AN INVESTIGATION OF ION EXCHANGE MEMBRANE

BATTERIES AS ELECTRICAL POWER SOURCES

By

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

HISTORY AND INTRODUCTION

Even before the turn of the century it was known that in
biological processes some membranes permit the passage of ions of
only one sign. The fact that such membranes would be useful in
studying natural phenomena and synthetic processes was recognized,
and some investigators fabricated artificial membranes which would
selectively pass ions having charges of a given sign; several
different materials were used, including cellulose and collodion
(3, 8). These membranes could be treated potentiometrically when
separating solutions of different activities, but they had the
common characteristic of offering high resistance to current flow.
Continued research with membranes coupled with the development of
efficient granular ion exchange resins and tough organic polymers
supplied the information needed to fabricate very durable membranes
of high electrical conductivity (1, 9).

The membranes used in this research are commercially available
Amberplex C-1 cation permeable membrane and Amberplex A-1 anion
permeable membrane manufactured by Rohm and Haas Company, Phila-
delphia. Amberplex membranes possess excellent mechanical dura-
bility; below 95° C they are highly resistant to concentrated
solutions of acids, bases and salts and to most common organic
solvents; (9). These membranes are similar to granular ion exchange
resins except that the former are in sheet form. The structure (7)
of the membranes is a three dimensional network of an insoluble

organic polymer. Their reactivity comes from active groups bound into the polymer. These groups can ionize and take part in ordinary ion exchange reactions. Cation permeable membranes have reactive acidic groups which ionize to leave negative charges attached to the polymer; anion permeable membranes have basic groups which ionize leaving positive charges on the polymer. In the interstices of the polymer there is water. Cation permeable membranes are capable of exchanging any cations which may be in this interstitial water. Anion permeable membranes can exchange anions in the interstitial water. It is evident that the mechanism of electrical conductivity is similar to that in aqueous solutions of electrolytes, i.e. by mobile ions. Random passages exist through the membranes, and in the ideal case the passages are just wide enough to permit ions to pass through only by displacing some of the ions on the groups lining the passages. The selectivity of a membrane for an ion of a given sign approaches the ideal case in dilute solutions; as the concentrations of the surrounding solutions increase, this selectivity decreases. It is necessary to use concentrated solutions in a battery in order for the battery to have minimum internal resistance.

There are theoretical expressions for the potential across a membrane (9). For membranes of perfect selectivity the potential is given by the Nernst potential expression

$$E = \frac{RT}{nF} \ln \frac{a_2}{a_1}$$

where

R = the gas constant

T = absolute temperature

F = Faraday's constant

n = charge per ion

a_1 and a_2 = mean ionic activities of the salts in the two solutions separated by a membrane.

For a membrane separating solutions which have large concentrations of ions compared to the fixed ion concentration of the membrane, the potential is the diffusion potential between two electrolytes

$$E = (t^+ - t^-) \frac{RT}{nF} \ln \frac{a_2}{a_1}$$

where

t^+ and t^- are the average transport numbers of cation and anion, respectively, in the solutions.

Figure 1 illustrates the battery of ten cells used in our work. The battery is essentially a series of concentration cells with transference. As in any apparatus in which a membrane separates solutions of different concentrations, osmotic pressure tends to force a particle through the membrane from the solution more concentrated with respect to the particle into the solution that is more dilute with respect to the particle. Hence, a solution of a salt will be negative with respect to a more dilute solution of that same salt when such solutions are separated by a cation permeable membrane. A dilute solution of a salt will be negative with respect to a more concentrated solution of that same salt when such solutions are separated by an anion permeable membrane. The series of compartments shown in Figure 1 is arranged, therefore, such that

```
 _                                                                          +
    1         2         3         4         5         6         7         8         9        10
        cpm       apm       cpm       apm       cpm       apm       cpm       apm       cpm

   2m        1m        2m        1m        2m        1m        2m        1m        2m        1m
   K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻     K⁺Cl⁻
```

charged battery

e⟶

K⁺⟶   ⟵Cl⁻ K⁺⟶   ⟵Cl⁻ K⁺⟶   ⟵Cl⁻ K⁺⟶   ⟵Cl⁻ K⁺⟶

Cl⁻   Cl⁻
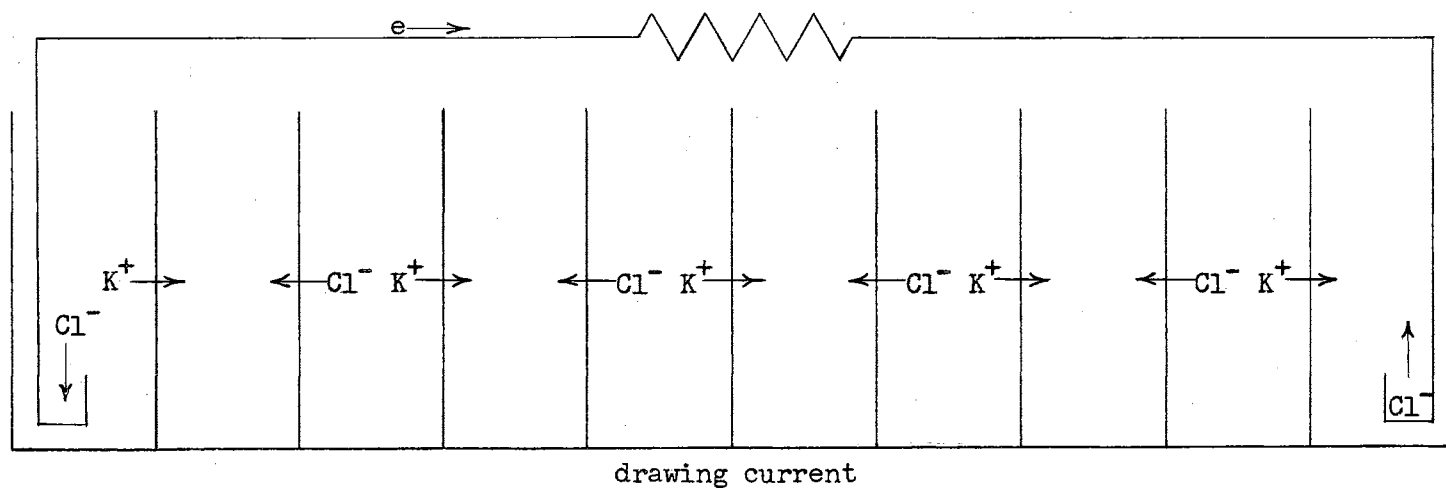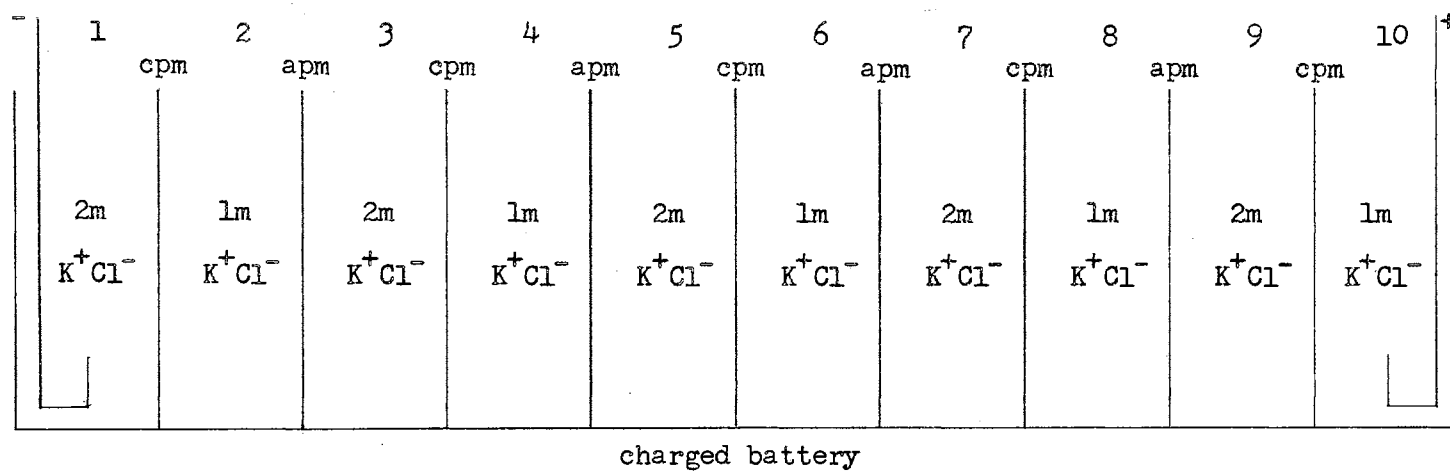
drawing current

Figure 1. Battery of 10 Cells

there is alternately a concentrated solution separated from a dilute solution by a cation permeable membrane and a dilute solution separated from a concentrated solution by an anion permeable membrane. Each compartment in this arrangement is positive with respect to the compartment to its left; therefore, the voltages across the membranes add just as in a series of electrochemical cells, and battery operation is obtained.

The electrodes need be reversible to only one ion. If the electrodes are in contact with solutions of the same concentrations, they do not contribute to the total electromotive force of the battery. If the electrodes are in contact with solutions of different concentrations, e.g. in a battery having an odd number of cells, the contribution of each is different.

SCOPE OF INVESTIGATION

This research has been entirely exploratory. The main idea has been to build a battery and to investigate its electrical characteristics when used as a source of power. Some study has been made of the chemical processes occuring in the battery.

ELECTRICAL APPARATUS

Potentiometers

Leeds and Northrup Student Potentiometer.

Brown Recording Potentiometer Model No. 153X12V-X-30; 0-10 millivolt range.

Resistance Boxes

Decade Resistors:

Central Scientific Co. No. 82821C with steps of 0.1, 1, 10, 100, and 1,000 ohms.

Industrial Instruments Inc. with steps of 0.1, 1, 10, 100, and 1,000 ohms.

Oklahoma A. and M. Department of Electrical Engineering with steps of 1, 10, 100, and 1,000 ohms.

Plug Resistance Box:

Central Scientific Co. with 12 plugs for 400, 300, 200, 100, 40, 30, 20, 10, 4, 3, 2, and 1 ohms.

Galvanometer for Manual Potentiometer Circuit

Leeds and Northrup Portable Pointer Type; sensitivity of 0.4 micro-amperes per scale division and period of 3 seconds.

## CONSTRUCTION OF APPARATUS AND ROUTINE PROCEDURES

Battery Construction

A battery was constructed from Plexiglass. Each cell was
fabricated from an individual piece of the plastic (Figures 2, 3).
The cells were bolted together with the membranes held firmly
between them. When the bolts were drawn up tightly, the membranes
acted as their own gaskets and prevented leakage of solutions. The
end cells differed from the center cells in that the former were
made larger to allow for the volume occupied by the electrodes.
Also, the end cells had larger holes in their tops to allow the
electrodes to be put inside.

Electrodes

Calomel, $Hg-Hg_2Cl_2$ electrodes were used. They differed from
ordinary calomel electrodes in that they were not used as reference
electrodes but only as receivers and sources of chloride ions. The
surface of $Hg-Hg_2Cl_2$ was exposed directly to the solutions in the
end cells of the battery. Figure 4 shows the electrode construction.
The layer of mercury needs only to be thick enough to make good con-
tact with the platinum wire. The layer of paste must be thick
enough to keep the mercury layer in place during handling of the
electrodes; a 3 to 5 mm layer is thick enough. The glass lead is
connected to the cup of the electrode by black sealing wax which
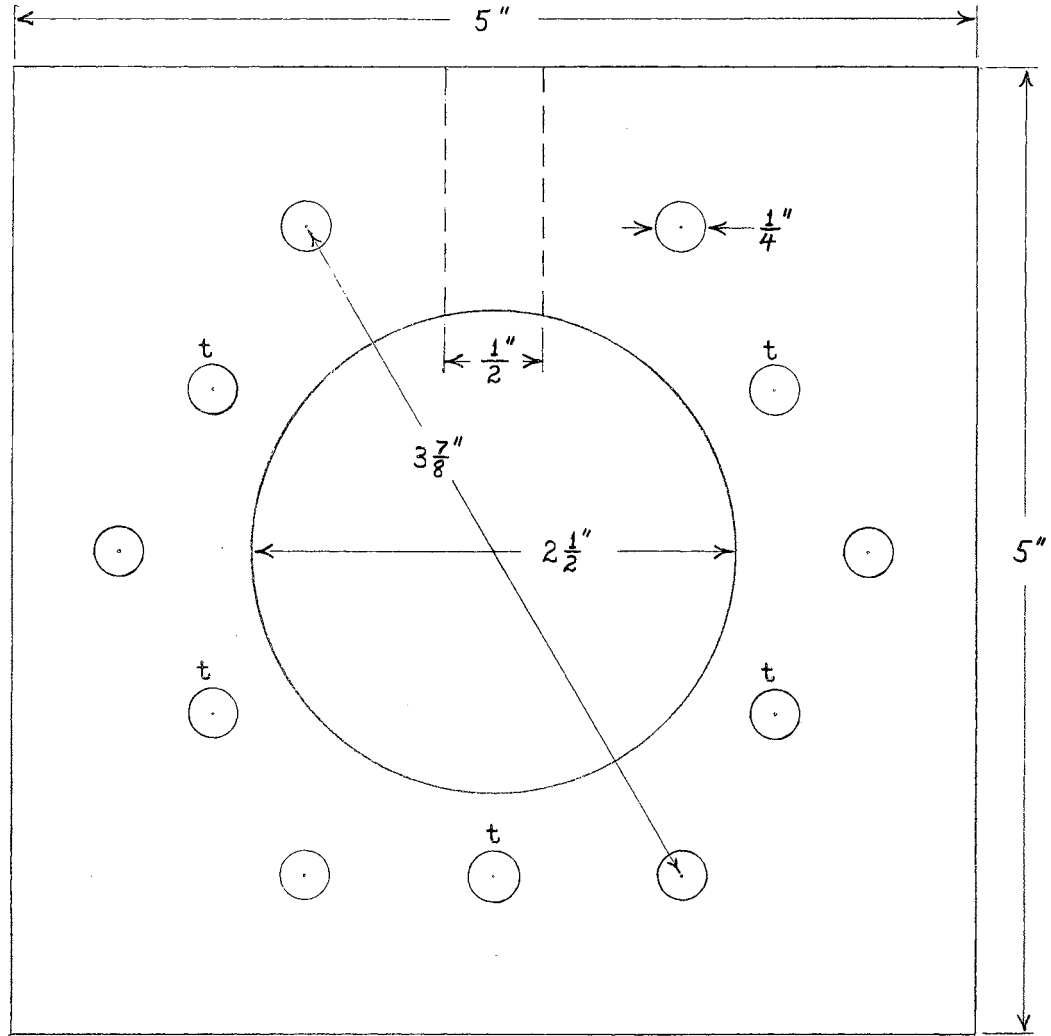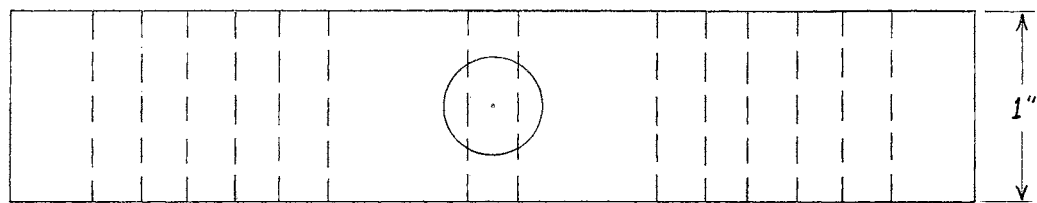is quite hard at room temperatures.
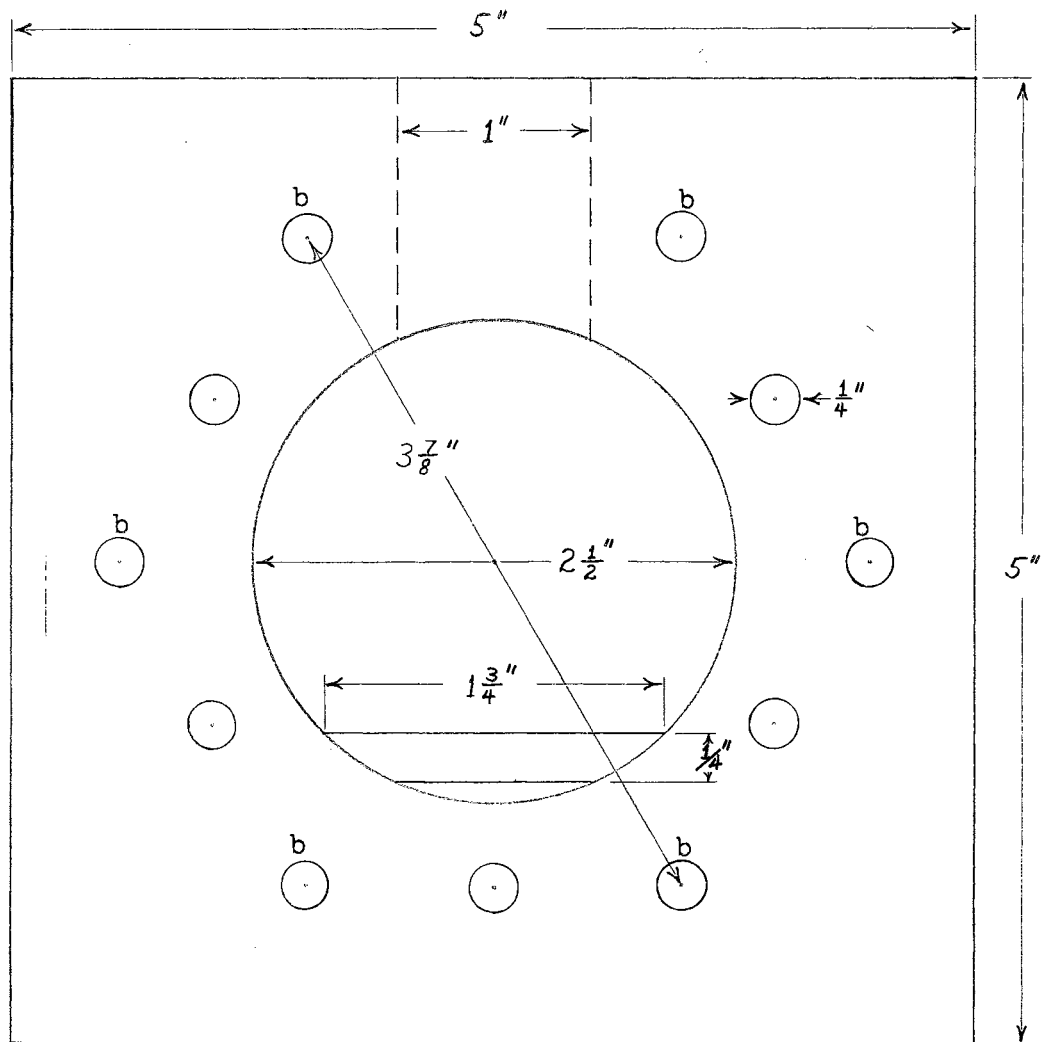
b = bolt hole

inside view
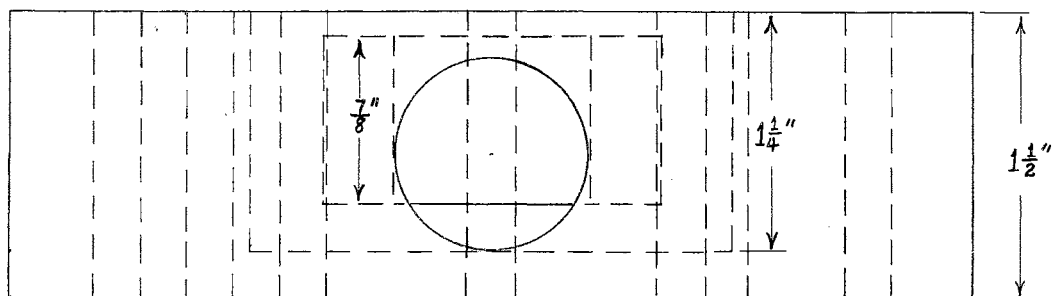
top view

Figure 2. End Cell of Battery

t = tubing hole

side view

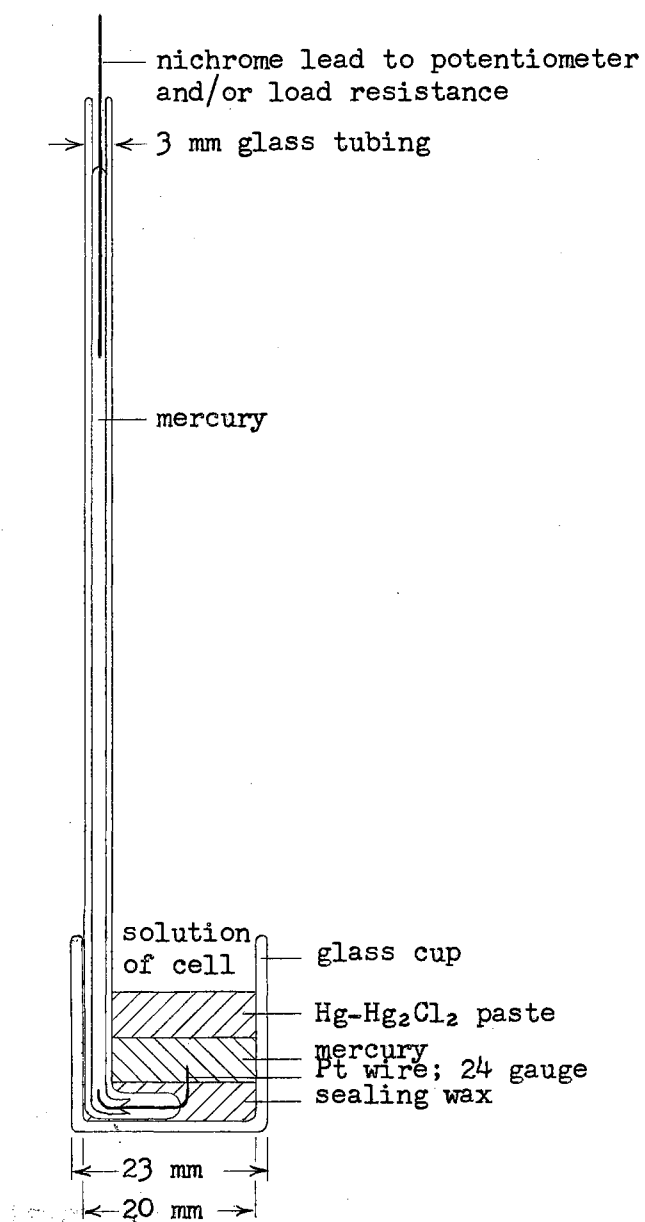top view

Figure 3. Middle Cell of Battery

nichrome lead to potentiometer
and/or load resistance

3 mm glass tubing

mercury

solution
of cell

glass cup

$Hg-Hg_2Cl_2$ paste

mercury
Pt wire; 24 gauge
sealing wax

23 mm

20 mm

Figure 4. Electrode

Electrolytes

Water solutions containing KCl of one and two molal concentrations were used as the electrolytes in the battery. KCl solutions were chosen because there are abundant thermodynamic data available for them, consequently, the theoretical treatment of the results is facilitated. Concentrations are recorded in molal units because they are easily handled in thermodynamic calculations. One and two molal solutions were chosen so that the battery would have reasonably low internal resistance. Time has not been available to extend the studies to solutions of different concentrations.

Preparation of Membranes

The membranes were obtained as sheets four inches square. In order to convert the mobile ions in them to potassium and chloride ions, if they were not as such when received, the membranes were removed from their packages and soaked in approximately 800 ml of potassium chloride solution of approximately 1.5 molal concentration. The solution was changed after the membranes soaked for about an hour; this was done five or six times. The membranes were then cut to shape (see Figure 5) and put between the cells. They were kept wet at all times to prevent shrinking and cracking; the cracking or splitting would occur if the membranes were held rigid and were allowed to dry out. While not in use, the battery was kept filled with potassium chloride solution of 1.5 molal concentration.
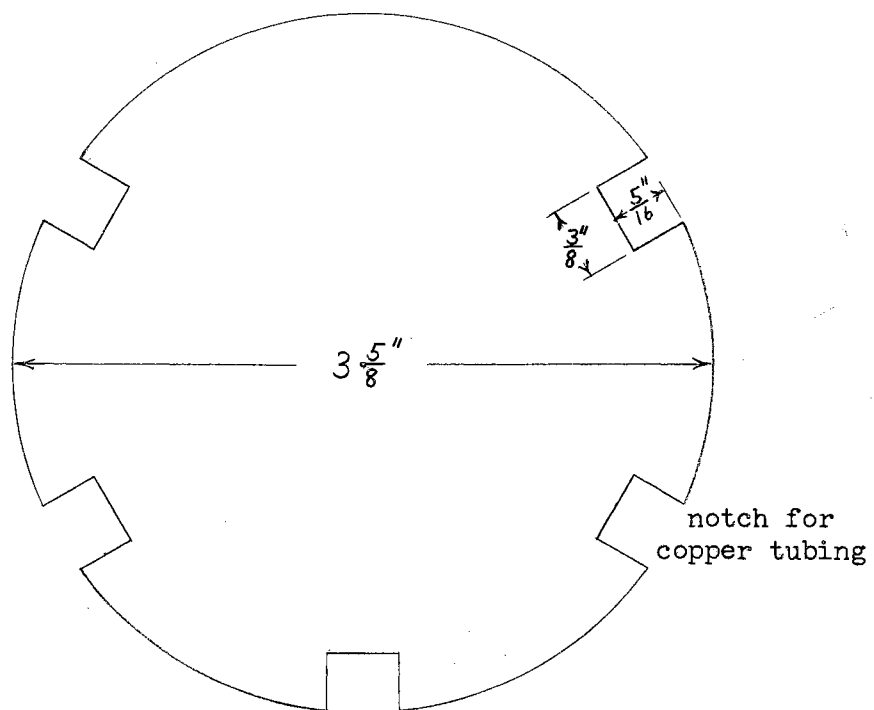
$3\frac{5}{8}''$

$\frac{5''}{16}$

$\frac{3''}{8}$

notch for
copper tubing

Figure 5. Membrane

Filling the Battery

The battery, filled with 1.5 molal KCl solution, was brought
to the desired temperature and kept there. The solutions to be
used for the actual experiments were brought to proper temperature.
The cells of the battery were emptied by means of a 100 ml pipet and
a suction bulb. The bulk of the solution was removed by putting the
end of the pipet with the large opening into the solution and drawing
on the other end with the bulb; the small remainder of the solution
was removed by putting the bulb over the large opening and sticking
the small tip of the pipet into the solution. Each cell was rinsed
with 15-20 ml portions of the solution with which it was to be
filled during the experiment. This rinse solution was then with-
drawn. The usual procedure in rinsing the cells was to rinse the
odd numbered cells first, i.e. those which were to contain 2 molal
KCl solution during the experiments. This left every other cell
full of solution, so that there would not be sufficient loss of
moisture from the membranes to cause them to shrink and split.
Then the other cells were emptied and rinsed. The time during
which there were no solutions in the battery was not long enough
to damage the membranes. The solutions were poured into the bat-
tery very rapidly from the bottles in which they were brought to
proper temperature. Each bottle held approximately one liter of
solution and was fitted with a rubber stopper with two holes through
which were two lengths of glass tubing bent above the stopper in
opposite directions. One of the pieces of tubing extended almost to
the bottom of the bottle, and the other passed only through the

stopper. Solutions can be poured quickly into the battery by means of this arrangement.

Temperature Control

Several methods of maintaining constant temperature were tried, but the most successful method for practical purposes, which was used exclusively after its worth had been proven, is described. Five holes were drilled the length of the battery between the bolt holes. Lengths of copper tubing were fitted snugly into these holes. Water at constant temperature was pumped through the tubing. The battery was insulated from the atmosphere on its bottom and sides by putting the battery in a cardboard box which was about two inches larger than the battery all around and filling the space between the box and the battery with mica packing.

Stirring of Solutions in the Battery

In order to reduce concentration polarization to a minimum, thereby permitting consistent results, the solutions were stirred by means of the stirrer shown in Figure 6. An adjustable speed stirring motor was mounted so that its chuck was in a horizontal position. The stirrer was connected to a bent shaft which was held in the chuck. The rotation of the bent shaft caused the stirrer to move up and down a distance of three-eighths inch per revolution. The speed of the motor was kept at approximately 240 revolutions per minute.
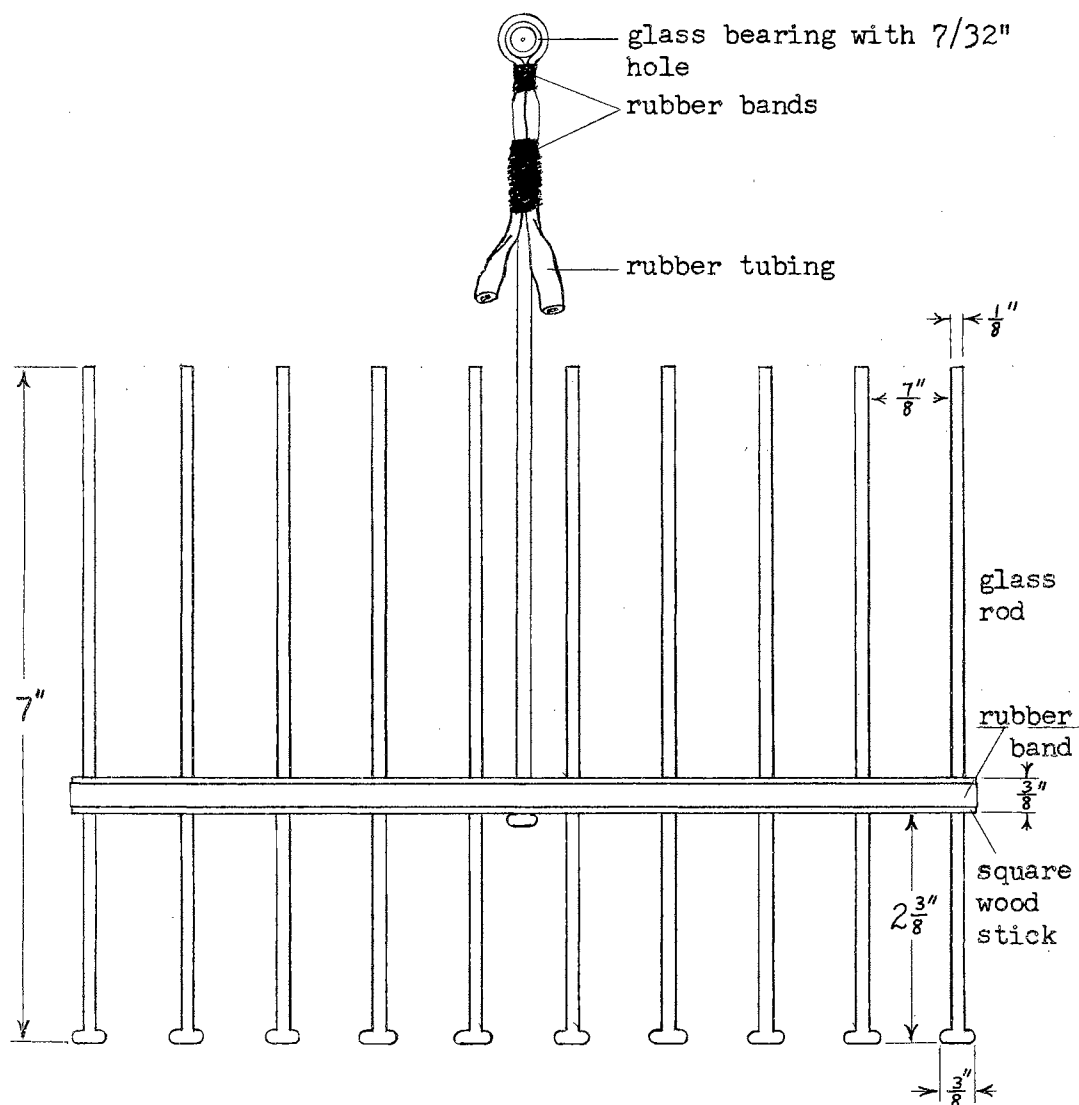
glass bearing with 7/32"
hole

rubber bands

rubber tubing

$\frac{1}{8}''$

$\frac{7}{8}''$

glass
rod

rubber
band

$\frac{3}{8}''$

$7''$

$2\frac{3}{8}''$

square
wood
stick

$\frac{3}{8}''$

Figure 6. Stirrer

Determination of Individual Cell Volumes

The pieces of Plexiglass used for the center cells were all machined to the same dimensions. The same is true for the two end cells. However, the volumes of the cells varied when the battery was assembled. This is due to the fact that the surfaces of the membranes were not perfectly flat when the membranes were held in the battery; they were somewhat convex or concave. This condition of the membranes was particularly noticeable after the battery, which had been assembled for several months, was taken apart and then reassembled with the same membranes. The membranes had a slight permanent set to them after being held tightly for so long a period of time. When new membranes, which were soaked in approximately 1.5 molal KCl, were used when the battery was reassembled, the center cells had approximately the same volumes, and the two end cells had approximately the same volumes. The volumes of the individual cells were determined by first filling all of the cells with 1.5 molal KCl so that the bottom of the meniscus of the solution in each cell was even with the top of the bolts which went lengthwise through the top of the battery. Then the solution was removed from a single cell. The empty cell was filled with 1.5 molal KCl from a burette. The volume of the solution needed to fill the cell to the original mark was taken as the volume of the cell. This procedure was repeated for each cell.

Volumes of Electrodes

The volume of each individual electrode was determined from the quantity of solution it displaced in a partly filled graduated cylinder.

Chloride Analysis

Fajen's method (4), which employs standard silver nitrate solution as titrant, dichloroflourescein as indicator, and dextrin as anti-coagulant, was used to determine chloride concentration of the solutions where analyses are mentioned in the next section of this paper. A very accurate pipette of 5 ml total delivery was used to transfer a sample of solution to a 125 ml Erlenmeyer flask. This sample was then diluted to approximately 50 ml with distilled water. One-tenth gram of dextrin and 0.3 ml indicator were added. The KCl solutions from the battery which were originally 2.00 molal were accurately titrated with 0.25 N silver nitrate solution, and those solutions which were originally 1.00 molal were titrated with 0.13 N silver nitrate. A graph of molal concentration against normal concentration was made from density values for solutions of various percentages of KCl in water (2). The normal concentrations obtained as a result of analysis were converted to molal concentrations by means of the graph.

Types of Batteries

Experiments were performed with two batteries. One battery was of an even number of cells, ten. Figure 1 is an illustration of such a battery. The other battery had eleven cells, and it is illustrated in Figure 7.
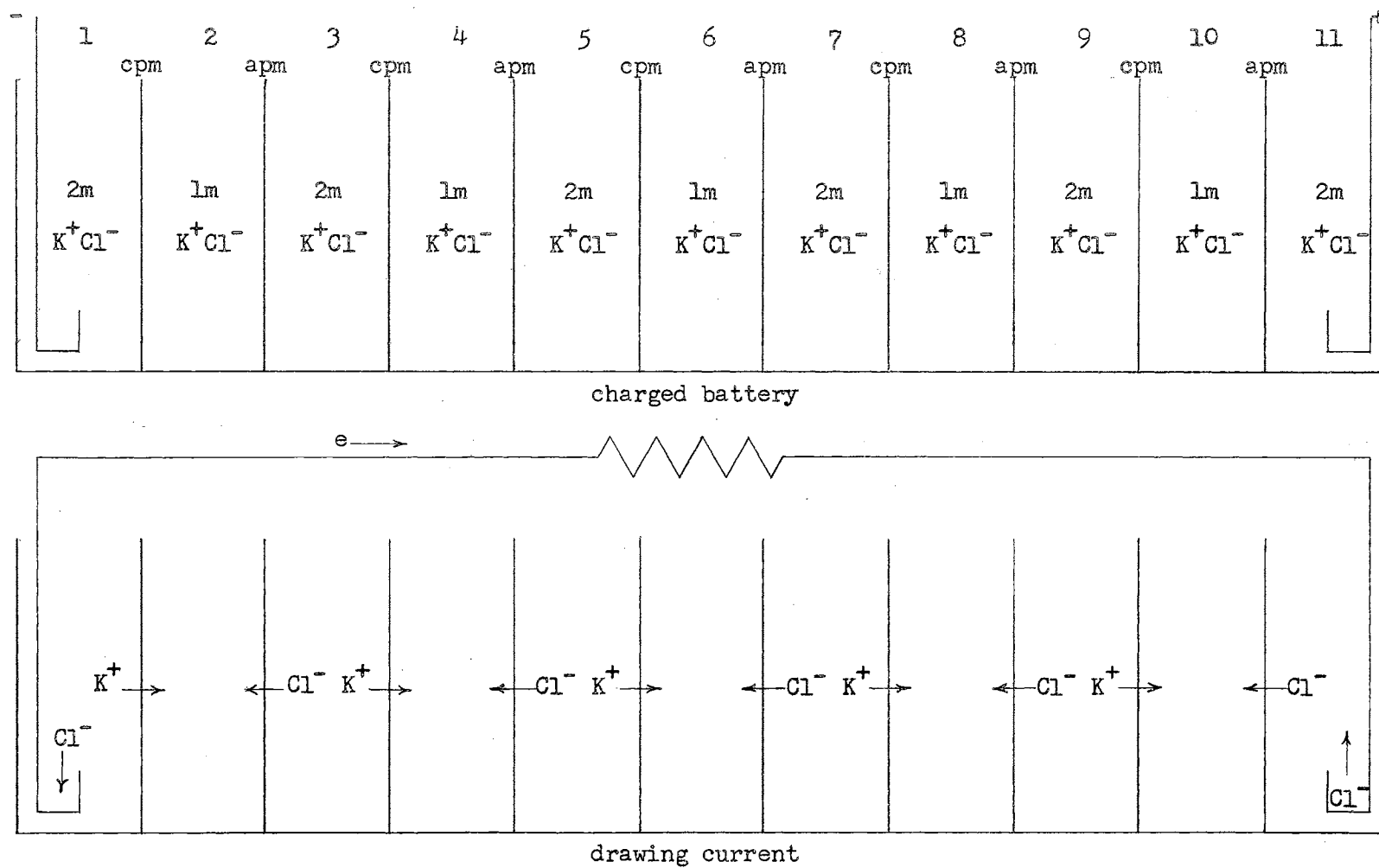
Figure 7. Battery of 11 Cells

ELECTRICAL EXPERIMENTS

In the first experiments the variations of the open circuit
potentials of the battery determined at 25 $\pm$ 1° centigrade were about
2 millivolts. Two major factors caused the two millivolt spread: (1)
the failure to make emf measurements at exactly the same time after
the battery was filled with solutions; the diffusion of cations
through the anion permeable membranes and anions through the cation
permeable membranes caused the potential to decrease from the moment
the solutions were put into the battery, and (2) temperature control
of $\pm$ 1° C was not close enough. Results which agreed more closely
at a single temperature were wanted so that change with temperature
of initial open circuit emfs could be determined accurately. The
problem was solved by (1) controlling temperature to $\pm$ 0.10° C or
better, and (2) timing the voltage measurements in each determination
with respect to a common starting point in the procedure. A stop-
watch was started at the point when cell number 6 was half full with
1.00 molal KCl. The temperature of the solution in each cell was
taken after the cells were filled. The solutions were then stirred
constantly and a series of emf measurements were taken until 25 - 30
minutes after the zero time. A graph of emf against time was made,
and the value obtained by extrapolating back to zero time was taken
as the initial open circuit potential. By this method initial open cir-
cuit potentials can be reproduced within 0.0005 volt. This procedure
was used for determinations at several temperatures. Figure 9 is a
graph of initial open circuit potentials versus temperature.
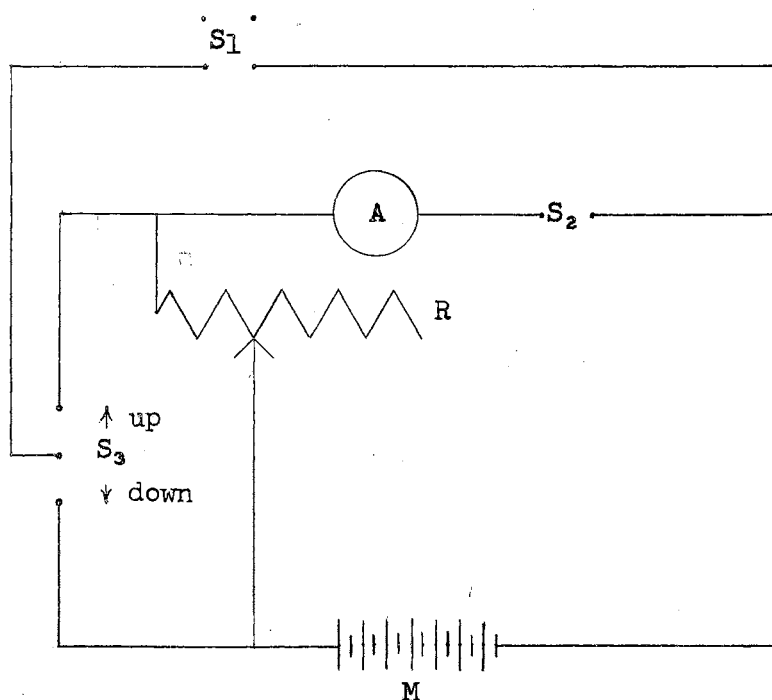
A — either a microammeter or a set known resistance

R — load resistance

M — ion membrane battery

S_1— dp switch to manual potentiometer

S_2— spst switch

S_3— spdt switch

S_2 closed, S_3 down — gives emf of ion membrane battery when current

is being drawn.

S_2 closed, S_3 up — gives emf drop across microammeter or a known

resistance.

S_2 open, S_3 down — gives open circuit potential of ion membrane

battery.

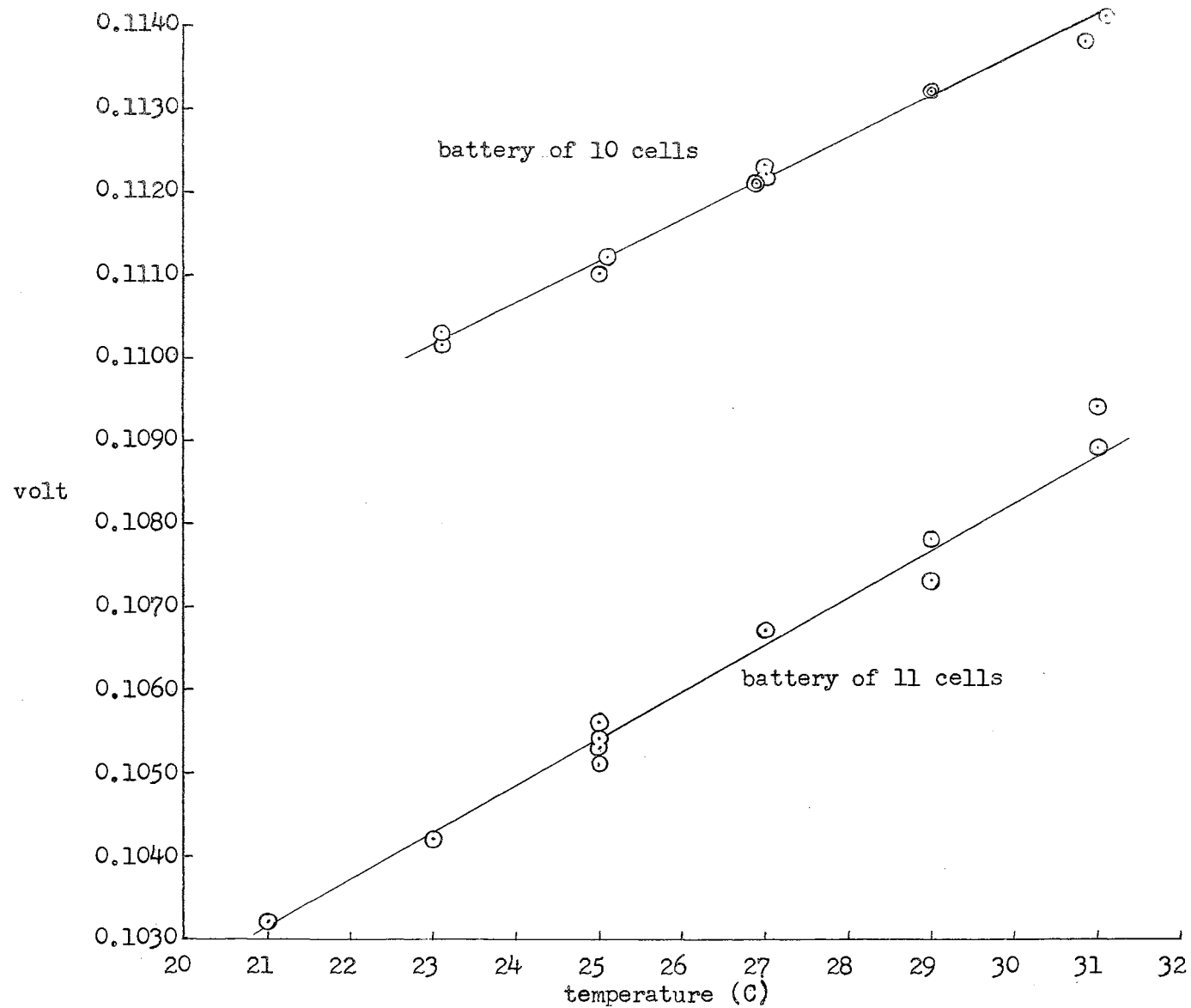Figure 8. Basic Circuit Used for Experiments with Battery

Figure 9. Initial Open Circuit Potentials Versus Temperature

Change of Open Circuit Electromotive Force with Time

Open circuit potentials were determined with the battery of 11 cells at three temperatures, 21, 25, and 29 degrees centigrade over a period of three days each. The battery was filled with 1.00 and 2.00 molal solutions, and the change in emf was followed with no current withdrawal. Temperature was controlled to $\pm$ 0.5°. Figure 9 shows that initial open circuit emf increases linearly with increase in temperature, so that the initial open circuit emf at 29° is greater than that at 25° which in turn is greater than the emf at 21°. The open circuit potential decreases quite rapidly due to diffusion of ions through the membranes. If a graph is made of emf against time for each of the three temperatures, and if the graphs are superimposed (Figure 10), it can be seen that the emfs at all three temperatures are about the same between 8 and 14 hours; at times greater than this, the open circuit emf at 21° is the greatest of the three and the emf at 29° is the least. The reason for this is believed to be the differences in mobilities of the ions at the different temperatures. When the temperature is 29°, there is greater rate of ion transfer through the membranes so that the ratios of concentrations separated by the membranes, upon which the potential depends, become smaller more rapidly than these ratios do at 21°. After 72 hours have elapsed, the potentials at all three temperatures are between 0.011 and 0.016 volt.

Performance of Batteries when Delivering Current

It has already been stated that the voltages of the batteries drop from the moment the solutions are put into the batteries. For
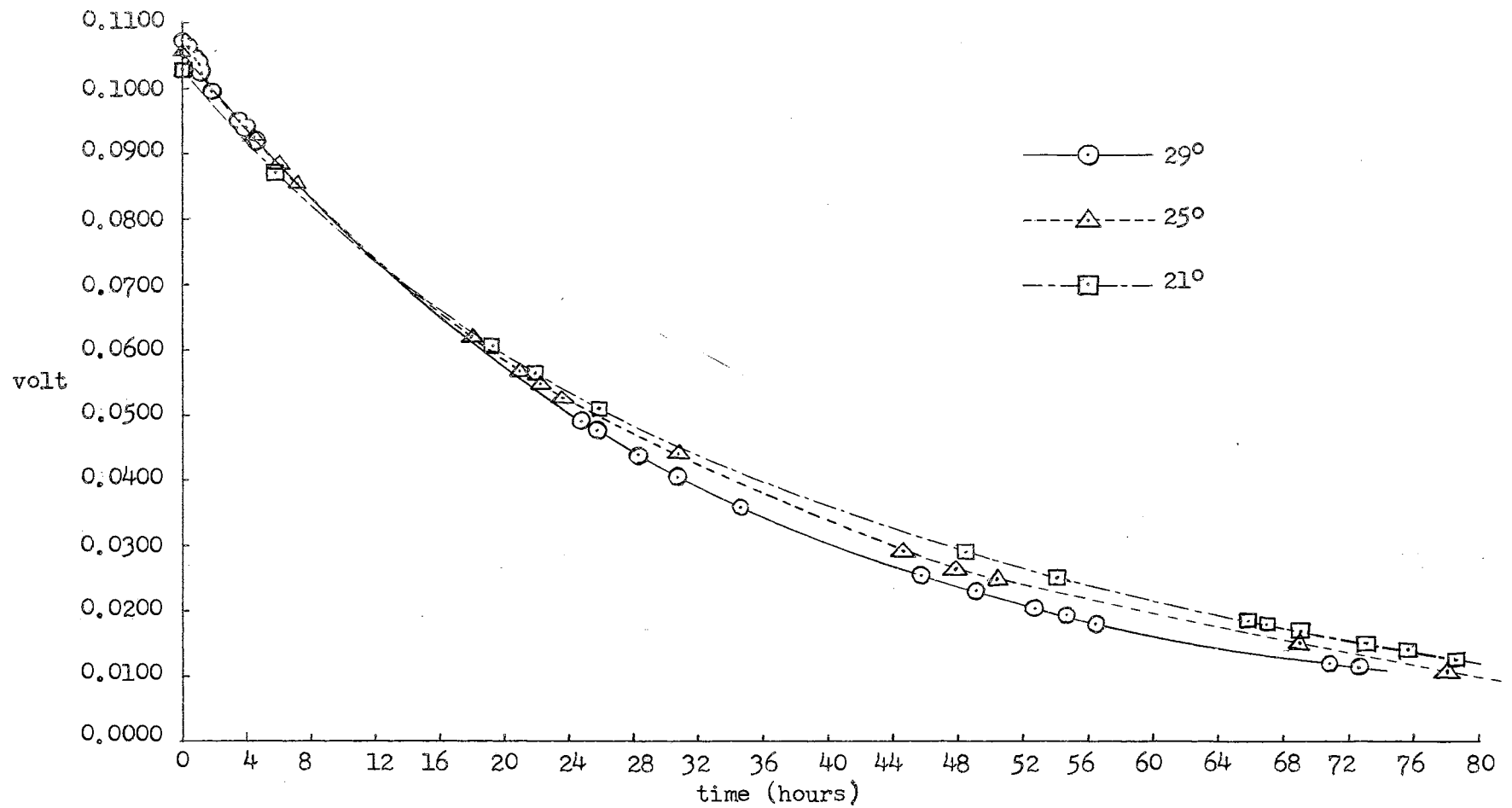
Figure 10. Change of Open Circuit Potentials with Time of 11 Cell Battery

these experiments where current was drawn, two electrodes were used
in each end cell. Constant current was maintained by connecting two
resistances, one fixed and the other variable, across the electrodes;
the variable resistance was adjusted every few minutes so that the
voltage drop across the fixed resistance remained constant. A graph
of the potential measured across the battery terminals against time
when the battery is delivering a constant current to a load is
parallel to a similar graph of the no-load voltage of the battery.
Results obtained for the battery of 11 cells and 10 membranes are
similar to the results obtained for the battery of 10 cells and 9
membranes except that the former battery has lower electromotive force.

Figures 11 and 12 show graphs of the drop in the emf across the
battery terminals against current drawn for batteries of 10 cells and
11 cells, respectively. The ordinate, which represents the difference
between open circuit and closed circuit voltages, shows the value of
the voltage which is subtracted from the open circuit potential when
a given current is being drawn. These graphs were obtained by mea-
suring the open circuit voltage, placing a fixed resistance across
the electrodes and following the potential drop for several minutes
until the drop became relatively constant; then the circuit was
opened, and the potential of the battery was followed until its rate
of decrease was again relatively constant. During the few minutes
when the circuit was closed and the voltage dropped constantly, the
current also decreased, but its decrease was not great compared to
the total current being drawn. Each value of current on the graph
is the average value of the current drawn.
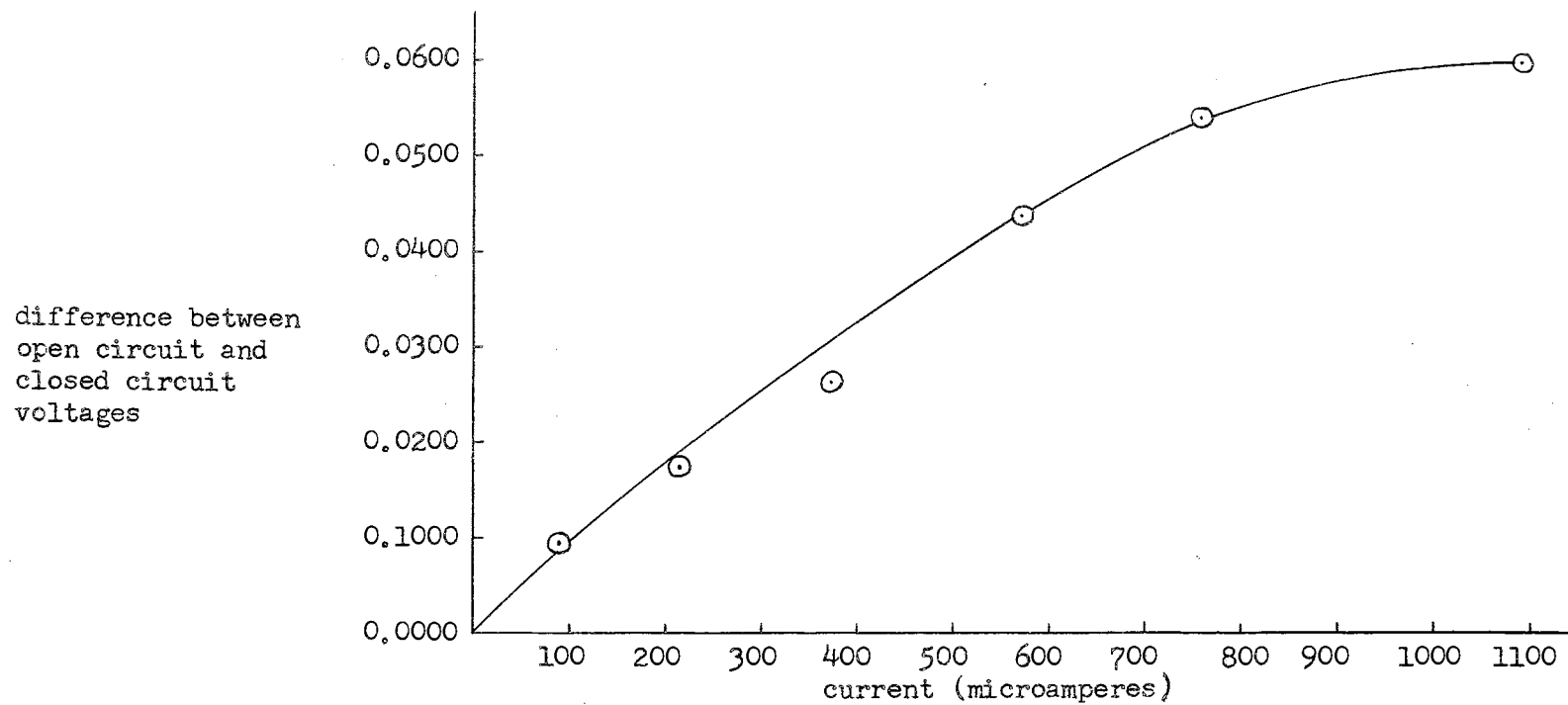
Figure 11. Decrease in Open Circuit Potential Against Current of 10 Cell Battery

difference between
open circuit and
closed circuit
voltages

0.0500

0.0400

0.0300

0.0200

0.0100

0.0000

100 200 300 400 500 600 700 800 900 1000 1100 1200
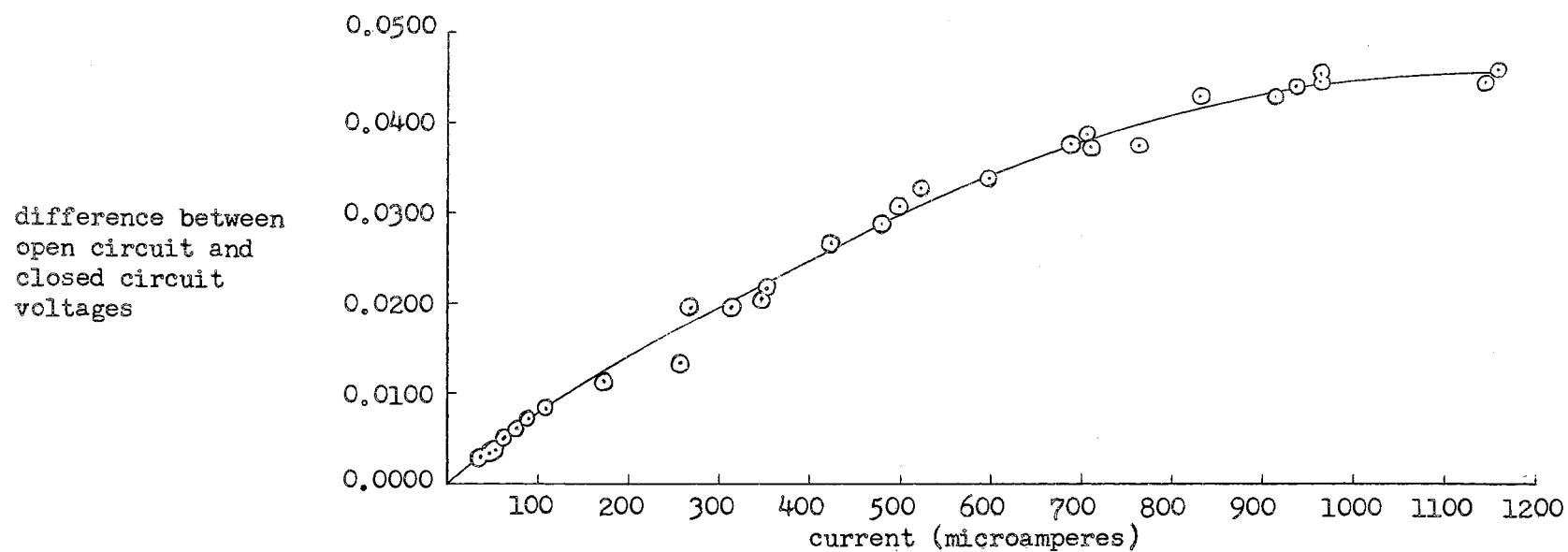
current (microamperes)

Figure 12. Decrease in Open Circuit Potential Against Current of 11 Cell Battery

Polarization and Internal Resistance of the Battery

Polarizability of the electrodes was studied independently of the batteries by placing the electrodes in the same solution and passing a constant current through them. They carried currents of up to 200 microamperes for several hours with only a slight change of resistance during the period. The results were the same with larger currents for shorter periods of time. The electrodes eventually became polarized, but when this happened, their resistances increased very rapidly within a minute of time; then only a small fraction of the former current passed through them. Breaking the circuit for a few minutes and then closing it again did not cause the electrodes to return to their original condition. These experiments indicate that electrode polarization cannot be the cause of the large drop in emf of the battery when current is drawn.

To investigate further the operation of these batteries, a recording potentiometer (Figure 13) was attached to one pair of electrodes in a battery of 10 cells while current was drawn from the battery through the other pair. The voltage measured across the electrodes which carried no current was much greater than the voltage across the electrodes which carried current. Figure 14 shows a graph of the drop of potential registered by the recording potentiometer against current drawn from the battery.

It seems justifiable to assume that the voltage across these recorder electrodes differs from the open circuit voltage of the battery for one reason only. In the former there may be polarization occurring at the membranes to reduce the potential. Since in neither case do
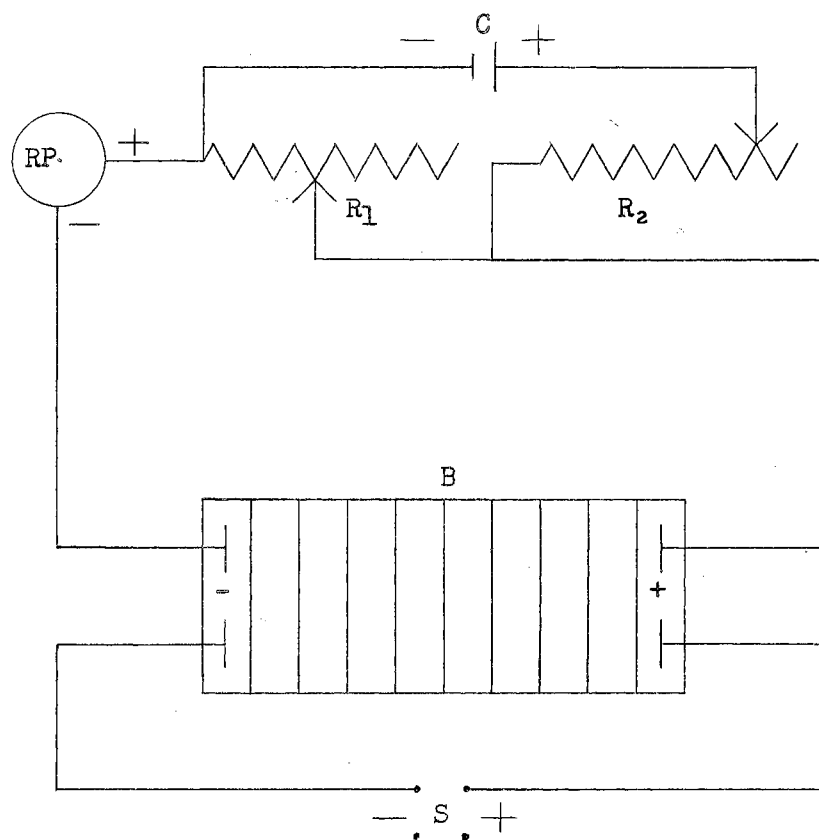
C - 1.5 volt dry cell

RP- recording potentiometer, 10 mv range

R_1- decade resistor

R_2- decade resistor

B - ion membrane battery; cell 10 opposes positive poles of
    potentiometers.

S - dp switch to manual potentiometer

R_1 and R_2 are kept adjusted so that the 10 mv range of the recorder
is utilized.

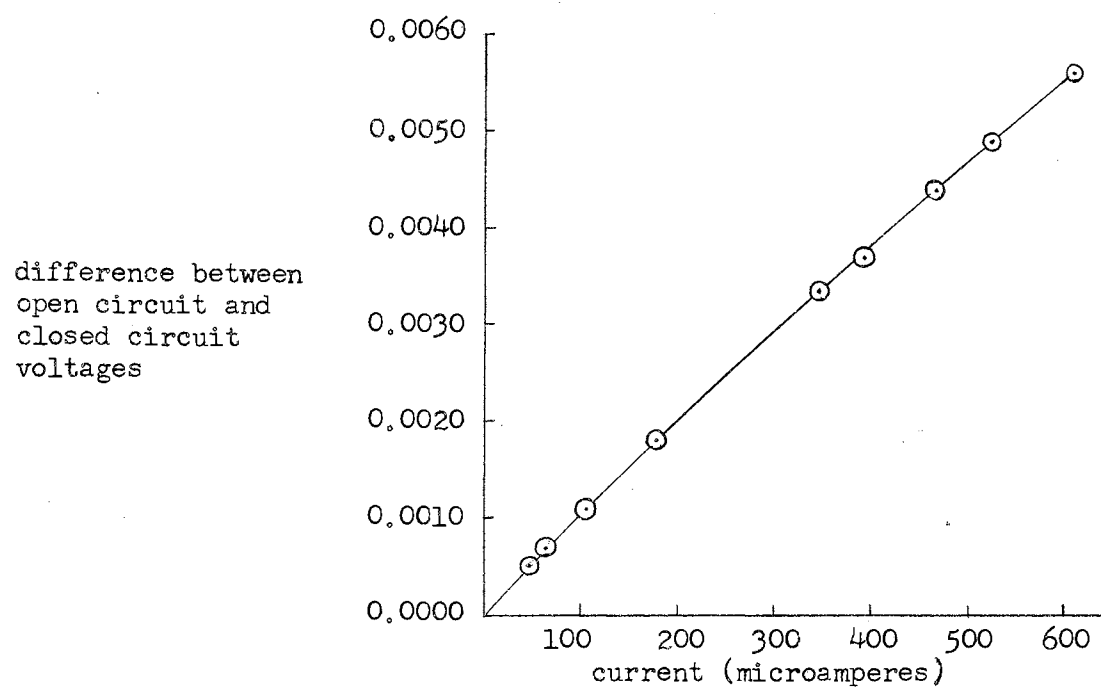Figure 13. Circuit with Recording Potentiometer

Figure 14. Decrease in Open Circuit Potential Registered by Recording
Potentiometer

the electrodes carry current, there can be no electrode polarization effects. In all cases the membrane polarization voltage was small, and since independent experiments described at the beginning of this section indicated electrode polarization to be negligible, it is believed proper to ascribe the remainder of the potential drop observed to the internal resistance of the battery.

Figure 15 is a schematic diagram of the battery used in the measurements together with circuits including potentiometers and resistances. The following symbols are used to represent the electrical quantities involved:

E_O = no load potential of the battery

E_n = potential across the electrodes carrying no current when the other pair of electrodes is delivering current

E_L = potential across the battery electrodes delivering the load current

R = the load resistance for the battery

R_i = the internal resistance of the battery

e_m = the polarization voltage across the membranes.

If one applies Kirchhoff's second law to the two closed circuits, one can write

$$0 = E_n - E_O + e_m \tag{1}$$

$$\text{and } 0 = E_O - e_m - I_L R_i - I_L R_L \tag{2}$$

$$\text{but } I_L R_L = E_L$$

$$\text{or } I_L = E_L / R_L$$

$$\text{therefore, } 0 = E_O - e_m - E (R_i / R_L + 1). \tag{3}$$

Adding equation 1 to equation 3, one obtains an expression for R_i

recording
potentiometer

E_o or E_n

ion membrane battery

R_i e_m

+   –

I_L E_L

R_L

manual
potentiometer
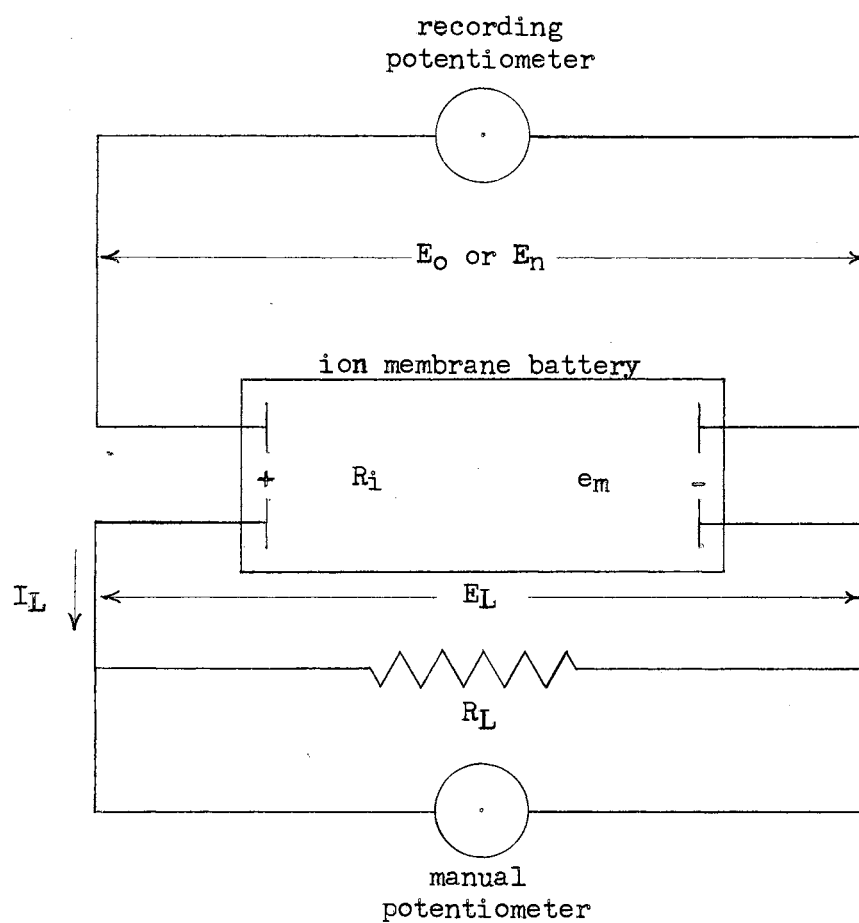
See text for definition of symbols and explanation.

Figure 15. Schematic Diagram of Circuits Used With Recording
Potentiometer

which is independent of e_m. Thus

$$R_i = R_L(E_n/E_L - 1).$$

The electrode polarization voltages have been ignored for reasons given earlier.

Table 1 shows the internal resistances of the battery calculated from data taken at several load currents.

Table 1

I_L (microamps)	49.4	63.3	106	178	346	393	467	525	610
R_i (ohms)		235	228	210	189	135	133	127	120	113

It is noteworthy that the internal resistance of the battery decreases with the increasing current. Such behavior is not commonly encountered in battery performance.

Comparison of Calculated and Experimental Open Circuit Potentials

In order to compare the value of the Nernst potential with experimental potentials, three similar experiments were performed: the 10 cell battery at 25° C was filled with 1 and 2 molal KCl solutions at 25° C, the potential was measured at a predetermined time in each of the three experiments and the solutions were quickly withdrawn from the battery for chloride analysis. The three experiments differed only in their predetermined times of 80, 160 and 240 minutes. For these experiments it was desired to have all of the cells of the battery hold the same volume of solution, so the battery was fitted with new membranes. The end cells were filled with the same quantity of solutions, 80-82 ml, that the center cells hold when full.

By using the E^o value for the calomel electrode (5) and activity values for KCl (6) the potential of the battery was calculated.

Table 2 shows the results. It is realized that the calomel electrodes were not in equilibrium with the KCl solutions during the experiments and that the E^O value is for equilibrium conditions.

Table 2

Time (minutes)	0	80	160	240
Experimental emf (volt)	0.1134	0.1112	0.1084	0.1056
Nernst emf (volt)	0.1528	0.1478	0.1434	0.1401

When all of the potentials, not just those shown in Table 2, of the above three experiments are plotted against time, they all are on the same line. Note that the initial emf for these experiments at 25° is higher than the potential at 25° shown in Figure 9. The reason for the difference is believed to be due to the use of membranes of different lot numbers, because the experimental procedure was the same throughout.

DISCUSSION AND SUMMARY OF THE RESULTS

The performance of the ion exchange membrane battery is determined largely by diffusion processes. This idea is borne out by two experimentally observable characteristics: a) the potential of the battery drops steadily with time whether or not a load current is flowing, and b) the rate of decrease of the potential with time increases with temperature.

The potentials of such batteries cannot be expected to be those calculated from thermodynamic data because the systems do not approximate equilibrium systems.

Polarization occurring either at the membranes or at the electrodes during the delivery of current seems to be quite small.

The internal resistance of the ion exchange membrane battery is relatively high, even when moderately concentrated salt solutions are used in its compartments, and the maximum potentials possible are those calculated for concentration cells, which have low potentials. Because of these facts the power obtainable from such a battery as constructed and tested is at best a few microwatts. Because some molecules and ions are continually passing through the membranes, the battery will not hold a charge for long. In its present form, the ion exchange membrane battery offers little promise as a power supply.

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