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A STUDY OF ELECTROCHEMICAL PROCESSES
IN SILVER-CADMIUM SECONDARY CELLS

July 14, 1965

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Status Report on NASA Order No. S-70008-G

A Study of Electrochemical Processes in Silver-Cadmium Secondary Cells

Immediately after receiving Forming-Cycle instructions from NASA in December 1964 a study of the discharge characteristics of Ag-Cd cells was begun. The first experiment was to conduct the normal 300 ma charge and 1.00 amp discharge at room temperature. This was to be followed by observing the effect on charge and discharge which resulted from changes in rates of charge and discharge, in temperature, in electrolyte level, in electrolyte concentration and in partial charge and discharge in both the AgO-Ag₂O and Ag₂O-Ag ranges. All cell tests were conducted in duplicate.

The effect of the charge rate, after discharge to 1.08V.

Two cells (S/N 1122 and 1055, made April 1961) received in the activated state, but of unknown history, initially had about 3.4 hr capacity to 0.90V, exclusively in the Ag₂O-Ag range. After charging at 300 ma to 1.65V, the capacity was about 1 hr to 1.08V and 5 more hrs to 0.90V. This was true whether the discharge was soon after charge or delayed 430 hrs. A recharge at 300 ma after stopping the discharge as soon as 1.08V was reached did not give a better subsequent capacity in the AgO-Ag₂O region. Again the cells were taken off discharge as soon as 1.08V were reached. This time the cells were recharged at 10 ma for 90 hrs, though the voltage climbed to 1.5V. Now the initial CCV's were 1.16 and 1.14, and the capacities to 1.08V were 0.5 and 0.1 hrs, respectively. Another stoppage was made at 1.08V. An overnight stand on open circuit resulted in a recovery of OCV to 1.40V, but further discharge of less than 0.02 hr. Hence, recovery is negligible. The cells were then charged at constant voltage of about 1.46V for 1800 mah, after having been brought back to 1.40V while charging at 300 ma. Sufficient current was passed through the cells for the production of considerable AgO, but none was produced, judging from the next discharge of the cells. The initial CCV on 1.00A discharge was 1.05V. A peak voltage of about 1.075V was reached after 2 hrs and 1 hr for the two cells. Full capacity of 6.5 hrs was achieved. (See figure 1.) This last discharge agrees with those shown on graphs by Sisemore, and Hennigan and Apelt, but with the differences that a high current (300 ma) may be used to a voltage of about 1.4V and that a low maximum voltage must be maintained.

The next discharge was not normal, though the charge was at 300 ma to 1.6 volts. The initial CCV's on 1 amp rates were 1.15 and 1.17V. These voltages dropped to 1.075 and 1.070 in 0.5 hr, recovered to 1.08 and 1.077V at 1.85 hrs and then proceeded to give more than 6 hrs to

0.90V. Complete recovery to normalcy resulted in the next cycle.

If the discharge of a cell is continued for 0.2 hr after the voltage has reached 1.08, the OCV is still 1.41V, that of AgO to Ag_2O with Cd. Just how much longer than that has not yet been observed. Maybe the AgO exists after it is thought to be decomposed to Ag_2O . A fully charged cell let stand on open circuit over night also has a 1.41 OCV.

Some peculiarities in voltages during cycling Ag-Cd cells.

Cells 1821 and 1828 were used exclusively to study in detail the voltages of cells during charge and discharge cycles at room temperature. Normal discharge capacities of about 6.8 hrs to 0.90V were obtained. In the second charge cycle after the forming period (and in later cycles) the initial CCV was 1.22. After 0.1 hr the voltage dropped to 1.17, but the minimum of 1.167 was not reached until 1.5 hrs elapsed. Inevitably during the change from the $\text{Ag-Ag}_2\text{O}$ to the $\text{Ag}_2\text{O-AgO}$ range a similar voltage fluctuation occurred. The greatest surge observed was from 1.300V to 1.553V to 1.505V, all this change taking only a few minutes.

During discharge no voltage drop-rise-drop was observed in the $\text{AgO-Ag}_2\text{O}$ range. This may be due to the rate of discharge being over three times the rate of charge and the voltage change might pass unnoticed. It is more likely that AgO is never the only compound present, even in a fully charged cell. When discharge begins, there is an Ag_2O phase present. As discharge exhausts the AgO the only active phase present is Ag_2O . This condition is such that it encourages a continued drop in voltage until Ag appears and polarizes the Ag_2O with a very conductive substance, thus causing the cell to function at a higher voltage. The rise is never more than 0.01 volt, but the cell remains high for about two hours. Curves illustrating these phenomena are shown in figure 2.

One of these cells, after being charged 0.5 hr at a 300 ma rate into the $\text{Ag}_2\text{O-AgO}$ region showed the next morning no sign of AgO . However, the other cell similarly charged, but for 0.7 hr showed AgO during discharge for nearly two minutes.

When the cells were charged at 150 to 300 ma for an input of 2.3 to 3.2 amp hrs, the succeeding discharge efficiencies approached 100%, but in one instance both cells charged at 40 ma for a 4.8 amp hr input seemed to give an amp hr efficiency of 125%. Judging from the voltages during charge and discharge it would appear that considerable AgO was formed during charge and that on discharge the oxide was in the lower state. Presumably AgO reacted with Ag to give Ag_2O and the latter caused the apparent 125% efficiency.

The effect of higher-rate charge

Cells 1822 and 1823 were used to determine the effect of charging at a higher rate than the customary 300 ma. After the usual forming cycles were completed one test cycle was made to see if the cells were normal. No. 1823 was somewhat defective, but was continued in test. Following 1.00 amp rate charges to full capacity the discharges for three cycles were a little shorter duration to 0.90V, but the first 1.5 hrs of discharge were at voltages of 1.36 to 1.33V, though after over night stand on open circuit. This voltage range is about 0.05V higher than when the charge is made at 300 ma. See figure 3.

The effect of electrolyte level

Cells 1881 and 1858 were used to study the effect of the height of electrolyte level on the capacity of the silver-cadmium cells. Following the forming period the first test cycle showed No. 1858 to be subnormal. During the second discharge of that cell the level of the excess electrolyte lowered to zero before the cell was down to 1.05V. When that cell was charged, the electrolyte depth rose to 1.6 cm. Since No. 1881 was deeper in electrolyte, 2 ml were added to No. 1858. The immediate effect was that No. 1858 became normal. More electrolyte (4.0 and 3.5 ml) was added to the two cells in the charged state bringing the electrolyte depth to 4.2 cm and 4.3 cm. (The height of the plates in the cell is 4.5 cm.) The performance of the cells was slightly improved, but the 15 to 30 minute improvement may have been due to aging the cells. Next, 2.5 and 2.8 ml electrolyte were removed leaving the discharged cells with a 1.1 cm depth. After charge the depth was 2.6 cm. The performance was still good during discharge, but slightly inferior. After two more removals of electrolyte none was visible 1.7 hrs after discharge was begun. A final removal when the cells were fully charged left not over 0.3 ml excess electrolyte. On overnight stand even the 0.3 ml was absorbed by the separators and electrodes, leaving no electrolyte visible. The average of two cycles of discharge of these very much "starved electrolyte" cells was about 6.6 hours. It is apparent then that, after cells are formed and the active parts well saturated with electrolyte, the surplus can be removed leaving no adverse condition, unless extended cycling removes electrolyte or water by some side reaction in the cell. Table 1 relates electrolyte depth to hours capacity to 0.90V.

The effect of high temperature

Cells 1883 and 1884 were cycled at 25°, 40° and 50°C. Table 2 gives the capacities to 0.90V and figure 4 shows typical curves. In the Ag₂O to Ag₂O region the CCV at 50° is about 0.08V higher than that at 25°, with that at 40° intermediate between the two. In the Ag₂O to Ag region there are the same relative positions, but the difference between the extremes is only 0.02V. To establish any superiority as far as amp hr capacity is concerned more data would have to be obtained.

Effect of concentration of electrolyte

It is hard to imagine that 44% KOH (11.64 molar) is the optimum concentration for the best performance for the particular application at hand, unless the very same cycling procedure used in the application was used in choosing the KOH concentration. According to information given by Paul Howard in May 1965 this was not done, but the concentration was specified in the Yardney patent for Ag-Cd cells. At the present time one pair of cells has been activated with a 49.3% KOH (13.24 molar) and the other pair with 33% KOH (7.9 molar) solution. The cells are in the forming period and nothing can be said as to what results to expect. Since the conductance is best in 7 molar KOH, one might expect this to be best for Ag-Cd cells. The conductance of 7 molar is twice that of the electrolyte supplied by Yardney.

A study of the gassing of a Ag-Cd cell

After cell No. 1065 was charged at 25° and let stand two months it was attached to a modified gasometer (see figure 5) in a 50° cabinet. On an 18 hour stand it took up 0.92 ml oxygen. On a 5.7 hr discharge to 0.76V it consumed 2.88 ml more. Even during 13.5 hrs charge at 300 ma (to 1.47V) it required 2.47 ml. While the voltage was rising from 1.47 to 1.49V evolution began. (0.57 ml evolved.) While the voltage was rising to 1.65 in 1.7 hrs 27.1 ml were given off. Twelve minutes later there were 9.3 ml additional. When the charge was stopped at a moment when pressure was relieved by a withdrawal of mercury, the evolution ceased within 5 seconds. If the cell was on open circuit and the gas pressure equilibrated, 10 seconds after beginning a 300 ma charge the evolution of gas was in evidence. Hence, it appears that at 50°C, at least, the changes from gassing to non gassing and vice versa on the plates are promptly reflected in the space above the plates. When the electrode plates were submerged in electrolyte, oxygen consumption was much slower during open circuit stand. In a complete cycle involving purposeful over-charge and a long stand period 61 ml of gas were evolved and 58 ml were consumed. (A study of gas evolution and consumption in normal cell cycling has not been made.) At some charge rate near 8 ma on a fully charged cell there is an equilibrium between gas evolved and gas consumed, the cell having a voltage of 1.52V.

It has been assumed that a lowering of gas pressure means consumption of oxygen and an increase means evolution. Changes of the structure of the Cd-Cd(OH)₂ and Ag-Ag₂O-AgO plates could account for some volume changes. Some preliminary steps were taken to maintain a certain electrolyte level at all times by adding or subtracting electrolyte as required. More work needs to be done at this point.

Composition of positive plate

A fully charged positive plate of cell No. 1855 was analyzed by X-ray diffraction methods. The composition reported was 50% AgO, 25% Cd(OH)₂, 20% Ag₂O and 0% Ag₂CO₃. The reason for such a large amount of Ag₂O is not apparent at the present time. This bears further investigation.

Examination of cell No. 1879

This cell was one that had been modified by Dr. Carson of the General Electric Company to include a third electrode consisting of platinum black and palladium enmeshed in a nickel grid. The cell had failed to hold a charge satisfactorily. One side of the cell case had been removed, a plastic separator inserted and then the additional electrode. To keep the electrode close to the nearby Cd electrode a spongy plastic grid was added and then a new rigid plastic cover-plate was cemented to the sides of the cell case.

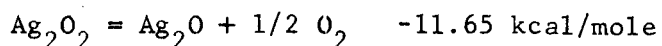
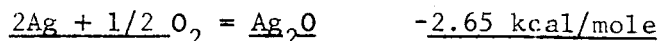
It seemed that, when the cell-case side was sawed, the separator had been cut and the Cd electrode exposed to the electrolyte and possibly "treed" to the third electrode. Inspection of the nearest Cd electrode showed that the face toward the added third electrode was black, especially at the bottom and near the damaged separator, while the other side and the other Cd electrodes were gray. Spectrographic analysis showed the questionable Cd plate to contain Pd 0.1 to 1%, Ni 0.1 to 1%, Pt 0.01 to 0.1% and Ag > 10%. See appended table 3.

The inclusion of platinum and/or palladium in a silver-cadmium cell is considered by some authorities to be very risky and most likely unsuccessful.

An examination of two other modified cells is under way. One of these had failed and the other had not. It will be interesting to study them both simultaneously.

Consideration of Equilibrium of the AgO-Ag₂O, O₂ system.

It was hoped that the type of study made on equilibria of MnO₂, [1] Mn₂O₃ [2] oxides of lead [3] and Ag₂O [4] might be extended to AgO. The values for the ΔF_f° 298 given by Glassner [5] were used to provide a rough calculation of the equilibrium oxygen pressure.



$$\frac{-\Delta F}{T} = 4.5756 \times 1/2 \log p\text{O}_2$$

$$\log p\text{O}_2 = 17.1 \text{ at } 298^\circ\text{K}$$

So the equilibrium pressure would be about 10^{17} atmospheres!
Consequently the plan to measure equilibrium pressures was abandoned.

Dissociation of AgO in KOH solution.

After approval was granted January 12 by Thomas Hennigan a program was undertaken to determine the rates of reaction of AgO in various concentrations of KOH solution. The ultimate study in this investigation should be that of the AgO which is produced during charge of a Ag-Cd cell. The use of commercially produced AgO is simpler, the material easier to get and the results may be of a more lasting value. Since there are practically no data in the literature, the results should be published.

The study was begun by putting some dry Merck AgO in a thermal balance and slowly raising the temperature stepwise. At 50°C the weight increased with time in a rather uniform manner. At 73°C there was a steady losing of weight. The increase at 50°C could be attributed only to the withdrawal of CO_2 from the air. On this account it was decided to change immediately to a closed system and measure oxygen evolution instead of weight loss. For the dissociation in KOH solution two lots of AgO have been used, —one from Handy and Harman and the other from the Ames Chemical Works. Both are reported to be 97 to 98% AgO.

The apparatus used was precisely that described in an article on the Gassing of Dry Cells [6]. (See figure 5.) A sample with KOH solution was put in an open bottle in the cell cup. After temperature stabilization and the stopcock atop the mercury reservoir was closed mercury was added to or taken from the reservoir until the tungsten rod made electrical contact with the mercury in the manometer. Then the air pressure reference tube was closed and the barometric pressure read.

When a suitable amount of O_2 was evolved, as judged by the movement of mercury in the manometer, mercury was removed from the reservoir until electrical contact was re-established. The weight of the mercury removed allowed the calculation of O_2 evolved and from this and the time elapsed the rate could be obtained.

Initially the Handy and Harman AgO was used separately with 44% KOH (11.64 molar, supplied by Yardney), water and no liquid. These were followed by diluted Yardney electrolytes, —a 1 to 2 (3.97 molar) and a

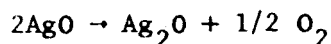
2 to 1 (7.90 molar) mixture with water. Also a much diluted solution was used in which there were 10 parts water to 1 part 44% KOH. This solution was 1.058 molar. So far dissociation has been studied only at 50°C. Preparations have been made to go to higher temperatures, the reasons being simply to gather the data and then to test a statement made in a Naval Ordnance Laboratory report (NOLTR 62-187) that Ag₂O decomposes "some" at 71°C. This is inconceivable from a thermodynamic standpoint. The dissociation of AgO has been found to be slow even at 50°C, but even so there should be a limited study made at 25°C and maybe 0°C.

Figure 6 shows the relative rates in the early stages for the components described above. When the supply of Handy and Harman AgO became exhausted, a change was made to the Ames product and a curve for it is included in figure 6. Thus the two products are quite alike but not identical. It should be observed that the weaker the KOH, the slower the dissociation. An anomalous fact is that water-wet AgO is slower than dry AgO.

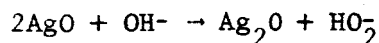
The AgO in 11.64 molar KOH has been in the process of dissociating for 2300 hours and, based on the weight of AgO sample, it has another month to get near completion. It is remarkable how constant the rate holds for more than half the dissociation after the first 100 hrs of stabilization. The volume is nearly linearly proportional to the time. See figure 7.

With 3.97 and 1.058 molar and with water, the plot of volume against time was parabolic after temperature stabilization. This suggested that the volume was proportional to the square root of time. Figure 8 shows how near this is true. The 7.90 molar gave no straight line either way. All this means that the evolution of oxygen by AgO takes place in two ways or more. Rates of dissociation determined in the 100 hr to 300 hr region have been intercompared. The rates were found to be no function of the %KOH, molarity or activity, but the log of the rate was directly proportional to the molality of the KOH. (See figure 9.)

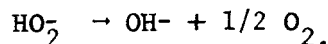
Since it has been shown above that the rate of dissociation of AgO increases greatly as the KOH concentration goes up, the equation for the reaction as it actually takes place must involve OH⁻ ions. The dissociation is usually written



This might be the mechanism when only water is present or when the sample is dry. For the concentrated solutions it is proposed that the reaction be written



followed by



Work in progress or contemplated

It is not yet known if 44% KOH is the best concentration for Ag-Cd cells for the NASA application. If so, why is it? Conductance points away from this concentration. Factors such as activity, viscosity, solution effect on the electrodes, etc. need to be considered. The solubilities of Cd (OH)₂, AgO and Ag₂O in high concentrations of KOH are not available. It is proposed to obtain these. It may be cited in this connection that oxides of manganese are very low in solubilities. Kozawa and Yeager [7] refer to Scholder and Kolb [8] in stating that the solubilities are very small (about 4×10^{-5} M) in neutral or the usual alkaline solutions but "solubility increases rapidly with increasing KOH or NaOH concentration"

The rate of dissociation of AgO in KOH solutions at 50°C is nearly completed. It is planned to get data at a higher temperature because the reaction should be faster and because NOL states that at 71°C some free silver is produced.

AgO should be removed from a fully charged cell and its decomposition rate compared with other rates given in this report.

To get some idea as to the efficiency of the cells on discharge the weights of active components should be determined. The manufacturer declines to provide this information.

The amount of gas produced and consumed during normal cycling has not been determined at any temperature.

The reversal of a cell during discharge of a battery of cells may occur. This can be simulated by charging a cell in reverse at a low rate. The production of hydrogen in this process and the possible disposal of the hydrogen need to be studied.

The study of a second cell-failure of General Electric modified cells is in process. It appears that a silver electrode is the cause of failure. During a 40 hour stand after charge the AgO seemed to have been lost. The evidence is that there was only the Ag₂O-Ag voltage level during discharge. The cell must be opened and examined.

It might be profitable to sever one of the cadmium plates and use it as a reference electrode in a cell. By this means both the silver and the working cadmium electrodes could be studied during charge and discharge.

So very little work has been done to produce a constant voltage cell that it has not been included in this report.

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- [2] Otto, ibid. 111, 88 (1964).
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Earl M. Otto

Earl M. Otto
Electrochemistry Section
Electricity Division, IBS

TABLE 1--THE EFFECT OF ELECTROLYTE LEVEL

(Cells Nos. 1881 and 1858)

CYCLE NO.	ELECTROLYTE DEPTH BEFORE DISCHARGE		1 AMP DISCHARGE TO 0.90V		ELECTROLYTE DEPTH AFTER DISCHARGE	
	1881	1858	1881	1858	1881	1858
	CM	CM	HRS	HRS	CM	CM
1			6.7	4.6		
2	1.7	1.4	6.9	5.1	TRACE	NONE
Added 2.2 ml electrolyte to No. 1858						
3	1.8	3.1	7.0	6.8	0.8	1.9
4	1.8	2.8	6.9	6.5	0.7	1.7
Removed 0.5 ml electrolyte from No. 1858						
5	1.8	2.1	6.8	6.2	0.8	1.0
Electrolyte added: 4.0 ml to 1881 and 3.5 ml to 1858						
6	4.2	4.3	6.6	5.8		
7	4.2	4.5	7.1	6.7	3.6	4.0
8	4.0	4.7	7.0	6.6		
9	4.3	4.4	7.0	6.7		
10			6.8	6.4	3.5	4.2
Electrolyte removed: 2.5 ml from 1881 and 2.8 from 1858						
					1.1	1.1
11	2.4	2.3	7.3?	6.9	1.6	1.6
12	2.6	2.5	6.7?	6.4		
	2.6	2.5	Electrolyte removed: 1.4 ml from each			
13	0.8	0.7	6.7	6.2	0	0
14	1.1	1.2	6.8	6.4	0	0
15	1.2	1.2	6.8	6.6	0	0
Electrolyte removed: 0.7 ml from 1881 and 0.9 ml from 1858						
16	TRACE	TRACE	6.6	6.2	0	0
17	"	"	6.5	6.6	0	0
18	"	"	6.8	6.6	0	0
19	"	"	6.8	6.5	0	0

TABLE 2--THE EFFECT OF HIGH TEMPERATURE

(Cells Nos. 1883 and 1884)

CYCLE NO.	TEMPERATURE		STAND CHARGED HOURS	1 AMP DISCHARGE To 0.90V	
	CHARGE °C	DISCHARGE °C		1883 HOURS	1884 HOURS
1	25	25	24	7.2	6.5
2	25	25	64	7.1	6.4
3	25	40	25	7.2	6.7
4	40	40	19	7.7	7.7
5	40	40	20	7.4	7.6
6	40	25	18	6.9	7.2
7	25	50	16	7.0	7.2
8	50	50	0.2	7.8	7.8
9	50	50	2.3	7.6	7.6
10	50	25	19	7.2	6.8

TABLE 3

TABLE OF RESULTS

Element	Sample					Element	Sample				
	1	2	3	4	5		1	2	3	4	5
Ag	>10		>10			Na	-?		.01-.1		
Al	.001-.01		.001-.01			Nb	-		-		
As	-		-			Nd					
Au	-		-			Ni	.01-.1		.1-1.0		
B	-		-			Os	-		-		
Ba	-		-			P	-		-		
Be	-		-			Pb	.001-.01		.001-.01		
Bi	-		-			Pd	.1-1.0		.1-1.0		
C						Pr					
Ca	.001-.01		.001-.01			Pt	.01-.1		.01-.1		
Cd	>10		>10			Ra					
Ce						Rb					
Co	-		-			Re					
Cr	<.001		<.001			Rh	-		-		
Cs						Ru	-		-		
Cu	.1-1.0		.01-.1			Sb	-		-		
Dy						Sc	-		-		
Er						Si	.01-.1		.01-.1		
Eu						Sm					
Fe	.01-.1		.001-.01			Sn	.001-.01		.01-.1		
Ga	-		-?			Sr	-		-		
Gd						Ta	-		-		
Ge	-		-			Tb					
Hf	-		-			Te	-		-		
Hg	-		-			Th	-		-		
Ho						Ti	-		-		
In	-		-			Tl	-		-		
Ir	-		-			Tm					
K						U	-		-		
La	-		-			V	-		-		
Li						W	-		-		
Lu						Y	-		-		
Mg	.001-.01		.001-.01			Yb					
Mn	<.001		<.001			Zn	-		-		
Mo	-		-			Zr	-		-		
No.	Laboratory No.		Label of Sample								
1	35440a		Metal Grid								
2											
3	35440b		Crumbly plate composition material								
4											
5											

Note: >, greater than; <, less than; -, not detected; values given in percent.

FIG. 1

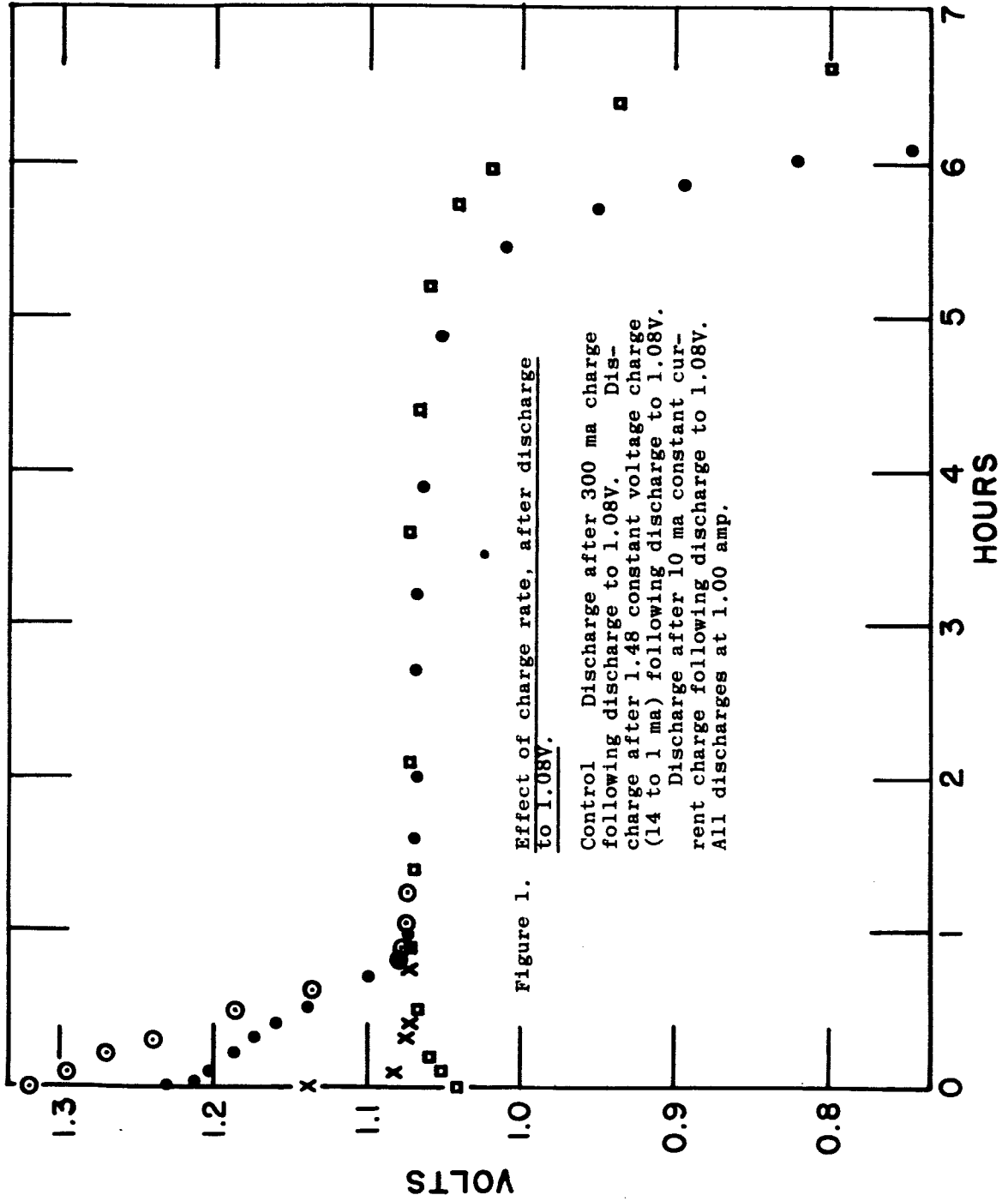


Figure 1. Effect of charge rate, after discharge to 1.08V.

Control Discharge after 300 ma charge following discharge to 1.08V. Discharge after 1.48 constant voltage charge (14 to 1 ma) following discharge to 1.08V. Discharge after 10 ma constant current charge following discharge to 1.08V. All discharges at 1.00 amp.

FIG. 2

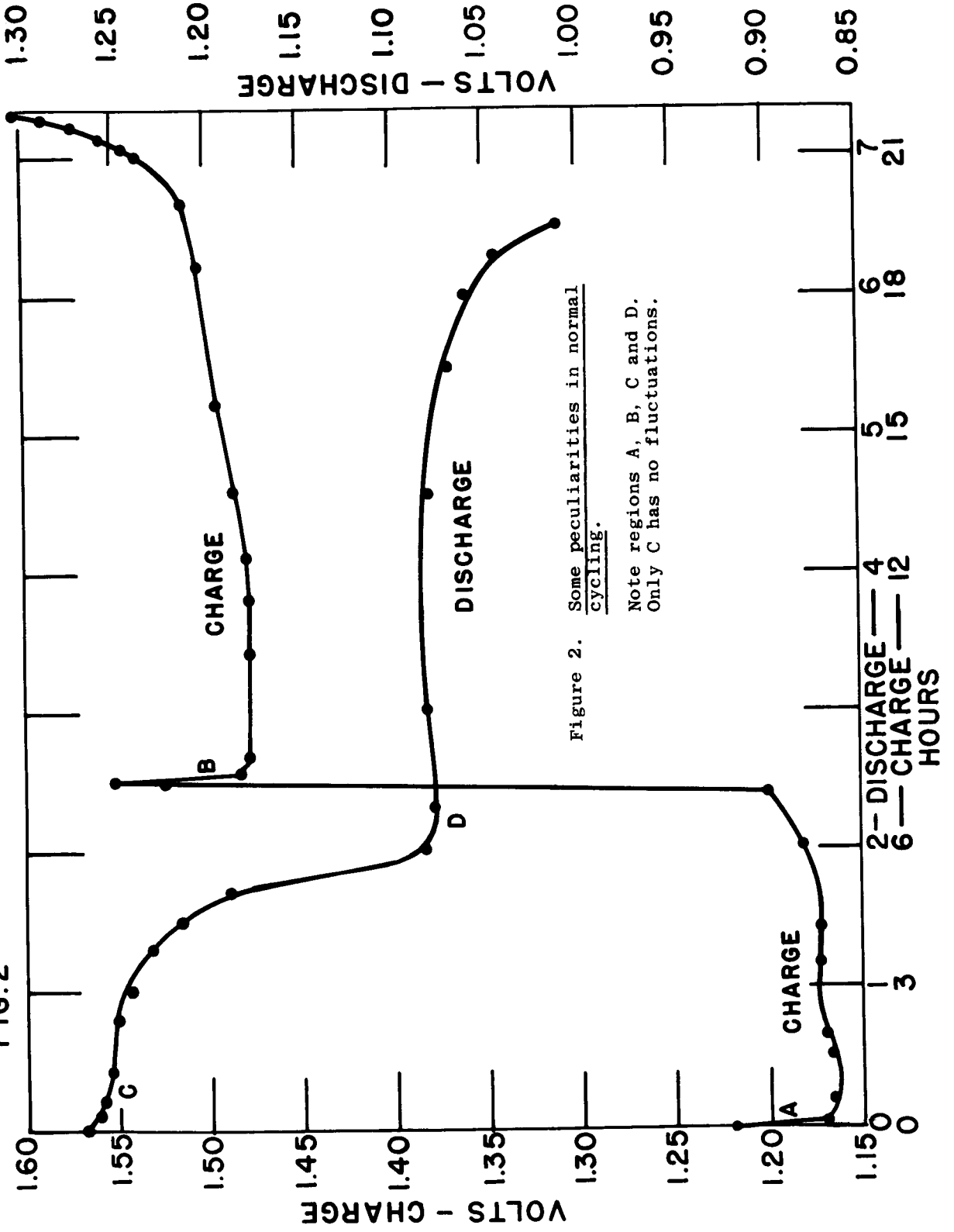


Figure 2. Some peculiarities in normal cycling.

Note regions A, B, C and D. Only C has no fluctuations.

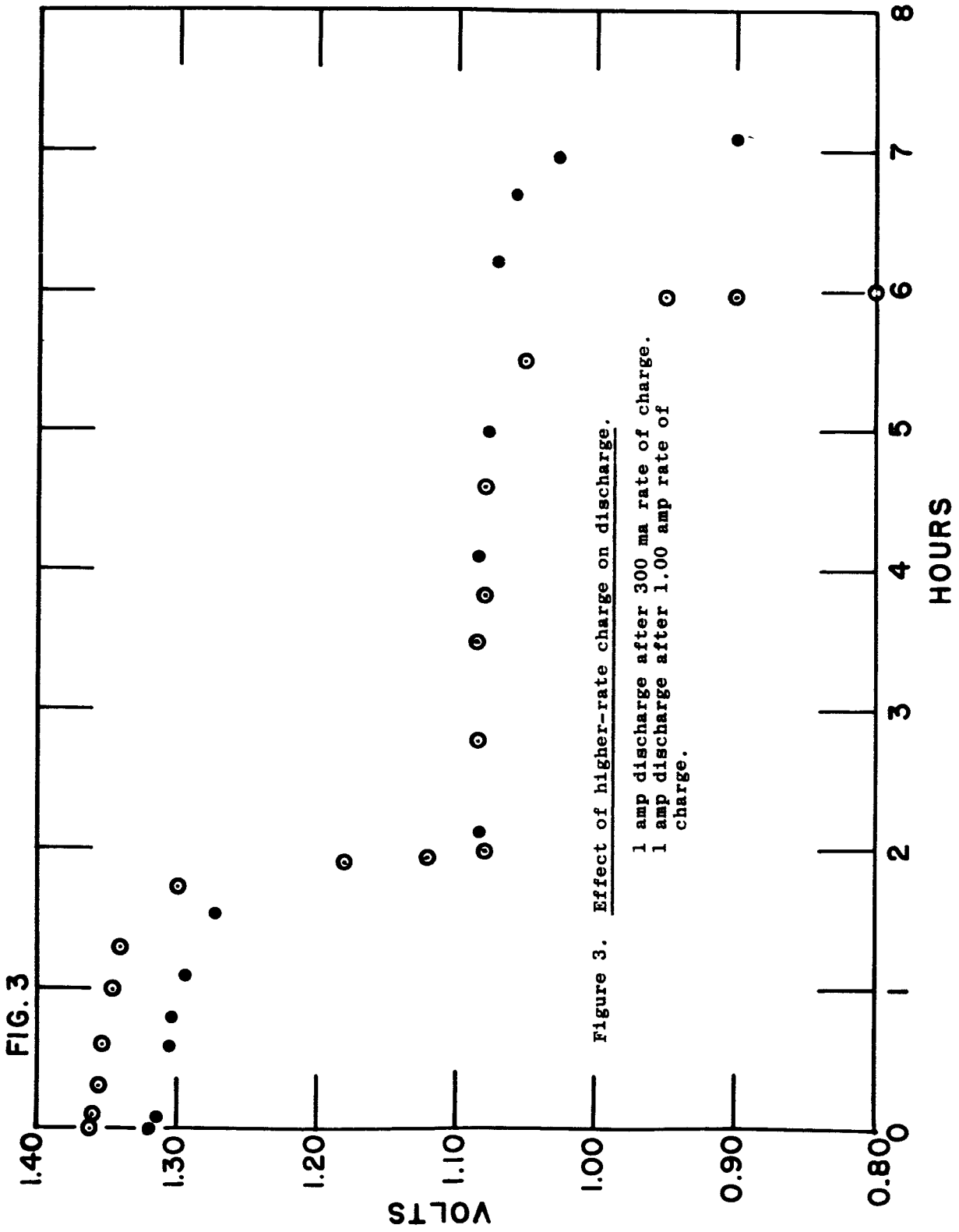
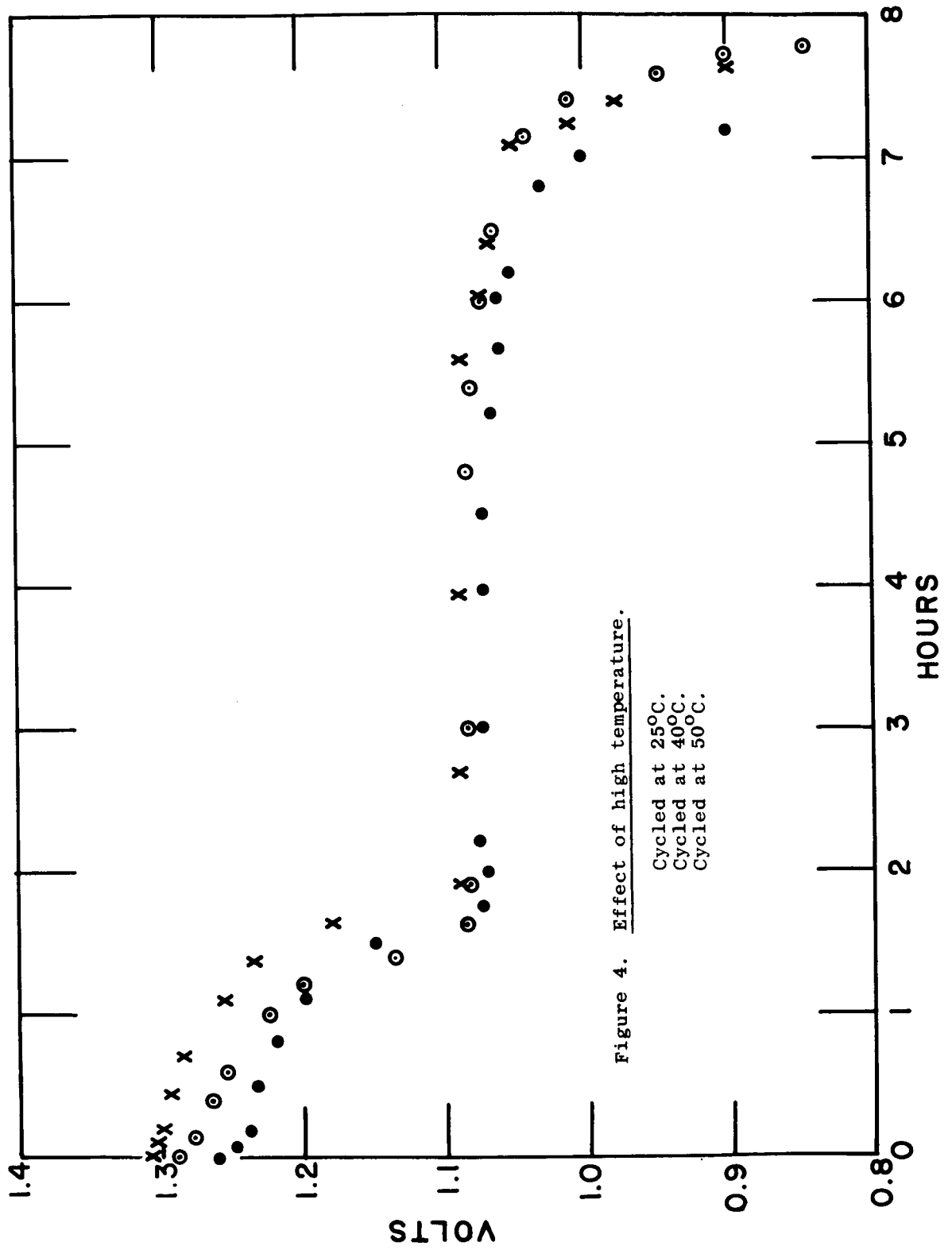


FIG. 4



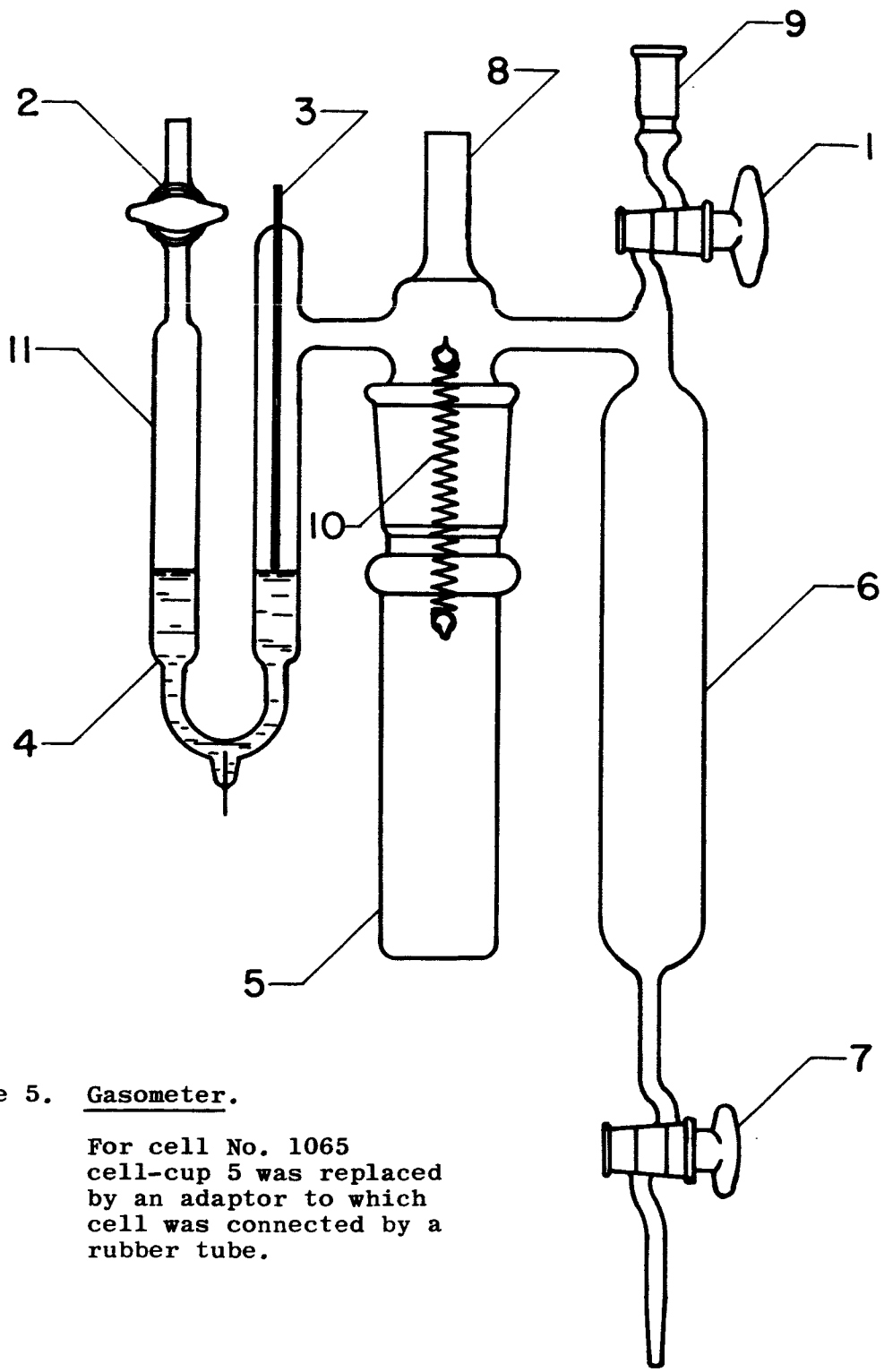


Figure 5. Gasometer.

For cell No. 1065
 cell-cup 5 was replaced
 by an adaptor to which
 cell was connected by a
 rubber tube.

FIG. 6

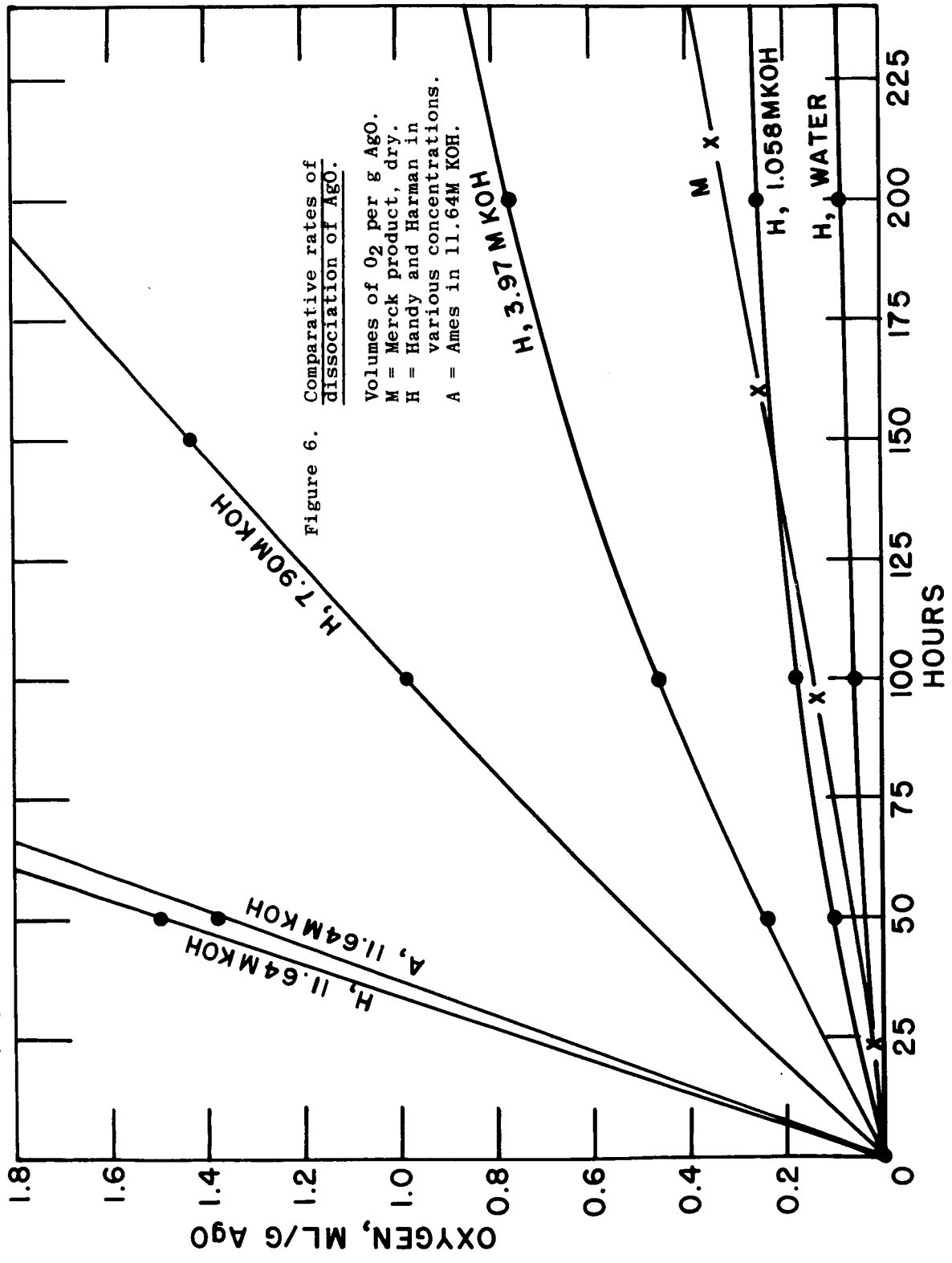


FIG. 7

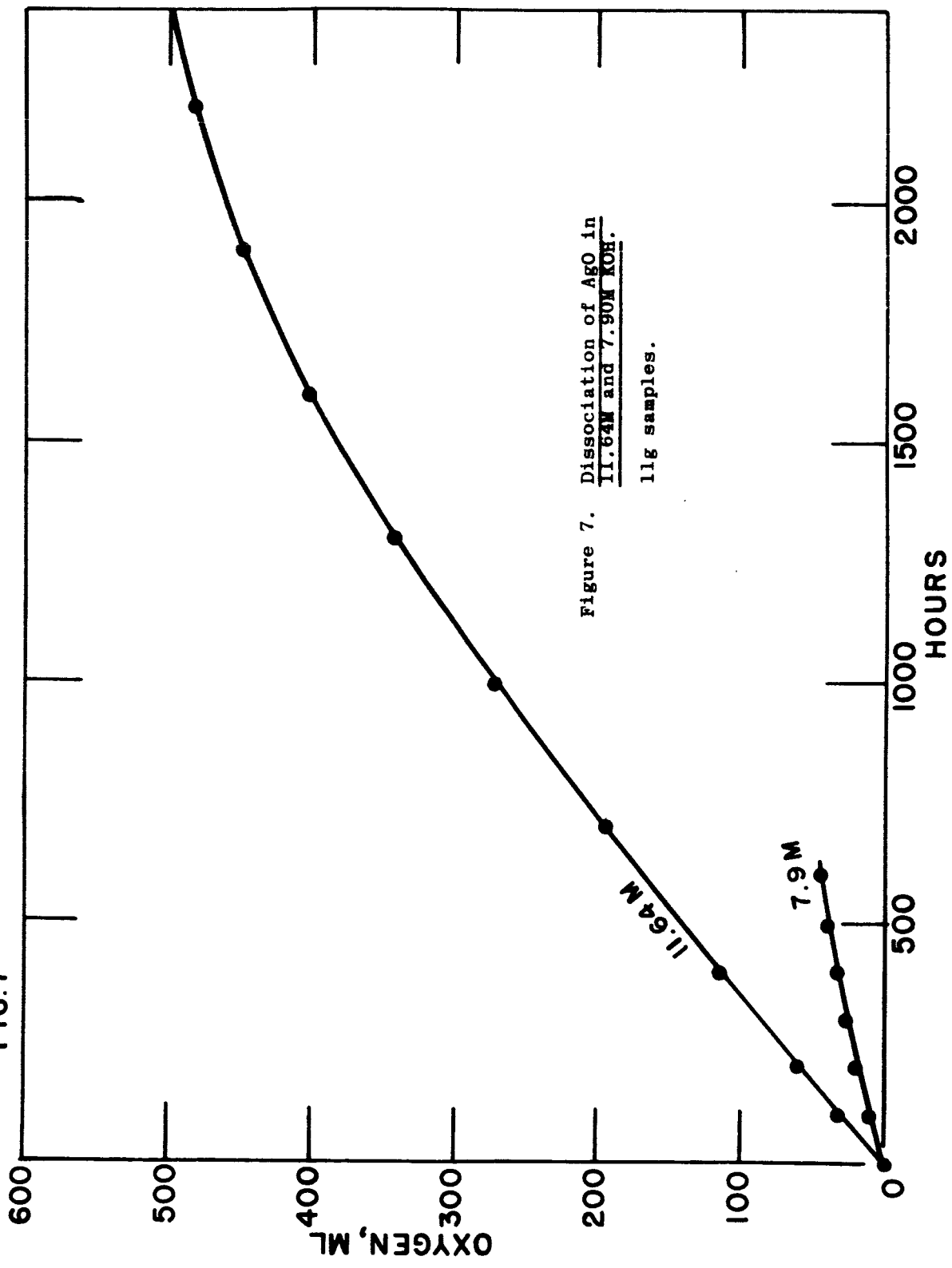


Figure 7. Dissociation of AgO in 11.64M and 7.90M KOH.
11g samples.

FIG. 8

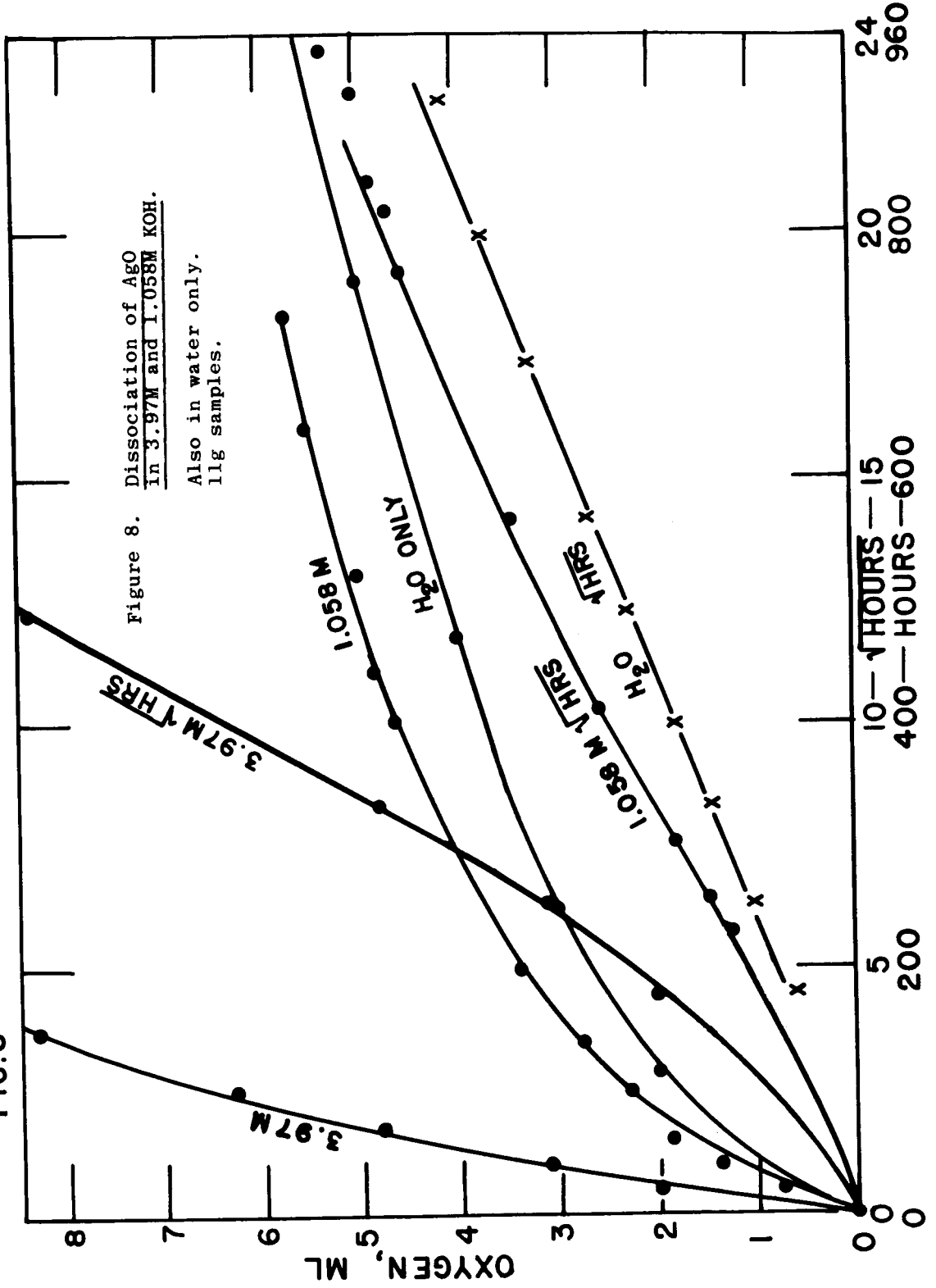


Figure 8. Dissociation of AgO
in 3.97M and 1.058M KOH.
Also in water only.
11g samples.

FIG. 9

