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ALKALINE · MnO₂ BATTERY

**Report No.1
1st Quarterly Report
Dated
August 27, 1962**

U.S. ARMY ELECTRONICS RESEARCH AND DEVELOPMENT LABORATORY

*Fort Monmouth, New Jersey
Contract DA-36-039-SC-89098
Covering the Period
May 1, 1962 · July 30, 1962
Project No. 3A99-09-002-02*

~~NO OTS~~

**UNION CARBIDE CONSUMER PRODUCTS COMPANY
DEVELOPMENT DIVISION
CLEVELAND, OHIO**

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ALKALINE-MnO₂ BATTERY

REPORT NO. 1
1ST QUARTERLY REPORT

Dated
AUGUST 27, 1962

U.S. ARMY ELECTRONICS RESEARCH AND DEVELOPMENT LABORATORY
FORT MONMOUTH, NEW JERSEY

CONTRACT DA-36-039-SC-89098


Covering the Period
MAY 1, 1962 - JULY 30, 1962

PROJECT NO. 3A99-09-002-02


TECHNICAL GUIDELINES FOR PR&C NO. 62-ELP/N-4212
Dated
SEPTEMBER 25, 1961

OBJECT - RESEARCH AND DEVELOPMENT WORK ON ALKALINE-MnO₂ BATTERY

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UNION CARBIDE CONSUMER PRODUCTS COMPANY
CLEVELAND, OHIO

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ABSTRACT CARDS

PURPOSE:

The purpose of this contract is to provide a substantially improved low temperature Alkaline-MnO₂ primary cell over the best existing commercial product, having significantly improved capacity performance at high rates. The cell shall also have design parameters optimized in all areas of performance and shall possess a high level of reliability.

In addition, the following conditions of shelf and service maintenance are to be met.

70% of 70°F. capacity after:

- A. 12 months storage at 113°F.
- B. 3 months storage at 130°F.
- C. 1 month storage at 160°F.

Seal improvement to provide a high degree of resistance to electrolyte leakage. The above will all be adaptable to a wide variety of sizes and shapes.

ABSTRACT:

This First Quarterly Report covers the initial effort to analyze the present factory product E-95 (D size) Alkaline-MnO₂ round cell with special emphasis placed on location of problem areas affecting low temperature, heavy drain service. In addition based on the above analysis empirical data was obtained from variations of electrode and electrolyte formulations as well as separator materials. From the above work, an optimized cell has been obtained for use in factory trials. This cell contains a cathode with a 4/1 ore to carbon ratio, an electrolyte with 40% KOH as dispensed into the cell (approximately 32% equilibrium value in the anolyte), a Viskon-Vinyon separator and a powdered zinc anode. The work to date has indicated a need for a higher surface area zinc. In addition, more work to perfect the seal is needed.

Uniformity of cathode mix formulations will also be characterized with respect to solution volumes, packing, density, etc.

CONFERENCES:

1. May 18, 1962 at Fort Monmouth, New Jersey, to discuss work plans.

Persons Attending:

<u>UCCPC</u>	<u>Signal Corps</u>
M. R. Hatfield	D. Linden
F. L. Granger	A. Daniel
E. F. Sipp, Jr.	J. Murphy
R. B. Klopfenstein	C. Nordell
P. B. Doll	C. Trigg

2. August 21, 1962 at Cleveland, Ohio, to discuss first quarterly progress.

Persons Attending:

<u>UCCPC</u>	<u>Signal Corps</u>
R. B. Klopfenstein	C. Nordell
J. Southworth	
P. B. Doll	
J. Winger	

FACTUAL DATA:

The work done in the first quarter of the contract was divided into two categories, namely:

1. Present E-95 cell analysis and characterization.
2. Substitution of various materials and electrode formulations in "D" size alkaline cells (E-95).

The goal for this quarter was to establish one or two cell models suitable for factory trials. The above categories of work were accomplished simultaneously and much information was relatable in both areas.

The following topics are a detailed account of the work done. Data and curves for these topics are presented in the Figures and Tables at the end of the text.

FACTORY PRODUCT E-95 CELL ANALYSIS.

This portion of the work consisted mainly of cell testing with the aid of reference electrodes and interrupted current techniques (Kordesch Sine Wave Apparatus) (Ref. K. Kordesch, Battery Conference of SCEL, May, 1955.) to determine gross factors affecting I.R. drop and polarization in the cell. For this use, three month old E-95 factory product cells were tested.

The reference electrode used was a zinc wire, polished with fine emery cloth, wrapped with two wraps of Viskon-Vinyon separator material and sealed at both ends to the wire by asphalt. The reference electrode was dipped in H₂O and then 45.2% KOH prior to insertion into the anode cavity of the E-95 cell (Figure 1)

Work reported was done on a sine wave pulser.

An oscilloscope was used to measure IR drops in each electrode. A vacuum tube voltmeter was used to measure open circuit (OCV) and closed circuit potentials (CCV) at each electrode with respect to a zinc reference wire. Since the scope measured only the voltage drop from open circuit to closed circuit associated with the peak pulse current, a VTVM was used to measure absolute voltages (OCV and CCV) associated with an average pulse current. (Figure 2) The average pulse current was read on an ammeter and the pulse current was calculated from the ammeter value by the relation:

$$\text{Pulse Current} = (\pi) \times (\text{Ave. Current}).$$

Resistance values were calculated by the following relations:

$$\text{Resistance} = \frac{\text{IR Loss (Oscilloscope Value)}}{\text{Pulse Current}}$$

$$\text{or Resistance} = \frac{\text{OCV} - \text{CCV (VTVM Values)}}{\text{Ave. Current}}$$

It was obvious that the resistance value was dependent on the accuracy of the average current reading. Since this was the case, resistance values were not calculated; instead percentages of total cell IR drop were calculated for each electrode by the relation:

$$\% \text{ Cathode IR} = \frac{\text{CR (IR of Cathode to Ref. from Oscilloscope)}}{\text{AC (IR of Anode to Cathode from Oscilloscope)}}$$

The accuracy to which each IR loss was determined was within 10% of the estimated true value.

Testing three months old factory product cells at 70°F., 0°F., and -40°F. has shown that the per cent anode IR loss was increasingly greater with decreasing temperatures. The percentages for a given temperature were fairly

constant regardless of drain (Table 1). Tables 2 and 3 show full discharges at 0°F. and -40°F. and the values were consistent with Table 1.

It is not yet known how these relationships hold with cell age or mix formulation. It was apparent, however, that the cathode provided a fertile area for reduction of IR losses and, in fact, later testing of fresh development cells showed the cathode IR losses were as much as 80% of the total cell IR losses. Work will, of course, continue in this area with our best model cells.

Factory product cell testing by conventional techniques across fixed resistors at a variety of loads and temperatures has shown the following characterization. (Tables 4 and 13)

<u>Temp.</u>	<u>Load</u>	<u>% Room Temperature Service</u>		
		<u>1.0 V.</u>	<u>0.9 V.</u>	<u>0.8 V.</u>
0°F.	1.0 ohm	3.5	5.6	8.2
	2.25 "	7.5	13.3	17.0
	10.0 "	27.6	39.6	48.7
-40°F.	2.25 ohm	1.6	2.2	3.2
	4.0 "	1.9*	3.5	5.3
	10.0 "	9.4	11.0	17.8

*Estimated from Table 13 data.

It can be seen that there is a good deal of service available at the lower voltage cutoffs on fairly heavy drains (2.25 and 4.0 ohm continuous). It is also seen that service decreases with decreasing temperature and increases with decreasing drain.

Intermittent service and delayed testing have not as yet been done. Gains in service through intermittent discharge are no doubt possible. Simulated BA - "A" section drains will be part of the future test schedule also.

CATHODE VARIATIONS.

Initial screening was done by varying cathode wall thicknesses and ore to carbon ratios in order to reduce cathode resistance.

Tables 5 and 14 show the effects from proceeding from 11.8/1 to 4/1 ore to carbon ratios. Definite improvements exist in going to 4/1 ore to carbon formulations on both 2.25 and 4.0 ohm tests. In addition, use of Air Spun Graphite (ASG) as sole conductor showed quite another improvement; however, the result was not entirely reproducible. (Figures 3 and 4)

Additional cell making and testing (Tables 6 and 14) confirmed the advantage of 4/1 ore to carbon ratio mixes over control.

Electrode thickness affected each formulation differently. 0.920 ID cathodes (approximately 0.160 inch thick electrodes) were best for 4/1 mixes. (Table 14) (Figure 5)

The most outstanding problem with the cell was uniformity of service levels from lot to lot with a given mix formulation. This was also true with the control product as shown in Table 13. Although substantial improvements have been made to high voltage cuts on heavy drains at -40°F., the level of service is not uniform for these conditions. This is partially a function of the limited (1.0-5.0%) capacity withdrawn to 1.0 volt and the differences in cell to cell IR values. Both of these factors even out as the cell approaches the lower (0.8 volt - 0.6 volt) but still usable cutoffs during heavy drain, low temperature discharge. (Tables 13, 14, 16)

It is not known what effect lighter drains, cell age or intermittency of discharge have on the above situation.

This effect is within the performance range of present dry battery service variation under the same conditions. One factor affecting uniformity

of service was cathode mix moisture content (Fig. 6). Cathode density as well as electrolyte wetting are both affected by mix moisture. (Table 14, Lots 55, 59, 60)

Factors which affect uniformity are as follows:

1. Cathode density - related to conductivity.
2. Cathode wetting - availability of liquid at reaction sites.
3. Raw material uniformity with respect to:
 - a. Moisture content.
 - b. Particle size distribution.
4. Proper mixing of formulations.

Comparative cell analysis with the aid of zinc reference electrodes and the Kordesch Sine Wave Apparatus further confirm the advantage of the 4/1 ore to carbon cathodes over control. (Figures 11A, 11B, 11C, 11D) Figure 11A shows the terminal cell voltages during the 0.250 amp (approximate) average discharge (0.785 amp pulsed, 1/2 time discharged). It can be seen that the 4/1 ore to carbon cathodes display higher discharge curves than control. (Readings taken with VTVM) Figure 11B shows the IR free discharges for these same cells. Here, again, the 4/1 cathodes demonstrate superior discharge curves. Since the IR free curves are a measure of cell polarization, it is apparent that in addition to reducing IR losses the 4/1 cathodes also demonstrate reduced cathode polarization. Figures 11C and 11D compare cell IR losses (Figure 11C) with cathode IR losses (Figure 11D) for these same cells. It is clear that the reduction in cell IR losses with the 4/1 ore to carbon cathodes vs. control is almost entirely due to reduction in cathode IR.

Areas for future cathode work are:

1. Establishing the parameters affecting service uniformity at -40°F. temperature.

2. Determination of best possible conductor or conductor combination.
3. Improvement of mixing operation through use of mullor-type mixing, if possible.
4. Establishing raw material specifications for more uniform electrode fabrication.
5. Determining a more precise measurement of moisture content.
6. Establish the effect of cell age prior to testing.

ELECTROLYTE VARIATIONS.

Work in this area (Tables 8 and 15) was centered around providing the correct electrolyte concentration for -40°F . performance by direct substitution into cells. All electrolyte concentrations were given as the concentration of the liquid being dispensed. From the table, it is apparent that between 37 and 40.5% KOH was the optimum value for balancing conductivity, eutectic and KOH availability.

Since the factory product is now 40.5%, it is felt that no large advantage is gained by going lower.

Other substitutions involved the use of $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ as a sequestering agent to limit the hydroxide reaction and promote the zincate reaction. It was hoped that this would provide a clean zinc surface. No elaborate trial of this material was attempted; therefore, no conclusions can be drawn other than the fact that the amount used gave 50% of control service to 0.9 V. on 4 ohm tests at -40°F . (The above general conclusions apply also to the use of $\text{Li}(\text{OH})$ in the electrolyte.)

ZINC VARIATIONS.

No full scale attempt has been made to correlate zinc surface area (particle size distribution) with cell performance at -40°F . The small amount of work done, however, has shown particle size to be a factor on IR losses to the high voltage cutoffs. It is expected that a higher surface area zinc would reduce anode polarization also.

Tables 9 and 16 show that screening of RM-976 zinc powder to various fractions has yielded as much as 150% of control service at -40°F . with the finer zincs. Figures 12A, 12B, and 12C demonstrate the effects of fine zinc by the use of the Kordes sine wave, interrupted current technique. Cell closed circuit voltages (CCV) vs. cell IR free voltages (OCV) show decreases in cell IR and polarization (Figure 12B) with the use of finer zincs. Figure 12C shows that the cathode IR for the control lot 61 and the fine zinc lot 62 was essentially the same, therefore, improvements in total cell IR and polarization must be related to the finer zinc. Finer zincs possibly may show higher gassing rates on high temperature shelf. Future areas of work will be with various forms of zinc (fibre, foam, etc.), uniformity control of RM-976 zinc or fractions thereof (Fig. 13) and their effect on low temperature service and high temperature shelf.

ANODE COLLECTOR VARIATIONS.

Efforts to improve the particle to particle contact of the anode by restricting the space available for the paste anode expansion have demonstrated that no improvement can be expected in this area of approach. Although 70°F . flash current was improved, the -40°F . service was reduced by reducing the total anode surface exposed to electrolyte and by blocking the paths for electrolyte migration (Table 17).

SEPARATOR VARIATIONS. (Table 18)

Only limited work was done in this area to ascertain the effects of multiple wraps of Viskon-Vinyon on -40°F. service. Based on past data, use of two wraps of Viskon-Vinyon provides adequate shelf stability, as well as acceptable service performance. It is felt that more uniform, better wetting, thinner materials would contribute to lower IR losses. Work will continue in the area of other material substitutions.

Use of cellophane (LSD-195) indicated 67% of control performance on 4 ohm -40°F. service to 1.0 volt, however, it is not known whether this is due to the increased numbers of wraps (4) versus control (2) or the properties of cellophane.

GENERAL.

Typical curves of all the above variations are shown in the Appendix. In addition, curves showing IR losses by sine wave current techniques with 4/1 ore to carbon ratio cathodes are also in the Appendix.

SEAL.

Work is in progress to improve the reliability of the nylon seal so as to meet the requirements of high temperature shelf performance.

CONCLUSIONS:

I. Electrode analysis with reference cells and interrupted sinusoidal current techniques has shown the following on 3 month old factory product cells.

<u>Temp.</u>	<u>Per Cent of Total Cell Internal Resistance</u>	
	<u>% of Cell IR Due to Cathode</u>	<u>% of Cell IR Due to Anode</u>
70°F.	95-97	3-5
0°F.	70-80	20-30
-40°F.	50-70	30-50

Cathode factors play a dominant part in cell IR losses. However, the anode contributes increasingly at the lower temperatures to these losses.

Effects of separator contribution or cell age to IR losses have not been fully determined.

II. Factory Product characterization on 3 month old cells has shown the following. (Table 4)

<u>Load</u>	<u>Per Cent of Room Temp. Service</u>					
	<u>-40°F.</u>			<u>0°F.</u>		
	<u>1.0 V.</u>	<u>0.9 V.</u>	<u>0.8 V.</u>	<u>1.0 V.</u>	<u>0.9 V.</u>	<u>0.8 V.</u>
1.0 ohm	-	-	-	3.5	5.6	8.2
2.25 "	1.6	2.2	3.2	7.5	13.3	17.0
4.0 "	1.9*	3.5	5.3	-	-	-
10.0 "	9.4	11.0	17.8	27.8	39.6	48.7

*Estimated from Table 13 data.

Thus it can be seen that considerable service is available to the lower voltage cutoffs at low temperatures, and that efficiency increases with decreasing drain and increasing temperature.

III. Formulating with varying ore to carbon ratios from 11.8/1 to 4/1 has shown that the more conductive 4/1 mixes (control = 5/1) offer between 50% and

100% improvement to 1.0 volt service at -40°F. (2.25 and 4.0 ohm loads) while maintaining the same extended low voltage service as control.

(Table 5)

- IV. Varying cathode wall thicknesses from 0.175 to 0.145 has shown 0.160 inch thick electrodes to be optimum for 4/1 ore to carbon mixes. This corresponds to a 0.920 O.D. cathode molding ram. (Table 5)
- V. Additional work is needed to determine which conductor or mixture of conductors is best. Use of air spun graphite as sole conductor or mixtures of acetylene black and #2624 graphite have shown essentially equivalent results in 4/1 ore to carbon ratio mixes.
- VI. The electrolyte concentration should be 40% KOH prior to dispensing in the cell. This is approximately a 31-33% equilibrium value when mixed with the other anolyte ingredients. (Table 15)
- VII. Use of finer powdered zincs with better control of particle size distribution shows indications of improving low temperature performance. It has not as yet been determined what effect this will have on high temperature shelf maintenance. (Table 16)
- VIII. Two wraps of Viskon-Vinyon separator material (approx. .008 inch/wrap) are optimum for overall cell performance. (Table 18)
- IX. Although substantial improvements have been made to high voltage cuts on heavy drains at -40°F. the level of service is not uniform for these conditions. This is partially a function of the limited (1.0-5.0%) capacity withdrawn to 1.0 volt and the differences in cell to cell IR values. Both of these factors even out as the cell approaches the lower (0.8 - 0.6 volt) but still usable cutoffs during heavy drain, low temperature discharge. (Tables 13, 14, 16)

It is not known what effect lighter drains, cell age, or intermittency of discharge have on the above situation. This non-uniformity effect is within the performance range of present day dry battery service variation under the same conditions.

FUTURE WORK OUTLINE:

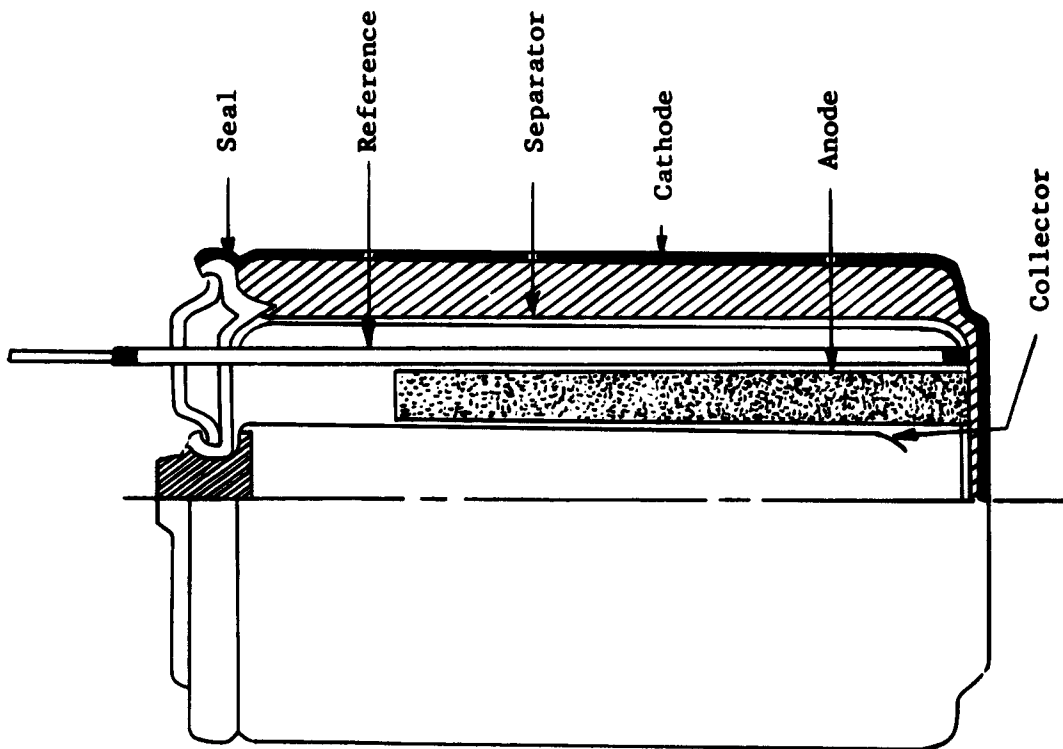
In the next quarter, the following work will be done:

1. Establishment of final cathode formulations.
2. Resolution within the limits of the contract those parameters affecting uniformity of service levels.
3. Finalized seal configuration to meet contract requirements.
4. Investigation of various separator materials.
5. Investigation of various zinc anode forms and/or powdered zinc fractions best suited for low temperatures and high temperatures.
6. Factory trial of best model with subsequent characterization.

PERSONS WORKING DIRECTLY ON SC-89098 CONTRACT:

	<u>Hours Worked from 5-1 to 7-31-62</u>
Project Supervisor - P. B. Doll B.S., M.S. in Mechanical Eng. University of Illinois, 5 yrs. experience on alkaline batteries	56.4
Senior Engineer - J. Winger B.S. in Chemical Engineering Michigan State, 5 yrs. experience on alkaline batteries	364.2
Senior Technician - L. O. Smith 15 years experience on all types of battery development work.	62.0
Senior Lab Assistant - B. J. Gorman 4 years experience on alkaline batteries.	405.9

FIGURE 1
COMPLETE E-95 CELL -
REFERENCE IN POSITION



REFERENCE ELECTRODE

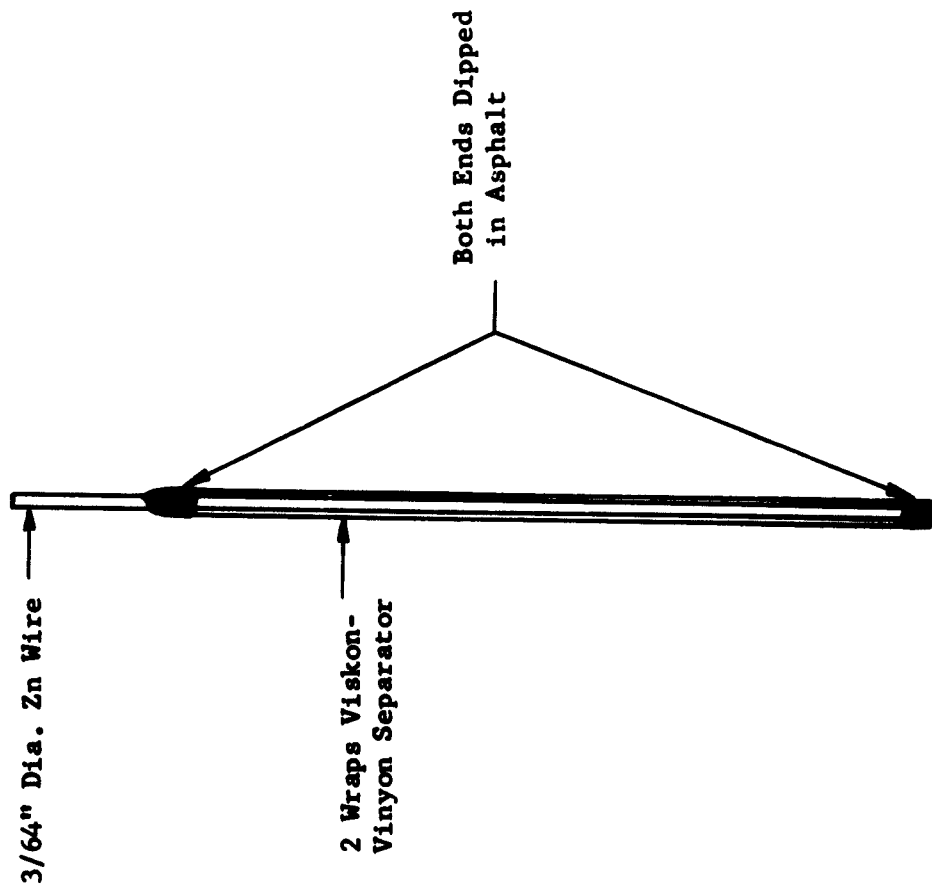


FIGURE 2
SINE WAVE, INTERRUPTED CURRENT, CELL ANALYSIS

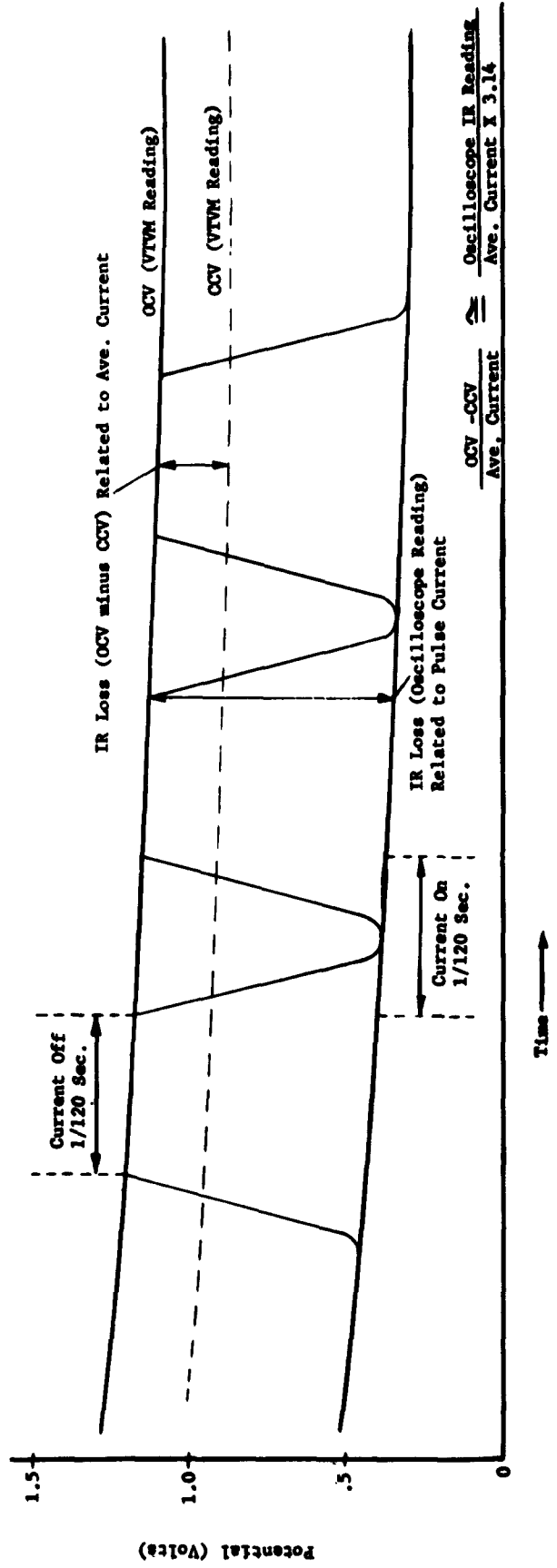
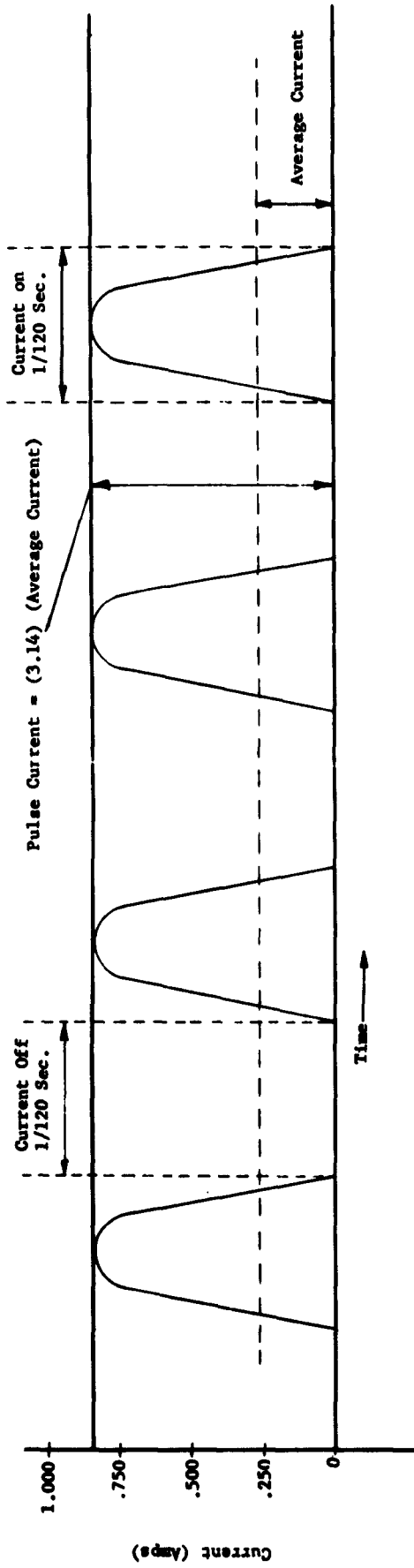


TABLE 1
SINE WAVE EVALUATION
LOT 18 ASHEBORO CELLS (FACTORY PRODUCT)
"D" SIZE CELLS FRESH TEST

Temp.	Ave. Current	(VTVM) AC		(Scope) IR		% CR	% AR
		O.C.V.	C.C.V.	AC	CR		
-40°F.	20 μA	1.56 V.	1.57 V.	0	.0004	0	
	100	1.56	1.57	.0002	.0004	.0001	
	520	1.56	1.56	.0009	.0004	.0004	44
	1020	1.56	1.56	.002	.0009	.001	56
	10.5 mA	1.55	1.55	.018	.0075	.0105	42
	100.0	1.43	1.39	.140	.070	.062	50
	500.0	1.20	0.98	.600	.370	.290	62
	1000.0	0.95	0.45	1.40	.750	.70	54
	22 μA	1.54	1.54	.0001	.0001	.0001	-
	100	1.54	1.54	.0001	.0001	.0001	-
0°F.	500	1.54	1.54	.0003	.0002	.0001	66
	1000	1.54	1.54	.0005	.0003	.0002	60
	9.7 mA	1.54	1.54	.005	.0036	.0016	73
	102.0	-	-	.055	.037	.016	68
	250.0	1.42	1.38	.124	.090	.031	73
	500.0	1.32	1.24	.225	.175	.050	76
	1000.0	1.15	1.01	.400	.310	.086	78
	22 μA	1.57	1.57	0	0	0	-
	100	1.57	1.56	0	0	0	-
	500	1.57	1.56	.0002	.0002	.0001	-
1000			.0004	.0004	.0001	-	
70°F.	9.75 mA	1.57	1.56	.0032	.0032	.0001	100
	102			.032	.027	.0005	85
	250	1.52	1.50	.058	.052	.002	90
	500			.108	.102	.0042	97
	1000	1.41	1.34	.132	.129	.0057	98
							2.7
						1.5	
						3.4	
						3.9	
						4.3	

Pulse Current = (77) (Ave. Current)
AC = Anode to Cathode Reading
AR = Anode to Reference Reading
CR = Cathode to Reference Reading

TABLE 2
SINE WAVE EVALUATION
LOT 18 CELLS
"D" SIZE ASHEBORO CELLS - FRESH TEST

Time	AC (Meter) O.C.V.	Oscilloscope				Amp. Min. Out
		AC IR	CR TR	AR IR	% CR IR	
0 min.	157 volts	0 V.	0 V.	0 V.		0
1	121	.58	.37	.28	63.6	0.5
2	114	.62	.40	.25	64.5	1.0
4	108	.66	.42	.23	63.6	2.0
5	105	.78	.54	.26	69.4	2.5
7	102	.67	.43	.24	64.4	3.5
9	98	.69	.46	.26	66.8	4.5
12	95	.72	.46	.23	64.0	6.0
14	91	.74	.47	.26	63.6	7.0
16	85	.79	.50	.28	63.3	8.0
18	82	.80	.51	.28	63.6	9.0
Drain 0.25 Amps. Ave. - 0.785 Amps Pulse						
1.5	131	.325	.180	.145	55.3	.37
3.0	127	.325	.190	.150	58.4	.75
6.0	122	.330	.195	.150	59.2	1.5
12.0	116	.350	.215	.155	61.5	3.0
18.0	111	.370	.230	.155	62.3	4.5
29.0	105	.415	.255	.185	61.5	7.2
47.0	97	.500	.300	.220	60.0	11.7
57.0	93	.530	.310	.240	58.5	14.2
77.0	85	.580	.300	.270	51.7	19.2
94.0	73	.700	.345	.340	49.3	23.5

AC = Anode to Cathode
AR = Anode to Reference
CR = Cathode to Reference

Refer to Figure 1 and Figure 2 for explanation of cell construction and experimental theory.

TABLE 3
SINE WAVE EVALUATION
LOT 18 CELLS

Temp. 0°F.
Drain 0.5 Amps. Ave.
1.57 Amps. Pulse

Time	O.C.V.		AC (Meter)		I.R.	Oscilloscope				Amp. Mins.	
	O.C.V.	AC (Meter)	C.C.V.	I.R.		AC	CR	AR	IR	%	Out
0.5 min.	1.36 V.	1.28 V.	0.8 V.	.08 V.	.202 V.	.155 V.	.054 V.		77	0.25	
3.0	1.30	1.23	.07	.07	.220	.155	.051		71	1.5	
5.0	1.28	1.20	.08	.08	.220	.165	.048		75	2.5	
11.0	1.22	1.14	.08	.08	.228	.170	.045		75	5.5	
35.0	1.09	1.00	.09	.09	.270	.200	.063		74	17.0	
45.0	1.08	0.97	.11	.11	.300	.220	.073		73	22.4	
68.0	1.00	0.83	.17	.17	.460	.305	.160		66	34.0	
78.0	0.97	0.78	.19	.19	.520	.370	.190		71	39.0	
95.0	0.90	0.69	.21	.21	.620	.430	.240		69		
119.0	0.75	0.52	.23	.23	.710	.450	.265		50		
175.0	0.68	0.49	.19	.19	.540	.380	.190		63		
185.0	0.60	0.40	.20	.20	.600	.460	.195		77		

AC = Anode to Cathode Reading
AR = Anode to Reference Reading
CR = Cathode to Reference Reading

Refer to Figures 1 and 2 for explanation of cell construction and experimental theory.

TABLE 5
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

CATHODE VARIATIONS WITH MnO₂ + CARBON CONDUCTORS

D-1026 Lot	Mix No.	Ore/ Carbon	Ram O.D.	Conductor	Other Variations	Mix Moist.	4 Ohm Cont. at -40°F.			
							% of Control 1.0 V.	% of 70°F. 1.0 V.	% of Control 0.9 V.	
39	Control	5/1	.920	AB + 2624	Cement Dynel	10.8	100	1.9	100	3.5
25	211	11.8/1	.950	"	"	10.0	112	2.3	107	4.0
21	211	11.8/1	.920	"	"	10.0	138	2.6	119	4.4
23	212	7.63/1	.950	"	"	10.0	192	4.0	155	5.9
20	212	7.63/1	.920	"	"	10.0	158	3.0	131	4.9
24	212	7.63/1	.885	"	"	10.0	165	3.0	134	4.9
28	210	5.36/1	.950	"	"	10.7	112	2.0	109	3.6
19	210	5.36/1	.920	"	"	10.7	162	3.0	138	4.9
26	210	5.36/1	.885	"	"	10.7	135	2.4	124	4.2
22	228	4/1	.920	"	"	10.8	138	2.6	122	4.4
27	228	4/1	.885	"	"	10.8	150	2.8	124	4.5
35	218	4/1	.920	ASG	"	12.4	208	4.0	171	6.0

TABLE 6
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

CATHODE VARIATIONS WITH MnO₂ + CARBON CONDUCTORS

D-1026 Lot	Mix No.	Ore/ Carbon	Ram O.D.	Conductor	Other Variations		Mix Moist.	% of Control 1.0 V.	% of 70°F. 1.0 V.	% of Control 0.9 V.	% of 70°F. 0.9 V.
					Cement	Dyne1					
43	Control	5/1	.920	AB + 2624	Cement	Dyne1	10.8	100	2.0	100	4.1
41	228	4/1	.950	" "	-	"	9.8	63	1.3	61	2.5
42	228	4/1	.920	" "	-	"	9.8	140	2.7	126	4.9
40*	228	4/1	.950	" "	-	"	9.8	133	2.9	85	3.8
49	Control	5/1	.920	" "	Cement	Dyne1	10.6	100	0.9	100	2.0
54	228	4/1	.920	" "	-	"	11.0	273	2.3	197	3.7
55	218	4/1	.920	ASG	-	-	11.0	64	0.5	77	1.4
59	218	4/1	.920	ASG	-	-	9.5	200	1.6	150	2.7
60	218	4/1	.920	ASG	-	-	7.0	164	1.4	127	2.4
57	223	9/1	.920	ASG	-	Dyne1	11.2	0	0	0	0
61	Control	5/1	.920	AB + 2624	Cement	Dyne1	9.7	100	0.9	100	1.6
63	218	4/1	.920	ASG	-	-	9.7	208	1.8	166	3.1
65	230	4/1	.920	ASG	Flake KOH		9.8	167	1.5	150	2.3
64**	218	4/1	.920	ASG	-	-	9.7	267	2.4	188	3.6

* 35% KOH electrolyte + 150-200 mesh zinc, .500 O.D. curtain rod collector.
** Anode was 150-Pan screened 1205 zinc.

TABLE 7
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

CATHODE VARIATIONS WITH MnO₂ + CARBON CONDUCTORS

D-1026 Lot	Mix No.	Ore/ Carbon	Ram O.D.	Conductors	Other Variations	Mix Moist.	4 Ohm Cont. at -40°F.			
							% of Control 1.0 V.	% of 70°F. 1.0 V.	% of Control 0.9 V.	% of 70°F. 0.9 V.
66	Control	5/1	.920	AB + 2624	Cement	Dynel 9.1	100	0.5	100	1.2
67	218	4/1	.920	ASG	-	9.2	167	0.8	137	1.6
69	231	4/1	.920	ASG	-	Dynel 9.2	133	0.6	121	1.4
68	228	4/1	.920	AB + 2624	-	Dynel 9.4	267	1.2	179	2.3

TABLE 8
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

ELECTROLYTE VARIATION

4 Ohm Cont. at -40°F.

<u>D-1026</u> <u>Lot</u>	<u>% KOH</u>	<u>Additive</u>	<u>% of Control</u> <u>1.0 V.</u>	<u>% of 70°F.</u> <u>1.0 V.</u>	<u>% of Control</u> <u>0.9 V.</u>	<u>% of 70°F.</u> <u>0.9 V.</u>
10	40.5	Control	100	No Data	100	No Data
17	27.0	No CMC	50	" "	51	" "
16	29.0	" "	77	" "	68	" "
15	31.0	" "	88	" "	75	" "
14	33.0	" "	100	" "	78	" "
39	40.5	Control	100	1.9	100	3.5
29	37.0		104	2.0	102	3.6
30	35.0		104	2.0	98	3.6
31	33.0		115	2.3	97	3.6
49	40.5	Control	100	0.9	100	2.0
50	45.0		55	0.5	57	1.1
51	50.0	Flake KOH	18	0.2	13	0.3
48	40.5	1% LiOH	100	0.8	10	1.9
52	40.5	.4% SiO ₂	50	0.4	52	1.0

TABLE 9
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

ZINC VARIATION

D-1026 Lot	Zinc Type	Screen	4 Ohm Testing at -40°F.		% of 70°F. 0.9 V.	
			% of Control 1.0 V.	% of Control 1.0 V.		
39	MM-976	Control	100	1.9	100	3.5
36	"	150-200	119	2.3	128	4.7
37	"	100-150	119	2.2	124	4.4
38	"	35-100	115	2.2	121	4.3
49	"	Control	100	0.9	100	2.0
53	"	150-Pan	155	1.4	140	2.8
61	"	Control	100	0.9	100	1.9
62	"	150-Pan	100	0.9	100	2.0
63*	"	Control	100	1.8	100	3.1
64*	"	150-Pan	128	2.4	95	3.6

* 4/1 ore to carbon cathodes with ASC (Mix 218).

TABLE 10
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

ANODE COLLECTOR VARIATIONS

<u>D-1026</u> <u>Lot</u>	<u>Collector</u> <u>Type</u>	<u>Collector</u> <u>O.D.</u>	<u>4 Ohm Cont. at -40°F.</u>		
			<u>% of Control</u> <u>1.0 V.</u>	<u>% of 70°F.</u> <u>1.0 V.</u>	<u>% of Control</u> <u>0.9 V.</u>
39	Leg	.396 Control	100	1.9	100
32*	Perforated tube.	.500	69	1.6	78
33*	Perforated tube.	.550	62	1.5	67
					3.5
					3.3
					3.0

* Preamalgamated, perforated brass tubes.

TABLE 11
LOW TEMPERATURE FRESH SERVICE SUMMARY RESULTS

SEPARATION VARIATIONS

<u>D-1026</u> <u>Lot</u>	<u>Separator Type</u>	<u>Wraps</u>	<u>4 Ohm Cont. at -40°F.</u>			
			<u>% of Control</u> <u>1.0 V.</u>	<u>% of 70°F.</u> <u>1.0 V.</u>	<u>% of Control</u> <u>0.9 V.</u>	<u>% of 70°F.</u> <u>0.9 V.</u>
10	Viskon-Vinyon	2	100	No Data	100	No Data
12*	Viskon-Vinyon	1	62	" "	78	" "
11*	Viskon-Vinyon	2	46	" "	58	" "
13*	Viskon-Vinyon	3	50	" "	64	" "
43	Control	2	100	2.0	100	4.07
44	Viskon-Vinyon + ISD-195 Cellophane	4	67	1.4	90	3.2

* No CMC in electrolyte.

TABLE 13
LOW TEMPERATURE FRESH SERVICE RESULTS

CONTROL LOTS

D-1026 <u>Lot</u>	<u>Mfr. Date</u>	<u>Cathode Mix Wt. gms.</u>	<u>% Cath. Mix Moist.</u>	<u>2.25 Ohm Cont. (minutes)</u>			<u>4 Ohm Cont. (minutes)</u>								
				<u>70°F.</u>	<u>0.8</u>	<u>-40°F.</u>	<u>70°F.</u>	<u>0.8</u>	<u>-40°F.</u>						
9	5-23-62 Devel.	46.11	12.8	558	740	910	2	5	14	1254	1510	1890	No data		
10	5-29-62 "	46.51	9.4	462	700	852	3	6	21	No Test	26	59	112		
18	Rec. 6-1-62 Asheboro			492	677	855	8	15	27	"	"	17	42	78	
39	7-6-62 Devel.	50.60	10.8	652	846	1016	6	15	33	1380	1640	2010	26	58	108
43	7-11-62 "	49.70	10.8	660	838	996	5	13	32	1344	1620	1960	27	66	137
49	7-26-62 "	48.0	10.6	660	780	948	2	4	14	1268	1520	1934	11	30	67
61	8-3-62 "	51.81	9.7	618	800	952	2	3	10	1357	1650	2060	12	32	73
66	8-7-62 "	50.38	9.1	626	808	956	0	1	5	1308	1580	1940	6	19	45

TABLE 14
LOW TEMPERATURE FRESH SERVICE RESULTS

CATHODE VARIATIONS WITH Na_2O_2 AND CARBON CONDUCTORS

D-1026 Loc	Mfg. Date	Mix No.	Ore/ Carbon	Ram O.D.	Conductor	Other Variations	% Mix Moist.	2.25 Ohm Cont. (min.)				4 Ohm Cont. (min.)							
								70°F. 1.0	0.9	0.8	0.8	70°F. 1.0	0.9	0.8	0.8				
39	7-6-62	Control	5/1	.920	AB + 2624	Cement Dynel	10.8	652	846	1016	6	15	33	1380	1640	2010	26	58	108
25	"	211	11.8/1	.950	"	-	10.0	550	714	844	1	14	37	1276	1540	1810	29	62	108
21	"	211	11.8/1	.920	"	-	10.0	620	748	794	4	17	35	1404	1578	1674	36	69	107
23	"	212	7.63/1	.950	"	-	10.0	576	736	940	0	11	31	1246	1530	1806	50	90	139
20	"	212	7.63/1	.920	"	-	10.0	564	726	830	0	16	33	1366	1544	1682	41	76	122
24	"	212	7.63/1	.885	"	-	10.0	606	760	802	2	14	33	1432	1590	1798	43	78	118
28	"	210	5.36/1	.950	"	-	10.7	679	860	980	0	7	26	1440	1740	2100	29	63	112
19	"	210	5.36/1	.920	"	-	10.7	676	810	848	7	16	36	1420	1620	1758	42	80	138
26	"	210	5.36/1	.885	"	-	10.7	678	802	826	2	14	35	1476	1720	1828	35	72	125
22	"	228	4/1	.920	"	-	10.8	636	790	850	7	20	41	1380	1610	1850	36	71	117
27	"	228	4/1	.885	"	-	10.8	608	734	764	0	7	27	1380	1590	1688	39	72	114
35	"	218	4/1	.920	ABC	-	12.4	678	828	938	10	23	44	1360	1650	1882	54	99	163

TABLE 14, Cont'd.

CATHODE VARIATIONS WITH MnO₂ AND CARBON CONDUCTORS

D-1026 Lot	Mfg. Date	Mix No.	Ore/ Carbon	Ram O.D.	Conductor	Other Variations	% Mix Moist.	2.25 Ohm Cont. (min.)			4 Ohm Cont. (min.)								
								70°F. 1.0	0.9	0.8	70°F. 1.0	0.9	0.8	-40°F. 1.0	0.9	0.8			
43	7-11-62	Control	5/1	.920	AB + 2624	Cement Dynel	10.8	660	838	996	5	13	32	1334	1620	1960	27	66	137
41	"	228	4/1	.950	"	"	9.8	702	870	1066	1	7	20	1326	1610	2050	17	40	81
42	"	228	4/1	.920	"	"	9.8	698	872	1014	7	17	39	1400	1710	2024	38	83	154
40*	"	228	4/1	.950	"	"	9.8	580	700	808	9	17	25	1242	1466	1880	36	56	70
* 35% KOH electrolyte + 150-200 mesh zinc, .500 O.D. Curtain Rod Collector.																			
49	7-26-62	Control	5/1	.920	AB + 2624	Cement Dynel	10.6	600	780	948	0	4	14	1268	1520	1934	11	30	67
54	"	228	4/1	.920	"	"	11.0	666	836	1060	7	17	28	1312	1578	2100	30	59	109
55	"	218	4/1	.920	ASG	"	11.0	624	826	1292	2	4	14	1370	1630	2060	7	23	57
59	"	218	4/1	.920	"	"	9.5	666	856	1050	0	10	22	1380	1640	2130	22	45	81
60	"	218	4/1	.920	"	"	7.0	620	746	816	0	5	14	1316	1552	1700	18	38	65
57	"	223	9/1	.920	"	"	11.2	638	874	1070	0	0	0	1438	1830	2200	0	0	0
61	8-3-62	Control	5/1	.920	AB + 2624	Cement Dynel	9.7	618	800	952	2	3	10	1357	1650	2060	12	32	73
63	"	218	4/1	.920	ASG	"	9.7	704	872	976	4	12	26	1380	1685	2150	25	53	94
65	"	230	4/1	.920	"	Flake KOH	9.8	640	840	1060	2	6	18	1340	1690	2110	20	48	97
64*	"	218	4/1	.920	"	"	9.7	708	900	1106	5	13	25	1358	1650	2016	32	60	95
* Anode was 150-Pan screened KM-976 zinc.																			
66	8-7-62	Control	5/1	.920	AB + 2624	Cement Dynel	9.1	626	808	956	0	1	5	1308	1580	1940	6	19	45
67	"	218	4/1	.920	ASG	"	9.2	648	844	966	1	3	11	1308	1610	1955	10	26	50
69	"	231	4/1	.920	"	"	9.2	618	810	902	0	1	6	1308	1610	1980	8	23	48
68	"	228	4/1	.920	AB + 2624	"	9.4	528	654	752	2	4	13	1296	1508	1930	16	34	62

TABLE 15
LOW TEMPERATURE FRESH SERVICE RESULTS

ELECTROLYTE VARIATION

D-1026 Lot	Mfg. Date	% KOH	Additive	2.25 ohm Cont. (min.)			4 Ohm Cont. (min.)								
				70°F.			70°F.								
				1.0	0.9	0.8	1.0	0.9	0.8						
10	5-29-62	40.5	Control	462	700	852	3	6	21	No Data	26	59	112		
17	"	27	No CMC	492	638	684	0	2	4	"	13	30	51		
16	"	29	"	510	665	781	1	3	11	"	20	40	63		
15	"	31	"	518	665	805	3	5	18	"	23	44	72		
14	"	33	"	436	561	665	4	9	24	"	26	46	75		
39	7-6-62	40.5	Control	652	846	1016	0	0	15	1380	1640	2010	26	58	108
29	"	37		670	836	972	3	12	31	1380	1620	1980	27	59	113
30	"	35		626	790	916	4	11	28	1354	1590	1876	27	57	96
31	"	33		600	758	870	0	6	16	1318	1570	1870	30	56	89
49	7-26-62	40.5	Control	600	780	948	2	4	14	1268	1520	1934	11	30	67
50	"	45.0		563	738	938	1	3	8	1238	1506	1952	6	17	43
51	"	50.0	Flake KOH	540	756	978	0	1	2	1140	1460	1866	2	4	13
48	"	40.5	1% LiOH	600	776	936	2	3	10	1274	1534	1938	10	29	62
52	"	40.5	.4% SiO ₂	622	808	970	2	6	15	1190	1496	1940	5	15	40

TABLE 16
LOW TEMPERATURE FRESH SERVICE RESULTS

ZINC VARIATION

D-1026 Lot	Mfg. Date	Zinc Type	Screen	2.25 Ohm Continuous (min.)			4 Ohm Continuous (min.)								
				-40°F.			-40°F.								
				1.0	0.9	0.8	1.0	0.9	0.8						
39	7-6-62	RM-976	Control	6	15	33	652	846	1016	26	58	108	1380	1640	2010
36	"	"	150-200	0	10	26	530	763	962	31	74	148	1334	1580	1970
37	"	"	100-150	0	9	24	665	816	968	31	72	152	1386	1650	1950
38	"	"	35-100	0	0	15	666	836	968	30	70	137	1380	1620	1970
49	7-26-62	"	Control	2	4	14	600	780	948	11	30	67	1268	1520	1934
53	"	"	150-Pan	5	10	23	612	792	960	17	42	83	1223	1509	1870
61	8-3-62	"	Control	2	3	10	618	800	952	12	32	73	1357	1650	2060
62	"	"	150-Pan	2	4	12	688	880	1033	12	32	72	1354	1610	1970
63*	8-3-62	"	Control	4	12	26	704	872	976	25	53	94	1380	1685	2150
64*	"	"	150-Pan	5	13	25	708	900	1106	32	60	95	1358	1650	2016

* 4/1 ore to carbon cathodes with ASG (Mix 218).

TABLE 17
LOW TEMPERATURE FRESH SERVICE RESULTS

ANODE COLLECTOR VARIATIONS

<u>Lot</u>	<u>Mfg. Date</u>	<u>Collector Type</u>	<u>Cell O.D.</u>	<u>2.25 Ohm Cont. (min.)</u>		<u>4 Ohm Cont. (min.)</u>	
				<u>70°F.</u>	<u>-40°F.</u>	<u>70°F.</u>	<u>-40°F.</u>
39	7-6-62	Leg	.396 (control)	1.0	0.9	1.0	0.9
				0.8	0.8	0.8	0.8
32*	"	Perforated Tube	.500	652	846	1016	6
				15	33	1380	1640
				2010	26	58	108
33*	"	Perforated Tube	.550	552	714	836	1
				6	15	1136	1382
				1720	18	45	87
				1612	16	39	77

* Prealgalgated, Perforated Brass Tubes.

TABLE 18
LOW TEMPERATURE FRESH SERVICE RESULTS

SEPARATOR VARIATIONS

D-1026 Lot	Mfg. Date	Separator Type	Wraps	2.25 Ohm Continuous			4 Ohm Continuous								
				70°F.			70°F.								
				1.0	0.9	0.8	1.0	0.9	0.8						
10	5-29-62	Control	2	462	700	852	3	6	21	No Data	26	59	112		
12*	"	V-V	1	582	732	819	4	12	28		16	46	98		
11*	"	V-V	2	548	735	877	3	7	20		12	34	80		
13*	"	V-V	3	590	721	818	3	7	22		13	38	85		
43	7-11-62	Control	2	660	838	996	5	13	32	1344	1620	1960	27	66	137
44	"	V-V + LSD-195	4	612	786	950	3	10	26	1318	1580	2030	18	51	112

* No CMC in clear gel.

FIGURE 3

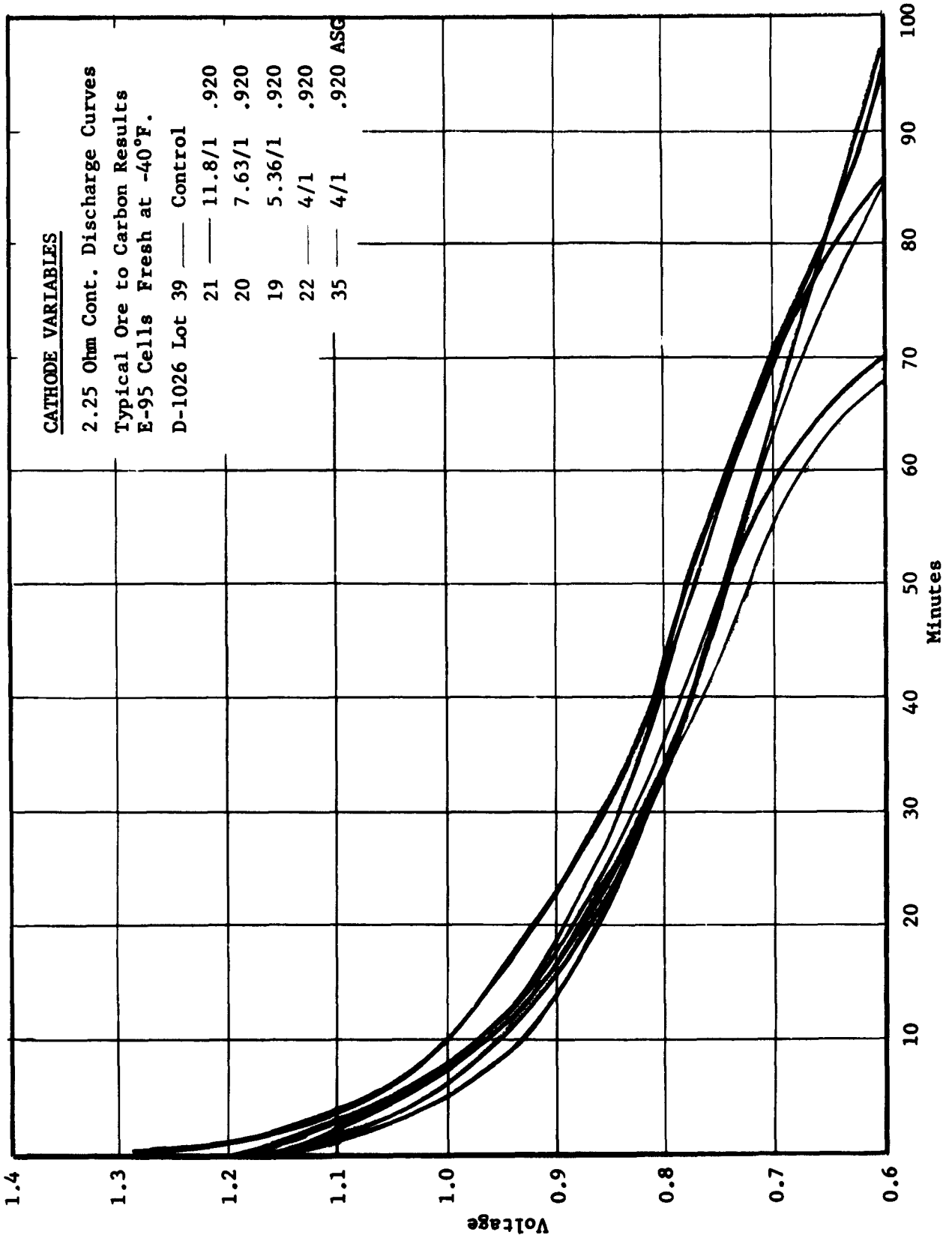


FIGURE 4

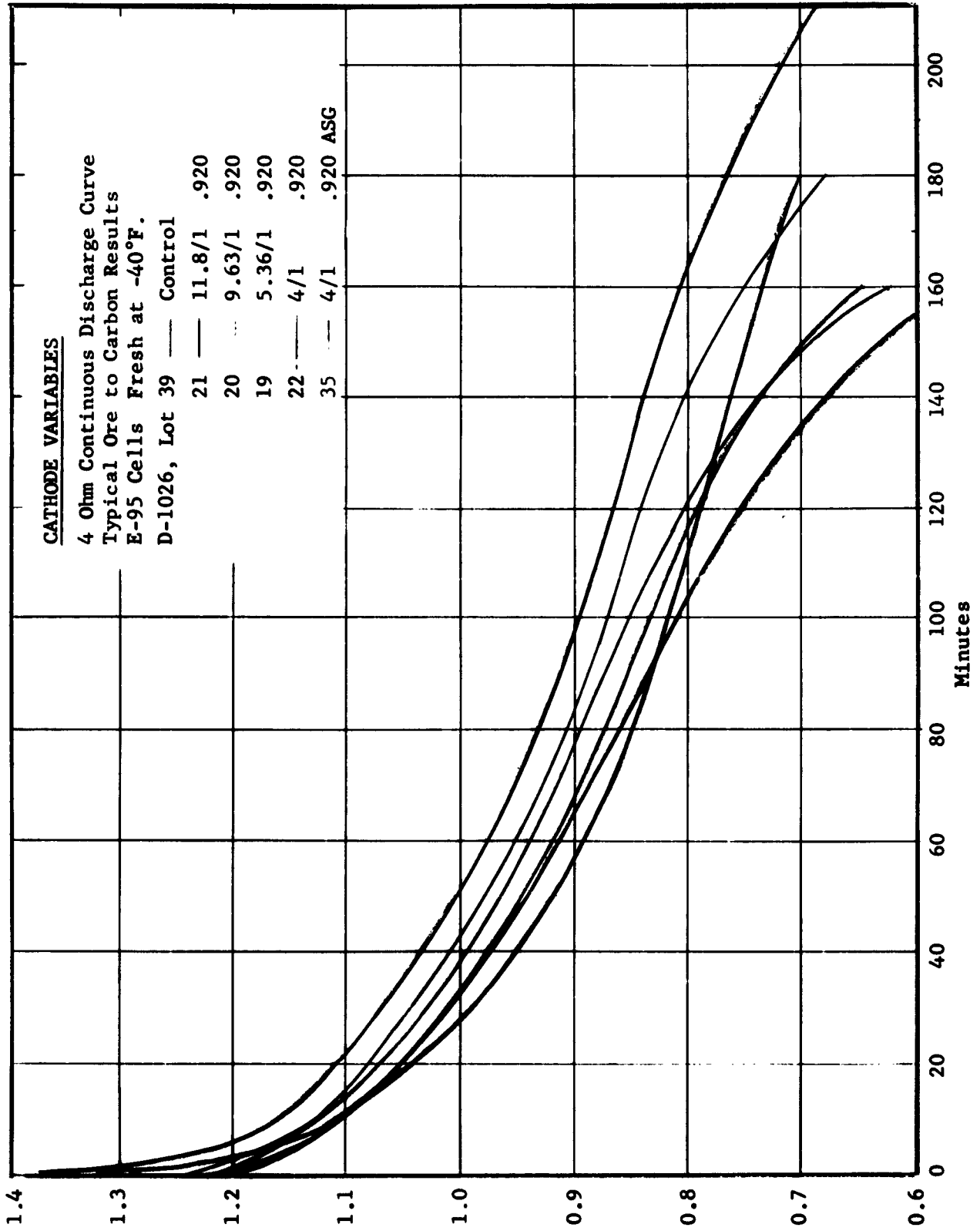


FIGURE 5

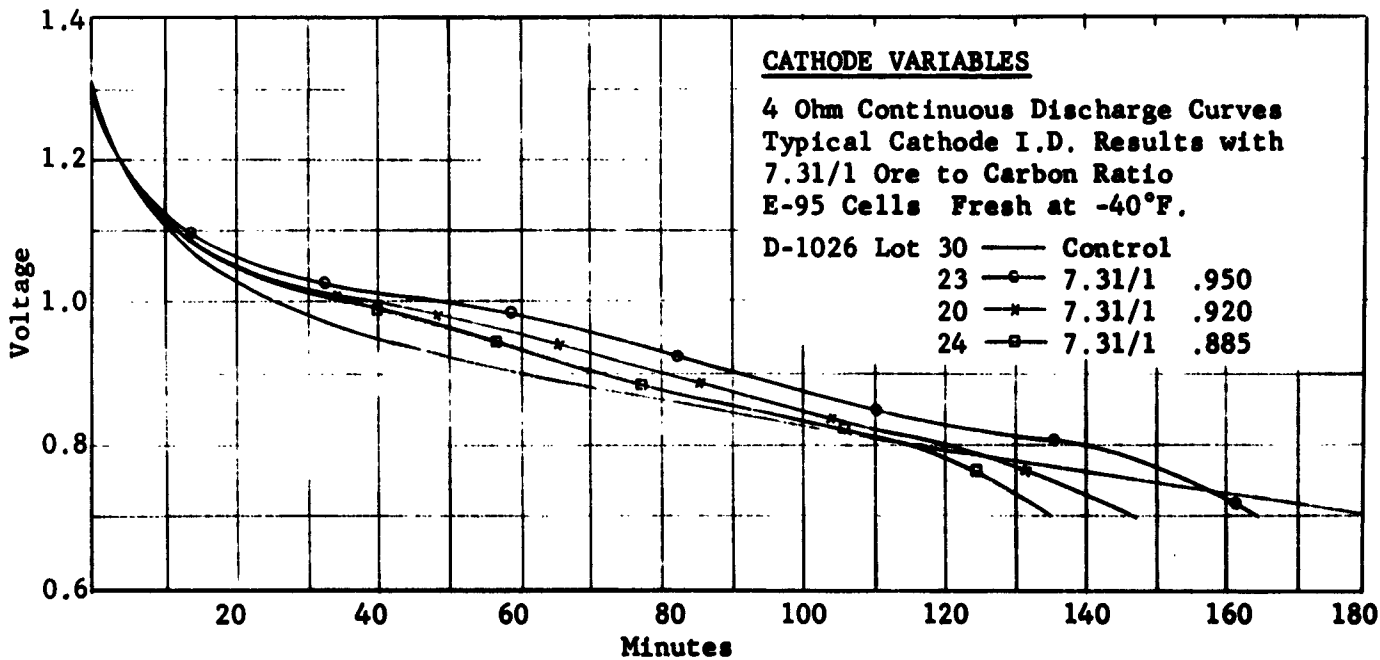
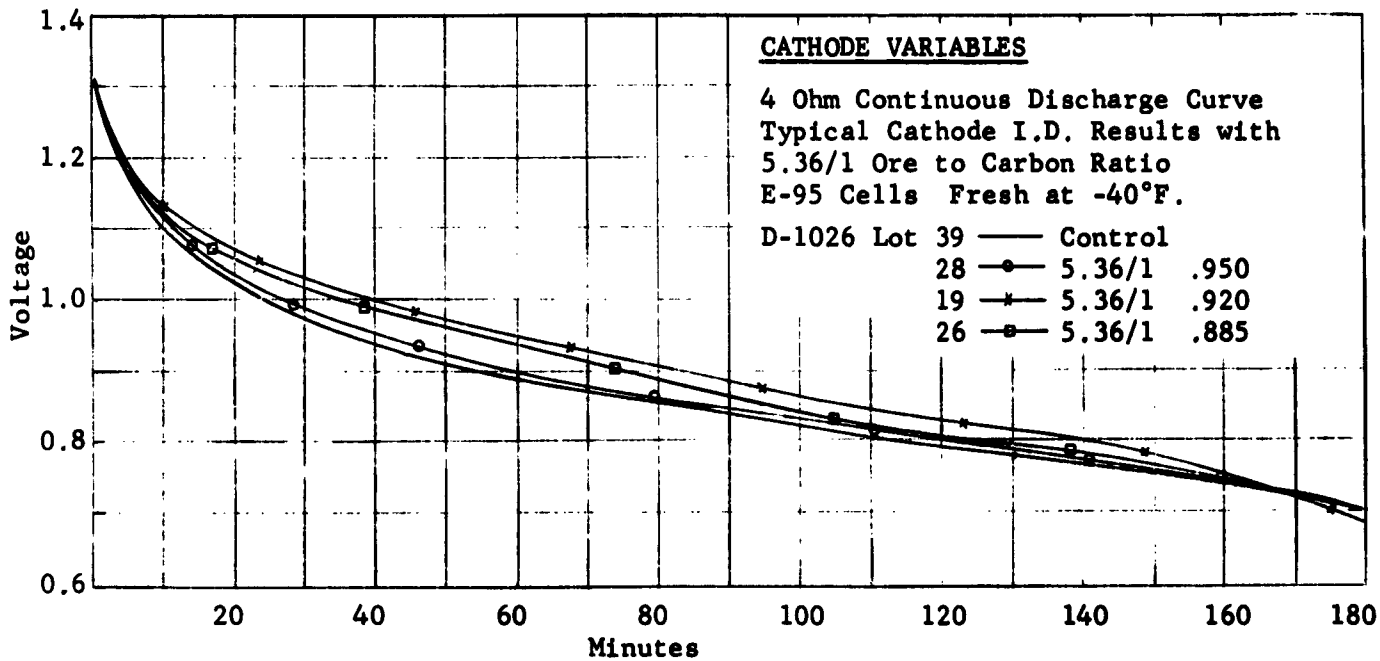
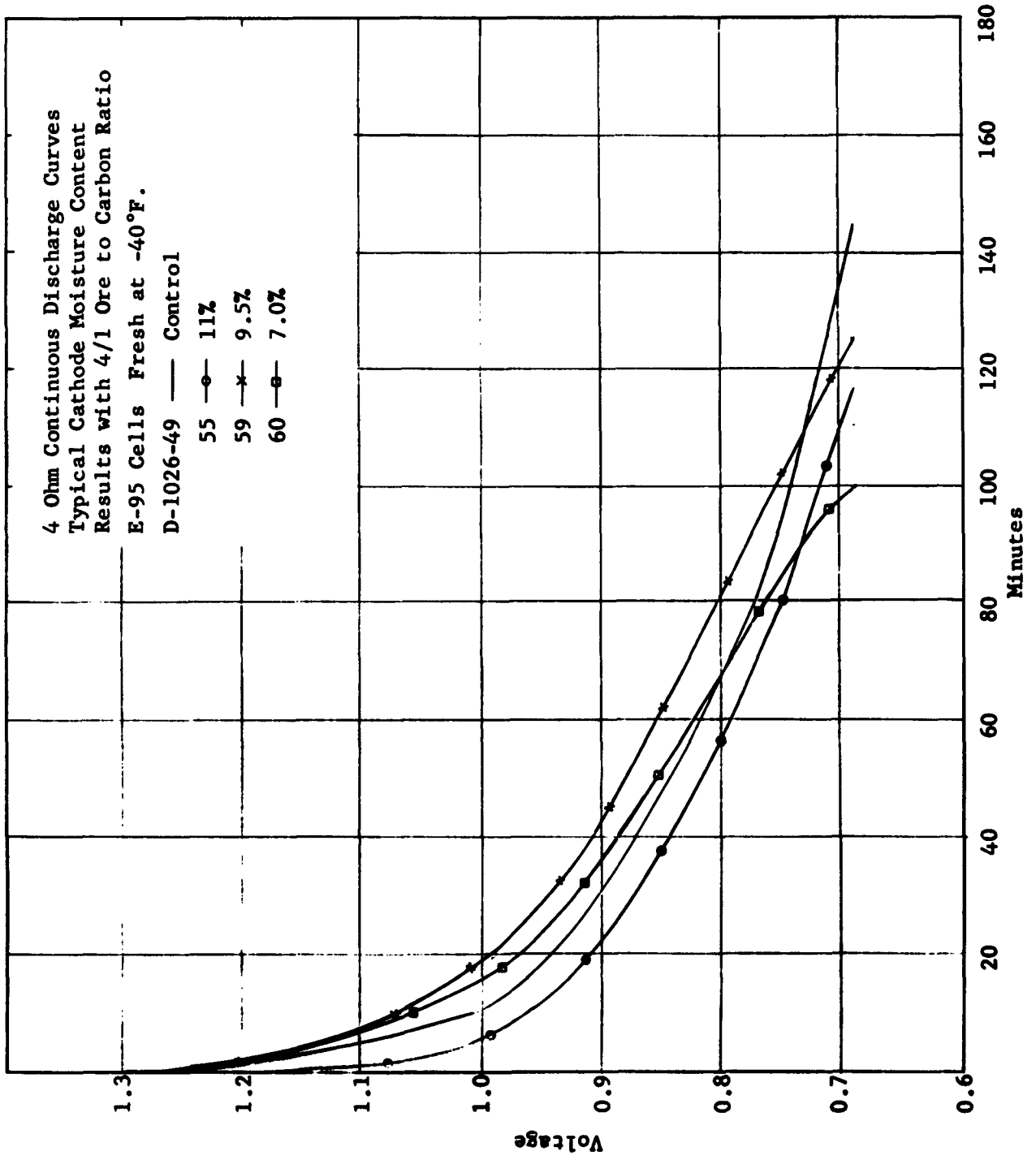


FIGURE 6



TYPICAL 4/1 ORE TO CARBON COMPARISON WITH ASG VS. AB + 2624 GRAPHITE
E-95 CELLS FRESH SERVICE AT -40°F.
2.25 OHM CONTINUOUS

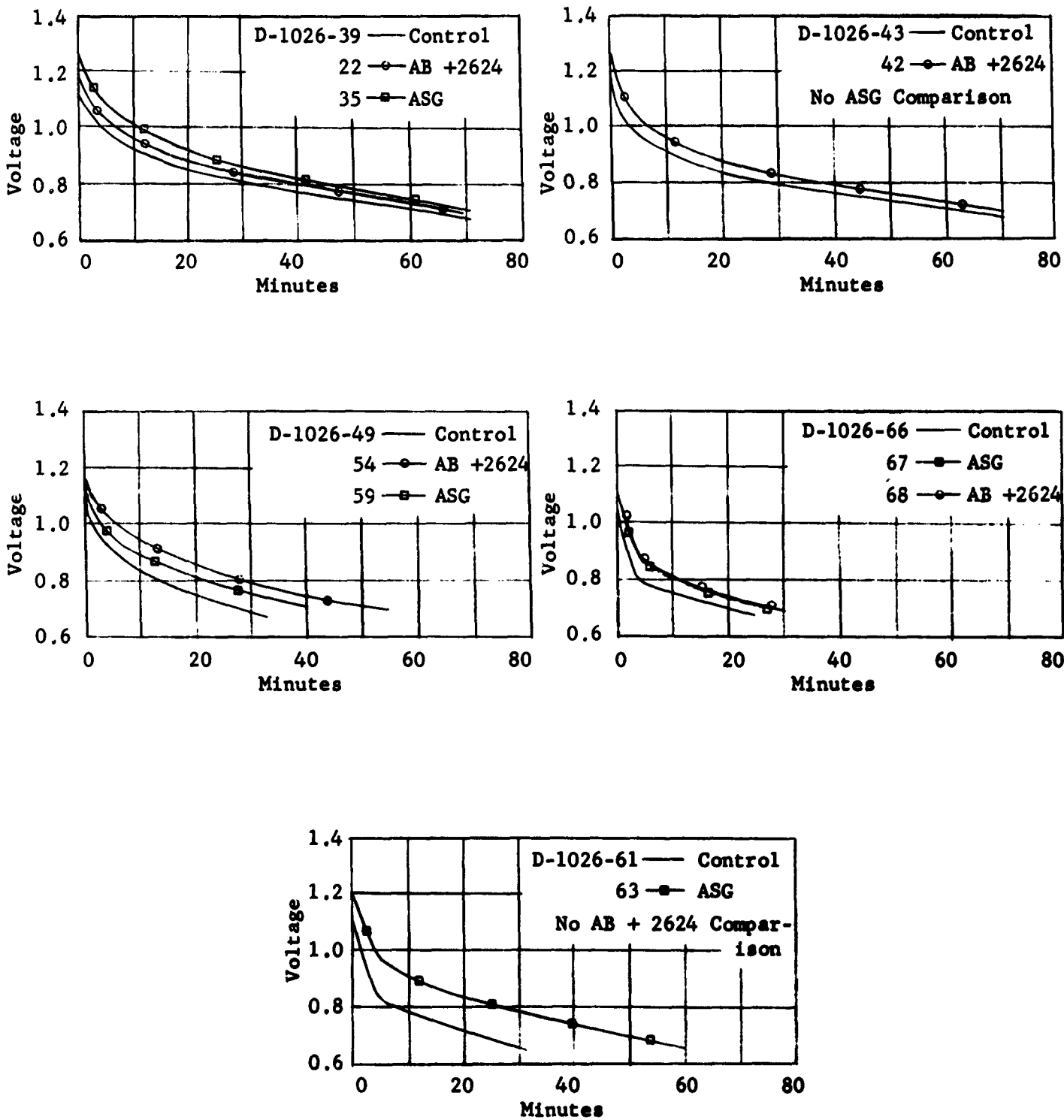


FIGURE 7

FIGURE 8
 TYPICAL 4/1 ORE TO CARBON COMPARISON WITH ASG VS. AB + 2624 GRAPHITE
 E-95 CELLS FRESH SERVICE AT -40°F.
 4 OHM CONTINUOUS

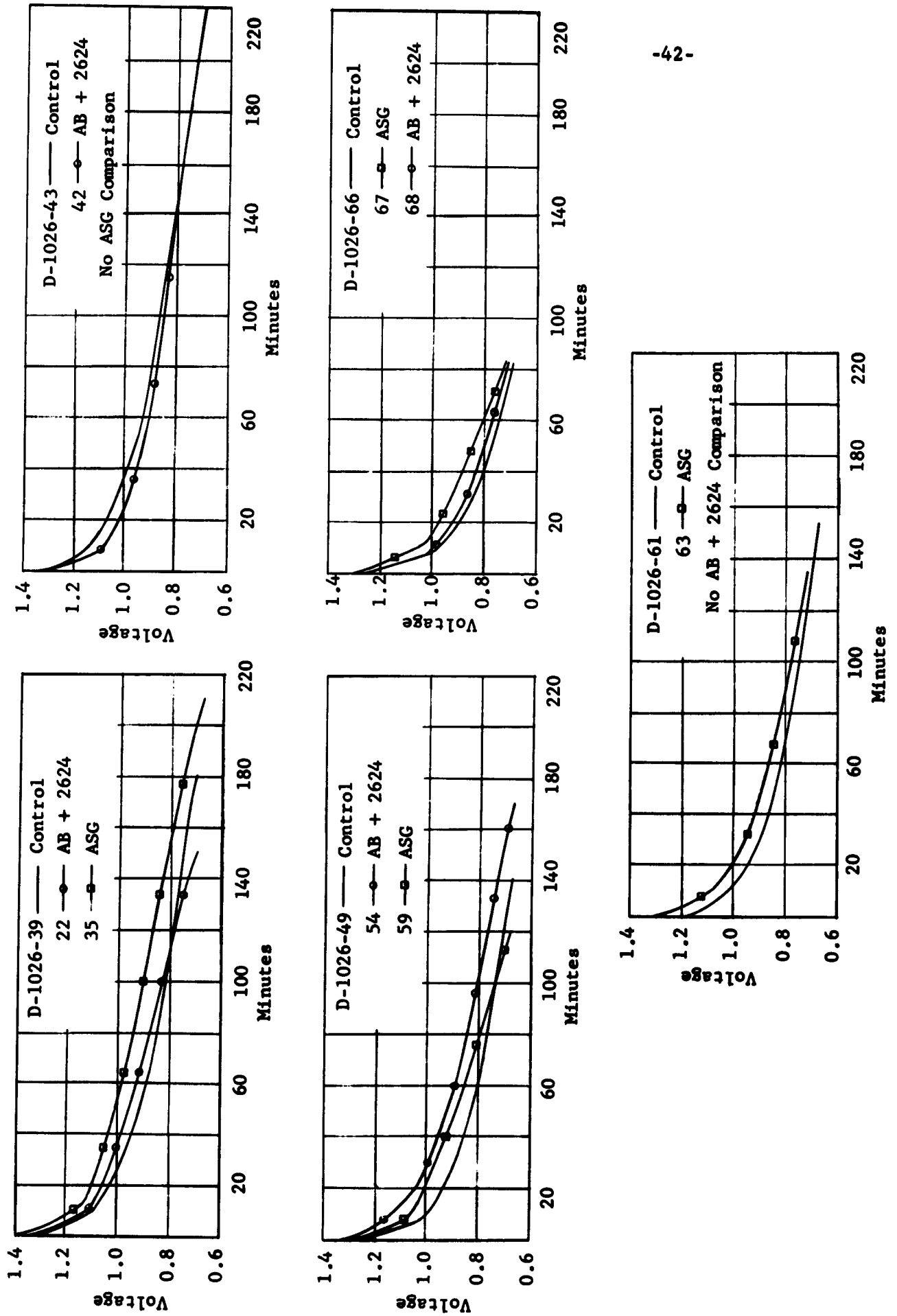


FIGURE 9

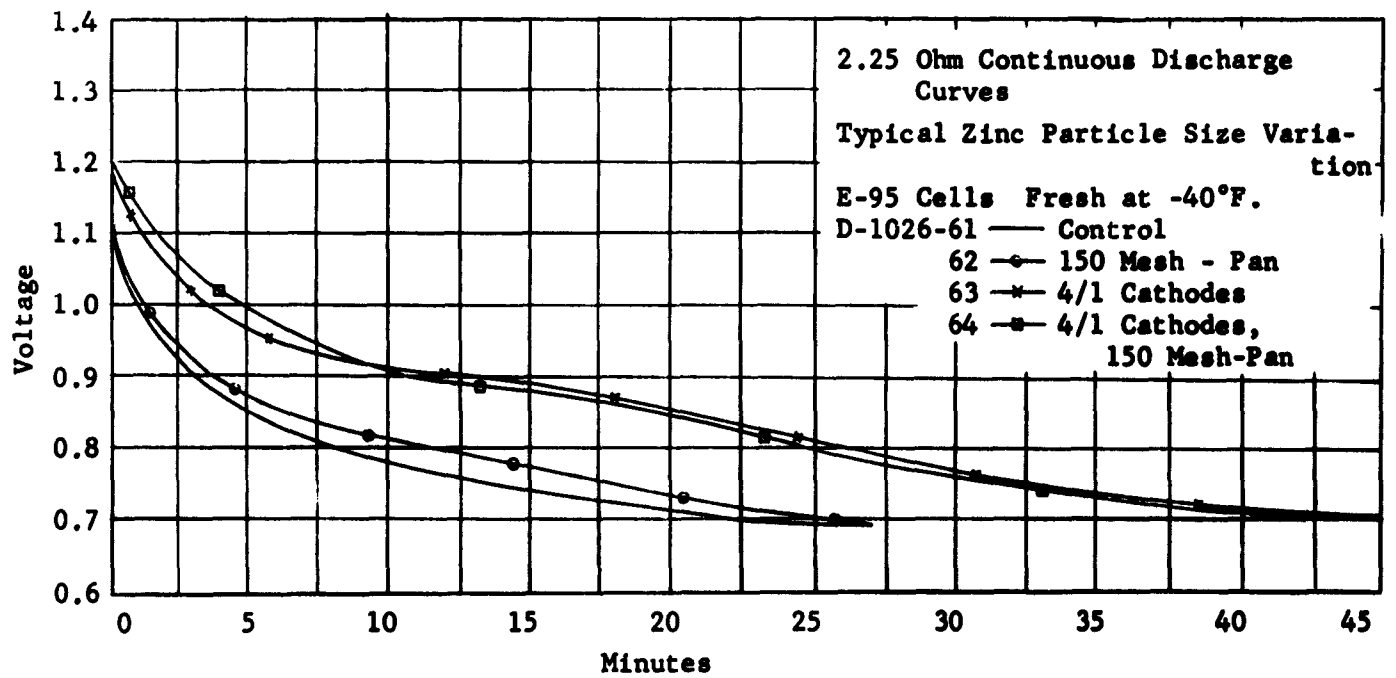
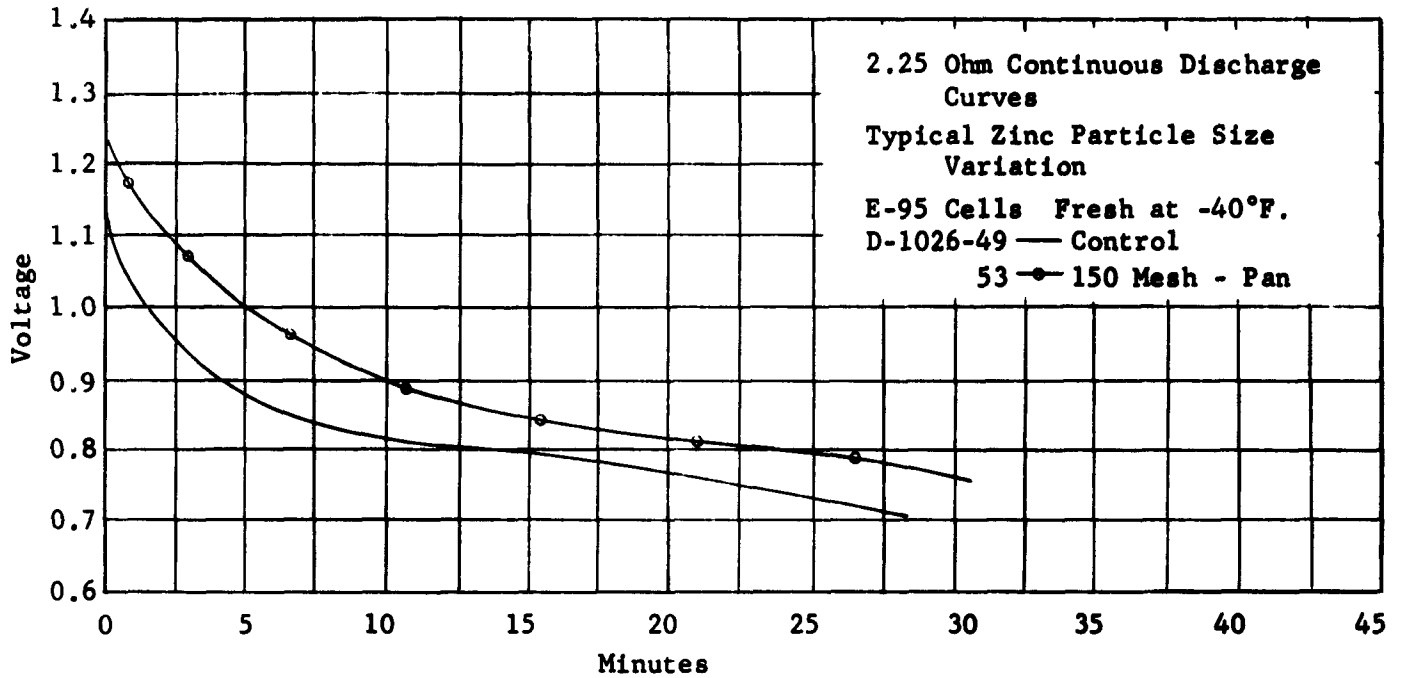
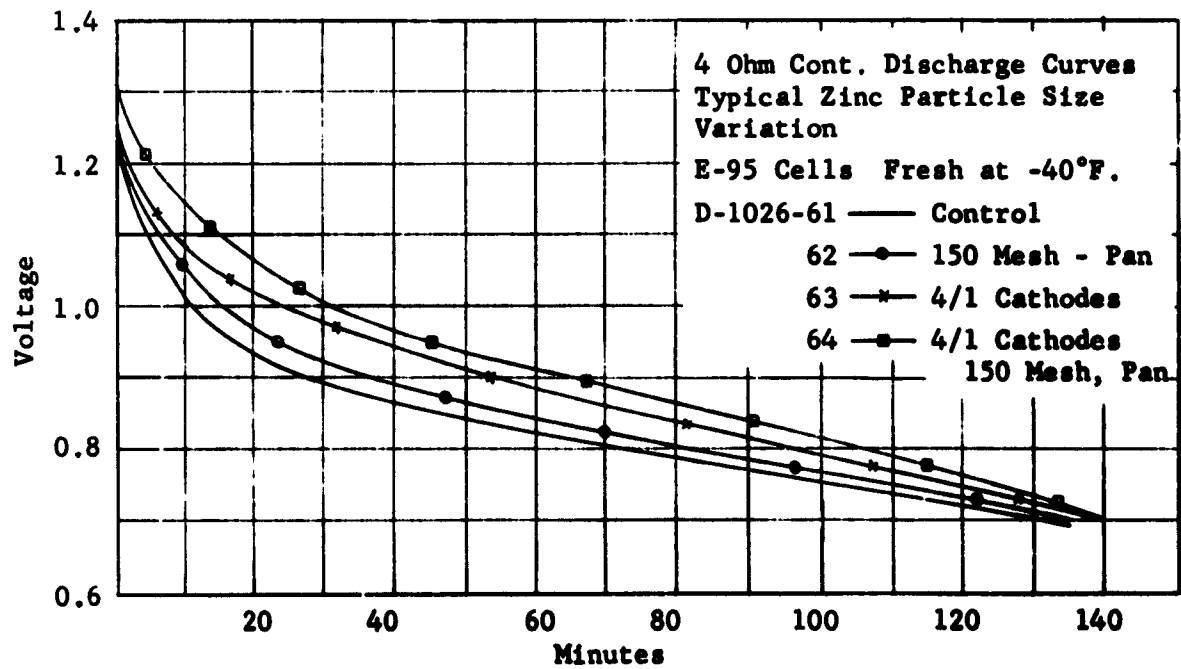
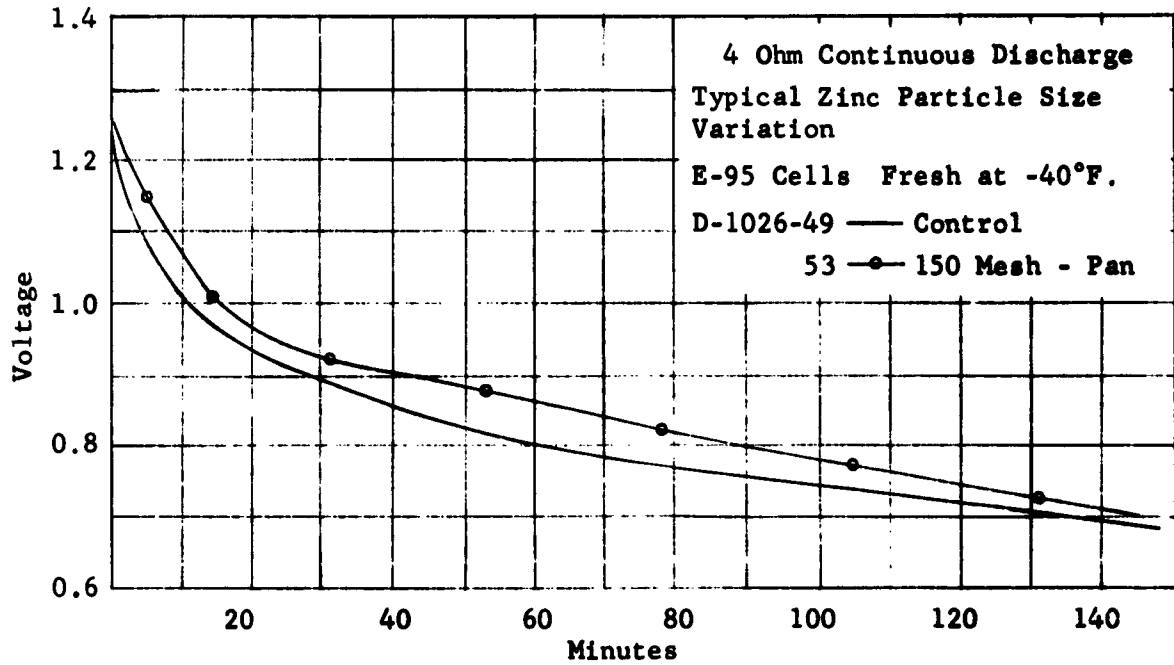


FIGURE 10



-40° SINE WAVE, .25 AMP. AVERAGE DISCHARGE

- D-1026-66 Control
- - - D-1026-67 Mix 218 (4/1 Cathode with ASG)
- * - D-1026-68 Mix 228 (4/1 Cathode with 2624 Graphite and Acetylene Black)
- o - D-1026-69 Mix 231 (4/1 Cathode with ASG and Dynel Binder)

FIGURE 11-A

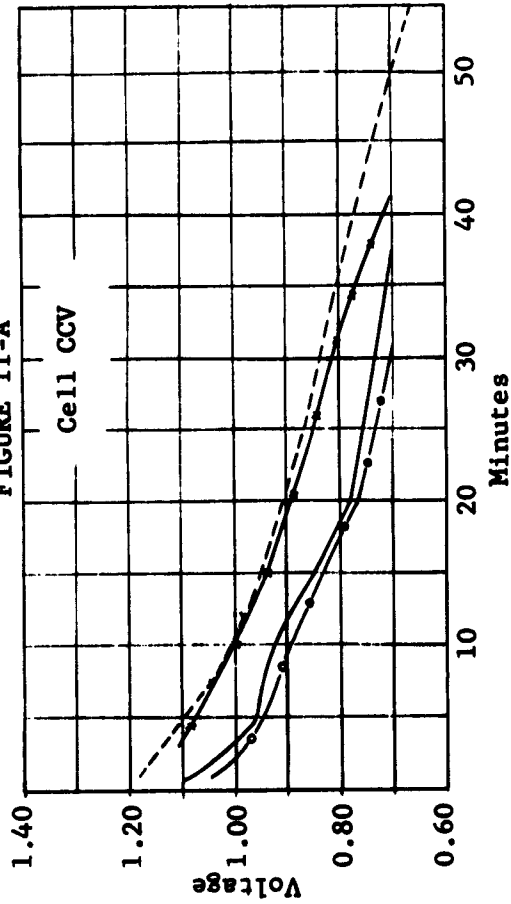


FIGURE 11-B

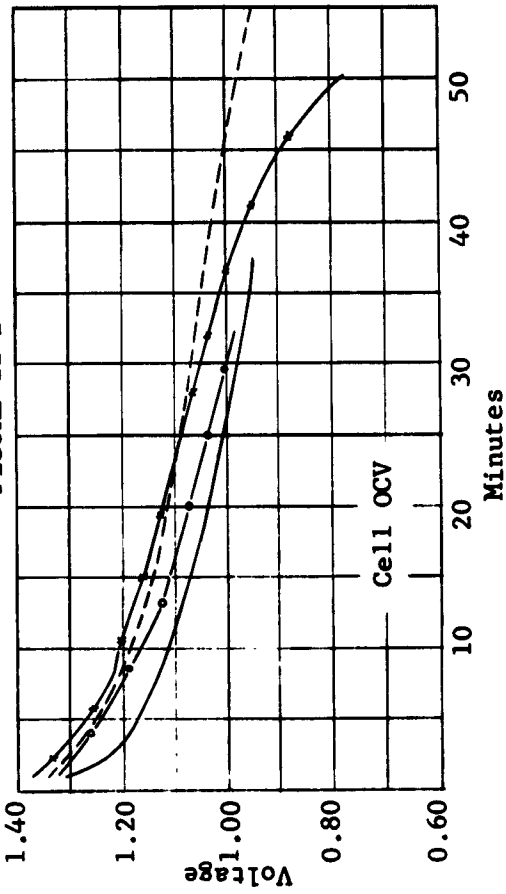


FIGURE 11-C

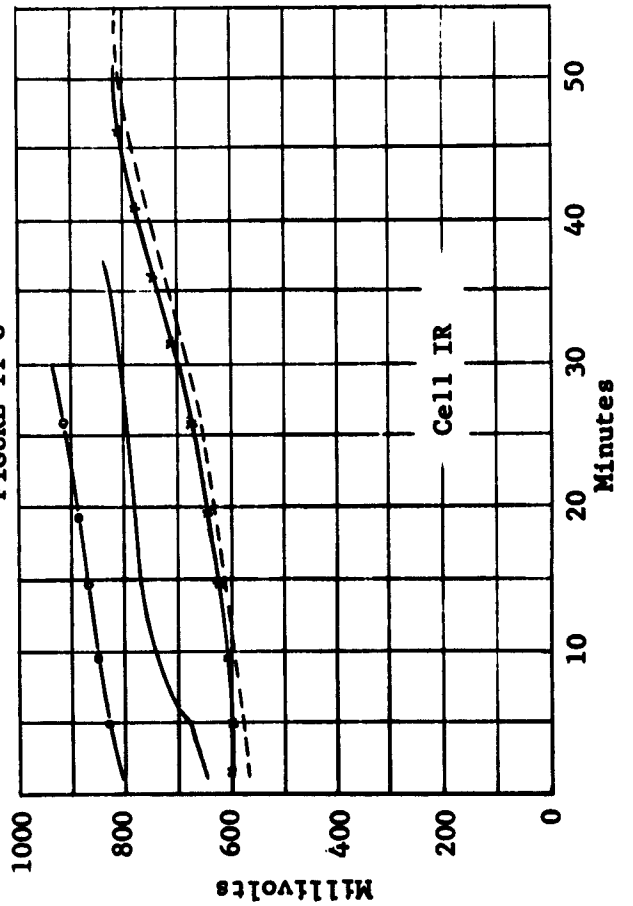
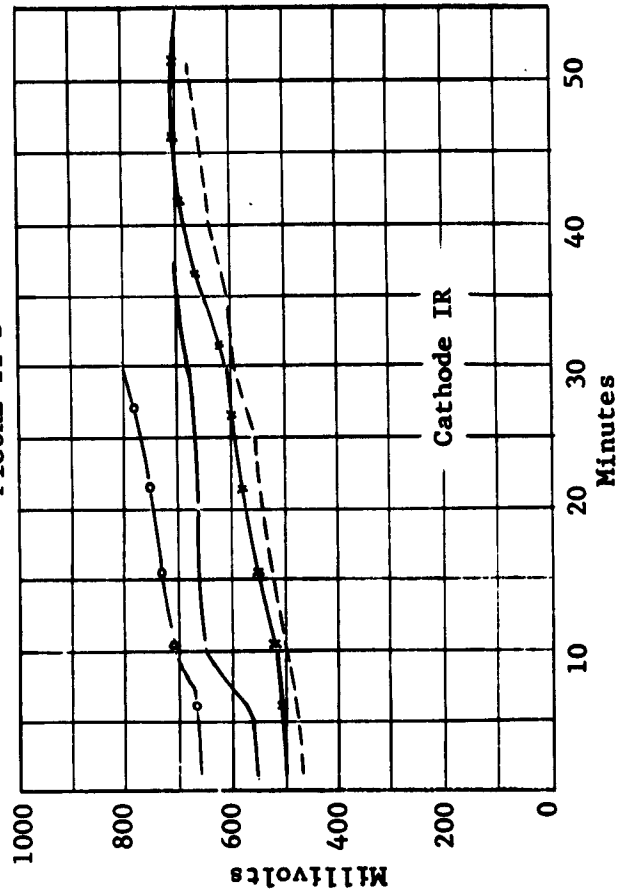


FIGURE 11-D



-40° SINE WAVE, .25 AMP. AVERAGE DISCHARGE

FIGURE 12-A

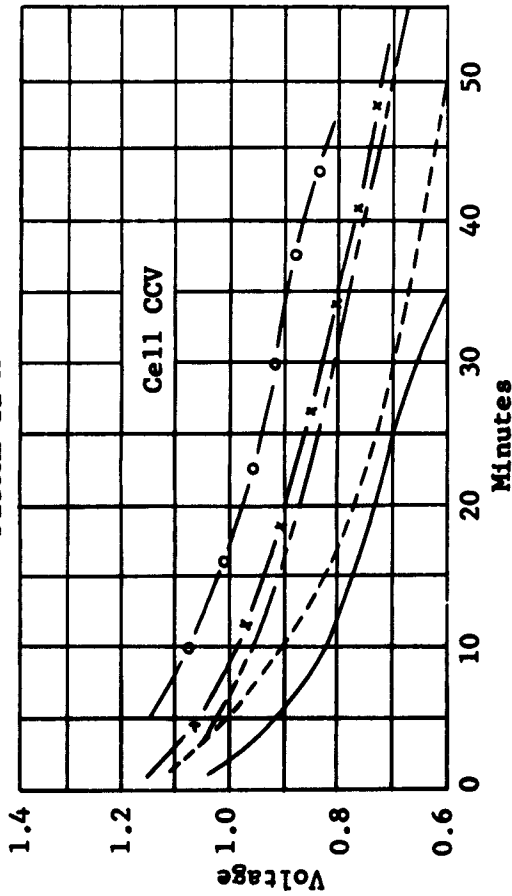
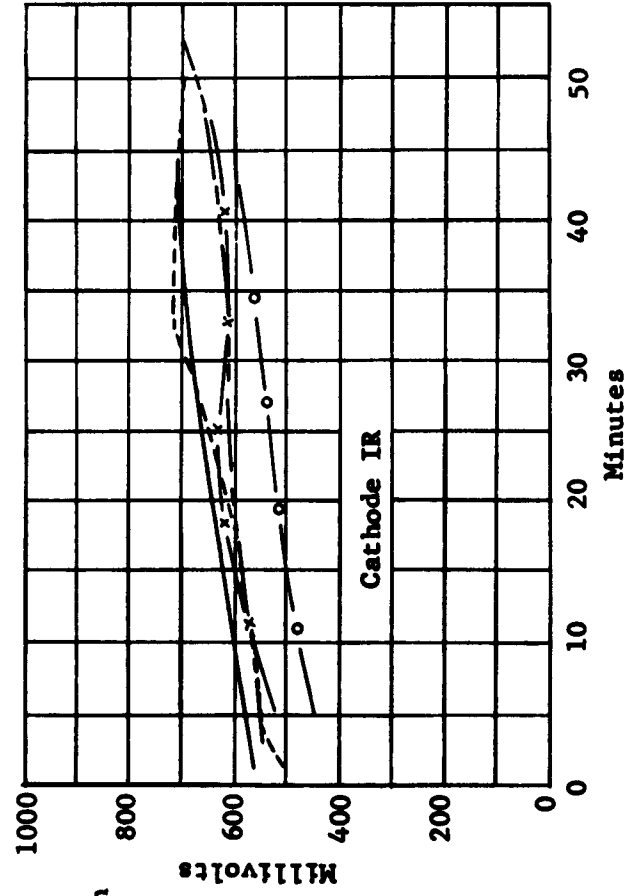
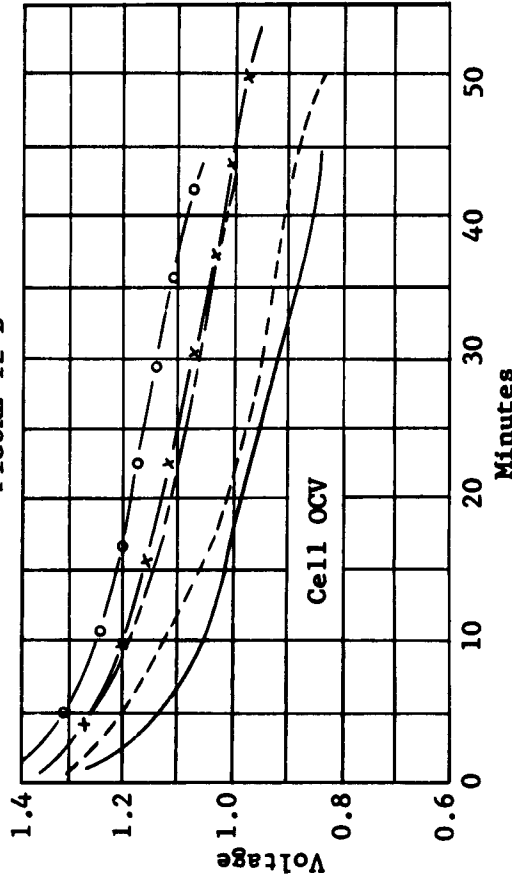
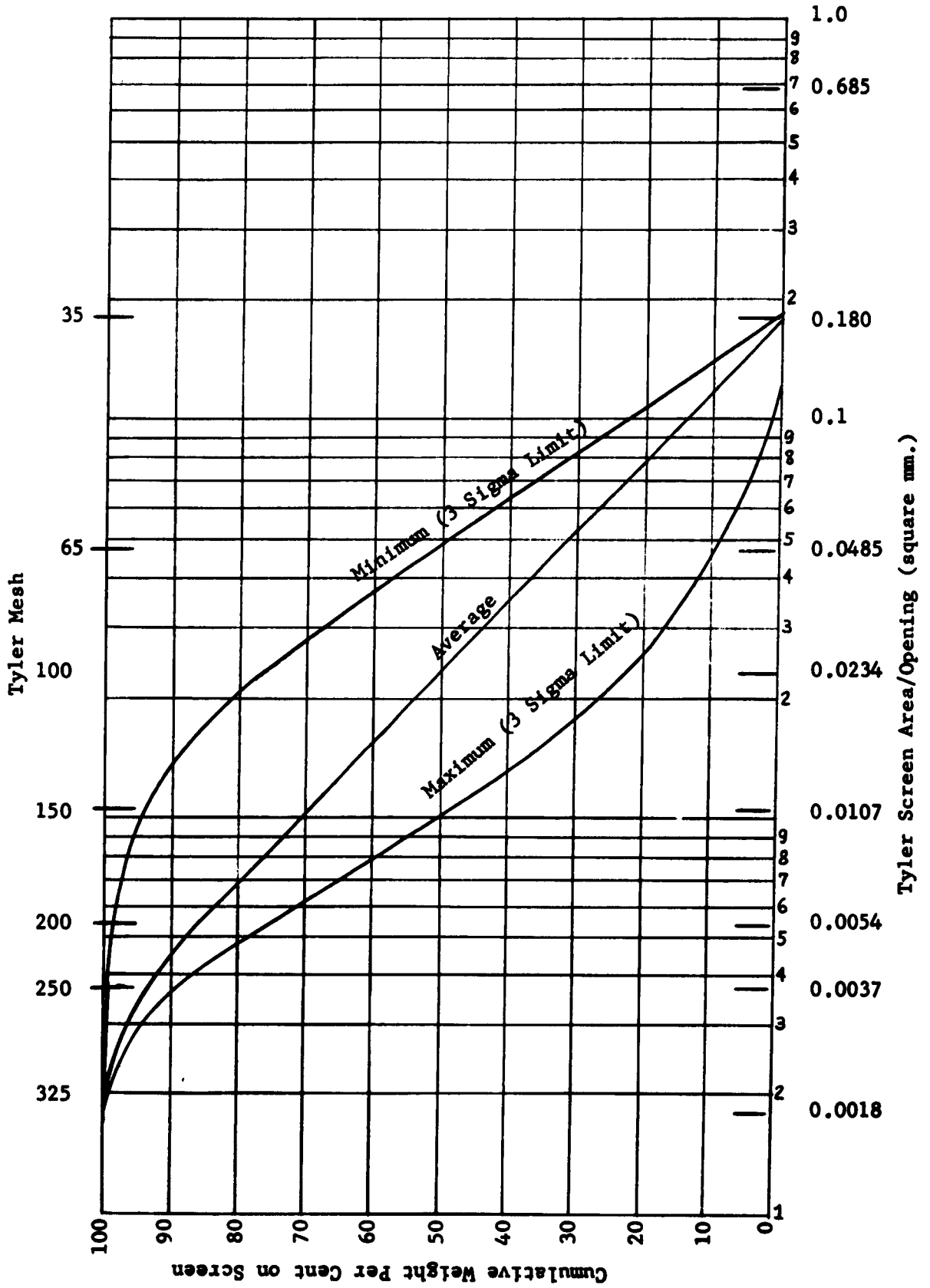


FIGURE 12-B



- D-1026-61, Control
- D-1026-62, Screened Zn
- x— D-1026-63, Mix 218
- o— D-1026-64, Mix 218 Screened Zn
- D-1026-65, Mix 230 KOH Flake

FIGURE 13
RM-976 ZINC ANALYSIS, PARTICLE SIZE VARIATION
(8 Lot Sample from 2 Shipments)



<p>AD _____ Accession No. _____</p> <p>Union Carbide Consumer Products Company, Electrochemical Product Development Laboratory, Cleveland, Ohio</p> <p>ALKALINE-H₂O₂ BATTERY</p> <p>J. Winger P. B. Bell</p> <p>Report No. 1, First Quarterly Report 1 May 1962 to 30 July 1962 47 pp. 13 Figures 18 Tables SC Contract MA-36-039-SC-89098 Project No. 3099-09-002-02</p> <p>Examination of present factory product E-95 "p" size alkaline-H₂O₂ cell at low temperatures with goal of optimizing acid cell for low temperature, heavy drain use. Discusses improvements in electrode formulations which improve -40°F. service through higher carbon phase cathodes and higher area zinc anodes. Relates problems of lot-let service uniformity with modified cathode formulation.</p>	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Present Product "p" size Alkaline-H₂O₂ examination at low temperatures 2. Improvements in electrode formulation. 3. Service uniformity with modified cathode formulations. 	<p>AD _____ Accession No. _____</p> <p>Union Carbide Consumer Products Company, Electrochemical Product Development Laboratory, Cleveland, Ohio</p> <p>ALKALINE-H₂O₂ BATTERY</p> <p>J. Winger P. B. Bell</p> <p>Report No. 1, First Quarterly Report 1 May 1962 to 30 July 1962 47 pp. 13 Figures 18 Tables SC Contract MA-36-039-SC-89098 Project No. 3099-09-002-02</p> <p>Examination of present factory product E-95 "p" size alkaline-H₂O₂ cell at low temperatures with goal of optimizing acid cell for low temperature, heavy drain use. Discusses improvements in electrode formulations which improve -40°F. service through higher carbon phase cathodes and higher area zinc anodes. Relates problems of lot-let service uniformity with modified cathode formulation.</p>	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Present Product "p" size Alkaline-H₂O₂ examination at low temperatures 2. Improvements in electrode formulation. 3. Service uniformity with modified cathode formulations.
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