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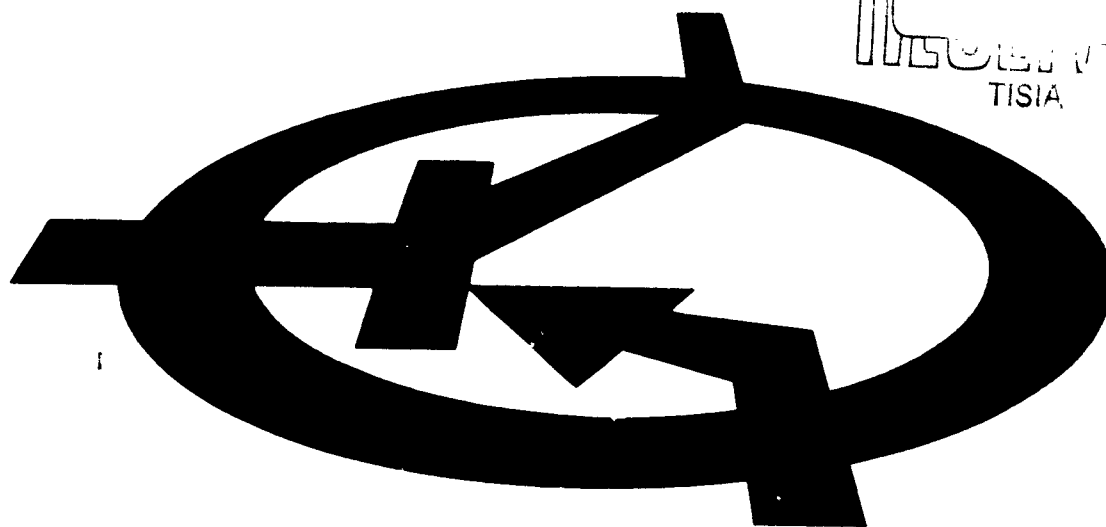
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ORGANIC DEPOLARIZED PRIMARY BATTERIES

REPORT NO. 2

SIGNAL CORPS CONTRACT NO.

DA-36-039-SC-87243

DEPARTMENT OF THE ARMY

PROJECT NO. 3A99-09-002

SECOND QUARTERLY PROGRESS REPORT
15 SEPTEMBER, 1961, TO 14 DECEMBER, 1961

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

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The objective of this research and development project is the development of primary batteries utilizing organic compounds as the active electrode components.

This report prepared by:

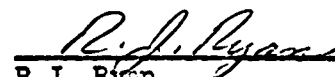
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1. PURPOSE

The purpose of this research and development contract is to:

1. Establish the role of carbon black in the nitro-organic-carbon cathode.
2. Determine the mechanism of the nitro reduction reaction.
3. Extend the development of the magnesium-m-dinitrobenzene cell to establish properties and reliability on shelf.

2. ABSTRACT

A method is presented for testing various carbon blacks used with organic nitro-cathode materials based on the ability of the carbon black to adsorb m-dinitrobenzene from 2N magnesium perchlorate saturated with m-dinitrobenzene. The data obtained from adsorption tests and capacity tests for adsorbed m-dinitrobenzene on Columbian carbon blacks and commercial carbon blacks of other manufacturers are summarized.

Effects of reaction products are discussed, and adsorption-capacity tests are summarized for two Columbian carbon blacks selected as superior to others tested. A-cell-capacity test data are given.

Eighteen-month storage-test results are given for AZ-10 magnesium A-cells made with Columbian 1100-CL 20212 carbon black. Three-month storage-test data are presented for AZ-21 magnesium A-cells made with Columbian 1100-P2142 HT 3232 and HR 1670-P3386 carbon blacks.

3. CONFERENCES

On September 26, 1961, Messrs. S. Bartosh and J. Pawlak of the U. S. Army Signal Research and Development Laboratories visited RCA in Somerville, New Jersey, to discuss progress under the contract. Present at the meeting were Dr. G. S. Lozier and Messrs. J. B. Eisen and R. J. Ryan of RCA.

On December 5, 1961, Messrs. J. B. Eisen, J. J. Meehan, and R. J. Ryan visited the U. S. Army Signal Research and Development Laboratories at Fort Monmouth, New Jersey, to discuss progress under the contract. Present at the meeting were Messrs. S. Bartosh, J. Hovendon, and J. Pawlak of the U. S. Army Signal Research and Development Laboratories.

4. EXPERIMENTAL AND FACTUAL DATA

4.1 RESEARCH STUDIES

A generalized five-step reaction mechanism for the m-dinitrobenzene (m-DNB) cathode was presented in the First Quarterly Report. The steps are:

- Step 1: Crystalline R-NO₂ is mechanically dispersed, but only partially in contact with carbon black.
- Step 2: R-NO₂ dissolves in electrolyte, and diffuses to carbon electrode.
- Step 3: R-NO₂ is in electrical contact with electrode; H₂O diffuses to reaction site.
- Step 4: R-NO₂ is reduced to R-NH₂ on the electrode surface.
- Step 5: R-NH₂ diffuses away to make room for another R-NO₂ molecule.

The above steps may be analyzed by the following quantitative measurements:

- (a) The ability of carbon black to adsorb m-DNB from an electrolyte saturated with the depolarizer.
- (b) The coulombic efficiency obtained upon discharge of the adsorbed m-DNB.
- (c) The effect of reaction products, as well as other cell mix components, upon the reduction of m-DNB.
- (d) The durability of a given carbon black in practical cell formulations.

A summary of studies based on the above factors is given in the following sections.

4.1.1 The Adsorption of m-Dinitrobenzene by Carbon Black

Adsorption studies were conducted with various Columbian and commercially available carbon blacks in a 2N $\text{Mg}(\text{ClO}_4)_2$ electrolyte saturated with m-DNB. The initial investigations evaluated the ability of various carbon blacks to adsorb m-DNB.

4.1.1.1 Adsorption Test

The choice of a test method was guided by

- (a) the low solubility of m-DNB in the 2N $\text{Mg}(\text{ClO}_4)_2$ electrolyte (1.0 gram per liter),
- (b) the time required to reach equilibrium following the introduction of the carbon-black sample into the solution, and
- (c) the ease of quantitative transfer of the carbon-black sample into the wet-test cell.

The apparatus is shown in Figure 4-1.

The carbon blacks tested showed wide variations in dry and wet bulk, and in wettability. For this reason, prolonged dispersion of the carbon black in the electrolyte was necessary. Various ratios of carbon/m-DNB were used during initial testing. It was determined that the optimum carbon black-electrolyte mixture consisted of 0.5 gram of carbon black dispersed in 250 ml of 2N $\text{Mg}(\text{ClO}_4)_2$ saturated with m-DNB. This ratio adequately meets the requirements for the m-DNB adsorption range, and provides a cathode of suitable size for the wet-cell test

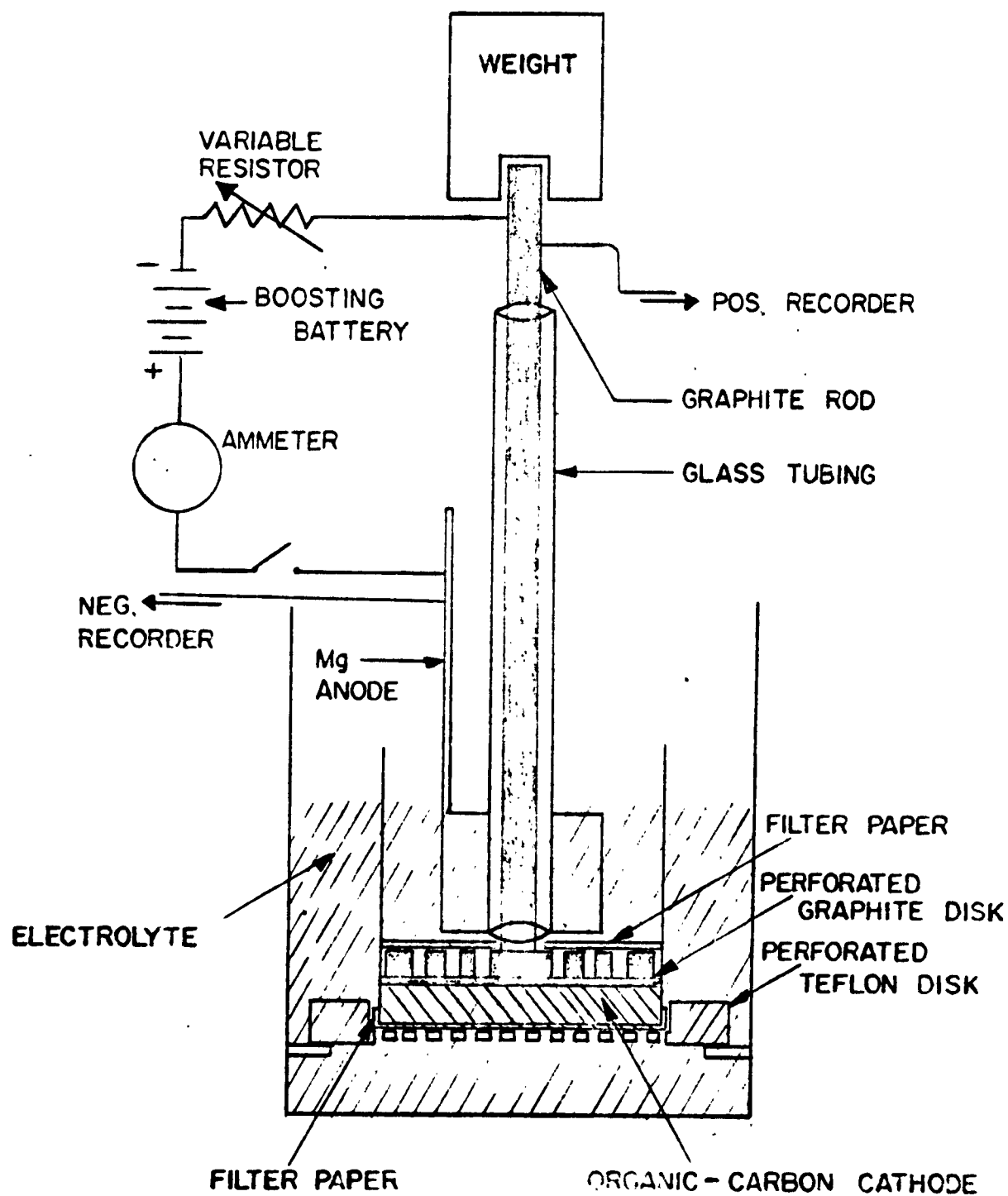


Figure 4-1. Wet-Cell Capacity Test Apparatus.

The 0.50-gram sample of carbon black is placed in a glass-stoppered 500-ml bottle with 250 ml of 2N $\text{Mg}(\text{ClO}_4)_2$ saturated with m-DNB (1 mg of m-DNB per ml of electrolyte). The mixture is agitated mildly by rolling the bottle on a ball mill. Although most carbon-black samples reached adsorption equilibrium in less than four hours, the procedure was standardized at 16 hours.

The carbon black-electrolyte mixture was filtered through a paper disc resting on a perforated Teflon plate. As a result, the carbon black formed a firm cake. Caution was exercised in filtering to insure the quantitative transfer of the carbon black. To avoid excessive packing of the precipitate, vacuum was applied only at the end of the filtration process. The moist, level, carbon-black cake was covered with a finely perforated graphite disc. The assembly, including the Teflon plate, was transferred to a 400-ml beaker. The graphite disc was covered with a filter paper disc provided with a central .25-inch-diameter hole. A graphite rod was placed in contact with the graphite disc under an applied weight of 1200 grams, forming a low-resistance contact.

An L-shaped, .016-inch-thick AZ-31B magnesium sheet, enclosed in a folded single layer of Nibroc Kraft separator paper, was bent into an upright annular anode. The lower portion of the graphite rod was sheathed with a glass tube and encircled by the anode. This anode structure permitted the free exit of hydrogen during cell discharge.

After the cell was assembled, 150 ml of 2N $\text{Mg}(\text{ClO}_4)_2$ was added. The cell voltage was recorded on a Leeds and Northrup Speedomax or a Varian Strip Chart Recorder. The electrolyte in the wet cell was agitated during initial testing by use of a magnetic stirrer. Subsequent testing indicated that this action was not necessary as it had no effect on the cell capacity.

A check test for effects of air depolarization was made on CBN P-1100-CL 20212 and CBN HR 1670-3386 cells by use of $\text{Mg}(\text{ClO}_4)_2$ purged with nitrogen. The results, given in Table 4-1, show that cell capacity was not affected.

4.1.1.2 Analytical Procedure

The amount of m-DNB adsorbed by the carbon black was determined by spectrophotometric analysis of residual m-DNB in the filtrate. The method is described in detail in Appendix 5.1.

4.1.1.3 Evaluation of Carbon Blacks

The ability of various carbon blacks to adsorb m-DNB from a 2N $\text{Mg}(\text{ClO}_4)_2$ electrolyte saturated with m-DNB ranges from zero removal to almost total removal. The lowest degree of adsorption occurred with carbon blacks supplied by Atlas (Darco), Cabot, and Shawinigan. In this property, these carbon blacks are similar to the CBN 10S67 series. Higher adsorption values were recorded for the CBN P-1100 series. The highest adsorption values were found with carbon blacks of the CBN HR 1670 series. Table 4-1 gives m-DNB adsorption and capacity data for

CARBON TYPE	ADSORP- TION TEST NO.	BULK	WETTING	SEDI- MENTA- TION OR FLOAT	FILTRA- TION	MG m-DNB ON 0.5 g CARBON BLACK	MIN- UTES AT 50 MA	EFFI- CIENCY (%)	A-CELL TEST TIME (hours) TO 0.9 VOLT		ABSORPTION OF WATER, g/g CARBON BLACK
									AT 4 OHMS	AT 16-2/3 OHMS	
<u>COLUMBIAN</u>											
P-1100-	36	Fluffy	Slow	50/50	Fast	174	300	75	4	29	5.0
-2142	37	Dense	Fast	Total		172	270	68			
-2142 #1	38			↓							
-2142 HT				Partial		178	360	88	3-5	30	4.1-4.6
3232	49										
-2142 HT	50		Slow			179	380	93			
-8417-1	51	Fluffy	↓			153	285	81	3.5	26.5	4.42
-8417-2	52		↓			167	390	100	4	28	5.13
-8417-3	82					171	300	76	3	29.5	4.23
-8417-4	8	Very Fluffy	Partial	Total		151	300	86	3.5	29	4.23
-CL 20212	9					184	340	80	5	33	4.32
	27					167	340	88			
	33					166	350	92			
	34					165	360	94			
	45					169	390	100			
	47					171	400	100			
11*	11*					170	360	92			
32**	32**					174	420	100			
						164	320	85			
10567	25		Slow	Partial	Fair	0	30	-	1.75	23	2.56
-Control	26		↓	↓	↓	0	30	-			
-3387	19		Fast		Fast	117	205	76	2.75	24	3.97
-3398	20		Slow	Flocculent		23	33	-	3	23.5	4.03
-3399	24		Partial	↓		133	280	92	2.25	24	4.54
-3522	15	Nodules	Fast	Not Dispersed		17	20	-	1.75	22.5	2.88
-3553	54	Fluffy	Slow	Partial		140	300	93	3	25.5	4.10

* Exposed to 500 ml of Electrolyte.

** In N₂ Atmosphere.

Table 4-1. Adsorption and Wet-Cell Capacity Data for m-Dinitrobenzene on Various Carbons Blacks. (Sheet 1 of 2)

CARBON TYPE	ADSORP- TION TEST NO.	BULK	WETTING	SEDI- MENTA- TION OR FLOAT	FILTRA- TION	MG m-DNB ON 0.5 g CARBON BLACK	MIN- UTES AT 50 MA	EFFI- CIENCY (%)	A-CELL TEST TIME (hours) TO 0.9 VOLT		ABSORPTION OF WATER, g/g CARBON BLACK
									AT 4 OHMS	AT 16-2/3 OHMS	
COLOMBIAN											
HR 1670											
-Control	17	Fluffy	Fast	Total	Fast	211	400	83	.3	27	2.43
-3379	16	↓ Dense	↓	↓	↓	195	370	83	1.75	23.5	2.43
-3380	18	↓	↓	Partial	Fair	83	120	62	0	14	2.05
-3381	21	↓	↓	Total	↓	111	240	93	0	0	1.47
-3382	22	↓	↓	↓	↓	160	240	65	1	15	1.54
-3383	23	↓	↓	Partial	↓	209	360	75	2.25	23.5	2.30
-3383	48	↓	↓	Slow	Slow	214	405	82			
-3386	13	Fluffy	Slow	Complete	Slow	234	420	78	3	25.5	4.48
-3386	34	↓	↓	↓	↓	233	420	78			
-3386	44	↓	↓	↓	↓	235	360	67			
-3386	46	↓	↓	↓	↓	234	380	70			
-3386**	10	↓	↓	↓	↓	233	400	75			
-3386**	12	↓	↓	↓	↓	231	440	83			
-3523	14	↓	Fast	Total	Fast	232	370	69	1.75	24	4.16
CABOT											
XC 72 F	6	Fluffy	Fast	Partial	Fast	52	100	83	3	22	2.95
Monarch 74	60	↓ Dense	↓	↓ Very	Fair	65	120	80			
Carbolac	61	↓	↓	Mobile	↓	109	270	100			
DARCO											
G-60	4	Dense	Fast	Total	Fast	99	145	63	3	20	1.36
G-60	5	↓	↓	↓	↓	96	120	55			
SHAWINIGAN											
50%											
Compressed	39	Fluffy	Fractional	Partial	Fair	25	35	-	2	18	3.60

* Exposed to 500 ml of Electrolyte.

** In N₂ Atmosphere.

Table 4-1. Adsorption and Wet-Cell Capacity Data for m-Dinitrobenzene on Various Carbons Blacks.
(Sheet 2 of 2)

various Columbian (CBN) and commercially available carbon blacks. A correlation of adsorption with capacity data for A-type AZ-10 magnesium cells is shown in Table 4-2. Manufacturers' data for several carbon blacks are given in Appendix 5-2.

Figure 4-2 presents a comparison of data obtained from testing three types of Columbian carbon blacks and several other carbon blacks commercially available from other suppliers. The samples of carbon black with adsorbed m-DNB were discharged at a constant current of 50 milliamperes. The curves show that m-DNB adsorbed on CBN P-1100-series and HR 1670-series carbon blacks is utilized more efficiently than it is with CBN 10S67 carbon blacks. The Columbian (CBN) samples are superior to the Darco, Cabot, and Shawinigan carbon blacks in this respect. The voltage decline was lowest for the CBN HR 1670-3386 sample.

The voltage data presented in Figure 4-2 are lower than the true polarization data because the values include an IR drop which is characteristic of the electrolyte, apparatus, and test procedure. Voltage levels will be important when an evaluation is made of the activation and polarization characteristics of carbon blacks.

In the studies conducted, the time required to reach the sharp break point on the voltage-time curve yields the coulombic capacity used in comparisons of the adsorption and efficiency of m-DNB with various carbon blacks.

The adsorption data show that CBN HR 1670-3386 carbon black has the greatest affinity for m-DNB under these test conditions. This carbon

CARBON TYPE	WET-CELL TEST MG m-DNB ON 0.5 g CARBON BLACK	MINUTES AT 50 MA	DRY-CELL TEST HOURS TO 0.90 VOLT	
			AT 4 OHMS	AT 16-2/3 OHMS
<u>COLUMBIAN</u>				
P-1100-2142	174	300	4	29
P-1100-2142 HT 3232	178	360	4	30
P-1100-8417-1	153	285	3.5	26.5
-2	167	390	4	28
-3	171	300	3	29.5
-4	151	80	3.5	29
P-1100-CL 20212	170	360	5.0	33
10S67-Control	0	30	1.75	23
-3387	117	205	2.75	24
-3398	23	33	3	23.5
-3399	133	280	2.25	24
-3522	17	20	1.75	22.5
-3553	140	300	3	25.5
HR 1670-Control	211	400	3	27
-3379	195	370	1.75	23.5
-3380	83	120	0	14
-3381	111	240	0	0
-3382	160	240	1	15
-3383	209	360	2.25	23.5
-3386	234	420	3	25.5
-3523	232	370	1.75	24.0
<u>CABOT</u>				
XC 72 F	52	100	3	22
<u>DARCO</u>				
G-60	99	145	3	20
<u>SHAWINIGAN</u>				
50% Compressed	25	35	2	18

Table 4-2. Comparison of Wet-Cell Capacity For m-Dinitrobenzene Adsorbed By Various Carbons With A-Cell Data.

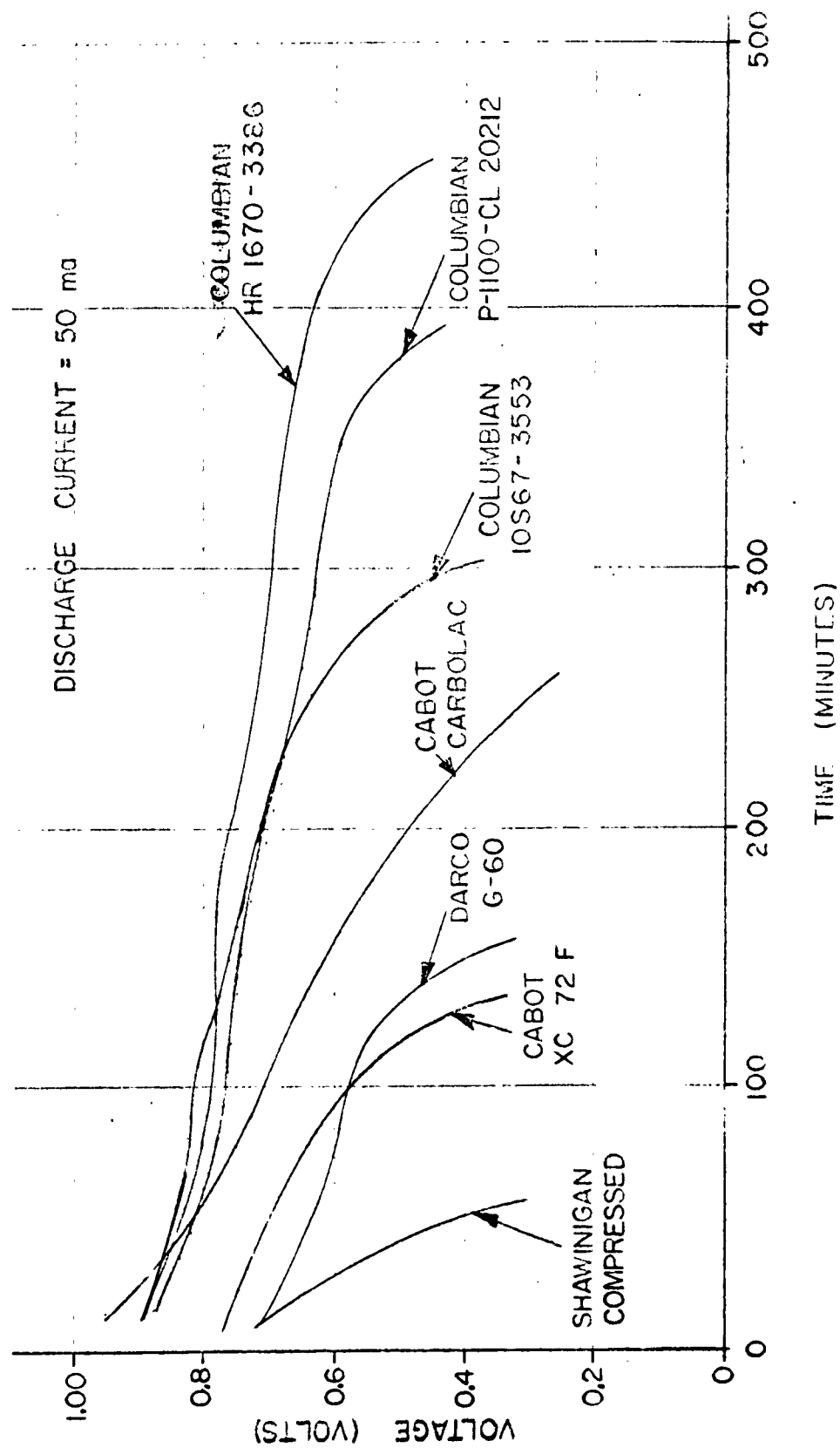


Figure 4-2. Capacity of m-Dinitrobenzene When Adsorbed By Various Carbon Blacks.

black also was superior to the others tested for capacity with the discharge current of 50 ma. Additional 0.5-gram samples of CBN P-1100-CL 20212 and CBN HR 1670-3386 carbon black were tested by exposing the samples to 500 ml of m-DNB-saturated 2N $\text{Mg}(\text{ClO}_4)_2$. The discharge current was 50 ma. The results showed an increase in m-DNB adsorption for CBN HR 1670-3386 carbon black with no loss in efficiency. No significant increase in adsorption was found with the CBN P-1100-CL 20212 carbon black. The data are summarized as follows for 0.5-gram samples of carbon black.

CARBON EXPOSED TO:	0.5 gm COLUMBIAN CARBON BLACKS	
	HR 1670-3386	P-1100-CL 20212
<u>250 ml Electrolyte</u>		
amount of m-DNB adsorbed	233 mg	167 mg
discharge time at 50 ma	420 min	390 min
cathode efficiency	78%	100%
<u>500 ml Electrolyte</u>		
amount of m-DNB adsorbed	335 mg	174 mg
discharge time at 50 ma	670 min	420 min
cathode efficiency	87%	100%

The data show general correlation between the adsorbed quantity of m-DNB and its 50-ma discharge capacity. The discrepancy between the adsorbed quantity of m-DNB and the 50-ma discharge capacity may be due to any of these reasons:

1. The active sites on the carbon black are capable of desorbing the discharge products, and are thus available for adsorbing additional m-DNB. This could permit a carbon black having an initially poor adsorption to give better results in a cell than a carbon black with a very high initial m-DNB adsorption value.
2. An optimum ration of $H_2O/m\text{-DNB}$ is not maintained during the life of the carbon black.
3. Structure breakdown of the carbon black by mass transport takes place during discharge.
4. Desorption of a quantity of m-DNB into the electrolyte takes place during the wet-cell test.

The m-DNB adsorption test also provides data on the dry bulk of the carbon black, wettability in the electrolyte, sedimentation, and behavior of the carbon black on filtration. As a general rule, the dense or nodular carbon blacks adsorbed less m-DNB than did the ultra-fine, fluffy carbon blacks. Slow or partial wetting suggests a heterogeneous surface condition. In the dense carbon blacks, this characteristic may inhibit dispersion and, possibly, the access of electrolyte. The degree of flocculation and sedimentation of carbon black dispersed in excess electrolyte may illustrate the persistence, development, or change of a heterogeneous surface condition. Considerable variation was observed in the speed of filtration with different carbon blacks. CBN HR 1670-3386 is particularly slow.

4.1.1.4 Effect of Reaction Products

The effect of reaction products was evaluated by the following two general methods:

1. Effect of the major reaction products on adsorption of m-DNB by carbon black.
2. Regeneration of carbon blacks, after discharge, by further treatment with 2N $\text{Mg}(\text{ClO}_4)_2$ electrolyte saturated with m-DNB.

The first method provides information concerning the preferred species adsorbed on carbon black. Studies were conducted on the adsorption of m-DNB from 2N $\text{Mg}(\text{ClO}_4)_2$ by carbon blacks in the presence of reaction products. Two samples of Columbian Carbon blacks, CBN P-1100-CL 20212 and HR 1670-3386, were examined. In one study, m-phenylenediamine was present; in the other, both m-phenylenediamine and n-nitroaniline were present.

Results showed that the capacity obtained from the testing of these carbon blacks in the wet cell was influenced by a reduced adsorption of m-DNB. Higher cell capacity was obtained from testing of HR 1670-3386 carbon black. Capacity data are presented in Figures 4-3 and 4-4.

The adsorption of m-DNB by 0.5 g of Columbian carbon black in the presence of reaction products is shown below. The quantities listed in columns 2 and 3 were adsorbed upon exposure of the carbon blacks to 250 ml of 2N $\text{Mg}(\text{ClO}_4)_2$ containing the compounds listed in column 1.

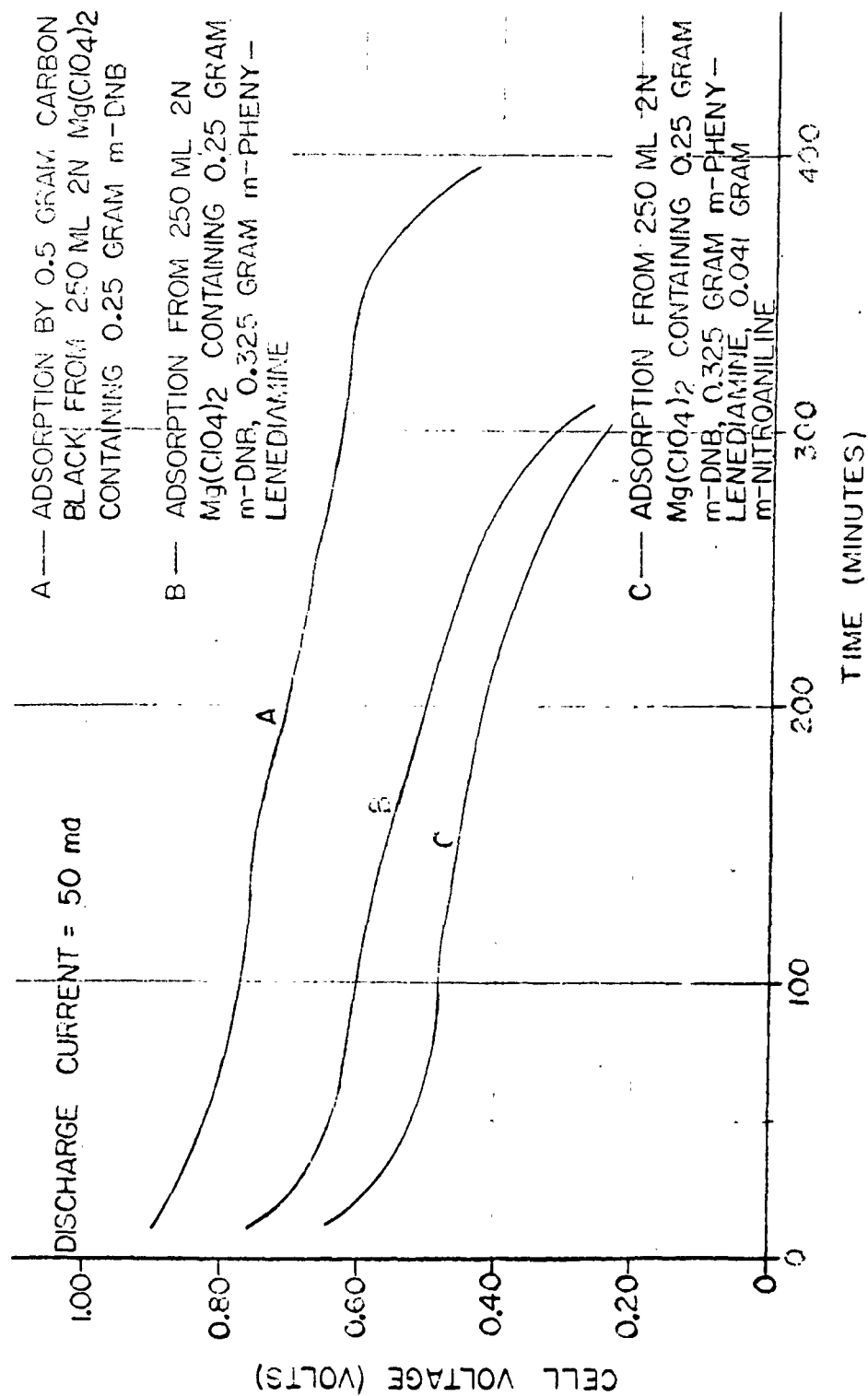


Figure 4-3. Capacity of m-Dinitrobenzene Adsorbed by Columbian P-1100-CL 20212 Carbon Black in the Presence of Selected Reaction Products.

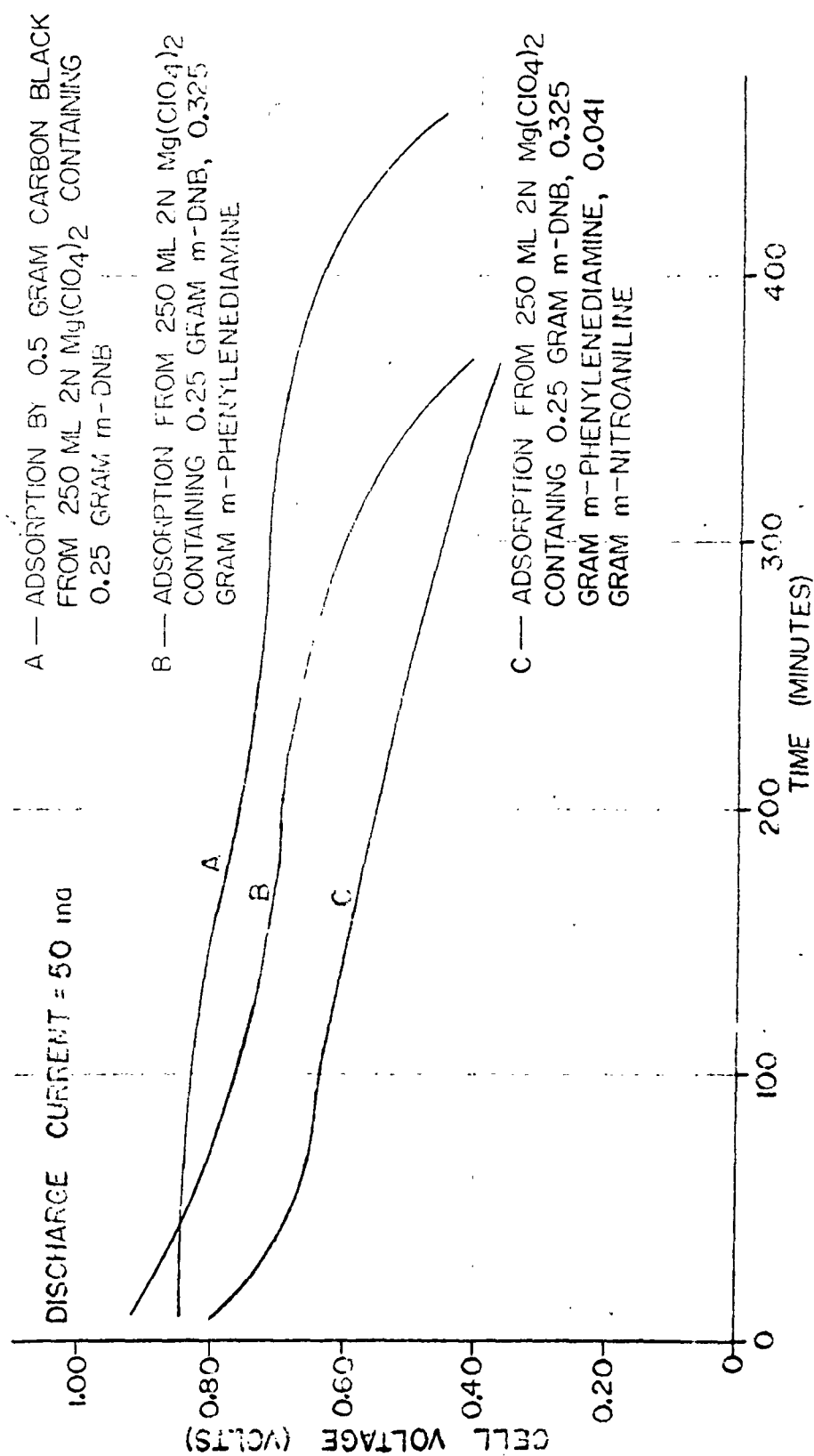


Figure 4-4. Capacity of m-Dinitrobenzene Adsorbed by Columbian HR 1670-3386 Carbon Black in the Presence of Selected Reaction Products.

REFER TO CURVE OF FIGURES 4.3 and 4.4:	250 ML OF 2N $\text{Mg}(\text{ClO}_4)_2$ CONTAINING	m-DNB (grams) ADSORBED BY 0.5 GRAM OF:	
		HR 1670-P3386	PL100-CL 20212
A	0.25 gm m-DNB	0.235	0.170
B	0.25 gm m-DNB and 0.325 gm m-phenylenediamine	0.197	0.124
C	0.25 gm m-DNB, 0.325 gm m-phenylenediamine, and 0.041 gm m-nitroaniline	0.179	0.112

The second method is directed toward determining the ability of the carbon black to desorb the reaction products formed during discharge. The effect of additional adsorption of m-DNB was studied by discharging the initially adsorbed m-DNB and exposing the carbon sample to a second 250-ml portion of electrolyte saturated with m-DNB by simple percolation through the packed test cell. Test results showed a definite decline of capacity on the second discharge with the same carbon black sample. For the third exposure the sample of carbon black was fully redispersed in 250 ml of electrolyte saturated with m-DNB, and formed into a wet cell. Performance decreased again. The data are presented in Figures 4-5 and 4-6. The data obtained from the two methods show that the active sites on the carbon blacks are blocked by reaction products. It can not be determined at this point if there is a desorption of reaction products followed by adsorption of more m-DNB, or if new active sites become available.

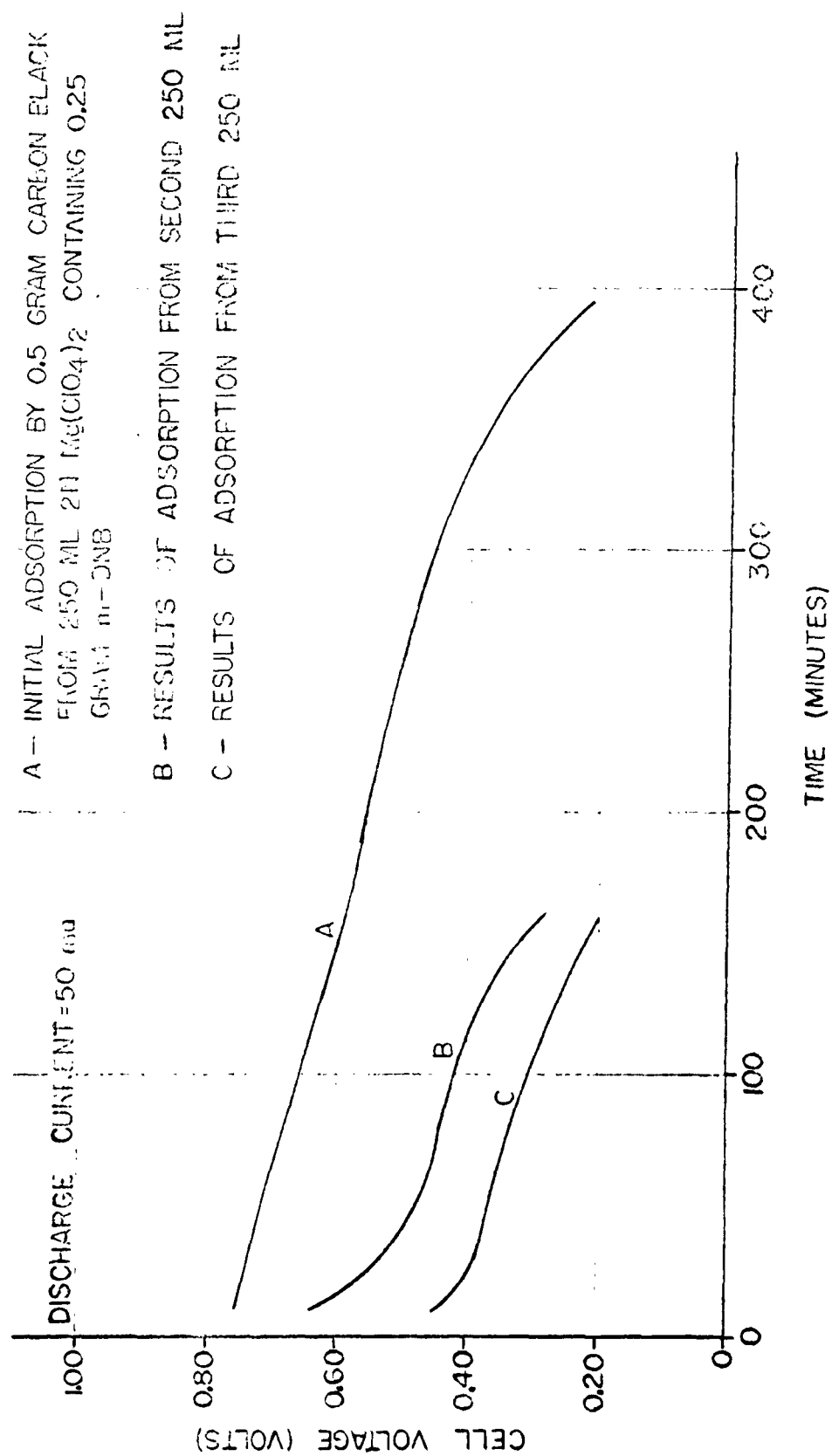


Figure 4-5. Effect of the Progressive Adsorption of m-Dinitrobenzene by Columbian P-1100-CL 20212 Carbon Black Upon Wet-Cell Capacity.

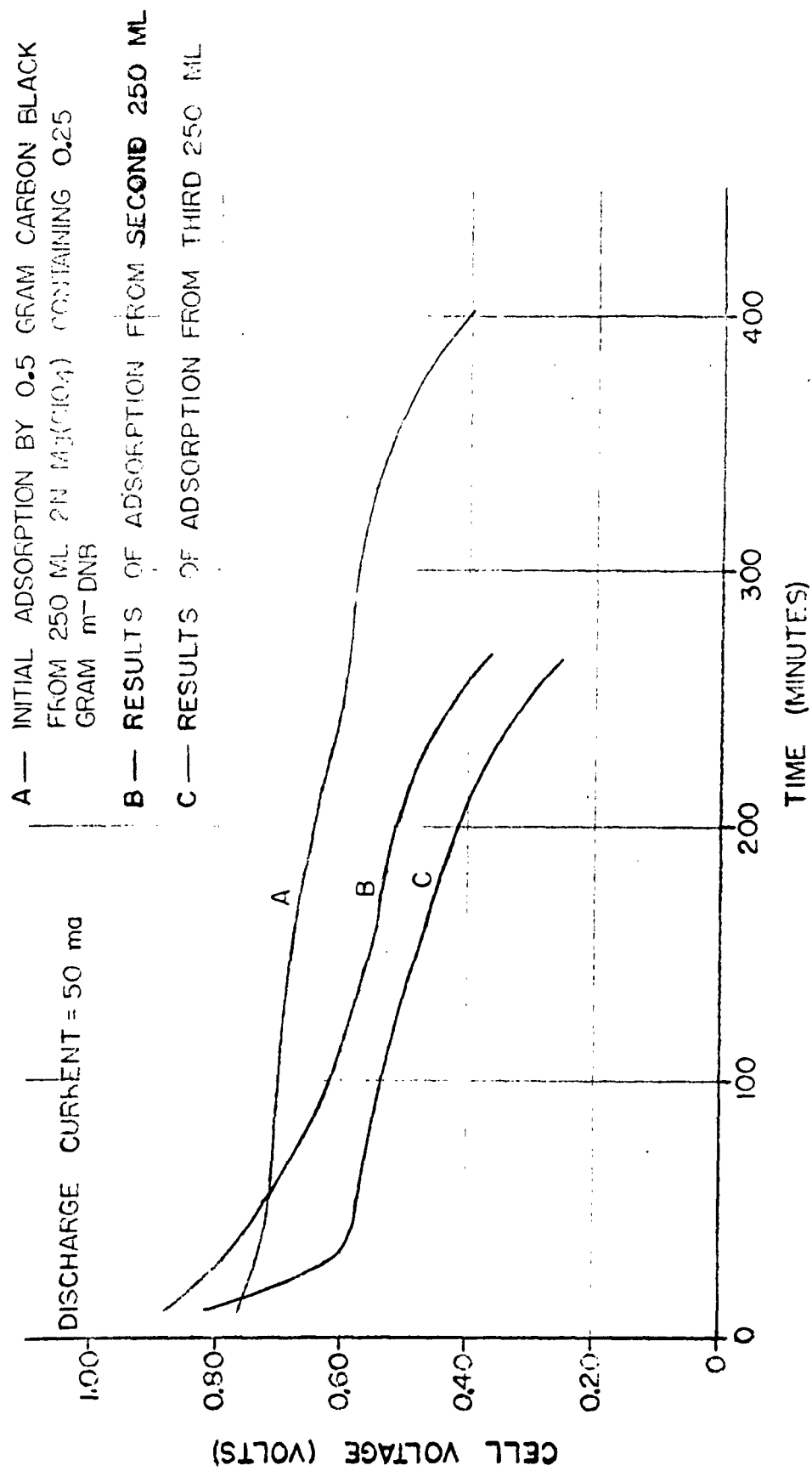


Figure 4-6. Effect of the Progressive Adsorption of m-Dinitrobenzene by Carbon Black Upon Wet-Cell Capacity.

The present data suggest partial desorption of some sites. An attempt will be made to determine the number of times active sites become available for reaction by adding the total ampere-minutes for a given carbon black sample. This should provide a reasonable approximation of the actual cell.

4.1.1.5 Effect of Other Cathode-Mix Components

Comparison of the adsorption and wet-cell test data shows that CBN HR 1670-3386 carbon black adsorbs more m-DNB and has greater capacity than CBN P-1100-CL 20212. These advantages, however, do not hold for A-cell tests, where the CBN P-1100-CL 20212 is superior. An examination of both carbon blacks was made by studying the effect of A-cell cathode-mix components on the wet-cell capacity. The transition from the wet cell to the composition of the A-cell mix was tested in this sequence:

MATERIALS	COMPOSITION (grams)			
	I	II	III	IV
carbon black	0.5	0.5	0.5	0.5
m-DNB	1.	1.	1.	1.
BaCrO ₄	none	.09	.09	none
Mg(OH) ₂	none	none	.03	.03

These components were milled gently for one hour, transferred in a level

layer into the wet-cell assembly previously described, saturated in 2N $\text{Mg}(\text{ClO}_4)_2$, and discharged at 50 ma. The discharge times were found to be as follows:

CARBON TYPE	COMPOSITION (hours)			
	I	II	III	IV
CBN HR 1670-3386	11	8	12	21
CBN P-1100-CL 20212	9	11	16	21

Examination of the discharged wet cells showed extreme hardening of the CBN HR 1670-3386 cathode with BaCrO_4 . The advantage of composition IV, which contained no BaCrO_4 , did not materialize in A-cells. The anomaly deserves further study. It suggests either a structure breakdown of the carbon black during discharge in A-cells (the observed hard setting would affect the diffusion process required for optimum cell performance), or some unknown cathode phenomenon not related to the anode inhibitor characteristic of BaCrO_4 .

4.1.1.6 Discussion of Test Results

A comparison of wet-cell capacities from adsorbed m-DNB, along with information on A-cell performance, is presented in Table 4-2. Variations noted in the performance of different carbon blacks make general conclusions difficult. The CBN 10S67-control sample and several CBN HR 1670 samples, although very fluffy, absorb less water than needed

for the 2:1 m-DNB:carbon black cathode mix. For example, the CBN HR 1670-control sample shows 27 hours for the 16-2/3-ohms test, which represents complete utilization of the water content. Simultaneous high values in water absorption and m-DNB adsorption are not unfailing criteria for superior A-cell capacity, as shown for CBN HR 1670-3386 and -3523 carbon blacks. It is reasonable, though, to expect poor A-cell performance from a combined low rating in both properties.

Carbon blacks of apparently "ideal" sorption properties often give relatively poorer 4-ohm A-cell test results than would be expected from the 16-2/3-ohm results. These results may be related to the apparent discrepancies noted above. Failure at the higher current density may be due to poor diffusion resulting from immobility of the electrolyte. The hard-setting mix cake, obtained in the CBN HR 1670-3386 carbon black with BaCrO_4 , is not conducive to a rapid exchange between reaction products and m-DNB necessary to sustain a high-current discharge.

Variations in data obtained from the 4-ohm A-cell tests with carbon blacks of the CBN 10S67 and HR 1670 types emphasize that the adsorption of m-DNB on carbon black is not the rate-controlling property. The data reveal that m-DNB can be discharged efficiently in a tightly packed A-cell with a carbon black which does not adsorb m-DNB from its electrolyte solution. Another factor which must be considered in the deviations between the wet-cell test and the dry-cell results is a structure breakdown within the cathode. During the discharge of a dry cell, two physical changes occur to alter the physical structure of the cathode. These changes are:

1. Movement of the free water from the cathode mix through the separator to the anode.
2. Transport of m-DNB to carbon black. This action is probably preceded by a dissolution process.

In a typical m-DNB (2-to-1 ratio) cathode, water comprises approximately 60 per cent of the total cathode volume. During the discharge of an A-cell with a $16\frac{2}{3}$ -ohm load for 27 hours, approximately 50 per cent of the water must be transferred out of the cathode mix through the separator to the Mg anode. Magnesium consumes water in the formation of its hydroxide and hydrogen. This means that the carbon black cathode structure must withstand a volume change of 30 per cent during discharge.

The solid m-DNB must dissolve before it can reach the carbon black. This process will also remove substance from the original cathode mix structure and, thus, inevitably alter it.

4.2 DEVELOPMENT STUDIES

4.2.1 A-Size AZ-21 Magnesium m-Dinitrobenzene Cells

Tests with two different Columbian carbon blacks showed little loss in cell capacity after three months storage at 113°F and 95 per cent R.H. Cells made with CBN P-1100-P2142 HT 3232 average $29\frac{1}{2}$ hours at $16\frac{2}{3}$ ohms, or 3 per cent less than initial-capacity results. Cells made with CBN HR 1670-P3317 averaged $26\frac{1}{2}$ hours at $16\frac{2}{3}$ ohms with no loss in capacity from initial results. Previous tests with CBN P-1100-CL 20212 showed a 15 per cent loss in capacity after mix storage of three months.

Other cell studies were limited to evaluation of the various experimental carbon blacks in AZ-10 A-cells. The data are summarized in Table 4-2.

4.2.2 Shelf Studies

A-size AZ-10 Mg cells made with Columbian carbon black P-1100-CL 20212 were stored under two different conditions for various periods, and then discharged continuously through 16-2/3 ohms to an end voltage of 0.9 volt. Corresponding lots of cathode mix were tightly sealed in glass jars for various storage periods and utilized in A-cells just before capacity tests. The following capacity data are average of 10 cells used in each test.

After cell storage at 70°F and 50 per cent R.H. for 12 and 18 months, the capacities dropped to 25 and 22.5 hours, yielding a capacity retention of 76 per cent and 68 per cent, respectively, of fresh cells. Cells freshly prepared from cathode mix stored 18 months gave 29.5 hours, which is a capacity retention of 89 per cent. After cell storage at 113°F and 95 per cent R.H. for 12 and 18 months, the capacities were 21.5 and 12.5 hours, respectively, indicating a capacity retention of 70.3 per cent and 41 per cent. Cells freshly prepared from cathode mix stored 3 and 18 months at 113°F in a sealed jar gave 25.5 and 25 hours at a capacity retention of about 83 per cent.

The large capacity loss from cells stored at 113°F is believed to result from seal failure and consequent escape of water. This same defect is responsible for the major portion of lost capacity in the cells stored at 70°F because freshly made cells with comparably stored cathode mix retained so much more of the original capacity. Examination of the magnesium cans

of discharged cells stored at 70°F showed practically no corrosion, as evidenced by retention of the bright pickle surface. The magnesium anodes of cells stored at 113°F had dark brownish films and slightly more corrosion than the cells stored at 70°F. The film resulted from slight reduction of soluble m-dinitrobenzene on the magnesium surface. The quantity of water required for magnesium corrosion and slight m-DNB reduction observed in these cells is not sufficient to account for the large loss of capacity after the 18 months of storage. Capacity of these cells on storage tests is limited, therefore, by water loss through the seal. The importance of magnesium-cell sealing must be emphasized in future storage programs.

5. APPENDIX

5.1 THE SPECTROPHOTOMETRIC DETERMINATION OF m-DNB IN 2N MAGNESIUM PERCHLORATE SOLUTIONS.

5.1.1 Scope

The procedure covers the range of 0.5 to 15 mg m-DNB/liter of 2N $\text{Mg}(\text{ClO}_4)_2$.

5.1.2 Theory

The ultra-violet absorption of m-DNB at 264 mμ is used as a quantitative measure of concentration in 2N $\text{Mg}(\text{ClO}_4)_2$ in the range 0.5 to 15 mg m-DNB/liter. Dilution with 2N $\text{Mg}(\text{ClO}_4)_2$ is used for concentrations greater than 15 mg m-DNB/liter.

5.1.3 Apparatus

A Beckman DU Spectrophotometer with hydrogen lamp source was used.

5.1.4 Reagents

The 2N $\text{Mg}(\text{ClO}_4)_2$ solution contains 331.3 grams of $\text{Mg}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ per liter; any residue is filtered off.

5.1.5 Procedure

- a. Avoid contact of the sample solution with the bottle cap. Swirl

the sample, and allow about one minute for any carbon black particles to settle.

- b. Warm the Beckman DU Spectrophotometer and the hydrogen lamp. Fix the sensitivity at its center of travel (5 turns from either end); fix the wavelength at 264 mμ.
- c. Match two 1-cm quartz cells containing 2N $\text{Mg}(\text{ClO}_4)_2$ solution to within 0.5% transmittance. The slit will read about .29 to .30 mm at the fixed sensitivity setting.
- d. Triple-rinse a 2-ml pipet with the supernatant solution, and pipet 2 ml into a dry, 100-ml volumetric flask rinsed with a few ml of 2N $\text{Mg}(\text{ClO}_4)_2$. Make up to volume with 2N $\text{Mg}(\text{ClO}_4)_2$, and mix well.
- e. Triple-rinse the sample cell with the volumetric solution. Read and record the absorbance (A_1).
- f. Use the table below to determine the final dilution of the sample.

A_1 RANGE	FINAL DILUTION BY VOLUME (Sample: 2N $\text{Mg}(\text{ClO}_4)_2$)
above 0.900	1:100
.400 to .399	use A_1 value obtained in step 5.
.200 to .399	3:100
.130 to .199	5:100
.028 to .129	10:100
.013 to .027	25:50
below .013	use original sample as is

g. When necessary, repeat steps 4 and 5 at the final dilution.

5.1.6 Calculations

mg m-DNB/liter 2N $\text{Mg}(\text{ClO}_4)_2 = A_1 \times 14.7$ dilution factor

where: A_1 = absorbance at the final dilution

14.7 = slope of calibration curve

5.1.7 Preparation of Calibration Curve

- a. Prepare solutions containing 1, 4, 8, and 15 mg of m-DNB per liter of 2N MgClO_4 .
- b. Measure the absorbance of the solutions as described under procedure.
- c. Plot the absorbance values on the y-axis of linear graph paper; plot mg m-DNB/liter 2N $\text{Mg}(\text{ClO}_4)_2$ on the x-axis.
- d. Draw a straight line through the points plotted.
- e. Calculate the slope of the curve.

5.2 SUMMARY OF TEST DATA FOR CARBON BLACKS

5.2.1 Columbian Carbon Co., Princeton, New Jersey

TYPE OF CARBON BLACK	SORPTOMETER N ₂ AREA m ² /gm	AREA BY I ₂ SORPTION m ² /gm	LINSEED OIL ABSORPTION gal/100 lb
<u>CBN HR_1670-</u>			
-Control	1520	940	31
-3386	1680	1150	45
-3303	1600	850	26
-3379	-	800	26
-3360	1320	725	21
-3381	1430	475	16
<u>CBN 10S67-</u>			
-Control	-	54	46
-3398	-	-	-
-3387	607	390	46
-3399	639	532	-
<u>CBN P-1100-</u>	670	659	34
-3385	670	565	31

CBN P-1100-8417 ASH DATA

SAMPLE NO	% ASH
-1	0.16
-2	0.041
-3	0.053
-4	0.52

5.2.2 Atlas Powder Co., Wilmington, Delaware

Darco G-60: Vegetable origin (wood charcoal) activated with heat and steam, and washed with strong mineral acid and water

Chloride (as NaCl)	less than 0.05%
Iron (as Fe_2O_3)	less than 0.05%
pH	5.0 to 7.0
Moisture	10%
Water-Solubles	0.3% max.
Acid-Solubles	0.9% max.
Ash	4.0% max.
Bulk Density (d = 0.4, i.e. 13 times that of CEN P-1100- CL 20212)	22 to 28 lb/ft ³
Mesh Size:	
through 100 mesh screen	95%
through 325 mesh screen	65-80%
Filterability	Excellent
Wettability	Excellent

5.2.3 G. L. Cabot, Inc., Boston, Massachusetts

<u>Monarch 74</u>	
Bulk Density	12.5 lb/ft ³ (d=0.2)
Surface Area	300 m ² /gram

<u>Vulcan XC-72R (formerly Battery Black XB-72F)</u>	
Electrolyte Sorption	3.2 ml/gram
Ash Content	0.1%
Area:	
N ₂ Adsorption	182 m ² /gram
Electron Microscope	84 m ² /gram
Apparent Density (d=0.06 to 0.1)	4 to 6.25 lb/ft ³

5.2.4 Shawinigen Chemicals, Ltd., Shawinigan Falls, Quebec

<u>50% Compresses</u>	
Surface Area	
N ₂	64.5 m ² /gram
Electron Microscope	70 m ² /gram
Apparent Density (d=0.1)	6.25 lb/ft ³
pH	7-8
% moisture	0.04%
% volatile	0.06%
% ash	0.04%

6. CONCLUSIONS

A method was developed to determine the ability of carbon blacks to absorb m-dinitrobenzene from aqueous salt solutions.

Studies of the adsorption properties of various Columbian and commercially available carbon blacks of other manufacturers showed wide variation in adsorption properties.

A correlation has been shown between a coulombic test and the amount of m-dinitrobenzene adsorbed by the carbon black.

Capacity data of A-size $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/\text{m-dinitrobenzene}$ cells did not correlate directly with the carbon black adsorption property. It is believed that the lack of complete correlation is due to changes in the cathode-mix structure which result from compression, material transfer, and phase inversion. The continuous liquid electrolyte phase of fresh cathode mix is reduced to a tenuous capillary film at some stage of discharge in the tightly packed A-cell.

Capacity Tests of A-size AZ-10 magnesium cells made with Columbian P-1100-CL 20212 carbon black and discharged after 18 to 19 months storage showed a capacity retention of 68% at 70°C and 41% at 113°F. The loss in capacity in the A-cell storage results mainly from water loss through the cell seal.

Capacity tests of A-size AZ-21 magnesium cells made with Columbian P-1100-P2142 HT 3232 and HT 1670-P3317 carbon blacks and discharged after three months storage showed no significant loss in capacity.

7. PROGRAM FOR THE NEXT QUARTER

1. Complete adsorption studies for various carbon blacks.
2. Continue wet-cell-capacity tests for various carbon blacks, and correlate data with A-cell test results.
3. Determine the effect of physical changes in the nitro-organic-carbon black cathode on cell performance.
4. Initiate developmental studies with the better Columbian carbon blacks.

8. KEY PERSONNEL

8.1 IDENTIFICATION

Biographies of most of the key personnel were presented in the previous report. Those given below pertain to persons who have recently joined the program.

MARY LOUISE KUETTEL, Chemical Technician, received a B. A. degree in Chemistry from the University of Delaware, Newark, Delaware, in 1960. Before joining RCA, Miss Kuettel was employed as a laboratory technician by Jersey Testing Laboratories, Inc., Newark, New Jersey.

From September, 1960, until May, 1961, Miss Kuettel was a part-time geology-laboratory employee of the University of Delaware, where she was engaged in graduate study in geology.

Miss Kuettel is a member of the American Chemical Society. She has been with Electrochemical Group since October, 1961.

DONALD G. TREVETHAN, Chemist, was graduated from the University of Pennsylvania in 1960 with a B. A. Degree in Chemistry. He has taken postgraduate work in chemistry at Temple University, Philadelphia.

Mr. Trevethan was employed from 1950 until 1952 as a biochemistry laboratory technician at the University of Pennsylvania, where he worked on the preparation of enzymes. From 1952 to 1954, he worked with the Franklin Institute, and per-

formed experiments to determine the ignition limits of various mixtures of carbon disulfide and oxygen.

From 1954 until 1958, Mr. Trevethan served as a nuclear technician with the U. S. Air Force, and specialized in the arming and fusing systems of special weapons. During the following three years, he was employed by the Philco Corporation in Philadelphia where he was engaged in research, fabrication, and testing of solar cells, fuel cells, and specialized electrochemical systems.

Mr. Trevethan joined the Electrochemical Group in September, 1961.

8.2 DISTRIBUTION OF HOURS

G. S. Lozier, Project Director	98
J. B. Eisen, Physical Chemist	493
R. J. Ryan, Physical Chemist	124
D. G. Trevethan, Physical Chemist.	96
M. Kuettel, Technician	303
A. Lindabery, Technician	202
Miscellaneous Analytical Personnel	75
Total	1391

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