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Quarterly Report No. 2

DEVELOPMENT OF THE DRY TAPE BATTERY CONCEPT

1 May to 1 August 1964

by

B. A. Gruber, J. J. Byrne, R. Kafesjian, K. W. Klunder,
E. A. McElhill, W. H. Power, and T. J. Wolanski

Prepared for:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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MONSANTO RESEARCH CORPORATION
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FOREWORD

In a meeting held with the Lewis Technical Manager on 21 July 1964 at Lewis Research Center, Cleveland, Ohio, it was decided that future work would be directed away from the use of block anodes. This decision necessitated a small change in the planned technical program on anode structures which may not be adequately reflected in the discussions contained in this report. This new planning will be treated more fully in subsequent reports.

SUMMARY

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Research on the development of an operating dry tape unit has continued to be concentrated on improving the performance of cathodic discharge characteristics on static and moving tapes; on evaluating incapsulation materials, techniques, and evaporation rate of incapsulated water and electrolytes; and on designing an operating tape unit.

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PHASE I. SINGLE COMPONENT CONFIGURATION DEVELOPMENT

The energy output obtained from the nitro compounds has been increased greatly during this report period as techniques for preparing the tape electrode were improved and operating conditions were optimized.

Picric acid was the most promising nitro compound depolarizer tested both because of its high energy capacity and because of its current efficiency at high current drains. For example, picric acid discharged with 62% efficiency and 572 watt-hr/lb energy production at 100 ma/in.^{2} in 2M $Mg(ClO_4)_2$ and with up to 50% efficiency in 2M $AlCl_3$ at 500 ma/in.^{2}. Under these conditions, picric acid was superior to the other nitro compounds tested.

The highest energy outputs of nitro compound depolarizers were obtained under the following conditions:

Depolarizer:	Picric acid
Depolarizer Loading:	Optimized for magnitude of current drain
Substrate Carbon:	Shawinigan Acetylene Black, 50% compressed
Carbon/Depolarizer Ratio:	1/1
Electrolyte:	2M $AlCl_3$
Anode:	Expanded Mg foil

The coulombic efficiency of the m-DNB/Mg couple in 2M $MgCl_2$ increased from 8 to 25% as the particle size of the m-DNB was reduced from 200μ to 48μ. Further particle size reduction produced no further improvement in efficiency.

For weight minimization, low substrate ratios are advantageous but, for efficient reduction of the nitro compounds, ratios in the range of 35 to 50% substrate will probably be required.

Shawinigan acetylene black, 50% compressed, was the most suitable carbon substrate found.

For aromatic nitro compounds, the best single neutral electrolyte found was 2M $Mg(ClO_4)_2$, and the best single electrolyte was 2M $AlCl_3$. Some advantage of $Mg(ClO_4)_2$ over $AlCl_3$ lies in the lower corrosion rate of Mg in $Mg(ClO_4)_2$. Electrolyte mixtures of $AlCl_3$ and $Mg(ClO_4)_2$, providing better electrode wetting than $AlCl_3$ alone, were used to improve the reduction efficiency of tapes with high loadings.

Organic acids were incorporated directly into the cathode coatings to supply acid at the reaction site without causing corrosion of the Mg anode.

Flexible and mechanically stable coatings of depolarizers on Dynel tapes were made by incorporating short Dynel fibers in the cathode mixture. Shrinking and cracking of the dried electrode was eliminated by the fibers. The discharge reproducibility was greatly improved by the presence of the fibers in the cathode, and some increase in reduction efficiency of picric acid was obtained on the improved electrode. The coating consisted of acetylene black/depolarizer (usually 1/1) mixed with about 6% Dynel fibers and bonded to the separator with a water-soluble adhesive (11% polyvinylpyrrolidone) that also acted as an efficient wetting agent.

PHASE II. DRY TAPE DESIGN

Power spraying, investigated as the first of several possibilities for tape manufacture, has produced the highest quality tapes to date. Further sophistication of the equipment is planned.

Plans are being made to evaluate extrusion as a manufacturing method with subsequent rolling or grinding to insure a uniformly flat surface.

Extensive testing of moving tapes on a breadboard device built for this purpose has provided valuable information on the design requirements of an operating tape unit. As a result of breadboard testing,

information has been obtained regarding electrolyte wet-out data, collector head design, anode configuration, cathode-tape physical characteristics, and tape feed. Most runs have been qualitative, and improvements, where appropriate, have been made following each experiment. Future plans call for an expanded capability to provide improved tapes and a more sophisticated testing set-up to permit further evaluation of limiting factors.

PHASE III. CONVERSION DEVICE DEVELOPMENT

A portable tape drive unit, complete with a dummy tape, was devised by modifying a commercial magnetic tape recorder. The device was operated with an external dc power source applied to a small dc motor. The moving incapsulation was punctured, the contents released, and the dummy tape wetted. Commercial dc motors are being tested for performance and efficiency for parasitic operation.

Magnesium alloy and polystyrene foam have been selected as lightweight structural materials for dry tape units.

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I. INTRODUCTION

Under Contract NAS3-2777, the feasibility of the Dry Tape Concept was demonstrated using a silver peroxide-coated porous polypropylene tape drawn between two current collectors. One of these current collectors was a zinc block that also served as an anode. The electrolyte was supplied by a second tape that contacted the silver peroxide coating.

The objectives of this continuation of that program are to develop the following:

1. Methods of efficiently utilizing high energy anodes and cathodes on tapes.

2. A method of incorporating high energy couples into a single tape configuration.

3. Methods of electrolyte incapsulation and tape activation.

4. A weight-optimized tape conversion device capable of supplying its own power for unattended start-stop operation.

5. Methods of supplying multiple cell voltages from the dry tape system.

During the first quarter, expanded magnesium, giving a current density of 1000 ma/in.^{2} at a polarization of 0.2 v, was tentatively selected as the best anode for combined tape construction. Anode development was concentrated on the use of magnesium foil, pressed powder, or sprayed metal forms. The preferred method of electrolyte supply was the continuous feed of macrocapsules of FEP Teflon, Aclar, or aluminum-Mylar laminate. Initial electrolyte payloads of greater than 80% were attained, and a 60% payload after three years' storage appeared to be possible. The organic oxidizer, 2,4,6-trichlorotriazinetrione (TCTT)* was tentatively selected as the cathode active material for the complete tape system because of its high discharge rate. Parasitic operation of a breadboard dry tape unit was achieved, with the drive motor consuming about 10% of the power output with a 15% motor efficiency.

This quarter's work included a continued search for high-energy cathode materials and the selection of optimum conditions for discharging with

*Monsanto Company's brand of 2,4,6-trichlorotriazinetrione, ACL-85®, is being used in this work. The compound will be referred to throughout this report as TCTT.

best possible energy efficiencies. In addition to the active material, experimental work was directed toward selecting the best depolarizer particle size, substrate carbon, carbon/depolarizer ratio, and the best electrolyte for efficient cathodic discharges. Cathodic reductions of depolarizers for evaluation purposes were made under both static conditions for the depolarizer/electrolyte/Mg cell and under dynamic conditions with a breadboard testing device. In addition to its use in evaluating cathode materials, the breadboard unit was used to obtain information on mechanical design requirements for the operating tape. A "first generation" tape drive unit was constructed to establish design concepts for the moving tape, the start-stop mechanisms, and the parasitic drive unit. Techniques of incapsulating and releasing electrolyte from the moving tape were developed, and the permeability of water vapor through the sealed plastic capsules was evaluated.

II. PHASE I. SINGLE COMPONENT CONFIGURATION DEVELOPMENT

A. HIGH ENERGY CATHODE DEVELOPMENT

1. Background

The recent interest in organic nitro compounds as electrochemical oxidants (depolarizers) stems from the fact that nitro compounds, because of their low equivalent weight, have theoretical energy densities far higher than those of inorganic cathodes. For example, the theoretical energy density of the m-dinitrobenzene (m-DNB)-Mg couple is estimated to be 855 watt-hr/lb of reactants, including the 8 moles of water that are consumed with every mole of m-DNB:

$$C_6H_4(NO_2)_2 + 6Mg + 8H_2O = C_6H_4(NH_2)_2 + 6Mg(OH)_2$$

$$\Delta F^\circ = 740 \text{ kcal/mole}; \; E^\circ = 2.67 \text{ v}; \; \text{Energy Density} = 855 \text{ watt-hr/lb}^*$$

The nitro compounds are known to be reduced with low efficiencies at the higher discharge rates in dry cell bobbins. However, we expect that the compact tape configuration will give improved discharge characteristics.

The development program during this period was designed to optimize the energy output at the highest current drains possible. Most of these tests were run in a simple static cell to define the necessary conditions for optimum energy output.

2. Method

a. Experimental Procedure

The simple plastic sandwich cell holder (Figure 2, First Quarterly Report) with a tape area of 3 in.^{2} and a grooved back on the anode side was used for testing under static conditions. The cell was not submerged in electrolyte. The separator and the charged tapes were prewet with electrolyte, and the test cell was placed on its back with the Mg anode on top. Voltages of the full cell Mg/electrolyte/nitro compound were measured. The Mg anode polarized 0.12 v at 100 ma/in.^{2} and 0.14 v at 500 ma/in.^{2} in 2M $Mg(ClO_4)_2$ when tested in a submerged cell (First Quarterly Report). Under similar test conditions therefore, full-cell failure up to 500 ma/in.^{2} will be caused by cathode failure (iR losses of full cell are also included in full-cell measurements). The current collector for the cathode was either a carbon sheet or a platinum foil.

*Note: Energy density is higher in acid electrolytes.

b. Tape Preparation (Static Tests)

The cathode electrode consisted of a mixture of carbon, depolarizer and (in the improved method) thin fibers bonded to the surface of a nonwoven fabric by a water-soluble binder. The dry components were blended by mixing them lightly with a glass mortar and pestle until no depolarizer was visible. An aqueous solution of binder was added to the dry components and the slurry mixed until homogeneous. (About 25 ml of water volume was required for 4 g of 50/50 depolarizer/carbon mix) The slurry was loaded on the tape and leveled to the desired thickness with a Gardner blade. The test samples were 3 in. by 1 in. (3 in.^{2}) cathode coats deposited on a Dynel, Nylon or polypropylene tape. (Nylon could not be used in strong acid because it was readily attacked by acid electrolyte.) Most of the tapes were allowed to dry at room temperature, but many of those containing o-DNB (which is stable and has a high melting point) were dried at 85°C.

The various binders, tapes, and fibrous fillers studied are listed in Table A-1 in the Appendix. The formulation for each tape that was tested is given in Table A-2 in the Appendix. Static cell tests (Table 1) indicated that none of the following candidate binders interfered with the electrochemical output of the various cathode-Mg couples. (Polyvinylacetate-polyvinyl alcohol binder was not stable in acid electrolytes.)

3. Development of Mechanically Stable, Coated Tapes

The organic depolarizers do not conduct and therefore must be mixed with a conducting substrate on the electrode. The method of binding silver peroxide to the tape fabric that we developed last year gave electrodes with these depolarizer-carbon mixtures that could be tested in the static cell but that had poor mechanical stability.

Most cathode mixtures consisted of a 1:1 ratio of depolarizer:Shawinigan acetylene black, 50% compressed. This carbon adsorbs a large amount of water. The coatings on the tapes, especially the thicker ones, shrank on drying and developed cracks. These cracks filled in well when rewetted, and this type coated tape was used in the early static cell tests. (The water-soluble binders used also acted as effective wetting agents for the nitro compound/Shawinigan acetylene black coatings.)

The addition of short (1/8- or 1/4in.) fibers to the cathode mixture eliminated the shrinking and cracking of the electrodes on drying. The coated tapes containing about 6% by weight 1/4- or 1/8-in., 3-denier Dynel fiber or fine graphite fibers were flexible and mechanically stable. The bond of the whole coating to the tape fabric (with 11% polyvinylpyrrolidine) was not always strong, but the coating was self-supporting.

Table 1

EFFECT OF BINDERS ON STATIC CELL OUTPUT

Test Cathode System: 50/50 wt. ratio depolarizer/ acetylene black
Electrolyte: 2M $AlCl_3$
Base Tape: 3 mil Dynel
Current Density: 100 ma/in.^{2}

Depolarizer Type	mg/in^2	Binder	Coulombic Efficiency to 0.8 volt, %
o-DNB	80	None (pressed powder)	50-55
o-DNB	74	PVP, 5 wt-%	50-60
o-DNB	70	Gelvatol 20-30, 5 wt-%	50-58
o-DNB	62	Gantrez AN-139, 5 wt-%	35-50
o-DNB	80	Methocel HG, 5 wt-%	30-50
Picric Acid	83	None (pressed powder)	55-60
Picric Acid	79	PVP (5 wt-%) + Dynel Fibers (1/4 in. & 1/8 in., 5 wt-%)	50-60
Picric Acid	68	Cyanamer P-26 (5 wt-%) + Dynel (1/8 in., 5 wt-%)	45-50

Incorporating fibers in the electrodes greatly improved their reproducibility on discharge and made possible the testing of the electrodes under dynamic conditions.

Also, some increase in the reduction efficiency of picric acid was obtained on the improved electrode. However, no difference in activity was found for the reduction of 2,5-dinitrothiophene on the two types of electrodes. This indicates that test results with the basic electrode without fibers are valid where the level of activity of the total system is limited by some process other than the physical characterizatics of the cathode. The increase in cathodic discharge efficiency found for the reduction of picric acid with the improved fiber electrode is illustrated in Table 2.

4. Optimization of Cell Conditions for High Energy Output of Nitro Compounds on Tape Configurations

a. Comparison of Different Nitro Compounds as Depolarizers

Nitro compounds with the highest theoretical coulombic capacities were chosen for testing. Compounds with different structural and physical characteristics were included. Previous work by us (ref. 1) and others (ref. 2) with nitro compounds as cathode materials in dry-cell bobbin configurations was used as a basis for choosing these compounds. The range of different types of compounds was kept broad, however, since the compound yielding the highest energy output on the tape configuration may not be the same as that giving the highest output in dry cell bobbins. These theoretical coulombic capacities of the organic nitro compounds that were tested as depolarizers are given in Table 3.

The discharge of picric acid on the improved fiber-type electrode in 2M $AlCl_3$ at different current densities is shown in Figure 1. The tape loadings of these electrodes are not optimized. (The area enclosed by the dotted lines of Figure 1 shows the variations in discharge at 500 ma/in.^{2} on a range of tape loadings.) A reduction efficiency of 95% was obtained at a very low current drain (16 ma/in.^{2}) in 2M $AlCl_3$ for a sample of picric acid catalyzed by Pd.

The potential drops steadily on discharging picric acid in the static test. Discharging the moving tape will enable us to obtain a constant energy output from this system. o-Dinitrobenzene was tested most extensively. Its energy output was improved during the report period as the cell variables were optimized. The best tests for o-DNB on the simple (no fiber) electrode did not give as high energy outputs as those of picric acid. It is expected that energy output would be increased somewhat by use of the fiber-type electrode (see Section A-3).

Table 2

EFFECT OF CATHODE FIBER CONTENT ON CATHODIC REDUCTION EFFICIENCIES OF PICRIC ACID

Cathode: Shawinigan Black:Picric Acid (1:1)

Anode: Magnesium

Electrolyte: 2M $AlCl_3$

Current Density: 500 ma/in.^{2}

Cathode Mixture Type	Cathode Coulombic Efficiency*, to 0.8v, %
No fiber	20-35
Dynel fiber	38-42
Graphite fiber	41-46

*At equivalent tape loadings

Table 3

COULOMBIC CAPACITIES OF ORGANIC DEPOLARIZERS

Compound	Theoretical Coulombic Efficiency (amp-min/g)	Special Characteristics
2,4,5-Trinitrotoluene	128	Very low sol. H_2O
Picric acid	125	Slight water sol.
1-Carboxymethyl-3,3,5,5-tetranitro piperidine	120	$-NO_2$ groups on saturated carbons
o-Dinitrobenzene	115	-
2,5-Dinitrothiophene 2,4-Dinitrothiophene	111 111	The nitro group in the 2,5-isomer is more active due to resonance interactions
3,6-Dinitrophthalic acid	76	Soluble in water

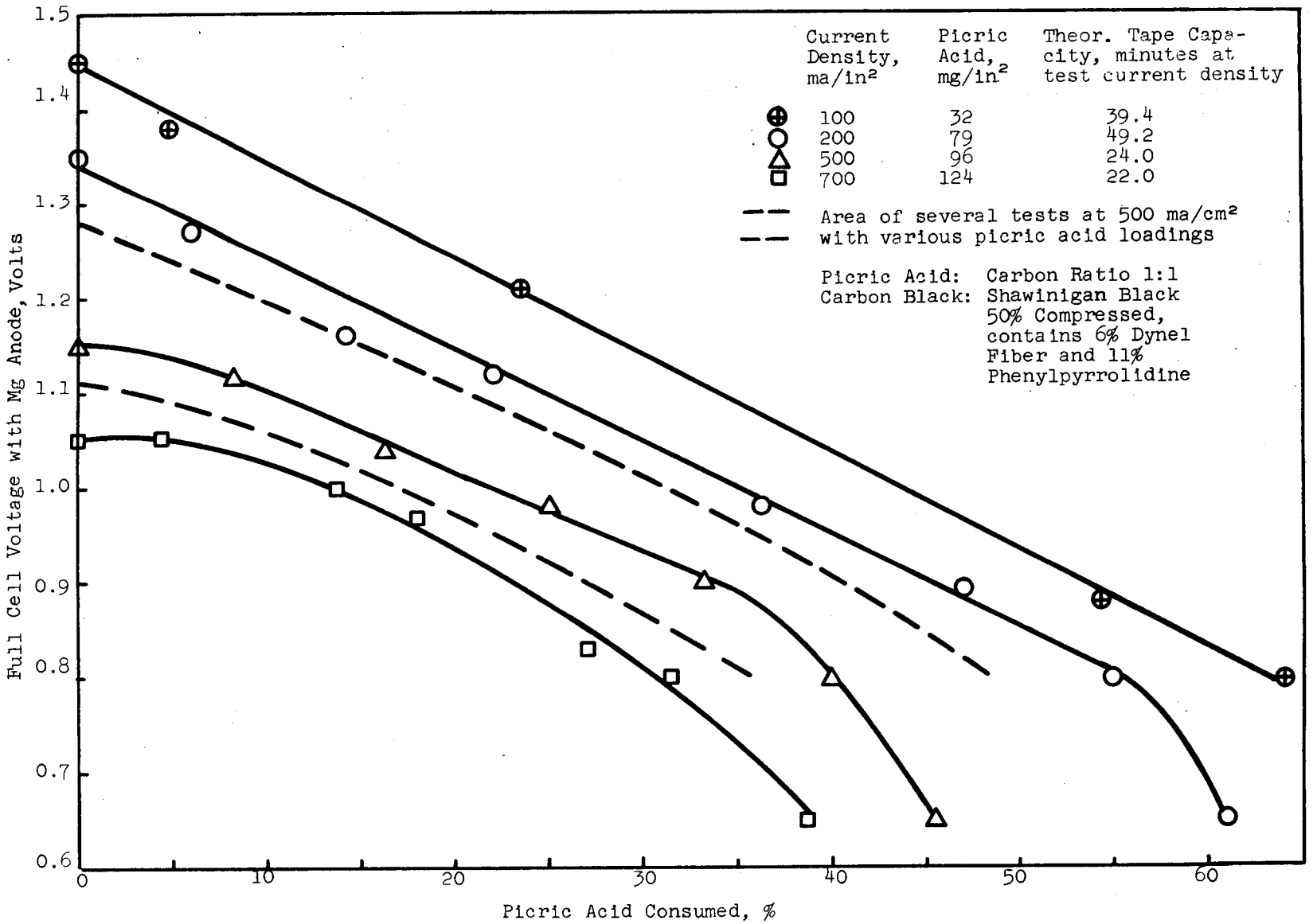


Figure 1. Static Discharge of Picric Acid in 2M $AlCl_3$ at Different Current Densities

The cathodic discharges of these compounds under different conditions of tape loading, electrolyte type, current density, and catalytic treatment are subsequently given in a series of figures and tables. Of all the nitro compounds tested, picric acid was the most promising depolarizer both because of its high energy capacity and its better current efficiency. Picric acid was the best when discharged at 100 ma/cm^2 in neutral electrolyte (Figures 2 and 3). These characteristics were somewhat better than those of o-DNB. Picric acid was also best in efficiency and energy output at 500 ma/in.^{2} in acidic electrolyte (Figure 4). Under these conditions, picric acid was markedly better than o-DNB and the other nitro compounds that were tested, as shown in Table 4. Energy capacities of the cell Mg/2M $AlCl_3$/picric acid at current densities from 16 to 700 ma/in.^{2} are given in Table 5.

2,4,5-Trinitrotoluene (TNT) would not maintain 0.8 v vs Mg at 500 ma/in.^{2} in 2M $AlCl_3$ and was reduced at the lowest potentials of all the depolarizers in 2M $Mg(ClO_4)_2$ at 100 ma/in.^{2} as shown in Figure 2. The energy output of TNT should be improved by using a smaller particle size (see Section A.4b).

1-Carboxymethyl-3,3,5,5-tetranitropiperidine was almost completely inactive in the acid electrolyte, 2M $AlCl_3$. Its discharge in 2M $Mg(ClO_4)_2$ was flat, but both reduction efficiency and discharge potential were low, as shown in Figure 3.

2,5-Dinitrothiophene was discharged at higher potentials than the 2,4- isomer (see Figures 3 and 4) as would be expected from its structure (the nitro group in the 5- position is made more reactive to reduction by the electron withdrawing effect of the nitro group in the 2- position). Both isomers had flat discharges in 2M $Mg(ClO_4)_2$ at 100 ma/in.^{2}, but efficiencies were low. The 2,4- isomer did not maintain 0.8 v vs Mg in 2M $AlCl_3$ at 500 ma/in.^{2}.

2,5-Dinitrothiophene was checked on fiber-type electrode configurations. Efficiencies of reduction were the same on both type electrodes in 2M $Mg(ClO_4)_2$ at 100 ma/in.^{2} (13%) and in 2M $AlCl_3$ at 100 ma/in.^{2} (38%), indicating that the depolarizer activity and not the limits of the electrode configurations were measured on both type electrodes.

3,6-Dinitrophthalic acid has the advantage of having two equivalents of acid in its molecule. It is also very soluble in water. It had a high potential in the early part of the discharge at 100 ma/in.^{2} in 2M $Mg(ClO_4)_2$ (Figure 3), but did not maintain higher current drains well. Its theoretical capacity is low compared with that of picric acid and it appears to offer no advantage to compensate for this.

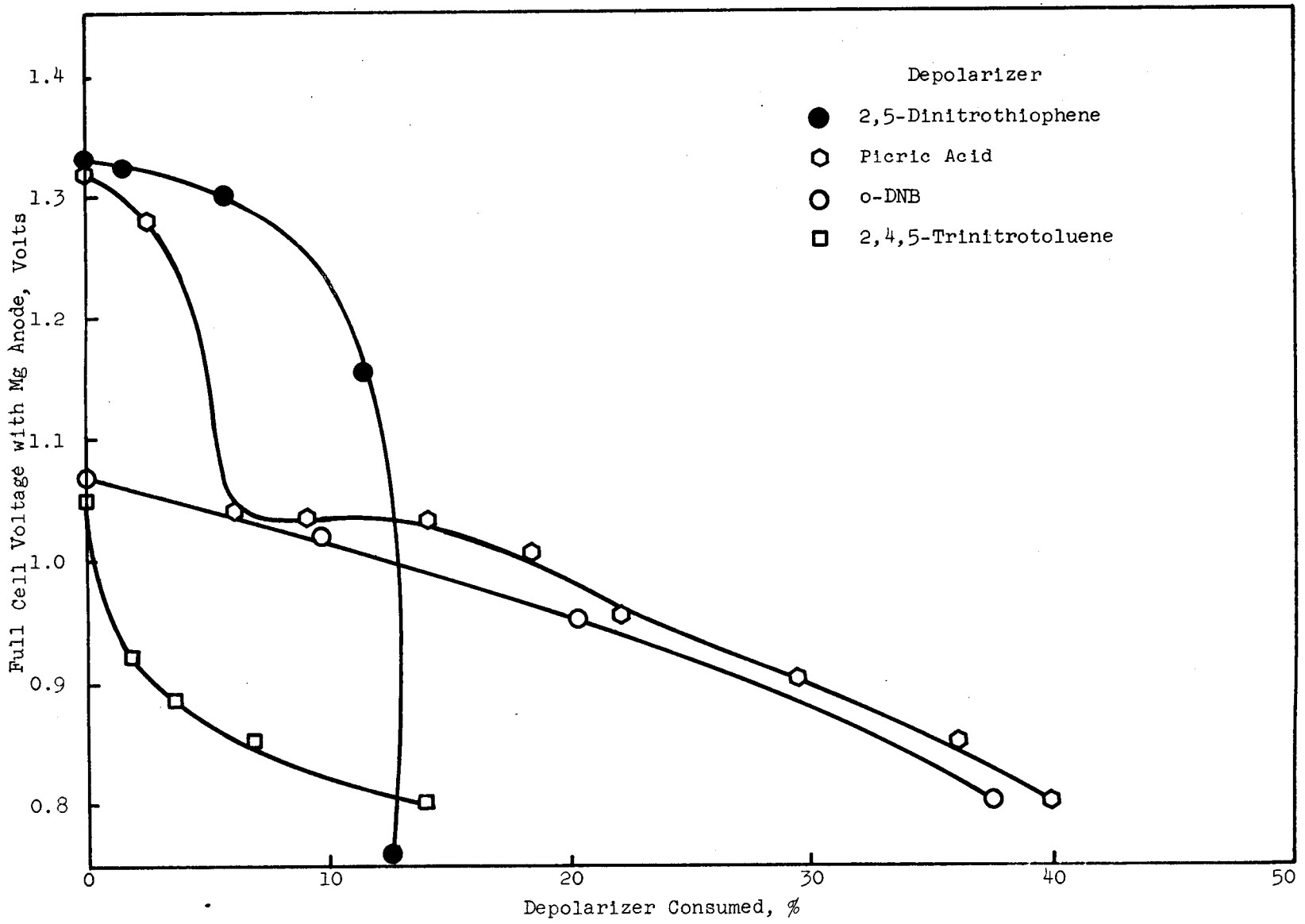


Figure 2. Static Discharges of Various Depolarizers in 2M $Mg(ClO_4)_2$ at 100 ma/in.^{2} (Part 1)

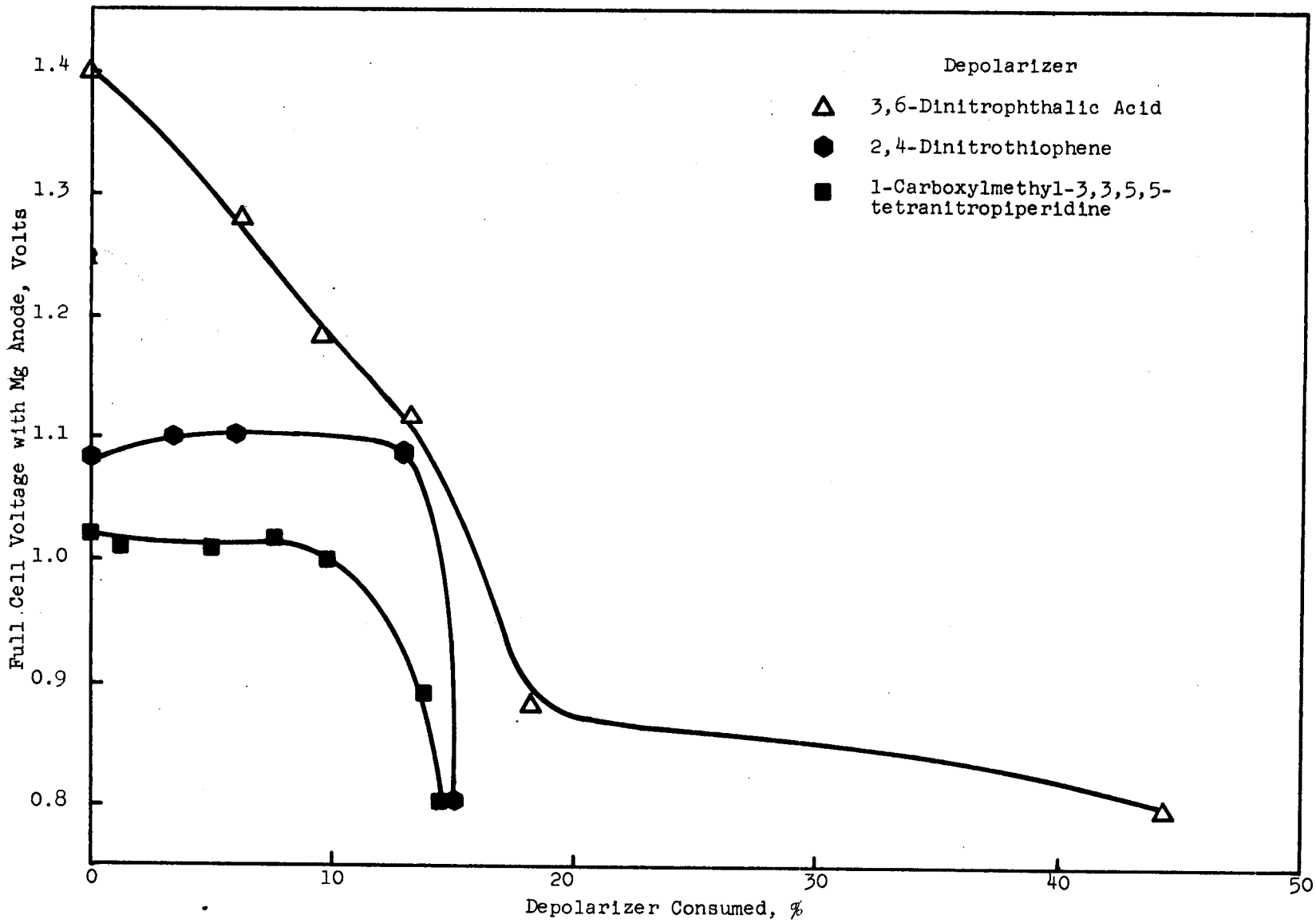


Figure 3. Static Discharge of Various Depolarizers in 2M $Mg(ClO_4)_2$ at 100 ma/in.^{2} (Part 2)

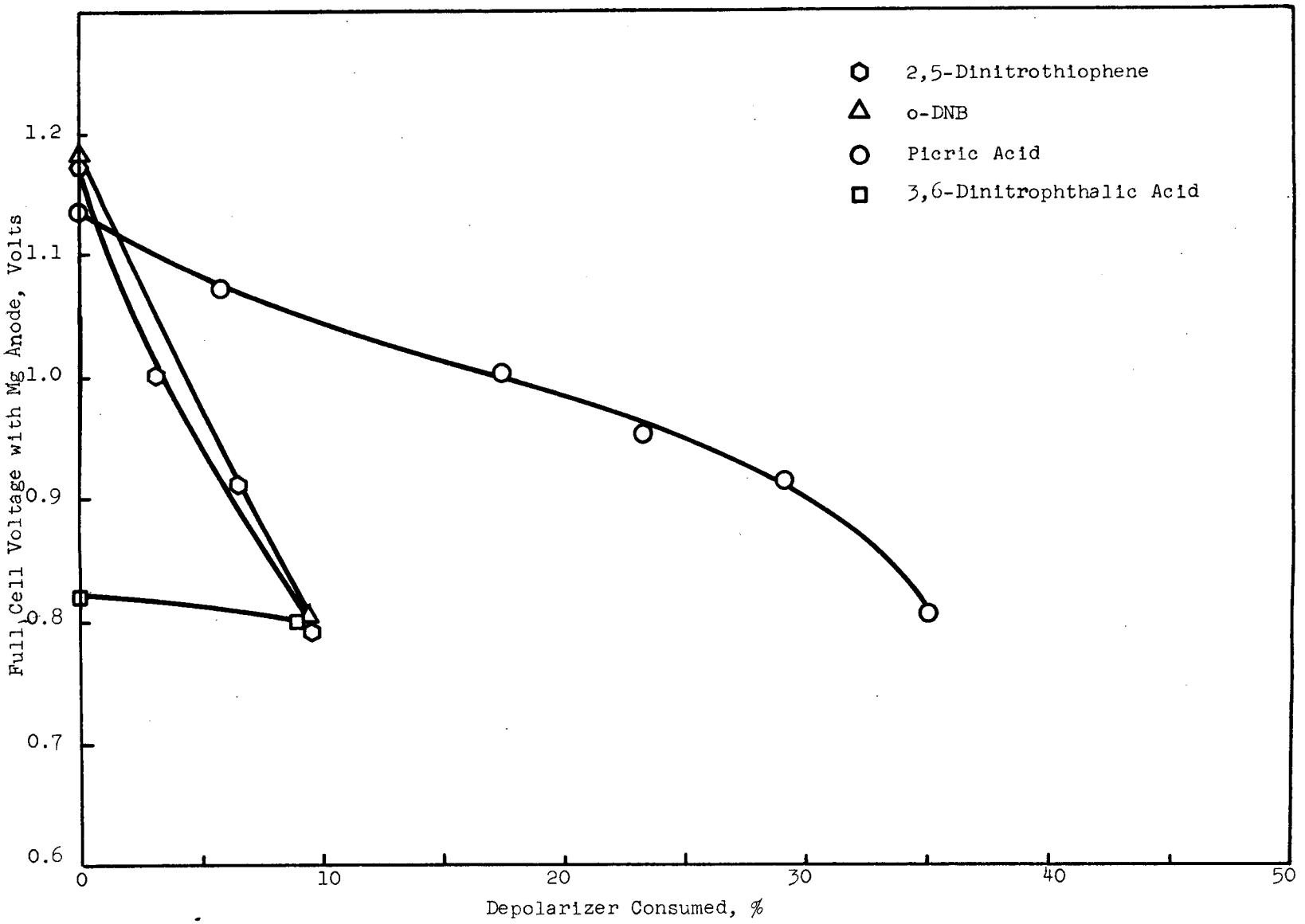


Figure 4. Static Discharges of Various Depolarizers in 2M $AlCl_3$ at 500 ma/in.^{2}

Table 4

CATHODIC DISCHARGE TESTS FOR PICRIC ACID AND o-DNB REDUCTION

Cell: Mg/Electrolyte/Nitro Compound

Cut-Off Voltage: 0.8v

Depolarizer	Current Density ma/in.^{2}	Electrolyte	Average Discharge Voltage, v	Depolarizer Efficiency,%	Depolarizer Energy Capacity, watt-hr/lb	Energy Capacity of Tape, watt-min/in.^{2}
Picric Acid	700	2M $AlCl_3$	0.95	19	170	3.3
Picric Acid	500	2M $AlCl_3$	1.00	35	330	3.0
o-Dinitrobenzene	500	2M $AlCl_3$	0.85	25	192	2.1
o-Dinitrobenzene	200	2M $AlCl_3$	1.0	62	540	7.5
Picric Acid	500	2M $Mg(ClO_4)_2$	0.85	14	112	2.1
Picric Acid	300	2M $Mg(ClO_4)_2$	0.95	42	378	2.9
Picric Acid	100	2M $Mg(ClO_4)_2$	1.03	40	389	3.2
o-Dinitrobenzene	300	2M $Mg(ClO_4)_2$	0.85	9.6	71	1.0
o-Dinitrobenzene	100	2M $Mg(ClO_4)_2$	0.93	49	396	2.1

Table 5

DISCHARGE TESTS OF Mg-PICRIC ACID CELL

Electrolyte: 2M $AlCl_3$

Current Density, ma/in.^{2}	Average Discharge Voltage, v	Total Tape and Coating Weight, mg/in.^{2}	Cathode Efficiency, %	Energy Capacity of Total Tape, watt-min/in.^{2}	Energy Capacity of Picric Acid watt-hr/lb
16 ‡	1.20	130	95	6.0	1070
100 †	1.10	330	85	8.05	887 (444)*
200	1.10	210	61	6.17	637
500 †	0.95	320	50	3.56	450 (225)*
500	1.05	120	42	2.10	417
700	1.00	270	33	3.51	310

* Based on weight of picric acid plus succinic acid.

‡ Contained 1.2% Pd Catalyst

† Contained succinic acid on tape.

b. Particle Size of Depolarizer

The coulombic efficiency of the m-DNB/Mg couple in 2M $MgCl_2$ increased from 8 to 25% as the particle size of the m-DNB was reduced from 200 to 48μ, respectively.

Reduction of particle size of o-DNB to 48μ, however, did not increase reduction efficiency. Particle sizes of other depolarizers (except TNT) were similar to that of o-DNB. The particle size of TNT was slightly larger. Since TNT has very low solubility in water, its cathodic discharge may be improved by particle size reduction. However, no further tests with TNT are planned because of the superior high energy output of picric acid.

c. Electrolytes

Our previous work (ref. 1) with m-DNB as a cathode material included a study of various neutral electrolytes. No single physical property of the neutral electrolytes determines its effectiveness but rather a combination as listed below:

(1) pH.

(2) Solubility and pH of base produced on reaction of electrolyte with hydroxide ion.

(3) Solubility of nitro compound in electrolyte.

(4) Effectiveness of electrolyte to salt-in the nitro compound onto the substrate carbon.

We have searched for the following types of electrolyte systems for use with the tape configurations:

(1) The best neutral electrolytes for m-DNB,

(2) Acid electrolytes which are compatible with the Mg anode at least for short periods of time,

(3) Solid acids with limited solubility for incorporation on the tape and use in combination with an aqueous electrolyte, and

(4) Mixed electrolytes with advantages over single electrolytes.

Most of the comparisons were made with o-DNB. A tabulation of o-DNB cathode efficiencies when discharged in various electrolytes is given in Table 6. The best single neutral electrolyte found was 2M $Mg(ClO_4)_2$, and the best single electrolyte was 2M $AlCl_3$. These electrolytes were therefore used in subsequent tests where other variables affecting discharge characteristics of the nitro compounds were studied.

Table 6

EFFECT OF ELECTROLYTE ON CATHODIC

DISCHARGE EFFICIENCY AT 100 ma/in^2

Cell: Mg/Electrolyte/o-DNB

Electrolyte	Carbon:Depolarizer Ratio	Reduction Efficiency to 0.8 v
Acid Salts		
2M $AlCl_3$*	50:50	62 (at 200 ma/in^2)
2M $Th(NO_3)_4$	50:50	29
Neutral Salts		
2M $Mg(ClO_4)_2$	50:50	20-49
2M $Mg(ClO_4)_2$	33:67	15,22
8M NH_4SCN	50:50	29
4M NH_4SCN	33:67	13,20
2M NH_4SCN	33:67	13, 18
3M $Mg(ClO_4)_2$	50:50	19, 15
1M $MgBr_2$	50:50	17-25
1.5M NH_4ClO_4	50:50	18
1M $MgBr_2$ saturated with $Mg(OH)_2$	50:50	9,12

* $FeCl_3$, $ZnCl_2$ and $AlCl_3$ + NH_4Cl mixture proved too corrosive to the Mg anode for use.

The electrolyte studies were conducted with the basic (no fiber) type tape cathodes. Where only slight variations in cathode performance were observed, the order of effectiveness of the electrolytes is not defined clearly. However, the considerable advantage of 2M $AlCl_3$ over other electrolytes tested is evident. Its superiority as an electrolyte has also been confirmed in tests with picric acid on the improved (fiber type) tapes.

The advantage of 2M $Mg(ClO_4)_2$ electrolyte over 2M $AlCl_3$ lies in the lower corrosion rate of Mg in $Mg(ClO_4)_2$. Cathodic discharges are better in $AlCl_3$, but Mg corrodes in this highly acidic salt which hydrolyzes in water to give HCl.

The selection of the best electrolyte for the dry tape system will be a compromise between cathode performance and anode attack.

Two combinations of mixed electrolytes were tested with picric acid. A mixture of $Mg(ClO_4)_2$ with $AlCl_3$ was used to improve the ease of wetting of the electrodes. (Electrodes were wet more readily in 2M $Mg(ClO_4)_2$ than in 2M $AlCl_3$.) The mixed electrolyte, 1.6M $AlCl_3$ and 0.4M $Mg(ClO_4)_2$ improved the reduction efficiency of o-DNB on tapes with high loadings as shown in Table 7. These tapes have extremely high loadings of depolarizer, and the effectiveness of wetting the electrode is most critical. (Note that the reduction efficiency of o-DNB in 2M $AlCl_3$ at lower loadings at 200 ma/in.^{2} is 62% (see Table 4). We will retest this electrolyte combination in the final system with picric acid.

A mixture of 2M $AlCl_3$ and 1M Li_2CrO_4 was tested as an electrolyte (Li_2CrO_4 is an inhibitor for Mg attack). However, the chromate was reduced at more positive potentials than the nitro compounds, o-DNB and picric acid. In addition, either the chromate or its reduction products inhibited discharge of the nitro compounds even after the chromate adsorbed on the electrode was consumed, as shown by Table 8.

Organic acids were incorporated directly into the cathode coating to supply acid at the reaction site without causing corrosion of the Mg anode. Initial attempts to improve discharge in neutral electrolytes of o-DNB by incorporation of oxalic acid in the tape were not successful. However, incorporation of solid acid (equal weights succinic acid with picric acid) in the new type fiber electrode gave increases in both discharge potential and reduction efficiency in 2M $AlCl_3$ as shown in Figure 5 for low current densities. At 500 ma/in.^{2}, (Figure 6) improvement in reduction efficiency was obtained in 2M $AlCl_3$. It is

[Note: The plots represent data assuming no loss of reagents on fabrication of electrode (both picric acid and succinic acid have slight solubility in water. A tape made in the same way as those used in the cell tests was analyzed to contain 89.3% of the succinic acid charged. Correction of efficiencies for this loss gives 47.8% efficiency at 500 ma/in.^{2} and 75.3% at 100 ma/in.^{2}).]

Table 7

EFFECT OF ELECTROLYTE ON CATHODIC DISCHARGE EFFICIENCY AT 200 MA/CM^2

Cell: Mg/Electrolyte/o-DNB

Electrolyte	o-DNB g/in.^{2}	Reduction Efficiency to 0.8 v, %
2M $Mg(ClO_4)_2$	0.125	26.4
1.6M $AlCl_3$; 0.4M $Mg(ClO_4)_2$	0.128 0.129	49.9 49.5
2M $AlCl_3$	0.111	33.0

Table 8

CELL DISCHARGES IN 2M $AlCl_3$ + 1M Li_2CrO_4

Cathode Material	Current Density ma/in.^{2}	Efficiency of Reduction Based on Cathode Material only, %	Amp-min to 0.8 v vs Mg
None	100	-	2.5
Picric Acid	500	20*	2.5
Picric Acid	100	54	3.2
o-Dinitrobenzene	500	20	2.5

*Normal value in 2M $AlCl_3$ is 40%

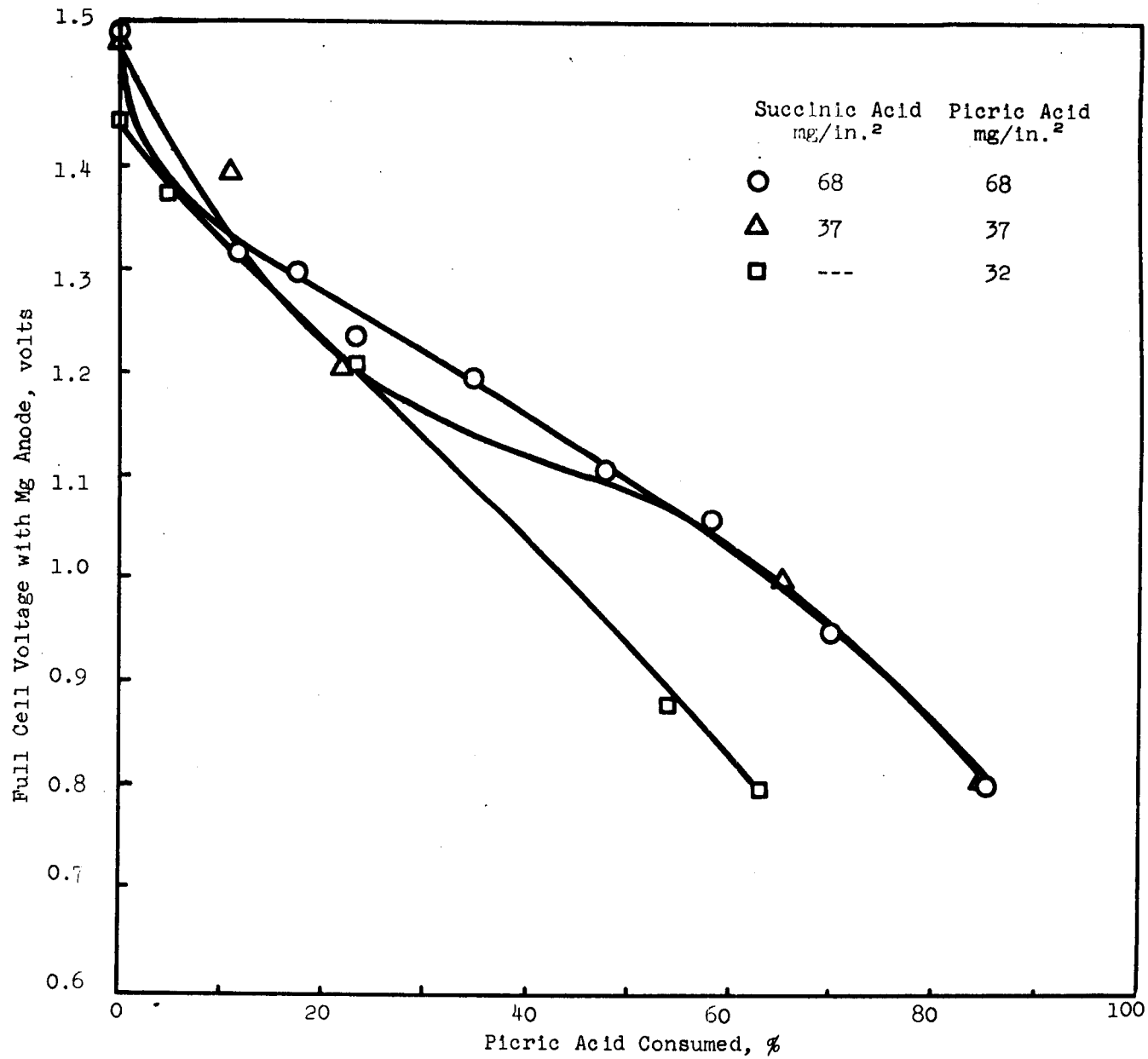


Figure 5. Effect of Succinic Acid on Static Discharge Characteristics of Picric Acid in 2M $AlCl_3$ at 100 ma/in.²

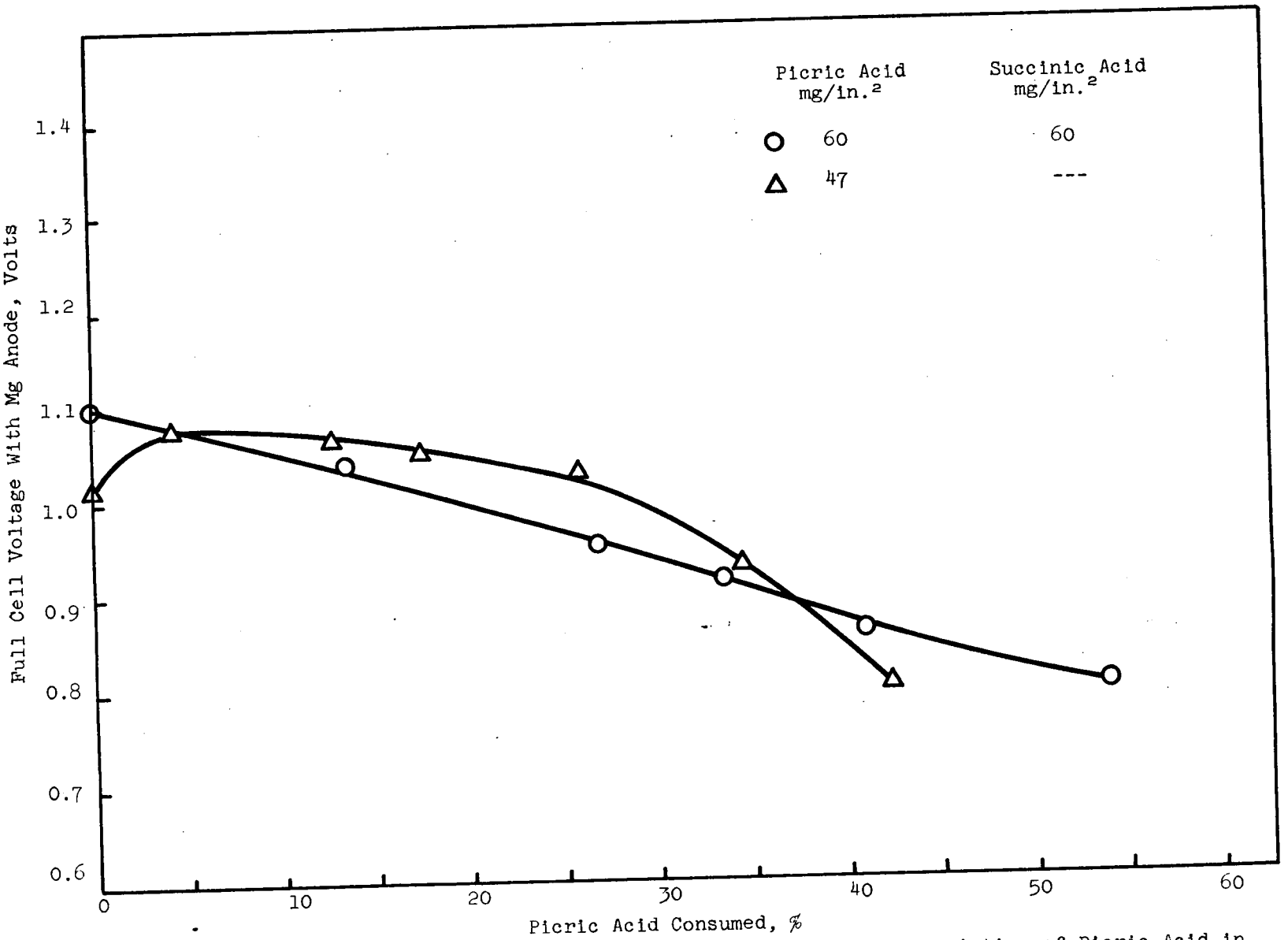


Figure 6. Effect of Succinic Acid Content on Static Discharge Characteristics of Picric Acid in 2M $AlCl_3$ at 500 ma/in.²

possible that an acid with slightly higher water solubility might be more effective at higher current drains. Lower loadings of acid will also be tested since the high loadings may give excessive IR loss due to lower conductivity caused by the organic acid.

d. Carbon Substrates

(1) Carbon Type

The proper carbon substrate is required for efficient discharge of the nitro compounds. It is not known specifically what properties of the carbon determine its suitability; high surface area, high adsorption of both depolarizer and water, and good conductivity are all necessary but are apparently not the only prerequisites.

Several different types of carbons were tested with o-DNB. Shawinigan acetylene black, 50% compressed, as used in dry cell bobbins with organic depolarizers, was the most suitable. Data in Table 9 illustrate this. Also, 50% compressed Shawinigan black is superior to the 100% compressed black at higher current drains as shown in Table 10. The 100% compressed black has a lower water adsorption capacity.

(2) Carbon: Depolarizer Ratio

For weight minimization, low substrate contents are advantageous, of course, but for efficient reduction of the nitro compounds, ratios in the range of 35 to 50% substrate will probably be required. At low current drains smaller amounts of substrate are required. With the basic type (no fiber) electrodes, efficiencies of reduction of three loadings of o-DNB on carbon substrates are given in Table 11.

Tests in 1M $MgBr_2$ with 1:1 and 3:1 ratios of C:o-DNB showed slightly higher efficiency for the 3:1 ratio. Efficiencies of the fiber type electrode are also affected by the carbon as shown in Table 10.

(3) Depolarizer Loading

High loadings of o-DNB and picric acid were necessary to maintain high current drains (e.g., 500 ma/in.^{2}) on the first basic tapes. At low current densities, reduction efficiencies were slightly lower with high depolarizer loadings.

Much higher current drains can be maintained with low loadings on the improved fiber tapes. For example, a picric acid tape containing only 18 mg/in.^{2} of picric acid was reduced with 47.5% efficiency at 500 ma/in.^{2} in 2M $AlCl_3$. The efficiency of reduction of tapes with low loadings was higher than that of those with high loadings, as shown by Figure 7, but the average discharge potential of the tapes with high loadings was higher in the initial part of the discharge.

Table 9

COMPARISON OF EFFECTIVENESS OF VARIOUS CARBONS AS SUBSTRATES FOR o-DNB

Cell: $Mg/2M\ Mg(ClO_4)_2$/o-DNB and Carbon Substrate or 2M $AlCl_3$

Carbon	Carbon: o-DNB Ratio	Cathode Efficiency to 0.8 v		
		2M $Mg(ClO_4)_2$ 100 ma/in.^{2}	2M $Mg(ClO_4)_2$	2M $AlCl_3$
Shawinigan, 50% compressed	1:1	21-49		62 (at 200 ma/in.^{2}) 35 (at 300 ma/in.^{2})
Shawinigan, 50% compressed	1:2	15,22	7 (300 ma/in.^{2})	22, 24 (at 500 ma/in.^{2})
Nuchar-CN	1:2		6 (200 ma/in.^{2})	17 (at 300 ma/in.^{2})
Pure Carbon FC-13	1:1	Does not hold		9 (at 100 ma/in.^{2})
Columbian Conductex-SC	1:1	12		15 (at 500 ma/in.^{2})
Asbury Graphite A625	1:1	1		5 (at 100 ma/in.^{2})

Table 10

EFFECT OF PICRIC ACID - CARBON RATIO ON CATHODIC REDUCTION EFFICIENCY

Cell: Mg/2M $AlCl_3$/o-DNB on Substrate
Cathodes: Fiber Type
Carbon: Shawinigan Black, 50% compressed

Carbon: Picric Acid Ratio	Cathodic Reduction Efficiency at 100 ma/in.^{2}, %	Cathodic Reduction Efficiency at 500 ma/in.^{2}, %
1:1	59.5	38, 42, 40
1:2	62.9	25, 28
1:1*	63.5	22, 27

* 100% Compressed Shawinigan Black

Table 11

EFFECT OF o-DNB - CARBON RATIO ON CATHODIC REDUCTION EFFICIENCY

Cell: Mg/2M $Mg(ClO_4)_2$/o-DNB on Substrate
Current Density: 100 ma/in.^{2}
Cathode Type: Basic (No Fiber)
Carbon: Shawinigan Black, 50% compressed

Carbon: o-DNB Ratio	Cathodic Reduction Efficiency, %
1:5	8-9
1:3	15-22
1:1	20-50

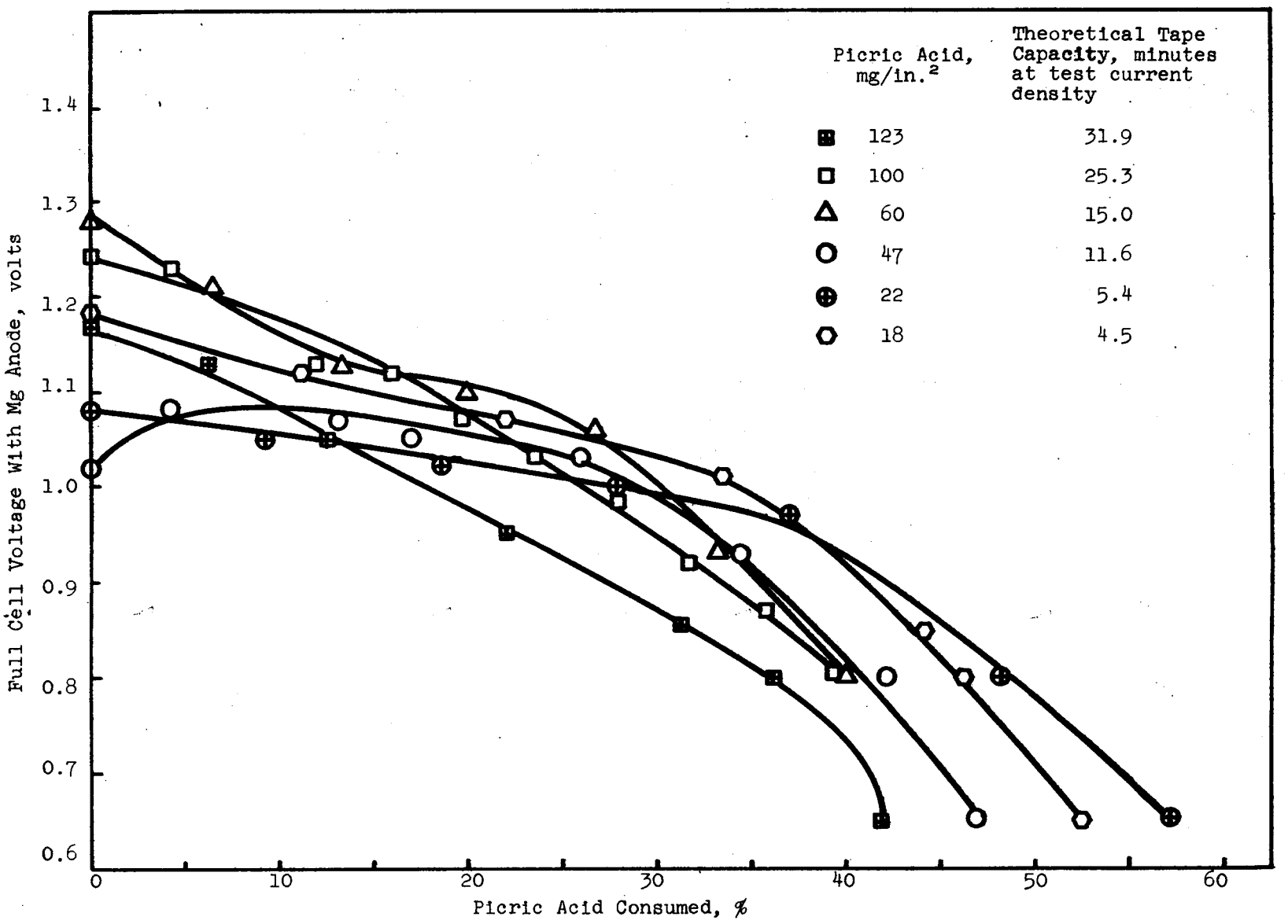


Figure 7. Effect of Picric Acid Loading on Static Discharge of Picric Acid in 2M $AlCl_3$ at 500 ma/in.^{2}

The same observations can be made about the discharge characteristics of picric acid in 2M $Mg(ClO_4)_2$ at 300 ma/in.^{2}, Figure 8. However, reduction efficiency did not vary as much. This may be a result of the superior wetting properties of the perchlorate electrolyte compared with those of $AlCl_3$, allowing high-load tapes to discharge at efficiencies comparable with lightly loaded tapes.

There is a large weight advantage to be gained by the use of heavy tape loadings in final tape system. At low tape loadings, the weight of the tape fabric for the total system is a high percentage of total weight as illustrated in Table 12. Therefore, we will attempt to improve the discharge efficiencies of the heavily loaded tapes by more effective wetting techniques. For example, higher loadings of binder (which is a wetting agent and has not adversely affected discharge of picric acid even at the high concentration of 29%) in the electrode and the use of the mixed electrolyte $AlCl_3$-$Mg(ClO_4)_2$ will be tried with high-load tapes.

One attempt to improve efficiency of a high-load tape by compressing the electrode (7000 lb/in.^{2}) resulted in a decrease in efficiency.

(4) Catalysts

Palladium appeared to catalyze the discharge of o-DNB in 2M $AlCl_3$. The best test result gave an increase of 0.2 v in the discharge potential of o-DNB at 200 ma/in.^{2} (Figure 9) for a 1.2% loading of Pd*. Catalysis was evident in a duplicate tape at low current densities but was ineffective at 500 ma/in.^{2}. This same cathode composition exhibited no catalytic effect in 2M $Mg(ClO_4)_2$. Another catalyst formulation, 2.5% Pd prepared by chemically reducing Pd on Shawinigan black by hydrazine, exhibited only a minor catalytic effect with o-DNB in 2M $AlCl_3$ (Figure 9). Similarly, small increases in discharge potential were obtained with picric acid as depolarizer. Palladium has been reported as a catalyst for m-DNB in sulfuric acid electrolyte (ref. 3).

All tests were run with the basic (no fiber) tape electrode. It is possible that mechanical factors were controlling the discharge and obscuring any catalytic effect. Also, the source of Pd catalyst may be critical. We will determine the effectiveness of Pd as catalyst on the improved fiber electrodes during the next period.

*5% Pd on carbon, Englehard Industries, Inc., Newark, New Jersey

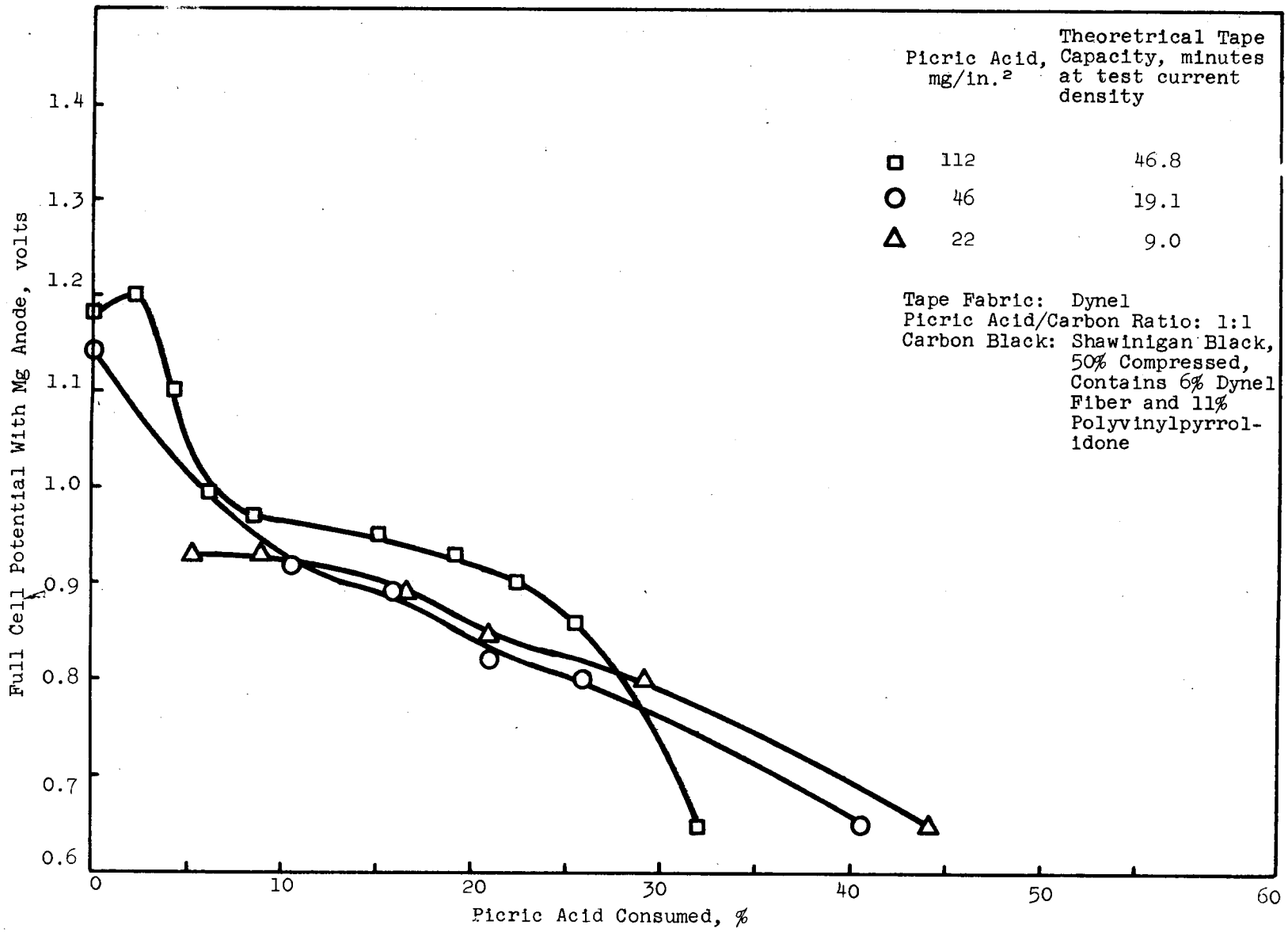


Figure 8. Effect of Picric Acid Loading on Static Discharge of Picric Acid in Mg $(ClO_4)_2$ at 300 ma/in.²

Table 12

WEIGHT DISTRIBUTION OF COATED TAPES

Carbon Depolarizer Ratio: 1:1

Tape Fabric: 3 mil Dynel

Picric Acid mg/in^2	Total weight coated tape mg/in^2	Total weight contributed by tape fabric %
18.0	70	35.6
21.6	80	31.2
46.7	140	17.8
60.0	180	13.8
99.7	270	8.9
123	330	7.5

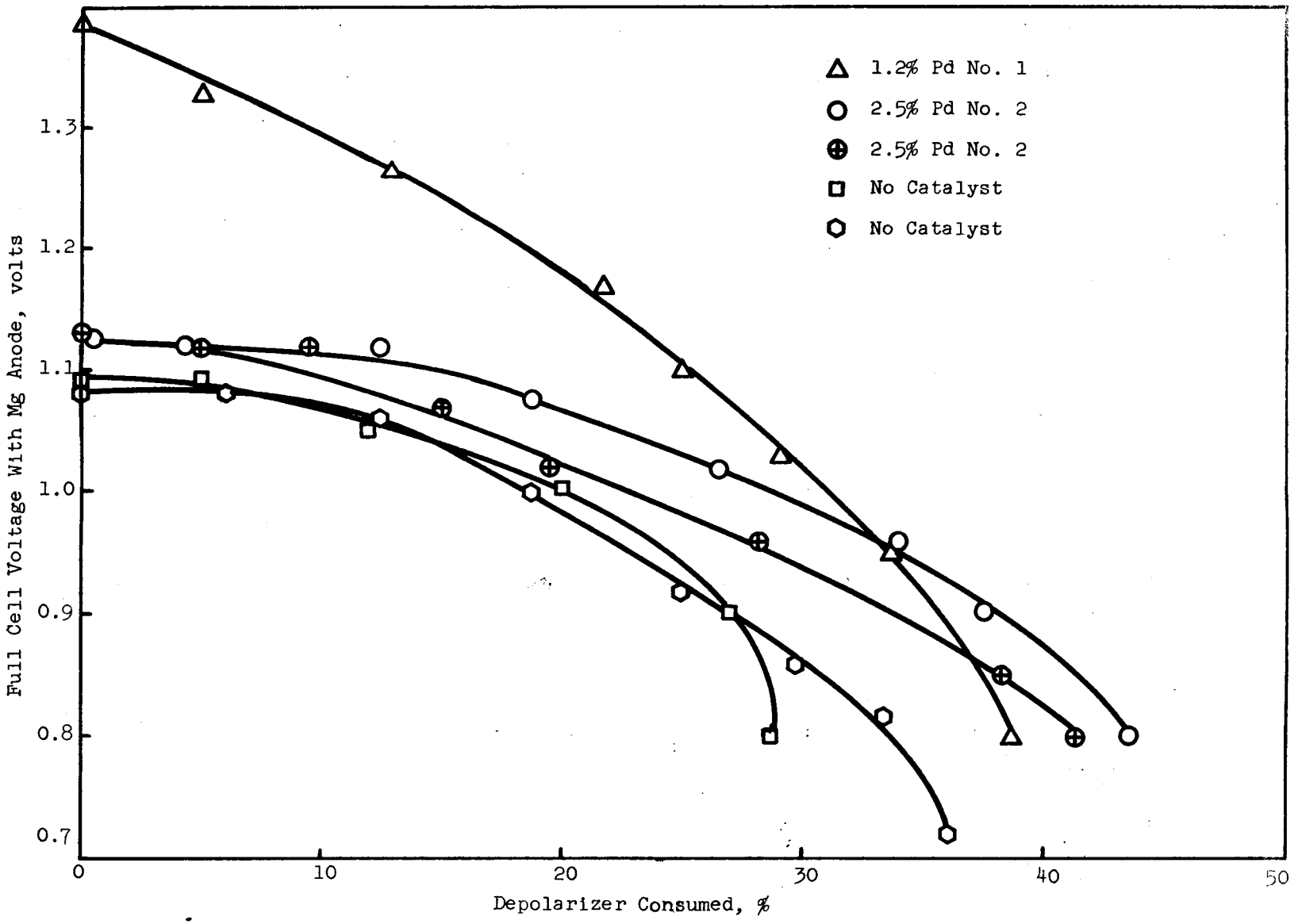


Figure 9. Effect of Catalysts on Static Discharge of o-DNB at Carbon Cathode in 2M $AlCl_3$ at 200 ma/in.^{2}

(5) Temperature

A series of o-DNB cathodic reduction tests on the basic type tapes was run at various temperatures to determine whether the reduction was limited kinetically. No improvement in discharge potential was obtained on increasing the temperature from 24°C to 40°C and then to 59°C in discharges at 100 ma/in.^{2} in 2M $Mg(ClO_4)_2$ (see Figure 10). Reduction efficiencies were slightly higher at the high temperatures (Table A-1, Appendix). Although these tests were run with the early tapes, coatings were thin, and it is thought that the conclusions drawn from both these data and data obtained on the effect of variation of substrate composition are still valid; that is, the discharge of nitro compounds is limited by complex mass transfer effects.

5. Future Plans (Cathode Development)

a. Optimize energy outputs of thick, heavily loaded tapes (e.g., by improving wetability).

b. Determine minimum electrolyte requirements for efficient discharge of picric acid tape electrodes.

c. Optimize energy output of the positive halogen compounds, TCTT, and hexachloromelamine.

B. ELECTROLYTE INCAPSULATION

1. Background

The operation of the dry tape battery requires the supply of a sufficient quantity of electrolyte to the discharge area for efficient discharge. During the previous quarter, various methods of supplying electrolyte in a storable form of minimum weight compatible with stop-start operation were considered. A macroincapsulation of dimensions suitable for supply with tape or on it offered a maximum in flexibility with a minimum weight. Design considerations, selection of thermoplastic films for incapsulation, and capsule fabrication were discussed in the first quarterly report and test results were presented on preliminary incapsulations. A payload greater than 60 wt-% after three years was estimated for the required incapsulation if a film permeability of the order of 0.002 g-mm/24 hr-sq in.-cm Hg could be obtained with a film thickness of about 2 mils.

This quarter's work has dealt primarily with testing of water and aqueous electrolyte incapsulations in thermoplastic films under various conditions. Methods of electrolyte release and dispensing have also been studied. Two methods of electrolyte release appear workable, one incorporating slitting of the packet, the other involving capsule puncture by a sharp-toothed sprocket. Dispensing devices incorporating these two methods of release have been built.

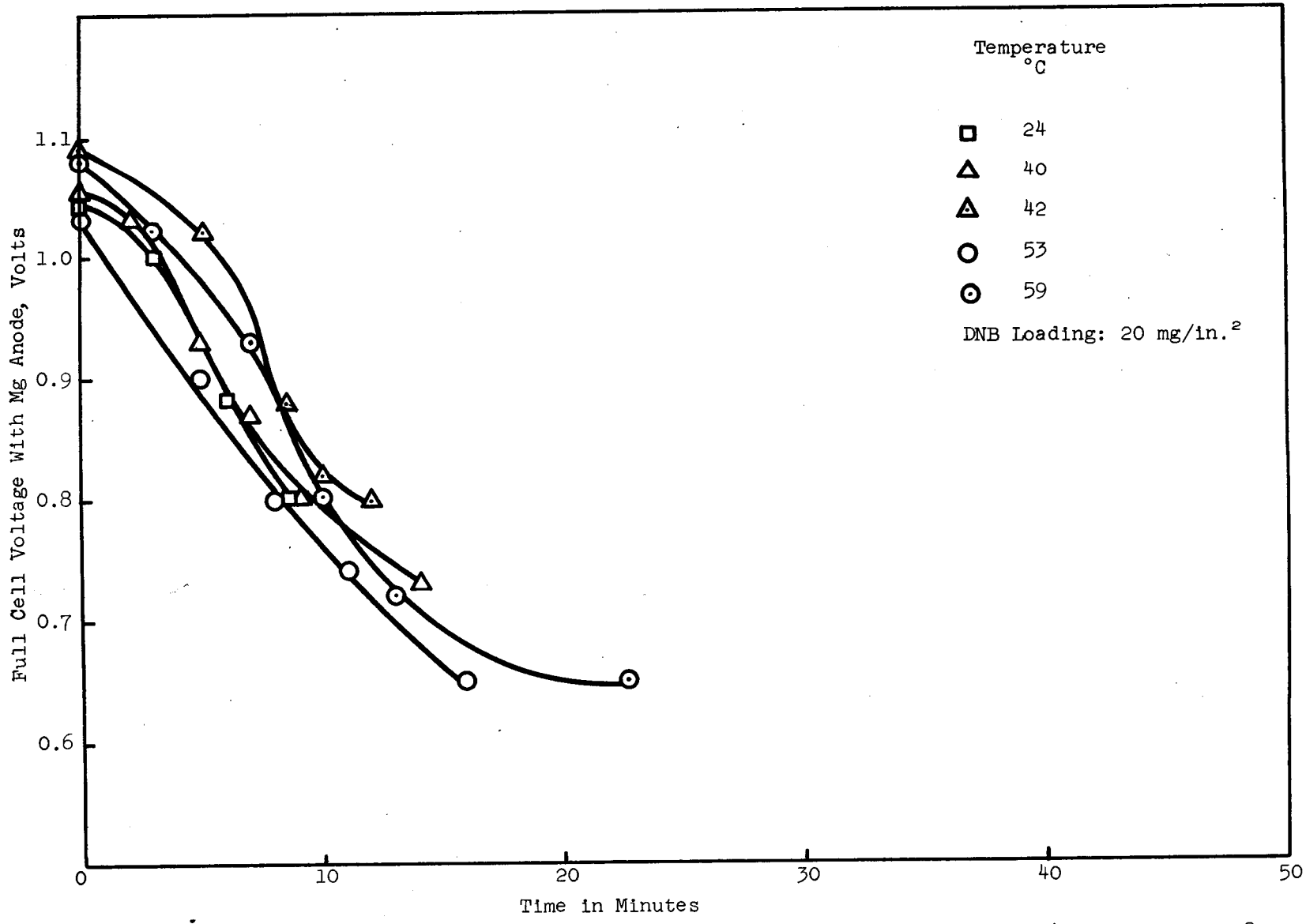


Figure 10. Effect of Temperature on Static Discharge of o-DNB at 2M Mg $(ClO_4)_2$, at 100 ma/in.^{2}

2. Capsule Fabrication

All capsules have been formed by thermal impulse sealing on our modified Sentinel Model 12-12AS laboratory sealer. Integral seals of good strength were obtained on all materials after the proper sealing conditions had been established. Both FEP and TFE types of Teflon sheet were sealed without undue problems. The TFE Teflon sheet* was supplied with one surface treated to make it heat bondable.

In forming incapsulations from film material, the film was first sealed down two sides and at one end to form a tube of the desired length. The tube was then filled with the desired liquid and heat sealed at one-inch intervals from the closed end to form a string of packets. When tubing was used as the starting material, the tube was sealed at one end, filled, and seals made at one-inch intervals.

A narrow heat seal is desirable to minimize reduction of capsule payload due to seals. The upper head of the heat sealing unit was redesigned to reduce the clamping width from one in. to 3/16-in. The seal width was also reduced by using 1/16-in. wide heating ribbon rather than the 1/8-in. ribbon originally supplied. Occasional failure of the thinner heating ribbon has been minimized by lowering the tension on the ribbon proportionately. A partial cross section of the modified sealing head is shown in Figure 11 with the heads in the open position.

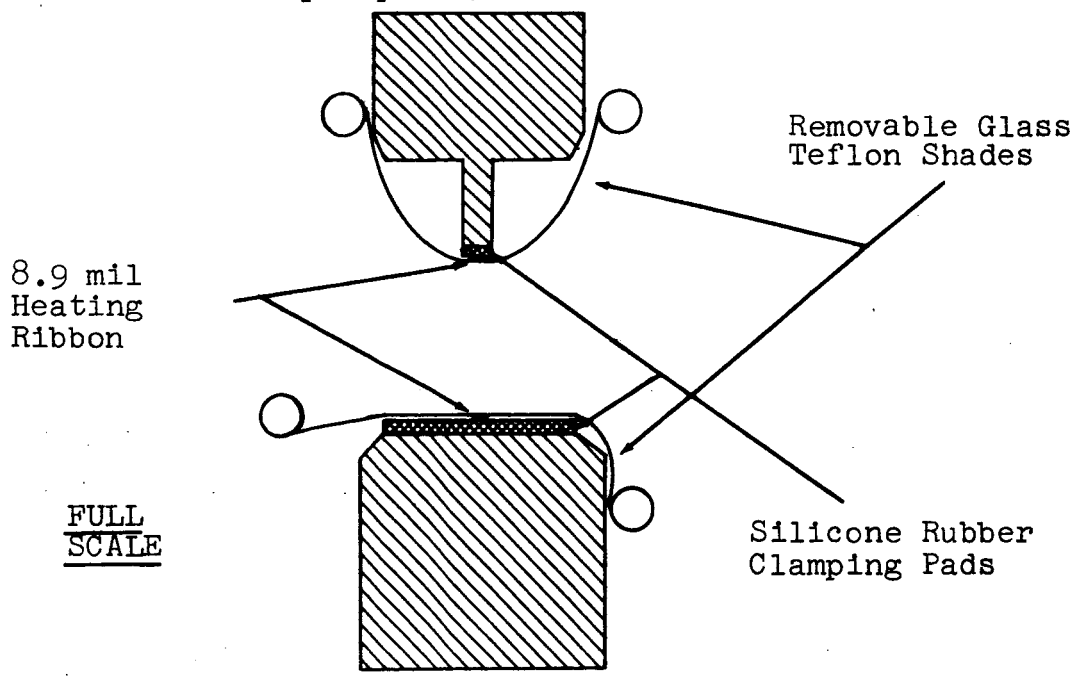


Figure 11. Cross Section of Modified Heat Sealer Head

*Dilectrix Corp., Farmingdale, L. I., N. Y.

The Sentinel Model 12-12AS sealer provides control of a wide range of sealing variables, allowing almost any thermoplastic film to be heat sealed. Jaw clamping pressure, clamping time, heat impulse time, and heater voltage can all be varied independently to determine the conditions required for a particular type of film.

Final incapsulation manufacture from the tentatively selected Kel-F 81 tubing can be readily accomplished in the lengths required with our present equipment. Finished incapsulation will be rapidly tested by short-term (2-3 days) measurement of weight loss in vacuum at 39°C. A defective incapsulation can be detected by this test.

3. Macrocapsule Design

An electrolyte requirement of 200 mg/in.^{2} of tape has been used previously as a basis for designing a macroincapsulation system. An incapsulation in Kel-F 81 thin-wall tubing has been tentatively selected as the best means of supplying electrolyte. To supply the quantity of electrolyte required, the tubing must be sized to the tape dimensions, loading, and number of tapes to be supplied. To cover the anticipated range of electrolyte requirements and to allow rapid estimation of payloads for design purposes, the volumetric capacity and payload of various tubing sizes should be known. The volumetric capacity of a tube is easily calculated from its dimensions. To allow for the reduction of this capacity due to heat seals, payloads were determined experimentally on finished packets made from several sizes of tubing. Using these data, a "squash factor" was calculated for each size tube where the "squash factor", β, is the ratio of the experimentally determined volume to the calculated volume of the tube. As would be expected, β decreases as the tube size increases, as shown in Figure 12.

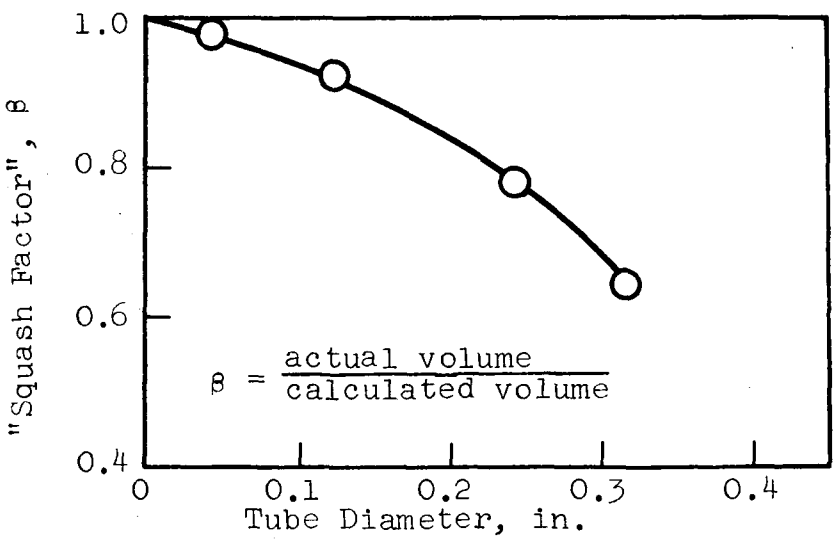


Figure 12. Reduction in Capacity of Kel-F 81 Tubing Due to Heat Seals

The squash factor will also vary with wall thickness and type of material, but these variations should not be important since a wall thickness and material selection has been made. The calculated payload and capacity for tubing covering the estimated range of electrolyte requirements is shown in Figure 13.* Experimental payloads are shown for comparison.

The effects of wall thickness and film density parameters on payload are readily apparent in Figure 13. Initial payloads and tubing sizes for a specified electrolyte requirement can readily be obtained by reference to Figures 12 and 13. Other factors needed to complete the incapsulation specification are film permeabilities (i.e., loss rates) and electrolyte release efficiency. Experimental results on loss rates of electrolytes from capsules under various conditions are detailed in Section B-6c. Preliminary work on electrolyte release indicates that at least 90% of the incapsulated electrolyte can be transferred to the tape.

Cathode tapes prepared to date have required from 0.15 to 0.3 ml/in.^{2} of electrolyte for good wet-out. It is hoped to reduce this to 0.1 to 0.2 ml/in.^{2} by some means of electrolyte conservation. Designs for multiple tape operation indicate the use of either two tapes or four tapes. It is anticipated that two tapes can be supplied with electrolyte efficiently from a single incapsulation. This would then require supplying about 0.2 to 0.4 ml/in.^{2} of incapsulation for a one-inch wide tape. Wider tapes will require a proportionately larger supply. Our tentative incapsulation selection will accomplish this supply with a payload greater than 80 wt-%.

4. Electrolyte Release

The electrolyte must be released from the incapsulation and distributed to the dry tape prior to the movement of the tape through the collector area. The time required for adequate distribution and wet-out will vary with the nature of the tape and electrolyte. Tapes with good discharge characteristics have had wet-out times of less than one minute. It has also been noted that electrolyte distribution is adequately achieved by the fibrous tape base material when the electrolyte is fed to the uncoated side of the cathode tape.

With these considerations in mind, release methods applicable to the tentative incapsulation selection of Kel-F tubing have been investigated. A number of the methods incorporated in a collector head are illustrated in Figure 14. Earlier tests on one method, slitting by a stainless steel blade followed by squeeze-rolling, were carried out with a rather flat packet of 2-mil polyethylene film. The test results were encouraging, showing both a low pulling force required and over 90% transfer of the incapsulated liquid to a dummy tape.

*In Table 1 of Quarterly Report No. 1, the diameter for the cylinder should be 130 mils rather than 65 mils.

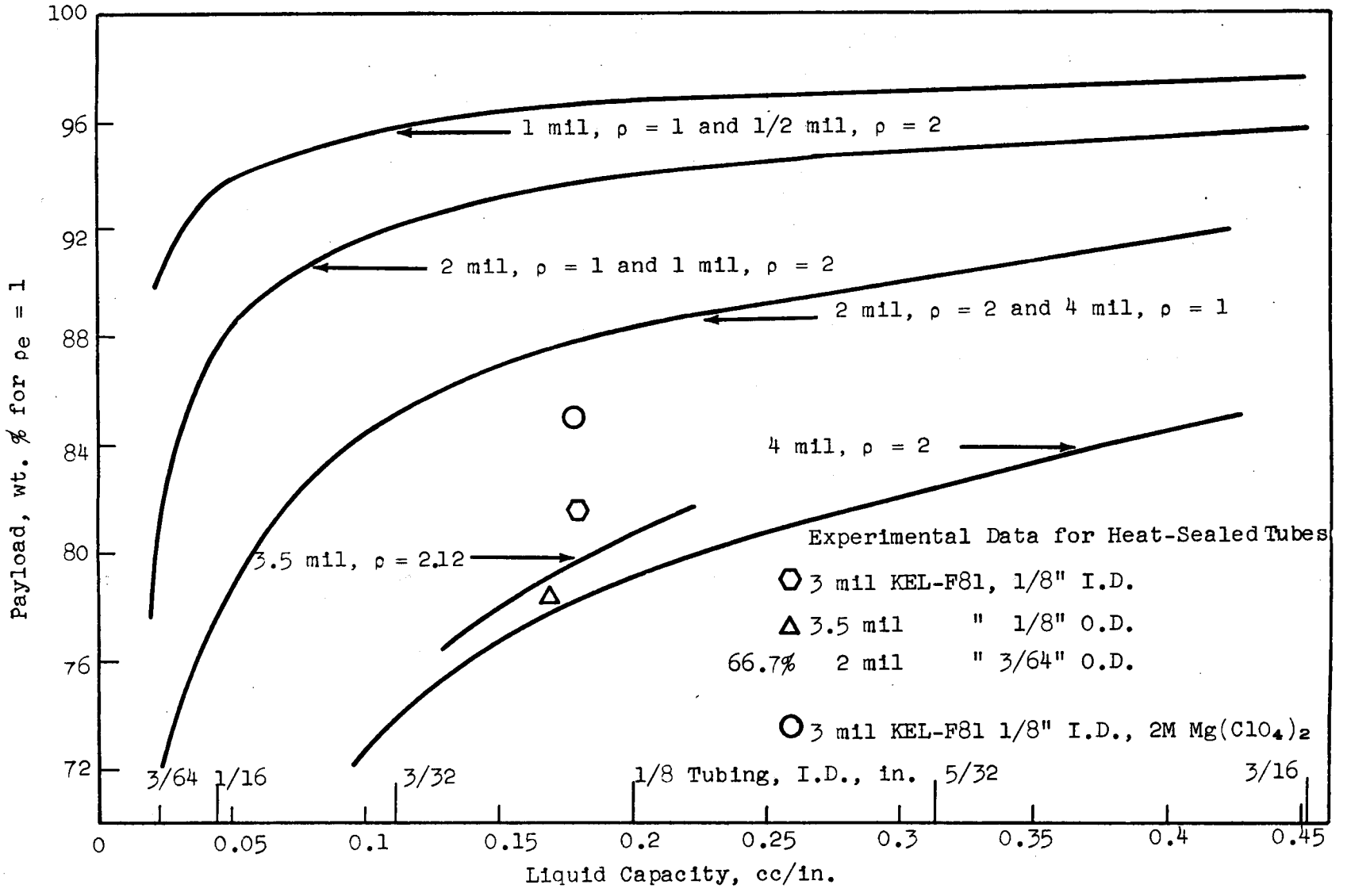
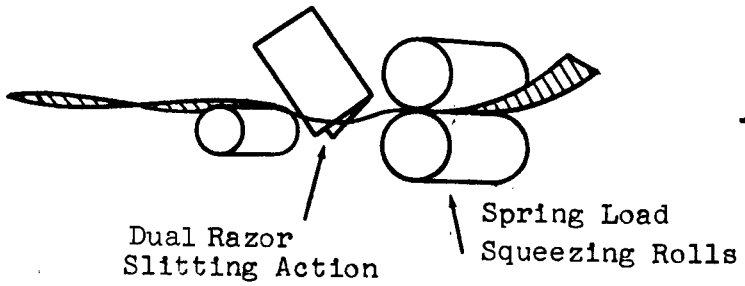
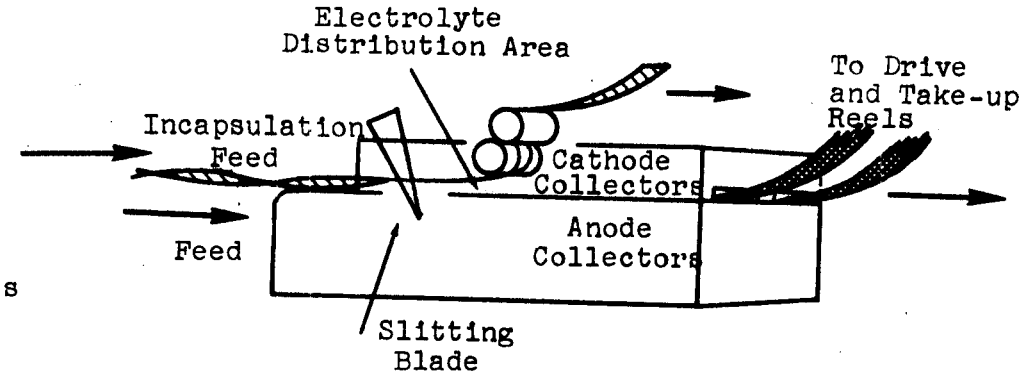


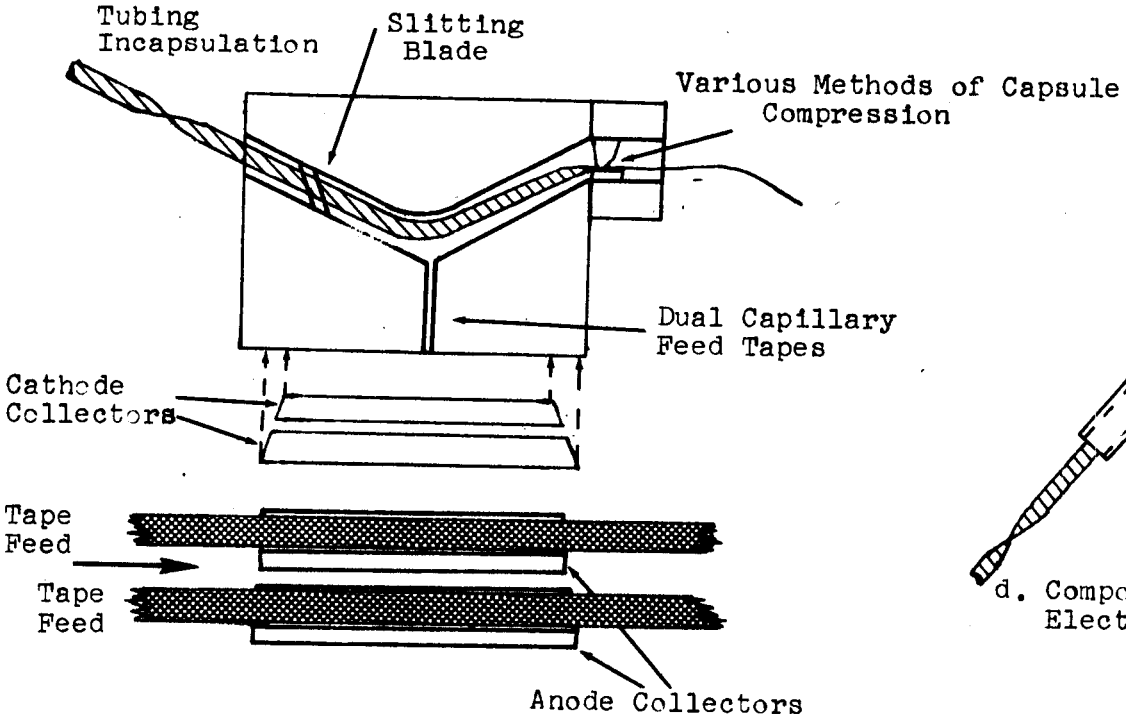
Figure 13. Calculated Film Thickness-Density-Payload Relationship for Tubing Incapsulations with no Heat Seals, and Comparison with Actual Incapsulations



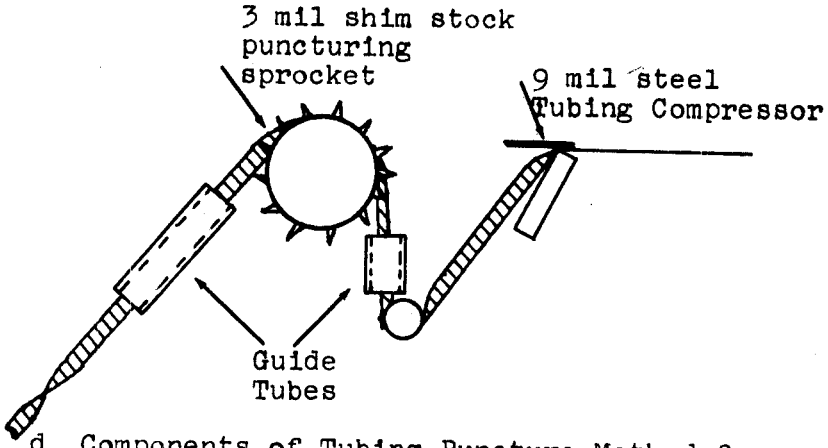
a. Slitting of Flat Capsules Followed by Squeeze Rolls



b. Slitting Method Incorporated into Tape Discharge Head for two Tape Operation



c. Tubing Incapsulation Slitting with Feed Through or Ahead of Discharge Area



d. Components of Tubing Puncture Method for Electrolyte Release

Figure 14. Methods of Electrolyte Release

With slight modification, the method was next tried on the custom-extruded Kel-F tubing subsequently obtained. Two drawbacks were noted in the slitting of the tubing, resulting both from the greater stiffness of the film and the rounder shape compared to that of the polyethylene incapsulation. First, a considerable increase in pulling force (rising from about 18 oz to over 2 lb) was usually necessary when the blade encountered the heat seal. Second, the orientation of the slit packets at the rollers varied causing occasional increase in pulling force.

A second method of release appears to present few difficulties and offers better reliability. This method employs sharp-toothed sprocket wheels of 0.003 in. thick stainless steel to puncture the capsules, a puncture being made about every 1/16- to 1/8-in. as the incapsulation is pulled over and against the wheel. The punctured capsules next move through a slot of controlled width to remove the electrolyte. This method has given a release efficiency better than 90% but the slot width required for good efficiency resulted in excessive drag. Rollers will be tried to reduce the drag.

5. Incapsulating Materials

Requirements outlined in the previous quarterly report (i.e., high electrolyte payload and low loss rate) indicated that thermoplastic films for incapsulation should have a permeability coefficient of the order of 0.002 g-mm/24 hr-m^2-cm Hg. The films used in this work were selected primarily on this basis. It should be noted that permeability values vary considerably from sample to sample and method of determination. Selected physical data on the films used are presented in Table 13.

6. Capsule Electrolyte Loss

a. Background

The transmission of a vapor or gas through a polymer film takes place primarily by a diffusion controlled mechanism, wherein the gas dissolves in one surface of the film, passes through by an activated diffusion process, and evaporates from the opposite surface. Under steady-state conditions, and assuming that the diffusion coefficient, D, is not concentration dependent, the rate of permeation through the film per unit area, q, can be expressed by

$$q = \frac{D(c_1 - c_2)}{t} \qquad (1)$$

where t is film thickness, and c_1 and c_2 are the concentrations at the high and low pressure surfaces, respectively. Applying Henry's law,

$$q = \frac{DS(p_1 - p_2)}{t} \qquad (2)$$

Table 13

PERMEABILITY TO GAS AND WATER VAPOR OF INCAPSULATION FILMS

Material	Thickness Mils	Approx. Density g/cc	Water vapor[1] Permeability g-mm/24 Hr. m^2-cm Hg at 25°C	Gas Permeability[1] cc(STP)-mil/100 in.^{2}-24 Hr-atm at 75°F	Polymer Type	Supplier
Teflon FEP Type A	2, 3	2.15	0.002			E. I. DuPont Wilmington, Del.
Teflon TFE Type HB	2	2.1-2.2			Polytetrafluoroethylene	Dielectrix Corp. Farmingdale, L. I. New York
Aclar Type 33C	2	2.12	0.001-0.002[2]	CO_2 - 16	Polytrichloroethylene	Allied Chemical Morristown, N. J.
Aclar Type 33C	5			O_2 - 7		Carmer Industries Parsipinee, N. J.
KEL-F-81 (Tube)	2, 2.5, 3.5	2.1-2.2	0.015[3]		Polytrichloroethylene	Adam Spence Corp. Union, N. J.
Mylar-Aluminum-Mylar	1-0.5-1	1.1	0.003[4]			3-M Company St. Paul, Minn.
Polyethylene	1, 2	0.9-0.96	0.02-0.08	CO_2 - 1000 O_2 - 350		
Polypropylene		0.9	0.06			
Saran		1.2-1.7	0.01-0.03	CO_2 - 5 O_2 - 1	Vinylidene chloride Vinylchloride copolymer	
Mylar		1.4	0.05-0.1[5]	CO_2 - 25 O_2 - 8	Polyester	

[1] Data from ref. 4 for water vapor transmission and ref. 5 for gas permeability

[2] Estimated from value of 0.015g/100 in^2 - 24 hr. for 2 mil film at 100°F and 90% relative humidity (RH), ref. 5.

[3] Estimated from data of Sivadjian and Ribeiro, ref. 6

[4] Estimated from value of 0.02g/100 in^2 - 24 hr. at 100°F, ref. 7

[5] Estimated from value of 110 g-mm/100in.^{2}-hr. at 103°F and 95% RH, ref. 4

where S is the solubility of the vapor in cc/cc of polymer at a pressure of 1 cm Hg with the volume corrected to S.T.P., and p is the vapor pressure; P, the permeability constant, may be defined as cc of gas at S.T.P. permeating per second through a film of 1 cm^2 area and 1 mm thickness under a pressure difference of 1 cm Hg and may be expressed as

$$P = DS = \frac{tq}{p_1 - p_2} \tag{3}$$

For gases, P should be independent of thickness and pressure, while for vapors it may increase with pressure.

The temperature dependence of P may be expressed as an Arrhenius relationship:

$$P = P_0 \exp(-E_p/RT) \tag{4}$$

where E_p is the activation energy associated with the overall permeation process, and P_0 is a constant. Since both the diffusion coefficient and the solubility depend on temperature, equation (4) will usually only approximate the temperature dependence of P. For pinhole-free films showing very low solubility for a vapor, the above relations should be quantitative and independent of whether a liquid or its vapor contact the film.

b. Method

Various methods (refs. 5,8,9,10,11,12) have been employed to measure the permeability of thin films. A diffusion cell method, employed by Barrer (ref. 8), depends on measurement of the pressure rise with time in a previously evacuated chamber that is sealed by the test sample. A modification of the Patra method (ref. 9) involves weighing a dish, filled with desiccant, enclosed by the film and stored in a cabinet of constant temperature and humidity. A hygrophotographic method, depending on the sensitivity of a double silver and mercury salt to moisture, has been developed recently by Sivadjian (ref. 13).

For our purposes, it was felt that tests on the completed incapsulations would be desirable since the results would then include any effects of heat seals, handling, etc. The gravimetric method used involves storing of capsules under the desired conditions and weighing them at intervals of time depending on the loss rate expected. The permeabilities of the films used are so low that the time during which the capsules are removed from controlled conditions for weighing is negligible compared to the total storage time and should not affect the test results.

Three test conditions were used in the evaluation of incapsulation - ambient storage (approximately 25°C and 50% R.H.), and storage under

vacuum at 25°C and 39°C (0% R.H.). Capsules of various dimensions were sealed as described in Section II-B-2. Water, 2M $Mg(ClO_4)_2$, 2M $AlCl_3$, and 37% KOH have been incapsulated in the films selected for tests. Physical data on the types of capsules tested are given in Table A-3 (Appendix).

c. Results and Discussion

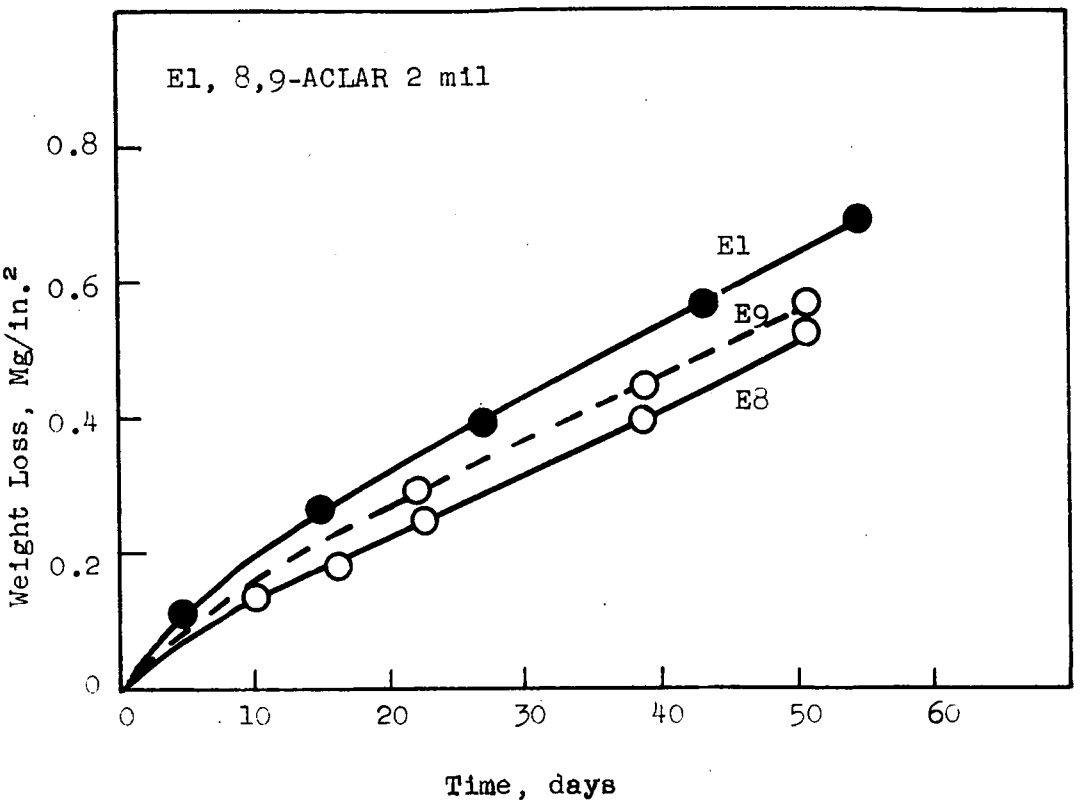
Test results on water incapsulations at ambient conditions are shown in Figures 15 through 18. The data are shown as weight loss per unit area of capsules, the area being estimated from the physical dimensions of the incapsulation. The fluorohalocarbon materials (Figures 15A and B) show the lowest vapor transmission with consistent results being obtained except for incapsulation number 4 in Figure 15B. The jump shown here after 14 days is believed to be due to a loss of a small portion of the film in handling since the continuation of the tests shows the same loss rate as obtained in the initial portion of the test. The Al-Mylar incapsulations show a much wider range of vapor transmission rates (Figure 18), the lowest of these rates being comparable with those obtained on the fluorohalocarbons. The wide variation is believed due to the heat seals since the higher loss rates were usually from capsules with proportionately greater heat seal length. The vapor transmission rates of FEP and TFE types of Teflon (Figure 16) are consistently higher than those of the fluorohalocarbon materials tested.

With the exception of capsule E20, which showed a rapid increase in transmission after 25 days, the results are consistent. The increase noted with E20 is believed due to a failure of a small portion of the seal. The polyethylene incapsulations showed high loss rates (Figure 17) as expected, and would be unsuitable for most applications even though the film density is low.

Permeability constants calculated from the vapor transmission rates are compared with values obtained from the literature in Table 14. Considerable disparity is evident in several cases, depending on the source of the values. Good agreement is evident for the fluorohalocarbon films except for reference 6 whose values are a factor of ten greater than ours.

A comparison of materials from a per cent weight loss standpoint is shown in Figure 19, all for incapsulations having approximately the same surface area per unit weight of liquid.

The superiority of the Aclar film is substantial. A comparison of incapsulations in Aclar with different surface area-to-volume ratios is shown in Figure 20. The importance of minimizing capsule surface area is evident from the increase in per cent loss proportional to the increase in surface area.



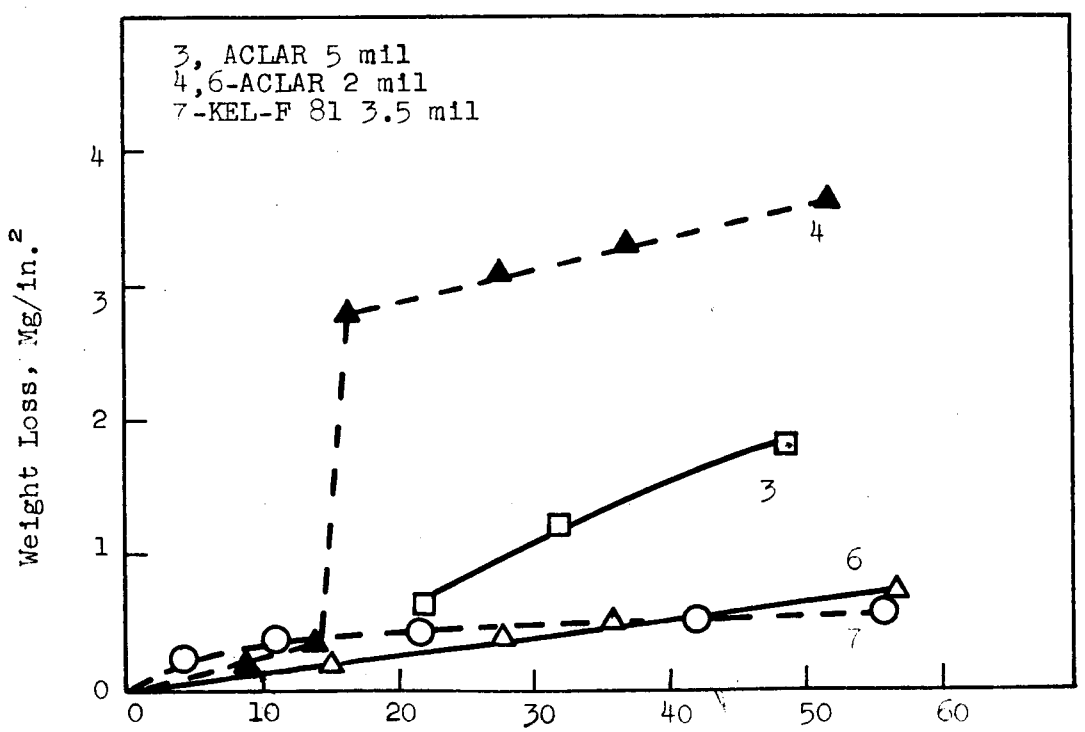


Figure 15. Weight Loss Data for Water Incapsulation in Fluorohalocarbon Films at Ambient Conditions

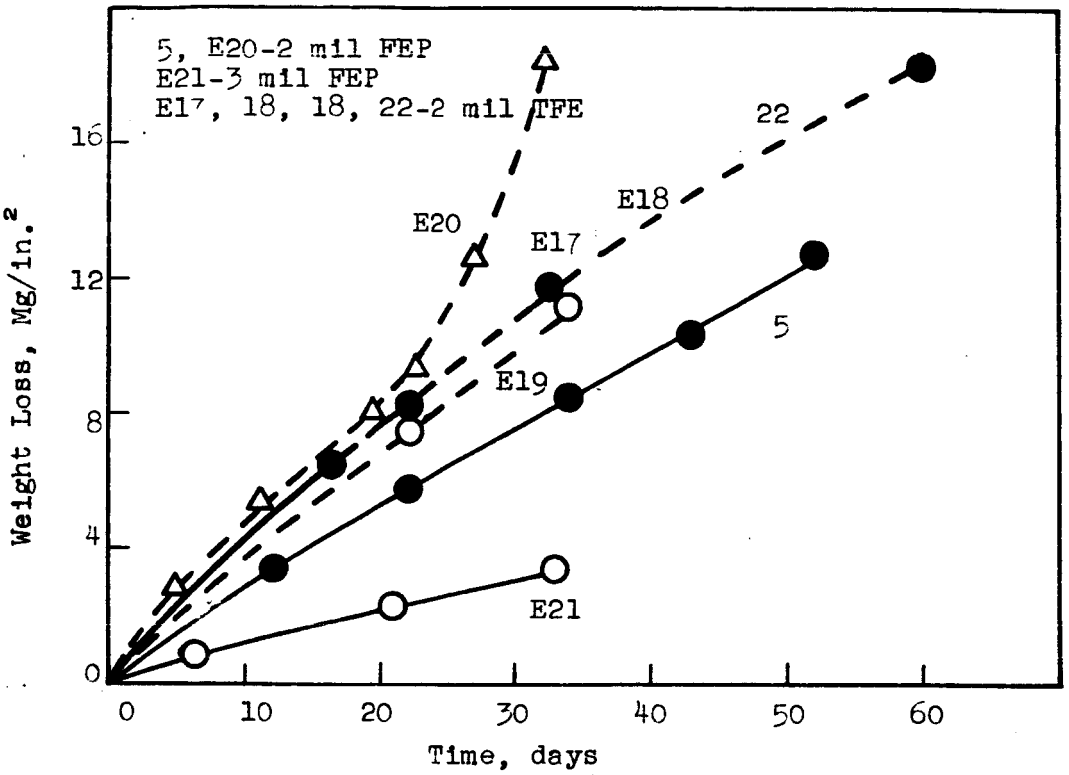


Figure 16. Weight Loss Data for Water Incapsulations in Teflon at Ambient Conditions

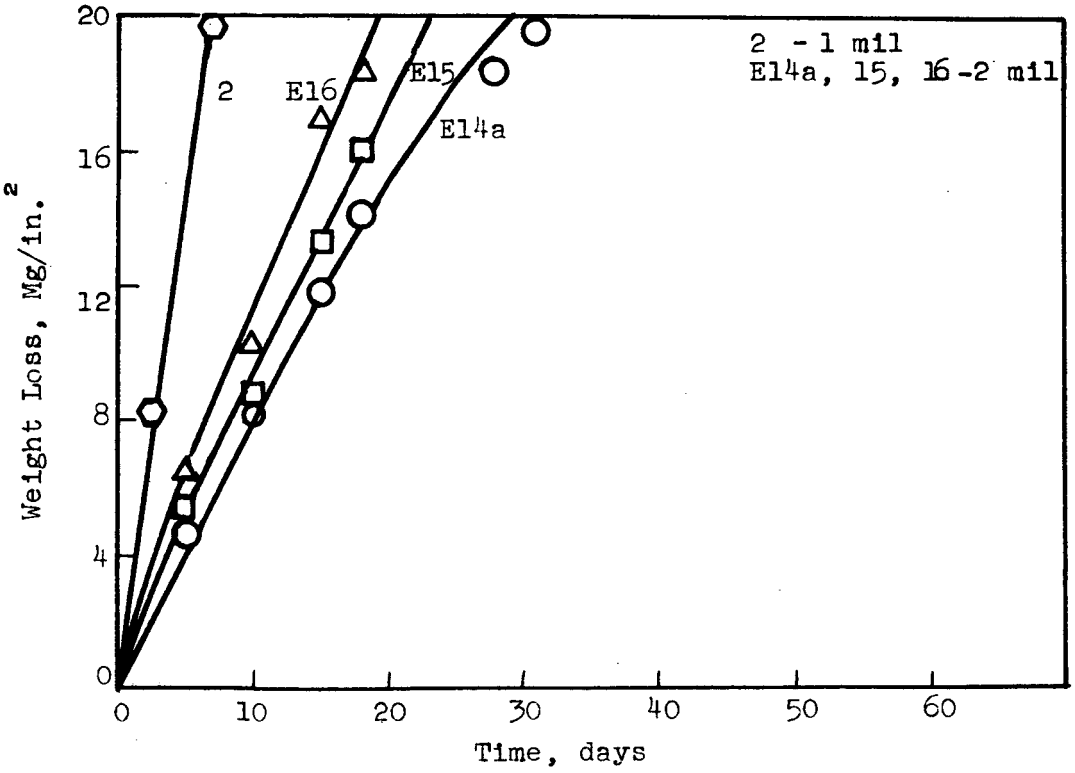


Figure 17. Weight Loss Data for Water Incapsulations in Polyethylene at Ambient Conditions

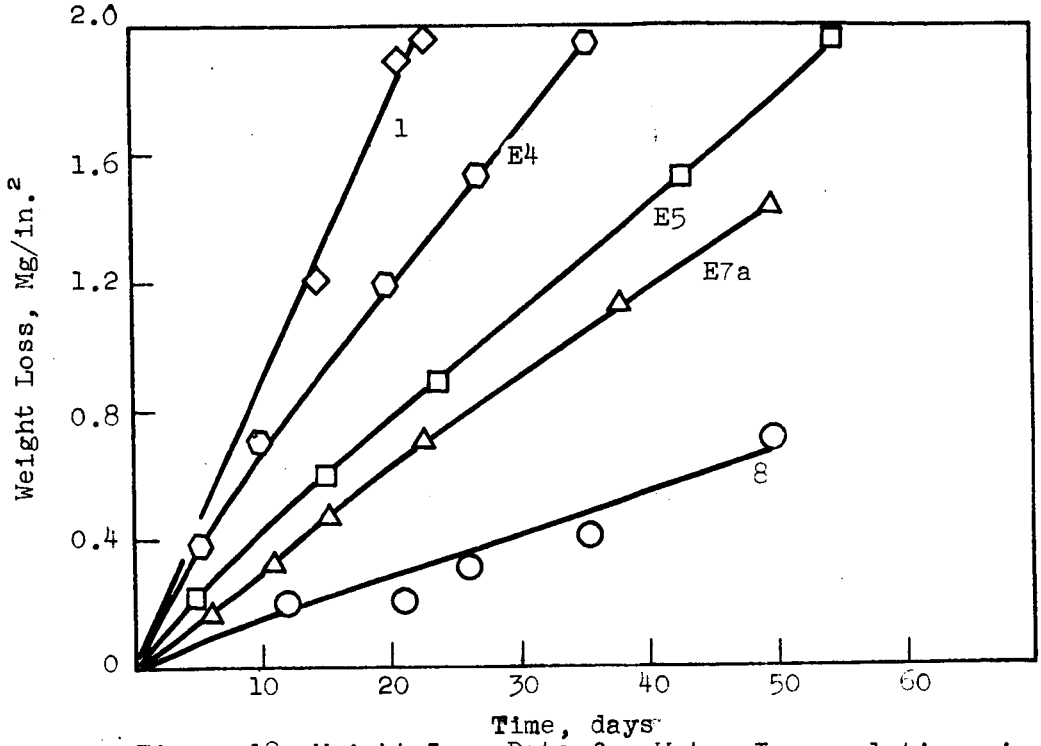


Figure 18. Weight Loss Data for Water Incapsulations in Al MYLAR (2.5 mil) Laminate at Ambient Conditions

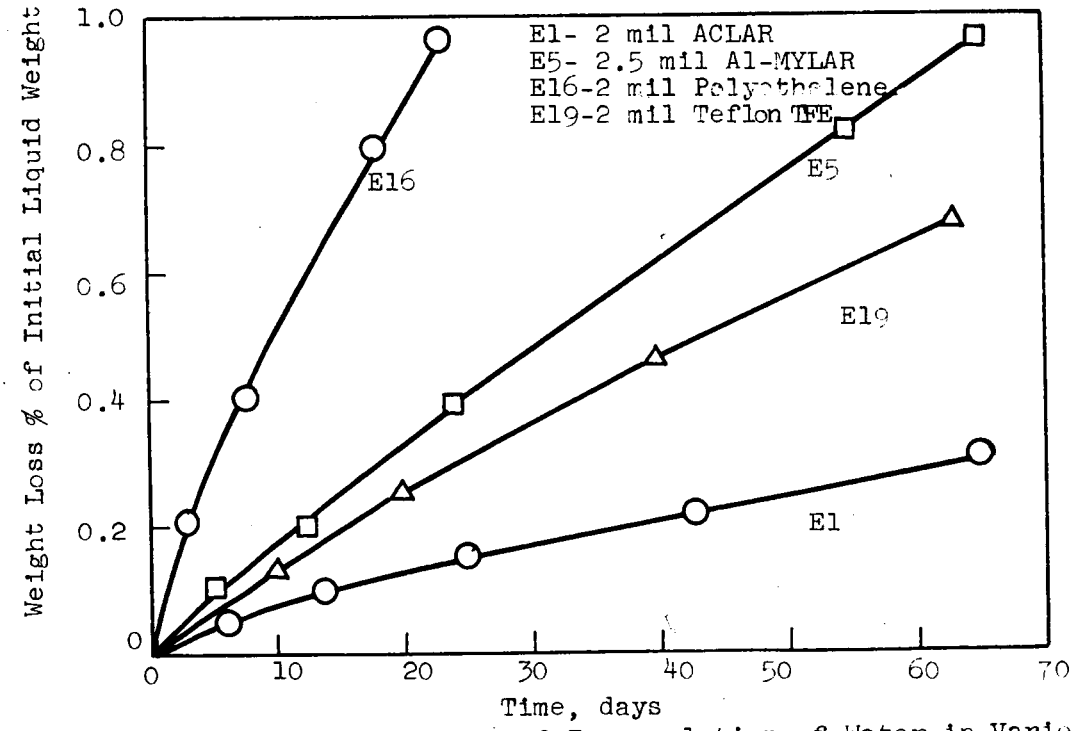


Figure 19. Comparison of Incapsulation of Water in Various Materials with a Specific Surface of 4 in.²/cc Liquid

Table 14

COMPARISON OF PERMEABILITY CONSTANTS ESTIMATED FROM THIS WORK AND AVAILABLE LITERATURE

Material	Sample Thickness Mils	Test Conditions	Reported Value	Permeability Constant Estimated at 250°C g-mm/24 hr-m²-cm Hg	Literature Reference
Polytrifluoro-chloroethylene		25°C	0.015-0.180 $\frac{\text{g-mm}}{\text{24 hr-m}^2\text{-cm Hg}}$	0.015-0.180 0.00	4 14
KEL-F 81	6.1	25°C Liquid	0.0015 $\frac{\text{g-mm}}{\text{m}^2\text{-hr}}$	0.015	6
KEL-F 81		Vapor	0.0015 $\frac{\text{g-mm}}{\text{m}^2\text{-hr}}$	0.015	6
KEL-F 300		25°C	2.9×10^{-10} $\frac{\text{cc-mm}}{\text{cm}^2\text{-sec-cm Hg}}$	0.0002	15
KEL-F 300 P25		25°C	2×10^{-9} $\frac{\text{cc-mm}}{\text{cm}^2\text{-sec-cm Hg}}$	0.002	15
Aclar 33C Aclar 33C	1 2	{100°F, 90% RH}	0.025 g/100 in²-24 hr 0.015 g/100 in²-24 hr	0.0022 (100°F) 0.0027 (100°F)	5
Aclar 33C Aclar	2 5	{25°C ≈50% RH}		0.001-0.002 0.0075	this work
KEL-F 81	3.5	{25°C ≈50% RH}		0.0018	this work
Teflon TFE		25°C		0.0	4
Teflon TFE (Type HB)	2	{25°C ≈50% RH}		0.025	this work
Teflon FEP		25°C		0.002	4
Teflon FEP		25°C	0.46 $\frac{\text{g-mil}}{\text{100 in}^2\text{-24 hr}}$	0.076	5
Teflon FEP	1.77	25°C Liquid	0.0013 $\frac{\text{g-mm}}{\text{m}^2\text{-hr}}$	0.013	6
		Vapor	0.0007 $\frac{\text{g-mm}}{\text{m}^2\text{-hr}}$	0.007	6
Teflon FEP (Type A) Teflon FEP (Type A)	2 3	{25°C ≈50% RH}		0.018-0.025 0.011	this work
Al-Mylar 25A20 Al-Mylar 25A20	2.5 2.5	100°F 25°C	0.02 g/100 in²-24 hr	0.0083 (100°F) 0.0014-0.005	7 this work
Polyethylene (Low density)	2 1	25°C 25°C {≈50% RH}		0.04 - 0.08 0.06 0.10	4 this work
Mylar		25°C		0.10	6
Saran		25°C		0.01-0.02	6

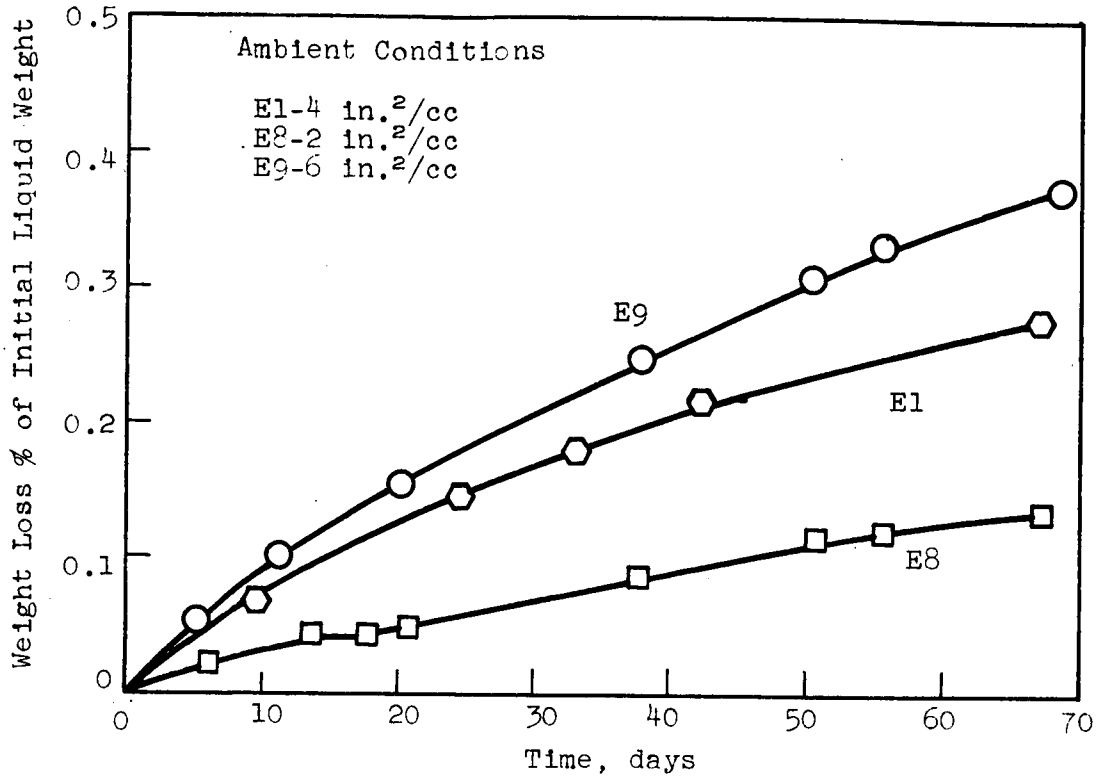


Figure 20. Comparison of Water Incapsulations in ACLAR with Different Specific Surface Areas

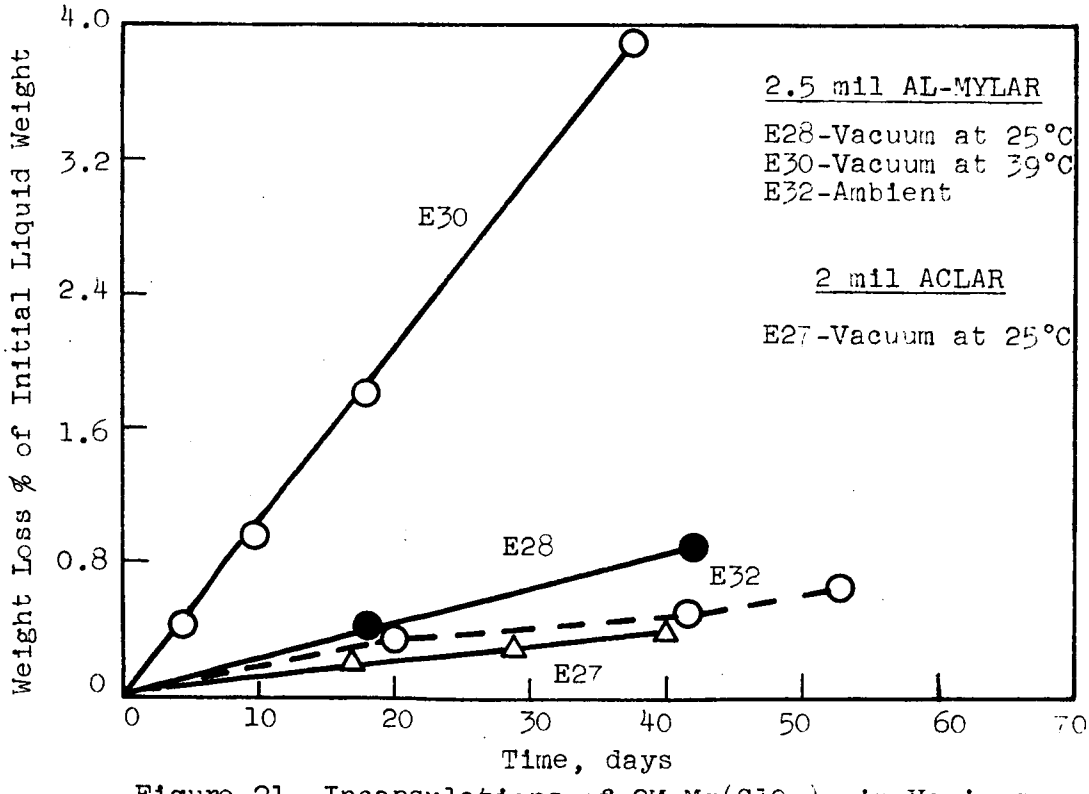


Figure 21. Incapsulations of 2M $Mg(ClO_4)_2$ in Various Environments

Results on electrolyte incapsulations and tests under vacuum and elevated temperatures are shown in Figure 21. These results are only partially completed. However, it appears that electrolyte incapsulations should provide results comparable with those for water incapsulations as would be expected since the driving force for transport is only slightly changed. The vacuum tests show slightly greater loss rates than those without vacuum, possibly due to the increase in the relative humidity difference from about 50% to 100%. By far, the more significant increase is that due to increased temperature. Here, the transmission rate is increased due to the greater vapor pressure of the liquid. This alone does not account for the total increase, the remainder being due to the effect of temperature on the film itself.

Weight loss data for incapsulations of dimensions compatible with the electrolyte requirements of the tape battery are shown in Figure 22. Also shown are the estimated results for a tubing incapsulation in Kel-F 81 supplying 0.18 cc/in. of tubing length. This tentative slection for incapsulation shows a weight loss of less than 1% per year at ambient conditions with an initial payload of about 85%. Larger amounts of electrolyte can be supplied with even better payloads.

7. Future Plans (Electrolyte Incapsulation)

a. Complete loss rate studies to provide further information on the effects of temperature and vacuum. The composition of the electrolyte will be analyzed to determine its suitability for use after prolonged storage. This will be of interest primarily for KOH electrolyte since uptake of CO_2 and resulting carbonation could reach significant levels.

b. Further development of electrolyte release methods to reduce the forces required and retain good efficiency of release.

c. Investigate methods of electrolyte conservation. This work has been delayed somewhat since electrolyte requirements and other tape characteristics are not yet fixed.

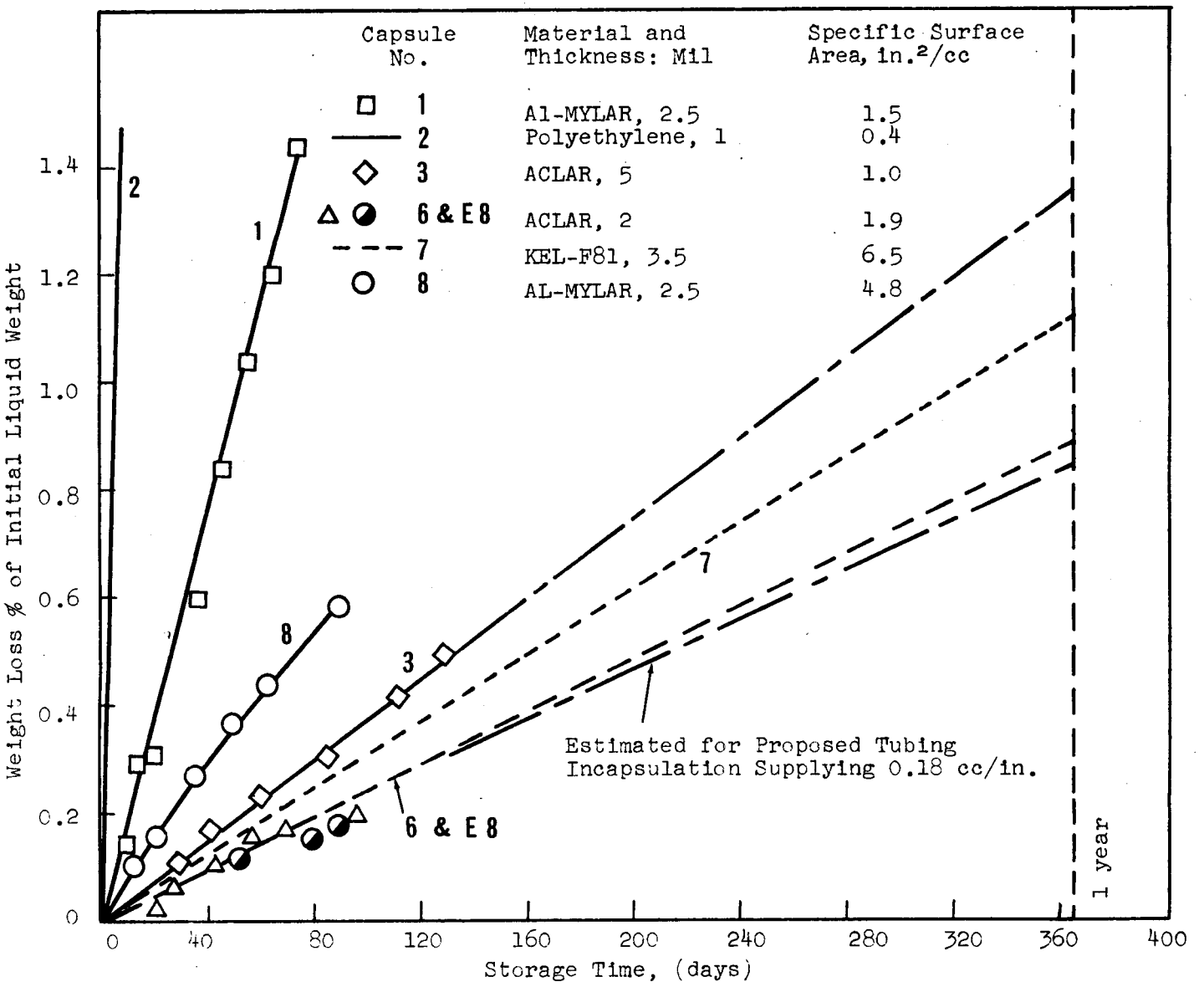


Figure 22. Percentage Weight Loss of Water from Various Shape Capsules at Ambient Conditions

III. PHASE 2 - DRY TAPE DESIGN

A. JOINT ANODE-CATHODE TESTING

1. Background

The joint-testing phase of the Dry Tape Program is concerned primarily with the engineering aspects of discharging moving tapes. The tapes used in this work are based on a variety of formulation of organic cathode materials and are manufactured by several techniques. Their physical characteristics must purposely cover a wide range. Anode performance is evaluated whether the anode is incorporated as a stationary block design or fed with the cathode as flame-sprayed magnesium metal. Data collected include current, voltage, tape speed, tape pull force, electrolyte quantity, capsule feed, electrolyte distribution, and tape integrity. The information resulting from this area has direct application in the final device phase of this program where it will provide a sound engineering basis for design and construction of a high energy density demonstration unit.

2. Cathode Tapes

Initial investigations of methods for depositing organic nitro cathode materials on tape have shown that three requirements must be met to insure satisfactory performance. The cathode coat must be (1) coherent to prevent cracking and shrinking, (2) adherent enough to prevent flaking from the tape, and (3) smooth enough to ensure continuous and intimate contact with the cathode current collector. These properties must be maintained during electrolyte wet-out to prevent scrape off and shorting while the tape is moving through the current collectors.

The tapes used in the breadboard testing were fabricated either by power spraying or by casting from a slurry with a Gardner knife. The spraying was done with a DeVilbiss spray gun in which separate, controllable air pressures are applied to both the slurry reservoir and the forced air stream (air of atomization) which carries the tiny solid particles to the tape. The Gardner knife process, described in previous reports, uses an adjustable blade behind a small quantity of slurry, both of which are drawn over a stationary tape. The casting solutions have included a variety of solvents, both aqueous and organic. Electrode compositions have covered several types of water soluble and water insoluble binders; in some samples fibrous Dynel has been included as a strength builder. Tape thicknesses ranged from 0.006-in. to 0.018-in. and base materials were varied to include Dynel, polypropylene and nylon. The majority of tests were run using 2,4,6-trichlorotriazotrione (TCTT) as the cathode active material on which approximately 50% coulombic efficiency can be expected (static half-cell tests) at current densities up to 200 ma/in^2.

3. Anode

A stationary anode, machined from cast Mg block, has been used in all tests covered in this report. Several designs have been incorporated, ranging from solid to slottted to machined blocks with drilled holes (see Figure 23). In most cases pure magnesium was used, although previous tests showed the suitability of magnesium alloy AZ31B for this purpose. Although tests of other anode configurations are planned (both flame sprayed and thin expanded metal forms), the availability of thin stock for expanding appears limited and some delay may be experienced in evaluating the latter. Hopefully, a planned trip to Dow Metal Products Division, Dow Chemical Company, may alleviate this problem as well as provide some new insight into the use of magnesium as an anode.

4. Breadboard Testing Parameters

a. Background

The workhorse for joing testing of anodes and cathodes under realistic conditions is the laboratory "breadboard" testing device. All elements required in a final tape unit are present or can readily be added, and sufficient freedom is present for detailed measurement of any particular area of operation. With some exceptions, major features of the testing procedures were the following:

(1) Anode - magnesium block, machined in several designs to facilitate gas removal, serving as both anode and current collector.

(2) Tape Speed - 0.1 to 1 in./min.

(3) Motor - Either 3Vdc for parasitic drive or 12 vdc with external power supply for adjustable speed operation

(4) Electrolyte - 2M $Mg(ClO_4)_2$ for most runs

(5) Electrolyte feed - Manual, usually through a 10 cc syringe to measure quantities

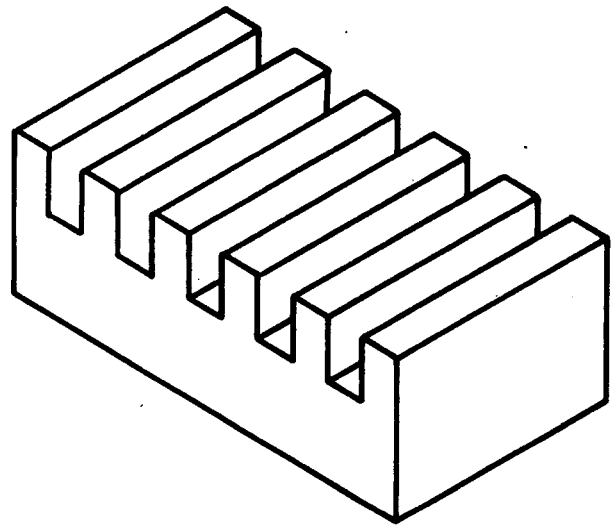
(6) Current density - varied from 0 to 500 ma/m.^{2}

(7) Tape material (separator) - 0.003 in, 0.0055 in. dynel, 0.005 in. polypropylene, 0.008 in. nylon

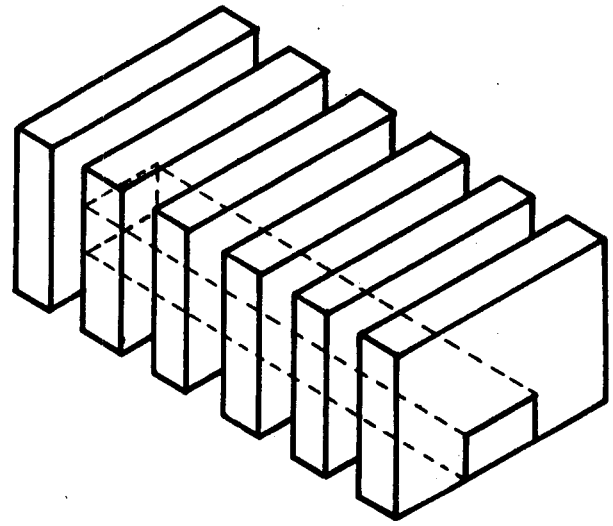
(8) Auxiliary separator - none (except for occasional usage in special tests).

b. Results of Breadboard Runs

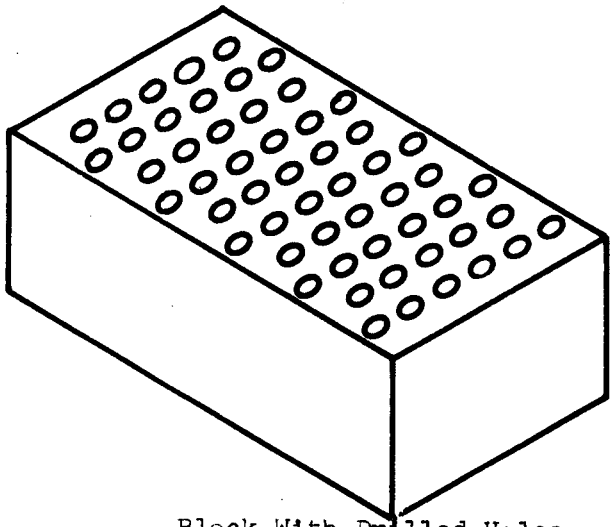
Although much of the testing was only qualitative a number of engineering and design features were established. These are summarized below.



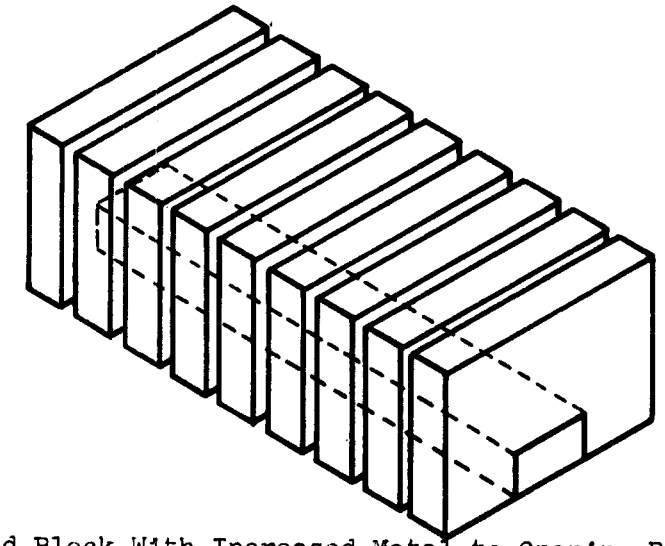
Slotted Block



Slotted Block With Open Base



Block With Drilled Holes



Slotted Block With Increased Metal-to-Opening Ratio

Figure 23. Solid Block Magnesium Anode Designs

(1) Cathode Collector Materials

Platinum, while not required for all cathode active materials, was found most suitable for general use. Platinum plating, used earlier in the series, was not sufficiently durable for reliable operation. Solid Pt was incorporated into all the later collector head designs.

(2) Cathode Collector Design

A flat, uniform, smooth collector area is preferred for tape contact, and a moderate chamfer on leading edges is helpful in preventing excessive scraping of tape coatings as they are fed to the discharge sites. Normal loadings must be uniformly applied. Uneven or erratic clamping of collector heads to the tape surfaces caused correspondingly uneven contact and irregular output.

(3) Physical Characteristics of Tape

Along with the uniformity of cathode collector mentioned above, a matching smoothness is required on the tape coating surface. The natural smoothing effect of the collector head on the moving tape (almost all tapes emerged smooth regardless of condition at entry) was insufficient to prevent voltage fluctuations at constant current discharges. Two factors were responsible: First, the leveling effect of the collector head did not occur immediately upon tape entry although the use of an inert lead-in section described below for both electrolyte dispensing and smoothing effects helped solve this. Second, and more basic, was the apparent electrical isolation of active material that resulted from physical rearrangement of the coating after it was wetted. Accordingly, more emphasis is being placed on uniformity of tape coatings for dynamic use than was required in the static discharge tests.

(4) Tape Composition

In achieving a uniform cathode tape, emphasis reverts back to both the manufacturing method and to the tape composition. In the latter area, the biggest effect was seen in the type of binder used and the nature of the slurry in which the ingredients were dispersed. Results from the early tests were adversely effected by excessive loss of cathode material at the collector sites. This was traced to a reduced binding action of the water-soluble binder after electrolyte wet-out. A switch to insoluble binders cast from organic solvents provided better integrity at the cathode collector, but these tapes were characterized by severe cracking of the coating. Some of the insoluble binders wetproofed the electrode and hindered rapid electrolyte wet-out. The addition of a small quantity of plasticizer promoted the desired soaking action but did not eliminate the cracking. The incorporation of Dynel fibers into the cathode mix added considerable strength to the water-soluble binders in particular, but generally resulted in an undesirable, rough surface.

Our efforts to combine the best features of the above approaches in a single process are described in Section B. . Spraying has produced the best quality tapes to date. Additional control over tape integrity has been obtained through improved collector and electrolyte dispensing point designs.

(5) Electrolyte Dispensing

Although only moderate attention has been given to electrolyte dispensing thus far, several engineering features have been determined. Liquid distribution is good on all tapes, virtually taking care of itself if proper amounts are supplied. This has permitted the use of a single supply port for each tape through which the electrolyte is channeled. The feeding of this manifold from a capsule source has proved quite feasible in preliminary tests. It is also advantageous to wet the non-coated side of the tape since the coating is not disturbed or scraped by the feed port. The loss of cathode materials described earlier has been partly overcome by an improved head design combining the current collectors and the electrolyte dispensing section. Since dry tapes maintained good integrity, electrolyte addition was delayed until the tape had passed between an inert extension of the cathode and anode collectors. The close confinement allowed no free space in which material could accumulate. Using this approach, several tapes not otherwise usable were satisfactorily discharged.

(6) Anode Design

Solid metallic magnesium served as both anode and current collector in all tests. Design changes were made frequently as the testing progressed. Initial runs were conducted with a slotted block in which both the lands and grooves were each approximately 1/8 in. wide. Excess electrolyte, however, tended to collect in the grooved sections. Accordingly, the unit was modified to eliminate the base of the grooves, leaving the area between the lands totally unfilled and removing all pockets in which liquid could collect. Nevertheless, maximum tape performance could not be obtained with either design. The difficulty lay in the uneven forces pressing against the tape surface by an anode which was alternately solid, space, solid, space. Because of correspondingly uneven cathode collector contact, cathode current density was not uniform and outputs suffered. Considerable improvement was obtained by the substitution of an anode with tiny holes drilled perpendicular to the exposed face and with the ratio of solid area to open area markedly increased. A slotted collector is also being fabricated in which the void spaces are considerably reduced.

A second problem area involved the creation of discharge paths around the sides of the tape due to the collection of even small amounts of cathode material. Although cathode tapes can be made on which an uncoated border is provided, this solution should be avoided if

possible since it represents a weight penalty. Anode collectors machined slightly less than the full width of the tape have been tried, but due to lack of pressure against the cathode collector, tape borders were not being discharged. A second test is planned in which the mechined-off sections of the collector are replaced by an inert plastic material. However, total discharge of border sections remains a question since a longer ion path from anode to cathode results.

(7) Drive Unit

The Zero-Max speed control system described in earlier report has been replaced with a direct drive dc geamotor for all future testing. The stop-and-go motion of the Zero-Max was reflected in output voltage fluctuations of discharging tapes. Tape linear speed is controlled by an adjustable resistance in the armature circuit of the dc motor.

(8) Electrode Coulombic Efficiencies

Tests to measure electrode output and efficiency have been conducted on the breadboard testing equipment. At times throughout the period, various sections of tapes have been monitored after a run has been started.

Data from two moving cathode tape runs are given in Table 15. The cathode reduction efficiencies of 16% and 28% at 60 and 80 ma/in.^{2}, respectively, are much lower than the 50% efficiency of static cathodes of the same type discharging at 100 ma/in.^{2}

One contributing factor to the low current efficiencies obtained during dynamic testing may be the roughness of the tapes. One series of static tests (Table 16) showed that high current collector pressure was required to enable these particular picric acid and TCTT tapes to carry high current densities with good potential. The voltage improvement as a result of increasing pressure on the TCTT cathode tapes indicates the importance of a smooth surface.

5. Future Program (Joint Anode-Cathode Testing)

(a) Improve testing equipment. The original equipment constructed to evaluate the design requirements of discharging moving tapes has outlived its usefullness. Incorporation of new modifications and added measuring equipment becomes increasingly more difficult.

Table 15

BREADBOARD DISCHARGE TESTS OF CATHODE TAPES

Cathode: ACL-85® 2,4,6-trichloro-triazine-trione
Anode: Solid Mg
Base Material: 0.0055 in. Dynel
Electrolyte: 2M $Mg(ClO_4)_2$

Item	Run A	Run B
Tape No.	S-6	S-6
Weight of Cathode Coating, g/in.^{2}	0.051	0.051
Electrolyte Usage, cc/in.	0.15	0.15
Collector Area, in.^{2}	1.68	1.68
Average Voltage, v	2.0	1.5
Average Current Density, ma/in.^{2}	60	80
Tape Speed, in./min	0.55	0.66
Run Time, min	19	26
Cathode Efficiency, %	16	28

*Cathode Formulation	%
ACL-85	45
Shawinigan Black	45
Opalon-506	7
Santicizer-160® (Plasticizer)	3

Table 16

CURRENT COLLECTOR PRESSURE EFFECT ON ATTAINABLE CURRENT DENSITY

Cell Type: Static
System: Picric Acid (46.5 wt %), acetylene black (46.5 wt %)
PVP (5 wt %), Dynel fiber (1/4 in.) (2 wt %)
Tape: Dynel 3 mil. - Coating Thickness 0.022 in. (Slurry Cast)
Electrolyte: 2M $AlCl_3$
Cathode Concentration: 0.0653 g/in.^{2}

Current Density ma/in.^{2}	Operating Voltage volts	Pressure Required lb/in.^{2}
100	1.1	2/3
250	1.0	1.5
500	0.95	3*

*increasing pressure did not increase voltage further

System- ACl 85 (50 wt %), acetylene black (50 wt %)
no binder - multiple spray technique used.
Cathode Concentration - 0.0501 g/in.^{2}

100	1.98	2/3
250	1.48	2/3
	1.66	2
	1.74*	3*
500	1.00	2/3
	1.12	1 1/3
	1.20	2
	1.30	3*

In addition, the increasing tempo of testing and the desire to measure more of the parameters involved has added to the burden on the present equipment. Accordingly, new facilities are under construction. These will not only contain most of the improvements determined above but will also include additional associated measuring equipment. Among the latter will be continual read-out of cell voltage, current, reference potentials, tape speed, electrolyte quantity, and similar information. The unit will also be adaptable to manual or capsule electrolyte feed.

(b) Continue testing with improved equipment. Continued testing will be performed on improved tapes as they are produced. Of foremost importance is the attainment of full output under dynamic conditions. An increase in the use of capsule electrolyte-feed is planned. Extensive testing of longer lengths of tape must be made since little is known about the time-effects on the design considerations discussed above.

B. TAPE MANUFACTURING METHODS

1. Background

The effort in this phase is concerned primarily with the investigation of manufacturing methods for dry tapes. A thorough process development program is not required. Rather our work must result in the selection of a suitable method for producing dry tapes and the attainment of sufficient capability to manufacture the electrodes needed for the completion of the main phases of the contract. A preliminary pilot plant set-up is envisioned.

2. Methods Investigated

a. Knife Casting

Most of the tapes have been made using this method both because of the simplicity of the process and because of its ready adaptibility to a variety of tape compositions. However, several limitations have become apparent. The reservoir of slurry behind the knife edge is pulled over the entire tape length and an impregnation rather than a surface coating tends to result. Consequently, thicker base tapes are usually required with this method. Casting a full coating thickness at once places a great strain on the drying process during which some contraction occurs. Organic solvents dry rapidly, causing severe cracking of the surface coating. Water solutions do not crack but the final traces of moisture during the longer drying period appear to concentrate in a multitude of small spots which result in an abundance of tiny mounds and an uneven surface. Temperature variations to speed up or slow down the drying had little effect. Nor did preshrinking (if any occurred) the base tapes. Nevertheless, the knife method does give reasonably good control over thickness of cast tapes

and, when used in conjunction with Dynel fibers, extremely rugged and sturdy tapes result. Accordingly, some attempts will be made to compensate for poor coating surfaces through incorporation of secondary treatments, possible with Teflon rollers while tapes are still moist.

b. Power Spraying

The DeVilbiss spray gun used for this work has two separate feed systems. The first employs an adjustable air pressure over the slurry material to be sprayed, forcing it through the exit nozzles at a controlled rate. The second system, equally controllable, supplies only the air stream (or air of atomization) on which the particles from the solution reservoir are carried to the work. To permit effective usage of the equipment, a rotating drum approximately 15 in. in diameter was attached by pulley and drive belt to an adjustable-speed motor. Base tape material was attached to the circumference of the drum, allowing production of cathode tapes approximately 1 foot x 4 feet.

Water and organic slurries were used, the water based type being eliminated immediately due to severe impregnation. The organic slurries, however, produced good quality tapes from the outset. The naturally rapid evaporation of these solvents plus their breakdown into a find mist by the atomization air resulted in almost instant drying. Thus, the solvents serve excellently as a carrier medium to the gun exit nozzle but the net effect from that point is virtually a spraying of solid matter. Barely sufficient moisture remains to promote adhesion to the tape material and the tapes are totally dry within 1-3 seconds. These very same factors permit layer to be sprayed upon layer as the drum rotates, since over 95% of the evaporation has taken place before the tape is coated. A secondary benefit is realized in that the added material is a true surface coating making possible the use of thinner base materials.

Tapes prepared by the spray method are more fragile than many of those cast with a Gardner knife, particularly when Dynel fibers are used. Nevertheless, flexibility is excellent and they are sufficiently durable to allow winding on a reel, unwinding, discharge, and rewinding without visible material loss. Care must be used when slitting to proper width. Surfaces are quite smooth which circumrents some of the design problems discussed earlier. A major problem area is an occasional uneven slurry flow which causes thickness variations ($\pm$0.001 in. to $\pm$0.002 in.). Some corrosion of passage ways is evident despite the purchase of stainless steel parts for all vital areas. To correct both these difficulties and to permit use of the gun with all of the cathode materials likely to be investigated, Teflon parts are being substituted for all slurry carrying passageways.

3. Future Plans (Tape Manufacturing Methods)

(a) Improve present methods. In addition to improvements on the spray gun itself, steps are being taken to enlarge slurry reservoir capacity for extended spraying times. This will allow greater coating thicknesses and the production of longer tapes. Since the method presently used requires repeated exposure of tape surfaces to the spray stream, a process to provide this for longer tape lengths must be developed. Also, closer control over the mass of material expelled per unit time must be maintained to achieve better coating thickness tolerances.

The versatile knife casting methods cannot yet be abandoned. Subsequent processes will be added to achieve better control over surface conditions. Initial trials will use Teflon rolls applied at different stages during the drying process.

(b) Evaluate extrusion coating. A possible tape coating technique which retains most of the advantages of the knife method is an extrusion process. As with the Gardner knife, subsequent surface conditioners are envisioned although multiple thin layer extrusion appears possible without additional controls. Thickness control should be simplified and, if necessary, fibers can be incorporated into the cathode mix (doubtful with spraying). Much of the extrusion experience available at Monsanto Company's Springfield location will be utilized in our initial efforts in this area.

IV. PHASE 3 - CONVERSION DEVICE DEVELOPMENT

A. TAPE DRIVE MECHANISM

1. Objectives

The major objectives during this development phase were:

a. To assemble a "first generation" portable tape transport device.

b. To investigate "start" devices, both mechanical and electrical, for tape transport following a shutdown period of the device.

c. To continue to investigate sources for suitable, efficient, commercially available motors - permanent magnet shunt and incremental stepping motors.

d. To investigate materials of construction - metals and plastics - as part of the weight optimization objective.

2. Portable Tape Transport Device

Analysis of our requirements for tape transport and comparison with the features found in commercial magnetic tape recorders led to the procurement of an inexpensive commercial recorder. Cannabalizing and adapting components, a portable prototype unit was assembled complete with dummy tape and electrolyte packets. The device was operated with an external dc power source applied to a small dc permanent magnet motor. Motion occurred; tape was transported from one reel to a take-up reel via a rotating capstan; electrolyte packets were punctured, contents released, and the dummy tape efficiently wetted.

No attempts were made to generate power. The sole purpose of these demonstrations was to observe the mechanical relationships of this specific "first generation" prototype and to establish the following design concepts for future optimized devices.

a. Tape tensioning devices.

b. Tape drive capstans - friction or positive drive.

c. Physical size requirements.

d. Optimum arrangement of components.

e. Tape guidance.

f. Tape storage.

No additional demonstrations are contemplated for this original prototype. New prototypes will be assembled, within the framework of the remaining project time schedule, incorporating the improvements that have been made in component testing.

3. Start Systems and Devices

a. Background

The tape transport device must be capable of parasitic operation. This is achievable only during the dynamic mode of operation. Activated ingredients are then continuously transported into the current collectors. Electrical power is continuously developed to sustain the driving motor and any external electrical load. Following a shutdown period, however, no active ingredients are available at the collector surfaces, and, unless otherwise provided for, no power is available to start the tape drive again. Thus, a supplementary energy system must be available to permit pulling a short section of "fresh" tape into the collector area. At this point, a portion of the tape output can again be delivered to the driving motor to perpetuate the tape transport. The frequency of start-stop operation, as required by the mission, will dictate the type of supplementary system to be used.

b. Methods for Starting Tape Battery

Three methods have been considered for this function:

(1) Rechargeable nickel-cadmium battery package with charging system.

(2) Primary silver oxide battery package with no charging system.

(3) An all-mechanical, spiral spring motor with spur gears, one-way clutches, and slip clutches driven by the shunt motor, which concurrently drives the tape capstan.

The weight of each of these systems is as follows:

	Weight, grams
Four Eveready N24 Ni-Cd battery with charger	12 + 40 = 60
Four Eveready S76E silver oxide batteries, no charger	10 + 0 = 10
Mechanical system of magnesium alloy	200

Each system under breadboard test conditions repeatedly performed the "start" function. Each is reliable within its specific sphere of application. Of the three, the mechanical system is the best for long mission reliability but has an attendant weight penalty.

The four Eveready S76E silver oxide batteries in series-parallel connection, produced the equivalent of 60 "starts" of 5 minutes duration per start in a twenty-hour period. Following each 5-minute start cycle, there was a 15-minute rest period. Under these conditions, this small, 10-gram package produced approximately 0.260 watt-hour of electrical power.

In addition, the primary battery system is the most efficient of the three for the following reasons:

(a) No parasitic power is required to charge and then continuously "trickle charge" the battery as is the case with rechargeable batteries.

(b) The all-mechanical system, because of its spur gearing arrangement and sliding clutches, is probably least efficient. Consequently, greater amounts of parasitic power would be required for the shunt motor to overcome these frictions while storing energy in the spiral spring.

The expected shelf life of these small primary batteries, at ambient conditions, is of the order of three to four years.

For these reasons, the primary battery package for starting the tape transport device must be considered for the short mission.

4. Motors

The efficiencies of two tape driven systems were measured using the same dc permanent magnet motor in both systems.

The first system featured a spur gear train (Zero-Max adaptation described in earlier reports) for varying the linear speed of tape. The second used a rheostat in series connection with the armature for varying the rpm of the motor shaft and its directly coupled capstan. Following are the efficiencies:

Mechanical linear speed changer:	4.2%
Resistive linear speed changer:	6.5%

These low efficiencies are principally due to the low efficiencies of the commercially available dc permanent magnet gear motors in the low nominal voltage range (3 to 4 v) and speeds (1/10 rpm) of interest to us.

A continuing effort is being made to procure commercial motors that have higher efficiencies in the torque and speed ranges common to our transport device. This will result in higher overall efficiencies for our device irrespective of the method for linear speed control - gear and linkage system or resistive control.

The following is a weight tabulation of the two existing devices for linear speed control:

	Overall Efficiency	Weight in Grams
Mechanical System	4.2%	150
Rheostat Control System	6.5%	30

In these small permanent magnet gear motors, the speed reduction ratios are of the order of 5000 to 1. With this amount of reduction, gear box efficiency runs about 55% resulting in the low overall efficiencies noted above.

It is logical that to attain highest gear motor efficiency at low rpm, the gear box should be eliminated. This is currently being done with so-called "stepping motors" or "steppers". This type of motor drives a mechanical load in a series of short steps.

There are many types of stepping motors but generally the ones investigated are all electrically powered by dc pulses from a pulser that varies the frequency of pulses and thus speeds up or slows down the incremental rotation of the shaft more efficiently than by resistive control of a dc shunt motor. Present state-of-the-art pulsers (sometimes called controllers) are composed of printed circuit and solid state electronic devices.

This type of motor system is currently being investigated for possible application to the tape transport development. Two manufacturers whose products look promising are:

(1) IMC Magnetics Corp., Maywood, California

(2) Sigma Instruments, Inc., Braintree, Mass.

As we discovered, stepping motors are not the panacea one would expect from a superficial examination of their merits.

Following are some of the problem areas attendant with their use in our application:

(a) To obtain our torque and speed requirement of 50 oz-in. at 0.1 rpm, we would require an external gear train in combination with the motor. This gear train, because of its inefficiency, partly offsets the advantage of this motor.

(b) The commercial pulser is heavier as a package than a commercial dc permanent magnetic motor with rheostat; 12 oz vs 4 1/2 oz, respectively.

(c) Tape reel inertia (fly-wheel effect) could be troublesome. A stepping motor reacts to a command pulse frequency. It must in a small increment of time (milliseconds) accelerate and decelerate the load. Depending upon pulse frequency and tape reel inertia, the motor may stall, the tape may break, or the tape reel may over-travel and unravel to an undesirable degree.

The stepping motor is not as directly applicable to our device as are commercially available dc permanent magnet motors. A custom engineering adaptation of both motor and control system is implicit even with the so-called commercial steppers.

5. Future Plans (Tape Drive Mechanism)

(a) Continue investigation of stepping motors before freezing a final design.

(b) Build portable tape transport unit using as starting energy sources:

(1) primary battery
(2) secondary battery
(3) mechanical device

B. MATERIAL INVESTIGATION

1. Structural Metals

Materials investigated included most structural metals and some plastics. If this device is to have some space implications and applications, we can narrow down the field to a few. These would be:

(a) Aluminum
(b) Magnesium
(c) Stainless steel
(d) Low carbon alloy steel

Table 17 compares the strength to stiffness of these metals, as alloys.

Table 17

RELATIVE COMPARISON OF VARIOUS SHEET MATERIALS AT EQUAL BENDING STRENGTH AND EQUAL STIFFNESS AT ROOM TEMPERATURE

Comparison	Alloy No.	Material	Thickness	Bending Strength	Stiffness	Weight
Equal Bending Strength	AZ31B-H24	magnesium	1.00	1.00	1.00	1.00
	6061-T6	aluminum	0.87	1.00	1.00	1.33
	5083-H113	aluminum	0.92	1.00	1.23	1.38
		steel	0.85	1.00	2.77	3.77
Equal Stiffness	AZ31B-H24	magnesium	1.00	1.00	1.00	1.00
	6061-T6	aluminum	0.87	1.02	1.00	1.33
	5083-H113	aluminum	0.86	0.87	1.00	1.29
	SAE 1025	steel	0.60	0.50	1.00	2.66
	Plastic (glass laminate)				1.00	3.2

It can be seen from the tabulation that magnesium alloy is a structural material of great merit for lightweight devices.

Aluminum is superior to magnesium in its position in the galvanic series and also in formability. Aluminum would be a logical choice if weight were not the prime consideration.

Appreciable stainless steel will not be used in this development because of the weight penalty.

We are cognizant of the potential corrosion problems attendant with magnesium, but Dow Metal Products Company has given us some recommendations and direction in this matter and we are hopeful that these will prove helpful in avoiding or mitigating the potential corrosion problems. Some of these protection methods are:

(a) Coating magnesium with a clear vinyl film.

(b) Coating with white titanium oxide as manufactured by Sherwin Williams.

(c) Anodizing to provide a coating of the order of 0.0001 in. thick as performed by two companies in Connecticut.

Some magnesium alloy sheet (AZ31B-24) has been purchased and a small prototype chassis and tape reel configurations assembled. In combination with the magnesium alloy, we employed polystyrene foams for additional stiffness, strength, and weight reduction. These two materials will appear in quantity in the final, optimized device.

2. Future Plans (Material Investigation)

Since the construction materials have been selected, no further work is planned for this section.

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APPENDIX

Table A-1. Binders, Tapes, and Fibrous Fillers used for Dry Tape Fabrication

Table A-2. Tape Formulations and Static Cell Characteristics of Mg/Nitro Compound

Table A-3. Physical Data on Capsules Prepared for Permeability Tests

Table A-1

BINDERS, TAPES, AND FIBROUS FILLERS
USED FOR DRY TAPE FABRICATION

Designation	Component	Source
	Water-Soluble Binders	
PVP NP-K30	Polyvinylpyrrolidone	General Aniline & Film
Gelvatol 20-60	Polyvinyl alcohol	Shawinigan Resin Corp.
Gontrez AN-139	Methyl vinyl ether-maleic anhydride copolymer	General Aniline & Film
Methocel HG	Hydroxymethyl cellulose	Dow
Cyanamer P-26	Polyacrylamide	American Cyanamide
Cyanamer P-26	Acrylamide-acrylic acid copolymer	American Cyanamide
	Reinforcing Fibers	
Dynel 1/4 - 1/8 in. 3 denier	Acrylonitrile-vinyl chloride copolymer	Microfiber, Inc.
	Tapes	
Dynel	Acrylonitrile-vinyl chloride copolymer (3 mil)	Kendall Corp.
	Polypropylene (3 mil)	Kendall Corp.
Pellon	Nylon (3 mil)	Pellon Corp.
	Conducting Substrates	
	Acetylene black (50% and 100% compression)	Shawinigan Resin Corp.
	Organic Depolarizers	
	Picric acid	Eastman Organic
	o-DNB	K&H Laboratories
	m-DNB	Fisher Scientific

Table A-2

TAPE FORMULATIONS AND STATIC CELL CHARACTERISTICS OF Mg/NITRO COMPOUND

Test No.	Depolarizer	Carbon Depolarizer (Note 1)	Fiber Content of Coating, % (Note 2)	Binder Content of Coating, % (Note 3)	Fabric (Note 4)	Depolarizer mg/in.^{2}	Electrolyte	Current Density ma/in.^{2}	Cell Efficiency % to 0.8 v	Cell Efficiency % to 0.65 v	Comments
60661	Picric Acid	1:1	6	11.4 PVP	D	99.7	2M $AlCl_3$	500	39.5	45.3	
60661	" "	1:1	6	11.4 PVP	D	96.5	2M $AlCl_3$/ 1M Li_2CrO_4	500	20.7	27.0	
60662	" "	1:1	6	11.4 PVP	D	53.1	2M $AlCl_3$/ 1M Li_2CrO_4	100	54.3	-	
63102	Blank	1:0	6	11.4 PVP	D	-	2M $AlCl_3$/ 1M Li_2CrO_4	100	(25 min)	(65 min)	
63103	Picric Acid	1:1	6	15 PVP	D	90.0	2M $AlCl_3$	500	44.3	51.1	
63103	" "	1:1	6		D	78.5	2M $AlCl_3$	200	55.2	61.2	
63106	" "	1:1	6		D	94.6	2M $AlCl_3$/ 1M Li_2CrO_4	500	21.1	35.9	
63107	" "	1:1	-	17 PVP	N-2	73.1	2M $AlCl_3$	200	61.3	70.0	
63107	" "	1:1	-	17 PVP	N-2	58.0	2M $Mg(ClO_4)_2$	100	59.3	71.7	
63108	" "	1:1	-	29 PVP	N-2	66.8	2M $Mg(ClO_4)_2$	100	55.2	74.3	
63109	" "	1:1	7.5	11 PVP	D	118	2M $Mg(ClO_4)_2$	300	26.6	36.7	
63109	" "	1:1	7.5	11 PVP	D	96.1	2M $AlCl_3$	500	39.5	45.7	
60662	" "	1:1	6	11 PVP	D	38.2	2M $AlCl_3$	500	41.8	-	
60662	" "	1:1	6	11 PVP	D	112	2M $Mg(ClO_4)_2$	300	28.9	40.8	
60663	" "	1:1	6	11 PVP	D	45.7	2M $Mg(ClO_4)_2$	300	25.7	40.8	
60623	o-DNB	1:1	-	11 PVP	N	115	2M $Mg(ClO_4)_2$	100	12	-	Conductex SC (Columbian)
60624	" "	1:1	-	11 PVP	N	117	2M $AlCl_3$	100	14.9	18.5	Conductex SC (Columbian)
60624	" "	1:1	-	9 PVP	N	114	2M $AlCl_3$	100	9		FC-13 Carbon
60623	" "	1:1	-	9 PVP	N	84.7	2M $Mg(ClO_4)_2$	100	≈ 1	6.5	Asbury Graphite A625
60623	" "	1:1	-	9 PVP	N	81.1	2M $AlCl_3$	100	5.4	-	Asbury Graphite A625
60619	" "	1:2	-	11 PVP	N	109	2M $Mg(ClO_4)_2$	200	6.4	9.5	Nuchar CN
60619	" "	1:2	-	11 PVP	N	98.5	2M $AlCl_3$	300	17.2	18	Nuchar CN

NOTES:

1. Shawinigan acetylene black, 50% compression unless otherwise noted.
2. 1/4 in. 3-denier Dynel fibers unless otherwise noted.
3. PVP = Polyvinylpyrrolidone, G = Polyvinylacetate-polyvinyl alcohol.
4. D = Webril nonwoven DYNEL No. 1410, N = Pellon Nylon No. 2505B, N-2 = 60 mil Nylon 524.

Table A-2 (continued)

TAPE FORMULATIONS AND STATIC CELL CHARACTERISTICS OF Mg/NITRO COMPOUND

Test No.	Depolarizer	Carbon Depolarizer (Note 1)	Fiber Content of Coating, % (Note 2)	Binder Content of Coating, % (Note 3)	Fabric (Note 4)	Depolarizer mg/in.^{2}	Electrolyte	Current Density ma/in.^{2}	Cell Efficiency % to 0.8 v	Cell Efficiency % to 0.65 v	Comments
60619	o-DNB	1:2	-	11 PVP	N	109	2M $Mg(ClO_4)_2$	300	9.6	19	A
60619	" "	1:1	-	11 PVP	N	92.8	2M $Mg(ClO_4)_2$	300	5.6	14	A
60631	" "	9:1	-	11 PVP	N	14.6	2M $Mg(ClO_4)_2$	100	41.7	53.5	A
60631	" "	9:1	-	11 PVP	N	20	2M $Mg(ClO_4)_2$	100	34.8	-	A
60633	" "	9:1	-	11 PVP	N	11.7	2M $Mg(ClO_4)_2$	50	40.8	44.7	A
60633	" "	9:1	-	11 PVP	N	13.0	2M $AlCl_3$	100	40.2	-	A
60629	" "	1:1	-	11 PVP	N	83.6	2M $AlCl_3$	500	26	31.2	A
60629	" "	1:1	-	11 PVP	N	120	2M $AlCl_3$	500	21.8	-	A
60629	" "	1:1	-	11 PVP	N	112	2M $Mg(ClO_4)_2$	200	≈10	-	A
60617	" "	1:5	-	11 PVP	N	61.7	2M $Mg(ClO_4)_2$	100	8.5	19.7	
60617	" "	1:5	-	11 PVP	N	47.0	2M $Mg(ClO_4)_2$	100	9.2	20.3	
60654C	Picric Acid	1:1	-	11.7 PVP	D	112	2M $AlCl_3$	500	21.4	28.6	
60636-2	" "	1:1	-	11.7 PVP	D	68.6	2M $AlCl_3$	500	35	-	
60646A	" "	1:1	-	11.7 PVP	D	146	2M $Mg(ClO_4)_2$	500	13.7	21.9	
60646B	" "	1:1	-	11.7 PVP	D	147	2M $AlCl_3$	500	19.0	24.7	
52292	" "	1:1	-	11.7 PVP	D	59.5	2M $AlCl_3$	17	77.5	81.0	
52292	" "	1:1	-	11.7 PVP	D	107	2M $AlCl_3$	200	35.9	41.8	
52297	" "	1:1	-	11.7 PVP	D	106	2M $AlCl_3$	500	32	43.4	
52230	" "	1:1	-	11.7 PVP	D	55.7	2M $AlCl_3$	200	28.8	33.1	
60665	" "	1:1	6	11.1 PVP	D	21.6	2M $AlCl_3$	500	48.2	57.5	
40665	" "	1:1	6	11.1 PVP	D	18.1	2M $AlCl_3$	500	46.2	55.2	
60665	" "	1:1	6	11.1 PVP	D	46.7	2M $AlCl_3$	500	42.1	47.3	
60665	" "	1:1	6	11.1 PVP	D	21.5	2M $Mg(ClO_4)_2$	300	29.1	44.7	
60666	" "	1:1	6	11.1 PVP	D	141	2M $AlCl_3$	500	23.5	26.4	Sample pressed
60666	" "	1:1	6	11.1 PVP	D	79.3	2M $AlCl_3$	100	34.3	40.6	
60667	" "	1:1	6	11.1 PVP	D	76.6	2M $AlCl_3$	100	59.5		10% by wt Pd
60670	" "	1:1	6	11.1 PVP	D	54.5	2M $AlCl_3$	500	27.2	33.8	B
60670	" "	1:1	6	11.1 PVP	D	61.5	2M $AlCl_3$	500	24.6	29.8	B
60670	" "	1:2	6	11.1 PVP	D	59.3	2M $AlCl_3$	500	28.4	33.7	
60670	" "	1:2	6	11.1 PVP	D	95.1	2M $AlCl_3$	500	25.2	38.3	
63114	" "	1:2	6	11.1 PVP	D	31.8	2M $AlCl_3$	500	62.9	65.7	
63115	" "	1:2	6	11.1 PVP	D	25.3	2M $Mg(ClO_4)_2$	100	38.0	57.0	

COMMENTS: A. 1:1 FC-13:Shawinigan black

B. 100% Compressed acetylene black

C. Succinic acid:Picric acid is 1:1

D. o-DNB under 300 mesh

Table A-2 (continued)

TAPE FORMULATIONS AND STATIC CELL CHARACTERISTICS OF Mg/NITRO COMPOUND

Test No.	Depolarizer	Carbon Depolarizer (Note 1)	Fiber Content of Coating, % (Note 2)	Binder Content of Coating, % (Note 3)	Fabric (Note 4)	Depolarizer mg/in.^{2}	Electrolyte	Current Density ma/in.^{2}	Cell Efficiency % to 0.8 v	Cell Efficiency % to 0.65 v	Comments
63115	Picric Acid	1:2	6	11.1 PVP	D	59.5	2M $Mg(ClO_4)_2$	300	32.4	40.4	
63116	" "	1:1	6	11.1 PVP	D	31.5	2M $AlCl_3$	100	63.5	68.5	B
63116	" "	1:1	6.6 Graphite	11.1 PVP	D	43.1	2M $AlCl_3$	500	46.4	55.7	
63117	" "	1:1	6	11.1 PVP	D	36.6	2M $AlCl_3$	100	85.2	87.5	C
63117	" "	1:1	6	11.1 PVP	D	111	2M $AlCl_3$	500	21.6	26.9	
63119	" "	1:1	6.6 Graphite	11.1 PVP	D	62.8	2M $AlCl_3$	500	41.2	47.2	
63119	" "	1:1	6	11.1 PVP	D	57.8	2M $AlCl_3$	100	59.3	63.6	
63120	" "	1:1	6	11.1 PVP	D	85.4	2M $AlCl_3$	300	32.8	36.1	
63121	" "	1:1	6	11.1 PVP	D	68.4	2M $AlCl_3$	100	85.3	87.6	C
63121	" "	1:1	6	11.1 PVP	D	59.5	2M $AlCl_3$	500	50.4	63.8	C
63125	" "	1:1	6	11.1 PVP	D	64.6	2M $Mg(ClO_4)_2$	100	49.4	61.7	
60661	" "	1:1	6	11.1 PVP	D	58.1	2M $Mg(ClO_4)_2$	200	38.6	57.8	C
60617	c-DNB	1:5	-	11 PVP	N	144	2M $AlCl_3$	400	12	-	
60617	" "	1:3	-	11 PVP	N	35.4	2M $Mg(ClO_4)_2$	100	14.7	41.8	
60616	" "	1:3	-	11 PVP	N	44.1	2M NH_4SCN	100	13.8	22.8	
60616	" "	1:3	-	11 PVP	N	52.4	4M NH_4SCN	100	13.3	29.9	
60616	" "	1:3	-	11 PVP	N	56.7	2M NH_4SCN	100	18.4	27.6	
60616	" "	1:3	-	11 PVP	N	40.9	4M NH_4SCN	100	23.3	38.0	
60614	" "	1:1	-	13 PVP	N	36.5	3M $Mg(ClO_4)_2$	100	19.0	35.6	
60614	" "	1:1	-	13 PVP	N	35.0	8M NH_4SCN	100	29.7	49.5	
60627	" "	1:1	-	16.5 PVP	N	43.8	1.5M NH_4ClO_4	100	17.8	31.7	
60603	" "	1:1	-	13 PVP	N	37.0	2M $Mg(ClO_4)_2$	100	49	-	
60603	" "	1:1	-	13 PVP	N	38.2	2M $Mg(ClO_4)_2$	100	37.6	91	
60603	" "	1:1	-	13 PVP	N	34.8	2M $Mg(ClO_4)_2$	100	20.2	52.7	
60603	" "	1:1	-	13 PVP	N	34.3	2M $Mg(ClO_4)_2$	200	12.5	27.5	
60604	" "	1:1	-	13 PVP	N	42.7	2M $Mg(ClO_4)_2$	100	44.8	91.7	
60604	" "	1:1	-	13 PVP	N	47.8	2M $Mg(ClO_4)_2$	200	21.9	40.2	
60604	" "	1:1	-	13 PVP	N	50.8	2M $Mg(ClO_4)_2$	200	15.4	34.3	
60605	" "	1:1	-	13 PVP	N	39.6	3M $Mg(ClO_4)_2$	100	15.3	28.5	
60605	" "	1:1	-	13 PVP	N	61.7	3M $Mg(ClO_4)_2$	100	19.7	39.4	
60606	" "	1:1	-	13 PVP	N	35.9	2M $Th(NO_3)_4$	100	29.1	38.8	
60607	" "	1:1	-	13 PVP	N	25.2	2M $Th(NO_3)_4$	50	31	37.9	
60607	" "	1:1	-	13 PVP	N	25.5	2M $AlCl_3$	50	51.2	56.2	
60611	" "	1:1	-	13 PVP	N	42.0	2M $AlCl_3$	200	62.2	74.5	
60611	" "	1:1	-	13 PVP	N	45.0	2M $AlCl_3$	300	35	52.5	
60612	" "	1:1	-	13 PVP	N	38.2	2M $Mg(ClO_4)_2$	100	34		Oxalic acid in cathode
55477	" "	1:1	-	9.7 G	N	39.2	1M $MgBr_2$	100	26.6	35.5	D
55477	" "	2:1	-	9.7 G	N	22.2	1M $MgBr_2$	100	31.5	43	D

Table A-2 (continued)

TAPE FORMULATIONS AND STATIC CELL CHARACTERISTICS OF Mg/NITRO COMPOUND

Test No.	Depolarizer	Carbon Depolarizer (Note 1)	Fiber Content of Coating, % (Note 2)	Binder Content of Coating, % (Note 3)	Fabric (Note 4)	Depolarizer mg/in.^{2}	Electrolyte	Current Density ma/in.^{2}	Cell Efficiency % to 0.8 v	Cell Efficiency % to 0.65 v	Comments
55480	o-DNB	1:1	-	9.7 G	N	48.0	1M $MgBr_2$	100	21.6	47	
55480	" "	2:1	-	9.7 G	N	25.2	1M $MgBr_2$	100	31.0	55	
55480	" "	2:1	-	9.7 G	N	38.4	1M $MgBr_2$	100	24.9	52.2	
55482	" "	1:1	-	9.7 G	N	50.5	1M $MgBr_2$ saturated with $Mg(OH)_2$	100	12	29	
55483	" "	1:1	-	9.7 G	N	54.8		100	8.7	17.5	
60643	" "	1:1	-	11.7 PVP	D	114	2M $AlCl_3$	500	17	21	1.2% Pd
60643	" "	1:1	-	11.7 PVP	D	119	2M $AlCl_3$	500	15	-	
60644	" "	1:1	-	11.7 PVP	D	95.8	2M $AlCl_3$	200	38	40.8	1.2% Pd
60644	" "	1:1	-	11.7 PVP	D	104	2M $AlCl_3$	200	29	-	
60644	" "	1:1	-	11.7 PVP	D	82.5	2M $Mg(ClO_4)_2$	200	10		1.2% Pd
60645	" "	1:1	-	11.7 PVP	D	131	2M $Mg(ClO_4)_2$	200	9.3	17	
60651	" "	1:1	-	11.7 PVP	D	105	2M $AlCl_3$	200	41.3	51.3	Pd
60651	" "	1:1	-	11.7 PVP	D	113	2M $AlCl_3$	200	43.2	-	Pd
60652	" "	1:1	-	11.7 PVP	D	174	2M $AlCl_3$	500	20.7	25.3	Pd
60653	" "	1:1	-	11.7 PVP	D	171	2M $AlCl_3$	200	32.7	36.4	Pd
60640	Picric Acid	1:1	-	17 PVP	D	57.3	2M $Mg(ClO_4)_2$	300	42	-	
60640	" "	1:1	-	17 PVP	D	52.7	2M $Mg(ClO_4)_2$	300	≈ 25	-	
60641	" "	1:4	-	17 PVP	D	57.8	2M $Mg(ClO_4)_2$	300	≈ 10	-	
60643	" "	1:4	-	17 PVP	D	112	2M $Mg(ClO_4)_2$	100	23.6	27.2	
60643	" "	1:4	-	17 PVP	D	105	2M $AlCl_3$	100	19	-	
52295	" "	1:1	-	11.7 PVP	D	71.5	2M $AlCl_3$	100	43.6	48.1	
52298	" "	1:1	-	11.7 PVP	D	116	2M $AlCl_3$	200	41.3	44.8	
60649	2,5-Dinitrothiophene	1:1	-	11.7 PVP	D	139	2M $AlCl_3$	500	9.7	22.7	
60649	" "	1:1	-	11.7 PVP	D	64.2	2M $AlCl_3$	100	43.7	50.7	
60649	" "	1:1	-	11.7 PVP	D	62.7	2M $Mg(ClO_4)_2$	100	13	13	
60650	" "	1:1	-	11.7 PVP	D	146	2M $Mg(ClO_4)_2$	100	18.5	19.4	
63125	" "	1:1	6.5	11.7 PVP	D	48.0	2M $AlCl_3$	100	37.5	39.4	
63125	" "	1:1	6.5	11.7 PVP	D	59.0	2M $Mg(ClO_4)_2$	100	13	13	
63101	2,4,5-Trinitrotoluene	1:1	-	11.7 PVP	D	44.7	2M $AlCl_3$	200	0		
63101	" "	1:1	-	11.7 PVP	D	128	2M $AlCl_3$	500	0		
63102	" "	1:1	-	11.7 PVP	D	43.8	2M $Mg(ClO_4)_2$	100	14.2	-	
52298	2,4-Dinitrothiophene	1:1	-	11.7 PVP	D	55.1	2M $Mg(ClO_4)_2$	200	8	-	
52299	" "	1:1	-	11.7 PVP	D	56.0	2M $Mg(ClO_4)_2$	100	14.5	15.5	
52299	" "	1:1	-	11.7 PVP	D	123	2M $AlCl_3$	500	0		
52230	" "	1:1	-	11.7 PVP	D	116	2M $AlCl_3$	200	29.5	35.7	

Table A-2 (continued)

TAPE FORMULATIONS AND STATIC CELL CHARACTERISTICS OF Mg/NITRO COMPOUND

Test No.	Depolarizer	Carbon Depolarizer (Note 1)	Fiber Content of Coating, % (Note 2)	Binder Content of Coating, % (Note 3)	Fabric (Note 4)	Depolarizer mg/in.^{2}	Electrolyte	Current Density ma/in.^{2}	Cell Efficiency % to 0.8 v	Cell Efficiency % to 0.65 v	Comments
60633	3,6-Dinitro-phthalic Acid	1:1	-	11.7 PVP	D	59.4	2M $Mg(ClO_4)_2$	100	44	-	
60633	" "	1:1	-	11.7 PVP	D	71.6	2M $AlCl_3$	500	9	-	
60634	" "	1:1	-	11.7 PVP	D	66.1	2M $Mg(ClO_4)_2$	100	17	26.1	
60634	" "	1:1	-	11.7 PVP	D	34.8	2M $Mg(ClO_4)_2$	100	15	-	
60650	1-Carboxymethyl-3,3,5,5-tetra-nitropiperidine	1:1	-	11.7 PVP	D	118	2M $AlCl_3$	100	14	-	
60651	" "	1:1	-	11.7 PVP	D	71.3	2M $AlCl_3$	100	4	-	
60650	" "	1:1	-	11.7 PVP	D	67.3	2M $Mg(ClO_4)_2$	100	14	15.5	
55491	o-DNB	1:1	-	11.7 PVP	N	28.3	2M $Mg(ClO_4)_2$	100	24.6	55.2	24°C
55491	"	1:1	-	11.7 PVP	N	21.0	2M $Mg(ClO_4)_2$	100	33.0	57.8	24°C
55491	"	1:1	-	11.7 PVP	N	19.9	2M $Mg(ClO_4)_2$	100	39.3	74.2	40°C
55491	"	1:1	-	11.7 PVP	N	18.4	2M $Mg(ClO_4)_2$	100	56.7	70.8	42°C
55493	"	1:1	-	11.7 PVP	N	19.2	2M $Mg(ClO_4)_2$	100	36.2	72.4	53°C
55493	"	1:1	-	11.7 PVP	N	23.5	2M $Mg(ClO_4)_2$	100	37.0	85.1	59°C

Table A-3

PHYSICAL DATA ON CAPSULES PREPARED FOR PERMEABILITY TESTS

Capsule Number	Capsule Material	Liquid	Conditions *	Film Thickness (mils)	Film Weight (g)	Total Weight (g)	Liquid Weight (g)	I. D. (in.)	O. D. (in.)	Flat Width (in.)	Length (in.)	Approximate Thickness (inches)	Surface Area (in.²)	Initial Payload Weight (g)
1	Al-Mylar	water	A	2.5	-	0.1980	-			0.5	0.5	0.062	0.5	83.8
2	Polyethylene	water	A	1	-	20.421	-			1.5	3.0	0.25	9.3	99.1
3	Aclar	water	A	5	-	1.0388	-			1.5	0.5	-	0.77	72.2
4	Aclar	water	A	2	-	5.4165	-			0.65	4.5	0.15	5.75	75.6
5	FEP-Teflon	water	A	2	-	0.7409	-			0.25	3.65	0.1	1.82	43.3
6	Aclar	water	A	2	-	1.8019	-			0.3	4.0	0.17	2.4	69.5
7	Kel-F 81	water	A	3.5	0.3346	1.5352	1.2006			0.12	7.0	-	8.6	78.2
8	Al-Mylar	water	A	2.5	0.2230	1.2282	1.0052			0.5	4.8	0.05	4.8	81.9
E1	Aclar	water	A	2	3.4873	15.5838	12.0965			1.75	14.0	-	49	77.7
E2	Aclar	water	A	2	1.7440	13.7782	12.0342			0.88	14.0	-	24.5	87.3
E3	Aclar	water	A	2	3.1337	36.9230	-			0.88	26.0	-	45.5	91.5
E4	Al-Mylar	water	A	2.5	1.2204	13.4590	12.2386			1.0	14.0	0.08	28	90.9
E5	Al-Mylar	water	A	2.5	1.9189	13.6187	11.6998			1.75	14.0	-	49	85.9
E6a	Aclar	water	A	2	0.7400	2.9273	2.1873			0.62	6.5	-	8.1	74.7
E7a	Al-Mylar	water	A	2.5	0.4780	2.5493	2.0713			0.94	6.0	0.05	11.2	81.4
E8	Aclar	water	A	2	0.7560	5.4208	4.6648			0.56	9.0	-	10.1	86.1
E9	Aclar	water	A	2	1.0701	6.3753	5.3052			1.75	8.25	-	29	83.2
E14a	Polyethylene	water	A	2	0.3036	2.7602	2.4566			0.75	6.0	0.11	9	89.0

* Conditions: A - Ambient
B - Vacuum at room temperature
C - Vacuum at 39°C

Table A-3 (continued)

PHYSICAL DATA ON CAPSULES PREPARED FOR PERMEABILITY TESTS

Capsule Number	Capsule Material	Liquid	Conditions *	Film Thickness (mils)	Film Weight (g)	Total Weight (g)	Liquid Weight (g)	I. D. (in.)	O. D. (in.)	Flat Width (in.)	Length (in.)	Approximate Thickness (inches)	Surface Area (in.^{2})	Initial Payload Weight (g)
E15	Polyethylene	water	A	2	0.8158	12.0105	11.1947			0.88	14.0	0.11	24.5	93.2
E16	Polyethylene	water	A	2	1.7005	12.8420	11.1415			1.75	14.0	-	49	86.7
E17	TFE-Teflon	water	A	2	2.0313	13.9479	11.9166			1.0	14.0	-	28	85.4
E18	TFE-Teflon	water	A	2	0.7393	2.9372	2.1979			0.75	6.0	-	9	74.9
E19	TFE-Teflon	water	A	2	3.1540	14.7175	11.5635			1.5	14.0	-	42	78.6
E20	FEP-Teflon	water	A	2	0.7910	7.7430	6.9520			0.81	6.0	0.17	10	89.8
E21	FEP-Teflon	water	A	3	0.8780	5.5890	4.7110			0.62	7.0	0.17	8.8	84.3
E22	TFE-Teflon	water	A	2	-	12.1148	-			1.12	4.0	0.45	9	-
E23	Kel-F 81	water	C	3.5	0.1306	0.6165	0.4859		0.12		2.88	-	1.12	78.8
E24	Kel-F 81	water	B	3.5	-	0.6837	-		0.12		3.0	-	1.18	-
E25	Al-Mylar	water	B	2.5	-	4.8883	-			0.75	6.0	-	9	-
E26	TFE-Teflon	water	B	2	-	7.2396	-			1.0	4.5	-	9	-
E27	Aclar	2 M $Mg(ClO_4)_2$	B	2	-	1.7497	-			0.43	7.5	0.03	6.3	-
E28	Al-Mylar	2 M $Mg(ClO_4)_2$	B	2.5	-	2.3568	-			0.5	7.25	0.06	7.3	-
E28a	Kel-F 81	water	B	2	-	0.1019	-		.047		3.0	-	0.044	-
E30	Al-Mylar	2 M $Mg(ClO_4)_2$	C	2.5	-	2.7925	-			0.5	7.25	0.08	7.3	-
E32	Al-Mylar	2 M $Mg(ClO_4)_2$	A	2.5	-	2.5605	-			0.5	7.25	0.07	7.3	-
E33	Al-Mylar	2 M $AlCl_3$	A	2.5	0.3321	2.7017	2.3696			0.56	6.0	-	6.8	87.7
E34	Al-Mylar	2 M $AlCl_3$	C	2.5	0.3246	2.6078	2.2832			0.56	6.0	-	6.8	87.3
E35	Al-Mylar	2 M $AlCl_3$	B	2.5	0.3321	2.6751	2.3430			0.56	6.0	-	6.8	87.5

Table A-3 (continued)

PHYSICAL DATA ON CAPSULES PREPARED FOR PERMEABILITY TESTS

Capsule Number	Capsule Material	Liquid	Conditions *	Film Thickness (mils)	Film Weight (g)	Total Weight (g)	Liquid Weight (g)	I. D. (in.)	O. D. (in.)	Flat Width (in.)	Length (in.)	Approximate Thickness (inches)	Surface Area (in.^{2})	Initial Payload Weight (%)
E36	Kel-F 81	2 M $AlCl_3$	C	3.5	-	0.6074	-		0.12		2.25	-	0.88	-
E37	FEP-Teflon	water	A	2	0.2878	1.0780	0.7902			0.36	4.0	0.08	3	73.2
E38	Aclar	2 M $Mg(ClO_4)_2$	C	2	0.7635	3.2884	2.5249			0.56	6.20	0.06	7	76.7
E39	Aclar	2 M $Mg(ClO_4)_2$	C	2	0.7479	2.9872	2.2393			0.56	6.20	0.06	7	75.1
E40	Aclar	2 M $Mg(ClO_4)_2$	B	2	0.7750	3.1044	2.3294			0.56	6.20	0.06	7	75.1
E41	Aclar	2 M $Mg(ClO_4)_2$	B	2	0.7152	2.9883	2.2731			0.56	6.20	0.06	7	76.0
E42	Aclar	2 M $Mg(ClO_4)_2$	A	2	0.7730	3.2693	2.4963			0.56	6.20	0.06	7	76.3
E43	Aclar	2 M $Mg(ClO_4)_2$	A	2	0.7018	3.2358	2.5340			0.56	6.20	0.06	7	78.2
E44	Kel-F 81	water	C	3	0.2733	1.4907	1.2174	0.12			6.0	-	2.36	81.7
E45	Kel-F 81	2 M $Mg(ClO_4)_2$	C	3	0.2674	1.8456	1.5782	0.12			6.0	-	2.36	85.3
E46	Kel-F 81	water	B	3	0.2703	1.5016	1.2313	0.12			6.0	-	2.36	82.1
E47	Kel-F 81	2 M $Mg(ClO_4)_2$	B	3	0.2704	1.8893	1.6189	0.12			6.0	-	2.36	85.6
E48	Kel-F 81	water	A	3	0.2833	1.6218	1.3385	0.12			6.0	-	2.36	82.6
E49	Kel-F 81	2 M $Mg(ClO_4)_2$	A	3	0.2696	1.8540	1.5844	0.12			6.0	-	2.36	85.6
E50	Kel-F 81	2 M $AlCl_3$	A	3	0.2798	1.8033	1.5235	0.12			6.0	-	2.36	84.6
E51	Kel-F 81	2 M $AlCl_3$	B	3	0.2760	1.8123	1.5363	0.12			6.0	-	2.36	84.9
E52	Kel-F 81	2 M $AlCl_3$	C	3	0.2788	1.8166	1.5378	0.12			6.0	-	2.36	84.5
E53	Kel-F 81	2 M $AlCl_3$	C	3	0.2739	1.7376	1.4637	0.12			6.0	-	2.36	84.1
E54	Kel-F 81	37% KOH	A	3	0.2785	1.8743	1.5958	0.12			6.0	-	2.36	85.3
E55	Kel-F 81	37% KOH	A	3	0.2792	1.8506	1.5714	0.12			6.0	-	2.36	84.9

Table A-3 (continued)

PHYSICAL DATA ON CAPSULES PREPARED FOR PERMEABILITY TESTS

Capsule Number	Capsule Material	Liquid	Conditions *	Film Thickness (mils)	Film Weight (g)	Total Weight (g)	Liquid Weight (g)	I. D. (in.)	O. D. (in.)	Flat Width (in.)	Length (in.)	Approximate Thickness (inches)	Surface Area (in.^{2})	Initial Payload Weight (%)
E56	Kel-F 81	37% KOH	B	3	0.2792	1.8760	1.5968	0.12			6.0	-	2.36	84.9
E57	Kel-F 81	37% KOH	B	3	0.2763	1.8892	1.6129	0.12			6.0	-	2.36	85.3
E58	Kel-F 81	37% KOH	C	3	0.2764	1.8361	1.5597	0.12			6.0	-	2.36	84.8
E59	Kel-F 81	37% KOH	C	3	0.2807	1.8330	1.5523	0.12			6.0	-	2.36	84.8
E60	Kel-F 81	37% KOH	C	3	0.2850	1.9113	1.6263	0.12			6.0	-	2.36	85.2
E61	Kel-F 81	37% KOH	C	3	0.2790	1.8954	1.6164	0.12			6.0	-	2.36	85.1