ANNUAL REPORT FOR 1979 ON RESEARCH, DEVELOPMENT AND DEMONSTRATION OF LEAD-ACID BATTERIES FOR ELECTRIC VEHICLE PROPULSION

Contract No. 31-109-38-4205

by

Globe Battery Division
Globe-Union Inc.
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Annual Report for 1979 on
RESEARCH, DEVELOPMENT AND DEMONSTRATION OF
LEAD-ACID BATTERIES FOR
ELECTRIC VEHICLE PROPULSION

Prepared for
The Office for Electrochemical Project Management
Argonne National Laboratory
Under Contract No. 31-109-38-4205

By
Globe-Union Incorporated
Milwaukee, Wisconsin

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June 1980
GLOBE BATTERY DIVISION
JOHNSON CONTROLS, INC.
5757 North Green Bay Ave.
Milwaukee, Wisconsin 53201

- ANNUAL REPORT -
1979

RESEARCH, DEVELOPMENT and DEMONSTRATION of
LEAD-ACID BATTERIES for ELECTRIC VEHICLE
PROPULSION

SUBMITTED UNDER CONTRACT NO. 31-109-38-4205

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1.0 EXECUTIVE SUMMARY - TECHNICAL STATUS

This report describes work performed by Globe-Union, Inc., a Subsidiary of Johnson Controls, Inc., under Contract Number 31-109-38-4205 to Argonne National Laboratory for the development of lead-acid batteries for electric vehicle propulsion. The period of performance of the work covered in this report is October 1, 1978 to September 30, 1979, the 1979 fiscal year.

Globe-Union's approach for development of both the Improved-State-of-the-Art (ISOA) and Advanced lead-acid batteries is three pronged. This approach concentrates on simultaneous optimization of battery design, materials and manufacturing processing. In the battery design area Globe is utilizing its proven computerized battery modeling programs to develop the most efficient current collecting system and active materials utilization for the electric vehicle battery application. In all design areas compatibility with efficient mass production techniques has been a prime consideration to insure that these batteries will be commercializable products which meet the cost goals of the program.

The materials development program covers virtually every battery component, with heavy emphasis on improvement of the active and conductive cell components. The results of this extensive program will primarily, but not solely, influence the Advanced battery and significantly contribute to the achievement of its ambitious life and specific energy goals.

Process development efforts involve both the design of a manufacturing system for these batteries which utilizes existing high speed production equipment and the optimization of those processes which are known to have a critical effect on the capacity and life of the product. Such processes include many of the steps in the preparation of both positive and negative electrodes and battery formation.

The 1979 fiscal year saw the achievement of significant progress in Globe-Union's program. Some of the major accomplishments of the year are as follows:

- A major module redesign effort was completed to incorporate a reliable electrolyte circulation system into our ISOA battery. Our original plans projected incorporation of electrolyte circulation only in the Advanced battery, but the advantages of such a system in improved electrical performance and life
precipitated the decision to include it in the ISOA design.

- The single point watering and venting system design was completed.
- Separator system design modifications were evaluated.
- Critical charging parameters were determined and charger design initiated.
- Design of composite grids for the Advanced battery was initiated.
- Commercially available and modified negative active material expanders were evaluated by the new microelectrode techniques, with strong indications that special expanders may be prepared which will improve the capacity and life of the negative electrode in deep cycle applications.
- A series of positive and negative grid alloys were characterized and the best candidates selected.
- Optimization studies were initiated in the paste preparation, curing and formation process areas.
- Cycle test facilities were completed and extensive cell testing initiated.
- ISOA cells were delivered to the National Battery Test Laboratory which exceeded the ISOA goals for specific energy, specific power, and roundtrip efficiency.

In retrospect then, it can be stated that the progress of this program in fiscal 1979, the first full year under the contract, has been substantial and has laid a sound base for continued success.
## TECHNICAL GOALS

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<th>ISOA</th>
<th>Advanced</th>
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<td>1. Battery Capacity (kW/hr)(^a) (100% rated)</td>
<td>20-30</td>
<td>30-40</td>
</tr>
<tr>
<td>2. Battery Dimension (cm H x cm W x cm L)</td>
<td>29.5x40.6x264</td>
<td>29.5x40.6x264</td>
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<tr>
<td>3. Specific Energy(^a) (W-hr/kg)</td>
<td>40</td>
<td>60</td>
</tr>
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<td>4. Specific Power (W/kg) Peak Battery-15 sec. avg.</td>
<td>100</td>
<td>150</td>
</tr>
<tr>
<td>5. Duty Cycle</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Charge (hr)</td>
<td>4-8</td>
<td>4-8</td>
</tr>
<tr>
<td>Discharge (hr)</td>
<td>2-4</td>
<td>2-4</td>
</tr>
<tr>
<td>6. Lifetime Deep Discharges(^b)</td>
<td>800</td>
<td>1000</td>
</tr>
<tr>
<td>7. Price/Energy(^c) ($/kW-hr)</td>
<td>50</td>
<td>40</td>
</tr>
<tr>
<td>8. Energy Efficiency (%)(^d)</td>
<td>&gt;60</td>
<td>&gt;60</td>
</tr>
<tr>
<td>9. Typical Installation Voltage(^e)</td>
<td>96V-120V</td>
<td>96V-120V</td>
</tr>
</tbody>
</table>

\(^a\)C/3 rate discharge; 8-hr charge. The 3 hr rate is defined as a 3 hr capacity of a battery to 1.75V/cells at 26.7\(^\circ\)C (80\(^\circ\)F).

\(^b\)80% depth of discharge from rated capacity.

\(^c\)Price FOB to auto manufacturer with production ~10,000/yr.

\(^d\)At battery terminals including auxiliary equipment, excluding charger.

\(^e\)For compact passenger car.
WBS 2.0 Expander Research Program

By the beginning of FY'79, the investigations had produced a microelectrode testing technique that allowed continuous cycling (discharge-charge) regime of numerous candidate expander materials without the need of constructing full size EV-type cells. In fact, it would have been an impossible task to investigate the many compounds and the multitude of test parameters if cells had to be constructed. From these studies came two performance 'evaluators', namely:

- High Rate Index - a measurement of expander action on the discharge behavior of the electrode over a wide range of current densities.
- Cycle Life Index - a quantitative measurement of the expander activity in maintaining electrode capacity during a well defined mode of cycling.

Concurrently, Globe-Union had progressed to the point where the data generated was collected and organized with a Doric 220 Data Logger which was controlled by software executed on a PDP 11/34 mini-computer.

Task 2.1 Centerline Expander Studies

Tests performed at equivalent lignosulfonate concentrations in 1.250 specific gravity acid have shown the superiority of the Type A lignosulfonate during high rate index testing and the Type B1 lignosulfonate in the cycle life index testing. SEM examination of the cycled electrodes has indicated considerable influence of all the lignosulfonates tested on the structure of the negative lead electro-deposit. Without lignosulfonates, the lead deposit is made up of discrete elongated particles (~0.8μ X 3μ) fused together in a honeycomb structure. Acid with lignosulfonates shows a finer particle (~0.5μ X 1μ) and less consolidation.

A technique used in organic chemistry to identify complex molecules was used to analyze solutions of Types B, B1 and A lignosulfonates. Comparative UV and IR spectra at pH2 and pH12 of the same materials were studied. In each case, the UV absorbance increased with increasing lignin structure. The IR spectra indicated the presence of OH-, aromatic, phenolic, and -c-o-c groups in each compound.
Additional differences among the samples were noted:

<table>
<thead>
<tr>
<th></th>
<th>Type B</th>
<th>Type B1</th>
<th>Type A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ash Content (%)</td>
<td>11.5</td>
<td>16.4</td>
<td>33.7</td>
</tr>
<tr>
<td>UV Absorbance at 28μm (+ 25%)</td>
<td>0.025</td>
<td>0.016</td>
<td>0.012</td>
</tr>
<tr>
<td>Organic S (%)</td>
<td>0.3</td>
<td>0.3</td>
<td>2.93 ± 10%</td>
</tr>
<tr>
<td>-OCH₃ (%)</td>
<td>13.7</td>
<td>10.4</td>
<td>8.0</td>
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In view of the significant differences in organic content indicated by ash analysis, the inverse relationship of the UV absorbance, which is due to the organic fraction, is understandable. However correcting the UV absorbances to unit weight of ash-free organic material suggests possible structural differences, particularly in Type B against Types B1 or A lignosulfonates.

The effect of Type B lignosulfonate concentration on the electrochemical performance of the lead electrode, in 1.250 specific gravity acid (22°C), has been determined. The discharge capacity was at a maximum at the c₁ concentration for discharge currents up to 500 μA/cm². At 660 μA/cm² the discharge capacity increased exponentially up to the c₂ Type B lignosulfonate concentration. The different behaviors at two discharge rates are believed associated with the different morphologies of the lead sulfate formed in each case.

Measurements of the cycle life index showed that, up to an optimum concentration (c₂), Type B lignosulfonate enhances the cycling ability of the electrode, while higher concentrations decreases it again. The B1 and A lignosulfonates are being similarly studied.

Recent tests on the lignosulfonates revealed similarities and differences (Table 1):
<table>
<thead>
<tr>
<th>Type of Analysis</th>
<th>Control</th>
<th>Type B1</th>
<th>Type B</th>
<th>Type A</th>
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<tr>
<td><strong>Chemical</strong></td>
<td></td>
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<td>Ash, %</td>
<td>16.4</td>
<td>11.5</td>
<td>33.7</td>
<td></td>
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<tr>
<td>Organics, %</td>
<td>&lt;0.3</td>
<td>&lt;0.3</td>
<td></td>
<td>2.93</td>
</tr>
<tr>
<td>-OCH₃, %</td>
<td>13.7</td>
<td>10.4</td>
<td>8.0</td>
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<td><strong>Electrode Capacity in Milli-coulombs</strong></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(with c₁ expander concentration in 1.250 sp.gr. H₂SO₄) at disch. current of 200 μA</td>
<td>258.1</td>
<td>502.2</td>
<td>498.0</td>
<td>452.4</td>
</tr>
<tr>
<td>400 μA</td>
<td>178.2</td>
<td>294.2</td>
<td>312.6</td>
<td>283.4</td>
</tr>
<tr>
<td>750 μA</td>
<td>124.6</td>
<td>231.1</td>
<td>184.1</td>
<td>202.5</td>
</tr>
<tr>
<td>1000 μA</td>
<td>103.6</td>
<td>138.0</td>
<td>144.0</td>
<td>178.0</td>
</tr>
<tr>
<td><strong>Electrode Capacity in Milli-coulombs</strong></td>
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<td>(with c₂ expander concentration in 1.250 sp.gr. H₂SO₄) at disch. current</td>
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<td>397.2</td>
<td>530.4</td>
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<tr>
<td>400 μA</td>
<td>178.2</td>
<td>276.0</td>
<td>213.0</td>
<td>423.6</td>
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<tr>
<td>750 μA</td>
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<td>170.8</td>
<td>158.9</td>
<td>270.0</td>
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<tr>
<td>1000 μA</td>
<td>103.6</td>
<td>137.4</td>
<td>167.1</td>
<td>264.0</td>
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<td><strong>Cycle life index</strong></td>
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<tr>
<td>(c₁final/cin)⁻¹</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>at 145 cycle based on cin., capacity at end of development as function of expander concentration</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>c₀</td>
<td>-.35</td>
<td>-.35</td>
<td>-.35</td>
<td></td>
</tr>
<tr>
<td>c₁</td>
<td>.36</td>
<td>.43</td>
<td>.66</td>
<td></td>
</tr>
<tr>
<td>c₂</td>
<td>.69</td>
<td>.68</td>
<td>1.05</td>
<td></td>
</tr>
<tr>
<td>c₃</td>
<td>.79</td>
<td>.79</td>
<td>.41</td>
<td></td>
</tr>
<tr>
<td>c₄</td>
<td>.36</td>
<td>.81</td>
<td>-0.36</td>
<td></td>
</tr>
<tr>
<td>c₅</td>
<td>.17</td>
<td></td>
<td>-.20</td>
<td></td>
</tr>
</tbody>
</table>
Spectrographic analyses have revealed sodium to be the major inorganic in each of the lignosulfonates with sodium concentrations greater than 2%. Type A lignosulfonate has 0.01% iron and 0.006% manganese compared to <.007% iron and <.002% manganese in both the Bl and B lignosulfonates.

UV, IR and ESR tests have shown strong similarities among the spectral behavior of the lignosulfonates. However, a differential UV at pH12 versus pH2 has picked up dissimilarities between Type A and the Type B or Bl lignosulfonates. (See Figure 1). This indicates a greater phenolic group content in the Type A lignosulfonate versus the others.

The effect of expander concentration on the discharge activity of the lead electrode is summarized in Table 2.

<table>
<thead>
<tr>
<th>Discharge Rate (µA)</th>
<th>Control</th>
<th>Type Bl c1</th>
<th>c2</th>
<th>Type B c1</th>
<th>c2</th>
<th>Type A c1</th>
<th>c2</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>258</td>
<td>502</td>
<td>544</td>
<td>498</td>
<td>379</td>
<td>452</td>
<td>530</td>
</tr>
<tr>
<td>400</td>
<td>178</td>
<td>294</td>
<td>276</td>
<td>313</td>
<td>214</td>
<td>283</td>
<td>424</td>
</tr>
<tr>
<td>750</td>
<td>125</td>
<td>231</td>
<td>171</td>
<td>184</td>
<td>159</td>
<td>203</td>
<td>270</td>
</tr>
<tr>
<td>1000</td>
<td>104</td>
<td>138</td>
<td>137</td>
<td>144</td>
<td>167</td>
<td>178</td>
<td>264</td>
</tr>
</tbody>
</table>

The above table shows the current density dependent behavior of the discharge capacity as a function of lignosulfonate concentration in 1.250 specific gravity acid at 75 ± 3°F for the Bl, B and A lignosulfonates. At the c1 lignosulfonate concentrations the discharge activities remain close to one another. However, at the c2 concentration and current densities = 200µA, the Type A out-performs Type Bl and the Type B lignosulfonates.

Tables 1 and 3 shows the effect of lignosulfonate concentration in 1.250 specific gravity acid (75 ± 3°F) on the cycle life index. The Type A again shows superiority over the Bl and B at the c1 concentration or lower.
Figure 1 Differential UV-Spectra of lignosulfonate candidates at solution pH 12 versus reference pH 2.
The variations in optimum concentration of lignosulfonates in cycling activities reflects their differences in surfactant activities; i.e., the concentration dependent adsorption-desorption kinetics on the electrode surface. Higher lignosulfonate concentrations in solution favors a stronger adsorption of lignosulfonates on the electrode surface. This in turn restricts the lead deposition sites and hinders the charging (electroconversion of PbSO4→Pb) operation. This subsequently shows up in a sharp decrease in cycling activity. The adsorption behavior of the test lignosulfonate candidates on the lead surface may be associated with their intrinsic properties, such as, the functional groups associated with the lignin molecule, molecular weight distribution and adsorptivity of various species on the lead surface.

The effect of Type B lignosulfonate in 1.050 specific gravity on the electrode capacity during development cycles and the capacity maintenance characteristics of these electrodes during test cycles, as a function of acid concentration, have been studied. This study has direct implications in explaining the lignosulfonate's role in lead-acid battery formation in low gravity acid and subsequent deep cycling of the same in relatively higher gravity acid. Table 4 and 5 and Figure 2 show summary data of the effect of the $c_1$ concentration of Type B lignosulfonate on the electrode capacity during development cycles and capacity retention characteristics during subsequent cycling as a function of acid concentration. The effect of lignosulfonate concentration in the development acid is shown in Table 5 and Figures 3 and 4.
Table 4

The Effect of the Concentration of Type B Lignin in the Developing Acid on the Electrode Capacity Development and Subsequent Capacity Maintenance During Cycling

<table>
<thead>
<tr>
<th>Electrolyte</th>
<th>Cycle No.</th>
<th>Electrode Capacity (Coulombs)</th>
<th>Electrolyte Concen., Sp.Gr.</th>
<th>Capacity at 145th Cycle, C&lt;sub&gt;final&lt;/sub&gt; (Coulombs)</th>
<th>Cycle Life Index C&lt;sub&gt;final&lt;/sub&gt;-C&lt;sub&gt;initial&lt;/sub&gt; C&lt;sub&gt;initial&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.050 H&lt;sub&gt;2&lt;/sub&gt;S&lt;sub&gt;4&lt;/sub&gt;</td>
<td>69</td>
<td>1.398</td>
<td>1.050</td>
<td>1.667</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>74</td>
<td>1.211</td>
<td>1.150</td>
<td>1.061</td>
<td>-0.12</td>
</tr>
<tr>
<td></td>
<td>74</td>
<td>1.147</td>
<td>1.250</td>
<td>0.879</td>
<td>-0.23</td>
</tr>
<tr>
<td></td>
<td>124</td>
<td>1.795</td>
<td>1.350</td>
<td>0.670</td>
<td>-0.63</td>
</tr>
<tr>
<td>1.050 H&lt;sub&gt;2&lt;/sub&gt;S&lt;sub&gt;4&lt;/sub&gt; with concentration Type B</td>
<td>116</td>
<td>3.861</td>
<td>1.050</td>
<td>3.450</td>
<td>-0.11</td>
</tr>
<tr>
<td></td>
<td>134</td>
<td>3.745</td>
<td>1.150</td>
<td>2.764</td>
<td>-0.26</td>
</tr>
<tr>
<td></td>
<td>136</td>
<td>3.522</td>
<td>1.250</td>
<td>2.105</td>
<td>-0.40</td>
</tr>
<tr>
<td></td>
<td>114</td>
<td>3.713</td>
<td>1.350</td>
<td>1.378</td>
<td>-0.63</td>
</tr>
</tbody>
</table>
Table 5
The Effect of Expander Concentration in the Developing Acid on the Electrode Capacity During Development and Subsequent Capacity Maintenance During Cycling in 1.250 sp.gr. H$_2$SO$_4$.

<table>
<thead>
<tr>
<th>Concentration of Type B in 1.050 sp.gr. H$_2$SO$_4$</th>
<th>Electrode Development</th>
<th>Test Cycles in 1.250 sp.gr. H$_2$SO$_4$</th>
<th>Cycle Life Index, $C_{final} - C_{initial}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Cycle No.</td>
<td>Electrode Capacity ($C_{initial}$) Coulombs</td>
<td>Electrode Capacity at 145th ($C_{final}$) Coulombs</td>
</tr>
<tr>
<td>0</td>
<td>144</td>
<td>1.562</td>
<td>1.110</td>
</tr>
<tr>
<td>$c_0$</td>
<td>144</td>
<td>3.823</td>
<td>2.244</td>
</tr>
<tr>
<td>$c_1$</td>
<td>136</td>
<td>3.522</td>
<td>2.105</td>
</tr>
<tr>
<td>$c_2$</td>
<td>144</td>
<td>2.250</td>
<td>2.159</td>
</tr>
<tr>
<td>$c_3$</td>
<td>144</td>
<td>1.690</td>
<td>2.478</td>
</tr>
<tr>
<td>$c_4$</td>
<td>144</td>
<td>1.288</td>
<td>2.438</td>
</tr>
</tbody>
</table>
Figure 2 The capacity degradation of the lead electrode with and without lignosulfonate during cycling as a function of acid concentration.
Figure 3 Potential sweep cycling voltammograms as a function of Type B lignosulfonate concentrations
Figure 4. Effect of Type B lignosulfonate concentration in 1.050 specific gravity acid on the developed electrode capacity and steady state capacity when cycled in 1.250 specific gravity H2SO4.
Test results reveal several interesting phenomena associated with the expander in the acid in a lead-acid battery:

1) The electrode capacity build-up during the development cycle is greatly influenced by small concentrations of lignosulfonate (Tables 4 and 5). This capacity increase is, to a greater extent, caused by the lignosulfonate's surface adsorptive influence in the charge inhibition process (Figure 3) which in turn helps in increasing the electrode's surface area. This is in agreement with the observation noted in the literature (B. Mahato, J. Electrochem Soc. 124, 1663 (1977); J.R.Pierson, P. Guralusky, A.C. Simon, S.M. Caulder, ibid 117, 1463 (1970); 121,463 (1974)).

2) During deep discharge cycling the lead electrode loses capacity almost linearly with the increasing acid concentration of the test solution (Table 4 and Figure 2). The absolute capacity loss is greater for electrodes developed in a solution with lignosulfonate than without lignosulfonate. This greater loss could be associated with the higher initial capacity. The rate of capacity loss (or cycle life index) is less for electrodes developed in acid with lignosulfonate than the control (Figure 2).

3) The lignosulfonate concentration in the developing acid appears to influence the electrode performance in two ways (Table 5 and Figures 3 and 4).

- It affects the capacity build-up development.

- It remains adsorbed on the electrode matrix which subsequently influences the capacity maintenance behavior during test cycling.

At low concentrations, the lignosulfonate helps to build up high capacity (or surface area) at the end of development and carries very little surface adsorbed material. At high concentrations it greatly restricts the capacity build-up during the development cycle and carries a significant amount of surface adsorbed material. During test cycling the adsorbed lignosulfonate, which is more concentrated on the surface of the
electrode formed in solutions with higher lignosulfonate concentrations, begins to influence the cyclic performance of the electrode in such a way that the electrode overcomes its low initial capacity and attains a steady state capacity close to the electrodes with high initial capacity. As a result, the electrodes developed with $c_1$ to $c_4$ lignosulfonate have shown almost identical steady state test cycling capacity (Figure 4).

4) There is an indication that surface desorption of the adsorbed lignosulfonate on the lead electrode surface occurs at higher end of the charge (cathodic) potential. (See Development Voltammograms at the $c_2$, $c_3$ and $c_4$ lignosulfonate concentrations, Figure 3).

The study of the effect of the Type A lignosulfonate concentration in the developing 1.050 specific gravity $H_2SO_4$ on micro-electrode capacity, both during development cycling and in subsequent cycling in pure 1.250 specific gravity $H_2SO_4$, was completed. It was determined that:

1) The $c_1$ concentration of Type A lignosulfonate in formation acid offers an optimum capacity at the end of development.

2) Electrodes with maximum developed capacity have maintained higher capacity during test cycling in spite of a more rapid capacity loss (lower cycle life index) than others.

3) The Type A lignosulfonate helps to develop electrode capacity better than Type B lignosulfonate during development but offers less capacity maintenance than the Type B during test cycling in 1.250 specific gravity $H_2SO_4$.

These results are summarized in Table 6 and Figure 5.

The SEM examination of 1000 $\mu$A discharged electrode ($\sim 1.57$ cm$^2$) surface has noted that the presence of Type B lignosulfonate in 1.250 specific gravity $H_2SO_4$ influences the particle size of the precipitated lead sulfate. The average particle size was about 2 microns without and 1 micron with lignosulfonates in solution (Figure 6). The smaller particle size in presence of lignosulfonates was caused by isotropic surface adsorption of
expander on the growing lead sulfate crystals. In spite of the smaller lead sulfate particle size, the delay in electrode passivation (Table 7, the greater discharge time in presence of additive) process had indicated a secondary role of the lignosulfonates in controlling the electrode kinetics. This could be related to its influence on the reacting interface by promoting the dissolution kinetics of lead and its role on easing the electron transfer process through the microporous discharge product matrix.

Table 6
Effect of Lignosulfonate Concentration in the Developing Acid on the Electrode Capacity During Development and Subsequent Capacity Maintenance During Cycling in 1.250 sp. gr. H₂SO₄.

<table>
<thead>
<tr>
<th>Electrode Development</th>
<th>Test Cycles in 1.250 sp.gr. acid Electrode Capacity at 145th Cycle final</th>
<th>Cycle Life Index</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Electrode Capacity at the End of Development initial</td>
<td>Cfinal-Cinitial</td>
</tr>
<tr>
<td>Lignosulfonate Type</td>
<td>Concent. in 1.050 sp.gr. Acid</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>c₀</td>
<td>0</td>
<td>1.562</td>
</tr>
<tr>
<td>c₁</td>
<td>4.738</td>
<td>2.865</td>
</tr>
<tr>
<td>c₂</td>
<td>6.113</td>
<td>3.006</td>
</tr>
<tr>
<td>c₃</td>
<td>4.923</td>
<td>2.839</td>
</tr>
<tr>
<td>c₄</td>
<td>3.357</td>
<td></td>
</tr>
<tr>
<td>c₅</td>
<td>2.433</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td></td>
<td></td>
</tr>
<tr>
<td>c₀</td>
<td>c₀</td>
<td>3.823</td>
</tr>
<tr>
<td>c₁</td>
<td>c₁</td>
<td>3.522</td>
</tr>
<tr>
<td>c₂</td>
<td>c₂</td>
<td>2.250</td>
</tr>
<tr>
<td>c₃</td>
<td>c₃</td>
<td>1.690</td>
</tr>
<tr>
<td>c₄</td>
<td>c₄</td>
<td>1.288</td>
</tr>
</tbody>
</table>
Lignosulfonate concentration in developing acid, ppm

Figure 5 The effect of lignosulfonate concentration in developing 1.050 specific gravity H₂SO₄ on developed capacity and steady state capacity in 1.250 specific gravity H₂SO₄.
Figure 6 Lead sulfate discharge product morphology (a) without Type B lignosulfonate (b) with $c_1$ concentration of Type B lignosulfonate.
Table 7
The Discharge Activity of Type B Lignosulfonates as Function of its Concentration in 1.240 sp.gr. \(\text{H}_2\text{SO}_4\) and Discharge Current

<table>
<thead>
<tr>
<th>Lignosulfonate</th>
<th>Concentration, ppm</th>
<th>Discharge Time to (-.500\text{V (Vs. Hg-Hg}_2\text{SO}_4)) in Minutes at Discharge Current of</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1000 (\mu\text{A})</td>
</tr>
<tr>
<td>Type B</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td></td>
<td>1.63</td>
</tr>
<tr>
<td>(c_0)</td>
<td></td>
<td>2.09</td>
</tr>
<tr>
<td>(c_1)</td>
<td></td>
<td>2.52</td>
</tr>
<tr>
<td>(c_2)</td>
<td></td>
<td>2.67</td>
</tr>
<tr>
<td>(c_3)</td>
<td></td>
<td>2.94</td>
</tr>
</tbody>
</table>

The adsorption behavior of Type B and Type A lignosulfonates in water solutions on reagent grade lead sulfate powder are shown in Figure 7 and 8, respectively. The lignosulfonate concentration is noted by UV absorption at 280 nm. Figure 9 shows the adsorptive behavior of the lignosulfonate expander on lead sulfate as a function of acid concentration. It indicates that the lignosulfonate adsorptivity on lead sulfate powder decreases with increasing acid concentration. It also indicates that the adsorptive influence of lead sulfate will be more pronounced in low acid concentrations than in high acid concentrations. This agrees closely with our previous observations in which we have noted increased expander activity with decreasing acid concentration.

Task 2.2 Equipment Development

All fifteen life cyclers have been received (beginning of FY'79) and are operational. The equipment has functioned well for seventy cycles over a period of one month. Data collection software has continued to function well with new features being
regularly appended. The data is routinely transmitted to the Honeywell computer for graphical display and long term storage on tape.

At this time, the cycling hardware has logged a combined total of >400 cycles on EV-type cells for the twelve units which have been activated. No major problems have arisen to date. Data collection software continues to function well under conditions of continuous operation.

Software, which allows for a graphical representation of cell parameters, continues to be developed. Discharge plots are the product of over 700 data points, while the charging plot is composed of over 1200 data points. No curve fitting or 'data smoothing' is employed. The curves represent only the inter-connection of accumulated data.

Data for these graphs was obtained via the minicomputer controlled data logger. Once the test has been completed, data may be listed in tabular form prior to its transmission to the Honeywell 66/40 mainframe computer. Once contained within the Honeywell computer, the data are catalogued as a disk file which may be accessed by plotting software to give a graphical representation of acquired data. Periodically, the catalogued disk files are transferred to magnetic tape for long term storage and future reference.

Later, revisions to the cell cycling data collection software were implemented. This resulted in added capacity for retention of end of discharge and end of charge cell parameters when performing continuous cycling, or, retention of capacity tests and a subsequent charge summary data are maintained in separate disk files for each cell. With this added feature, a cell's cycling characteristics may easily be retained for every cycle during its test life. This ordering of information will greatly reduce the effort required to make observations on charge/discharge behavior as a function of cycle number, number of cycles between equalization, depth of discharge, etc.

As with all the data, these summary files will be transferred to the Honeywell computer for extended storage on magnetic tape. To make the system more versatile, graphic display programs were developed which allows instant, graphical printouts of any tests, or combination of testing plots, contained in storage.
Figure 7 Adsorption behavior of Type B lignosulfonate on lead sulfate in water at 22°C.
Figure 8 Adsorption behavior of Type A lignosulfonate on lead sulfate in water at 22°C.
Figure 9 Effect of acid concentration on the adsorption behavior of Type A lignosulfonate on 50 grams lead sulfate powder.
Task 2.3 Deep Cycling Tests

The influence of the Type B lignosulfonate in pasted plates was carried out by negative limited test cells constructed with six negatives and seven positives. In these test cells, the ratio of negative to positive paste was maintained close to 0.26 (Table 8) and theoretical capacity ratio between positive to negative around 3.65. The test negatives had 0, 0.02, 0.10, and 0.50 weight percent Type B lignosulfonate based on the weight of oxide. Three test cells of each group were electroformed in 1.100 specific gravity H₂SO₄. The mode of cycling, arbitrarily selected for this test, was 132 minutes discharge at 40 amperes followed by 228 minutes charge.

Eₙₑḡ, Eₚₒˢ, ᵅₑᶜₑˡˡ, ᵃᶜₑˡˡ (charge) and E₂80 (concentration of lignosulfonate as noted from UV-absorption at 280 nm wave length) were monitored during formation. The results are summarized in Table 9. Very little changes have been noted in cell parameters (Eₙₑḡ, Eₚₒˢ, ᵅₑᶜₑˡˡ) for lignosulfonate concentrations in the paste up to 0.10 weight percent. The cells with 0.5 weight percent lignosulfonate in the paste were noted to have a noticeable increase in negative potential and a decrease in formation current (Table 9). The electrolyte analysis for lignosulfonate have shown very little changes between the cells with 0.1 and 0.5 weight percent Type B lignosulfonate in the negative paste. The initial capacity (Figure 9) and material utilization of the negative active masses (Table 10) have shown an increase with increasing lignosulfonate concentration in the negative matrix.

The fifth capacity test after formation had shown average cell capacities to be 72, 90, 99 and 108 amp hours for lignosulfonate concentrations of 0, .02, .10, and .50 weight percent in the paste respectively. Due to the large deviation in capacity, in spite of identical net active mass, the cyclic mode of these four test series were varied to represent 90% depth of discharge per cycle as stated in Table 11. The summary results of the net and gross capacity characteristics of the test cells, as obtained at the end of charge cycle and after full charge, are given in Figure 10 and 11. The negative electrode characteristics during cycling and at early capacity tests are shown in Figures 12 and 13 respectively.

These results suggest the presence of an optimum concentration of lignosulfonate in the pasted electrode for maximum capacity and cyclic performance as previously observed from the study of the lignosulfonates influence on the microelectrode
In addition to the role of the lignosulfonate in modifying the plate morphology during electroformation, these results have also indicated that the presence of lignosulfonate in the matrix (residual) helps to build up capacity during early cycling (Figure 11). This is evidenced by the half cell potential data at the end of discharge during cycling (Figure 12) and the potential characteristics of the lead electrode during capacity tests at the start and around the 70th cycle point (Figure 13). This again parallels our observations with the microelectrode study.

The gradual loss of the capacity of the lead electrodes containing lignosulfonate, after attainment of maximum capacity (Figure 11) could be associated with the physical loss of lignosulfonate from the plate matrix by the dissolution process followed by oxidation of the dissolved species at the positive electrode or, by the chemical degradation of the material lead to the loss in the lignosulfonate activity. It is possible that it is a combination of the two.

At the end of the year, these EV-type cells (0-0.5 weight percent Type B expander) were placed on accelerated life cycling. The cycling regime was programmed to be four cycles per day, at a constant discharge current of 40 amperes. Discharge time is adjusted so as to remove 90% of the apparent cell capacity, as determined by the average of the five 100% DOD discharges performed immediately after formation.
Table 8
Particulars of the Negative Limited EV Cells Used for Lignosulfonate Activity Evaluation in the Pasted Type Negative Electrodes

<table>
<thead>
<tr>
<th>w/o of Type B in negative, based on the wt. of oxide</th>
<th>n</th>
<th>0.02</th>
<th>0.10 (Control)</th>
<th>0.50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Negative Paste per Cell, gm.</td>
<td>768</td>
<td>764</td>
<td>760 (1600)</td>
<td>765</td>
</tr>
<tr>
<td>Positive Paste per Cell, gm.</td>
<td>2902</td>
<td>2876</td>
<td>2918 (2320)</td>
<td>2903</td>
</tr>
<tr>
<td>Theoretical Negative Capacity AH 1</td>
<td>185</td>
<td>184</td>
<td>183 (385)</td>
<td>184</td>
</tr>
<tr>
<td>Theoretical Ratio 1 of Positive and Negative Capacity</td>
<td>3.66</td>
<td>3.64</td>
<td>3.72 (1.41)</td>
<td>3.68</td>
</tr>
</tbody>
</table>

1 Capacity calculations are based on 0.93 gm of lead per gram of negative paste and 1.04 gm lead dioxide per gram of positive paste.

2 Figures in parenthesis represent baseline EV-cells.
Table 9

The Influence of Lignosulfonate Concentration in Pasted Type Negatives on the Formation Behavior of the Negative Limited EV-Cells

<table>
<thead>
<tr>
<th>Particulars When Formed in 1.100 sp. gr. Sulfuric Acid Solution</th>
<th>Type B Concentration in Pasted Negative (w/o based on the weight of oxide)</th>
<th>0</th>
<th>0.02</th>
<th>0.10</th>
<th>0.50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duration, min.</td>
<td>500 2500 500 2500 500 2500 500 2500 500 2500</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E cell, volts</td>
<td>2.467 2.489 2.484 2.500 2.469 2.500 2.500 2.500</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E neg (vs. Hg/Hg₂SO₄), volts</td>
<td>1.376 1.269 1.383 1.278 1.369 1.269 1.444 1.394</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E pos (vs. Hg/Hg₂SO₄), volts</td>
<td>1.091 1.220 1.101 1.222 1.105 1.231 1.056 1.106</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Icell, amps</td>
<td>30.0 3.0 30.0 3.1 30.0 3.2 11.0 4.7</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>QAH, formation charge, AH</td>
<td>259.3 658.0 252.0 612.0 250.0 654.0 203.0 452.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(280, UV-absorption of the formation electrolyte*</td>
<td>- - 0.209 0.247 0.243</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*relative concentration of Type B lignosulfonate in formation electrolyte.
Table 10

Effect of Lignosulfonate Concentration on Discharge Capacity and Material Utilization of the Lead Electrode After Formation

<table>
<thead>
<tr>
<th>Particulars</th>
<th>Type B Concentration in Pasted Negatives (w/o based on the Weight of Oxide)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>Capacity at 40A/cell (52A/kg or 24A/lb. of negative paste), Ah.</td>
<td>85.3</td>
</tr>
<tr>
<td>2</td>
<td>81.0</td>
</tr>
<tr>
<td>3</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>-</td>
</tr>
<tr>
<td>AVERAGE</td>
<td>83.2</td>
</tr>
<tr>
<td>Material Utilization of lead-active mass, %</td>
<td>45.0</td>
</tr>
</tbody>
</table>
Table 11

Initial Capacity and Cycle Mode of the Negative Limited Test Cells

<table>
<thead>
<tr>
<th>Test Series with Type B Lignosulfonate Concentration w/o</th>
<th>Fifth Capacity after Formation, Ah.</th>
<th>Six Hours Cycle</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Charge</td>
</tr>
<tr>
<td></td>
<td></td>
<td>time * min.</td>
</tr>
<tr>
<td>0</td>
<td>71.7</td>
<td>265 33 2.50</td>
</tr>
<tr>
<td>0.02</td>
<td>90.2</td>
<td>241 33 2.50</td>
</tr>
<tr>
<td>0.10</td>
<td>98.5</td>
<td>228 33 2.50</td>
</tr>
<tr>
<td>0.50</td>
<td>108.2</td>
<td>228 33 2.60</td>
</tr>
</tbody>
</table>

*Time varied to accommodate capacities
Figure 10  Capacity of the negative limited test cells as a function of cycle number. Capacity determined as described in Table 11.
Figure 11 Capacity of the negative limited test cells as a function of cycle number. Capacity determined after 16 hours charge.
Figure 12  The end of discharge negative electrode voltage as a function of cycle number for Type B lignosulfonate in the paste
Figure 13  Half cell voltage characteristics during discharge
WBS 3.1 ISOA Processing and Material Screening

Task 3.1.1.1 Grids

Experiments were conducted to select the optimum alloy composition for both the positive and negative grids from nine positive alloy candidates (PI-P9) and four negative alloy candidates (N1-N4). Samples were cast for age hardening strength and corrosion tests. These samples were cut to exact dimensions and chemically cleaned. The results of the age hardening tests for alloys PI-P9 are depicted in Figures 14, 15 and 16.

The test results indicated, processability-wise, that all the positive alloy candidates exceeded Globe-Union's minimum required strength levels. However, that is only one criteria of many.

In a similar manner, age hardening tests were run for the negative alloy candidates (Figure 17). Alloy number N3 did not meet minimum strength specifications.

The corrosion samples were tested in a refluxing apparatus. The equipment was designed to allow the testing of four samples simultaneously under identical conditions. The H₂SO₄ acid was held at a constant temperature (+0.5°C) by the use of a water bath. Seven positive half cell voltages (as referenced to a Hg/Hg₂SO₄ electrode) were chosen and maintained using custom-built potentiostats. In all cases, the potential variations were held to ±1mV. One set of samples was corroded for 72 hours at each, fixed potential.

The extent of corrosion was measured in terms of weight loss of lead. The lead content, in parts per million, was determined by atomic absorption. The fourth sample from each set was mounted for microscopic examination of the corrosion layer.

Results of the corrosion tests are shown in Figures 18 through 20. The majority of the alloys exhibited expected corrosion characteristics, however, alloys P1, P2 and P3 showed lower rates of corrosion and alloys P3 and P6 had non-characteristic curves.

After candidate alloys were selected, several grid casting runs were made and the acceptable grids were placed in inventory. For the large ISOA grid, the debugging and casting runs proceeded remarkably well. Small modifications were required on grid molds and the new 'trimmer die'.

Figure 14 Yield strength versus age time
Figure 15  Yield strength versus age time
Figure 16 Yield strength versus age time
Figure 17 Yield strength versus age time
NOTE: Alloy numbers P1, P2 and P3

Figure 18 Corrosion as a function of the positive half cell voltage
Figure 19  Corrosion as a function of the positive half cell voltage
Positive half cell voltage (referenced to Hg/Hg\textsubscript{2}SO\textsubscript{4})

Figure 20  Corrosion as a function of the positive half cell voltage

NOTE: Alloy numbers P7, P8 and P9
Task 3.1.1.2 Oxides

Screening tests on various oxide processing methods began in December. Three different oxide mixing methods were examined and the subsequent paste parameters analyzed. Test cells were constructed and placed on a life cycling test regime (Figure 21):
- 2 cycles/day
- 80% DOD @ the C3/3 rate

Considerable differences were noted in the active materials produced. To date, insufficient data is available to project an optimum method.

Task 3.1.1.3 Plate Curing

Studies were conducted in the way the pasted grids were cured. This is a chemical process where the high percent levels of free lead are reduced to <4.0%. It is a complex process where the final, dried active materials are affected by time, moisture in the paste, chamber humidity and temperature.

It was known that an infinite number of combinations could be pursued, but would also be impractical. The approach chosen was to use in-house data, of batteries/curing regimes that nearly mirrored the ultimate EV performance specification, and focus the experiments to three prime candidate processes. It should be noted that any optimized process may be sensitive to future battery design improvements. This is also true for any of the other optimization programs.

Cells were constructed and put on life cycling testing which were constructed with plates that were cured per the experimental curing methods (Figure 22). Analysis of the test data suggested that significant performance differences can be obtained. It was felt that the investigations of higher curing chamber temperatures was advantageous and that considerable more cycling would be required before definitive decisions could be made.

Task 3.1.1.4 Formation

All the inherent Process selection problems concerning the 'Curing' experiments are applicable to the 'Formation' optimization task. In fact, they are magnified. The processes of formation, the electrochemical forming of the cured plates to lead sponge negatives and lead dioxide positives, are more complex and
Figure 21  Capacity/cycle life as a function of positive material processing method
Figure 22  Capacity/cycle life as a function of curing procedure
candidates more numerous. Some of the parameters investigated were:

- Time
- Total ampere hour input
- The rate (i.e. C3/3) at which the input is limited
- Temperature profiles
- Specific gravity of the electrolyte
- Upper current limits
- Upper voltage limits
- Open circuit regimes

Candidate selections were more difficult than the curing process selections. Again, the same technique used for the curing selection was employed. Even with considerable screening, sixteen different formation processes were deemed sufficiently promising to explore.

The impact of this work load forced this optimization program to come on board later than the rest. The result of this is that the efforts to date have produced minimal data.

The sixteen formation experiments were considered a starting point. It was acknowledged that updated analysis would either reduce or increase the total number of experiments. This would be particularly germane when results from chemical and morphology analyses were available.

Task 3.1.1.5 Separators, 3.1.2.3 Separator Assembly

The original scope of work for this subtask was to study methods to transform separator 'blanks' into an envelope-type configuration by designing appropriate machinery to fold and seal this component. In addition, plans called for the establishment of various processing parameters to accomplish this function. Preliminary testing indicated that this subtask would have to be expanded considerably to include studies not originally foreseen.

As test data accumulated and inputs were received from the other engineering groups, it became apparent that the standard separator configuration was a major cell failure mode component. This was a critical problem that had to be solved, less this defect bias the results and conclusions of the cells then on test.

Schedules and coordinated manpower priorities were realigned
to attack this problem(s). Post mortem of many cells confirmed that three failure modes were present:

1. 'Mossing' shorts
2. Dendritic shorting
3. Unreliable sealing process

Within several weeks, the failure modes were defined and corrected. Cells that were under test were retrofitted with separators that were of the new design optimized sealing method.

When confidence was restored in the functional reliability of the separator component, efforts were redirected to the original scope of work. Although a breakthrough had been made, it did cause a delay in the progress of this subtask, as originally defined, for the following reasons:

1. Redirection of manpower
2. The solution process opened other areas to be addressed for investigation
3. Forced reordering of blank separator stock (very long lead times for the procurement)

A subtask of the separator program involved new separator designs. A much lighter separator was assembled in cells and placed on life cycling. These tests were initiated to determine whether or not if this separator could survive the brutal EV battery test regime. If successful, this would translate into:

1. A weight reduction for the battery system or additional volume for electrolyte
2. Lower internal resistance
3. Increased sustained power

These tests are still in progress and while the results appear favorable, firm conclusions are not available (Figure 23).

Task 3.1.1.7 Cell Testing

Prior to contract award, Globe-Union suspected that, in order to realistically meet the ambitious goals of the program, an electrolyte circulation system would be required. Work was programmed in this area in the subtask number 4.2.1.1.4 and WBS 5.3[1]. These efforts were scheduled for the 'Advanced' EV

Figure 23 Performance comparison of separator back web thickness
battery system and were considered a high risk venture. It was hoped that such a subsystem would be achievable.

Several events prompted Globe-Union to 'rescope' its internal program that accelerated this task and committed our efforts to design into the ISOA EV battery a workable circulation system. First, continuing in-house testing of batteries (with testing regimes close to the expected EV test specifications) showed that the electrolyte stratified and this resulted in limiting cycle life. Secondly, logic dictated that when the ISOA battery design was frozen, without the electrolyte circulation subsystem, and not knowing the eventual configuration, that it might be impossible to execute a retrofit. Additionally, it was very possible that the numerous optimization programs would have to be repeated as the circulation system would constitute a major design change.

For the purpose of this report, tasks 3.1.1.7 and 4.2.1.1.4 should be considered as one, as the design of a circulation system impacts all aspects of cell testing. Whereas this section deals with the concepts and initial cell testing of the circulation systems, other subtasks (4.1.2 and 5.3) deal with the problems of the design of the required hardware and the incorporation of the cell concepts into a viable battery circulation system.

When preliminary testing specifications became available, EV-type cells were placed on accelerated life cycling (Figure 24). Cell 'A' represented a modified EV State-of-the-Art (SOA) cell construction, however, cell 'C' was equipped with an auxiliary device to circulate the electrolyte. These tests confirmed earlier observations and opinions. Stratification of the electrolyte occurred early in cycling and was found to be one of the numerous parameters that limits the obtainment of the cycle life goals of this contract.

While these tests were under way, efforts were being directed towards translating the concept into a workable design (this was successfully accomplished in FY'79). When the initial tests on stratification started to show favorable results (the negation of stratification), three major decisions were made:

1. Cells presently on test would be converted to have an electrolyte circulation device.
2. All future cell/module tests would have electrolyte circulation devices.
3. It would be assumed that a viable electrolyte system would be a reality and thus, as design improvements were available, the cells/module would be retrofitted with the latest hardware improvements.

It should be noted that Globe-Union's electrolyte circulation system is an 'on-board' subsystem and its weight has been included in the total battery system weight for all published electrical performance results.

When the first group of ISOA grids became available, the results of the various optimization studies (best results/directions then available) were analyzed to provide the baseline for the first ANL-ISOA cell design. Eight cells were constructed, five of which were delivered to the National Battery Test Laboratory for testing.

The cells that remained in-house were subjected to various testing regimes. One series of tests was the measuring of the cell/battery power capability (Figure 25). The test procedure was determined by a joint effort between ANL and Globe-Union. The resulting test regime for the sustained power test was deemed a severe test as cell voltage was allowed to drop 30% (rather than the standard 50%) and the time under load was increased to 20 seconds.

The 'first-cut' test was extremely encouraging when considering the following:

The U.S. Department of Energy's 'Second Annual Report to Congress for Fiscal 1978, Electric and Hybrid Vehicle Program', DOE/CS-0068, page 54, Table 4-6, documents projected performance on sustained power of the lead-acid system to be:

<table>
<thead>
<tr>
<th>Table 12</th>
<th>Peak Power at 20 Seconds, 50% S.O.C. (voltage drop undefined)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Watts/Kg of Battery</td>
<td>90 90 95 118</td>
</tr>
</tbody>
</table>

1 The subsystem weights are calculated weights.
Figure 24 Comparison of cycle life of cells with and without electrolyte circulation.
Figure 25 Watts per kilogram of battery as a function of state of charge (%)

NOTE:
Voltage drop to 1.4V/cell
The companion curve was equally encouraging (Figure 26). Results showed that at the 20% S.O.C. (empty fuel tank), the cell/battery could deliver 680 amperes for 20 seconds. Not knowing the downstream EV power requirements, it cannot be stated factually, but it appears that minimal acceleration capability would be lost when the fuel gage registers close to 'zero'.

The cells were placed on life cycling test regimes (80% DOD at the C3/3 rate). Two of the cells were tested on an accelerated regime of 2.55 cycles per day (Figure 27). The remaining cell was put on a more 'real-life' test regime of one cycle per day (Figure 28). Both curves show the end of discharge cell voltage as a function of total cycles (1.75 volts/cell being considered end of service life).

As of August 24, 1979, cell number 2VD3A-7 (one cycle/day) had reached the 135th cycle and was still on life cycling testing with an end-of-discharge voltage of 1.8905 volts.

Cell number 2VD3A-6 exhibited a service life of 247 cycles on the accelerated testing regime. It is not known how many cycles this represents on a single cycle/day test regime. It has been our experience that with increasing cycles/day results in decreasing the total life cycles, however, this 'K' factor is unknown.

The cell performance of the first ANL-ISOA design have been very encouraging and this design did not benefit from the final results of any of the optimization programs.
Figure 26: Sustaining current profile as a function of state of charge (%)
Figure 27 End of discharge voltage versus number of cycles

NOTE: Cell number 2VD3A-6
80% DOD
2.55 cycles/day
Figure 28 End of discharge voltage versus number of cycles

NOTE: Cell number 2VD3A-7
80% DOD
1 cycle/day
WBS 4.1 ISOA Battery Design and Construction

Task 4.1.2 Design Updates

During FY’79, the majority of the battery components were subjected to the 'fine-tuning' of the ISOA design. (The notable exception being the substantial container and cover modifications required to interface with the evolving electrolyte circulation subsystem.) Of the former group, the following accomplishments were obtained:

1. Changes to the drawing package required to convert the twenty-four volt module to the twelve volt module were completed.

2. A new ISOA terminal bushing was designed and incorporated into the cover design. The new bushing will increase resistance to terminal acid leakage and increase the terminal twist resistance.

3. Polarity designations were added to the terminal posts (positive and negative) to insure proper element insertion into the container.

4. The watering/vent manifold was designed.

The majority of the time expended in this subtask was devoted to the design(s) of the electrolyte circulation subsystem hardware and the subsequent impact to the container and cover designs. The pump hardware evolved through six major designs. As each design matured, 'fit and function' studies were carried out. Additionally, each design was studied to evaluate methods available to convert hand-made prototypes into a viable, cost effective, mass produced components.

The circulation component design impact on the container and cover designs was considerable. Major design changes were made to both pieces to accommodate the new subsystem.

Task 4.1.3 Performance Testing

Testing was done on the prototype flame arrester and the weld buttons to assist component designs. The flame arrester was tested using a modified BCI test procedure. A 108 volt battery pack was used and the gases vented through one flame arrester (two flame arrestors will be used in the Argonne battery system).
Figure 29 Model of the 12 volt ISOA module
The test procedure used was:

1. Charge to a 100% State-of-Charge condition
2. Immediately overcharge continuously at 20 amperes until the flow rate of the gases plateau.
3. Using a high voltage supply, initiate sparks near the flame arrestor.

The gases ignited, however, the flame front was unable to propagate to the batteries.

The weld buttons were subjected to electrical and thermal tests (figures 30 and 31). The results of these tests, along with the sustained power tests, were used to make final design decisions.

Task 4.1.4 Deliverables

ISOA containers and covers were not available in December, 1979, therefore, special cells (interim design) were fabricated for subsequent cell deliveries to NBTL. These cells were modified to provide features which would aid in the testing program.

The first five cells were delivered to NBTL in April 1979. An electrolyte circulation subsystem was provided. This subsystem was designed to accommodate the operation of one cell or the simultaneous operation of two to five cells. The results of the testing at NBTL have been extremely encouraging.

WBS 4.2 Advanced Battery Design and Construction

The 'Advanced' design logically must be based on the knowledge, performance testing, and resultant data analysis of the 'ISOA' design. This is a logical sequence, but not necessarily practical. If 'go - no go' decisions are held in abeyance for complete testing (one being life cycling), an 'Advanced' design would never leave the drawing boards. Hence, the dilemma, not only in execution but also in the forecast narrative.

To achieve the goals of the Advanced Electric Vehicle Battery (to present specifications and without benefit of interfacing with the Electric Vehicle Manufacture) requires:
Figure 30 Resistance versus weld button configuration
Figure 31 Temperature rise versus current of three weld button configurations
• Increase cycle life
• Increase specific energy
• Increase sustained power

The methods by which the above will be achieved will be accomplished by the combination (some now available; others in the planning stages) of the following:

• Lead-plastic composite electrodes
• Increased specific gravity of electrolyte
• Synthetic organic lignosulfonate, optimized
• Reduction of weight
• Sophisticated charger/battery marriage
• Optimization of the electrolyte circulation subsystem
• Inputs from all optimization programs

Task 4.2.1 Operating Supplies

The baseline design work on the plastic-lead composite grid was completed during FY'79. The complexity of this task is such that the baseline is only the first step of the long and ambitious project. The goal of the plastic-lead composite grid(s) is to reduce total module weight by the replacement of structural lead with plastic.

Globe-Union's proven computerized battery modeling program was used extensively to assist in the design of the plastic-lead grids. Many designs were examined as the optimization program continued. Not only were the mechanical and electrical parameters optimized and studied, but also design considerations had to be given to the mass production manufacturability of each grid design.

The computer program, given the proper inputs, is also capable of predicting electrical discharge curves, on a constant current test regime. The 'first-cut' projections showed an increase in Whr/kg of ≥ 5.4%, assuming a 7% total module weight decrease (Figure 32).

WBS 5.0 Battery Subsystems

Task 5.1 Fuel Gage

It was decided to delay investigations of the 'Fuel Gage' task until such time that a reasonable baseline battery design
Figure 32  Module weight reduction versus single panel grid weight
was established. It was concluded that the battery design and fuel gage subsystem were so interrelated that a small change in the battery design could force a major redesign of the fuel gage.

Task 5.2 Charger Development

Preliminary charger designs were completed (first phase experimental charger) and three units were constructed. These units were designed to provide maximum operational flexibility for studying various charging concepts. Directions were not given on charger input voltage (110 or 230 volts). Therefore, the prototype chargers are operational at either voltage level.

Design features of the chargers are:

- Switch selection of one of 4 modes (output wave form)
- Accomodates 1 to 6 cells
- Short circuit protection
- Reverse polarity protection
- Current and voltage limits

The chargers were bench-cycled and tested to obtain preliminary engineering data. Later, voltage/temperature compensation circuits were added. After initial engineering tests were analyzed and a literature search completed, the conclusions made were:

- Provide one charger, 'on-board'.
- Use a 230 volt charger supply.

Globe-Union believes that a sophisticated, rugged, and reliable charger is a prerequisite for a successful EV battery system.

Task 5.3 Acid Circulation

The main challenge of this task was to move the circulation concept from a high risk engineering idea, single cell category to a workable 48 cell subsystem. The initial single cell tests proved that the acid circulation design was required. Building on those preliminary tests, confirmatory tests on the negation of stratification, and the subsequent trade-offs, were run.

A semi-porous block of material (to simulate the cell element) was positioned inside an empty cell container. \( \text{H}_2\text{SO}_4 \)
Figure 33 Experimental chargers
electrolyte was carefully entered into the cell container in three steps such that:

1. Bottom 1.400 specific gravity
2. Center 1.265 specific gravity
3. Top 1.100 specific gravity

In this 'worst case condition', the pump mixed the stratified electrolyte to specific gravity homogeneity within forty-five minutes.

Throughout FY'79 numerous system concepts were studied to design a reliable, simple subsystem that would overcome the obvious problems of weight, parasitic power losses, reliability and costs. After many months and numerous tests, a novel subsystem was conceived that met the rigid requirements. Calculations projected an energy density of 43.8 Whr/kg, in which the weight and parasitic power losses were included.

As each test was run, more cells were added to the subsystem test. To date, eighteen cells have been successfully operated simultaneously. The eventual scale-up to forty-eight cells is not considered to be a problem.
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SECTION 2.0 SUMMARY
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2.0 SUMMARY

Globe-Union is quite pleased with the progress that has been made during FY'79. It was during this period that the results from many of the subtasks were married and resulted in the design and submission of the first ANL ISOA cells to NBTL. It is the achievements made during FY'79 as measured against the contractural goals that is the 'bottom line' of our program. Table 13 gives an overview of the major areas for comparison.

For the ANL ISOA cells being tested in-house:

- **Cell number 2VD3A-6 (accelerated testing)**
  
  Obtained 247 cycles to the 80% capacity level. For the sake of obtaining additional data, this cell was placed on cycling again (@70% D.O.D.) and bottomed-out at 261 cycles.

- **Cell number 2VD3A-3 (accelerated testing)**
  
  Obtained 260 cycles to the 80% capacity level. Continued on the 70% D.O.D. testing regime and achieved 289 cycles.

- **Cell number 2VD3A-7 (one cycle/day)**
  
  As of August 24, 1979 the cell was on the 135th cycle and exhibited an 'End-of-Discharge' voltage of 1.8905 volts. It continues to cycle.

Within several weeks of testing it was determined that the design was obsolete. This is reported as a positive event as it represents how rapidly the lead-acid technology is progressing. Failure analysis has not been completed but it appears that shedding of the active material of the positive electrode will be the failure. Simply, the shedding of the active material results in the loss of electronic and mechanical continuity of the active material, and hence, the ultimate loss in capacity.

Of major importance to the program, three design successes were realized:

1. The electrolyte circulation concept was proven essential and provided performance improvements far in excess than originally thought.
<table>
<thead>
<tr>
<th></th>
<th>Battery Capacity (kW-hr)</th>
<th>20-30</th>
<th>23.4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(100% rated)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>Battery Dimension</td>
<td>29.5x40.6x264</td>
<td>29.5x38.1x264.2</td>
</tr>
<tr>
<td></td>
<td>(cmH x cmW x cmL)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>Specific Energy (W-hr/kg)</td>
<td>40</td>
<td>42.5</td>
</tr>
<tr>
<td>4.</td>
<td>Specific Power (W/kg)</td>
<td>100</td>
<td>148(1)</td>
</tr>
<tr>
<td></td>
<td>Peak Battery - 15 sec x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>Lifetime</td>
<td>800</td>
<td>&gt;260(2)</td>
</tr>
<tr>
<td></td>
<td>Deep Discharges</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6.</td>
<td>Energy Efficiency</td>
<td>&gt;60</td>
<td>~83</td>
</tr>
<tr>
<td>7.</td>
<td>Typical Installation</td>
<td>96-120V</td>
<td>96V</td>
</tr>
<tr>
<td></td>
<td>Voltage</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(1) 100% SOC, 20 seconds sustained, to 1.40 volts/cell (30% drop)
(2) Accelerated cycling, total cycles lost due to test regime unknown
2. Of the many deep cycled test cells tested to date, these are the first two cells which did not fail due to separator failure. This speaks highly of the successful work done on the 'Separator' sub-task effort.

3. The mechanism responsible for the shedding process has been defined. This will enable Globe-Union to attack this area and provide cells/modules in the future to exhibit quantum improvements in cycle life.

Unfortunately, there is still no credible information available to help define the 'K' factor between the multi-cycle/day accelerated tests and the 'real life' one cycle/day test. To make matters more difficult, it is very probably that these three cells will not provide the data being sought.

The sister ANL ISOA cells being life tested at NBTL confirm our excitement with this first design. Although the test regimes are dissimilar, the resultant, exceptional 'first-cut' performances compliment one another.

Other 'characterization' tests at NBTL and Globe-Union look equally good. Sustained power test, charging, roundtrip efficiencies, and thermal management data look especially good.

It should be pointed out that the first ISOA cells do not have the benefit of all the sub-task conclusions. It is not known what total performance benefits will be realized, after the second cell/module submission, when more outputs are available and translated into battery design.

These sub-tasks continue, all at different stages of completion. Probably the most successful and most visible results have been made in the area of electrolyte circulation and separator processing/design. These studies continue.

As one would expect with a R, D and D type of program, priorities constantly shift. This is further complicated, by the realization that the battery performance is a moving target trying to hit the moving target of vehicle design.

The substantial efforts being expended in the research area of expanders has produced significant results and advanced the technology. However, this program is still not far enough for
its results/conclusions to be incorporated into battery design. Certainly its impact will be felt and the magnitude unknown for the near future. The milestones accomplished are:

- Two electrochemical techniques based on the microelectrode testing technique have been developed for quantitative evaluation of expander activity on the high rate discharge cycling. These techniques were noted to be efficient and economical ways to determine expander activity outside a test battery.

- Good qualitative correlation between the microelectrode and pasted electrode was found and a more exacting correlation is being sought.

- An automatic cycling facility capable of simultaneous testing of 15 units has been designed and installed. The cell cycling uses a constant current discharge and constant current or constant potential/current limiting charging. The software permits the storage of relevant data, such as test time, cell voltage, half cell potentials, electrolyte temperature, amp hours and watt hours as a function of time as well as their tape storage and retrieval for graphical presentation.

- Accelerated cycling of 98 amp hour, negative limited test cells at 90% D.O.D. per cycle established 200 cycles as a base line cycle life for the standard negative.

- Selective evaluation of conventional expanders and six (6) modified expanders were completed.

As the expander research work continues, it is expected that the following activities will take place in FY'80:

- Extend the cycling facility to accelerate the expander optimization testing program.

- Modify the data management software for efficient data retrieval and graphical representation of the test data.

- Select the best expander candidate or combination of candidates from the available lignosulfonates.
Optimize the best expander in the pasted electrode to maximize the electrode performance.

Develop means to minimize the amount of lead in the negative active matrix by modifying the paste composition, including the addition of inert paste extenders.

The ISOA grids evolved through many computer optimizations and designs. The state-of-the-art was pushed with the designs and its impacts on casting, pasting, processing and formation. The design also taxed other areas merely because of its size. Rushed studies were done to ascertain whether or not the design was compatible with all the needed requirements.

Initial casting results were more than acceptable. Problems did occur with minor mold defects, which was expected and which have been corrected. At no time did the defects delay casting schedules. Some are still being worked on and total solutions will be achieved.

Throughout the FY'79, it was reported that non-typical corrosion curves for alloys P6 and P3 were abnormal and several retests were done on P6. However, the latest corrosion tests and subsequent mechanical processing now suggest that the original tests were correct. This was totally unexpected and additional investigations will be made.

In the areas of oxides, plate curing, and formation, we are in the early stages of investigation. Some activities are considered high risk exploratory adventures; the others not. It is too early to make conclusions but the tests show considerable ranges of response. This could result in breakthroughs or a 'bust'. It will be reported as data is accumulated. It should be pointed out that this is one area, (material/processing) where the optimization studies are ultra sensitive to battery design. To make this task more difficult, the different combinations of the three processes produce different results. The pyramid effect may be complex.

The separator studies have been highly successful. Up to the time of the testing of the ANL ISOA first submission cells, every deep discharged, life cycled test cell failure has been traced to separator failure. Even non-EV cell testing, of similar test regimes, have failed due to separator failure.
The failure mode, in retrospect, has caused problems in the data analysis of test results. All cell test performances were biased with this failure mode. The one question that immediately comes to mind is, "What would the performance and failure mode have been had the short through the separator not occurred?" Unfortunately, at the beginning of the program, many cells had to go on test simultaneously. The weakness in the separator component was known and every effort was made to minimize it. It wasn't enough; the separator studies finally produced the answers and directions.

The separator component is a highly complex part. Now that the shorting problem has been solved, studies will continue in other areas:

- Geometrics of the ribs
- Backweb configurations
- Electrical properties
- Glass mat

The battery design has gone through many changes. The first, and most ambitious, change was a complete redesign of the ISOA battery. This was done to update to the newest technology that occurred during contracts. Later, it was decided that the module would change from a 24 volt to a 12 volt configuration and this required another redesign.

After the decision was made to commit to an electrolyte circulation system in the ISOA battery, the design was changed several times to:

- accommodate the marriage of the pump hardware to the container
- update container design to reflect latest pump advances

The development of the pump hardware was put on a priority basis and required numerous designs that could successfully interface with the container design. Considerable time was expended in determining the best process and most competent vendor to produce the pump hardware. Changes are still being made to the components related to the electrolyte circulation subsystem, as it is still being developed system-wise.

The design of the lead-plastic composite electrodes continues. The design has been changed numerous times as the result of many computer and optimization inputs. Again, this is in the area of pushing the state-of-the-art. The emerging
designs dictate new demands on the processing capabilities of the equipment to produce the parts. We expect failures and additional redesigns but ultimate success.

Work on the 'on-board' charger has been continual but at a lower priority. Until a firm baseline cell design was established, the charger program could not proceed, less we would have to repeat the work. To date, three prototype chargers have been constructed and delivered to the three areas that will do the investigations. It is probable that this work will begin during the construction of the second submission of the ANL ISOA cells. Charging is an important area. The total success of the EV battery is dependent on the charger/battery combination. Once these studies begin, the priority level will immediately be upgraded.
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