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BATTERY RESOURCE ASSESSMENT
SUBTASK II.5
BATTERY MANUFACTURING CAPABILITY
RECYCLING OF BATTERY MATERIALS

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Recycle of Battery Materials

Summary

Studies have been conducted on the recycling of advanced battery system components for six different battery systems. These include: Nickel/Zinc, Nickel/Iron, Zinc/Chlorine, Zinc/Bromine, Sodium/Sulfur, and Lithium-Aluminum/Iron Sulfide.

For each battery system, one or more processes has been developed which would permit recycling of the major or active materials. This recycling is necessary for at least the following reasons: 1) conserve critical or valuable raw materials, 2) reduce battery manufacturing costs by reuse of costly manufactured sub-assemblies, 3) permit disposal by removal of hazardous or toxic materials.

Each recycle process has been designed to produce a product material which can be used directly as a raw material by the battery manufacturer. Metal recoverabilities are in the range of 93-95% for all processes. In each case, capital and operating costs have been developed for a recycling plant which processes 100,000 electric vehicle batteries per year. These costs have been developed based on material and energy balances, equipment lists, factored installation costs, and manpower estimates. Table 1 shows the processes considered for each battery type. Table 2 shows the capital and operating costs for the processes considered. The nickel recovery processes for the Nickel/Zinc and the Nickel/Iron battery systems are the same and are only shown once.

In general, there are no technological barriers for recycling in the Nickel/Zinc, Nickel/Iron, Zinc/Chlorine and Zinc/Bromine battery systems. The recycling process are based on essentially conventional technology which has been demonstrated in industry or at least in pilot scale R & D units.

~~The lead times required to build battery recycling plants based on these~~
processes is comparable to that of any other new plant. It is expected that confirmation of the process parameters would require some laboratory development. The total elapsed time required from inception to plant operation is approximately 3-5 years.

The recycling process for the sodium/sulfur and lithium-aluminum/sulfide battery systems are not based on conventional technology. In particular, mechanical systems for dismantling these batteries must be developed. In the sodium/sulfur battery system recycling operation these machines will be handling a large number of cells and may be operating in a solvent-laden environment. The chemistry for the dissolution steps must also be confirmed in order to ensure proper plant design. The elapsed time from inception to plant operation will be on the order of 6 years.

Table 1 - Recycling Processes

A. Nickel/Zinc Battery System

1. Zinc Electrode - Zinc oxide product
 - a) Acid Leach - hydroxide precipitation
 - b) Ammonia Leach - carbonate precipitation
 - c) Pyrometallurgical Reduction-oxidation
2. Nickel Electrode - Nickel Sulfate solution product
 - a) Acid Leach - solvent extraction
 - b) Ammonia Leach - solvent extraction
3. Nickel Electrode - Nickel powder product
 - a) Acid Leach - hydrogen reduction
 - b) Ammonia Leach - hydrogen reduction
 - c) Pyrometallurgical Reduction

B. Nickel/Iron Battery System

1. Nickel Electrode - Nickel sulfate solution product
 - a) Acid Leach - solvent extraction
 - b) Ammonia Leach - solvent extraction
2. Nickel Electrode - Nickel powder product
 - a) Acid Leach - hydrogen reduction
 - b) Ammonia Leach - hydrogen reduction
 - c) Pyrometallurgical Reduction

C. Zinc/Chlorine Battery System

1. Dismantling - Zinc Chloride Electrolyte Recovery

D. Zinc/Bromine Battery System

1. Dismantling - Zinc Bromide Electrolyte Recovery

E. Sodium/Sulfur Battery System

1. Dismantling - Sodium Polysulfide Recovery

Table 2 - Recycle Process Capital and Operating Costs

<u>Process</u>	<u>Capital Cost \$1,000</u>	<u>Operating Cost \$1,000/Year</u>
1. Zinc-Acid Leach	4,830	7,300
2. Zinc - Ammonia Leach	5,470	4,590
3. Zinc - Pyrometallurgical	4,770	2,800
4. Nickel Solution - Acid Leach	4,670	5,590
5. Nickel Solution - Ammonia Leach	5,010	6,270
6. Nickel Powder - Acid Leach	11,310	9,190
7. Nickel Powder - Ammonia Leach	6,630	6,230
8. Nickel Powder - Pyrometallurgical	6,440	3,940
9. Zinc/Chlorine	1,620	2,330
10. Zinc/Bromine	2,240	2,620
11. Sodium/Sulfur	10,610	8,460
12. Lithium-Aluminum/Sulfide		

Discussion

Process Descriptions

Flowsheets have been developed for recycling of advanced battery system components for six battery systems. These battery systems are: Nickel/Zinc, Nickel/Iron, Zinc/Chlorine, Zinc/Bromine, Sodium/Sulfur, and Lithium-Aluminum/Iron Sulfide.

For each battery system, one or more recycle process has been defined which will recover one or more of the major components or active materials.

The detailed process flowsheets and process descriptions are given in Appendix A.

In the nickel/zinc and nickel/iron battery systems, processes for recovering the zinc and nickel are described. The iron in the nickel/iron battery will be discarded rather than recycled. Processes for nickel electrodes which involve only washing and direct reuse have not been included in detail.

Capital and Operating Costs

Capital and operating cost estimates for each of the flowsheets have been prepared. The capital costs are based on equipment lists for each process. The equipment was sized using major stream flow material balances for a recycling plant processing 100,000 batteries per year. Standard factors were used to relate the capital cost to the purchased equipment costs. The capital costs for each recycle process are given in Appendix B. The costs shown are calculated in mid-1980 dollars.

The operating cost estimates are based on raw material and utility requirements, labor dependent items and capital related items. No credits are shown for materials such as copper, case materials or other components which may be recovered for recycle in addition to the primary materials. The operating costs are also given in Appendix B.

Technological Barriers

The processes developed for the nickel and zinc recovery processes are based on conventional technology which is either currently practiced or has been demonstrated in advanced pilot plant programs.

The processes for the zinc/chlorine and zinc/bromine batteries are primarily dismantling operations with recovery of electrolyte solutions. The purification of the electrolyte solutions may require additional process steps if significant quantities of other battery components dissolve in the electrolyte. It has been assumed that no significant deterioration of the bromine stabilizer occurs in the zinc/bromine battery and that no chemical treatment is required.

The dismantling and recovery of sodium polysulfide in the sodium/sulfur battery system requires that dismantling equipment be developed and that the solvent system be further characterized. The dismantling equipment required handles a large number of components since the batteries contain a large number of individual cells. The solvent system, based on dimethylacetamide in the process described, requires further development in order to determine the solubility, extent of reaction and reaction rates of the various polysulfides which may be formed in the recovery process.

Lead Time Requirements

The design and construction of the battery recycling processes closely parallels that of many other metallurgical operations.

The nickel and zinc recycle processes, which are based on conventional technology, would require approximately 5 years from inception to plant operation. This period of time is required to define and confirm design parameters, executive detailed plant engineering, construction of the facilities and plant start up.

The recycle processes for the zinc/chlorine and zinc/bromine batteries

would require somewhat less time to implement since the processes are primarily dismantling operations. Plant engineering, construction and startup would require about 3 years.

The recycle process for the sodium/sulfur process would required about 6 years to implement because of the additional time required to develop the mechanical equipment to dismantle the large number of cells involved and to further develop the polysulfide dissolution reaction chemistry.

Appendix A - Detailed Process Flowsheets and Descriptions

I. Nickel Recycle Processes (Common to Nickel/Zinc and Nickel/Iron Batteries)

A. Description of Nickel Recovery Process A

The flowsheet Nickel Recovery Process A is shown in Figure 1. After separation and washing nickel electrodes are conveyed to a storage pile. This storage contains a sufficient quantity of electrodes to supply the processing plant for a period of one week.

The size reduction and separation plant is operated one shift/day. The first step is to reclaim electrodes from the storage pile. The electrodes are crushed and then further reduced in size by a hammer mill. The final product from the size reduction plant is smaller than 20 mesh. Metallic material is removed from the crushed material in a dry magnetic separator. The non-magnetic fraction is conveyed to the Ground Electrode Storage Bins. The size reduction and separation equipment are connected to a baghouse for dust control.

The ground electrodes are fed to the leach system by feeders from the storage bins. Dissolution of the metal values is performed in sulfuric acid in a countercurrent leach. The nickel laden liquor is separated from unreacted solids and stored in a surge tank before proceeding to purification. The remaining solids residue is washed on a filter and then conveyed to a storage pile from which it is reclaimed for sale to a secondary metal processor.

The pregnant liquor from leaching contains approximately 10 g/l Ni^{++} and has a pH of 2-3. The solution is pumped to an agitated vessel where copper is removed from solution by the addition of hydrogen sulfide gas. The copper sulfide formed is insoluble and, therefore, can be recovered by filtration.

The filtered solution, now cleaned of copper, is reacted with ammonia to raise the pH to 8-8.5.

FIGURE 2. PROCESS B: AMMONIACAL DISSOLUTION OF NICKEL ELECTRODES

The solvent extraction plant selectively removes nickel from the pregnant solution and results in a concentrated acidic nickel solution for use by the battery manufacturer.

B. Description of Nickel Recovery Process B

The flowsheet for Nickel Recovery Process B is shown in Figure 2. The electrode preparation areas (i.e. storage, size reduction, metallic separation) are identical to process A. The difference in process B is that an ammoniacal leach is utilized to dissolve the metals of interest from the electrodes.

The dissolution is accomplished in two stages of countercurrent leaching where 95% of the nickel is leached. The nickel bearing solution is pumped to a pregnant liquor surge tank.

Unreacted solids are dewatered and washed for ammonia on a filter. Washed residue is stockpiled before being reclaimed for sale to a secondary metals processor.

Pregnant liquor is cleaned of any particulates in a polishing filter before entering the solvent extraction plant where the nickel is recovered as a relatively pure solution for reuse. Nickel, along with any copper present, is extracted from the pregnant liquor in the first stages of organic/aqueous contacting. The raffinate from this section is sent to the zinc recovery process. Nickel is selectively stripped from the loaded organic to produce a concentrated nickel solution which is supplied to the battery manufacturer for reuse. Any copper in the organic is then stripped to form a concentrated copper solution which is sold to a secondary processor or sent to a copper recovery process.

C. Description of Nickel Recovery Process C

Process C consists of directly recycling nickel electrodes with no chemical treatment. In this process the batteries are open, drained, and disassembled as before. After separation and washing, the nickel electrodes

are visually and physically tested to insure their suitability for reuse. Those electrodes which pass the tests are shipped to the battery manufacturer. Electrodes which are not acceptable are stored for eventual sale to a secondary metal producer.

D. Description of Nickel Recovery Process D.

The flowsheet for Nickel Recovery Process D is shown in Figure 3. The electrode sorting and grinding steps are essentially the same as in Process A. In Process D, however, a nickel powder product is produced for recycle to the battery manufacturer. This process is designed to provide nickel for sintered electrodes.

The ground electrodes are leached in two countercurrent stages of agitated leaching. Sulfuric acid is used as the leachant. Any unreacted material is separated from solution, washed on a filter, and stored for eventual shipment to a secondary metal processor.

Pregnant liquor containing the metals of interest is treated further to produce nickel powder. Any copper in solution is removed by reaction with hydrogen sulfide to produce insoluble copper sulfide. The purified solution is autoclaved where nickel in solution is reduced with hydrogen to form nickel powder.

The slurry from the autoclave is flashed and the powder removed by filtration. The nickel powder is dried in a rotary dryer and packaged for recycle to the battery manufacturer.

E. Description of Nickel Recovery Process E.

The flowsheet for Nickel Recovery Process E is shown in Figure 4. Process E also produces a nickel powder product, but from an ammoniacal solution. The electrode separation, grinding, and dissolution steps are identical to those described in Process B. The pregnant solution from the leach clarifier is purified and treated to form nickel powder.

FIGURE 3. PROCESS OF NICKEL POWDER PRODUCTION FROM NICKEL ELECTRODES

FIGURE 4. PROCESS E: NICKEL POWDER FROM AMMONIACAL DISSOLUTION OF NICKEL ELECTRODES

The solution is preheated and then treated to remove copper using sodium hydrosulfide to precipitate the copper as a sulfide. The product sulfides are collected and shipped to a secondary metal processor. The copper-free liquor is treated to precipitate the nickel values by steam stripping.

The nickel forms a basic carbonate during steam stripping where the ammonia level in solution is decreased. The ammonia and carbon dioxide vapors resulting from the steam stripping are sent to ammonia recovery where they are reabsorbed in water to form fresh leach solution.

The slurry from the strippers is discharged to a flash tank followed by a thickener where the solids and liquid are separated.

The relatively pure basic nickel carbonate is redissolved in concentrated ammoniacal solution. The nickel-containing solution is preheated and pumped to an autoclave where nickel powder is produced by hydrogen reduction. The nickel powder is filtered and then dried to yield the final product.

F. Description of Nickel Recovery Process F.

The flowsheet for Process F is shown in Figure 5. Process F utilizes the nickel carbonyl process to make nickel powder. This operation differs from the other processes in that the important reactions are gas-solid rather than liquid-solid.

The nickel electrodes, after separation and washing, are conveyed to a storage pile as in all the other processes. The electrodes are reclaimed and conveyed to the size reduction equipment. Only crushing is required to produce feed for this process. The magnetic separator is used to remove any ferrous material, such as plated steel grids and tramp metal, from the nickeliferous material. The granulated electrodes are then stored in storage bins.

The granulated electrodes are fed to a series of two reducers. In the

reducers, nickel oxide or hydroxide is reacted with water gas (mixture of hydrogen and carbon monoxide) or other hydrogen-rich gas to form nickel.

The metallic nickel is reacted with carbon monoxide in vessels very similar to the reducers to form nickel carbonyl gas. Nickel carbonyl gas is taken from the top of the volatilizers and piped to decomposing units.

The decomposers form nickel from the carbonyl gas. The gas decomposes to nickel powder and carbon monoxide on the hot walls of the decomposer unit. The powder falls to the bottom of the decomposer and is collected for packaging and shipping to the battery manufacturer. The carbon monoxide gas is recycled to the volatilizers.

II. Zinc Recovery Process Options for Nickel/Zinc Batteries

A. Description of Zinc Recovery Process A.

The flowsheet for Zinc Recovery Process A is shown in Figure 6. After separation and washing, the zinc electrodes are transferred to a storage area. The zinc electrodes are reclaimed from storage and conveyed to the processing plant.

The first step of the processing consists of size reduction. The zinc electrodes are conveyed to a crusher and then reduced further in size to about 20 mesh. A magnetic separator after the mill removes any tramp magnetic material from the ground material.

The ground electrodes are conveyed to storage bins. These bins serve as the feed bins for the recovery process. The crushing, grinding and storage operations are connected to a baghouse for dust control.

The zinc electrodes are dissolved in sulfuric acid solution in a counter current leach. The zinc-rich solution is pumped to a surge tank prior to purification. The solids, which contain some metallics as well as other

FIGURE 6. ZINC RECOVERY PROCESS A

materials (plastics, etc), are filtered and washed. The filtered solids are drummed for disposal or sale to a secondary processor.

The zinc-rich solution is pumped from the surge tank to the copper removal tank. In this tank, the solution is mixed with zinc powder to cement out most of the copper which dissolved in the leach. The solution is filtered and then pumped to a surge tank which feeds the solvent extraction unit to remove dissolved nickel and any residual copper.

The pure zinc solution is mixed with filtered battery electrolyte (KOH) or other hydroxides to precipitate $\text{Zn}(\text{OH})_2$ in the precipitation reactor. The resultant zinc hydroxide cake is heated in the kiln to drive off the water and convert to $\text{Zn}(\text{OH})_2$ to Zn. The final zinc oxide powder is drummed for shipment to the battery manufacturer.

B. Description of Zinc Recovery Process B.

The flowsheet for this Zinc Recovery Process B is shown in Figure 7. The zinc electrode storage, crushing and grinding are identical to Process A. In this process, however, the ground electrode material is leached in an ammoniacal leach liquor to dissolve the metal values.

The leaching of the ground material takes place in a counter-current leach step utilizing an ammonium hydroxide/ammonium carbonate solution. The zinc-rich solution is pumped to the purification section. The residual solids are filtered, washed and then drummed for disposal.

In order to remove any dissolved nickel and copper, the zinc-rich leach liquor is purified using solvent extraction. The purified zinc-rich liquor is pumped to a steam stripper. In the stripper, the ammonia in solution is driven off, causing the zinc to precipitate as zinc carbonate. The precipitate is collected, filtered and washed.

The zinc carbonate filter cake, is transferred to a rotary drier/kiln where water is removed and the zinc carbonate calcined to give zinc oxide. The

calcined zinc oxide is milled to break up any lumps formed in the kiln and then drummed for shipment to the battery manufacturing facility.

C. Description of Zinc Recovery Process C.

The flowsheet for Zinc Recovery Process C is shown in Figure 8. The zinc electrodes are stored and crushed as in Process A. The crushed electrodes are then transferred to storage bins which serve as feed hoppers for the zinc recovery process.

The recovery method is a pyrometallurgical process in which the crushed electrodes are first reduced to fume off zinc vapor which is then oxidized to form zinc oxide. The resultant zinc oxide is collected by passing the gas stream through bagcollectors.

III. Description of Recycling Process for Zinc Chlorine Batteries.

The flowsheet for the process is shown in Figure 9. The zinc/chlorine battery recycle consists of recovery of the mechanical components, such as the pumps, reservoirs and battery plate stacks, and the chemical components, zinc, zinc chloride and chlorine.

The battery is first vented into a gas collection system. The zinc chloride electrolyte and the chlorine hydrate slurry are then drained into a collection tank. The solution is filtered and pumped to a storage tank.

The battery case is then opened, washed and dismantled. The various components are separated and inspected. Damaged or otherwise unusable components are discarded for sale or disposal.

The zinc electrodes, which may contain metallic zinc, are washed with hydrochloric acid to dissolve the zinc and then either reused or discarded.

All of the solutions, after passing through the collection tank, purification, filtration and storage, are sent back to the battery manufacturing operation.

FIGURE B. ZINC RECOVERY PROCESS C
- PYROMETALLURGICAL -

FIGURE 9. ZINC CHLORIDE BATTERY RECYCLE

IV. Description of Recycling Process for Zinc Bromine Batteries

The flowsheet for the process is shown in Figure 10. The zinc/bromine battery recycle consists of recovery of the mechanical components such as the pumps, battery stacks and storage tanks and the chemical components, zinc, zinc bromide electrolyte, and bromine.

The battery is first vented into a gas collection system. The electrolyte and bromine solutions are drained into collection tanks. These solutions, are filtered and sent to storage.

The battery is then opened, washed and dismantled. The various components are separated and inspected. Damaged or otherwise unusable components are set aside for sale or disposal.

Residual zinc in the electrodes is removed by washing with hydrobromic acid which is sent to the collection system. The electrodes are inspected and either sent for reuse or discarded.

The solutions, after adjustment to the required chemical composition, are sent to the battery manufacturing operation.

IV. Sodium-Sulfur Battery Recycling Process

The recycling process for the sodium/sulfur battery, as shown on Figure 11, consists of battery dismantling, cell separation, inner and outer cell dismantling, removal of the sodium/sodium polysulfide/sulfur and solvent recovery.

The batteries are brought from the collection and storage area and placed on a dismantling conveyor where the outer battery cases are removed. The cases, after inspection are either recycled or sent for disposal. The inner battery, consisting of the cell modules, is then dismantled. The cell connectors and other hardware are removed and the individual cells set onto a transfer conveyor.

FIGURE 10. ZINC BROMIDE BATTERY RECYCLE

FIGURE 11 - SODIUM/SULFUR BATTERY RECYCLE

The cell separating machine opens the cells and sorts the inner and outer cell components. The outer cell, consisting of the steel case and sulfur/polysulfide impregnated carbon felt, is further dismantled by removing the steel case. The sulfur/polysulfide is dissolved in solvent. The felt is drained of solvent and then rinsed with fresh (or recycled) solvent. The felt is drained, dried, and then sent to disposal. The cleaned steel outer cases are either recycled to the battery manufacturer or scrapped. Solvent solution is also used to wash the inner cell components which have been dismantled and separated. The inner cell components are drained and then rinsed with solvent. The components are drained and then dried to remove any residual solvent. The inner cell components, free of sodium polysulfide and solvent are sent to disposal. The cleaned inner cases, are either recycled to the battery manufacturer or scrapped.

The solvent, loaded with sodium polysulfide, is collected and then evaporated to recover the solvent and produce a sodium polysulfide product. The sodium polysulfide is drummed for shipment.

APPENDIX B - CAPITAL AND OPERATING COST SUMMARIES

Process 1 - Zinc-Acid Leach - 100,000 Batteries/Year

Product - 5,282 tons Zinc Oxide per Year

Capital Cost - \$4,833,000

Operating Costs Components - \$ per year

Raw Materials	3,392,900
Utilities	129,900
Labor Related	2,954,000
Capital Related	<u>821,300</u>
Total Operating Costs	\$7,298,100 per year

Process 2 - Zinc - Ammonia Leach - 100,000 Batteries/Year

Product - 4,020 tons Zinc Oxide per Year

Capital Cost - \$5,470,500

Operating Cost Components - \$ per year

Raw Materials	173,600
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Utilities	323,000
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Labor Related	3,161,000
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Capital Related	<u>930,000</u>
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Total Operating Cost	\$4,587,600 per year
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Process 11 - Sodium/sulfur - 100,000 Batteries per Year

Product 46,640 tons per year Na_2S_x per year

Capital Cost - \$10,605,100

Operating Cost Components - \$ per year

Raw Materials 3,258,600

Utilities 409,600

Labor Related 2,990,000

Capital Related 1,802,800

Total Operating Cost \$8,461,00 per year

Process 3 - Zinc Pyrometallurgical - 100,000 Batteries/Year

Product - 4,090 tons Zinc Oxide per Year

Capital Cost - \$4,772,600

Operating Cost Components - \$ per year

Raw Materials 127,500

Utilities 270,800

Labor Related 1,586,000

Capital Related 812,300

Total Operating Costs \$2,796,600 per year

Process 4 - Nickel Solution - Acid Leach - 100,000 Batteries Per Year

Product - 3,766 tons Nickel as Sulfate per Year

Capital Cost - \$4,670,100

Operating Costs Components - \$ per year

Raw Materials	1,969,900
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Utilities	111,300
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Labor Related	2,718,000
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Capital Related	<u>794,000</u>
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Total Operating Cost	\$5,593,200 per year
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Process 5 - Nickel Solution - Ammonia Leach - 100,000 Batteries per year

Product - 3,766 tons Nickel as Sulfate per Year

Capital Cost - \$5,009,900

Operating Cost Components - \$ per year

Raw Materials 2,581,600

Utilities 115,600

Labor Related 2,718,000

Capital Related 851,200

Total Operating Cost \$6,266,400 per year

Process 6 - Nickel Powder - Acid Leach - 100,000 Batteries per Year

Product - 8,107 tons Nickel Powder per Year

Capital Cost - \$11,306,200

Operating Cost Components - \$ per year

Raw Materials	2,420,500
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Utilities	1,905,700
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Labor Related	2,944,500
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Capital Related	<u>1,922,000</u>
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Total Operating Cost	\$9,192,700 per year
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Process 7 - Nickel Powder - Ammonia Leach - 100,000 Batteries per Year

Product - 3,749 tons Nickel Powder per year

Capital Cost - \$6,634,400

Operating Cost Components - \$ per year

Raw Materials	559,200
Utilities	1,597,000
Labor Related	2,944,500
Capital Related	<u>1,127,900</u>
Total Operating Cost	\$6,228,600 per year

Process 8:- Nickel Powder - Pyrometallurgical - 100,000 Batteries per Year

Product - 8,167 tons Nickel Powder per Year

Capital Cost - \$6,441,900

Operating Cost Components - \$ per year

Raw Materials	148,500
Utilities	463,600
Labor Related	2,229,000
Capital Related	<u>1,095,100</u>
Total Operating Cost	\$3,936,200 per year

Process 9 - Zinc/Chlorine - 100,000 Batteries per Year

Product - 8,423 tons Zinc Chloride per year

Capital Cost - \$1,622,100

Operating Cost Components - \$ per year

Raw Materials	123,700
Utilities	22,800
Labor Related	1,904,000
Capital Related	<u>275,200</u>
Total Operating Cost	\$2,325,700 per year

Process 10 - Zinc/Bromine - 100,000 Batteries per Year

Product - 10, 218 tons Zinc Bromide per Year

Capital Cost - \$2,240,000

Operating Cost Components - \$ per year

Raw Materials	314,700
Utilities	22,800
Labor Related	1,904,000
Capital Related	<u>381,000</u>
Total Operating Cost	\$2,622,500 per year