SYNTHESIS REPORT

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TITLE: **LEAD RECOVERY FROM LEAD OXIDE SECONDARIES**

PROJECT COORDINATOR: TECNICAS REUNIDAS, S.A.

PARTNERS:
UNIVERSITY OF LISBON
UNIVERSIDAD DE ALICANTE
DIFUS
T.N.O.
QUIMITECNICA

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1. TITLE AND AUTHORS

Under the auspicious of the CEU, a consortium composed of six European organisations has developed **the PLACID prows** into the Brite Euram II Programme. The title of the project is *"Lead Recovery From Lead Oxide Secondaries"* and has the acronym 'LEREFLEOS'. The project began on 1 January 1993 and was finished on 30 April 1996.

European organisations involved in the LEREFLEOS project has been:

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University of Lisbon	Portugal	Partner
Universidad de Alicante	Spain	Partner
Difus (DAPL)	U.K.	Partner
TNO-MEP	The Netherlands	Partner
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BRITE-EURAM 11 PROGRAMME

Proyect:

LEAD RECOVERY FROM LEAD OXIDE SECONDARIES

Partnership:

Técnicas Reunidas, S.A. (SPAIN), University of Lisbon (PORTUGAL), Universidad de Alicante {SPAIN), Difus (U.K.), T.N.O. (THE NETHERLANDS), Quimitécnica (PORTUGAL)

2. ABSTRACT

The PLACID process applied to the recycling of spent lead-acid batteries has been developed by an European Consortium within the Brite-Euram II programme. The findings and results of the project entitled "***Lead Recovery from Lead Oxide Secondaries***" ***is*** described in this paper.

Main aim of this project has been to develop the PLACID process, which is based on a novel technology able to deal with different lead bearing materials, mainly lead oxide secondaries: lead-acid battery pastes, lead fumes, furnace slags, etc., producing "four nines" pure lead in an efficient manner with a benign influence on the environment.

Basic developments of the process and pilot plant results are summarised. The description of the final feasibility study applied to a selected base case is also included.

An interesting global approach is proposed, integrating the PLACID process in the existing spent battery recycling pyrometallurgical facilities. This yields important improvements in the overall process. Grids and metallic parts would be easily treated by the conventional pyrometallurgical route, obtaining lead alloys, while battery pastes, slags and fumes would be fed to the PLACID line, obtaining pure electrolytic lead (> 99.99 %), ideal for battery paste manufacturing. Other advantages of this combination are greater overall lead recovery, lower operating costs, reduced waste production, environmentally safer residues and much better lead product quality.

Based on the results achieved, the Consortium establish two final conclusions. First, a new technological package to deal with scrap lead-acid batteries and other lead secondaries recycling has been fully developed in a soundly environmental way, technically feasible and economically attractive; and second, the integration of the PLACID process with conventional pyrometallurgical treatment is a feasible and very attractive alternative route for spent lead-acid battery recycling.

3. INTRODUCTION

This paper deals with the project entitled *"Lead Recovery From Lead Oxide Secondaries"* and has the acronym 'LEREFLEOS'. It is an Industrial Project 50% funded by the Commission of the European Union within Brite-Euram-II Programme. The 'LEREFLEOS' project began on 1 January 1993 and was finished on 30 April 1996.

Lead metal production and manufacturing industries produce large amounts of dusts and powders collected by several procedures. Those materials are mainly composed by lead oxides and lead metal, besides metallic impurities and matrix components.

Spent lead-acid batteries recycling also produces lead oxides from the battery pastes rather similar to those mentioned above.

Those lead oxide secondary materials are hazardous and difficult to handle by usual procedures. Existing processes to treat lead oxides inflict considerable damage on the environment, and the main aim of this project is to minimise the need for this environmental damage by developing a well-founded hydrometallurgical process for that purpose, it is called the PLACID process.

Main objectives of this project has been:

- lst. To design and demonstrate a reliable full process capable of coping efficiently with hazardous materials containing lead oxides and metalliclead.
- 2nd. To directly manufacture a commercially acceptable final product in high purity metallic lead, having a higher added value than that of lead ingot.
- 3rd. To promote water, common salt and other components full recycling and guarantee liquid effluents, if any, within the actual EC legal requirements.
- 4th. To minimise solid wastes production, obtaining non-hazardous residues.

4. TECHNICAL DESCRIPTION

4.1. PROCESS DESCRIPTION

The PLACID process is based on a novel technology able to deal with different lead bearing *materials*, mainly lead oxide secondaries: lead-acid battery pastes, lead fumes, furnace slags, oxided residues, etc., producing "four nines" pure lead in an efficient manner with a benign influence on the environment.

In this process, lead is leached in warm, slightly acidic, brine to form soluble lead chloride. This solution is purified by cementation with lead powder. Pure lead is then won from the lead chloride electrolyte on the cathode of the electrowinning cell and is collected. This electrolytic cell is the heart of the process and it was especially developed to give optimum performance. Hydrochloric acid is reformed in the cell and returned to the leaching bath; reagent net consumption in the process is irrelevant.

The conceptual description of the PLACID process is shown in the block diagram of the Figure 1.

The core of the process is the PLACID electrowinning cell that is shown diagrammatically in the Figure 2. This is a cell with two compartments, cathodic and anodic, separated by a permoselective membrane. Electrolysis produces spongy lead on the cathode and oxygen evolution on the anode being the proton ion the electrical current carrier. The produced HCl acidifies the spent catholyte which is returned to the leaching process. Lead sponge is taken out from the bottom of the cell by a belt conveyor.

4.2. FUNDAMENTALS

The fundamentals of the process are summed up in the following chemical reactions:

- Leaching: Equation (1) to (4)

$$PbO + 2HCl \Rightarrow PbCl_2 + H_2O \quad (1)$$
$$Pb + PbO_2 + 4HCl \Rightarrow 2PbCl_2 + 2H_2O \quad (2)$$
$$PbSO_4 + 2NaCl \Rightarrow PbCl_2 + Na_2SO_4 \quad (3)$$
$$Na_2SO_4 + 2HCl + Ca(OH)_2 \Rightarrow CaSO_4 + 2NaCl + 2H_2O \quad (4)$$

Main lead compounds present in the raw material are lead dioxide, lead oxide, lead metal and lead sulphate. The process uses the dissolution capability of the HC1 at low concentrations and the oxidation-reduction balance shown in reactions 1 and 2 to produce $PbCl_2$. Lead sulphate is directly solubilised in hot brine, controlling the sulphate level in the brine by mean of neutralisation with milk of lime, reactions 3 and 4.

As summary, all leaching reactions lead to $PbCl_2$ formation which is soluble in hot brine. Neutralisation with milk of lime controls sulphate and impurities building up.

- Purification: Equation (5)

$$MeCl_2 + Pb \Rightarrow PbCl_2 + Me \quad (5)$$

Purification uses cementation with lead powder to get the best quality achievable for the lead product. “Me” signifies any metallic impurity: Cu, Bi, As, Sb...

- Electrolysis: Equation (6) to (8)

Cathodic: $$PbCl_2 + 2e^- \Rightarrow Pb^o + 2Cl^- \quad (6)$$
Anodic: $$H_20 \Rightarrow 2H^+ + ½O_2 + 2e^- \quad (7)$$

GLOBAL: $$PbCl_2 + H_2O \Rightarrow Pb^o + 2HC1 + ½O_2 \quad (8)$$

Electrolysis produces lead metal through the cathodic reaction and water hydrolysis in the anode. Oxygen is released and proton ions cross the membrane regenerating the acidity of the catholyte, and thereby closing the brine loop.

The overall ideal reaction (9) summarises the process in steady state and with all the compounds in balance.

$$PbO \Rightarrow Pb + \frac{1}{2}O_2 \qquad (9)$$

4.3. THE 'LEREFLEOS' PROJECT

An intensive R&D work has been performed during 40 months project duration, aiming to achieve the general objectives of the project.

The work program was divided into fifteen tasks and covered three levels of development: laboratory studies, prototypes design and construction and pilot plant testing of the proposed flowsheet, with two steps concerning feasibility studies to make decision on the process viability.

Activities included in the R&D programme were as follows.

A) **Initial** definition of the proposed prms. Main sections of the process that required experimentation works were identified. The work programme was started up.

B) Searching lead oxide secondaries. An inventory of suitable feed materials in Europe, including contacting with producers and validation of the need for the PLACID process, was carried out.

C) Laboratory works aiming to develop and/or enhance the hydrometallurgical steps of the process were performed. A special care was devoted to theelectrowinning cell development.

D) Process simulation by computer model. This task provided a reliable and very useful tool for using in different steps of the process development: laboratory, pilot plant and industrial base case study.

E) Process re-definition. Based on the information obtained on laboratory works, the proposed process was revised and re-defined, performing a pre-feasibility study and defining the optimum flowsheet for the pilot plant experimentation.

F) Piloting the process. A complete pilot plant was installed to test the proposed process. This facility included the main steps of the process, being remarkable the new developed electrowinning cell with 16 Kg/h electrolytic lead production.

G) Final feasibility study. The final evaluation of the developed process, applied to a selected base case was accomplished. The feasibility study included the basic engineering package, determining operating and investment costs, as well as, process economics, performing also a sensitivity analysis on the results.

H) Environmental aspects. A special care was devoted to the environmental aspects of the PLACID process, aiming to produce non hazardous residues or effluents. New techniques to deal with the bleed of the process were developed, yielding the complete recycling of components and no production of any liquid effluent at all.

5. RESULTS

5.1. EUROPEAN LEAD MARKET SEARCHING

Lead-acid battery pastes and lead smelting fumes and slags were identified as the most appropriate feed-materials to the PLACID process, focusing all the attention of the Consortium in contacting this kind of lead recycling companies.

The use of "four nines" lead grade seems not necessary to manufacture standard car batteries with sufficient life-time, according to the information supplied by battery recycling firms. Perhaps it is true for the metallic parts of the battery, however, battery makers intend to use primary lead (high purity) for battery pastes manufacturing.

The situation could be critical in a near future where lead-acid batteries could be considered as the primary battery for powering near-term electric road vehicles, so a new generation of sealed lead batteries is presently under development. Essential to this new VRLA (Valve Regulated Lead-Acid) type of batteries is the use of very pure lead which must presents low level of gas forming elements. For this application, a 99,99 % lead quality might be indispensable.

It is clear that conventional pyrometallurgical battery recycling plants are not able to produce "four nines" lead in a profitable route. For instance, bismuth and silver are almost impossible to be removed by this method.

The Consortium detected a clear opportunity for the PLACID process development because this new process can produce "four nines" lead at low cost and having benign influence on the environment.

5.2. BASIC DEVELOPMENTS

The operating conditions of the leaching-neutralisation step were clearly defined at 100 L batch experiments. Lead leaching efficiency was higher than 99.5 %, feeding battery pastes and lead fumes or slags. Solid-liquid separation parameters were determined. A Standard Leaching Test procedure was defined and checked.

The operating conditions of the purification step by cementation with lead powder was also completely defined. Globally, the purified pregnant brine got by the process is able to produce +99.99 % electrowon lead.

The electrowinning cell components and the operating conditions were chosen after a carefully laboratory and bench scale prototype study. The system performance required for obtaining top quality lead and satisfactory current efficiencies were well established. Energy consumption was as expected, about 0,8 Kwh(DC) per Kg of lead.

Several routes for lead sponge handling were studied to produce different commercial lead products. The key processes are extrusion, agglomeration and melting and casting when the final products are to be rods and bars, pellets and ingots respectively.

The bleed of the close circuit is treated to avoid any build up of the impurities and to keep in balance the water and components of the process. Lead is efficiently recycled from the bleed by PbS precipitation with sodium hydrosulphide. Iron removal by goethite precipitation for recycling back to the leaching step is over 99% efficiency.
Removal of heavy metals through precipitation by lime addition is the cheapest method of dealing with small traces of non-ferrous metal impurities. A negligible amount of solid waste is produced.

Evaporation has been chosen from among several processes for water recycling. Contacts with suppliers confirmed that available evaporation processes do not require any additional research work.

5.3. PILOTING THE PROCESS

A complete pilot plant, having a production of 16 Kg/h electrolytic lead and 1,000 L/h flowrate of the main circuit, was installed; a perspective view of the PLACID electrolytic cell is presented in the attached photograph. The principal sections of this facility were:

Feed preparation
Leaching-neutralisation
Cementation
Electrowinning
Lead sponge handling
Bleed treatment

The work programme carried out included four experimental campaigns having a duration of twelve continuous days each. Operating time was 24 hours a day.

The starting up campaign of the pilot plant was satisfactory accomplished, proving the process fundamentals at that scale. Obtained data were very approximated to the theoretical material balance, showing the good design works performed. First run was a very important training for the operators because they acquired a great experience about the equipment and process behaviour that was very useful for the next trials.

Main objectives of the **second run** were to study raw material, equipment and process behaviour, determining the principal variables and their effects on the process performance. A special care was devoted to the PLACID electrowinning cell, which is the heart of the process. Obtained results were satisfactory, producing about 1,600 Kg electrolytic lead with 99.988 % Pb. Only minor operating problems appeared in this run.

Process was optimised during the **third run**. Only **slightly changes** in process variables were performed, adjusting the operating conditions to attain the steady state at least 100 accumulated hours, obtaining the frost process data set, parameters and performance.

The pilot plant was running very efficiently and without any relevant operating problem. The behaviour of the electrowinning cell was satisfactory, confirming the good design of this equipment. About 2,200 Kg electrolytic lead were produced, having 99.994 % Pb.

Process performance were demonstrated in the **fourth campaign. The** plant parameters were adjusted and kept to steady state conditions. It was aiming to get at least 200 hours contiguous operation at steady conditions.

Relevant results achieved were the following:

+ Lead recovery:	99.7 %.
+ Electrolytic lead production:	3,100 **Kg**.
+ Electrowinning:	
Operating time=	204 **hours.**
C. D.=	1200 **A/m²**.
Cell voltage=	3.5 **V.**
Current efficiency =	**100 %** approx.
Energy Consum. =	0.9 **Kwh/Kg Pb**.
+ Lead quality:	**99.996 %.**

Average lead sponge composition (ppm) was:	Cu	Sb	As	Sn	Bi
	9	19	2	1	2

Main results of this 4th. run demonstrated successfully the core technology of the PLACID process, achieving the main objectives of the project and obtaining the necessary technical and designing information for its scaling up.

5.4. FINAL EVALUATION STUDY

After several contacts with lead recycling companies discussing topics related to the actual pyrometallurgical route, a Base Case was selected for the final feasibility study of the PLACID process based on 'realistic conditions'.

The selected base case implementation is schematically shown in the Figure 3. It presents the following relevant characteristics:

a. Although the PLACID process could be implemented alone for battery recycling, there are technical as well as economic and environmental advantages in using it integrated with a pyrometallurgical facility so, this has been the selected alternative.

In a combined PYRO + PLACID process, for example, the amount of waste that might be disposed off is half of the actual production, global lead recovery is increased from 95% to above 99.5%, operating costs can be shared, and hence, minimised, etc.

b. Lead productions are the following:

Pure lead	18,700 t/y (20.000 t/y electrolytic lead)
Alloy Pb-Sb	18,350 t/y
Bismuthed lead	950 t/y (for shot production)
Total	38,000 t/y

c. The energy consumption has been optimized. After a detailed study about electricity cost optimization it was decided to implement a co-generation energetic plant by natural gas to supply electricity and heat to the process. It has been a very attractive option, reducing substantially the operating cost.

The chosen base case was developed in detail, obtaining material and heat balances, consumable, manpower requirements, etc. Operating costs were estimated from the previous information.

Main consumption are shown on the Table I. The most important is natural gas to produce electricity for the electrolytic process through the co-generation plant.

Table I. Main Consumption

CONCEIT	VALUE
Lime Kg/T Pb	143
Sodium Hydrosulphide, Kg/T Pb	1.5
Process Water, m^3/t Pb	2.2
Natural Gas. Mcal/h	4,150

An operating cost break down at current prices in Spain is depicted in Table II in US Dollars at the prevalent exchange rate, 128 PTA per USD. Main cost is that of electricity production from natural gas. A 10% contingency factor has been allowed.

The raw material cost was estimated, based on scrap battery and scrap lead market prices as 95$/t of Pb. A lead ingot price 665 $/t of Pb has been assumed based on LME price at date, January 1996.

Table II. Operating Cost Break Down

CONCEPT	$/T Pb
Utilities	46
Reagents	9
Residue disposal	20
Labour	42
Maintenance	19
Contingency	14
TOTAL............................	150

The estimated total investment cost, based on a good practice engineering standard with a +25 % contingency factor is '23 Million USD. The conservatism of the raw material cost and the high contingency factors assumed make this study fairly conservative.

Discounted cash flow calculations (DCF) were undertaken to determine the economic value of the process. A net discount rate of 13% per annum was added after tax deflacted cash flow, and the net present value was found to be 30 million US$. The fact that the lead market price was assumed constant against a background of 5 % per annum inflation is an example of the conservatism of this calculations.

Relevant results of the DCF study are the following:

Table III. DCF Study Results

Item	value
Total Investment Cost, million US$	23
Net Present Value, million US$	30
Pay-back time, years	3.2
Internal Rate of Return (Net), %	24.1

The payback time was found to be 3.2 years, which is a satisfactory result. The internal rate of return, reaches 24.1% per annum after payment of tax, which is considered to be a very high rate.

As a general conclusion, can be said that the Base Case presents a very attractive economic figures so, it could facilitate the industrial implementation of this new technology.

Based on the computer DCF program, a sensitivity analysis of the base case to the main economic parameters has been performed. Obtained results show that process economics are mainly sensible to the lead price and total investment cost. However, the PLACID process presents very attractive economics even considering worse data; for example, with a -20 % lead price, +20 % TIC and +10 % electricity cost, the IRR value is 18.3 %.

5.5. ENVIRONMENTAL ASPECTS

The combination of a PLACID line together a PYRO line presents important environmental advantages, such as:

Less amount of solid residue production, 250 versus 350 kg/t Pb and lower lead content in the residue, [Pb] < 1.5 %, in comparison to the existing slags from smelters which present about 4-5 % Pb as a minimum, being classified as hazardous,

The PLACID process works in a close circuit with no liquid effluent emission.

Gasses emissions are minimized because only metallic lead is melted at low temperature operation (500 'C), while in the conventional pyrometallurgical process battery pastes must be fed into the furnaces at high temperature (1100 'C), with a potential production of pollutant gases like SO_2, HCl, dioxins, etc..

The PLACID process opens the possibility to eliminate a great variety of secondary lead oxides by-products and/or residues coming from different lead related industries that presently are disposed off because their pyrometallurgical treatment is not feasible.

Other important considerations related to the environment is the capability of the PLACID process to maintain a continuously sustainable cycle for lead-acid battery recycling in present and foreseen conditions because the produced electrolytic lead is top quality, ideal for battery pastes manufacturing.

6. CONCLUSIONS

Initial foreseen objectives of this project were fully achieved.

After more than 1,000 hours operating time, the results obtained in the pilot plant trials have demonstrated successfully the core technology of the PLACID process.

About 10,000 Kg of electrolytic lead were produced after four continuous operating campaigns.

The process is able to treat different raw materials. A blend of battery pastes and lead fumes were fed to the pilot plant. The leaching efficiency was above 99.5 % Pb recovery.

The quality of the electrolytic lead produced is maximum. Above 99.99% lead grade was reached working at nominal conditions. For instance, bismuth content of the lead product was 2 ppm.

Although the PLACID process could be implemented alone for battery recycling, there are technical as well as economic and environmental advantages in using it integrated with a pyrometallurgical facility.

The final evaluation study of the PLACID process, applied to a selected Base Case producing 20,000 T/y electrolytic lead presented very satisfactory results:

+ Obtained operating cost represents a saving of about 25 % in respect to existing pyrometallurgical plants and obtained lead quality is “four nines”.

+ Outputs from the Discount Cash Flow study show very attractive economic data: pay-back time of 3.2 years and net internal rate of return of 24.1 %, for a total investment cost of about 23 million US$.

The PLACID technology presents important environmental advantages in respect to conventional pyrometallurgical lead recycling plants, such as: non-hazardous residue production, lower quantity and lead content in the residue, no liquid effluent emission, no gasses and dusts evolution.

As a general conclusion can be said, that a new technological package to deal with scrap lead-acid batteries and other lead secondaries recycling has been fully developed in a soundly environmental way, technically feasible and economically attractive.

7. ACKNOWLEDGEMENTS

The European organisations that have developed the LEREFLEOS project into the Brite-Euram II programme show their gratitude to the European Community for its encouragement, support and partial funding of the project.

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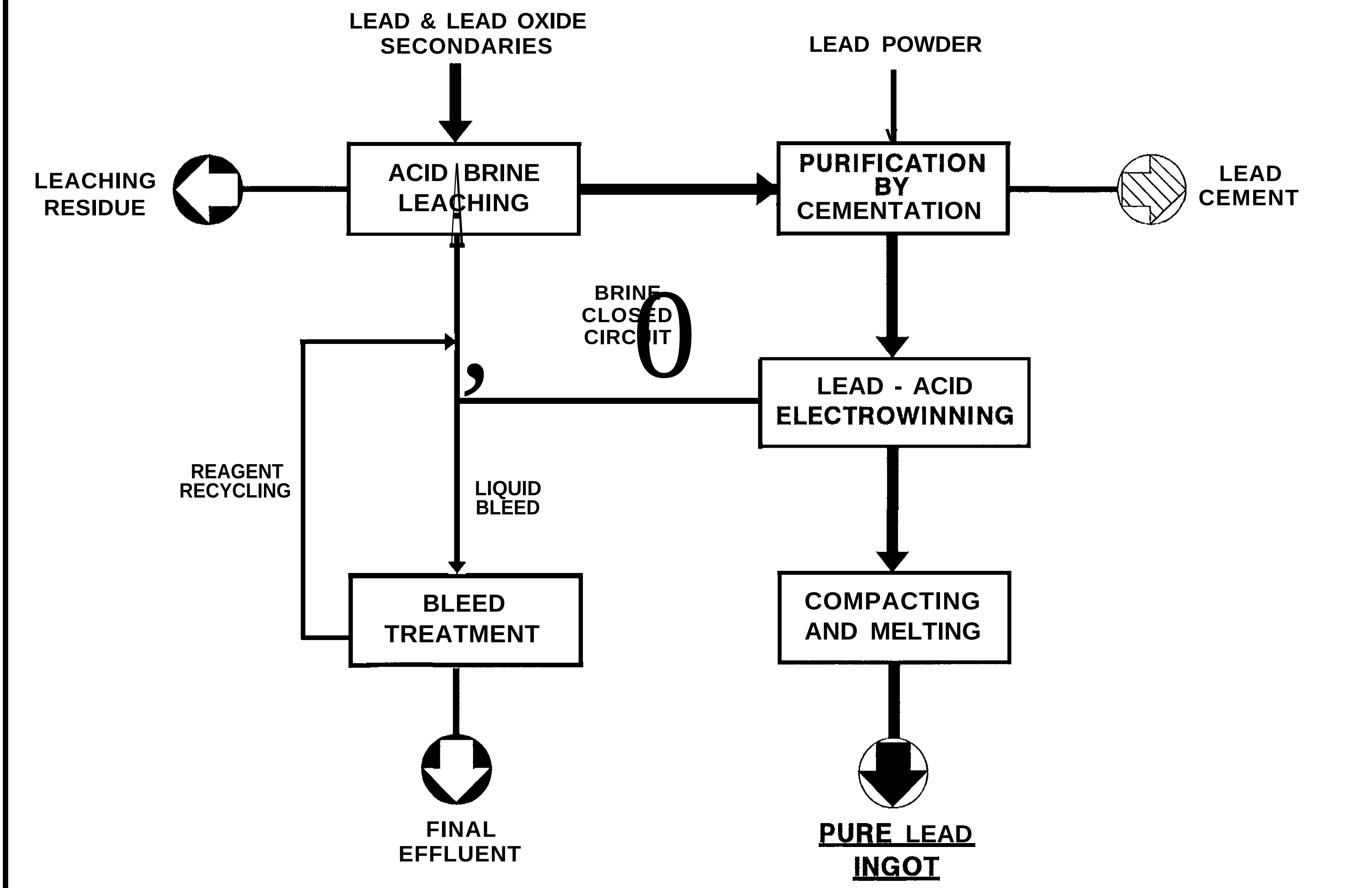


FIGURE 1.- THE PLACID PROCESS. CONCEPTUAL BLOCK DIAGRAM

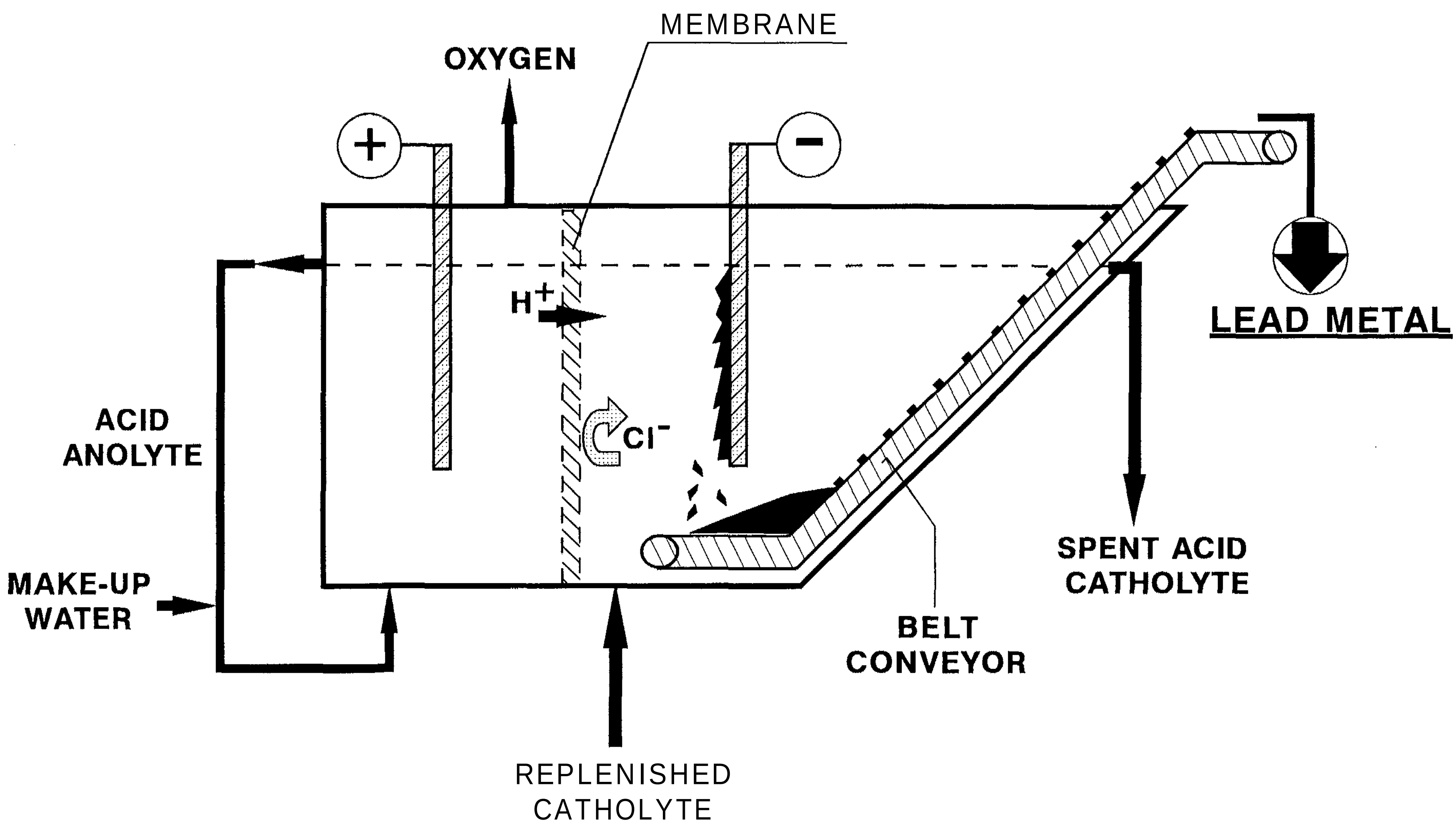
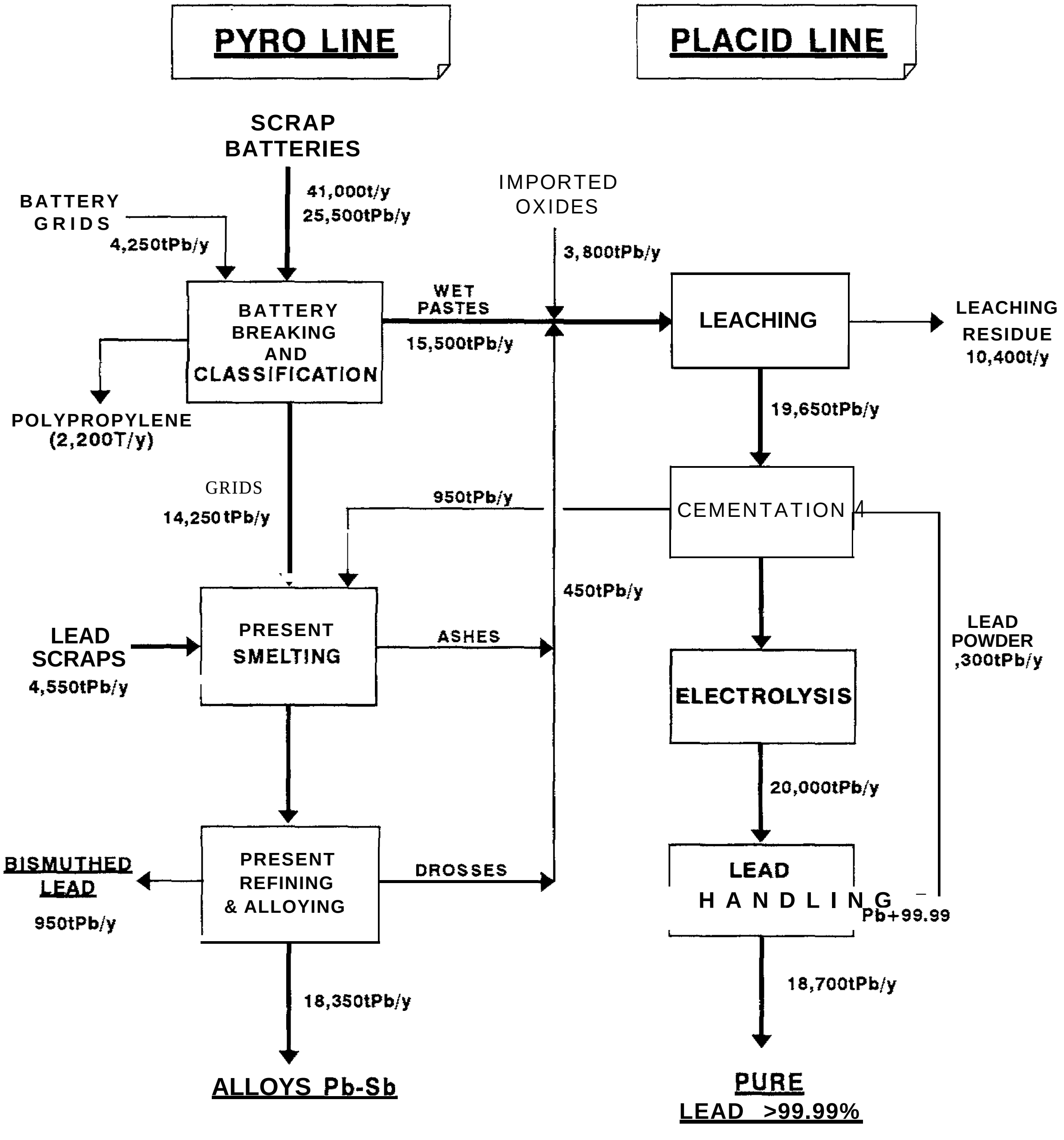


Figure 2 - The PLACID **Electrowinning** Cell



Kg OF NON-HAZARDOUS RESIDUE/t OF PRODUCT = 275

LEAD PRODUCTION = 38,000 t/y
GLOBAL RECOVERY = 99.8%

FIGURE 3.- BASE CASE IMPLEMENTATION

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