## For Inspection Centra

### \* GOVERNMENT OF INDIA: THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17. Specification No. 59250, 29th December 1956. (Accepted 14th December 1957.)

ELECTROWINNING OF LEAD FROM LEAD SULPHATE.

VEERARAGAVA ARAVAMUTAAN AND PALAMUTHI RAMASWAMI RAJA-BIMAN BEHARI DEY, GOPALAN, ALL OF THE CENTRAL ELECTRO-CHEMICAL RESEARCH INSTITUTE, KARAIKUDI, INDIA, ALL CYTIZENS.

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This invention relates to the electrowinning of lead from lead sulphate which has hitherto been attempted for a long time without much success. Possible raw materials include electrolytic zinc plant tailings, lead sulphate in the flue dust, lead sulphate from sulphuric acid plants, lead sulphate from stores but

These sheets have grooves for the two anodes and cathode. The electrodes are arranged at one end of the vessel leaving space at the opposite end for the stirrer (not shown). Leached plumbite solution is fed into this vessel and the electrolysis is conducted between 85° and 90°C. For

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GOVERNMENT OF INDIA THE PATENT OFFICE, 214, Lower Circular Road, Calcutta-17.

Conditions. by the leaching of with 26% caustic int up to 45 grams

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wround the anode.

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The following misprint in the above specification should be corrected :--

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In page 1, between lines 3 and 4, in the heading-

Insert "Council of Scientific and Industrial Research, Old Mill Road, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860)".

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described in 1 ft. is found to

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J.H./lb. of lead

MGIPC-S5-32 Patent/60-3-10-60-100.

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mesas deposited, under the conditions described in 1 to 9;

R. VASUDEVA PAI,

Deputy Controller of Patents and Designs.

Price: TWO RUPEES.

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LELECTROWINNING OF LEAD FROM LEAD SULPHATE.

BIMAN BEHARI DEY, VEERARAGAVA ARAVAMUTHAN AND PALAMUTHI RAMASWAMI RAJA-GOPALAN, ALL OF THE CENTRAL ELECTRO-CHEMICAL RESEARCH INSTITUTE, KARATRUDI, INDIA, ALL INDIAN CITIZENS.

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This invention relates to the electrowinning of lead from lead sulphate which has hitherto been attempted for a long time without much success. Possible raw materials include electrolytic zine plant tailings, lead sulphate in the flue dust, lead sulphate from sulphuric acid plants, lead sulphate from storage battery scrap and also from primary sources, viz., galena concentrates. In previous attempts brine was used as the leaching agent for lead sulphate. Handling of hot brine, containing lead in solution, evolution of chlorine during electrolysis etc., stood in the way of commercial success. Realising the enormous difficulties in brine leaching, Prof. Colin. G. Fink attempted the leaching of lead sulphate wastes with caustic soda solution. [Fink C. G., and Greenspan L., Tran Am. Elec. Chem. Soc. 63, 465-473 (1930).]

Prof. Fink employed caustic soda of 20-24 per cent. strength for leaching. The resulting plumbite was electrolysed employing lead-silver anode and lead cathode. An alundum diaphragm separated the analyte from the catholyte. At 60-70°C., employing a current density of 15 amps/sq. ft., a current efficiency of 95-98 per cent. was reported. No colloids other than glue were tried.

The present invention consists in the electrowinning of lead from lead sulphate such as from battery wastes, battery dust, galena concentrates or the like by conducting electrolysis between 85° and 90°C, using leach-liquor obtained by leaching pure lead sulphate with 20 per cent. caustic soda, containing up to 45 grams per litre of Pb as electrolyte.

Advantages claimed for the new process:

- vi. In Prof. Fink's process 0.8 lb. of oxides of lead were formed for every lb. of lead deposited as metal, while in the new process, all the lead value is recovered as pure metal and none is used up in the formation of oxides of indeterminate composition the overall recovery is very high.
- (2) Prof. Fink employed alundum as diaphragm in alkaline medium. While alundum might serve well for a limited number of experiments, it is not resistant to the prolonged action of alkali and cannot be employed for large scale work. In the new process, blue asbestos rope is used as the diaphragm. Besides being corroion resistant, it completely eliminates the oxidation at the anode by nascent oxygen, and thus ensures quantitative recovery of lead as metal.
- (3) Rosin has been found to be the most suitable addition agent. Good and adherent but peclable deposits are obtained. Glue is not so efficacious as rosin for use in an alkaline medium.
- (4) The process of caustic leaching and subsequent electrowinning which has been worked out for application to lead sulphate wastes from batteries, is quite flexible and can easily be adapted for application to other lead wastes or ores.

Description of the process:

The invention will now be described with the help of accompanying drawings wherein Figure 1 shows a sectional view of an electrolytic cell used in the process.

A stainless steel vessel 1  $(9'' \times 8'' \times 6'')$  i.d.) constitutes the cell. Two graphite anodes 2  $(10'' \times 3'' \times 1'')$  are wrapped sufficiently with blue ashestos rope 3, and kept in position by means of a grooved perspex sheet 7 kept at the bottom of the vessel. A stainless steel cathode 5  $(9'' \times 3'' \times 1/6'')$  also rests on the perspex sheet and has a frame 6 also made of perspex, to avoid "treeing". All the electrodes are kept rigidly without any lateral play by two strips of perspex 4, fixed at the top of the vessel.

These sheets have groones for the two anodes and cathode. The electrodes are arranged at one end of the vessel leaving space at the opposite end for the stirrer (not shown).

Leached plumbite solution is fed into this vessel and the electrolysis is conducted between 85° and 90°C. For this purpose, the cell is kept inside another bigger vessel containing water, the whole arrangement being kept on a hot plate. All the experiments are carried out for 8 hours. Rosin is added at the beginning.

Operating Data.

Optimum Conditions.

Electrolyte:

Leach-liquor obtained by the leaching of pure lear sulphate with 20% caustic soda, with lead content up to 45 grams per litre of pb.

Addition agent . 0.25% to 0.5% of rosin in solution.

Cathode: . . Stainless Steel.

Anode: . . Acheson graphite.

Diaphragm . . Blue asbestos wrapped around the anode.

Voltage: • . . 1.3 to 1.6.

Current density . 8-10 amps./sq. ft.

Current efficiency . 95-

. 95-100%.

Power constmption 0.17 K. W. H./lb.

The cathode product is found to be of high purity.

The following are among the noteworthy features of his process:

- 11) Lach-liquor obtained by leaching pure lead sulphate with 20 per cent, caustic soda, containing upto 45 grams per ittre of Pb, is employed as a suitable electrolyte fr the electrowinning of lead from lead sulphate from battery wastes, battery dust, galena concentrates etc., at temperatures between 85-90°C.;
- (2) standess steel is the best material of construction for the electrowinning of lead from sodium plumbite solution containing sodium sulphate mentioned in (1);
- (3) plates of Acheson graphite, well wrapped with blue asbestos rope (Bestobel), are used as anodes, being free from disintegration and decay under the conditions of electrolysis,
- (4) blue asbestos (commercially called Bestobel) is a suitable diaphragm material for the electrowinning of lead and there is no increase in voltage and no oxides of lead are formed under the conditions described in 1 to 3 above;
- (5) stainless steel, covered upto 1/16" on three sides by perspex is suitable for use as cathode, under conditions mentioned in 1 to 4. There is no "treeing" of lead. Perspex is the best material for the stop off at the edges to prevent 'treeing' under conditions described in 1 to 4;
- (6) rosin at a concentration of 0.25 to 0.5 per cent, in solution is found to be the best addition agent for the electrowinning of lead from the electrolyte under conditions mentioned in 1 to 5:
- (7) under the conditions of electrolysis described in 1 to 6, a current density of 8-10 amps./sq.ft. is found to be optimum;
- (8) under the conditions described in 1 to 7, the current efficiency is between 95 and 100 per cent.;
- (9) the cell voltage is between 1.3 and 1.6 under the conditions described in 1 to 8;
- (10) the power consumption is 0.17 K.W.H./lb. of lead metal deposited, under the conditions described in 1 to 9;

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- (11) under the conditions of electrowinning described in 1 to 10, the overall recovery is more than 85 per cent. and a "strip" of 30 grams per litre of Pb can be obtained;
- (12) process for the electrowinning of lead is specially and particularly suited for the treatment of battery wastes, battery dust (including process wastes), and also for the electrowinning of lead from galena concentrates. The latter may be converted into lead sulphate by any one of the known methods such as by air-oxidation at about 500°C., or brought into solution by "high pressure leaching" in autoclaves.

#### We claim:

- 1. A process for the electrowinning of lead from lead sulphate such as from battery wastes, battery dust, galena concentrates or the like by conducting electrolysis between 85° and 90°C. using leach-liquor obtained by leaching pure lead sulphate with 20 per cent caustic soda, containing up to 45 grams per litre of Pb as electrolyte.
- 2. A process as claimed in Claim 1 wherein a stainless steel cathode is used for the electrolysis.
- 3. A process as claimed in Claim 1 or 2 wherein plates of Acheson graphite, well wrapped with blue asbestos rope (commercially known as Bestobel) are used as anodes.
- 4. A process as claimed in any of the preceding claims wherein blue asbestos (commercially called Bestobel) is used as diaphragm material for the electrowinning of lead.
- 5. A process as claimed in any of the preceding claims wherein stainless steel covered up to 1/16" on three sides by perspex is used as cathode.

- 6. A process as claimed in any of the preceding claims wherein rosin at a concentration of 0.25 to 0.5 per cent, in solution is added to the electrolyte.
- A process as claimed in any of the preceding claims wherein the current density is 8-10 amps./sq. ft.
- 8. A process as claimed in any of the preceding claims wherein the current efficiency is between 95 and 100 per cent.
- 9. A process as claimed in any of the preceding claims wherein the cell voltage is between 1.3 and 1.8.
- 10. A process as claimed in any of the preceding claims wherein the power consumption is 0.17 K.W.H./Ib. of lead metal deposited.
- 11. A process as claimed in any of the preceding claims wherein the overall recovery is more than 85 per cent. and a "strip" of 30 grams per litre of Pb is obtained.
- A process for the electrowinning of lead from lead sulphate substantially as hereinbefore described.
- 13. Lead whenever obtained from lead sulphate by electrolysis according to a process substantially as hereinbefore described.

R. BHASKAR PAT,

Patent Officer,

Council of Scientific & Industrial Research.

Dated this 24th day of December 1956.

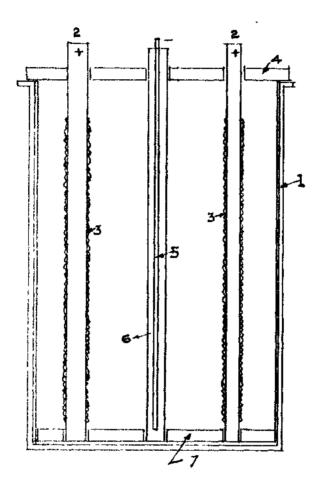


Fig. I.

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