ELECTROLYTIC METHOD FOR PRODUCING TETRAETHYL LEAD

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Our invention relates to an improved method and apparatus for the electrolysis of organo-metallic compounds. More particularly, our invention relates to a superior method of producing tetraethyl lead and an apparatus to be used in such process.

It has previously been suggested to produce metal alkyls by the electrolysis of alkyl complexes of a metal or metals other than the product metal alkyl. Such processes are described in Belgian Patents Nos. 575,595 and 575,641 and British Patent No. 797,093. These processes, operated in conventional equipment, while being effective, are not completely desirable as industrial processes in that the conventional electrolytic cell must be shut down periodically so as to permit removal and replacement of the spent anodes. Furthermore, it is also necessary to remove periodically the cathodes due to the build-up of undesirable deposits thereon. In addition to interrupting the process for such removals, the removal operations themselves are difficult and dangerous due to the presence of very poisonous products, e.g., tetraethyl lead.

It is an object of our invention to provide a continuous process for the production of tetraethyl lead by electrolysis and an apparatus for use in the process of our invention.

The method of our invention includes flowing a liquid phase electrolyte comprising a tetraethyl derivative of an alkali metal and aluminum through an electrolytic cell. A liquid lead amalgam is also flowed through the cell as the anode of the cell. The electrolyte is then electrolyzed by imposing a voltage across the anode and cathode of the cell thereby depositing the alkali metal as a liquid phase at the cathode and producing tetraethyl lead at the anode where the lead of said amalgam is in liquid phase. The spent amalgam is removed from the cell. The spent electrolyte, liquid alkali metal, and tetraethyl lead are also removed from the cell and the tetraethyl lead is separated from the spent electrolyte and the liquid alkali metal. Fresh electrolyte is added to the cell and the separated tetraethyl lead is recovered as product.

In defining the electrolyte of our invention the term "tetraethyl derivative of an alkali metal and aluminum" is to be construed as indicating an organo-metallic complex rather than a mixture of derivatives of the two metals. Thus, in a particularly advantageous employment of the process of our invention, the alkali metal of the electrolyte is sodium and the tetraethyl derivative of sodium and aluminum is a complex having the formula

\[ \text{NaAl}(\text{C}_2\text{H}_5)_2 \]

Desirably, the alkali metal of the complex employed in our electrolyte is an alkali metal having an atomic number of 11 to 19, e.g., sodium and potassium. Sodium, however, is strongly preferred.

The amalgam which is employed as the anode of our cell, is normally composed of 5 to 20 weight percent of lead, however, a greater or lesser amount of lead can also be employed. In operation, the amalgam and electrolyte enter the cell at a temperature within the range of from about 100°C to about 120°C, or perhaps somewhat more and are maintained within this temperature range throughout the cell. The temperature, however, should not be so high as to cause decomposition of tetraethyl lead.

In the application to which our process is particularly suited, namely, the production of tetraethyl lead, wherein the electrolyte contains a sodium and aluminum tetraethyl complex, the liquid sodium formed in the process is of approximately the same density as the electrolyte. To insure that the sodium, as produced, will rise to the top of the cell, it is necessary to maintain a concentration of the product (tetraethyl lead) in the electrolyte, thus maintaining a density greater than that of the sodium. Furthermore, in the production of tetraethyl lead, if only sodium-aluminum tetraethyl is used as the electrolyte, triethyl aluminum \((\text{Al}(\text{C}_2\text{H}_3)_2)\) will be released in solution. Sodium will then react with the triethyl aluminum depositing free aluminum, which would in turn build up sufficiently to short-circuit the cell. To obviate this difficulty a small amount of a scavenger compound which will react with the aluminum triethyl without depositing aluminum is needed in the electrolyte. A suitable compound for this use has the formula

\[ \text{MAI}(\text{C}_2\text{H}_5)_2 \]

where \( M \) is an alkali metal, preferably sodium or potassium, and where \( R \) is an alkyl group with 2 to 8 carbon atoms. This type of compound is described in Belgian Patent No. 575,641. The following equation is illustrative of the reaction of such a compound with aluminum triethyl:

\[ \text{Al}(\text{C}_2\text{H}_3)_2 + \text{MAI}(\text{C}_2\text{H}_5)_2 \rightarrow \text{NaAl}(\text{C}_2\text{H}_5)_2 + \text{Al}(\text{C}_2\text{H}_3)_2 \]

Such scavenger compound is present in the electrolyte in a quantity sufficient to provide at least 4 moles of compound for each mole of tetraethyl lead formed. The compound will usually constitute from about 2 to about 10% by weight of the electrolyte; however, it can also be present in greater quantities. The \( \text{NaAl}(\text{C}_2\text{H}_5)_2 \) OR is regenerated from \( \text{Al}(\text{C}_2\text{H}_3)_2 \) OR by adding NaH and C6H6, as described in Belgian Patent No. 575,641.

Thus, for the production of tetraethyl lead the electrolyte of our invention comprises advantageously the sodium-aluminum tetraethyl complex together with tetraethyl lead and a compound of the formula

\[ \text{NaAl}(\text{C}_2\text{H}_5)_2 \]

preferably the tetraethyl lead concentration is about 5 to 15% by weight of the electrolyte entering the cell and the \( \text{NaAl}(\text{C}_2\text{H}_5)_2 \) OR compound is about 2 to 10% by weight of the electrolyte. The apparatus of our invention includes an electrolytic cell to be employed in the process described above. The cell of our invention comprises an electrically conductive, generally flat, inclined cell bottom for directly supporting the flowing amalgam described above. An inlet means for said amalgam is provided at the upper end of the inclined cell bottom and a discharge means for the spent amalgam is provided at the lower end of the inclined cell bottom opposite the inlet means. An electrically conductive, generally flat, inclined cell top is provided as the cathode for the cell. Advantageously, the cell top is inclined similarly to the cell bottom and is parallel to the cell bottom. Such members are generally spaced from about 1/8 to 2 inches apart. An inlet means and an outlet means for the electrolyte are provided at the opposite ends of the cell.

The inclined top of the cell causes the liquid metal formed at the cathode to flow towards the electrolyte outlet means; whereby it is continuously removed from the cell together with the spent electrolyte and tetraethyl lead product. The top portion and bottom portion of the cell are electrically insulated from each other.
The voltage applied to the electrodes of our apparatus must be sufficiently low so as to prevent transferring mercury from the amalgam (anode) to the cathode. We have found that when the top and bottom members are spaced apart by 2 inches, an acceptable range is approximately from 1 to 3 volts. There is, however, no operable limit in regard to current density. It is merely a matter of increasing production as the top and bottom members are moved together at a given voltage and the amperage varies only with electrode spacing for a given electrode surface area and electrolyte composition.

In order to understand our invention more completely, reference is made to the attached drawing in which:

FIGURE 1 is a plan view of a particularly advantageous embodiment of the cell of our invention.

FIGURE 2 is a sectional view of the embodiment of our taken along line 2—2 of FIGURE 1.

FIGURE 3 is a flow diagram illustrating the process of our invention.

FIGURES 1 and 2 show the structure and operation of the cell of our invention. Structurally our cell includes a top member 10 and a bottom member 12 (not seen in FIGURE 1). Each of the members 10 and 12 are fabricated of an electrically conductive material and insulated from each other by gasket 14, which also serves as a liquid tight seal between members 10 and 12. It will be noted that gasket 14 extends only about the periphery of top and bottom members 10 and 12 and is not a diaphragm-like device interposed between the operating surfaces of the anode and cathode.

As shown in FIGURE 2, bottom member 12 is provided with an amalgam inlet well 16 and amalgam outlet well 18 located at opposite ends of bottom member 12. Intermediate to inlet and outlet wells 16 and 18 is located an inclined amalgam support 20 of a generally rectangular shape. Support 20 is inclined downwardly along its longitudinal dimension from the inlet well 16 to the outlet well 18 with a slope of 1:100.

Separating inlet well 16 and support 20 is a sharp-crested vertical inlet weir 22. Also separating support 20 and outlet well 18 is a sharp-crested vertical outlet weir 24. Intermediate inlet and outlet weirs 22 and 24 are a series of sharp-crested vertical weirs 26 positioned at equal distances along support 20 transverse to its longitudinal dimension.

Positioned along the exterior surface of amalgam support 20 and electrically connected thereto are a plurality of bus bars 28. Each of these bus bars 28 is electrically interconnected, one to the other, by a plurality of connecting rods 30. The entire bus bar system is also connected to a source of electric power by means of conductor 32.

The amalgam inlet well 16 of bottom member 12 is provided with a downwardly opening amalgam inlet pipe 34 positioned such that its opening is substantially lower than the crest of inlet weir 22. At the opposite end of bottom member 12, the amalgam outlet well 18 is provided with an amalgam outlet pipe 36 located near the bottom of outlet well 18 and substantially lower than the crest of outlet weir 24. Intermediate amalgam outlet pipe 36 and the crest of outlet weir 24 amalgam outlet well 18 is also provided with an auxiliary product outlet pipe 37.

The top member 10 is provided with a product riser 38 located at the same end of the cell as, and positioned above, amalgam inlet well 16. A product outlet means 39 consisting of a plurality of upwardly opening pipes 40, communicating with the interior of a manifold 42, and a discharge pipe 44 also communicating with the interior of manifold 42 is located within product riser 38. At the opposite end of top member 10 and positioned above amalgam outlet well 18 is located the electrolyte inlet means 45 consisting of a plurality of horizontally opening pipes 46 communicating with the interior of manifold 48 and inlet pipe 50 also communicating with the interior of manifold 48.

The portion of top member 10 intermediate product riser 38 and electrolyte inlet means 45 is inclined downwardly from the product riser 38 to the electrolyte inlet means 45 with a slope of 1:100. Thus, opposing faces of top member 10 and bottom member 12 are substantially parallel.

Positioned along the exterior surface of top member 10 and electrically connected thereto are a plurality of bus bars 52. Each of these bus bars 52 is electrically interconnected, one to the other, by a plurality of connecting rods 54. This entire bus bar system is also connected to a source of electric power by means of conductor 56.

In operation, the bottom member 12 is maintained at a high positive potential (relative to top member 10) by having conductor 32 and thus the remainder of the bus bar system, including bus bars 28 and connecting rods 30, electrically connected to a power source of high positive potential. Conversely, top member 10 is maintained at a substantially negative in relation to the potential of bottom member 12 by connecting conductor 56 and thus the remainder of the bus bar system, consisting of bus bars 52 and connecting rods 54, to a power source of negative potential.

Liquid amalgam 33 is introduced into amalgam inlet well 16 by means of amalgam inlet pipe 34. As the level of amalgam 33 rises within well 16, the amalgam 33 eventually reaches the level at which it spills over inlet weir 22. As more liquid amalgam 33 is introduced, the level of the amalgam 33 in the cell reaches the point at which it spills over each of the weirs 26 and finally spills over outlet weir 24 into outlet well 18. Inlet weir 22, intermediate weirs 26 and outlet weir 24 are of such height as to maintain the amalgam liquid 33 in a depth of approximately one eighth inch across the amalgam support 20. The direction of amalgam flow is indicated by arrows in FIGURE 2. The amalgam 33 in outlet well 18 is removed from the cell by means of amalgam outlet pipe 36.

An electrolyte 43 consisting of a sodium aluminum tetraethyl complex, tetraethyl lead and NaAl(C2H5)4OR is introduced into the cell through electrolyte inlet means 45. The electrolyte 43 then flows through the cell countercurrent to the amalgam flow, as shown by arrows in FIGURE 2, and is discharged from the cell by arrow 41 shown in FIGURE 2, where it is constantly removed along with the tetraethyl lead product and spent electrolyte 43. The auxiliary product outlet pipe 37 provided in amalgam outlet well 18 also serves as a means for withdrawing some of the denser tetraethyl lead product 41 which may collect in outlet well 18 above the amalgam pool 33.

FIGURE 3 is a schematic diagram illustrating the operation of the process of our invention. The cell illustrated in FIGURES 1 and 2 is designated by reference number 100 in FIGURE 3. The top member 10, bottom member 12 and gasket 14 are represented in this FIGURE 3 also.

The top member 10 is interconnected to electrical line 101 by means of conductor 56 shown in FIGURE 2. The bottom member 12 is interconnected to electrical line 103 by means of conductor 32 shown in FIG-
Amalgam inlet line 102 is connected to the amalgam inlet pipe 34 shown in FIGURES 1 and 2 and amalgam outlet line 104 is connected to the amalgam outlet pipe 36 shown in FIGURES 1 and 2. The amalgam outlet line 104 leads to a surge tank 106. Outlet line 108 connects surge tank 106 to pump 110, which then pumps a solution to the liquid amalgam in surge tank 106 and passes the amalgam by means of line 112 through heat exchanger 114 and thence into line 102 by means of which the liquid amalgam is returned to amalgam inlet pipe 34 of outlet 108.

Another pump 116 removes a relatively small amount of liquid amalgam from line 108 by means of line 118. This small amount of liquid amalgam is discharged through line 120, heat exchanger 122, line 124, furnace 136, line 138 and line 130 into an enclosed stirred mixing vessel 132. The liquid amalgam is at a temperature of about 600°F when introduced into mixing vessel 132. The stirring mechanism of vessel 132 is powered by an electric motor 134.

Solid lead is introduced into a melting furnace 136 by means of chute 137. Liquid lead is withdrawn from melting furnace 136 and pump 140 and is then discharged into stirred mixing vessel 132 by means of lines 142 and 139. Regenerated liquid amalgam is withdrawn from stirred mixing vessel 132 by means of line 144 and pump 146 and is then passed by means of line 148 through heat exchanger 122, pump 150, cooler 152, line 154, and into line 102. As previously explained, line 102 connects with amalgam inlet pipe 34 and cell 100.

Electrolyte inlet line 156 is connected to inlet pipe 50 shown in FIGURES 1 and 2. The section of electrolyte inlet line 156 immediately adjacent to cell 100 and marked "A" is composed of electrical insulating material. Outlet line 158 is connected to discharge pipe 44 shown in FIGURES 1 and 2. The product liquid from cell 100 is withdrawn through outlet line 158 and pump 160 and is then discharged through line 162 into settling tank 164.

Outlet line 171 is connected to auxiliary product outlet pipe 37 shown in FIGURES 1 and 2. The denser project tetraethyl lead product is removed from tank 164 by means of line 165 and pump 167 and is then passed by means of line 169 to a product purification process, not shown. That section of line 165 immediately adjacent to settling tank 164 and marked "A" is composed of electrical insulating material. The constituent of the lowermost liquid layer, other than tetraethyl lead, are returned to the cell 100 from the purification process via an electrolyte regeneration process, also not shown. The middle liquid layer containing the spent electrolyte is withdrawn through line 166 and pump 168 and is then passed by line 170 to cell 100 by means of line 156. That section of line 166 immediately adjacent to settling tank 164 and marked "A" is composed of electrical insulating material.

When employing the sodium aluminum complex mentioned above as the electrolyte, a quantity of make-up NaAl(C₂H₅)₂OR can be added to the electrolyte stream prior to returning the regenerated electrolyte to cell 100 by means of line 156 and/or the NaAl(C₂H₅)₃OR can be regenerated as mentioned above.

The upper liquid layer containing the liquid sodium formed at the cathode is removed from settling tank 164 by means of line 172 and pump 174 and is then passed by line 176 to plastic double block and bleed valve systems. Line 176 is connected to lines 178 and 180, each of which leads to a double block and bleed valve system.

Line 178 is connected to valve system I which comprises two block valves 182 and 184 and one bleed valve 186 interconnected by means of a T- joint 188. The discharge path leads from line 178 through block valve 182, the arms of T-joint 188, block valve 184 and discharge line 190 into tank 192. The bleed path leads through the leg of T-joint 188, bleed valve 186 and bleed line 194. The portions of block and bleed valve system I contained within the dotted lines are composed of electrical insulating material.

Similarly, line 190 leads to block and bleed valve system II which consists of block valves 196 and 198 and bleed valve 200 interconnected by means of a T- joint 202. The discharge path leads from line 190 through block valve 196, the arms of T-joint 202, block valve 198 and discharge line 204 into tank 206. The bleed path leads from the leg of T-joint 202 through bleed valve 200 and into bleed line 208.

Line 210 interconnects tank 212 with block and bleed valve system III consisting of block valves 214, bleed valve 216, T-joint 218, discharge line 220 and bleed line 222. The structure and composition of block and bleed valve system III is similar to that of block and bleed valve system I.

Line 224 interconnects tank 206 and block and bleed valve system IV consisting of block valves 225 and 228, bleed valve 230, T-joint 232, discharge line 234 and bleed line 236. The structure and composition of block and bleed valve system IV is similar to that of block and bleed valve system I.

Bleed lines 194 and 208 connect with line 238 and bleed lines 222 and 236 connect with pipe 240. Line 238 and 240 in turn connect with line 242 which discharges into settling tank 164.

Discharge lines 220 and 234 connect with line 244 which discharges into surge drum 246. The liquid is removed from surge drum 246 by means of line 248 and pump 250 and passed to an electrolyte production process, not shown, by means of line 252.

Due to the fact that the streams leaving cell 100 are all good conductors of electricity it is important to give due consideration to the electrical potential of the various components of equipment. With this in mind the anode itself and all equipment in contact with the circuit and amalgam stream are grounded to earth. Thus, it can be seen that electrical line 103 which is connected to cell bottom 12 is grounded. Surge tank 106, heat exchanger 122, furnace 136, melting furnace 136 and heat exchanger 114 are also grounded.

The top member 10 of the cell 100 is electrically insulated from the bottom member 12, as previously explained, and is insulated from the ground. The electrolyte enters cell 100 through line 156, the portion marked "A" of which is composed of electrical insulating material. The first metallic section of pipe 156 (away from the cell) is grounded to earth. An electrical circuit thus exists from the cathode through the flowing electrolyte to earth and back to the electrical supply, but the resistance of this path is very high in comparison to the resistance path through the cell itself so that the current loss is small.

The settling tank 164 is insulated from earth and stands at cathode potential. Spent electrolyte is withdrawn from settling tank 164 through pipe 166 the portion marked "A" of which is composed of an electrically insulating material. This section of pipe operates in the same manner as pipe 156 through which electrolyte enters the cell 100. As mentioned above, the portions of lines 165 and 171 marked "A" are also composed of electrically insulat-
ing material. These sections of lines 165 and 171 operate in the same manner as lines 156 and 166 to insulate the cell 100 and settling tank 164 from earth. The stream containing the liquid sodium formed at the cathode is an excellent conductor of electricity and since it is in effective contact with the cathode stream, this stream must be physically disrupted before being passed to equipment grounded to earth. To accomplish this end, the stream containing the liquid sodium is discharged alternately through the electrical insulating block and bleed valve systems I and II to one of the tanks 192 or 206. The liquid sodium then flows by gravity alternately from tanks 192 and 206 through electrical insulating double block and bleed valve systems III and IV to surge drum 246 which is grounded to earth.

In operation, the double block and bleed valve systems make it possible to isolate electrically the tank (either 192 or 206) from which the liquid sodium is flowing from the cathode potential and to isolate the tank (either 192 or 206) to which the liquid sodium is flowing from the earth. This isolation is accomplished by closing both block valves and opening the bleed valve in one system, thus draining the tank, and opening the line between the block and bleed valves and interrupting the line path. For example, when pump 174 is delivering the liquid sodium to tank 192 by means of lines 176, 178 and 190, the block valves 182 and 184 in system I are open and the bleed valve 186 is closed; in system III block valves 212 and 214 are closed and bleed valve 216 is open; in system II the block valves 196 and 198 are closed and the bleed valve 200 is open; in system IV the block valves 226 and 228 are open and the bleed valve 230 is closed. Thus tank 192 is being filled by the stream of liquid sodium at the cathode potential and at the same time such liquid stream is separated from the grounded surge drum 246 by the operation of block and bleed valve system III. At the same time tank 206, which has been previously filled with liquid sodium, is now separated from the stream of liquid sodium at cathode potential by the operation of block and bleed valve system II. The liquid sodium in tank 206 thus separated from the cathode potential, is drained through block and bleed valve system IV and into surge drum 246. When tank 192 has been filled and tank 206 has been drained in the manner explained above, the flow of liquid sodium streams is alternately reversed by reversing the positions of the valves in the four block and bleed valve systems, i.e., in system I block valves 182 and 184 are closed and bleed valve 186 is opened; in system III block valves 212 and 214 are opened and bleed valve 216 is closed; in system II block valves 196 and 198 are opened and bleed valve 200 is closed; in system IV block valves 226 and 228 are closed and bleed valve 230 is open.

When the block and bleed valves are in the positions first described above, the liquid sodium contained in the T-joint 218 of system III is drained through bleed valve 216, line 222 and line 240. The liquid sodium contained in T-joint 202 of system II is drained through bleed valve 206, line 208 and line 238. When the block and bleed valves are in the positions second described above, the liquid sodium in T-joint 122 of system I is drained through bleed valve 156, line 194 and line 238. The liquid sodium in T-joint 232 of system IV is drained through bleed valve 330, line 235 and line 240. Lines 238 and 240 both connect to line 242 by means of which the liquid sodium is returned to settling tank 164.

As a practical matter tanks 192 and 206 are sized for a long cycle (8 hrs) so as to minimize the valve switching problems.

Example

The following process is carried out in accordance with the method of our invention employing the cell shown in FIGURES 1 and 2 of the drawing.

In operation, 1.2 cubic feet per hour of lead amalgam containing 8% by weight lead at a temperature of 212°F. is introduced into cell by means of amalgam inlet pipe 34. The lead amalgam is maintained at an average depth of 3/4 inch as it flows across amalgam support 20. Spent amalgam at a rate of 1.15 cubic feet per hour and a temperature of 212°F. is removed from the cell by means of amalgam outlet pipe 36. The electrolyte employed has the following composition:

<table>
<thead>
<tr>
<th>Component</th>
<th>Wt. percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tetraethyl lead</td>
<td>7.8</td>
</tr>
<tr>
<td>Sodium aluminum tetraethyl (NaAl(C₂H₅)₄)</td>
<td>85.7</td>
</tr>
<tr>
<td>Sodium butoxy-diethyl aluminum I (Al(C₂H₅)₃OC₂H₅)</td>
<td>4.5</td>
</tr>
<tr>
<td>Butoxy-diethyl aluminum I (Al(C₂H₅)₃OC₂H₅)</td>
<td>2.0</td>
</tr>
</tbody>
</table>

* From recycle electrolyte.

This electrolyte is introduced into the cell at a temperature of 212°F. by means of electrolyte inlet means 45 at the rate of 121.7 cubic feet per hour. Liquid at a temperature of 212°F. is removed at the rate of 121.4 cubic feet per hour from the cell by means of product outlet means 39. After separation by settling, this liquid forms a sodium layer, a spent electrolyte layer and a tetrathyli lead layer. The constituents of these layers are removed at the following rates:

<table>
<thead>
<tr>
<th>Component</th>
<th>Lb./hr.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium layer: Sodium</td>
<td>15.0</td>
</tr>
<tr>
<td>Spent electrolyte layer:</td>
<td></td>
</tr>
<tr>
<td>NaAl(C₂H₅)₄</td>
<td>6228.2</td>
</tr>
<tr>
<td>NaAl(C₂H₅)₃OC₂H₅</td>
<td>164.0</td>
</tr>
<tr>
<td>Tetrathyli lead</td>
<td>568.2</td>
</tr>
<tr>
<td>Al(C₂H₅)₃OC₂H₅</td>
<td>142.0</td>
</tr>
<tr>
<td>Tetrathyli lead layer:</td>
<td></td>
</tr>
<tr>
<td>Tetraethyl lead</td>
<td>63.2</td>
</tr>
<tr>
<td>Al(C₂H₅)₃OC₂H₅</td>
<td>123.2</td>
</tr>
<tr>
<td>NaAl(C₂H₅)₄</td>
<td>3.8</td>
</tr>
</tbody>
</table>

Both the top member 10 and the bottom member 12 of the cell have a slope of 1:100 and are thus maintained substantially parallel. The inner surface of top member 10 is maintained at a distance of 0.75 inch from the upper surface of amalgam support 20. The bottom member 12 is maintained at a positive potential (relative to top member 10) of 3 volts.

We claim:

1. A continuous process for the production of tetrathyli lead by electrolysis which includes introducing into and flowing through an electrolytic cell a liquid phase electrolyte consisting essentially of a tetrathyli complex of aluminum and an alkali metal having an atomic number of 11 to 19, tetrathyli lead and a scavenger compound of the formula MAI(C₂H₅)₃O wherein M is selected from the group consisting of sodium and potassium and R is an alkyl group having from 2 to 8 carbon atoms, continuously flowing liquid lead amalgam through said cell as the anode of said cell, electrolyzing said electrolyte whereby said alkali metal is deposited as a liquid phase at the cathode which is positioned above the anode and tetrathyli lead is produced at the anode where the lead of said amalgam is in the liquid phase, said tetrathyli lead being initially present in the electrolyte in a concentration sufficient to maintain the density of said electrolyte greater than that of said alkali metal, removing the spent amalgam from the cell, removing spent electrolyte, said alkali metal deposited in liquid phase at the cathode and tetrathyli lead produced at the anode, separating tetrathyli lead from the spent electrolyte and said alkali metal in liquid phase, adding fresh electrolyte to the cell and recovering tetrathyli lead as product. 2. The process of claim 1 in which said alkali metal is sodium.
3. The process of claim 1 in which the spent amalgam is regenerated and recycled to the cell and in which the spent electrolyte is regenerated and recycled to the cell.

4. The process of claim 1 in which M is sodium and R is a butyl radical.

5. The process of claim 1 in which tetraethyl lead comprises from about 5 percent to about 15 percent by weight of the total electrolyte and in which said scavenger compound comprises from about 2 percent to about 10 percent by weight of the total electrolyte.

6. The process of claim 1 in which said amalgam is composed from about 5 percent to about 20 percent by weight of lead and in which said amalgam and said electrolyte are maintained at a temperature within the range from about 100° C. to about 120° C.

References Cited in the file of this patent

UNITED STATES PATENTS

2,704,743 Deprez ---------------- Mar. 22, 1955
2,734,855 Buck et al. ---------------- Feb. 14, 1956
2,741,587 Sindeband ---------------- Apr. 10, 1956
2,848,406 Szechman ---------------- Aug. 19, 1958
2,944,948 Giraltia ---------------- July 12, 1960
2,985,568 Zeigler et al. -------------- May 23, 1961

FOREIGN PATENTS

848,364 Great Britain --------------- Sept. 14, 1960
614,688 Canada ------------------- Feb. 14, 1961

OTHER REFERENCES