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G. H. F. CALINGAERT

METHOD FOR PRODUCING LEAD COMPOUNDS

Filed July 2, 1923

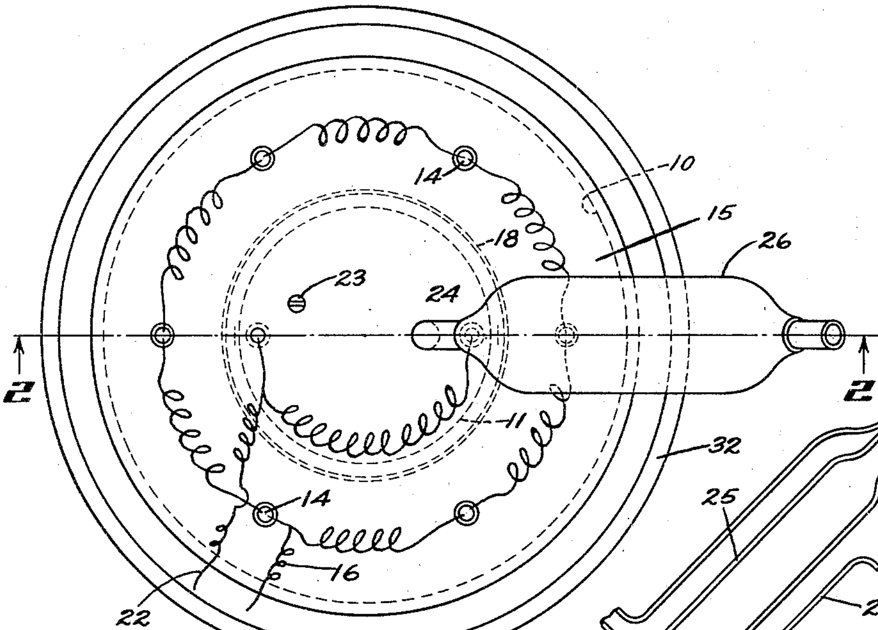

Fig. 1

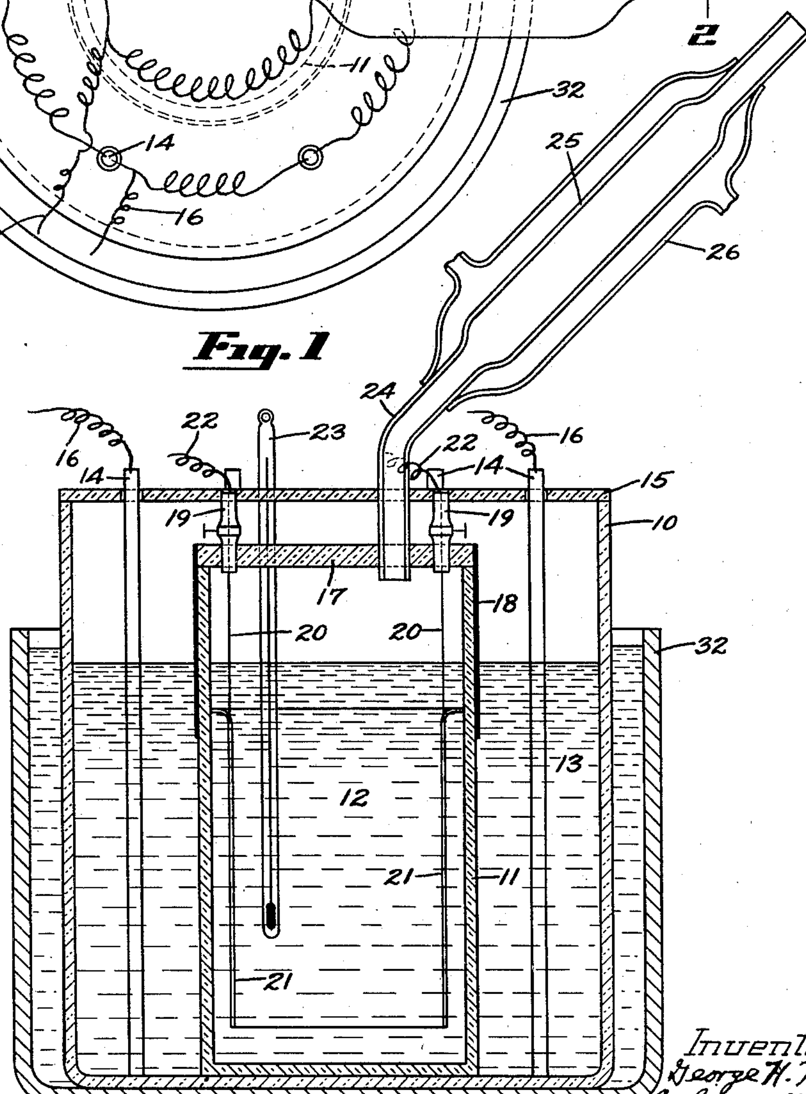

Fig. 2

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UNITED STATES PATENT OFFICE.

GEORGE H. F. CALINGAERT, OF WATERTOWN, MASSACHUSETTS, ASSIGNOR, BY MESNE ASSIGNMENTS, TO GENERAL MOTORS CORPORATION, OF DETROIT, MICHIGAN, A CORPORATION OF DELAWARE.

METHOD FOR PRODUCING LEAD COMPOUNDS.

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To all whom it may concern:

Be it known that I, George H. F. Calingaert, a subject of the King of the Belgians, residing at Watertown, in the county of Middlesex, Massachusetts, have invented certain new and useful Improvements in Methods for Producing Lead Compounds, of which the following is a full, clear, and exact description.

This invention relates to modes of producing lead tetra alkyls, and, more particularly, lead tetraethyl, and its principal objects are to promote an intimate relation between the substances employed and to electrically reduce the reaction mass.

In the accompanying drawings:

Fig. 1 is a plan view of an apparatus adapted to carry out my process; and

Fig. 2 is a sectional view thereof, taken substantially on the line 2—2 of Fig. 1.

In these drawings 10 is a tank containing a porous cup 11 made of clay or other material, which divides the interior of the tank into an inner chamber 12 within the cup and an outer chamber 13 between the cup and the tank walls. A series of graphite anodes 14, (shown herein as six in number) are held in vertical positions in the outer chamber 13 by a tank cover 15 through which the anodes project. The outer ends of the anodes are connected in an electrical circuit by a wire 16.

The porous cup 11 has a cover 17 of nonconducting material, for example, hard rubber, sealed along its periphery by asphalt 18. Metal ferrules 19, with rubber attachments, seated in the cover 17 support leads 20 which are attached at their inner ends to an annular lead cathode 21 and at their outer ends to a wire 22 connected in the electrical circuit with wire 16. The cathode may be formed of spongy lead to promote a reaction between the lead and the catholyte. 23 is a thermometer, and 24 a reflux condenser having a condensing chamber 25 communicating with the inside of cup 11, and a cooling chamber 26 having connections for circulating a cooling medium about the condensing chamber 25.

The cell may be placed in a cooling bath 32 for maintaining the temperature of the catholyte below the boiling point of the alkyl halide.

The chamber 12 within the cup is partially filled with a catholyte comprising, by weight, 90 parts of commercial ethyl alcohol, 10 parts of NaOH and 10 parts of ethyl iodide, and the chamber 13 around the cup is partially filled with an anolyte comprising 10 parts of NaOH and 90 parts of alcohol. If desired, a lead compound, such as lead acetate, may be dissolved in the catholyte to promote a reaction with the lead, preferably by the formation of spongy lead at the cathode. 2 parts of lead acetate may be used in the catholyte given above.

The current is turned on, a current density of about 1.15 amperes per dm^2 being preferred. The current heats the reaction mass, and forms hydrogen. The temperature of the reaction mass given above is preferably maintained at about 55° C., which temperature is below the boiling point of ethyl iodide, but high enough to maintain a relatively high rate of reaction in the cell. The reflux apparatus condenses and returns to the cup the ethyl iodide which vaporizes during the reaction. Apparently the hydrogen formed at the cathode reduces the reaction mass, forming lead di-ethyl, which is unstable at the temperature of the catholyte, and breaks up thermally into lead and lead tetraethyl. The lead tends to settle to the bottom of the cup.

When the reaction is completed, the alcohol and ethyl iodide are distilled over by heating the catholyte to a temperature below about 98° C., the heavier lead tetraethyl is then steamed distilled out, preferably by passing steam into the lead tetraethyl, and collected in another vessel, and the caustic which remains in the cell may be recovered.

The composition of the reaction mass may be varied by using in place of ethyl iodide, other alkyl halides, for example, methyl iodide, amyl iodide, and methyl, amyl or ethyl bromide, and by employing other substances than ethyl alcohol as a solvent for the alkyl halide and the NaOH or its equivalents, such as KOH.

Slight alkalinity of the electrolyte promotes the reaction and avoids acidity which is detrimental to the reaction.

I claim:

1. The process of producing a lead alkyl, which comprises forming a solution of an alkyl halide and an alkali, and reducing the reaction mass thus formed in contact with lead.

2. A process as set forth in claim 1 in which the temperature of the reaction mass is maintained slightly below the boiling point of the alkyl halide.

3. A process as set forth in claim 1 in which the alkyl halide is an ethyl halide and lead tetraethyl is formed.

4. A process as set forth in claim 1 in which the alkyl halide is an alkyl iodide.

5. A process as set forth in claim 1 in which the alkyl halide is ethyl iodide and lead tetraethyl is formed.

6. A process as set forth in claim 1, in which the alkyl halide and alkali are dissolved in ethyl alcohol.

7. The process of producing a lead alkyl, which comprises dissolving an alkyl halide in an alcoholic electrolyte, placing a lead cathode in the electrolyte, and passing an electric current through the cathode and the electrolyte.

8. A process as set forth in claim 7, in which the solution is alkaline.

9. A process as set forth in claim 7, in which an alkali and a lead compound are added to the solution.

10. A process as set forth in claim 7, in which NaOH and lead acetate are added to the solution.

11. The process of producing a lead alkyl, which comprises forming a catholyte comprising NaOH and an alkyl halide dissolved in ethyl alcohol, placing a lead cathode in the catholyte, and passing an electric current through the cathode and catholyte.

12. A process as set forth in claim 11, in which the temperature of the catholyte is maintained below the boiling point of the alkyl halide.

13. A process as set forth in claim 11, in which the alkyl halide is an ethyl halide and lead tetraethyl is formed.

14. A process as set forth in claim 11, in which the alkyl halide is an alkyl iodide.

15. A process as set forth in claim 11, in which the alkyl halide is ethyl iodide and lead tetraethyl is formed.

16. A process as set forth in claim 11, in which lead acetate is added to the catholyte.

17. The process of producing lead tetraethyl, which comprises forming a catholyte of NaOH, an ethyl halide and ethyl alcohol; and passing an electric current through a lead cathode in contact with the catholyte.

18. A process as set forth in claim 17, in which lead acetate is added to the catholyte.

In testimony whereof I hereto affix my signature.

GEORGE H. F. CALINGAERT.