B. MEAD

METHOD FOR PRODUCING LEAD COMPOUNDS

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Fig. 1

Fig. 2

Inventor
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By           His Attorney
To all whom it may concern:

Be it known that I, BRIAN MEAD, a subject of the King of Great Britain, residing at 61 Mountfort Street, Boston, 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 non-conducting material, for example, hard rubber, sealed along its periphery by asphalt 18. Metal ferrules 19 with rubber attachment 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. 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. Mounted on the cover 17 is a mercury seal 27 for an agitator comprising a rod 28 projecting through the seal, gears 29 connecting the outer end of the rod with a power shaft 30 and propeller blades 31 on the inner end of the rod.

The cell is placed on a hot plate or similar means for controlling the temperature of the electrolyte.

The chamber 12 within the cup is partially filled with a catholyte comprising by weight 90 parts of water, 10 parts of NaOH, 10 parts of ethyl iodide, and 1 part casein, and the chamber 13 around the cup is partially filled with an anolyte comprising 10 parts NaOH in 90 parts of water. The electrolyte is heated to about 55° C, or just below the boiling point of the ethyl iodide, the agitator is started, and the current is turned on, a current density of about 1.15 ampere per dm² being preferred. The reflux apparatus condenses and returns to the cup the ethyl iodide which vaporizes during the reaction. The stirring produces an emulsion of casein and ethyl iodide in the water and this emulsion promotes homogeneity of the mixture and an intimate contact between the ingredients of the cell and, more particularly, between the ethyl iodide and the lead. Apparently the hydrogen formed at the cathode reduces the reaction mass, forming lead diethyl which is unstable at the temperature used 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 NaOH is decanted off and the heavier lead tetraethyl is steam distilled out and collected in another vessel.

The composition of the reaction mass may be varied by using in place of the ethyl iodide, other alkyl halides, for example, methyl iodide, amyl iodide, and methyl, amyl, and ethyl bromides.

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 an emulsion of an alkyl halide in water, placing lead in contact with the emulsion, and reducing the reaction mass thus formed.

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. The process of producing a lead alkyl which comprises forming an electrolyte comprising an emulsion of an alkyl halide, and reducing the electrolyte by an electric current through lead in contact with the electrolyte.

7. A process as set forth in claim 6 in which the emulsion is formed in an alkaline solution.

8. A process as set forth in claim 6 in which the emulsion is formed with casein.

9. The process of producing a lead alkyl which comprises forming an electrolyte comprising an emulsion of an alkyl halide in an alkaline water solution, and reducing the electrolyte by an electric current through a lead cathode.

10. A process as set forth in claim 9 in which the temperature of the reaction mass is maintained slightly below the boiling point of the alkyl halide.

11. A process as set forth in claim 9 in which the alkyl halide is an ethyl halide and lead tetraethyl is formed.

12. A process as set forth in claim 9 in which the alkyl halide is an alkyl iodide.

13. A process as set forth in claim 9 in which the alkyl halide is ethyl iodide and lead tetraethyl is formed.

14. A process as set forth in claim 9 in which an emulsion of an alkyl halide and casein is formed in an alkaline water solution.

In testimony whereof I hereto affix my signature.

BRIAN MEAD.