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METHOD OF PRODUCING ALLOYS OF LEAD AND ALKALI METALS

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 1 Claim. (Cl. 75-167)

The invention relates to a new method of producing alloys of lead and alkali metals, particularly the alloys used in the production of organic lead compounds, especially tetraethyl lead.

Monosodium lead alloys corresponding to the formula $PbNa$ and containing substantially 90% of lead and 10% of sodium by weight, are at the present time prepared by mixing the two metals in the fused state. The sodium used in these alloys is almost always obtained by electrolysis of a sodium salt in the dry-way.

Various methods have been proposed for separating the alkali metals from the dilute amalgams obtained by electrolysis of aqueous solutions of alkali metal salts in mercury cathode cells.

However, these processes all present difficulties due to the great affinity of the alkali metals for mercury, which renders separation difficult.

The present invention has as object a process permitting the obtaining of alloys of lead and alkali metals from alkali metal amalgams and in particular from electrolytic amalgam, without separation of the alkali metal in the isolated state.

It is based on the property possessed by lead of modifying, in a manner favourable to distillation, the partial pressures of mercury and of alkali metal of binary mercury-alkali metal alloys.

According to the invention, the process of preparation of alloys of lead and an alkali metal from electrolytic amalgams obtained in mercury cathode cells consists in dissolving lead in the amalgam and eliminating the mercury of the ternary alloy by distillation.

The invention is applicable to the production of alloys of lead with one or more alkali metals, but in order to facilitate the understanding of the process, the invention will be described hereinbelow in its application to the production of lead-sodium alloys.

The dissolution of the lead in the amalgam is advantageously effected in the heat. When the dissolution is completed, the ternary amalgam is distilled in order to eliminate the mercury, which is condensed, collected, and returned to the electrolytic cells. A lead-sodium alloy containing only traces of mercury is obtained as distillation residue.

The mercury distilled off and recycled to the electrolytic cells should contain only small amounts of sodium. For this reason the atomic ratio Na/Pb in the alloy preferably lies within the range 0.1 to 3. The alloy which is particularly suitable for the production of tetraethyl lead may be prepared directly at the desired concentration. It is also possible to prepare an alloy having a greater or smaller concentration of sodium, the composition of which can subsequently be modified by dissolving a further quantity of lead or of metallic sodium therein.

The lead need not necessarily be introduced into the dilute electrolytic amalgam. It is sometimes advantageous to eliminate a part of the mercury from the dilute amalgam in order to increase the sodium content of the amalgam, and then to add the lead. The lead may be added in one or more times, the advantages resulting from the introduction of the lead being the greater, the higher the sodium content of the amalgam subjected to distillation.

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Continuous distillation of the mercury contained in the ternary mercury-lead-sodium amalgam can be effected in a flow distillation apparatus in which the amalgam flows over a metal wall, through which is effected the transfer of the heat required for evaporation of the mercury. This apparatus has the advantage of permitting distillation of a thin layer, thus avoiding bumping due to distillation.

The distillation may also be carried out continuously in one or more stages in stills which may or may not be surmounted by extraction columns.

Distillation in several stages offers the advantage that a large part of the mercury is evaporated at a pressure close to atmospheric pressures. It also permits a higher degree of extraction of mercury from the final lead-sodium alloy.

The advantages of the invention are illustrated by the examples given below, which are in no way limitative.

Example 1

In dilute amalgam, containing 0.5% of sodium by weight and issuing from mercury cathode electrolytic cells, lead is dissolved so as to form a ternary alloy in which the ratio of sodium and lead concentration is equal to 0.5 at. $Na/at. Pb$.

This amalgam is introduced continuously into a still operating at a temperature of $550^{\circ} C.$ at an absolute pressure of 100 mm./Hg. The vapours emitted are condensed by cooling and the condensate, composed of mercury containing only 1.0 at. percent of sodium, is recycled to the electrolytic cells.

A binary lead-sodium alloy corresponding approximately to the formula Pb_2Na and containing only 2 at. percent of mercury is continuously collected from the still.

Example 2

To a dilute amalgam containing 0.5% by weight of sodium issuing from electrolytic cells, lead is added so as to form a ternary amalgam of a composition such that the ratio of the sodium and lead concentrations will be equal to 1 at. $Na/at. Pb$.

This ternary alloy is introduced into the top of a trickle distillation apparatus and uniformly distributed over the internal face of the externally heated apparatus. This apparatus works at the normal pressure of 760 mm./Hg, while the temperature varies between 370 and $550^{\circ} C.$ from the top to the bottom.

The vapours emitted are condensed and the condensate composed of practically pure mercury is recycled to the electrolytic cells.

The liquid ternary alloy leaving this distillation apparatus has the following composition:

	At. percent
Sodium -----	41
Lead -----	41
Mercury -----	18

This alloy is continuously introduced into the boiler of a still functioning at $550^{\circ} C.$ at an absolute pressure of 10 mm./Hg. The dilute amalgam obtained by condensation of the vapours leaving said still has a sodium concentration of 5 at. percent. This amalgam is mixed with amalgam leaving the electrolytic cells for treatment in the trickle still after adding lead. The liquid alloy collected from the bottom of the boiler-still corresponds to the formula $PbNa$ and its mercury content is only 2 at. percent.

It is obvious that numerous modifications exist in the carrying out of the process forming the subject of the invention. In particular, trickle stills may be replaced by boiler stills, and vice versa. Moreover, in multi-stage distillation processes an almost pure mercury fraction

obtained by condensation of the vapours leaving the first stage may be utilised to condense vapours produced in a subsequent stage. This operation enables the sodium concentration of the vapours leaving the last stage to be reduced and thus avoids fractional condensations and the obstructions which may result from the formation of solid amalgams.

I claim:

A method of preparing directly an alloy of lead and of an alkali metal from an amalgam previously produced by electrolysis in a mercury cathode cell without prior recovery of the alkali metal from said amalgam, which comprises dissolving lead in said previously-produced amalgam to form a ternary amalgam in which the atomic ratio of sodium to lead concentration is equal to 1.0, introducing said ternary amalgam into a trickle distillation zone operating at atmospheric pressure and at a temperature varying from 370-550° C. from top to bottom, condensing the vapors emitted from said distillation zone,

recycling the condensate to said electrolytic cell, introducing the residue from said distillation zone into a second distillation zone operating at a temperature of 550° C. and a pressure of 10 mm. of mercury, condensing the vapors leaving said second zone, mixing said condensate with said amalgam obtained from said electrolytic cell and collecting said alloy from the bottom of said second zone.

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