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TETRA ALKYL LEAD

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This invention relates to the manufacture of tetra alkyl lead compounds and more particularly to the recovery of such compounds from a mixture of reaction products.

The process in most general use, at the present time, for the manufacture of tetra alkyl lead compounds comprises reacting an alkyl halide with a lead sodium alloy, cooling the resulting lead sludge, then drowning it in water and then distilling with steam to remove the tetra alkyl lead compound. During this distillation, the particles of lead in the sludge tend to cohere and form balls and sheets which render effective agitation impractical or impossible and which cannot be flushed from the still but require manual removal. Furthermore, the lead sludge remaining in the still retains substantial proportions of the tetra alkyl lead which it has, heretofore, been considered to be impossible to recover by means of continued distillation.

In order to prevent this agglomeration of the metallic lead and thus overcome the many problems presented thereby, it has been proposed to add to the lead sludge, before distillation, compounds which have a dispersing action on the metallic lead and prevent the agglomeration thereof. Some of these compounds are glue, agar-agar, gums, engine oil, lard oil, toluene, and the like; and also certain inorganic salts such as sodium sulfide, sodium carbonate, sodium sulfate, and the corresponding potassium salts.

While these compounds greatly improve the condition of the sludge and render it more fit for proper working in the still, they have not been proved to be entirely satisfactory as they do not entirely prevent agglomeration of the lead. Furthermore, even with the use of these compounds, it has been impossible to recover all of the tetra alkyl lead, as much as 6% being retained in the sludge at the completion of the distillation.

An object of the present invention is to improve the process of producing tetra alkyl lead compounds and particularly the recovery of the tetra alkyl lead compounds from the lead sludge. A further object is to place the lead sludge in better condition for removal of the tetra alkyl lead compounds therefrom. A still further object is to provide a process for the more complete removal of tetra alkyl lead compounds from the lead sludge. Other objects will appear hereinafter.

These objects are accomplished by the following invention which comprises treating the alkyl lead sludge with a compound which efficiently and completely prevents the agglomeration of the lead in the sludge, distilling off the major portion of the tetra alkyl lead, then adding a wetting agent to the residue in the still, and then continuing the distillation until substantially all of the tetra alkyl lead is recovered.

We have found that when engine oil and thiocarbanilide are both added to the diluted reaction mass and the resulting mass subjected to steam distillation, the metallic lead forms coarsely granular particles which do not agglomerate. These lead particles are of such a size that they can be readily washed in a stream of water to remove sodium chloride and like impurities without washing away any of the particles. The wash water will readily drain from the resulting mass of lead particles and such mass may be readily dried. Neither engine oil nor thiocarbanilide nor any of the other dispersing agents heretofore employed, when used alone, can produce such results.

Certain other substances such as sodium chromate or dichromate, sodium thiosulfate, sodium silicates and their corresponding water soluble salts of other cations, may be employed in place of the engine oil and thiocarbanilide but without obtaining all of the advantageous features thereof.

The above dispersing agents have the disadvantage, common to all the prior dispersing agents, of failing to permit complete removal of the tetra alkyl lead compounds from the sludge, as the metallic lead particles retain substantial proportions of the tetra alkyl lead compound, up to 6% of the tetra alkyl lead formed in the reaction. However, we have found that, by subjecting a lead sludge so produced to a distillation with steam until the major portion of the tetra alkyl lead has been removed or until no further tetra alkyl lead appears in the distillate, then adding a wetting agent to the sludge remaining in the still and then continuing the distillation, substantially all of the tetra alkyl lead may be recovered.

The wetting agents, which we propose to employ, include those substances which lower the surface tension of water, thereby increasing its tendency to wet metallic lead. The wetting agents, which we have found to be particularly desirable, are sulfonated castor oils known as Turkey red oil and monopole oil and other sulfonated natural oils. Other wetting agents, which may be employed but which are less desirable because of certain problems attendant upon the use thereof, include the salts of alkyl naphthalene sulfonic acids; the condensation products

of naphthol sulfonic acids and formaldehyde; the reaction products of oleic acid and the aliphatic diamines; the higher fatty and unsaturated acids and their soaps including both the inorganic cations, such as sodium and potassium stearates, and the organic soaps, such as triethanolamine stearate and oleate; the resinates; and certain commercial products.

In order to more clearly disclose our invention and to illustrate the preferred method of carrying the same into effect, the following examples are given:

Example 1.—Ethyl chloride is caused to react with a lead sodium alloy in an autoclave. After the reaction is complete, any pressure in the autoclave is released and the sludge is drowned in water to which has been added thiocarbanilide equal in weight to from 0.01 to 5% of the total weight of the lead in the charge and engine oil equal in weight to from 0.01 to 5% of the total weight of the lead in the charge. The tetra ethyl lead is then removed by steam distillation from the agitated mass.

Example 2.—The process, as disclosed in Example 1, is employed and the distillation is continued for some time, e. g. 90 minutes, at which time the distillation appears to be substantially complete as substantially no further tetra ethyl lead passes off into the distillate. At the end of this time, from 0.01 to 5% of Turkey red oil is added to the still and the distillation with steam continued. Within a short time, substantially all of the tetra ethyl lead is removed from the lead sludge.

In the above description, we have specifically disclosed the use of from about 0.01 to 5% of each of the agents. However, we have found that from about 0.1 to 1% thereof is preferred, under ordinary conditions. By the use of the wetting agent as described in Example 2, the recovery of the tetra ethyl lead is increased from about 1 to 6%.

While we have disclosed certain specific processes employing certain agents in specific proportions, it will be apparent to those skilled in the art that many changes and variations may be made therein without departing from the spirit of our invention. Accordingly, the scope of our invention is to be limited solely by the appended claims construed as broadly as is permissible in view of the prior art.

We claim:

1. The method of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding engine oil and thiocarbanilide to the lead sludge and then distilling off the tetra alkyl lead.

2. The method of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding to the lead sludge from about 0.01 to 5% of engine oil and about 0.01 to 5% of thiocarbanilide and then distilling off the tetra alkyl lead.

3. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding a dispersing agent to the lead sludge, distilling off the major portion of the tetra alkyl lead, then adding a wetting agent to the undistilled sludge, and then continuing the distillation.

4. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding a dispersing agent to the lead sludge, distilling off tetra alkyl lead until substantially no tetra alkyl lead appears in the distillate, then adding a wetting agent to the undistilled lead sludge, and then continuing the distillation.

5. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding a dispersing agent to the lead sludge, distilling off a major portion of the tetra alkyl lead, then adding a sulfonated natural oil to the undistilled sludge, and then continuing the distillation.

6. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding a dispersing agent to the lead sludge, distilling off the major portion of the tetra alkyl lead, then adding a sulfonated castor oil to the undistilled sludge, and then continuing the distillation.

7. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding a dispersing agent to the lead sludge, distilling off a major portion of the tetra alkyl lead, then adding a member of the group consisting of Turkey red oil and monopole oil to the undistilled sludge and then continuing the distillation.

8. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding to the lead sludge engine oil and thiocarbanilide, distilling off the major portion of the tetra alkyl lead, then adding a wetting agent to the undistilled sludge, and then continuing the distillation.

9. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding to the lead sludge engine oil and thiocarbanilide, distilling off the major portion of the tetra alkyl lead, then adding a sulfonated natural oil to the undistilled sludge, and then continuing the distillation.

10. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding to the lead sludge engine oil and thiocarbanilide, distilling off the major portion of the tetra alkyl lead, then adding a sulfonated castor oil to the undistilled sludge and then continuing the distillation.

11. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding engine oil and thiocarbanilide to the lead sludge, distilling off the major portion of the tetra alkyl lead, then adding a member of the group consisting of Turkey red oil and monopole oil to the undistilled sludge, and then continuing the distillation.

12. The method of recovering tetra ethyl lead from a lead sludge containing the same which comprises adding engine oil and thiocarbanilide to the lead sludge, distilling off the tetra ethyl lead until substantially no further tetra ethyl lead passes off, then adding to the undistilled sludge from about 0.01 to 5% of Turkey red oil based on the lead in the sludge, and then continuing the distillation to remove further amounts of tetra ethyl lead.

13. The method of recovering tetra ethyl lead from a lead sludge containing the same which comprises adding to the lead sludge from 0.01 to 5% of engine oil and from 0.01 to 5% of thiocarbanilide based on the weight of the lead in the sludge, distilling off the tetra ethyl lead with steam until substantially no further tetra ethyl lead passes off, then adding to the undistilled sludge from 0.01 to 5% of Turkey red oil based on the lead in the sludge, and then continuing the distillation to remove all of the tetra ethyl lead.

14. The method of manufacturing tetra alkyl

lead which comprises reacting an alkyl halide with a lead-sodium alloy, drowning the reaction products in water containing engine oil and thiocarbanilide, distilling off the major portion of the tetra alkyl lead with steam, then adding a wetting agent to the residue, and then distilling off the rest of the tetra alkyl lead with steam.

15. The method of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding from about 0.01 to about 5% of sodium thiosulphate to the lead sludge and then distilling off the tetra alkyl lead.

16. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding sodium thiosulfate to the lead sludge, distilling off the major portion of the tetra alkyl lead, then adding a wetting agent to the undistilled sludge, and then continuing the distillation.

17. The process of recovering tetra alkyl lead from a lead sludge containing the same which comprises adding sodium thiosulfate to the lead sludge, distilling off the major portion of the tetra alkyl lead, then adding a sulfonated natural oil to the undistilled sludge, and then continuing the distillation.

18. The process of recovering tetra ethyl lead from a lead sludge containing the same which comprises adding sodium thiosulfate to the lead sludge, distilling off the major portion of the tetra ethyl lead, then adding a wetting agent to the undistilled sludge, and then continuing the distillation.

19. The process of recovering tetra ethyl lead from a lead sludge containing the same which comprises adding sodium thiosulfate to the lead sludge, distilling off the major portion of the tetra ethyl lead, then adding a sulfonated natural oil to the undistilled sludge, and then continuing the distillation.

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