

UNITED STATES PATENT OFFICE

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PROCESS OF PRODUCING TETRA ALKYL LEAD

No Drawing.

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This invention relates to the manufacture of tetra alkyl lead and more particularly to an improvement in the process of producing tetra ethyl lead.

dropped into a still which is previously heated and maintained at a temperature approximately 200 to 300° C. Operating the process in this way results in a mixture of tetra alkyl lead and water flash-distilling out of the still. 55

Several methods have been known for the recovery of tetra ethyl lead from the metallic lead sludges always present after its formation by the reaction of ethyl halides with the sodium alloys of lead. These methods have consisted principally of low temperature, high vacuum distillation, steam distillation, and extraction with organic solvents. The latter, however, is objectionable because the removal of the solvents is necessary, and this is difficult and expensive. Also, vacuum distillation presents many difficulties and dangers. When steam distillation is employed it has been the practice to use low-pressure saturated steam, the temperature of which is approximately not above 110-115° C. The literature on this subject discloses that tetra ethyl lead will decompose at temperatures not much above 125° C.

The following examples illustrate preferred embodiments of our invention, but it is to be understood that these are not to be taken as limitations thereof. 60

Example I.—A mixture of tetra ethyl lead and lead sludge such as is obtained by the interaction between lead mono-sodium alloy and ethyl chloride is mixed with approximately 10 times its weight of water. This mixture is then brought to a boil by the application of external heat by the use of a steam jacket for large scale operation or on a small scale, by direct gas flame. When the mixture is boiling the external heat is removed and steam, superheated to 200-300° C. is introduced through orifices in the bottom of the still. Distillation by this procedure is generally complete in two to three hours. The tetra ethyl lead is recovered from the water fraction of the distillate in the usual manner and the yield obtained will be approximately between three per cent and four per cent higher than it would have been had ordinary saturated steam been employed. 80

An object of our invention is to overcome the difficulties heretofore experienced in the recovery of tetra ethyl lead from the metallic sludges inherently present in such processes. A further object of our invention is to improve generally on the process of manufacturing tetra alkyl lead compounds. A particular object of our invention is to increase the yield of tetra alkyl lead. A further object of our invention is an improvement in the distillation of tetra alkyl lead from the sludges present. Other objects will appear hereinafter.

While we prefer the temperatures specified, these may be lower than 200° C. and even as high as 400° C.

These objects are accomplished by the following invention in which we have found that tetra alkyl lead and particularly tetra ethyl lead, can be distilled from the sludges formed in the process of manufacture, with an increased completeness of removal as is indicated by increases in yield, provided that high temperature steam, and particularly steam superheated to temperatures between 200° C. and 300° C., is employed in the step of distillation, instead of the ordinary saturated steam, and that tetra alkyl lead may also be recovered without decomposition if a mixture of the sludge and water is slowly 50

Example II.—An iron still is provided with an oil bath or other baths capable of being raised to 300° C. The temperature of the bath is elevated until the internal temperature of the still reaches approximately 275° C. There is then run into the still at such a rate that the temperature does not fall below 200° C. a mixture of one part of lead sludge containing tetra ethyl lead and ten parts of water. The tetra ethyl lead and water immediately flash off the lead sludge and are recovered in the usual fashion. The tetra ethyl lead is separated from the water fraction in the normal manner and the yield will be found to be from three per cent to four per cent higher than could have been obtained. 100

tained by the distillation of a like sample with ordinary saturated steam.

Similar improved results may be obtained by flash-distilling in like manner other tetra alkyl lead compounds such as tetra methyl lead and tetra butyl lead, etc.

As many apparently widely different embodiments of this invention may be made without departing from the spirit thereof, it is to be understood that we do not limit ourselves to the foregoing examples or descriptions except as indicated in the following patent claims.

We claim:

1. In the process of producing tetra alkyl lead by the interaction of alkyl halides and a lead sodium alloy, the step of removing the tetra alkyl lead from the metallic lead sludge present which comprises steam distilling the mixture with superheated steam heated to a temperature of from about 200° C. to about 400° C.
2. In the process of recovering tetra alkyl lead from metallic lead sludge which comprises contacting the mixture with superheated steam at a temperature of between 200° C. and 300° C.
3. In the process of recovering tetra ethyl lead from metallic lead sludge which comprises steam distilling the mixture with superheated steam at a temperature of between 200° C. and 300° C.
4. In the process of recovering tetra alkyl lead from metallic lead sludge which comprises flash-distilling the mixture at a temperature of from about 200° C. to about 400° C.
5. In the process of recovering tetra ethyl lead from metallic lead sludge which comprises flash-distilling the mixture at a temperature of from about 200° C. to about 400° C.
6. The process of recovering tetra alkyl lead from metallic lead sludge which comprises mixing the sludge with water, raising the temperature of a still to from 200° C. to 300° C. and gradually introducing the mixture of the sludge and water into the heated still while maintaining the still at such temperatures.
7. The process of recovering tetra ethyl lead from metallic lead sludge which comprises mixing the sludge with water, maintaining the temperature of a still at approximately from 200° C. to 300° C. and gradually contacting the mixture of the sludge and water with the heated surfaces of the still.
8. The process of recovering tetra alkyl lead from metallic lead sludge which comprises distilling the mixture in the presence of water at a temperature above that at which the tetra alkyl lead will decompose in the absence of water at atmospheric pressure.
9. The process of recovering tetra alkyl

lead from metallic lead sludge which comprises distilling the mixture in the presence of water at a temperature of from about 200° C. to about 400° C.

In testimony whereof we affix our signatures.

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