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# UNITED STATES PATENT OFFICE

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### **PRODUCTION OF ALKYLATED LEAD**

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#### 2 Claims. (Cl. 266-34)

This invention relates to the production of alkylated lead, and in particular to the making of the alloy of lead and alkali metal, e.g. sodium, which is to be reacted with an alkyl halide, e.g. 5 ethyl chloride, in the production of the alkylated lead.

A particular object of the invention is to provide an apparatus for the production of alkylated lead. Other objects will appear hereinafter. 10 These objects are accomplished as follows:

Without restricting the invention thereto, it is described in detail with particular reference to the accompanying drawing in which: Figure 1 is a conventional somewhat diagrammatic view

- 15 of an apparatus embodying the invention; and Figure 2 is a detail cross section showing a specimen of the jacketing-and-chaser lines that may be used. Referring now to the drawing:-
- The illustrated apparatus comprises a steel ves-20 sel 1 for melted lead and in which the lead may be melted or into which molten lead may charged as desired; a lead weigh tank 2; an alloy manufacturing pot 3; a storage pot 4; a liquid sodium scale tank 5; a grinder 6; and a conveyor 7.
- 25 These are connedcted by the piping 8, 9, 10, 11 and 12 as shown for the feed of the liquid lead from the pot 1 through the pipe 8 to the scale tank 2 and thence to the pot 3 through the pipe 9, the feed of the liquid sodium from the scale
- 30 tank 5 through the pipe 10 to the pot 3, the transfer of the lead mono-sodium alloy from the pot 3 by the pipe 11 to the storage pot 4, and the transfer of the alloy by the pipe 12 to the grinder 6 and thence to the conveyor 7. 35

The pot I, a steel vessel capable of holding 100,000 pounds of melted lead in the present instance, is made with a hemispherical bottom 13 and an external steel jacket 14 completely surrounding that portion of the vessel containing

<sup>40</sup> the molten lead and for the circulation of heating medium for maintaining the lead in the molten state. On the cover 15 (desirably of cast iron) is supported a motor drive 16 for a lead pump 17 for pumping the molten lead from the pot I to  $^{45}$  the tank 2.

The pump 17 is, as here indicated, a centrifugal pump of the submerged type supported from the cover 15. It comprises the impeller 18 completely submerged in the liquid and the drive 50 mechanism 19 extending upward through the

cover 15 and connecting with the motor drive 16. The scale tank 2, preferably a standard cylindrical steel tank of such proportions as to safely hold upwards to 75,000 pounds of liquid lead, 55 properly jacketed for the circulation of a heating medium to maintain the lead in a molten state and properly provided with steam chasers and a heat resisting coating, is mounted upon a heavy duty weight scale 20. This is a standard scale of the beam and lever type and of sufficient 5 sensitivity to accurately weigh or measure out the lead to the manufacturing pot 3. As will be understood, the flexibility of the connected piping permits proper operation of the scale, a proper tare being readily determined; this being also 10 the case with tank 5 and scale 31. From the scale tank 2, after a given quantity of liquid lead has been weighed, the liquid lead is allowed to flow by gravity to the pot 3 through the pipe line 9. The manufacturing pot 3 is similar to 15 the lead pot I as regards construction, shape and capacity, and is similarly jacketed for circulation of heating medium to maintain the mass molten. Similarly, it is provided with submerged type centrifugal pump 21 with impeller 22, driving 20 mechanism 23 and motor drive 24, for pumping out the lead sodium alloy. It is further provided with two agitators 25, 25 with paddles so designed as to thoroughly stir and mix the liquid lead and sodium to make a proper alloy. The drive shafts 2526. 26 of the agitators extend up through the cover plate 27 and are connected to motor drives and speed reducers conventionally indicated at 28 and 29.

The sodium melt and weight tank 5 is a con-30ventional cylindrical steel tank of such proportions as to safely hold a suitable quantity of liquid sodium. It is jacketed on the outside, as indicated at 30, for the circulation of a heating medium of sufficient temperature to hold the so- 35 dium in a molten state, and is also suitably lagged with a proper heat resisting material to prevent the loss of heat from the liquid sodium to the surrounding atmosphere. The tank is mounted on a heavy duty weight scale 31 similar to the 40scale 20.

The storage pot 4 is, as here indicated, identical in type and construction with the lead pot I and similarly provided with jacket 32 for heating medium, pump 33 and so forth. While its inclusion  $^{45}$ in the assembly is not essential, it desirably provides for the preparation of a second batch in the pot 3 while the first is being fed to the grinder.

The grinder 6 is desirably of a type in which the molten alloy is cast against the cooled shell 50walls where it solidifies and is immediately removed, or cut away, by rotary cutters, into a finely divided state of suitable structure for tetraethyl lead manufacture, such for example as that covered in the application of Stecher, Amick 55

and Daniels, Serial No. 698,384, filed November 17, 1933, for Machine and method for making solid comminuted material.

- The conveyor 7 may be either of the rotating 5 screw or vibrating type, the containing shell of which is provided with a jacket through which a cooling medium may be circulated for the purpose of removing the residual heat still in the ground alloy. The length of the conveyor is suffi-
- 10 cient to provide adequate cooling to the alloy before it is discharged to storage. The temperature of the ground alloy in the storage bin should be about  $40^{\circ}$  C. From storage the ground alloy is loaded in portable conveyors that will
- 15 hold a sufficient amount of ground alloy for one charge of a unit of the system.
  - To ensure that the lead, the sodium, and the alloy shall be held in molten condition as required, the various containers, pipe lines, valves,
- 20 fitting, etc. are jacketed for heating medium, and provided with steam chaser lines along the outside of the jacket for initially heating the jackets before passing the heating medium therethrough; and the entire piping system, including
- 25 the jackets, chasers, valves and fittings, is heavily lagged or covered with a heat resisting material to prevent loss of heat in the pipe line to the surrounding atmosphere. For simplicity in the drawing Figure 1, detailed showing of these fea-
- <sup>30</sup> tures is omitted. In Figure 2 is illustrated a typical arrangement, wherein is shown a pipe 34 (e.g. pipe 8) with the jacket 35, chaser 36 and lagging 37. It is obvious that a comparable chaser arrangement may be used in connection with e.g.
  - $^{35}\,$  a pipe for conducting heating fluid, the chaser being in contact with the exterior of the pipe.

All of the apparatus that it is necessary to so treat in order that the molten materials shall so remain, is jacketed for heating medium and pro-

- 40 vided with chaser lines for e.g. steam, as required. The heating medium may be any one suitable to the conditions and purposes. Desirably it may be one of low vapor pressure at high temperature, a temperature of 400° C, being re-
- 45 quired to keep the lead and lead sodium alloy in molten state. For example it may be a medium comprising a diphenyl compound of the group consisting of diphenyl and diphenyl oxide. Either may be used alone, or a mixture thereof, the pres-
- 50 ent apparatus being adapted to the use of any of these. At 400° C. diphenyl has a vapor pressure of approximately 160 lbs. per square inch abs., diphenyl oxide approximately 146 lbs. per square inch abs. and a mixture has, of course, a
- 55 pressure dependent on the proportions. Desirably the medium may be adapted to use in accordance with copending application Marion G. Amick, James C. Lawrence, and Joseph L. Stecher, Serial Number 746,642, Temperature control,
- <sup>60</sup> filed of even date herewith. On occasion, the medium may be circulated to the jacket of the reaction pot in relatively cool condition to remove excess heat from the exothermic reaction. In order to keep oxygen from contact with
- 65 either the liquid lead or the lead-sodium alloy, the system includes provisions for the admission of nitrogen thereinto at a pressure higher than atmospheric in order to displace all of the air and provide an inert atmosphere surrounding the 70 material within this part of the system. These
- provisions are desirably as here somewhat conventionally illustrated. Referring to Figure 1, the system shown comprises a nitrogen reservoir

tank 38 receiving nitrogen from the plant service line 39 at a pressure of 25 lbs. per square inch gauge, and from which tank the nitrogen is piped: line 40 from tank 38 to lead scale tank 2; line 41 from tank 2 to storage pot 4; line 42 from 5 tank 38 to manufacturing pot 3; line 43 from tank 38 to pipe 8; and line 44 from the top of storage pot 4 to the end of conveyor 7. From the conveyor the nitrogen enters the storage bin 45 along with the ground alloy. 10

Among the advantages of the present invention are, uniformity, ability to use larger charges with a given size manufacturing pot than are possible when adding the ingredients in solid form, and a better quality of products. 15

It is apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof and, therefore, it is not intended to be limited except as indicated in the appended claims. 20

What is claimed is:

1. In an apparatus for the production of lead sodium alloy, a manufacturing pot, a container for melting sodium and supplying the same in measured amounts to the manufacturing pot, a 25 container for melted lead for supplying the same in measured amount to the manufacturing pot, a container for melted lead for supplying the firstnamed lead container, connections for the flow of molten lead from the second-named lead con- 30 tainer to the first-named lead container, connections for conducting the sodium in molten state from the sodium container to the manufacturing pot, connections for conducting the lead in molten state from the first-named lead container 35 to the manufacturing pot, means for heating the portions of the apparatus for molten material, and means for supplying an inert medium for preventing oxidation of the materials in process in the apparatus, such means including provi- 40 sions for supplying said medium to the connections between said two lead containers.

2. In an apparatus for the production of lead sodium alloy, a manufacturing pot, a container for melting sodium and supplying the same in 45 measured amount to the manufacturing pot. a container for melted lead for supplying the same in measured amount to the manufacturing pot, a container for melted lead for supplying the firstnamed lead container, connections for the flow of 50 molten lead from the second-named lead container to the first-named lead container, connections for conducting the sodium in molten state from the sodium container to the manufacturing pot, connections for conducting the lead in molten 55 state from the first-named lead container to the manufacturing pot, a device for simultaneously cooling and finely dividing the alloy as manufactured, and connections for the flow of molten alloy from the manufacturing pot to the said de- 60 vice. said connections including a storage pot for the molten alloy, means for heating the portions of the apparatus for molten materials, and means for supplying an inert medium for preventing oxidation of the materials in process in 65the apparatus, such means including provisions for supplying said medium to the connections between said two lead containers and for supplying said medium to the storage pot.

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