Patented Jan. 12, 1943 2,307,944

UNITED STATES PATENT OFFICE

2,307,944

RECOVERY OF METAL SALTS

William H. Osborn, New York, Sidney B. Tuwiner, Kew Gardens, and John R. Smith, Flushing, N. Y., assignors to Phelps Dodge Corporation, New York, N. Y., a corporation of New York

Application August 3, 1940, Serial No. 350,906

11 Claims. (Cl. 75—63)

This invention relates to the recovery and production of a metal salt from and in a mixture thereof with another metal salt and to a process for the separation of metals utilizing such a recovery and production. It may be used, for example, in the recovery and production of tin chloride from and in mixtures thereof with lead chloride and especially as a step in a process for the separation of tin from lead.

In the art of removing lead from molten, metallic tin, liquid stannous chloride may be applied in molten form to the surface of a kettle of tin containing lead and an equilibrium is established between the lead chloride in the stannous chloride and the lead dissolved in the tin. This equilibrium is such that by repeated additions of fresh stannous chloride into a drossing kettle, substantially all of the lead may be removed from the molten tin. For example, by treating successive batches of molten tin metal containing approximately 3 to 4% lead in solution with successive batches of molten stannous chloride, it is possible to obtain final mixed chlorides running 30% or better in lead, and final tin running as low as .003% lead, which tin is of increased value.

Stannous chloride may be separated from the lead chloride dissolved in it by utilizing the difference in boiling points between the two materials, since at atmospheric pressure stannous chloride boils at approximately 1166° F. and lead chloride at approximately 1652° F. However, such a separation by distillation presents difficulties due to the corrosive action of the chlorides and the fact that as the percentage of stannous chloride is reduced in the mixed chlorides, the partial pressure of stannous chloride vapor will be reduced to such an extent that separation by distillation becomes uneconomical, since excessive temperatures or excessive times would have to be used.

It is an object of the present invention to provide an improved method for recovering and producing a metal salt from and in a mixture of such salt with another metal salt. A more specific object is to provide an improved method whereby stannous chloride, or another metal salt, may be distilled from and produced in a mixture thereof with tin chloride, or other metal salt. It is also an object to provide a new cyclic process for the recovery of metals in which such a recovery and production of a metal salt is utilized in the regeneration of the metal salt. More specifically it is an object to provide a cyclic process by which lead may be separated from tin, in which stannous chloride is used to effect the separation and the stannous chloride used in that step is regenerated in and recovered from the mixture of stannous chloride and lead chloride resulting from such separation. It is also an object to recover metallic tin from the alloy as a pure metal and to recover lead as a component of a commercially salable product such as a 50–50 lead-tin solder. Other objects will become apparent.

We have found that when a mixture of stannous and lead chlorides is placed in a suitable distilling vessel over a bath of molten tin, or of a molten mixture containing tin and particularly one containing a predominance of molten tin over molten lead, and a temperature is applied (with or without vacuum or pressure and with or without passage of an inert gas through the liquids) such that the stannous chloride will vaporize, the chloride element of the mixed salts may be removed as stannous chloride vapor. For, as the percentage of stannous chloride in the mixture of chlorides is reduced by vaporization, lead chloride, in excess of that in the equilibrium mixture between lead chloride dissolved in stannous chloride and lead dissolved in tin in the metals of the molten bath, will be converted to lead, which will dissolve in the tin or mixture containing tin, and a corresponding amount of tin will be converted to stannous chloride which will be distilled off as vapor. The course of the reaction, which is illustrated by the reaction equation:

$$PbCl_2+Sn \rightarrow Pb+SnCl_2 \uparrow \text{(vapor)}$$

is continuously moved to the right by maintaining the mixture of chlorides over the mixture of metals at such a temperature that the more volatile stannous chloride is continuously removed from the zone of the reaction as a vapor.

Thus by distilling a mixture of lead and tin chlorides over a bath of tin, it is possible to distill substantially all of the chlorine content of the chlorides as stannous chloride and to throw down substantially all of the lead content of the original mixed chlorides as metallic lead. If the amount of tin in the molten bath is just sufficient to satisfy all of the chlorine in the lead chloride, a pure lead may be recovered. Or the amount of tin in the molten bath may be in excess of that required to satisfy the chlorine in the lead chloride and the lead may be recovered as a lead tin alloy that is commercially salable. For example, the amount of tin to be used in the molten bath may be calculated, from the amount of lead chloride in the metal salts to be separated, to give a solder containing 50% lead and 50% tin at the end of the distillation, thus providing, without further cost, a product of commercial value. By this means it is possible to substantially completely separate stannous chloride from lead chloride and to reduce the lead to pure metallic lead or to lead alloyed with tin to form a solder metal, without loss of chlorine.

The temperature of the distillation may be reduced by bubbling an inert gas through the mixture of chlorides to help carry off the stannous chloride vapors at temperatures below the actual