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RECOVERY OF ANTIMONY

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This invention relates to the recovery of antimony from other metals with which it is commonly associated and particularly to the separation of antimony from tin or lead-tin mixtures, such as solder metals.

In the refining of tin or solder metals containing metallic antimony as an impurity, it is difficult to obtain a satisfactory and economical separation of the antimony from the other metals. It is an object of the present invention to provide an improved method for effecting a separation of antimony from other metals. Another object is to provide an improved method for the recovery of antimony from drosses containing metallic aluminum or containing other active drossing metals. Other objects will become apparent.

In the refining of tin or solder metal or other metal mixtures containing metallic antimony as an impurity, metallic aluminum or metallic sodium, or some other active drossing metals, may be stirred into a pot of the tin or solder metal that is being refined, in such a manner that it will combine with the antimony present in the bath to form intermetallic compounds of relatively high melting point. These intermetallic compounds, such as the intermetallic compound of antimony and aluminum, generally given the formula AlSb, will rise to the surface of the pot as a mushy dross that can be easily separated from the rest of the bath by skimming it. These drosses necessarily entrain considerable proportions of the tin, solder metal or other metals with which they are associated. By subjecting such drosses to high pressure in a mold at temperatures above the melting point of the tin or solder metal, etc. entrained in them, it is possible to expel a large part of this entrained metal and leave a dross containing a higher content of antimony. For example, a skimmed dross containing approximately 8% antimony, 2.2% aluminum, and the balance principally tin, when subjected to pressures of approximately 30,000 pounds per square inch in a mold, at about 700° F. gave, by expulsion of tin and other molten metals, a remaining dross cake containing about 52% antimony, 14.25% aluminum and 30% tin.

Such a cake containing the intermetallic compounds AlSb, plus remaining entrained tin, etc. metal, may be finely ground in an apparatus, such as a ball mill, and then subjected to heat in an atmosphere of steam, whereby the aluminum may be oxidized to alumina and the antimony may be oxidized to the trioxide which, at temperatures in the neighborhood of 1600° F., is volatile and may be carried off in the current of steam. The higher oxides of antimony, that is, the so-called tetraoxide Sb_2O_4 and the pentoxide Sb_2O_5, are relatively non-volatile at those temperatures. Therefore, if any excess of air or free oxygen is contained in the current of gas (steam) which is used in the oxidation of the ground charge, at least some of the antimony will be oxidized to one or more of the higher oxides and so will not be removed.

A certain amount of arsenic is almost always present in the original kettle of molten metals. In forming the drosses this arsenic also forms intermetallic compounds with the aluminum and other active metals used as a drossing agent and is thus incorporated in the final press cake. If the press cake is ground in water, a very poisonous gas, arsine, or arsenous hydride (AsH_3), may be formed. It is also possible that the poisonous gas stibine, or antimonous hydride (SbH_3), may be formed. To prevent the formation of these poisonous gases in the crushing of the antimony dross cake, the cake may be crushed as it comes out of the hot press and while it is still above the temperatures at which those poisonous gases are formed and then may be dropped immediately as a coarse, crushed product into water-free oil, such as common fuel oil distillate. The crushed cake, together with the oil, is then charged into a ball mill where it may be ground without danger of generation of these poisonous gases. As a precaution against the possibility of any water or water vapor getting into the ball mill, it is advisable to connect the ball mill directly through a relief valve to a gas vent by which such gases may be exhausted. After the cake has been crushed so that the particle sizes are about 200 mesh, the charge may be removed from the ball mill and allowed to settle from the oil, and the excess oil may be decanted, or otherwise separated from the crushed particles, and reused. The crushed material from which the bulk of the oil has been decanted or otherwise removed may be dried, preferably at a temperature of approximately 500° F. in an inert atmosphere, such as carbon dioxide gas, whereby the excess oil is distilled off and a substantially dry and oil-free powdered product is obtained.

This powdered product consists essentially of crystals of aluminum-antimony intermetallic compound plus particles of metallic tin or solder. It may be introduced into an externally heated rotary kiln, through which a current of steam is passing, where it is heated to about 1600° F. The steam breaks down in the presence of the metals