This invention relates to the manufacture of the lead salts of stypnic acid. These lead salts, especially the normal salt, find extensive use in the explosives industry, being specially desired as ingredients of ammunition priming mixtures. In the patent to Herz, No. 1,443,228, there is disclosed a method for the preparation of the normal lead salt of stypnic acid from the more soluble alkaline metal or alkaline earth metal salts, particularly the sodium salt. Preparation of the lead salt in this way presents substantial technical difficulty due to the relatively slight solubility of even the sodium salts, the other salts of this class being still less soluble. In the patent to Herz, No. 1,899,728, a method is disclosed which involves the use of the magnesium salt. Prior to the present invention, no other soluble stypnic acid salt have been known or known to be available and suitable for the manufacture of such relatively insoluble salts as the lead salts. It has now been discovered that soluble stypnates of several metals can be prepared and utilized to substantial advantage in the preparation of lead and other relatively insoluble stypnates. Among such soluble salts may be mentioned those of cadmium, zinc, manganese, iron, cobalt and nickel. Solutions of the stypnates of any of these metals may be prepared by the method of dissolving together the requisite amounts of stypnic acid and the oxide or carbonate of the metal, and filtering off the solid residues. From the resulting solution of a soluble stypnate, which may be stored in the usual manner, lead stypnate can be made by the method of slowly adding to the solution of the soluble stypnate a solution of a soluble lead salt, for example, lead nitrate. If the stypnic acid used is of the requisite purity, the lead stypnate is precipitated in crystals of a form and size suitable for use as the explosive ingredient of ammunition priming mixtures.

Specific examples of the preparation of soluble stypnates of the group above mentioned and of the making of lead stypnate therefrom are as follows:

**Example 1.—Manufacture from Zinc Stypnate**

72 grams of stypnic acid and 24 grams of zinc oxide are dissolved in about 200 c. c. of water. This solution is filtered, and diluted by the addition of 700 c. c. (more or less) of water to a specific gravity of 1.04. An acidity determination is made and the acidity adjusted to the point at which 100 c. c. of the 1.04 specific gravity solution diluted with 250 c. c. of water is neutralized by 6 to 8 c. c. of a one-half normal solution of sodium hydroxide. 240 c. c. of the zinc stypnate solution thus prepared is precipitated with 80 c. c. of 30% lead nitrate solution, the temperature of the solutions being preferably about 60° or 65° C. This results in a yield of about 17.5 grams of crystalline lead stypnate, which may be recovered by filtration and washing in the usual manner.

**Example 2.—Manufacture from Manganese Stypnate**

34 grams of manganese carbonate and 72 grams of stypnic acid are dissolved in 200 c. c. of water. The solution is filtered and diluted to a specific gravity of about 1.04. Acidity is adjusted, as described in conjunction with the preparation of the zinc stypnate solution. 240 c. c. of the manganese stypnate solution thus prepared is precipitated with 80 c. c. of 30% lead nitrate solution, preferably at a temperature of about 60° to 65° C. The precipitate of about 15.8 grams of normal lead stypnate may be recovered by filtration and washing.

This application is a continuation-in-part of this inventor’s prior co-pending application, Serial No. 35,063, filed on August 7, 1935.

What is claimed is:

1. The method of making lead stypnate, which comprises the precipitation thereof by the addition of an approximately 30% solution of lead nitrate to a solution having a specific gravity of about 1.04 of a soluble stypnate selected from the group consisting of the stypnates of cadmium, zinc, manganese, iron, cobalt and nickel, said lead nitrate solution and soluble stypnate solution being in the proportions of about 80 cc. of lead nitrate solution to 240 cc. of soluble stypnate solution.

2. The method of making lead stypnate, which comprises the precipitation thereof by the addition of an approximately 30% solution of lead nitrate to a solution having a specific gravity of about 1.04 of a soluble stypnate selected from the group consisting of the stypnates of cadmium, zinc, manganese, iron, cobalt and nickel, said lead nitrate solution and soluble stypnate solution being in the proportions of about 80 cc. of lead nitrate solution to 240 cc. of soluble stypnate solution, and the reacting solutions being at a temperature of about 60° C.

3. The method of making lead stypnate, which comprises the precipitation thereof by the addition of an approximately 30% solution of lead nitrate to a solution having a specific gravity of about 1.04 of a soluble stypnate selected from the group consisting of the stypnates of cadmium, zinc, manganese, iron, cobalt and nickel, said lead nitrate solution and soluble stypnate solution being in the proportions of about 80 cc. of lead nitrate solution to 240 cc. of soluble stypnate solution, and the reacting solutions being at a temperature of about 60° C.
nitrato to a solution of nickel stypnate having a specific gravity of about 1.04, said reacting solutions being in the proportions of about 80 cc. of lead nitrate solution to 240 cc. of zinc stypnate solution.

4. The method of making lead stypnate, which comprises the precipitation thereof by the addition of an approximately 30% solution of lead nitrate to a solution of zinc stypnate having a specific gravity of about 1.04, said reacting solutions being in the proportions of about 80 cc. of lead nitrate solution to 240 cc. of manganese stypnate.

5. The method of making lead stypnate, which comprises the precipitation thereof by the addition of an approximately 30% solution of lead nitrate to a solution of manganese stypnate having a specific gravity of about 1.04, said reacting solutions being in the proportions of about 80 cc. of lead nitrate solution to 240 cc. of manganese stypnate.

WILLI BRUN.