

UNITED STATES PATENT OFFICE

2,493,551

PRODUCTION OF NORMAL LEAD
DINITRORESORCINATELeon Rubenstein, Saltcoats, Scotland, assignor to
Imperial Chemical Industries, Limited, a corpo-
ration of Great BritainNo Drawing. Application July 8, 1947, Serial No.
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7 Claims. (Cl. 260—435)

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The present invention is concerned with a new or improved process for the production of normal lead salt of 2:4 dinitroresorcinol, whereby said salt may be produced in free-running form without the necessity for corning it.

In Hale U. S. P. 2,116,514 there are described priming compositions for propellant powders and other explosives, consisting of normal lead 2:4 dinitroresorcinate prepared by the action of a lead salt such as lead nitrate on sodium dinitroresorcinate. When prepared in the usual manner by the addition of a solution of soluble normal lead salt to an aqueous solution of a normal salt of 2:4 dinitroresorcinol normal lead 2:4 dinitroresorcinate has no free flowing properties, and hitherto it has been necessary to corn it with gum arabic before it can be used for certain purposes, such as the production of delay fuses, or as an ignition agent in detonators.

According to the present invention the process for the production of normal lead 2:4 dinitroresorcinate comprises mixing together at a temperature not lower than 60° C. an aqueous solution of normal lead acetate with an aqueous solution containing a magnesium salt of 2:4 dinitroresorcinol having about $\frac{1}{2}$ to $\frac{3}{4}$ the quantity of magnesium present in the normal magnesium salt, the said lead acetate and magnesium salt being admixed in stoichiometrical equivalent weights.

The precipitate thus formed may then be washed with water and dried.

By the term "magnesium salt of 2:4 dinitroresorcinol having about $\frac{1}{2}$ to $\frac{3}{4}$ the quantity of magnesium present in the normal magnesium salt" is meant a product such as is obtained by interaction of 2:4 dinitroresorcinol and for example magnesium oxide in $\frac{1}{2}$ to $\frac{3}{4}$ the proportion necessary to give the normal magnesium salt of 2:4 dinitroresorcinol.

The pH of the solution containing the magnesium salt of 2:4 dinitroresorcinol having about $\frac{1}{2}$ to $\frac{3}{4}$ the quantity of magnesium present in the normal magnesium salt is about 3.8 to 5.4 when measured at ordinary temperature. This solution may obviously be formed either by addition of 2:4 dinitroresorcinol to a hot solution of the normal magnesium salt thereof, or by adding to a hot suspension of 2:4 dinitroresorcinol in water from about $\frac{1}{2}$ to $\frac{3}{4}$ of the amount of magnesium oxide or carbonate required to form the normal magnesium salt.

In carrying out the precipitation the normal lead acetate solution may be gradually added to the aforesaid solution containing the said

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magnesium salt of 2:4 dinitroresorcinol, or both solutions may be run together into water. The precipitation temperature is preferably about 70° C.

The resulting free-running normal lead 2:4 dinitroresorcinate may be used as a substitute for lead styphnate as an igniting agent in detonators and the like, for instance in place of lead styphnate in compositions containing lead styphnate and lead azide together. Mixtures of the said normal lead 2:4 dinitroresorcinate and lead azide show less tendency to segregation than the azide with lead styphnate. It may also be used as a fast burning delay fuse composition in military and commercial detonators. Since it does not require to be corned it can be loaded very easily by apparatus employing charging plates.

The invention is illustrated by the following example:

Example

10 gm. of 2:4 dinitroresorcinol are mixed into 150 c. c. of water at 80° C. with 1.0 gm. magnesium oxide ($\frac{1}{2}$ equivalent), until substantially complete solution is attained. To the filtered solution of the resulting acid magnesium salt, maintained at a temperature near 70° C. and stirred, 157 c. c. of 12% lead acetate solution are added at a constant rate over 11 minutes. A curdy orange-yellow precipitate is formed at first, but soon this changes to a dark red dense granular precipitate. After cooling, filtering and washing with water, the material is dried at 40° C.

The slightly lumpy product breaks down on gentle pressure to a dark red free-running powder.

The product is found to be less sensitive to percussion and friction but somewhat more sensitive to flame than lead styphnate. It mixes well with lead azide, being superior to lead styphnate in this respect, and the resulting mixtures in proportions around 70% lead azide: 30% normal lead 2:4 dinitroresorcinate ignite satisfactorily in detonators for safety fuse or electric fuseheads, and initiate satisfactorily a base charge of tetryl.

I claim:

1. A process for the production of normal lead 2:4 dinitroresorcinate which comprises mixing together at a temperature not lower than 60° C. an aqueous solution of normal lead acetate with an aqueous solution containing a magnesium salt of 2:4 dinitroresorcinol having about $\frac{1}{2}$ to $\frac{3}{4}$ the quantity of magnesium present in the normal magnesium salt, the said lead acetate and mag-

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nesium salt being admixed in stoichiometrical equivalent weights.

2. A process as claimed in claim 1 wherein the solution containing the said magnesium salt of 2:4 dinitroresorcinol is formed by addition of 2:4 dinitroresorcinol to a hot solution of the normal magnesium salt thereof.

3. A process as claimed in claim 1 wherein the solution containing the said magnesium salt of 2:4 dinitroresorcinol is formed by adding about $\frac{1}{2}$ to $\frac{3}{4}$ of the amount of magnesium oxide required to form the normal magnesium salt to a hot suspension of 2:4 dinitroresorcinol in water.

4. A process as claimed in claim 1 wherein the solution containing the said magnesium salt of 2:4 dinitroresorcinol is formed by adding $\frac{1}{2}$ to $\frac{3}{4}$ of the amount of magnesium carbonate required to form the normal magnesium salt to a hot suspension of 2:4 dinitroresorcinol in water.

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5. A process as claimed in claim 1 wherein the solution of normal lead acetate and the solution containing the said magnesium salt of 2:4 dinitroresorcinol are run together into water.

6. A process as claimed in claim 1 wherein the temperature of mixing is about 70° C.

7. A process as claimed in claim 1 wherein the precipitate of normal lead 2:4 dinitroresorcinate formed is washed with water and dried.

LEON RUBENSTEIN.

REFERENCES CITED

The following references are of record in the file of this patent:

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