Oct. 10, 1939. L. A. BURROWS ET AL 2,175,249

IGNITION COMPOSITION

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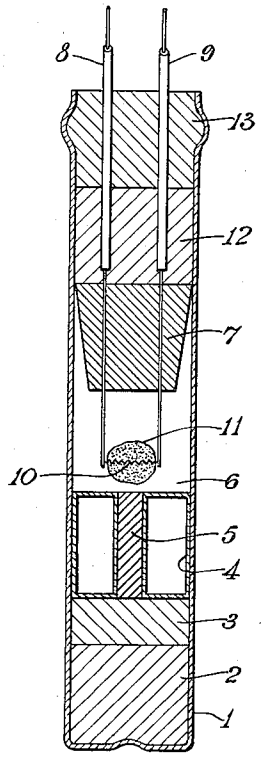

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IGNITION COMPOSITION

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16 Claims. (Cl. 52—4)

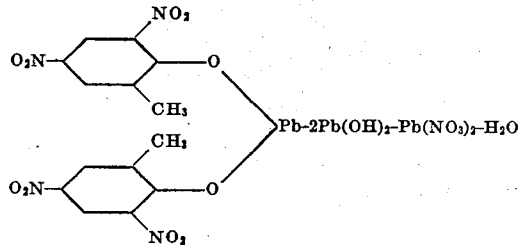
This invention relates to new and improved ignition compositions adapted for efficient use in blasting initiators and particularly in electric blasting initiators.

An ignition composition to meet the commercial requirements satisfactorily in electric blasting caps and other initiators must possess certain characteristics and properties. It is essential, for example, that it be capable of ready ignition on application of the heat from the passage of an electric current through a fine, highly-resistant wire in contact with the material. Not only must ignition be possible, but it must take place with the use of a low firing current. In the case of initiators where ignition is brought about by means of the spit from a fuse, such ignition must be certain also. The composition must be sufficiently stable so that its ability to ignite is a constant property not affected by storage even under unfavorable conditions. In addition to ready ignitibility by the initiating means, a composition to be satisfactory must be capable of giving off sufficient heat and flame on ignition or of initiating a shock impulse sufficient to bring about the firing of an adjacent main explosive charge.

While many compositions have been proposed for such use in electric blasting caps and other initiators, no one of them has been entirely satisfactory in every respect.

An object of the present invention is the production of improved ignition agents. A further object is the preparation of ignition agents suitable for use in electric blasting caps and other blasting initiators. A still further object is the preparation of new chemical compounds of a desirable degree of sensitiveness and suited for use in the manner described. Additional objects will be disclosed as the invention is described more fully hereinafter.

We have found that the foregoing objects are accomplished by the preparation of an ignition agent comprising a complex or double salt of lead nitrate with a lead salt of a nitrophenol. These salts are sometimes found to be hydrated salts, containing water of crystallization. The bis basic double salts are the ones preferably used. The lead salts of various nitrophenols are adapted for use in this manner; for example, the salts of dinitrocresols, dinitrophenols, trinitrophenol, and the like. Preferably we make use of the double salt of 4,6-dinitro-ortho-cresol which may be designated more exactly as 3,5-dinitro-2-hydroxy-1-methyl benzene. Wherever we may hereafter refer in this application to 4,6-dinitro-ortho-cresol, it will be understood that this is identical with 3,5-dinitro-2-hydroxy-1-methyl benzene. Our preferred complex salt of dinitro-ortho-cresol may be designated as lead nitrato-bis basic lead-4,6-dinitro-ortho-cresylate monohydrate. This salt has the following structural formula:



As examples of methods of preparing the complex salts in accordance with our invention, the following will serve:

EXAMPLE 1.—*Lead nitrato-bis-basic lead-4,6-dinitro-ortho-cresylate monohydrate*

A solution of 95.1 gms. of 4,6-dinitro-ortho-cresol in 2250 cc. of water, containing 57.6 gms. of sodium hydroxide, was added in a small, steady stream over 27 minutes to a solution of 348 gms. of lead nitrate in 6000 cc. of water maintained at 60 to 61° C. After the addition, stirring was continued while the suspension was cooled to room temperature by a cold water bath. The supernatant liquid was decanted and the crystalline solid washed once by decantation with 300 cc. of cold water and twice by stirring with 350 cc. of cold water and filtering. The orange colored crystalline product was air-dried at 70 to 75° C. A weight of 335.9 gms. of product was obtained, containing 58.69% lead and 1.81% nitrate nitrogen. That this product was a monohydrate is indicated by the fact that the loss in weight in vacuo at 150° C. amounted to 1.3% as against 1.26% for the theoretical amount of water in the monohydrate.

EXAMPLE 2.—*Lead nitrato-bis basic lead picrate*

A solution of 18.32 gms. of picric acid in 400 cc. of water containing 9.6 gms. of sodium hydroxide, was added dropwise during 30 minutes to a well-stirring solution of 55.6 gms. of lead nitrate in 1000 cc. of water maintained at 70 to 72° C. After all had been added, the suspension was cooled to room temperature, filtered, washed once by decantation and twice by agitating with cold water and filtering. The air-dried product

consisted of glistening yellow prisms and weighed 58.2 gms. The lead content was 56.17%.

Example 3.—Lead nitrato-bis basic lead-2,4-dinitrophenolate

A solution of 14.72 gms. of 2,4-dinitrophenol in 400 cc. of water, containing 9.6 gms. of sodium hydroxide, was added dropwise during 20 minutes to a well-agitated solution of 58 gms. of lead nitrate in 1000 cc. of water maintained at 70 to 72° C. After the addition, the mixture was stirred and heated to 80° C. for 30 minutes. The solution was then cooled and filtered. The precipitate was washed once by decantation and twice by stirring with 100 cc. of cold water and filtering. The crystals were dried by washing on the filter with acetone and consisted of bright yellow needles weighing 46.7 gms. The product contained 58.91% lead and 1.5% nitrate nitrogen.

The foregoing examples show methods of preparing complex lead salts of nitrophenols and lead nitrate for use in accordance with our invention. It will be understood that many other salts may be applied successfully also, and that the method of procedure may be varied considerably. The examples given are by way of illustration only.

While the above complex salts may be used by themselves, preferably they will be admixed with other materials; for example, oxidizing agents such as potassium chlorate, barium peroxide, calcium peroxide, and the like, and agents giving off heat and sparks, such as finely-divided oxidizable metals, for example, zirconium, magnesium, aluminum, and the like. As specific compositions, we cite the following:

Complex lead salt of—	1	2	3
	Percent	*Percent*	*Percent*
4, 6-dinitro-ortho-cresol	80	80	70
Zirconium	20	----------	15
Potassium chlorate	----------	20	15

All three of the foregoing compositions, when included as charges in electric blasting caps, are readily ignited by the heat of an incandescent bridge wire, and have low firing current requirements. Moreover, all are less sensitive to shock than mercury fulminate, and throw out hot igniting flames. It will be understood, however, that the sensitiveness to shock as well as to electro-static effects will depend on the nitrophenol used, the double salts of the trinitro-compounds being more sensitive in general than those of the dinitro-compounds. Not only are the above compositions of a desirable range of properties when prepared, but these properties remain constant on storage. For example, the compositions shown have been stored for six months at a temperature of 110° F. and have been found to undergo no appreciable change during such heated storage.

It will be understood that the compositions described, containing our new complex salts, may be used as ignition compositions under varying conditions. For example, they may be employed as cap charges for ignition by means of fuse, as loose charges in electric blasting caps or electric squibs, or as the ignition charge in caps of the so-called concave plug type. Preferably, we use the composition containing the lead nitrato-bis basic lead-4,6-dinitro-ortho-cresylate as an ignition charge in the form of a bead surrounding a bridge wire. A desirable composition will comprise more than 50% of the double salt, 5 to 30% potassium chlorate, and 5 to 30% zirconium. It has particular advantage in such use for igniting delay charges in electric blasting caps because of its ready ignition, low firing current requirements, certain action, and because of the hot flame given off on ignition.

In order to disclose this latter preferred embodiment of our invention more fully, reference is made to the accompanying drawing. It is to be understood, however, that this is to be taken solely by way of illustration and is not to be regarded as a limitation upon the scope of our invention which has many important applications other than those herein particularly illustrated.

Referring generally to the drawing, the sole figure represents a vertical section of an electric blasting initiator of the delay type provided with the special bridge plug bead type of ignition.

In particular, the electric blasting initiator comprises the rigid shell **1**, suitably of metal; in the base of the shell is placed a base charge **2**, of either detonating or deflagrating material, depending on whether a squib or a cap is desired. Superimposed on the base charge is an intermediate charge capable of initiating said base charge. Above said intermediate charge **3** and in firing relation therewith, is placed the delay element comprising the metal carrier **4**, which contains the delay composition **5**, preferably of the type which burns substantially without the evolution of gas. The delay element is, in turn, in firing relation with the electrical ignition means above it through the chamber **6** to permit the expansion and cooling of the gases. The electrical ignition means above this chamber comprises the bridge plug **7**, through which are passed the leading wires **8** and **9**, the ends of which are brought into electrical connection by means of the high resistance bridge wire **10**. The ignition composition according to our invention is fixed on the bridge wire in the form of the compact bead **11**, which bead is preferably suspended thereon without the aid of additional support. Closure is effected by means of the waterproofing layer **12** and the sealing layer **13**.

While our invention has been described in detail in the forgoing, it will be understood that many variations may be made from the compositions shown and the details of preparation and use, without departing from the spirit of the invention. We wish to be limited only by the following patent claims.

We claim:

1. A chemical compound consisting of a complex salt of lead nitrate with a lead salt of a nitrophenol.

2. A chemical compound consisting of a complex salt of lead nitrate with a basic lead salt of a nitrophenol.

3. A chemical compound consisting of a complex salt of lead nitrate with a bis basic lead salt of a nitrophenol.

4. A chemical compound consisting of a complex salt of lead nitrate with a bis basic lead dinitrophenolate.

5. A chemical compound consisting of a complex salt of lead nitrate with a bis basic lead picrate.

6. A chemical compound consisting of a complex salt of lead nitrate with a bis basic lead dinitro-ortho-cresylate.

7. A chemical compound consisting of a complex salt of lead nitrate with a bis basic lead salt of 4,6-dinitro-ortho-cresol.

8. An ignition agent comprising a complex salt of lead nitrate with a lead salt of a nitrophenol.

9. An ignition agent comprising a complex salt of lead nitrate with a basic lead salt of a nitrophenol.

10. An ignition agent comprising a complex salt of lead nitrate with a bis basic lead salt of a nitrophenol.

11. An ignition agent comprising a complex salt of lead nitrate with a bis basic lead dinitrophenolate.

12. An ignition agent comprising a complex salt of lead nitrate with a bis basic lead picrate.

13. An ignition agent comprising a complex salt of lead nitrate with a bis basic lead dinitro-ortho-cresylate.

14. An ignition agent comprising a complex salt of lead nitrate with a bis basic lead salt of 4,6-dinitro-ortho-cresol.

15. An ignition composition comprising 5 to 30% potassium chlorate, 5 to 30% zirconium, and more than 50% of a double salt of lead nitrate with a bis basic lead salt of 4,6-dinitro-ortho-cresol.

16. In an electric blasting initiator of the delay type, an ignition charge in the form of a bead surrounding the bridge wire, which charge comprises a complex salt of lead nitrate with a lead salt of a nitrophenol.

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