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EXPLOSIVE

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This invention relates generally to explosives and particularly to nitroso derivatives of resorcinol and the compounds thereof.

In the explosive compositions, such as those employed in the priming mixtures of ammunition, the ingredients must be selected with reference not only to the explosive activity in the office or function which the ingredient is to fulfill upon detonation, but care must also be taken to select ingredients which are compatible with each other and which are stable under adverse atmospheric conditions. In some cases the severe atmospheric conditions may be artificial, but, where there is liability that the ammunition may be subjected to them, it obviously must be capable of withstanding the severity and adversity of such conditions.

Heretofore considerable difficulty has been countered with priming mixtures insofar as maintenance of stability under adverse storage conditions is concerned. The usual priming mixture is composed of a number of ingredients which fall, generally, into three classes, to-wit: initiators, oxidizers, and fuels and in some cases abrasives. With such a mixture it is apparent that under certain conditions of temperature, moisture and the like, the respective ingredients are not only liable to decompose of themselves, but reactions are liable to be set up among the respective ingredients resulting in deterioration of the primer with consequent unreliability of the ammunition.

The object of the present invention, generally stated, is to provide an explosive compound having a high order of stability under a variety of conditions.

Another object of the present invention is to provide a fuel ingredient for an ammunition priming mixture which is not only stable but is compatible with the other ingredients of the priming mixture under a variety of conditions.

A more specific object of the invention is to provide a nitrosoresorcinol compound capable of use as the fuel ingredient in a priming mixture for ammunition.

A further object of the present invention is to provide a process of making nitrosoresorcinol compounds of an explosive character.

Other objects will become apparent to those skilled in the art when the following description is understood.

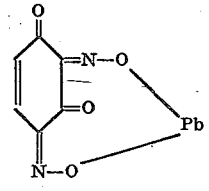
In accordance with the present invention, compounds of nitroso derivatives of resorcinol are provided and employed as ingredients of explosive compositions, such for instance as in the priming charge of ammunition and in other detonating charges. The heavy metal salts of the polynitrosoresorcinols particularly exhibit characteristics which indicate that they may be advantageously used in such explosive compositions. While the individual isomers, and the various salts thereof with different metals, vary somewhat as to such specific characteristics as sensitivity and flash point, the entire group of explosive nitrosoresorcinol compounds is characterized by a high order of stability even under extremely severe and adverse conditions. On account of the relative ease and economy of their manufacture, compounds of dinitrosoresorcinol particularly commend themselves for use. For instance, the lead salts of dinitrosoresorcinol prepared in any suitable manner may be used with particular advantage as the fuel ingredients in an ammunition priming mixture. In addition to fulfilling the office and function of the ordinary fuel ingredient in such a priming mixture, the lead salts of dinitrosoresorcinol are somewhat sensitive to friction and impact and hence their presence contributes to the sensitivity of the priming composition, and, in fact, smaller quantities of the initiating ingredient may be employed than with other known fuels, such as antimony sulphide or lead thiocyanate. Likewise other heavy metal salts of dinitrosoresorcinol may be used, the silver salt, for instance, being somewhat less sensitive than the lead salt, and likewise the cadmium and copper salts.

Such salts of dinitrosoresorcinol may be prepared from dinitrosoresorcinol which latter may have been produced by any suitable process such, for instance, as that described by Orndorff and Nichols in the Journal of the American Chemical Society, vol. 45, page 1536 (1923). The salts of dinitrosoresorcinol may be obtained by reaction with the desired base in the usual manner for obtaining salts. The water soluble salts of dinitrosoresorcinol may be readily made by treating a given amount of dinitrosoresorcinol with an equivalent amount of a metal hydroxide or carbonate in aqueous solution and then evaporating the water. Water insoluble salts of dinitrosoresorcinol can also be formed by this method provided the hydroxide or carbonate of the desired metal is itself soluble in water. In case, however, the hydroxide or carbonate of the metal desired is insoluble in water, as is the case with lead, a double decomposition reaction between two salts is resorted to as, for instance, by first making an alkali salt of dinitrosoresorcinol. This alkali salt may then be reacted in the presence of a soluble lead salt such, for instance, as lead acetate or ni-

trate with the result that lead dinitrosoresorcinate is precipitated while the other products of the reaction remain in solution.

Lead salts may be obtained in either an amorphous or granular form, the explosive properties of each form being substantially the same, but, because of the greater convenience in handling the material, the granular form is the more advantageous to use when the conventional loading and mixing machinery is to be employed in the preparation and charging of primer compositions. Moreover, the lead salts may be obtained as either the normal or the basic salt or in the form of a mixture of the normal and the basic salts as will be more particularly described hereinafter.

As one illustrative embodiment of the present invention, normal lead dinitrosoresorcinate may be obtained by treatment of dinitrosoresorcinol as follows: 60 grams of 2,4 dinitrosoresorcinol monohydrate are suspended in 500 cc. of water and a solution of 28.5 grams of sodium hydroxide in 100 cc. of water is added with stirring. This reacts to produce a solution of sodium dinitrosoresorcinate. 15 cc. of this solution are added to a solution of 132 grams of lead acetate and 45 grams of glacial acetic acid in 2,000 cc. of water heated to a temperature of 80° C. The lead salt of the dinitrosoresorcinol immediately appears as a brown amorphous precipitate, but, upon vigorous agitation of the bath for about five minutes, the amorphous precipitate is converted into black granules which are substantially spherical in shape. The remainder of the sodium dinitrosoresorcinate solution is then gradually added to the bath while vigorous agitation thereof continues and the temperature maintained at about 80° C. Substantially forty-five minutes may be consumed in completing the mixture of the two solutions and agitation should proceed for a short time as, for instance, five minutes after the two solutions have been completely mixed, the mixture may then be permitted to stand for a time sufficient to permit the precipitate to settle, the supernatant spherical granules of lead salt of dinitrosoresorcinol washed with water and dried at a suitable temperature, such for instance as 50° C. The product of this reaction has been identified as normal lead dinitrosoresorcinate having the following formula:

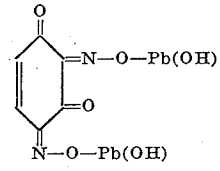


From the procedure above described, a yield of approximately 112 grams of the black spherical granular normal lead dinitrosoresorcinate may be expected. This material has a density of 3.665 at 25° C. It is substantially insoluble in water at any temperature. The material flashes instantaneously when dropped into a metal cup immersed in a Wood's metal bath at 274° C. and retains this flash point after treatment and storage under severe conditions such, for instance, as after three weeks storage at 74° C. at 90% relative humidity. Even after being boiled for four hours in water, the flash point of 274° C. was retained. When touched with a match flame, the salt deflagrates rapidly leaving an amorphous carbonaceous deposit. On analysis, the salt was found to contain 55.53% lead, in close agreement with 55.52%, the theoretical lead content corresponding to the formula given above.

As a further illustrative embodiment basic lead nitrosoresorcinate may be prepared as follows:

A solution containing 25 grams of 2,4 dinitrosoresorcinol monohydrate, 21.5 grams of sodium hydroxide, and 1000 cc. of water is gradually added over a period of 50 minutes to a solution of 100 grams of lead nitrate in 500 cc. of water, which is maintained at 80° C. and well agitated. After standing one-half hour, the product is obtained as a light brown precipitate consisting of agglomerated amorphous particles. The resultant product may be washed and dried as with the previous example.

The product has been determined to be basic lead dinitrosoresorcinate having the following formula:



Analysis shows the lead content of the product to be 67.09% in substantial agreement with the theoretical value of 67.29% based upon the formula.

The material has a flash point of 242° C. but is somewhat less sensitive to impact than the normal lead salt of dinitrosoresorcinate described in the previous embodiment. When touched with a match flame the substance, however, deflagrates more violently than the normal salt and the carbonaceous residue is spread over a wider area.

In the apparatus employed for determining the flash points of the materials as hereinbefore stated, the flash points of other known explosive materials are indicated as follows:

	Degrees Centigrade
Normal lead styphnate	328
Basic lead styphnate	254
Diazodinitrophenol	225

As a further example of an illustrative process in which the resultant product is composed of both the normal and basic salts of lead dinitrosoresorcinate the following procedure may be carried out.

A solution containing 25 grams of 2,4 dinitrosoresorcinol monohydrate, 21.5 grams of sodium hydroxide, and 1000 cc. of water, is gradually added over a period of 50 minutes to a solution containing 100 grams of lead acetate, 8 grams of glacial acetic acid, and 750 cc. of water, maintained at 80° C. and well agitated. Until after about one-third of the first solution has been added, the precipitate consists of the black spherical granules characteristic of the normal salt. Following this, the color of the precipitate changes to the brown color of the basic salt. After all the solution has been added, 8 grams of glacial acetic acid are added to the suspension in order to complete the agglomeration of the particles, the addition causing a darkening of the precipitate which is probably due to the superficial conversion of basic to normal salt. The precipitate is then thoroughly washed with water and dried.

From the last described procedure, using the proportions of ingredients specified, a product consisting of approximately one-third normal lead dinitrosoresorcinate and two-thirds basic dinitrosoresorcinate may be obtained. This

product is more sensitive to detonation by shock than the normal salt described in the first illustrative embodiment. The behavior of this material on exposure to a match flame is, however, intermediate that of the normal and basic salts.

While in describing the illustrative embodiments reference has been made to specific proportions and conditions of treatment, it is to be understood that variations therein are contemplated. For instance, as a general rule, the coarseness of the precipitate may be increased by raising the temperature, increasing the time, and supplying more vigorous agitation during the precipitation.

As hereinbefore pointed out, the explosive compounds of nitrosoresorcinol particularly commend themselves for use in explosive compositions on account of the higher order of stability exhibited by these materials even under severe and adverse conditions of storage and treatment. Consequently priming compositions and other explosive mixtures which are formulated with such nitrosoresorcinol compounds as, for instance, the lead salts of polynitrosoresorcinols are particularly advantageous insofar as the stability of the compound is concerned, it being observed in this connection that instability of priming mixtures has heretofore been frequently attributed to instability of fuel ingredients therein.

As illustrating the application of such explosive compounds of nitrosoresorcinol in priming compositions, several illustrative embodiments are set forth in the following tables, it being observed that each table sets forth a range of percentages for each ingredient as well as a specific example in each instance, the figures representing percentages by weight:

	Range	Specific
	Percent	*Percent*
1. Primer for shot shells:		
Mercury fulminate	30–40	40
Lead salt of dinitroso resorcinol	3–15	5
Antimony sulfide	12–25	18
Barium nitrate	20–40	32
Lead dioxide	0–10	5
2. Rim-fire primers:		
a. Normal lead styphnate	35–50	40
Lead salt of dinitrosoresorcinol	3–15	6
Guanylnitrosaminoguanyl tetrazene	1–8	4
Barium nitrate	20–40	29
Ground glass	10–30	20
Gum arabic	1–2	1
b. Mercury fulminate	35–45	40
Lead salt of dinitrosoresorcinol	5–15	11
Barium nitrate	15–35	22
Lead chromate	5–15	11
Ground glass	15–25	15
Gum arabic	1–2	1
c. Lead azide	10–20	14
Lead salt of dinitroso-resorcinol	15–40	30
Guanylnitrosaminoguanyl tetrazene	1–5	3
Barium nitrate	25–55	40.5
Ground glass	5–20	10
Ester gum	1–4	2.5

In any of the mixtures above referred to, either the normal or the basic lead salt of dinitrosoresorcinol, as described hereinbefore, or mixtures thereof may be employed.

While the foregoing tables are given for the purpose of illustrating a representative range of priming mixtures embodying the nitrosoresorcinol compounds of the present invention, it is not to be understood that the present invention, or its application to priming mixtures, is limited to the examples set forth in the foregoing tables. On the contrary, it is to be distinctly understood that the compounds or combinations of compounds having a recognized function in priming mixtures may be substituted and replaced by other known compounds or combinations thereof having the same functions, adjustment being made when necessary in the proportions of the ingredients. While in each of the foregoing examples a lead salt of dinitrosoresorcinal is included as the fuel ingredient in the priming mixtures, it is to be understood that dinitrosoresorcinol compounds may be used either alone or in combination with other known and commonly employed fuels, such, for instance, as antimony sulfide, lead thiocyanate, calcium silicide and the like.

While the invention has been described with particular reference to explosive compositions and particularly in connection with priming and detonating compositions, and while the nitrosoresorcinol compounds of the present invention are particularly useful and advantageous in the explosive field, it is not to be understood that the invention is limited thereto. On the contrary the present invention is to be understood as contemplating, generally, the novel compounds which have been disclosed herein regardless of the use or application which is made of them.

While in the foregoing description reference has been made and a complete disclosure given of several embodiments of the present invention, it is not to be understood that the invention is limited to the precise details of those embodiments. It is apparent that many modifications of the process of manufacture and many variations in the resulting compositions themselves will present themselves to those skilled in the art without departing from the spirit of this invention, and it is to be distinctly understood, therefore, that such modifications and the use of such combinations and subcombinations of features of the present invention as do not depart from the spirit of this invention are, although not specifically described herein, contemplated by and within the scope of the appended claims.

Having thus described the invention, what is claimed is:

1. An explosive comprising a salt of a nitrosoresorcinol.

2. An explosive comprising a heavy metal salt of a nitrosoresorcinol.

3. A priming composition containing a lead salt of dinitrosoresorcinol.

4. A priming composition containing normal lead dinitrosoresorcinate.

5. A priming composition containing basic lead dinitrosoresorcinate.

6. A priming composition containing a mixture of normal and basic lead dinitrosoresorcinate.

7. A manufacture comprising spherical granules of a lead nitrosoresorcinate.

8. A priming composition comprising, an initiating ingredient, a lead salt of dinitrosoresorcinol, and an oxidizing agent.

9. An explosive comprising 2, 4 lead dinitrosoresorcinate.

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