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# EXPLOSIVE COMPOUND, PROCESS OF MAKING SAME AND A COMPOSITION THEREOF

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This invention relates to a new compound suitable for use as an explosive and to the manufacture thereof.

There has been a pressing need in the oil industry for explosives of relatively high heat stability for the purpose of firing missiles through the side walls of well casings far beneath the earth's surface. Numerous types of devices have been developed for this purpose, generally known as oil well gun perforators. Gun powder has been used in such devices but has the limitation of being relatively heat sensitive so that upon being lowered to great depths premature firing and the like has occurred. The temperature in such casings, of course, increases with depth of penetration into the earth and for very deep wells explosives of greater heat stability are required.

It is therefore an object of this invention to provide an explosive suitable for use under such higher temperature conditions. Another object is to provide a novel propellant. Another object of this invention is to provide a novel compound having characteristics making it eminently suitable for an explosive. A still further object of the invention is to provide a novel explosive and a method for the manufacturing thereof. A more specific object is to provide improved ammunition priming compositions as well as other novel explosive compositions.

The foregoing objects as well as others which will become apparent from the following description are accomplished in accordance with this invention, generally speaking, by providing as the new compound, the lead salt of ethylenedinitramine, also referred to herein as lead ethylenedinitramine. The lead salt of ethylenedinitramine has been found to be resistant to higher temperatures than smokeless powder, black powder, nitro-starch, trinitrotoluene, tetryl, ethylenedinitramine and the like. Some of such explosives are unsuitable at the higher temperatures because of their relatively low melting points, others because of their relatively low flash points. Hale in U. S. 2,011,578 discloses that ethylenedinitramine may be used as a substitute for tetryl, picric acid, and trinitrotoluene in booster and high explosive charges but that it explodes spontaneously when heated to 180° C. In contrast it has been found in accordance with this invention that the lead salt of ethylenedinitramine does not spontaneously explode until temperatures of around 300° C. and above are attained and that it is a relatively powerful explosive without admixture with other explosives. It has been found that due to the characteristics of the lead salt of ethylenedinitramine it can be used to advantage in compositions containing other explosives, as will become apparent hereinafter.

In accordance with this invention the lead salt of ethylenedinitramine is prepared by first forming a soluble alkali metal salt of ethylenedinitramine and then replacing the alkali metal with lead to effect a precipitation of the insoluble lead salt. As an example of one embodiment of this invention, about 15 parts ethylenedinitramine are suspended in about 500 parts water at ambient temperature and containing about 16.5 parts sodium hydroxide. The slurry is agitated until substantially all of the ethylenedinitramine has dissolved to form a water soluble sodium salt. The resulting sodium salt is then converted to the lead salt of ethylenedinitramine by adding to the solution about 66 parts lead nitrate dissolved in about 400 parts water at room temperature. The lead salt is insoluble in the solution and is rapidly formed but agitation is continued for a few minutes to insure complete mixing of the two solutions. The suspension is then filtered and the precipitate air dried at room temperature for about sixteen hours. The lead ethylenedinitramine can then be loaded without further treatment or purification into oil well gun perforator devices for use at temperatures as high as 300° C. or thereabouts.

The foregoing process can be modified in many respects without deleteriously affecting the product. For instance, other lead compounds such as lead acetate can be substituted for the lead nitrate and potassium hydroxide can be substituted for the sodium hydroxide, if desired. The method of mixing the various solutions together can be reversed, the reactions can be carried out at elevated temperatures and drying can be accomplished at elevated temperatures below that at which the compound would explode.

This new explosive due to its low impact and friction sensitivity can be handled in the dry state with a reasonable degree of safety while loading, has moderate power or brisance (crushes about 17 grams of sand when initiated in a sand bomb), produces large volumes of gas rather slowly which along with a relatively high flash point are requirements for explosives used in oil well perforator guns. Explosive charges consisting of the lead salt of ethylenedinitramine or in combination with other heat stable propellants are suitable for operations of this type.

In addition to having advantages as a propellant, the compound is useful in ammunition. The ballistic properties of ammunition and particularly improved ignition of the propellant by the primer is obtained with priming mixes which contain the lead salt of ethylenedinitramine. While its relatively low impact sensitivity eliminates it as a substitute for the more impact sensitivity normal lead styphnate, mercury fulminate, diazodinitrophenol and similar explosives utilized in percussion primers, it can be used in combination with such explosives. Because of its low impact sensitivity, a priming mix can contain relatively large percentages thereof as an explosive fuel or sensitizer which when ignited by the more impact sensitive explosive will produce a hot flame, thus improving the ignition characteristics of the primer and improving the ballistics of the cartridge by reducing the number of squibs, hangfires and other malfunctions due to poor ignition. As as example, a priming mix having the following formula has been found to satisfactorily ignite some of the most difficultly ignitable propellent powders:

| | Parts |
|---|---|
| Diazodinitrophenol | 20 |
| Tetracene | 5 |
| Lead ethylenedinitramine | 25 |
| Calcium silicide | 20 |
| Barium nitrate | 24 |
| Lead peroxide | 6 |
| Gum arabic | 0.5 |

Variations can be made in the foregoing priming composition in order to adjust its characteristics for proper functioning with various types of propellants. For example, other oxidizers such as lead nitrate, potassium nitrate and the like are suitable. Non-explosive fuels other than calcium silicide such as antimony sulfide or a metal, for example, zirconium, aluminum and the like, might be advantageous in some priming mixes. Normal lead styphnate and mercury fulminate are examples of

other primary explosives which might be substituted for the diazodinitrophenol and in some instances the explosive sensitizer such as tetracene can be eliminated from the composition.

It should be apparent from the foregoing description that the present invention accomplishes its objects and provides a process for making a novel explosive compound and new explosive compositions having unusual characteristics which are advantageous in several types of operations requiring explosive charges.

While specific embodiments have been given and the invention has been described with particularity in the foregoing, modifications which will occur to those skilled in the art may be made without departing from the spirit and scope of the invention.

Having thus described the invention what is claimed and desired to secure by Letters Patent is:

1. Lead ethylenedinitramine.

2. A process for making lead ethylenedinitramine which comprises the steps of reacting in solution and in molecular proportions, ethylenedinitramine and an alkali metal hydroxide, reacting, in solution and in molecular proportions, the resulting alkali metal salt of ethylenedinitramine and a lead salt, and thereafter separating the resulting precipitate of lead ethylenedinitramine from the reaction liquor.

3. The process of claim 2 wherein said alkali metal hydroxide is sodium hydroxide.

4. The process of claim 2 wherein said lead salt is water soluble.



5. The process of claim 2 wherein said lead salt is lead acetate.

6. A process for making lead ethylenedinitramine which comprises reacting, in solution and in molecular proportions, ethylenedinitramine and sodium hydroxide, reacting, in solution and in molecular proportions, the product of this reaction with lead nitrate, and thereafter separating the resulting precipitate of lead ethylenedinitramine from the reaction liquor.

7. A priming mix having a composition substantially as follows:

| | Parts |
|---|---|
| Diazodinitrophenol | 20 |
| Tetracene | 5 |
| Lead ethylenedinitramine | 25 |
| Calcium silicide | 20 |
| Barium nitrate | 24 |
| Lead peroxide | 6 |
| Gum arabic | 0.5 |


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