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PROCESS FOR PRIMARY EXPLOSIVES CONTAINING BORON HAVING REDUCED ELECTROSTATIC SENSITIVITY

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The invention relates to primary explosive compositions which may be used as initiatory or fast delay explosives in fuses, detonators and the like and which will explode under mild stimulus with sufficient violence to produce a detonation wave capable of exploding quantities of less sensitive explosive. Primary explosives according to the invention are metal azides and metal salts of nitro-substituted resorcinols.

In order to be of practical value, a primary explosive composition must be able to fulfil a number of exacting requirements. In particular, although it must be capable of being exploded consistently, when required, by a small pulse of energy (from a small electric current or mechanical impact, for example), a primary explosive composition must be reasonably safe and not easily exploded during manufacture and subsequent handling.

Although there is a substantial hazard of mechanical shock causing accidental detonation of primary explosives during their manufacture and handling, many accidents are caused by accidental ignition by electrostatic spark, to which many primary explosives especially various normal and basic salts of nitro-substituted resorcinols, are very sensitive. Copper acetylide, for example, although capable of functioning satisfactorily as an initiatory explosive, has never been developed because its prohibitively low energy for electrostatic ignition of a few ergs renders it virtually impossible to handle in manufacture.

The present invention provides a process for the manufacture of primary explosive compositions in a modified form which have reduced sensitivity to electrostatic ignition thus enabling various primary explosives which have an electrostatic hazard to be manufactured and handled in greater safety.

The present invention is thus particularly applicable to primary explosives which are very sensitive to ignition by electrostatic sparks of very low energies such as, for example, various normal and basic metal salts of nitro-substituted resorcinols.

In a process for producing a primary explosive composition in accordance with the invention, crystals of a primary explosive are formed in a liquid medium in the presence of a suspension of finely-divided boron to form crystals having an increased resistance to electrostatic ignition containing boron.

An important advantage of the present process is that the handling of sensitive dry primary explosive is avoided and the primary explosive composition is initially obtained in the dry state with reduced electrostatic sensitivity and with the boron more fully dispersed throughout the crystal granules than is readily possible with mechanical mixing.

In order to achieve a good interspersion of the boron particles with those of the primary explosive, the boron particles should be finely divided so that they are appreciably finer than the primary explosive particles. A suitable mean boron particle size (i.e., diameter of particles) is between about 1–10 microns as primary explosive granules generally have a mean size of the order of 50 microns.

The presence of boron in the primary explosive composition can provide important advantages besides the reduction of electrostatic hazard which can make the incorporation of boron in the particles very advantageous, even if the electrostatic hazard without boron is insufficient for its reduction to be vital. Thus the igniting power of certain primary explosives, such as barium styphnate, to ignite other explosives can be increased advantageously by the incorporation of boron although there is no real electrostatic hazard.

For other primary explosives, such as normal lead 2:4-dinitroresorcinate and lead styphnate, the presence of boron can advantageously reduce the electrostatic hazard and also increase the stab and percussion sensitivity to a desirable extent. The presence of boron also advantageously increases the ability of these primary explosives to ignite the next element in the firing train as the boron produces hot particles.

The boron may be incorporated in the primary explosive particles in proportions as desired, the electrostatic sensitivity decreasing progressively with increasing additions generally up to about 20%.

The formation of crystalline particles of a primary explosive composition in accordance with the invention is conveniently carried out in a liquid medium by precipitating the primary explosive by a double decomposition reaction.

A typical example of the production of a primary explosive composition in accordance with the invention wherein the primary explosive is precipitated by a double-decomposition reaction between two reactant aqueous solutions A and B is as follows.

Solution A is added to the stirred solution B which contains a suspension of finely-divided boron. The primary explosive composition precipitates from the mixed solutions as crystalline particles which incorporate the boron dispersed in the individual particles. Substantially all the boron in suspension is incorporated by this procedure and the separated, dried primary explosive composition has a greatly reduced sensitivity to electrostatic ignition as compared with the primary explosive prepared similarly by double decomposition without the incorporation of boron.

Three examples of processes for producing primary explosive compositions in accordance with the invention will now be described.

Example 1

50 mls. of an aqueous solution of lead nitrate (260 g. $Pb.(NO_3)_2$ per litre) is added with stirring over a period of eight minutes to 80 mls. of an aqueous solution of magnesium styphnate (150 g. styphnic acid per litre) in which is suspended 2.1 g. of finely-divided boron (90–92% pure) having an average particle size of 4.0 microns. The temperature of the magnesium styphnate/boron solution is maintained at 75° C. during the addition of the lead nitrate solution, and the precipitated lead styphnate is washed by decantation twice with 120 mls. distilled water and twice with acetone. After drying at 50°, 21 g. of free-flowing product are obtained substantially free of unincorporated boron. Normal lead styphnate prepared in this way contains 10% by wt. of boron and has a threshold energy for ignition by electrostatic spark of about 5,000 ergs. The threshold energy for normal lead styphnate prepared in a known manner from lead nitrate and magnesium styphnate without the incorporation of boron or other additives is about 80 ergs.

Example 2

22.4 mls. aqueous lead acetate solution (containing 7.05 g. $Pb(Ac)_2.3H_2O$) are added dropwise over a period of twenty minutes to 80 mls. of a stirred aqueous solution of sodium 2:4-dinitroresorcinate (equivalent to 4 g. 2:4-dinitroresorcinol and 0.72 g. sodium hydroxide per litre) in which is suspended 0.67 g. of finely-divided boron having a mean particle size of about 4 microns.

The temperature of the sodium 2:4-dinitroresorcinate/boron solution is maintained at 65° C. during the addition of the lead acetate solution, and the precipitated product is washed twice with 120 mls. distilled water and then twice with acetone or methanol. After drying at 45° C., 6.5 g. of granular, free-flowing normal lead 2:4-dinitroresorcinate are obtained substantially free from unincorporated boron. The product contains 10% boron by weight and has a threshold energy for electrostatic ignition of about 5,500 ergs as compared with the threshold energy of 160 ergs for normal lead 2:4-dinitroresorcinate prepared in a similar manner but without the incorporation of boron.

Example 3

3 litres aqueous magnesium styphnate solution (containing 164 g. styphnic acid and 2.2 g. sodium carboxymethyl cellulose per litre) are added over a period of 15 minutes to two litres of a stirred aqueous solution of barium chloride (244.3 g. $BaCl_2.2H_2O$ per litre) in which 80 g. finely-divided boron (mean particle size about 4 microns) are suspended, the temperature of the barium chloride solution being maintained at 75° C. during the addition. The precipitated barium styphnate is washed with water by decantation and, after drying at 50° C., 750 g. of free-flowing barium styphnate containing 6% by weight boron is obtained substantially free of unincorporated boron. This product has a threshold energy for electrostatic ignition of about 600,000 ergs, whereas the threshold energy for barium styphnate prepared in a similar manner without the incorporation of boron is about 25,000 ergs.

The increase in the resistance of primary explosives to electrostatic ignition resulting from the incorporation of various percentages of boron is illustrated by the following results.

Primary explosive	Boron incorporated (percent by weight)	Electrostatic Energy for Ignition (ergs)
Normal lead styphnate	0	80 (C)
	½	130 (C)
	1	140 (C)
	2½	300 (C)
	5	1,500 (P)
	[1] 8.5	500 (P)
	10	5,000 (P)
Basic lead styphnate	0	15
	10	500 (P)
	12½	900 (P)
Normal lead 2:4-dinitroresorcinate	0	160 (C)
	10	5,500 (P)
Normal lead trinitrophloroglucinate	0	165 (C)
	7	900 (P)
Barium styphnate monohydrate	0	25,000
	6	600,000

[1] Mechanical mixing.

Except where indicated, boron is incorporated from suspension in a liquid in the manner described in the preparative examples. (C) and (P) indicate respectively whether complete or partial ignition of the explosive composition occurs at threshold ignition energies, partial ignition being a case where at least a third, but not all, of the composition ignites. The results for normal lead styphnate demonstrate the superiority of the described processes for incorporating boron from a suspension in a liquid over a mechanical mixing of the primary explosive and boron.

What we claim is:

1. A process for producing a primary explosive composition which comprises forming the primary explosive in a liquid medium in the presence of a suspension of finely divided boron to give crystalline particles of primary explosive individually containing boron having an increased resistance to electrostatic ignition and separating said crystalline particles from the liquid medium.

2. A process as in claim 1 wherein the said primary explosive is a compound selected from the group consisting of normal and basic salts of a nitro substituted resorcinol.

3. A process as in claim 1 wherein the said crystalline particles of the primary explosive are formed in a liquid medium by a double decomposition reaction.

4. A process as in claim 1 wherein said primary explosive is a compound selected from the group consisting of normal lead styphnate, basic lead styphnate, normal lead 2,4-dinitroresorcinate and barium styphnate.

5. A process as in claim 1 wherein the boron is present in the liquid medium in a proportion of up to about 20% by weight of the theoretical yield of the primary explosive and wherein the primary explosive is formed in the liquid medium by a double decomposition reaction.

6. In a process for producing a primary explosive composition the steps comprising precipitating a primary explosive from a liquid medium by a double decomposition reaction in the presence of a suspension of boron having a mean particle size of up to 10 microns, the boron being present in the liquid medium in the proportion of up to 20% by weight of the product primary explosive; separating the primary explosive and drying it to form crystalline particles of primary explosive containing finely-divided boron.

7. In a process for producing a primary explosive composition the steps comprising precipitating a primary explosive selected from the group consisting of normal lead styphnate, basic lead styphnate, normal lead 2,4-dinitroresorcinate and barium styphnate from a liquid medium in the presence of a suspension of boron having a mean particle size of up to 10 microns, the boron being present in the liquid medium in the proportion of up to 20% by weight of the product primary explosive; separating the said primary explosive and drying it to form crystalline particles of said primary explosive containing up to 20% by weight boron.

8. In a process for producing a normal lead styphnate primary explosive composition the steps comprising reacting lead nitrate and magnesium styphnate in about stoichiometric amounts in an aqueous solution containing a suspension of finely divided boron having an average particle size of about 0.9 micron in the proportion of about 16 grams per liter; separating and drying the resulting lead styphnate to form crystalline particles of lead styphnate containing about 10% by weight boron.

9. In a process for producing a normal lead 2,4-dinitroresorcinate primary explosive composition the steps comprising reacting in about stoichiometric amounts an aqueous solution of lead acetate and an aqueous solution of sodium 2,4-dinitroresorcinate containing a suspension of finely divided boron having an average particle size of about 4 microns in the proportion of about 8.3 grams per liter; separating and drying the resulting normal lead 2,4-dinitroresorcinate to form crystalline particles of normal lead 2,4-dinitroresorcinate containing about 10% by weight boron.

10. In a process for producing a barium styphnate primary explosive composition the steps comprising reacting in about stoichiometric amounts an aqueous solution of magnesium styphnate and an aqueous solution of barium chloride containing a suspension of finely divided boron having an average particle size of about 4 microns in the proportion of about 40 grams per liter; separating and drying the resulting barium styphnate to form crystalline particles of barium styphnate containing about 6% by weight boron.

11. A primary explosive composition comprising crystalline particles of a primary explosive containing dis-

persed within said particles up to about 20% by weight of boron particles having a mean particle size of up to 10 microns.

12. A primary explosive composition comprising crystalline particles of a compound selected from the group consisting of normal and basic lead styphnate, normal lead 2,4-dinitroresorcinate, and barium styphnate, containing dispersed within said particles up to about 20% by weight of boron particles having a mean particle size of up to 10 microns.

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