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## PROCESS OF HANDLING SENSITIVE MATERIALS

Frederick R. Seavey and Edward B. W. Kerone,  
Alton, Ill., assignors to Western Cartridge Com-  
pany, East Alton, Ill., a corporation of Dela-  
ware

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This invention relates generally to the handling of sensitive materials and particularly to a process of handling lead azide in the formation of primer compositions for ammunition, or initiating charges for detonators.

Although lead azide has gone into quite general use as an initiator in primer compositions, severe accidents have not infrequently occurred during the mixing and charging steps in the preparation of primer compositions containing lead azide. Although the sensitivity of lead azide has generally been regarded as lower than the sensitivity of mercury fulminate to such shock and friction as is liable to occur during the mixing and charging procedure, this is probably due to the fact that the lead azide is generally reduced to a crystal size so fine that it approaches the size of an impalpable powder such, for instance, as will pass through a 200 mesh screen, while mercury fulminate is generally of a much larger size. It is recognized that the sensitivity of lead azide is particularly dependent upon the crystal size, the larger the crystals the greater the sensitivity, and accordingly it is the usual practice in the handling of lead azide to employ the fine powder which may be obtained by precipitation so that the sensitivity of the composition during the mixing and charging procedure is as low as possible. Although the number of accidents which are occasioned from the premature ignition or explosion of lead azide is not so great with the use of finely divided crystals as would probably occur if the crystals were of a larger size such as for instance, the usual mercury fulminate size, it is nevertheless true that once lead azide is ignited, the rapidity with which the maximum rate of detonation is achieved is much greater than that of mercury fulminate, and accordingly the ignition of a mixture containing lead azide will usually be found to produce strong shattering effects which are much more violent than those obtained with a similar composition containing mercury fulminate. As a consequence if a priming mixture, for instance, is permitted to become too dry during charging or mixing operation, slight friction may produce violent detonation.

For this reason, it is necessary to accurately control the moisture content of the mixture during the mixing and charging operations, but it is nevertheless difficult to accurately control this content since the tendency is for the moisture to evaporate. The rapidity of such an evaporation, of course, depends upon the temperature and humidity of the atmosphere.

The object of this invention, generally stated, is to provide a process of handling such sensitive materials as should be maintained moist, in which the tendency for the moistening medium to evaporate is substantially avoided.

A further object of the present invention is to provide a process of handling sensitive materials in which the usual water content of a paste is replaced by a liquid having a substantially lower volatility than water.

A further object of this invention is to provide a process of handling lead azide in which the sensitivity thereof is substantially reduced and liability to premature detonation is avoided.

A more specific object of this invention is to provide a process of handling lead azide in which the sensitivity thereof is substantially reduced and liability to detonation is avoided throughout the procedural steps involved in the preparation and charging of priming compositions subsequent to the precipitation and purification of the lead azide up to and including the first stages of drying at which time the azide has been divided into many small charges—primers for example.

Other objects will become apparent to those skilled in the art when the following description of one illustrative embodiment is read. It is to be understood, however, that the invention is not limited to the particular embodiment referred to hereinafter for the purpose of illustration, but that the principles of this invention are susceptible of use in the process of handling sensitive materials generally.

Generally stated, in accordance with the present invention, which is adapted particularly for use in the handling of material such as lead azide which must be maintained moist at all times during the handling thereof, such as in mixing and charging operations, in order to prevent premature detonation thereof, the tendency or possibility of the moistening medium such as water, which has heretofore been employed, to evaporate during the course of the procedure and thus render the material liable to detonation upon the occurrence of slight friction, flame, sympathetic detonation or the like is overcome by the use of a moistening medium which is not so readily volatile as is water. Such moistening media are selected must, however, be capable of complete removal during the subsequent drying operations such, for instance, as are generally followed after the deposit of a priming composition in position within its case. The low volatile medium selected is preferably non-inflammable and when, as in accordance with one

embodiment of this invention it is desired to employ a binding agent in the primer composition, the particular low volatile moistening medium selected should be one which is operative as a solvent for the binding material.

As illustrative of a typical procedure which may be practiced in handling such sensitive materials as lead azide, such as in the mixing and charging of a priming composition, the lead azide which may in accordance with the usual practice be obtained by precipitation may be first screened, preferably through a silk bolting cloth having a fineness corresponding approximately to a 200 mesh screen. Two duplicate screens of this type are preferably employed, one being placed in such a position as to receive the screened material from the other in order to avoid the possibility of crystals above the desired size being present in the screened material. When two duplicate screens are thus employed, it is apparent that the possibility of distortion in the weave of the two screens occurring simultaneously is substantially remote and accordingly when crystals above the desired size appear on the second screen it will indicate that a distortion of the weave in the first screen has occurred.

After the screening of the lead azide in order to remove the larger crystals having a relatively high sensitivity is accomplished, as just described, the water content of the lead azide paste is preferably then replaced by a liquid medium of appreciably lower volatility than water. The volatility of the liquid employed is preferably such as to preclude the possibility of the lead azide becoming dry under the atmospheric conditions which will be encountered during the mixing and charging operations. In order to accomplish this replacement of the water by the low volatile medium, the lead azide-water mixture may be first filtered and the lead azide collected on a suitable filter medium such, for instance, as a lintless filter paper. The lead azide crystals thus collected may then be washed with a medium capable of removing the water content, such as for instance, any suitable anhydrous medium, for example anhydrous methanol. The methanol may then be readily replaced by applying a liquid medium which is soluble in methanol. In accordance with the present invention the medium which is selected for replacing the methanol is one having a comparatively low volatility as compared to water and when, as in the case of priming mixtures, it is desired that the resultant composition be non-hygroscopic, it is of importance to select a low volatile medium which is not soluble in water. A suitable low volatile liquid which fulfills all these requirements and which, in addition is substantially non-inflammable, is dichlorethyl ether.

A suitable binding agent, preferably one which operates as a deterrent and thus decreases the sensitivity of the lead azide crystals which may be mixed therewith, may be incorporated with the priming mixture at any time during the mixing procedure. Such a binding agent as for instance, ester gum is particularly adapted for use in this connection, as described and claimed in our co-pending application, 598,530, filed March 12, 1932.

When dichlorethyl ether is employed as the low volatile medium, the process of this invention is particularly adapted for controlling priming mixtures, containing ester gum as a binding material, since the binding material, ester gum, is

readily soluble in dichlorethyl ether and accordingly the dichlorethyl ether or other low volatile medium may be employed as a vehicle for carrying the binding material during incorporation with the other sensitive ingredients, such as lead azide or the priming composition. During the subsequent drying stages the dichlorethyl ether will eventually all volatilize and a relatively hard rigid mass tenaciously adhering to the walls of the metallic case will result due to the action of such a binding agent as cellulose acetate.

The foregoing description is illustrative of an embodiment in which the presence of water is completely eliminated. With such an anhydrous method waterproofing of the resultant mix is facilitated. It is apparent, however, that the water content in whole or in part may be replaced directly with the low volatile medium, such as dichlorethyl ether.

From the foregoing description, it is apparent that a course of procedure has been described by which the liability to premature detonation of such materials as lead azide during mixing and charging operations is substantially lessened, particularly since the tendency of the moistening medium to evaporate is in accordance with the present invention substantially avoided. Furthermore the process of the present invention is particularly adapted for use in the treatment of a priming composition containing a deterrent binder operating to decrease to some extent the sensitivity of the mixture and consequently to reduce the liability of accidents resulting from premature detonation of the sensitive material employed is substantially avoided. It is to be understood, however, that the replacement of the water content of the material by a low volatile liquid may be accomplished either directly or indirectly by other methods than that hereinbefore specifically referred to for the purpose of illustration.

From the foregoing description, it is apparent that many modifications of the process hereinbefore described 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 the invention is neither limited to the specific details nor to use with the particular materials hereinbefore referred to for the purpose of illustration, but that such modifications and the use of such individual features and subcombinations of features as do not depart from the spirit of this invention are contemplated by and within the scope of the appended claims.

Having thus described the invention, what is claimed is:

1. In the art of making explosives, the process of handling normally moist highly sensitive materials, comprising, wetting the materials with a substantially non-inflammable liquid which is non-solvent for the highly sensitive material and which has a lower volatility than water but is capable of complete removal by drying.

2. In the art of making explosives, the process of handling highly sensitive materials, comprising, wetting the materials with a substantially non-inflammable liquid which is non-solvent for the highly sensitive material and which has a lower volatility than water but is capable of complete removal by drying and which is insoluble in water.

3. In the art of making explosives, the process of handling sensitive materials, comprising, wetting the materials with a substantially non-in-

flammable water-repellant liquid which is non-solvent for the highly sensitive material and which has a lower volatility than water but is capable of complete removal by drying.

4. In the art of making ammunition, the process of handling sensitive water wet materials, comprising, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a substantially non-inflammable liquid having a lower volatility than water.
5. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a substantially non-inflammable low volatile liquid soluble therein.
6. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a substantially non-inflammable low volatile liquid soluble therein but insoluble in water.
7. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a binder solution the solvent of which is of low volatility.
8. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a water repellent binder solution the solvent of which is of low volatility.
9. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the moisture with a dehydrating liquid, and displacing the dehydrating liquid with a low volatile non-inflammable liquid.
10. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a low volatile liquid medium containing a binder having deterrent properties.
11. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the moisture with a dehydrating liquid, and displacing the dehydrating liquid with a low volatile non-inflammable liquid medium containing a binder having deterrent properties.
12. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with anhydrous methanol, and displacing the methanol with dichlorethyl ether.
13. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with anhydrous methanol, and displacing the methanol with dichlorethyl ether containing a binding substance.
14. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, displacing the water with an-

hydrous methanol, and displacing the methanol with dichlorethyl ether containing a binding substance having deterrent properties.

15. In the art of making ammunition, the process of handling sensitive materials, comprising wetting the material, displacing the water with a dehydrating liquid, and displacing the dehydrating liquid with a low volatile non-inflammable desensitizing liquid medium containing a binder having deterrent properties.

16. In the art of making ammunition, the process of handling sensitive materials, comprising, wetting the material, displacing the moisture with a dehydrating liquid, and displacing the dehydrating liquid with a low volatile non-inflammable liquid medium containing a binder having deterrent and desensitizing properties.

17. In the art of making ammunition, the process of handling sensitive materials, comprising, wetting the material, displacing the moisture with a dehydrating liquid, and displacing the dehydrating liquid with a low volatile non-inflammable desensitizing liquid medium containing a binder having deterrent and desensitizing properties.

18. In the art of making ammunition the process of handling sensitive materials containing water, comprising, replacing the water with dichlorethyl ether.

19. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, replacing the water with dichlorethyl ether containing a binding substance.

20. In the art of making ammunition, the process of handling sensitive materials containing water, comprising, replacing the water with dichlorethyl ether containing a binding substance having deterrent properties.

21. In the art of making ammunition, the process of handling sensitive materials which are normally maintained moist comprising, desensitizing the material by wetting with a non-solvent non-inflammable liquid of lower volatility than water, and removing the liquid after handling.

22. In the art of making and charging priming mixtures for ammunition, the process comprising, wetting the normally moist highly sensitive ingredient with a non-solvent desensitizing liquid of lower volatility than water, charging the mixture into shells, and thereafter removing the desensitizing liquid.

23. In the art of handling sensitive materials containing water, the process comprising, wetting the material with a liquid having a lower volatility than water.

24. In the art of handling sensitive materials containing water, the process comprising, wetting the material with dichlorethyl ether.

25. A manufacture comprising lead azide wetted with dichlorethyl ether.

26. A manufacture comprising a highly sensitive explosive wetted with dichlorethyl ether.

27. A manufacture comprising lead azide wetted with a non-inflammable liquid of lower volatility than water.

FREDERICK R. SEAVEY.  
EDWARD B. W. KERONE.