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EXPLOSIVE DEVICE CONTAINING CHARGE OF ELONGATED  
CRYSTALS AND AN EXPLODING BRIDGEWIRE  
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FIG-1

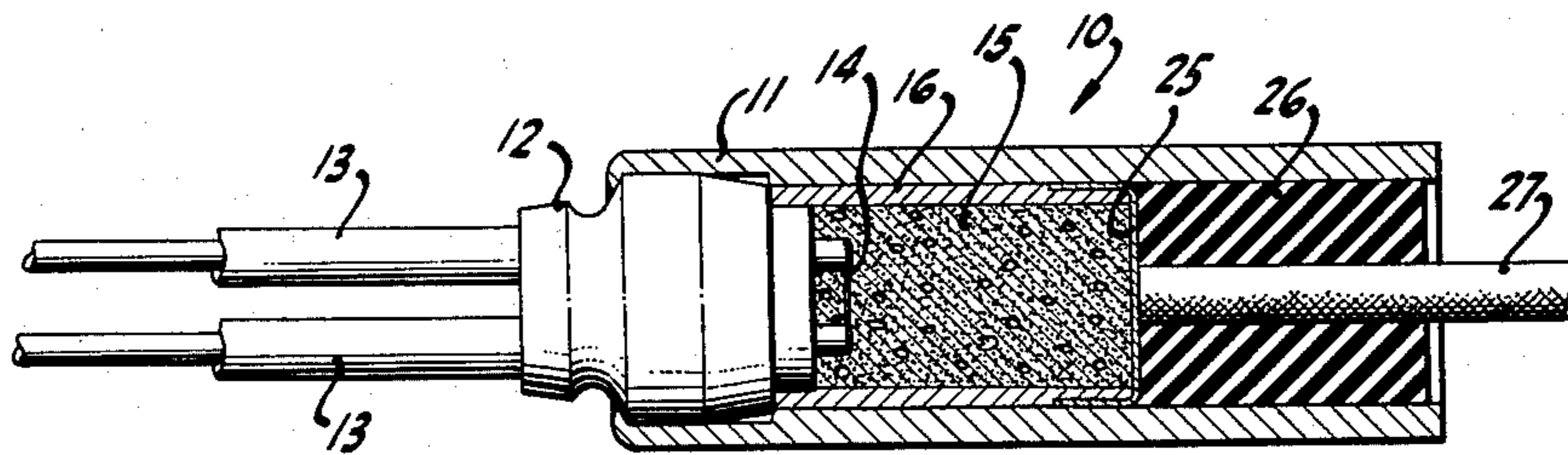
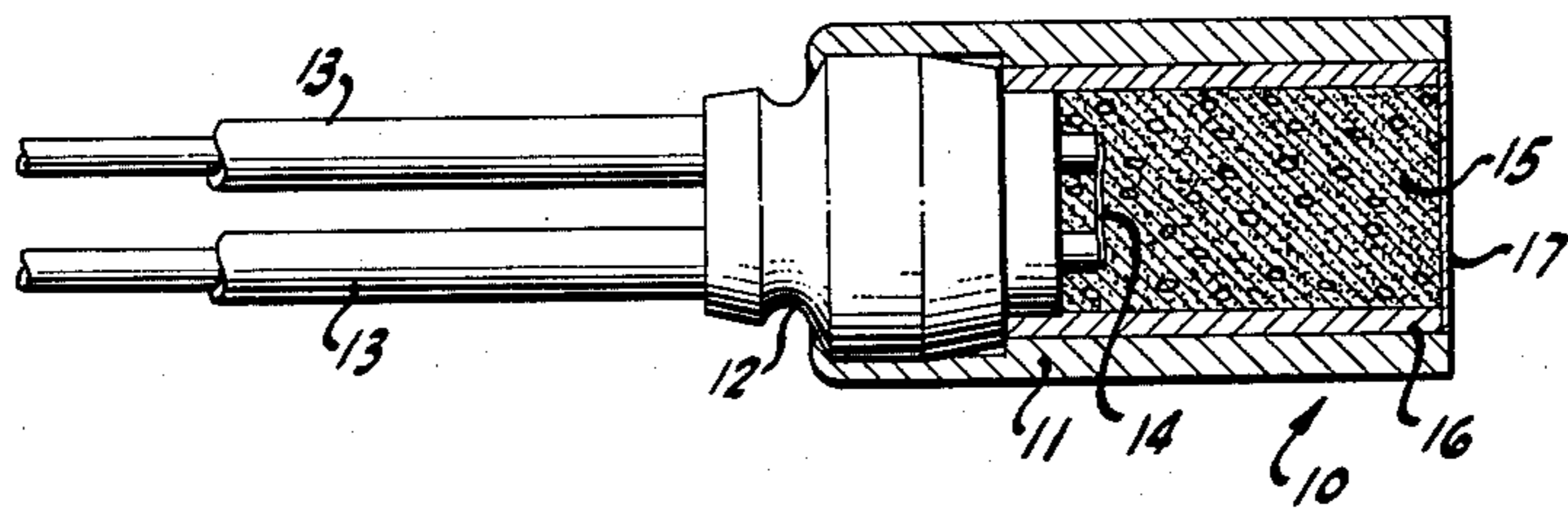


FIG-2

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**EXPLOSIVE DEVICE CONTAINING CHARGE OF ELONGATED CRYSTALS AND AN EXPLODING BRIDGEWIRE**

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6 Claims. (Cl. 102-28)

This invention relates to explosives and propellants. More particularly, this invention relates to a method of producing explosive and propellant compositions in a more desirable form; to the end products of such method; and to explosive or propellant devices employing the same.

In the manufacture of detonators (such as used to initiate a propellant or an explosive, or to initiate an explosive cord or cordeau) it is a common practice to provide a body of explosive material such as PETN (which is pentaerythritol tetranitrate) encased in a suitable housing, and to initiate the explosive material in the detonators by means of an exploding bridgewire. This practice avoids the use of highly sensitive primary explosives such as fulminates, lead azide and lead styphnate. As is well known in the explosive art an exploding bridgewire unit comprises a short length of wire of small diameter and a power source which stores electrical energy and releases it upon demand in the form of a short pulse at a high voltage. This results in vaporization of the wire, explosion of the metal vapor, the release of a large amount of heat and the creation of a shock wave which together detonate the explosive.

PETN is useful for this purpose but, when it is used in this manner and is initiated by an exploding bridgewire (hereinafter referred to as an "EBW"), it is important that the PETN be insensitive to stray currents which may heat (but not explode) the bridgewire, yet remain sensitive to an EBW.

It is known that the sensitivity of PETN is related to its density. It is desirable to pack PETN in a casing or housing at a relatively high density in order to obtain a greater energy output and to maintain a uniform, intimate contact of the bridgewire with the PETN.

If PETN is packed loosely within a detonator, it may separate from the wire during handling or storage with resultant failure to initiate. On the other hand, as the density increases it reaches a point at which the detonator fails to function. This presents the manufacturer with the problem of working between relatively close lower and upper limits of density. This requires much greater production control and it results in a greater proportion of rejects or of products which may pass inspection but fail in service.

It is an object of the present invention to provide improvements in explosive and propellant materials.

It is a further object of the invention to provide PETN and other explosives in a more desirable physical form than has been available heretofore.

It is a particular object of the invention to provide a means of treating PETN, and other explosives which are susceptible to initiation by an EBW, whereby the explosive material can be packed to a higher density without failure to perform.

The above and other objects of the invention will be

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apparent from the ensuing description and the appended claims.

FIG. 1 illustrates a first embodiment of the invention.

FIG. 2 illustrates a further embodiment of the invention primarily adapted for use as an initiator for an explosive fuse.

We have discovered that when PETN and other explosive or propellant materials are employed in the form of elongated, e.g. needle-like or rod-shaped crystals of small size (rather than in the usual amorphous, globular or spherical form as produced by mechanical grinding), the explosive has substantially superior properties.

For example, in the case of PETN, if it is of suitably small particle size and if the particles are needle-like crystals rather than spheres or globules, the material is much less sensitive to density and can be packed more tightly. Also, PETN in this form can be packed into a mass having greater structural strength than the previously available product, and it presents a larger surface area per unit of weight than previously available.

These and other properties are among advantages of the needle- or rod-like crystalline shape when the explosive is PETN. Some or all of these advantages exist with other explosives if they have a similar particle structure, e.g., RDX, HMX, TNT and even inorganic explosives such as ammonium nitrate. (RDX is cyclotrimethylene trinitramine, HMX is cyclotetramethylene tetranitramine, and TNT is trinitrotoluene.) It will be understood that the advantages of the crystalline form differ somewhat from one explosive to another, particularly when the type of explosive differs radically. For example, with PETN the crystalline form of the invention has, as an important advantage, the fact that the material can be packed within a relatively wide range of density, yet it remains susceptible to initiation by an exploding bridgewire. With TNT this advantage is absent because TNT is insensitive to an exploding bridgewire regardless of density. However, the crystalline form of the invention is advantageous in the case of TNT when mixed with an oxidizer such as  $\text{NH}_4\text{NO}_3$  or  $\text{KClO}_4$ . Such mixtures can be initiated by an EBW at energy inputs of 3000 volts from a 12 micro farad source, giving rise to low order detonations. Also, ammonium nitrate is benefitted by the crystalline structure of the invention because of the resulting small particle size; for example, it produces better ammonal explosives with powdered aluminum. In general, any explosive or propellant material is benefitted by the crystalline form of the invention because, for a given particle size, the material presents a greater surface area per unit of weight and because packed bodies of the material have greater structural strength.

The explosive material of the present invention preferably has a small particle size, e.g., a length (L) of about 30 to 200 microns and a width (W) of about 5 to 20 microns.

We have also discovered an advantageous method of producing the crystalline particles of the invention, such method being as follows:

The explosive or propellant material is dissolved in a solution in which it has a substantial solubility. Preferably, the amount dissolved is substantially less than the saturation concentration at the temperature involved, preferably not more than about 1 to 5% of the saturation concentration. Then a nonsolvent or precipitant is added which is miscible with the solvent but in which the solute

is insoluble or in which the solute has a relatively low solubility. This addition of nonsolvent to solution is continued, preferably slowly and with constant agitation, until the solute begins to precipitate and until the desired or optimum yield of precipitate has been obtained.

By this means—i.e., by gradually reducing the solubility of the solute in a solvent-nonsolvent system—we are able to produce an explosive or propellant material which is in the form of small rod-like or needle-like crystals.

We have also discovered that the process and product are greatly benefitted by conducting the precipitation in the presence of a surfactant which is dissolved or dispersed in the solvent-nonsolvent system.

Therefore, in the preferred form of our invention we dissolve the explosive or propellant material in a suitable solvent, but only to a concentration which is a small fraction of the saturation concentration; we also incorporate in the solution a surfactant; and we then add a nonsolvent slowly and with agitation to produce a crop of small needle-like or rod-like crystals.

The following specific examples will serve further to illustrate the practice and advantages of the invention:

#### EXAMPLE 1

12.5 grams of PETN were dissolved in 3,125 ml. of reagent grade acetone at 72° F. with stirring and 25 ml. of Tergitol Non-ionic NPX were added and dissolved with stirring (Tergitol Non-ionic NPX is the trademark of Carbide and Carbon Chemicals Corp. of New York, New York, for a nonyl phenyl ether of polyethylene glycol). Then 6,000 ml. of distilled water were added with constant stirring at the rate of 250–300 cc. per minute, the temperature remaining 72° F. After about 5,000 ml. had been added, PETN commenced to precipitate as shown by the cloudy appearance of the solution. Meanwhile the temperature of the solution rose to about 82–84° F. After 6,000 ml. of water had been added an additional volume of water was added equal to that in the vessel. Such additional increment was added at the rate of 1,000 ml. per minute. Stirring was continued meanwhile and for 15 minutes thereafter to complete the precipitation of the PETN, which was separated by filtration and was washed repeatedly with small volumes (100 mls.) of distilled water. The washed PETN was air dried under suction for 15 minutes and then dried in an oven at 70° C. for 24 hours.

The PETN as received prior to dissolving and precipitation as described in Example 1, was in the form of globules of irregular shape. As precipitated by the process of Example 1 this same material was in the form of needle-like crystals. The "as received" PETN was minus 325 mesh and had a typical particle size of 15 microns minimum, 60 microns maximum. The recrystallized product of the invention, by way of contrast, has typically a maximum dimension (length of the needle) of about 40 to well over 100 microns and a width of about 5–15 microns. Thus, notwithstanding the fact that the average particles of the improved product of the invention are larger in volume, the cross sectional area of the needles is smaller than the minimum cross sectional area of the "as received" PETN. Accordingly, the product of the invention has a smaller mesh size than the "as received" PETN.

As a measure of sensitivity or adaptability to an exploding bridgewire detonator, a test structure of the character shown in FIGURE 1 was constructed. This test detonator is generally indicated by the reference numeral 10. It has an aluminum case 11 and a header 12 of known construction. Insulated wires 13 are provided whose inner ends are connected to a bridgewire 14, which is of small diameter and whose composition and construction are well known. The other ends of the wires 13 are connected to a suitable power supply which is capable, upon being triggered, of creating a very high, instantaneous voltage drop across, and a very high surge of current through the bridgewire 14, whereby the latter is exploded

to create intense heat and a shock wave. Suitable circuits employing banks of condensers are well known and require no description herein.

Within the casing 11 is a body of explosive (e.g., PETN as produced by the present invention) and between this body of explosive and the casing 11, there is a steel sleeve 16. A tape or other sealing closure which is readily ruptured is applied to the outer end (i.e., to the right hand end as viewed in FIGURE 1) of the structure.

Referring now to FIGURE 2 in which like reference numerals indicate like parts, the casing 11 is longer, the outer (right-hand) end of sleeve 16 is closed by an aluminum cap 25, a body of rubber 26 is molded into the outer (right-hand) extension of casing 11, and an explosive cord such as a Primacord 27 is embedded within the rubber 26 and is in contact with the cup 25. Such an article is a commercial detonator having wide utility, e.g., as an initiator for an explosive fuse or cord, to initiate or ignite mix in a rocket motor, etc.

Test detonators constructed as in FIGURE 1 were used in steel dent tests. In each test the taped end of the detonator was placed on a 5/8" steel plate with no external confinement and the detonator was fired (or an attempt was made to fire it) with a pulse of current from a 2,000 volt, 1 mfd. source. Conditions were identical in each case except that the loading density and the character of the PETN were varied from test to test. In one series of tests, globular minus 325 mesh PETN as purchased was loaded into the detonator at densities of 0.8, 0.9, 1.0, 1.1 and 1.2 grams per cc. In another series of tests the PETN was prepared from the same lot of purchased PETN but was processed in accordance with the present invention and had, accordingly, the crystal structure and particle size described hereinabove. This processed PETN was loaded at densities of 0.8, 0.9, 1.0, 1.1, 1.18, 1.2, 1.27 and 1.32 grams per cc. In the first series of tests with "as received" PETN the detonators exploded and dents were made in the steel plates up to a density of 1.1. But at a density of 1.2 the detonator would not explode. In the second series of tests, with the same PETN processed in accordance with the invention, the detonators exploded and dents were made in the steel plate up to and including a density of 1.27, failure occurring with a density of 1.32.

Therefore, it will be apparent that a considerably greater packing density (1.27–1.1—an increment of 0.17 gram per cc.) is possible with the PETN of this invention. This is important because higher packing densities are desirable. Among other things, at higher densities more explosive and more energy are available. Also, the more tightly the explosive is packed the less likely it is to separate from the bridgewire during handling and storage. Also, the present invention permits a wider range of operable densities, which means that density control is more easily achieved. If, for example, a piston is used to pack the explosive in a detonator it may not apply precisely the same pressure during each loading cycle. Moreover the explosive itself and other factors will offer more or less resistance to compaction. The higher packing densities made possible by the present invention, and the broader operating range made possible thereby greatly facilitate production control.

Detonators of the type shown in FIGURE 1, loaded with PETN of the present invention and having a density of 0.9 gram per cc., were subjected to and passed a series of safety tests as follows:

(1) Passage of current through the bridgewire at 500 volts D.C. and 1.0 microfarad. Sixteen detonators were tested, none were initiated and there were no outward physical changes. All the bridgewires fused.

(2) 37 volts D.C. and 0.1 W impedance. Sixteen tests produced no initiation of physical change.

(3) A current of 1.5 amperes (calculated bridgewire temperature of 245° C.) and a current of 1.62 amperes (calculated bridgewire temperature of 299° C.). Many tests were made under these conditions and none of them

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produced initiation. All bridgewires were intact after the tests. Ten of the detonators were then tested at 2,000 volts and 1.0 microfarad but no initiation occurred.

(4) Nine detonators were subjected to 5 milliamp. current through the bridgewire for 10 minutes. No initiation occurred. All but one of these units were then successfully fired with 2,000 volts and 1 microfarad.

### EXAMPLE 2

RDX was processed by the following procedure:

5.5 grams of RDX were dissolved in 1,000 ml. of acetone and 6 ml. of Tergitol NPX (the same surfactant as used in Example 1) were also dissolved in the acetone solution. The solution was filtered and the filter paper was washed with 500 ml. of acetone to bring the total volume of acetone to 1,500 ml. Then the acetone solution was aspirated into a stream of tap water at the rate of 1 gallon per minute of water with a pickup of 400 ml. per minute of the acetone solution. That is to say, the acetone solution was diluted by a factor of about 9.5. The temperature was 73° F. The resulting dilute solution of acetone in water was allowed to stand 30 minutes and was then filtered. The recovered precipitate of RDX was washed with water and dried.

Needle-like crystals of RDX were obtained by this procedure. The crystals were rhombic and had a Coulter size of 10-100 microns with a mean size of about 50 microns. ("Coulter size" refers to the Coulter test which is well known and is a measure of volume of the individual particles whereas the usual sieve test gives a mesh size which is a measure of the smallest cross sectional area.) The RDX from which these rhombic needles were made had no crystalline structure observable through a microscope.

### EXAMPLE 3

This example relates to the application of the invention to a radically different type of explosive, namely ammonium nitrate. Five grams of ammonium nitrate were dissolved in 10 ml. of distilled water and 500 ml. of acetone were added slowly and with shaking and stirring. This procedure was repeated with another 5 gram quantity of ammonium nitrate but with the addition of 10 drops of Tergitol Anionic No. 4 (a trademark of Carbide and Carbon Chemical Corporation of New York, New York for a branched chain sodium alkylsulfate) to the aqueous solution of ammonium nitrate.

The acetone caused precipitation of ammonium nitrate which, after filtering and drying, was examined under the microscope. The crystals made without the surfactant were needle-shaped and had a size of about 60-100 microns in length and about 10 microns in width. The crystals made with the surfactant were rounded rhombic crystals, contained a few needles and had a length dimension of 20-50 microns and a width of 10-20 microns.

The method of the invention has also been applied to HMX and TNT, both of which yielded needle-like crystals.

Other solvents have also been employed. For example, methyl ethyl ketone has been used instead of acetone in the case of RDX. Needle-like crystals were obtained but were of a larger size than with acetone. Also, dimethyl sulfoxide was used as the solvent for RDX as follows: 5 grams of RDX and 2 ml. of Tergitol NPX were dissolved in 946 ml. of dimethyl sulfoxide and the solution was aspirated into a stream of tap water as in Example 2. Prismatic leaflet crystals were obtained with a small proportion of needles. Crystal length was 20-80 microns and width was 5-10 microns.

As will be seen the method of the invention does not always produce needle-like crystals, but the crystals are in any event elongated.

Many other solvent-precipitant combinations are available depending on the solute which is to be precipitated. The following table shows explosive materials in the left

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hand column, suitable solvents in the middle column, and suitable precipitants in the right hand column.

Table 1

Explosive	Solvent	Precipitant
PETN	Acetone	Water.
	Methyl ethyl ketone	Do.
	Methyl alcohol	Do.
	Methyl acetate	Do.
RDX	Benzene	CCl <sub>4</sub> .
	Acetone	Water.
	Ethyl alcohol	Water or isopropanol.
	Methyl alcohol	Water.
HMX	Ethyl acetate	Do.
	Methyl ethyl ketone	Do.
	Dimethyl sulfoxide	Do.
	Acetone	Do.

In addition to the surfactants described hereinabove, others may be used which are nonionic, anionic and cationic such as the ordinary soaps such as sodium stearate and oleate; other anionic surfactants such as turkey red oil, the various alkyl sulfates, alkyl benzene sulfonates such as the Oronite Detergents (Oronite Chemical Co., San Francisco, California) and the Naconols (Allied Chemical & Dye Corp., New York, N.Y.); cationic surfactants such as the higher alkyl, alkaryl, etc. quaternary ammonium salts; and various nonionic surfactants such as the polyethenoxy ethers of alkyl phenols, the polyethenoxy ethers of alcohols, pentaerythritol monostearate, polyethenoxy esters of tall oil acids, polyethenoxy alkalolamides of fatty acids such as the Ethomids of Armour and Co., and the fatty acid esters of sugar alcohols such as sorbitol and mannitol (e.g., the Spans of Atlas Powder Co.).

In practicing the invention the dilution of the starting solution (i.e., solution of the explosive material in the selected solvent before the precipitant is added) may vary considerably. Very dilute solutions, produce the smallest crystals, but more concentrated solution may be used. The degree of dilution of the solvent with precipitant will also vary depending upon the yield desired and the problem of solvent recovery if it is to be recovered.

It will, therefore, be apparent that a novel method of processing explosive and propellant materials has been provided; that explosive and propellant materials have been provided in a novel and useful form; and that certain novel and useful fabricated explosive articles have been provided.

We claim:

1. An article of manufacture comprising a housing having a cavity therein, a bridgewire within said cavity capable of exploding upon application of sudden high voltage, and an explosive solid in said cavity and in intimate contact with said bridgewire, said explosive being of a type which is initiated by an exploding bridgewire and substantially all of said explosive being in the form of small, elongated crystals having a density within the range of 1.2 to 1.27 grams per cc.

2. The article of claim 1 wherein said explosive is PETN.

3. The article of claim 2 wherein the PETN crystals have a length-to-width ratio not less than about 10 and a width not greater than about 20 microns.

4. The article of claim 1 wherein the explosive is RDX.

5. An explosive device comprising a housing, a charge of PETN within said housing and means for initiating said charge, substantially all of said PETN being present in the form of elongated crystals having widths of about 5 to 20 microns, said crystals being coated with a nonylphenyl ether of polyethylene glycol surfactant, said PETN elongated crystals being packaged within said housing at a density within the range of 1.2 to 1.27 grams per cc.

6. An EBW device comprising a housing having a cavity therein, a charge of PETN in said cavity and an exploding bridgewire in contact with said charge and capable, upon application of a high voltage from an

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EBW power source, of initiating said charge of PETN, substantially all of said PETN being in the form of elongated crystals having a width not exceeding about 20 microns, said charge of PETN having a density within the range of 1.1 to 1.27 grams per cc., said crystals being coated with a surfactant.

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