United States Patent Office 3,354,010 Patented Nov. 21, 1967

3,354,010

FLEXIBLE EXPLOSIVE CONTAINING RDX AND/OR HMX AND PROCESS THEREFOR

John D. Hopper, Succasunna, and Franklin B. Wells, Hackettstown, N.J., assignors to the United States of America as represented by the Secretary of the Army

No Drawing. Filed Jan. 27, 1967, Ser. No. 612,766
5 Claims. (Cl. 149—18)

ABSTRACT OF THE DISCLOSURE

A method for the preparation of a self-supporting high power, flexible sheet explosive of low impact sensitivity comprising the steps of mixing a high explosive from the group consisting of cyclotetramethylenetetranitramine (HMX), cyclotrimethylenetrinitramine, (RDX) and mixtures thereof, with a plasticizer to form a pasty mass, mixing into said pasty mass a high viscosity, alcohol-wet nitrocellulose containing about 12% nitrogen and a pigment for camouflage purposes, adding ethanol to the mixture to form a stiff dough and then mixing and roll-milling said dough at about 135° F. to form a flexible sheet explosive.

This invention is a continuation-in-part of our pending application Ser. No. 505,445, filed Oct. 27, 1965, now Patent No. 3,317,361, and relates to an explosive composition and more particularly concerns a flexible, self-supporting, water-resistant explosive and method for its preparation.

Our composition comprises a particulate explosive and a plasticized binder, the composition being useful in explosive metal-forming processes and admirably well suited for demolition purposes. The composition is characterized further by safety in handling, and it may be formed readily into any desired shape as by rolling, extrusion, compression-molding, etc. into strips, blocks, sheets, and the like.

It is therefore an object of this invention to provide an explosive having the aforementioned characteristics.

Another object of this invention is to provide a flexible explosive of high power (at least the equivalent of TNT) and brisance (rate of detonation at least about 7,000 m./sec.).

Still another object of this invention is to provide an explosive which, in sheet form, possesses sufficient flexibility so that it may be made to conform to the contour of uneven surfaces with a minimum of manipulation, thus aiding in the complete destruction of the device to be demolished.

A further object of the invention is to provide a flexible explosive having high resistance to impact and friction while retaining good cap sensitivity characteristics.

A still further object of this invention is to provide a flexible explosive which is more heat-stable than similar materials currently available.

Yet another object of this invention is to provide a flexible explosive which is not adversely affected by water.

A final object of this invention is to provide an explosive of sufficient resiliency so that, when made in block or other massive form, it will resist breaking up upon impact with a hard surface when striking such a surface with a velocity of at least 150 feet/second.

Other and further objects of this invention will become apparent as the invention is further described hereinafter.

We have found that the foregoing objects may be attained through provision of an explosive composition comprising about 45–76% of the cyclic polynitramines cyclotetramethylenetetranitramine, hereinafter referred to as HMX, and/or cyclotrimethylenetrinitramine, hereinafter

referred to as RDX, the said HMX and/or RDX having an average particle size of up to about 25 microns, preferably not over 13 microns. One hundred percent of the HMX and/or RDX must pas through a #200 U.S. Standard sieve, and at least 90%, and preferably not less than 97.5% thereof, should pass through a #325 U.S. Standard sieve.

Our composition will also contain about 17–39%, preferably about 25–30%, of a plasticizer such as tributyl acetylcitrate (preferred), dioctyl sebacate, triethylene glycol di(2-ethylbutyrate), or other similar materials, preferably those having pour points of —40° C. or below, about 5–15%, preferably 6–8%, of nitrocellulose preferably containing about 12.1–12.5% nitrogen and having such a degree of polymerization as to have a viscosity, using a 4% solution of nitrocellulose and a 5/16-inch diameter steel ball, of at least 90 seconds and preferably greater than 140 seconds, and about 0.2–1.0%, preferably about 0.3–0.8%, of a pigment to impart any desired color to the finished product.

In the determination of viscosity aforementioned, the steel ball shall weigh about 2.025 to 2.045 grams, the viscosimeter shall consist of a glass tube 14 inches in length with an internal diameter of 1 inch, the tube being immersed to the height of its liquid content in a constant temperature bath maintained at 25°±0.2° C., and the time of passage of the ball between markings shall be noted.

The aforementioned viscosity test is run in accordance with Specification MIL–N–224A, paragraph 4.4.5 et seq., dated Feb. 13, 1962, with the exception that the quantities of materials used are: 8 grams nitrocellulose, 21.3 grams ethyl alcohol, and 170.7 grams of acetone. The 21.3-gram/170.7 gram alcohol/acetone ratio is used to maintain the 1/8 ratio required in the specification.

While varying the proportions of all three of the major ingredients of our explosive results in some variations in the properties thereof, we have found in a series of tests that, in general, the presence of less than 45% of the particulate explosive results in reduced self-supporting properties and reduced power; whereas inclusion of more than 76% particulate explosive results in reduced cohesiveness. A content of less than 17% plasticizer results in reduced strength and cohesiveness; whereas the presence of more than 39% plasticizer results in a sticky product having reduced self-supporting properties. Less than 5% nitrocellulose in the product results in reduced strength whereas the presence of more than 15% nitrocellulose results in a product that is not sufficiently moldable. We have found, also, that substitution of even a minor proportion of the specified nitrocellulose by ordinary low-viscosity nitrocellulose results in a sticky product with reduced self-supporting properties.

For purposes of camouflage, an olive-drab coloration of the explosive is desirable. Less than 0.2% pigment is insufficient to give a satisfactory coloration to the product where a color other than white is desired, and more than 1.0% pigment is not necessary for effective coloring. The preferred pigment, which imparts an olive-drab coloration to our explosive composition, comprises one part lampblack and 8 parts chrome yellow, medium (lead chromate).

Addition of 0.4% (5% based on nitrocellulose present) diphenylamine, referred to hereinafter as DPA, to our explosive composition results in improvement of the product with respect to thermal stability to such a marked extent that when it is subjected to the 110° C. Vacuum Stability Test for 40 hours the volume of gas evolved is only about half that evolved when the composition without added DPA is similarly tested. Quantities other than 0.4 DPA all resulted in a reduced stabilizing effect. Other commonly used nitrocellulose stabilizers were found to be less ef-

fective. The Vacuum Stability Test is described in Method 503.1 of Military Standard MIL–STD–650.

Our product employing HMX and/or RDX as the particulate high explosive base provides unexpected and unpredictable advantages over flexible explosives containing pentaerythritol tetranitrate, hereinafter referred to as PETN, as the particulate high explosive base.

Compositions containing HMX and/or RDX but no DPA showed substantially greater thermal stability in the Vacuum Stability Test than did those containing PETN. According to Picatinny Arsenal Technical Report No. 1740, Revision 1, April 1958, vacuum stability values of PETN, HMX, and RDX at 100° C. are 0.5 ml., 0.37 ml., and 0.7 ml., respectively. It would be expected, therefore, that flexible explosive compositions differing only in which of these three explosive materials they contain should have almost identical thermal stabilities, but this has not been found to be the case. A series of vacuum stability results, not included in the examples, show that in the 40-hour 100° C. Vacuum Stability Test, using 5-gram samples, the PETN-based flexible explosive composition yielded about 1.5 ml. of gas, while similar HMX- and/or RDX-based flexible explosive compositions yielded about 0.75 ml. of gas. The advantage of the use of HMX and/or RDX becomes much more striking in the 110° C. Vacuum Stability Test in which unstabilized HMX- and/or RDX-based flexible explosive compositions yielded 4–5 ml. of gas in 40 hours, and various batches of similar PETN-based compositions yielded 11+ ml., usually in much less than 40 hours. The capacity of the apparatus used is 11 ml. The improved vacuum stability found for the HMX- and/or RDX-based flexible explosive compositions, therefore, is not predictable on the basis of the cited vacuum stability values for PETN, HMX, and RDX. Further indications of the improved thermal stability obtained through the use of HMX and/or RDX is found in the several examples which follow.

To further reveal the superiority of our cyclic-polynitramine-based product over a commercial PETN-based flexible explosive, samples of both were tested in a circulating-hot-air oven at 160° ±5° F. Some of the samples of the PETN-based explosive developed wrinkles and puckers in 2–3 days which grew to patches of one inch or more in maximum dimension until the 7th or 8th day of the test after which they remained static. None of the samples of our cyclic-polynitramine-based compositions developed any such imperfections during the test period of 6 weeks. This further illustrates an unpredictable advantage of our cyclic-polynitramine-based flexible explosive.

Again, in exudation properties, our flexible explosive was found to be superior to the PETN-based flexible explosive. The exudation test at 160° F., as outlined in Military Specification MIL–E–46776A (MU), is carried out as follows: A sample of the flexible explosive ¼-inch thick and 1½ inches square is placed on 15 layers of filter paper in the form of 2-inch diameter discs. A porous release paper is placed between the sample of explosive and the layers of filter paper. A 125-gram brass weight, 1½ inches square, is placed on top of the sample. This assembly is then placed on a wire screen and stored in a forced-draft oven for 24 hours at 160° F. Before assembly, the brass weight is weighed by itself and then reweighed with the sample. After hot storage, the sample and brass weight are removed from the filter paper while still warm, no attempt being made to separate the brass weight from the sample, and allowed to cool with the sample uppermost. When cool, the sample and brass weight are weighed together. The test is run in triplicate and the average of three results is reported to determine compliance with the requirement.

Our cyclic-polynitramine-based material readily met the 0.10% maximum weight loss requirement of the specification in the above exudation test by yielding values ranging between 0.090% and 0.094%; whereas the PETN-based flexible explosive failed by yielding results ranging between 0.11% and 0.20%.

In cap sensitivity tests it was found that the commercial PETN-containing flexible explosives showed wide variations in sensitivity to initiation, no only from lot to lot, but also within a given lot. This is exemplified by two extremes of one lot which both accepted and failed to accept initiation from standard #3, #4, #5, and #6 caps and accepted initiation consistently from standard #8 caps, and another lot which both accepted and failed to accept initiation from standard #1 caps and accepted initiation consistently from standard #2 caps. Our HMX-based material, on the other hand, accepted initiation from caps containing 13.5 grains of RDX as the base charge (M6 caps), but failed to do so from standard #8 caps, which have 8.5 grains of PETN as the base charge. Our RDX-based material accepted initiation from standard #8 caps but failed to do so from standard #6 caps. The erratic behavior of the PETN-containing material with respect to cap sensitivity constitutes an unexpected safety feature inherent in our HMX- and/or RDX-containing flexible explosives. In caps, as is well known in the art, the smaller the number of the cap, the smaller the explosive charge contained therein.

The cap sensitvity test was conducted as follows: strips of explosive material ¼ inch thick by 1 inch wide by 12 inches long were indented at one end by butting a notch 1.4 inch wide and 3.4 inch deep and indented at the other end with a similar notch ¼ inch deep. The initiating cap was inserted into the 3.4 inch-deep notch and taped in place. One end of a 3-foot-long piece of 50-grain Primacord was inserted into the 1.4 inch deep notch and taped in place. The assembly was placed on a smooth clean steel plate and the cap fired. Caps which initiated the strip of explosive caused the Primacord to fire and make a readily detectable mark on the plate. In cases where the cap failed to initiate the strip of explosive, the undetonated Primacord and a large portion of the test strip were always present after the test.

We have also devised a method for preparing our inventive compositions which offers distinct advantages over conventional methods. An outstanding advantage to be derived from our method is the granular form of the product which renders it particularly well suited for extrusion and compression-molding purposes.

In accordance with this method, nitrocellulose of the high viscosity type aforementioned containing about 12.1 to 12.5% nitrogen is dissolved in about 30–70 parts (preferably 48–50 times its weight) of a solvent such as butyl (preferred), propyl, or ethyl acetate with mechanical agitation. When solution of the nitrocellulose is complete, the stabilizer is added and dissolved and the plasticizer, in the amounts aforementioned, is then stirred into the solution. The stabilizer preferably will be 0.4% DPA, as aforementioned. The HMX and/or RDX and the pigment are separately suspended in about 20–80 (preferably 58–60) times their weight of water held at 80° C. in an open vessel. The HMX or RDX may be suspended without the presence of the other or a mixture of the two in any proportions would also be satisfactory. The nitrocellulose solution is added slowly in a fine stream to the vigorously agitated HMX and/or RDX suspension so that it is well dispersed therein. When all the nitrocellulose solution has been added, the temperature of the constantly stirred mixture is raised to 95–98° C. to distill off the solvent, which, preferably, is collected for reuse. The mixture is then cooled to 40° C. as rapidly as possible and filtered, after which the product in the form of small somewhat irregular pellets is air-dried until all apparent moisture has evaporated. It is then oven-dried to constant weight at 60° C. The granular product thus obtained is relatively free-flowing when first prepared. Upon standing, the granules tend to adhere lightly to one another, but the aggregate may be broken up readily, if desired. It may

be processed in any desired manner as by extrusion, rolling, molding, etc.

The products and process of our invention are further described and illustrated by the examples presented hereinunder. All proportions expressed in parts by weight unless stated otherwise.

Example I

The flexible explosive of this first example of our invention had the following composition:

	Grams
HMX (Grade II, Class B)[1]	315
Tributyl acetylcitrate	141
Nitrocellulose, alcohol-wet (dry basis)	40
Pigment (8 parts chrome yellow, medium/1 part lampblack)	4

[1] Specification MIL–H–45444A (Ord) w/Amendment 3 dated July 31, 1962. The HMX used in this and subsequent examples contained 1.55% RDX unless otherwise noted.

The HMX and tributyl acetylcitrate were mixed for three minutes in an open sigma blade mixer held at 135° ±5° F. The high viscosity nitrocellulose containing about 12.1–12.5% nitrogen and the pigment were then added and mixed in and about 60 ml. of ethyl alcohol added to lighten the consistency of the mix to that of a stiff dough. The dough was mixed about 15 minutes at 135°±5° F. in the open mixer and then formed into sheets on a roll mill, also held at 135°±5° F. The mix was first rolled at a roll gap setting of 0.010 inch until the alcohol was driven off. The roll gap was then increased to 0.210 inch and the thin sheets consolidated by rerolling into a smooth uniform sheet about one-quarter inch in thickness. This material is partially characterized by the following test results:

Vacuum stability test (5 g. for 40 hours) at 110° C.	4.58 ml. gas.
Cap sensitivity test	M6 cap.[1]

[1] In accordance with Specification MIL–C–45468A(MU) w/Amendment 5 dated May 15, 1964.

Example II

The composition of the explosive of this example was identical with that of Example I with the exception that the cyclic polynitramine used was Type B, Class E RDX.[1]

This material formed a smooth uniform sheet. It is partially characterized by the following test results:

Vacuum stability test (5 g. for 40 hours) at 110° C.	4.94 ml. gas.
Cap sensitivity test	#8 cap.

A similar flexible material based on PETN and prepared by the Du Pont Company gave a vacuum stability test value of 11+ ml. of gas when similarly tested. The superior thermal stability of our material is thus illustrated.

Example III

The composition of the flexible explosive of this example was identical in formulation with that of Example II except that RDX containing about 2.5% HMX was used. The characteristics of the resultant product were substantially identical with those of the product of Example II.

Example IV

The composition of the flexible explosive of this example was identical in formulation with that of Example I with the exception that HMX containing about 1.2% RDX was used. A smooth homogeneous sheet was obtained having characteristics and physical properties similar to those of the product of Example I. As in Example I, an M6 cap was required for initiation in the cap sensitivity test.

[1] Specification MIL–R–398C dated Aug. 22, 1962. The RDX used in this and subsequent examples contained 7.3% HMX unless otherwise noted.

Example V

The composition of the flexible explosive of this example was identical with that of Example II except that it contained 0.4 added part of DPA (based on 100 parts of flexible explosive as prepared in Example II), which was dissolved in the alcohol used to wet the nitrocellulose. The mixture gave a product very similar to that of Example II except that its vacuum stability value at 110° C. was 2.85 ml. of gas evolved, a considerable improvement over the value found for the unstabilized material.

Example VI

The composition of the explosive of this example was identical with that of Example I with the exception that 0.4 part by weight of the stabilizer DPA was added. The DPA was dissolved in the alcohol with which the nitrocellulose was wet and the nitrocellulose-alcohol-DPA mixture was allowed to age at least one day before use. The composition of the material of this example was:

	Parts
HMX	63.0
High viscosity nitrocellulose	8.0
Tributyl acetylcitrate	28.2
Pigment (of Example I)	0.8
DPA	0.4

The volume of alcohol used in this example amounted to 1.75 ml./g. of nitrocellulose, 0.5 ml. thereof being used to dissolve the DPA and provide the solution used to moisten the nitrocellulose and incorporate therewith the DPA, the rest being worked into the mixture to assure a heavy doughlike consistency. This material was rolled into a ¼ inch thick sheet which was smooth and uniform. It is partially characterized by the following test results:

d_4^{20} [1]	1.515.
Vacuum stability test (5 g. for 40 hours) at 110° C.	2.78 ml. gas.
Picatinny Arsenal impact sensitivity test [2]	15 inches.
Picatinny Arsenal explosion temperature test [3]	270° C. smoke.
Cap sensitivity test	M6 cap.

[1] The symbol d_4^{20} indicates the ratio of the density of the explosive sheet at 20° C. to the density of water at 4° C.

[2] The Picatinny Arsenal impact sensitivity test is described in "Standard Laboratory Procedures for Sensitivity, Brisance and Stability of Explosives," PATR No. 1401, Mar. 18, 1944, revised Feb. 28, 1950, W. H. Rinkenbach and A. J. Clear.

[3] The Picatinny Arsenal explosion temperature test is described in "Standard Laboratory Procedures for Sensitivity, Brisance and Stability of Explosives," PATR No. 1401, Mar. 18, 1944, revised Feb. 28, 1950, W. H. Rinkenbach and A. J. Clear.

Comparison of the result obtained in the vacuum stability test of this example with the corresponding test result shown under Example I clearly shows the value of DPA for stabilization of the mixture.

Example VII

The composition and procedure of this example were identical with those of Example V and Example VI except that the cyclic polynitramine used consisted of a mixture containing equal amounts of HMX and RDX. This mixture was prepared by mixing 47 parts by weight of the HMX of Example I with 53 parts by weight of the RDX of Example II. The composition of the flexible explosive prepared in this example was thus:

	Parts
HMX	31.5
RDX	31.5
High viscosity nitrocellulose	8.0
Tributyl acetylcitrate	28.2
Pigment (of Example I)	0.8
DPA	0.4

Test results obtained with this material are:

d_4^{20}	1.057.
Vacuum stability test (5 g. for 40 hours) at 110° C.	2.79 ml. gas.
Picatinny Arsenal impact sensitivity test	17 inches.
Picatinny Arensal explosion temperature test	246° C. smoke.
Detonation velocity	7008 m./sec.
Cap sensitivity test	M6 cap.

In the following three examples (Examples VIII, IX, and X) the cyclic polynitramine used was wet with 25–35% water. The water-wet polynitramine was machine-mixed with the plasticizer tributyl acetylcitrate before the other ingredients were added. Mixing was then stopped and the mixture was allowed to stand for a few minutes to allow the displaced water to rise to the top and be removed by decantation. Then addition of ingredients and machine-mixing were continued.

Example VIII

	Lbs.
RDX (as used in Example II but containing 30% water (dry basis)	12.60
Tributyl acetylcitrate	5.64
High viscosity nitrocellulose, alcohol-wet (dry basis)	1.60
Pigment (of Example I)	0.16

The RDX and plasticizer were mixed 3 minutes at 130° F. The mixer was then stopped and tilted to decant the water. The alcohol-wet nitrocellulose and the pigment were then added and the whole mixed 30 minutes with the mixer held at 130° F. and the top open. The mix was then rolled as described in Example I to give uniform homogeneous sheets having a vacuum stability value of 5.04 ml. gas at 110° C. Two PETN-containing analogs of this material (other than that noted for comparative purposes in Example II) showed vacuum stability values of 11+ ml. when tested at 110° C., demonstrating a substantially lower degree of stability.

Other test values:

d_4^{20}	1.490.
Detonation velocity	7044 m./sec.
Cap sensitivity test	#8 cap.
Friction pendulum test (steel shoe)[1]	No crackles, flame, or explosion.
Ballistic mortar test value (TNT value=1.0) [2]	1.0.
Picatinny Arsenal impact sensitivity test	14 inches.

[1] The friction pendulum test is described in "Picatinny Arsenal Testing Manual," May 8, 1950, J. H. McIvor, Manual 7–1.

[2] The ballistic mortar test is described in "Picatinny Arsenal Testing Manual," May 8, 1950, J. H. McIvor, Manual 7–2.

Rifle bullet impact test [1]	No fire or explosion.
Bar drop impact test [1]	Do.
Elevated temperature test [1]	No change.
Hot water immersion test [1]	Do.
Exudation test	0.09% loss.
Picatinny Arsenal explosion temperature test	208° C. (decompn.).

[1] The rifle bullet impact test, elevated temperature test, bar drop impact test, and hot water immersion test are described in Military Specification MIL–E–46676A(MU) dated Apr. 17, 1964, in paragraphs 4.3.7, 4.3.9, 4.3.11, and 4.3.15, respectively. In the elevated temperature test our samples were subjected to six weeks at 160° F. in lieu of the seven days specified in the above-noted specification.

Example IX

Several 80-pound batches of flexible explosive composition containing the DPA stabilizer (see Example V) were prepared by the mixing technique described in Example VII. A portion of these mixtures was processed by rolling as described in the above examples and the remainder by extrusion. The materials used in each batch were:

	Lbs.
RDX (as used in Example II but water-wet (dry basis)	50.40
Tributyl acetylcitrate	22.56
High viscosity nitrocellulose, alcohol wet (dry basis)	6.40
Pigment of Example I	0.64
DPA (dissolved in the alcohol wetting the nitrocellulose)	0.32

Typical test values obtained with this material are:

d_4^{20} of rolled one-quarter-inch thick sheet	1.466.
d_4^{20} of extruded 2-inch x 4-inch x 12-inch blocks	1.485.
Vacuum stability test (5 g. for 40 hours at 110° C.	2.85 ml. gas.
Picatinny arsenal impact sensitivity test	16 inches.
Picatinny arsenal explosion temperature test	250° C. smoke.
Detonation velocity (d_4^{20} 1.466)	7011 m./sec.
Cap sensitivity test	#8 cap.
Ballistic bortar test value (TNT value=1.0	1.0.
Rifle bullet impact test	No fire or explosion.
Elevated temperature test	No change.
Hot water immersion test	Do.
Bar drop impact test	No fire or explosion.
Flame sensitivty test [1]	No explosion.

[1] The flame sensitivity test is described in Specification MIL–E–46676A(MU), Apr. 17, 1964, in paragraph 4.3.8.

Extruded blocks of the flexible explosive of this example were found to withstand relatively high-velocity impact without breakage, undue distortion, or excessive bounce. In addition to blocks, ¼-inch thick sheets and filaments only ⅛ inch in diameter were prepared readily by extrusion. Blocks of the flexible explosive of this invention, similar to those produced by extrusion, have been produced by compression molding.

Example X

Using the same technique (of Example VIII), a 500-gram batch having the composition of the explosive of Example VI was prepared in a small sigma blade mixer and rolled into a smooth homogeneous sheet. As expected, this material was substantially identical with the material of Example VI.

Example XI

The composition of the flexible explosive prepared according to this example was identical with that of Example V, a new precipitation method being employed in its preparation. In accordance with this method, 24 grams of high viscosity nitrocellulose was dissolved in 1176 grams butyl acetate with mechanical agitation, 1.2 grams DPA added, and 84.6 grams of tributyl acetylcitrate then stirred in. This solution was then added slowly in a fine stream to a vigorously agitated suspension of 189 grams of the RDX of Example II and 2.4 grams of the lampblack-chrome yellow, medium pigment in 3,780 ml. of distilled water held at 80° C. When all the nitrocellulose solution had been added, the temperature of the vigorously agitated mixture was raised gradually to 98° C. to drive off the solvent. The suspension was then cooled quickly to 40° C. by passing water at about 16° C. through the jacket of the vessel used, and the whole vacuum-filtered. The granular product, consisting of somewhat rounded particles having a maximum dimension range of about 1/64 inch to 3/16 inch, was placed in shallow trays and air-dried overnight. It was then oven-dried at 60° C. to constant weight.

The granular product thus prepared was further processed conventionally by rolling into sheets as described in Example I except that rolling at the smaller roll-gap setting was only of such duration as to obtain a product of uniform appearance, there being no solvent to evaporate. This material was also formed into billets by compression-molding. The processed flexible explosive prepared according to this example was substantially identical in properties with that of Example V with the exception that its vacuum stability test value at 110° C. was 2.24 ml. gas evolved.

Example XII

The composition of the flexible explosive prepared according to this example was identical with that of Example VI, the method of preparation of Example XI being employed. In accordance with the method of this example 12 grams of high viscosity nitrocellulose was dissolved in 588 grams of butyl acetate with mechanical agitation, 0.6 gram DPA added, and 42.3 grams of tributyl acetylcitrate then stirred in. This solution was then added slowly in a fine stream to a vigorously agitated suspension of 94.5 grams of HMX (of Example I) and 1.2 grams of pigment (as used in Example I) in 5,544 ml. of distilled water held at 80° C. When all the nitrocellulose solution had been added, the vigorously agitated mixture was heated to 98° C. in an open vessel and held at that temperature until virtually all the butyl acetate had been driven off. The suspension was then cooled quickly to 40° C. by passing water at about 16° C. through the jacket of the vessel used, and the whole vacuum-filtered. The granular product, consisting of somewhat rounded particles having a maximum dimension range of about 1/64 inch to 3/16 inch, was placed in shallow trays and air-dried overnight. It was then oven-dried at 60° C. to constant weight.

The granular product thus prepared was further processed conventionally in exactly the same manner as the granular product of Example XI was processed. The processed flexible explosive prepared according to this example was substantially identical with that of Example VI. Some test results obtained with this material are:

d_4^{20}	1.513.
Vacuum stability test (5 g. for 40 hours) at 110° C.	2.89 ml. gas.
Picatinny Arsenal impact sensitivity test	18 inches.
Picatinny Arsenal explosion temperature test	267° C. smoke.

While the present invention has been described in detail, it will be apparent to those skilled in the art that there are many variations possible without departing from the scope of this invention, which is limited only by the appended claims.

We claim:

1. A method for the preparation of a self-supporting, resilient, high-powered, brisant, flexible sheet explosive composition of moderate cap sensitivity, low impact sensitivity, and good thermal stability and water resistance comprising the steps of

mixing about 63 parts of a high explosive comprising dry cyclotetramethylenetetranitramine (HMX) containing about 1.2–1.55% dry cyclotrimethylenetrinitramine (RDX) and about 28.2 parts tributyl acetylcitrate at about 135° F. to form a pasty mass,

mixing thereinto about 8 parts of high viscosity, alcohol-wet nitrocellulose containing about 12.1 to 12.5% nitrogen and 0.8 part of a pigment consisting of 1 part lampblack and 8 parts chrome yellow, medium, to form a mixture,

adding ethanol to said mixture to form a stiff dough,

mixing said dough at a temperature in the neighborhood of about 135° F. and

roll-milling said mixed dough at about 135° F. to form said flexible sheet explosive composition.

2. The method as described in claim 1 wherein said high explosive is dry RDX containing about 2.5–7.3% dry HMX.

3. The method of claim 1 further characterized by adding 0.4 part of diphenylamine stabilizer dissolved in the alcohol with which the nitrocellulose is wet and allowing the nitrocellulose-alcohol-diphenylamine mixture to age at least one day before use.

4. The method described as in claim 1 wherein the high explosive used consists of equal parts HMX and RDX.

5. The method of claim 2 further characterized by adding 0.4 part of diphenylamine stabilizer dissolved in the alcohol with which the nitrocellulose is wet and allowing the nitrocellulose-alcohol-diphenylamine mixture to age at least one day before use.

References Cited

UNITED STATES PATENTS

3,138,501	6/1964	Wright	149—92
3,235,420	2/1966	Murphy	149—18

BENJAMIN R. PADGETT, *Primary Examiner.*