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(54) Title: CAST PRIMER AND SMALL DIAMETER EXPLOSIVE COMPOSITION

(57) Abstract

A solid explosive composition is made by curing a mixture of a from about 20% to about 50% by weight of a liquid matrix, from about 50% to about 80% by weight of a dry or monohydrate inorganic chlorate or perchlorate salt, and from 0% to about 22% of a salt selected from the group consisting of nitrate oxidizer salts and inert chloride salts, after transferring said mixture to a mold. The matrix can include from about 50% to about 84% by weight of a non-explosive liquid fuel selected from the group consisting of polyhydric alcohols, lower aliphatic alcohols, ketones, and hydrocarbons; from 0% to about 15% by weight of a nitrate oxidizer salt; from 0% to about 15% by weight water; from 0% to about 15% by weight of a thickener; from 0% to about 5% by weight of an acid; and from 0% to about 2% by weight of a surfactant.

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SPECIFICATION

CAST PRIMER AND SMALL DIAMETER EXPLOSIVE COMPOSITION

Background of the Invention

5 Field: The invention is in the field of explosives of the solid cast primer or booster explosive type as well as explosives in general of small diameter.

10 State of the Art: With the advent of ammonium nitrate and fuel oil mixtures, known as ANFO, into the explosives industry in the early 1950's, and of other lesser or more sensitive explosive mixtures since that time, ways were sought to develop an initiator for these materials in surface and underground blasting. As a result, the cast primer or booster was developed. The cast primer is made from self-explosives such as trinitrotoluene [TNT], cyclotrimethylenetrinitramine [RDX], pentaerythritol tetranitrate [PETN], and mixtures thereof such as Composition B which contains about 60% RDX, 40% TNT, and some wax as a desensitizer. The first cast products were made with a variety of sensitive cores such as PETN or a coiled detonating fuse.

20 The known and presently used process for making such cast primers or boosters consists of melting the above-mentioned sensitive explosives or combinations thereof, often at temperatures of 100 degrees Centigrade or higher, and casting the melted explosive into molds. The greatest hazard posed by the operation is the risk that the temperature control means may fail, and thus, the explosive may reach decomposition temperature and explode. Aside from this grave risk, the process is inherently dangerous in that self-explosives are being handled. These are subject to shock detonation even at lower temperatures. Furthermore, in handling the cast products, an extremely sensitive dust may be produced which is even more hazardous than the cast primer itself. For example, following shipment, pentolite primers have been observed to leave a residue of abraded, fine, particulate dust in their shipping containers.

35 There are disadvantages other than the risk of explosion. The materials used are expensive and some are not available from domestic sources. The molds must, of course, be able to withstand the heat of the melted explosive. Nevertheless,

such primers are widely used because they have the advantage of being relatively water resistant. Other combinations of primers and boosters based on water gel and or emulsion-based compositions may display only some water resistance or no water resistance at all.

In the past, there have been attempts to formulate insensitive slurry or emulsion type explosive compositions using aqueous solutions of inorganic chlorate and perchlorate salts, i.e., salts of either the chlorate (ClO_3) or perchlorate (ClO_4) ions. These inorganic chlorate and perchlorate slurries and emulsions have serious drawbacks. Their density has to be carefully controlled by using gases (as small bubbles), micro-balloons or similar inert, insensitive, materials that complicate processing and detract from the energy of the primers.

It was a principal object in the making of the present invention to provide a castable primer explosive that would maximize borehole pressure (which is a function of both velocity and density), and that would use insensitive inorganic chlorate and perchlorate salts, especially the relatively shock-insensitive sodium perchlorate, instead of self-explosives. Other objects were to be able to make a cast primer or booster at ambient temperatures and thus avoid the hazard of working with explosives at elevated temperatures; to provide a cast primer or booster that, during handling, does not produce sensitive and hazardous dust; and to make a cast primer or booster that increases in sensitivity after it is formulated and placed in a container so as to enhance the margin of safety in handling the explosive.

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Summary of the Invention

According to the invention, a castable primer explosive composition is made up by combining about 20 to about 50% by weight of a liquid matrix and about 50% to about 80% of a dry, insensitive, oxidizer salt or mixture of such oxidizer salts. Preferred salts are inorganic chlorate or perchlorates.

The composition of the invention is hereinafter referred to as matrix-dry salt mixture. The inventors discovered that such a mixture cures to a solid if it contains a substantial

amount of the dry perchlorate or chlorate salt or salts. Curing takes place rather quickly, but, before it does, the matrix-dry salt mixture can be pressed, poured, or cast into a mold. The molded product can be detonated by a pentolite 5 detonator of about 8 grams. Preferred embodiments contain amounts of inorganic perchlorates that can be detonated with a No. 6 or a No. 8 blasting cap.

A typical liquid matrix includes from about 50% to about 84% by weight of a non-explosive liquid fuel, preferably a 10 polyhydric glycol such as diethylene glycol; 0 to about 22% by weight of an inorganic nitrate oxidizer salt, such as ammonium nitrate, potassium nitrate, sodium nitrate, or calcium nitrate; 0% to about 15% percent by weight water; 0% to about 15% by weight of a water-soluble polymer thickener 15 such as guar gum; 0% to about 5% by weight of an acid such as glacial acetic acid; and 0 to about 2% by weight of a surfactant. Except for the water soluble polymer thickeners, the preferred fuel is a water soluble, oxygenated, organic material of low volatility. Examples of the preferred fuel 20 include polyhydric alcohols, such as glycerol, ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, propylene glycol, dipropylene glycol, tripropylene glycol, and mixtures thereof. Also preferred are still bottoms remaining from the purification of the above, because 25 of their low cost. Depending on their composition, still bottoms can be used alone or in combination with any of the above polyhydric alcohols. As an additional option the matrix can include a cross-linking agent such as potassium pyro-antimonate.

30 The final mixture includes about 50 to about 80% by weight of a dry, inorganic chlorate or perchlorate salt. Sodium perchlorate is preferred but sodium chlorate, ammonium chlorate, and ammonium perchlorate can be used. The final mixture can also include an additional 0% to about 22% by 35 weight of a dry salt such as ammonium nitrate, calcium nitrate, sodium nitrate, potassium nitrate, or mixtures thereof in addition to any nitrate salt that may be included in the matrix. Some or all of this additional dry salt can be sodium chloride. Thus, as the price and availability of

the ingredients vary, the primers can be formulated to minimize cost.

A rough guide to formulating the primers of this invention is to proportion the inorganic oxidizers and the 5 organic fuels so that all of the hydrogen atoms in the formulations are converted to water and all the oxygen atoms in the formulations are converted to carbon dioxide. However, wide departures from this guide yield primers with good 10 mechanical properties that detonate reliably with a No. 8 blasting cap.

Initially, upon mixing the liquid matrix with the dry, inorganic, oxidizer salt or salts, a pourable or pumpable mixture is formed. On standing anywhere from twenty minutes 15 to several hours, depending on the formulation and temperature, the mixture cures to a solid, waxy mass with good mechanical properties. The cure mechanism is not well understood, especially with those formulations in which the water soluble polymer is not deliberately crosslinked, but may involve interactions in which the matrix polyols act as 20 polydentate ligands toward the oxidizer cations.

This method of making a solid, castable explosive is a safety improvement over prior art cast primers, especially when sodium perchlorate is employed, because none of the starting materials is self-explosive and it is not necessary 25 to heat the mixture in order to obtain an extrudable, pourable, or pumpable composition. In fact, only a mildly exothermic reaction takes place in making up the liquid matrix. Cooling the matrix before adding the dry product can 30 keep the mixture pourable for a longer period of time, i.e., extend the pour life or pot life. The resulting cast primer is economical, heat resistant, modestly water resistant, and has an explosive strength equivalent to that of prior art cast primers. The use of a hot melt seal on top of the explosive solid in the container renders the container and contents 35 water-tight.

Another surprising safety feature of the invention is that the primer tends to increase in detonation sensitivity as it cures, thereby allowing for safer mixing and handling in a less sensitive state. This phenomenon is opposite that

of conventional primers or boosters.

Another distinct advantage of the primers of the present invention over the prior art is the fact that their performance in terms of shock wave velocity improves as their 5 density increases. The optimum density for certain formulations is 1.80 grams/cc. Surprisingly, formulations show only slightly diminished activity and performance down to 1.50 grams/cc.

This is in direct contrast with many primers, which give 10 reduced velocity as the density increases. For example, micro-balloons or air entrapment must be used to lower density in order to make primers from materials such as ANFO emulsions or slurries. Also, the self-explosives usually require 15 blending to give an optimum density of about 1.5 to 1.6 grams/cc.

Of course, the objective is to maximize borehole pressure which is a function of both velocity and density.

Detailed Description of the Best Mode Contemplated

20 The solid explosive of the invention is made from a mixture of a liquid matrix and a dry oxidizer salt or salts, i.e., a matrix-dry salt mixture. After combining, the matrix-dry salt mixture is transferred to a primer container or mold. The resulting molded mixture cures to a solid product in about 25 two hours, more or less, depending on the temperature, the fluidity of the mixture when made, the particle size and particle size distribution of the dry solid oxidizers, and the amount of dry salt or salts used. If desired, a surfactant may be added to the liquid matrix to slow down the rate of 30 cure.

A typical matrix of the invention comprises from about 50% to about 84% by weight of a non-explosive liquid fuel, such as diethylene glycol, other polyhydric glycols, lower aliphatic alcohols, ketones, and hydrocarbons, such as fuel 35 oil or mixtures thereof; from 0% to about 15% by weight of water; from 0% to about 22% by weight of an organic or inorganic nitrate salt, such as calcium nitrate, ammonium nitrate, or sodium nitrate; from 0 to about 15% of a water soluble polymeric thickener, such as guar gum; from 0% to

about 5% by weight of an acid, such as glacial acetic acid; and from 0% to about 2% by weight of a surfactant.

The oxidizer salt is an inorganic chlorate or perchlorate salt, such as ammonium chlorate, sodium chlorate, ammonium perchlorate, sodium perchlorate, or mixtures thereof. A substantial portion of the oxidizer salt must be supplied in a dry form in order to obtain primers with good mechanical properties. The dry oxidizer salt can include lesser amounts of dry nitrate salts. The matrix-dry salt mixture comprises from about 50% to about 80% by weight of a dry inorganic chlorate or perchlorate, from 0% to about 15% by weight of a dry nitrate salt; and from about 20% to about 50% by weight matrix.

In the examples below of matrix-dry salt mixtures, the 15 68% perchlorate and 32% matrix blend illustrates the greatest tendency to shorter pot life or pour life. The use of 10% sodium nitrate, substituting for perchlorate, extends the pot life in mixes ranging from 10 lb. to 25 lb. (4.5 kg to 11.3 kg), for a matter of 30 seconds to 1.5 minutes. Also, the 20 addition of various surfactants, such as Amphoteric L and Amphoteric C, both available from Exxon Chemical Company, Milton, Wisconsin, in amounts of 0.25% to 0.5% of the final batch weight, increase pot life. However, the final density may be reduced from 0.5 gm/cc to 0.1 gm/cc because of the 25 nature of the surfactants. This pot life extension becomes moot if continuous mixing is employed.

It has also been found that particulate size of the sodium perchlorate has an effect on pot life. The following 30 table shows the percent retained in U.S. Standard Sieves for three samples identified as #1, #2, and #3.

U.S. Standard Sieve	#1	#2	#3
25	---	---	0.0%
40	---	0.4%	6.8%
50	20.6%	9.2%	56.4%
70	29.6%	47.8%	88.4%
80	32.3%	---	---
100	35.2%	82.4%	96.0%

40 As indicated only 35.2% of the #1 sample was retained on a 100 mesh sieve. When added to matrix, this sodium

perchlorate set up to be not pourable in less than one minute. The #2 and #3 samples had acceptable pour times with 10 lb. to 30 lb. (4.5 kg to 13.6 kg) batches of up to 3 to 4 minutes. With 80% to 90% retained on 100 mesh sieve, pour times have 5 been found to be adequate for batch mixing and pouring.

Sodium perchlorate is the preferred salt from a safety standpoint. Ammonium perchlorate is also quite safe to handle but is much more expensive. These salts yield a final product that is much safer to handle than the usual primers.

10 The higher the amount of perchlorate, the greater the density and ease with which the primers are detonated.

In a first example of the invention, the matrix had the following formula:

15	Diethylene Glycol	75%
	Water	10%
	Calcium Nitrate	12.4%
	Guar Gum	2.5%
	Glacial Acetic Acid	0.1%

20 In making up this matrix, the calcium nitrate was first dissolved in water. This solution was added to the non-explosive liquid fuel, i.e., diethylene glycol, wherein a mild exothermic reaction took place. It was desirable to keep the temperature of the matrix low, or to lower it after the exothermic reaction. Keeping the temperature low extended the time in which the freshly made-up matrix-dry salt mixture remained transferable.

After the aqueous solution containing the nitrate was 30 added to the non-explosive liquid fuel, guar gum was suspended in an aliquot of the liquid fuel, water, and the nitrate salt. Once suspended it was added to the liquid fuel-water-nitrate mixture.

The liquid matrix in such a formulation can include part 35 of the overall amount of sodium perchlorate as an aqueous solution thereof such as is available from commercial sources.

Of course, if added to the matrix as a solution, the amount of water used as such is reduced to keep the overall amount of water within an acceptable range.

The glacial acetic acid was added next and mixed.

5 Glacial acetic acid is a viscosity enhancer in the guar system. The matrix was now ready to be mixed with the dry salt. The matrix was of low viscosity initially and thickened with time as the guar dissolved. On standing for several hours it became thick and honey-like. However, it has been

10 found that there is no observed change or difference in the handling and performance of the final product whether the matrix is used fresh or aged for several days.

Dry sodium perchlorate salt was added to the liquid matrix to make up a matrix-dry salt mixture that was 67% by

15 weight sodium perchlorate and 33% by weight matrix. The sodium perchlorate used in this example and the other examples below was essentially dry, i.e., less than a percent or so water. However, it is believed that since some water is present in all acceptable formulations, it is not necessary

20 that the dry salt be essentially anhydrous and that perhaps even the monohydrate is dry enough to be used to form the fluid liquid matrix-dry salt mixture.

It has been discovered in accordance with the invention, that the fluid mixture is not sensitive to a No. 8 blasting

25 cap, while the finished product that hardens after about two hours is sensitive to a No. 8 blasting cap. This greatly adds to the margin of safety in handling the mixture. The final product had a density of 1.49 grams/cc. A one pound (454 gram) charge was detonated on a steel plate of $\frac{3}{4}$ inch (1.9 centimeter) thickness and blasted a hole in the plate.

In a second example, the liquid matrix had the following formula:

35	Aqueous Solution of Sodium Perchlorate (61%)	30%
	Calcium Nitrate	10%
	Diethylene Glycol	57%
	Guar Gum	2.9%
	Glacial Acetic Acid	.1%

40 Dry sodium perchlorate was added to the matrix to make a final product that was 60% dry sodium perchlorate and 40%

matrix. The final product had a density of 1.40 grams/cc. A one pound (454 gram) charge was sensitive to a No. 8 blasting cap. The charge was detonated on a $\frac{3}{4}$ inch (1.9 centimeter) thick steel witness plate and caused spalling of

5 the plate.

In a third example, the liquid matrix had the following formula:

10	Aqueous Solution of Sodium Perchlorate (61%)	20%
	Calcium Nitrate	10%
	Diethylene Glycol	67%
	Guar Gum	3%
	Glacial Acetic Acid	.1%

15 Dry sodium perchlorate was added to the matrix to make a final product that was 65% dry sodium perchlorate and 35% matrix. The final product had a density of 1.40 grams/cc. A one pound (454 gram) charge detonated with a No. 8 blasting cap. The charge was detonated on a $\frac{3}{4}$ inch (1.9 centimeter) thick steel witness plate and caused spalling of the plate.

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In a fourth example, the liquid matrix had the following formula:

25	Aqueous Solution of Sodium Perchlorate (61%)	35%
	Calcium Nitrate	10%
	Diethylene Glycol	53.5%
	Guar Gum	1.5%

30 Dry sodium perchlorate was added to the matrix to make a final product that was 55% dry sodium perchlorate and 45% matrix. The final product had a density of 1.40 grams/cc. A one pound (454 gram) charge detonated with a No. 8 blasting cap.

35 In a fifth example, the liquid matrix had the following formula:

40	Aqueous Solution of Sodium Perchlorate (61%)	25%
	Diethylene Glycol	73%
	Guar Gum	2%

Dry sodium perchlorate was added to this matrix to make a final product that was 62% dry sodium perchlorate and 38% matrix. The final product had a density of 1.54 grams/cc.

A one pound (454 gram) charge was sensitive to a No. 8 blasting cap. The charge was detonated on a $\frac{1}{4}$ inch (1.9 centimeter) thick steel witness plate and blasted a hole in the plate.

5 In sixth, seventh, eighth, and ninth examples, the liquid matrix had the following formula:

10	Diethylene Glycol	74%
	Water	11%
	Calcium Nitrate	12%
	Guar Gum	2%
	Glacial Acetic Acid	1%

15 In the sixth example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 68% dry sodium perchlorate and 32% matrix. A 250 gram charge was sensitive to a No. 8 blasting cap. The charge was detonated on a $\frac{1}{4}$ inch (1.9 centimeter) thick steel witness plate and blasted a hole in the plate.

20 In the seventh example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 53% dry sodium perchlorate and 32% matrix. The remaining 15% of the final product was made up with additional calcium nitrate. A 250 gram charge was sensitive to a No. 8 blasting cap.

25 In the eighth example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 53% dry sodium perchlorate, 32% matrix. The remaining 15% of the final product was made up with sodium nitrate. A 250 gram charge was sensitive to a No. 8 blasting cap.

30 In the ninth example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 60.5% dry sodium perchlorate and 32% matrix. The remaining 7.5% of the final product was made up with additional sodium nitrate. The final product had a density of 1.72 grams/cc. A one pound (454 gram) charge was sensitive to a No. 8 blasting cap. The charge was detonated on a $\frac{1}{4}$ inch (1.9 centimeter) thick steel plate and blasted a $\frac{1}{2}$ to $\frac{3}{4}$ inch (1.27 to 1.9 centimeter) hole in the plate.

35 40 In a tenth and eleventh example, the liquid matrix had

the following formula:

5	Diethylene Glycol	84%
	Water	12.5%
	Guar Gum	2.4%
	Glacial Acetic Acid	1.1%

10 In the tenth example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 64.5% dry sodium perchlorate and 28% matrix. The remaining 7.5% of the final product was made up with calcium nitrate. The final product had a density of 1.67 grams/cc. A one pound (454 gram) charge was sensitive to a No. 8 blasting cap.

15 In the eleventh example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 53% dry sodium perchlorate and 32% matrix. The remaining 15% of the final product was made up with calcium nitrate. The final product had a density of 1.64 grams/cc. A one pound (454 gram) charge was sensitive to a No. 8 blasting cap.

In a twelfth example, the matrix had the following formula:

25	Diethylene Glycol	75%
	Water	11.5%
	Calcium Nitrate	13.5%

30 In this twelfth example using the above matrix, dry sodium perchlorate was added to the matrix to make a final product that was 58% dry sodium perchlorate and 32% matrix. The remaining 10% was sodium nitrate. The final product had a density of 1.75 to 1.80 grams/cc with good mechanical properties. A one pound (454 gram) charge was sensitive to 35 a No. 8 blasting cap and blasted a hole 1.0 to 1.5 inches (2.54 to 3.81 centimeters) in diameter in a $\frac{3}{4}$ inch (1.9 centimeter) thick steel witness plate.

40 In a thirteenth example using the above matrix, dry ammonium perchlorate and sodium nitrate was added to the matrix to make a final product that was 58% dry ammonium perchlorate, 10% sodium nitrate, and 32% matrix. The final

product had a density of 1.75 to 1.80 grams/cc with good mechanical properties. A one pound (454 gram) charge was sensitive to a No. 8 blasting cap and blasted a hole 1.0 to 1.5 inches (2.54 to 3.81 centimeters) in diameter in a $\frac{3}{4}$ inch (1.9 centimeter) thick steel witness plate. Similar results were obtained using a final product that was 68% ammonium perchlorate and 32% matrix.

5 Tests on the various examples showed that velocity ranged from 19,000 to 23,000 ft/sec (5,791 meters/sec to 7,010 meters/sec) for the various final products depending on the formulation.

10 Whereas this invention is here illustrated and described with reference to embodiments thereof presently contemplated as the best modes of carrying out such invention in actual practice, it is to be understood that various changes may be made in adapting the invention to different embodiments without departing from the broader inventive concepts disclosed herein and comprehended by the claims that follow.

We claim:

1. An explosive product in solid form, comprising a mixture of:

about 50 to about 80% of a dry, inorganic, oxidizer salt
5 selected from the group consisting of sodium perchlorate, potassium perchlorate, ammonium perchlorate, sodium chlorate, potassium chlorate, ammonium chlorate, and mixtures thereof;

from 0% to about 22% by weight of a salt selected from the group consisting of calcium nitrate, sodium nitrate, 10 potassium nitrate, ammonium nitrate, inert chloride salts and mixtures thereof; and

from about 20% to about 50% of an initially liquid matrix material containing from about 50% to about 84% of a non-explosive liquid fuel selected from the group consisting of 15 polyhydric alcohols, lower aliphatic alcohols, ketones, and hydrocarbons; from 0 to about 22% of a salt selected from the group consisting of nitrate oxidizer salts and inert chloride salts; from 0 to about 15% of a thickener; from 0 to about 5% of an acid; and from 0 to about 2% of a surfactant.

20 2. An explosive product according to Claim 1 wherein the non-explosive liquid fuel was a water soluble, oxygenated, organic material of low volatility.

3. An explosive product according to Claim 2, wherein the water soluble, oxygenated, organic material of low 25 volatility was selected from the group consisting of glycerol, ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, propylene glycol dipropylene glycol, tripropylene glycol, still bottoms remaining from the purification of the above, and mixtures thereof.

30 4. An explosive product according to Claim 1, wherein the non-explosive liquid fuel was diethylene glycol.

5. An explosive product according to Claim 1, wherein the nitrate salt in the matrix was calcium nitrate.

35 6. An explosive product according to Claim 1, wherein the thickener was guar gum.

7. An explosive product according to Claim 1, wherein the acid was glacial acetic acid.

8. An explosive product according to Claim 1, wherein a cross-linking agent was included.

9. An explosive product according to Claim 8, wherein the cross-linking agent was potassium pyro-antimonate.

10. An explosive product according to Claim 1, wherein the initially liquid matrix material included from about 57% to about 84% diethylene glycol; from about 7% to about 15% water; from about 7.5% to about 10% calcium nitrate; from about 0.1% to about 3% guar gum; and from about 0.1% to about 1.1% glacial acetic acid.

11: A method of preparing a solid explosive product as claimed in any one of Claims 1 to 10 which comprises the steps of:

preparing a liquid matrix that includes from about 50% to about 84% by weight of a non-explosive liquid hydrocarbon fuel; from 0% to about 15% water, from 0% to about 15% of a thickener, from 0% to about 22% of a nitrate salt, and from 0% to about 5% of an acid;

mixing said liquid matrix with a dry oxidiser salt and dry nitrate salt making up a final fluid mixture that is from about 20 to about 50% liquid matrix, from about 50% to about 80% dry oxidiser salt, and from 0% to about 22% dry nitrate salt;

placing the resulting mixture into molds; and curing the molded mixture until it forms a solid.

12. The method of Claim 12, wherein the several steps are carried out at ambient temperatures.

13: A method according to any one of Claims 11 or 12 wherein the final fluid mixture increases in sensitivity upon curing to a solid form.