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## DESENSITIZED PENTAERYTHRITOL TETRANITRATE EXPLOSIVE

Joseph A. Wyler, Allentown, Pa., assignor to  
Trojan Powder Company, Allentown, Pa.

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This invention relates in general to explosive compositions and more particularly to desensitized pentaerythritol tetranitrate explosives.

It is well known that pentaerythritol tetranitrate possesses the disadvantageous property of being unusually sensitive to friction and impact. It is also known, although not so commonly, that pentaerythritol tetranitrate is very sensitive to detonation even when wetted with an equal weight of water. These factors are the main reason why pentaerythritol tetranitrate has not found greater usefulness, although many of its other explosive properties are of a distinctly superior nature.

The present invention is concerned with the preparation of certain pentaerythritol tetranitrate explosive mixtures having low friction and impact sensitiveness and at the same time having excellent sensitivity toward detonation by means of the usual initiators used in the explosives industry.

I have discovered that the hydrated calcium nitrates as  $\text{Ca}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ ,  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and  $\text{Ca}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$ , which melt at temperatures less than  $100^\circ \text{C}$ . can be used in combination with pentaerythritol tetranitrate to produce granular or pulverulent mixtures of excellent explosive properties.

In order to more clearly point out my invention, the following examples are given by way of illustration:

### Example 1

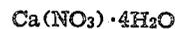
22 lbs. of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  are placed in a steam jacketed graining kettle provided with a suitable stirrer, and heated to a temperature above  $40^\circ \text{C}$ . in order to produce a thin liquid. Then 78 lbs. of pentaerythritol tetranitrate (preferably previously heated to above  $40^\circ \text{C}$ .) are incorporated into this liquid by stirring the mixture at a temperature above the melting point of the hydrous calcium nitrate. After about fifteen minutes of this stirring the mixture is allowed to cool slowly, with further stirring, until a granular or pulverulent mixture results. This mixture is then passed through an 18 mesh sieve to produce my preferred product.

In a similar manner I have prepared mixtures

of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and pentaerythritol tetranitrate of following compositions and properties:

PETN	$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	Grams fulminate of mercury required to cause detonation of (1" x 4" cartridges)	Standard drop hammer sensitiveness (10 kg. weight)
<i>Percent</i>	<i>Percent</i>		<i>Centimeters</i>
100	0	Less than .26 gms.	5-10
95	5	Less than .26 gms.	15
90	10	Less than .26 gms.	40
80	20	Less than .26 gms.	65
70	30	Less than .26 gms.	95-100
60	40	Less than .26 gms.	100+
50	50	Less than .26 gms.	100+
30	70	.4 gm.	100+
20	80	(Partial detonation .65 gm. Detonates with .8 gm.)	100+
0	100	Non-explosive	-----

These results show that the addition of



applied in the manner described above has a pronounced effect upon the impact sensitiveness of pentaerythritol tetranitrate without causing a deleterious drop in the detonative sensitiveness toward the proper initiating agents.

### Example 2

Ninety pounds of a mixture consisting of:

	Percent
$\text{Ca}(\text{NO}_3)_2$ .....	76.8
$\text{NH}_4\text{NO}_3$ .....	4.1
$\text{H}_2\text{O}$ .....	19.1

100.0

are heated to above the melting point (below  $100^\circ \text{C}$ .) in a suitable steam jacketed graining kettle provided with a stirrer. Then 10 pounds of pentaerythritol tetranitrate are added and the mixture stirred with cooling, until a granular or pulverulent product results. This product was detonatable by means of about .33 gm. of mercury fulminate and was insensitive toward impact (a 10 kg. weight did not cause a detonation when dropped from a height of 100 cm.).

Similarly, I may prepare mixtures of pentaerythritol tetranitrate with any of the hydrated calcium nitrates which melt below  $100^\circ \text{C}$ . Also, I may use mixtures of these calcium nitrates

with other nitrates as  $\text{NH}_4\text{NO}_3$ ,  $\text{NaNO}_3$ , magnesium nitrates, zinc nitrates, aluminum nitrates, etc. provided the mixtures melt at temperatures below  $100^\circ\text{C}$ . and above  $35^\circ\text{C}$ .

It should be noted that my pentaerythritol tetranitrate compositions are not merely mechanical mixtures in which the individual grains of pentaerythritol tetranitrates are in juxtaposition with the individual grains of the hydrated nitrate mixture as would be obtained if finely crystalline pentaerythritol tetranitrate and finely powdered nitrates are simply mixed below the melting point of the nitrates. My compositions are characterized by the individual crystals or agglomerations of crystals of pentaerythritol tetranitrate being imbedded within the solidified nitrate and by the resulting mixture consisting of discrete particles which may be spherical, granular or pulverulent. These mixtures are capable of being poured or pressed into molds or used in admixture with other materials commonly used in commercial explosives.

My pentaerythritol tetranitrate compositions are useful directly as explosives, as for example, a mixture consisting of 78 parts (by weight) of pentaerythritol tetranitrate and 22 parts (by weight) of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  which is essentially oxygen-balanced, has an unusually high brisance and is adapted for special uses where high shattering ability is required. Compositions containing more of the nitrates than is required for an oxygen balance (all C to form  $\text{CO}_2$ , all H<sub>2</sub> to form  $\text{H}_2\text{O}$ , the metals to form oxides and the nitrogen to be liberated as  $\text{N}_2$ ) are particularly useful for admixture with oxygen-deficient explosives as nitrostarch, nitrocellulose, trinitrotoluene, dinitrotoluene, dipentaerythritol hexanitrate, tetryl, nitrated alkyl lactates, nitrated alkyl glycolates, etc., or mixtures of these with oxidizing agents as  $\text{NH}_4\text{NO}_3$ ,  $\text{NaNO}_3$ , and the like or with reducing agents as woodpulp, charcoal, ivory nuts, paraffin, rosin, mineral oils, vaseline, and the like; or with oxygen-deficient or combustible materials in general.

It is important to note that I am using the term pentaerythritol tetranitrate in a broad manner. Persons familiar with the explosives art know that all commercial pentaerythritol tetranitrate contains some dipentaerythritol hexanitrate (often as much as 10-12%) and, at times, other impurities absorbed from the mother liquor obtained in the pentaerythritol crystallization. Consequently, the term pentaerythritol tetranitrate is used not only to mean pure pentaerythritol tetranitrate, but also the impure product normally obtained by the various commercial processes.

I wish to emphasize that the examples given above are merely illustrative of my invention and

are not intended to limit me to the method given therein, for the preparation of my compositions. I may, for instance, melt the hydrated-nitrate-mixtures, stir, add the pentaerythritol tetranitrate, stir, then allow the molten mixture to flow in the form of a thin stream, and then be subjected to a jet of air under high pressure so as to form small globules which upon falling through the cool air, solidify and form spheres; or I may use any suitable process which produces the product of characteristics described above. I, therefore, do not limit myself in any way except as indicated in the following claims.

I claim:

1. An explosive comprising pentaerythritol tetranitrate and a hydrated calcium nitrate of melting point greater than  $35^\circ\text{C}$ . and less than  $100^\circ\text{C}$ ., said pentaerythritol tetranitrate being substantially embedded within the hydrated nitrates and the mixture being composed of discrete particles.

2. An explosive comprising pentaerythritol tetranitrate and  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ , said pentaerythritol tetranitrate being substantially embedded within the hydrated nitrate and the mixture being composed of discrete particles.

3. A composition of matter comprising pentaerythritol tetranitrate and  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  in the proportions, by weight, of approximately 78 parts of pentaerythritol tetranitrate to 22 parts of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ , said composition being made up of solid, discrete particles in which the pentaerythritol tetranitrate is substantially embedded within the  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ .

4. An explosive comprising crystalline pentaerythritol tetranitrate, hydrated calcium nitrate of melting point between  $35^\circ\text{C}$ . and  $100^\circ\text{C}$ ., and an oxygen-deficient explosive; said pentaerythritol tetranitrate being substantially embedded within the hydrated calcium nitrate and this resulting mixture of pentaerythritol tetranitrate and hydrated calcium nitrate being composed of discrete particles; and said oxygen-deficient explosive being intimately mixed with the pentaerythritol tetranitrate-calcium nitrate mixture.

5. An explosive comprising crystalline pentaerythritol tetranitrate, hydrated calcium nitrate of melting point between  $35^\circ\text{C}$ . and  $100^\circ\text{C}$ ., and nitrostarch; said pentaerythritol tetranitrate being substantially embedded within the hydrated calcium nitrate and this resulting mixture of pentaerythritol tetranitrate and hydrated calcium nitrate being composed of discrete particles; and said nitrostarch being intimately mixed with the pentaerythritol tetranitrate calcium nitrate mixture.

JOSEPH A. WYLER.