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GRANULAR CYCLOTRIMETHYLENETRINITRAMINE EXPLOSIVE COATED WITH ALKYL AMIDE AND MICROCRYSTALLINE WAX

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This application is a continuation-in-part of copending application Serial Number 843,915, now abandoned, filed October 2, 1959.

This invention relates to explosives compositions and more particularly to such compositions containing desensitized cyclonite.

Cyclotrimethylenetrinitramine, which is frequently called cyclonite, is a commercially attractive explosive since it is exceedingly powerful, relatively non-toxic, and waterproof. Because of its great strength, it is used extensively in seismic exploration operations, in blasting, and in all applications requiring a high brisant explosive. This explosive compound is relatively insensitive in comparison with nitroglycerin compositions. However, pure dry cyclonite is too sensitive to permit shipping in interstate commerce. In order to pass the regulations of the Interstate Commerce Commission, this material must be sufficiently insensitive that it will not detonate when an 8 pound weight is dropped on it from a height of 7 inches.

The customary preparation of cyclonite for commercial shipping involves coating with 4 percent or 5 percent of a microcrystalline wax which desensitizes it sufficiently to comply with I.C.C. regulations. Although such wax coatings are generally satisfactory, they materially reduce the amount of explosive per pound of desensitized material. Thus, when a given weight of cyclonite is needed, the handling and shipping of inordinately large quantities of desensitizing material is involved. Also, the force of the explosive charge is necessarily diluted by the presence of the inert coating material.

It is, therefore, an object of this invention to provide new and novel desensitized cyclonite compositions and a method of preparing the same. Another object of this invention is to provide desensitized explosive compositions having maximum power. Another object of this invention is to provide cyclonite desensitized with a minimum of non-explosive material.

In accordance with this invention, these and other objects are accomplished, generally speaking, by providing granular cyclonite with a coating of a mixture of a microcrystalline wax and long chain fatty acid amide or amine. The basic amide or amine is an alkyl amide or amine containing between 8 and 20 carbon atoms in the alkyl chain. Suitable amides and amines include prelargonamide, capramide, myristamide, palmitamide, stearamide, octylamine, nonylamine, dodecylamine, tetradecylamine, octadecylamine, etc. and mixtures thereof. More specifically this invention contemplates cyclonite desensitized with between about 0.8 percent and about 2.5 percent of a mixture of a microcrystalline wax and an alkyl amide or amine with the organic base representing at least 0.65 percent of the weight of the cyclonite. Compositions containing less than about 0.65 percent of the organic base are not reliably desensitized. Likewise, cyclonite coated with less than about 0.8 percent of the wax-base mixture is inadequately desensitized. Amounts of the coating mixture in excess of 2.5 percent do not enhance the properties of the explosive but merely act as a diluent.

Any type of microcrystalline wax having a melting point in excess of about 75° C. can be used in accordance with the present invention. Suitable microcrystalline waxes include tank bottom microcrystalline wax, motor

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oil wax, residual microcrystalline wax, and the like, as well as mixtures thereof. Any of the various microcrystalline waxes can be obtained by processes well known to the art.

Cyclonite can be coated with the wax-organic base mixture in any conventional manner. Thus, the dry material can be tumbled consecutively with each component of the mixture until a uniform coating is obtained. Alternately, the material to be desensitized can be tumbled with a premixture of the coating ingredients. It is preferred, however, to suspend the explosive material in an aqueous slurry which is heated prior to the addition of the coating mixture. The cyclonite is then mixed with the coating mixture above its melting point until a substantially uniform coating is obtained. At this point, the water is removed by filtration, decantation, or by any other suitable method and the product is dried.

A major problem experienced previously with all explosive materials desensitized with a paraffin wax has been that of migration of the wax from the crystal surface of the explosive material when the explosive compositions are subjected to elevated temperatures such as encountered in oil well perforation operations. In such operations cyclonite is exploded with an initiator charge. When the wax employed to desensitize the cyclonite migrates into the initiator charge at the high temperatures encountered in deep wells the result is that the initiator itself is desensitized to such an extent that it may not fire. It has been found that when cyclonite is coated with a mixture of a microcrystalline wax and an alkyl amine or amide containing between 8 to 20 carbon atoms in the alkyl chain as described herein the problem of wax migration is substantially decreased because of the high melting point of the wax-base mixture.

Microcrystalline wax and the alkyl amide or amine when melted together form a mixture which when applied to cyclonite in the manner described herein yields a coating with extremely desirable properties. Such coatings have high melting points, they are flexible even at low temperatures, they possess excellent adhesive as well as good cohesive properties, and, in addition, they are pliable. Paraffin wax cannot be utilized in place of microcrystalline wax to prepare the valuable products of this invention because the films formed with it and the alkyl amine or amide are low melting, brittle and they exhibit a lack of adhesion.

In order to clarify the invention and to more fully describe the method by which it is carried out to achieve its various objects, the following is a detailed description of a preferred embodiment thereof. In this embodiment and throughout the specification, all proportions are expressed in parts by weight.

About 500 parts of crystalline cyclonite having a granulation such that it will pass through a U.S. No. 10 screen and be retained on a U.S. No. 325 screen were introduced into a jacketed vessel containing approximately 2,000 parts of water. The cyclonite and water were agitated to form a uniform aqueous slurry. Agitation was continued throughout the process. The vessel was heated by steam in the jacket to about 80° C. When this temperature was reached, about 4 parts of a fatty acid amide having an average carbon chain length of about 18 and a melting point of about 100° C. to 102° C. together with 1 part of microcrystalline wax were added to the aqueous suspension. The microcrystalline wax used was a highly refined paraffinic wax having a melting point of about 77° C. to 80° C. The wax and the organic amide were readily compatible and formed a mixture with a melting point from about 93° C. to 95° C. After the components of the coating composition were added to the vessel, the temperature of the slurry was elevated to about 97° C., slightly above the melting point of the coating material.

The agitated slurry was maintained at this temperature for about 15 minutes and then cooled to about 65° C. by running cold water through the jacket of the vessel. When the suspension was thus cooled, it was removed from the vessel and poured over a filter bed to remove the water from the coated cyclonite. The resultant product was then dried at 75° C. for 48 hours to obtain a material having a moisture content less than 0.1 percent.

The sensitivity of the cyclonite treated in this manner was determined by the Bureau of Explosives Impact Apparatus Test. In accordance with this test, an 8 pound steel ball is dropped on a compressed pellet of the explosive .003 of an inch thick and .2 of an inch in diameter confined rigidly between hard steel surfaces. In carrying out this test, 10 samples are subjected to the impact of an 8 pound ball dropping from a height of 7 inches. If any one of the samples detonates, the material cannot be shipped in interstate commerce.

When subjected to this test, the desensitized cyclonite of the present invention did not detonate, whereas the same material coated with 2 percent microcrystalline wax alone failed consistently. The results of these tests on the product of the present invention and on cyclonite coated with wax only is set forth in the following table:

Fall in Inches	Coating		
	2 Per-cent Wax	1 Per-cent Wax Mixture	5 Per-cent Wax
7-----	2/10	0/10	0/10
9-----		1/10	2/10
10-----		0/10	2/10
12-----		3/5	1/5
14-----		1/3	1/3
16-----		2/3	2/3

Each entry in the above table represents the results of a series of tests on specimens of coated cyclonite. The first digit indicates the number of specimens detonated, while the second number gives the total number of samples tested. For example, 10 samples of cyclonite coated with the desensitizer of the present invention were subjected to the impact of the ball falling from a height of 10 inches,

but none detonated. From the tabulated results, it is clear that cyclonite coated with 1 percent of the mixture of the present invention is more thoroughly desensitized than cyclonite coated with 2 percent wax. Also, it is self-evident that cyclonite thus coated has been desensitized to substantially the same degree as cyclonite coated with the conventional 5 percent wax. Thus, in accordance with this invention, cyclonite can be desensitized with a much smaller amount of coating material than have been heretofore found practical.

The above description is directed exclusively to cyclonite alone. It will be understood, however, that the cyclonite prepared in accordance with this invention can be mixed with other explosives and components in the preparation of commercial explosive compositions.

Although the invention has been described in considerable detail in the foregoing, it is to be understood that the purpose of such detail is only for clarification of the invention and that many modifications can be made by those skilled in the art without departing from the spirit and scope of the invention except as it is limited by the appended claims.

What is claimed is:

1. Granular cyclotrimethylenetrinitramine coated with about 0.8 percent to about 2.5 percent of a mixture of a microcrystalline wax and an alkyl amide containing between 8 and 20 carbon atoms in each alkyl chain, said alkyl amide representing at least 0.65 percent of the weight of the cyclotrimethylenetrinitramine.

2. Granular cyclotrimethylenetrinitramine coated with about 1 percent of a mixture of a microcrystalline wax and an alkyl amide containing between 8 and 20 carbon atoms in each alkyl chain, the weight ratio of said alkyl amide to said microcrystalline wax being at least 4:1.

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