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METHOD OF CRYSTALLIZING EXPLOSIVES FROM SOLVENTS

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The invention described herein may be manufactured and used by or for the Government for governmental purposes without the payment to me of any royalty thereon.

This invention relates to a method of crystallizing explosives from solvents.

In the recrystallization of explosives from solvents for the purpose of purification, the rate of cooling in the general manner of conducting the process is extremely slow, although precipitation of the crystals may be hastened somewhat by agitating the solvent. There is present, however, a serious tendency for the explosive to crystallize in large masses on the sides and bottom of the vessel. This is objectionable because it is difficult and even dangerous to remove the deposit and furthermore because the mass of crystals usually have to be crushed or broken before they can be brought to a physical condition where they are suitable for use.

In order to greatly reduce the period of time consumed in the crystallizing process, I make use of a cooling medium, and I have discovered that by regulating the rate of cooling of the explosive solution the nature and size of the crystals may be controlled and the deposit yielded in such a form that it may easily be removed with safety and in suitable condition for subsequent use. Inasmuch as certain solvents, such as benzene, toluene, xylene, carbon tetrachloride, and others which may be used in the crystallization process are insoluble or practically insoluble in water, I may conveniently employ water as the cooling medium.

The invention, therefore, consists essentially in hastening the crystallization of explosives from solutions of them in solvents, by bringing the solution directly in contact with a cooling medium, such as water, in such a manner that the explosive crystallizes in a form and manner which are desirable.

The method of carrying out the process to accomplish this result is as follows:

Should the explosive be required in the form of very fine crystals, the procedure consists in admitting a small stream of the hot and nearly saturated solution into a large volume of a cooling medium such as water which is vigorously agitated. Should the explosive be desired in the form of fairly large, uniform crystals, the procedure is reversed and a stream of water is admitted into a warm or hot and saturated or nearly saturated solution of the explosive and the mixture is kept in agitation. In the former case the cooling of the solution is effected more rapidly than in the latter instance. While the foregoing is a general method followed in producing very fine or fairly large crystals the intermediate sizes may be controlled either by regulating the temperature of the water or the rate of its admixture with the solution.

I have found that when the operation is carried out according to either of the above procedures the crystals of the explosive do not enter the water to any considerable degree but are retained in the solvent from which they are separated. The existence of this condition permits frequent renewal of the water supply to more rapidly cool the solution, it being only necessary to discontinue the agitation and allow the water to separate from the crystals and the solution. The importance of this feature in the practical application of the invention is evident since it allows the use of a much smaller vessel and a correspondingly smaller quantity of water than would otherwise be required to reduce the temperature of a given amount of explosive solution to the desired point, and so effect the crystallization of a portion of the explosive from the solution.

The exact manner in which the crystals and the mother liquor from which they have separated are to be handled depends upon the characteristics of the particular solvents. If the mother liquor is of lower specific gravity than water it usually separates, upon cessation of agitation, into two portions, one floating on the top of the layer of water and containing very few crystals, and the other forming a doughlike mass with the separated crystals and sinking to the bottom of the vessel. The elements which form the mixture are consequently conveniently disposed to be drawn off separately, the bulk of the mother liquor which is on top being first

removed without filtration, whence it can be used in dissolving another charge of explosive.

Where the mother liquors are of greater specific gravity than water, the water rises to the surface and can be easily drained.

While the invention is especially adapted to the purification of tetryl by recrystallization from benzene, toluene, and similar solvents which are insoluble in water, it is pointed out that its application is general to all classes of substances which are good solvents for various explosives and also to all practical non-miscible cooling mediums.

I claim:

1. A method of treating tetryl which includes forming a solution of tetryl in a solvent which is practically insoluble in water and bringing the solution in direct contact with cold water.

2. A method of treating tetryl which includes forming a solution of the tetryl and bringing the solution into direct contact with a non-miscible cooling element.

3. A method of treating explosives derived from a normally solid aromatic nitro amine which includes forming a solution of the explosive in a solvent which is practically insoluble in water and bringing the solution into direct contact with cold water.

4. A method of treating explosives derived from a normally solid aromatic nitro amine which includes forming a solution of the explosive, and bringing the solution into direct contact with a non-miscible cooling liquid.

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