Patented Feb. 20, 1940 2,190,703

UNITED STATES PATENT OFFICE

2,190,703

PERCHLORATE EXPLOSIVE

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No Drawing. Application March 8, 1938, Serial No. 194,605

7 Claims. (Cl. 52—18)

The present invention relates to new and improved explosive compositions and methods of preparing them, and more particularly to explosive compositions containing inorganic salts of chloric or perchloric acid as important ingredients.

Dynamites containing nitroglycerin have become the standards for high explosives by reason of their reliability and consistency of performance. Over a period of many years, however, there has been a trend toward the replacement of a portion of the nitroglycerin by inorganic explosive salts, particularly ammonium nitrate. This has meant increased safety in the manufacture of such explosives because of the great stability of ammonium nitrate and its insensitiveness to accidental shock, together with great economic advantage by reason of the availability of ammonium nitrate.

Less attention has been given to another attractive class of inorganic explosive salts, namely the alkali and alkali earth perchlorates and chlorates. These have been less widely used in commercial explosives and are less readily available; however, the widespread occurrence of their starting materials gives them an economic attractiveness. One of the disadvantages of ammonium nitrate lies in its high degree of hygroscopicity, and ammonium perchlorate possesses considerable superiority in this respect. In addition, the higher density of the perchlorates makes it possible to obtain compositions of higher density and higher bulk strength.

Heretofore it has been the practice, in the preparation of perchlorate explosives, to incorporate in the compositions a considerable amount of a high velocity explosive sensitizer—nitroglycerin, nitrostarch, trinitrotoluene, or like explosive material—since blending of the perchlorate with oxygen-accepting materials, which were non-explosive, gave a product of an insufficient degree of sensitivity.

The object of my invention is a new and improved explosive composition of desirable properties. A further object is an explosive composition containing as an ingredient an inorganic salt of chloric or perchloric acid. A further object is such an explosive which contains an inorganic perchlorate as an ingredient. A still further object is an improved method for producing such explosive compositions. Additional objects will be disclosed as the invention is described in more detail hereinafter.

I have found that the foregoing objects are accomplished by a process in which an inorganic perchlorate is dissolved in substantially anhydrous liquid ammonia, a sensitizing agent or oxygen-accepting ingredient is dispersed therein, the free ammonia is expelled, and a product is thereby formed in which the sensitizing material is dispersed in the solid inorganic perchlorate, the latter compound being the continuous phase. It is essential that the perchlorate employed be one soluble in liquid ammonia, and the perchlorates applicable according to my invention will be in general taken from the salts of the group comprising the alkali and alkali earth metals. More specifically, and preferably, they will be taken from the group consisting of the ammonium, sodium, calcium, magnesium, and barium perchlorates.

A large number of inorganic and organic compounds and materials are adapted for use as sensitizing ingredients in accordance with my invention, such sensitizers being ordinarily either explosive compounds themselves or compounds containing insufficient oxygen for complete combustion, hence capable of utilizing the excess oxygen present in the perchlorate molecule. According to my invention, a dispersion of the sensitizer in the perchlorate takes place. As examples of suitable sensitizers, I may cite such inorganic materials as aluminum, magnesium, antimony, zirconium, ferrosilicon, calcium silicide, sulfur, and the like; in fact, any inorganic oxygen-acceptor which is stable under atmospheric conditions and in the presence of the perchlorate used and of ammonia.

Likewise many organic sensitizers are highly efficient as sensitizing agents according to my invention. As examples of such compounds I may cite diphenylamine, dicyandiamide, acetamide, mono-nitronaphthalene, dinitrotoluene, trinitrotoluene, pentaerythritol, various sugars, paraffin, ammonium salts of organic acids, and many others. Thus either explosive or non-explosive organic sensitizers may be employed. I find it advantageous in many cases to employ a sensitizer which is itself soluble in liquid ammonia, since extreme intimacy of contact between the inorganic perchlorate and sensitizer is thereby readily effected, with increased control of sensitiveness to initiation and propagation.

It is not essential, however, that the sensitizer be soluble in liquid ammonia, since insoluble materials may be readily dispersed in solutions of the soluble perchlorate. A very efficient sensitization results also when the sensitizer is employed insoluble in liquid ammonia, together with a soluble dispersing agent.

In the foregoing, the preparation of perchlorate explosives has been particularly emphasized. It should be understood, however, that my ammonia process is equally applicable to the incorporation of the ingredients of chlorate explosives. My invention includes only those salts which are soluble in anhydrous liquid ammonia and which are stable under atmospheric conditions. Ammonium chlorate, unlike ammonium perchlorate, is definitely excluded from the scope of the invention, since it is an unstable salt. Sodium chlorate, on the other hand, is advantageously applicable.

The materials of the types named may be introduced into the system in any desired sequence in carrying out my invention, but the following procedure is satisfactory. The determined amount of sensitizer or blend of sensitizers in finely divided form is introduced into a solution of alkali perchlorate in substantially anhydrous liquid ammonia. The solution is preferably nearly saturated with respect to said perchlorate. It will be understood that the sensitizer may itself be soluble in liquid ammonia, may be insoluble therein, or again may be one that has increased solubility when first mixed with a dispersing agent. In the latter case, the substantially insoluble sensitizer is preferably thoroughly mixed with the dispersing agent before addition to the liquid ammonia solution, preferably with agitation. The free ammonia is then expelled by evaporation, with employment of reduced pressure if desired. The mass is preferably agitated vigorously during the escape of the free ammonia. The resulting material may be described as a dispersion product in which extremely minute regions of the sensitizer are disseminated within crystals of the alkali perchlorate, the latter being the continuous phase.

The following examples are illustrative of my invention and demonstrate various methods and compositions adaptable for use:

Example 1.—4182 grams of ammonium perchlorate and 354 grams of paraffin were introduced into a jacketed mixing machine provided with agitating blades. Sufficient liquid anhydrous ammonia was then introduced to take the ammonium perchlorate into solution, and agitation was maintained during solution of the ammonium perchlorate. The free ammonia was then expelled and the apparatus vacuated. The dry product comprised minute regions of paraffin disseminated within the crystals of ammonium perchlorate. The material was punched into cartridges to a density such that there were 123 1¼x8" cartridges to the 50-lb. case. The explosive, shot with a blasting cap, had a sensitiveness of 7 inches by the double cartridge test and a velocity of detonation of 4100 meters per second. A similar composition, when incorporated by dry mixing, was found to be insensitive to a commercial blasting cap.

Example 2.—2517 grams of ammonium perchlorate and 1769 grams of sodium nitrate were blended in a jacketed mixer, and 227 grams of paraffin was introduced, together with about 23 grams of a dispersing agent. Liquid anhydrous ammonia was introduced into the mixer to dissolve the ammonium perchlorate and the sodium nitrate. Agitation was maintained during solution of the above salts and the paraffin was dispersed throughout the solution. The free ammonia was then expelled, with final evacuation of the apparatus. The dry product was punched to a density such that there were 108 1¼x8" cartridges to the 50-lb. case. The explosive composition showed a sensitiveness by the double cartridge test of 7 inches and a velocity of 4100 meters per second.

Example 3.—60 grams of trinitrotoluene was dissolved in 140 grams of dinitrotoluene at a temperature between 50 and 60° C. This solution was added to a solution of 1600 grams of ammonium nitrate and 200 grams of sodium perchlorate in anhydrous liquid ammonia. Agitation was maintained during solution of the inorganic salts for the dispersion of the nitrotoluenes therethrough. The free ammonia was then expelled from the system. The dry product was punched into 1¼x8" cartridges at two densities, 114 and 167 cartridges respectively per 50-lb. case. At these densities, the explosive showed a sensitiveness by the double cartridge test of 3 inches and 9 inches respectively, and velocities of detonation of 4000 and 3500 meters per second. The explosive was insensitive to accidental impact.

Example 4.—The following composition was incorporated according to the procedure set forth in Example 1:

	Per cent
Sodium chlorate	85
DNT oil	10
TNT (crude)	5

The compositions prepared according to my invention may be employed as explosives, either by themselves or blended with other materials to obtain the desired oxygen balance and other properties. It will be understood that various degrees of sensitiveness may be obtained depending on the relative proportions of perchlorate and sensitizer used and on the nature of the sensitizing material.

In the examples given in the foregoing, ammonium perchlorate was used as the perchlorate soluble in liquid ammonia. It will be understood, however, that various alkali and alkali earth perchlorates are adaptable with advantage to my process, the essential being that the perchlorate be soluble in anhydrous liquid ammonia. In addition to the ammonium salt, I find the sodium, calcium, and barium salts satisfactory. Potassium perchlorate possesses only a low solubility in liquid ammonia, hence is less readily utilizable. When ammonium perchlorate is employed, it will ordinarily be desirable to include a sufficient amount of a material such as sodium nitrate to fix the liberated chlorine. This prevents the formation of the obnoxious chlorine gas in the fumes.

In my copending application Serial No. 194,551 filed March 8, 1938, I have disclosed explosive compositions comprising ammonium nitrate and prepared by the ammonia process. It will be understood that perchlorate explosives have important differences from, and advantages over, ammonium nitrate explosives. The present method of incorporating the alkali and alkali earth perchlorates with sensitizing material gives a much more intimate mixture of the two types of ingredients and allows a careful control of sensitiveness.

While my invention is directed toward the formation of explosive compositions by solution of said perchlorate in liquid ammonia and dispersion of the sensitizer thereby through the solidified perchlorate, I may employ other soluble inorganic salts with the perchlorate. Example 2 has shown a composition containing a considerable amount of sodium nitrate also. Likewise,

Example 3 illustrates an ammonium nitrate composition containing dissolved ammonium perchlorate also. Such compositions should be understood to be covered by my invention and are important embodiments thereof. If a chlorate is used, no ammonium salt should be present in the composition, because of the low order of stability of ammonium chlorate, which would form by metathesis.

My invention offers the additional advantage that explosives containing no organic explosive ingredients may be prepared thereby. Thus of all the possible sensitizers listed in the foregoing, only one, trinitrotoluene, is an organic explosive material.

As many different embodiments of this invention may be made without departing from the spirit thereof, I wish to be limited only by the following patent claims.

I claim:

1. An explosive composition comprising a solid dispersion of an oxygen-accepting ingredient in an inorganic salt of an acid taken from the group consisting of chloric and perchloric acids, which salt is soluble in anhydrous liquid ammonia and stable under atmospheric conditions, said dispersion resulting from crystallization from substantially anhydrous liquid ammonia.

2. A perchlorate explosive composition, comprising a solid disperson of a sensitizing material in an inorganic perchlorate soluble in anhydrous liquid ammonia, said dispersion resulting from crystallization from substantially anhydrous liquid ammonia.

3. The explosive composition of claim 2, in which the inorganic perchlorate is ammonium perchlorate.

4. The explosive composition of claim 2, in which the inorganic perchlorate is sodium perchlorate.

5. The explosive composition of claim 2, in which the inorganic perchlorate is barium perchlorate.

6. A perchlorate explosive composition comprising a solid dispersion of a sensitizing material within an inorganic perchlorate.

7. A perchlorate explosive composition comprising a solid dispersion of an ammonia-soluble sensitizing material in an inorganic perchlorate soluble in liquid ammonia, said dispersion resulting from crystallization from substantially anhydrous liquid ammonia.

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