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PROCESS OF TREATING SMOKELESS
POWDER

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The object of my invention is to restore the chemical stability of nitrocellulose smokeless powders which have undergone deterioration during storage and at the same time to conserve the ballistic qualities of the powder. My invention, while not limited to such, is particularly applicable to smokeless powder of the so-called Pyro type, that is, powder made from nitrocellulose with a nitrogen content of 12.50 to 12.70% having a solubility in ether-alcohol mixture in excess of 95% of which more than one billion pounds were made during the World War in the United States for the use of the Allied Governments.

This application is a continuation-in-part of my application, Serial No. 678,731, filed July 1, 1933, which resulted in U. S. Patent 2,033,217, on March 10, 1936.

It is well known that smokeless powder deteriorates during storage. As more than 16 years have elapsed since the end of the World War the Pyro cannon powder made during the stress of war time conditions is approaching the end of its useful life. This is particularly true since Pyro cannon powder of war time manufacture was for the greater part "water-dried", that is, steeped in hot water in order to remove to the desired degree the volatile solvents, ether and alcohol, used in its manufacture.

Up to the present time deteriorated nitrocellulose smokeless powders of the Pyro type have been reworked by a process involving pulverizing the powder grains under water by means of a heavy wheel mill and subjecting the finely divided material to a repurification treatment similar to that employed in the regular poaching treatment of the original nitrocellulose. Smokeless powder reworked in this manner has a relatively limited storage life because the process does not completely remove the deteriorated products. For that reason the reworked powder is usually assigned for practice use.

A principal object of my invention is to restore the chemical stability of the deteriorated powder while still retaining the powder grains in their original form, thus avoiding the expense of grinding and reworking deteriorated nitrocellulose smokeless powder, besides the accompanying hazards of manufacture.

In my co-pending application, Serial No. 678,731, aforesaid, I have described one embodiment of my invention in which distortion and cracking of the powder grains are minimized by subjecting them to a soaking treatment at room temperature, in order to permit the grains to

swell slowly, before subjecting them to extraction in boiling alcohol. However, this treatment tends to increase the brittleness of the powder grains, particularly the larger grains, since the viscosity of the nitrocellulose is reduced in proportion to the temperature and the time of extraction by means of the boiling alcohol.

I have now found that increased brittleness of the treated powder grains can be minimized and a still further decrease in the distorted and cracked grains can be obtained by carrying out the entire rejuvenation treatment at a temperature considerably below the boiling point of the ethyl alcohol, as, for example, at a temperature of 30° C. to 40° C.

One embodiment of my invention, as applied to deteriorated Pyro cannon powder of the 155 m/m granulation, is more fully described as follows:

The nitrocellulose powder grains are covered with ethyl alcohol (95% by volume) and subjected to a soaking treatment of five days at a temperature of about 30° C. The temperature of the alcohol is then gradually raised to 40° C. and the treatment continued at this temperature for about nine days with several changes of alcohol until the diphenylamine content of the powder grains has been reduced to about 0.02%. The extracted powder grains are then subjected to treatment in an alcoholic solution of new diphenylamine, containing about 1.25% of diphenylamine based on the weight of the powder. This treatment is carried on for two to three days at 40° C., preferably with circulation of the solution, until the powder grains have taken up the desired amount of diphenylamine, in this case about 0.50%. The amount of diphenylamine put back into the powder grains can be varied according to the amount of diphenylamine present in the dipping or rejuvenating solution. The excess alcohol is removed from the powder by draining or by wringing in a centrifugal wringer after which the powder is steeped in hot water at a temperature of 40° to 55° C. until the residual solvent or alcohol content of the powder grains is reduced to the desired figure. The powder grains are then air dried at about 55° C. for one or more days and finally conditioned to the desired moisture content.

The alcohol used may, of course, be denatured alcohol, such as formerly known as 2B containing 0.5 gallon benzol per 100 gallons ethyl alcohol, or other suitable grades of denatured alcohol.

The time of treatment can of course be varied so long as it is maintained until the powder is freed from objectionable products of decomposition.

5 As many apparently widely different embodiments of this invention may be made without departing from the spirit thereof, it is understood that I do not limit myself to the foregoing examples or descriptions except as indicated in the following patent claims.

10 I claim:

1. The process of restoring the chemical stability of diphenylamine-stabilized nitrocellulose powder grains while retaining the grains in substantially their original form, which comprises
15 treating the grains with ethyl alcohol at a tem-

perature of approximately 30° C., gradually raising the temperature to about 40° C., maintaining such temperature until the powder is freed from objectionable products of decomposition, and impregnating the powder grains with fresh diphenylamine in alcoholic solution. 5

2. The process of restoring the chemical stability of diphenylamine-stabilized nitrocellulose powder grains while retaining the grains in substantially their original form, which comprises
10 treating the grains with ethyl alcohol while maintaining the temperature within the range of 30° C. and 40° C., and impregnating the powder with fresh diphenylamine. 15

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