

UNITED STATES PATENT OFFICE.

ALFRED LUCK, OF DARTFORD, AND CHARLES F. CROSS, OF LONDON;
ENGLAND.

PROCESS OF INCREASING STABILITY OF NITROCELLULOSE.

SPECIFICATION forming part of Letters Patent No. 647,420, dated April 10, 1900.

Application filed March 27, 1899. Renewed March 9, 1900. Serial No. 8,078. (No specimens.)

To all whom it may concern:

Be it known that we, ALFRED LUCK, chemist, residing at Brencote, Dartford, in the county of Kent, and CHARLES FREDERICK CROSS, chemist, residing at 4 New Court, Carey street, London, England, citizens of England, have invented a certain new and useful Process for Increasing Stability of Nitrocellulose, (for which we have made application for a patent in Great Britain, No. 18,868, dated September 3, 1898,) of which the following is a specification.

Our invention relates to a process for rendering cellulose nitrates stable, this process being based upon observation of the nature and properties of impurities eliminated from nitrocellulose under the action of diluted solvents. The solutions thus obtained, when freed from any dissolved nitrate of cellulose by further dilution and filtration, are found to contain an acid substance or substances which unite with metallic bases, such as lead oxide, forming insoluble compounds. These precipitates when dried and analyzed are found to contain a high proportion of the metallic oxide—for example, three per cent. of PbO—and they also contain nitric nitrogen—for instance, three per cent. N, which gives them the characteristics of explosive bodies. It appears that when these acid compounds, which are present in the original guncotton, are combined with basic oxides they are rendered relatively inert and incapable of upsetting the stability of the guncotton. This result is brought about by treating the cellulose nitrates with solutions of the metallic salts. These may be dilute aqueous solutions, or the action may be carried out in presence of acetone. When no acetone is present, the guncotton is digested at 80° to 100° centigrade with sufficient quantity of a one-per-cent. solution of lead acetate or zinc chloride to entirely cover the guncotton. After thirty to sixty minutes digestion the excess of solution is removed and the nitrate is thoroughly washed until it is free from all traces of the soluble metallic salt, or the guncotton may be covered with acetone diluted by twice or thrice its weight of alcohol, and containing also the metallic salt in a one-per-cent. solution added to the extent of about one per cent.

by weight of the diluted acetone. The digestion in this case is effected at ordinary temperature in about half an hour, and then the solution is drained off and the nitrate is washed with water in successive small quantities until the bulk of the acetone is removed, the wash-waters being retained for the recovery or further use of the acetone. The product is then exhaustively washed.

The product of the treatment retains about two per cent. of the basic oxide (PbO) in combination with the acid groups before mentioned. We have obtained products showing satisfactory stability by treating cellulose nitrates with salts of lead, zinc, cadmium, calcium, barium, magnesium, and other metals. The most satisfactory results are obtained by using zinc, magnesium, and lead salts. We prefer to use the salts of these metals with acids of low hydrolizing function, such as acetates.

We are aware that certain metallic compounds have been used in association with guncottons with a view to increase their stability. Of these we may instance calcium and magnesium carbonates, which are in some cases added for the purpose to the explosive nitrate, but more often find their way into the product as the result of the boiling and washing with ordinary water. Mercuric chloride has also been added to guncottons, and the presence of this salt has been assumed to raise the stability of the explosive; but this conclusion has been shown to result from an interference of the mercury salt with the chemical reactions involved in the usual "heat test." These facts are fully set forth in the recent publications of Simon Thomas, *Zeitschrift Angewandte Chemie* for 1898, pp. 1003-6 and 1027-31, which contains no suggestion of the presence of such acid compounds in guncottons as we have particularized, nor of such treatment as we propose with metallic salts for the purpose of bringing them into a relatively inert form by combination with basic metallic oxides.

Having thus described the nature of this invention and the best means we know of carrying the same into practical effect, we claim—

The herein-described process for increasing the stability of nitrocellulose, which con-

sists in freeing the nitrocellulose from the nitrating acid, treating it with a solution of acetone and metallic salts, and alcohol, and washing the nitrocellulose, in successive
5 washes to remove the acetone, substantially as specified.

In testimony whereof we have hereunto set

our hands in presence of two subscribing witnesses.

ALFRED LUCK.
CHARLES F. CROSS.

Witnesses:

JOSEPH LAKE,
GERALD L. SMITH.