UNITED STATES PATENT OFFICE.

ALFRED LUCK, OF DARTFORD, COUNTY OF KENT, AND CHARLES F. CROSS, OF THE COUNTY OF LONDON, ENGLAND.

PROCESS OF TREATING NITRO-CELLULOSE.

SPECIFICATION forming part of Letters Patent No. 670,346, dated March 19, 1901.

Application filed September 17, 1898. Serial No. 691,216. (No specimens.)

To all whom it may concern:

Be it known that we, ALFRED LUCK, residing at Brentcote, Dartford, Kent, and CHARLES FREDERICK CROSS, residing at No. 4 New Court, Carey street, in the county of London, England, chemists, citizens of England, have invented a certain new and useful Improvement in the Treatment of Nitro-Cellulose for the Manufacture of Explosives and other Products, (for which we have applied for patents in Austria July 9, 1898; in Germany July 7, 1898; in Great Britain March 3, 1898, No. 5,286, and in Hungary July 13, 1898,) of which the following is a specification.

Nitro-cellulose as it is usually manufactured retains a fibrous structure which occasions considerable difficulties in the subsequent treatment. Even in ordinary granulated nitro-cellulose the structure of the fibers still remains in the granules, not having been always destroyed by granulation.

Our invention relates to means of avoiding these difficulties by reducing the nitro-cellulose at once in its original fibrous form to a structureless condition without mechanical treatment. For this purpose we subject nitro-cellulose, either in the mass or in a more or less finely-divided condition, to the action of solvents, such as acetone, acetic ether, pyridine, nitrobenzene, and the like diluted with a proportion of inert liquid. In the case of acetone or other solvent miscible with water we use, preferably, water as the diluent. When the solvent is not miscible with water—as, for instance, nitrobenzene—it may be diluted with alcohol. Such liquids rapidly and completely destroy the fibrous condition of the nitro-cellulose, and at the same time it is freed from certain impurities which remain dissolved in the liquid even after further dilution. The case of acetone may be taken as typical, and we shall therefore describe the process as carried out with this solvent. A cellulose nitrate of, say, thirteen per cent. nitrogen (taken not necessarily in a dry condition) is placed in a suitable vessel, and a diluted acetone—that is, acetone diluted with water in the proportion of about ninety-two parts acetone to eight parts water, by volume—is poured upon the fibrous nitrate in quantities sufficient to cover it. The change of physical condition rapidly ensues, the swelling of the fiber being followed by a disintegration, so that on slight agitation the last remnants of the original fibrous structure are destroyed. This effect is attended by a certain limited solvent action upon the fibrous nitrates, whereby certain constituents are taken up in solution and, in fact, in a permanently-soluble form. The solution thus formed is diluted with water, whereby any structureless cellulose nitrates which may have been dissolved are precipitated; but there remains in solution a group of constituents of strongly acid character, which it is desirable to eliminate. The insoluble nitrates are therefore separated from the solution containing these by-products by filtering and washing first with dilute acetone and then with water to recover the acetone as completely as possible. The washed product is then dried and is found to possess the characteristics of stability required for working up into the special forms required in commerce. Thus, besides effecting the physical change from the fibrous to the structureless form, the process chemically purifies the product.

For certain purposes the fibrous nitrate may in the first place be granulated or reduced to a more or less finely-divided condition in the wet state, the granules being then treated as above described, rendering them individually more or less structureless and coherent without any tendency to agglomerate into masses. On further dilution of the acetone and washing the granules acquire density and hardness and on being dried form a free granulated powder. The product is also chemically "stable." The simultaneous changes in form and composition furnish a product which may be directly worked up for smokeless powders suitable for the class of explosives known as "sporting-powders."

Instead of entirely removing the fibrous character of the product we can so treat nitro-cellulose that it becomes a fine fibrous powder. For this purpose we prefer to employ a mixture of acetone and alcohol, for instance, in equal proportions by volume. The material is covered with this mixed solution, and

on then adding water the material is reduced to the condition of an extremely fine fibrous powder.

The removal of by-products causing instability may be effected as a separate treatment independently of disintegration by treating the fibrous nitrate with a more dilute acetone, such as equal parts, by volume, of acetone and water.

From any of the treatments above described there is a dilute acetone residue from which the acetone may be recovered by distillation at a low temperature.

The acid by-products remaining in solution have the property of forming insoluble lead compounds and may be precipitated by the addition of a solution of lead acetate. The lead salts thus obtained contain fifty to sixty-three per cent. PbO and three to four per cent. of nitric nitrogen. They are explosive and may be used in fireworks.

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

The herein-described process of reducing nitro-cellulose to an approximately structureless condition for the manufacture of explosives or for other purposes, which consists in subjecting the nitro-cellulose in its original fibrous form to the action of a diluted solvent in a sufficient quantity to cover the nitro-cellulose to render it structureless, in greater or less degree, and simultaneously therewith remove the impurities, and finally washing.

In testimony whereof we have hereunto set our hands in presence of two subscribing witnesses.

ALFRED LUCK.
C. F. CROSS.

Witnesses:
FRED C. HARRIS,
JNO. P. M. MILLARD.