

UNITED STATES PATENT OFFICE.

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SMOKELESS POWDER AND METHOD OF MAKING SAME.

SPECIFICATION forming part of Letters Patent No. 781,926, dated February 7, 1905.
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To all whom it may concern:

Be it known that I, HARVEY W. WILEY, a citizen of the United States, residing at Washington, in the District of Columbia, have invented certain new and useful Improvements in Smokeless Powder and Methods of Manufacturing the Same, of which the following is a specification.

Experience has shown that the utility of nitrated cellulose for producing explosives which are safe and effective depends, first, upon the degree of nitration of the cellulose of whatever origin; second, on the solubility of the product in certain solvents and the character and properties of the colloids produced. In addition to these the form in which the powder is used is of great importance, and the properties of the colloid permitting the formation of the desired shapes and sizes and the toughness and other properties of the dried mass must be taken into consideration.

My invention relates to the methods of securing a colloid of the properties above mentioned with any desired percentage of nitrogen, and a description of the way in which this is done follows.

It is well known that in the nitrating of cellulose the theoretical conditions of nitration—viz., the replacement of all the hydrogen possible in the cellulose compound by the radical NO₂ is rarely if ever attained. If such a complete replacement could be secured in pure cellulose, the percentage of nitrogen in the compound would be 14.14. In point of fact, in the manufacture of smokeless powders the nitrated cellulose employed usually has a nitrogen content varying between twelve and thirteen per cent. It is well known that if the content of nitrogen rises above thirteen per cent. there is formed what is known as a "guncotton," which is practically insoluble in a mixture of ether-alcohol. If the content of nitrogen falls below twelve per cent., colloids are formed, which, indeed are soluble, but have far too small a content of nitrogen for practical purposes. Hence the efforts of inventors have been centered in securing, if possible, some compound of nitrated cellulose

having a content of nitrogen lying between twelve and thirteen per cent. and at the same time preserving the viscosity, toughness, and hardness of the colloid produced by solution in an appropriate solvent which is necessary to secure a proper and effective powder. I am aware of the various methods which have been employed by other inventors to secure this object, viz: in the first place, in the attempts to secure an original nitration of any given degree, say, for instance, 12.45 per cent. of nitrogen; second, in mixing nitrated cellulose containing different proportions of nitrogen together in such quantities as to secure a given content of nitrogen, as, for instance, 12.75 per cent.; third, by dissolving out of nitrated products of a low content of nitrogen certain proportions of the lower forms of the collodion present, so as to raise the content of nitrogen in the residual mass to any given degree—viz., say, 12.50 per cent. The proportions given above are simply used for illustrative purposes to show the conditions upon which these desired contents of nitrogen are secured.

It is evident that in any given case the content of nitrogen should depend upon the purpose to which the powder is to be applied. Hence it often happens that for one purpose a powder of a low content of nitrogen is desirable, while for another a powder of a higher content is preferred. My invention relates to the manufacture of a powder of any desired content of nitrogen to secure the purposes above mentioned. To this end I make use of the well-known facts of the solubility of nitrated cellulose containing varying proportions of nitrogen in different solvents. For instance, it is well known that guncotton—that is, nitrated cellulose—having so high a content of nitrogen that it is insoluble in ether-alcohol is soluble in acetone, acetic ether, benzol, nitro-benzol, and other bodies. The colloid, however, formed by most of these solvents is somewhat too hard and brittle to make a powder of the best quality. On the other hand, those forms of nitrocellulose which are known as "collodion" are more or

less completely soluble in mixtures of two parts of ether and one part of alcohol or other varying quantities of the solvents. I make use of these different reactions toward different reagents for the purpose of securing a powder of any desired nitrogen content whatever and at the same time of a physical structure which is best for the purpose mentioned. It is evident that the more nitrogen introduced into a molecule of this kind the more oxygen also, since for each atom of nitrogen introduced two atoms of oxygen are incorporated. In burning an ordinary smokeless powder which contains, say, 12.5 per cent. of nitrogen only there is not a sufficient quantity of oxygen to completely oxidize the hydrogen and the carbon, and for this reason considerable quantities of carbon monoxid and free hydrogen are produced. It is evident that if any additional quantity of oxygen could be incorporated in the molecule a more effective powder would be made, provided the physical state of the powder were not injured. To this end in my invention I take a highly-nitrated guncotton containing above thirteen per cent. of nitrogen, in fact as high a degree of nitration as may be possible approaching the theoretical limit of 14.14 per cent., and dissolve this in acetone. As has before been pointed out, a colloid made of this alone would be too brittle for practical use, and hence in order to secure the requisite degree of plasticity and toughness in the colloid I dissolve a guncotton of a lower degree of nitration—say below thirteen per cent.—in a mixture of ether-alcohol. I now have two dissolved portions of nitrated cotton, one containing a very high percentage of nitrogen and the other moderately low percentage, and I mix these two together in any desired proportions to secure, on the one hand, a very high content of nitrogen and, on the other, a moderate content of nitrogen. This I illustrate by the following examples. In the first place I take a nitrated cellulose containing 13.75 per cent. of nitrogen and dissolve a given weight of it in acetone. In the second place I take a nitrated cellulose containing 12.75 per cent. of nitrogen and dissolve it in ether-alcohol. Now if I desire to make a smokeless powder with a content of nitrogen of 13.25 per cent. I take equal portions of these two viscous masses and mix them together, thus securing a higher content of nitrogen than has ever before been secured in a practical way, while at the same time I have a colloid mass which is tough and resistant and has the valuable properties of the ordinary colloid made by dissolving collodion in ether-alcohol. It is evident that I may vary the proportions of the two constituents at will, and thus be able, on the one hand, to make a powder containing almost the maximum content of nitrogen found in the portions dissolved in acetone, and, on the other, almost the minimum content of nitrogen of the por-

tions dissolved in ether-alcohol. Thus in the simplest possible manner and most effective way I am able to produce a powder of any desired quality for any specific purpose.

The smokeless powder made in the manner above described differs in its properties from other smokeless powders in these and other respects—namely, it is not soluble in any single solvent, either in the non-colloidal or colloidal form nor in a mixture of ether and alcohol in any proportions. In order to dissolve a powder of this kind, it is necessary to treat the small particles successively with different solvents, separating the insoluble portions by centrifugal action or otherwise and subjecting them to treatment with a different solvent. For instance, powder of this kind would be partially soluble in ether and partially in alcohol and more soluble in a mixture of ether and alcohol; but in all these cases a considerable residue would remain undissolved, and to dissolve this residue it would be necessary to separate by centrifugal action or otherwise and treat it with another solvent, such as an acetone. By proceeding in this way it would be possible to bring it completely in solution.

While I have mentioned in the above specification the use of acetone and a mixture of ether and alcohol as the solvents employed, I do not confine myself to those solvents, but to any solvents whatever which will dissolve the nitrated cellulose in its various degrees of nitration.

Having thus described my invention, what I claim is—

1. The method of producing a smokeless powder having a desired percentage of nitrogen which consists in separately dissolving nitrated celluloses having known contents of nitrogen and combining the solutes thus obtained in proportions necessary to form a powder having a predetermined content of nitrogen.

2. The method of producing a smokeless powder having a desired percentage of nitrogen which consists in separately dissolving nitrated celluloses, having known contents of nitrogen, and of different solubility, in their appropriate solvents, and combining the solutes thus obtained in proportions necessary to form a powder having a predetermined content of nitrogen.

3. A smokeless powder having a predetermined content of nitrogen in a colloidal form, insoluble in any single solvent or in a mixture of ether and alcohol in any proportion.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

HARVEY W. WILEY.

Witnesses:

A. E. T. HANSMANN,
W. CLARENCE DUVAL.