# UNITED STATES PATENT OFFICE.

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## PROCESS OF MAKING NITRO-DEXTRINE.

SPECIFICATION forming part of Letters Patent No. 242,893, dated June 14, 1881.

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*To all whom it may concern:*

Be it known that I, GILBERT S. DEAN, of the city and county of San Francisco, and State of California, have invented an Improvement in the Preparation of Nitro-Dextrine; and I do hereby declare the following to be a full, clear, and exact description thereof.

The object of my invention is the preparation of a new variety of nitro-dextrine. Briefly stated, the manner of its accomplishment consists in treating vegetable fiber with dilute acid, whereby its structure is destroyed and dextrination commenced, and afterward nitrating the same with cold concentrated nitro-sulphuric acid.

It is well known that if cloth, paper, or any similar substance be immersed in cold sulphuric acid it becomes converted into dextrine or "parchment." If, however, the sulphuric acid contains nitric, nitro-substitution takes place, but there is no dextrination.

The object of the present invention is to bring about both these changes at once, and thereby to produce a material new to the arts.

The process divides itself naturally into three stages—to wit, first, the preparation of the material; second, the nitration; third, the washing.

The material used is old rag which has been thoroughly bleached. Unbleached cotton, hemp, &c., may be used; but the first-named material is that which I consider best.

The object of the first step is to start the dextrination; for if this be once commenced it will be continued and completed in the nitrating-acids. To this end the rag is immersed in sulphuric acid; but the cold concentrated acid commonly employed for dextrinating paper acts far too rapidly for the present purpose. It is therefore largely diluted with water and used hot. The degree of dilution is not of importance. I use, generally, four or five volumes of water to one of acid. If too weak, it rapidly concentrates by heat and soon reaches the point where it "cuts" nicely. I therefore generally use a mixture of waste sulphuric and nitric acids, which is to be had in abundance and at small cost in a nitration establishment. I place the acid in a pot or boiler of porcelain or other acid-proof material, and heat it nearly or quite to boiling. The rag being immersed in the hot acid is watched by the operator. As fast as it becomes tender he removes it and replaces it by fresh rag. It is not generally desirable that the boiling be continued till the fiber is completely broken down, for in that case it becomes more difficult to wash; but there is a point, easily known after one or two trials, where the action of the acid is sufficient, yet where the fiber is not so completely destroyed as to render the washing slow and tedious. This point is judged of by the tenderness of the rag. It should be as tender as possible, yet not reduced to pulp. If "overcut," it may still be used, but, as before stated, it becomes difficult to wash. After removal from the boiler the material is thoroughly washed, either with water or weak alkaline solutions, or both, till all acid is removed, the washing being in all cases completed with water. It is then thoroughly dried and cooled, and is ready for the next operation.

*The nitration.*—This operation somewhat resembles the preparation of nitro-glycerine, being conducted with cold acids, stirring and cooling. For the nitration I use a mixture of about six parts, by weight, of the strongest sulphuric acid, and two parts, by weight, of the strongest nitric, adding thereto one part, by weight, of the prepared material. It is desirable that these proportions be not widely departed from, for experience has shown that one part of the dry material requires not less than one and three-fourths part of strong nitric acid (48° to 50° Baumé) for its nitration, and requires at least seven parts of mixed acid to form a mass sufficiently liquid for pouring. The acids are, of course, mixed, and allowed to cool before using. The mixed acids may be placed in a tank and the dried material added and stirred in; but I prefer to draw into the tank alternately, or simultaneously and gradually, the mixed acid and the organic material, with a view to obtaining a product as nearly uniform as possible. Constant stirring is kept up during the operation, and care is taken that all material used be in a cool state, and that heating of the tank and its contents be pre-

vented by external cooling. In these respects the process resembles that ordinarily followed in the manufacture of nitro-glycerine. When all the materials have been introduced into the tank and the whole thoroughly stirred together dextrination continues and nitration commences, and both proceed to completion. The nitro-dextrine should now be in a state of quasi solution in the acids, resembling mush or thick ropy mucilage. If, however, it be so thick that it will not pour out readily, it must be thinned by the addition of more strong acid. The perfection of the result depends largely on the perfect management of the preliminary process described, termed by the workmen the "cutting;" for if the rag be not sufficiently cut it nitrates in the strong acid, but does not dextrinate, and consequently retains its original form of rag. It appears, in fact, that the process of dextrination must be fairly started in the preliminary operation, and that when so started it is continued in the strong acids, *pari passu* with the nitration. If, therefore, the rag be withdrawn from the cutting acids before it has become very tender it may yield some nitro-dextrine, but will give, principally, nitro-cellulose; whereas, if the cutting process be pushed sufficiently far complete dextrination will take place in the nitrating acids.

*The washing.*—The nitration process being finished, the mixture of acids and nitro-dextrine should be drawn off into water. Special precautions are here necessary to prevent decomposition of the nitro-compound; for though it appears in a state of semi-solution in the acid it precipitates immediately when drawn into water. It is therefore necessary—

First, that a somewhat large volume of water be used to receive the mixture—say about eight or ten times the amount of the acid mixture. Still more water is better. If the water becomes more than lukewarm during the washing its quantity is insufficient.

Second, that the water be violently agitated and beaten up while the mixture is flowing into it.

Third, that the mixture be not added to the water in a lump, but be poured in gradually, and not too rapidly.

Without these precautions there is certain to be more or less of overheating and decomposition. The nitro dextrine falls to the bottom of the washing-tank, whence it is withdrawn and washed and freed from acids by simple and well-known processes.

I regard a finely granular, pulverulent, and semi-crystalline appearance of the finished product as evidence that the cutting has been well conducted; and I regard the combustion of the finished article without leaving any notable amount of carbonaceous residue as evidence that a sufficient amount of strong nitric acid has been employed, and that the washing has been well conducted. The nitro-dextrine, however, is scarcely so combustible as the fibrous nitro compounds.

I am aware that heretofore desiccated vegetable material previously reduced to granules has been treated with strong sulphuric acid, washed, and then treated with a bath of nitric and sulphuric acids for the purpose of producing an explosive compound, and hence I lay no claim to the consecutive treatment with sulphuric acid, water, and mixed sulphuric and nitric acids, broadly, but confine my invention to the various consecutive steps comprising my precise process as described and claimed.

Having thus described my invention, what I desire to secure by Letters Patent is—

The process herein described for producing nitro-dextrine, consisting, essentially, in treating rag with warm dilute sulphuric acid to start dextrination, washing the rag, then submitting same to a mixture of sulphuric and nitric acids to produce the required nitration and dextrination, precipitating the product by water and then thoroughly washing it, as specified.

In witness whereof I have hereunto set my hand.

GILBERT S. DEAN.

Witnesses:
FRANK A. BROOKS,
WM. F. BOOTH.