UNITED STATES PATENT OFFICE.

EBENEZER KENNARD MITTING, OF CHICAGO, ILLINOIS.

PROCESS OF MAKING NITRO-GLYCERINE.

SPECIFICATION forming part of Letters Patent No. 457,002, dated August 4, 1891.

Application filed September 1, 1890. Serial No. 363,696. (No specimens.)

To all whom it may concern:

Be it known that I, EBENEZER KENNARD MITTING, a British subject, residing at Chicago, in the county of Cook and State of Illinois, have invented a certain new and useful Improvement in the Manufacture of Nitro-Glycerine; and I do hereby declare that the following is a full, clear, and exact description of the invention, which will enable others skilled in the art to which it appertains to make and use the same.

Hitherto nitro-glycerine has been produced by the cautious mixing together of thin streams of glycerine and the nitrating-acid, or by allowing the glycerine to flow in a small stream into the acid, suitable precautions being taken to intermingle the liquid without objectionable rise in temperature, as is well understood by those versed in the art. The nitrating-acid consists of a mixture of sulphuric and nitric acids in varying proportions; but usually a mixture of about two parts of sulphuric acid with one part of nitric acid is employed, and about eight to nine parts of this mixture are usually employed for the nitration of one part of glycerine.

Three varieties of nitro-glycerine are known to chemists—viz., the mononitro-glycerine represented by the formula $C_3H_5(OH_2)(O.NO_2,)$ dinitro-glycerine $C_3H_5(OH)(O.NO_2)_2$, and trinitro-glycerine $C_3H_5(O.NO_2)_3$,—and of these the trinitro is the only variety which is of practical utility as an explosive, and for the production of this the strongest or most concentrated nitric acid is required, otherwise the dinitro and the mononitro varieties are formed, and it is for this reason that the large proportion of sulphuric acid is added to the nitric acid employed, such sulphuric acid acting as an absorbent of the water generated and set free in the reaction, and thus preventing to a considerable extent the weakening of the nitric acid. The mixture of the glycerine and acid having been effected it is allowed to stand for a short time until the nitro-glycerine has separated from the "spent" acid and formed a supernatant layer. The spent acid is then drawn off, and in the best-conducted factories purified for use in the arts, and the nitro-glycerine is also drawn off to suitable receptacles, washed from impurities, and employed in the fabrication of high explosives.

The foregoing is an outline of the operation as usually conducted. It is, however, defective, owing to the fact that the last portions of the glycerine added to the acid are not converted into trinitro-glycerine by reason of the weakening of the acid mixture by the water formed in the reaction earlier in the operation, the presence of a comparatively large excess of anhydrous or nearly anhydrous nitric acid being essential to thoroughly convert the glycerine into trinitro-glycerine. Consequently the full theoretical yield of trinitro-glycerine (or a close approach thereto) is never obtained in practice, a certain loss always resulting from such portion of the glycerine which has not been converted at all or only converted into the mononitro variety, being dissolved in and carried away by the wash-water, while another and variable proportion may have been converted into the dinitro variety, and a portion of this may remain after washing, together with the bulk of the trinitro, reducing its specific gravity and explosive force. Various means have been proposed to overcome this defect and improve the yield of trinitro-glycerine. It was thought that the admixture of the acid with the glycerine in thin streams, allowing the whole to presently run into a collecting-tank, would overcome the difficulty; but this device did not succeed, owing to the fact that the reaction is not completed, except (as stated above) in the presence of a large excess of anhydrous nitric acid. Consequently the same conditions were brought about in the collecting-tank as obtained in the case of running the whole of the acid first into the tank and then adding the glycerine slowly—viz., that the last portions of the glycerine were not fully converted. Again, it was proposed to first treat the glycerine with only a portion of the usual amount of acid to remove the spent acid and then treat with the remainder, and for the better prosecution of this process to vary the quality of the acids used for the first and last treatment, and even to make the process a continuous one. So far as I am aware, this mode has not proved successful in practice, because it fell short of providing the necessary excess of nitric acid at the close of the operation. It has also been proposed to use double and treble the usual quantity of acid;

but this device has not been successful, and on the other hand the cost has been so largely increased as to be almost prohibitive.

The object of my invention is to overcome the difficulty set forth and explained above and to convert the whole or practically the whole of the glycerine into trinitro-glycerine, and thus produce a yield more nearly approaching the theoretical quantity and to effect this improvement without the use of additional acid beyond the usual quantity now employed.

To carry my invention into effect, I first proceed with the nitration of the glycerine in the usual manner—viz., by charging the nitrating-vessel with the mixed acid, say about eight parts, by weight, for every one part of glycerine to be nitrated, and at the close of the operation and after separation has taken place I draw off the spent acid in the usual manner. In the meantime I prepare another lot of mixed acid for the next succeeding lot of glycerine; but before using it for such next lot I run into and mix with it the nitro-glycerine produced from the first operation. The effect of this is to expose the nitro-glycerine produced in the first operation to the full effect of the large charge of anhydrous nitric acid intended for the second operation, and thus convert any of the lower nitro-glycerine into the trinitro variety. After allowing the mixture to settle I draw off the supernatant trinitro-glycerine to the washing-tanks and proceed with the nitration of the second lot of glycerine with the acid (originally intended for it) in the usual way, and the nitro-glycerine thus produced is in its turn fed into and mixed with the lot of acid for the next succeeding nitration, and so on continuously, using always the fresh acid first upon the last preceding lot of glycerine which has been nitrated, and then to nitrate a fresh lot of glycerine, as described. The fresh acid after acting upon the product of a previous nitration contains a little water, such water being that produced in the reaction, as will be readily understood. This, however, is of comparatively small amount and does not seriously affect the succeeding nitration, especially as same is in reality completed by exposure to the next lot of fresh acid.

I perform the foregoing operations in one nitrating-vessel by proceeding as follows: I first nitrate a charge of glycerine in said nitrating-vessel and allow the mixture of nitro-glycerine and spent acid to settle and separate, and then draw off the spent acid only, leaving the nitro-glycerine in the nitrating-vessel. I next run into that nitro-glycerine the charge of mixed acid intended for the next nitration, (this operation is performed comparatively quickly and with perfect safety under the usual precautions,) and having mixed the liquids I allow them to settle and separate and next draw off the supernatant nitro-glycerine (to be washed and otherwise dealt with) by a faucet fixed at a proper level or equivalent means, leaving the acid in the nitrating-vessel ready to receive a charge of glycerine, which I now run into it. I next allow the mixture to settle and separate, draw off the spent acid, and proceed to run into the nitro-glycerine the charge of fresh acid, as before, and so on in regular order, as described, or two or more nitrating-vessels may be employed and preferably fixed at different levels, as will be readily understood by those versed in the art. By working in this manner an increased yield of nitro-glycerine is obtained, of full specific gravity and explosive power, without increased quantity of acid beyond that usually employed and at only a slightly-increased cost for manipulation, which is more than repaid by the increased yield, quality, and safety, as the fully-converted trinitro-glycerine is far less liable to spontaneous decomposition than a mixture of such nitro-glycerine with lower nitro compounds.

Having now described my invention, what I claim, and desire to secure by Letters Patent, is—

1. The improvement in the method of manufacturing nitro-glycerine, which consists in first nitrating a charge of glycerine and separating the product from the spent acid, then treating said product anew with a fresh charge of nitrating-acid in excess, and finally separating the nitrated glycerine from the fresh excess charge of acid, substantially as described.

2. The improvement in the method of manufacturing nitro-glycerine, which consists in first nitrating a charge of glycerine and separating the product from the spent acid, then treating said product anew with a fresh charge of nitrating-acid, separating the nitrated glycerine from the acid, and employing the acid to nitrate a second charge of glycerine, substantially as described.

3. The improvement in the method of manufacturing nitro-glycerine, which consists in first nitrating a charge of glycerine and drawing off the spent acid, next treating the product with a fresh charge of nitrating-acid, then drawing off the nitro-glycerine and nitrating a fresh charge of glycerine with the same acid, and repeating the operation in the same nitrating-vessel, substantially as described.

EBENEZER KENNARD MITTING.

Witnesses:
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