

UNITED STATES PATENT OFFICE.

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METHOD OF MAKING UNFREEZABLE EXPLOSIVES.

1,029,519.

Specification of Letters Patent.

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To all whom it may concern:

Be it known that I, VEZIO VENDER, a subject of the King of Italy, residing at Milan, Italy, have invented certain new and useful
5 Improvements in Methods of Making Unfreezable Explosives, of which the following is a specification.

The present invention has for its object primarily to provide a method of making
10 practically unfreezable explosives having a plastic and horny consistency and a low explosive temperature.

At the present time it is regarded as essential that explosives and smokeless powder
15 have a plastic and horny consistency, but such substances as have been heretofore made containing nitro-glycerin are objectionable in that they freeze at a temperature below 50° F., they lose their valuable prop-
20 erties and they are also slow of ignition and are very dangerous.

The present invention relates to the preparation and the use of nitrated esters of glycerin with formic or acetic acid, nitro-
25 formin or nitracetin for making practically unfreezable explosives or for lowering the freezing point of nitro-glycerin explosives. The use of nitracetin and nitro-formin, for the purpose just described, is particularly
30 advantageous over processes heretofore known in that they are not volatile substances but are very permanent, they do not contain chlorin, they are not hygroscopic and, moreover, are easy to make.

By nitrating acetins (containing free OH) with ordinary nitro-sulfuric acid for making nitro-glycerin, a very poor yield of nitro-glycerin is obtained which moreover
35 contains little nitracetin. It has been found that the sulfuric acid of the acid mixture separates acetyl, and that on the other hand, a good yield of pure nitracetin can be obtained by nitrating the basic acetins with
40 nitric acid of more than 65% NO_3H or with nitro-sulfuric acid containing more nitric than sulfuric acid, and that a good yield of mixtures of nitracetin and nitro-glycerin can be obtained by nitrating acetin-glycerin
45 mixtures, if acid mixtures are employed containing as much more nitric acid than sulfuric acid as there is acetin in proportion to glycerin. By obtaining pure nitracetins, as mentioned above, it has been discovered that these nitracetins possess certain
50 valuable properties which are of especial

utility in the manufacture of explosives, that is to say, it has been found that nitracetin does not freeze even at very low temperatures nor does nitracetin freeze at very low temperatures when mixed with nitro-glycerin; that nitracetin is extremely non-
60 susceptible to heat and shock and it is so great a solvent for gun cotton even when cold that gun cotton of 13.4% N may be gelatinized. Moreover, dinitracetin can not
65 be exploded or ignited by means of a hammer or by striking iron against iron, but when exploded with a suitable primer, it possesses an explosive power as great as
70 nitro-glycerin although it has a lower explosion temperature. Nitro-formins heretofore unknown can also be obtained by nitrating formin, and it has been found that these nitro-formins have a great explosive
75 power and they also lower the freezing point of nitro-glycerin to such an extent that a mixture for instance of about 25% dinitro-formin with nitro-glycerin may be regarded as practically unfreezable.

More specifically, the invention may be
80 disclosed more clearly by the following which are given as examples:

Example No. 1: 100 kgs. of glycerin (99%) are heated with 20 kgs. of anhydrous oxalic acid at 100° C. and the mixture is
85 then heated for about 20 hours at a temperature ranging between 140° and 150° C., the product obtained which consists of about 125 kgs. of a mixture of formin and glycerin is directly nitrated in 275 kgs. of nitric acid
90 (96%) and 90 kgs. of fuming sulfuric acid (25% SO_3) at 25° C. after which the oil separated after washing with water and a warm soda solution is of a pale yellow color and has a specific gravity of 1.57 at 15° C.
95 and about 15.7% N corresponding to a mixture of 33% dinitro-formin and 67% trinitroglycerin. 100 kgs. of this nitro-glycerin mixture is gelatinized with 10 kgs. of collo-
100 dion cotton to obtain a blasting gelatin or with 200 kgs. of nitro-cellulose to obtain horny explosives; the blasting gelatin or
105 horny explosives thus obtained remaining unfrozen even at a temperature of minus 17° C.

Example No. 2: 100 kgs. of glycerin (99%) and 75 kgs. acetic acid (99%) are boiled for about 24 hours and acetic acid is
110 distilled off by heating to 240° C. Of the raw monacetin obtained, 125 kgs. are ni-

trated with 340 kgs. of nitric acid of 95% and 110 kgs. fuming sulfuric acid (25% SO_3) at 25°C .; the whole is poured into water and the separated oil washed with
5 water and a warm soda solution. A pale yellow oil having a specific gravity of 1.45 at 15°C . is obtained which is insoluble in water, benzin and sulfid of carbon, soluble in nitric acid, alcohol, acetone and nitro-
10 glycerin; it contains 12.5% N, it does not explode under the hammer, but it may be exploded with a suitable percussion cap, 10 grams of the explosive causing in the
15 Tranzyl test an expansion of about 450 cubic centimeters.

I claim as my invention:

1. A method of making mixed esters of glycerin, consisting in nitrating a suitable

basic glycerin ester of a monobasic acid of the acetic series with acid containing not less
20 than sixty-five per cent. of nitric acid to obtain practically unfreezable explosives.

2. A method of making mixed esters of glycerin, consisting in nitrating a suitable
25 basic glycerin ester of a monobasic acid of the acetic series with a mixture of nitric and sulfuric acid containing more nitric than sulfuric acid.

In testimony whereof I have hereunto set my hand in presence of two subscribing wit-
30 nesses.

VEZIO VENDER.

Witnesses:

N. LYLE ROBB,
CHAS. H. FLISCHER.