

# UNITED STATES PATENT OFFICE.

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## PROCESS OF MAKING NITROGLYCERIN.

No. 804,817.

Specification of Letters Patent.

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Continuation of application Serial No. 161,694, filed June 16, 1903. This application filed February 4, 1904. Serial No. 191,961.

*To all whom it may concern:*

Be it known that I, CHARLES L. REESE, a citizen of the United States, residing at Wilmington, county of Newcastle, and State of Delaware, have invented a new and useful Improvement in the Manufacture of Nitroglycerin and Treatment of Materials Used in the Manufacture of Nitroglycerin, of which the following is a full, clear, and exact description.

10 In the manufacture of nitroglycerin, as is well known, the glycerin is admitted into a bath of a mixture of nitric and sulfuric acids, in which bath the nitration takes place, and the nitroglycerin is formed and suspended in the remaining acids in the form of an emulsion. This emulsion of nitroglycerin and acids is allowed to stand in a suitable vessel, into which it is run after nitration to allow the globules of nitroglycerin to collect together and rise to the surface of the acids, while the acids settle by gravity. This process of separation is in many cases extremely sluggish, often the proper amount of nitroglycerin not being recovered and often requiring the subsequent drowning of the charge or a part of the charge, causing considerable waste. The item of time caused by the sluggish separation of nitroglycerin is a considerable factor, as it adds materially to the cost and danger of the manufacture. It is also well known that emulsions are made and held more permanent by the use of gelatinous or colloidal substances, and after many experiments I have discovered that when gelatinous or colloidal silica is present in glycerin in the form of soluble silica or a soluble silicate or as a glycerosol or a glycerogel or when soluble silica is present in the acids used the time of separation is increased very materially.

40 In many cases the glycerin or the acids contain some one or more of these or similar substances, and very minute quantities are sufficient to retard the separation. I have discovered that if I add a reagent which will dissolve or change the silicious or other colloidal material to such a form as to neutralize their emulsifying effects the rapidity of separation out of nitroglycerin will be materially increased. I have found that if I even add soluble silica to the glycerin or mixed acids or the separate acids and at the same time add the proper reagent, such as fluorid or hydrofluoric acid, so as to combine

with the silica forming silica tetrafluorid the time of separation will be materially shortened. I have used many reagents for this purpose, working principally with the halide group. I have had excellent results using a compound of fluorin. Preferably I use a fluorid of the alkalies and of these sodium fluorid. In practice I have added the reagent to the glycerin before its entry into the nitrator, or in the process of manufacture to the separate acids, (sulfuric and nitric,) to these acids when mixed, and to the acids when admixed in the nitrator, and either before or after the glycerin has been added. Preferably I add it either to the glycerin before entering into the nitrator or to the admixture of glycerin and acids in the nitrating-tank. As is well known, the nitrating-tank is provided with arrangements for stirring or agitating, which enables the reagent added to be thoroughly disseminated throughout the mass. I have also added the reagent to both the glycerin and the acids before their entry into the nitrator, which is often advisable, and I consider my invention involved where the addition is made to both the acids and glycerin. As to the quantity of the reagent I have found an addition of one thousandth of one per cent. (.001%) of sodium fluorid added to glycerin before entering into the tank and the same quantity after admixture in the tank to give good results; but I do not intend to limit myself to this amount or method of addition. I give the same only as an example of an amount and method which may be used.

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

1. The hereinbefore-described method of accelerating the separation of nitroglycerin, in the manufacture of nitroglycerin, which consists in adding, during the manufacture of nitroglycerin, to the materials used in the manufacture of nitroglycerin, a reagent which will reduce any emulsifying or colloidal substance contained therein to a non-emulsifying or non-colloidal condition.

2. The hereinbefore-described method of accelerating the separation out of nitroglycerin in the manufacture of nitroglycerin which consists in adding, during the manufacture of nitroglycerin, to the materials used in the manu-

facture of nitroglycerin, a compound of fluorin.

3. The hereinbefore-described method of accelerating the separation out of nitroglycerin, in the manufacture of nitroglycerin, which consists in adding, to the materials used in the manufacture of nitroglycerin, during the manufacture of nitroglycerin, a fluorid of the alkalies.

4. The hereinbefore-described method of accelerating the separation of nitroglycerin, in the manufacture of nitroglycerin, which consists in adding, during the manufacture of nitroglycerin, both to the acids and glycerin, a reagent which will reduce any contained emulsifying or colloidal substance to a non-emulsifying or non-colloidal condition.

5. The hereinbefore-described method of accelerating the separation out of nitroglycerin in the manufacture of nitroglycerin which consists in adding, during the manufacture of nitroglycerin, to both the acids and glycerin, a compound of fluorin.

6. The hereinbefore-described method of accelerating the separation out of nitroglycerin, in the manufacture of nitroglycerin, which consists in adding, during the manufacture of ni-

troglycerin, to both the acids and glycerin, a fluorid of the alkalies.

7. The hereinbefore-described method of accelerating the separation of nitroglycerin in the manufacture of nitroglycerin which consists in admixing the acids and glycerin in association with a reagent which will change emulsifying or colloidal substances to a non-colloidal condition.

8. The hereinbefore-described method of accelerating the separation of nitroglycerin in the manufacture of nitroglycerin which consists in admixing the acids and glycerin in association with a compound of fluorin.

9. The hereinbefore-described method of accelerating the separation of nitroglycerin in the manufacture of nitroglycerin which consists in admixing the acids and glycerin in association with a fluorid of the alkalies.

In testimony of which invention I have hereunto set my hand, at Philadelphia, on this 30th day of January, 1904.

CHARLES L. REESE.

Witnesses:

M. M. HAMILTON,  
WILLIAM B. MARKS.