

UNITED STATES PATENT OFFICE.

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EXPLOSIVE.

994,841.

Specification of Letters Patent. Patented June 13, 1911.

No Drawing.

Application filed June 15, 1910. Serial No. 566,934.

To all whom it may concern:

Re it known that I, HAROLD HIBBERT, a subject of the King of Great Britain, residing at Wilmington, county of Newcastle, and State of Delaware, have invented a new and useful Improvement in Explosives, of which the following is a full, clear, and exact description.

When glycerin is treated with the mixed acids, nitric acid and sulfuric acid, for the production of nitro-glycerin, nitro-glycerin is produced, the formula of which is:



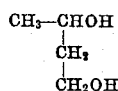
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This nitro-glycerin has certain defects, among which it may be stated that the glycerin which is its base is expensive, and the temperature of explosion of the explosive mixture of the nitro-glycerin is very high, which makes it in many cases not a good ingredient for smokeless powder.

I have discovered that if butylene glycol nitrate be formed, a product is obtained whose temperature of explosive ignition is lower than that of nitro-glycerin, which enables it to be freely used as an ingredient of smokeless powder, safety explosives and dynamite. Further, I have discovered that such compound may be combined with nitrates or nitro compounds, such, for instance, as examples of nitrates nitro-cellulose or nitro starch, to form a colloid of such mixtures. It also may be mixed with nitro-glycerin, and the resulting mixture has a lower freezing point and is more stable than nitro-glycerin.

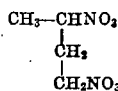
While butylene glycol is known and is described in text books, I believe myself to be the first to have discovered nitrated-butylene glycol and its value for the purposes named. Further, the method of making butylene glycol, as set out in the text books, is so complicated as to render it very expensive and difficult to carry out. My discovery, therefore, includes a simple method for directly producing this butylene glycol as well as the nitrated butylene glycol.

The butylene glycol is represented in formula in the following manner:



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and when nitrated the formula is thus represented:



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I produce the butylene glycol in the following manner: I take ethyl alcohol ($\text{C}_2\text{H}_5\text{OH}$), which may be de-natured without affecting the result. This ethyl alcohol is first oxidized. Preferably I accomplish this oxidation by the ordinary catalytic oxidation from the air, such for instance, by using metallic platinum or copper and heat. The resultant is acetaldehyde ($\text{C}_2\text{H}_4\text{O}$). This acetaldehyde is then condensed. I have used for this condensation an aqueous solution of an alkaline carbonate, for instance, potassium carbonate; 450 grams of acetaldehyde were cooled to -5°C . and 200 grams of powdered ice then added. A few drops of a cooled solution of potassium carbonate containing 19 grams of the carbonate in 60 cubic centimeters of water were introduced into this mixture the temperature not being allowed to rise above $+10^\circ\text{C}$. After standing some 10 or 15 minutes a further quantity of the carbonate solution was introduced and again allowed to stand, contents being kept as nearly as possible at a temperature of 0°C . After the whole of the carbonate had been added, the reaction-product was kept in ice water for several hours to prevent any rise of temperature and then for a further 20 hours at $+8$ to $+10^\circ\text{C}$. If the pure acetaldol is required this may be extracted with ether, but for the purpose of obtaining butylene glycol this is unnecessary. The condensation can be produced by other alkaline carbonate or a borate, phosphate, acetate or cyanid, of the alkaline metals or alkaline earths. The resultant of this condensation is acetaldol ($\text{C}_4\text{H}_8\text{O}_2$). This condensation product is

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then reduced to butylene glycol by means of a reducing agent, and I have employed the following method: The aqueous solution of acetaldol, obtained by condensing

5 450 grams of acetaldehyde with potassium carbonate was diluted to six liters with ice-water, rendered neutral by the addition of hydrochloric acid and then 142 grams of aluminum amalgam added. After standing

10 for two days, the aluminum hydroxid was filtered off and a further 58 grams of the amalgam added, and the mixture left for a further two days. After filtering the solution, the butylene glycol was recovered

15 by evaporating off the water under reduced pressure. Other amalgams may be used and, further, the aluminum or other metal or an alloy or several metals may be used and also without the use of mercury. In

20 this condition the butylene glycol exists in an aqueous solution and is isolated by simple distillation, preferably under vacuum. This butylene glycol may then be nitrated in the same manner that glycerin is nitrated, by means of nitric acid and sulfuric

25 acid in the following manner: 350 grams of ordinary nitrating acid such as is used for the nitration of glycerin were cooled to -5° C. and then 50 grams of butylene glycol slowly introduced into this, under the

30 surface of the liquid in a fine spray, the acid mixture being kept at -5° C. and agitated vigorously throughout the addition. The butylene glycol dinitrate at once rises

35 to the surface as a yellow oil and may be separated without further difficulty from the acid mixture. The resultant nitrated compound may then be used by itself or mixed with a nitrate or nitrates, as for instance, nitroglycerin, nitro-celluloses or

40 nitro-starch. Indeed, I have found it quite valuable when mixed with nitroglycerin as it then lowers the freezing point of nitroglycerin.

45 Butylene glycol nitrate has great stability, its temperature of ignition is lower than that of nitro-glycerin, it is non-hygroscopic, it has a low freezing point, and is very stable toward acids and alkalis. Its

50 insolubility in nitrating acids renders its separation after nitration a very simple matter. When mixed with nitro-glycerin it lowers the freezing point and promotes its stability. It colloids nitro-cellulose

55 more effectively than does nitro-glycerin and such mixtures are very stable.

The following are examples of admixtures containing butylene glycol nitrate:—

Butylene glycol nitrate, 1 part by weight; nitro-glycerin, 3 parts by weight. 60

Butylene glycol nitrate, 3 parts by weight; nitro-starch, 1 part by weight.

Butylene glycol nitrate, 1 part by weight; nitro-glycerin, 2 parts by weight; nitro-starch, 1 part by weight. 65

Butylene glycol nitrate, 1 part by weight; nitro-glycerin, 2 parts by weight; nitro-cellulose, 1 part by weight.

Butylene glycol nitrate, 1 part by weight; nitrocellulose, 1 part by weight. 70

Butylene glycol nitrate, 1 part by weight; nitrocellulose, 2 parts by weight; nitro-glycerin, 1 part by weight.

When I speak of nitro-glycerin, of course I intend to include the mono- and di-nitro-glycerins, although I have shown only the formula of the tri-nitro-glycerin. In like manner, when I use the term nitro-butylene glycol I intend to include the mono-nitro-butylene glycol and the di-nitro-butylene glycol, although I have only shown the formula of the di-nitro-butylene glycol. 75

I do not herein claim the method of producing butylene glycol, as the same forms the subject matter of an application filed 85 September 15, 1910, Serial No. 582,144, a division of this application. Nor do I claim herein specifically the explosive wherein a nitro derivative is used with nitrated butylene glycol, as this also is claimed in an application filed by me September 15, 1910, Serial No. 582,145. 90

Having now fully described my invention, what I claim and desire to protect by Letters Patent is:— 95

1. An explosive containing butylene glycol nitrate.

2. An explosive containing an admixture of butylene glycol nitrate and a nitrate.

3. An explosive containing an admixture 100 of butylene glycol nitrate and nitrates.

4. An explosive containing an admixture of butylene glycol nitrate and nitro-glycerin.

In testimony of which invention, I have 105 hereunto set my hand, at Wilmington, on this 7th day of June, 1910.

HAROLD HIBBERT.

Witnesses:

J. ED. WILSON,
J. A. CASEY.