UNITED STATES PATENT OFFICE.

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METHOD OF PRODUCING EXPLOSIVES CONTAINING NITRO DERIVATIVES OF COMMON RESIN.

1,043,042. Specification of Letters Patent. Patented Oct. 29, 1912.

No Drawing. Original application filed February 12, 1910, Serial No. 543,534. Divided and this application filed July 31, 1912. Serial No. 712,482.

To all whom it may concern:

Be it known that I, OSCAR BIRGER CARLSON, a subject of the King of Sweden, and resident of Månsbo, Avesta, in the Kingdom of Sweden, have invented certain new and useful Improvements in the Method of Producing Explosives Containing Nitro Derivatives of Common Resin, of which the following is a specification.

The present invention is a division of my application Serial No. 543,534, filed February 12, 1910.

In the manufacture of explosives suitable oxygen carriers, such as nitrates, chlorates and perchlorates, have been used for a long time. However, the combustibles hitherto employed, have not been quite satisfactory and this is why no considerable competition has taken pla[illegible] between explosives containing a combust[illegible]e and an oxygen carrier and such explosives which are based on the use of nitroglycerin and nitrocellulose. Hitherto several explosives containing a combustible and highly oxidized salts as oxygen carriers have no doubt been produced, and some of them have also been usable in many cases, but as to explosive power they have, however, been inferior to dynamite and similar explosives. On account of their low specific gravity they have a low quantity of energy per unit of volume (in the following called density of energy), and, moreover, they lack the plasticity of dynamite, this plasticity or elasticity making it possible to press the dynamite into the bore-hole, so that it fills up all unevennesses in the same, whereby its effect is furthermore increased.

Several nitro compounds, especially aromatic compounds, have hitherto been used as combustibles in explosives, but hereby neither plasticity nor high specific gravity is attained. An explosive which may be used for some purposes can be obtained by coating the grains of the oxygen carrier with the nitro derivative of resin. This explosive obtains a certain degree of plasticity because the slippery grains slide in all directions, when they are exposed to pressure; the density of energy is, however, low. The volume weight of such an explosive, having for instance potassium chlorate as a base, is only about 0.8, while that of dynamite is about 1.5.

The present invention refers to a method of producing a combustible which, mixed with a suitable quantity of an oxygen carrier, forms an explosive with high density of energy and which may also if desired, be given the required plasticity. It has been found that, by the action of nitro derivatives of common resin in crude or pure state upon organic nitro compounds, tough and viscous products with low fusing points are formed. The reaction is executed at an increased temperature of at least 40° C. and for a time of at least a number of hours or often days. By choosing suitable nitro compounds and by suitably varying the proportions of the same the toughness of the product may be varied as desired and the fusing point may also be varied between the freezing-point and higher temperatures. A common characteristic of all these compositions is that their physical and chemical properties are different from those of their constituents: All of them are dark brown, very viscous liquids which at rising temperatures gradually become more thin, and which are easily absorbed by pulverulent salts, but not at all or very little by paper, the latter not being acted upon by the same (in distinction from nitro compounds). Other phenomena at the production of the present combustibles also indicate that not only an ordinary dissolving action takes place, but also a slow chemical reaction between the nitro derivatives of common resin on one side and the organic nitro compounds employed on the other. A development of gas takes place when the said products are fused though the temperature is generally held at 60°–70° Cent. and the inventor has in no case used higher temperatures than 90°–100° Cent. No distillation of the constituents can cause the development of gas. Furthermore, the properties of the product will not be constant before it has been heated to the fusing point during a long time, varying from some hours to some days. The fusing point of the mass varies within wide limits: In certain cases the same may rise during the heating, but generally it descends. Other properties of the product also change.

As to the product formed by the chemical reaction the composition of the same is

not known, nor is it known which constituents of nitro derivatives of common resin take part in the reaction, and it is supposed to be very difficult to determine this, especially in consideration of the fact that the composition of many constituents of the nitro derivatives of common resin is not known. However, it seems to be evident, that all the constituents of the nitro derivatives of common resin do not take part in the said reaction, but that the mass obtained forms a solution of the products of the reaction in the unchanged constituents. This seems to be clear by the fact that sometimes certain constituents crystallize out prior to other constituents, and a mass of buttery character is formed.

Real nitrated common resin may be produced in many ways but oxidation will, to some extent at least, take place during the process. It is generally desired to limit this oxidation as much as possible, although nitrated resin containing oxidized resin may also be employed. While, by nitrating the resin, excluding as far as possible oxidation, an increase in weight of 30 to 40 per cent. is obtained, an essential loss of substance and energy (production of heat) is caused when the same is oxidized. Thus from an economic as well as from an energetic point of view it will be desirable to prevent oxidation as far as possible. The resin may be nitrated as follows.

1. Pulverulent resin is gradually stirred into an excess of nitric acid (sp. gravity about 1.4) without permitting the temperature to rise far above the ordinary temperature of the room. If the resin is introduced so quickly that the temperature rises far above the temperature of the room, too much oxidation will take place and several oxy-acids will be formed, for instance, trimellitic $C_6H_3(CO_2H)_3$; terebinic $C_7H_{10}O_4$; isophthalic $C_6H_4(COOH)_2$, etc.

2. To 5 kilograms of coarse ground resin within a vessel of stone ware, add while stirring, 3 liters of nitric acid (sp. gravity 1.35) heated to about 75° to 80° C. In the same manner, a mixture of 90% of resin and 10% of meal may be treated. If the process is carried on carefully, a bright yellow resin-like, very light, porous and spongy product is obtained, which, on account of its brittleness, may easily be pulverized, the said product readily reacting with other nitrated substances.

Some examples of combustibles according to the present invention are cited in the following:

1. 1 kilogram of 2.5-dinitrotoluene+1 kilogram of nitro derivatives of common resin (fusing point 150°). This combustible is between 20°-30° a viscous liquid, which solidifies at 18°.

2. 1 kilogram of 2.5-dinitrotoluene+1 kilogram of nitro derivatives of 90 parts of common resin and 10 parts of meal of wheat (fusing point 180°). Solidifies at 18°.

3. 1 kilogram of 2.4-dinitrotoluene+1 kilogram of nitro derivatives of common resin. Solidifies at 75°.

4. 9 kilograms of 2.5-dinitrotoluene+2 kilograms of o-nitrotoluene+9 kilograms of nitro derivatives of common resin; fusing point 32°.

5. 1 kilogram of 2.4-dinitrotoluene (fusing point 70.4°)+1 kilogram of 2.5-dinitrotoluene (fusing point 52°)+1 kilogram of nitro derivatives of common resin; (fusing point about 180°). The product is rather thin, but viscous at ordinary temperature, solidifies at +12°.

6. 2 kilograms of 2.4-dinitrotoluene+4 kilograms of 2.5-dinitrotoluene+1 kilogram of trinitrotoluene+1 kilogram of m-dinitrobenzene+7 kilograms of nitro derivatives of common resin.

7. 1 kilogram of 2.4-dinitrotoluene+2 kilograms of 2.5-dinitrotoluene+1 kilogram of m-dinitrobenzene+4 kilograms of nitro derivatives of common resin; does not solidify at the freezing point.

8. 1 kilogram of 2.4-dinitrotoluene+1 kilogram of nitrotoluene+1 kilogram of nitro derivatives of common resin; solidifies at +2°.

9. 2 kilograms of 2.4-dinitrotoluene+1 kilogram of trinitrotoluene+2 kilograms of nitro derivatives of common resin; solidifies at +32°.

10. 1 kilogram of 2.4-dinitrotoluene+1 kilogram of m-dinitrobenzene+2 kilograms of nitro derivatives of common resin.

11. 5 kilograms of 2.5-dinitrotoluene+5 kilograms of azobenzene+9 kilograms of nitro derivatives of common resin+1 kilogram of nitro derivatives of meal of wheat; the product is viscous at ordinary temperature.

In the indicated examples 1-11 the reaction is effected at a temperature of 60-70° C., but the same may be allowed to increase to 90-100° C. without inconvenience. If this temperature be exceeded, dark, burned and charred products will easily arise which do not dissolve in the smelting but form solid grains. An explosive produced from such a burned combustible does not keep unaltered when stored up but it gets hard after some time, and it may only with difficulty be brought to explosion. Consequently, it is of the greatest importance that the temperature used in the production of the combustible be not allowed to increase so much that such burned products arise. On the other hand it is also important that the heating becomes high enough and is continued so long that the said chemical reaction which takes place slowly between ni-

trated resin and organic nitro-compounds be accomplished, as otherwise neither the qualities of the combustible nor those of the explosive become stable and constant.

By varying the percentage of the constituents in the mixture explosives can be obtained which are adapted to different climates, seasons, etc., and which are plastic or not. Some examples of explosives, containing a combustible according to the present invention and an oxygen carrier are indicated in the following:

1. 1 kilogram of combustible No. 4+4 kilograms of ammonium perchlorate; this explosive is plastic and tough at ordinary temperature; specific gravity 1.71.

2. 2 kilograms of combustible No. 4+7 kilograms of potassium chlorate; this explosive is plastic and tough, even at low temperatures; specific gravity 1.84.

3. 1 kilogram of combustible No. 4+3 kilograms of potassium perchlorate; this explosive is plastic and tough; specific gravity 2.02.

The production of explosives of the combustibles according to the present invention is very simple and requires simple apparatus on account of the fact that the mixing of the constituents and the molding of the mixture is very much facilitated by the doughy character of the product. If the explosives are adapted to be exported to countries with hot climates it may, for instance, be favorable to transport the constituents of the same separately and to mix them together just before they are to be used.

Explosives produced according to the present invention can be caused to explode only by very powerful impulses. When ignited they burn calmly and are very insensitive to strokes. Hereby the danger connected with the producing and employment of the same is reduced to a minimum.

The combustibles produced according to the present invention are not soluble in water, and, thus, the explosives made of the same become sufficiently waterproof. It is to be observed that the combustibles according to the present invention are much cheaper than the nitro compounds which have hitherto been used in the manufacture of explosives.

The term "an organic nitro compound" in the claims is intended to cover a mixture of two or more organic nitro compounds as well as one only.

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

1. The method of producing combustibles for explosives comprising heating a mixture of a nitro derivative of common resin and an organic nitro compound to a temperature of at least 40° C. for a time of at least a number of hours, substantially as described.

2. The method of producing explosives consisting in heating a mixture of a nitro derivative of common resin and an organic nitro compound to a temperature of at least 40° C. for a time of at least a number of hours and adding a solid oxidizing salt, substantially as described.

In witness whereof, I have hereunto signed my name in the presence of two subscribing witnesses.

OSCAR BIRGER CARLSON.

Witnesses:
GRETA GIVEN,
AXEL EHRNER.