

F. S. WASHBURN.
 PROCESS OF MAKING CALCIUM CYANAMID.
 APPLICATION FILED JUNE 27, 1911.

1,009,705.

Patented Nov. 21, 1911.

Fig. 1.

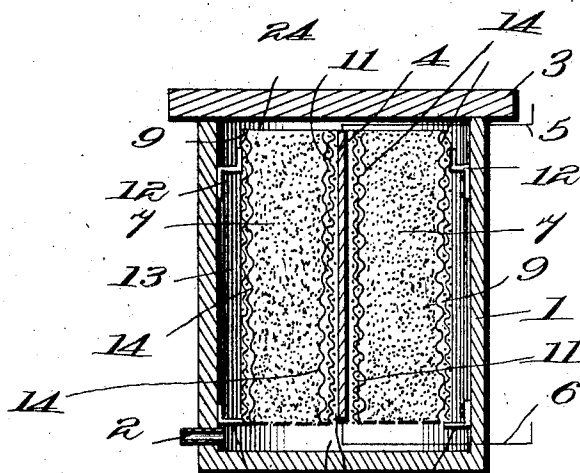
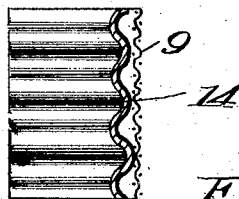
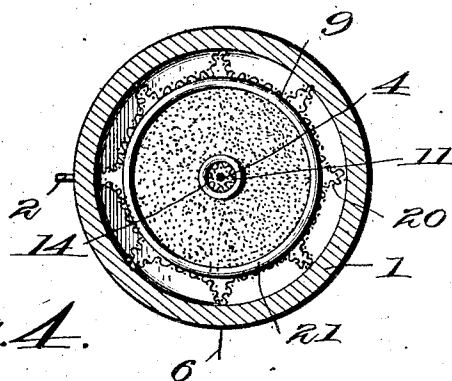
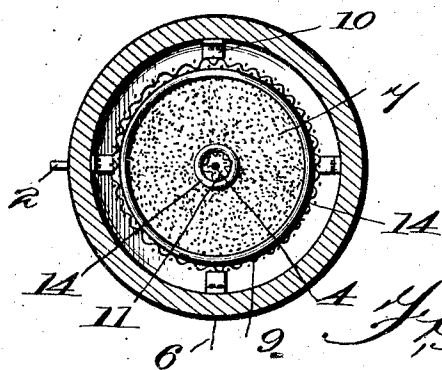


Fig. 2. *Fig. 3.*



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PROCESS OF MAKING CALCIUM CYANAMID.

1,009,705.

Specification of Letters Patent. Patented Nov. 21, 1911.

Original application filed March 4, 1910, Serial No. 547,357. Divided and this application filed June 27, 1911. Serial No. 635,688.

To all whom it may concern.

Be it known that I, FRANK S. WASHBURN, a citizen of the United States, residing at Nashville, in the county of Davidson and State of Tennessee, have invented certain new and useful Improvements in Processes for Making Calcium Cyanamid; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to a process of producing calcium cyanamid in ovens or retorts and has for its object the improvement of the method of compelling the reaction between the nitrogen and carbid, and for holding the same in position during its manufacture, thereby not only improving the uniformity of the product but also producing a greater yield of calcium cyanamid.

In the retorts heretofore employed the central open space through the mass of carbid has not been as efficient as has been desired in affording a ready means for the nitrogen gas to attack the finely divided carbid, and, therefore, it has been found desirable in practice to improve this means. And, further, in the older methods, after the reaction is once started any passages which might have existed between the outer walls of the mass of carbid and the inner walls of the retort were soon more or less obstructed and, therefore, the free passage of the nitrogen in a measure cut off.

This invention is a division of my prior, co-pending application #547,357, filed March 4, 1910, entitled Apparatus for producing calcium cyanamid, now Patent No. 999,071, dated July 25, 1911, and it obviates these objections, and consists in the novel steps constituting the process more fully hereinafter set forth and particularly pointed out in the claims.

Referring to the accompanying drawings forming a part of this specification, in which like numerals refer to like parts in all the views:—Figure 1, is a diagrammatic view illustrating in longitudinal section, an oven suitable for carrying out this invention; Fig. 2, is a transverse sectional view of the parts shown in Fig. 1; Fig. 3, is a view similar to Fig. 2, but illustrating a slightly modified

form of means for holding the carbid; and, Fig. 4, is a sectional view of a portion of the foraminous vessel.

1 illustrates any suitable retort or vessel, 2 any suitable means for introducing nitrogen gas into the vessel 1, 3 any suitable closure for said vessel, and 4 any suitable means for creating the desired temperature inside the vessel, in this instance shown as a resistance heater connected to the lead wires 5 and 6.

As stated above, it has heretofore been customary to provide centrally through the mass of carbid 7 a space 8 through which the nitrogen gas passed and attacked the carbid all around the same. But it has been found in practice that the passages of the nitrogen into the interior of the mass soon became clogged with the result that the reaction was found to have been imperfect in the center of the mass. To obviate these objections, I provide a suitable foraminous can or vessel 9 preferably made of wire cloth and suitably supported as by the brackets or other supports 10 on the interior of the vessel 1. The bottom of the foraminous vessel 9 may be of the same material as the outer wall or of any other suitable foraminous or perforated substance. Through the center of the vessel 9, I provide a foraminous hollow tube 11 inclosing the space 8 through which nitrogen may be freely passed, and if a resistance heater such as 4 is employed said heater may also be located in said tube 11, as illustrated. The vessel 9 is preferably held away from the inside of the vessel 1 by brackets, such as 12 in order to leave an annular space 13 in which the nitrogen may freely circulate.

The calcium carbid is preferably used in a finely divided condition, and, therefore, it is liable to find its way through the walls of the vessel 9 and tube 11. In order to prevent this, I prefer to line the interior of the vessel 9 and to cover the exterior of the tube 11 with layers of corrugated paper 14, as illustrated.

In the form shown in Fig. 3, instead of the brackets 12 I may corrugate the wire netting 9 as at 20 and permit the outwardly projecting portions of the same to contact with the interior wall of the retort 1, as will be clear from an inspection of said

figure. In such case the paper 21 may be smooth, as indicated, or it may be corrugated, if desired.

The operation of the retort will be obvious from the foregoing but may be briefly summarized as follows:—Upon admitting nitrogen through the tube 2 and upon turning on the current through the resistance heater 4, or upon otherwise raising the temperature of the interior of the vessel 1 to the required degree, the now well known reaction between the nitrogen and carbid will take place. This reaction, as it well known, is strongly exothermic and when once started, the reaction will continue either without the aid of heat or with some slight assistance therefrom until the entire mass is transformed into calcium cyanamid and its accompanying by-products, chiefly free carbon and free lime. As the temperature rises the paper 14 or 21, as the case may be, will become charred so that the nitrogen is freely admitted through the same, while at the same time, the powdered carbid now partially converted into calcium cyanamid will not pass through the perforations of the vessel 9. The spaces 8 and 13 being kept always open by the wire cloth will afford free passages for the nitrogen, and thereby a means throughout the reaction by which the nitrogen may pass through the entire mass of powdered carbid. In fact, it is preferred to also maintain spaces 23 at the bottom and 24 at the top of the can 9 so that the nitrogen may at all times freely pass completely around as well as through the center of the mass of carbid. It results from this structure that the reaction is not only much quicker than has heretofore been the case, but it is very much more perfect and the yield is also much greater than it has been possible to attain with the old style of retort. This is shown by the fact that in actual practice when using a screen made of wire of about one-sixteenth of an inch in diameter, woven so as to form a mesh of about three-sixteenths of an inch square and employing 89 retorts without these screens and 34 retorts with these screens, the conditions being otherwise identical, the cans without the screens resulted in 58.5 hours in an average of combined nitrogen amounting to 17 per cent. of the weight of their contents. On the other hand, the retorts with the screens, under identical conditions, resulted after 45.6 hours in an average of combined nitrogen amounting to 21.1 per cent. of the weight of their contents. In addition to this, the cans with no screens showed comparatively wide variations be-

tween high and low percentages of combined nitrogen, while the minimum amount of combined nitrogen in any of the screened cans was 21 per cent., thus showing the great uniformity of the product with the new retorts as compared with the old retorts. The difference between these results is made clearer when it is stated that the retorts without screens combined the nitrogen at the rate of 3.15 pounds per hour, while the cans with screens combined the nitrogen at the rate of 4.72 pounds per hour, which is a gain of about 53 per cent. over the old process.

It is clear that this process may be carried out by apparatus entirely different from that disclosed, for that its essential characteristics consist in maintaining at all times a free passage for the nitrogen through and around the mass of carbid, while, of course, providing a means for the nitrogen to readily enter the body of the mass of carbid. It is therefore obvious that those skilled in the art may vary the details of construction and arrangement of parts without departing from the spirit of my process, and therefore, I do not wish to be limited to such features, except as may be required by the claims.

What I claim is:—

1. The process of producing calcium cyanamid from calcium carbid, which consists in providing a mass of said carbid in a finely divided condition; in surrounding said finely divided carbid with a readily combustible envelop; in providing and maintaining a free passage for nitrogen through the body of said mass; and in subjecting said mass to the requisite temperature in the presence of free nitrogen to carbonize the envelop and to form said cyanamid, substantially as described.

2. The process of increasing the yield and improving the quality of calcium cyanamid during the manufacture of the same, which consists in providing a mass of finely divided calcium carbid; in providing and maintaining free passages for nitrogen through and around said mass during the reaction; and in heating said mass to the temperature required to produce calcium cyanamid in the presence of free nitrogen, substantially as described.

In testimony whereof, I affix my signature, in presence of two witnesses.

FRANK S. WASHBURN.

Witnesses:

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