(No Model.)

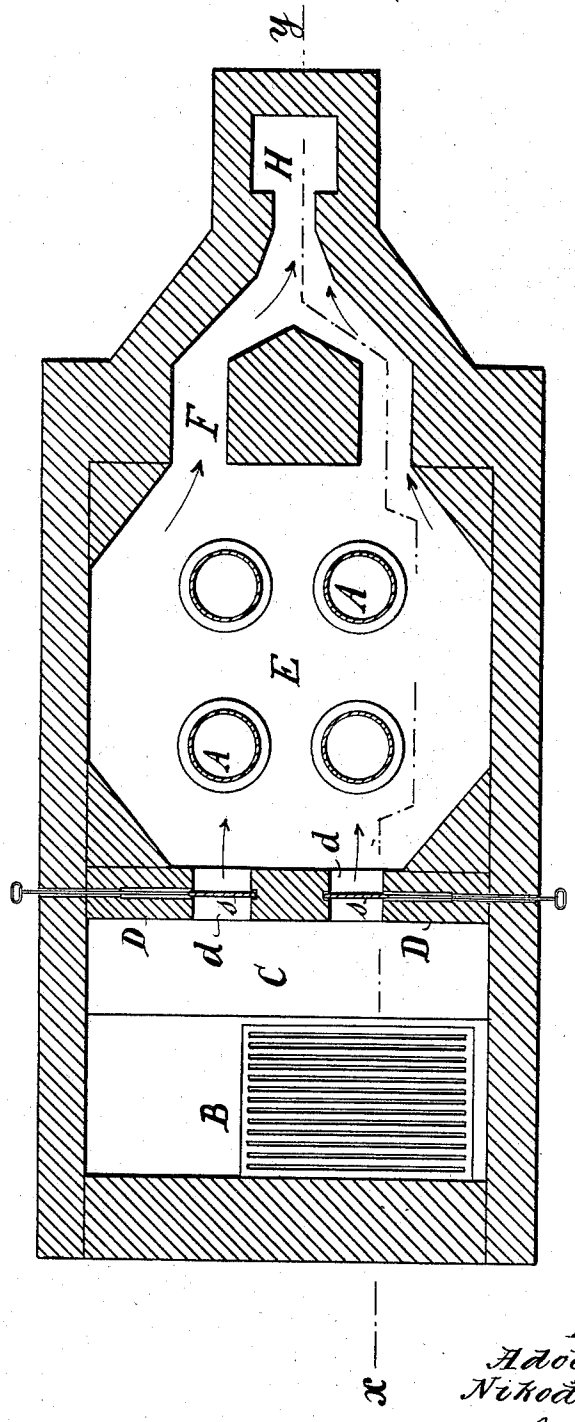
2 Sheets—Sheet 1.

A. FRANK & N. CARO.

PROCESS OF MAKING CYANIDS.

No. 590,217. Patented Sept. 21, 1897.

Fig. 1.



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2 Sheets—Sheet 2.

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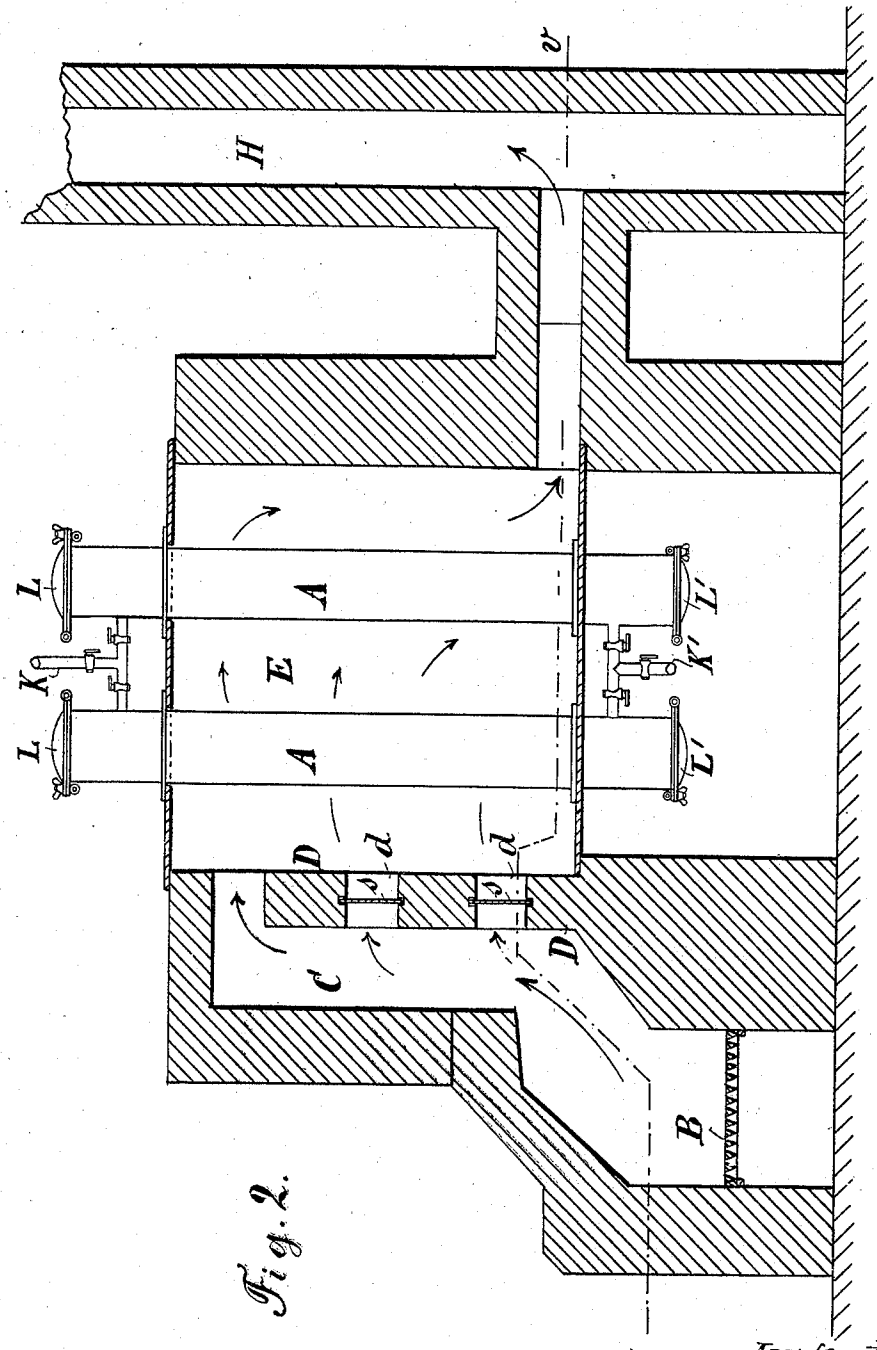

Fig. 2.

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UNITED STATES PATENT OFFICE.

ADOLPH FRANK, OF CHARLOTTENBURG, AND NIKODEM CARO, OF BERLIN, GERMANY.

PROCESS OF MAKING CYANIDS.

SPECIFICATION forming part of Letters Patent No. 590,217, dated September 21, 1897.

Application filed November 5, 1895. Serial No. 568,023. (No specimens.) Patented in Belgium August 10, 1895, No. 116,953, and in Luxemburg August 11, 1895, No. 2,346.

To all whom it may concern:

Be it known that we, ADOLPH FRANK, residing at Charlottenburg, and NIKODEM CARO, residing at Berlin, Germany, have invented certain new and useful Improvements in and Relating to the Production of Cyanogen Combinations, (for which Letters Patent have been obtained in Belgium, No. 116,953, bearing date August 10, 1895, and in Luxemburg, No. 2,346, bearing date August 11, 1895;) and we 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, reference being had to the accompanying drawings, and to letters of reference marked thereon, which form a part of this specification.

Our invention has relation to the production of cyanogen combinations; and it has for its object a novel process of producing the same.

According to the statement of Moissan, (*Comptes Rendus*, 1894, p. 503,) the passing of nitrogen over the heated carbids of calcium, barium, and strontium does not result in a reaction or combination. We have discovered, however, that nitrogen is taken up by carbids in presence of steam in a closed vessel, as in a tubular or other retort or the like, the carbids having previously been heated to a temperature approximating that of red heat. The formation of the cyanids takes place in the manner described, whether the carbids of the metals of the alkaline earths—such as calcium, barium, strontium, and magnesium—or the metals of the alkalies—such as potassium, sodium, or lithium—are employed, this being likewise the case when mixtures of these carbids are used or when the carbids contain any other substance, as an alkali (soda or potash) or an alkaline earth (lime, baryta, strontia, or magnesia) or the salts of the alkalies or alkaline earths. The mixture of carbids may be effected by means of the different carbids themselves, or by forming the carbids from raw materials, as the oxids or carbonates of the alkalies and alkaline earths, or the free metals by admixture with carbon in any well-known or desired manner, or by mixing the respective carbids of the metals of the alkaline earths or a mixture thereof with an alkali or alkalies or a salt or salts thereof.

In the production of carbids from the free metals and carbon practical experiments have shown it to be advisable to subject the same to the required high temperature in a closed retort or vessel, especially when metals are used that volatilize at the said temperature—as potassium, sodium, and magnesium, as well as the zinc chemically allied or similar to the latter—in order to produce the carbids by the action of the vapors of the metals upon the carbon, the reaction in the production of sodium carbid, for instance, taking place according to the following equation:

$$C_x + Na_y = C_xNa_y.$$

We have further discovered that when iron or iron combinations are mixed with the substances from which the carbids are produced and the mixture is treated as described the corresponding cyanogen combinations can be obtained.

We will now describe the mode of carrying out our invention by the following examples:

First. Comminuted barium carbid is placed in a refractory vessel, preferably of fire-clay and of tubular form. The retort is provided with a suitable inlet and an outlet for the induction and eduction of the nitrogen or gaseous matter. The nitrogen (which may be air freed from its oxygen) is saturated with steam, and this may be conveniently effected by simply passing the nitrogen through water, which latter is converted into steam within the heated retort. After the retort and its contents have been heated to about a red heat the moisture-laden nitrogen is caused to flow or is forced into the same. For a charge of from fifteen to seventeen kilos barium carbid from two to two and one-half cubic meters of nitrogen will be required, which is preferably

forced into the retort at a moderate pressure and at such a rate of speed that the operation will be concluded in about two hours. The material in the retort is then allowed to cool and treated with water. From the unconverted carbid acetylene is evolved, which may be collected separately, while barium cyanid goes over into solution and is recovered from the liquid in a well-known manner.

In the production of barium cyanid, as described, the reaction takes place in accordance with the following equation:

$$BaC_2+2N=Ba(CN)_2.$$

Second. Calcium carbid when treated as just described does not give very favorable results, while excellent results are obtained with a mixture of this carbid and barium carbid, also with a mixture of calcium and sodium carbid, the latter being obtained in the usual manner from soda-lime and carbon. The reaction product obtained by the action of nitrogen and steam upon the described mixtures can likewise be washed out with water, as described in the first example, the acetylene evolved being collected and the cyanogen combination separated from the solution. The latter can be readily effected by adding to the solution an acid, preferably carbonic acid, whereby cyanid of hydrogen is driven out, the latter gas being then brought into contact with the oxid of the metal the cyanogen salt of which it is desired to obtain. The desired cyanid may also be obtained from its solution by double decomposition—for instance, by addition of an alkali carbonate—whereby the cyanid of a metal of the alkaline earths is converted into the cyanid of the corresponding metal of the alkalies which remains in solution and into an alkaline-earth carbonate.

Third. The result obtained by the treatment described in the first example of barium as well as calcium carbid may be greatly improved by mixing the carbids or a mixture thereof with a caustic alkali or an alkali carbonate and then subjecting the same to the action of heat, nitrogen, and steam, as described in said first example, the product of the reaction being then treated as described in example 2.

Fourth. Carbids of the metals of the alkalies, as those of potassium, sodium, and lithium, yield *per se*, or when mixed, even in presence of an alkaline earth, as lime, baryta, strontia, magnesia, or their salts, the corresponding cyanids when treated as described in the first example, the cyanids being obtained from the reaction products by washing out with water and then separating the same from their solution in a well-known manner.

The reaction in the production of potassium cyanid, for instance, takes place according to the following equation:

$$K_2C_2+N_2=2KCN.$$

The temperature most favorable to the production of the described cyanogen combinations is a dark-red heat, the action of the nitrogen being very sluggish or slow at lower temperatures, while at materially-higher temperatures the yield is reduced by reason of a partial decomposition taking place.

For the best utilization of the nitrogen a number of retorts can be arranged in battery through which the gas is successively forced, and means provided whereby any one or more of such retorts may be cut out without interrupting the operation of the remaining retorts, thereby making the operation a continuous one.

Any suitable apparatus may be employed in carrying out my invention, and in the accompanying drawings I have shown an apparatus of simple construction for the purpose.

Figure 1 is a horizontal section on line *v w* of Fig. 2, and Fig. 2 a vertical section on line *x y* of Fig. 1.

In the figures of drawings, B indicates the fuel-grate; C, a flue leading from said grate to the heating-chamber E, said flue formed by extending the fire-bridge D nearly to the upper wall of the apparatus, openings *d* being formed in said fire-bridge controlled by suitable dampers *s*, whereby the heat and products of combustion can be led into the heating-chamber at different levels, as may be required in heating the retorts A A, which latter, as shown, are arranged vertically in said heating-chamber. The retorts are provided with charging and discharging covers or caps L L′ at their upper and lower ends, which ends project, respectively, above and below the top and bottom of the heating-chamber E, and are connected at those points with gas-supply and exhaust-pipes K and K′, respectively, the products of combustion escaping from chamber E to a stack H through a suitable flue F. Any desired number of retorts A may be arranged within the heating-chamber. I have shown four such retorts as an example.

It will be observed that the described process is of the simplest nature, capable of being carried out at a low temperature (dark-red heat) as compared with the temperatures required in the known processes, while the yield of cyanogen combinations greatly exceeds that of any other process known to us.

Having thus described our invention, what we claim as new, and desire to secure by Letters Patent, is—

1. In a process for producing cyanogen combinations, subjecting a described carbid

of a suitable metal as a metal of the alkalies, to the action of heat and nitrogen in the presence of steam.

2. In a process for producing cyanogen combinations, subjecting a described carbid of a suitable metal, as a metal of the alkalies, to the action of heat and of nitrogen, in the presence of steam and of a described combination of a metal belonging to the series of metals of the alkalies and of the alkaline earths.

3. In a process for producing cyanogen combinations, subjecting a described carbid of a suitable metal, as a metal of the alkalies, to the action of heat and nitrogen in the presence of steam and of a feriferous substance, substantially as described.

In testimony that we claim the foregoing as our invention we have signed our names in presence of two subscribing witnesses.

ADOLPH FRANK.
NIKODEM CARO.

Witnesses:
RICHARD SCHMIDT,
W. HAUPT.