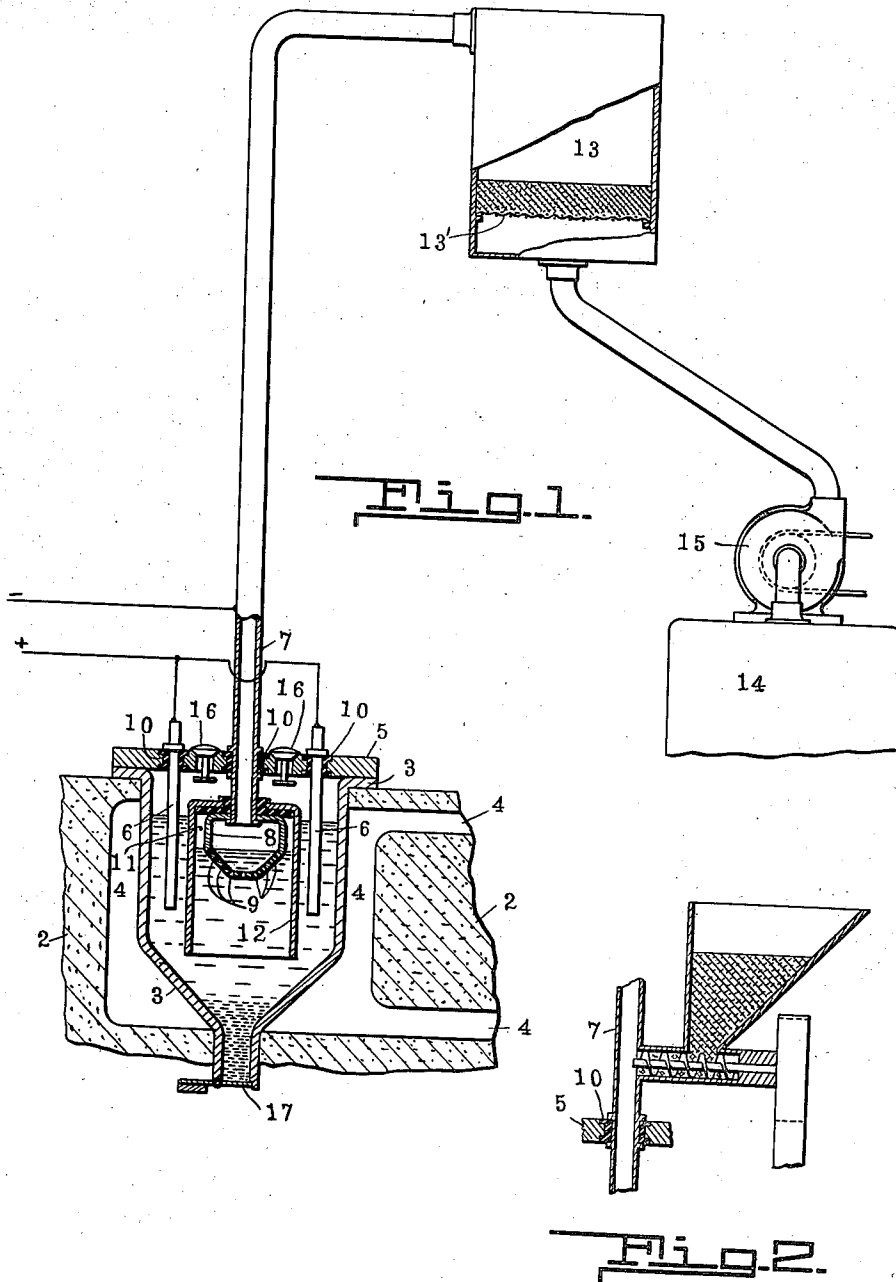


C. B. JACOBS.
PROCESS OF MAKING NITROGEN COMPOUNDS.
APPLICATION FILED DEC. 26, 1914.

1,311,231.

Patented July 29, 1919.



WITNESSES
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PROCESS OF MAKING NITROGEN COMPOUNDS.

1,311,231.

Specification of Letters Patent.

Patented July 29, 1919.

Application filed December 26, 1914. Serial No. 878,995.

To all whom it may concern:

Be it known that I, CHARLES B. JACOBS, a citizen of the United States, residing at Bloomfield, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in Processes of Making Nitrogen Compounds, of which the following is a full, clear, and exact description, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to the processes and apparatus for making nitrogen compounds and with respect to its more specific features to processes and apparatus for the production of cyanids and substances from which cyanids may be formed.

One object of the invention is the provision of a practical and relatively inexpensive process for the production of a cyanid.

Another object of the invention is the provision of a simple and efficient process for the production of compounds from which cyanids, etc., may be readily made.

The invention accordingly consists in the several steps and the relation and order of one or more of such steps with relation to each of the others thereof, which will be exemplified in the hereinafter disclosed process, and the scope of the application of which will be indicated in the claim that follows.

Figure 1 is a diagrammatic view of an apparatus suitable for carrying out the process; and Fig. 2 is a view of a modified detail of the apparatus.

The drawing is to be regarded as more or less of a diagrammatic representation of an apparatus suitable for carrying out the process, and it is believed that it will be conducive to understanding of the disclosure if the processes carried out be first described, leaving the description of the apparatus for the latter part of the disclosure.

The processes herein referred to relate to synthetic production of nitrogen compounds by the treatment of alkali metals, and also the production of alkali metals in form and condition suitable for such treatment.

While any of the alkali metals may be treated in accordance with the process so as to result in related cyanids, the embodiment of the invention is herein specifically disclosed in connection with the cyanid of

sodium, this latter being relatively inexpensive and more commonly used than the others. A suitable alkali compound, such as sodium hydroxid or "caustic soda" is treated so as to produce an electrolyte thereof. This is readily accomplished by fusing caustic soda. The caustic soda in a fused condition is placed in an electrolytic cell provided with the customary positive and negative electrodes, and a current of electricity from a suitable source is passed through the caustic soda electrolyte whereupon metallic sodium will be liberated at the negative pole. While the liberated sodium is in a nascent state, carbon and nitrogen are brought into contact therewith, said contact being effected at a temperature sufficiently high to cause chemical combination between the nascent sodium, the carbon and the nitrogen, and under these conditions the sodium will unite with the carbon and nitrogen, as will further appear, and sodium cyanid will be formed. A suitable temperature at which the sodium, carbon and nitrogen will combine to form the reactions referred to has been found to be between 600° C. and 900° C., although any other temperature lending itself efficiently to the result may be employed. It is found that carbon lends itself more readily to the carrying out of the process when it is in a finely divided condition, as for instance, would be provided by lamp black or finely ground charcoal. The nitrogenous gas may be either in the form of a pure dry nitrogen gas or in the form of dry ammonia. Whether nitrogen alone or nitrogen in the form of ammonia be employed, it is found that the nascent sodium unites with the carbon and the nitrogen and results ultimately in the production of sodium cyanid which may be readily separated from the electrolyte. When dry nitrogen and carbon are employed in the process, it is believed that reactions may take place in either one of two ways or the combination of the two. On the one hand the carbon and nitrogen combine to form the unsaturated cyanogen radical,

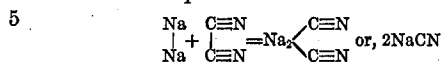


due to the influence of the nascent sodium acting as a catalytic agent. The cyanogen radical immediately combines with the sodium on account of its unsaturated condi-

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tion and nascent state of the sodium to form the fully saturated compound of sodium cyanid according to the following structural equation:

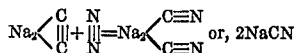


Each carbon atom present in the cyanogen has four chemical bonds or affinities, three of which are satisfied by the affinity of the trivalent nitrogen atoms leaving one remaining bond for each carbon atom to be satisfied by the affinity of the carbon atoms for each other. The chemical bonds of the two sodium atoms just satisfy these affinities and make a fully satisfied stable compound of sodium cyanid.

On the other hand the nascent sodium might be said to combine directly with the carbon to form sodium carbid,



Also an unsaturated compound with three of the four bonds of each carbon atom in condition to be satisfied by trivalent nitrogen atoms as follows:



However the intermediate steps of the reactions may take place either through the formation of cyanogen or through the formation of a carbid, the intermediate products are believed to be formed only "in situ" and immediately pass on to the same end product of the stable sodium cyanid.

When dry ammonia is used as the source of nitrogen, it is believed that is to be regarded as a hydrogen amid H.NH_2 , in which the hydrogen not contained in the amidogen group NH_2 is capable of replacement by the alkali metal, in the present case sodium, as in the well-known reaction of the formation of soda amid from metallic sodium and dry ammonia. In the presence of nascent sodium and carbon, the soda usually formed by the contact of dry ammonia with metallic sodium is replaced by sodium cyanamid



due to the polymerization of the amidogen group NH_2 to the di-amidogen group $\text{NH}_2.\text{NH}_2$ and its combination with two atoms of sodium and the replacement of four hydrogen atoms by one carbon atom. The sodium cyanamid takes up a further atom of carbon and passes on to the fully saturated sodium cyanid.

It is to be observed that the formation of the cyanid by means of the dry ammonia may be carried out either by bringing the carbon into contact with the ammonia and the nascent sodium so that the three make the quick transposition or chemical com-

bination into cyanid, as before described, or the dry ammonia alone may be used as a preliminary step, the same being brought into contact with the nascent sodium at a temperature sufficient to effect chemical combination. Under the latter circumstances soda amid is produced and this substance may be subsequently employed in connection with carbon to form the cyanid in a manner well known.

Reverting now to a form of apparatus for carrying out the process, the numeral 1 indicates an iron or steel container or crucible set in brickwork 2, and supported by its flanges 3, there being passageways 4 in the brickwork surrounding the container for the purpose of conducting heating gases therearound to heat the cell. The crucible is provided with a tight iron or steel lid 5 which supports the positive carbon electrodes 6, 6 and the negative electrode 7, the latter being composed, preferably, of an iron pipe terminating in an enlarged hollow head 8 having openings 9 leading into the electrolyte space within the crucible. The electrodes pass through insulating bushings 10 in the lid, and a chamber 11 is formed around the end of the negative electrode by means of an iron hood 12 supported by the negative electrode and connected thereto above the openings 9, said hood being insulated from the electrode. The hood extends downwardly into the cell and has an open bottom communicating with the electrolyte space of the container. The numeral 13 indicates a gas-tight compartment or chamber which communicates with the pipe 7 and contains finely divided carbon, such as lamp black or charcoal resting on a fine screen 13' and 14 indicates a supply of nitrogen gas or dry ammonia. The numeral 15 indicates a pump by means of which the gas from the container 14 may be fed under some pressure through the carbon box 13 and through the passageway in the pipe 7 to the openings 9. Caustic soda is placed in the crucible 1, the cover or lid is applied, and the caustic soda is brought to a state of fusion, and a temperature sufficient to carry on the reactions is maintained, as before explained. The electric circuit is made through the electrodes in the customary manner and the pump is started. The caustic soda will be decomposed and sodium will be liberated at the negative electrode in the presence of the nitrogen or ammonia which, as will be understood, will be permeated by the finely divided carbon suspended therein. In this manner the nitrogen and carbon are brought into close and intimate contact with the sodium, while the latter is in a nascent state, whereupon the reactions hereinbefore referred to will take place. The sodium cyanid formed will gravitate toward the bottom of the crucible and may be disposed

of in any convenient manner, as by tapping, while the process is being carried on, or the process may be discontinued for a moment, the lid lifted, and the cyanid removed. The
5 hopper shape of the bottom of the crucible facilitates the removal of the cyanid through the pivoted tap cover 17.

The hood 12 forms a continuation of the passageway or pipe 7, and facilitates the
10 depression of the surface of the electrolyte at the negative electrode, so that when in operation the electrolyte is substantially restricted to contact with the negative electrode at the level of the uppermost of the
15 openings 9. In this manner the sodium is liberated at the place where it will at once come in contact with the gases and carbon delivered through the tube 7.

Thus, by the above described process and
20 apparatus are accomplished, among others, the objects hereinbefore referred to.

As many changes could be made in carrying out the above process, and in the above construction, and many apparently widely
25 different embodiments of this invention could be made without departing from the scope thereof, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be in-

terpreted as illustrative and not in a limiting sense. 30

It is also to be understood that the language used in the following claim is intended to cover all of the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language, might be said to fall there-between. 35

Having described my invention, what I claim as new and desire to secure by Letters Patent is: 40

A process of producing an alkali metal cyanid which comprises electrolyzing an alkali metal compound so as to liberate alkali metal of the compound at one pole and
45 delivering a nitrogenous gas and carbon in the form of a plurality of relatively fine streams at the point of liberation of said metal so that the same contacts with the said metal while it is in a nascent state and at
50 a temperature sufficient to effect chemical combination.

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

CHARLES B. JACOBS.

Witnesses:

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