C. E. ACKER.

PROCESS OF PRODUCING NITRIDS.

APPLICATION FILED MAR. 18, 1909.

943,132.

Patented Dec. 14, 1909.

Fig. 1.

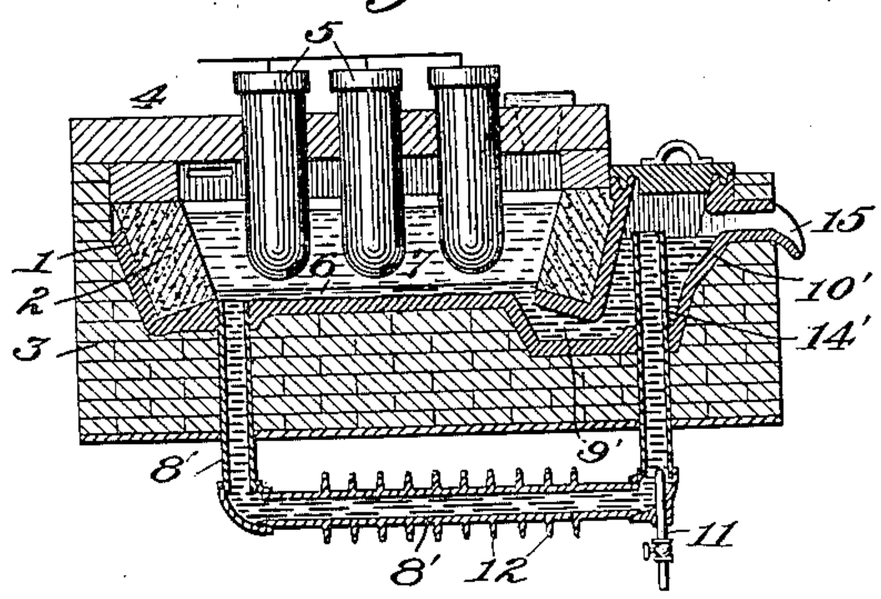


Fig. 2.

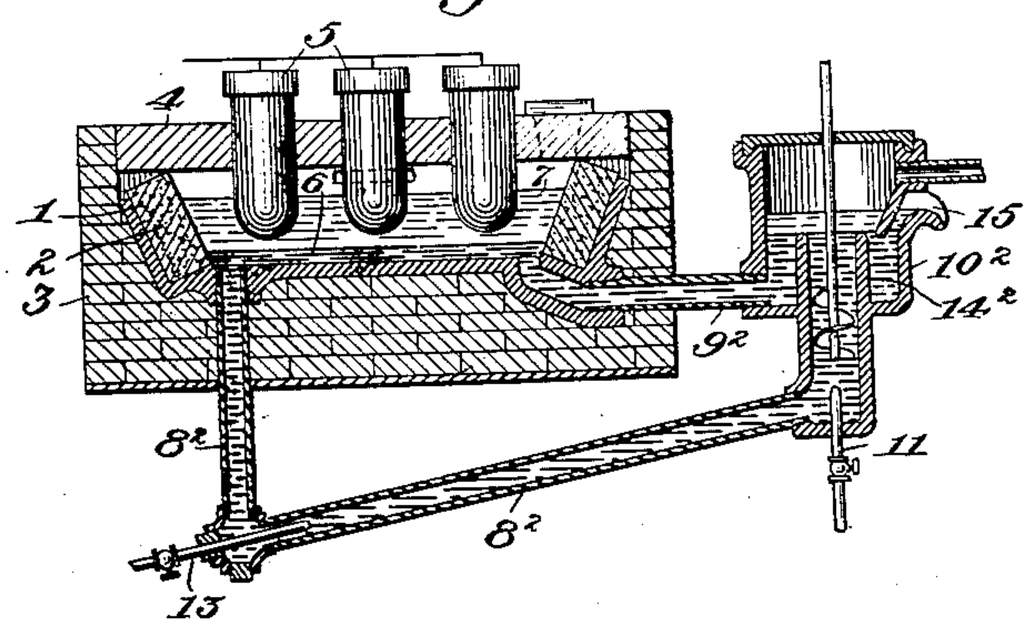
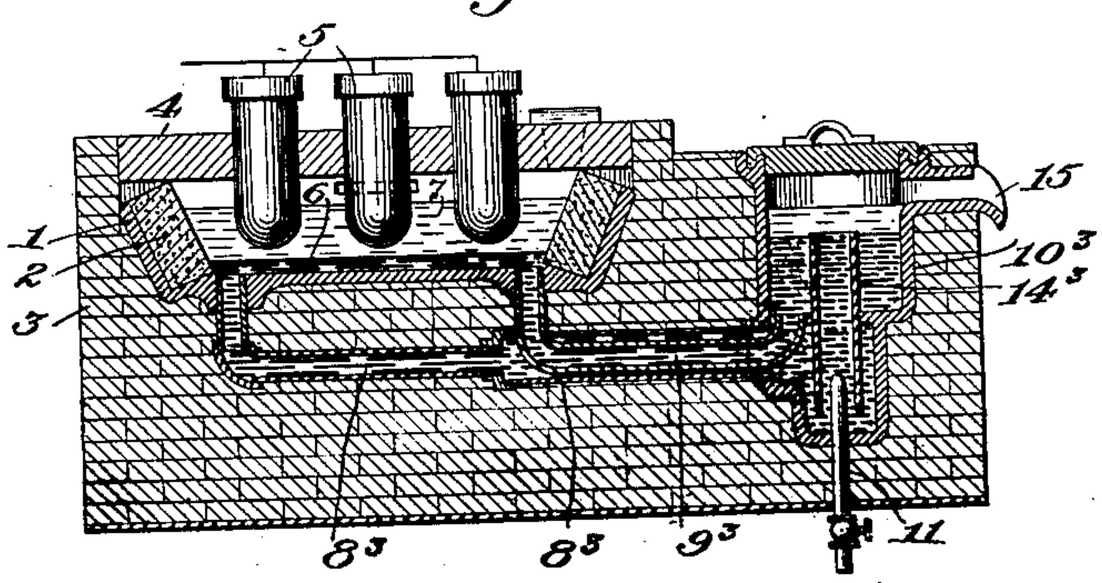


Fig. 3.



Witnesses:
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UNITED STATES PATENT OFFICE.

CHARLES E. ACKER, OF NEW YORK, N. Y., ASSIGNOR TO THE NITROGEN COMPANY, A CORPORATION OF NEW YORK.

PROCESS OF PRODUCING NITRIDS.

943,132. Specification of Letters Patent. **Patented Dec. 14, 1909.**

Application filed March 18, 1909. Serial No. 484,187.

To all whom it may concern:

Be it known that I, CHARLES E. ACKER, a citizen of the United States, residing at New York, in the county of New York and State of New York, have invented certain new and useful Improvements in Processes of Producing Nitrids, of which the following is a specification.

This process relates to the electrolytic production of nitrids, especially those of the alkali and alkaline-earth metals.

The preferred process comprises the steps of electrolyzing a molten compound of the metal which is to be combined with nitrogen, alloying the separated metal with a cathode metal, reacting on the alloyed metal with a nitrogenous gas, and returning the residual metal to the cathode. Incidental steps are the injection of the nitrogenous gas in a direction to cause or assist the circulation of the alloy and residual metal; and the cooling of the alloy before treatment with the gas, especially by maintaining opposed adjacent streams of the alloy and residual metal.

Apparatus which may be used in carrying out the process is shown in the accompanying drawings, in which—

Figures 1, 2, 3 are vertical longitudinal sections of three different constructions, each comprising an electrolytic cell and a separate connected chamber for treating the alloy.

Each apparatus illustrated has the general construction of that heretofore devised by me for the production of caustic alkali, the electrolytic cell comprising a cast iron vessel 1 lined at the sides with magnesia brick 2, inclosed in brickwork 3 and covered by a refractory slab 4, through which pass the depending carbon anodes 5. In the bottom of the vessel is a shallow layer 6 of a molten heavy inert metal such as lead, constituting the cathode, upon which is the molten electrolyte 7. The vessel 1 is connected by delivery and return pipes or ducts 8, 9 to a separate chamber 10, into which the nitrogenous gas may be introduced by a valved pipe 11. The chamber 10 may be inclosed in the brickwork 3, as shown in Figs. 1, 3, or may be outside it as shown in Fig. 2, depending on the temperature which it is desired to maintain therein.

The delivery pipe $8'$ of the apparatus shown in Fig. 1 is provided with a series of annular heat-radiating flanges 12, to cool the alloy therein. The delivery pipe 8^2 of the apparatus shown in Fig. 2 has a lower portion which receives a supplemental gas inlet pipe 13 and thence inclines upward to the chamber 10, the gas injected through the pipe 13 and the light compounds produced by the gas rising and moving along the pipe, facilitating the reaction therein. In the apparatus of Fig. 3, a portion of the delivery pipe 8^3 is enlarged and receives the return pipe 9^3, so that heat may be transferred from the outflowing alloy to the returning residual metal. The circulation of the alloy and metal may be effected either solely by the current of injected gas, as in the apparatus of Figs. 1, 3, or by a mechanical device, for example the screw shown in Fig. 2, or by both. If desired, the alloy may be cooled by injecting a regulated amount of a cooled and compressed gaseous reagent.

The reaction chamber 10 is of the general construction of that employed in my caustic alkali apparatus, comprising a vertical central pipe 14, which receives the molten alloy and injected gas. The nitrid resulting from the reaction, being usually lighter than the residual metal, rises and collects as a floating layer, which may be run out through a suitable outlet 15, the metal returning to the electrolytic cell to reconstitute the cathode.

A specific process which may be carried out is the production of calcium nitrid by the injection of nitrogen into an electrolytic calcium-lead alloy.

I claim:

1. The cyclic process of producing nitrids, which consists in repeatedly reacting with nitrogen on an alloy comprising an inert metal and a nitrid-forming metal, separating the resulting nitrid, and alloying more of the nitrid-forming metal with the residual inert metal.

2. The cyclic process of producing nitrids, which consists in repeatedly reacting with nitrogen on a molten alloy comprising an inert metal and a nitrid-forming metal, separating the resulting nitrid, and alloying more of the nitrid-forming metal with the residual inert metal.

3. The cyclic process of producing nitrids, which consists in repeatedly reacting with nitrogen on a molten alloy comprising an inert metal and a nitrid-forming metal, sepa-

rating the resulting nitrid, and alloying more of the nitrid-forming metal with the residual molten inert metal.

4. The cyclic process of producing nitrids, which consists in injecting a nitrogenous gas into a molten alloy comprising an inert metal and a nitrid-forming metal, separating the resulting nitrid, and alloying more of the nitrid-forming metal with the residual metal for further treatment.

5. The electrolytic process of producing nitrids, which consists in electrolyzing a molten compound of a nitrid-forming metal, alloying the separated metal with an inert cathode metal, reacting on the molten alloy with nitrogen, separating the resulting nitrid, and reëmploying the residual inert metal in the cathode.

6. The electrolytic process of producing nitrids, which consists in electrolyzing a molten compound of a nitrid-forming metal, alloying the separated metal with an inert cathode metal, injecting a nitrogenous gas into the molten alloy, separating the resulting nitrid, and reëmploying the inert residual metal in the cathode.

7. The cyclic process of producing alkaline-earth metal nitrids, which consists in repeatedly reacting with nitrogen on an alloy comprising an inert metal and an alkaline-earth metal, separating the resultant nitrid, and alloying more of the alkaline-earth metal with the residual inert metal.

8. The cyclic process of producing calcium nitrid, which consists in repeatedly reacting with nitrogen on an alloy comprising an inert metal and calcium, separating the resultant nitrid, and alloying more calcium with the residual inert metal.

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

CHARLES E. ACKER.

Witnesses:
ROBERT HARRISON,
SUSIE E. SAMPSON.