E. A. ASHCROFT.

PROCESS FOR THE MANUFACTURE OF ALKALI METAL COMPOUNDS.

APPLICATION FILED MAR. 12, 1912.

1,197,329.

Patented Sept. 5, 1916.

2 SHEETS—SHEET 1.

Fig. 1.

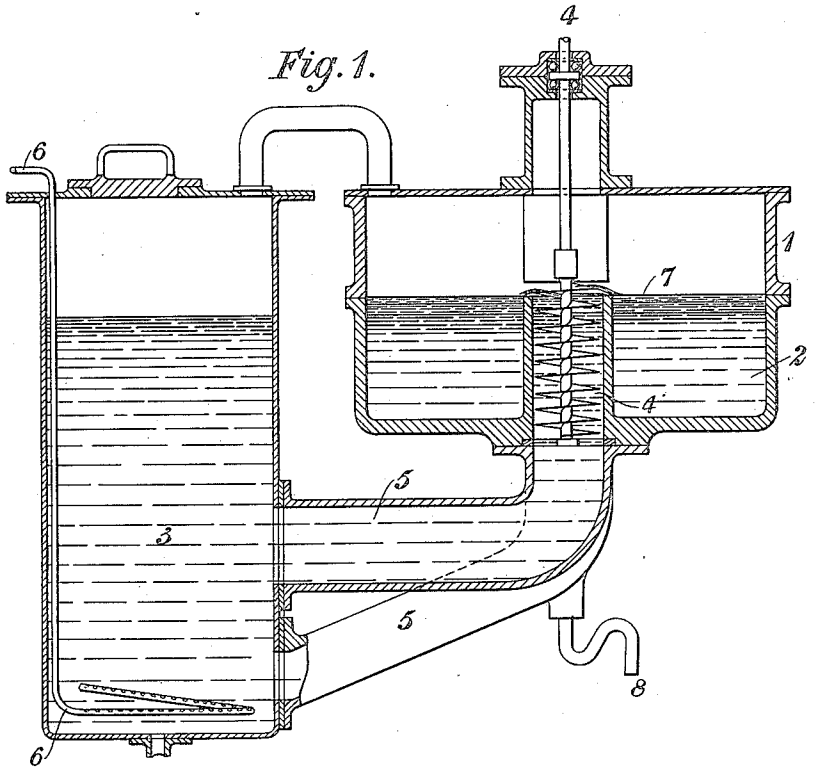
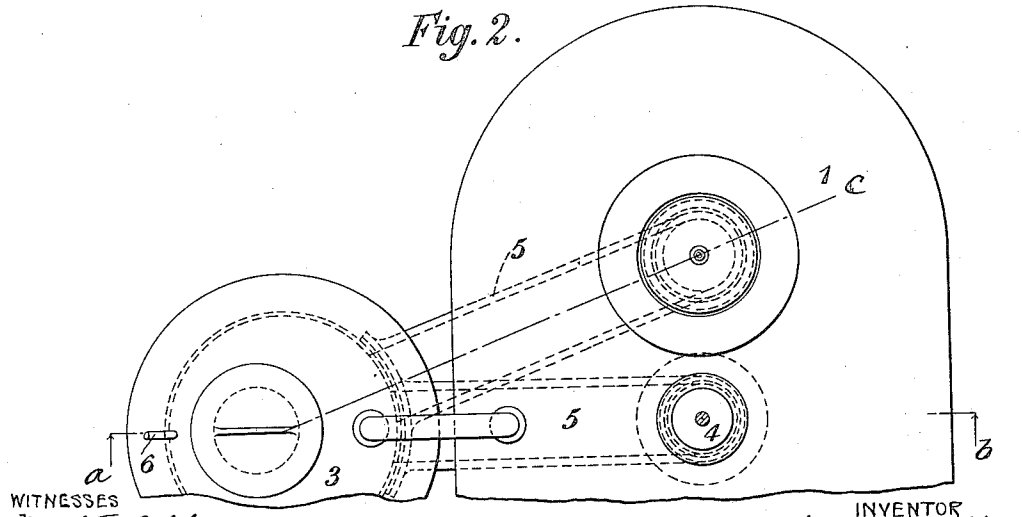


Fig. 2.



WITNESSES

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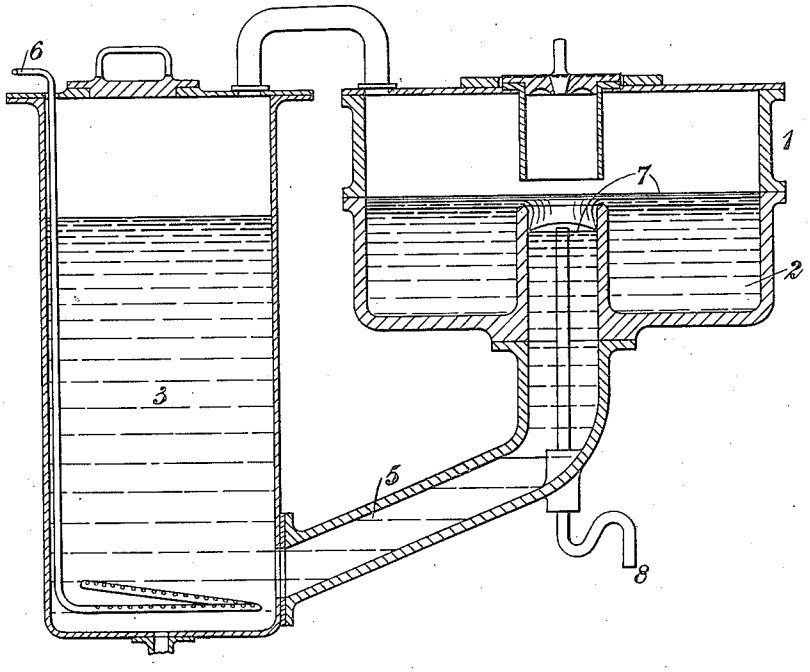
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2 SHEETS—SHEET 2.

Fig. 3.



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PROCESS FOR THE MANUFACTURE OF ALKALI-METAL COMPOUNDS.

1,197,329. Specification of Letters Patent. Patented Sept. 5, 1916.

Application filed March 12, 1912. Serial No. 683,376.

To all whom it may concern:

Be it known that I, EDGAR ARTHUR ASHCROFT, a subject of the King of Great Britain and Ireland, and residing at 99 Buckingham Palace road, in the county of London, England, and at Sande Gaard, Balestrand, Sogn, Norway, electrochemical engineer, have invented new and useful Improvements in Processes for the Manufacture of Alkali-Metal Compounds, of which the following is a specification.

It is known that reagents such as ammonia, carbon, hydroxyl (from steam) and the like will react with sodium-lead and potassium-lead alloys producing the corresponding compounds usually in the fused state but hitherto such processes have not been extensively applied, nor have the necessary conditions for success been ascertained, or described, the reactions being mostly too slow, or attended by other serious difficulties.

The object of my present invention is to effect the economical manufacture of alkali metals, amids, cyanamids, cyanids and other products (hydrates for instance) from alloys of alkali metals in one continuous operation.

In carrying my invention into effect advantage is taken of the discovery that the aforesaid products in a fused state if brought into contact with alloys of the alkali metals (for instance with sodium-lead or potassium-lead alloys) under suitable conditions as hereinafter explained will dissolve large quantities of the alkali metal without dissolving the lead and the dissolved alkali metal may be afterward very conveniently reacted to produce further quantities of the original compound or any derivative thereof. It is not essential to this invention whether the phenomenon observed be a true solution of the metal in the fused melt or a chemical union therewith forming another compound, as the original or desired compound is always generated on reacting the melt with the required reagents exactly as if a pure solution of metal in the melt takes place. As an example I will explain the production of sodium or potassium amids from sodium or potassium alloys with lead.

I have found by experiment that the alloy must exceed 6 per cent. sodium or 5 per cent. potassium and must be maintained at a temperature of or about 400° centigrade below which point the reactions become very much slower and impracticable on a large scale. The temperature of the fused amid should not however be allowed to exceed 440° centigrade for sodium or about 400° centigrade for potassium, or otherwise decomposition will begin. These conditions having been established it is only necessary to promote intimate contact of the alloy with the fused melt, which may be done in any convenient manner for instance by mechanical agitation or stirring. The means however which I prefer to employ for this purpose are as follows: The body of the fused amid is contained in a kettle or reservoir which is attached by means of pipes to the second cell of an electrolytic apparatus in which the alloys are continuously produced. By means of a worm or other pump the amid is caused to circulate somewhat rapidly and to spread out and flow continuously in a shallow stream over a large flat surface of the molten alloy and finally to collect in a well or chamber into which it flows over a weir of a suitable height (for example one centimeter) above the constant level of the alloy. From the chamber or well it flows back into the kettle or reservoir. It is convenient to so direct the discharge from the pump into the cell that rotary motion of the melt and alloy is set up and maintained. The reaction may be further assisted by agitators.

In the kettle or reservoir and also in the well or any other convenient part of the apparatus in which there is no free access to the alloy the melt may be continuously treated by passing in slowly a stream of ammonia, preferably through a perforated ring-pipe having many small outlets. The ammonia reacts with the dissolved metal and forms additional quantities of fused amid which may be caused to overflow from the apparatus continuously by means of a fixed level overflow in the well or elsewhere. In this manner a perfectly continuous process is obtained together with a high rate of reaction and all other conditions necessary for successful operation, while the apparatus is simple and durable. The kettle or reservoir and the secondary cell may be of any convenient form but I prefer a circular form with the well in the center of the secondary cell and the kettle or reservoir on one side of it. It is also possible to employ a kettle or reservoir contained within

or practically within the second cell or immediately beneath it as described under my application for Letters Patent Serial No. 683,377.

The accompanying drawing represents in vertical section in Figure 1 and plan in Fig. 2 an apparatus adapted for the purposes of this invention. Fig. 3 is a vertical section on the line *a*, *b*, *c*, Fig. 2, but I do not limit myself to the precise details illustrated.

1 is the secondary cell in connection with any electrolytic or other alloy producing apparatus. The alloy 2 circulates through this cell.

3 is the amid kettle or reservoir, 4 the pump for circulating the amid and 5 the well and circulating pipes to the kettle 3.

The inlet pipe for ammonia or the like is marked 6.

The surface of the moving body of amid or other product is indicated at 7. The alloy for this process may be produced by any known or suitable means but I prefer to employ the electrolytic process and apparatus described under my application Ser. No. 683,375. The described apparatus then becomes the second cell and attachment of that electrolytic plant and the whole constitutes means for the continuous manufacture of the finished product from the raw materials. Instead of reacting the dissolved metal in the amid with ammonia it may be subjected to electrolysis in a separate or connected cell with insoluble anodes, as described under my application Ser. No. 683,377 when the amid will be regenerated in an active state for further solution of metal from the alloy and the alkali metal will be obtained at the cathode.

Similar reactions may be carried out with other bodies for instance fused cyanid dissolves the alkali metal from the alloy and forms a compound which may be at once reacted with cyanogen (HCN), trimethylamin, benzol, or other hydrocarbons, ammonia and the like and will produce more cyanid, cyanamid, or other compound according to known reactions. The strength of alloy required is the same as for the amid but the temperature must be higher namely about 600° centigrade. Caustic soda dissolves the metal freely from the alloy at an even lower percentage of alkali metal if the temperature be above 550° centigrade and according to this invention the fused melt is reacted either with hydroxyl (from water in the form of steam) direct or with ammonia first forming amid which is afterward reacted with hydroxyl forming more caustic alkali, all as described under my application Ser. No. 683,378. It is also obvious that according to my application Ser. No. 683,377 alkali metals may be manufactured by electrolyzing the amid and returning the ammonia. I prefer to maintain an atmosphere of hydrogen (or other gaseous reagent in use) in the whole apparatus in order to completely exclude air and at the same time assist the reactions or, if desired, I may maintain an atmosphere of ammonia in the kettle. This condition is automatically produced by the condition of the process in practical work. It is convenient to put in communication the free space in the second cell and that in the kettle, or reservoir when this does not contain an atmosphere of ammonia and also to seal the overflow by a trap 8 to prevent ingress of air.

What I claim is —

1. The process of manufacturing an alkali metal product from an alloy of the alkali metal, which comprises bringing a fused body of the alkali metal product into contact with an alloy of said alkali metal with a heavy metal, transferring the fused alkali metal product with its absorbed alkali metal, to a separate reaction chamber apart from that containing the heavy metal alloy, and therein subjecting the absorbed alkali metal to the action of a reagent serving to convert the same into the desired product, substantially as described.

2. The process of manufacturing an alkali metal product from an alloy of the alkali metal, which comprises circulating a fused melt of the alkali metal product in intimate contact with an alloy of said alkali metal with a heavy metal, transferring the fused alkali metal product with its absorbed alkali metal, to a separate reaction chamber apart from that containing the heavy metal alloy, and therein subjecting the absorbed alkali metal to the action of a reagent serving to convert the same into the desired product, substantially as described.

3. The process of manufacturing sodium amid which comprises bringing a body of sodium amid into contact with a sodium lead alloy containing at least 6 per cent. sodium, transferring the melt so obtained into a separate reaction vessel and therein reacting the absorbed metal with ammonia.

4. The process of manufacturing sodium amid which comprises bringing a body of sodium amid into contact with a sodium-lead alloy containing at least 6 per cent. sodium at a temperature of substantially 400° C., transferring the melt so obtained into a separate reaction vessel and therein reacting the absorbed metal with ammonia.

5. The process of manufacturing alkali metal substances which comprises the step of passing a fused melt of alkali metal substance in a shallow stream over a large surface of an alloy of the alkali metal with a heavy metal, transferring the melt into a separate reaction vessel apart from that containing the heavy metal alloy, and therein subjecting the melt to the action of

reagents to transform the absorbed alkali metal into the desired product.

6. The process of manufacturing alkali metal substances which comprises the step of passing a shallow stream of a fused melt of alkali metal substance over a large surface of an alloy of the alkali metal substance, transferring the melt so obtained into a separate reaction vessel and therein agitating the melt in the presence of reagents to produce the desired product, and continuously circulating the alkali metal substance between the second cell of an electrolytic apparatus and the second reaction vessel, substantially as described.

7. The process of manufacturing an alkali metal product from an alkali metal alloy, which comprises bringing a fused melt of the alkali metal product into contact with an alloy of the alkali metal with a heavy metal containing at least five per cent. of the alkali metal, transferring the said product with its absorbed alkali metal to a separate reaction vessel and therein subjecting the melt to the action of a suitable reagent to obtain the desired product.

8. The process of manufacturing an alkali metal product from an alkali metal alloy, which comprises bringing a fused melt of the alkali metal product into contact with an alloy of the alkali metal with a heavy metal containing at least five per cent. of the alkali metal, and at a temperature of about 450° C., transferring the said product with its absorbed alkali metal to a separate reaction vessel and therein subjecting the melt to the action of a suitable reagent to obtain the desired product.

9. The continuous process of manufacturing an alkali metal compound from an alloy of the alkali metal, which comprises circulating from a separate reaction vessel a quantity of fused alkali metal compound into contact with a current of fused alkali-metal-heavy metal alloy, leading back said compound with the alkali metal absorbed from the alloy to said separate reaction chamber and therein subjecting the melt to the action of a suitable reagent to convert the absorbed alkali metal into the desired compound.

10. The continuous process of manufacturing an alkali metal compound from an alloy of the alkali metal, which comprises circulating from a separate reaction vessel a quantity of fused alkali-metal compound in a shallow stream over the surface of a fused alkali-metal-heavy-metal alloy, leading back said compound with the alkali metal absorbed from the alloy to the said separate reaction chamber and therein subjecting the melt to the action of a suitable reagent to convert the absorbed alkali metal into the desired compound.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

EDGAR ARTHUR ASHCROFT.

Witnesses:
ULYSUS J. BYWATER,
PAUL ARRAS.