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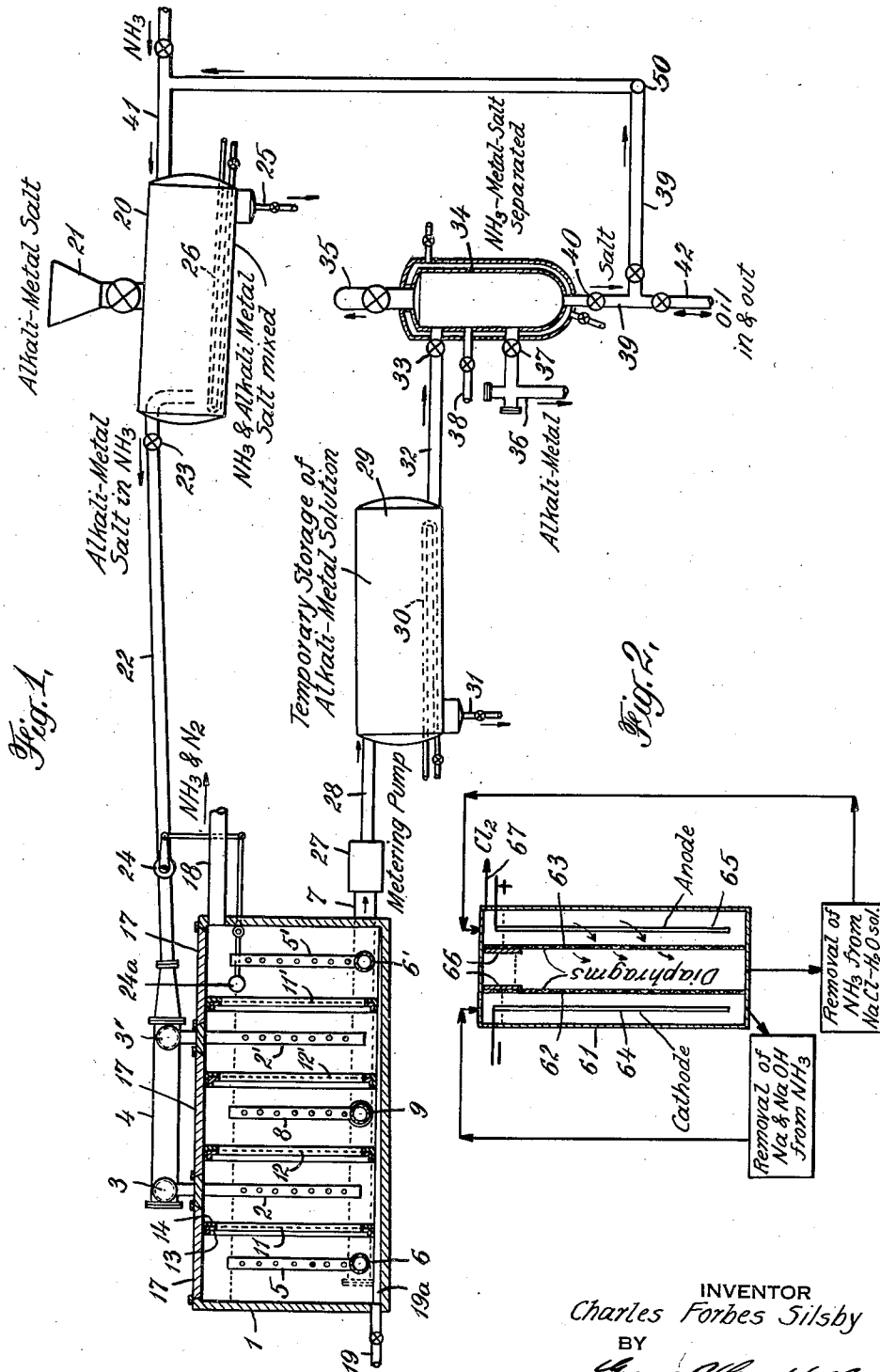
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ELECTROLYSIS OF SALTS IN LIQUID AMMONIA

Filed Feb. 15, 1938

2 Sheets-Sheet 1



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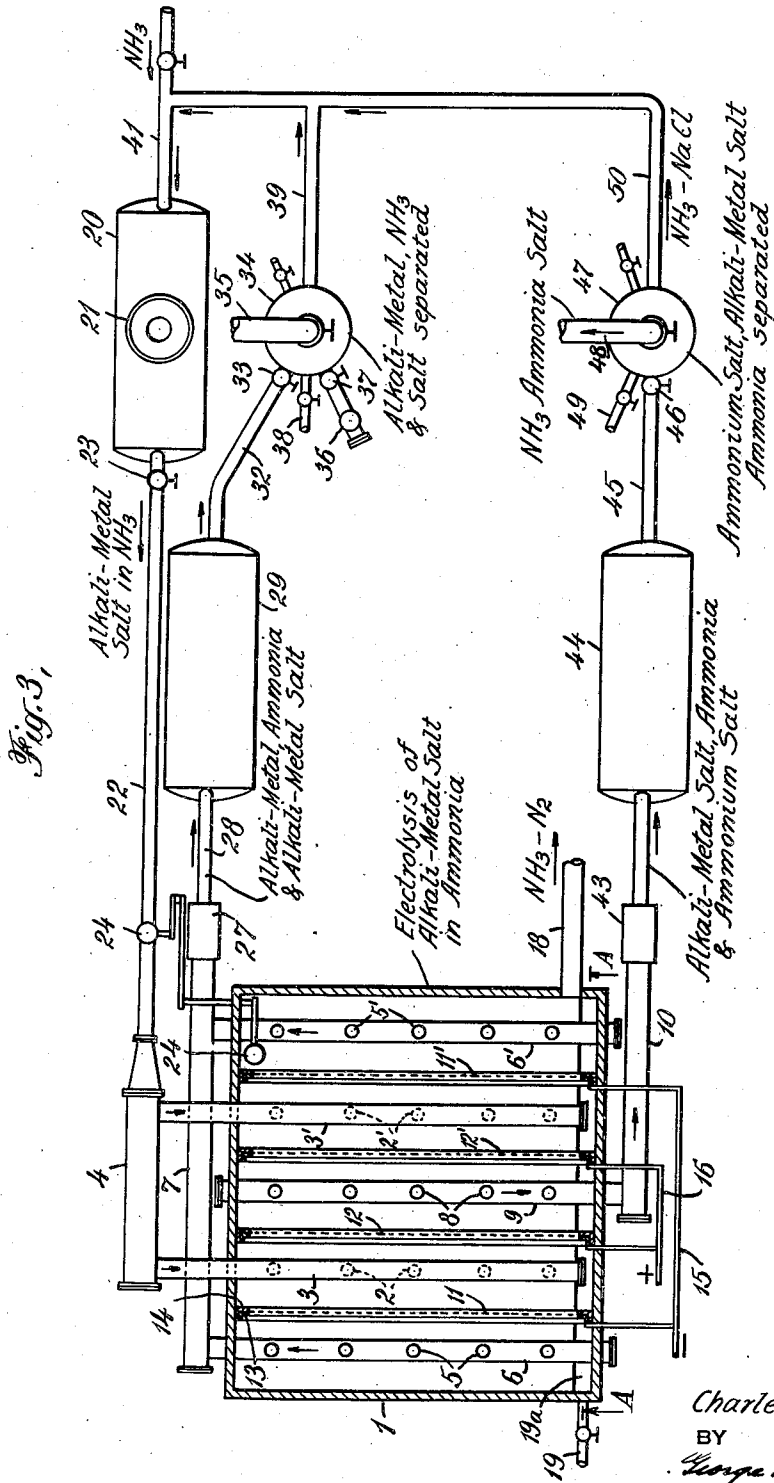
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ELECTROLYSIS OF SALTS IN LIQUID AMMONIA

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9 Claims. (Cl. 204—59)

This invention relates to electrolysis of alkali-metal salts in liquid ammonia. It is especially concerned with the electrolysis of the alkali-metal halides in anhydrous ammonia to liberate the alkali-metal, either as such or as the alkali-metal amide.

In the past it has been proposed to electrolyze the alkali-metal salts, such as sodium chloride, in anhydrous ammonia in order to produce a solution of metallic sodium in ammonia or, alternatively, to produce sodamide. To the best of my knowledge none of these prior proposals have become of commercial interest.

Further investigation of this subject has led to the development of an improved method of operation which overcomes difficulties inherent in operation of the prior procedures, which difficulties it is believed account for the failure of such prior procedures to attain commercial realization.

Foremost among the factors, the effect of which was not understood in prior processes, is the character of a solution of sodium in ammonia. Thus, in the electrolysis of sodium chloride, for example, although the salt may be decomposed into sodium and chlorine, the former being liberated at the cathode and the latter at the anode, the chlorine reacts readily with the ammonia and the sodium dissolves equally readily. The sodium solution then tends to diffuse through the liquid ammonia until it reaches proximity to the anode or the chlorine formed at the anode and which may be in the form of ammonium chloride. Since solutions of sodium in ammonia possess high electrical conductivity approaching that of a metallic conductor, dissolved sodium diffusing through the cell tends to short-circuit the electrodes. The sodium can also react with the anode product ammonium chloride. These phenomena vitiate the desired cell reactions.

In accordance with the present invention electrolyte is caused to flow through the cell at a rate greater than the rate of diffusion of metallic sodium through the electrolyte, whereby diffusion into the anode compartment of sodium liberated at the cathode, with consequent loss in yield and in current efficiencies, is prevented.

For instance, in one embodiment of the present invention there is introduced between the anode and cathode, at a sufficient rate to prevent substantial intermingling of liberated alkali-metal with liberated anion, a solution of alkali-metal salt in liquid ammonia, substantially free from an excess of either alkali-metal or salt anion; the solution of alkali-metal salt in ammonia is caused to flow gradually toward the cathode and anode at a sufficient rate to compensate for diffusion of the alkali-metal so that

a liquid partition between the anolyte and catholyte is provided. The resulting anolyte and catholyte are gradually drawn off separately for recovery of the products of electrolysis.

After removal of alkali-metal solution from the cell the alkali-metal may be separated from the ammonia and residual salt in any convenient manner and the latter materials may be recirculated in the process.

My process is illustrated in the following description and in the accompanying drawings, wherein Fig. 1 shows diagrammatically an elevation of one form of apparatus with the electrolytic cell shown in vertical section along the line A—A of Fig. 3; Fig. 2 shows diagrammatically (in vertical section) a second type of apparatus embodying my invention; Fig. 3 is a plan view of the apparatus of Fig. 1.

With especial reference to Figs. 1 and 3, the numeral 1 designates the electrolytic cell unit which may be a container of any suitable material, such as ceramic ware, silica brick, wrought iron, mild steel, chromium-iron, nickel, or the ferrous or non-ferrous alloys thereof. Any material of adequate structural strength may be used if it is suitably coated so as not to react with the materials in the electrolytic cell. If the cell is to be employed for production of alkali-metals in metallic form, the use of materials which act as catalysts for the formation of the amides should be avoided.

Within the cell are a series of inlet pipes 2 and 2' having apertures along their surface distributed so as to compel as uniform a flow of liquid across the cell as possible. These inlet pipes are fed by branch headers 3, 3' which in turn may be fed by the main header 4. Corresponding outlets 5 and 5' are provided communicating with withdrawal headers 6 and 6', leading to the main withdrawal pipe 7. Other outlet pipes 8 lead to header 9 connected to withdrawal pipe 10.

Between inlet pipes 2 and outlet pipes 5, and between inlet pipes 2' and outlet pipes 5', are arranged electrodes 11 and 11' constituting the cathodes. Between inlets 2 and 2' and outlets 8 are electrodes 12 and 12' constituting the anodes. All of the electrodes may have the same general construction, their two essential features being that they be capable of conducting an electric current and that they be suitably perforated to permit flow of liquid from inlets 2 and 2' to outlets 5, 5', and 8. The electrodes may be wire mesh or punched or expanded metal and the perforations may be uniformly distributed over the surface or may be concentrated locally in order to increase the uniformity of flow of the solution therethrough. Each electrode may be provided with a frame 13 fitting into insulating supports 14. Cathodes 11 and 11' may be connected to a common bus 15, and an-

odes 12 and 12' to a common bus 16, connected to a suitable source of direct current.

Suitable closures 17 prevent introduction of air into or escape of gases from the cell.

A gas conduit 18 is provided for drawing off the gases from the cell during the electrolysis.

For drainage purposes a valve-controlled outlet pipe 19 is provided, the cell bottom being sloped slightly toward the side at which the bus-bars are located and having a channel 19a along this side leading to the outlet. The insulating supports 14 may be cut out or recessed to facilitate flow to this outlet if desired.

In addition to the screen electrodes additional screens may be provided between them if desired. Such screens serve the purpose of preventing lateral agitation while permitting vertical agitation, such as may be caused by boiling or gas evolution in the electrolyte, thereby assuring a maximum uniformity of reaction and efficiency of operation.

A supply tank 20, having a hopper 21 for introduction of salt, is connected to header 4 by a pipe 22 having a shut-off valve 23. Normally flow will be controlled through this line by a regulating valve 24 controlled by float 24a. The supply tank 20 may have a drainage outlet 25 and may be provided with a cooling coil 26.

From the electrolytic cell outlet pipe 7 leads to a metering pump 27, which in turn is connected by pipe 28 to a storage tank 29. This storage tank may be similar to tank 20 and may have a cooling coil 30 therein and drain pipe 31.

From the tank 29 a pipe 32 having a valve 33 leads to a flash distillation chamber 34. The flash distillation chamber may be steam- or oil-jacketed and has a vapor outlet conduit 35, an outlet pipe 36 for liquid sodium provided with a valve 37, an inlet 38 for fresh liquid ammonia, and a second liquid outlet pipe 39 having a valve 40. This outlet pipe leads from the bottom of the distillation chamber back to the main ammonia inlet 41 on tank 20. A valve-controlled pipe 42 may be provided for removing liquid ammonia from the system at this point and also for the introduction and withdrawal of oil.

From the cell 1, pipe 10 leads to a metering pump 43 and thence to a tank 44 similar to tank 29. Pipe 45, having a valve 46, leads to a second flash chamber 47 having a vapor outlet 48, a liquid ammonia inlet 49, and a liquid ammonia outlet pipe 50 connecting with pipe 39 for return of liquid to the main ammonia inlet 41.

The operation of the above apparatus may be conducted as described below for the production of sodium from sodium chloride. The sodium chloride should be free from undesirable constituents; for instance, iron salts which would catalyze, or would form reaction products which would catalyze, the reaction of sodium and ammonia to sodamide.

Finely divided sodium chloride which has previously been dried to eliminate contained moisture, and liquid anhydrous ammonia are introduced into tank 20. A relatively large quantity of solid salt may be maintained in this tank at all times and anhydrous ammonia, in passing through the solid salt, will take up the salt in the form of a saturated solution thereof. This solution, containing on the order of ten to fourteen grams of sodium chloride per hundred grams of liquid ammonia passes through pipe 22 and headers 4, 3, and 3' to the inlet pipes 2 and 2' in the electrolytic cell until the cell is filled to the operating level whereupon the flow is

checked by float-controlled valve 24. By supplying direct current to the bus-bars 15 and 16, the solution in the cell is electrolyzed so that a solution of sodium in liquid ammonia is formed at the cathodes and a solution of ammonium chloride is formed at the anodes.

While the electrolysis may be conducted at any temperature and pressure at which ammonia and the solutions involved are liquid, it is preferable to employ a temperature in the range of about -14°C. to about $+2^{\circ}\text{C.}$ and to employ a pressure approximately corresponding to the vapor pressure of the ammonia at the operating temperature. In this manner vaporization of the ammonia serves to maintain the low temperature of the solution and to effect agitation thereof. This avoids the necessity for providing additional cooling means, such as cooling coils within the cell, or a cooling jacket about the cell, and thus simplifies construction. The agitation assists in keeping the solution uniform across the various sections of the apparatus.

While the incoming solution preferably should be saturated with salt, the electrolysis removes salt from the solution and therefore evaporation in the cell serves the further purpose of maintaining a relatively high concentration of salt as the electrolysis proceeds. It will be appreciated, of course, that with an efficient system the evaporation of ammonia will be less than with an inefficient system since the only sources of heat are the surrounding atmosphere and the heat generated by the electrolysis.

Nitrogen generated by the reaction of chlorine with ammonia and any hydrogen generated by reaction of sodium with ammonia, together with the ammonia vapor produced, are withdrawn through gas conduit 18 to refrigeration and compression apparatus for condensation of the ammonia, which may then be returned to tank 20 without further treatment. Where the formation of sodamide in the cell is minimized, there will be little formation of hydrogen; consequently, the uncondensed gases from the electrolytic cell will consist of practically pure nitrogen, which may be used as desired or wasted to the atmosphere. Where it is desired to produce sodamide directly in the electrolytic cell, the vapor space may be divided so that evolved hydrogen can be collected separately.

During the electrolysis metering pumps 27 and 43 are operated to withdraw liquid from the cell through outlet pipes 7 and 10 respectively. Pipe 7 and metering pump 27 withdraw the catholyte and pipe 10 and pump 43 withdraw the anolyte. The pumps are coordinated so that there will be substantially no flow of liberated sodium to the anode nor of ammonium chloride to the cathode.

The rate of flow will depend upon the rate of electrolysis within the cell. This rate should be such that the maximum practical electrolysis of sodium chloride is effected.

As long as there is a fairly steady, gradual flow of liquid from inlets 2 and 2' through the anode and cathode screens, the anode screens will serve as containers for anolyte and the cathode screens as containers for catholyte so there will be practically no diffusion or migration of anolyte toward the cathode or catholyte toward the anode and the objects of my invention will be attained.

Metering pump 27 delivers the catholyte to tank 29, which serves as a temporary storage

vessel for the solution at temperatures substantially below atmospheric. Refrigeration may be supplied by means of a refrigerating coil 30. Since the next step is an evaporation step, it is not necessary or desirable to effect any great cooling at this point but merely to compensate for gain of heat so as to avoid undesirably high back pressures on the metering pump. The liquid in this storage tank will contain metallic sodium in solution and undecomposed salt. If any sodamide has been formed, this too will be in solution or solution and suspension in the liquid ammonia. The solution is drawn off intermittently to flash distillation chamber 34 maintained under a sufficiently lower pressure to effect vaporization of the ammonia, which passes off through vapor outlet 35 to suitable condensation and recompression apparatus (not shown), from which it may be returned to tank 20. The evaporation of ammonia leaves as residue a mixture of metallic sodium, salt, and frequently a small amount of sodamide.

The flash distillation preferably is conducted until an amount of salt has been accumulated sufficient so that upon melting of the sodium the salt will substantially fill the bottom of the flash chamber up to the sodium outlet 36. When this point is reached the introduction of ammonia is discontinued and steam or other heating fluid is introduced into the jacket surrounding the flash chamber so as to heat it to a temperature slightly above the melting point of the sodium; the salt, being heavier than the liquid sodium, settles to the bottom of the mixture. The liquid sodium may then be drawn off by opening valve 37.

In order to assist in eliminating the liquid sodium, hot hydrocarbon oil of low vapor pressure at the temperature employed may be introduced through pipe 42 and permitted to rise through the body of settled salt so as to assist in driving the liquid sodium up and out at outlet 36. The hydrocarbon, of course, should be of a density intermediate that of the sodium chloride and that of the sodium. Since the sodium may be stored in the same hydrocarbon oil, the oil may be allowed to flow out after the sodium through outlet 36 thereby flushing this outlet.

When all or practically all of the sodium has been expelled, the hydrocarbon oil remaining in the chamber may be withdrawn through the same pipe 42. The heating fluid passing through the jacket is then turned off and liquid ammonia is introduced into the flash still at 38 to cool the walls by its evaporation. When the chamber has cooled sufficiently so that liquid ammonia remains in liquid phase, this liquid ammonia, which dissolves the sodium chloride remaining in the chamber, may be withdrawn through pipe 39 and returned to supply tank 20, or, in order to avoid contamination of the ammonia supply with hydrocarbon oil, may be withdrawn at 42. In the latter case it is desirable to employ a minimum of the liquid ammonia and to flush out as much as possible of the sodium chloride as solid in order to avoid using the large amounts of ammonia necessary to effect its complete solution. It is possible, of course, with the arrangement shown, to permit the hydrocarbon oil remaining with the salt in the flash distillation chamber to pass along through the system to supply tank 20 and to draw off accumulated oil from this tank through drain pipe 25.

Since the pressure resulting from the vaporization of liquid ammonia in the flash chamber may be utilized for returning liquid from the flash tank to the supply tank, no pumps are required.

When the salt has been eliminated from the chamber, vacuum may again be applied and further flash distillation of sodium solution effected.

Anolyte withdrawn through pipe 10 by metering pump 43 passes to storage tank 44 similar to tank 29. This solution will contain ammonium chloride and sodium chloride which should be separated before recirculation. For this purpose a flash chamber 47 is provided which, like chamber 34, may be maintained under vacuum to cause flash evaporation of ammonia therein, leaving ammonium chloride and sodium chloride as residue. The ammonia, after recondensation, may be returned to tank 20 for reuse. Steam or hot oil is then introduced into the jacket of the flash vaporizer to raise the temperature thereof sufficiently to decompose the ammonium chloride, which may be passed to a suitable sublimator or absorber for its recovery. Residual salt may be washed out by means of liquid ammonia introduced through pipe 49 and may be passed via pipes 50 and 39 to supply tank 20.

In both distillation chambers the distillation of ammonia may, of course, be assisted by application of heat, as by passing a heating fluid through the jacket. This fluid may be the ammonia vapor condensing at higher pressure or may be any other suitable vapor or liquid. If the ammonia vaporized in the flash chamber is recompressed and returned to the jacket either as liquid or as vapor, the fluid employed in the jacket during liquefaction of sodium should be selected so as not to introduce undesired constituents into the ammonia. It is possible, of course, to provide separate passages in the jacket for the two heating fluids but this tends to increase the mass of the still and to make its structure more complicated. In any event, it is desirable to avoid excessive heating in order to maintain the thermal efficiency of the distillation operation at a maximum and to avoid greater removal of heat from ammonia vapor than necessary to effect its recondensation for recirculation.

Wherever in the system heating or cooling is called for, it is desirable, in order to maintain the high thermal efficiency necessary for commercial operation, to provide for heat interchange with material to be cooled or heated, either by direct or indirect heat exchange or by interposition of an intermediate heat transfer fluid. The ammonia itself serves admirably as a heating or cooling medium where the temperatures required do not demand excessive pressure changes.

As previously mentioned, it is desirable to avoid any introduction of moisture into the electrolytic cell. This may be accomplished by thoroughly drying the salt and ammonia before introducing them into the system. However, in starting the electrolysis, the apparatus will contain some moisture and this will react with the sodium liberated in the hydrolysis to form sodium hydroxide. Since the sodium hydroxide is insoluble in liquid ammonia, it will settle out in the electrolytic vessel and, as long as it is not present in sufficient quantities to seriously coat the electrodes, no adverse effect upon the system will be caused. Settled caustic may be withdrawn through outlet 19 together with any other accumulated material deposited from the solution in the cell.

If it is desired to operate for the production of sodamide instead of metallic sodium, the same system described above may be employed; a catalyst for this reaction may be provided in the electrolytic cell or the storage tank 29. The catalyst may be provided by constructing the

cathode of high carbon steel or steel coated with iron oxide, for example, or the walls of the electrolytic cell or storage tank may be constructed of these materials. Preferably the catalyst is provided by dissolving in the electrolyte a salt of iron, cobalt, or nickel, for instance ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$. In this modification the salt carried by a suitable support is maintained in the electrolyte preferably in the ratio of 0.001 mol or less of the salt per gram atom of sodium liberated and a small amount of sodium oxide is introduced into the electrolyte either by the action of atmospheric oxygen on the sodium solution or by adding a small amount of sodium oxide or peroxide. Removal of solid sodamide from the solution may be effected by filtration or decantation as it seems to have little or no tendency to adhere to the catalyst. Since dissolved sodamide may be recirculated, the distillation chamber 34 may be omitted.

Sodium hydroxide may be made by injecting an amount of water just sufficient to react with the sodium in the ammonia solution. This is slightly more than the theoretical amount for forming anhydrous NaOH . By such treatment, precipitation of solid sodium hydroxide is effected. The solid product settles out from the ammonia and may be removed mechanically and washed with anhydrous ammonia to produce a solid sodium hydroxide of high purity.

In a similar manner the halides of the other alkali-metals may be converted into their dissociation products. Since in each case the alkali-metal or alkali-metal amide possesses a low melting point and density compared with that of the corresponding chloride, bromide, or iodide and the ammonium salts of all these halogens are relatively volatile (or in the case of the chloride, decomposable), the same recovery system may be applied and the only modifications necessary are those procedural changes required to take care of the different solubilities and melting points. For example, a different solution concentration requires a change in the flow rates and a different density requires the selection of a different oil to effect gravity separation. Such changes, however, will be obvious to those charged with the conduct of the process.

In the embodiment illustrated in Fig. 2, a diaphragm cell 61 having diaphragms 62 and 63, a cathode 64 and an anode 65 is employed. In this cell an aqueous anolyte is employed separated from the liquid ammonia catholyte by an aqueous solution of salt flowing through the central compartment. Baffles 66 are provided to permit a higher column of liquids in the anolyte and catholyte chambers than in the intermediate chamber. The height of the columns of liquid should be adjusted so that anolyte passes through diaphragm 63 as shown by the arrows. The column of liquid in the catholyte chamber should be sufficient to prevent substantial diffusion of aqueous solution through diaphragm 62. Some water may diffuse through diaphragm 62 without seriously affecting the operation of the cell. This water merely reacts with sodium generated to produce sodium hydroxide, which may be separated from the ammonia by filtration. Similarly ammonia may diffuse from the catholyte compartment through diaphragm 62 and may mix with the aqueous salt solution in the intermediate compartment. Solution is withdrawn from the intermediate compartment at a rate such as to maintain a substantially uniform level of

liquid to provide the proper flows of solution through the cell as just described.

After its removal from the central compartment, the solution may be treated for removal of ammonium chloride and ammonia or hydrogen chloride; for instance, by adding a small amount of caustic soda and warming or reducing the pressure on the solution to vaporize the ammonia. After this treatment the solution may have additional salt added to it or additional aqueous salt solution and may be again introduced into the anolyte chamber. The amounts and proportions of make-up materials will, of course, depend upon the rate of electrolysis, the amount of water lost by evaporation or reaction, etc. The catholyte solution may acquire a small amount of sodium hydroxide resulting from reaction of water as above indicated. This solution may be withdrawn from the catholyte chamber, filtered to remove any contained sodium hydroxide, and then treated to remove sodium in any convenient manner. If desired, the solution may be treated to form sodamide from the sodium. Ammonia and salt, with the addition of make-up solution, may be returned to the catholyte chamber. Ammonia removed from the aqueous solution may be added to this solution upon its return to the cell. Chlorine gas liberated at the anode may be withdrawn through a gas outlet 67 near the top of the cell.

Because of the electrical conductivity of the alkali-metals in liquid ammonia, especially in the case of the more concentrated solutions, the entire inner surface of the vessel in which the electrolysis is conducted may become a cathode. For this reason care must be exercised in arranging the electrical circuit in either of the above procedures so that a reduction of electrical efficiency from this source may be avoided. This can be accomplished by treating all of the apparatus as a cathode and insulating it accordingly. While similar results can be secured by providing an insulating coating on the interior of the apparatus (if it is constructed of metal), it should be borne in mind that passage of the solution through a metal valve farther along in the system may negatively charge the entire apparatus.

Instead of operating the diaphragm cell of Fig. 2 in the manner described above, the pressures in the several compartments may be adjusted to provide flow of ammonia through diaphragm 62 into the central compartment and withdrawal of ammonia containing small amounts of aqueous solution diffusing through diaphragm 63; or the mixture withdrawn from the central compartment may contain ammonia and water in more nearly equal proportions. With such modifications of the process the treatment of the liquid withdrawn from the central compartment should be modified to correspond to its composition.

Still another method of operation involves the introduction of a saturated solution of salt in anhydrous ammonia through the central compartment from top to bottom or from one end to the other, freeing the resultant solution from impurities acquired by diffusion during passage thereof through the central compartment and returning the purified solution to this compartment. The principal impurity to be removed will be water.

The saturated solution of sodium chloride in the anode compartment might be caused to recirculate through the anode compartment after treatment exteriorly for removal of any acid

formed. In the cathode compartment it may be desirable to operate with a flow of solution up rather than down through the cell in order to take advantage of the relatively low density of the solution of sodium in ammonia as compared with the original solution free from sodium.

I claim:

1. In the electrolysis of an alkali-metal salt wherein the catholyte comprises liquid ammonia, the improvement which comprises maintaining between the anode and cathode a liquid partition of an alkali-metal salt solution substantially free from alkali-metal uncombined with the salt anion and substantially free from the salt anion uncombined with alkali-metal by maintaining a gradual flow of said solution through the space between the anode and cathode at a sufficient rate to prevent substantial migration of alkali-metal to the anode.

2. In the electrolysis of a sodium halide wherein the catholyte comprises liquid ammonia and the anolyte comprises a member of the group consisting of liquid ammonia and water, the improvement which comprises maintaining between the anode and cathode a liquid partition of sodium halide solution substantially free from sodium and halogen by maintaining a gradual flow of said solution between the anode and cathode in a general direction from the former toward the latter at a sufficient rate to prevent substantial intermingling of liberated sodium with liberated halogen.

3. In the electrolysis of an alkali-metal salt wherein the catholyte comprises liquid ammonia and the anolyte comprises a member of the group consisting of liquid ammonia and water, the improvement which comprises introducing between the anode and cathode, at a sufficient rate to maintain between the anode and cathode a liquid partition of alkali-metal salt in liquid ammonia substantially free from alkali-metal uncombined with the salt anion thereby to prevent substantial intermingling of liberated alkali-metal with liberated anion, a solution of alkali-metal salt in liquid ammonia, substantially free from alkali-metal uncombined with the salt anion and substantially free from the salt anion uncombined with alkali-metal.

4. In the electrolysis of an alkali-metal halide wherein the catholyte comprises liquid anhydrous ammonia and the anolyte comprises a member of the group consisting of liquid ammonia and water, the improvement which comprises introducing between the anode and cathode, at a sufficient rate to maintain between the anode and cathode a liquid partition of alkali-metal salt in liquid ammonia substantially free from alkali-metal uncombined with the salt anion thereby to prevent substantial intermingling of liberated alkali-metal with liberated halide, a solution of alkali-metal halide in liquid anhydrous ammonia, substantially free from alkali-metal uncombined with halide and substantially free from halide uncombined with alkali-metal.

5. In the electrolysis of an alkali-metal halide wherein the catholyte comprises liquid ammonia and the anolyte comprises a member of the group consisting of liquid ammonia and water, the improvement which comprises gradually and continuously withdrawing anolyte and catholyte separately from the electrolytic cell at such a rate as to compensate for diffusion thereof toward the cathode and anode respectively, sepa-

5 rately evaporating ammonia from solids in the anolyte and catholyte, separating the ammonia vapor from the solids, reliquefying the separated ammonia, mixing it with additional alkali-metal halide, and returning it to the electrolytic cell between the anode and cathode at a rate sufficient to prevent substantial intermingling of liberated alkali-metal with liberated anion so as to maintain a liquid partition free from alkali-metal uncombined with salt anion between the anolyte and catholyte therein.

6. In the electrolysis of sodium chloride wherein the catholyte comprises liquid ammonia and the anolyte comprises a member of the group consisting of liquid ammonia and water, the improvement which comprises gradually and continuously withdrawing anolyte and catholyte separately from the electrolytic cell at such a rate as to compensate for diffusion thereof toward the anode and cathode respectively, separately evaporating ammonia from solids in the anolyte and catholyte, separating the ammonia vapor from the solid, reliquefying the separated ammonia, mixing it with additional sodium chloride and returning it to the electrolytic cell between the anode and cathode at a rate sufficient to prevent substantial intermingling of liberated sodium with liberated chlorine so as to maintain a liquid partition free from sodium uncombined with chlorine between the anolyte and catholyte therein.

7. In the electrolysis of an alkali-metal salt in liquid ammonia, the method which comprises providing a catholyte of liquid ammonia and an aqueous anolyte and maintaining between the anode and cathode a liquid partition of alkali-metal salt solution substantially free from alkali-metal uncombined with salt anion and substantially free from the salt anion uncombined with alkali-metal, maintaining a gradual flow of said solution between the anode and cathode in a general direction from the former toward the latter at a sufficient rate to prevent substantial intermingling of liberated alkali-metal with liberated anion.

8. In the electrolysis of an alkali-metal halide in liquid ammonia, the method which comprises providing a catholyte of liquid ammonia and an aqueous anolyte and maintaining between the anode and cathode a liquid partition of alkali-metal halide solution substantially free from alkali-metal uncombined with halogen and substantially free of halogen uncombined with alkali-metal by maintaining a gradual flow of said solution between the anode and cathode in a general direction from the former toward the latter at a sufficient rate to prevent substantial intermingling of liberated alkali-metal with liberated halogen.

9. In the electrolysis of sodium chloride in liquid ammonia, the method which comprises providing a catholyte of liquid ammonia and an aqueous anolyte and maintaining between the anode and cathode a liquid partition of sodium chloride solution substantially free from sodium uncombined with chlorine and substantially free from chlorine uncombined with sodium by maintaining a gradual flow of said solution between the anode and cathode in a general direction from the former toward the latter at a sufficient rate to prevent substantial intermingling of liberated sodium with liberated chlorine.

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