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3,135,673

PROCESS AND APPARATUS FOR ELECTROLYZING SALT SOLUTIONS

Filed May 5, 1961

FIG. 1

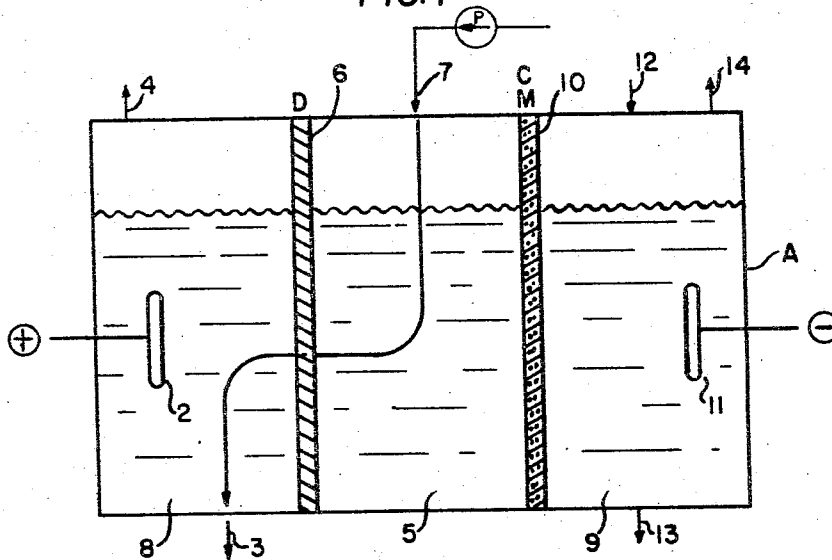


FIG. 2

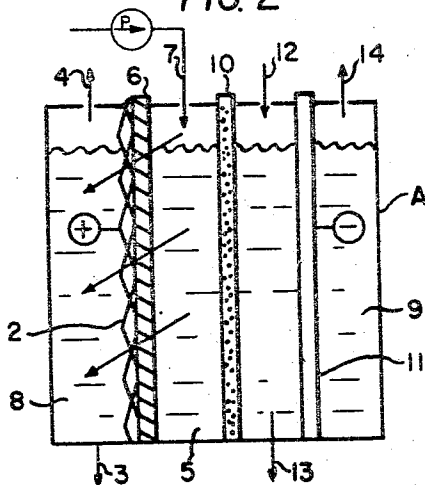
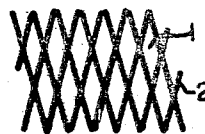


FIG. 3



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PROCESS AND APPARATUS FOR ELECTROLYZING SALT SOLUTIONS

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This invention relates to electrolytic processes and apparatus for the electrochemical conversion of aqueous electrolyte solutions; and more particularly relates to processes and apparatus which employ a three-compartment electrolytic cell containing an ion-exchange membrane selectively permeable to cations defining the cathode compartment and a spaced acid-resistant hydraulically permeable diaphragm defining the anode chamber and into the center compartment of which cell is passed the electrolytic salt solution. Tap water or other conducting solutions are passed into the cathode compartment and an electric current is impressed upon the cell causing migration of cationic ions of the electrolyte through the cation permselective membrane into the cathode compartment where combination with hydroxyl ions produced by the electrolysis of water at the cathode produces the corresponding metal hydroxide; and migration of the anionic groups of the electrolyte plus a small portion of the cationic ions of said electrolyte through the hydraulically permeable diaphragm into the anode compartment where combination of the anionic groups of the electrolyte with hydrogen ions produced by the electrolysis of water at the anode produces the corresponding acid which, mixed with the original electrolyte forms an anolyte effluent product such a product, for example, acid salt.

The processes of this invention relating to the conversion of soluble salts into their corresponding metal hydroxides and acid salts may be directed in general toward those applications wherein it is desired to recover acid salt and base values from substantially waste salt solutions, in particular, the waste sodium sulfate liquor derived from the neutralization of caustic cellulose acetate in the spinning bath operation of the viscose rayon industry or from pickle liquor of the steel industry.

Electrolytic cells, for example caustic-chlorine cells, now commonly employed for salt conversion are of two general types: (1) diaphragm and (2) mercury, and are distinguished chiefly by the purity of the corresponding metal hydroxide produced and production per unit floor space. Mercury cells are of such design that the product hydroxide solution is of a high degree of purity, i.e., the hydroxide may have a contaminating foreign anion concentration of less than about 0.05% of the concentration of the hydroxide, this advantage being the mercury cell's principal inducement toward its employment in commercial installations. Mercury cells typically employed in the electrolysis of waste sodium sulfate spinning liquor consist of an electrolyzing chamber containing a sulfate-resistant alloyed anode and a cathode consisting of a flowing bed or rotating vortex of mercury which reacts with sodium ions to form a sodium amalgam film upon its surface; subsequently this sodium amalgam-mercury composition is withdrawn from the cell and passed through an amalgam decomposing chamber wherein a flow of water countercurrent to the flow of mercury effluent converts the amalgam to a substantially pure aqueous solution of sodium hydroxide. Disadvantages of this process are the high initial capital investment necessitated by the large inventory of mercury required, a relatively high energy consumption, an undesirably high ratio of floor space per unit of product and relatively low power

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efficiencies resulting from the wider spacing between the electrodes.

Diaphragm type electrolytic cells overcome these cited disadvantages to a satisfactory extent. However, they are characterized by the production of a product metal hydroxide solution which has a considerably low degree of purity. Cells of this category employ one or more diaphragms permeable to flow of electrolyte solution, but impervious to passage of gas bubbles, thus separating the cell into two or more compartments; for example, a three-compartment cell containing two porous diaphragms is described in U.S. Patent No. 1,126,627. The processes associated with this type of cell comprise the steps transferring from either or both the anode and the center compartment to the cathode compartment by means of permeation through the diaphragm(s) a relatively concentrated aqueous salt solution which is passed into the anode or center compartment at a rate sufficient to repress the flow of hydroxyl ions toward the anode, thereby producing as a catholyte effluent the corresponding metal hydroxide and free hydrogen gas; while at the anode, free oxygen gas is produced and, in the case of the original salt solution consisting of sodium sulfate, a mixture of the salt and the corresponding acid. Employment of diaphragm-type electrolytic cells achieves relatively high production of products per unit floor space, lower energy requirements and higher current efficiencies but incurs the serious disadvantage of producing as the catholyte effluent product the hydroxide solution in a very dilute and impure form, i.e., the product may consist of about 12% hydroxide and about 12% of the original salt. In the use of a saturated NaCl feed solution, concentration and separation of the hydroxide by the use of three-stage evaporators is required wherein the salt crystallizes out, and a final NaOH concentration of about 50% is achieved. The hydroxide obtained using these supplementary and expense-incurring processes is still contaminated by about 1% of the original salt and therefore falls short of the purity achieved in a mercury cell which produces a caustic well within the specification limits of rayon grade caustic soda. It has been a consistent feature of every diaphragm-type electrolytic cell proposed heretofore, that the product hydroxide is characterized by a purity below that value signifying a "rayon grade" caustic, due to the permeation of the anion groups of the original salt through the non-permselective diaphragm separating the cathode from the feed electrolyte solution.

It has been proposed in U.S. Patent No. 2,681,320 that permselective membranes be interposed in an electrolytic cell in place of non-permselective diaphragms in order that such "rayon grade" caustic be achieved. However, no provision is made for a three-compartment electrolytic cell containing both a permselective membrane and a non-permselective diaphragm. In the instance of employment of one or more cation permselective membranes, the hydrogen ions formed by the electrolysis of water at the anode will migrate through the membrane or membranes with the metallic cations to neutralize what hydroxyl ions are being formed at the cathode. In the instance of employment of a three-compartment cell, the center compartment of which is bounded on the anode side by an anion permselective membrane and on the cathode side by a cation permselective membrane, the same phenomenon of migration toward the cathode of hydrogen ions formed at the anode through intervening hydrogen ion-permeable barriers will occur due to the inherent inefficiency toward repression of hydrogen ion permeation of those anion permselective membranes known in the art. For example, starting with a 1.0 normal sodium sulfate solution introduced into the center compartment, a comparatively high efficient anion membrane will function in such a manner that about 50% of the current passing

through said membrane is conveyed by the passage of hydrogen ions through said membrane toward the cathode. The efficiency is lower with higher and commercially more suitable concentrations of salt solution. Consequently, the insertion of an anion permselective membrane in place of a non-permselective diaphragm in this case, is of no value due to the membranes low permselectivity.

In U.S. Patent No. 3,017,338 an electrolytic process and cell for making caustic soda and chlorine are disclosed employing a spaced porous diaphragm and cation exchange membrane defining a three chamber cell. The present invention employs a different novel arrangement of the porous and cation exchange membrane with the procedure for conversion of the feed solution such as to effect a much wider and useful application of the principle of electrolytic conversion of feed electrolytes as will be more fully hereinafter disclosed.

Construction of a three-compartment electrolytic cell wherein the partition between the anode compartment and the center compartment is a porous non-selective diaphragm and wherein the partition between the center compartment and the cathode compartment is a cation permselective membrane allows management of the electrolysis in such a manner that passage of the feed electrolytic solution into the center compartment may be controlled to such a rate that the consequent flow of electrolytic solution through the diaphragm is sufficiently rapid to substantially prevent the undesired migration of hydrogen ions from the anode to the cathode. The minimizing of the migration of hydrogen ions toward the cathode by a countercurrent flow of electrolytic solutions is effected substantially by means of a porous non-permselective diaphragm inserted between the anode and the influent electrolytic solution.

It has been discovered in accordance with the present invention, that such a three-compartment cell not only prevents substantially the flow of hydrogen ions toward the cathode but also furnishes as the catholyte effluent product a "rayon grade" caustic. The same corresponding purity of product is similarly obtained in the electrolytic conversion of other feed salt electrolyte such as pickle liquor, waste Na_2SO_4 liquor, etc.

Another object of this invention is the provision of a method for electrochemical conversion of aqueous electrolyte solutions of high purity products overcoming many of the disadvantages of the prior art.

Another object of the present invention is to provide an improved process for the regeneration of waste sodium sulfate liquor obtained from the spinning operations in the viscose rayon industry.

It is a further object to provide such a process wherein the resultant caustic soda product is of "rayon grade" purity and the product sodium acid sulfate is of a composition suitable for use in rayon spinning operations.

It is a further object to provide a method whereby electrolytic salt solutions can be electrolyzed in such manner as to produce salt-free product hydroxide and the corresponding acid salt.

It is a further object to provide an electrolytic method for the preparation of organic acids and inorganic acids of slight ionization from the corresponding soluble salts.

It is a further object to provide a method for the electrolysis of salt solutions wherein the migration to the cathode of hydrogen ions formed at the anode is effectively prevented.

Still further objects will appear from the following description and appended drawing and claims.

The foregoing and related objects are accomplished by employment of a three-compartment electrolytic cell containing one ion-exchange membrane selectively permeable to cations and an acid-resistant hydraulically permeable diaphragm, such cells diagrammatically illustrated in vertical cross-section in the drawings which do not in any way represent an attempt to construct the

concept of the invention which is sufficiently broad to permit variations and modifications of such electrolytic cells.

FIGURE 1 is a diagrammatic representation of the electrolytic cell of the present invention in vertical cross section.

FIGURE 2 is a slight variation of the cell of FIGURE 1 wherein the anode is of expanded metal and forms the support for the porous diaphragm.

FIGURE 3 is a further representation of the configuration of the expanded metal anode of FIGURE 2.

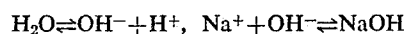
In FIGURES 1 to 3 the anode compartment 8 of cell A contains an acid resistant anode and is provided with outlet 3 for the anolyte effluent product and outlet 4 for gaseous anode products such as oxygen. The anode compartment is separated from the center compartment 5 by means of an acid resistant hydraulically permeable non-permselective diaphragm 6, said center compartment 5 containing an inlet 7 through which the electrolytic feed solution is introduced. The cathode compartment 9 is separated from the center compartment by a cation-exchange membrane 10 selectively permeable to cations and such cathode compartment is provided with a cathode 11 and conduit 12 through which water is passed into the cathode compartment. Outlet 13 serves to withdraw the catholyte effluent product, and exit pipe 14 removes gaseous cathodic products such as hydrogen. Some modifications of this general assembly of apparatus will be described hereinafter.

In the general aspect of operation of this invention, the electrolyte solution, for example, a nearly saturated solution of sodium sulfate, is introduced into the cell through conduit 7 at a rate and pressure sufficiently high so that the passage of said electrolyte through the porous diaphragm 6 is at a rate sufficiently rapid to curtail substantially the migration of hydrogen ions from the anode toward the cathode.

Simultaneously water is passed into the cathode compartment at a rate corresponding to the concentration of hydroxide desired in the resultant catholyte effluent product, and a direct electric current is impressed upon the cell. Upon the influence of the impressed electric current, the cationic constituents of the electrolytic solution for example, sodium ions, pass through the cation permselective membrane into the cathode compartment where combination with hydroxyl ions produced at the cathode by the electrolysis of water produces the corresponding hydroxide, sodium hydroxide, which is withdrawn thorough outlet 13 in a concentration dependent upon the rate of flow of water into the cathode compartment. The electrolytic solution of the center compartment 5 now having been partially depleted of its positive ions passes through the diaphragm into the anode compartment where combination of its free anionic groups with hydrogen ions produced by electrolysis of water at the anode produces the corresponding acid, for example, sulfuric acid, which is withdrawn together with unreacted electrolytic solution through outlet 3 as a mixture of the original salt and its corresponding acid salt, for example, sodium bisulfate. In the case, for example wherein 50% of the sodium ions of a sodium sulfate solution are effectively transferred to the cathode compartment, the resultant anolyte effluent product will be a stoichiometric solution of sodium bisulfate, such solution being suitable for use in rayon spinning operations.

The processes may be more clearly understood with reference to the following series of equations wherein the electrolytic salt employed is sodium sulfate.

- (1) At the cation exchange membrane 10: Na^+ [center compartment] $\rightarrow$ Na^+ [cathode compartment]
- (2) At the cathode 11:

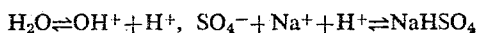


- (3) At the diaphragm 6: $\text{Na}^+ + \text{SO}_4^-$ (excess) [center

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compartment] $\rightarrow$ $\text{Na}^+ + \text{SO}_4^-$ (excess) [anode compartment]

(4) At the anode 2:



The electrolyte employed may be any water soluble electrolytic material such as inorganic salts, acids and bases and organic salts. In the case that the electrolyte is of a basic or acid nature, the processes disclosed herein are applicable toward its purification of ionic impurities; such as cationic impurities, in the instance of the electrolyte being of acidic nature, and anionic impurities in the instance of the electrolyte being of basic character. The employment of an electrolytic salt whose corresponding acid is weakly ionized, for example, sodium acetate or potassium fluoride, permits, as a result of such anodic formation of the slightly ionized acid a relatively small quantity of free hydrogen ions available for migration to ward the cathode, with a corresponding reduction in the flow rate of the feed electrolytic solution to conform to the degree of ionization of the corresponding acid, whereby said reduction in flow rate permits a comparatively high degree of transference of the alkali metal cation into the cathode compartment, and correspondingly, less of the original electrolyte solution passing through the diaphragm into the anolyte compartment. Employment of such salts in the process of this invention thereby produces a catholyte effluent product, of the corresponding caustic at a comparatively high rate and a production of anolyte effluent of the corresponding acid, associated with a comparatively small amount of the original electrolytic salt. It is apparent that the processes of this invention thus afford efficacious methods for the preparation of organic acids and inorganic acids of slight ionization from their corresponding soluble salts.

Application of these processes toward the electrolysis of inorganic salts whose corresponding acids are strongly acidic, for example, sodium sulfate or potassium chloride, results in the production, as the anolyte effluent product, of a mixture of such acid with the original inorganic salt, the ratio between the two constituents being determined by the rate at which the electrolytic solution is introduced into the center compartment. In the case of the electrolyte being a salt of a dibasic or tribasic acid, the anolyte product will be the corresponding acid salt and accompanied to a small degree by some of the original salt. The flow rate of the electrolytic solution may be regulated so that the salt content of the anolyte product is of a desired value, for example, in the case of sodium sulfate, the flow of an aqueous solution of the same into the center compartment may be regulated so that the anolyte effluent product is solely a solution of sodium bisulfate, i.e., the rate will be controlled so that the equivalents of the original salt entering the anode compartment are equal to the equivalents of sulfuric acid being formed therein.

The temperature of the electrolytic feed solution and the catholyte influent water may vary from above its freezing point to below its boiling point. However, in general, it is preferred that relatively high temperatures be employed, i.e., above about 60° C., since the impressed voltage required to pass a specific current through an electrolytic cell tends to vary inversely with the temperature of the electrolytic medium.

The anode employed is of acid-resistant conductive material and may be, for example, of platinum, rhodium or noble metal coated tantalum or titanium. The cathode is of conventional construction, being conveniently of steel or a carbonaceous material. The electrodes may properly be positioned in either of two methods. An electrode may be fixed to a side of its compartment or suspended in it by conventional means. It may, depending upon the nature of the diaphragm or permselective membrane employed, be necessary or desirable to utilize the electrode as a support for such diaphragm or membrane

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in which case the electrode is constructed in the form of a screen or expanded metal and positioned contiguous to the partition being supported.

Such a cell incorporating an expanded metal anode is diagrammed in FIGURE 2, and a section of the anode itself in FIGURE 3.

Expanded metal is available commercially. The expanded mesh is made by cutting a series of fine slots in a sheet of the desired metal and pulling the sheet lengthwise, resulting in the expansion of the slots to form triangular diamond-shaped holes. As seen in FIGURE 3, the triangular shaped holes (1) can be made in various dimensions so that the total available surface area of the metal (2) will vary correspondingly. For use as an anode the expanded material is fabricated from an electrolytic valve metal preferably tantalum, titanium or niobium which is coated with a noble metal such as platinum or rhodium.

Although the use of noble metal coatings of electric valve metals as anodes is well known in the art, the combination of such expanded metals as both anode and a support media for a porous diaphragm makes it unique. Such support makes it possible to employ diaphragms (less than .010" thick) in electrolytic cells without adding appreciably to the total electrical resistance of the electrolytic cell. Without such support, it is not possible to use thin and fragile diaphragms by themselves. Expanded metal anodes have an advantage over microporous metal or carbonaceous anodes in that disengagement of those gases produced at the anode surface during electrolysis is more readily accomplished.

The non-permselective diaphragm is of such design that it will allow passage of electrolyte solution but restrict flow of gaseous products, such as oxygen and is preferably of such suitable microporous materials such as rubber, ceramic, polyethylene, canvas, asbestos, Teflon and other synthetic fabrics.

The cation permselective membrane is commonly of the type consisting of cation exchange resin prepared in the form of thin sheets; said membranes being substantially impervious to water and to ions carrying a negative charge but permeable to ions carrying a positive charge. Permselectivity toward cations is defined as the membrane's possession of a higher transport number for cations than that of the solution in which it performs. It is essential that the membrane employed in the processes of the present invention have as high a cation transport number as possible and be substantially non-permeable to anions.

The art contains many examples of cation exchange materials which can be formed into cation permselective membranes. The mechanism underlying the operation of an ion-exchange membrane is determined from its construction which consists of a polymeric structure containing dissoluble ionizable radicals, one ionic compound being fixed into the polymeric matrix, the other a mobile and replaceable ion electrostatically associated with the fixed component. The replacement of the mobile ions by ions of like sign in the solution in which the membrane is immersed is the particular property of such membranes which characterizes it as an ion exchange material. The type of cation exchange membrane ordinarily affording the highest permselectivity toward cations is that in which carboxylic acid groups are fixed into a polymeric matrix, the preparation of one such preferred type of membrane being described in U.S. Patent 2,731,408, wherein is disclosed a method for preparing membranes consisting of a matrix of copolymers of divinyl benzene and an olefinic carboxylic compound. For example, divinyl benzene and an olefinic carboxylic compound such as an anhydride, ester or chloride derivative of acrylic acid are copolymerized in a suitable solvent. The solid polymeric material recovered is then saturated with water or an aqueous solution of an acid or base to convert the anhydride, ester or acid radicals in the polymeric matrix to their acidic or

alkali metal salt form. Alternative cation exchange materials may be prepared by the condensation of resorcylic acid with formaldehyde, by the use of sulfonated or carboxylated humic materials, etc.

The resinous material is incorporated into a sheet-like reinforcing matrix in order to increase the mechanical strength and heat resistance of the membranes. Suitable reinforcing materials include, for example, woven or felted materials such as glass filter cloth, polyvinylidene chloride screen, cellulose paper, asbestos, Teflon or Saran cloth and similar porous materials of adequate strength.

The process of this invention relating to the conversion of soluble salts into their corresponding metal hydroxide and acid salts with the recovery of such products are illustrated by the following examples which are not to be construed as limiting.

Example 1

An electrolytic cell as shown in FIGURE 2 was operated to convert an electrolyte of sodium sulfate into sodium hydroxide and its acid salt of sodium bisulfate. It employed a platinized expanded titanium anode 2 on which was supported a .010" microporous polyethylene diaphragm 6. This diaphragm thus defines and separates the anode compartment 8 from the center compartment 5. The cathode compartment 9 contained a nickel screen cathode 11 and was separated from the center cell by a self-supporting carboxylic type cation-exchange membrane 10 made as a copolymer of acrylic acid and divinyl benzene. The spacing between electrodes was about 1/4". A 10% solution of sodium sulfate was introduced into the center compartment through conduit 7 at such a rate so that the percolation of this electrolyte through the porous diaphragm and expanded anode is sufficient so as to appreciably prevent the migration or diffusion of anodic products into the center compartment. Since the positively charged hydrogen ions formed at the anode surface tend to travel in the direction of the negatively charged cathode, the sodium sulfate electrolyte is allowed to percolate through the microporous diaphragm at a velocity greater than that of the opposing migrating hydrogen ions. The result being that the hydrogen ions are effectively washed back into the anode compartment. Water is introduced into the cathode compartment through a conduit 12 at a rate depending upon the caustic concentration desired as the catholyte product. The catholyte product and anode product effluents are recovered from outlets 13 and 3. The electrolysis was conducted at a current density of 100 amps per square foot of membrane area. The cell voltage was 4.3 volts at a solution temperature of 80° C. At steady conditions the catholyte effluent analyzed to be a 2 normal NaOH and the anode effluent analyzed to 0.75 normal acid concentration, representing a current efficiency of 90%.

Example 2

The cell configuration and components were used here as indicated in FIGURE 2, and an aqueous solvent brine solution was employed as the feed electrolyte to the center compartment. During the electrolysis of sodium chloride, the products produced at the cathode were sodium hydroxide and hydrogen gas, and at the anode, hydrochloric acid, chlorine gas and a small amount of sodium hypochlorite. The use of the porous diaphragm effectively prevented the diffusion of these highly corrosive anodic products from entering the center cell and thus not subjecting the cation membrane to chemical and physical oxidation degradation. Because of the highly corrosive nature of the anolytic solution, an inert porous Teflon diaphragm was used to separate the anode and center compartment.

The electrolysis of sodium chloride was conducted at a membrane current density of 125 amps per square foot, a cell voltage of 4.2 volts and at a temperature of 95° C. The steady state product of the cathode was 15% sodium hydroxide of a purity consistent with rayon grade caustic, representing a current efficiency of better than 90%.

The above examples represent a few typical methods of operation that can be performed with electrolytic cells of this configuration and as such are not intended to limit the area of this invention to only such operations.

It is proposed as a novel application of the processes of the present invention that they be directed toward the removal from air or other gases of carbon dioxide, said application being of particular significance with respect to purification of air in closed systems wherein it is either inconvenient or impossible to introduce a continuing supply of materials such as carbondioxide absorbing fluids; an example of such a closed system being a submarine. This application comprises the following steps and installations: (1) An electrolytic cell equivalent in design and function to that described herein is constructed and operated with an electrolytic influent feed solution of sodium sulfate, for example, 3 N. The catholyte effluent product is aqueous sodium hydroxide and the anolyte effluent product is sodium bisulfate. The anode compartment is also the site for the production of oxygen which is passed into the atmosphere of the particular system being purified. The product sodium hydroxide solution is conducted to a "scrubbing" apparatus wherein the atmosphere or particular gas whose purification is desired is passed and its content of carbon dioxide absorbed by the caustic, forming sodium carbonate. The carbonate-containing solution is then (3) combined with an equivalent amount of the anolyte effluent product sodium bisulfate to regenerate the original sodium sulfate solution. This sodium sulfate solution is recycled (4) to the electrolytic cell. Since the sodium sulfate is thus substantially regenerated, the production of oxygen is dependent wholly upon the electrolysis of water, whose source is ordinarily readily available.

What we claim is:

1. The method of converting aqueous electrolytic salt solutions to their corresponding acid and base solutions comprising: passing said salt solution into the center compartment of a three-compartment electrolytic cell having a cathode compartment separated from the center compartment by a cation-selective ion exchange membrane and a spaced fluid-permeable diaphragm separating the center compartment from the anode compartment, maintaining a greater pressure in said center compartment than the pressure in the anode compartment to cause said feed salt solution to flow from the center compartment through said porous diaphragm into and out of said anode compartment, introducing an electrolyte solution into said cathode compartment, passing a direct electric current transversely through said compartments, diaphragm, and membrane and removing the corresponding acid and base solutions from the anode and cathode compartments respectively.

2. The method of claim 1 wherein the solution introduced into said cathode chamber is tap water.

3. The method of claim 1 wherein the feed salt solution is sodium sulfate and the products obtained are essentially solutions of sodium hydroxide and sodium bisulfate.

4. The method of claim 1 wherein the feed salt solution is sodium chloride and the products obtained are essentially solutions of sodium hydroxide and hydrochloric acid.

5. A three compartment electrolytic cell comprising a cathode compartment, a center compartment, and an anode compartment, the cathode compartment being separated from the center compartment by a cation selective ion exchange membrane, the center compartment being separated from the anode compartment by a fluid-permeable diaphragm, the center compartment containing only an inlet for passing a feed salt solution therein, means for maintaining a pressure in said center compartment greater than the pressure in the anode compartment to cause said feed solution to pass from said center compartment into said anode compartment, the anode compartment containing only an outlet for withdrawing a product solution

from said compartment, an inlet and outlet in said cathode compartment for passing an aqueous electrolyte solution there-through, and an anode and a cathode in the respective end compartments for passing a direct current transversely through said compartments, membrane and diaphragm.

6. The cell of claim 5 wherein the fluid-permeable diaphragm is an asbestos sheet.

7. The cell of claim 5 wherein the anode consists of expanded metal backed against said diaphragm for physical support of the latter.

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