United States Patent Office 3,799,849 Patented Mar. 26, 1974

3,799,849

REACTIVATION OF CATHODES IN CHLORATE CELLS

Edward H. Cook, Jr., Lewiston, and Daniel J. Snyder, Depew, N.Y., assignors to Hooker Chemical Corporation, Niagara Falls, N.Y.

No Drawing. Continuation-in-part of application Ser. No. 98,178, Dec. 14, 1970, which is a continuation-in-part of application Ser. No. 688,985, Dec. 8, 1967, both now abandoned. This application June 26, 1972, Ser. No. 266,090

Int. Cl. C01b *11/26*

U.S. Cl. 204—95 4 Claims

ABSTRACT OF THE DISCLOSURE

Steel cathodes which have become deactivated by exposure to an electrolyte containing in excess of 300 grams per liter alkali metal chlorate during a shutdown period in chlorate cell operation are reactivated by resuming electrolysis with an alkali metal chloride brine having a chlorate concentration below 300 grams per liter, continuing electrolysis while maintaining this lower level of chlorate concentration in the brine until the hydrogen efficiency of the cell reaches at least 95% and, thereafter, increasing the chlorate concentration in the electrolyte to above 300 grams per liter.

This application is a continuation-in-part of copending application Ser. No. 98,178, filed Dec. 14, 1970, now abandoned, which application is a continuation-in-part of Ser. No. 688,985, filed Dec. 8, 1967, now abandoned.

This invention relates to an improved process for the operation of electrolytic cells for the production of alkali metal chlorates and, more particularly, relates to a method for reactivating the steel cathodes utilized in alkali metal chlorate cells.

Heretofore, alkali metal chlorates have conventionally been produced in diaphragmless electrolytic cells having graphite anodes and steel cathodes. Typically, these cells have been operated at temperatures of from about 40 to 45 degrees C. and the alkali metal chlorate content of the electrolyte has been from about 50 to 500 grams per liter. In these operations, the hydroxide ion migrates to and reacts with chlorine produced at the anode, the reactions occurring in the process being expressed by the following equations:

$$(1) \quad 3Cl_2+6OH^-\rightarrow 3ClO^-+3Cl^-+3H_2O$$

$$(2) \quad 2ClO^-+2H^+\rightleftharpoons 2HClO$$

$$(3) \quad 2HClO+ClO^-\rightarrow ClO_3^-+2Cl^-+2H^+$$

The overall reaction taking place in the process may be expressed as follows:

$$(4) \quad 3Cl_2+6OH^-\rightarrow ClO_3^-+5Cl^-+3H_2O$$

In a typical commercial operation, the cells are operated with a slightly acid electrolyte so as to favor the formation of HClO, as shown by Equation 2, above. Although the cells may be operated to approach a current efficiency of about 90% and a production of hydrogen at the cathode which is at nearly 100% efficiency, i.e. at at least about 95% efficiency, a reduction in product yield and loss and current efficiency may take place, generally as a result of the reduction of hypochlorite and chlorate ions at the cathode.

To minimize the reduction of hypochlorite and chlorate ions at the steel cathode, it is presently the practice to add a small amount of an alkali metal dichromate, such as sodium dichromate, to the feed brine of the cell. The alkali metal dichromate is utilized in an amount sufficient to inhibit the oxidation of the steel cathode during startup periods and temporary shutdowns. Where the alkali metal dichromate used is sodium dichromate amounts of from about 0.01 to about 1% by weight of the feed solution are typical. Although, as an alternative, the reduction of chlorate at the cathode may be minimized by forming the cathode from stainless steel or a chromium containing steel, this adds appreciably to the equipment cost. The use of such cathodes is, therefore, not the most preferred expedient for minimizing chlorate reduction at the cathode.

Recently, there have been two innovations in the art of electrolytic chlorate production which have resulted in a material change in operating conditions from those utilized in conventional chlorate cell operations. These two improvements are the use of dimensionally stable or metallic anodes instead of the conventional graphite anodes and the development of diaphragm type chlorate cell processes, as disclosed in U.S. Pats. 3,464,901 and 3,516,918. When using either or both of these developments, the cells are operated at temperatures above 50 degrees C., typically in the range of about 70 to 95 degrees C., and the alkali metal chlorate content of the electrolyte is generally in excess of about 300 grams per liter. Although operation under these conditions is advantageous, particularly in terms of attaining higher anode current efficiencies and lower cell voltages, deactivation of the cathode during shutdown periods is found to occur much more rapidly and to be much more severe than has heretofore been experienced.

It has been found that when there is a temporary shutdown of the cells which are operating under these conditions of higher temperature and electrolyte chlorate content, upon startup of the cells again, the deactivation of the cathode has been sufficient that there is an appreciable reduction in the efficiency of hydrogen production at the cathode, with a corresponding drop in the product yield and cathode current efficiency. Frequently, the production of hydrogen efficiency is reduced from the normal 95 to 100% range to 10%, or even less. Attempts to overcome this problem by the addition of alkali metal dichromates to the electrolyte have not been successful.

It is, therefore, an object of the present invention to provide an improved method for reactivating the steel cathodes used in the electrolytic production of alkali metal chlorates.

A further object of the present invention is to provide an improved method for reactivating cathodes of electrolytic chlorate cells, which cells are operated at temperatures above about 50 degrees C. and electrolyte chlorate contents in excess of about 300 grams per liter.

These and other objects will become apparent to those skilled in the art from the description of the invention which follows.

Pursuant to the above objects, the present invention is directed to an improvement in the process for producing alkali metal chlorates wherein an aqueous alkali metal chloride electrolyte containing from about 300–650 grams per liter chlorate is electrolyzed, at a temperature from about 50–105 degrees C., by passing an electric current through the electrolyte between an anode and a steel cathode and the passage of the electric current is discontinued, while the cathode remains in contact with the electrolyte for a period sufficient to deactivate the cathode, which improvement comprises reducing the chlorate content of the electrolyte to below 300 grams per liter, passing an electric current through the electrolyte between the anode and the steel cathode for a period sufficient to increase the hydrogen efficiency of the steel cathode to 95% and increasing the chlorate content of the electrolyte to above 300 grams per liter when the hydrogen efficiency of the cathode reaches 95%, thereby reactivating the steel cathode. By this method, reactivation is effected of deactivated steel cathodes which would not be reactivated

by the addition of even relatively large amounts of sodium dichromate to the electrolyte solution.

More specifically, in the practice of the method of the present invention, electrolytic cells having an anode and steel cathodes, are operated with an aqueous alkali metal chloride electrolyte which also contains from about 300 to 650 grams per liter chlorate. This electrolyte is electrolyzed, at a temperature of from about 50 to 105 degrees C., by passing an electric current through the electrolyte between an anode and the steel cathode. In the course of this process, the passage of the electric current is discontinued while the steel cathode remains in contact with the electrolyte. The discontinuance of the passage of electric current results from temporary shutdowns of the cell operation, which may be caused by a disruption in the transmission of electrical power or by the need for maintenance or repair of the cells, or the like. As a result of the high temperatures and chlorate content of the electrolyte, the steel cathodes become deactivated even after a shutdown period of only a few minutes. The deactivation which occurs is sufficiently severe that, upon startup of the electrolysis operation, the hydrogen efficiency of the cell has been reduced appreciably below 95%, which is the desired minimum efficiency for operation.

To overcome this problem and effect reactivation of the steel cathode, the chlorate concentration of the electrolyte is reduced to below 300 grams per liter, prior to resuming electrolysis and the electrolysis is then carried out at this reduced chlorate concentration until the hydrogen efficiency of the cathode reaches 95%, whereupon the chlorate content of the electrolyte is increased to above 300 grams per liter. This reduction in the chlorate content of the electrolyte to below 300 grams per liter, may be effected in any convenient manner. Preferably, this is done by adding to the electrolyte an aqueous alkali metal chloride brine which is substantially free of chlorate ions. In this manner, not only is the chlorate content of the electrolyte reduced but, additionally, the chloride ion content of the electrolyte is increased from its normal range of about 90 to 140 grams per liter up to from about 200 to 280 grams per liter, which increased chloride content has, in many instances, also been found to be desirable. Alternatively, the reduction of the chlorate content of the lectrolyte may be accomplished simply by adding water to the electrolyte in amounts sufficient to dilute it to the point that the desired lower chlorate content is obtained.

Although it is not known for certain, it is believed that the deactivation of the steel cathode may be the result of the formation of a surface coating of an iron oxide which may serve to catalyze the decomposition of chlorate and hypochlorite ions. Where the alkali metal chlorate content of the electrolyte is in excess of about 300 grams per liter when the cell is started after a temporary shutdown, for some unknown reason, the presence of these chlorate ions and/or hypochlorite ions appears to stabilize the deactivating factor of the steel cathode. Thus, although in theory, the chlorate concentration should be decreasing in the electrolyte, it appears to be in a state of equilibrium and must be physically decreased, in the manner indicated hereinabove, before the cathode will be reactivated during electrolysis processes carried out at temperatures between 50–105 degrees C.

As has been heretofore set forth, the production of alkali metal chlorates in accordance with the present invention may be carried out in processes such as those disclosed in U.S. Pats. 3,464,901 and 3,516,918. In these processes, the electrolysis is effected in a diaphragm type chlor-alkali cell which is operated under optimum conditions for the production of chlorine and caustic soda and these products are then reacted chemically to produce chlorate. This reaction is carried out at a point removed from the active electrode region and the electrolyte feed to the cell contains the chlorate which has been thus-produced chemically. The recirculation of the chlorate containing electrolyte, with makeup chloride, under conditions of continuous electrolysis, produces a cell effluent with ever increasing amounts of chlorate, so that a portion of the cell effluent may be removed to a crystallizer for chlorate recovery.

The chlor-alkali cell used in such processes to produce alkali metal chlorates can be any of the numerous types of chlor-alkali diaphragm cells in which gaseous chlorine and aqueous alkali metal hydroxide cell liquor are produced, as are known in the art. A typical cell of this type is described in U.S. Pat. 1,862,244 to Stewart. For further descriptions of typical chlor-alkli diaphragm cells, reference is also made to the ACS Monograph Series, Book No. 154, entitled "Chlorine," by Sconce (1962), Reinhold Publishing Co., pp. 81–126.

The diaphragms used in these cells may be any porous material which is resistant to the conditions within the cell. Exemplary of preferred materials which may be used are asbestos, polytetrafluoroethylene such as Teflon, after chlorinated polyvinylchloride, polyvinylidene chloride, and similar materials. Of these, the most preferred diaphragm materials are the more porous and acid resistant asbestoses such as anthophyllite and asbestos mixtures containing anthophyllite. Preferably, the diaphragm used in the cell in such processes is more porous than that which is conventionally used for normal chlorine-caustic production. Typically, when a diaphragm of deposited asbestos is used, the diaphragm weight is from about 25 to 100% of that used for normal chlor-alkali production.

The cathodes of these cells are typically of steel, while the anodes may be graphite or dimensionally stable anodes. The dimensionally stable anodes are electrodes having an electrically conductive film-forming base material supporting on its surface a coating or deposit of at least one member of the group of noble metals, noble metal alloys, noble metal oxides, lead dioxide or magnetite. Suitable film-forming substrate metals are titanium, tantalum, or niobium (columbium) either as a single metal or as thin sheets or layers of metal which are electrically connected to another electrically conducted metal, such as copper, aluminum, iron, and similar less expensive metals or their alloys.

The electrically conductive film-forming base or substrate metal of the dimensionally stable anodes are those metals and metal alloys which become passivated when polarized anodically and remain passive well beyond the anodic potential which is needed to convert chloride ions to chlorine. Thus, the base metal will remain passive under the conditions of operation of the electrolytic cell. This base metal need not be homogenous in cross-section but may be clad or bonded to an electrically conductive material such as aluminum, steel, copper, or the like, either by mechanical bonding or by means of an electrically conductive adhesive material. Moreover, the substrate metal may have a hollow core which contains a metal or mixture of metals such as sodium, potassium, or the like. These metals may be liquid at the temperature of cell operation so as to form a completely encapsulated liquid core having excellent electrical conductivity.

The surface of the substrate metal may be made active by methods known in the art, such as by the electro-deposition of a noble metal. Mixtures of noble metals as well as their alloys or oxides, may be employed to provide the active electrode surface of the substrate metal on these dimensionally stable anodes. Similarly, lead dioxide and magnetite may also be used to provide the active electrodes surface. In general, however, the preferred active metal surface is provided by at least one member selected from ruthenium, rhodium, irridium, platinum, their alloys and oxides.

It is to be further appreciated that in addition to their use in conventional chlor-alkali diaphragm cells in processes wherein the chlorate is produced externally of the cell by the chemical reaction of the caustic and chlorine,

the dimensionally stable anodes may also be used in place of the graphite anodes in conventional diaphragmless chlorate cells. In either instance, the cells are operated at temperatures in excess of about 50 degrees C., preferably within the range of about 60–105 degrees C. and most preferably at temperatures within the range of about 70 to 95 degrees C. These temperatures are at least about 5 to 15 degrees higher than those used for the production of chlorate in diaphragmless cells with graphite andoes. Additionally, the chlorate content of the electrolyte in these cells is within the range of about 300–650 grams per liter, which concentration is in excess of the concentration of alkali metal chloride in the electrolyte.

When the cells as have been described above are operated under these conditions, i.e. relatively high temperatures and high chlorate electrolyte content, undergo shut-down during which the steel cathode remains in contact with the electrolyte, it is found that in many instances substantially no hydrogen is produced at the steel cathode when the cells are again started up. This has been found to be true even when a current load of up to 6000 amperes is applied to the cells, where the steel cathode has been deactivated by exposure to the electrolyte during the shut-down period. Although chlorine is produced at the anode and caustic is produced at the cathode, the steel cathode remains deactivated and the chlorate concentration of the cell liquor remains constant. This indicates that the chlorate ion is being reduced as fast as it is formed. This situation is not overcome even by the addition of relatively large amounts of sodium dichromate to the electrolyte. It is only by reducing the chlorate content of the electrolyte to below 300 grams per liter, typically to an amount within the range of about 10 to 250 grams per liter, in the manner as has been described herein above, and continuing the electrolysis with this reduced chlorate content in the electrolyte that the steel cathodes are reactivated. It is to be noted that once the hydrogen efficiency of the cathode has been increased to 95%, the chlorate concentration of the electrolyte can again be increased to above 300 grams per liter, e.g., by the addition of alkali metal chlorate to the electrolyte.

It is to be appreciated that the processes which have been described herein for generating alkali metal chlorates may be used for the production of all of the alkali metal chlorates, such as lithium chlorate, potassium chlorate, sodium chlorate, cessium chlorate, rubidium chlorate and the like, from their respective alkali metal chlorides. In general, however, it is preferred to produce sodium chlorate from sodium chloride, by the process of the present invention, and subsequently to convert the sodium chlorate to other desired chlorates. Accordingly, the description of the present invention herein has been directed more particularly to the production of sodium chlorate. It is to be understood, however, that in this description of the production of sodium chlorate, other alkali metal chlorates are also applicable.

In order that those skilled in the art may better understand the present invention and the manner in which it may be practiced, the following specific examples are given. In these examples, unless otherwise indicated, parts and percent are by weight and temperatures are in degrees centigrade. It is to be appreciated, however, that thse examples are merely exemplary of the present invention and are not to be taken as a limitation thereof.

EXAMPLE 1

A diaphragm type chlorate cell in which the steel cathode was cleaned in hydrochloric acid and protected by application of sodium dichromate solution on the bare steel surface, was employed in this experiment. Sodium dichromate was also added to the asbestos slurry used for diaphragm deposition. The anolyte feed brine contained two grams per liter sodium dichromate. Electrolysis was first started using a 200 gram per liter sodium chloride solution containing the sodium dichromate (2 grams per liter). This solution was drained from the cell after 45 minutes and replaced with a sodium chlorate (500 grams per liter)-sodium chloride (150 grams per liter) solution containing 2 grams per liter sodium dichromate.

At a cathode current density of 0.83 ampere per square inch, the initial cell temperature was 36 degrees centigrade. During the first hour of operation the hydrogen efficiency was 97 percent. As the temperature increased to 78 degrees centigrade over an eight-hour period, the hydrogen efficiency fell to 90 percent. However, assay of the anolyte showed that the sodium chloride had decreased to 99 grams per liter sodium chloride. Attempting to increase the temperature to 83 degrees centigrade under these conditions lowered the hydrogen efficiency to 75 percent. The temperature of the cell was lowered back to 78 degrees centigrade, and additional sodium chloride was added to bring the sodium chloride content up to 133 grams per liter. After thirteen hours, a hydrogen efficiency of 96 percent was obtained at 79 degrees centigrade. Additional sodium dichromate (1 gram per liter makeup) was added to the cell. Within a few minutes, a hydrogen efficiency of 98 percent was obtained at 78 degrees centigrade. The cell was gradually heated to 86 degrees centigrade, and after fifteen hours of operation a hydrogen efficiency of 97 plus percent was obtained. After a total of seventeen hours of operation, the cell temperature was 90 to 92 degrees centigrade, and hydrogen efficiencies were still at 98 percent. The cell continued to operate at about 97 degrees centigrade until it was shut down after a total of twenty-two hours of continuous operation. The cell operated at about 90 degrees centigrade for a total of five hours. During this time the average hydrogen efficiency was 96 percent. After the thirteenth hour of operation, the sodium chloride content in the anolyte did not go below 124 grams per liter. At the end of the experiment, the sodium chlorate content in the solution was 610 grams per liter. The sodium chlorate increased with time because the catholyte was constantly neutralized to pH 3 with hydrochloric acid and added to the recirculated anolyte without correcting for evaporation losses. The cell was operated at an anode current efficiency of 97.8%.

The following data demonstrate the results of this pretreatment of the mild steel cathode in the operation of a chlorate cell. The cell was operated with an applied current of 20 amperes.

TABLE I

Temperature, °C.	H_2 evolution from cathode, cc./second	Temperature, °C.	H_2 evolution from cathode, cc./second	Temperature, °C.	H_2 evolution from cathode, cc./second
35		57	2.50	79	2.60
36	2.60	58	2.45	80	2.55
37	2.57	59		81	2.40
38	2.57	60		82	[1] 1.85
39	2.57	61	2.45	83	2.55
40	2.60	62	2.45	84	
41		63		85	2.45
42		64		86	2.55
43		65	2.40	87	2.60
44	2.50	66	2.45	88	2.65
45		67	2.45	89	2.65
46		68	2.45	90	2.65
47		69	2.45	91	2.65
48	2.45	70		92	2.65
49		71	2.40	93	2.55
50		72		94	
51		73	2.40	95	2.60
52	2.37	74		96	2.60
53		75	2.40	97	2.60
54	2.45	76	2.45	98	
55	2.40	77	2.45	99	
56	2.45	78	2.50		

[1] Low NaCl (99 g./l.).

EXAMPLE 2

A mild steel cathode having a coating of surface rust was exposed as a cathode in a chlorate-free alkali metal chloride brine solution. Electrolysis of the alkali metal chloride brine was conducted for a short time interval, less than one hour, to activate the steel cathode surface. Subsequently, a chlorate containing brine was introduced as the anolyte feed for operation of the cell as a chlorate

diaphragm cell. The results from this operation were similar to those obtained in the preceding experiment.

EXAMPLE 3

A sodium chlorate cell, having a steel cathode, was operated at a temperature of 80 degrees C. with an aqueous electrolyte containing 500 grams per liter sodium chlorate and 150 grams per liter sodium chloride. Under these conditions, the hydrogen efficiency of the cell was found to be 97%. Thereafter, the passage of current in the cell was stopped for a period of 10 minutes while the steel cathode was maintained in contact with the electrolyte solution. Upon again supplying current to the cell, it was found that the hydrogen efficiency of the cathode was only 75%. An aqueous sodium chloride brine was then added to the cell until the sodium chlorate content of the electrolyte was reduced to 250 grams per liter and the sodium chloride concentration was 170 grams per liter. After about 2 minutes of electrolysis with this reduced chlorate content electrolyte, the hydrogen efficiency of the cathode had increased to 97%, at which point the chlorate content of the electrolyte was increased to 500 grams per liter, by the addition of sodium chlorate. The electrolysis was continued and the hydrogen efficiency was maintained at 97%.

EXAMPLE 4

For purposes of illustrating the effect of dichromate addition and temperature control in conjunction with the use of a mild steel cathode which was not properly activated, the following data were obtained. A 100 percent current efficiency in this series of experiments would be represented by a hydrogen evolution of 2.67 cubic centimeters per second.

An electrolytic cell employing a graphite anode, a steel mesh cathode, and a deposited asbestos diaphragm was operated at a cathode current density of 0.83 ampere per square inch using an electrolyte containing about 500 grams per liter $NaClO_3$ plus 150 grams per liter NaCl. The rate of hydrogen evolution was monitored as a function of cell temperature and sodium dichromate concentration.

TABLE II

Temperature, ° C.	Cathode H_2 evolution rate (cc./second) at 20 amperes to the cell
No sodium dichromate:	
48	0.83–0.91
65	0.28–0.42
0.2 gram/liter $Na_2Cr_2O_7$:	
45	1.45–1.74
67	0.58–1.0
75	0.38–0.88
0.5 gram liter $Na_2Cr_2O_7$:	
60	0.75–1.74
80	0.59
1.0 gram/liter $Na_2Cr_2O_7$:	
65	1.60–1.74
80	1.38–1.44
85	1.22–1.32
2.0 grams/liter $Na_2Cr_2O_7$:	
50	2.13–2.47
60	2.17–2.32
70	1.90–2.00
80	1.43–1.54
90	1.32–1.39
5 grams/liter Na_2CrO_7:	
50	2.31–2.53
60	2.32
70	2.20–2.22
80	2.06–2.14
90	1.54–1.94

This experimental data demonstrates that at the desired operating temperatures (50 to 95 degrees centigrade), no amount of sodium dichromate (soluble hexavalent chromium) is sufficient to afford a 100 percent current efficiency when operating with an untreated or improperly treated steel cathode.

It was observed that the rate of hydrogen evolution increased with increasing sodium dichromate concentration and decreased with increasing temperature. These experiments were conducted by, first, starting the cell at 45 to 50 degrees centigrade in the absence of dichromate and observing the rate of hydrogen evolution as the temperature increased. Dichromate was then added to the anolyte feed, the cell was rapidly cooled, and once again, rates of hydrogen evolution were observed at increased temperatures. This procedure was repeated for various additions of sodium dichromate.

EXAMPLES 5–11

The steel cathode in each of the following experiments was treated preliminary to exposure to the electrolyte by etching in 10 percent hydrochloric acid followed by a caustic rinse. The cathodes were then exposed to the air. Subsequently, the cathodes were employed in the electrolysis of a solution containing 500 grams per liter of sodium chlorate, 150 grams per liter sodium chloride, and 2 grams per liter of sodium dichromate. The solution being electrolyzed was maintained at a temperature between 85 to 90 degrees centigrade and the current applied was one ampere per square inch.

	Time exposed to air	Time elapse prior to H_2 evolution
Example:		
5	0 minutes	0 minutes.
6	11 minutes	2 minutes.
7	20 minutes	4 minutes.
8	30 minutes	6 minutes.
9	60 minutes	7 minutes.
10	18 hours	Do.
11	45 hours	Do.

EXAMPLES 12–15

Control cathode for Examples 5–11

(12) The cathode used in Example No. 7 supra was air dried overnight (16 hours) and then exposed as a cathode in the same solution. No hydrogen was evolved. After 24 minutes the experiment was discontinued.

(13) A rust covered steel cathode was employed in electrolysis of the solution described in Examples 5–11. No hydrogen was evolved during a period of 27 minutes at which time the experiment was discontinued.

(14) 304 stainless steel containing 18–20 percent chromium plus 8–12 percent nickel was employed as the cathode in the solution described for Examples 5–11. A strong evolution of hydrogen was observed after one minute.

(15) A clean surface of a mild steel cathode was exposed to the anolyte solution described above for a few minutes. When an electrical current was impressed across the solution, no hydrogen was evolved at the cathode.

EXAMPLES 16–18

Three mild steel cathodes were etched in 10 percent HCl followed by a water rinse only (caustic rinse was eliminated). Upon exposure to the chlorate-containing solution at 85–90 degrees centigrade, the following results were obtained.

	Time exposed to air after rinsing	Elapsed time until H_2 evolution
Example:		
16	0 minutes	2 minutes.
17	15 minutes	7 minutes.
18	60 minutes	None after 24 minutes of observation.

The cathode used in Example 18 visibly displayed evidence of a ferric oxide surface stain.

While there have been described various embodiments of the invention, the compositions, methods and apparatus described are not intended to be understood as limiting the scope of the invention as changes therein are possible and it is intended that each element recited in any of the following claims is to be understood as referring to all equivalent elements for accomplishing substantially the

same results in substantially the same or equivalent manner, it being intended to cover the invention broadly in whatever form its principle may be utilized.

What is claimed is:

1. In the process for producing alkali metal chlorates wherein an aqueous electrolyte consisting essentially of an alkali metal chloride and 300–650 grams per liter of alkali metal chlorate is electrolyzed, at a temperature of about 50 to 105° C., by passing electric current through said electrolyte between an anode and a steel cathode, and the passage of said electric current is discontinued, while the cathode remains in contact with said electrolyte for a period sufficient to deactivate said cathode, the improvement which comprises reducing the chlorate content of the electrolyte to below 300 gm./liter by ading to said electrolyte an aqueous alkali metal chloride brine which is substantially free of alkali metal chlorate and dichromate, passing electric current through the electrolyte between the anode and the steel cathode for a period sufficient to increase the hydrogen efficiency of the steel cathode to 95% and increasing the chlorate content of the electrolyte to above 300 gm./liter when the hydrogen efficiency of the cathode reaches 95%, thereby reactivating the steel cathode.

2. The process as claimed in claim 1 wherein the alkali metal chlorate is sodium chlorate and the alkali metal chloride is sodium chloride.

3. The method as claimed in claim 2 wherein the electrolysis is carried out at a temperature of from about 70 to 95 degrees C.

4. The process as claimed in claim 3 wherein the sodium chlorate content of the electrolyte is reduced to an amount within the range of about 10 to 250 grams per liter.

References Cited

UNITED STATES PATENTS

536,848	4/1895	Blumenberg	204—95
2,687,993	8/1954	Cox	204—145 R
3,464,901	9/1969	Grotheev et al.	204—95

FREDERICK C. EDMUNDSON, Primary Examiner