Sept. 29, 1936. J. W. BOSS 2,055,962

APPARATUS FOR PRODUCTION OF CHROMIC ACID AND CAUSTIC ALKALI

Original Filed Feb. 16, 1935

Fig. 1.

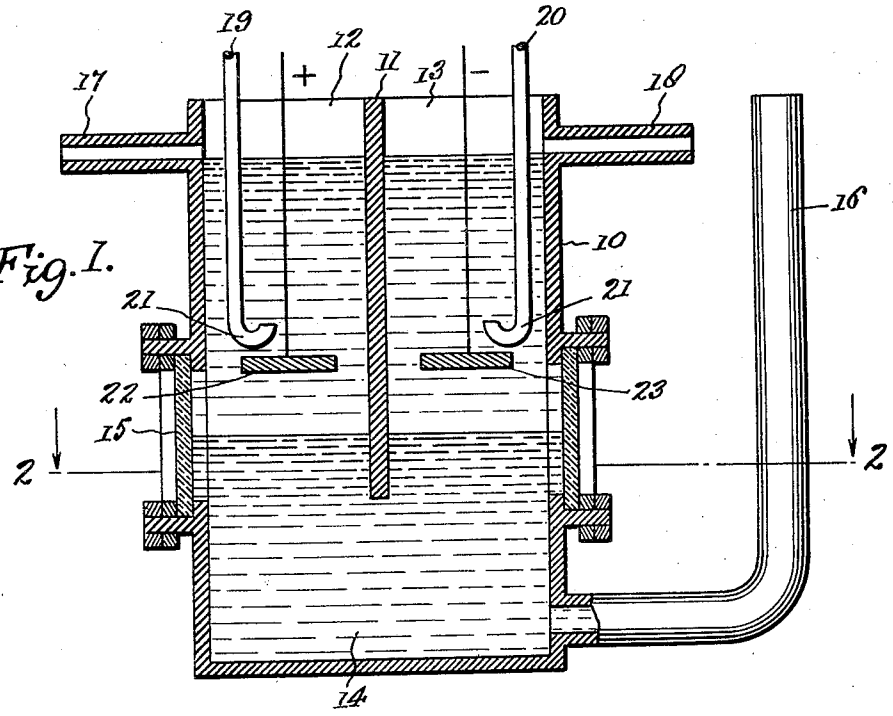
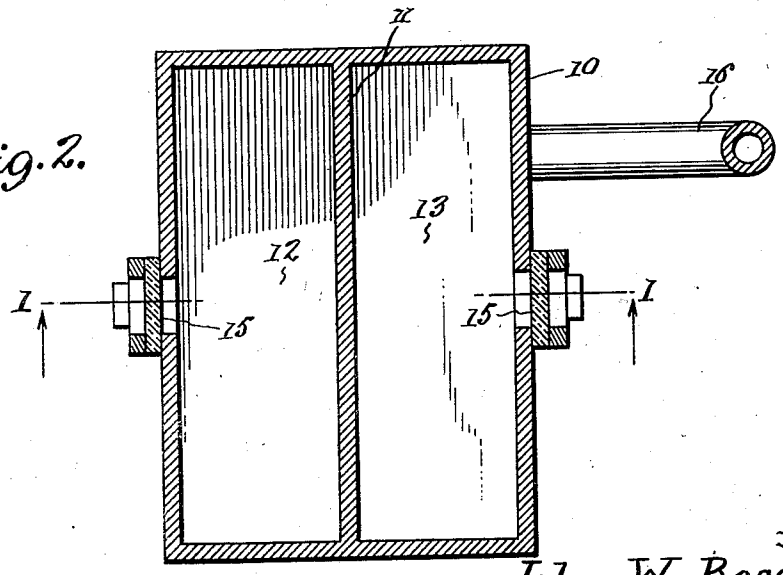


Fig. 2.



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APPARATUS FOR PRODUCTION OF CHROMIC ACID AND CAUSTIC ALKALI

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Original application February 16, 1935, Serial No. 6,904. Divided and this application June 8, 1935, Serial No. 25,653

5 Claims. (Cl. 204—5)

The present application is a division of my copending application No. 6,904 filed February 16, 1935.

This invention relates to a process and apparatus for the electrolytic production of chromic acid and of caustic alkali.

In the prior art it is customary to produce chromic acid by the action of an acid such as sulphuric on an alkali chromate or dichromate such as sodium chromate or dichromate. Separation of the chromic acid is accomplished by crystallization and draining off the mother liquor containing in solution other products of the reaction.

Some of the disadvantages of such a process are that difficulty is encountered in removing other acid radicals from the chromic acid, and that the alkali originally combined as chromate or dichromate appears as a salt such as sodium sulphate. This salt is not of much value and is not suitable for use in preparing new quantities of sodium chromate.

I am aware that in the prior art, an electrolytic cell containing a diaphragm has been used for the purpose of converting alkali chromates to alkali dichromates. This process is limited to the preparation of dichromates and employs a diaphragm rendering the construction of the cell more complex and greatly increasing the electrical resistance encountered. An object of the present invention is to produce by electrolysis from water soluble chromates, dichromates or mixtures of chromic acid and salts of chromic acid; a solution floating in the anode chamber of the cell which solution has a substantially greater ratio of chromic acid to base than has the feed solution, and a floating solution in the cathode chamber of the cell which has a substantially greater ratio of base to the chromic acid than has the feed solution, the above described result to be attained without the use of a diaphragm.

A further object of the invention is to produce chromic acid free from other acid radicals and from alkali salts.

A further object is to produce chromic acid electrolytically. An additional object is to revivify used chromium plating solutions.

Another object of the invention is to provide an apparatus or multiple chamber cell adapted to function with three different solutions and wherein means are provided for maintaining these solutions at their desired compositions.

The invention has for a further object to provide a multiple chamber cell wherein the planes of separation of the solutions may be observed.

With the foregoing and other objects in view, the invention will be more fully described hereinafter, and will be more particularly pointed out in the claims appended hereto.

In the drawing, wherein like symbols refer to like or corresponding parts throughout the several views,

Figure 1 is a vertical transverse section taken through a cell constructed according to the present invention substantially on the line 1—1 of Figure 2 and

Figure 2 is a horizontal section taken through the lower end of the cell on the line 2—2 of Figure 1.

Referring now to the drawing which shows one form of the improved apparatus of this invention by means of which the improved process of this invention may be practiced, 10 designates the body of the cell which may be of substantially rectangular form and having appreciable depth and which may be made of glass, stoneware or other suitable material for containing chromic acid and caustic alkali. The cell is divided longitudinally by an intermediate partition 11 extending from the upper end of the cell down to about two-thirds or three-quarters of the cell depth and dividing the cell into opposed chambers 12 and 13 in the upper part thereof. The chambers 12 and 13 intercommunicate at their lower ends through a bottom chamber 14. The cell 10 is provided in one or both sides with windows 15 which may comprise panes of glass or other transparent material sealed over openings in the walls of the cell and which are disposed above the horizontal plane of the lower end of the partition 11. Of course these windows 15 may be located in other walls of the cell or disposed at any other points of vantage for carrying out the purpose. The bottom chamber 14 of the cell is open to a feed pipe 16 which may open at any suitable point into this chamber for supplying thereto feed solution.

The cell body 10 is provided at its upper end with overflow pipes 17 and 18 which communicate at their inner ends with the upper chambers 12 and 13 respectively and at a desired height in the upper chamber to maintain a definite surface level in said chambers.

The solution supply pipes 19 and 20 lead into the tops of the chambers 12 and 13 respectively for introducing thereto water or for introducing into each upper chamber a diluted solution of its product and communicating outside the cell with

any suitable source of supply. Each of the solution supply pipes 19 and 20 extends into its respective chamber not lower than the plane of the electrode and is provided at its lower end with a returned portion 21 to prevent disturbance when introducing the solutions. In each of the chambers 12 and 13 is suspended by any suitable means an electrode 22 and 23. The electrode 22 may be the anode and 23 the cathode of the cell, the anode 22 being preferably of lead or antimonial lead and the cathode 23 being of iron, steel or copper. The electrodes 22 and 23 may be connected to any suitable source of direct current for operating the cell.

The process or method of the present invention, set forth and described in connection with the above specifically described embodiment of the cell of this invention, is as follows:

In the following description, sodium chromate is used as an example but it is to be understood that other alkali salts of chromic acid or spent chromic acid solutions may be used. A sodium chromate solution having for example a specific gravity of 1.400 is introduced into the bottom chamber 14 through the feed pipe 16 to a surface level above the horizontal plane of the lower edge of the partition 11 and within the line of vision of the windows 15. When making pure chromic acid, acid radicals other than the chromate radical should be excluded from this solution. In the anode chamber 12 is floated a solution of chromic acid having a specific gravity materially less than the feed solution, for example 1.200. The solution supply pipe 19 is used to accomplish this floating in order to maintain the plane of separation between the anode solution and the feed solution. Similarly a caustic soda solution of approximately the same specific gravity is floated in the cathode chamber being introduced through the solution supply pipe 20.

The cell is now placed in operation by connecting the electrodes 22 and 23 to the proper poles of a source of direct current. Passage of current through the cell is necessary for the maintenance of the planes of separation.

Stoppage of current supply is followed by diffusion across and eventual disappearance of both planes of separation. In cells constructed of metal the supply voltage should not be high enough to cause passage of current through the walls of the cell itself. About 4.5 volts can usually be used with good results. Warming of the cell is beneficial in decreasing the resistance to be overcome. In operation of the cell, passage of current causes chromic acid together with a small quantity of basic ions to accumulate in the layer floating in the anode chamber and caustic soda together with unused sodium chromate in the floating layer in the cathode chamber. As electrolysis proceeds the plane of separation at the top of the feed solution may be observed through the windows 15 to move downward in both chambers. This movement is to be balanced by the introduction of additional feed solution through the feed pipe 16.

The solution in the anode chamber increases in volume and density during the operation of the cell. The increase in volume is kept down by means of the overflow pipe 17. The density of the solution is maintained at the standard by the introduction of water or diluted overflow through the solution supply pipe 19. The overflow consists in the chromic acid solution produced by the cell.

Likewise in the cathode chamber 13 the volume is controlled by the overflow pipe 18 and the requisite density maintained by dilution through the solution supply pipe 20. In this chamber the overflow solution consists of caustic soda and sodium chromate.

The chromic acid produced as described may not be satisfactory for use in a plating bath. Therefore I call this solution crude chromic acid and it may be refined by passing through a second cell identical with the first. This crude solution may be evaporated to reduce the volume and increase the specific gravity and then fed through the feed pipe 16 of the second cell. The floating layers are maintained as in operation of the first cell. The acid overflow from the tube 17 will now be found to be a nearly pure chromic acid solution suitable for use in chromium plating baths. Overflow from the cathode will be comparatively richer in sodium chromate and poorer in caustic soda than is the case of a cell fed with sodium chromate solution. This last overflow may be reworked in a cell using a sodium chromate feed to produce crude chromic acid and strong caustic solution.

To revive chromium plating baths that have become inoperative because of an accumulation of basic ions, the solution to be revived is fed through a feed pipe 16 and the cell operated as described for refining crude chromic acid.

Basic ions present in used plating solutions accumulate in the floating layer in the cathode chamber and any insoluble hydroxides or oxides will appear as a suspension or sludge. Working of the cell is benefited by maintaining a fairly rapid flow of solution through the solution supply tube 20 and out of overflow tube 18. In this way the insoluble hydroxides or oxides are washed out of the cell. Clear red solution will be discharged from the overflow tube 17 as distinguished from the usual dark color of used plating solutions. The product discharged from overflow tube 17 when made up to its original volume and CrO_3 content will be found to be restored in its plating efficiency.

In some methods of cell construction it is not practical to provide two observation windows as shown. Normal operation of such a cell can be obtained with the use of only one observation window if the following conditions are fulfilled. Overflow tubes 17 and 18 should be at the same level and the overflows from such tubes be maintained at the same or nearly the same specific gravity, variable for example between the limits 1.190 and 1.210. It will be evident that if the specific gravities of the solutions in each chamber are equal then the planes of separation will be at the same level. If the cell is constructed of transparent material windows are not necessary.

A convenient way of assuring separation as before described is to provide a flow of solution through each upper compartment such that the rise of specific gravity is not disturbing, for example the solution feed may have a specific gravity of 1.190 and the overflow a specific gravity of 1.200.

It must be noted that in operation of the cell the plane of separation of the feed solution and upper chamber solutions should be kept at a point below the level of the electrodes 22 and 23 and above the plane of the lower edge of the partition 11. The cell will not operate if the plane of separation rises above the electrodes or yet if the two upper chamber solutions intermix below the partition 11. The windows 15 are so positioned that when the planes of separation

are kept within their line of vision, those adverse conditions are prevented.

The above description refers to a simple cell having only two upper chambers. The cell may have two or more upper chambers, in each of which is suspended an electrode and if composed of more than two upper chambers, each alternate chamber will have suspended in it an anode and the intervening chamber will have suspended in it a cathode.

It will be obvious that various changes and modifications may be made in the apparatus and process above described without departing from the spirit of this invention and limited only by the scope of the following claims.

I claim:

1. An apparatus for an electrolytic process employing three solutions, one of greater density than the other two, consisting of a vessel provided with an inpermeable partition at its upper portion forming upper anode and cathode chambers open at their lower ends, a common chamber beneath said partition and communicating with said upper chambers, the densest solution in said common chamber and extending partly into the upper chambers, the light solutions floated on said dense solution one in each of the upper chambers and separated by the dense solution, means for observing the plane of separation between the solutions at a point above the lower edge of the partition, an electrode in each of the upper chambers, feeding means for the common chamber, and feeding and overflow means for each of the upper chambers.

2. An apparatus for the electrolytic separation of a solution of an alkali salt of chromic acid or of a solution containing chromic acid and basic constituents into two parts, said apparatus consisting of a vessel containing an anode chamber, a chromic acid solution of relatively low density in said anode chamber, a cathode chamber, an alkali solution of relatively low density in said cathode chamber, said chambers communicating at their lower ends to provide a common chamber, a feed solution of relatively high density in said common chamber and on which the anode and cathode solutions are floated, said feed solution filling the common chamber and extending partly into the electrode chambers to separate the anode and cathode solutions, an electrode in each anode and cathode chamber, and means for permitting visual observation of the plane of separation between the high density solution and a low density solution.

3. An electrolytic cell comprising a vessel provided with an impermeable partition at its upper part forming anode and cathode chambers, product solutions of relatively low density in said chambers, a common chamber in the vessel below the edge of said partition, a feed solution of relatively high density in said common chamber, said anode and cathode chambers opening at their lower ends into said common chamber, electrodes in the anode and cathode chambers, and means in the vessel for visual observation of the line of separation between the electrode chamber solutions and the common chamber solution.

4. An electrolytic cell comprising a vessel, a partition dividing the upper part of the vessel into two chambers communicating at their lower ends to provide a common chamber, a relatively dense feed solution in said common chamber, relatively light product solutions floated upon said feed solution in said upper chambers, a window in the vessel above the lower edge of the partition, electrodes suspended one in each of the said upper chambers, a feed solution pipe communicating with the common chamber, overflow pipes leading from the vessel near the upper edges of the upper chambers, and a solution feed pipe extending down into each of the upper chambers.

5. An electrolytic cell comprising a vessel, a partition dividing the upper part of the cell into chambers communicating at their lower ends to provide a common chamber, a relatively dense feed solution in said common chamber, relatively light product solutions floated upon said feed solution in said upper chambers, means for observing the plane of separation between a light and the dense solution at a point above the lower edge of the partition, electrodes suspended in each of said upper chambers, and means for maintaining the solutions at desired density and volume values.

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