

[54] **ELECTROLYTIC CELL FOR THE
MANUFACTURE OF OXYHALOGENS**

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204/284, 204/286

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[58] Field of Search **204/269, 268, 286,**
204/95, 284

[56] **References Cited**

UNITED STATES PATENTS

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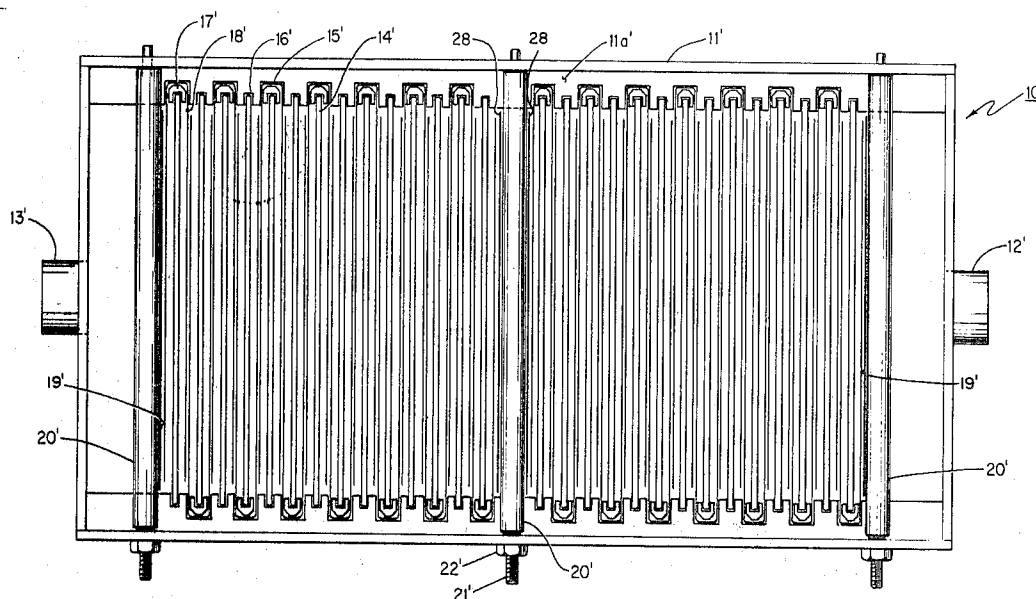
Attorney—Roy Davis et al.

[57]

ABSTRACT

An electrolytic cell for generation of sodium hypochlorite and other products comprising a cell chamber and plurality of vertical spaced parallel electrically insulating partitions dividing said chamber into individual compartments, monopolar electrodes in the first and last compartments of the group, bipolar electrodes constructed of two spaced parallel straight segments closed at one end intermediate the terminal electrodes, positioned with the straight elements of the electrodes on opposite sides of each partition and with the closed end of adjacent electrodes on opposed lateral edges of adjacent partitions, and means for applying a decomposition electrical potential between the terminal electrodes in the first and last compartments, respectively. Means are provided for creating electrolyte flow in a side-to-side or top-to-bottom manner in each compartment as the electrolyte flows through the chamber. Alkali metal hypochlorite and other chemical compounds are produced by electrolyzing alkali metal halide and other electrolyte solutions which form a gas at the electrode surfaces during electrolysis of the solutions.

14 Claims, 6 Drawing Figures



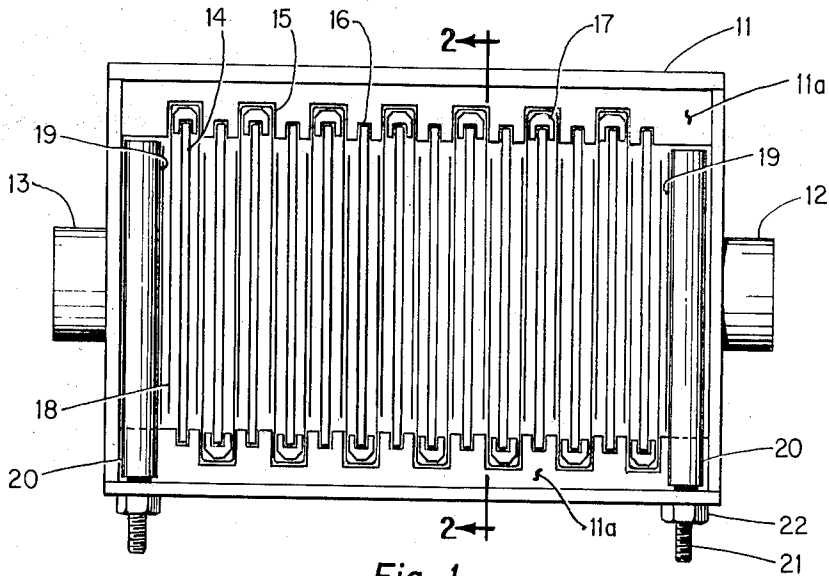


Fig. 1

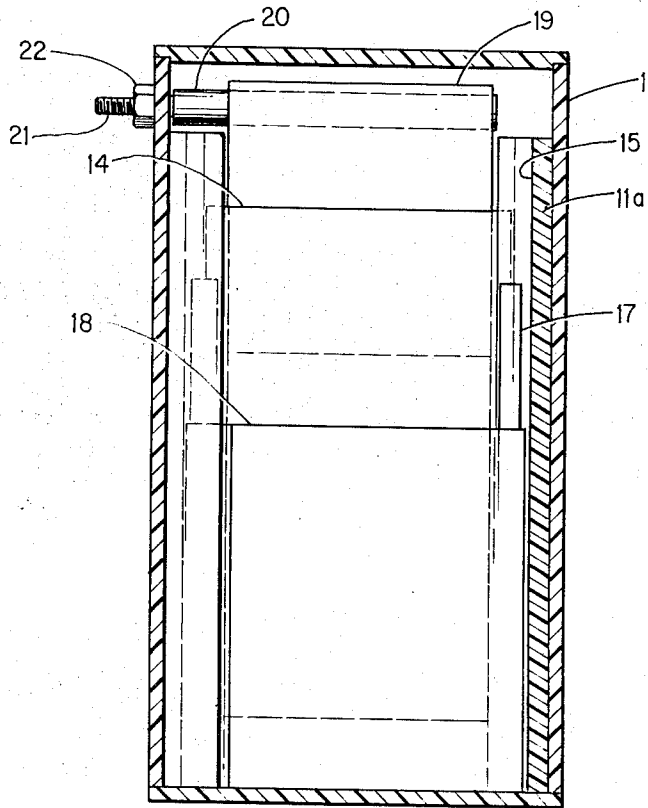


Fig. 2

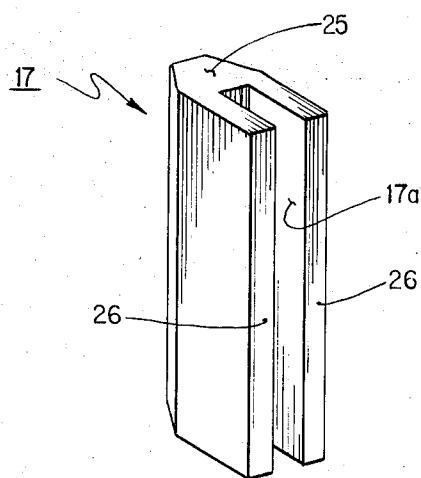


Fig. 3

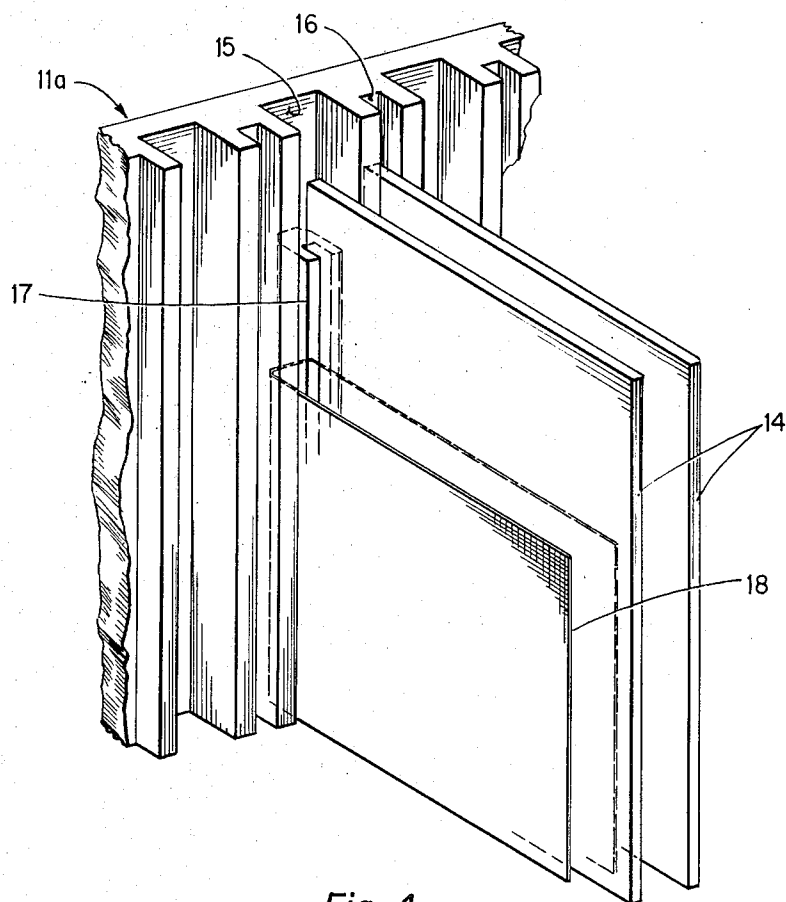


Fig. 4

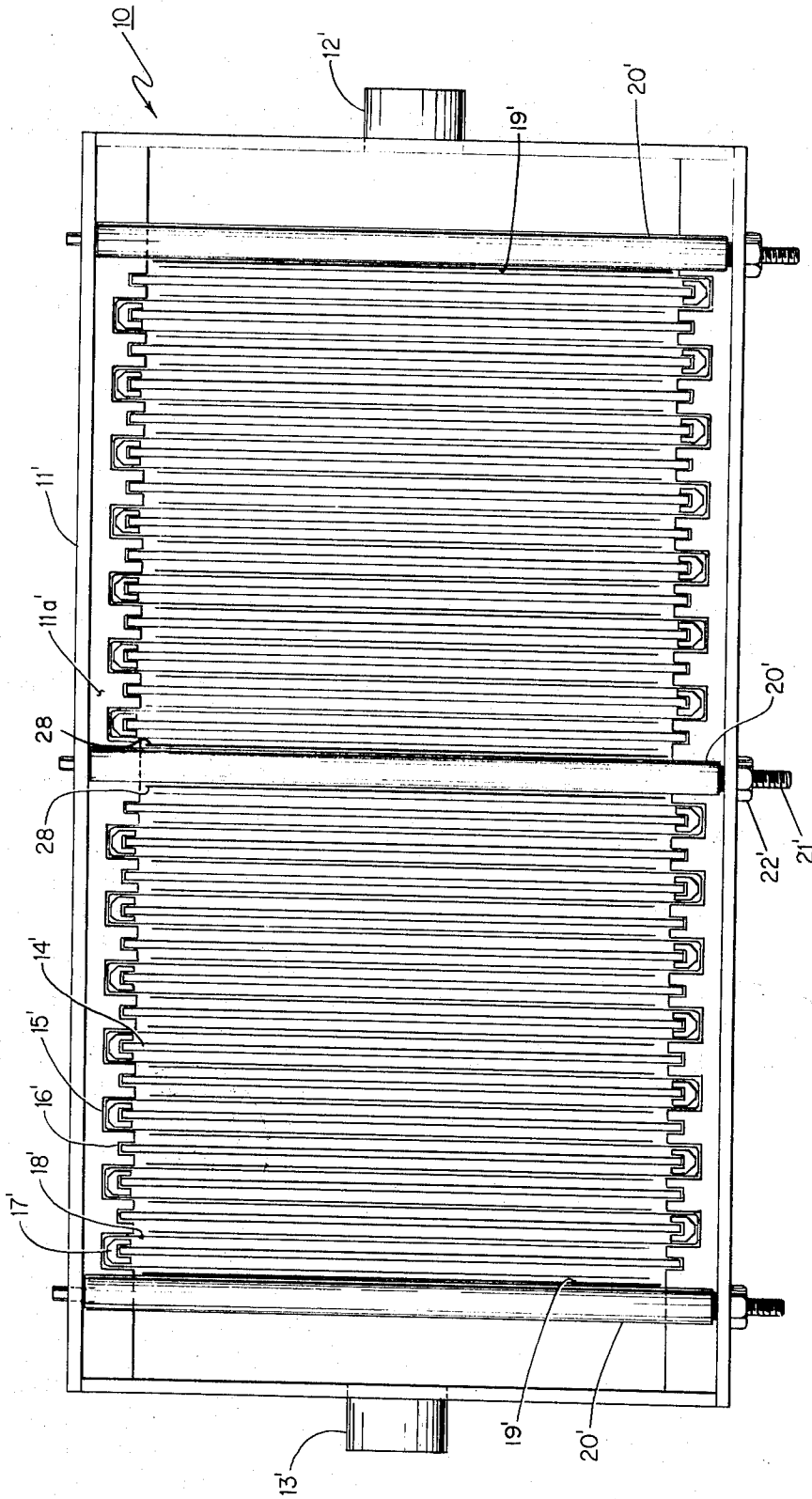


Fig. 5

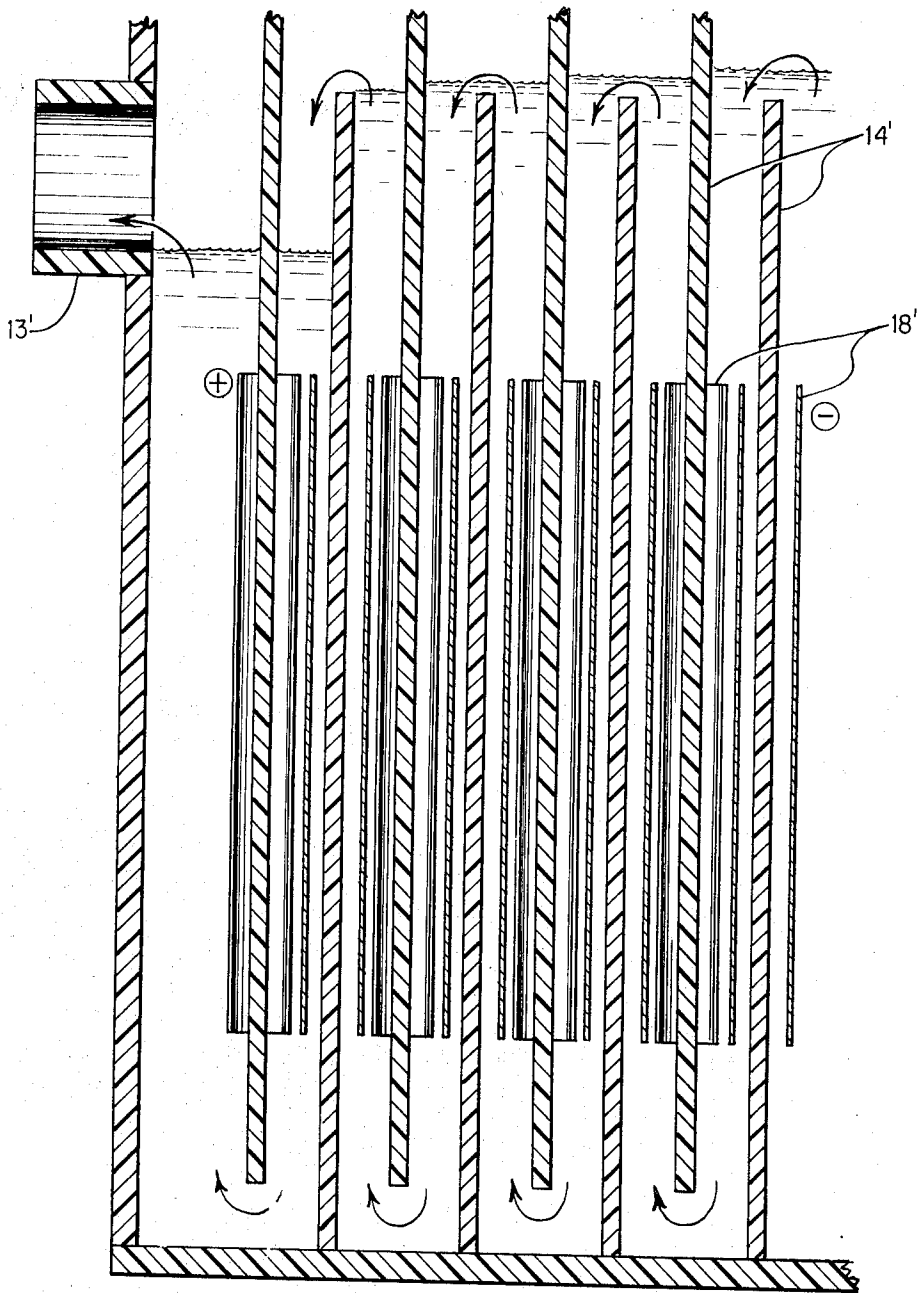


Fig. 6

ELECTROLYTIC CELL FOR THE MANUFACTURE OF OXYHALOGENS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to an electrolytic cell and process for manufacturing chemical compounds wherein gaseous products are formed at an electrode surface during electrolysis. In greater detail, this invention concerns a diaphragm-less electrolytic cell and the manufacture of sodium hypochlorite and other chemicals by operation of the cell.

2. Description of the Prior Art

Sodium hypochlorite has previously been produced directly in electrolytic cells. Such cells are generally so large that they are expensive to construct, require frequent maintenance and are transportable only by dismantling and reassembly. Additional disadvantages of such cells in production operations are the requirement of high consumption of saline solution for the quantity of available chlorine provided, production of undesirable by-products such as oxygen and sodium chlorate, inability to provide high hypochlorite concentration and poor electrical power efficiency. Because of the drawbacks and disadvantages of such prior cells sodium hypochlorite has been generally produced in recent years by the chemical reaction of sodium hydroxide and chlorine. Such production is carried out in large size plants, the hypochlorite product being widely distributed to various users. While such method of production is satisfactory the solution is corrosive and reasonably stable during storage only in dilute liquid form. Shipment of material in such dilute form presents an unavoidable expense since such solutions are objectionably unstable when shipped in more concentrated condition.

The above noted problems of the shipment and storage of sodium hypochlorite solutions and the disadvantages of large scale apparatus for producing such solutions indicate an obvious need for small scale portable electrolytic cells which are readily accessible and easily maintainable in such locations as laundries, hospitals, water treatment facilities and the like.

SUMMARY OF THE INVENTION

It is a principal object of this invention to provide an electrolytic cell of simple inexpensive design which is easily assembled, readily portable in assembled condition, and may be used to produce sodium hypochlorite and other compounds by electrolysis and in situ reaction of the products of electrolysis.

It is another object of this invention to provide such an electrolytic cell which may be easily dismantled, requires minimum maintenance and is especially suitable for small scale production of sodium hypochlorite or other compounds at locations such as laundries, small sewage treatment plants, hospitals and the like.

It is a further object of this invention to provide an electrolytic cell suitable for a small scale production of sodium hypochlorite and a method for the production of sodium hypochlorite wherein formation of objectionable by-products is minimized.

Other additional objects and advantages of this invention will be gained from the following specification, appended claims and by reference to the drawings wherein like numerals and characters represent the same or similar parts and in which:

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a top plan view of an electrolytic cell in accordance with this invention.

FIG. 2 is a cross-section taken on the line 2—2 of FIG. 1.

FIG. 3 is an elevational view of one of the elements used for positioning the electrodes in the cell in spaced relation to the partitions.

FIG. 4 is a perspective view illustrating one of the U-shaped electrodes positioned on one of the electrically insulating partitions by a spacing and positioning element, the assembly being removed from the cell chamber of FIG. 1 for clarity of illustration.

FIG. 5 is a top plan view of another embodiment of the cell of this invention wherein two monopolar electrodes of the same electrical charge are positioned in the cell at the midpoint of the electrode units and separate the cell and the electrode units into two regions of equal numbers of electrodes and partitions.

FIG. 6 is a somewhat diagrammatic view of another embodiment of this invention wherein alternating partitions are spaced from the bottom wall of the cell chamber and extend above the top edges of adjacent partitions.

Referring to the drawings, a cell chamber shown generally at 10 has side walls 11 and inlet and outlet means 12 and 13 respectively. Electrically insulating partitions 14 are mounted in the cell chamber in vertical channels 15 and 16. U-shaped electrodes 18 having two spaced parallel straight segments joined together at one end are positioned with each segment spaced from opposite faces of each partition. Wall sections 11a are mounted within the cell chamber and are provided with U-shaped vertical channels 15 and 16 for receiving the partitions 14. The sections 11a may be separately mounted for ease of insertion and removal from the cell chamber or they may be integral with the cell walls 11. Vertical channel 15 is adapted to receive spacer 17 and channel 16 is adapted to receive one end of partitions 14. Spacer 17 generally of U-shaped configuration is constructed with a vertical slotted portion 17a which is adapted to receive the other end of partitions 14. The partitions 14 are thus held in parallel spaced relation by insertion of one lateral edge of the partition in the longitudinally spaced vertical channels 16 and the other end snugly fitted in the longitudinal spaced parallel vertical slotted portion 17a of spacer 17. In addition to being adapted to engage one lateral edge of partitions 14, spacers 17 are also adapted to snugly engage bipolar electrodes 18 at the inner surface of the closed end of said electrodes between the outer periphery of the spacers 17 and the interior surfaces of the vertical channels 15. The spacers are adapted to position the bipolar electrodes so that each parallel segment is held in spaced parallel relation to opposite sides of partitions 14. Wall sections 11a are constructed so that the vertical channels 15 and 16 alternate in end-to-end and opposed side-by-side position in the longitudinal direction of the cell chamber. By this arrangement one segment of the U-shaped electrodes is closely spaced in parallel relationship to a segment of an adjacent U-shaped electrode, both segments being in face-to-face relationship between two electrically insulating partitions. The opposed end walls of the chamber are provided with inlet 12 and outlet 13, respectively, which may be constructed of any suitable material and are

adapted for connection to other conduits. Terminal monopolar electrodes 19 are provided within each terminal compartment of the cell and are connected in any suitable manner, e.g., by welding to busbars 20 which are preferably constructed of titanium but may be of any suitable material resistant to the cell environment and capable of conducting electric current. The busbar may be made of any suitable configuration and conveniently may be cylindrical. A direct current power source (not shown) is connected to the terminal electrodes 19 through busbar 20 by connecting the power source leads to brass stud 21 which is threadably connected to the busbar 20. Brass nuts 22 serve to connect the lead from the power source in secure position to stud 21. Spacer 17 is shown in more detail in FIG. 3 where 25 illustrates the generally U-shaped configuration of the spacer, in which vertical U-shaped channel 17a extends between the two straight wall segments 26 of the spacer. The spacer is adapted to fit within vertical U-shaped channel 15 in such manner that it tightly engages one end of partition 14 in channel 17a and also snugly engages the U-shaped electrode firmly between its outer periphery and the inner surface of channel 15.

In another embodiment of the invention shown in FIG. 5 a similar design to that shown in FIGS. 1-3 is utilized. FIG. 5 varies from FIG. 1 in that monopolar electrodes are positioned to divide the bipolar electrodes and partitions into two equal sections. The monopolar electrodes 28 may be the same type as the terminal electrodes 19 and serve to supply or withdraw electric current from the bipolar electrodes. Thus the surface of each bipolar segment positioned adjacent the central monopolar electrodes 28 in closely spaced face-to-face parallel relation will be opposite to the electric charge of the monopolar electrodes and will vary dependent upon electric current being supplied to or withdrawn from the monopolar electrodes 28. The central monopolar electrodes 28, of which there are at least two, are generally of the same polarity. Busbars 20' are connected to the monopolar electrodes, with the central electrodes' busbar connected to one lead from the power source and the terminal electrodes' busbars connected to the other lead. Brass nuts 22' serve to connect the lead from the power source in secure position to stud 21', which is threadably connected to the busbar 20'. In the embodiments of the invention shown in FIGS. 1-5 the electrolyte solution enters the cell chamber and flows through the cell by passing through the small space between the outer perimeter of spacer 17 and vertical channel 15 at the lower portion of the partition and around the edges of partition 14 which extend above the upper edges of the electrodes 18. In this manner the electrolyte traverses the cell from the inlet to the outlet in a tortuous path by circulating through the channels 15 around the periphery of spacers 17 and the joined or yoked portion of electrodes 18 and the edges of partitions 14 extending vertically in spaced relation from the side walls of the chamber above the electrodes and spacers. The path is tortuous because of the alternating arrangement of the parallel longitudinally spaced vertical channels 15 in which the spacers are arranged, said channels being alternately spaced in end-to-end and opposed relation in a longitudinal direction of the side walls of the cell with channels 16. Consequently, in each unit compartment of the multi-unit cell the electrolyte solution must flow around the partition to enter and exit from each unit, the electro-

lyte entering the unit in the space between the lateral edge of the partition and the side wall of one side of the cell and leaving the unit compartment via passage around the space at the opposed edge of the next adjacent partition. In another embodiment of the invention shown in FIG. 6 the spaced parallel electrically insulating partitions are arranged so that alternating partitions are spaced from the bottom wall of the chamber and extend vertically a sufficient distance to prevent solution from flowing over the upper edge into the adjacent succeeding compartment. The partitions alternating with the partitions spaced from the bottom wall are arranged with their lower edges in electrolyte separating engagement with the bottom wall of the chamber and their upper edges below the upper edges of said spaced partitions. By this arrangement the electrolyte solution is caused to flow underneath the partitions spaced from the bottom of the chamber wall and over the upper edges of the adjacent partitions. Such arrangement provides for an undulating over and under flow of the electrolyte solution throughout the cell which provides thorough mixing of the products of the electrolysis and in this manner provides improved current efficiency and uniformity of the composition of the electrolyte solution. A distinct advantage of this type of flow is the prevention of formation of quiescent pockets of the electrolyte solution in any area of the cell and particularly in an area adjacent or surrounding the electrodes.

The cell chamber cover, electrically insulating partitions and other structural elements may be constructed of any material which is not adversely affected by the environment of use and is usually made from plastic material such as polyvinyl chloride, polyvinylidene chloride, chlorinated polyvinyl chloride, acrylates, tetrafluoroethylene, polyethylene and the like. A preferred material of construction of the cell chamber and similar elements of the cell is polyvinylchloride.

The bipolar electrodes comprise a dimensionally stable anode segment and a cathode segment which are of equal dimensions. Each segment comprises one-half of the dimension of the bipolar electrode. The configuration of the bipolar electrode may vary but is generally in the form of a flat sheet and preferably is foraminous. The bipolar electrodes are constructed of two straight parallel laterally spaced segments closed at one end. The straight segments are adapted for location on opposite sides of the lateral surfaces of insulating partitions. Where the closed end is adapted for arrangement in a vertical channel of the chamber side wall it will vary in shape with the configuration of the channel. The closed end may be bonded to an edge of an insulating partition by any suitable means such as adhesives or by softening the partition edge and embedding the electrode in the softened material. In this embodiment the vertical wall channels are deleted and the partition is positioned adjacent the side walls in liquid-tight engagement. The electrolyte solution in this embodiment does not flow through the cell via the spaces between the edges of the partitions and the side walls but rather over the top edge and under the bottom edge of adjacent partitions.

The bipolar electrode may be so constructed that the metal is unitary or integral in construction or the segments of the electrode may be joined, preferably at the midpoint of the closed end in any suitable manner such as welding and the like. The dimensionally stable anode segments of the bipolar electrode comprise an electri-

cally conductive substrate with a surface coating thereon of an electrically conductive and electrocatalytically active material. The active coating must be on at least a portion of the surface of the substrate and may be a solid solution of at least one precious metal oxide and at least one valve metal oxide. The electrically conductive substrate may be any metal which is not adversely effected by the cell environment during use and which also has the capability, if a breakdown in the surface coating develops, of preventing detrimental reaction of the electrolyte with the substrate. Generally, the substrate is selected from the valve metals including titanium, tantalum, niobium and zirconium. Expanded mesh titanium sheet is preferred at the present time for the substrate.

In the solid solutions an interstitial atom of a valve metal oxide crystal lattice host structure is replaced with an atom of precious metal. This solid solution structure distinguishes the coating from physical mixtures of the oxides since pure valve metal oxides are, in fact, insulators. Such substitutional solid solutions are electrically conductive, catalytic and electrocatalytic.

In the above-mentioned solid solution host structure the valve metals include titanium, tantalum, niobium and zirconium while the implanted precious metals encompass platinum, ruthenium, palladium, iridium, rhodium and osmium. Titanium dioxide-ruthenium dioxide solid solutions are preferred at this time. The molar ratio of valve metal to precious metal varies between 0.2-5:1, approximately 2:1 being presently preferred.

If desired, the solid solutions may be modified by the addition of other components which may either enter into the solid solution itself or admix with same to attain a desired result. For instance, it is known that a portion of the precious metal oxide, up to 50 percent, may be replaced with tin dioxide without substantial detrimental effect on the overvoltage. Likewise, the defect solid solution may be modified by the addition of cobalt compounds, particularly cobalt titanate. Solid solutions modified by the addition of cobalt titanate, which serves to stabilize and extend the life of the solid solution, are described more completely in co-pending application Ser. No. 104,703 filed Jan. 7, 1971 now U.S. Pat. No. 3,726,995. Other partial substitutions and additions are encompassed. Another type of dimensionally stable anode coating which may be used with good results in the practice of this invention consists of mixtures of chemically and mechanically inert organic polymers and solid solutions of valve metal and precious metal oxides as at least a partial coating on the electrically conductive substrate. Particularly useful materials in such anode coatings are the above-described solid solutions in admixture with fluorocarbon polymers such as polyvinyl fluoride, polyvinylidene fluoride and the like coated on at least part of the surface of an electrically conductive substrate consisting of the above-described valve metals and other suitable metals. Such anode coatings and preparation thereof are disclosed and more completely described in co-pending application Ser. No. 111,752 filed Feb. 1, 1971.

One other type of dimensionally stable anode capable of satisfactory use in this invention consists of a valve metal substrate bearing a coating of precious metals or precious metal alloys, particularly platinum alloys thereof on at least part of its surface.

The above-mentioned preferred solid solution coatings are described in more detail in British Pat. No. 1,195,871.

The cathode segment may be any metal capable of sustaining the corrosive cell conditions and a useful metal is generally selected from the group consisting of stainless steel, nickel, titanium, steel, lead and platinum. In some cases the cathode segments may be coated with the solid solutions above-described for coating the dimensionally stable anode segments. The cathode segments may be flat sheets and are preferably flat, foraminous sheets. At the present time expanded mesh titanium sheets are preferred.

When the bipolar electrode metal substrate is integral, the anode segment is always at least partially coated with one of the above-described solid solutions and the cathode segment is either at least partially coated with a solid solution or uncoated substrate material. Where the bipolar electrode is constructed by joining the two segments, the cathode metal may be of the same substrate metal as the anode or a different metal and either coated or uncoated with the same or different solid solution coating than the anode. The dimensions of the cell chamber partitions and the bipolar electrodes will vary in accordance with the amount of product desired and the use of the cell but generally the cell will be of dimensions suitable for ease of assembly and portability. The dimensions of the bipolar electrodes are selected in accordance with the quantity of product desired and the optimum electric current efficiency for such production.

It will be noted from the above description that an anode and a cathode segment of adjacent bipolar electrodes are positioned between two adjacent electrically insulating partitions in closely spaced parallel substantially face-to-face relation. The gap or space between the adjacent anode and cathode segments is generally from about 0.03 inches to about 0.08 inches and preferably is maintained at about 0.04 inches. Because of such closely spaced positioning of the anode-cathode segments electrically non-conductive separators are generally interwoven through or positioned within the openings of foraminous electrodes to prevent electrical contact of the segments. When flat or cylindrical elements are used as separators they are generally interwoven through alternate openings on the outer edges of the lateral surfaces of the electrodes but may also be interwoven through other openings in the foraminous electrodes. The electrically non-conductive separators should be constructed of materials inert to the cell environment and may have any suitable geometric configuration. Generally, the separators are polyvinylidene chloride, polyvinyl chloride, chlorinated polyvinylfluoride, polyvinylfluoride, tetrafluoroethylene and the like and may be of solid or hollow, cylindrical, flat or other suitable configuration. Other types of spacers capable of satisfactory use are electrically nonconductive strips provided with projections adapted to be tightly engaged within the electrode openings and button-type members such as semi-spherical elements arranged in opposite sides of the electrode openings and joined by an engaging member such as a stem extending through the electrode openings. The separators are preferably arranged to prevent electrical contact for shorting between the electrodes and, at the same time, provide maximum flow of the electrolyte solution through the openings in the electrodes.

In operation of the cell electrolyte solution is introduced into the cell through the inlet, a decomposition potential is applied across the terminal electrodes to decompose the electrolyte, the electrolyzed solution is withdrawn through the outlet means and the desired compounds obtained by the decomposition are recovered. The parameters of the electrolytic process such as temperature, pH, electrolyte solution concentration, amperage, voltage and the flow rate of the solution are adjusted in accordance with the quantity and type of product desired. The cell may be operated continuously or batchwise.

The following examples of the production of sodium hypochlorite presented in Table I below are intended for purposes of illustration only and are not to be considered limitative of the invention in any manner. In Examples 1 to 4 of Table I a brine solution containing 28 grams of sodium chloride per liter of solution was continuously introduced to an electrolytic cell of the type illustrated in FIGS. 1 through 4. The temperature of the inlet brine solution was 19° C. and the current density was 1.0 ampere/in.². The solution was continuously electrolyzed within the parameters included in the table and the sodium hypochlorite product continuously withdrawn. A similar procedure was followed for Examples 5 to 8 except the inlet brine temperature was 14° C. and the current density was 0.95 amp/in.². The data of the examples show that sodium hypochlorite can be manufactured by the cells and process of this invention in varying concentrations and with good current and power efficiency.

TABLE I

Example No.	Flow rate ml/min.	Outlet temp. °C.	Temp. in- crease °C.	Sodium hypochlo- rite as available Cl ₂ -gpl	Sodium hypochlo- rite produc- tion current efficiency, percent	Direct current KWH/lbs. of available chlorine
1.....	410	31	12	9.22	57	1.74
2.....	650	26	7	7.87	77	1.30
3.....	1110	24	5	5.22	88	1.16
4.....	1560	22	3	3.72	88	1.55
5.....	440	25	11	9.2	65	1.55
6.....	690	23	9	7.1	77	1.33
7.....	980	21.5	7.5	5.65	87	1.18
8.....	1320	19.0	5	4.08	90	1.16

Although the present invention has been described with detailed reference to specific embodiments thereof, it is not intended to be so limited since modifications and alterations therein may be made which are within the complete intended scope of this invention as defined by the appended claims.

I claim:

1. A multi-compartment electrolytic cell comprising:
 - a. a cell chamber having side, bottom and end walls and provided with inlet and outlet means for electrolyte solution;
 - b. a plurality of electrically insulating vertical, substantially parallel, spaced partitions dividing said chamber into compartments;
 - c. monopolar electrodes mounted in each of the terminal compartments;
 - d. a like plurality of foraminous bipolar electrodes having a closed end and a pair of parallel opposed straight segments extending from the end, the segments adapted to be positioned in spaced parallel relation on opposed faces of each partition, the closed ends of adjacent electrodes being mounted

on opposed lateral exterior edges of adjacent partitions;

- d. means for mounting the closed end of each electrode on the opposed lateral exterior edge of adjacent partitions; and

- f. the lateral electrode-mounted edge of each partition being spaced from a sidewall of said chamber a sufficient distance to permit passage of the electrolyte solution from one cell compartment to the adjacent cell compartment.

2. The cell as claimed in claim 1 in which the bipolar electrodes are U-shaped.

3. The cell as claimed in claim 1 in which the bipolar electrodes are spaced in equal proportions (dimensions) on each side of each partition.

4. The cell as claimed in claim 1 in which at least one segment of said electrode has an electrically conductive coating on at least a portion of its surface.

5. The cell as claimed in claim 1 in which the entire electrode is coated with an electrically conductive coating on at least a portion of the surface of each segment.

6. The cell as claimed in claim 1 in which a cell compartment is disposed centrally of the cell chamber and intermediate an equal number of cell compartments, and at least two monopolar electrodes of like charge are mounted in the intermediate compartment.

7. The cell as claimed in claim 1 in which the electrodes are bonded to the lateral edges of the partitions by adhesive connection means.

8. A multi-compartment electrolytic cell comprising:

- a. a cell chamber having side, bottom and end walls and provided with inlet and outlet means for electrolyte solution;

- b. a plurality of parallel vertical channels spaced equidistant longitudinally on each side wall of the cell in directly opposed position;

- c. spacers adapted to be slidably mounted within one group of channels alternating end to end and transversely of the side walls of the cell chamber with a second group of channels, said spacers having a slotted portion;

- d. vertical partitions slidably mounted within said channels, one lateral edge of each partition positioned within the slotted portion of each spacer, and the opposite edge positioned in the other channel;

- e. substantially U-shaped foraminous bipolar electrodes having a closed end and a pair of opposed straight segments extending from the end, the segments adapted to be positioned in spaced parallel

relation on opposite faces of each partition;

- f. the closed end of each adjacent electrode being mounted between the rear surface of the spacer and the side wall surface adjacent the spacer; and
- g. the rear surface of each spacer being spaced from the sidewall of said chamber a sufficient distance to permit passage of the electrolyte solution from one cell compartment to adjacent cell compartments.

9. The cell as claimed in claim 8 in which the U-shaped electrodes are spaced in equal proportions (dimensions) on each side of each partition.

10. The cell as claimed in claim 8 in which at least one segment of said electrode has an electrically conductive coating on at least a portion of its surface.

11. The cell as claimed in claim 8 in which the entire electrode is coated with an electrically conductive coating on at least a portion of the surface of each segment.

12. The cell as claimed in claim 8 in which a cell compartment is disposed centrally of the cell chamber and intermediate an equal number of cell compartments and at least two monopolar electrodes of like charge are mounted in the intermediate compartment.

13. A multi-compartment electrolytic cell comprising:

- a. a cell chamber having side, bottom, and end walls and provided with inlet and outlet means for electrolyte solution;
- b. a plurality of electrically insulating, vertical, substantially parallel, spaced partitions dividing said chamber into compartments and substantially traversing said chamber from side wall to side wall, alternate partitions being spaced from the cell bottom to permit electrolyte flow;
- c. monopolar electrodes mounted in each of the ter-

minal compartments;

- d. a like plurality of foraminous bipolar electrodes having a closed end and a pair of parallel opposed straight segments extending from the end, the segments adapted to be positioned in spaced parallel relation on opposed faces of each partition, the closed end of each electrode being mounted between a lateral partition edge and a side wall.

14. A multi-compartment electrolytic cell comprising:

- a. a cell chamber having side, bottom, and end walls and provided with inlet and outlet means for electrolyte solution;
- b. a plurality of parallel vertical channels spaced equidistant longitudinally on each side wall of the cell in directly opposed position;
- c. spacers adapted to be slidably mounted within one group of channels alternating end to end and transversely of the side walls with a second group of channels, said spacers having a slotted portion;
- d. vertical partitions slidably mounted within said channels, one lateral edge of each partition positioned within the slotted portion of each spacer, the opposite edge positioned in the other channel, alternate partitions being spaced from the cell bottom to permit electrolyte flow;
- e. substantially U-shaped foraminous bipolar electrodes having a closed end and a pair of opposed straight segments, the segments adapted to be positioned in spaced parallel relation on opposite faces of each partition; and
- f. the closed end of each electrode being held between the rear surface of the spacer and the side wall.

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