June 25, 1968 R. M. WISEMAN 3,390,072

DIAPHRAGM ELECTROLYTIC ALKALI HALOGEN CELL

Filed May 6, 1965 3 Sheets-Sheet 1

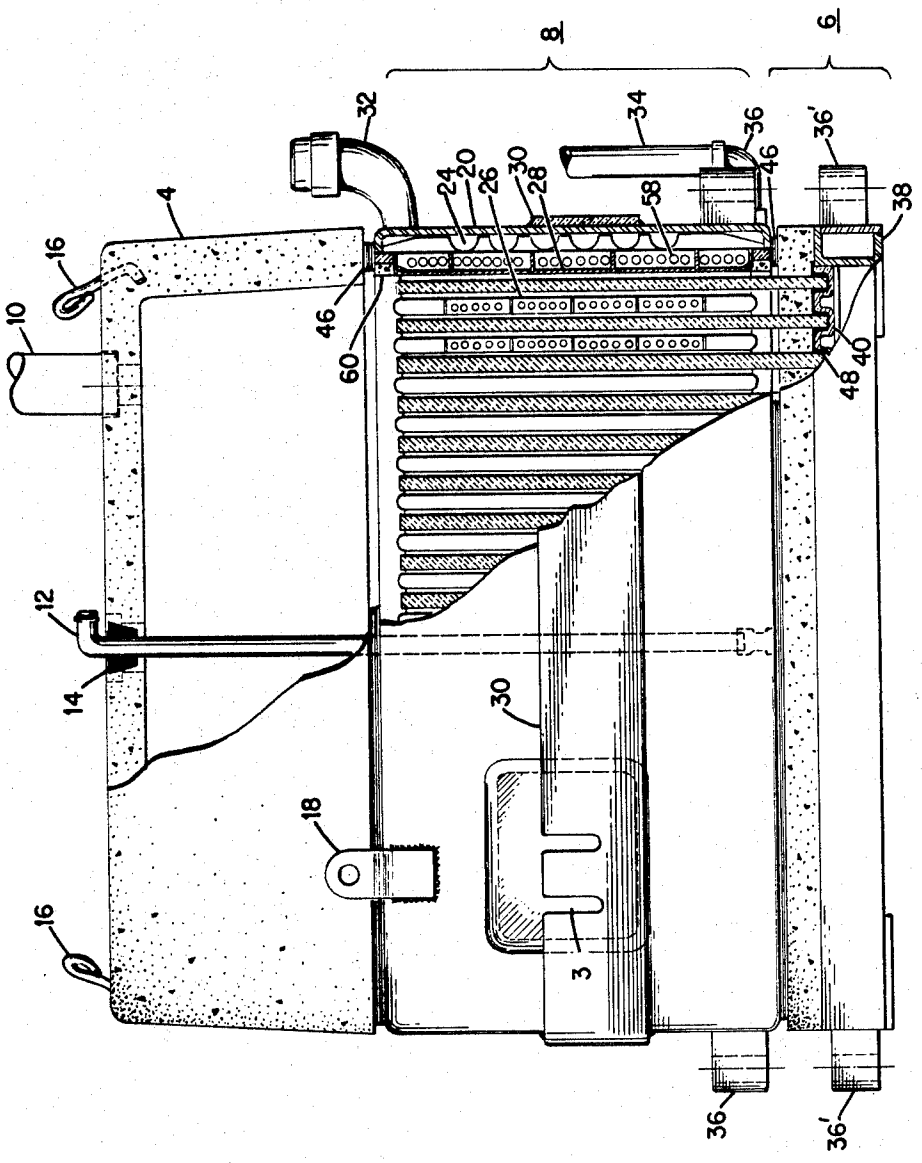


Fig. 1

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Filed May 6, 1965 3 Sheets-Sheet 2

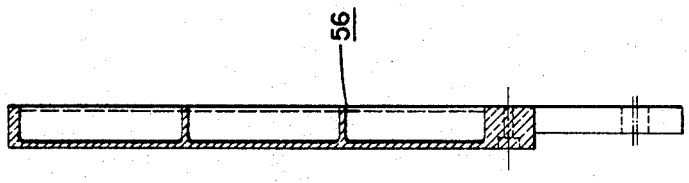


Fig. 4

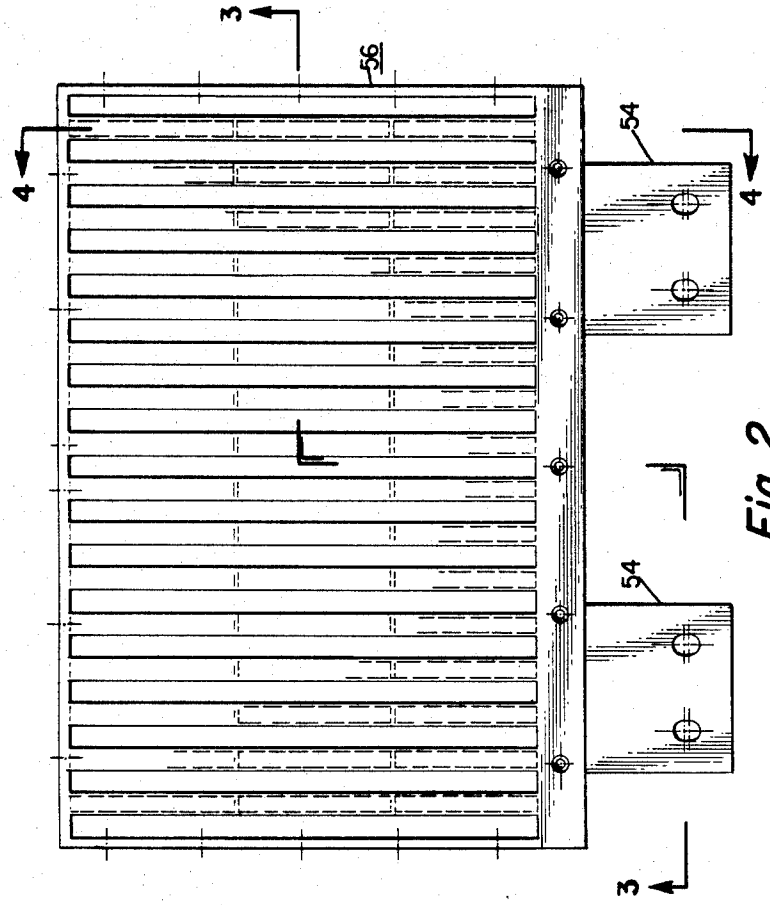


Fig. 2

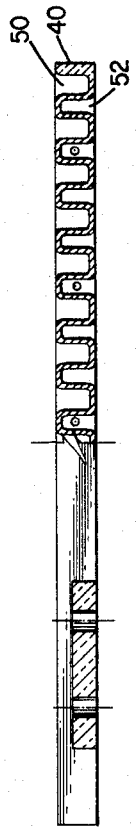


Fig. 3

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DIAPHRAGM ELECTROLYTIC ALKALI HALOGEN CELL

Filed May 6, 1965 3 Sheets-Sheet 3

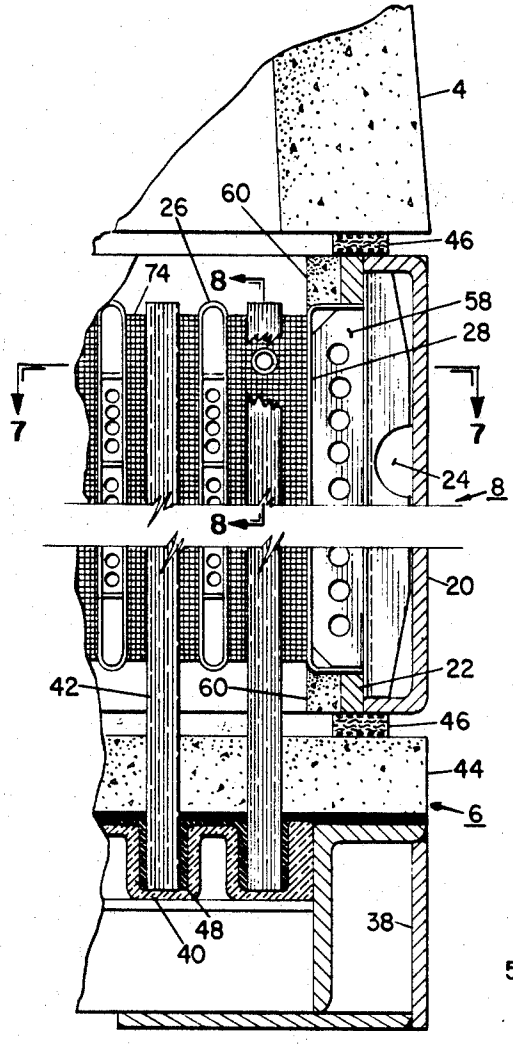


Fig. 5

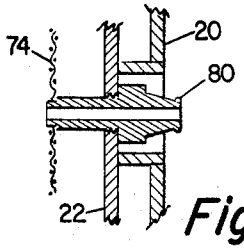


Fig. 8

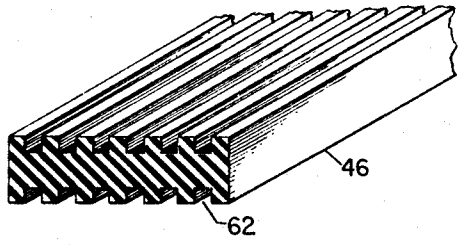


Fig. 6

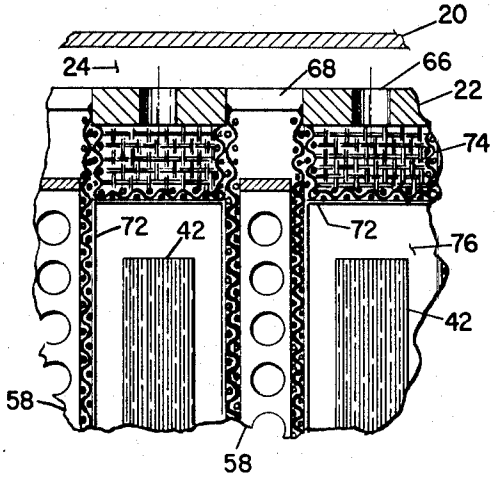


Fig. 7

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United States Patent Office 3,390,072 Patented June 25, 1968

3,390,072

DIAPHRAGM ELECTROLYTIC ALKALI HALOGEN CELL

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Filed May 6, 1965, Ser. No. 453,616
5 Claims. (Cl. 204—266)

This invention relates to high amperage electrolytic cells and more specifically pertains to an improved chlor-alkali cell of the diaphragm type and of compact design capable of operation at a capacity of 30,000 amperes or higher.

Diaphragm electrolytic alkali halogen cells are widely used in the preparation of caustic soda, chlorine and hydrogen via electrolysis of sodium or potassium chloride brines. Sodium chloride brines, for example, are continuously fed into the cells which employ foraminous metallic cathodes with fluid permeable diaphragms which essentially cover said cathodes and thus allow the brine, hereafter referred to as the electrolyte, to permeate the diaphragm and flow into the cathode chamber from the anode chamber, thereby producing H_2 gas and caustic soda in the cathode chamber and chlorine gas in the anode chamber.

In chlor-alkali cells of the above-described type and more particularly as described in U.S. Patent No. 2,987,463, of which the novel features of this invention, hereafter described, constitute improvements therein, the goal in industry today is increased circuit amperage which, in turn, introduces problems in cell operation caused primarily by corresponding rapid increases in cell temperature. When increased operating currents are employed, the corresponding high temperature directly associates itself with rapid deterioration of various cell elements, e.g., the anode blades which, in turn, necessitates frequent and costly component changes as well as seriously reducing the "on stream" time of the cell.

An additional outstanding aspect of disadvantages which have come to light in prior art cells when operated at increased currents resides in current loss encountered in certain locations of the cell, especially in the vicinity of the anode support area and betwen the anode blades and surrounding cathode member. This current loss is caused principally by three factors, (1) poor electrical contact between the anode support means which previous to the instant invention employed excessive amounts of soft lead metal for conductive and support purposes, (2) failure to attain or maintain precise positional proximity and conformation between the anode blades and associated cathode chambers and (3) inactive current conductive areas of the anode surrounding cathode-chambers caused primarily by previous diaphragm cell design wherein no or irregularly spaced circular cathode end screens were employed.

An additional disadvantage to those of undesired electrolyte leakage, current loss and rapid temperature gain caused by high amperage operating conditions, is the accumulation and fouling of instrumentalities by organic sealing putty material heretofore required with conventional gasket members. Such conventional gasket members requiring excessive amounts of putty are, by necessity, exposed to the oxidizing action of dissolved chlorine in the electrolyte. As a result of this action, significant quantities of organic degradation products form which contaminate the chlorine, contribute materially to plugging diaphragm pores and to forming undesirable quantities of sludge in the bottom of the cell.

Thus, in accordance with the present invention, novel cell construction is provided which eliminates many of the above problems faced in high amperage diaphragm cell operation. By virtue of the instant invention, advantageous results are obtained by employing the following features either taken singly or in combination:

(1) An improved diaphragm electrolytic alkali halogen cell adapted to be operated at a capacity of 30,000 amperes or higher;

(2) An improved anode support and current conducting grid member for such cells;

(3) An improved cathode end screen construction for such cells which allows maximum and uniform current density and avoids disadvantages of stray currents and current losses; and

(4) An improved gasket member for such cells which permits cell operation with little or no component fouling sealing putty accumulation while also preventing hazardous as well as corrosive cell leakage.

The invention will be more fully understood by reference to the attached drawings, in which:

FIGURE 1 is a side elevational section of the electrolytic diaphragm cell of the instant invention partially cut away to show, in section, the arrangement of anodes and cathodes and the preferred mode of affixing the anodes to the corrugated pan or base as well as means for introducing the electrolyte and removal means for products of the cell;

FIGURE 2 is a top plan view of the pan or base to show the electrically conductive corrugated grid pan support means adapted to be used for the anodes of the invention:

FIGURE 3 is a cross-section view of the pan or base taken along line 3—3 of FIGURE 2;

FIGURE 4 is a side elevated ivew of a portion of the apparatus of FIGURE 2 taken along line 4—4, showing the cross-members in each channel to guide and support the anode instrumentalities;

FIGURE 5 is an enlarged fragmentary perspective view of a portion of the apparatus of FIGURE 1;

FIGURE 6 is an enlarged cross-section view of the gasket member of the instant invention;

FIGURE 7 is an enlarged fragmentary view, partially in section, showing the new flat or box type end screens of the cathode chamber embodying the instant invention; and

FIGURE 8 is an enlarged fragmentary sectional view along line 8—8 of FIGURE 5 showing a convenient manometer connection (manometer not shown) for determining the anolyte level within the anode compartment.

Referring more particularly to FIGURES 1 and 2, FIGURE 1 illustrates a partially cutaway side view of the cell which includes a gas collecting and cooling cover member 4, bottom anode support and aligning portion indicated generally at 6, and intermediate cathode carrying section indicated generally at 8.

Gas collecting and cooling cover 4 is constructed of a halogen resistant, non-conductive material such as concrete or rubber-lined steel; however, concrete is generally preferred, typically consisting of the materials set forth in col. 4 of U.S. Patent No. 2,987,463. Provided on said cover 4 is a gas exit member or conduit 10 for escaping halogen gas. A centrally located conduit 12 provided with a gasket 14 provides a convenient electrolyte inlet directly to the cell bottom, therefore, providing electrolyte dispersion on impact. Gasket 14 prevents undesired gas escape around the conduit and may be formed of any chemically inert gas-impermeable material such as rubber or the like. Conveniently positioned lifting eyes 16 are provided securely embedded in the cover 4. Proper alignment of the cover with the intermediate cathode carrying section 8 is maintained by members 18 of conventional female and male insert design.

Intermediate cathode carrying section 8 comprises outer side walls 20 and inner walls 22 (best shown in FIGURE 5) which form a peripheral chamber 24 for

collection of evolving cathode gas (in the case of the instant invention, hydrogen) formed within cathode tubes 26 and half cathode members 28. Side walls 20 are constructed of electrical conductive material so as to facilitate supplying current through conductive inner walls 22 to cathode tubes 26 and half cathode members 28 from an encircling buss 30 which is adapted to be connected to any convenient source of electrical potential.

Side walls 20 and 22 are constructed of metal sheets, preferably of steel sheets since openings therein, as hereinafter described, are easily made and welds can be used to join them in good electrical connection to cathode tubes 26 and half members 28. A well placed multiplicity of electrically conductive metal straps 58 (see also FIGURE 7) furnish support and stability to the cathode tube assembly. These straps can be welded to the tubes to assure a good electrical connection. Cathode tubes 26 and half-cathode members 28 are advantageously connected electrically at both their ends to the electrically conductive side walls 20. This can be done by welding, if both tubes and side walls are steel. It is then possible to supply current at each end of the tubes by connecting side walls 20 to a source of potential through buss member 30.

Conduit 32 permits withdrawal of hydrogen present in peripheral chamber 24 and conduit 34 permits convenient exit for the catholyte liquor, i.e., the alkali metal hydroxide containing unelectrolyzed salt.

Elements 36 and 36' are fixed locators providing a novel alignment feature between intermediate cathode carrying section 8 and cell bottom 6 of this invention.

In referring more particularly to FIGURE 5 (which is exaggerated for clarity), several important aspects of the invention will be readily apparent. Anodes 42, each unit of which is composed of several sections are mounted in each channel of grid support pan 40 which, in turn, is detachably mounted to the open structure or conventional base 38. The anode grid pan is composed of electrically conductive copper and the novel corrugated feature is completely cast in one piece, a feature that affords better electrical conductivity, better electrical distribution and completely eliminates the expensive and relatively inefficient tie bars previously relied on for connection to a source of positive potential. The anodes 42 can be maintained in proper spaced and electrically conductive relationship by conductive bonding layer 48, suitably of lead. Anode member 42, although as such forming no part of the instant invention can be formed of any good electrically conductive material such as graphite, magnetite, or magnetite coated material. Corrugated grid pan member 40 can be conviently formed using electrolytic grade copper which normally has a resistivity factor of about 1.724×10^{-6} ohm./cm.^{3}.

Open structure steel base 38 may be formed of any convenient structural steel such as welded angle iron, I beam material; or, if desired, the conventional cast grid support means may be used, for example, the grid support as heretofore described in U.S. Patent No. 2,987,463. Base slab 44, previously described, may be cast of concrete. Alignment means 36 and 36' are preferably formed of welded steel plates of varying thicknesses and having accurate openings in each for proper positioning of intermediate cathode carrying section 8 in relation to cell bottom portion 6. Channeled gasket members 46 and 46' may be of rubber, plastic-coated silicon rubber or any good resilient substitute preferably not subject to electrolytic action or corrosive effects of residual dissolved chlorine.

Various views of the corrugated anode grid pan support of the invention are illustrated in FIGURES 2 to 4 where is illustrated anode supporting channels 50, spaces 52, which, in association with structural steel base, allows cooling air to circulate freely around the base portion of anodes 42 (not shown in FIGURE 3) and anode spacing and grid pan cross-members 56 for each anode supporting channel to support and provide alignment for each of three anodes adapted to be contained therein. Another important function of cross-member 56 resides in affording tight electrical connection to the anodes thus giving good electrical conductivity and cutting the I^2R loss to a minimum. The grid pan is conveniently attached to a source of proper electrical potential by anode lugs 54.

Turning now to FIGURE 6, there is shown an exaggerated cross sectional view illustrating an additional novel portion of the invention, namely the gasket member 46 which embodies a multiplicity of parallel channels 62 the construction of which affords another important aspect of the invention. Gasket members 46 are adapted to be used between the cell cover 4 and cathode carrying section 8 as well as between this section and cell bottom 6. Such gaskets, when in proper position with the weight of the cell components 4 and 8 resting thereon, provide good suction engagement therewith to prevent corrosive and hazardous electrolyte leakage.

The form and supporting structure of cathode tubes 26 are perhaps best shown in FIGURE 7, which is a top plan view of a portion of the apparatus of FIGURE 5. The tubes extend from inner sidewall to inner sidewall of the cathode-carrying section of the cell; however, since the structure for each end is the same, only one end has been shown. Slots 66 and 68 communicate directly with peripheral chamber 24 providing convenient outlet for the catholyte liquor and cathode gas from the cathode tubes and ultimately out of the cell proper through conduits 32 and 34, previously mentioned. The cathode tube may be constructed from perforated metal sheets by forming into a tube and welding the seam or alternatively heavy gage wire cloth is suitable with wire cloth being illustrated and preferred. The method of fitting to the inner side walls 22 provides easy access to the cathode assembly which can be detached from the remainder of the cell and removed for cleaning or repair.

Fluid permeable diaphragm 72 completely separates the cathode from the anodes and is constructed of halogen-resistant material such as particulate asbestos used in conventional cells of the diaphragm type and can be deposited in situ upon the outer surfaces of the wire mesh cathode. Other type diaphragm materials are equally readily adaptable as will be apparent from those skilled in the art. The cathode tubes are horizontally disposed across the cell and are spaced above concrete base member 44 to permit complete circulation of the cell liquor therebeneath. End members 74, construction of which constitutes an important aspect of this invention, are overlaid with the aforementioned diaphragm material thereby completing the enclosure of the sides of the anodes by cathodic material and forming anode compartments 76. As previously mentioned, the cathode tubes are preferably constructed of heavy gage steel wire cloth and are adapted to be fitted to inner side walls 22 in good electrical connection and may be serviced to the sidewall by spot welding. When fully assembled, the tubes are from about ¾" to 1⅛" in total thickness.

The flat or box-type end screens 74 are separately mounted and are adapted to be fitted between the corresponding cathode tubes either by a frictional engagement or weldment. Of course, to insure optimum current conductivity and positioning, spot welding of the end screen members is preferred. Additionally to insure a good fitting and current conducting relationship between the cathode tubes and conductive side walls, the end screen members are preferably first constructed outside the cell in a V or other similar dihedral manner somewhat less in width (approximately 2") between the sides then when finally assembled in the cell. This permits stretching the dihedral into a flat box type arrangement upon assembly; thus an excellent mounting as well as precise positional conformation between the ends of the anode blades are obtained.

Electrical connection with other cells in series can be provided as shown in FIGURES 1 and 2. Connection is made to the anode lug 54 by a convenient buss (not shown) and attached to the corresponding adjacent cell to cathode recessed lug 31. This recess reduces the length of the electrical buss connection and saves space required for the cells without effecting individual cell operation.

Thus, from the preceding detailed description, it will be apparent to one skilled in the art that the improvements embodied in diaphragm cells of this type constitute a timely advancement in cell design.

More particularly, the improved channeled gasket means not only stands up to the advanced temperatures characteristic of cells operated under high amperages but also its improved design acts to prevent cell leakage without the use of costly, element fouling, sealing putty normally used in the design of diaphragm cells. Moreover, the channels or pockets afford a "tortuous path" between the cell interior and exterior further to guard against leakage. Should, however, cell putty be desired as an added insurance feature against cell leakage, the channels incorporated in the gasket act as a perfect containing and retaining means thereby preventing direct contact between the putty and the oxidative effects of the liquor. As a consequence, the putty has no chance, should it deteriorate, to continually foul cell instrumentalities thus causing frequent and expensive shutdown time.

Furthermore, the improved corrugated grid pan, construction of which is discussed in detail above, permits substantial saving in time and in improved cell operation. In prior art cells where conventional grid construction is employed, costly and excessive amounts of current robbing soft, low melting, conductive metal such as lead are used between the more efficient copper current conducting leads and the anode buss member. Additionally in cell assembly or construction, each row of anode members had to be aligned and set in the metal individually to ensure proper performance in the cell. This vastly costly and time-consuming operation had to be repeated each time the anode members were replaced, which incidentally, is considerably more often in high amperage cells than in cells operating at lower amperages. In the grid pan design of the present cell wherein supporting and electrically conductive corrugations are used, the anode blades can be rapidly positioned in the entire grid pan, either by frictional engagement or by allowing slight tolerance if lead is to be used. In either case, fast and correct alignment between the anodes and cathode surfaces is assured. Should, however, the latter method, e.g., utilizing soft lead be preferred, the present design permits pouring the entire grid at one time, an additional expedient that permits a tremendous overall savings notwithstanding a considerable saving in the amount of lead always used. For example, only about one-quarter of as much lead is necessary as compared to the amount previously employed by conventional grid support means.

The novel cathode and screen construction of the instant invention also constitutes a vast improvement in overall cell efficiency. Other than virtually eliminating stray currents and obtaining additional active product producing surfaces on the anode and cathode members, such design reduces high concentration of currents in localized areas which contribute materially to anode wear. Again, the cells of the prior art, if any end screen is used at all, employ a semi-circular continuation of the cathode tubes which permits varying distances between the anode and cathode thereby promoting currents of varying intensity on the active surfaces of the anode-cathode members. Uneven distribution of current with especially high concentration in a particular location, sometimes referred to as "hot spots," contributes materially to anode wear and ultimate failure of the anode blades notwithstanding inefficient current densities on the anode and cathode surfaces. Hence, the flat box type end screen employed in the instant invention allows even current distribution over virtually the entire anode surface thus improving considerably the merits of efficient current density and advantages of even component wear aside from making a good sealed section for the diaphragm members.

In the operation of a cell of this invention an alkali metal chloride, for example, sodium chloride, brine stream of desired concentration is fed to the cell through opening 12 in the cover 4. The brine level is brought to a point somewhat above the upper surface of the anodes, suitably more than one inch and preferably about one to four inches above the anodes, and an electric current passed through the cell by means of the electric connections, some of which are not shown. It has been found that the anolyte level is preferably maintained well above the tops of the anode elements of the cell to aid in ensuring uniform chloride ion distribution in the anolyte compartments.

Although the precise manner in which uniform chloride ion concentration is obtained is not completely understood, there is evidence that the feed brine entering through the inlet 12 and through the tube downwardly to the bottom of the cell is dispersed, upon impact with the base of the cell, assisted by natural circulation within the anode compartment, uniformly in the region of the lower portions of the anodes and cathodes. The hottest part of the cell of the present invention is at the center, unlike cells having an open center aisle, where there are no anodes. The brine is heated in the central portion of the cell, during electrolysis.

Chlorine gas forms at the surfaces of the anodes 42, rises along the anodes and is exhausted from the cell through the vent 10. The rising chlorine as well as the heat generated during electrolysis, particularly at and near the center of the cell, causes upward movement of the anolyte in the electrolytically active central regions of the anolyte compartments and this is accompanied by a compensating circulation of the anolyte downwardly at the edges of the cell in the more open end spaces of the compartments. The result of the placement of brine introduction and this natural convection—and chlorine lift—aided circulation is to give substantially uniform chloride ion concentration throughout the anolyte compartments. This circulation is greatly assisted by the placement of the cathode tubes above the base of the cell.

Concurrently with the evolution of chlorine from the anodes, anolyte, percolates through the porous cathode diaphragm and the metallic cathode members, as well as the box type end members of the cathodes, where it forms alkali metal hydroxide and hydrogen, into the catholyte compartments. The alkali metal hydroxide solution and hydrogen escape from the catholyte compartments into the peripheral chamber 24 by way of the slots 66 and other openings 68. The catholyte solution ultimately leaves the peripheral chamber of the cell through the take-off conduit 34 here shown at the side of the cell. However, the catholyte take-off conduit may conveniently be located otherwise than as shown, without detrimental effect. Hydrogen and traces of other gases present in the peripheral chamber may be suitably removed as through the hydrogen outlet 32.

The above-described design offers considerable advantages over prior cells. The most important is the efficient operation at a large current, of the order amperes and higher, which permits a considerably greater production. The cathode design permits an exceedingly low hydrogen content in the chlorine cell gas. The improved copper corrugated grid super-imposed upon the base member gives a much better electrical connection in the proximity of the anode, reducing to a minimum, or none at all, the distance current must travel in order to reach the anode. The anode and cathode arrangement allows cell repair and assembly to be accomplished readily and efficiently outside the cell room, because each of these components is readily removed. It also is possible to

achieve a considerable stability of operation under varying load conditions simply by adjusting the feed of brine to the cell and withdrawal of cell liquor from the cell to fit the need.

The following data is typical of performance of the cell of the invention at amperages of 27,000, 30,000 and 33,000 with a sodium chloride brine, and is compared with a typical 20,000 ampere cell of more conventional design:

TABLE I.—PERFORMANCE DATA

Cell Amperes	Typical 20,000 Ampere Diaphragm Cell	30,000 Ampere Diaphragm Cell		
		27,000	30,000	33,000
Current Efficiency Avg. Cell Voltage (Inc. Buss.)	96.5	96.5	96.5	96.5
	[1] 3.78	3.66	[1] 3.82	[1] 3.94
Power, KWHDC/Ton Cl_2	2,690	2,630	2,720	2,800
Graphite, lbs./Ton Cl_2	7.5	7.5	7.5	7.5
Avg. Anode Life, Days	230	255	230	210
Avg. Diaphragm Life, Days	115	115	115	110
Cell Liquor Temp.,° F.	196	193	196	199
Percent NaOH in Cell Liquor	[1] 10.5	[1] 10.5	[1] 10.5	[1] 10.5
$NaClO_3$/1,000 NaOH in C.L.	0.54	0.54	0.54	0.54
Chlorine Production Tons/Day	0.675	0.91	0.11	1.11
NaOH Production Tons/Day	0.760	1.02	1.4	1.25
Anolyte Temp.,° F.		200	203	206

[1] The cell can be operated so as to produce cell liquor with an 11.2% NaOH content. This results in a cell voltage increase of 0.02 volt.

Brine cell feed specification

NaCl	g./l.	318–325
NaOH	g./l.	.006–0.12
Na_2SO_4	g./l.	0–5.0
Na_2CO_3	g./l.	0.32–0.72
pH, minimum		10.2
Calcium	p.p.m.	<10

The cell liquor characteristics are as follows:

Cell liquor

		Percent
NaOH	g./l.	133–140
$NaClO_3$	g./l.	0.07
Na_2SO_4	g./l.	0–7
Na_2CO_3	g./l.	0.6
NaCl	g./l.	210
NaCl decomposed percent		48.1

The above data show that the 30,000 ampere diaphragm cell of the invention does not require an appreciably higher cell voltage than the 20,000 ampere cell. In fact, at 27,000 amperes the cell voltage needed is markedly less than that needed by the 20,000 ampere cell. At 30,000 amperes the voltages are comparable, and so also are the average lives of the anodes and of the diaphragms. A higher cell liquor temperature is not necessary. The percentage of sodium hydroxide in the cell liquor is the same, and the chlorine and sodium hydroxide production are increased. As would be expected from this, the current efficiencies are substantially the same.

The purity of the chlorine obtainable from the cell of the invention is evident from the following:

TABLE II

Chlorine cell gas:	Mole percent
Cl_2	97.6
CO_2	0.9
O_2	0.7
H_2	<0.1
N_2	0.7

The cell structure of the invention is readily adapted for use at any available amperages to suit the available electrical equipment, merely by adjusting the number of electrode pairs. It can be used at amperages well below 30,000 amperes, when so modified, although of course it is most economically operated at 30,000 amperes and above. Amperages of 35,000 to 40,000 amperes are not excessive.

The cell is useful for the electrolysis of alkali metal chlorides in general, including not only sodium chloride, as indicated above, but also potassium chloride, lithium chloride, rubidium chloride, and caesium chloride. These collectively are encompassed by the term "brine" as used in the specification and claims.

It is to be understood that although the invention has been described with specific reference to particular embodiments thereof, it is not to be so limited, since changes and alterations therein may be made which are within the full intended scope of this invention as defined by the appended claims.

What is claimed is:

1. In an electrolytic cell the improvements comprising:
(A) a base member having associated therewith an electrically conductive one piece grid support member which comprises alternately facing corrugation adapted to support said plurality of anodes in good positional proximity between alternately associated cathode members anodes;
(B) a plurality of hollow, tubular, foraminous electrically conductive metal cathodes interposed between said anodes in an alternating array and which comprise;
(C) a flat, separately mounted electrically conductive member adapted to be fitted between two corresponding cathode members constituting part of said array.

2. An electrolytic cell in accordance with claim 1 in which the grid member is separately cast and is electrically conductive over its entire surface.

3. Apparatus as in claim 1 wherein said anodes are supported by a frictional engagement with said corrugations.

4. Apparatus as in claim 1 and wherein said grid member is of pan type construction and is adapted to contain about ¼ the amount of low melting, fusible current conducting support metal normally used.

5. In an electrolytic cell for the electrolysis of sodium chloride brine the improvements comprising:
(A) an open grid base member having associated therewith an electrically conductive one piece pan type grid support member comprising alternately facing corrugations adapted to guide and support a plurality of vertically disposed anodes,
(B) a plurality of hollow, tubular foraminous electrically conductive metal cathodes interposed between said anodes,
(C) said cathode members constituting a horizontally disposed alternating array and extending from an inner side wall to inner side wall of an inner liquid-containing chamber,
(D) said tubular cathodes being electrically connected to at least one of said side walls,
(E) a flat, separately mounted electrically conductive cathode active screen member between and in electrical connection with, said cathode members adapted to provide an active current conductive and thereby product forming surface,
(F) said end screen member and said cathodes acting to completely surround said anodes and thereby defining separate catholyte and anolyte compartments,

(G) a fluid-permeable diaphragm associated with the surface of said cathode tubes and said cathode end screen.

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D. R. JORDAN, *Assistant Examiner.*

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,390,072 June 25, 1968

Russell M. Wiseman

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 31, "ivew" should read -- view --. Column 3, line 51, "conviently" should read -- conveniently --. Column 8, line 34, after "members" cancel "anodes".

Signed and sealed this 18th day of November 1969.

(SEAL)
Attest:

Edward M. Fletcher, Jr.
Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents