Oct. 29, 1968 R. D. BARNARD ETAL 3,408,281

VERTICAL MERCURY CATHODE ELECTROLYTIC CELL WITH DIAPHRAGM ENCLOSED PERFORATED CATHODE SUPPORT

Filed July 15, 1965 4 Sheets-Sheet 1

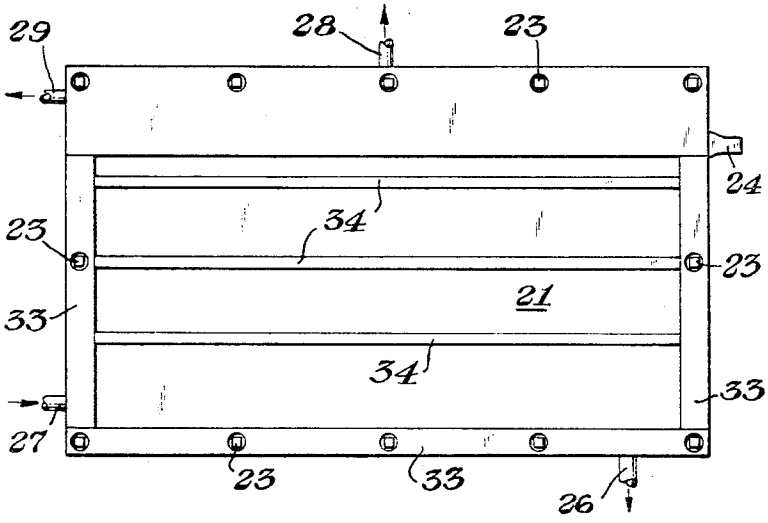


Fig. 1

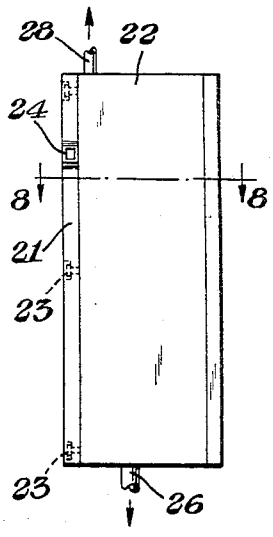


Fig. 2

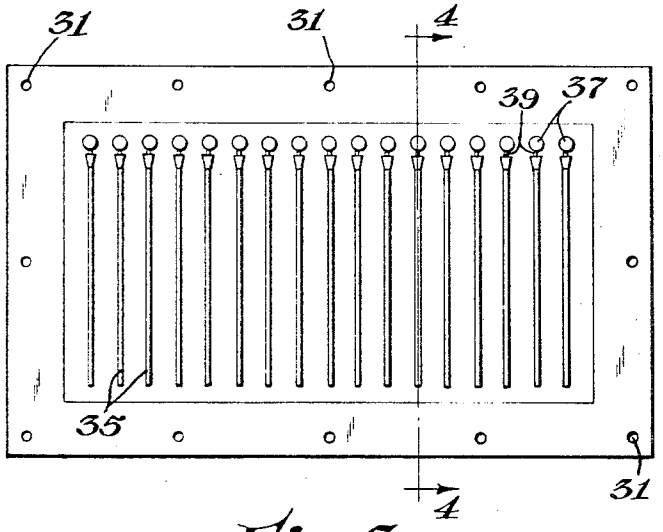


Fig. 3

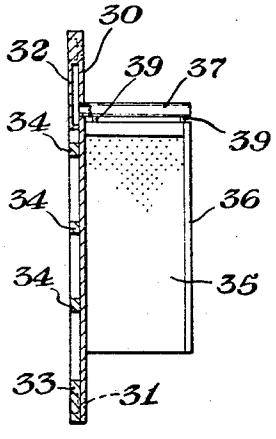


Fig. 4

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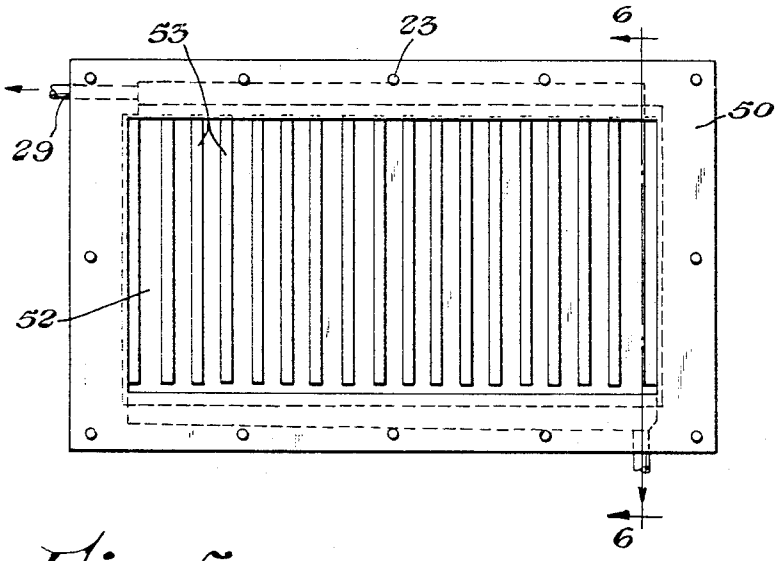


Fig. 5

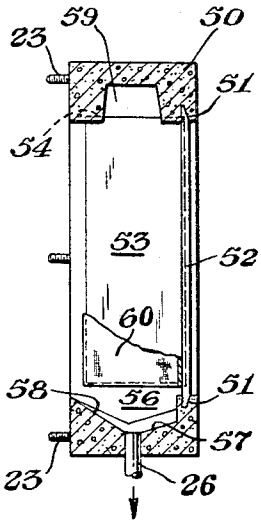


Fig. 6

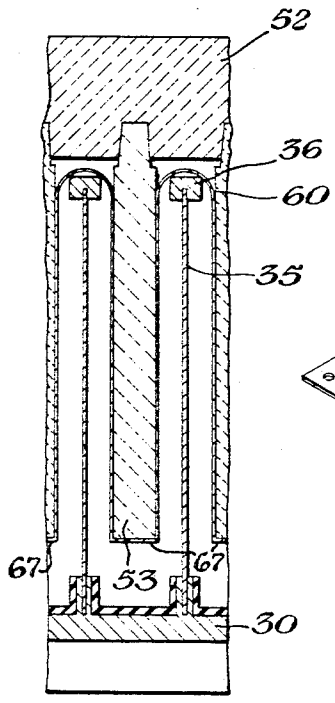


Fig. 8

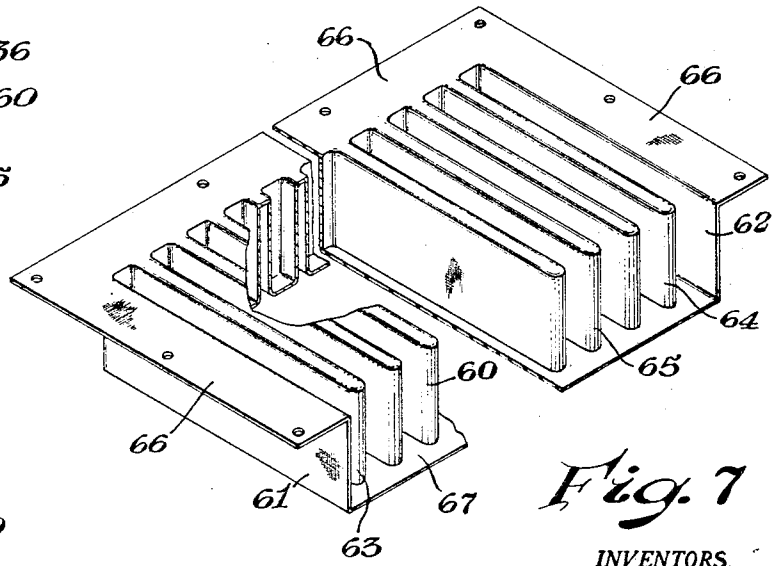


Fig. 7

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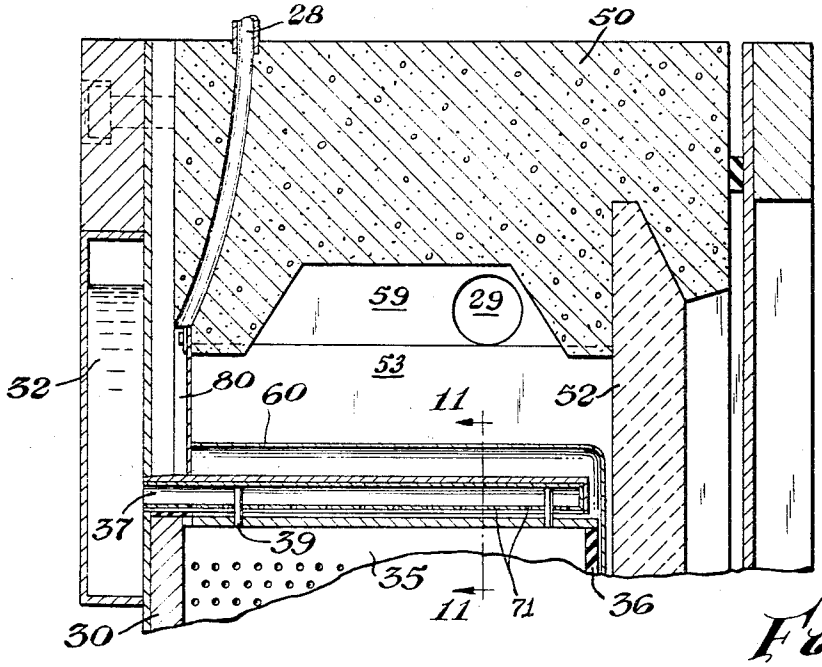


Fig. 9

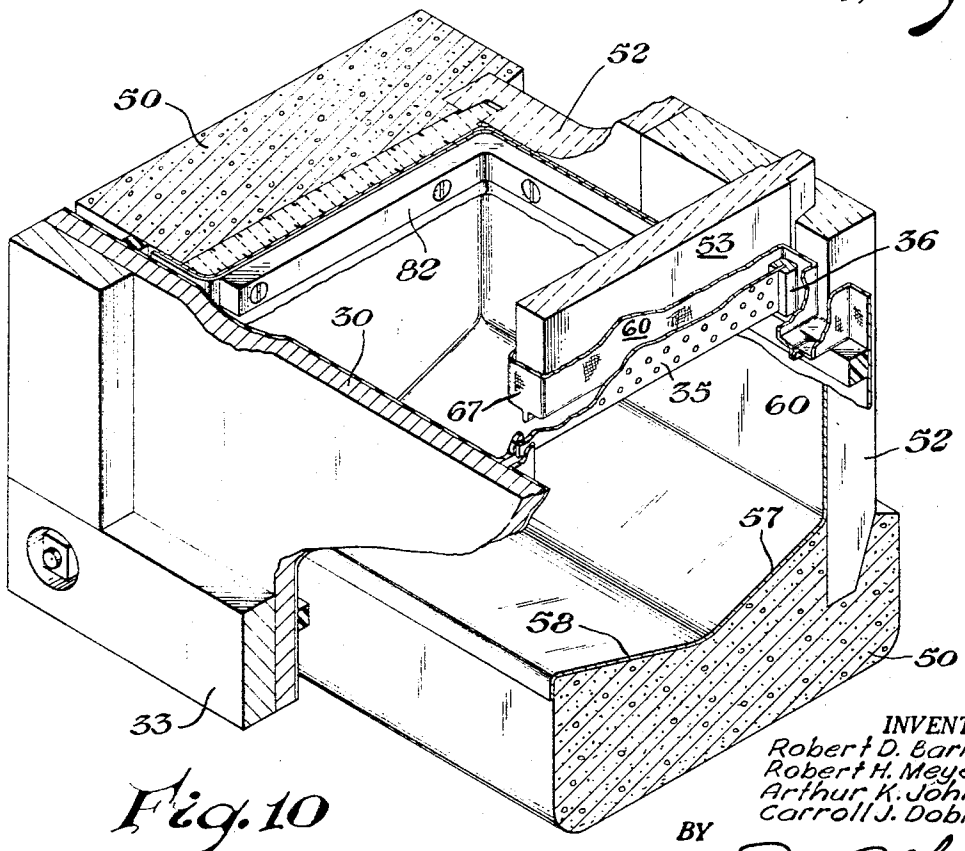


Fig. 10

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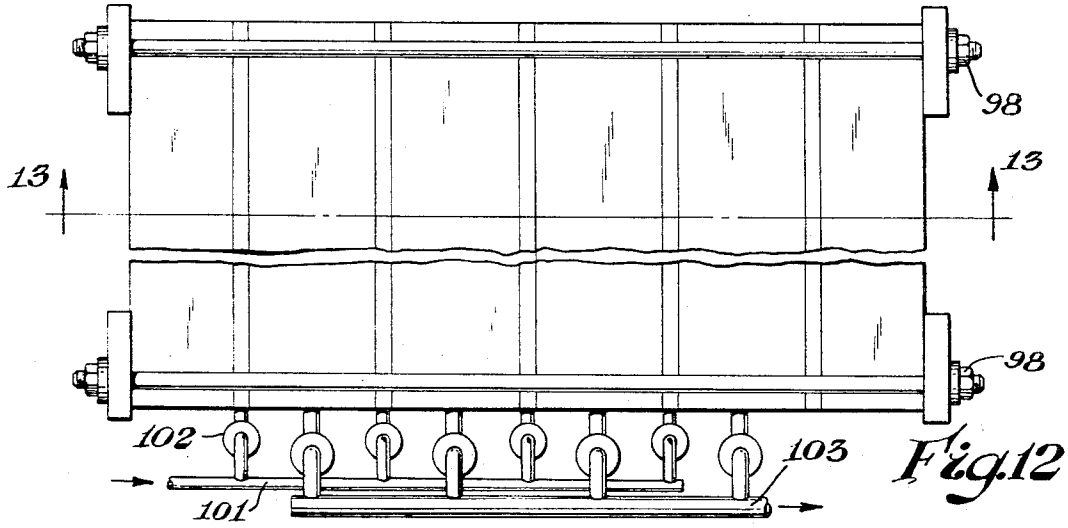


Fig. 12

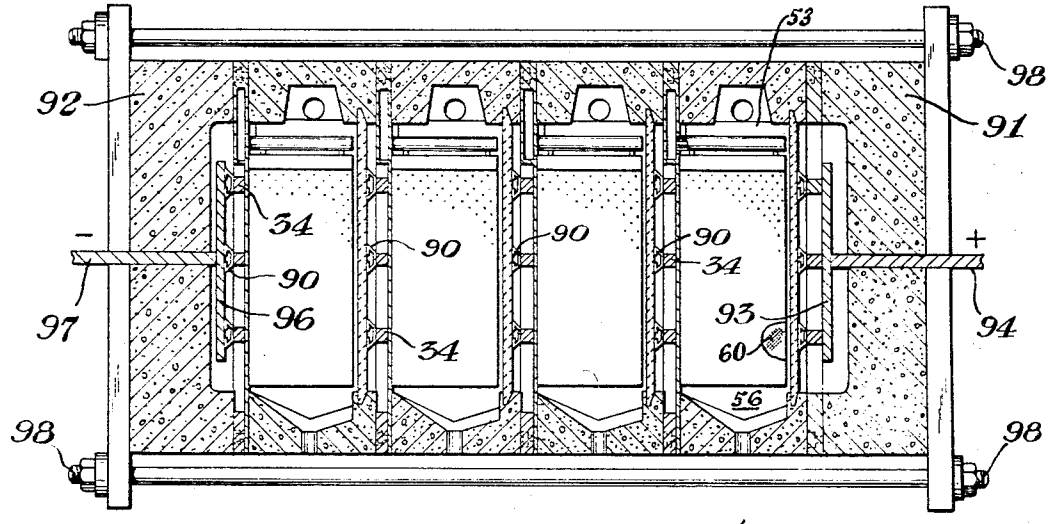


Fig. 13

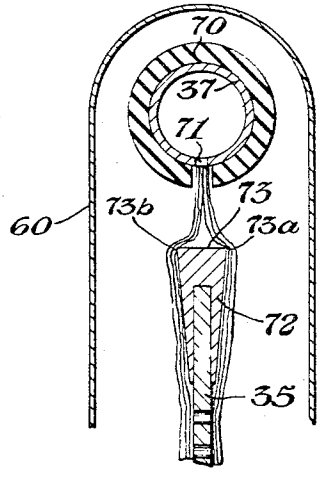


Fig. 11

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2 Claims. (Cl. 204—219)

This invention relates to a method and apparatus for effecting the electrolysis of an aqueous electrolyte, such as alkali metal halides and brines. More particularly, it relates to a method and apparatus for the electrolysis of brines in a mercury cathode cell of the vertical type.

One of the main advantages of the vertical arrangement of the effective anode and cathode areas in a mercury cathode cell over the horizontal arrangement is the conservation of floor space. While all the advantages of a horizontally arranged cell may be obtained in a mercury vertical cathode cell, the operating conditions to achieve high efficiency in the vertical arrangement are much more critical. For example, in the vertical cathode cell as well as in the horizontal flowing mercury cells, some impurities are normally present in the brine and the cell such as iron and other metals as well as graphite dust. These promote the formation of globules of a mercury emulsion which appear as lumps in the mercury and is often referred to in the trade as "butter." In horizontal cells the mercury circulates in relatively thick sheets and is sufficiently turbulent so that these lumps if present are carried along and collected and removed at the weir end of the cell. For economic consideration, the mercury being passed over a vertical cathode is in a considerably thinner sheet or stream. As a result of this reduced flow, the undesirable effects of the presence or formation of the butter or other impurities in the mercury are much more pronounced. It is essential to minimize the butter formation, and, if it is formed, to prevent its accumulation within the cell by providing for its continuous removal. The presence of the butter or lumps not only disrupts the flow pattern of the mercury over the cathode support but also results in the decomposition of the amalgam and the formation of hydrogen with a corresponding decrease in current efficiency.

It is therefore an object of this invention to provide an economical method and apparatus for electrolytic decomposition of alkali metal halide brines to produce the halogen and an alkali metal amalgam in a mercury vertical cathode cell. Another object is to provide an apparatus and method whereby high current efficiency is obtained. A still further object is to provide a method and apparatus for electrolytic decomposition of alkali metal brines in a mercury vertical cathode cell whereby the formation and the deleterious effects of impurities present in the mercury, such as the butter, are minimized. Other objects and advantages of the invention will become apparent from the following description of the invention taken in connection with the accompanying drawings in which:

FIGURE 1 is a front elevation of a typical mercury cathode cell of the invention.

FIGURE 2 is an end view of the cell shown in FIGURE 1.

FIGURE 3 is an elevation of the cell opened up to show the cathode arrangement of the cell.

FIGURE 4 is a cross section of the cathode arrangement taken along the line **4—4** in FIGURE 3.

FIGURE 5 is an elevation of the cell opened up to show the anode arrangement of the cell.

FIGURE 6 is a cros section of the anode arrangement taken along **6—6** in FIGURE 5.

FIGURE 7 is an isometric view of the diaphragm used in the cell.

FIGURE 8 is a portion of the cross section of the cell taken along line **8—8** in FIGURE 2.

FIGURE 9 is a sectional end view showing the details of the top portion of the cell.

FIGURE 10 is a partial perspective view of the bottom portion of the cell with parts cut away to show the internal arrangement of the diaphragm and cell parts.

FIGURE 11 is a section view of the mercury distribution system taken along line **11—11** in FIGURE 9.

FIGURE 12 is a top view of a plurality of the individual cells shown in FIGURE 1 assembled in a battery.

FIGURE 13 is a sectional view of the battery of cells shown in FIGURE 12 taken along line **13—13**.

Generally the cell is constructed of two sections joined face to face with a fluid tight seal along a vertical plane to form a box-like structure. To one of the sections the rectangular cathode supports are attached with the necessary mercury distribution system, while to the other the anodes are attached.

As shown in FIGURES 1 and 2, the box-like structure of the cell comprises a cathode section **21** attached to an anode section **22** by means of a plurality of bolts **23**. The cathode section is equipped with a mercury inlet **24** near the top of the section, while the anode section is provided with a mercury outlet **26** and a brine inlet **27** near the bottom and a gas vent **28** and brine outlet **29** near the top.

In FIGURES 3 and 4, the cathode section of the cell is shown in more detail. It comprises a cathode backing plate of a conductive material such as iron or steel **30** having a plurality of holes **31** near the outer edge by which means it is bolted to the anode section. A mercury header **32** is provided on the outer side of the plate which communicates with inlet **24**. As shown in FIGURE 4, a filler strip **33** is also attached to the outer surface of the cathode backing plate near the outer edges. The filler strip is recessed in the vicinity of the holes **31** so that the nuts used in bolting the cathode section to the anode section will not protrude beyond the filler strip. The filler strip is especially useful when the individual cell units are to be assembled in series to form a battery as shown in FIGURES 12 and 13. Also on the outer surface, metallic strips **34** are attached which provide means of attaching electrical contacts when the cells are used in series. For example these strips may be drilled and tapped so that the electrical contacts can be attached by means of bolting.

To the inner surface of the cathode backing plate, the cathode supports **35** are attached transversely. The support plates are attached to the cathode backing plate by means such that good electrical contact is obtained between the cathode supports and the cathode backing plate. To the edge of the supports distanct from the cathode backing plate a spacer **36** is attached which acts as a stiffener. A distributor pipe **37** extending also perpendicularly from the inner face of the cathode backing plate serves as a means of distributing the mercury over the cathode support. Distributor **37** communicates with mercury header **32** and may be further supported by being attached to the cathode supports by means of rods **39**.

The anode section of the cell as shown in FIGURES 5 and 6 comprises a rectangular frame **50** constructed from an electrically non-conducting and chemically resistant material, e.g. concrete or from steel or similar material which has been coated to provide electrical insulation and protection from corrosive fluids. A channel **51** is provided in the inner wall of frame **50** near one face. This channel provides a means of inserting an anode backing plate **52** made of conducting material to which the anode members **53** are attached. The anode backing material is usually a

graphite plate in which the rectangular anode members **53** are press fitted into grooves providing an electrical contact with the anode backing plate. Frame **50** is also provided with slots **54** at the top in which the anode members are also inserted. The anode members are positioned a distance from the bottom of the inner wall of frame **50** to provide a space **56** between the bottom of the anode members and the bottom inner walls **57** and **58** of frame **50**. Walls **57** and **58** are substantially free of any obstruction and are inclined inwardly and downwardly to form a bottom having a trough-shaped section. The trough-shaped bottom is also inclined along its length toward the mercury outlet **26** located at one end of the anode section. The inner wall of the top of frame **50** is constructed as an inverted trough to provide a space **59** communicating with brine and gas outlet **29** above the anode members. This can be seen in more detail in FIGURE 9 where the top portion of the assembled cell is shown. Bolts **23** on the face of frame **50** are attached to the frame and serve as a means of attaching the cathode section to the anode section in assembling the cell.

The cathode support plates and the anode members are attached to their respective backing plates at a predetermined distance apart such that when the cell is assembled the cathode support plates fit in between the anode members as shown in FIGURE 8. The cathode support plate and the anode members are separated by diaphragm **60**.

As shown in FIGURE 7 diaphragm **60** is a rectangular unitary structure made of a porous material having two side walls **61** and **62**, respectively. Between the side walls a multiplicity of corrugation-type folds of substantially equal height and length are inserted with the folds **63** and **64**, closest to the side walls, being attached to the side wall at the bottom. The folds are alternately open at one end and closed at the other. As shown the ends **65** of the folds are attached together to close the ends as by sewing when the diaphragm is made of a fabric. An outwardly extending flange **66** attached to the top of side walls and top of folds at one end extends transversely from the top of the side walls and longitudinally from the top of the folds. A second flange **67** attached to the folds at the bottom at the other end of the folds and the side walls extends longitudinally from the bottom of the folds and the side wall. The flanges provide a means of attaching the diaphragm to the anode section of the cell. Flange **66** may be bolted to frame **50** or inserted and held in place by flange **66** being located between the face of frame **50** and the cathode backing plate **30** when the cell is assembled. Flange **67** is attached to the anode section at the bottom as shown in FIGURE 8.

The diaphragm is generally made of a non-rigid type porous material such as fabric but it may be made of a rigid support material such as a non-conducting plastic screen which may be coated with an inert filler material such as asbestos to form a unitary rigid porous structure.

The diaphragm used in the cell is such that the anodes and cathodes are separated by the folds and also the interior of the cell is partitioned by means of the diaphragm into anode and cathode compartments so that the cathode compartments communicate with space **56** or bottom of the cell and the anode compartments communicate with space **59** at the top of the cell. However, since in the operation of the cell a small amount of gas may be generated in the cathode compartments, arrangements are made to vent this gas from on top of the cell. As shown in FIGURE 9, this is accomplished by attaching the top portion of the diaphragm to frame **50** at the top to provide a space **80** between the diaphragm and the cathode backing plate **30**. This space communicates with the cathode compartments and thus any gas formed is vented through outlet **28**. As shown in FIGURE 9 diaphragm **60** encompasses the cathode support plate at the top including the mercury distribution system, both sides of the support plate adjacent to the anode, and the edge of the support plate adjacent to the anode backing plate **52**. The diaphragm encompassing the cathode support plate extends to the bottom of the supports but does not enclose the plate at the bottom or at the edge adjacent to the cathode backing plate **30**. On the other hand the diaphragm encloses the anode plates at the bottom but not at the top. As shown in FIGURE 10, the diaphragm **60** encloses the anode member **53** completely at the bottom and the flange **67** at the bottom of the diaphragm is attached to anode backing plate **52** and frame **50** by means of a bar clamp **82** bolted to the anode backing plate and frame.

FIGURE 11 illustrates in cross section detail the system used for the distribution of mercury uniformly across the cathode support plate. As shown in FIGURE 11, distributor **37** is a pipe jacketed by a resistant material **70** to protect it from the corrosive fluids in the cell. In this manner it is possible to use as a distributor an ordinary steel pipe coated with a resistant material such as a plastic coating or plastic pipe. On the underside of the distributor directly above the cathode support, small holes **71** are drilled to provide a means of discharging the mercury in small diameter streams. Distributor **37** and the holes are situated directly above cathode support **35**. To aid in obtaining the initial distribution of the mercury from small streams into a uniform mercury sheet, a spreader bar **72** having a triangular cross section is attached to the top edge of the cathode support with the base that is the upper surface of the triangular bar being located below and centered with the holes in perforated pipe **37**. Thus, the small streams of mercury passing through holes **71** strike the base **73** of the spreader bar **72** and are dispersed into a uniform sheet covering the base. The mercury then flows over the edges **73***a* and **73***b* of the base of the bar and uniformly down both sides of the cathode support **35**.

In the operation of the cell, brine is introduced at one end of the cell through inlet **27** and flows through the unrestricted bottom of the cell to be distributed throughout the cell. Since the cathode support plates communicate with the bottom of the cell, the brine flows up from the bottom adjacent to the cathode support plate, through the diaphragm, past the anode and then with the chlorine the electrolyzed brine is discharged through outlet **29** at the top. The mercury used as a cathode is introduced in inlet **24** to maintain the required head within header **32**. From header **32** the mercury flows into distributor **37** where it passes through the holes in the bottom and spreads over the cathode supports flowing down each side of the support as a sheet completely covering it. At the bottom of the cathode support the mercury or amalgam resulting from the electrolysis falls through the incoming fresh brine to the bottom of the cell. It is directed to the center by the sloping bottom walls, flows to the discharge end of the cell, and is discharged through outlet **24**. The bottom of the cell is unobstructed and as a result of the sloping of the cell bottom the mercury immediately flows from the cell carrying with it any butter or other impurities which may be present in the mercury. By having the rich amalgam pass through the incoming brine aids in decreasing the butter formation upon the supports and elsewhere in the cell. Impurities in the brine tend to promote the butter formation and thus by having the rich amalgam passing through the incoming brine the amalgam entrains the impurities and induces butter formation at this point instead of upon the cathode plate surface where it would interfere with the mercury distribution or flow over the cathode support magnifying its undesirable effect. The butter or any impurities entrained in the mercury at the bottom are immediately carried out of the cell and may be recovered and separated from the mercury in the further processing of the amalgam. The sloping of the walls forming the bottom of the cell and the inclining of the bottom toward the outlet result in a low mercury inventory and any mercury lumps or impurities in the

mercury are carried along and not deposited within the cell where they can accumulate.

While each of the cells may be operated individually, the cells may be very conveniently assembled and operated as a bi-polar battery of individual cells in series. As shown in FIGURES 12 and 13, the cells are assembled together in a row between two end units **91** and **92**. When assembled the electrical contact clips **90** attached to each cathode backing plate contact the anode backing plate of the next proceeding cell. An anode contact plate **93** is attached to a bus bar **94** which extends through end unit **91** and is attached to an electrical source. To anode plate **93** electrical contact clips are attached such that they are in contact with the anode backing plate of the first cell. A similar means is used at the other end of the cell where a cathode plate **96** is attached to bus bar **97** through which the circuit is completed. Cathode plate **96** does not have the electrical contact clips but is positioned so it is in contact with the electrical contact clips of the cathode backing plate of the last cell. The individual cells can be held together by means of bolts **98** as shown. Thus, when assembled the cathode plate of one cell is in contact with the anode of the next cell so that during the operation the current passes through each cell in series. The brine is fed to each cell from a common header and then discharged to a common header. The mercury passing to each cell comes from a common header **101** and passes through a known type of circuit interrupter **102** prior to entering of the cell. Similar arrangement is used to collect the amalgam coming from the cells in a common header **103**. By assembly of the units as shown in FIGURES 12 and 13 the amount of floor space required to obtain a certain production is considerably decreased.

In addition to the operation of the cell such that the butter formation is minimized, the economy or efficiency of a cell is improved whenever a thin stream or sheet of mercury flowing down the cathode support plate can be maintained. By using a thin film of mercury, the concentration of the alkali metal in the amalgam is increased, while the inventory of mercury is decreased. Thus, an appreciable saving is obtained in handling and pumping costs in addition to having an amalgam from which the alkali metal may be more easily recovered. Often this may make the difference between an economical and uneconomical operation of the cell.

Attempts to use a thin film or sheet of mercury considerably complicates the operation of the cell, if it is remembered that it is essential at all times to have all of the surface of the cathode support plate covered with mercury or amalgam. Exposure of the cathode support plate to the brine will result in butter formation and hydrogen resulting in a decrease in efficiency of the cell. When a thin film of mercury is used, the film is easily disrupted and complete coverage of the support plate is not obtained. Thus, any advantage gained by use of a thin film may be offset by the decrease in efficiency resulting from not periodically completely covering the cathode supports.

The objective of completely covering the cathode support plate with a thin film or flowing sheet of mercury is accomplished by distributing the mercury in a thin sheet and maintaining this sheet as it flows on the support. The initial distribution is obtained by the employment of the mercury distribution system described above. Maintaining a thin sheet of mercury upon the cathode support as it flows down the support without interruptions is aided by using a perforated metal plate as a cathode support having a certain number and size of perforations. The employment of this combination in the instant cell further increases the efficiency; however, it is apparent that the distribution system as well as the combination can be used in any vertical type mercury cathode cell.

The desired results are achieved by maintaining critical limitations as to the diameter of the holes in the bottom of the distributor pipe or the diameter of the mercury distribution streams issuing from the distributor with respect to the distance it falls before impinging upon a substantially flat surface of a given width to disperse the mercury.

The cathode supports used are perforated plates to allow for more equal distribution of the mercury on both sides of the support. They are of sufficient thickness to provide a required rigid surface free from flexing which is generally obtained with a plate of from 3/64 to 1/8 inch in thickness in cells of the size usually used. Plates over 1/8 inch are generally undesirable since butter will collect in the perforations. It is essential that the cathode supports have a substantially smooth surface. Protrusions of any size will not be covered by the mercury film but will be exposed to the brine. Generally, the perforations are obtained by drilling or punching holes through the plate in a staggered fashion at predetermined distance apart. Sufficient number of perforations are used such that they represent from 10 to 50 percent of the total area of the cathode support plate. Perforations of a size of about 3/4 to 1 1/2 times the thickness of the cathode support plate balance the mercury flowing on both sides of the plate, are small enough to be easily maintained filled with mercury, but are large enough not to become plugged with impurities or any butter which may be formed. Thus by having the perforations of a given size equal to a given portion of the total surface of the contact plate, a thin stream of mercury distributed over the plate at the top will flow down the plate on both sides of the plate as a sheet without breaking or interrupting.

In the distributor pipe relatively small holes are used resulting in obtaining small diameter streams of mercury to impinge upon the base of the spreader bar. By employing small holes or streams and a mercury head of from 2 to 5 inches, the variation in rate of flow due to normal changes in the mercury head in the header are minimized. Holes from 3/64 to 1/8 inch in diameter or an opening having the equivalent cross sectional area are employed.

The spreader bar attached to the top edge of the support to provide the initial distribution of the mercury has a base or width of at least 1/4 inch and generally not greater than 8 times the diameter or size of the holes in the distributor. The preferred width is 1/4 to 1/2 inch.

The distance that the small streams of mercury fall before striking the spreader is also critical. The distributor must be maintained at a distance sufficiently above the base of the triangular section of the spreader bar so that the small mercury streams impinging upon the base are dispersed or spread in circular continuous overlapping pools prior to flowing over the edges of the bar while clinging to the sides and onto the plate. If the distance between the distributor and the base of the spreader bar is too great the mercury stream upon hitting the spreader bar is broken up in small fractions and the unity is lost. On the other hand, if the distance is not sufficient, proper spreading of the mercury is not obtained to give a thin sheet. To obtain the desired mercury sheet on the surfaces of the plate, the distance from the bottom of the distributor to the base of the spreader bar is maintained such that it is equal to from about 1 to 8 times the diameter of the mercury holes in the bottom of the distributor, preferably being from 1/8 to 1/2 inch. The spacing of the holes are generally located 3/4 to 1 1/4 inches apart and are such that the pools of mercury formed by the impingement of the mercury stream upon the spreader bar intercept and overlap each other and completely cover the spreader bar.

In a vertical cell constructed as shown in the attached drawings employing the above described distribution system, the average flowing mercury films on the cathode supports obtained were in the range of 0.1 to 0.5 mm.

The cathode support plates were 1/16 inch thick perforated plates having perforations of 1/16 inch in diameter.

The distributor system comprised a steel distributor tube encased in a plastic tube having mercury stream holes at the bottom of 1/16 inch in diameter spaced on 15/16 inch centers. The spreader bar attached to the top of the cathode support plates was a triangular piece having a 3/8 inch wide base centered about 3/16 inch below the mercury stream holes in the distributor tube.

In operation of the cell using a mercury head of about 2 inches of mercury in the header, flow of mercury obtained indicated that the thickness of the film on the support plates was about 0.2 mm.

What is claimed is:

1. In a mercury vertical cathode cell, a two piece shell-like cell body joinable in a fluid tight seal to form a cell cavity, said body pieces having the inner walls at the bottom which are substantially free of obstructions and which slope inwardly and downwardly with respect to the horizontal axis to form a trough-shaped bottom within the cell body, said sloping walls and resulting trough-shaped bottom being inclined with respect to the horizontal axis towards an outlet in the bottom of the cell body, a plurality of rectangular leaf-like cathode supports attached by at least one edge substantially perpendicularly to one piece of said cell body, said cathode supports being plates of ferruginous material having a thickness of between 3/64 inch and 1/8 inch, said plates having an array of perforations which are staggered with respect to adjacent perfortions but are substantially uniformly distributed over the side surfaces of the plates, said perforations having a diameter equal to between 3/4 and 1 1/2 times the thickness of said plate, the area of the array being between 10 percent and 50 percent of the area of the side surfaces of the plate, a plurality of rectangular leaf-like anode members attached substantially perpendicularly by at least one edge to the second piece of said cell body, said cathode supports and anode members being attached to their respective body pieces in a predetermined spatial relationship such that the leaf-like cathode supports are disposed between the leaf-like anode members, said cathode supports and anode members being attached to their respective body pieces at a distance from the bottom of the pieces to thereby provide a space between the bottoms of the cathode supports and anode members and the sloping bottom of the cell cavity, a diaphragm, said diaphragm being disposed between said cathode supports and anode members, said diaphragm enclosing the cathode support at the top and being open at the bottom to thereby provide communication of the cathode support with the bottom of the cell, said diaphragm enclosing the bottom of the anodes and being open at the top of the anodes to provide communication of the anodes with the top of the cell, means to introduce electrolyte between the diaphragm and cathode supports at the bottom of the cathode supports, means to discharge the electrolyte from between the anode members and the diaphragm from the top of the anodes, means to flow amalgam over the cathode supports, means to vent gases from the cell, and means to impress an electrolysis current between said anode members and said cathode.

2. In a mercury vertical cathode cell a frame constructed of electrically non-conductive material, said frame having the bottom inner walls substantially free of obstructions said bottom inner wall sloping inwardly and downwardly with respect to the horizontal axis to form a trough-shaped bottom, said trough-shaped bottom being inclined with respect to the horizontal axis toward an outlet located in the bottom, an anode backing plate of electrically conductive material attached to said frame at one face, a cathode backing plate of conductive material removably attached to the second face of said frame, a plurality of parallel spaced graphite anode members, said anode members being transversely attached to said anode backing plate to project into the area enclosed by said frame with a space being provided between the anode member and the frame at the top and bottom, a plurality of rectangular cathode supports attached transversely to said cathode backing plate to project into the area enclosed by said frame with a space being provided between the cathode supports and the frame at the top and bottom, said cathode supports being interleaved between and spaced from said anode member, said cathode supports being plates of ferruginous material which are between 3/64 and 1/8 inch thick and which have an array of perforations which are staggered with respect to one another, each of said perforations having a diameter equal to between 3/4 and 1 1/2 times the thickness dimension of said plate, said array of perforations covering between 10 percent and 50 percent of the surface area of the sides of said plates, a mercury distribution system for distributing the mercury over the cathode supports, said distribution system comprising a mercury carrying conduit having perforations on the side facing said cathode supports equivalent to opening of from 3/64 to 1/8 inch in diameter, said conduit communicating with a mercury header, a spreader bar having a triangular cross section with the base being of from 1/4 inch to 8 times the diameter of the perforations on the underside of the conduit, said spreader bar being attached to the top edge of each cathode support, said conduit being centered above the base of the spreader bar at a distance of from 1 to 8 times the diameter of the perforations in the conduit, said perforations being spread apart such that the pools of mercury formed upon the spreader bar by the impingement of individual streams of mercury discharging from the perforations upon the bar intercept each other to completely cover the spreader bar, a diaphragm separating the cathode supports and the mercury distribution system from the anodes such that the anodes communicate with the space at the top and the cathode supports and mercury distribution system communicate with the space at the bottom, means to introduce electrolyte in the space at the bottom of the cell below the cathode supports, means to discharge the electrolyte from the space at the top of the cell, means to vent gases from the cell, and means to impress an electrolysis current between said anodes and said cathodes.

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