Dec. 8, 1959 C. E. VAN WINCKEL ET AL 2,916,430

ELECTROLYTIC CELL

Filed Jan. 13, 1958 6 Sheets-Sheet 1

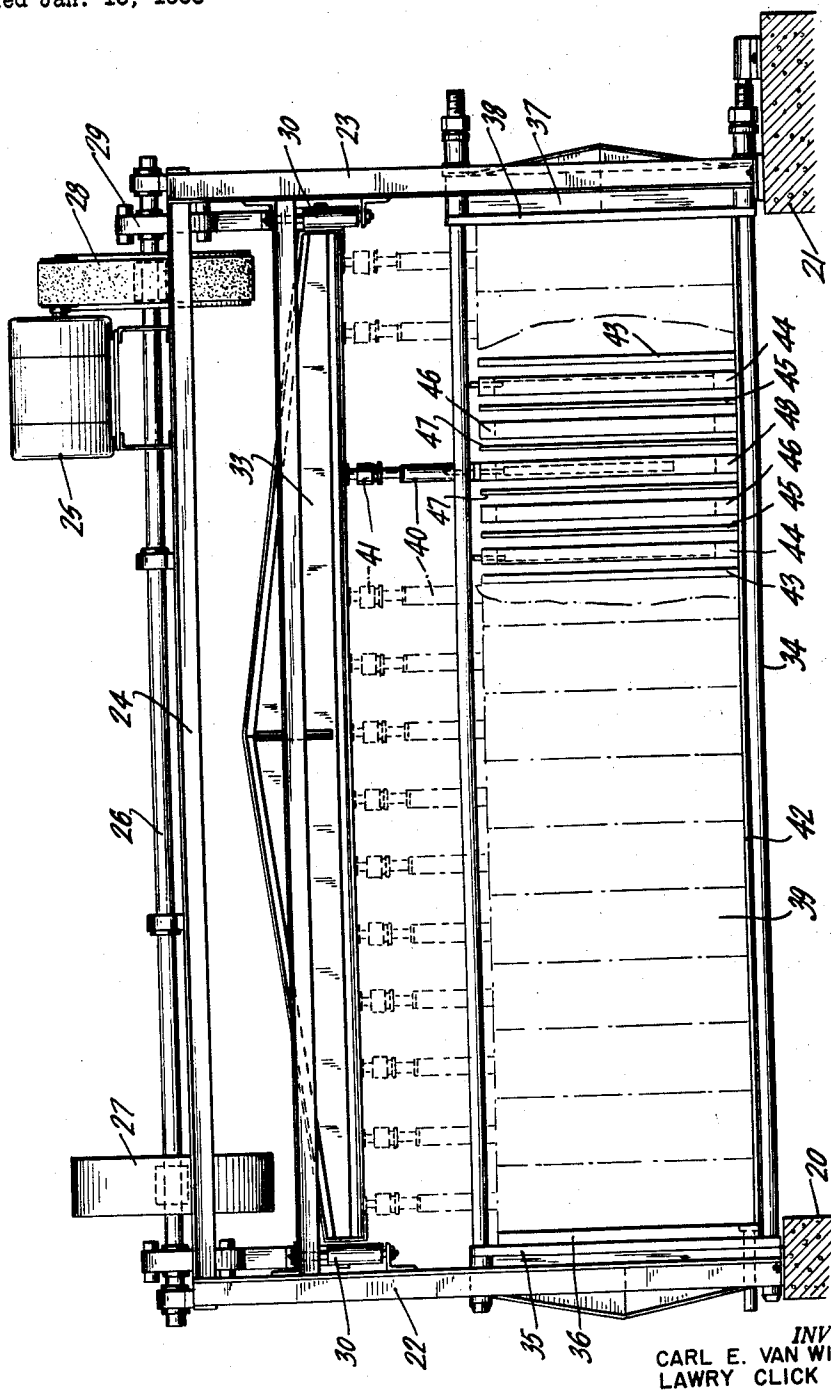


FIG. 1.

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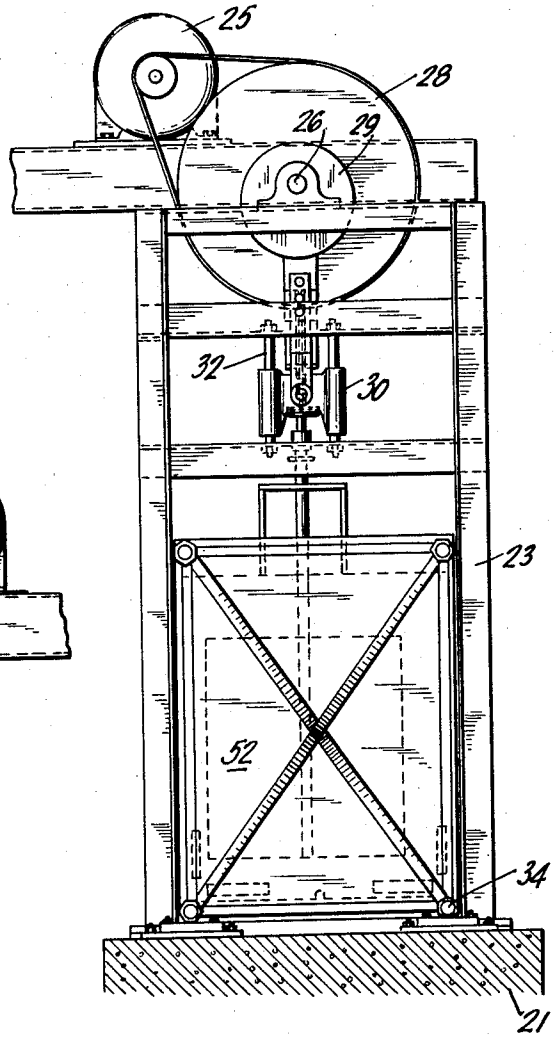
BY
Campbell Brumbaugh Free & Graves
Their ATTORNEYS.

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ELECTROLYTIC CELL

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FIG.3.



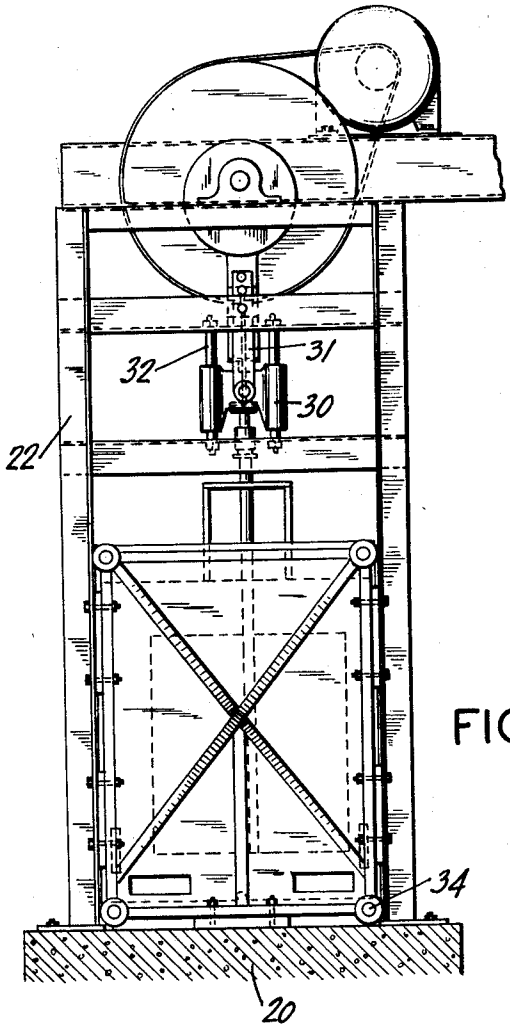


FIG.2.

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ELECTROLYTIC CELL

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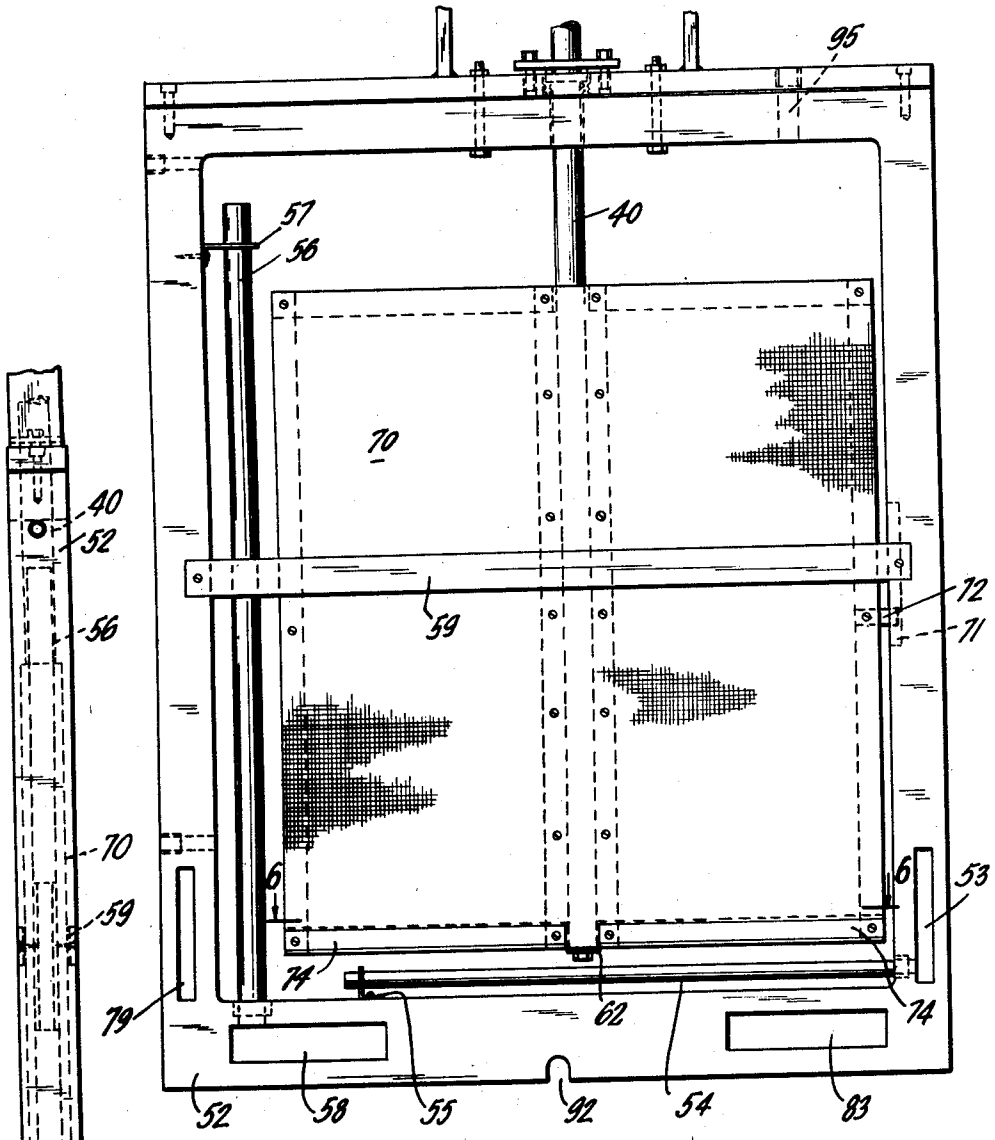


FIG.4.

FIG.5.

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ELECTROLYTIC CELL

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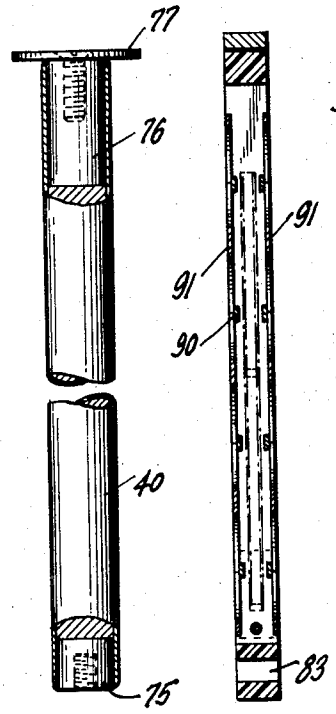


FIG. 7.

FIG. 11.

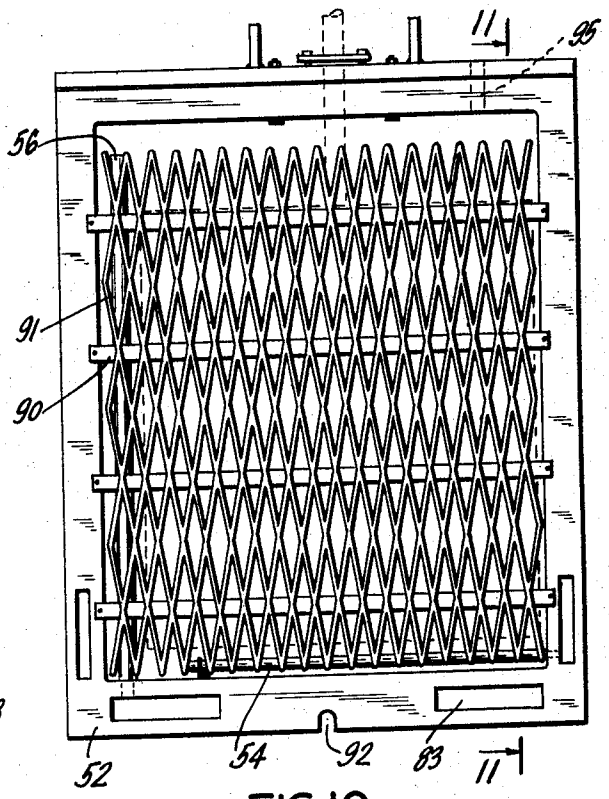


FIG. 10.

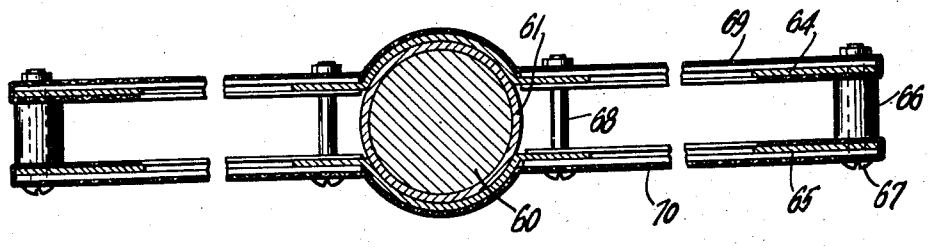


FIG. 6.

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ELECTROLYTIC CELL

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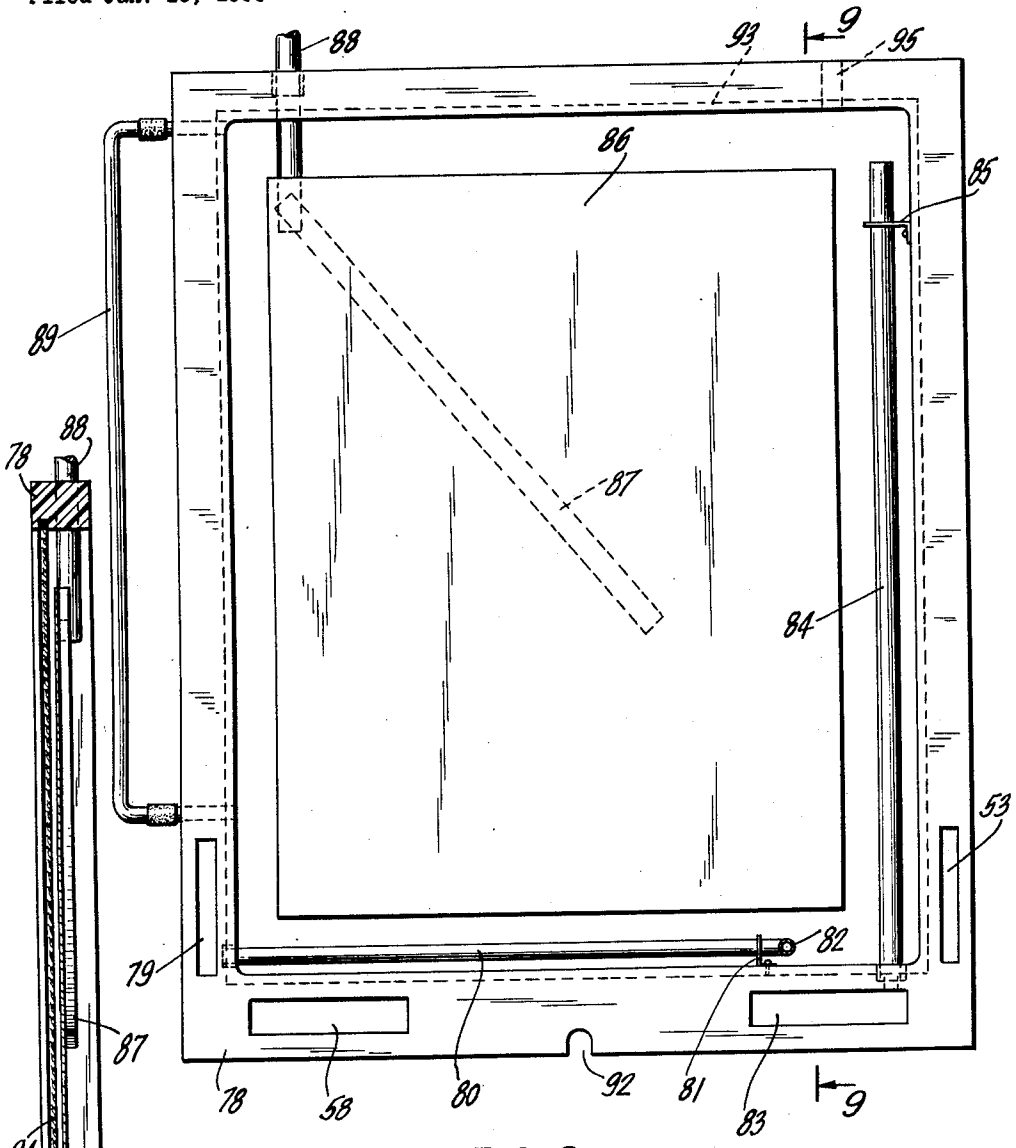


FIG. 8.

FIG. 9.

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ELECTROLYTIC CELL

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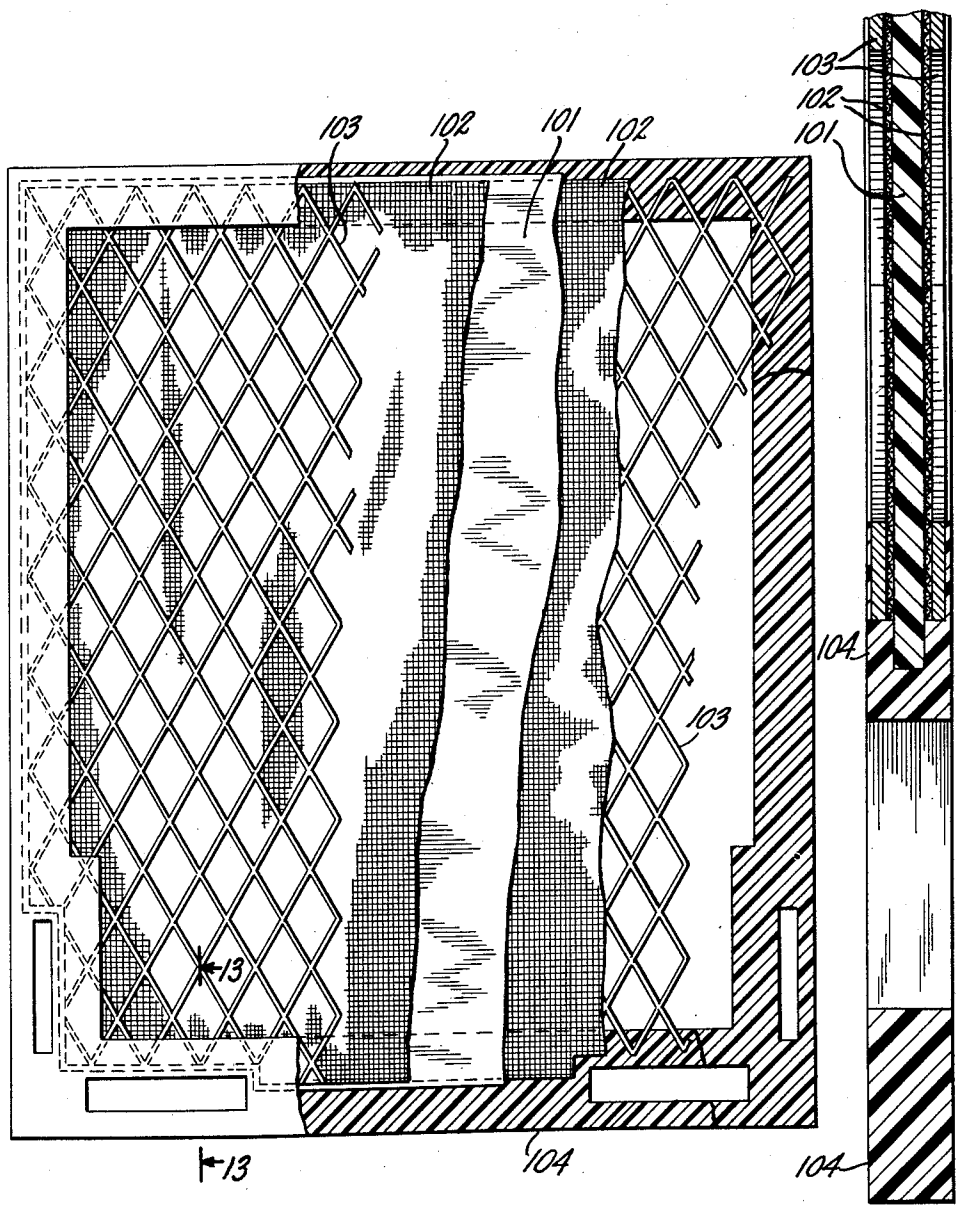


FIG. 12.

FIG. 13.

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2,916,430

ELECTROLYTIC CELL

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Application January 13, 1958, Serial No. 709,130

4 Claims. (Cl. 204—222)

In the application of Van Winckel, Smith, and Hutz, Serial No. 253,896, filed October 30, 1951, for "Electrolytic Apparatus," now abandoned, there is shown and described apparatus by means of which electrolytic processes may be practiced, and this invention embodies an improved apparatus by means of which the electrolytic treatment of organic compounds may be accomplished with certain attendant advantages hereinafter described.

The electrolytic treatment of certain organic compounds presents difficulties which are well recognized by those who are experienced in this art. Not only must the structure of the apparatus be such as to avoid the occurence of objectionable chemical reactions, but numerous controls over the conditions of treatment must be carefully watched, and the conditions maintained substantially uniform or within relatively narrow permissible ranges of variations.

As in the above-mentioned application, the present invention contemplates the use of a multiplicity of electrolytic cells, the structure of the cells being such as to facilitate their convenient assembly and maintenance and to insure that the treatment intended to be performed in the cells is actually performed in the manner desired.

Yet another object of the invention is to provide an apparatus by means of which organic compounds may be treated electrolytically while being maintained in a constant state of agitation and circulation, yet without generating conditions that tend to inhibit successful treatment of the compound or the resulting electrolytic cell structure.

Other and further objects of the invention will appear as it is described in connection with the accompanying drawings, wherein:

Figure 1 is a view in front elevation showing electrolytic apparatus constructed in accordance with the present invention, and showing a plurality of similar cell units, one of which has been expanded to show its component parts;

Figure 2 is a view in end elevation, as viewed from the left in Figure 1;

Figure 3 is a view in end elevation, as viewed from the right in Figure 1;

Figure 4 is an enlarged partial view showing the structure of the cathode and cathode cell unit;

Figure 5 is a view in side elevation of the structure shown in Figure 4;

Figure 6 is a view in horizontal section, taken on the plane indicated by the line 6—6 of Figure 4, this view being enlarged and broken away;

Figure 7 is a detail view of the cathode supporting rod shown in Figures 4 and 6;

Figure 8 is a detail view showing the anode, together with the cell frame structure in which it is supported;

Figure 9 is a view in vertical section, taken on the plane indicated by the line 9—9 of Figure 8, and looking in the direction of the arrows;

Figure 10 is a view in somewhat reduced scale showing a modified version of the cathode cell unit and the manner in which the diaphragm is supported in proper position with respect to the cathode;

Figure 11 is a view in vertical section, taken on the plane indicated by the line 11—11 of Figure 10, and looking in the direction of the arrows;

Figure 12 is a view similar to Figure 10 showing a modified form of diaphragm structure, being partly broken away and in section; and

Figure 13 is a greatly enlarged partial view in section taken on the line 13—13 of Figure 12, and looking in the direction of the arrows.

Referring to the above drawings and particularly to Figures 1, 2 and 3, the apparatus of the present invention is mounted upon a suitable foundation 20 and 21, the foundation 20 serving to support the head, while foundation 21 supports the tail end of the apparatus.

The end frames 22 and 23 include horizontal frame members 24 upon which a driving motor 25 may be mounted, the driving motor serving to rotate a drive shaft 26 that is journaled upon the frame structure hereinabove described. If desired, a fly wheel 27 may be driven by the shaft 26, and a belt or other suitable form of drive mechanism 28 may be utilized to transmit power between the motor 25 and the drive shaft 26.

At each end of the drive shaft 26 eccentric mechanisms 29 are provided, these mechanisms serving to reciprocate crossheads 30 by means of connecting links 31, the crossheads 30 being slidably mounted upon guide rods 32 upon the respective end frame members 22 and 23. A crossarm 33 is connected between the crossheads 30 and serves as the mechanism by means of which the cathodes of the electrolytic cells (as hereinafter described) may be reciprocated.

Between the end frames 22 and 23 a plurality (four, as shown in the drawings) of stay bolts 34 is provided, these stay bolts serving to maintain a plurality of electrolytic cell structures in a desired assembled position, as will be presently described. A head plate 35 is bolted to the frame 22 and has secured thereto a resin head plate 36 against which the adjacent electrolytic cell is secured in assembled position.

At the opposite end of the apparatus, the end frame 23 is provided with a follower plate 37 upon which is supported a resin follower plate 38, the latter being secured against the adjacent electroltyic cell when the apparatus is assembled as an operating unit.

Referring to Figure 1, it will be seen that a plurality of electrolytic cell units 39 is assembled between the head and follower plates and secured in position by the stay bolts 34. The cathode of each cell is adapted to be reciprocated by a compound shaft 40 (the details of which will be described presently), the upper end of the shaft being secured to the crossarm 33 by means of flexible couplings 41. The cells, moreover, are positioned upon an insulated supporting plate 42 that rests upon the lower stay bolts 34.

As will be seen from the portion of Figure 1 illustrating the units of a cell in expanded or exploded relationship, each cell unit consists of spacer or divider plates 43 positioned upon the outer sides of anode frames 44. Inwardly of the anode frames there are provided diaphragms 45 that are positioned against intermediate frames 46 that serve to space the diaphragms 45 from adjacent and inwardly disposed diaphragms 47 which are maintained against the cathode frame unit 48. Suitable sealing material may be provided, if desired, in order to assure an effective seal between the several elements above-mentioned. The material of the anode and cathode frames, as well as the intermediate frames 46 and the spacer plates 43, consists of an impervious furfuryl

alcohol resin asbestos mixture suitably formed in sheet or frame formation and provided with the ports and other configurations presently to be described. The furfuryl alcohol resin asbestos material may be cast- or pressure-molded and, as above stated, suitable gaskets or other sealing material may be provided to insure an effective seal between the adjacent faces of the assembled elements.

Details of the structure of these cell units will appear from an inspection of Figures 4, 5, 8, 9, 10 and 11 and, referring to Figures 4 and 5, the cathode structure will be seen to include a cell frame member **52** having, in the lower right-hand portion thereof (as viewed in Figure 4), a duct **53** which extends from face to face, and thus forms, with adjacent cell units, a manifold by means of which the catholyte may be furnished to the cells. It is preferred that each frame unit **52** be provided with a glass or other non-conducting tube **54** sealed within the frame and communicating with the duct **53**, the outer end thereof being suitably supported by means of a bracket **55** in order that the catholyte may be directed into the cell at a point where optimum electrolyte action will be facilitated.

The catholyte is withdrawn from the cell by means of a glass or other non-conducting tube **56**, the upper end of which is supported in the frame **52** by means of a bracket **57** and terminates at a level at which it is desired to maintain the catholyte within the cell during operation. The tube **56** is sealed to the bottom portion of the frame **52** and communicates with a through duct **58** which forms, with adjacent cells, a discharge manifold by means of which the catholyte may be withdrawn from the cells together with any gases generated therein.

In the form of the invention shown in Figure 4, a transverse plate or bar **59** is secured to the opposite sides of the frame **52** and flush therewith in order that the adjacent diaphragm **47** may be supported and maintained in their desired positions during operation.

In order that the cathode may be reciprocated to insure constant agitation of the catholyte within the cell and thus enhance the diffusion at the surface of the cathode, the shafts **40** are formed with a core **60** of aluminum and a stainless steel shell **61**. This structure serves to protect the aluminum from the catholyte material and, at the same time, provides a conductor having adequate capacity and minimum weight to carry the current required during the operation of the apparatus. At its lower end the shaft is sealed by means of a plug **62** welded thereto and supports a cathode frame including spaced spiders **64** and **65**, these being secured together by spacers **66** and bolts **67**. Adjacent the shell **61** the spiders are clamped thereto by means of bolts **68**, and stainless steel wire screens **69** and **70** are secured over the respective spiders **64** and **65**. One side of the frame **52** may be formed with a groove **71** within which a guide finger **72** rides, the guide finger being secured to the cathode frame in order to insure that the cathode will be maintained in its desired position during operation. The lower extremities of the cathode are preferably formed with angle flanges **73** and **74** in order to insure effective agitation.

Figure 7 illustrates, in greater detail, the structure of the compound shaft and in order that the aluminum core may be sealed effectively from the catholyte, a lower plug **75** is welded therein, the upper end of the shaft being provided with an upper plug **76** to which an operating flange **77** may be secured by means of a screw.

The structure of the anode cell unit is illustrated in Figures 8 and 9 and will be seen to include an anode frame member **78** having in its lower left-hand side a through port **79** which, together with the ports of adjacent members, forms an anolyte manifold, the port discharging into the cell unit through a glass or other non-conducting tube **80**, the outer end of which may be supported by bracket **81**. As shown, the outer end of the tube **80** is provided with an L-shaped extremity **82** in order that the anolyte may be discharged in a direction perpendicular to the plane of the cell unit.

Within the bottom of the cell unit a through port **83** is formed to serve as the discharge manifold for the anolyte, this duct communicating with the interior of the cell through a vertically positioned glass or other non-conducting tube **84** which is secured in position by a bracket **85**. The height of the tube **84** is such that the level of the anolyte within the cell will be maintained in a desired position, the anolyte and any gases generated during the operation being withdrawn through the tube **84**.

Within the cell **78** there is shown an anode **86**, this anode being formed as a stainless steel plate provided with an angularly positioned conductor bar **87** and secured to a stainless steel supporting shaft **88**, the shaft being secured within the frame **78**. If desired, a gauge glass **89** may be provided in order to indicate the height of the anolyte within the cell.

In the form of the invention illustrated in Figures 10 and 11, the cathode cell frame unit is shown (upon its opposite sides) as being provided with a plurality of transversely extending supporting bars **90** upon which there has been secured an expanded metal grid **91** by means of which the adjacent diaphragms may be positioned when the apparatus is assembled for operation.

To facilitate proper alignment of the cell units in the assembled position of the cell, the bottoms of each unit are formed with a notch **92** which engages a positioning rib (not shown) formed upon the bottom plate **42**.

As an important aspect of the present invention, the anode frame **78** is formed with an interior groove **93** within which there is secured a cloth skirt or shield **94** in order to prevent oxygen or other gases liberated at the anode from contaminating the anolyte or the structure (especially the diaphragm) in communication with the anolyte. The material of this skirt is one which will resist the action of the anolyte and the small amounts of organic materials with which the anolyte is sometimes contaminated, and also of a material that presents a minimum of resistance to the current flow of the material. It has been found that nylon and asbestos cloth serve satisfactorily in this respect. Obviously where conditions make it unnecessary, the skirt is dispensed with.

As above mentioned, in place of gaskets or other sealing layers between the components of the cells, uncured rubber (cured after assembling the cells) may be used, or other suitable flexible material resistant to the compounds present in the cells may be used. The anolyte and catholyte inlet tubes **80** and **54**, respectively, provide a relatively long path for the passage of electricity between the cells, thus increasing the resistances thereof and minimizing the amount of electric current by-passed from one cell to the other. These inlet tubes also serve as a convenient means of draining the several compartments of the cell. As illustrated at **95**, vent ports are provided in the tops of the anode and cathode cells to prevent the creation of a partial vacuum therein due to the wavering effect of the anolyte and catholyte during operation, thus relieving the cell structure incident to such a vacuum.

It will be observed that the outlet tubes **56** and **84** of the cathode and anode compartments, respectively, have their upper or entrance ends adjacent the top of the respective cells or compartments, whereas the respective inlet tubes **54** and **80** have their discharge openings at points remote from the inlet manifolds. This results in a relatively long path of the electrolyte between the cells, the path being of a relatively constricted nature in view of the size of the tubes and, as a result, a relatively high resistance path is provided to prevent the by-passing of appreciable current around the cells and through the electrolyte in these channels and tubes. Providing the overflow at the top of the cells with the vents above mentioned

results in the separate removal of liquid and gas, thus preventing surging within the cells and electrolyte circulating mechanism. With the diaphragm supporting mechanism hereinabove described, it may be possible to dispense with the vent in the cathode compartment. Plugging of the vent for the cathode compartment tends to occur by reason of sublimation of the organic liquid toward the end of the reduction cycle. By providing discharge ducts and manifolding for the cathode vents and by utilizing steam or other heating media, the tendency toward plugging of the cathode vents will be reduced.

Figures 12 and 13 show a preferred form of reinforced diaphragm structure for use in connection with the cell structure hereinabove described. In this structure, the diaphragm (hereinabove described) is indicated at **101**, being provided on opposite sides thereof with screens **102**, upon the outer sides of which expanded metal **103** is provided. The diaphragm, screens, and expanded metal members are all assembled and maintained in unitary relationship as a single element by means of a frame **104** preferably formed of furfuryl alcohol resin. The frame is provided with apertures adapted to align with the corresponding apertures in the frames of the cells as hereinabove described and, to accomplish this, the expanded metal, screens, and diaphragm are placed in the proper position in a mold in which the uncured resin is placed about the edges of the mold. The mold is then closed and the entire charge of resin cured under pressure to form a one-piece molded diaphragm including supports in the form of the frame, as illustrated in Figures 12 and 13. Also asbestos cement mixtures and sheets of asbestos impregnated with furfuryl resin serve satisfactorily as diaphragms.

Instead of the form of cell illustrated in Figure 1, the intermediate frame may be eliminated and the anode frames may be provided on either side of the cathode frame spaced only by the diaphragm and, of course, suitable gaskets to provide effective sealing.

In the cell structure illustrated in Figure 1, intermediate spacer frames **46** are provided in which a neutral solution can be circulated between the adjacent diaphragms. For example, in an operation wherein the reduction of nitro-compound is accomplished electrolytically, diaphragms of furfuryl alcohol resin asbestos mixture could be utilized between the intermediate frames **46** and cathode frame **48**. The intermediate frames **46** would be formed of furfuryl alcohol resin, and within these frames a neutral solution of sodium sulfate would be circulated. The diaphragms **45** would be formed of fluorinated aliphatic resins, and the anode frames **44** would be of lead or resin lined metal. The anode itself would be of lead and the anolyte would be a sulfuric acid solution of chromic sulfate. In this operation, the manifolds of the anode frame would be lined with a non-conductor such as furfuryl alcohol resin or fluorinated aliphatic resins, and the connections to the anode compartment would be made preferably externally through glass or other suitable non-conducting flexible couplings.

Instead of complete and full manifolding of the cell structure, as hereinabove described, it is sometimes preferable to manifold the connections to the cathode and intermediate compartments and to use external connections for the anode compartment.

It will be understood, of course, that where operating conditions make it expedient, the intermediate frames and added diaphragms may be dispensed with.

This application is a continuation-in-part of our copending application Serial No. 365,134, filed June 30, 1953, now abandoned.

We claim:

1. Electrolytic apparatus comprising an upright generally rectangular planar hollow cell frame having at least one open side of relatively large dimension as compared with the axial frame thickness, said frame enclosing a cell chamber, an electrode housed within said cell chamber mounted for reciprocation, a diaphragm secured at said open side of said frame in liquid-tight relationship with respect thereto, and means supported by the frame and insulated from said electrode to reinforce the diaphragm against forces applied thereto having a component parallel to the axis of the frame, said reinforcing means guarding the diaphragm from engaging the electrode, and stabilizing said diaphragm to prevent excessive motion thereof when the electrode is reciprocated.

2. Apparatus, according to claim 1, wherein the reinforcing means comprises elements extending across the open side of the frame contiguous to the plane of the diaphragm.

3. Apparatus, according to claim 1, wherein the reinforcing means comprises at least one bar extending across the open side of the frame and lying flush with said side.

4. Apparatus, according to claim 1, wherein the reinforcing means comprises a grid extending across the open side of the frame and lying flush with said side.

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