Aug. 20, 1935. J. E. JEWETT 2,012,046

ELECTROLYTIC PROCESS AND APPARATUS

Filed Jan. 31, 1930 2 Sheets-Sheet 1

Fig. 1.

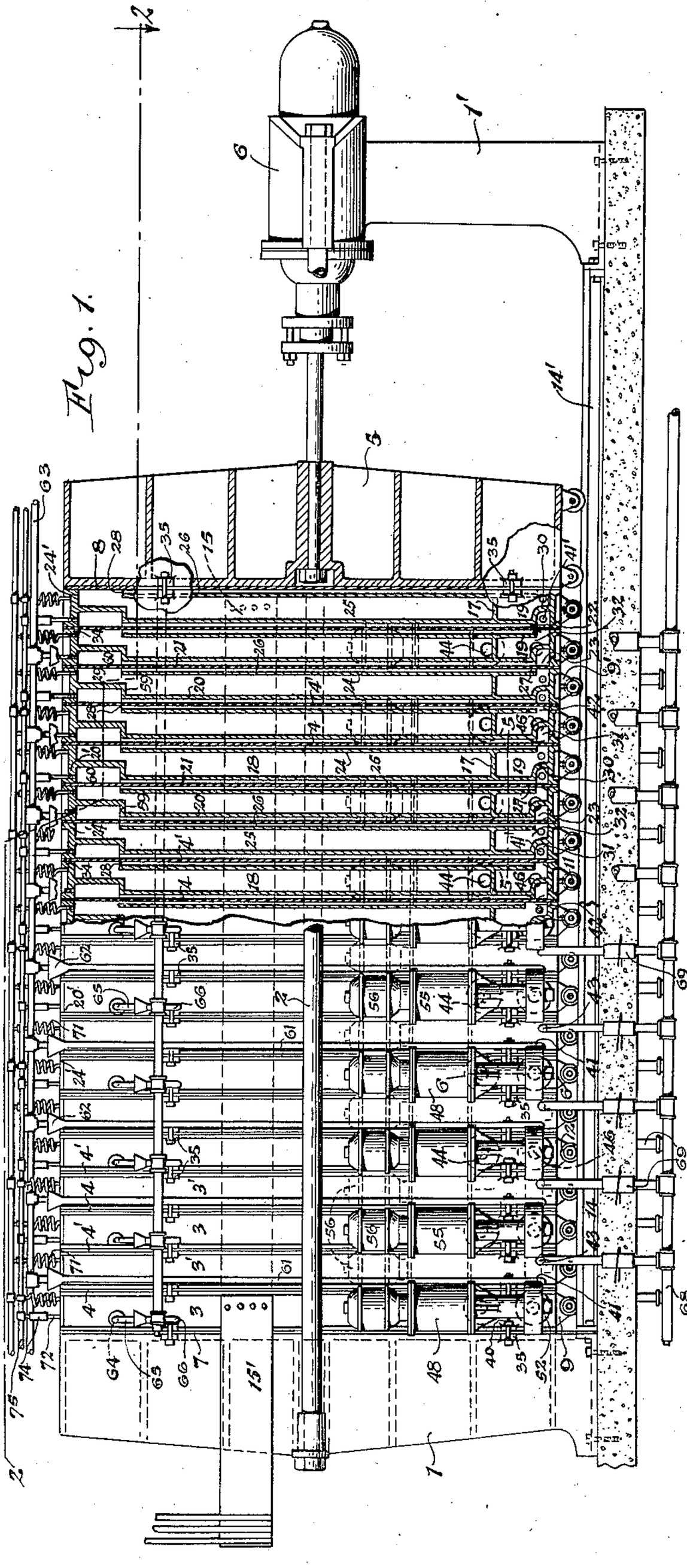
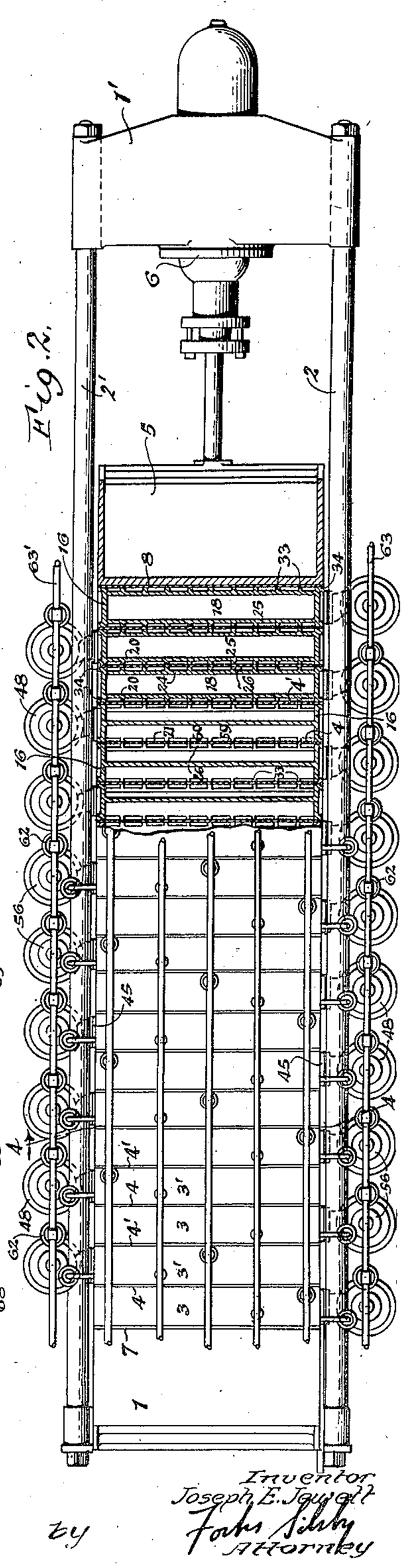


Fig. 2.



Inventor
Joseph E. Jewett
By Fahs Sibley
Attorney

Aug. 20, 1935. J. E. JEWETT 2,012,046

ELECTROLYTIC PROCESS AND APPARATUS

Filed Jan. 31, 1930 2 Sheets-Sheet 2

Fig. 3

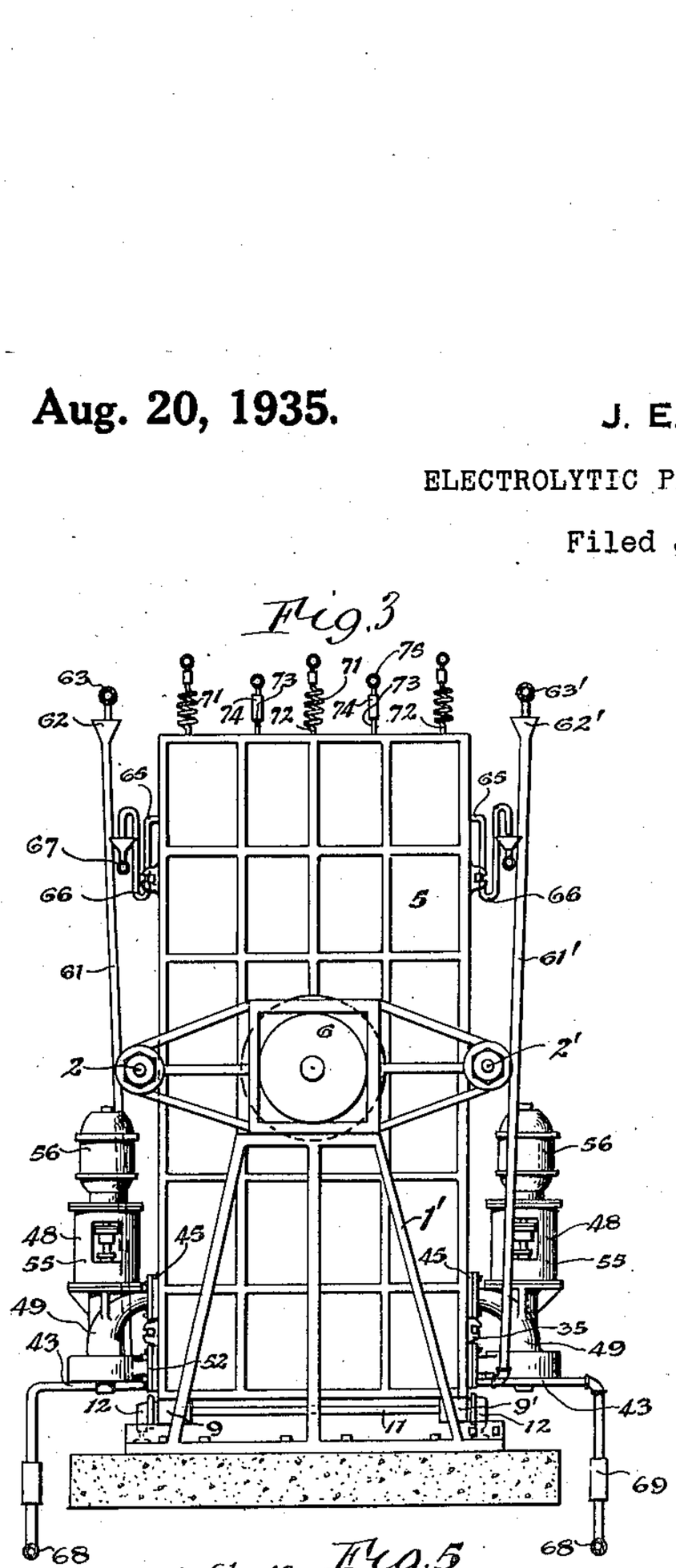


Fig. 4.

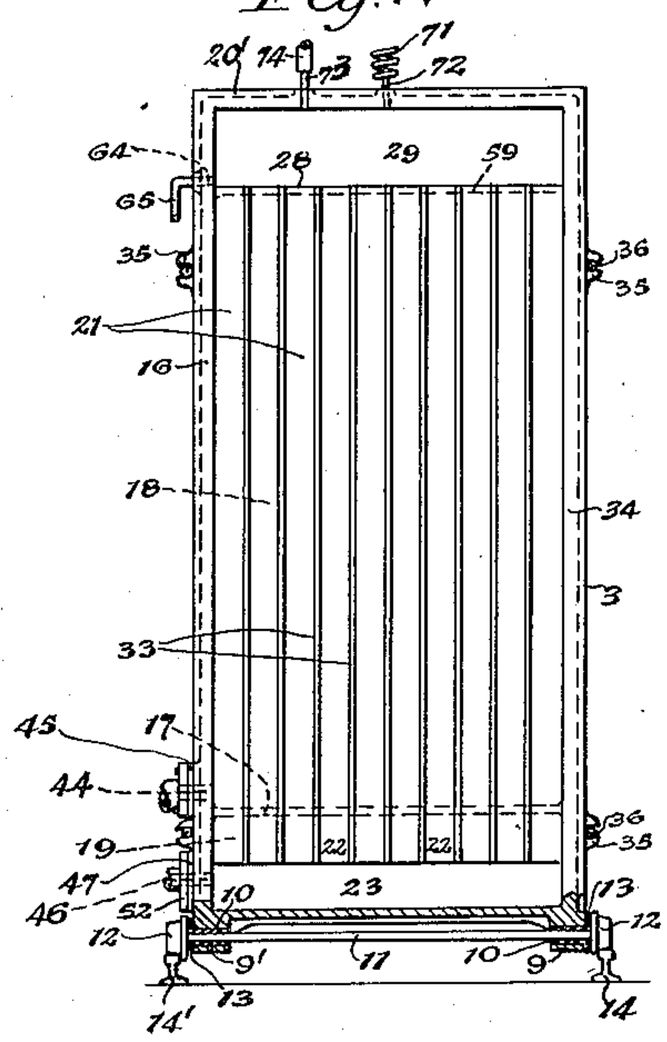


Fig. 5

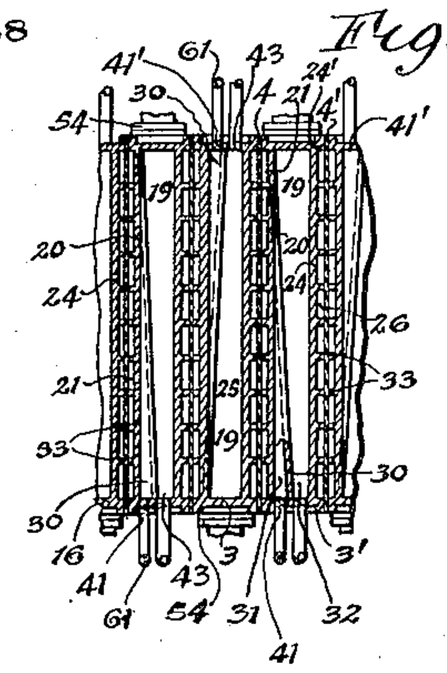


Fig. 6

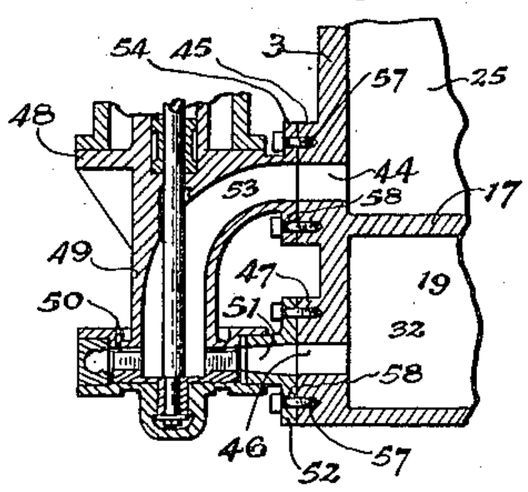


Fig. 7.

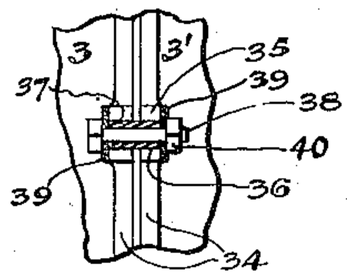
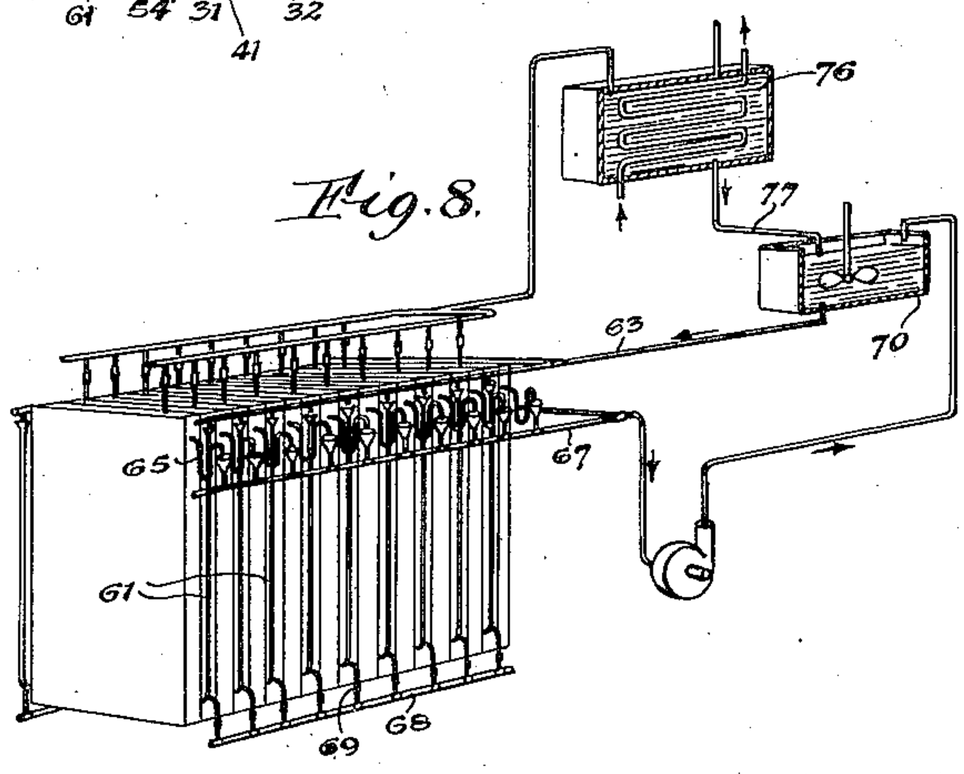


Fig. 8.



Inventor
Joseph E. Jewett

by Fohs Sibley
Attorney

Patented Aug. 20, 1935 2,012,046

UNITED STATES PATENT OFFICE

2,012,046

ELECTROLYTIC PROCESS AND APPARATUS

Joseph E. Jewett, Buffalo, N. Y., assignor to National Aniline and Chemical Company, Inc., New York, N. Y., a corporation of New York

Application January 31, 1930, Serial No. 424,910

17 Claims. (Cl. 204—9)

The present invention relates to improvements in processes and apparatus for carrying out electrolytic reactions, and is particularly adapted for carrying out the electrolytic reduction of nitro, azoxy, or azo bodies.

In the manufacture of hydrazobenzenes by the electrolytic reduction of the corresponding nitro, azoxy or azo bodies, the reduction products are precipitated as solids which stick to the diaphragms and walls of the electrolyzer and decrease the efficiency of the apparatus. Frequently, the solids are deposited as a hard mass which is difficult to remove.

One object of the present invention is the provision of a process for conducting electrolytic reactions wherein the electrolytic solution is passed between the diaphragm and the electrode to keep the surfaces free from incrustation. Another object is the provision of an electrolyzer wherein the liquor in the space between the electrodes and diaphragm is kept in circulation to prevent the deposition of solids on the walls of the apparatus. Another object is the design of an electrolytic cell which is compact and is convenient to assemble and disassemble, and which is inexpensive to manufacture, occupies little floor space, has a high output per unit of capacity and a low power cost in comparison with cells of the same type. These and other objects are accomplished in the practice of the present invention.

An illustrative embodiment of a manner in which the process may be carried out in practice and of an apparatus in which the process may be practiced is shown in the accompanying drawings wherein Figure 1 is a side view partly in section of an assembled apparatus; Figure 2 is a plan view; Figure 3 is an end view; Figure 4 is a section of a detail taken on line 4—4 of Figure 2; Figure 5 is a section of a detail taken on line 5—5 of Figure 1; Figure 6 is a section on an enlarged scale of a detail taken on line 6—6 of Figure 1; Figure 7 is an enlarged view of the cell fastening means; and Figure 8 is a diagrammatic view of the circuit through which the anode liquor flows.

Referring to the drawings, the numerals 1, 1' indicate a pair of end cheeks or standards which are connected by a pair of longitudinally extending parallel bars 2, 2'. The electrolyzer is constructed of a plurality of cells or units in pairs 3, 3' which may be of any number desired, and are clamped together, side by side, separated by diaphragms 4, 4', etc. The units 3, 3' and diaphragms 4, 4' are held in clamped position between the cheek 1 and follower 5 by a suitable clamping mechanism indicated generally by the numeral 6, which may be hydraulically operated. A sheet 7 of insulation material is interposed between the cheek 1 and first unit 3, and a similar sheet 8 of insulation material is interposed between the follower 5 and the last unit, to insulate the units from electrical contact with the follower 5 and cheek 1. The units 3, 3' are each equipped at the bottom on each side with lugs 9, 9', through which pass the insulation sleeves 10, suitably keyed to the lugs. A shaft 11 passes through the insulation sleeves 10 and is suitably keyed thereto, and at its outer ends carries the flanged wheels 12 rotatably held thereon by suitable means and insulated from electrical contact with the lugs by insulation washers 13. The wheels 12 operate on the guide rails 14 and 14' to permit easy manipulation of the units in assembling and disassembling the electrolyzer. The electric current enters the apparatus at the terminal 15 and leaves the apparatus at the terminal 15'.

Each unit 3, 3' comprises a hollow casing or frame 16 of generally rectangular shape which may be constructed of cast iron or other suitable conducting material. A horizontal web or partition 17 spaced from the diaphragms 4, 4' divides the interior of the frame 16 into an upper compartment 18 and a lower compartment 19. A wall 20 forming an anode completely closes the side 20' of the upper compartment 18 and is spaced from the diaphragm 4 to form an anode chamber 21 therebetween, which communicates at its bottom 22 through the opening 23, with the lower compartment 19. A second wall 24, forming a cathode, blocks the other side 24' of the upper compartment 18 and is spaced from the anode 20 to form a cathode reservoir 25 therebetween, and is spaced from the other diaphragm 4' to form a cathode chamber 26 therebetween. The cathode chamber 26 communicates at its bottom 27 with the lower compartment 19, and at its top communicates with the cathode reservoir 25 through the passage 29. A vertically extending warped wall or partition 30 diagonally divides the lower compartment 19 into a lower anode compartment 31 and a separate lower cathode compartment 32.

On the sides of the anode 20 and cathode 24 are cast the parallel spacing ribs 33 which lie flush with the flanges 34 which extend around the perimeter of the units 3, 3' at both sides 20', 24'. Lugs 35 are cast integral with the flanges 34 and are slotted at 36 to receive the

insulation sleeves 37. In assembly the units 3, 3' are clamped together with the diaphragms 4, 4', etc., therebetween, resting against the flanges 34 and ribs 33, and are held in clamped position between the end cheek 1 and follower 5. The bolts 38 pass through the insulation washers 39 and sleeves 37 in the slotted lugs 35. Nuts 40, insulated from electrical contact with the lugs 35 by the insulation washers 39, are threaded onto the ends of the bolts 38 and hold the diaphragms 4, 4', etc., and units 3, 3', etc., in assembled position when pressure on the follower 5 is released to disassemble the electrolyzer or for any other reason.

The lower anode liquor chamber 31 communicates with the anode compartment 21 through the opening 23, and receives its supply of anode liquor through supply passage 41. The lower cathode liquor chamber 32 communicates with the cathode compartment 26 through the opening 42 at its bottom 27, and receives its supply of cathode liquor through supply passage 43. Each unit is also provided with a circulating passage 44, flanged at its outer edge 45, which communicates with the cathode reservoir 25. A passage 46, also flanged at its outer edge 47, communicates with the lower cathode compartment 19. Each unit carries a pump assembly 48 of conventional construction and need not be described in detail. The pump assembly 48 consists of an integral pump casting indicated generally by the numeral 49 having a rotor chamber 50 therein. The pump casting 49 has a pump discharge passage 51 therein, leading from the periphery of the rotor, which is flanged at its outer end, as indicated at 52, and a curved pump supply passage 53 leading to the center of the rotor, which is also flanged at its outer end as indicated at 54. At one end of the pump casting 49 the spacer 55 is suitably secured, and at the other end of the spacer the motor 56 is secured. The entire assembly comprising the pump casting 49, the spacer 55 and motor 56 is secured to the respective units by the bolts 57 which pass through alined bores 58 in the flanges 52 and 47, respectively, and flanges 54 and 45, respectively, thus forming a continuous passageway from cathode reservoir 25, passage 44, passage 53, rotor chamber 50, passage 51 and passage 46.

In assembly, an upper anode compartment 21 is formed between the diaphragm 4 and the wall 20, which functions as an anode, communicating at its bottom with the lower anode chamber 31 through the opening 23. The upper cathode compartment 26 formed between the wall 24, which functions as a cathode, and diaphragm 4', communicates at its bottom 27 with the lower cathode chamber 19 through the opening 42. The wall 24 does not extend completely to the top of the casing, but terminates at 28, some distance from the top, leaving an open space 29 which communicates with the cathode reservoir 25. The wall 20 extends fully to the top of the unit, but is offset at its top at 59 to provide a compartment 60 communicating with the upper anode compartment 21, but is closed off from the cathode reservoir 25.

The operation of the apparatus will now be described in connection with the reduction of nitrobenzene to hydrazobenzene, but it is to be understood that the invention is not limited thereto. In operation, anode liquor is supplied to the lower anode compartments 31 as a continuous stream through the supply passages 41 from lead-in supply pipes 61. Lead-in supply pipes 61 have funnels 62 at their upper ends, each receiving a dripping stream from main anode liquor supply pipe 63 which runs parallel to the assembled electrolyzer. As shown, the supply passages 41 and 41' are on alternate sides of alternate units. Supply passages 41 and 41', communicating with lead-in supply mains 61 and 61' on opposite sides of the electrolyzer, correspond in the respective units 3 and 3'. A main anode liquor supply pipe 63' supplies anode liquor to the lead-in pipes 61' in a small trickling stream. The anode liquor flows into compartments 31, and flows upward through the openings 23 into the upper anode compartment 21, formed between the anodes 20 and the diaphragms 4. Overflow outlets 64 at the top of each unit communicate with compartments 60 to carry off the overflow through pipes 65 and traps 66. The overflow drips into a run-off pipe 67 through which it is pumped to a mixing tank 70 where it is mixed and flows back to the supply lines 63 and 63' to be reused. The anode liquor may be reused till exhausted.

Cathode liquor is introduced into each unit through the inlets 43 communicating with the lower cathode compartments 32 and is shut off when the liquor fills the upper cathode compartment 26 and cathode reservoir 25 to the upper edge 28 of the cathode 24. The cathode liquor is supplied to the units from main supply pipes 68 through rubber connections 69 to the inlets 43. The pumps 48 are set in operation and circulate the cathode liquor through pump discharge 51, passage 46, lower cathode chamber 19, through opening 42, into upper cathode chamber 26 and overflows at the top 28 of the cathode 24 into cathode reservoir 25, whence it is withdrawn through passage 44 and pump supply passage 53 to the pump to be recirculated. The liquor in the cathode compartments is kept at about 95° to 98° C. by suitable heating means (not shown). The electric current enters the apparatus at the terminal 15 joined to an anode and passing through the anode liquor, diaphragm and cathode liquor successively, leaves the apparatus at the terminal 15' joined to a cathode. The nitrobenzene in the cathode compartments is reduced to hydrazobenzene, which is prevented from depositing on the walls of the diaphragms and cathodes by the circulation of liquor through the passages 26. When the reduction is complete, the cathode liquor is drained from the cathode compartments through the pipes 43 which serve as both a drain and supply pipe and the apparatus is ready to receive another charge of cathode liquor.

Each unit 3, 3' is provided with a vented coil 71 connected to the cathode chamber at its top through the pipe 72. Material which volatilizes in the cathode chamber passes into the condenser coil 71 where it is condensed and returned to the cathode chamber. A pipe 73 connected to each anode chamber, is connected by the insulation coupling 74 to a common vent line 75. Vapor from the anode liquor passes through the pipe 73, and coupling 74 into the vent line 75, and is conducted to the condenser 76 where it is condensed and discharged into a mixing tank 70 by the pipe 77. From the mixing tank the liquor flows into the mains 63 and 63' and is fed back into the cells.

When it is required to take down the apparatus for cleaning or for any other purpose, the nuts 40 are turned off the bolts 38 at the desired separation point, and the follower is retracted, drawing with it along the rails 14, 14' units on that side of the separation point. After cleaning,

the follower carrying with it the respective units, may be returned to operative position, and the units again bolted together at the point of separation.

What I claim as new and desire to secure by Letters Patent is:

1. In the electrolytic reduction of nitrobenzene, the process which comprises flowing the cathode liquor in a stream through the space formed between the cathode and diaphragm to keep the surfaces of the cathode and diaphragm free from incrustations.

2. In the electrolytic reduction of nitro compounds, the process which comprises flowing the cathode liquor in a stream through the space formed between the diaphragm and cathode plate, cascading the cathode liquor over the cathode plate into a reservoir, and recirculating the cathode liquor.

3. An electrolyzer comprising an open-sided frame having its sides closed by diaphragms, an anode parallel to the one diaphragm and spaced therefrom to provide an anode chamber, a cathode parallel to the other diaphragm and spaced therefrom to provide a cathode chamber, a cathode reservoir, means for supplying anode liquor to the anode chamber in a continuous stream, means for supplying cathode liquor to the cathode chamber and reservoir, means for flowing cathode liquor through the cathode chamber and for circulating the cathode liquor between the cathode chamber and cathode reservoir.

4. An electrolyzer comprising a plurality of units having diaphragms interposed therebetween, a press for holding said units in position, insulation members separating the end units from electrical contact with the press, said units each comprising an anode plate slightly spaced from and parallel to a diaphragm to form a separate anode chamber therebetween, a cathode plate slightly spaced from and parallel to the preceding diaphragm to form a separate cathode chamber therebetween, means for supplying anode liquor to each anode chamber in a continuous stream, means for carrying away the overflow from each anode chamber, means for supplying cathode liquor to each cathode chamber, and independent means for each unit for circulating cathode liquor through the cathode chamber to keep the walls thereof free from incrustation.

5. An electrolyzer of the character described comprising a plurality of units clamped together and separated by diaphragms, a press for holding said units in clamped position, insulation members separating the end units from electrical contact with the press, each unit comprising an open-sided frame flanged at its sides to provide bearing surfaces for the diaphragms, and means independent of said press for holding the units and diaphragms in clamped position when the pressure of the press is released.

6. An electrolyzer comprising a plurality of units clamped side by side and separated by diaphragms to form a cell, each unit comprising an open-sided frame having its sides closed by the diaphragms, an anode plate in electrical contact with a frame, spaced from and disposed parallel to a diaphragm, a cathode plate on the opposite side of the diaphragm, in electrical contact with an adjacent frame, spaced from and disposed parallel to said diaphragm, reinforcing ribs interposed between said cathode plate and diaphragm against which said diaphragm bears, forming a cathode chamber between the cathode plate and diaphragm, and reinforcing ribs interposed between said anode plate and diaphragm against which said diaphragm bears on its opposite side, forming an anode chamber between the anode plate and diaphragm.

7. An electrolyzer of the character described comprising a plurality of units clamped side by side and separated by diaphragms, each unit comprising an open-sided frame flanged at its sides to provide a bearing surface for the diaphragms, a cathode plate spaced from one diaphragm to form a cathode compartment therebetween, an anode plate parallel to and spaced from the other diaphragm to form an anode compartment therebetween, a pump clamped to the unit, a pump discharge passage communicating with the lower part of the cathode compartment, and a pump supply passage communicating with the upper part of the cathode compartment.

8. An electrolyzer comprising a plurality of units clamped side by side and separated by diaphragms, each interior unit comprising an open-sided frame flanged at its sides to provide bearing faces for the diaphragms, a partition spaced from the diaphragms, dividing the interior of the frame into an upper and a lower compartment, a plate forming an anode completely closing one side of the upper compartment and spaced from the diaphragm to form an anode compartment therebetween which communicates at its bottom with the lower compartment, auxiliary bearing means interposed between the diaphragm and anode plate to provide auxiliary support for the diaphragm, a cathode plate on the opposite side of said unit, said cathode plate being spaced from and disposed parallel to the adjacent diaphragm to form a cathode chamber which communicates at the bottom with said lower compartment auxiliary bearing means interposed between the latter diaphragm and cathode plates to provide auxiliary support for the latter diaphragm, and a partition dividing said lower compartment into separate lower anode and lower cathode compartments.

9. An electrolyzer of the character described comprising a plurality of units clamped side by side, and separated by diaphragms, each unit comprising an open-sided frame having a partition spaced from the diaphragms and dividing the interior of the frame into an upper compartment and a lower compartment, a wall forming an anode completely closing one side of the upper compartment and spaced from one of the diaphragms to form an anode chamber therebetween which communicates at its bottom with the lower compartment, a second wall forming a cathode blocking the other side of the upper compartment, spaced from the anode to form a cathode reservoir therebetween, and spaced from the other diaphragm to form a cathode chamber therebetween, the cathode chamber communicating at its bottom with the lower compartment and communicating at the top with the cathode reservoir, a passage communicating with the cathode reservoir, a partition dividing the lower compartment into a lower anode compartment and a separate lower cathode compartment, a passage communicating with the lower cathode compartment, means for supplying anode liquor to the anode compartment, means for supplying cathode liquor to the cathode compartment, and means for circulating the cathode liquor through the lower cathode compartment, cathode chamber and cathode reservoir.

10. An electrolyzer comprising an anode chamber, a cathode chamber, a diaphragm between said chambers, a cathode spaced from said diaphragm forming a passage therebetween, a wall dividing said cathode chamber into two compartments, and means for circulating cathode liquor from one compartment through said passage and into the other compartment.

11. An electrolyzer comprising an open-sided frame having its sides closed by diaphragms, an anode parallel to the one diaphragm and spaced therefrom to provide an anode chamber, a cathode parallel to the other diaphragm and spaced therefrom to provide a cathode chamber, a cathode reservoir, and means for circulating cathode liquor through the cathode chamber between the cathode and the diaphragm and into the cathode reservoir.

12. An electrolyzer comprising an open-sided frame having its sides closed by diaphragms, an anode parallel to the one diaphragm and spaced therefrom to provide an anode chamber, a cathode parallel to the other diaphragm and spaced therefrom to provide a cathode chamber, a web dividing the space between the anode and the cathode into an upper and a lower cathode compartment, and means for circulating cathode liquor from the lower cathode compartment through the cathode chamber and into the upper cathode compartment.

13. An electrolyzer of the character described, comprising a plurality of units clamped together and separated by diaphragms, a press for holding the said units in clamped position, and means independent of said press and carried by said units for holding the units and diaphragms in clamped position when the pressure of the press is released.

14. An electrolyzer of the character described comprising a plurality of units clamped together and separated by diaphragms, a press for holding said units in clamped position, insulation members separating the end units from electrical contact with the press, each unit comprising an open-sided frame having diaphragms positioned at and closing its sides, and means independent of said press carried by said units for holding the units and diaphragms in clamped position when the pressure of the press is released.

15. An electrolyzer of the character described comprising a plurality of units clamped side by side and separated by diaphragms, each unit comprising an open-sided frame flanged at its sides to provide a bearing surface for the diaphragms, a cathode plate spaced from one diaphragm to form a cathode compartment therebetween, an anode plate parallel to and spaced from the other diaphragm to form an anode compartment therebetween, a web dividing the space between the cathode plate and the anode plate into an upper and a lower cathode compartment, and means for passing cathode liquor from the lower compartment through the cathode chamber and into the upper cathode compartment.

16. An electrolyzer comprising a plurality of units clamped side by side and separated by diaphragms, each interior unit comprising an open-sided frame, a partition spaced from the diaphragms dividing the interior of the frame into an upper and a lower compartment, a partition dividing said lower compartment into separate lower anode and lower cathode compartments, a plate forming an anode completely closing one side of the upper compartment and spaced from the diaphragm to form an anode compartment therebetween which communicates at its bottom with the lower anode compartment and a cathode plate on the opposite side of said unit, said cathode plate being spaced from the adjacent diaphragm to form a cathode chamber which communicates at the bottom with said lower cathode compartment.

17. In the electrolytic reduction of nitrobenzene, wherein the reduction product is obtained in the form of a solid precipitated from the cathode liquor, the improvement which comprises flowing the cathode liquor in a stream through the space formed between the cathode and the diaphragm to keep the surfaces of the cathode and diaphragm free from incrustations of said precipitated solid.

JOSEPH E. JEWETT.