Feb. 16, 1960 C. E. VAN WINCKEL ET AL 2,925,371

ELECTROLYTIC CELL

Filed June 1, 1956 3 Sheets-Sheet 1

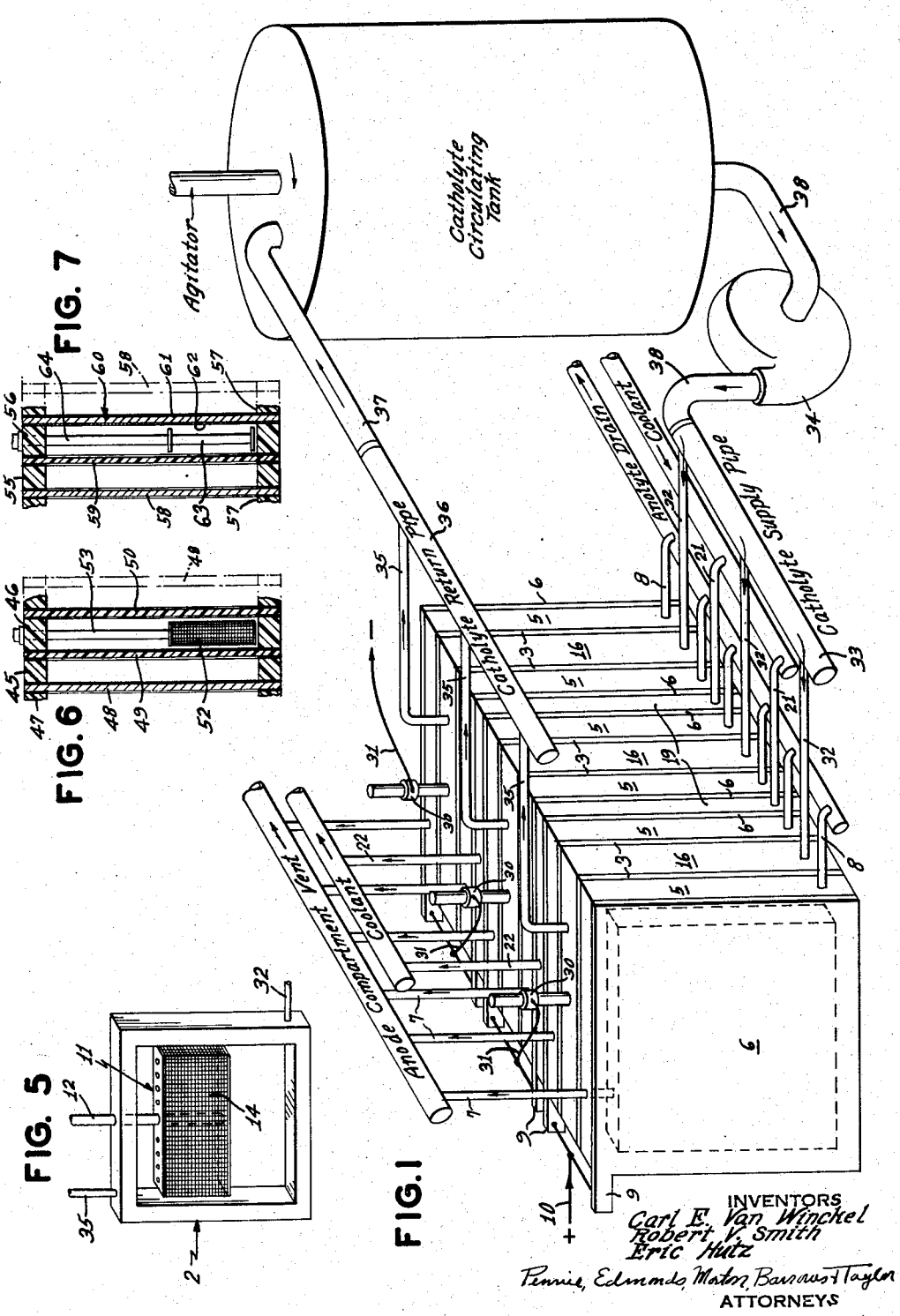


FIG. 1

FIG. 6

FIG. 7

FIG. 5

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ELECTROLYTIC CELL

Filed June 1, 1956 3 Sheets-Sheet 2

FIG. 3

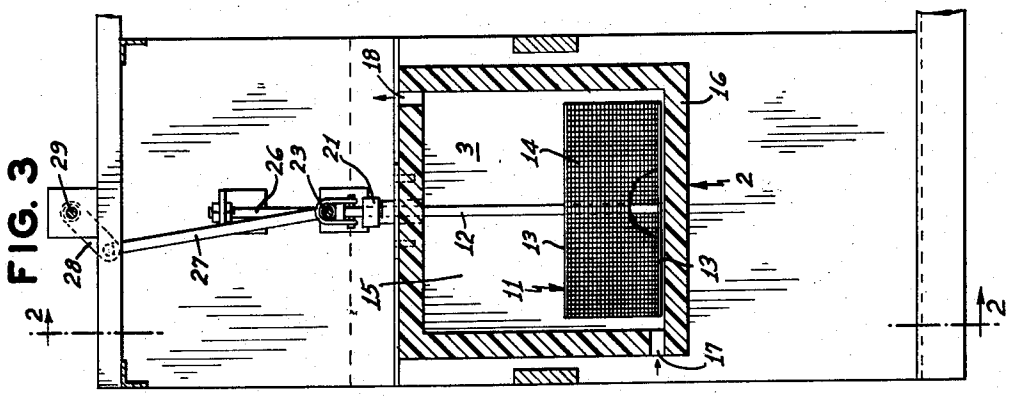


FIG. 2

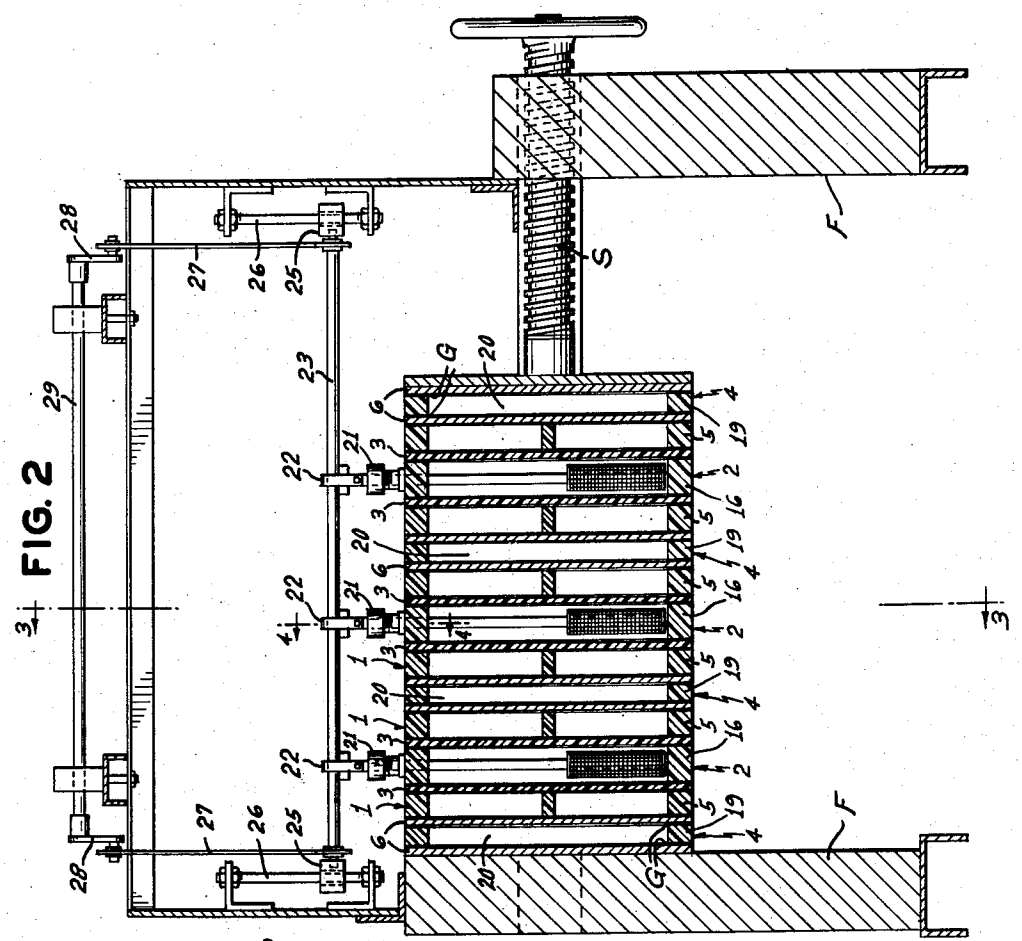
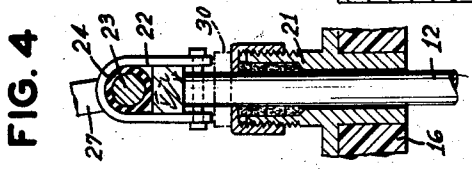


FIG. 4



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ELECTROLYTIC CELL

Filed June 1, 1956 3 Sheets-Sheet 3

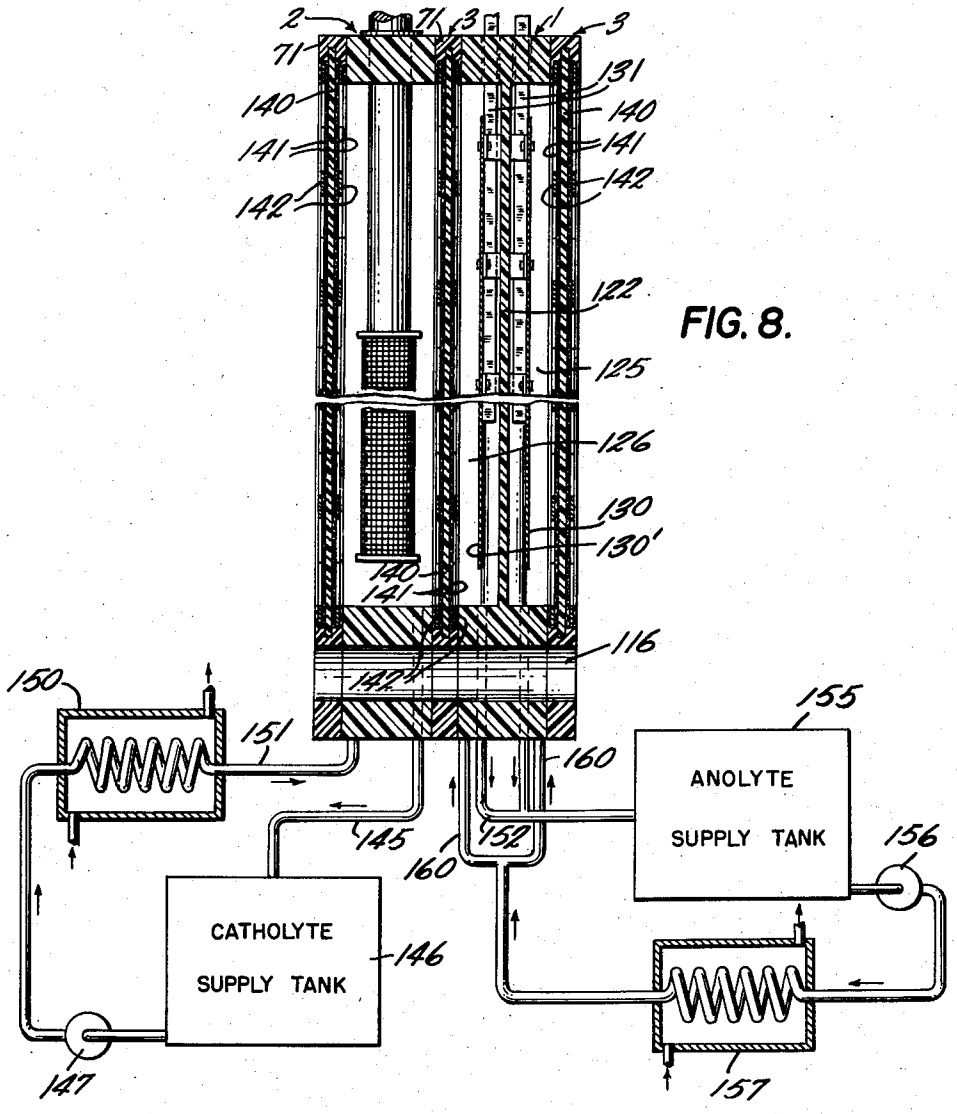


FIG. 8.

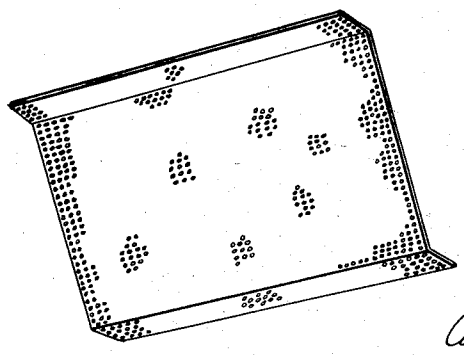


FIG. 9.

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2,925,371

ELECTROLYTIC CELL

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Application June 1, 1956, Serial No. 588,926

5 Claims. (Cl. 204—222)

This invention is directed to a method and apparatus for carrying out electrolytic processes and especially the electrolysis of organic compounds which are in suspension in the electrolyte. This application is a continuation-in-part of the copending application of Van Winckel, Smith and Hutz, Serial No. 253,896, filed October 30, 1951, and now abandoned for "Reciprocating Cathode for Electrolytic Reduction of Organic Nitrogen Compounds" in which there is shown and described an apparatus and method by means of which electrolytic processes may be practiced.

The electrolytic treatment of organic compounds presents serious difficulties which have not submitted to an easy solution. It has become apparent that successful operation is dependent upon the control of variables and upon structural features of the electrolytic cell that have not been clearly understood nor disclosed in the prior art.

One object of the invention is to provide improvements in electrolyzing dispersions of insoluble organic compounds with increased yields of desired products and with reduced yields of less desired by-products.

Yet another object of the invention is to provide improvements by means of which organic compounds may be treated electrolytically without generating conditions that tend to inhibit successful treatment of the compound or to cause deterioration of the electrolytic cell structure.

Still another object of the invention is to provide improvements in cooling the anolyte with a view to increasing the efficiency of the electrolytic operation.

Another object lies in improvements that will provide simultaneously the many conditions that are necessary for the efficient reduction of organic nitro compounds to form useful products.

According to the invention, reducible insoluble organic compounds, such as organic nitro compounds, are subjected to electrolysis in an electrolytic cell comprising anode and cathode chambers and intervening semi-permeable diaphragms by providing a cathode in the cathode chamber comprising a supporting framework and a screen supported by said framework having a mesh size in the range from about 20 to about 120 and by maintaining in the cathode chamber an aqueous catholyte comprising a suspension of such reducible organic compound and agitating the catholyte in the cathode chamber so as to maintain said organic compound dispersed in the catholyte and mix the dispersed material uniformly into all portions of the cathode chamber. In this way, the need for emulsifying agents, dispersing agents or solubilizing agents (referred to herein generically as "solubilizing agents") which have heretofore been considered to be necessary in reactions of this type, is avoided. Without such solubilizing agents increased yields of the desired products and reduced yields of less desired by-products can generally be obtained. The supporting frame of the cathode which is of stainless steel or other material that is refractory to the catholyte, is preferably connected to means for moving the cathode continuously during operation, preferably to effect a reciprocable up and down

movement within the cathode compartment to agitate the catholyte. This movement of the cathode gives improved results in treating catholytes which do not contain solubilizing agents because it effects an efficient dispersing action on the insoluble organic material in the catholyte, and an efficient mixing of the dispersed material uniformly into all portions of the cathode chamber. For example, in the electrolytic reduction of orthonitrochlorobenzene in caustic solution, it is efficient in both dispersing the orthonitrochlorobenzene as small droplets in the caustic solution, and in mixing the dispersion in the cathode chamber.

The cathodes are located within a cathode compartment formed within a surrounding frame of inert nonconducting material such as furfuryl alcohol resin, having at least one side closed by a diaphragm and the opposite side closed by another diaphragm. In a preferred form the cathode is reciprocated within the cathode compartment in which the catholyte is circulated. The cathodes are preferably entirely within the cathode compartments and supported on rods which are slidable in stuffing boxes where the rods enter the cathode compartment frames, and the rods are connected to any suitable operating structure for reciprocating the cathodes with the required stroke and frequency. The cathode compartment of each unit may be continuously supplied with fresh catholyte, or they may be supplied with a circulating batch of catholyte which is treated until the practical end of the reaction, at which time the batch is removed and replaced with a fresh batch.

The diaphragm may be advantageously formed of fibrous material bonded with furfuryl alcohol resin or other resin as described and claimed in the copending application, Serial No. 405,038, filed January 19, 1954. The diaphragms are preferably formed as sheets or slabs and are interposed between the anodes and cathodes, thereby providing one side for the cathode compartment and one side for the anode compartment. The diaphragms do not exhibit exceptionally high potentials and do not permit migration of organic material into the anolyte. The diffusion of electrolyte is at a minimum and the composition of the diaphragm has satisfactory stability.

The improved complete anode assembly of the invention comprises a surrounding frame, preferably rectangular, formed of inert nonconducting material such as furfuryl alcohol resin preferably having the same exterior dimensions as the cathode compartment frame, with a semi-permeable diaphragm on one side and an anodic metal plate or an impermeable diaphragm on the other side enclosing an open center or compartment for the anolyte. The anodic metal plate is advantageously made of a metal having a high resistance to corrosion even at high current densities, such as the alloy Inconel or stainless steel for alkaline anolytes, and stainless steel or other metals for acidic anolytes. The diaphragms are secured in fluid tight relationship to each side of the anode frame, thereby to define between the diaphragms an anode chamber having an anodic plate mounted therein. The chamber also is provided with means for adding an anolyte to and withdrawing anolyte from anodic chamber.

It has been found that the temperature in the cathode chamber does not submit itself to easy regulation merely by exterior cooling of the circulated catholyte because of the liberation of heat in the cathode chamber at a high rate and the generation of heat due to electrical resistance, particularly in the diaphragm. It is not feasible to extract all of the heat necessary to maintain the desired temperature in the cathode chamber. In operating the electrolytic cell according to a preferred procedure of the invention, the heat which is generated in the cathode by the resistance and chemical reaction is removed from

the cathode in a novel way thereby to maintain the cathode chamber temperature within the range which will produce the desired chemical reaction and avoid undesirable side reactions as heretofore indicated. That is, the anolyte is withdrawn from the anolyte chamber, cooled, and thereafter returned to the anolyte chamber, thereby to maintain the anolyte chamber at a temperature substantially below that of the catholyte. The surface to volume relations of the cathode chamber are so high and hence the potential heat interchangeable between the anolyte and the catholyte through the diaphragm are such that the temperature of the catholyte in the cathode chamber may be controlled by circulating the anolyte through the anode chamber as described, and cooling the anolyte before it is returned to the anode chamber.

Certain features of the invention including the procedural features and the manner of cooling the anolyte will be better understood after considering the following description taken in conjunction with the accompanying drawings.

Figure 1 is a perspective view, with parts omitted, of a electrolytic cell and liquid supply means embodying one form of the invention for the electrolysis of organic compounds.

Figure 2 is a sectional side view at 2—2 of Figure 3.

Figure 3 is a sectional view at 3—3 of Figure 2.

Figure 4 is an enlarged sectional view at 4—4 of Figure 2.

Figure 5 is a perspective view of a cathode compartment frame and cathode of the invention.

Figures 6 and 7 are sectional side views of other embodiments of cell units of the invention.

Figure 8 is a partially schematic illustration of the assembled cathode, diaphragms and anode of the invention with the apparatus which is used in circulating and cooling the anolyte and catholyte.

Figure 9 is a detailed view of a perforated plate which may be used on the cathode instead of a screen.

The electrolytic cell unit illustrated in Figs. 1 to 5 comprises several cells each consisting of an anode and anode compartment **1** on each side of a cathode and cathode compartment **2**, with a diaphragm **3** between the cathode and anode compartments and a coolant chamber **4** between the adjacent anodes. Each member of the unit has the same rectangular cross-sectional shape and dimensions so that the cells may be arranged in series one after the other in any desired number in the complete cell unit.

Each anode compartment comprises a rectangular frame **5** formed of furfuryl alcohol resin and a flat anodic plate **6** which is preferably formed of the alloy Inconel, a vent connection **7** through the top of the frame and an anolyte drain connection **8** at the bottom of the frame which connects to the anolyte drain pipe. The anolyte may be under such a pressure that it stands in pipes **7** up almost to the anode compartment vent pipe. We prefer to construct tubes **7**, **8** and the anolyte drain of a nonconducting material such as furfuryl alcohol resin tubing or pipes. The projecting lugs **9** include any suitable means for connecting the anodic plates to the electrical circuit **10**.

The diaphragms **3** are preferably formed as a flat sheet consisting of fibrous material bonded with furfuryl alcohol resin and other resin, such as described more fully and claimed in copending application Serial No. 405,038, filed January 19, 1954, and now abandoned.

The cathodes **11** of the combinations **2** comprise operating and supporting rods **12** to which are permanently attached perforated spaced plates **13** which provide a support for the covering **14** of stainless steel wire cloth which covers both ends and both sides. Perforated end plates may be used for the ends with the screen just covering the large opposite sides. The rods **12** and all parts of the cathode in contact with the catholyte are preferably of stainless steel or other conductive material that is refractory to the catholyte. The cathode is supported within the space **15** inside the cathode compartment frame **16**, which is mounted between adjacent diaphragms **3**. The frame **16** is provided with an inlet connection **17** for the supply of catholyte and an outlet connection **18** for the removal of catholyte from the space **15**. It will be noted that the cathode is arranged to reciprocate while being entirely immersed in the catholyte.

The coolant members **4** consist of rectangular frames **19**, also preferably formed of furfuryl alcohol resin, having interior spaces **20** for the circulation of the coolant which are closed off by the adjoining plates **6** of the anodes. The frames **19** have inlet pipe connections **21** at the bottoms and outlet pipe connections **22** at the tops for the circulation of any suitable nonconducting liquid such as lubricating oil from the coolant main.

The furfuryl alcohol resin frames are advantageously formed according to the method of copending application Serial No. 405,038, filed January 19, 1954, and are preferably filled with various materials, such as asbestos. The furfuryl alcohol resin of which this cell is largely constructed has unique properties for the purpose of this invention. It is a nonconductor and is resistant to non-oxidizing acids, alkalies and organic solvents at temperatures up to 120° C.

The assembly of a cell unit is arranged with the cells alongside of each other, repeated one after the other, and the entire assembly is held in a rigid horizontal position in a frame F which is similar to a filter press skeleton. Between the adjoining faces of the members, any suitable gasket, such as asbestos tape G, is interposed and the screw means S presses the members into fluid-tight contact with the gaskets, thereby sealing the compartments from each other. Instead of a gasket, we may use a sealing resin or inert plastic material.

The cathode rods **12** pass through stuffing boxes **21'** and are connected by stirrups **22'** to the lift rod **23**. A layer of electrical insulation **24** is interposed between the stirrups and the rod **23**. The bearings **25** guide the rod **23** in its up-and-down movement on the guide rods **26**. The rod **23** is operated by the links **27**, crank arms **28** and rotatably driven shaft **29**. The cathodes **11** are accordingly reciprocated in an up-and-down movement at any desired stroke and frequency.

At any convenient places on the rods **12** conductor clamps **30** are attached for connection to the flexible electrical conductors **31** which lead to the anode conductors **10** of an adjacent cell.

In the more complete embodiment of apparatus shown somewhat diagrammatically in Figure 1, the catholyte inlet connections **17** are connected by short sections of nonconducting tubing such as furfuryl alcohol resin tubing or hose **32** to the catholyte supply pipe **33** which is supplied from the tank by the centrifugal pump **34**. The catholyte circulates through the cathode compartments and leaves through the furfuryl alcohol resin tube sections **35** which connect the outlet connections **18** to the return main **36**. The catholyte in the tank is preferably agitated with any suitable device and cooled by means of a heat exchanger if desired. The anolyte drain pipes **8**, the catholyte supply pipes **32**, and outlet pipes **35** are preferably of such small diameter, around ½ to 1 inch, and such length, around 2 to 4 feet, that the catholyte therein offers such high resistance that there is practically no current flowing in the anolyte drain and catholyte supply and return pipes. The catholyte circulating supply tank and the sections of pipes **37** and **38**, as well as the pump **34**, are also formed of stainless steel.

The alloy Inconel is especially suitable for the anodic plates. It showed no corrosive action when used for 250 ampere hours at a current density of 21 amps./dm.^{2}. It has also shown no attack when used re-

peatedly in a cell of our invention operating at approximately 14 amps./dm.^{2}.

The diaphragm **3** which is indicated generally in Figure 2 is formed so that it will transmit ions without permitting the passage of particles which are dispersed but not dissolved, e.g., insoluble organic compounds of the type which may be reduced by electro-chemical methods. One type of diaphragm in its frame is shown in detail in Figures 12 and 13 of the copending application Serial No. 365,134, filed June 30, 1953, and is described in Figure 8 of the present application. The diaphragm assembly comprises a molded frame **71**, a centrally disposed semipermeable diaphragm **140**, a screen **141** on each side of the diaphragm and a sheet of expanded metal **142** on the exterior side of each screen.

The electrolytic reaction may be carried out by retaining all of the catholyte in the cathode chamber until operation is complete and then draining the reaction product from the chamber. However, it is preferred to circulate the catholyte through the cathode chamber during the operation and for this purpose there is provided in Figure 8 a pipe **145** of nonconducting material leading to a catholyte supply tank **146**. From the supply tank, catholyte is withdrawn through a pump **147** thence through a cooler or heat interchanger **150**, and back to the cathode chamber through another nonconducting pipe **151**.

The anolyte in the anode chamber is also withdrawn through the pipes **152** which are formed of nonconducting material to an anolyte supply tank **155**. From the tank **155** the anolyte passes through the pump **156**, the cooler or heat interchanger **157** and back to the anode chambers through the pipes **160**, also made of nonconducting material.

The anode frame and chamber shown in Figure 8 includes a centrally disposed liquid impervious web **122** which forms two anode chambers **125** and **126**, one on each side of the web. Each chamber has an anode plate **130** or **130'** mounted therein and secured to the web **122**. The conductor bars **131** supply the electric current to the anode plates. The manifolds **116** may be used to supply anolyte or catholyte to the respective compartments, through connecting piping not shown. Another cathode chamber not shown may be provided to the right of the anode chamber and diaphragm shown in Figure 8, and another anode chamber to the left of the cathode chamber and diaphragm thereof.

As heretofore indicated a serious problem in carrying out electrolytic processes is the control of the temperature to produce the desired product. Since the amount of the liquid in the cathode chamber is small and the amount of heat generated relatively large, temperature control is difficult. It is an outstanding feature of the present invention that an improved way is provided to control the temperature of the catholyte in the cathode chamber in the operation of an electrolytic cell. This is accomplished by providing an electrolytic cell having a cathode and an anode in which the catholyte and anolyte are in direct contact with opposite sides of a semipermeable diaphragm and recirculating and cooling the anolyte, as for example, in the cooler or heat exchanger **155** shown in Figure 8. In this way advantage is taken of the relatively large area-to-volume relation between the diaphragm and the volume of the cathode chamber to transmit heat through the diaphragm from the catholyte to the anolyte, thereby maintaining temperatures in the cathode chamber which are more nearly uniform and within the desired range for optimum control of the electrolytic reaction.

An electrolytic cell of the invention especially suitable for the reduction of orthonitrochlorobenzene has cell units of the approximate following dimensions:

Overall outside width 12 to 30 inches (inside 10 to 26 inches)
Overall outside height 12 to 30 inches (inside 10 to 26 inches)
Anode frame about 1 to 2 inches thick
Cathode compartment frame about 1½ to 3 inches thick
Coolant compartment frame about ¾ to 1½ inches thick
Diaphragm about ¼ to ½ inch thick
Anodic plate about ¼ to ½ inch thick

It is to be understood that these are not limiting specifications since the cell and cathode can be of any suitable dimensions within the inventive concept described herein. In a cell having outside cross-sectional dimensions of 12 by 12 inches, the cathode compartment has an inside square dimension of 10 inches and the cathode is 9½ inches wide by 4 inches high by 1½ inches thick and can be moved in the cathode chamber without contacting the compartment frame or the diaphragm. The modification of cell unit illustrated in Figure 6 comprises an anode compartment frame **45**, a cathode compartment frame **46**, and a coolant compartment frame **47**, all rectangular in cross section, and preferably formed of furfuryl alcohol resin. The frames are provided with inlet and outlet conections for the circulation of anolyte, catholyte, and coolant as in Figure 1. The anodic plate of Inconel **48** is sealed between frames **45** and **47** and the diaphragm **49** is sealed between the frames **45** and **46**. The plate **50** of glass or stainless steel is just an inert wall member which closes one side of the coolant compartment on one side of the anodic plate **48**. We omit the coolant compartment and form the anode of a coil of Inconel tubing and cool the anode by circulating the coolant therein. We may also eliminate the coolant members entirely and circulate the anolyte to a heat exchanger as described above. The cathode **52** is reciprocably mounted on the rod **53** in a manner similar to the cathode of Figure 1.

The modification of cell unit illustrated in Figure 7 comprises anode compartment frame **55**, cathode compartment frame **56**, and coolant compartment frame **57**, all rectangular in cross-section and formed preferably of furfuryl alcohol resin. The anodic plate **58** is preferably formed of Inconel and is sealed between the frames **55** and **57**. The diaphragm **59** is sealed between the frames **55** and **56** and the cathodic plate or active cathode **60** is sealed between the frames **56** and **57'**. The cathode comprises a stainless steel plate **61** which seals the cathode compartment from the coolant compartment and an active cathode member **62** of stainless steel mesh. The catholyte is agitated with the stainless steel agitator **63** which is moved up and down by the rod **64**. The anode, cathode, and when provided the coolant compartments have inlet and outlet means, as in Figure 1, for the circulation of anolyte, catholyte, and coolant.

The diaphragms of Figures 6 and 7 are preferably made of granular furfuryl resin, as previously described, and the units may be arranged in a frame as in Figure 1.

The cathodes of the invention are preferably of stainsteel such as 18–8 low carbon steel and the cathodic surfaces may be coated with an even layer of sponge lead, electrolytically deposited on the stainless steel. As stated above the cathodic element comprises a stainless steel screen in the mesh range of 20 to 120. Screens having mesh size in the range 80 to 120 are preferred because generally they bring about reduction of nitro compounds at high current efficiencies. Meshes lower than 80 gradually decrease in their ability to produce complete reduction to the hydrazo compound as evidenced by increasing amounts of azoxy and azo intermediate isolated from the product.

By the addition of small amounts of lead, i.e., 25 mg./dm.^{2} of cathode screen area, in the form of lead monoxide, to the catholyte prior to the addition of the nitro compound, or by coating the cathode screen with the sponge lead, or by adding lead to the cathode in other

ways, an improvement in reducing the nitro compounds to the hydrazo compound may be realized.

The cell is compact and makes possible the use of a comparatively high voltage source of current due to the large number of cells that may be connected in series. In fact several cell units may be connected in series to utilize even higher voltages. The construction of the cell also makes possible better and more uniform mixing of the charge and provides a ready means of controlling the temperature and of charging and discharging the cells without an undue loss of current efficiency. Other important features are the low resistance per cell, comparatively cheap construction, ease of maintenance, and long life. Moreover, the rectangular cathodes, operable in rectangular cathode compartments, with means for reciprocation in an up-and-down movement permits the use of an elongated narrow cell unit which may be employed in banks of 10 to 12 cell units, each connected in series.

One important feature of the invention is the adaptability of the units to operation in a series circuit. In connecting the individual cells in series it is possible to use high voltage current, the voltage across each cell being from 3.5 to 5 volts. Several units, each having several cells may be connected in series giving the advantage of using relatively high generator voltage. It is advantageous to connect the anodes on each side of a cathode in parallel and the cathode in series to the next pair of anodes and so on through the series. In this way the electron movement is from a cathode toward the adjacent side of each anode.

Example I

The following is an example of an operation carried out in 30 inch cells of the type described in Figures 6 and 8 which are provided with reciprocating cathodes having 80 mesh stainless steel screen. The ratio of the effective cathode chamber volume to the cathode screen surface area was 684 cc. per square decimeter. The catholyte solution was prepared by mixing orthonitrochlorobenzene with 10% aqueous sodium hydroxide in the ratio of one part of the nitro compound to five parts of the aqueous caustic. The internal temperature in the electrolytic cells was raised to about 90° C. (by circulating and heating the anolyte and catholyte in the heat exchangers), and the organic matter was dispersed in the catholyte by reciprocating the cathodes at the rate of about 150 cycles per minute in strokes of about 4 inches. Direct current was applied across the unit of cells connected in series and the voltage was controlled to provide a current density of 11.7 amp. per square decimeter of cathode screen area per cell. The temperature in the cathode chamber during the reaction was maintained at 90° C. by circulating and cooling the anolyte and catholyte in the external heat exchangers shown in Figure 8. After 104% of the theoretical current which is required to reduce the orthonitrochlorobenzene to o,o'-dichlorohydrazobenzene had passed through the cell, the catholyte which was then colorless, was discharged from the cells and the o,o'-dichlorohydrazobenzene was recovered by solvent extraction. The byproducts orthochloroaniline and orthodichlorobenzene were also recovered. The analysis showed that the following percentages of the orthonitrochlorobenzene were converted to the materials designated:

	Percent
To o,o'-dichlorohydrazobenzene	80.8
To o-chloroaniline	5.7
To o,o'-dichloroazobenzene	13.5

Example II

The following is an example of the operation carried out in the apparatus of Figure 1. The anode compartments of the cells are charged with 30% aqueous caustic soda solution. Next 7,000 parts of 10% caustic soda solution is placed in the steel catholyte circulating tank and is heated to 90° C. Circulation and agitation is then started and continued until the cell is heated as indicated by the temperature of the returning solution. 1000 parts of orthonitrochlorobenzene is then added to the catholyte circulating tank and the current is passed through the cells at a density of 4 amps./dm.^{2}. It is desirable to control the ratio of nitro compound to caustic solution within certain limits. For best results a ratio of nitro compound to 10% caustic solution of not greater than about 1:5 is used. Although the lower limit of this ratio is not as critical, ratios of at least about 1:15 are preferred. The temperature during the reduction is maintained at 90° by cooling both the anode compartment and the catholyte in the catholyte tank. When the reduction is about 75% complete, hydrogen may be evolved and the current can be reduced, if desired, to prevent excessive hydrogen evolution and to obtain greater current efficiency. When the reaction is complete as indicated by the rate of evolution of hydrogen, the passage of about 110% of the theoretical current and the color change from orange to yellow, the current is shut off and the cells drained into the catholyte tank. Agitation is stopped and the molten hydrazobenzene or a derivative thereof is allowed to settle and is then drawn off. The remaining caustic solution may be adjusted as to strength and reused or discarded.

Example III

The procedure described in the preceding examples was repeated using a three-cell unit, of the type described in Figures 1 to 5 inclusive, each cathode chamber (10" x 10" internal dimensions) having a reciprocating cathode (about 140 strokes per minute). Cathodes were provided with 80 mesh stainless steel screen. The effective volume of the cathode chamber to the cathode screen area was 475 cc. per square decimeter. The orthonitrochlorobenzene was mixed with the aqueous caustic in the ratio of 1:5.5, the amount of catholyte provided was such that the ratio of catholyte in the cathode chambers to the amount of catholyte in the external circulating system was 3:1. The current density was 11.2 amp. per square decimeter of cathode screen area. After 132% of the amount of current theoretically necessary to reduce all of the nitrochlorobenzene to dichlorohydrazobenzene had been passed through the cells, the operation was discontinued and the catholyte analyzed for materials described in Example I. It was determined that the following amounts of orthonitrochlorobenzene were converted to the materials designated:

	Percent
To o,o'-dichlorohydrazobenzene	80.7
To o-chloroaniline	10.9
To o,o'-dichloroazobenzene	8.4

Following are examples of other materials that have been successfully reduced using the procedure and apparatus described in Example I:

Reactant	Catholyte	Product
Nitrobenzene	Caustic	Hydrazobenzene.
o-Nitrotoluene	Caustic	Azoxytoluene.
o-Nitroanisole	Caustic	Hydrazoanisole.
Nitrobenzene	20% H_2SO_4*	p-aminophenol.

*80 mesh Monel screen cathode.

Although the best mode of carrying out the invention and the preferred electrolytic cells has been described, there are many modifications thereto within the scope of the invention. Instead of screens, perforated plates as shown in Figure 9 having the same number of openings and total weight per square inch as the screens may be used. Accordingly, it is intended that such modifica-

tions and equivalents be included within the scope of the appended claims.

We claim:

1. Electrolytic apparatus comprising an upright, generally rectangular hollow cell frame having an axis perpendicular to the plane of the rectangle and at least one open side of relatively large dimension as compared with the axial frame thickness, said frame enclosing a cell chamber for containing a catholyte comprising an aqueous suspension of a reducible, organic compound that is insoluble in the aqueous suspension, an electrode housed within said cell chamber mounted for vertical reciprocation and attached to means for reciprocating said electrode in a direction perpendicular to said axis, a diaphragm secured at said open side of said frame in liquid-tight relationship with respect thereto, said electrode comprising a framework and a screen supported by said framework having a mesh size in the range from about 20 to 120, so that reciprocation of said electrode maintains said organic compound dispersed in the catholyte and mixes the dispersed material uniformly into all portions of said cathode chamber.

2. The apparatus described in claim 1 wherein the mesh size of the screen is in the range from about 80 to 120.

3. The apparatus described in claim 1 comprising an anode cell frame enclosing an anode chamber on the side of said diaphragm opposite to said cathode chamber, said anode chamber containing liquid anolyte, means for withdrawing said anolyte from said anode chamber, means for cooling said anolyte externally of said anode chamber and means for returning said cooled anolyte to said anode chamber thereby to control the temperature of the catholyte in the cathode chamber by heat transfer to the anode chamber through the diaphragm.

4. The apparatus described in claim 3 including means for withdrawing the catholyte from the cathode chamber, means for cooling said withdrawn catholyte externally of the cathode chamber and means for returning the cooled catholyte to the cathode chamber.

5. The apparatus described in claim 1 in which the screen is stainless steel.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,925,371 February 16, 1960

Carl E. Van Winckel et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 65, for "and other" read -- or other --; column 6, line 23, for "conections" read -- connections --; line 30, before "omit" insert -- may --.

Signed and sealed this 9th day of August 1960.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents