E. A. SPERRY.

OSMOTIC DIAPHRAGM.

APPLICATION FILED DEC. 26, 1918.

1,368,227. Patented Feb. 8, 1921.

Fig. 1.

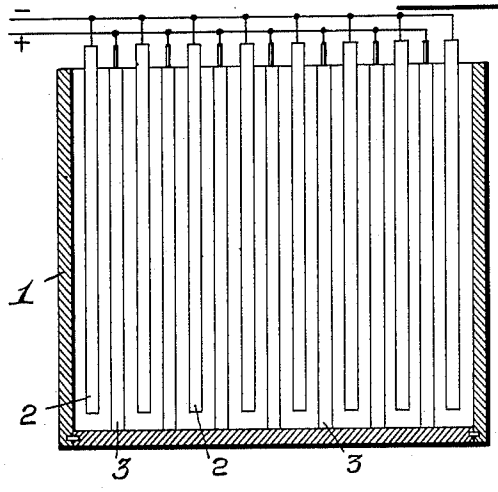


Fig. 2.

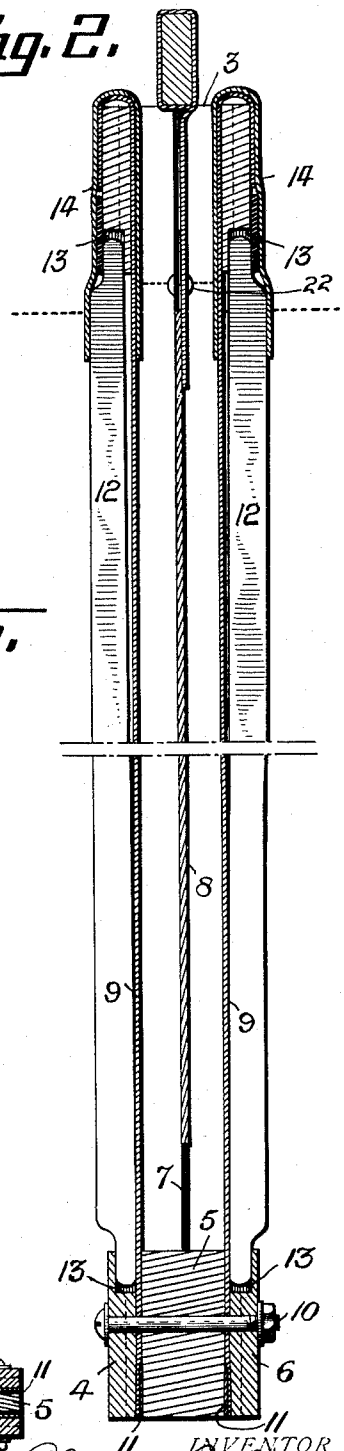


Fig. 3.

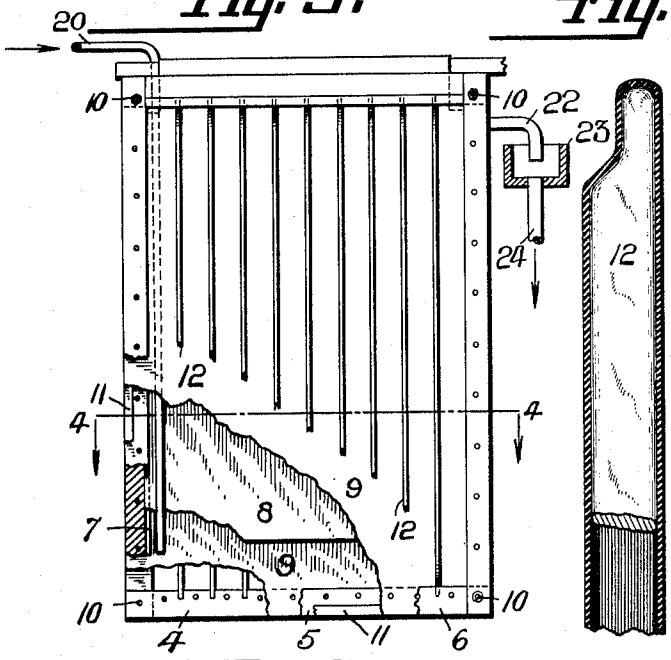
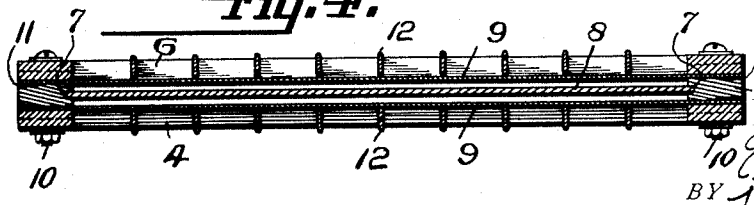


Fig. 5.

Fig. 4.



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UNITED STATES PATENT OFFICE.

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OSMOTIC DIAPHRAGM.

1,368,227. Specification of Letters Patent. Patented Feb. 8, 1921.

Application filed December 26, 1918. Serial No. 268,319.

To all whom it may concern:

Be it known that I, ELMER A. SPERRY, a citizen of the United States of America, residing at 1505 Albermarle road, Brooklyn, in the county of Kings and State of New York, have invented certain new and useful Improvements in Osmotic Diaphragms, of which the following is a specification.

This invention relates to porous diaphragms for use in bifluid cells. More specifically it relates, not only to such diaphragms *per se*, but to a method of making an apparatus for using the same. While such diaphragms are capable of wide application I will, to avoid surplusage, restrict the present disclosure principally to the application of the invention to apparatus designed for use in the method of manufacturing white lead, or other lead salts, by electrolysis.

Such a method is disclosed in the application for United States Letters Patent by Ralph M. Harrington, "Method of producing lead salts," Serial No. 193,415, filed September 27, 1917. This method involves the employment of a bifluid electrolytic cell in which the anolyte and catholyte are separated by a porous diaphragm or series of such diaphragms. In carrying out this process considerable difficulty has been experienced in obtaining diaphragms possessing the necessary mechanical strength, durability and the proper degree of porosity. Diaphragms consisting of paper, parchmentized or otherwise treated, have been tried as have also others consisting of cloth treated in various ways. None of these proved successful due to the fact that they not only lacked the desired mechanical strength but due also to the fact that the necessary osmotic, percolating, or combined characteristics were lacking. In fact until the present diaphragm was invented it was practically impossible to maintain a cell in uninterrupted operation for more than a few days. By employing the diaphragm hereinafter disclosed continuous operation of many weeks has been attained.

One of the primary objects of the present invention is to provide a durable diaphragm preferably osmotic having the proper qualities and possessing great mechanical strength.

Another object is to provide a method of making and using such diaphragms.

A further object is to provide an electrode compartment, comprising a plurality of such diaphragms, which permits ready removal and replacement of the diaphragms, which supports the latter in such a manner as to prevent rupture thereof and short-circuiting of the electrodes, and which supports one of the electrodes. Further objects and advantages will appear as the invention is hereinafter disclosed.

Referring to the drawings which illustrate what I now consider a preferred physical embodiment of one part of my invention:

Figure 1 is a somewhat diagrammatic sectional elevation of a bifluid electrolytic cell.

Fig. 2 is an enlarged sectional elevation of one of the electrode compartments shown in Fig. 1.

Fig. 3 is a side elevation, with parts broken away, of the compartment shown in Fig. 2.

Fig. 4 is an enlarged section taken on the line 4—4 of Fig. 3.

Fig. 5 is a fragmentary sectional detail of one of the retaining bars employed.

Referring to Fig. 1, it will be seen that I have shown an electrolytic cell comprising a tank or container 1 in which are mounted a plurality of spaced anode plates 2 between which are located a plurality of cathode compartments 3. A preferred form of such compartments is illustrated in Figs. 2, 3, and 4 and may be constructed substantially as follows. A plurality of clamping frames, preferably of wood or other insulating material, are provided the main frame 5 being U-shaped in form and provided with guide grooves 7 which the cathode 8 is adapted to enter. The diaphragms 9, 9 are each clamped between the main frame and one or the other of the side frames 4, 6. The latter are closed frames and are detachably secured to the main frame and to each other by means of bolts 10. In order to prevent leakage of liquid, cloth or other strips of compressible material 11 may be provided between the frames. Rods or retaining bars 12, adapted to seat in notches 13 provided in the upper and lower members of the side frames 4 and 6, may be provided to prevent rupture of the diaphragms due to the pres-

sure of the catholyte and to prevent contact between the diaphragms, the anode and cathode which would give rise to a short circuit. These bars may comprise an inner core of steel coated with a layer of hard rubber or other insulating material.

The diaphragms preferably consist of parchmentized cloth or fabric. One method of making one of such diaphragms, especially adapted for use in an electrolytic cell in the manufacture of white lead, is as follows. A piece of duck canvas of suitable size and preferably of comparatively fine weave is first freed of grease and other impurities. This may be accomplished by boiling it in a weak solution, say about one per cent. solution of sodium hydrate for about ten minutes. The fabric may then be washed, boiled in a weak solution of hydrochloric or other acid for about ten minutes, to neutralize the caustic solution, and then washed and dried. The fabric is now ready for parchmentizing which may be accomplished as follows.

The fabric is subjected to a strong sulfuric acid bath, of say 1.750 specific gravity, for a length of time depending on the osmotic and impervious properties desired. The extent of the anastomosing of the weave may be varied of course by changing the length of time of the bath or by otherwise varying the process. If it is desired to form an osmotic diaphragm for use in an electrolytic cell for the manufacture of white lead, thirty minutes, or from thirty to forty-five minutes, has been found satisfactory. The sulfuric acid converts the fabric (cellulose) beginning at the surface into amyloid which forms a gelatinous-like surface and cements the fibers together. After the sulfuric acid bath the fabric is placed immediately into water to wash off the excess acid and then subjected to a bath containing about five per cent. of sodium or ammonium hydroxid. The diaphragm is now ready to be mounted in the frames.

It has been found in practice that osmotic diaphragms made in accordance with the method set forth above, swell when moist, and tend to crack and tear when allowed to become dry, causing tearing of diaphragm away from the frames. It is, therefore, important that they be kept wet, moist or damp both before and after they are mounted on the frames. This may preferably be accomplished by keeping them in water until ready to be placed into the cells. When the diaphragms are in the cell the problem of preventing tearing and cracking is more difficult. If it were possible to keep the upper edge of the diaphragms submerged or flush with the surface of either or both of the electrolytes, said diaphragms would never become dry. It is, however, practically impossible to do this. I solve the problem by allowing the upper end of the diaphragms to project above the surface of the electrolyte, as shown in Fig. 2 in which the dotted line represents the electrolyte level, and by providing means to prevent the exposed or unimmersed portion from becoming dry. This means may assume various forms, one form being illustrated in Figs. 2 and 3.

The upper edge of each diaphragm is made sufficiently long to lap over the upper edge of the corresponding side frame 4 or 6. A strip of cloth 14, preferably having capillary or absorbent properties, is placed over each of said upper edges and may, if desirable, be stitched or otherwise secured to the corresponding diaphragm. These strips 14 should be of sufficient area to cover completely the unimmersed portions of the diaphragms. It will be obvious that if the cloth 14 is kept wet or moist, so also will the unimmersed portions of the diaphragms. One method of keeping the said cloth 14 moist is by applying glycerin to it. Another and preferable method is to allow the lower edge, or edges, of said cloth to project into one or both of the electrolytes and thus convey liquid to the unimmersed portions of the diaphragms 9 by capillary attraction. The fabric so used should, of course, be treated to prevent deterioration in the electrolyte used.

Any suitable form of means may be employed to maintain the catholyte, *i. e.* the liquid in the cathode compartment, at its predetermined level. For reasons which will be apparent on inspection of said prior application as well as the present application I prefer to employ the system shown somewhat diagrammatically in Fig. 3. The catholyte is continuously introduced into each cathode compartment by means of a long tube 20 which extends into and substantially to the bottom of said compartment. The latter is provided also with an overflow pipe 22 which empties into a trough 23 from which the catholyte is removed at 24. Obviously the liquid within the compartment will never rise above the level of the overflow tube 22.

In accordance with the provisions of the patent statutes, I have herein described the principle of operation of my invention, together with the apparatus, which I now consider to represent the best embodiment thereof, but I desire to have it understood that the apparatus shown is only illustrative and that the invention can be carried out by other means. Also, while it is designed to use the various features and elements in the combination and relations described, some of these may be altered and others omitted without interfering with the more general results outlined, and the invention extends to such use.

Having described my invention, what I

claim and desire to secure by Letters Patent is:

1. A porous diaphragm for use in a bifluid cell, said diaphragm consisting of cloth parchmentized to a predetermined extent.

2. An osmotic diaphragm for use in an electrolytic cell, said diaphragm consisting of cloth parchmentized to a predetermined extent.

3. In combination, a parchmentized diaphragm for a bifluid cell and means for preventing the unimmersed portion of said diaphragm from becoming dry.

4. An electrolytic cell comprising a parchmentized fibrous diaphragm and means for automatically conveying moisture to the unimmersed portion of said diaphragm to prevent the latter from becoming dry.

5. An electrolytic cell comprising a parchmentized diaphragm and means for automatically conveying electrolyte to the unimmersed portion of said diaphragm to prevent the latter from becoming dry.

6. An electrolytic cell comprising a parchmentized diaphragm and capillary means for automatically conveying electrolyte to the unimmersed portion of said diaphragm to prevent the latter from becoming dry.

7. An electrode compartment comprising a plurality of diaphragm-clamping frames and a plurality of retaining bars secured thereto across the open space thereof.

8. An electrode compartment comprising a main frame, a plurality of diaphragm-clamping frames, one for each side of said main frame and electrode-guide means on said main frame.

9. An electrode compartment comprising a main frame and side frames, a plurality of diaphragms each clamped between said main frame and a corresponding side frame and a plurality of retaining bars carried by each side frame between the latter and the corresponding diaphragm.

10. An electrode compartment comprising a main frame and side frames, a plurality of diaphragms each clamped between said main frame and a corresponding side frame, electrode guide means on said main frame, and a plurality of retaining bars carried by each side frame between the latter and the corresponding diaphragm.

11. An electrode compartment comprising a main frame, a plurality of diaphragm-clamping frames, one for each side of said main frame, electrode-guide means on said main frame, and a diaphragm supporting strip at the top of the frame.

12. An electrode compartment comprising a main frame, a plurality of diaphragm-clamping frames, one for each side of said main frame, electrode-guide means on said main frame, and a diaphragm supporting strip at the top of the frame on each side of the cathode.

13. In combination, a diaphragm for a bifluid cell, and a wick engaging said diaphragm adjacent its upper edge.

14. In combination, a diaphragm for a bifluid electrolytic cell comprising parchmentized cloth, and an absorbent material engaging said diaphragm adjacent one of its edges.

15. In combination, a diaphragm for a bifluid electrolytic cell, means for maintaining said diaphragm in stretched position, and absorbent material covering the upper portion of said diaphragm.

In testimony whereof I have affixed my signature.

ELMER A. SPERRY.