

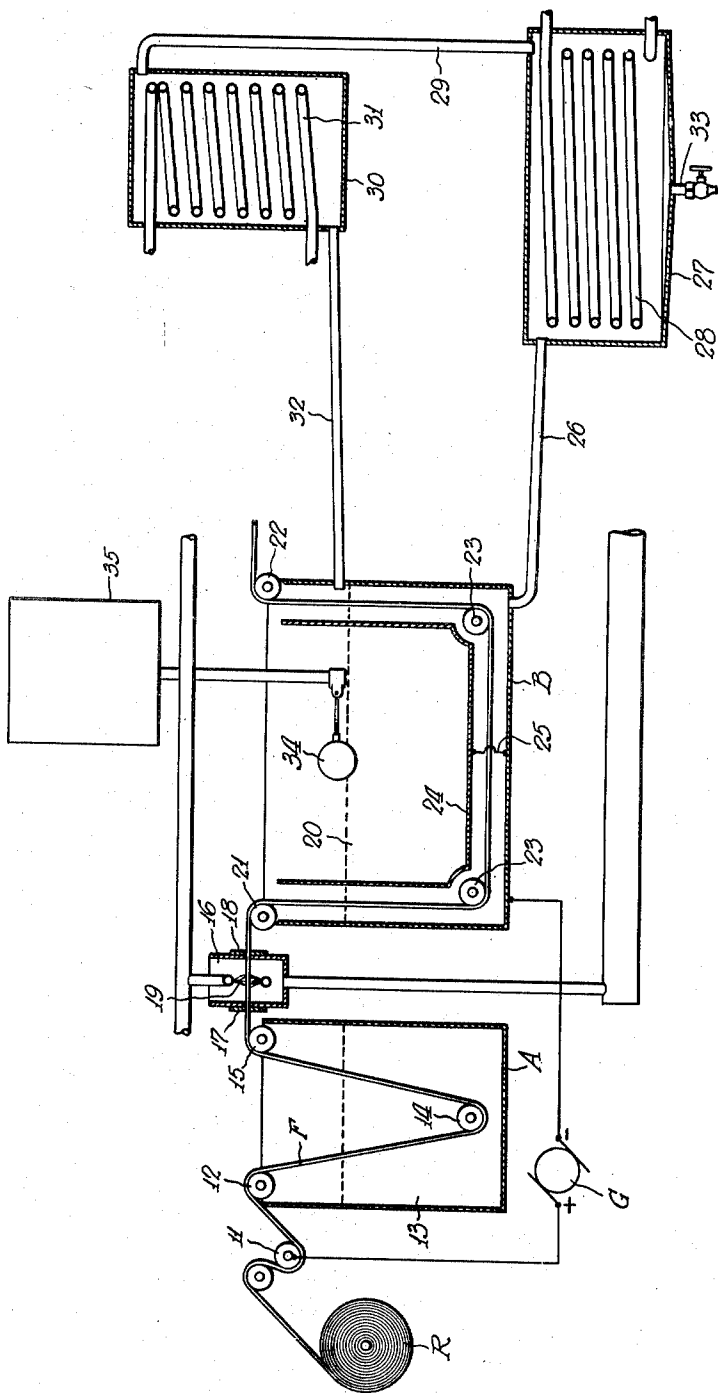
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ART OF RECLAIMING REAGENT

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ART OF RECLAIMING REAGENT

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While my present invention has a particularly useful field of application in the reclaiming of waste acid in the process of etching aluminum foil for use in electrolytic condensers, the invention in some aspects has a wider field of application in other arts especially where reagent solutions of constant boiling point proportions are or may be used.

Among the objects of the invention are to provide a process by which considerable economy may be effected in the re-use of otherwise waste reagent and in which such re-use may be effected by a substantially continuous and self-controlling action which requires no elaborate apparatus or skilled supervision.

According to the present invention the reagent solution which is in such degree of concentration that the boiling point remains substantially constant is progressively distilled and the distillate returned for re-use. Since the vapor or gas ingredients given off from the solvent and the solute in such distillation are in the same proportion by weight as are the solvent and solute in the reagent solution, the returned recondensed distillate will not disturb the concentration of the reagent in which the chemical or electro-chemical process is conducted. Fresh reagent need be added only at rate sufficient to compensate for that removed with the products of reaction.

In the indicated application of the invention for the production of etched aluminum foil, hydrochloric acid is desirably used as the etching reagent in a 20.2 per cent aqueous solution at which concentration the boiling point of the solution remains constant at 760 mm. pressure.

In the accompanying drawing in which is shown one of various possible embodiments of the several features of the invention, the figure is a purely diagrammatic view indicating the general correlation of the elements.

Referring now to the drawing, there is shown diagrammatically, apparatus for carrying out the process in one specific embodiment. Foil F from the supply roll R, preferably of annealed aluminum, is directed over to the cleansing tank, preferably for chemical or electro-chemical treatment, in manner well known to the art. The cleansing is performed in tank A, which contains a cleansing liquid 13. The foil is passed over inlet roller 12 at the top, one or more feed rollers 14 at the bottom, and an outlet roller 15 at the top of the tank A. If originally clean foil is used, this step may be omitted altogether. The cleansed foil leaves outlet roller 15 to pass

on through washer 16, which includes wipers 17 and 18 and an intervening hot water spray 19, thereby to remove all traces of the cleansing liquid.

The clean or cleansed foil is then passed through the etching tank B. This etching tank is made of a material such as iron, not subject to appreciable erosion with the etching fluid 20 employed therein. The tank is preferably equipped with an inlet roller 21 and an outlet roller 22, at the top, and a pair of feed rollers 23 near the bottom, over which the foil is passed in its traverse through the etching tank. In the electrolytic action of the etching fluid, the foil F serves as the anode and the tank B together with an inner metal plate 24 electrically connected to the tank by a conductor 25 serves as the cathode. As shown, the tank B and the inner cathode piece 24 are equidistant from the foil F in order to produce an equi-resistant conductive path through the electrolyte at opposite faces of the foil, thereby to effect a substantially uniform degree of etching thereat. By conventional shielding (not shown) the edges of the foil can be protected against excessive etching thereat. A roller 11 is provided over which the foil is fed to roller 12 and this is preferably metallic for electric contact, the positive terminal of a direct current generator G being connected thereto, the negative being connected to tank B. In practice the etched foil may proceed through a series of tanks (not shown) for further processing, but as this is no part of the present invention, it need not be further described but reference for an illustrative showing is made to my copending application Serial No. 46,082, jointly with Elliott Rippere, filed October 22, 1935.

The arrangement, which as thus far described, is not per se new is used with an etching reagent in tank B comprising a solution of about 20.2 percent by weight of hydrochloric acid in water. At that concentration the solution has a constant boiling point, that is to say, the concentration of the distillate remains constant as evaporation proceeds. The vapor or gas given off in boiling will have the same proportion of ingredients, namely 20.2 parts by weight of hydrochloric acid gas to 100 parts of water vapor.

According to the present invention that property is utilized for reclaiming the acid solution as it is being spent, by the combination of hydrochloric acid with the aluminum etched out, to precipitate $AlCl_3$ as a product of reaction. To this end the tank B is slowly and progressively

drained through a downwardly inclined pipe 26 to a hydrochloric acid boiler or still 27 that has a steam coil 28 therein which serves to evaporate the solution in the still and to cause it to pass through pipe 29 to a condenser 30 kept cool by the circulation of cold water through associated coil 31. The distillate having thus 100 parts of water to approximately 20.2 parts of hydrochloric acid in solution therein, the same concentration as the original solution in tank B, need merely be fed back through pipe 32 for re-use in the tank B. Accordingly the process may be and preferably is carried on in a closed system.

It is only necessary to replenish the tank B with a fresh supply of a 20.2% hydrochloric acid solution to compensate for the hydrochloric acid used up in forming the precipitate $AlCl_3$ in the etching reaction which precipitate is discharged through a drain 33 at the bottom of the still 27.

By resort to a float controlled valve 34 or the like the level of reagent in the tank B may be kept constant by gravity feed from a supply tank 35 containing hydrochloric acid at the concentration set forth. In practice, fresh acid is supplied at rate less than 30 per cent of that at which the used acid flows to the still 27, more than 70 per cent of the reagent being reclaimed.

According to the process described, the tank is thus continuously being freed of the aluminum chloride resulting from the etching process and the concentration of the liquid in the tank automatically and inherently remains substantially constant, so that the etching process will automatically be controlled to produce that uniformity of etching which is important in electrolytic condenser manufacture.

While the invention is of special utility when carried out in the particular manner set forth, it is in its broader aspects of wider range of applicability. The principle broadly can be applied to a wide variety of uses with a suitable reagent solution that has a constant boiling point at the concentration at which it is to be used in practicing the invention.

While in the particular application of the invention for etching aluminum foil for use in electrolytic condensers, it is generally satisfactory to use that degree of concentration of the etching chemical at which the boiling point remains substantially constant, situations arise in this and in other applications where it may be desirable to use a solution of a higher degree of concentration or of a lower degree of concentration. In such cases, it is of course clear that the concentration of the distillate will necessarily be different from that of the reagent solution. It will be obvious to those skilled in the art that the concentration of the etching fluid in the tank B even in such cases, may be kept uniform by replenishing the tank B with liquid from the supply tank 35 at concentration correspondingly less where the returned distillate is of greater concentration than that in the tank, or of concentration correspondingly greater where the concentration of the returned distillate is less than that in the tank B.

While the specific embodiment shown in the drawing involves an application to an electrolytic process, it is understood that the invention is by no means to be confined thereto, but is of wider application to chemical processes in which no electric current is used.

As many changes could be made in the above process and apparatus and as many apparently widely different embodiments of this invention

could be made without departing from the scope of the claims, it is intended that all matter contained in the above description or shown in the accompanying drawing shall be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. The process of reclaiming an aqueous solution of acid of the type which in a given concentration has a constant boiling point, which consists in progressively distilling the reagent solution, thereby to withdraw the products of reaction and to produce a distillate of said given concentration, returning the distillate and supplying fresh acid in said given concentration to make up for the loss of reagent in the product of reaction.

2. In the process of treatment with hydrochloric acid, the use of an aqueous solution of such acid in the constant boiling point proportions, the progressive distillation of the used acid solution, the return of the distillate for re-use and the replenishment with reagent in the constant boiling point proportions to make up for the removal thereof with the products of reaction.

3. In the process of chemical treatment in a hydrochloric acid tank, the use of an aqueous solution of said acid in the constant boiling point proportions, the progressive removal from the tank of used acid, the distillation thereof to remove the products of reaction, resulting in a distillate in the constant boiling point proportions, the return thereof to the tank, and the addition to the tank of aqueous solution of hydrochloric acid in the constant boiling point proportions to replenish that which has combined in the reaction product.

4. In the process of etching sheet aluminum, the step of passing such sheet progressively through a tank containing hydrochloric acid in aqueous solution and in constant boiling point proportions, progressively withdrawing the spent acid, distilling the same, condensing the distillate to the inherently constant boiling point proportion of acid and water and returning the same to the tank.

5. In the process of etching aluminum foil, the use of a 20.2 percent hydrochloric acid solution in water while subjecting the foil progressively to the action thereof, the progressive removal of the spent acid with its aluminum chloride precipitate, the vaporization of said spent acid with the separation of the aluminum chloride resulting from the reaction, and the condensation and return of the vaporized solution for re-use.

6. The process set forth in claim 5 in which a 20.2 percent aqueous solution of hydrochloric acid is added to the tank at substantially the rate at which hydrochloric acid is combined chemically with the aluminum.

7. An installation for using and simultaneously reclaiming a reagent solution in a chemical process, comprising a tank containing reagent solution of concentration having a constant boiling point, a boiler connected thereto in which products of reaction are progressively removed by distillation, a condenser reliequifying the distillate, means for returning the same to the tank and a source of supply of reagent solution in said concentration, connected to said tank and means for automatically controlling the feed therefrom to said tank at rate corresponding to that at which the reagent is lost from the system with the product of reaction.

8. The process of reclaiming a reagent solution which at a given concentration has a constant boiling point while using such solution in a container and at a concentration differing from such constant boiling point concentration, which consists in progressively distilling the used reagent to remove the product of reaction and returning the distillate to the reagent container, and adding reagent at rate and in concentration to compensate for the difference in the concentration of the distillate with respect to that of the reagent.

9. In the process of treatment with hydrochloric acid, the use in a tank of an aqueous solution of such acid, in proportions differing from those of constant boiling point, viz. 20.2 per cent, the progressive distillation of the used acid solution, the return of the distillate for re-use and the replenishment with reagent at concentration complementary to that of the returned distillate, whereby the mixture of the returned distillate and of the reagent added for replenishment is equal to that of the reagent in the tank, thereby to maintain such reagent substantially of constant concentration at all times.

10. In the process of etching sheet aluminum,

the step of passing such sheet progressively through a tank, containing hydrochloric acid in aqueous solution and at concentration differing from that of constant boiling point, viz. 20.2 per cent, progressively withdrawing the spent acid, distilling the same, condensing the distillate, returning the same to the tank and concurrently replenishing the tank with reagent of concentration complementary to that of the returned distillate, so that the mixture of the returned distillate and the reagent added for replenishment is substantially the same as that of the reagent in the tank, whereby substantial constancy of concentration of the reagent in the tank is maintained.

11. The process of reclaiming a reagent solution, which at a given concentration has a constant boiling point, which consists in progressively distilling the used reagent to remove the product of reaction and returning the distillate to the reagent container, and adding reagent at rate and in concentration to maintain the reagent solution at the original concentration.

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