July 1, 1947. M. C. TAYLOR 2,423,351

APPARATUS FOR AMALGAM DECOMPOSITION

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Fig. 1.

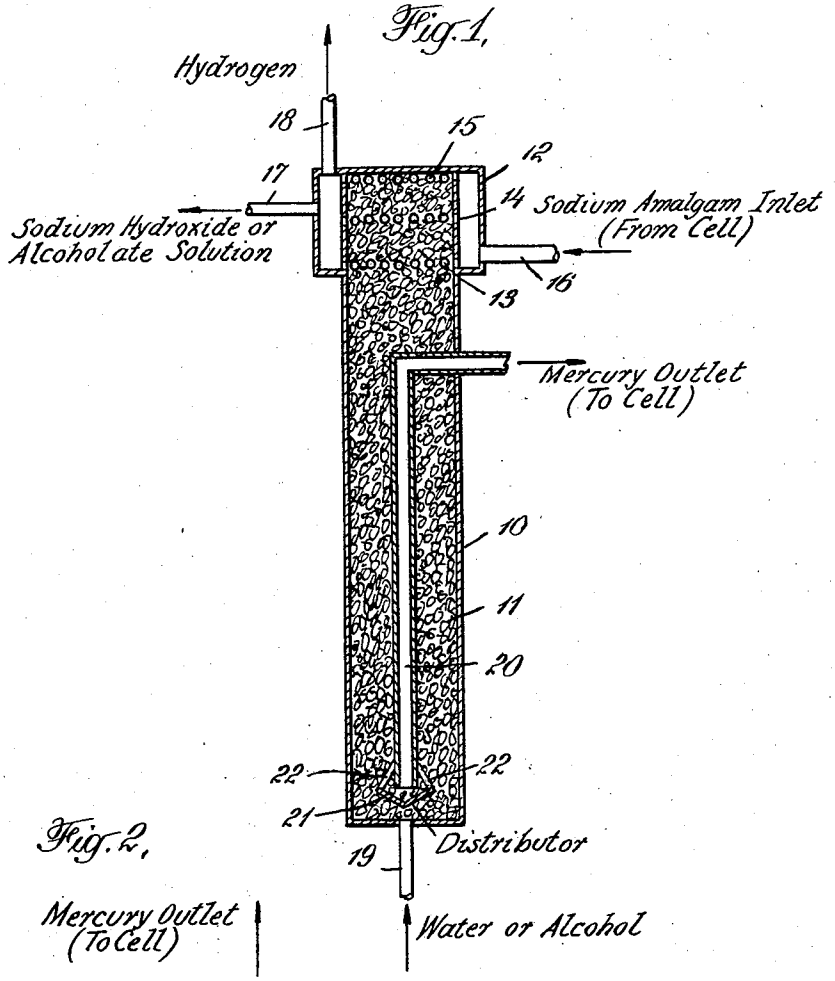
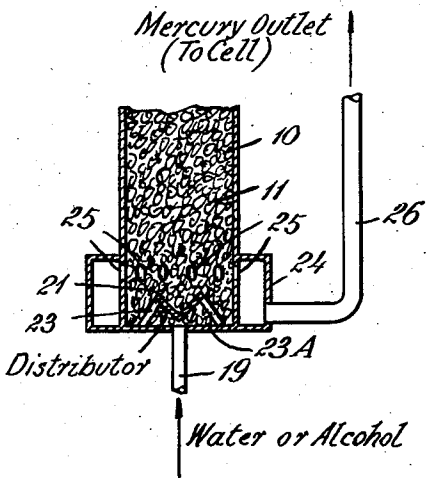


Fig. 2.



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Fig. 3,

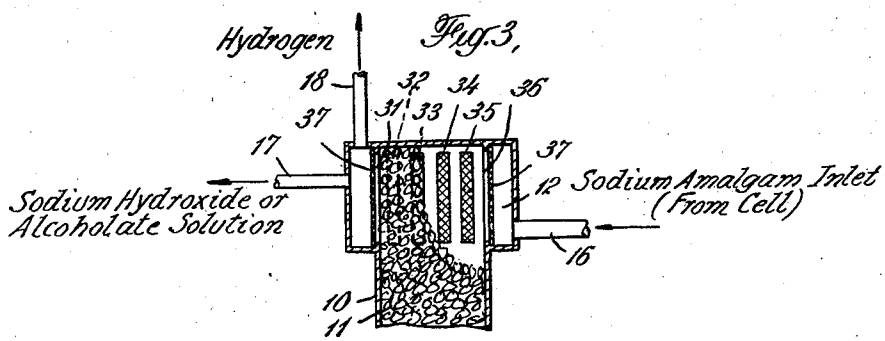


Fig. 4,

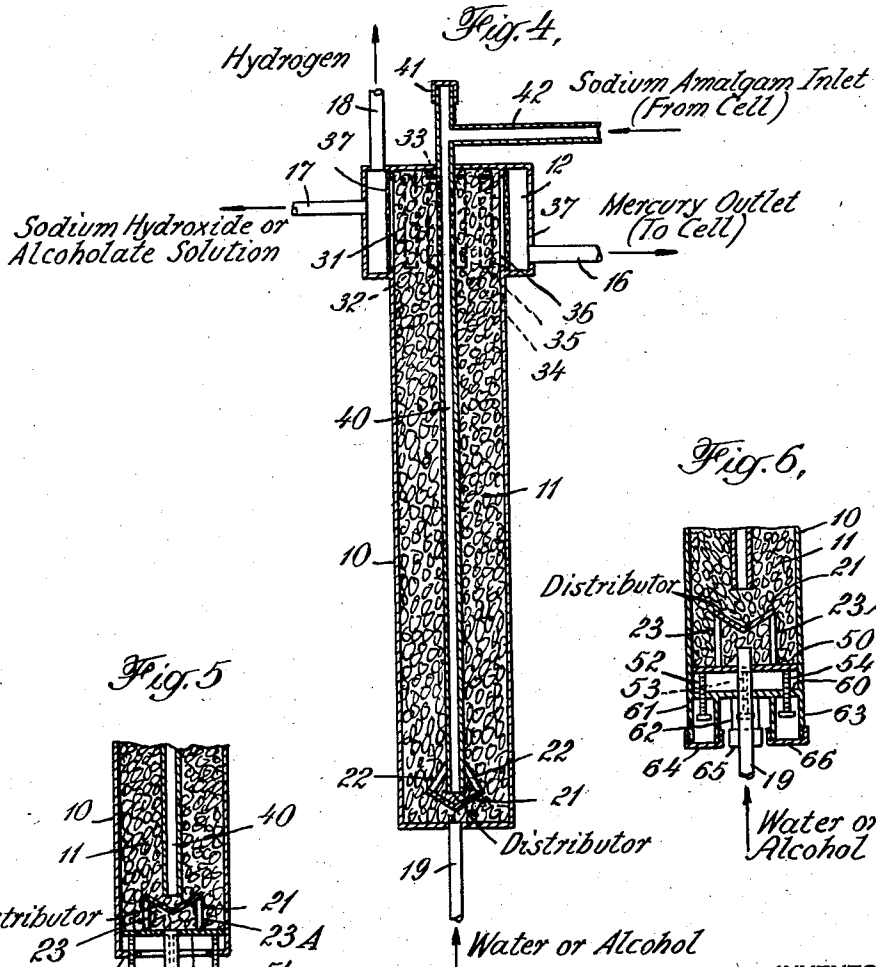


Fig. 6,

Fig. 5

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APPARATUS FOR AMALGAM DECOMPOSITION

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Application February 1, 1943, Serial No. 474,263

1 Claim. (Cl. 204—219)

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This invention is concerned with the decomposition of alkali metal amalgam in the presence of water or an alcohol having less than four carbon atoms to form alkali metal hydroxide or alcoholate.

I have discovered that the decomposition of amalgam of sodium potassium or lithium with water or alcohols having less than four carbon atoms is facilitated to a pronounced degree if the amalgam and the water or alcohol are passed through a tower containing a decomposition electrode in the form of a packing of electrically conductive material with which mercury does not amalgamate substantially and which is preferentially wetted by the water or alcohol, under conditions such that the water or alcohol forms a thin moving film on the packing and travels countercurrent to the amalgam through the tower.

As described in my copending application Serial No. 378,917 filed February 14, 1941, now Patent 2,336,045, of which this is a continuation in part, my invention contemplates (in a process involving reaction of alkali metal from amalgam with a liquid selected from the group consisting of water and alcohols having less than four carbon atoms per molecule) the improvement which comprises passing the amalgam and liquid in countercurrent contact with each other through a space containing an electrode in the form of a packing of electrically conductive material with which mercury or the amalgam does not amalgamate substantially and which is preferentially wetted by the liquid so that the liquid forms a thin moving film on the packing, the space in the packing, except for that occupied by the thin film, being substantially filled by a pool of mercury or the amalgam. Preferably, the packing is in such form that it provides a plurality of tortuous passages or channels, the walls of which are defined by the packing itself. In operating this process, the liquid preferably is passed upwardly in the tower while the amalgam is passed downwardly, and improved results are obtained if the packing is flooded by a downwardly moving stream of mercury or amalgam through which the liquid passes in countercurrent.

My invention further contemplates, in apparatus for reacting alkali metal from an amalgam thereof with a liquid selected from the group consisting of water and alcohols having less than four carbon atoms per molecule, the combination which comprises a tower, an electrode disposed in the tower and consisting of packing providing a plurality of passages through the tower and

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formed of electrically conductive material with which mercury does not amalgamate substantially and which is preferentially wetted by the liquid, means for admitting the amalgam and the liquid to the tower, means (such as a bottom in the tower) for maintaining a pool of mercury or amalgam in the passages through the tower and means for withdrawing the products of the reaction of the amalgam and the liquid from the tower. The packing, conveniently, may be of graphite. Preferably, the amalgam is admitted to the tower adjacent the top thereof while the liquid is admitted adjacent the bottom of the tower. The mercury in such case should be withdrawn from an intermediate portion of the tower, preferably through a conduit the outlet of which is substantially lower than the inlet of the conduit for mercury into the tower. The conduit for withdrawing hydrogen gas preferably communicates with the tower adjacent the top thereof and the conduit employed for withdrawing the liquid product of the alkali metal and the liquid (alkali metal hydroxide or alcoholate solution) preferably communicates with the tower near the top thereof, above the level at which the mercury is withdrawn.

In the preferred form of my apparatus, there is an annular chamber around the upper portion of the tower. Communication between this annular chamber and the tower is afforded by means of apertures in the tower wall, at three or more different levels or by means of elongated slots extending between the extreme levels. With such an apparatus the amalgam may flow from the annular chamber into the tower through the lowest of the three levels, with hydrogen flowing from the tower into the chamber at the uppermost level and with hydroxide or alcoholate solution flowing into the annular chamber at an intermediate level.

A preferred practice of my invention is described in detail hereinafter with reference to the accompanying Figure 1 which is a diagrammatic vertical section of a tower through which sodium amalgam and water are passed in countercurrent.

Fig. 2 illustrates a modified form of the apparatus of Fig. 1 provided with an improved means for withdrawing mercury.

Fig. 3 illustrates a modification of the apparatus of Fig. 1 in which the communication between the tower and the annular chamber surrounding it at the top thereof is by means of vertical screened slots instead of through rows of apertures at different levels.

Fig. 4 illustrates a modification of the appara-

tus of Figs. 1 and 3 in which the amalgam is introduced through a downcomer disposed at the center of the tower and provided with a clean-out, the flow of amalgam and liquid being, in general, concurrent.

Fig. 5 illustrates a modification of the apparatus of Figs. 1 to 4, provided with means for compressing the packing in the tower.

Fig. 6 illustrates a modification of the apparatus of Fig. 5.

The tower of Fig. 1 comprises a vertically disposed shell 10, preferably of uniform cross section. The shell may be constructed of any material that is not attacked by the materials passing therethrough. Thus, the shell may be in the form of a hollow cylinder of iron or ceramic closed at both ends. The shell is packed throughout with electrode material 11 that is electrically conductive, non-amalgamating and preferentially wetted by the water or alcohol. Graphite is a suitable material and in the apparatus illustrated the packing is formed of irregular shaped pieces of graphite about ⅜ inch in size. The interstices between the pieces form a plurality of tortuous passages from top to bottom of the tower.

The upper portion of the tower is surrounded by a closed annular chamber 12 that communicates with the interior through three rows 13, 14, 15 of holes drilled through the shell at different elevations. The holes are smaller than the pieces of packing and, conveniently, may be about $\frac{3}{16}$ or $\frac{7}{32}$ inch in diameter. A conduit 16 for introducing sodium amalgam to be decomposed is connected to the annular chamber near the bottom thereof adjacent the lowermost row 13 of the holes. A second conduit 17 for withdrawing the product (say, sodium hydroxide solution) is connected to the side of the annular chamber at approximately the level of the intermediate row 14 of holes and a third conduit 18 for withdrawing hydrogen is connected to the top of the annular chamber above the upper row 15 of holes.

The liquor (say, water) to be reacted with the amalgam is introduced into the bottom of the tower through an inlet pipe 19. The liquid must be introduced under a pressure sufficient to overcome the head of the contents of the tower and a pump (not shown) may be employed to force the liquid into the tower.

Mercury from which the sodium has been removed is withdrawn from the lower portion of the tower through a riser pipe 20 that passes upwardly through the tower to a point below the annular chamber and thence through the shell. An inverted distributor cone 21 is disposed below the riser and attached thereto by a spider 22. The cone serves as a seal to prevent water from entering the riser and leaving with the spent amalgam and as a distributor to cause the water entering through the inlet pipe 19 to spread throughout the cross section of the tower.

In the operation of the apparatus, sodium amalgam from an electrolytic cell, not shown, or other suitable source is admitted into the lower portion of the annular chamber and flows through the lowermost holes into the interior of the tower. Sufficient amalgam is introduced into the tower to submerge a predominating portion of the mass of packing material. Water or alcohol is forced into the bottom of the tower under pressure sufficient to overcome the head of amalgam in the tower. The water or alcohol

is lighter than the amalgam and mercury and tends to rise in the tower countercurrent to the amalgam flow. Reaction takes place between the amalgam and the water or alcohol so that as the amalgam travels down the tower it loses its sodium content until it is substantially mercury and, as such, is withdrawn through the riser. The reaction between the water or alcohol and the sodium of the amalgam produces sodium hydroxide or sodium alcoholate, as the case may be, together with hydrogen gas. The hydrogen gas and the resulting solution of sodium hydroxide in water or sodium alcoholate in alcohol both rise toward the top of the tower. The hydrogen rises to the uppermost row of holes and passes therethrough into the upper portion of the annular chamber from which it is withdrawn. Sodium hydroxide or alcoholate solution passes through the intermediate row of holes into the annular chamber and thence is withdrawn into the conduit provided for this purpose.

Since the water or alcohol preferentially wets the surface of the graphite electrode packing, it follows a path which takes the form of an upwardly moving thin film of solution on the electrode surfaces. This film moving up through the submerged mass of packing in the tower decomposes the amalgam in a most efficient way since the film is continuously replenished. The violent evolution and rise of hydrogen inside the tower tends to carry the amalgam as well as the other fluids in an upward direction. However, this tendency may be overcome by employing a tower of relatively large cross section in order to give more space for the escape of hydrogen or by increasing the head of amalgam on the tower.

The means provided for withdrawing the mercury from the tower is particularly suitable since it insures that a predominating portion of the tower packing will always be flooded with mercury or amalgam. The height of the sodium amalgam inlet above the mercury outlet represents the head required to force the amalgam countercurrent through the rising hydrogen and solution of hydroxide or alcoholate.

Fig. 2, which is a fragmentary vertical section through the lower portion of a tower similar to that of Fig. 1, shows a preferred means for withdrawing mercury from the tower. Water or alcohol to be reacted with the amalgam is, as in the case of the apparatus of Fig. 1, introduced into the tower through the inlet pipe 19 in the bottom thereof and is distributed throughout the tower cross section by the inverted cone 21 disposed immediately above the inlet and held in position by braces 23, 23A attached to the bottom. An annular jacket 24 is disposed around the lower portion of the tower. Mercury enters this jacket through a row of holes 25 in the upper portion thereof and is withdrawn through an upwardly extending riser pipe 26 that communicates with a lower portion of the jacket, which acts as a seal to prevent the water or alcoholate from escaping with the mercury. Otherwise, the apparatus partially illustrated by Fig. 2 is the same as that of Fig. 1.

Referring now to Fig. 3, it will be observed that the apparatus illustrated is the same as that of Fig. 1, except that a series of vertical slots 31, 32, 33, 34, 35, 36 are provided around the periphery of the tower in place of the three rows 13, 14, 15 of holes. Thus, the vertical slots extend from the level of the holes 13 to the level of the holes 15, and are covered with fine screen

37 which permits the passage of hydrogen through the top of the slot, sodium hydrogen or alcoholate solutions from the mid-portions of the slots and amalgam or mercury through the bottom of the slots, so that the three conduits provided, respectively, for the movement of hydrogen, liquid and amalgam or mercury have a screen in front of them.

The apparatus of Fig. 4 is similar to the apparatus of Figs. 1 and 3, except in the following particulars: The amalgam from the cell, instead of being admitted to the annular chamber 12 through the pipe 16, is admitted into the tower through a downcomer pipe 40 which is sealed into the top of the tower and passes downwardly and centrally therein to the distributor 21 which is fastened to the downcomer 40 by the spider 22. The top of the downcomer is covered by a removable clean-out cap 41, and a horizontally extending branch 42 of the downcomer is provided for admitting sodium amalgam thereto. The amalgam flows into the tower through the downcomer and thence rises concurrently with the water or alcohol introduced at the bottom of the tower. As the amalgam rises, it gives up its sodium and becomes mercury which flows through the slots 31—36 into the annular chamber and is withdrawn from this chamber through the conduit 16, which in this case acts as a mercury outlet.

The apparatus of Fig. 4 has the advantage of easy cleaning, in addition to the fact that the mercury passing through the downcomer is in countercurrent heat exchange relation with the rising column of liquid and mercury or amalgam in the tower.

It should be noted that in practically every case the packing will have specific gravity lower than that of the mercury or the amalgam, so that there will be a tendency to buoy the packing upwardly. If the packing is composed of a plurality of pieces, which is the more convenient form, it should have sufficient mechanical strength to resist crumbling and should be so confined in the tower that it will not crowd into the upper portion thereof to the extent that it interferes with flow through the tower.

Unless the packing is held firmly in place in the tower, the evolved hydrogen as it rises in the tower tends to grind together the pieces of packing and produces fines which may clog the tower or pass out through the upper portion thereof. This effect is more noticeable when the tower is employed for the production of sodium hydroxide than when it is employed to produce alcoholates, but in either case it is highly desirable that the packing be held firmly and tightly in the tower. This can be accomplished by providing the tower with means for compressing the packing therein. One such means is illustrated in Fig. 5, which illustrates a modification of the apparatus of Figs. 1 and 4.

Referring to Fig. 5, it will be seen that the shell 10 is provided with a movable bottom 50. An internal flange 51 around the lower edge of the tower wall is provided with a series of threaded adjusting screws 52, 53, 54. These screws, when turned in the proper direction, force upwardly the movable bottom which is provided at its edges with suitable gaskets (not shown) or is fitted to the wall tightly enough for sealing purposes. As the bottom is pushed upward the packing in the tower is compressed and is held firmly in position so that it does not tend to be ground together through the action of the hydrogen in the tower.

Fig. 6 illustrates a modified form of the apparatus of Fig. 5, provided with improved sealing means at the bottom. As in the apparatus of Fig. 5, the distributor 21 is fastened to the movable bottom plate 50 by uprights 23, 23A. The inlet pipe 19 projects upwardly through a central aperture in the movable bottom 50 and the movable bottom may be forced upward by means of screws 52, 53, 54 threaded through the bottom 60. The heads of the screws are covered by nipples 61, 62, 63 closed by threaded caps 64, 65, 66, so that the entire bottom of the structure may be sealed after adjustment. Otherwise the apparatus of Fig. 6 is the same as that of Fig. 5 and operates in the same manner.

I claim:

In an apparatus for reacting alkali metal from amalgam with a liquid selected from the group consisting of water and alcohols having less than 4 carbon atoms per molecule, the combination which comprises a tower having apertures arranged in horizontal rows near the upper end thereof, an annular chamber extending around the exterior of the upper portion of the tower, said apertures providing communication between the tower and the annular chamber at levels adjacent the lower portion of said annular chamber, the upper portion of said annular chamber and the intermediate portion of said annular chamber, an electrode disposed in the tower and consisting of a mass of packing providing a plurality of tortuous passages through the tower and formed of electrically conductive material with which mercury does not amalgamate substantially and which is preferentially wetted by the liquid, a conduit communicating at its outlet end with the lower portion of said tower for admitting the liquid to the tower adjacent the bottom thereof, a second conduit communicating at its outlet end with said annular chamber at substantially the level of the lowermost row of said apertures for admitting amalgam to the upper portion of the tower, whereby the amalgam will flow downwardly through the tower and the packing material therein countercurrent to rising liquid introduced into the tower at its lower end, a third conduit for discharging from the tower mercury resulting from the decomposition of the amalgam, said third conduit having its inlet end communicating with said tower near the lower end thereof, said third conduit also having a portion thereof disposed at a level below the outlet end of said second conduit but above the position occupied by a major portion of said packing material, whereby in operation a major portion of the mass of packing material is automatically maintained submerged in a pool of the amalgam and mercury resulting from the decomposition of the amalgam, baffling means disposed opposite and below the inlet end of said third conduit to form a seal whereby the liquid admitted to the lower portion of said tower is prevented from entering the inlet end of said third conduit, a fourth conduit communicating at its inlet end with said annular chamber at a point adjacent the upper end thereof for removing hydrogen gas from the upper portion of the tower, and a fifth conduit communicating at its inlet end with said annular chamber at a level intermediate the junctures of said second and fourth conduits with said annular chamber and at substantially the level of the intermediate row of said apertures, for withdrawing from the tower

the liquid products of the reaction of the alkali metal and said liquids.

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