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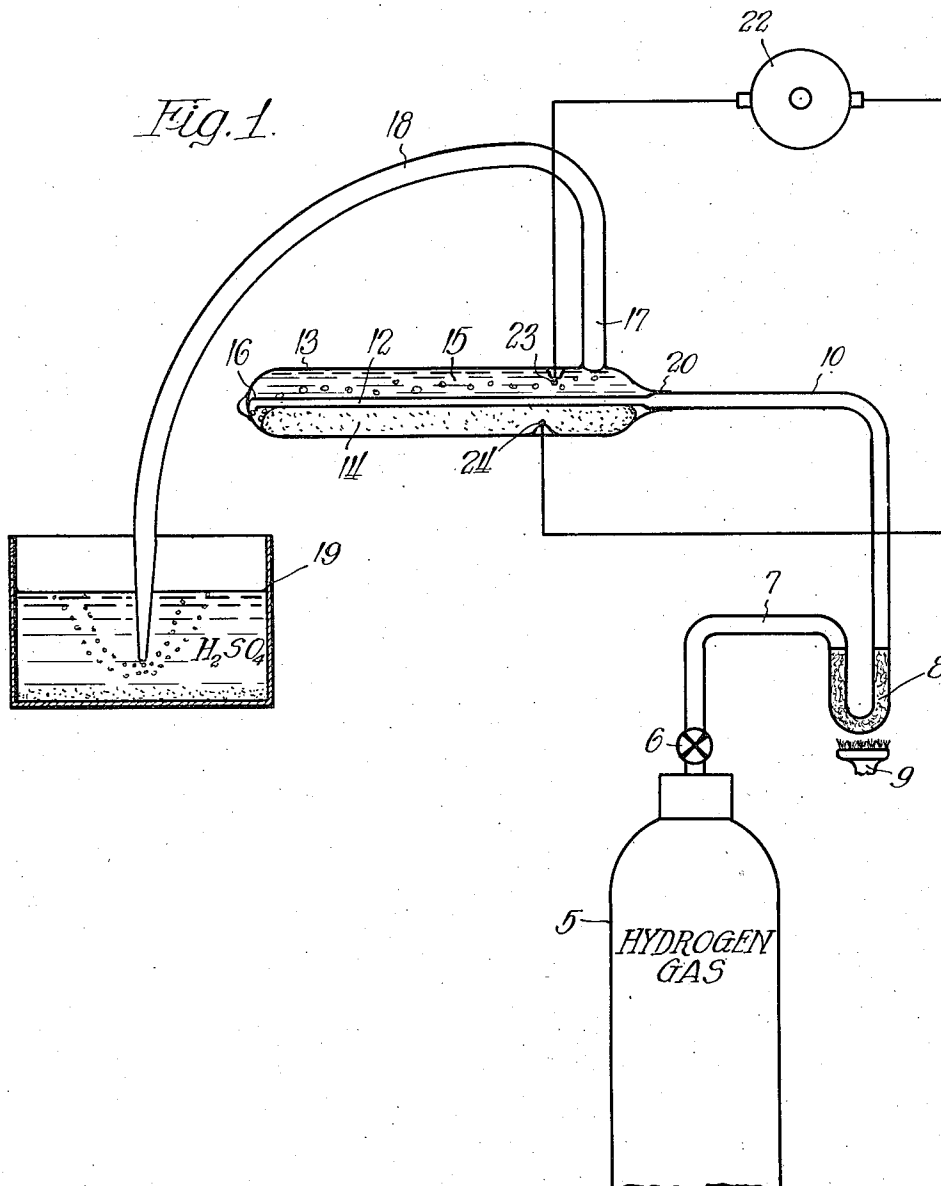
P. S. BEAR

2,134,694

PREPARATION OF MERCURY FOR USE IN MERCURY SWITCHES

Original Filed March 9, 1936

2 Sheets-Sheet 1



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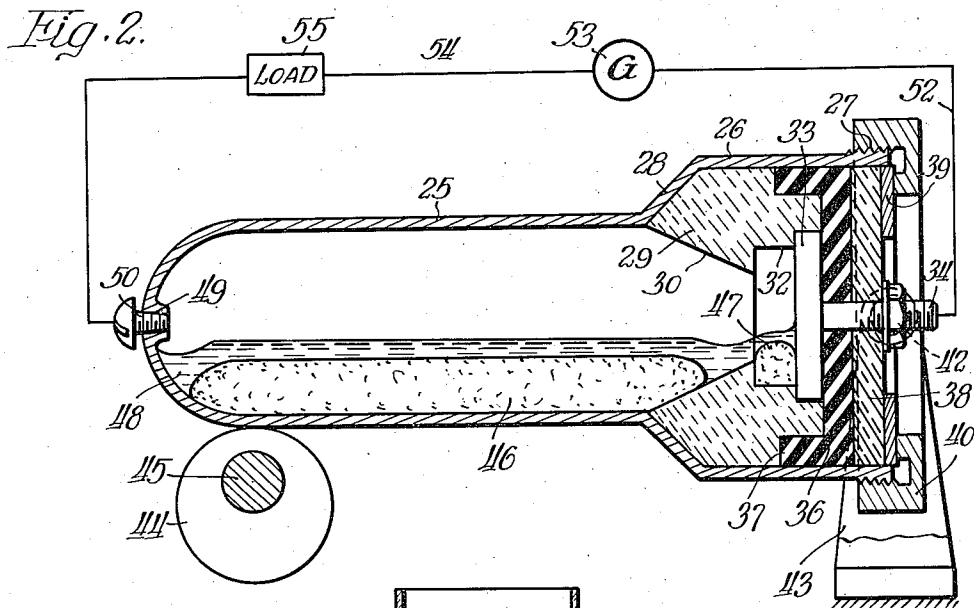
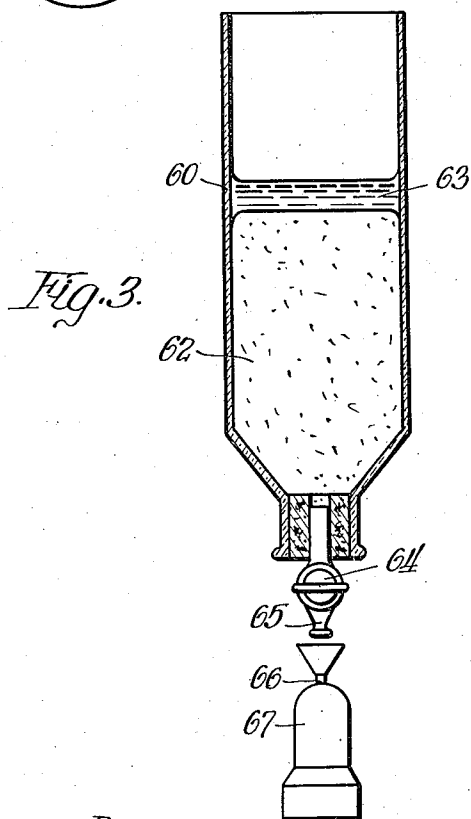
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2 Sheets-Sheet 2



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UNITED STATES PATENT OFFICE

2,134,694

PREPARATION OF MERCURY FOR USE IN
MERCURY SWITCHES

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Application March 9, 1936, Serial No. 67,816
Renewed January 24, 1938

13 Claims. (Cl. 75—81)

This invention relates to a method of preparing mercury for use in switches, and more particularly is directed to the cleaning and purification of mercury for use in metal envelope switches of the type shown in my copending application, Serial No. 745,842, filed September 28, 1934.

I have found that ordinary commercially pure acid washed mercury, when introduced into a metal envelope type of switch which employs a liquid fill, contains a certain amount of impurities, such as dust and foreign material, and also usually has a thin scum of mercury oxide over the top thereof. Since metal envelope switches, as well as the ordinary glass envelope type of switch, usually have occluded gases, such as oxygen, contained in the walls and on the interior surfaces thereof, even when the envelope has been subject to evacuation, in a short time an increased amount of mercury oxide is formed. The formation of the oxide obviously is detrimental to the operation of the switch and should be avoided as far as possible, where the mercury switch is to be depended upon to carry and interrupt any appreciable amount of current over long periods of time.

As described in my copending application, I prefer to use triethanolamine as the liquid fill in the switch, due to its volatility, and its other desirable characteristics as set forth in my copending application which serve to improve the operation of the switch. Among these characteristics may be mentioned the ability to increase the meniscus height of the mercury to an extent not obtainable in commercially pure mercury by the use of the present process.

The present invention, in its preferred embodiment, is directed to a method of removing oxygen, mercury oxide and other impurities from the mercury in the presence of triethanolamine, and then introducing the cleaned mercury and fresh triethanolamine into a switch envelope whereby during subsequent operation of the switch substantially no mercury oxide will be formed.

I have found that when acid washed mercury and triethanolamine are placed in a switch with impurities which may be deposited on the inside of the switch case, the arc formed in the switch will produce a change in the triethanolamine which results in possible formation of water due to hydrogen combining with the oxygen of the mercury oxide, and the reduction of the triethanolamine toward a di-ethanolamine and ethyl alcohol, or even possibly mono-ethanolamine.

In my present invention I propose to process the mercury prior to insertion thereof into a switch envelope, whereby the foreign matters and the oxygen and oxides are removed from the mercury and suspended in the triethanolamine used in the cleaning treatment of the mercury. Thus when the cleaned and purified mercury, with new triethanolamine, is placed in a switch envelope and the switch is operated, none of the reactions between the oxygen, hydrogen and other impurities takes place in the switch, and the mercury and triethanolamine remain in the same condition throughout the life of the switch.

This is decidedly advantageous to the operation of the switch, since triethanolamine has a higher boiling point than diethanolamine and also is more viscous, providing a better film forming medium. Furthermore, because of its higher boiling point, the internal pressure in the switch will be reduced for a given thermal condition.

Another advantage gained by this prior cleaning and treatment of the mercury is that it prevents the depositing of foreign matter on the inner walls of the envelope, and thus a switch filled with triethanolamine-cleaned mercury will initially operate at a narrower angle and with much less change in its operating angle during its flashing or "run in" period. Thus the shift in the angle of operation during the "run in" period will be materially reduced, as well as the time necessary to condition it for constant repeat performance. After the "run in" period the angle of operation does not change.

Still another advantage is that with the triethanolamine washed mercury in a switch and the pure triethanolamine added to this mercury in the proper limited quantity, the switch will have a lower operating angle after the "run in" period than with the usual acid washed mercury.

Other objects and advantages of my present invention will appear more fully from the following detailed description which, with the accompanying drawings, will disclose to those skilled in the art one manner in which my method may be carried out.

In the drawings:

Figure 1 is a schematic layout of a laboratory test set-up for determining the reactions which occur during cleaning of the mercury;

Figure 2 is a sectional view through cleaning apparatus for cleaning the mercury on a commercial scale; and

Figure 3 illustrates the manner in which the cleaned mercury is introduced into a switch envelope.

Figure 1 is illustrative of laboratory apparatus that may be employed to wash the mercury with triethanolamine in the presence of an arc and hydrogen to remove impurities from the mercury, to determine the reactions occurring in the washing process, and to fix the mercury in a condition such that when the washed mercury is placed in a switch with new triethanolamine, it will remain in such fixed condition during the life of the switch and will not adversely affect the condition of the triethanolamine.

Referring now in detail to the drawings, a hydrogen tank is indicated at 5, containing hydrogen gas under considerable pressure, which gas is passed through the control valve 6 and a conduit 7 into contact with heated copper filings indicated at 8, which copper filings are heated by means of a burner shown diagrammatically at 9. The purpose of passing the hydrogen through the copper filings is to insure removal of all oxygen therefrom, since any oxygen present will, in the presence of heat, combine with the copper filings to form copper oxide. The purified hydrogen gas then passes through the conduit 10 and through the extension 12 thereof into the rear end of a washing chamber 13. The washing chamber 13 contains the body of mercury 14 which is to be treated, and a body of triethanolamine 15 which lies on top of the mercury 14, and through which the hydrogen gas bubbles from the end 16 of the tube 12 toward the outlet 17. The purpose of extending the tube 12 throughout the length of the chamber 13 is to insure that the gas will pass through the entire portion of the triethanolamine prior to its exit through the outlet 17 which leads thru the conduit 18 to below the surface of the sulphuric acid in the tank 19.

The tube 10 is sealed, as indicated at 20, to the inlet of the tube or chamber 13, in order that the materials within the tube will be prevented from escaping through the junction between the tube 10 and the inlet of the chamber. A suitable generator 22, driven in any desired manner, is connected to the two electrodes 23 and 24, to produce a high voltage arc within the chamber 13 to effect combination of the hydrogen and oxygen.

While I have not been able to determine precisely the chemical action which occurs during this washing process, I have found that it can be generally indicated by the following formula:



It will be noted that the triethanolamine, during this operation, is reduced toward a diethanolamine, with the consequent formation of ethyl alcohol and water. The hydrogen for forming the water and alcohol is produced from the tank 5, and the oxygen which is necessary to complete the formation of these substances is drawn from mercury oxide which may be formed over the surface of the mercury 14, from included oxygen which may be in the chamber 13, and from oxygen which may find its way into the chamber through sources unknown. As long as there is an excess of hydrogen in the chamber 13, the water and ethyl alcohol will remain suspended in the triethanolamine within the chamber, and at the same time, any foreign particles, dirt or the like carried by the mercury 14 will be floated by the triethanolamine 15, so that these impurities are removed from the mercury.

Thus, any oxides or oxygen which might be contained in the mercury are removed and carried away in the triethanolamine. This stabilizes

the mercury in a condition such that when it is introduced into a switch in the presence of fresh purified triethanolamine, it will remain substantially in the condition in which it was introduced, that is, upon flashing of the switch there will be no tendency for the mercury to form mercury oxide, due to the fact that there now is no oxygen or oxides carried in the mercury, and due to the fact that all other impurities of the mercury have also been removed so that it is operating in the switch substantially only in the presence of triethanolamine, the switch tube having been previously thoroughly cleaned and evacuated, and hence the mercury will have no opportunity to combine with any oxygen in order to form the oxides, since substantially all the oxygen has been removed therefrom in the treating process.

Of course, it is to be understood that the invention is not limited merely to the treatment of mercury for use in mercury switches, since mercury, treated in this manner, may find other uses in which it is desired that it be entirely free of any included oxygen or the like.

In the carrying out of this method in the laboratory for test purposes, I conducted the gases formed during the treatment of the mercury through the conduit 18 into the sulphuric acid tank 19, in which may be placed caustic potash. In this way I was able to determine whether or not any ammonia was being driven off from the triethanolamine, since the caustic potash will drive off ammonia introduced into sulphuric acid. Such a process is especially adapted for detecting slight traces of ammonia. From the tests which I made I found that there was no ammonia being driven off, but that a slight trace of an alcohol was noticeable after the caustic potash was put into the sulphuric acid. I believe that the reason for this phenomenon is that the triethanolamine was being changed toward a diethanolamine or even possibly to a mono-ethanolamine, and that the trace of alcohol came from the one leg of the ethanolamine being converted to ethyl alcohol, while the ammonia portion formed by the basic nitrogen molecule stayed in the compound and formed the diethanolamine.

In the commercial embodiment of my invention, shown in Figures 2 and 3, I provide an enclosing envelope 25, which may be cylindrical in section and is provided with the enlarged end portion 26 suitably threaded adjacent its outer periphery as indicated at 27.

Disposed within the envelope 25, and bearing against the tapered portion 28 thereof, we provide a ceramic insert 29, which is provided with an axially extending passageway defined by the tapered or conical opening 30 and the cylindrical surface 32, defining therebetween an acute cutting edge disposed radially inwardly of the surface 32.

Placed rearwardly of the surface 32 I provide the electrode stud 33, which is preferably shaped in the form of a disc and provided with an outwardly extending threaded stud 34 projecting outwardly of the enlarged end of the envelope 25.

The stud is held in position against the shoulder formed at the right hand edge of the annular surface 32 by means of a compression gasket 36, formed of soft rubber or the like, which has an extending skirt portion 37 projecting axially beyond the stud adjacent the inner surface of the enlarged portion 26 of the envelope. A suitable Bakelite gasket 38 bears against the outer surface of the compression member 36, and is held

in position by an annular sealing ring 39 which is clamped against the peripheral surface of the gasket 38 by means of the clamping nut 40, which is threaded over the outer end of the shell 25.

5 This serves to lock the assembly in fixed position, with the stud 33 held against the ceramic 29, and with the ceramic itself held against the tapered surface 28 of the envelope. The nut 40 is provided with oppositely extending trunnion members 42 which are suitably supported in a bearing bracket 43 to allow rotative movement of the envelope 25 thereabout under the influence of an eccentric cam 44 carried by a rotating shaft 45. This is merely one optional manner of securing making and breaking of the circuit within the envelope 25.

10 The envelope 25, with the electrode 33 and the ceramic 29 in position, is thoroughly evacuated, and then mercury as indicated at 46, is introduced into the envelope, a portion of the mercury being retained within the annular groove formed by the surface 32, as indicated at 47, and as described in detail in my copending application, Serial No. 53,146, filed December 6, 1935.

15 After the mercury has been introduced, a predetermined amount of triethanolamine, as indicated at 48, is also introduced into the envelope through the opening 49, which is then sealed by the plug 50. The electrode stud 33 is then connected by the conductor 52 to one side of a generator 53, the opposite side of the generator being connected to the conductor 54 and load 55 with the plug 50 which is in electrical contact with the envelope 25.

20 Rotation of the shaft 45 produces making and breaking of contact between the bodies of mercury 46 and 47, causing arcing within the envelope 25, and this arc, produced while the triethanolamine is in contact with the mercury, serves to eliminate any included oxygen or oxides from the mercury, these foreign materials being carried in suspension in the triethanolamine during the arcing.

25 In one preferred embodiment of the invention, the envelope 25, with the parts assembled as shown, has an internal capacity of 300 cc. Into this envelope is introduced 140 cc. of mercury, and 30 cc. of triethanolamine, and the switch is operated at 200 amperes for an hour, opening and closing at a rate of approximately ten times per minute.

30 After the washing operation has been completed, and the triethanolamine washed mercury has been purified by removal of all oxides or other included impurities into the triethanolamine, the mercury is poured from the envelope into a burette indicated at 60 in Figure 3. The mercury introduced into the burette 60 falls to the bottom, as indicated at 62, and the enclosing envelope of triethanolamine, which passed outwardly from the envelope 25 as the mercury was poured therefrom, and thus prevented the mercury from coming into contact with air during its removal into the burette 60, is forced, by the difference in densities between the mercury and the triethanolamine, into the position shown at 63 in Figure 3, closing off the upper surface of mercury from contact with the air.

35 A suitable stop cock 64 is provided, which has an outlet 65 centered with respect to the filling opening 66 in a mercury switch envelope 67. The cleaned mercury 62 from the burette 60 is passed through the cock 64 and the outlet 65 into the interior of the switch envelope 67, which has been previously washed and cleaned.

This mercury is fresh, and has not been in any substantial contact with air since it has been washed with the triethanolamine. The cleaned mercury introduced into the switch 67, together with fresh pure triethanolamine, produces a switch which has a smaller running in period, and which operates at a much smaller angle, due to the fact that the mercury employed in the switch has substantially no impurities therein, and substantially all of the oxides have been removed therefrom in the washing process shown in Figure 2.

40 Of course, it is obvious that any desired enclosing envelope might be employed for washing the mercury with the triethanolamine in the presence of an electric arc, and I do not intend to limit the invention to the particular apparatus shown in Figure 2 although this is a preferred form of carrying out my present process. Further, the purified mercury may be introduced into a switch envelope in manners other than as shown in Figure 3, but the principles underlying the washing and subsequent filling of a switch envelope with a purified mercury remain substantially the same regardless of the particular apparatus which I may employ in the various steps of my process.

45 It will be noted that the proportion of triethanolamine employed with respect to the amount of mercury in the envelope 25 during the washing process is considerable greater than is normally used in a mercury switch, as disclosed in my copending application of Herbert E. Bucklen and myself Serial No. 2,990, filed January 23, 1935. The larger amount of the triethanolamine will pick up more impurities out of the mercury, and will hold them in solution for a longer time without becoming saturated. Further, more triethanolamine will be presented for reaction with any foreign gases so that a more complete cleaning process is obtainable.

50 While I have disclosed my invention specifically in connection with the treatment of mercury to rid it of impurities and oxygen-forming compounds which may be present therein in order to employ it satisfactorily in a mercury type switch, I do not intend to limit myself to the preparation of mercury only for this purpose.

I claim:—

1. In the process of preparing mercury to remove impurities therefrom, the step of washing said mercury with triethanolamine in the presence of an electric arc.

2. The method of removing oxides and other impurities from mercury which comprises washing said mercury with triethanolamine in a washing chamber, simultaneously producing an arc in said chamber, and separating the washed mercury from said triethanolamine.

3. The method of conditioning mercury for use with triethanolamine in a switch which comprises washing said mercury with triethanolamine in the presence of an arc to remove oxygen and other impurities therefrom while reducing said triethanolamine, suspending said removed impurities in the reduced triethanolamine, and removing the impure triethanolamine.

4. The process of preparing mercury for use in metal-envelope type mercury switches which comprises washing said mercury with triethanolamine in a washing chamber, establishing an electric arc within said chamber to effect removal of oxides or free oxygen carried in said mercury, removing said washing triethanolamine, and enveloping said treated mercury in a film of tri-

ethanolamine to prevent any substantial contact of said treated mercury with air.

5 5. A method of removing oxygen from mercury containing the same which comprises washing said mercury with triethanolamine, and passing an electric arc therethrough to suspend impurities from said mercury in said triethanolamine.

10 6. A method of removing free oxygen and oxides from mercury which comprises contacting said mercury in the presence of a washing medium and an electric arc, and pouring said mercury within an enclosing film of said medium into a container to prevent air from contacting
15 said treated mercury.

7. The method of stabilizing mercury for use in a mercury switch which comprises flashing said mercury in the presence of triethanolamine to suspend the impurities from said mercury in
20 said triethanolamine, removing said triethanolamine and suspended impurities, and enveloping such portions of said mercury as remain in contact with air in a film of triethanolamine.

8. The method of treating mercury to remove
25 any oxygen-containing compounds therefrom which comprises oscillating a body of mercury in the presence of triethanolamine and an electric arc to suspend said compounds in said triethanolamine, drawing off said treated mercury within
30 a protecting film of said triethanolamine, and

displacing said film of triethanolamine to only such portion of said treated mercury as remains in contact with air.

9. A method of purifying and simultaneously increasing the meniscus height of mercury which
5 comprises washing said mercury with triethanolamine, and simultaneously passing an electric arc therethrough to suspend the impurities from said mercury in said triethanolamine.

10 10. The method of increasing the meniscus height of mercury which comprises washing said mercury with triethanolamine in the presence of an electric arc, and drawing off said mercury in an enclosing film of said triethanolamine.

11. The method of preparing mercury which
15 comprises washing said mercury with triethanolamine in the presence of a hydrogen atmosphere.

12. The method of preparing mercury which
20 comprises forming an electric arc between a body of mercury and an electrode in the presence of a film of triethanolamine.

13. A method of maintaining a body of mercury free from oxides and other impurities in a metal container which comprises successively
25 producing an electric arc in said shell, and maintaining said body of mercury covered by a film of triethanolamine which is capable of being punctured by said arc.

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