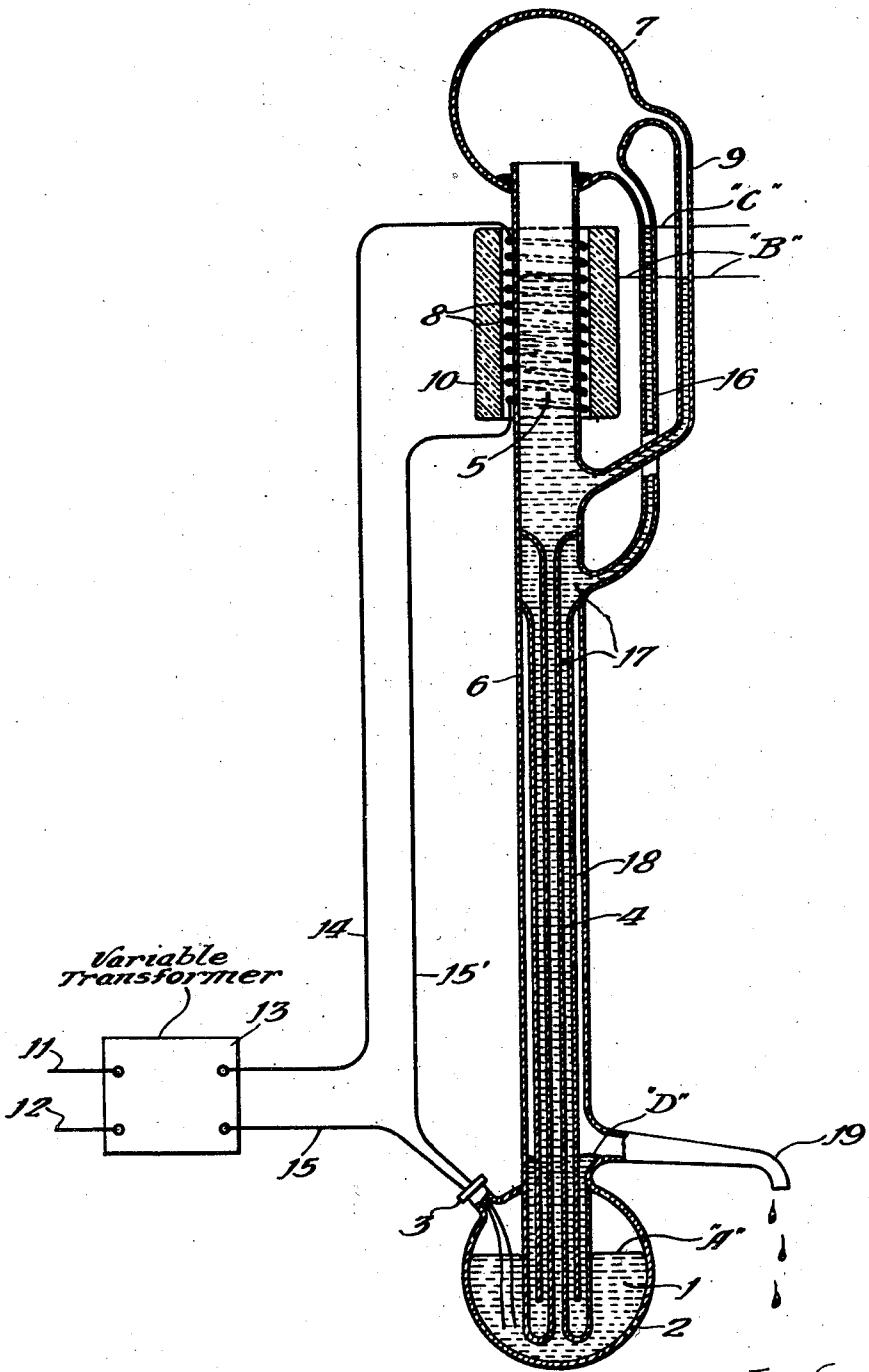
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MERCURY VACUUM STILL

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MERCURY VACUUM STILL

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This invention relates to an improved and novel type of mercury distillation apparatus which automatically maintains a vacuum in the heating and vaporizing zone.

Many chemical and research laboratories make use of considerable quantities of mercury in their testing and analysis work so that the mercury becomes contaminated with impurities as it is used. Most ordinary dirt, oils, oxides, sulphur compounds, or materials of lower boiling point than mercury may be washed from the mercury with nitric acid and acetone. However, it has been found advantageous by many laboratories to maintain a mercury still in order to remove other impurities such as copper, silver, aluminum, or other metals which amalgamate with the mercury, or other materials which may have a higher boiling point than the mercury.

This type of still need not be limited in size or capacity, or to being used by small laboratories only; for relatively large units may be constructed for commercial installations, as well as in connection with laboratories handling considerable quantities of mercury.

The principal advantage of this vacuum still is that it is not necessary to have a vacuum pump or other vacuum producing means continually working in conjunction with the vaporizing operation. Another advantage of this device is that with the electrical heating method and the safety shut-off that is provided, it is not necessary to have anyone tend the operation of the apparatus.

The still makes use of the barometric principle in its construction and operation. In the preferred embodiment of the still, concentric tubes are used which are sealed one from another in such a manner that two independent barometric legs or columns are formed.

The inner concentric tube is open at the bottom to a reservoir or an enlarged chamber which contains mercury that is to be distilled. Atmospheric pressure maintains a raised column of the mercury in this inner concentric tube; thus, the upper part of the tube is used as a heating and vaporizing zone and has an electrical heating coil encasing it. Above the heating coil, the top of this inner tube opens into an enlarged vapor condensing zone in which a vacuum is maintained. The condensed or distilled mercury collects in a well or collecting ring, provided in the bottom of this upper condensing chamber, and then runs down into an outer barometric leg or the annular space around the inner mercury feed tube of the device. This outer annular space is sealed from the inner tube and from the lower reservoir containing the

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impure mercury. At the lower end of the discharge column, the mercury flows through a trap arrangement to an outlet spout. The special method of removing the condensed mercury maintains an air seal and the vacuum in the condenser flask at the top.

The distillation of mercury should be carried out under vacuum conditions, preferably at an absolute pressure which is of the order of 1 mm. of mercury or less, in order to have the mercury vaporize at a lower temperature than its normal boiling point of approximately 358° C. It is also advisable to operate the still so that boiling or "bumping" does not occur and thus prevent any of the impure mercury from bouncing or carrying over into the clean mercury column.

The electrical heating coil provides an ideal means of supplying the heat for vaporization of the mercury. The amount of heat is easily controllable by a variable transformer, and a safety shut-off is easily provided by routing the flow of electrical current through the mercury supply in the lower reservoir. Thus, if the mercury level recedes to a point that approximates the empty point, the current is automatically broken and the distillation operation is stopped.

The accompanying drawing which shows the still in a cross-sectional elevation view, and the following description thereof, should make the novel features and construction details of the invention more apparent.

Referring now to the drawing, the impure mercury 1, which is to be distilled, is poured into the bottom flask 2 through the opening 3. The mercury is maintained raised in the tube 4 by atmospheric pressure, in the manner of a barometer, reaching the heating zone 5 in the upper part of the apparatus. The outer tube 6 forms the wall of the heating or vaporizing zone 5 and it extends upward into the spherical chamber 7, which forms the condenser for the mercury vapor.

Since the normal atmospheric pressure will maintain the level of the mercury in zone 5 approximately 30 inches above the level A in the lower reservoir, it is thus evident that the still has minimum limitations as to its height. The bottom of the condenser 7 should be something over 30 inches above the top of the reservoir 2, and the top of the heating coil 8 should be maintained at approximately 30 inches above the level A. The level B to which the impure mercury rises will of course vary slightly with variations in atmospheric pressure. The auxiliary tube 9, connecting between zone 5 and the condensing sphere 7, acts as a level gauge-glass to show the height of the

mercury in zone 5, which is concealed by the electrical heating coil 8 and the surrounding insulation 16.

The current for heating the resistance coil 8 is supplied through lines 11 and 12 to a variable type of transformer 13 which will allow adjustability as to the amount of heat. The electric current leaves the transformer 13 through lines 14 and 15 to be carried to coil 8. Line 15 is shown to be broken with the circuit being maintained by the conductivity of the mercury 1, which carries current from line 15 to line 15'. This feature makes an automatic safety control. As the impure mercury level A falls to the bottom of the flask or reservoir 2, the ends of wires 15 and 15' lose contact with the mercury and the current supply to the heating coil 8 is broken.

The vaporized mercury cools and condenses on the walls of the upper zone 7 and collects around the upper extension of tube 6; it then empties into tube 16. The tube 16 connects with the annular space 17 between tube 4 and tube 18. The distilled mercury column maintains a high level C, also in the manner of a barometer. As mercury enters the top of tube 16 then an equivalent amount of mercury is discharged down around the lower end of tube 18 and out of the spout 19. A trap or liquid seal is formed by the mercury pocket between the discharge level D and the bottom end of tube 18. With atmospheric pressure acting on level D, then the upper level C of the discharging column of clean mercury, will be approximately 30 inches above level D.

This novel method of discharging the distilled mercury from the still is such that it maintains a vacuum in the condenser 7 and on the vaporizing zone 5. When initially starting up the still, it is necessary to obtain the vacuum in the upper portion of the apparatus, and to pull mercury into the barometric columns. This may be done by attaching a vacuum pump to the outlet spout 19, evacuating the air in the still, and pulling mercury (which may be fed into the lower chamber 2) up the inlet leg 4 and over the top rim of tube 6 in the chamber 7, until the discharge leg or annular column 17 is filled. Then upon disconnecting the vacuum pump, the mercury will fall to levels B and C in each of the independent barometric columns, as determined by the atmospheric pressure, and a vacuum is formed in the closed top portion of the apparatus.

Once having obtained a vacuum in the still, it is always ready for operation. By turning on the electric current to the coil 8 vaporization can be started, and distilled mercury will be gradually discharged from the outlet 19. Impure batches of mercury may be periodically poured into the lower reservoir 2, otherwise as previously stated the operation is automatic, with the still requiring no other aid.

The apparatus should preferably be constructed of "pyrex" glass; however, steel or any other material which will not react with the mercury, may be used. Glass has an advantage in that the mercury levels and the operation may be observed. The discharge barometric column of course need not be annular shaped, or in contact with the feed tube. The preferred form is shown in this manner because of compactness and good design features. Also other means of heating, than that shown and described, may be used within the scope of this invention, although electrical heating seems to be the most practical for automatic operation of the still.

I claim as my invention:

1. A vacuum still for mercury comprising a bottom reservoir for impure mercury, concentric tubes for barometric mercury columns, an inner tube of said concentric tubes for a barometric column of impure mercury extending into the lower part of said reservoir, a vaporizing zone provided by flaring the upper end of said inner tube into the outer of said concentric tubes, a heat providing means adjacent to and exterior of said vaporizing zone, a vapor condensing chamber positioned above and connecting with said vaporizing zone, a collecting well in said vapor condensing chamber, an outlet conduit from said well connecting with a discharge barometric column of distilled mercury, an intermediate concentric tube positioned between the inner and the outer of said concentric tubes forming thereby an annular space around said inner tube for said discharge mercury column, a pocket formed by connecting said inner and said outer concentric tubes at their lower ends within said reservoir, said intermediate concentric tube projecting into said pocket such that said annular discharge column feeds around the bottom submerged end of said intermediate tube, and a drawoff spout from the side of said outer tube at a point above said submerged end of said intermediate concentric tube.

2. A vacuum still for mercury comprising a bottom reservoir for impure mercury, concentric tubes for barometric mercury columns, an inner tube of said concentric tubes for a barometric column of impure mercury extending into the lower part of said reservoir, a vaporizing zone provided by flaring the upper end of said inner tube into the outer of said concentric tubes, an electric heating coil around said outer tube at said vaporizing zone, a vapor condensing chamber positioned above and connecting with said vaporizing zone, a collecting well in said vapor condensing chamber, an outlet conduit from said well connecting with a discharge barometric column of distilled mercury, an intermediate concentric tube positioned between the inner and the outer of said concentric tubes forming thereby an annular space around said inner tube for said discharge mercury column, a pocket formed by connecting said inner and said outer concentric tubes at their lower ends within said reservoir, said intermediate concentric tube projecting into said pocket such that annular discharge column feeds around the bottom submerged end of said intermediate tube, and a drawoff spout from the side of said outer tube at a point above said submerged end of said intermediate concentric tube.

3. The apparatus of claim 2 further characterized in that the electric circuit to said heating coil is routed such that said circuit will be broken when the mercury level in said reservoir recedes to the bottom thereof.

4. A mercury still comprising a reservoir for impure mercury, a condenser for mercury vapors disposed above said reservoir, an inlet tube extending from within said reservoir to said condenser, heating means disposed exteriorly of and adjacent to the upper portion of said inlet tube to form a mercury vaporizing zone within said upper portion intermediate said reservoir and said condenser, a concentric jacket around said inlet tube extending from a point below said vaporizing zone downwardly into said reservoir to form an annular passage around said inlet tube, said annular passage terminating within said reservoir in a sealing trap, means for collecting condensed mercury vapors and discharging

the same from said condenser into the upper part of said annular passage, and a drawoff for withdrawing distilled mercury from the bottom of said annular passage through said sealing trap whereby to prevent loss of vacuum in said condenser during the withdrawal of distilled mercury.

5. The apparatus of claim 4 further characterized in that said heating means comprises an electric heater disposed in a circuit comprising a source of electric current and contact points so positioned in said reservoir that when the level of mercury therein recedes below said contact points the circuit is broken.

6. The apparatus of claim 4 further characterized in that said sealing trap comprises a U-shaped extension of said annular passage.

7. A mercury still comprising a reservoir for impure mercury, a condenser for mercury vapors disposed above said reservoir, an inlet tube extending from within said reservoir to said condenser, heating means for vaporizing mercury in the upper portion of said inlet tube intermediate said reservoir and said condenser, a concentric jacket around said inlet tube extending from a point below said vaporizing zone downwardly into said

reservoir to form an annular passage around said inlet tube, said annular passage terminating within said reservoir in a sealing trap, means for collecting condensed mercury vapors and discharging the same from said condenser into the upper part of said annular passage, and a drawoff for withdrawing distilled mercury from the bottom of said annular passage through said sealing trap whereby to prevent loss of vacuum in said condenser during the withdrawal of distilled mercury.

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