

Sept. 17, 1929.

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2 Sheets-Sheet 1

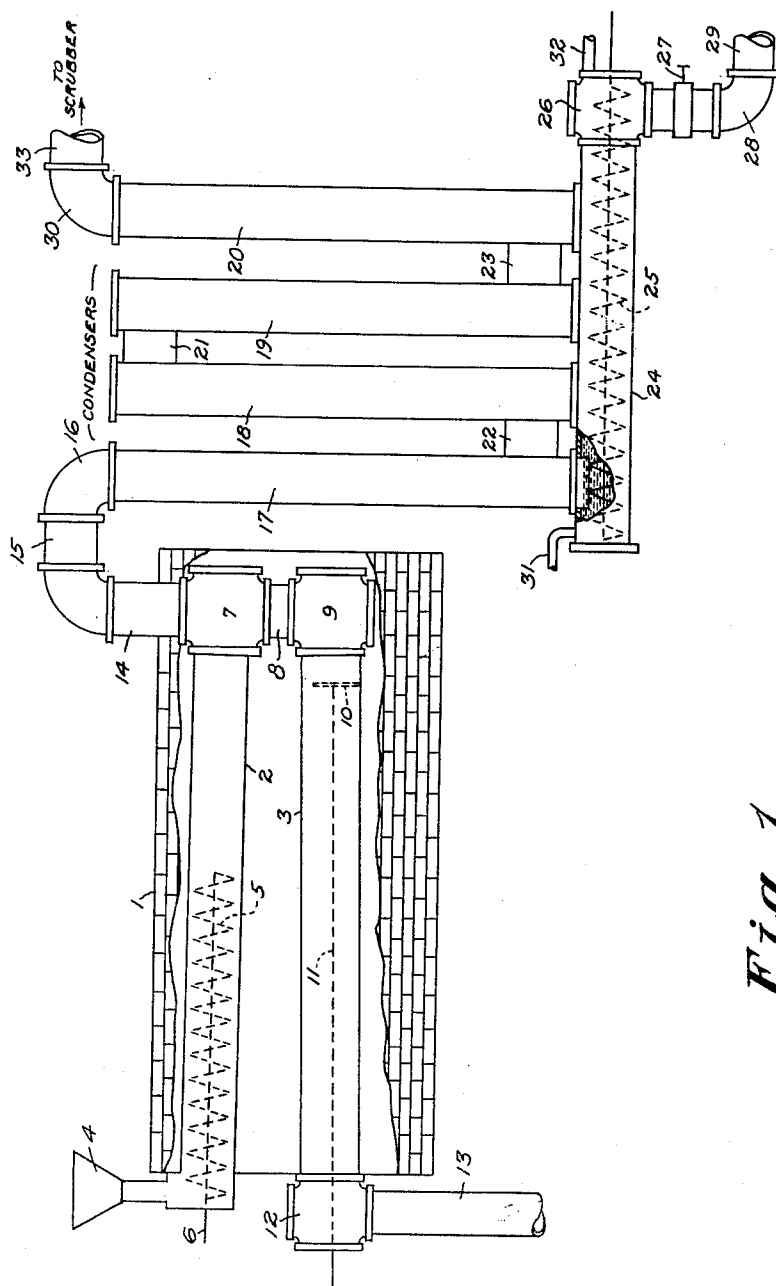


Fig. 1

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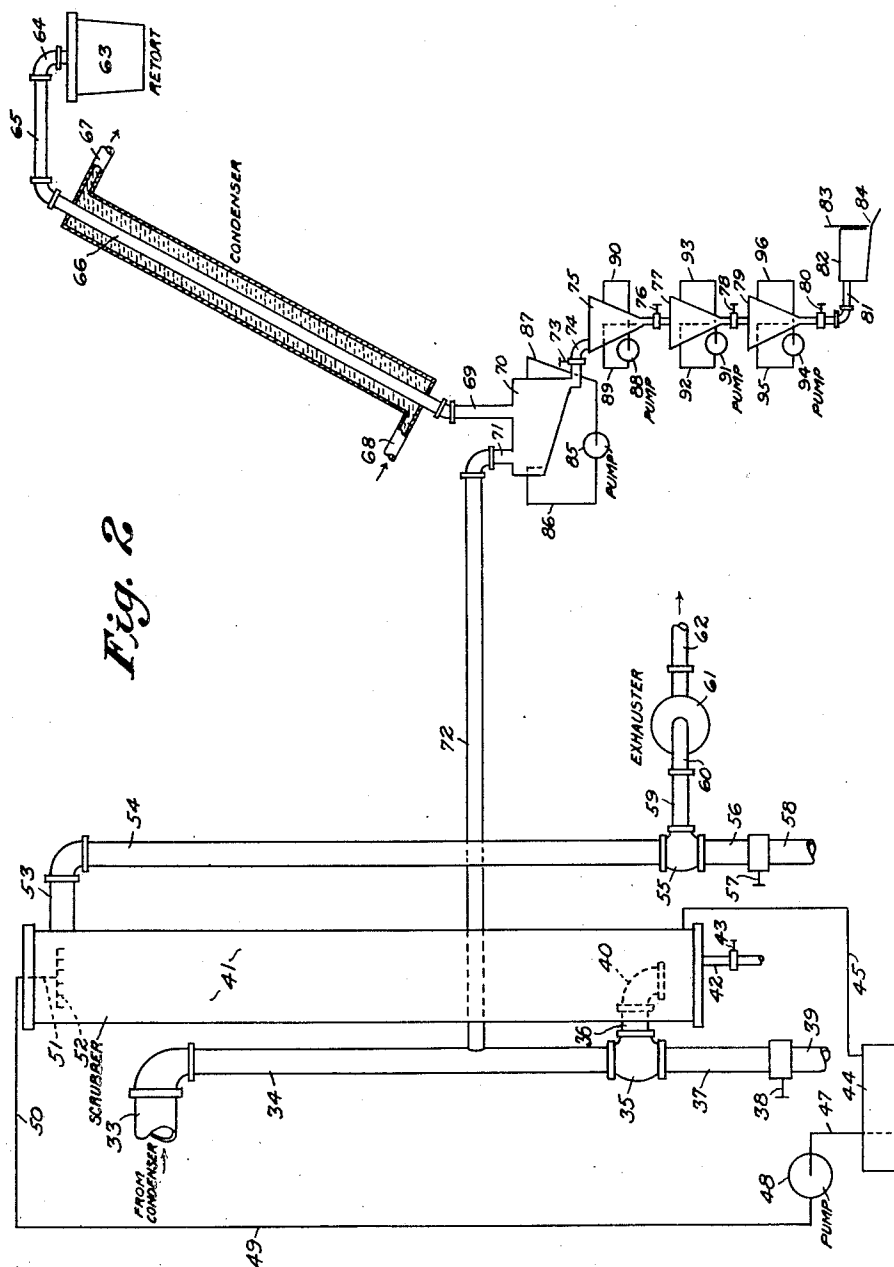
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2 Sheets-Sheet 2



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UNITED STATES PATENT OFFICE

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PROCESS OF AND APPARATUS FOR RECOVERING VOLATILE METALS

Application filed August 1, 1927. Serial No. 209,899.

This invention relates to the recovery of volatile metals from material containing the same and has for its object to provide a process and an apparatus for conducting said process which will be more efficient in operation and less costly of carrying out than those heretofore proposed.

With these and other objects in view the invention consists in the novel details of construction and arrangements of parts constituting the apparatus, as well as in the novel steps and combinations of steps constituting the process which will be more fully disclosed hereinafter and particularly pointed out in the claims.

Referring to the accompanying drawings forming a part of this specification in which like numerals designate like parts in all the views,—

Fig. 1 is a diagrammatic view illustrating an apparatus for vaporizing and condensing the raw material; and

Fig. 2 is a diagrammatic view illustrating an apparatus for purifying the volatile metals obtained as a result of condensing the same in such an apparatus as disclosed in Fig. 1.

In order that this invention may be the better understood it is said that there are many volatile metals of considerable value employed in the manufacture of articles of commerce and, further, much waste is encountered in said manufacture so that a considerable saving in costs could be had by carrying out the process such as hereinafter disclosed, under the operation of which said waste is recoverable. The process herein disclosed will be valuable in other ways as will be evident from the following, said value lying in the higher purity of product obtained, and the lessening of dangerous operating conditions especially in connection with the life and health of employees, and the people in the immediate vicinity of the recovery plant. Chief among the aforesaid valuable volatile metals are mercury, antimony, and arsenic, the recovery of all of which is accompanied by the abovementioned danger and relatively high costs. Further this process is carried out by simple and inexpensive apparatus and

is made continuous whereby there results a lesser handling of the material with consequent decrease in cost.

Referring to Fig. 1 there is disclosed a furnace 1 in the upper region of which is located a melting chamber which may be a pipe 2 and a vaporizing chamber therebelow which may be another pipe 3, both of said chambers inclined to the horizontal. The material is fed through the hopper 4 and moved by the conveyor 5, actuated in any suitable manner as through the shaft 6, to a point well within the chamber 2, said material being subjected in said chamber to a heat sufficient to cause the same to become more or less fluid. That is to say, the furnace 1 is or may be provided with any suitable source of heat which will create a temperature in the upper region thereof sufficient to melt or liquefy all or substantially all of the material therein so that the fluid material will pass into and through the chamber 2 due to the inclination of both chambers. In practice a temperature of about 500° F. has been found sufficient to liquefy said material.

The fluid material leaving the chamber 2 enters the connection 7 and then flows by gravity through the connections 8 and 9 into the elevated end of the chamber 3 where it is substantially completely vaporized by the increased temperature of the lower region of the furnace. In practice this temperature is about 1000° F. Should any carbonization of the material take place in the chamber 3 the same can be readily scraped or withdrawn therefrom as by the blade 10 secured to the end of an operating rod 11 passing out of said chamber, said carbon dropping to a suitable receptacle therefor as through the connections 12 and 13. By inclining the chambers 2 and 3 it will readily be seen that no positive means is necessary to convey the material there-through, the conveyor 5 serving to prevent a clogging of the chamber 2 at the hopper end, thus utilizing gravity to avoid unnecessary expense of operation. On the other hand, it is within the scope of this invention to provide separate sources of heat for the chambers 2 and 3, as well as to separate said chambers so that they are not necessarily contained in

the same furnace structure. Also a single chamber could be utilized, receiving two degrees of heat, the important feature being the treatment of the material with two temperatures, the first to liquify said material and the second to vaporize and/or decompose the liquified material.

The material in the chamber 3 is vaporized by the high temperature to which it is subjected and these vapors escape counter to the flow of the material, or in other words, pass out of the chamber 3 through the connections 9, 8 and 7, entering the conduit formed by the connections 14, 15 and 16 leading to the vertically disposed condensers, 17, 18, 19 and 20 which are joined in series as by the connections 22, 21 and 23 respectively. Any vapors given off from the material in the chamber 2 may readily pass along with the vapors from chamber 3 due to the fact that an exhaust pressure is maintained in the vaporizing and condensing apparatus. In other words, an exhaustor is employed, later to be described, which will insure a current in the above mentioned apparatus from the input end to, and through the entire length of, said condensers.

The condensers 17, 18 and 19 and 20 are not provided with closures at their bottoms but extend into a closed collecting main 24 provided internally with a suitable expelling device 25 for conveying the condensates out of said main as through the connections 26, 27, 28 and 29 into any suitable receptacle, not shown. On the other hand, these condensates may be led directly to and into a retort such as shown at 63 in Fig. 2 in order to make the process more continuous.

The main 24 is adapted to be filled with water or other suitable liquid entering and leaving the same as through the pipes 31 and 32 so located as to provide a liquid seal for the open bottom of each condenser. This liquid is prevented from passing out of said main along with the condensates in any suitable manner either through the employment of a valve in the connection 27 or simply due to the fact that the bottom of the main 24 is covered by the condensate containing the heavy volatile metal, which completely fills the outlet to connection 27.

With more particular reference to Fig. 2 the vapors and gases remaining after condensation leave the last condenser 20 as at 30 and pass through the connections 33, 34, 35, 36 and 40 to and into the lower portion of a scrubber 41, passing upwardly therein through any material such as gas coke adapted to furnish a large contact surface for scrubbing purposes. A suitable drain 42 having a valve 43 is provided at the bottom of said scrubbing tower, and a relief passage 37 having a valve 38 provides means for removing any sediments formed as by condensation in the passage 34 to the scrubber.

A supply tank 44 receives the scrubbing

fluid through a pipe 45 from the tower 41 from which it is drawn through the pipe 47 by the pump 48 and forced through the piping 49, 50 and 51 to the spray nozzle 52 disposed in the upper part of said tower, said nozzle being adapted to cause the fluid to be finely sprayed down and through the vapors and gases passing upwardly through the tower. The preferred absorbing liquid is a solution of caustic soda in water, but other solutions may be used within the scope of this invention, the purpose being to remove the obnoxious odors in the vapors. A fresh solution of the scrubbing fluid will of course be supplied as necessary. That is to say, one source of mercury such as to be recovered is found in vermilion which is found in substantial percentage in the barrels of fountain pens. In manufacturing said barrels there is considerable waste, and this process is peculiarly adapted to the recovery of the mercury in said waste from the rubber constituent. Therefore, it is very desirable to prevent the obnoxious rubber fumes from escaping to the atmosphere, hence the employment of the scrubbing tower.

After scrubbing, the residual vapors and gases leave the tower 41 through the connections 53, 54, 55, 59 and 60 being withdrawn therefrom by the action of an exhaustor 61. Said gases and vapors may be disposed of in any suitable manner, including conveyance as through the conduit 62 to the burners or fuel chamber of the furnace 1. Any residue material such as condensate may be withdrawn from the system as through the trap 56, controlled as by a valve 57, connected to a conduit 58 leading to any suitable receptacle.

The condensates from the collecting main 24 as hereinbefore stated are placed in a retort such as 63 at an elevated position where they are heated for redistillation, the vapors therefrom passing through the connections 64 and 65 to and into a condensing pipe 66 provided with a water jacket having the outlet 67 and inlet 68. The condensate then passes through the connection 69 into a closed receptacle 70 having an inclined bottom leading to the outlet provided with a control valve 73. This condensate contains the volatile metal to be recovered and any other substance that may have passed over with said metal. Should there be traces of rubber or other similar material in said condensate, the same is removed therefrom by a washing action suitable to dissolve the same or to put it into solution. For rubber it is found desirable to use xylol as a washing fluid but for other substances other chemicals may be found more desirable, none of said chemicals, however, having any action on the product being recovered. The particular washing fluid to be used is forced by a pump 85 through the pipe 86 into the receptacle 70 from which it escapes through the overflow

87 to be used over again if desired. Any vapors and gases uncondensed upon reaching the receptacle 70 may escape therefrom through the connections 71 and 72 leading to the connection 34 joining the last condenser 20 with the scrubbing tower, for the purpose of further treatment and disposition in accordance with the foregoing.

The impure volatile metal from the receptacle 70 is then passed through one or more further washing actions as found necessary, depending on the particular metal to be recovered. In the case of the recovery of mercury the impure metal is passed into a tank 75 for washing with sulphuric acid solution to remove organic and other impurities attacked by said acid. The washed metal then passes through the valved connection 76 into another tank 77 where the acid is neutralized with a solution containing sodium carbonate or caustic soda. Then the metal is passed through another valved connection 78 into still another tank 79 where the alkali is washed out with water. From this tank the metal then passes through the valved outlet 80 through the pipe 81 into a receiving tank 82 from which it may be drawn into suitable shipping containers. Associated with the tank 82 is a means of removing any physical impurities such as surface oxidation, scum or the like including any floating particles, said means conveniently being a vertically adjustable member in the nature of a blade or scraper for mechanically removing said impurities as the metal is withdrawn.

In these final washings the sulphuric acid solution is circulated by a pump 88 and connections 89 and 90. The sodium carbonate solution is circulated similarly as by the pump 91 and connections 92 and 93. And the last washing with water is accomplished by means of the pump 94 and connections 95 and 96. In each washing, as indicated in Fig. 2, the solution is admitted well below the surface of the metal in each tank so that said solution may bubble up through said metal and therefore better come in contact therewith, the outlet for each solution being in the nature of an overflow from each tank. A sight gage, not shown, may be provided on each tank 70, 75, 77 and 79, and only sufficient metal be admitted to each tank for best washing results, to be ascertained through trial.

It is obvious that those skilled in the art may vary the details of construction as well as the arrangements of parts constituting the apparatus, as well as varying the steps and combinations of steps constituting the process without departing from the spirit of the invention and therefore it is not desired to be limited to the foregoing except as may be required by the claims.

What is claimed is:—

1. The process of freeing chemically combined mercury from a matrix containing rub-

ber which consists in heating said matrix to a temperature sufficient to melt said rubber; submitting the rubber and the heated combined mercury to a higher temperature to completely vaporize the combined mercury; 70 subjecting the combined vapor to condensation to recover mercury and rubber as a condensate; subjecting the condensate to heat to vaporize the mercury and rubber; and washing the product to remove the rubber. 75

2. The process of separating mercury from articles containing the same in combination with rubber which consists in subjecting said articles to heat to melt the same; vaporizing the melt to obtain a combined vapor of 80 mercury and rubber; and recovering the mercury by condensing the same and washing to dissolve out the rubber constituent.

3. The process of separating mercury from articles containing the same in combination 85 with rubber which consists in subjecting said articles to heat to melt the same; vaporizing the melt to obtain a combined vapor of mercury and rubber; and recovering the mercury by condensing the same, dissolving the 90 rubber constituent therefrom, and washing the product to leave the mercury.

4. The process of separating mercury from articles containing the same in combination 95 with rubber which consists in subjecting said articles to heat to melt the same; vaporizing the melt to obtain a combined vapor of mercury and rubber and recovering the mercury by subjecting the combined vapor to condensation, a xylol bath to dissolve out the rubber, washing with acid, and neutralizing the wash. 100

5. The process of separating mercury from articles containing the same in combination 105 with rubber which consists in subjecting said articles to heat to melt the same; vaporizing the melt to obtain a combined vapor; and recovering the mercury by subjecting the combined vapor to condensation to recover mercury and rubber as a condensate, distillation 110 and recondensation of said condensate, a xylol bath to remove the rubber, acidification of the product to purify the mercury, neutralization of the acidified product, and washing to free the mercury. 115

6. In an apparatus of the class described the combination of means to receive and condense a combined vapor of mercury and rubber; a tank to receive said condensate and means to continuously circulate therethrough 120 a solution adapted to dissolve said rubber; means to convey the fumes from said tank for retreatment; and another tank for receiving the treated condensate, said tank provided with means to continuously circulate 125 therethrough a solution adapted to purify the rubber-freed mercury.

7. In an apparatus of the class described the combination of means to receive and condense a combined vapor of mercury and rub- 130

ber; a tank to receive said condensate and
means to continuously circulate therethrough
a solution adapted to dissolve said rubber;
means to convey the fumes from said tank for
5 retreatment; and a plurality of tanks for re-
ceiving the treated condensate said tanks pro-
vided with means to continuously circulate
therethrough solutions to successively acid-
ify, neutralize and wash with water the
10 rubber-freed mercury.

In testimony whereof I affix my signature.
BERNARD ORMONT.

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