Feb. 19, 1924. 1,484,260

H. D. GIBBS

APPARATUS FOR SUBLIMATION

Filed Jan. 24, 1919 2 Sheets-Sheet 1

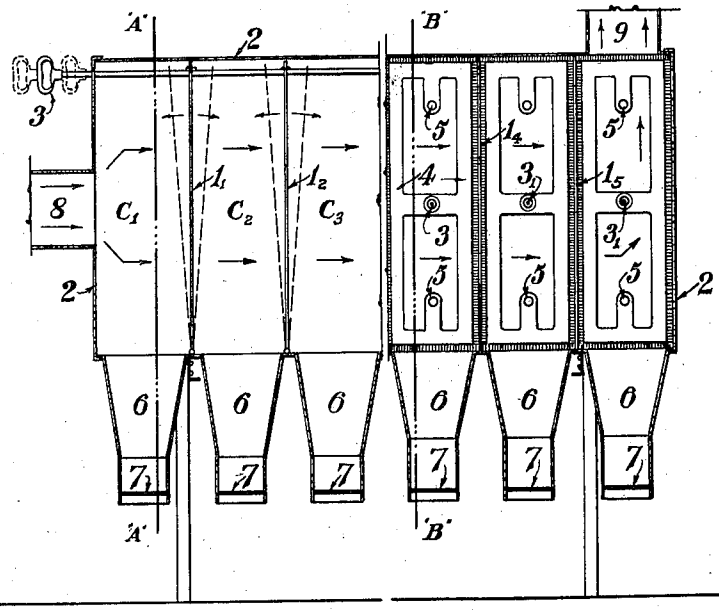


Fig. 3

Fig. 4

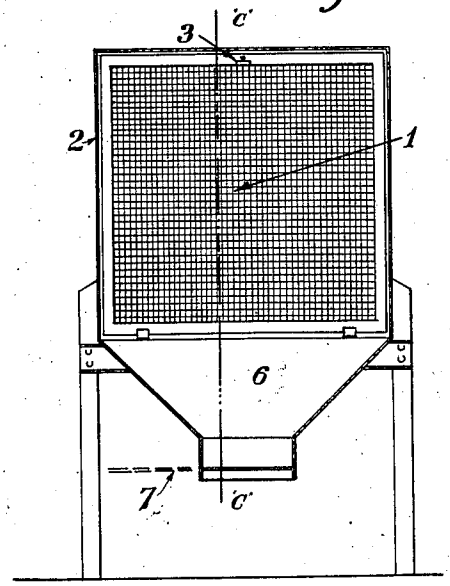


Fig. 1

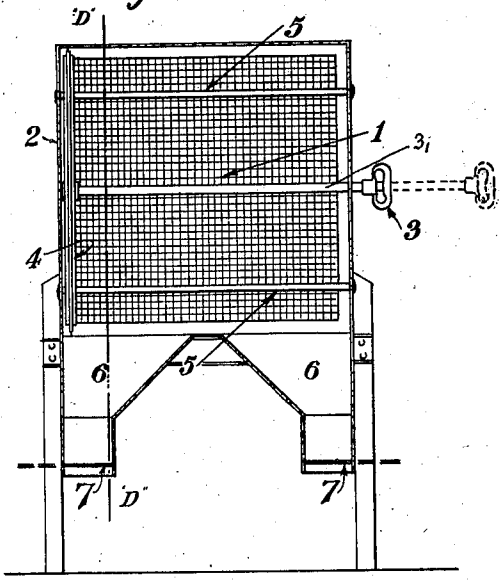


Fig. 2

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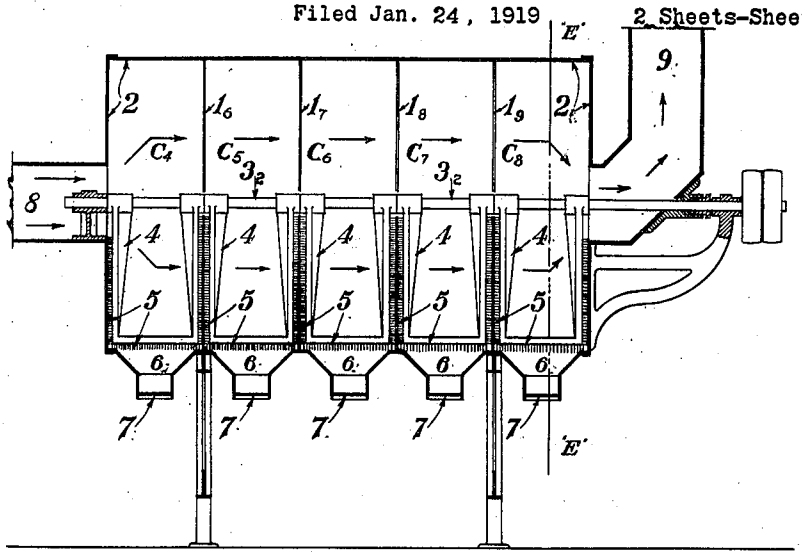


Fig. 6

Fig. 7.

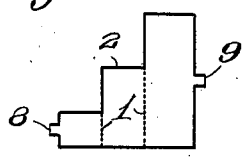


Fig. 5

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Patented Feb. 19, 1924.

1,484,260

UNITED STATES PATENT OFFICE.

HARRY D. GIBBS, OF SAN FRANCISCO, CALIFORNIA, BY MESNE ASSIGNMENTS DEDICATED TO THE PEOPLE OF THE UNITED STATES FOR THEIR FREE USE AND ENJOYMENT.

APPARATUS FOR SUBLIMATION.

Application filed January 24, 1919. Serial No. 272,902.

(FILED UNDER THE ACT OF MARCH 3, 1883, 22 STAT. L., 625.)

To all whom it may concern:

Be it known that I, HARRY D. GIBBS, a citizen of the United States of America, and an employee of the Department of Agriculture, residing in the city of San Francisco, county of San Francisco, State of California (whose post-office address is Department of Agriculture, Washington, D. C.), have invented a new and useful Improvement in Apparatus for Sublimation.

This application is made under the act of March 3, 1883, chapter 143 (22 Stat., 625), and the invention herein described and claimed may be used by the Government of the United States or any of its officers or employees in the prosecution of work for the Government or by any person in the United States, without payment to me of any royalty thereon.

The object of my invention is to purify substances that separate from a gas or mixture of gases upon cooling the gas or gases and separate substances that are suspended in the gas or are incorporated with the gas at elevated temperatures as part of the gas phase.

The usual method of so purifying such substances is to heat them to a high degree, producing vaporization, and conduct the vapors, either with or without a current of air, into a cooling chamber where the solid particles separate out. Or the gas is heated first and conducted over the substances to be vaporized and thus the gas becomes charged with the volatile substances. By this process all of the substances that separate from the gas condense in the chamber in a condition in which they are mixed together, either in the form of crystals or powder that settles to the floor of the chamber or collects upon the walls. In some cases a succession of chambers have been employed, these chambers connected with each other by large openings or doors situated at various places in the partition through which all of the vapors that escape from the first chamber pass into the second, and so on.

Reference is made to the drawing, wherein;—

Figures 1, 2, 3, and 4 represent the rectangular form of apparatus, and Figures 5 and 6 represent the cylindrical form of apparatus. Figure 1 is a cross section on line A—A of Figure 3; Figure 2 is a cross section on line B—B of Figure 4; Figures 3 and 4 represent longitudinal sections of the apparatus; Figure 5 represents a cross section of the apparatus; and Figure 6 represents a longitudinal section. Fig. 7 represents a vertical cross section of a modified type of the apparatus.

Further describing my invention with reference to the drawing wherein like numerals denote corresponding parts throughout: 1 denotes the screens, which may be made of metal cloth, fibers, perforated plates or other construction that provides a multiplicity of orifices, through which the gases are passed; 2 denotes the outside shell or casing; 3 represents a rod attached to the screens 1 in such a way that the screens can be oscillated or jarred for the purpose of removing adhering substances; 3_1, represents a rod attached to scrapers 4, arranged in such a way that by moving the rod 3_1, the scrapers 4 are dragged back and forth on guide rods 5, across the screens 1 dislodging material from the screens. The material dislodged from the screens 1 or walls falls to the bottom and collects in hoppers 6 where it is removed by drawing slides 7. The gases, charged with the substances to be separated, enter through the pipe 8, pass through the series of screens 1, and exit through pipe 9.

3_2 represents the shaft that revolves and carries the scrapers 4 that move against the screens 1 so as to dislodge material from the screens 1 and thus keep the orifices open. The scrapers 4 are fitted with bristles 5_1 of wire or other material forming a brushing surface. The scrapers 4 in their revolution loosen the material collected upon the screens 1 or circumference of the apparatus. This material falls to the bottom of the cylinder and collects in hoppers 6 where it can be removed by moving slides 7. The vapors carrying the materials to be separated enter through the pipe 8, pass through a series of screens 1, and exit through pipe 9.

To further illustrate the operation of my process as carried out with either one of

the above-described forms of apparatus, I will give the preferred procedure in the case of phthalic anhydride.

A current of gas, consisting of atmospheric air through gases, nitrogen, carbon dioxide or similar gases, is passed over or through melted phthalic anhydride, whereby the gas becomes charged with the phthalic anhydride in the vapor phase, the amount of phthalic anhydride carried by the gas being dependent upon the temperature of the gas, which should be above 150° centigrade. This gas, charged with the vapors of phthalic anhydride, is passed into the apparatus through the opening 8 and successively into the compartments separated from each other by the series of screens. The first compartment, C_1 in Figure 3, is maintained by the heated gas stream at a temperature of approximately 100° centigrade. In this compartment a certain amount of heat is lost from the gas and the least volatile substances crystallizing out upon the screens and upon the walls of the apparatus. The gas stream flows through the screens 1_1 into compartment C_2, where a further quantity of heat is lost from the gas and solid substances crystallize out upon the screens 1_1 and 1_2 and the walls of this compartment. A further quantity of heat is lost from the gas in this compartment and it passes through the screens 1_2 into the compartment C_3, where it is further cooled and other solid substances crystallize. The exit gases are carried off through the outlet 9, shown in Figure 4. When the material crystallizing upon the screens tends to clog the orifices, the handle 3 can be moved to oscillate or jar the screens and remove the adhering material. In Figure 4 the rakes 4 moving across the screens 1 are moved by handle 3. The material as it flows from the screens in the various compartments collects in hopper 6, where it is removed by drawing slides 7.

The same procedure is employed with the cylindrical apparatus described in Figures 5 and 6. The gases charged with phthalic anhydride enter at the orifice 8 and pass through the screens 1_6, 1_7, 1_8, 1_9, into the first compartment C_4, where a certain amount of cooling takes place with the deposition of some of the phthalic anhydride. The gases then pass through screens 1_6 into the compartment C_5 where a further cooling and precipitation takes place, and so on successively into the compartments C_6, C_7, C_8, through screens 1_7, 1_8, 1_9.

The phthalic anhydride ordinarily manufactured by the air oxidation process is contaminated by naphthalene and the separation of these two constituents is very easily effected by this apparatus, the phthalic anhydride separating out into pure form in the first compartment, while the naphthalene separates out in the latter compartments.

In describing my process I shall first give an example describing the steps of the complete process, it being understood that while all of the steps are not novel, they have not been heretofore employed in connection with the novel steps, for which reason they have not accomplished the results obtained in the practice of my invention.

When a current of gas is passed over or through mixtures with various volatile compounds the gas will take up the volatile substances, and the proportion of volatile substances taken up will depend upon the nature of the substances, the gas, and the temperature, both of the substances themselves and of the gas. On cooling the gas some of the constituents separate out, the substances having the highest melting, sublimation and boiling points usually separating out first.

I have found out that when the current of gas is charged with these volatile substances and it is caused to move through small orifices, the least volatile constituents separate or congregate or crystallize on the edges or adjacent to the orifices. Crystallization and separation from the vapor phase is thus assisted by the orifices and the passage of the gas through the orifices. I do not wish to confine myself to the use of any particular gas, for I have found atmospheric air, furnace gases, flue gases, carbon dioxide, water vapor, and various other gases may be advantageously employed.

The orifices may be arranged in large numbers in approximately the same plane in a sheet, plate or screen composed of metal, fiber or other substances, or the aggregation of orifices may be attained by the use of gauze, screen or cloth woven of wire or spun fibers, or any material producing a partition permeable to gases.

I have also found that a multiple arrangement of these planes of orifices on the plan of connecting rooms or compartments afford the additional advantage of forming a temperature gradient varying from the highest temperatures where the stream of heated gases enters, to the lowest temperature of the farthest compartment, the drop in temperature of the gases being quite sharp near the point where the gases pass through the orifices or just after passing through the orifices.

I do not wish to confine myself to the use of these orifices or screens in multiple in plane surfaces, but these orifices may be arranged in circles or in segments of circles, in spheres or parts of spheres, concentrically, or otherwise, or in any figure of revolution, or warped or bent surfaces or any form of a partition constructed of material permeable to gases. I have found that one arrangement of orifices possesses advantage

over other arrangements depending upon the character and state of the material that is being separated and also depending upon the size and capacity desired in the apparatus.

The air current may be charged with the volatile constituents in various ways, for example, by heating the material in a vessel and passing the air over or through the heated material; by feeding the solid or molten material into the air stream or passing the air stream over or through baffles containing the material to be volatilized. The air stream thus charged is passed into the first compartment where it loses a portion of its heat and precipitates a portion of the materials carried. From the first compartment it passes through the orifices, screens or permeable partition into the second compartment, where the process of cooling and precipitation of particles continues, and from this compartment it may be passed through another series of orifices or screens or a permeable partition into a third compartment and so on into, and through, any number of compartments desired.

The material collecting on the screen or on the orifices may be removed by shaking or jarring the screens or by scraping or brushing by hand intermittently or continuously by well known mechanical device. The material falling from the screens and condensing in the various compartments may be removed from time to time by means of chutes in the bottom of the compartment or it may be scraped from the floors of the compartment through openings placed in the side or bottom walls.

To further illustrate my process I will give the preferred procedure in the case of phthalic anhydride. In the manufacture of this compound by the catalytic air oxidation process the phthalic anhydride may be contaminated by greater or lesser quantities of naphthalene, naphthaquinones and other substances. The gas stream containing these substances is made to enter the first chamber at a temperature of approximately 120° C. The temperature of this chamber is maintained at a range between 80° C. and 120° C. The permeable partition separating this chamber from the second is made of wire cloth of mesh that may be one-quarter of an inch to one inch in size. I do not wish to confine myself to wire mesh of this size for in some instances larger or smaller mesh may be desirable. The rate of the gas stream passing through this permeable partition may be approximately three cubic feet of gas per minute for each square foot of surface in the screen separating the first and second compartments. In some cases the gas stream may be greatly increased with corresponding increase in the amount of the substances purified in a given time. Large masses of phthalic anhydride crystals separate and form on the screen. These may fall to the floor of the compartment or may be shaken or scraped from the screen and from time to time removed from the compartment.

Impurities of various nature, including naphthalene and naphthaquinone and some pthalic anhydride, pass with the air stream into the second compartment, which is preferably larger than the first and separated from the third compartment by a similar screen or permeable partition. In the second compartment an impure phthalic anhydride condenses and in the third compartment the larger proportion of the naphthalene and some phthalic anhydride condense. If the air current is very rapid and the temperature of the third compartment remains too high to insure a good condensation, a fourth or a fifth compartment may be provided, each increasing in size over the preceding, thus affording more rapid cooling and slowing the rate of passage of the gas through the screens.

As an example of some of the useful applications of this process, I will cite—

1. The separation of phthalic anhydride from naphthaquinone, toluic acid, naphthalene and other impurities.

2. The separation of anthracene pressed cake into its various constituents whereby carbazole, anthracene, and phenanthrene are obtained in a higher state of purity.

3. Benzoic acid from chlorbenzoic acid and other impurities.

4. Salicylic acid from impurities.

5. Beta-naphthol from impurities.

6. Naphthalene from impurities.

7. Iodin from impurities.

8. Sulphur from impurities.

9. Arsenious oxide from impurities.

10. Anthraquinone from impurities.

11. Phenanthraquinone from impurities.

Having now fully described my invention, what I claim and desire to protect by Letters Patent is—

1. In condensing mechanism of the character set forth, the combination with a casing, of means for introducing vapors to be condensed into the casing, means for permitting the escape of the residues, screens in the casing between the introducing and escape-permitting means, brushes acting against the screens, means for effecting a complete relative rotation between the screens and brushes, and means operating over the bottom between the screens for separately collecting the materials condensed and deposited in the different compartments formed by the screens and separately discharging the same from the casing.

2. In condensing mechanism of the character set forth the combination with a casing, of means for introducing vapors to be

condensed into the casing, means for permitting the escape of the residues, screens in the casing between the introducing and escape permitting means, brushes acting against the screens, means for effecting a relative movement between the screens and brushes, and means operating over the bottom between the screens for separately collecting the materials condensed and deposited in the different compartments formed by the screens, and separately discharging the same from the casing.

3. In condensing mechanism of the character set forth the combination with a casing, of means for introducing vapors to be condensed into the casing, means for permitting the escape of the residues, screens in the casing between the introducing and escape permitting means, and means for removing the condensed materials from the screens and means for separately collecting the materials condensed and deposited in the different compartments formed by the screens, and separately discharging the same from the casing.

4. In condensing mechanism of the character set forth the combination with a casing, of means for introducing vapors to be condensed into the casing, means for permitting the escape of the residues, screens in the casing between the introducing and escape permitting means, means for removing the condensate from the screens, and means operating over the bottom between the screens for separately collecting the materials condensed and deposited in the different compartments formed by the screens, and separately discharging the same from the casing.

5. In apparatus of the character set forth, the combination with a casing, of means for introducing thereinto in the vapor phase and at a temperature sufficiently high to maintain the least volatile materials in said vapor phase, a mixture of materials that respectively condense under different temperature conditions, outlet means for permitting the outflow of the residues at a separate point from the introducing means, said casing having heat-radiating walls for effecting a cooling action upon the interior of the casing and combining with the vapor therein to form zones of different temperatures at different points, a plurality of screens in the casing located at different points across the path of the vapors between the introducing and outlet points and substantially defining said different zones, means for dislodging the materials from the screens and causing the same to be individually deposited at different points, and means for permitting the separate individual withdrawal of the condensed materials from the casing.

6. In apparatus of the character set forth, the combination with a casing having portions of consecutively increasing capacity, of means for introducing materials of different characteristics in the vapor stage into a portion of relatively small capacity, means for effecting the separate condensation of such materials, respectively, in the said different portions of the casing, and means permitting the discharge of the residue from a portion of relatively great capacity.

In testimony whereof, I affix my signature in the presence of two subscribing witnesses.

HARRY D. GIBBS.

Witnesses:
L. A. SKINNER,
COURTNEY CONOVER.