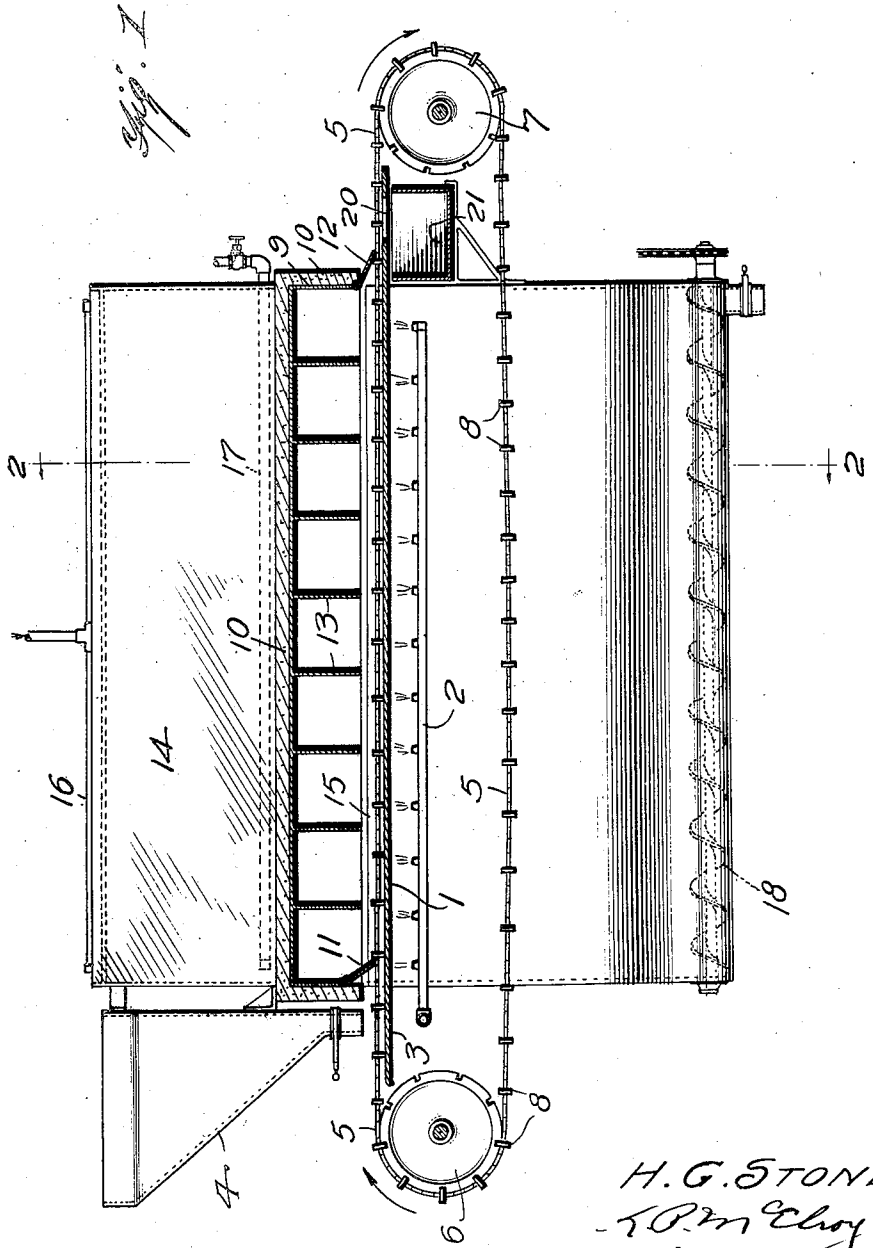
Sept. 11, 1928. 1,683,931

H. G. STONE

SUBLIMING APPARATUS

Filed June 2, 1922 2 Sheets-Sheet 1

Fig. 1.



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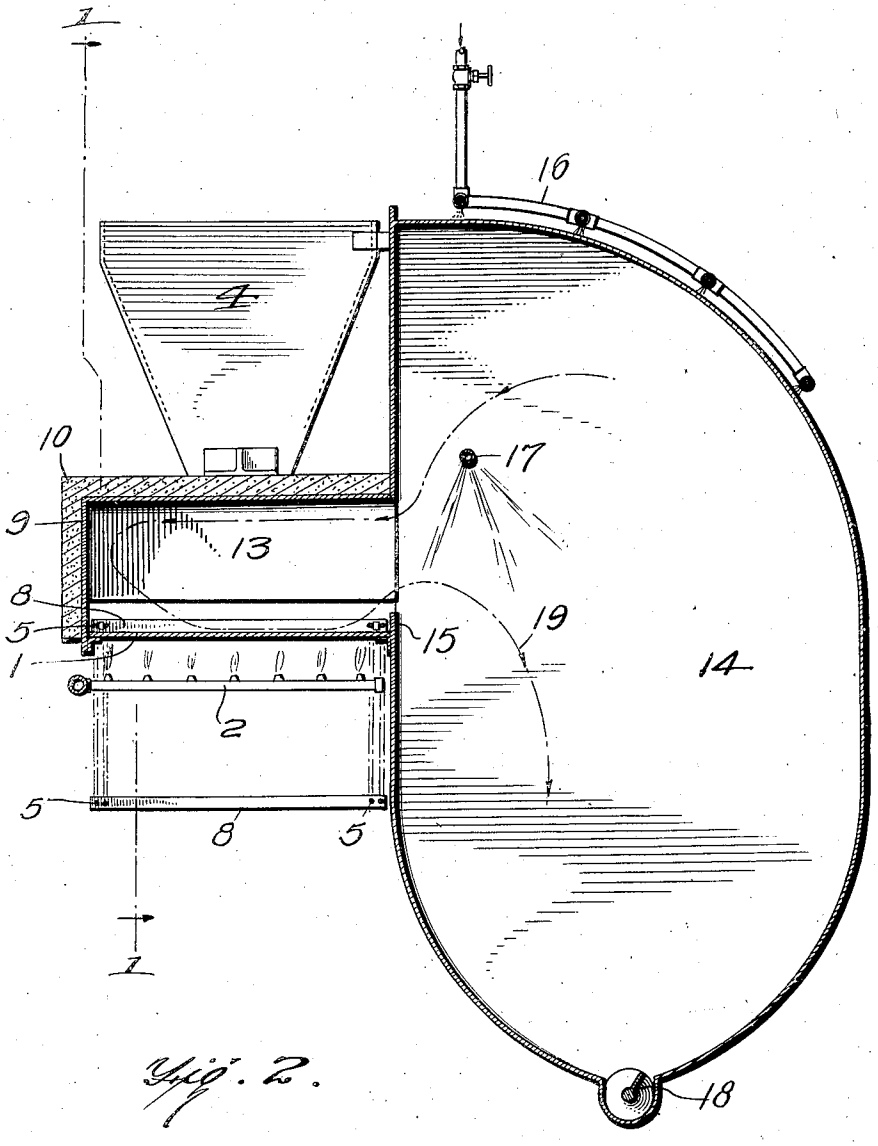


Fig. 2.

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Patented Sept. 11, 1928. 1,683,931

UNITED STATES PATENT OFFICE.

HERBERT G. STONE, OF BRISTOL, TENNESSEE.

SUBLIMING APPARATUS.

Application filed June 2, 1922. Serial No. 565,448.

This invention relates to subliming apparatus; and it comprises a relatively long heated plane surface, regulable means for imparting heat thereto, conveyor means for passing pulverulent material thereover from end to end, a vapor chamber enclosing said surface, a series of transverse depending baffles in said chamber and a subliming chamber laterally communicating with said vapor chamber and extending below the level of said heated plane surface, adapted for the reception and removal of sublimate; all as more fully hereinafter set forth and as claimed.

The present invention relates to an apparatus for efficiently and economically subliming solids from materials containing (or producing) the same and is particularly, although not exclusively, designed for the recovery and purification of volatile solid carbon compounds or "organic compounds", such as anthraquinone, anthracene, naphthalene, camphor, etc. etc. Like other volatilization operations, sublimation requires a considerable amount of heat at a definite temperature; the temperatures required being sometimes quite near those at which the substance to be sublimed alters or decomposes. It is therefore desirable in sublimation to be able to supply a relatively large amount of heat within a limited temperature range. The temperature must of course be above the subliming point but it must also be below any point dangerous to the substance to be sublimed or to materials associated therewith.

In the present invention, the material is treated as a relatively thin traveling layer or film passing along a plate with its upper surface kept at the subliming temperature or a few degrees above; the length (or time) of travel being sufficient to enable the delivery of the necessary number of thermal units at this temperature to the material under treatment. The vapors evolved are allowed to pass into a lateral chamber for condensation. Commonly, I make this chamber of rather large cross-section and, as these vapors are usually rather heavy and tend to fall rather than rise I give it a downward extension into which vapors can flow, considerably below the level of the hot plate. Advantageously, it also extends above this level for some distance. I provide a hopper or the like at the bottom of the downward extension. This may be provided with conveying means. With such a chamber, a considerable volume of gas is afforded to mix with and quickly cool the inflowing vapors to produce condensation thereof. Radiation from the chamber walls is usually sufficient to dispose of the heat taken up by the gaseous contents; although positive wall cooling means may, in some cases, be provided. A simple method of affording the additional cooling, in many cases, is to spray the walls with water. Sometimes it is necessary to provide more or less heat within the chamber to prevent premature condensation; this being particularly the case with vapors forming only at rather high temperatures. This may be done by the introduction of steam, which may be superheated. Such an introduction of steam also offers a means for diluting the vapors somewhat and thereby making the operation more controllable. Sometimes, however, in dealing with high temperature vapors a little heat insulation on the subliming chamber is all that is necessary.

In the accompanying illustration I have shown more or less diagrammatically, one embodiment of the described invention. In this showing

Fig. 1 is a longitudinal vertical section through the sublimation chamber along line 1—1 of Fig. 2 with certain accessory parts and parts of the condensation chamber shown in elevation, and

Fig. 2 is a transverse section through the subliming chamber and condensation chamber along line 2—2 of Fig. 1.

Referring first to Fig. 1, element 1, as shown, is a relatively long smooth surfaced plate of any convenient material, such as cast iron or steel. It is shown as heated by a bank of burners 2. Any convenient regulable means of heating, however, may be employed, such as electrical heating, circulating hot oil, furnace gases, etc. Material moves along this plate. As shown, it moves from left to right. At the entrance is an unheated extension 3 of the plate upon which material can be deposited by any suitable feeding means, here shown as hopper 4. Any convenient mechanical feeding means adapted to deposit material on 3 as a relatively uniform layer, usually rather thin, may be used. Passing over the surface of the plate is a conveyor, here shown as a drag means 5, passing over wheels 6 and 7 and returning through the base of the apparatus. The drag elements can be of any desired type. As shown it carries transverse blade members 8. If these be of a plow-like form the speed of the

conveyor may be considerably greater than that of the material passing through the apparatus; and in this event, the conveyor has the additional function of disturbing the traveling layer of material and constantly exposing fresh surfaces for release of vapors. Enclosing the heated plate is vapor subliming chamber 9. As shown, it is provided with heat insulating means 10 and with swinging gates 11 and 12 at the two ends, these gates riding upon the conveying means and serving to prevent drafts. Usually, the operation is so controlled as to maintain about the normal atmospheric pressure within both chambers, giving no tendency either for an indraft or an outdraft. Within the vapor chamber, as shown, are provided a plurality of depending baffles 13 breaking it up into a plurality of sub-chambers and decreasing any tendency towards longitudinal gas and vapor movements. Referring now to Fig. 2, the vapor chamber, as shown, has connected with it condensation chamber 14. As shown this is made of metal plates. A low longitudinal lateral bridge-wall or dam 15 along the side of the subliming chamber prevents solid matter carried forward by the drag from entering the condensation chamber. Vapors arising from the hot material overflow this lateral dam member and sink downward in the lateral condensation chamber. As shown, spray cooling means 16 may be provided for assisting in the cooling of the gaseous contents of this chamber. Inlet means 17 may be provided for admitting steam or other gas or vapor into the condensation chamber. At the bottom of the condensation chamber is shown conveyor means 18 for removing condensed solids.

Presuming the apparatus so far shown is to be used for recovering anthraquinone from associated tarry matter and impurities in crude anthraquinone, the material to be treated is delivered on the unheated extension 3 of the hot plate and is carried forward within the chamber by the forward movement of the conveyor drags 8. Anthraquinone boils at 382° C. and sublimes at temperatures considerably below this. Presuming that it is to be sublimed at, say, 350° C., and that because of the presence of impurities or for other reasons the material cannot be safely heated above, say, 370° C., the hot plate may be heated to a temperature of 350° to 360° C. As the crude anthraquinone moves forward over the plate the drag constantly exposes fresh surfaces for volatilization and anthraquinone sublimes in proportion to the heat delivered to the material from the hot plate. The vapors which are very heavy do not tend to rise far within the subliming chamber which, it may be noted, is desirable to have, as shown, rather low. These vapors tend, on the other hand, to flow laterally across bridge wall 15 into condensation chamber 14 where they mix with the gases present and are cooled thereby, producing solid anthraquinone which falls to the bottom of chamber 14. The hot gases separated from the solid sublimate rise in the chamber and are cooled by its walls. There is to a greater or less extent, a gas circulation within the vapor chamber as indicated by the arrow 19. The crude anthraquinone is given a sufficient time of exposure to the heat in the vapor chamber to insure that the material delivered at the right hand end is substantially free of volatilizable matters. The spent material falls through opening 20 in plate 1 into waste receiving means 21.

The operation of the device is exactly the same, save for the temperatures to be maintained, in subliming camphor, naphthalene, anthracene, etc. The apparatus may also be used for inorganic sublimable material, such as corrosive sublimate, sal ammoniac, etc.

The vapor chamber and condensation chamber may be filled with suitable inert vapors or gases, nitrogen, steam, products of combustion, etc. where this is desirable. As a rule, however, the materials treated evolve enough moisture, gases, etc. to furnish their own inert atmosphere.

What I claim is:

1. In a subliming apparatus, a plate-like heating member having a low lateral dam thereon, conveying and distributing means passing longitudinally thereover, regulable heating means therefor, a vapor chamber above the heating member and a condensation chamber in free lateral communication with the vapor chamber above the dam.

2. Subliming apparatus comprising a vapor chamber, means for heating a horizontal surface therein, a lateral dam on said surface, means for moving and distributing the material to be heated longitudinally over said heated surface and a condensing chamber in free lateral communication with the vapor chamber, said condensing chamber being of substantially greater capacity than the said vapor chamber.

3. Subliming apparatus comprising a long narrow vapor chamber, means for heating a horizontal surface therein, a lateral dam on said surface, means for moving and distributing the material to be heated longitudinally over said heated surface, and a condensing chamber in free lateral communication with the vapor chamber, said condensing chamber being substantially co-extensive with said vapor chamber and in communication therewith throughout its length but of relatively greater capacity.

4. Subliming apparatus comprising a vapor chamber, means for heating a horizontal surface therein, a lateral dam on said surface, means for moving and distributing the material to be heated longitudinally over said heated surface, and a condensing chamber in

free lateral communication with the vapor chamber, said condensing chamber being of substantially greater capacity than the said vapor chamber and provided with exterior cooling means.

5. Subliming apparatus comprising a vapor chamber, means for heating a horizontal surface therein, a lateral dam on said surface, means for moving and distributing the material to be heated longitudinally over said heated surface, and a condensing chamber in free lateral communication with the vapor chamber, said condensing chamber being of substantially greater capacity than the said vapor chamber and provided with means for admitting fluid interiorly thereof.

6. Subliming apparatus comprising a plate-like heating member, conveying and distributing means passing thereover, heating means therefor, a vapor chamber above the heating member, said vapor chamber being relatively long and narrow, and a condensing chamber in lateral communication with the vapor chamber throughout its length.

7. Subliming apparatus comprising a vapor chamber, means for heating material therein, means for conveying material to be heated therethrough, a condensing chamber in communication therewith, and baffles in the vapor chamber preventing substantial passage of sublimed vapors longitudinally therein.

8. Subliming apparatus comprising a plate-like heating member, conveying means passing thereover, heating means therefor, a vapor chamber above the heating member, a condensation chamber in lateral communication with the vapor chamber and extending above and below said vapor chamber, and means in the condensation chamber for the conveying and removing of condensed solids.

In testimony whereof, I have hereunto affixed my signature.

HERBERT G. STONE.