No. 651,942. Patented June 19, 1900.

J. WHEELER.

PROCESS OF MAKING PYROBETULIN.

(Application filed Dec. 30, 1897.)

(No Model.) 3 Sheets—Sheet 1.

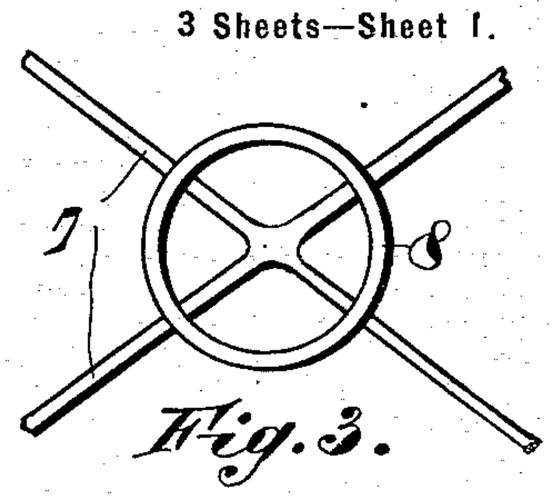

Fig. 3.

Fig. 1.

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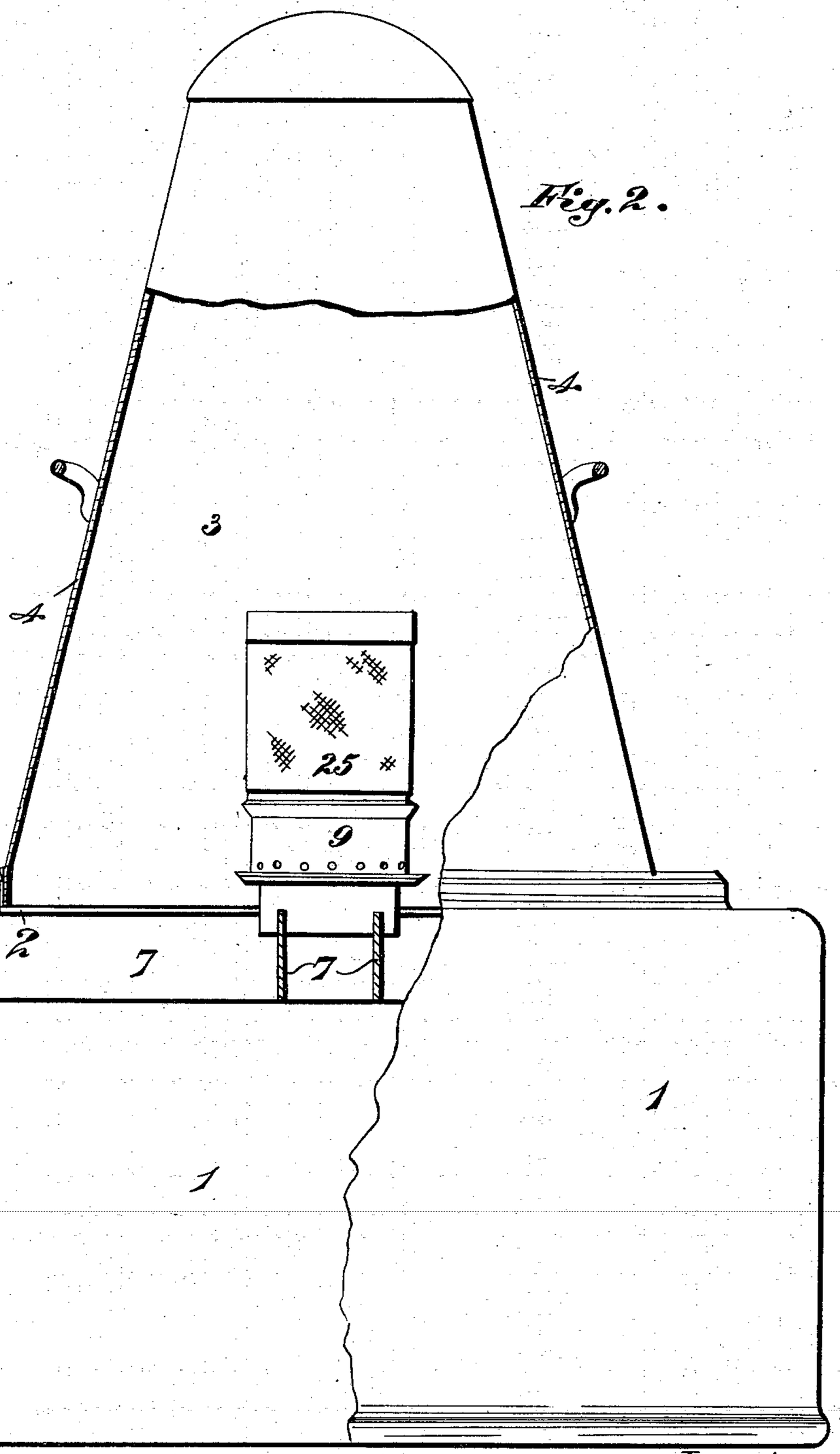
(No Model.) 3 Sheets—Sheet 2.

Fig. 2.



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(No Model.) 3 Sheets—Sheet 3.

Fig. 4.

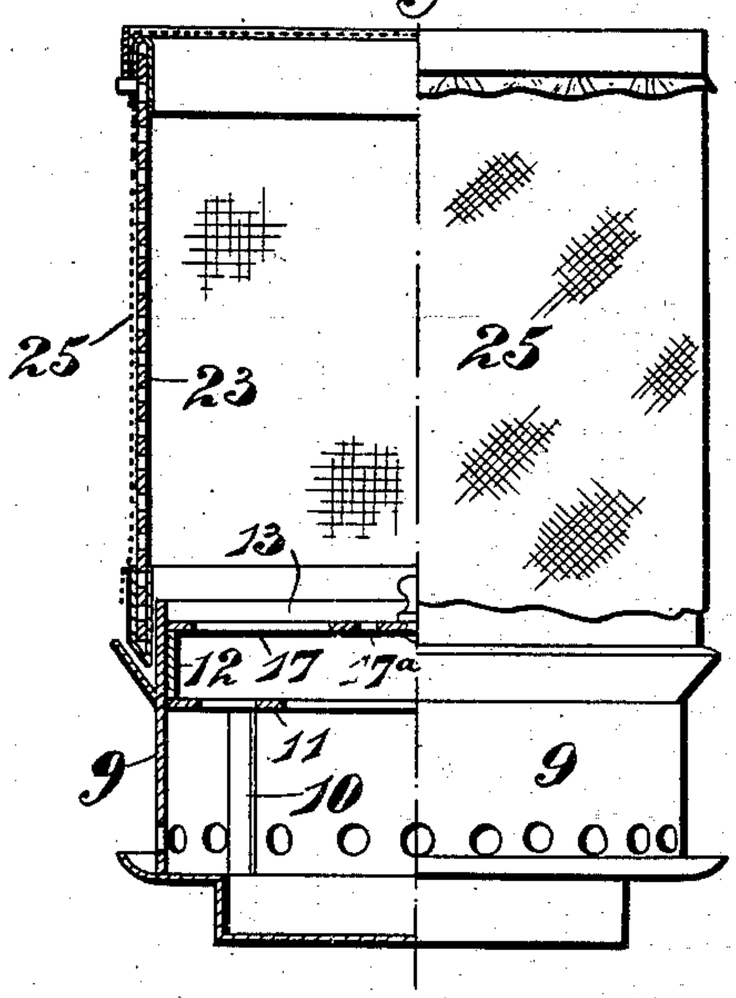


Fig. 5.

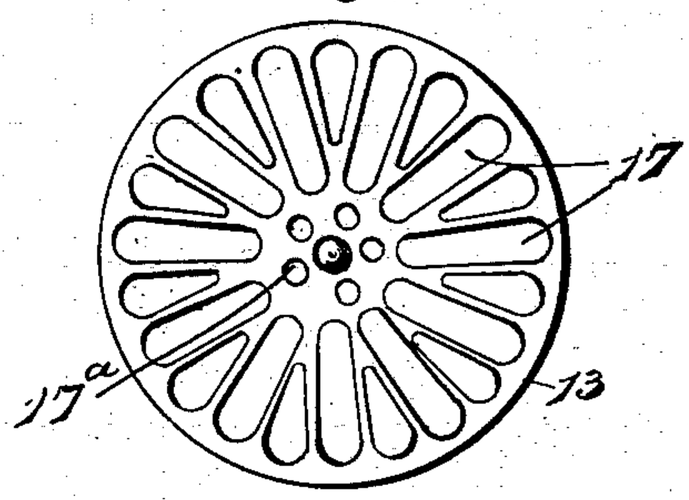


Fig. 7

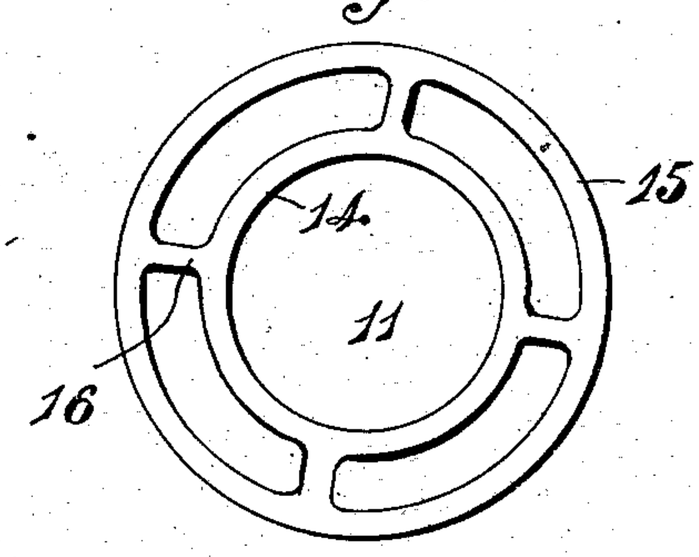


Fig. 6.

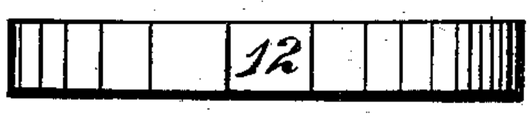


Fig. 8.

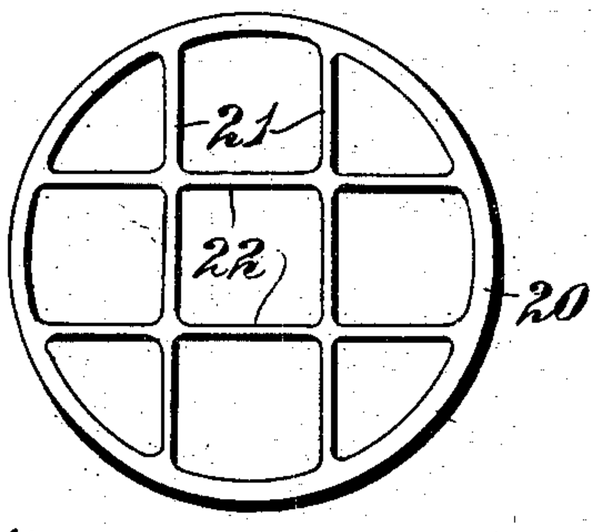
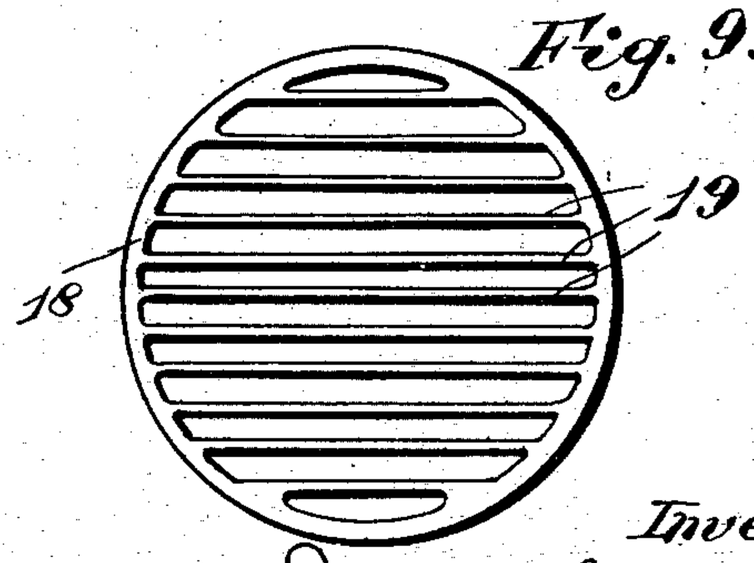


Fig. 9.



Witnesses.
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UNITED STATES PATENT OFFICE.

JAMES WHEELER, OF ILFRACOMBE, ENGLAND.

PROCESS OF OBTAINING PYROBETULIN.

SPECIFICATION forming part of Letters Patent No. 651,942, dated June 19, 1900.

Original application filed June 16, 1897, Serial No. 641,041. Divided and this application filed December 30, 1897. Serial No. 664,758. (No specimens.)

To all whom it may concern:

Be it known that I, JAMES WHEELER, a subject of the Queen of Great Britain and Ireland, residing at Ilfracombe, in the county of Devon, England, have invented an Improvement in the Manufacture or Production of Pyrobetulin or Pyrobetulin Anhydrids, of which the following is a specification.

In the specification of other Letters Patent granted to me, dated September 20, 1898, and numbered 611,047, I have described and claimed a method and apparatus for producing films. Now this application, which is a division of the said application, relates to the manufacture or production from birch-bark or the like of certain substances hereinafter called "pyrobetulin" or "pyrobetulin anhydride"—*i. e.*, betulin ($C_{36}H_{60}O_3$) or betulin anhydride ($C_{36}H_{58}O_2$) (*vide Watts' Chemistry*, 1888 edition) or products analogous thereto, obtained by burning and consequent dry distillation. According thereto pyrobetulin or pyrobetulin anhydride is produced by burning in a closed chamber without flame and in a current of air material containing betulin to an amount bearing a suitable ratio to the capacity of the said chamber. In order to obtain pyrobetulin, the material is burned with an abundant air-supply, whereas to obtain pyrobetulin anhydride the material is burned with a restricted air-supply, with the result that in the former case there is burned away the cortical investment of the betulin of the bark and in the latter case the pyrobetulin thus produced is converted into its anhydride by the more prolonged heating and slower evolution of the fumes.

In carrying my invention into practical effect I may employ the combustible material in the form of tablets or small blocks, (hereinafter called "tablets,") made by compressing together a mixture comprising finely-ground epidermis of birch-bark, preferably that of white birch or *betula alba*, and an oxygen-carrier, such as nitrate of potassium. The percentage of nitrate of potassium in the material may vary. For the production of pyrobetulin the percentage may vary from four to eight, and for producing pyrobetulin anhydride it may vary from one to four. When less than two per cent. of nitrate of potassium is used, the material should be dried for a considerable time both before and after compression. In the case of high percentages of nitrate of potassium it is advisable, in order to lessen the tendency of the charge (when burning) to burst into flame, to dissolve the nitrate of potassium either in plain water or in a weak solution of dextrin, so as, for example, to give one per cent. of dextrin to the amount of bark used. In order to produce pyrobetulin, the tablets are arranged so as to give a space around each to allow of the free passage of an ample supply of air, whereas to produce pyrobetulin anhydride the tablets are placed near together, so as to insure a restricted passage of air, whereby a slower combustion will be attained than with the former arrangement.

Figures 1 and 2 of the accompanying drawings are sectional views taken at right angles to each other of an apparatus suitable for producing pyrobetulin or pyrobetulin anhydride according to this invention. Fig. 3 is a partial plan representing a support for a lamp. Fig. 4 shows the lamp half in vertical section and half in elevation. Figs. 5, 6, and 7 are respectively a plan of a charge-plate, an elevation of an annular distance-piece, and a plan of a floor-plate used in producing pyrobetulin; and Figs. 8 and 9 are respectively plans of a charge-plate and a floor-plate used in producing pyrobetulin anhydride. Figs. 4 to 9, inclusive, are drawn to a larger scale than the other figures.

The apparatus shown comprises a chamber 1, of sheet-zinc, which has in its top an opening 2, preferably of circular shape, through which it communicates with an upper compartment 3, formed by a conical cover 4, that fits upon the top of the chamber 1, forming therewith around the opening 2 an air-tight joint by means of an india-rubber ring 5 and a flange 6 or other suitable means. The bottom of the chamber 1 may be closed in a sufficiently air-tight manner by placing it upon a thick cloth or other compressible material. At the center of the opening 2 there is supported from the top of the chamber 1, by means of bars 7, (see Fig. 3,) soldered or otherwise secured thereto, a suitable stand 8 for a lamp in which to burn the tablets.

This lamp may, as shown, comprise a cylindrical cup 9, (see Fig. 4,) perforated, as shown, at its lower part to admit air and adapted in its upper part to support, by means of brackets 10, a floor-plate 11, on which is placed a ring or distance-piece 12, (see also Fig. 6,) which supports a charge-plate 13, in which the tablets are arranged with their lower edges resting more or less on the floor-plate 11. The lower portion of the cup 9 may be surrounded by a ring (not shown) having apertures in it corresponding to the perforations in the cup, so that by slightly turning the ring on the cup the supply of air passing through the perforations of the cup may be accurately adjusted.

To produce pyrobetulin, the floor-plate may advantageously consist, as shown in Fig. 7, of two concentric rings 14 and 15, of metal, connected by radial pieces 16, of metal, and the charge-plate 13 may, as shown in Fig. 5, be a plate of metal having a number of radial openings 17 made through it for the due retention of the tablets, with an air-space around each, (17 are air-holes,) other openings, some radial and between adjacent ones of the aforesaid radial openings and some near the center of the plate, being made through the plate for the passage of air therethrough. The edges of the tablets will then rest more or less on the inner ring. For a larger lamp the floor-plate may have two or more concentric inner rings and the charge-plate a corresponding number of concentric rings of tablet-containing holes (of any desired capacity, according to the size of the tablets employed) with intervening air-holes.

For producing pyrobetulin anhydride the floor-plate may advantageously consist, as shown in Fig. 9, of a metal ring 18, having opposite sides connected together by a number of parallel bars 19, and the charge-plate may consist, as shown in Fig. 8, of a similar ring 20, having two intersecting sets of bars 21 and 22, respectively, that connect opposite sides, but are placed at a comparatively-great distance apart, and between which the tablets are arranged near together transversely to the bars 19 of the floor-plate.

The tablet-containing holes or spaces of the charge-plates, whether for producing pyrobetulin or for producing pyrobetulin anhydride, may be made with sinuous outlines or otherwise, so as to prevent the checking of the extrication of the fumes that might otherwise occur through the swelling of the tablets, especially in the case of large tablets.

The lamp is provided with a chimney 23, of wire-gauze, which rests on and within a collar 24, secured to the cup 9. Besides acting as a support to the chimney, the collar 24 serves to prevent air from entering the chimney between the latter and the cup 9 to any injurious degree.

In using apparatus constructed as described the tablets, having been suitably arranged in the lamp, as hereinbefore described, are ignited simultaneously by means of a jet or jets of gas caused to play upon their upper surfaces, and, any flame produced having been extinguished, the chimney 23 is placed upon the cup 9 and the lamp put upon its stand 8. The conical cover 4 is then placed in position, when the fumes, on being formed, rise upward to the top 26 of the cover 4, which deflects them downward and causes them to descend into the chamber 1.

It is advisable, especially in cold weather, that the lamp and its charge should be preparatorily heated as much as consistent with its proper handling, when arranged for producing pyrobetulin anhydride, in order more completely to prevent at the outset the evolution of low-temperature fumes adapted to produce pyrobetulin.

36 and 37 are apertures provided at opposite ends of the chamber 1 and normally closed by screw-plugs, as shown. After a charge has been burned and before igniting a new one the cover is removed and fresh air to support combustion is pumped or blown through the apertures 36 into the chamber, whence it displaces through the aperture 37 the air or gas that is no longer capable of supporting combustion.

I have obtained satisfactory results with an apparatus, as above described, in which the chamber 1 had a length of twenty-two inches, a breadth of sixteen inches, and a depth of eight inches, while the opening in its top was nine inches in diameter, the height of the cover twelve inches, and the diameter of the cup of the lamp two and one-fourth inches, this being adapted for small circular tablets of about five-eighths of an inch in diameter. With this apparatus I have employed tablets to the full extent of the respective charge-plates. The cover here described would be large enough for a chamber very considerably larger than the one here referred to.

In the case of a chamber 1 of very large area a hook or ring at each corner and connected with a common rope or chain passing over an overhead pulley is convenient for raising the chamber to allow of the removal of the deposited material from the floor of the chamber. Furthermore, the conical cover may be made in two parts forming an air-tight joint with each other by means of flanges, the lower part supporting the stand for the lamp and having either one or more internal annular grooved depressions or channels or corresponding projections adapted to prevent condensed moisture from running down and falling upon the deposited pyrobetulin or pyrobetulin anhydride.

For reducing the epidermis of birch-bark to a sufficiently-fine powder to produce the combustible material or tablets I first pass it through a disintegrator until it is reduced to a finely-shredded condition. I then dry it by exposing it to the air freely and to a temperature of 150° Fahrenheit during three days, and I then pass it repeatedly under heavy

stone edge runners until sufficiently fine to pass through a sieve having sixty holes to the linear inch.

When material containing betulin is burned under the conditions hereinbefore described, the cortical investment (in the case of birch-bark) or other matter surrounding or inclosing the betulin becomes by contact with the burning zone heated to such an extent that aided by the current of air produced by the heat of combustion it volatilizes the betulin. When the tablets are arranged in close contact, so as to give a limited air-supply, the temperature of combustion and relatively-long heating are such as to eliminate more or less completely a molecule of water, and thus to produce pyrobetulin anhydride; but when the tablets are so arranged as each to have an abundant air-supply the temperature of combustion is too low and the evolution of the fumes too rapid to effect this elimination, with the result that pyrobetulin itself is deposited in the chamber 1.

What I claim is—

1. The hereinbefore-described process, consisting in burning, without flame, by the unaided heat of its own combustion, and in a chamber of suitable size closed to the outer air, a mixture containing betulin and an oxygen-carrier.

2. Process of obtaining pyrobetulin or pyrobetulin anhydride by compressing together into blocks or tablets a mixture comprising or containing finely-ground epidermis or outer rind of birch-bark and an oxygen-carrier such as nitrate of potassium, and burning in a closed chamber without flame, a quantity of the said blocks or tablets bearing a suitable proportion to the air capacity of the said chamber, substantially as hereinbefore described.

3. Process of obtaining pyrobetulin or pyrobetulin anhydride by compressing together into blocks or tablets a mixture comprising or containing finely-ground epidermis or outer rind of birch-bark and an oxygen-carrier such as nitrate of potassium, arranging in a closed chamber a quantity of the said blocks or tablets bearing a suitable proportion to the air capacity of the said chamber in such a manner that when ignited they will all have either an abundant or a limited supply of air, and burning the said blocks or tablets in the said chamber without flame, substantially as hereinbefore described.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

JAMES WHEELER.

Witnesses:
W. C. HUTCHINGS,
S. ROTTENBURG.