No. 877,261. PATENTED JAN. 21, 1908

C. P. TOWNSEND.

METHOD OF PRODUCING STANNIC CHLORID.

APPLICATION FILED DEC. 6, 1904.

2 SHEETS—SHEET 1

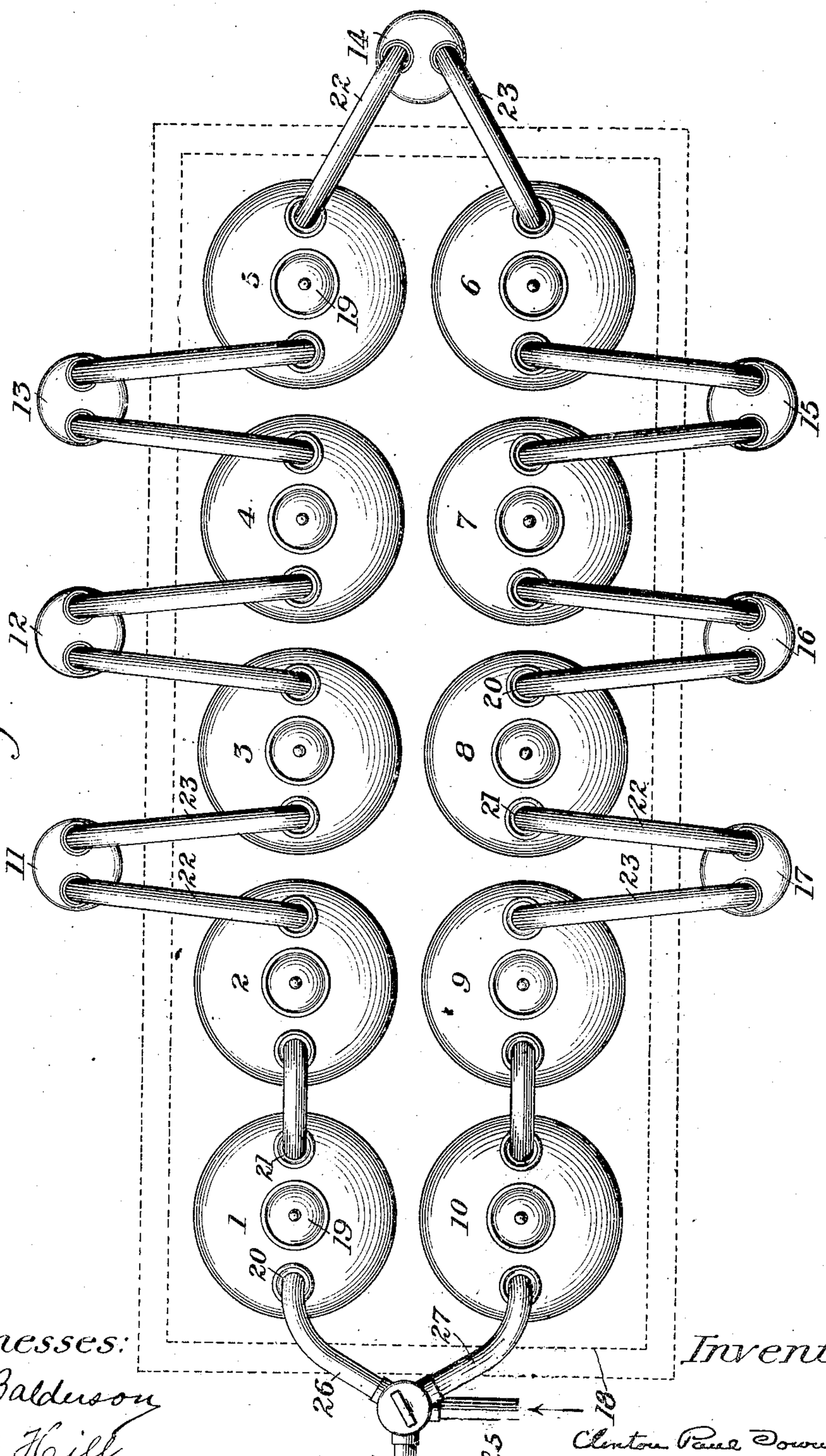

Fig. 1.

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Fig. 2

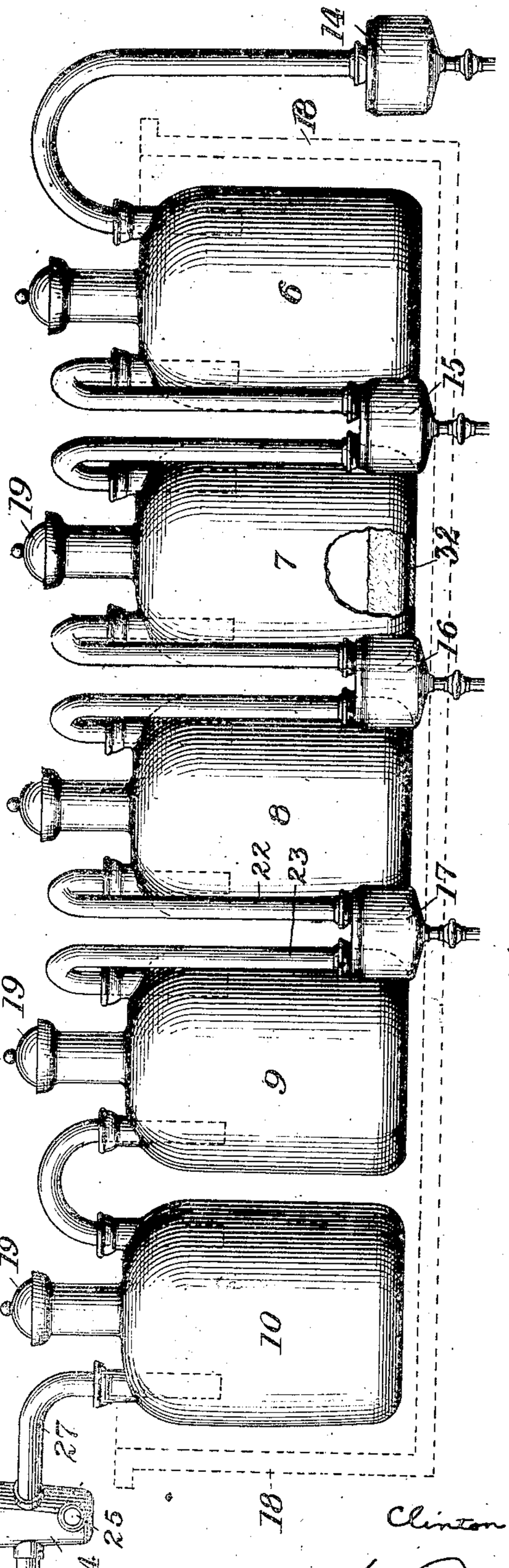


Fig. 4.

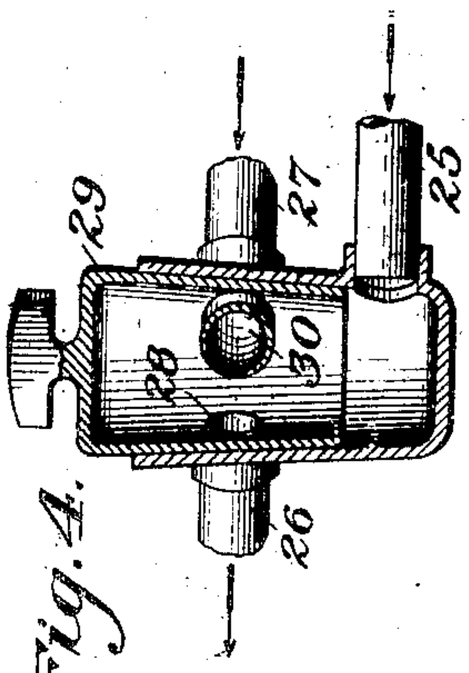
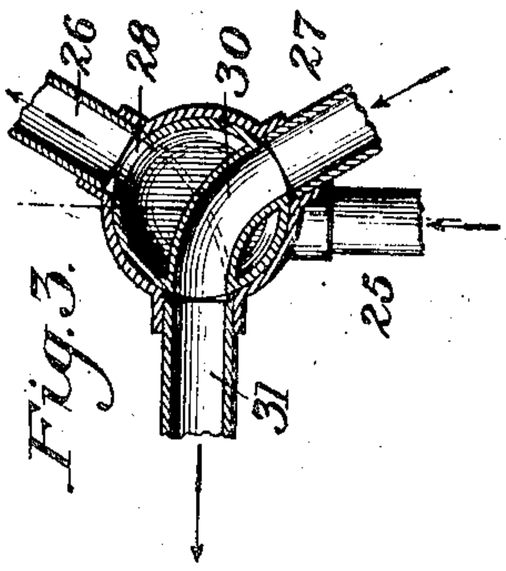


Fig. 3.



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UNITED STATES PATENT OFFICE.

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METHOD OF PRODUCING STANNIC CHLORID.

No. 877,261. Specification of Letters Patent. Patented Jan. 21, 1908.

Application filed December 6, 1904. Serial No. 235,765.

To all whom it may concern:

Be it known that I, CLINTON PAUL TOWNSEND, a citizen of the United States, residing at Washington, in the District of Columbia, have invented certain new and useful Improvements in Method of Producing Stannic Chlorid, of which the following is a specification.

This invention is a method of producing and recovering stannic chlorid from metallic tin, or from any material which contains tin in form capable of yielding a chlorid of tin by reaction with chlorin. The method is adapted to the recovery of stannic chlorid from the product resulting from the detinning of tin-scrap or cuttings, and will be described as applied to such product by way of example. This product is commonly obtained as a powder containing from 80 to 95 per cent. of tin, largely as metal, and relatively small proportions of lead and iron or their compounds. Tin in this form can be refined and rendered marketable only at considerable cost, but is well adapted to the production of stannic chlorid according to the present method.

For a full understanding of the invention reference is made to the accompanying drawings, wherein,—

Figure 1 is a plan view of one form of apparatus for carrying out the method; Fig. 2 is a side elevation of the same; and Figs. 3 and 4 are details.

Referring to the figures, reference numerals 1—10 represent a series of vessels or retorts of which any desired or suitable number may be employed: These retorts are preferably arranged in a continuous series with a number of interposed condensers. The retorts shown constitute a series and are closely grouped, suitable means being provided for the application of heat as, for instance, by embedding all retorts in sand or other medium contained in a pan indicated in outline by dotted lines 18, Figs. 1 and 2: Heat is applied to this pan in any suitable way, as by the contact of flue gases or the application of steam under suitable pressure. The retorts may, of course, be separately heated by any convenient means.

The retorts are indicated as consisting of acid proof stoneware. Each retort is provided with a central aperture having a cover 19, this aperture serving for charging the retort and discharging the residuum; and two lateral apertures 20, 21 for the passage of gases and vaporized reaction products. The vessel or retort 1 communicates directly with retort 2, and retort 9 with 10, but the remaining retorts of a series communicate only through interposed condensers. These condensers may be of any desired form, those illustrated comprising the pipes 22, 23 and the collecting vessels 11—17. It will be understood that in the production of stannic chlorid condensation occurs also in the exit pipe from the retort, and means for withdrawing the liquid from this pipe may be used instead of the collecting vessels shown.

24 is a cock shown in detail in Figs. 3, 4. Chlorin is introduced through inlet 25, and is directed into either of the pipes 26 or 27 in accordance with the position of the opening 28 in the cap 29. When the cock is turned as illustrated in Fig. 3 the chlorin introduced at 25 will pass through pipe 26 into the vessel 1, thence in succession through the several retorts and interposed condensers, returning through pipe 27 to the cock and being directed by the tube 30, carried by the cap 29, to the outlet 31 for waste gases. When the cock is revolved into the position indicated in dotted lines in Fig. 3 the direction of flow through the retorts and condensers is reversed, the inlet and outlet remaining unchanged. Any convenient means for controlling and reversing the flow of chlorin may be substituted. The pipes and collecting chambers constituting the condensers may, if desired, be provided with a heat insulating cover. The collecting chambers are conveniently of glass, or other means are provided for watching the progress of the operation. The condenser pipes may advantageously extend for a considerable distance into the retorts, as indicated by dotted lines in Fig. 2, whereby the current of gas is directed toward the surface of the charge.

In using the above described apparatus the operation is conducted substantially as follows: The retorts 2—9 are charged with the tin-bearing material, preferably as a layer of relatively slight depth, and chlorin is admitted to the series, the chlorin being preferably admitted faster than it can be absorbed by the tin in a single retort. The retorts are maintained during the distillation period at a suitable temperature above the vaporizing point of stannic chlorid (114° C.), and the vaporized and entrained stannic chlorid to-

gether with the excess of chlorin passes into the condensing and collecting apparatus 22, 11, 23, the uncondensed portions passing thence to the retort 3 where a further absorption or condensation occurs. At this stage of the operation the distillation will proceed most rapidly in retort 2, and the number of retorts in series will be so related to the rate of supply of chlorin that the first retort will cease to yield stannic chlorid before the distillation has begun in the last retort of the series.

For a clear understanding of the purpose of the series arrangement of retorts and interposed condensing means it should be understood that in order to insure a rapid production and distillation of stannic chlorid it is necessary that an excess of chlorin be brought into presence of the tin; and that it is extremely difficult to secure a complete condensation of stannic chlorid, particularly from a gas containing chlorin. It is clear, therefore, that if it be attempted to produce stannic chlorid in a single retort followed by a condensing system, there will result a loss of chlorin representing the excess used, and a loss of stannic chlorid representing the uncondensed portion. According to this invention such excess of chlorin as is desirable for rapid and economical production and distillation of stannic chlorid is used, and no particular necessity exists for providing the complex apparatus which would be necessary for securing an approximately complete condensation of the stannic chlorid. The gases passing from the first condenser to the second retort ordinarily contain both chlorin and stannic chlorid, both of which are quickly absorbed or condensed in presence of the tin in the second and following retorts. As each retort in succession ceases to absorb chlorin and stannic chlorid it will begin the delivery of stannic chlorid to its condenser and the succeeding retorts. Before the last retort in series has ceased absorbing, one or more of the retorts first in series will have ceased distilling and should then be recharged.

In the particular form of apparatus shown it is advisable at this stage to reverse the direction of flow of chlorin through the retorts by rotating the cap 29 of the cock 24, permitting the chlorin to enter the retorts through pipe 27 and the waste gases to escape through pipe 26. The retorts 9, 8, 7, 6 will then yield stannic chlorid and the freshly charged retorts 2, 3, 4, 5 will act as absorbers. The number of retorts yielding stannic chlorid and the number acting as absorbers for the uncondensed gases will, of course, be dependent upon the total quantity of chlorin which has been introduced before reversing the flow.

The apparatus may be so arranged as to obviate the necessity for reversing the flow of chlorin at intervals: For instance, the retorts may be so connected that as the distillation ceases in the first in series, the chlorin is introduced into the second, the first being re-charged and connected as an absorber at the end of the series. According to such arrangement the direct chlorin connection will be transferred successively to the retorts constituting the series.

The apparatus as above described is suited for use with chlorin which is approximately dry. If the chlorin contains considerable moisture it should be dried, or, preferably, a part or all of the contained moisture should be converted into hydrochloric acid. This is very conveniently effected as follows: The vessels 1 and 10 are intended to contain portions of the residue which remains in the retorts 2—9 after the completion of the distillation. This residue contains a considerable proportion of ferric chlorid, which, upon contact with wet chlorin at a suitable temperature, reacts with the contained moisture to produce hydrochloric acid as follows;

$$XCl_2 + 3H_2O + 2FeCl_3 = XCl_2 + Fe_2O_3 + 6HCl.$$

This is advantageous because the reaction is accelerated by the presence of hydrochloric acid, and also because that portion of the chlorin which would otherwise be lost as ferric chlorid is recovered as stannic chlorid. It is, of course, not essential that the vessels 1—10 for treating the moist chlorin should be grouped with the distilling retorts.

Instead of stoneware retorts, retorts of lead or of iron lined with lead, or such vessels as are employed as generators for chlorin by the manganese dioxid method may be used.

If the chlorin be substantially undiluted a wide range of temperatures above the vaporizing point of stannic chlorid is applicable; if, however, the chlorin be diluted with a considerable proportion of air such strong ignition as will result in oxidizing the tin should be avoided.

The tin bearing material is preferably introduced in successive small charges until the retorts are filled. The volume of the residue at the close of the operation in case crude powder is treated equals or exceeds that of the original charge, but its weight is, of course, relatively small. Its apparent specific gravity is therefore very low and it may conveniently be discharged by a suction pipe introduced into the central aperture of the retort.

The heat developed by the reaction between fresh bodies of tin and chlorin is frequently sufficient to melt the tin, whereby the retorts are endangered: these may be protected by applying to the interior surface a layer of a suitable material such as asbestos paper, cement, etc., indicated at 32 in Fig. 2. In case the retorts are independently heated, heat will preferably be applied only as re-

quired: during the early stages of the absorption the application of heat is not necessary.

The apparatus described herein is claimed in my co-pending application Serial No. 302,218, filed February 21, 1906.

I claim:—

1. The method of producing stannic chlorid, which consists in subjecting a tin-bearing material to the action of chlorin and vaporizing stannic chlorid, condensing stannic chlorid from the vapor, and bringing the uncondensed vapor into presence of tin-bearing material, substantially as described.

2. The method of producing stannic chlorid, which consists in subjecting a tin-bearing material to the action of an excess of chlorin and vaporizing stannic chlorid, condensing stannic chlorid from the vapor, and bringing the uncondensed vapor into presence of tin-bearing material, substantially as described.

3. The method of producing stannic chlorid, which consists in subjecting a tin bearing material to the action of chlorin and vaporizing stannic chlorid, condensing stannic chlorid from the vapor, and bringing the uncondensed vapor into presence of tin-bearing material at a temperature sufficient to vaporize stannic chlorid, substantially as described.

4. The method of producing stannic chlorid, which consists in subjecting a tin-bearing material to the action of an excess of chlorin and vaporizing stannic chlorid, condensing stannic chlorid from the vapor, and bringing the uncondensed vapor into presence of tin-bearing material at a temperature sufficient to vaporize stannic chlorid, substantially as described.

5. The method of producing stannic chlorid, which consists in subjecting a tin-bearing material to the action of an excess of chlorin and vaporizing stannic chlorid, condensing stannic chlorid from the vapor, bringing the uncondensed vapor into presence of tin-bearing material at a temperature sufficient to vaporize stannic chlorid, and repeating the process until the excess of chlorin is absorbed, substantially as described.

6. The method of producing stannic chlorid, which consists in subjecting a tin-bearing material to the action of chlorin and hydrochloric acid and vaporizing stannic chlorid, condensing stannic chlorid from the vapor, and bringing the uncondensed vapor into presence of tin-bearing material, substantially as described.

7. The method of producing stannic chlorid, which consists in passing a stream containing chlorin successively in contact with separate bodies of a tin-bearing material, and shifting the point of supply of chlorin when the tin in any one of said bodies is substantially exhausted, substantially as described.

8. The method of producing stannic chlorid, which consists in alternately passing a stream containing chlorin in contact with separate bodies of a tin-bearing material and condensing stannic chlorid from said stream, substantially as described.

9. The method of producing stannic chlorid, which consists in alternately passing a stream containing chlorin in contact with separate bodies of a tin-bearing material and condensing stannic chlorid from said stream, and shifting the point of supply of chlorin when the tin in any one of said bodies is substantially exhausted, substantially as described.

10. The method of producing stannic chlorid which consists in continuously supplying chlorin to a body of metallic tin-bearing material in the presence of anhydrous stannic chlorid.

11. The method of producing stannic chlorid, which consists in reacting on tin-bearing material with chlorin in presence of anhydrous stannic chlorid at a temperature above the vaporizing point of anhydrous stannic chlorid, maintaining the reaction by supplying chlorin to the reacting mass, and recovering anhydrous stannic chlorid.

12. The method of producing stannic chlorid which consists in continuously supplying chlorin to a body of metallic tin-bearing material in the presence of anhydrous stannic chlorid, and controlling the temperature of the reaction.

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

CLINTON PAUL TOWNSEND.

Witnesses:
EUGENE A. BYRNES,
JULIA B. HILL.