No. 885,391. PATENTED APR. 21, 1908.

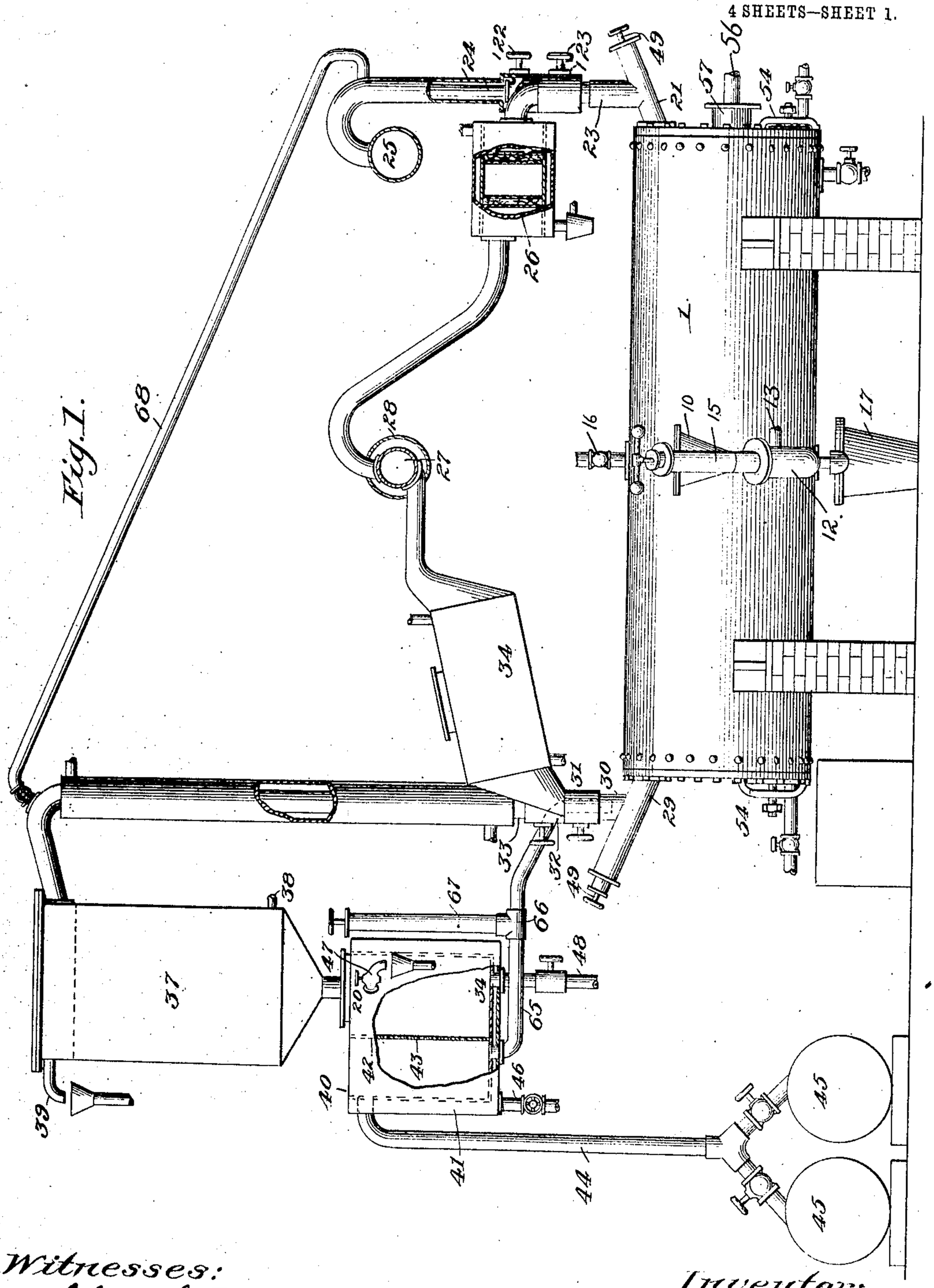
E. A. SPERRY.

APPARATUS FOR EFFECTING REACTIONS.

APPLICATION FILED DEC. 29, 1906.

4 SHEETS—SHEET 1.

Fig. 1.



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4 SHEETS—SHEET 2.

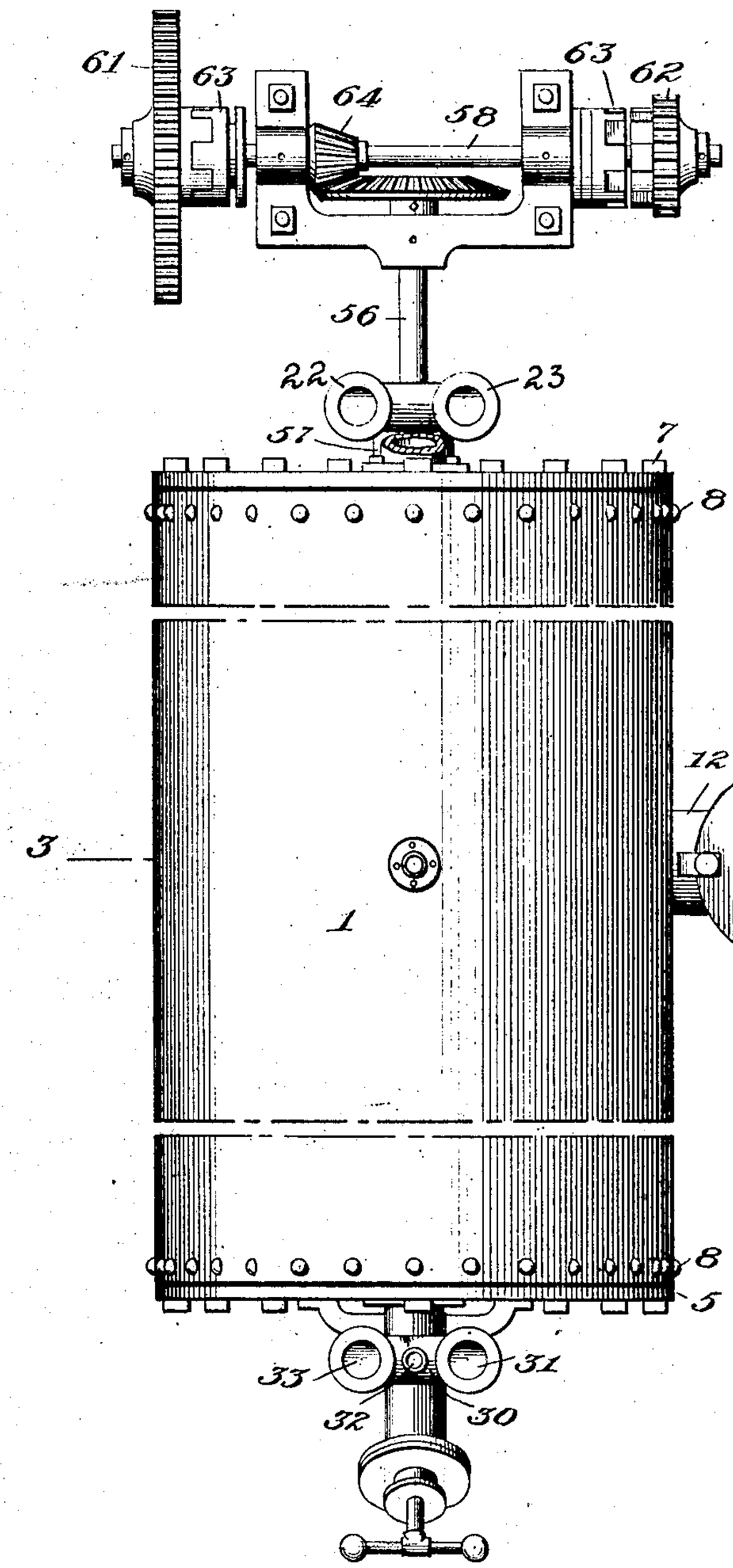

Fig. 2.

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4 SHEETS—SHEET 3.

Fig. 3.

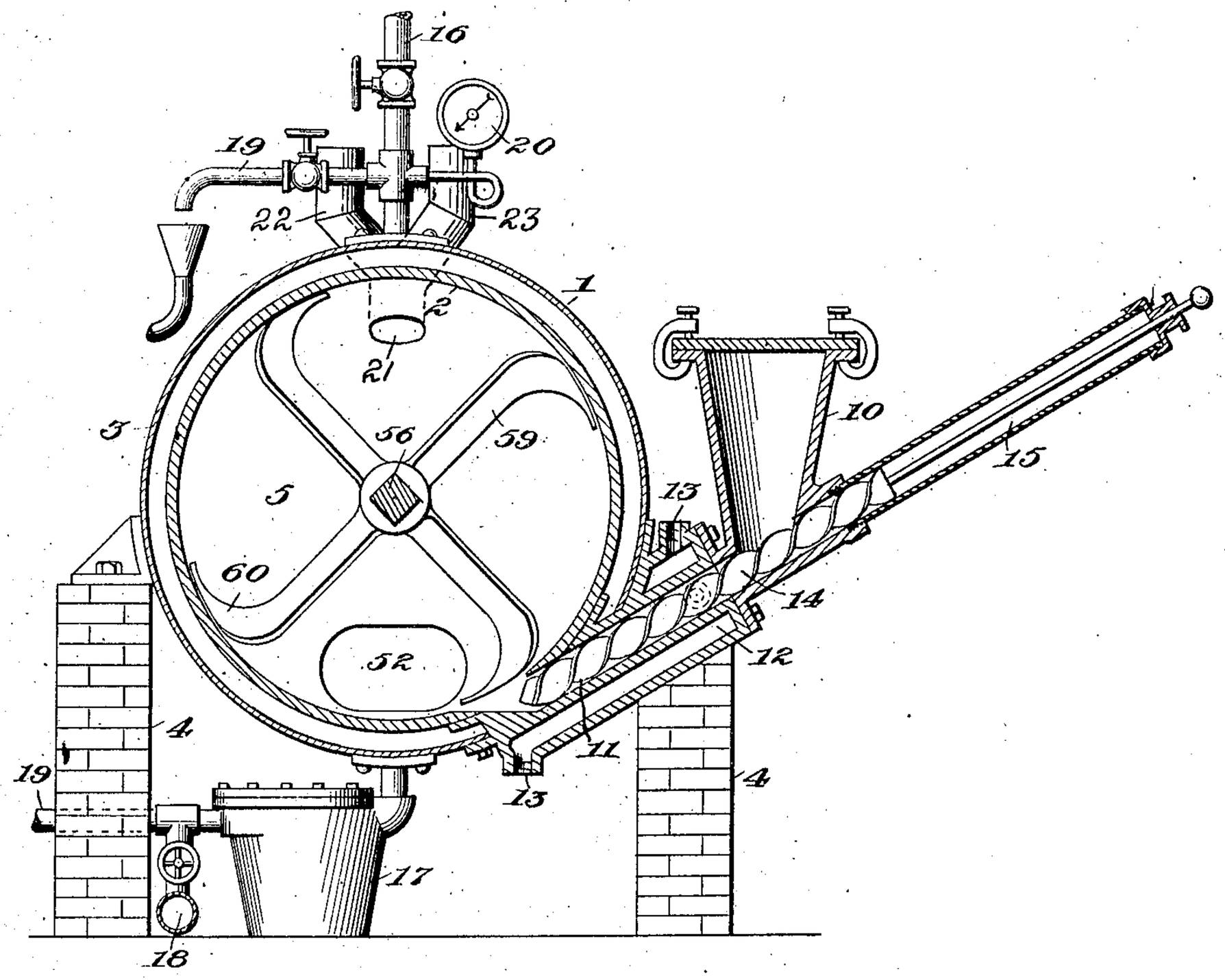
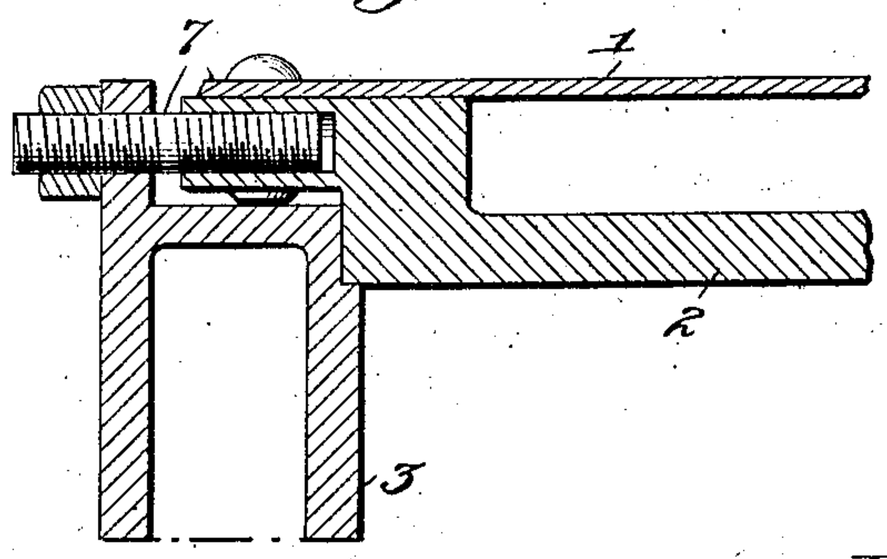


Fig. 5.



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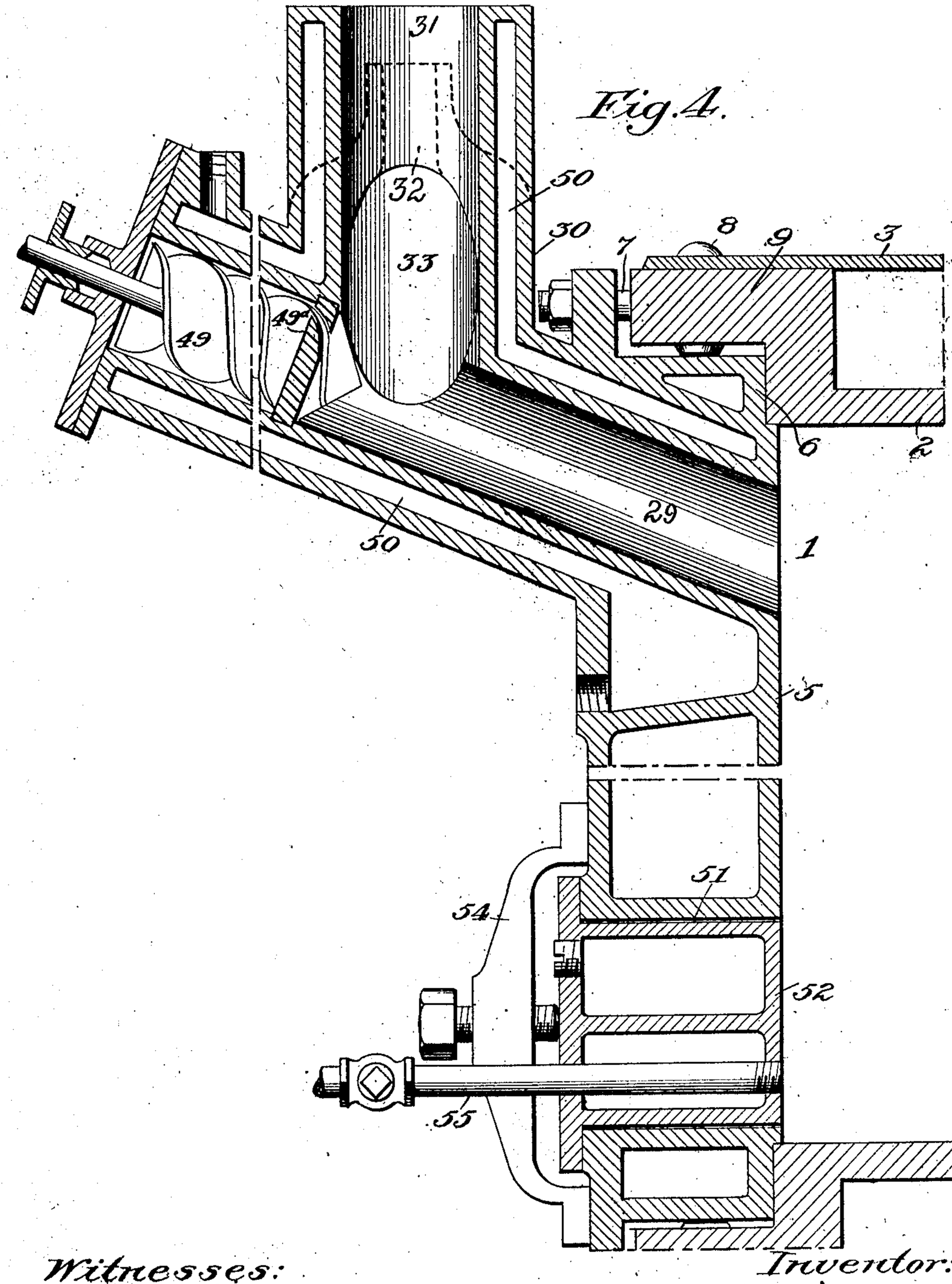
4 SHEETS—SHEET 4.

Fig. 4.



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UNITED STATES PATENT OFFICE.

ELMER A. SPERRY, OF BROOKLYN, NEW YORK.

APPARATUS FOR EFFECTING REACTIONS.

No. 885,391. Specification of Letters Patent. Patented April 21, 1908.

Application filed December 29, 1906. Serial No. 350,093.

To all whom it may concern:

Be it known that I, ELMER A. SPERRY, a citizen of the United States, residing at Brooklyn, in the county of Kings and State of New York, have invented certain new and useful Improvements in Apparatus for Effecting Reactions, of which the following is a specification.

The object of this invention is to provide an apparatus for effecting reactions, and particularly for the production of stannic chlorid or similar anhydrous liquids by reactions involving the use of gaseous chlorin.

For the production of stannic chlorid the chlorin, which usually contains a certain proportion of moisture, is caused to react upon a tin-bearing material. The apparatus is designed to effect such reactions at a rapid rate, while securing high efficiency both in the production and recovery of the compound.

For a full understanding of the invention reference is made to the accompanying drawings, showing a preferred form of apparatus.

In said drawings: Figure 1 is a side elevation of an apparatus which may constitute one unit of a plant for the production of stannic chlorid; Fig. 2 is a plan view of the reaction and distillation vessel and the driving gear therefor, upon a somewhat larger scale, parts being broken away; Fig. 3 is a transverse section on line 3—3 of Fig. 2; Fig. 4 is a sectional view on an enlarged scale of one of the heads of the reaction vessel, showing the gas or vapor connection; and Fig. 5 is a sectional detail showing a preferred means of attaching the head to the drum, and the means for securing the jacket to the drum.

Referring to the drawings, 1 represents a reaction and distillation vessel comprising a tubular casting 2, preferably of iron, jacketed with boiler plate as indicated at 3 and suitably mounted on columns 4. The heads 5 are shown as comprising hollow castings, although the provision of temperature regulating means for the heads is not essential. The casting 2 and the heads 5 are provided with abutting surfaces 6, and the heads are adapted to be secured in position by studs 7. The jacket 3 is secured by rivets 8, passing through the flange 9 of the casting 2. In the treatment of anhydrous liquids it is essential to avoid leakage of water or steam from the jacket to the interior of the reaction vessel, and this object is fully attained by the above construction in which it will be observed there are no bolts, rivets or the like passing into the interior of the vessel or into the space constituting the jacket; consequently there can be no leakage of water or water vapor which will affect the contents of the vessel.

The feeding means comprises a covered hopper 10 communicating with the lower portion of the vessel 1 by a conduit 11 having a jacket 12 provided with steam connections 13. The feeding means shown comprises a screw 14 which may be hand or power operated and which is preferably contained in an elongated casing 15 so that in case of necessity it may be fully retracted; this construction also permits the feed to be effected by turning or ramming as may be desired.

The space between the jacket and the drum may be supplied with a heating or cooling medium as desired. For heating, steam may be admitted through the pipe 16, the water of condensation being delivered through the trap 17 to a hot return 18 which may be common to several reaction vessels; for cooling, water may be admitted through pipe 19, passing upwardly through the jacket and overflowing through the visual indicator 19. Suitable valves are shown for cutting off the steam supply when using the water and vice versa, and a steam gage 20 is indicated. When jacketed or hollow heads are employed they may likewise be coupled both to the steam and water supply, and a steam supply is also afforded for the jacket 12 of the feed device and for the gas inlet and outlet 21, 29 hereafter described.

As shown at the right side of Fig. 1 the chlorin inlet pipe 21 branches upwardly in the form of a Y having branches 22 and 23, and valves 122 and 123 in the respective branches; one of these branches is in direct communication through jacketed pipe 24 with the chlorin main 25. The remaining branch connects through a jacketed filter 26 with a main 27, leading to a tower, scrubber or other means for recovering residual vapors. This main is also jacketed as indicated at 28. The outlet 29 for chlorid vapors is provided with a Y connection 30 similar to that above described, but having also a central branch as most clearly shown in Fig. 2, thus providing three apertures 31, 32, 33, each having a suitable valve. The aperture 31 communi-

cates through a condenser 34 with the main 27 before referred to. The aperture 33 communicates directly with a vertical conduit 35 provided with a jacket 36 having both steam and water connections. The central aperture 32 communicates with a separating vessel hereinafter described.

The vertical conduit 35 is connected at its upper end with a condenser 37 for the anhydrous vapors, the condenser jacket being provided with an inlet 38 for water and a visual overflow 39. The condenser 37 communicates at its lower end with a separator 40, the purpose of which is to part the liquid anhydrous chlorid from any hydrated salt which may be present. This separator 40 is jacketed as indicated at 41, and is provided with a vertical partition 42 perforated at its central portion as indicated at 43. The purpose of the imperforate upper and lower parts of the partition 42 is to prevent the passage to the drums of any crystalline hydrated chlorid of tin which may either float upon the surface of the anhydrous liquid or settle to the bottom. The separator 40 communicates on the side removed from the condenser 37 through overflow pipe 44 with the drums 45 or other storage receptacles for anhydrous chlorid. The inlet 46 to the jacket of the separator 40 is usually connected with a source of hot water or of steam, an overflow being indicated at 47. 48 represents a valved outlet for residues from the separation. The separator 40 communicates through pipe 65, preferably jacketed, with the central apertures 32 of the Y connection 30 above referred to, and therefore with the interior of the vessel 1. In the pipe 65 I provide a valve 66, also jacketed, the jacket extending upwardly around the valve stem to a point above the top of the separator 40. In practice this jacket extension is filled with an anhydrous liquid, usually stannic chlorid, the purpose of which is to provide a liquid seal for the valve and to prevent sticking due to access of moisture. 68 represents a valved pipe leading from the top of the vapor pipe 35 and communicating with the chlorin main 25.

By reason of the tendency of the inlet and outlet to the vessel 1 to become clogged with crystalline hydrated chlorids when operating with moist chlorin it is preferred to provide both the inlet and outlet conduits with clearing means conveniently in the form of a scraper or screw conveyer 49 as best shown in Fig. 4, this scraper rotating in the conduit and having also a longitudinal movement therein. A wiper 49[a] is so disposed as to clear the scraper 49 from accumulations tending to adhere to the blades. The thorough jacketing of these parts as shown at 50 in Fig. 4, provides means whereby the temperature may be raised or maintained above the melting point of the crystalline hydrates, and not only tends to prevent clogging of the pipes but greatly facilitates the clearing of the same should clogging occur. The residue of the operation is removed through an aperture 51 in the head 5, this aperture being normally closed by a plug 52 secured by the usual yoke 54. 55 represents a valved air-inlet pipe, which may conveniently pass through the plug or closure 52.

Referring particularly to Figs. 2 and 3, 56 represents a central shaft, passing through a stuffing box 57 at one end of the vessel and connected to suitable power mechanism 58 (Fig. 2). The shaft 56 carries a plurality of agitating arms or plows 59, the blades 60 of the plows being so constructed as to thoroughly agitate and lift the mass undergoing reaction or distillation and to thoroughly distribute the same. The arms 59 also greatly facilitate the absorption of chlorin by the anhydrous liquid, not only by agitating it and thereby increasing the exposed surface, but by conveying films of the liquid into the stream of gas.

Referring particularly to Fig. 2, the power device 58 comprises a two-speed drive for the shaft 56, slower from the gear 61 and faster from the gear 62. Suitable clutches 63 are provided and the power is transmitted to the shaft 56 through bevel gears 64.

The operation of the apparatus is as follows: In starting there is admitted to the vessel 1 through pipe 65 a quantity of anhydrous stannic chlorid, the dimensions of the separator 40 being such that the chlorid remaining therein will be sufficient for this purpose. A quantity of tin-bearing material is supplied from the hopper 10, and chlorin gas is led in through inlet 21. The agitator is driven at its higher speed, usually about 48 revolutions per minute and the temperature within the vessel is controlled by the admission of water or other cooling medium to the jacket. Under these conditions the production of stannic chlorid is rapid, and such heat is developed by the reaction as to necessitate energetic cooling of the vessel. If the chlorin be at all diluted, as is ordinarily the case, the residual gases conveying some stannic chlorid are caused to pass through condenser 34 to deposit a portion of the chlorid, and thence by the main 27 to a scrubber or the like for extracting any residual portions; preferably these residual gases are drawn off by means of an exhaust fan or the like arranged in connection with the scrubber, and the operation is preferably so conducted as to maintain a slightly reduced pressure in the vessel 1 in order that no outward leakage may occur. The tin-bearing material is added continuously or from time to time until the chlorid has accumulated in the vessel to the desired extent; in practice the vessel is usually about two-thirds full at the close of the operation. Thereupon the

valves governing communication with the chlorin main 25 and the condenser 34 are closed, and connection is established with the vertical conduit 35, the jacket of which is cooled at this stage, and a further small quantity of tin is supplied to the reaction vessel. Agitation is continued, and any chlorin escaping at this stage is returned through pipe 68 to the system, the stannic chlorid being mostly condensed in vapor pipe 35 and returned to the vessel 1. When the last traces of chlorin have disappeared the valve in pipe 68 is closed, steam is admitted to the jacket 3 and to the jacket 36 of the vapor pipe 35, and the stannic chlorid in the vessel 1, including any hydrated salt which may be present, is quickly distilled over and condensed in the condenser 37. During this stage of the operation the lower gear 61 is preferably employed and the agitator shaft is revolved at about eight revolutions per minute, which enables it to thoroughly agitate the mass even in a pasty or heavy condition, without the use of excessive power.

The chlorids pass freely to the separator 40 and the pure anhydrous chlorid flows through partitions 42 and thence to the shipping drums 45. The imperforate upper and lower parts of this partition retain the floating as well as the subsided hydrated chlorid, which may afterward be withdrawn through pipe 48. When the distillation has substantially ceased, even under the conditions of agitation and heat above described, communication with the vapor pipe 35 is closed, and valve 22 and the valve in the air-inlet pipe 55 are opened. Under suction through main 27 heated air or gas is drawn in through pipe 55, further quantities of chlorid are removed, and the residue is distilled to dryness. During this operation considerable dust is carried forward, and this is removed by the heated filter 26, contamination of the absorbing medium in the scrubber being thus avoided. At the close of the operation the residue is removed through aperture 51, and after adjustment of the several valves the operation is repeated. The temperature of the filter 26 should be sufficiently high to prevent any condensation or separation of chlorid therein.

I claim:

1. Apparatus for effecting reactions, comprising a vessel, a vapor connection therefor, mechanically operated means for clearing said vapor connection, and heating means for said connection.

2. Apparatus for effecting reactions, comprising a vessel having a vapor connection, a rotary clearing device for said connection, and a wiper for said clearing device.

3. Apparatus for effecting reactions, comprising a vessel, an agitator therein, a jacket for said vessel, and separate induction and eduction connections for supplying steam and water to said jacket.

4. Apparatus for effecting reactions, comprising a vessel, a mechanical feeding device one end of which enters the vessel, and heating means for said feeding device.

5. Apparatus for effecting reactions, comprising a vessel, a mechanical feeding device one end of which is connected with the lower portion of the vessel, and heating means for said feeding device.

6. Apparatus for effecting reactions, comprising a vessel having a vapor connection, a filter in said vapor connection, and means for heating said connection and the filter.

7. In apparatus for effecting reactions, a condenser and a separator for the solid and liquid condensed products in operative relation with said condenser.

8. In apparatus for effecting reactions, a condenser, and a continuous-flow separator for the solid and liquid condensed products in operative relation with said condenser.

9. In apparatus for effecting reactions, a separator comprising a pressure tight vessel, a substantially vertical partition therein having a central aperture, and inlet and outlet apertures on opposite sides of said partition.

10. In apparatus for effecting reactions, a vessel having a valved connection, and an anhydrous liquid seal for the moving part of said valve.

11. In apparatus for effecting reactions, a jacketed vessel, an agitator therein, and a plural speed driving mechanism for said agitator.

12. In apparatus for effecting reactions, a vessel having an outlet, a condenser connected with said outlet, and means located between the condenser and the vessel for trapping a portion of the condensed product and for returning the same to the vessel.

13. In apparatus for effecting reactions, an elongated reaction vessel, a jacket therefor, a gas inlet and outlet at opposite ends of said vessel, and an agitator within the vessel adapted to carry films of liquid above the normal liquid level.

14. In apparatus for effecting reactions, an elongated reaction vessel, a jacket therefor, a gas inlet and outlet at opposite ends of said vessel, and an agitator within the vessel adapted to agitate the material therein and to carry films of liquid above the normal liquid level.

15. In apparatus for effecting reactions, a reaction vessel, a plurality of jacketed condensers, and jacketed conduits between said vessel and condensers.

16. In apparatus for effecting reactions, a reaction vessel, a plurality of jacketed condensers, jacketed conduits between said vessel and condensers, and jacketed valves in said conduits.

17. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein, a jacket for said vessel, steam and water-supply connections for said jacket, and a visual indicator for the water-supply.

18. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein, a jacket for said vessel, steam and water-supply connection for said jacket, and a gas inlet and outlet for the vessel opposite each other, situated upon the longest axis of said vessel.

19. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein with a horizontal axis, a jacket for said vessel, steam and water-supply connections for said jacket, gas induction and eduction openings at the opposite ends of the longest axis of said vessel in the upper part thereof, and a feeding device in the lower part of the vessel.

20. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein, a jacket for said vessel, steam and water-supply connections for said jacket, a gas inlet and outlet for the vessel opposite each other, situated upon the longest axis of said vessel, and a feeding device located in the middle of the elongated vessel.

21. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein, a jacket for said vessel, steam and water-supply connections for said jacket, and a manhole for the vessel at the end thereof.

22. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein, a jacket for said vessel, steam and water-supply connections for said jacket, and a vent for the vessel at a low point located at the end of the vessel.

23. Apparatus for effecting reactions, comprising an elongated horizontal vessel and agitator therein, a jacket for said vessel, steam and water-supply connections for said jacket, a gas inlet and outlet for the vessel opposite each other, situated upon the longest axis of said vessel, a feeding device located in the middle of the elongated vessel, and a closure for the outer end of the feeding device.

24. Apparatus for effecting reactions, comprising a vessel having vapor connections, a filter in said vapor connection, means for heating said filter, a vent for the filter, and means for heating the vent.

25. Apparatus for effecting reactions, a separator comprising a closed vessel, a substantially vertical partition therein having a centrally perforated strainer portion and inlet and outlet apertures on opposite sides of said partition.

26. Apparatus for effecting reactions, comprising an elongated cylindrical vessel, a rotating agitating device interior to the vessel, and a feeding device for the vessel, disposed tangentially to the cylinder in a transverse plane thereto entering at the bottom of the vessel.

27. Apparatus for effecting reactions, comprising an elongated cylindrical vessel, a rotating agitating device interior to the vessel, and a feeding device for the vessel, disposed tangentially to the cylinder in a transverse plane thereto entering in the direction of rotation of the agitating device.

28. Apparatus for effecting reactions, comprising an elongated cylindrical vessel, a rotating agitating device interior to the vessel, a feeding device for the vessel, disposed tangentially to the cylinder in a transverse plane thereto, and a mechanism within the feeding device for effecting a positive feed of the materials therein.

29. Apparatus for effecting reactions consisting of a cylinder, a head upon the cylinder, screw threaded fixtures for securing the head, the cylindrical casing having an enlargement upon its ends, performing the threefold function of receiving the screw threaded fixtures, the outer casing forming the jacket of the cylinder and the inner portion of such enlargement serving as a seating for the head.

30. Apparatus for effecting reactions, comprising an elongated horizontal cylinder and agitator therein having journals for its shaft, a jacket for the cylinder, removable heads for the cylinder, gas inlets and outlets for the cylinder, and a manhole for the cylinder, and journals for the said shaft, the gas passages, and manholes located in the said removable heads.

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

ELMER A. SPERRY.

Witnesses:
L. A. WELLES,
W. P. PALMER.