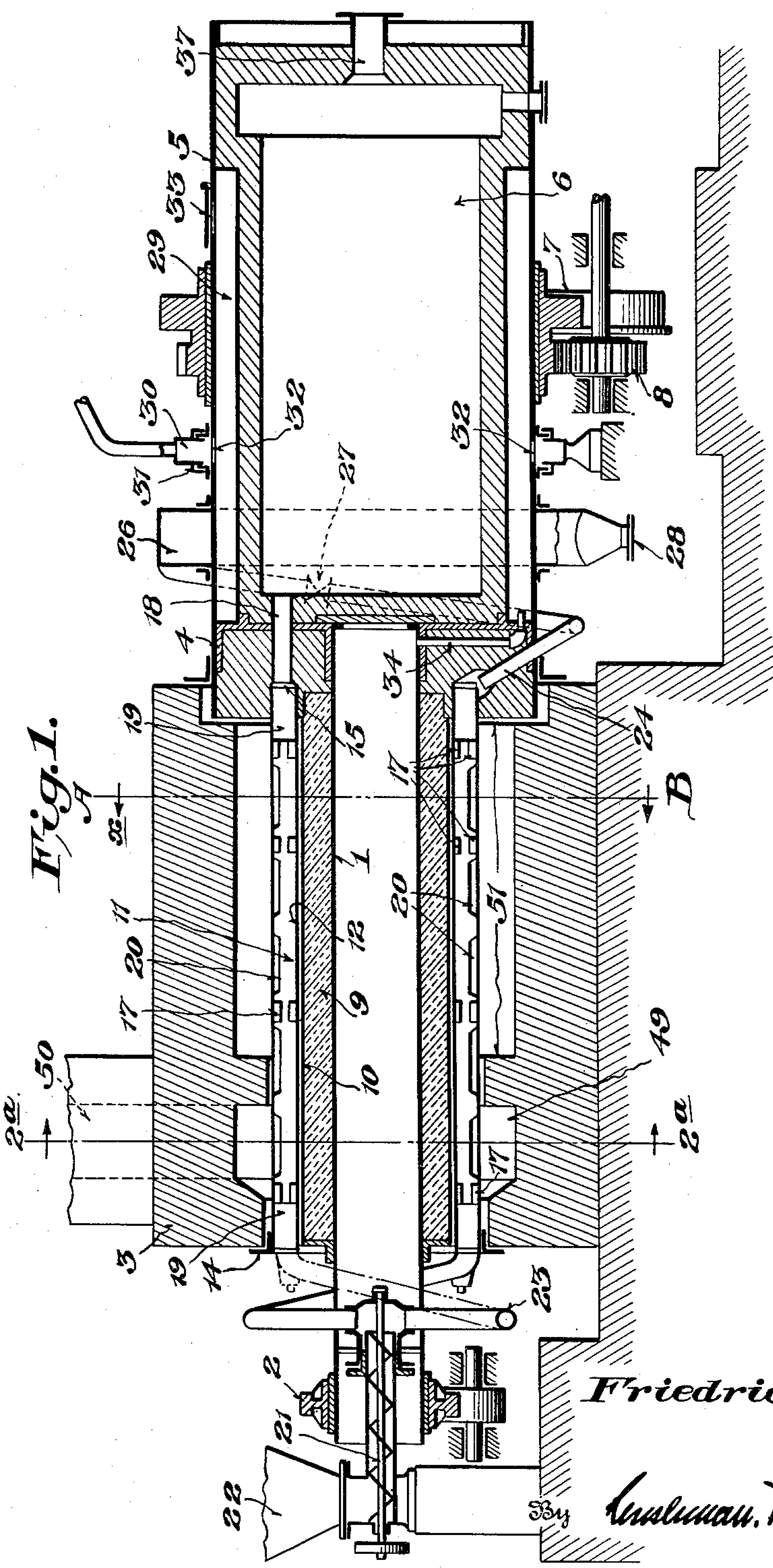
July 9, 1935. F. JOHANNSEN 2,007,332

APPARATUS FOR THE DISTILLATION OF ZINC AND OTHER VOLATILE METALS

Filed Oct. 2, 1933 4 Sheets-Sheet 1



Inventor

Friedrich Johannsen,

By [signature] Attorneys

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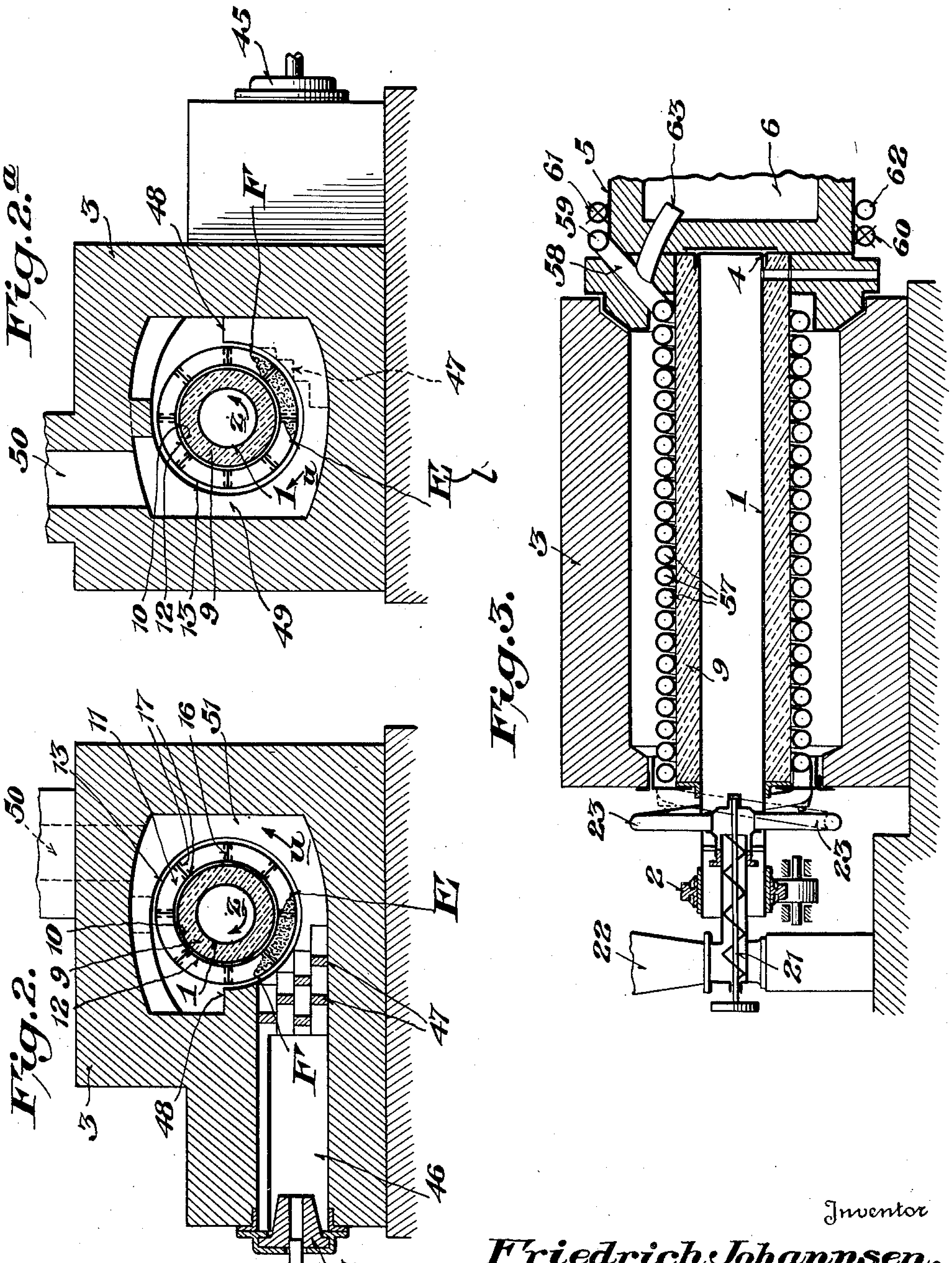
Filed Oct. 2, 1933 4 Sheets-Sheet 2



Inventor

Friedrich Johannsen,

By Cushman, Darby & Cushman

Attorneys

APPARATUS FOR THE DISTILLATION OF ZINC AND OTHER VOLATILE METALS

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Fig. 4

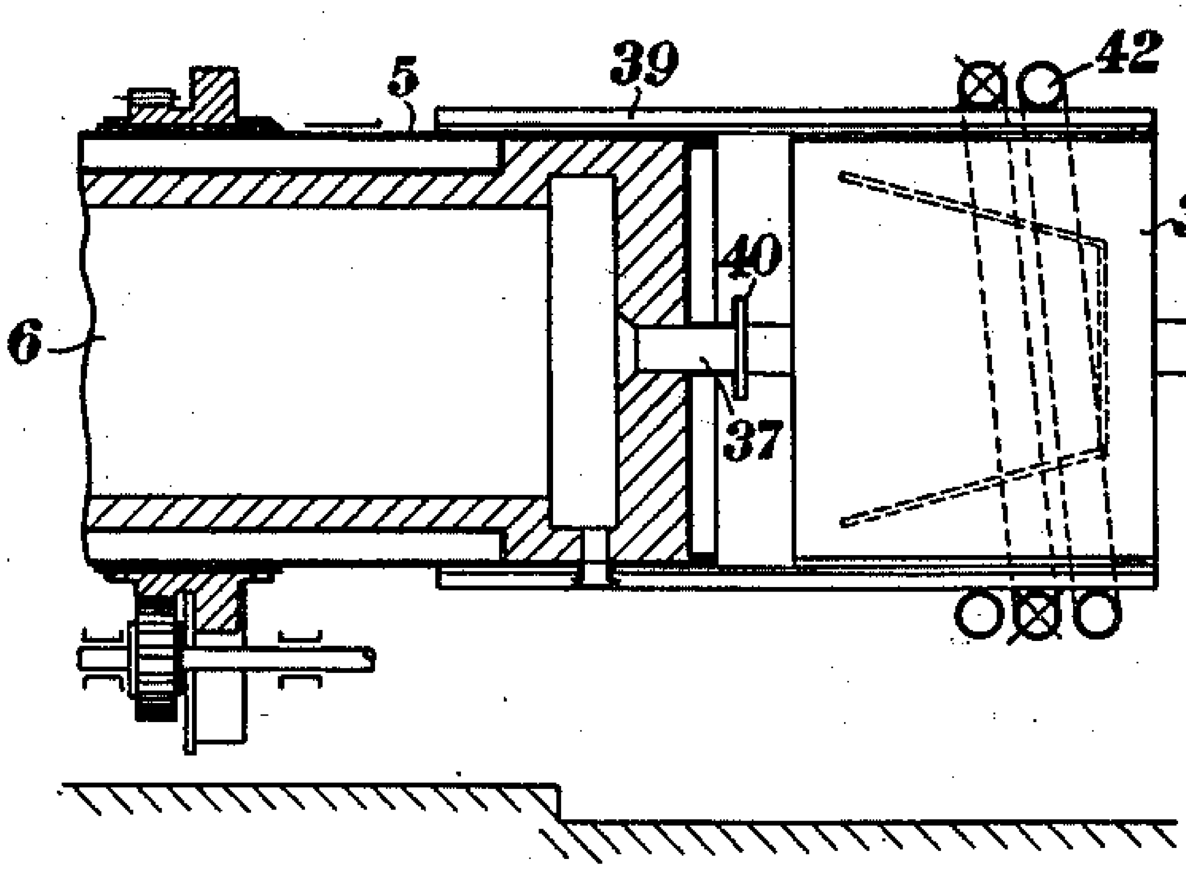


Fig. 5

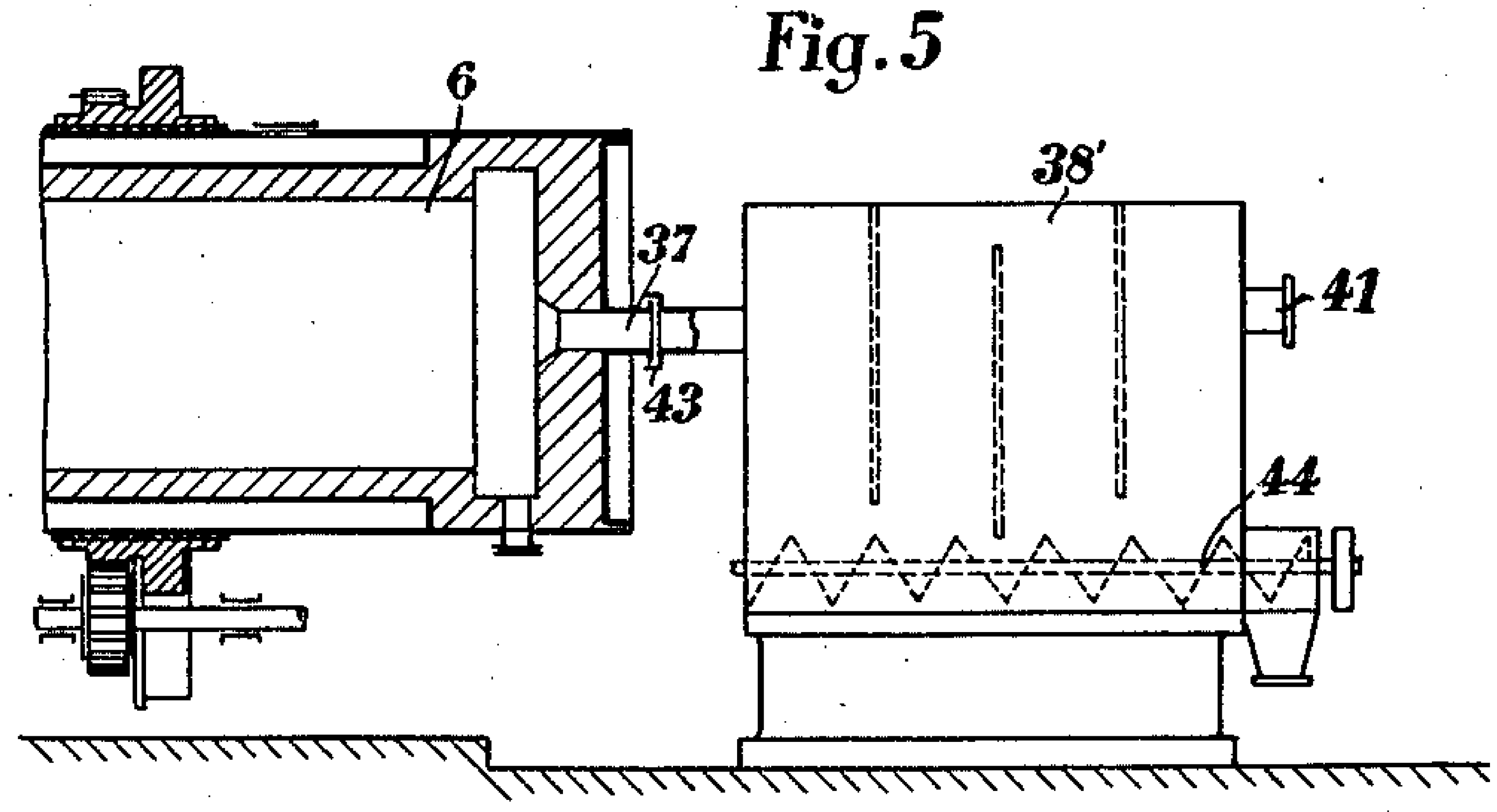


Fig. 6

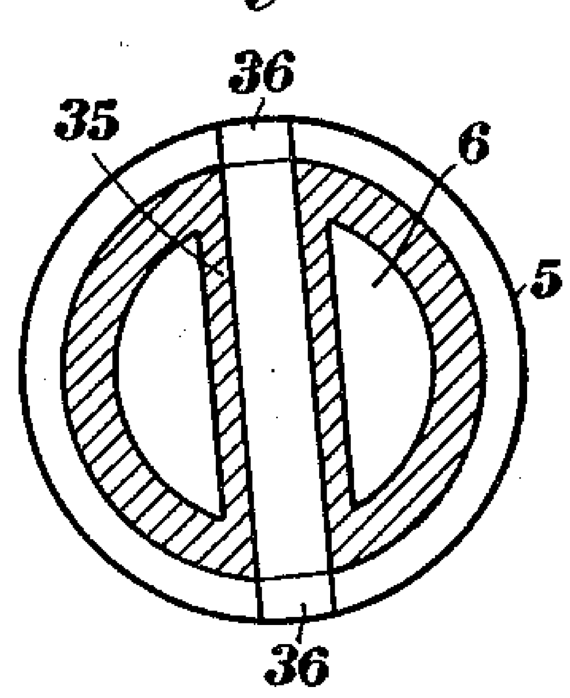
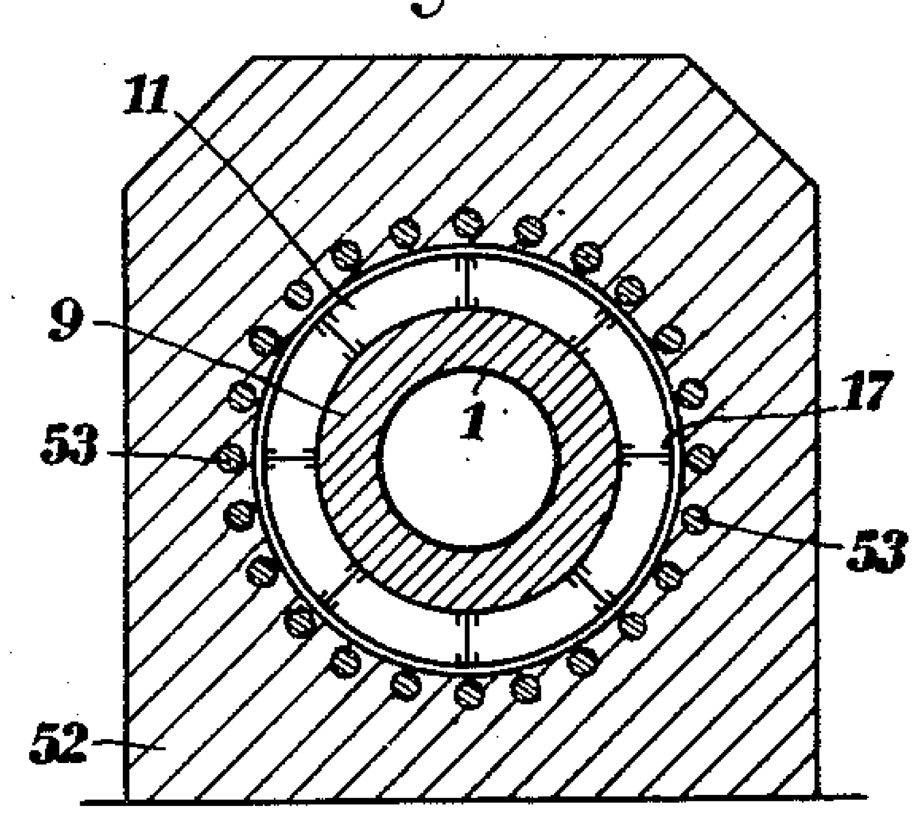


Fig. 9



Inventor
Friedrich Johannsen
By Cushman, Darby & Cushman
Attys

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APPARATUS FOR THE DISTILLATION OF ZINC AND OTHER VOLATILE METALS

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Fig. 7

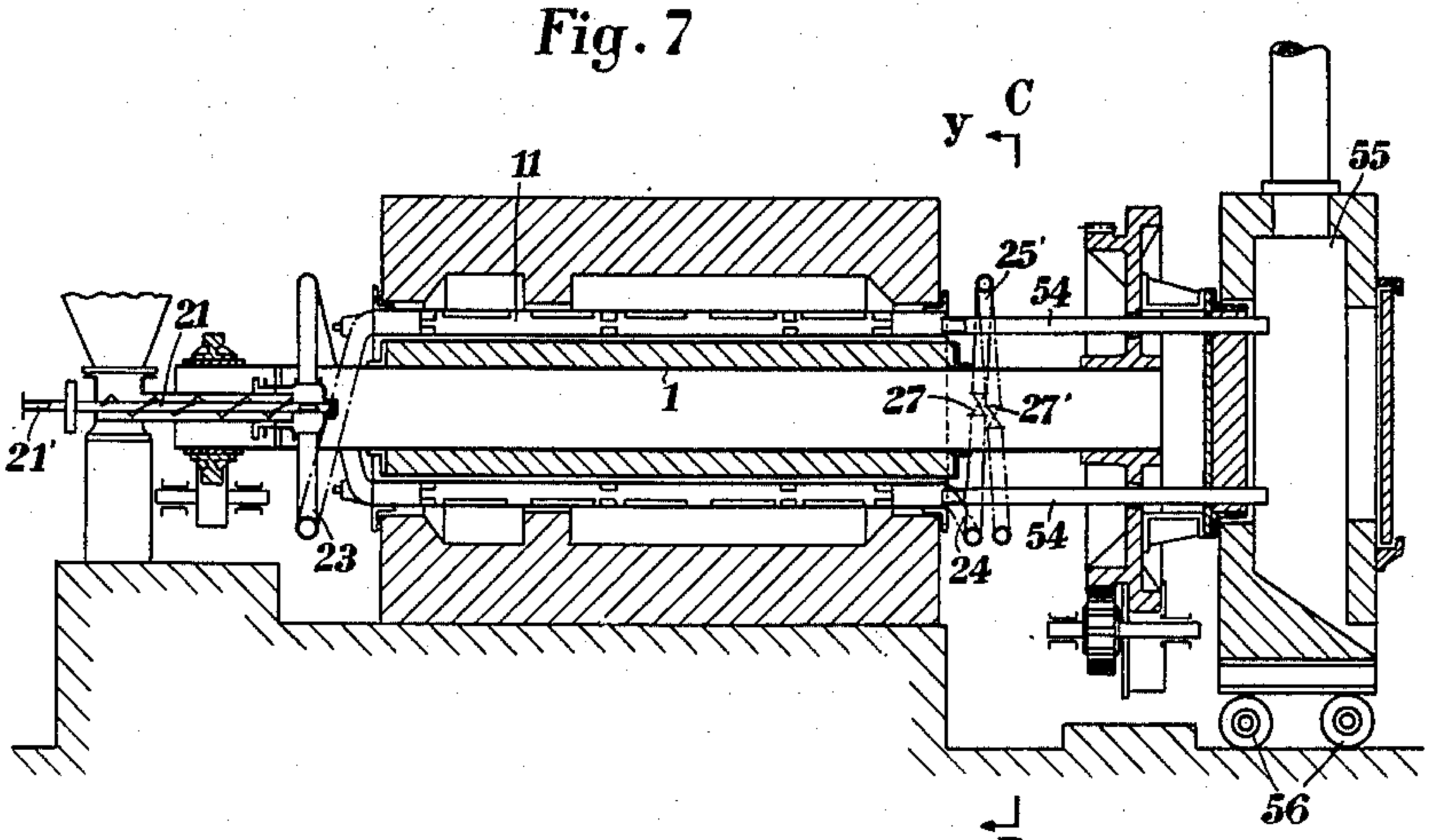
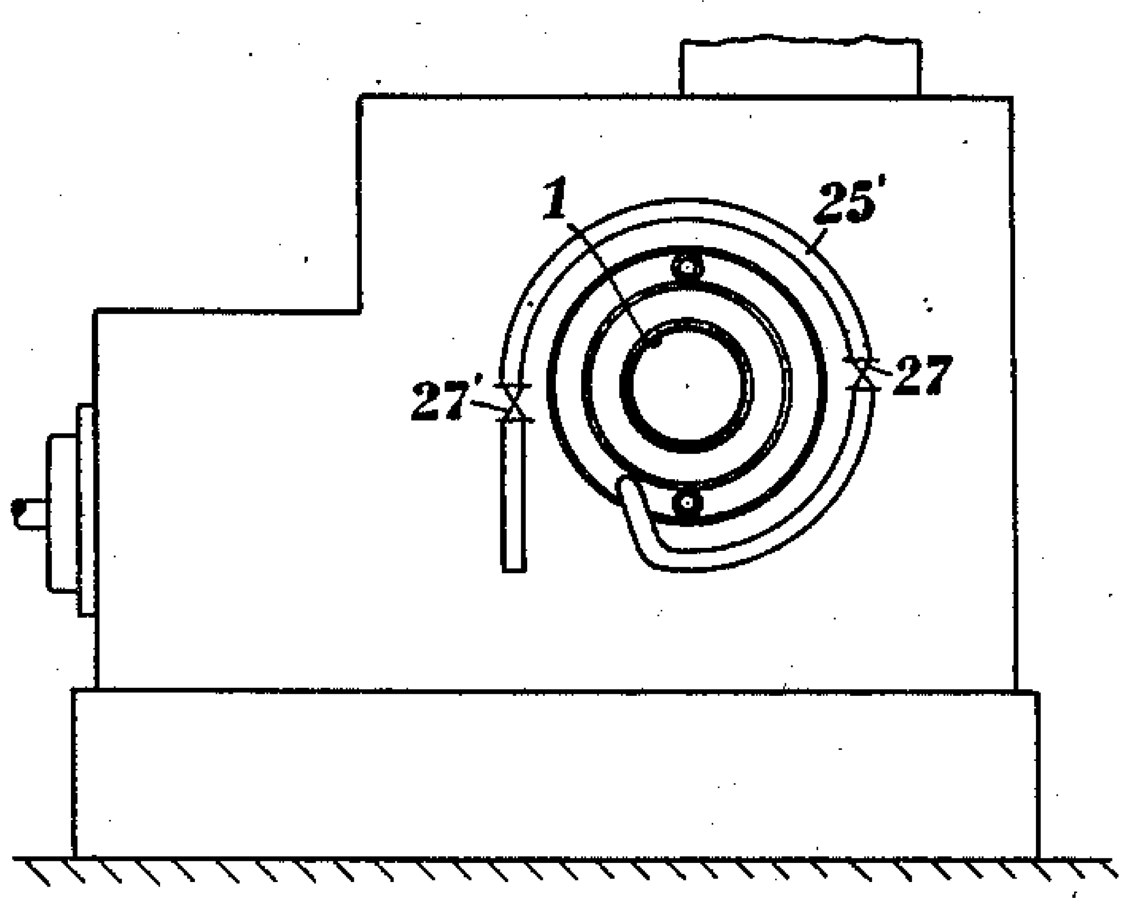


Fig. 8



Inventor
Friedrich Johannsen
By Cushman, Darby & Cushman
Attys

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2,007,332

APPARATUS FOR THE DISTILLATION OF ZINC AND OTHER VOLATILE METALS

Friedrich Johannsen, Magdeburg, Germany, assignor to Fried. Krupp Grusonwerk Aktiengesellschaft, Magdeburg-Buckau, Germany, a joint-stock company of Germany

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9 Claims. (Cl. 266—18)

It is known to distil easily distillable metals, such as zinc, cadmium and mercury, in a rotating muffle which is built in a revolving tubular furnace in such a manner as to rotate with it. The heating of the muffle is, as a rule, effected from the outside by a flame in the revolving tubular furnace. It has further been proposed to carry out the distillation of such metals in a muffle which is itself in the form of a revolving furnace which is partially enclosed within a stationary furnace chamber, the heating of the muffle that is in the form of a revolving furnace being effected in this furnace chamber.

These previously proposed muffle constructions have not been satisfactory, because in both cases the heated wall of the muffle must not only support itself but must also at the same time serve as a supporting frame for the whole muffle construction. It has consequently been necessary, when building one or more muffles into a revolving tubular furnace, to arrange the supports of the muffle at such small distances from each other that there was insufficient heating space on the outside wall of the muffle between these supports for the heat that is necessary for the endothermic distillation process. In the case of the proposal to make the muffle itself in the form of a revolving furnace, it cannot be supported on an outer brickwork revolving furnace wall as it is practically impossible to obtain a sufficient stability of construction.

According to the invention, there is to be used as a distillation vessel a muffle which is mounted on a carrying shaft and rotates therewith within a stationary heated furnace chamber. The muffle is formed as an annular chamber. This is arranged about a carrying shaft which is preferably insulated from the annular chamber. The material is fed continuously with exclusion of air to the annular muffle chamber, while the residue or reduction gases are also continuously discharged with exclusion of air. The muffle may be made of metals, especially of fireproof alloys, or of other suitable material—e. g., silicon carbide. The two muffle walls are closed at the ends in a gas-tight manner by rings which, if metals are employed for making the muffle, are advantageously welded to the muffle walls. This construction has, in comparison with those of the known proposals, the great advantage that the outer muffle wall which is directly heated and therefore has the highest temperatures, now no longer serves for supporting the whole construction but there is employed for supporting the construction an inner muffle wall which is mounted on a carrying shaft. The inner wall is not heated directly but entirely by conduction of heat—i. e. it is heated through the charge. It has a lower temperature than the charge, and is therefore more resistant than the outer heating wall whose temperature is about 200° C. higher. The construction has the further advantage that it enables muffles having a large diameter to be employed. According to experience, it is difficult when carrying out endothermic processes to introduce sufficient heat into the heart of a muffle. In the case of a rotating muffle in which the charge rotates during the rotation of the muffle and is turned over in doing so, it is, it is true, easier to obtain a uniform temperature in the whole of the charge, because the several parts of the charge are, during the rotation, continually mixed and frequently brought directly to the wall. Whilst, therefore, in the case of small rotating muffles, of a diameter of up to about 300 mms., a sufficiently practical equalization of the temperature within the charge can be obtained, in the case of an enlargement of the muffle to a diameter of about a metre, large differences of temperature within the charge are, in practice, to be considered. The heart of the muffle is therefore, in the case of large muffles, to be regarded as an unsatisfactory space. Now in the case of the present construction, this objectionable interior space is avoided by making the muffle merely an annular chamber; the space gained in this way enables a holding and rotating device that is very certain of operation to be built into the muffle in the form of the preferably insulated carrying shaft. In certain circumstances it may be advantageous to form the annular chamber of the muffle not of cylindrical form but of prisms whose bases may have three or more sides. The bearings of the carrying shaft are outside the furnace chamber. It will generally be advantageous to make the carrying shaft a hollow shaft; it may also be cooled by air or water in order to prevent excessive heating in working.

In many cases, especially in the case of difficultly distillable metals, it is advantageous not to employ a unitary annular muffle chamber but to resolve it as it were into one or more spiral passages which are placed round the carrying shaft. In this form the heating surface is substantially greater in relation to the throughput, so that the endothermic operations, such as reduction and vaporization, are carried out in a shorter time and in a more complete manner. This form has the further advantage that it is constructionally extremely simple and that, when the muffle is damaged, a replacement can be rapidly made at comparatively small cost by exchanging a spiral tube portion.

The muffle can, in that case, consist of one continuous spiral tube or of a number of parallelly arranged spiral tubes. The form that is preferable depends principally on the time that is necessary for the reduction and vaporization. In the spiral tube construction, the conveying of the material is positive, because the charge is advanced through one turn on every rotation. It is therefore advantageous, in the case of difficult distillation, to employ a single continuous spiral tube, whilst the arrangement of a number of parallel tubular passages offers advantages in the case of distillations which are easily effected. It is possible in this construction to adjust and regulate exactly the velocity of travel of the material, and, consequently, the time that is necessary for the distillation, by varying the number of revolutions.

When it is a question of distilling mercury, in which case the danger of a re-oxidation of the mercury vapour is absent, it is advantageous to pass the waste gases of the muffle into a stationary receiver. The same method is advantageous when, for example, zinc or cadmium is reduced and vaporized in the muffle and the vapours evolved are oxidized in a second process. In this case the gases would be brought to combustion after leaving the muffle. If it is a question of distilling zinc and cadmium and obtaining them as metals, it is advantageous to arrange the condenser chamber that is necessary for the condensation of metallic vapours in the extension of the carrying shaft and to connect it rigidly therewith. In this case, one bearing of the carrying shaft is advantageously fitted on the condenser chamber. The rigid connection of the condenser chamber with the carrying shaft has the advantage that a gas-tight closure can be made between the muffle and the condenser without difficulty. The rotation of the condenser chamber has the further advantage that its inner surface is continually wetted with liquid metal and the metallic vapours are more readily deposited on a wet wall. Fittings such as baffle plates etc. may be provided within the condenser for the purpose of enhancing the condensation action.

In order that the invention may be clearly understood and readily carried into effect the same will now be described more fully with reference to the accompanying drawings, which illustrate a number of embodiments of the invention, wherein:—

Figure 1 shows a muffle according to the invention in vertical longitudinal section, a condenser being directly connected to the muffle to rotate therewith.

Figure 2 is a cross-section on the line A—B of Figure 1 as seen in the direction of the arrow *x*.

Figure 2*a* is a cross section of Figure 1 on line 2*a*—2*a*, looking in the direction of the arrows associated therewith.

Figure 3 shows a construction of the muffle as a continuous spiral tube.

Figures 4 and 5 are longitudinal sections showing alternative forms of construction of the condenser.

Figure 6 is a cross-section through a further construction of condenser.

Figure 7 shows in longitudinal section a form of construction of the muffle without a condenser that rotates therewith.

Figure 8 is a section on the line C—D of Figure 7 as seen in the direction of the arrow *y*.

Figure 9 shows in cross-section an embodiment of a muffle of a special construction.

In Figures 1, 2 and 3, **1** denotes the carrying shaft which, in this case, is drawn as a hollow shaft and is mounted in a bearing **2** outside the furnace block **3** and is rigidly connected with the outer supporting jacket **5** of the condenser chamber **6** by the part **4**. In Figures 1 and 2 there is located at **7** a second bearing and at **8** there is located the drive of the whole rotatable part of the furnace. Around the carrying shaft **1** there is disposed an insulating layer **9** (Figures 1 to 3) which may be held together by a metallic jacket or metallic bands **10** (Figures 1 and 2). Over this metallic jacket **10** there is placed, according to Figures 1 and 2 the muffle **11** which consists of an inner cylindrical jacket **12** and an outer cylindrical jacket **13**. In this diagrammatic representation it is assumed that both jackets consist of a metallic alloy—e. g. a nickel-chromium-iron alloy or a silicon-chromium-iron alloy. The two cylindrical furnace walls are connected together at the ends **14** and **15** in a gas-tight manner by a ring which is welded to the walls. In order to maintain the distance between the two muffle walls throughout the whole length of the muffle, and, consequently, to support the outer muffle, there are provided, as indicated in Figures 2 and 2*a*, eight stays, comprising elongated radial plates **16** which may be rigidly connected with one or both furnace walls or may only be kept in their position in corresponding U-section guides **17** (see, for example, Figure 9). The muffle **11** communicates with the condenser chamber **6** through one or more openings **18**. The number of stays **16** between the walls of the muffle depends, in addition, on the size of the muffle. The stays divide the muffle into separate chambers, so that if the stays extend from one end of the muffle to the other, the annular chamber consists of many separate muffles. The charge of the muffle accordingly travels separately through each of these muffle sections. It may be advantageous, as shown in Figures 1 and 7, to provide the elongated plates or stays with interruptions or openings for the purpose of obtaining a sufficient communication between the individual sections. This may also be effected by all the stays being completely broken away at certain parts of the muffle, especially at its two ends. There are then produced, as shown in Figure 1, the two annular spaces **19**. The interruption of the stays may also be effected by their being only partially cut away at certain places. It is advisable to have these recesses at the outer wall of the muffle as shown at **20** in Figure 1. In this construction, the stays act for their greater part as scoops which, on the rotation of the furnace, lead the material away to the outer wall of the muffle, so that the material slides along this outer heated wall of the muffle and always remains collected in the lower portion of the whole muffle. The recesses or cut-away portions **20** in the stays may be arranged in all the stays to lie in similar positions or they may be so displaced in relation to each other that they together act as a worm and influence the progression of the material in the longitudinal direction of the muffle by acceleration or retardation. In this way a constant thorough mixing and eventual onward movement of the material within the muffle is ensured. The muffle may be slightly inclined or horizontal in accordance with the material to be worked up. The charging of the muffle is advantageously effected continuously and by means of a feeding device—e. g. a feeding worm 21 which is arranged in the axis of rotation of the carrying shaft and affords an air-tight closure owing to the fact that it is secured directly to the bottom portion of the charging bunker 22. The feeding worm may be firmly connected with the muffle by means of tubes 23 which are advantageously in the form of spirals. It is advisable to make these tubes of such a material—e. g. thin sheet copper—that, when the muffle expands on heating, they can yield sufficiently but are absolutely air-tight. The residue from the distillation leaves the muffle 11 (Figures 1 and 2) through one or more connecting pieces 24 which, in the construction according to Figure 1, are connected by a spiral tube 25 with the sluice chamber 26. A valve 27 is arranged at any desired position in the spiral tube 25 and a lid 28, which closes in an air-tight manner, is placed on the discharge end of the sluice chamber 26. By alternate operation of the valve 27 and the lid 28, the distillation residue is discharged with exclusion of air. Instead of the sluice chamber 26, there may be employed a continuous spiral tube 25′ in which the two shutting-off members 27 and 27′, in the form of valves or the like, are arranged one behind the other (see Figures 7 and 8).

On the condensation of the metal vapours, a considerable quantity of heat of condensation is liberated, which, in order to keep up the condensation sufficiently, must be continually carried away during working. It is therefore advisable to provide, in the wall of the condenser, cooling spaces through which is passed the air or water for carrying away the excess of heat.

In the construction shown in Figure 1, a hollow space 29 is provided within the outer jacket 5 of the condenser. This hollow space communicates, through suitably placed openings 32, with a stationary annular chamber 30 which is tightened against the rotating outer jacket 5 of the condenser by the packing 31. The annular chamber 30 is connected with a fan which can, in a regulatable manner, suck cooling air through the hollow jacket 29. The cooling air enters the cooling space through, for example, openings 33 that are adapted to be closed as required and, if desired, also through the pipes 34 from the hollow shaft. Many alternative means for cooling the condenser will readily occur to one skilled in the art.

Figure 6 represents a cross-section of such a construction. In this figure, 5 again denotes the outer jacket of the condenser and 6 denotes the hollow condensation space. A cross-tube 35, that is coated with fire-clay material for example, is passed through the condenser. On the rotation of the condenser, this cross-tube acts as a chimney and carries away a portion of the excess of heat out of the, for the time being, upper opening of the tube in the form of warm air. The arrangement of such cross-tube has the advantage that the condensation surface is substantially increased. In working, it is possible to close the openings of the cross-tubes at 36 wholly or partially by slides in any desired manner, so that, in accordance with their particular adjustment, the temperatures can be adjusted in the individual zones of the condenser. Instead of cooling by air, cooling by water or by water-spray may be employed, for example in the case of the arrangement of fittings diagrammatically represented in Figure 6. The waste gases of the condenser, which, in the example shown in Figure 1, leave it through the opening 37, may be directly burnt. The metal vapour that is still present then burns into metallic oxides which can be obtained from the waste gases in a small filter plant—e. g. a bag-filter house. Instead of this, the waste gases may first be allowed to pass through an auxiliary condenser or prolongation 38 (see Figures 4 and 5) which is advantageously made of a material that is a good conductor of heat—e. g. thin sheet iron—and effects the further cooling of the waste gases to below the melting point of the metal. The metals that are still present in the waste gases of the condenser, as in the present-day muffle process, are obtained in the greater part in the form of metallic dust.

Figure 4 represents diagrammatically how an auxiliary condenser 38 can be connected with the condenser 6. A supporting device—e .g. an angle iron construction 39—is rigidly connected with the outer jacket 5 of the condenser and the auxiliary condenser 38 is inserted into it and is firmly connected with the connecting piece 37, for example, by a flange 40. The auxiliary condenser thus rotates with the condenser and, like the condenser, can be cooled by air or water. The waste gases of the auxiliary condenser 38 leave it through the connecting piece 41. They still contain only a small quantity of metallic dust and can, if required, after further purification—e. g. a wet purification—be employed to contribute to the heating of the muffle 11. In this construction, the metallic dust can be removed from the auxiliary condenser 38 by, for example, a coil 42 provided wih shutting-off members. Instead of the rotatable auxiliary condenser, a stationary condenser 38′ can be employed, as diagrammatically represented in Figure 5. 6 again denotes the rotating condenser chamber, the waste gases of which pass out through the connecting piece 37. 38′ is a stationary condenser and 43 is a packing which connects the said condenser with the rotating connecting piece. 44 denotes a discharging device for the metallic dust in the form of a worm.

The flame-heating of the annular-chambered muffle is to be effected within a furnace block 3 as represented diagrammatically in Figures 1 and 2. The production of the flame is effected by first bringing the fuel mixture—e. g. gas, oil or coal dust and air—that is introduced through the burner 45, to ignition in the ante-chamber 46, and the formation of a too hot flame tip may be avoided by the use of Actinolite or Strahlite bricks 47. As shown in Figure 2, it is advantageous to rotate the muffle and the carrying tube in the direction of the arrow z, in which case the charge comes to lie in the annular-chamber muffle 11 between about the points E and F. The flame that is formed in the ante-chamber 46 consequently encounters the muffle directly in the filled zone, so that the hottest part of the flame can only encounter the muffle at a part where it is covered with the charge. In the case of the example of construction shown in Figure 2, the flame then passes in the direction of the arrow u—i. e. it passes in the opposite direction to that of the travelling movement of the muffle and in contact with the muffle up to the projection 48 of the brickwork. At this place it is carried away into an ante-chamber 49, as shown in the longitudinal section of Figure 1 and, in this preheating zone, again passes round the muffle and is finally carried away out of the furnace chamber by the chimney connection 50 that may be seen in Figures 2 and 2a. The employment of the waste heating gases from the main reaction zone 51 (Figure 1) for the preheating of the material in the preheating zone 49 reduces the fuel consumption that is necessary for carrying out the process.

The construction represented in Figures 7 and 8 corresponds substantially, with regard to the construction of the muffle itself, to the embodiment according to Figures 1 and 2. In this construction there is only omitted the condenser that, according to Figures 1 to 6, rotates with the muffle. The gases from the annular chamber 11 of the muffle pass through the tube 54 into a stationary collecting head 55 which serves for example as a condensation chamber for the material being worked up. This receiver may be mounted on the rollers 56.

Figure 9 represents diagrammatically a second method of heating the revolving furnace—i. e. with the aid of electrical heating resistances. 1 again denotes the carrying shaft which is surrounded by the insulating ring 9 and the annular chamber 11. Electrical resistances 53 are fitted in the furnace block 52 near its inner surface. The distance between the revolving muffle and the resistances is such that the small deflections of the outer heating wall that are possible in working cannot lead to a contact of the muffle wall with the resistances. The resistances themselves may consist of carbon, graphite or the like or they may be formed of resistance metals. The resistances are advantageously in the form of rods which lie parallel to the axis of rotation of the muffle, because the result obtained by this construction is that the individual parts of the muffle wall are carried past the resistances in a perpendicular direction and in this way a very uniform heating of the heating muffle can be obtained. The insulating material of the furnace block 52 that surrounds the heating resistances 53 may be divided up in such a manner that the individual resistances can be exchanged without any great interruption of the working.

According to Figure 3, the muffle is in the form of a spiral tube 57. The material goes from the bunker 22 through the worm 21 and thence by means of two distributing spirals into the spiral 57. At 58, the reduced material is let into an outer spiral 59 which is provided with sluice valves at 60 and 61 and periodically discharges at 62 the cooled reduced ashes that are left. In front of the connecting piece 58 there is arranged a connecting piece 63 for gas, which piece is connected with the condenser. The construction of the furnace and the condensing device may in this case be of different kinds.

The above described devices enable a continuous distillation process to be carried out for easily distillable metals such as zinc, cadmium, mercury and others, the charge that is to be reduced, and which normally consists of the oxides of these metals, being introduced into the annular-chambered muffle 11 or spiral 57 by the feeding device 21 and the tubular connection 23. As a reducing substance there is generally to be used a solid reducing agent such as fine coke, anthracite etc. A gasiferous coal, a liquid hydrocarbon or a gaseous hydrocarbon may, however, also be used as reducing agent, the supply of the liquid or gaseous hydrocarbon in the tubes 23 being effected through the shaft 21' of the worm 21 (see Figure 7) which is then formed as a hollow shaft. The residues of the distillation are carried away by the connecting piece 24 or into the spiral 59 and are discharged after suitable cooling and with the interposition of a sluice chamber. According to Figures 1 to 6, the metallic vapours pass through the connecting pieces 18 or 63 into the condenser chamber 6, where the greater quantity of the metal is obtained in liquid form, whilst the quantity of metallic vapour still contained in the waste gases is burnt to form oxide or is obtained in an auxiliary condenser as metallic dust. The waste gases of the auxiliary condenser may, if desired after purification from the metallic dust particles still contained therein, be employed for heating the muffle.

The embodiment according to Figures 7 and 8 principally comes into question for the production of pure metallic oxides such as zinc oxide and cadmium oxide. In this process, the metallic vapours are burnt in the receiver 55 by the addition of fresh air. The same embodiment may be employed in the distillation of mercury, for example, from rich flotation concentrates. In this case the receiver 55 serves merely for the condensation of a portion of the gases and the waste gases can then be cooled by a method that is at present commonly in use. Naturally, the embodiment according to Figures 1 and 2 with the condenser that is rigidly connected with the annular-chambered muffle may likewise be used for the distillation of mercury.

What I claim and desire to secure by Letters Patent of the United States is:—

1. Apparatus for the distillation of metals, such as zinc, cadmium, mercury and other volatilizable metals, comprising a stationary furnace chamber having heating channels therein, a rotatable shaft extending through said chamber, a body of insulating material on said shaft, a muffle surrounding said shaft and mounted thereon for rotation therewith, said muffle having its radially inner surface adjacent said insulating material and its outer surface exposed to said heating channels in the furnace chamber, means for charging material into one end of said muffle with the exclusion of air, and means for discharging the residue of the reduction gases from the muffle at the other end with the exclusion of air.

2. Apparatus for the distillation of metals according to claim 1 in which the stationary furnace further comprises a separate chamber in which primary combustion takes place and the heating channels, leading therefrom to the chamber in which the rotary muffle is located, debouch upon the outer wall of the muffle at its lower part in the zone where the material in the muffle forms a layer during rotation of the same.

3. Apparatus in accordance with claim 1, wherein radially arranged stays are disposed between the radially inner and outer walls forming an annular muffle by means of which stays the outer of the two walls is carried by the inner, the stays being at least partially interrupted in places between the muffle walls.

4. Apparatus in accordance with claim 1, wherein radially arranged stays are disposed between the radially inner and outer walls forming an annular muffle, by means of which stays the outer of the two walls of the muffle is carried by the inner, the stays being provided with openings therein which are staggered in relation to one another whereby the stays act as mixing and conveying means.

5. Apparatus in accordance with claim 1, wherein the supporting shaft for the muffle, adjacent the discharge end of the muffle, is rigidly connected to a rotating condenser, and a conduit leading from the muffle to the condenser.

6. Apparatus in accordance with claim 1, characterized by the fact that for discharging the completely reduced charge there is provided a sluice chamber rotating simultaneously with the muffle and shaft, said chamber having two successively operated closing members, which are connected with the muffle by one or more pipe unions.

7. Apparatus in accordance with claim 1 in which the rotating muffle discharges metal in gaseous state with a rotating condenser and wherein cooling spaces are arranged in the wall of the condenser for cooling off the condensation heat.

8. Apparatus in accordance with claim 1 in which the rotating muffle discharges metal in gaseous state into a rotating condenser and wherein air-cooled or water-cooled built-in devices are disposed inside the condenser chamber for cooling the condenser.

9. Apparatus in accordance with claim 1 in which the rotating muffle discharges metal in gaseous state into a rotating condenser and wherein an extension member connected with the condenser and rotating therewith is employed for recovering metal dust from the waste gases.

FRIEDRICH JOHANNSEN.