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C. T. KNIPP.

DEVICE FOR THE PURIFICATION OF METALS.

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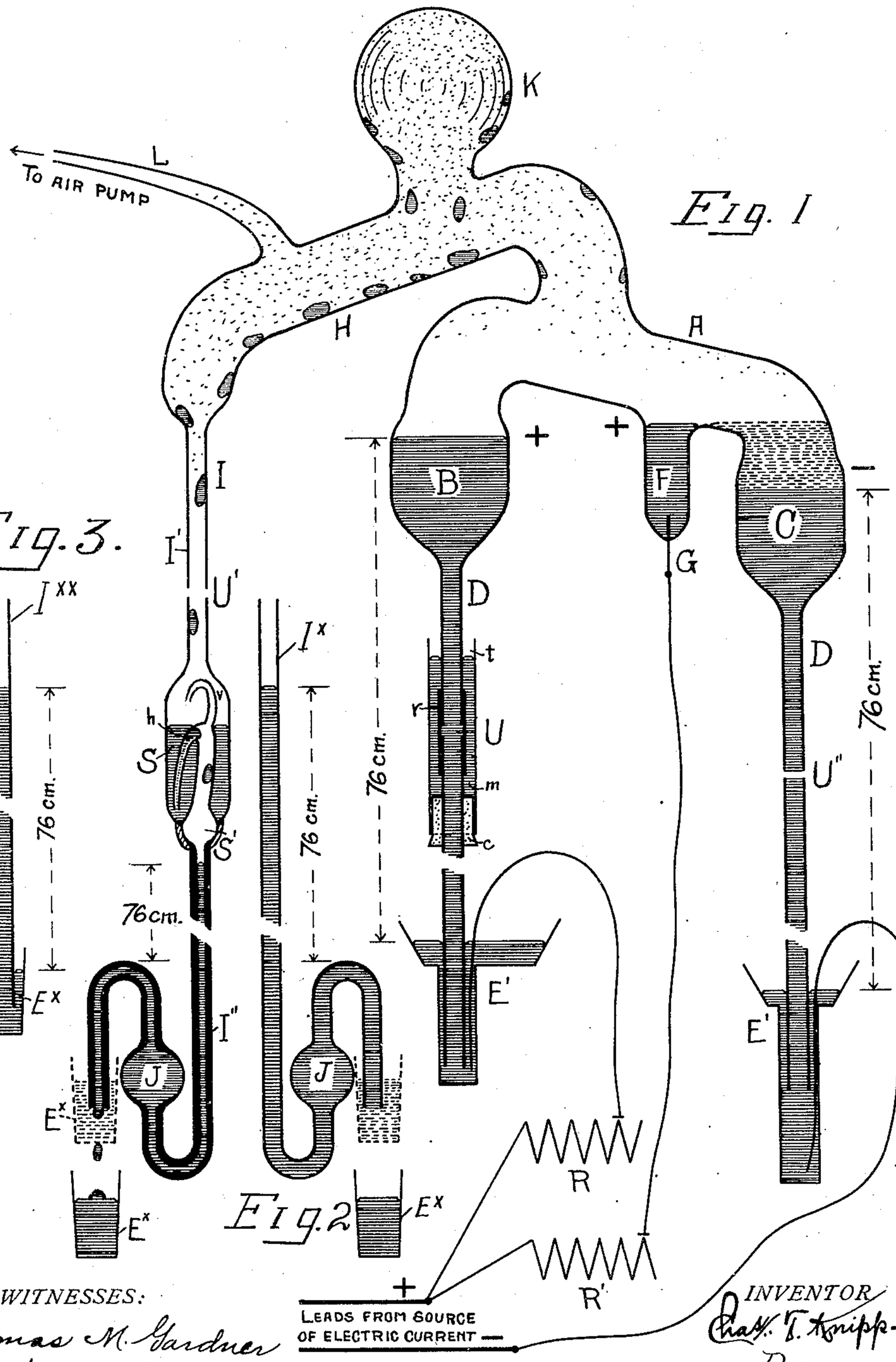

Fig. 1

Fig. 3.

Fig. 2

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DEVICE FOR THE PURIFICATION OF METALS.

No. 891,264. Specification of Letters Patent. Patented June 23, 1908.

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To all whom it may concern:

Be it known that I, CHARLES T. KNIPP, a citizen of the United States, residing at Urbana, in the county of Champaign and State of Illinois, have invented certain new and useful Improvements in Devices for the Purification of Metals, of which the following is a specification, reference being had to the accompanying drawings.

My invention relates to improvements in devices for the purification of metals and particularly to electrical devices for that purpose.

The mode of operation is to vaporize the metal by generating an arc and to condense the vapor. My new device is, therefore, particularly adapted to the purification of metals in a liquid state, and of mercury in particular.

The object of my invention is to provide a device of the character described which will give a metal distillate of a high degree of purity, and which will operate at an extremely low cost and require little skill in its attendance.

In the drawing illustrating the principle of my invention and the best mode now known to me of applying that principle, Figure 1 is an elevation of my new device; and Figs. 2 and 3 illustrate modifications hereinafter referred to.

The ends of the tube A are bent downwardly and formed with the bulbs B, C from each of which extends a small bore tube D, the lower end of which dips into a vessel E′. Intermediate the bulbs B, C the tube A is formed with a recess or pocket F in the bottom of which is fused a platinum wire G for a purpose hereinafter referred to.

The tube A is in communication with (opens into) one end of the condensation tube H the other end of which is extended downwards by the tube I which terminates in an S provided with an enlargement or reservoir J. The upper portion I′ of the tube I is of comparatively large bore, while the lower portion I″ of the tube I is of small bore, or a capillary tube. Between the portions I′ and I″ is an enlargement or chamber S within which is a hollow valve S′, which prevents any flow of mercury from the reservoir S into the capillary tube I″ except through the valve itself. From near the top of the valve S′ extends downwardly a tubular inlet arm *h* which reaches to near the bottom of the chamber S; and extending upwardly from the valve is a vent-piece *v* which permits air to be exhausted from the capillary tube I″ through the valve S′. The latter traps the mercury in the bulb S; and when the mercury has reached the level of the point of junction of the arm or inlet tube *h* with the top of the valve S′, the mercury will escape through the valve S′ into the capillary tube I″. In this way any dross or impurity floating on the top of the mercury in the chamber S is prevented from escaping into the capillary tube I″.

The condensation tube H is provided with the condensation chamber K and is formed with a nipple L for the attachment of a rubber hose or like means of connection to a Geryk hand or power air-pump (not shown), or other equally effective air-pump.

The upper portions of the tubes D and I are made separate from their lower portions as is indicated at U, U′ U″. This construction enables the upper part of the apparatus to be separated from the lower part, for the purpose of cleaning. When the apparatus is in use, the spaces U, U′ U″ are bridged over or closed by a piece of rubber tubing *r*. When the tubing is new, it will be found generally to suffice in keeping the joints airtight. In order to reinforce the tubing *r* and hermetically seal the joint, a jacket made up of a cork *c* and tube *t* filled with mercury *m* is used. Although only one of the joints (that marked U) is shown fitted with this fermeture, it will be understood that the other two joints U′, U″ are also sealed, when the apparatus is in use.

The mercury in one of the vesels E′ is connected with the negative pole of a suitable source of electric current, as a dynamo, storage battery or the like. Preferably direct current is used. The mercury in the other vessel E′ is electrically connected through the resistance R with the positive pole of the source of electric current; and with this same positive pole the platinum wire G of the side connection F is connected through the resistance R′. Of course, it will be understood that the mercury in the tubes D and the bulbs B, F and C serve as conductors for the electric current. The mercury in the bulb C acts as a return circuit for the current which flows through the mercury in the bulbs B and F.

The length of the capillary portion I″ of

the tube I is such that the level of the column of mercury sustained by the atmosphere in the capillary tube I″ is about five centimeters below the bottom of the chamber S. The volume of the bulb J is sufficient to insure against the admission of air.

The vessels E′ are made with their upper portions wide and shallow or dish-shaped, and resemble somewhat a frying pan from which extends downwardly a pipe. This shape enables the vessels E′ to hold a large volume of mercury without altering in a great degree the mercury level in the bulbs B and C.

To start the still: Place a beaker E× of clean mercury at the lower end of the tube I″ allowing the latter to dip into it, as shown by the dotted outline. Place the vessels E′ in position as shown and fill with the mercury to be distilled. Connect the nipple L to an air-pump, (a Geryk hand or power pump is well adapted for this purpose), and slowly exhaust the air from the still. The mercury will rise in the tubes I and D, and after 15 or 20 minutes of pumping, it will become stationary. Clearly the columns of mercury are supported by the pressure of the atmosphere. Now adjust the position of one of the vessels E′ until the mercury stands in the bulb B at the level indicated in the drawing. Next raise the other vessel E′, until the mercury rises in the bulb C and overflows, filling the pocket F, after which the vessel E′ is lowered so that the column stands in the bulb C at the level indicated by the heavy shading.

The volume of the bulb J is sufficient to insure against the admission of air, hence, the beaker E× at the lower end of I″ may now be lowered to the position indicated by the full line sketch. Electrical connections are now made as indicated in the drawing. There is a divided circuit, one branch of which includes the resistance R and the mercury in the bulb B, while the other branch includes the resistance R′ and the mercury in the pocket F; the common return for the two branches is the mercury in the bulb C. The side connection F is purely to start the arc between the electrodes B and C as described by Weintraub, *Phil. Mag.*, Vol. VII, February, 1904, but the arc may be started quite readily by dispensing with the side connection F and simply raising the level of the mercury columns in the bulbs B and C by raising the vessels E′ until the two columns touch, and then lowering the vessels. Or it may be started by the method of using a high potential discharge, as originally used by Hewitt and others.

After the arc is once started between the bulbs B and C (by the Weintraub method), the circuit through the side connection F is broken. The mercury vapor fills the tube A, the tube H and the condensation bulb or chamber K, upon the walls of which it condenses. The shape and position of the tube H and the condensation chamber K are such that the condensed mercury is diverted into the tube I and thence is discharged through the valve S′ into the beaker E× placed to receive the distillate as shown in the full lines in the drawing. As the purified metal falls drop by drop through the capillary tube I″, it operates as in an air-pump of the Sprengel type and tends to maintain the vacuum produced by the Geryk pump used in starting the apparatus. However, experience indicates that it is advisable to operate the latter pump from time to time, especially if the impurities give off gases, as they are very liable to do. If desired, the tube may be made of the same bore throughout, the bore being the same as that of the upper portion I′, in which case the chamber S is omitted but the S-shaped end and the bulb J are retained. (See Fig. 2, in which I× is the tube.) In this case the dropping of the purified mercury will not operate to maintain the vacuum. Again, the discharge of the purified mercury may be made to take place through a straight tube I×× (Fig. 3) dipping into a vessel, as do the tubes D into the vessels E′.

While the physical conditions existing, such as temperature, vacuum and the like, are such as to favor the vaporization of zinc present as an impurity in the mercury, yet the results of tests with my new device indicate a suppression of the zinc ions by the electric forces, the distillate of mercury being practically entirely free of all trace of zinc. The action of the device is equally effective in freeing the mercury of other impurities commonly found in it, such as gold, copper, tin, brass and the like.

I claim:

1. In combination, means for generating *in vacuo* an electric arc and vaporizing the metal thereby; means for condensing the vapor; and means for separating and discharging the metal distillate.

2. In combination, electric means for vaporizing the metal *in vacuo;* means for condensing the vapor; and means for separating and discharging the metal distillate.

3. In combination with a suitable source of electrical energy, a receptacle containing the metal to be purified; a condensation chamber which communicates with said receptacle; and a discharge tube for the metal distillate leading from said chamber, the air pressure in said receptacle and chamber being less than atmospheric pressure.

4. In combination with a suitable source of electrical energy, a pair of coöperating receptacles containing the metal to be purified; a condensation chamber which communicates with said receptacles; and a discharge tube for the metal distillate leading from said chamber, the air pressure in said recep-

tacles and chamber being less than atmospheric pressure.

5. In combination, electric means for vaporizing the metal to be purified *in vacuo;* a condensation chamber wherein the metal vapor is condensed; and a capillary discharge tube leading from said chamber, said tube serving to maintain the vacuum.

6. In a device for purifying mercury by distillation, the combination with means for vaporizing the metal *in vacuo* and condensing the metal vapor, of a discharge tube formed with a chamber intermediate its ends, the portion of said tube which leads downwardly from said chamber being capillary; and a hollow valve provided with an inlet tube extending from the top of said valve to the bottom of said chamber.

7. In a device for purifying mercury by distillation, the combination with means for vaporizing the metal *in vacuo* and condensing the metal vapor, of a discharge tube formed with a chamber intermediate its ends; and a hollow valve in said chamber, said valve controlling the passage from chamber.

Dated at said Urbana this 31st day of July, 1906, in the presence of two witnesses.

CHARLES T. KNIPP.

Witnesses:
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