No. 788,315. PATENTED APR. 25, 1905.

W. HOOPES.

METHOD OF ELECTROLYTIC SEPARATION.

APPLICATION FILED NOV. 30, 1904.

Fig. 1.

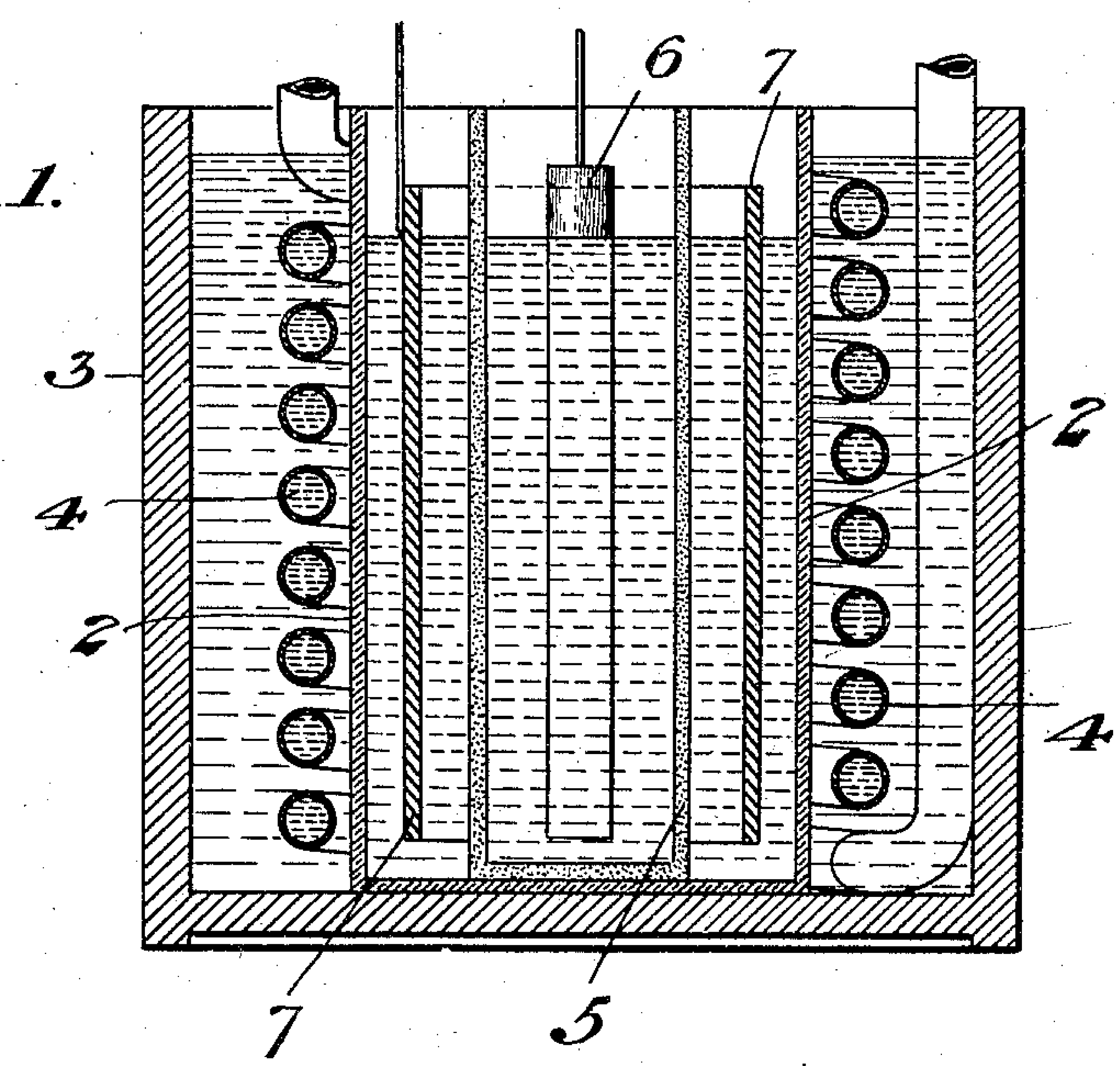
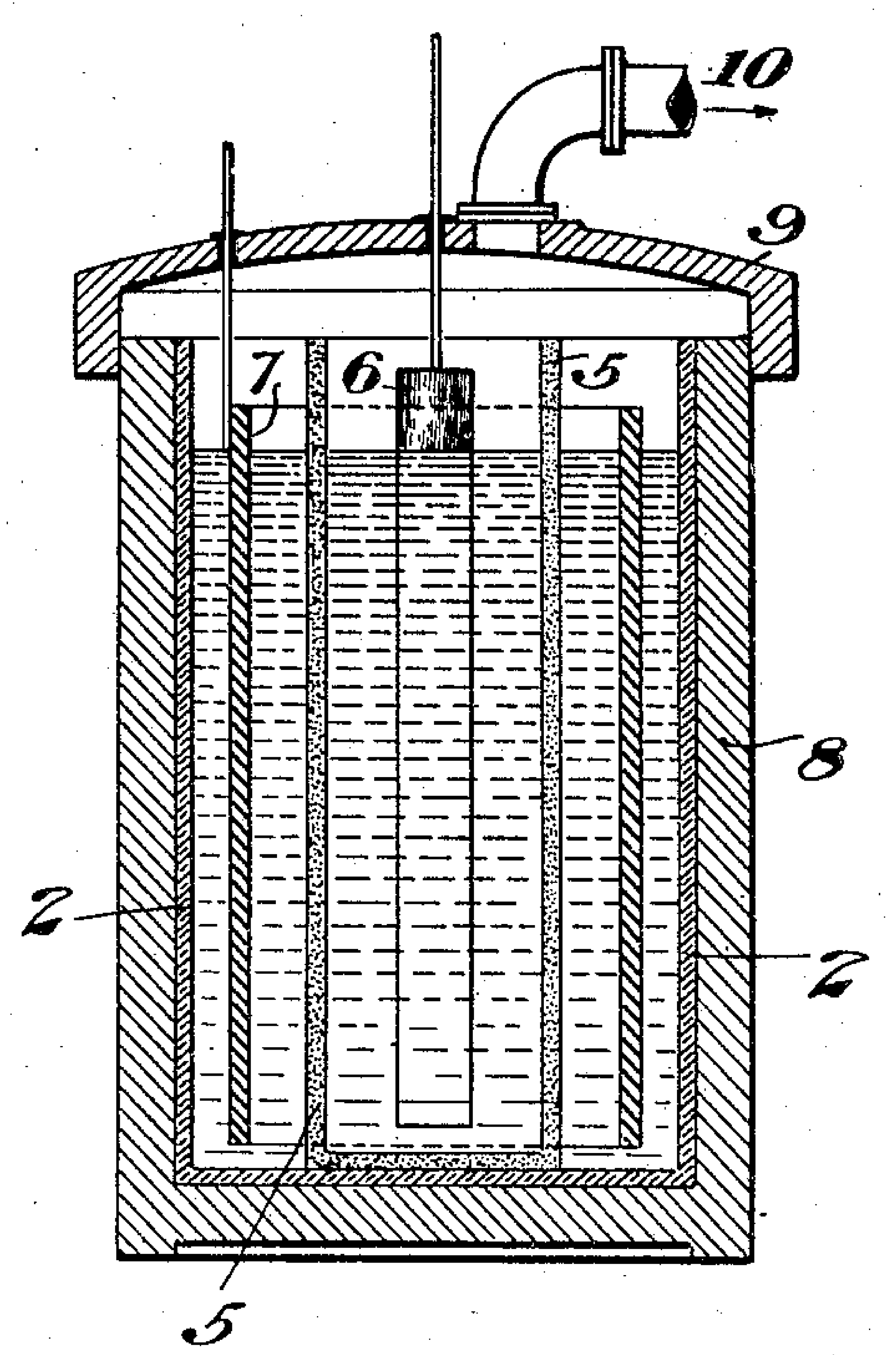


Fig. 2.



WITNESSES

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No. 788,315. Patented April 25, 1905.

UNITED STATES PATENT OFFICE.

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METHOD OF ELECTROLYTIC SEPARATION.

SPECIFICATION forming part of Letters Patent No. 788,315, dated April 25, 1905.

Application filed November 30, 1904. Serial No. 234,886.

To all whom it may concern:

Be it known that I, WILLIAM HOOPES, of Pittsburg, Allegheny county, Pennsylvania, have invented a new and useful Method of Electrolytic Separation, of which the following is a full, clear, and exact description, reference being had to the accompanying drawings, forming part of this specification, in which—

Figure 1 is a sectional side elevation of one form of apparatus adapted to carry out my improved method of electrolytic separation. Fig. 2 is a similar view of a modified form of apparatus.

Many of the metals cannot be readily separated from aqueous solutions of their salts by reason of the fact that, the heat of formation of the oxids of the metals being greater than the heat of formation of water, the deposited metal immediately decomposes the water and produces as the result of the operation the oxid of the metal and hydrogen. I have discovered, however, that if the salts of these metals be dissolved in liquid anhydrous ammonia (NH_3) the solution can be readily electrolyzed and the metal obtained therefrom without trouble, since the metals do not decompose ammonia.

My invention is especially applicable to the obtaining of magnesium by electrolysis of a solution of magnesium chlorid in anhydrous ammonia; but sodium and potassium can be produced freely in the same way, and in general I believe that the process is applicable to the electrolytic extraction of the metal of any anhydrous salt which is soluble in anhydrous ammonia, such as the cyanids, acetates, &c.

I will describe the invention with reference to the separation of magnesium, which is almost impossible to effect by electrolysis of an aqueous solution; but it should be understood that I do not limit my invention thereto, but intend to claim it generically.

My invention requires the use of a vessel in which the ammonia can be held in liquid condition. For this purpose I may employ the apparatus shown in Fig. 1, in which 2 is a glass vessel adapted to contain the liquid ammonia. It is set in an outer vessel 3, made of or lined with wood or other material which is a poor conductor of heat. The outer vessel 3 is filled with alcohol or other liquid of very low freezing-point, and it contains a coil of pipe 4, through which a refrigerating fluid—such as carbonic acid, ammonia, or the like—is caused to circulate. This chills the alcohol, which in turn chills the liquid ammonia in the vessel 2 and prevents substantial loss by evaporation.

The vessel 2 contains a porous cup 5, in which is an anode 6, preferably of carbon, and in the space between the walls of the cup 5 and vessel 2 is a cathode 7, made, preferably, of iron. Both compartments of the cell are filled with liquid anhydrous ammonia. Magnesium or other metallic chlorid is dissolved in the anode-compartment, and anhydrous magnesium chlorid with or without a little sodium chlorid is dissolved in the cathode-compartment. Both the solution of sodium chlorid in the anode-compartment and the solution of magnesium chlorid in the cathode-compartment are preferably saturated solutions, an excess of magnesium chlorid being preferably added either loosely on the bottom of the vessel or in a suspended bag. Upon passing current through the cell from the anode to the cathode magnesium is deposited on the cathode and chlorin is liberated in the porous cell, combining with the ammonia in case an insoluble anode, as carbon, is used and with the material of the anode itself in case a soluble anode, as zinc, is used. I have found a current of five volts and a current density of .1 ampere per square inch of anode-surface suitable in the separation of magnesium; but my invention is not limited thereto. As the solution in the cathode-compartment is impoverished it is replenished from time to time by the addition of more magnesium chlorid.

Instead of the refrigerating-coil shown in Fig. 1 I may employ the apparatus shown in Fig. 2, in which the vessel 2 is simply inclosed in a heat-insulating jacket 8, so as to retard the evaporation of the ammonia as much as possible, and the vessel is provided with a cover 9, having a pipe 10, by which the ammonia-vapor as it is liberated is conducted to a suitable condenser. Many other forms of apparatus suitable for the purpose may be devised by the skilled mechanic.

Although my process is more particularly applicable to the separation of such electropositive metals as can be deposited with difficulty or not at all from aqueous solutions, it nevertheless possesses general advantages for carrying out electrochemical operations which may also proceed in presence of water. Thus the migration velocities of the ions are higher in ammonia than in water, which renders solutions in anhydrous ammonia particularly available for electrochemical work. Further, certain metals—such, for instance, as iron and zinc—which can be deposited from aqueous solutions are obtainable with less expense, in a better physical state, and in a condition of higher purity when separated from solutions of their salts in ammonia. I have separated sodium, potassium, magnesium, chromium, iron, and silver by following methods similar to that described for magnesium, and there appears to be no reason why many other metals cannot be so produced. The process can also be used for the electrolytic refining of metals in like manner as it is done with those metals which are now purified by electrolysis in aqueous solution. Thus I have purified iron by immersing an iron cathode and an iron anode containing silicon and carbon in a solution of iron cyanid and potassium cyanide in ammonia, and on passing current have obtained pure iron on the cathode, the carbon and silicon not being dissolved at the anode. The obtaining of pure iron is a valuable commercial application of the process, since pure iron is now very costly, but very desirable for the production of grades of iron and steel free from phosphorus and other deleterious impurities.

I claim—

1. The method of electrolysis, which consists in passing an electric current through a solution of a compound in anhydrous ammonia.

2. The method of separating metal electrolytically, which consists in passing an electric current through a solution of a compound of such metal in anhydrous ammonia.

3. The method of separating metal electrolytically, which consists in passing an electric current through a solution of an anhydrous salt of such metal in anhydrous ammonia.

4. The method of separating metal electrolytically, which consists in passing an electric current through a solution of an anhydrous salt of such metal in anhydrous ammonia, a porous diaphragm being interposed between the electrodes.

5. The method of separating magnesium electrolytically, which consists in passing an electric current through a solution of a compound of magnesium in anhydrous ammonia.

6. The method of separating metal electrolytically, which consists in passing an electric current through a solution of a compound of such metal in anhydrous ammonia, and applying a refrigerating medium to the solution to prevent or retard evaporation of the solvent.

7. The method of refining metals, which consists in electrolyzing a solution of the salt of the metal to be refined in anhydrous ammonia, by the passage of the current through such solution, using as an anode a mass of the metal containing impurities of such a nature that all of them will remain undissolved or if dissolved will remain in solution, the pure metal only being deposited on the cathode.

In testimony whereof I have hereunto set my hand.

WILLIAM HOOPES.

Witnesses:
JOHN MILLER,
H. M. CORWIN.