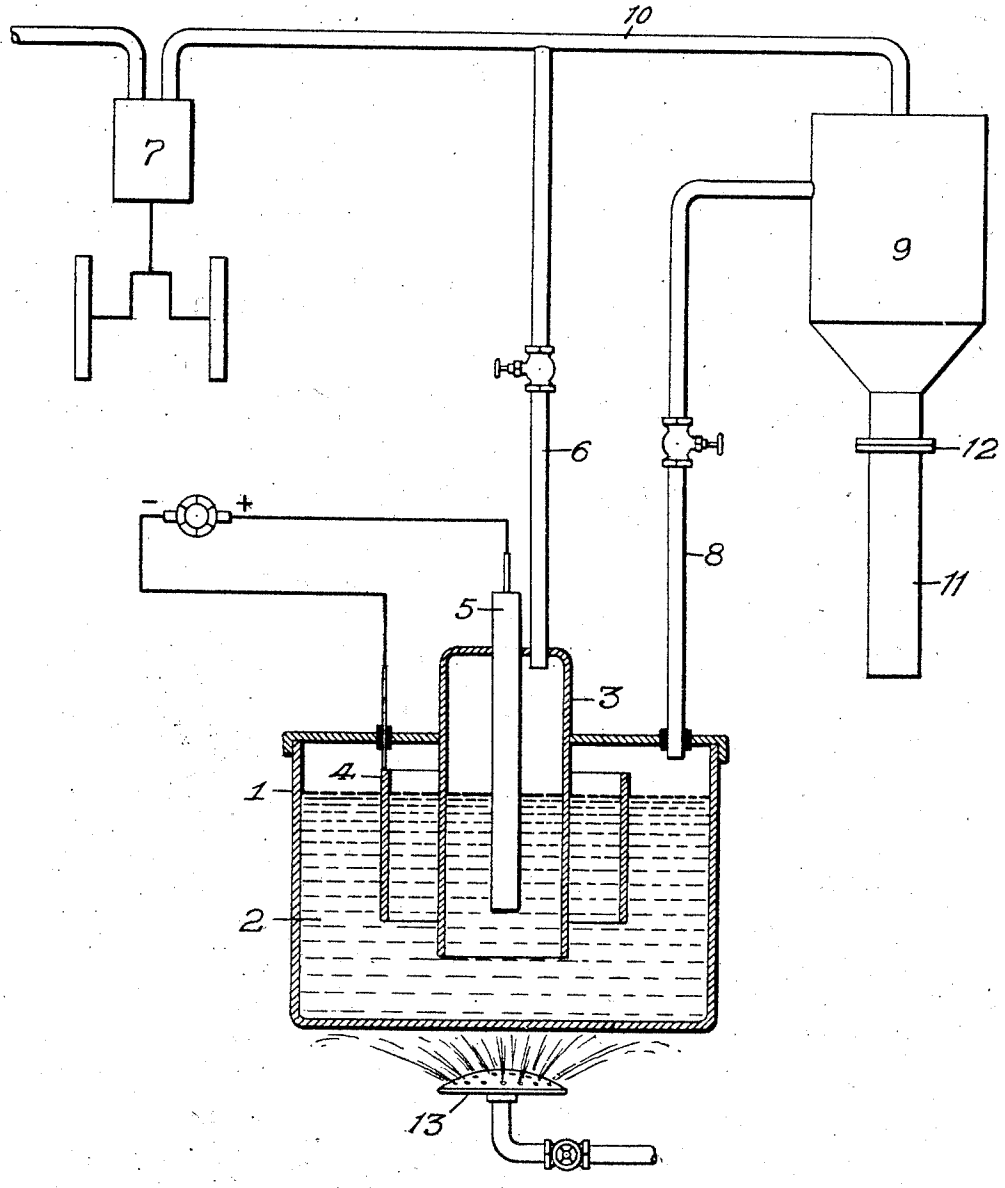
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ELECTROLYTIC PRODUCTION OF ALKALI METALS

Filed March 23, 1922



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ELECTROLYTIC PRODUCTION OF ALKALI METALS.

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The chief object of this invention is to provide a process for the manufacture of alkali metals, particularly sodium, potassium and magnesium, whereby such metals may be produced in greater purity and at a higher efficiency than by present methods.

The alkali metals are now commonly made by the electrolysis of molten or fused solutions of their compounds. The chief difficulty of the present processes is the protection of the metal from atmospheric oxygen and nitrogen as well as from some of the byproducts of the electrolytic decomposition itself.

My invention in its preferred form consists in electrolyzing molten compounds containing the desired metal, removing some or all of the products including the metal as vapors and afterwards condensing the metal separately.

Taking magnesium as a specific example, fused carnallite or any suitable compound of magnesium is melted and electrolyzed at a suitable temperature, preferably above 650 degrees centigrade, in an air-tight vessel capable of withstanding an external pressure of, say, one atmosphere or more. The magnesium will evaporate if the internal pressure is reduced to less than one inch of mercury, and the evaporation is at a sufficient rate to remove it from the solution rapidly. It will of course evaporate at a lesser rate if the pressure in the cell is greater. At the lower pressure, however, the reaction between magnesium and various gases is greatly reduced in intensity due to the greater attenuation of the gases and magnesium vapor. The result is a larger yield of metal and the occlusion of less of these gases in the metal produced.

Sodium, potassium, and magnesium are especially adapted to treatment by this process on account of their low melting and boiling points. The generally accepted melting and boiling points of these materials are as follows:

	Melting point.	Boiling point.
	° C.	° C.
Sodium	97.6	877.5
Potassium	62.5	757.5
Magnesium	650	1,120

Each of these metals is easily vaporized at several hundred degrees below its boiling point if the pressure is sufficiently reduced.

Apparatus convenient and suitable for practicing the invention in the preferred manner is illustrated diagrammatically in the accompanying drawing, in which the apparatus is shown partly in section.

Referring to the drawing, 1 is a closed electrolytic cell partially filled with a molten electrolyte 2 containing one or more compounds of the metal desired. The electrolyte is separated into two portions near the surface by a porcelain or other suitable hood or partition 3. Upon subjection of the mass to a direct or continuous current between cathode 4 and anode 5, preferably composed of iron and carbon, respectively, the metal is produced around the cathode and certain gaseous by-products are generated around the anode. The gaseous portions of the anodic products are withdrawn through valved pipe 6 by vacuum pump 7. Due to the low specific gravity of the metallic product it rises to the surface of the molten liquid around cathode 4 and there vaporizes. The vapors are withdrawn through valved pipe 8 to condenser 9 which is exposed to the cooling effect of the atmosphere or any other heat absorbing medium. The vapor of the metal withdrawn into condenser 9 by the vacuum pump 7 acting through pipe 10 is liquefied and falls into receptacle 11 which is removably attached to condenser 9. The liquefied metal product is retained in receptacle 11 until the end of the run and is then recovered by detaching receptacle 11 at the point 12 and withdrawing the product in either the liquid or solid condition.

Where an extremely low pressure is attainable or where an extremely pure metal is not desired, the hood 3 may be dispensed with and all of the products of the electrolysis drawn through pipe 8 and condenser 9 where the metal will be deposited by condensation and the uncondensed vapors withdrawn through pipe 10 by vacuum pump 7.

Some additional heat may be required for the evaporation of the metal product and it is therefore desirable in some cases to utilize more or less of the heat of condensation to maintain or aid in maintaining the temperature of cell 1, or to supply this extra heat by the use of suitable auxiliary means. As indicative of the latter method a gas burner is shown at 13.

The use of a vacuum electrolytic cell not only excludes air but as previously stated diminishes the occlusion of the gases by

the metal and the recombination of the gases and metal. Both these results are believed to be due to the attenuation and consequent reduced mass action of the gases.

As raw materials for my process the alkali metal chlorids are preferred. Moreover, they are cheap and have relatively low melting points. Carnallite is a double chlorid of potassium and magnesium ($KCl.MgCl_2.6H_2O$) occurring in the Stassfurt deposits. It is also possible to use the uncrystallized residue from the manufacture of sodium chlorid from sea water or terrestrial brines, commonly known as bittern. These residues nearly always contain all the alkali metals. For the manufacture of sodium it is also possible to use sodium hydroxid, sodium carbonate or sodium nitrate, and the corresponding potassium compounds are also usable for making that metal, but at present their cost is in general prohibitive. In some cases, especially when using naturally occurring mixtures containing the salt or salts, it is desirable to add one or more substances to obtain a suitable or advantageous melting point or prevent side reactions. Among the substances suitable for such purpose may be mentioned sodium chlorid, sodium sulfid, ammonium chlorid, and calcium fluorid. It is generally desirable to operate at a rather higher temperature than has commonly been employed heretofore, in order to get the increased vapor tension which is an advantage in the present process but which in the past has been a source of loss and annoyance.

The voltage and current employed may vary considerably, depending upon the usual factors, such as the resistance of the bath; the size of the cell and the effectiveness of the heat-insulation used with it, if any; the amount of heat supplied from an external source, if any; etc. Hence no general rule as to voltage and current can be given. It may be stated, however, that for making magnesium from carnallite with extraneous heat, a voltage of 5 or 6 volts is sufficient, with a current density of from 10 to 15 amperes per square decimeter. Without extraneous heat it is generally desirable to operate with 8 to 10 volts or higher and 40 or more amperes per square decimeter.

It is to be understood that the invention is not limited to the procedure and apparatus herein specifically described and illustrated but can be practised in other ways without departure from its spirit.

What I claim is:

1. A process for the production of metals, comprising electrolyzing a molten bath containing a compound of the metal desired, and withdrawing the products separately in vapor or gaseous form at a pressure less than atmospheric.

2. A process for the production of metals, comprising electrolyzing a molten bath containing a compound of the metal desired, at a temperature above the melting point of the metal, and withdrawing the anodic and cathodic products in vapor or gaseous form at less than atmospheric pressure.

3. A process for the production of metals, comprising electrolyzing a molten bath containing a compound of the metal desired, at a temperature above that of the melting point of the metal, and withdrawing the anodic and cathodic products in vapor or gaseous form separately at a pressure less than atmospheric.

4. A process for the production of metals, comprising electrolyzing a molten bath containing a compound of the metal desired, at a temperature at which the metal has an appreciable vapor tension, and withdrawing the anodic and cathodic products in vapor or gaseous form at a pressure below atmospheric.

5. A process for the production of metals, comprising electrolyzing a molten bath containing a compound of the metal desired, at a temperature at which the metal has an appreciable vapor tension and withdrawing the anodic and cathodic products separately in vapor or gaseous form at a pressure below atmospheric.

6. A process for making magnesium, comprising electrolyzing a fused salt of magnesium at a temperature sufficient to vaporize the magnesium produced, withdrawing the anodic and cathodic products separately in vapor or gaseous form, and condensing the magnesium vapor so withdrawn.

In testimony whereof I hereto affix my signature.

PIERRE E. HAYNES.