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PROCESS FOR PRODUCING ANHYDROUS ALUMINUM CHLORIDE.
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1,343,662.

Patented June 15, 1920.

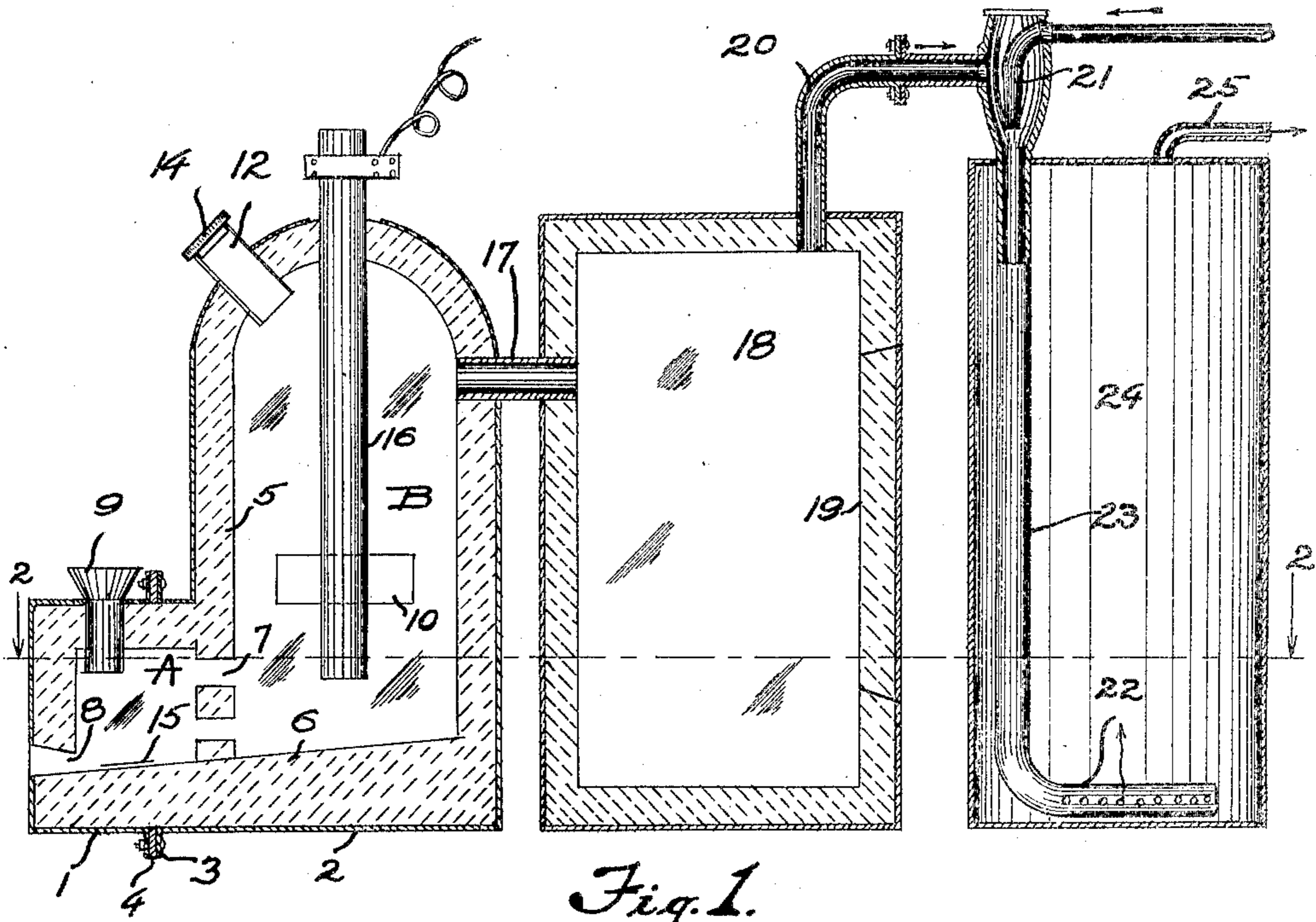


Fig. 1.

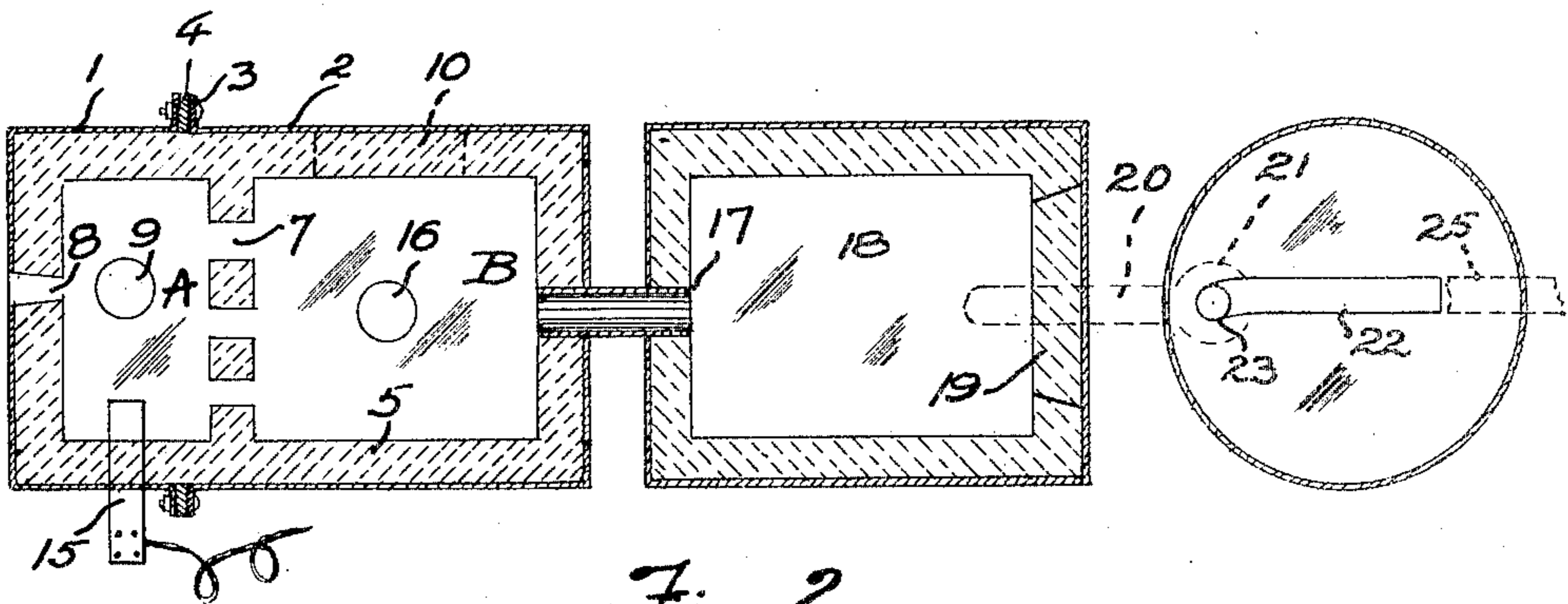


Fig. 2.

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UNITED STATES PATENT OFFICE.

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PROCESS FOR PRODUCING ANHYDROUS ALUMINUM CHLORID.

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Specification of Letters Patent. Patented June 15, 1920.

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To all whom it may concern:

Be it known that I, PAUL DANCKWARDT, a citizen of the United States, residing in the city and county of Denver and State of Colorado, have invented certain new and useful Improvements in Processes for Producing Anhydrous Aluminum Chlorid; and I do declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same, reference being had to the accompanying drawings, and to the characters of reference marked thereon, which form a part of this specification.

This invention relates specifically to a process for producing aluminum chlorid by electrolysis of a molten metal chlorid, while a mixture of substances containing an aluminum compound is in contact with the positive pole, or is itself forming the positive pole, the current in decomposing the metal chlorid, causing metal to be separated at the negative pole and chlorin at the positive pole. The metal is withdrawn at intervals; the chlorin reacts with the aluminum of the aluminum compound in the vicinity of the positive pole, forming anhydrous chlorid, which volatilizes and is condensed in a suitable chamber. This part of the process takes place at an elevated temperature in that part of the apparatus where electrolysis is taking place.

As a raw material for the supply of the aluminum compound, I may utilize the cake or residue obtained in the process of treating mineral oils with anhydrous aluminum chlorid, as carried out in making gasolene from such oils. This residue at present is a waste product and practically of no value, as the aluminum chlorid contained therein has become ineffective. Or, I may use a mixture of aluminum oxid and carbon. The aluminum oxid may be that obtained as a by-product in a certain process for producing zinc chlorid from the residue above described. As an electrolyte, I prefer to use zinc chlorid, although other chlorids may be used, for instance, lead or copper chlorids. The chief object of this invention is to utilize the ineffective aluminum chlorid in the residue above described directly, and thereby regenerate it, and also to provide a use for the aluminum oxid and zinc chlorid produced as above mentioned.

In the accompanying drawing:

Figure 1 is a vertical section through one

form of apparatus in which the process may be carried on.

Fig. 2 is a horizontal section, taken on the line 2—2 of Fig. 1.

The apparatus comprises an electrolyzer composed of two sections having steel shells, 1 and 2, with their flanges 3 bolted together and insulated by asbestos layers 4. The two shells 1 and 2 are provided with an inside lining of fire brick 5, which is projected downward and rests upon the inclined bottom fire brick lining 6, and has a number of openings 7 in the lower part thereof to offer free communication between the two compartments A and B, as shown. At the extreme left of the compartment A, an opening 8 through the wall serves as a tap-pole for drawing off the molten contents. A pipe 9, projecting down into compartment A, enters the molten bath when the apparatus is filled, and serves for the introduction of fresh material. This pipe 9 should be made of non-conducting and heat-resisting material.

The compartment B is provided with a door 10 for the removal of charge, and a charging duct 12, with cap 14, is provided for the introduction of charge.

The negative electrode 15 is formed by a strip of iron bolted to the steel shell and projecting therethrough to form a communication between a layer of molten zinc inside the apparatus and the current supplying means outside. A carbon rod 16, forming the positive pole, passes through the top of compartment B, its outer end being connected with the current.

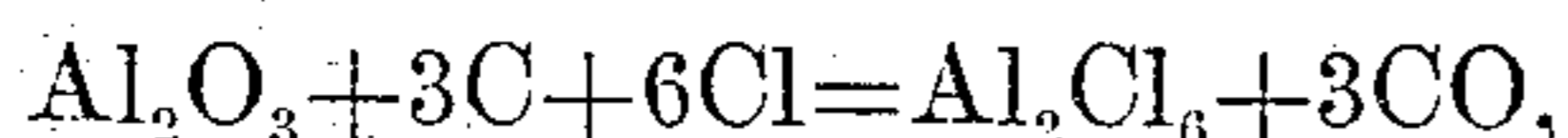
A conduit 17 connects compartment B of the electrolyzer with a condensing chamber 18, which is built of brick and provided with a smooth surface on the inside, preferably glazed bricks, from which the solid, condensed salt may be easily detached for removal through the door 19. Any material not condensed is sucked off through the pipe 20, to a steam exhaust 21, by means of which it is forced through the perforated extension 22 of the pipe 23, which extends down into the lower portion of the absorber 24, the latter, when in operation, being filled with water holding zinc oxid in suspension. The latter is for the purpose of collecting any hydrochloric acid or free chlorin escaping from the condenser 18. The absorber 24 consists of a lead-lined, iron or wooden tank, and has an outlet 25 for waste gases.

In carrying on the process with this ap-

paratus, the absorber 24 is filled with water and zinc oxid, as stated. A mixture containing aluminum compound is then introduced through the duct 12 into the chamber B.

5 The amount of carbon in this material should be such that it is at least sufficient to take up all the oxygen of the aluminum oxid, forming carbon monoxid. The residue before mentioned contains hydrocarbons and
10 carbon sufficient for this purpose, while if aluminum oxid is used, carbon must be added in proper proportions.

Melted zinc chlorid, or other electrolyte, is then introduced through the pipe 9 in
15 sufficient quantity to fill up to the line 2—2 of Fig. 1. The current is then turned on immediately. As the current finds the greatest resistance in passing from the carbon electrode through the mixture contain-
20 ing the aluminum compound, it is here that the most heat is produced. Therefore it is advisable in the beginning not to let the carbon pole itself dip into the molten electrolyte, as the zinc chlorid is a very good
25 conductor of electricity and otherwise there would not be sufficient current transformed into heat to bring the charge up to the required temperature. When the charge becomes red hot, a large amount of gas is
30 evolved, consisting principally of carbon monoxid and hydrochloric acid, with some hydrocarbons, if the above-described residue is used, and almost entirely of carbon monoxid with a little chlorin if carbon and
35 aluminum oxid are used. As the charge becomes hotter and the electrolysis of the zinc chlorid increases, copious fumes of anhydrous aluminum chlorid are generated. The gases and vapors not condensed in condenser
40 18 are forced through the water containing the zinc oxid in suspension in the absorber 24. The zinc set free at the bottom of the electrolyzer is drawn off through the outlet
45 8 before its level becomes high enough to short-circuit the apparatus by spreading out into the compartment B, where it would come in contact with the conducting charge around the positive pole. The liberated chlorin permeates the charge around the
50 positive pole and will react with the aluminum oxid and carbon, according to the equation



55 if a mixture of aluminum oxid and carbon is used. If the above-described residue is used, the reaction is more complicated and may be expressed by some such equation as



60 This latter equation is hypothetical, but it expresses the fact that a certain amount of free hydrochloric acid is liberated.

The aluminum chlorid condenses in the
65 condensing chamber 18 from which it is re-

moved through the door 19, whenever a sufficient amount has accumulated. The exhaust 21 is operated at such a rate that no aluminum chlorid, but all of the gases, are
70 carried over and forced through the emulsion of zinc oxid. Zinc chlorid will be formed by the reaction of the hydrochloric acid with the zinc oxid, and when the solution becomes saturated with zinc chlorid, it
75 may be filtered, the filtrate evaporated, and the residue of zinc chlorid melted down and used in the process as a part of the electrolyte.

The aluminum chlorid thus produced is not pure, as it invariably contains some zinc
80 chlorid volatilized along with the aluminum chlorid. This contamination, however, is not objectionable when the aluminum chlorid is to be used for making gasolene. However, it may be freed of the zinc chlorid by
85 resubliming in a retort fired from the outside, the zinc chlorid remaining behind.

I claim:

1. A process of producing anhydrous aluminum chlorid which consists in electro-
90 lyzing a metal chlorid in contact with an aluminum compound so that the chlorin in nascent state can react with it, forming anhydrous aluminum chlorid.

2. The process of producing anhydrous
95 aluminum chlorid, which consists in electrolyzing molten metal chlorid while the positive pole is in contact with a charge containing carbon and an aluminum compound, allowing the chlorin from the chlorid to re-
100 act with the charge to form an aluminum chlorid, and condensing the vapors of aluminum chlorid so formed.

3. The process of producing anhydrous aluminum chlorid, which consists in elec-
105 trolyzing molten metal chlorid, using a mixture of carbon and an aluminum compound at the positive pole at such temperature that the aluminum chlorid formed volatilizes, and condensing the latter.
110

4. The process of producing anhydrous aluminum chlorid, which consists in elec-
115 trolyzing molten zinc chlorid in contact with carbon and an aluminum compound at the positive pole, causing the liberated chlorin to react with the charge to form anhydrous aluminum chlorid, maintaining the temperature sufficient to volatilize the anhydrous aluminum chlorid formed, and condensing
120 the latter.

5. The process of producing anhydrous aluminum chlorid, which consists in elec-
125 trolyzing molten zinc chlorid, thereby separating it into metallic zinc and chlorin, causing the latter in nascent state to react with aluminum oxid and carbon to form aluminum chlorid, and condensing the volatilized aluminum chlorid.

In testimony whereof I affix my signature.
PAUL DANCKWARDT.