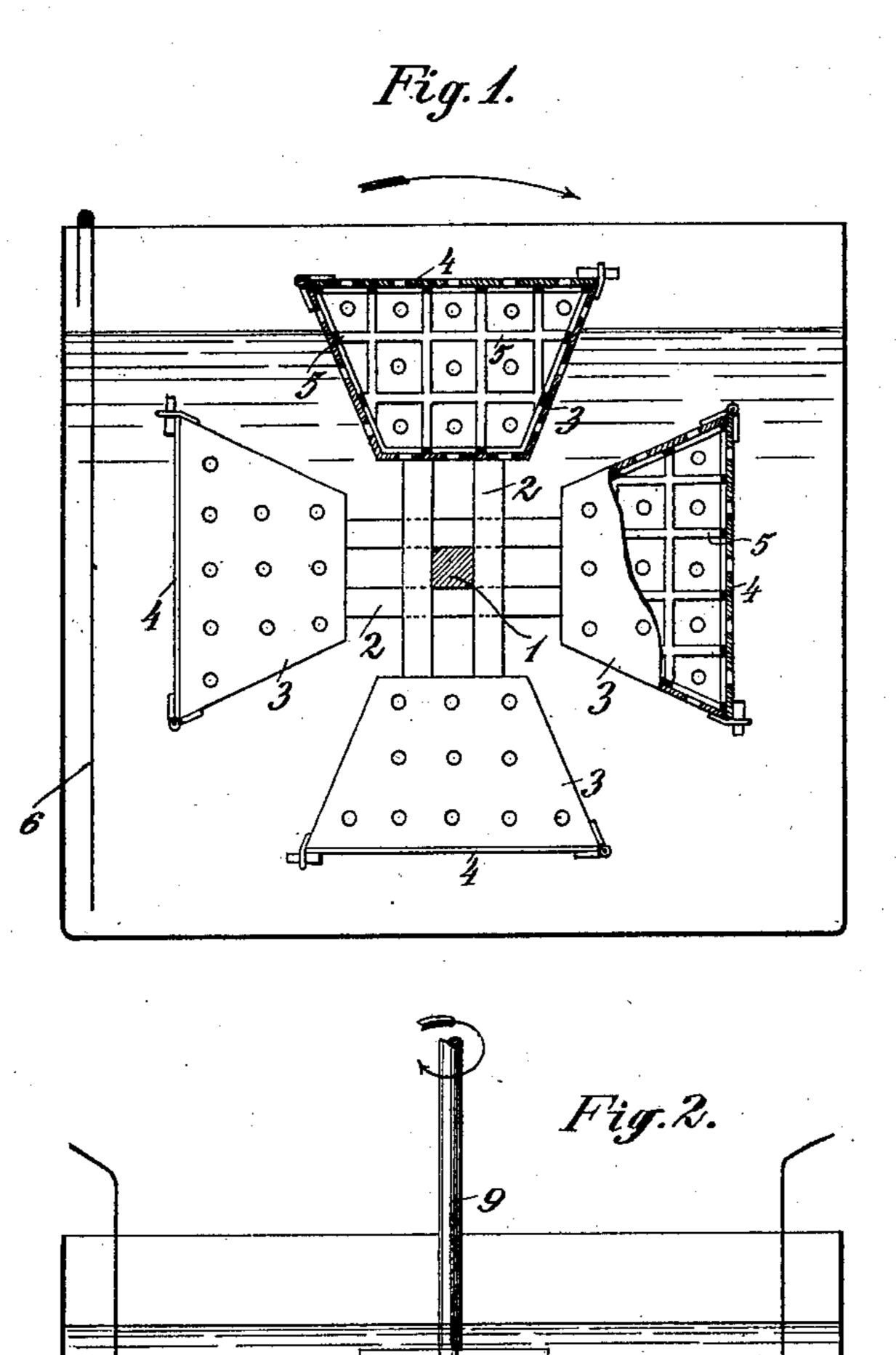
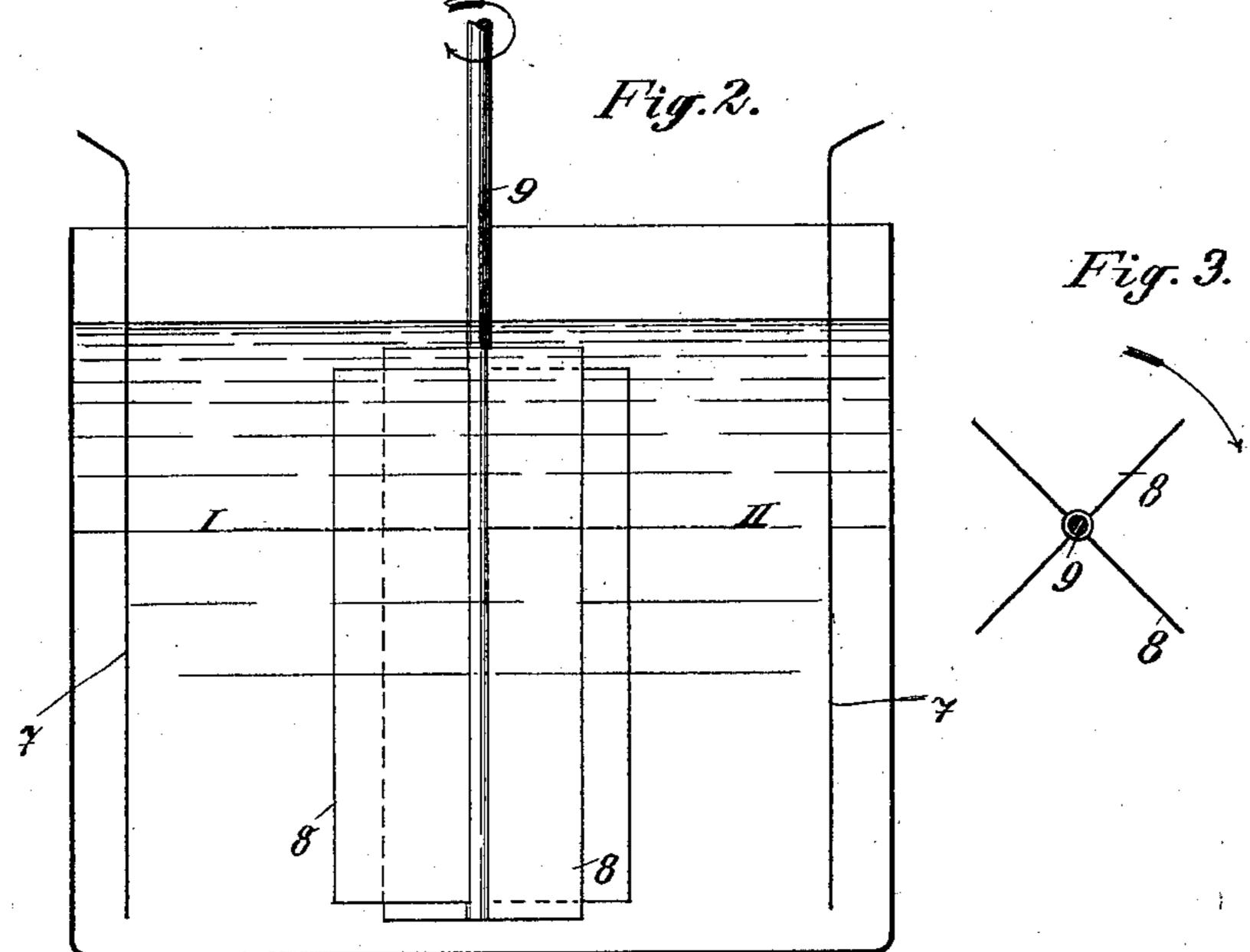
## H. PAWECK. ELECTROLYTIC REFINING OF ZINC. APPLICATION FILED MAR. 7, 1902.

NO MODEL.





WITNESSES: Deabella Waldron: Adelaide Clave Glesson. Sinrich Paweck.

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## United States Patent Office.

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## ELECTROLYTIC REFINING OF ZINC.

SPECIFICATION forming part of Letters Patent No. 745,378, dated December 1, 1903.

Application filed March 7, 1902. Serial No. 97,074. (No specimens.)

To all whom it may concern:

Be it known that I, HEINRICH PAWECK, Ph. D., engineer and chemist, of 36 Alseggerstrasse, Vienna XVIII, Austria-Hungary, 5 have invented certain new and useful Improvements in the Electrolytic Separation, Deposit, and Refining of Zinc, of which the fol-

lowing is a specification.

This invention relates to the electrolytic 10 separation, deposit, and refining of zinc, the process according to this invention being characterized by the use of new baths and special mechanical devices, as hereinafter described. The baths differ from those hitherto 15 employed mainly in the use of boric acid or borates in solutions of salts, some of which salts, but not others, have already been used in the electrolytic separation of zinc.

The aforesaid special mechanical devices 20 have for their object to insure in even especially difficult cases a fine deposit of metallic

zinc.

By means of the process according to this invention it is possible to readily, rapidly, 25 and thickly deposit zinc on objects of iron or other metals or of alloys or on objects not made of metals, but specially prepared for receiving a deposit of zinc or to deposit zinc on plastic masses, the deposit being effected 30 in all cases without having to resort to special measures of precaution. The separation of zinc takes place without the production of heat and generally without its being necessary to stir the bath mechanically or to re-35 move and scrape the articles upon which the deposit is being made. However, in some special cases the electrolyte may be agitated with advantage, simple devices for this purpose being hereinafter described.

As by the process according to this invention very thick layers of zinc can be deposited, it is also possible to successfully carry out the refining of zinc in the bath, as its composition is eminently suitable for this purpose,

45 this being of importance for zinc-etching. Likewise the electrolyte itself may be used for the lixiviation of ores and zinc waste. Solutions already prepared in a different manner may be readily transformed into an elec-

50 trolyte in accordance with the present invention and this electrolyte be used for the separation of zinc. The process is therefore gen-

erally applicable.

Numerous investigations which I have made have demonstrated that the salts of boric 55 acid, and especially borax, (the sodium salt of the bibasic tetraboric acid,) when decomposed by hydrolyses in aqueous solution present special advantages over the free orthoboric acid, which advantages are utilized in 60 accordance with this invention to compose new baths for the electrolytic separation of zinc.

According to the purpose in view the proportions of the salts may be varied. Exam- 65 ples will now be given of compositions of baths which I have found practically successful. Besides the quantity of the substances used the degree of concentration, current intensity, and electrode area play an important 70 part. For instance, about seventeen kilograms of zinc sulfate (chemically pure and crystalized) are dissolved in water and one half of the solution is introduced into a warm solution of about nine kilograms of borax. 75 The precipitate thereby formed is dissolved in concentrated sulfuric acid and stirred until congo paper turns only perceptibly blue, when the solution is applied thereto. The other half of the solution of zinc sulfate is 80 then added. About one hundred and seventy liters of water is the most suitable proportion of the aforesaid weights of salts.

Another suitable weight proportion of the mixture of salts is, for instance, as follows: 85 About one hundred grams of borax and eighty grams of zinc sulfate treated as before with one liter of water; further, eighty grams of zinc sulfate, fifty grams of boric acid, thirty-eight grams of sodium sulfate or the like, and, fur- 90 ther, zinc sulfate about seventy-eight parts, by weight, (precipitated by soda-lye and the precipitate again dissolved with an acid,) and about fifty-seven and one-half parts, by weight, of boric acid each with one liter of water, so 95 that a highly-concentrated electrolyte is obtained.

The quantities of the substances used may be varied according to circumstances.

The composition of this kind of bath is 100 simple, cheap, and permits of high current densities—say from one to three amperes per

square decimeter—being used. Especially favorable results are obtained when the concentration of baths is 20° Baumé and over, which has not before been used. The evolu-5 tion of the gases is counteracted by the existence of an extra large amount of the metal ions. The high degree of concentration of the bath, which is in itself of great advantage, assists the action of the boron comro pounds added. The concentration is obtained by a correspondingly large addition of zinc salts.

The process when applied, for instance, to coating iron plates may be carried out as fol-15 lows: The iron plates are freed from scale in any suitable scouring-bath, preferably sulfuric acid. They are then thoroughly ruled, brushed mechanically with water, washed, and suspended in the bath at distances apart of 20 about ten centimeters. The bath need be neither heated nor agitated. The plates are removed from the bath after from a quarter of an hour to three hours or more, according to the thickness of the deposit required. The 25 metallic deposit is absolutely free from spongy portions and is regular and dense. After being brushed once this deposit acquires a luster between that of aluminium and that of silver.

Instead of zinc sulfate, zinc chlorid or other zinc salts may be used in combination with borax or boric acid, the process being carried out in the same manner as in the case of using zinc sulfate. To redissolve the pre-35 cipitates, other acids, such as hydrochloric acid, may be used. Even with the zinc-baths already known the addition of boric acid or boron compounds insures a favorable deposit of metal. The same applies to alkaline baths 40 and chiefly those that are highly concentrated. The presence of boric acid or boron compound, such as borax, is always one of the most essential factors for the separation of zinc. As further examples of combinations which have 45 withstood my tests in practice may be mentioned: the sole addition of boric acid or of borax to zine salts—such as zine chlorid, zine

of alkali chlorids, sulfates, and the like. The 50 disseminating power of ammonium salts, and especially of sal-ammoniac, may also be used in special cases in connection with these baths. Of course the various baths may be combined one with another, and my trials made there-55 with have produced very good results, so that the process according to this invention may be carried out with all the zinc-baths already known and conducting-salts (salts) be used in the most varied combinations with baths 60 to which boron compounds have been added.

sulfate, and the like—or the further addition

When dealing with a large number of articles, especial devices are required to obtain faultless zinc deposits. In the bath stationary sieves, baskets, or the like may be used 65 as cathode and as receptacles for articles treated in large numbers. Iron nails may, for instance, be placed on galleries arranged

in the bath, or, still better, the following device may be used: In a wooden bath a rotatable shaft is arranged, preferably horizon- 7c tal, and carries around it, somewhat after the fashion of paddle-wheels, baskets of thick iron or copper or brass, wire-netting, or of perforated metal, which baskets are filled with iron nails which have sufficient contact 75 and are scoured while the baskets revolve slowly.

If it be wished to galvanize in a bath in motion objects which are especially difficult to coat with zinc, the goods may be hung in 80. the bath while the anodes are arranged radially on a vertical or horizontal spindle rotated by any suitable means. The bath is thus vigorously stirred and the electric current increased and decreased intermittingly.

Figures 1 and 2 of the accompanying drawings represent in vertical sections two forms of apparatus suitable for carrying out the process as described. Fig. 3 shows the rotatable anode-plates in horizontal section on 90 the line 1 2 of Fig. 2.

1 is a shaft of wood, on both ends of which are fixed arms 2, serving as carriers of four trough-shaped baskets 3, extending over the whole length of the bath and being provided 95 with a cover 4. Into these baskets there are introduced nails and similar objects to be covered with zinc and serving as cathode. From the drawings it may be seen that the bottom as well as all the walls and the cover of the roo baskets are perforated and provided at their inner surfaces with ribs 5. The whole device is rotated in the bath by means of a drivinggear or any other means provided for that purpose at one of the ends of shaft 1.

6 is the anode.

An insulated wire connects the baskets 3 conductively with the shaft, the latter being surrounded by a metallic shell and is supplied with the electric current by any of the 110 well-known means.

In consequence of the walls of the baskets 3 being perforated the electrolyte is permanently in contact with the objects to be galvanized. The ribs 5 establish the metallic 115 contact of the objects with the walls of the baskets.

If the device is being rotated, and thus acts simultaneously as an agitator or stirring device, the objects contained in the troughs 120 are continuously turned over and over, whereby they are brought into intimate contact with the electrolyte and are exposed to a varying action of the current.

Fig. 2 shows an arrangement of the appa-125 ratus for galvanizing sheet-iron. 7 indicates the sheet-irons to be covered with zinc by galvanization The anodes 8 may be rotated on a vertical shaft. In view of the rotation of the anodes the electrolyte is being main- 130 tained in a continuous movement and further on the anodes are placed intermittently opposite the cathodes 7, with their surface and with their edges only in consequence, whereof

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the cathodes are intermittently acted upon by an increasing and decreasing electric current without any necessity of moving the articles to be treated. It is obvious that the 5 shaft of the anodes may be disposed inclined or horizontally.

Having fully described my invention, what I desire to claim and secure by Letters Pat-

ent is—

1. The electrolytic process herein described of depositing zinc by electrolyzing in a bath containing zinc salts, conductive salts and boron compounds, substantially as described.

2. The electrolytic process described of de-15 positing zinc by electrolyzing in a bath con-

taining metallic salts, conductive salts and boron compounds, said bath being concentrated by the addition of zinc salts.

3. The electric process herein described of depositing zinc by electrolyzing in a bath con- 20 taining zinc salt and a boron compound, substantially as described.

In witness whereof I have hereunto signed my name this 21st day of February, 1902, in the presence of two subscribing witnesses.

HEINRICH PAWECK.

Witnesses:

JOHANN LUSE, ALVESTO S. HOGUE.