

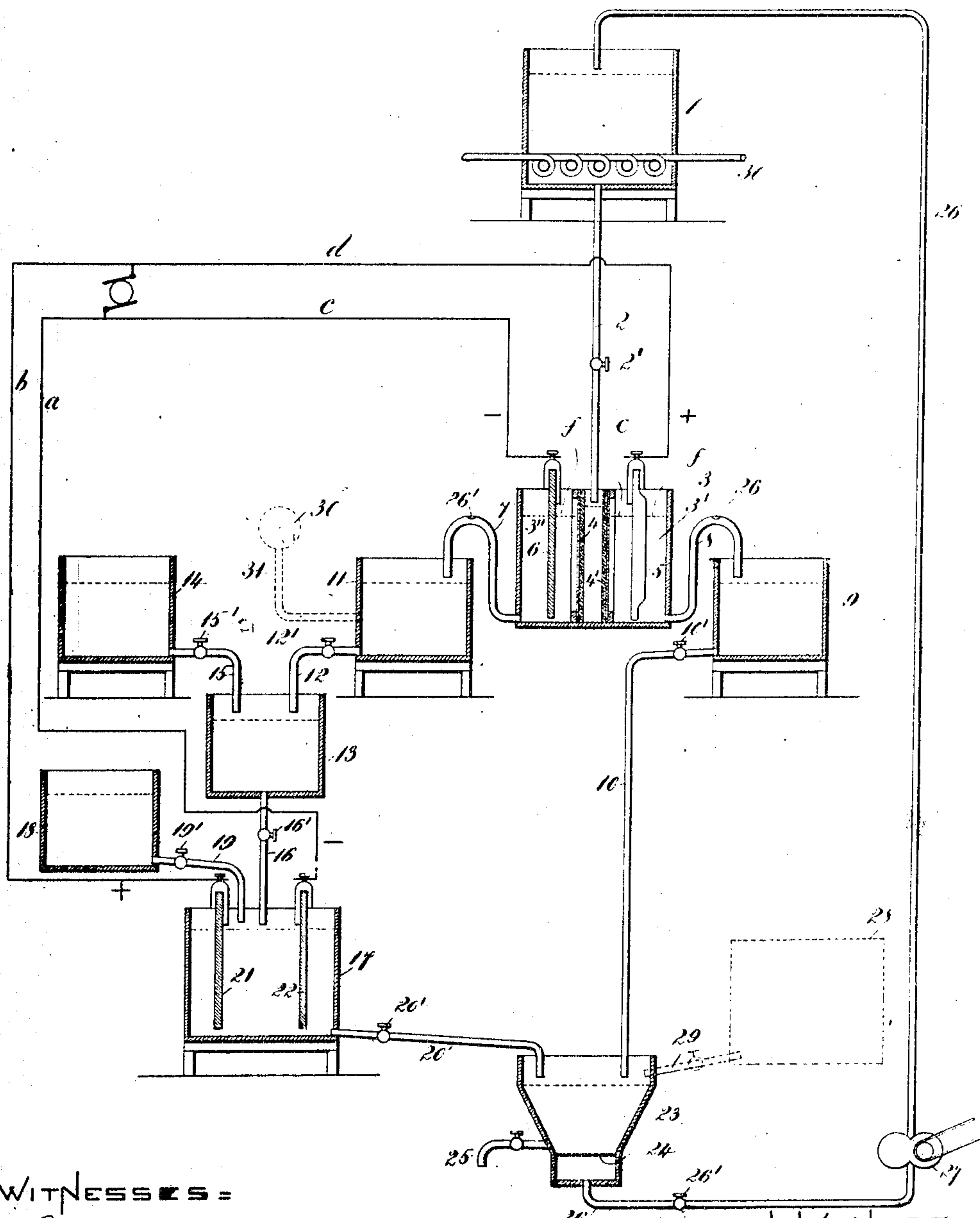
No. 871,162.

PATENTED NOV. 19, 1907.

E. D. CHAPLIN.

ART OF PRODUCING PIGMENTS BY ELECTROLYSIS.

APPLICATION FILED FEB. 3, 1906.



WITNESSES =  
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*his attorneys.*

# UNITED STATES PATENT OFFICE.

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## ART OF PRODUCING PIGMENTS BY ELECTROLYSIS.

No. 871,162.

Specification of Letters Patent.

Patented Nov. 19, 1907.

Application filed February 3, 1906. Serial No. 299,238.

*To all whom it may concern:*

Be it known that I, EDWIN D. CHAPLIN, a citizen of the United States, and a resident of Boston, in the county of Suffolk and State of Massachusetts, have invented a new and useful Improvement in the Art of Producing Pigments by Electrolysis, of which the following is a specification.

My invention relates to the art of producing pigments by electrolysis, and it relates more particularly to a process for so producing lead chromate.

In the manufacture of lead chromate by electrolysis, I prefer to employ an electrolyte which under the influence of the electric current will separate into a solvent of lead and an alkaline hydrate and thereby to produce in the anode compartment containing metallic lead, which may be the anode of the electrolyzer, a solution of a soluble salt of lead, and in the cathode compartment, an alkaline hydrate. I have found it essential to the commercial production of lead chromate to maintain the lead solution and the alkaline hydrate separate and not to produce any lead precipitate in the electrolyzer itself. I have also found it essential to employ some means, such as an oxidizing agent, to prevent the formation of insoluble salts of lead in the electrolyzer.

As clearly set out in Letters Patent No. 555,232 and 563,553, granted to A. B. Browne and myself, and in Letters Patent No. 675,555 granted to H. G. Halloran and myself, for processes of producing lead carbonate by electrolysis, certain insoluble basic salts of lead are formed in the electrolyzer, thereby rendering the processes uncommercial, unless certain precautions are taken in order to prevent such formation.

The object of the present invention is to provide a process of producing lead chromate by electrolysis in which the formation of insoluble basic salts of lead in the electrolyzer may be prevented by any means suitable for the purpose. While the formation of the aforesaid basic lead salts may be prevented in a variety of ways, I prefer in the process herein set forth, to render such formation impossible by electrolytically producing a soluble acid salt of lead obtained, preferably, by electrolytically dissolving metallic lead in the presence of a soluble salt capable of preventing the formation of in-

soluble salts of lead in the electrolyzer,—for example, a soluble chlorid salt or an oxidizing agent such as a soluble chlorate of an alkaline base.

In Letters Patent No. 538,998, granted to A. B. Browne and myself is described a process of producing lead chromate by electrolysis, to which reference may be had for a more complete disclosure of the principles involved in the present application than is necessary to set forth herein.

My invention may best be understood by having reference to the drawing which accompanies and forms a part of this specification, and which illustrates in diagrammatic form an arrangement of apparatus and circuits whereby my process may be carried into effect; but it is to be understood that my invention is broader than mere apparatus and may be carried into effect by a great variety of apparatus and circuit arrangements.

In the drawing the figure represents an apparatus and system of circuits for electrolytically producing lead chromate.

In the figure, 1 represents a supply tank, containing an electrolyte, which under the influence of the electric current, will be separated into an alkaline hydrate and a solvent of lead, such for example as an aqueous solution of a non-alkaline salt, preferably sodium nitrate, and containing also an aqueous solution of a soluble chlorid or chlorate salt, which may be a chlorid or a chlorate of an alkaline base, preferably sodium chlorid or chlorate. I shall hereinafter refer to the solution in tank 1 as the "neutral solution" and shall assume that it consists of sodium nitrate and sodium chlorid.

3 represents an electrolyzer divided into an anode compartment 3' and a cathode compartment 3'', by means of the foraminous diaphragms 4 4', between which is formed a compartment for receiving the electrolyte from the tank 1 through the pipe 2, which is provided with a valve 2'. For the purpose of preventing a mixture of the anolyte and catholyte in the electrolyzer, I may maintain a preponderance of pressure on the inner sides of said diaphragms, preferably by maintaining the level of the electrolyte between the diaphragms higher than the level of the anolyte and catholyte. The level of the electrolyte in the compartment between the diaphragms is indicated by the dotted



line *e*, and the level of the anolyte and catholyte, which is governed by the height of the bends of the outlet pipes 7 and 8, is indicated by the dotted lines *ff*. In order to prevent said outlet pipes from siphoning the liquids out of the electrolyzer, the upper portion of each of said bends may be provided with an opening 26, 26' to equalize the atmospheric pressure on the columns of liquid in the two portions of each of said pipes.

The lead to be operated upon is placed in the anode compartment and may form the anode of the electrolyzer. In the present case the lead is shown in the form of a lead pig 5, connected by the conductor *d* to the positive pole of the dynamo D, or other suitable source of current. The cathode 6 of the electrolyzer may consist of copper or any other suitable metal, and is connected by the conductor *c* to the negative pole of the dynamo. When the electrolyzer is charged with the neutral solution and the electric current passed therethrough, a soluble acid salt of lead is formed in the anode compartment 3' and an alkaline hydrate, in the present case assumed above, sodium hydrate, is formed in the cathode compartment 3''. Presumably lead nitrate is at first formed in the anode compartment and this, by the action of the sodium chlorid and under the influence of the electric current, is converted into lead chlorid, which is soluble at the temperature at which the electrolyte is maintained by the steam coil 30, or by any other suitable means, and, finally by the continued action of the current, the lead chlorid is converted into lead oxychlorid, which is soluble at ordinary temperatures. The lead oxychlorid being an acid salt of lead, renders impossible the formation of insoluble basic salts of lead. The lead oxychlorid or soluble acid salt of lead so formed in the anode compartment 3' is drawn off by the pipe 8 into the settling tank 9 and the alkaline hydrate formed in the cathode compartment 3'' is drawn off by the pipe 7 into the settling tank 11. By means of the pipe 10, provided with a valve 10' the acid salt of lead is discharged from the tank 9 into the precipitating tank 23, or as is obvious, it may be discharged into said precipitating tank 23 directly from the anode compartment 3'.

In the precipitating tank 23 the acid salt of lead may be converted into lead chromate by treating it with any suitable chromate or bichromate, or mixture of the two, such for example, as a mixture of sodium or potassium chromate and bichromate, an aqueous solution of which may be contained in the tank 28 and discharged into the precipitating tank by the pipe 29. The resulting lead chromate settles at the bottom of the tank 23 upon the filter 24, while the filtrate, which is substantially the original neutral solution,

is pumped back to the supply tank 1 by the pump 27 through the pipe 26 provided with the valve 26'. The lead chromate may be withdrawn by the pipe 25, washed, and dried for use. The alkaline hydrate which in the present case is assumed to be sodium hydrate, may be carbonated by passing carbon-dioxid therethrough from the generator 30, connected with the tank 11 by means of the pipe 31, in order to produce salsoda which is a marketable product or else the said hydrate may be utilized in the production of the aforesaid chromate and bichromate mixture. In order to utilize said alkaline hydrate in the production of said chromate and bichromate mixture, I may discharge said hydrate herein assumed to be sodium hydrate through the pipe 12, provided with a valve 12', into the tank 13 and mix therewith a solution of chrome alum contained in the tank 14 and drawn off into the tank 13 by the pipe 15 provided with a valve 15'. By producing an excess of sodium hydrate, I precipitate and redissolve chrome hydrate in the tank 13 and convey such solution of chrome hydrate into the electrolyzer 17 through the pipe 16 provided with the valve 16'. A solution of sodium chlorid is conveyed from the tank 18 by the pipe 19, provided with the valve 19', into the electrolyzer 17, and under the influence of the electric current from the dynamo D, the poles of which are connected by the wires *a* and *b*, respectively, to the electrodes 21 and 22, of the cell 17, a suitable mixture of sodium chromate and bichromate is produced from the mixture of chrome hydrate and sodium chlorid. This mixture is then conveyed from the cell 17 by the pipe 20, provided with the valve 20', into the tank 23, where it precipitates lead chromate from the solution of the acid salt of lead discharged into said tank by the pipe 10.

I claim:

1. As an improvement in the art of producing lead chromate by electrolysis, the process herein described which consists in electrolytically producing from metallic lead a soluble acid salt of lead and converting said soluble acid salt of lead into lead chromate.

2. As an improvement in the art of producing lead chromate by electrolysis, the process herein described which consists in electrolytically dissolving metallic lead in the presence of a soluble chlorid salt, converting the resulting lead chlorid into lead oxychlorid, and producing lead chromate from said lead oxychlorid.

3. As an improvement in the art of producing lead chromate by electrolysis, the process herein described which consists in electrolytically dissolving metallic lead in the presence of a soluble chlorid of an alkaline base, thereby producing an alkaline hy-



drate and lead chlorid, converting said lead  
chlorid into lead oxychlorid, withdrawing  
said alkaline hydrate and lead oxychlorid  
into separate receptacles, electrolytically pro-  
5 ducing a mixture of chromate and bichro-  
mate of an alkaline base from said alkaline  
hydrate, and treating said lead oxychlorid  
with said mixture to produce lead chromate.

In testimony whereof, I have hereunto  
subscribed my name this fifth day of Jan. 10  
1906.

EDWIN D. CHAPLIN.

Witnesses:

CHARLES C. KURTZ,  
GEO. K. WOODWORTH.