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METHOD FOR DETERMINING THE COATING WEIGHT OF TIN ON TIN PLATES

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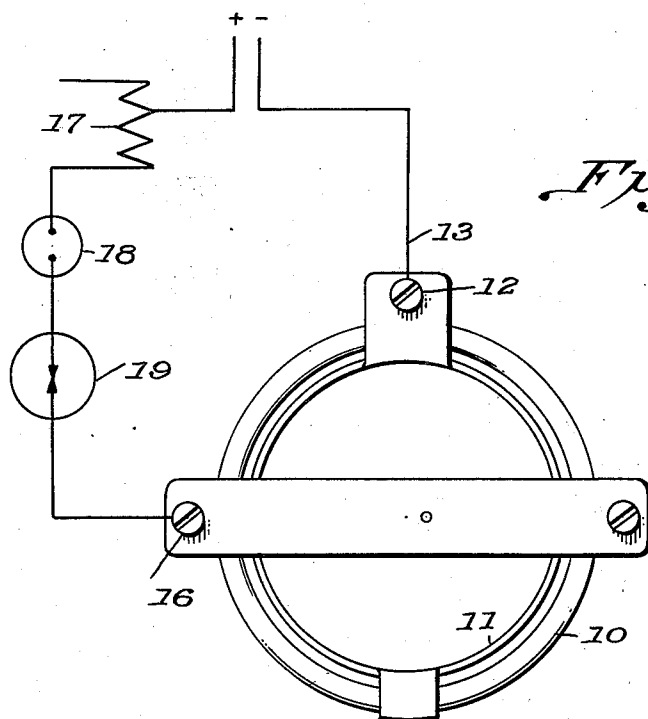


Fig. 1.

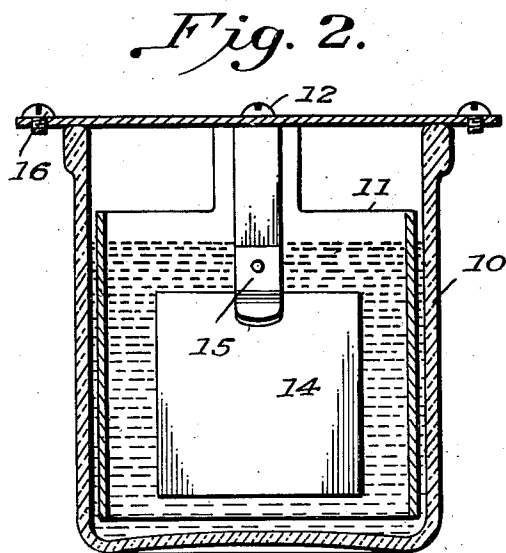


Fig. 2.

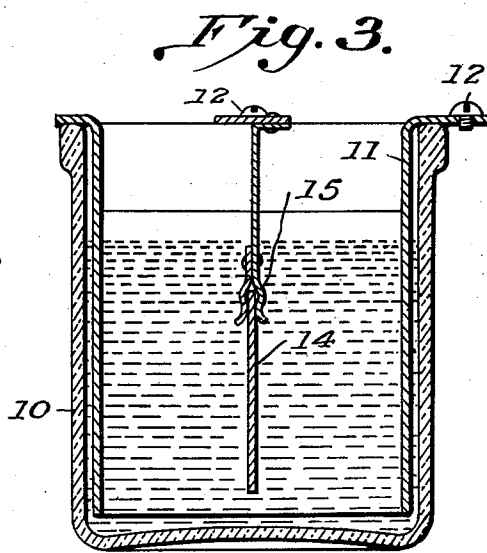


Fig. 3.

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METHOD FOR DETERMINING THE COATING
WEIGHT OF TIN ON TIN PLATES

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2 Claims. (Cl. 204—1)

The present invention relates generally to the method for the determination of thickness or weight of certain metal coatings and is more particularly directed to the determination of the coating of tin on tin plate.

It is the general object of the present invention to provide a method which will facilitate the performance of such determinations and will yield results which are accurate for practical purposes.

By the present method the tin coating is removed from the sample electrolytically in the presence of a known quantity of an oxidizing agent. The excess of the oxidizing agent present in the electrolyte is then determined and the weight or thickness of the tin coating is then calculated from the data obtained.

While the method described herein may be applied to many types of metal coatings, it will be described herein specifically for the determination of the coatings of tin on tin plate. The reagents employed in the present process may also be varied as is well understood by persons skilled in the art.

In the present method a sample of tin plate of standard dimensions is suspended in the electrolyte of an electrolytic cell, the cell being connected so that the sample constitutes the anode of the cell, carbon, preferably in cylindrical form, being employed for the cathode. A constant current is permitted to flow through the cell for a predetermined period of time, the current and time being so determined that substantially all of the tin will be removed from the tin plate. It is important that in the conduct of this process the current should not be permitted to pass through the cell after substantially all of the tin has been removed, for the reason that such continued flow of current through the cell may produce reactions between the ingredients in the electrolyte which may interfere with the results of the determination.

The electrolyte preferably contains hydrochloric acid, potassium iodate, and potassium iodide, all in aqueous solution. The passage of electricity through this cell results in the dissolution of the tin, which in the absence of the oxidizing agent present would be converted into the salt of the lowest valence and result in the formation of stannous chloride. In the present case, however, the oxidizing agent present causes the oxidation of the bivalent tin to its tetravalent form, resulting in the formation of stannic chloride. The oxidation in the present case is the result of the action of the iodine which is liberated by the

reaction between potassium iodate and potassium iodide in acid solution.

In the conduct of the present process an excess of the oxidizing agent or of the reagents which produce the oxidizing agent should be had. When the detinning is completed, the flow of current through the cell is interrupted, and the excess of iodine or potassium iodate still remaining is determined by titrating the electrolyte with sodium thiosulphate in the presence of starch as an indicator.

Knowing the amount of potassium iodate originally introduced into the cell and knowing the amount of potassium iodate which remained in the cell after the detinning was completed, the amount of potassium iodate consumed in reaction with the dissolved tin is a measure of the tin removed from the sample, and thus is a measure of the thickness or the weight of the tin coating.

It will now be understood that while in the preferred form of the present invention the tin is removed electrolytically in the presence of an excessive amount of an oxidizing agent, instead of an oxidizing agent, other agents may be employed, provided they be such as will react quantitatively with the dissolved tin. In the preferred method as described herein the conduct of the determination is greatly facilitated for the reason that the electrolytic solution can be titrated immediately after the completion of the detinning step without any intermediate treatment of the solution.

The chemical reactions upon which the present process is based are as follows:

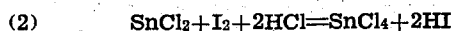
In the aqueous solution of acid, iodate, and iodide, the following reaction takes place:



The solution employed should contain a known quantity of potassium iodate and an excess of potassium iodide and acid.

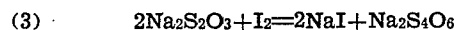
Instead of the potassium iodate, other agents for liberating iodine may be employed, such as potassium biiodate, potassium permanganate, or potassium dichromate.

When the tin goes into solution by electrolysis SnCl_2 is originally formed and then is acted upon by the iodine as follows:



Equations (1) and (2) thus show that the amount of iodine liberated is determined by the amount of potassium iodate or other such iodine liberating oxidizing agent employed. The free

iodine oxidizes the tin. The excess iodine is determined by titration with sodium thiosulphate in accordance with the following equation:



Thus, the amount of tin which was placed in solution by electrolysis is determined by the aid of the above equations.

The drawing accompanying the present application illustrates an apparatus that may be employed in the conduct of the present process.

In the drawing:

Fig. 1 is a plan view of the electrolytic cell employed and also contains a diagrammatic showing of the electrical wiring system employed;

Fig. 2 is a front partly sectional view of the apparatus; and

Fig. 3 is a side partly sectional view of the apparatus.

Referring to the drawing, 10 designates a glass container, the glass container being employed for the reason that the titration of the excess potassium iodate may, if desired, be conducted in this container, taking care, however, that the solution adhering to the anode and cathode should be washed into the container. The carbon cathode 11, preferably in the form of a hollow cylinder, is placed in the container and is connected to the source of direct current by means of the cathode connector 12 and wire 13. The sample of tin plate 14 is suspended within the cathode by means of the anode clamp 15 and the anode connector 16 which rests on top of the container 10. Interposed between the anode and the positive side of the source of current is the rheostat 17, the ammeter 18, and the automatic time switch 19.

In the conduct of the present process the electrolyte consisting of a known amount of standard potassium iodate solution together with an excess of potassium iodide and of hydrochloric acid, and enough distilled water to cover the anode, is placed in the glass container. A constant electric current is passed through the cell for a definite period of time, the automatic time switch controlling this time interval.

For removing the coating from a sample two inches square and having a coating weight of 1½ lbs. per base box, a current of 1 ampere for a period of three minutes is employed. After the lapse of three minutes, at which time the automatic switch will open the circuit, the sample is removed from the electrolyte and washed, the wash water being added to the electrolyte. Similarly, the cathode is removed and washed, the wash water being added to the electrolyte. The excess potassium iodate or its equivalent, the excess iodine liberated by the potassium iodate, is now determined by titration with standard sodium thiosulphate solution, starch being used as an indicator.

In the calculation of the weight of the tin coating allowance is made for a certain constant which is characteristic of the cell as operated. This cell constant is obtained by comparing the results obtained by a chemical analysis of a specimen of tin plate with the results given by the present process on a specimen of the same tin plate.

It is found that in the deposition of tin during the manufacture of tin plate some iron-tin alloy is formed which does not yield up its tin content by the present process. There is also a small amount of tin deposited in metallic form on the cathode. The electrolysis also has some effect

upon the chemical reagents. It is found that in the present process, where the current is constant and the time is constant, these factors stay constant. Together these are here termed a cell constant which is determined originally by comparing the results obtained by a chemical analysis of a specimen of tin plate with the results given by the present process of a specimen of the same tin plate. In carrying out my present process I employ for my cell a glass beaker within which are mounted two carbon electrodes three inches by one inch by ⅜ inch with faces parallel and 1½ inches apart. The specimen two inches square is disposed between the electrodes. The electrolyte consists of 200 cubic centimeters of hydrochloric acid (1 part concentrated HCl and 20 parts water) and 15 cubic centimeters of standard potassium iodate solution equivalent to exactly .007 gram of tin per cubic centimeter. The potassium iodate solution contains dissolved therein .04 gram of potassium iodide per cubic centimeter. A current of 1 ampere is passed through the cell for a period of three minutes, sufficient to remove the tin from a specimen having a coating weight of 1½ lbs. per base box, typical of commercial tin. The amount of tin is then determined by the amount of potassium iodate used from the electrolyte. It has been found by a comparison of over a thousand tests made simultaneously by the electrolytic method using the cell described above and a standard chemical method, that the electrolytic method gives results which show the tin content to be .007 gram higher than that shown by the standard chemical method. Therefore the cell constant for the above described cell is minus .007 gram of tin, which constant remains the same under the above mentioned conditions of time, current, concentration of electrolyte and cell construction.

While the process is described herein as specifically applied to tin coatings, the process is also applicable to other metal coatings, as chromium, nickel, zinc, and the like, provided that the reagents employed are such that they will react quantitatively with the dissolved metal.

In lieu of the oxidizing agents that have been set forth in earlier portions of the present specification, other reagents may be employed which have the quality of reacting quantitatively with the dissolved tin or other metal. Among such other reagents may be soluble sulphides, hydrogen sulphide in aqueous solution, and oxalates.

Other iodine solutions than the type described hereinabove may be employed, such as iodine in excess potassium iodide; chlorates, and bromates may be used for the liberation of iodine.

Instead of mineral acid in the electrolyte, an organic acid such as acetic acid may be employed.

Having thus described my invention, I claim:

1. The method of rapidly and accurately determining the weight of tin coatings on a plurality of specimens of tin plate or the like, which comprises first determining the correction factor of an electrolytic cell in which the electrolyte contains hydrochloric acid, potassium iodide and an iodine liberating agent of the group consisting of potassium iodate, potassium permanganate, and potassium dichromate and having a tin plate anode, which factor is derived from a comparison of the results obtained by chemical analysis with the results obtained by passing a predetermined constant electric current through said cell for a predetermined time, and thereafter subjecting each specimen to electrolysis for the same period of

time and under the same constant current conditions in the same cell thereby dissolving substantially all the tin from the specimen, determining the excess of iodine and correcting the results thus obtained by said correction factor.

2. The method of rapidly and accurately determining the weight of tin coatings on a plurality of specimens of tin plate or the like, which comprises first determining the correction factor of an electrolytic cell in which the electrolyte contains hydrochloric acid, potassium iodide and an iodine liberating agent of the group consisting of potassium iodate, potassium permanganate, and potassium dichromate and having a tin plate

anode, which factor is derived from a comparison of the results obtained by chemical analysis with the results obtained by passing a predetermined constant electric current through said cell for a predetermined time, and thereafter subjecting each specimen to electrolysis for the same period of time and under the same constant current conditions in the same cell thereby dissolving substantially all the tin from the specimen, determining the excess of iodine by titration with sodium thiosulphate and correcting the results thus obtained by said correction factor.

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