

UNITED STATES PATENT OFFICE.

JOSEF WEBER, OF ESSEN-ON-THE-RUHR, GERMANY.

PROCESS OF REGENERATING STANNIC-CHLORID SOLUTIONS.

958,986.

Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that I, JOSEF WEBER, a subject of the King of Prussia, German Emperor, and resident of Essen-on-the-Ruhr, in the Province of the Rhine, German Empire, have invented a new and useful Process of Regenerating Stannic-Chlorid Solutions, of which the following is a specification.

This invention relates to a continuous process of regenerating stannic chlorid solutions, and the main object of the invention is to restore to a working condition an aqueous solution of stannic chlorid that has been used as a bath for a considerable period of time in silk dyeing and has become so loaded with impurities, especially lime and lead, as to be unfit for further use while in that condition.

In the art of silk dyeing as practiced for many years, it has been customary to employ baths containing solutions of stannic chlorid of from 20° to 30° Bé. for the purpose of weighting the silk. After the baths had been used for a considerable period of time and had become weakened by the passage through them of a predetermined quantity of silk it was formerly the practice to restore them to working condition by adding a concentrated solution of stannic chlorid of 50° to 60° Bé. This method of concentrating a weakened solution of stannic chlorid has, however, been superseded by the use of anhydrous tetrachlorid of tin. It is now the practice to transfer a part of the weakened solution from the bath to another vessel, for example, an enameled receptacle, and concentrate it, while in such vessel, up to 50° to 60° Bé. by adding a suitable quantity of anhydrous tetrachlorid of tin. I have frequently observed that during the concentration of such a weakened solution by the addition of the anhydrous tetrachlorid a precipitate is formed. Numerous analyses have shown that this precipitate consists of chlorid of lead, sulfate of lead and calcium sulfate, that is, gypsum.

The lime present in the weakened bath comes chiefly from the water that is drawn into the bath by the silk, while the lead is dissolved from the walls of the apparatus containing the stannic chlorid bath used in the process. The lead is present in considerable quantities after the bath has been used for a long period of time, as it has been found that lead is quite soluble in dilute solutions of stannic chlorid, although it is absolutely in-

soluble in a concentrated solution of stannic chlorid. The silk treated by such a weakened solution of stannic chlorid contaminated with considerable quantities of lime is very injuriously affected by both of these impurities, and especially by the lead, which destroys or seriously impairs the luster and "touch" of the silk. Because of this it has been customary to renew the baths that become weakened by long use and to recover the tin content from the solution that is removed from the apparatus. This recovery of the tin has been effected by transforming the stannic chlorid of the weakened bath into oxid of tin; but the process involves a large financial loss, the main elements of which are the cost of converting the tin from one form into another and the reduced value of the oxid of tin in this form as compared with that of stannic chlorid and of tin itself. I have found that all of this loss that has resulted from the removal of weakened solutions of stannic chlorid from the baths and their transformation into oxid of tin can be entirely avoided, and that the weakened stannic chlorid solution can be concentrated and used in a continuous silk treating process without converting it into any other form of tin. In order to accomplish this result it is necessary first to determine quantitatively the principal impurities in the weakened stannic chlorid solution, and then to precipitate them out by means of a suitable reagent, after which the solution from which such impurities have been eliminated may be concentrated and used again.

The amount of lime in the weakened stannic chlorid solution is, according to my method, first determined quantitatively by analysis of the solution. As a result of numerous experiments I have found that by adding to the weakened stannic chlorid solution of the bath an amount of sulfuric acid calculated according to the amount of lime present in the bath, all the lime will be precipitated in the form of calcium sulfate, together with the lead. By the addition of the exact amount of sulfuric acid necessary to precipitate all the lime in the bath the chief impurities in the weakened stannic chlorid solution are eliminated, and the weakened solution after settling and filtration is then in condition for concentration. The concentration of the weakened solution is then effected in the manner be-

fore stated by the addition of anhydrous tetrachlorid of tin, up to about 60° Bé. By this process the tin content of the bath is maintained at all times in the form of stannic chlorid, the impurities removed by precipitation after determination of the quantities present, and the bath restored to working condition by the mere addition of the proper amount of the anhydrous tetrachlorid, thus making it possible in a continuous process to utilize all the tin in the baths for weighting the silk.

What I claim is:

1. The process of purifying an aqueous solution of stannic chlorid that has been contaminated with lime by use in silk dyeing, which consists in adding to said aqueous solution a sufficient amount of sulfuric acid to precipitate all the lime content.

2. The process of purifying an aqueous

solution of stannic chlorid that has been contaminated with lime and lead by use in silk dyeing, which consists in adding to said aqueous solution a sufficient amount of sulfuric acid to precipitate all the lime and lead contents.

3. The process of purifying and concentrating an aqueous solution of stannic chlorid that has been contaminated with lime and lead by use in silk dyeing, which consists in first adding to said aqueous solution a sufficient amount of sulfuric acid to precipitate all the lime and lead content, and afterward suitably concentrating said purified solution by adding anhydrous tetrachlorid of tin.

JOSEF WEBER. [L. S.]

Witnesses:

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