

UNITED STATES PATENT OFFICE.

HERMANN LEY, OF ELBERFELD, GERMANY.

PROCESS OF PURIFYING SODIUM-PHOSPHATE WASTE LIQUORS.

1,049,740.

Specification of Letters Patent.

Patented Jan. 7, 1913.

No Drawing.

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To all whom it may concern:

Be it known that I, HERMANN LEY, a subject of the King of Prussia, German Emperor, and a resident of Elberfeld, German Empire, have invented certain new and useful Improvements in Processes of Purifying Sodium-Phosphate Waste Liquors, of which the following is an exact specification.

In the art of silk weighting, it is well known that after the baths of sodium phosphate have been used several times for the purpose of weighting silk they should no longer be employed, for the reason that serious deterioration, and especially putrefaction, of the silk would result. It has been found that such deterioration results from the fact that in the course of time the baths of sodium phosphate employed in silk-weighting will take up compounds of tin which accumulate in these baths. When the percentage of tin contained in a bath exceeded a certain limit it was necessary to abandon the bath and substitute a new one, as there existed no useful process for separating these injurious compounds of tin from a bath of sodium phosphate and restoring the bath to a condition in which it was fit for use again for the purpose of weighting silk. The separation of the compounds of tin from such a bath is attended with considerable difficulties, as the precipitating agents generally used for precipitating compounds of tin either cause no separation at all or else an utterly incomplete separation of the compounds of tin—and this is true both in the case of chemical action, as by means of caustic alkali, sulfureted hydrogen, etc., and in the case of electrolytic action.

Now I have found that tin separates quickly and quantitatively from such a bath if first there be added to the bath small quantities of the salts, soluble in water, of such metals as have phosphates insoluble in water, and the bath then be suitably heated. The soluble salts of calcium, barium, strontium, magnesium, zinc and aluminum may be used for this purpose. Of the soluble salts tested by me up to the present time I have found that the following give a greater or less precipitation of the tin, according to the quantities added: Calcium—oxid, chlorid, nitrate and acetate. Barium—oxid, chlorid and nitrate. Strontium—oxid, chlorid and nitrate. Magnesium—oxid, chlorid, nitrate, sulfate, acetate, and mixture of magnesia.

Zinc—chlorid, nitrate, sulfate, acetate. Aluminum—chlorid, sulfate, acetate, acetotartrate. By adding the proper amount of any such salt to what would ordinarily be the waste liquor of the bath a precipitate will be formed containing all of the tin present in the bath. By filtration a perfect separation of the precipitated compounds of tin may be effected and the bath restored to a condition in which it is fit for use again for weighting silk.

In order that my process may be clearly understood I give the following example illustrating one mode of practising it: 4 kilos. of magnesium sulfate dissolved in a small quantity of water are added to 1,000 liters of a bath of sodium phosphate containing 2 g. of tin in each liter of solution. These baths, as used in silk-weighting, on an average contain from 160 to 200 g. of sodium phosphate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) in one liter. The highest possible concentration, according to my experience, would be from 250 g. to 270 g. of sodium phosphate in one liter. After the magnesium sulfate has been added to the bath the whole is carefully stirred. The bath, which has become slightly turbid, is then heated up to the boiling point and after a few minutes is left to cool, after which it is forced through a filter-press. The solution obtained is entirely free from tin and may be used again for silk-weighting purposes. The precipitate contains about 30 per cent. of tin, which may be recovered by treating the precipitate in any of the well-known ways.

The great importance of this process in the silk-weighting industry will be readily understood when it is considered that it has been necessary heretofore not only to abandon the baths themselves after they had accumulated a certain percentage of tin, but also to lose a large part of the sodium phosphate taken up by the silk in passing through the baths. After treatment this silk is usually wrung out or subjected to centrifugal action for removing the surplus sodium phosphate. It has been found by experiments that when the silk is subjected to centrifugal action after having passed through four baths, and been normally weighted, about 50 per cent. of the sodium phosphate taken up in the baths by the silk will be removed. This considerable amount of sodium phosphate, which has been a total

loss heretofore, may be recovered at very small expense by the present process.

What I claim is:

1. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a soluble salt of a metal the phosphate of which is insoluble in water.
2. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a plurality of soluble salts of metals the phosphates of which are insoluble in water.
3. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a salt, soluble in water, of a metal the phosphate of which is insoluble in water, and heating the liquor.
4. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a soluble salt of a metal the phosphate of which is insoluble in water, and heating the liquor up to the boiling point.
5. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with magnesium sulfate.

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6. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a small quantity of a solution of magnesium sulfate, and then heating the liquor.

7. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a small quantity of a solution of magnesium sulfate, and then heating the liquor up to the boiling point.

8. A process of purifying sodium phosphate waste liquors, which consists in treating such a liquor with a salt, soluble in water, of a metal the phosphate of which is insoluble in water, heating the liquor to the boiling point, and after cooling separating the precipitate.

In witness whereof I have hereunto set my hand in the presence of two witnesses.

HERMANN LEY. [L.S.]

Witnesses:

OTTO KÖNIG,

WALTER VONNEGUT.