REGENERATION OF LEAD OXIDE FROM THE WASTE PRODUCTS OF THE BATUMSKII CAFFEINE FACTORY

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In the production of a series of pharmaceutical chemistry compounds from vegetable raw materials, a solution of basic lead acetate [1, 2] is used for the precipitation of tannins and other inert compounds.

At the Batumskii Caffeine Factory, in the stage of purification of the first aqueous caffeine solutions, the consumption of the reagent indicated above reaches 120 t/day. Acetate and phosphate precipitates, containing up to 30.6-33.4% of lead (upon recalculation to lead oxide 33-36%) are formed in the process of this purification. Based on literature data on hydrometallurgical recovery of ore [3, 4], we studied the possibility of regenerating lead from the indicated precipitates by the following scheme: combustion of the precipitate – solution of it in nitric acid – precipitation with ammonium in the form of the hydroxide – calcining the latter.

The results of consolidated laboratory experiments are presented in this work. In all, up to 40 kg of a mixture of acetate and phosphate precipitates were processed.

EXPERIMENTAL

In the first stage of the technological process, making use of the presence of carbon in the precipitates, their autocombustion was carried out. In the combustion of 10 kg of precipitates, 4.35-4.4 kg of residue remains, which contains 64.9-70.5% of lead (recalculated to lead oxide 70-76%). The residue in amounts of 2 kg was treated with 12 l of 16% nitric acid and the reaction mixture was stirred 1-1.5 h at 80-90°C to complete solution. The obtained solution of lead nitrate was filtered and evaporated to a density d = 1.324-1.34. After 24 h (10-12°), the precipitated crystals of lead nitrate were separated on a suction filter, and the filtrate was again evaporated to formation of a crystalline film on the surface of the solution; the mixture was cooled and the crystals of lead nitrate were separated.

The obtained lead nitrate requires a second purification as the precipitates after combustion contain admixtures of silicon, iron, aluminum, and other elements, which upon further treatment, pass into solution. Therefore, the pressed-out precipitate of lead nitrate is again dissolved with stirring in freshly boiled water at 80-90° in a weight ratio of 1:2.2. In this case, a solution of lead nitrate and 180-220 g of a residue, which is insoluble in water and contains lead, which on an average reaches 73.05%, are formed. After separation of the precipitate, the solution of lead nitrate is subjected to precipitation.

As was shown by preliminarily determined potentiometric titration curves of standard solutions of lead nitrate, of suitable concentration, prepared by us, the upper limit of pH for the precipitation of the hydroxide is found in the region 7.8-8.0. In connection with this, to the obtained solution of lead nitrate was added, with intense mixing, a 25% solution of ammonia to pH 8.0. The precipitated lead hydroxide was washed to a negative reaction for NO_3^- ion, pressed out, and placed into a muffle furnace where it was converted to the oxide at 580-600°. From separate treatments of the lead nitrate, separated from the main solution and the first mother liquor, were obtained 2 fractions of technical material with a lead oxide content of 95.5-96.7 and 90.5-93.6%, respectively. Therefore, in the subsequent experiments, a common treatment of both fractions of crystals was carried out, during which a technical material of lead oxide was obtained having an average content of PbO of about 93-95%, in a yield of 73-76% of the original amount of lead oxide in the residue after baking.

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After obtaining the principle amount of lead oxide, there remain residues insoluble in water with high contents of lead (see above). They were treated analogously to give on an average 6.61% more of the initial amount of lead oxide. In this way, the yield of technical lead oxide reaches 82.85%.

A solution of basic lead acetate [5] suitable for the precipitation of inert compounds in phytochemical production is prepared from the obtained technical lead oxide.

To obtain lead oxide which satisfies the requirements of the GF SSSR IX publication, it is necessary to repeat the purification of the technical product. In precipitation of the technical lead oxide from nitric acid, a pharmacopoeic preparation of 99.3-99.8% purity was obtained. In the regeneration of lead oxide, ammonium nitrate is formed as a by-product.

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