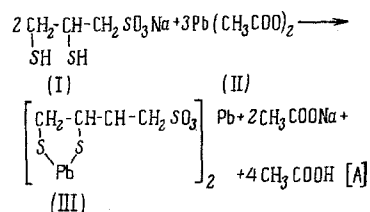


# UTILIZATION OF LEAD CHLORIDE - A BY-PRODUCT OF UNITHIOL PRODUCTION

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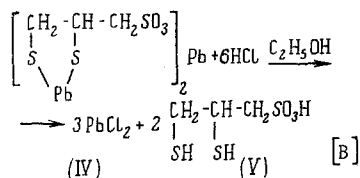
The isolation of sodium 2,3-dimercaptopropanesulfonate (unithiol) is performed from an aqueous solution containing considerable amounts of inorganic and organic impurities (potassium bromide and sodium bromide, acetic acid, and potassium acetate) by converting the unithiol into the water-insoluble complex III [1]:



A part of the lead is tied up in the difficultly soluble lead bromide.

The complex III so obtained is filtered off, washed with a large amount of hot water to partially remove lead bromide, dried to a moisture content of 5-7%, and then decomposed with hydrogen chloride in ethyl alcohol.

The solution of 2,3-dimercaptopropanesulfonic acid, after filtration from lead chloride and refining with activated charcoal, is routed to the stage of unithiol isolation.



As is evident from scheme B, all the lead used in forming complex III is separated in the form of lead chloride - a production by-product, which amounts to about 80% of that introduced into the process, according to data of the "Farmakon" plant.

A result of the investigations which we conducted with the objective of utilizing the lead chloride was a means which we have suggested for preparing complex III using lead chloride instead of lead acetate.

The complete decomposition of complex III with hydrogen chloride is connected with the formation of the alcohol-insoluble lead chloride and the readily soluble acid, V.

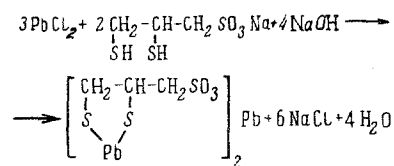
There is a similar situation when the complex III is decomposed in water [1].

The interaction of acid V with lead chloride can be carried out in aqueous medium if the hydrogen chloride set free is neutralized with sodium hydroxide or some other alkali. Thereupon complex III will be formed as a precipitate, and sodium chloride will be formed in solution.

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Formation of complex III takes place according to the following scheme:



All the components are charged in equimolar amounts. The end of the reaction is determined from the absence of unithiol in the reaction mixture by titration of a precise sample of the mother liquor with 0.1 N iodine solution. At the end of the reaction, complex III is filtered off, washed with water, and dried (the yield based on unithiol is 79%).

The lead salt obtained using lead chloride is distinguished by a high content of complex III (90-95%) and by a correspondingly smaller content of lead bromide than when lead acetate is used (75-80%); and three or four times less water is consumed thereby in washing than is provided for in the existing procedure.

The decrease in the amount of lead bromide impurity is explained first by the large affinity of sulfhydryl groups for lead and the very low solubility of complex III in water, and second by the same order of magnitude of solubility of lead chloride and lead bromide in water.

It is natural that the side reaction of lead bromide formation proceeds at a very low rate, and the lead is used up principally in forming complex III.

As a result, when IV is charged in no excess, complex III is obtained in the same yield as when lead acetate is used in 5.5% excess.

The unithiol obtained from complex III formed by the method described conforms completely to the requirements of technical conditions for this preparation.

The reduction in reaction rate in the heterogeneous medium leads to an approximately two-fold increase in time for formation of complex III. However, as a result of reduction in amount of aqueous washes, the over-all duration of the process is appreciably shortened.

The introduction of this method of preparing III into production will permit returning to process about 4 tons of waste IV calculated as lead in putting out one ton of unithiol.

#### EXPERIMENTAL

Lead Complex of 2,3-Dimercaptopropanesulfonic Acid (III). To 150 ml of water was added 33.9 g (0.122 mole) of lead chloride and 6.5 g (0.163 mole) of sodium hydroxide, and the mixture was stirred for 30 min; it was heated to 50-60°, and over a one-hour period at this same temperature there was added an aqueous solution of I containing 17 g of main product; the mixture was allowed to stand at this temperature for 2 h. The end of reaction was determined from the absence of a yellow precipitate when a lead acetate solution was added to a filtered sample of the mother liquor. At the end of the reaction, the hot mixture was filtered, the solid was washed with 30 ml of hot (60°) water, and the precipitate was dried. There was obtained 34.5 g of III. The yield of III was 79%, based on I charged.

#### LITERATURE CITED

1. V. E. Petrun'kin, *Urk. Khim. Zh.*, **22**, 604 (1956).