However, the data of gas chromatograph with mass spectroscopy show that the oxidation products of phenol are hydroquinone, butyric acid, butenoic acid, and preferably acetic acid. And the presence of p-benzoquinone was not observed. It was found out only p-hydroquinone.

To confirm the results of gas chromatography with mass selective detector we analyzed our samples by high performance liquid chromatography without extraction. Sulfuric acid was neutralized by solution of ammonium hydroxide. Benzoquinone was not observed too. It was identified p-hydroquinone (absorption at 290 nm) and carboxylic acids.

Thus, according to UV-spectroscopy the phenol oxidation intermediates are p-benzoquinone and carboxylic acids, and gas chromatograph with mass spectroscopy determines the p-hydroquinone without p-benzoquinone.

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Titanium powder segregation from electrolyte precipitate

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This article is a continuation of the work about fluoridation and electrolysis of titanium concentrates. It is dedicated to the development of one of the most important stages of the proposed fluoride technology of titanium – the process of washing the cathode deposit formed during the electrolytic separation of titanium fluoride melts from the electrolyte salts

As a result of titanium powder electrolytic extraction of fluoride salt melt, cathode precipitate containing 42.50% Ti and impurities are formed: LiF, KF, NaF and complex titanium salts such as Me_2TiO_3, where Me = Na, K, Li, adsorbing on the obtained product during the electrolysis process. There are two methods of segregation: 1) dissolution of previously crushed cathodic precipitate in inorganic acids with the subsequent filtration, drying and packing ("an acid segregation"); 2) segregation of cathodic precipitate with waterless HF and the subsequent its regeneration.

When carrying out an segregation of cathodic precipitate, it is shown, that the content of impurity in titanic powder during "an acid segregation " is much less, than in the titanic sponge synthesized by Kroll's method. However, significant amounts of chemical reagents, which can't be regenerated and returned in the process of segregation are necessary for such process of segregation. Therefore, the "acid" method can be used in vitro for receiving pilot batches of Ti-powder. Essentially new method allowing to carry out regeneration of the used reagent, to minimize quantity of waste, and also to reduce cost price of Ti-powder is necessary for carrying out an segregation in industrial conditions. The cathodic precipitate after electrolysis consists of salts of electrolyte (FLiNaK) in the mixture with Ti-powder, therefore the best solvent of these salts is waterless HF. Segregation should be carried at –20 °C , as pressure of HF vapors doesn't exceed 3–5 %. Titanic powder at such temperature isn't dissolved in HF and remains in a solid phase. The liquid phase enter the HF distillation stage, and then HF comes to the dissolution reactor again. After HF distillation from fluoride salts they are directed to a reutilization in the electrolyzer, and waterless HF is returned in the segregation process again. The offered technology of segregation of titanic powder from a cathodic precipitate with waterless HF completely excludes emission of solid, liquid and gaseous chemically harmful substances in environment, is almost reagentless, has low cost processing of cheap initial raw materials.

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