THE DESALKYLATION OF PHYSCION

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In the isolation of hydroxy derivatives of anthraquinone from plant raw material, products with mp 158-162° C, 159-164° C, and 165-172° C are frequently obtained which consist of mixtures of chrysophanol with physcion which have various quantitative compositions and are clearly separated on paper chromatography if dry pentane is used as the solvent.

In the separation of considerable amounts of this mixture, a method of desalkylation with the subsequent separation and purification of the reaction products is suitable. Desalkylation was previously [1, 2] performed in conc. sulfuric acid at 160° C with a yield of chrysophanol of ~20%. Sarget et al. [3] have described the desalkylation of physcion by pyridine hydrochloride on heating to 165° C for 9 h (yield of emodin 73%) and with 48% HBr in glacial acetic acid for 1 h (yield 60%). Other workers [4] have desalkylated 2-methoxyanthraquinones by heating them in ethylene glycol for 15 h. In selecting conditions for the separation of a mixture of chrysophanol and physcion we have checked, in addition to the methods mentioned, methods using anhydrous $AlCl_3$ in nitrobenzene and dry ether [5], KBr in an acid medium, KBr in organic solvents with the addition of acid at the end of the reaction, and others.

As a result, we propose the following reaction conditions for the desalkylation of the mixtures mentioned. A mixture of chrysophanol and physcion (0.5 g) was heated with 10 g of KI, and 80 ml of ethylene glycol (n_D^{20} 1.4304) for 23 h. Then the reaction mixture was diluted with water and acidified with 10% hydrochloric acid. The yellow precipitate that deposited was filtered off, washed free from chloride ion, dried, and chromatographed on paper in toluene and pentane. The chromatograms showed that the precipitate contained chrysophanol and emodin.

The benzene solution of the reaction product was separated on a column of $NaHCO_3$ –silica gel (8 : 2). The eluate was concentrated giving chrysophanol with mp 190-192° C (from ethanol). Yield 86%.

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