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4-(Tetracyanoethyl)-2,6-dimethylphenol

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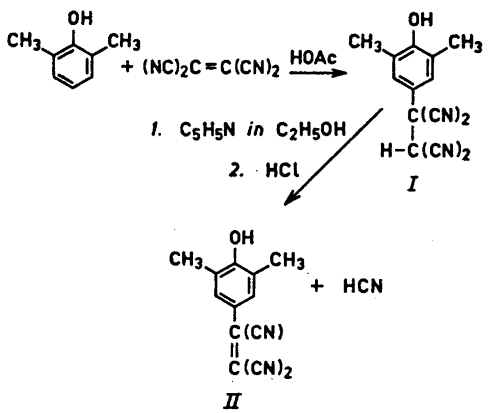
Sausen, Engelhardt and Middleton [1] have described the formation of 4-(tricyanovinyl)-2,6-dimethylphenol (II) in a reaction between tetracyanoethylene and 2,6-dimethylphenol in tetrahydrofuran-pyridine solution. They assumed that the reaction proceeded over an intermediate 4-(tetracyanoethyl)-2,6-dimethylphenol (I). We wish to report the preparation of the latter compound from tetracyanoethylene and 2,6-dimethylphenol in acetic acid solution. It is a white solid which is reasonably stable in acid solution but, in alkaline solution, is converted to the red 4-(tricyanovinyl)-2,6-dimethylphenol (II). It can, however, be dissolved in sodium bicarbonate solution and reprecipitated with hydrochloric acid.



I *II*

We are now studying the scope and limitations of the reaction between tetracyanoethylene and phenols in acidic solution as well as the properties of the tetracyanoethylphenols formed. The results of the study will be presented in a forthcoming paper.

Tetracyanoethylene (0.173 g, 1.35 mmole) and 2,6-dimethylphenol (0.167 g, 1.37 mmole) were dissolved in 15 ml glacial acetic acid and then 1 ml water was added. The reddish-violet solution was discoloured after a few hours and, upon addition of water (10 ml), a white precipitate was formed. It was collected and recrystallized from glacial acetic acid and water (1:1). Yield 0.205 g (61 %). M.p. 154–156° (decomp.). (Found: C 67.4; H 3.97; N 22.2. Calc. for (I), $C_{14}H_{10}ON_4$: C 67.19; H 4.02; N 22.39).

4-(Tetracyanoethyl)-2,6-dimethylphenol (0.145 g, 0.58 mmole) was dissolved in 10 ml ethyl alcohol and 0.5 ml pyridine was added. The solution was heated on a boiling water bath for half an hour. On acidification with 5 N hydrochloric acid, the colour of the dark red solution changed to orange-yellow. Addition of water (10 ml) caused an orange-yellow substance to precipitate. It was collected and recrystallized from glacial acetic acid and water (1:1). Yield 0.122 g (95 %). M.p. 179–181° (decomp.). M.p. for 4-(tricyanovinyl)-2,6-dimethylphenol (II) according to Sausen *et al.*[1] 182–183° (decomp.) *. A specimen of the latter compound obtained from tetracyanoethylene and 2,6-dimethylphenol in alcohol-pyridine solution was found not to depress the melting point of the compound formed from 4-(tetracyanoethyl)-2,6-dimethylphenol.

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* Determined on a preheated block.