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REACTIONS OF TETRACYANOETHYLATED KETONES WITH HYDROCHLORIC AND HYDROBROMIC ACIDS. SYNTHESIS OF 2-CHLORO(BROMO)-3,4-DICYANOPYRIDINES

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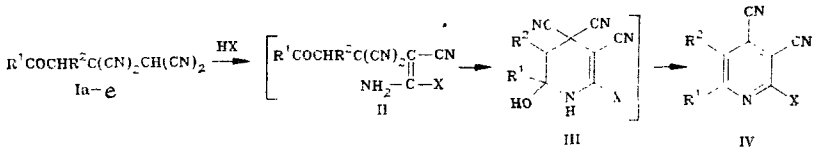
We have discovered that tetracyanoethylated ketones Ia-e [1, 2] react with concentrated aqueous solutions of hydrochloric and hydrobromic acids for 6-120 h at room temperature in an excess of acid, forming 5,6-dialkyl-2-halo-3,4-dicyanopyridines (IVa-e). With hydrobromic acid, the pyridines IV were isolated only with the ketones Ib, e. All the ketones I react with hydrochloric acid. The reaction probably proceeds according to the scheme:



I—IV a $R^1=CH_3$, $R^2=H$; b $R^1=R^2=CH_3$; c $R^1-R^2=-(CH_2)_4-$; d $R^1-R^2=-(CH_2)_3-$; e $R^1=C_2H_5$, $R^2=H$; X=Cl, Br

The structure of the pyridines IV was confirmed by the IR, ^{13}C NMR, and mass spectra, as well as by chemical conversions. The yield in percent; melting point, °C; and IR spectroscopic data, cm^{-1} (suspension in liquid petrolatum) are cited below for each substance. 5,6-Dimethyl-2-chloro-3,4-dicyanopyridine: 84; 72-74; 2250 (C≡N), 1560, 1538 (arom. ring); 5,6-dimethyl-2-bromo-3,4-dicyanopyridine: 61; 103-104; 2248 (C≡N), 1565, 1537 (arom. ring); 6-methyl-2-chloro-3,4-dicyanopyridine: 73; 98-100; 2242 (C≡N), 1560, 1540 (arom. ring); 5,6-tetramethylene-2-chloro-3,4-dicyanopyridine: 62; 95-96; 2245 (C≡N), 1558, 1540 (arom. ring); 5,6-trimethylene-2-chloro-3,4-dicyanopyridine: 70; 77-79; 2250 (C≡N), 1580, 1553 (arom. ring). All the pyridine derivatives obtained have satisfactory analytical characteristics.

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