LETTERS TO THE EDITOR

SYNTHESIS OF BIS(3-CYANO-2-PYRROLYL) DISULFIDES

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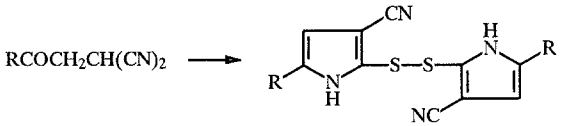
It was observed that substituted bis(3-cyano-2-pyrrolyl) disulfides IIa-d are formed when hydrogen sulfide is passed through solutions of β,β-dicyano ketones Ia-d in organic solvents in the presence of catalytic amounts of triethylamine.



Ia-d II a-d

I, II a R=CH_3; b R=C_6H_5; c R=*p*-$CH_3C_6H_4$; d R=*p*-ClC_6H_4

Bis[5-(p-tolyl)-3-cyano-2-pyrrolyl] Disulfide (IIc, $C_{24}H_{18}N_4S_2$). Hydrogen sulfide was passed through a solution of 0.05 mole of 1-(p-tolyl)-3,3-dicyano-1-propanone in 70 ml of acetone containing a catalytic amount of triethylamine until the starting dicyano ketone vanished (monitoring by TLC). The reaction mixture was then diluted with water, and the precipitate was removed by filtration, washed with water, dried, and recrystallized from acetonitrile to give IIc with mp 248-259°C. IR spectrum (in mineral oil): 3245 (NH), 2240 cm^{-1} (CN). PMR spectrum: 2.33 (CH_3), 7.09 (4-H), 7.24-7.66 (C_6H_4), 12.78 ppm (NH). The yield was 94%.

The compounds listed below were similarly obtained.

Compound IIa ($C_{12}H_{10}N_4S_2$). This compound had mp 207-208°C. IR spectrum: 3270 (NH), 2240 cm^{-1} (CN). The yield was 80%.

Compound IIb ($C_{22}H_{14}N_4S_2$). This compound had mp 253-256°C. IR spectrum: 3280 (NH), 2240 cm^{-1} (CN). The yield was 94%.

Compound IId ($C_{22}H_{12}Cl_2N_4S_2$). This compound had mp 263-265°C. IR spectrum: 3200 (NH), 2240 cm^{-1} (CN). The yield was 85%.

The structure of IIb was established by x-ray diffraction analysis.

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