

Preparation of $1 \cdot C_6H_6 \cdot 3H_2O$

A solution of TCNE (512 mg, 4 mmol) in acetonitrile (20 ml) was added to bis(benzene)vanadium(0) (207 mg, 1 mmol) under vacuum with stirring. The reaction mixture was kept for 2 h under vacuum and then exposed to moist air for 3 h. The dark-green solution was separated, evaporated to dryness under vacuum, and the resulting residue was taken up in dry benzene. Careful removal of benzene under vacuum afforded **I** in 60% yield. Anal.: Calc. for $C_{30}N_{16}H_{12}VO_4$: C, 50.65; N, 31.50; H, 1.70%. Found: C, 50.68; N, 31.50; H, 2.23%.

Preparation of $2 \cdot 4CH_3C_6H_5 \cdot 2.5THF$

A solution of TCNE (192 mg, 1.5 mmol) in dry THF (10 ml) was added to bis(indenyl)ytterbium(II)2THF⁵ (164 mg, 0.3 mmol) in THF (1 ml). After 3 h the reaction mixture was extracted with dry acetonitrile to afford a dark-green solution which was separated and evaporated to dryness. The resulting residue was taken up in dry toluene. Careful removal of toluene under vacuum afforded **II** in 50% yield. Anal.: Calc. for $YbC_{68}N_{20}H_{42}O_{2.5}$: C, 60.40; N, 20.72; H, 3.13%. Found: C, 60.81; N, 20.37; H, 3.15%.

Unfortunately, **1** and **2** cannot be purified by chromatography owing to significant CN hydrolysis in solution in the presence of moisture.