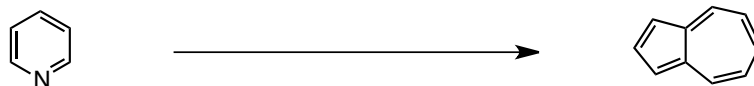


Yu, W. L.; Nunns, T.; Richardson, J.; Booker-Milburn, K. I. *Org. Lett.* **2018**, *20*, 1272-1274.

Explain the following transformation by indicating the chemical entities of (1)-(3).



1-Chloro-2,4-dinitrobenzene (20.3 g, 100 mmol) and anhydrous pyridine (120 mL) under nitrogen atmosphere were heated with stirring at 90 °C for 4 h (formation of a voluminous precipitate₍₁₎, initially colorless, then turning from yellow to finally brown), cooled to 0 °C, then slowly treated dropwise with pyrrolidine (18.1 mL, 220 mmol, clearing and turning to red₍₂₎) and stirred at r.t. for 16 h. The red solution was then treated with freshly (!) distilled cyclopentadiene (8.68 mL, 105 mmol) and then a solution of sodium methoxide in methanol [freshly prepared from sodium (2.30 g, 100 mmol) and MeOH (40.0 mL)] was added dropwise. The reaction mixture was then stirred at r.t. for 16 h, then concentrated by distillation with the application of a Vigreux column (30 cm, bath 130 °C) until 95 °C of the distillate, and subsequently allowed to cool to r.t. The distillate₍₃₎ was treated with anhydrous pyridine (200 mL), then heated to reflux for 6 d (bath 135 °C), and allowed to cool to r.t. After addition of distilled water (200 mL), the reaction flask was attached to a phase separator according to Figure 2, equipped with distilled water (300 mL) and isohexane (300 mL), heated to reflux (bath 125 °C) so that steam distillation and phase separation proceeded simultaneously and steam distilled and extracted until colorless condensation of isohexane (1–2 days). The deep-blue phase of isohexane was washed with 2 M aqueous HCl (3 x 100 mL), distilled water, then dried with Na₂SO₄, evaporated, purified by column separation (basic alumina, activity II, isohexane, first intensely blue band), filtrated through a D5 glass filter and evaporated. Yield: 4.4 g (36%); bluish black leaflets; mp 99–100 °C (Lit. 99–100 °C); R_f = 0.67 (Al₂O₃ neutral, isohexane).

Langhals, H.; Eberspächer, M. *Synthesis* **2018**, *in press*.

DOI: 10.1055/s-003601591906