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4-Nitrophenylferrocene

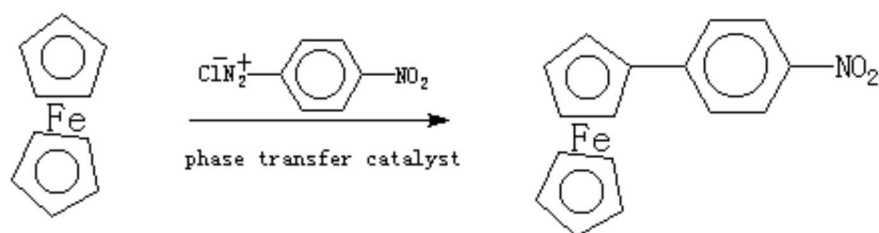
Ping Hu, Ke-Qing Zhao*, and Hong-Bo Xu

Department of Chemistry, Sichuan Normal University, Chengdu, 610066, China

Tel./Fax: 86-28-4764743. Email: zkq2@yahoo.com (Present e-mail: zhao@hrz2.hrz.tu-darmstadt.de)

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4-Nitrophenylferrocene is a candidate for the study of second order non-linear optical materials containing electron donating and electron withdrawing groups in the molecule. It is also an important intermediate for the synthesis of ferrocene-containing liquid crystals [1]. Here we report a modified synthetic method for the preparation of 4-nitrophenylferrocene through arylation of ferrocene by a diazonium salt under phase transfer conditions[2].

4-Nitroaniline (14 g, 100 mmol), 30 ml of water and 30 ml of concentrated hydrochloric acid are mixed together and cooled to 0–5°C. A solution of sodium nitrite (7 g, 100 mmol) in 10 ml of water is added dropwise with stirring. After the addition is complete, the solution is stirred 30 min and kept below 5°C during this period. Ferrocene (9.5 g, 50 mmol), and 1 g hexadecyltrimethylammonium bromide are added to 100 ml ethyl ether and cooled to 0–5°C. The above prepared diazonium salt solution is added dropwise with stirring. After the addition is complete, the reaction mixture is stirred for an additional 5 h at room temperature. The mixture is concentrated by rotary evaporation and the residue washed by water before the solid is steam distilled to recover unreacted ferrocene. The residual crude product is recrystallized from petroleum ether (90–120°C) to give 4-nitrophenylferrocene as violet plates (13.5 g, 70%).

M.p. 169.5–170°C (lit., 163°C, dec.).

IR(KBr, cm^{−1}): 3075, 3100, 1596, 1507, 1341, 1106, 1011.

¹HNMR(DMSO, d₆): 4.03(s, 5H, C₅H₅), 4.5(s, 2H, C₅H₄), 4.7(s, 2H, C₅H₄), 7.5(d, 2H, C₆H₄), 8.2(d, 2H, C₆H₄)

Elemental analysis for C₁₆H₁₃FeNO₂: calculated, C, 61.94; H, 4.19; N, 4.52%. Found: C, 61.73; H, 4.11; N, 4.44%.

References

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Sample Availability: Available from the authors and from MDPI.

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