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4-Formylphenyl 4-Ferrocenylbenzoate

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Ferrocene unit is comparatively large volume and it needs a longer molecular shape in geometry in to support the liquid crystal property [1]. 4-Formylphenyl 4-ferrocenylbenzoate contains a formyl group and can further condenses with amine, so it is a key intermediate for the synthesis of ester-Schiff's base ferocene-containg liquid crystal material. Following is the synthetic procedure. 4-Ferrocenylbenzoic acid [2] (9.6 g, 30 mmol) is dissolved in 150 ml of CHCl₃ and 7.5 ml oxalyl chloride is added. The mixture is refluxed 24 h with stirring under protection of CaCl₂ dry tube. The mixture is concentrated in vacuum. The residue is extracted with 180 ml of petroleum ether (boiling range 90-120°C) three times under refluxing. After cooling, the extracts give product as solid. The product is filtered and dried in vacuum to give 4-ferrocenylbenzoylchloride (8.2 g, 80% in yield, m.p. 134-135°C) and it can be used for next step without further purification. The mixture of 4-hydroxybenzaldehyde(3.1g, 25 mmol), 4-ferrocenylbenzoyl chloride (8.2 g, 25 mmol) and 2 ml pyridine in 100 ml benzene is refluxed 10 h under the protection of a CaCl₂ drying tube until the disappearance of acid chloride as monitored by thin-layer chromatography. After reaction has finished, the clear upper layer is decanted from the oily lower layer. After cooling, the product is collected by filtration to give 4-formylphenyl 4-ferrocenylbenzoate (8.0 g, yield 78%).

M.p.: 195-196°C.

IR(KBr, cm⁻¹): 1720, 1693, 1272.

¹HNMR (CDCl₃, 200 MHz): 10.04(1H, S, CHO), 7.57-8.13(4H, dd, J=8.26Hz, ArCHO), 7.41-8.01(4H, dd, J=8.38Hz, Fc-Ar), 4.46-4.78(4H, d, C₅H₄), 4.08(5H, S, C₅H₅).

Elemental analysis for C₂₄H₁₈FeO₃: calculated, C, 69.73; H, 4.36%. Found: C, 69.55; H, 4.52%.

Reference

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- 2. Zhao, Ke-Qing; Hu, Ping and Xu, Hong-Bo. 4-Ferrocenylbenzoic Acid. *Molecules* **2001**, *6*, M246.

Sample Availability: Available from the authors and from MDPI.

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