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Short Note

1,3,5-Tris{[*N*-(1,3-diphenyl-1*H*-pyrazol-4-yl)methylene]-4-aminophenyl}benzene

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Abstract: 1,3,5-Tris{[*N*-(1,3-diphenyl-1*H*-pyrazol-4-yl)methylene]-4-aminophenyl}-benzene was synthesised from *N*-(4-bromophenyl)-*N*-[(1,3-diphenyl-1*H*-pyrazol-4-yl)-methylene]amine and 1,3,5-tribromobenzene by an Ullman coupling reaction. The synthesized compound was charecterised by NMR, IR, MS and elemental analysis.

Keywords: 1,3-diphenyl-1*H*-pyrazol-4-carboxaldehyde; 1,3,5-tribromobenzene; Ullman coupling

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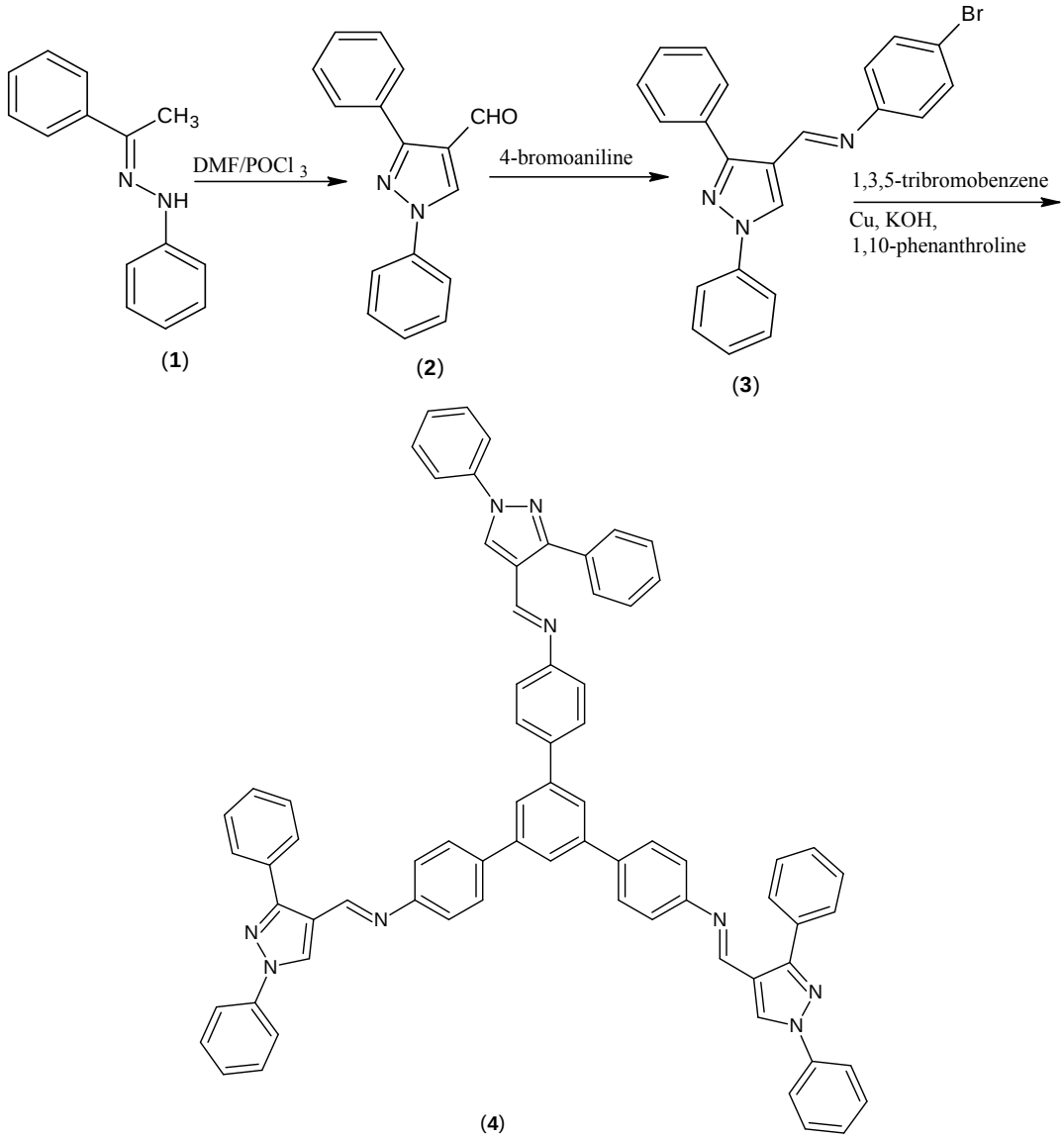
The title compound, 1,3,5-tris{[*N*-(1,3-diphenyl-1*H*-pyrazol-4-yl)methylene]-4-aminophenyl}-benzene, is a highly conjugated pyrazole derivative. Due to this extensive conjugation, the above compound can be potentially used as hole transporting material in photoelectric conversion devices such as solar cells. Usually used hole transporting materials are arylamines [1].

1,3-Diphenyl-1*H*-pyrazole-4-carboxaldehyde (**2**) was prepared according to the known procedure [2]. The yield of the product was 72% and its m.p. was found to be 135 °C. The aldimine derivative, *N*-(4-bromophenyl)-*N*-[(1,3-diphenyl-1*H*-pyrazol-4-yl)methylene]amine (**3**) [2,3] was obtained from 1,3-diphenyl-1*H*-pyrazole-4-carboxaldehyde and 4-bromoaniline and it was recrystallised from ethyl acetate. The yield of the product was 60% with m.p. 127 °C. The aldimine derivative (**3**) (0.3 mmol) and 1,3,5-tribromobenzene (0.1 mmol) were dissolved in DMF. To this solution, 0.1 g of electrolytic copper, 0.01 g of KOH and 0.001 g of 1,10-phenanthroline were added and the reaction mixture was heated for 15 h at 140 °C to get the title compound (**4**). The hot solution was filtered to remove the copper compounds and the base [4,5]. The solvent DMF was evaporated under vacuum and the solid was triturated with ethyl acetate, filtered and further purified by

chromatography on silica gel using ethyl acetate-hexane (1:3) as eluent. According to a theoretical calculation, the formed compound may be the *E* isomer (least steric hindrance). Furthermore, the ^{1}H NMR spectral data indicate that there is only one product formed.

Scheme 1. Synthesis of 1,3,5-tris{[*N*-(1,3-diphenyl-1*H*-pyrazol-4-yl)methylene]-4-aminophenyl}benzene.



Yield: 60%.

M.p. 149 °C.

IR (KBr) cm^{-1}: 3019.2 (Ar C-H$_{str}$), 1026.9 (Ar C-H$_{def}$), 1422.3 (Ar C=C$_{str}$), 1599.7 (C=N$_{str}$), 1338.4 (Ph-N$_{str}$).

^{1}H NMR (500 MHz, $CDCl_3$) δ ppm: 10.07 (s, 3H, pyrazole 5-H), 8.56 (s, 3H, HC=N), 7.85-7.80 (m, 12H, N-Ph-), 7.55-7.35 (m, 30 H, Ph), 7.27 (s, 3H, Ph).

^{13}C NMR (75 MHz, DMSO-d_6): 163.7, 158.0, 147.9, 137.6, 136.5, 135.0, 129.1, 129.0, 128.7, 128.5, 128.1, 127.0, 126.0, 125.2, 122.5, 118.8, 108, 99.7.

MS: m/z (ES), 1042 [(M+1)$^{+}$].

Elemental analysis calculated for $C_{72}H_{51}N_9$ (1042.24): C, 82.97%; H, 4.93%; N, 12.10%. Found: C, 83.00%; H, 4.90%; N, 12.10%.

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