Electronic Supplementary Material for CrystEngComm


**Synthesis of 1,4-bis[(pyrazol-1-yl)methyl]benzene**

Pyrazole (.776 g, 11.4 mmol) was dissolved in 20 mL dry THF with NaOH (4.56 g, 114.0 mmol) in a 250 mL round-bottomed flask under a $N_2$ atmosphere and stirred for 2 h, upon which a solution of α,α′-dichloro-*p*-xylene (1 g, 5.68 mmol) in 10 mL dry THF was added. The mixture was stirred at room temperature overnight under a $N_2$ atmosphere, after which 200 mL distilled water was added to dissolve the NaOH and NaCl. 200 mL ethyl acetate was added and the two layers were separated using a separatory funnel. The organic layer was concentrated *via* rotorary evaporation to produce a light yellow oil. The product was isolated by column chromatography (hexanes → ethyl acetate) to yield a white solid. Yield 0.947 g (70 %); mp 92˚ C; $^1$H NMR, 200 MHz, DMSO-d6, 5.291 (s, 4H), 6.248 (t, 2H, *J*=4 Hz), 7.162 (s, 4H), 7.434 (d, 2H, *J*=1), 7.792 (d, 2H, *J*=2.2 Hz).

**Synthesis of 1,4-bis[(3,5-dimethylpyrazol-1-yl)methyl]benzene**

3,5-dimethylpyrazole (2.19 g**,** 22.8 mmol) was dissolved in 40 mL dry THF with NaOH (9.12 g, 228.0 mmol) in a 250 mL round-bottomed flask under a $N_2$ atmosphere and stirred for 2 h, upon which a solution of α,α′-dichloro-*p*-xylene (2 g**,** 11.36 mmol) in 20 mL dry THF was added. The mixture was stirred at room temperature for 48 hours under a $N_2$ atmosphere, after which 200 mL distilled water was added to dissolve the NaOH and NaCl. 200 mL ethyl acetate was added and the two layers were separated using a separatory funnel. The organic layer was dried over $MgSO_4$, then concentrated *via* rotorary evaporation to produce a white solid. The product was isolated by column chromatography (hexanes → ethyl acetate) to yield a white solid. Excess 3,5-dimethylpyrazole was removed by dissolving product in ethyl acetate and washing with 1:1 HCl/water. Yield 2.684 g (79 %); mp 87-90˚ C; $^1$H NMR, 200 MHz, DMSO-d6, 2.071 (s, 6H), 2.134 (s, 6H), 5.130 (s, 4H), 5.821 (s, 2H), 7.035(s, 4H).

**Synthesis of 1,4-bis[(pyrazol-1-yl)methyl]benzene:3,5-dinitrobenzoic acid (1a)**

1,4-bis[(pyrazol-1-yl)methyl]benzene (.015 g, .063 mmol) and 3,5-dinitrobenzoic acid (.027 g, .126 mmol) were dissolved in 2 mL of ethanol. Light yellow crystals were obtained after six days of slow evaporation of the solvent. mp 135˚ C

**Synthesis of 1,4-bis[(3,5-dimethylpyrazol-1-yl)methyl]benzene:2,6-dichlorobenzoic acid (2a)**

1,4-bis[(3,5-dimethylpyrazol-1-yl)methyl]benzene (.015 g, .043 mmol) and 2,6-dichlorobenzoic acid (.016 g, .086 mmol) were dissolved in 2 mL of ethanol. Colorless crystals were obtained after a week of slow evaporation of the solvent. mp 130-132˚ C

**Synthesis of 1,4-bis[(3,5-dimethylpyrazol-1-yl)methyl]benzene:pentamethylbenzoic acid (2b)**

1,4-bis[(3,5-dimethylpyrazol-1-yl)methyl]benzene (.015 g, .043 mmol) and pentamethylbenzoic acid (.017 g, .086 mmol) were dissolved in 2 mL of ethanol. Colorless square prisms were obtained after a week of slow evaporation of the solvent. mp 156-158˚ C