Supporting Information for

2-D Coordination polymers of hexa(4-cyanophenyl)[3]-radialene and silver(I): anion···π-interactions and radialene C-H···anion hydrogen bonds in the solid-state interactions of hexaaryl[3]-radialenes with anions

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4,4'-Diiododiphenylmethane.

4,4'-Diaminodiphenylmethane (3.0g, 15.3mmol) in concentrated sulfuric acid (15mL) was stirred at 0°C. Sodium nitrite (3.0g, 43.2mmol) in water (10mL) was added dropwise over a period of 10 min. The resultant solution was stirred at 0°C for 30 min followed by the addition of potassium iodide (17.5g, 102mmol) in water (60mL). The reaction mixture was heated at 50°C for 1 h then cooled to room temperature and neutralized with 50% sodium hydroxide solution (20mL). The mixture was extracted with dichloromethane (6×50mL). The organic fractions were combined and washed with 1M hydrochloric acid solution (100mL), 1M sodium thiosulfate solution (100mL), dried over anhydrous magnesium sulfate and the solvent evaporated under vacuum to yield a brown solid. Purification via silica chromatography eluting with hexane yielded diiododiphenylmethane as white needles (3.40g, 53%). Mp. 80-82°C (lit.[1] 85-86°C); 1H NMR ($CDCl_3$): δ 3.85 (s, 2H, CH_2); 6.90 (d, 4H, [J = 8.2 Hz], H2, H6); 7.60 (d, 4H, [J = 8.2 Hz], H3, H5).

4,4'-Dicyanodiphenylmethane.

A mixture of 4,4'-diidodiphenylmethane (1.0g, 2.4mmol) and copper cyanide (0.66g, 7.2mmol) in dry N,N-dimethylformamide (15mL) was heated at 100°C for 2 days. The cooled reaction mixture was diluted with ethyl acetate (40mL) and the resultant solution washed with concentrated ammonia solution (30mL), water (20mL), brine (20mL), dried over anhydrous magnesium sulfate and the solvent evaporated under vacuum to yield dicyanodiphenylmethane as an off white solid (0.5g, 95%). Mp. 160-163°C (lit.[2] 166-167°C); 1H NMR ($CDCl_3$): δ 4.09 (s, 2H, CH_2); 7.27 (d, 4H, [J = 8.2 Hz], H2, H6); 7.61 (d, 4H, [J = 8.2 Hz], H3, H5).

References

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