Electronic Supplementary Information

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Phosphine Oxide-based Conjugated Microporous Polymers with Excellent CO_2 Capture Properties

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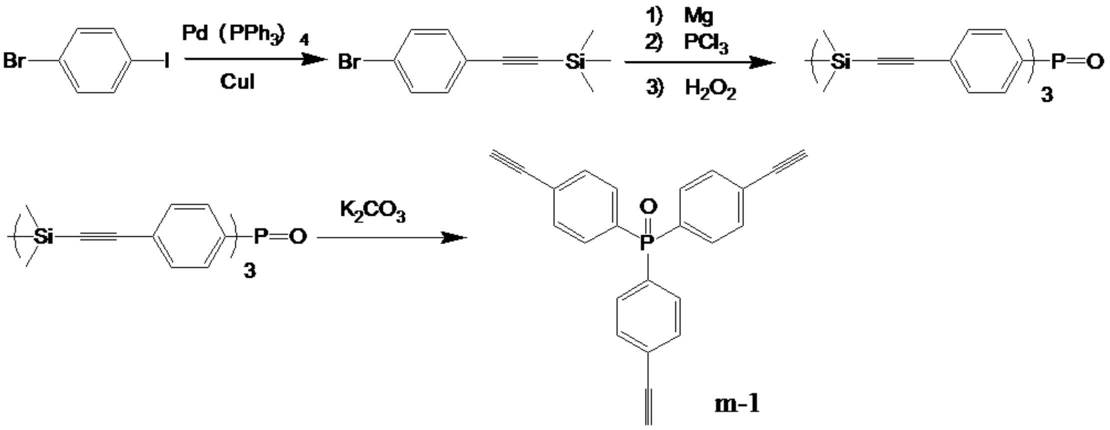
Experimental section

m-1



m-1

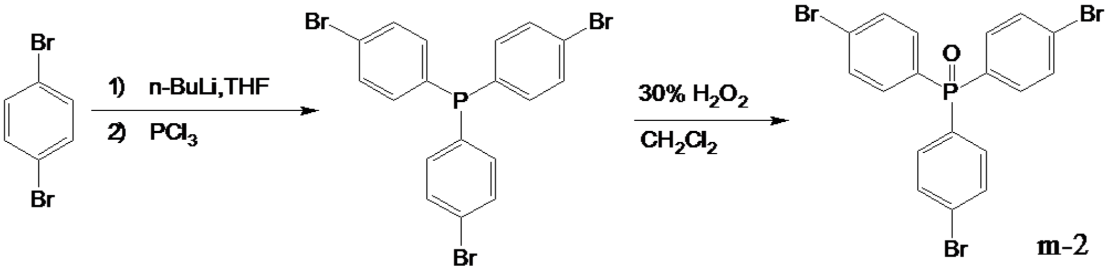
Tris(4-ethynylphenyl)phosphine oxide (m-1):

Tris-[4-(trimethylsilylethynyl)-phenyl]-phosphine oxide was synthesized by the procedure in literature.[1,2] To a solution of tris[4-(trimethylsilylethynyl)-phenyl] -phosphine oxide (920 mg, 1.64 mmol) in a mixture of dichloromethane (8 mL) and methanol (15 mL) was added potassium carbonate (224 mg, 1.64 mmol). The resulting mixture was stirred at room temperature for 30 minutes and then concentrated under reduced pressure to get a white solid (536 mg, 94%).

^{1}H NMR ($CDCl_3$, 600 MHz): 7.64-7.59 (m, 12 H), 3.22 (s, 3 H).

^{13}C NMR ($CDCl_3$, 150MHz): 132.5, 132.2, 131.9, 126, 82, 80.

m-2



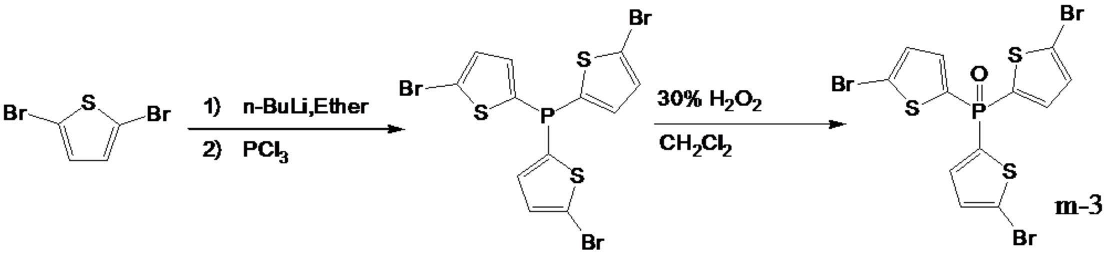
m-2

Tris(4-bromophenyl)phosphine oxide (m-2): The compound was synthesized as the procedure in literature.[3] To a solution of 1, 4-dibromobenzene (2.36 g, 10 mmol) in anhydrous THF (100 mL), n-butyl lithium (2.26 M in hexane, 4.2 mL, 9.5 mmol) was added dropwise at −78 °C. The reaction was kept at this temperature for 2 h, and then phosphorus trichloride (0.62 mL, 4.6 mmol) was added. After the mixture was stirred for 1 h at −78 °C, it was allowed to warm to room temperature, stirred overnight followed by quenching with 5 mL of methanol. Water was added, and the mixture was extracted with CH_2Cl_2, washed with water, and dried over anhydrous Na_2SO_4. After the solvent had been completely removed, 30% hydrogen peroxide (15 mL) and CH_2Cl_2 (30 mL) were added to the obtained residue, and the mixture was stirred overnight at room temperature. The organic layer was separated and washed with water and then brine. The extract was evaporated to dryness, and the residue was purified by column chromatography on silica gel using dichloromethane/methanol (30:1 by vol) as the eluent to give a white solid (663 mg, 28%).

^{1}H NMR ($CDCl_3$, 600 MHz): 7.67-7.60 (m, 6H), 7.54-7.44 (m, 6H).

^{13}C NMR ($CDCl_3$, 150 MHz): 137, 135, 131, 122.

m-3



m-3

Tris(5-bromothiophen-2-yl)phosphine oxide (m-3):

Tri(2-bromo-5-thienyl)phosphine was synthesized as the procedure described in our previous paper.[4] Tri(2-bromo-5-thienyl)phosphine (1 g, 1.93 mmol) and hydrogen peroxide (5 mL, 35% in water) were dissolved in 50 mL of dichloromethane. The mixture was stirring at room temperature for 2h. The organic phase was washed three times with 100 mL of water and then dried over $MgSO_4$. After evaporation of solvent, the residue was purified by column chromatography (silica gel, dichloromethane) to give m-3 as a white solid (823 mg, 80%).

^{1}H NMR ($CDCl_3$, 600 MHz): 7.36 (m, 3H), 7.17 (m, 3H).

^{13}C NMR ($CDCl_3$, 150 MHz): 137, 134, 131, 122.

1. V. H. Gessner and T. D. Tilley, *Organic letters*, 2011, **13**, 1154-1157.
2. R. Métivier, R. Amengual, I. Leray, V. Michelet and J.-P. Genêt, *Organic letters*, 2004, **6**, 739-742.
3. C. Liu, Y. Li, Y. Li, C. Yang, H. Wu, J. Qin and Y. Cao, *Chemistry of Materials*, 2013, **25**, 3320.
4. X. Chen, S. Qiao, Z. Du, Y. Zhou and R. Yang, *Macromolecular rapid communications*, 2013, **34**, 1181-1185.