

Supporting Information

Organic nanotubes assembled from isophthalamides and their application as templates to fabricate Pt nanotubes

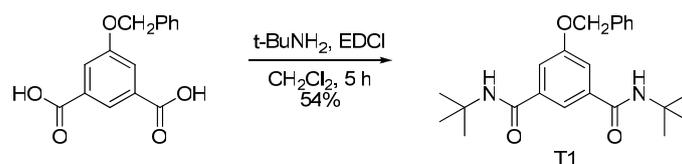
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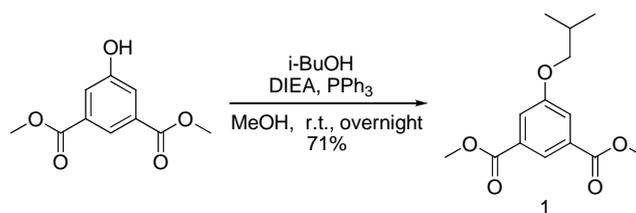
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Synthesis and Characterization

General methods. Melting points are uncorrected. All reagents and chemicals were obtained from commercial sources and used without further purification unless otherwise noted. The solvents have been purified by standard procedures before use. Silica gel (10- 40 μ) was used for all column chromatography. The ^1H NMR spectra were recorded on Bruker Avance 300 MHz or Varian Mercury 300 MHz spectrometers in the indicated solvents. Chemical shifts are expressed in parts per million (δ) using residual proton resonances of the deuterated solvents as the internal standards. MS and HR-MS were carried out in Shanghai Institute of Organic Chemistry (SIOC) MS center. Elemental analyses were obtained on a Vario EL Elemental Analyzer. X-ray crystallography was carried on a Bruker AXS Smart apex II diffractometer. Powder X-ray diffraction (XRD) patterns were recorded on a Bruker D8 Avance diffractometer. High- resolution transmission electron microscopy (HR-TEM) images and EDX were recorded on a JEOL JEM-2010 microscope equipped with energy-dispersive X-ray spectroscopy; Low-resolution transmission electron microscopy (LR-TEM) images were recorded on a JEOL JEM-1230 microscope. Scanning electron microscopy (SEM) experiments were conducted on a JEOL JSM-6390-LV microscope. 5-(benzyloxy)isophthalic acid was prepared according to the literature [Diederich, F.; Schuermann, G.; Chao, I. *J. Org. Chem.* 1988, 53, 2744-2757.]

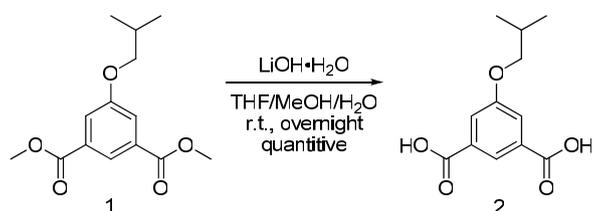


N,N'-di-tert-butyl-3-(benzyloxy)isophthalamide (T1). A mixture of 5-(benzyloxy)-isophthalic acid (0.27 g, 1.0 mmol), 1-(3-dimethylamino-propyl)-3-ethylcarbodiimide hydrochloride (EDCI) (0.58 g, 3.0 mmol) and tert-butylamine (0.26 mL, 2.5 mmol) in dichloromethane (15 mL) was stirred at room temperature for 5 hours. After the solvent was removed, the resulting residue was subjected to flash column chromatography (CH₂Cl₂ to CH₂Cl₂/methanol 100:1) to give compound T1 as white solid (0.21 g, 54%). M.p. 257-258 °C. ^1H NMR (CDCl₃, 300 MHz): δ 7.65 (s, 1H), 7.44-7.27 (m, 7H), 6.09 (br, 2H), 5.11 (s, 2H), 1.46 (s, 18H). ^{13}C NMR (100 MHz, CDCl₃): δ 165.8, 158.8, 137.4, 136.1, 128.7, 128.3, 127.7, 117.2, 115.7, 70.4, 51.9, 28.7. MS (MALDI-FT): m/z 383.2 [M + H]⁺, 405.2 [M + Na]⁺. Anal. Calcd for C₂₃H₃₀N₂O₃: C, 72.22; H, 7.91, N 7.32. Found: C, 72.30; H, 8.01; N, 7.08.

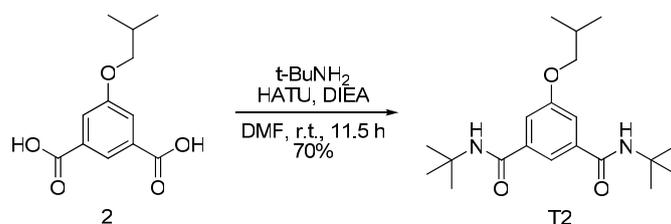


Dimethyl 5-(isobutyloxy)isophthalate (1). To a stirred solution of dimethyl 5-hydroxyisophthalate (4.00 g, 19.0 mmol), triphenylphosphine (5.24 g, 20.0 mmol) and isobutanol (2.40 ml, 25.9 mmol) in methanol (50 ml), N,N-diisopropyl ethylamine (4.04 g, 20.0 mmol) was added at 0 °C. The mixture was kept at 0 °C for 0.5 hour and then stirred at room

temperature overnight. After removal of the solvent with a rotavapor, the resulting residue was subjected to flash column chromatography (CH₂Cl₂/petroleum ether 1:1) to give compound **1** as a white solid (3.59 g, 71%). M.p. 50-53 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.26 (s, 1H), 7.75 (s, 2H), 3.94 (s, 6H), 3.80 (d, *J* = 6.6 Hz, 2H), 2.18-2.05 (m, 1H), 1.04 (d, *J* = 6.9 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 166.2, 159.4, 131.7, 122.7, 119.9, 75.0, 52.3, 28.2, 19.1. MS (EI): *m/z* 266 [M]⁺. Anal. Calcd for C₁₄H₁₈O₅: C, 63.15; H, 6.81. Found: C, 63.21; H, 6.68.



5-(isobutyloxy)isophthalic Acid (2) To a stirred solution of compound **1** (0.69 g, 2.59 mmol) in THF (12 mL), methanol (4 mL), and water (4 mL) was added lithium hydroxide monohydrate (0.24 g, 5.70 mmol). The mixture was stirred at room temperature overnight and the solvent removed with a rotavapor. The resulting residue was acidified with hydrochloric acid (1 N) to pH = 1 and the formed precipitate was filtered, washed with water (20 mL × 2), dried under vacuum to give compound **2** as a white solid (0.62 g, 100%). M.p. 230 °C (sublimation) ¹H NMR (300 MHz, DMSO-*d*₆): δ 13.25 (s, 2H), 8.09 (s, 1H), 7.65 (s, 2H), 3.87 (m, 2H), 2.08-2.00 (m, 1H), 1.01 (d, *J* = 4.5 Hz, 6H). ¹³C NMR (100 MHz, DMSO-*d*₆): δ 166.4, 158.9, 132.6, 122.1, 119.0, 74.2, 27.6, 18.9. MS (ESI): *m/z* 237.1 [M - H]⁻. HRMS (EI) *m/z* Calcd for C₁₂H₁₄O₅ [M]⁺ 238.0841, Found 238.0837. Anal. Calcd for C₁₂H₁₄O₅: C, 60.50; H, 5.92. Found: C, 60.38; H, 5.92.



N,N'-di-tert-butyl-3-(isobutyloxy)isophthalamide (T2) A mixture of 5-(isobutyloxy)isophthalic acid (0.24 g, 1.00 mmol), tert-butylamine (0.26 mL, 2.5 mmol), *o*-(7-azabenzotriazol-1-yl)-*N,N,N',N'*-tetramethyluroniumhexafluorophosphate (HATU) (1.14g, 3.00 mmol), and *N,N*-diisopropylethylamine (DIEA) (1.55 mL, 9.00 mmol) in DMF (10 mL) was stirred at room temperature for 11.5 hours. Upon removal of the solvent, the resulting residue was subjected to flash column chromatography (CH₂Cl₂/petroleum ether 1:2 to 1:1) to give **T2** as a white solid (0.24 g, 70%). Single crystal for X-ray crystallography was grown from ethyl acetate-methanol by slow evaporation. M.p. 260 °C (decomp.) ¹H NMR (CDCl₃, 300 MHz): δ 7.60 (s, 1H), 7.35 (s, 2H), 6.09 (s, 2H), 3.78 (d, *J* = 6.6 Hz, 2H), 2.12-2.04 (m, 1H), 1.46 (s, 18H), 1.01 (d, *J* = 6.6 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 166.0, 159.3, 137.3, 116.6, 115.4, 74.8, 51.8, 28.8, 28.2, 19.1. MS (EI): *m/z* 348 [M]⁺. Anal. Calcd for C₂₀H₃₂N₂O₃: C, 68.93; H, 9.26; N 8.04. Found: C, 69.35; H, 9.40; N, 7.71.

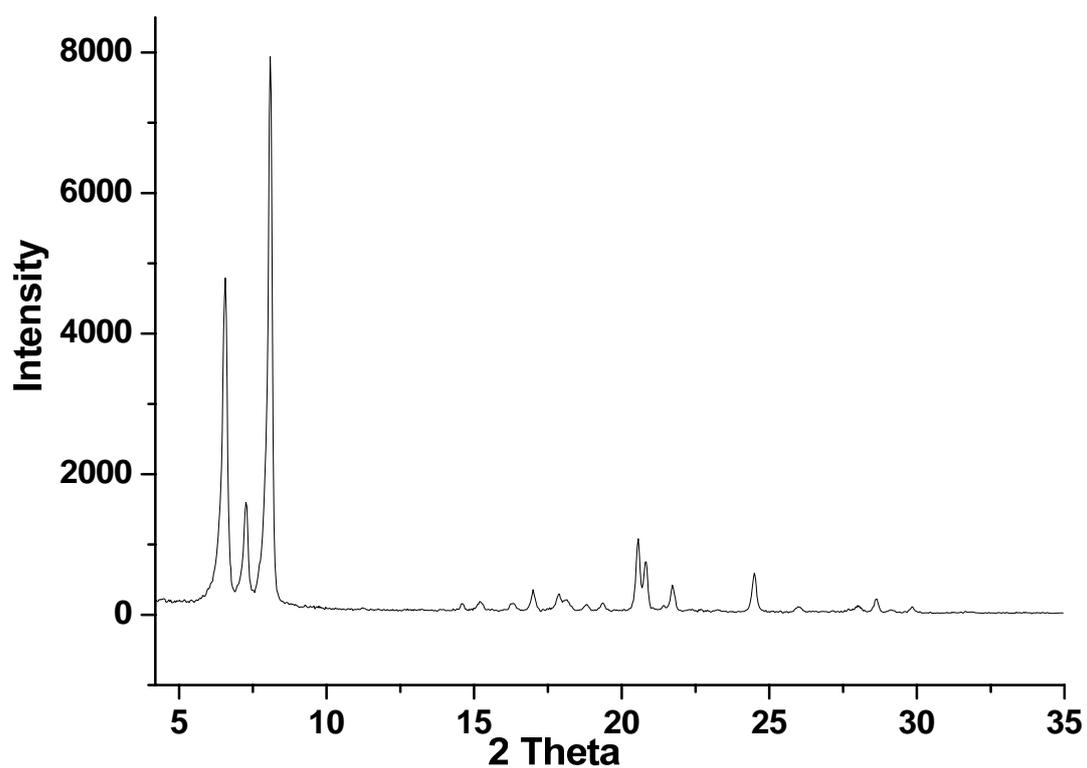


Figure S1. Powder XRD pattern of the T1 nanotubes obtained from methanol.



Figure S2. TEM image of Pt-organic nanotubes after standing for two weeks.

Table S1 Geometrical parameters of the NH...O hydrogen bonds^a

Atoms	Bond angle (°)	Bond length (Å)
N1 H1A O4	152.77	2.089
N2 H2B O5	161.38	2.056
N3 H3A O7	157.90	2.101
N4 H4B O8	144.70	2.087
N5 H5A O2A	154.82	2.084
N6 H6B O1A	148.34	2.101

^a Symmetry code: $-x + 1/2, y - 1/2, -z + 1/2$.

