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Preparation of 2-(4-Fluorophenyl)-6-(methanesulfonyloxy)benzothiophene (2i):

2-(4-Fluorophenyl)-6-hydroxybenzothiophene. A mixture of 2-(4-fluorophenyl)-6-methoxybenzothiophene (prepared by methods described in Ref. 11,14) (1.0 g, 3.9 mmol) and dry pyridine hydrochloride (7.0 g) was heated at 170-200°C for 2h. The mixture was then cooled to 150°C and poured into ice water (200 mL) and the resulting precipitate collected, washed with excess water, and dried. Recrystallization from methanol gave 0.54 g (57%) of the phenol as an off-white crystalline solid: MS (EI) m/e 244 (M⁺). Anal. calc'd. for C₁₄H₉FOS: C, 68.83; H, 3.71. Found: C, 68.58; H, 3.93.

2-(4-Fluorophenyl)-6-(methanesulfonyloxy)benzothiophene (2i). A solution of the phenol obtained above (1.9 g, 7.8 mmol) and *N,N*-dimethylaminopyridine (10 mg) in dry pyridine (40 mL) at 22°C was treated with methanesulfonyl chloride (1.1 g, 9.3 mmol). After stirring 18 h, the reaction was poured into water (150 mL) and the resulting precipitate was collected, washed with excess water and dried. Recrystallization from ethanol/ethyl acetate (9:1) gave 2.3 g (92%) of the methanesulfonyl ester. ¹H NMR δ 3.25 (s, 1H), 7.20 (t, J = 9 Hz, 2H), 7.35 (m, 1H), 7.51 (s, 1H), 7.73 (m, 2H), 7.83 (m, 2H).

Preparation of 2-[4-(4-Fluorobenzoyloxyphenyl)]-7-(4-fluorobenzoyloxy)benzothiophene (2j):

2-Carbomethoxy-7-methoxybenzothiophene. A solution of 3-methoxy-2-nitrobenzaldehyde (20 g, 110 mmol) in DMF (200 mL) and methyl thioglycolate (14.7 mL, 160 mmol) was stirred at 0°C while lithium hydroxide (9.2 g, 380 mmol) was added portionwise over 1 h. After warming to room temperature, the mixture was stirred an additional 30 min then poured into ice water to give a light pink precipitate that was filtered and dried *in vacuo* providing 6.2 g (26% yield) of the title compound: ¹H NMR δ 3.96 (s, 3H), 4.02 (s, 3H), 6.88 (d, J = 8 Hz, 1H), 7.37 (m, 1H), 7.50 (d, J = 8 Hz, 1H), 8.06 (m, 1H); MS (FD) m/e 213 (M⁺).

7-Methoxybenzothiophene-2-carboxylic acid. A sample of 2-carbomethoxy-7-methoxybenzothiophene (10 g, 45 mmol) was dissolved in THF/water(1:1, 1 L), and treated with lithium hydroxide (1.4 g, 56 mmol) for 18 h at room temperature. The mixture was concentrated *in vacuo* to a smaller volume, diluted with 100 mL of water, washed with Et₂O, acidified with 60 mL 1 N HCl, and extracted with EtOAc (3x). The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated and triturated with Et₂O to give 8 g (85% yield) of the title product as an off-white solid, mp 244-246°C: ¹H NMR δ 3.70 (s, 3H), 6.58 (d, J = 8 Hz, 1H), 7.05 (m, 1H), 7.18 (d, J = 8 Hz, 1H), 7.68 (s, 1H); MS (FD) m/e 208 (M⁺).

7-Methoxybenzothiophene. A mixture of 7-methoxybenzothiophene-2-carboxylic acid (10.4 g, 50 mmol), copper (2.5 g, 40 mmol), copper(II)oxide (3 g, 38 mmol), and dipyriddy (0.3 g, 2 mmol) in quinoline (90 mL) was vigorously stirred at 180-190 °C for 2.5 h. After cooling to room temperature, the mixture was filtered through a pad of sea sand and the filter pad washed with Et₂O (2x150 mL). The quinoline/Et₂O filtrate was washed successively with brine, 0.5 N HCl (4x), then brine, dried (Na₂SO₄), and concentrated. The residue was purified by preparative HPLC followed by flash chromatography (hexane:EtOAc gradient) to give 7.7 g (94% yield) of the title product as a yellow oil: ¹H NMR δ 4.14 (s, 3H), 6.92 (d, J = 8 Hz, 1H), 7.48 (m, 2H), 7.59 (m, 2H); MS (FD) m/e 164 (M⁺).

7-Methoxybenzothiophene-2-boronic acid. A solution of 7-methoxybenzothiophene (5g, 30 mmol) in THF (70 mL) was stirred at -70°C while *n*-butyllithium (1.6 M in hexanes, 21 mL, 33 mmol) was added dropwise over 15 min. After an additional 15 min at -70°C, the mixture was treated dropwise with triisopropylborate (7.8 mL, 33 mmol) stirred 15 min at -70°C and allowed to warm to 0°C. The mixture was then poured into 250 mL 1 N HCl/ ice, stirred 20 min and extracted with EtOAc (4x). The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and triturated with petroleum ether to give 5.8 g (93%) of the title product as a white solid: ¹H

NMR δ 3.82(s, 3H), 6.57 (d, $J = 8$ Hz, 1H), 7.0-7.1 (m, 2H), 7.22 (m, 2H), 7.65 (s, 1H).

2-(4-Methoxyphenyl)-7-methoxybenzothiophene. A solution of 4-bromoanisole (2.8 mL, 22 mmol) and tetrakis(triphenylphosphine)palladium(0) (0.8 g, 0.7 mmol) in degassed toluene (250 mL) was treated with 7-methoxybenzothiophene-2-boronic acid (5 g, 24 mmol) in EtOH (10 mL) and aqueous Na_2CO_3 (2 M, 12 mL, 24 mmol) and heated at reflux for 2 h. After cooling to room temperature, the mixture was poured into saturated aqueous NaHCO_3 and extracted with EtOAc (3x), and the combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated. Purification by flash chromatography (hexane:EtOAc gradient) followed by trituration with Et_2O :pet ether gave 2.75 g (47%) of the product as a crystalline solid, mp 125-126°C, ^1H NMR δ 3.86 (s, 3H), 4.02 (s, 3H), 6.73 (m, 2H), 6.95 (m, 2H), 7.28-7.44 (m, 3H), 7.66 (m, 2H); MS (FD) m/e 270 (M^+).

2-(4-Hydroxyphenyl)-7-hydroxybenzothiophene. A mixture of 2-(4-methoxyphenyl)-7-methoxybenzothiophene (0.27 g, 1 mmol) and pyridine hydrochloride (5g, 43 mmol) was stirred at 170-180 °C for 4 h, then cooled, stirred in dilute aqueous HCl and extracted with EtOAc (3x). The organic extracts were combined, washed with dilute aqueous HCl followed by brine, dried (Na_2SO_4), and concentrated to give 0.2 g (82%) of the title product as a tan solid: ^1H NMR ($\text{DMSO}-d_6$) δ 6.67 (m, 1H), 6.82 (m, 2H), 7.18 (m, 2H), 7.27 (m, 2H), 7.30 (m, 1H), 8.90 (s, 1H), 9.30 (s, 1H); MS (FD) m/e 242 (M^+).

2-[4-(4-Fluorobenzoyl)oxyphenyl]-7-(4-fluorobenzoyloxy)benzothiophene (2j). A solution of 2-(4-hydroxyphenyl)-7-hydroxybenzothiophene (0.25 g, 1 mmol) and N,N -dimethylaminopyridine (~0.2 g, 0.1 mmol) in pyridine (10 mL) was treated with 4-fluorobenzoyl chloride (0.28 mL, 2.4 mmol) and the reaction mixture stirred for 20 h at room temperature. The reaction mixture was poured into ice water to give a white precipitate which was filtered and dried. The filtrate

was extracted with EtOAc:EtOH (10:1, 2x), and the combined organic extracts were washed with 0.1 N HCl, saturated aqueous NaHCO₃, brine, dried (Na₂SO₄), and concentrated to give 0.1 g of material that was combined with the original precipitate to give a total of 0.45 g (92%) of **2j** as a white solid, mp 210-215 °C: ¹H NMR δ 6.8-7.0 (m, 7H), 7.13 (m, 1H), 7.4-7.5 (m, 3H), 7.88 (m, 2H), 7.97 (m, 2H); MS (FD) m/e 486 (M⁺). Anal. calc'd. C₂₈H₁₆F₂O₄S: C, 69.13; H, 3.32. Found: C, 68.93; H, 3.49.