Supporting Information

A General, Efficient and Inexpensive Catalyst System for the Coupling of Aryl Iodides and Thiols

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General considerations:

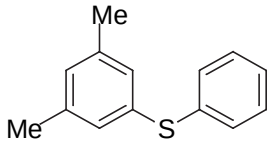
Copper(I) iodide (98% purity) was purchased from Strem Chemical. Potassium carbonate, 2-propanol, ethylene glycol and hexane were purchased from Mallinckrodt. It should be noted that 2-propanol (reagent grade, 4 L bottle) and ethylene glycol (bench grade, 4 L bottle) were used directly *without* drying or degassing. All thiols and aryl halides were used as received. Silica gel (230-400 mesh) and ethyl acetate were purchased from Merck. Elemental analysis were performed by Atlantic Microlabs, Inc., Norcross, GA 30091. ^{1}H NMR and ^{13}C NMR were recorded on a Varian 300 MHz instrument with chemical shifts reported relative to residual deuterated solvent peaks. Gas chromatographic analysis were performed on a Hewlett Packard 6890 instrument with FID detector and a Hewlett Packard 10 m × 0.2 mm i.d. HP-1 capillary column. Mass spectra (GC-MS) were recorded on a Hewlett Packard model GCD. All yields reported represent an average of at least two independent runs. Characterization data for previously unknown compounds were determined from a single run with isolated yields. Compounds described in the literature were characterized by comparing their ^{1}H, ^{13}C NMR and GC-MS to the previously reported data.

General procedure for copper-catalyzed carbon-sulfur bond formation.

Cu(I) iodide (10 mg, 0.05 mmol), potassium carbonate (276 mg, 2.0 mmol) and the aryl iodide (1.0 mmol) (if a solid) were added to a screw-capped test tube with

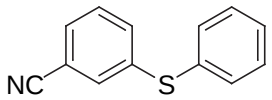
Teflon-lined septum. The tube was evacuated and backfilled with argon (3 cycles). 2-Propanol (1.0 mL), ethylene glycol (111µL, 2.0 mmol), aryl iodide (1.0 mmol) (if liquid) and the thiol (1.0 mmol) were added by syringes at room temperature. The tube was heated to 80 ˚C and stirred for 18-24 h. The reaction mixture was then allowed to reach room temperature. Ethyl acetate (approx. 5 mL) and dodecane (227 µL, GC standard) were added. The aliquot was analyzed GC. The reaction mixture was then filtered and concentrated. The crude product was purified by flash column chromatography on silica gel to afford the desired thioether.

Characterization data for products shown in Table 1



3,5-Dimethylphenyl phenyl sulfide[1] (Table 1, entry 1).

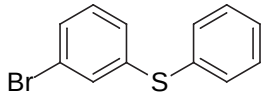
The general procedure was followed (18 h). 5-Iodo-*m*-xylene (144 µL, 1.0 mmol), thiophenol (103 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3,5-dimethylphenyl phenyl sulfide (196 mg, 92% yield) as colorless liquid. Column chromatography solvent (hexane). R_f = 0.5 (hexane). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.19-7.29 (m, 5 H), 6.97 (s, 2 H), 6.87 (s, 1 H). MS (EI) *m/z* (relative intensity) 214 (100), 137 (30).



3-Cyanophenyl phenyl sulfide[2] (Table 1, entry 2).

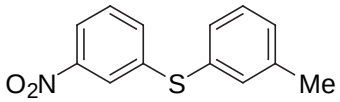
The general procedure was followed (20 h). 3-Iodobenzonitrile (229 mg, 1.0 mmol), thiophenol (103 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-cyanophenyl phenyl sulfide (181 mg, 86% yield) as colorless liquid. Column

chromatography solvent (hexane/ethyl acetate = 25/1). R_f = 0.4 (hexane/ethyl acetate = 20/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.30-7.45 (m, 9 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 140.2, 133.6, 132.9, 132.3, 131.7, 130.0, 129.7, 129.6, 129.1, 118.5, 113.5. MS (EI) *m/z* (relative intensity) 211 (30), 185 (20), 134 (100).



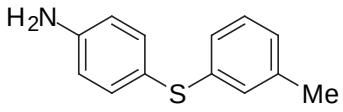
3-Bromophenyl phenyl sulfide[3] (Table 1, entry 3).

The general procedure was followed (20 h). 3-bromoiodobenzene (283 mg, 1.0 mmol), thiophenol (103 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-bromophenyl phenyl sulfide (240 mg, 91% yield) as colorless liquid. Column chromatography solvent (hexane). R_f = 0.6 (hexane). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.11-7.22 (m, 2 H), 7.28-7.40 (m, 7 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 139.2, 134.1, 132.5, 132.4, 130.6, 129.8, 129.7, 128.6, 128.2, 123.2. MS (EI) *m/z* (relative intensity) 266 (40), 264 (40).



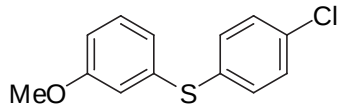
3-Nitrophenyl 3-tolyl sulfide[2] (Table 1, entry 4).

The general procedure was followed (22 h). 3-Nitroiodobenzene (249 mg, 1.0 mmol), *m*-thiocresol (119 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-nitrophenyl 3-tolyl sulfide (208 mg, 85% yield) as yellow liquid. Column chromatographic solvent (hexane/ethyl acetate = 20/1). R_f = 0.4 (hexane/ethyl acetate = 20/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.95-7.99 (m, 2 H), 7.46 (dt, 1 H, J = 7.5 Hz, 0.9 Hz), 7.38 (t, 1 H, J = 8.1 Hz), 7.12-7.28 (m, 5 H), 2.36 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 141.0, 140.0, 134.3, 134.2, 131.7, 130.8, 130.0, 129.9, 129.8, 123.1, 122.9, 120.9. MS (EI) *m/z* (relative intensity) 245 (100), 184 (80).



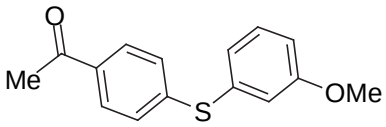
4-(3-Tolyl)sulfanylaniline (Table 1, entry 5).[4]

The general procedure was followed (22 h). 4-Iodoaniline (219 mg, 1.0 mmol), *m*-thiocresol (119 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 4-(3-tolyl)sulfanylaniline (194 mg, 90% yield) as pale yellow liquid. Column chromatographic solvent (hexane/ethyl acetate = 4/1). R_f = 0.4 (hexane/ethyl acetate = 4/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.28 (dt, 2 H, *J* = 7.5 Hz, 0.9 Hz), 7.08 (t, 1 H, *J* = 7.8 Hz), 6.91 (t, 3 H, *J* = 7.5 Hz), 3.71 (brs, 2 H), 2.26 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 138.8, 138.1, 136.2, 128.9, 128.1, 126.4, 124.6, 117.5, 116.1, 100.0, 21.8. IR (neat, cm^{-1}) 3460 (broad), 3377 (broad), 3211, 3051, 3027, 2919. MS (EI) *m/z* (relative intensity) 219 (100), 92 (80). HRMS (EI), Cald. for $C_{13}H_{13}NS$ 215.0769; Found 215.0766.



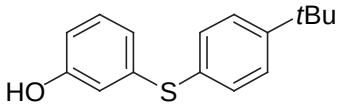
3-Methoxyphenyl 4-chlorophenyl sulfide (Table 1, entry 6).

The general procedure was followed (22 h). 3-Iodoanisole (234 mg, 1.0 mmol), 4-chlorothiophenol (145 mg, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-methoxyphenyl 4-chlorophenyl sulfide (203 mg, 81% yield) as colorless liquid. Column chromatographic solvent (hexane/ethyl acetate = 50/1). R_f = 0.4 (hexane/ethyl acetate = 40/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.13-7.26 (m, 2 H), 6.88 (ddd, 2 H, *J* = 1.2 Hz, 1.8 Hz, 7.8 Hz), 6.83 (t, 2 H, *J* = 1.5 Hz), 6.78 (ddd, 2 H, *J* = 0.9 Hz, 2.7 Hz, 8.1 Hz), 3.75 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.2, 136.6, 134.3, 133.4, 132.5, 130.3, 129.5, 123.4, 116.4, 113.3, 55.7. IR (neat, cm^{-1}) 3065, 3002, 2962, 2949, 2820. MS (EI) *m/z* (relative intensity) 252 (30), 250 (100). Anal Cald for $C_{13}H_{11}ClOS$, Cald. C: 62.27, H: 4.42; Found C: 62.58, H: 4.46.



4-(3-Methoxyphenyl)sulfanylacetophenone (Table 1, entry 7).

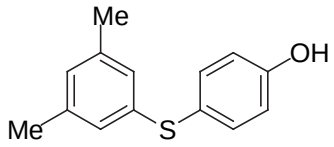
The general procedure was followed (22 h). 3-Iodoacetophenone (246 mg, 1.0 mmol), 3-methoxythiophenol (124 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 4-(3-methoxyphenyl)sulfanylacetophenone (209 mg, 81% yield) as pale yellow liquid. Column chromatographic solvent (hexane/ethyl acetate = 10/1). R_f = 0.3 (hexane/ethyl acetate = 10/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.80 (dd, 2 H, *J* = 6.6 Hz, 2.1 Hz), 7.29 (t, 1 H, *J* = 7.8 Hz), 7.20-7.24 (m, 2 H), 7.03-7.07 (m, 1 H), 7.00 (m, 1 H), 6.90 (ddd, 1 H, *J* = 8.4 Hz, 2.7 Hz, 1.2 Hz), 3.79 (s, 3 H), 2.55 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 196.2, 160.4, 144.7, 134.7, 133.4, 130.6, 129.1, 127.8, 126.0, 118.6, 114.9, 55.7, 26.9. IR (neat, cm^{-1}) 3064, 3002, 2962, 2939, 2836, 1698. MS (EI) *m/z* (relative intensity) 258 (80), 243 (100). Anal Cald for $C_{14}H_{15}O_2S$, Cald. C: 69.74, H: 5.46; Found C: 69.47, H: 5.39.



4-(3,5-Dimethylphenyl)sulfanylphenol (Table 1, entry 8).

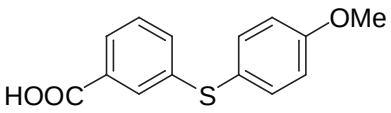
The general procedure was followed (22 h). 5-Iodo-*m*-xylene (144 μL, 1.0 mmol), 4-mercaptophenol (126 mg, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 4-(3,5-dimethylphenyl)sulfanylphenol (207 mg, 90% yield) as colorless liquid. Workup procedure: ethyl acetate (approx. 5 mL) and dodecane (227 μL, GC standard) were added to the reaction mixture after the reaction was completed. The organic layer was neutralized by dilute HCl to pH 8. The aqueous layer was extracted by ethyl acetate (4 × 10 mL). The combined organic layers were concentrated and purified by column chromatography on silica gel using hexane/ethyl acetate = 10/1 as the eluent to afford the titled product. R_f = 0.3 (hexane/ethyl acetate = 10/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ

7.33 (dt, 2 H, *J* = 1.8 Hz, 8.4 Hz), 6.77-6.81 (m, 5 H), 4.83 (s, 1 H), 2.22 (s, 6 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.6, 138.8, 137.7, 135.4, 128.1, 126.4, 125.3, 116.6, 21.6. IR (neat, cm^{-1}) 3371 (broad), 3031, 2917, 2860. MS (EI) *m/z* (relative intensity) 230 (100), 215 (20). Anal Cald for $C_{14}H_{14}OS$, Cald. C: 73.01, H: 6.13; Found C: 73.21, H: 6.11.



3-(4-*tert*-Butylphenyl)sulfanylphenol (Table 1, entry 9).

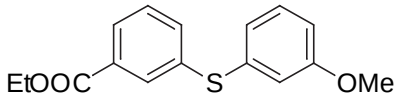
The general procedure was followed (22 h). 3-Iodophenol (220 mg, 1.0 mmol), 4-*tert*-butylthiophenol (168 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-(4-*tert*-butylphenyl)sulfanylphenol (232 mg, 90% yield) as the colorless oil. Workup procedure: ethyl acetate (approx. 5 mL) and dodecane (227 µL, GC standard) were added to the reaction mixture after the reaction was completed. The organic layer was neutralized by dilute HCl to pH 8. The aqueous layer was extracted by ethyl acetate (4 × 10 mL). The combined organic layers were concentrated and purified by column chromatography on silica gel using hexane/ethyl acetate = 10/1 as the eluent to afford the titled product. R_f = 0.2 (hexane/ethyl acetate = 10/1) (Note: same R_f value as the starting material). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.21-7.34 (m, 2 H), 7.14 (t, 1 H, *J* = 8.1 Hz), 6.95 (t, 1 H, *J* = 8.1 Hz), 6.84-6.88 (m, 1 H), 6.78-6.81 (m, 1 H), 6.64-6.71 (m, 2 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.0, 132.5, 131.2, 130.3, 130.1, 124.8, 122.1, 116.3, 115.2, 113.8, 35.0, 31.7. IR (neat, cm^{-1}) 3375 (broad), 2962, 2904, 2867. MS (EI) *m/z* (relative intensity) 258 (100). HRMS (EI), Cald. for $C_{16}H_{18}OS$, 258.1073; Found 258.1068.



3-(4-Methoxyphenyl)sulfanylbenzoic acid (Table 1, entry 10).

Cu(I) iodide (10 mg, 0.05 mmol), potassium carbonate (414 mg, 3.0 mmol) and 3-iodobenzoic acid (248 mg, 1.0 mmol) were charged into a screw-capped test tube with

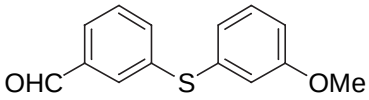
Teflon-lining. The tube was evacuated and backfilled with argon (3 cycles). 2-Propanol (1.0 mL, bench grade solvent without degassing and pre-drying), ethylene glycol (111μL, 2.0 mmol, bench grade solvent) and 4-methoxythiophenol (123 μL, 1.0 mmol) were added by syringes at room temperature. The tube was heated to 80 ˚C and stirred for 24 hours. The reaction mixture was then allowed to reach room temperature. Ethyl acetate (~5 mL), water (~10 mL) and dil. HCl were added to reach pH 3-4. The reaction mixture was extracted with ethyl acetate (2 × 10 mL) and CH_2Cl_2 (2 × 10 mL). The combined organic phase was passed through a short pad of silica (0.5 cm diameter × 1 cm height). Solvent was removed and the yellow residue was redissolved in minimum amount of CH_2Cl_2. Hexane was added slowly and the solution was stand overnight at room temperature. White crystal was obtained as the titled product (221 mg, 85% yield). R_f = 0.2 (hexane/ethyl acetate = 2/1) (Note: same R_f value as the starting material). Melting point; 121-123 ˚C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 10.68 (brs, 1 H), 7.86 (s, 1 H), 7.82 (dt, 1 H, *J* = 1.8 Hz, 6.6 Hz), 7.42 (dt, 2 H, *J* = 2.1 Hz, 8.7 Hz), 7.28-7.31 (m, 2 H), 6.90 (dt, 2 H, *J* = 2.1 Hz, 8.7 Hz), 3.83 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 172.0, 160.3, 140.3, 136.1, 132.8, 130.2, 129.2, 129.1, 127.4, 123.1, 55.7. IR (neat, cm^{-1}) 2964 (broad), 2943, 2902, 2875, 2856, 2840, 2813, 2360, 2342, 1688. MS (EI) *m/z* (relative intensity) 260 (100). Anal. Cald. for $C_{14}H_{12}O_3S$, Cald. C: 64.60, H: 4.65; Found C: 64.52, H: 4.68.



Methyl 3-(3-methoxyphenyl)sulfanylbenzoate (Table 1, entry 11).

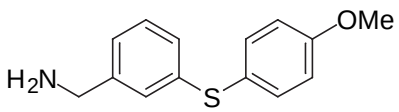
Cu(I) iodide (10 mg, 0.05 mmol), potassium carbonate (276 mg, 2.0 mmol) and Methyl 3-iodobenzoate (276 mg, 1.0 mmol) were charged into a screw-capped test tube with Teflon-lining. The tube was evacuated and backfilled with argon (3 cycles). Anhydrous DME (1.0 mL) and 3-methoxythiophenol (124 μL, 1.0 mmol) were added by syringes at room temperature. The tube was heated to 80 ˚C and stirred for 22 hours. The reaction mixture was then allowed to reach room temperature. Ethyl acetate (approx. 5 mL) and dodecane (227 μL, GC standard) were added. The aliquot was analyzed GC. The reaction mixture was then filtered and concentrated. The crude product was purified by

column chromatography on silica gel using hexane/ethyl acetate = 20/1 to afford colorless liquid as the titled product (220 mg, 81% yield). R_f = 0.5 (hexane/ethyl acetate = 10/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.02-8.03 (m, 1 H), 7.89 (dt, 1 H, *J* = 1.2 Hz, 8.1 Hz), 7.45-7.49 (m, 1 H), 7.34 (t, 1 H, *J* = 7.5 Hz), 7.21 (t, 1 H, *J* = 8.1 Hz), 6.86-6.92 (m, 2 H), 6.79 (ddd, 1 H, J = 0.6 Hz, 2.4 Hz, 8.1 Hz), 4.34 (q, 2 H, *J* = 7.2 Hz), 3.76 (s, 3 H), 1.37 (t, 3 H, J = 7.2 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 166.0, 160.2, 136.5, 136.3, 135.2, 132.0, 131.7, 130.3, 129.3, 128.4, 123.7, 116.6, 113.5, 61.5, 55.7, 14.7. IR (neat, cm^{-1}) 3064, 2981, 2960, 2937, 2360, 2342, 1717. MS (EI) *m/z* (relative intensity) 288 (100), 243 (30). Anal. Cald. for $C_{16}H_{16}O_3S$, Cald. C: 66.64, H: 5.59; Found C: 66.87, H: 5.66.



3-(3-Methoxyphenyl)sulfanylbenzaldehyde (Table 1, entry 12).

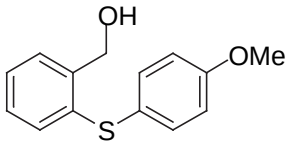
The general procedure was followed (22 h). 3-Iodobenzaldehyde (232 mg, 1.0 mmol), 3-methoxythiophenol (124 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-(3-methoxyphenyl)sulfanylbenzaldehyde (205 mg, 85% yield) as colorless liquid. Column chromatographic solvent (hexane/ethyl acetate = 15/1). R_f = 0.4 (hexane/ethyl acetate = 10/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.91 (s, 1 H), 7.76-7.77 (m, 1 H), 7.71 (dt, 1 H, *J* = 1.2 Hz, 7.2 Hz), 7.45-7.53 (m, 1 H), 7.42 (t, 1 H, *J* = 7.5 Hz), 7.25 (t, 1 H, *J* = 7.5 Hz), 6.91-6.98 (m, 2 H), 6.84 (ddd, 1 H, *J* = 1.2 Hz, 2.4 Hz, 8.1 Hz), 3.77 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 191.7, 160.3, 138.4, 137.3, 135.8, 135.0, 131.1, 130.5, 129.9, 127.9, 124.6, 117.6, 114.0, 55.7. IR (neat, cm^{-1}) 3060, 3006, 2960, 2937, 2834, 2726, 1698. MS (EI) *m/z* (relative intensity) 244 (100), 227 (30), 211 (40). Anal. Cald. for $C_{14}H_{12}O_2S$, Cald. C: 68.83, H: 4.95; Found C: 69.04, H: 4.94.



3-(4-Methoxyphenyl)sulfanylbenzylamine (Table 1, entry 13).

The general procedure was followed (22 h). 3-Iodobenzylamine (133 μL, 1.0 mmol), 4-methoxythiophenol (123 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-(4-methoxyphenyl)sulfanylbenzylamine (218 mg, 89% yield) as light yellow liquid. Column chromatographic solvent (CH_2Cl_2 (saturated with NH_3)/MeOH = 30/1). R_f = 0.4 (CH_2Cl_2 (saturated with NH_3)/MeOH = 30/1), (Note: same R_f value as the starting material). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.40 (dt, 2 H, *J* = 1.8 Hz, 8.7 Hz), 7.19 (t, 1 H, *J* = 7.8 Hz), 7.06-7.13 (m, 2 H), 7.01 (dt, 1 H, *J* = 1.5 Hz, 7.5 Hz), 6.89 (dt, 2 H, *J* = 2.1 Hz, 8.7 Hz), 3.82 (s, 3 H), 3.79 (s, 2 H), 1.40 (brs, 2 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.9, 144.3, 139.0, 135.6, 129.3, 126.9, 126.7, 124.8, 124.3, 115.2, 55.7, 46.6. IR (neat, cm^{-1}) 3373 (broad), 3072, 3060, 2939, 2836. MS (EI) *m/z* (relative intensity) 245 (100), 106 (60). HRMS (EI), Cald. for $C_{14}H_{15}NOS$ 245.0869; Found 245.0862.

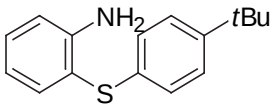
Characterization data for product shown in Table 2



2-(4-Methoxyphenyl)sulfanylbenzylalcohol (Table 2, entry 1).

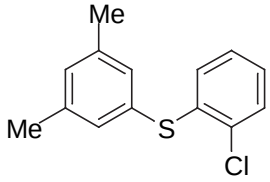
The general procedure was followed (20 h). 2-Iodobenzylalcohol (234 mg, 1.0 mmol), 4-methoxythiophenol (123 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 2-(4-methoxyphenyl)sulfanylbenzylalcohol (219 mg, 89% yield) as colorless liquid. Column chromatographic solvent (hexane/ethyl acetate = 5/1). R_f = 0.3 (hexane/ethyl acetate = 5/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.40 (dd, 1 H, *J* = 6.6 Hz, 1.5 Hz), 7.29 (dt, 2 H, *J* = 8.7 Hz, 2.1 Hz), 7.13-7.21 (m, 2 H), 7.08 (dd, 1 H, *J* = 8.1 Hz, 2.1 Hz), 6.86 (dt, 2 H, J = 9.0 Hz, 2.1 Hz), 4.78 (d, 2 H, *J* = 6.3 Hz), 3.80 (s, 3 H), 2.12 (t, 1 H, *J* = 6.3 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.7, 140.0, 135.9, 134.3, 130.8, 128.6, 128.5, 127.1, 124.5, 115.3, 63.9, 55.7. IR (neat, cm^{-1}) 3365 (broad), 3015, 2962, 2904, 2867. MS (EI)

m/z (relative intensity) 246 (100), 138 (40), 108 (70). HRMS (EI), Cald. for $C_{14}H_{14}O_2S$ 246.0709; Found 246.0707.



2-(4-*tert*-Butylphenyl)sulfanylaniline (Table2, entry 2).

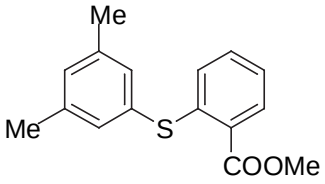
The general procedure was followed (22 h). 2-Iodoaniline (219 mg, 1.0 mmol), 4-*tert*-butylthiophenol (166 mg, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 2-(4-*tert*-butylphenyl)sulfanylaniline (231 mg, 90% yield) as light yellow liquid. Column chromatographic solvent (hexane/ethyl acetate = 20/1). R_f = 0.4 (hexane/ethyl acetate = 10/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.42 (dd, 1 H, *J* = 1.5 Hz, 7.5 Hz), 7.17-7.24 (m, 4 H), 7.00 (dt, 2 H, *J* = 1.8 Hz, 8.4 Hz), 6.70-6.78 (m, 2 H), 4.28 (brs, 2 H), 1.26 (s, 9 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 148.8, 148.7, 137.5, 133.3, 131.1, 126.5, 126.3, 118.9, 115.5, 115.0, 34.8, 31.7. IR (neat, cm^{-1}) 3471, 3375, 3072, 3062, 3020, 2962, 2902, 2867. MS (EI) *m/z* (relative intensity) 257 (70), 242 (100). Anal. Cald. for $C_{16}H_{19}NS$, Cald. C: 74.66, H: 7.44; Found C: 74.64, H: 7.30.



3,5-Dimethylphenyl 2-chlorophenyl sulfide[5] (Table 2, entry 3).

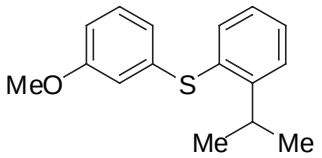
The general procedure was followed (22 h). 5-Iodo-*m*-xylene (144 µL, 1.0 mmol), 2-chlorothiophenol (145 mg, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3,5-dimethylphenyl 2-chlorophenyl sulfide (215 mg, 87% yield) as colorless liquid. Column chromatographic solvent (hexane). R_f = 0.5 (hexane/ethyl acetate = 50/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.32-7.36 (m, 1 H), 7.05-7.10 (m, 4 H), 6.97 (s, 1 H), 6.88-6.92 (m, 1 H), 2.30 (s, 6 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 139.5, 137.4, 132.7, 131.7,

131.5, 130.6, 129.8, 129.6, 127.3, 126.9, 21.6. IR (neat, cm^{-1}) 3060, 3037, 2950, 2917, 2860. MS (EI) *m/z* (relative intensity) 250 (30), 248 (100).



Methyl 2-(3,5-dimethylphenyl)sulfanylbenzoate (Table 2, entry 4).

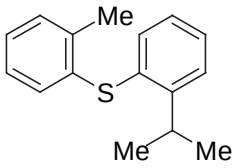
The general procedure was followed (22 h). 5-Iodo-*m*-xylene (144 µL, 1.0 mmol), Methyl thiosalicylate (138 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), and DME (1.0 mL) were used to obtain the methyl 2-(3,5-dimethylphenyl)sulfanylbenzoate (236 mg, 86% yield) as colorless liquid. Column chromatographic solvent (hexane/ethyl acetate = 20/1). R_f = 0.5 (hexane/ethyl acetate = 10/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.95 (dd, 1 H, *J* = 1.8 Hz, 8.1 Hz), 7.19-7.25 (m, 1 H), 7.17 (s, 2 H), 7.08 (dt, 1 H, *J* = 1.2 Hz, 7.8 Hz), 7.03 (s, 1 H), 6.81 (dd, 1 H, *J* = 0.9 Hz, 8.1 Hz), 3.94 (s, 3 H), 2.32 (s, 6 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.0, 144.0, 139.6, 133.4, 132.5, 131.2, 127.4, 126.5, 124.2, 117.1, 52.5, 21.6. IR (neat, cm^{-1}) 2950, 2916, 1712, 1711. MS (EI) *m/z* (relative intensity) 272 (100), 197 (70). HRMS (EI), Cald. for $C_{16}H_{16}O_2S$ 272.0866; Found 272.0858.



3-(2-Isopropylphenyl)sulfanylanisole (Table 2, entry 5).

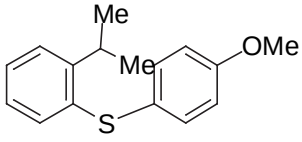
The general procedure was followed (22 h). 3-Iodoanisole (234 mg, 1.0 mmol), 2-Isopropylbenzenethiol (90% purity, 168 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-(2-isopropylphenyl)sulfanylanisole (241 mg, 93% yield) as colorless liquid. Column chromatographic solvent (hexane/ethyl acetate = 40/1). R_f = 0.3 (hexane/ethyl acetate = 40/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.29-7.36 (m, 3 H), 7.09-7.17 (m, 2 H), 6.67-6.73 (m, 3 H), 3.72 (s, 3 H), 3.54 (hept, 1 H, *J* = 6.9 Hz), 1.22 (s, 3

H), 1.19 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.1, 150.9, 139.0, 134.5, 132.1, 129.9, 128.9, 126.8, 126.3, 121.4, 114.4, 111.9, 55.6, 31.1, 24.0. IR (neat, cm^{-1}) 3060, 2962, 2867, 2834, 2362, 2343. MS (EI) *m/z* (relative intensity) 258 (100), 241 (30), 225 (30). HRMS (EI), Cald. for $C_{16}H_{18}OS$ 258.1078; Found 258.1080.



2-Tolyl 2-Isopropylphenyl sulfide (Table 2, entry 6).

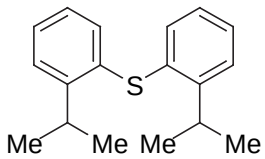
The general procedure was followed (22 h). 2-Iodotoluene (218 mg, 1.0 mmol), 2-Isopropylbenzenethiol (90% purity, 168 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 2-tolyl 2-Isopropylphenyl sulfide (213 mg, 88% yield) as colorless liquid. Column chromatographic solvent (hexane). R_f = 0.4 (hexane). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.32 (d, 1 H, *J* = 7.5 Hz), 7.18-7.26 (m, 2 H), 7.13 (dd, 1 H, *J* = 1.5 Hz, 7.2 Hz), 7.09-7.11 (m, 1 H), 7.01-7.08 (m, 2 H), 7.00 (dd, 1 H, *J* = 1.5 Hz, 7.2 Hz), 3.49 (hept, 1 H, *J* = 6.6 Hz), 2.37 (s, 3 H), 1.25 (s, 3 H), 1.22 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 149.6, 138.6, 135.5, 133.1, 132.2, 131.1, 130.6, 127.9, 127.0, 126.8, 126.7, 126.1, 30.9, 23.9, 20.9. IR (neat, cm^{-1}) 3060, 3012, 2962, 2867. MS (EI) *m/z* (relative intensity) 242 (100), 225 (80). Anal. Cald. for $C_{16}H_{18}S$, Cald. C: 79.29, H: 7.49; Found C: 79.33, H: 7.62.



4-(2-Isopropylphenyl)sulfanylanisole[5] (Table 2, entry 7).

Cu(I) iodide (38 mg, 0.2 mmol) and potassium carbonate (276 mg, 2.0 mmol) were charged into a screw-capped test tube with Teflon-lined septum. The tube was evacuated and backfilled with argon (3 cycles). *tert*-Amyl alcohol (2-methyl-2-butanol) (1.0 mL, bench grade solvent without degassing and pre-drying), ethylene glycol (111µL, 2.0 mmol, bench grade solvent), 2-isopropyliodobenzene (246 mg, 1.0 mmol) and 4-

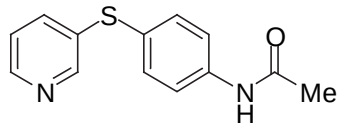
methoxythiolphenol (147 μL, 1.2 mmol) were added by syringes at room temperature. The tube was heated to 100 ˚C and stirred for 24 hours. The reaction mixture was then allowed to reach room temperature. Ethyl acetate (approx. 5 mL) and dodecane (227 μL, GC standard) were added. The aliquot was analyzed GC. The reaction mixture was then filtered and concentrated. The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate = 40/1 as eluent to afford white solid as the titled product (241 mg, 94% yield). R_f = 0.6 (hexane/ethyl acetate = 20/1). Melting point; 63-65 ˚C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.32 (dt, 2 H, *J* = 2.1 Hz, 8.7 Hz), 7.25-7.28 (m, 1 H), 7.19 (dt, 1 H, *J* = 2.1 Hz, 8.1 Hz), 7.03-7.06 (m, 2 H), 6.88 (dt, 1 H, *J* = 2.4 Hz, 9.0 Hz), 3.82 (s, 3 H), 3.53 (hept, 1 H, *J* = 6.9 Hz), 1.27 (s, 3 H), 1.25 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.5, 147.8, 135.9, 134.5, 130.2, 126.9, 126.5, 125.7, 125.6, 115.2, 55.7, 30.7, 23.7. IR (neat, cm^{-1}) 3071, 3068, 3011, 2952, 2857. MS (EI) *m/z* (relative intensity) 258 (100), 241 (20), 149 (30). Anal. Cald. for $C_{16}H_{18}OS$, Cald. C: 74.38, H: 7.02; Found C: 74.57, H: 7.04.



Di(2-isopropylphenyl) sulfide (Table 2, entry 8).

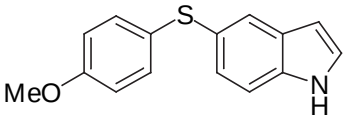
Cu(I) iodide (38 mg, 0.2 mmol) and potassium carbonate (276 mg, 2.0 mmol) were charged into a screw-capped test tube with Teflon-lined septum. The tube was evacuated and backfilled with argon (3 cycles). *tert*-Amyl alcohol (2-methyl-2-butanol) (1.0 mL, bench grade solvent without degassing and pre-drying), ethylene glycol (111μL, 2.0 mmol, bench grade solvent), 2-isopropyliodobenzene (246 mg, 1.0 mmol) and 2-isopropylbenzenethiol (90% purity, 202 μL, 1.2 mmol) were added by syringes at room temperature. The tube was heated to 100 ˚C and stirred for 24 hours. The reaction mixture was then allowed to reach room temperature. Ethyl acetate (approx. 5 mL) and dodecane (227 μL, GC standard) were added. The aliquot was analyzed GC. The reaction mixture was then filtered and concentrated. The crude product was purified by column chromatography on silica gel using hexane as eluent to afford colorless oil as the titled product (245 mg, 91% yield). R_f = 0.5 (hexane). ^{1}H NMR ($CDCl_3$, 300 MHz) δ

7.30 (d, 2 H, J = 7.2 Hz), 7.18-7.24 (m, 2 H), 7.03-7.05 (m, 4 H), 3.50 (hept, 2 H, J = 6.9 Hz), 1.25 (s, 3 H), 1.23 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 149.2, 134.2, 132.1, 127.6, 126.7, 125.9, 30.9, 23.8. IR (neat, cm^{-1}) 3058, 2962, 2929, 2867. MS (EI) *m/z* (relative intensity) 270 (100), 211 (90). HRMS (EI), Cald. for $C_{18}H_{22}S$ 270.1442; Found 270.1445.



3-(4-Acetamidophenyl)sulfanylpyridine (Table 2, entry 9).

The general procedure was followed (20 h). 3-Iodopyridine (205 mg, 1.0 mmol), 4-acetamidothiophenol (167 mg, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 3-(4-acetamidophenyl)sulfanylpyridine (202 mg, 83% yield) as white solid. Column chromatographic solvent (ethyl acetate). R_f = 0.4 (ethyl acetate). Melting point; 138-140 ˚C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.42 (d, 1 H, *J* = 1.8 Hz), 8.38 (dd, 1 H, *J* = 0.9 Hz, 4.5 Hz), 8.29 (s, 1 H), 7.52 (d, 2 H, *J* = 8.7 Hz), 7.35 (d, 2 H, *J* = 8.4 Hz), 7.18 (dd, 1 H, *J* = 3.0 Hz, 7.8 Hz), 2.17 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.0, 149.5, 147.1, 138.9, 136.8, 135.3, 134.0, 127.3, 124.2, 120.9, 24.9. IR (neat, cm^{-1}) 3286, 3245, 3176, 3105, 2360, 2342, 1650. MS (EI) *m/z* (relative intensity) 244 (100), 202 (100). Anal. Cald. for $C_{13}H_{12}N_2OS$, Cald. C: 63.91, H: 4.95; Found C: 63.80, H: 4.93.

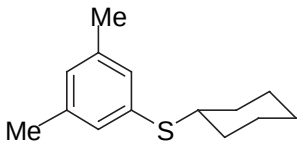


5-(4-Methoxyphenyl)sulfanylindole (Table 2, entry 10).

The general procedure was followed (20 h). 5-Iodoindole (243 mg, 1.0 mmol), 4-methoxythiophenol (123 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 5-(4-methoxyphenyl)sulfanylindole (228 mg, 90% yield) as white solid. Column chromatographic solvent (hexane/ethyl acetate = 5/1). R_f = 0.3 (hexane/ethyl acetate = 5/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.14 (brs, 1 H), 7.69 (s, 1 H), 7.30 (d, 1 H, *J* = 8.4

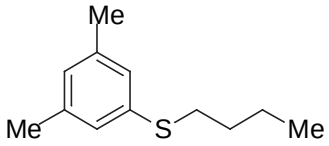
Hz), 7.12-7.28 (m, 4 H), 6.79 (dt, 2 H, *J* = 2.1 Hz, 9.0 Hz), 6.47-6.49 (m, 1 H), 3.76 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 158.7, 135.2, 132.1, 128.9, 128.8, 126.5, 126.2, 125.2, 124.9, 114.8, 112.1, 102.9, 55.7. IR (neat, cm^{-1}) 3417 (broad), 2958, 2939, 2834. MS (EI) *m/z* (relative intensity) 255 (100), 223 (15). HRMS (EI), Cald. for $C_{15}H_{13}NOS$ 255.0712; Found 255.0702. Anal. Cald. for $C_{15}H_{13}NOS$, Cald. C: 70.56, H: 5.13; Found C: 70.37, H: 5.09.

Characterization data for product shown in Table 3



Cyclohexyl 3,5-dimethylphenyl sulfide (Table 3, entry 1)

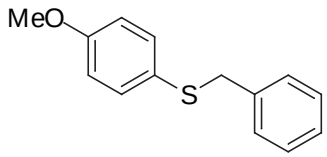
The general procedure was followed (20 h). 5-Iodo-*m*-xylene (144 µL, 1.0 mmol), cyclohexylmercaptan (122 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 µL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the cyclohexyl 3,5-dimethylphenyl sulfide (156 mg, 71% yield) as colorless oil. Column chromatographic solvent (hexane). R_f = 0.4 (hexane). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.99 (s, 2 H), 6.82 (s, 1 H), 3.02-3.10 (m, 1 H), 2.28 (s, 6 H), 1.96-2.00 (m, 2 H), 1.74-1.77 (m, 2 H), 1.56-1.63 (m, 1 H), 1.21-1.42 (m, 4 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 138.4, 134.7, 129.7, 128.7, 46.8, 33.8, 26.5, 26.2, 21.6. IR (neat, cm^{-1}) 3088, 3060, 3012, 2962, 2867. MS (EI) *m/z* (relative intensity) 220 (40), 138 (100), 105 (30). Anal Cald for $C_{14}H_{20}S$, Cald. C: 76.30, H: 9.15; Found C: 76.30, H: 9.17.



***n*-Butyl 3,5-dimethylphenyl sulfide (Table 3, entry 2).**

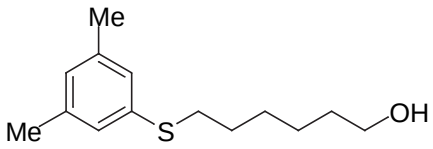
The general procedure was followed (20 h). 5-Iodo-*m*-xylene (144 µL, 1.0 mmol), 1-butanethiol (107 µL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol),

ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the *n*-butyl 3,5-dimethylphenyl sulfide (184 mg, 95% yield) as colorless oil. Column chromatographic solvent (hexane). R_f = 0.5 (hexane). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.92 (s, 2 H), 6.77 (s, 1 H), 2.89 (t, 2 H, *J* = 7.2 Hz), 2.27 (s, 6 H), 1.41-1.65 (m, 4 H), 0.92 (t, 3 H, *J* = 7.5 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 138.5, 136.7, 127.7, 126.6, 33.5, 31.6, 22.4, 21.7, 14.1. IR (neat, cm^{-1}) 3083, 3050, 3011, 2965, 2867. MS (EI) *m/z* (relative intensity) 194 (70), 138 (100). Anal Cald for $C_{12}H_{18}S$, Cald. C: 74.16, H: 9.34; Found C: 73.89, H: 9.32.



4-Benzylsulfanylanisole (Table 3, entry 3)

The general procedure was followed (20 h). 4-Iodoanisole (234 mg, 1.0 mmol), benzylmercaptan (117 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0 mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 4-benzylsulfanylanisole (206 mg, 90% yield) as colorless solid. Column chromatographic solvent (hexane/ethyl acetate = 50/1). Melting point; 48-50 ˚C. R_f = 0.3 (hexane/ethyl acetate = 40/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.14-7.24 (m, 7 H), 6.76 (dt, 2 H, *J* = 8.7 Hz, 2.1 Hz), 3.97 (s, 2 H), 3.76 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.3, 138.3, 134.3, 129.1, 128.5, 127.2, 126.2, 114.6, 55.6, 41.6. IR (neat, cm^{-1}) 3043, 3012, 2982, 2861. MS (EI) *m/z* (relative intensity) 230 (30), 91 (100). Anal Cald for $C_{14}H_{14}OS$, Cald. C: 73.01, H: 6.13; Found C: 72.86, H: 5.93.



6-(3,5-Dimethylphenyl)mercaptohexanol (Table 3, entry 4).

The general procedure was followed (20 h). 5-Iodo-*m*-xylene (144 μL, 1.0 mmol), 6-mercaptohexanol (137 μL, 1.0 mmol), CuI (10 mg, 0.05 mmol), K_2CO_3 (276 mg, 2.0

mmol), ethylene glycol (111 μL, 2.0 mmol) and 2-propanol (1.0 mL) were used to obtain the 6-(3,5-dimethylphenyl)mercaptohexanol (212 mg, 92% yield) as colorless oil. Column chromatographic solvent (hexane/ethyl acetate = 3/1). R_f = 0.4 (hexane/ethyl acetate = 2/1). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.92 (s, 2 H), 6.78 (s, 1 H), 3.62 (q, 2 H, *J* = 4.8 Hz), 2.90 (t, 2 H, *J* = 7.2 Hz), 2.27 (s, 6 H), 1.30-1.68 (m, 9 H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 138.6, 136.5, 127.8, 126.7, 63.2, 33.8, 33.0, 29.5, 28.9, 25.7, 21.7. IR (neat, cm^{-1}) 3320 (broad), 3060, 3022, 2972, 2847. MS (EI) *m/z* (relative intensity) 238 (40), 138 (100). Anal Cald for $C_{14}H_{22}OS$, Cald. C: 70.54, H: 9.30; Found C: 70.29, H: 9.33.

References:

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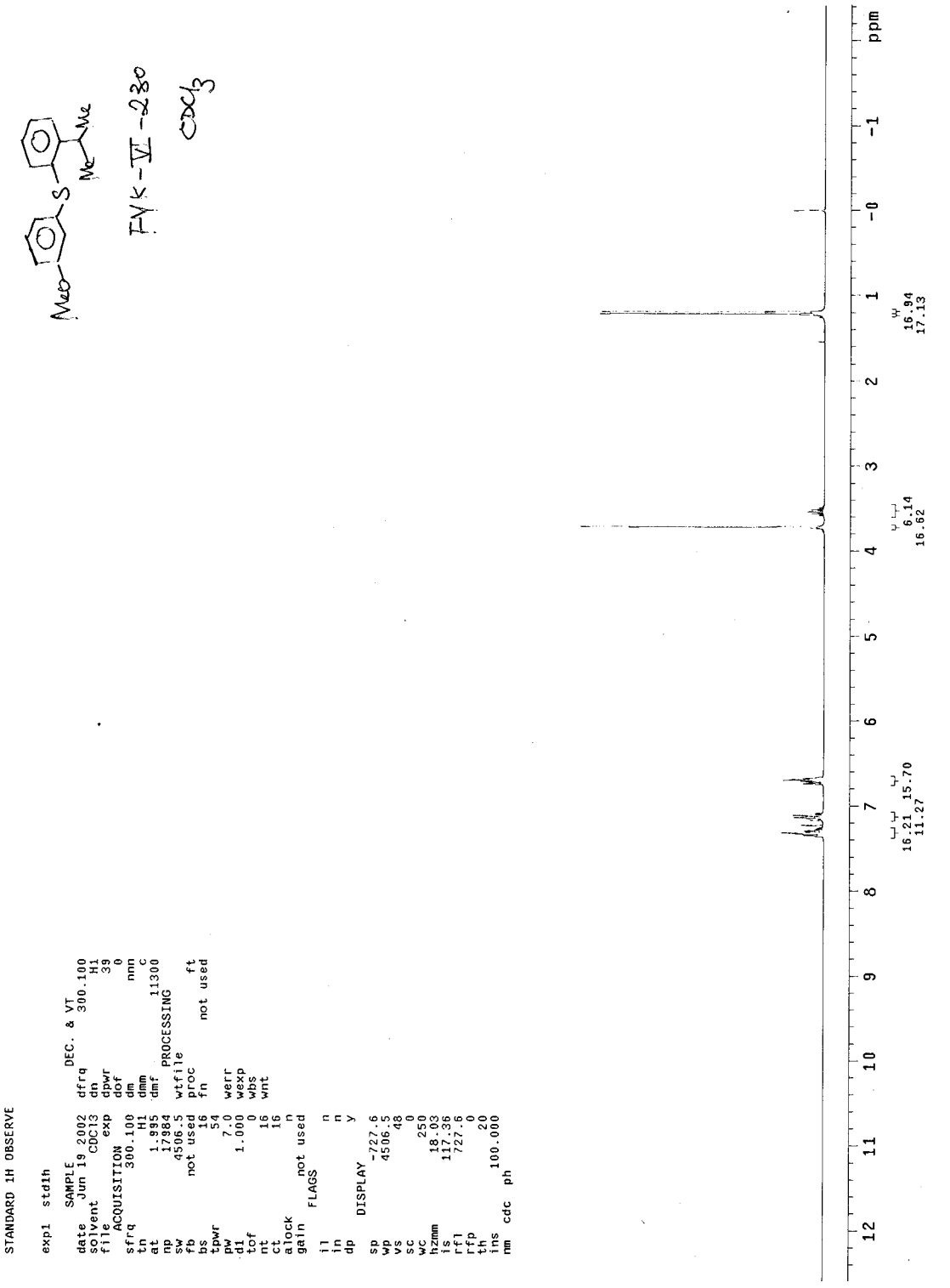
(2) Petrillo, G.; Novi, M.; Garbarino, G.; Dell'erba, C. *Tetrahedron* **1987**, *43*, 4625-4634.

(3) Barbero, M.; Degani, I.; Diulgheroff, N.; Dughera, S.; Fochi, R.; Migliaccio, M. *J. Org. Chem*. **2000**, *65*, 5600-5608.

(4) Commercially available from Solar, reg. 76590-36-8.

(5) Palomo, C.; Oiarbide, M.; López, R.; Gómez-Bengoa, E. *Tetrahedron Lett*. **2000**, *41*, 1283-1286.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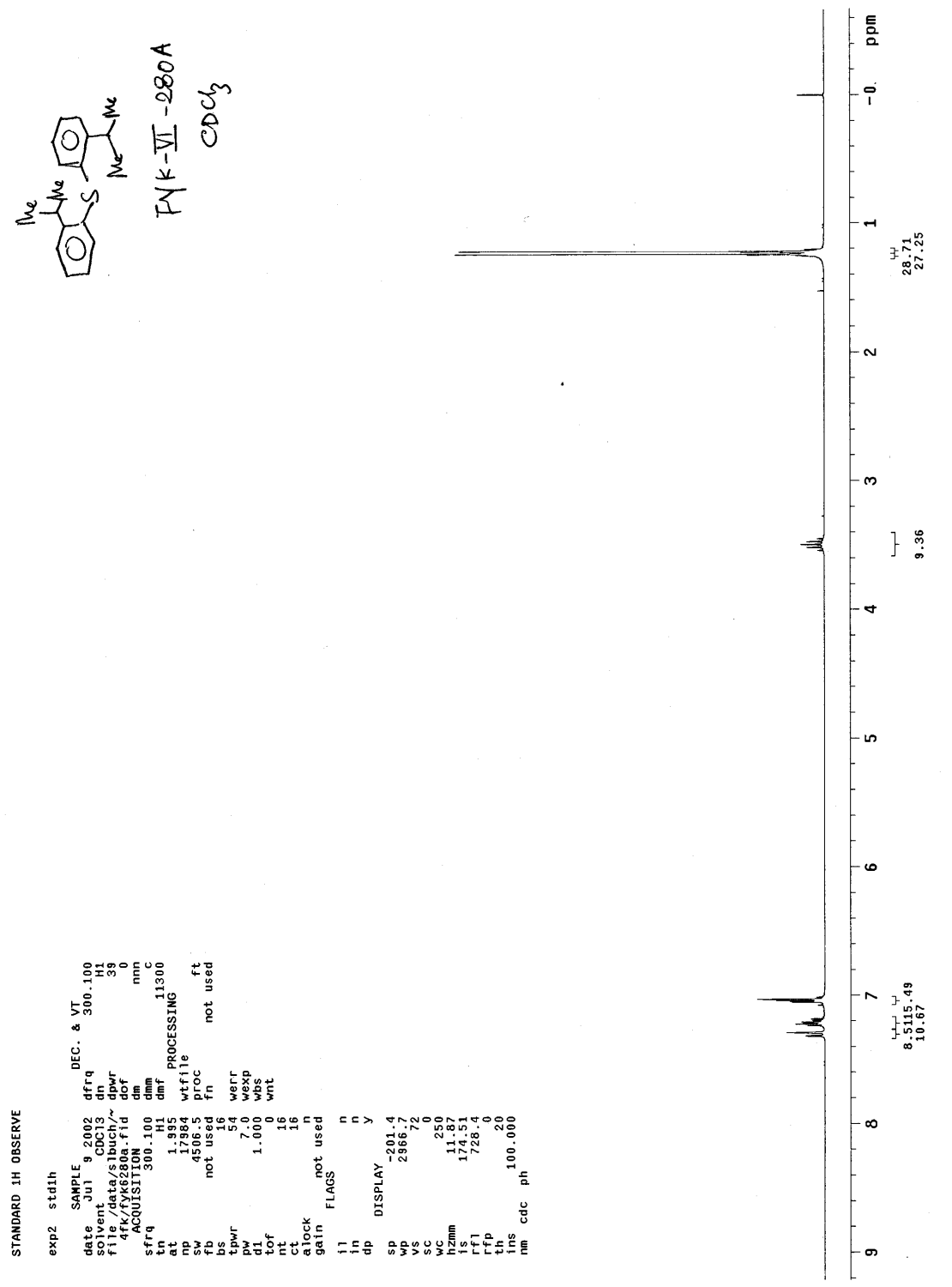
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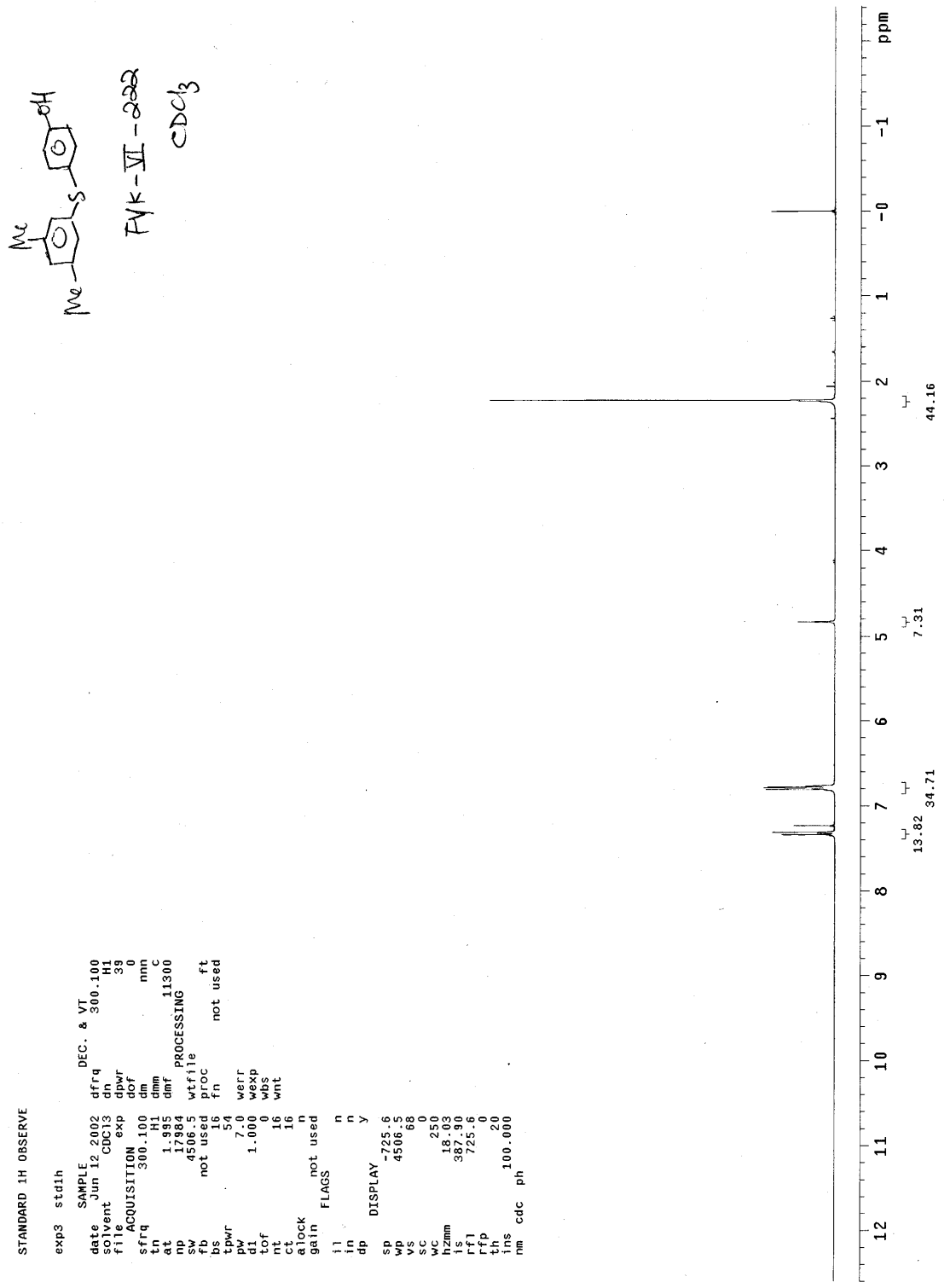
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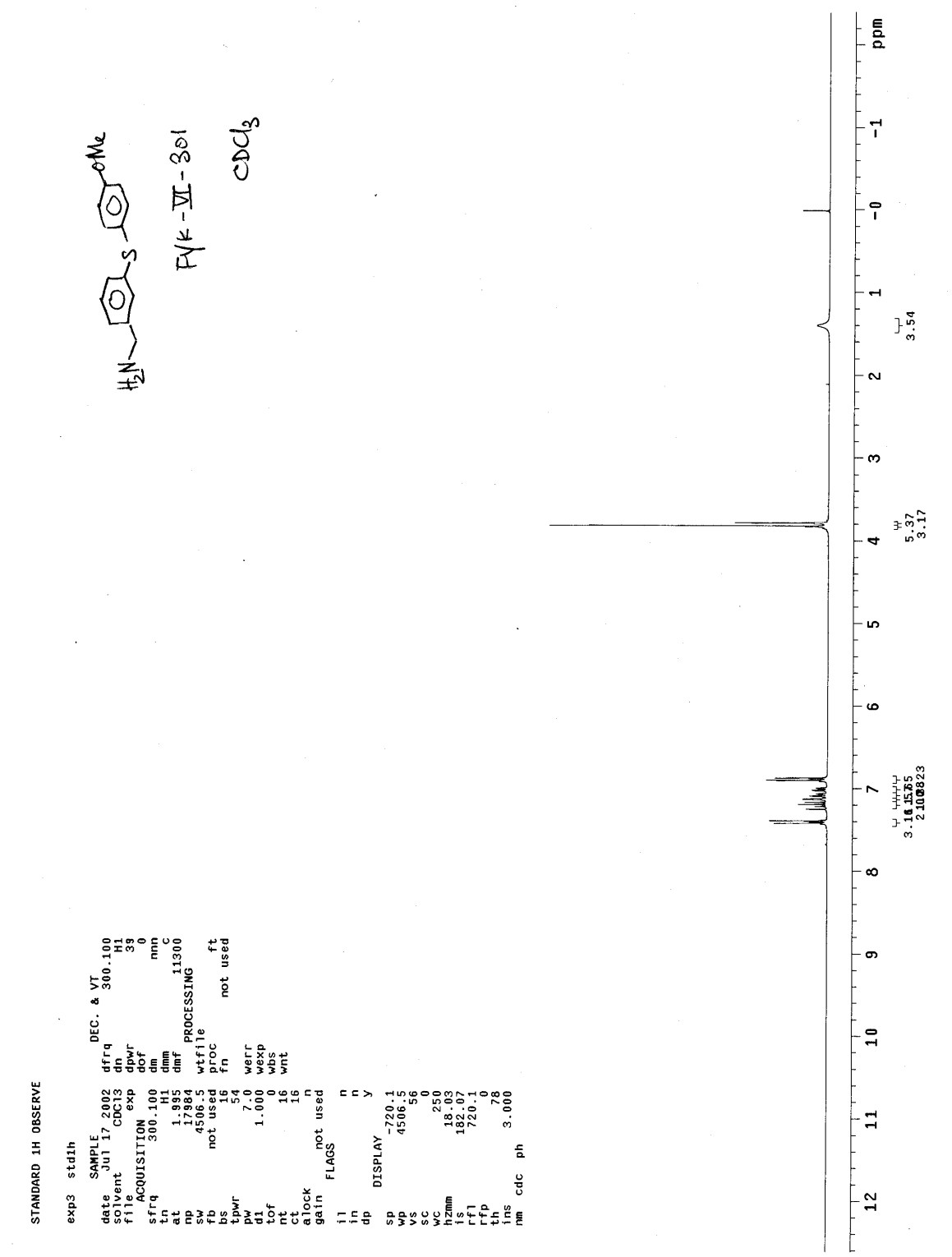
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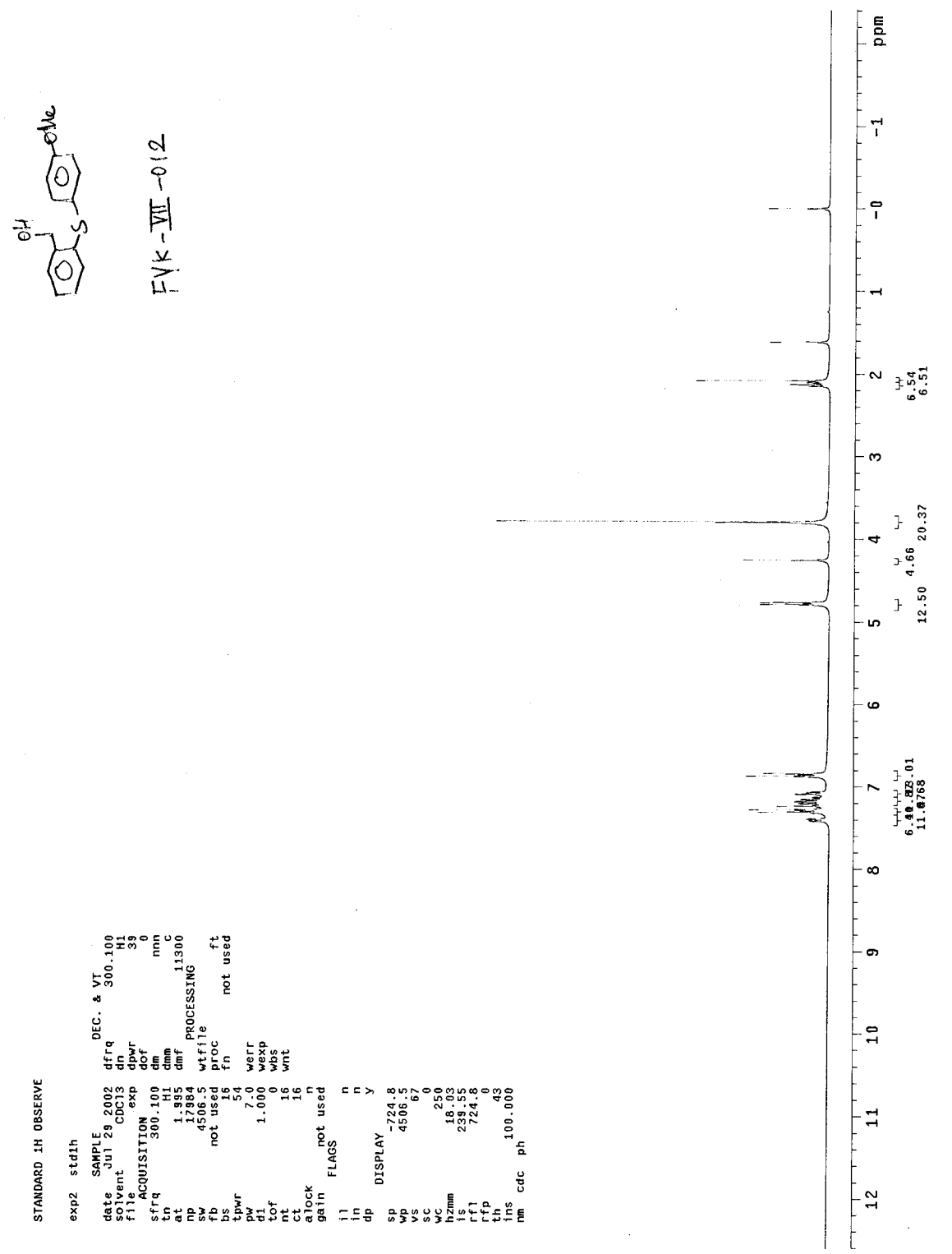
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