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One-Pot Synthesis of Unsymmetrical Diaryl Thioethers by Palladium-Catalyzed Coupling of Two Aryl Bromides and a Thiol Surrogate

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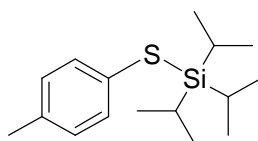
Experimental Part

General Considerations: All reactions were assembled under an inert atmosphere. Reactions were conducted in 4 mL vials sealed with a cap containing a PTFE septum. All glassware was oven-dried, evacuated and purged with nitrogen immediately prior to use. All reaction temperatures refer to bath temperatures. All common reagents were obtained from commercial suppliers and used without further purification. CyPF-*t*Bu (1-dicyclohexylphosphino-2-di-*tert*-butylphosphinoethylferrocene) was obtained from commercial suppliers and used without purification. Toluene was degassed by purging with nitrogen for 45 min and dried with a solvent purification system containing a 1 m column of activated alumina. 1,2-Dimethoxyethane (DME, 99.9% purity, HPLC grade) was used without further purification, but was stored under nitrogen. All other chemicals were used as received from commercial sources. ^1H and ^{13}C spectra were recorded in CDCl_3 on 400 MHz or 500 MHz spectrometers with tetramethylsilane or residual protiated solvent used as a reference. Abbreviations for ^1H NMR splitting patterns are: s, singlet; bs, broad singlet; d, doublet; t, triplet; sept, septet; dd, doublet of doublets; td, triplet of doublets; ddd, doublet of double doublets; m, multiplet. In the case of compound **2d** which bears F, the abbreviation refers to the splitting pattern due to C-F coupling (the corresponding coupling constant is also reported). The coupling constants are reported in hertz (Hz). Flash column chromatography was carried out on silica gel (230-240

mesh). The yields of the coupled products included in all tables refer to isolated yields. Products that had been reported previously were isolated in greater than 95% purity, as determined by ^1H NMR spectroscopy and capillary gas chromatography (GC). GC and GC/MS analyses were conducted with an HP-1 methyl silicone column.

Preparation of stock solution A (1.0×10^{-2} M): Toluene (1.0 mL) was added to a mixture of $\text{Pd}(\text{OAc})_2$ (2.2 mg) and CyPF-*t*Bu (5.5 mg). The resulting orange solution was stirred at room temperature for 1 min before using

General procedure for the coupling of aryl bromides with triisopropylsilanethiol: The appropriate quantity of stock solution **A** was added to a 4 mL vial containing the aryl bromide or chloride (1.00 mmol) and LiHMDS (184 mg, 1.10 mmol) in toluene (1.5 mL). TIPS-SH (214 μL , 1.00 mmol) was then added, and the vial sealed with a cap containing a PTFE septum. The mixture was heated at 110 $^\circ\text{C}$ until the aryl halide was consumed, as determined by GC. Silica gel (0.5 g) was added, and the solvents were evaporated under reduced pressure. The crude residue was purified by column chromatography on silica gel using hexane or a mixture of hexane and ethyl acetate as eluent. Protected aryl thiols were isolated in the yields reported in Table 1.



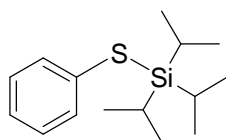
4-Methylphenyl triisopropylsilyl sulfide (Table 1, entry 1).^[1] 50 μL of stock solution A were employed. 98% yield. Colorless liquid. ^1H NMR (CDCl_3): d 7.32 (d, $J = 8.0$ Hz, 2H), 6.96 (d, $J = 8.0$ Hz, 2H), 2.24 (s, 3H), 1.24-1.15 (m, 1H), 1.03 (d, $J = 7.6$ Hz, 18H). ^{13}C NMR (CDCl_3): d 136.4, 135.2 (2C), 129.3 (2C), 127.5, 20.9, 18.4 (6C), 13.0 (3C).

4-Methylphenyl triisopropylsilyl sulfide (Table 1, entry 2).^[1] 50 μL of stock solution A was employed. Reaction performed using NaOtBu (106 mg, 1.10 mmol) as base. 88% yield.

4-Methylphenyl triisopropylsilyl sulfide (Table 1, entry 3).^[1] 50 μL of stock solution A was employed. Reaction conducted in DME (1.5 mL). 90% yield.

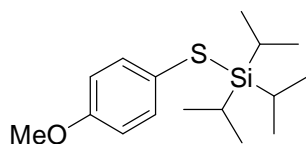
4-Methylphenyl triisopropylsilyl sulfide (Table 1, entry 4).^[1] 100 μL of stock solution A was employed and the mixture heated at 90 $^\circ\text{C}$. 91% yield.

4-Methylphenyl triisopropylsilyl sulfide (Table 1, entry 5).^[1] 100 μL of stock solution A was employed and the reaction performed using 4-chlorotoluene. 96% yield

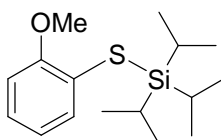


Phenyl triisopropylsilyl sulfide (Table 1, entry 6).^[2] 50 μL of stock solution A was employed. 91% yield. Colorless liquid. ^1H NMR (CDCl_3): d 7.41-7.39 (m, 2H), 7.11-7.09 (m, 3H), 1.19-1.11 (m, 3H), 0.99 (d, $J = 7.3$ Hz, 18H). ^{13}C NMR

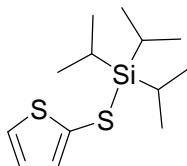
(CDCl₃): d 135.4 (2C), 131.4, 128.5 (2C), 126.6, 18.3 (6C), 13.0 (3C).



4-Methoxyphenyl triisopropylsilyl sulfide (Table 1, entry 7).^[1] 250 μ L of stock solution A was employed. 96% yield. Colorless liquid. ¹H NMR (CDCl₃): d 7.31 (d, *J* = 8.8 Hz, 2H), 6.68 (d, *J* = 8.8 Hz, 2H), 3.70 (s, 3H), 1.18-1.10 (m, 3H), 1.00 (d, *J* = 7.3 Hz, 18H). ¹³C NMR (CDCl₃): d 158.7, 136.5 (2C), 121.5, 114.2 (2C), 55.2, 18.4 (6C), 12.9 (3C).

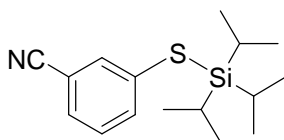


2-Methoxyphenyl triisopropylsilyl sulfide (Table 1, entry 8).^[1] 250 μ L of stock solution A was employed. 99% yield. Colorless liquid. ¹H NMR (CDCl₃): d 7.41 (m, 1H), 7.12 (m, 1H), 6.75-6.72 (m, 2H), 3.77 (s, 3H), 1.21-1.11 (m, 3H), 0.99 (d, *J* = 7.3 Hz, 18H). ¹³C NMR (CDCl₃): d 160.1, 137.5, 128.3, 120.4, 119.3, 110.7, 55.3, 18.2 (6C), 13.3 (3C).

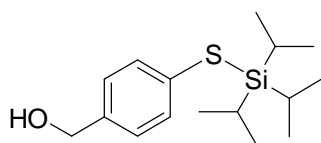


Thiophen-2-yl triisopropylsilyl sulfide (Table 1, entry 9).^[1] 250 μ L of stock solution A was employed. Deactivated silica gel was employed in the purification to minimize desilylation. 76% yield. Colorless liquid. ¹H NMR (CDCl₃):

d 7.02 (dd, $J = 1.3$ Hz and 5.4 Hz, 1H), 6.87 (dd, $J = 1.3$ Hz and 3.5 Hz, 1H), 6.74 (dd, $J = 3.5$ Hz and 5.4 Hz, 1H), 1.18-1.07 (m, 3H), 0.94 (d, $J = 7.6$ Hz, 18H). ^{13}C NMR (CDCl_3): d 133.8, 129.2, 127.5, 127.2, 18.3 (6C), 12.9 (3C).

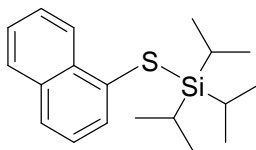


3-Cyanophenyl triisopropylsilyl sulfide (Table 1, entry 10). 250 μL of stock solution A was employed. Deactivated silica gel was employed in the purification to minimize desilylation. 87% yield. Colorless liquid. ^1H NMR (CDCl_3): d 7.56 (s, 1H), 7.52 (d, $J = 7.9$ Hz, 1H), 7.30 (d, $J = 7.9$ Hz, 1H), 7.12 (t, $J = 7.9$ Hz, 1H), 1.09-1.00 (m, 3H), 0.88 (d, $J = 7.3$ Hz, 18H). ^{13}C NMR (CDCl_3): d 139.6, 138.1, 134.5, 130.1, 129.1, 118.2, 112.7, 18.3 (6C), 13.0 (3C). Elem. Anal. Calcd for $\text{C}_{16}\text{H}_{25}\text{NSSi}$: C, 65.92; H, 8.64; N, 4.80. Found: C, 65.66; H, 8.38; N, 4.96.



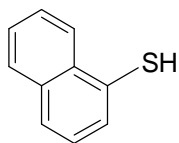
4-Hydroxymethylphenyl triisopropylsilyl sulfide (Table 1, entry 11). 250 μL of stock solution A was employed. 74% yield. Pale yellow oil. ^1H NMR (CDCl_3): d 7.34 (d, $J = 8.0$ Hz, 2H), 7.06 (d, $J = 8.0$ Hz, 2H), 4.49 (s, 2H), 1.73 (bs, 1H), 1.16-1.07 (m, 3H), 0.94 (d, $J = 7.3$ Hz, 18H). ^{13}C NMR (CDCl_3): d 139.4, 135.4 (2C), 130.7, 127.1 (2C), 64.7, 18.4

(6C), 13.0 (3C). Elem. Anal. Calcd for C₁₆H₂₈OSSi: C, 64.80; H, 9.52. Found: C, 64.96; H, 9.38.



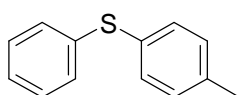
1-Naphthyl triisopropylsilyl sulfide (Table 1, entry 12).^[2]

50 μ L of stock solution A was employed. 97% yield. Colorless liquid. ¹H NMR (CDCl₃): d 8.61 (d, *J* = 8.5 Hz, 1H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.60–7.57 (m, 1H), 7.42–7.39 (m, 1H), 7.33–7.30 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 1.18–1.10 (m, 3H), 0.89 (d, *J* = 7.5 Hz, 18H). ¹³C NMR (CDCl₃): d 135.9, 134.1, 133.8, 129.6, 128.2, 127.6, 127.2, 126.0, 125.9, 125.2, 18.3 (6C), 13.3 (3C).

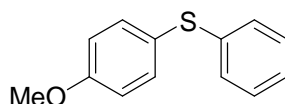


1-Naphthalenethiol (Table 1, entry 13).^[2] 50 μ L of stock solution A was employed. TBAF (2 equiv.) was added to the crude mixture of the coupling reaction and the mixture was stirred for 30 min at rt. The crude residue was purified by column chromatography on silica gel using hexane as eluent. 95% yield from 1-bromonaphthalane. Colorless oil. ¹H NMR (CDCl₃): d 8.23–8.20 (m, 1H), 7.75–7.72 (m, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.61 (d, *J* = 7.3 Hz, 1H), 7.40–7.35 (m, 2H), 7.29 (t, *J* = 7.8 Hz, 1H), 4.32 (s, 1H). ¹³C NMR (CDCl₃): d 134.0, 133.3, 131.8, 130.9, 128.5 (2C), 126.6, 126.2, 125.4, 125.1.

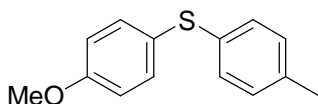
General procedure for the one-pot synthesis of unsymmetrical diaryl sulfides 2 from two aryl bromides and triisopropylsilanethiol: Pd(OAc)₂ (1.1 mg) and CyPF-*t*Bu (2.75 mg) were added to a 4 mL vial containing the aryl bromide (1.00 mmol) and LiHMDS (184 mg, 1.10 mmol) in toluene (1.5 mL). TIPS-SH (214 μ L, 1.00 mmol) was then added, and the vial sealed with a cap containing a PTFE septum. The mixture was heated at 90 °C until the aryl bromide was consumed (1-2 h) as determined by GC. The second bromide (1.00 mmol) and CsF (608 mg, 4.00 mmol) were then added, and the mixture was heated at 110 °C until the aryl bromide was consumed (4-24 h), as determined by GC. Silica gel (0.5 g) was added, and the solvents were evaporated under reduced pressure. The crude residue was purified by column chromatography on silica gel using hexane or a mixture of hexane and ethyl acetate as eluent. Diaryl sulfides **2** were isolated in the yields reported in Scheme 2.



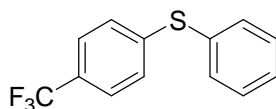
4-Methylphenyl phenyl sulfide 2a.^[3] 97% yield (98% yield when 0.25 mol% catalyst was employed). Colorless liquid. ¹H NMR (CDCl₃): d 7.21 (d, *J* = 8.0 Hz, 2H), 7.19-7.14 (m, 4H), 7.11-7.05 (m, 1H), 7.03 (d, *J* = 8.0 Hz, 2H), 2.25 (s, 3H). ¹³C NMR (CDCl₃): d 137.5, 137.0, 132.2 (2C), 131.2, 130.0 (2C), 129.7 (2C), 128.9 (2C), 126.3, 21.0.



4-Methoxyphenyl phenyl sulfide 2b.^[4] The coupling of *p*-methoxybromobenzene with TIPS-SH, followed by bromobenzene, afforded the title compound in 97% yield. The reaction conducted with the opposite order of addition of the aryl bromides (first bromobenzene, then *p*-methoxybromobenzene) formed the sulfide in 95% yield. Colorless liquid. ¹H NMR (CDCl₃): d 7.23 (d, *J* = 8.8 Hz, 2H), 7.06-7.02 (m, 2H), 7.00-6.93 (m, 3H), 6.71 (d, *J* = 8.8 Hz, 2H), 3.62 (s, 3H). ¹³C NMR (CDCl₃): d 159.7, 138.5, 135.3 (2C), 128.8 (2C), 128.1 (2C), 125.7, 124.2, 114.9 (2C), 55.3.

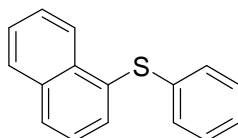


4-Methylphenyl 4-methoxyphenyl sulfide 2c.^[5] 98% yield. White solid. ¹H NMR (CDCl₃): d 7.47 (d, *J* = 8.8 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H), 2.40 (s, 3H). ¹³C NMR (CDCl₃): d 159.3, 135.9, 134.21 (2C), 134.15, 129.6 (2C), 129.2 (2C), 125.4, 114.7 (2C), 55.1, 20.8.

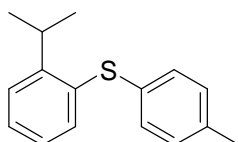


Phenyl 4-trifluoromethylphenyl sulfide 2d.^[6] The coupling of TIPS-SH with *p*-trifluoromethylbromobenzene, followed by bromobenzene, afforded the title compound in 98% yield. The reaction conducted with the opposite order of addition of the aryl bromides (first bromobenzene, then *p*-trifluoromethylbromobenzene) gave the desired unsymmetrical thioether in 92% isolated yield. When the coupling was

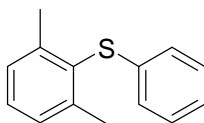
conducted in this order, the product was contaminated with 5% of the undesired symmetrical sulfides. Colorless liquid. ^1H NMR (CDCl_3): d 7.47–7.45 (m, 4H), 7.39–7.35 (m, 3H), 7.28–7.24 (m, 2H). ^{13}C NMR (CDCl_3): d 142.8, 133.5 (2C), 132.4, 129.6 (2C), 128.6, 128.2 (2C), 128.1 (q, $^2J_{\text{C-F}} = 32.6$ Hz), 125.7 (d, $^3J_{\text{C-F}} = 3.8$ Hz), 124.0 (q, $^1J_{\text{C-F}} = 271.6$ Hz).



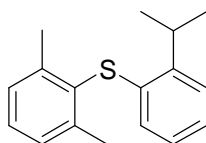
1-Naphthyl phenyl sulfide 2e.^[7] 91% yield. Colorless liquid. ^1H NMR (CDCl_3): d 8.26 (s, 1H), 7.71–7.53 (m, 3H), 7.37–7.0 (m, 8H). ^{13}C NMR (CDCl_3): d 136.8, 134.1, 133.5, 132.4, 131.1, 129.1, 129.0 (2C), 128.9 (2C), 128.5, 126.8, 126.3, 126.0, 125.7, 125.5.



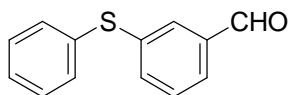
2-Isopropylphenyl 4-methylphenyl sulfide 2f.^[8] 85% yield. Colorless liquid. ^1H NMR (CDCl_3): d 7.32 (dd, $J = 7.7$ Hz and 1.4 Hz, 1H), 7.26–7.14 (m, 4H), 7.11–7.06 (m, 3H), 3.54 (sept, $J = 6.8$ Hz, 1H), 2.32 (s, 3H), 1.22 (d, $J = 6.8$ Hz, 6H). ^{13}C NMR (CDCl_3): d 149.2, 136.4, 133.8, 132.7, 132.3, 130.6 (2C), 129.8 (2C), 127.6, 126.4, 125.7, 30.4, 23.4 (2C), 20.9.



2,6-Dimethylphenyl phenyl sulfide 2g.^[9] Reaction performed with 2 mol % catalyst (4.4 mg of Pd(OAc)₂ and 11 mg of CyPF-*t*Bu). 95% yield. Colorless liquid. ¹H NMR (CDCl₃): δ 7.33–7.29 (m, 1H), 7.27–7.23 (m, 4H), 7.15–7.11 (m, 1H), 7.02–6.99 (m, 2H), 2.51 (s, 6H). ¹³C NMR (CDCl₃): δ 143.8 (2C), 137.9, 130.4, 129.2, 128.8 (2C), 128.4 (2C), 125.5 (2C), 124.5, 21.8 (2C).

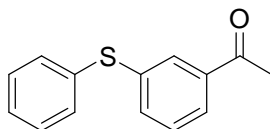


2,6-Dimethylphenyl 2-isopropyl sulfide 2h. Reaction performed with 3 mol % catalyst (6.6 mg of Pd(OAc)₂ and 16.5 mg of CyPF-*t*Bu). 94% yield. White solid. ¹H NMR (CDCl₃): δ 7.17–7.08 (m, 4H), 6.96 (td, *J* = 7.3 Hz and 1.3 Hz, 1H), 6.81 (ddd, *J* = 7.9 Hz, 7.3 Hz and 1.5 Hz, 1H), 6.32 (dd, *J* = 7.9 Hz and 1.3 Hz, 1H), 3.42 (sept, *J* = 6.6 Hz, 1H), 2.31 (s, 6H), 1.25 (d, *J* = 6.6 Hz, 6H). ¹³C NMR (CDCl₃): δ 145.2, 144.0 (2C), 135.8, 130.9, 129.0, 128.4 (2C), 126.3, 125.1, 124.8, 124.6, 30.2, 23.0 (2C), 21.7 (2C). Elem. Anal. Calcd for C₁₇H₂₀S: C, 79.63; H, 7.86. Found: C, 79.87; H, 7.87.

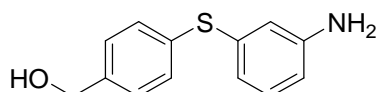


3-Phenylsulfanylbenzaldehyde 2i.^[10] 71% yield. Colorless oil. ¹H NMR (CDCl₃): δ 9.83 (s, 1H), 7.66 (t, *J* = 1.8 Hz, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 7.7 Hz, 1H), 7.35–7.31 (m, 2H), 7.28–7.20 (m, 3H). ¹³C NMR (CDCl₃): δ

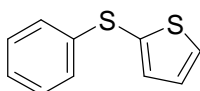
191.5, 138.6, 137.0, 135.2, 133.5, 132.3 (2C), 130.4, 129.6, 129.4 (2C), 128.0, 127.5.



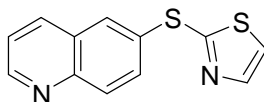
3-Acetylphenyl phenyl sulfide 2j. 77% yield. Colorless liquid. ^1H NMR (CDCl_3): d 7.78 (bs, 1H), 7.65 (d, $J = 7.9$ Hz, 1H), 7.32 (d, $J = 7.9$ Hz, 1H), 7.26–7.13 (m, 6H), 2.4 (s, 3H). ^{13}C NMR (CDCl_3): d 197.1, 137.7, 137.3, 134.3, 134.1, 131.6 (2C), 129.7, 129.2 (2C), 129.1, 127.5, 126.3, 26.4. Elem. Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{OS}$: C, 73.65; H, 5.30. Found: C, 73.34; H, 5.38.



3-Aminophenyl (4-hydroxymethyl)phenyl sulfide 2k. Reaction performed with 1 mol % catalyst (2.2 mg of $\text{Pd}(\text{OAc})_2$ and 5.5 mg of CyPF-*t*Bu. A portion of the crude product contained an O-TMS group; thus, aqueous 1 N HCL was added prior to the chromatographic purification to remove the silyl group. 69% yield. Yellow oil. ^1H NMR (CDCl_3): d 7.26 (d, $J = 8.3$ Hz, 2H), 7.20 (d, $J = 8.3$ Hz, 2H), 6.99 (t, $J = 7.7$ Hz, 1H), 6.63 (m, 1H), 6.55 (t, $J = 1.9$ Hz, 1H), 6.47 (m, 1H), 4.57 (s, 2H), 2.95 (bs, 3H). ^{13}C NMR (CDCl_3): d 147.0, 139.7, 136.5, 134.8, 131.3 (2C), 129.9, 127.7 (2C), 120.9, 116.9, 113.9, 64.7. Elem. Anal. Calcd for $\text{C}_{13}\text{H}_{13}\text{NOS}$: C, 67.50; H, 5.66; N, 6.06. Found: C, 67.72; H, 5.81; N, 5.78.



Phenyl thiophen-2-yl sulfide 2l.^[4] Colorless liquid. 92% yield. ¹H NMR (CDCl₃): d 7.32 (d, *J* = 5.1 Hz, 1H), 7.18-7.00 (m, 6H), 6.95-6.93 (m, 1H). ¹³C NMR (CDCl₃): d 138.5, 135.9, 131.2, 130.9, 128.9 (2C), 127.8, 127.0 (2C), 125.9.



Quinol-6-yl thiazol-2-yl sulfide 2m. Reaction performed with 1 mol % catalyst (2.2 mg Pd(OAc)₂ and 5.5 mg of CyPF-*t*Bu). 85% yield. White solid. ¹H NMR (CDCl₃): d 8.86 (dd, *J* = 4.2 Hz and *J* = 1.6 Hz, 1H), 8.03 (m, 2H), 7.99 (d, *J* = 1.6 Hz, 1H), 7.75 (dd, *J* = 8.8 Hz and *J* = 1.6 Hz, 1H), 7.68 (d, *J* = 3.5 Hz, 1H), 7.35 (dd, *J* = 8.2 Hz and *J* = 4.1 Hz, 1H), 7.20 (d, *J* = 3.5, 1H). ¹³C NMR (CDCl₃): d 163.7, 151.3, 147.9, 143.6, 135.7, 133.0, 132.0, 130.9, 130.6, 128.5, 121.8, 121.2. Elem. Anal. Calcd for C₁₂H₈N₂S₂: C, 58.99; H, 3.30; N, 11.47. Found: C, 58.94; H, 3.39; N, 11.12.

Tandem reaction between *p*-bromotoluene, TIPS-SH and chlorobenzene:

Pd(OAc)₂ (2.2 mg) and CyPF-*t*Bu (5.5 mg) were added to a 4 mL vial containing 4-bromotoluene (121 μL, 1.00 mmol), chlorobenzene (102 μL, 1.00 mmol), LiHMDS (184 mg, 1.10 mmol) and CsF (608 mg, 4.00 mmol) in toluene (1.5 mL). The vial was sealed with a cap containing a PTFE septum, and the mixture was heated at 100 °C until the chlorobenzene was consumed, as determined by GC. Silica gel (0.5 g) was added, and the solvents were evaporated under reduced pressure. The crude residue was purified by column

chromatography on silica gel using hexane as eluent. 4-Methylphenyl phenyl sulfide **2a**^[3] was obtained as the major product in 80% yield (160 mg). This isolated material contained 8% of the symmetrical sulfides, which could not be separated from the unsymmetrical sulfide, in this case, by column chromatography.

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^1H and ^{13}C NMR Spectra Of Reaction Products

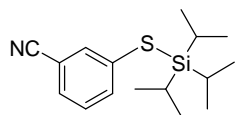
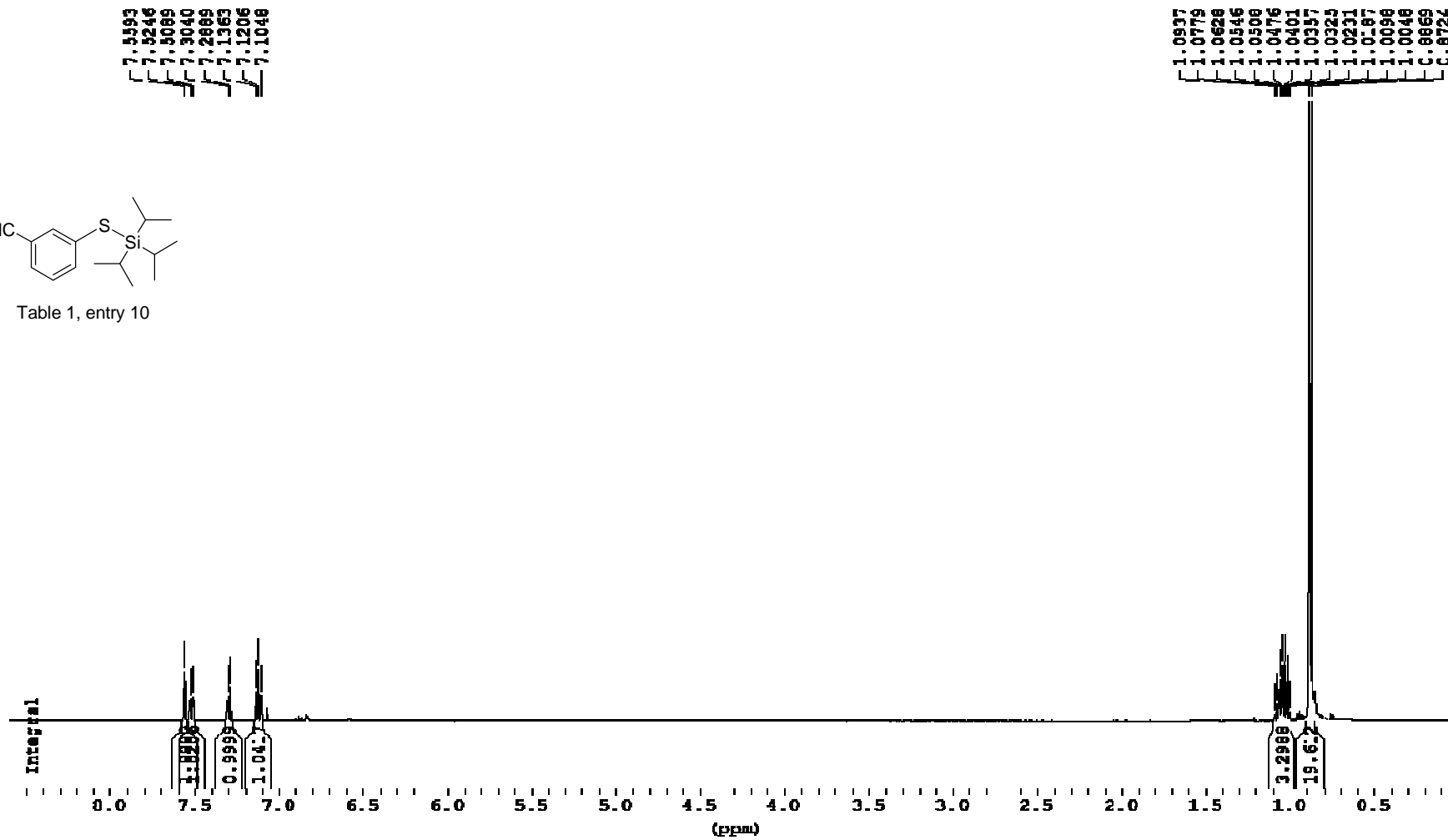


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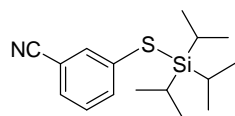
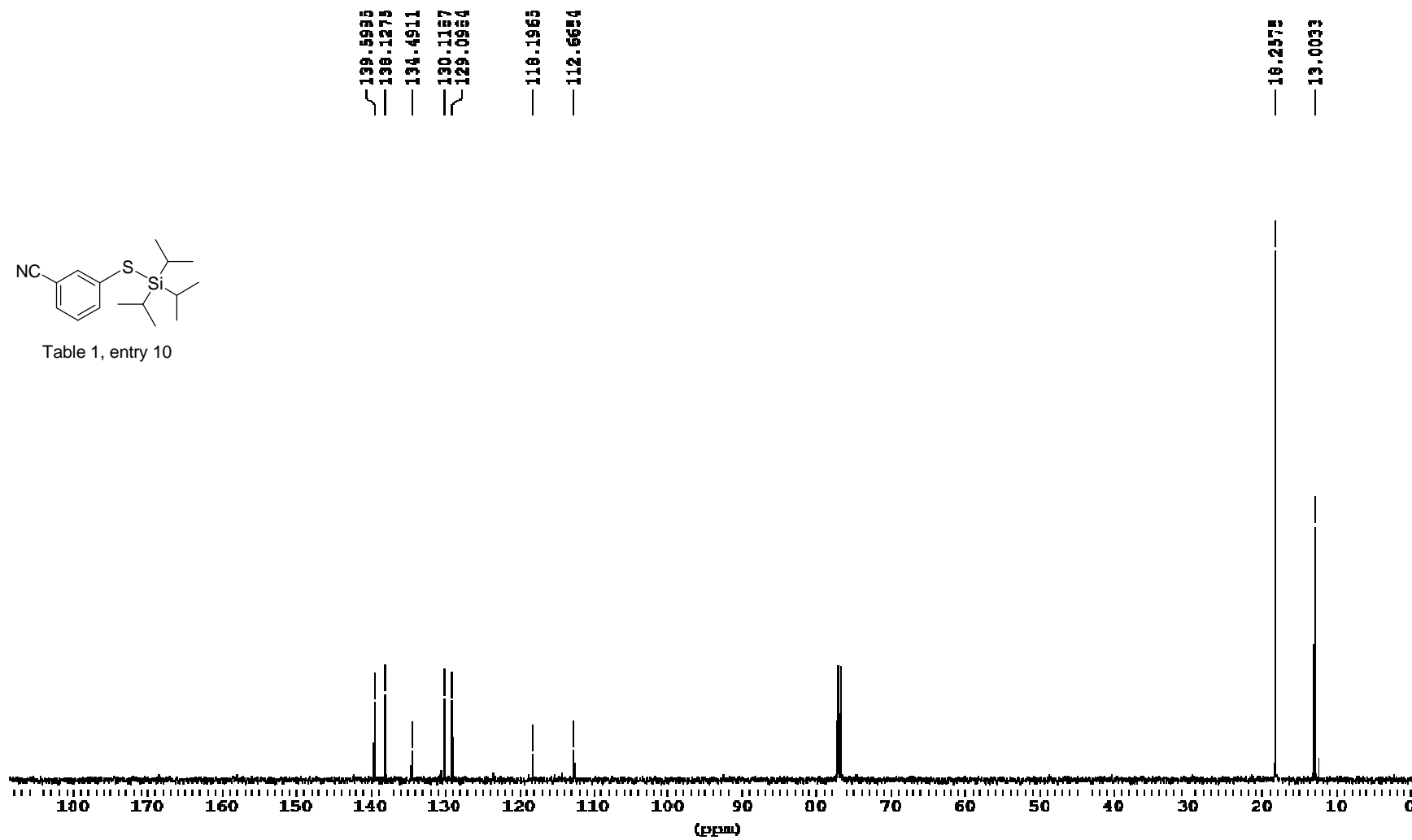


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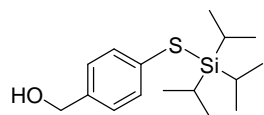
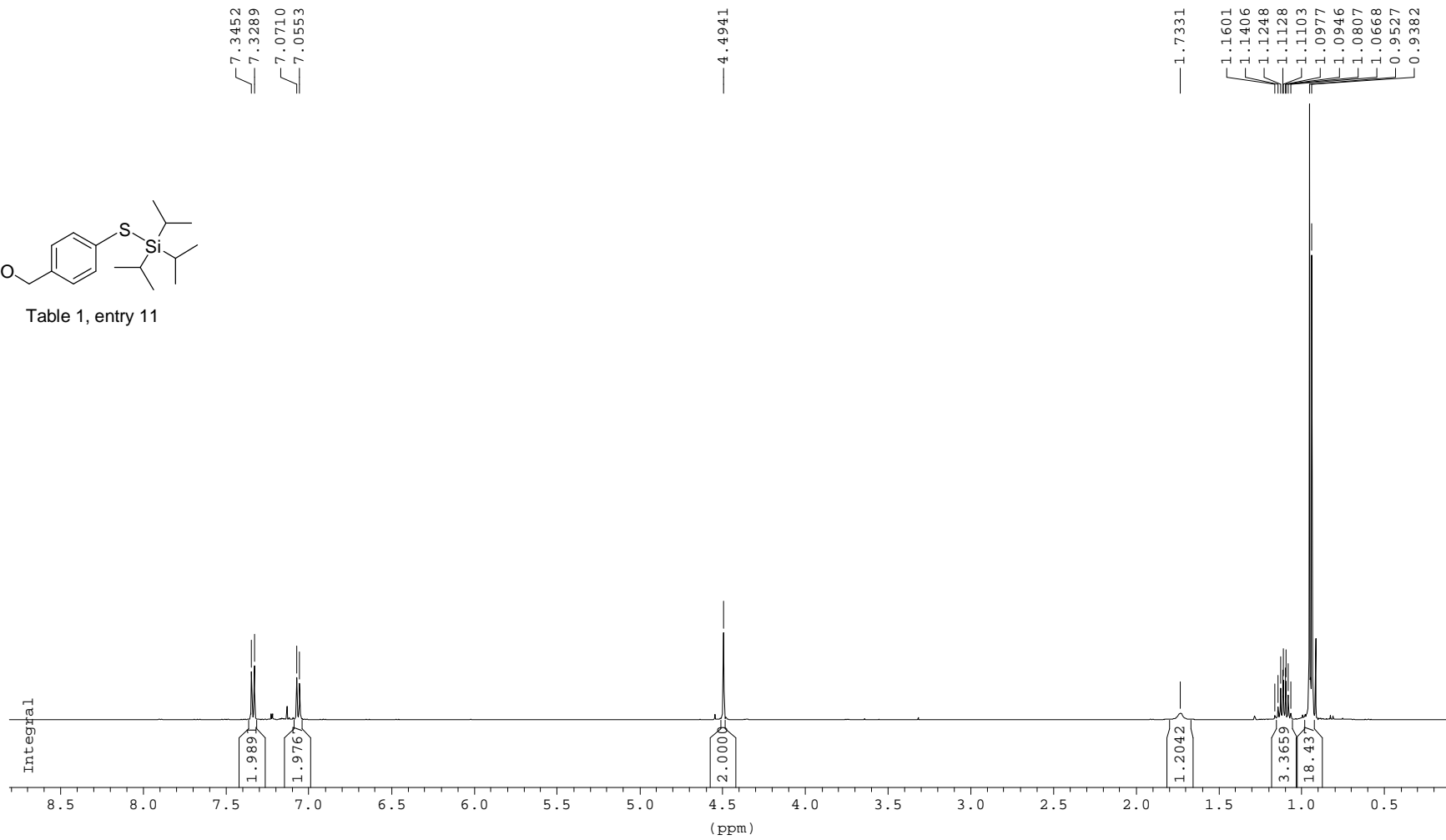


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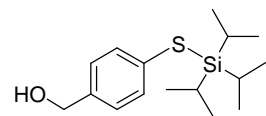
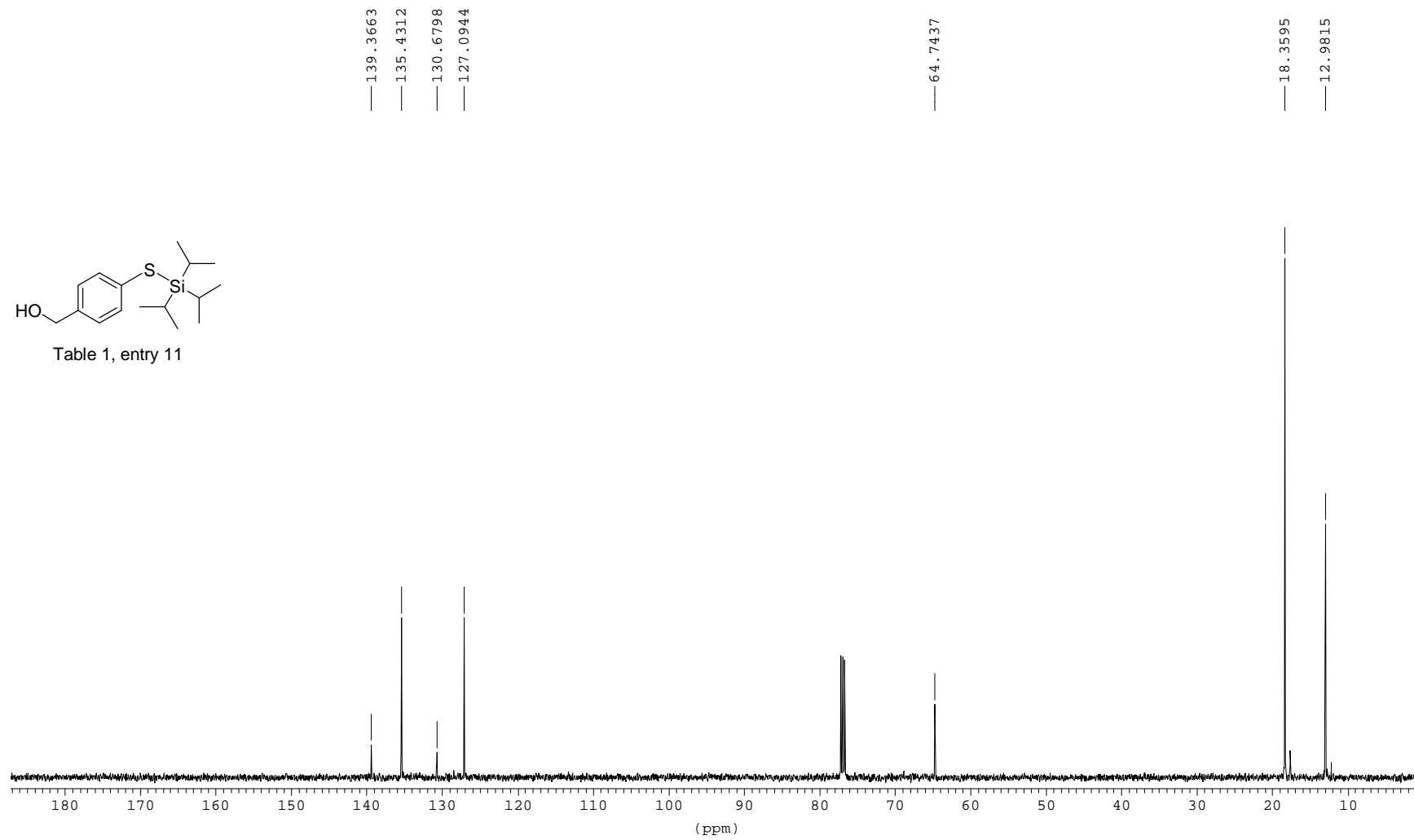
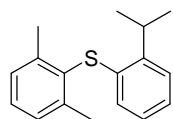
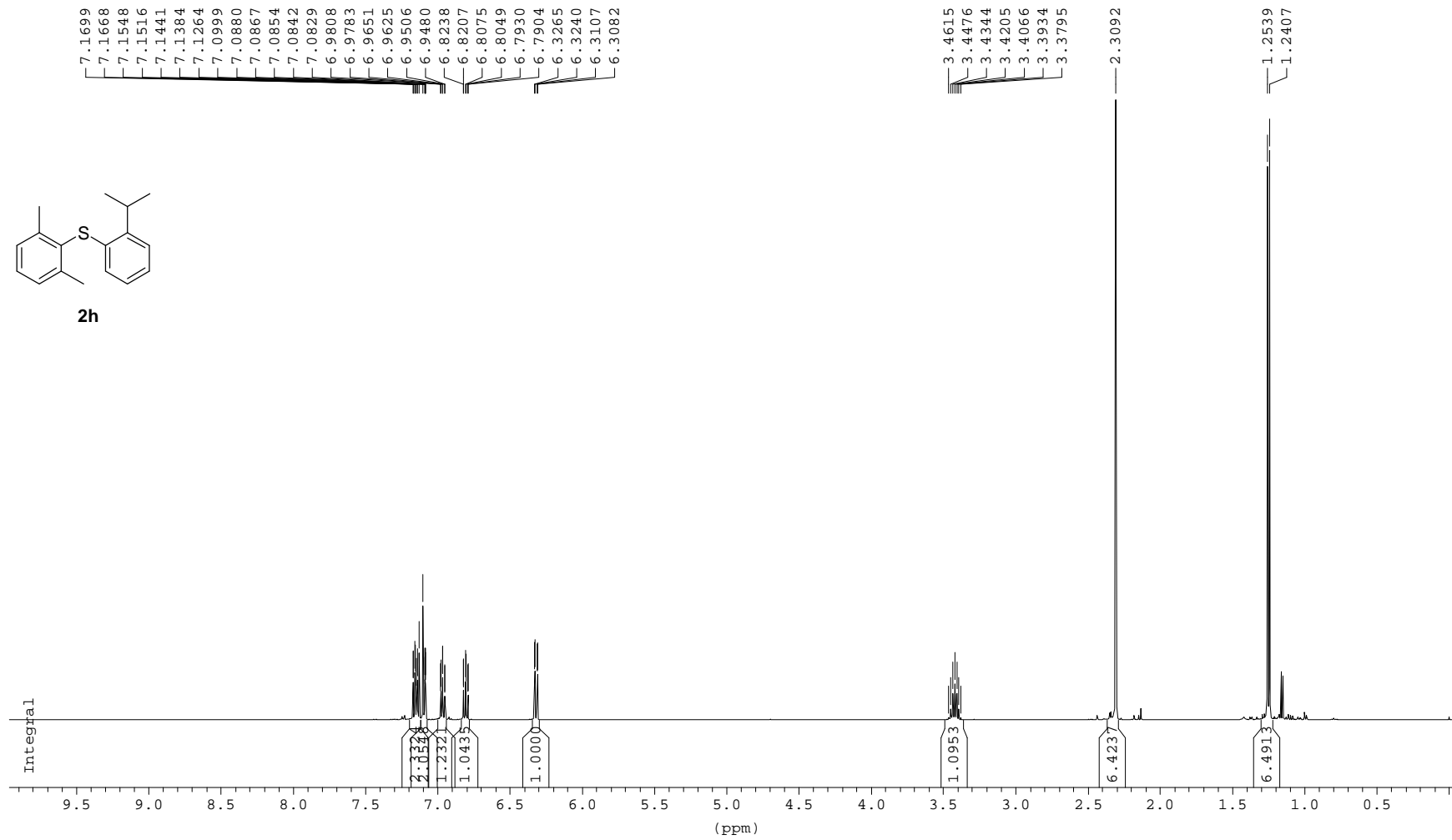


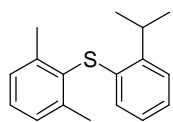
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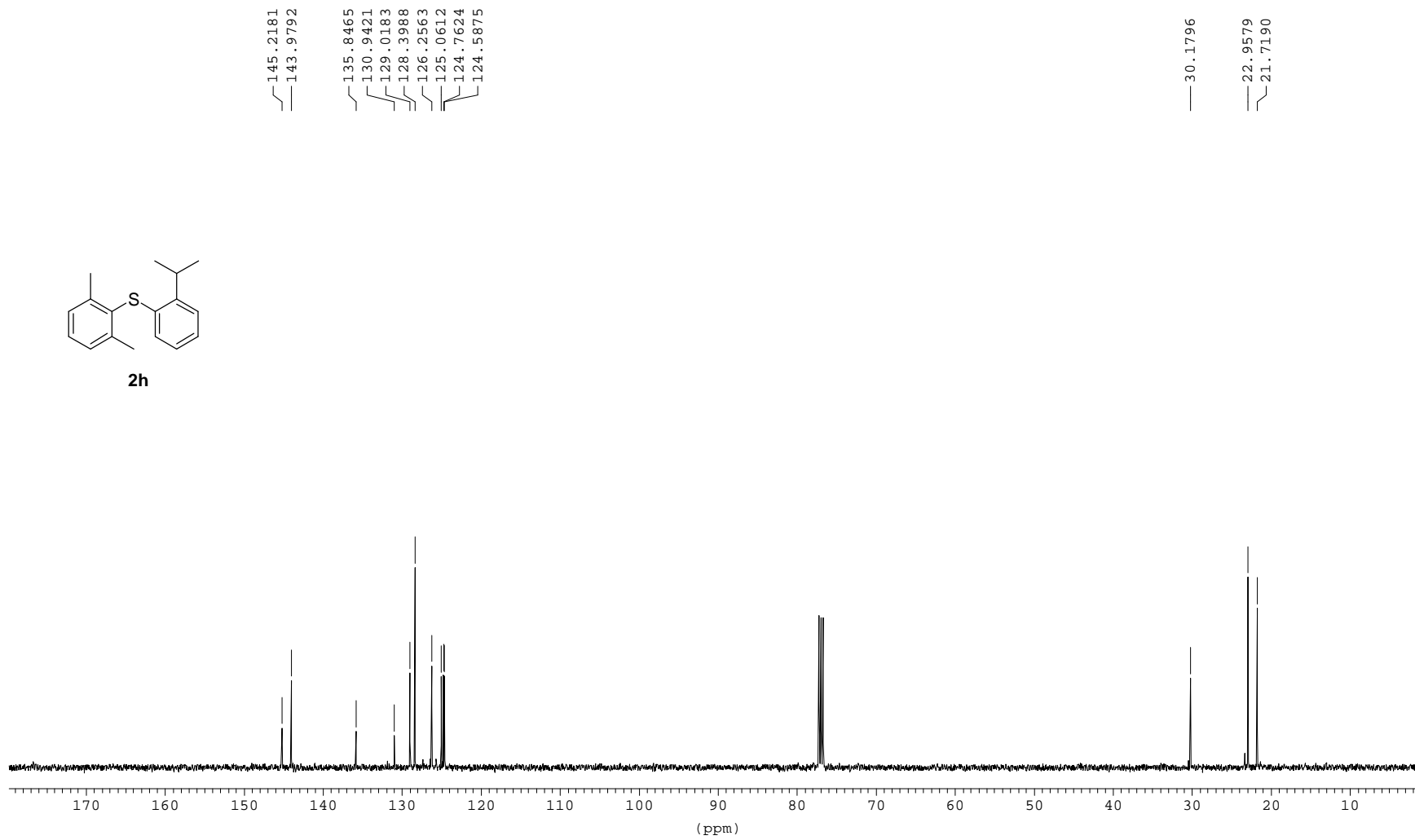


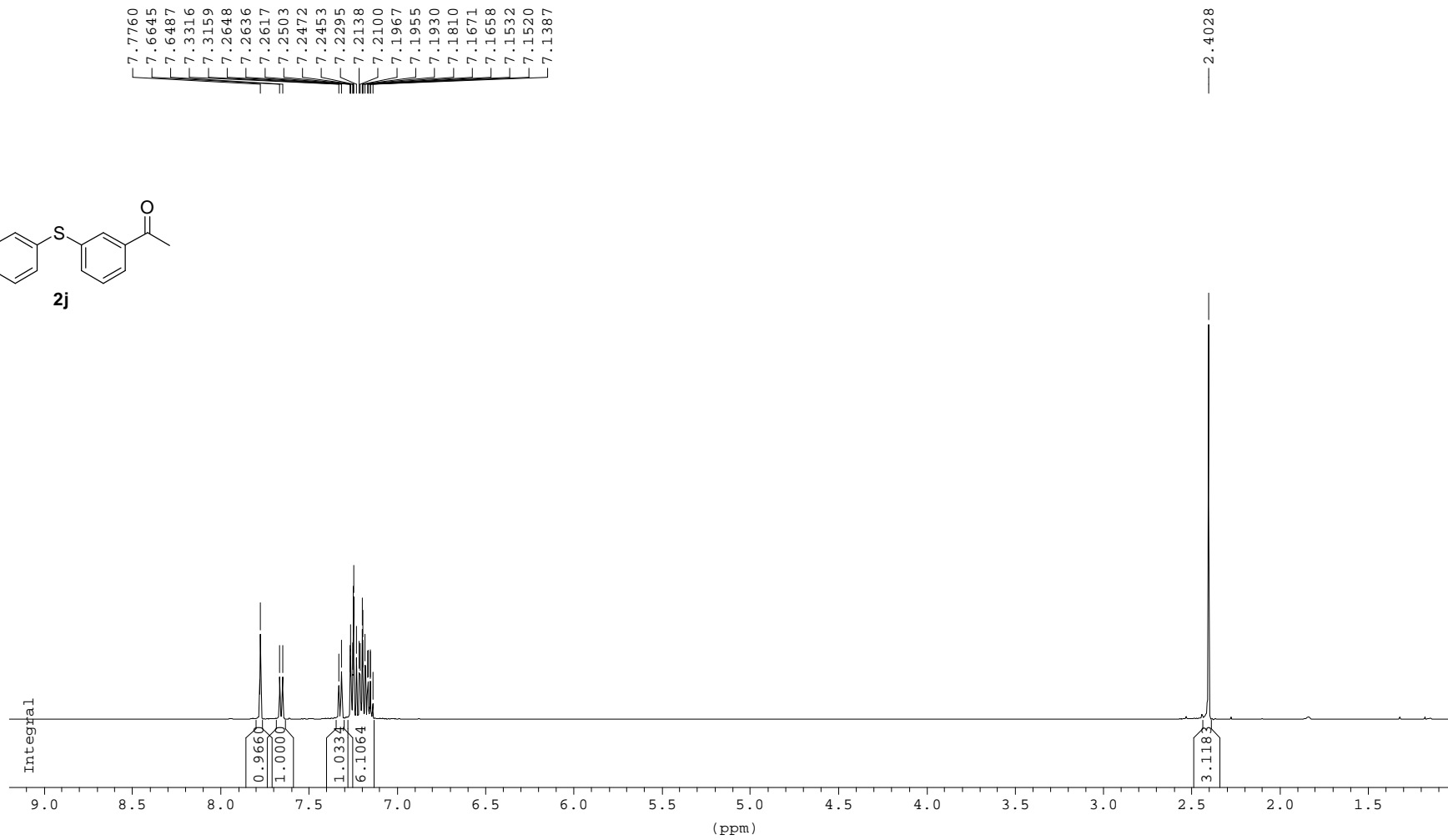
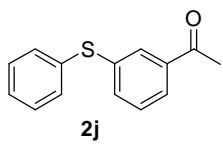
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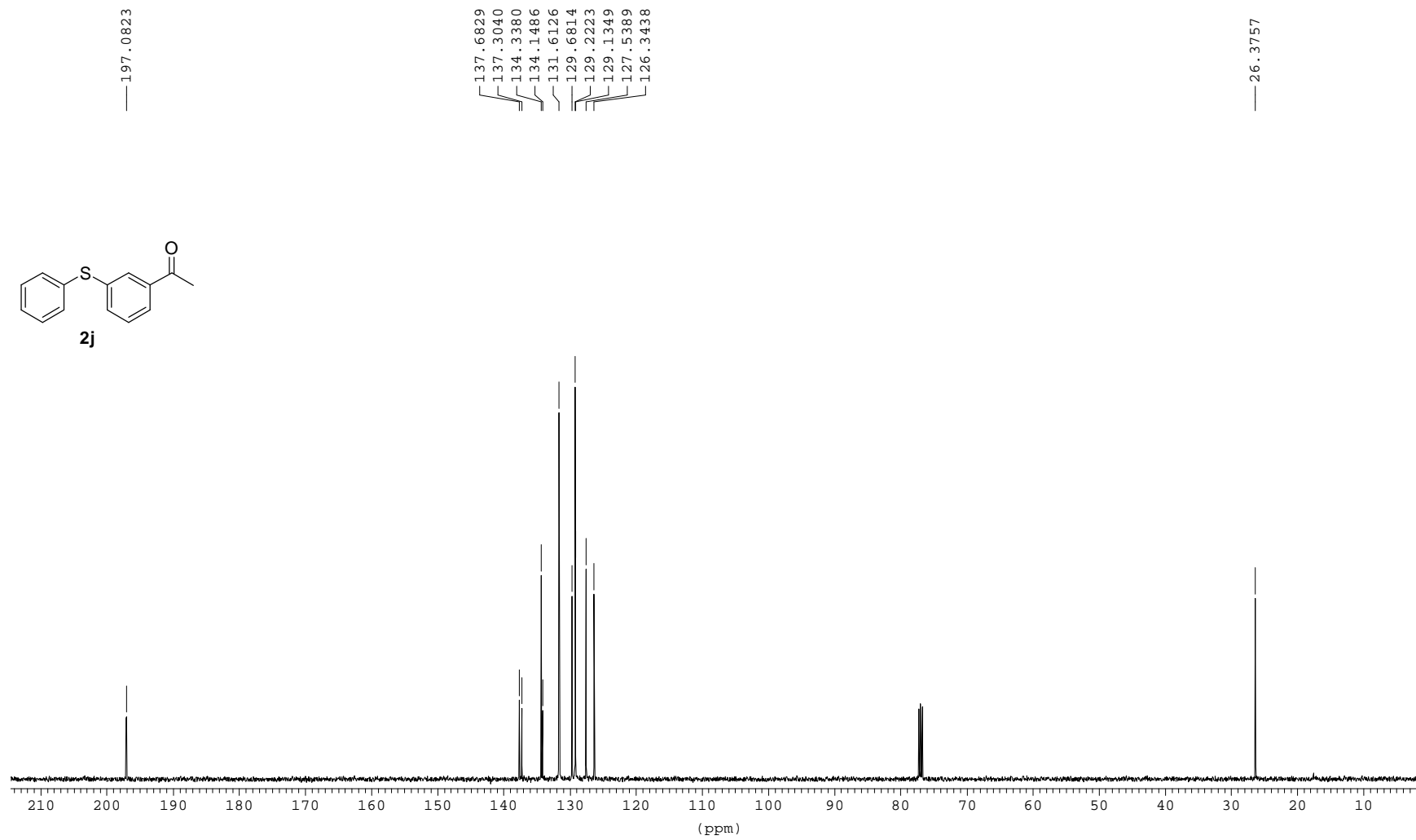
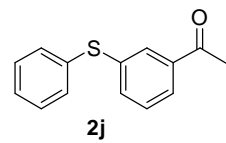


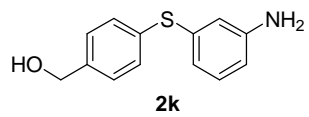


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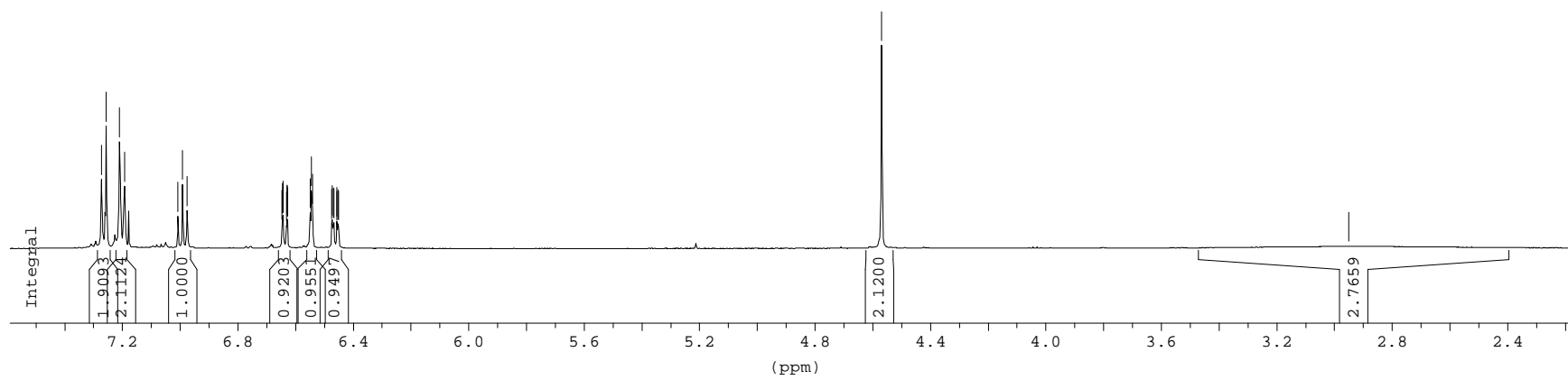


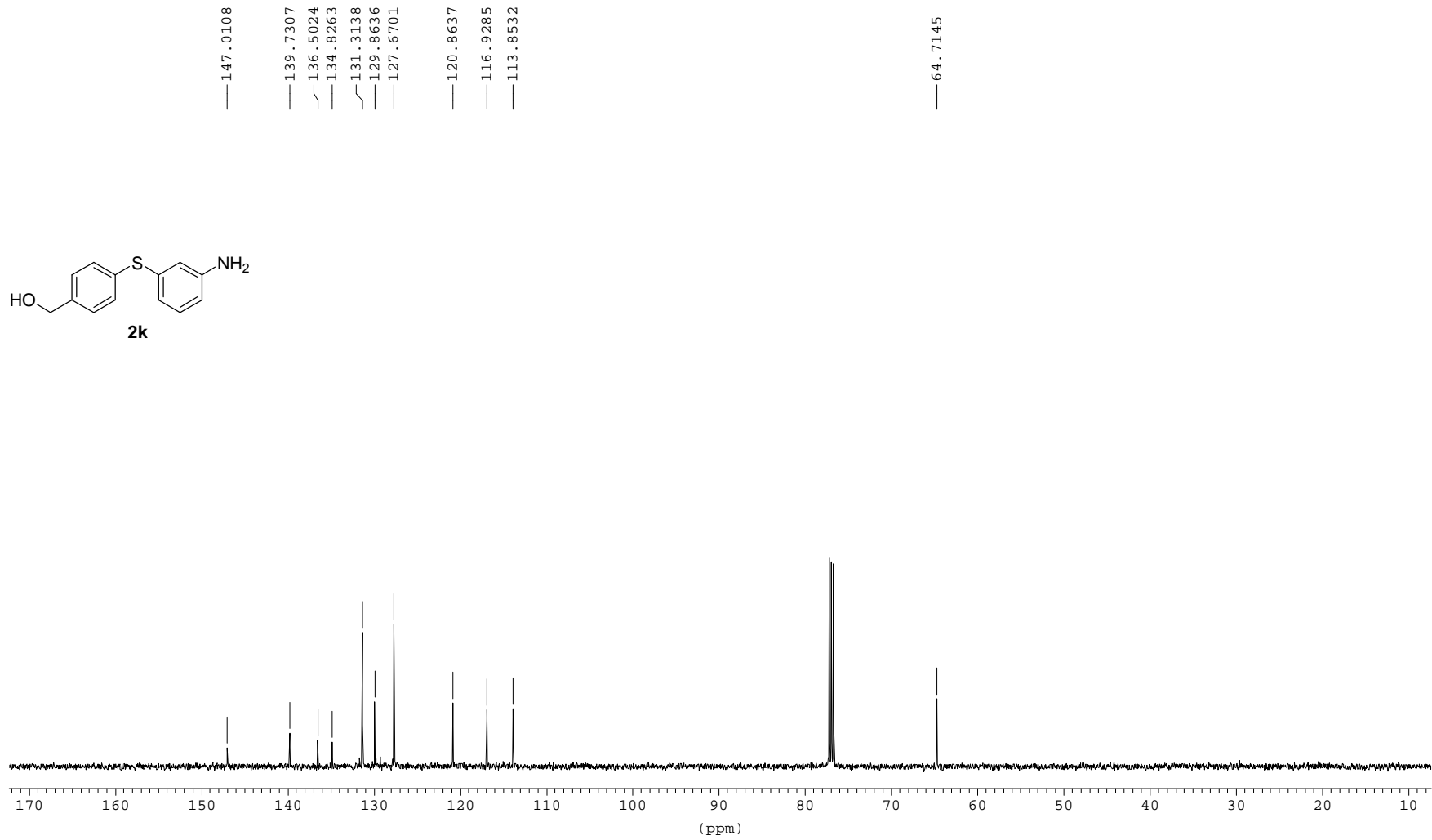
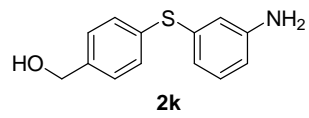


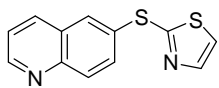
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