

Indium-Catalyzed C-S Cross-Coupling of Aryl Halides with Thiols

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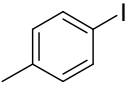
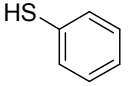
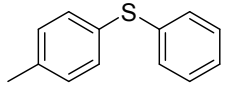
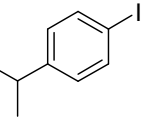
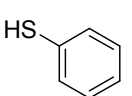
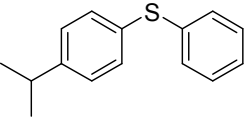
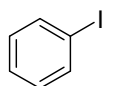
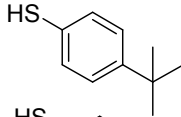
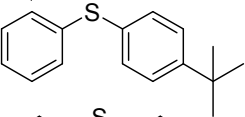
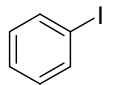
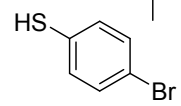
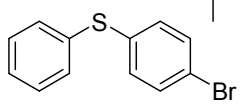
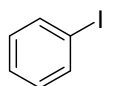
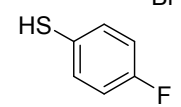
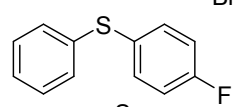
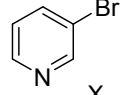
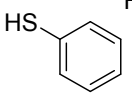
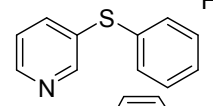
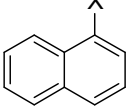
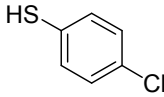
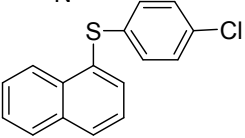
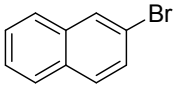
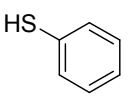
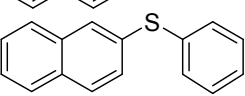
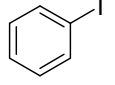
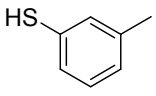
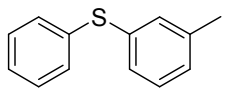
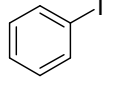
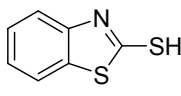
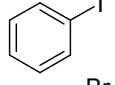
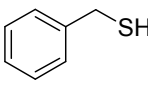
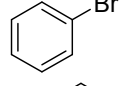
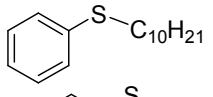
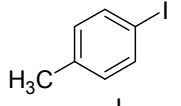
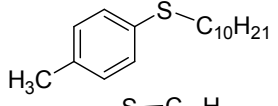
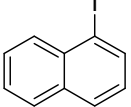
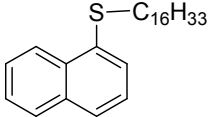
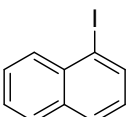
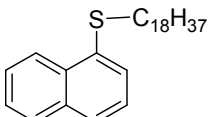
General Information:

Analytical thin layer chromatography (TLC) was carried out using silica gel 60 F₂₅₄ pre-coated plates. Visualization was accomplished with UV lamp. ¹H NMR and ¹³C was recorded on 200 and 300 MHz, in CDCl₃ using TMS as the internal standard, Chemical shifts were reported in parts per million (ppm, δ) downfield from tetramethylsilane. Melting points were determined on a Fischer-Johns melting point apparatus.

General procedure for the synthesis of Aryl Sulfides:

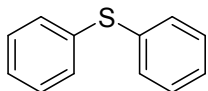
To a stirred solution of iodobenzene (1.1 mmol) and In(OTf)₃ (10 mol%) in dry DMSO (2.0 mL) was added thiophenol (1.0 mmol) followed by TMEDA (20 mol%) and KOH (2.0 eq) under nitrogen atmosphere. The reaction mixture was then stirred at 135 °C for 24 h. The progress of the reaction was monitored by TLC and then allowed to cool to room temperature. Diethyl ether (5 mL) was added and the organic phase was separated, dried over anhydrous Na₂SO₄, and purified by chromatography on silica gel to afford the corresponding coupling product.

Table 3. C-S cross-coupling of aryl halides with thiols^a

entry	aryl iodide	thiol	product	yield ^b (%)
26				89
27				85
28				90
29				93
30				94
31				19
32	 X = I Br			85
33				32
34				51
35				86
36			-	0
37			-	0
38		$C_{10}H_{21}SH$		25
39		$C_{10}H_{21}SH$		80
40		$C_{16}H_{33}SH$		81
41		$C_{18}H_{37}SH$		80

^a Reaction conditions: thiophenol (1.0 mmol), iodobenzene (1.1 mmol), 0.1 equiv of $In(OTf)_3$, 0.2 equiv of TMEDA, 2.0 equiv of KOH, DMSO (2.0 mL), 135 °C, 24 h, nitrogen atmosphere. ^b Yield of isolated product after flash chromatography.

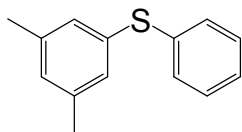
Spectroscopic Data



diphenylsulfane (entries 1,2,3, Table 3):¹

colorless oil

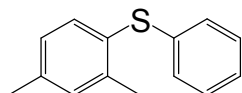
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.34 – 7.14 (m, 10H); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 135.7, 131.0, 129.1, 127.0



(3,5-dimethylphenyl)(phenyl)sulfane (entry 4, Table 3):²

colorless oil

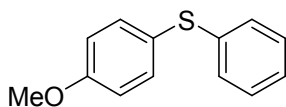
¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.26 – 7.12 (m, 5H), 6.94 (s, 2H), 6.82 (s, 1H), 2.25 (s, 6H) ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 138.8, 136.3, 134.7, 130.4, 129.1, 126.6, 21.4



(2,4-dimethylphenyl)(phenyl)sulfane (entry 5, Table 3):

colorless oil

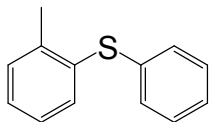
¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.24 (d, 1H, J = 7.5 Hz), 7.21 – 7.15 (m, 2H), 7.10 – 7.01 (m, 4H), 6.92 (d, 1H, J = 7.5 Hz) ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 140.7, 138.4, 137.3, 134.4, 131.5, 129.2, 128.9, 128.2, 127.5, 125.6, 21.0, 20.5; Mass (ESI): m/z 237[M+Na]; Anal calcd for: C₁₄H₁₄S (214) C: 78.46, H: 6.58, S: 14.96; found C: 78.37, H: 6.49, S: 14.89



(4-methoxyphenyl)(phenyl)sulfane (entries 6, 9, Table 3):¹

colorless oil

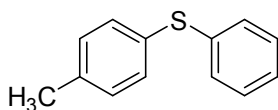
¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.42 (d, 2H, J = 8.0 Hz), 7.23 – 7.19 (m, 2H), 7.16 – 7.12 (m, 3H), 6.82 (d, 2H, J = 8.0 Hz), 3.78 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 159.7, 138.6, 135.3, 128.8, 128.1, 125.6, 124.2, 114.9, 55.2



phenyl(*o*-tolyl)sulfane(entries 7, Table 3):⁶

colorless oil

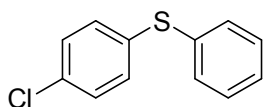
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.06 – 7.23 (m, 9H), 2.51(s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 139.7, 136.0, 132.7, 130.6, 130.1, 129.3, 127.7, 127.1, 126.6, 21.1



phenyl(*p*-tolyl) sulfane(entries 8, 26, Table 3):¹

colorless oil

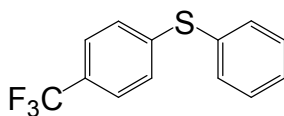
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.28 (d, 2H, *J* = 8.0 Hz), 7.25 – 7.14 (m, 5H), 7.10 (d, 2H, *J* = 8.0 Hz), 2.32 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 137.5, 137.0, 132.2, 131.2, 130.0, 129.7, 129.0, 126.3, 21.1



(4-chlorophenyl)(phenyl)sulfane(entry 10, Table 3):⁴

colorless oil

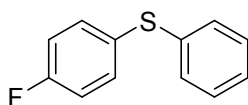
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.40 – 7.22 (m, 7H), 7.16 (d, 2H, *J* = 8.5 Hz); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 135.4, 134.7, 132.1, 132.0, 131.4, 129.3, 127.5



(4-trifluoromethylphenyl)(phenyl)sulfane (entry 11, Table 3):⁸

colorless oil

¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.47 – 7.43 (m, 4H), 7.38 – 7.31 (m, 3H), 7.22 (d, 2H, *J* = 7.6 Hz); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 142.8, 133.5, 132.5, 129.6, 128.6, 128.2, 127.8, 125.7, 122.2



(4-fluorophenyl)(phenyl)sulfane (entry 12, 30 Table 3):¹

colorless oil

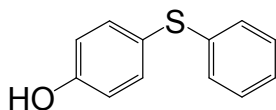
¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.36 – 7.30 (m, 2H), 7.26 – 7.13 (m, 5H), 6.99 (t, 2H, J = 8.3 Hz); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 163.9, 160.6, 136.6, 134.0, 133.9, 130.1, 129.8, 129.1, 126.7, 116.5, 116.2.



(2-nitrophenyl)(phenyl)sulfane(entry 13, Table 3):

colorless oil

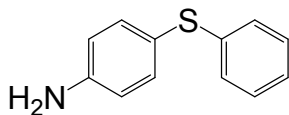
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.44 – 7.41 (m, 1H), 7.23 – 7.16 (m, 3H), 7.09 – 7.01 (m, 3H), 6.75 – 6.69 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 148.8, 137.4, 136.7, 131.1, 129.0, 126.3, 125.3, 118.7, 115.3; Mass (ESI): m/z 254 [M+Na]; Anal calcd for(C₁₂H₉NO₂S) C: 62.32, H: 3.92, S: 13.86; found C: 62.25, H: 3.84, S: 13.75



4-(phenylthio)phenol (entry 14, Table 3):¹

colorless oil

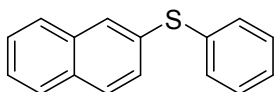
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.39 (d, 2H, J = 8.05), 7.31 – 7.24 (m, 2H), 7.20 – 7.12 (m, 3H), 6.83 (d, 2H, J = 8.05), 4.90 (bs, 1H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 155.8, 138.1, 135.0, 128.5, 127.8, 125.7, 124.5, 116.4



4-(phenylthio)benzenamine (entry 15, Table 3):¹

white solid, mp 89-90 °C

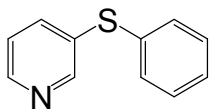
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.31 – 7.24 (m, 2H), 7.17 – 7.13 (m, 2H), 7.06 – 6.99 (m, 3H), 6.59 (d, 2H, J = 8.05) 3.69 (bs, 2H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 147.1, 139.7, 136.2, 128.7, 127.7, 125.4, 121.1, 116.0



(naphthalen-6-yl)(phenyl)sulfane(entries 16, 34, Table 3):⁵

white solid mp: 49-50 °C

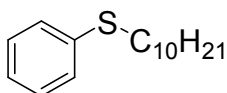
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.96 – 7.62 (m, 4H), 7.58 – 7.05 (m, 8H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 135.8, 133.7, 132.9, 132.2, 130.8, 129.8, 129.1, 128.8, 128.7, 127.6, 127.3, 127.0, 126.5, 126.1



3-(phenylthio)pyridine(entry 17, Table 3):¹⁰

colorless oil

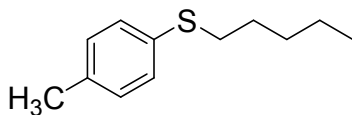
¹H NMR (200 MHz, CDCl₃, TMS): δ = 8.53 – 8.43 (m, 2H), 7.53 (d, 1H, J = 7.32 Hz), 7.35 – 7.23 (m, 5H), 7.17 (dd, 1H, J = 5.17, 2.91 Hz); **NMR** (100 MHz, CDCl₃, TMS): δ = 148.4, 136.8, 134.6, 129.7, 129.6, 128.7, 128.2, 125.6, 121.4



decyl(phenyl)sulfone(entries 18, Table 3):

colorless oil

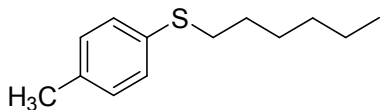
IR (KBr): ν 3019, 2911, 2932, 1668, 1560, 1234, 1059, 931 cm⁻¹; **¹H NMR** (200 MHz, CDCl₃, TMS): δ = 7.31 – 7.04 (m, 5H), 2.87 (t, 2H, J = 7.0), 1.70 (p, 2H, J = 7.0Hz), 1.44 – 1.12 (m, 14H), 0.88 (t, 3H, J = 7.0); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 137.4, 129.4, 125.9, 33.6, 31.9, 29.5, 29.3, 29.2, 28.8, 22.7, 14.1; **Mass** (ESI): m/z 273[M+Na]; **Anal** calcd for: (C₁₆H₂₆S) C: 76.73, H: 10.46, S: 12.80
found C: 76.65, H: 10.39, S: 11.99



pentyl(p-tolyl) sulfane(entry 19, Table 3):

colorless oil

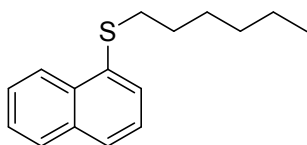
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.17 (d, 2H, J = 8.5 Hz), 7.02 (d, 2H, J = 8.5 Hz), 2.82 (t, 2H, J = 7.5 Hz), 2.31 (s, 3H), 1.60 (p, 2H, J = 7.0 Hz), 1.43 – 1.25 (m, 4H), 0.89 (t, 3H, J = 7.0 Hz); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 135.7, 133.1, 129.7, 129.5, 34.3, 30.9, 28.9, 22.2, 20.9, 13.9; **Mass** (ESI): m/z 217 [M+Na]; **Anal** calcd for: (C₁₂H₁₈S) C: 74.16, H: 9.34, S: 16.50; **found** C: 74.07, H: 9.25, S: 16.41



hexyl(p-tolyl)sulfane(entry 20, Table 3):¹

colorless oil

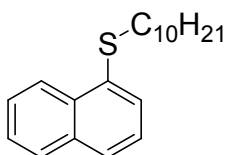
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.15 (d, 2H, J = 7.5 Hz), 6.99 (d, 2H, J = 7.5 Hz), 2.80 (t, 2H, J = 7.0 Hz), 2.29 (s, 3H), 1.64 – 1.51 (m, 2H), 1.45 – 1.16 (m, 6H) 0.86 (t, 3H, J = 7.0 Hz); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 135.77, 133.14, 131.04, 129.74, 129.58, 34.35, 31.37, 29.23, 28.50, 22.55, 20.98, 14.02



hexyl(naphthalen-5-yl)sulfane(entry 21, Table 3):

colorless oil

IR (KBr): ν 3053, 2954, 2924, 1641, 1563, 1255, 1022, 974, 665 cm⁻¹; ¹H NMR (300 MHz, CDCl₃, TMS): δ = 8.34 (d, 1H, J = 8.5 Hz), 7.79 (d, 1H, J = 7.5 Hz), 7.67 (d, 1H, J = 7.5 Hz), 7.48 (q, 3H, J = 7.0, 7.5 Hz), 7.35 (t, 1H, J = 7.5 Hz), 2.93 (t, 2H, J = 7.5 Hz), 1.65 (p, 2H, J = 7.0, 7.5 Hz), 1.49- 1.20 (m, 6H), 0.88 (t, 3H, J = 7.0 Hz); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 134.2, 133.8, 132.8, 128.4, 127.2, 126.7, 126.19, 126.1, 125.5, 124.9, 34.1, 31.3, 29.1, 28.5, 22.5, 14.0; Mass (ESI): m/z 267 [M+Na]; Anal calcd for: (C₁₆H₂₀S) C: 78.63, H: 8.25, S: 13.12; found C: 78.52, H: 8.16, S: 13.03

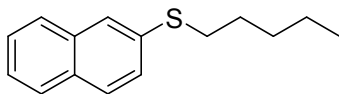


decyl(naphthalen-5-yl)sulfane(entry 22, Table 3):

colorless oil

IR (KBr): ν 3045, 2953, 2920, 1633, 1543, 1262, 1011, 984, 675 cm⁻¹; ¹H NMR (200 MHz, CDCl₃, TMS): δ = 8.36 (d, 1H, J = 8.3 Hz), 7.76 (d, 1H, J = 7.0 Hz), 7.66 (d, 1H, J = 8.0 Hz), 7.52 – 7.39 (m, 3H), 7.34 (t, 1H, J = 8.0 Hz), 2.92 (t, 2H, J = 7.0 Hz), 1.64 (p, 2H, J = 7.0 Hz), 1.49 – 1.37 (m, 2H), 1.34 – 1.13 (m, 12H), 0.87 (t, 3H, J = 6.5 Hz); ¹³C NMR (100 MHz, CDCl₃, TMS): δ = 134.2, 133.8, 132.8, 128.4, 127.3, 126.7, 126.19, 126.11, 125.5, 124.9, 34.1, 31.8, 29.5, 29.3, 29.18, 29.12, 28.8, 22.6, 14.1; Mass (ESI):

m/z 323[M+Na]; Anal calcd for: (C₂₀H₂₈S) C: 79.94, H: 9.39, S: 10.67; found C: 79.83, H:9.31, S:10.58



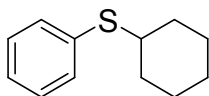
(naphthalene-6-yl)(pentyl)sulfane (entry 23, Table 3):

colorless oil

IR (KBr): ν 3043, 2972, 2914, 1632, 1541, 1225, 1018, 931 cm⁻¹

¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.73 -7.66 (m, 4H), 7.42 – 7.33 (m, 3H), 2.97 (t, 2H, J = 7.5 Hz), 1.68 (p, 2H, J = 7.0 Hz), 1.48 – 1.24 (m, 4H), 0.90 (t, 3H, J = 7.5 Hz);

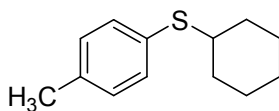
¹³C NMR (100 MHz, CDCl₃, TMS): δ =134.6, 133.7, 131.5, 128.2, 127.6, 127.1, 126.9, 126.4, 126.1, 125.4, 33.3, 31.0, 28.7, 22.2, 13.9; Mass (ESI): m/z 253 [M+Na]; Anal calcd for: (C₁₅H₁₈S) C: 78.21, H: 7.88, S: 13.92; found C: 78.12, H: 7.79, S: 13.81



cyclohexyl(phenyl)sulfane(entry 24, Table 3):⁶

colorless oil

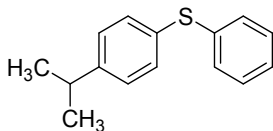
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.38 (d, 2H, J = 8.0 Hz), 7.30 – 7.17 (m, 3H), 3.14 – 3.05 (m, 1H), 2.01 – 1.92 (m, 2H), 1.81 – 1.74 (m, 2H), 1.65 – 1.57 (m, 1H), 1.44 – 1.20 (5H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 135.1, 131.8, 128.7, 126.5, 46.5, 33.3, 26.0, 25.7



cyclohexyl(p-tolyl)sulfane(entry 25, Table 3):¹

colorless oil

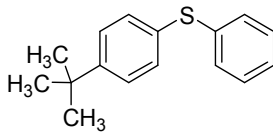
¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.23 (d, 2H, J = 8.0 Hz), 7.02 (d, 2H, J = 8.0 Hz), 3.01 – 2.85 (m, 1H), 2.32 (s, 3H), 1.98 – 1.51 (m, 5H), 1.42 – 1.20 (m, 5H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ =136.7, 132.7, 131.1, 129.4, 47.0, 33.3, 26.0, 25.7, 21.0



(4-isopropylphenyl)(phenyl)sulfane(entry 27, Table 3):

colorless oil

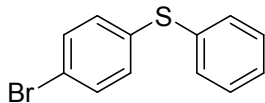
IR (KBr): ν 3045, 2931, 2921, 1671, 1561, 1232, 1067, 971 cm^{-1} ; **^1H NMR** (200 MHz, CDCl_3 , TMS): δ = 7.44 – 7.11 (m, 9H), 3.03 – 2.76 (m, 1H), 1.28 (d, 6H, J = 7.0 Hz); **^{13}C NMR** (100 MHz, CDCl_3 , TMS): δ = 148.3, 136.8, 131.9, 131.7, 130.0, 129.0, 128.1, 127.3, 127.1, 126.4, 33.7, 23.8 Mass (ESI): m/z 251[M+Na]; Anal calcd for: $\text{C}_{15}\text{H}_{16}\text{S}$ (228) C: 78.90, H: 7.06, S: 14.04; found C: 78.81, H: 6.99, S: 13.97



(4-tert-butylphenyl)(phenyl)sulfane(entry 28, Table 3):⁹

colorless oil

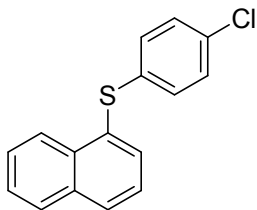
^1H NMR (300 MHz, CDCl_3 , TMS): δ = 7.35 – 7.08 (m, 9H), 1.31 (s, 9H); **^{13}C NMR** (100 MHz, CDCl_3 , TMS): δ = 150.5, 136.7, 131.4, 130.4, 130.2, 129.0, 126.7, 126.2, 35.0, 31.



(4-bromophenyl)(phenyl)sulfane(entry 29, Table 3):³

colorless oil

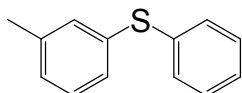
^1H NMR (200 MHz, CDCl_3 , TMS): δ = 7.38 – 7.20 (m, 9H); **^{13}C NMR** (100 MHz, CDCl_3 , TMS): δ = 135.0, 134.6, 132.9, 131.9, 131.2, 129.2, 127.4



(4-chlorophenyl)(naphthalen-5-yl)sulfane(entries 32, 33, Table 3):⁷

colorless oil

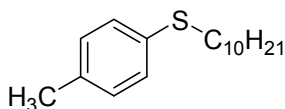
¹H NMR (200 MHz, CDCl₃, TMS): δ = 8.31 (dd, 1H, *J* = 3.0 Hz), 7.87 – 7.84 (m, 2H), 7.68– 7.66 (m, 1H), 7.49 (dd, 2H, *J* = 3.0, 3.7 Hz), 7.42 (t, 1H, *J* = 8.5 Hz), 7.14(d, 2H, *J* = 9.0 Hz), 7.04(d, 2H, *J* = 9.0 Hz); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 135.7, 134.2, 133.5, 133.0, 131.8, 130.3, 129.7, 129.6, 129.1, 128.6, 127.1, 126.5, 125.8, 125.4



phenyl(*m*-tolyl) sulfane(entry 35, Table 3):⁸

colorless oil

¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.49 – 7.42 (m, 4H), 7.39 – 7.29 (m, 4H), 7.19 (m, 1H), 2.51 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 138.9, 136.0, 135.1, 131.7, 130.6, 129.0, 128.9, 128.2, 127.9, 126.7, 21.2

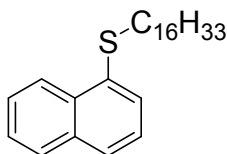


decyl(*p*-tolyl)sulfane(entry 39, Table 3):

colorless oil

IR (KBr): ν 3049, 2941, 2913, 1680, 1571, 1242, 1079, 974 cm⁻¹

¹H NMR (200 MHz, CDCl₃, TMS): δ = 7.17 (d, 2H, *J* = 8.0 Hz), 7.01 (d, 2H, *J* = 8.0 Hz), 2.82 (t, 2H, *J* = 7.5 Hz), 2.31 (s, 3H), 1.59 (p, 2H, *J* = 7.5 Hz), 1.42 – 1.17 (m, 14H), 0.88 (t, 3H, *J* = 7.0 Hz); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ = 135.7, 133.1, 129.7, 129.5, 34.3, 31.8, 29.5, 29.3, 29.2, 29.1, 28.8, 22.6, 20.9, 14.1; Mass (ESI): *m/z* 287 [M+Na]; Anal calcd for: (C₁₇H₂₈S) C: 77.20, H: 10.67, S: 12.12; found C: 77.12, H: 10.59, S: 11.99

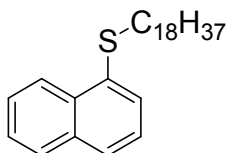


hexadecyl(naphthalen-5-yl)sulfane(entry 40, Table 3):

white solid, mp: 55-56 °C

IR (KBr) : ν 3052, 2918, 2850, 1623, 1585, 1267, 1074, 941, 736 cm⁻¹; **¹H NMR** (200 MHz, CDCl₃, TMS): δ = 8.36 (d, 1H, *J* = 8.0 Hz), 7.76 (d, 1H, *J* = 7.5 Hz), 7.64 (d, 1H, *J* = 7.5 Hz), 7.56 – 7.31 (m, 4H), 2.92 (t, 2H, *J* = 7.5 Hz), 1.64 (p, 2H, *J* = 7.0 Hz), 1.45 – 1.38 (m, 2H), 1.32 – 1.24 (m, 24H), 0.88 (t, 3H, *J* = 7.0 Hz); **¹³C NMR** (100 MHz,

CDCl₃, TMS): δ =134.2, 133.8, 132.8, 128.4, 127.3, 126.7, 126.19, 126.1, 125.5, 124.9, 34.1, 31.8, 29.5, 29.3, 29.18, 29.12, 28.8, 22.6, 14.1; Mass (ESI): m/z 407 [M+Na]; Anal calcd for: (C₂₆H₄₀S) C: 81.18, H: 10.48, S: 8.34; found C: 81.09, H:10.37, S: 8.29



(naphthalen-5-yl)(octadecyl)sulfane(entry 41, Table 3):

white solid, mp: 41-42 °C

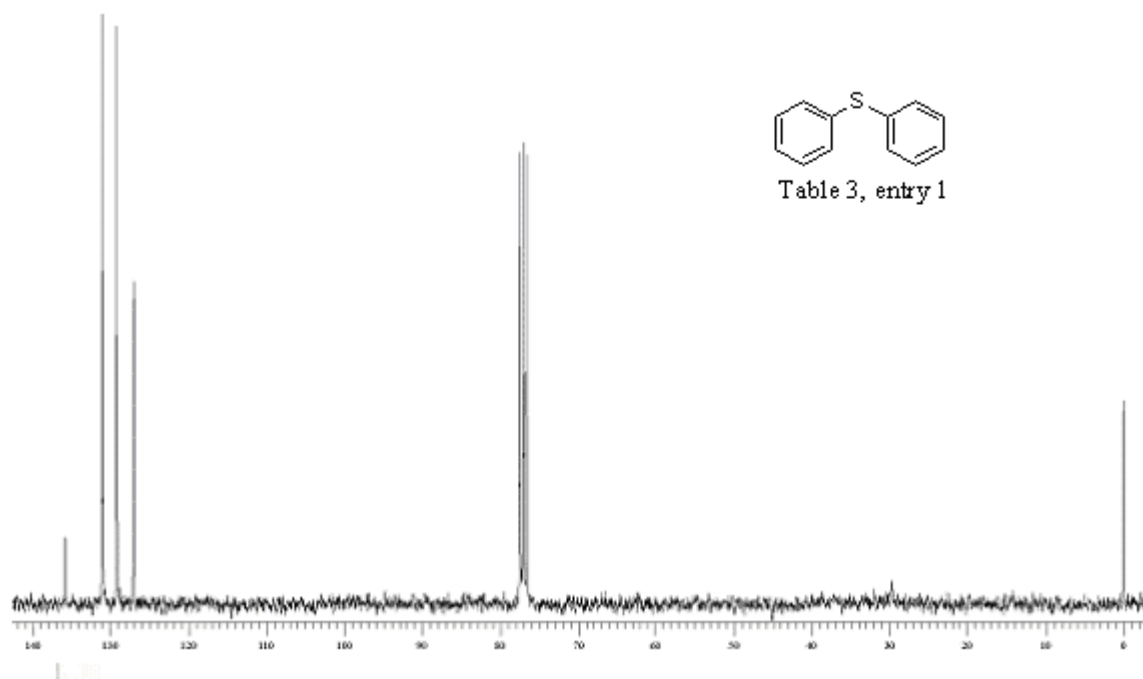
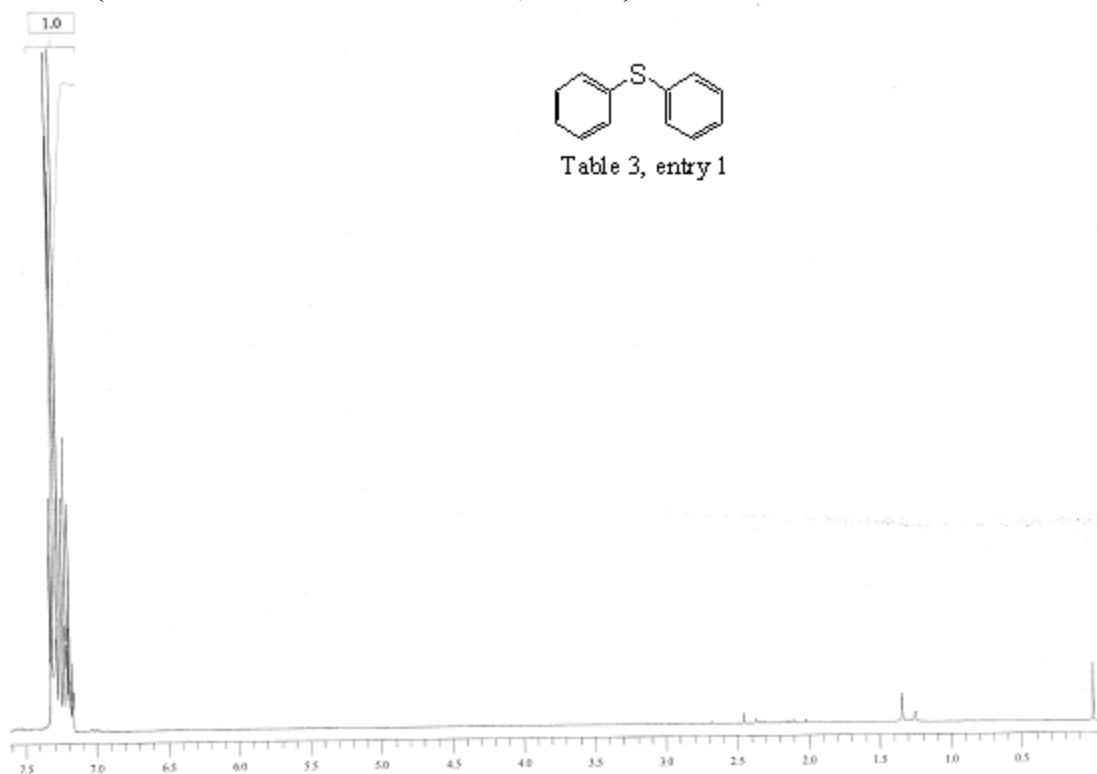
IR (KBr): ν 3055, 2955, 2925, 1648, 1492, 1213, 1017, 802 cm⁻¹

¹H NMR (300 MHz, CDCl₃, TMS): δ = 7.79 – 7.69 (m, 4H), 7.48 – 7.35 (m, 3H), 3.01 (t, 2H, J = 7.5 Hz), 1.72 (p, 2H, J = 7.0 Hz), 1.55 – 1.44 (m, 2H), 1.38 – 1.18 (m, 28H), 0.92 (t, 3H, J = 6.0 Hz); **¹³C NMR** (100 MHz, CDCl₃, TMS): δ =134.2, 133.8, 132.8, 128.4, 127.2, 126.7, 126.1, 126.0, 125.5, 124.9, 34.1, 31.9, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 28.9, 22.7, 14.1; Mass (ESI): m/z 435 [M+Na]; Anal calcd for: (C₂₈H₄₄S) C: 81.48, H: 10.75, S: 7.77; found C: 81.39, H: 10.64, S: 7.64

References:

1. Wong, Y.C.; Jayanth, T. T.; Cheng, C. H. *Org. Lett.* **2006**, 8, 5613.
2. Correa, A.; Carril, M.; Bolm, C. *Angew. Chem. Int. Ed.* **2008**, 47, 2880.
3. Rout, L.; Sen, T. K.; Punniyamurthy, T. *Angew. Chem.* **2007**, 119, 5679.
4. Rout, L.; Saha, P.; Jammi, S, Punniyamurthy, T. *Eur. J. Org. Chem.* **2008**, 640.
5. Zhang, Y.; Ngeow, K. C.; Ying, J. Y. *Org. Lett.* **2007**, 9, 3495.
6. Itoh, T.; Mase, T. *Org. Lett.* **2004**, 6, 4587.
7. Ranu, B.C.; Saha, A.; Jana, R. *Adv. Synth. Catal.* **2007**, 349, 2690.
8. Zhang, Y.; Ngeow, K. C.; Ying, J. Y. *Org. Lett.* **2007**, 9, 3495.
9. Sperotto, E.; Klink, G. P. M. V.; De Vries, J. G.; Koten, G. V. *J. Org. Chem.* **2008**, 73, 5625.
10. Kumar, S.; Engman, L. *J. Org. Chem.* **2006**, 71, 5400.

Copies of ^1H NMR and ^{13}C NMR of Compounds:
NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

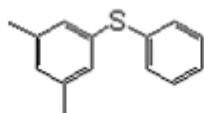


Table 3, entry 4

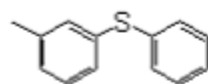
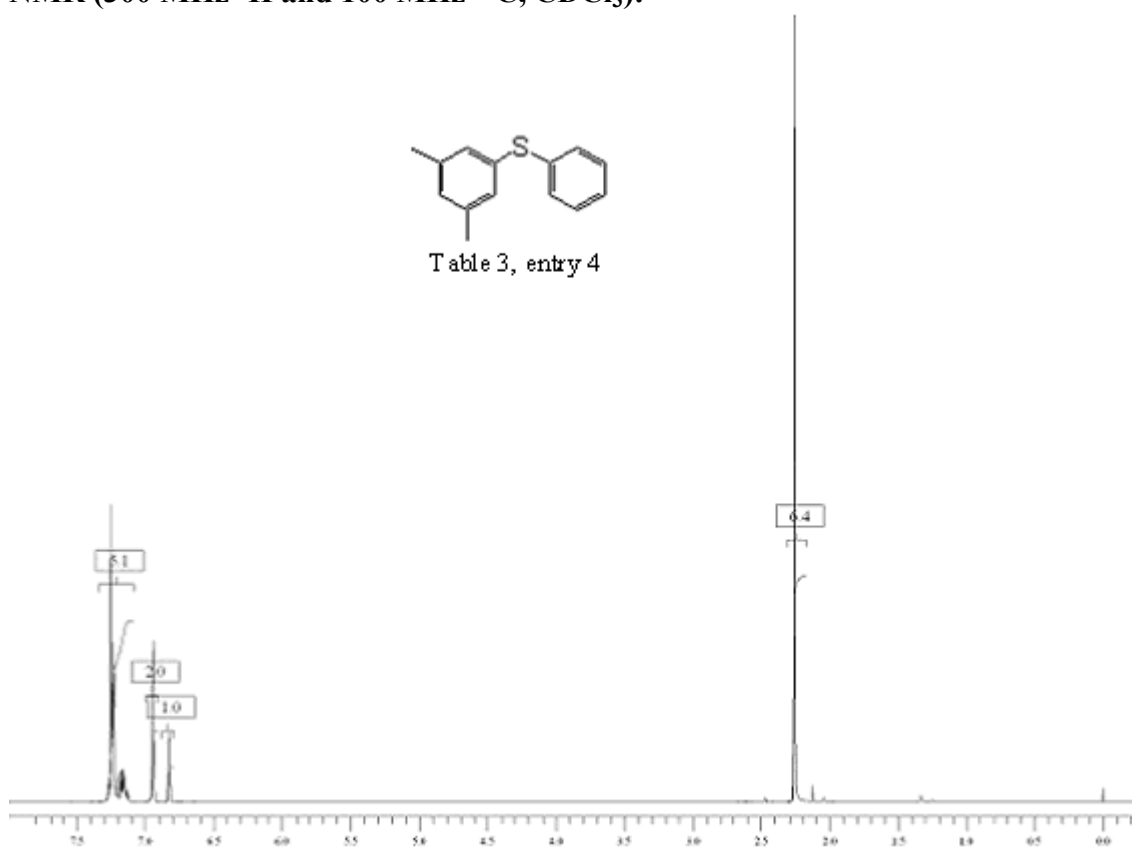
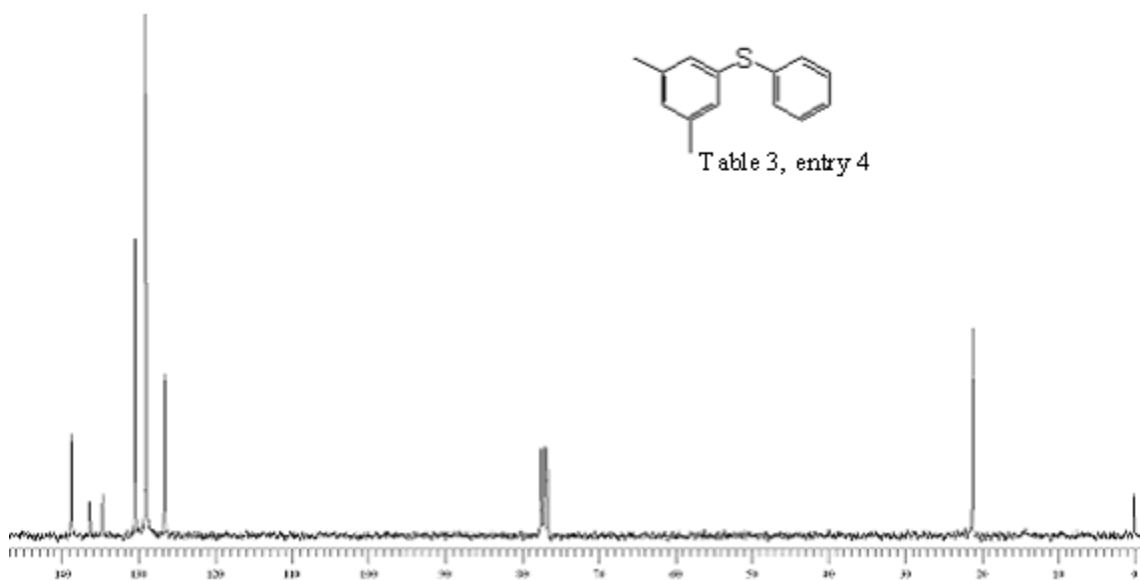
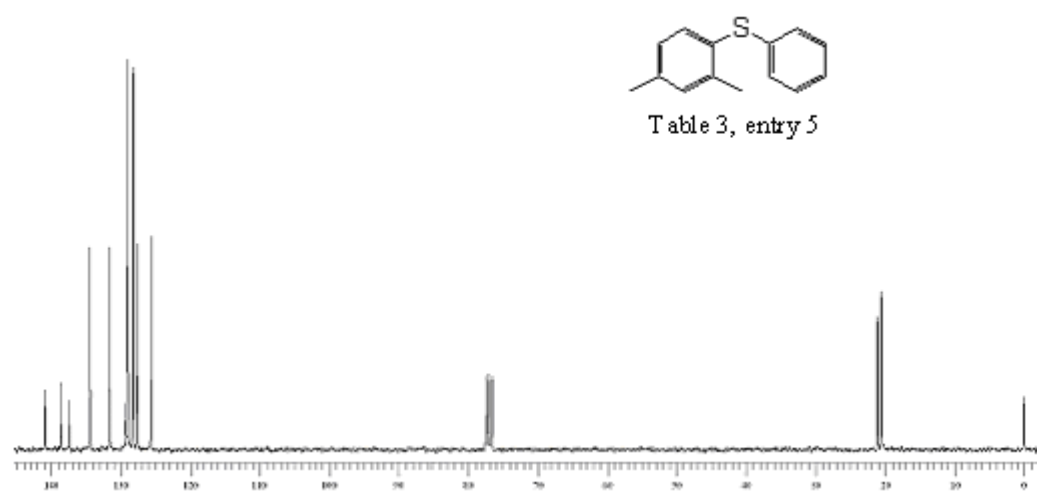
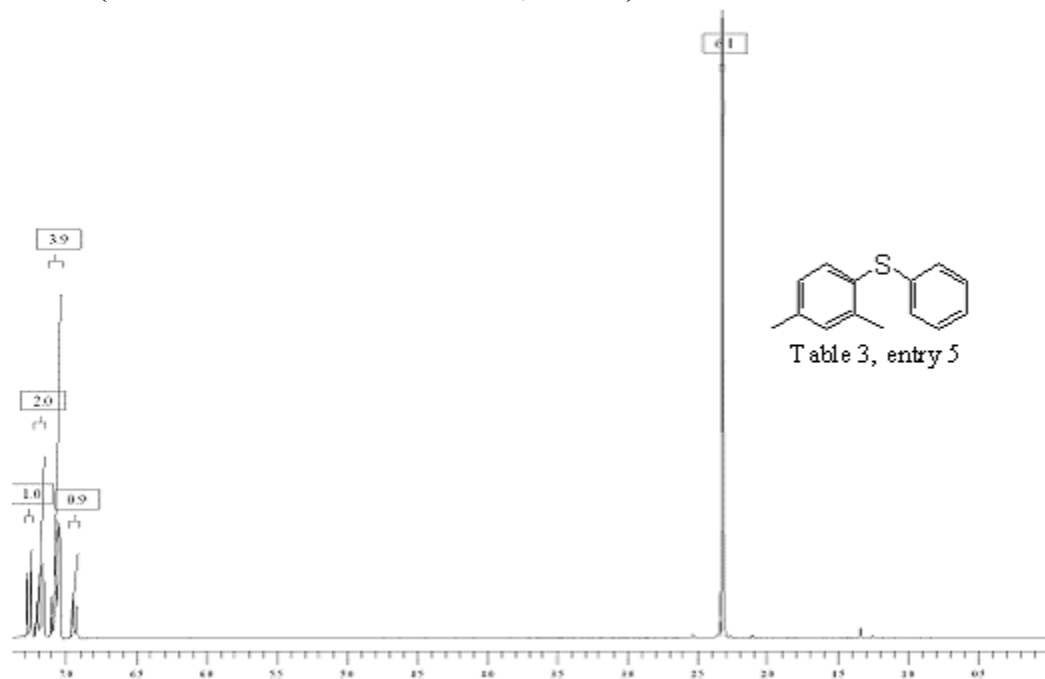


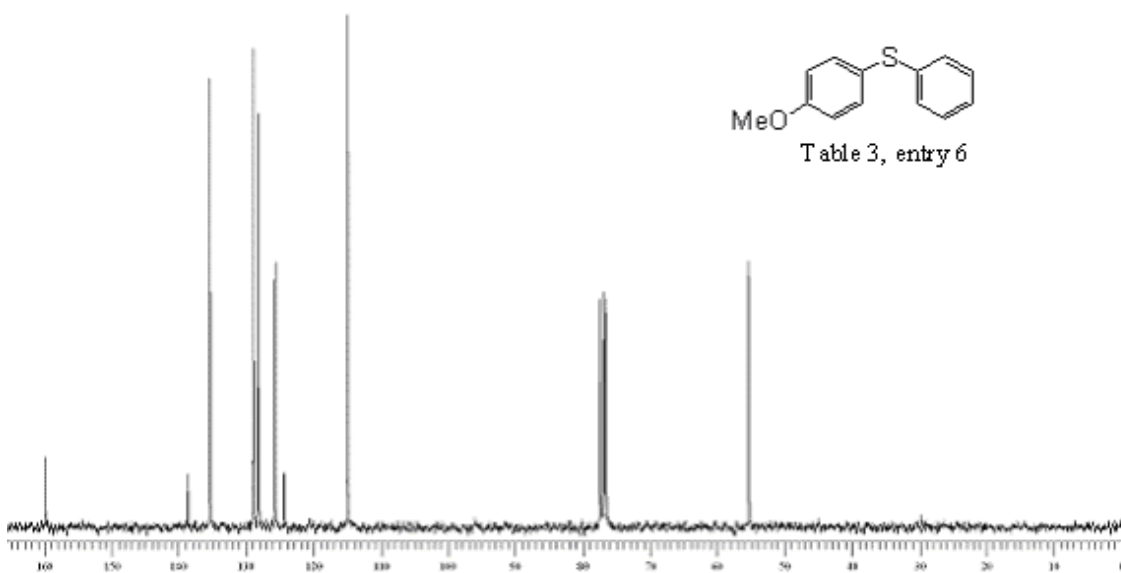
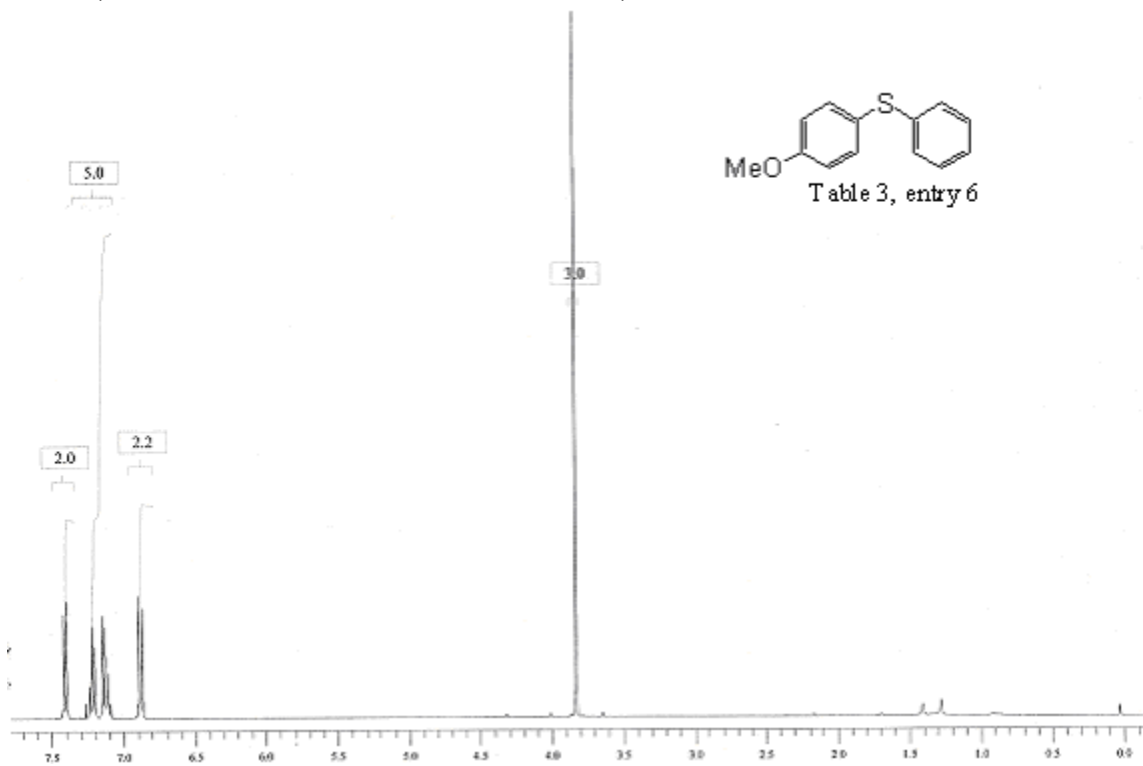
Table 3, entry 4



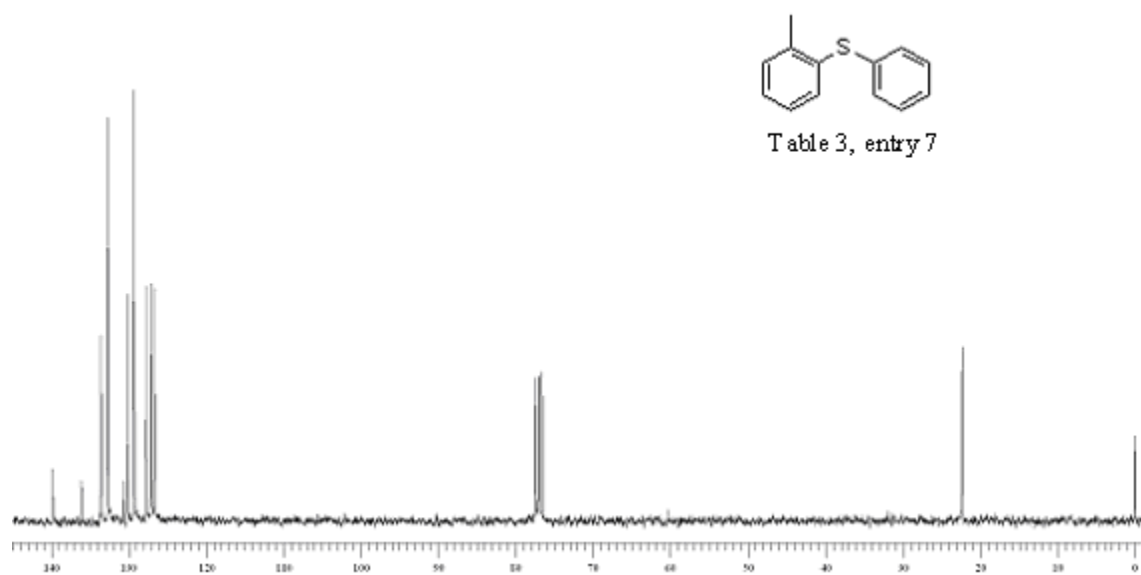
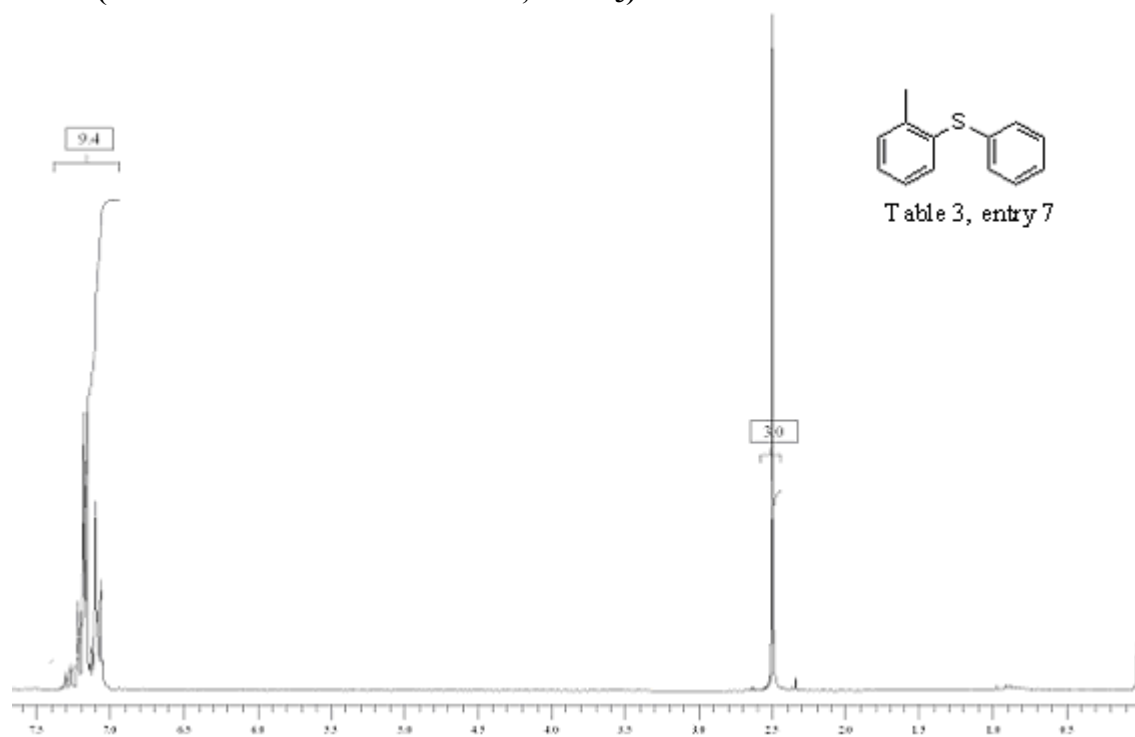
NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



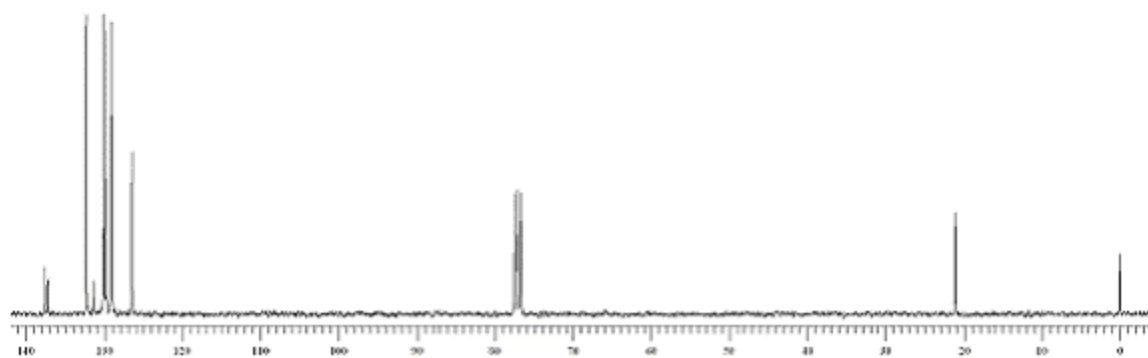
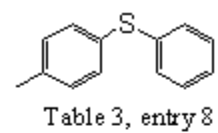
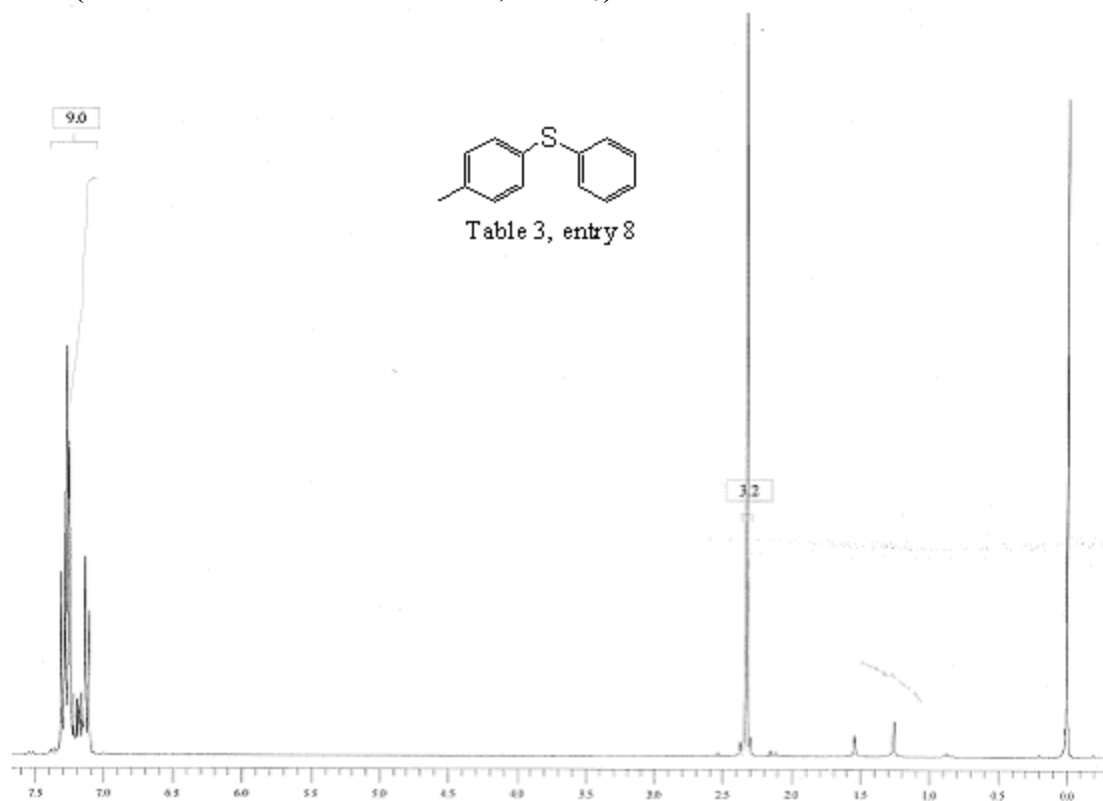
NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



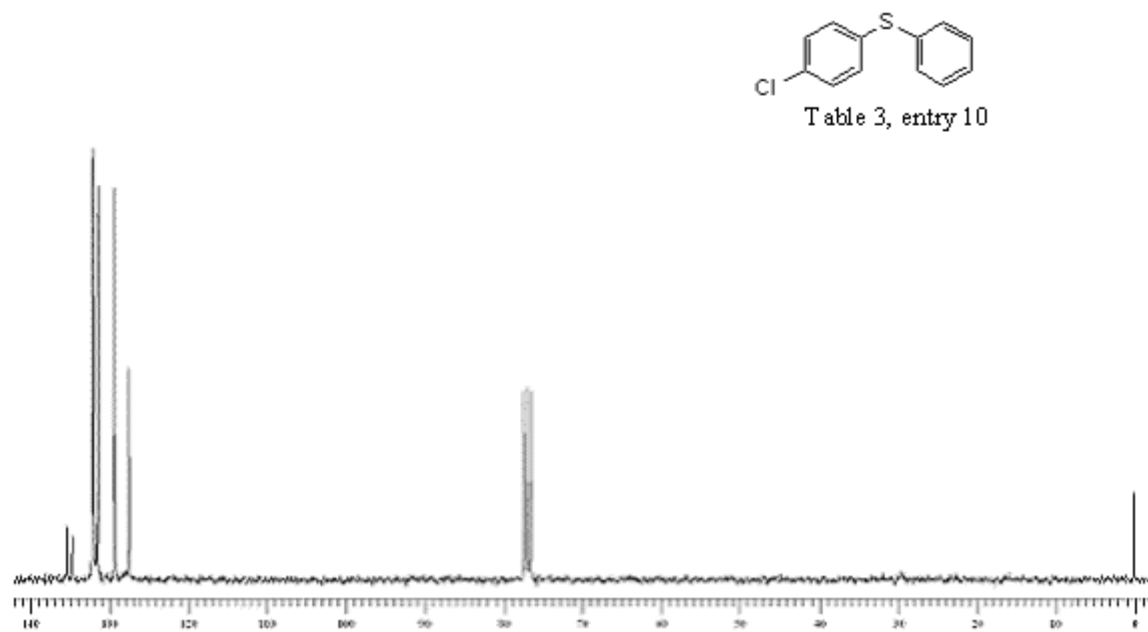
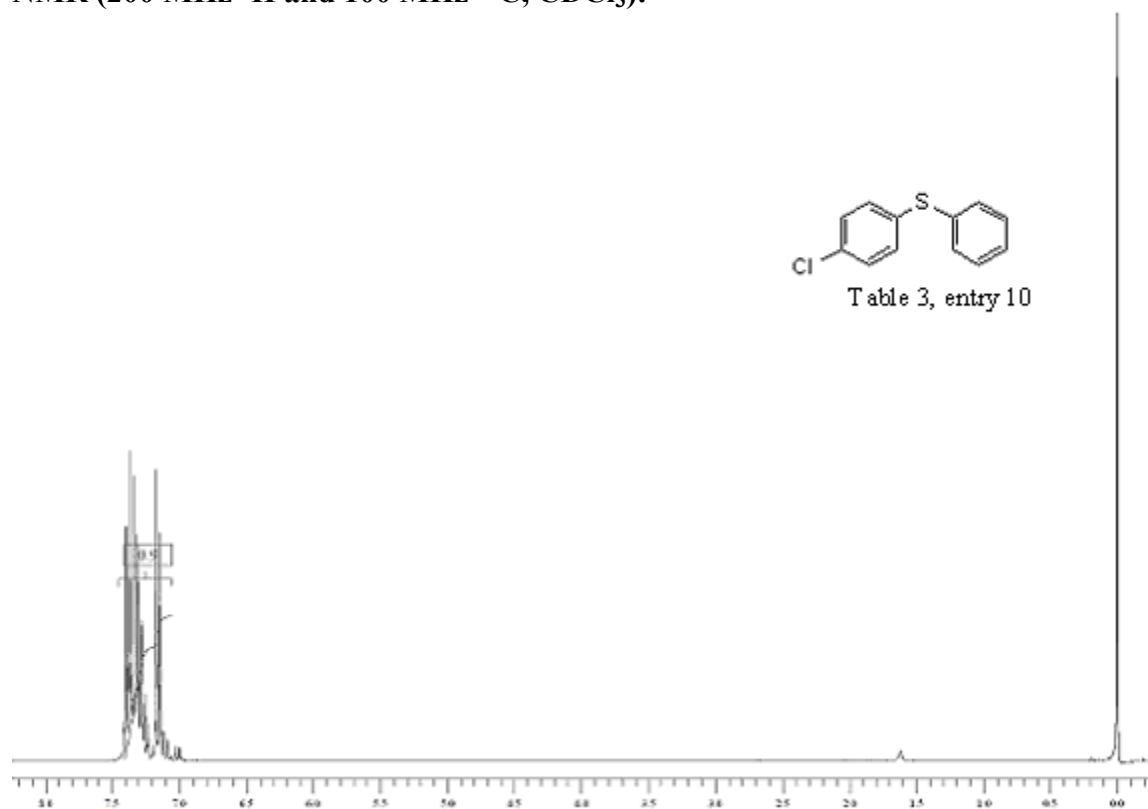
NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



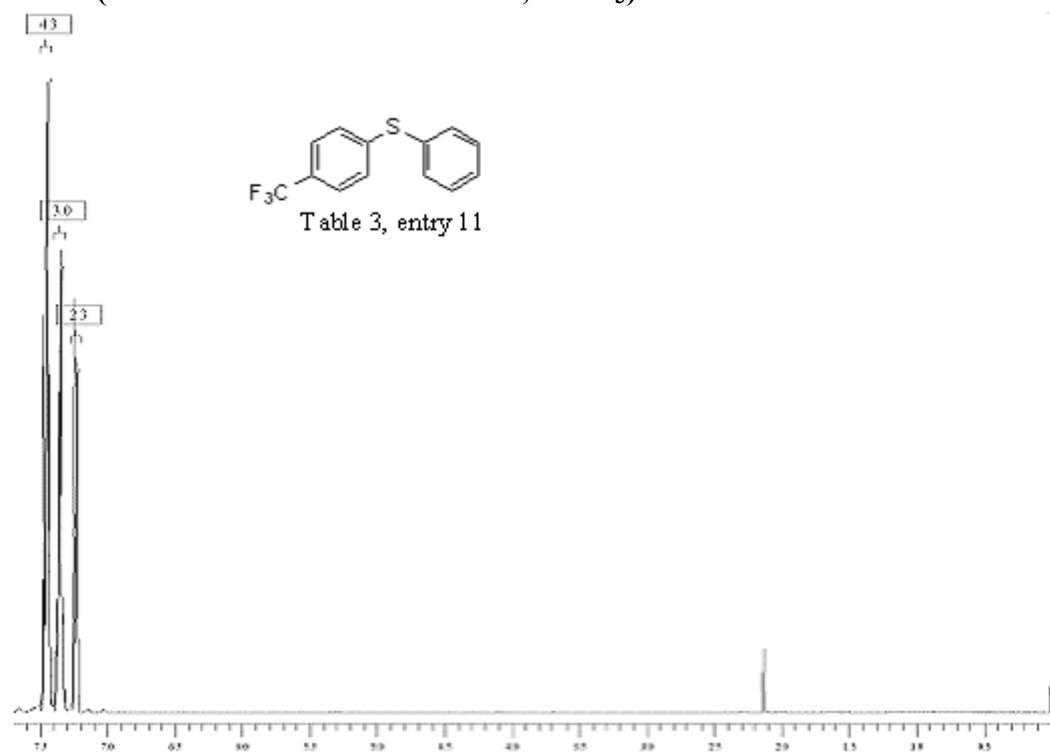
NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

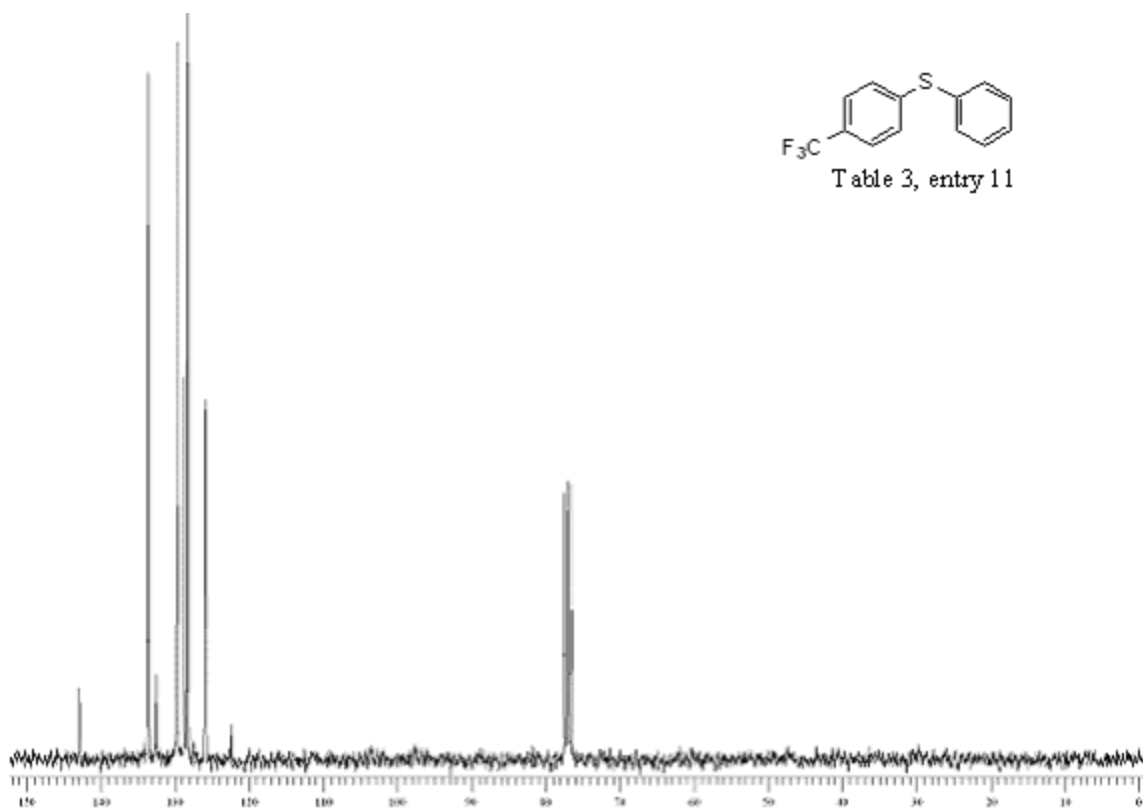


NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

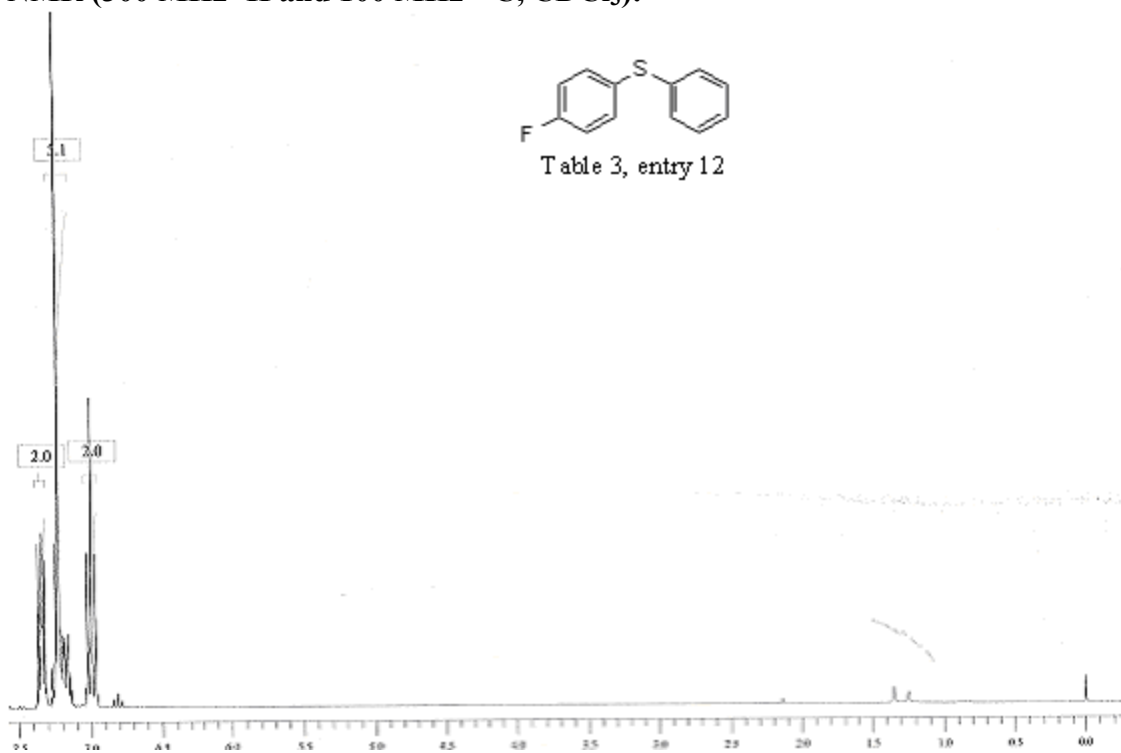


NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):





NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



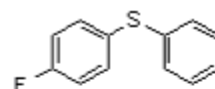
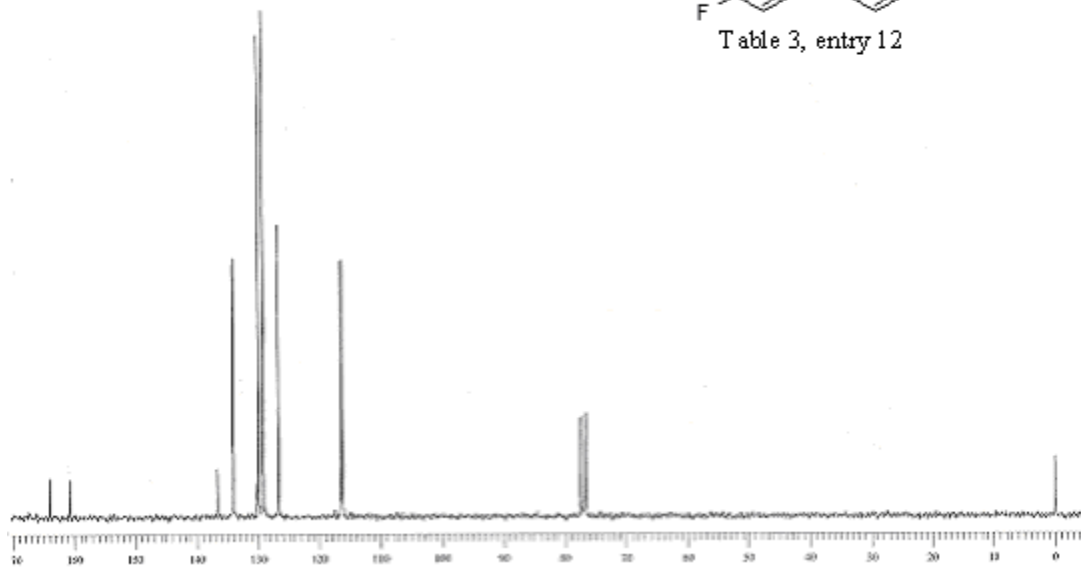


Table 3, entry 12



NMR (200 MHz ¹H and 100 MHz ¹³C, CDCl₃):

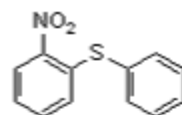
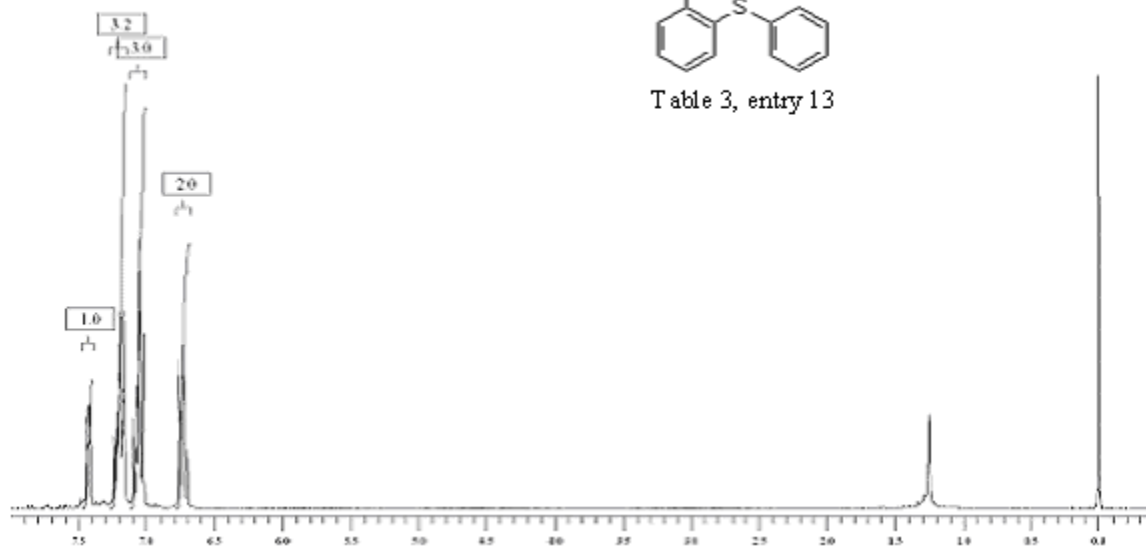
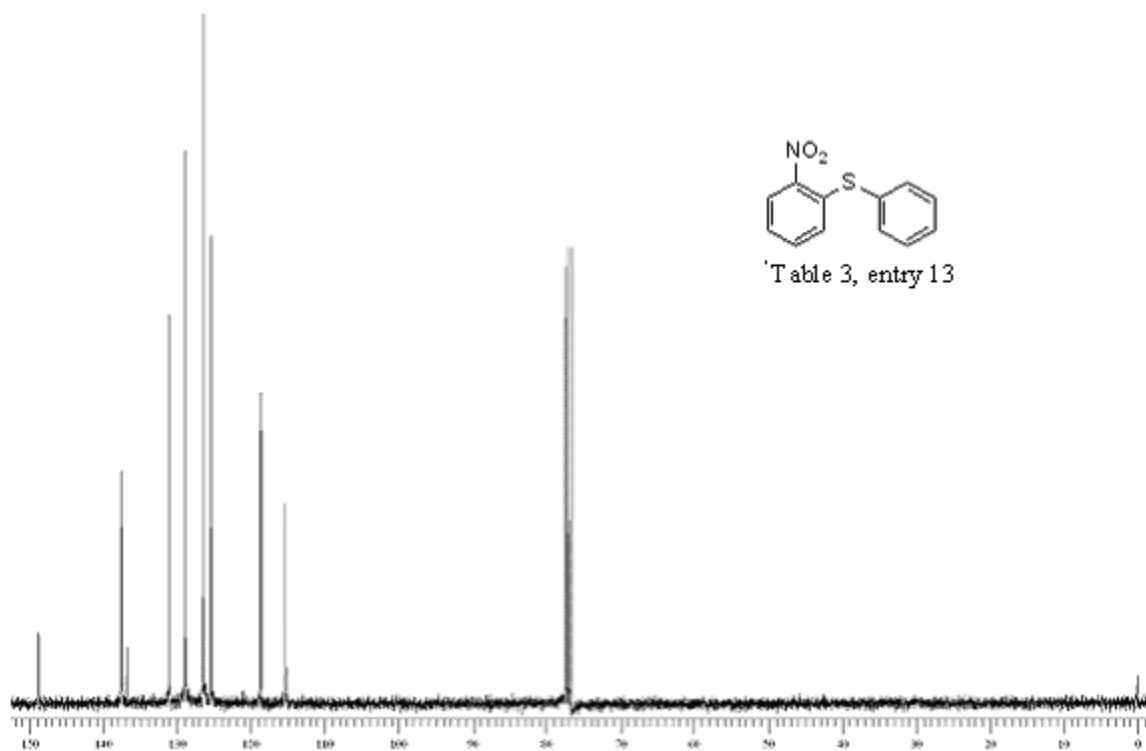
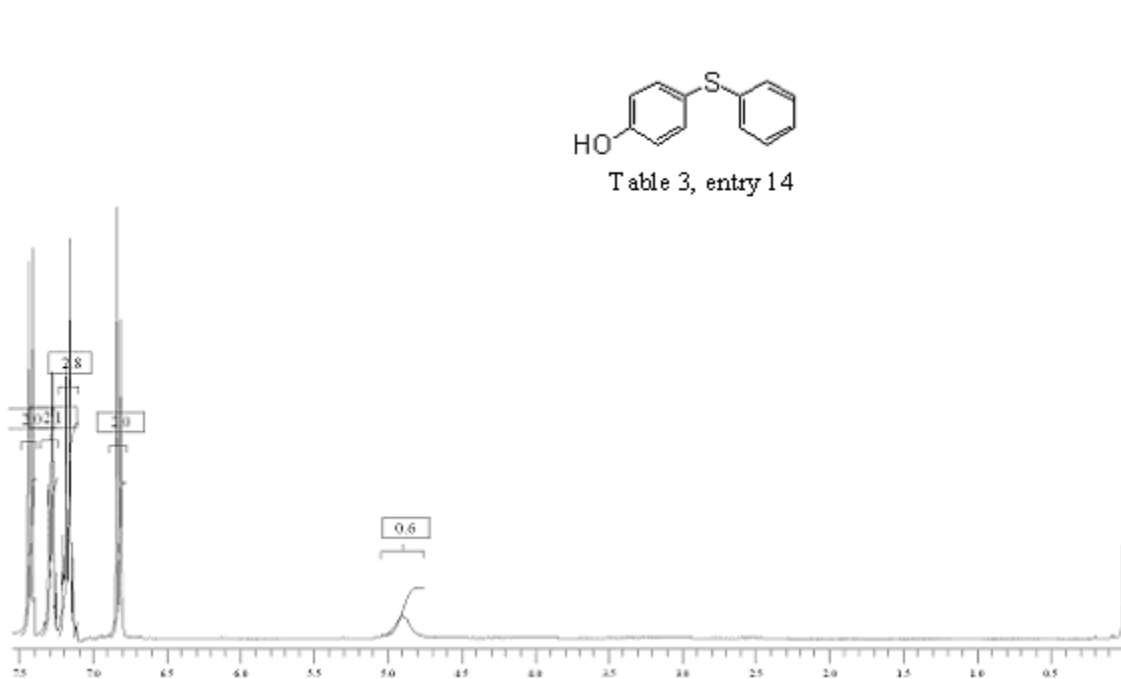


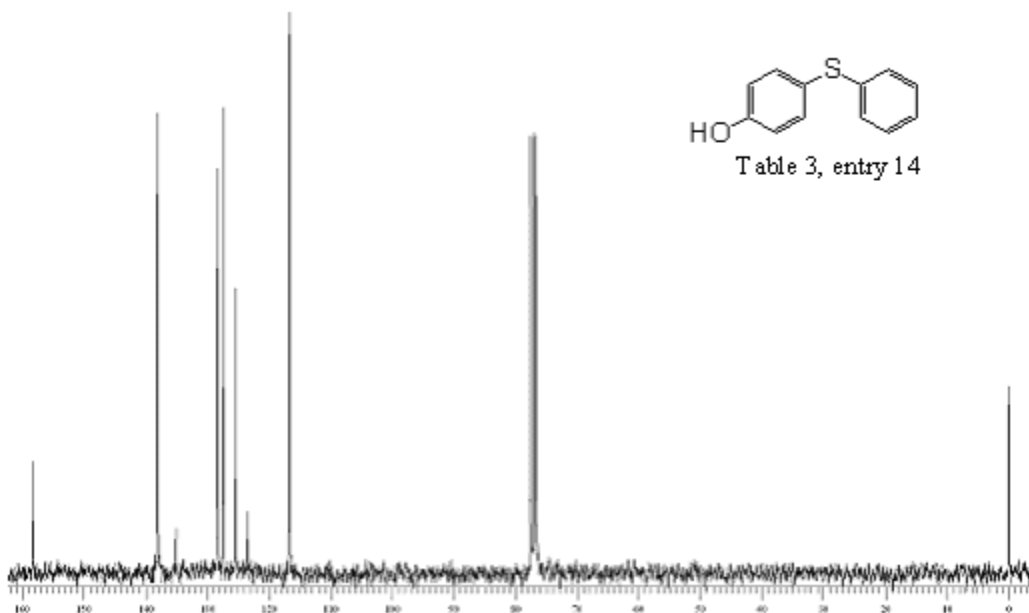
Table 3, entry 13



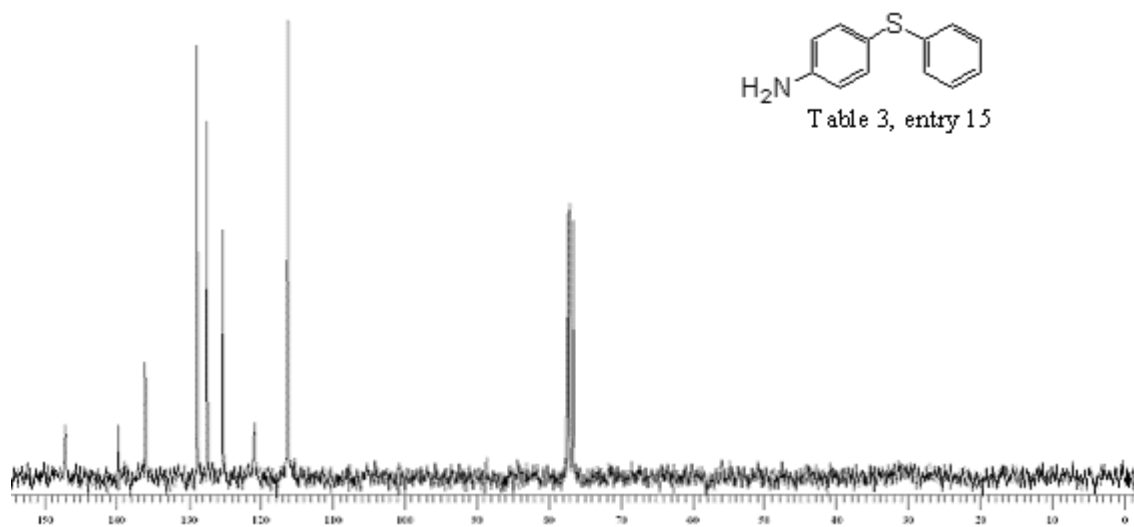
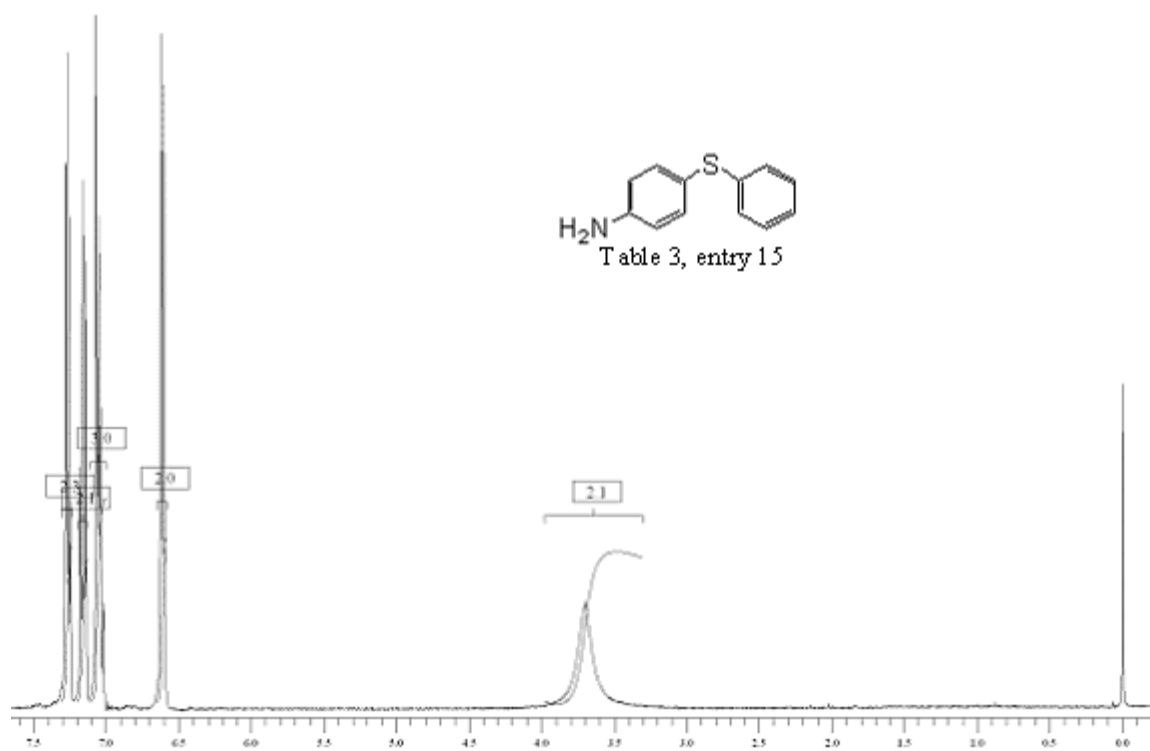


NMR (200 MHz ¹H and 100 MHz ¹³C, CDCl₃):

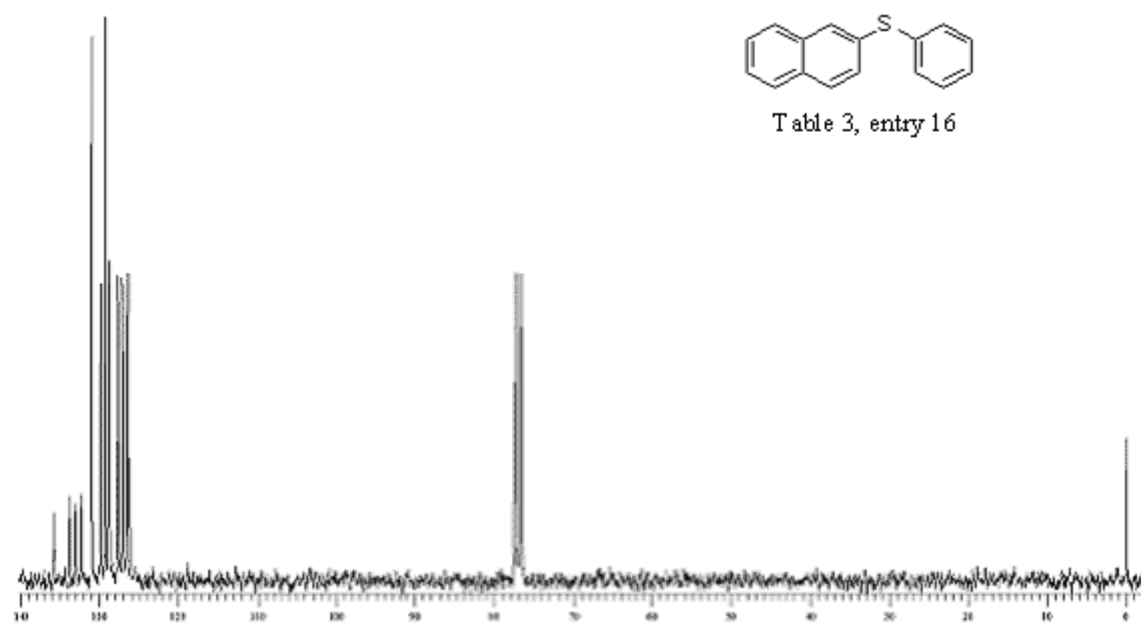
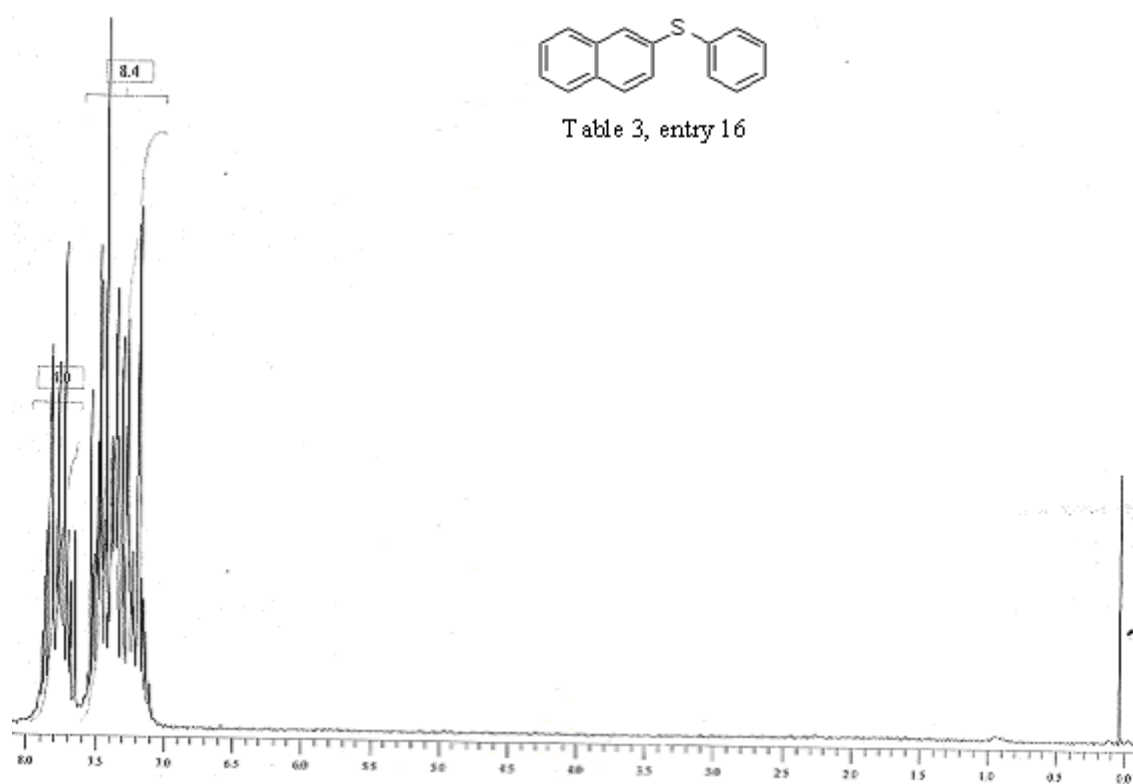




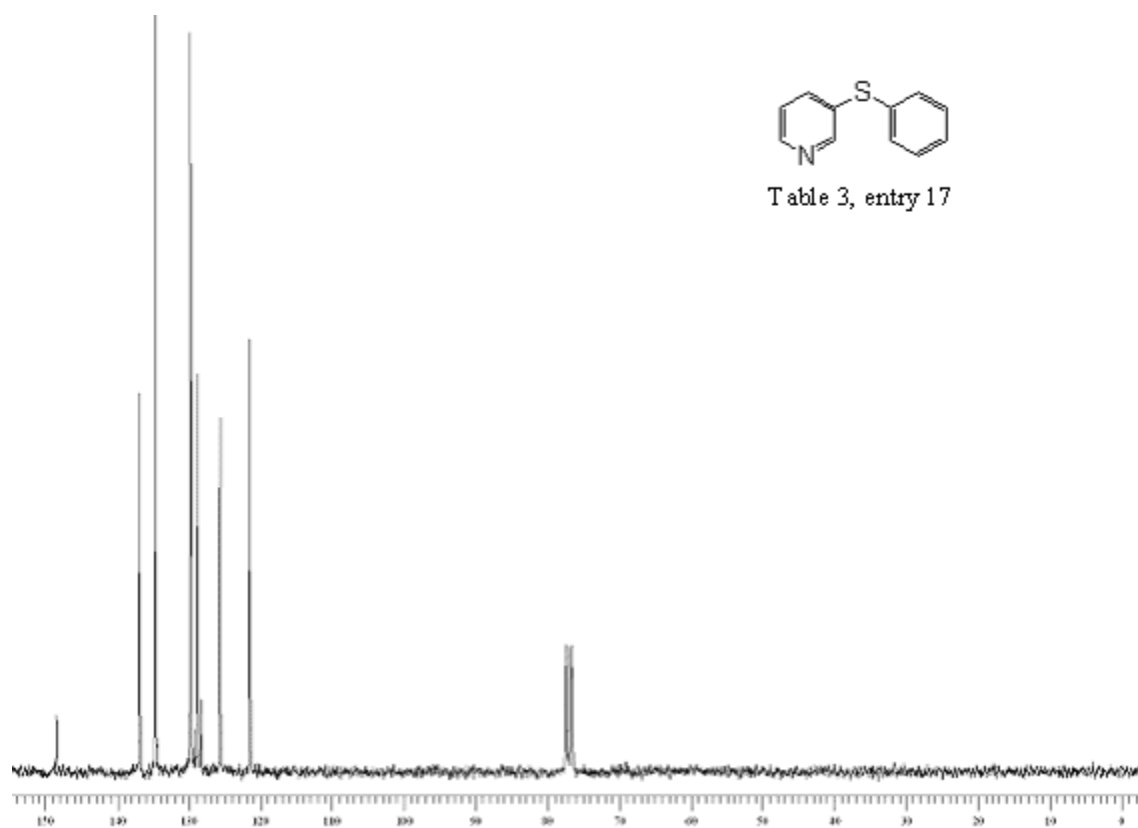
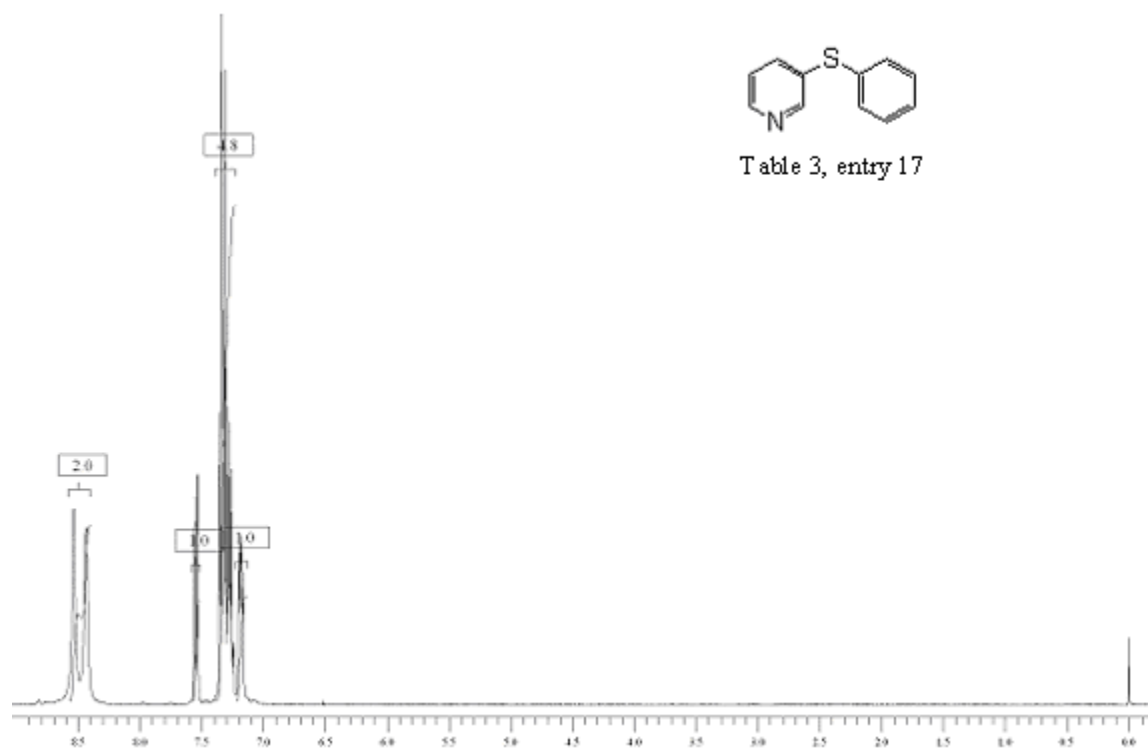
NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



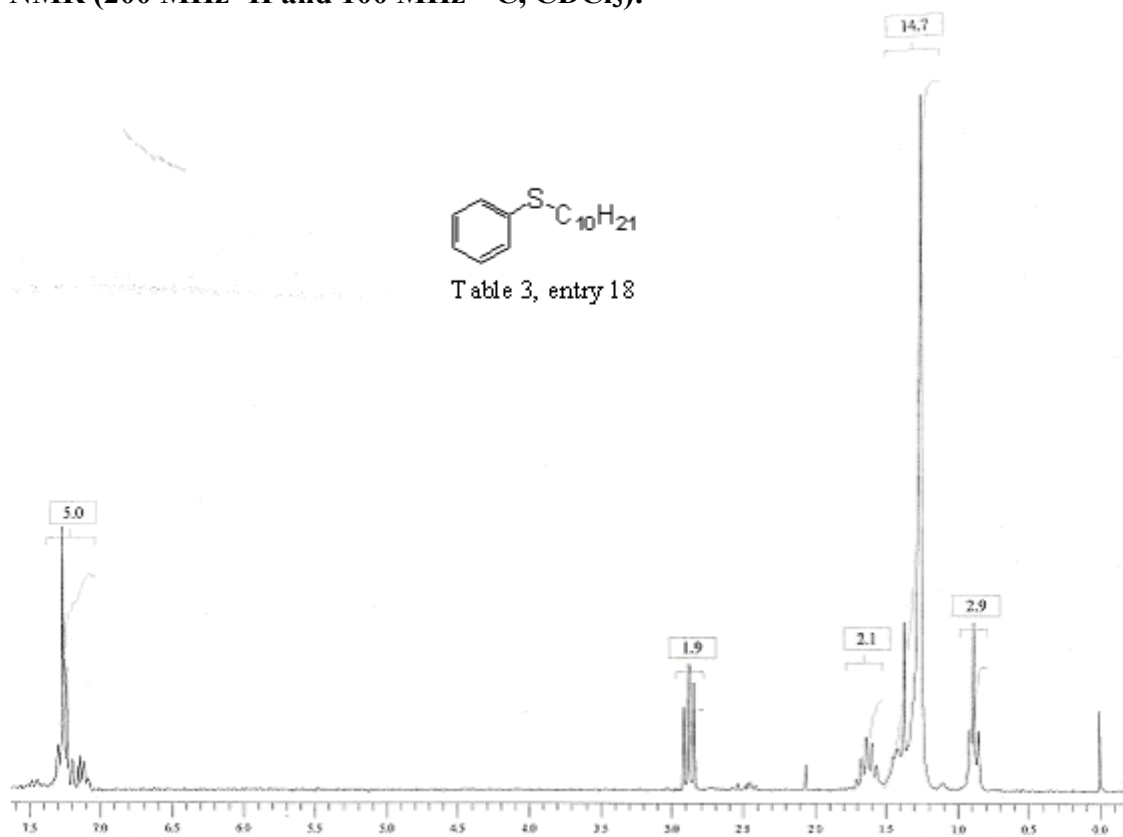
NMR (200 MHz ¹H and 100 MHz ¹³C, CDCl₃):

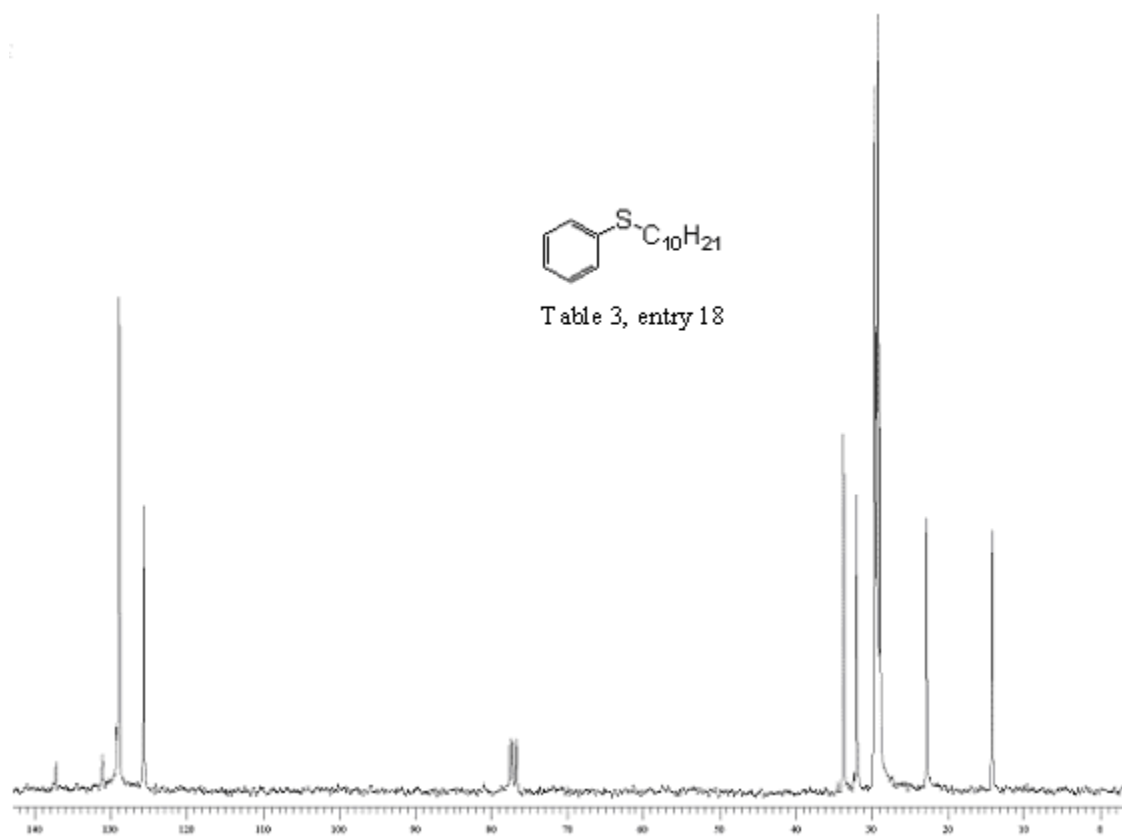


NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):





NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

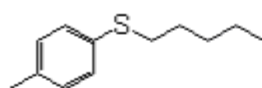


Table 3, entry 19

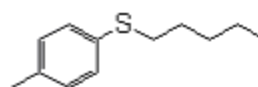
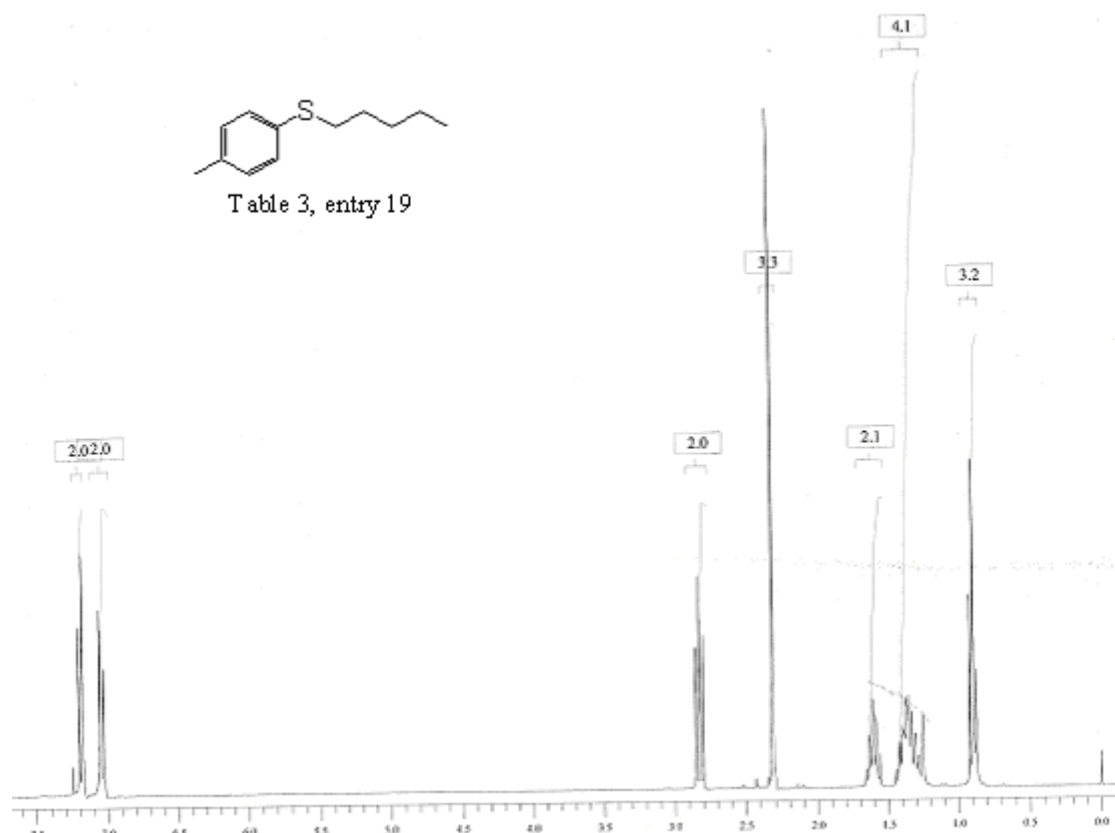
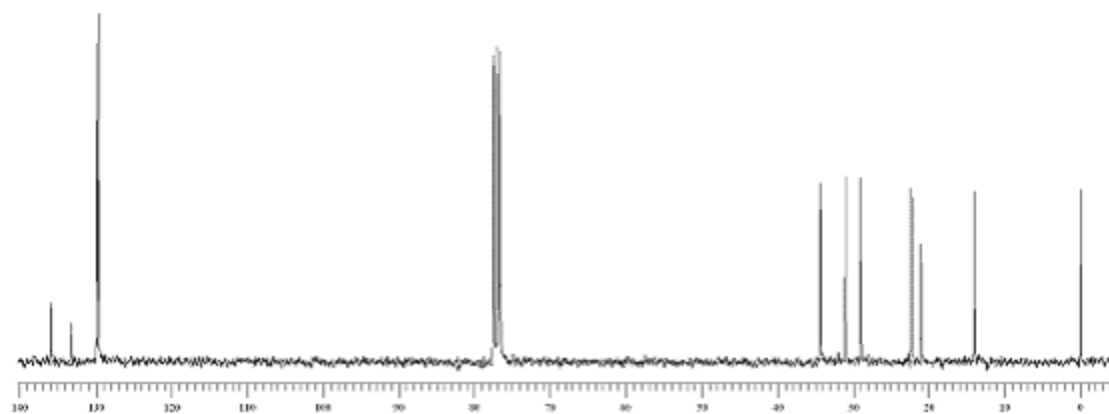
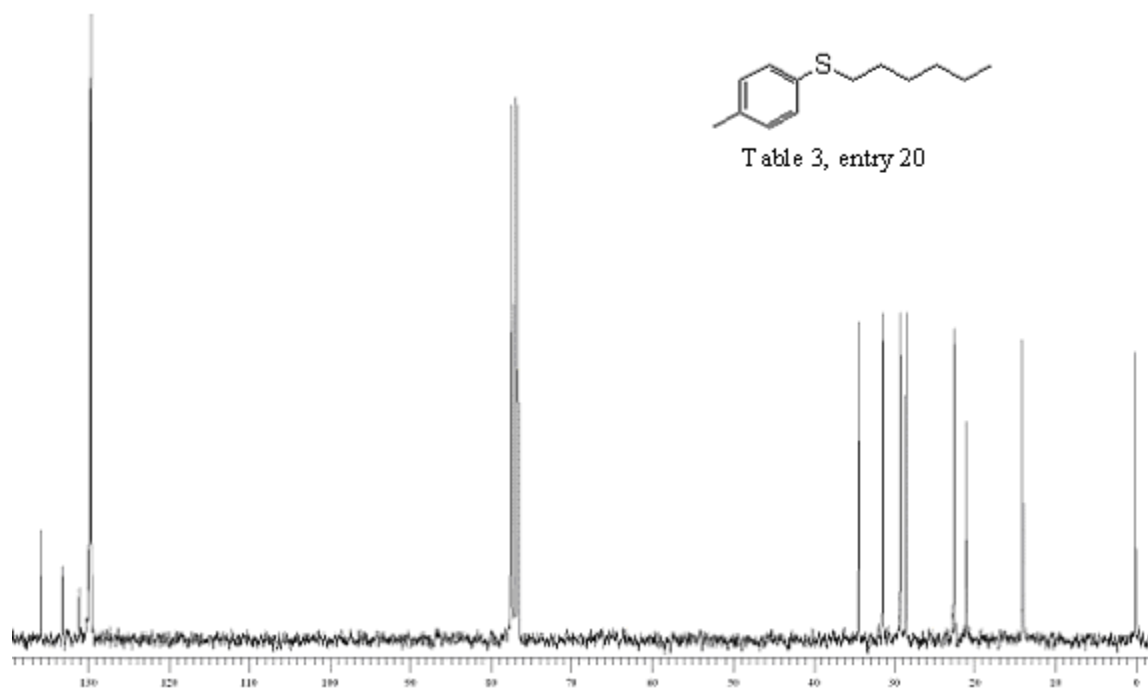
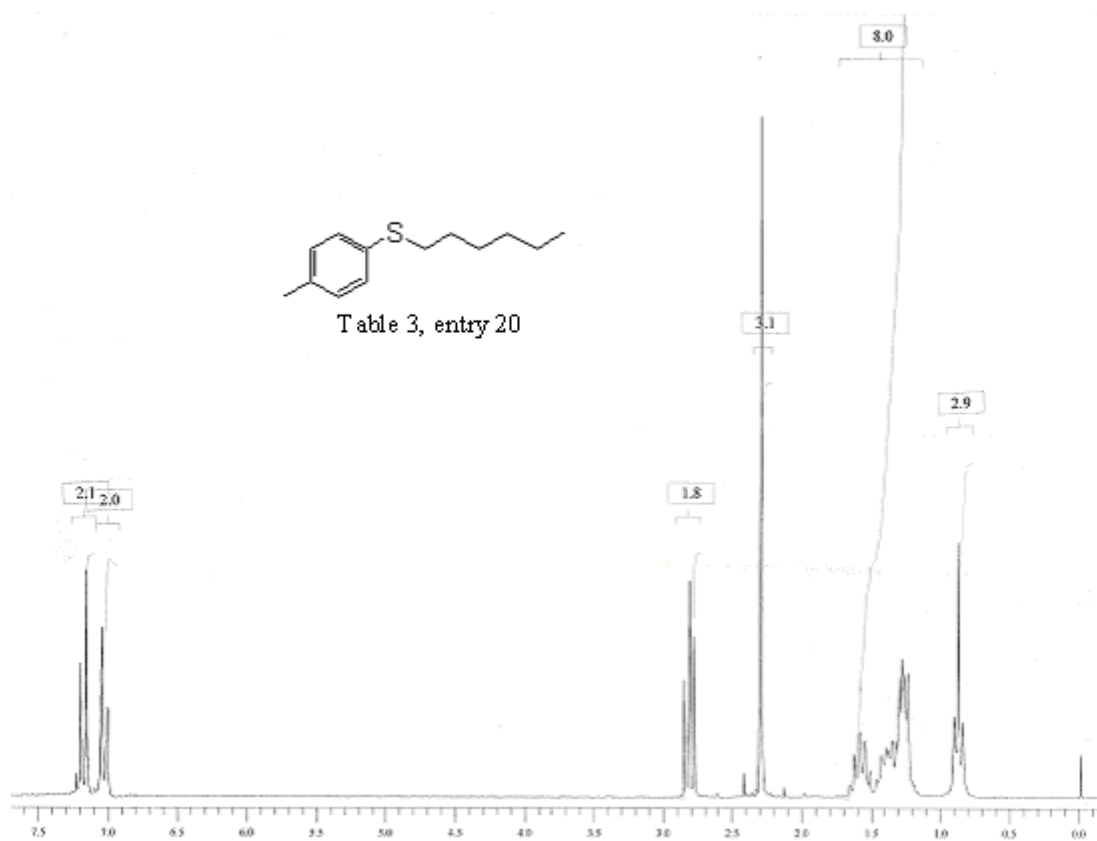


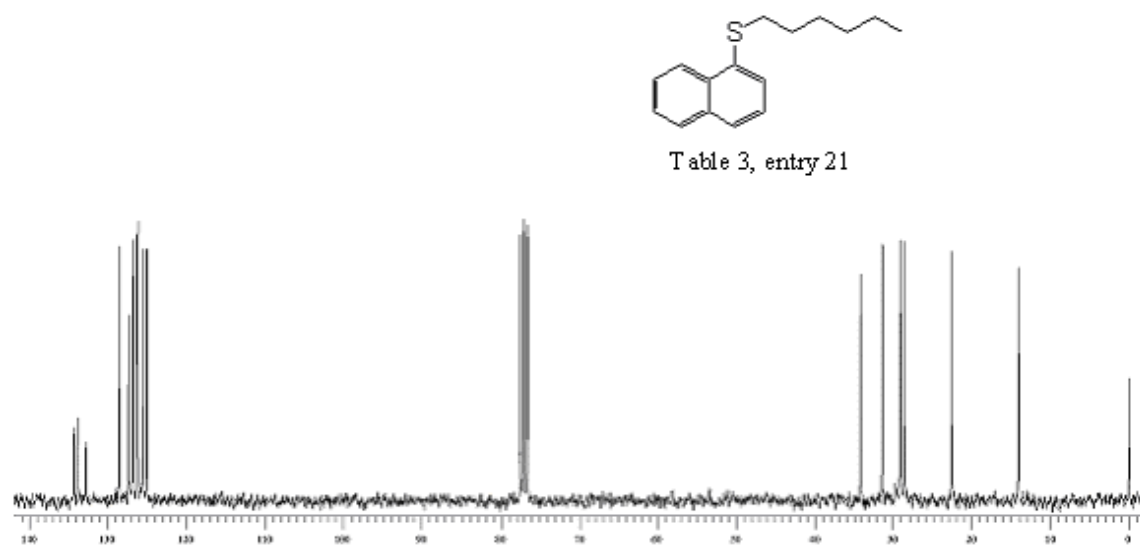
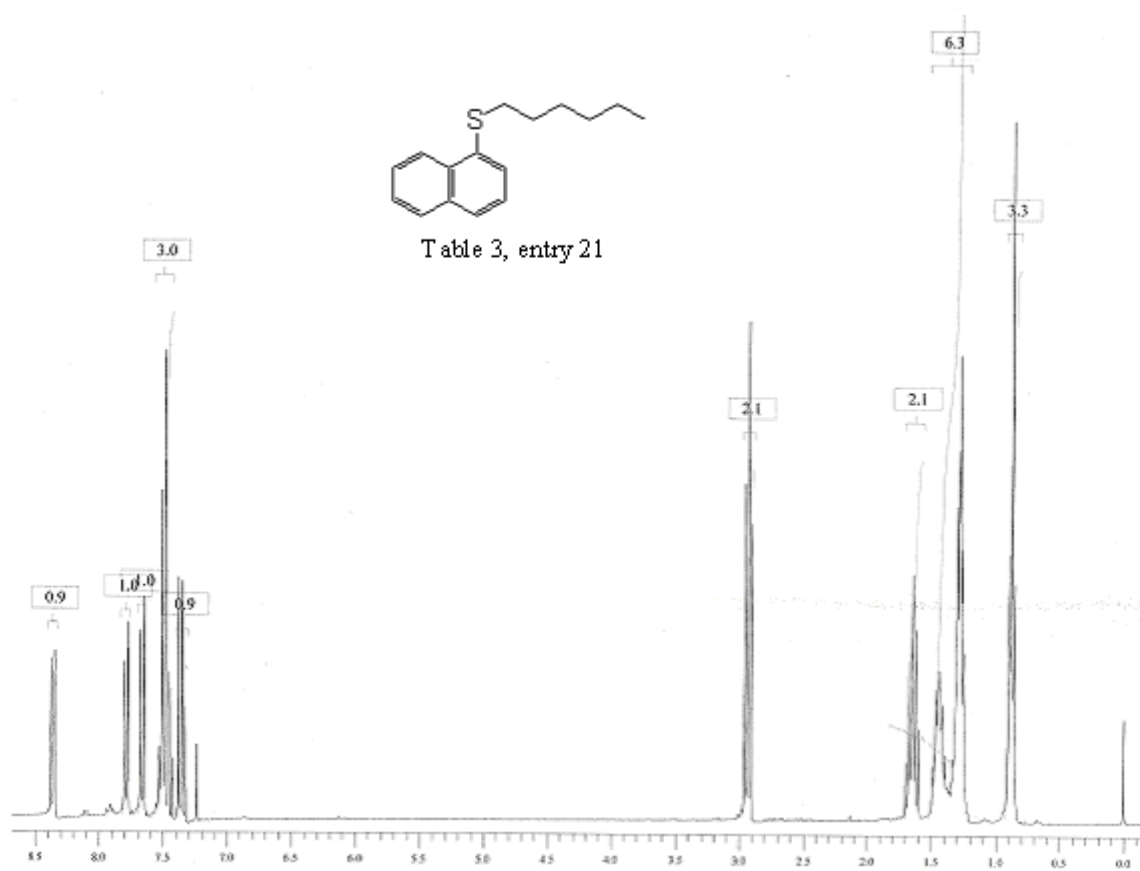
Table 3, entry 19



NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

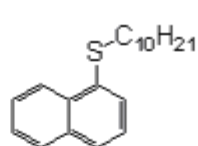


Table 3, entry 22

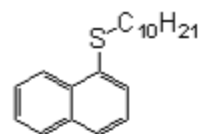
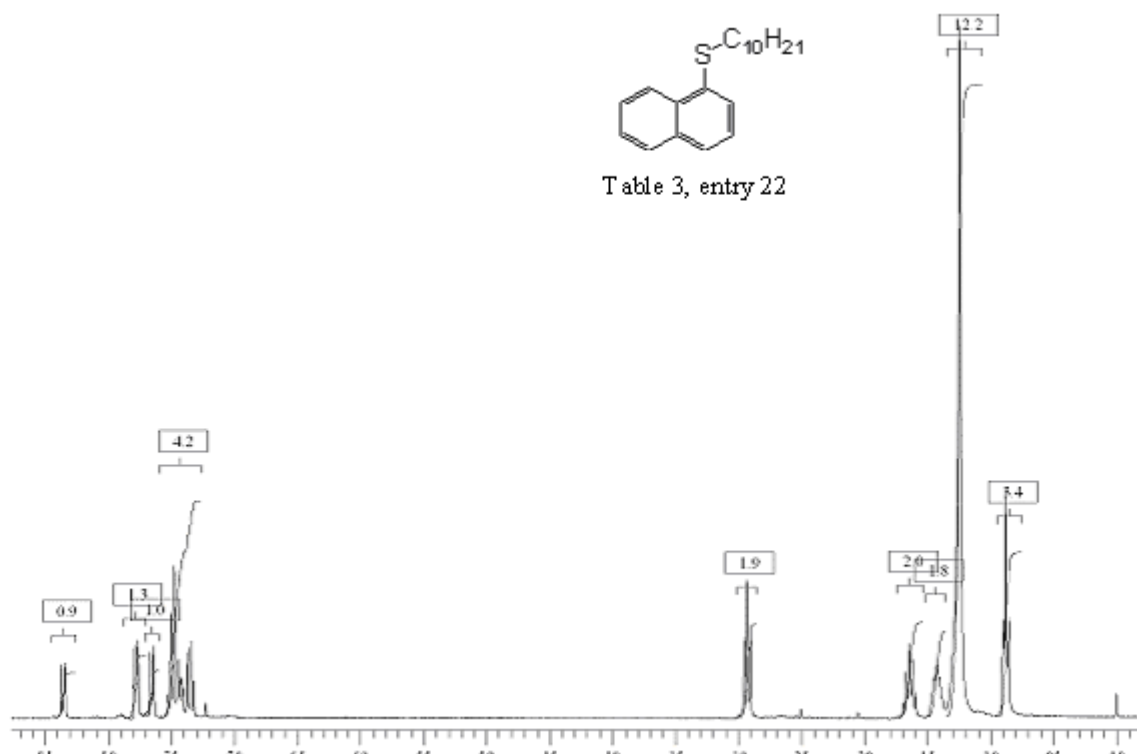
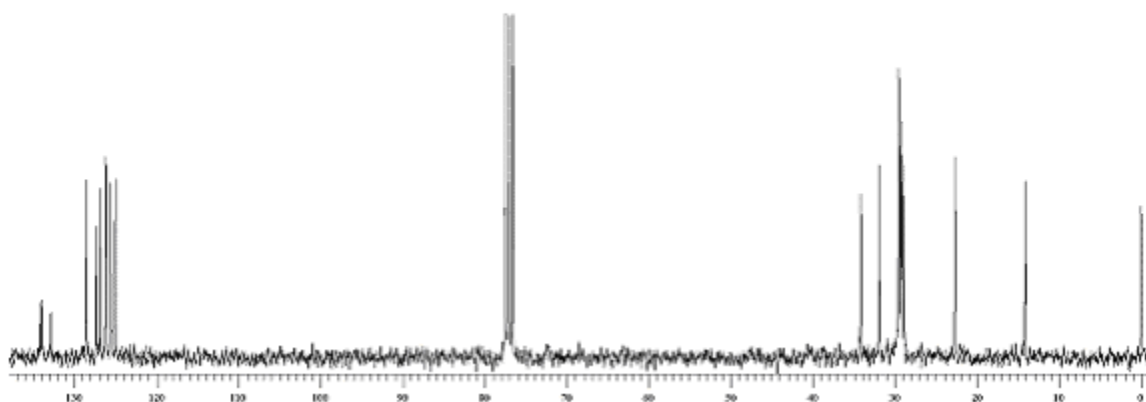
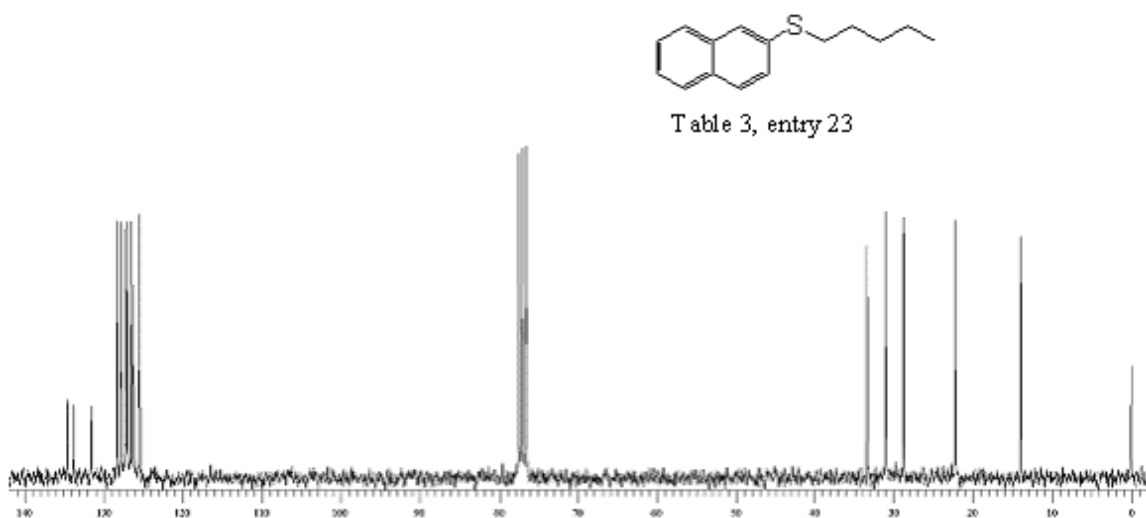
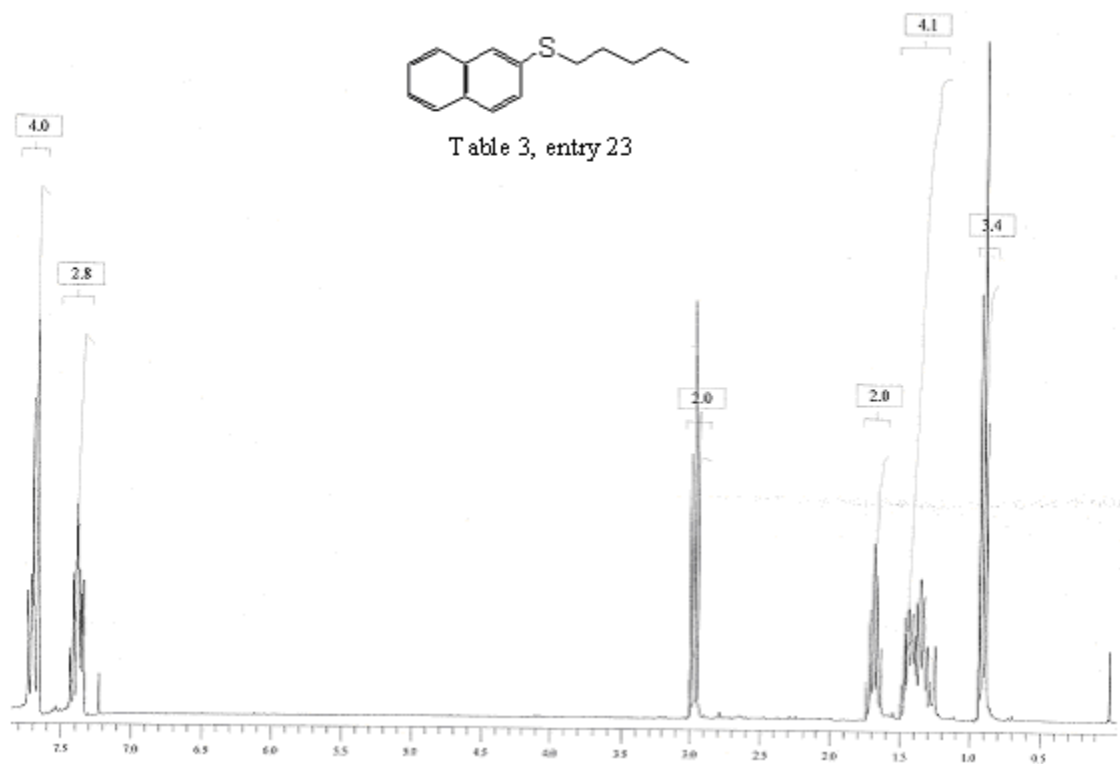


Table 3, entry 22



NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

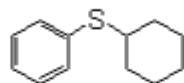


Table 3, entry 24

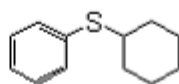
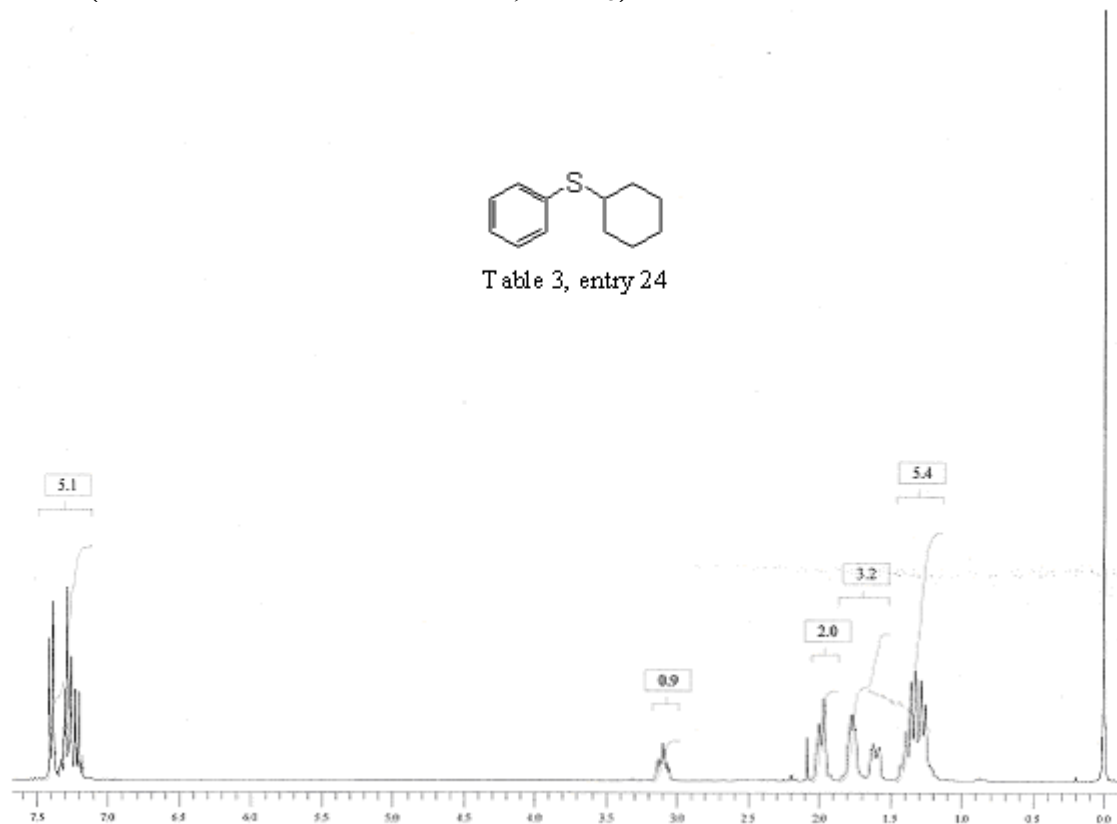
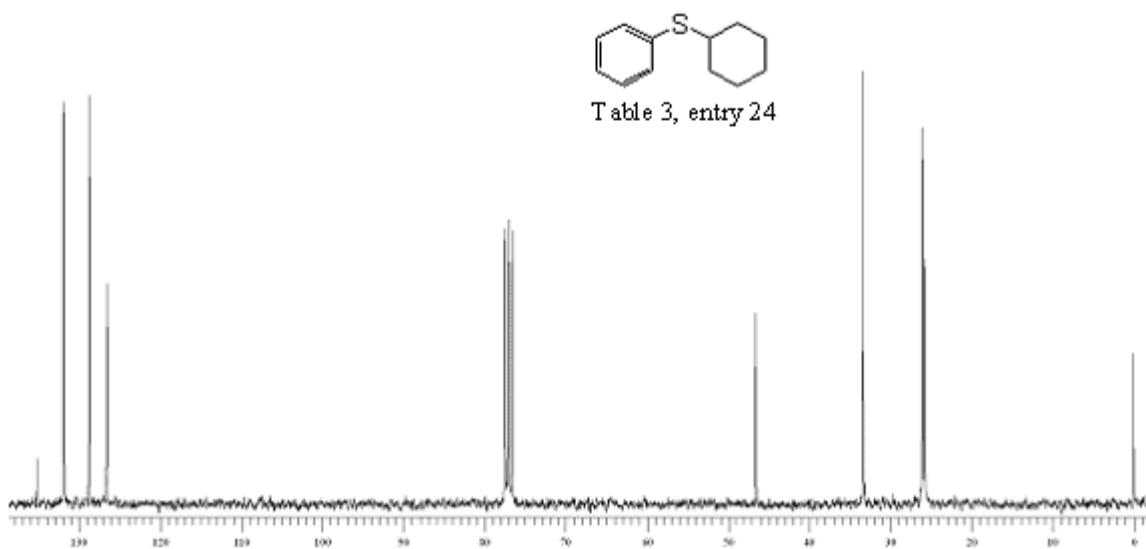
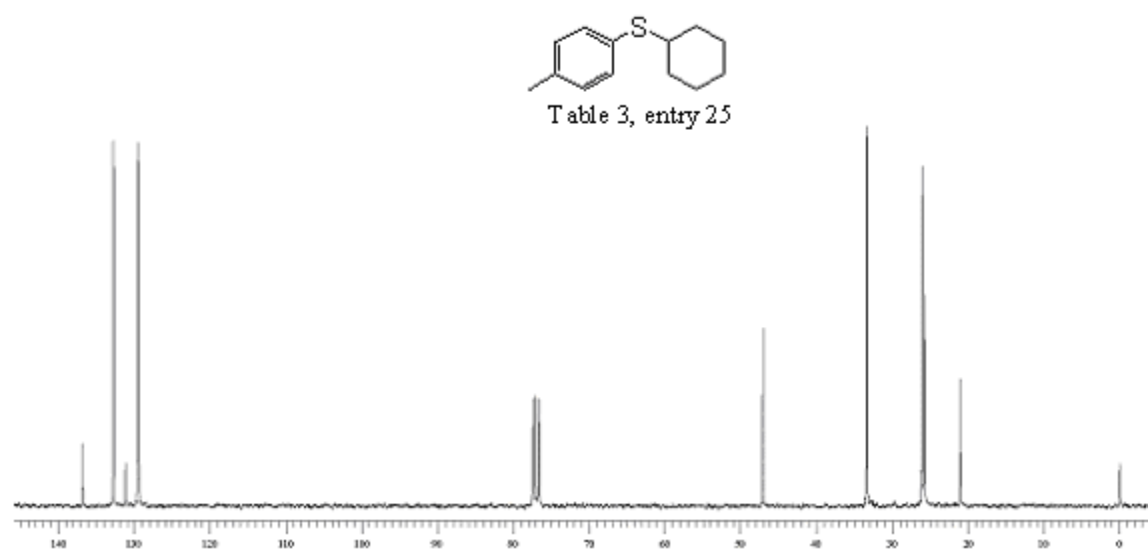
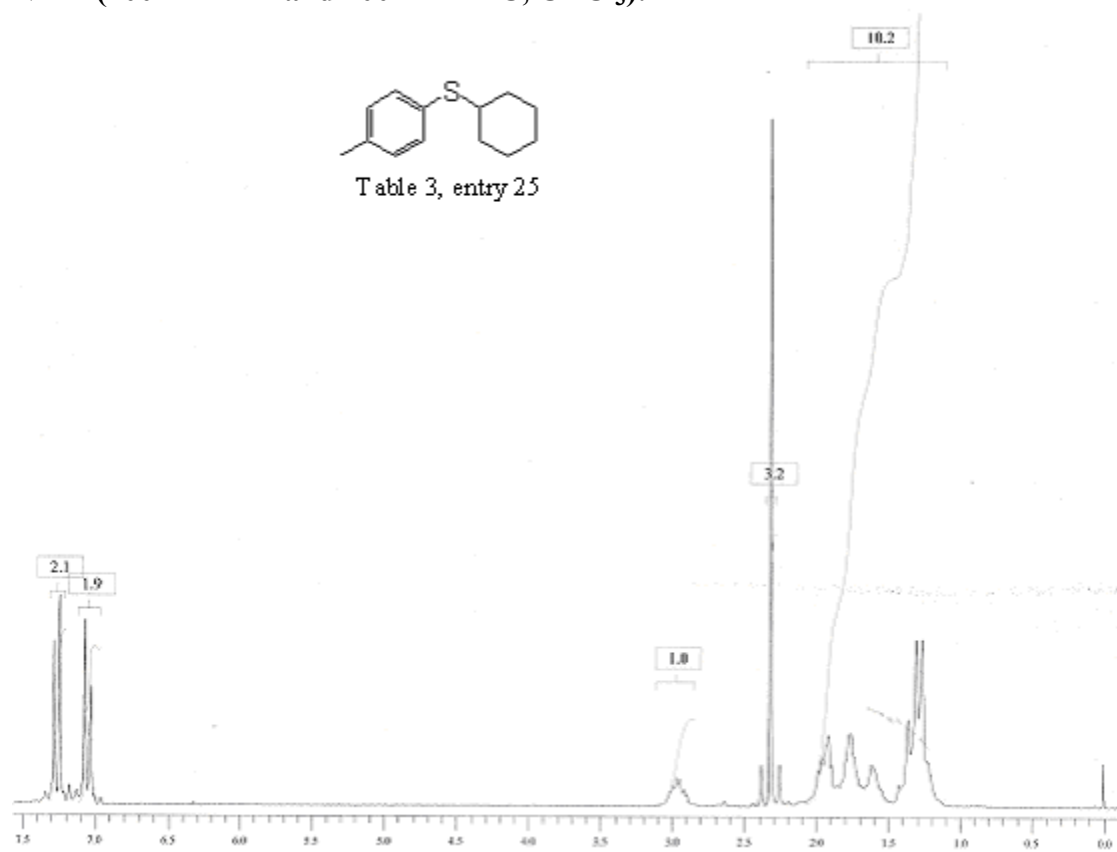


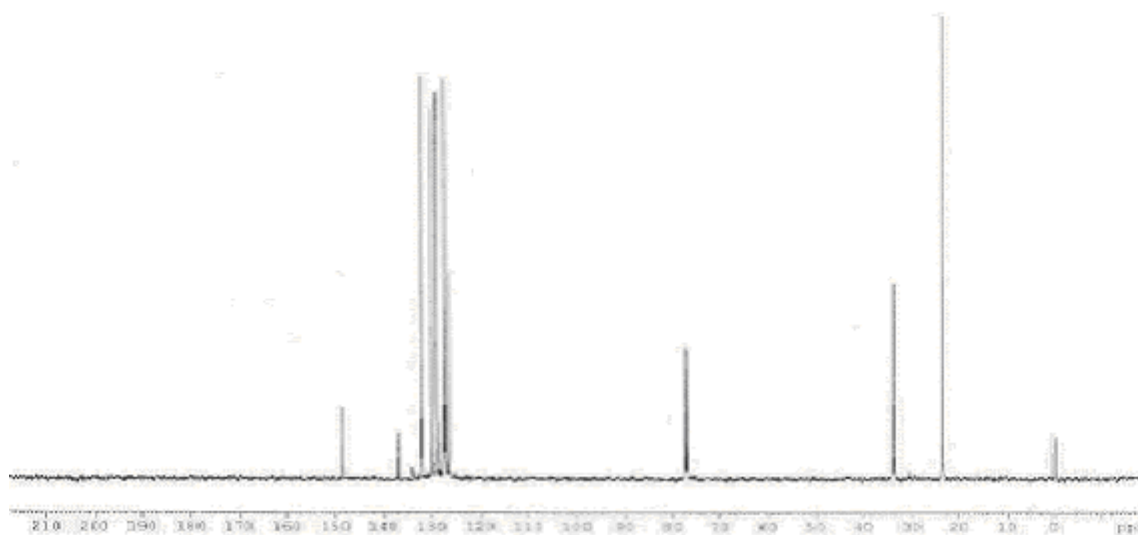
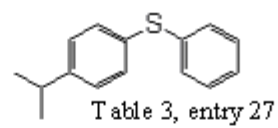
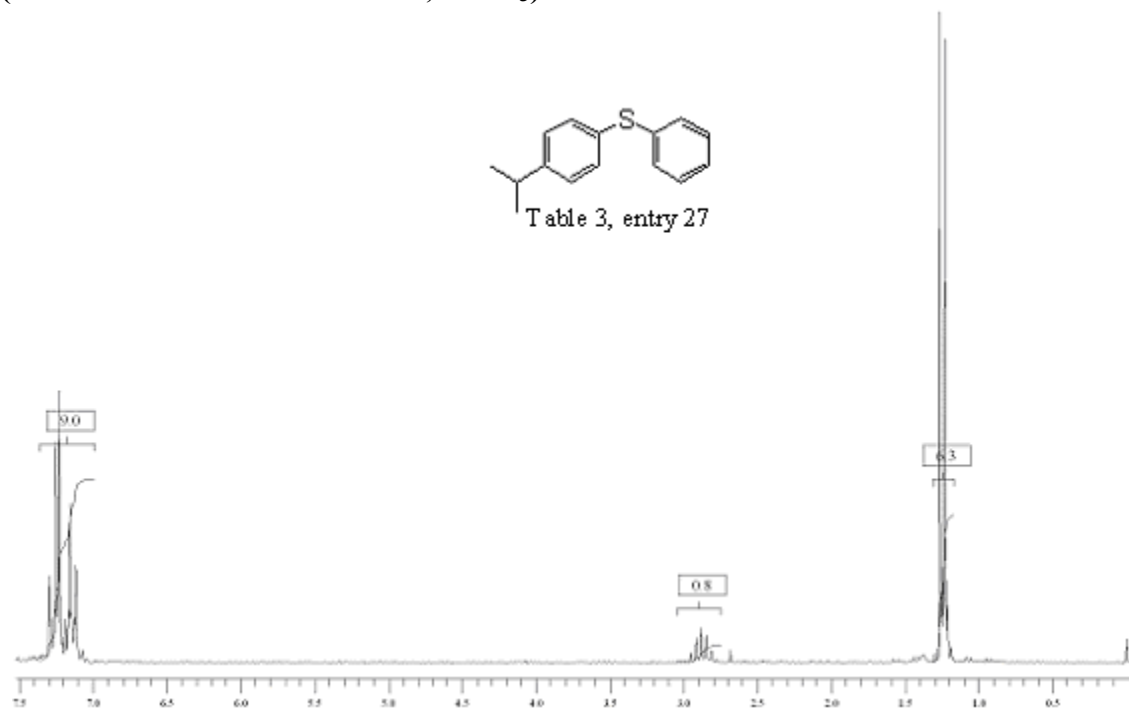
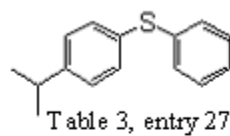
Table 3, entry 24



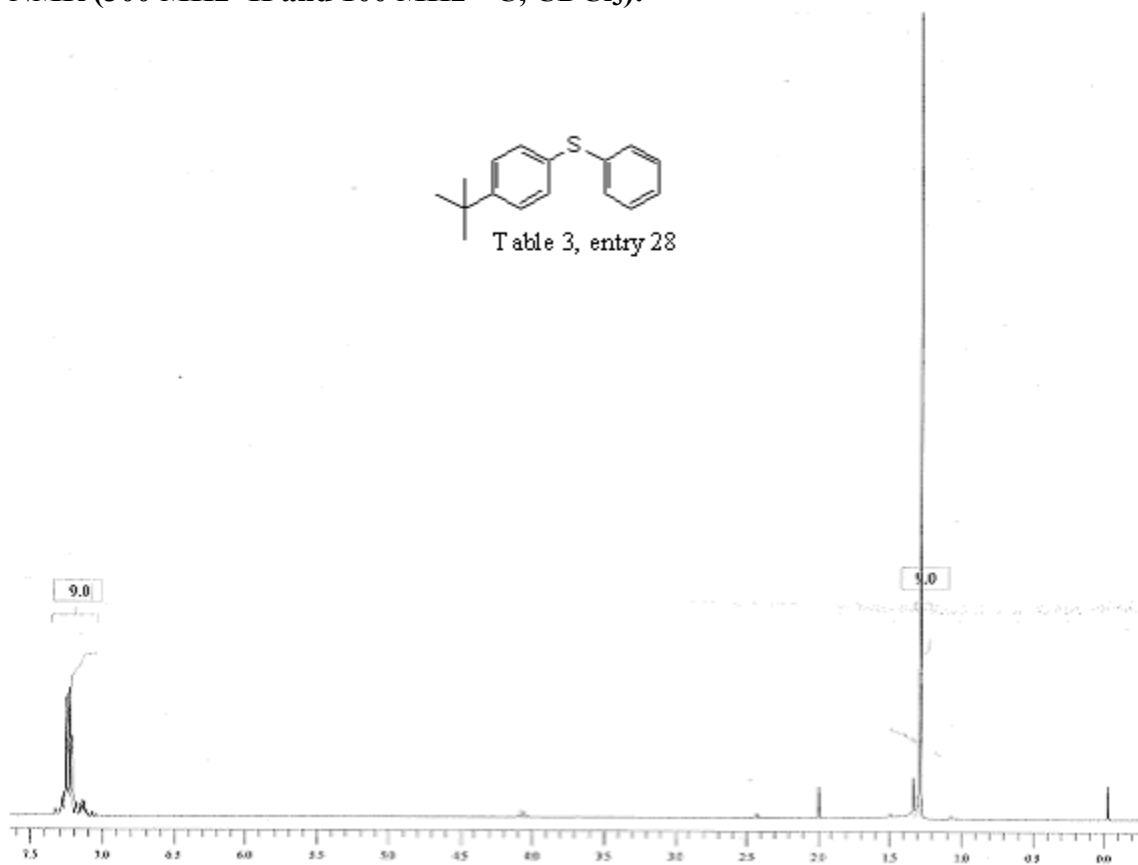
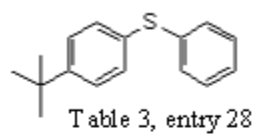
NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

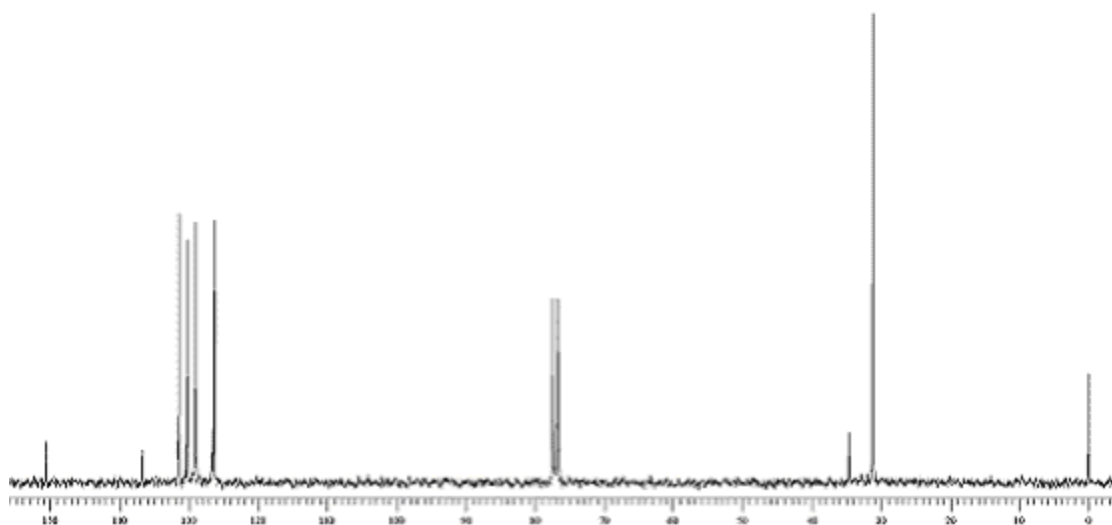
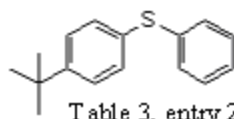


NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

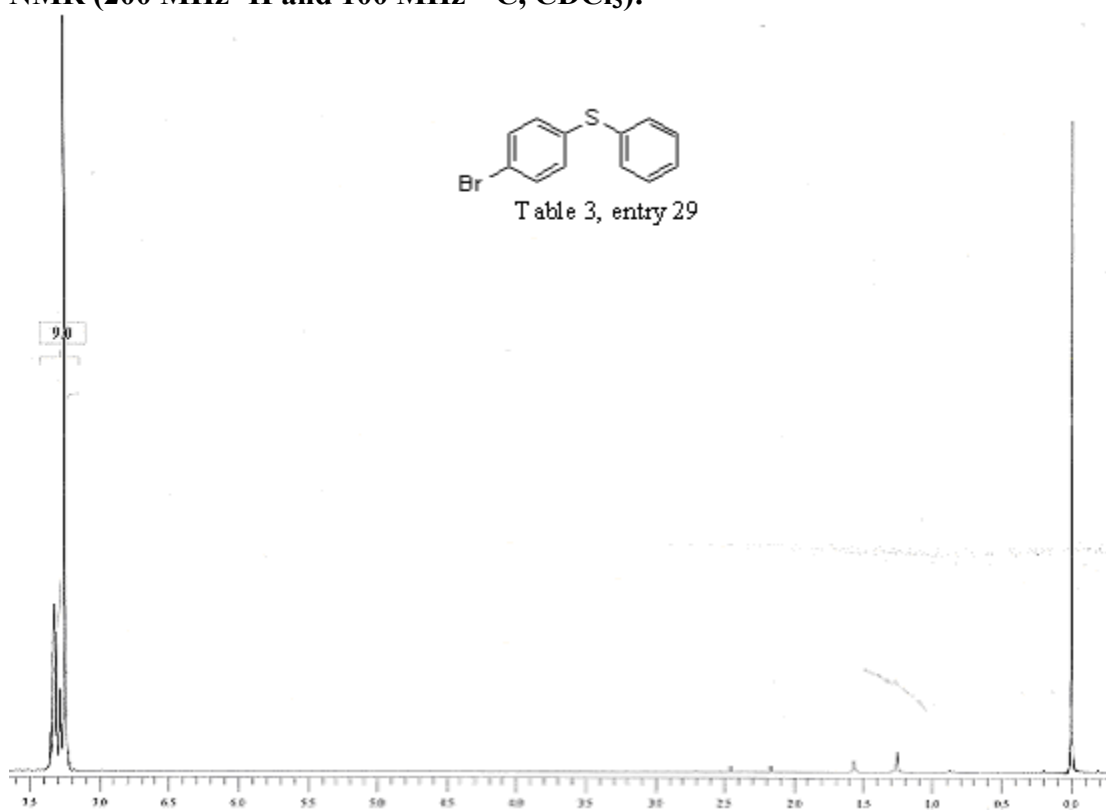
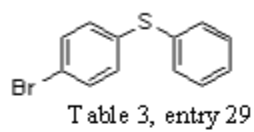


NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):





NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):



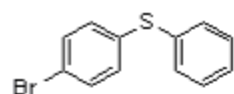
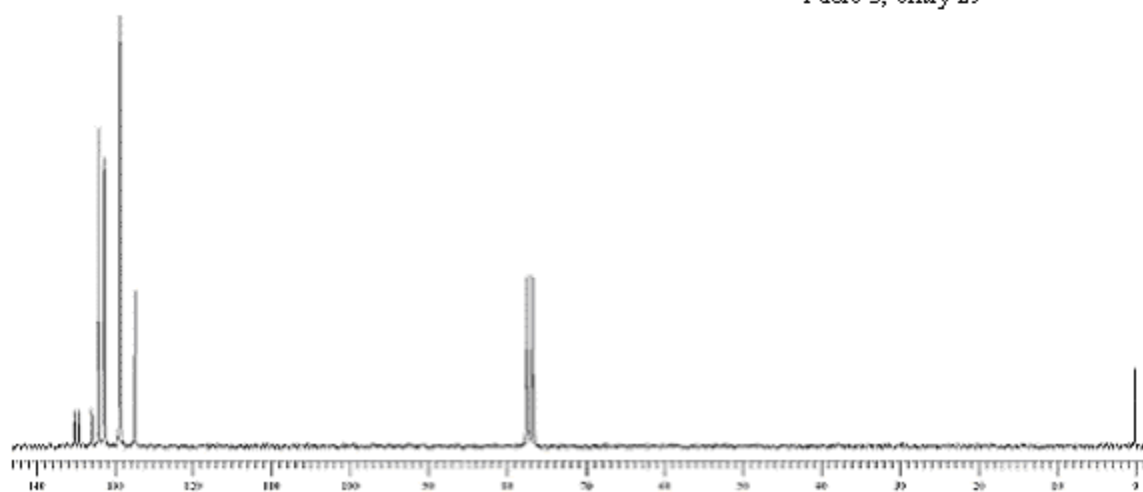


Table 3, entry 29



NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

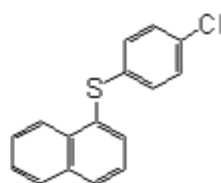


Table 3, entry 32

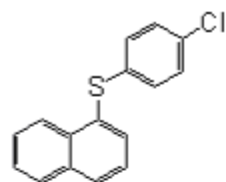
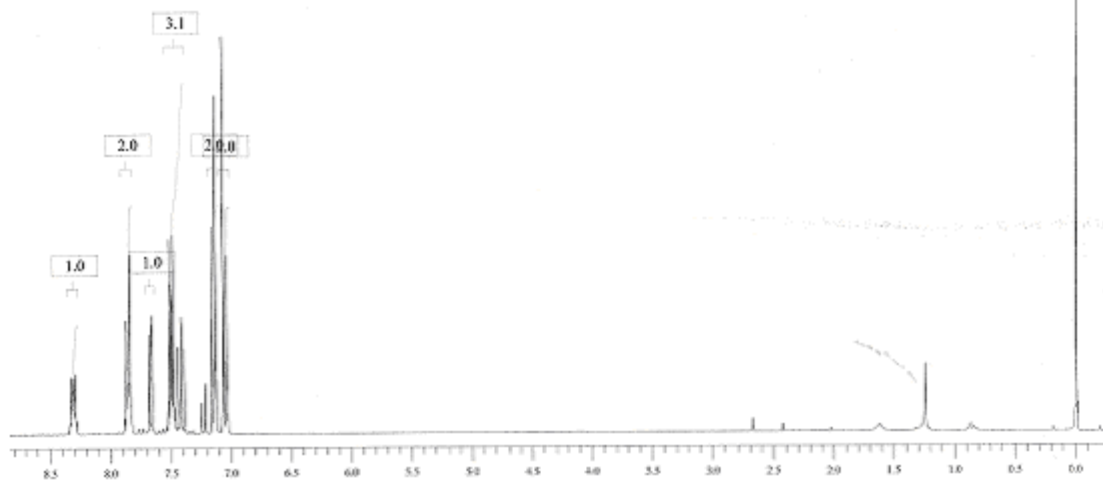
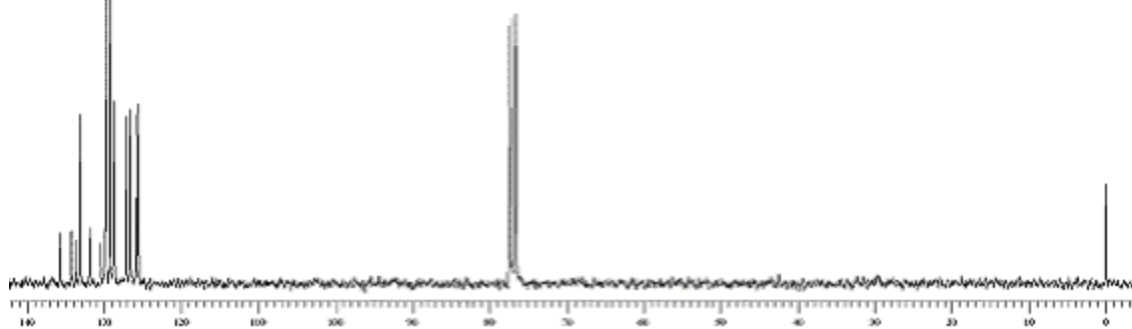
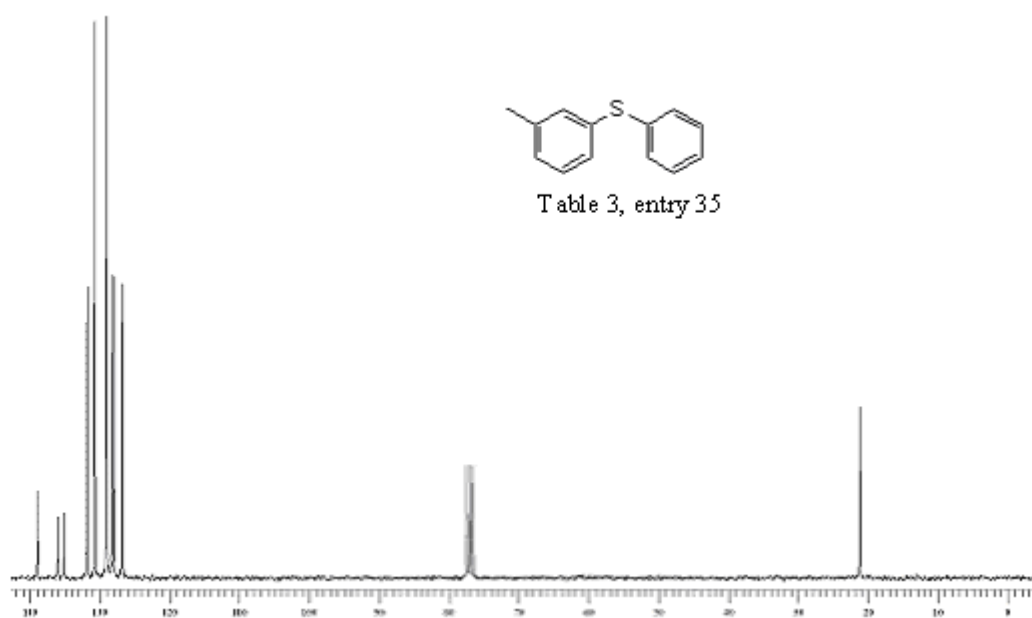
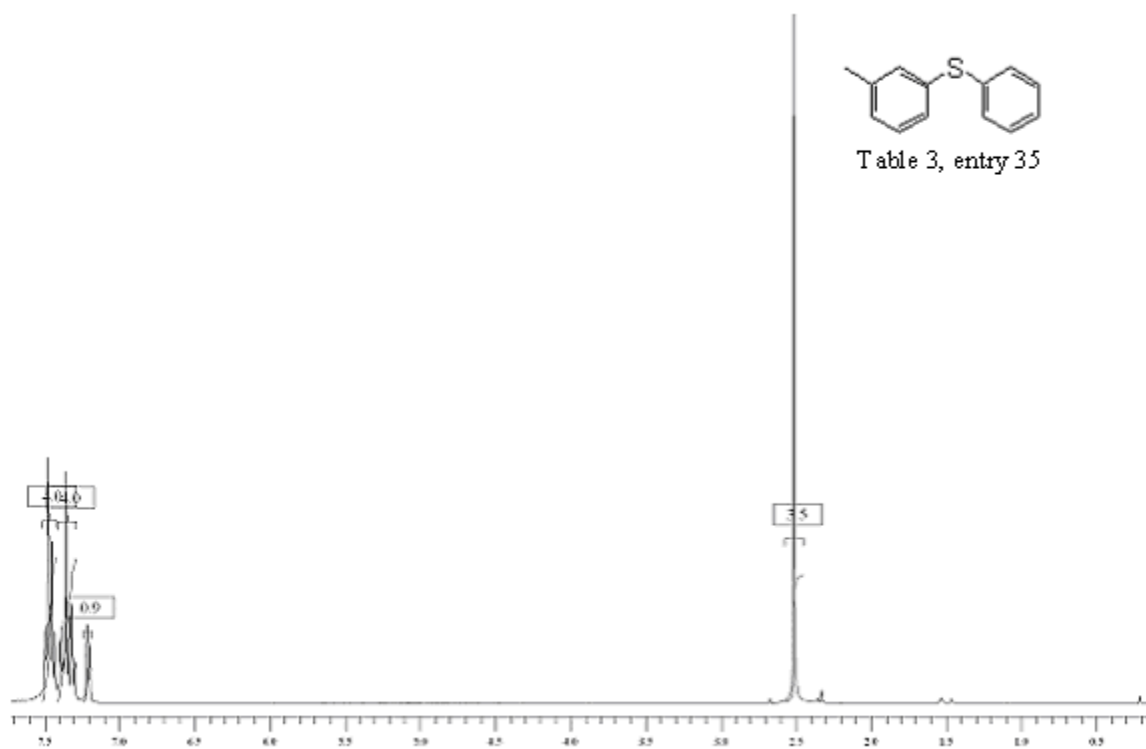


Table 3, entry 32



NMR (300 MHz ¹H and 100 MHz ¹³C, CDCl₃):



NMR (200 MHz ¹H and 100 MHz ¹³C, CDCl₃):

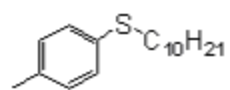


Table 3, entry 39

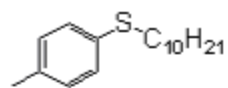
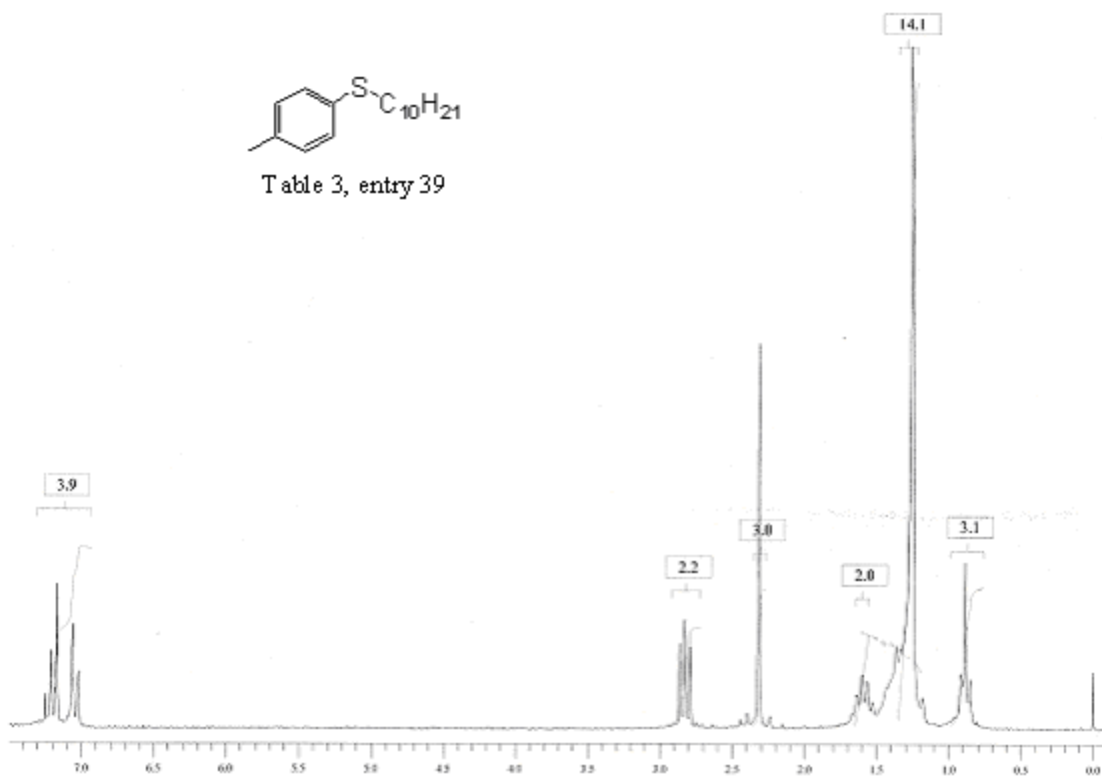
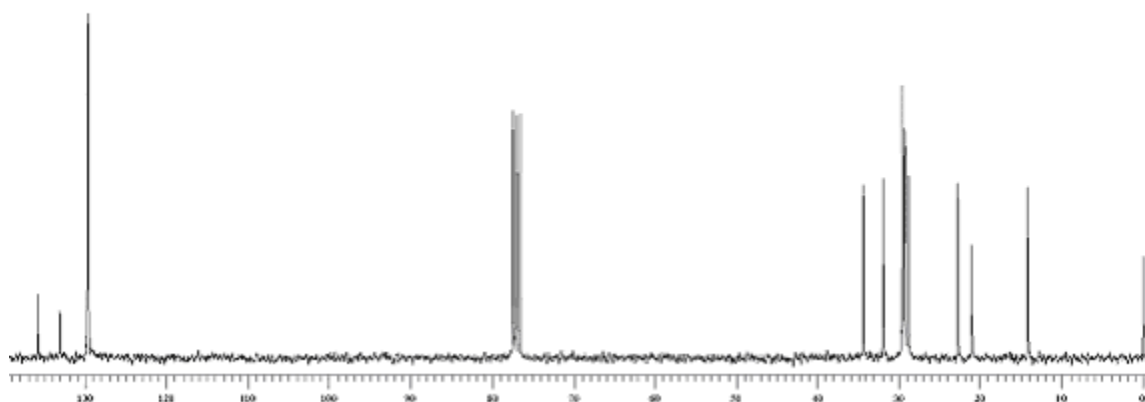


Table 3, entry 39



NMR (200 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

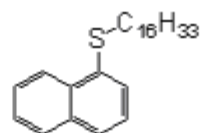


Table 3, entry 40

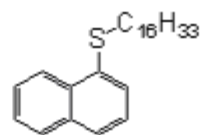
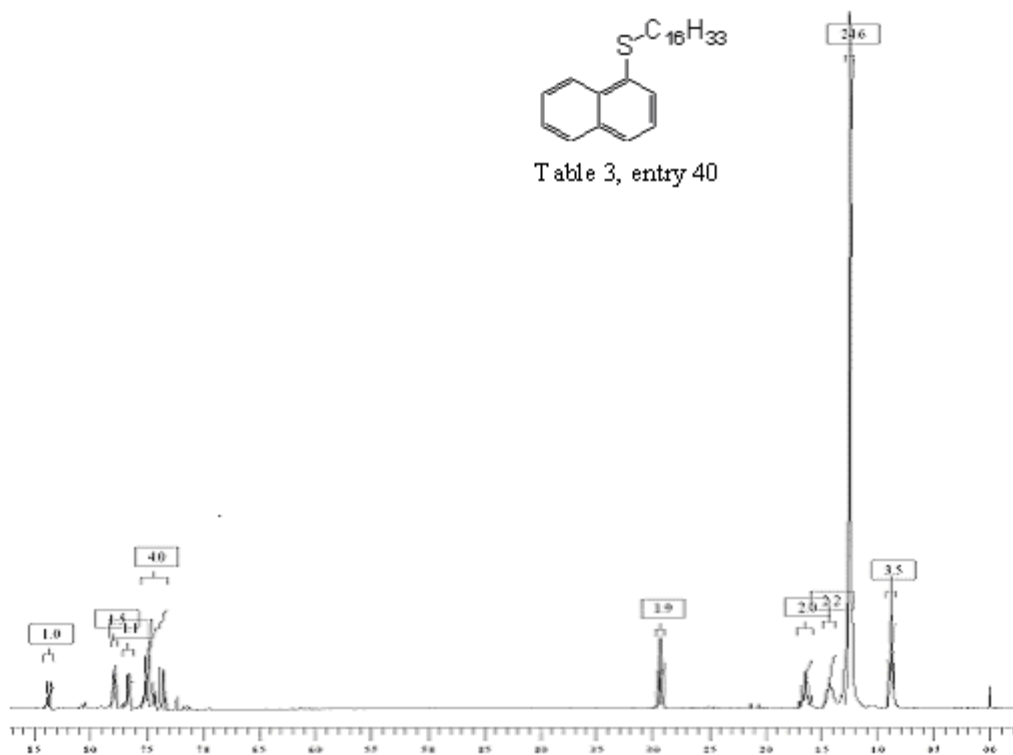
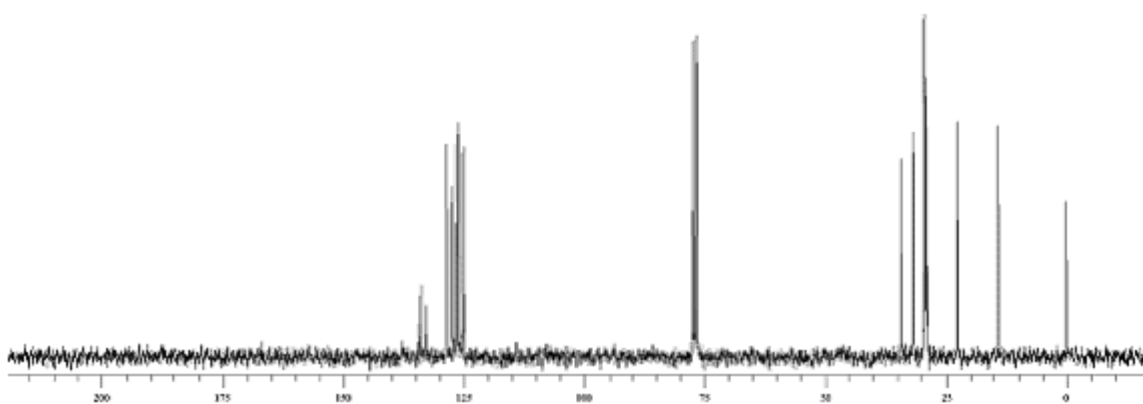


Table 3, entry 40



NMR (300 MHz ^1H and 100 MHz ^{13}C , CDCl_3):

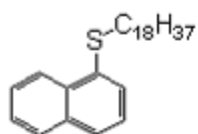


Table 3, entry 41

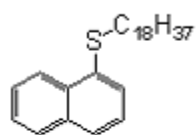
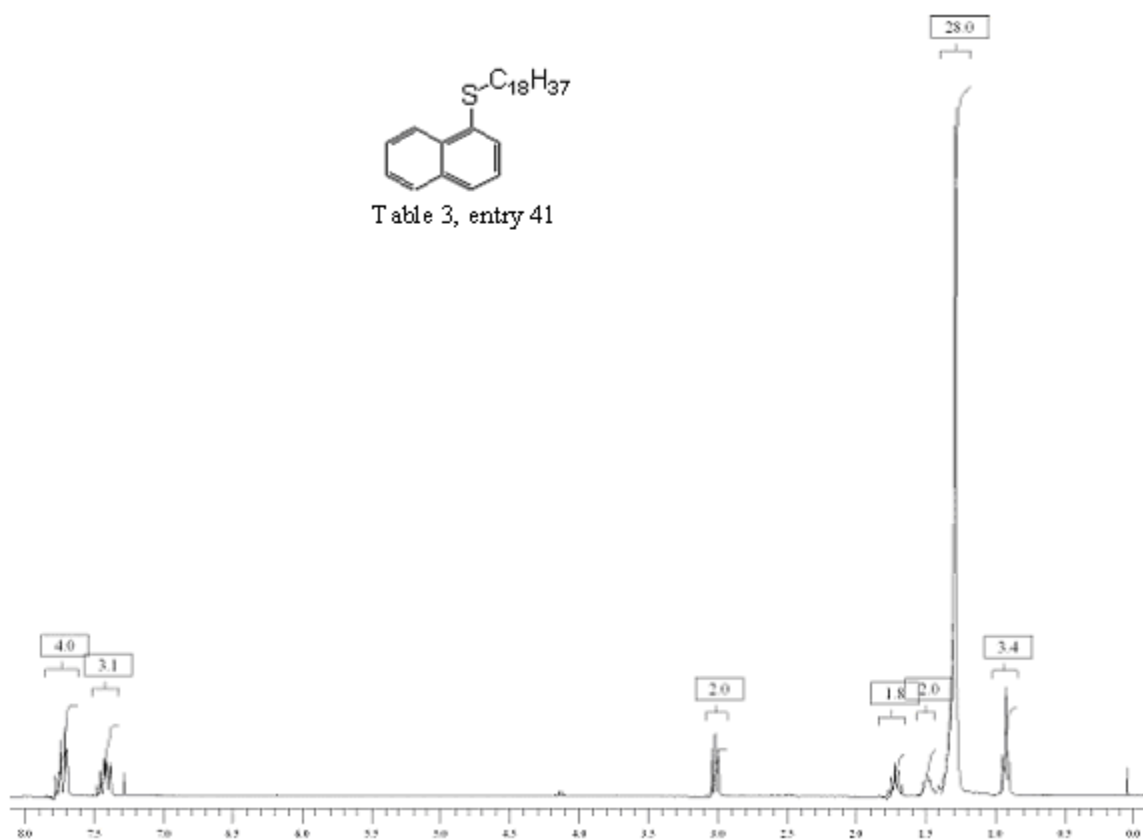


Table 3, entry 41

