Enantioselective Carbon–Sulfur Bond Formation: γ Additions of Aryl Thiols to Allenoates Catalyzed by a Chiral Phosphepine

Yuji Fujiwara, Jianwei Sun, and Gregory C. Fu*

Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

SUPPORTING INFORMATION

Table of Contents

I. General Information

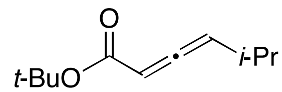
The following reagents were purchased and used as received: toluene (anhydrous; Sigma-Aldrich), pivalic acid (98%; TCI), sodium hydride (dry, 95%; Sigma-Aldrich), and phenylphosphine (Alfa–Aesar).

HPLC analyses were carried out on an Agilent 1100 Series system, and supercritical fluid chromatography (SFC) analyses were carried out on a Berger SFC MiniGram system. Daicel CHIRALCEL® columns or Daicel CHIRALPAK® columns (internal diameter 4.6 mm, column length 250 mm, particle size 5 μm) were used for both HPLC and SFC analysis.

II. Preparation of Allenes

General Procedure. (*tert*-Butoxycarbonylmethylene)triphenylphosphorane (11.3 g, 30.0 mmol) and a stir bar were added to a 250-mL flask, which was then evacuated and back-filled with nitrogen three times. CH_2Cl_2 (90 mL) and Et_3N (3.50 mL, 25 mmol) were added via syringe, and the resulting solution was stirred at r.t. in a water bath. The acid chloride (30.0 mmol) was then added dropwise via syringe over 2 min to the stirred solution, during which time the temperature was maintained at r.t. After 1 h of stirring at r.t., the reaction mixture was concentrated under reduced pressure to one-third of the original volume, and pentane (100 mL) and silica gel (5 g) were added. After stirring at r.t. for 1 h, the mixture was passed through a pad of silica gel and washed with 4:1 hexane/Et_2O (150 mL). The combined filtrate was concentrated under reduced pressure, and the residue was purified by column chromatography, which furnished the allenoate as a colorless oil.

The yields have not been optimized.



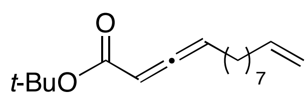
(±)-*tert*-Butyl 5-methylhexa-2,3-dienoate. Prepared from isovaleroyl chloride according to the General Procedure (purification by flash chromatography: 30:1 hexane/Et_2O; 23% yield).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.50 (dd, *J* = 6.0 Hz, *J* = 6.0 Hz, 1H), 5.44 (dd, *J* = 6.0 Hz, *J* = 2.8 Hz, 1H), 2.32-2.44 (m, 1H), 1.40 (s, 9H), 1.00 (d, *J* = 6.4 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, *J* = 6.4 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 210.7, 165.4, 102.1, 90.7, 80.4, 28.0, 27.6, 22.22, 22.18;

IR (film) 2966, 2933, 2873, 2254, 1959, 1708, 1458, 1411, 1392, 1368, 1322, 1279, 1257, 1148 cm^{-1};

LRMS (EI) calcd for $C_{11}H_{18}NaO_2$ (M+Na) 205.12, found 205.12.



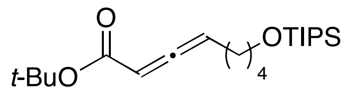
(±)-*tert*-Butyl trideca-2,3,12-trienoate. Prepared from 10-undecenoyl chloride according to the General Procedure (purification by flash chromatography: 20:1 hexane/Et_2O; 28% yield).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.76 (dddd, *J* = 17.2 Hz, *J* = 10.4 Hz, *J* = 6.4 Hz, *J* = 6.4 Hz, 1H), 5.49-5.56 (m, 1H), 5.42-5.46 (m, 1H), 4.95 (dd, *J* = 17.2 Hz, *J* = 1.2 Hz, 1H), 4.89 (ddd, *J* = 10.4 Hz, *J* = 1.2 Hz, *J* = 1.2 Hz, 1H), 2.04-2.11 (m, 2H), 1.97-2.04 (m, 2H), 1.44 (s, 9H), 1.23-1.43 (m, 10H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 211.8, 165.6, 139.1, 114.1, 95.0, 89.7, 80.6, 33.7, 29.2, 29.0, 28.84, 28.80, 28.7, 28.0, 27.5;

IR (film) 3077, 2978, 2928, 2856, 1961, 1719, 1641, 1457, 1415, 1392, 1368, 1282, 1257, 1145 cm^{-1};

LRMS (EI) calcd for $C_{17}H_{28}NaO_2$ (M+Na) 287.20, found 287.20.



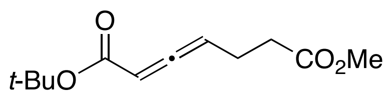
(±)-*tert*-Butyl 8-((triisopropylsilyl)oxy)octa-2,3-dienoate. Prepared from 6-(triisopropylsilyloxy)hexanoyl chloride[1] according to the General Procedure (purification by flash chromatography: 30:1 hexane/Et_2O; 16% yield).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.51-5.56 (m, 1H), 5.43-5.47 (m, 1H), 3.66 (t, *J* = 6.0 Hz, 2H), 2.09-2.16 (m, 2H), 1.46-1.61 (m, 4H), 1.44 (s, 9H), 1.00-1.10 (m, 21H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 211.8, 165.6, 95.0, 89.8, 80.6, 63.0, 32.2, 28.1, 27.4, 25.2, 18.0, 12.0;

IR (film) 3420, 2943, 2866, 1962, 1708, 1463, 1415, 1391, 1368, 1281, 1147, 1108, 1070, 1014 cm^{-1};

LRMS (EI) calcd for $C_{21}H_{40}NaO_3Si$ (M+Na) 391.26, found 391.26.



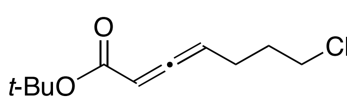
(±)-1-*tert*-Butyl 7-methyl hepta-2,3-dienedioate. Prepared from methyl 5-chloro-5-oxopentanoate according to the General Procedure (purification by flash chromatography: 9:1 hexane/ethyl acetate; 38% yield).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.58-5.63 (m, 1H), 5.46-5.50 (m, 1H), 3.63 (s, 3H), 2.35-2.46 (m, 4H), 1.42 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 211.5, 172.8, 165.0, 93.8, 90.0, 80.8, 51.6, 32.8, 28.0, 22.6;

IR (film) 2979, 1962, 1740, 1708, 1480, 1438, 1416, 1393, 1368, 1286, 1257, 1147, 1055 cm^{-1};

LRMS (EI) calcd for $C_{12}H_{18}NaO_4$ (M+Na) 249.11, found 249.11.



(±)-*tert*-Butyl 7-chlorohepta-2,3-dienoate. Prepared from 5-chlorovaleroyl chloride according to the General Procedure (purification by flash chromatography: 20:1 hexane/Et_2O; 51% yield).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.46-5.55 (m, 2H), 3.58 (t, *J* = 6.4 Hz, 2H), 2.20-2.29 (m, 2H), 1.84-1.95 (m, 2H), 1.43 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 211.8, 165.2, 93.4, 90.4, 80.9, 43.7, 31.1, 28.0, 24.5;

IR (film) 3404, 2979, 2936, 1962, 1706, 1479, 1456, 1415, 1393, 1368, 1283, 1257, 1146, 1041 cm^{-1};

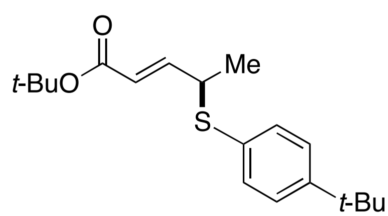
(1) Duffy, R. J.; Morris, K. A.; Romo, D. *J. Am. Chem. Soc.* **2005**, *127*, 16754–16755.

LRMS (EI) calcd for $C_{11}H_{17}ClNaO_2$ (M+Na) 239.08, found 239.08.

III. Phosphine-Catalyzed Enantioselective γ Additions of Aryl Thiols

General Procedure. In the air, phosphepine (*R*)-**1** (27 mg, 0.050 mmol) and pivalic acid (26 mg, 0.25 mmol) were added to an oven-dried 20-mL vial, which was then capped (if the thiol is a solid, then it was also added to the vial at this time). The vial was evacuated and back-filled with nitrogen three times. Toluene (anhydrous; 5 mL) and the thiol (0.60 mmol) were added via syringe, and the vial was cooled to 10 °C. Next, the allene (0.50 mmol) was added in one portion via syringe, and the reaction mixture was stirred at 10 °C for 72 h. To quench the reaction, *tert*-butyl hydroperoxide (3.0 M solution in isooctane; 100 μL) was added dropwise over 1 min. After 15 min, the reaction mixture was warmed to r.t., and a solution of $Na_2S_2O_3$ (20% aqueous solution; 5 mL) was added. The aqueous layer was extracted with Et_2O (three times), and the combined organic layers were washed with brine, dried (Na_2SO_4), and concentrated under reduced pressure. The residue was purified by column chromatography.

Note: Thiols are susceptible to air oxidation to form disulfides. A control experiment indicated that the presence of a disulfide does not significantly affect the enantioselectivity of the γ-addition process.



(*E*)-*tert*-Butyl 4-(4-(*tert*-butyl)phenylthio)pent-2-enoate (Table 2, entry 1). The compound was prepared according to the General Procedure from (±)-*tert*-butyl penta-2,3-dienoate (77 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 μL, 0.60 mmol). After purification by flash chromatography (4:1 → 2:1 hexane/CH_2Cl_2), the title compound was isolated as a colorless oil (90 mg, 56% yield) with 83% ee.

$[\alpha]^{23}_{D}$ = +97 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALPAK AD-H column; 2.5% MeOH in CO_2; 3.0 mL/min; retention times: 2.72 min (major), 3.27 min (minor).

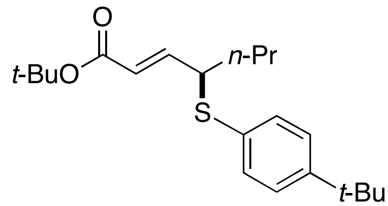
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (96 mg, 60% yield) with 79% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28-7.32 (m, 4H), 6.74 (dd, *J* = 15.6 Hz, *J* = 8.4 Hz, 1H), 5.46 (dd, *J* = 15.6 Hz, *J* = 1.2 Hz, 1H), 3.66-3.75 (m, 1H), 1.43 (s, 9H), 1.39 (d, *J* = 6.8 Hz, 3H), 1.28 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.6, 151.1, 147.3, 133.7, 129.8, 125.8, 122.1, 80.2, 44.9, 34.6, 31.2, 28.1, 19.5;

IR (film) 2967, 2930, 2906, 2869, 1909, 1714, 1649, 1596, 1490, 1478, 1458, 1392, 1367, 1339, 1287, 1263, 1220, 1156, 1137, 1120, 1064, 1014 cm^{-1};

LRMS (EI) calcd for $C_{19}H_{28}O_2S$ (M+) 320.18, found 320.20.



(*E*)-*tert*-Butyl 4-(4-(*tert*-butyl)phenylthio)hept-2-enoate (Table 2, entry 2). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 µL, 0.60 mmol). After purification by flash chromatography (7:1:1 → 2:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless oil (143 mg, 83% yield) with 90% ee.

$[\alpha]^{22}_{D}$ = +115 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALPAK IA-H column; 2.5% MeOH in CO_2; 3.0 mL/min; retention times: 2.81 min (major), 3.08 min (minor).

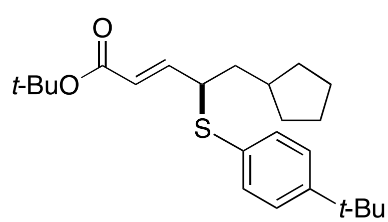
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (138 mg, 79% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28 (s, 4H), 6.64 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.38 (dd, *J* = 15.6 Hz, *J* = 0.4 Hz, 1H), 3.50-3.56 (m, 1H), 1.55-1.74 (m, 2H), 1.38-1.47 (m, 2H), 1.42 (s, 9H), 1.28 (s, 9H), 0.90 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.6, 151.1, 146.6, 133.8, 129.9, 125.8, 122.8, 80.2, 50.7, 35.7, 34.6, 31.3, 28.1, 20.5, 13.8;

IR (film) 3078, 2963, 2933, 2872, 1714, 1647, 1597, 1559, 1540, 1490, 1458, 1393, 1367, 1337, 1292, 1268, 1257, 1232, 1153, 1132, 1120, 1089, 1014 cm^{-1};

LRMS (EI) calcd for $C_{21}H_{32}O_2S$ (M+) 348.21, found 348.20.



(*E*)-*tert*-Butyl 4-(4-(*tert*-butyl)phenylthio)-5-cyclopentylpent-2-enoate (Table 2, entry 3). The compound was prepared according to the General Procedure from (±)-*tert*-butyl 5-cyclopentylpenta-2,3-dienoate (111 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 µL, 0.60 mmol). After purification by flash chromatography (7:1:1 → 6:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless semisolid (139 mg, 72% yield) with 91% ee.

$[\alpha]^{23}_D$ = +88 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALPAK IA column; 2.5% MeOH in CO_2; 3.0 mL/min; retention times: 4.62 min (major), 5.07 min (minor).

The second run was performed with (*S*)-**1**. The product was isolated as a colorless semisolid (125 mg, 64% yield) with 90% ee.

Recrystallization from *n*-hexane gave a colorless needle, which was subjected to X-ray crystallography.

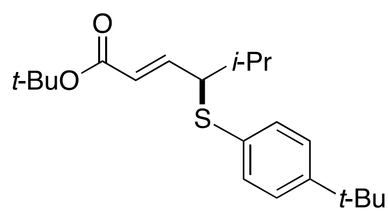
mp 69–72 °C;

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28 (s, 4H), 6.63 (dd, *J* = 15.2 Hz, *J* = 9.4 Hz, 1H), 5.34 (d, *J* = 15.2 Hz, 1H), 3.50-3.57 (m, 1H), 1.88-1.98 (m, 1H), 1.71-1.81 (m, 2H), 1.65-1.70 (m, 2H), 1.45-1.62 (m, 4H), 1.42 (s, 9H), 1.28 (s, 9H), 1.01-1.12 (m, 2H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 151.0, 146.7, 133.8, 129.8, 125.7, 122.5, 80.1, 50.3, 39.9, 37.7, 34.5, 32.8, 32.1, 31.2, 28.1, 25.02, 24.99;

IR (film) 2954, 2868, 2361, 2340, 1714, 1646, 1559, 1540, 1506, 1490, 1457, 1393, 1366, 1327, 1294, 1232, 1144 cm^{-1};

LRMS (ESI) calcd for $C_{24}H_{36}NaO_2S$ (M+Na) 411.23, found 411.23.



(*E*)-*tert*-Butyl 4-(4-(*tert*-butyl)phenylthio)-5-methylhexa-2-enoate (Table 2, entry 4). The compound was prepared according to the General Procedure from (±)-*tert*-butyl 5-methylhexa-2,3-dienoate (91 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 μL, 0.60 mmol). After purification by flash chromatography (4:1 → 3:1 hexane/CH_2Cl_2), the title compound was isolated as a colorless semisolid (104 mg, 60% yield) with 95% ee.

$[\alpha]^{23}_D$ = +159 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALPAK AD-H column; 2.5% MeOH in CO_2; 3.0 mL/min; retention times: 2.73 min (major), 3.15 min (minor).

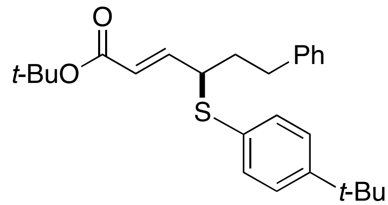
The second run was performed with (*S*)-**1**. The product was isolated as a colorless semisolid (108 mg, 62% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27 (s, 4H), 6.68 (dd, *J* = 15.2 Hz, *J* = 10.0 Hz, 1H), 5.34 (dd, *J* = 15.2 Hz, *J* = 0.6 Hz, 1H), 3.35 (ddd, *J* = 10.0 Hz, *J* = 6.4 Hz, *J* = 0.6 Hz, 1H), 1.92-2.01 (m, 1H), 1.42 (s, 9H), 1.28 (s, 9H), 1.06 (d, *J* = 6.8 Hz, 3H), 1.01 (d, *J* = 6.8 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 150.8, 144.9, 133.5, 130.5, 125.8, 123.3, 80.1, 58.9, 34.5, 31.9, 31.2, 28.1, 20.6, 19.8;

IR (film) 2965, 2905, 2871, 1714, 1645, 1489, 1462, 1367, 1322, 1286, 1248, 1153, 1014 cm^{-1};

LRMS (ESI) calcd for $C_{21}H_{32}O_2S$ (M+) 348.21, found 348.20.



(*E*)-*tert*-Butyl 4-(4-*tert*-butylphenylthio)-6-phenylhex-2-enoate (Table 2, entry 5). The compound was prepared according to the General Procedure from (±)-*tert*-butyl 6-phenylhexa-2,3-dienoate (129 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 μL, 0.60 mmol). After purification by flash chromatography (7:1:1 → 6:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless semisolid (149 mg, 73% yield) with 92% ee.

$[\alpha]^{24}_D$ = +63 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALCEL OJ-H column; 5% MeOH in CO_2; 3.0 mL/min; retention times: 4.05 min (minor), 4.72 min (major).

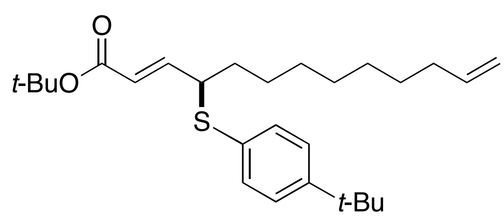
The second run was performed with (*S*)-**1**. The product was isolated as a colorless semisolid (137 mg, 67% yield) with 90% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.29 (s, 4H), 7.16-7.28 (m, 5H), 6.71 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.41 (d, *J* = 15.6 Hz, 1H), 3.47-3.56 (m, 1H), 2.71-2.85 (m, 2H), 1.91-2.09 (m, 2H), 1.45 (s, 9H), 1.30 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.4, 151.2, 146.1, 140.9, 133.8, 129.4, 128.43, 128.41, 126.0, 125.8, 123.0, 80.2, 49.9, 34.9, 34.5, 33.2, 31.2, 28.1;

IR (film) 2965, 2867, 1714, 1647, 1603, 1559, 1490, 1456, 1393, 1367, 1292, 1148, 1120, 1014 cm^{-1};

LRMS (ESI) calcd for $C_{26}H_{34}NaO_2S$ (M+Na) 433.22, found 433.22.



(*E*)-*tert*-Butyl 4-(4-(*tert*-butyl)phenylthio)trideca-2,12-dienoate (Table 2, entry 6). The compound was prepared according to the General Procedure from (±)-*tert*-butyl trideca-2,3,12-trienoate (132 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 μL, 0.60 mmol). After purification by flash chromatography (8:1:1 → 7:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless oil (154 mg, 72% yield) with 88% ee.

$[\alpha]^{24}_D$ = +86 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALCEL OJ-H column; 5.0% MeOH in CO_2; 3.0 mL/min; retention times: 2.25 min (minor), 2.67 min (major).

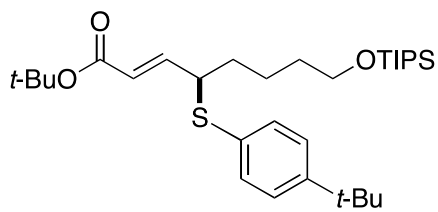
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (148 mg, 69% yield) with 90% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28 (s, 4H), 6.64 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.73-5.85 (m, 1H), 5.37 (d, *J* = 15.6 Hz, 1H), 4.89-5.01 (m, 2H), 3.46-3.54 (m, 1H), 1.55-1.75 (m, 2H), 1.43 (s, 9H), 1.30-1.44 (m, 4H), 1.28 (s, 9H), 1.23-1.28 (m, 8H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 151.0, 146.6, 139.1, 133.7, 129.9, 125.8, 122.7, 114.2, 80.2, 50.9, 34.5, 33.8, 33.6, 31.2, 29.21, 29.20, 29.0, 28.8, 28.1, 27.2;

IR (film) 3077, 2966, 2929, 2856, 2361, 2340, 1714, 1645, 1490, 1459, 1392, 1367, 1292, 1269, 1256, 1150, 1120, 1014 cm^{-1};

LRMS (ESI) calcd for $C_{27}H_{42}NaO_2S$ (M+Na) 453.28, found 453.28.



(*E*)-*tert*-Butyl 4-(4-(*tert*-butyl)phenylthio)-8-triisopropyloxyoct-2-enoate (Table 2, entry 7). The compound was prepared according to the General Procedure from (±)-*tert*-butyl 8-(triisopropylsilyloxy)octa-2,3-dienoate (191 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 μL, 0.60 mmol). After purification by flash chromatography (4:1:1 → 3:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless oil (179 mg, 67% yield) with 95% ee.

$[\alpha]^{24}_{D}$ = +63 (c = 1.0, $CHCl_3$).

HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 0.6% 2-PrOH in hexanes; 1.0 mL/min; retention times: 14.3 min (major), 18.4 min (minor).

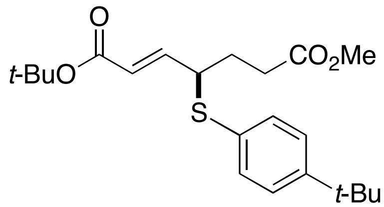
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (184 mg, 69% yield) with 94% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28 (s, 4H), 6.64 (dd, *J* = 15.2 Hz, *J* = 9.4 Hz, 1H), 5.38 (d, *J* = 15.2 Hz, 1H), 3.62-3.68 (t, *J* = 6.0 Hz, 2H), 3.48-3.54 (m, 1H), 1.45-1.78 (m, 6H), 1.41 (s, 9H), 1.24 (s, 9H), 1.03 (s, 21H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 151.0, 146.5, 133.8, 129.8, 125.8, 122.8, 80.1, 63.0, 50.9, 34.5, 33.4, 32.6, 31.2, 28.1, 23.7, 18.0, 12.0;

IR (film) 2942, 2866, 1715, 1647, 1490, 1463, 1392, 1367, 1293, 1255, 1150, 1108, 1070, 1014 cm^{-1};

LRMS (ESI) calcd for $C_{31}H_{54}NaO_3SSi$ (M+Na) 557.35, found 557.35.



(*E*)-*tert*-Butyl 7-methyl 4-(4-*tert*-butylphenylthio)hept-2-enedioate (Table 2, entry 8). The compound was prepared according to the General Procedure from (±)-*tert*-butyl 7-methyl hepta-2,3-dienedioate (108 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 µL, 0.60 mmol). After purification by flash chromatography (25:1 → 10:1 hexane/ethyl acetate; second column: 45:1 → 35:1 toluene/Et_2O), the title compound was isolated as a colorless oil (146 mg, 74% yield) with 91% ee.

$[\alpha]^{23}_{D}$ = +79 (c = 1.0, $CHCl_3$).

HPLC analysis of the product: Daicel CHIRALPAK AS-H column; 3.0% MeOH in hexanes; 1.0 mL/min; retention times: 5.14 min (major), 6.00 min (minor).

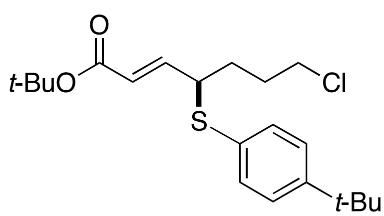
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (149 mg, 76% yield) with 91% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28 (s, 4H), 6.64 (dd, *J* = 15.6 Hz, *J* = 8.8 Hz, 1H), 5.43 (dd, *J* = 15.6 Hz, *J* = 0.8 Hz, 1H), 3.63 (s, 3H), 3.53-3.60 (m, 1H), 2.45-2.50 (m, 2H), 1.90-2.05 (m, 2H), 1.42 (s, 9H), 1.27 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.0, 165.2, 151.3, 145.4, 133.9, 129.0, 125.9, 123.3, 80.3, 51.6, 49.8, 34.5, 31.4, 31.1, 28.5, 28.0;

IR (film) 3410, 2965, 2870, 1739, 1713, 1647, 1490, 1437, 1393, 1367, 1291, 1256, 1149, 1120, 1014 cm^{-1};

LRMS (ESI) calcd for $C_{22}H_{32}NaO_4S$ (M+Na) 415.19, found 415.19.



(*E*)-*tert*-Butyl 4-(4-*tert*-butylphenylthio)-7-chlorohept-2-enoate (Table 2, entry 9). The compound was prepared according to the General Procedure from (±)-*tert*-butyl 7-chlorohepta-2,3-dienoate (108 mg, 0.50 mmol) and 4-*tert*-butylthiophenol (103 µL, 0.60 mmol). After purification by flash chromatography (4:1 → 3:1 hexane/CH_2Cl_2), the title compound was isolated as a colorless semisolid (122 mg, 64% yield) with 90% ee.

$[\alpha]^{24}_{D}$ = +96 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALCEL OD-H column; 5.0% MeOH in CO_2; 3.0 mL/min; retention times: 3.35 min (minor), 3.56 min (major).

The second run was performed with (*S*)-**1**. The product was isolated as a colorless semisolid (128 mg, 67% yield) with 90% ee.

Recrystallization from *n*-hexane gave a colorless needle, which was subjected to X-ray crystallography.

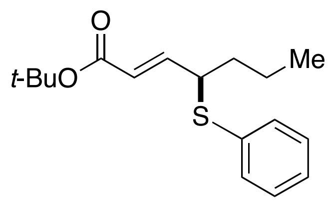
mp 67–68 °C;

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.29 (s, 4H), 6.65 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.40 (d, *J* = 15.6 Hz, 1H), 3.49-3.56 (m, 3H), 1.74-1.98 (m, 4H), 1.43 (s, 9H), 1.28 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.3, 151.3, 145.7, 134.0, 129.2, 125.9, 123.1, 80.3, 50.1, 44.4, 34.5, 31.2, 30.8, 30.1, 28.0;

IR (film) 2965, 2870, 1713, 1647, 1490, 1458, 1393, 1367, 1304, 1252, 1150, 1120, 1014 cm^{-1};

LRMS (ESI) calcd for $C_{21}H_{31}ClNaO_2S$ (M+Na) 405.16, found 405.16.



(*E*)-*tert*-Butyl 4-phenylthiohept-2-enoate (Table 3, entry 1). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and thiophenol (62 μL, 0.60 mmol). After purification by flash chromatography (6:1:1 → 2:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless oil (105 mg, 72% yield) with 89% ee.

$[\alpha]^{22}_{D}$ = +131 (c = 1.0, $CHCl_3$).

SFC analysis of the product after reduction of the ester by DIBAL-H: Daicel CHIRALPAK AD-H column; 10% 2-PrOH in CO_2; 3.0 mL/min; retention times: 3.05 min (major), 3.30 min (minor).

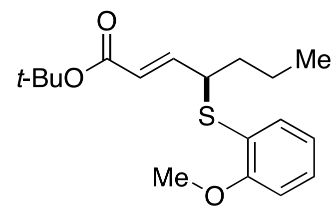
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (106 mg, 73% yield) with 91% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.32-7.36 (m, 2H), 7.20-7.28 (m, 3H), 6.64 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.41 (dd, *J* = 15.6 Hz, *J* = 0.4 Hz, 1H), 3.53-3.61 (m, 1H), 1.56-1.74 (m, 2H), 1.38-1.50 (m, 2H), 1.42 (s, 9H), 0.90 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.4, 146.4, 133.53, 133.46, 128.7, 127.6, 122.9, 80.2, 50.4, 35.7, 28.0, 20.4, 13.7;

IR (film) 3059, 3005, 2961, 2933, 2873, 1951, 1713, 1647, 1584, 1478, 1457, 1439, 1392, 1367, 1338, 1292, 1257, 1232, 1152, 1091, 1069, 1025 cm^{-1};

LRMS (EI) calcd for $C_{17}H_{24}O_2S$ (M+) 292.15, found 292.10.



(*E*)-*tert*-Butyl 4-((2-methoxyphenyl)thio)hept-2-enoate (Table 3, entry 2). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 2-methoxythiophenol (73 μL, 0.60 mmol). After purification by flash chromatography (40:1 → 20:1 hexane/Et_2O), the title compound was isolated as a colorless oil (105 mg, 65% yield) with 89% ee.

$[\alpha]^{22}_D$ = +162 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALCEL OD-H column; 5% MeOH in CO_2; 3.0 mL/min; retention times: 3.28 min (minor), 4.07 min (major).

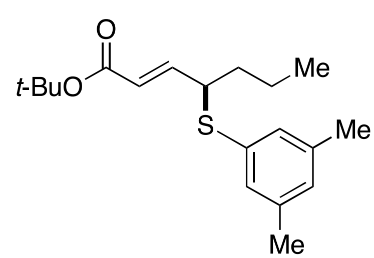
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (111 mg, 69% yield) with 90% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.30 (dd, *J* = 7.6 Hz, *J* = 1.6 Hz, 1H), 7.20-7.25 (m, 1H), 6.80-6.87 (m, 2H), 6.64 (dd, *J* = 15.4 Hz, *J* = 9.2 Hz, 1H), 5.36 (d, *J* = 15.4 Hz, 1H), 3.83 (s, 3H), 3.66-3.74 (m, 1H), 1.55-1.74 (m, 2H), 1.37-1.49 (m, 2H), 1.39 (s, 9H), 0.89 (t, *J* = 7.4 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 159.1, 146.7, 135.1, 129.4, 122.5, 121.4, 120.7, 110.7, 80.0, 55.6, 48.4, 35.7, 28.0, 20.5, 13.7;

IR (film) 3064, 3004, 2961, 2934, 2873, 2837, 1712, 1646, 1583, 1477, 1465, 1433, 1392, 1367, 1341, 1294, 1274, 1245, 1153, 1132, 1094, 1071, 1026 cm^{-1};

LRMS (EI) calcd for $C_{18}H_{26}O_3S$ (M+) 322.16, found 322.10.



(*E*)-*tert*-Butyl 4-((3,5-dimethylphenyl)thio)hept-2-enoate (Table 3, entry 3). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 3,5-dimethylthiophenol (82 μL, 0.60 mmol). After purification by flash chromatography (6:1:1 → 2:1:1 hexane/toluene/CH_2Cl_2), the title compound was isolated as a colorless oil (128 mg, 80% yield) with 92% ee.

$[\alpha]^{22}_D$ = +130 (c = 1.0, $CHCl_3$).

HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 1.0% 2-PrOH in hexanes; 1.0 mL/min; retention times: 8.2 min (major), 9.6 min (minor).

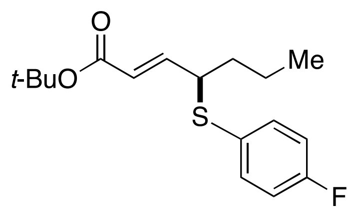
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (131 mg, 82% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.97 (s, 2H), 6.87 (s, 1H), 6.65 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.39 (dd, *J* = 15.6 Hz, *J* = 0.4 Hz, 1H), 3.50-3.57 (m, 1H), 2.25 (s, 6H), 1.56-1.73 (m, 2H), 1.38-1.48 (m, 2H), 1.44 (s, 9H), 0.90 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.6, 146.6, 138.3, 132.9, 131.4, 129.6, 122.8, 80.2, 50.5, 35.7, 28.1, 21.1, 20.5, 13.8;

IR (film) 2961, 2932, 2873, 2361, 1714, 1647, 1600, 1582, 1540, 1457, 1392, 1367, 1339, 1293, 1257, 1232, 1153, 1132, 1072, 1039 cm^{-1};

LRMS (EI) calcd for $C_{19}H_{28}O_2S$ (M+) 320.18, found 320.20.



(*E*)-*tert*-Butyl 4-((4-fluorophenyl)thio)hept-2-enoate (Table 3, entry 4). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 4-fluorothiophenol (64 μL, 0.60 mmol). After purification by flash chromatography (4:1 → 3:1 hexane/CH_2Cl_2), the title compound was isolated as a colorless oil (96 mg, 62% yield) with 92% ee.

$[\alpha]^{22}_{D}$ = +97 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALPAK AD-H column; 2.5% 2-PrOH in CO_2; 3.0 mL/min; retention times: 3.47 min (major), 3.80 min (minor).

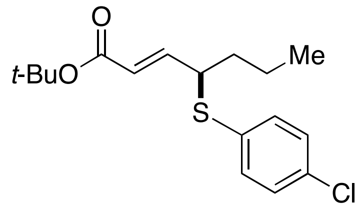
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (100 mg, 65% yield) with 90% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.31-7.37 (m, 2H), 6.92-6.99 (m, 2H), 6.59 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.31 (d, *J* = 15.6 Hz, 1H), 3.42-3.50 (m, 1H), 1.53-1.71 (m, 2H), 1.37-1.46 (m, 2H), 1.42 (s, 9H), 0.89 (t, *J* = 7.6 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.3, 162.7 (d, *J* = 247 Hz), 146.1, 136.6 (d, *J* = 8 Hz), 128.3 (d, *J* = 3 Hz), 122.9, 115.8 (d, *J* = 21 Hz), 80.3, 51.3, 35.4, 28.0, 20.4, 13.7;

IR (film) 2962, 2933, 2874, 2362, 2339, 1890, 1716, 1647, 1590, 1559, 1540, 1490, 1457, 1393, 1368, 1339, 1292, 1231, 1154, 1090 cm^{-1};

LRMS (EI) calcd for $C_{17}H_{23}FO_2S$ (M+) 310.14, found 310.10.



(*E*)-*tert*-Butyl 4-((4-chlorophenyl)thio)hept-2-enoate (Table 3, entry 5). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 4-chlorothiophenol (87 mg, 0.60 mmol). After purification by flash chromatography (9:2 → 1:1 hexane/CH_2Cl_2), the title compound was isolated as a colorless oil (97 mg, 60% yield) with 92% ee.

$[\alpha]^{22}_{D}$ = +127 (c = 1.0, $CHCl_3$).

SFC analysis of the product: Daicel CHIRALPAK AD-H column; 2.5% 2-PrOH in CO_2; 3.0 mL/min; retention times: 6.18 min (major), 7.36 min (minor).

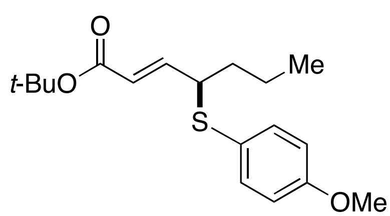
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (93 mg, 57% yield) with 90% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 2H), 6.60 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.39 (dd, *J* = 15.6 Hz, *J* = 0.8 Hz, 1H), 3.50-3.57 (m, 1H), 1.58-1.72 (m, 2H), 1.37-1.47 (m, 2H), 1.43 (s, 9H), 0.90 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.4, 146.1, 135.0, 134.0, 132.1, 129.0, 123.2, 80.5, 50.8, 35.6, 28.1, 20.5, 13.7;

IR (film) 2962, 2933, 2873, 1713, 1647, 1573, 1477, 1458, 1391, 1368, 1338, 1293, 1258, 1233, 1153, 1133, 1095, 1040, 1014 cm^{-1};

LRMS (EI) calcd for $C_{17}H_{23}ClO_2S$ (M+) 326.11, found 326.10.



(*E*)-*tert*-Butyl 4-((4-methoxyphenyl)thio)hept-2-enoate (Table 3, entry 6). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 4-methoxythiophenol (74 μL, 0.60 mmol). After purification by flash chromatography (4:1 → 1:1 hexane/CH_2Cl_2), the title compound was isolated as a colorless oil (127 mg, 79% yield) with 89% ee.

$[\alpha]^{22}_{D}$ = +100 (c = 1.0, $CHCl_3$).

HPLC analysis of the product: Daicel CHIRALPAK AS-H column; 1% 2-PrOH in hexanes; 1.0 mL/min; retention times: 5.23 min (major), 5.67 min (minor).

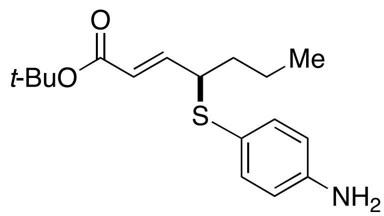
The second run was performed with (*S*)-**1**. The product was isolated as a colorless oil (118 mg, 73% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.29 (d, *J* = 7.8 Hz, 2H), 6.78 (d, *J* = 7.8 Hz, 2H), 6.61 (dd, *J* = 15.6 Hz, *J* = 9.2 Hz, 1H), 5.28 (d, *J* = 15.6 Hz, 1H), 3.75 (s, 3H), 3.36-3.43 (m, 1H), 1.50-1.70 (m, 2H), 1.36-1.46 (m, 2H), 1.42 (s, 9H), 0.88 (t, *J* = 7.4 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 159.8, 146.6, 136.8, 123.4, 122.6, 114.2, 80.1, 55.2, 51.5, 35.4, 27.9, 20.5, 13.7;

IR (film) 3004, 2961, 2933, 2873, 2838, 2533, 2390, 2290, 2045, 1888, 1710, 1646, 1592, 1571, 1494, 1464, 1405, 1392, 1367, 1337, 1287, 1248, 1150, 1104, 1072, 1033, 1008 cm^{-1};

LRMS (EI) calcd for $C_{18}H_{26}O_3S$ (M+) 322.16, found 322.20.



(*E*)-*tert*-Butyl 4-((4-aminophenyl)thio)hept-2-enoate (Table 3, entry 7). The compound was prepared according to the General Procedure from (±)-*tert*-butyl hepta-2,3-dienoate (91 mg, 0.50 mmol) and 4-aminothiophenol (75 mg, 0.60 mmol). The ee of the unpurified mixture was 86%. Purification by semi-preparative HPLC (Daicel Chiralcel OD-H, 1 cm x 25 cm, 92:8 hexane/2-PrOH, 10 cycles) gave the title compound as a light-yellow oil (96 mg, 63% yield).

$[\alpha]^{23}_{D}$ = +118 (c = 1.0, $CHCl_3$, 86% ee).

HPLC analysis of the product: Daicel CHIRALCEL OD-H column; 10% 2-PrOH in hexanes; 1.0 mL/min; retention times: 12.7 min (major), 13.5 min (minor).

The second run was performed with (*S*)-**1**. The ee of the unpurified mixture was 86%. Purification by semi-preparative HPLC gave the title compound as a light-yellow oil (100 mg, 65% yield).

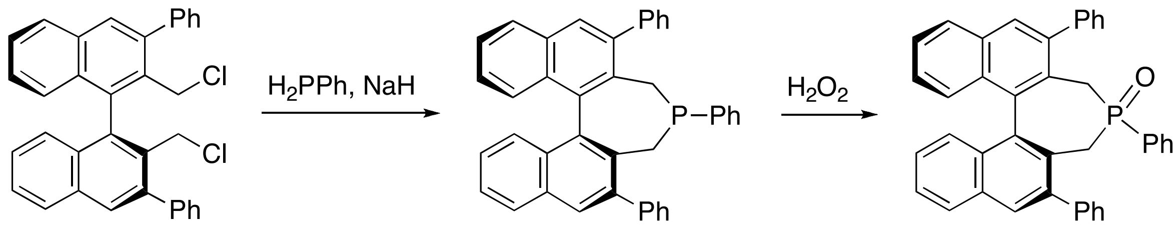
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.16 (d, *J* = 8.4 Hz, 2H), 6.61 (dd, *J* = 15.4 Hz, *J* = 9.4 Hz, 1H), 6.54 (d, *J* = 8.4 Hz, 2H), 5.29 (d, *J* = 15.4 Hz, 1H), 3.75 (br s, 2H), 3.29-3.36 (m, 1H), 1.57-1.67 (m, 1H), 1.47-1.56 (m, 1H), 1.43 (s, 9H), 1.33-1.45 (m, 2H), 0.87 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.6, 146.9, 146.8, 137.0, 122.3, 120.0, 115.1, 80.1, 51.6, 35.3, 28.1, 20.4, 13.7;

IR (film) 3471, 3375, 3223, 3027, 2961, 2932, 2873, 1700, 1624, 1598, 1496, 1457, 1424, 1392, 1368, 1338, 1294, 1257, 1234, 1153, 1095, 1072, 1040 cm^{-1};

LRMS (ESI) calcd for $C_{17}H_{25}NNaSO_2$ (M+Na) 330.15, found 330.15.

IV. Preparation of Catalyst 1



(*S*)-**1**

(*S*)-2,2'-Bis(chloromethyl)-3,3'-diphenyl-1,1'-binaphthalene[2,3] (6.78 g, 13.5 mmol) and a stir bar were added to an oven-dried 1000-mL flask, which was then evacuated and back-filled with nitrogen (three cycles). THF (650 mL; degassed, anhydrous), phenylphosphine (1.60 mL, 14.5 mmol), and sodium hydride (782 mg, 32.6 mmol) were added in turn, the flask was purged with argon for 1 min, and then the resulting mixture was stirred vigorously at 60 °C for 1 day. Next, phenylphosphine (0.30 mL, 2.7 mmol) and sodium hydride (170 mg, 7.08 mmol) were added in turn, the flask was purged with argon for 1 min, and the reaction was stirred at 60 °C for an additional 14 h (the disappearance of starting material was monitored by reverse-phase HPLC after oxidation of an aliquot by *t*-BuOOH). After the starting material had been consumed, the THF was removed under reduced pressure, CH_2Cl_2 (200 mL) was added, and then water (200 mL) was cautiously added with stirring. The phases were separated, and the aqueous phase was extracted with CH_2Cl_2 (100 mL). The combined organic phases were treated with 30% H_2O_2 (7.63 g, 67.3 mmol) and stirred at r.t. for 30 min. Then, an aqueous solution of $Na_2S_2O_3$ (150 mL; 10%) was cautiously added. The phases were separated, and the aqueous phase was extracted with CH_2Cl_2 (100 mL). The combined organic phases were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (9:0 → 9:1 CH_2Cl_2/Et_2O), which furnished 6.94 g of impure phosphine oxide. This material was dissolved in hot toluene (30 mL), and then the solution was allowed to cool to r.t. over 1 h and stirred at r.t. for an additional 30 min. The resulting white solid was collected by filtration, washed with toluene (3 mL) and hexane (10 mL), and dried under reduced pressure to afford the desired phosphine oxide (4.70 g, 63%) as a white powder.

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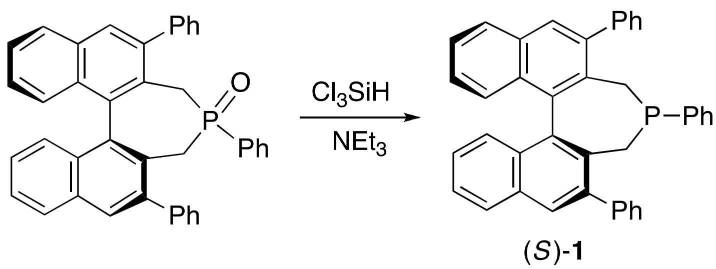
(2) Ooi, T.; Kameda, M.; Maruoka, K. *J. Am. Chem. Soc.* **2003**, *125*, 5139–5151.
(3) Zhou, Y.-G.; Zhang, X. *Chem. Commun.* **2002**, 1124–1125.



(11bR)-2,4,6-Triphenyl-4,5-dihydro-3H-dinaphtho[2,1-c:1',2'-e]phosphepine 4-oxide (4.70 g, 8.44 mmol) and a stir bar were added to an oven-dried 500-mL flask, which was then evacuated and back-filled with nitrogen (three cycles). Toluene (169 mL; degassed, anhydrous) was added via syringe. Triethylamine (8.23 mL, 59.2 mmol) and trichlorosilane (4.26 mL, 42.2 mmol) were added in turn, and the resulting slurry was stirred at 80 °C for 14 h. Next, the reaction mixture was allowed to cool to r.t., and degassed water (170 mL) was added dropwise over 5 min. The resulting slurry was stirred for 10 min, and then Celite (10 g) was added. In the air, the mixture was filtered through a pad of Celite, and the solid was washed with additional toluene (100 mL). The aqueous phase was separated, and the organic phase was washed successively with an aqueous solution of NaOH (170 mL; 0.5 M) and water (170 mL), dried over Na_2SO_4, and concentrated by rotary evaporation under reduced pressure. Next, the flask was back-filled with nitrogen, and under a nitrogen atmosphere the residue was dissolved in hot toluene (4.6 mL) and then cooled to 60 °C. Next, hexane (13.8 mL) was added dropwise over 1 min, and the resulting solution was cooled to r.t. with stirring, during which time a white precipitate was observed. After a significant amount of precipitation had occurred, the slurry was warmed to reflux for 1 min (white slurry) and then allowed to cool to 60 °C. Hexane (23 mL) was added dropwise over 3 min while maintaining the temperature near 60 °C. Next, the slurry was allowed to cool to r.t. with stirring for 2.5 h. The crystals were filtered in the air, washed with hexane (3 mL x3), and dried under reduced pressure to afford phosphepine **1** (3.57 g, 78%) as a white powder.

$[\alpha]^{24}_D = -117$ (c = 1.0, $CHCl_3$).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.93 (d, *J* = 8.0 Hz, 1H), 7.90 (s, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 7.2 Hz, 2H), 7.64 (s, 1H), 7.33-7.48 (m, 5H), 7.03-7.28 (m, 11H), 6.80-6.86 (m, 2H), 6.50-6.90 (br s, 1H), 3.21 (dd, *J* = 14.4, 4.4 Hz, 1H), 2.96 (dd, *J* = 14.8, 11.6 Hz, 1H), 2.80 (dd, *J* = 11.6, 2.4 Hz, 1H), 2.76 (dd, *J* = 14.4, 11.6 Hz, 1H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 141.3 (*J* = 33 Hz), 140.14 (*J* = 5 Hz), 140.12 (*J* = 8 Hz), 136.1 (*J* = 23 Hz), 134.7 (*J* = 5 Hz), 134.0 (*J* = 1 Hz), 132.2, 132.1 (*J* = 2 Hz), 131.66 (*J* = 1 Hz), 131.64 (2C), 131.62, 131.56, 131.4, 131.2, 129.8 (*J* = 5 Hz), 129.5, 129.2 (*J* = 2 Hz), 128.7, 128.23, 128.18, 128.14, 128.12, 128.10, 127.2, 126.63, 126.60, 126.2, 125.9, 125.8, 125.5 (*J* = 1 Hz), 125.2, 28.0 (*J* = 25 Hz), 25.6 (*J* = 16 Hz);

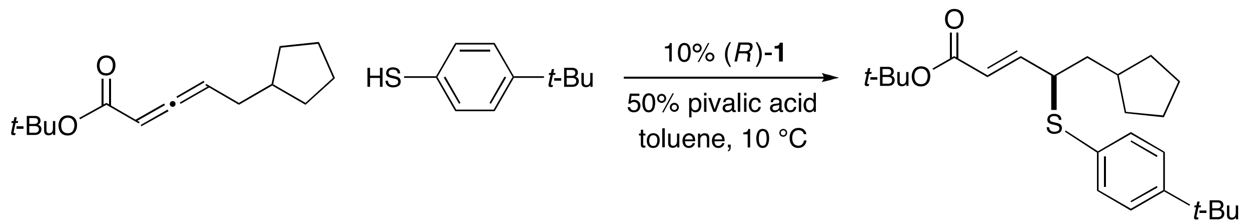
^{31}P NMR ($CDCl_3$, 162 MHz) δ 5.2;

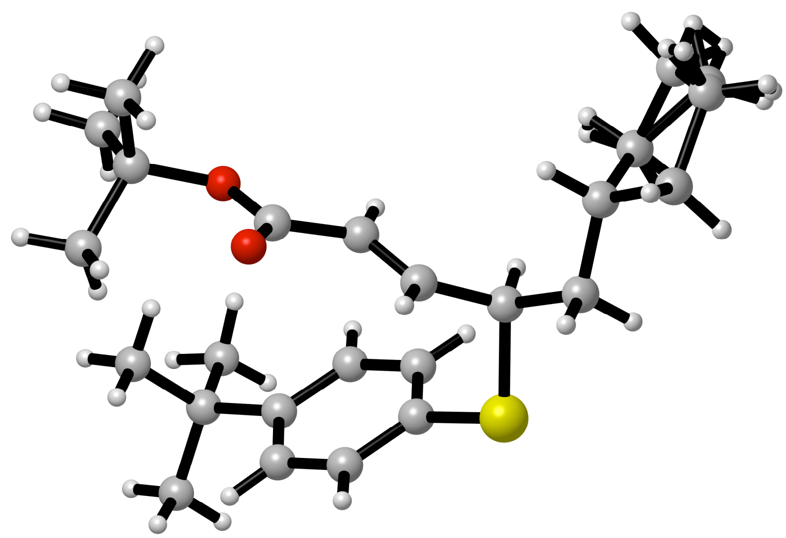
IR (film) 3054, 1587, 1494, 1433, 1328, 1214, 1072, 1026, 1001 cm^{-1};

LRMS (ESI) calcd for $C_{40}H_{30}P$ (M+H) 541.21, found 541.21.

V. Determination of Absolute Configuration

Table 2, entry 3:





The cyclopentane ring is disordered.

Table 1. Crystal data and structure refinement for d10056.

Identification code	d10056	
Empirical formula	C24 H36 O2 S	
Formula weight	388.59	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 6.01460(10) Å	α= 90°.
	b = 18.8648(3) Å	β= 90°.
	c = 20.1082(3) Å	γ = 90°.
Volume	2281.56(6) $Å^3$	
Z	4	
Density (calculated)	1.131 Mg/m^3	
Absorption coefficient	1.360 mm^{-1}	
F(000)	848	
Crystal size	0.40 x 0.35 x 0.30 mm^3	
Theta range for data collection	3.21 to 69.32°.	
Index ranges	-7<=h<=7, -22<=k<=20, -24<=l<=24	
Reflections collected	45400	
Independent reflections	4267 [R(int) = 0.0304]	
Completeness to theta = 69.32°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.6857 and 0.6122	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4267 / 12 / 260	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0279, wR2 = 0.0721	
R indices (all data)	R1 = 0.0285, wR2 = 0.0728	
Absolute structure parameter	0.008(11)	
Largest diff. peak and hole	0.171 and -0.179 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for d10056. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	1378(1)	6617(1)	4737(1)	21(1)
C(1)	3628(2)	6513(1)	4117(1)	19(1)
C(2)	4185(2)	5743(1)	4070(1)	20(1)
C(3)	6027(2)	5450(1)	4308(1)	20(1)
C(4)	6342(2)	4669(1)	4294(1)	18(1)
O(1)	5136(2)	4257(1)	4008(1)	22(1)
O(2)	8160(2)	4493(1)	4649(1)	20(1)
C(5)	8830(2)	3743(1)	4728(1)	19(1)
C(6)	6957(2)	3317(1)	5042(1)	24(1)
C(7)	9562(2)	3448(1)	4058(1)	26(1)
C(8)	10796(2)	3794(1)	5202(1)	24(1)
C(9)	2757(2)	6831(1)	3463(1)	20(1)
C(10)	4567(2)	6906(1)	2939(1)	23(1)
C(11)	3711(3)	7177(1)	2266(1)	31(1)
C(12)	5769(3)	7471(1)	1913(1)	36(1)
C(13)	7515(9)	7576(4)	2445(2)	34(1)
C(13A)	7070(12)	7847(5)	2460(3)	33(2)
C(14)	6388(3)	7446(1)	3114(1)	33(1)
C(21)	2645(2)	6220(1)	5445(1)	20(1)
C(22)	1713(2)	5615(1)	5724(1)	22(1)
C(23)	2725(2)	5296(1)	6266(1)	23(1)
C(24)	4698(2)	5558(1)	6538(1)	19(1)
C(31)	5822(2)	5165(1)	7112(1)	23(1)
C(32)	4165(3)	5026(1)	7677(1)	31(1)
C(33)	7787(3)	5580(1)	7402(1)	29(1)
C(34)	6686(3)	4453(1)	6844(1)	34(1)
C(25)	5597(2)	6163(1)	6249(1)	24(1)
C(26)	4583(3)	6494(1)	5711(1)	25(1)

Table 3. Bond lengths [Å] and angles [°] for d10056.

S(1)-C(21)	1.7809(13)
S(1)-C(1)	1.8507(14)
C(1)-C(2)	1.4924(18)
C(1)-C(9)	1.5379(17)
C(1)-H(1A)	1.0000
C(2)-C(3)	1.3273(19)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.4873(18)
C(3)-H(3A)	0.9500
C(4)-O(1)	1.2078(17)
C(4)-O(2)	1.3478(16)
O(2)-C(5)	1.4793(14)
C(5)-C(7)	1.5213(18)
C(5)-C(8)	1.5215(18)
C(5)-C(6)	1.5219(18)
C(6)-H(6A)	0.9800
C(6)-H(6B)	0.9800
C(6)-H(6C)	0.9800
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-C(10)	1.5213(19)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-C(14)	1.536(2)
C(10)-C(11)	1.5365(19)
C(10)-H(10A)	1.0000
C(11)-C(12)	1.531(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(13)	1.513(5)

C(12)-C(13A)	1.525(6)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(12)-H(12C)	0.9900
C(12)-H(12D)	0.9900
C(13)-C(14)	1.526(5)
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(13A)-C(14)	1.572(6)
C(13A)-H(13C)	0.9900
C(13A)-H(13D)	0.9900
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(14)-H(14C)	0.9900
C(14)-H(14D)	0.9900
C(21)-C(26)	1.383(2)
C(21)-C(22)	1.3890(19)
C(22)-C(23)	1.386(2)
C(22)-H(22A)	0.9500
C(23)-C(24)	1.3964(19)
C(23)-H(23A)	0.9500
C(24)-C(25)	1.3915(19)
C(24)-C(31)	1.5292(18)
C(31)-C(33)	1.533(2)
C(31)-C(32)	1.534(2)
C(31)-C(34)	1.537(2)
C(32)-H(32A)	0.9800
C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800
C(33)-H(33A)	0.9800
C(33)-H(33B)	0.9800
C(33)-H(33C)	0.9800
C(34)-H(34A)	0.9800
C(34)-H(34B)	0.9800
C(34)-H(34C)	0.9800
C(25)-C(26)	1.389(2)

C(25)-H(25A)	0.9500
C(26)-H(26A)	0.9500

C(21)-S(1)-C(1)	100.42(6)
C(2)-C(1)-C(9)	113.69(10)
C(2)-C(1)-S(1)	108.07(9)
C(9)-C(1)-S(1)	106.58(9)
C(2)-C(1)-H(1A)	109.5
C(9)-C(1)-H(1A)	109.5
S(1)-C(1)-H(1A)	109.5
C(3)-C(2)-C(1)	124.71(13)
C(3)-C(2)-H(2A)	117.6
C(1)-C(2)-H(2A)	117.6
C(2)-C(3)-C(4)	120.82(12)
C(2)-C(3)-H(3A)	119.6
C(4)-C(3)-H(3A)	119.6
O(1)-C(4)-O(2)	125.52(12)
O(1)-C(4)-C(3)	124.79(12)
O(2)-C(4)-C(3)	109.70(11)
C(4)-O(2)-C(5)	120.86(10)
O(2)-C(5)-C(7)	109.51(10)
O(2)-C(5)-C(8)	102.60(10)
C(7)-C(5)-C(8)	110.62(11)
O(2)-C(5)-C(6)	110.35(10)
C(7)-C(5)-C(6)	112.89(11)
C(8)-C(5)-C(6)	110.38(11)
C(5)-C(6)-H(6A)	109.5
C(5)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
C(5)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
C(5)-C(7)-H(7A)	109.5
C(5)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(5)-C(7)-H(7C)	109.5

H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(5)-C(8)-H(8A)	109.5
C(5)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(5)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(10)-C(9)-C(1)	112.62(11)
C(10)-C(9)-H(9A)	109.1
C(1)-C(9)-H(9A)	109.1
C(10)-C(9)-H(9B)	109.1
C(1)-C(9)-H(9B)	109.1
H(9A)-C(9)-H(9B)	107.8
C(9)-C(10)-C(14)	114.41(12)
C(9)-C(10)-C(11)	113.66(12)
C(14)-C(10)-C(11)	102.71(12)
C(9)-C(10)-H(10A)	108.6
C(14)-C(10)-H(10A)	108.6
C(11)-C(10)-H(10A)	108.6
C(12)-C(11)-C(10)	104.94(13)
C(12)-C(11)-H(11A)	110.8
C(10)-C(11)-H(11A)	110.8
C(12)-C(11)-H(11B)	110.8
C(10)-C(11)-H(11B)	110.8
H(11A)-C(11)-H(11B)	108.8
C(13)-C(12)-C(13A)	21.89(17)
C(13)-C(12)-C(11)	106.3(2)
C(13A)-C(12)-C(11)	104.4(3)
C(13)-C(12)-H(12A)	110.5
C(13A)-C(12)-H(12A)	129.3
C(11)-C(12)-H(12A)	110.5
C(13)-C(12)-H(12B)	110.5
C(13A)-C(12)-H(12B)	91.6
C(11)-C(12)-H(12B)	110.5
H(12A)-C(12)-H(12B)	108.7

C(13)-C(12)-H(12C)	90.4
C(13A)-C(12)-H(12C)	110.9
C(11)-C(12)-H(12C)	110.9
H(12A)-C(12)-H(12C)	21.6
H(12B)-C(12)-H(12C)	125.3
C(13)-C(12)-H(12D)	127.4
C(13A)-C(12)-H(12D)	110.9
C(11)-C(12)-H(12D)	110.9
H(12A)-C(12)-H(12D)	89.9
H(12B)-C(12)-H(12D)	20.7
H(12C)-C(12)-H(12D)	108.9
C(12)-C(13)-C(14)	107.1(3)
C(12)-C(13)-H(13A)	110.3
C(14)-C(13)-H(13A)	110.3
C(12)-C(13)-H(13B)	110.3
C(14)-C(13)-H(13B)	110.3
H(13A)-C(13)-H(13B)	108.6
C(12)-C(13A)-C(14)	104.3(4)
C(12)-C(13A)-H(13C)	110.9
C(14)-C(13A)-H(13C)	110.9
C(12)-C(13A)-H(13D)	110.9
C(14)-C(13A)-H(13D)	110.9
H(13C)-C(13A)-H(13D)	108.9
C(13)-C(14)-C(10)	102.8(2)
C(13)-C(14)-C(13A)	21.40(17)
C(10)-C(14)-C(13A)	108.2(2)
C(13)-C(14)-H(14A)	111.2
C(10)-C(14)-H(14A)	111.2
C(13A)-C(14)-H(14A)	90.2
C(13)-C(14)-H(14B)	111.2
C(10)-C(14)-H(14B)	111.2
C(13A)-C(14)-H(14B)	124.8
H(14A)-C(14)-H(14B)	109.1
C(13)-C(14)-H(14C)	93.7
C(10)-C(14)-H(14C)	110.0
C(13A)-C(14)-H(14C)	110.0

H(14A)-C(14)-H(14C)	124.6
H(14B)-C(14)-H(14C)	19.0
C(13)-C(14)-H(14D)	130.1
C(10)-C(14)-H(14D)	110.0
C(13A)-C(14)-H(14D)	110.0
H(14A)-C(14)-H(14D)	21.3
H(14B)-C(14)-H(14D)	90.8
H(14C)-C(14)-H(14D)	108.4
C(26)-C(21)-C(22)	119.41(13)
C(26)-C(21)-S(1)	120.86(11)
C(22)-C(21)-S(1)	119.70(11)
C(23)-C(22)-C(21)	119.77(13)
C(23)-C(22)-H(22A)	120.1
C(21)-C(22)-H(22A)	120.1
C(22)-C(23)-C(24)	121.90(12)
C(22)-C(23)-H(23A)	119.1
C(24)-C(23)-H(23A)	119.1
C(25)-C(24)-C(23)	117.12(12)
C(25)-C(24)-C(31)	122.81(12)
C(23)-C(24)-C(31)	120.03(12)
C(24)-C(31)-C(33)	112.32(12)
C(24)-C(31)-C(32)	110.76(12)
C(33)-C(31)-C(32)	107.84(12)
C(24)-C(31)-C(34)	107.93(11)
C(33)-C(31)-C(34)	108.59(13)
C(32)-C(31)-C(34)	109.35(13)
C(31)-C(32)-H(32A)	109.5
C(31)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(31)-C(33)-H(33A)	109.5
C(31)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(31)-C(33)-H(33C)	109.5

H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(31)-C(34)-H(34A)	109.5
C(31)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
C(31)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(26)-C(25)-C(24)	121.58(13)
C(26)-C(25)-H(25A)	119.2
C(24)-C(25)-H(25A)	119.2
C(21)-C(26)-C(25)	120.20(13)
C(21)-C(26)-H(26A)	119.9
C(25)-C(26)-H(26A)	119.9

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for d10056. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

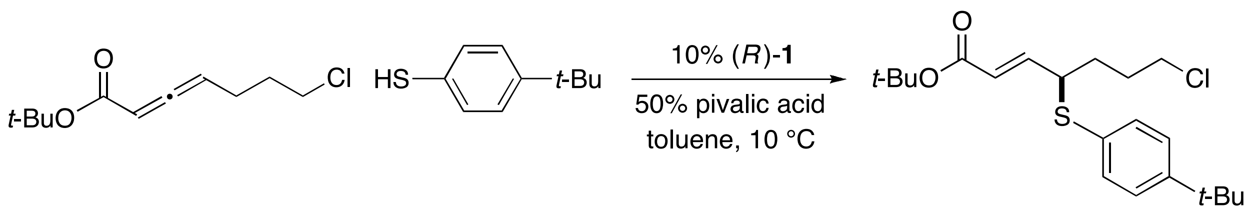
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	23(1)	19(1)	20(1)	3(1)	0(1)	5(1)
C(1)	19(1)	19(1)	20(1)	0(1)	1(1)	1(1)
C(2)	23(1)	20(1)	16(1)	0(1)	-1(1)	0(1)
C(3)	20(1)	18(1)	21(1)	1(1)	-1(1)	0(1)
C(4)	17(1)	21(1)	17(1)	1(1)	3(1)	2(1)
O(1)	24(1)	20(1)	24(1)	-2(1)	-4(1)	1(1)
O(2)	19(1)	15(1)	25(1)	1(1)	-3(1)	3(1)
C(5)	21(1)	14(1)	22(1)	2(1)	1(1)	3(1)
C(6)	26(1)	20(1)	26(1)	4(1)	1(1)	0(1)
C(7)	29(1)	24(1)	24(1)	1(1)	2(1)	8(1)
C(8)	22(1)	22(1)	28(1)	0(1)	-3(1)	4(1)
C(9)	21(1)	18(1)	23(1)	2(1)	-4(1)	2(1)
C(10)	26(1)	21(1)	21(1)	1(1)	0(1)	6(1)
C(11)	35(1)	34(1)	24(1)	6(1)	-4(1)	-4(1)
C(12)	39(1)	38(1)	29(1)	11(1)	7(1)	5(1)
C(13)	32(2)	29(3)	39(2)	6(2)	8(2)	-2(2)
C(13A)	26(3)	31(4)	43(2)	7(2)	9(2)	3(2)
C(14)	24(1)	44(1)	31(1)	4(1)	0(1)	-4(1)
C(21)	24(1)	16(1)	19(1)	-1(1)	2(1)	5(1)
C(22)	19(1)	24(1)	23(1)	0(1)	0(1)	-2(1)
C(23)	23(1)	22(1)	23(1)	5(1)	1(1)	-4(1)
C(24)	22(1)	20(1)	15(1)	-3(1)	3(1)	0(1)
C(31)	26(1)	25(1)	18(1)	2(1)	0(1)	0(1)
C(32)	34(1)	41(1)	20(1)	8(1)	0(1)	-4(1)
C(33)	28(1)	38(1)	19(1)	1(1)	-4(1)	-1(1)
C(34)	40(1)	30(1)	33(1)	0(1)	-10(1)	10(1)
C(25)	29(1)	22(1)	21(1)	-2(1)	-3(1)	-7(1)
C(26)	35(1)	16(1)	24(1)	1(1)	-2(1)	-7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for d10056.

	x	y	z	U(eq)
H(1A)	4969	6783	4266	23
H(2A)	3146	5442	3854	23
H(3A)	7158	5744	4490	23
H(6A)	6408	3566	5437	36
H(6B)	7514	2848	5171	36
H(6C)	5743	3262	4721	36
H(7A)	8304	3455	3749	39
H(7B)	10083	2960	4115	39
H(7C)	10772	3740	3880	39
H(8A)	11933	4105	5009	36
H(8B)	11423	3321	5273	36
H(8C)	10297	3990	5628	36
H(9A)	1559	6524	3286	25
H(9B)	2107	7303	3554	25
H(10A)	5278	6433	2870	28
H(11A)	3039	6787	2006	37
H(11B)	2584	7554	2329	37
H(12A)	6303	7132	1572	43
H(12B)	5419	7927	1693	43
H(12C)	6659	7083	1715	43
H(12D)	5341	7807	1557	43
H(13A)	8114	8065	2426	40
H(13B)	8758	7239	2381	40
H(13C)	6655	8354	2485	40
H(13D)	8690	7809	2381	40
H(14A)	5739	7888	3294	40
H(14B)	7447	7248	3442	40
H(14C)	7694	7199	3305	40
H(14D)	5824	7788	3448	40
H(22A)	387	5421	5543	26

H(23A)	2057	4889	6458	27
H(32A)	3519	5477	7824	47
H(32B)	4938	4801	8051	47
H(32C)	2979	4713	7518	47
H(33A)	8934	5638	7060	43
H(33B)	8407	5320	7781	43
H(33C)	7276	6047	7549	43
H(34A)	5430	4165	6690	52
H(34B)	7477	4201	7198	52
H(34C)	7703	4540	6472	52
H(25A)	6937	6355	6423	29
H(26A)	5223	6910	5526	30

Table 2, entry 9:



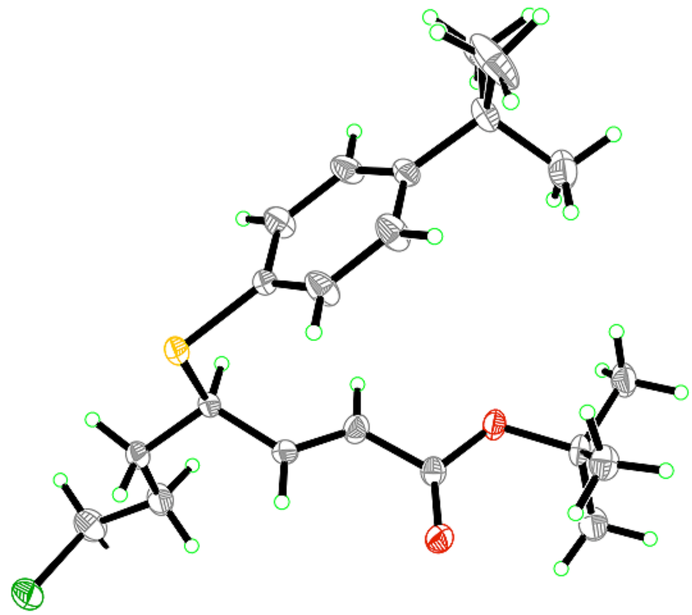


Table 1. Crystal data and structure refinement for yf1.

Identification code	yf1	
Empirical formula	C21 H31 Cl O2 S	
Formula weight	383.00	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21	
Unit cell dimensions	a = 5.8536(7) Å	α= 90°.
	b = 21.778(2) Å	β= 99.580(2)°.
	c = 17.441(2) Å	γ = 90°.
Volume	2192.4(4) $Å^3$	
Z	4	
Density (calculated)	1.179 Mg/m^3	
Absorption coefficient	0.281 mm^{-1}	
F(000)	848	
Crystal size	0.40 x 0.25 x 0.15 mm^3	
Theta range for data collection	1.18 to 29.57°.	
Index ranges	-8<=h<=8, -30<=k<=30, -24<=l<=24	
Reflections collected	58806	
Independent reflections	12186 [R(int) = 0.0482]	
Completeness to theta = 29.57°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9591 and 0.8959	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12186 / 385 / 464	
Goodness-of-fit on F^2	1.010	
Final R indices [I>2sigma(I)]	R1 = 0.0351, wR2 = 0.0773	
R indices (all data)	R1 = 0.0387, wR2 = 0.0796	
Absolute structure parameter	-0.04(4)	
Largest diff. peak and hole	0.244 and -0.171 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for yf1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	5513(1)	9620(1)	5612(1)	21(1)
Cl(1)	5227(1)	7822(1)	7985(1)	35(1)
O(1)	-1880(3)	8656(1)	3462(1)	22(1)
O(2)	902(3)	7931(1)	3818(1)	27(1)
C(1)	3114(4)	9135(1)	5832(1)	19(1)
C(2)	2418(4)	8710(1)	5158(1)	20(1)
C(3)	468(4)	8781(1)	4654(1)	23(1)
C(4)	-113(4)	8396(1)	3944(1)	20(1)
C(5)	3948(4)	8814(1)	6612(1)	22(1)
C(6)	2116(4)	8383(1)	6830(1)	28(1)
C(7)	2515(5)	8218(1)	7695(1)	32(1)
C(8)	4064(4)	10024(1)	4787(1)	19(1)
C(9)	2289(5)	10431(1)	4861(1)	27(1)
C(10)	1137(4)	10749(1)	4215(1)	27(1)
C(11)	1718(4)	10666(1)	3482(1)	20(1)
C(12)	3515(5)	10263(1)	3422(1)	29(1)
C(13)	4676(5)	9942(1)	4057(1)	28(1)
C(14)	419(4)	10996(1)	2757(1)	24(1)
C(15)	-1325(5)	11475(1)	2967(2)	37(1)
C(16)	-939(6)	10520(1)	2222(1)	41(1)
C(17)	2153(5)	11341(2)	2348(2)	56(1)
C(18)	-2773(4)	8385(1)	2693(1)	20(1)
C(19)	-846(4)	8362(1)	2201(1)	26(1)
C(20)	-3840(5)	7761(1)	2797(1)	30(1)
C(21)	-4627(5)	8839(1)	2349(1)	29(1)
S(2)	8896(1)	5615(1)	3111(1)	21(1)
Cl(2)	16990(1)	7413(1)	5357(1)	37(1)
O(3)	13943(3)	6515(1)	845(1)	19(1)
O(4)	11537(3)	7221(1)	1251(1)	22(1)
C(31)	11695(4)	6034(1)	3294(1)	18(1)
C(32)	11709(4)	6465(1)	2630(1)	18(1)

C(33)	12765(4)	6336(1)	2033(1)	18(1)
C(34)	12624(4)	6749(1)	1348(1)	18(1)
C(35)	11892(4)	6341(1)	4091(1)	20(1)
C(36)	14145(4)	6707(1)	4298(1)	22(1)
C(37)	14432(4)	6941(1)	5124(1)	27(1)
C(38)	9270(4)	5202(1)	2264(1)	18(1)
C(39)	8151(4)	5387(1)	1536(1)	21(1)
C(40)	8522(4)	5072(1)	868(1)	22(1)
C(41)	10038(4)	4577(1)	912(1)	20(1)
C(42)	11158(4)	4397(1)	1652(1)	26(1)
C(43)	10775(4)	4703(1)	2318(1)	25(1)
C(44)	10471(4)	4228(1)	184(1)	26(1)
C(45)	9686(5)	3561(1)	234(2)	32(1)
C(46)	13070(5)	4239(1)	146(2)	43(1)
C(47)	9129(6)	4506(1)	-565(1)	38(1)
C(48)	14211(4)	6850(1)	132(1)	19(1)
C(49)	15451(4)	7456(1)	343(1)	24(1)
C(50)	11870(4)	6924(1)	-396(1)	24(1)
C(51)	15751(4)	6420(1)	-250(1)	26(1)

Table 3. Bond lengths [Å] and angles [°] for yf1.

S(1)-C(8)	1.778(2)
S(1)-C(1)	1.848(2)
Cl(1)-C(7)	1.803(3)
O(1)-C(4)	1.344(3)
O(1)-C(18)	1.480(2)
O(2)-C(4)	1.212(3)
C(1)-C(2)	1.497(3)
C(1)-C(5)	1.535(3)
C(2)-C(3)	1.329(3)
C(3)-C(4)	1.487(3)
C(5)-C(6)	1.521(3)
C(6)-C(7)	1.531(3)
C(8)-C(9)	1.388(3)
C(8)-C(13)	1.389(3)
C(9)-C(10)	1.396(3)
C(10)-C(11)	1.387(3)
C(11)-C(12)	1.387(3)
C(11)-C(14)	1.542(3)
C(12)-C(13)	1.388(3)
C(14)-C(16)	1.527(3)
C(14)-C(17)	1.531(4)
C(14)-C(15)	1.545(3)
C(18)-C(21)	1.517(3)
C(18)-C(20)	1.518(3)
C(18)-C(19)	1.527(3)
S(2)-C(38)	1.774(2)
S(2)-C(31)	1.856(2)
Cl(2)-C(37)	1.807(3)
O(3)-C(34)	1.360(2)
O(3)-C(48)	1.472(2)
O(4)-C(34)	1.206(2)
C(31)-C(32)	1.492(3)
C(31)-C(35)	1.530(3)
C(32)-C(33)	1.327(3)

C(33)-C(34)	1.488(3)
C(35)-C(36)	1.532(3)
C(36)-C(37)	1.510(3)
C(38)-C(39)	1.389(3)
C(38)-C(43)	1.392(3)
C(39)-C(40)	1.399(3)
C(40)-C(41)	1.389(3)
C(41)-C(42)	1.403(3)
C(41)-C(44)	1.536(3)
C(42)-C(43)	1.390(3)
C(44)-C(45)	1.530(3)
C(44)-C(47)	1.533(4)
C(44)-C(46)	1.534(4)
C(48)-C(49)	1.519(3)
C(48)-C(50)	1.527(3)
C(48)-C(51)	1.529(3)
C(8)-S(1)-C(1)	100.04(10)
C(4)-O(1)-C(18)	121.12(16)
C(2)-C(1)-C(5)	114.84(16)
C(2)-C(1)-S(1)	107.92(15)
C(5)-C(1)-S(1)	107.64(15)
C(3)-C(2)-C(1)	122.61(19)
C(2)-C(3)-C(4)	122.53(19)
O(2)-C(4)-O(1)	126.09(19)
O(2)-C(4)-C(3)	124.7(2)
O(1)-C(4)-C(3)	109.16(17)
C(6)-C(5)-C(1)	111.64(19)
C(5)-C(6)-C(7)	113.1(2)
C(6)-C(7)-Cl(1)	111.80(19)
C(9)-C(8)-C(13)	118.58(19)
C(9)-C(8)-S(1)	120.30(16)
C(13)-C(8)-S(1)	121.11(17)
C(8)-C(9)-C(10)	120.6(2)
C(11)-C(10)-C(9)	121.3(2)
C(12)-C(11)-C(10)	117.20(19)

C(12)-C(11)-C(14)	120.66(19)
C(10)-C(11)-C(14)	122.14(19)
C(11)-C(12)-C(13)	122.3(2)
C(12)-C(13)-C(8)	119.9(2)
C(16)-C(14)-C(17)	111.5(2)
C(16)-C(14)-C(11)	108.61(18)
C(17)-C(14)-C(11)	109.67(19)
C(16)-C(14)-C(15)	107.8(2)
C(17)-C(14)-C(15)	107.2(2)
C(11)-C(14)-C(15)	112.06(18)
O(1)-C(18)-C(21)	102.66(16)
O(1)-C(18)-C(20)	109.83(17)
C(21)-C(18)-C(20)	110.47(19)
O(1)-C(18)-C(19)	109.86(17)
C(21)-C(18)-C(19)	110.21(18)
C(20)-C(18)-C(19)	113.29(18)
C(38)-S(2)-C(31)	99.46(10)
C(34)-O(3)-C(48)	120.71(15)
C(32)-C(31)-C(35)	114.87(16)
C(32)-C(31)-S(2)	107.14(14)
C(35)-C(31)-S(2)	107.58(14)
C(33)-C(32)-C(31)	122.59(18)
C(32)-C(33)-C(34)	122.11(18)
O(4)-C(34)-O(3)	124.89(18)
O(4)-C(34)-C(33)	125.80(19)
O(3)-C(34)-C(33)	109.31(16)
C(31)-C(35)-C(36)	111.97(17)
C(37)-C(36)-C(35)	110.80(18)
C(36)-C(37)-Cl(2)	111.25(17)
C(39)-C(38)-C(43)	118.97(18)
C(39)-C(38)-S(2)	120.68(15)
C(43)-C(38)-S(2)	120.30(16)
C(38)-C(39)-C(40)	120.31(19)
C(41)-C(40)-C(39)	121.37(19)
C(40)-C(41)-C(42)	117.65(18)
C(40)-C(41)-C(44)	122.03(19)

C(42)-C(41)-C(44)	120.31(19)
C(43)-C(42)-C(41)	121.2(2)
C(42)-C(43)-C(38)	120.47(19)
C(45)-C(44)-C(47)	107.9(2)
C(45)-C(44)-C(46)	109.1(2)
C(47)-C(44)-C(46)	109.1(2)
C(45)-C(44)-C(41)	109.35(19)
C(47)-C(44)-C(41)	112.10(18)
C(46)-C(44)-C(41)	109.23(19)
O(3)-C(48)-C(49)	109.88(16)
O(3)-C(48)-C(50)	110.50(17)
C(49)-C(48)-C(50)	113.49(17)
O(3)-C(48)-C(51)	102.56(15)
C(49)-C(48)-C(51)	110.18(18)
C(50)-C(48)-C(51)	109.68(18)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for yf1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	24(1)	22(1)	17(1)	3(1)	-2(1)	0(1)
Cl(1)	43(1)	28(1)	30(1)	9(1)	-2(1)	-2(1)
O(1)	22(1)	27(1)	17(1)	-5(1)	-1(1)	2(1)
O(2)	28(1)	25(1)	25(1)	-6(1)	-2(1)	6(1)
C(1)	22(1)	18(1)	15(1)	0(1)	-1(1)	2(1)
C(2)	24(1)	17(1)	18(1)	0(1)	2(1)	2(1)
C(3)	23(1)	26(1)	20(1)	-6(1)	2(1)	3(1)
C(4)	18(1)	24(1)	18(1)	-1(1)	3(1)	0(1)
C(5)	28(1)	22(1)	16(1)	3(1)	-1(1)	-1(1)
C(6)	27(1)	31(1)	24(1)	5(1)	-1(1)	-5(1)
C(7)	33(1)	36(1)	28(1)	8(1)	8(1)	2(1)
C(8)	24(1)	17(1)	16(1)	1(1)	0(1)	-1(1)
C(9)	36(1)	29(1)	18(1)	2(1)	9(1)	12(1)
C(10)	32(1)	24(1)	24(1)	4(1)	8(1)	12(1)
C(11)	21(1)	21(1)	19(1)	5(1)	2(1)	0(1)
C(12)	34(1)	38(1)	18(1)	5(1)	10(1)	13(1)
C(13)	28(1)	34(1)	22(1)	6(1)	8(1)	15(1)
C(14)	20(1)	33(1)	19(1)	7(1)	1(1)	2(1)
C(15)	40(2)	35(1)	32(1)	6(1)	-2(1)	12(1)
C(16)	47(2)	43(1)	27(1)	-4(1)	-10(1)	9(1)
C(17)	28(1)	89(2)	50(2)	48(2)	5(1)	0(1)
C(18)	19(1)	27(1)	13(1)	-3(1)	2(1)	-5(1)
C(19)	27(1)	32(1)	21(1)	1(1)	9(1)	-3(1)
C(20)	31(1)	30(1)	26(1)	4(1)	1(1)	-10(1)
C(21)	27(1)	35(1)	23(1)	0(1)	-5(1)	-1(1)
S(2)	24(1)	25(1)	16(1)	-2(1)	8(1)	-4(1)
Cl(2)	34(1)	29(1)	43(1)	-5(1)	-6(1)	-5(1)
O(3)	22(1)	22(1)	15(1)	3(1)	9(1)	3(1)
O(4)	26(1)	23(1)	20(1)	2(1)	9(1)	6(1)
C(31)	21(1)	18(1)	15(1)	0(1)	5(1)	-3(1)
C(32)	21(1)	16(1)	18(1)	2(1)	4(1)	-1(1)

C(33)	20(1)	18(1)	17(1)	4(1)	6(1)	0(1)
C(34)	20(1)	19(1)	14(1)	1(1)	2(1)	-3(1)
C(35)	28(1)	20(1)	14(1)	0(1)	5(1)	-2(1)
C(36)	25(1)	24(1)	17(1)	2(1)	6(1)	-4(1)
C(37)	32(1)	26(1)	23(1)	-2(1)	4(1)	-4(1)
C(38)	21(1)	17(1)	18(1)	-2(1)	6(1)	-3(1)
C(39)	20(1)	23(1)	20(1)	0(1)	5(1)	3(1)
C(40)	20(1)	27(1)	17(1)	1(1)	2(1)	2(1)
C(41)	20(1)	19(1)	21(1)	-4(1)	4(1)	-3(1)
C(42)	30(1)	19(1)	29(1)	-4(1)	3(1)	6(1)
C(43)	30(1)	23(1)	18(1)	0(1)	-2(1)	2(1)
C(44)	27(1)	29(1)	22(1)	-7(1)	8(1)	2(1)
C(45)	35(1)	29(1)	33(1)	-11(1)	8(1)	-3(1)
C(46)	30(1)	53(2)	50(2)	-25(1)	18(1)	-10(1)
C(47)	48(2)	43(1)	22(1)	-3(1)	9(1)	6(1)
C(48)	21(1)	22(1)	16(1)	4(1)	8(1)	0(1)
C(49)	23(1)	27(1)	21(1)	4(1)	4(1)	-7(1)
C(50)	26(1)	28(1)	19(1)	1(1)	3(1)	-1(1)
C(51)	30(1)	30(1)	23(1)	3(1)	15(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for yf1.

	x	y	z	U(eq)
H(1)	1768	9404	5886	22
H(2)	3410	8377	5088	24
H(3)	-596	9090	4751	28
H(5A)	5374	8578	6579	27
H(5B)	4334	9128	7024	27
H(6A)	2105	8001	6522	33
H(6B)	575	8578	6691	33
H(7A)	1230	7954	7805	38
H(7B)	2509	8599	8006	38
H(9)	1853	10494	5356	33
H(10)	-67	11027	4278	32
H(12)	3966	10204	2929	35
H(13)	5888	9667	3993	33
H(15A)	-2070	11685	2494	55
H(15B)	-505	11777	3330	55
H(15C)	-2505	11268	3211	55
H(16A)	-1790	10725	1761	62
H(16B)	-2035	10309	2500	62
H(16C)	137	10220	2062	62
H(17A)	3092	11045	2113	84
H(17B)	3165	11593	2727	84
H(17C)	1313	11606	1941	84
H(19A)	-104	8766	2207	39
H(19B)	-1505	8252	1665	39
H(19C)	307	8055	2416	39
H(20A)	-2642	7482	3056	44
H(20B)	-4506	7594	2287	44
H(20C)	-5060	7805	3115	44
H(21A)	-5736	8894	2707	44
H(21B)	-5439	8683	1850	44

H(21C)	-3905	9235	2267	44
H(31)	12991	5733	3306	21
H(32)	10930	6848	2635	22
H(33)	13640	5968	2044	22
H(35A)	11831	6023	4492	24
H(35B)	10557	6620	4092	24
H(36A)	15477	6442	4238	26
H(36B)	14124	7059	3938	26
H(37A)	14550	6588	5486	32
H(37B)	13050	7183	5193	32
H(39)	7127	5728	1491	25
H(40)	7722	5199	375	26
H(42)	12197	4058	1699	31
H(43)	11545	4571	2813	30
H(45A)	10612	3362	686	48
H(45B)	9897	3342	-240	48
H(45C)	8046	3552	288	48
H(46A)	13583	4665	113	65
H(46B)	13355	4012	-315	65
H(46C)	13932	4046	614	65
H(47A)	7465	4496	-546	56
H(47B)	9443	4269	-1013	56
H(47C)	9621	4933	-614	56
H(49A)	14483	7719	612	36
H(49B)	15740	7660	-132	36
H(49C)	16929	7377	684	36
H(50A)	11116	6523	-481	37
H(50B)	12103	7097	-896	37
H(50C)	10888	7201	-149	37
H(51A)	17216	6354	104	40
H(51B)	16064	6604	-734	40
H(51C)	14960	6026	-364	40

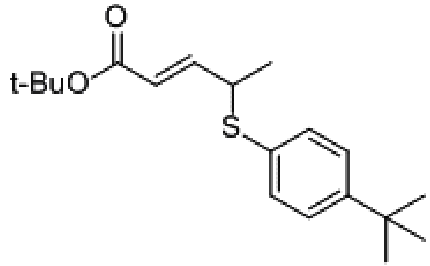
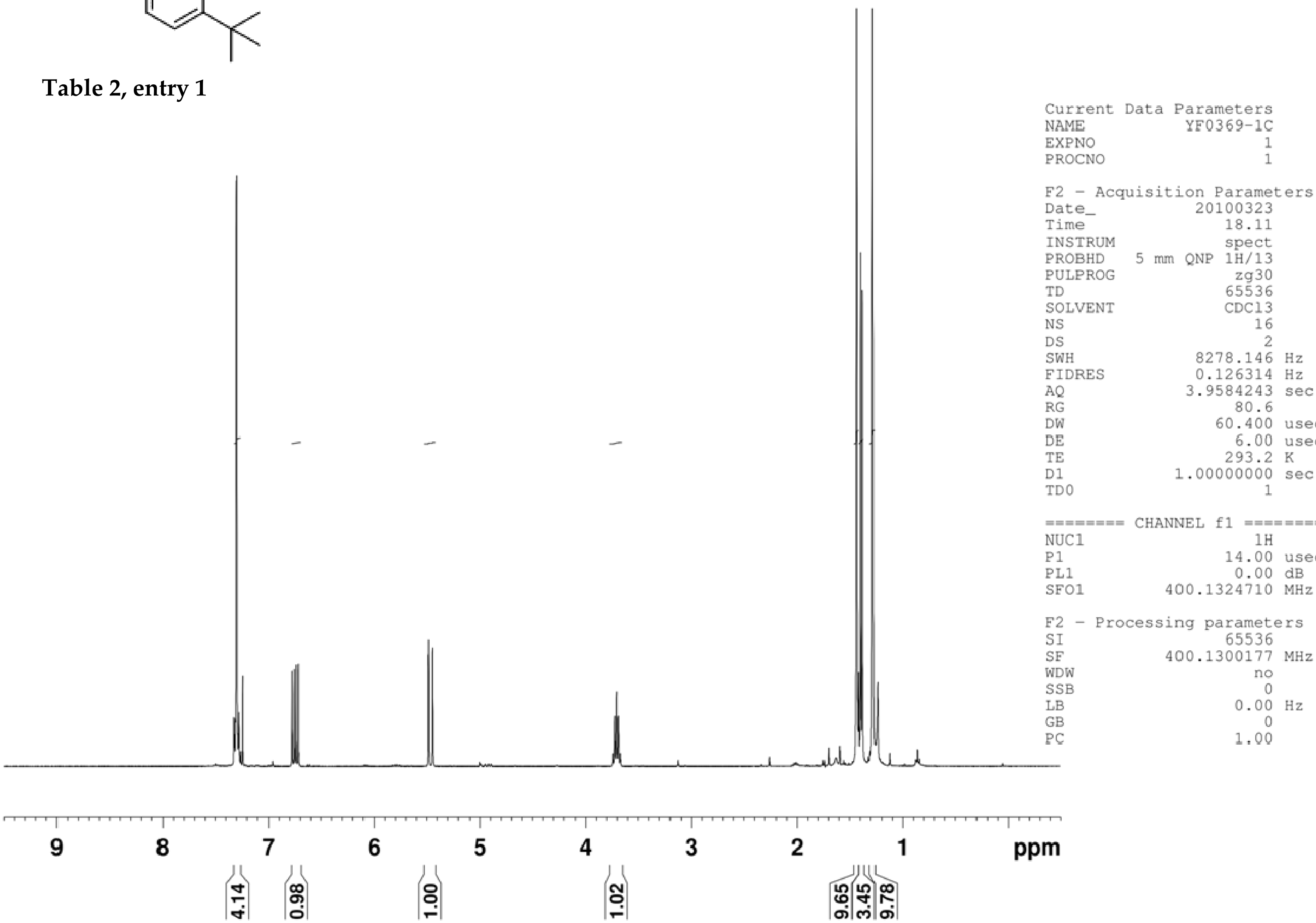


Table 2, entry 1



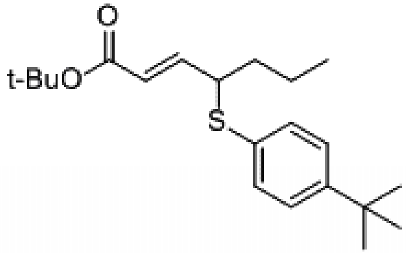
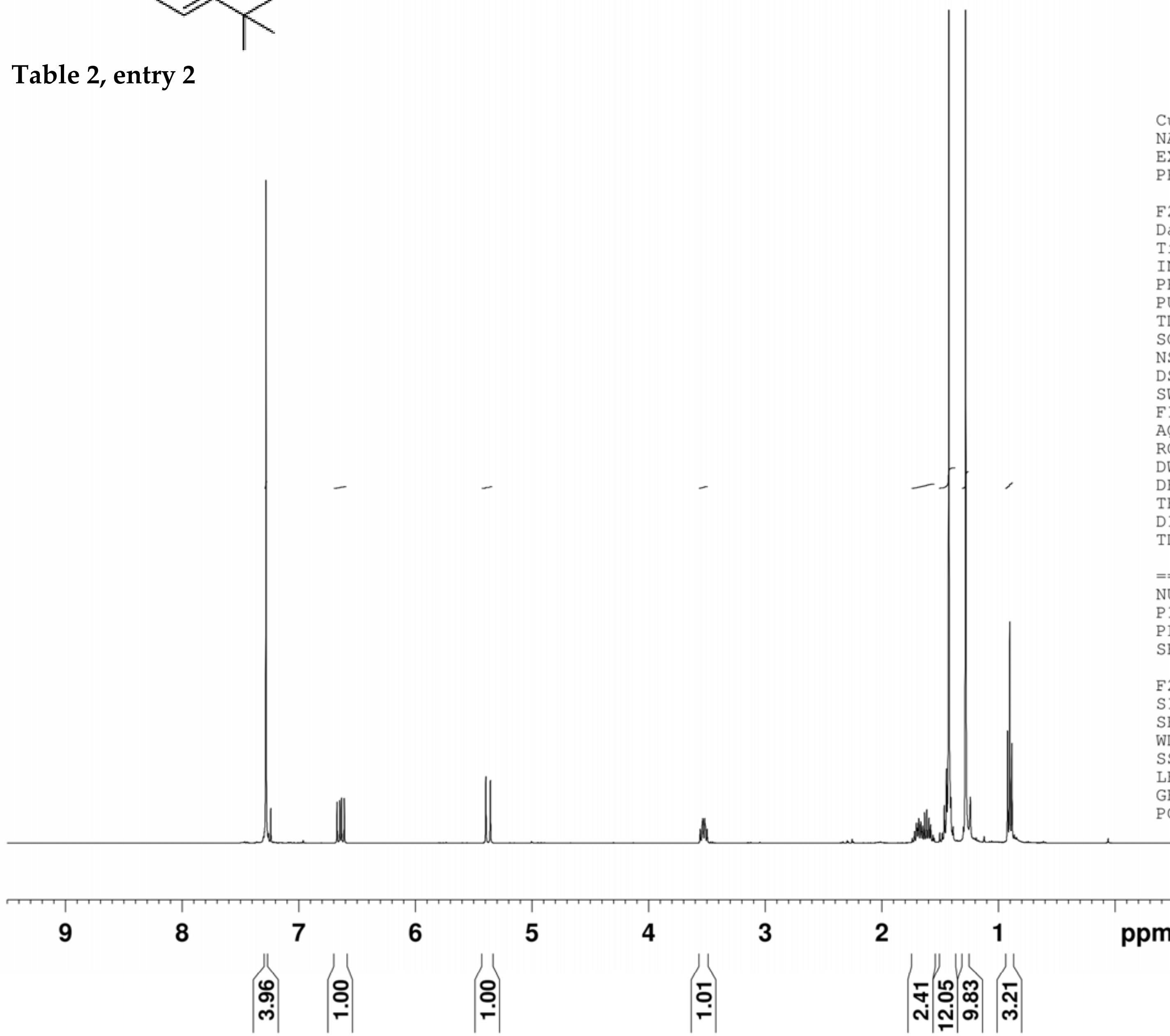


Table 2, entry 2




```
Current Data Parameters
NAME            YF0350-1C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100318
Time                15.45
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                     57
DW                 60.400 usec
DE                   6.00 usec
TE                  294.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300177 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

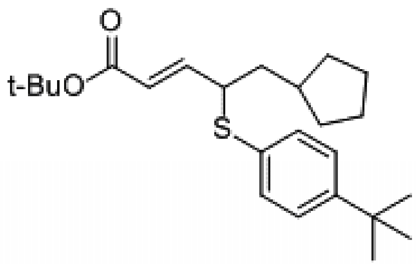
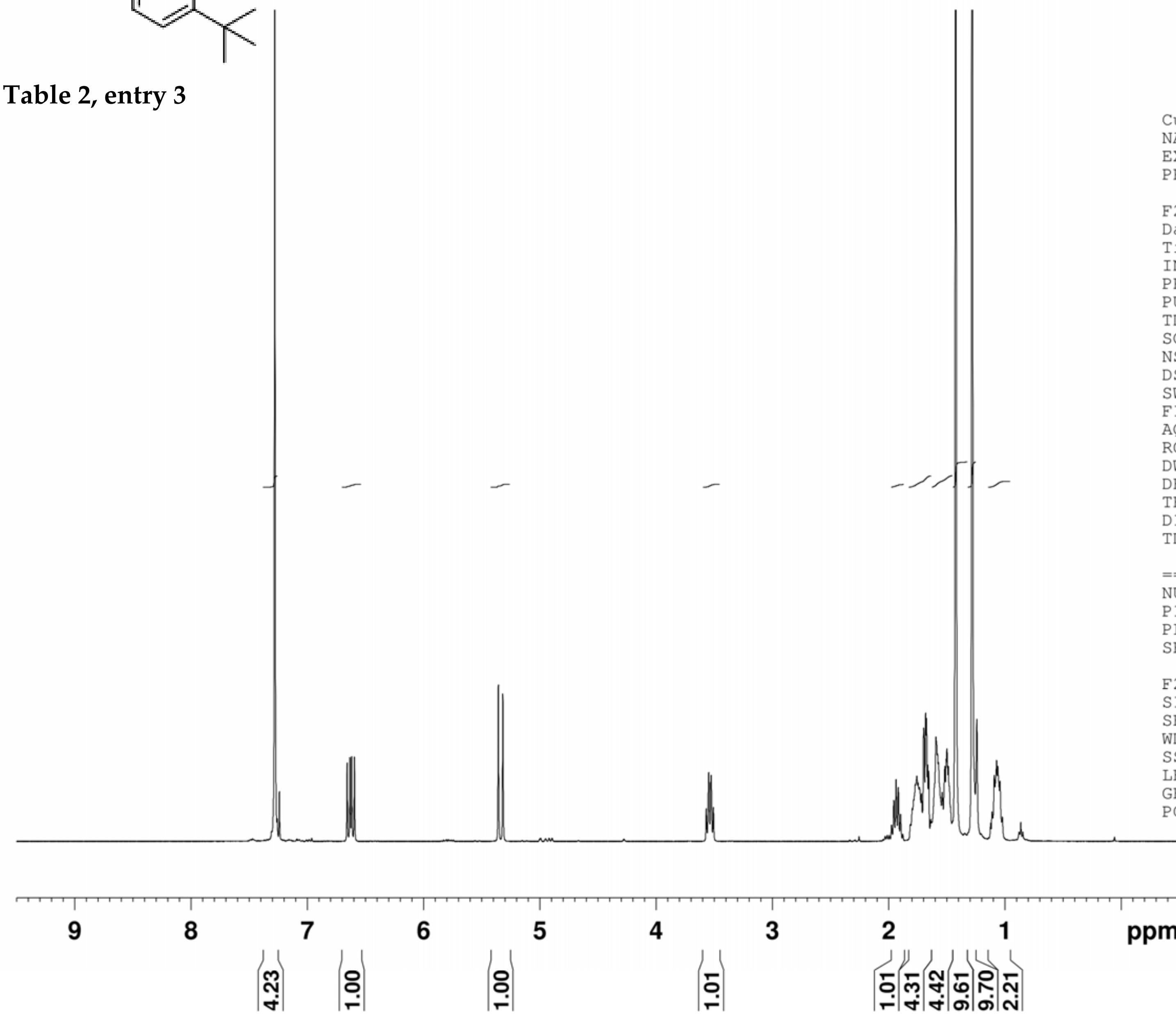


Table 2, entry 3




```
Current Data Parameters
NAME            YF0372-1B
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100325
Time                12.34
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                   40.3
DW                 60.400 usec
DE                   6.00 usec
TE                  295.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.07 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300170 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

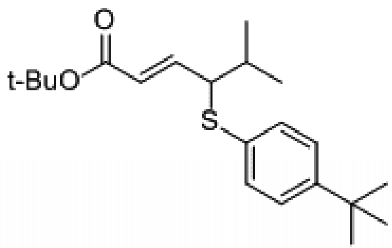
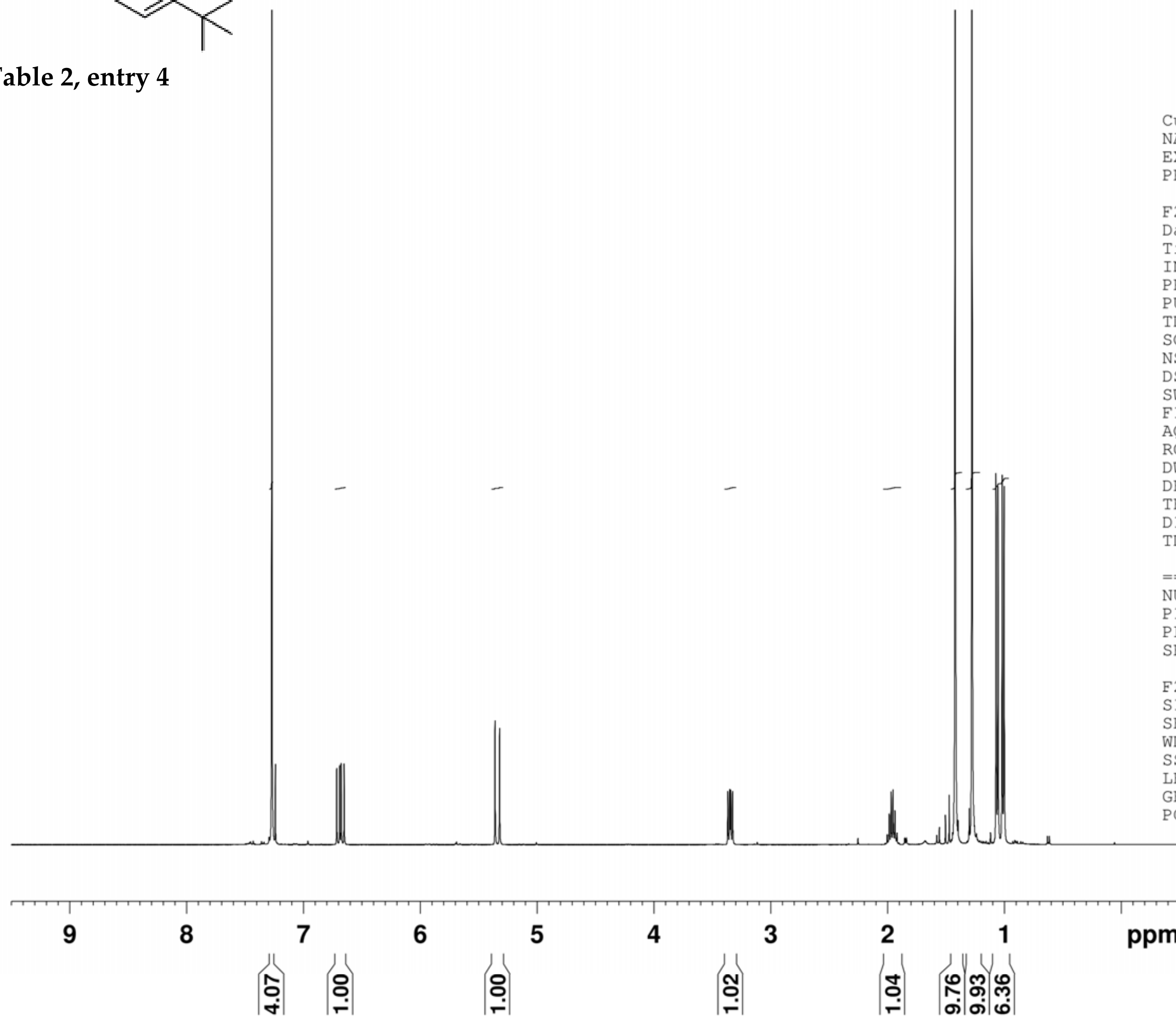


Table 2, entry 4




```
Current Data Parameters
NAME           YF0371-1C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100323
Time               12.50
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                    57
DW                60.400 usec
DE                  6.00 usec
TE                 293.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300176 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

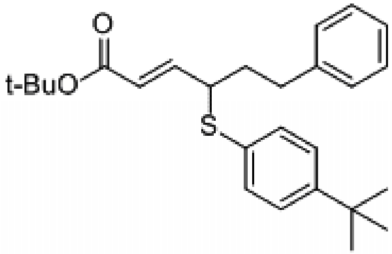
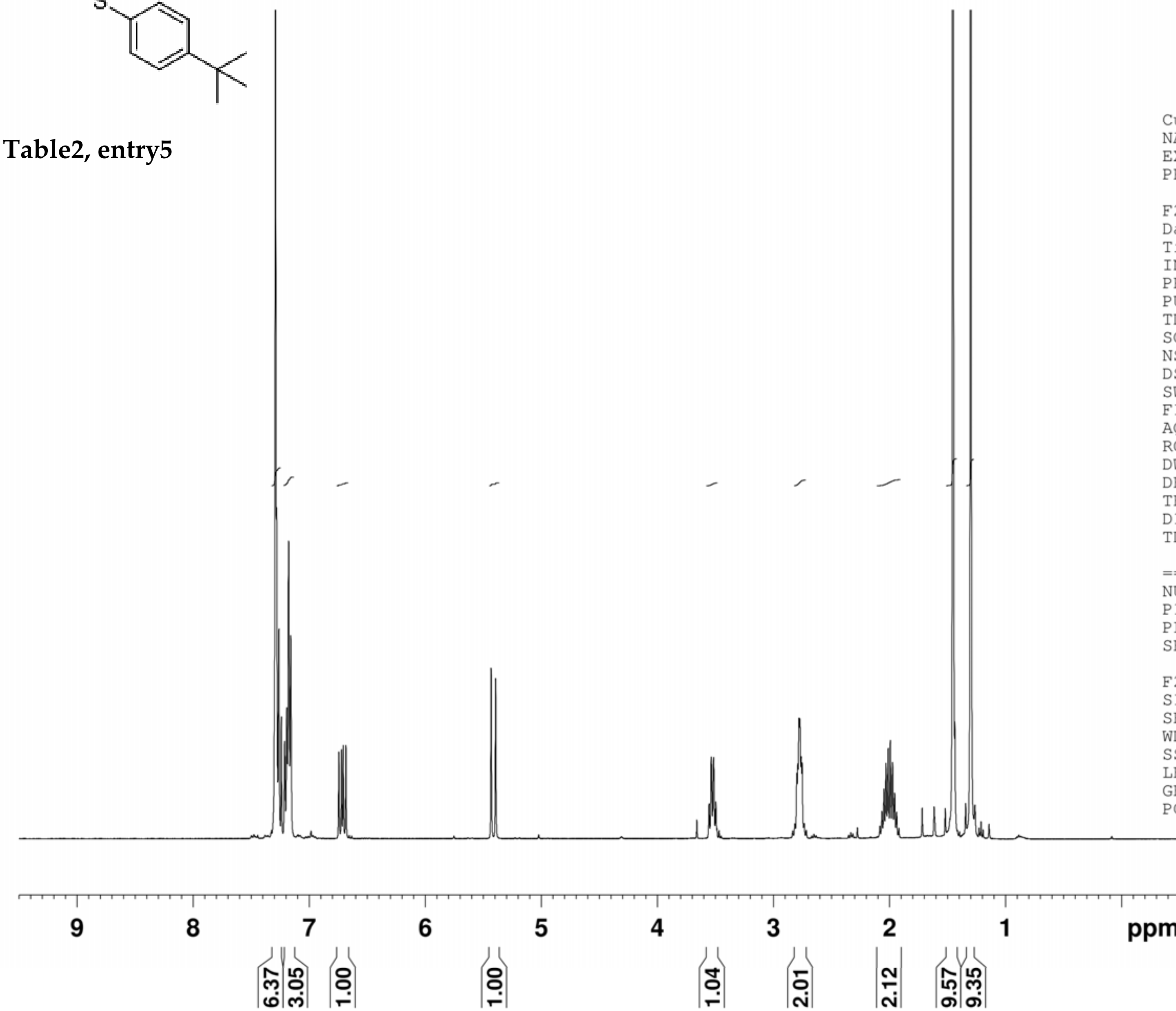


Table2, entry5




```
Current Data Parameters
NAME             YF0376-1B
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100325
Time                 12.24
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT
NS                      16
DS                       2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ               3.9584243 sec
RG                    40.3
DW                  60.400 usec
DE                    6.00 usec
TE                   295.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   15.07 usec
PL1                   0.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300172 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

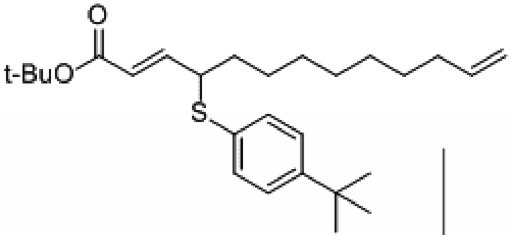
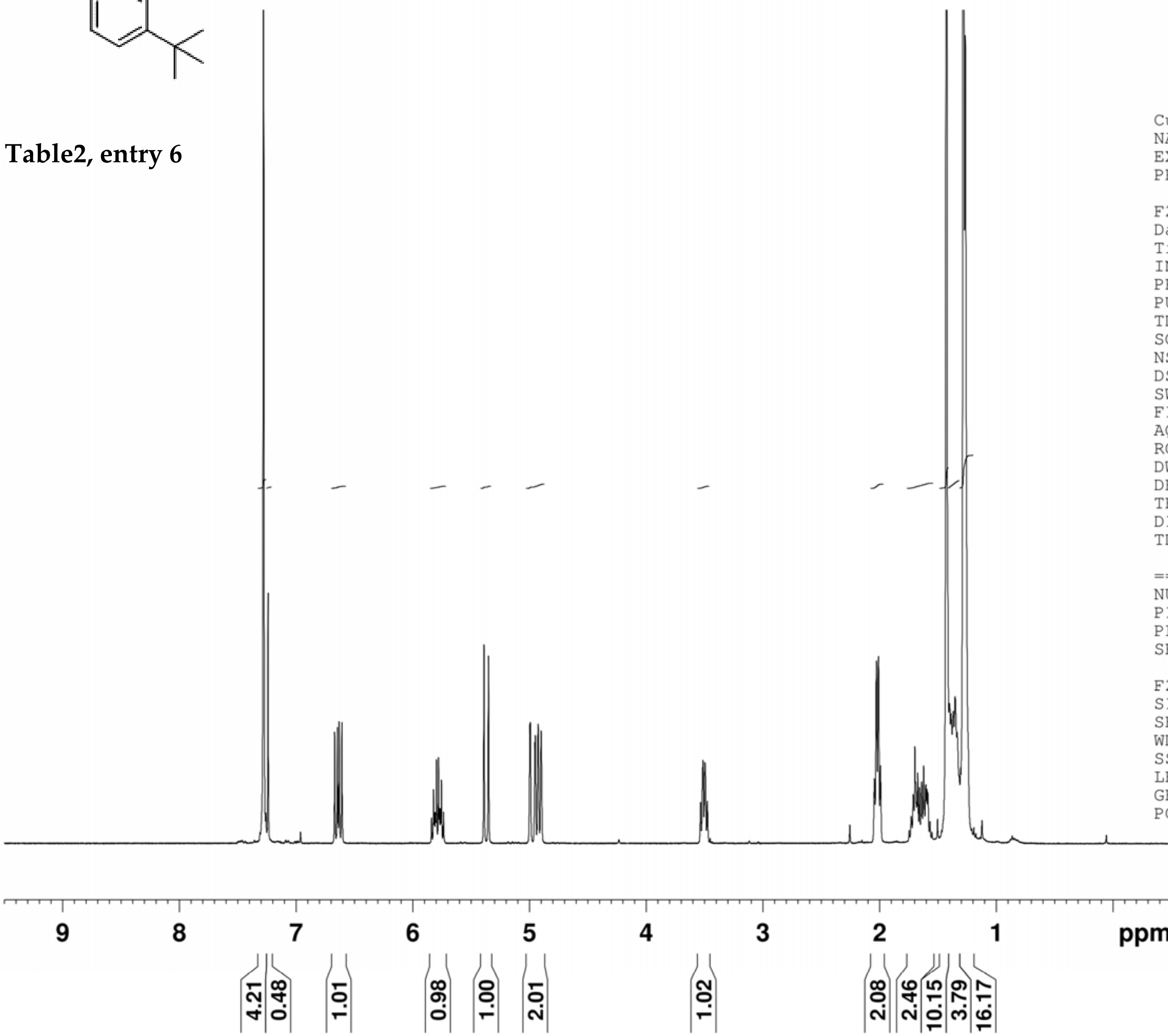


Table2, entry 6




```
Current Data Parameters
NAME            YF0375-1C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100326
Time                17.31
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                   50.8
DW                 60.400 usec
DE                   6.00 usec
TE                  296.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.07 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300172 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

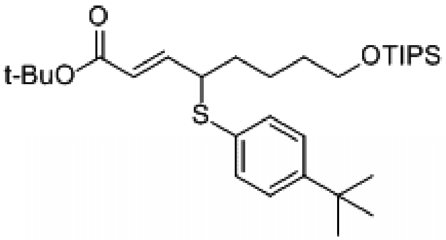
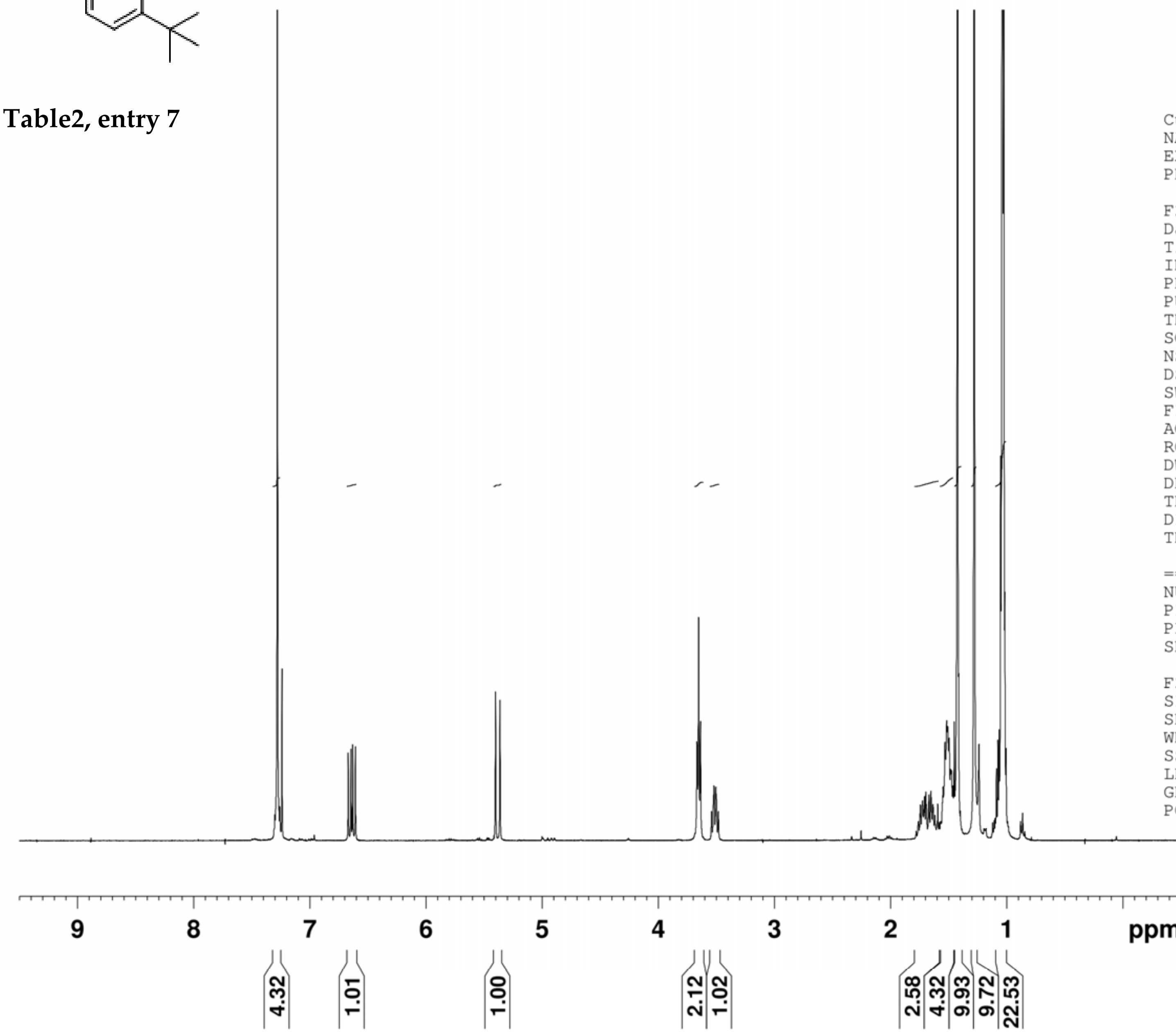


Table2, entry 7




```
Current Data Parameters
NAME            YF0378-1B
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100325
Time                18.52
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                     57
DW                 60.400 usec
DE                   6.00 usec
TE                  293.2 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300178 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

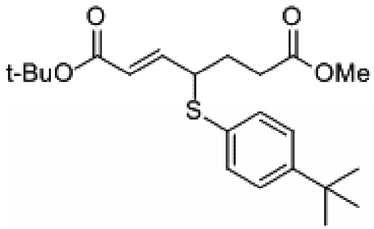
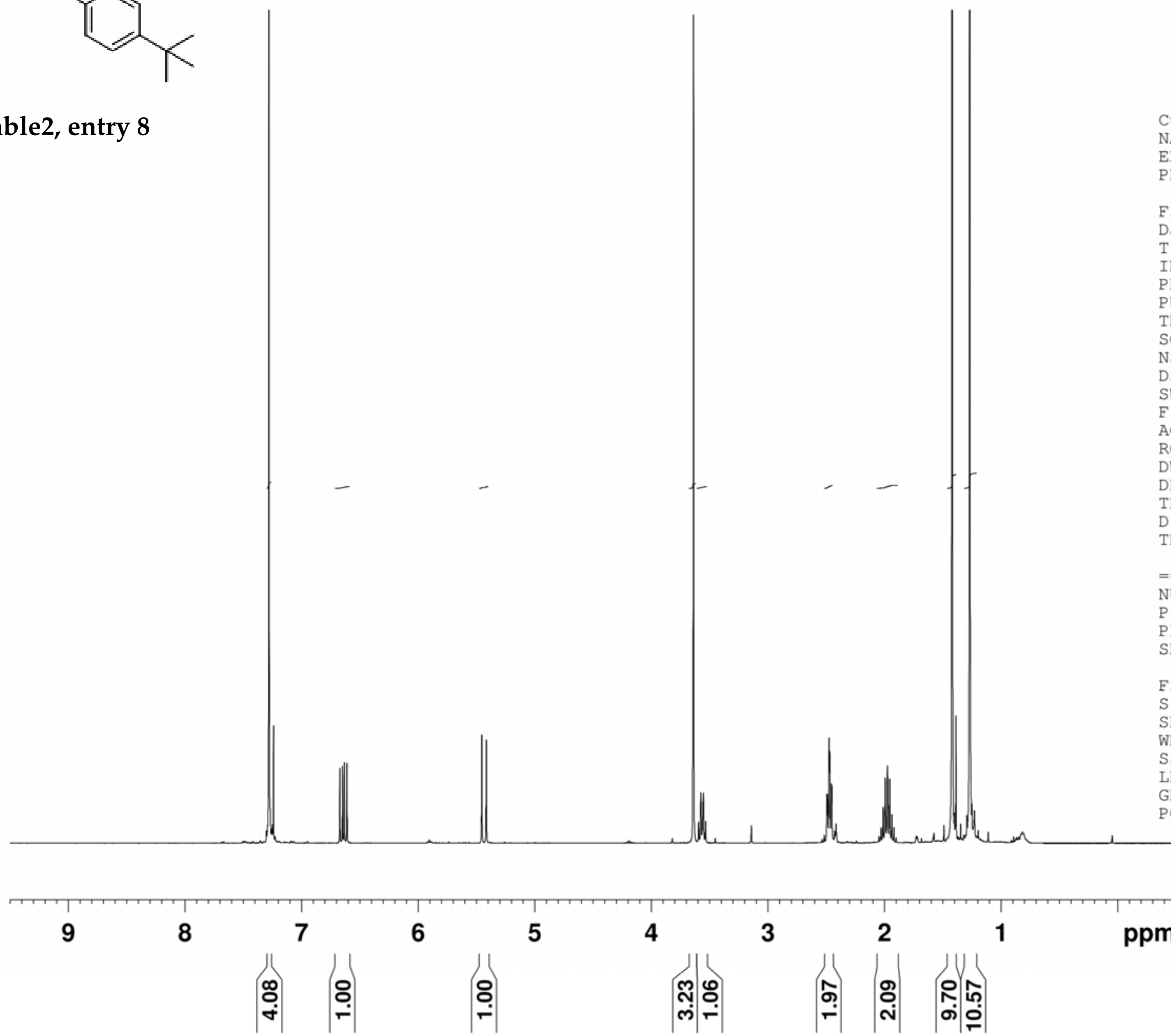


Table2, entry 8




```
Current Data Parameters
NAME          YF0407-2B
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20100420
Time              13.45
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ            3.9584243 sec
RG                 35.9
DW               60.400 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.07 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                65536
SF          400.1300167 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

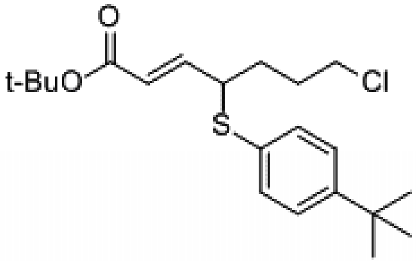
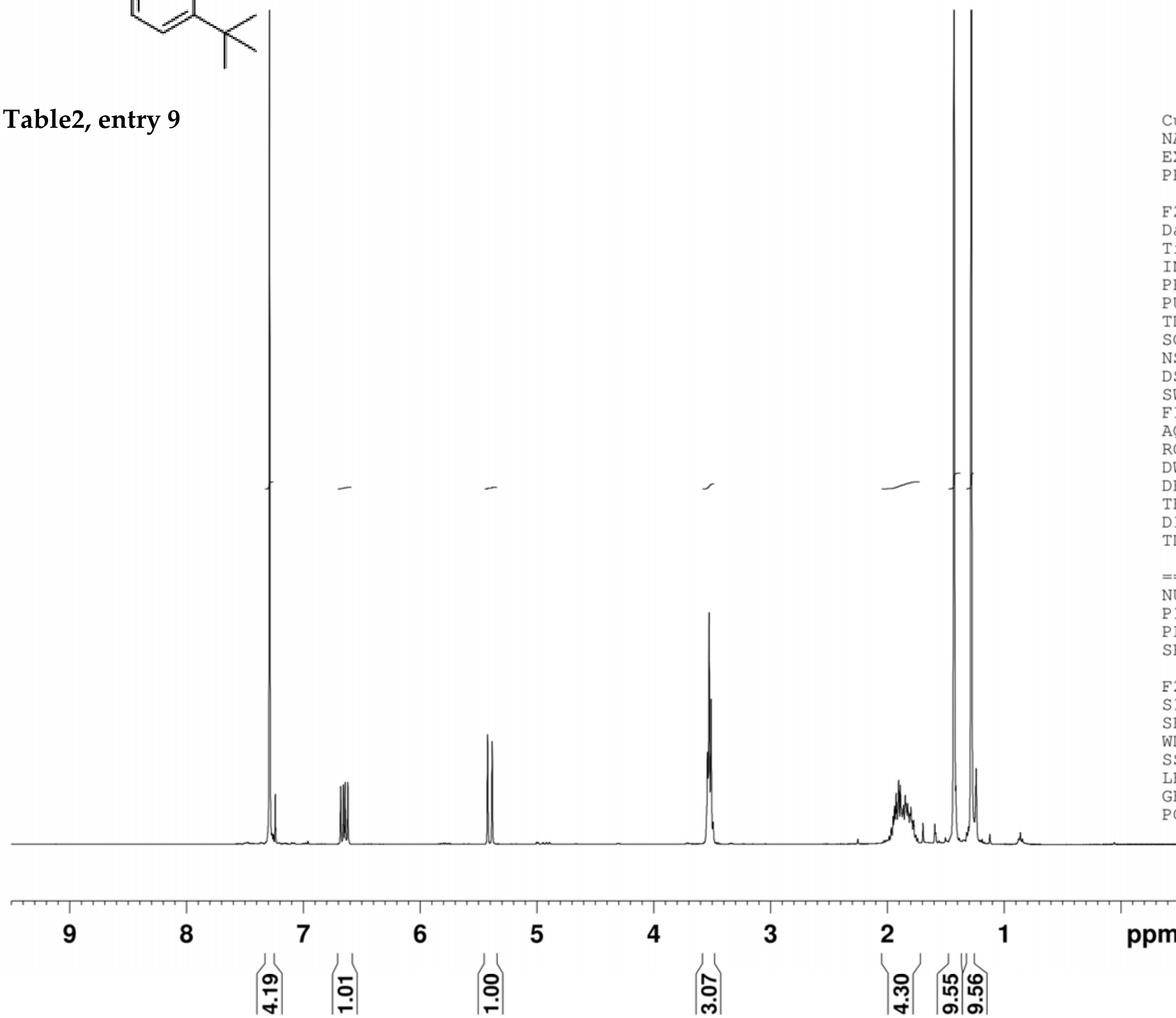


Table2, entry 9




```
Current Data Parameters
NAME            YF0374-1C
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100325
Time                12.43
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                   35.9
DW                 60.400 usec
DE                   6.00 usec
TE                  295.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.07 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300170 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

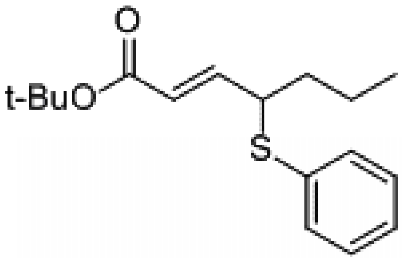
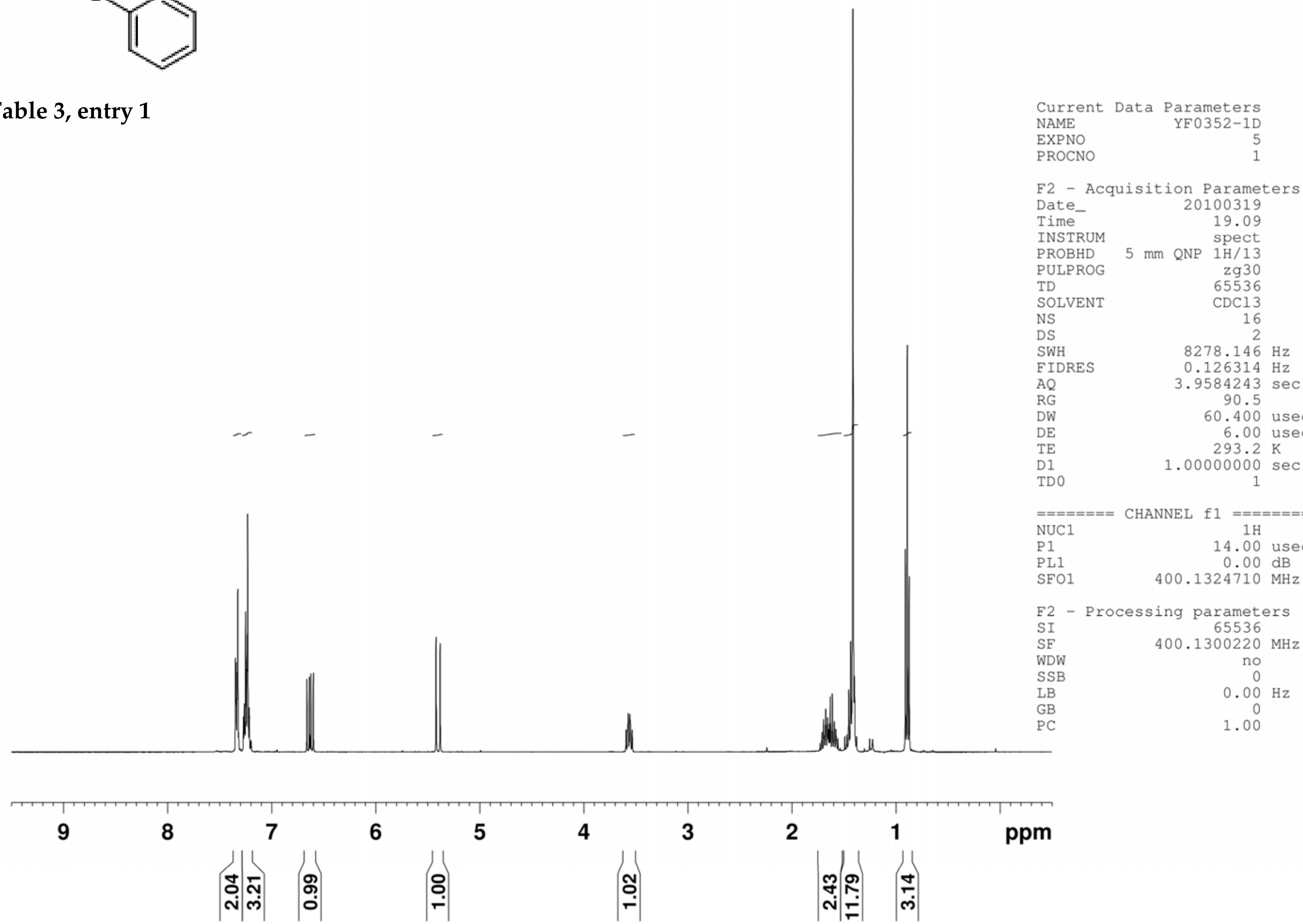


Table 3, entry 1



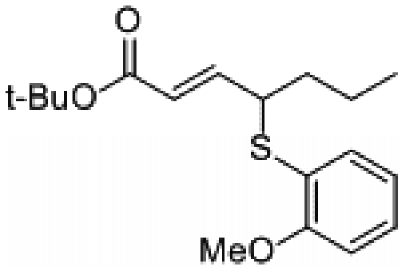
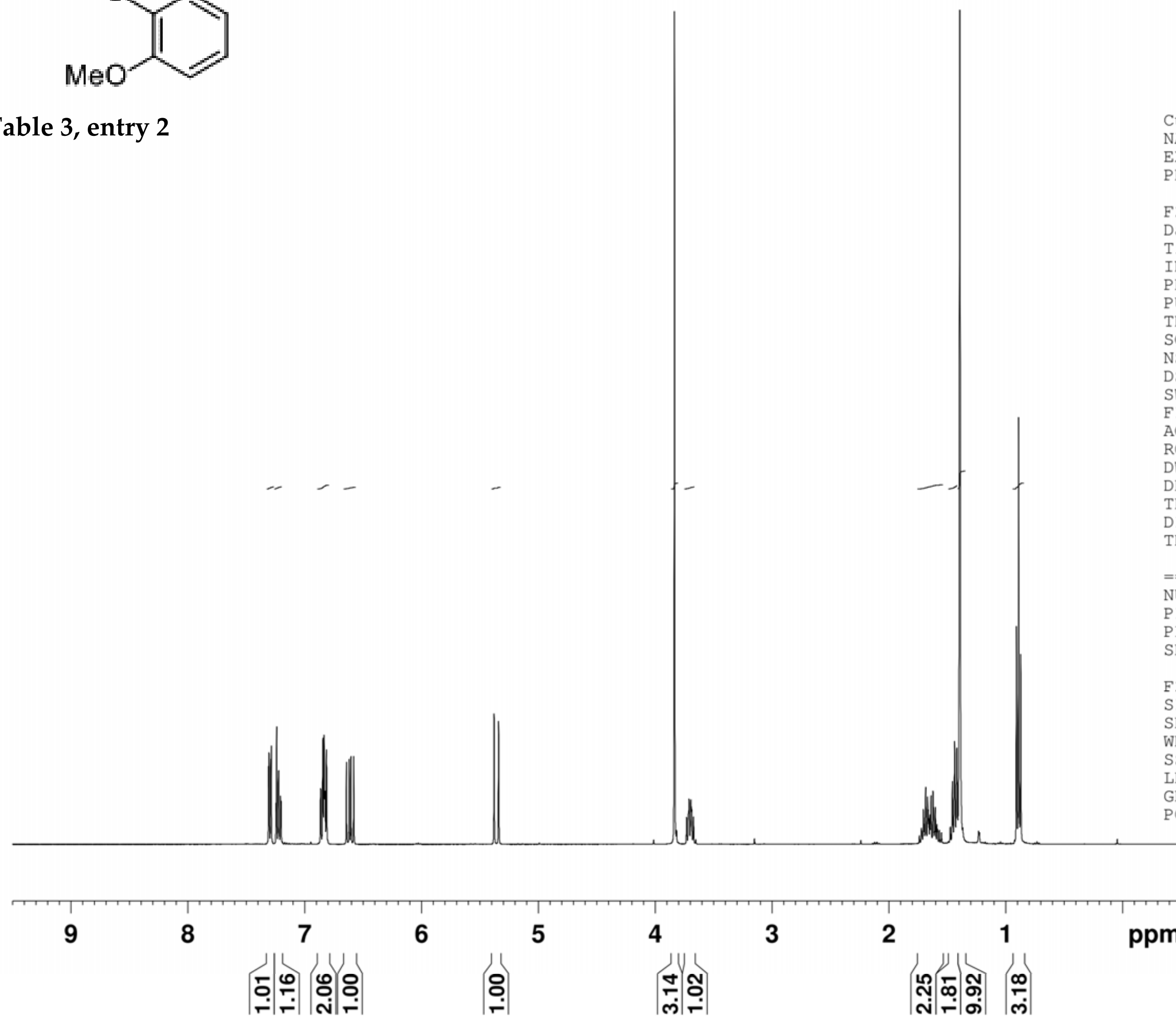


Table 3, entry 2




```
Current Data Parameters
NAME                 YF0357
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100315
Time                  18.50
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                3.9584243 sec
RG                       57
DW                   60.400 usec
DE                     6.00 usec
TE                    294.2 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.00 usec
PL1                    0.00 dB
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF              400.1300177 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```

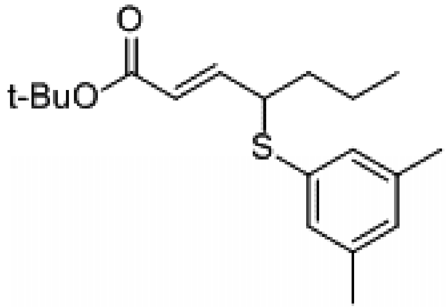
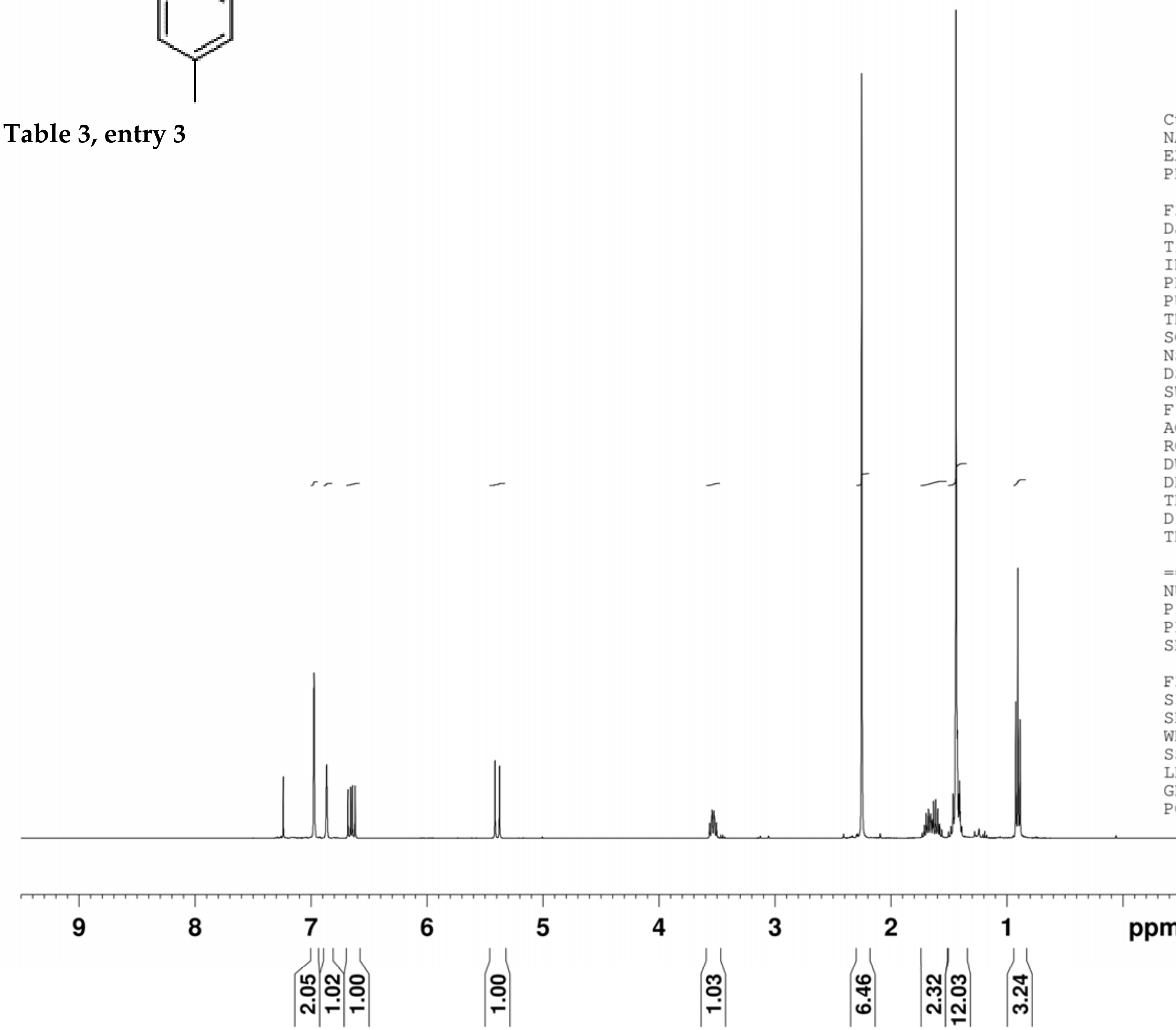


Table 3, entry 3




```
Current Data Parameters
NAME            YF0351-2B
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100318
Time                18.05
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                     57
DW                 60.400 usec
DE                   6.00 usec
TE                  294.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300178 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

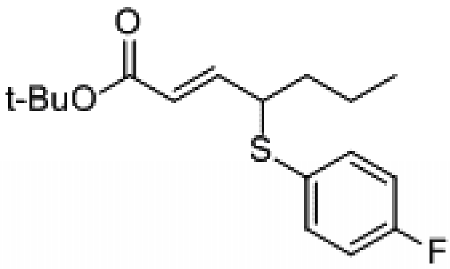
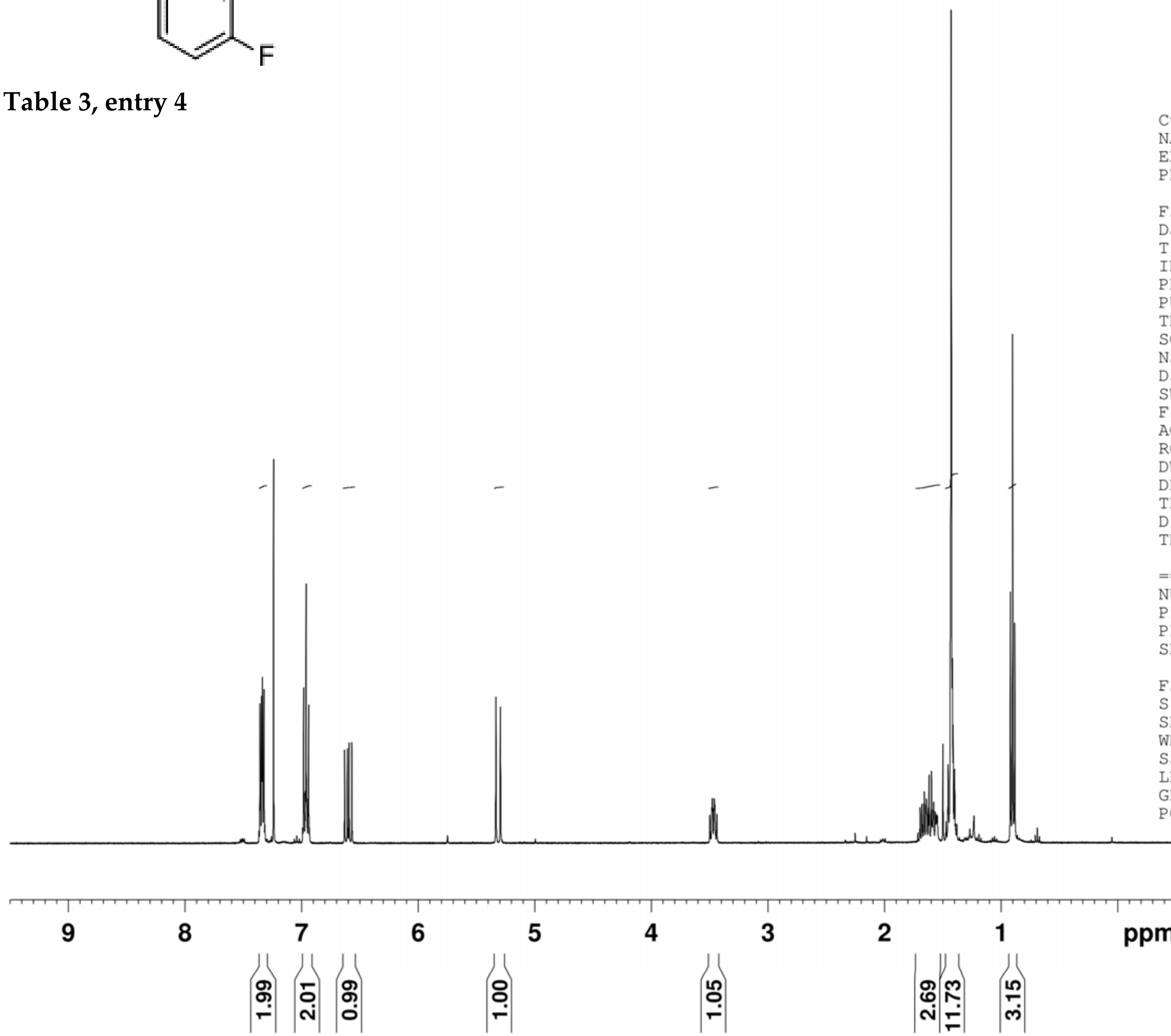


Table 3, entry 4




```
Current Data Parameters
NAME            YF0355-2B
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100317
Time                21.20
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ              3.9584243 sec
RG                    256
DW                 60.400 usec
DE                   6.00 usec
TE                  294.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300177 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

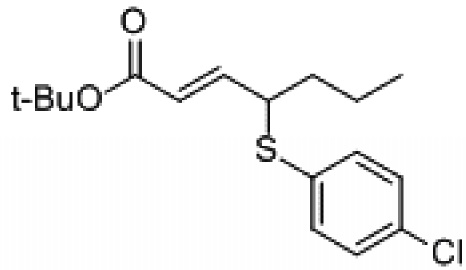
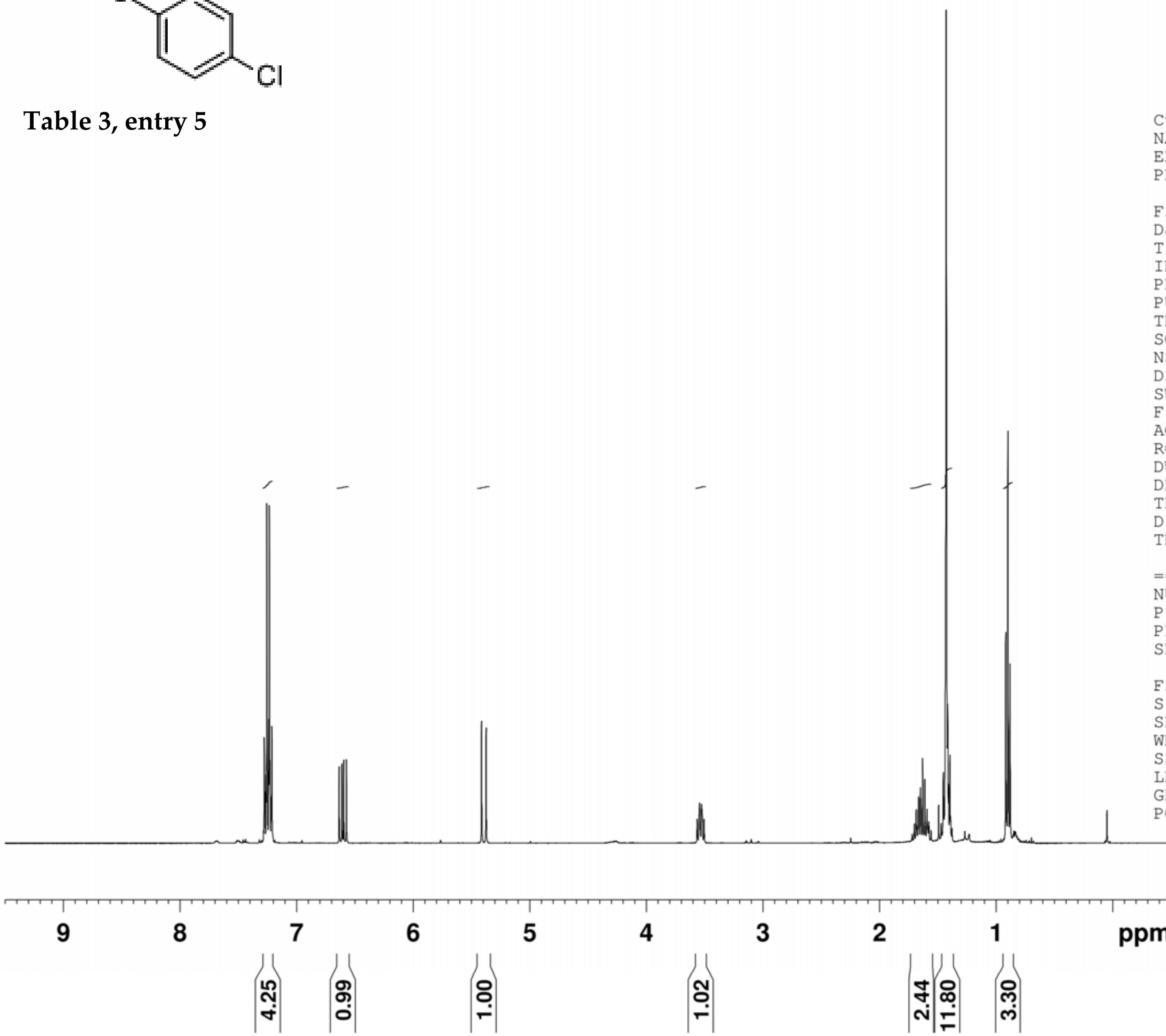


Table 3, entry 5




```
Current Data Parameters
NAME            YF0358-1B
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100316
Time                19.40
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ            3.9584243 sec
RG                  101.6
DW                 60.400 usec
DE                   6.00 usec
TE                  294.2 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300178 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

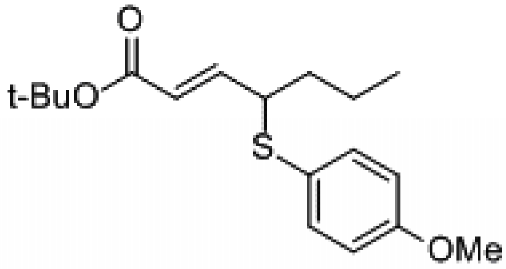
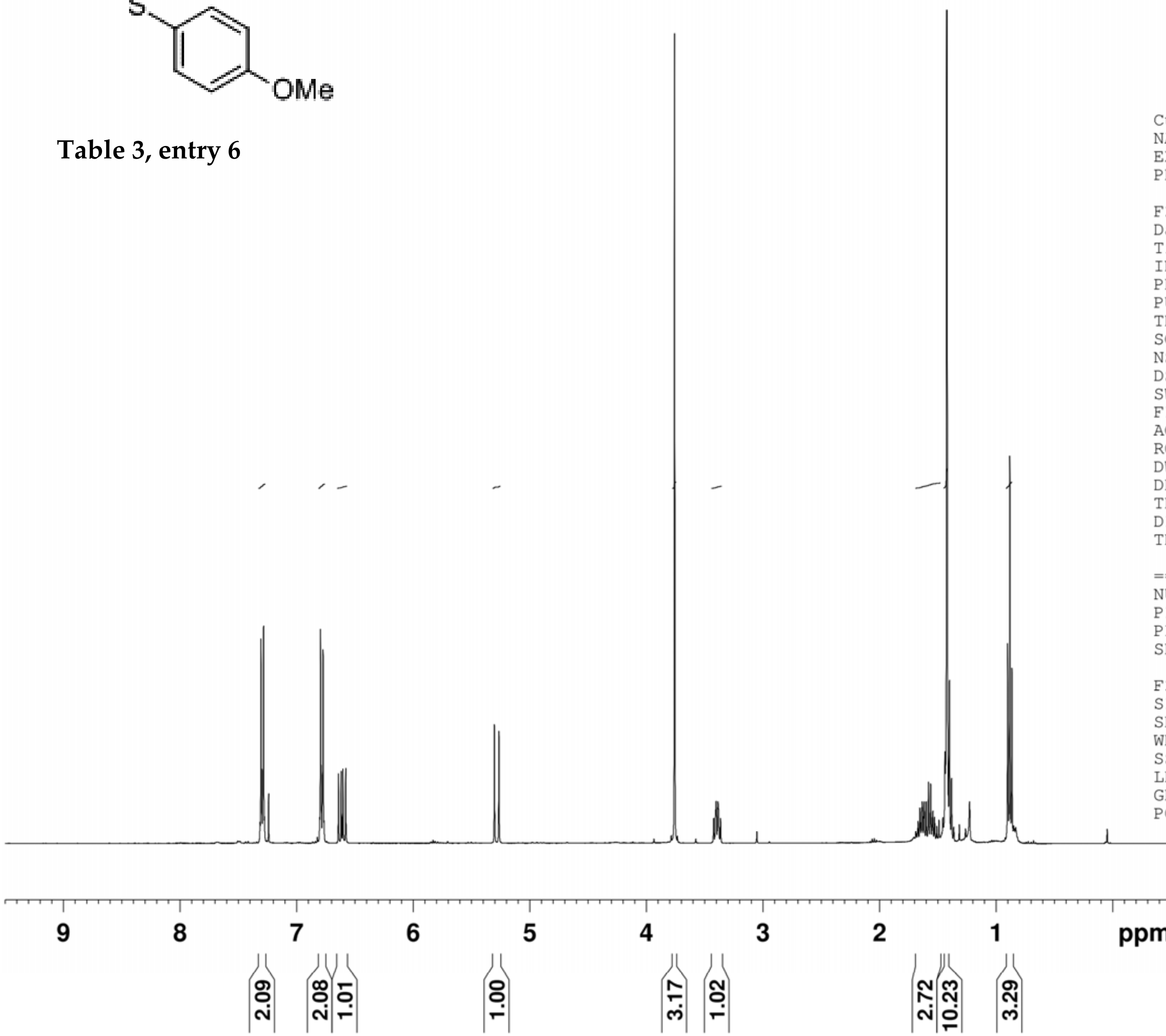


Table 3, entry 6




```
Current Data Parameters
NAME             YF0356
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20100315
Time              18.39
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ            3.9584243 sec
RG                   57
DW               60.400 usec
DE                 6.00 usec
TE                294.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                65536
SF          400.1300176 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

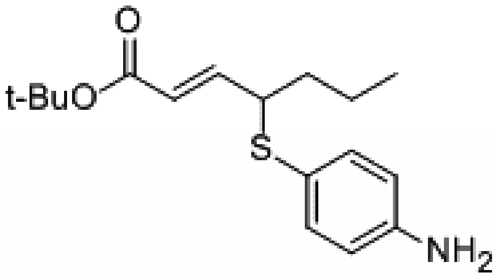


Table 3, entry 7