

SUPPORTING INFORMATION

Borostannylation of Alkynes and Enynes. Scope and Limitations of the Reaction and Utility of the Adducts

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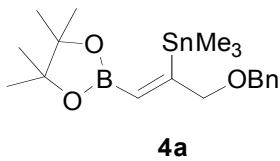
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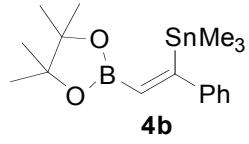
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General procedure for the Pd-catalyzed, borostannylation of alkynes and enynes with [B-Sn]-reagent 1, followed by pinacolate formation:

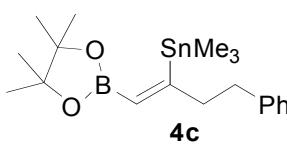
To a solution of 1,3-dimethyl-2-(trimethylstannyl)-2-bora-1,3-diazocyclopentane (156 mg, 0.6 mmol) in benzene (4 mL) was added $\text{PdCl}_2(\text{PPh}_3)_2$ (3.5 mg, 0.005 mmol) and the mixture was stirred at room temperature for 15 min followed by addition of a solution of alkyne or enyne (0.5 mmol) in benzene (1 mL). After stirring the reaction mixture at room temperature for 6 h, a solution of pinacol (84 mg, 0.72 mmol) in benzene (0.5 mL) followed by *p*-toluenesulfonic acid (*p*-TSA, 136 mg, 0.72 mmol) were added. After 2 h, the reaction was quenched by the addition of Et_3N (0.12 mL, 0.86 mmol) and the solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel chromatography using 2-5% EtOAc/hexane as the mobile phase to yield the desired product.



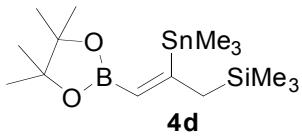
Borostannylation of propargyl alcohol benzyl ether: synthesis of 4a: Compound **4a** was synthesized from propargyl alcohol benzyl ether **2a** in 83 % isolated yield using the general procedure described above. ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.25 (m, 5 H), 6.42 (d, $J = 2.0$ Hz, $J_{\text{Sn}-\text{H}} = 71.6$ Hz, 1 H), 4.47 (s, 2 H), 4.21 (d, $J = 1.6$ Hz, $J_{\text{Sn}-\text{H}} = 13.6$ Hz, 2 H), 1.24 (s, 12 H), 0.17 (s, 9 H, $J_{\text{Sn}-\text{H}} = 26.8$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 171.89, 138.74, 128.48, 127.78, 127.59, 83.57, 79.58, 77.43, 72.33, 25.02, -7.42; IR (neat, cm^{-1}): 2977.0, 2924.1, 2858.1, 2339.8, 1683.9, 1540.0, 1495.6, 1351.8, 1096.1, 967.5; MS (ESI): m/z: 461.11 $[\text{M}+\text{Na}]^+$.



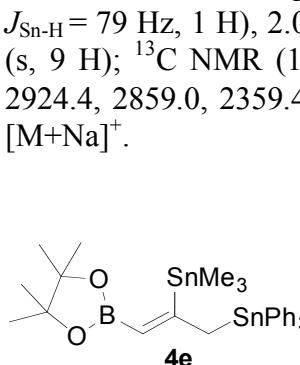
Borostannylation of phenylacetylene: synthesis of the compound 4b: Compound **4b** was synthesized from phenylacetylene in 82 % isolated yield using the general procedure described above. ^1H NMR (400 MHz, CDCl_3) δ 7.27 (d, $J = 6.8$ Hz, 2 H), 7.17 (t, $J = 6.0$ Hz, 1 H), 7.02 (d, $J = 6.8$ Hz, 2 H), 6.26 (s, $J_{\text{Sn}-\text{H}} = 72.0$ Hz, 1 H), 1.28 (s, 12 H), 0.16 (s, $J_{\text{Sn}-\text{H}} = 26.8$ Hz, 9 H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.23, 149.36, 128.10, 126.35, 83.69, 25.08, -5.62; IR (neat, cm^{-1}): 2977.7, 2924.9, 2539.4, 1683.8, 1558.2, 1487.1, 1338.7, 1143.0, 968.9, 846.4; MS (ESI): m/z: 417.08 $[\text{M}+\text{Na}]^+$.



Borostannylation of 4-phenyl-1-butyne: synthesis of the compound 4c: Compound **4c** was synthesized from 4-phenyl-1-butyne in 81 % isolated yield using the general procedure described above. ^1H NMR (400 MHz, CDCl_3) δ 7.26 (d, $J = 7.6$ Hz, 2H), 7.17-7.148 (m, 3H), 6.15 (s, $J_{\text{Sn}-\text{H}} = 82.8$ Hz, 1H), 2.63 (s, 4H), 1.24 (s, 12H), 0.18 (s, $J_{\text{Sn}-\text{H}} = 26.4$ Hz, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.19, 142.22, 128.67, 128.53, 126.02, 83.47, 47.04, 36.37, 25.04, -5.84; IR (neat, cm^{-1}): 2977.3, 2923.6, 2856.2, 2359.4, 1652.2, 1495.4, 1455.8, 1535.2, 1234.7, 1146.2; MS (ESI): m/z: 445.12 $[\text{M}+\text{Na}]^+$.

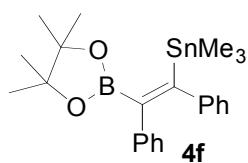


Borostannylation of trimethyl(propargyl)silane: synthesis of the compound 4d: Compound **4d** was synthesized from trimethyl(propargyl)silane in 80 % isolated yield using the general procedure described above. ^1H NMR (500 MHz, CDCl_3) δ 5.87 (s, $J_{\text{Sn}-\text{H}} = 79$ Hz, 1 H), 2.05 (s, $J_{\text{Sn}-\text{H}} = 49.5$ Hz, 2 H), 1.23 (12 H), 0.14 (s, 9H, $J_{\text{Sn}-\text{H}} = 27$ Hz), -0.24 (s, 9 H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.36, 83.16, 53.62, -1.23, -6.82; IR (neat, cm^{-1}): 2924.4, 2859.0, 2359.4, 1652.2, 1558.1, 1349.9, 1133.9, 1026.1, 824.5; MS (ESI): m/z: 427.03 $[\text{M}+\text{Na}]^+$.



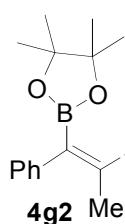
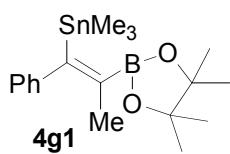
Borostannylation of triphenyl(2-propynyl)stannane: synthesis of the compound 4e: Compound **4e** was synthesized from triphenyl(2-propynyl)stannane in 80 % isolated yield using the general procedure described above.

^1H NMR (400 MHz, CDCl_3) δ 7.60-7.40 (m, 6 H), 7.362-7.326 (m, 9 H), 6.07 (s, $J_{\text{Sn}-\text{H}} = 84$, 20 Hz, 1 H), 2.99 (d, $J = 0.8$ Hz, $J_{\text{Sn}-\text{H}} = 44$, 36 Hz, 2 H), 1.19 (12 H), -0.11 (s, $J_{\text{Sn}-\text{H}} = 27$ Hz, 9 H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.60, 137.50, 129.18, 128.43, 83.13, 24.90,-0.6; IR (neat, cm^{-1}): 3063.8, 2924.3, 2359.7, 1733.6, 1558.3, 1348.3, 1143.8; MS (ESI): m/z: 701.08 $[\text{M}+\text{Na}]^+$.



Borostannylation of diphenylacetylene: synthesis of the compound 4f: Compound **4f** was synthesized from diphenylacetylen in 84 % isolated yield using the general procedure described above. ^1H NMR (400 MHz, CDCl_3) δ 6.95-6.87 (m, 8 H), 6.62 (d, $J = 7.6$ Hz, 2 H), 1.28 (s, 12 H), 0.08 (s, 9H, $J_{\text{Sn}-\text{H}} = 26$ Hz, 9 H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.38, 146.82, 142.56, 129.72, 127.54, 127.14, 127.01, 126.93, 125.29, 124.57, 84.22, 77.44, 25.04, -4.86. IR (neat, cm^{-1}): 2923.8, 1732.9, 1652.0, 1557.9, 1493.9, 1455.9, 1049.4; MS (ESI): m/z: 493.13 $[\text{M}+\text{Na}]^+$.

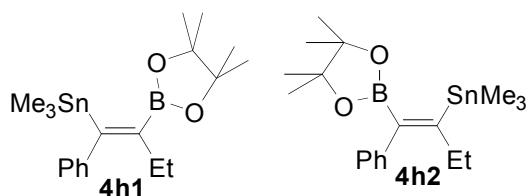
Borostannylation of 1-phenyl-1-propyne: synthesis of the compounds 4g1 and 4g2: Borostannylation of 1-phenyl-1-propyne using the general procedure described above yielded the two region isomers **4g1** and **4g2** in 88:12 ratio respectively in 90 % combined isolated yield.



4g1: ^1H NMR (500 MHz, CDCl_3) δ 7.26 (t, $J = 7.2$ Hz, 2 H), 7.06 (t, $J = 7.2$ Hz, 1 H), 6.78 (d, $J = 7.2$ Hz, 1 H), 1.64 (t, $J = 5.5$ Hz, 3 H), 1.28 (s, 12 H), -0.02 (t, $J = 26.0$ Hz, 9 H); ^{13}C NMR (125 MHz, CDCl_3) δ 167.79, 147.50, 128.18, 127.84, 125.93, 124.81, 83.91, 25.12, 18.92, -5.32; IR (neat, cm^{-1}): 2977.9, 2923.7, 2856.6, 1683.7, 1575.3, 1488.3, 1352.1, 1308.0, 1114.2, 760.9; MS (ESI): m/z: 431.11 $[\text{M}+\text{Na}]^+$.

4g2: ^1H NMR (500 MHz, CDCl_3) δ 7.27 (t, $J = 7.6$ Hz, 2 H), 7.16 (t, $J = 7.6$ Hz, 1 H), 7.02 (d, $J = 7.6$ Hz, 1 H), 1.89 (t, $J = 28.5$ Hz, 3 H), 1.21 (s, 12 H), 0.19 (t, $J = 27.0$ Hz, 9 H).

Borostannylation of 1-phenyl-1-butyne: synthesis of the compounds 4h1 and 4h2: Borostannylation of 1-phenyl-1-butyne using the general procedure described above yielded the two region isomers **4h1** and **4h2** in 85:15 ratio respectively in 89 % combined isolated yield. Regioselectivity was improved to 94:6 when the reaction was run at 60°C for 12 h. in the presence of $\text{PdCl}_2(\text{CH}_3\text{CN})_2$, $\text{P}(t\text{-Bu})_3$.

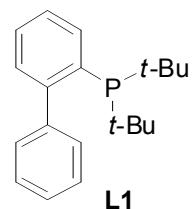


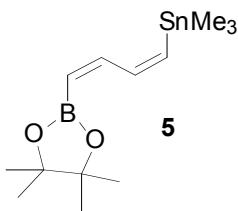
4h1: ^1H NMR (500 MHz, CDCl_3) δ 7.25 (t, $J = 7.5$ Hz, 2 H), 7.06 (t, $J = 7.5$ Hz, 1 H), 6.78 (d, $J = 7.5$ Hz, 2 H), 2.03 (q, $J = 10.0$ Hz, 2 H), 1.28 (s, 12 H), 0.85 (t, $J = 10.0$ Hz, 3 H), -0.03 (t, $J = 26.0$ Hz, 9 H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.37, 147.38, 128.04, 125.95, 125.88, 125.81, 124.67, 83.77, 26.32, 25.08, 15.70, -5.06; IR (neat, cm^{-1}): 2976.9, 2926.0, 2867.9, 1683.8, 1488.5, 1351.7, 1128.9, 968.4, 867.8; MS (ESI): m/z: 443.14 [M+Na]⁺.

4h2: ^1H NMR (500 MHz, CDCl_3) δ 7.26 (t, $J = 7.5$ Hz, 2 H), 7.17 (t, $J = 7.5$ Hz, 1 H), 6.99 (d, $J = 7.5$ Hz, 2 H), 2.30-2.10 (m, 2 H), 1.20 (s, 12 H), 0.80 (t, $J = 8.2$ Hz, 3 H), 0.19 (t, $J = 26.0$ Hz, 9 H)

Effect of ligands in improving the regioselectivity of the borostannylation of the internal unsymmetrical acetylene **2h** [Table]

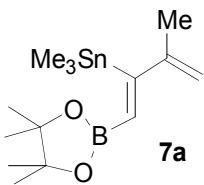
entry	reaction conditions	ratio of 4h1 : 4h2 (% yield)
1	a) $\text{PdCl}_2(\text{PPh}_3)_2$, C_6H_6 , rt, 2h.	86 : 13 (89)
	b) PTSA, Pinacol, rt. 12h.	
2	a) $\text{PdCl}_2(\text{CH}_3\text{CN})_2$, C_6H_6 , 80 °C, 12h.	No reaction, recovered starting material
	b) PTSA, Pinacol, rt. 12h.	
3	a) $\text{PdCl}_2(\text{CH}_3\text{CN})_2$, $\text{P}(\text{o-tolyl})_3$, C_6H_6 , rt, 6h.	87 : 13 (82)
	b) PTSA, Pinacol, rt. 12h., 82%	
4	a) $\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$, $\text{P}(\text{o-tolyl})_3$, C_6H_6 , rt, 24h.	87 : 13 (82)
	b) PTSA, Pinacol, rt. 12h.	
5	a) $\text{PdCl}_2(\text{CH}_3\text{CN})_2$, $\text{P}(t\text{-Bu})_3$, C_6H_6 , 60 °C, 12h.	94 : 6 (75)
	b) PTSA, Pinacol, rt. 12h.	
6	$\text{PdCl}_2(\text{CH}_3\text{CN})_2$, L1 , C_6H_6 , rt to 60 °C, 6h.	No reaction, recovered starting material
7	$\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$, L1 , C_6H_6 , rt to 60 °C, 24h.	No reaction, recovered starting material





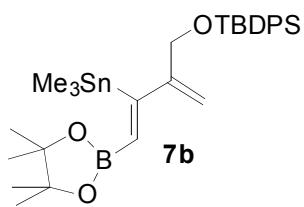
Borostannylative dimerization of acetylene: synthesis of the compound 5: Solution of mixture of 1,3-dimethyl-2-(trimethylstannyl)-2-bora-1,3-diazocyclopentane (156 mg, 0.6 mmol) and $\text{PdCl}_2(\text{PPh}_3)_2$ (3.5 mg, 0.005 mmol) in benzene (4 mL) was stirred at room temperature for 6 h under acetylene atmosphere (balloon pressure). After stirring the reaction mixture at room temperature for 6 h, a solution of pinacol (84 mg, 0.72 mmol) in benzene (0.5 mL) followed by *p*-toluenesulfonic acid (*p*-TSA, 136 mg, 0.72 mmol) were added. After 2 h, the reaction was

quenched by the addition of Et_3N (0.12 mL, 0.86 mmol) and the solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel chromatography using 2% EtOAc/hexane as the mobile phase to yield the desired product **5** in 86 % isolated yield.
 ^1H NMR (400 MHz, CDCl_3) δ 7.69 (t, $J = 12$ Hz, $J_{\text{Sn}-\text{H}} = 75$ Hz, 1 H), 6.79 (t, $J = 13.5$ Hz, 1 H), 6.38 (d, $J = 12.5$ Hz, $J_{\text{Sn}-\text{H}} = 24.5$ Hz, 1 H), 5.47 (d, $J = 13.5$ Hz, 1 H), 1.26 (s, 12 H), 0.18 (s, $J_{\text{Sn}-\text{H}} = 28$ Hz, 9 H); ^{13}C NMR (400 MHz, CDCl_3) δ 151.93, 146.48, 141.08, 83.40, 25.08, -0.8; IR (neat, cm^{-1}): 2977.9, 2939.6, 1733.4, 1606.3, 1418.2, 1143.1; MS (ESI): m/z: 367.079 [$\text{M}+\text{Na}$]⁺.



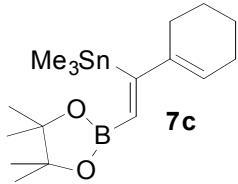
Borostannylation of 2-methyl-1-buten-3-yne: synthesis of the compound 7a: Compound **7a** was synthesized from 2-Methyl-1-buten-3-yne **6a** in 84 % isolated yield using the general procedure described above.

^1H NMR (500 MHz, CDCl_3) δ 6.12 (t, $J = 87.6$ Hz, 1 H), 4.74 (s, 1 H), 4.57 (s, 1 H), 1.78 (s, 3 H), 1.24 (s, 12 H), 0.18 (t, $J = 20.0$ Hz, 9 H); ^{13}C NMR (125 MHz, CDCl_3) δ 178.17, 152.96, 110.50, 83.57, 25.07, 22.49, -0.46; IR (neat, cm^{-1}): 2926.1, 2339.9, 1733.6, 1576.0, 1456.3, 1338.0, 1727.8, 1144.2, 960.5.

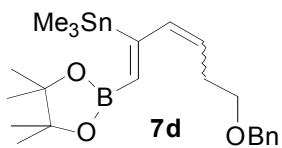


Borostannylation of alkyne 6b: synthesis of the compound 7b:

Compound **7b** was synthesized from alkyne **6b** in 82 % isolated yield using the general procedure described above. ^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, $J = 7.6$ Hz, 4 H), 7.48-7.30 (m, 6 H), 6.05 (t, $J = 92.0$ Hz, 1 H), 5.24 (s, 1 H), 4.70 (s, 1 H), 4.14 (s, 2 H), 1.22 (s, 12 H), 1.04 (s, 9 H), 0.13 (t, $J = 26.4$ Hz, 9 H); ^{13}C NMR (100 MHz, CDCl_3) δ 174.53, 154.97, 135.73, 133.85, 129.80, 127.86, 107.73, 83.59, 65.25, 27.04, 25.01, 19.53, -5.04; IR (neat, cm^{-1}): 2928.8, 2856.3, 2359.5, 1575.8, 1472.0, 1337.6, 1111.2, 969.1, 885.9; MS (ESI): m/z: 635.19 [$\text{M}+\text{Na}$]⁺.

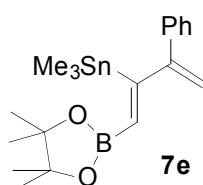


Borostannylation of 1-Ethynylcyclohexene: synthesis of the compound 7c: Compound **7c** was synthesized from 1-ethynylcyclohexene **6c** in 85 % isolated yield using the general procedure described above. ^1H NMR (400 MHz, CDCl_3) δ 6.07 (t, $J = 92.0$ Hz, 1 H), 5.46-5.35 (bs, 1 H), 2.16-1.98 (m, 4 H), 1.68-1.49 (m, 4 H), 1.24 (s, 12 H), 0.16 (t, $J = 28$ Hz, 9 H); ^{13}C NMR (125 MHz, CDCl_3) δ 178.68, 145.46, 124.18, 83.43, 27.98, 25.73, 25.06, 23.11, 22.54; IR (neat, cm^{-1}): 2976.8, 2926.3, 1733.2, 1652.1, 1558.1, 1335.9, 1143.5, 969.5, 768.7; MS (ESI): m/z: 421.13 [$\text{M}+\text{Na}$]⁺.



Borostannylation of alkyne 6d: synthesis of the compound 7d:

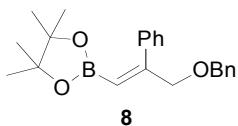
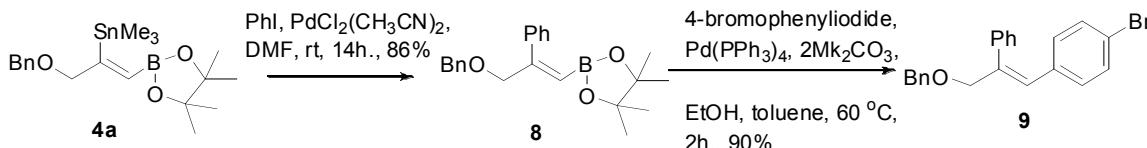
Compound **7d** was synthesized from alkyne **6d** in 81 % isolated yield using the general procedure described above. ¹H NMR (500 MHz, CDCl₃) δ 7.31-7.23 (m, 10 H), 6.34 (dd, *J* = 15.5, 1.0 Hz, 1 H), 6.22 (dd, *J* = 11.5, 2.0 Hz, 1 H), 6.17 (d, *J* = 1.0 Hz, 1 H), 6.07 (d, *J* = 2.0 Hz, 1 H), 5.82-5.72 (m, 1 H), 5.34-5.27 (m, 1 H), 4.50 (s, 2 H), 4.47 (s, 2 H), 3.50 (t, *J* = 6.5 Hz, 2 H), 3.44 (t, *J* = 7.0 Hz, 2 H), 2.42-2.36 (m, 4 H), 1.29-1.19 (m, 24 H), 0.28-0.12 (m, 18 H); IR (neat, cm⁻¹): 2976.8, 2924.3, 2855.9, 2359.5, 1575.8, 1455.8, 1343.5, 1143.1, 1103.8, 968.6, 850.7; MS (ESI): m/z: 501.15 [M+Na]⁺.



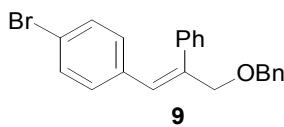
Borostannylation of alkyne 6e: synthesis of the compound 7e:

Compound **7e** was synthesized from alkyne **6e** in 10 % isolated yield using the general procedure described above. ¹H NMR (500 MHz, CDCl₃) δ 7.22-7.05 (m, 5 H), 6.27 (s, 1 H), 5.17 (d, *J* = 1.5 Hz, 1 H), 4.92 (d, *J* = 1.5 Hz, 1 H), 1.51 (s, 12 H), 0.25 (t, *J* = 24.3 Hz, 9 H).

Selective Stille Coupling followed by Suzuki Coupling:



Selective Stille coupling: Synthesis of the compound 8: The substrate **4a** (20 mg, 0.05 mmol) in DMF (0.8 mL) was added PdCl₂(CH₃CN)₂ (0.6 mg, 0.0025 mmol) followed by the solution of iodobenzene (12 mg, 0.06 mmol) in DMF (0.2 ml), and the reaction mixture was stirred at the rt for 12 h. The reaction mixture was diluted with diethyl ether and washed successively with water, brine and dried over Na₂SO₄. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 3% EtOAc/hexane as the eluent to yield **8** (15 mg, 0.04 mmol) in 86% yield as a viscous oil. ¹H NMR (500 MHz, CDCl₃) δ 7.31-7.24 (m, 5H), 5.81 (t, *J* = 1.5 Hz, 1H), 4.54 (s, 2H), 4.29 (d, *J* = 1.5 Hz, 2H), 1.12 (s, 9H).

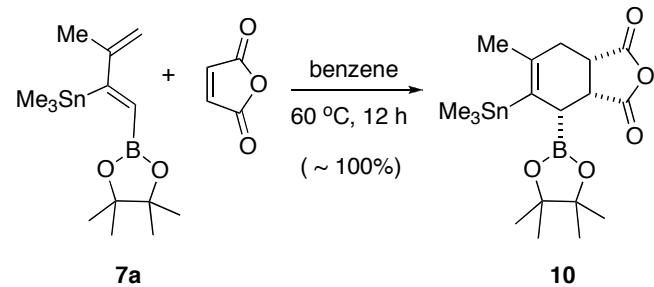


Suzuki Coupling for the Synthesis of 9: To the substrate **8** (8 mg, 0.025 mmol) in toluene(0.5 mL) under an atmosphere of nitrogen was added the 1-bromo-4-iodobenzene (14 mg, 0.05 mmol) in 0.32 mL toluene followed by 0.3 mL of 2 M potassium carbonate solution and 0.3 mL EtOH . To this mixture was added *tetrakis*-(triphenylphosphine)palladium (1.5 mg), and the reaction was heated at 60 °C for 2 h. The reaction mixture was diluted with diethyl ether and the solution was washed successively with water, brine and dried over Na₂SO₄. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 5 % EtOAc/hexane as the mobile phase to yield the compound **9** (8 mg, 0.022 mmol) in 90% yield as a viscous oil. ¹H NMR (400 MHz,

solution and 0.3 mL EtOH . To this mixture was added *tetrakis*-(triphenylphosphine)palladium (1.5 mg), and the reaction was heated at 60 °C for 2 h. The reaction mixture was diluted with diethyl ether and the solution was washed successively with water, brine and dried over Na₂SO₄. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 5 % EtOAc/hexane as the mobile phase to yield the compound **9** (8 mg, 0.022 mmol) in 90% yield as a viscous oil. ¹H NMR (400 MHz,

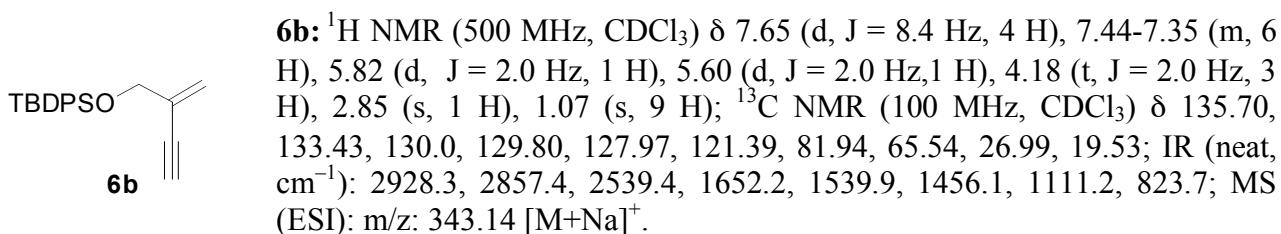
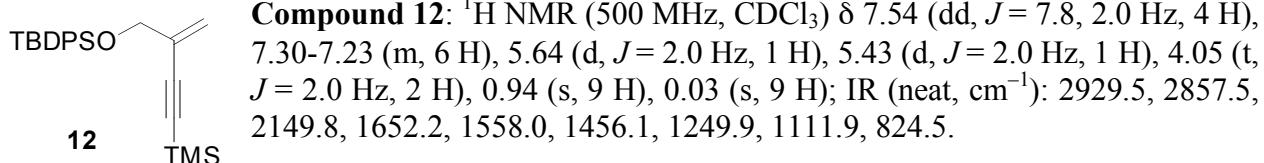
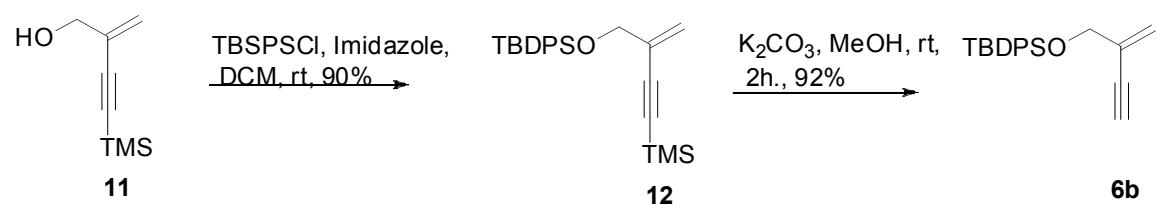
CDCl_3) δ 7.32-7.16 (m, 12H), 6.83 (d, $J = 8.0$ Hz, 2H), 6.63 (s, 1H), 4.59 (s, 2H), 4.29 (d, $J = 1.2$ Hz, 2H).

Diels-Alder reaction of **7a**

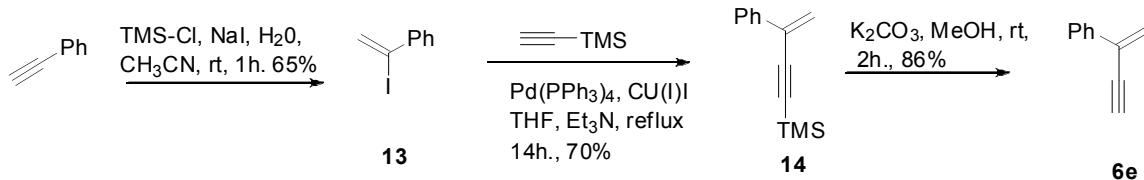


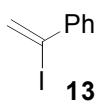
Diene **7a** (18 mg, 0.05 mmol) and maleic anhydride (10 mg, 0.05 mmol) in benzene (1.5 mL) were refluxed for 12 h. Evaporation of the solvent under reduced pressure yielded the desired product **10** in quantitative yield. ^1H NMR (500 MHz, CDCl_3) δ 3.17-3.12 (m, 1 H), 3.05 (dd, $J = 8.0, 11.0$ Hz, 1 H), 2.71 (dd, $J = 8.0, 30.0$ Hz, 1 H), 2.53-2.42 (m, 2 H), 1.84 (dt, $J = 25.0, 5.5$ Hz, 3 H), 1.12 (s, 6 H), 1.09 (s, 6 H), 0.19 (t, $J = 26$ Hz, 9 H). The configuration was assigned as shown based on the ample literature precedents on reactions of similar compounds.

Synthesis of enyne **6b:** Enyne **6b** was synthesized from the known compound **11**, which was obtained from the propargyl alcohol using the literature known procedure (*J. Org. Chem.* **2005**, *70*, 2364-2367).

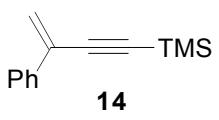


Synthesis of enyne **6e:** Enyne **6e** was synthesized from the phenyl acetylene using the procedure described for the synthesis of enyne **6b** from the propargyl alcohol. (*J. Org. Chem.* **2005**, *70*, 2364-2367).

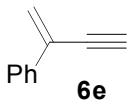




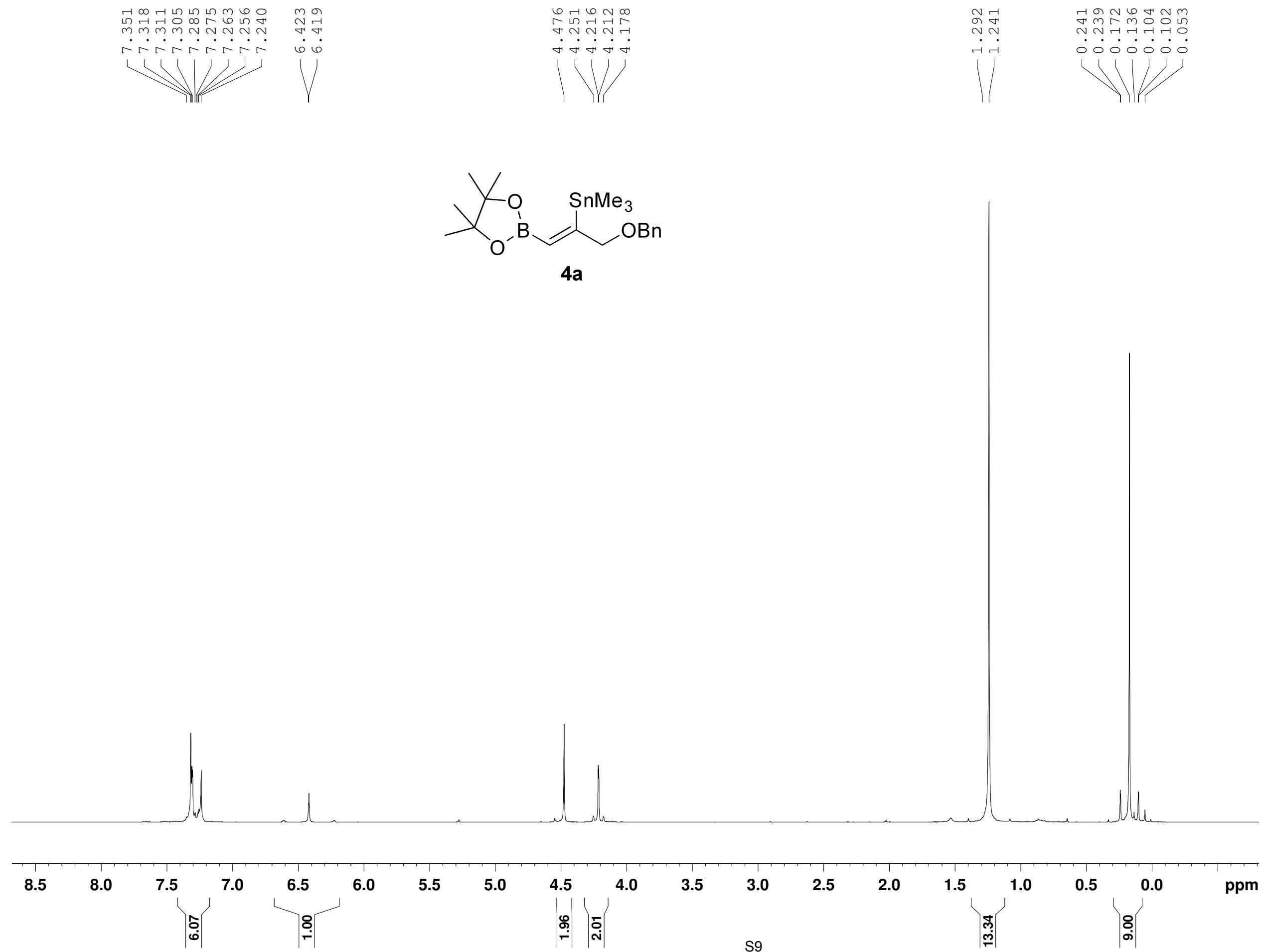
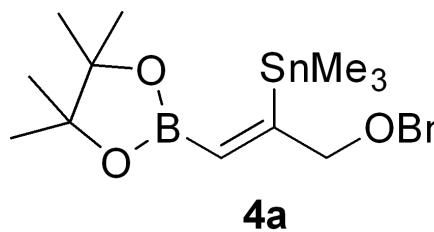
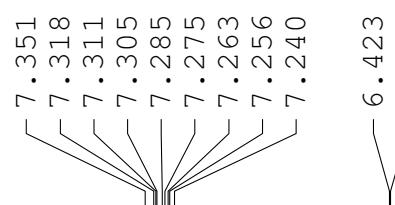
¹H NMR (500 MHz, CDCl₃) δ 7.45 (dd, *J* = 8.0, 2.0 Hz, 2 H), 7.27-7.19 (m, 3 H), 6.40 (d, *J* = 2.0 Hz, 1 H), 6.02 (d, *J* = 2.0 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃) δ 141.28, 129.14, 128.52, 128.37, 127.59, 107.88.



¹H NMR (400 MHz, CDCl₃) δ 7.63 (dd, *J* = 7.8, 1.5 Hz, 2 H), 7.36-7.29 (m, 3 H), 5.92 (s, 1 H), 5.70 (s, 1 H), 0.24 (s, 9 H); ¹³C NMR (100 MHz, CDCl₃) δ 137.16, 130.87, 128.59, 128.55, 126.28, 121.68, 104.33, 96.14, 0.18; IR (neat, cm⁻¹): 2958.0, 2898.2, 2162.0, 2066.7, 1733.1, 1645.7, 1598.3, 1491.2, 1447.9, 1249.5, 1036.0, 842.0.



¹H NMR (400 MHz, CDCl₃) δ 7.65 (d, *J* = 7.6 Hz, 2 H), 7.37-7.28 (m, 3 H), 5.97 (s, 1 H), 5.75 (s, 1 H), 3.10 (s, 1 H); ¹³C NMR (100 MHz, CDCl₃) δ 136.85, 129.96, 128.66, 128.62, 126.20, 122.45, 82.98, 78.86; IR (neat, cm⁻¹): 2925.1, 2857.0, 2162.0, 1733.0, 1652.2, 1493.8, 1448.2, 1397.7, 1049.6.



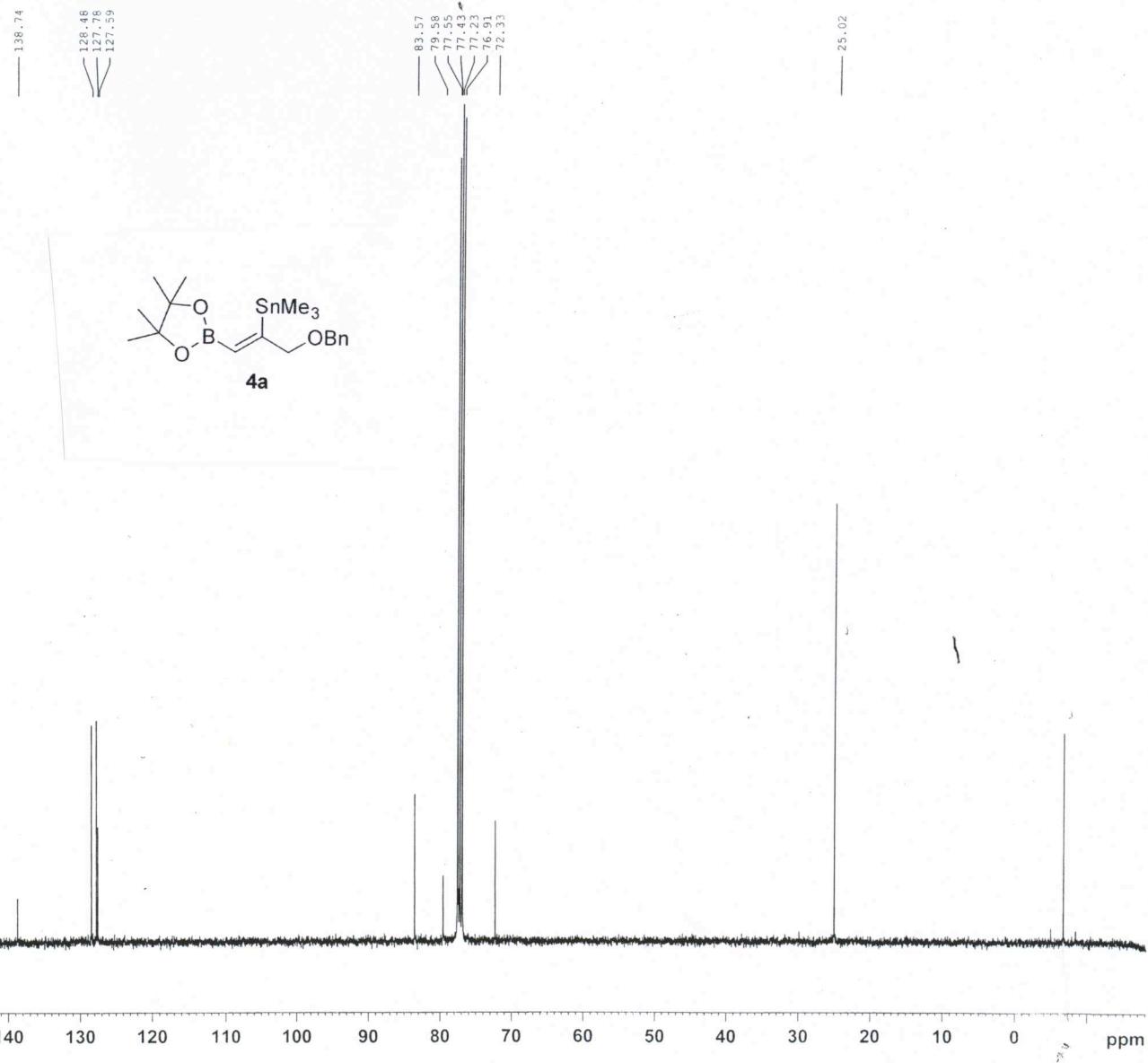
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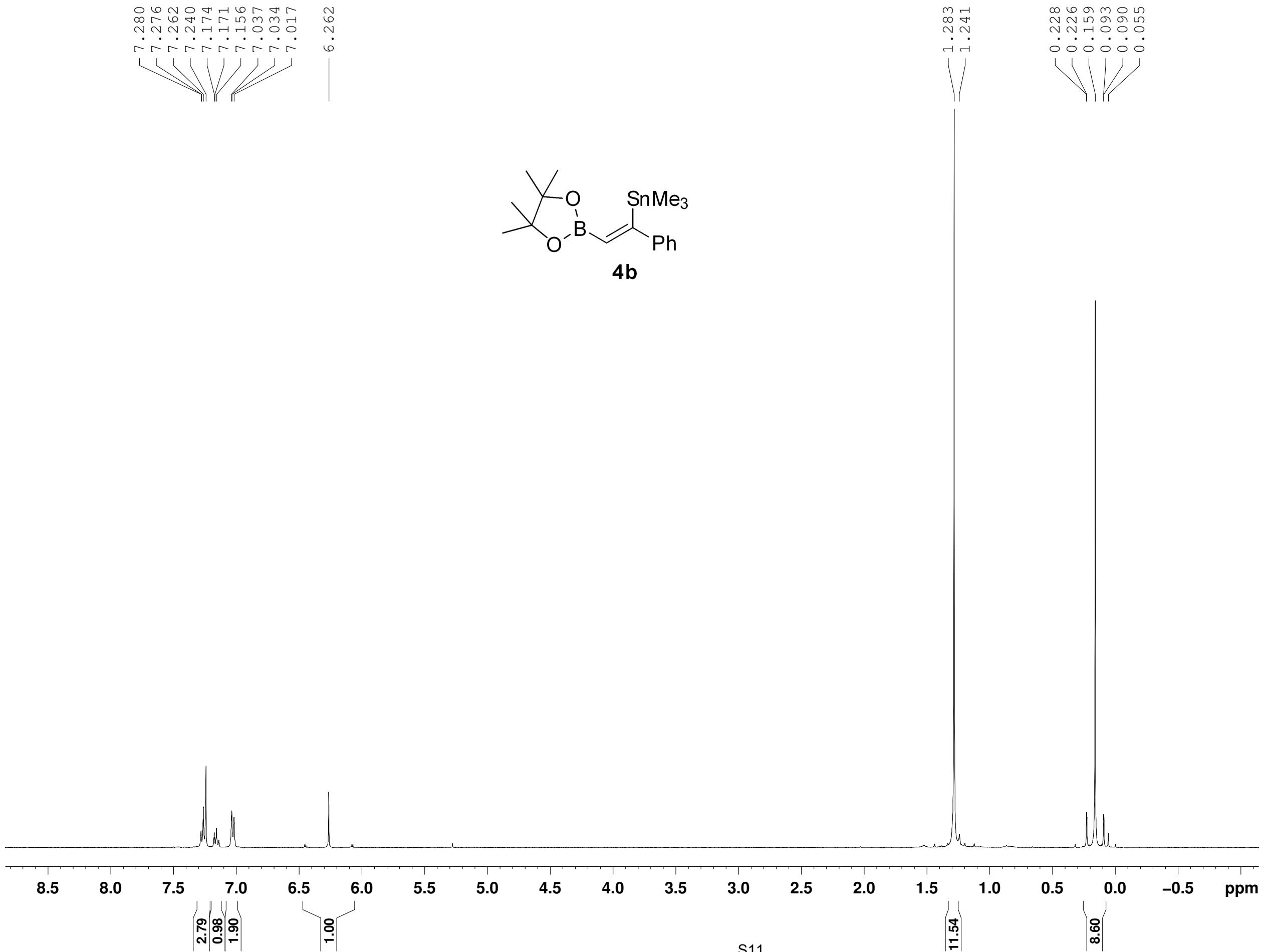
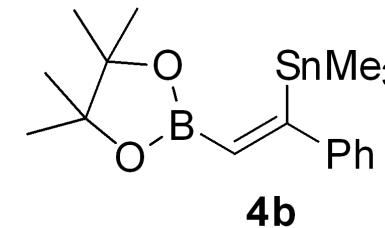
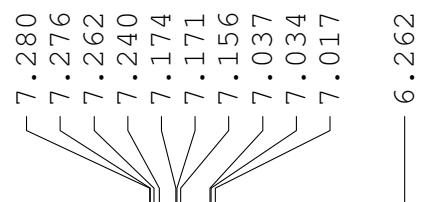
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FIDRES 0.126314 Hz
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RG 114
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DE 6.00 use
TE 300.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

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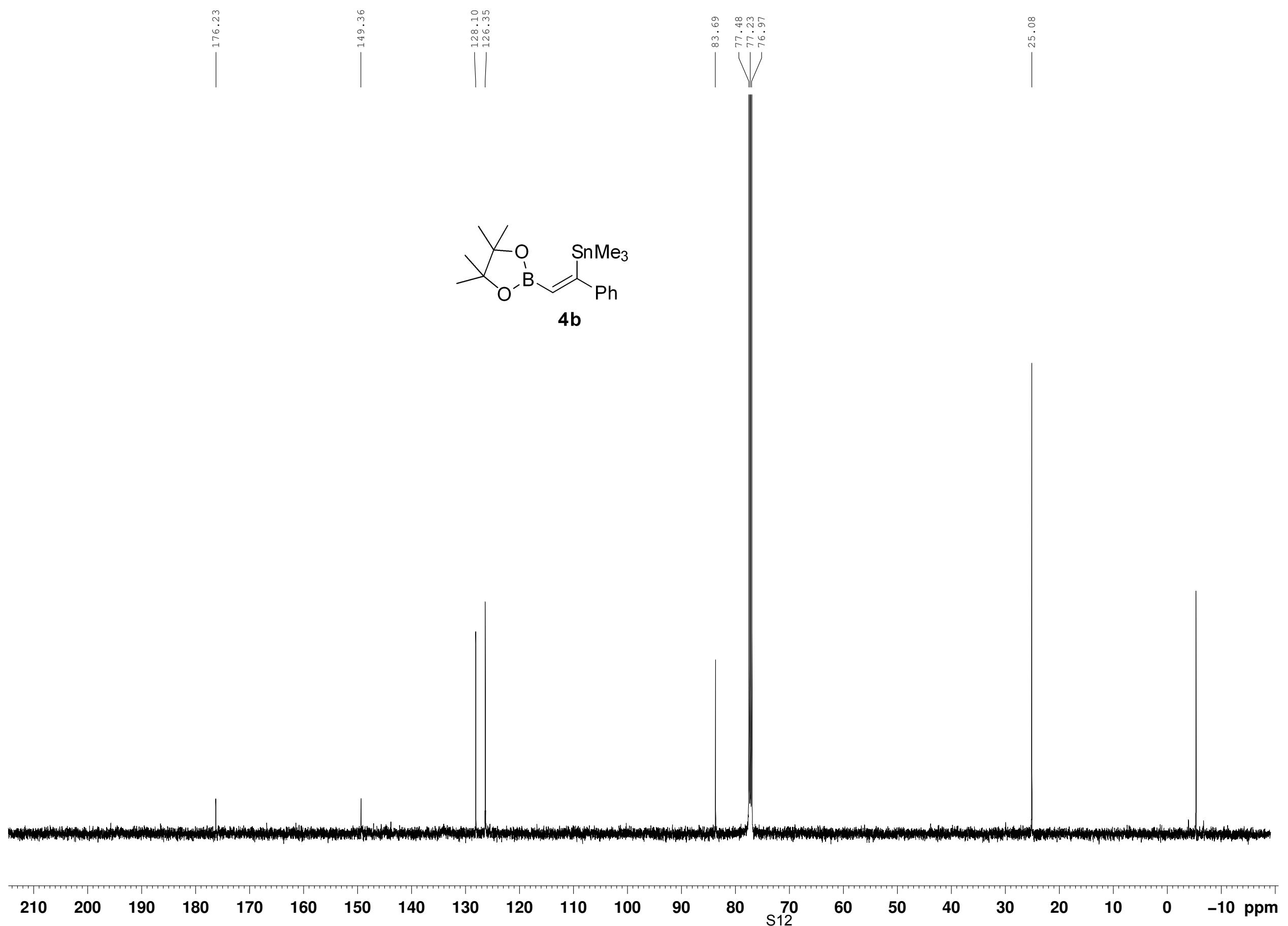
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SOLVENT CDCl3
NS 16
DS 2
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RG 912.3
DW 60.400 use
DE 6.00 use
TE 300.2 K
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PL1 0.00 dB
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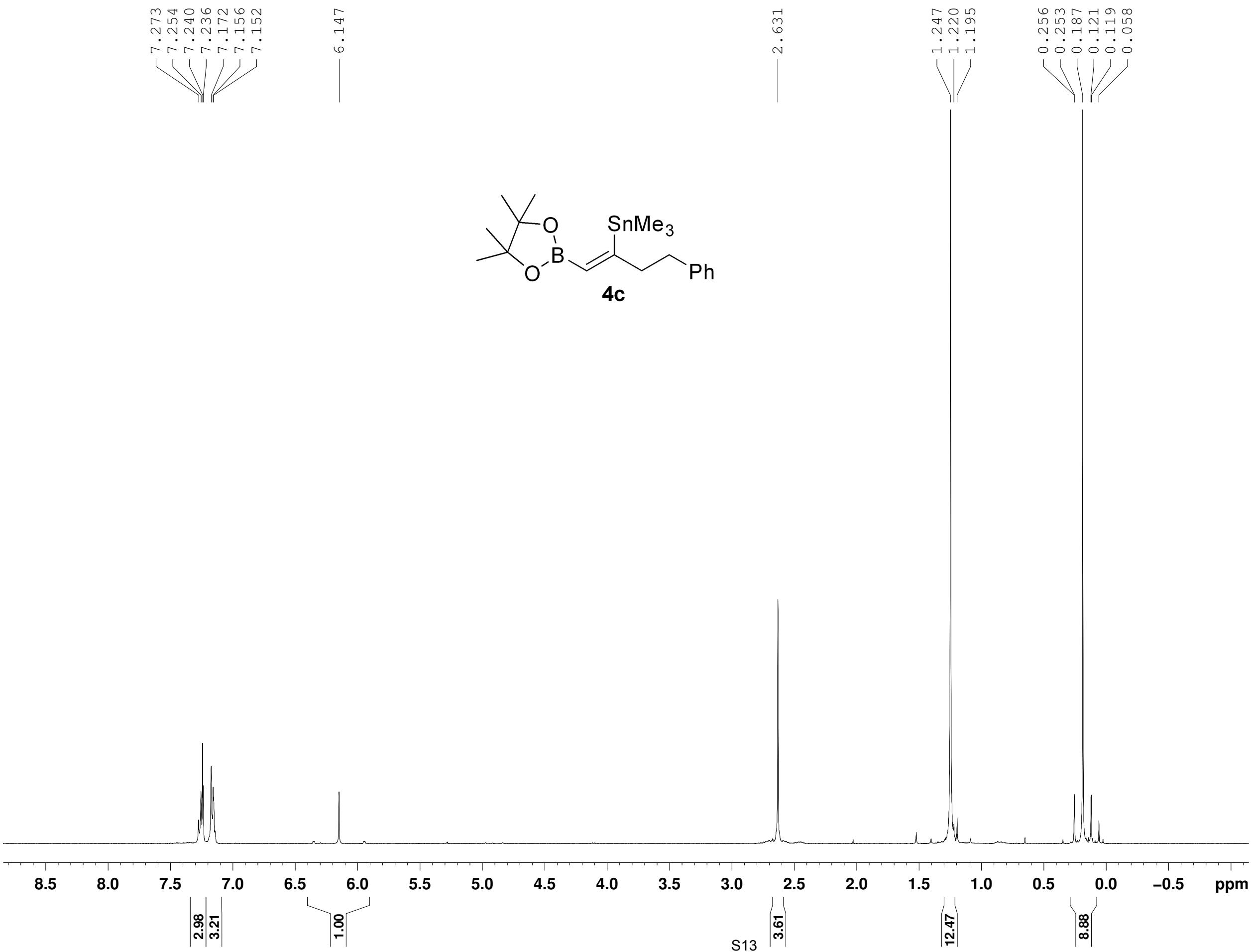
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 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 6502
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 D1 2.0000000 sec
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 PL1 3.00 dB
 SFO1 125.7427020 MHz

===== CHANNEL f2 =====
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 PL2 -1.00 dB
 PL12 15.59 dB
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F2 - Processing parameters
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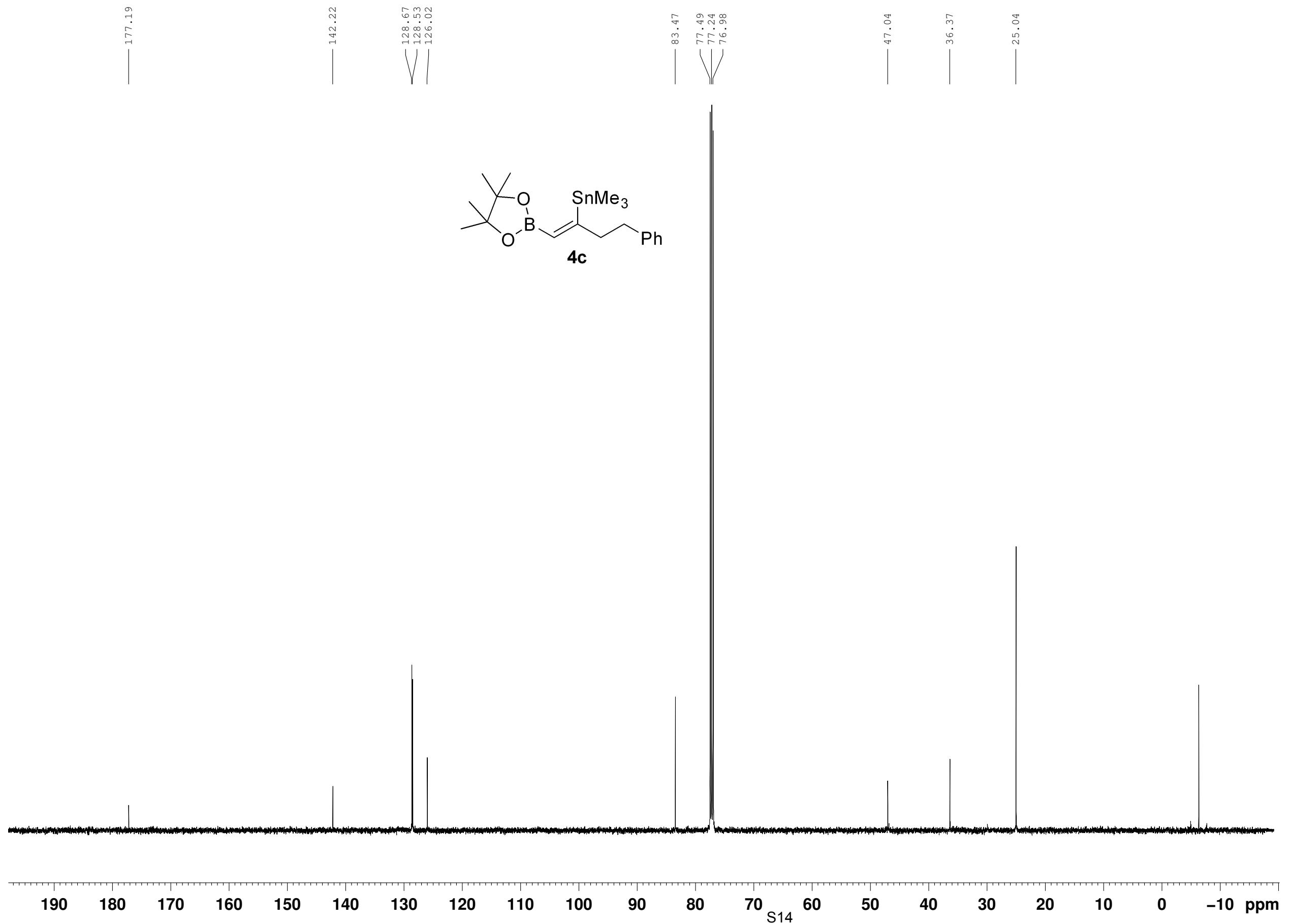
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FIDRES 0.126314 Hz
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RG 645.1
DW 60.400 use
DE 6.00 use
TE 300.2 K
D1 1.0000000 sec
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===== CHANNEL f1 ======

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F2 – Processing parameters
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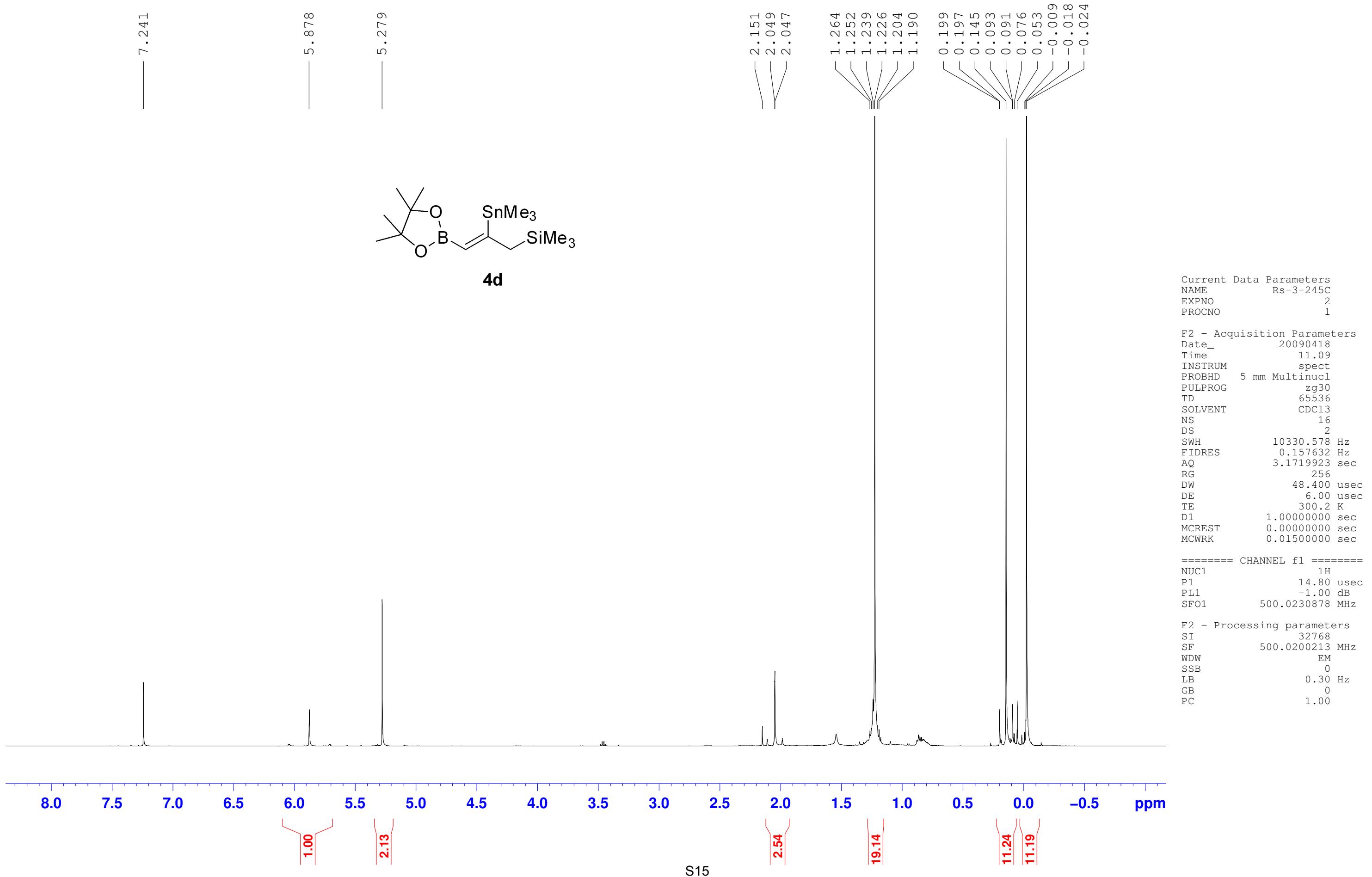
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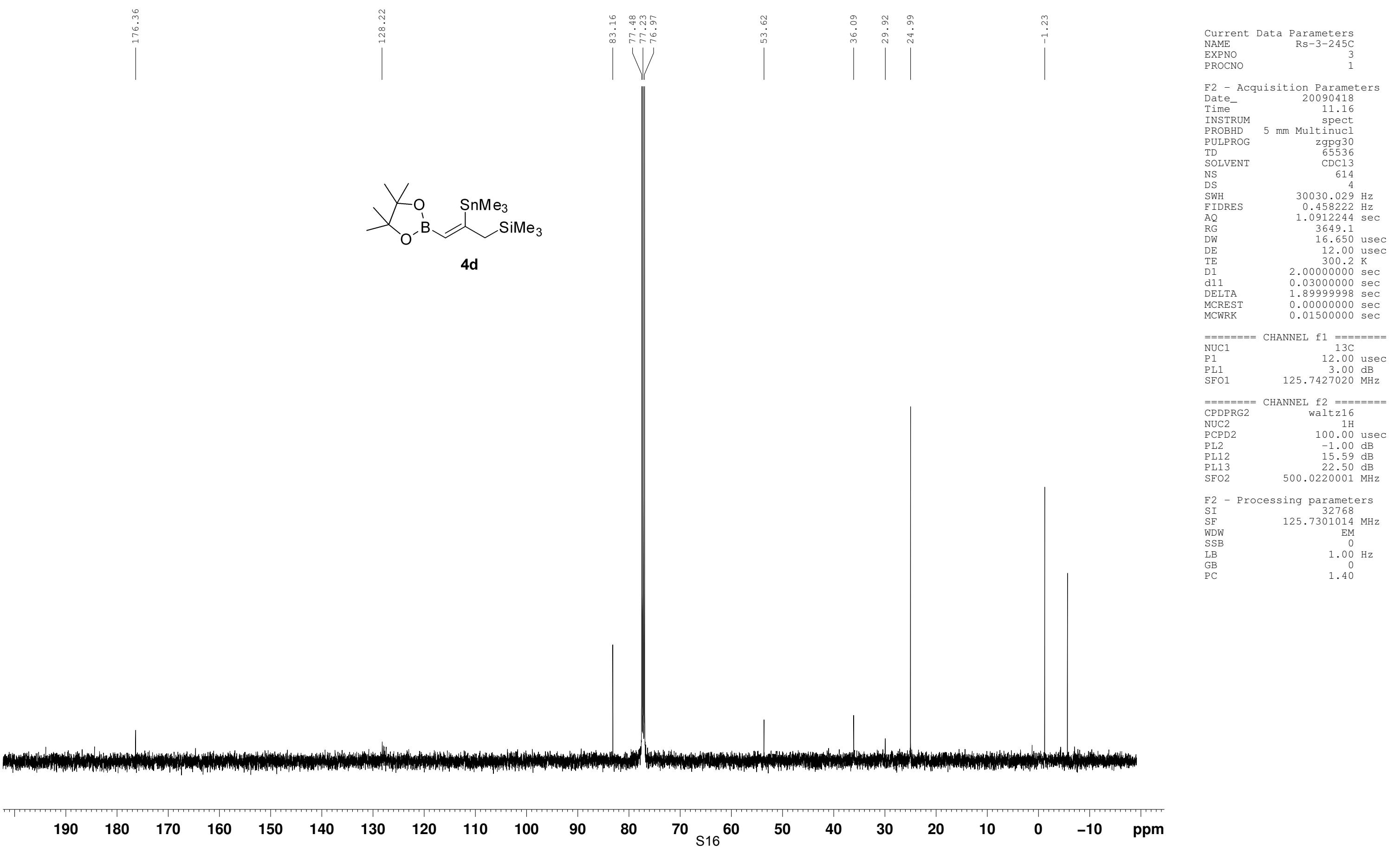
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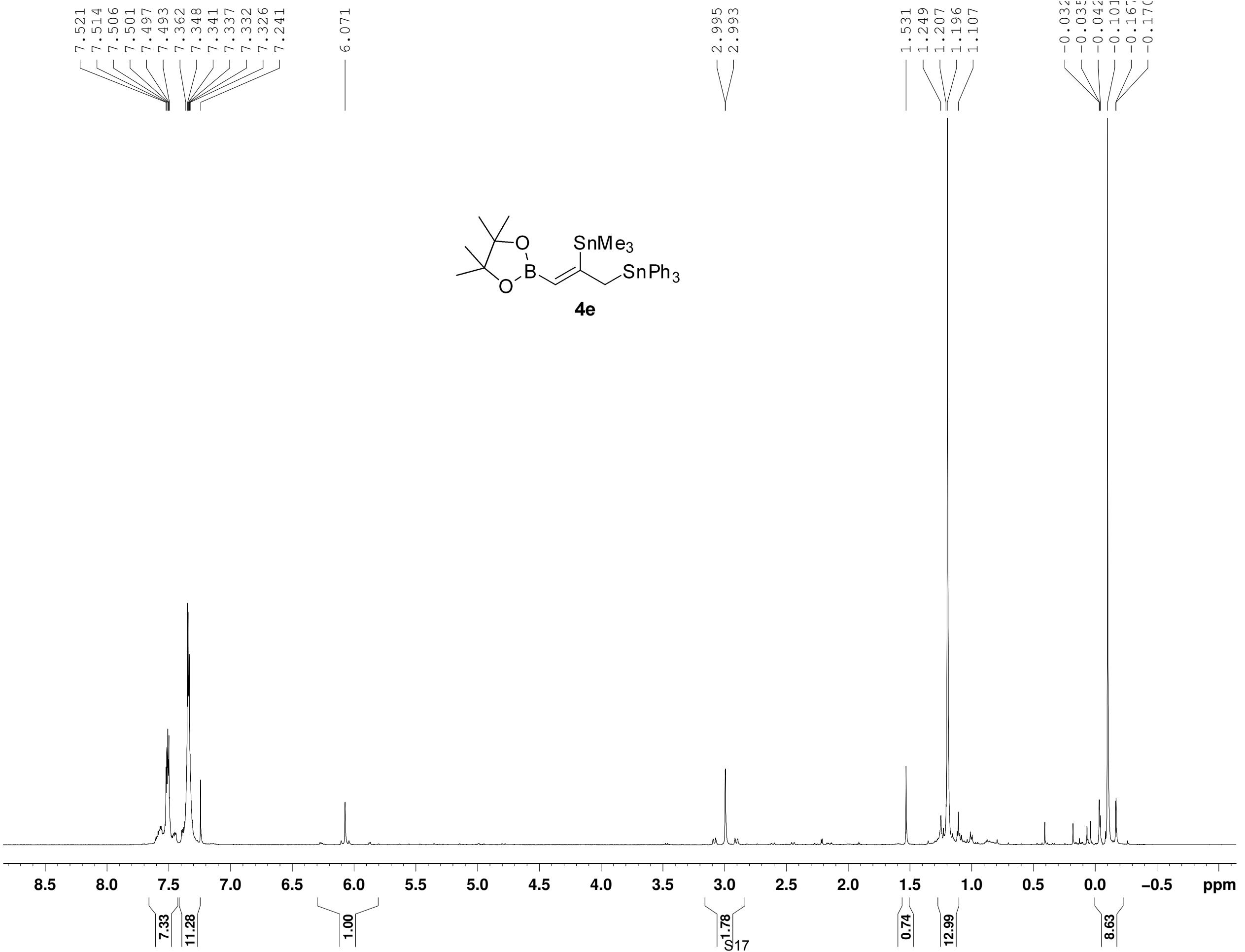
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PCPD2 100.00 usec
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PL12 15.59 dB
PL13 22.50 dB
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F2 - Processing parameters
SI 32768
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Current	Data	Parameters
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FIDRES        0.126314 Hz
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RG              128
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F2 - Processing parameters
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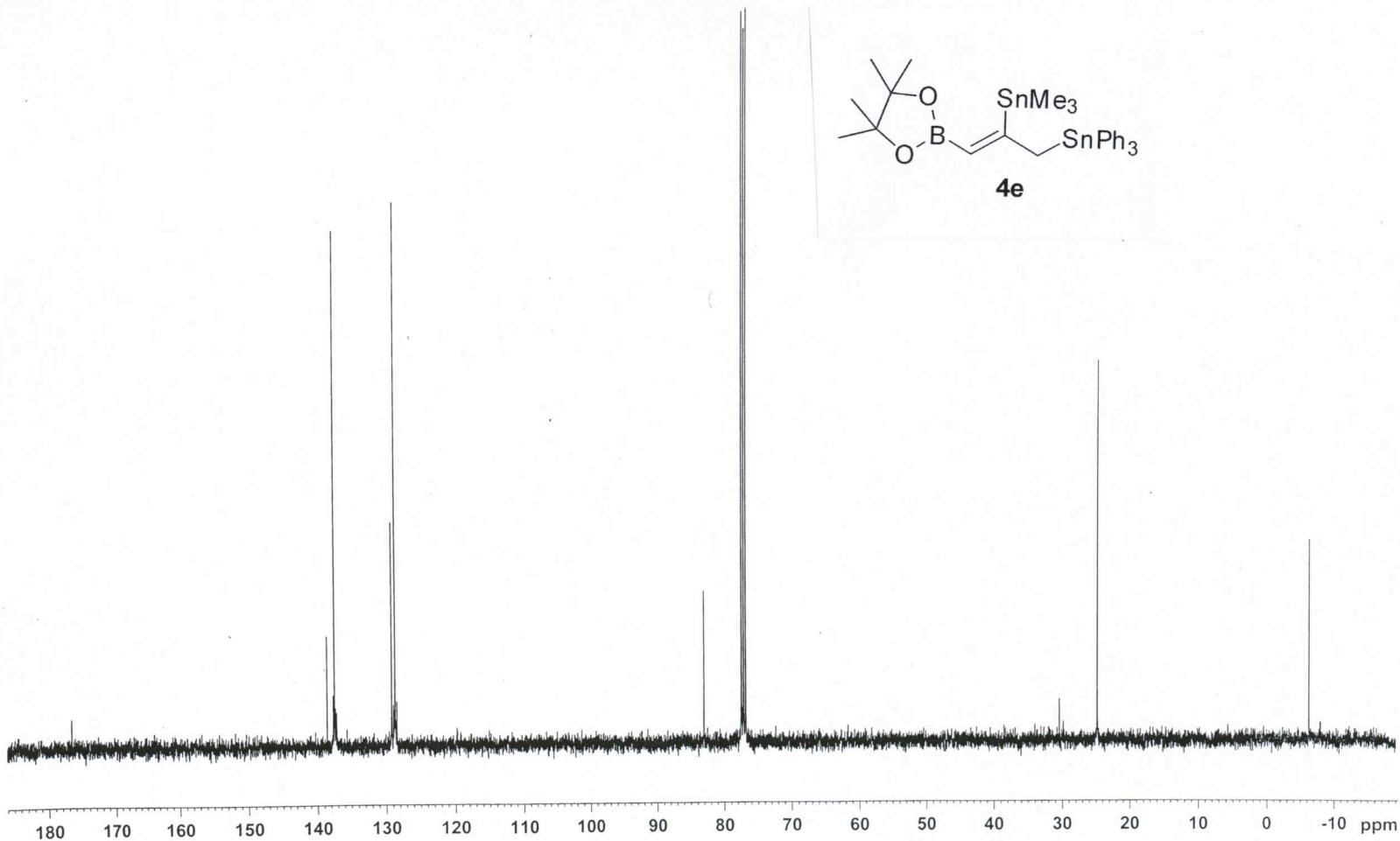
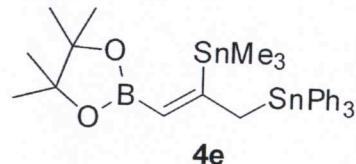
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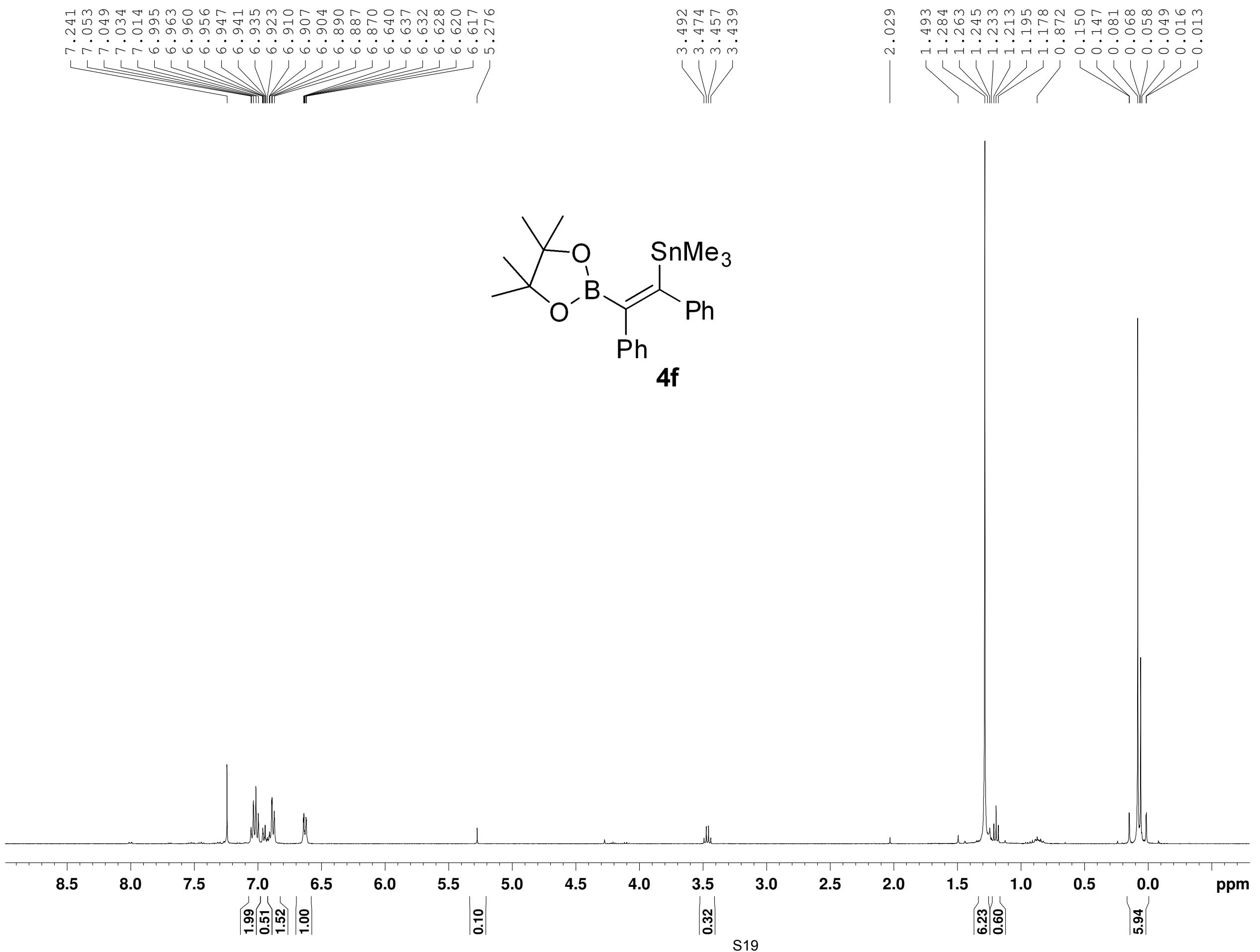
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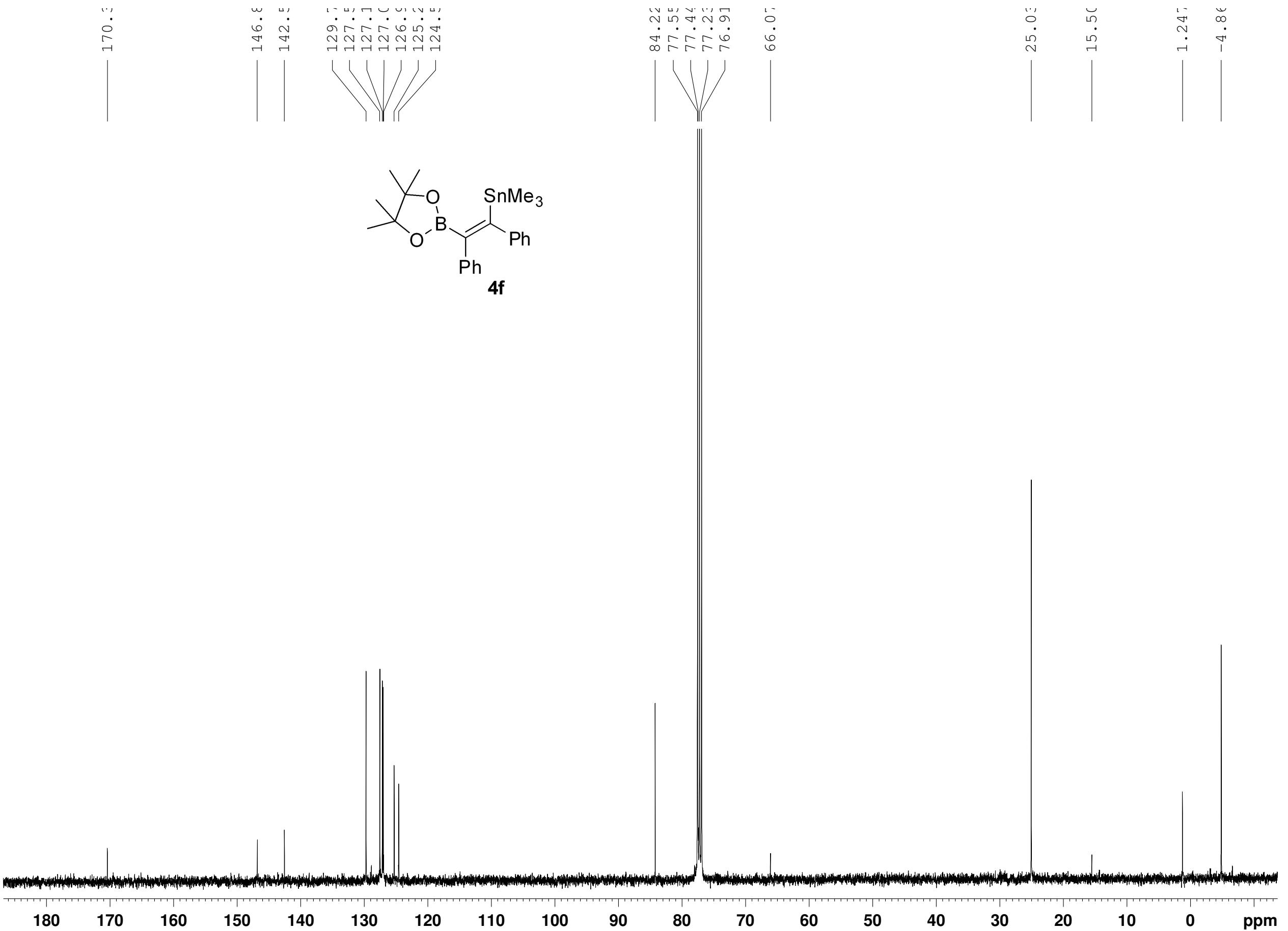
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NS 16
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FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 724.1
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MCREST 0.0000000 sec
MCWRK 0.0150000 sec

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F2 – Processing parameters
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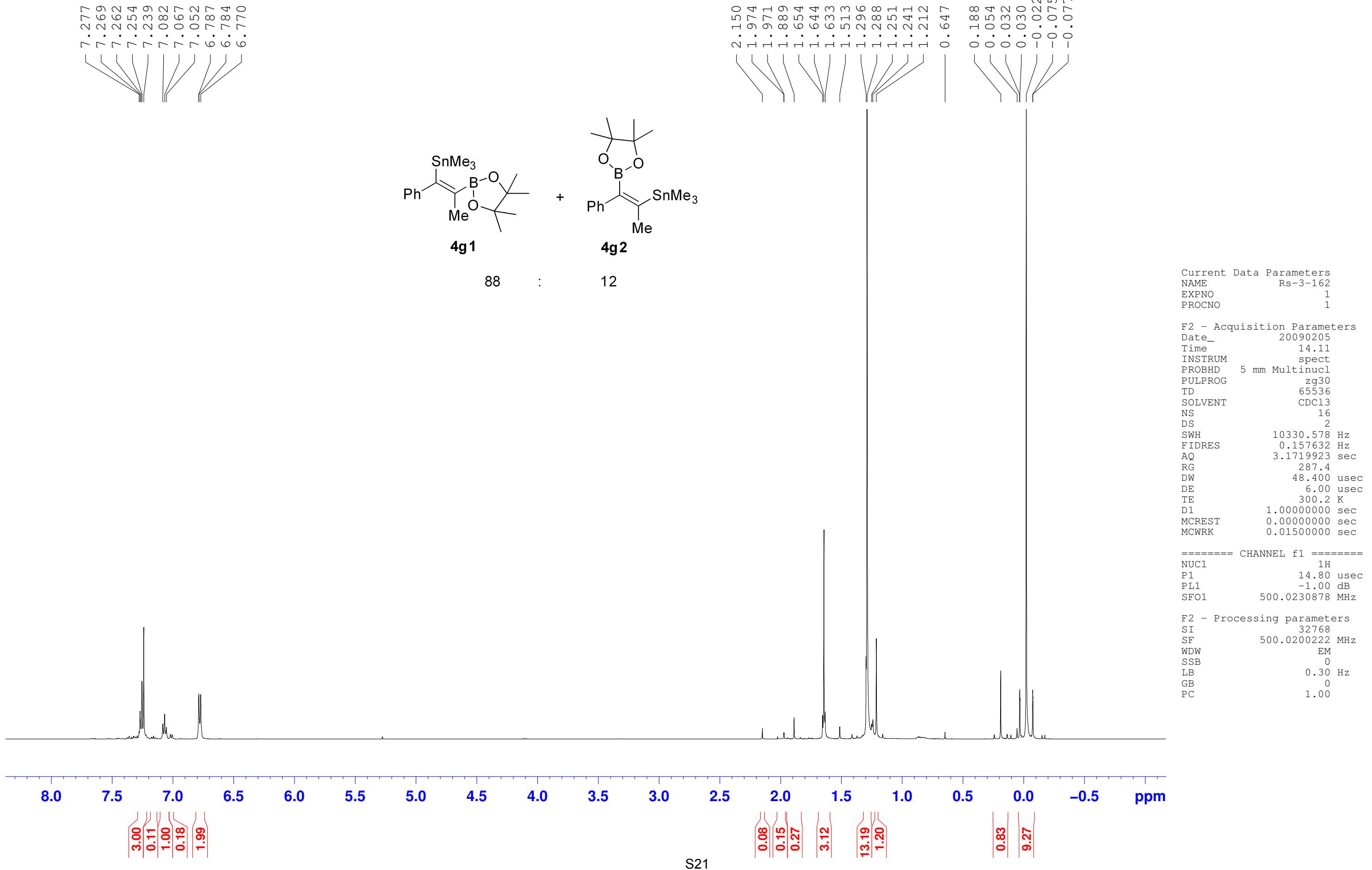
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EXPNO 2
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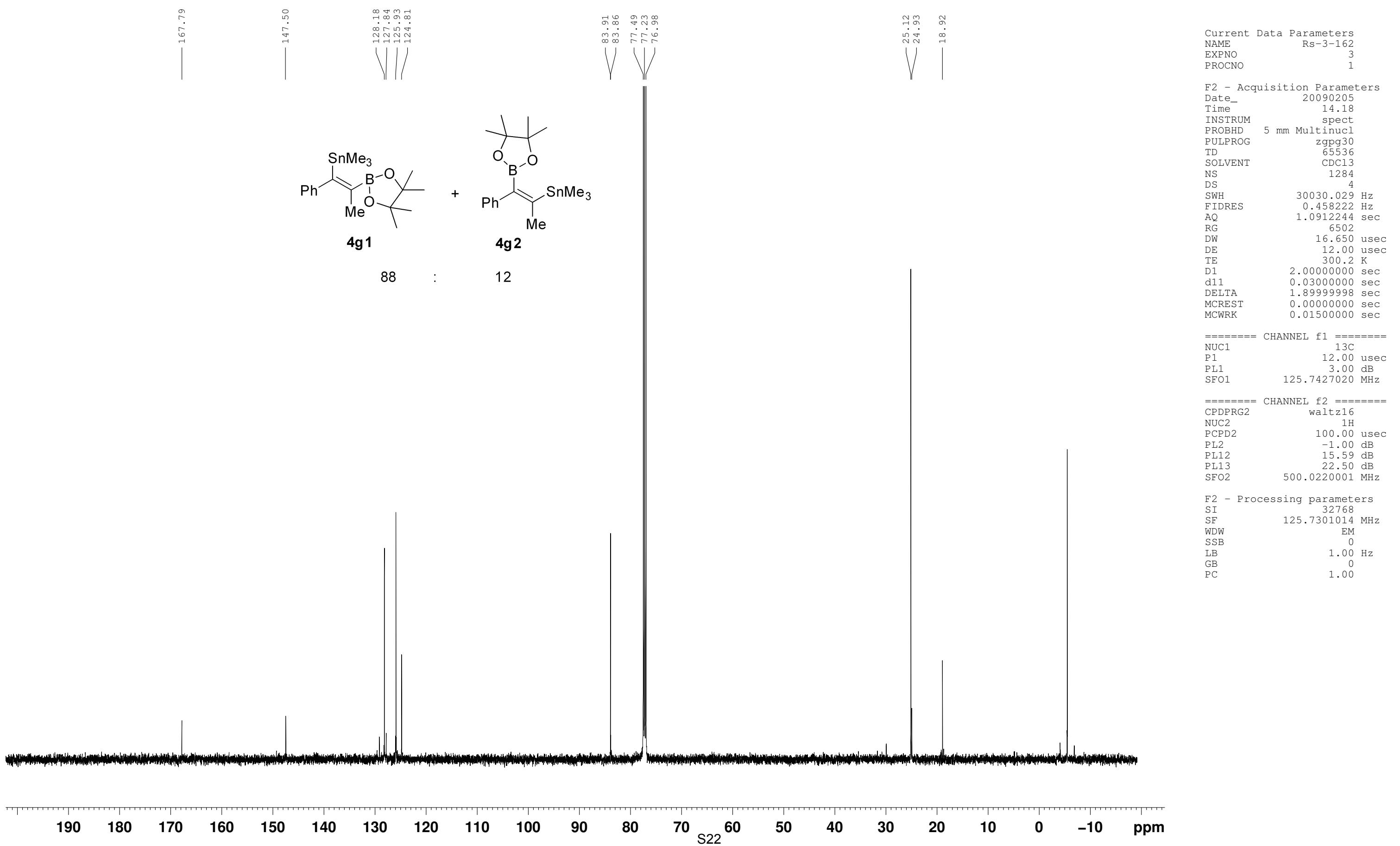
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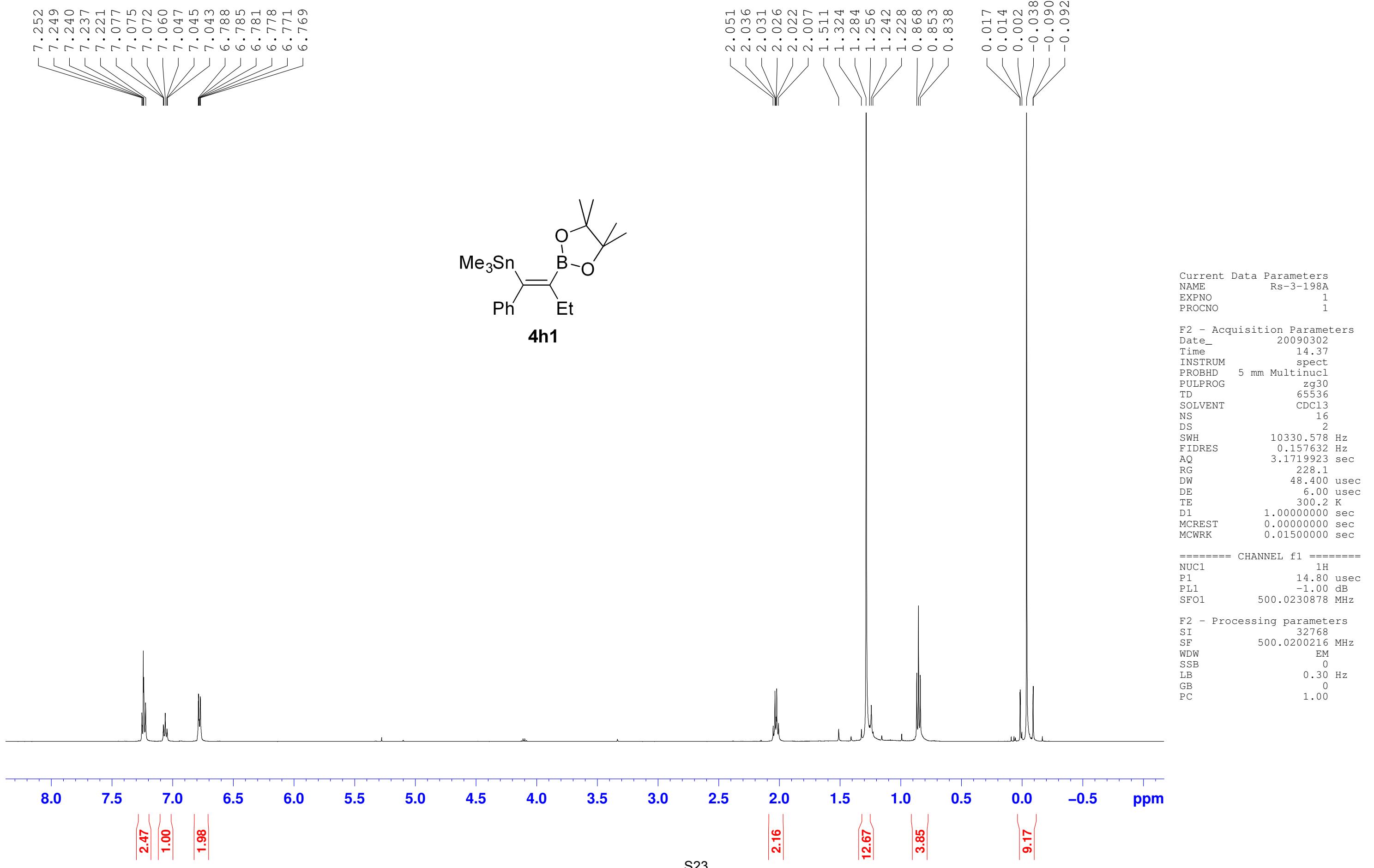
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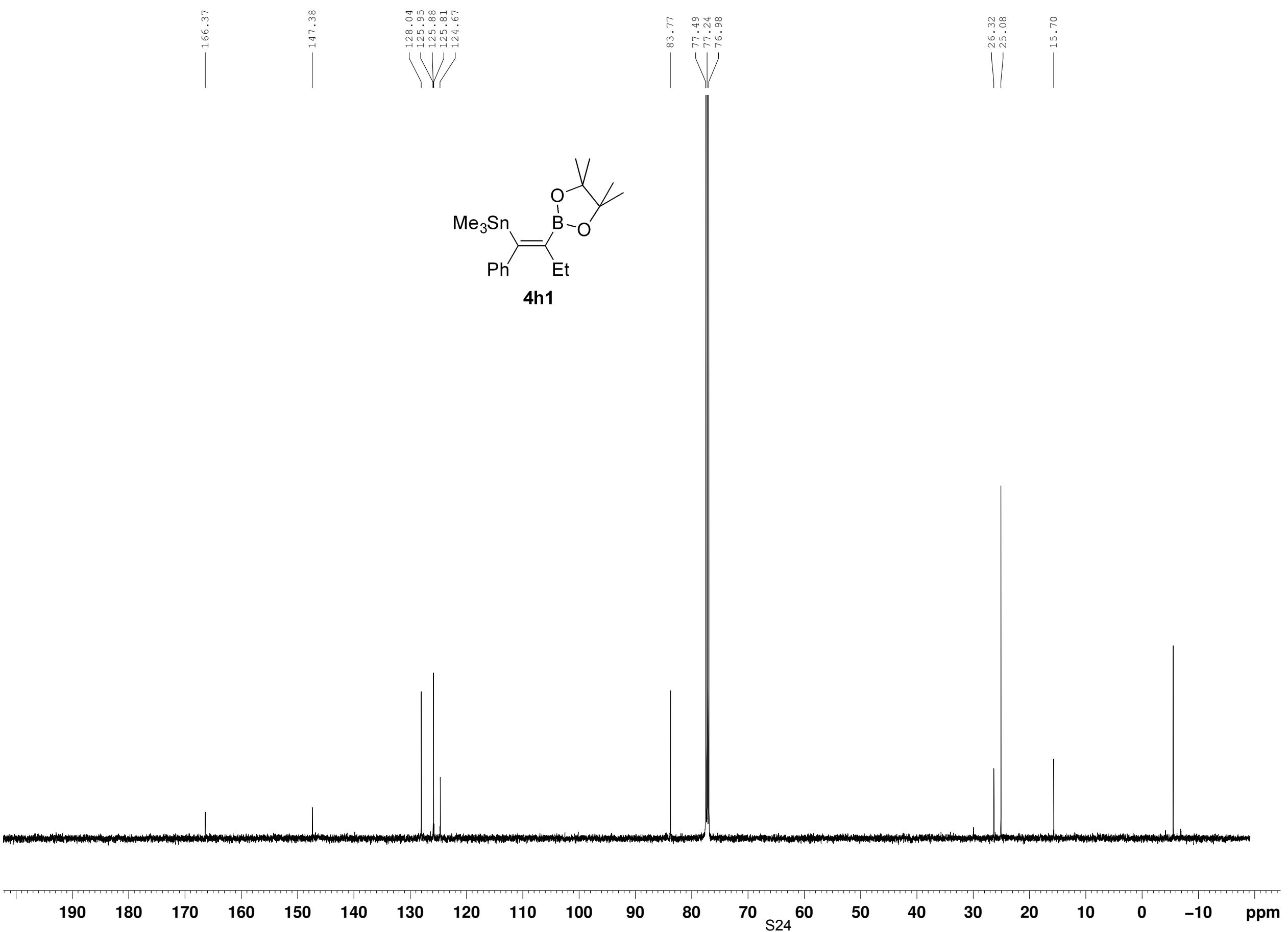
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F2 – Processing parameters
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SF 100.6127469 MHz
WDW EM
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LB 1.00 Hz
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PC 1.40









Current Data Parameters
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 PROCNO 1

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 PULPROG zpgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 870
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 2580.3
 DW 16.650 usec
 DE 12.00 usec
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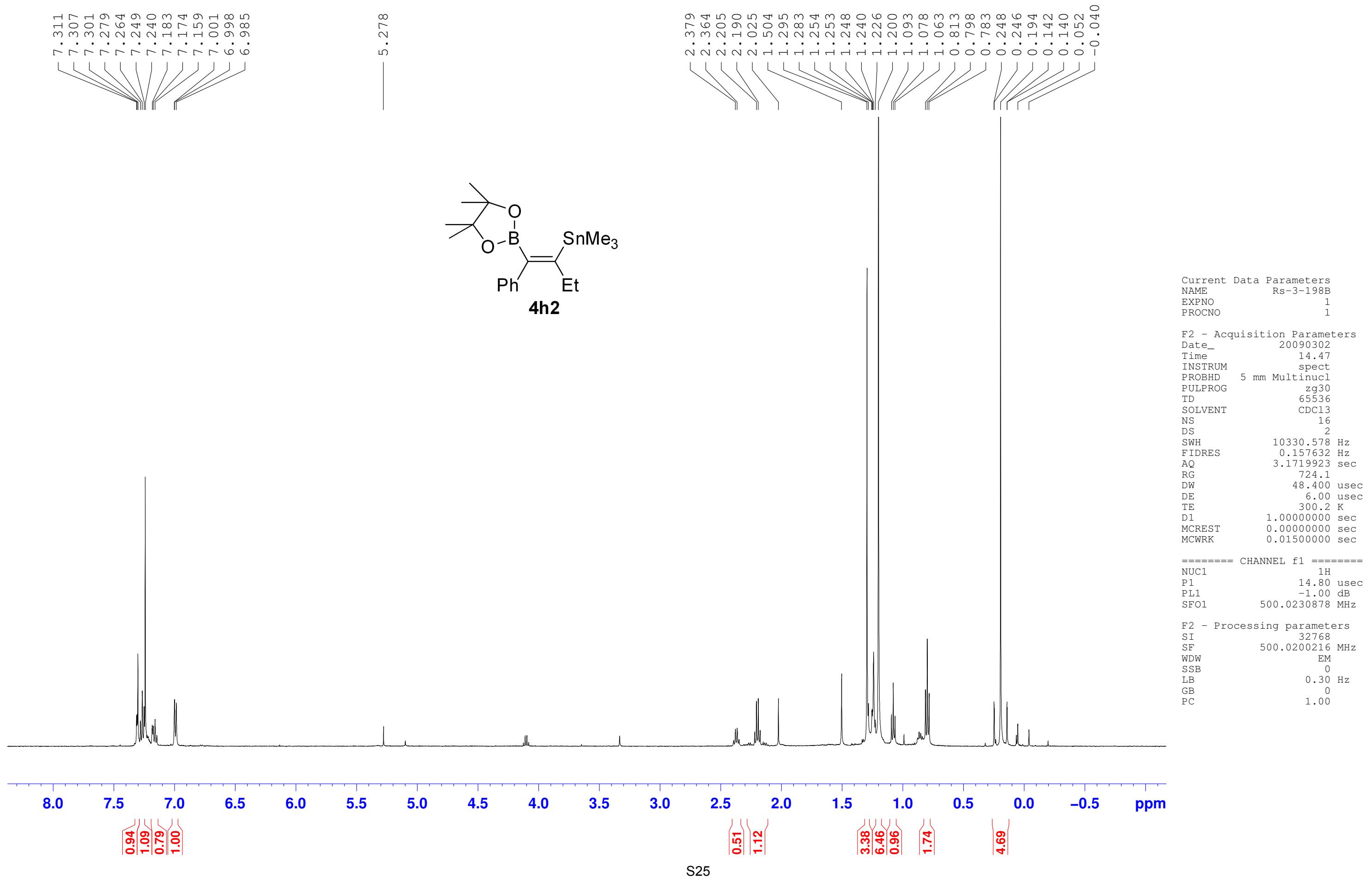
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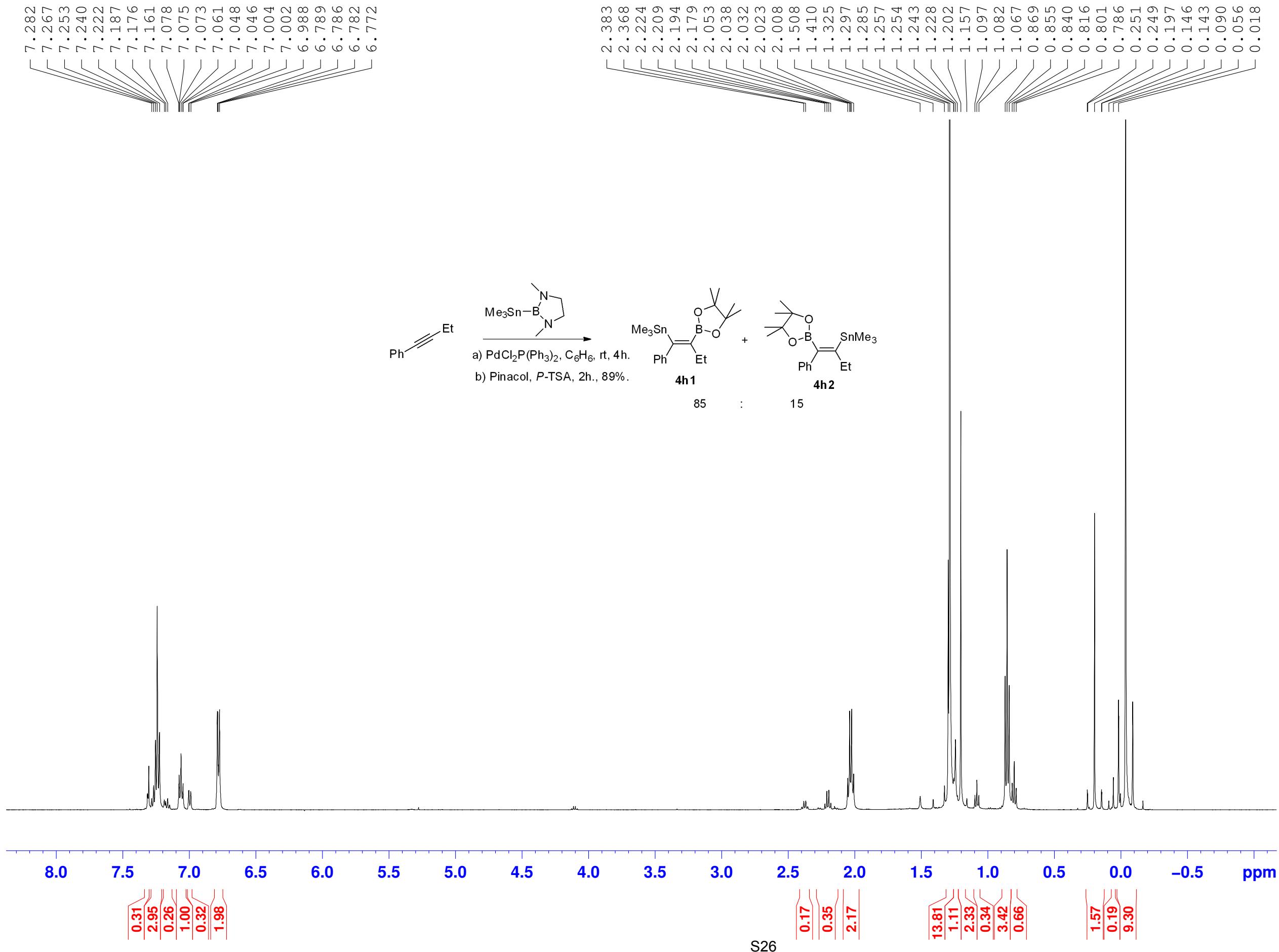
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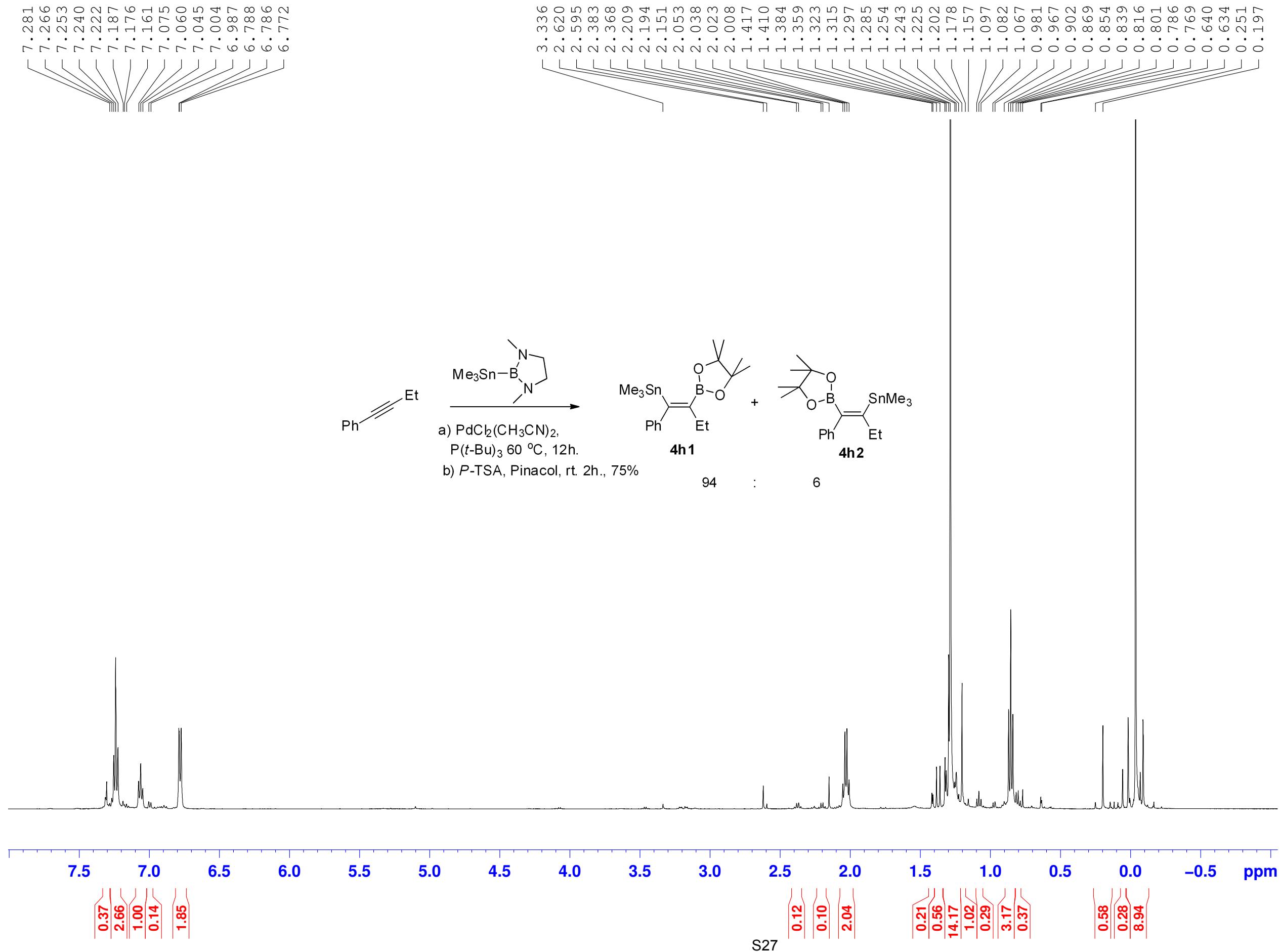
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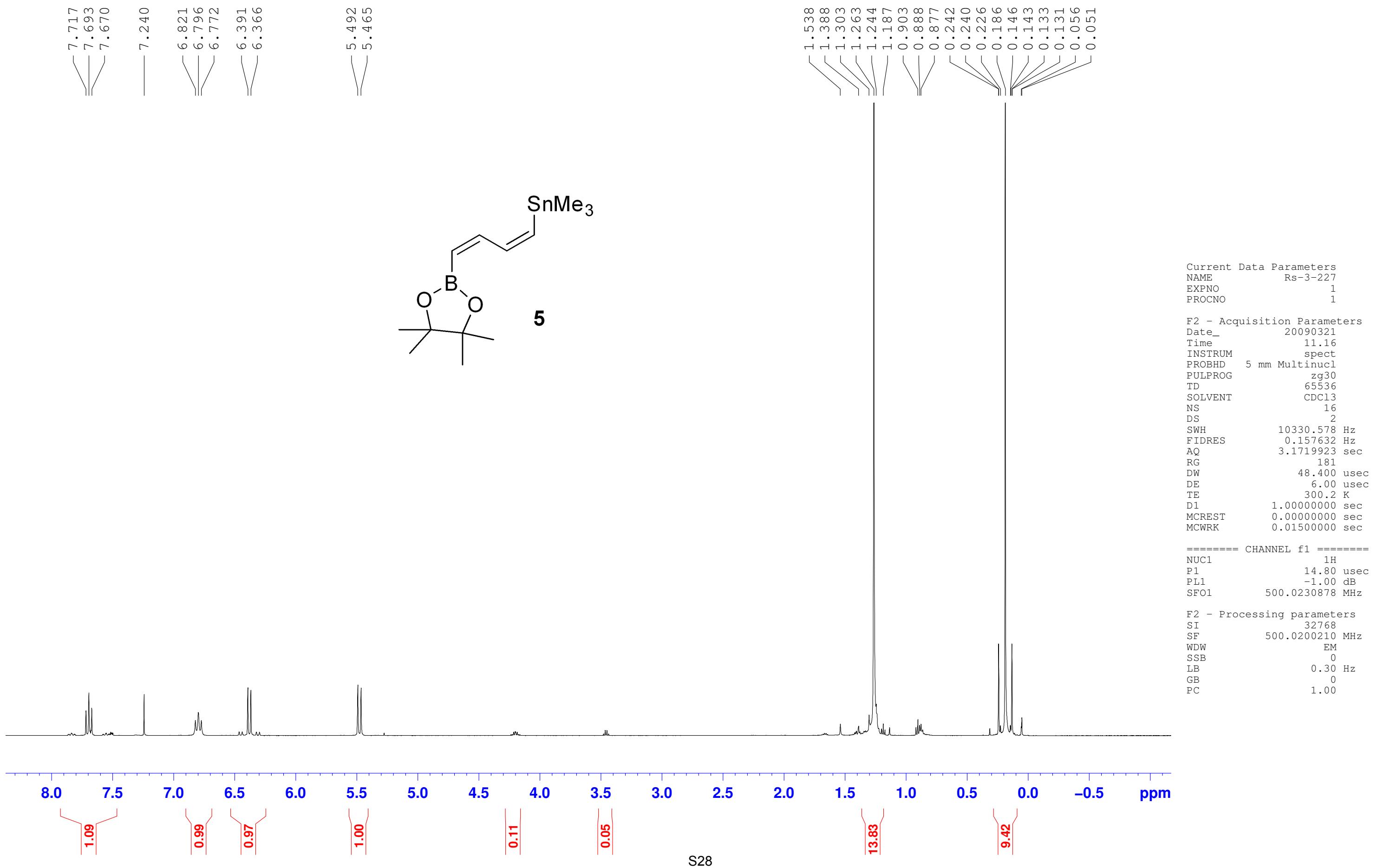
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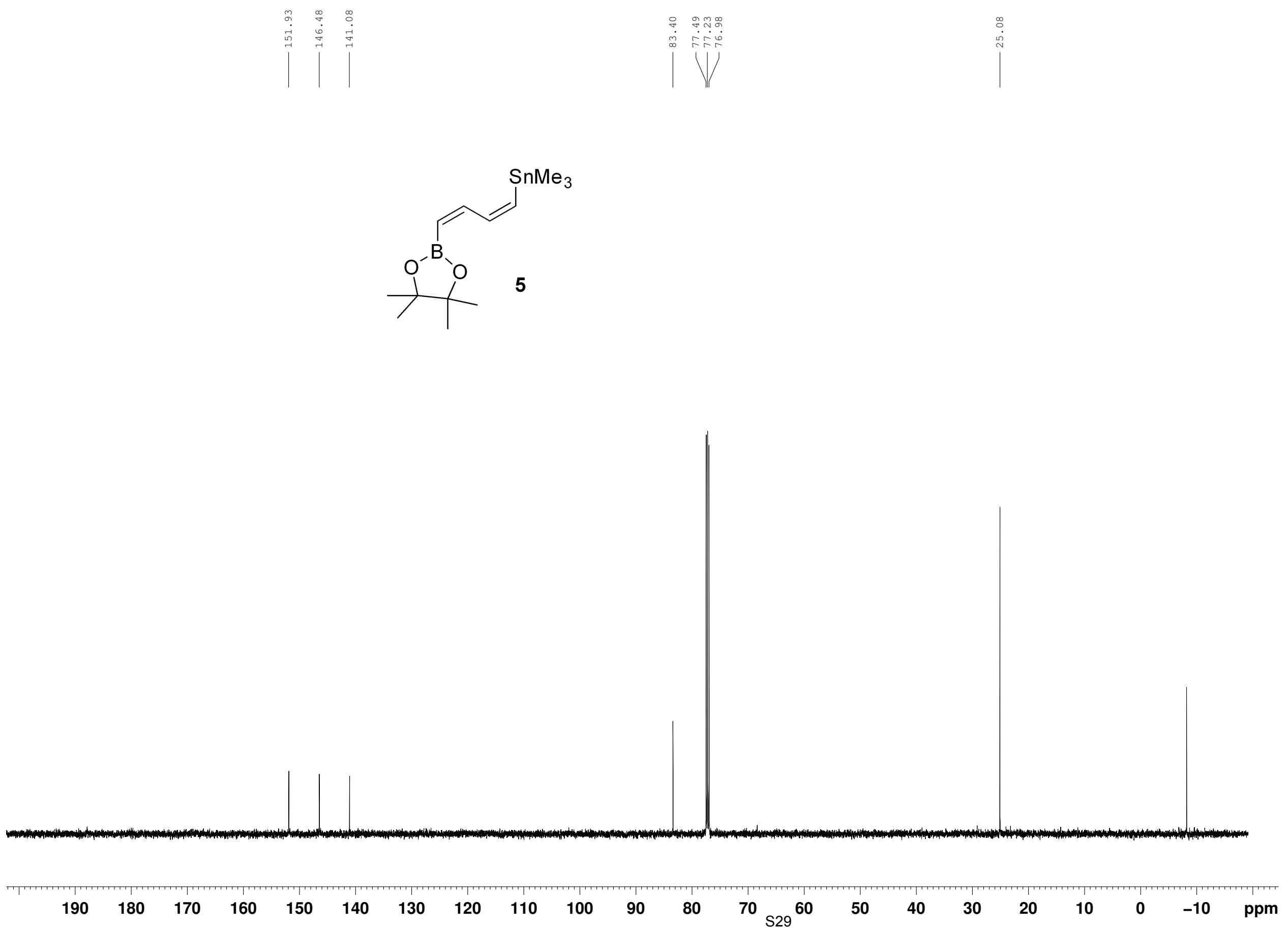
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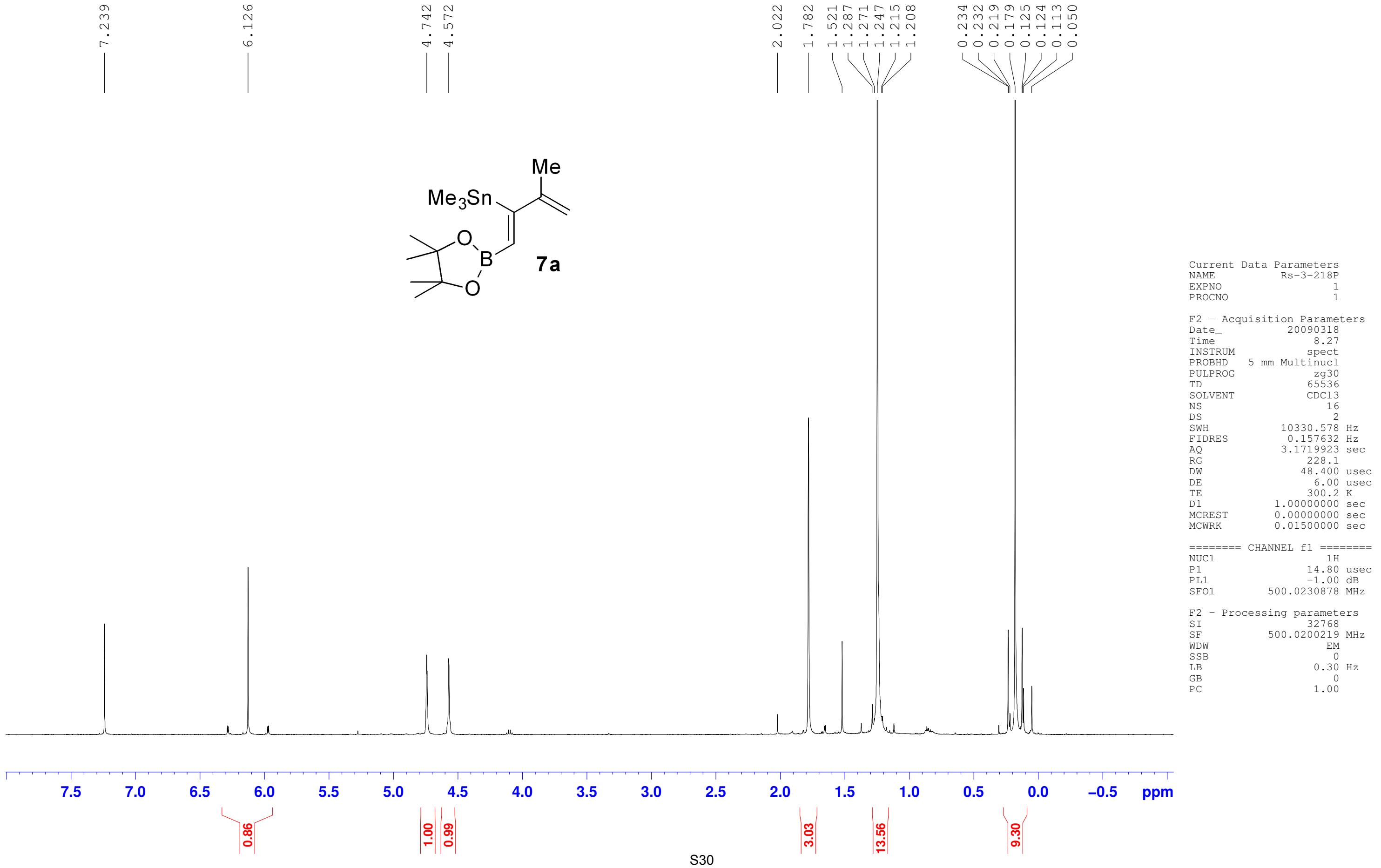
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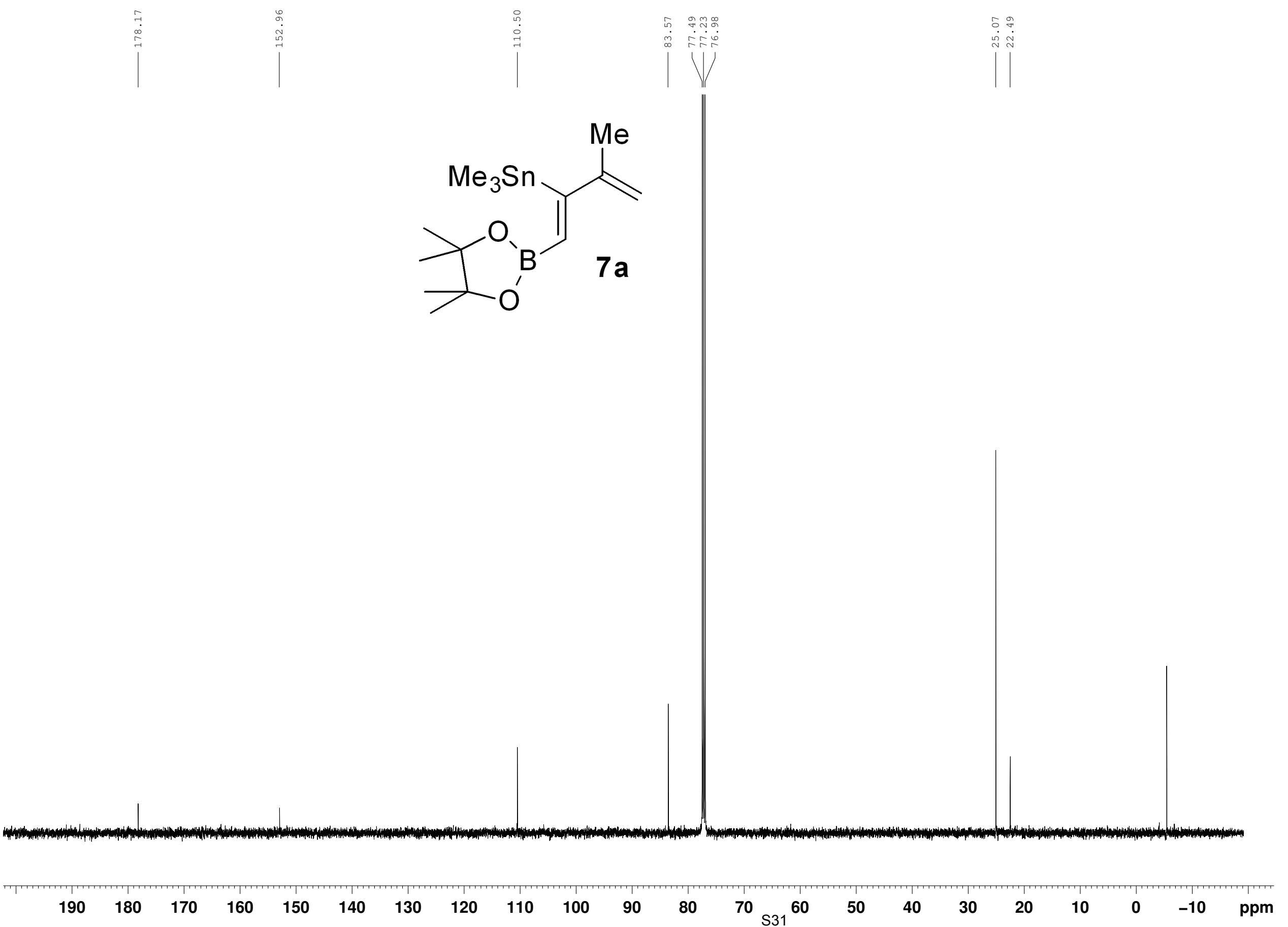
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===== CHANNEL f1 =====
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 PL1 3.00 dB
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===== CHANNEL f2 =====
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 PL2 -1.00 dB
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 PL13 22.50 dB
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F2 - Processing parameters
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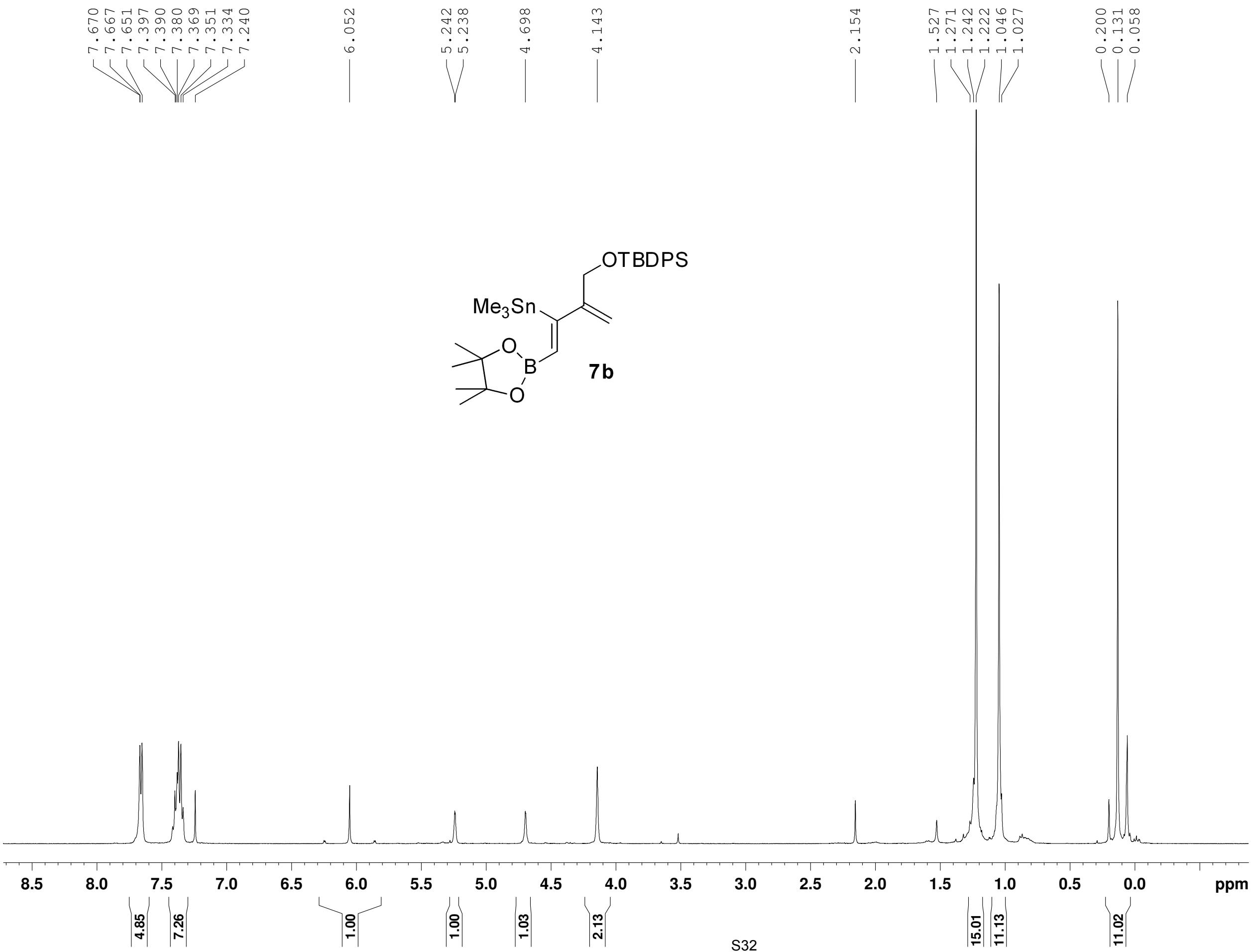
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 SOLVENT CDCl₃
 NS 571
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 2580.3
 DW 16.650 usec
 DE 12.00 usec
 TE 300.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999999 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 12.00 usec
 PL1 3.00 dB
 SFO1 125.7427020 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ^{1H}
 PCPD2 100.00 usec
 PL2 -1.00 dB
 PL12 15.59 dB
 PL13 22.50 dB
 SFO2 500.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7301005 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



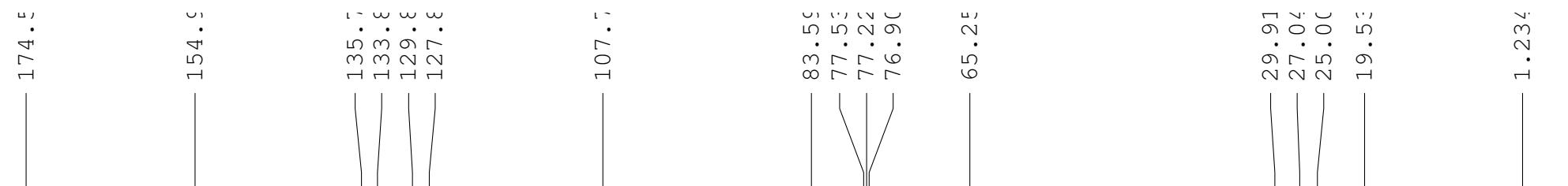
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Current Data Parameters
NAME Rs-3-248P
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090417
Time 13.38
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 143.7
DW 60.400 use
DE 6.00 use
TE 299.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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Current Data Parameters
NAME Rs-3-248P
EXPNO 2
PROCNO 1

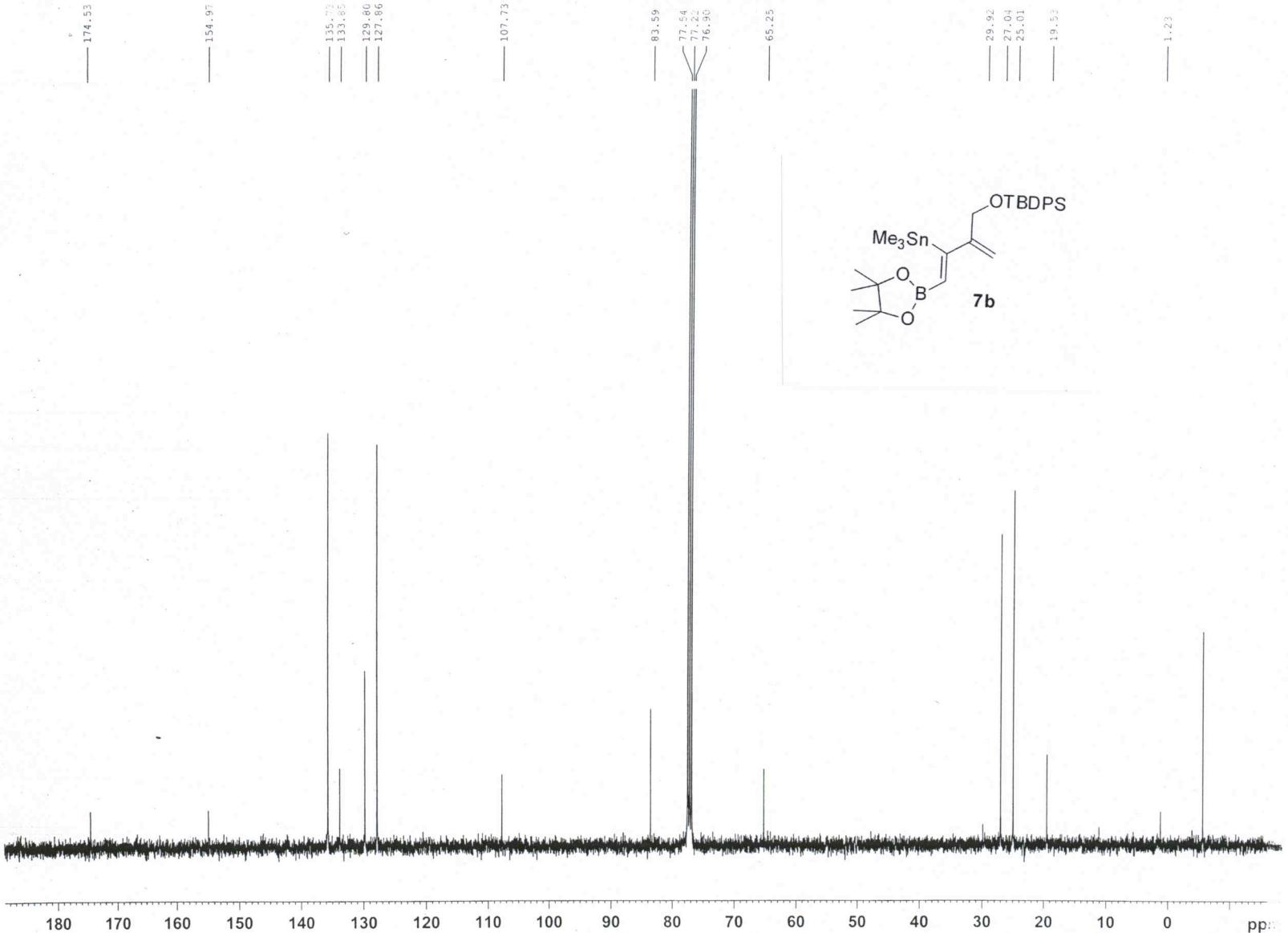
F2 - Acquisition Parameters
Date_ 20090417
Time 13.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 428
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 14596.5
DW 20.850 use
DE 6.00 use
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999999 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 use
PL1 0.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127476 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm



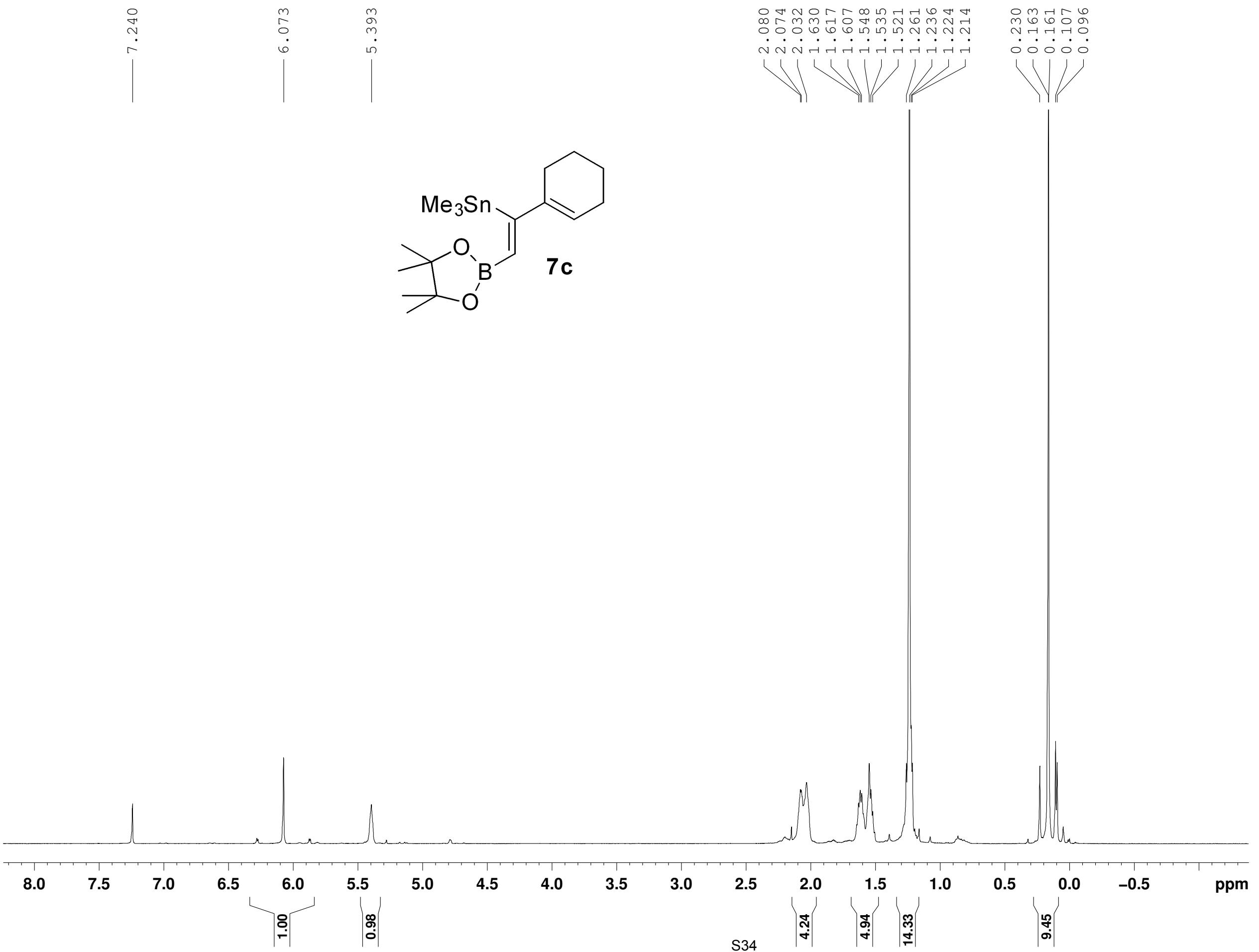
Current Data Parameters
NAME Rs-3-248P
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20090417
Time 13.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 428
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 14596.5
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.8999998 sec
MCREST 0.0000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127483 MHz
WDW EM
SSB 0
LB 1.00 Hz
TP 0
TIME 1.40



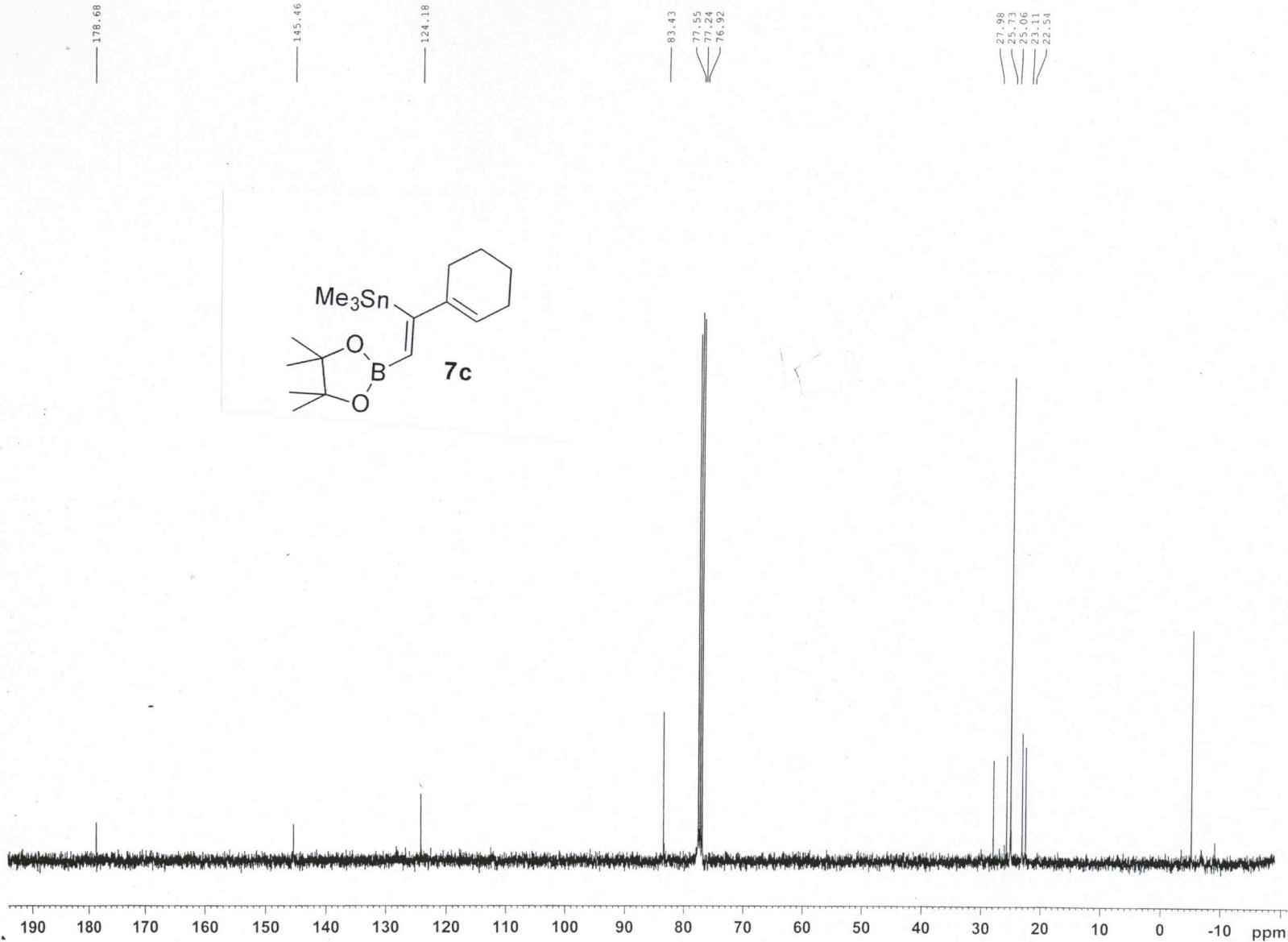
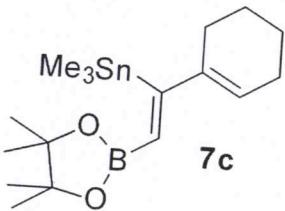
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Current Data Parameters
NAME Rs-3-261P
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20090429
Time 14.37
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 80.6
DW 60.400 use
DE 6.00 use
TE 299.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SF01 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



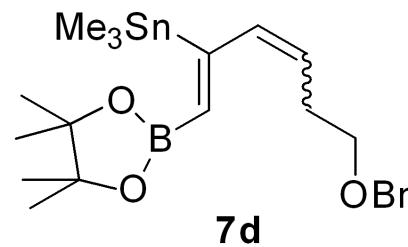
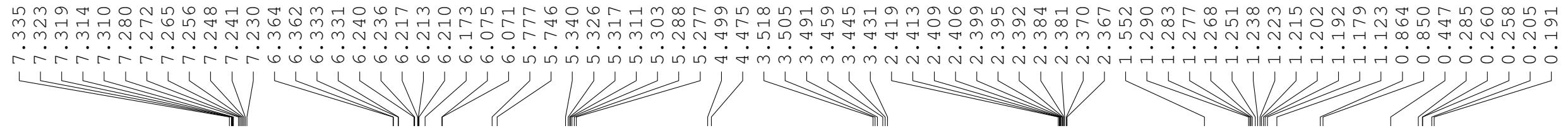
Current Data Parameters
 NAME Rs-3-261P
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090429
 Time 14.40
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 210
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 5160.6
 DW 20.850 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.8999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 14.56 dB
 PL13 16.50 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127469 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

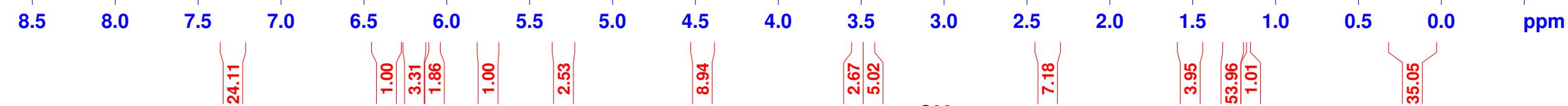


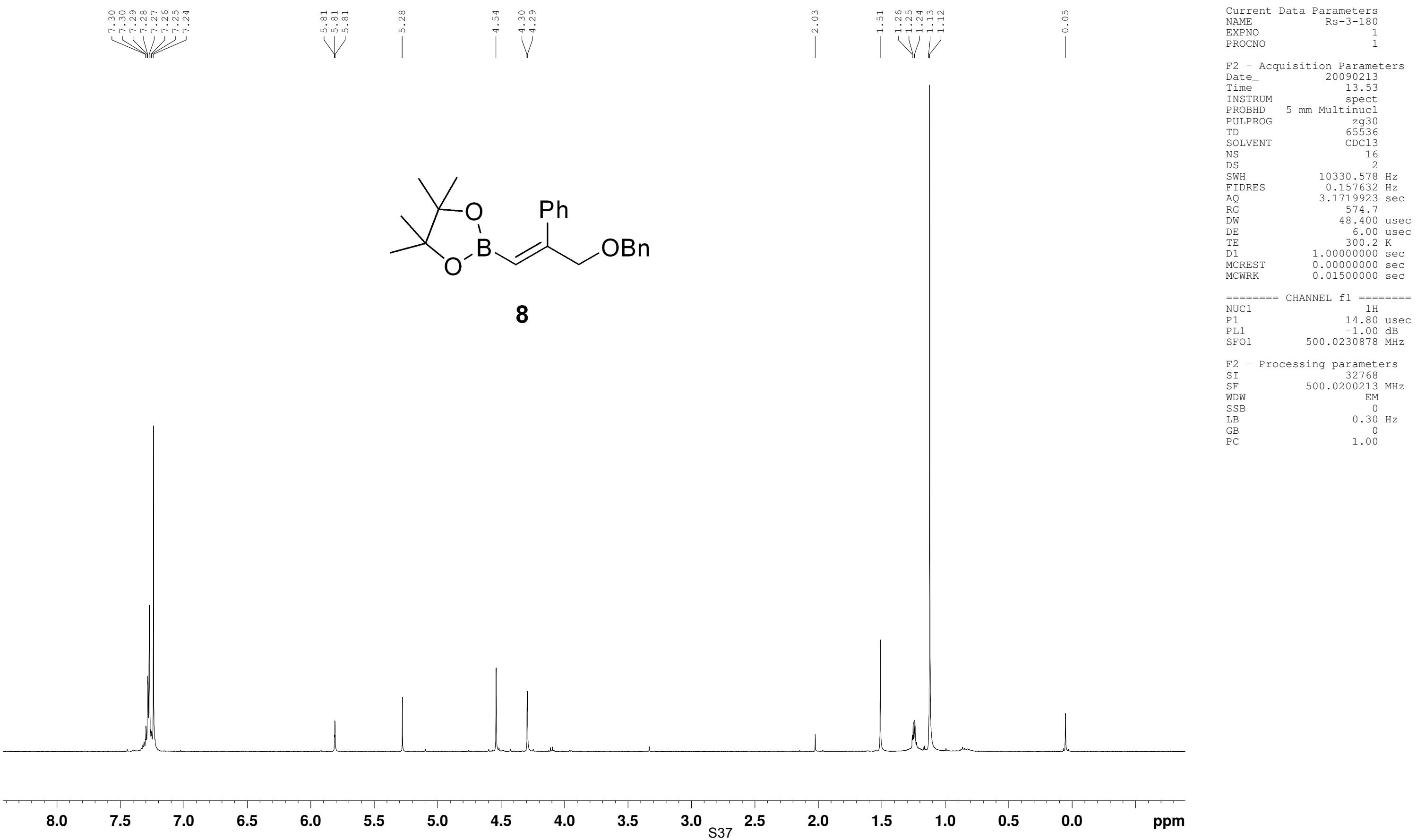
Current Data Parameters
NAME Rs-3-249
EXPNO 3
PROCNO 1

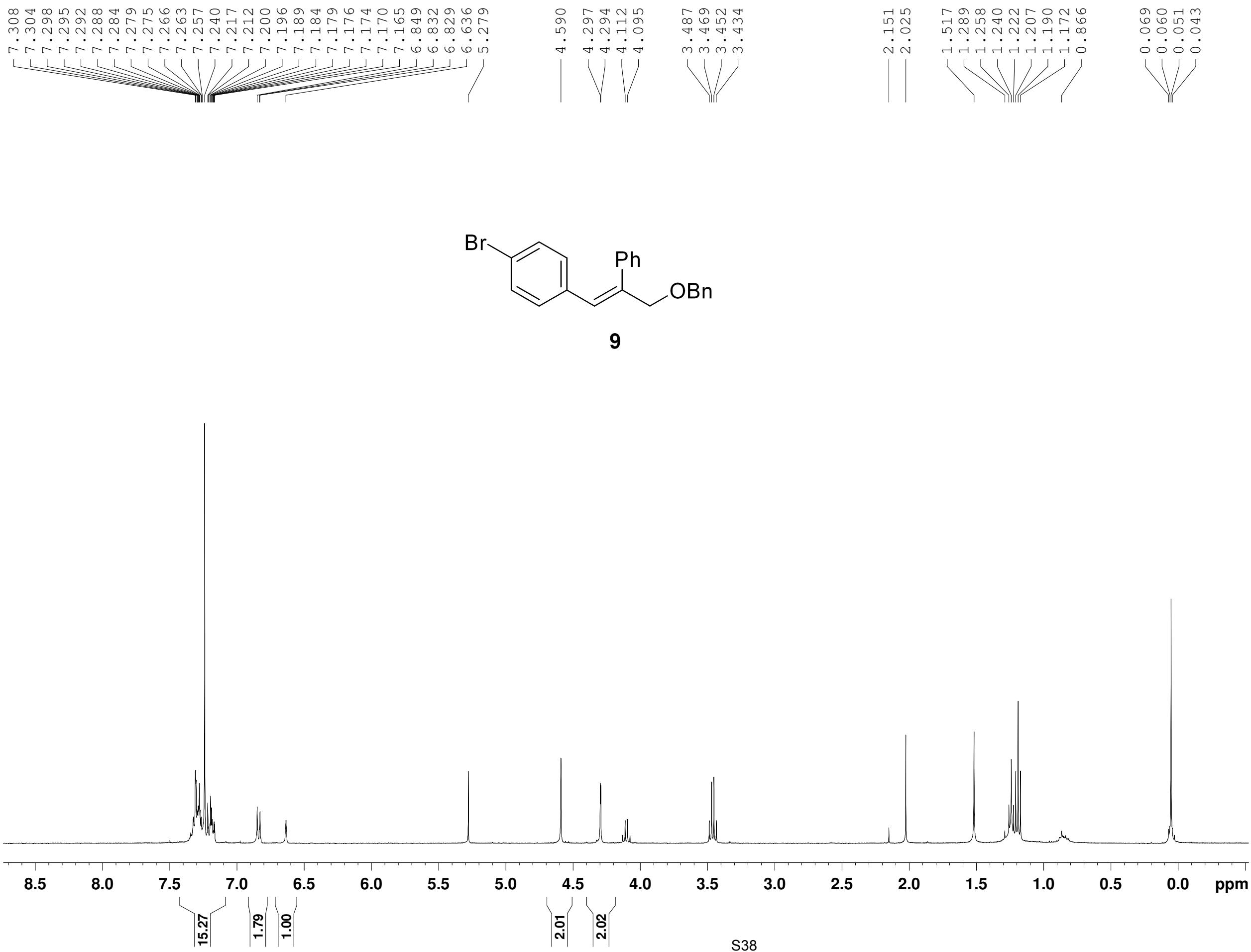
F2 - Acquisition Parameters
Date_ 20100401
Time 11.21
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 14.80 usec
PL1 -1.00 dB
SFO1 500.0230878 MHz

F2 - Processing parameters
SI 32768
SF 500.0200213 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







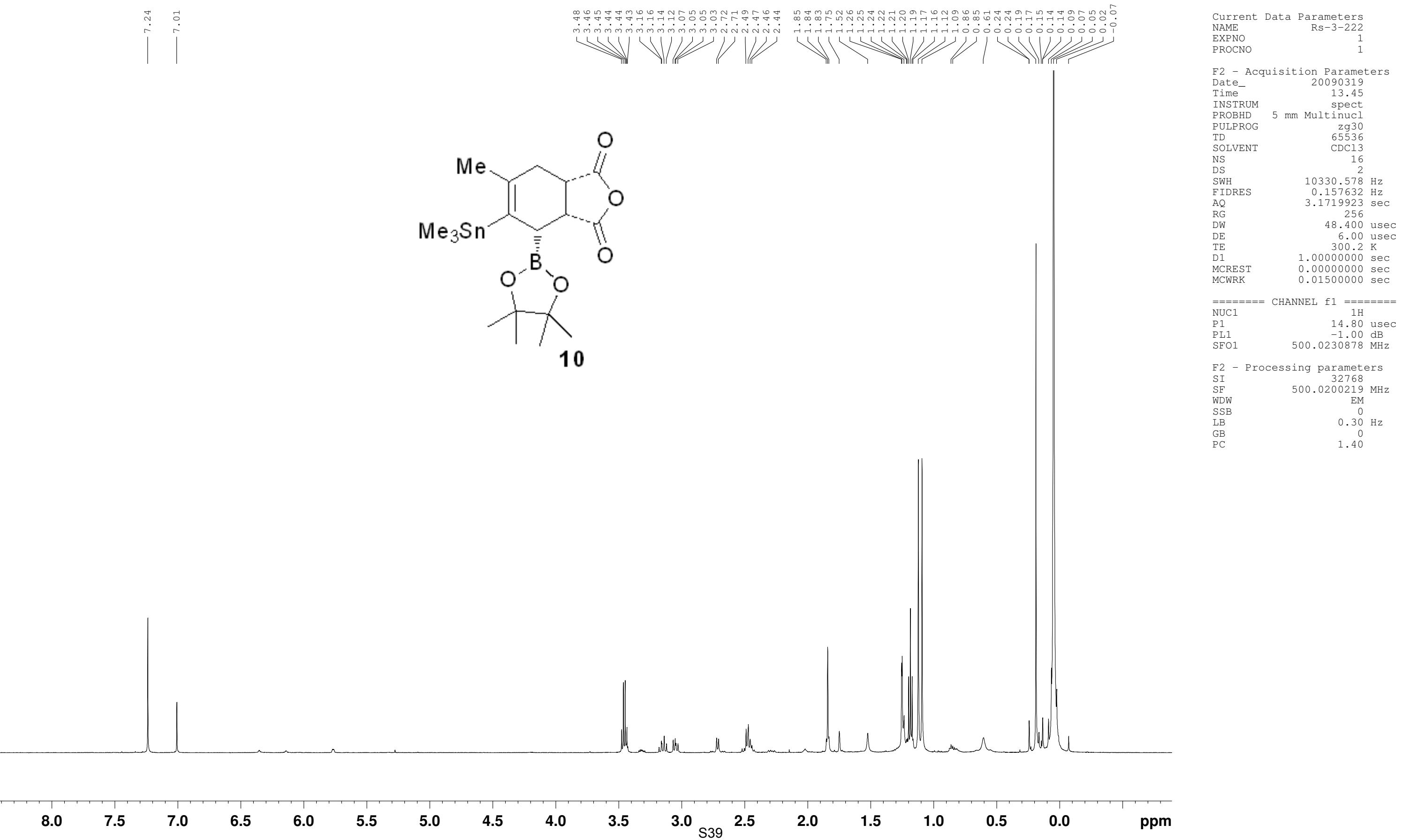
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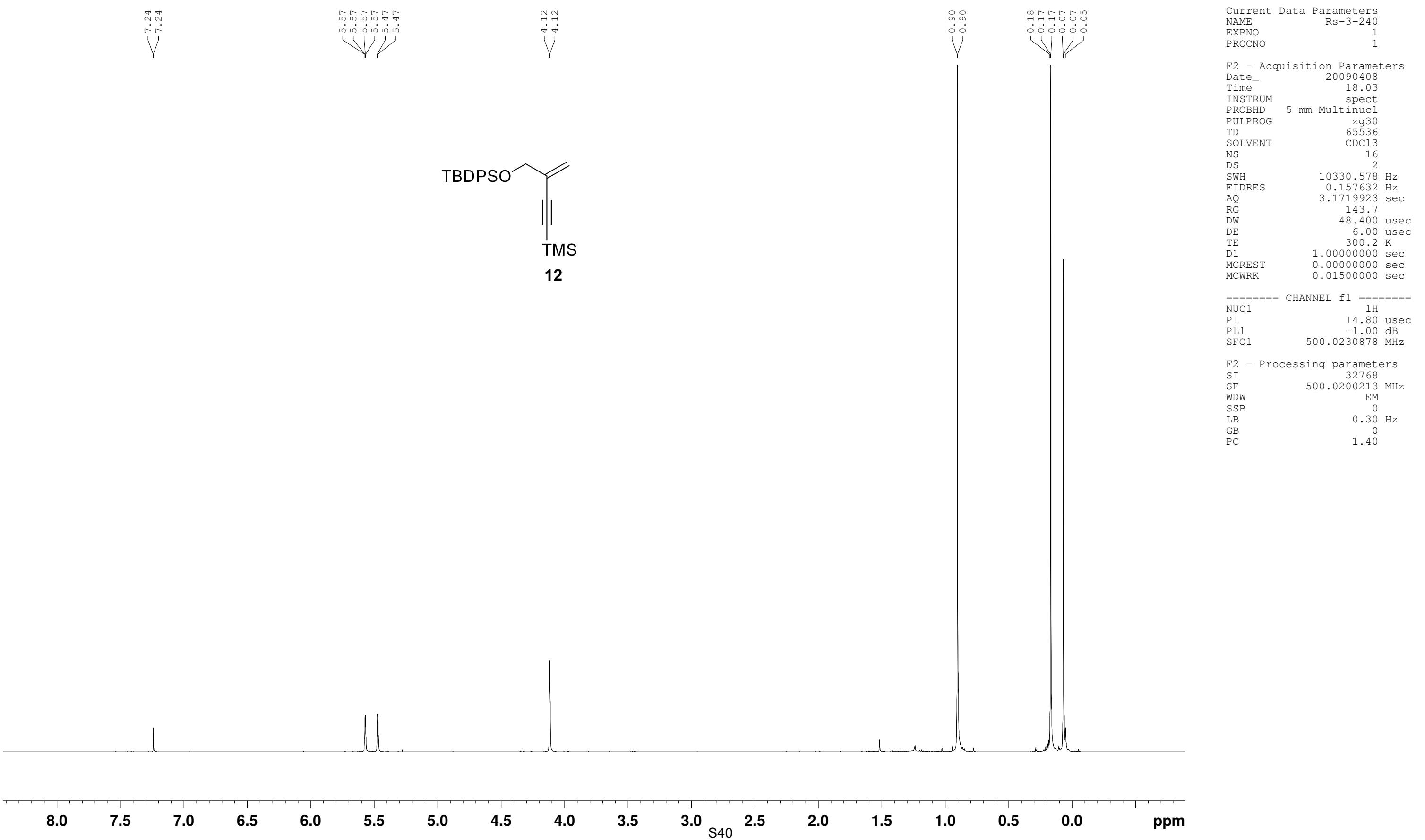
Current Data Parameters
NAME Rs-3-181-LP
EXPNO 1
PROCNO 1

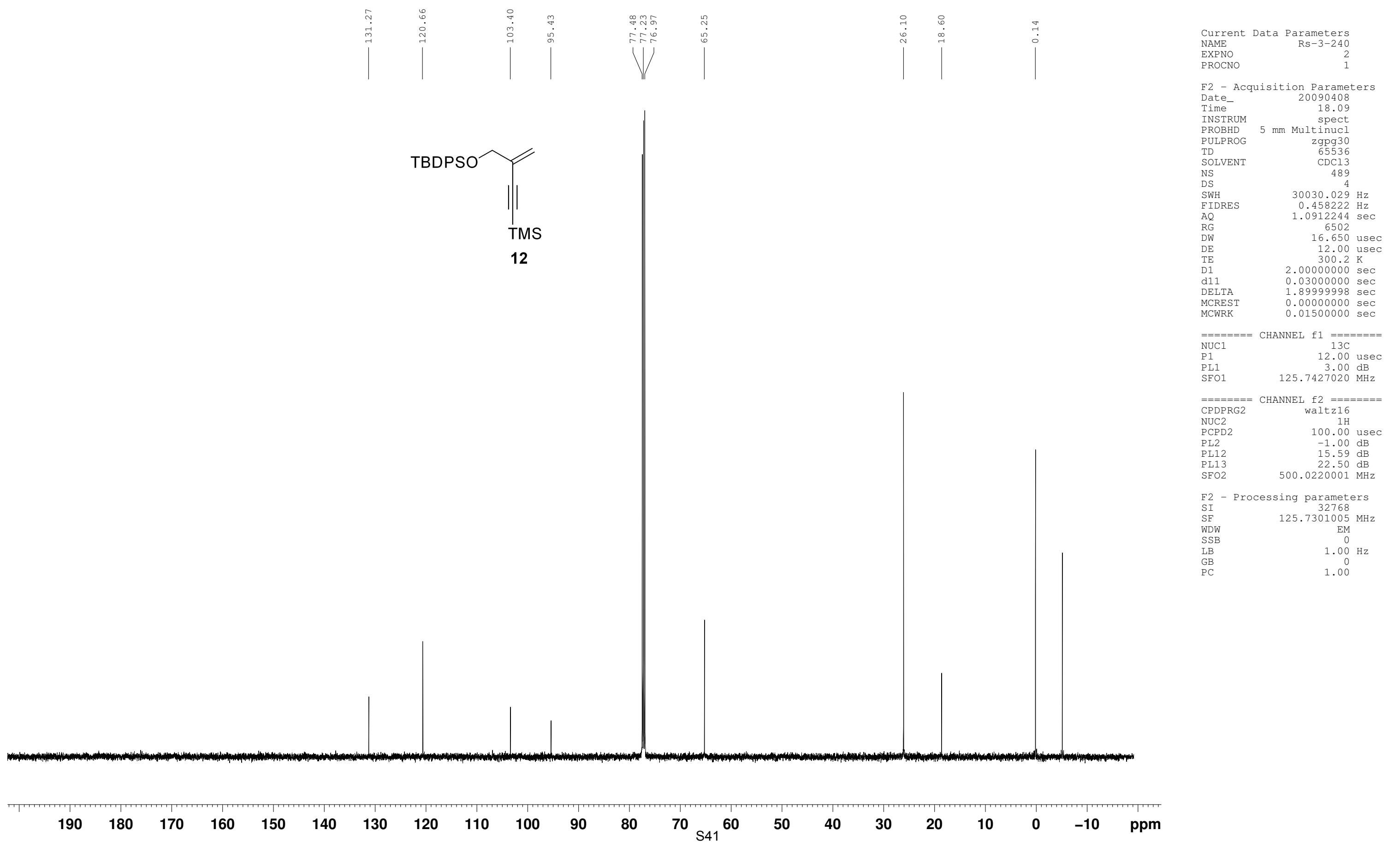
F2 – Acquisition Parameters
Date_ 20090214
Time 11.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 use
DE 6.00 use
TE 300.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

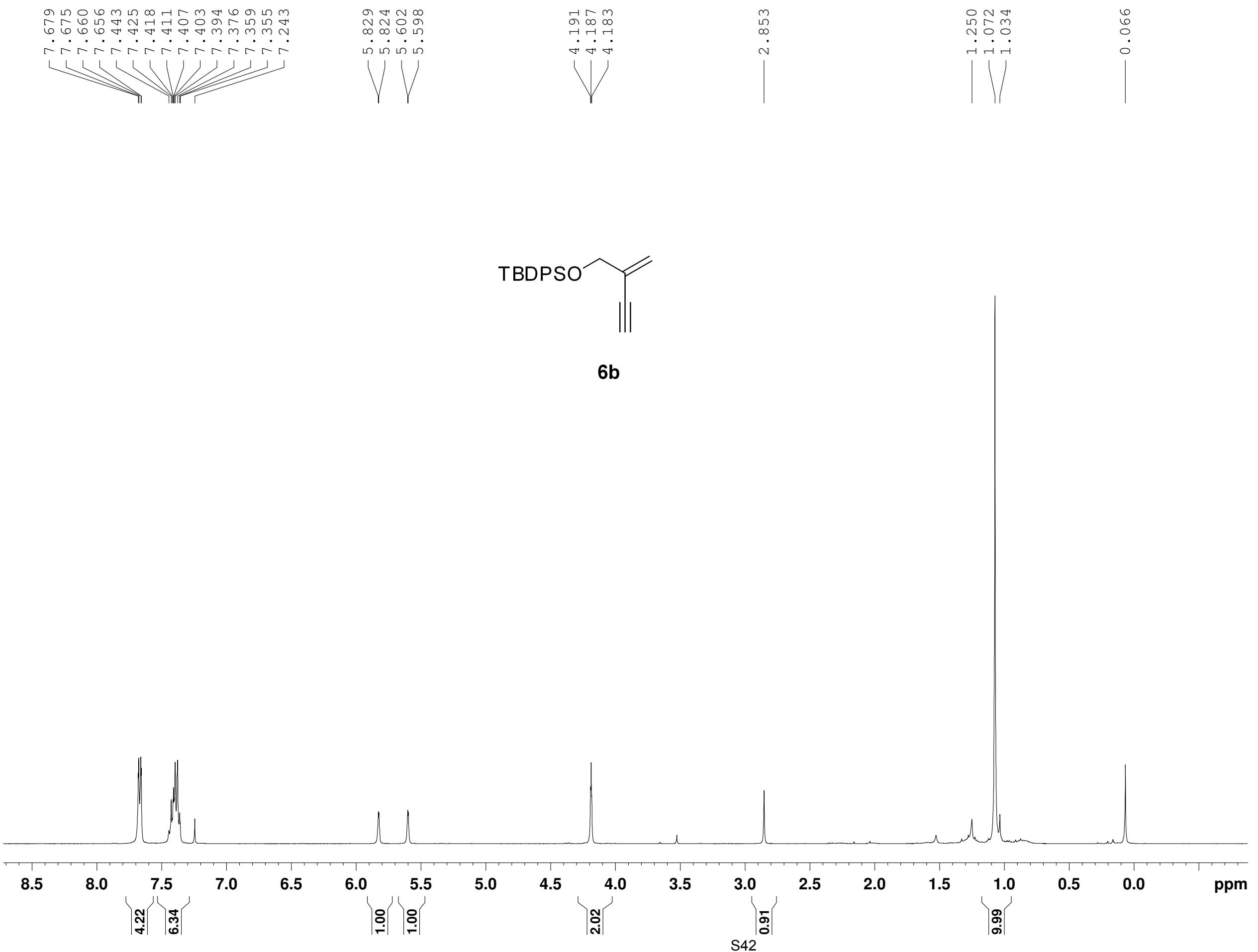
===== CHANNEL f1 =====
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SF01 400.1324710 MHz

F2 – Processing parameters
SI 32768
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

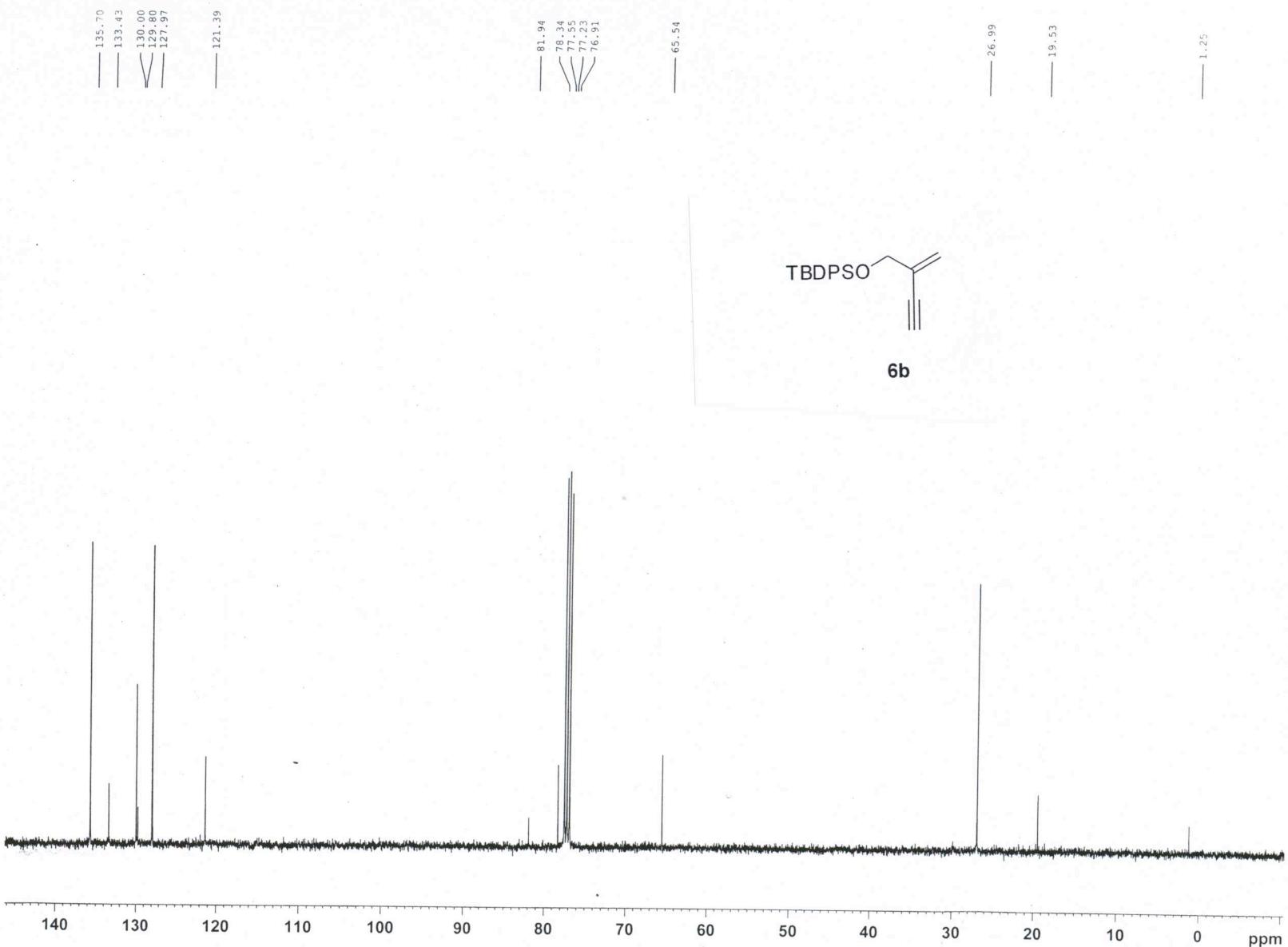


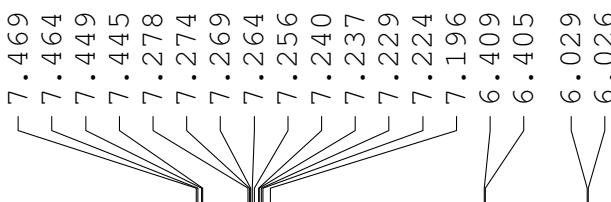






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Current Data Parameters
NAME Rs-3-251
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090421
Time 11.42
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 203.2
DW 60.400 use
DE 6.00 use
TE 298.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300351 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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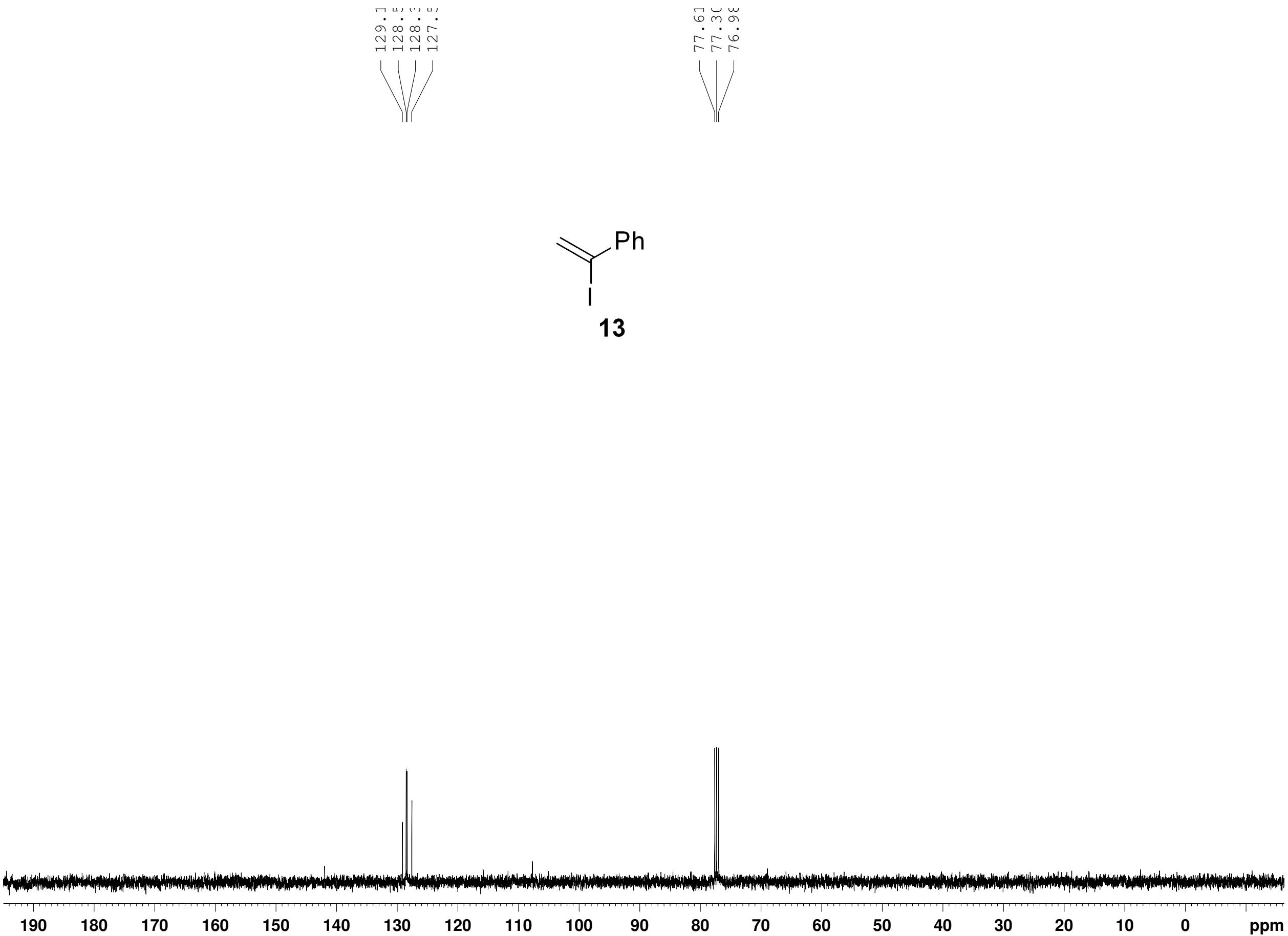
Current Data Parameters
NAME Rs-3-251
EXPNO 4
PROCNO 1

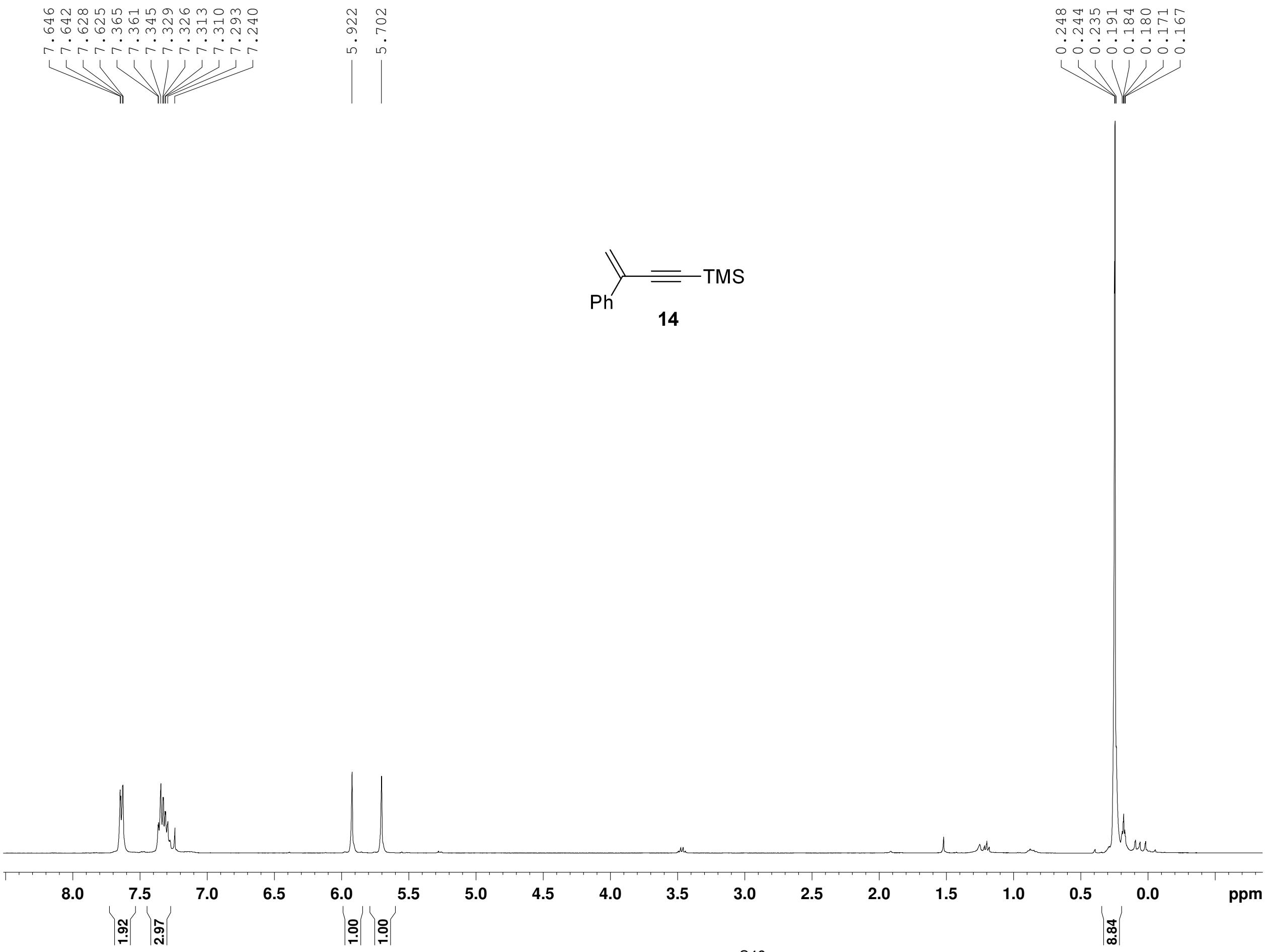
F2 – Acquisition Parameters
Date_ 20090421
Time 11.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 20
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 use
DE 6.00 use
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 use
PL1 0.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 – Processing parameters
SI 32768
SF 100.6127413 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





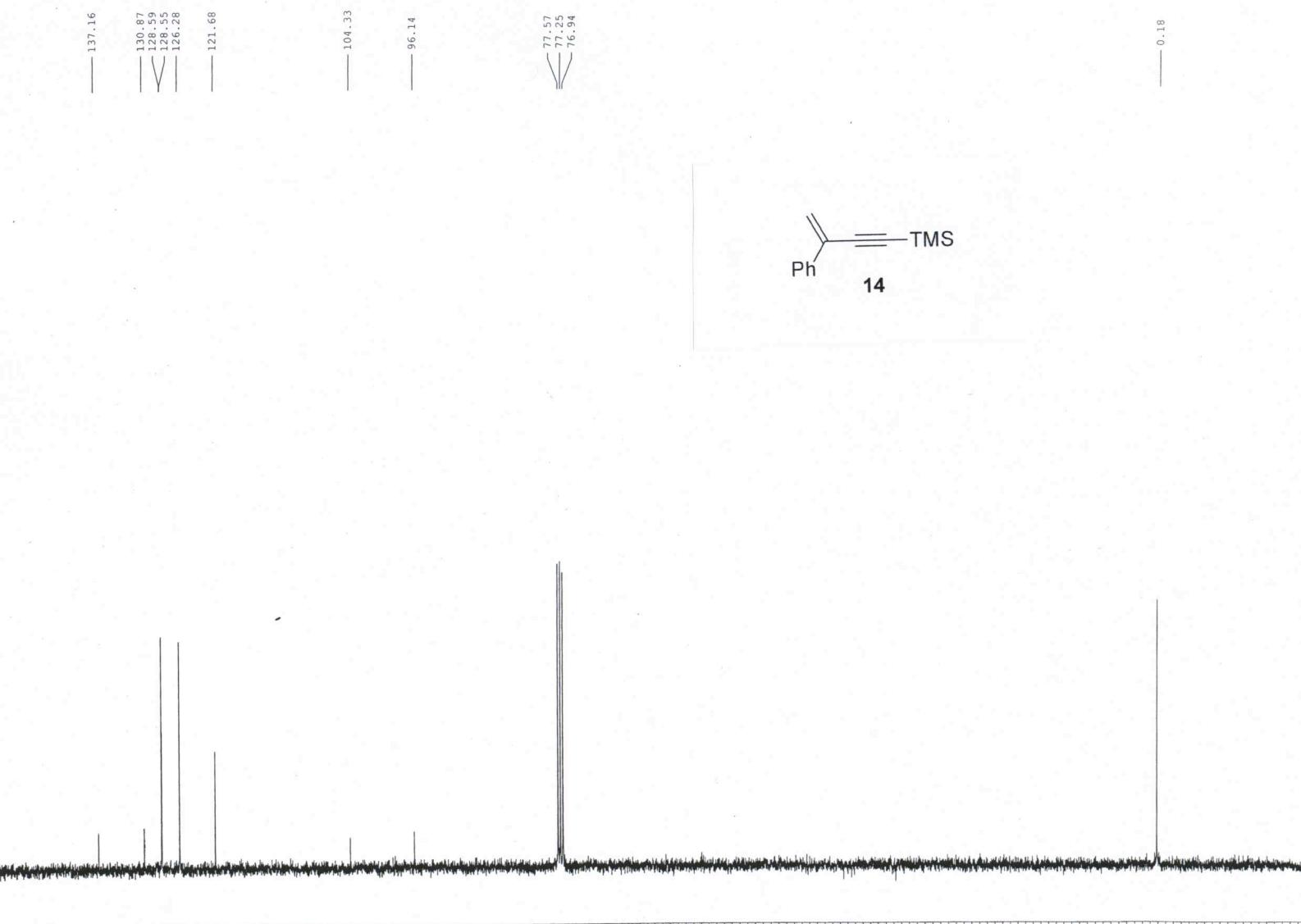
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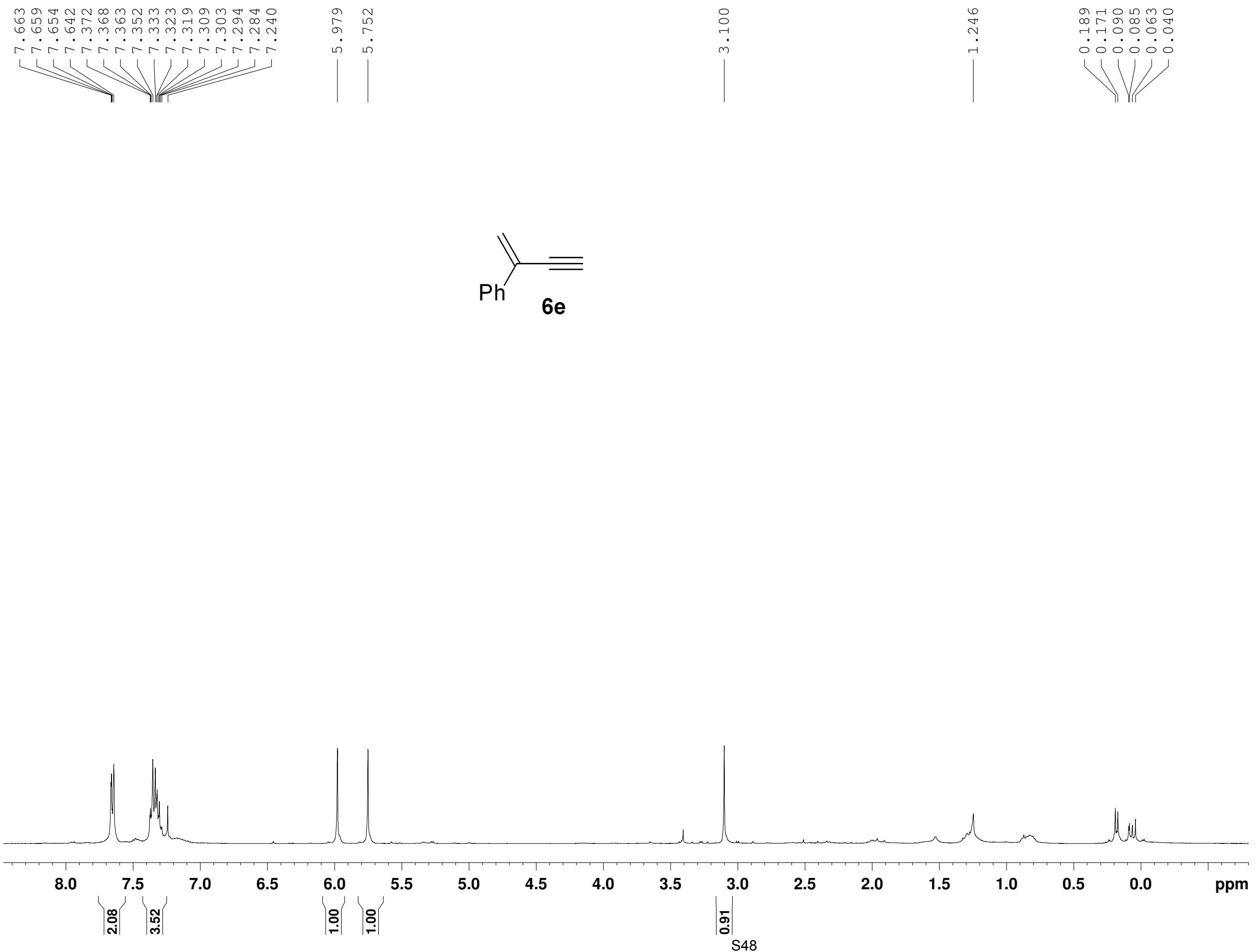
Current Data Parameters
NAME Rs-3-253
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090422
Time 9.09
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 114
DW 60.400 use
DE 6.00 use
TE 298.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





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Current Data Parameters
NAME Rs-3-254
EXPNO 1
PROCNO 1

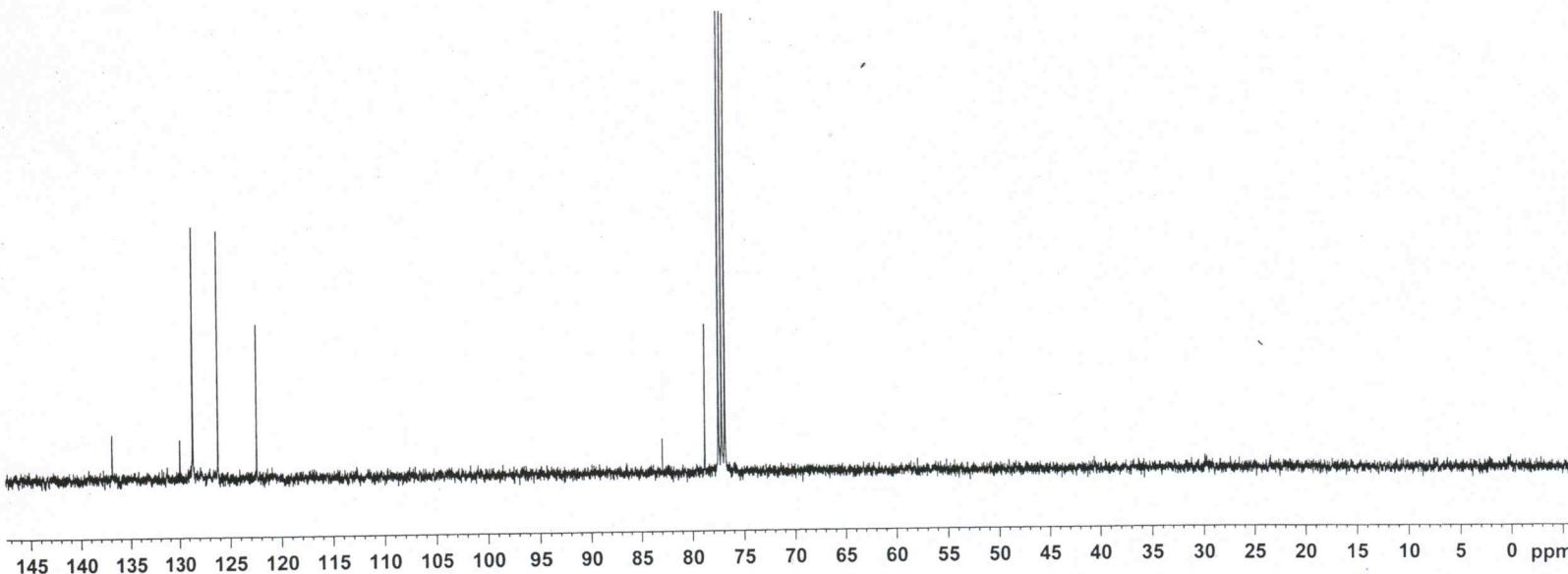
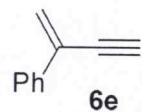
F2 - Acquisition Parameters
Date_ 20090422
Time 14.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 use
DE 6.00 use
TE 298.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

136.854
129.966
128.665
128.622
126.199
122.451

82.982
78.860
77.550
77.232
76.915



Current Data Parameters
NAME Rs-3-254
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090422
Time 14.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 245
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127483 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00