

Supporting Information

Copper-Catalyzed γ -Selective Allyl–Alkyl Coupling between Allylic Phosphates and Alkylboranes

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Instrumentation and Chemical

NMR spectra were recorded on a Varian Gemini 2000 spectrometer, operating at 300 MHz for ^1H NMR and 75.4 MHz for ^{13}C NMR, and a JEOL ECA-600, operating at 600 MHz for ^1H NMR, 150.9 MHz for ^{13}C NMR and 192.6 MHz for ^{11}B NMR. Chemical shift values for ^1H and ^{13}C are referenced to Me_4Si and the residual solvent resonances, respectively. Chemical shift values for ^{11}B is referenced to $\text{BF}_3\cdot\text{OEt}$ (δ 0 ppm). Chemical shifts are reported in δ ppm. IR spectra were recorded on a Perkin-Elmer Spectrum One. Mass spectra were obtained with Thermo Fisher Scientific Exactive, JEOL JMS-T100 GC or JEOL JMS-T100LP at the Center for Instrumental Analysis, Hokkaido University. Elemental analysis was performed at the Center for Instrument Analysis of Hokkaido University. TLC analyses were performed on commercial glass plates bearing 0.25-mm layer of Merck Silica gel 60F₂₅₄. Silica gel (Kanto Chemical Co., Silica gel 60 N, spherical, neutral) and aluminum oxide (Nacalai Tesuque, Alumina Activated 200) were used for column chromatography. HPLC analyses were conducted on a HITACHI ELITE LaChrom system with a HITACHI L-2400 UV detector or a Shimadzu LC-6A system with a Shimadzu SPD-10A UV detector. Gas chromatographic (GLC) analyses were conducted on a Shimadzu GC-14B equipped with a flame ionization detector. Gel permeation chromatography (GPC) was performed by LC-908 (Japan Analytical Industry Ltd., two in-line JAIGEL-2H, CHCl_3 , 3.5 mL/min, UV and RI

detectors).

All reactions were carried out under nitrogen or argon atmosphere. Materials were obtained from commercial suppliers or prepared according to standard procedures unless otherwise noted. *t*-BuOK (1.0 M THF solution) and CuOAc were purchased from Aldrich Chemical Co., stored under nitrogen, and used as it is. THF was purchased from Kanto Chemical Co., stored under argon. Alkenes **1a–g** was well known compounds. 20(21)-Methylene steroid **1n** was prepared from pregnenone acetate according to the reported procedure.¹

Preparation of Allylic Phosphates

Preparation of Allylic Phosphates 3a, a' and 3c–e. Allylic substrates were prepared by the phosphorylation of the corresponding allylic alcohols. The allylic alcohols were prepared by the reduction of the corresponding propargylic alcohols by $\text{Cp}_2\text{TiCl}_2/i\text{-BuMgBr}$ according to the reported procedure.² The obtained crude allylic alcohols were purified by silica gel chromatography and GPC (CHCl_3).

The preparation of **3a** is representative. To a solution of (*Z*)-1-phenyl-4-nonen-3-ol (437.7 mg, 2 mmol) in pyridine (2.2 mL, 5 mL per 1 gram of alcohol), $(\text{EtO})_2\text{P}(\text{O})\text{Cl}$ (405 μL , 2.8 mmol) and DMAP (61.1 mg, 0.5 mmol) were sequentially added at 0 °C. After being stirred at rt for 2 h, the reaction mixture was diluted with EtOAc (66 mL) and quenched with H_2O (5 mL). The resulting mixture was washed with sat. CuSO_4 (10 mL $\times$ 3) and brine, and was dried over anhydrous MgSO_4 , filtered, and evaporated under reduced pressure. The residue was purified through a short plug of aluminum oxide (ether) to provide **3a** in 86% yield (612.8 mg, 1.7 mmol).

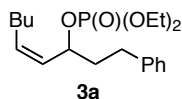
Preparation of Allylic Phosphates 3b, 3f and 3g. Allylic phosphate **3b** was synthesized as follows: 2,3-*O*-isopropylidene-glyceraldehyde was subjected to *Z*-selective Horner-Wadsworth-Emmons-type reaction with pentyltriphenylphosphonium bromide in the presence of NaHMDS to give the corresponding *Z*-alkene. Next, deprotection of the acetal, silylation and phosphorylation produced **3b**. Allylic substrates **3f** and **3g** were obtained by the phosphorylation of the corresponding commercial available alcohols.

Preparation of (*S*, *Z*)-Diethyl 3-Octen-2-yl Phosphate (3h). (*S*)-3-Octyn-2-ol was prepared by the asymmetric reduction of 3-octyn-2-one according to the reported procedure.³ (*S*)-3-Octyn-2-ol was reduced with $\text{Cp}_2\text{TiCl}_2/i\text{-BuMgBr}$ according to the reported procedure,² producing (*S*, *Z*)-3-octen-2-ol, which then was converted to the corresponding allylic phosphate (*S*)-(Z)-**3h**. The ee value of (*S*)-(Z)-**3h** (95% ee) was determined by HPLC analysis of the *p*-nitrobenzoate derivative of (*S*, *Z*)-3-octen-2-ol (CHIRALCEL[®] OD-3 column, 4.6 mm $\times$ 250 mm, Daicel Chemical Industries, hexane, 0.5 mL/min, 40 °C, 254 nm UV detector, retention time = 32.8 min for the *R* isomer and 34.3 min for the *S* isomer). The absolute configuration of (*S*)-(Z)-**3h** was determined by optical rotations of the precursor compounds, 3-octyn-2-ol.³

Preparation of Cyclic Allylic Phosphates. Allylic substrates **3i** and **3k–m** were prepared by the phosphorylation of the corresponding known allylic alcohols. Allylic phosphate (1*R*,4*S*)-**3j** was synthesized from commercial available (1*R*,3*S*)-*cis*-4-cyclopentene-1,3-diol 1-acetate by silylation, deacetylation and phosphorylation.

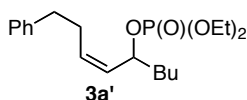
Characterization Data for Allylic Phosphates

(*Z*)-Diethyl 1-Phenyl-4-nonen-3-yl Phosphate (**3a**)



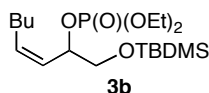
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.88 (t, $J = 7.2$ Hz, 3H), 1.28–1.35 (m, 10H), 1.80–1.89 (m, 1H), 2.06–2.16 (m, 3H), 2.65–2.71 (m, 2H), 4.02–4.15 (m, 4H), 5.15 (dq, $J = 9.0, 6.9$ Hz, 1H), 5.46 (dd, $J = 11.1, 9.0$ Hz, 1H), 5.59 (dt, $J = 11.1, 7.5$ Hz, 1H), 7.18–7.21 (m, 3H), 7.26–7.31 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.77, 15.90 (d, $J = 4.0$ Hz), 16.00 (d, $J = 3.4$ Hz), 22.18, 27.38, 31.03, 31.48, 38.06 (d, $J = 6.9$ Hz), 63.40 (d, $J = 5.7$ Hz), 63.42 (d, $J = 5.7$ Hz), 74.28 (d, $J = 6.3$ Hz), 125.99, 128.30 (d, $J = 3.4$ Hz), 128.40, 128.45, 134.18, 141.44. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{31}\text{O}_4\text{PNa}$, 377.18522; found, 377.18552.

(*Z*)-Diethyl 1-Phenyl-3-nonen-5-yl Phosphate (**3a'**)



Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.87 (t, $J = 6.9$ Hz, 3H), 1.17–1.43 (m, 11H), 1.64 (m, 1H), 2.40–2.52 (m, 2H), 2.56–2.81 (m, 2H), 3.98–4.14 (m, 4H), 5.03 (dq, $J = 9.3, 6.6$ Hz, 1H), 5.41 (dd, $J = 11.1, 9.3$ Hz, 1H), 5.59 (dt, $J = 11.1, 7.5$ Hz, 1H), 7.16–7.21 (m, 3H), 7.22–7.31 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.85, 15.93 (d, $J = 2.9$ Hz), 16.30 (d, $J = 2.9$ Hz), 22.31, 26.80, 29.52, 35.50, 35.81 (d, $J = 6.3$ Hz), 63.34 (d, $J = 5.7$ Hz), 63.36 (d, $J = 5.7$ Hz), 74.73 (d, $J = 6.3$ Hz), 126.02, 128.41, 128.59, 129.50 (d, $J = 2.9$ Hz), 132.42, 141.47. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{31}\text{O}_4\text{PNa}$, 377.1858; found, 377.1862.

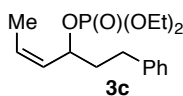
(*Z*)-1-(*tert*-Butyldimethylsiloxy)-3-octen-2-yl Diethyl Phosphate (**3b**)



Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.06 (s, 6H), 0.88–0.92 (m, 12H), 1.28–1.40 (m, 10H), 2.12–2.16 (m, 2H), 3.61 (ddd, $J = 10.8, 5.1, 1.8$ Hz, 1H), 3.73 (dd, $J = 10.8, 6.3$ Hz, 1H), 4.02–4.17 (m, 4H), 5.12 (m, 1H), 5.40 (ddt, $J = 10.8, 9.3, 1.2$ Hz, 1H), 5.64 (dtd, $J = 10.8, 7.5, 1.2$ Hz, 1H). ^{13}C NMR (75.4 MHz, CDCl_3) δ –5.56, 13.81, 15.91, 16.01, 18.25, 22.23, 25.75, 27.58, 31.57, 63.38

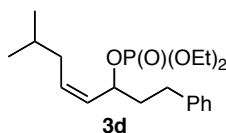
(d, $J = 5.6$ Hz), 63.42 (d, $J = 5.6$ Hz), 65.85 (d, $J = 7.4$ Hz), 74.96 (d, $J = 5.6$ Hz), 125.67 (d, $J = 2.8$ Hz), 135.60. HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{18}H_{39}O_5PSiNa$, 417.22021; found, 417.22031.

(Z)-Diethyl 6-Phenyl-2-hexen-4-yl Phosphate (3c)



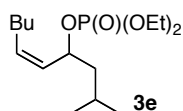
Oil. 1H NMR (300 MHz, $CDCl_3$) δ 1.28 (td, $J = 6.6, 0.9$ Hz, 3H), 1.30 (td, $J = 6.6, 0.9$ Hz, 3H), 1.70 (dd, $J = 6.9, 1.5$ Hz, 3H), 1.82 (m, 1H), 2.10 (m, 1H), 2.63–2.72 (m, 2H), 4.02–4.15 (m, 4H), 5.17 (m, 1H), 5.50 (ddq, $J = 13.2, 9.0, 1.5$ Hz, 1H), 5.70 (dq, $J = 13.2, 6.9$ Hz, 1H), 7.17–7.21 (m, 3H), 7.27–7.31 (m, 2H). ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 13.30, 15.92 (d, $J = 3.4$ Hz), 16.01 (d, $J = 3.4$ Hz), 31.00, 37.81 (d, $J = 6.9$ Hz), 63.43 (d, $J = 6.3$ Hz), 63.45 (d, $J = 6.3$ Hz), 73.89 (d, $J = 6.3$ Hz), 126.01, 128.42, 128.45, 128.48, 129.40 (d, $J = 3.4$ Hz), 141.41. HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{16}H_{25}O_4PNa$, 335.13881; found, 335.13924.

(Z)-Diethyl 7-Methyl-1-phenyl-4-octen-3-yl Phosphate (3d)



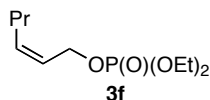
Oil. 1H NMR (300 MHz, $CDCl_3$) δ 0.87 (d, $J = 6.9$ Hz, 3H), 0.90 (d, $J = 6.9$ Hz, 3H), 1.30 (dt, $J = 6.9, 1.2$ Hz, 3H), 1.33 (dt, $J = 6.9, 1.2$ Hz, 3H), 1.63 (septet, $J = 6.9$ Hz, 1H), 1.86 (m, 1H), 1.99 (t, $J = 7.2$ Hz, 2H), 2.10 (m, 1H), 2.61–2.77 (m, 2H), 4.02–4.17 (m, 4H), 5.16 (dt, $J = 14.7, 7.2$ Hz, 1H), 5.57 (m, 2H), 7.17–7.19 (m, 3H), 7.21–7.31 (m, 2H). ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 15.95 (d, $J = 4.0$ Hz), 16.05 (d, $J = 4.0$ Hz), 22.18, 22.21, 28.39, 31.07, 36.62, 38.12 (d, $J = 6.3$ Hz), 63.43 (d, $J = 6.3$ Hz), 63.45 (d, $J = 6.3$ Hz), 74.40 (d, $J = 6.3$ Hz), 126.02, 128.45, 128.48, 129.09 (d, $J = 3.4$ Hz), 132.90, 141.52. HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{19}H_{31}O_4PNa$, 377.18576; found, 377.18750.

(Z)-Diethyl 2-Methyl-5-decen-4-yl Phosphate (3e)



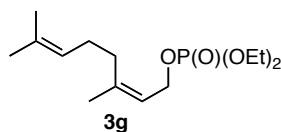
Oil. 1H NMR (300 MHz, $CDCl_3$) δ 0.88–0.97 (m, 9H), 1.28–1.39 (m, 11H), 1.62–1.74 (m, 2H), 2.12–2.19 (m, 2H), 4.01–4.15 (m, 4H), 5.17 (m, 1H), 5.39 (tt, $J = 10.8, 1.2$ Hz, 1H), 5.54 (dt, $J = 10.8, 7.5$ Hz, 1H). ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 13.81, 15.91 (d, $J = 3.4$ Hz), 16.01 (d, $J = 3.4$ Hz), 22.24 (d, $J = 3.4$ Hz), 22.70, 24.08, 27.35, 31.54, 45.40 (d, $J = 6.3$ Hz), 63.26 (d, $J = 5.7$ Hz), 63.30 (d, $J = 5.7$ Hz), 73.35 (d, $J = 5.7$ Hz), 128.99 (d, $J = 2.9$ Hz), 133.62. HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{15}H_{31}O_4PNa$, 329.18522; found, 329.18579.

(Z)-Diethyl 2-Hexenyl Phosphate (3f)



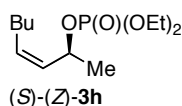
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.90 (t, $J = 7.5$ Hz, 3H), 1.34 (td, $J = 7.2, 1.2$ Hz, 6H), 1.36–1.46 (m, 2H), 2.07 (q, $J = 6.9$ Hz, 2H), 4.11 (quint., $J = 7.2$ Hz, 4H), 4.60 (dd, $J = 8.1, 6.0$ Hz, 2H), 5.55–5.70 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.34, 15.80, 15.89, 22.25, 29.22, 62.84, 62.91, 63.51 (d, $J = 5.7$ Hz), 124.09 (d, $J = 6.8$ Hz), 135.11. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{10}\text{H}_{21}\text{O}_4\text{PNa}$, 259.10697; found, 259.10698.

(Z)-Diethyl 3,7-Dimethylocta-2,6-dienyl Phosphate (3g)



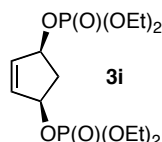
Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.33 (t, $J = 6.9$ Hz, 6H), 1.60 (s, 3H), 1.68 (s, 3H), 1.77 (s, 3H), 2.05–2.15 (m, 4H), 4.10 (quint., $J = 6.9$ Hz, 4H), 4.54 (t, $J = 7.5$ Hz, 2H), 5.08 (m, 1H), 5.41 (t, $J = 7.5$ Hz, 1H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 15.95, 16.04, 17.53, 23.37, 25.56, 26.48, 32.01, 63.53 (d, $J = 5.7$ Hz), 63.71 (d, $J = 5.7$ Hz), 119.96 (d, $J = 6.8$ Hz), 123.51, 132.36, 142.74. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{27}\text{O}_4\text{PNa}$, 313.15392; found, 313.15407.

(S, Z)-Diethyl 3-Octen-2-yl Phosphate (3h).

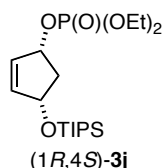


Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.90 (t, $J = 6.9$ Hz, 3H), 1.29–1.36 (m, 10H), 1.38 (d, $J = 6.3$ Hz, 3H), 2.09–2.16 (m, 2H), 4.02–4.14 (m, 4H), 5.31 (dq, $J = 12.6, 6.3$ Hz, 1H), 5.41–5.55 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.79, 15.90 (d, $J = 1.1$ Hz), 15.99 (d, $J = 1.1$ Hz), 22.17, 22.67 (d, $J = 5.1$ Hz), 27.22, 31.53, 63.55 (d, $J = 5.7$ Hz), 63.40 (d, $J = 5.7$ Hz), 71.28 (d, $J = 5.7$ Hz), 129.92 (d, $J = 5.1$ Hz), 132.76. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{12}\text{H}_{25}\text{O}_4\text{PNa}$, 287.13881; found, 287.13795. $[\alpha]_D^{24} +46.4$ (c 1.54, CHCl_3).

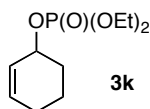
cis-4-Cyclopentene-1,3-diyl Tetraethyl Diphosphate (3i)



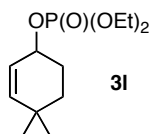
Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.35 (t, $J = 7.2$ Hz, 12H), 2.05 (dt, $J = 14.4, 4.5$ Hz, 1H), 2.93 (dt, $J = 14.4, 6.9$ Hz, 1H), 4.12 (quint., $J = 7.2$ Hz, 8H), 5.23 (dd, $J = 6.9, 4.5$ Hz, 2H), 6.15 (s, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 16.00 (d, $J = 5.7$ Hz), 39.51, 63.81 (d, $J = 5.7$ Hz), 79.14 (d, $J = 5.7$ Hz), 135.15 (d, $J = 5.1$ Hz). HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{13}\text{H}_{26}\text{O}_8\text{P}_2\text{Na}$, 395.09951; found,

Diethyl (1*R*, 4*S*)-4-(Triisopropylsiloxy)-2-cyclopentenyl Phosphate (3j)

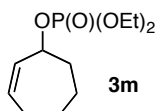
Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.05–1.08 (m, 21H), 1.34 (td, $J = 7.2, 0.9$ Hz, 6H), 1.80 (dt, $J = 13.5, 5.4$ Hz, 1H), 2.86 (dt, $J = 13.5, 7.2$ Hz, 1H), 4.11 (quint., $J = 7.2$ Hz, 4H), 4.75 (t, $J = 5.1$ Hz, 1H), 5.18 (q, $J = 6.0$ Hz, 1H), 5.96 (dm, $J = 5.4$ Hz, 1H), 6.02 (dm, $J = 5.4$ Hz, 1H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 11.91, 15.95, 16.04, 17.80, 17.81, 42.61 (d, $J = 4.5$ Hz), 63.59 (d, $J = 1.2$ Hz), 63.67 (d, $J = 1.2$ Hz), 74.54, 79.84 (d, $J = 5.8$ Hz), 131.84 (d, $J = 5.8$ Hz), 139.21. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{37}\text{O}_5\text{PSiNa}$, 415.20401; found, 415.20462. $[\alpha]_{\text{D}}^{25} -16.4$ (c 1.1, C_6H_6).

2-Cyclohexenyl Diethyl Phosphate (3k)

Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.34 (t, $J = 7.2$ Hz, 6H), 1.55–2.18 (m, 6H), 4.11 (quint., $J = 7.2$ Hz, 4H), 4.88 (brs, 1H), 5.79 (d, $J = 10.2$ Hz, 1H), 5.95 (dt, $J = 10.2, 3.6$ Hz, 1H),. ^{13}C NMR (75.4 MHz, CDCl_3) δ 15.89, 15.98, 18.27, 24.56, 29.69 (d, $J = 4.6$ Hz), 63.38 (d, $J = 5.7$ Hz), 63.45 (d, $J = 5.7$ Hz), 72.03 (d, $J = 5.7$ Hz), 126.29 (d, $J = 4.6$ Hz), 132.73. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{10}\text{H}_{19}\text{O}_4\text{PNa}$, 257.09186; found, 257.09175.

Diethyl 4,4-Dimethyl-2-cyclohexenyl Phosphate (3l)

Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.97 (s, 3H), 1.03 (s, 3H), 1.34 (td, $J = 6.6, 1.2$ Hz, 6H), 1.42 (m, 1H), 1.63 (m, 1H), 1.82–2.01 (m, 2H), 4.11 (quintet d, $J = 7.2, 1.2$ Hz, 4H), 4.82 (q, $J = 5.4$ Hz, 1H), 5.63 (s, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 15.91, 16.00, 26.90 (d, $J = 4.0$ Hz), 28.44, 28.82, 31.56, 32.88, 63.40, 63.47 (d, $J = 6.3$ Hz), 72.32 (d, $J = 5.7$ Hz), 123.75 (d, $J = 5.1$ Hz), 142.56. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{12}\text{H}_{23}\text{O}_4\text{PNa}$, 285.12262; found, 285.12304.

2-Cycloheptenyl Diethyl Phosphate (3m)

Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.34 (t, $J = 7.2$ Hz, 6H), 1.42 (m, 1H), 1.56–1.84 (m, 3H), 1.92–2.09 (m, 3H), 2.19 (m, 1H), 4.11 (quint., $J = 7.2$ Hz, 4H), 5.01 (t, $J = 9.0$ Hz, 1H), 5.74–5.87 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 16.00, 16.06, 26.09, 26.34, 28.29, 34.37 (d, $J = 4.6$ Hz), 63.53 (d, $J = 6.3$ Hz), 63.54 (d, $J = 6.3$ Hz), 78.34 (d, $J = 5.7$ Hz), 131.71, 134.15 (d, $J = 5.1$ Hz). HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{11}\text{H}_{21}\text{O}_4\text{PNa}$, 271.10751; found, 271.10732.

Procedures for Copper-Catalyzed Allyl–Alkyl Coupling

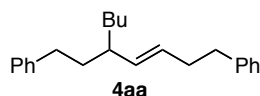
Typical Procedure for Hydroboration/Allyl–Alkyl Coupling Sequence (Scheme 1). In a glove box, (9-BBN-H) $_2$ (91.5 mg, 0.375 mmol) was placed in a vial containing a magnetic stirring bar. Then, the vial was sealed with a cap equipped with a Teflon-coated silicon rubber septum. The vial was removed from the glove box. THF (0.3 mL) and styrene (105 μL , 0.9 mmol) were sequentially added, and the mixture was stirred at 60 $^\circ\text{C}$ for 1 hour to prepare alkylborane **2a**. On the other hand, in a glove box, CuOAc (6.1 mg, 0.05 mmol) was placed in another vial. *t*-BuOK (1 M in THF, 0.5 mL, 0.5 mmol) was added to alkylborane **2a** prepared in advance at 25 $^\circ\text{C}$, and the mixture was stirred at 25 $^\circ\text{C}$ for 5 min to produce the corresponding alkylborate. Next, the alkylborate was then transferred to another vial containing Cu salt. Finally, allylic phosphate **3a** (177.2 mg, 175 μL , 0.5 mmol) was added. After 8 h stirring at 60 $^\circ\text{C}$, CH_2Cl_2 was added to the mixture. Then, the mixture was filtered through a short plug of silica gel, which was washed with diethyl ether. After the solvent was removed under reduced pressure, flash chromatography on silica gel (hexane) provided **4a** (122 mg, 0.4 mmol) in 80% yield.

Procedure for Hydroboration/Allyl–Alkyl Coupling Sequence (Scheme 2). In a glove box, (9-BBN-H) $_2$ (33.6 mg, 0.1375 mmol) and steroid **1n** (102.8 mg, 0.286 mmol) were placed in a vial containing a magnetic stirring bar. Then the vial was sealed with a cap equipped with a Teflon-coated silicon rubber septum. The vial was removed from the glove box. The mixture was dissolved in THF (0.37 mL) at 0 $^\circ\text{C}$, and the mixture was stirred at 25 $^\circ\text{C}$ for 24 hour to prepare alkylborane **2n**. After the vial was brought into a glove box, *t*-BuOK (1 M in THF, 0.25 mL, 0.25 mmol) was added at 25 $^\circ\text{C}$, and the mixture was stirred at 25 $^\circ\text{C}$ for 5 min to produce the corresponding alkylborate. Next, CuOAc (3.1 mg, 0.025 mmol) was added, and then the vial was removed from the glove box. Finally, allylic phosphate (1*R*,4*S*)-**3j** (98.1 mg, 0.25 mmol) was added. After 8 h stirring at 60 $^\circ\text{C}$, CH_2Cl_2 was added to the mixture. Then, the mixture was filtered through a short plug of silica gel, which was washed with diethyl ether. After the solvent was removed under reduced pressure, flash chromatography on silica gel (hexane) and GPC (CHCl_3) provided **5** (74.1 mg, 0.125 mmol) in 50% yield.

Characterization Data for Coupling Products

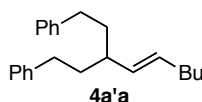
Coupling products **4k**⁴ and **4m**⁵ were reported in the literature.

(E)-5-(2-Phenylethyl)-1-phenyl-3-nonene (4aa)



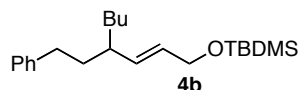
Oil. ¹H NMR (300 MHz, CDCl₃) δ 0.86 (t, *J* = 6.9 Hz, 3H), 1.08–1.37 (m, 6H), 1.45 (m, 1H), 1.62 (m, 1H), 1.88 (m, 1H), 2.36 (q, *J* = 7.2 Hz, 2H), 2.44 (ddd, *J* = 13.8, 10.2, 6.3 Hz, 1H), 2.57 (ddd, *J* = 13.8, 10.2, 5.2 Hz, 1H), 2.70 (t, *J* = 7.2 Hz, 2H), 5.16 (dd, *J* = 15.3, 8.9 Hz, 1H), 5.40 (dt, *J* = 15.3, 7.2 Hz, 1H), 7.12–7.20 (m, 6H), 7.24–7.30 (m, 4H). ¹³C NMR (75.4 MHz, CDCl₃) δ 14.00, 22.70, 29.29, 33.53, 34.30, 35.16, 36.13, 37.22, 42.45, 125.55, 125.77, 128.27, 128.31, 128.49, 128.59, 129.78, 135.51, 142.15, 143.19. Anal. Calcd for C₂₃H₃₀: C, 90.13; H, 9.87%. Found: C, 89.95; H, 10.07%. The regioselectivity of **4aa** was assigned on the basis of ¹H and ¹³C NMR of 3-phenylpropanoic acid obtained by ozonolytic cleavage followed by Jones oxidation.

(E)-3-(2-Phenylethyl)-1-phenyl-4-nonene (4a'a)



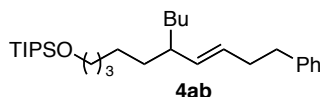
Oil. ¹H NMR (300 MHz, CDCl₃) δ 0.92 (t, *J* = 7.2 Hz, 3H), 1.25–1.44 (m, 4H), 1.47–1.60 (m, 2H), 1.63–1.75 (m, 2H), 1.93–2.10 (m, 3H), 2.50 (ddd, *J* = 13.8, 10.2, 6.3 Hz, 2H), 2.64 (ddd, *J* = 13.8, 10.2, 5.4 Hz, 2H), 5.20 (dd, *J* = 15.3, 8.7 Hz, 1H), 5.43 (dt, *J* = 15.3, 6.9 Hz, 1H), 7.11–7.18 (m, 6H), 7.21–7.29 (m, 4H). ¹³C NMR (75.4 MHz, CDCl₃) δ 13.86, 22.10, 31.83, 32.27, 33.54, 37.37, 42.24, 125.61, 128.32, 128.49, 131.79, 134.08, 143.07. Anal. Calcd for C₂₃H₃₀: C, 90.13; H, 9.87%. Found: C, 90.10; H, 9.88%. The regioselectivity of **4a'a** was assigned on the basis of ¹H and ¹³C NMR of pentanoic acid obtained by ozonolytic cleavage followed by Jones oxidation.

(E)-1-(tert-Butyldimethylsiloxy)-4-(2-phenylethyl)-2-octene (4b)



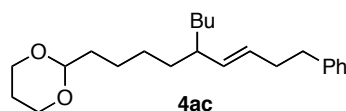
Oil. ¹H NMR (300 MHz, CDCl₃) δ 0.88 (s, 6H), 0.84–0.89 (m, 3H), 0.92 (s, 9H), 1.12–1.40 (m, 6H), 1.54 (m, 1H), 1.66 (m, 1H), 1.99 (m, 1H), 2.50 (ddd, *J* = 13.8, 10.2, 6.6 Hz, 1H), 2.63 (ddd, *J* = 13.8, 10.2, 5.4 Hz, 1H), 4.17 (d, *J* = 5.1 Hz, 2H), 5.41 (dd, *J* = 15.3, 8.4 Hz, 1H), 5.53 (dt, *J* = 15.3, 5.1 Hz, 1H), 7.15–7.18 (m, 3H), 7.24–7.30 (m, 2H). ¹³C NMR (75.4 MHz, CDCl₃) δ –5.19, 13.98, 18.33, 22.70, 25.86, 29.26, 33.51, 34.90, 36.99, 41.96, 63.99, 125.61, 128.32, 128.48, 129.74, 135.31, 143.04. Anal. Calcd for C₂₂H₃₈OSi: C, 76.23; H, 11.05%. Found: C, 76.15; H, 10.94%. The regioselectivity of **4b** was assigned on the basis of ¹H and ¹³C NMR of 2-(2-phenylethyl)hexanoic acid⁶ obtained by ozonolytic cleavage followed by Jones oxidation.

(E)-5-Butyl-10-triisopropylsiloxy-1-phenyl-3-decene (4ab)



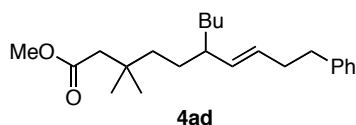
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.87 (t, $J = 7.2$ Hz, 3H), 1.00–1.25 (s, 33H), 1.49–1.54 (m, 2H), 1.84 (m, 1H), 2.31 (td, $J = 7.8, 6.6$ Hz, 2H), 2.67 (t, $J = 7.8$ Hz, 2H), 3.66 (t, $J = 6.6$ Hz, 2H), 5.10 (dd, $J = 15.3, 8.7$ Hz, 1H), 5.34 (dt, $J = 15.3, 6.6$ Hz, 1H), 7.17–7.19 (m, 3H), 7.25–7.30 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 11.88, 14.00, 17.92, 22.70, 25.82, 26.95, 29.36, 32.95, 34.33, 35.11, 35.39, 36.18, 42.64, 63.49, 125.70, 128.26, 128.59, 129.00, 135.96, 142.27. Anal. Calcd for $\text{C}_{29}\text{H}_{52}\text{OSi}$: C, 78.31; H, 11.78%. Found: C, 78.10; H, 11.80%.

(E)-5-Butyl-10,10-trimethylenedioxy-1-phenyl-3-decene (4ac)



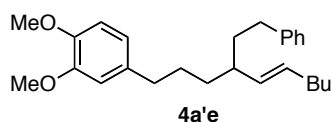
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.86 (t, $J = 6.9$ Hz, 3H), 1.11–1.36 (m, 13H), 1.53–1.60 (m, 2H), 1.82 (m, 1H), 2.07 (m, 1H), 2.31 (td, $J = 7.5, 6.6$ Hz, 2H), 2.66 (t, $J = 7.5$ Hz, 2H), 3.76 (td, $J = 12.0, 2.4$ Hz, 2H), 4.10 (ddd, $J = 12.0, 5.1, 1.2$ Hz, 2H), 4.49 (t, $J = 5.1$ Hz, 1H), 5.09 (dd, $J = 15.0, 8.7$ Hz, 1H), 5.34 (dt, $J = 15.0, 6.6$ Hz, 1H), 7.14–7.22 (m, 3H), 7.24–7.30 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 14.01, 22.71, 23.97, 25.76, 26.93, 29.34, 34.34, 35.10, 35.16, 35.23, 36.18, 42.55, 66.88, 102.50, 125.72, 128.27, 128.59, 129.07, 135.88, 142.26. HRMS–EI (m/z): $[\text{M}-\text{H}]^+$ calcd for $\text{C}_{23}\text{H}_{35}\text{O}_2$, 343.26370; found, 343.26306.

(E)-Methyl 6-Butyl-3,3-dimethyl-10-phenyl-7-decenoate (4ad)



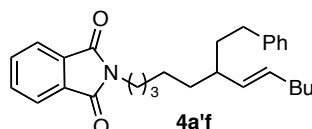
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.87 (t, $J = 6.9$ Hz, 3H), 0.95 (s, 6H), 1.06–1.29 (m, 10H), 1.75 (m, 1H), 2.17 (s, 2H), 2.32 (q, $J = 7.2$ Hz, 2H), 2.68 (t, $J = 7.2$ Hz, 2H), 3.64 (s, 3H), 5.09 (dd, $J = 15.3, 8.7$ Hz, 1H), 5.34 (dt, $J = 15.3, 7.2$ Hz, 1H), 7.17–7.20 (m, 3H), 7.26–7.30 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 14.00, 22.69, 27.14, 27.19, 29.33, 29.58, 33.05, 34.25, 35.12, 36.14, 39.82, 43.25, 45.74, 51.00, 125.74, 128.28, 128.58, 129.27, 135.77, 142.20, 173.09. HRMS–EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{23}\text{H}_{36}\text{O}_2$, 344.27153; found, 344.24148.

(E)-1-(3,4-Dimethoxyphenyl)-7-(2-phenylethyl)-5-decene (4a'e)



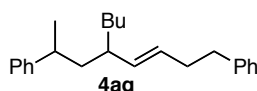
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.90 (t, $J = 6.9$ Hz, 3H), 1.18–1.69 (m, 10H), 1.92–2.06 (m, 3H), 2.43–2.53 (m, 3H), 2.64 (ddd, $J = 14.1, 9.9, 5.4$ Hz, 1H), 3.86 (s, 3H), 3.87 (s, 3H), 5.13 (dd, $J = 15.3, 6.9$ Hz, 1H), 5.39 (dd, $J = 15.3, 6.9$ Hz, 1H), 6.67–6.71 (m, 2H), 6.76–6.80 (m, 1H), 7.14–7.19 (m, 3H), 7.24–7.29 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.83, 22.07, 29.22, 31.83, 32.22, 33.55, 35.07, 35.48, 37.30, 42.32, 55.69, 55.84, 111.12, 111.72, 120.16, 125.58, 128.29, 128.49, 131.25, 134.31, 135.60, 143.12, 147.06, 148.80. Anal. Calcd for $\text{C}_{26}\text{H}_{36}\text{O}_2$: C, 82.06; H, 9.53%. Found: C, 82.01; H, 9.55%.

(E)-7-(2-Phenylethyl)-12-phthalimide-5-dodecene (4a'f)



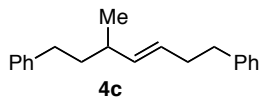
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.89 (t, $J = 6.9$ Hz, 3H), 1.18–1.36 (m, 10H), 1.45 (m, 1H), 1.60–1.69 (m, 3H), 1.89 (m, 1H), 2.01 (q, $J = 6.3$ Hz, 2H), 2.48 (ddd, $J = 13.5, 10.2, 6.6$ Hz, 1H), 2.62 (ddd, $J = 13.5, 10.2, 5.1$ Hz, 1H), 3.66 (t, $J = 7.2$ Hz, 2H), 5.11 (dd, $J = 15.0, 8.7$ Hz, 1H), 5.35 (dt, $J = 15.0, 6.6$ Hz, 1H), 7.14–7.17 (m, 3H), 7.24–7.34 (m, 2H), 7.70 (dd, $J = 6.0, 3.0$ Hz, 2H), 7.84 (dd, $J = 6.0, 3.0$ Hz, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.83, 22.06, 26.68, 26.87, 28.50, 31.79, 32.20, 33.55, 35.27, 37.29, 38.00, 42.41, 123.21, 125.55, 128.29, 128.48, 131.14, 132.26, 133.91, 134.34, 143.18, 168.62. Anal. Calcd for $\text{C}_{28}\text{H}_{35}\text{NO}_2$: C, 80.53; H, 8.45; N, 3.35%. Found: C, 80.40; H, 8.45; N, 3.31%.

(E)-1-Phenyl-5-(2-phenylpropyl)-3-nonene (4ag)



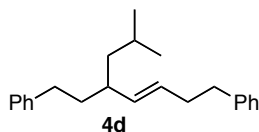
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.80 (t, $J = 7.2$ Hz, $0.5 \times 3\text{H}$), 0.88 (t, $J = 7.2$ Hz, $0.5 \times 3\text{H}$), 1.05–1.36 (m, $0.5 \times 9\text{H}$, $0.5 \times 9\text{H}$), 1.40 (m, $0.5 \times 1\text{H}$), 1.46–1.48 (m, $0.5 \times 2\text{H}$), 1.58–1.65 (m, $0.5 \times 2\text{H}$), 1.97 (m, $0.5 \times 1\text{H}$), 2.31–2.38 (m, $0.5 \times 2\text{H}$, $0.5 \times 2\text{H}$), 2.62–2.73 (m, $0.5 \times 3\text{H}$, $0.5 \times 3\text{H}$), 5.08 (dd, $J = 15.5, 9.0$ Hz, $0.5 \times 1\text{H}$), 5.08 (dd, $J = 15.5, 9.0$ Hz, $0.5 \times 1\text{H}$), 5.23 (dt, $J = 15.5, 6.3$ Hz, $0.5 \times 1\text{H}$), 5.36 (dt, $J = 15.5, 6.3$ Hz, $0.5 \times 1\text{H}$), 7.09–7.31 (m, $0.5 \times 10\text{H}$, $0.5 \times 10\text{H}$). ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.94, 13.99, 20.93, 22.67 ($\times 2\text{C}$), 23.61, 29.13, 29.22, 34.21, 34.29, 35.30, 35.62, 36.09 ($\times 2\text{C}$), 36.75, 37.24, 40.43, 40.52, 43.91, 44.28, 125.71, 125.74, 125.76 ($\times 2\text{C}$), 126.98, 127.33, 128.30 ($\times 4\text{C}$), 128.60 ($\times 2\text{C}$), 129.41, 129.66, 135.49, 135.68, 142.21 ($\times 2\text{C}$), 147.60, 148.78. HRMS–EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{24}\text{H}_{32}$, 320.25040; found, 320.25048. The regioselectivity of **4ag** was assigned on the basis of ^1H and ^{13}C NMR of 3-phenylpropanoic acid obtained by ozonolytic cleavage followed by Jones oxidation.

(E)-5-Methyl-1,7-diphenyl-3-heptene (4c)



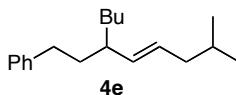
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.97 (d, $J = 6.9$ Hz, 3H), 1.50–1.62 (m, 2H), 2.10 (septet, $J = 6.9$ Hz, 1H), 2.33 (q, $J = 6.9$ Hz, 2H), 2.46–2.61 (m, 2H), 2.69 (t, $J = 7.2$ Hz, 2H), 5.33 (dd, $J = 15.9, 6.9$ Hz, 1H), 5.43 (dt, $J = 15.9, 7.2$ Hz, 1H), 7.14–7.21 (m, 6H), 7.24–7.30 (m, 4H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 20.86, 33.55, 34.33, 36.09, 36.33, 38.78, 125.60, 125.78, 128.24, 128.31 ($\times 2$), 128.49, 128.60, 136.78, 142.20, 143.04. Anal. Calcd for $\text{C}_{20}\text{H}_{24}$: C, 90.85; H, 9.15%. Found: C, 90.81; H, 9.19%.

(E)-7-Methyl-1-phenyl-5-(2-phenylethyl)-3-octene (4d)



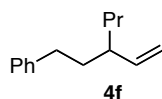
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.79 (d, $J = 6.6$ Hz, 3H), 0.82 (d, $J = 6.6$ Hz, 3H), 1.10 (dt, $J = 8.7, 5.7$ Hz, 2H), 1.35–1.65 (m, 3H), 2.00 (m, 1H), 2.38 (m, 2H), 2.45 (ddd, $J = 13.8, 10.2, 6.3$ Hz, 1H), 2.56 (ddd, $J = 13.8, 10.2, 5.4$ Hz, 1H), 2.70 (t, $J = 7.5$ Hz, 2H), 5.12 (dd, $J = 15.3, 9.0$ Hz, 1H), 5.40 (dt, $J = 15.3, 6.6$ Hz, 1H), 7.12–7.20 (m, 6H), 7.24–7.30 (m, 4H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 21.68, 23.47, 25.08, 33.52, 34.31, 36.12, 37.60, 40.34, 44.85, 125.55, 125.77, 128.27, 128.30, 128.48, 128.61, 129.68, 135.51, 142.13, 143.19. Anal. Calcd for $\text{C}_{23}\text{H}_{30}$: C, 90.13; H, 9.87%. Found: C, 90.04; H, 9.81%. The regioselectivity of **4d** was assigned on the basis of ^1H and ^{13}C NMR of 3-phenylpropanoic acid obtained by ozonolytic cleavage followed by Jones oxidation.

(E)-2-Methyl-6-(2-phenylethyl)-4-decene (4e)



Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.86 (t, $J = 6.6$ Hz, 3H), 0.89 (d, $J = 6.6$ Hz, 3H), 0.90 (d, $J = 6.6$ Hz, 3H), 1.13–1.39 (m, 6H), 1.41–1.72 (m, 3H), 1.89–1.94 (m, 3H), 2.51 (ddd, $J = 13.5, 10.2, 6.6$ Hz, 1H), 2.64 (ddd, $J = 13.5, 10.2, 5.2$ Hz, 1H), 5.13 (dd, $J = 15.0, 9.0$ Hz, 1H), 5.36 (dt, $J = 15.0, 7.2$ Hz, 1H), 7.13–7.18 (m, 3H), 7.24–7.30 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 14.00, 22.19, 22.22, 22.66, 28.45, 29.39, 33.66, 35.22, 37.40, 42.03, 42.59, 125.55, 128.29, 128.48, 129.59, 135.84, 143.28. Anal. Calcd for $\text{C}_{19}\text{H}_{30}$: C, 88.30; H, 11.70%. Found: C, 88.01; H, 11.85%.

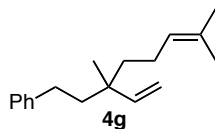
3-(2-Phenylethyl)-1-hexene (4f)



Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.86 (t, $J = 6.3$ Hz, 3H), 1.24–1.37 (m, 4H), 1.57 (m, 1H), 1.68 (m, 1H), 2.00 (m, 1H), 2.51 (ddd, $J = 13.8, 10.2, 6.3$ Hz, 1H), 2.65 (ddd, $J = 13.8, 10.2, 5.4$ Hz,

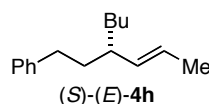
1H), 4.98 (dd, $J = 16.8, 2.1$ Hz, 1H), 5.02 (dd, $J = 10.2, 2.1$ Hz, 1H), 5.57 (ddd, $J = 16.8, 10.2, 9.0$ Hz, 1H), 7.16–7.18 (m, 3H), 7.25–7.30 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 14.03, 20.08, 33.45, 36.76, 37.20, 43.47, 114.66, 125.63, 128.32, 128.48, 143.02, 143.20. Anal. Calcd for $\text{C}_{14}\text{H}_{20}$: C, 89.29; H, 10.71%. Found: C, 89.10; H, 10.83%.

3,7-Dimethyl-3-(2-phenylethynyl)-1,6-octadiene (**4g**)



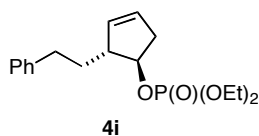
Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.05 (s, 3H), 1.25–1.38 (m, 2H), 1.54–1.62 (m, 2H), 1.59 (s, 3H), 1.68 (s, 3H), 1.87–1.95 (m, 2H), 2.48–2.54 (m, 2H), 4.96 (dd, $J = 17.7, 1.5$ Hz, 1H), 5.06 (dd, $J = 11.1, 1.5$ Hz, 1H), 5.10 (m, 1H), 5.76 (dd, $J = 17.7, 1.1$ Hz, 1H), 7.15–7.19 (m, 3H), 7.25–7.30 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 17.49, 22.41, 22.69, 25.61, 30.63, 39.56, 40.65, 42.88, 112.06, 124.99, 125.63, 128.40 ($\times 2\text{C}$), 131.27, 143.46, 146.97. HRMS–EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{18}\text{H}_{26}$, 242.20345; found, 242.20325.

(*S*, *E*)-4-(2-Phenylethyl)-2-octene (**4h**)



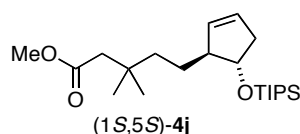
Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.87 (d, $J = 6.9$ Hz, 3H), 1.18–1.35 (m, 6H), 1.53 (m, 1H), 1.69 (dd, $J = 6.3, 1.5$ Hz, 3H), 1.70 (m, 1H), 1.90 (m, 1H), 2.48 (ddd, $J = 13.8, 10.2, 6.6$ Hz, 1H), 2.63 (ddd, $J = 13.8, 10.2, 5.4$ Hz, 1H), 5.18 (ddq, $J = 15.0, 8.7, 1.5$ Hz, 1H), 5.38 (dq, $J = 15.0, 6.3$ Hz, 1H), 7.14–7.18 (m, 3H), 7.23–7.29 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 14.01, 17.89, 22.74, 29.36, 33.59, 35.19, 37.24, 42.49, 125.01, 125.55, 128.29, 128.49, 135.91, 143.25. Anal. Calcd for $\text{C}_{16}\text{H}_{24}$: C, 88.82; H, 11.18%. Found: C, 88.54; H, 11.17%. $[\alpha]_{\text{D}}^{24} +1.0$ (c 0.8, CHCl_3). The ee value of (*S*)-(*E*)-**4h** was determined by chiral HPLC (CHIRALCEL[®] OD-3 column, 4.6 mm $\times$ 250 mm, Daisel Chemical Industries, hexane, 0.5 mL/min, 40 $^\circ\text{C}$, 220 nm UV detector, retention time = 8.57 min for the *S* isomer and 8.98 min for the *R* isomer). The (*S*) absolute configuration of **4h** was determined by optical rotation of 2-(2-phenylethyl)-1-hexanol obtained in two steps from (*S*)-(*E*)-**4h**. (*S*)-(*E*)-**4h** was converted to 2-(2-phenylethyl)-1-hexanol according to ozonolytic cleavage followed by reduction with NaBH_4 . (*S*)-2-(2-phenylethyl)-1-hexanol (46% ee): $[\alpha]_{\text{D}}^{25} +0.4$ (c 1.1, CHCl_3) [Lit^6 , (*R*) isomer, $[\alpha]_{\text{D}} -0.5$ (c 3.2, CHCl_3)].

Diethyl *trans*-2-(2-Phenylethyl)-3-cyclopentenyl Phosphate (**4i**)



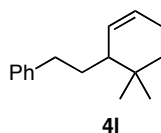
Oil. ^1H NMR (300 MHz, CDCl_3) δ 1.32 (td, $J = 7.2, 0.9$ Hz, 3H), 1.33 (td, $J = 7.2, 0.9$ Hz, 3H), 1.62–1.80 (m, 2H), 2.52 (m, 1H), 2.69 (t, $J = 7.8$ Hz, 2H), 2.76–2.83 (m, 2H), 4.09 (quint., $J = 7.2, 2.4$ Hz, 4H), 4.75 (septet, $J = 3.3$ Hz, 1H), 5.67–5.74 (m, 2H), 7.18–7.30 (m, 5H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 15.88, 15.98, 33.50, 34.36, 39.63 (d, $J = 3.9$ Hz), 52.41 (d, $J = 6.3$ Hz), 63.51, 63.60, 82.92 (d, $J = 6.3$ Hz), 125.84, 127.58, 128.36, 128.38, 132.31, 141.99. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{17}\text{H}_{25}\text{O}_4\text{PNa}$, 347.13827; found, 347.13850. The *anti* stereochemistry of **4i** was assigned on the basis of ^1H NMR of *trans*-2-(2-phenylethyl)-3-cyclopentenol⁷ obtained by dephosphorylation with LiAlH_4 . The *cis* isomer was not detected by ^1H NMR.

(1*S*,5*S*)-1-Triisopropylsiloxy-5-[(4-methoxycarbonyl-3,3-dimethyl)butyl]-3-cyclopentene (4j)



Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.97 (s, 6H), 1.05–1.08 (m, 21H), 1.15–1.57 (m, 4H), 2.19 (s, 2H), 2.27 (m, 1H), 2.49 (m, 1H), 2.62 (m, 1H), 3.64 (s, 3H), 4.15 (dt, $J = 6.3, 3.3$ Hz, 1H), 5.62–5.69 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 12.10, 17.93, 27.07, 27.12, 27.64, 33.04, 39.99, 42.30, 45.64, 51.05, 55.45, 78.30, 127.87, 132.93, 172.94. Anal. Calcd for $\text{C}_{22}\text{H}_{42}\text{O}_3\text{Si}$: C, 69.05; H, 11.06%. Found: C, 68.99; H, 10.93%. $[\alpha]_{\text{D}}^{24} +82.1$ (c 1.00, CHCl_3). The *de* value of (1*S*,5*S*)-**4j** was determined by chiral HPLC analysis of the *p*-nitrobenzoate derivative obtained by desilylation followed by benzylation from (1*S*,5*S*)-**4j**. HPLC analysis (CHIRALCEL[®] OD-3 column, 4.6 mm $\times$ 250 mm, Daisel Chemical Industries, hexane/2-propanol = 99:1, 0.5 mL/min, 40 $^\circ\text{C}$, 254 nm UV detector, retention time = 28.4 min for the *cis* isomer and 36.7 min for the *trans* isomer) revealed that the diastereomeric excess of the *p*-nitrobenzoate derivative was 94%. The *anti* stereochemistry of **4j** was determined by comparison of the coupling constants in the ^1H NMR for the corresponding alcohol (obtained by desilylation) with that for *trans*-2-(2-phenylethyl)-3-cyclopentenol.⁷ The alcohol from **4j**: δ 4.11 (CHOH , dt, $J = 6.0, 3.0$ Hz).

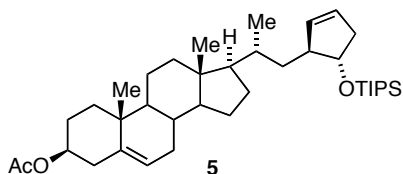
4,4-Dimethyl-3-(2-phenylethyl)-1-cyclohexene (4l)



Oil. ^1H NMR (300 MHz, CDCl_3) δ 0.76 (s, 3H), 0.88 (s, 3H), 1.23–1.41 (m, 3H), 1.74 (m, 1H), 1.83 (m, 1H), 1.98–2.03 (m, 2H), 2.51 (ddd, $J = 13.5, 10.5, 6.3$ Hz, 1H), 2.84 (ddd, $J = 13.5, 10.5, 4.8$ Hz, 1H), 5.58–5.78 (m, 2H), 7.15–7.21 (m, 3H), 7.25–7.31 (m, 2H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 21.48, 22.96, 28.76, 31.55, 32.21, 34.19, 35.87, 44.57, 125.72, 126.01, 128.37, 128.51, 129.50, 143.02. HRMS–EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{16}\text{H}_{22}$, 214.17215; found, 214.17219. The

regioselectivity of **4l** was determined on the basis of ^{13}C NMR and DEPT experiments. The chemical shift of the quaternary homoallylic carbon is diagnostic of the regiochemistry. The corresponding ^{13}C resonance appeared at higher field (δ 31.55) than that expected for the quaternary allylic carbon.

Steroid **5**



White Solid. ^1H NMR (300 MHz, CDCl_3) δ 0.68 (s, 3H), 0.87–1.23 (m, 36H), 1.43–1.60 (m, 8H), 1.79–2.05 (m, 4H), 2.03 (s, 3H), 2.22–2.32 (m, 3H), 2.59–2.64 (m, 2H), 4.05 (dt, $J = 6.3, 3.3$ Hz, 1H), 4.58 (m, 1H), 5.38 (m, 1H), 5.63 (dq, $J = 6.0, 1.5$ Hz, 1H), 5.73 (dq, $J = 6.0, 1.8$ Hz, 1H). ^{13}C NMR (75.4 MHz, CDCl_3) δ 11.76, 12.10, 17.93, 18.31, 19.19, 20.90, 21.33, 24.11, 27.64, 28.30, 31.71, 31.77, 34.35, 36.48, 36.88, 38.01, 39.65, 40.38, 42.04, 42.33, 49.92, 52.25, 56.66, 56.89, 73.94, 79.36, 122.67, 127.74, 132.98, 139.74, 170.71. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{38}\text{H}_{64}\text{O}_3\text{SiNa}$, 619.45169; found, 619.45145. $[\alpha]_{\text{D}}^{24} +33.6$ (c 2.9, CHCl_3). The *anti* stereochemistry of **5** was determined by comparing the coupling constants in the ^1H NMR of the corresponding alcohol (obtained by desilylation) with that of *trans*-2-(2-phenylethyl)-3-cyclopentenol.⁷ The alcohol derived from **5**: δ 4.01 (*CHOH*, dt, $J = 6.0, 3.0$ Hz). The *cis* isomer was not detected by ^1H NMR.

NMR Studies (Figures S1a–f, Table S1)

The mixture of (9-BBN-H)₂ (122.4 mg, 0.5 mmol) and styrene (109.4 μ l, 1.05 mmol) in THF-*d*₈ (0.4 mL) was stirred at 60 °C for 1 hour to prepare alkylborane **2a** [δ 65.7 ppm (¹¹B), Figure S1a. See also Figure S1d for the ¹H NMR spectrum].⁸ Next, *t*-BuOK (1 M in THF, 1.0 ml, 1.0 mmol) was added at 25 °C, and the mixture was stirred at 25 °C for 5 min. The ¹¹B NMR spectrum of the mixture showed a peak corresponding to a tetravalent borate (δ −1.4 ppm) (Figure S1b. See also Figure S1e for the ¹H NMR spectrum).⁹ Subsequently, CuOAc (121.6 mg, 1.0 mmol, B/Cu 1:1) was added to the borate solution, and the solution was heated at 60 °C for 1 h: the peak of the borate disappeared completely and a signal that corresponds to 9-BBN-O'Bu¹⁰ appeared at δ 55.1 ppm as a major peak in the ¹¹B NMR spectrum (Figure S1c). Meanwhile, the formation of styrene and ethylbenzene were observed by ¹H NMR spectroscopy (4% and 13% NMR yields, respectively. Dibenzyl was used as an internal standard) (Figure S1f). These compounds seem to be produced by β -hydride elimination and protonation of an alkylcopper species. The signals for styrene and ethylbenzene did not increase furthermore with prolonged heating (60 °C, 20 h).

Figure S1a. [¹¹B NMR spectrum (192.6 MHz, THF-*d*₈)]

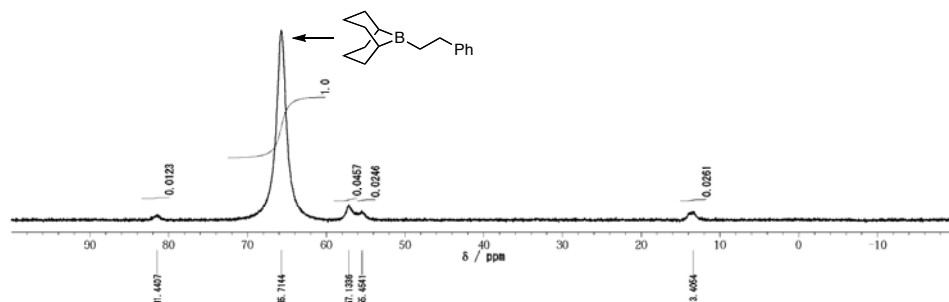


Figure S1b. [¹¹B NMR spectrum (192.6 MHz, THF-*d*₈)]

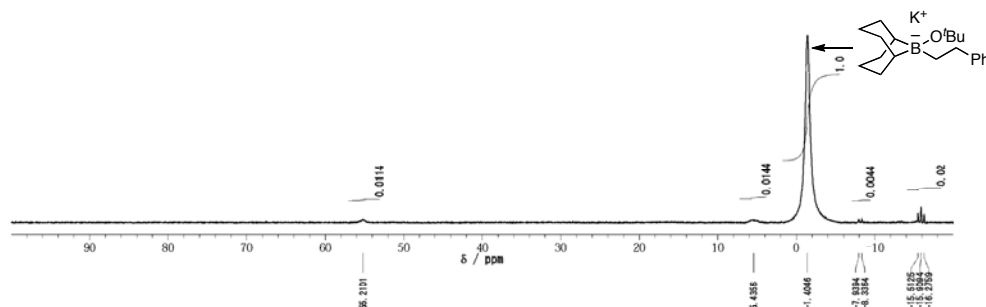
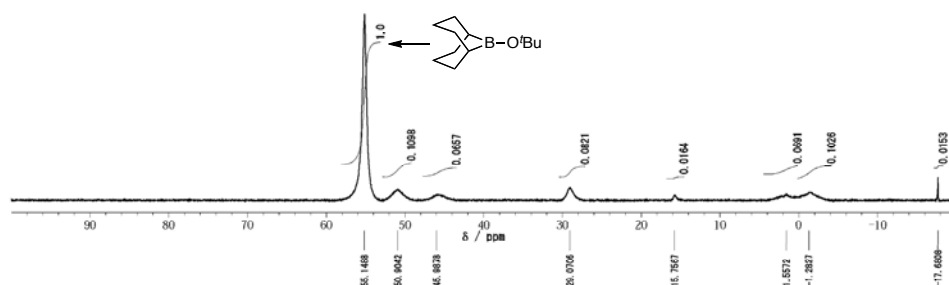
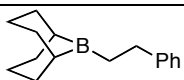
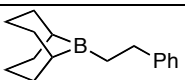
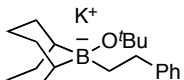
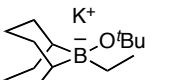
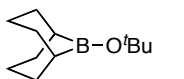
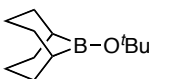


Figure S1c. [¹¹B NMR spectrum (192.6 MHz, THF-*d*₈)]



[illegible][illegible][illegible]

Table S1. Summary of the ^{11}B NMR Data for the Organoboron Compounds

this work	literature	
 δ 65.7 ppm (THF- d_8)	Soderquist, J. A.; Kock, I.; Estrella, M. E. <i>Org. Process Res. Dev.</i> 2006 , <i>10</i> , 1076–1079.	 δ 72.2 ppm (C_6D_6)
 δ -1.4 ppm (THF- d_8)	Köster, R.; Seidel, G.; Wagner, K.; Wrackmeyer, B. <i>Chem. Ber.</i> 1993 , <i>126</i> , 305–317.	 δ -1.5 ppm (THF- d_8)
 δ 55.1 ppm (THF- d_8)	Brown, H. C.; Cha, J. S.; Nazer, B. <i>J. Org. Chem.</i> 1985 , <i>50</i> , 549–553.	 δ 55.5 ppm (neat)

References

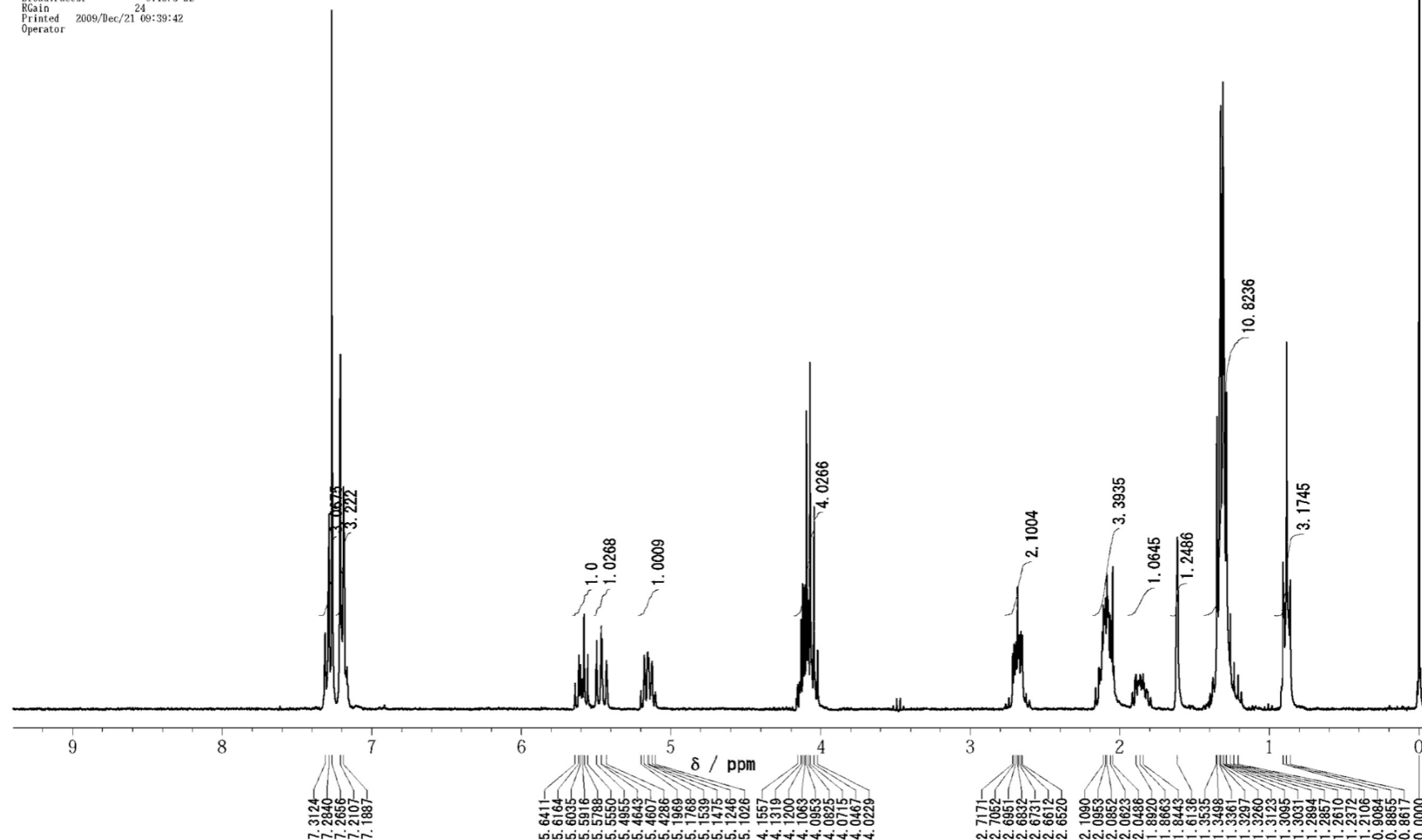
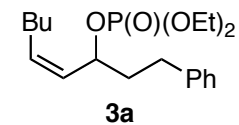
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- (2) Sato, F.; Ishikawa, H.; Sato, M. *Tetrahedron Lett.* **1981**, *22*, 85–88.
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- (4) Lysenko, I. L.; Kim, K.; Lee, H. G.; Cha, J. K. *J. Am. Chem. Soc.* **2008**, *130*, 15997–16002.
- (5) Langlois, J.-B.; Alexakis, A. *Chem. Comm.* **2009**, 3868–3870.
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- (7) (a) Partridge, J. J.; Chadha, N. K.; Uskoković, M. R. *J. Am. Chem. Soc.* **1973**, *95*, 532–540. (b) Danheiser, R. L.; Martinez-Davila, C.; Auchus, R. J.; Kadonaga, J. T. *J. Am. Chem. Soc.* **1981**, *103*, 2443–2446.
- (8) Soderquist, J. A.; Kock, I.; Estrella, M. E. *Org. Process Res. Dev.* **2006**, *10*, 1076–1079.
- (9) (a) Köster, R.; Seidel, G.; Wagner, K.; Wrackmeyer, B. *Chem. Ber.* **1993**, *126*, 305–317. (b) Fry, A.; Vishwakarma, L. C. *J. Org. Chem.* **1980**, *45*, 5306–5308.
- (10) Brown, H. C.; Cha, J. S.; Nazer, B. *J. Org. Chem.* **1985**, *50*, 549–553.

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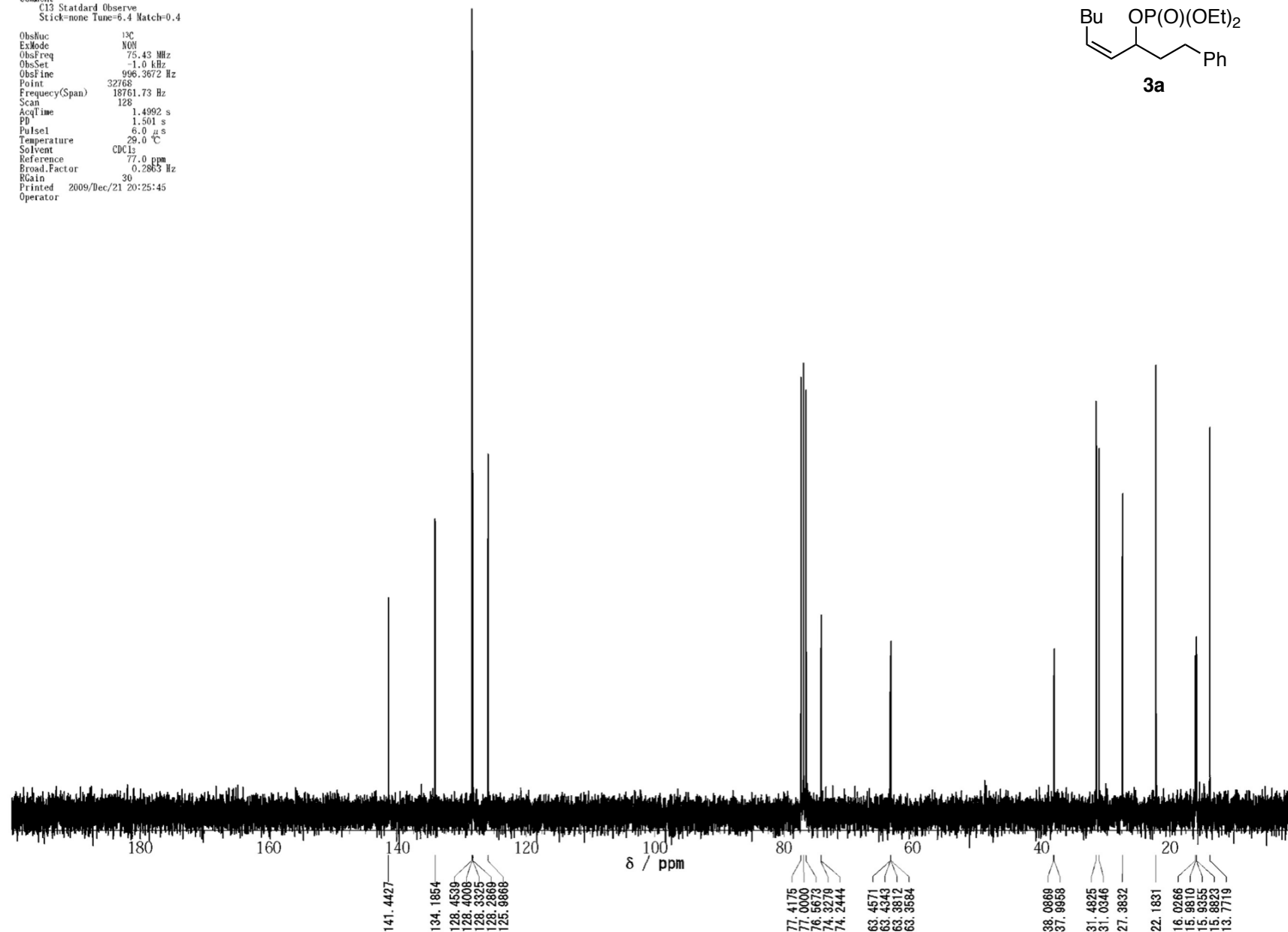
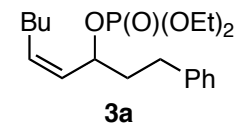
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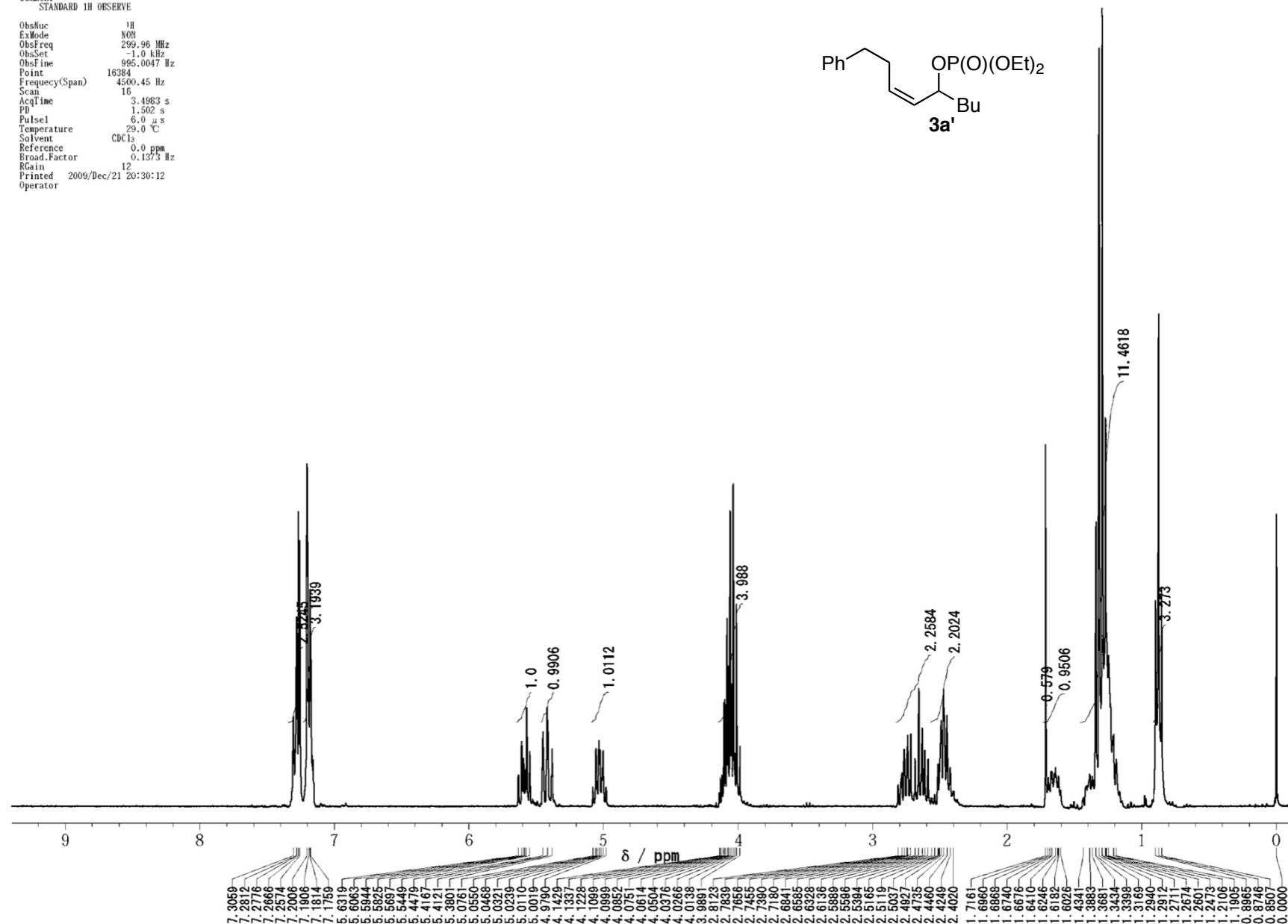
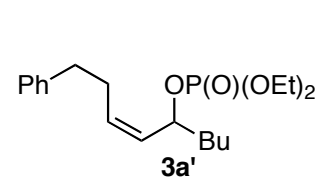
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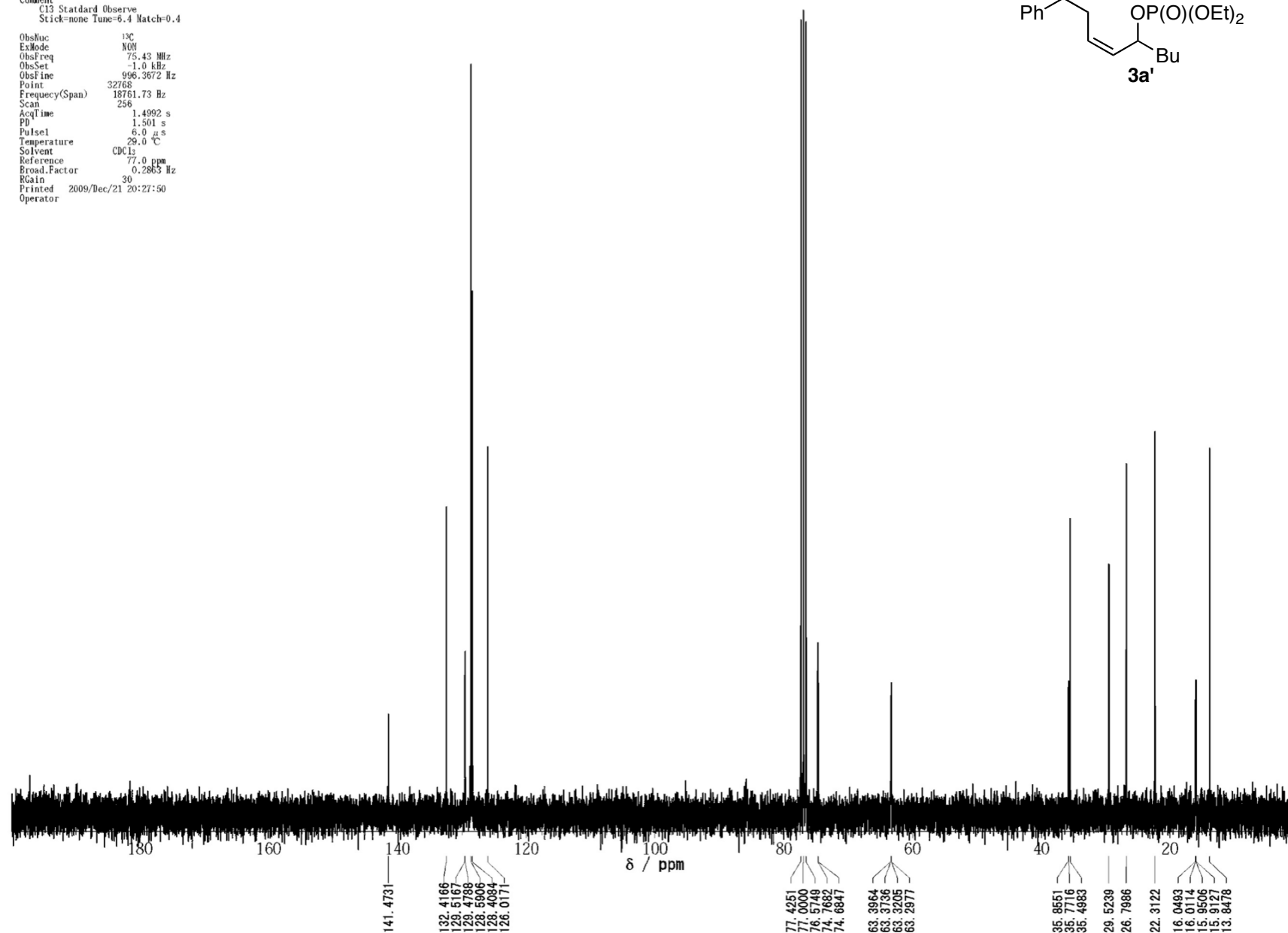
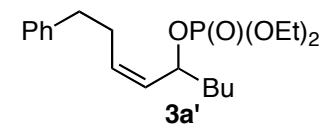
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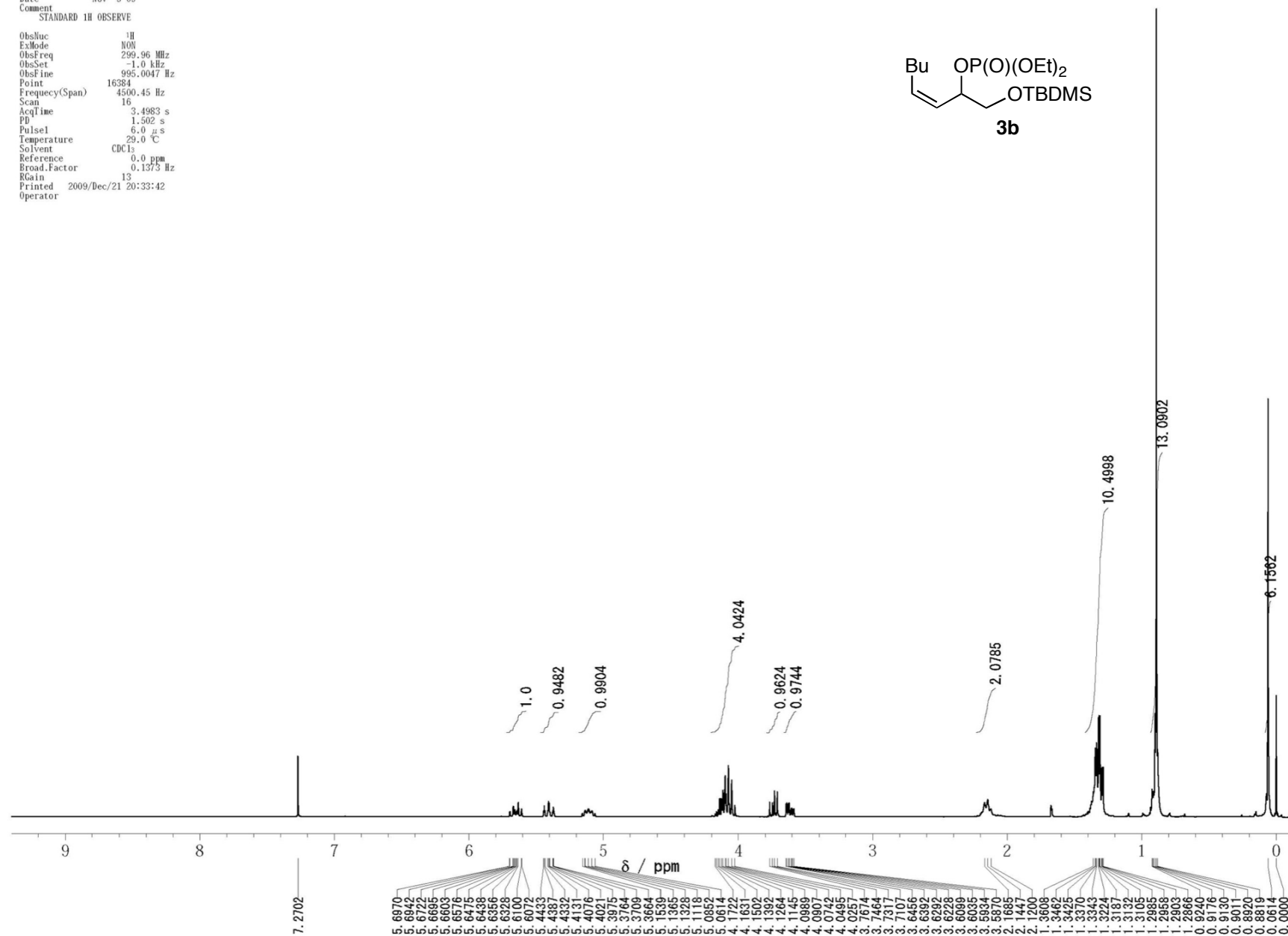
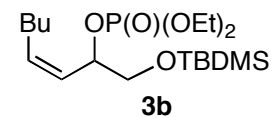
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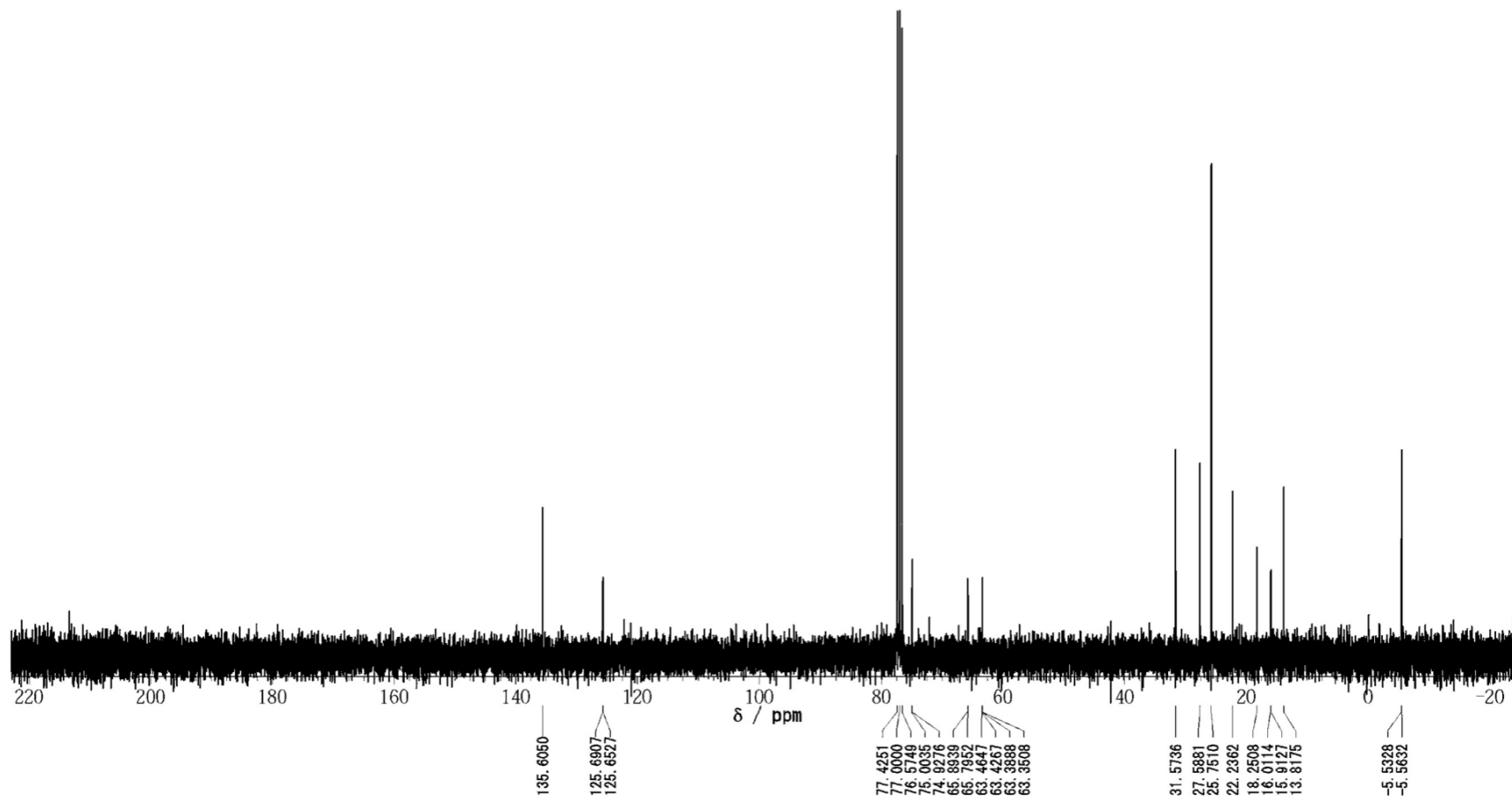
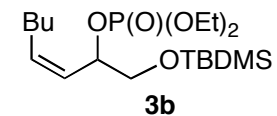
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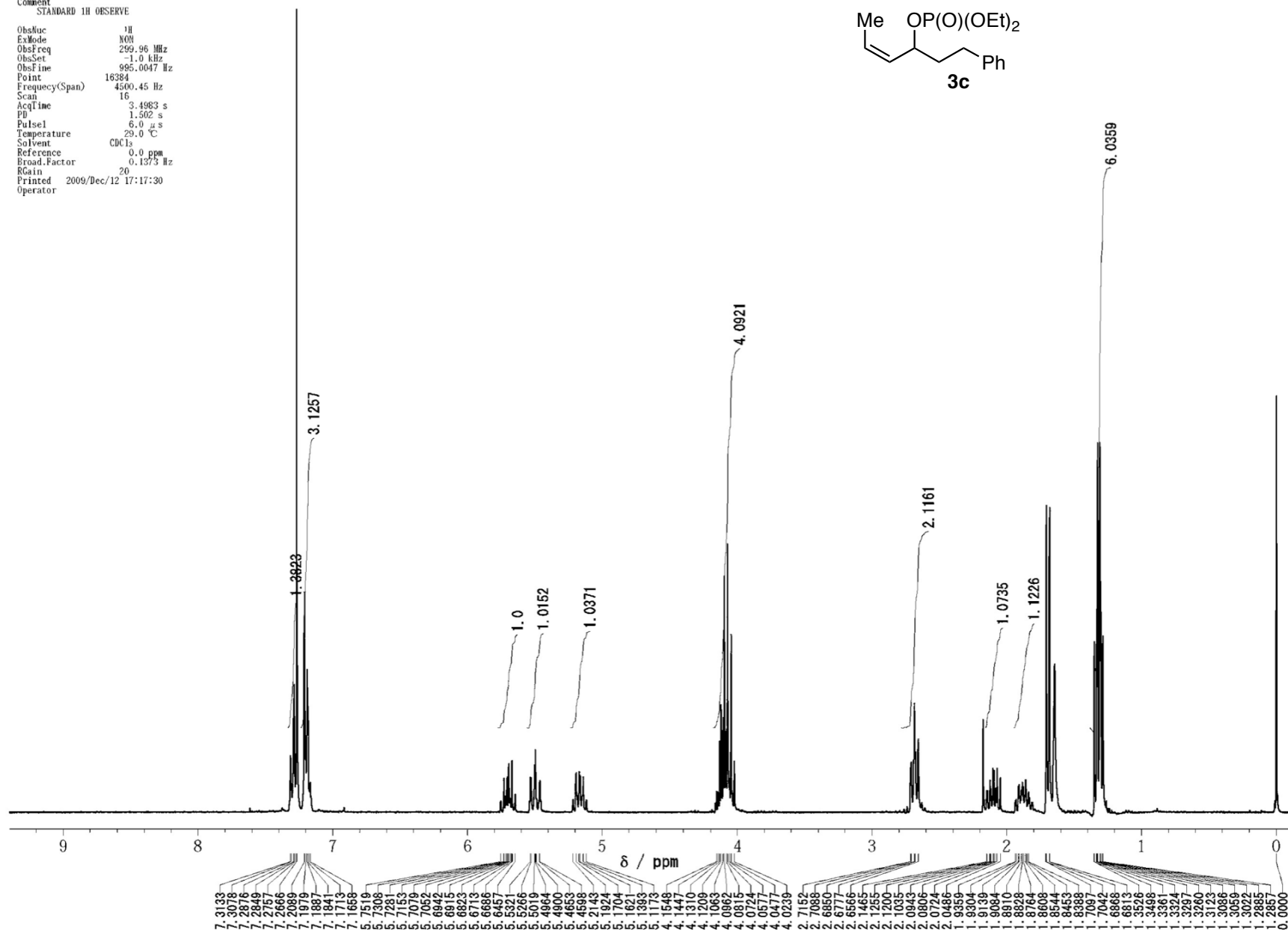
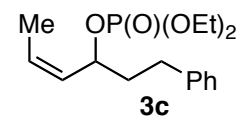
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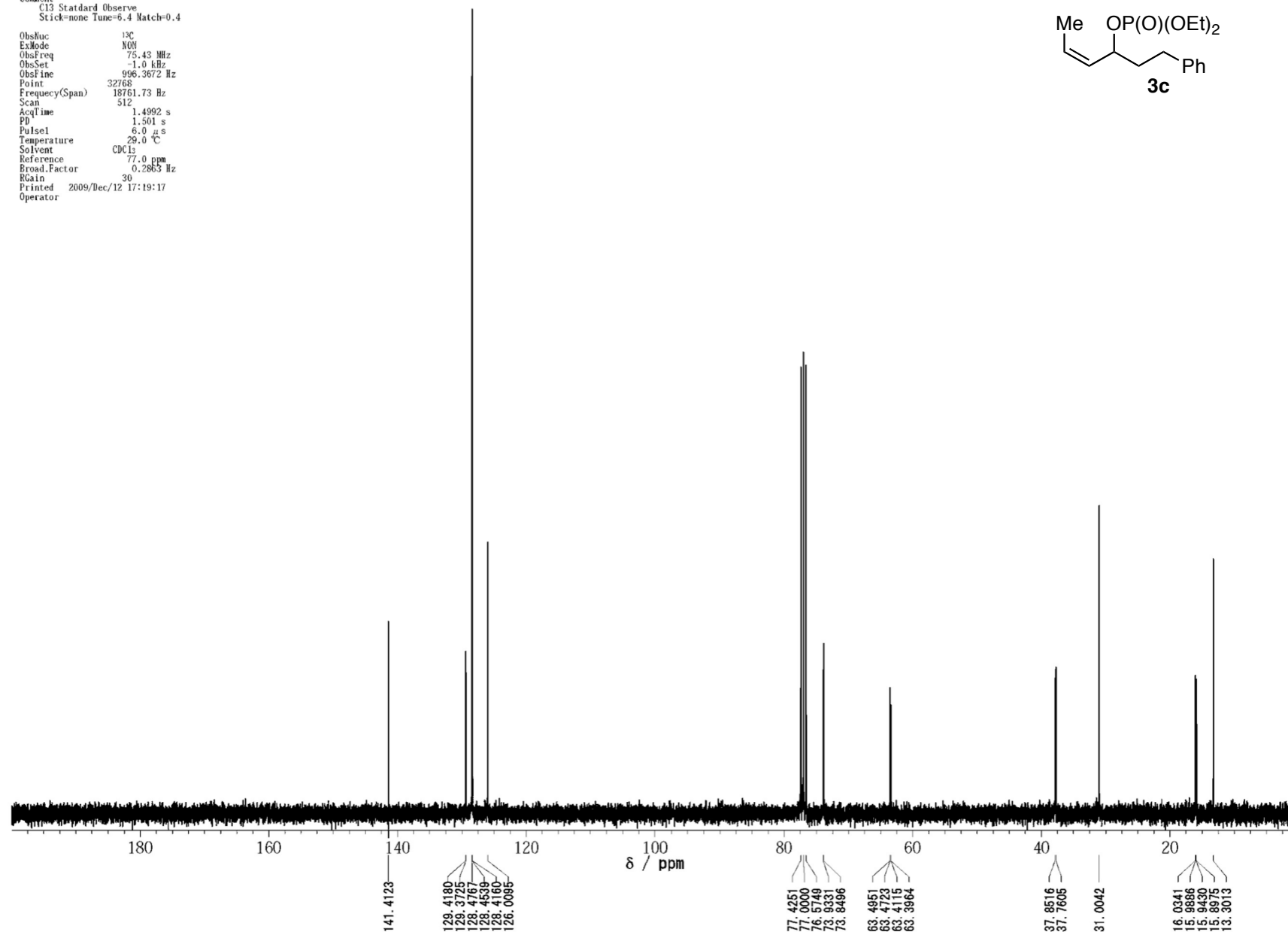
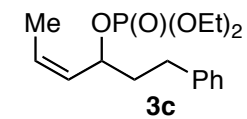
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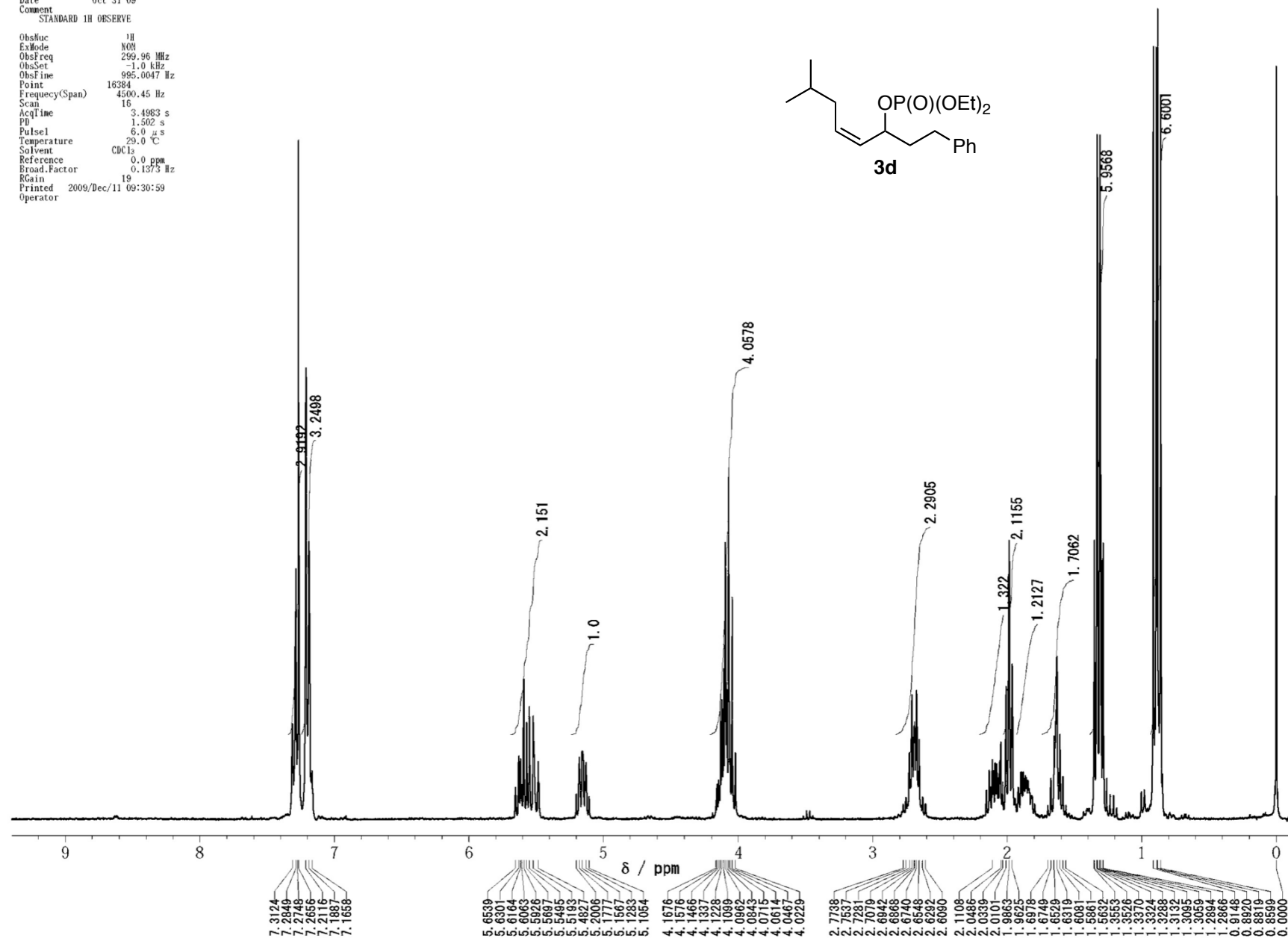
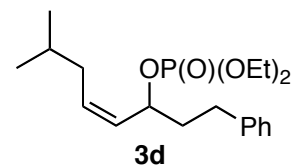
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 Freqd.Factor 0.1373 Hz
 RGain 20
 Printed 2009/Dec/12 17:17:30
 Operator



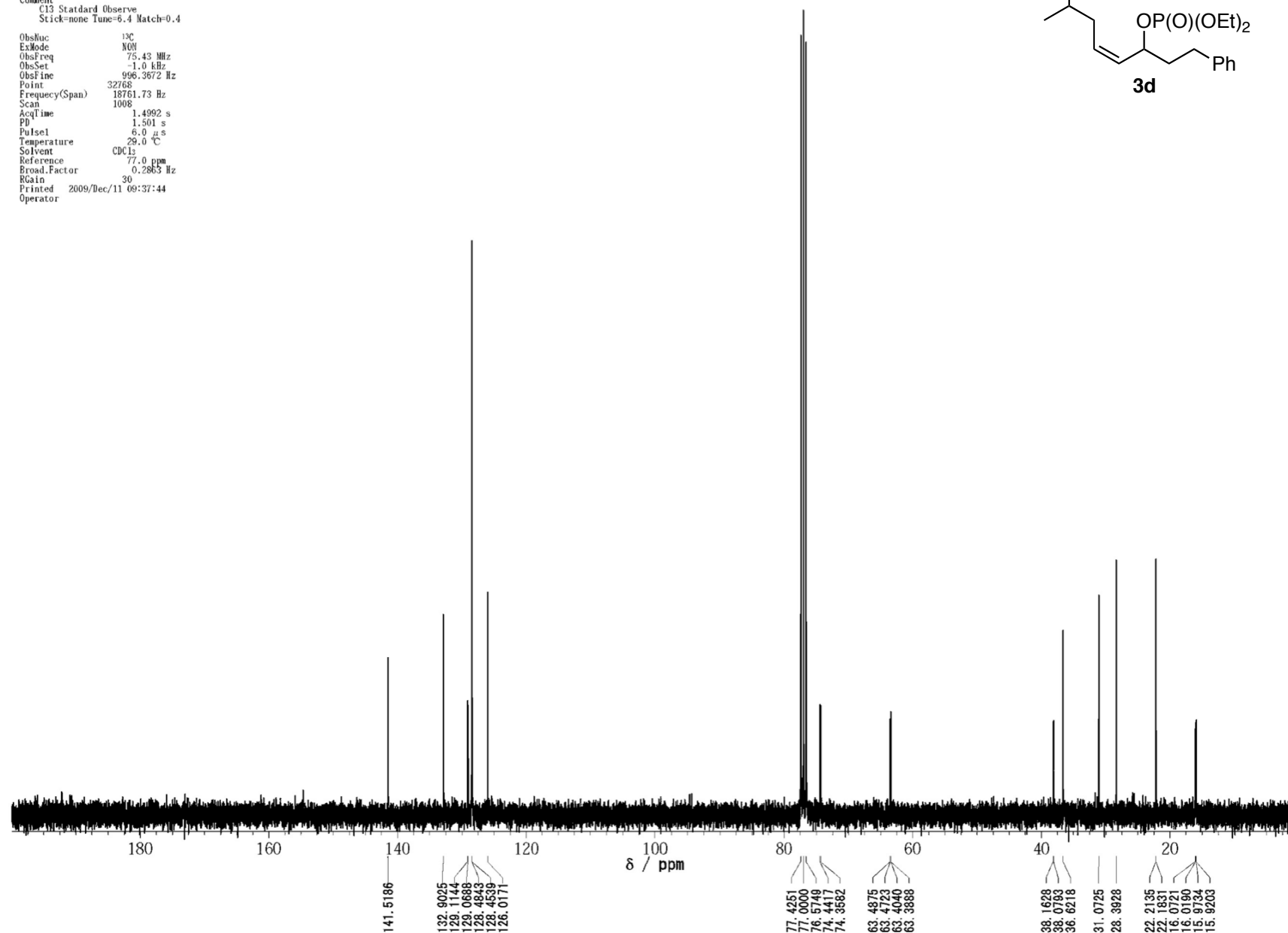
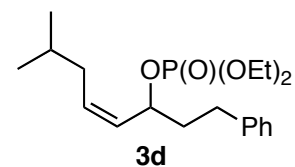
Original File:
 Date Jun 15 09
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 512
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/12 17:19:17
 Operator



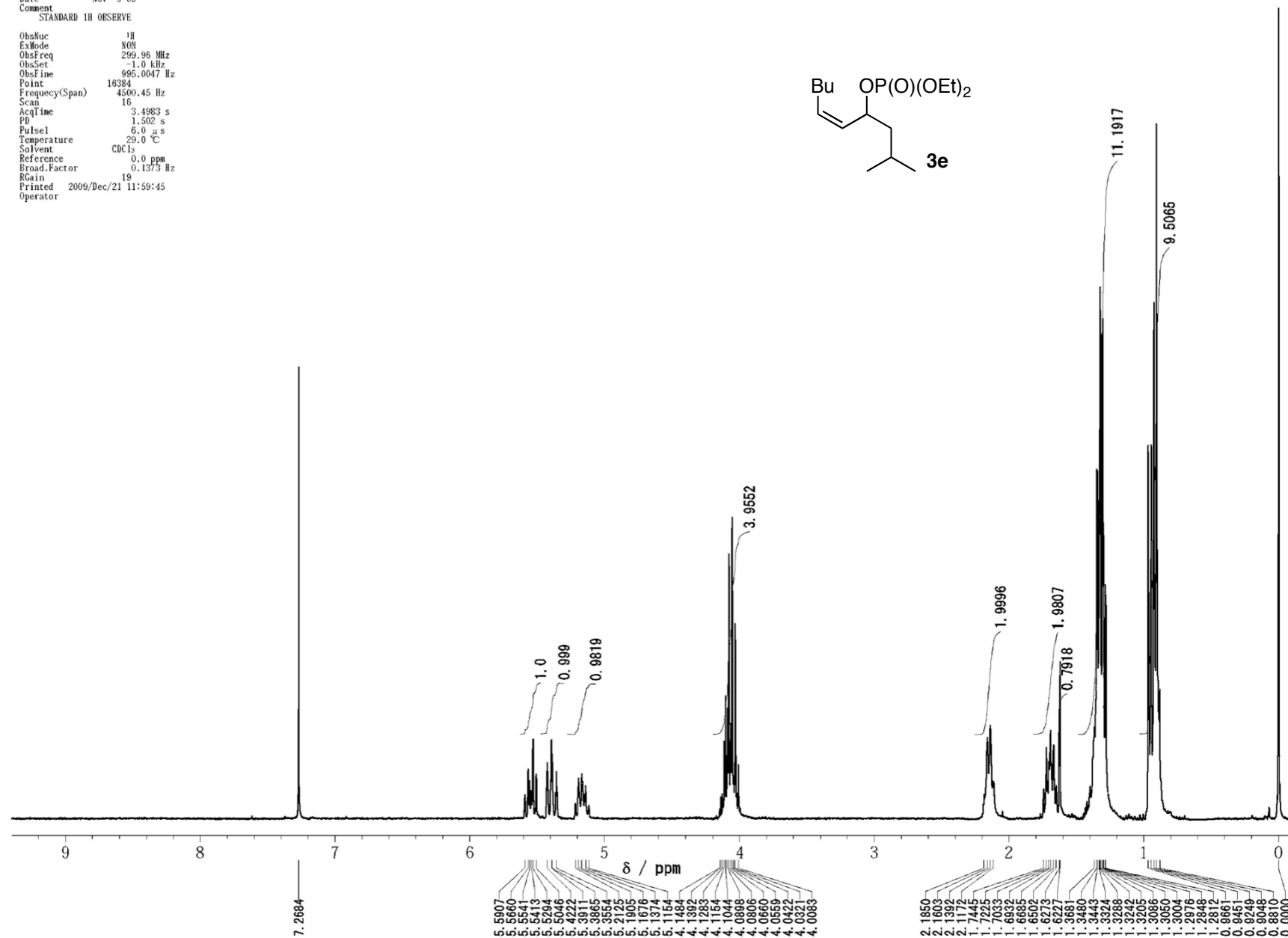
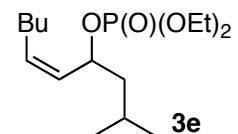
Original File:
 Date Oct 31 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 19
 Printed 2009/Dec/11 09:30:59
 Operator



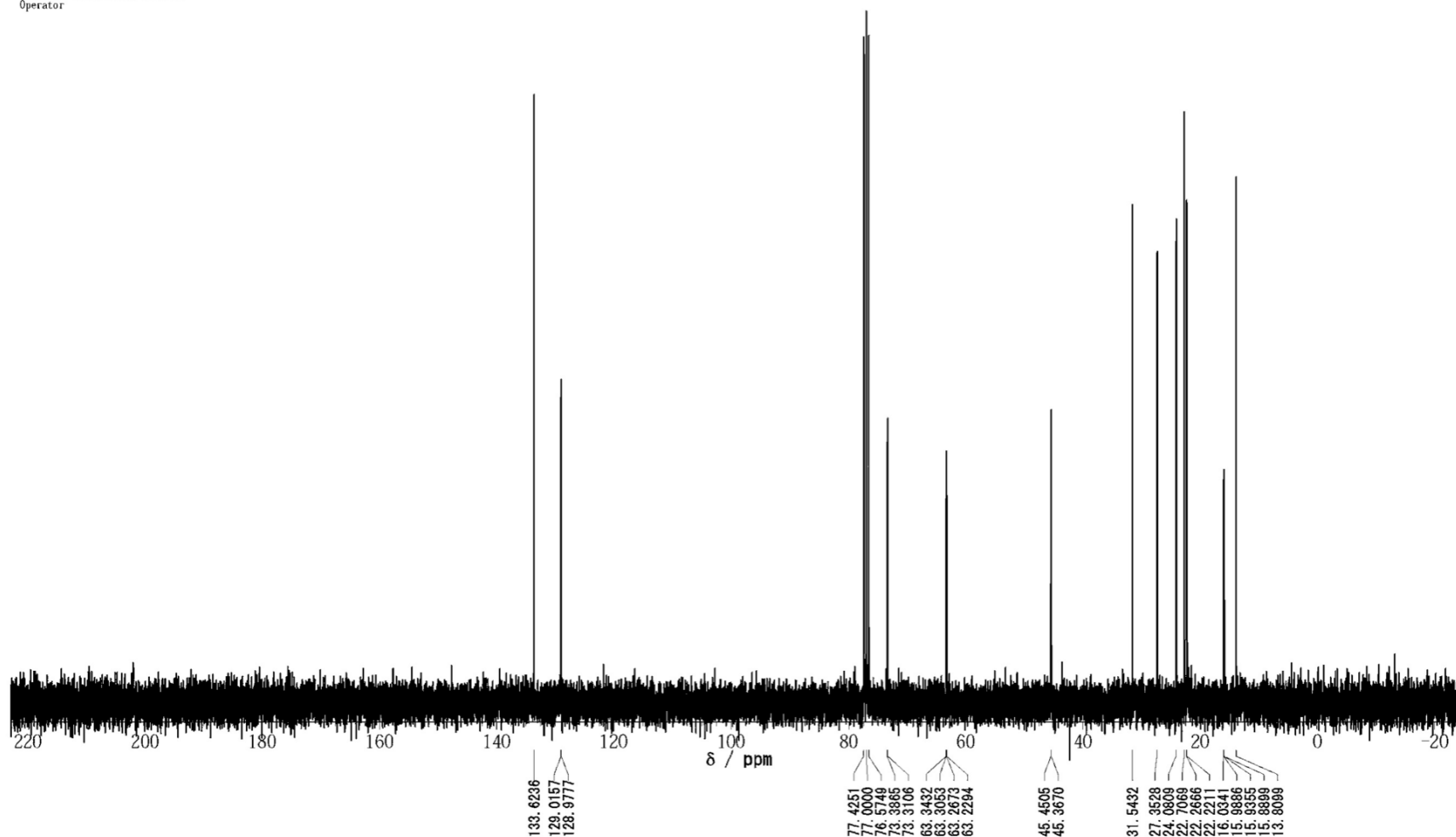
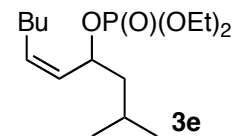
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 Comment
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 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 1008
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/11 09:37:44
 Operator



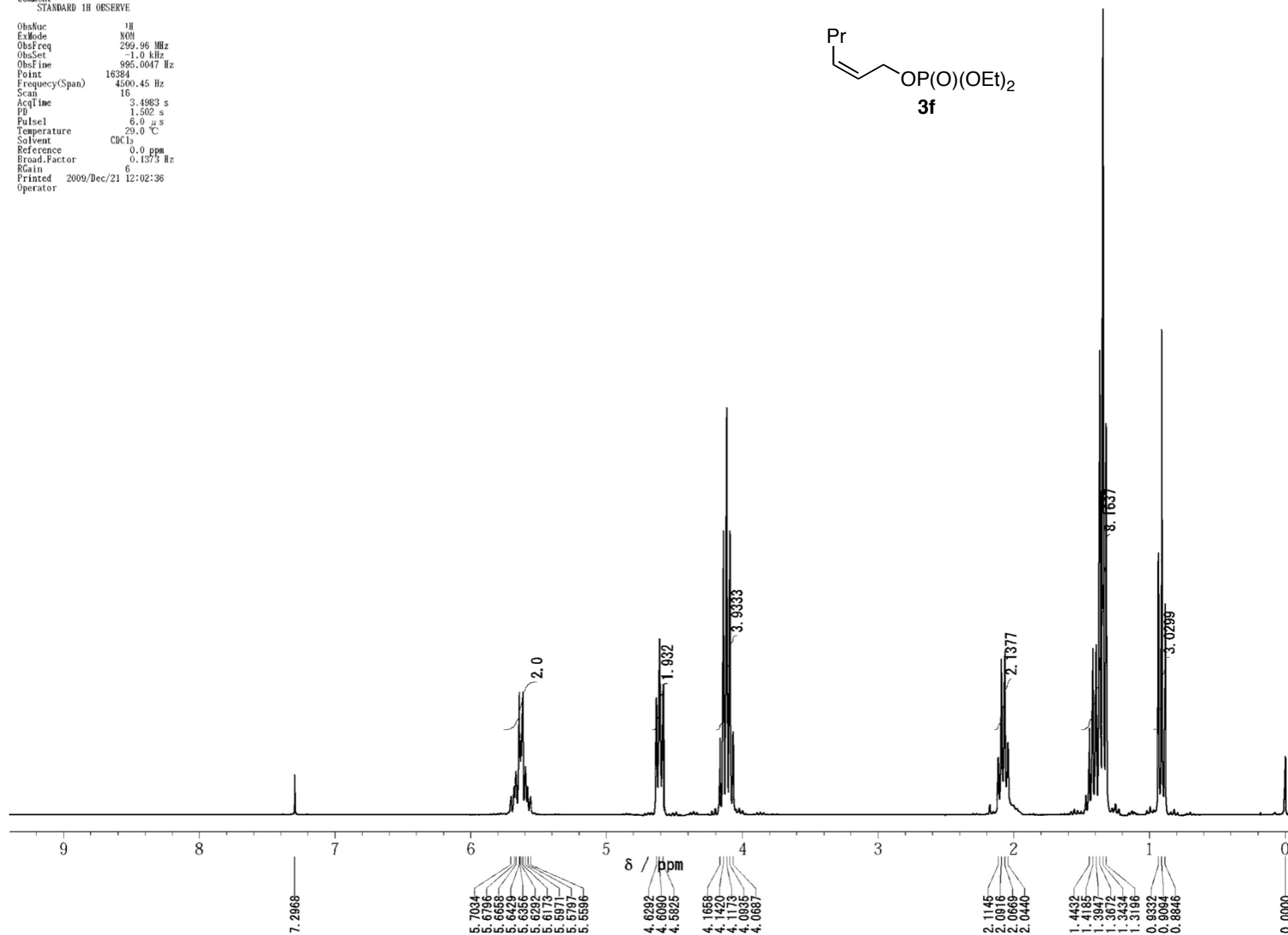
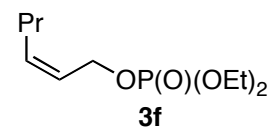
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 Date Nov 9 09
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 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 19
 Printed 2009/Dec/21 11:59:45
 Operator



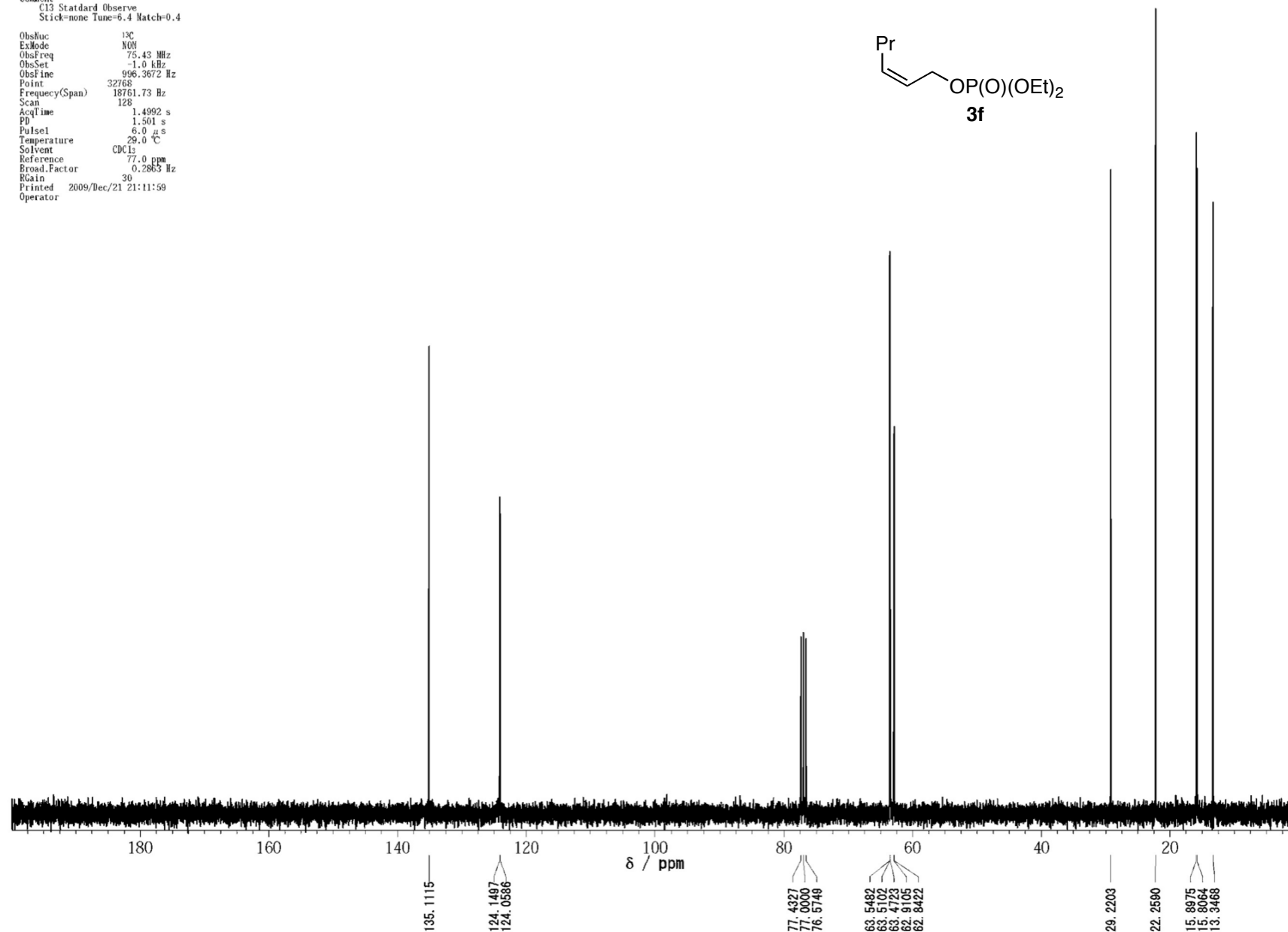
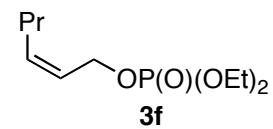
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 Date Nov 26 09
 Comment
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 ObsMuc 13C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 256
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 20:37:38
 Operator



Original File:
 Date Jun 3 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 6
 Printed 2009/Dec/21 12:02:36
 Operator

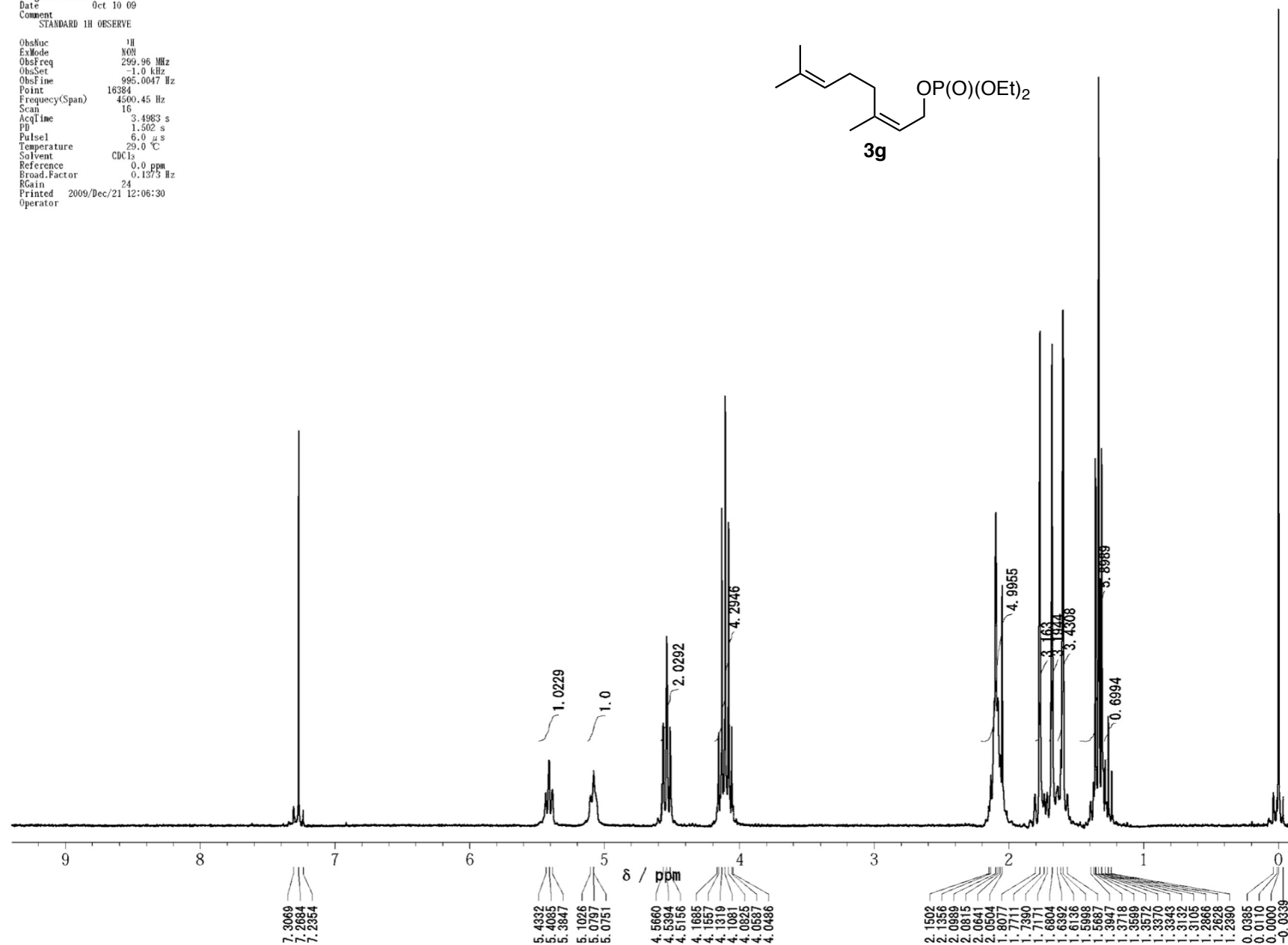
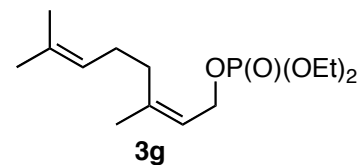


Original File:
 Date Jun 5 09
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 C13 Standard Observe
 Stick-none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 21:11:59
 Operator

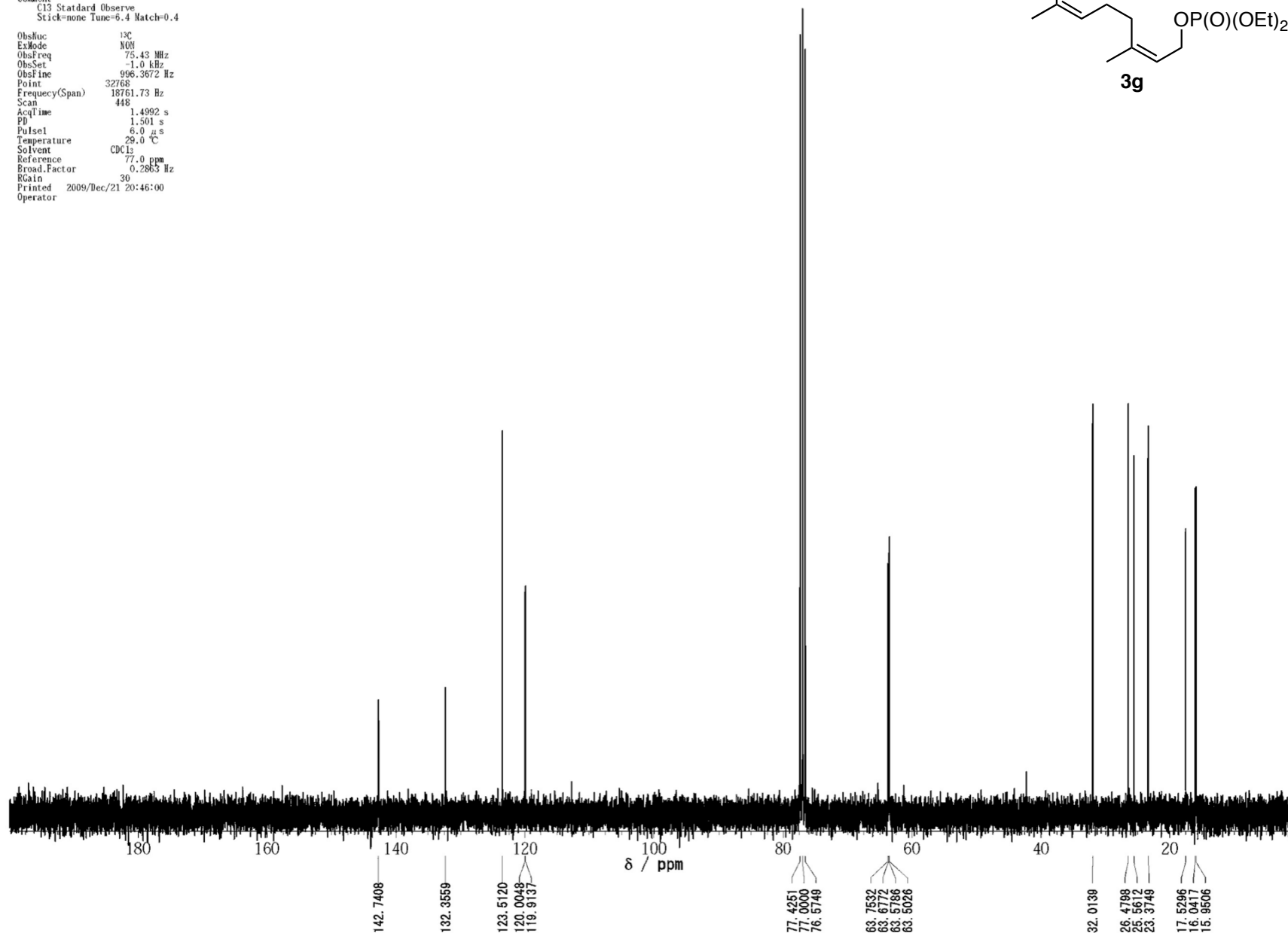
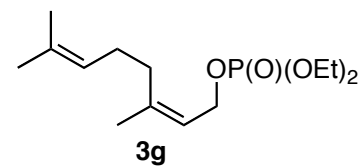


Original File:
 Date Oct 10 09
 Comment
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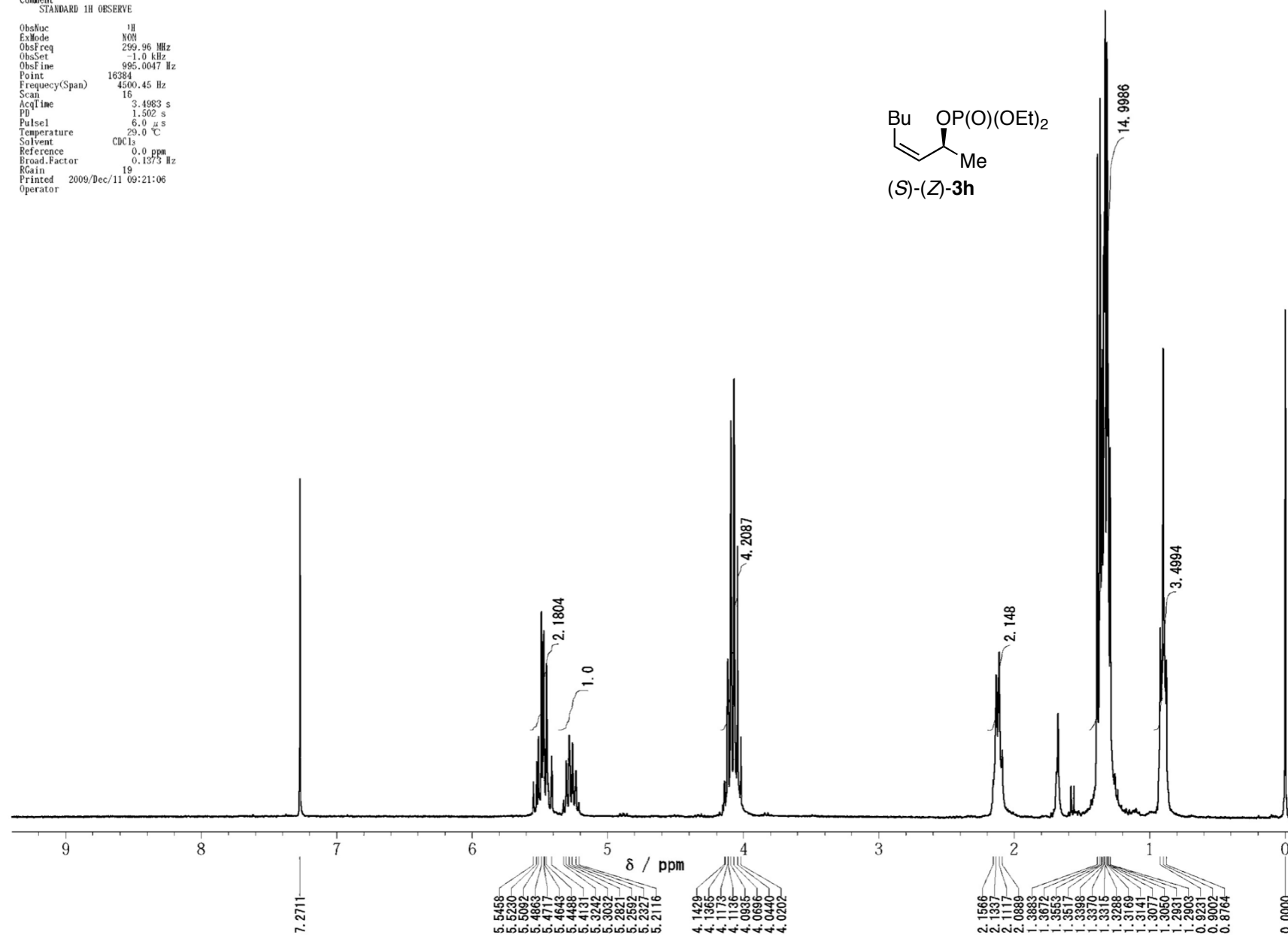
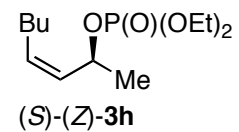
ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 24
 Printed 2009/Dec/21 12:06:30
 Operator



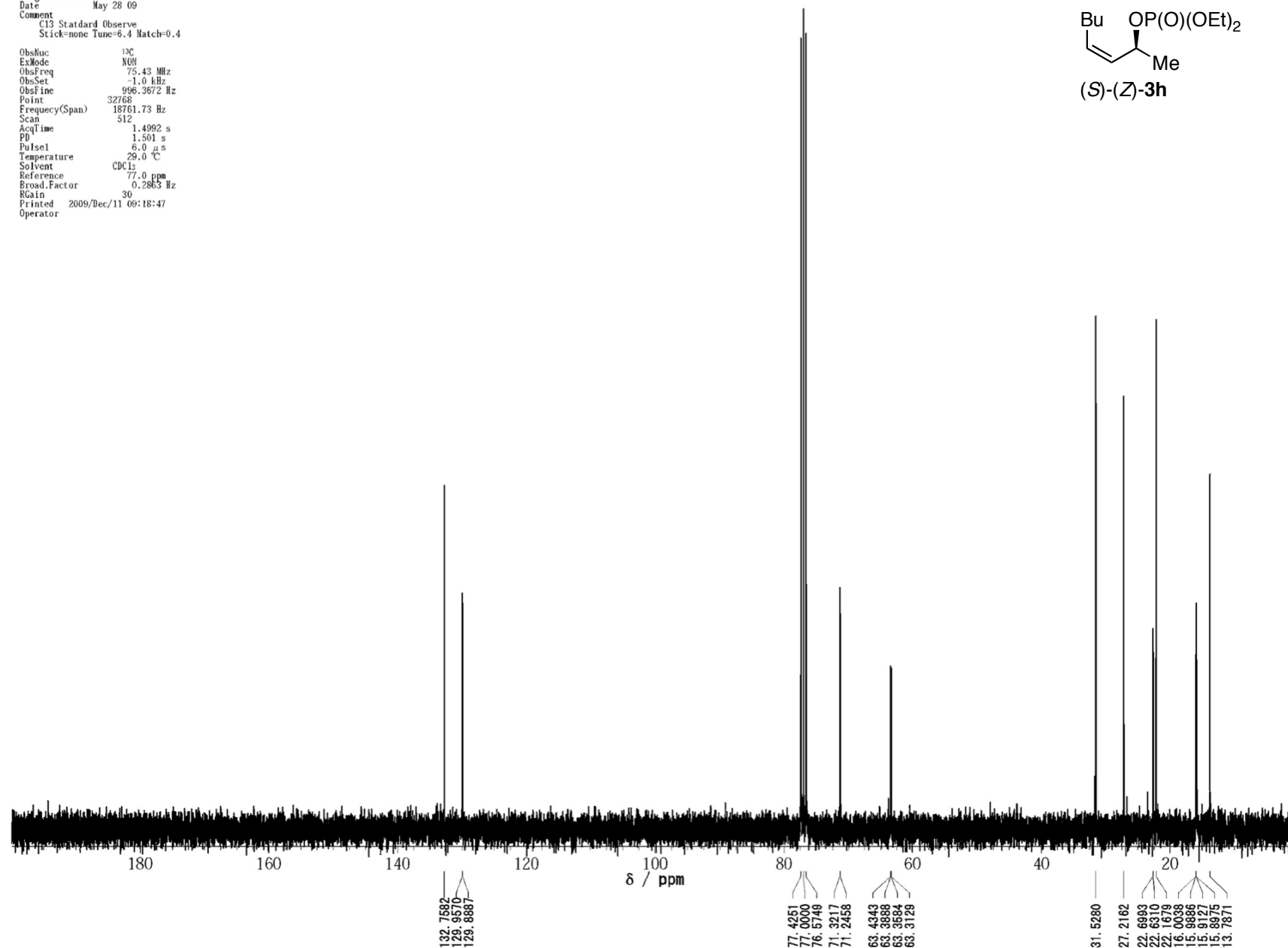
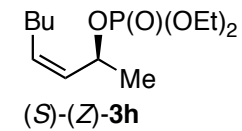
Original File:
 Date Nov 26 09
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 448
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 20:46:00
 Operator



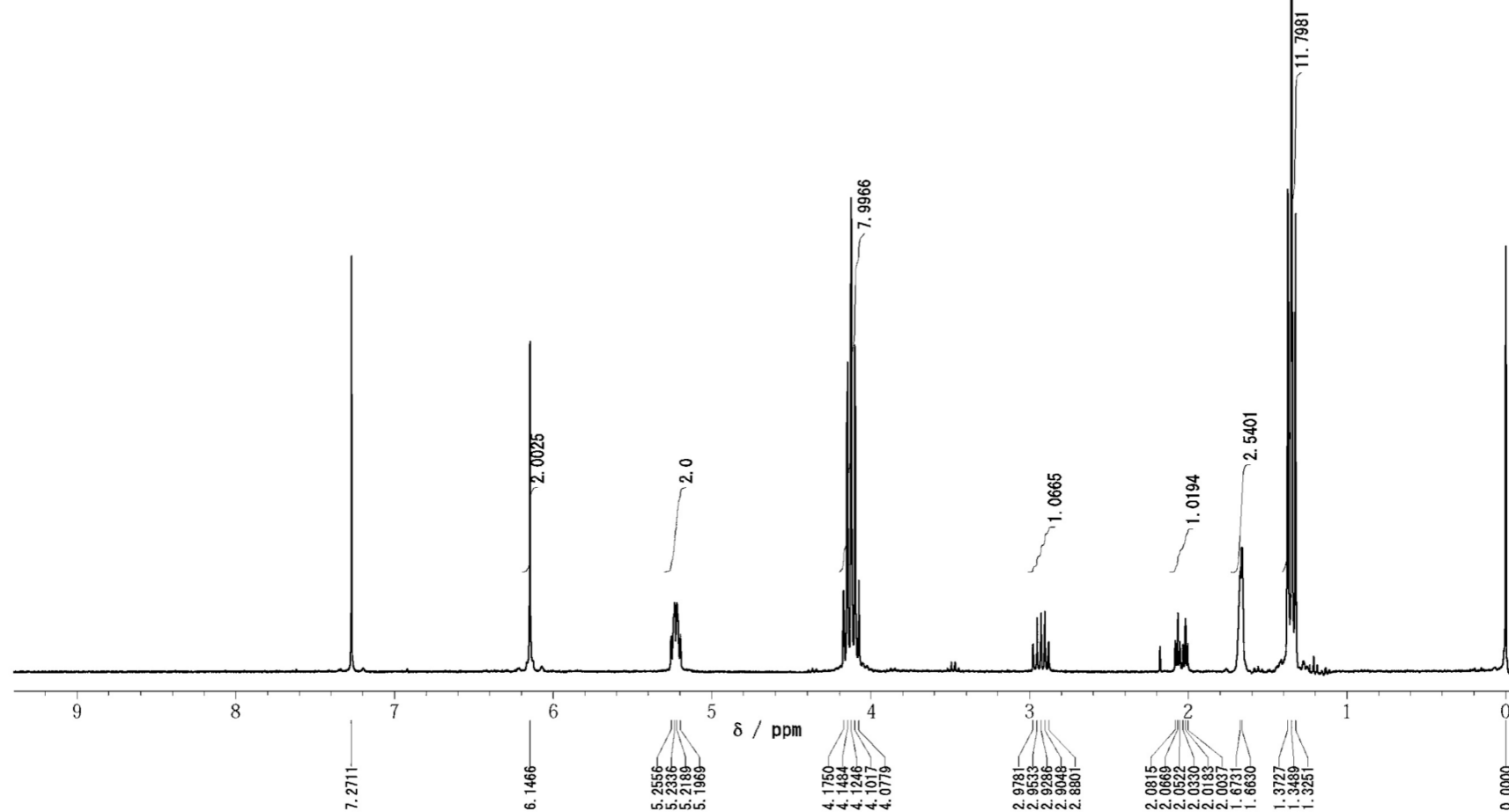
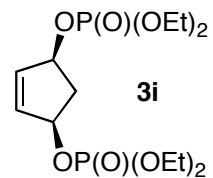
Original File:
 Date Sep 16 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 0.0 ppm
 Froad.Factor 0.1373 Hz
 RGain 19
 Printed 2009/Dec/11 09:21:06
 Operator



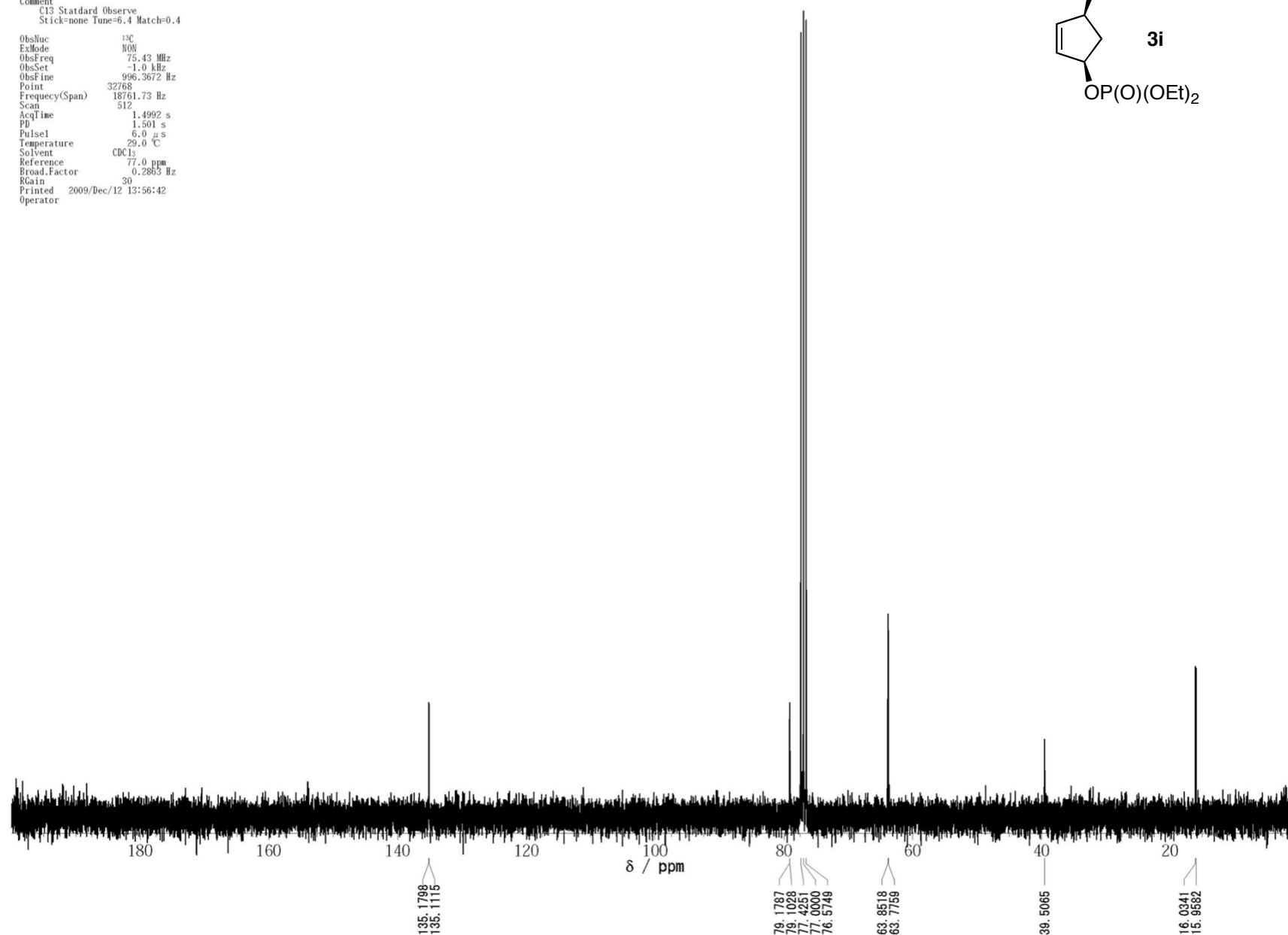
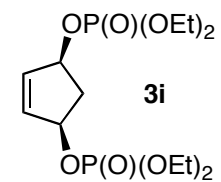
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 Date May 28 09
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 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 512
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/11 09:18:47
 Operator



Original File:
 Date Jun 10 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Froad.Factor 0.1373 Hz
 RGain 22
 Printed 2009/Dec/12 13:54:50
 Operator

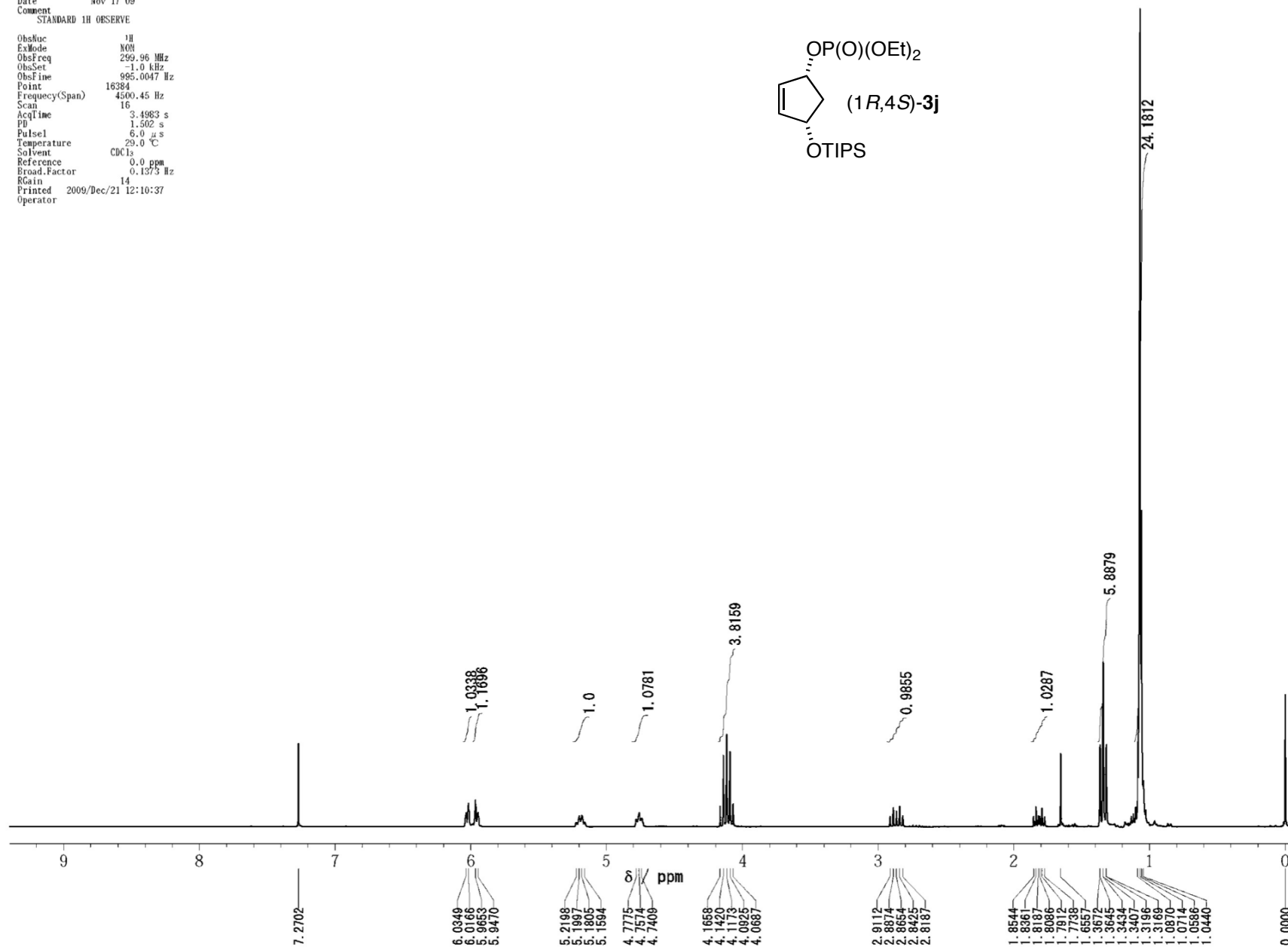
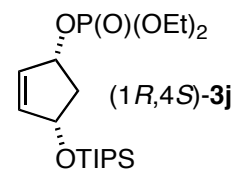


Original File:
 Date Jun 15 09
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 ObsNuc ¹³C
 ExMode R0N
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 512
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/12 13:56:42
 Operator

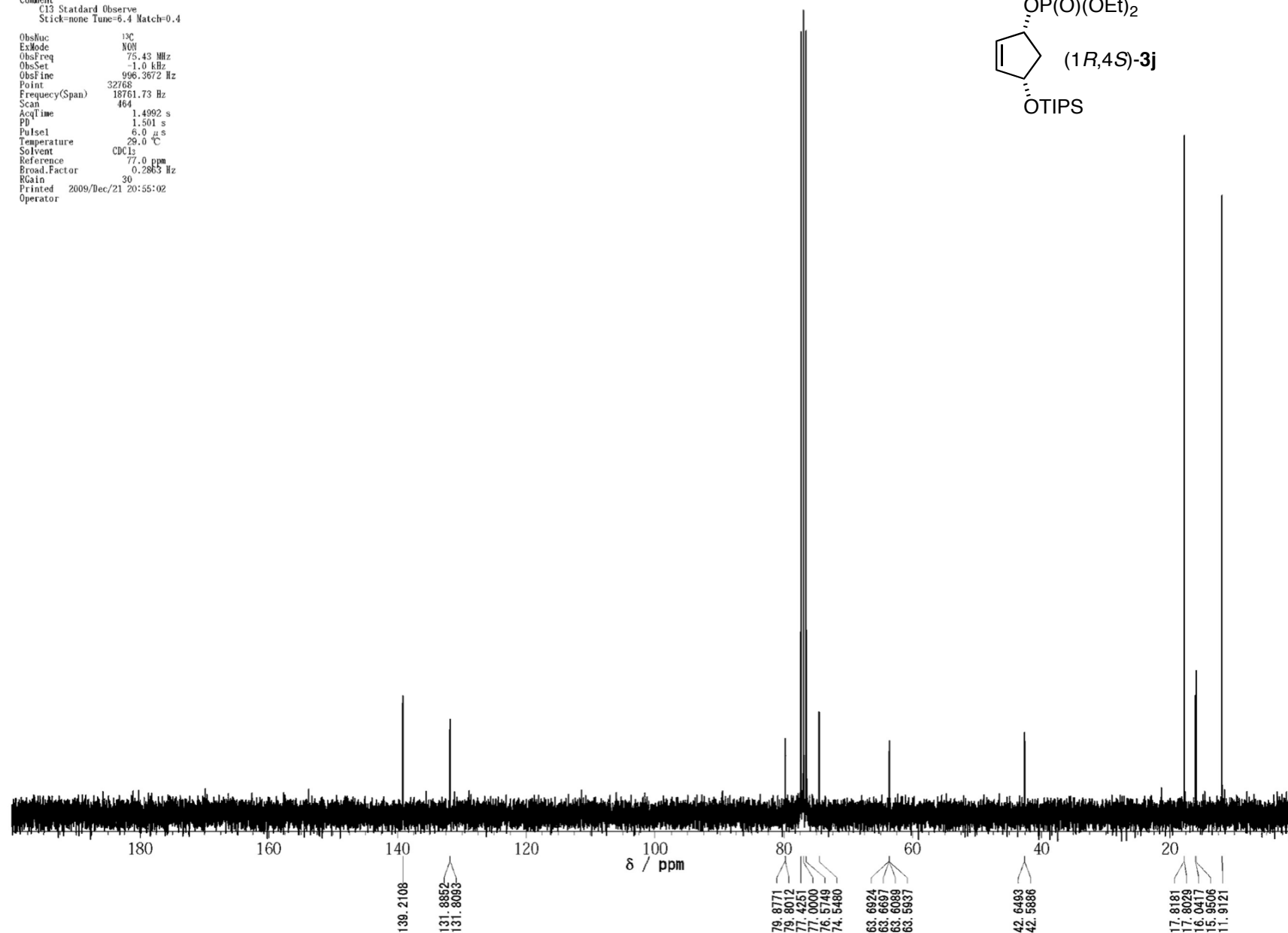
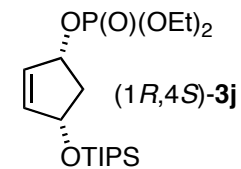


Original File:
 Date Nov 17 09
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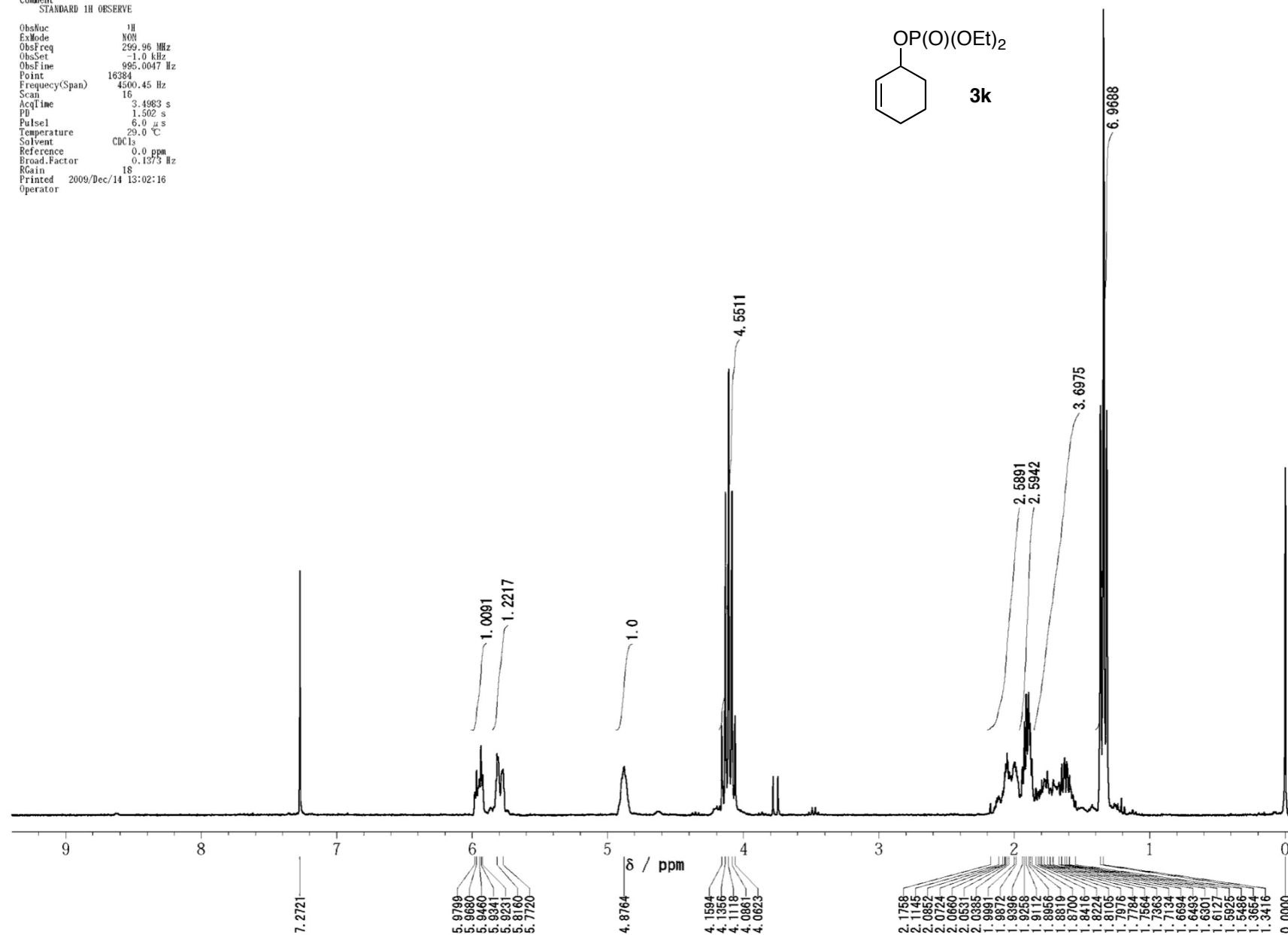
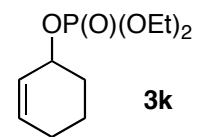
ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 14
 Printed 2009/Dec/21 12:10:37
 Operator



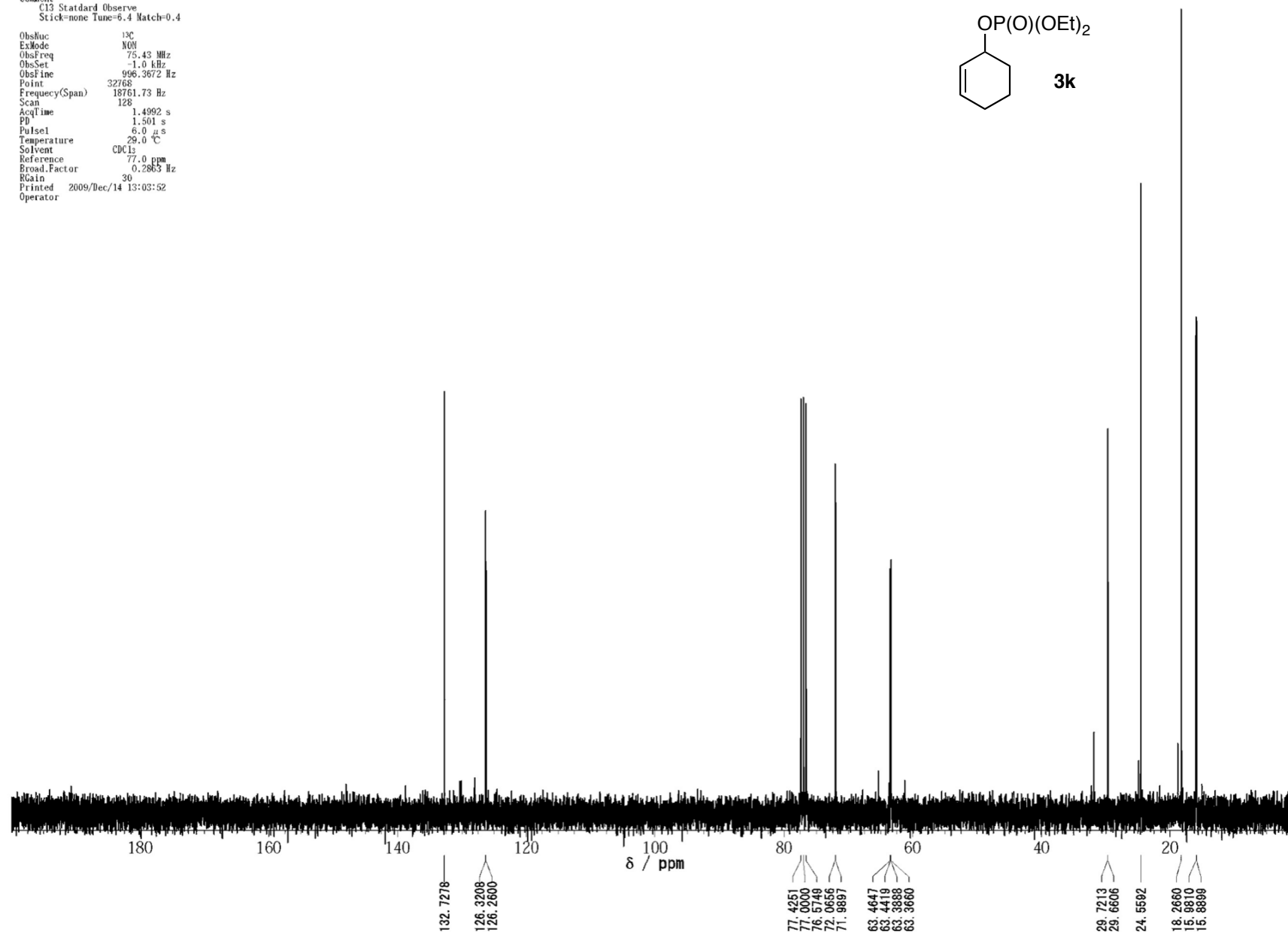
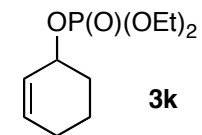
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 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 464
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 20:55:02
 Operator



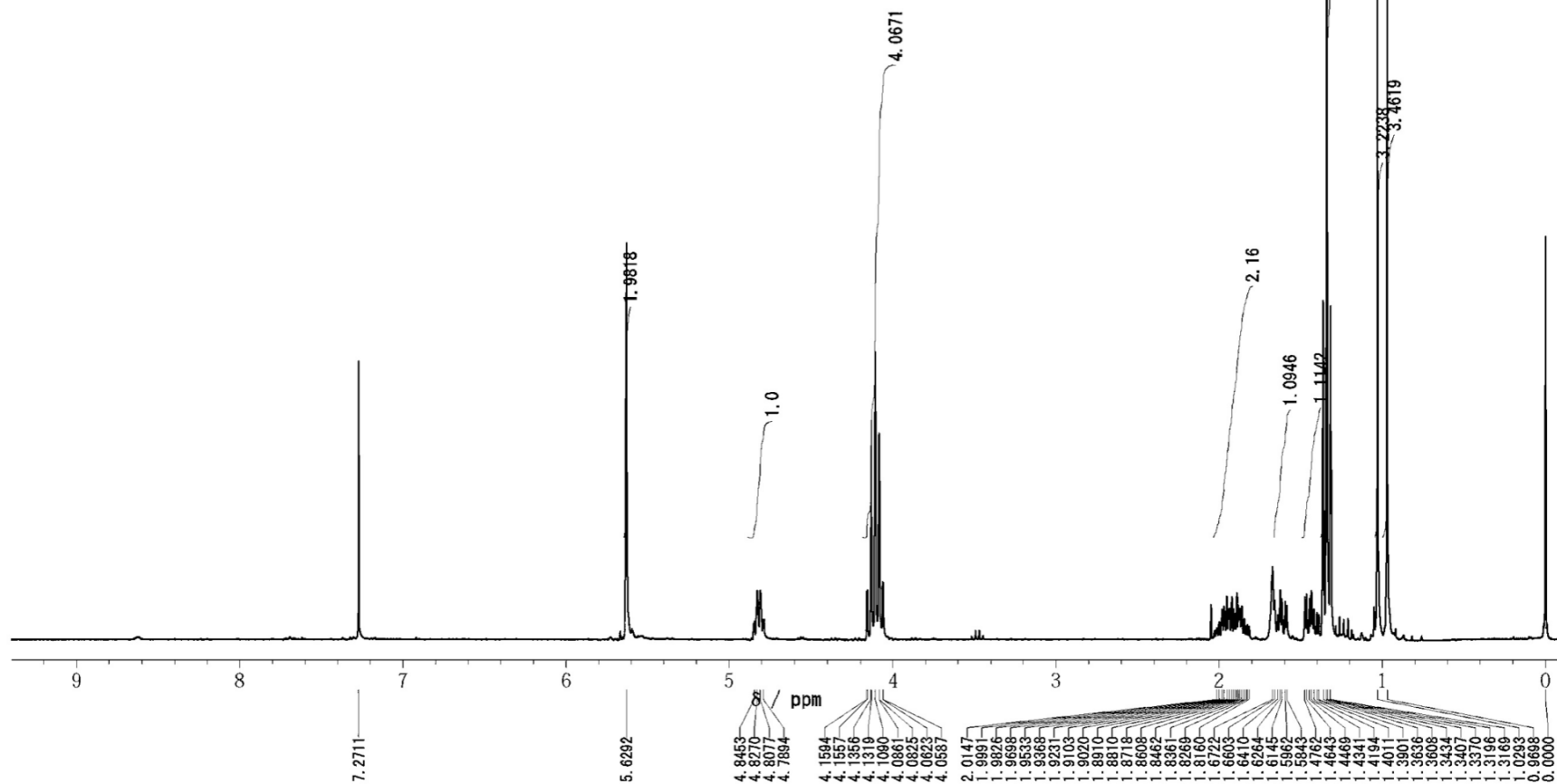
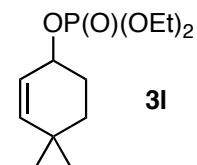
Original File:
 Date Sep 15 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 18
 Printed 2009/Dec/14 13:02:16
 Operator



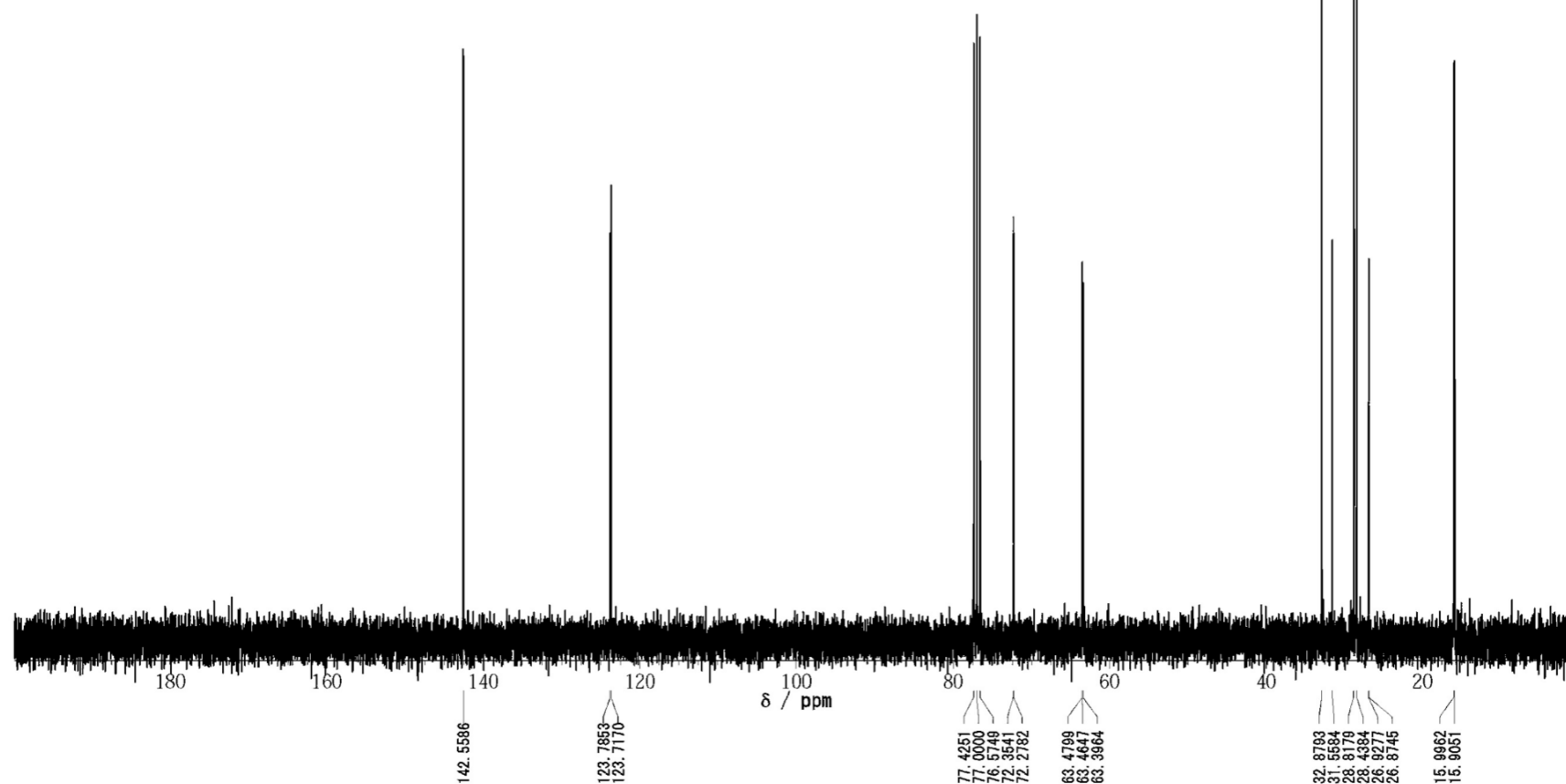
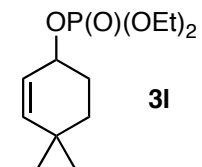
Original File:
 Date Sep 15 09
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 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/14 13:03:52
 Operator



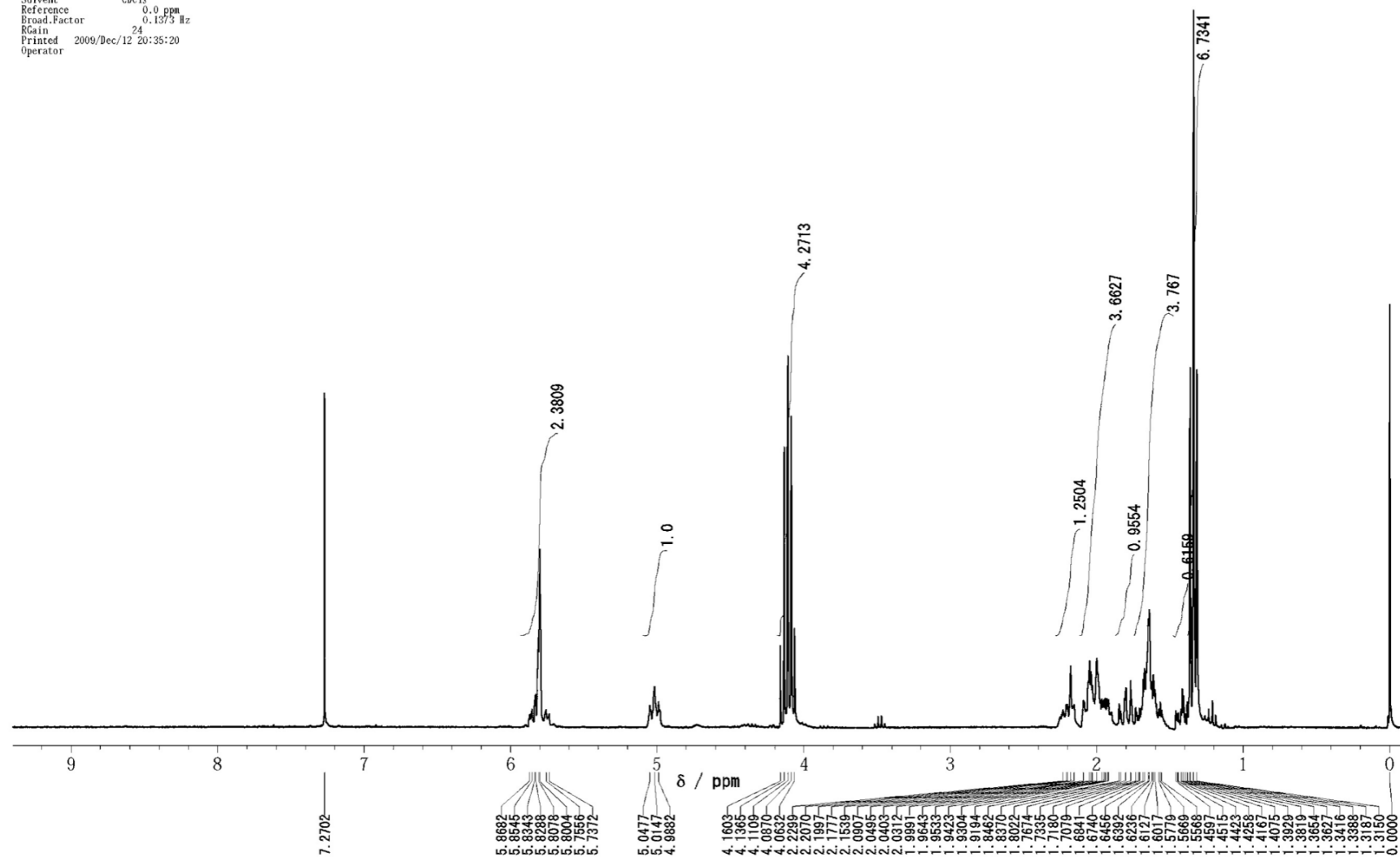
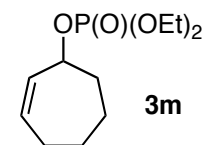
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 Comment
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 ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 19
 Printed 2009/Dec/17 09:33:16
 Operator



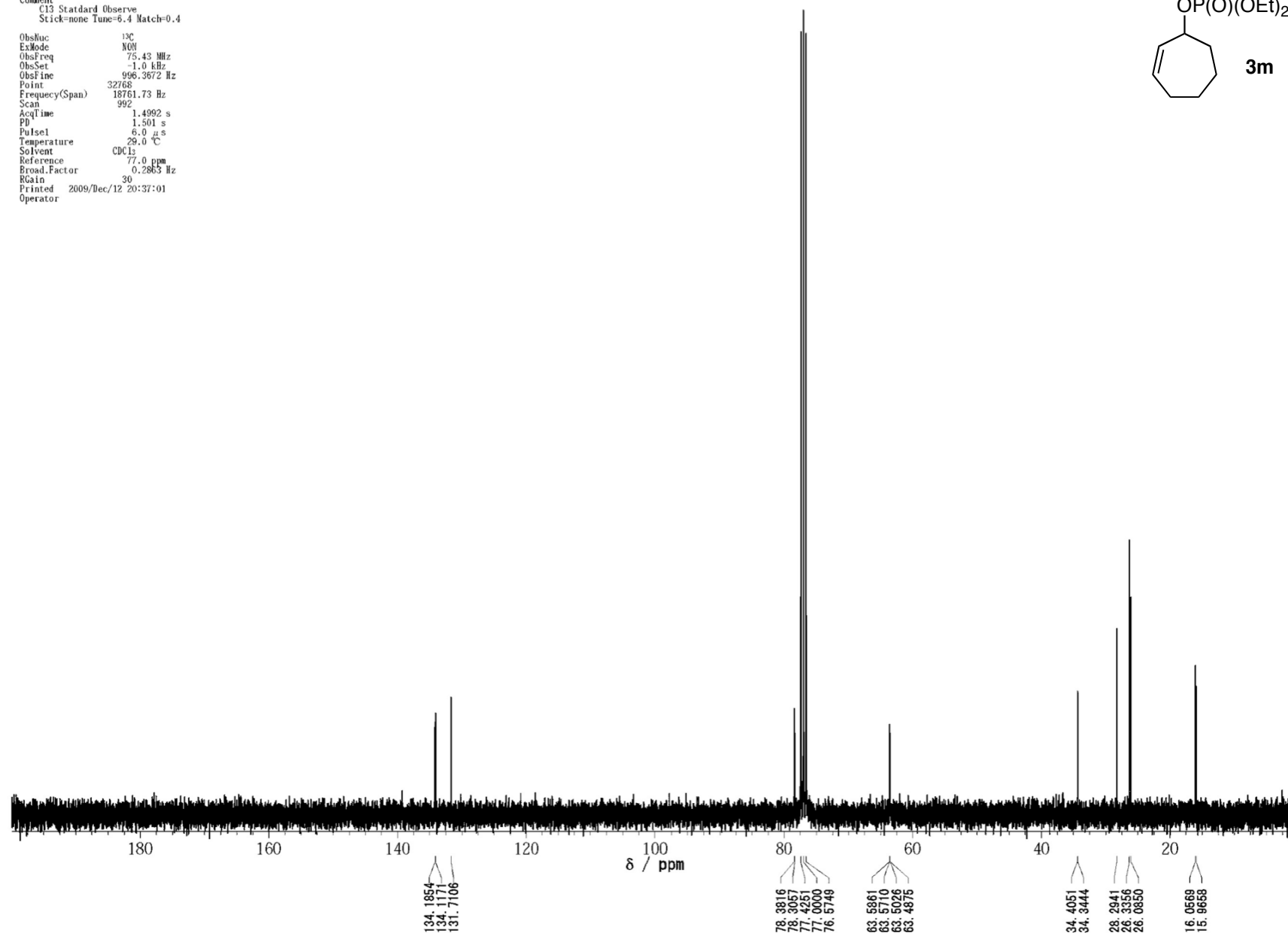
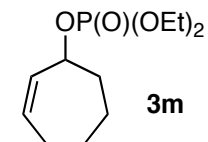
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 Date Aug 27 09
 Comment
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 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/17 09:35:40
 Operator



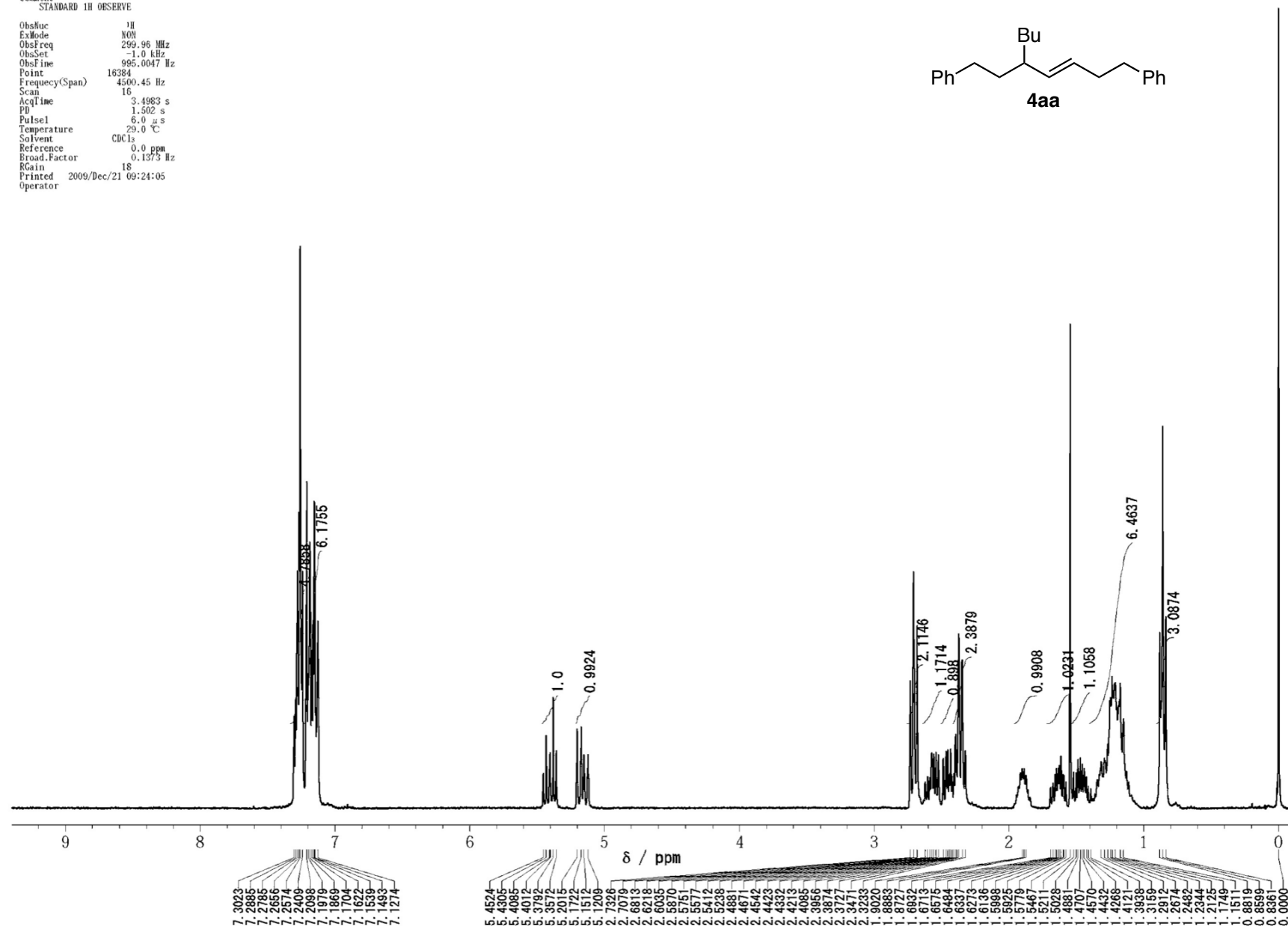
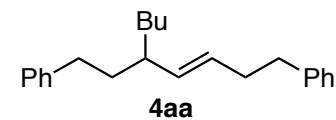
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 Date Oct 16 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 24
 Printed 2009/Dec/12 20:35:20
 Operator



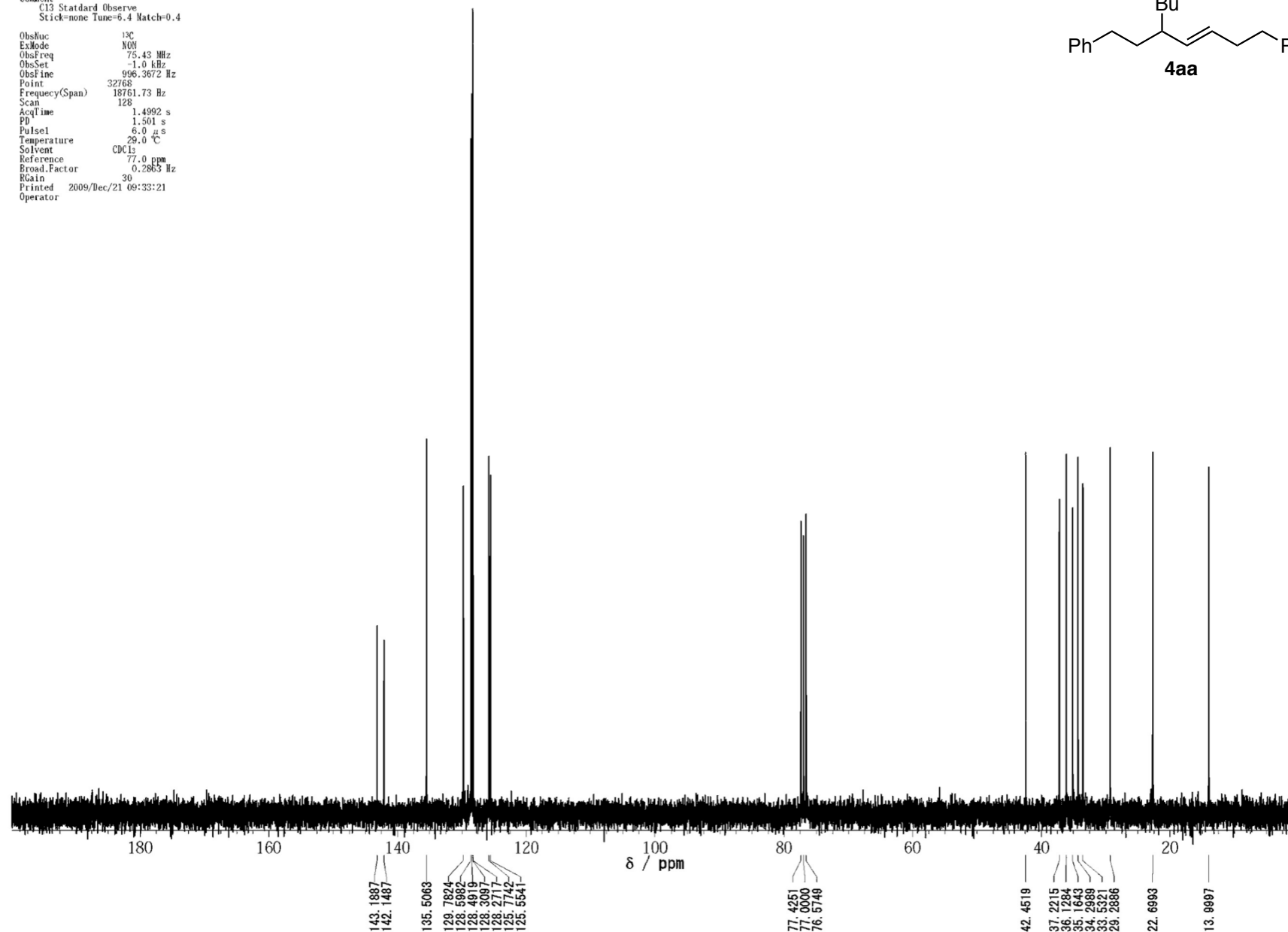
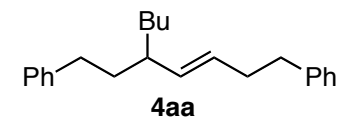
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 Date Nov 25 09
 Comment
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 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 992
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/12 20:37:01
 Operator



Original File:
 Date Aug 31 09
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 18
 Printed 2009/Dec/21 09:24:05
 Operator

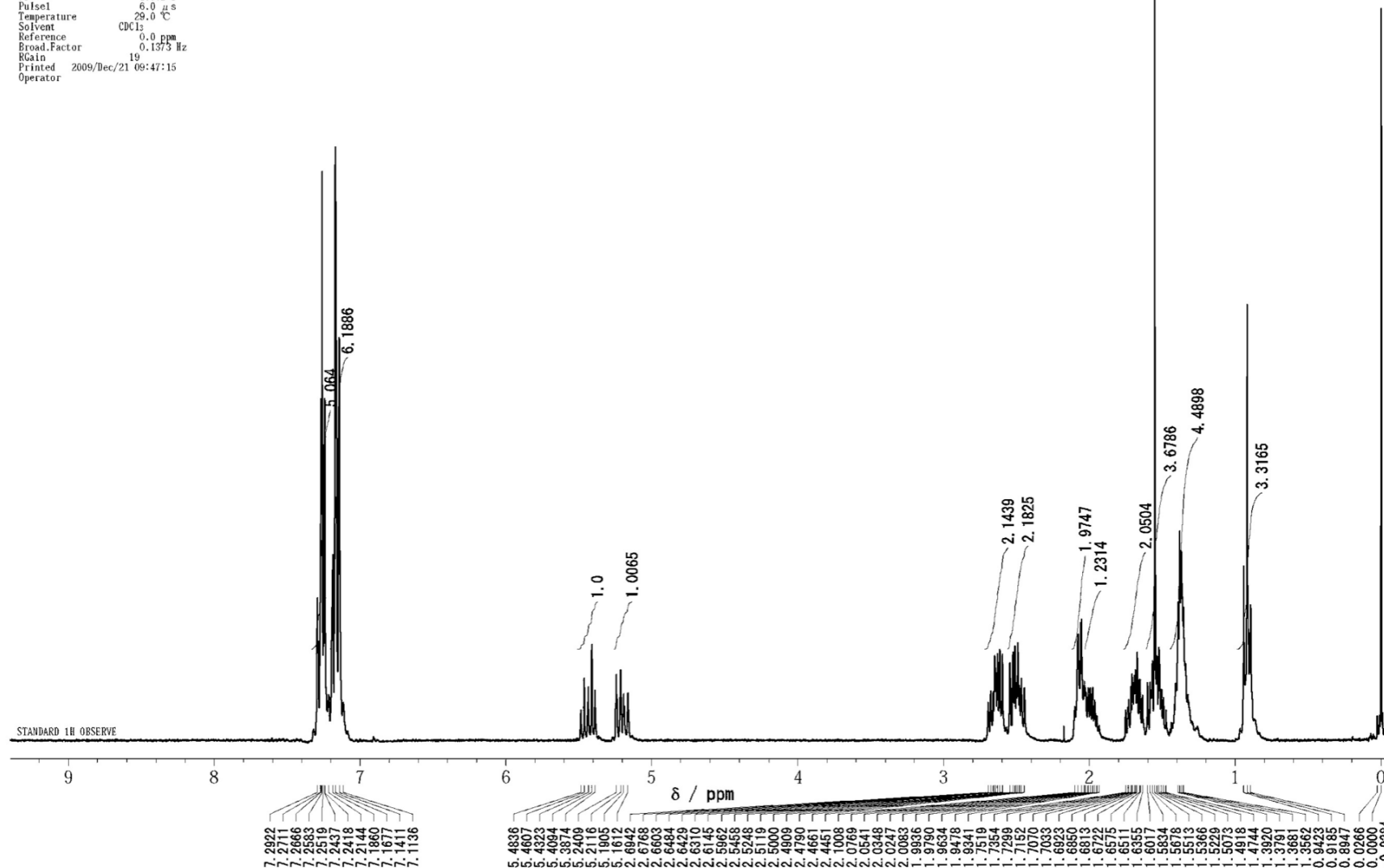
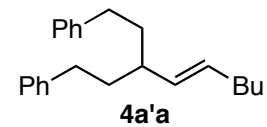


Original File:
 Date Aug 31 09
 Comment
 C13 Standard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 09:33:21
 Operator

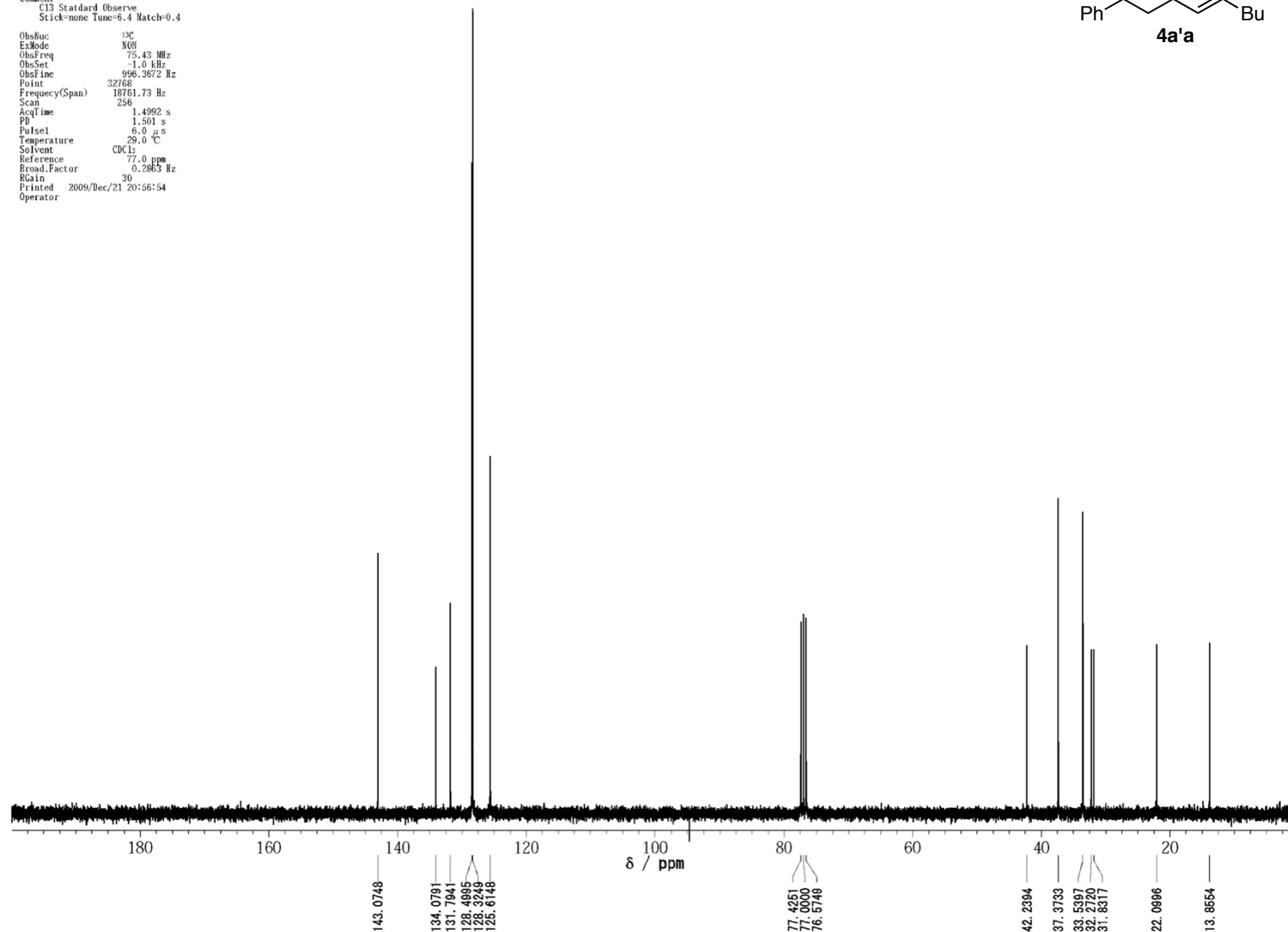
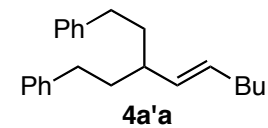


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 Original File:
 Date Oct 27 09
 Comment STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet 1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 19
 Printed 2009/Dec/21 09:47:15
 Operator



Original File:
 Date Oct 27 09
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 ExMode NON
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 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 256
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 20:56:54
 Operator



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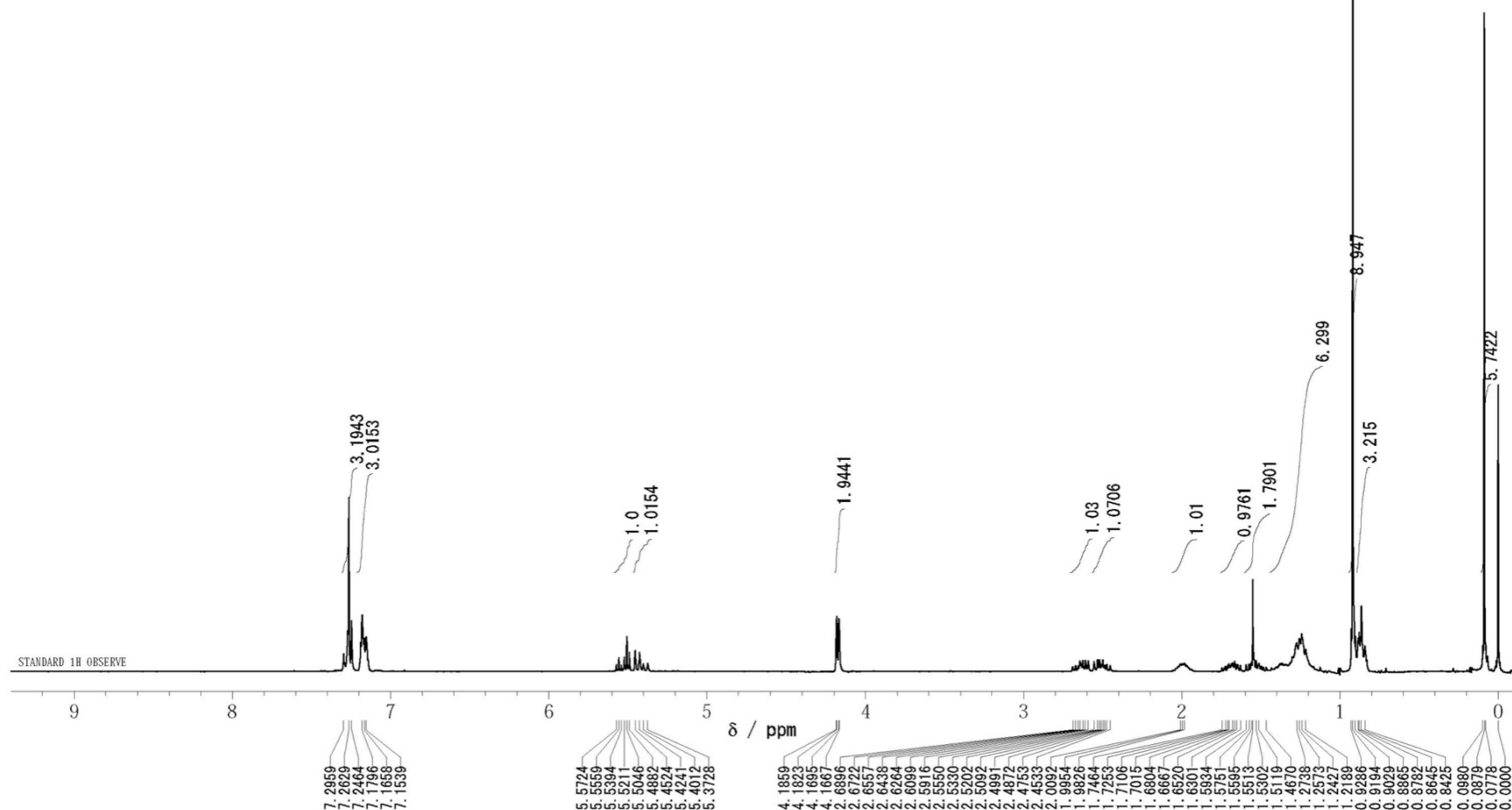
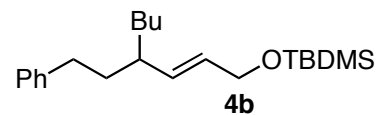
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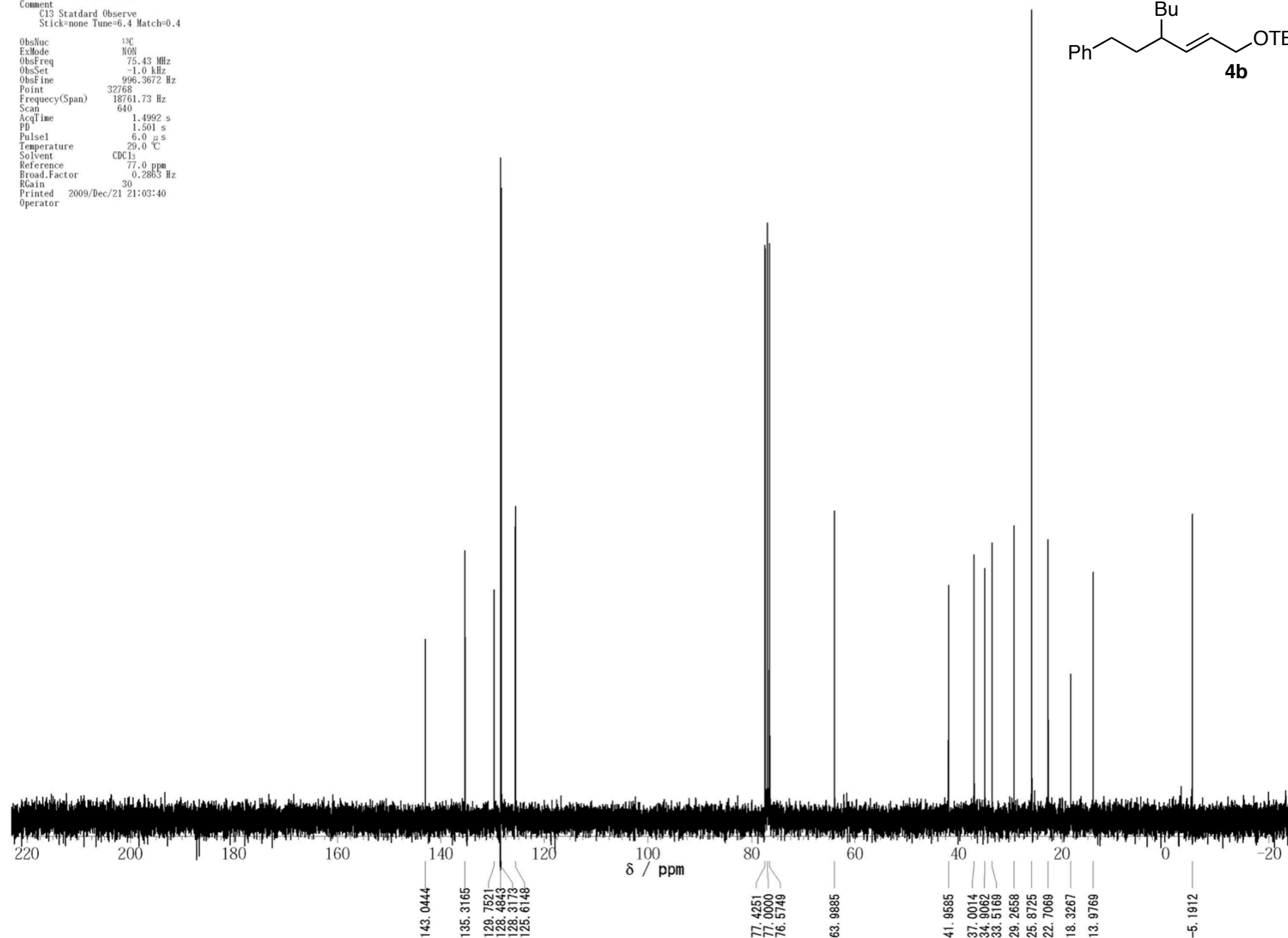
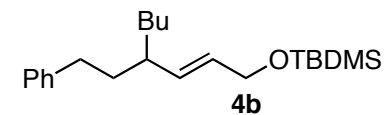
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STANDARD 1H OBSERVE

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ObsSet -1.0 kHz
ObsFine 995.0047 Hz
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Frequency(Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse1 6.0 μs
Temperature 29.0 °C
Solvent CDCl₃
Reference 0.0 ppm
Broad.Factor 0.1373 Hz
RGain 24
Printed 2009/Dec/21 09:55:54
Operator



Original File:
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 Frequency(Span) 18761.73 Hz
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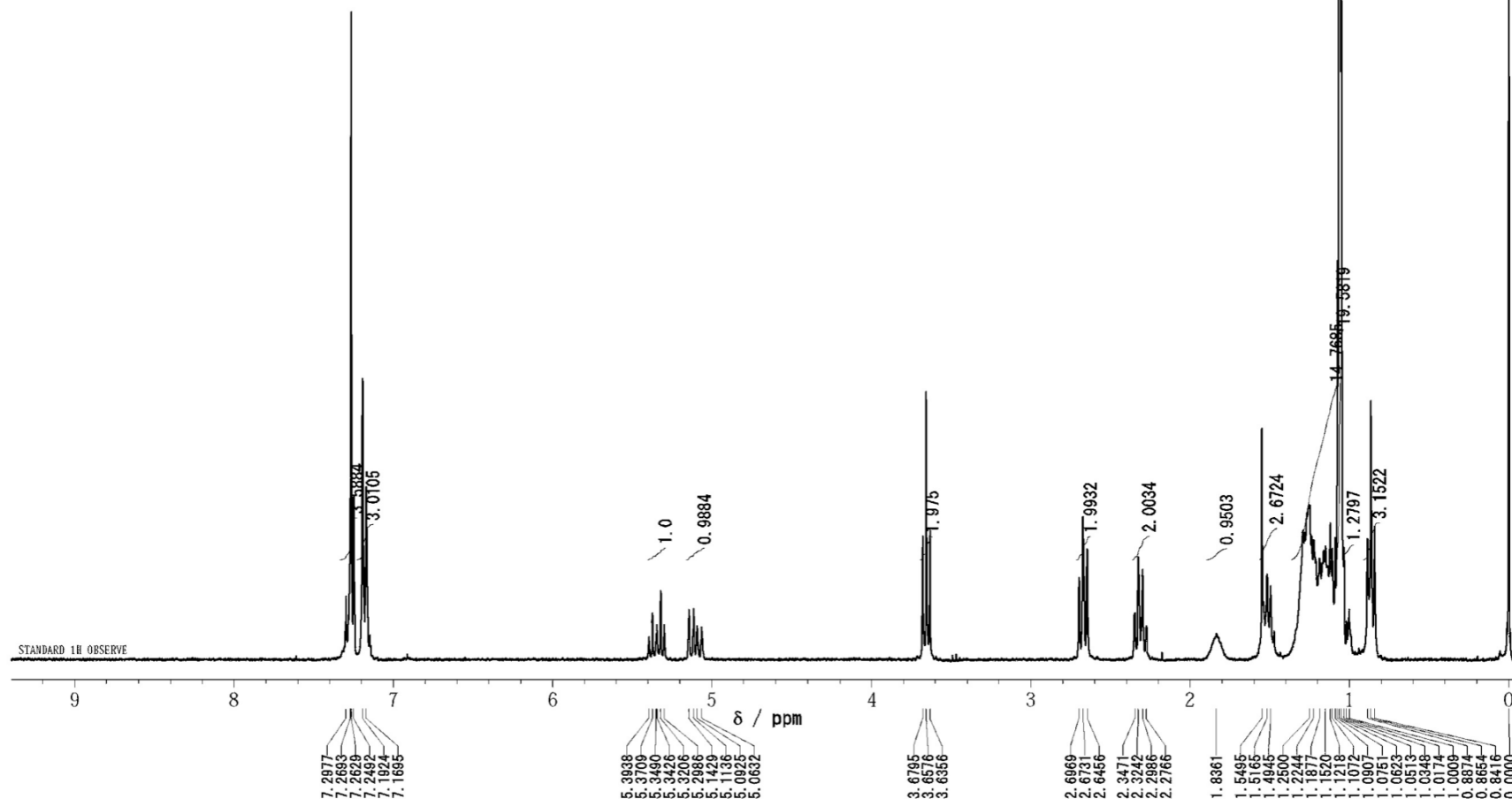
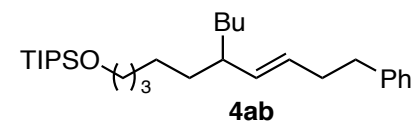


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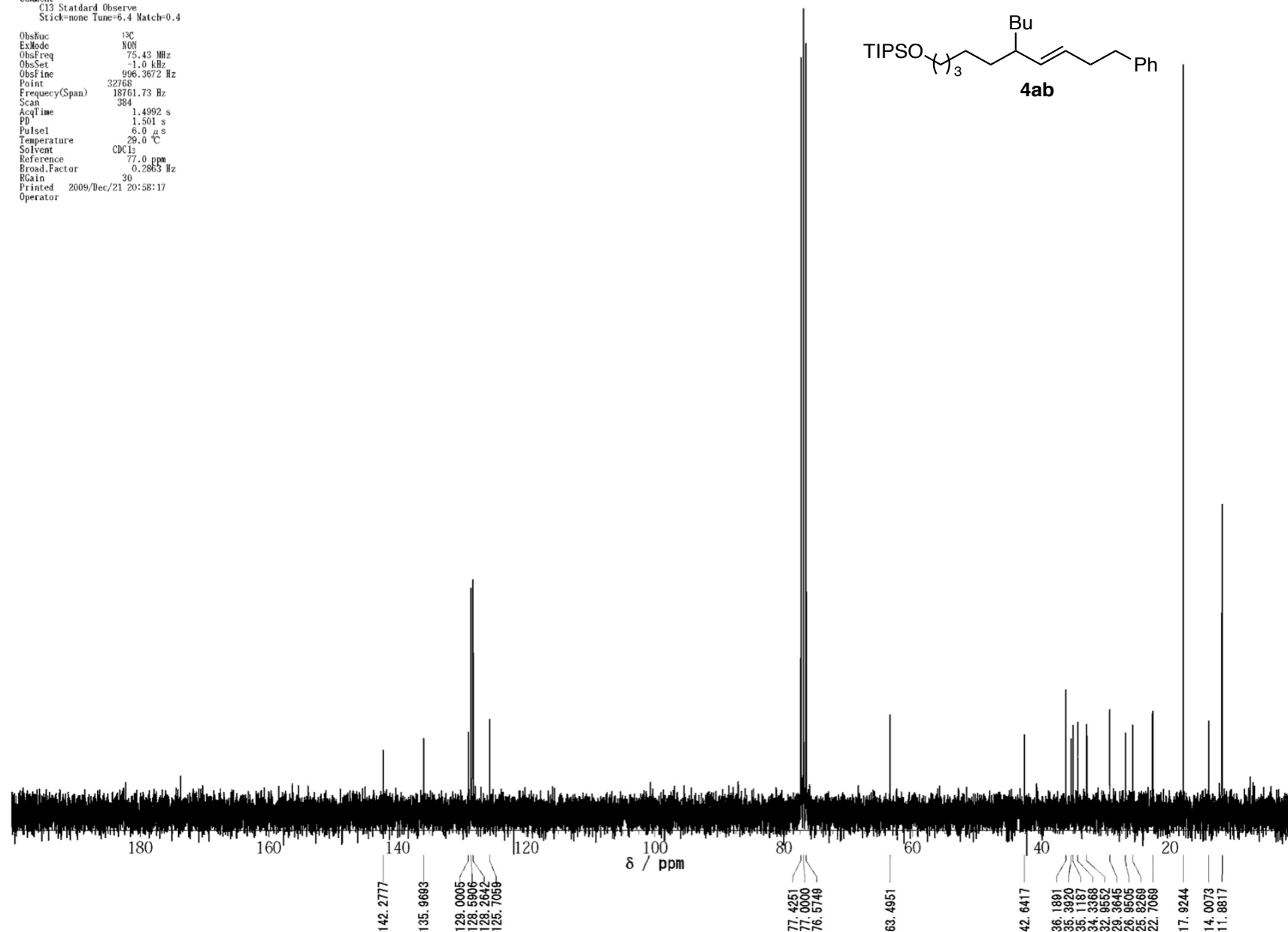
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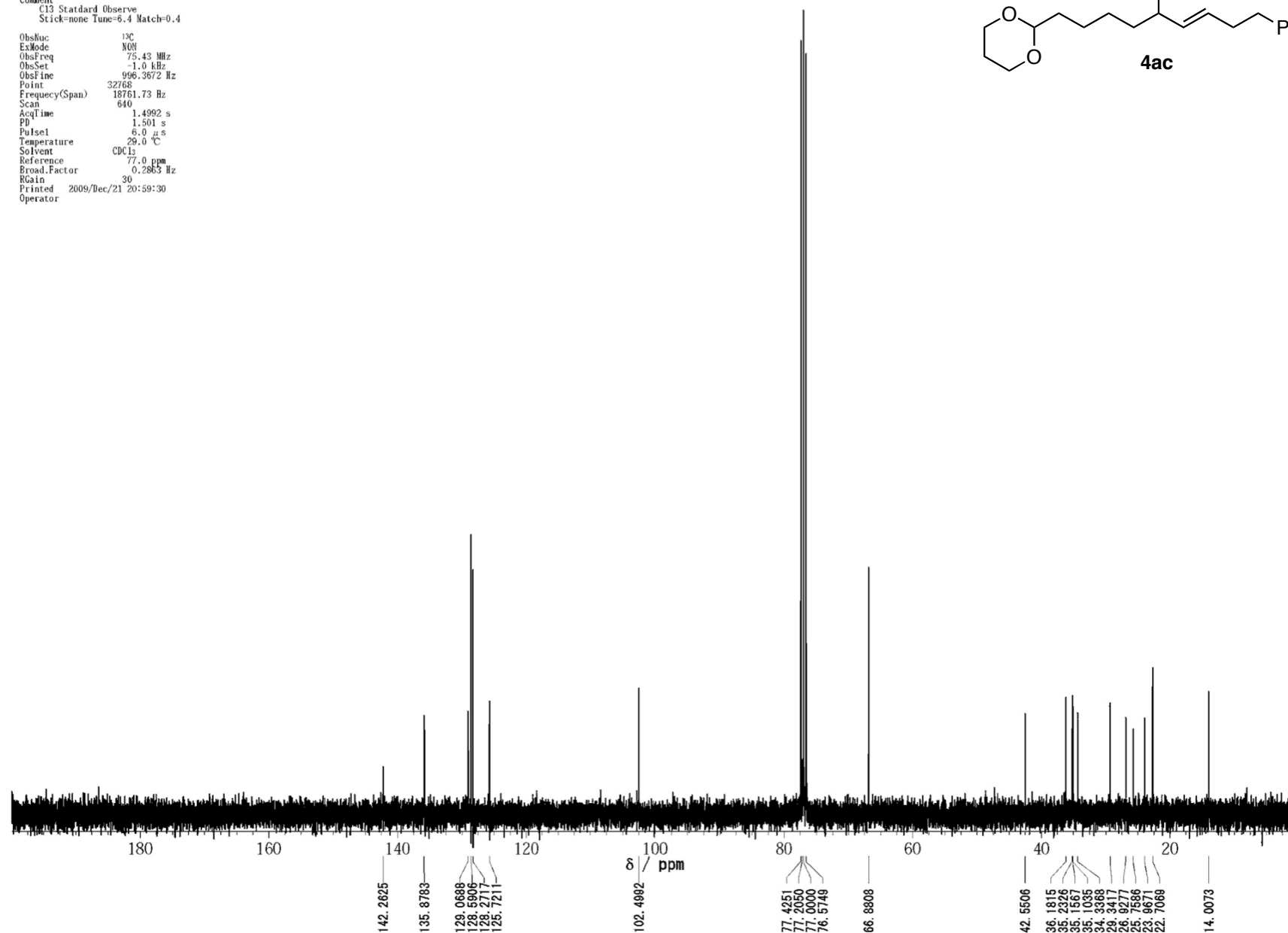
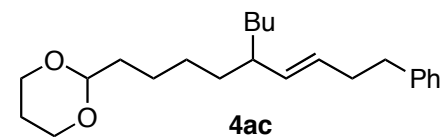
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PD 1.562 s
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Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
BroadFactor 0.1373 Hz
RGain 24
Printed 2009/Dec/21 10:01:09
Operator



Original File:
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 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 384
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
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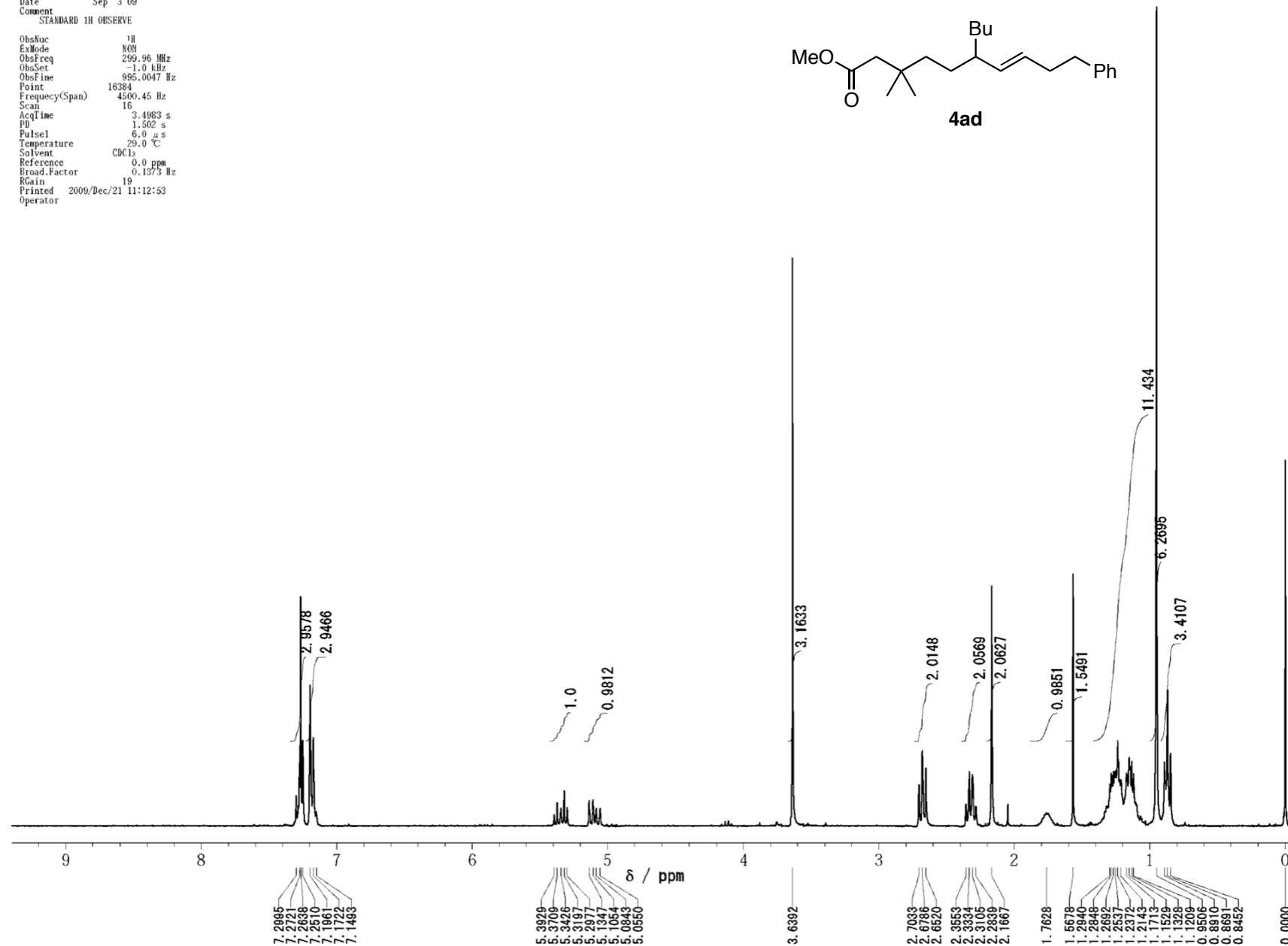
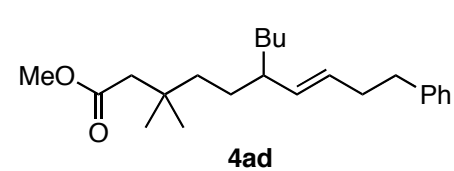


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 PD 1.501 s
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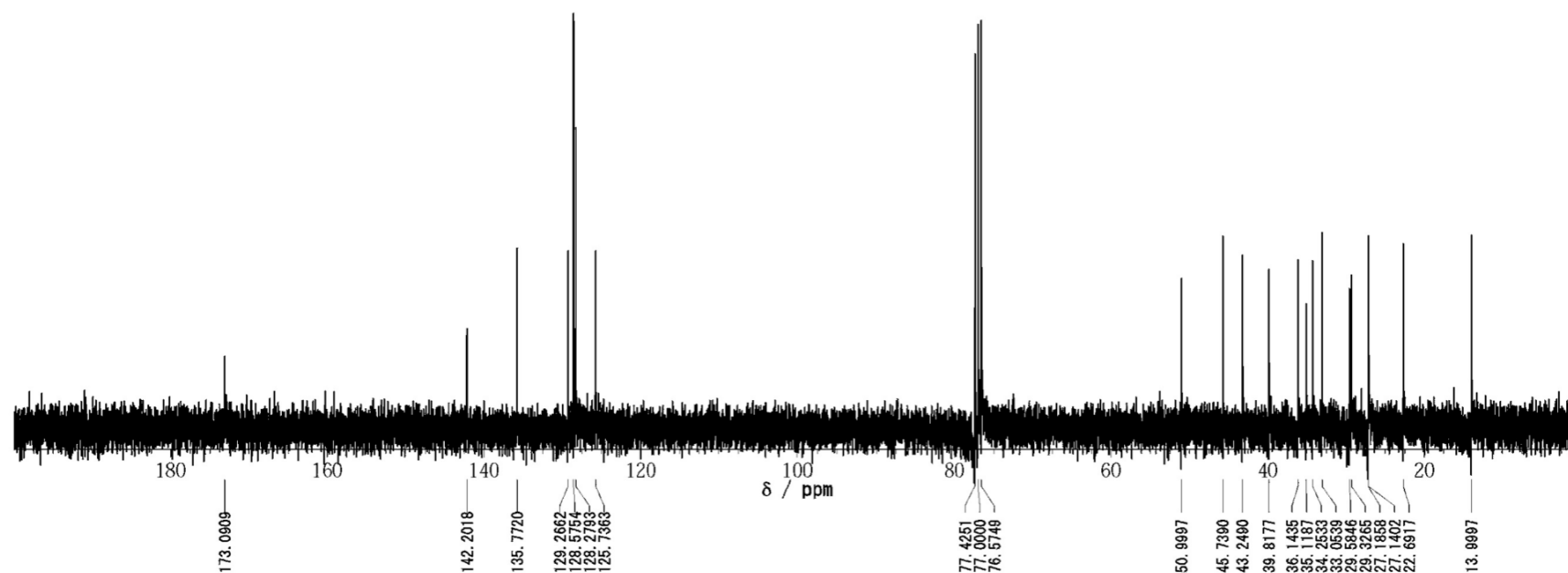
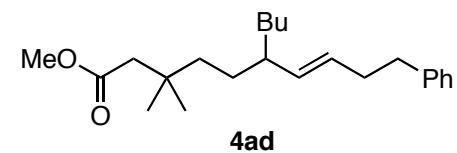


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 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Freqd.Factor 0.1373 Hz
 RGain 19
 Printed 2009/Dec/21 11:12:53
 Operator



Original File:
 Date Sep 4 09
 Comment
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 ExMode NON
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 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
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 Broad.Factor 0.2863 Hz
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 Operator



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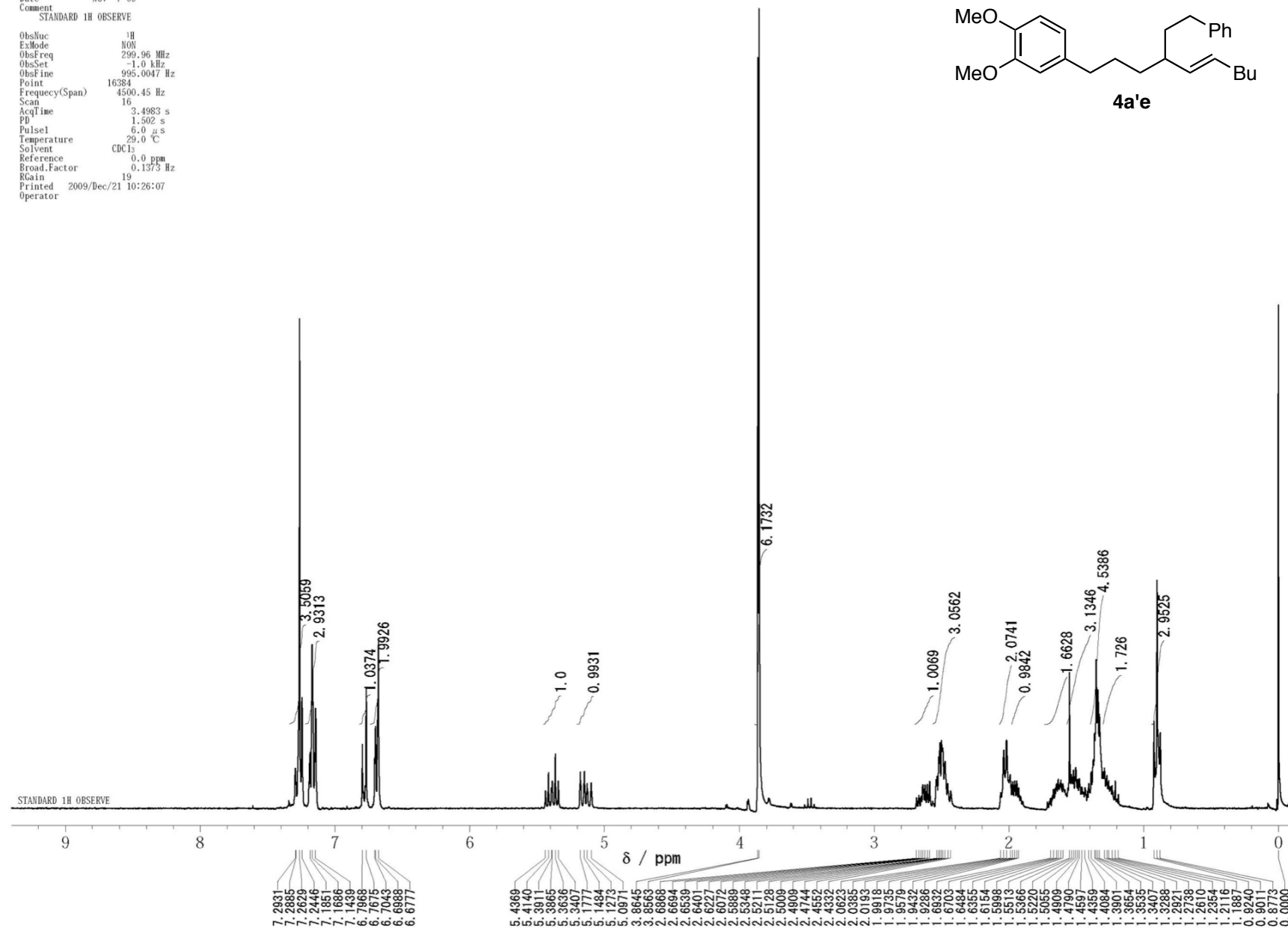
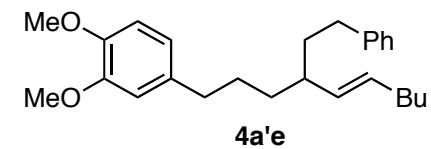
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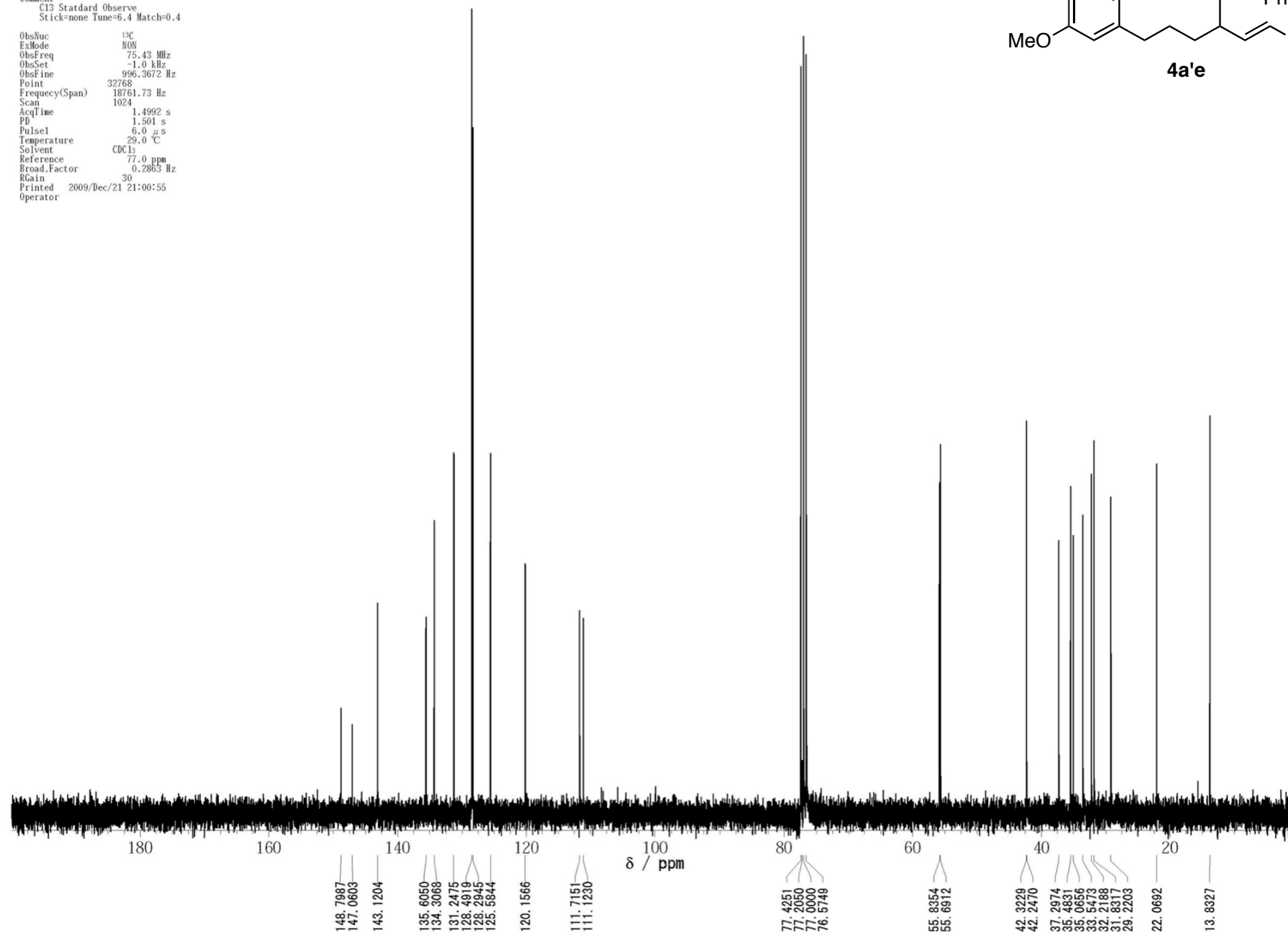
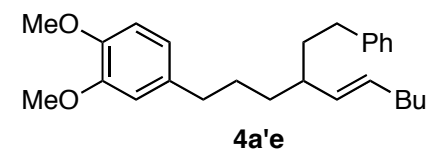
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STANDARD 1H OBSERVE

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Scan 16
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PD 1.502 s
Pulse1 6.0 μs
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Operator



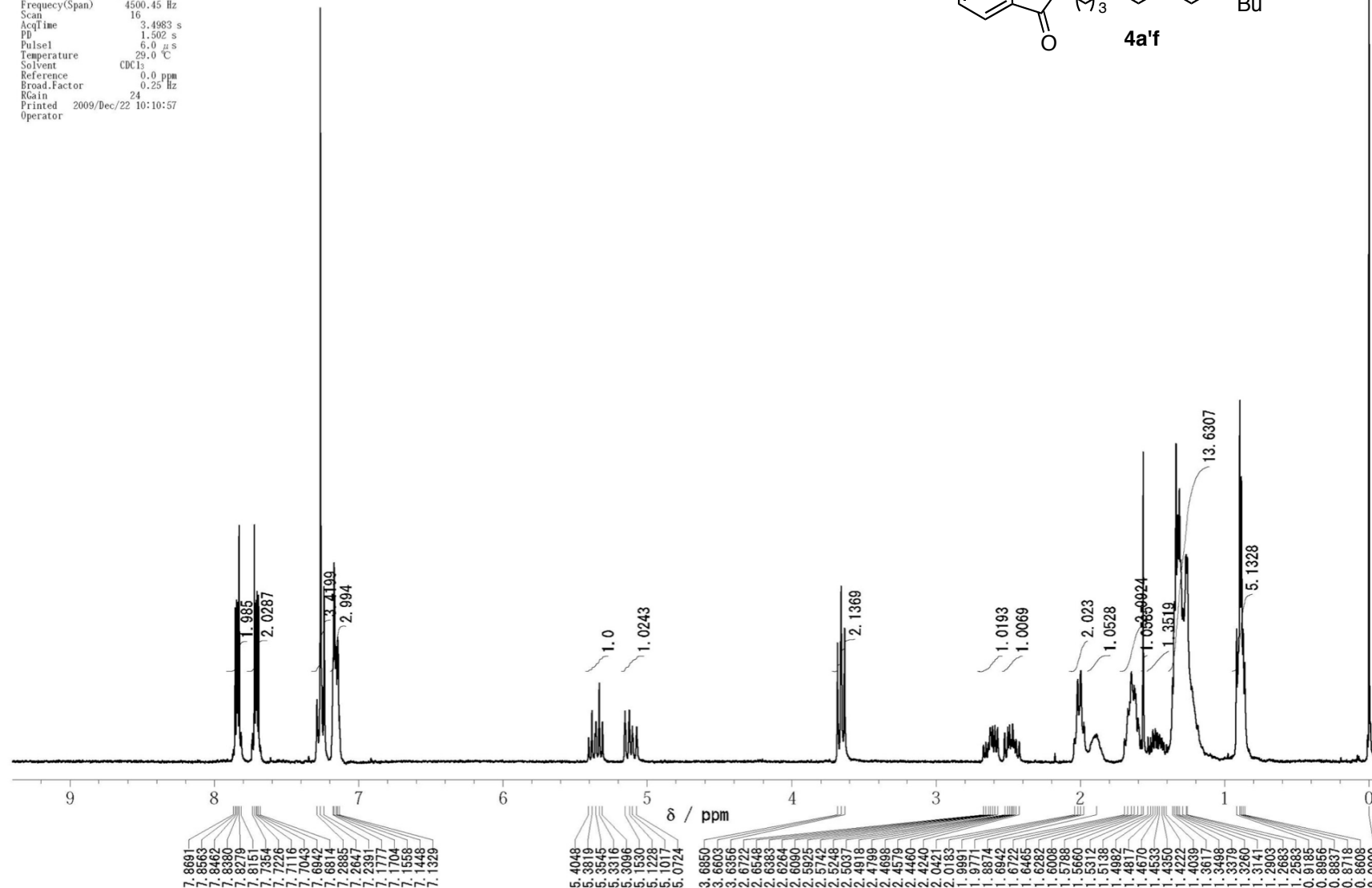
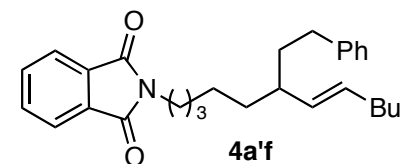
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 ObsFine 996.3672 Hz
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 Frequency(Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
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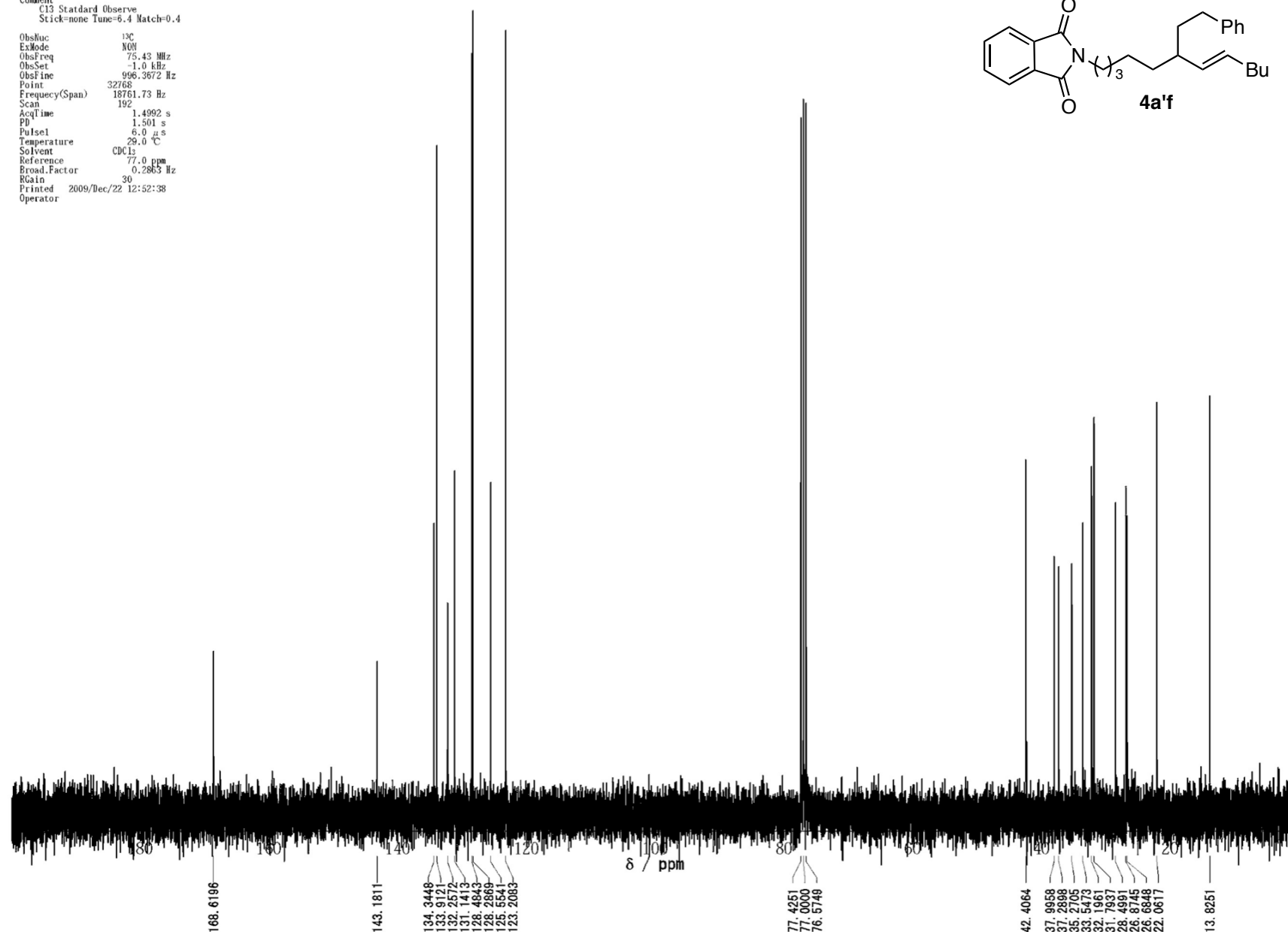
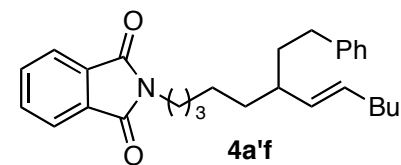
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Date Dec 21 09
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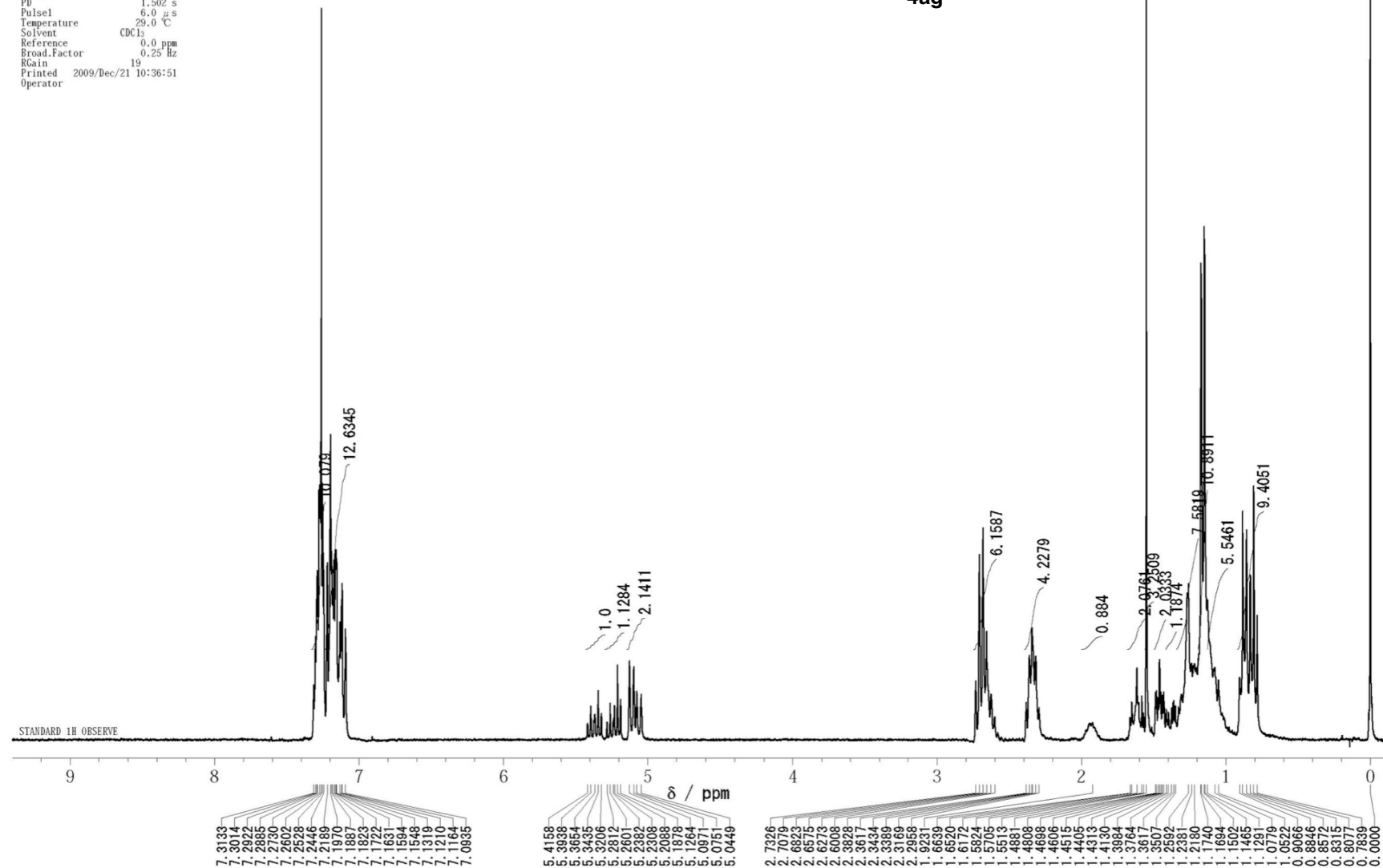
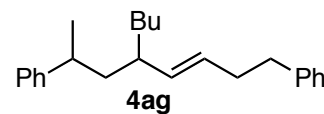
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PD 1.502 s
Pulse 6.0 μs
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Operator



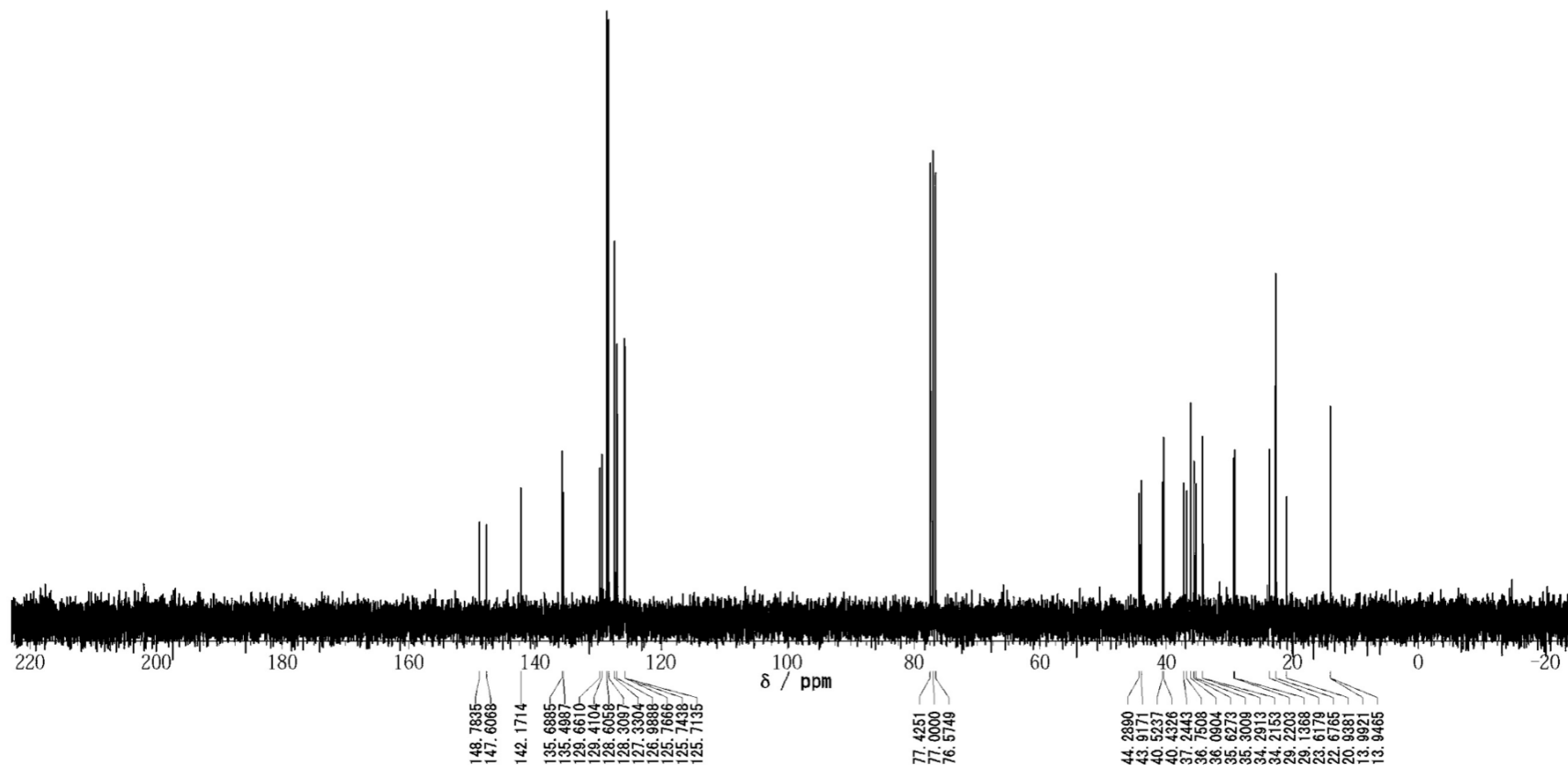
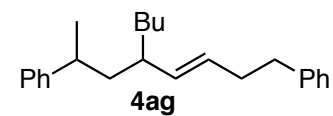
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 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/22 12:52:38
 Operator



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 PD 1.502 s
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Original File:
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 Printed 2009/Dec/21 21:02:09
 Operator



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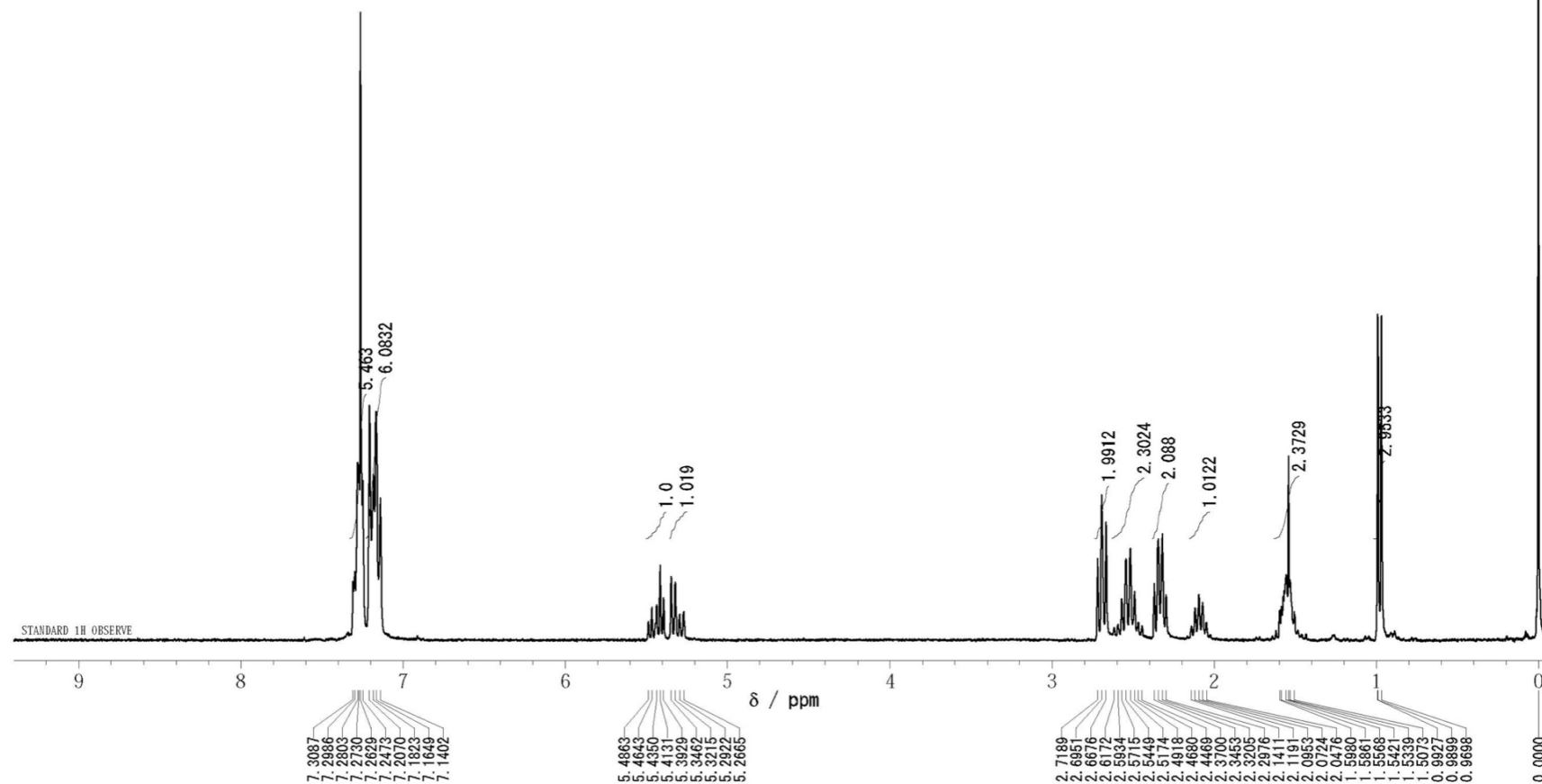
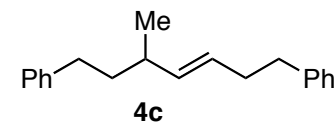
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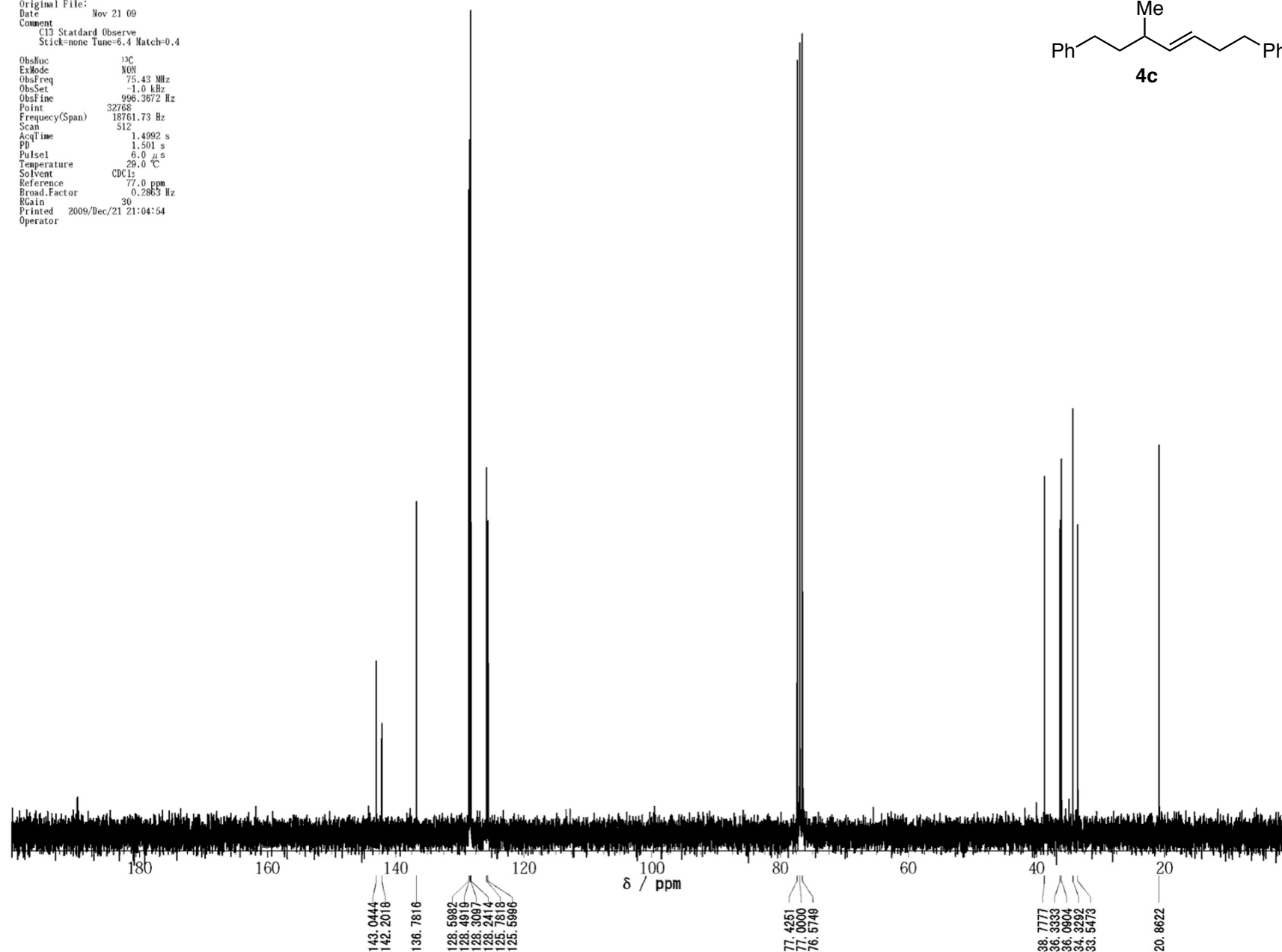
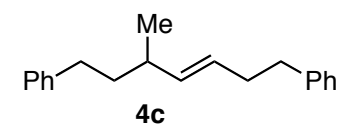
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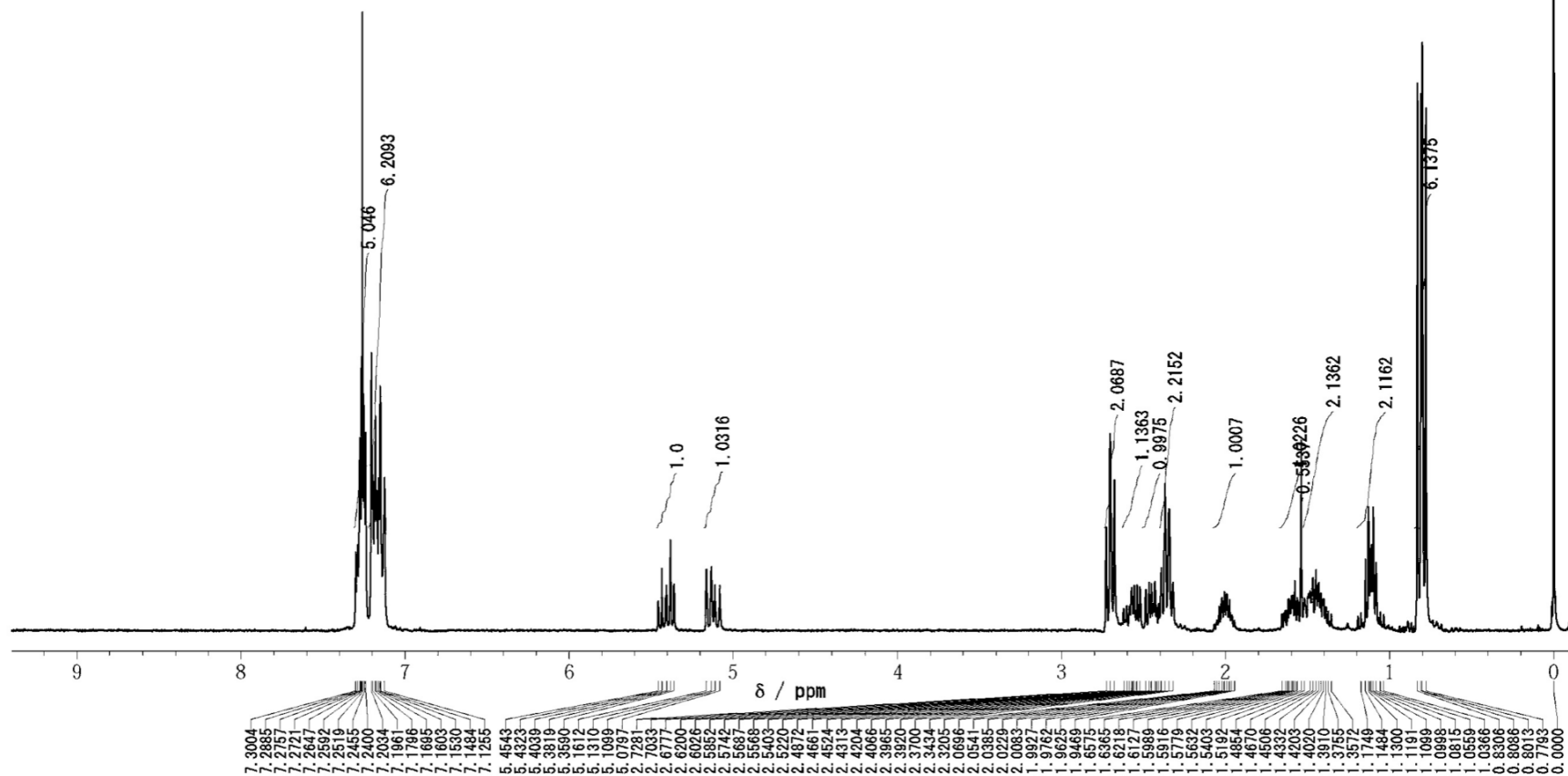
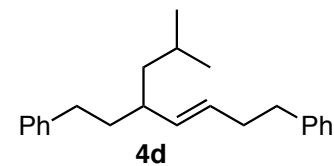
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PD 1.502 s
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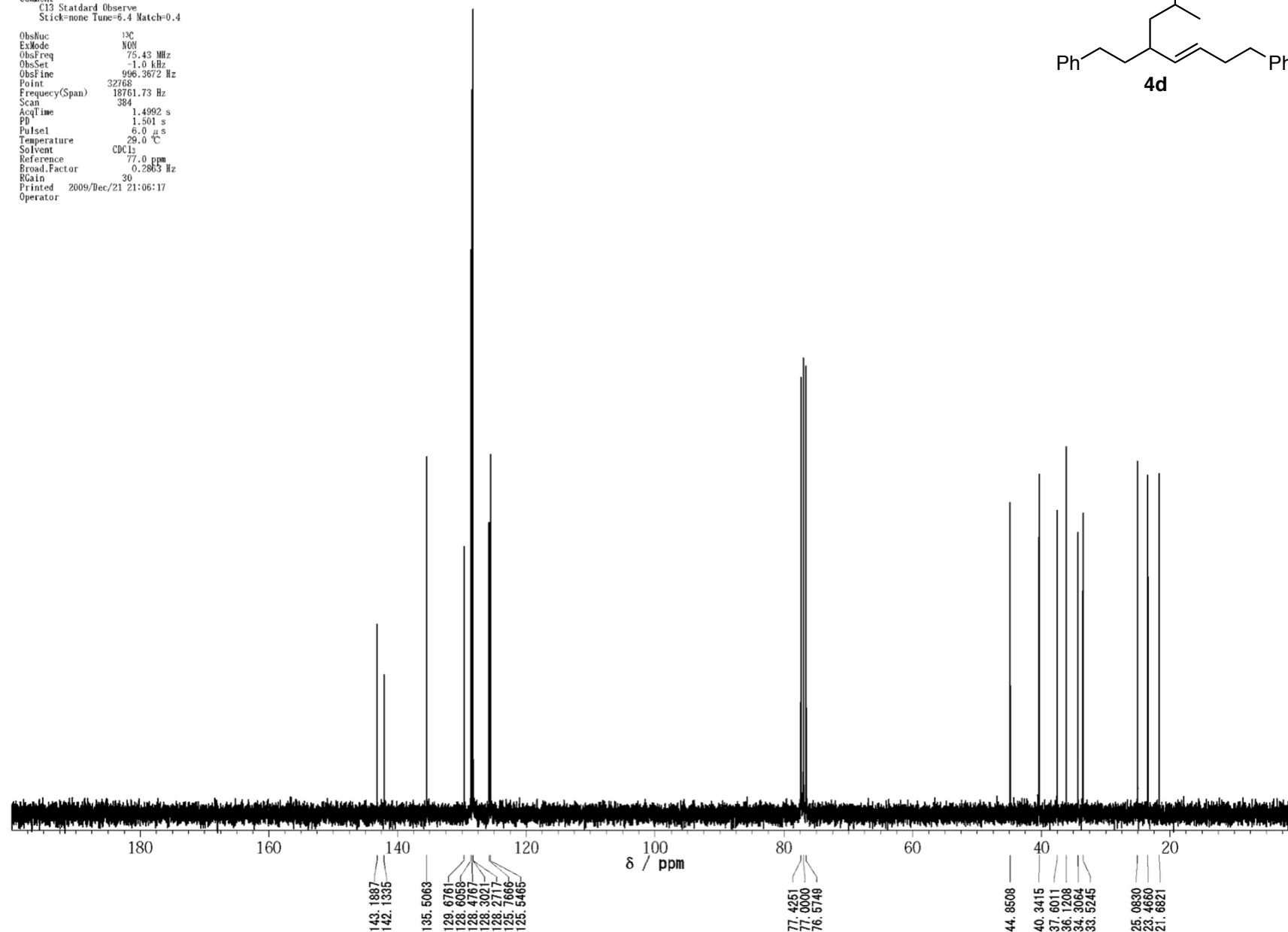
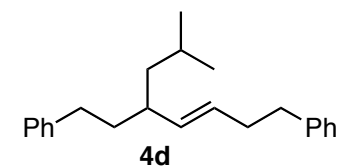
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 Point 32768
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 RGain 30
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 Operator



Original File:
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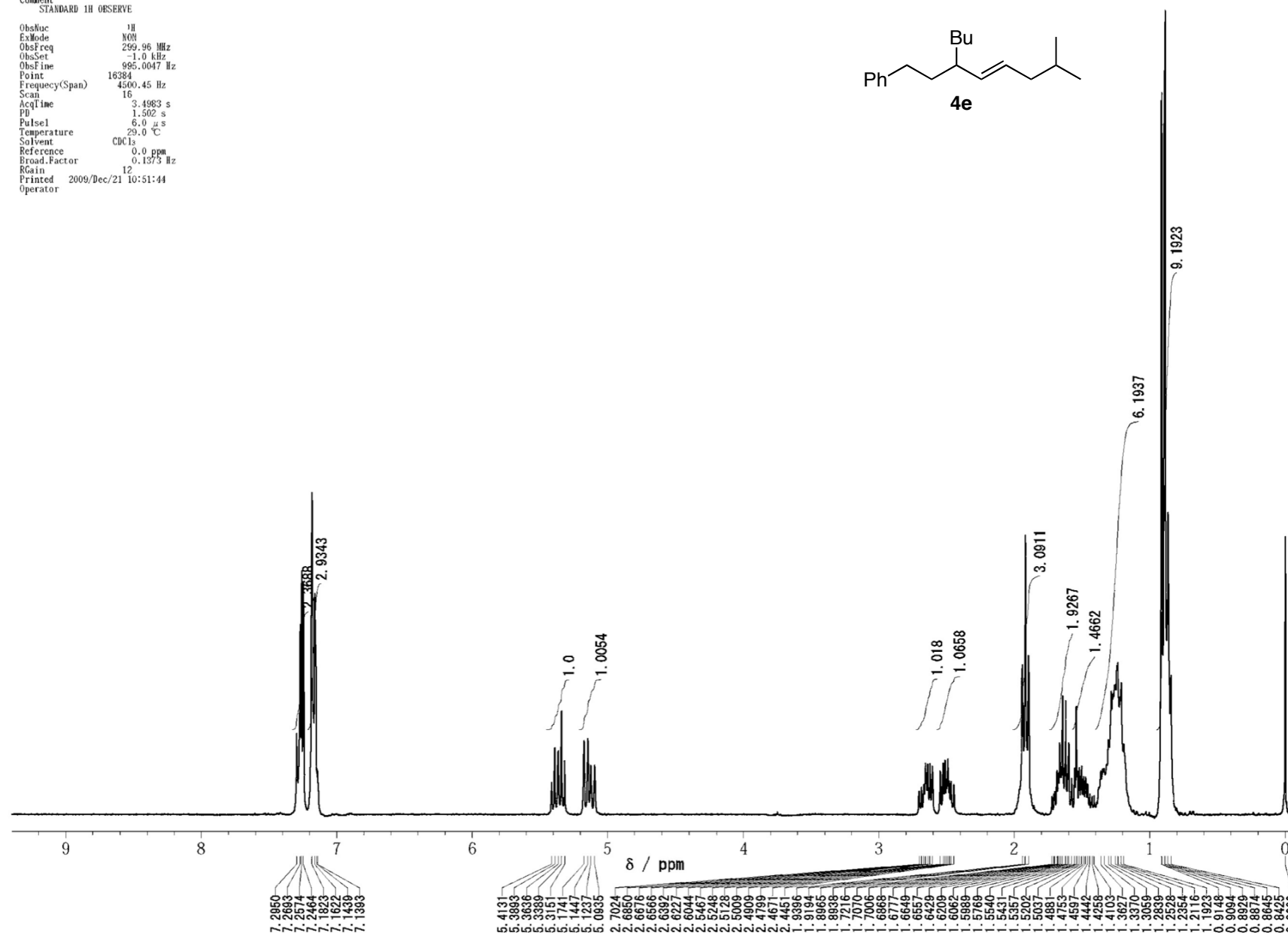
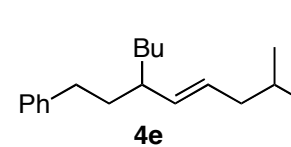


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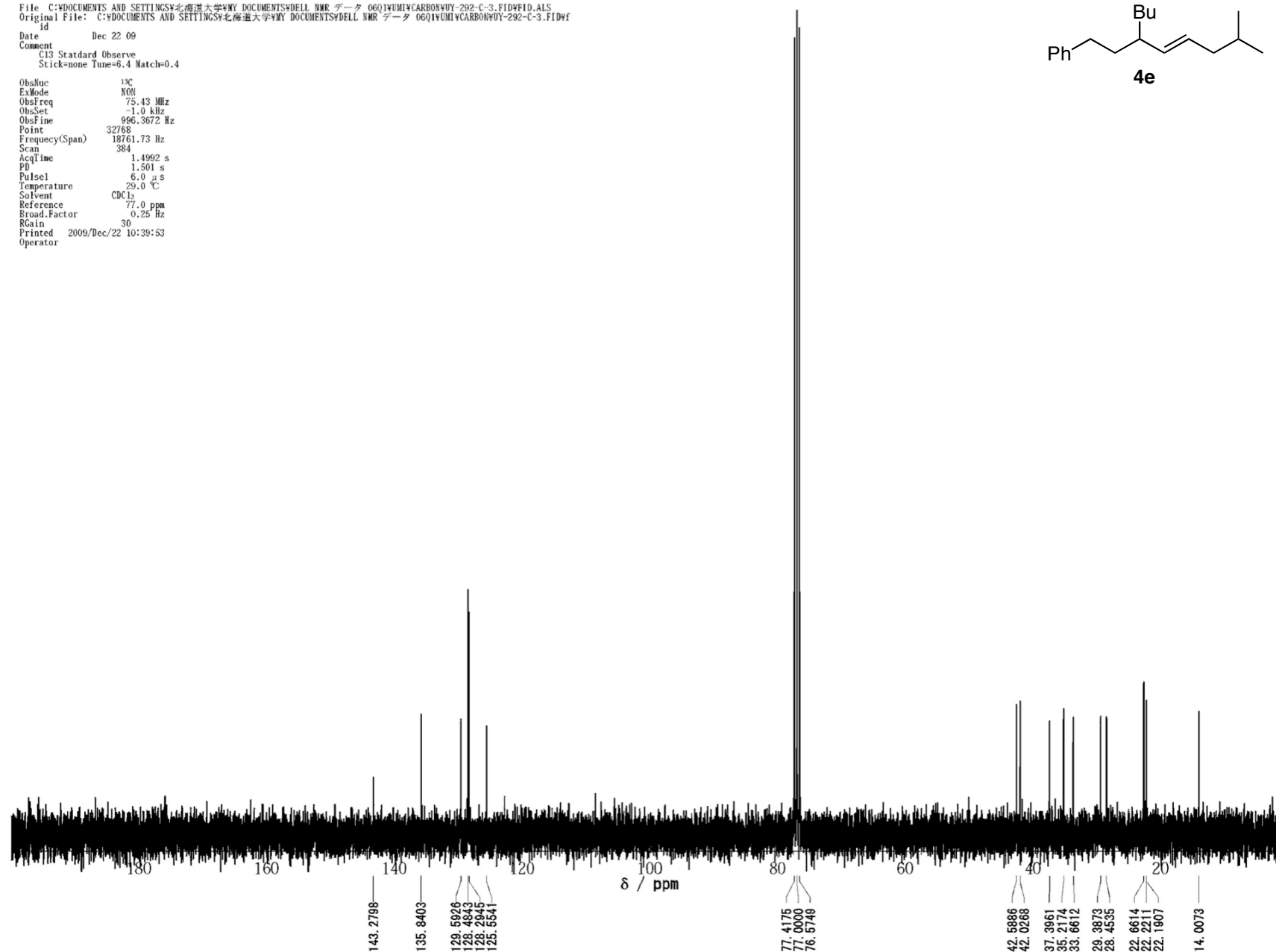
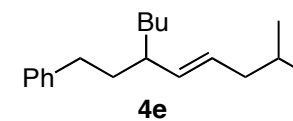


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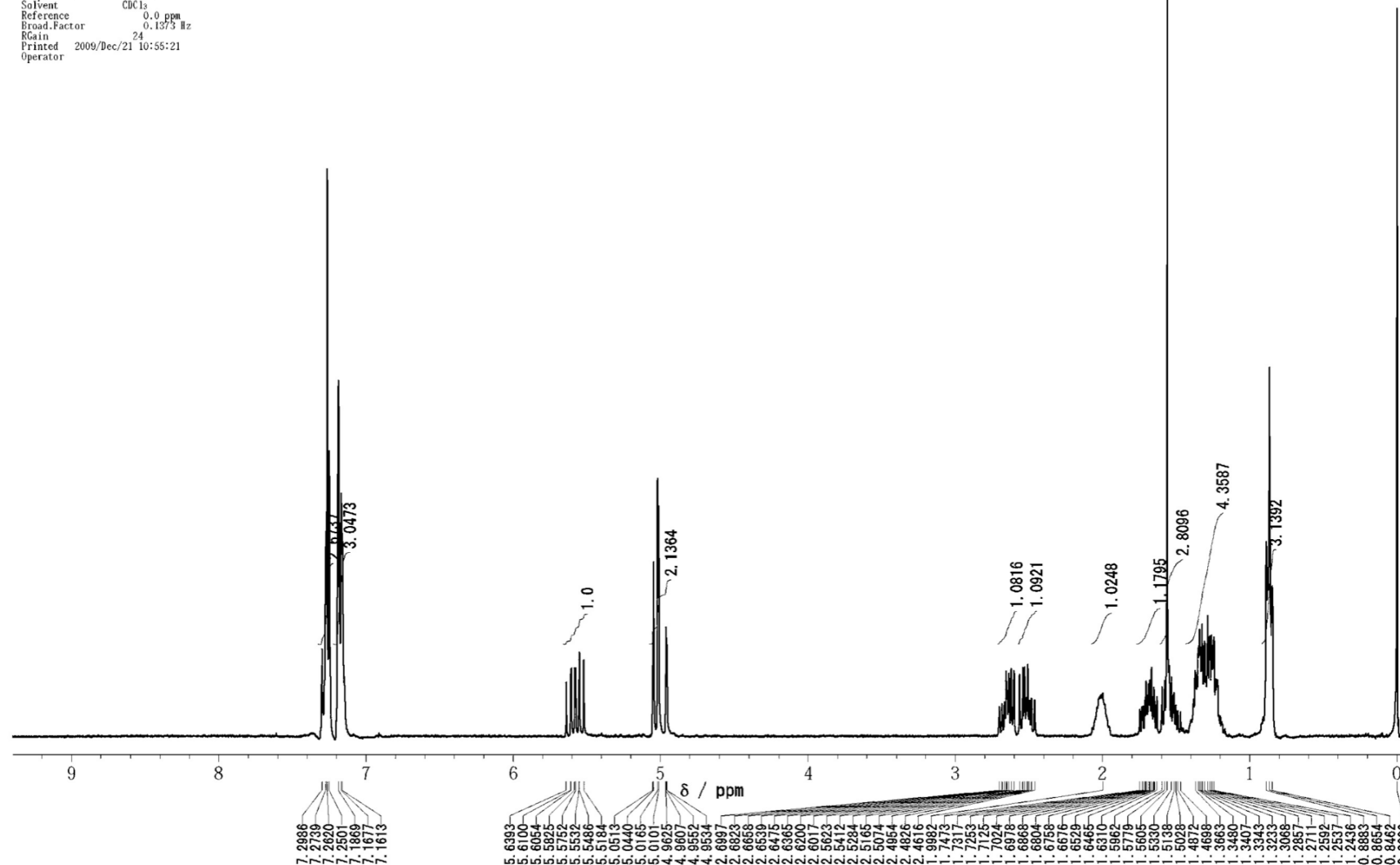
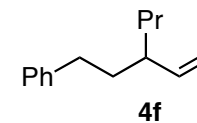
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 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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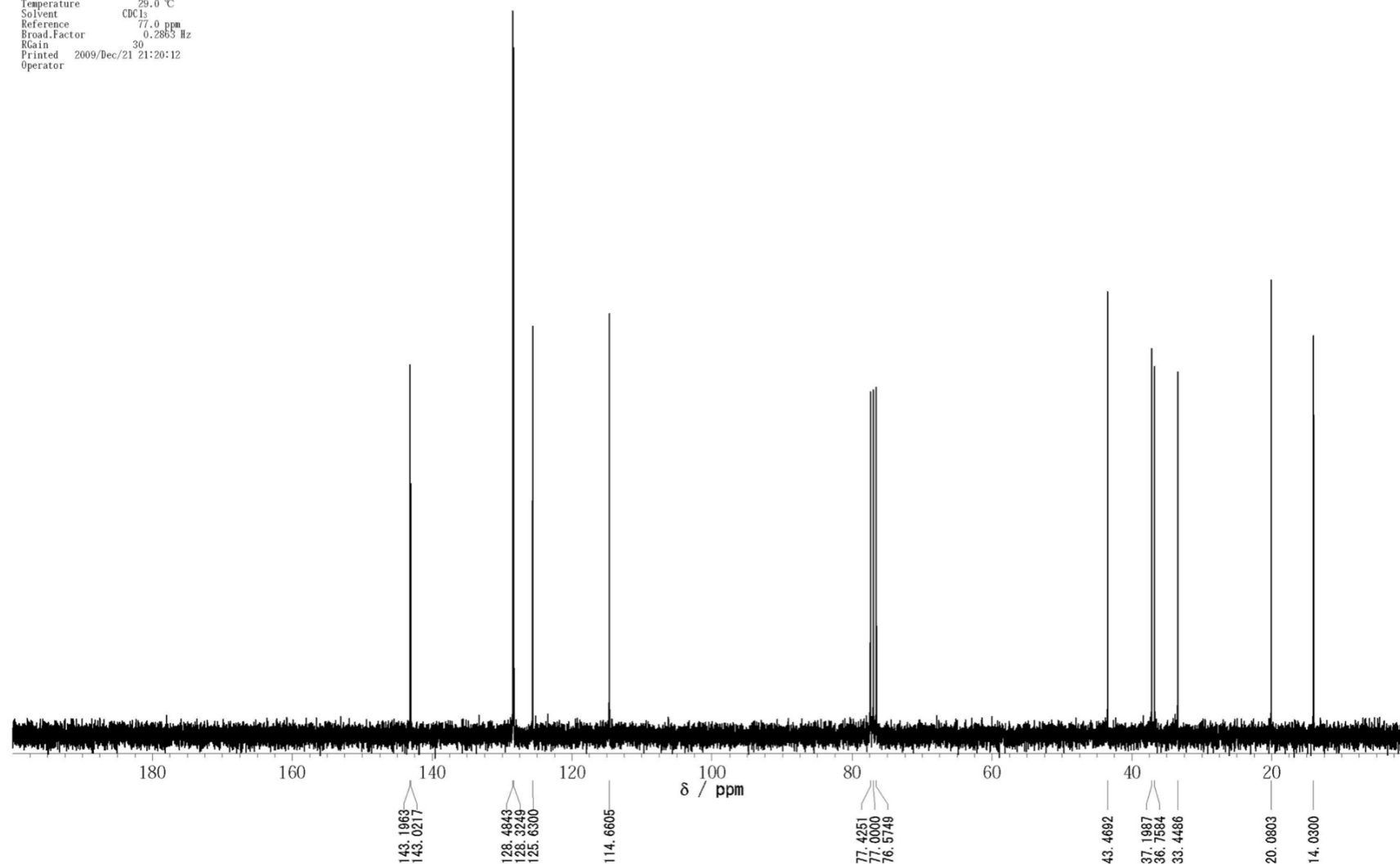
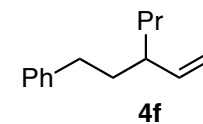
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 Point 32768
 Frequency(Span) 18761.73 Hz
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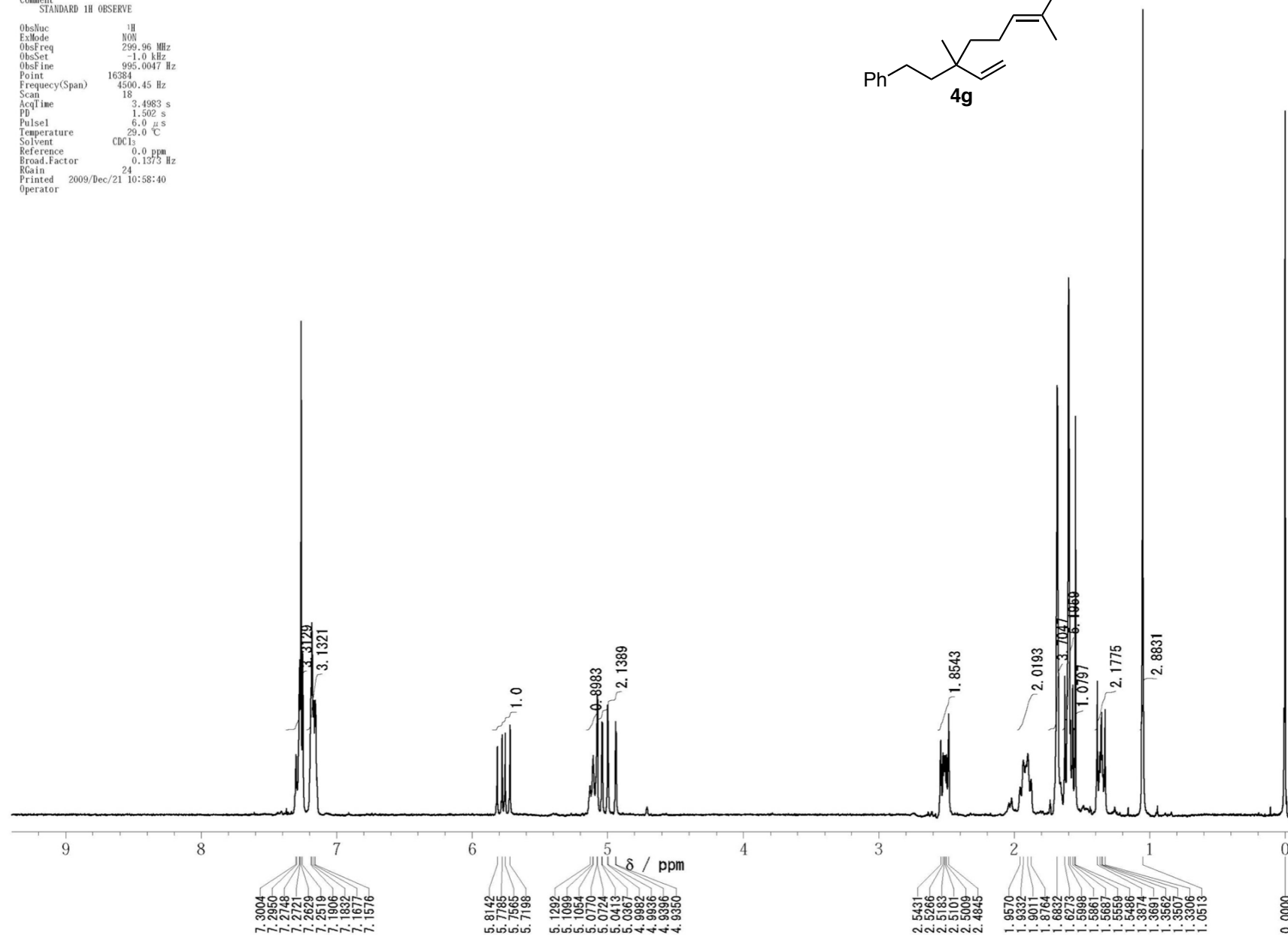
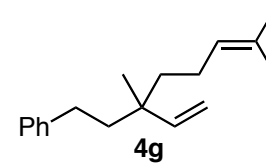
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 Pulse1 6.0 μ s
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 Operator



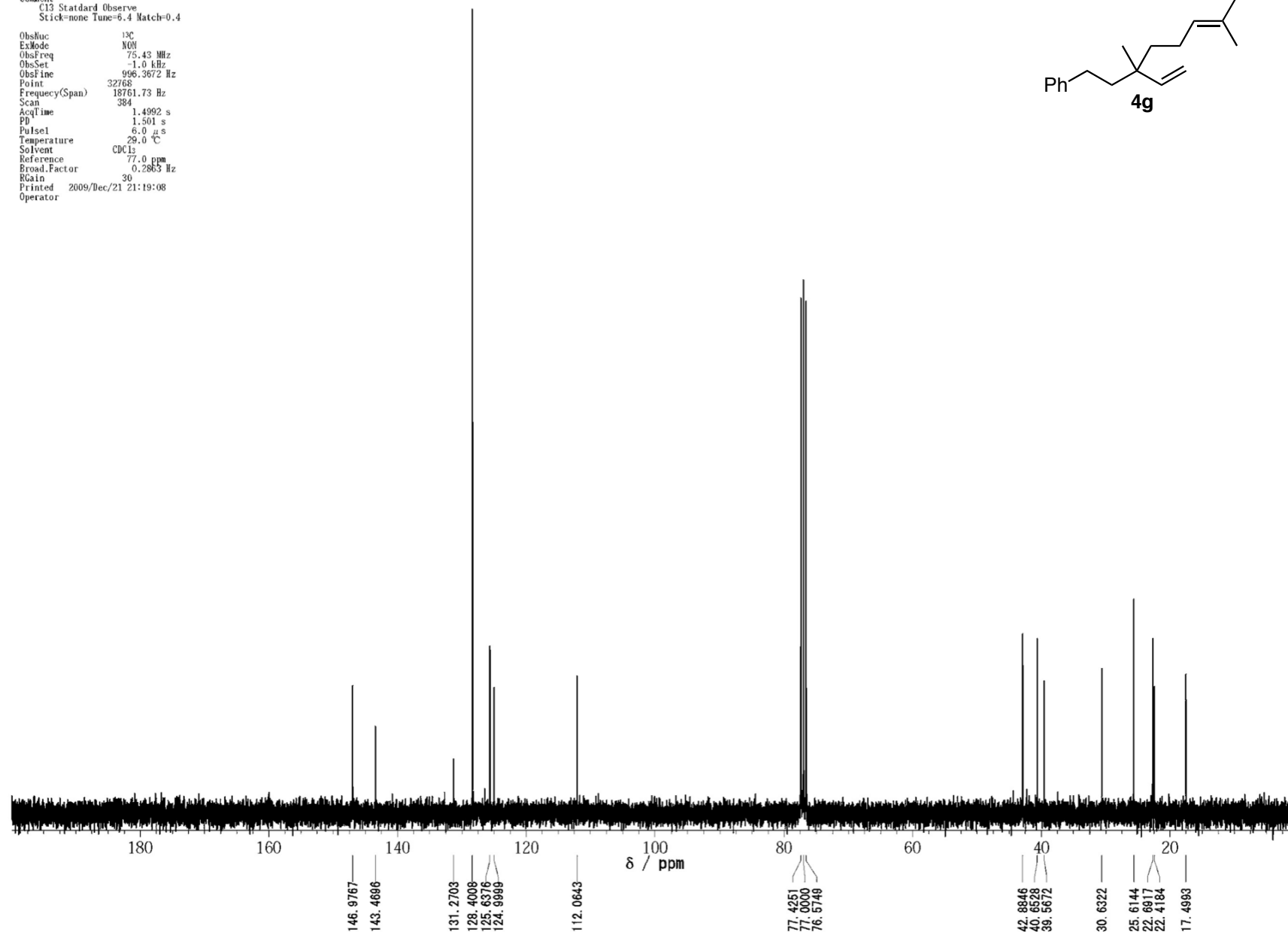
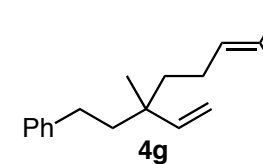
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 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 192
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 21:20:12
 Operator



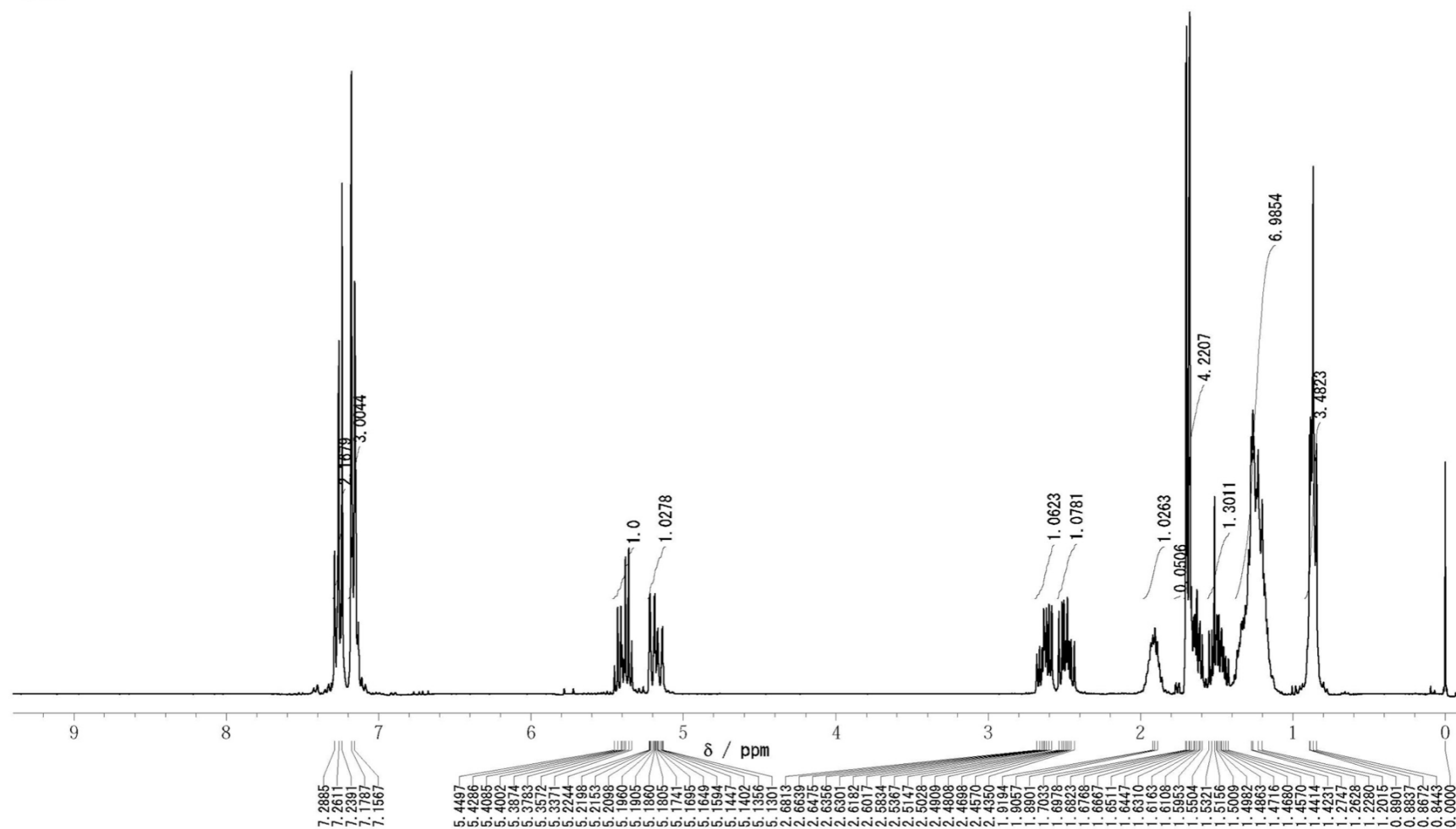
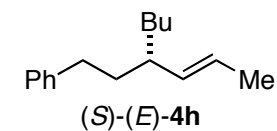
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 ExMode NON
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 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 18
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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 Printed 2009/Dec/21 10:58:40
 Operator



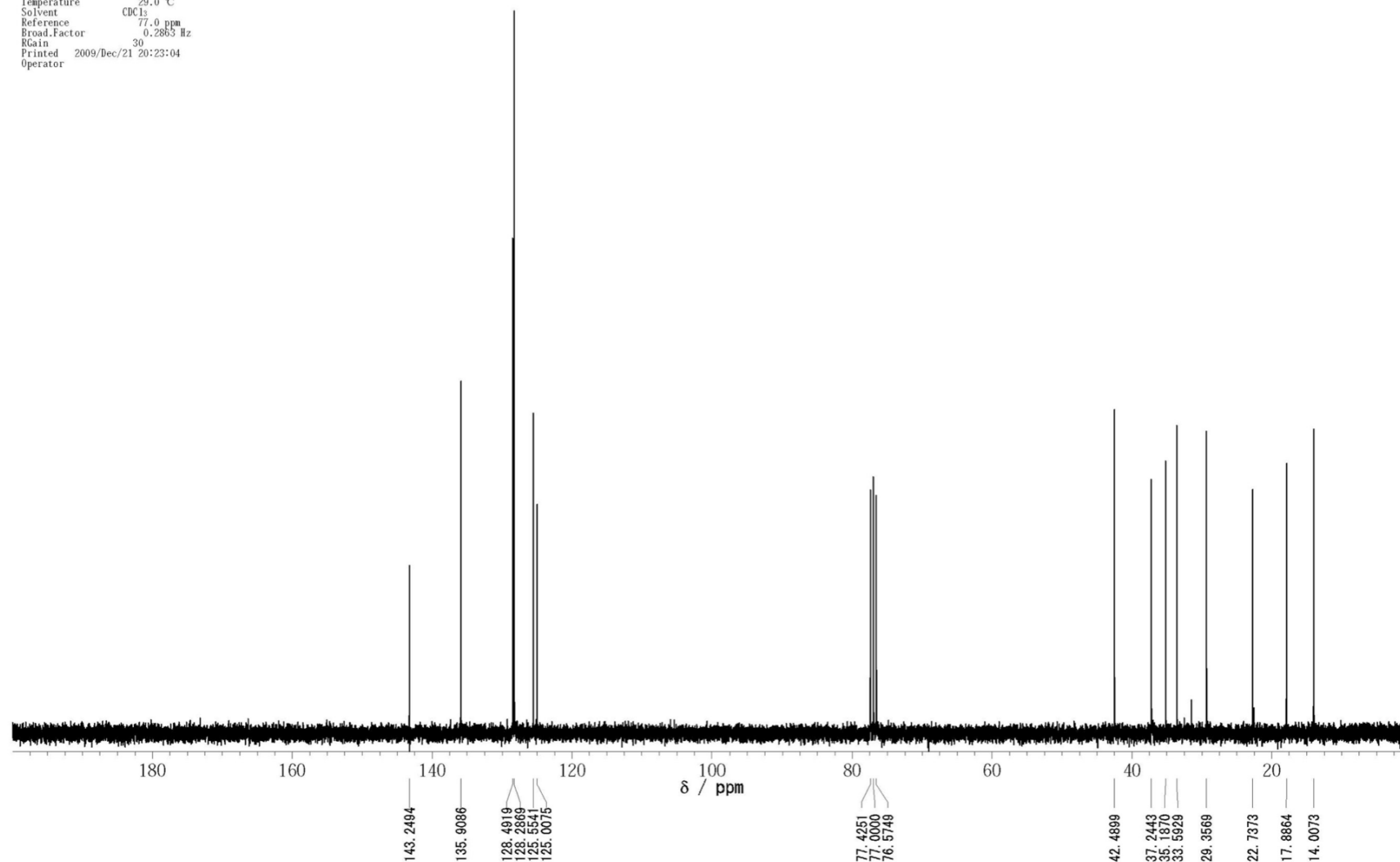
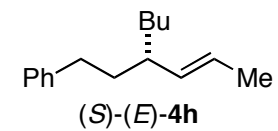
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 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 384
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2009/Dec/21 21:19:08
 Operator



Original File:
 Date Sep 26 09
 Comment
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 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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 Operator

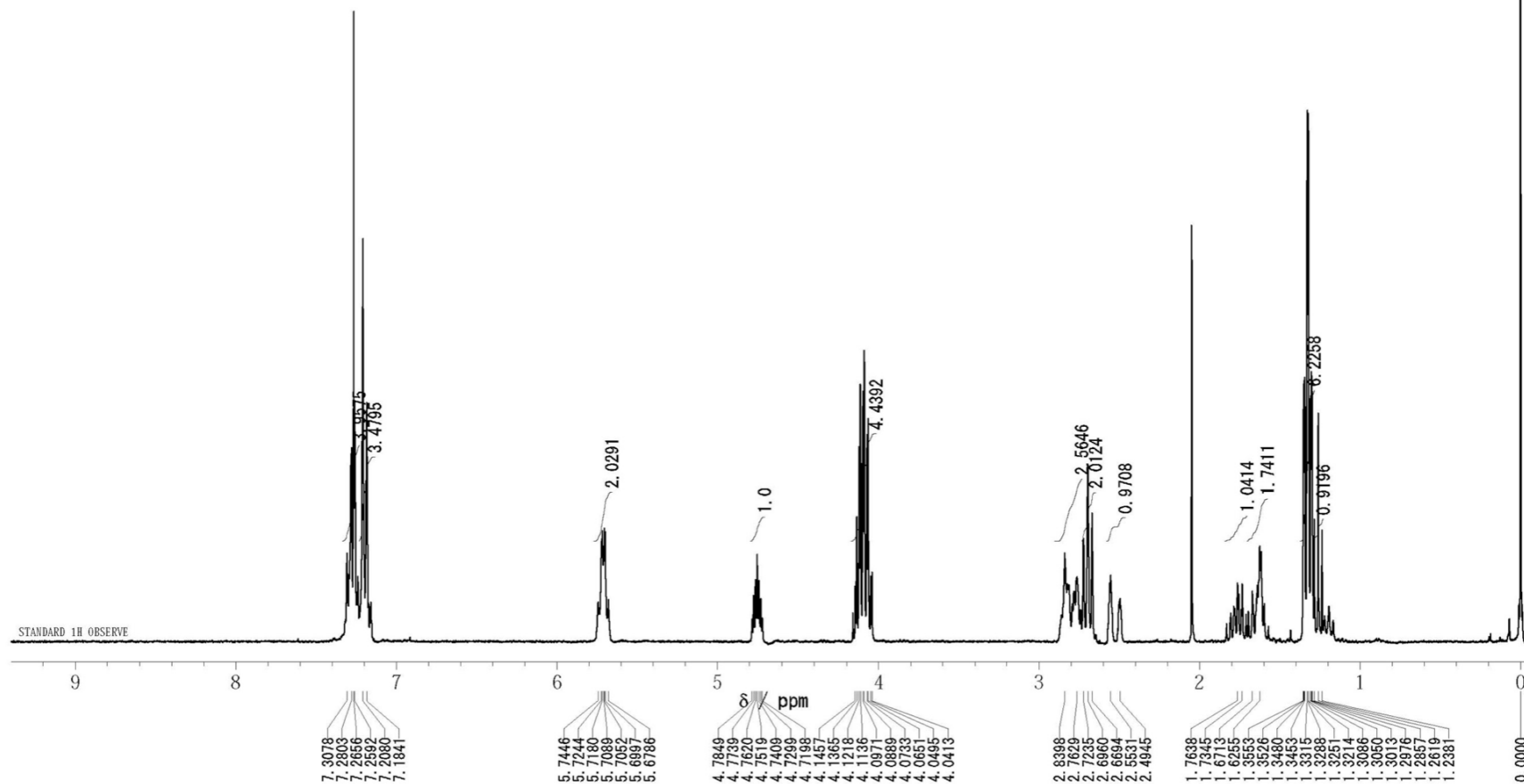
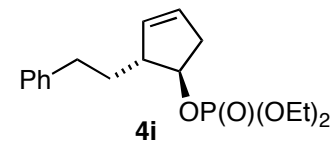


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 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
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 Operator

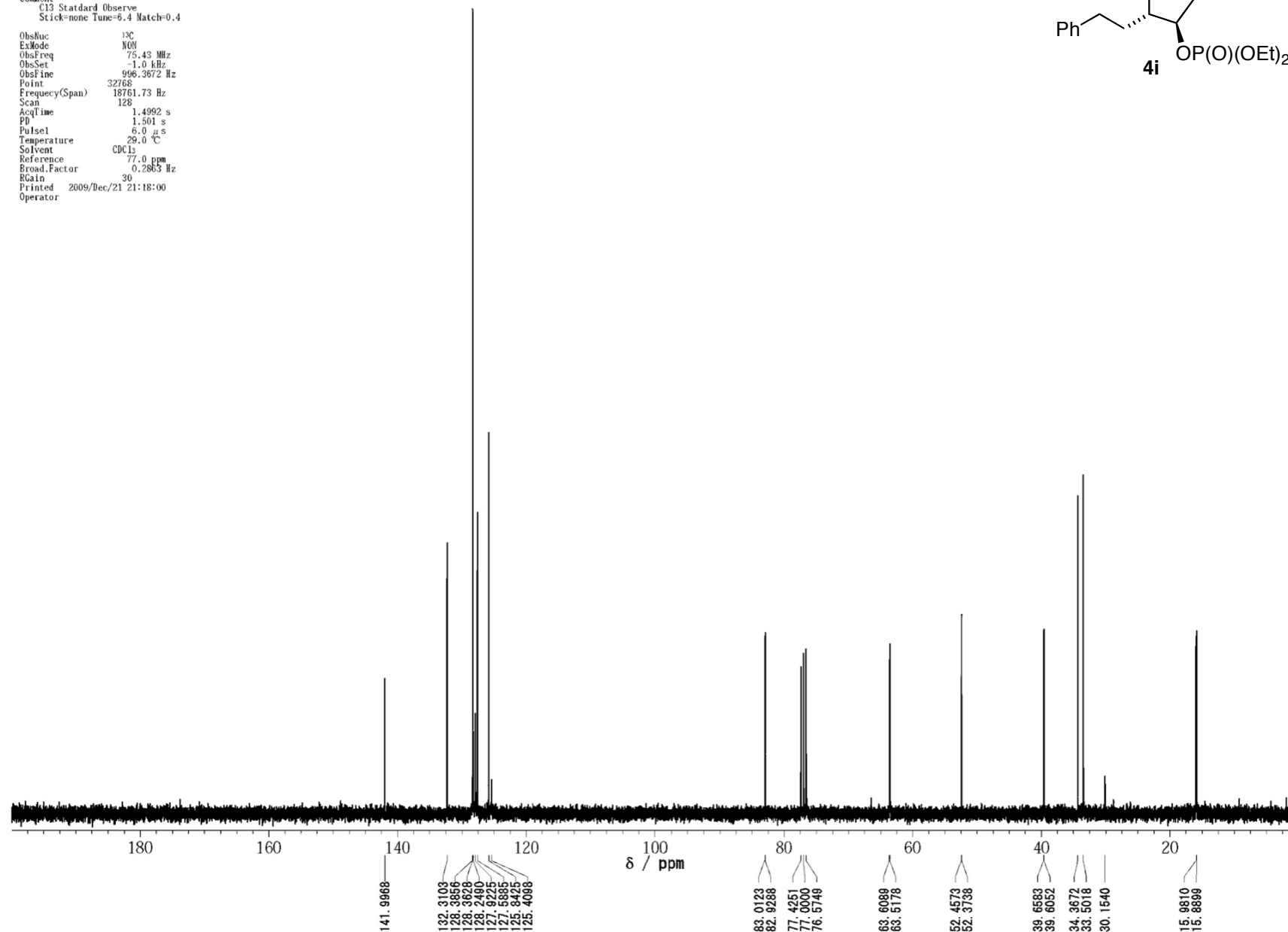
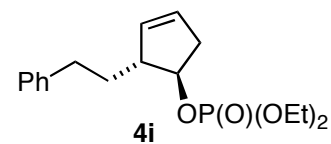


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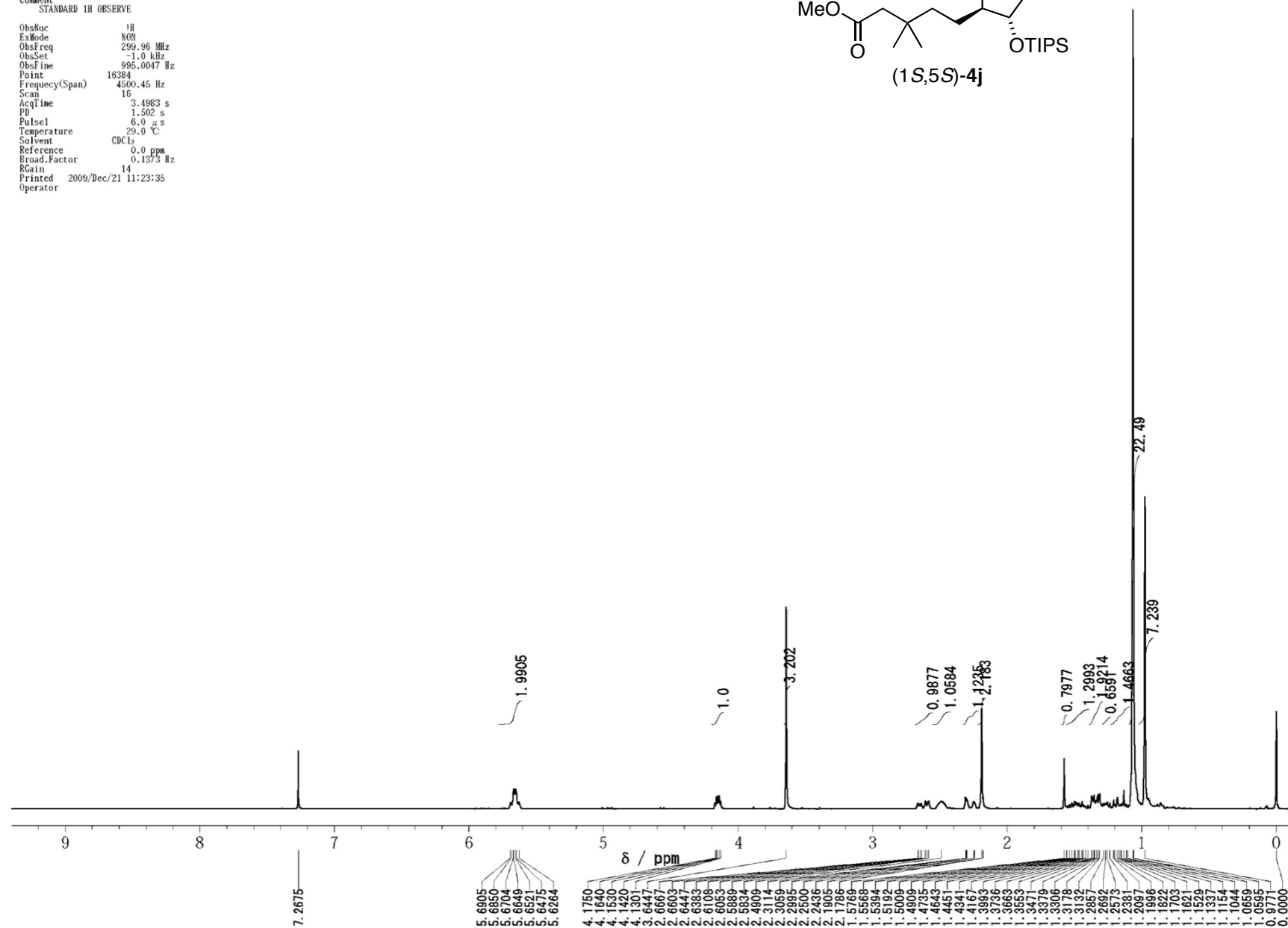
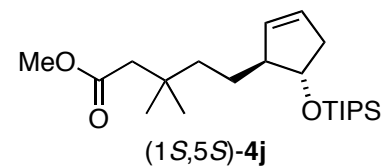
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 ObsFine 995.0047 Hz
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 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μs
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 Solvent CDCl₃
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 Broad.Factor 0.1373 Hz
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 Operator



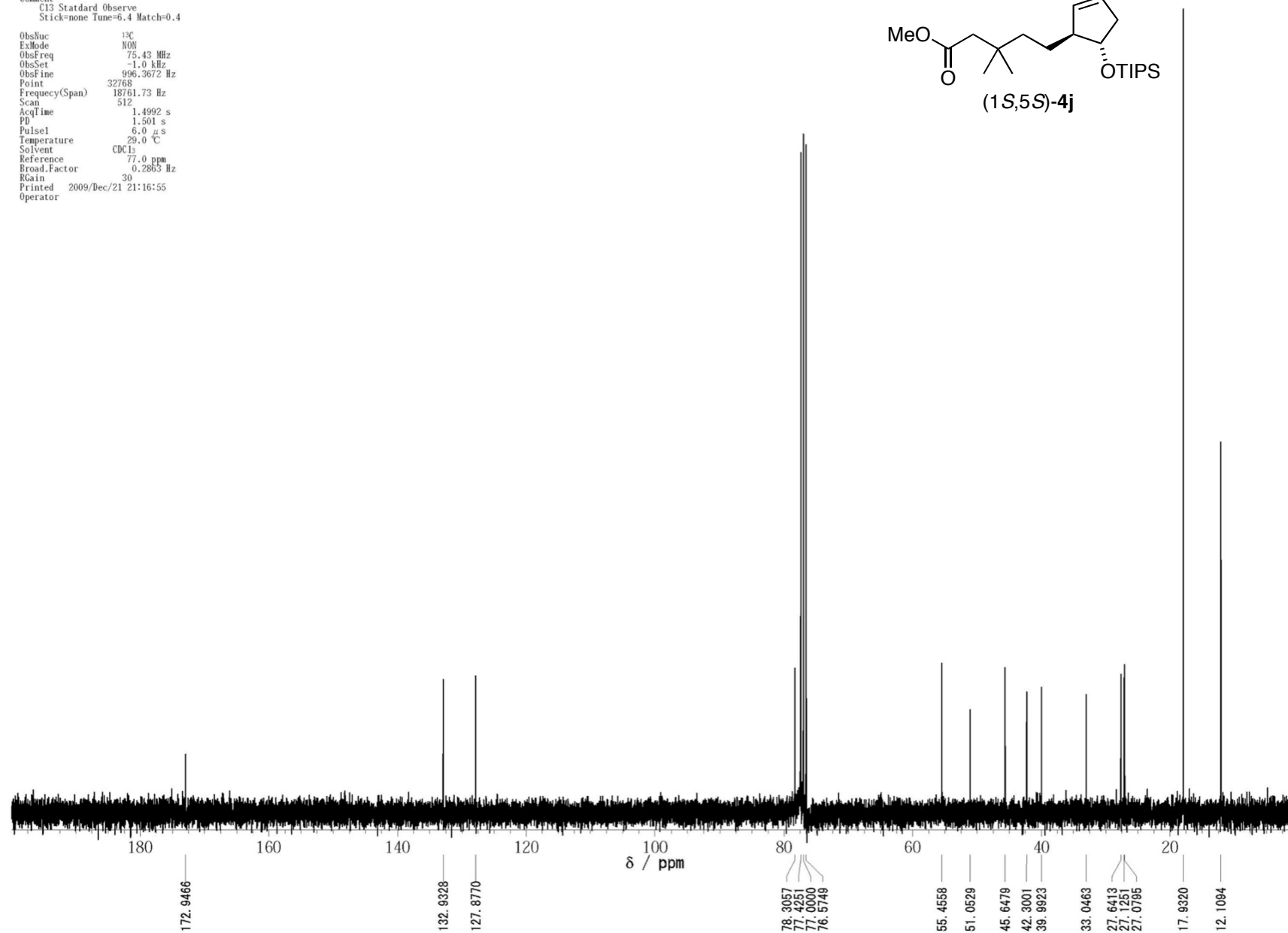
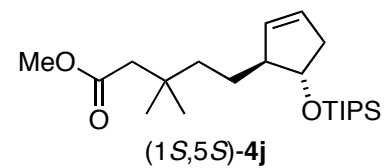
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 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
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 Solvent CDCl₃
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 Broad.Factor 0.2863 Hz
 RGain 30
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 Operator



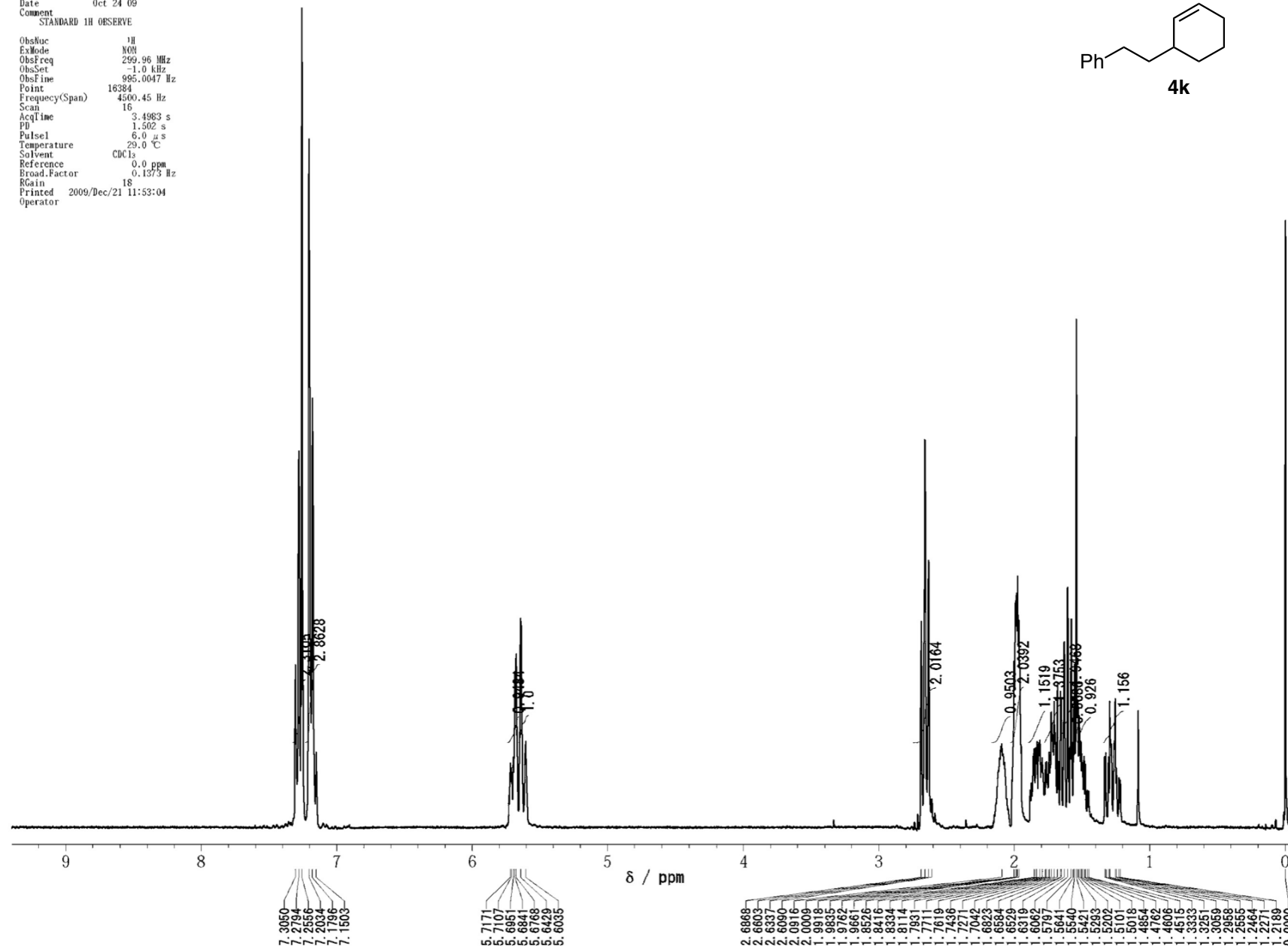
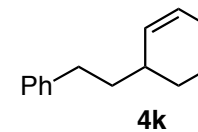
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 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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 Solvent CDCl₃
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 Operator



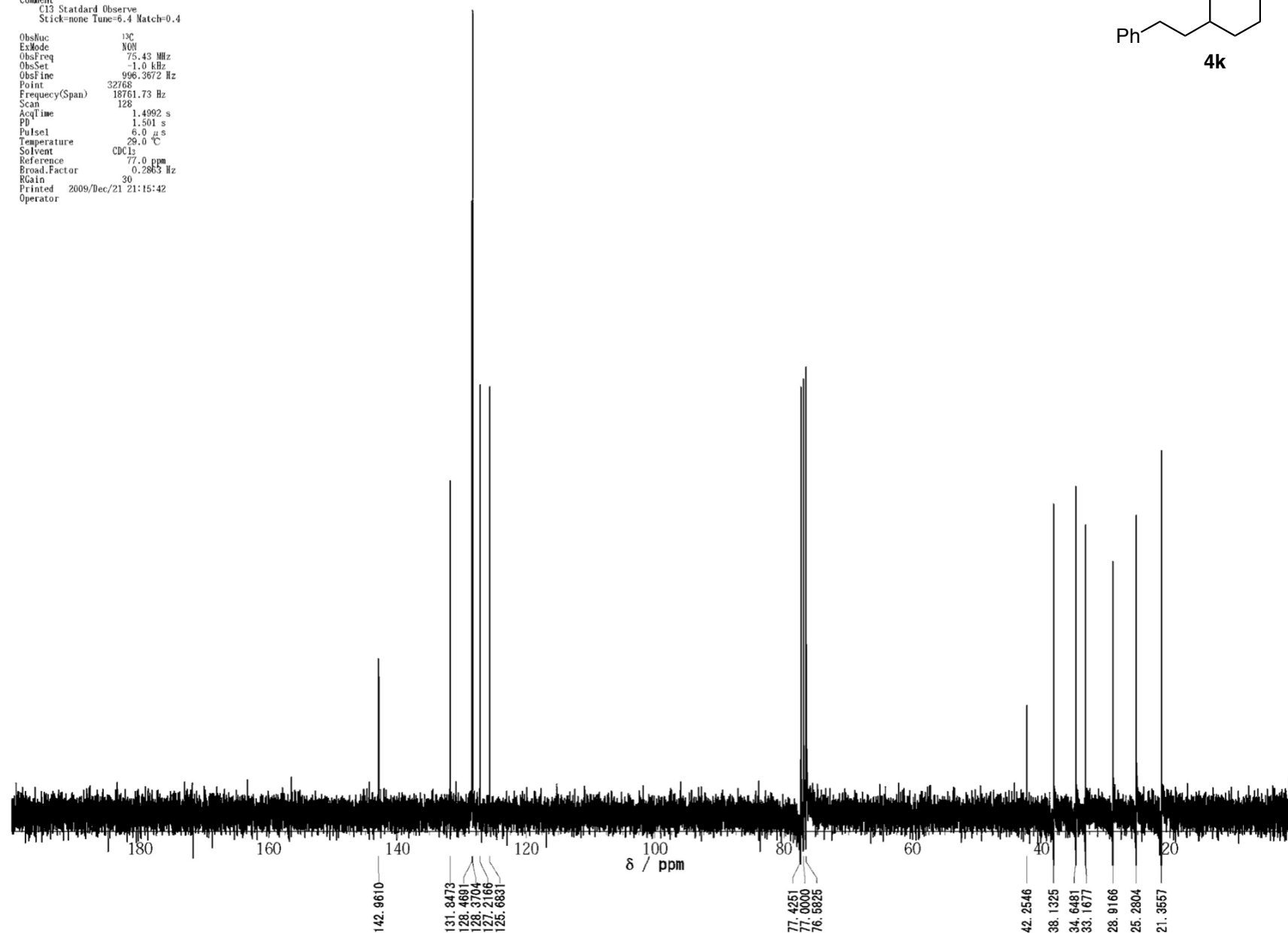
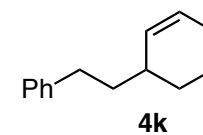
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 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 512
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
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 Operator



Original File:
 Date Oct 24 09
 Comment
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 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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 Operator

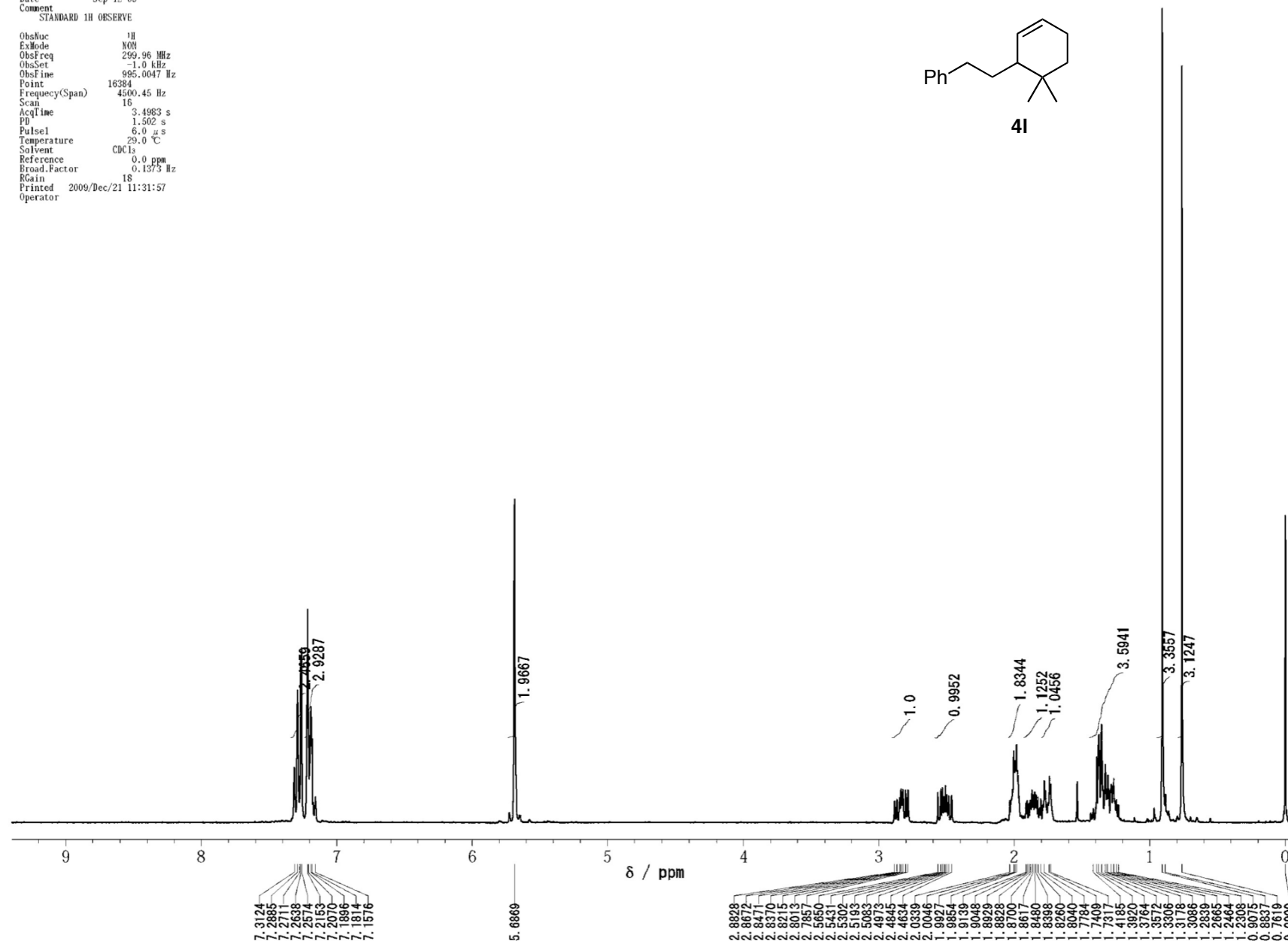
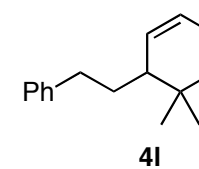


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 ObsFine 996.3672 Hz
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 Frequency(Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
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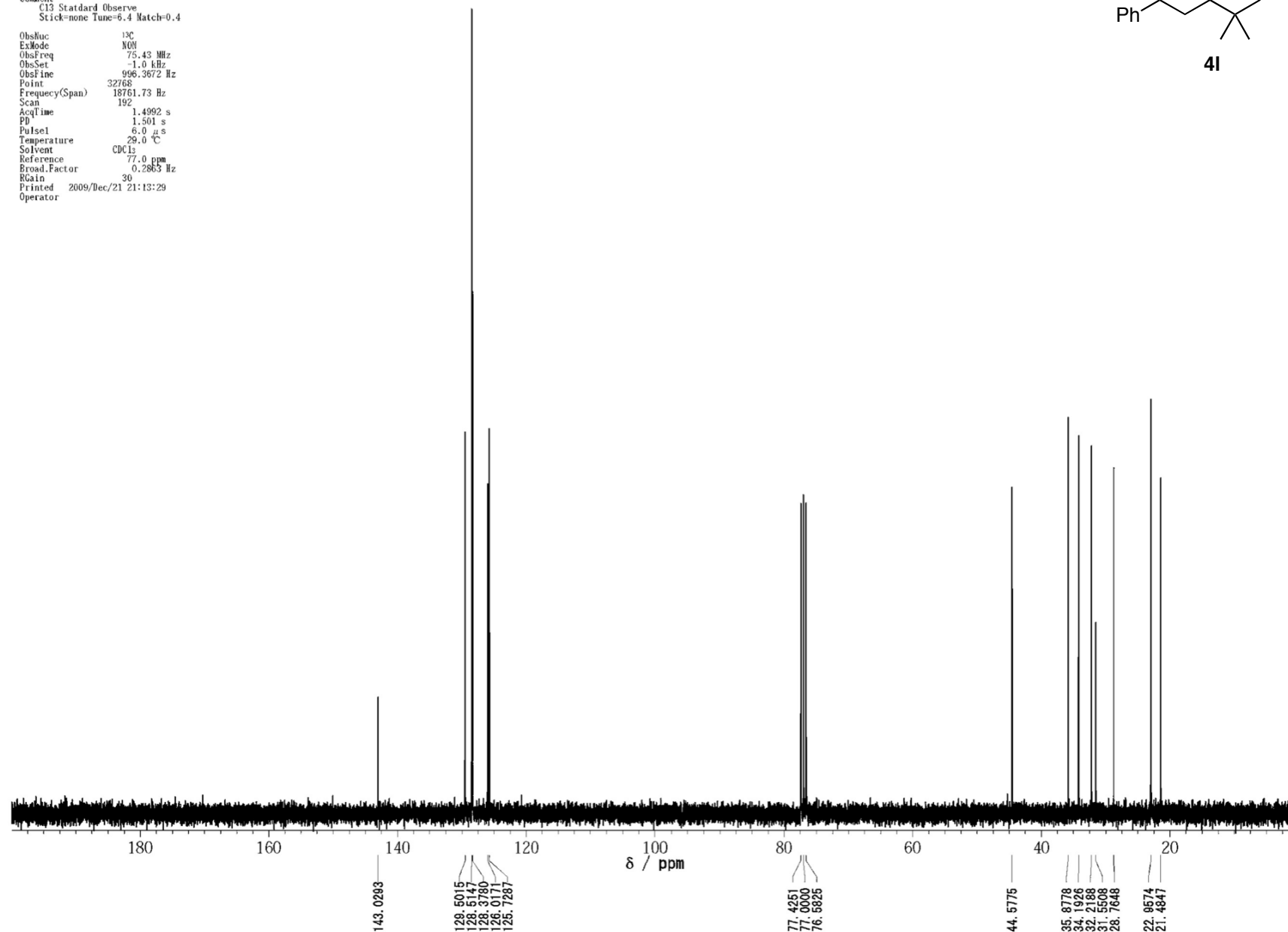
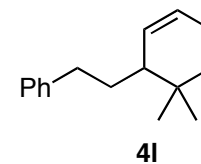


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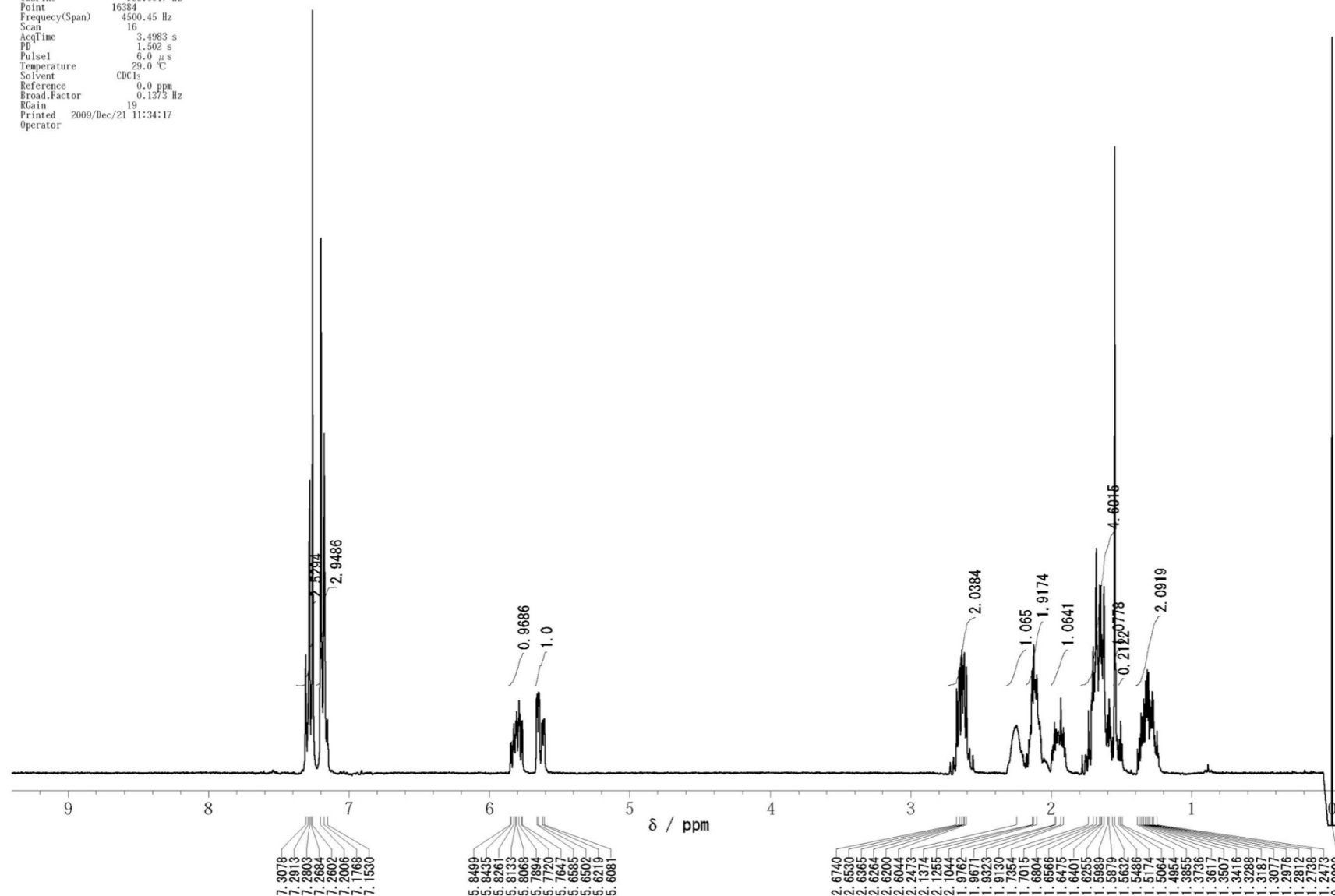
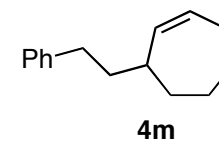
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 ObsFine 995.0047 Hz
 Point 16384
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 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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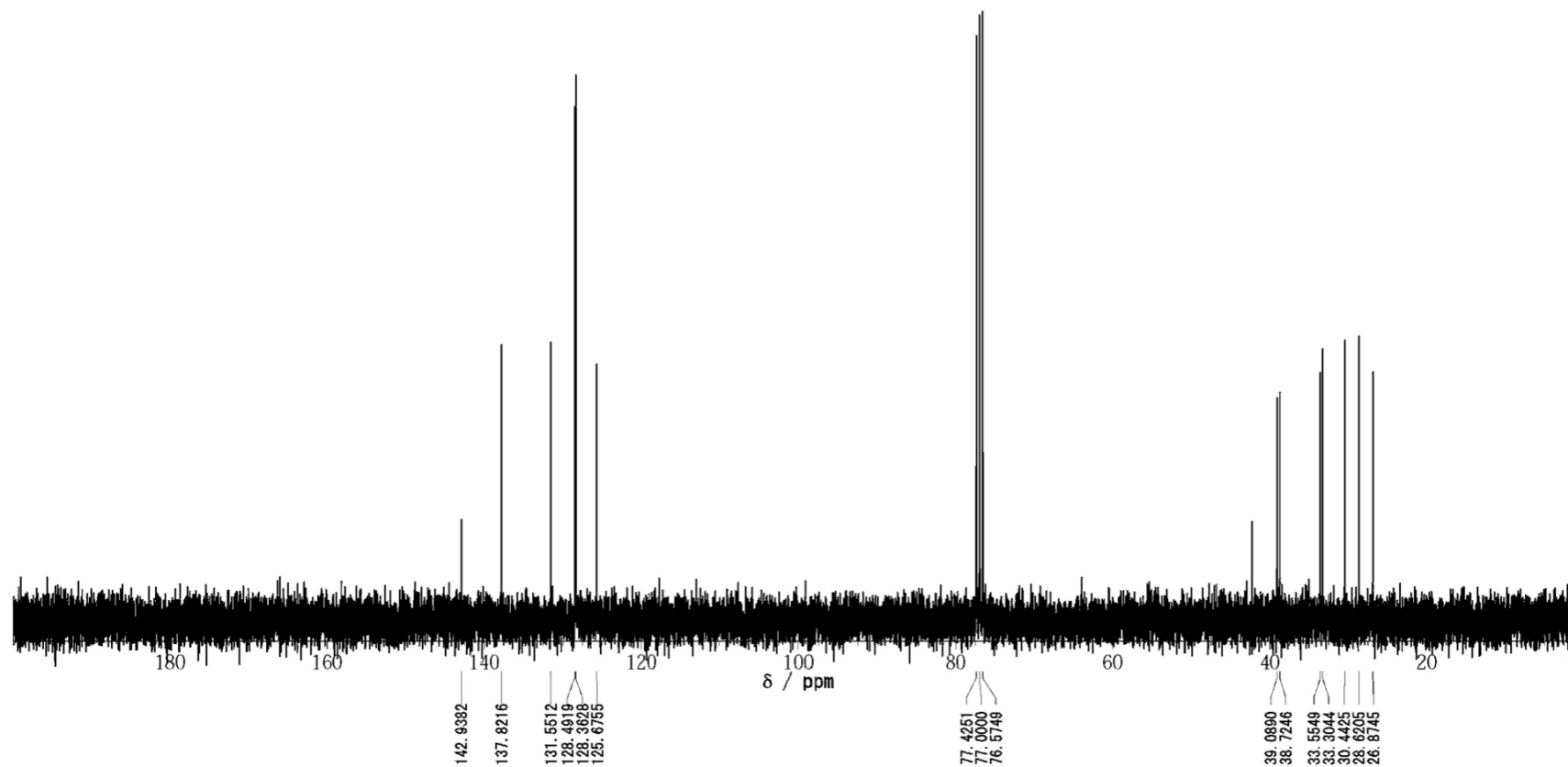
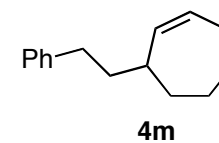
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 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 192
 AcqTime 1.4992 s
 PD 1.501 s
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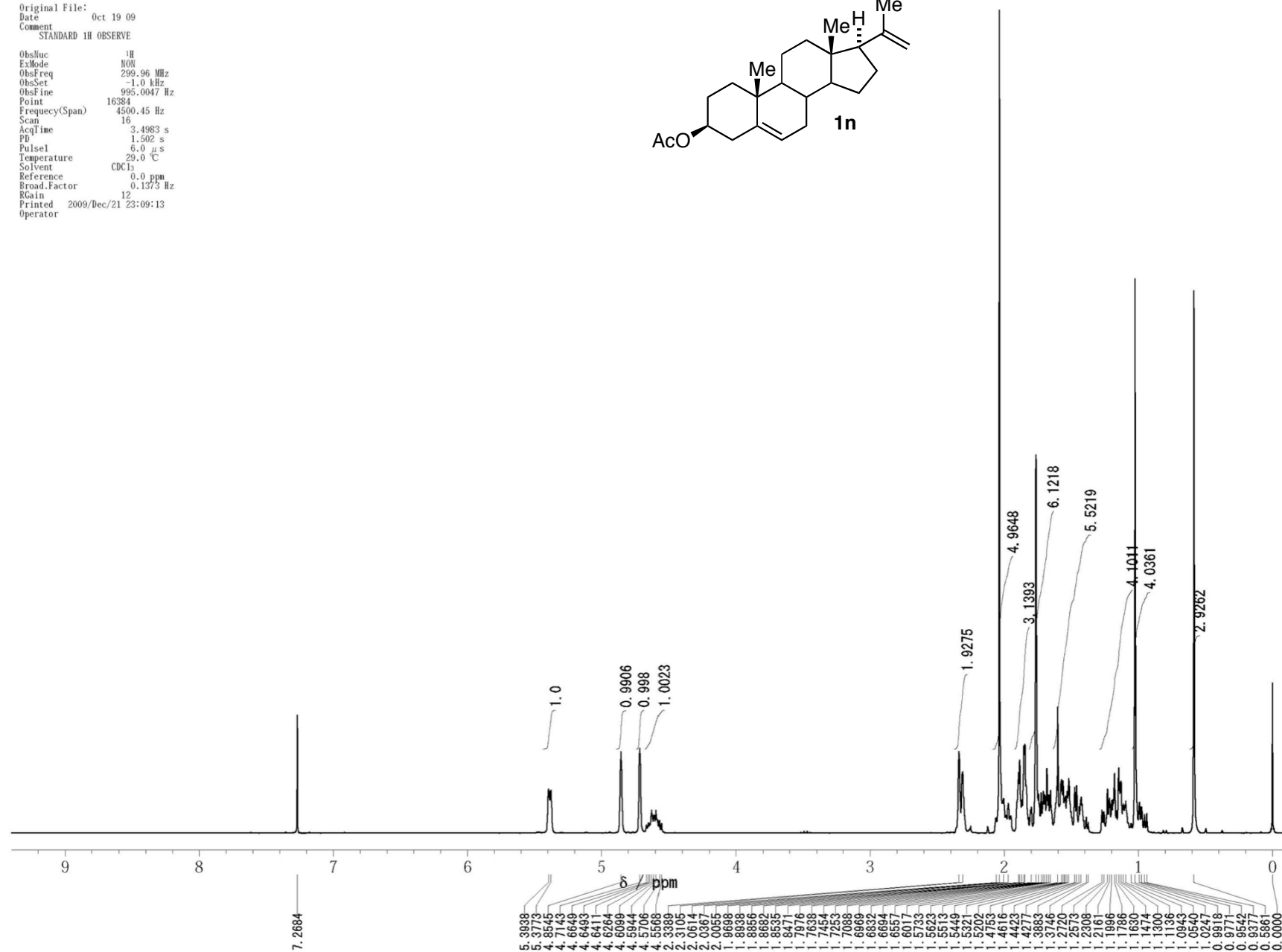
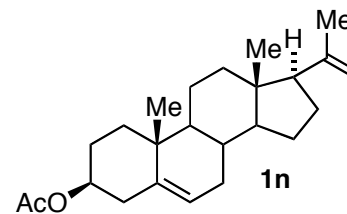
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 Point 16384
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 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
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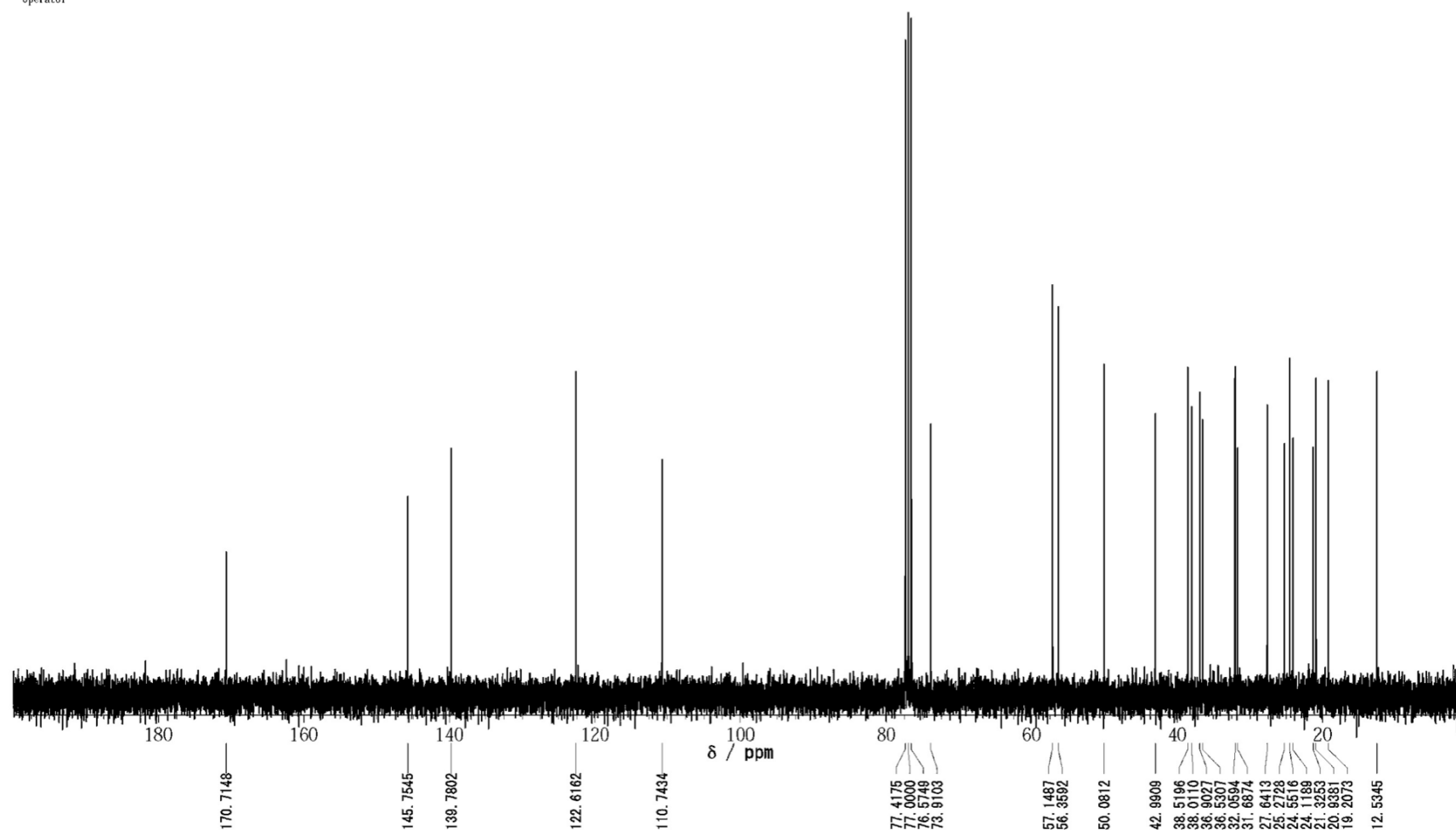
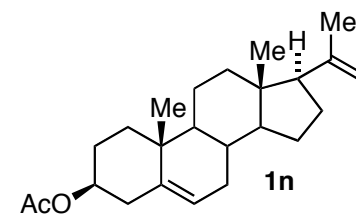
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 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
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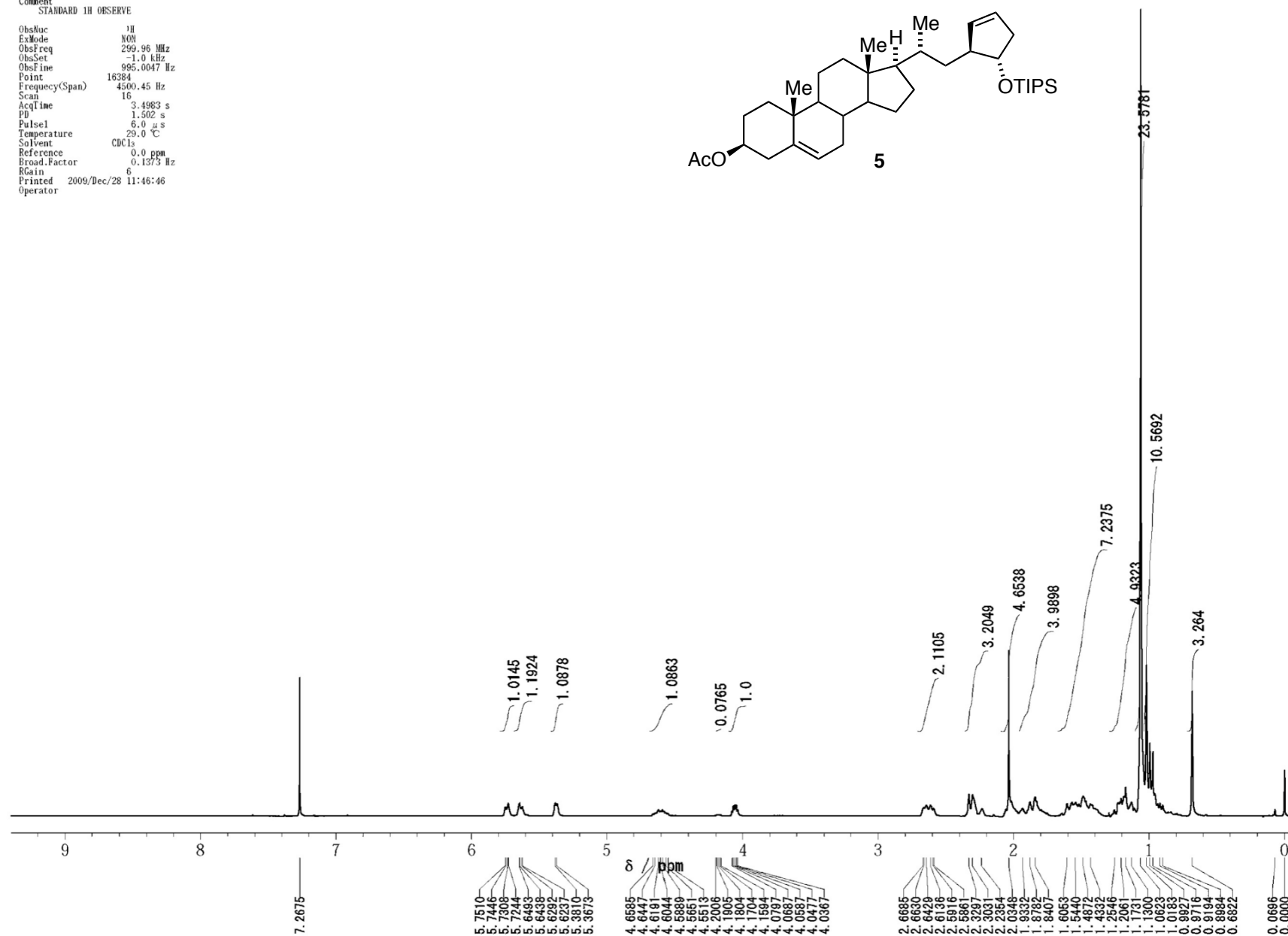
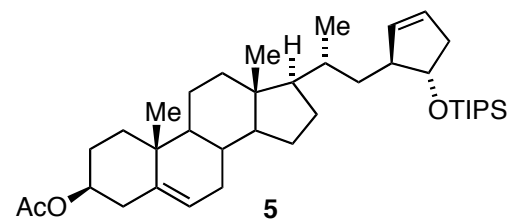


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Original File: Dec 22 09
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Frequency(Span) 18761.73 Hz
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Pulse 6.0 μs
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Operator

