Electronic Supplementary Material (ESI) for Chemical Communications

Catalytic Asymmetric Construction of Tetrasubstituted Carbon Stereocenters by Conjugate Addition of Dialkyl Phosphine Oxides to β,β-Disubstituted α,β-Unsaturated Carbonyl Compounds

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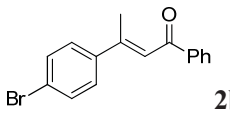
General remarks

All reactions were performed under an argon atmosphere and solvents were dried according to established procedures. ^{1}H NMR (300 M), ^{13}C NMR (75 M) and ^{31}P NMR (121 M) spectra were obtained in $CDCl_3$. The chemical shifts are reported in ppm relative to internal standard TMS (^{1}H NMR), to residual signals of the solvents ($CHCl_3$, 7.26 ppm for ^{1}H NMR and 77.0 ppm for ^{13}C NMR) and to external standard 85% H_3PO_4 (^{31}P NMR). The enantiomeric excess was determined by HPLC analysis.

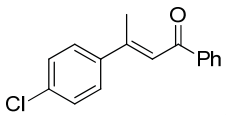
Materials

Diethylphosphine oxide was commercially available. Other dialkylphosphine oxides were prepared according to literature procedures.[1][2] **L2** was prepared according to the previous reported procedures.[3] Et_2Zn was freshly prepared[4] and diluted to 1.0 M in toluene before use. Pyridine was freshly distilled before use. β,β-Disubstituted α,β-unsaturated carbonyl compounds were prepared according to the previous reported procedures.[5] New substrates are given below:



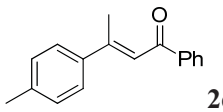
2b

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 – 7.79 (m, 2H), 7.54 – 7.43 (m, 1H), 7.43 – 7.31 (m, 4H), 7.12 – 7.03 (m, 2H), 6.75 (d, *J* = 1.4 Hz, 1H), 2.28 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.4, 151.4, 139.5, 137.9, 132.8, 131.2, 129.0, 128.7, 128.4, 124.4, 122.0, 26.5.



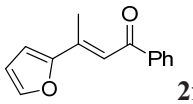
2c

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.07 – 7.91 (m, 2H), 7.61 – 7.43 (m, 5H), 7.38 (d, *J* = 8.4 Hz, 2H), 7.14 (dd, *J* = 2.4, 1.1 Hz, 1H), 2.56 (d, *J* = 1.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.7, 153.4, 141.0, 139.1, 135.1, 132.7, 128.8, 128.6, 128.2, 127.8, 122.3, 18.7.



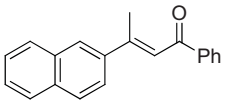
2e

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 – 7.94 (m, 2H), 7.59 – 7.42 (m, 5H), 7.27 – 7.19 (m, 2H), 7.19 – 7.14 (m, 1H), 2.59 (d, *J* = 1.3 Hz, 3H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.8, 155.2, 139.8, 139.5, 139.3, 132.4, 129.3, 128.5, 128.2, 126.4, 121.2, 21.2, 18.7.



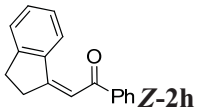
2f

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.17 – 7.83 (m, 2H), 7.64 – 7.37 (m, 5H), 6.75 (d, *J* = 3.4 Hz, 1H), 6.51 (dd, *J* = 3.4, 1.7 Hz, 1H), 2.52 (d, *J* = 1.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.4, 154.8, 144.1, 141.9, 139.6, 132.3, 128.5, 128.1, 116.5, 112.4, 112.3, 15.66.



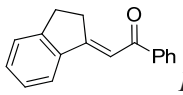
2g

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 5.2, 3.1 Hz, 3H), 7.95 – 7.80 (m, 3H), 7.70 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.61 – 7.44 (m, 5H), 7.36 – 7.29 (m, 1H), 2.71 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.9, 154.8, 139.9, 139.4, 133.6, 133.2, 132.6, 128.6, 128.5, 128.3, 128.2^{7}, 127.62, 126.8, 126.6, 126.2, 124.1, 122.5, 18.9.



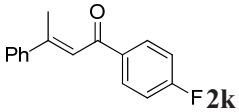
***Z*-2h**

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 5.3, 3.3 Hz, 2H), 7.56 (tt, *J* = 7.6, 1.3 Hz, 1H), 7.50 – 7.42 (m, 3H), 7.39 – 7.27 (m, 2H), 7.22 (td, *J* = 7.2, 1.3 Hz, 1H), 6.34 (s, 1H), 4.22 (dd, *J* = 3.3, 1.7 Hz, 2H), 3.38 (d, *J* = 1.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.2, 144.5, 143.9, 137.5, 136.4, 133.1, 131.8, 128.4^{9}, 128.4^{5}, 126.1, 124.8, 123.7, 119.0, 38.1, 38.0.



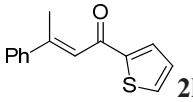
***E*-2h**

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.09 – 7.98 (m, 2H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.58 – 7.43 (m, 4H), 7.39 (d, *J* = 3.9 Hz, 2H), 7.34 – 7.25 (m, 1H), 3.51 – 3.42 (m, 2H), 3.16 – 3.07 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.7, 164.7, 150.4, 140.4, 139.8, 132.1 131.3, 128.5, 127.9, 126.8, 125.8, 121.6, 111.3, 32.5, 30.8.



2k

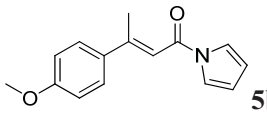
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.09 – 7.96 (m, 2H), 7.63 – 7.50 (m, 2H), 7.47 – 7.34 (m, 3H), 7.20 – 7.07 (m, 3H), 2.59 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 165.4 (d, *J* = 254.0 Hz), 155.4, 142.6, 135.7, 130.8 (d, *J* = 9.2 Hz), 129.2, 128.6, 126.4, 121.6, 115.6 (d, *J* = 21.8 Hz), 18.9.



2l

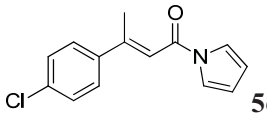
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 3.8, 1.1 Hz, 1H), 7.61 (dd, *J* = 4.9, 1.1 Hz, 1H), 7.59 – 7.51 (m, 2H), 7.47 –

7.37 (m, 3H), 7.13 (dd, *J* = 4.9, 3.8 Hz, 1H), 7.08 (d, *J* = 1.3 Hz, 1H), 2.65 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.5, 155.9, 147.0, 142.7, 133.2, 131.0, 129.2, 128.6, 128.1, 126.5, 121.1, 18.9.



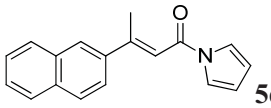
5b

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.9 Hz, 2H), 7.44 – 7.34 (m, 2H), 6.94 (d, *J* = 8.9 Hz, 2H), 6.72 (d, *J* = 1.2 Hz, 1H), 6.38 – 6.17 (m, 2H), 3.85 (s, 3H), 2.60 (d, *J* = 1.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4, 160.8, 157.6, 134.1, 127.8, 119.2, 114.0, 113.9[6], 112.7, 55.4, 18.7.



5c

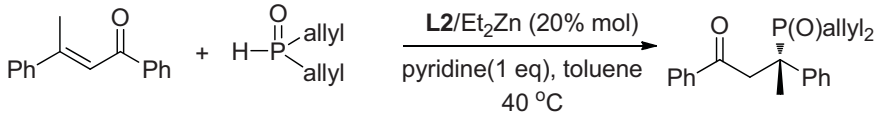
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 – 7.42 (m, 2H), 7.42 – 7.34 (m, 4H), 6.72 (d, *J* = 1.3 Hz, 1H), 6.32 (t, *J* = 3.0 Hz, 2H), 2.58 (d, *J* = 1.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.1, 156.3, 140.3, 135.5, 128.9, 127.7, 119.2, 116.3, 113.1, 18.8.



5d

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 1.5 Hz, 1H), 7.81 – 7.70 (m, 3H), 7.52 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.47 – 7.37 (m, 2H), 7.38 – 7.28 (m, 2H), 6.77 (d, *J* = 1.2 Hz, 1H), 6.29 – 6.16 (m, 2H), 2.61 (d, *J* = 1.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.3, 157.6 139.1, 133.6, 133.0, 128.5, 128.4, 127.6, 127.0, 126.7, 126.1, 123.7, 119.2, 116.2, 112.9, 18.9.

Typical procedure for the asymmetric hydrophosphinylation of β,β-Disubstituted α,β-unsaturated carbonyl compounds

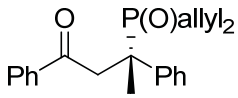


2a **1d** **3a**

Typical experimental procedure: To a stirred solution of **L2** (33.1 mg, 0.05 mmol) in toluene (0.5 mL) was added diethylzinc (100 μL, 1.0 M in toluene, 0.1 mmol) under an argon atmosphere. The mixture was stirred at room temperature for 0.5 h to generate the zinc catalyst. Then the resulting solution of catalyst was transferred by syringe to a stirred mixture of pyridine (20 μL, 1 eq), **2a** (55.5 mg, 0.25 mmol), and diallyl phosphine oxide **1d** (48.8 mg, 0.375 mmol) in toluene (2.0 mL) at rt under an argon atmosphere. After the addition, the mixture was stirred at 40 °C for 12 h.

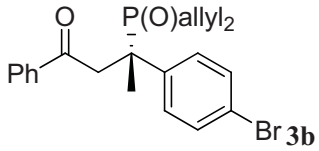
Then the reaction was quenched with saturated NH_4Cl and extracted with CH_2Cl_2. The combined organic layer was dried over Na_2SO_4, and concentrated under vacuum. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate 4:1- ethyl acetate/methanol 40:1).

The racemic products were prepared according to the procedure described above by using racemic **L1**/EtZn.



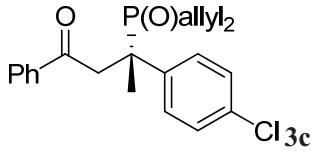
3a

Colorless oil; 94% yield; 99% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 14.5 min, t_{major} = 12.9 min); $[\alpha]^{rt}_D$ = -75.9 (*c* = 1.19, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.90 (d, *J* = 7.2 Hz, 2H), 7.59 – 7.38 (m, 5H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.18 (m, 1H), 6.13 – 5.86 (m, 1H), 5.76 – 5.53 (m, 1H), 5.38 – 4.95 (m, 4H), 4.43 (dd, *J* = 18.0, 8.4 Hz, 1H), 3.57 (dd, *J* = 17.9, 4.7 Hz, 1H), 2.89 – 2.58 (m, 2H), 2.28 (dd, *J* = 13.6, 7.5 Hz, 2H), 1.88 (d, *J* = 16.5 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.2 (d, *J* = 14.1 Hz), 139.4 (d, *J* = 4.2 Hz), 137.3 (d, *J* = 2.1 Hz), 133.1, 128.6 (d, *J* = 8.5 Hz), 128.5^7, 128.5^2, 128.2 (d, *J* = 8.2 Hz), 127.9, 127.0 (d, *J* = 4.7 Hz), 126.9 (d, *J* = 2.9 Hz), 120.4 (d, *J* = 10.8 Hz), 120.1 (d, *J* = 11.0 Hz), 43.9 (d, *J* = 56.8 Hz), 42.5, 31.2 (d, *J* = 58.6 Hz), 30.8 (d, *J* = 62.1 Hz), 19.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.9 ppm; **IR** (neat): 2925, 1691, 1635, 1447, 1352, 1218, 1160, 998, 919, 848, 755, 695 cm^{-1}; **HRMS** (ESI): $C_{22}H_{25}O_2P$ $[M+H]^+$ calcd: 353.1670, found: 353.1670.



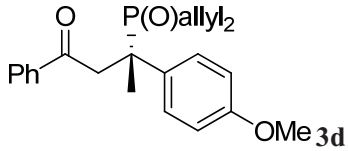
3b

Colorless oil; 98% yield; 96% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 12.1 min, t_{major} = 17.4 min); $[\alpha]^{rt}_D$ = -77.6 (*c* = 1.07, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.89 (d, *J* = 8.3 Hz, 2H), 7.55 (t, *J* = 6.8 Hz, 1H), 7.48 – 7.30 (m, 6H), 6.14 – 5.89 (m, 1H), 5.73 – 5.52 (m, 1H), 5.41 – 4.95 (m, 4H), 4.39 (dd, *J* = 18.1, 8.1 Hz, 1H), 3.56 (dd, *J* = 18.1, 4.2 Hz, 1H), 2.87 – 2.61 (m, 2H), 2.26 (dd, *J* = 13.9, 7.3 Hz, 2H), 1.84 (d, *J* = 16.5 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 195.8 (d, *J* = 14.0 Hz), 138.6 (d, *J* = 4.6 Hz), 137.0 (d, *J* = 1.8 Hz), 133.2, 131.5 (d, *J* = 2.4 Hz), 128.7 (d, *J* = 4.6 Hz), 128.5, 128.3 (d, *J* = 8.5 Hz), 127.8, 127.7 (d, *J* = 7.0 Hz), 121.0 (d, *J* = 3.6 Hz), 120.5 (d, *J* = 11.0 Hz), 120.3 (d, *J* = 11.1 Hz), 43.5 (d, *J* = 56.4 Hz), 42.5, 31.2 (d, *J* = 58.7 Hz), 30.7 (d, *J* = 62.3 Hz), 19.2 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.2 ppm; **IR** (neat): 2979, 1690, 1635, 1490, 1350, 1218, 1161, 1003, 919, 854, 754, 692 cm^{-1}; **HRMS** (ESI): $C_{22}H_{24}BrO_2P$ $[M+H]^+$ calcd: 431.0770, found: 431.0782.



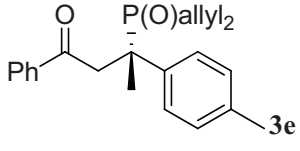
3c

Colorless oil; 94% yield; 98% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 12.5 min, t_{major} = 18.3 min); $[\alpha]^{rt}_D$ = -86.0 (*c* = 1.07, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.89 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.48 – 7.38 (m, 4H), 7.30 (d, *J* = 8.6 Hz, 2H), 6.12 – 5.94 (m, 1H), 5.73 – 5.55 (m, 1H), 5.40 – 4.94 (m, 4H), 4.40 (dd, *J* = 18.1, 8.2 Hz, 1H), 3.55 (dd, *J* = 18.1, 4.4 Hz, 1H), 2.85 – 2.60 (m, 2H), 2.26 (dd, *J* = 13.4, 7.4 Hz, 2H), 1.85 (d, *J* = 16.6 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 195.9 (d, *J* = 14.0 Hz), 138.1 (d, *J* = 4.5 Hz), 137.0 (d, *J* = 2.0 Hz), 133.3, 132.8 (d, *J* = 3.6 Hz), 128.6, 128.5, 128.4 (d, *J* = 4.5 Hz), 128.3[7] (d, *J* = 8.7 Hz), 127.7[9], 127.7[7] (d, *J* = 8.2 Hz), 120.5 (d, *J* = 10.9 Hz), 120.3 (d, *J* = 11.0 Hz), 43.4 (d, *J* = 56.5 Hz), 42.5, 31.2 (d, *J* = 58.6 Hz), 30.7 (d, *J* = 62.3 Hz), 19.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.3 ppm; **IR** (neat): 2922, 1690, 1635, 1493, 1218, 1161, 998, 919, 755, 692 cm^{-1}; **HRMS** (ESI): $C_{22}H_{24}ClO_2P$ $[M+H]^+$ calcd: 387.1275, found: 387.1281.



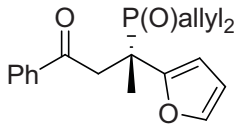
3d

Colorless oil; 97% yield; 98% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{minor} = 9.7 min, t_{major} = 20.3 min); $[\alpha]^{rt}_D$ = -73.0 (*c* = 1.28, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.96 (d, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.47 – 7.38 (m, 3H), 7.38 (d, *J* = 2.3 Hz, 1H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.10 – 5.90 (m, 1H), 5.74 – 5.55 (m, 1H), 5.38 – 5.00 (m, 4H), 4.41 (dd, *J* = 18.0, 8.4 Hz, 1H), 3.76 (s, 3H), 3.70 – 3.45 (m, 1H), 2.93 – 2.61 (m, 2H), 2.49 – 2.23 (m, 2H), 1.87 (d, *J* = 17.0 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.1 (d, *J* = 14.5 Hz), 158.3 (d, *J* = 2.9 Hz), 137.2 (d, *J* = 1.9 Hz), 133.1, 130.7, 128.5, 128.2 (d, *J* = 9.2 Hz), 128.1 (d, *J* = 4.7 Hz), 128.0, 127.8 (d, *J* = 9.0 Hz), 120.6 (d, *J* = 11.0 Hz), 120.3 (d, *J* = 10.5 Hz), 113.9 (d, *J* = 2.5 Hz), 55.1, 43.1 (d, *J* = 58.1 Hz), 42.4, 30.8 (d, *J* = 58.0 Hz), 30.4 (d, *J* = 61.1 Hz), 19.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.6 ppm; **IR** (neat): 2955, 1691, 1635, 1609, 1513, 1254, 1187, 1032, 920, 854, 755, 691 cm^{-1}; **HRMS** (ESI): $C_{23}H_{27}O_3P$ $[M+H]^+$ calcd: 383.1771, found: 383.1775.



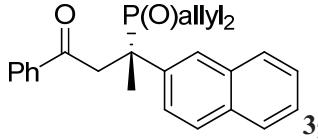
3e

Colorless oil; 94% yield; 96% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 11.7 min, t_{major} = 16.3 min); $[\alpha]^{rt}_{D}$ = -66.0 (*c* = 1.09, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.93 (d, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.34 (dd, *J* = 8.4, 2.3 Hz, 2H), 7.12 (d, *J* = 8.2 Hz, 2H), 6.09 – 5.89 (m, 1H), 5.78 – 5.58 (m, 1H), 5.33 – 4.98 (m, 4H), 4.40 (dd, *J* = 18.0, 8.4 Hz, 1H), 3.60 (dd, *J* = 17.7, 4.0 Hz, 1H), 2.92 – 2.57 (m, 2H), 2.43 – 2.20 (m, 2H), 2.30 (d, *J* = 1.7 Hz, 3H), 1.87 (d, *J* = 16.7 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.1 (d, *J* = 14.3 Hz), 137.2 (d, *J* = 2.0 Hz), 136.4 (d, *J* = 3.2 Hz), 136.0, 133.0, 129.2 (d, *J* = 2.7 Hz), 128.5 (d, *J* = 7.3 Hz), 128.4, 128.1 (d, *J* = 7.7 Hz), 127.9, 126.8 (d, *J* = 4.7 Hz), 120.4 (d, *J* = 11.6 Hz), 120.1 (d, *J* = 11.6 Hz), 43.5 (d, *J* = 57.3 Hz), 42.3, 30.9 (d, *J* = 58.2 Hz), 30.5 (d, *J* = 62.7 Hz), 20.8, 19.2 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.0 ppm; **IR** (neat): 2978, 1691, 1635, 1514, 1450, 1352, 1218, 1161, 999, 919, 853, 756, 613 cm^{-1}; **HRMS** (ESI): $C_{23}H_{27}O_2P$ $[M+H]^+$ calcd: 367.1821, found: 367.1815.



3f

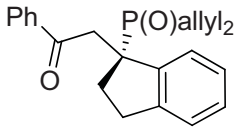
Colorless oil; 93% yield; 98% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 14.3 min, t_{major} = 12.2 min); $[\alpha]^{rt}_{D}$ = -39.3 (*c* = 1.07, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.92 (d, *J* = 7.3 Hz, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.35 (s, 1H), 6.38 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.23 (t, *J* = 3.4 Hz, 1H), 5.95 – 5.67 (m, 2H), 5.31 – 5.10 (m, 4H), 3.97 (dd, *J* = 16.8, 7.1 Hz, 1H), 3.67 (dd, *J* = 16.8, 5.2 Hz, 1H), 2.77 – 2.43 (m, 4H), 1.80 (d, *J* = 14.5 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.2 (d, *J* = 13.5 Hz), 153.4 (d, *J* = 5.5 Hz), 141.5 (d, *J* = 3.3 Hz), 137.1 (d, *J* = 1.9 Hz), 133.1, 128.4, 128.1 (d, *J* = 8.6 Hz), 127.9, 127.8 (d, *J* = 9.0 Hz), 120.3 (d, *J* = 11.0 Hz), 120.2 (d, *J* = 11.0 Hz), 111.1 (d, *J* = 3.0 Hz), 107.7 (d, *J* = 7.1 Hz), 41.5 (d, *J* = 60.0 Hz), 39.7, 31.5 (d, *J* = 60.1 Hz), 30.4 (d, *J* = 61.2 Hz), 17.3 (d, *J* = 2.4 Hz) ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.4 ppm; **IR** (neat): 2916, 1692, 1635, 1450, 1353, 1221, 1160, 998, 932, 757, 693, 603 cm^{-1}; **HRMS** (ESI): $C_{20}H_{23}O_3P$ $[M+H]^+$ calcd: 343.1458, found: 343.1452.



3g

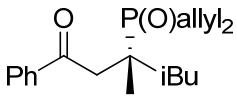
Colorless oil; 94% yield; 96% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 30.7 min, t_{major} = 20.6 min); $[\alpha]^{rt}_{D}$ = -85.5 (*c* = 1.17, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.97 – 7.86 (m, 3H), 7.85 – 7.74 (m, 3H), 7.67 (d, *J* = 8.8 Hz, 1H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.48 – 7.33 (m, 4H), 6.13 – 5.90 (m, 1H), 5.77 – 5.57 (m, 1H), 5.37 – 4.91 (m, 4H), 4.55 (dd, *J* = 18.0, 8.4 Hz, 1H), 3.69 (dd, *J* = 18.0, 4.6 Hz,

1H), 2.93 – 2.60 (m, 2H), 2.29 (dd, *J* = 13.3, 7.6 Hz, 2H), 2.00 (d, *J* = 16.5 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.0 (d, *J* = 14.1 Hz), 137.2 (d, *J* = 2.0 Hz), 136.9 (d, *J* = 3.8 Hz), 133.0^9, 133.0^6 (d, *J* = 4.2 Hz), 132.0 (d, *J* = 2.1 Hz), 128.5, 128.4 (d, *J* = 7.1 Hz), 128.0^7, 128.0^5, 127.9 (d, *J* = 7.2 Hz), 127.8^6, 127.4 (d, *J* = 1.1 Hz), 126.0^9 (d, *J* = 1.5 Hz), 126.0^5 (d, *J* = 4.0 Hz), 125.9 (d, *J* = 0.8 Hz), 125.0 (d, *J* = 3.5 Hz), 120.4 (d, *J* = 10.9 Hz), 120.1 (d, *J* = 11.0 Hz), 44.1 (d, *J* = 56.7 Hz), 42.6, 31.1 (d, *J* = 58.5 Hz), 30.8 (d, *J* = 62.0 Hz), 19.4 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.0 ppm; **IR** (neat): 2978, 1691, 1634, 1349, 1218, 1162, 999, 920, 752, 691, 627 cm^{-1}; **HRMS** (ESI): $C_{26}H_{27}O_2P$ $[M+H]^+$ calcd: 403.1821, found: 403.1823.



3h

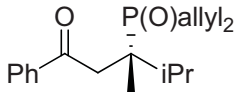
Colorless oil; From Z substrate, 72% yield; 96% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 13.7 min, t_{major} = 15.3 min); $[\alpha]^{rt}_D$ = -27.6 (*c* = 1.09, $CHCl_3$); From *E* substrate, 90% yield; 80% *ee*; $[\alpha]^{rt}_D$ = -23.7 (*c* = 1.01, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 7.5 Hz, 2H), 7.47 – 7.34 (m, 2H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.16 – 6.97 (m, 3H), 5.95 – 5.73 (m, 1H), 5.67 – 5.47 (m, 1H), 5.27 – 4.83 (m, 4H), 3.95 (dd, *J* = 17.2, 9.8 Hz, 1H), 3.67 (dd, *J* = 17.2, 6.0 Hz, 1H), 3.28 – 3.06 (m, 1H), 3.01 – 2.59 (m, 3H), 2.59 – 2.18 (m, 4H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 197.3 (d, *J* = 11.7 Hz), 144.1 (d, *J* = 6.7 Hz), 141.5 (d, *J* = 3.3 Hz), 137.1 (d, *J* = 1.3 Hz), 133.0, 128.3^4 (d, *J* = 6.3 Hz), 128.2^9, 127.9, 127.8 (d, *J* = 2.5 Hz), 127.7 (d, *J* = 7.8 Hz), 126. 7 (d, *J* = 2.5 Hz), 124.7 (d, *J* = 3.1 Hz), 124.5 (d, *J* = 2.1 Hz), 120.2 (d, *J* = 10.9 Hz), 120.0 (d, *J* = 11.0 Hz), 52.2 (d, *J* = 61.1 Hz), 42.1, 31.5 (d, *J* = 58.0 Hz), 31.3 (d, *J* = 3.2 Hz), 30.6 (d, *J* = 62.2 Hz), 30.4 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.5 ppm; **IR** (neat): 2947, 1690, 1635, 1451, 1354, 1216, 1157, 998, 919, 754, 691, 615 cm^{-1}; **HRMS** (ESI): $C_{23}H_{25}O_2P$ $[M+H]^+$ calcd: 365.1665, found: 365.1660.



3i

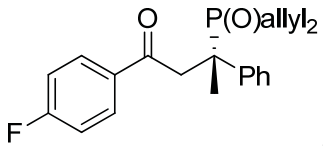
Colorless oil; 90% yield; 93% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 9.8 min, t_{major} = 7.3 min); $[\alpha]^{rt}_D$ = +1.8 (*c* = 1.08, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.97 (d, *J* = 7.1 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 2H), 6.07 – 5.83 (m, 2H), 5.30 – 5.16 (m, 4H), 3.57 (dd, *J* = 17.4, 10.9 Hz, 1H), 3.27 (dd, *J* = 17.4, 14.9 Hz, 1H), 2.96 – 2.64 (m, 4H), 1.99 – 1.82 (m, 2H), 1.79 – 1.62 (m, 1H), 1.43 (d, *J* = 15.9 Hz, 3H), 0.94 (d, *J* = 6.4 Hz, 3H), 0.90 (d, *J* = 6.4 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 198.7 (d, *J* = 7.7 Hz), 137.5, 133.1, 128.9 (d, *J* = 8.2 Hz), 128.8^6 (d, *J* = 8.3 Hz), 128.6, 128.0, 120.0 (d, *J* = 10.9 Hz,

overlapped), 42.1, 41.6, 40.5 (d, J = 60.4 Hz), 31.8 (d, J = 58.6 Hz), 31.7 (d, J = 58.2 Hz), 25.2, 25.1, 24.3 (d, J = 8.1 Hz), 21.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +52.9 ppm; **IR** (neat): 2957, 1689, 1635, 1451, 1361, 1218, 1160, 917, 754, 692, 616 cm^{-1}; **HRMS** (ESI): $C_{20}H_{29}O_2P$ $[M+H]^+$ calcd: 333.1978, found: 333.1970.



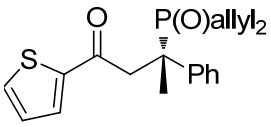
3j

Colorless oil; 92% yield; 90% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 8.3 min, t_{major} = 7.7 min); $[\alpha]^{rt}_D$ = -0.9 (c = 1.09, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.98 (d, J = 7.2 Hz, 2H), 7.58 (t, J = 7.3 Hz, 1H), 7.47 (t, J = 7.5 Hz, 2H), 6.07 – 5.86 (m, 2H), 5.31 – 5.10 (m, 4H), 3.50 – 3.24 (m, 2H), 3.05 – 2.67 (m, 4H), 2.66 – 2.47 (m, 1H), 1.31 (d, J = 15.2 Hz, 3H), 1.08 (d, J = 6.9 Hz, 3H), 0.98 (d, J = 6.8 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 198.9 (d, J = 3.8 Hz), 137.2, 133.2, 129.3^1 (d, J = 8.1 Hz), 129.2^6 (d, J = 8.8 Hz), 128.6, 128.0, 119.9 (d, J = 11.7 Hz), 119.7 (d, J = 11.5 Hz), 43.5 (d, J = 58.8 Hz), 41.7, 33.7 (d, J = 58.7 Hz), 33.1 (d, J = 57.8 Hz), 30.3, 19.0 (d, J = 4.1 Hz), 18.2 (d, J = 6.7 Hz), 17.9 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +53.8 ppm; **IR** (neat): 2970, 1687, 1635, 1450, 1394, 1221, 1157, 998, 916, 747, 692, 615 cm^{-1}; **HRMS** (ESI): $C_{19}H_{27}O_2P$ $[M+H]^+$ calcd: 319.1821, found: 319.1827.



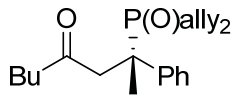
3k

White solid, m.p. 113-115 ^{o}C; 91% yield; 98% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 9.1 min, t_{major} = 13.5 min); $[\alpha]^{rt}_D$ = -71.0 (c = 1.06, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.92 (dd, J = 8.9, 5.4 Hz, 2H), 7.48 (dd, J = 7.6, 1.7 Hz, 2H), 7.34 (t, J = 7.7 Hz, 2H), 7.28 – 7.20 (m, 1H), 7.08 (t, J = 8.7 Hz, 2H), 6.09 – 5.89 (m, 1H), 5.71 – 5.53 (m, 1H), 5.35 – 4.95 (m, 4H), 4.40 (dd, J = 17.9, 8.4 Hz, 1H), 3.53 (dd, J = 17.8, 4.6 Hz, 1H), 2.89 – 2.59 (m, 2H), 2.27 (dd, J = 13.7, 7.5 Hz, 2H), 1.88 (d, J = 16.6 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 194.6 (d, J = 14.1 Hz), 165.6 (d, J = 255.0 Hz), 139.3 (d, J = 4.2 Hz), 133.7 (dd, J = 3.0, 2.3 Hz), 130.5 (d, J = 9.3 Hz), 128.5^2 (d, J = 8.7 Hz), 128.4^7 (d, J = 2.5 Hz), 128.1 (d, J = 8.2 Hz), 126.8^9 (d, J = 4.7 Hz), 126.8^8 (d, J = 2.7 Hz), 120.4 (d, J = 10.8 Hz), 120.1 (d, J = 11.0 Hz), 115.5 (d, J = 21.8 Hz), 43.8 (d, J = 56.7 Hz), 42.4, 31.2 (d, J = 58.6 Hz), 30.7 (d, J = 62.2 Hz), 19.2 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.8 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$): δ = -105.0 ppm; **IR** (neat): 2923, 1692, 1597, 1504, 1415, 1220, 1159, 999, 921, 837, 700, 614 cm^{-1}; **HRMS** (ESI): $C_{22}H_{24}FO_2P$ $[M+H]^+$ calcd: 371.1571, found: 371.1580.



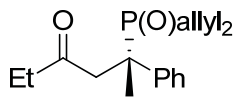
3l

Colorless oil; 97% yield; 98% *ee* determined by HPLC on a Chiralpak OJ-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 15.9 min, t_{major} = 10.3 min); $[\alpha]^{rt}_D$ = -42.9 (*c* = 1.12, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.79 (s, 1H), 7.58 (d, *J* = 4.3 Hz, 1H), 7.50 (d, *J* = 7.7 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.28 – 7.19 (m, 1H), 7.08 (dd, *J* = 4.7, 4.0 Hz, 1H), 6.08 – 5.87 (m, 1H), 5.71 – 5.51 (m, 1H), 5.37 – 4.92 (m, 4H), 4.33 (dd, *J* = 17.3, 8.3 Hz, 1H), 3.53 (dd, *J* = 17.3, 4.7 Hz, 1H), 2.90 – 2.58 (m, 2H), 2.29 (dd, *J* = 13.5, 7.5 Hz, 2H), 1.88 (d, *J* = 16.7 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 189.2 (d, *J* = 14.6 Hz), 144.7 (d, *J* = 2.7 Hz), 138.9 (d, *J* = 4.0 Hz), 133.8, 132.2, 128.4 (d, *J* = 2.5 Hz), 128.3 (d, *J* = 8.5 Hz), 128.1, 127.8 (d, *J* = 8.1 Hz), 127.0[1] (d, *J* = 4.6 Hz), 126.9[6] (d, *J* = 2.5 Hz), 120.5 (d, *J* = 11.0 Hz), 120.2 (d, *J* = 11.0 Hz), 43.9 (d, *J* = 56.8 Hz), 43.0, 31.0 (d, *J* = 58.6 Hz), 30.6 (d, *J* = 62.4 Hz), 19.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.3 ppm; **IR** (neat): 2980, 1666, 1517, 1416, 1228, 1160, 1060, 917, 847, 729, 700 cm^{-1}; **MS** (ESI): $C_{18}H_{25}O_3P$ $[M+H]^+$ calcd: 359.1, found: 359.3.



3m

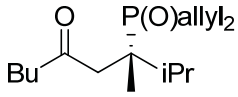
Colorless oil; 98% yield; 99% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, t_{minor} = 9.2 min, t_{major} = 10.3 min); $[\alpha]^{rt}_D$ = -5.3 (*c* = 1.14, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.48 (d, *J* = 7.8 Hz, 2H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.31 – 7.21 (m, 1H), 6.04 – 5.83 (m, 1H), 5.64 – 5.47 (m, 1H), 5.35 – 4.91 (m, 4H), 3.78 (dd, *J* = 16.8, 8.4 Hz, 1H), 2.91 (dd, *J* = 16.8, 4.8 Hz, 1H), 2.81 – 2.55 (m, 2H), 2.35 – 2.07 (m, 4H), 1.76 (d, *J* = 16.7 Hz, 3H), 1.45 – 1.31 (m, 2H), 1.14 (dq, *J* = 14.3, 7.3 Hz, 2H), 0.80 (t, *J* = 7.3 Hz, 3H)ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 207.6 (d, *J* = 13.4 Hz), 139.1 (d, *J* = 4.2 Hz), 128.5 (d, *J* = 2.5 Hz), 128.4 (d, *J* = 8.7 Hz), 127.9 (d, *J* = 8.2 Hz), 127.0 (d, *J* = 3.1 Hz), 126.9[9] (d, *J* = 4.5 Hz), 120.3 (d, *J* = 10.8 Hz), 120.0 (d, *J* = 11.0 Hz), 46.7 (d, *J* = 1.2 Hz), 44.1 (d, *J* = 1.4 Hz), 43.5 (d, *J* = 56.7 Hz), 31.2 (d, *J* = 58.5 Hz), 30.8 (d, *J* = 62.0 Hz), 25.4, 22.0, 19.0, 13.7 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.4 ppm; **IR** (neat): 2957, 1716, 1635, 1419, 1378, 1163, 917, 847, 701, 617 cm^{-1}; **HRMS** (ESI): $C_{20}H_{29}O_2P$ $[M+H]^+$ calcd: 333.1978, found: 333.1968.



3n

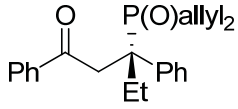
Colorless oil; 84% yield; >99% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 9.2 min, t_{major} = 11.9 min); $[\alpha]^{rt}_D$ = +0.9 (*c* = 1.10, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ =

7.49 (d, J = 7.7 Hz, 2H), 7.36 (t, J = 7.6 Hz, 2H), 7.31 – 7.22 (m, 1H), 6.03 – 5.85 (m, 1H), 5.67 – 5.46 (m, 1H), 5.33 – 4.91 (m, 4H), 3.77 (dd, J = 16.7, 8.5 Hz, 1H), 2.92 (dd, J = 16.7, 4.9 Hz, 1H), 2.68 (pd, J = 14.6, 7.5 Hz, 2H), 2.41 – 2.04 (m, 2H), 2.20 (dd, J = 13.8, 7.2 Hz, 2H), 1.76 (d, J = 16.6 Hz, 3H), 0.87 (t, J = 7.3 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 207.9 (d, J = 13.4 Hz), 139.2 (d, J = 4.2 Hz), 128.5 (d, J = 2.3 Hz), 128.4 (d, J = 8.0 Hz), 128.0 (d, J = 8.2 Hz), 127.0[3] (d, J = 3.3 Hz), 127.0[2] (d, J = 4.4 Hz), 120.3 (d, J = 10.8 Hz), 119.9 (d, J = 11.0 Hz), 46.6 (d, J = 1.2 Hz), 43.6 (d, J = 56.8 Hz), 37.6 (d, J = 1.5 Hz), 31.2 (d, J = 58.5 Hz), 30.9 (d, J = 61.9 Hz), 19.1, 7.4 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.2 ppm; **IR** (neat): 2978, 1717, 1635, 1419, 1163, 918, 847, 701, 617 cm^{-1}; **HRMS** (ESI): $C_{18}H_{25}O_2P$ $[M+H]^+$ calcd: 305.1665, found: 305.1670.



3o

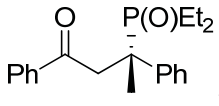
Colorless oil; 87% yield; 96% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, t_{minor} = 9.4 min, t_{major} = 8.8 min); $[\alpha]^{rt}_D$ = +1.4 (c = 1.46, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 6.07 – 5.81 (m, 2H), 5.29 – 5.11 (m, 4H), 2.99 – 2.62 (m, 6H), 2.47 (t, J = 7.4 Hz, 2H), 2.46 – 2.30 (m, 1H), 1.62 – 1.48 (m, 2H), 1.31 (dd, J = 15.1, 7.4 Hz, 2H), 1.21 (d, J = 15.4 Hz, 3H), 1.00 (t, J = 7.1 Hz, 6H), 0.91 (t, J = 7.3 Hz, 3H)ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 210.0 (d, J = 4.1 Hz), 129.2[3] (d, J = 8.1 Hz), 129.1[8] (d, J = 8.7 Hz), 119.6 (d, J = 11.0 Hz), 119.5 (d, J = 11.1 Hz), 45.3, 43.5, 43.1 (d, J = 58.9 Hz), 33.5 (d, J = 58.3 Hz), 32.9 (d, J = 57.4 Hz), 30.4, 25.6, 22.1, 18.7 (d, J = 4.6 Hz), 18.2 (d, J = 6.3 Hz), 17.6, 13.7 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +53.1 ppm; **IR** (neat): 2960, 1713, 1635, 1463, 1378, 1159, 914, 612 cm^{-1}; **HRMS** (ESI): $C_{17}H_{31}O_2P$ $[M+H]^+$ calcd: 299.2134, found: 299.2139.



3p

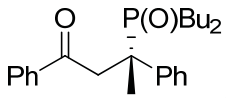
Colorless oil; 84% yield; 94% *ee* determined by HPLC on a Chiralpak OJ-H column (hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, t_{minor} = 12.8 min, t_{major} = 9.4 min); $[\alpha]^{rt}_D$ = -58.9 (c = 1.00, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 8.01 (d, J = 7.3 Hz, 2H), 7.57 (t, J = 7.3 Hz, 1H), 7.51 – 7.39 (m, 4H), 7.34 (t, J = 7.6 Hz, 2H), 7.30 – 7.18 (m, 1H), 6.06 – 5.80 (m, 1H), 5.79 – 5.60 (m, 1H), 5.31 – 4.97 (m, 4H), 4.11 (dd, J = 18.7, 8.9 Hz, 1H), 3.97 (dd, J = 18.7, 11.2 Hz, 1H), 2.91 (td, J = 15.4, 6.9 Hz, 1H), 2.72 – 2.48 (m, 3H), 2.47 – 2.18 (m, 2H), 1.05 (t, J = 7.4 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.6 (d, J = 10.3 Hz), 139.6 (d, J = 2.8 Hz), 136.9 (d, J = 1.0 Hz), 133.1, 128.7[1] (d, J = 8.0 Hz), 128.6[8] (d, J = 8.6 Hz), 128.5, 128.3 (d, J = 2.7 Hz), 127.8, 127.3 (d, J = 4.6 Hz), 126.8 (d, J = 3.1 Hz), 120.0 (d, J = 11.2 Hz), 119.6 (d, J = 11.0 Hz), 48.2 (d, J = 55.0 Hz), 38.4, 32.0 (d, J = 60.6 Hz), 31.5 (d, J = 59.6 Hz), 25.3, 9.8 (d,

J = 5.3 Hz) ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.7 ppm; **IR** (neat): 2973, 1691, 1635, 1448, 1216, 1159, 918, 754, 696, 613 cm^{-1}; **HRMS** (ESI): $C_{23}H_{27}NO_2P$ $[M+H]^+$ calcd: 367.1821, found: 367.1820.



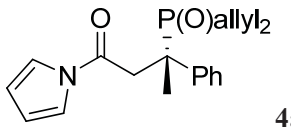
3q

White solid, m.p. 159-161 °C; 82% yield; 99% *ee* determined by HPLC on a Chiralpak OJ-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 11.0 min, t_{major} = 6.9 min); $[\alpha]^{rt}_D$ = -82.6 (*c* = 1.11, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.91 (d, *J* = 7.5 Hz, 2H), 7.58 – 7.37 (m, 5H), 7.35 – 7.25 (m, 2H), 7.25 – 7.11 (m, 1H), 4.43 (dd, *J* = 17.8, 7.9 Hz, 1H), 3.46 (dd, *J* = 17.8, 4.6 Hz, 1H), 2.04 – 1.78 (m, 1H), 1.83 (d, *J* = 15.7 Hz, 3H), 1.78 – 1.60 (m, 1H), 1.53 – 1.34 (m, 2H), 1.24 (dt, *J* = 15.3, 7.7 Hz, 3H), 0.84 (dt, *J* = 15.4, 7.7 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.5 (d, *J* = 13.8 Hz), 139.8 (d, *J* = 4.2 Hz), 137.4 (d, *J* = 1.8 Hz), 133.0, 128.4, 128.3 (d, *J* = 2.5 Hz), 127.9, 126.9 (d, *J* = 4.4 Hz), 126.5 (d, *J* = 2.7 Hz), 42.8 (d, *J* = 58.0 Hz), 42.2, 19.3, 17.8 (d, *J* = 61.5 Hz), 16.8 (d, *J* = 65.3 Hz), 6.5 (d, *J* = 5.5 Hz), 6.1 (d, *J* = 5.4 Hz) ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +56.3 ppm; **IR** (neat): 2977, 2940, 1691, 1449, 1351, 1217, 1156, 1030, 1000, 754, 696, 618 cm^{-1}; **HRMS** (ESI): $C_{20}H_{25}O_2P$ $[M+H]^+$ calcd: 329.1665, found: 329.1655.



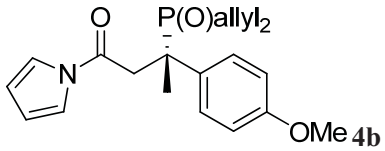
3r

White solid, m.p. 108-110 °C; 90% yield; 99% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 10.6 min, t_{major} = 8.1 min); $[\alpha]^{rt}_D$ = -86.5 (*c* = 1.04, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.92 (d, *J* = 7.4 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.49 – 7.36 (m, 4H), 7.35 – 7.25 (m, 2H), 7.25 – 7.15 (m, 1H), 4.44 (dd, *J* = 17.8, 7.9 Hz, 1H), 3.45 (dd, *J* = 17.8, 4.5 Hz, 1H), 1.97 – 1.75 (m, 1H), 1.82 (d, *J* = 15.8 Hz, 3H), 1.74 – 1.58 (m, 2H), 1.56 – 1.32 (m, 5H), 1.28 – 1.11 (m, 3H), 1.09 – 0.88 (m, 1H), 0.95 (t, *J* = 7.2 Hz, 3H), 0.75 (t, *J* = 7.0 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 196.6 (d, *J* = 14.0 Hz), 139.9 (d, *J* = 3.9 Hz), 137.5 (d, *J* = 1.8 Hz), 133.1, 128.5, 128.4 (d, *J* = 2.5 Hz), 128.0, 127.0 (d, *J* = 4.4 Hz), 126.6 (d, *J* = 2.4 Hz), 43.0 (d, *J* = 57.8 Hz), 42.3, 25.3 (d, *J* = 60.2 Hz), 24.7 (d, *J* = 13.7 Hz), 24.3[8] (d, *J* = 13.7 Hz), 24.3[7] (d, *J* = 4.6 Hz), 24.1 (d, *J* = 65.2 Hz), 24.0 (d, *J* = 4.4 Hz), 19.4, 13.7, 13.5 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +54.3 ppm; **IR** (neat): 2958, 1692, 1449, 1350, 1218, 1159, 999, 754, 696 cm^{-1}; **HRMS** (ESI): $C_{24}H_{33}O_2P$ $[M+H]^+$ calcd: 385.2291, found: 385.2286.



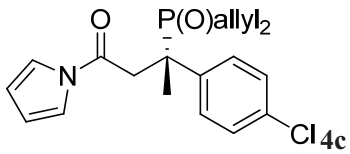
4a

Colorless oil; 88% yield; 99% *ee* determined by HPLC on a Chiralpak OJ-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 11.0 min, t_{major} = 7.6 min); $[\alpha]^{rt}_{D}$ = -57.5 (*c* = 1.04, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.50 (d, *J* = 7.7 Hz, 2H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.32 – 7.23 (m, 3H), 6.25 (t, *J* = 2.1 Hz, 2H), 6.08 – 5.90 (m, 1H), 5.71 – 5.52 (m, 1H), 5.41 – 4.97 (m, 4H), 4.20 (dd, *J* = 17.5, 8.1 Hz, 1H), 3.44 (dd, *J* = 17.5, 4.5 Hz, 1H), 2.85 – 2.59 (m, 2H), 2.25 (dd, *J* = 13.5, 7.4 Hz, 2H), 1.90 (d, *J* = 16.4 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 166.6 (d, *J* = 17.0 Hz), 138.8 (d, *J* = 4.3 Hz), 128.7 (d, *J* = 2.5 Hz), 128.4 (d, *J* = 8.5 Hz), 127.9 (d, *J* = 8.2 Hz), 127.2 (d, *J* = 2.8 Hz), 126.9 (d, *J* = 4.5 Hz), 120.6 (d, *J* = 10.9 Hz), 120.3 (d, *J* = 11.1 Hz), 119.0, 113.1, 43.7 (d, *J* = 56.8 Hz), 39.2, 31.3 (d, *J* = 58.8 Hz), 30.9 (d, *J* = 62.7 Hz), 19.2 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.6 ppm; **IR** (neat): 2980, 1724, 1635, 1469, 1363, 1270, 1160, 918, 844, 744, 617 cm^{-1}; **HRMS** (ESI): $C_{20}H_{24}NO_2P$ $[M+H]^+$ calcd: 342.1617, found: 342.1610.



4b

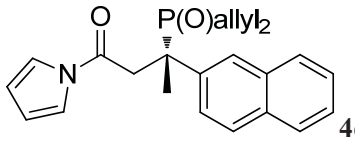
Colorless oil; 91% yield; 96% *ee* determined by HPLC on a Chiralpak As column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 14.3 min, t_{major} = 27.8 min); $[\alpha]^{rt}_{D}$ = -71.8 (*c* = 1.02, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.41 (dd, *J* = 8.8, 2.1 Hz, 2H), 7.27 (s, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.26 (t, *J* = 2.1 Hz, 2H), 6.11 – 5.90 (m, 1H), 5.74 – 5.55 (m, 1H), 5.41 – 4.99 (m, 4H), 4.14 (dd, *J* = 17.4, 8.2 Hz, 1H), 3.79 (s, 3H), 3.41 (dd, *J* = 17.4, 4.4 Hz, 1H), 2.86 – 2.57 (m, 2H), 2.25 (dd, *J* = 13.6, 7.5 Hz, 2H), 1.87 (d, *J* = 16.5 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 166.7 (d, *J* = 17.2 Hz), 158.5 (d, *J* = 2.7 Hz), 130.6 (d, *J* = 4.5 Hz), 128.5 (d, *J* = 8.4 Hz), 128.1 (d, *J* = 4.6 Hz), 128.0 (d, *J* = 7.1 Hz), 120.5 (d, *J* = 10.8 Hz), 120.2 (d, *J* = 11.0 Hz), 119.0, 114.0 (d, *J* = 2.4 Hz), 113.1, 55.1, 43.1 (d, *J* = 58.0 Hz), 39.1, 31.3 (d, *J* = 58.0 Hz), 30.9 (d, *J* = 62.9 Hz), 19.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.0 ppm; **IR** (neat): 2932, 1723, 1610, 1513, 1468, 1269, 1256, 920, 850, 738, 615 cm^{-1}; **HRMS** (ESI): $C_{21}H_{26}NO_3P$ $[M+H]^+$ calcd: 372.1723, found: 372.1731.



4c

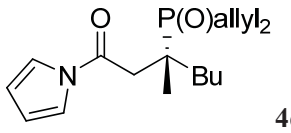
Colorless oil; 96% yield; 94% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow

rate = 1.0 mL/min, t_{minor} = 14.6 min, t_{major} = 15.6 min); $[\alpha]^{rt}_D$ = -63.4 (*c* = 1.10, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.45 (dd, *J* = 8.8, 2.1 Hz, 2H), 7.34 (d, *J* = 8.6 Hz, 2H), 7.25 (t, *J* = 2.3 Hz, 2H), 6.26 (t, *J* = 2.4 Hz, 2H), 6.10 – 5.92 (m, 1H), 5.71 – 5.53 (m, 1H), 5.40 – 4.96 (m, 4H), 4.16 (dd, *J* = 17.6, 8.0 Hz, 1H), 3.42 (dd, *J* = 17.6, 4.3 Hz, 1H), 2.84 – 2.66 (m, 2H), 2.30 – 2.15 (m, 2H), 1.86 (d, *J* = 16.4 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 166.4 (d, *J* = 16.8 Hz), 137.5 (d, *J* = 4.5 Hz), 133.2 (d, *J* = 3.4 Hz), 128.7 (d, *J* = 2.5 Hz), 128.4 (d, *J* = 4.5 Hz), 128.2 (d, *J* = 8.5 Hz), 127.5 (d, *J* = 8.3 Hz), 120.7 (d, *J* = 10.9 Hz), 120.5 (d, *J* = 11.1 Hz), 118.9, 113.3, 43.4 (d, *J* = 56.5 Hz), 39.2, 31.3 (d, *J* = 58.8 Hz), 30.9 (d, *J* = 62.8 Hz), 19.1 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +49.1 ppm; **IR** (neat): 2981, 1723, 1635, 1469, 1361, 1272, 1162, 1121, 921, 851, 741 cm^{-1}; **HRMS** (ESI): $C_{20}H_{23}ClNO_2P$ $[M+H]^+$ calcd: 376.1228, found: 376.1235.



4d

Colorless oil; 93% yield; 94% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 21.2 min, t_{major} = 15.9 min); $[\alpha]^{rt}_D$ = -81.0 (*c* = 1.12, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.91 (s, 1H), 7.88 – 7.77 (m, 3H), 7.68 (d, *J* = 8.7 Hz, 1H), 7.54 – 7.41 (m, 2H), 7.30 (s, 2H), 6.25 (t, *J* = 2.4 Hz, 2H), 6.12 – 5.92 (m, 1H), 5.72 – 5.55 (m, 1H), 5.39 – 4.90 (m, 4H), 4.33 (dd, *J* = 17.6, 8.1 Hz, 1H), 3.57 (dd, *J* = 17.6, 4.4 Hz, 1H), 2.93 – 2.60 (m, 2H), 2.26 (dd, *J* = 13.7, 7.5 Hz, 2H), 2.02 (d, *J* = 16.4 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 166.5 (d, *J* = 17.1 Hz), 136.3 (d, *J* = 4.9 Hz), 133.1 (d, *J* = 2.6 Hz), 132.2 (d, *J* = 2.1 Hz), 128.4 (d, *J* = 2.0 Hz), 128.1[9] (d, *J* = 8.3 Hz), 128.1[7], 127.7 (d, *J* = 8.6 Hz), 127.5 (d, *J* = 1.1 Hz), 126.3, 126.2, 126.1[6] (d, *J* = 4.5 Hz), 124.8 (d, *J* = 3.4 Hz), 120.8 (d, *J* = 11.0 Hz), 120.5 (d, *J* = 10.9 Hz), 119.0, 113.2, 44.0 (d, *J* = 56.7 Hz), 39.3, 31.2 (d, *J* = 58.7 Hz), 30.9 (d, *J* = 62.7 Hz), 19.3 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +50.3 ppm; **IR** (neat): 2980, 1723, 1634, 1468, 1363, 1272, 1162, 1121, 919, 736, 610 cm^{-1}; **HRMS** (ESI): $C_{24}H_{26}NO_2P$ $[M+H]^+$ calcd: 392.1774, found: 392.1763.



4e

Colorless oil; 90% yield; 98% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 7.9min, t_{major} = 6.7 min); $[\alpha]^{rt}_D$ = +5.8 (*c* = 1.03, $CHCl_3$); From Z substrate, 95% yield; -97% *ee*; $[\alpha]^{rt}_D$ = -6.4 (*c* = 1.09, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 7.36 (s, 2H), 6.29 (t, *J* = 2.4 Hz, 2H), 6.04 – 5.85 (m, 2H), 5.34 – 5.16 (m, 4H), 3.32 (dd, *J* = 16.3, 9.5 Hz, 1H), 3.08 (dd, *J* = 16.3, 12.4 Hz, 1H), 2.91 – 2.61 (m, 4H),

1.97 – 1.70 (m, 2H), 1.54 – 1.22 (m, 4H), 1.37 (d, J = 15.6 Hz, 3H), 0.89 (t, J = 7.1 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 168.1 (d, J = 11.2 Hz), 128.5 (d, J = 8.6 Hz), 128.4 (d, J = 8.5 Hz), 120.2[8] (d, J = 11.0 Hz), 120.2[5] (d, J = 10.9 Hz), 119.2, 113.3, 39.8 (d, J = 60.7 Hz), 37.0, 34.5, 31.8 (d, J = 58.9 Hz), 31.5 (d, J = 58.8 Hz), 26.2 (d, J = 5.7 Hz), 23.2, 20.7, 13.8 ppm; **^{31}P NMR** (121 MHz, $CDCl_3$): δ = +52.1 ppm; **IR** (neat): 2958, 1715, 1635, 1469, 1272, 1163, 1117, 1071, 921, 846, 745, 618 cm^{-1}; **HRMS** (ESI): $C_{18}H_{28}NO_2P$ $[M+H]^+$ calcd: 322.1930, found: 322.1937.

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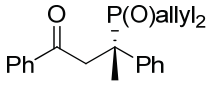
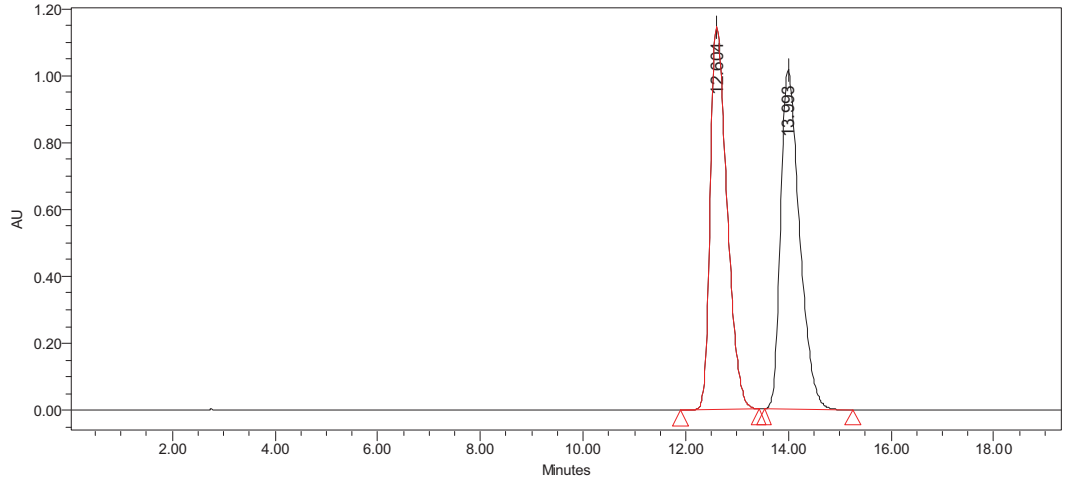
HPLC results

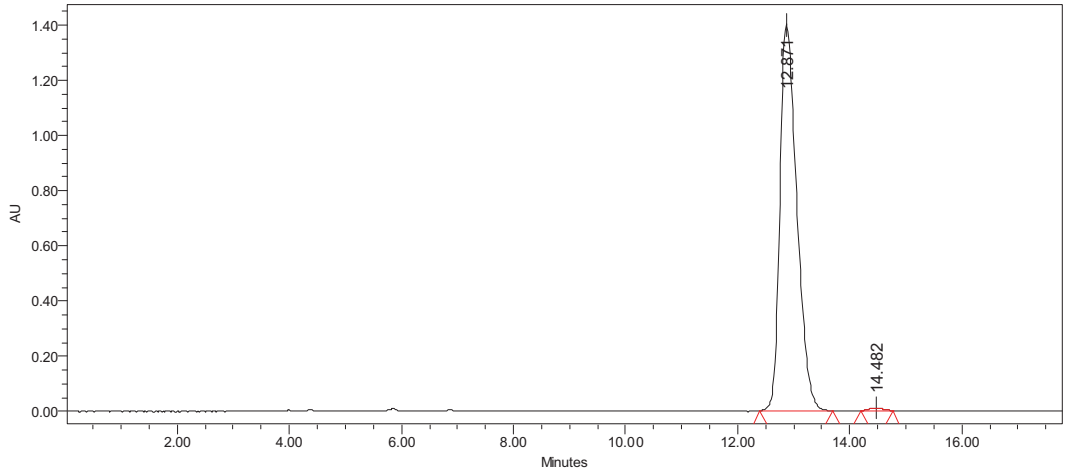


3a

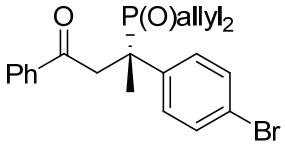
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		12.604	26021791	49.99	1143854
2		13.993	26028727	50.01	1015798

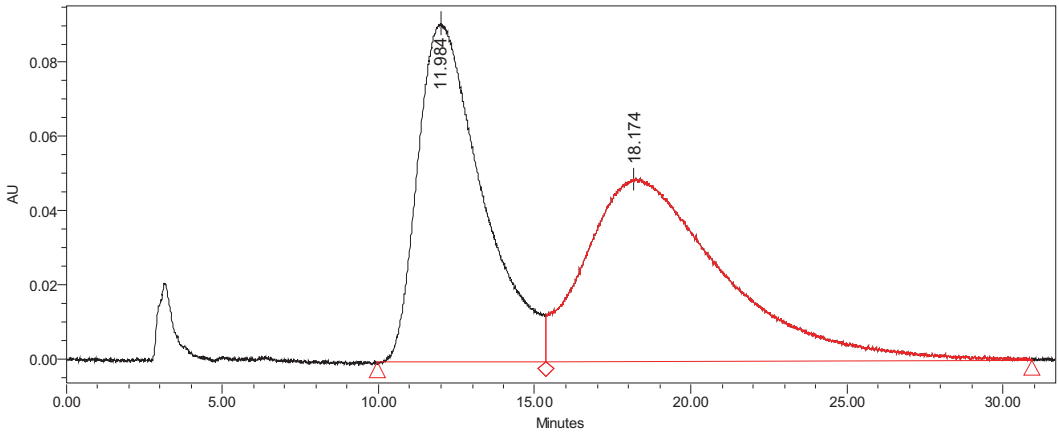


	Name	Retention Time	Area	% Area	Height
1		12.871	29491205	99.36	1401036
2		14.482	189248	0.64	10200

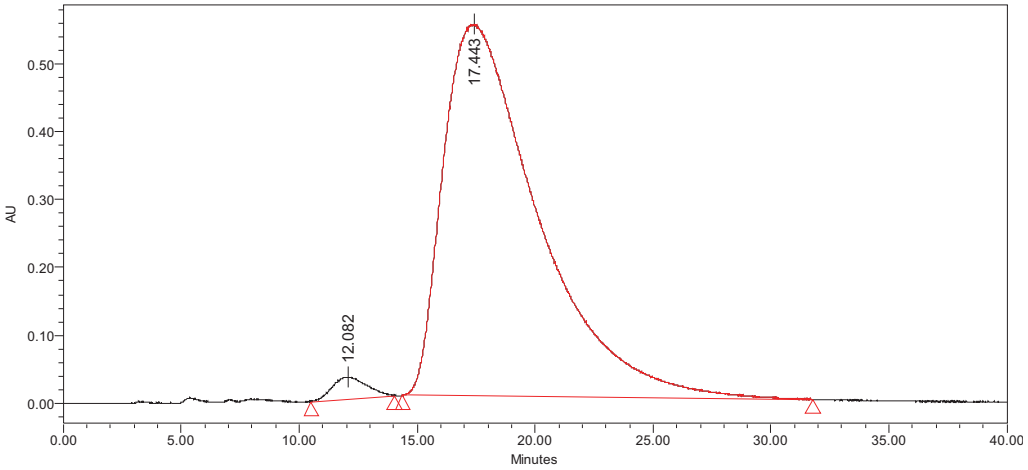


3b

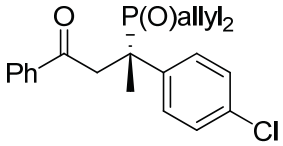
HPLC using an As (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	11.984	13074669	46.50	91251
2	18.174	15040359	53.50	49114

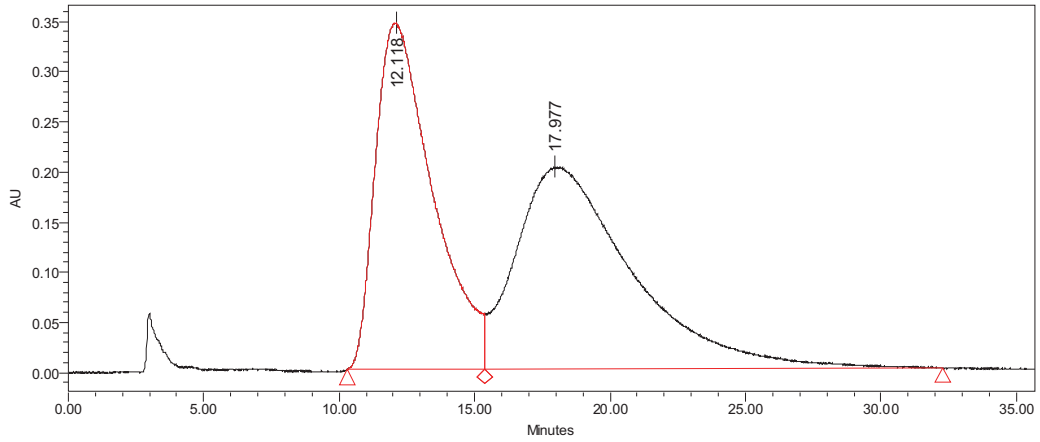


Name	Retention Time	Area	% Area	Height
1	12.082	3278198	2.09	32531
2	17.443	153504631	97.91	548075

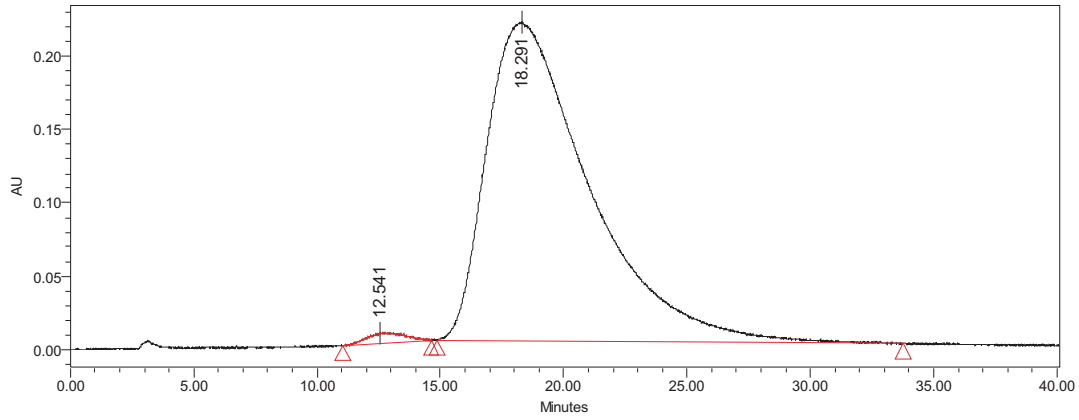


3c

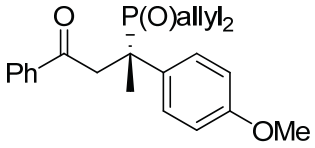
HPLC using an AS (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	12.118	51061177	45.97	345969
2	17.977	60015705	54.03	201624

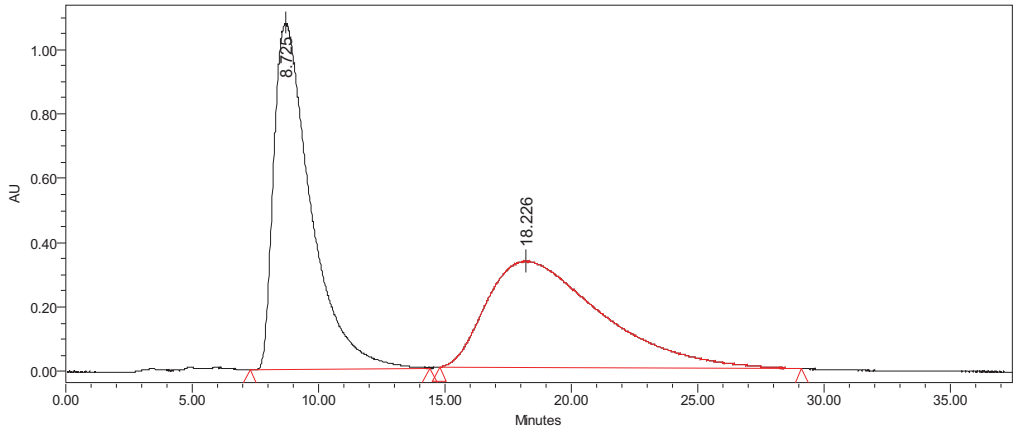


Name	Retention Time	Area	% Area	Height
1	12.541	712322	1.12	6874
2	18.291	62920285	98.88	217679

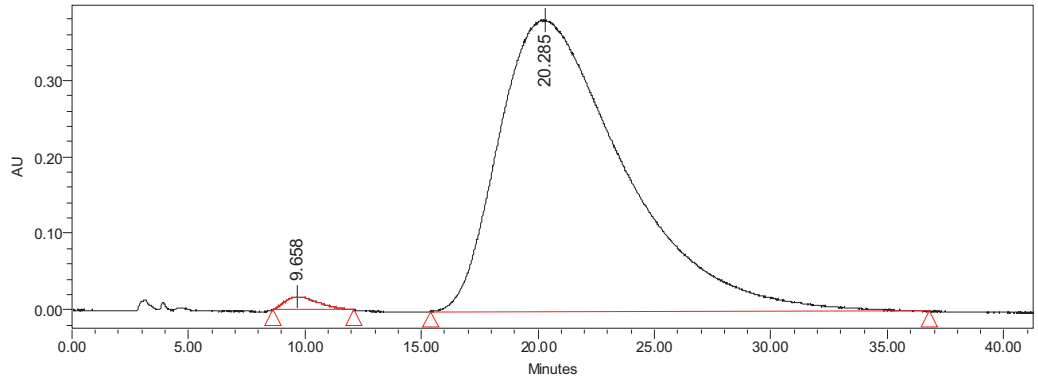


3d

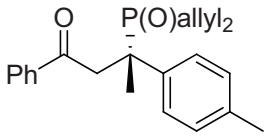
HPLC using an AS (*n*-Hexane/*i*PrOH=80/20, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		8.725	109285754	51.20	1076204
2		18.226	104173139	48.80	330616

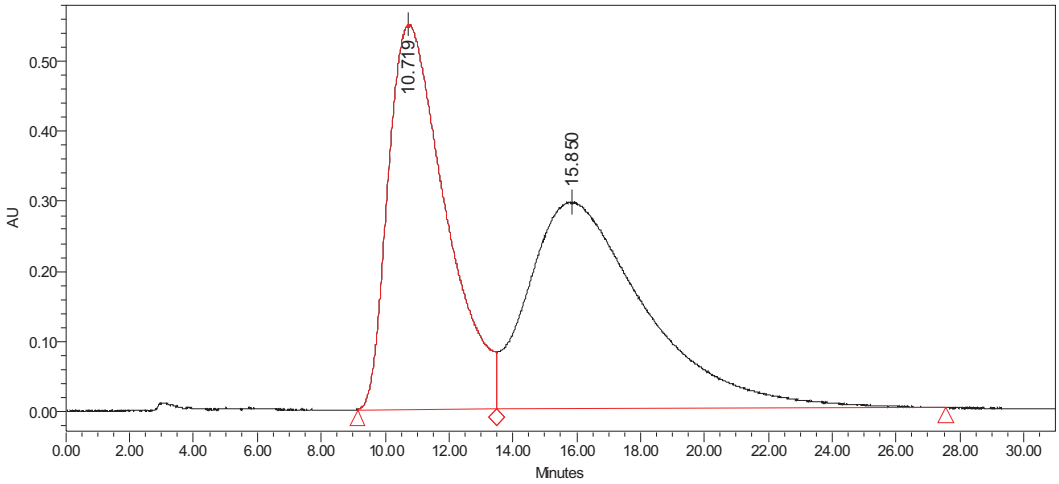


	Name	Retention Time	Area	% Area	Height
1		9.658	1699976	1.18	17277
2		20.285	142266606	98.82	381393

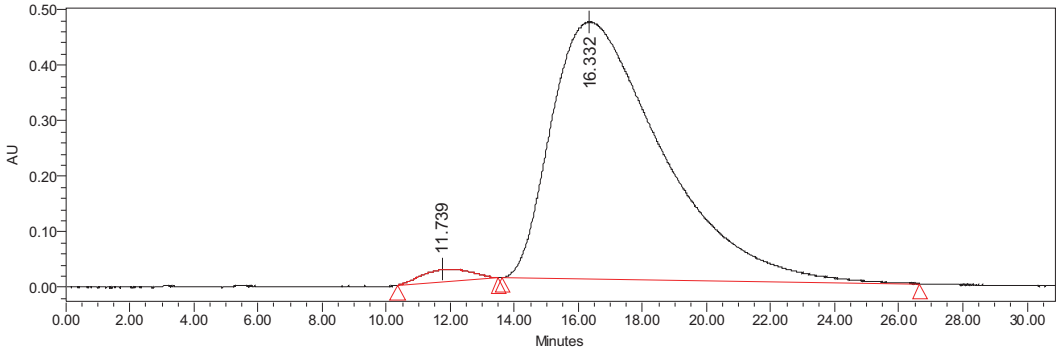


3e

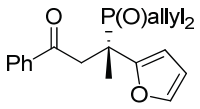
HPLC using an AS (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		10.719	68967113	47.72	550608
2		15.850	75566915	52.28	295827

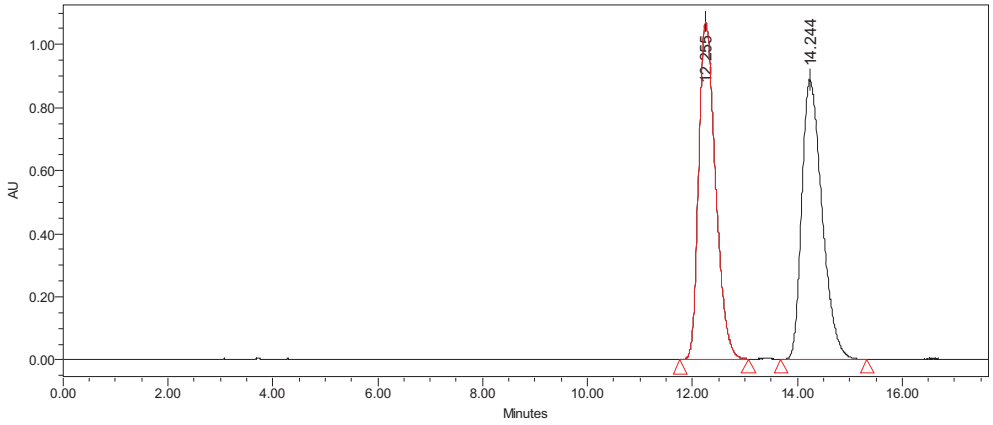


	Name	Retention Time	Area	% Area	Height
1		11.739	2347983	2.08	22572
2		16.332	110793648	97.92	463180

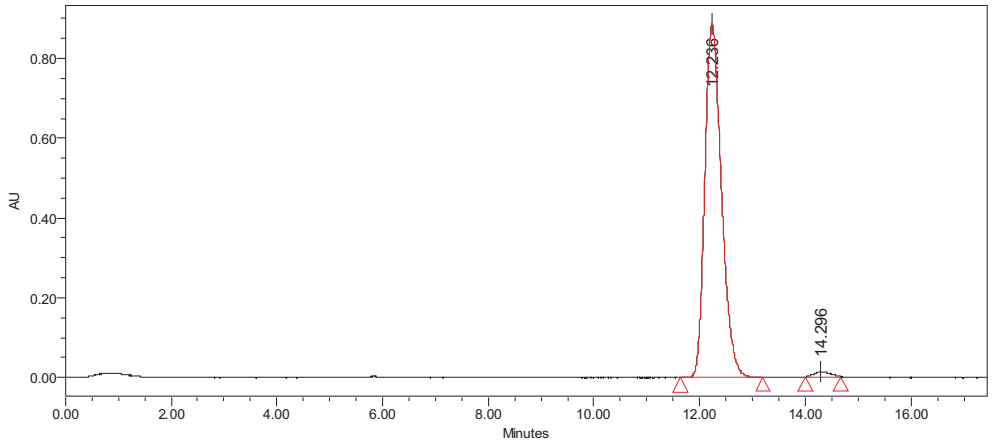


3f

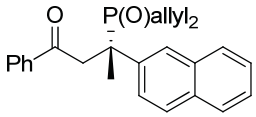
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		12.255	23736844	49.95	1070205
2		14.244	23785498	50.05	886362

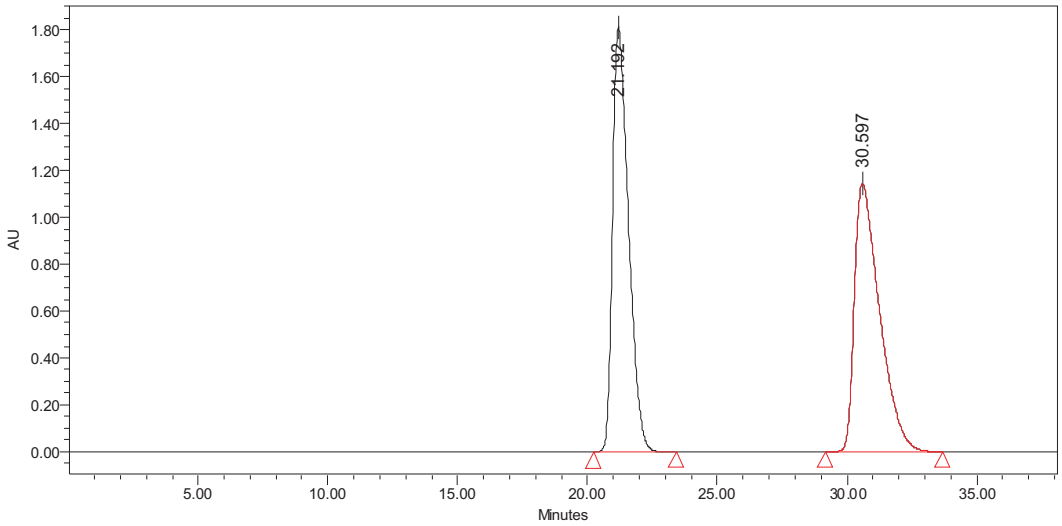


	Name	Retention Time	Area	% Area	Height
1		12.236	19356854	98.75	888682
2		14.296	245481	1.25	11726

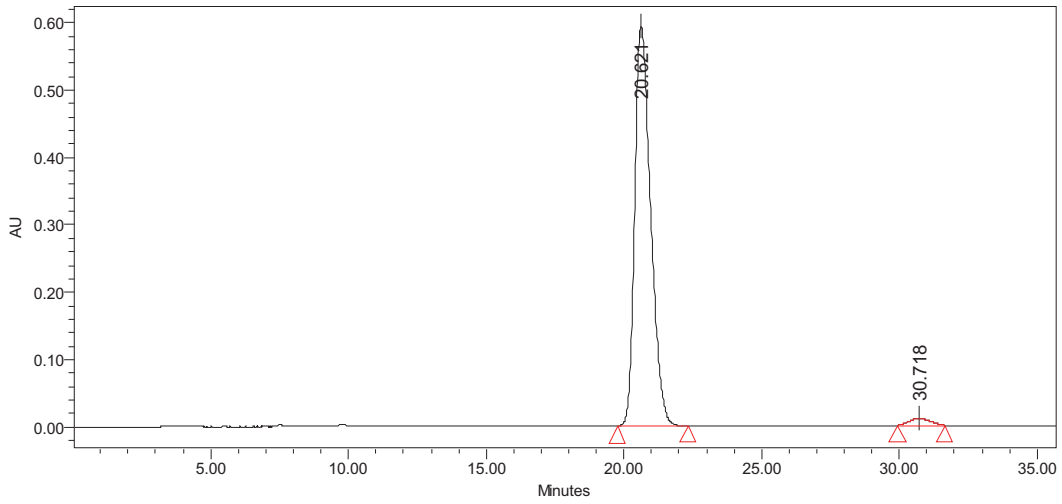


3g

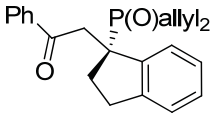
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		21.192	77426809	50.16	1810261
2		30.597	76934418	49.84	1142390

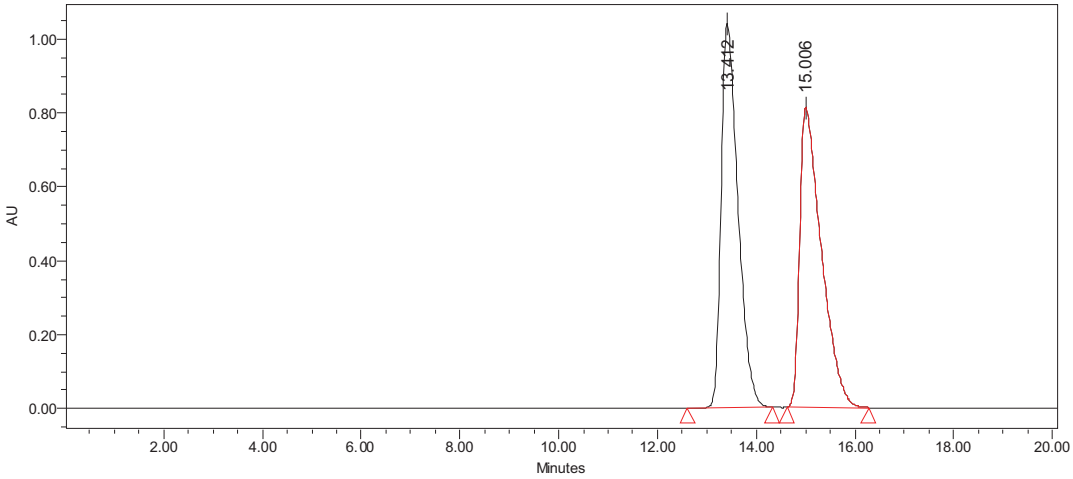


	Name	Retention Time	Area	% Area	Height
1		20.621	23903013	97.79	593108
2		30.718	539319	2.21	10311

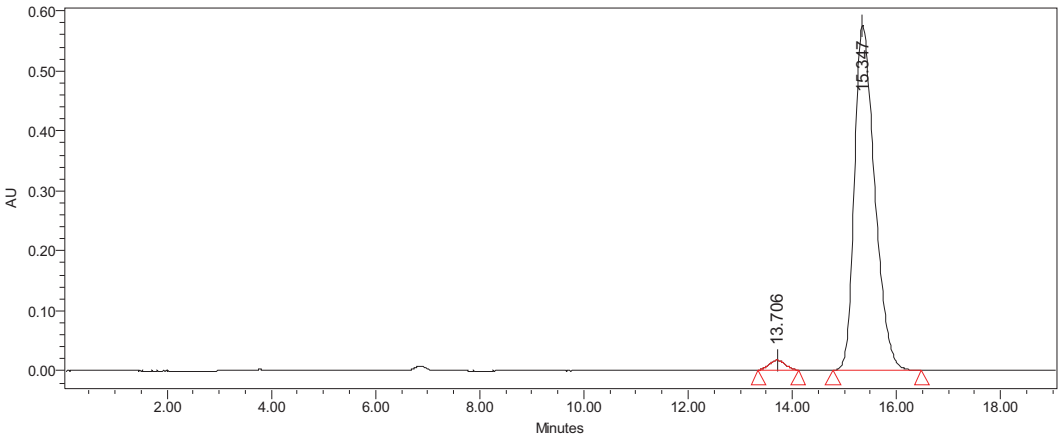


3h

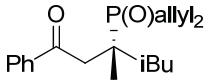
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		13.412	24721071	50.06	1040508
2		15.006	24666105	49.94	809860

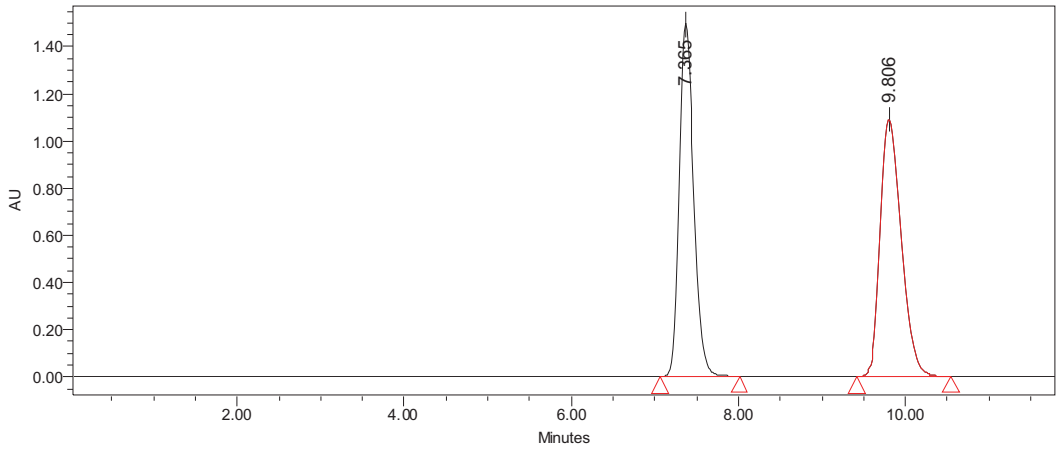


	Name	Retention Time	Area	% Area	Height
1		13.706	351175	2.20	16383
2		15.347	15598847	97.80	575184

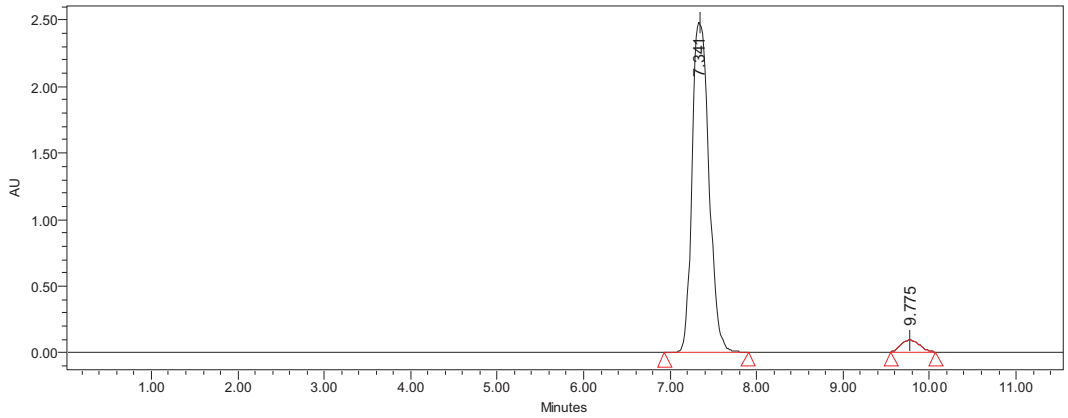


3i

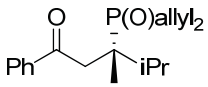
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		7.365	18606356	49.66	1495558
2		9.806	18860132	50.34	1092304

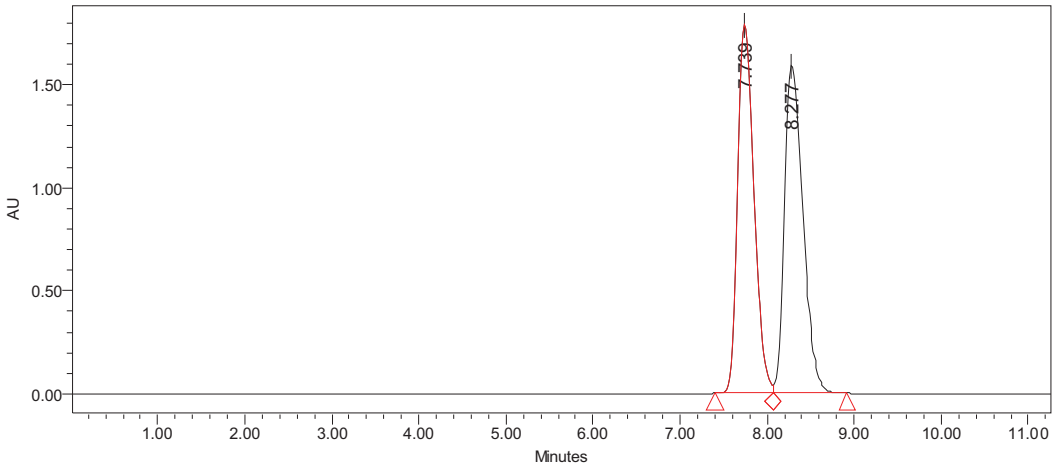


	Name	Retention Time	Area	% Area	Height
1		7.341	33804548	96.33	2473294
2		9.775	1287948	3.67	88857

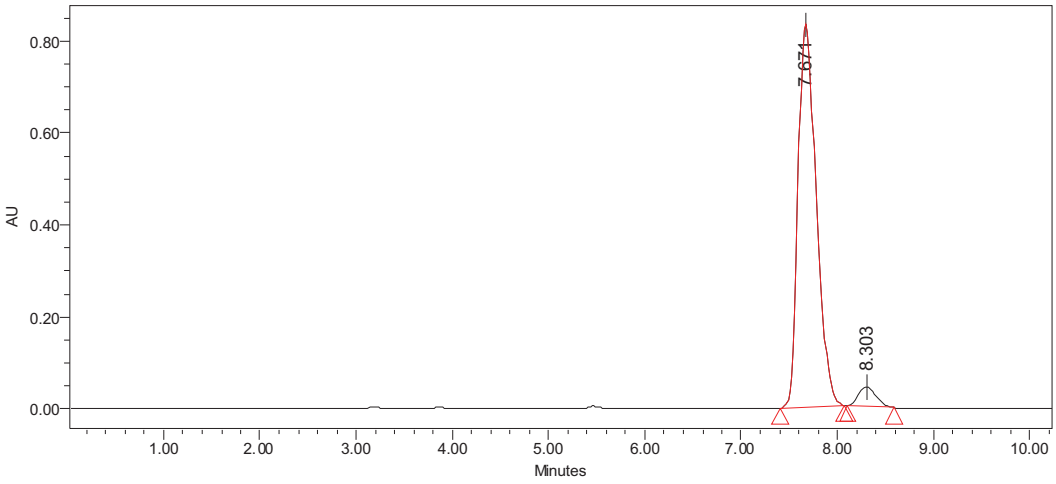


3j

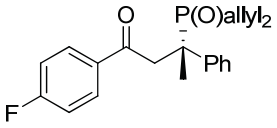
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		7.739	23282759	49.55	1791218
2		8.277	23708242	50.45	1593194

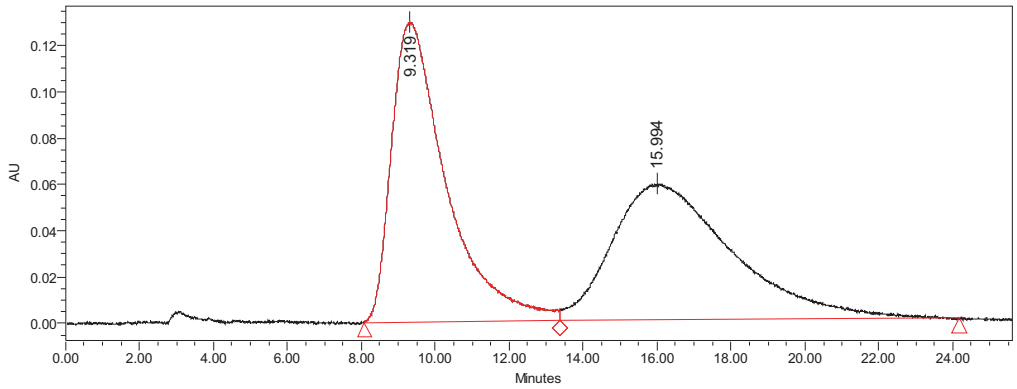


	Name	Retention Time	Area	% Area	Height
1		7.671	10950553	95.24	832106
2		8.303	547817	4.76	42310

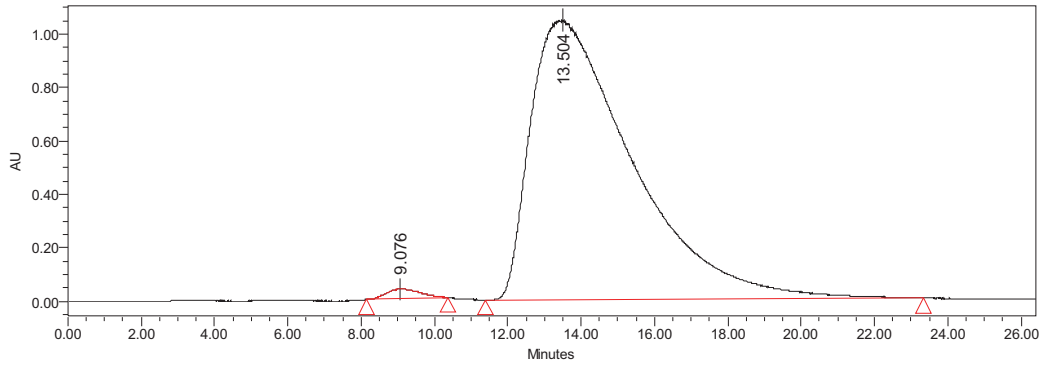


3k

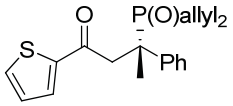
HPLC using an AS (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	9.319	13351926	50.11	129778
2	15.994	13294194	49.89	58833

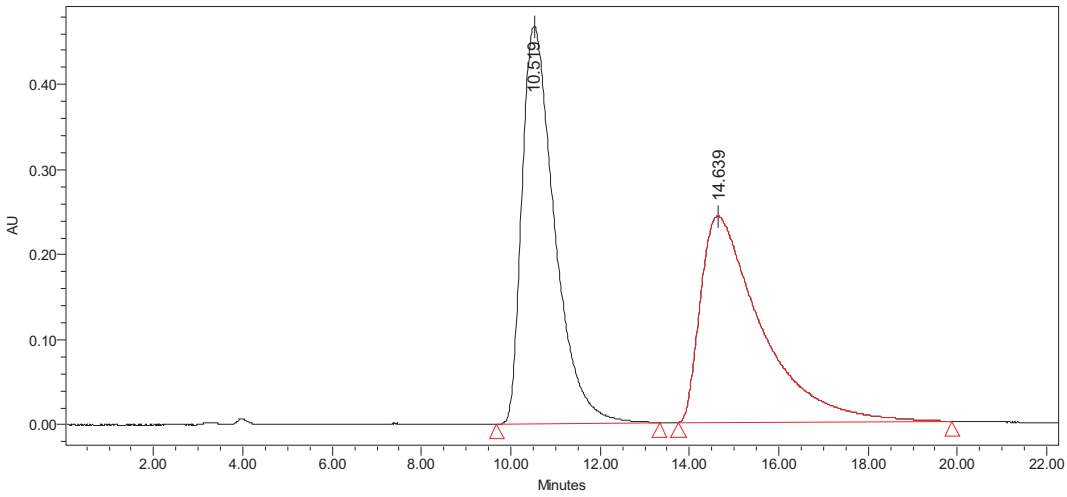


Name	Retention Time	Area	% Area	Height
1	9.076	2404455	1.18	37479
2	13.504	201726555	98.82	1047633

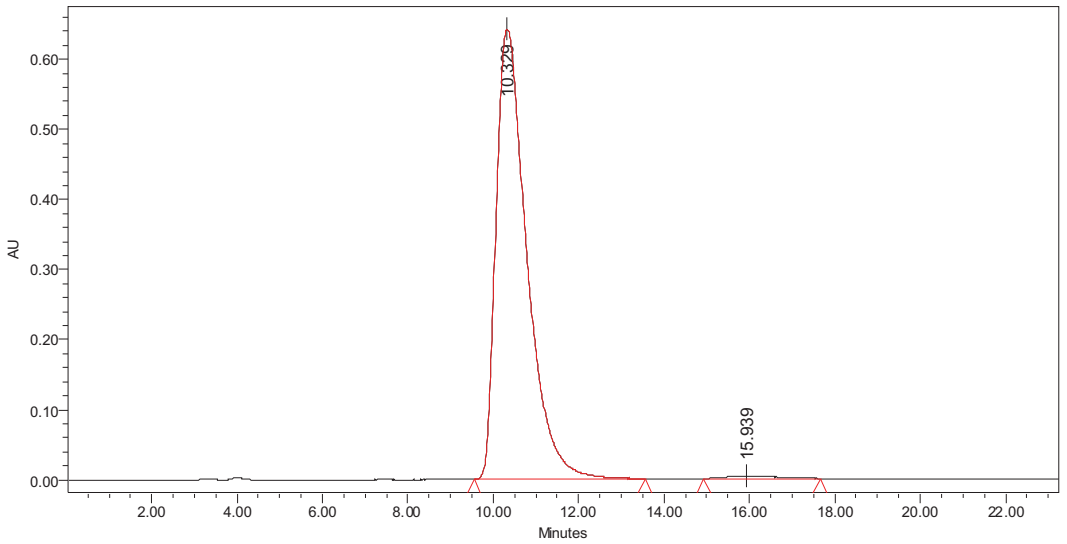


3l

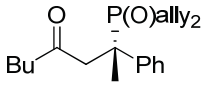
HPLC using an OJ-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	10.519	23757642	50.93	467140
2	14.639	22891545	49.07	242354

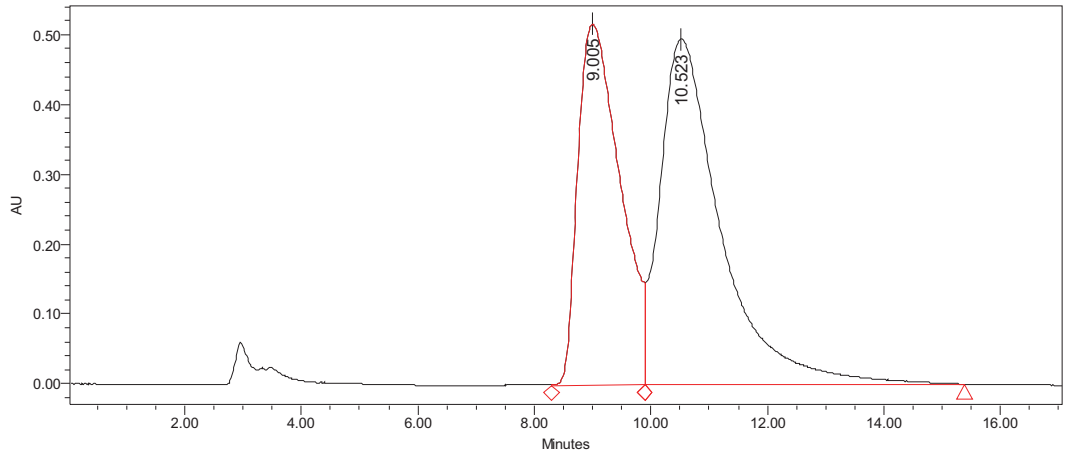


Name	Retention Time	Area	% Area	Height
1	10.329	32118814	99.06	641873
2	15.939	303279	0.94	3572

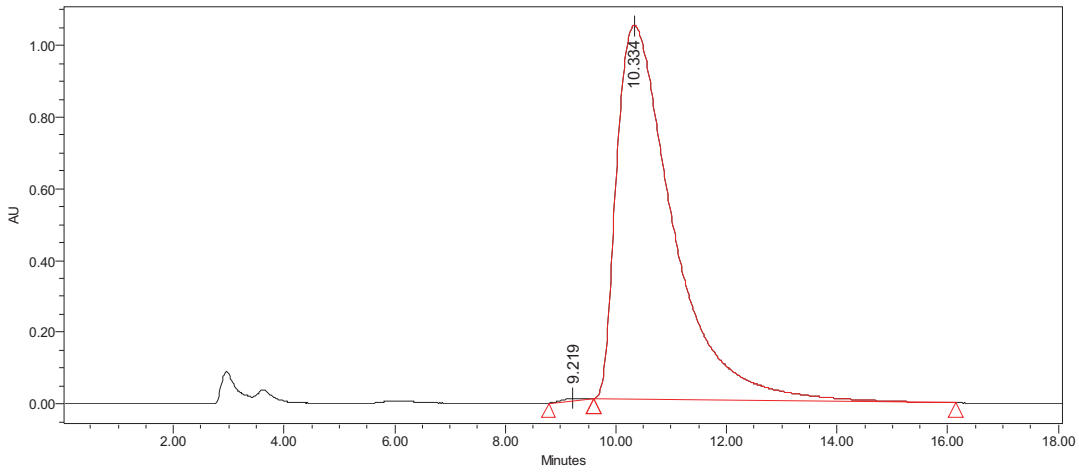


3m

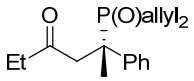
HPLC using an AS (*n*-Hexane/*i*PrOH=95/5, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		9.005	26063395	42.02	516900
2		10.523	35963883	57.98	495495

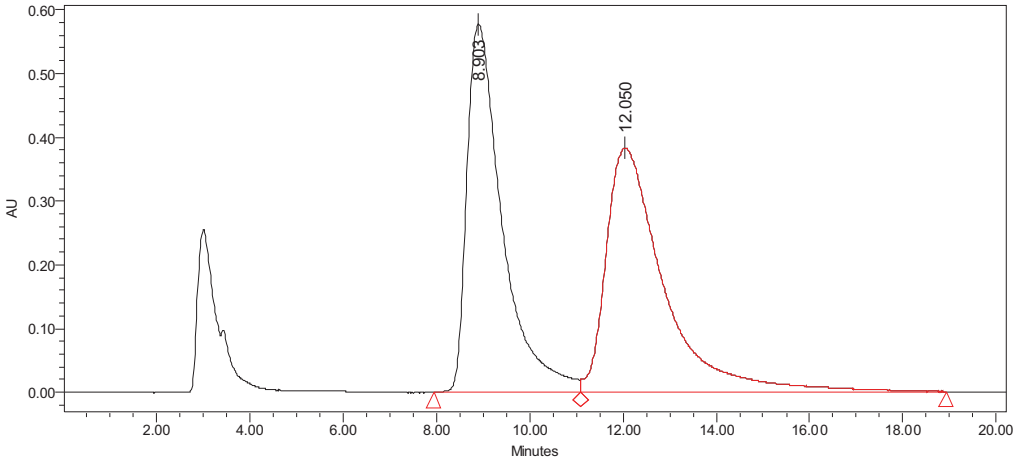


	Name	Retention Time	Area	% Area	Height
1		9.219	184167	0.25	7287
2		10.334	74277683	99.75	1043070

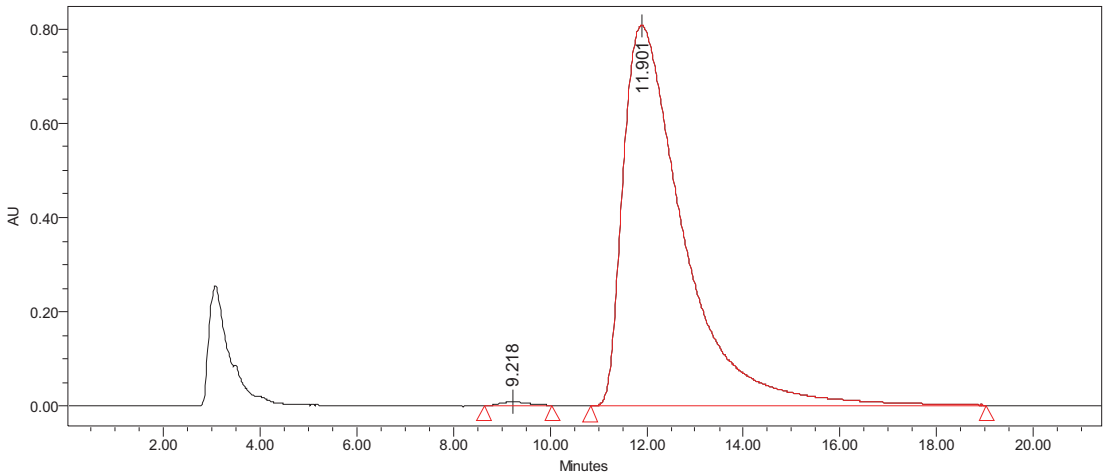


3n

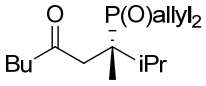
HPLC using an AS (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		8.903	30462978	48.04	577241
2		12.050	32952935	51.96	382925

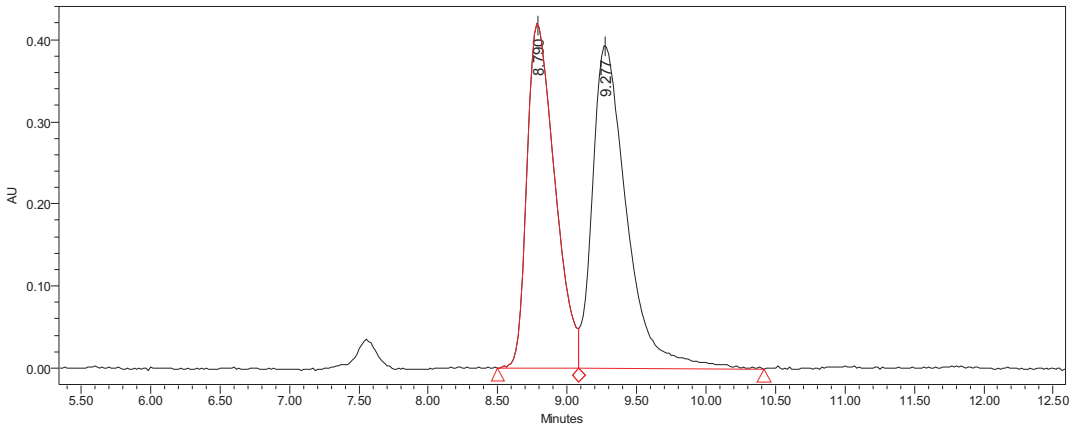


	Name	Retention Time	Area	% Area	Height
1		9.218	327726	0.47	8212
2		11.901	70026919	99.53	806822

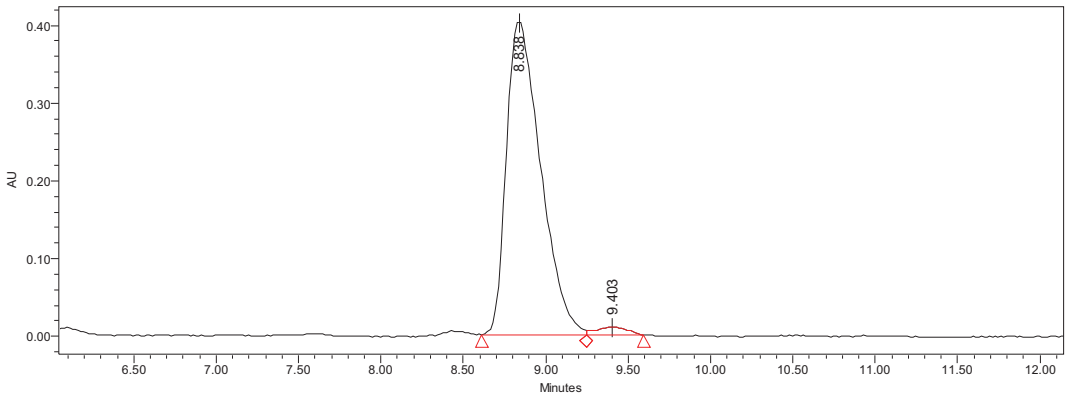


3o

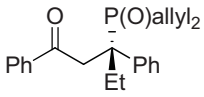
HPLC using an AD-H (*n*-Hexane/*i*PrOH=95/5, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	8.790	5859818	46.36	420583
2	9.277	6779868	53.64	394429

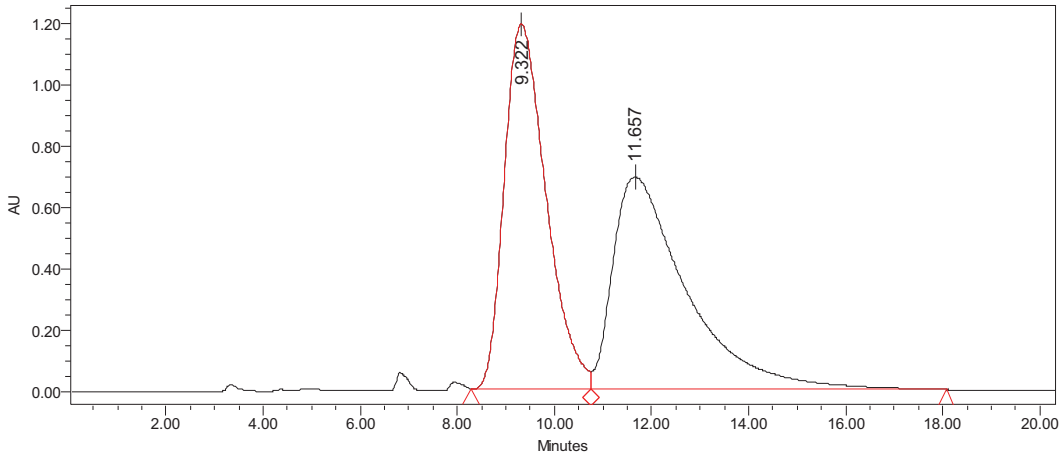


Name	Retention Time	Area	% Area	Height
1	8.838	5794150	97.85	402671
2	9.403	127496	2.15	9608

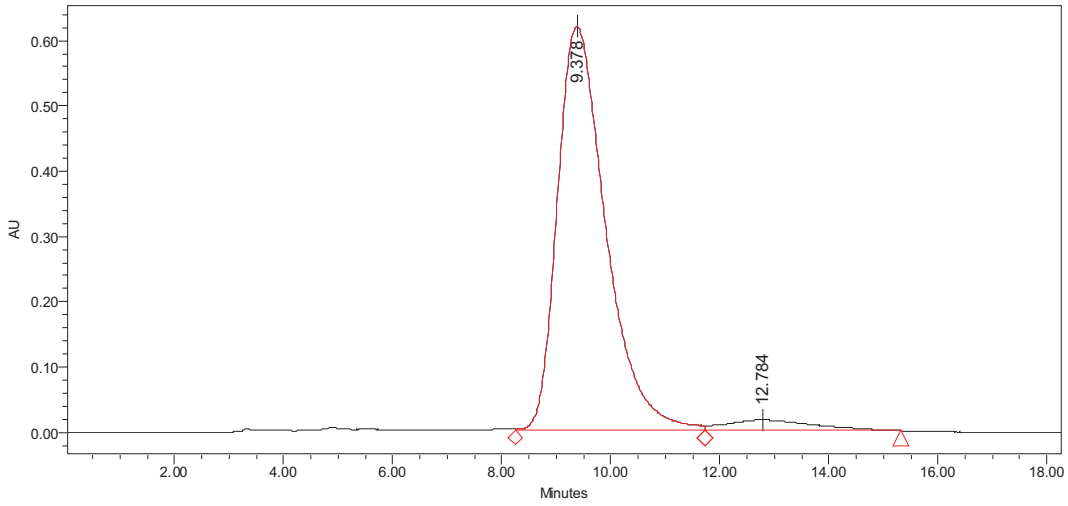


3p

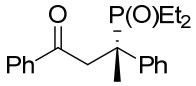
HPLC using an OJ-H (*n*-Hexane/*i*PrOH=95/5, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		9.322	71192167	49.31	1188509
2		11.657	73183031	50.69	691285

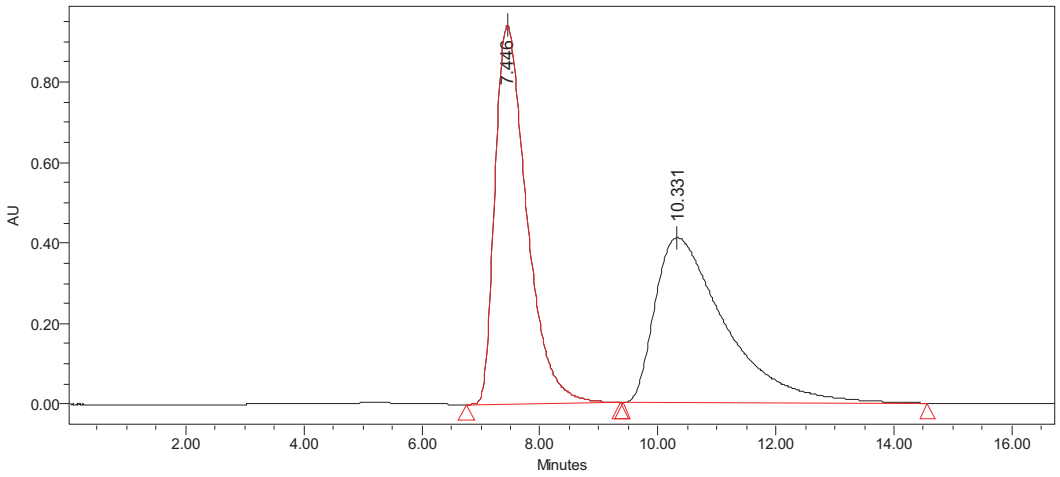


	Name	Retention Time	Area	% Area	Height
1		9.378	38107992	95.54	618334
2		12.784	1779740	4.46	15887

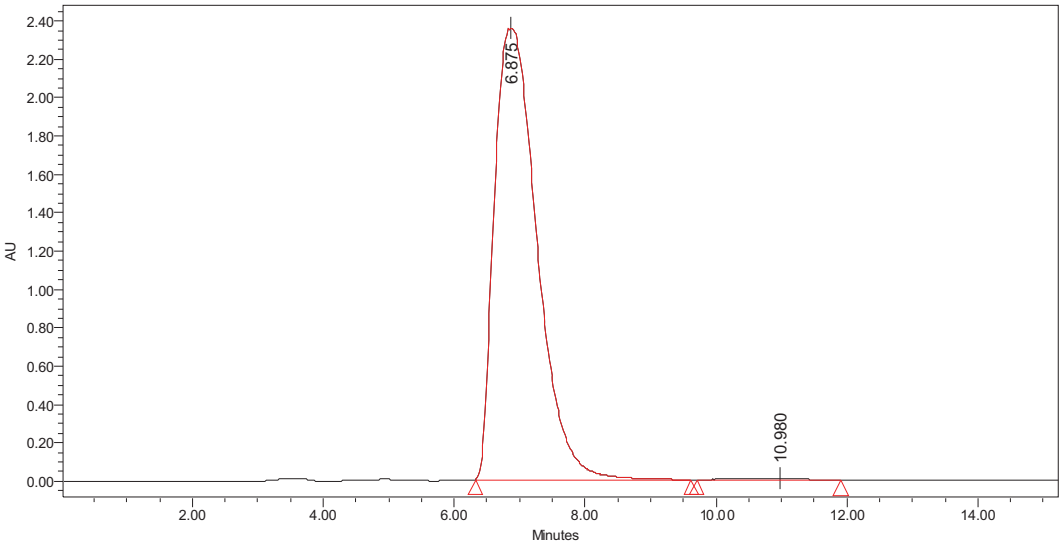


3q

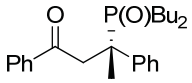
HPLC using an OJ-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	7.446	35104961	50.94	939282
2	10.331	33808714	49.06	409327

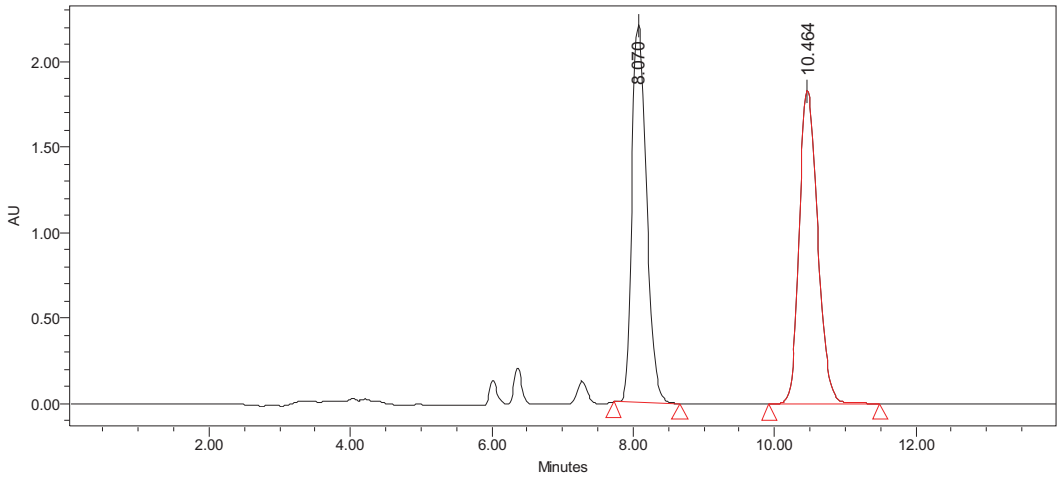


Name	Retention Time	Area	% Area	Height
1	6.875	106147372	99.54	2354015
2	10.980	486335	0.46	8402

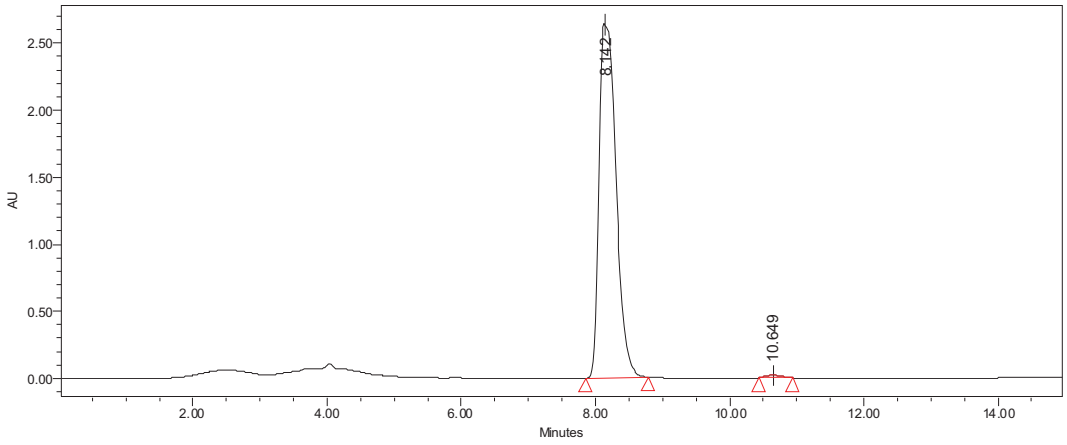


3r

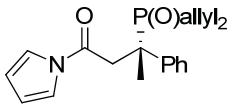
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		8.070	31777093	48.59	2204004
2		10.464	33623088	51.41	1829774

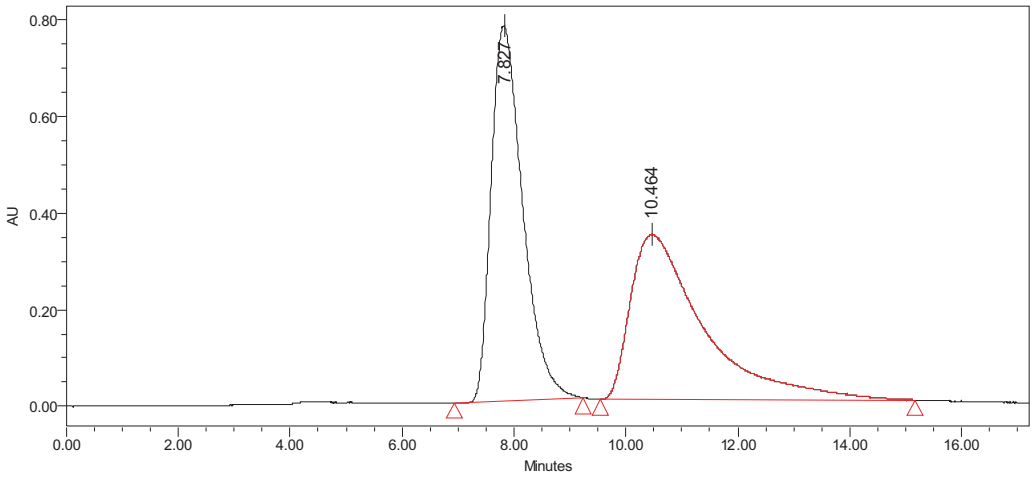


	Name	Retention Time	Area	% Area	Height
1		8.142	46960891	99.31	2645477
2		10.649	327421	0.69	20295

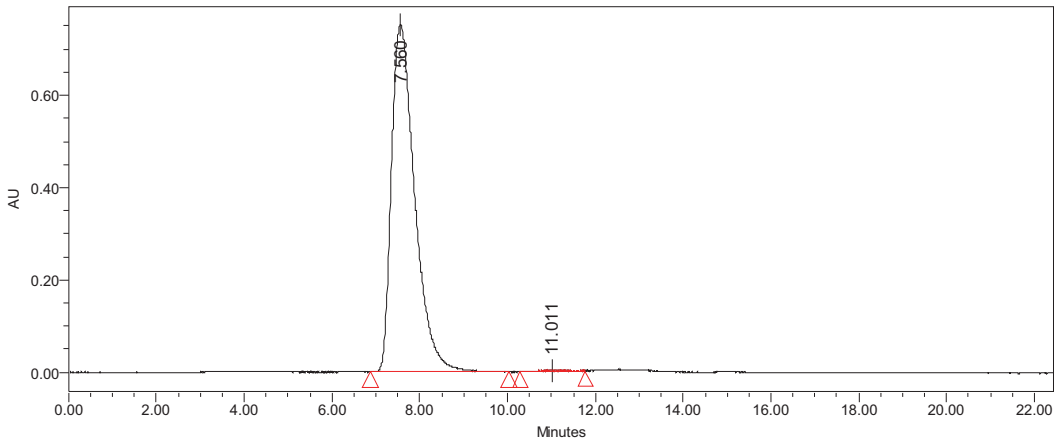


4a

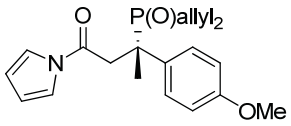
HPLC using an OJ-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		7.827	30533051	49.66	778118
2		10.464	30952613	50.34	342295

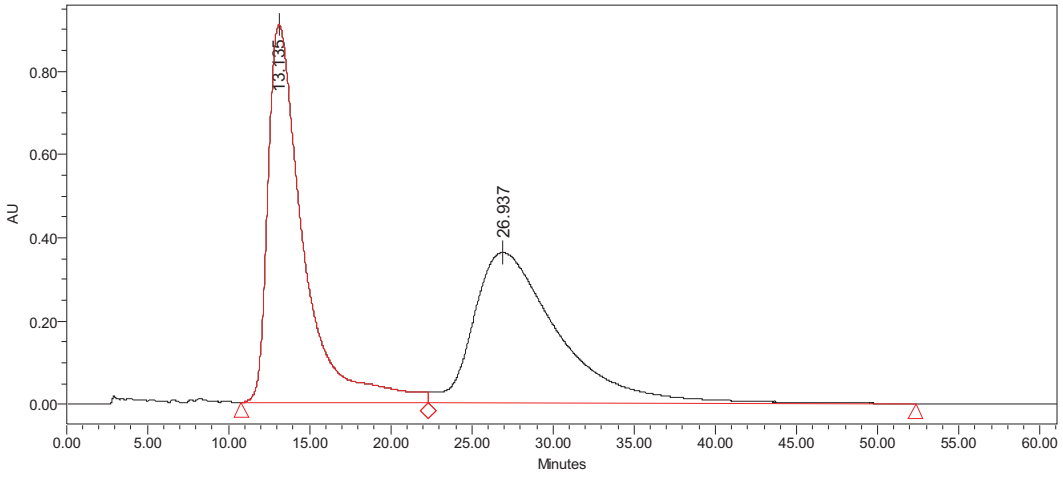


	Name	Retention Time	Area	% Area	Height
1		7.560	27840568	99.78	753550
2		11.011	61746	0.22	1906

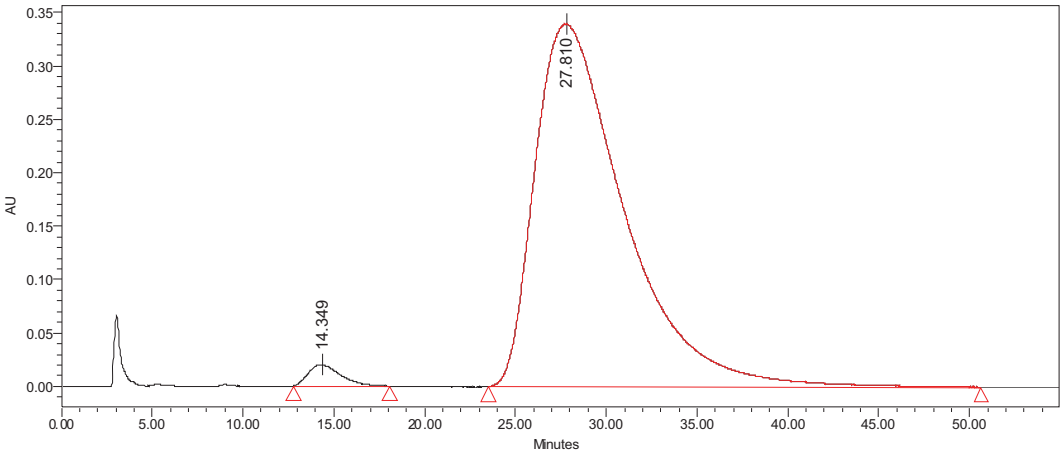


4b

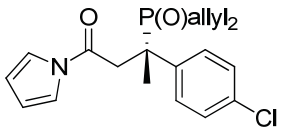
HPLC using an As (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	13.135	137181310	51.17	908757
2	26.937	130882962	48.83	361551

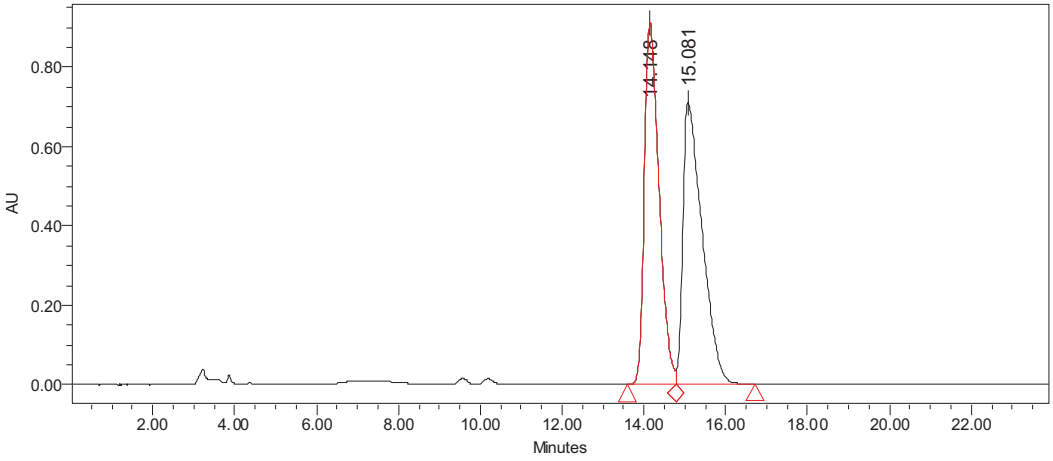


Name	Retention Time	Area	% Area	Height
1	14.349	2484268	2.09	20012
2	27.810	116514951	97.91	339537

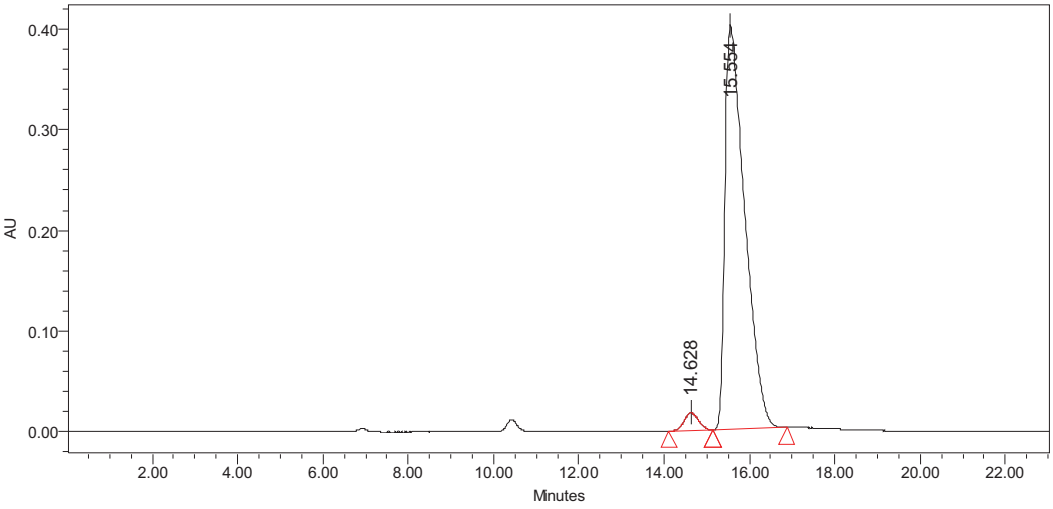


4c

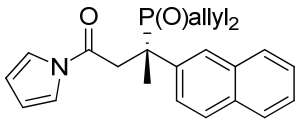
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		14.148	22689977	49.41	912514
2		15.081	23227245	50.59	709267

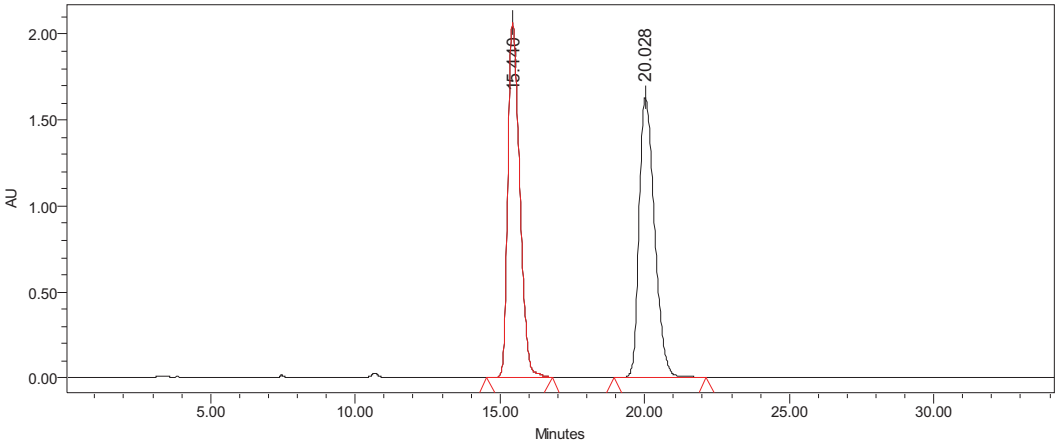


	Name	Retention Time	Area	% Area	Height
1		14.628	427375	3.12	18078
2		15.554	13249420	96.88	402993

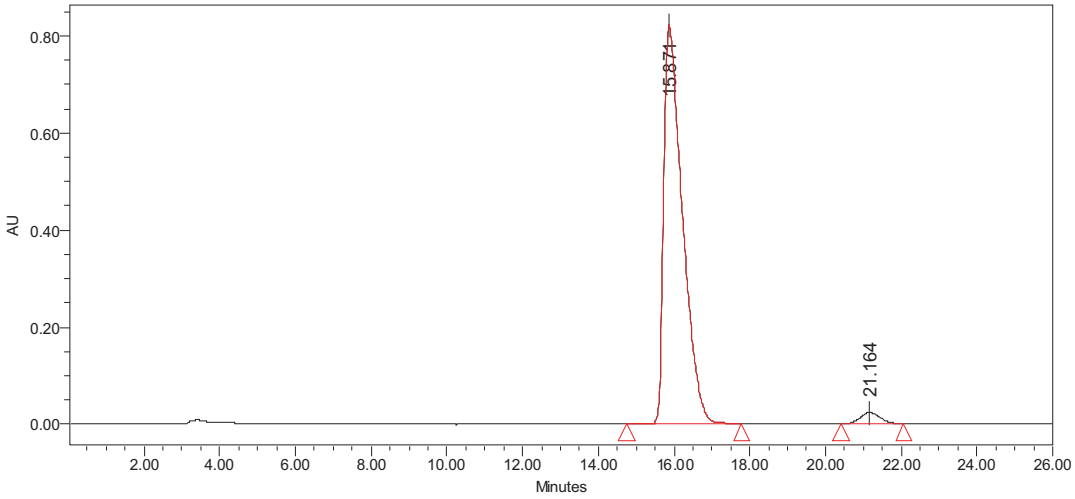


4d

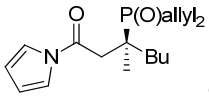
HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	15.440	59039093	49.32	2061191
2	20.028	60671372	50.68	1631041



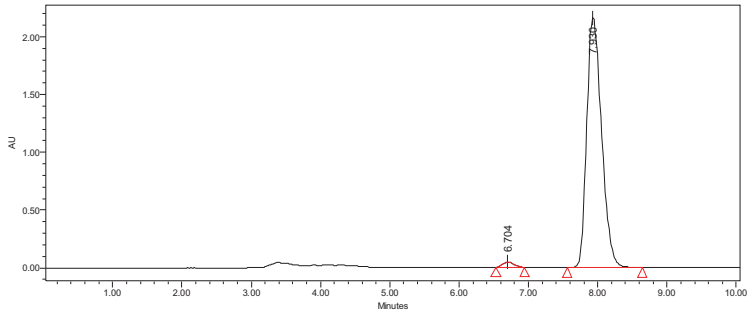
Name	Retention Time	Area	% Area	Height
1	15.871	28014889	96.92	822476
2	21.164	891133	3.08	23436



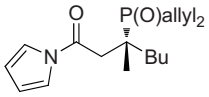
4e

HPLC using an AD-H (*n*-Hexane/*i*PrOH=90/10, flow rate 1.0 mL/min)

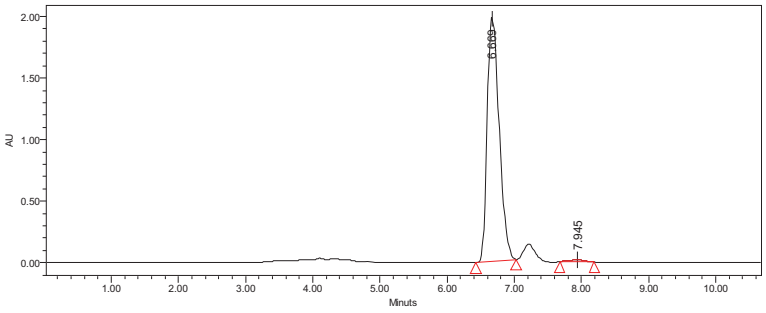
From *Z*-**5e**:



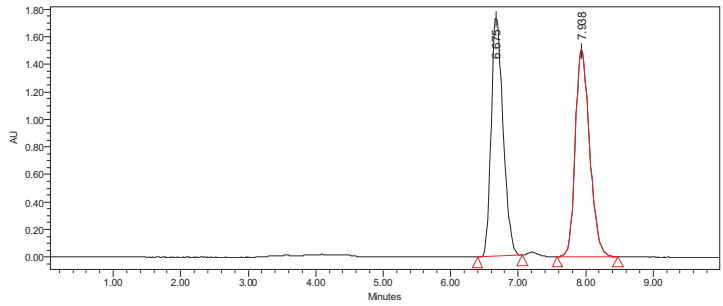
Name	Retention Time	Area	% Area	Height
1	6.704	536497	1.64	47597
2	7.930	32195468	98.36	2160587



From *E*-**5e**:

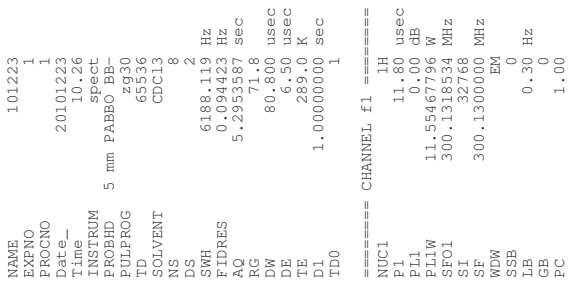


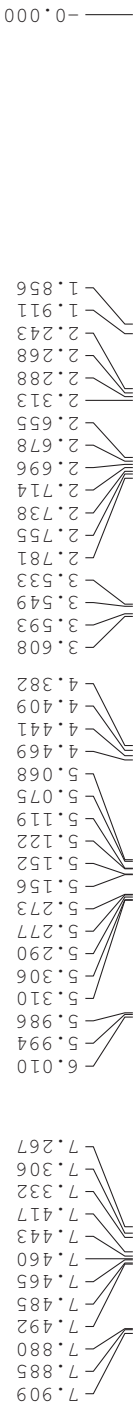
Name	Retention Time	Area	% Area	Height
1	6.669	24715906	98.90	1987575
2	7.945	274047	1.10	16645

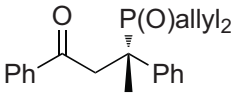


Name	Retention Time	Area	% Area	Height
1	6.675	21244250	48.96	1732522
2	7.938	22147292	51.04	1495482

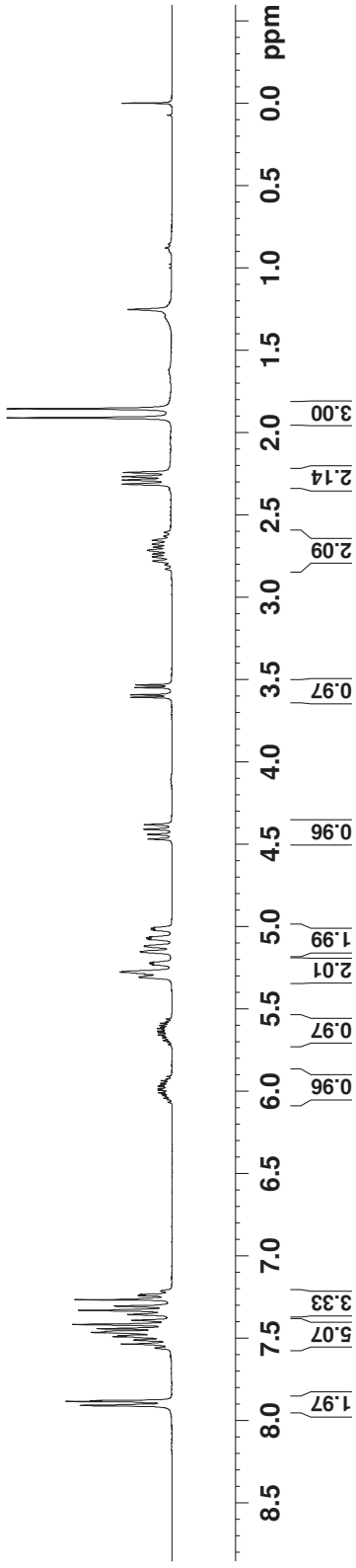
Copies of NMR spectra

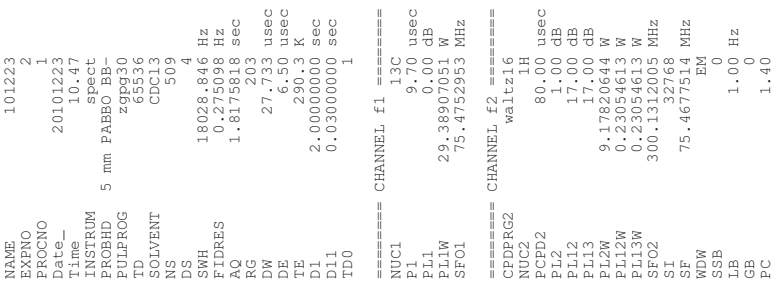




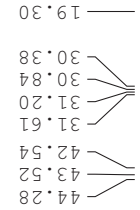


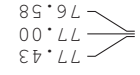
3a

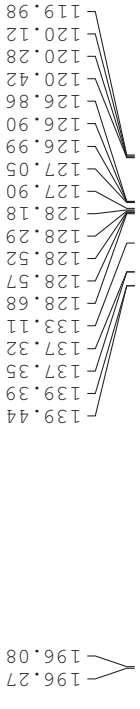


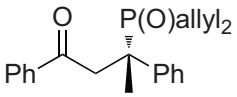


```
NAME                 101223
EXPNO                     2
PROCNO                    1
Date_              20101223
Time                 10.47
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        509
DS                          4
SWH                18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175818 sec
RG                          203
DW                     27.733 usec
DE                      6.50 usec
TE                     290.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.70 usec
PL1                       0.00 dB
PL1W                29.38907051 W
SFO1                75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                   80.00 usec
PL2                       1.00 dB
PL12                     17.00 dB
PL13                     17.00 dB
PL2W                 9.17820644 W
PL12W                0.23054613 W
PL13W                0.23054613 W
SFO2               300.1312005 MHz
SI                        32768
SF                 75.4677514 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40
```

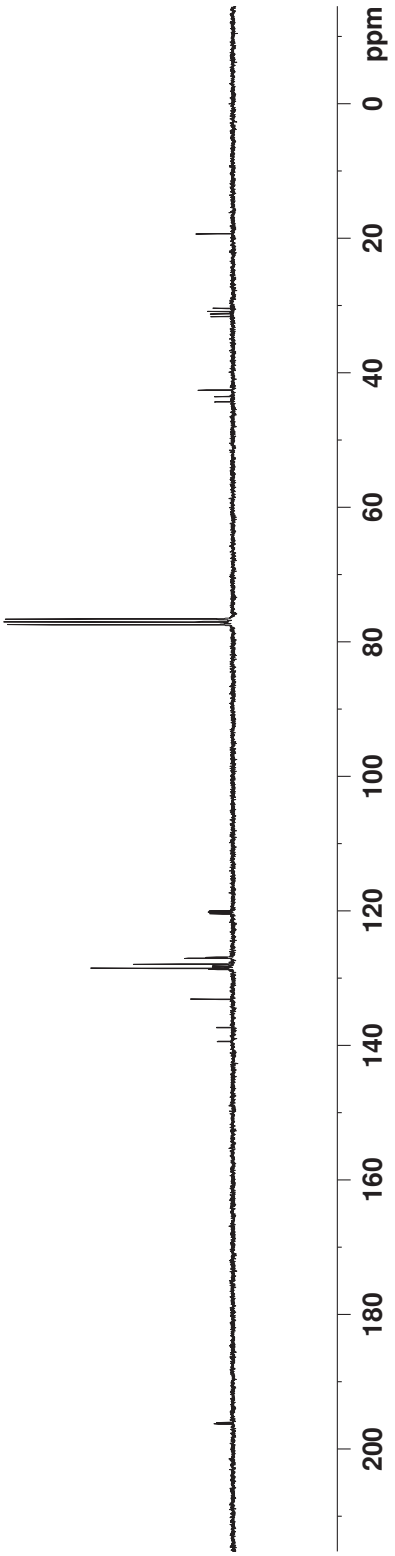




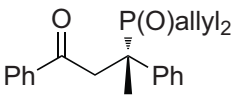




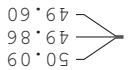
3a

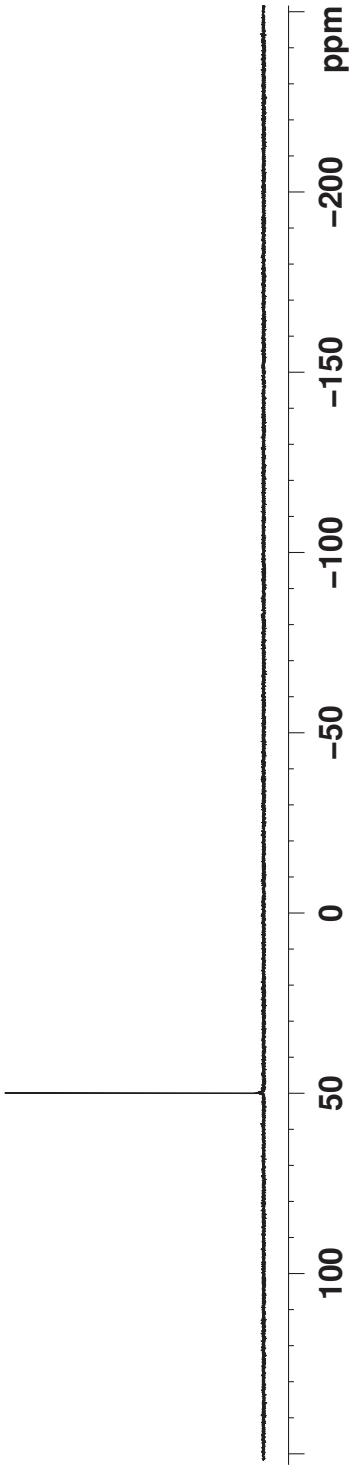


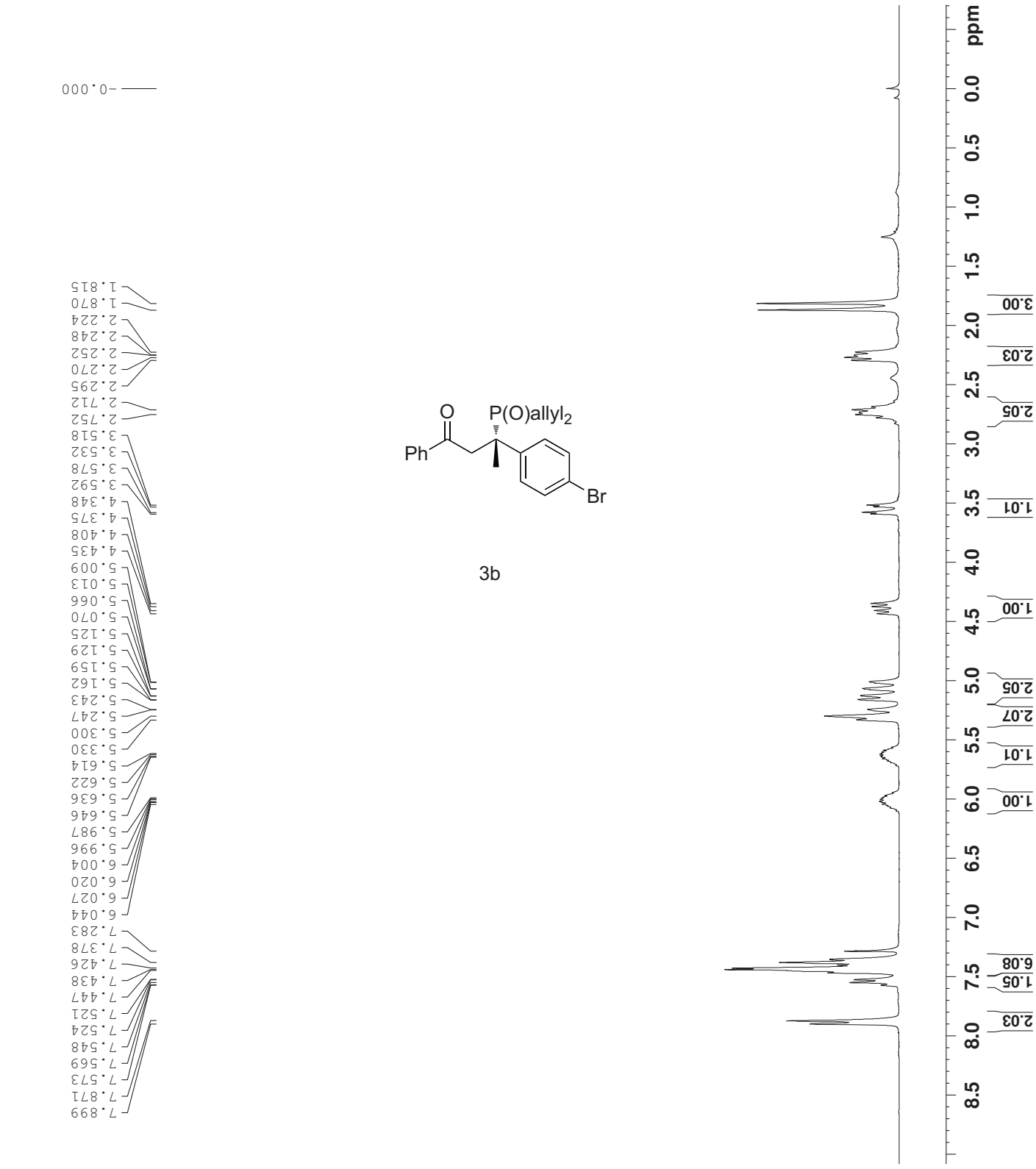
```
NAME                 101223
EXPNO                     3
PROCNO                    1
Date_              20101223
Time                 11.04
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                 49019.609 Hz
FIDRES              0.747980 Hz
AQ                  0.6685172 sec
RG                        203
DW                     10.200 usec
DE                      6.50 usec
TE                     289.9 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W                 9.17820644 W
PL12W                0.23054613 W
PL13W                0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



3a






NAME 110112
EXPNO 7
PROCNO 1
Date_ 20110112
Time 11.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 285.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299965 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.000
1.815
1.870
2.224
2.248
2.252
2.270
2.295
2.712
2.752
3.518
3.532
3.578
3.592
4.348
4.375
4.408
4.435
5.009
5.013
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5.070
5.125
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5.247
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6.027
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2.05
1.01
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1.01
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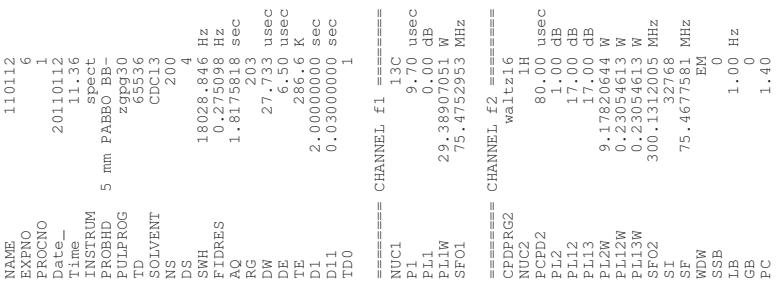
1.05
6.08
2.03

O
P(O)allyl2
Ph
Br

3b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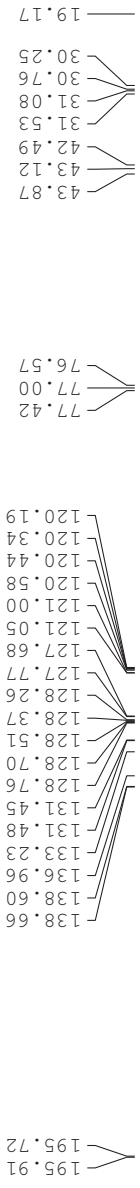
EXPNO 6
PROCNO 1
Date_ 20110112
Time 11.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 286.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677581 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


19.17
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31.08
31.53
42.49
43.12
43.87
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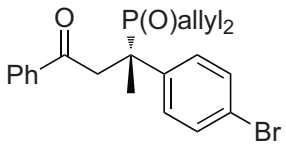
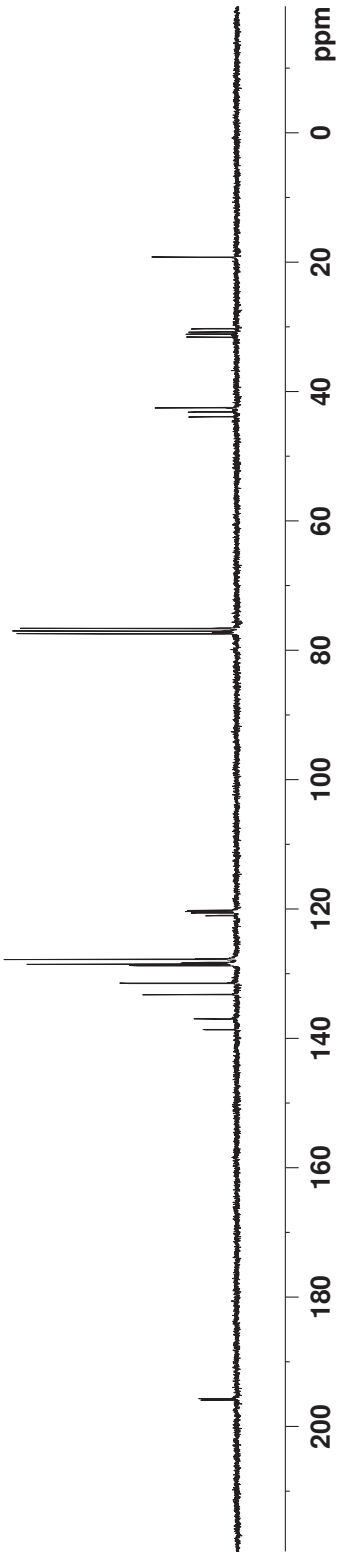
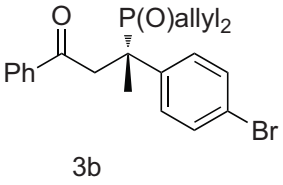
77.00
77.42
120.19
120.34
120.44
120.58
121.00
121.05
127.68
127.77
128.26
128.37
128.51
128.70
128.76
131.45
131.48
133.23
136.96
138.60
138.66
195.72
195.91


O
P(O)allyl2
Ph
Br

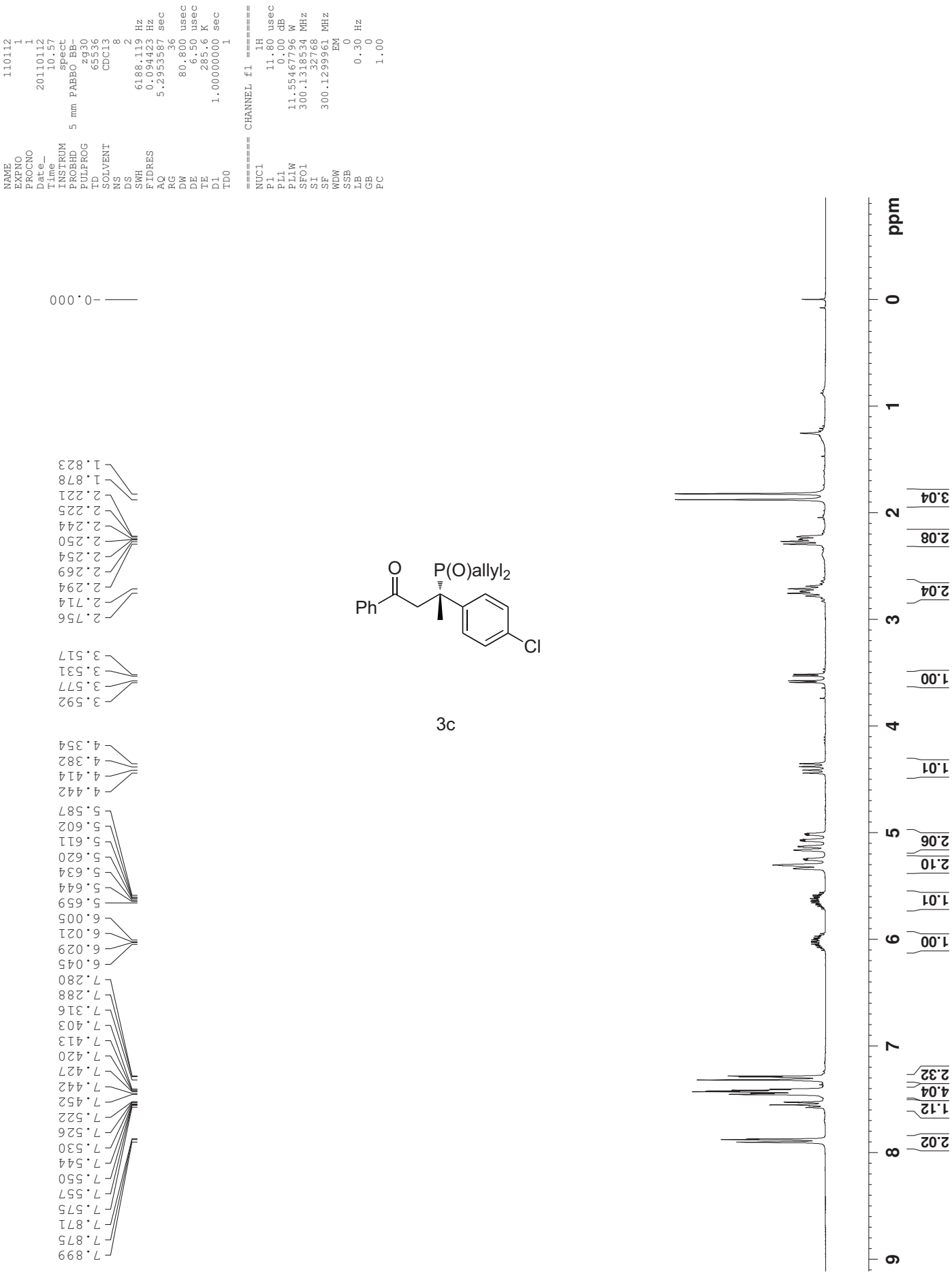
3b


ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                 110112
EXPNO                     8
PROCNO                    1
Date_              20110112
Time                 11.52
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     286.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                     9.10 usec
PL1                    0.00 dB
PL1W              36.92473221 W
SFO1              121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2              300.1312005 MHz
SI                      32768
SF                121.4948510 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40
```

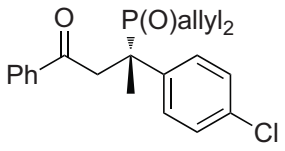


3b

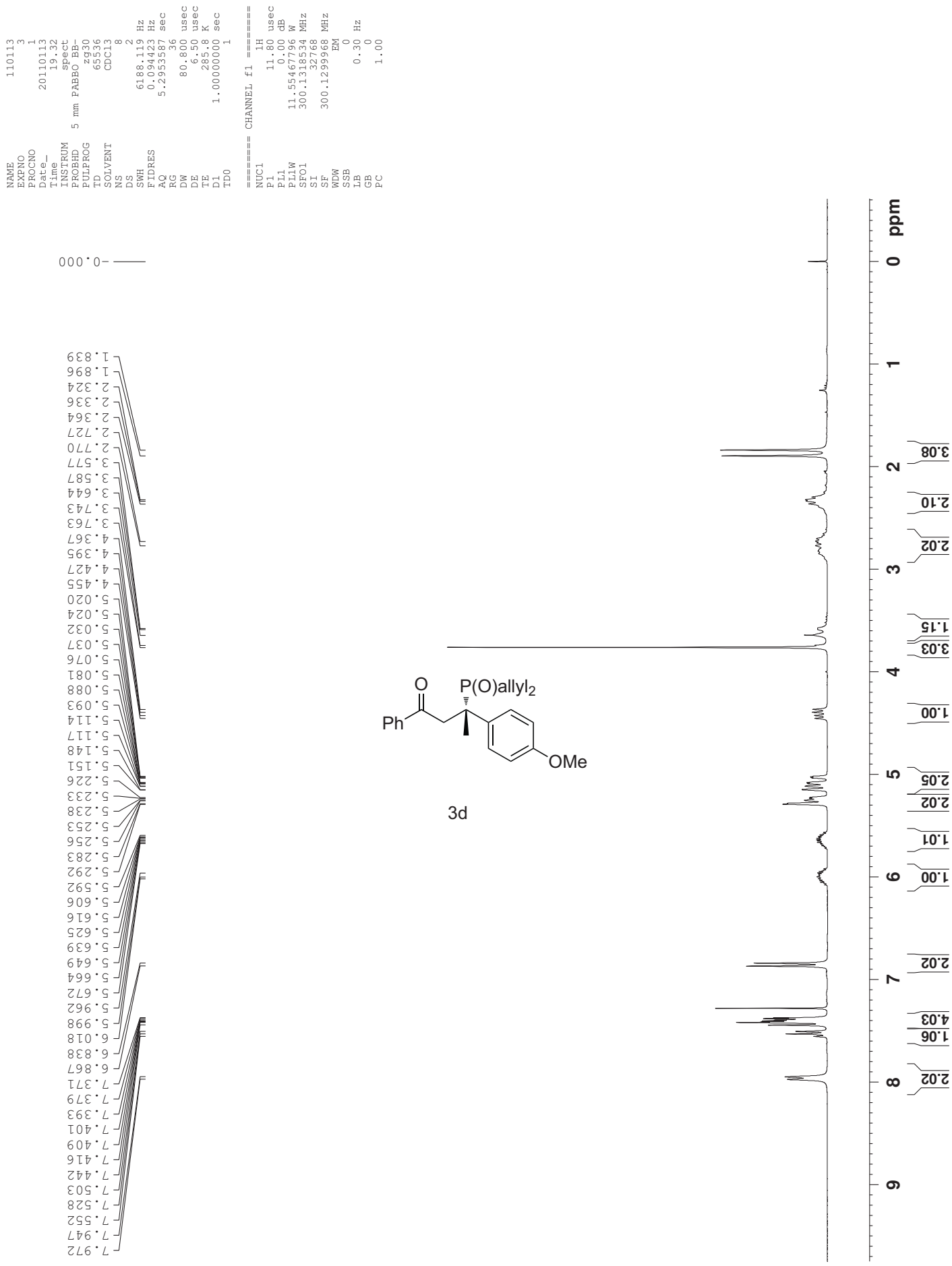

NAME 110112
EXPNO 1
PROCNO 1
Date_ 20110112
Time_ 10.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 285.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299961 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.823
1.878
2.221
2.225
2.244
2.250
2.254
2.269
2.294
2.714
2.756
3.517
3.531
3.577
3.592
4.354
4.382
4.414
4.442
5.587
5.602
5.611
5.620
5.634
5.644
5.659
6.005
6.021
6.029
6.045
7.280
7.288
7.316
7.403
7.413
7.420
7.427
7.442
7.452
7.522
7.526
7.530
7.544
7.550
7.557
7.575
7.871
7.875
7.899
O
P(O)allyl2
Ph
Cl
3c
ppm
0
1
2
3
4
5
6
7
8
9
3.04
2.08
2.04
1.00
1.01
2.06
2.10
1.01
1.00
2.32
4.04
1.12
2.02

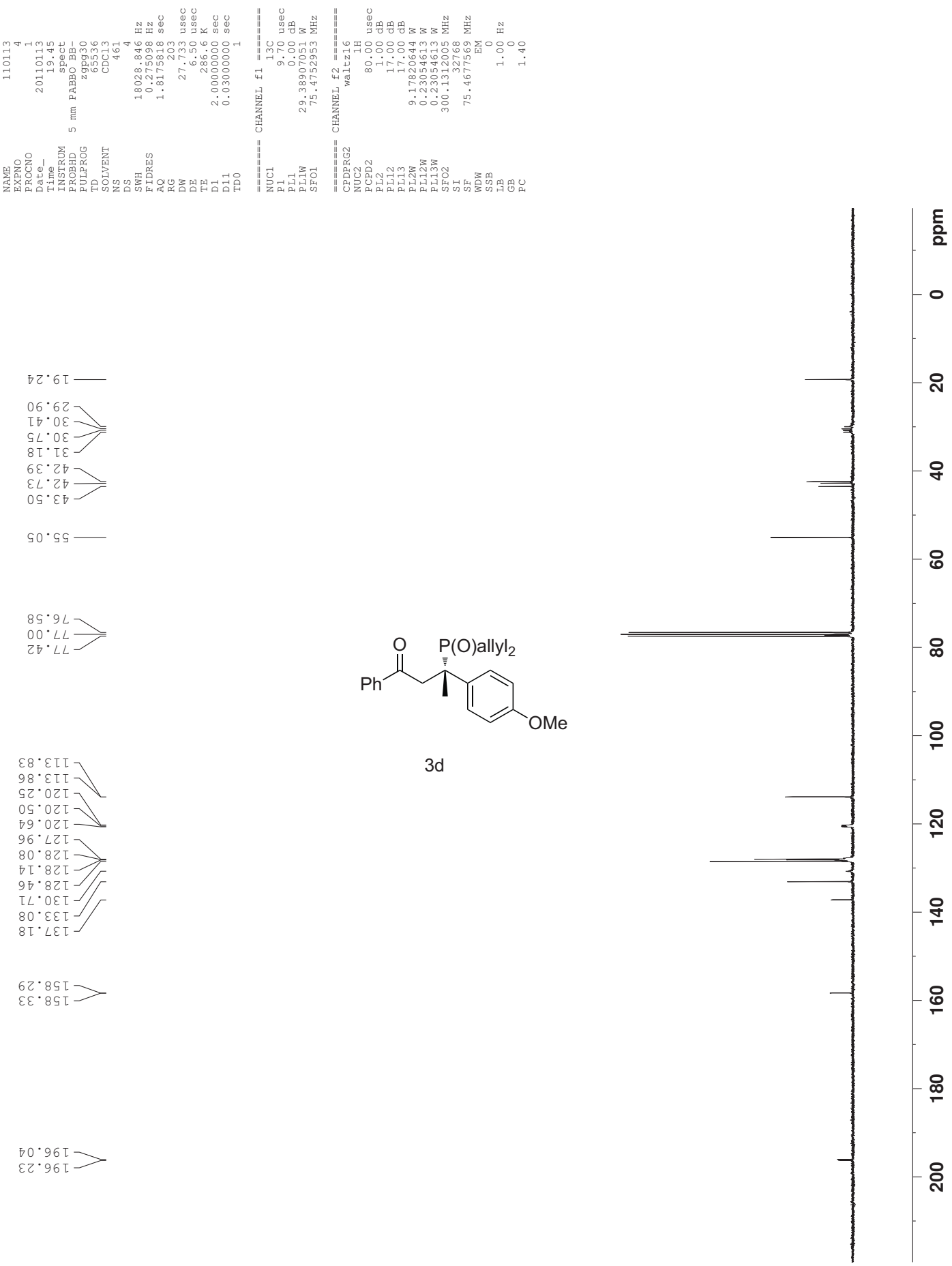

NAME 110112
EXPNO 2
PROCNO 1
Date_ 20110112
Time_ 11.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 255
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 286.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677565 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.25
30.28
30.81
31.11
31.58
42.53
43.06
43.81
76.58
77.00
77.43
120.18
120.33
120.44
120.58
127.73
127.79
127.83
128.31
128.36
128.42
128.53
128.57
132.79
132.84
133.25
137.00
137.03
138.07
138.13
195.79
195.97
O
P(O)allyl2
Ph
Cl
3c
ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                  110112
EXPNO                      3
PROCNO                     1
Date_               20110112
Time                   11.18
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                         16
DS                          4
SWH                 49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                        203
DW                     10.200 usec
DE                      6.50 usec
TE                     286.3 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                      9.10 usec
PL1                     0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  80.00 usec
PL2                     1.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2                300.1312005 MHz
SI                      32768
SF                  121.4948510 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

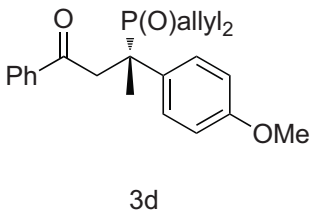


3c

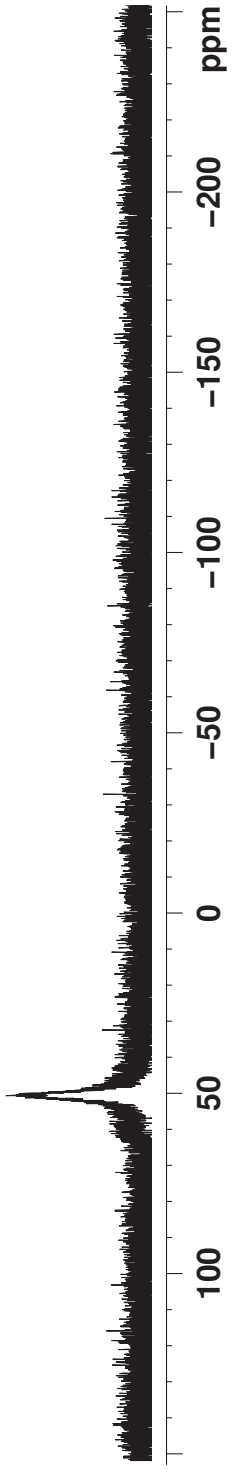

NAME 110113
EXPNO 3
PROCNO 1
Date_ 20110113
Time 19.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 285.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299968 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)allyl2
Ph
OMe
3d
ppm
3.08
2.10
2.02
1.15
3.03
1.00
2.05
2.02
1.01
1.00
2.02
4.03
1.06
2.02

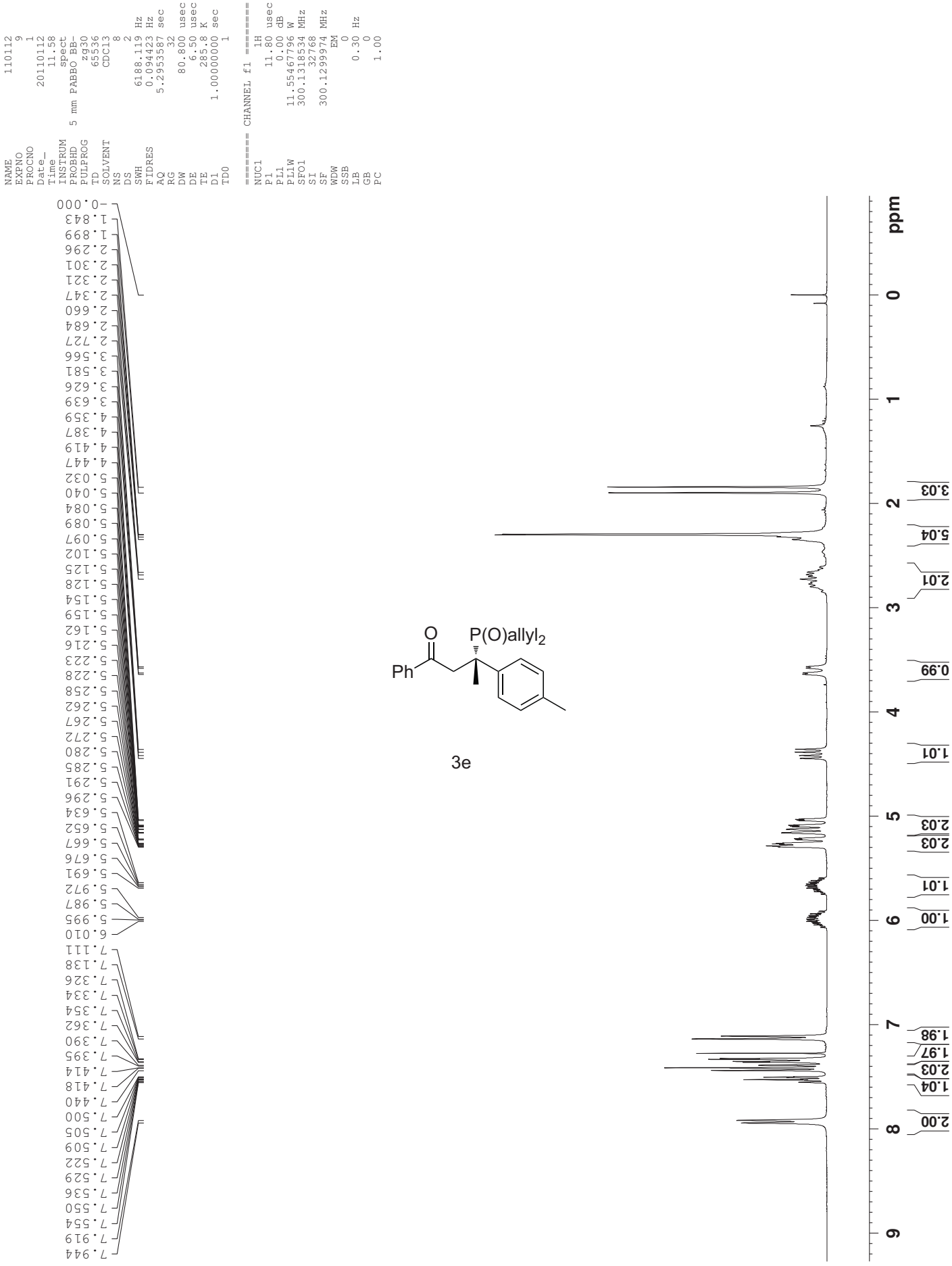

NAME 110113
EXPNO 4
PROCNO 1
Date_ 20110113
Time 19.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 461
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 286.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677569 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.24
29.90
30.41
30.75
31.18
42.39
42.73
43.50
55.05
76.58
77.00
77.42
113.83
113.86
120.25
120.50
120.64
127.96
128.08
128.14
128.46
130.71
133.08
137.18
158.29
158.33
196.04
196.23
O
P(O)allyl2
Ph
OMe
3d
ppm
0
20
40
60
80
100
120
140
160
180
200

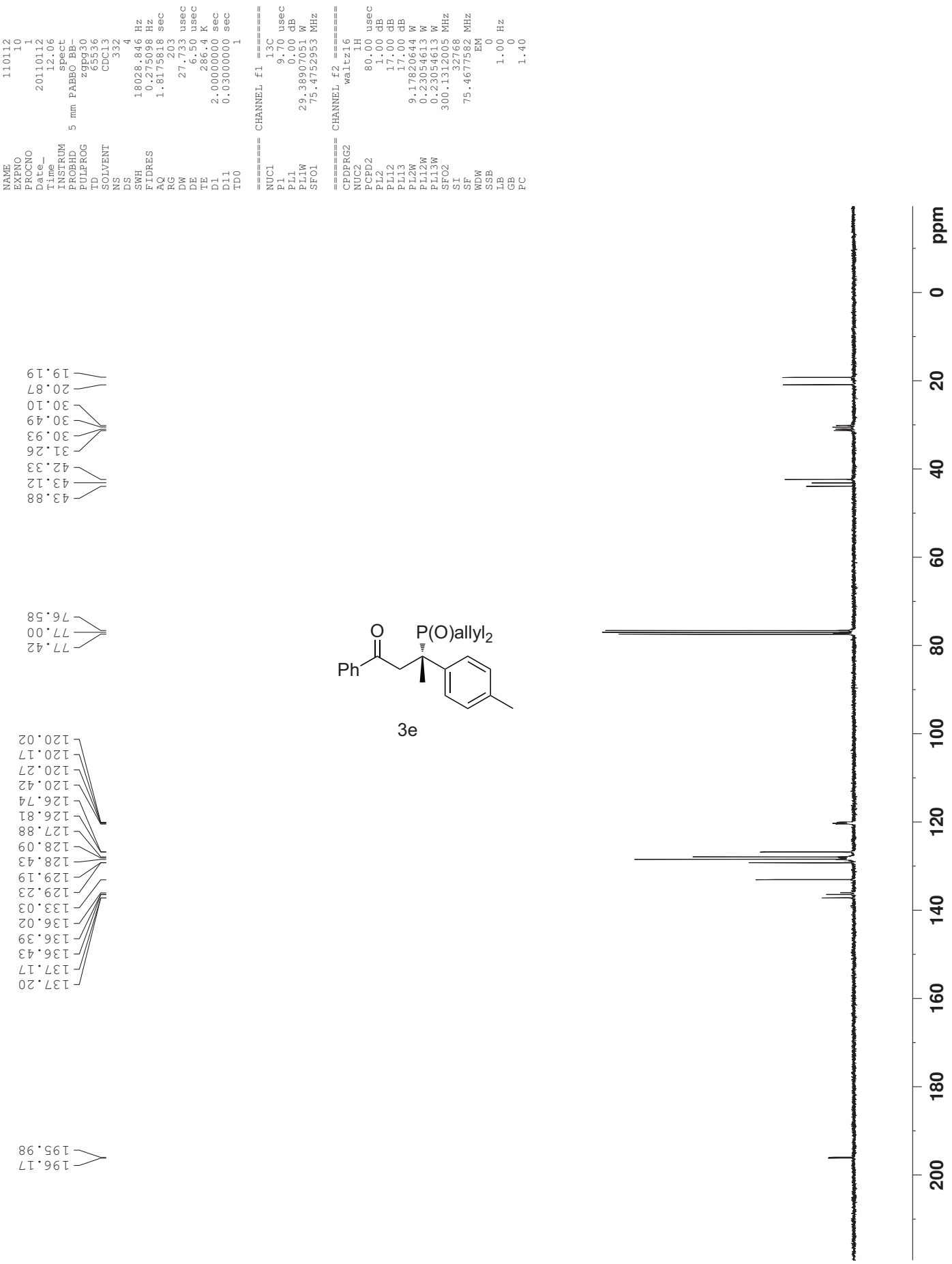
```
NAME                 110113
EXPNO                     2
PROCNO                    1
Date_              20110113
Time                 19.29
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                 49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                        203
DW                     10.200 usec
DE                      6.50 usec
TE                     285.9 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W                 9.17820644 W
PL12W                0.23054613 W
PL13W                0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



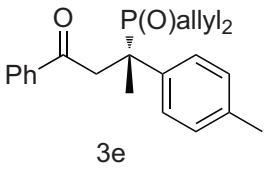
3d




NAME 110112
EXPNO 9
PROCNO 1
Date_ 20110112
Time 11.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 285.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299974 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.03
5.04
2.01
0.99
1.01
2.03
2.03
1.01
1.00
1.98
1.97
2.03
1.04
2.00
O
P(O)allyl2
Ph
3e

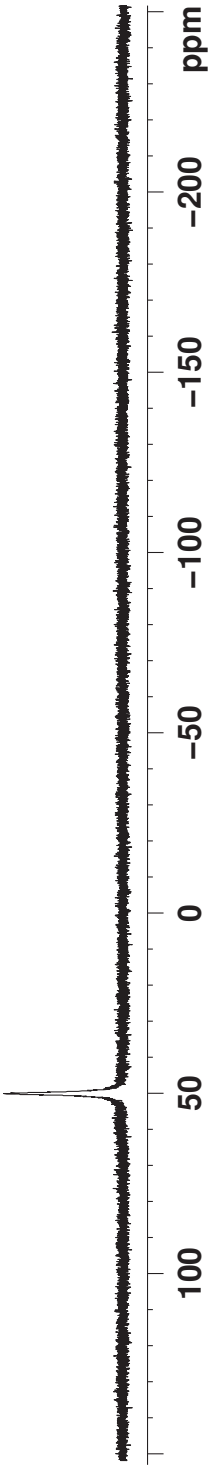

NAME 110112
EXPNO 10
PROCNO 1
Date_ 20110112
Time 12.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 332
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 286.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677582 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.19
20.87
30.10
30.49
30.93
31.26
42.33
43.12
43.88
77.42
77.00
76.58
120.02
120.17
120.27
120.42
126.74
126.81
127.88
128.09
128.43
129.19
129.23
133.03
136.02
136.39
136.43
137.17
137.20
196.17
195.98
O
P(O)allyl2
Ph
3e
ppm
0
20
40
60
80
100
120
140
160
180
200

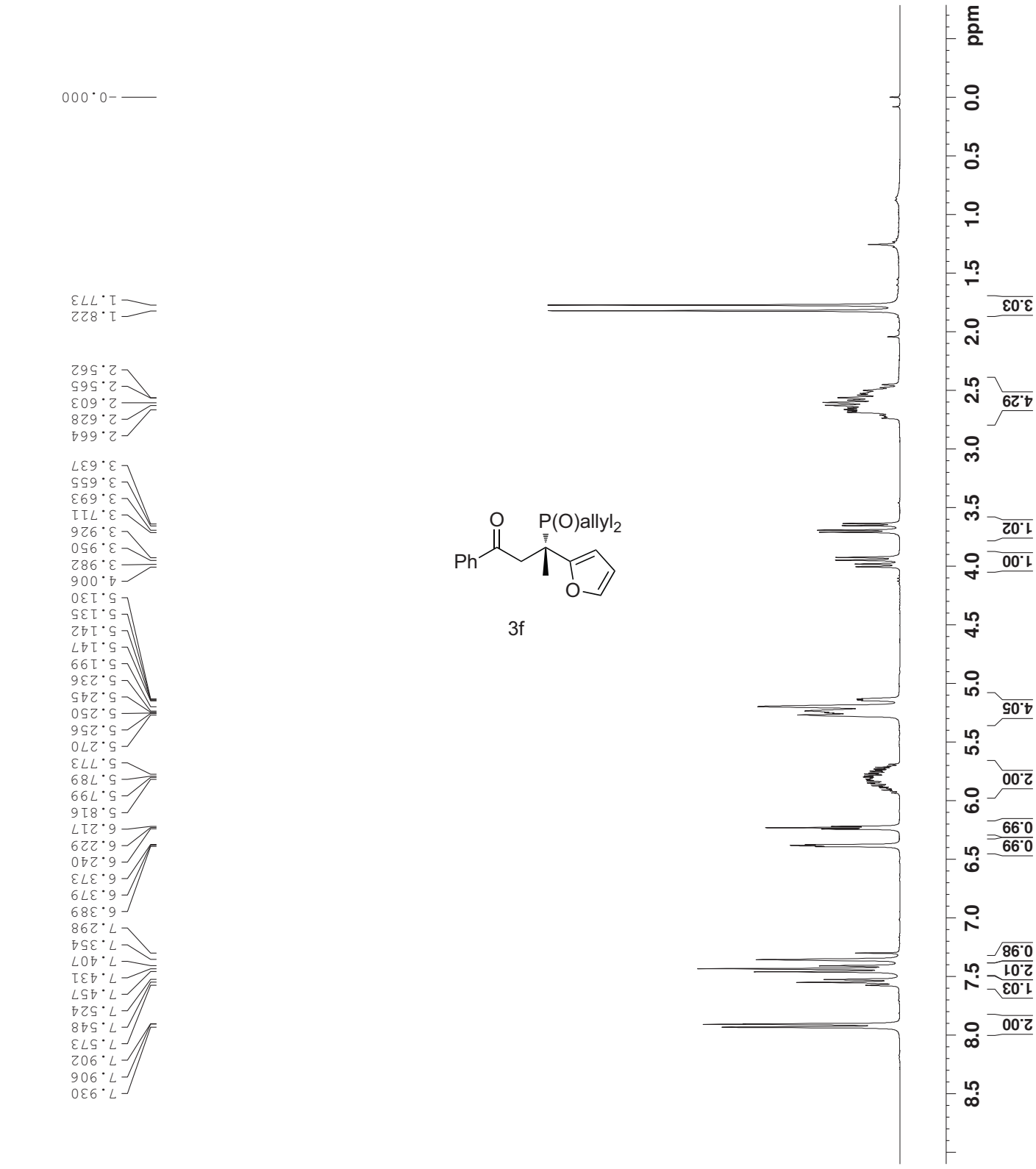
```
NAME                 110112
EXPNO                    12
PROCNO                    1
Date_              20110112
Time                  12.34
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     286.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W               36.92473221 W
SFO1               121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2               300.1312005 MHz
SI                       32768
SF                 121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

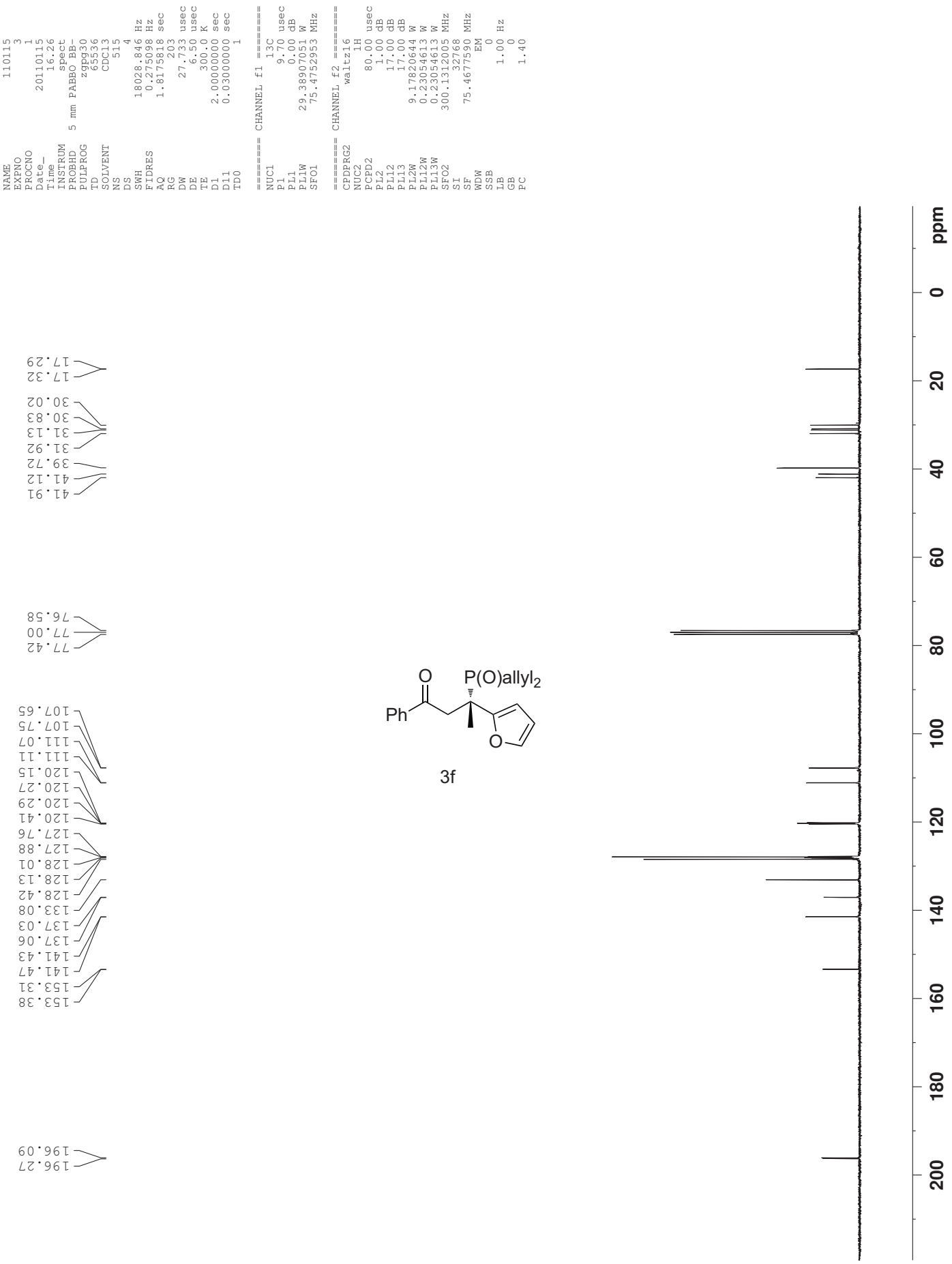


3e

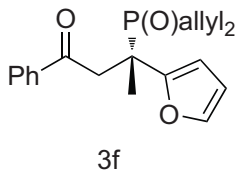
50.04



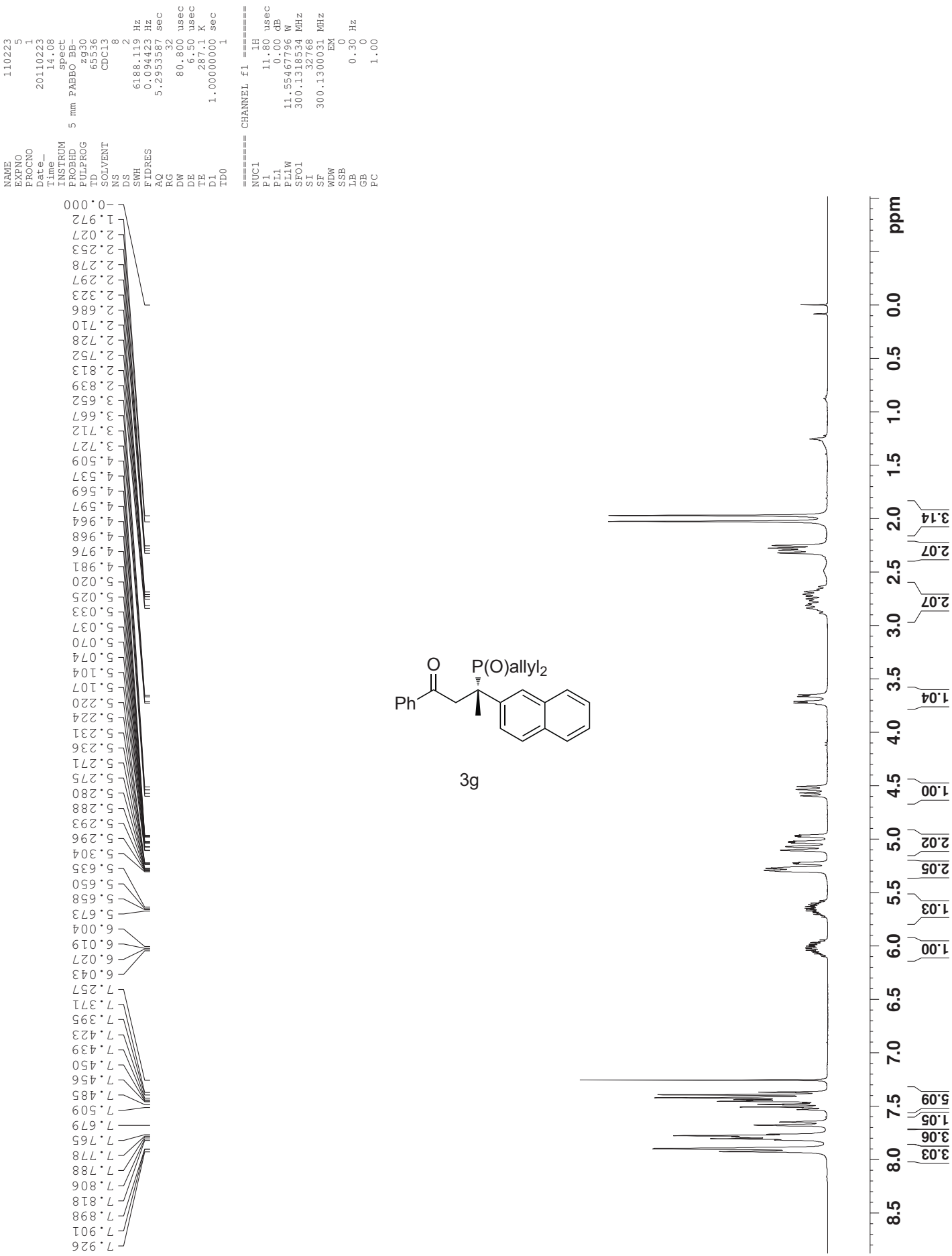

NAME 110115
EXPNO 2
PROCNO 1
Date_ 20110115
Time 15.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 28.5
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299908 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.822
1.773
2.562
2.565
2.603
2.628
2.664
3.637
3.655
3.693
3.711
3.926
3.950
3.982
4.006
5.130
5.135
5.142
5.147
5.199
5.236
5.245
5.250
5.256
5.270
5.773
5.789
5.799
5.816
6.217
6.229
6.240
6.373
6.379
6.389
7.298
7.354
7.407
7.431
7.457
7.524
7.548
7.573
7.902
7.906
7.930
O
P(O)allyl2
Ph
O
3f
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.03
4.29
1.02
1.00
4.05
2.00
0.99
0.99
0.98
2.01
1.03
2.00

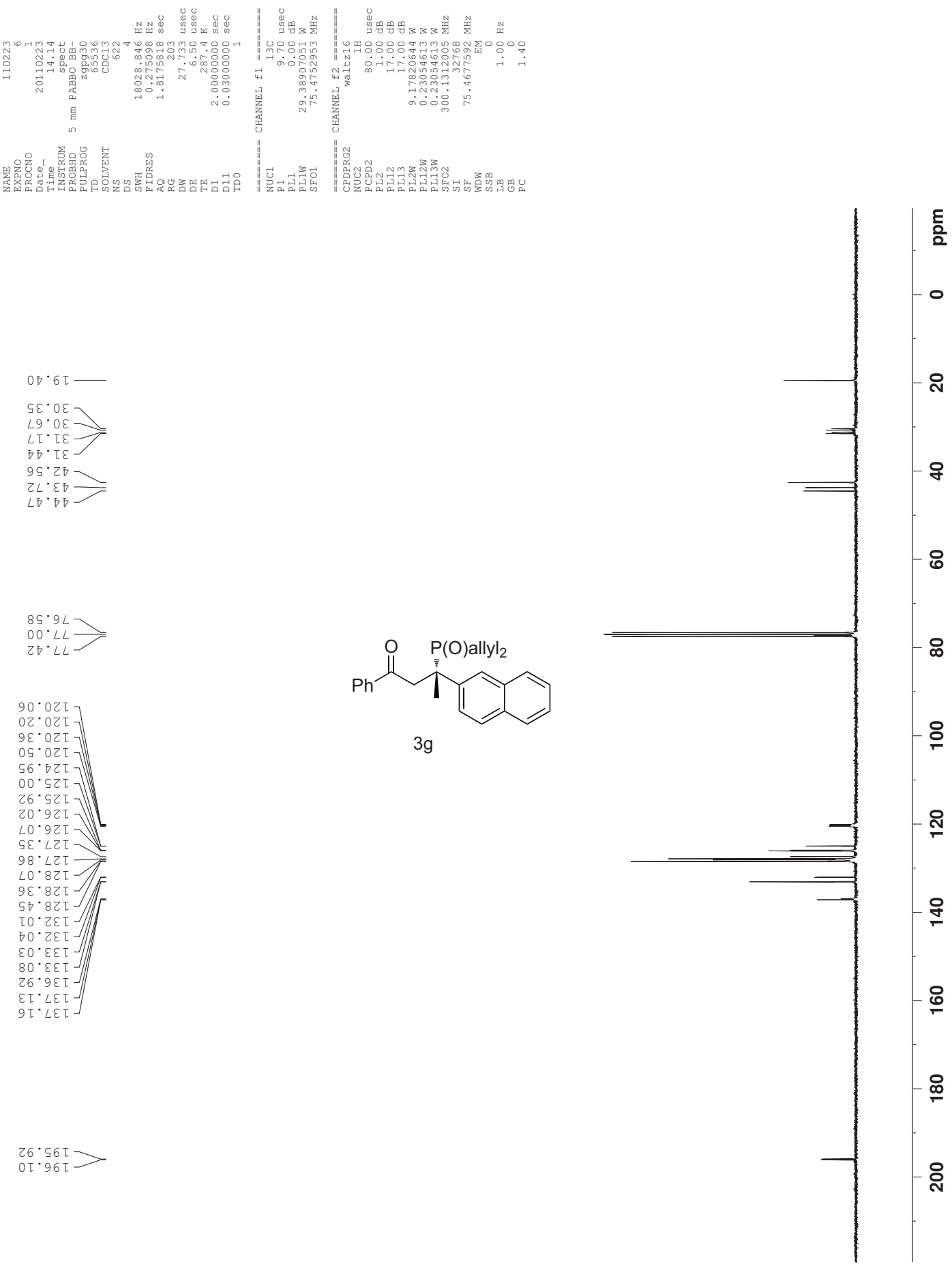

NAME 110115
EXPNO 3
PROCNO 1
Date_ 20110115
Time 16.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 515
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
17.29
17.32
30.02
30.83
31.13
31.92
39.72
41.12
41.91
76.58
77.00
77.42
107.65
107.75
111.07
111.11
120.15
120.27
120.29
120.41
127.76
127.88
128.01
128.13
128.42
133.08
137.03
137.06
141.43
141.47
153.31
153.38
196.09
196.27
O
P(O)allyl2
Ph
O
3f
ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                  110115
EXPNO                      1
PROCNO                     1
Date_               20110115
Time                   15.38
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                         16
DS                          4
SWH                 49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                        203
DW                     10.200 usec
DE                      6.50 usec
TE                     300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                      9.10 usec
PL1                     0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  80.00 usec
PL2                     1.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2                300.1312005 MHz
SI                      32768
SF                  121.4948510 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

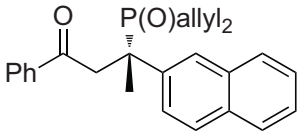


3f

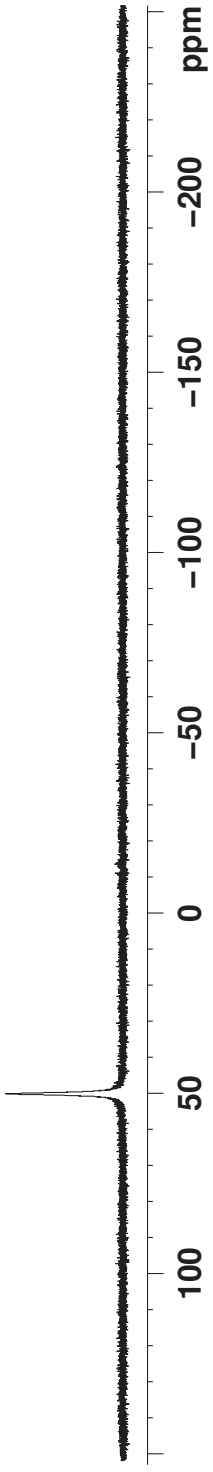

NAME 110223
EXPNO 5
PROCNO 1
Date_ 20110223
Time 14.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 287.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.14
2.07
2.07
1.04
1.00
2.02
2.05
1.03
1.00
5.09
1.05
3.06
3.03
O
P(O)allyl2
Ph
3g


NAME 110223
EXPNO 6
PROCNO 1
Date_ 20110223
Time 14.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 622
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677592 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.40
30.35
30.67
31.17
31.44
42.56
43.72
44.47
76.58
77.00
77.42
120.06
120.20
120.36
120.50
124.95
125.00
125.92
126.02
126.07
127.35
127.86
128.07
128.36
128.45
132.01
132.04
133.03
133.08
136.92
137.13
137.16
195.92
196.10
O
P(O)allyl2
Ph
3g
ppm
0
20
40
60
80
100
120
140
160
180
200

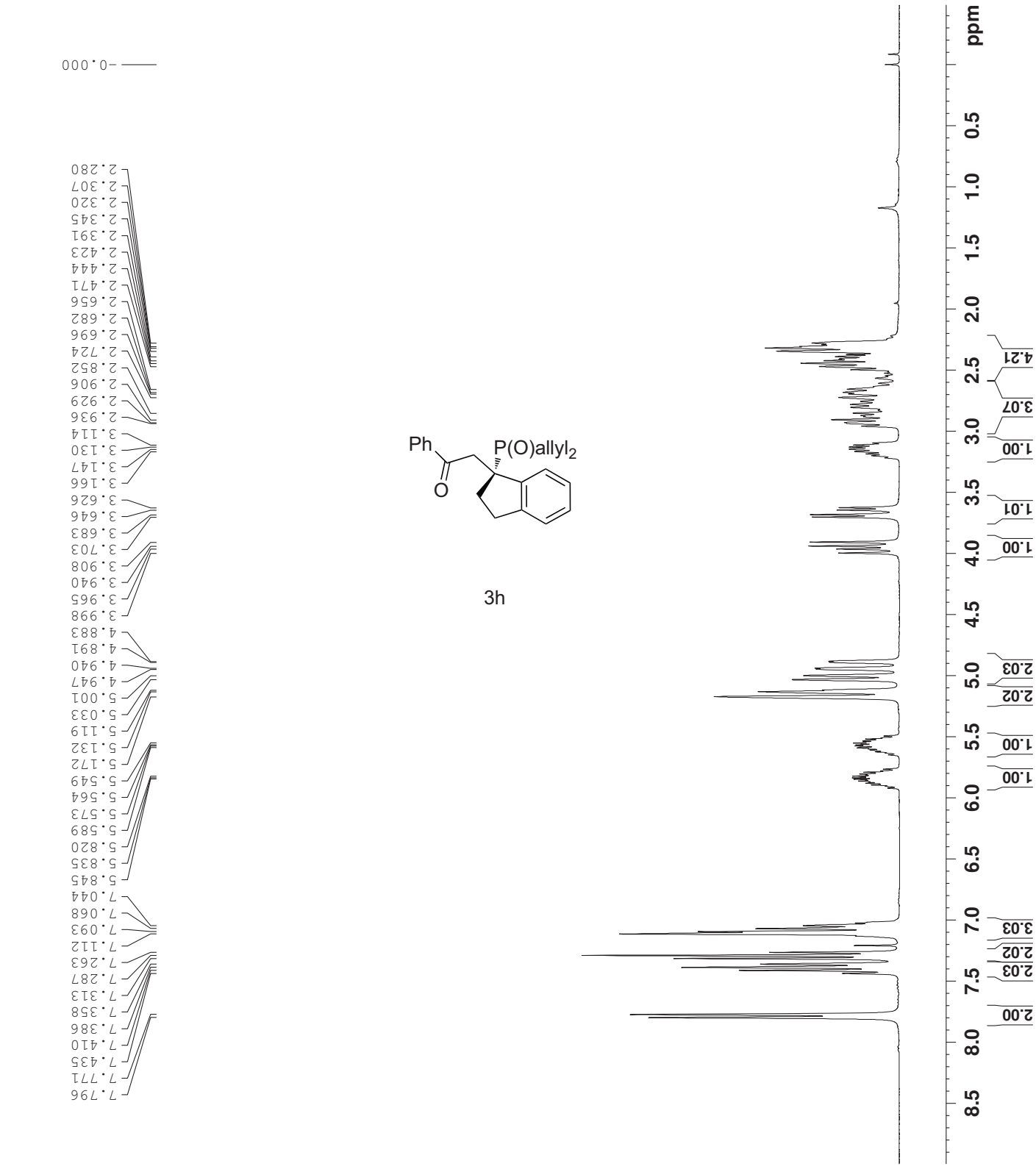
```
NAME                110223
EXPNO                    4
PROCNO                   1
Date_             20110223
Time                 14.05
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                      203
DW                     10.200 usec
DE                      6.50 usec
TE                    286.9 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                     31P
P1                      9.10 usec
PL1                     0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     1.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                      1.40
```

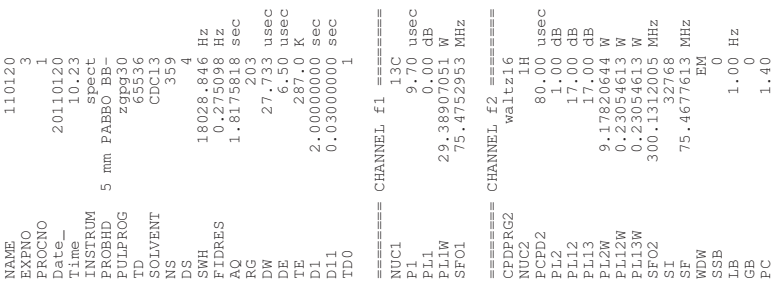


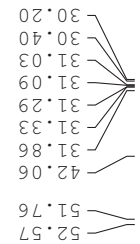
3g



50.04


NAME 110120
EXPNO 2
PROCNO 1
Date_ 20110120
Time 10.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 25.4
DW 80.800 usec
DE 6.50 usec
TE 286.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300183 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
P(O)allyl2
3h
ppm
4.21
3.07
1.00
1.01
1.00
2.03
2.02
1.00
1.00
3.03
2.02
2.03
2.00


NAME 110120
EXPNO 3
PROCNO 1
Date_ 20110120
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 359
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677613 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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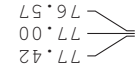
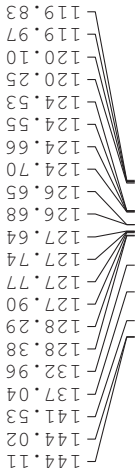
30.40
31.03
31.09
31.29
31.33
31.86
42.06
51.76
52.57


76.57
77.00
77.42


119.83
119.97
120.10
120.25
124.53
124.55
124.66
124.70
126.65
126.68
127.64
127.74
127.77
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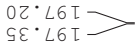
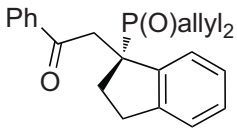
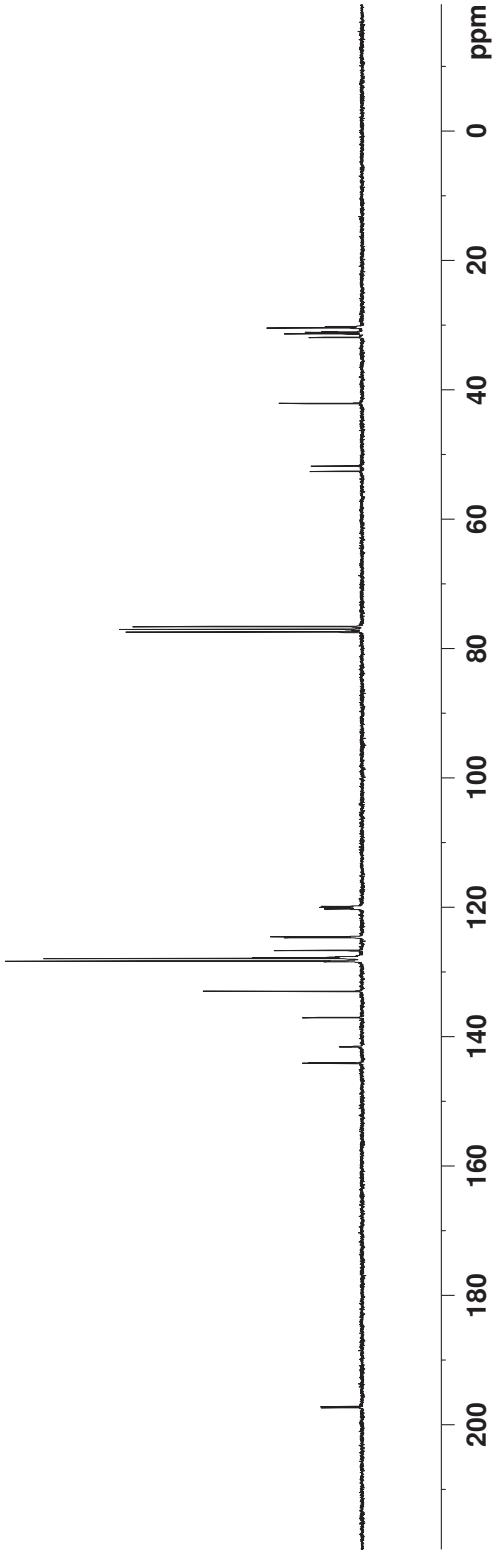
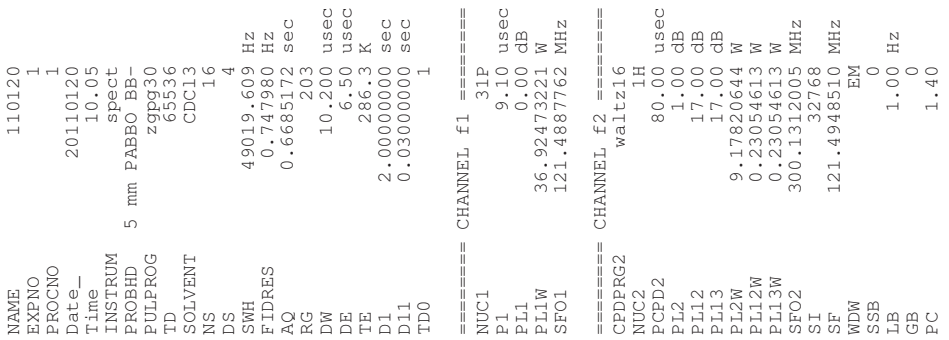
128.38
132.96
137.04
141.53
144.02
144.11


197.35
197.20

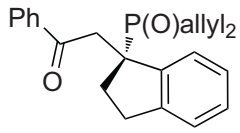

Ph
P(O)allyl2
O

3h

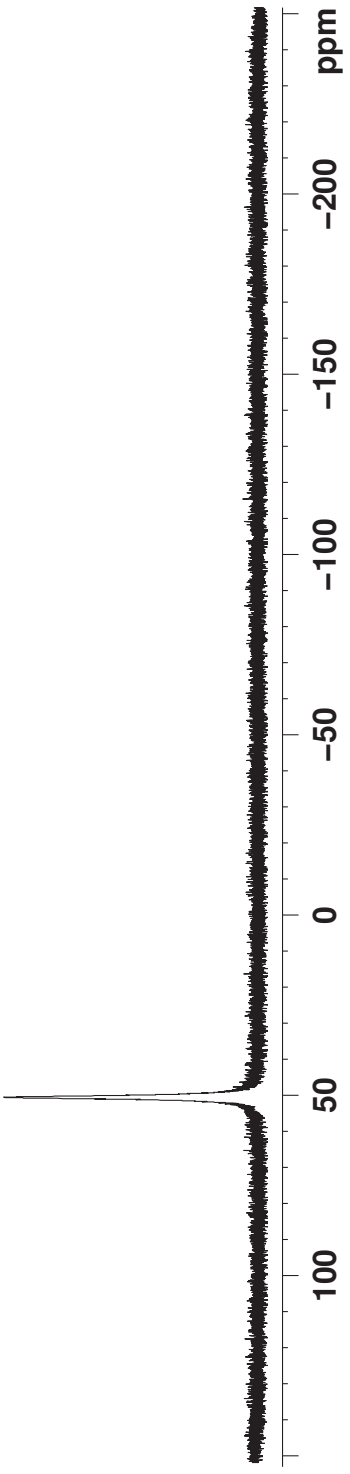

ppm
0
20
40
60
80
100
120
140
160
180
200

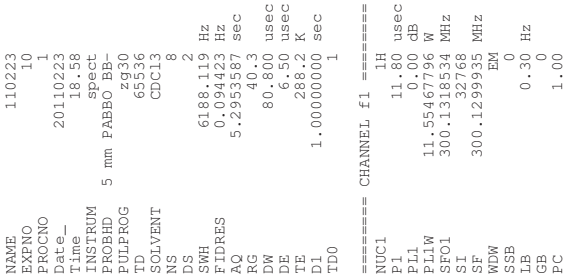


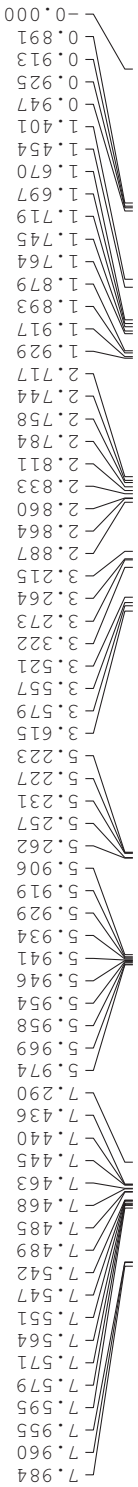
```
NAME                 110120
EXPNO                     1
PROCNO                    1
Date_              20110120
Time                  10.05
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES             0.747980 Hz
AQ                 0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                    286.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     31P
P1                    9.10 usec
PL1                   0.00 dB
PL1W           36.92473221 W
SFO1           121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W           9.17820644 W
PL12W          0.23054613 W
PL13W          0.23054613 W
SFO2           300.1312005 MHz
SI                    32768
SF             121.4948510 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



3h




NAME 110223
EXPNO 10
PROCNO 1
Date_ 20110223
Time_ 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 40.3
DW 80.800 usec
DE 6.50 usec
TE 288.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299935 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


0.000
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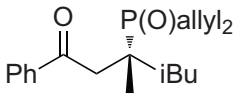
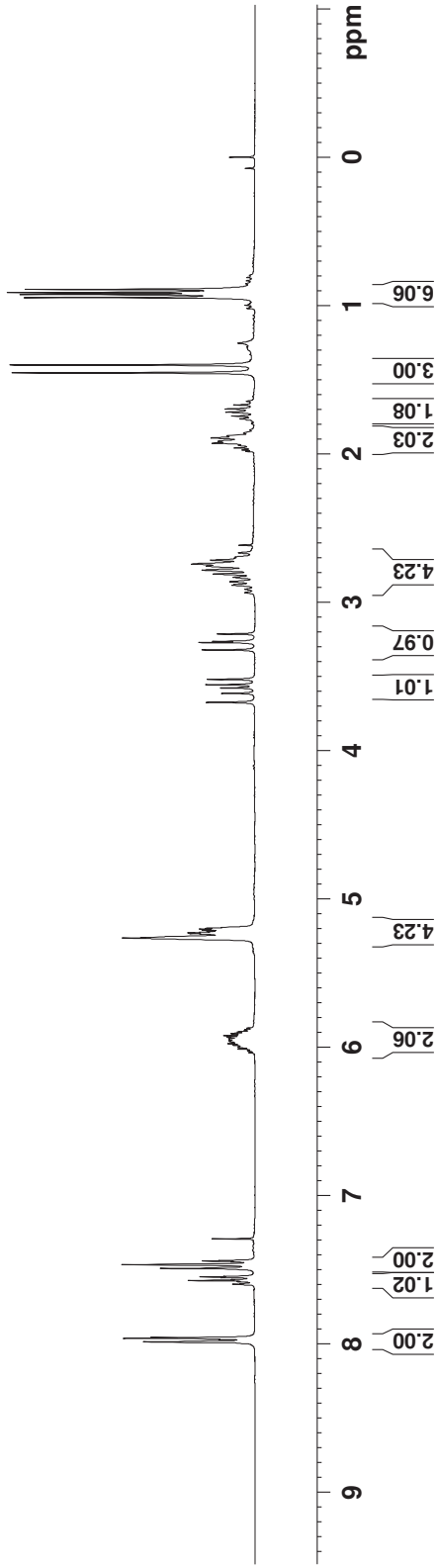
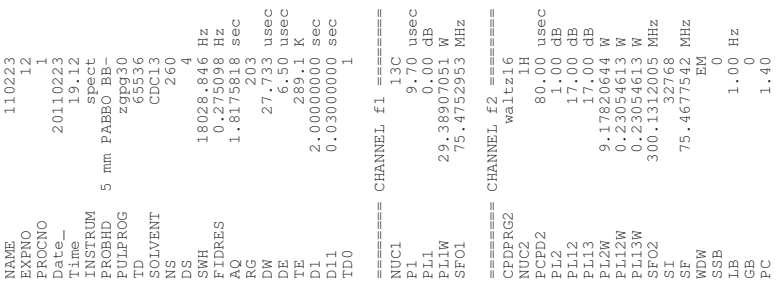
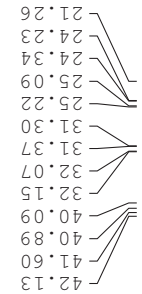
0.913
0.925
0.947
1.401
1.454
1.670
1.697
1.719
1.745
1.764
1.879
1.893
1.917
1.929
2.717
2.744
2.758
2.784
2.811
2.833
2.860
2.864
2.887
3.215
3.264
3.273
3.322
3.521
3.557
3.579
3.615
5.223
5.227
5.231
5.257
5.262
5.906
5.919
5.929
5.934
5.941
5.946
5.954
5.958
5.969
5.974
7.290
7.436
7.440
7.445
7.463
7.468
7.485
7.489
7.542
7.547
7.551
7.564
7.571
7.579
7.595
7.955
7.960
7.984

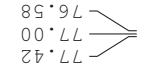

O
P(O)allyl2
Ph
iBu

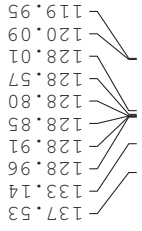
3i

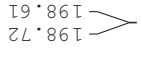

ppm
0
1
2
3
4
5
6
7
8
9
6.06
3.00
1.08
2.03
4.23
0.97
1.01
4.23
2.06
2.00
1.02
2.00

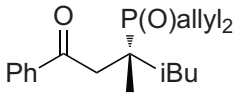

NAME 110223
EXPNO 12
PROCNO 1
Date_ 20110223
Time 19.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 260
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81775818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


21.26
24.23
24.34
25.09
25.22
31.30
31.37
32.07
32.15
40.09
40.89
41.60
42.13

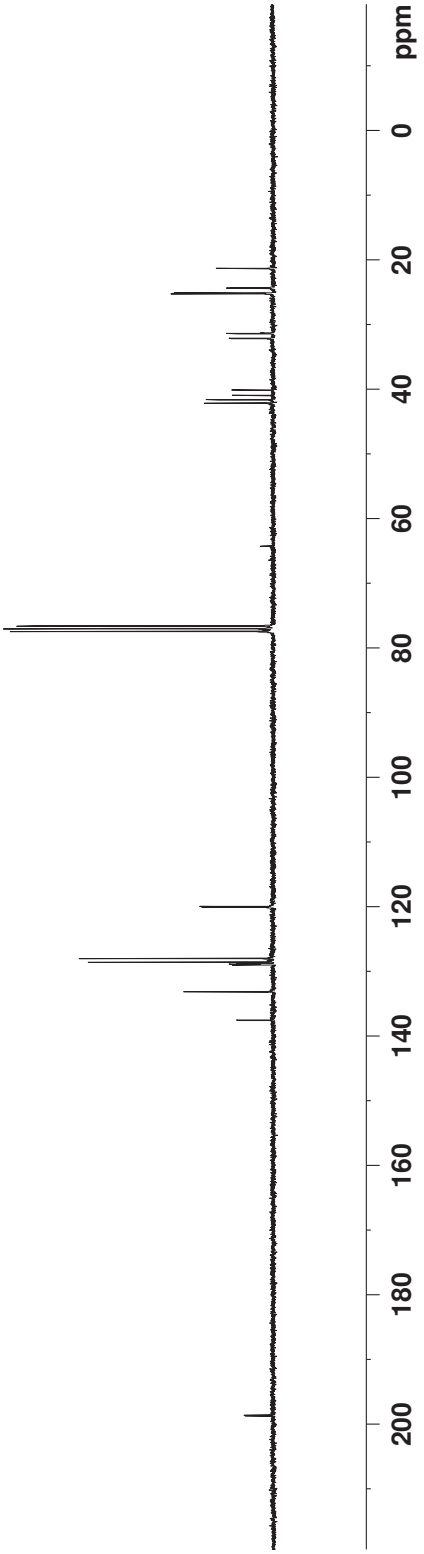

76.58
77.00
77.42


119.95
120.09
128.01
128.57
128.80
128.85
128.91
128.96
133.14
137.53


198.61
198.72


O
P(O)allyl2
Ph
iBu

3i

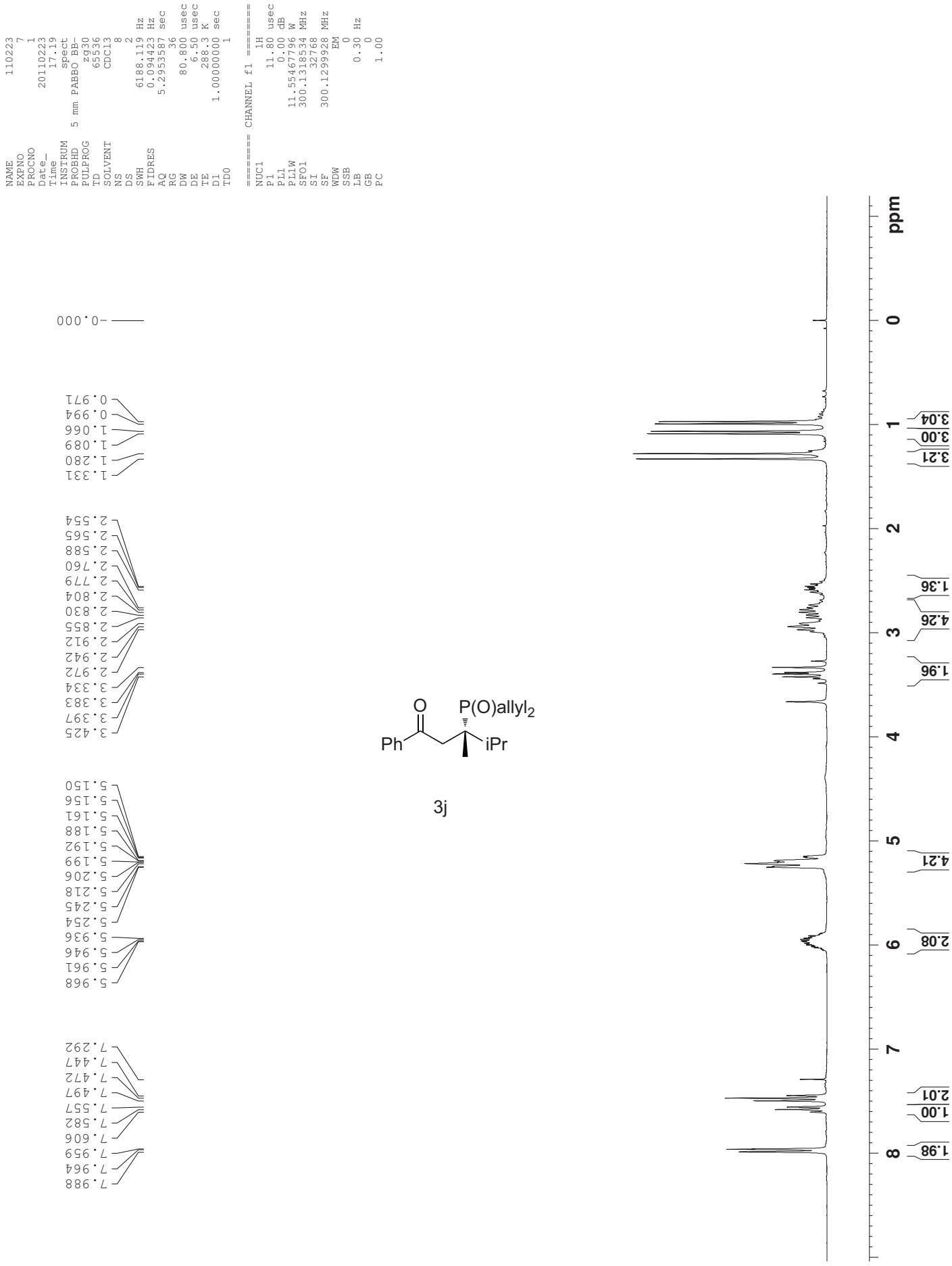

ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                 110223
EXPNO                    11
PROCNO                    1
Date_              20110223
Time                 19.02
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     288.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W               36.92473221 W
SFO1               121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2               300.1312005 MHz
SI                       32768
SF                 121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```


3i


NAME 110223
EXPNO 7
PROCNO 1
Date_ 20110223
Time 17.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 288.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299928 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.000
0.971
0.994
1.066
1.089
1.280
1.331
2.554
2.565
2.588
2.760
2.779
2.804
2.830
2.855
2.912
2.942
2.972
3.334
3.383
3.397
3.425
5.150
5.156
5.161
5.188
5.192
5.199
5.206
5.218
5.245
5.254
5.936
5.946
5.961
5.968
7.292
7.447
7.472
7.497
7.557
7.582
7.606
7.959
7.964
7.988
ppm
0
1
2
3
4
5
6
7
8
3.04
3.00
3.21
1.36
4.26
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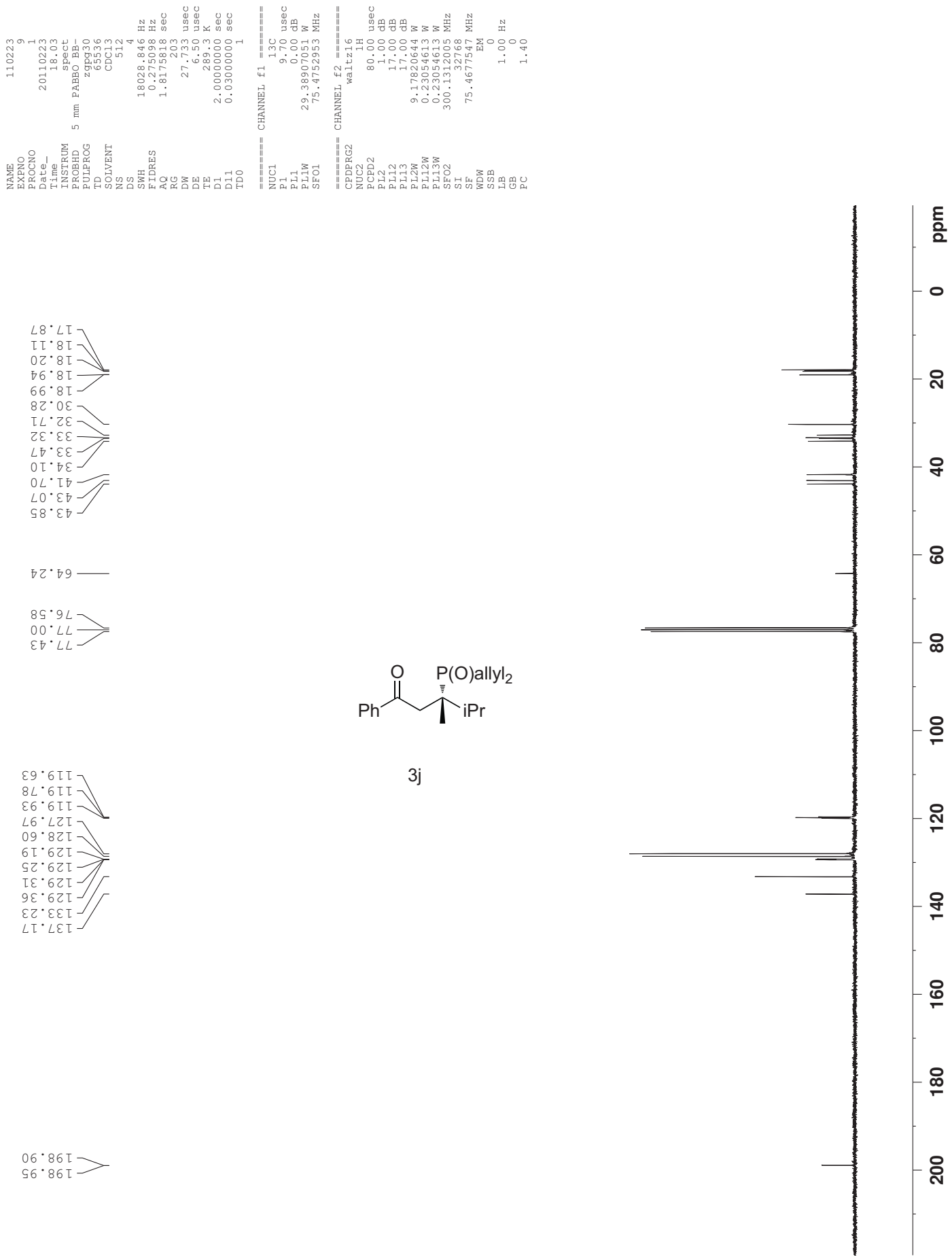
4.21
2.08
2.01
1.00
1.98
O
P(O)allyl2
Ph
iPr

3j


NAME 110223
EXPNO 9
PROCNO 1
Date_ 20110223
Time_ 18.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
17.87
18.11
18.20
18.94
18.99
30.28
32.71
33.32
33.47
34.10
41.70
43.07
43.85
64.24
76.58
77.00
77.43
119.63
119.78
119.93
127.97
128.60
129.19
129.25
129.31
129.36
133.23
137.17
198.90
198.95
O
P(O)allyl2
Ph
iPr
3j
ppm
0
20
40
60
80
100
120
140
160
180
200

NAME 110223
EXPNO 8
PROCNO 1
Date_ 20110223
Time 17.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15
DS 4
SWH 49019.609 Hz
FIDRES 0.747980 Hz
AQ 0.6685172 sec
RG 203
DW 10.200 usec
DE 6.50 usec
TE 288.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 9.10 usec
PL1 0.00 dB
PL1W 36.92473221 W
SFO1 121.4887762 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

O
P(O)allyl2
Ph
iPr

3j

53.76
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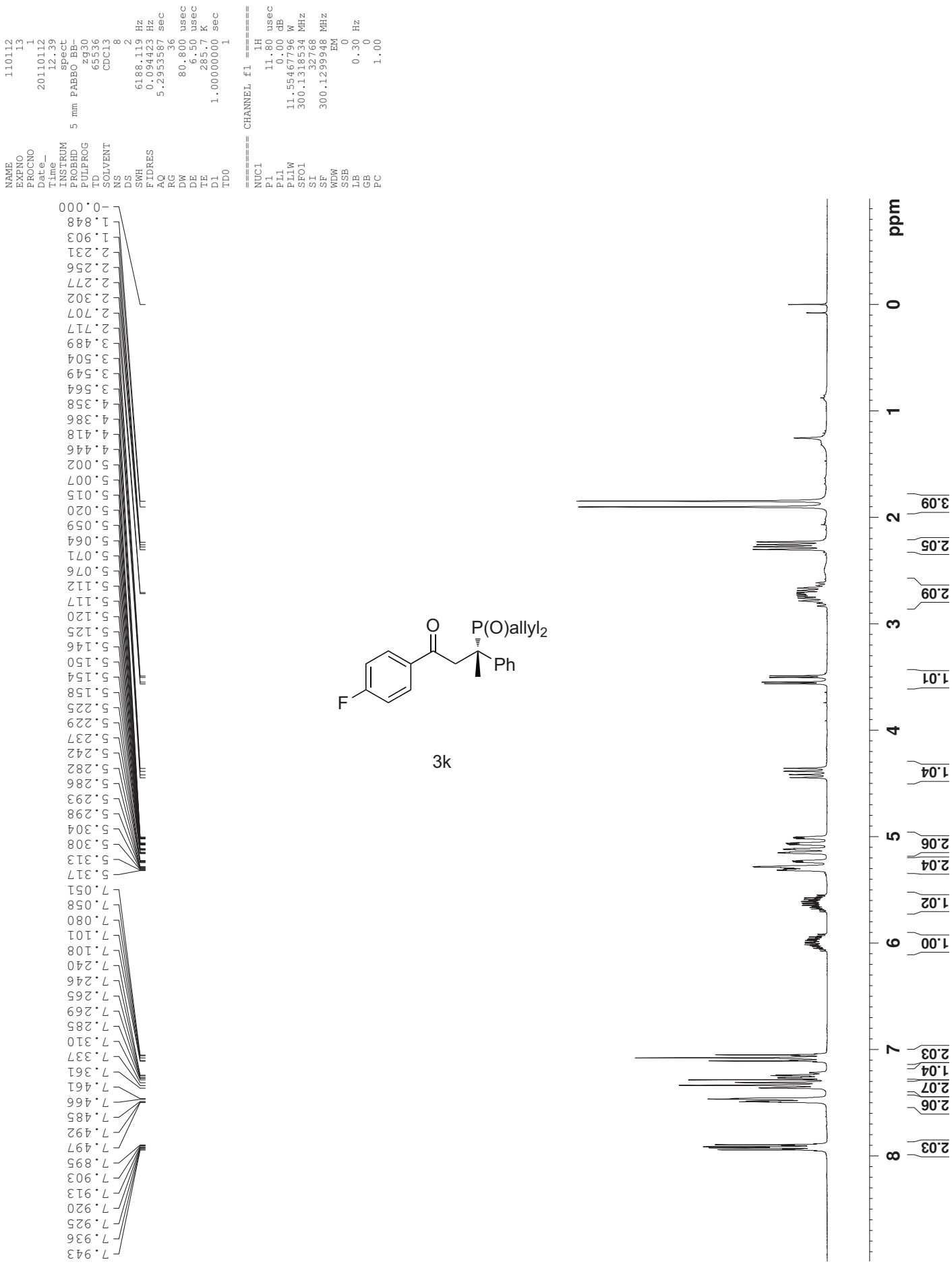
50
0
−50
−100
−150
−200
ppm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EXPNO 13
PROCNO 1
Date_ 20110112
Time_ 12.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 285.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299948 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
P(O)allyl2
Ph
F
3k
ppm
3.09
2.05
2.09
1.01
1.04
2.06
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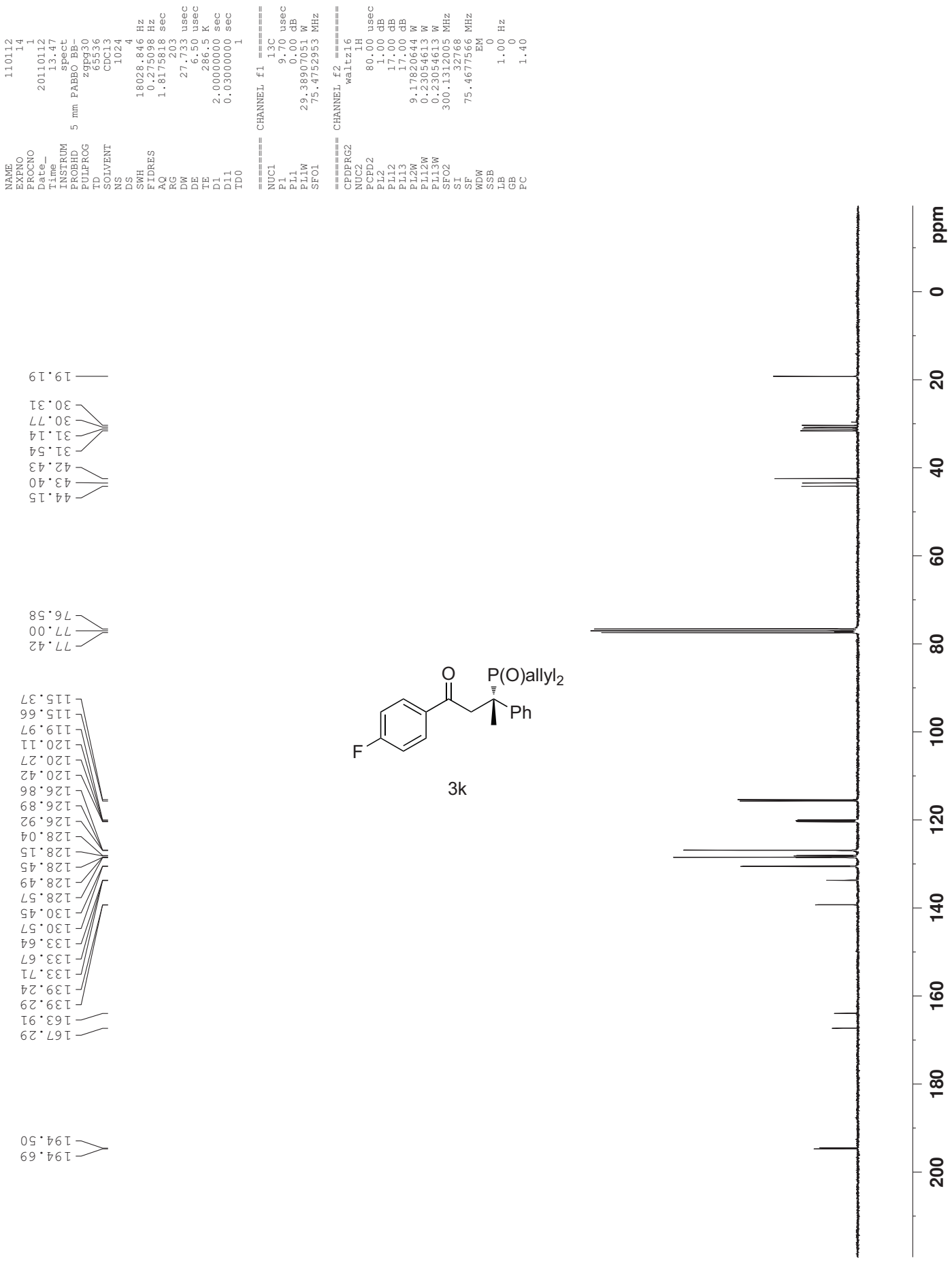
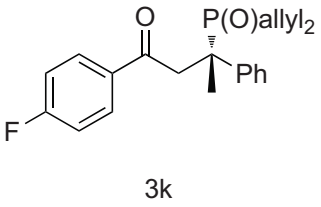
1.00
2.03
1.04
2.07
2.06
2.03


NAME 110112
EXPNO 14
PROCNO 1
Date_ 20110112
Time 13.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 286.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677566 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.19
30.31
30.77
31.14
31.54
42.43
43.40
44.15
76.58
77.00
77.42
115.37
115.66
119.97
120.11
120.27
120.42
126.86
126.89
126.92
128.04
128.15
128.45
128.49
128.57
130.45
130.57
133.64
133.67
133.71
139.24
139.29
163.91
167.29
194.50
194.69
O
P(O)allyl2
Ph
F
3k
ppm
0
20
40
60
80
100
120
140
160
180
200

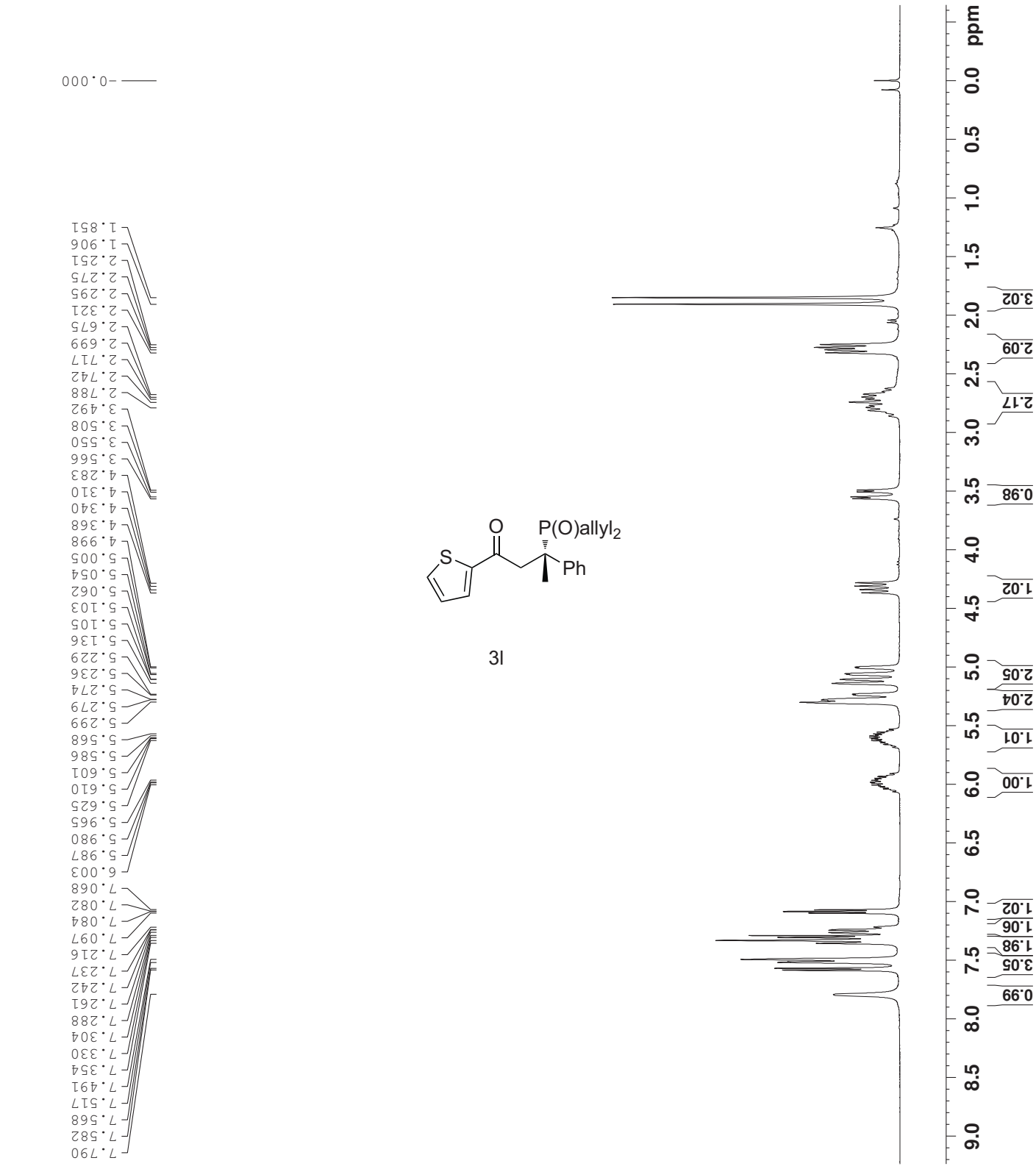
```
NAME                 110112
EXPNO                    15
PROCNO                    1
Date_              20110112
Time                  14.53
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     285.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     31P
P1                     9.10 usec
PL1                    0.00 dB
PL1W              36.92473221 W
SFO1              121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2              300.1312005 MHz
SI                      32768
SF                121.4948510 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.40
```



3k


NAME 110123
EXPNO 1
PROCNO 1
Date_ 20110223
Time_ 10.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 286.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299938 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.851
1.906
2.251
2.275
2.295
2.321
2.675
2.699
2.717
2.742
2.788
3.492
3.508
3.550
3.566
4.283
4.310
4.340
4.368
4.998
5.005
5.054
5.062
5.103
5.105
5.136
5.229
5.236
5.274
5.279
5.299
5.568
5.586
5.601
5.610
5.625
5.965
5.980
5.987
6.003
7.068
7.082
7.084
7.097
7.216
7.237
7.242
7.261
7.288
7.304
7.330
7.354
7.491
7.517
7.568
7.582
7.790
O
P(O)allyl2
S
Ph
3l
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
3.02
2.09
2.17
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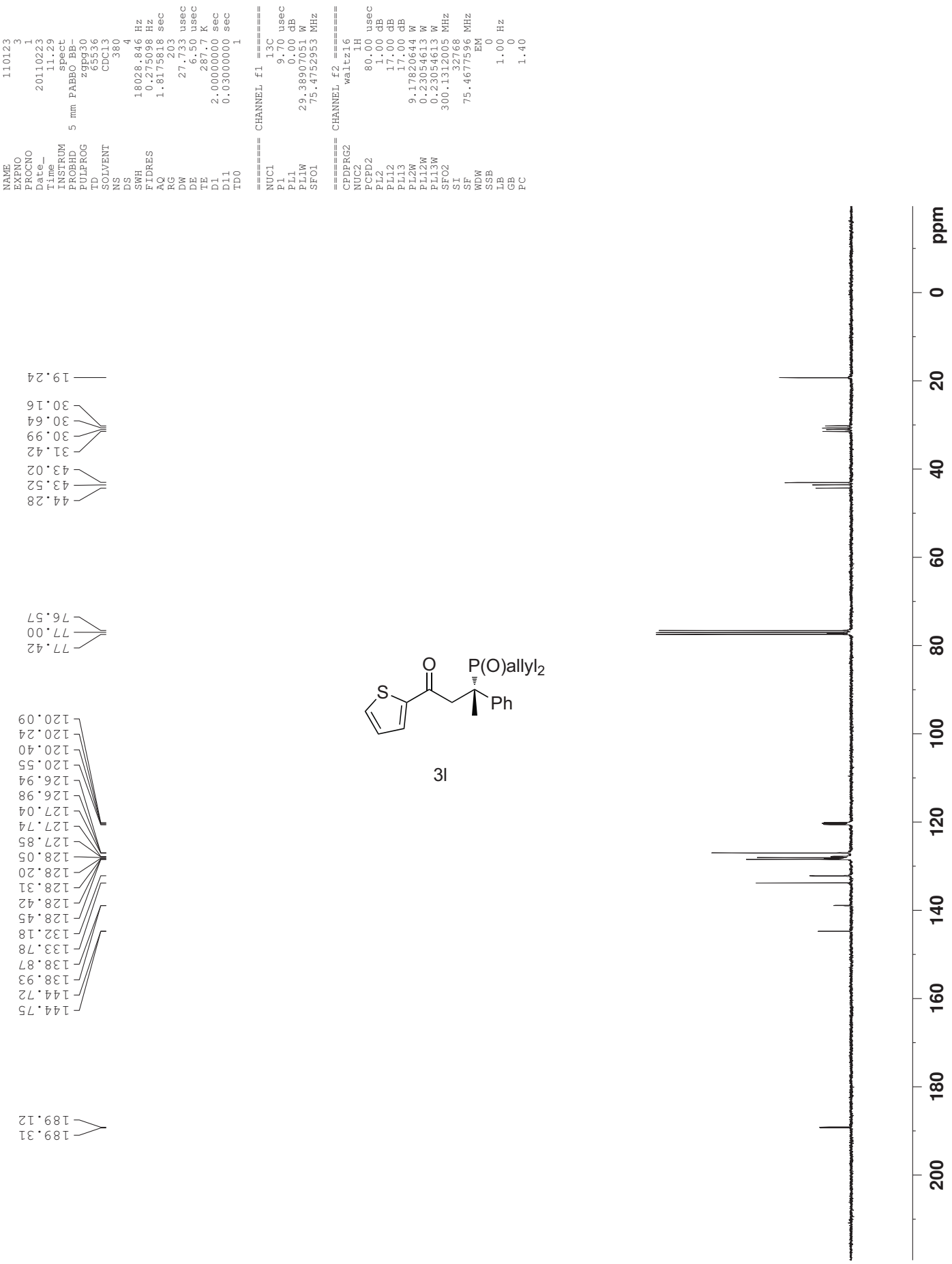
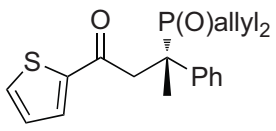
1.02
2.05
2.04
1.01
1.00
1.02
1.06
1.98
3.05
0.99


NAME 110123
EXPNO 3
PROCNO 1
Date_ 20110223
Time 11.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 380
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677596 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.24
30.16
30.64
30.99
31.42
43.02
43.52
44.28
76.57
77.00
77.42
120.09
120.24
120.40
120.55
126.94
126.98
127.04
127.74
127.85
128.05
128.20
128.31
128.42
128.45
132.18
133.78
138.87
138.93
144.72
144.75
189.12
189.31
O
P(O)allyl2
S
Ph
3l
ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                110123
EXPNO                    2
PROCNO                   1
Date_             20110223
Time                11.02
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       4
SWH              49019.609 Hz
FIDRES           0.747980 Hz
AQ               0.6685172 sec
RG                     203
DW                  10.200 usec
DE                   6.50 usec
TE                  287.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   31P
P1                  9.10 usec
PL1                 0.00 dB
PL1W            36.92473221 W
SFO1            121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                17.00 dB
PL13                17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                   32768
SF              121.4948510 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



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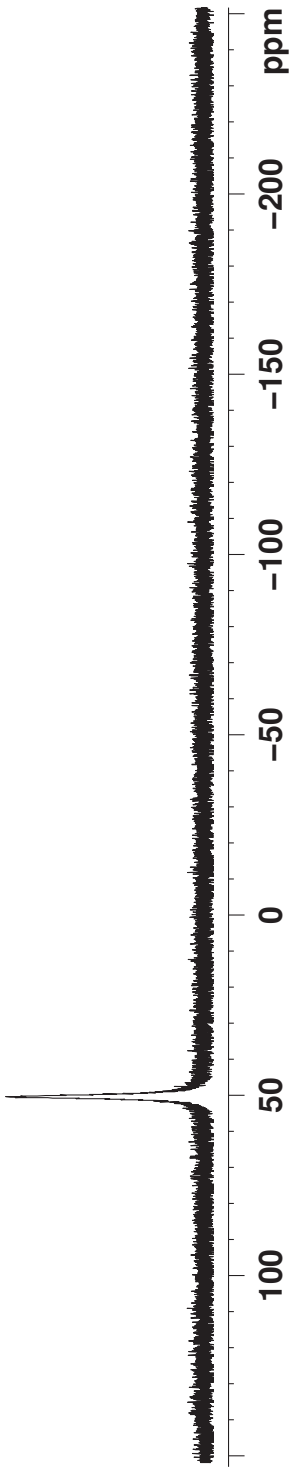
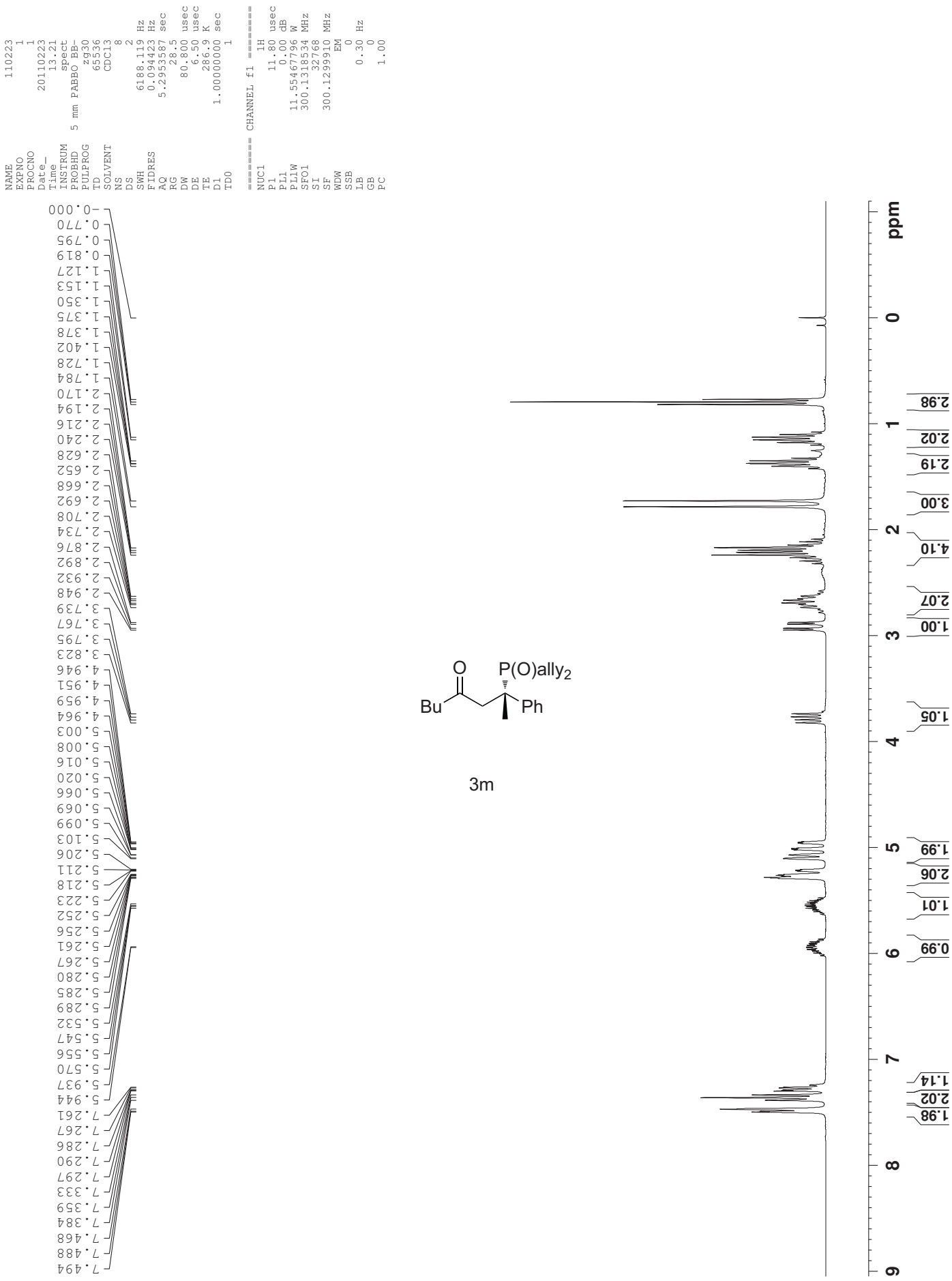
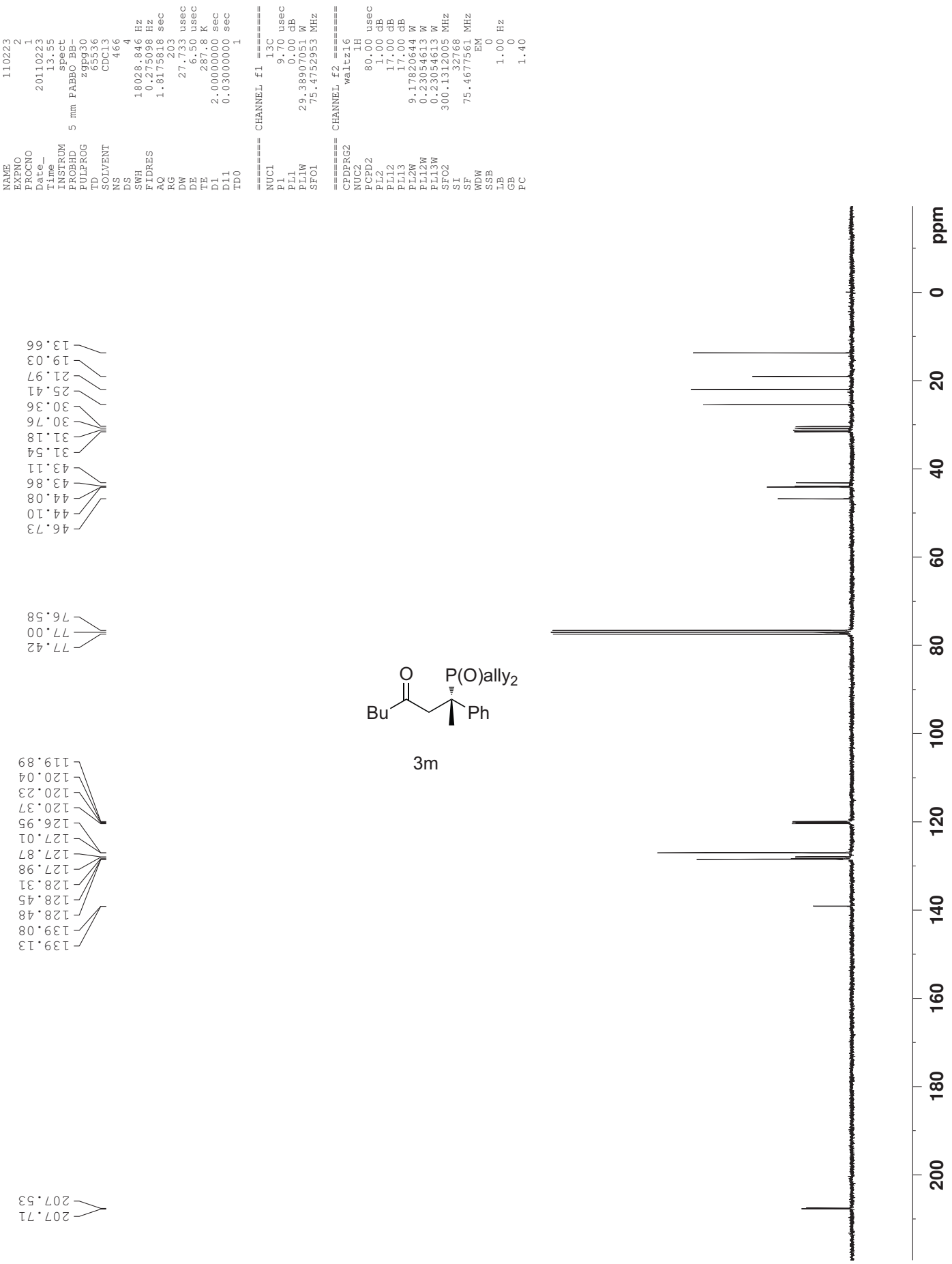


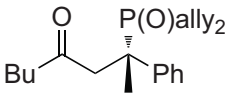

NAME 110223
EXPNO 1
PROCNO 1
Date_ 20110223
Time 13.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 28.5
DW 80.800 usec
DE 6.50 usec
TE 286.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299910 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
2.98
2.02
2.19
3.00
4.10
2.07
1.00
1.05
1.99
2.06
1.01
0.99
1.14
2.02
1.98
O
P(O)ally2
Bu
Ph
3m


NAME 110223
EXPNO 2
PROCNO 1
Date_ 20110223
Time 13.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 466
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677561 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.66
19.03
21.97
25.41
30.36
30.76
31.18
31.54
43.11
43.86
44.08
44.10
46.73
76.58
77.00
77.42
119.89
120.04
120.23
120.37
126.95
127.01
127.87
127.98
128.31
128.45
128.48
139.08
139.13
207.53
207.71
O
P(O)ally2
Bu
Ph
3m
ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                110223
EXPNO                    3
PROCNO                   1
Date_             20110223
Time                 13.59
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                      203
DW                     10.200 usec
DE                      6.50 usec
TE                     287.3 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     31P
P1                      9.10 usec
PL1                     0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     1.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2               300.1312005 MHz
SI                      32768
SF                 121.4948510 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



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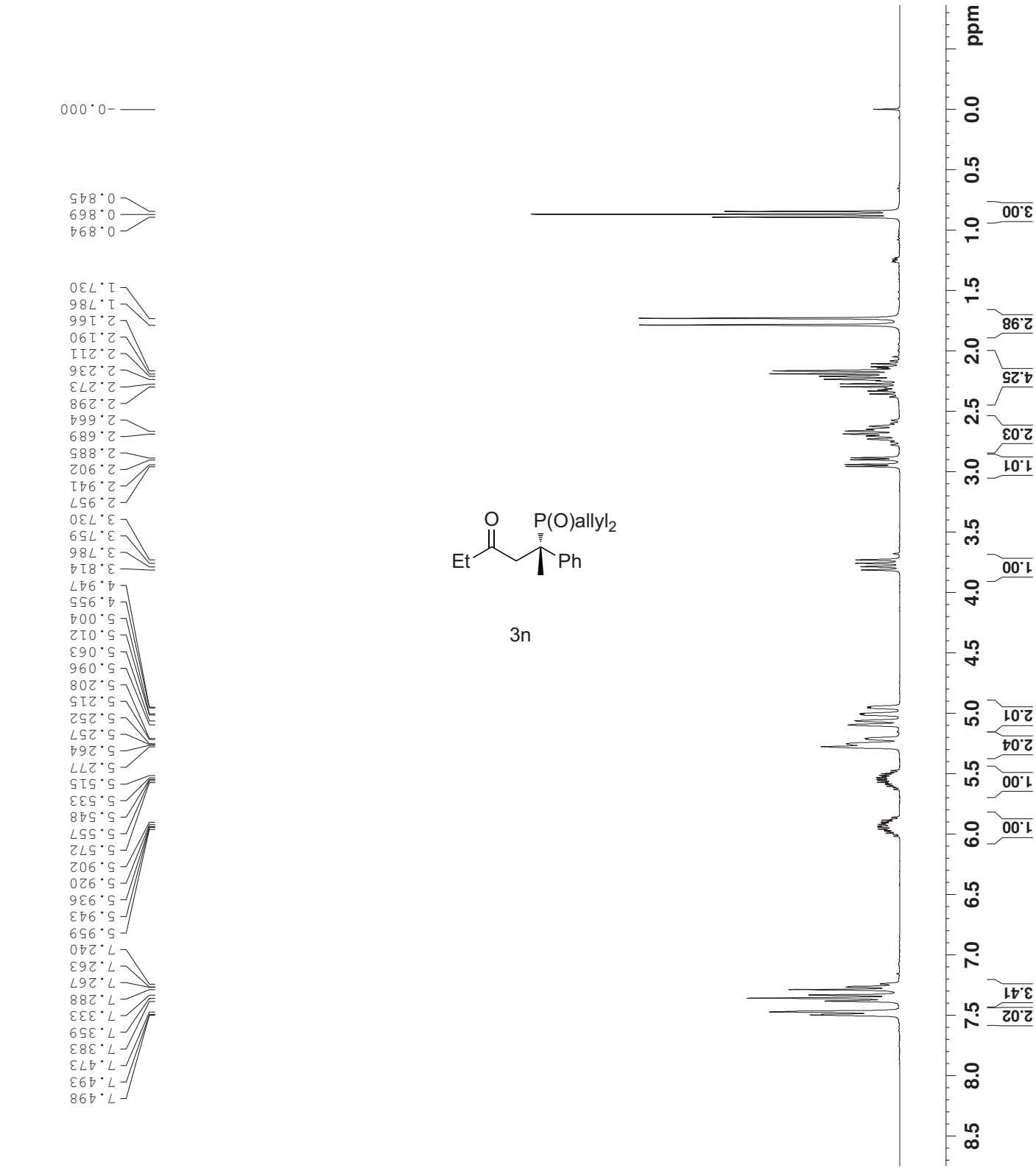
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EXPNO 5
PROCNO 1
Date_ 20110309
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 45.2
DW 80.800 usec
DE 6.50 usec
TE 290.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299937 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.845
0.869
0.894
1.730
1.786
2.166
2.190
2.211
2.236
2.273
2.298
2.664
2.689
2.885
2.902
2.941
2.957
3.730
3.759
3.786
3.814
4.947
4.955
5.004
5.012
5.063
5.096
5.208
5.215
5.252
5.257
5.264
5.277
5.515
5.533
5.548
5.557
5.572
5.902
5.920
5.936
5.943
5.959
7.240
7.263
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7.383
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ppm
3.00
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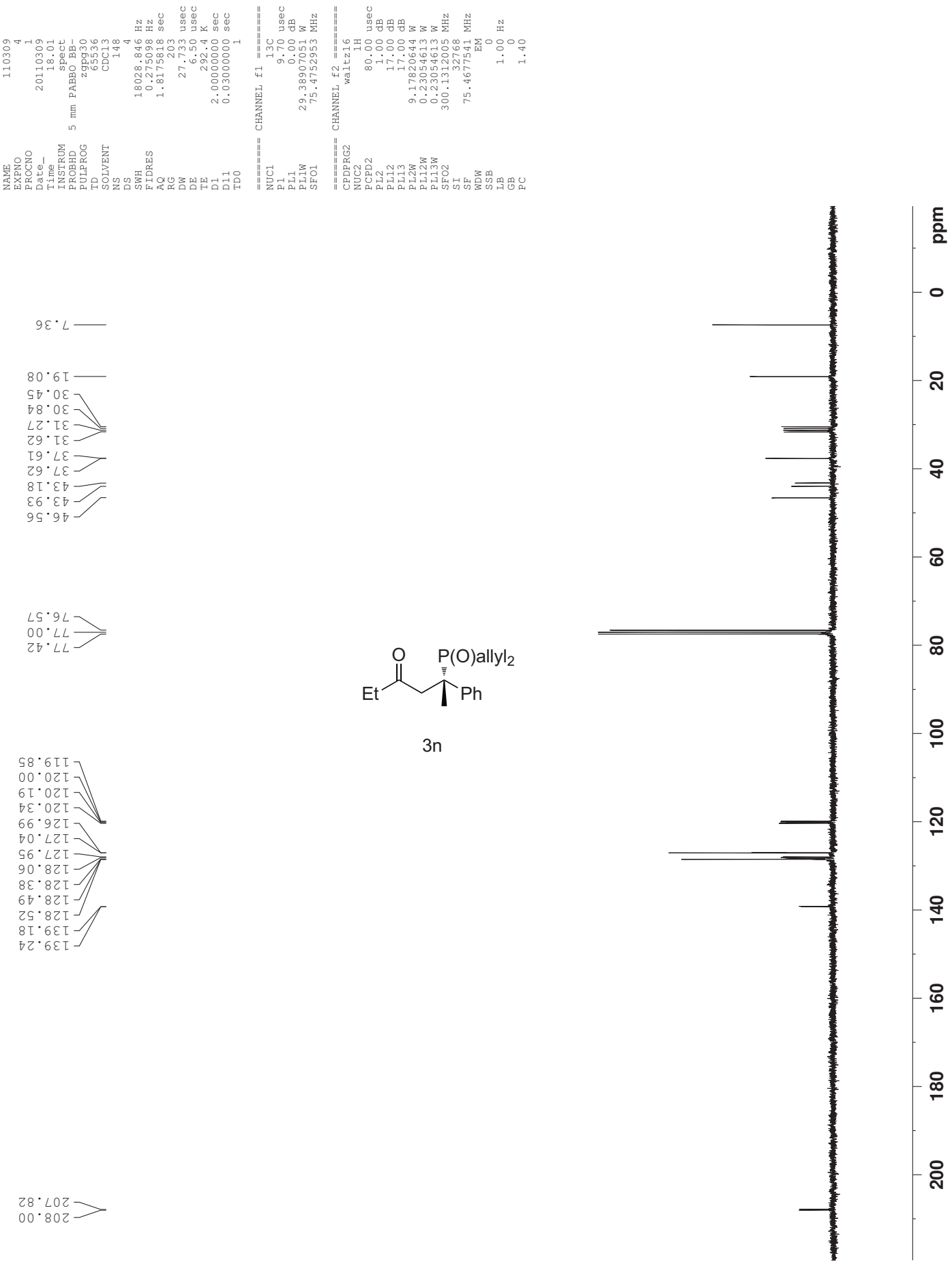
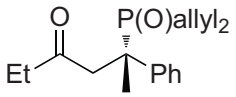
2.03
1.01
1.00
2.01
2.04
1.00
1.00
2.02
3.41

O
P(O)allyl2
Et
Ph

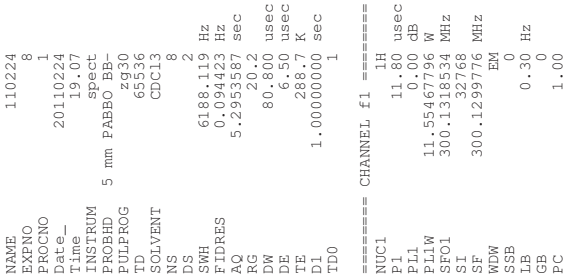
3n

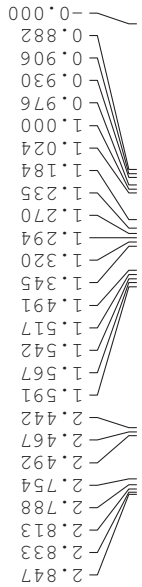

NAME 110309
EXPNO 4
PROCNO 1
Date_ 20110309
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 148
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81775818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
7.36
19.08
30.45
30.84
31.27
31.62
37.61
37.62
43.18
43.93
46.56
76.57
77.00
77.42
119.85
120.00
120.19
120.34
126.99
127.04
127.95
128.06
128.38
128.49
128.52
139.18
139.24
207.82
208.00
O
P(O)allyl2
Et
Ph
3n
ppm
0
20
40
60
80
100
120
140
160
180
200

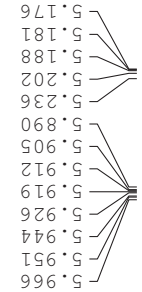
```
NAME                 110309
EXPNO                     6
PROCNO                    1
Date_              20110309
Time                 19.04
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES             0.747980 Hz
AQ                 0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                   290.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     31P
P1                     9.10 usec
PL1                    0.00 dB
PL1W              36.92473221 W
SFO1              121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2              300.1312005 MHz
SI                      32768
SF                121.4948510 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.40
```



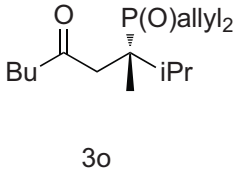
3n


NAME 110224
EXPNO 8
PROCNO 1
Date_ 20110224
Time 19.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 20.2
DW 80.800 usec
DE 6.50 usec
TE 288.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299776 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

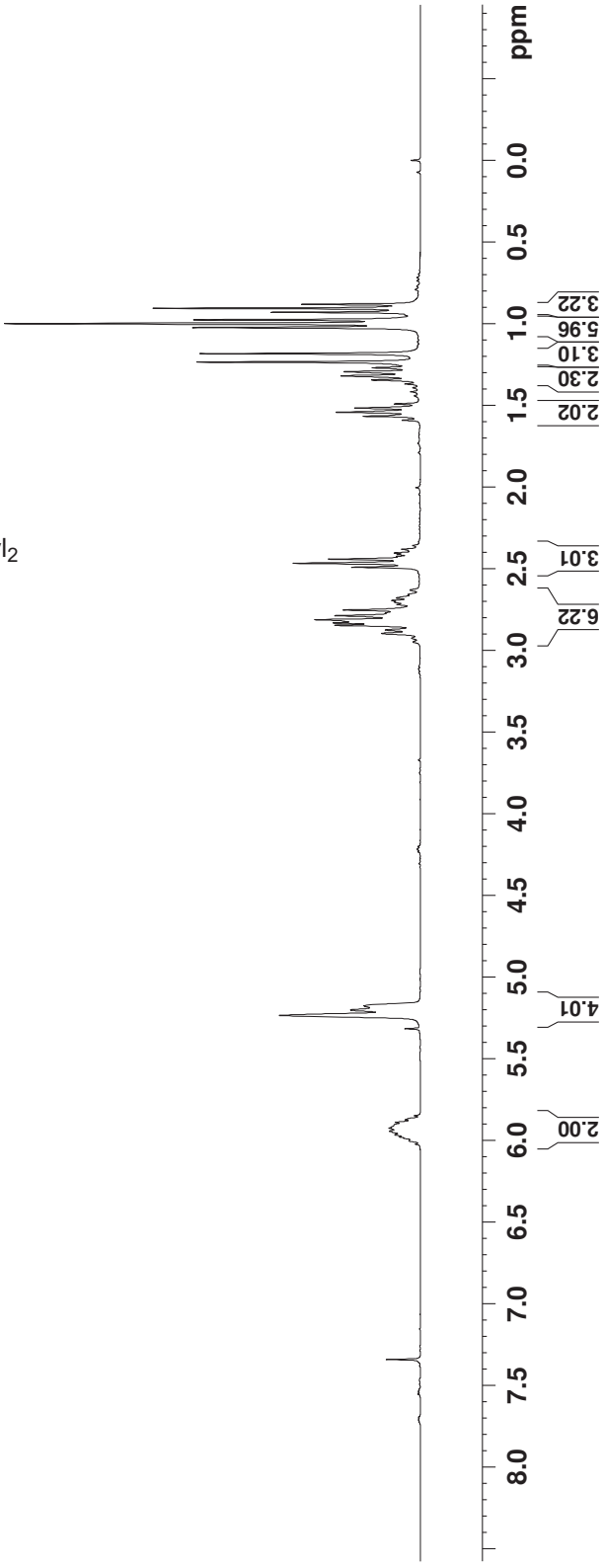

0.000
0.882
0.906
0.930
0.976
1.000
1.024
1.184
1.235
1.270
1.294
1.320
1.345
1.491
1.517
1.542
1.567
1.591
2.442
2.467
2.492
2.754
2.788
2.813
2.833
2.847

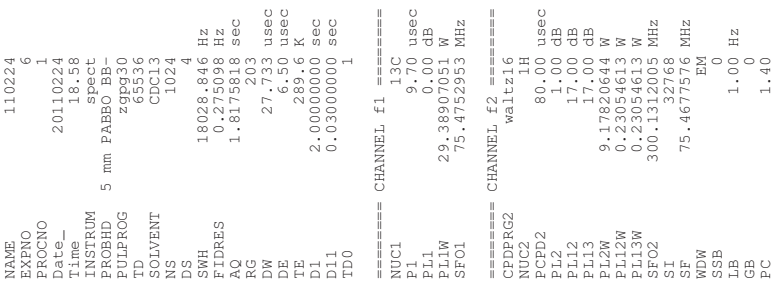

5.176
5.181
5.188
5.202
5.236
5.890
5.905
5.912
5.919
5.926
5.944
5.951
5.966

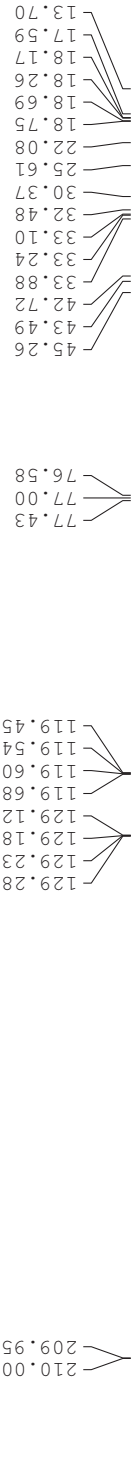
7.342

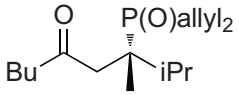

O
P(O)allyl2
Bu
iPr

3o

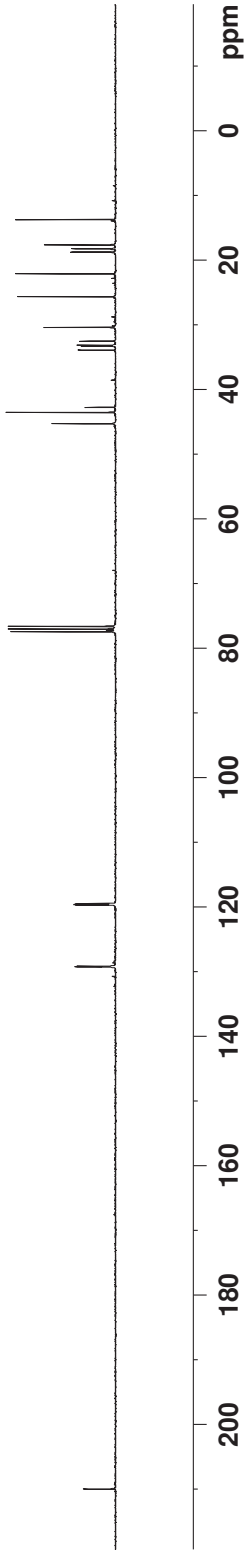

ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
3.22
5.96
3.10
2.30
2.02
3.01
6.22
4.01
2.00


NAME 110224
EXPNO 6
PROCNO 1
Date_ 20110224
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81775818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677576 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

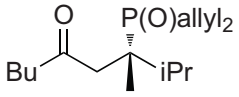

13.70
17.59
18.17
18.26
18.69
18.75
22.08
25.61
30.37
32.48
33.10
33.24
33.88
42.72
43.49
45.26
76.58
77.00
77.43
119.45
119.54
119.60
119.68
129.12
129.18
129.23
129.28
209.95
210.00


O
P(O)allyl2
Bu
iPr

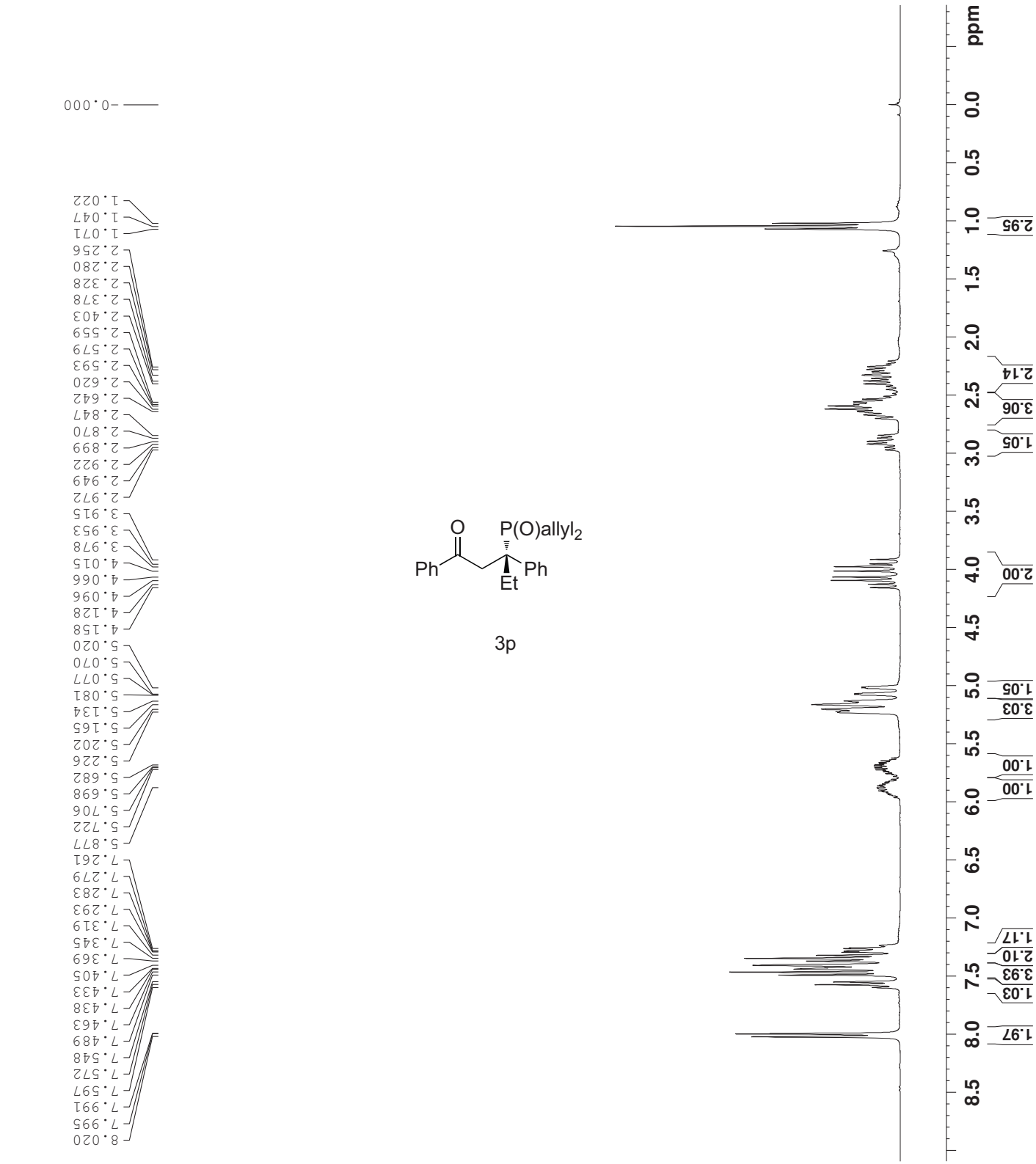
3o

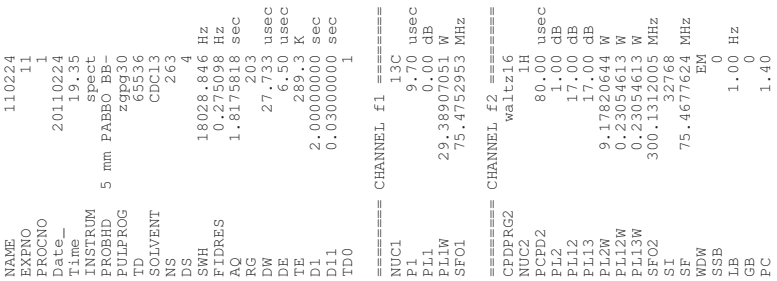

ppm
0
20
40
60
80
100
120
140
160
180
200

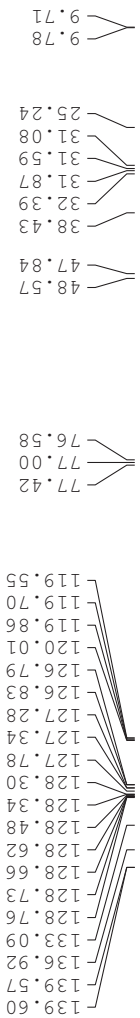
```
NAME                 110224
EXPNO                     7
PROCNO                    1
Date_              20110224
Time                 19.04
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                         16
DS                          4
SWH                 49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                        203
DW                     10.200 usec
DE                      6.50 usec
TE                     288.9 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                  80.00 usec
PL2                     1.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



3o


NAME 110224
EXPNO 9
PROCNO 1
Date_ 20110224
Time_ 19.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 22.6
DW 80.800 usec
DE 6.50 usec
TE 288.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299923 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.022
1.047
1.071
2.256
2.280
2.328
2.378
2.403
2.559
2.579
2.593
2.620
2.642
2.847
2.870
2.899
2.922
2.949
2.972
3.915
3.953
3.978
4.015
4.066
4.096
4.128
4.158
5.020
5.070
5.077
5.081
5.134
5.165
5.202
5.226
5.682
5.698
5.706
5.722
5.877
7.261
7.279
7.283
7.293
7.319
7.345
7.369
7.405
7.433
7.438
7.463
7.489
7.548
7.572
7.597
7.991
7.995
8.020
O
P(O)allyl2
Ph
Ph
Et
3p
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
2.95
2.14
3.06
1.05
2.00
1.05
3.03
1.00
1.00
1.17
2.10
3.93
1.03
1.97


NAME 110224
EXPNO 11
PROCNO 1
Date_ 20110224
Time_ 19.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 263
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81775818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677624 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


9.71
9.78
25.24
31.08
31.59
31.87
32.39
38.43
47.84
48.57
76.58
77.00
77.42
119.55
119.70
119.86
120.01
126.79
126.83
127.28
127.34
127.78
128.30
128.34
128.48
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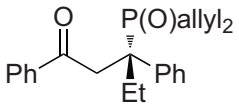
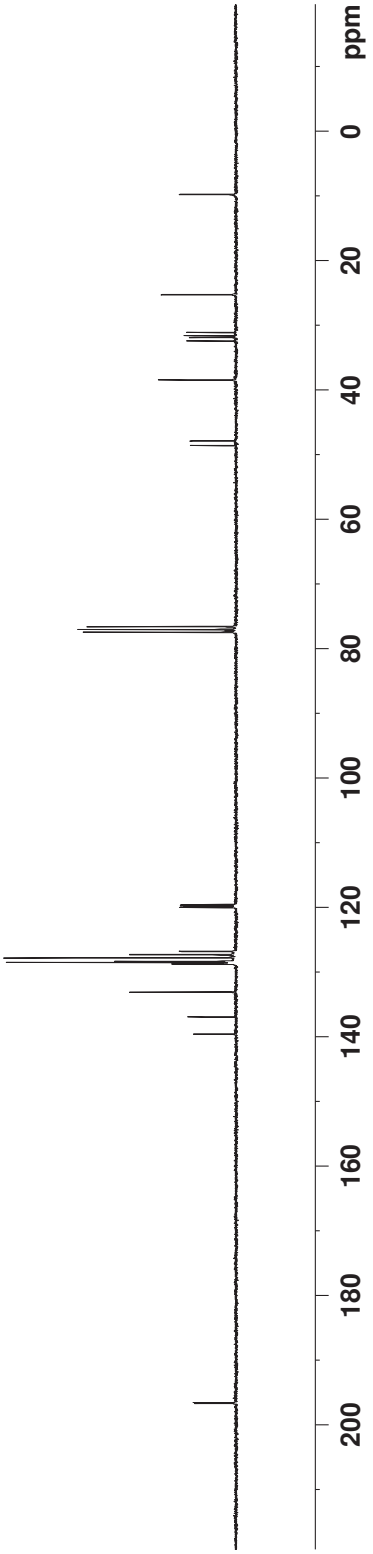
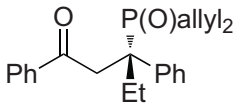
128.66
128.73
128.76
133.09
136.92
139.57
139.60
196.57
196.71


O
P(O)allyl2
Ph
Ph
Et

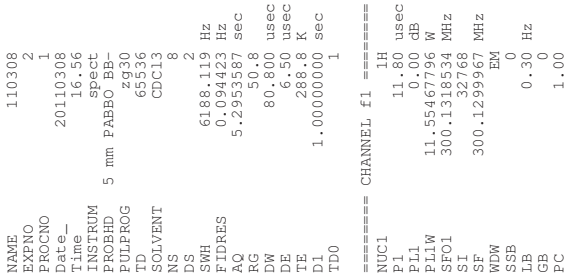
3p

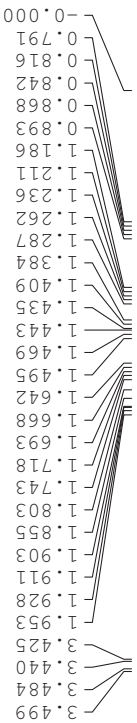

ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                110224
EXPNO                   10
PROCNO                   1
Date_             20110224
Time                19.16
INSTRUM              spect
PROBHD  5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                      203
DW                     10.200 usec
DE                      6.50 usec
TE                     288.7 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                  36.92473221 W
SFO1                  121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.00 dB
PL12                     17.00 dB
PL13                     17.00 dB
PL2W                  9.17820644 W
PL12W                 0.23054613 W
PL13W                 0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



3p


NAME 110308
EXPNO 2
PROCNO 1
Date_ 20110308
Time_ 16.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 50.8
DW 80.800 usec
DE 6.50 usec
TE 288.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299967 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00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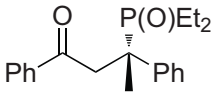
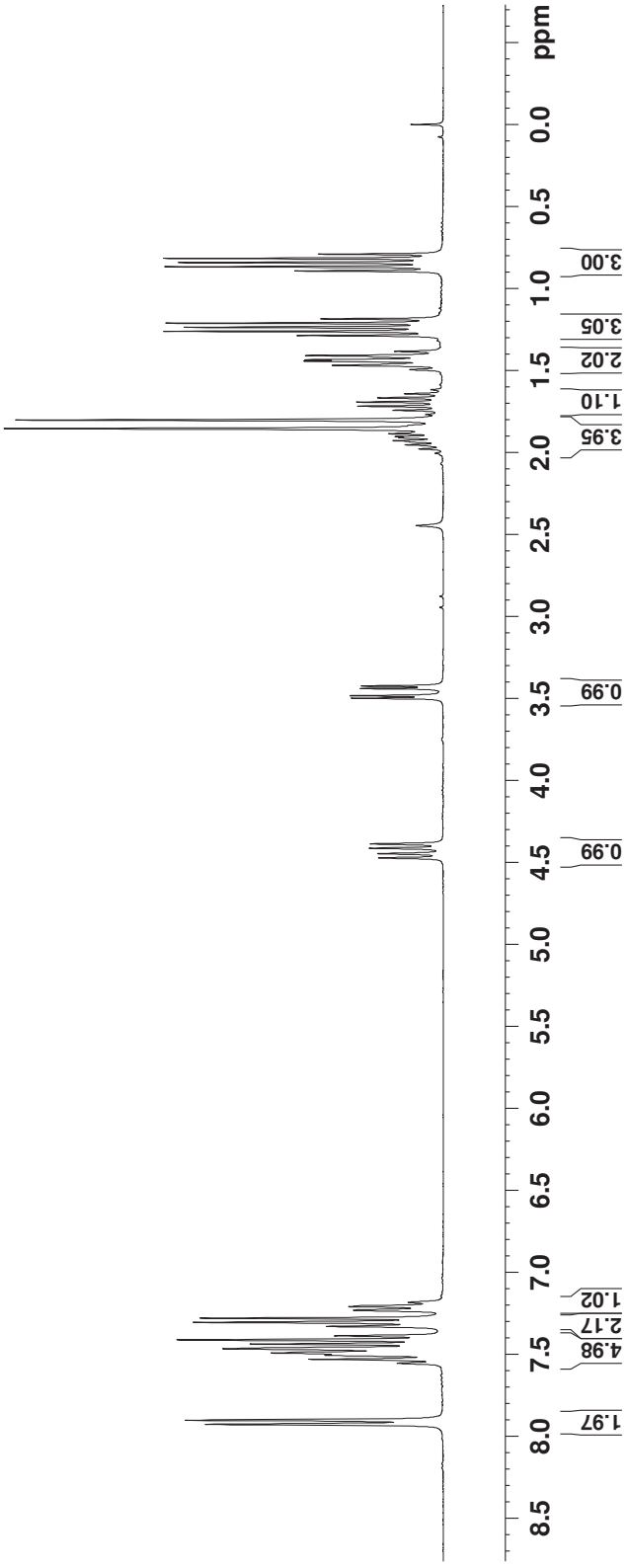
1.211
1.236
1.262
1.287
1.384
1.409
1.435
1.443
1.469
1.495
1.642
1.668
1.693
1.718
1.743
1.803
1.855
1.903
1.911
1.928
1.953
3.425
3.440
3.484
3.499
4.233
4.388
4.415
4.447
4.474
7.182
7.207
7.229
7.278
7.303
7.328
7.385
7.410
7.436
7.465
7.490
7.503
7.529
7.553
7.900
7.925


O
P(O)Et2
Ph
Ph

3q


ppm
0.0
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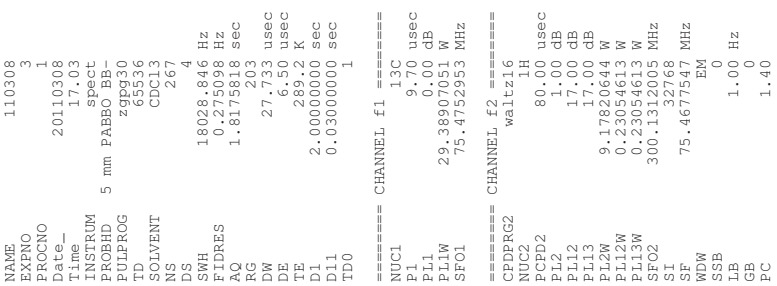
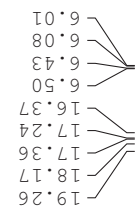
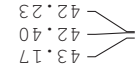
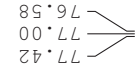
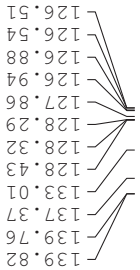
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.00
3.05
2.02
1.10
3.95
0.99
0.99
1.02
2.17
4.98
1.97

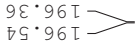

110308
NAME
3
EXPNO
1
PROCNO
20110308
Date_
17.03
Time
spect
INSTRUM
5 mm PABBO BB-
PROBHD
zgpg30
PULPROG
65536
TD
CDCl3
SOLVENT
267
NS
4
DS
18028.846 Hz
SWH
0.275098 Hz
FIDRES
1.81175818 sec
AQ
203
RG
27.733 usec
DW
6.50 usec
DE
289.2 K
TE
2.00000000 sec
D1
0.03000000 sec
D11
1
TD0
======== CHANNEL f1 ========
13C
NUC1
9.70 usec
P1
0.00 dB
PL1
29.38907051 W
PLW1
75.4752953 MHz
SFO1
======== CHANNEL f2 ========
waltz16
CPDPRG2
1H
NUC2
80.00 usec
PCPD2
1.00 dB
PL2
17.00 dB
PL12
17.00 dB
PL13
9.17820644 W
PL2W
0.23054613 W
PL12W
0.23054613 W
PL13W
300.1312005 MHz
SFO2
32768
SI
75.4677547 MHz
SF
EM
WDW
0
SSB
1.00 Hz
LB
0
GB
1.40
PC

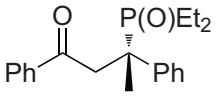

19.26
18.17
17.36
17.24
16.37
6.50
6.43
6.08
6.01


43.17
42.40
42.23

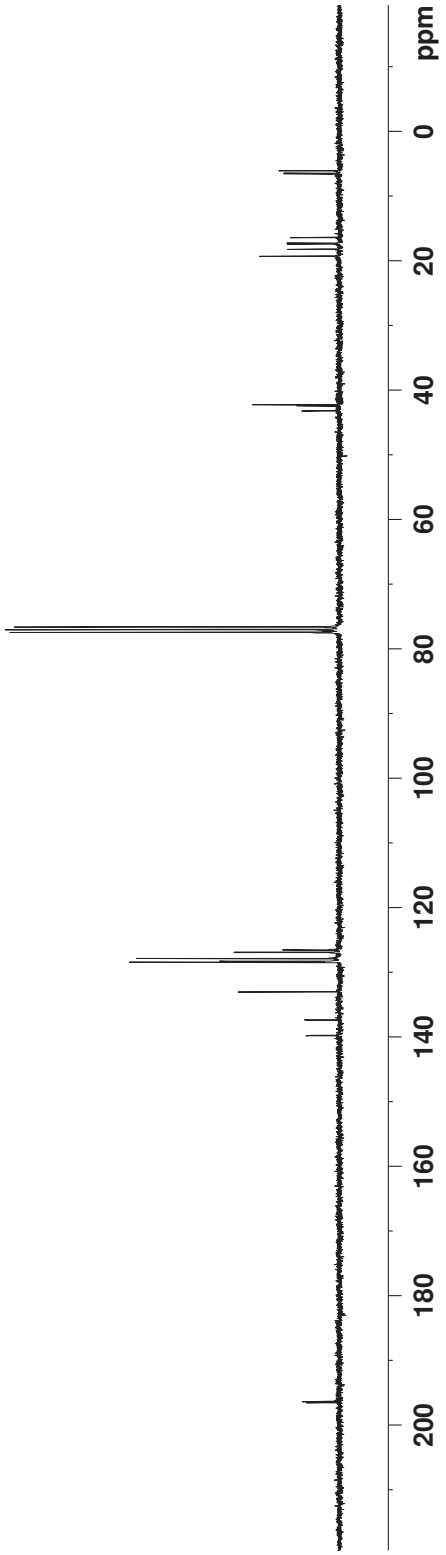

77.42
77.00
76.58


139.82
139.76
137.37
133.01
128.43
128.32
128.29
127.86
126.94
126.88
126.54
126.51

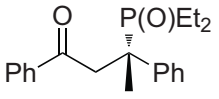

196.54
196.36


O
P(O)Et2
Ph
Ph

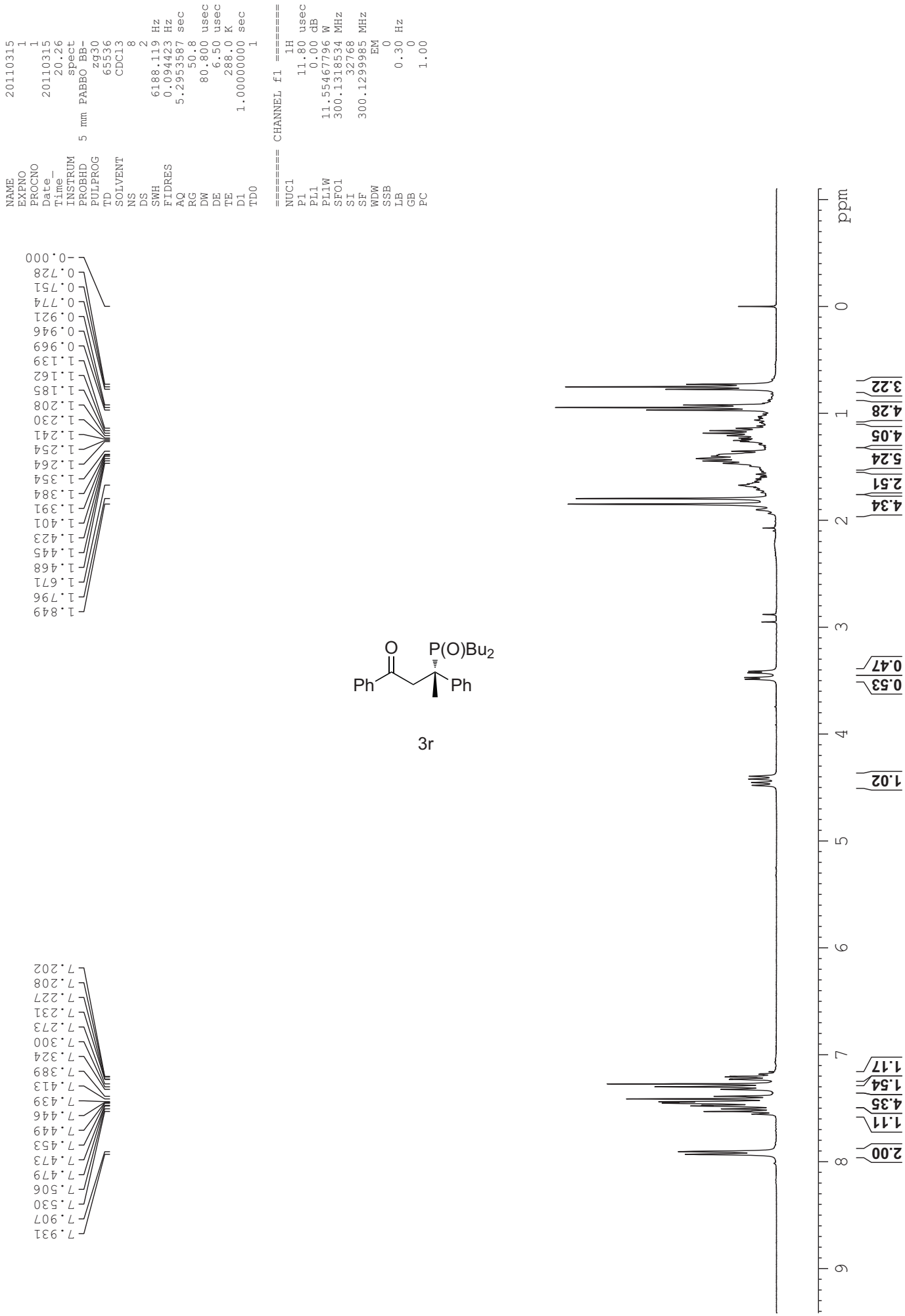
3q

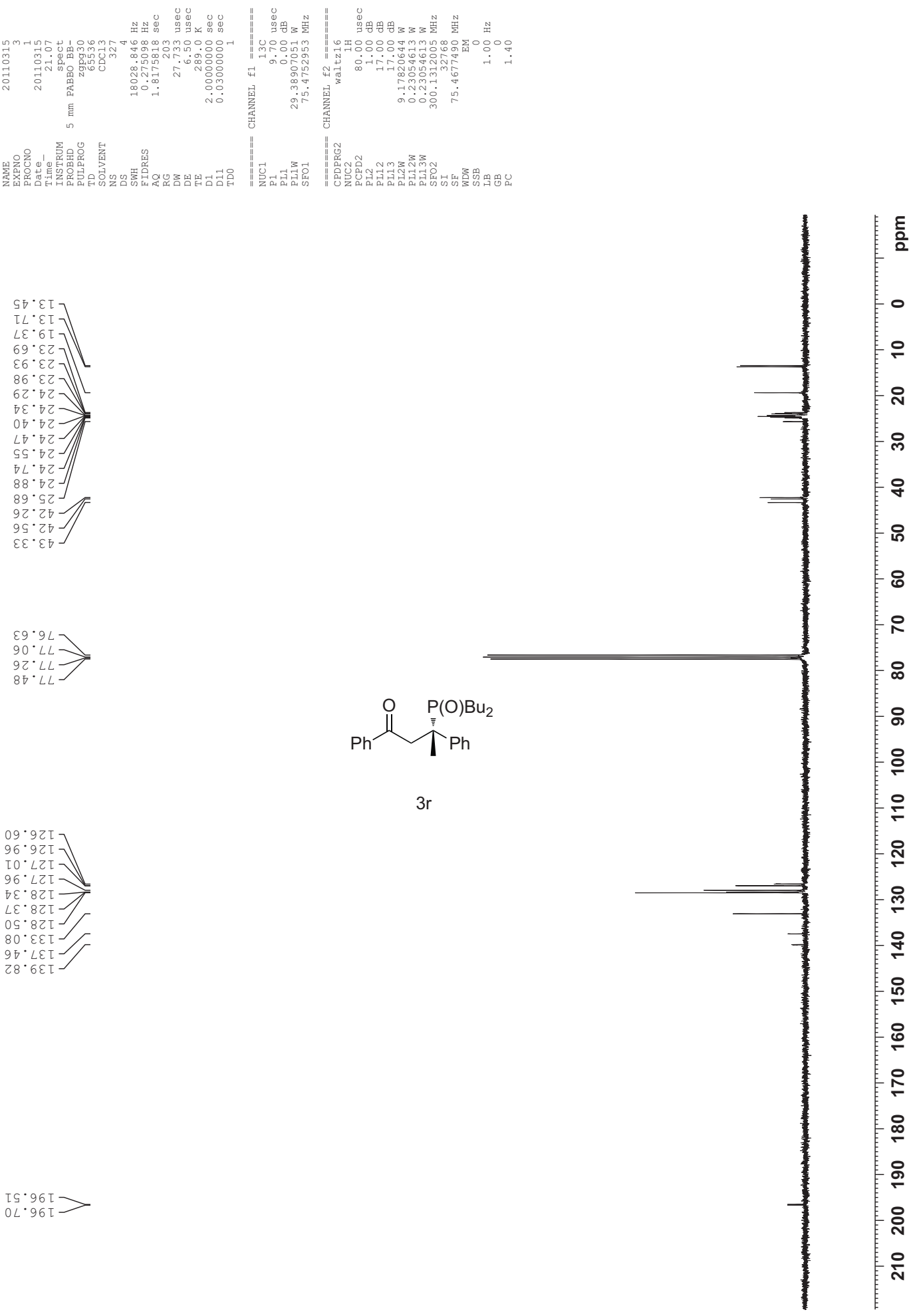

ppm
0
20
40
60
80
100
120
140
160
180
200

```
NAME                110308
EXPNO                    1
PROCNO                   1
Date_             20110308
Time                 16.54
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                      203
DW                     10.200 usec
DE                      6.50 usec
TE                    289.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    31P
P1                     9.10 usec
PL1                    0.00 dB
PL1W                36.92473221 W
SFO1               121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2              300.1312005 MHz
SI                      32768
SF                121.4948510 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.40
```

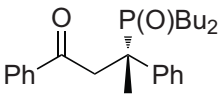


3q

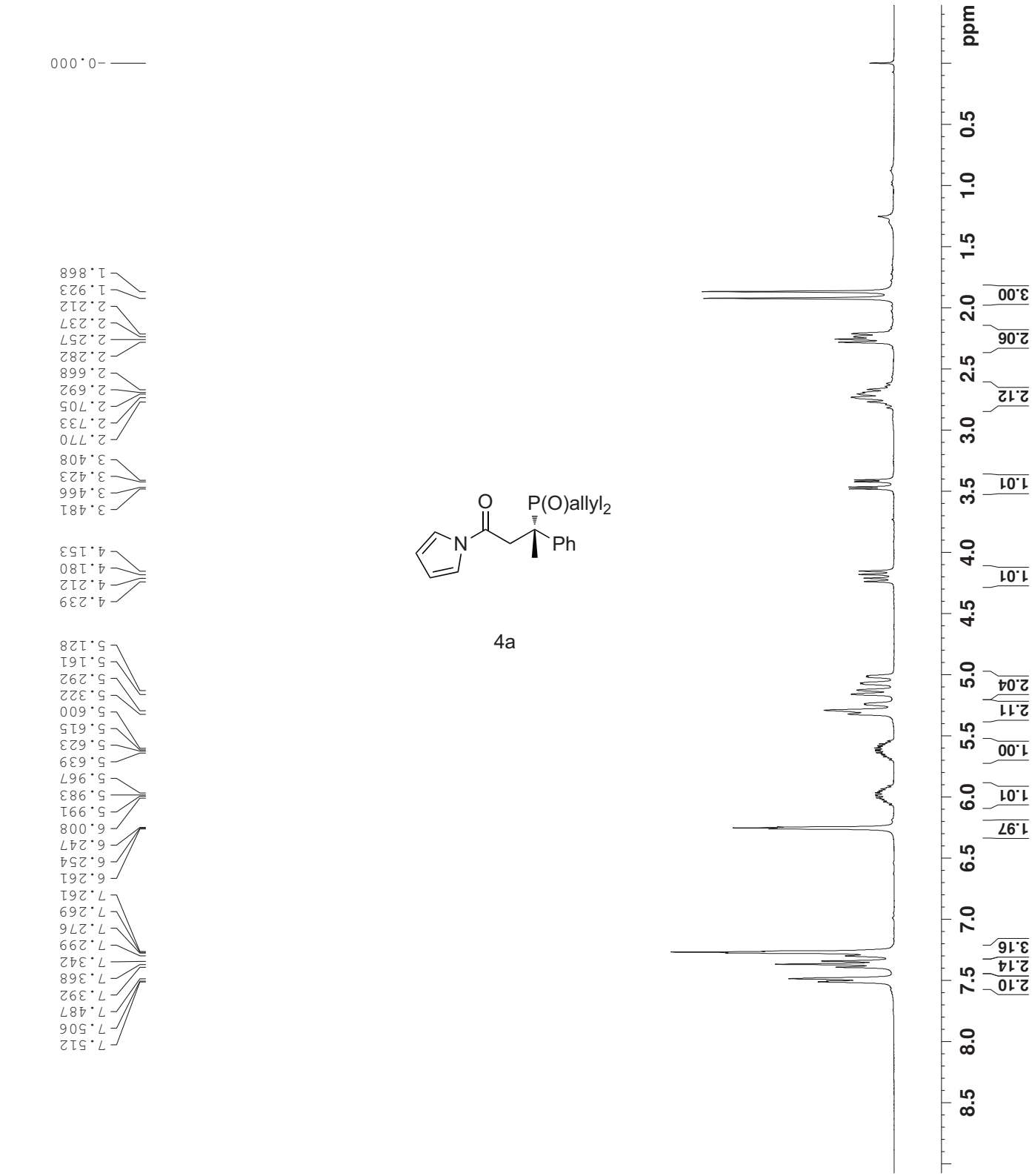

NAME 20110315
EXPNO 1
PROCNO 1
Date_ 20110315
Time 20.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 50.8
DW 80.800 usec
DE 6.50 usec
TE 288.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299985 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.22
4.28
4.05
5.24
2.51
4.34
0.53
0.47
1.02
1.17
1.54
4.35
1.11
2.00
O
P(O)Bu2
Ph
Ph
3r


NAME 20110315
EXPNO 3
PROCNO 1
Date_ 20110315
Time_ 21.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 327
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.45
13.71
19.37
23.69
23.93
23.98
24.29
24.34
24.40
24.47
24.55
24.74
24.88
25.68
42.26
42.56
43.33
76.63
77.06
77.26
77.48
126.60
126.96
127.01
127.96
128.34
128.37
128.50
133.08
137.46
139.82
196.70
196.51
O
P(O)Bu2
Ph
Ph
3r
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

```
NAME                  20110315
EXPNO                        2
PROCNO                       1
Date_              20110315
Time_                  20.29
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                         203
DW                      10.200 usec
DE                       6.50 usec
TE                      288.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                 36.92473221 W
SFO1                 121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      1.00 dB
PL12                     17.00 dB
PL13                     17.00 dB
PL2W                  9.17820644 W
PL12W                 0.23054613 W
PL13W                 0.23054613 W
SFO2                 300.1312005 MHz
SI                       32768
SF                   121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

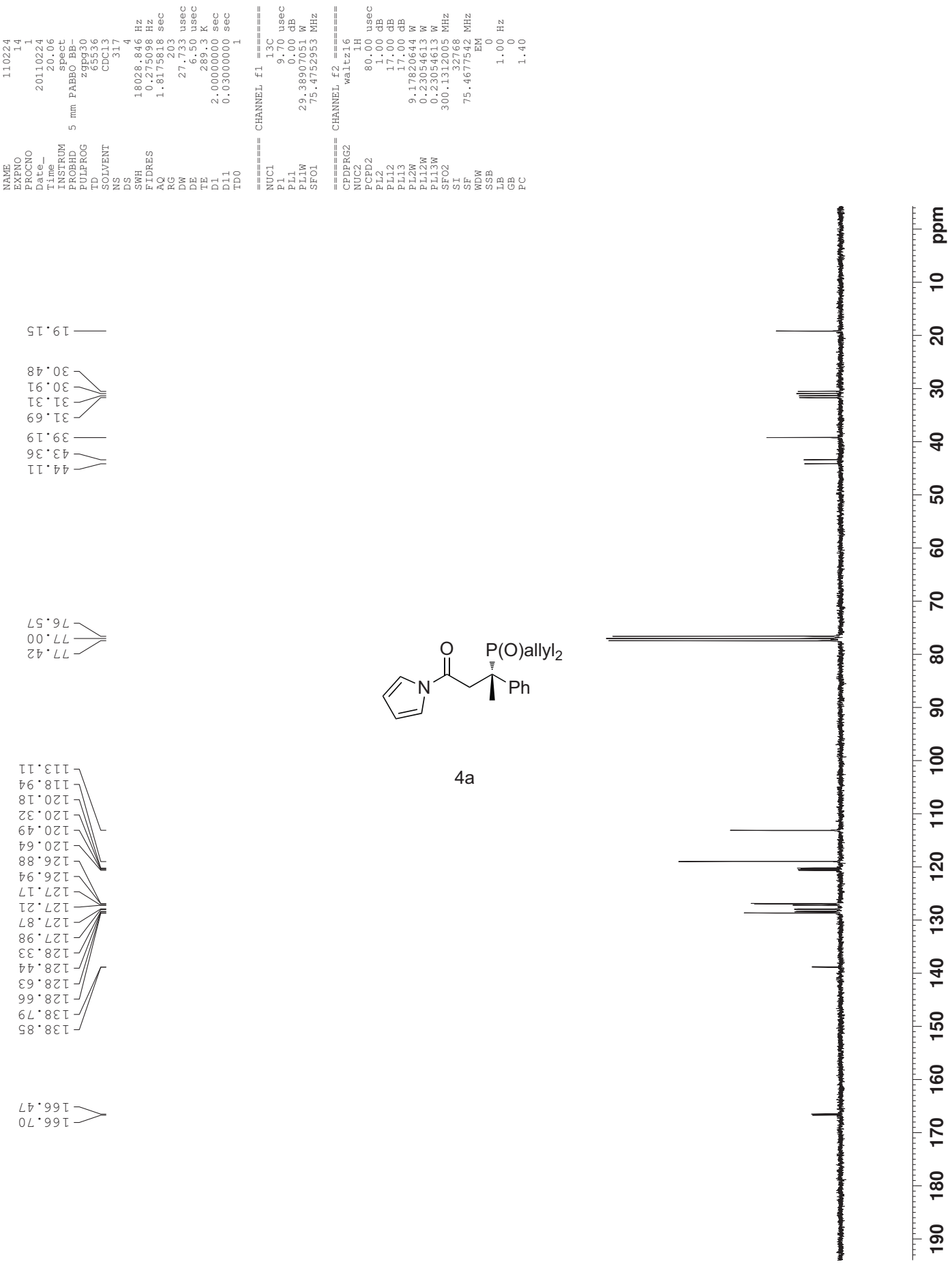


3r

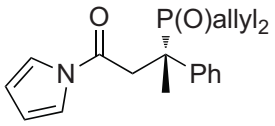

NAME 110224
EXPNO 12
PROCNO 1
Date_ 20110224
Time 19.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 288.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.00
2.06
2.12
1.01
1.01
2.04
2.11
1.00
1.01
1.97
3.16
2.14
2.10

O
P(O)allyl2
N
Ph

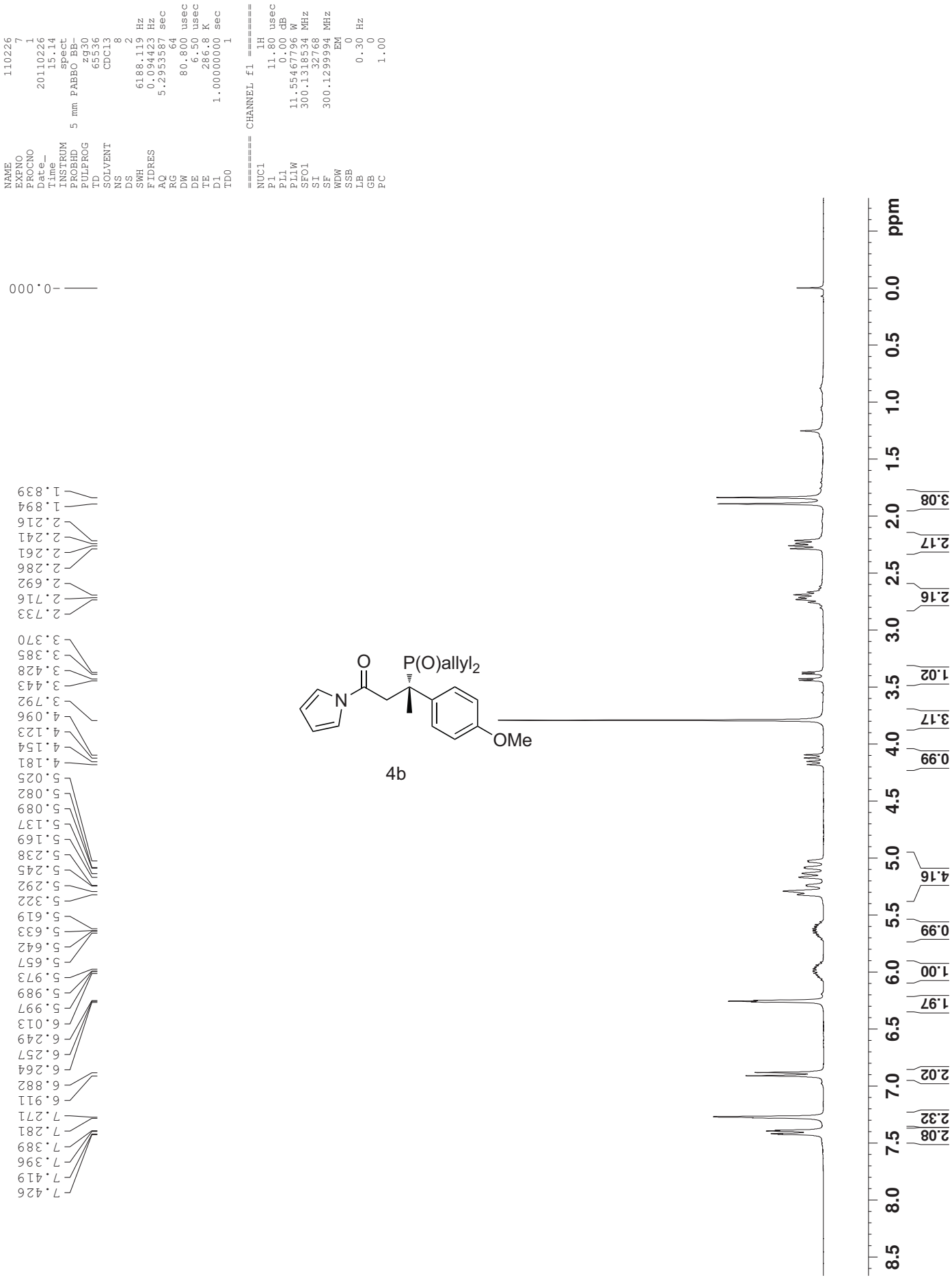
4a


NAME 110224
EXPNO 14
PROCNO 1
Date_ 20110224
Time_ 20.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 317
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.15
30.48
30.91
31.31
31.69
39.19
43.36
44.11
76.57
77.00
77.42
113.11
118.94
120.18
120.32
120.49
120.64
126.88
126.94
127.17
127.21
127.87
127.98
128.33
128.44
128.63
128.66
138.79
138.85
166.47
166.70
O
P(O)allyl2
N
Ph
4a
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

```
NAME                110224
EXPNO                   13
PROCNO                   1
Date_             20110224
Time                 19.44
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                      10.200 usec
DE                       6.50 usec
TE                     288.7 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                  36.92473221 W
SFO1                  121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       1.00 dB
PL12                     17.00 dB
PL13                     17.00 dB
PL2W                  9.17820644 W
PL12W                 0.23054613 W
PL13W                 0.23054613 W
SFO2                  300.1312005 MHz
SI                         32768
SF                    121.4948510 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

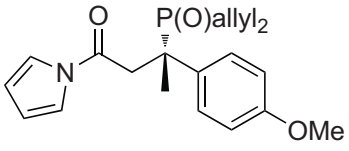


4a

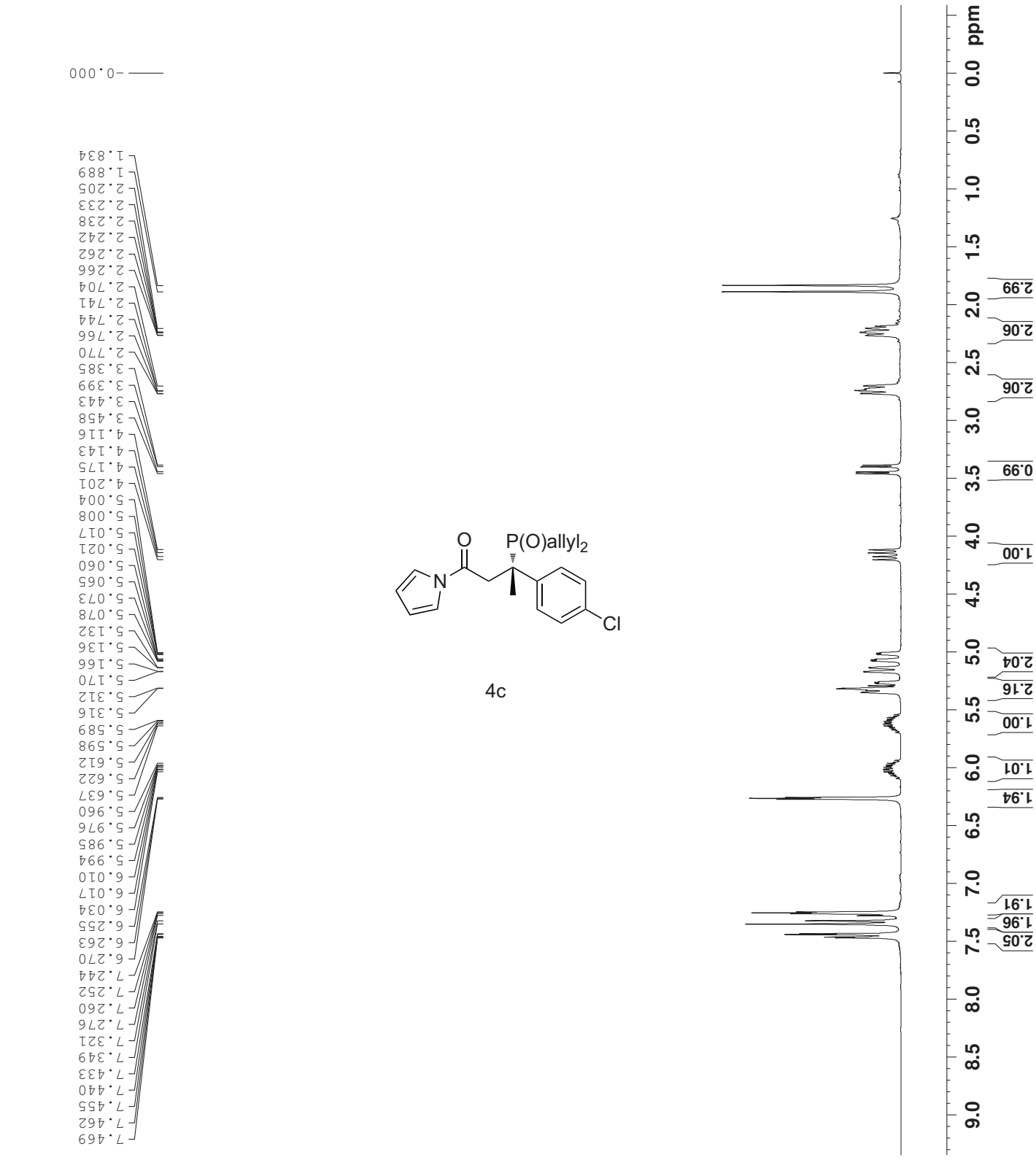

110226
7
1
20110226
15.14
spect
5 mm PABBO BB-
zg30
65536
CDCl3
8
2
6188.119 Hz
0.094423 Hz
5.2955587 sec
64
80.800 usec
6.50 usec
286.8 K
1.00000000 sec
1
======== CHANNEL f1 ========
1H
11.80 usec
0.00 dB
11.55467796 W
300.1318534 MHz
32768
300.1299994 MHz
EM
0
0.30 Hz
0
1.00
NAME
EXPNO
PROCNO
Date_
Time_
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
TD0
NUC1
P1
PL1
PL1W
SFO1
SI
SF
WDW
SSB
LB
GB
PC
O
P(O)allyl2
N
OMe
4b
ppm
3.08
2.17
2.16
1.02
3.17
0.99
4.16
0.99
1.00
1.97
2.02
2.32
2.08

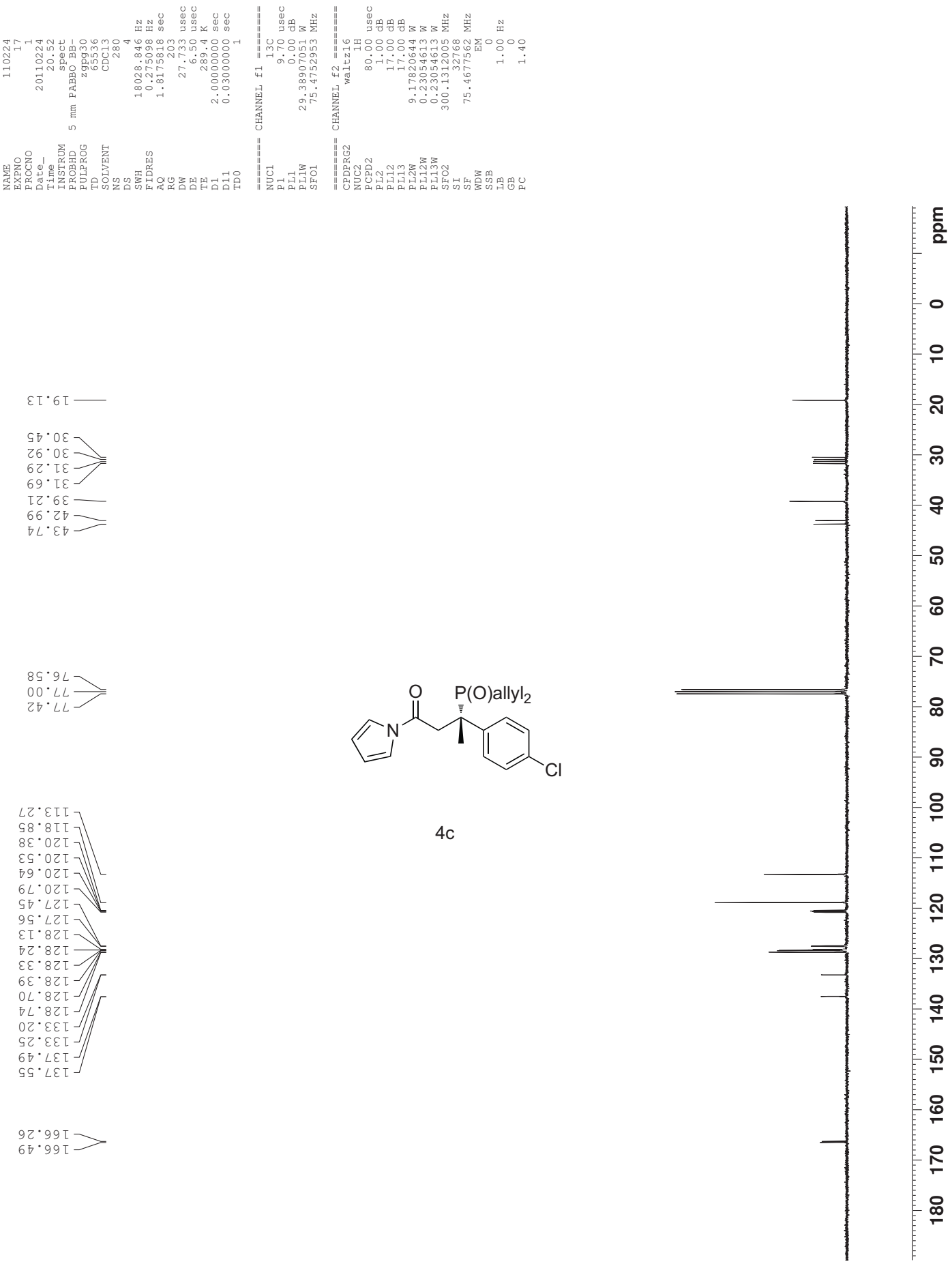

NAME 110226
EXPNO 8
PROCNO 1
Date_ 20110226
Time 15.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 454
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.26
30.48
30.92
31.32
31.68
39.13
42.71
43.48
55.14
76.58
77.00
77.43
113.12
114.00
114.04
118.98
120.15
120.30
120.44
120.59
127.99
128.03
128.09
128.43
128.54
130.55
130.61
158.55
158.51
166.61
166.83
O
P(O)allyl2
N
OMe
4b
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

```
NAME                 110226
EXPNO                     9
PROCNO                    1
Date_              20110226
Time                 15.50
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     287.5 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                     9.10 usec
PL1                    0.00 dB
PL1W              36.92473221 W
SFO1              121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2              300.1312005 MHz
SI                      32768
SF                121.4948510 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

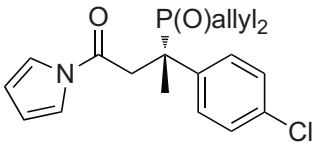


4b

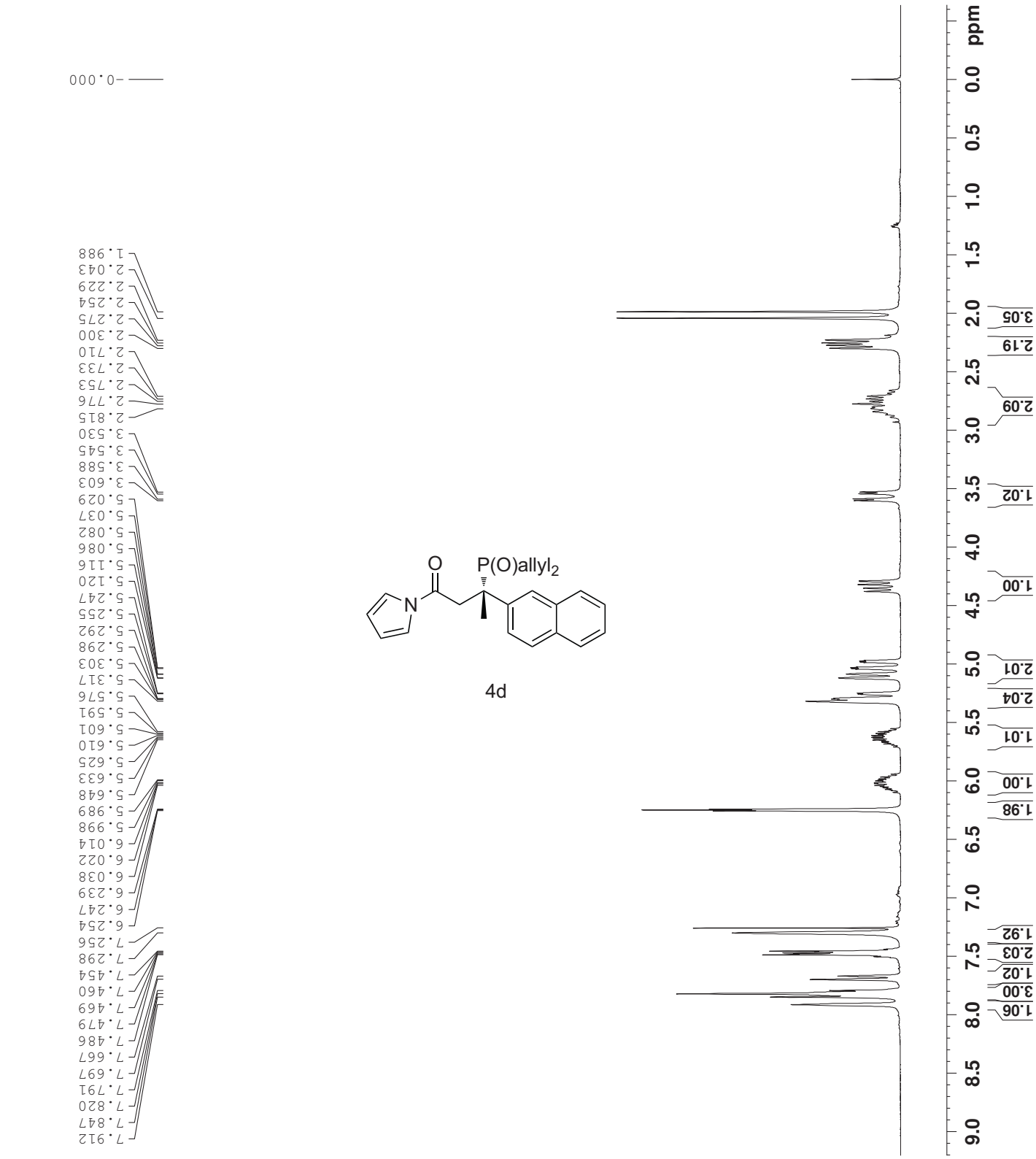

NAME 110224
EXPNO 15
PROCNO 1
Date_ 20110224
Time 20.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 45.2
DW 80.800 usec
DE 6.50 usec
TE 288.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299974 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.000
1.834
1.889
2.205
2.233
2.238
2.242
2.262
2.266
2.704
2.741
2.744
2.766
2.770
3.385
3.399
3.443
3.458
4.116
4.143
4.175
4.201
5.004
5.008
5.017
5.021
5.060
5.065
5.073
5.078
5.132
5.136
5.166
5.170
5.312
5.316
5.589
5.598
5.612
5.622
5.637
5.960
5.976
5.985
5.994
6.010
6.017
6.034
6.255
6.263
6.270
7.244
7.252
7.260
7.276
7.321
7.349
7.433
7.440
7.455
7.462
7.469
O
P(O)allyl2
N
Cl
4c
ppm
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
2.99
2.06
2.06
0.99
1.00
2.04
2.16
1.00
1.01
1.94
1.91
1.96
2.05


NAME 110224
EXPNO 17
PROCNO 1
Date_ 20110224
Time_ 20.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 280
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.13
30.45
30.92
31.29
31.69
39.21
42.99
43.74
76.58
77.00
77.42
113.27
118.85
120.38
120.53
120.64
120.79
127.45
127.56
128.13
128.24
128.33
128.39
128.70
128.74
133.20
133.25
137.49
137.55
166.26
166.49
O
P(O)allyl2
N
Cl
4c
ppm
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

```
NAME                 110224
EXPNO                    16
PROCNO                    1
Date_              20110224
Time                  20.36
INSTRUM                spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     289.1 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      31P
P1                     9.10 usec
PL1                    0.00 dB
PL1W              36.92473221 W
SFO1              121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2              300.1312005 MHz
SI                      32768
SF                121.4948510 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



4c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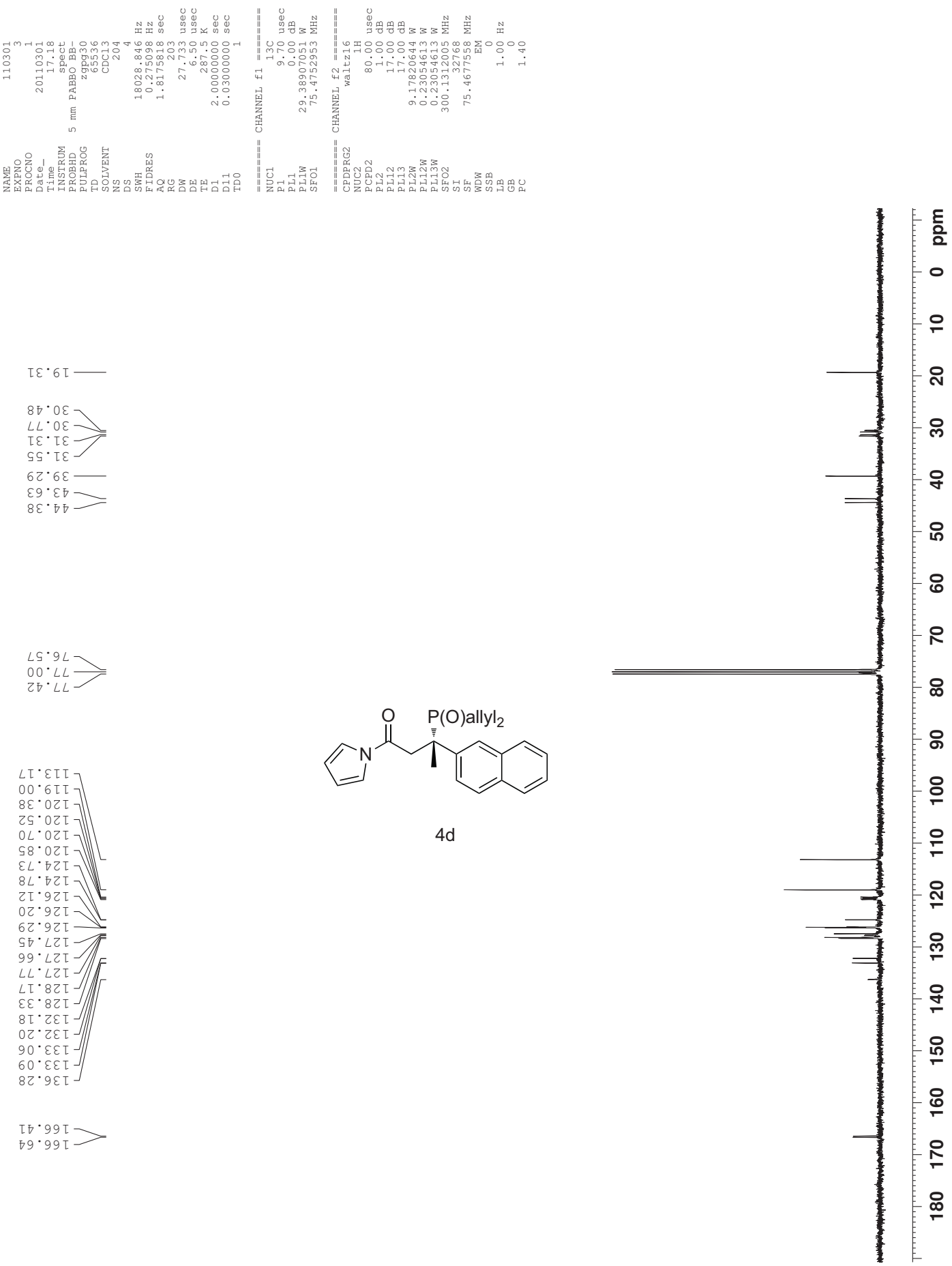
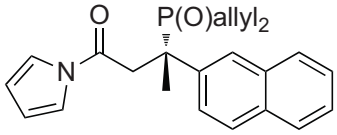
EXPNO 1
PROCNO 1
Date_ 20110301
Time 17.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 45.2
DW 80.800 usec
DE 6.50 usec
TE 286.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.05
2.19
2.09
1.02
1.00
2.01
2.04
1.01
1.00
1.98
1.92
2.03
1.02
3.00
1.06

O
P(O)allyl2
N

4d

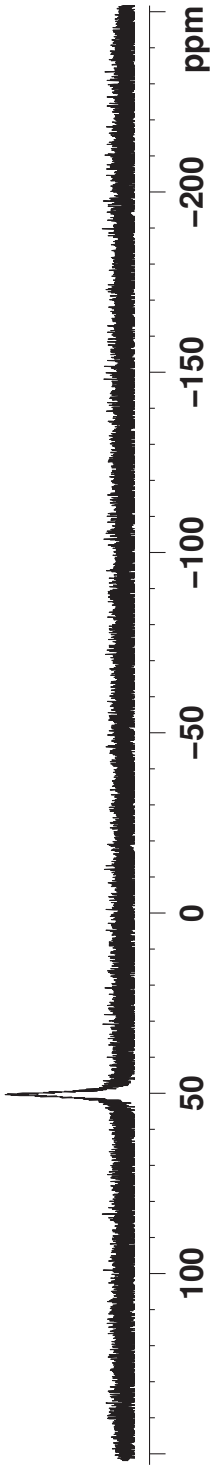

NAME 110301
EXPNO 3
PROCNO 1
Date_ 20110301
Time_ 17.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 204
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677558 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.31
30.48
30.77
31.31
31.55
39.29
43.63
44.38
76.57
77.00
77.42
113.17
119.00
120.38
120.52
120.70
120.85
124.73
124.78
126.12
126.20
126.29
127.45
127.66
127.77
128.17
128.33
132.18
132.20
133.06
133.09
136.28
166.41
166.64
O
P(O)allyl2
N
4d
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

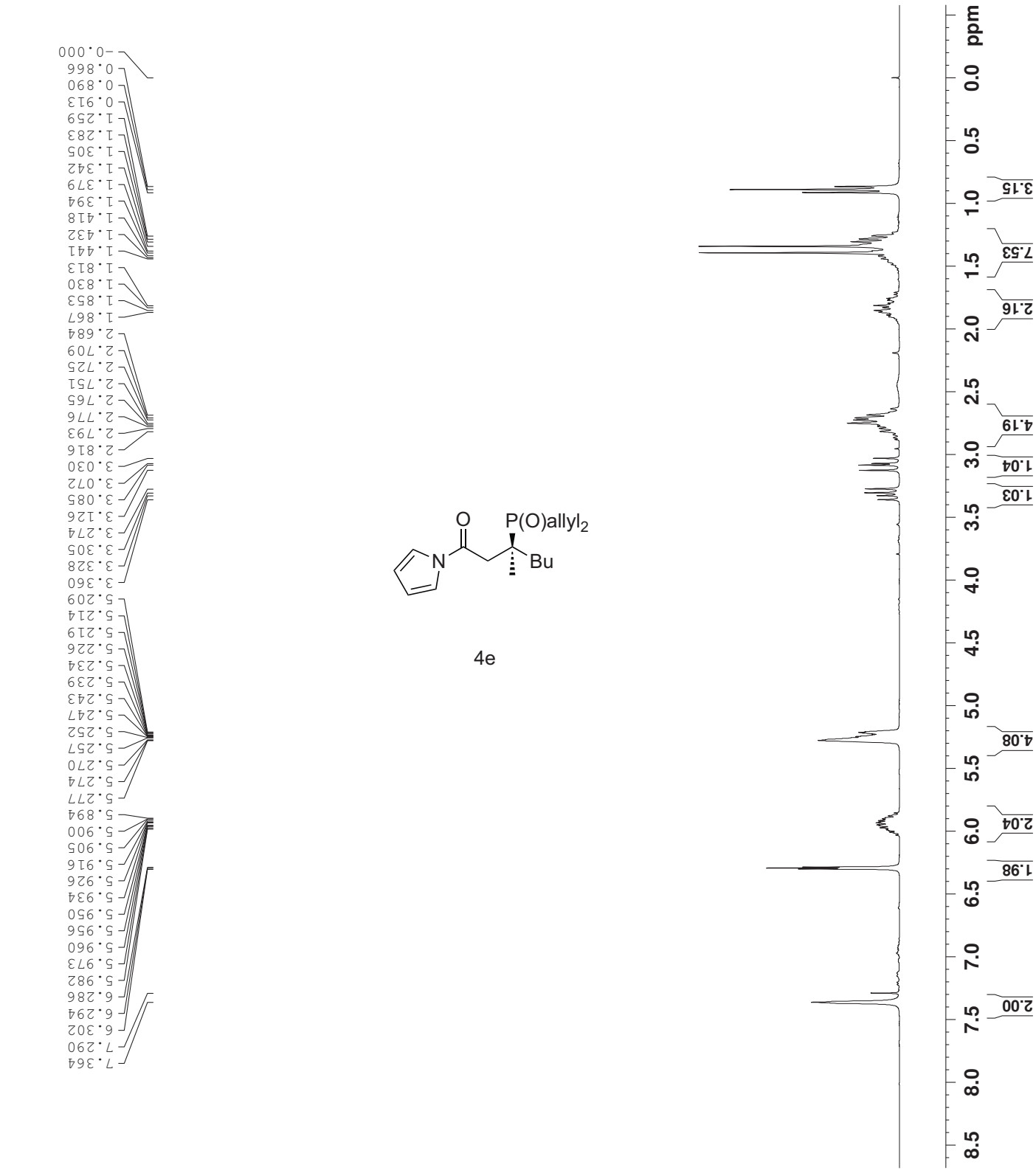
```
NAME                 110301
EXPNO                     2
PROCNO                    1
Date_              20110301
Time                 17.08
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                 49019.609 Hz
FIDRES              0.747980 Hz
AQ                  0.6685172 sec
RG                        203
DW                     10.200 usec
DE                      6.50 usec
TE                     286.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W                 9.17820644 W
PL12W                0.23054613 W
PL13W                0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



4d

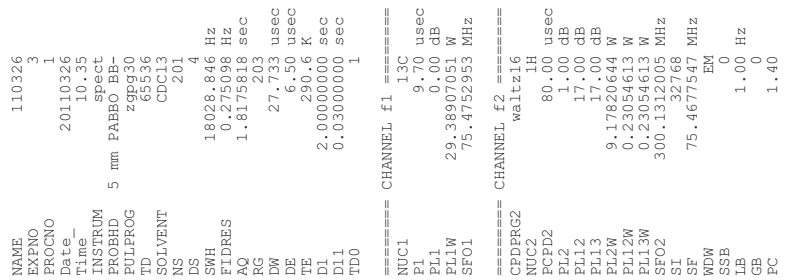
50.31



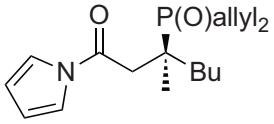

NAME 110326
EXPNO 1
PROCNO 1
Date_ 20110326
Time 10.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 289.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299932 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.15
7.53
2.16
4.19
1.04
1.03
4.08
2.04
1.98
2.00

O
P(O)allyl2
N
Bu

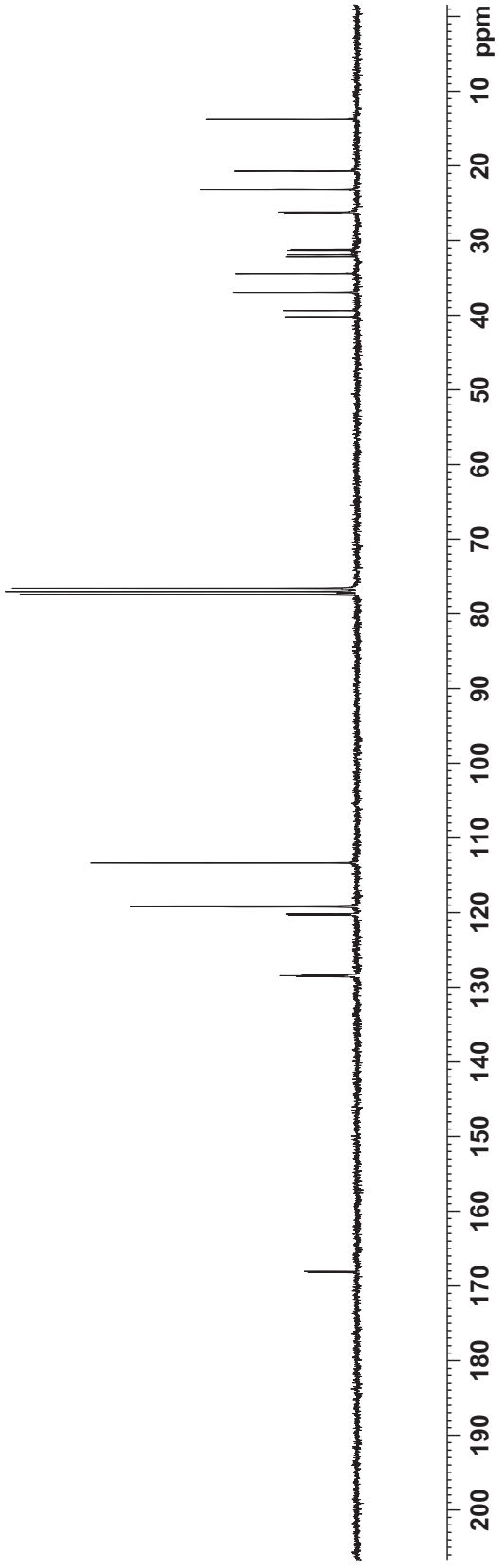
4e


NAME 110326
EXPNO 3
PROCNO 1
Date_ 20110326
Time_ 10.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 201
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

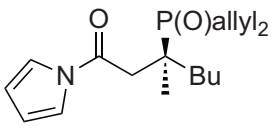

13.77
20.70
23.18
26.19
26.27
31.14
31.41
31.92
32.19
34.47
36.98
39.41
40.21
76.58
77.00
77.42
113.33
119.24
120.17
120.20
120.32
120.35
128.35
128.47
128.59
168.07
168.21


O
P(O)allyl2
N
Bu

4e


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

```
NAME                110326
EXPNO                    2
PROCNO                   1
Date_             20110326
Time                 10.24
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                49019.609 Hz
FIDRES                0.747980 Hz
AQ                    0.6685172 sec
RG                       203
DW                    10.200 usec
DE                     6.50 usec
TE                     290.1 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      31P
P1                       9.10 usec
PL1                      0.00 dB
PL1W                36.92473221 W
SFO1                121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.00 dB
PL12                     17.00 dB
PL13                     17.00 dB
PL2W                 9.17820644 W
PL12W                0.23054613 W
PL13W                0.23054613 W
SFO2                300.1312005 MHz
SI                       32768
SF                  121.4948510 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



4e