

Supporting Information

Copper-Mediated Coupling of 1,1-Dibromo-1-alkenes with Dialkylphosphonates: A Convenient Synthesis of 1-Alkenylphosphonates

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General Information.

All reactions were carried out in oven or flame-dried glassware under an argon atmosphere employing standard techniques in handling air-sensitive materials. Copper-catalyzed cyclization reactions were carried out in resealable pressure tubes.

All solvents were reagent grade. Toluene was freshly distilled from sodium/benzophenone under argon and degassed immediately prior to use.

Copper(I) iodide (99,999% purity) was purchased from Aldrich and used as supplied. Finely powdered potassium phosphate tribasic was purchased from Riedel-de Haën through Sigma Aldrich. All other reagents were used as supplied.

Unless otherwise noted, reactions were magnetically stirred and monitored by thin layer chromatography using Merck-Kieselgel 60F₂₅₄ plates. Flash chromatography was performed with silica gel 60 (particle size 35-70 μm) supplied by SDS. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise noted.

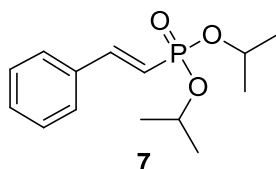
Proton NMR spectra were recorded using an internal deuterium lock at ambient temperature on a Bruker 300 MHz spectrometer. Internal reference of δ_{H} 7.26 was used for CDCl_3 . Data are presented as follows: chemical shift (in ppm on the δ scale relative to δ_{TMS} = 0), multiplicity (s = singlet, d = doublet, t = triplet, quint. = quintuplet, m = multiplet, br. = broad, app. = apparent), coupling constant (J/Hz) and integration. Resonances that are either partially or fully obscured are denoted obscured (obs.). Carbon-13 NMR spectra were recorded at 75 MHz and an internal reference of δ_{C} 77.16 was used for CDCl_3 . Phosphorus-31 NMR spectra were recorded at 121.5 MHz and are referenced to H_3PO_4 (85% solution in D_2O , 0 ppm). ^{19}F NMR spectra were recorded at 188 MHz.

Melting points were recorded on a Buchi B-545. Infrared spectra were recorded on a Nicolet iS 10 (SMART iTR diamond ATR) spectrophotometer. Mass spectra were obtained on a Waters XevoQTof spectrometer.

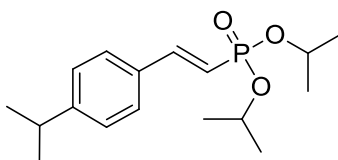
Experimental Procedures and Characterization Data

General procedure for the synthesis of vinylphosphonates from 1,1-dibromo-1-alkenes:

A 15 mL pressure tube was charged with dialkylphosphonate (2.5 mmol), 1,1-dibromo-1-alkene (1.0 mmol), K_3PO_4 (1.3 g, 6.0 mmol), and copper(I) iodide (76 mg, 0.40 mmol). The tube was fitted with a rubber septum, evacuated under high vacuum and backfilled with argon. Dry and degassed toluene (3 mL) and *N,N'*-dimethylethylenediamine (85 μ L, 0.8 mmol) were next added, the rubber septa was replaced by a Teflon-coated screw cap and the light blue-green suspension was heated at 120 $^{\circ}C$ for 20-24 h. The brownish suspension was cooled to rt, filtered over a plug of silica gel (washed with EtOAc), and concentrated. The crude residue was purified by flash chromatography over silica gel.



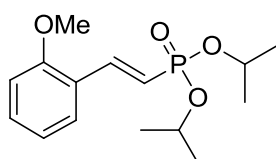
(E)-Diisopropyl styrylphosphonate 7.^{S1} Scale: 150 mg of compound obtained (*E/Z*: 90/10); yield: 60%. Pale yellow liquid; 1H NMR ($CDCl_3$, 300 MHz): δ 7.17-7.65 (m, 6 H), 6.18 (t app., J = 16.8 Hz, 1 H), 4.47-4.68 (m, 2 H), 1.35 and 1.29 (d, J = 6.1 Hz, d, J = 6.1 Hz, 12H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.5 (d, J = 7.1 Hz), 135.0 (d, J = 23.6 Hz), 129.8, 128.6, 127.4, 115.5 (d, J = 191.2 Hz), 70.1 (d, J = 5.5 Hz), 24.0 (d, J = 4.1 Hz), 23.8 (d, J = 4.1 Hz); ^{31}P NMR (121.5 MHz, $CDCl_3$): δ 17.2; IR (ATR) ν_{max} : 2970, 1620, 1445 1385, 1240, 1100, 980, 890, 825 cm^{-1} ; ESIMS (positive mode): 559.2, 291.1, 269.1, 185.0; ESI-HRMS (positive mode): calcd for $C_{14}H_{21}PO_3Na$ [$M+Na$] $^+$ 291.1126, found 291.1135.



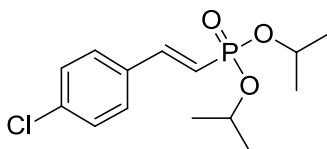
(E)-Diisopropyl 4-isopropylstyrylphosphonate. Scale: 180 mg of compound obtained (*E/Z*: 92/8); yield: 58%. Brown liquid; 1H NMR ($CDCl_3$, 300 MHz): δ 7.39 (dd, J = 22.5 and 17.6

^{S1} Inoue, H.; Tsubouchi, H.; Nagaoka, Y.; Tomioka, K. *Tetrahedron* **2002**, 58, 83.

Hz, 1H), 7.36 (d, $J = 8.2$ Hz, 2H), 7.16 (d, $J = 8.2$ Hz, 2H), 6.14 (t app., $J = 17.6$ Hz, 1H), 4.47-4.70 (m, 2H), 2.84 (sept., $J = 6.9$ Hz, 1H), 1.29, 1.23 (d, $J = 6.1$ Hz, d, $J = 6.1$ Hz, 12H), 1.17 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ 151.2, 148.0 (d, $J = 6.6$ Hz), 132.5 (d, $J = 23.1$ Hz), 127.7, 126.8, 114.0 (d, $J = 192.1$ Hz), 70.3 (d, $J = 5.5$ Hz), 34.0, 24.0 (d, $J = 3.8$ Hz), 23.8 (d, $J = 4.91$ Hz), 23.7, 23.6; ^{31}P NMR (121.5 MHz, CDCl_3): δ 17.7; IR (ATR) ν_{max} : 2975, 2930, 2865, 2160, 2030, 1965, 1710, 1610, 1470, 1380, 1250, 1175, 1100, 975 cm^{-1} ; ESIMS (positive mode): 643.3, 621.3, 334.2, 333.2, 311.2, 269.1, 227.1; ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{27}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 333.1596, found 333.1600.

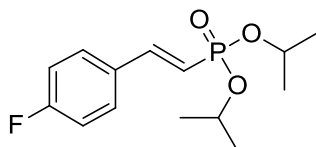


(E)-Diisopropyl 2-methoxystyrylphosphonate. Scale: 200 mg of compound obtained (E/Z : 89/11); yield: 67%. Pale yellow liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.74 (dd, $J = 23.6$ and 17.7 Hz, 1H), 7.39 (d, $J = 7.7$ Hz, 1H), 7.24 (t, $J = 8.4$ Hz, 1H), 6.73-6.88 (m, 2H), 6.27 (dd, $J = 19.3$ and 17.7 Hz, 1H), 4.55-4.69 (m, 2H), 3.76 (s, 3H), 1.28 and 1.23 (d, $J = 6.1$ Hz, d, $J = 6.6$ Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 157.7, 143.0 (d, $J = 7.6$ Hz), 131.0, 128.0, 124.0 (d, $J = 23.0$ Hz), 120.3, 116.8 (d, $J = 190.4$ Hz), 111.1, 70.5 (d, $J = 5.5$ Hz), 55.2, 24.1 (d, $J = 4.4$ Hz), 24.0 (d, $J = 5.0$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 18.4; IR (ATR) ν_{max} : 2975, 2930, 2200, 2160, 2035, 1980, 1710, 1660, 1490, 1460, 1380, 1290, 1245, 1105, 970 cm^{-1} ; ESIMS (positive mode): 620.3, 619.2, 322.1, 299.1, 257.1, 215.0; ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{23}\text{PO}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 321.1232, found 321.1231.

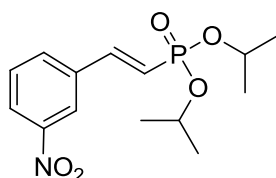


(E)-Diisopropyl 4-chlorostyrylphosphonate. Scale: 160 mg of compound obtained (E/Z : 89/11); yield: 56%. Yellow liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.19-7.38 (m, 5H), 6.17 (t app., $J = 17.0$ Hz, 1H), 4.54-4.68 (m, 2H), 1.27 and 1.23 (d, $J = 6.1$ Hz, d, $J = 6.1$ Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 145.2 (d, $J = 6.8$ Hz), 134.9, 132.5 (d, $J = 23.6$ Hz), 128.0, 127.8, 115.4 (d, $J = 191.0$ Hz), 69.5 (d, $J = 5.6$ Hz), 23.1 (d, $J = 4.5$ Hz), 23.0 (d, $J = 4.7$ Hz);

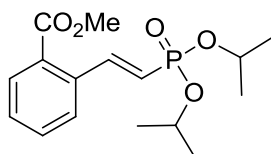
^{31}P NMR (121.5 MHz, CDCl_3): δ 16.9; IR (ATR) ν_{max} : 2790, 1620, 1590, 1490, 1400, 1220, 1090, 995, 860, 800 cm^{-1} ; ESIMS (positive mode): 629.2, 627.2, 327.1, 325.1, 303.1, 219.0; ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{20}\text{ClPO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 325.0736, found 325.0733.



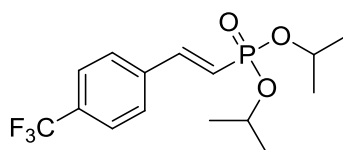
(E)-Diisopropyl 4-fluorostyrylphosphonate. Scale: 150 mg of compound obtained (*E/Z*: 95/5); yield: 52%. Light yellow liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.40 (dd, J = 6.5 Hz, 2H), 7.32 (d, J = 17.3 Hz, 1H), 6.98 (t, J = 8.8 Hz, 2H), 6.10 (t app., J = 17.3 Hz, 1H), 4.52-4.70 (m, 2H), 1.28 and 1.23 (d, J = 6.2 Hz, d, J = 6.2 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 163.8 (d, J = 251.0 Hz), 146.3 (d, J = 7.1 Hz), 131.0 (dd, J = 3.3, 23.0 Hz), 129.3 (d, J = 8.8 Hz), 115.7 (d, J = 22.0 Hz), 115.0 (d, J = 192.0 Hz), 70.3 (d, J = 5.5 Hz), 23.9 (d, J = 3.8 Hz), 23.8 (d, J = 4.9 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 17.2; ^{19}F NMR (188.0 MHz, CDCl_3): δ -110.30 - -110.10 (m); IR (ATR) ν_{max} : 2980, 2930, 2160, 2025, 1970, 1715, 1620, 1600, 1510, 1380, 1230, 1100, 975 cm^{-1} ; ESIMS (positive mode): 596.2, 595.2, 310.1, 309.1, 203.0; ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{20}\text{FPO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 309.1032, found 309.0984.



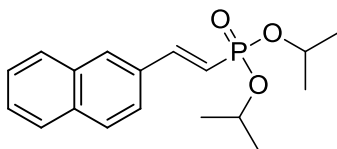
(E)-Diisopropyl 3-nitrostyrylphosphonate. Scale: 250 mg of compound obtained (*E/Z*: 87/13); yield: 84%. Yellow solid; Mp: 58-62 $^{\circ}\text{C}$; ^1H NMR (CDCl_3 , 300 MHz): δ 8.26 (s, 1H), 8.11 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 7.7 Hz, 1H), 7.31-7.56 (m, 2H), 6.38 (t app., J = 15.8 Hz, 1H), 4.55-4.73 (m, 2H), 1.29 and 1.25 (d, J = 6.1 Hz, d, J = 6.1 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 148.2, 144.3 (d, J = 7.1 Hz), 136.6 (d, J = 24.4 Hz), 133.0, 129.6, 124.0, 121.0, 119.3 (d, J = 191.6 Hz), 70.5 (d, J = 5.5 Hz), 24.0 (d, J = 4.4 Hz), 24.0 (d, J = 3.8 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 15.3; IR (ATR) ν_{max} : 2980, 1530, 1350, 1250, 1105, 985, 895, 810, 730 cm^{-1} ; ESIMS (positive mode): 650.2, 649.2, 337.1, 336.1, 314.1, 272.1, 230.0; ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{20}\text{NPO}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 336.0977, found 336.0945.



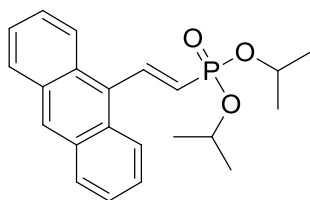
(E)-Diisopropyl 2-(carbomethoxy)styrylphosphonate. Scale: 170 mg of compound obtained (*E/Z*: 83/17); yield: 52%. Brown liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 8.09 (dd, J = 22.4 and 17.4 Hz, 1H), 7.86 (dd, J = 7.6 and 1.0 Hz, 1H), 7.42-7.52 (m, 1H), 7.31-7.38 (m, 2H), 6.10 (t app., J = 17.4 Hz, 1H), 4.62-4.75 (m, 2H), 3.84 (s, 3H), 1.31 and 1.28 (d, J = 6.5 Hz, d, J = 6.5 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 167.0, 146.3 (d, J = 8.2 Hz), 137.1 (d, J = 24.1 Hz), 132.2, 130.6, 129.5, 129.1, 127.6, 119.2 (d, J = 191.0 Hz), 70.5 (d, J = 5.5 Hz), 52.3, 24.0 (d, J = 4.4 Hz), 23.9 (d, J = 4.4 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 16.3; IR (ATR) ν_{max} : 2975, 2850, 1715, 1620, 1480, 1435, 1295, 1265, 1195, 1130, 850 cm^{-1} ; ESIMS (positive mode): 675.2, 350.1, 349.1, 327.1, 307.1, 243.0, 211.0; ESI-HRMS: calcd for $\text{C}_{16}\text{H}_{23}\text{PO}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 349.1181, found 349.1165.



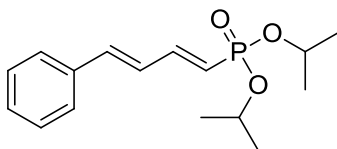
(E)-Diisopropyl 4-(trifluoromethyl)styrylphosphonate. Scale: 220 mg of compound obtained (*E/Z*: 80/20); yield: 67%. Yellow oily solid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.48-7.56 (m, 4H), 7.41 (dd, J = 22.3 and 17.3 Hz, 1H), 6.30 (t app., J = 17.2 Hz, 1H), 4.58-4.72 (m, 2H), 1.29 and 1.24 (d, J = 6.1 Hz, d, J = 6.1 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 145.6 (d, J = 6.6 Hz), 138.0 (d, J = 23.6 Hz), 131.3 (d, J = 32.4 Hz), 127.6, 125.6 (q, J = 3.8 Hz), 123.9 (q, J = 281.4 Hz), 118.8 (d, J = 191.0 Hz), 70.5 (d, J = 6.0 Hz), 24.0 (d, J = 4.1 Hz), 23.8 (d, J = 4.1 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 16.0; ^{19}F NMR (188 MHz, CDCl_3): δ -65.0; IR (ATR) ν_{max} : 2970, 2360, 1750, 1605, 1320, 1165, 1175, 1015, 885 cm^{-1} ; ESIMS (positive mode): 695.2, 359.1, 337.1, 295.1, 253.0; ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{20}\text{F}_3\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 359.1000, found 359.0993.



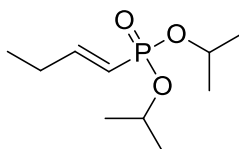
(E)-Diisopropyl 2-(naphthalen-2-yl)vinylphosphonate. Scale: 190 mg of compound obtained (*E/Z*: 92/8); yield: 60%. Dark brown liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 8.21 (dd, $J = 22.4$ and 17.2 Hz, 1H), 8.08 (d, $J = 8.2$ Hz, 1H), 7.32-7.81 (m, 6H), 6.28 (t app., $J = 17.3$ Hz, 1H), 4.60-4.77 (m, 2H), 1.29 and 1.23 (d, $J = 6.1$ Hz, d, $J = 6.1$ Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 144.8 (d, $J = 7.1$ Hz), 133.6, 132.7 (d, $J = 23.0$ Hz), 131.1, 130.3, 128.7, 126.8, 126.2, 125.4, 124.6, 123.3, 118.8 (d, $J = 190.0$ Hz), 70.6 (d, $J = 5.5$ Hz), 24.0 (d, $J = 4.1$ Hz), 23.8 (d, $J = 4.1$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 16.8; IR (ATR) ν_{max} : 2980, 2365, 1610, 1380, 1240, 1110, 980, 890, 790 cm^{-1} ; ESIMS (positive mode): 659.3, 637.3, 615.2, 342.1, 341.1, 297.1, 275.1, 247.1; ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{23}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 341.1283, found 341.1287.



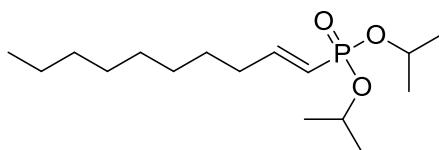
(E)-Diisopropyl 2-(anthracen-9-yl)vinylphosphonate. Scale: 180 mg of compound obtained (*E/Z* > 95/5); yield: 48%. Brown liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 8.24 (dd, $J = 22.8$ and 17.8 Hz, 1H), 8.23 (s, 1H), 8.07 (d, $J = 8.4$ Hz, 2H), 7.82 (d, $J = 7.7$ Hz, 2H), 7.29-7.38 (m, 4H), 6.20 (dd, $J = 20.8$ and 18.1 Hz, 1H), 4.68-4.83 (m, 2H), 1.33 and 1.31 (d, $J = 4.2$ Hz, d, $J = 4.2$ Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 145.0 (d, $J = 6.0$ Hz), 131.2, 130.5 (d, $J = 22.5$ Hz), 129.0, 128.8, 128.0, 127.0 (d, $J = 185.5$ Hz), 126.3, 125.4, 125.1, 70.5 (d, $J = 5.5$ Hz), 24.3 (d, $J = 3.3$ Hz), 24.2 (d, $J = 3.3$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 15.2; IR (ATR) ν_{max} : 2975, 2930, 2160, 2030, 1970, 1710, 1660, 1600, 1450, 1390, 1240, 1105, 980, 895, 730 cm^{-1} ; ESIMS (positive mode): 791.3, 760.3, 759.3, 737.3, 423.1, 392.1, 391.1, 369.2, 327.1, 285.1; ESI-HRMS: calcd for $\text{C}_{22}\text{H}_{25}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 391.1439, found 391.1440.



(1E,3E)-Diisopropyl 4-phenylbuta-1,3-dienylphosphonate. Scale: 210 mg of compound obtained (*E/Z* > 95/5); yield: 71%. Yellow solid; Mp: 68-70 °C; ^1H NMR (CDCl_3 , 300 MHz): δ 7.33-7.36 (m, 2H), 7.06-7.27 (m, 4H), 6.73 (d, J = 5.4 Hz, 2H), 5.72 (t app., J = 17.7 Hz, 1H), 4.48-4.70 (m, 2H), 1.26 and 1.22 (d, J = 6.1 Hz, d, J = 6.1 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 147.5 (d, J = 6.0 Hz), 139.1, 135.7 (d, J = 2.2 Hz), 128.7, 128.5, 127.2, 126.9, 118.6 (d, J = 192.1 Hz), 70.5 (d, J = 5.5 Hz), 24.0 (d, J = 4.1 Hz), 23.8 (d, J = 4.1 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 16.9; IR (ATR) ν_{max} : 2980, 1530, 1350, 1250, 1105, 985, 895, 810, 730 cm^{-1} ; ESIMS (positive mode): 612.3, 611.3, 318.1, 317.1, 295.1, 253.1, 211.1; ESI-HRMS: calcd for $\text{C}_{16}\text{H}_{23}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 317.1283, found 317.1291.

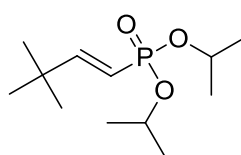


(E)-Diisopropyl but-1-en-1-ylphosphonate. Scale: 100 mg of compound obtained (*E/Z* > 95/5); yield: 45%. Light brown liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 6.74 (ddt, J = 22.4, 17.6 and 5.6 Hz, 1H), 5.57 (br. t app., J = 17.6 Hz, 1H), 4.53-4.61 (m, 2H), 2.10-2.22 (m, 2H), 1.26, 1.23 (d, J = 6.1 Hz, d, J = 6.1 Hz, 12H), 0.99 (t, J = 8.1 Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ 154.0 (d, J = 4.9 Hz), 117.0 (d, J = 189.4 Hz), 70.0 (d, J = 6.0 Hz), 26.8 (d, J = 22.5 Hz), 24.0 (d, J = 4.9 Hz), 24.0 (d, J = 4.4 Hz), 11.8; ^{31}P NMR (121.5 MHz, CDCl_3): δ 17.3; IR (ATR) ν_{max} : 2970, 2350, 1245, 1100, 1000, 960, 970, 890 cm^{-1} ; ESIMS (positive mode): 463.2, 244.1, 243.1, 201.1; ESI-HRMS: calcd for $\text{C}_{10}\text{H}_{21}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 243.1126, found 243.1116.

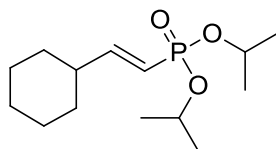


(E)-Diisopropyl dec-1-en-1-ylphosphonate. Scale: 120 mg of compound obtained (*E/Z* > 95/5); yield: 59%. Colorless liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 6.68 (ddt, J = 21.9, 17.0

and 6.8 Hz, 1H), 5.57 (ddt, $J = 21.0, 17.0$ and 1.6 Hz, 1H), 4.52-4.63 (m, 2H), 2.13 (q app., $J = 6.9$ Hz, 2H), 1.25 and 1.22 (d, $J = 6.1$ Hz, d, $J = 6.5$ Hz, 12H), 1.14-1.39 (obs. m, 12H), 0.81 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ 153.0 (d, $J = 4.4$ Hz), 118.0 (d, $J = 118.3$ Hz), 70.0 (d, $J = 5.5$ Hz), 34.0 (d, $J = 2.2$ Hz), 31.7, 29.2, 29.1, 29.0, 27.6, 23.9 (d, $J = 4.4$ Hz), 23.8 (d, $J = 4.4$ Hz), 22.5, 14.0; ^{31}P NMR (121.5 MHz, CDCl_3): δ 17.0; IR (ATR) ν_{max} : 3000, 2850, 2360, 1460, 1260, 1004, 865, 820, 780 cm^{-1} ; ESIMS (positive mode): 935.6, 632.4, 631.4, 609.4, 327.2; ESI-HRMS: calcd for $\text{C}_{16}\text{H}_{33}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 327.2065, found 327.2075.

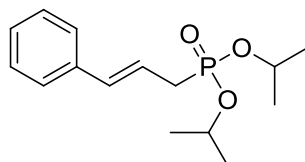


(E)-Diisopropyl 3,3-dimethylbut-1-en-1-ylphosphonate.^{S1} Scale: 120 mg of compound obtained ($E/Z > 95/5$); yield: 48%. Colorless liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 6.51 (dd, $J = 22.9$ and 17.3 Hz, 1H), 5.31 (t app., $J = 19.8$ Hz, 1H), 4.37-4.46 (m, 2H), 1.10 and 1.06 (d, $J = 6.1$ Hz, d, $J = 6.1$ Hz, 12H), 1.0 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3): δ 162.0 (d, $J = 3.8$ Hz), 113.0 (d, $J = 188.8$ Hz), 70.0 (d, $J = 5.5$ Hz), 34.5 (d, $J = 18.6$ Hz), 28.2, 23.8 (d, $J = 4.4$ Hz), 23.7 (d, $J = 4.4$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 18.4; IR (ATR) ν_{max} : 2950, 2860, 2365, 1620, 1480, 1370, 1250, 1175, 1010 cm^{-1} ; ESIMS (positive mode): 615.1, 520.3, 519.3, 271.1, 249.2, 207.1, 165.1; ESI-HRMS: calcd for $\text{C}_{12}\text{H}_{25}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 271.1439, found 271.1442.

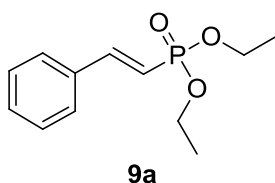


(E)-Diisopropyl 2-cyclohexylvinylphosphonate.^{S1} Scale: 140 mg of compound obtained ($E/Z > 95/5$); yield: 54%. Colorless liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 6.51 (ddd, $J = 22.5, 17.0$ and 6.3 Hz, 1H), 5.31 (t app., $J = 19.0$ Hz, 1H), 4.51-4.62 (m, 2H), 1.93-2.09 (m, 1H), 1.53-1.75 (m, 4H), 1.25 and 1.21 (d, $J = 6.1$ Hz, d, $J = 6.1$ Hz, 12H), 0.98-1.20 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ 157.4 (d, $J = 3.8$ Hz), 115.2 (d, $J = 188.8$ Hz), 69.8 (d, $J = 5.5$ Hz), 41.6 (d, $J = 20.8$ Hz), 31.2, 25.9, 25.6, 24.0 (d, $J = 4.4$ Hz), 23.8 (d, $J = 3.8$ Hz); ^{31}P NMR

(121.5 MHz, CDCl₃): δ 17.5; IR (ATR) ν_{max} : 2980, 2925, 2850, 2365, 2195, 2160, 1720, 1630, 1450, 1385, 1255, 980 cm⁻¹; ESIMS (positive mode): 845.5, 571.3, 297.2; ESI-HRMS: calcd for C₁₄H₂₇PO₃Na [M+Na]⁺ 297.1596, found 297.1608.



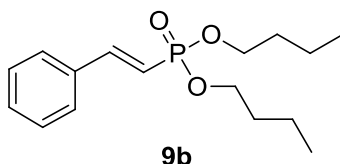
(E)-Diisopropyl cinnamylphosphonate.^{S2} Scale: 100 mg of compound obtained (*E/Z* > 95/5); yield: 62%. Colorless liquid; ¹H NMR (CDCl₃, 300 MHz): δ 7.09-7.29 (m, 5H), 6.40 (dd, *J* = 15.8 and 5.1 Hz, 1H), 6.05 (ddd, *J* = 15.8, 7.6 and 7.3 Hz, 1H), 4.43-4.68 (m, 2H), 2.63 (dd, *J* = 22.3 and 7.3 Hz, 2H), 1.22 and 1.19 (d, *J* = 6.1 Hz, d, *J* = 6.1 Hz, 12H); ¹³C NMR (75 MHz, CDCl₃): δ 135.1 (d, *J* = 3.3 Hz), 132.6 (d, *J* = 14.8 Hz), 126.7, 125.6, 124.3, 117.5 (d, *J* = 12.1 Hz), 69.0 (d, *J* = 5.5 Hz), 30.2 (d, *J* = 140.5 Hz), 23.8 (d, *J* = 3.3 Hz), 23.8 (d, *J* = 2.2 Hz); ³¹P NMR (121.5 MHz, CDCl₃): δ 25.0; IR (ATR) ν_{max} : 2980, 2155, 2025, 1975, 1735, 1550, 1390, 1230, 985 cm⁻¹; ESIMS (positive mode): 459.1, 305.1, 263.1, 221.0, 199.0, 117.1; ESI-HRMS: calcd for C₁₅H₂₃PO₃Na [M+Na]⁺ 305.1283, found 305.1283.



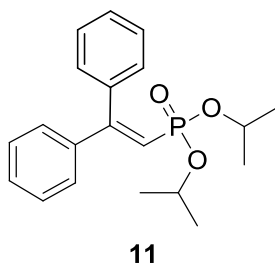
(E)-Diethyl styrylphosphonate 9a.^{S3} Scale: 180 mg of compound obtained (*E/Z*: 94/6); yield: 77%. Pale yellow liquid; ¹H NMR (CDCl₃, 300 MHz): δ 7.22-7.51 (m, 6H), 6.17 (t app., *J* = 17.3 Hz, 1H), 3.98-4.09 (m, 4H), 1.26 (t, *J* = 7.7 Hz, 6H); ¹³C NMR (75 MHz, CDCl₃): δ 148.4 (d, *J* = 7.1 Hz), 134.4 (d, *J* = 23.6 Hz), 130.0, 128.5, 127.4, 113.4 (d, *J* = 191.0 Hz), 61.5 (d, *J* = 5.5 Hz), 16.0 (d, *J* = 6.6 Hz); ³¹P NMR (121.5 MHz, CDCl₃): δ 19.6; IR (ATR) ν_{max} : 2980, 2370, 1620, 1450, 1390, 1245, 1165, 1045, 1025 cm⁻¹; ESIMS (positive mode): 539.2, 503.2, 264.1, 263.1, 241.1; ESI-HRMS: calcd for C₁₂H₁₇PO₃Na [M+Na]⁺ 263.0813, found 263.0825.

^{S2} Wender, P. A.; Smith, T. E. *Tetrahedron* **1998**, 54, 1255.

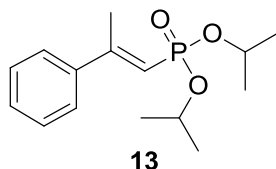
^{S3} Thielges, S.; Bisseret, P.; Eustache, J. *Org. Lett.* **2005**, 7, 681.



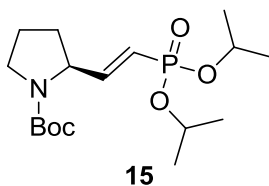
(E)-Dibutyl styrylphosphonate 9b. Scale: 170 mg of compound obtained (*E/Z*: 84/16); yield: 55%. Pale yellow liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.23-7.61 (m, 6H), 6.17 (t app., J = 17.7 Hz, 1H), 3.97 (q app., J = 6.9 Hz, 4H), 1.14-1.63 (m, 8H), 0.84 (t, J = 7.3 Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ 148.3 (d, J = 7.1 Hz), 134.5 (d, J = 23.0 Hz), 129.8, 128.5, 127.3, 113.6 (d, J = 191.0 Hz), 65.2 (d, J = 5.5 Hz), 32.2 (d, J = 6.6 Hz), 18.7, 13.2; ^{31}P NMR (121.5 MHz, CDCl_3): δ 20.3; IR (ATR) ν_{max} : 2980, 1625, 1370, 1240, 1180, 1110, 970 cm^{-1} ; ESIMS (positive mode): 911.5, 615.3, 593.3, 319.1, 297.2; ESI-HRMS: calcd for $\text{C}_{16}\text{H}_{25}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 319.1439, found 319.1454.



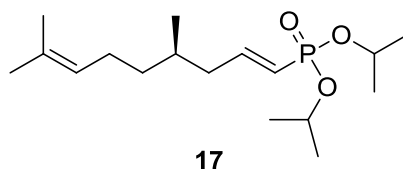
Diisopropyl 2,2-diphenylvinylphosphonate 11. Scale: 100 mg of compound obtained; yield: 58%. Colorless liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.12-7.37 (m, 10H), 6.10 (d, J = 15.8 Hz, 1H), 4.39-4.49 (m, 2H), 1.13 (d, J = 6.1 Hz, 6H), 1.02 (d, J = 6.1 Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ 159.3 (d, J = 5.5 Hz), 142.0 (d, J = 22.5 Hz), 139.0, 138.9, 130.0, 129.0, 128.4, 128.1, 127.6, 116.4 (d, J = 194.3 Hz), 70.2 (d, J = 6.6 Hz), 23.9 (d, J = 4.9 Hz), 23.6 (d, J = 3.8 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 14.5; IR (ATR) ν_{max} : 2970, 1600, 1450, 1385, 1245, 1110, 970, 840 cm^{-1} ; ESIMS (positive mode): 711.3, 689.3, 367.1, 345.2; ESI-HRMS: calcd for $\text{C}_{20}\text{H}_{25}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 367.1439, found 367.1421.



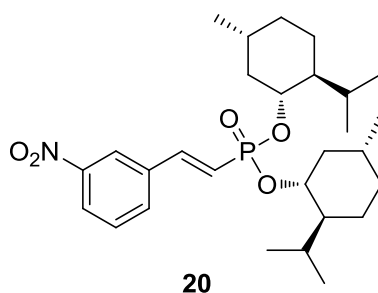
(E)-Dibutyl 2-phenylprop-1-enylphosphonate 13. Scale: 110 mg of compound obtained (*E/Z* > 95/5); yield: 78%. Colorless liquid; ^1H NMR (CDCl_3 , 300 MHz): δ 7.22-7.41 (m, 5H), 5.85 (d, J = 16.6 Hz, 1H), 4.56-4.72 (m, 2H), 2.42 (dd, J = 1.2, 3.5 Hz, 3H), 1.29 and 1.26 (d, J = 6.1 Hz, d, J = 6.1 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 157.0 (d, J = 8.2 Hz), 141.8 (d, J = 24.1 Hz), 128.9, 128.4, 128.4, 125.8, 115.2 (d, J = 190.4 Hz), 70.0 (d, J = 5.5 Hz), 24.1 (d, J = 4.9 Hz), 24.0 (d, J = 3.8 Hz); ^{31}P NMR (121.5 MHz, CDCl_3): δ 16.2; IR (ATR) ν_{max} : 3055, 2365, 1605, 1570, 1495, 1445, 1320, 1255, 1100, 995, 960, 880 cm^{-1} ; ESIMS (positive mode): 587.3, 538.2, 305.1, 283.1, 241.1, 199.0; ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{23}\text{PO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 305.1283, found 305.1296.



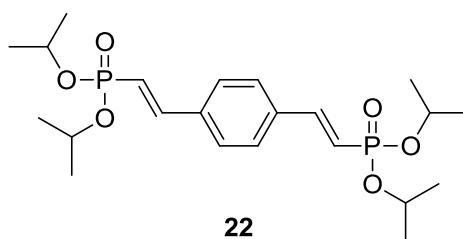
(S,E)-1-tert-Butoxycarbonyl-2-(2-(diisopropoxyphosphoryl)vinyl)pyrrolidine 15. Scale: 200 mg of compound obtained (*E/Z* > 95/5); yield: 55%. Colorless liquid; $[\alpha]_{\text{D}}^{20}$ -17 (c 1.0, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz): δ 6.18-6.42 (m, 1H), 5.41 (t app., J = 15.1 Hz, 1H), 5.03 (q app., J = 7.4 Hz, 1H), 4.51-4.63 (m, 2H), 3.39-3.52 (br. m, 1H), 3.26-3.33 (m, 1H), 2.22-2.32 (m, 1H), 1.69-1.80 (m, 2H), 1.55-1.66 (m, 1H), 1.36 (s, 9H), 1.27 and 1.24 (d, J = 6.5 Hz, d, J = 6.2 Hz, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ 155.5, 154.6, 115.5 (d, J = 187.7 Hz), 79.6, 70.0 (d, J = 5.5 Hz), 56.6, 46.8, 34.0, 28.4, 24.0 (d, J = 4.1 Hz), 23.8 (d, J = 4.1 Hz) (*1C not observed*); ^{31}P NMR (121.5 MHz, CDCl_3): δ 14.3; IR (ATR) ν_{max} : 2950, 2360, 1695, 1390, 1365, 1155, 1115, 920 cm^{-1} ; ESIMS (positive mode): 745.4, 384.2, 362.2, 262.2, 220.1, 178.1; ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{34}\text{NPO}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 384.1916, found 384.1880.



(*R,E*)-Diisopropyl (4,8-dimethylnona-1,7-dien-1-yl)phosphonate 17. Scale: 140 mg of compound obtained (*E/Z*: 87/13); yield: 44%. Brown liquid; $[\alpha]_D^{20} +17$ (*c* 1.0, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 6.64 (ddt, *J* = 21.8, 17.0 and 7.2 Hz, 1H), 5.60 (dd, *J* = 21.2 and 17.0 Hz, 1H), 5.00 (t, *J* = 6.2 Hz, 1H), 4.45-4.70 (m, 2H), 2.30-2.55 (m, 1H), 2.08-2.24 (m, 1H), 1.80-2.01 (m, 3H), 1.60 (s, 3H), 1.52 (s, 3H), 1.25 and 1.22 (d, *J* = 6.2 Hz, d, *J* = 6.2 Hz, 12H), 1.10-1.26 (obs. m, 2H), 0.82 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 150.4 (d, *J* = 4.4 Hz), 131.2, 124.3, 119.5 (d, *J* = 187.2 Hz), 70.0 (d, *J* = 5.5 Hz), 40.5 (d, *J* = 22.0 Hz), 36.5, 31.7 (d, *J* = 1.6 Hz), 25.6, 25.3, 23.9 (d, *J* = 3.8 Hz), 23.8 (d, *J* = 4.9 Hz), 19.3, 17.5; ³¹P NMR (121.5 MHz, CDCl₃): δ 16.5; IR (ATR) $\nu_{\max}$: 2970, 1755, 1625, 1455, 1385, 1240, 1100, 985 cm⁻¹; ESIMS (positive mode): 687.4, 655.4, 371.2, 339.2, 317.2, 275.2, 233.1; ESI-HRMS: calcd for C₁₇H₃₃PO₃Na [M+Na]⁺ 339.2065, found 339.2074.



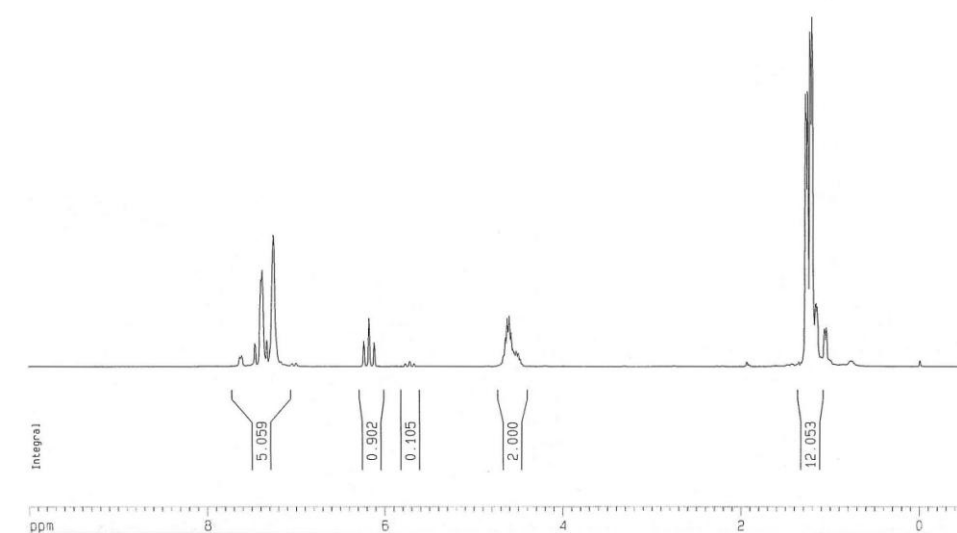
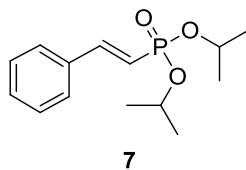
Bis[(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl] (*E*)-3-nitrostyrylphosphonate 20. Scale: 330 mg of compound obtained (*E/Z* > 95/5); yield: 65%. Pale yellow solid Mp: 155-160 °C; $[\alpha]_D^{20} -93$ (*c* 1.0, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 8.35 (s, 1H), 8.21 (d, *J* = 8.1 Hz, 1H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.47-7.60 (m, 2H), 6.40 (dd, *J* = 17.4 and 15.2 Hz, 1H), 4.23-4.35 (m, 1H), 4.10-4.18 (m, 1H), 2.01-2.37 (m, 4H), 1.63-1.70 (m, 4H), 0.68-1.47 (m, 28H); ¹³C NMR (75 MHz, CDCl₃): δ 148.7, 144.5 (d, *J* = 7.5 Hz), 137.0 (d, *J* = 24.0 Hz), 133.4, 129.9, 124.2, 121.8, 120.1 (d, *J* = 191.0 Hz), 77.8 (d, *J* = 6.8 Hz), 77.4 (d, *J* = 5.4 Hz), 48.6 (d, *J* = 6.5 Hz), 48.5 (d, *J* = 6.8 Hz), 43.5, 43.2, 34.0, 31.6, 31.5, 25.5, 22.8, 22.7, 21.9, 21.0, 15.9, 15.5; ³¹P NMR (121.5 MHz, CDCl₃): δ 15.0-16.0 (m); IR (ATR) $\nu_{\max}$: 2970, 1376, 1245, 1102, 972 cm⁻¹; ESIMS (positive mode): 528.3, 277.1, 229.1; ESI-HRMS: calcd for C₂₈H₄₄NPO₅Na [M+Na]⁺ 528.2855, found 528.2873.



Tetraisopropyl ((1*E*,1'*E*)-1,4-phenylenebis(ethene-2,1-diyl))bis(phosphonate) 22. Scale: 380 mg of compound obtained (*E/Z*: 88/12); yield: 83%. Yellow crystals; Mp: 135-140 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.44 (s, 4H), 7.39 (dd, *J* = 22.3 and 17.3 Hz, 2H), 6.23 (t app., *J* = 17.3 Hz, 2H), 4.56-4.72 (m, 4H), 1.30 and 1.25 (d, *J* = 6.1 Hz, d, *J* = 6.1 Hz, 24H); ¹³C NMR (75 MHz, CDCl₃): δ 146.5 (d, *J* = 7.1 Hz), 136.6 (d, *J* = 23.6 Hz), 128.0, 116.8 (d, *J* = 191.5 Hz), 70.6 (d, *J* = 5.5 Hz), 24.1 (d, *J* = 1.6 Hz), 24.0 (d, *J* = 1.6 Hz); ³¹P NMR (121.5 MHz, CDCl₃): δ 17.1; IR (ATR) ν_{max}: 2970, 1620, 1460, 1380, 1230, 1110, 970, 895 cm⁻¹; ESIMS (positive mode): 481.2, 439.2, 417.2, 375.1, 359.1, 253.2; ESI-HRMS: calcd for C₂₂H₃₆P₂O₆Na [M+Na]⁺ 481.1885, found 481.1800.

Supporting Information

^1H and ^{13}C NMR spectra



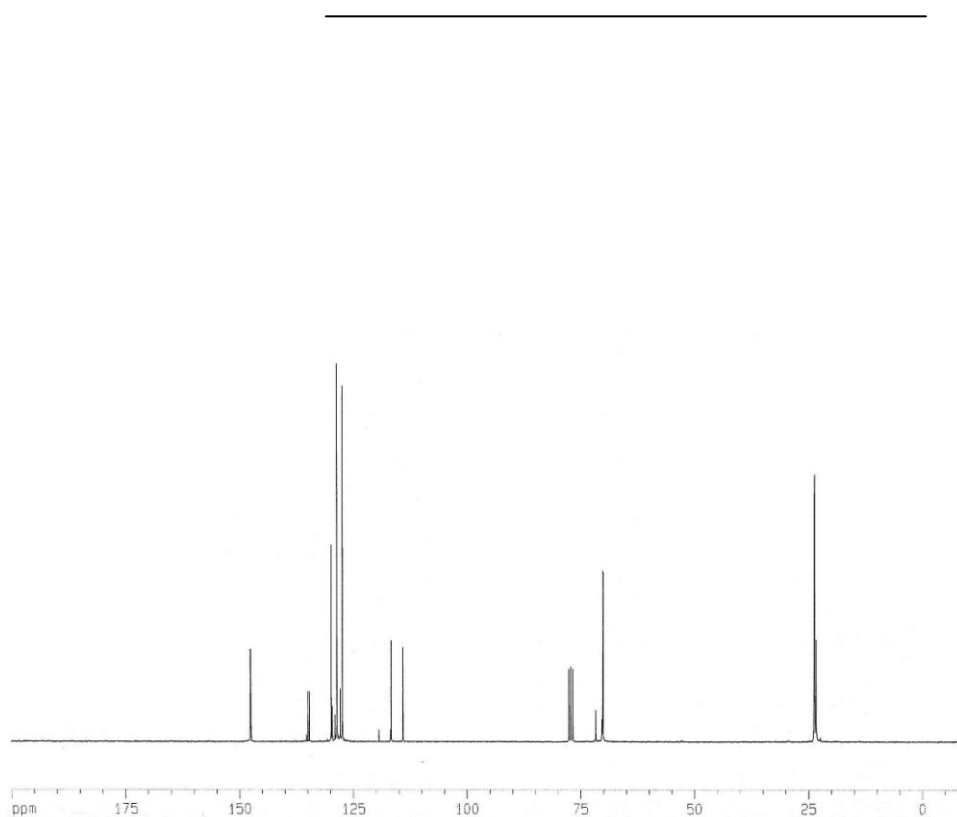
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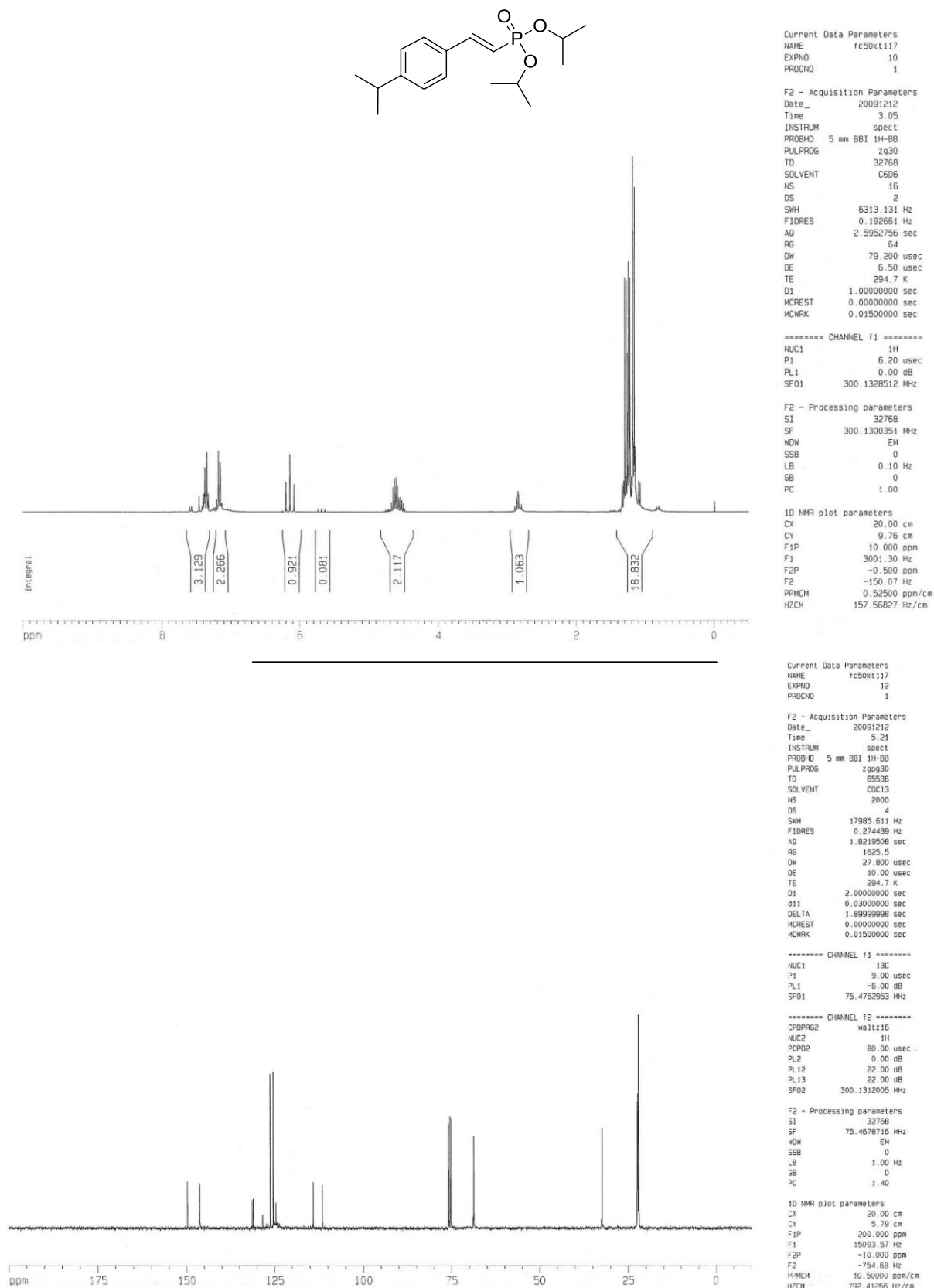
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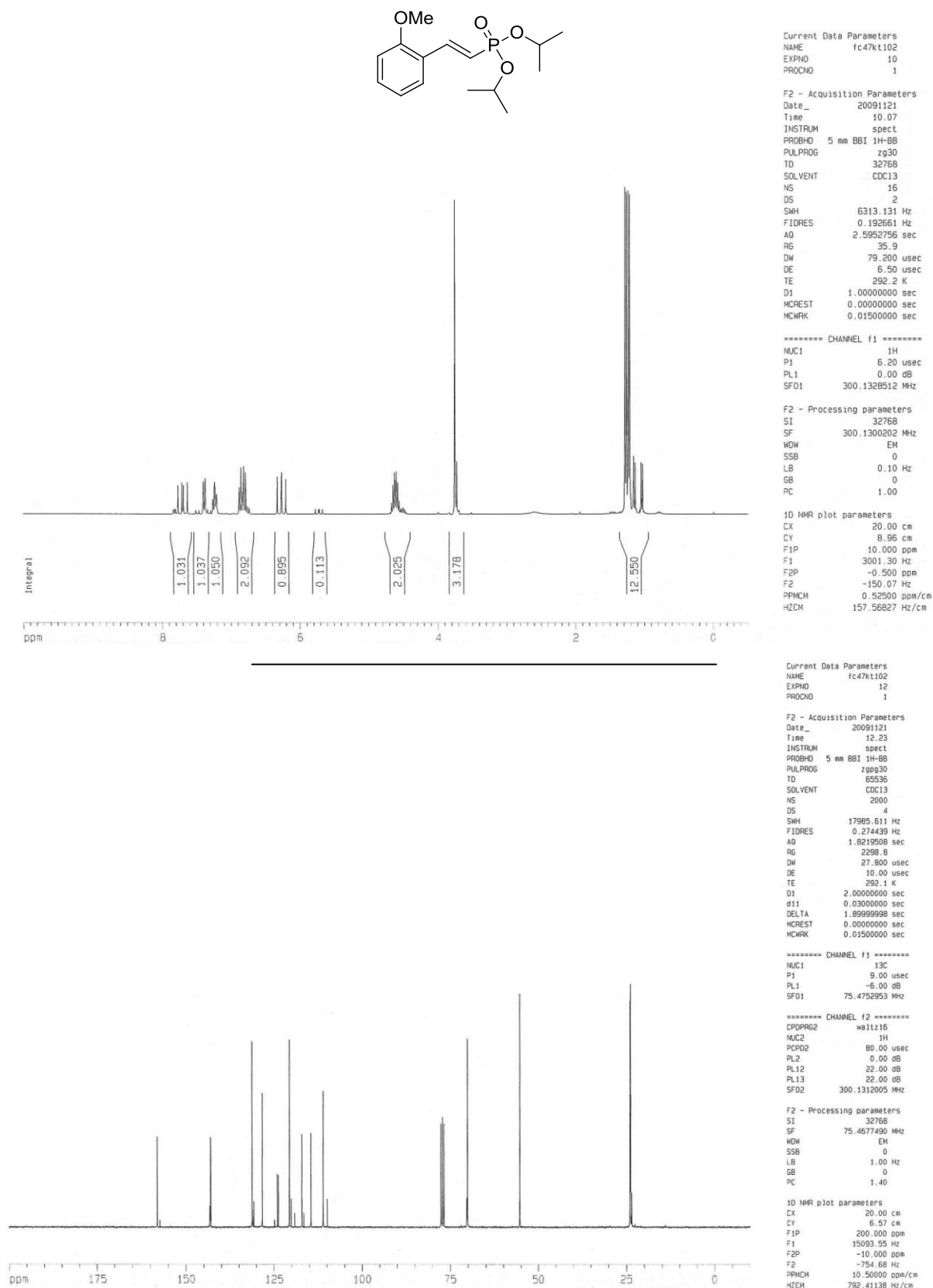
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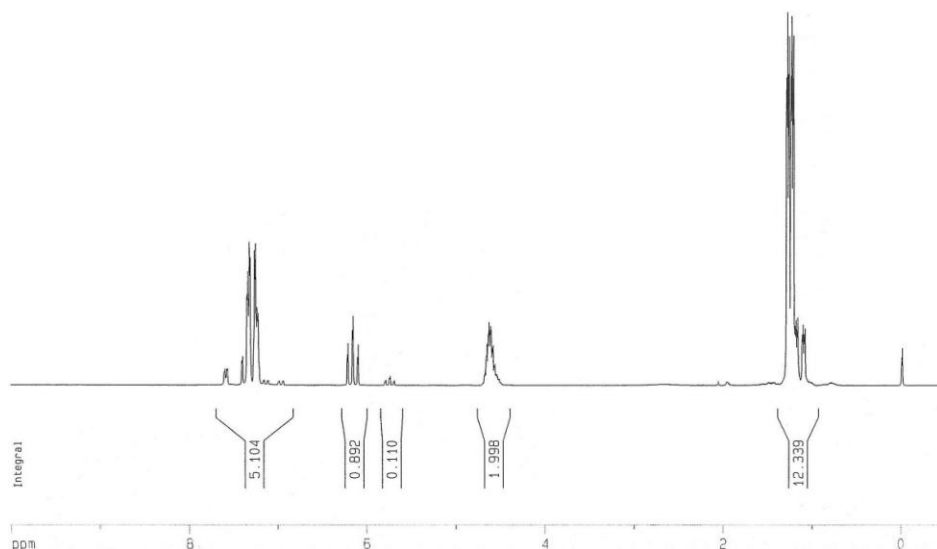
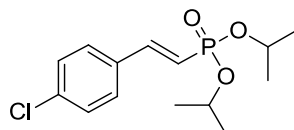
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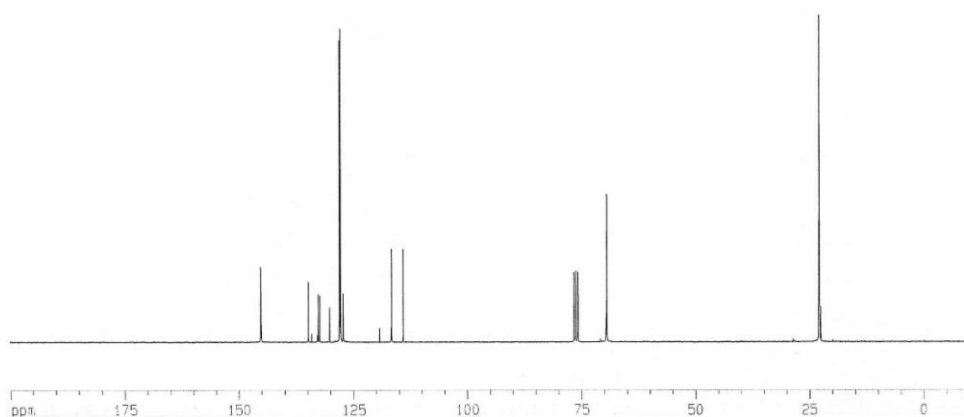
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Current Data Parameters
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EXPNO 10
PROCNO 1

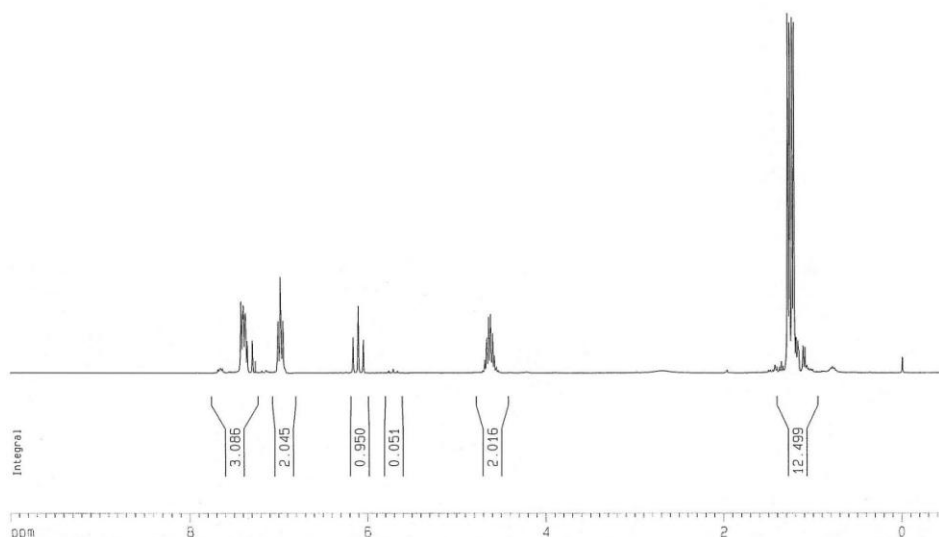
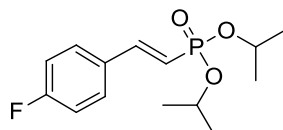
F2 - Acquisition Parameters
Date_ 20091118
Time 7.53
INSTRUM spect
PROBHD 5 mm BBI 1H-8B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 10.00 usec
TE 294.9 K
D1 2.0000000 sec
S11 0.03000000 sec
DELTA 1.89999999 sec
MCREST 0.0000000 sec
MCNRK 0.0150000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SF01 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4678235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.93 cm
F1P 200.000 ppm
F1 15093.57 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCH 10.50000 ppm/cm



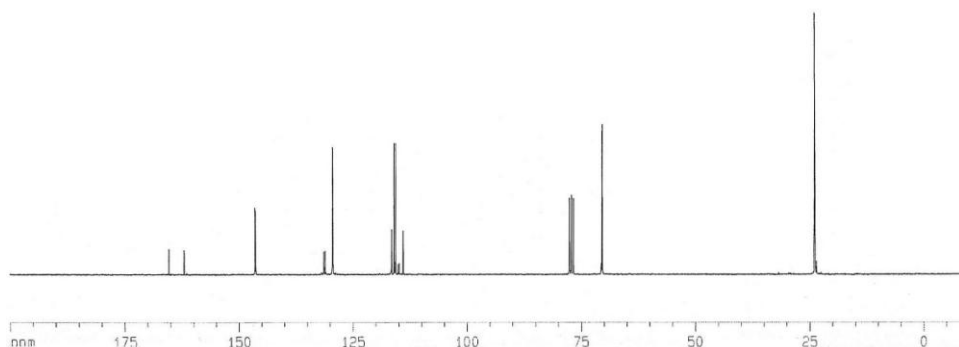
Current Data Parameters
NAME fc4Bkt105
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091123
Time 18.59
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 40.3
DW 79.200 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SF01 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EN
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 7.66 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPHCH 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME fc4Bkt105
EXPNO 11
PROCNO 1

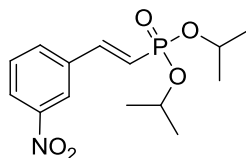
F2 - Acquisition Parameters
Date_ 20091123
Time 21.10
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 10.00 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SF01 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 5.50 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPHCH 10.50000 ppm/cm



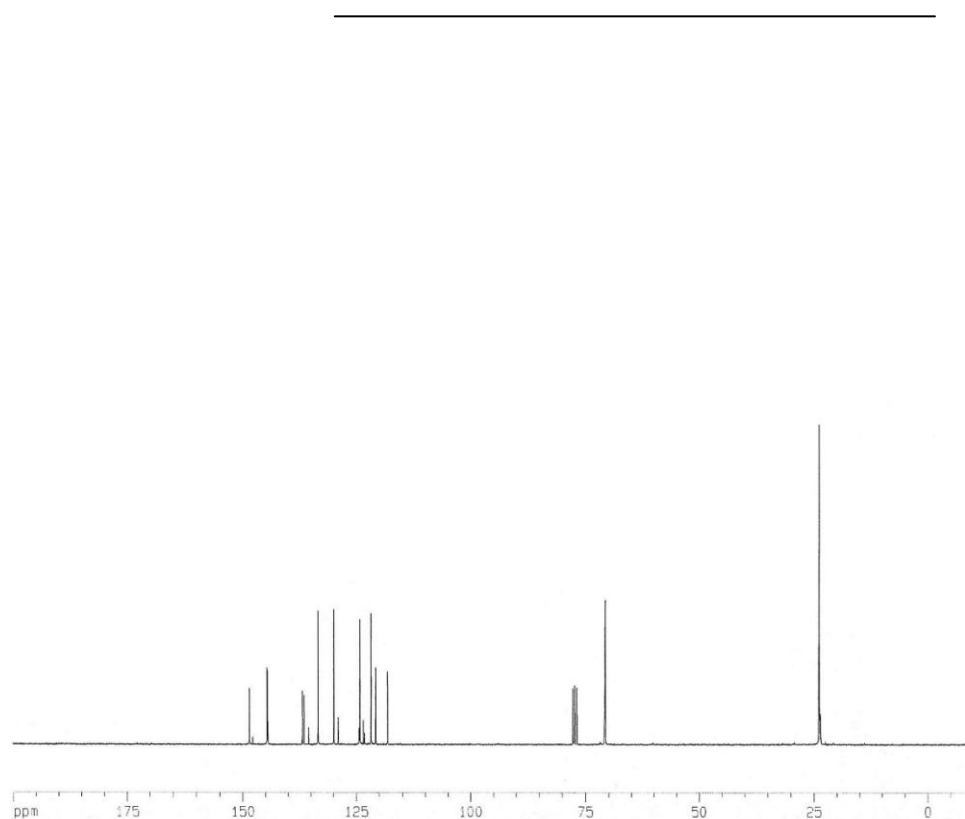
Current Data Parameters
NAME fc47kt101
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091119
Time 7.14
INSTRUM spect
PROBHD 5 mm BBI 1H-8B
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 28.5
DW 79.200 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SF01 300.1328512 MHz

F2 - Processing parameters
S1 32768
SF 300.1299932 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 6.44 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME fc47kt101
EXPNO 21
PROCNO 1

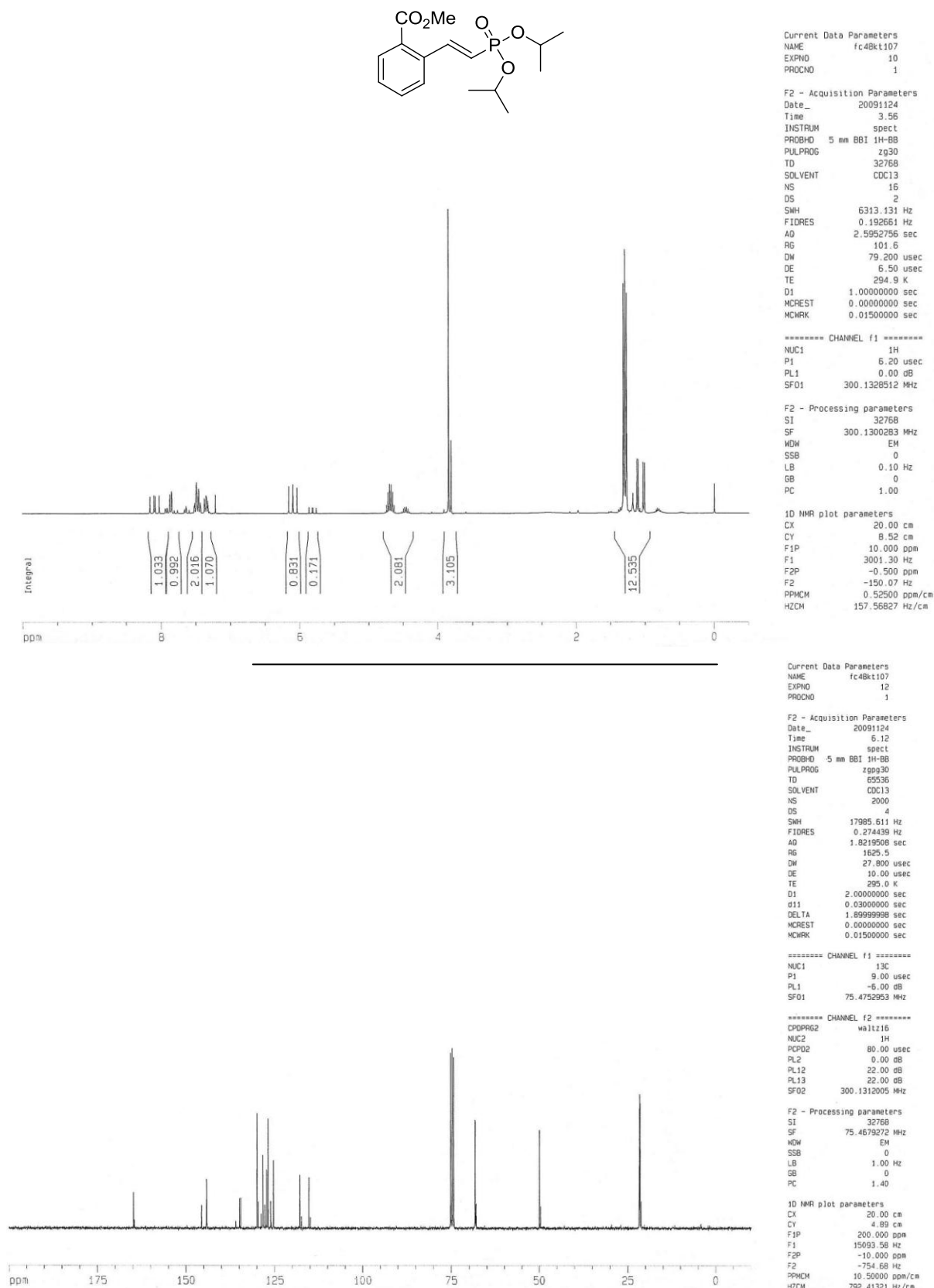
F2 - Acquisition Parameters
Date_ 20091119
Time 9.06
INSTRUM spect
PROBHD 5 mm BBI 1H-8B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 10.00 usec
TE 294.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

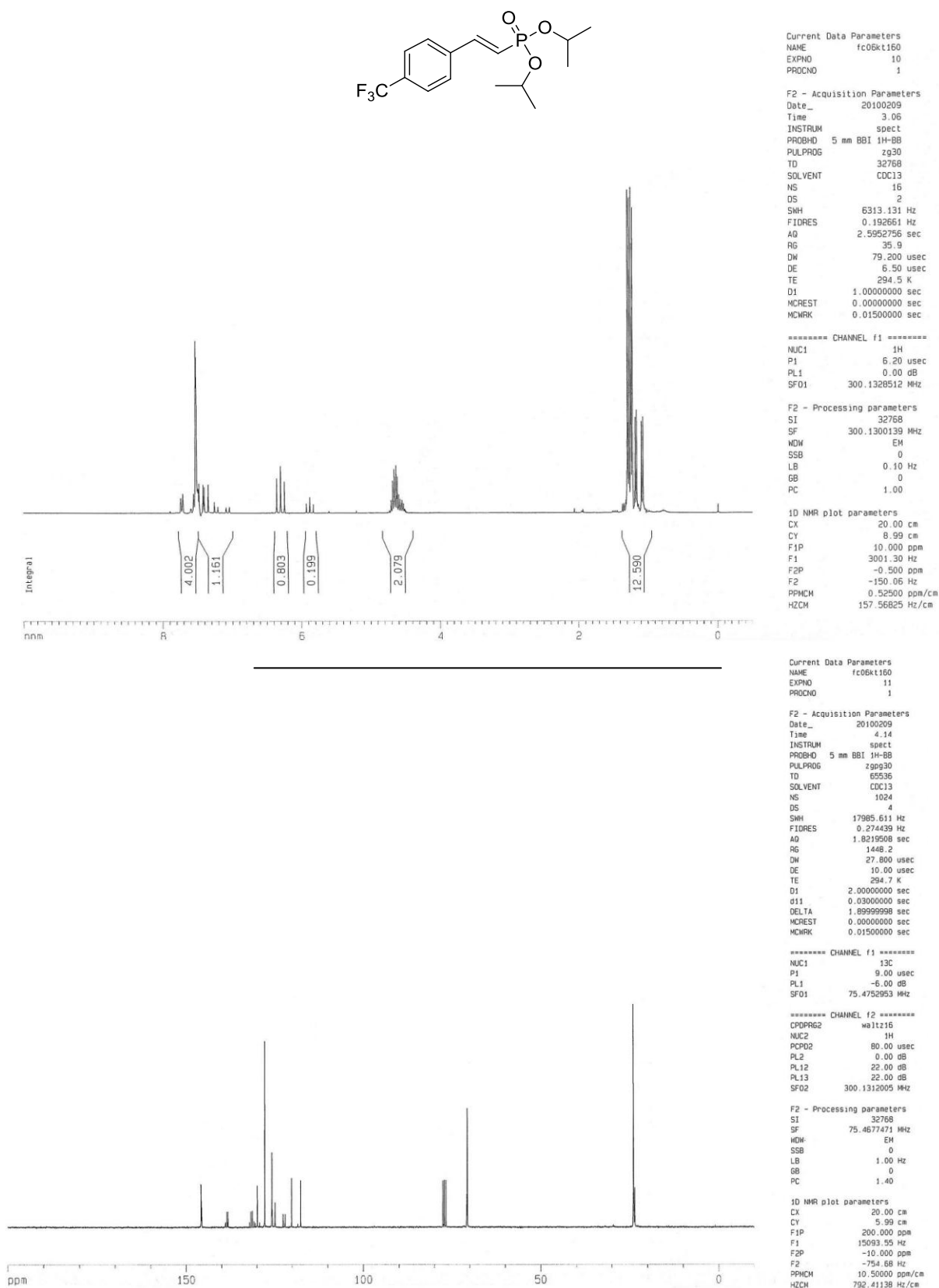
***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 75.4752953 MHz

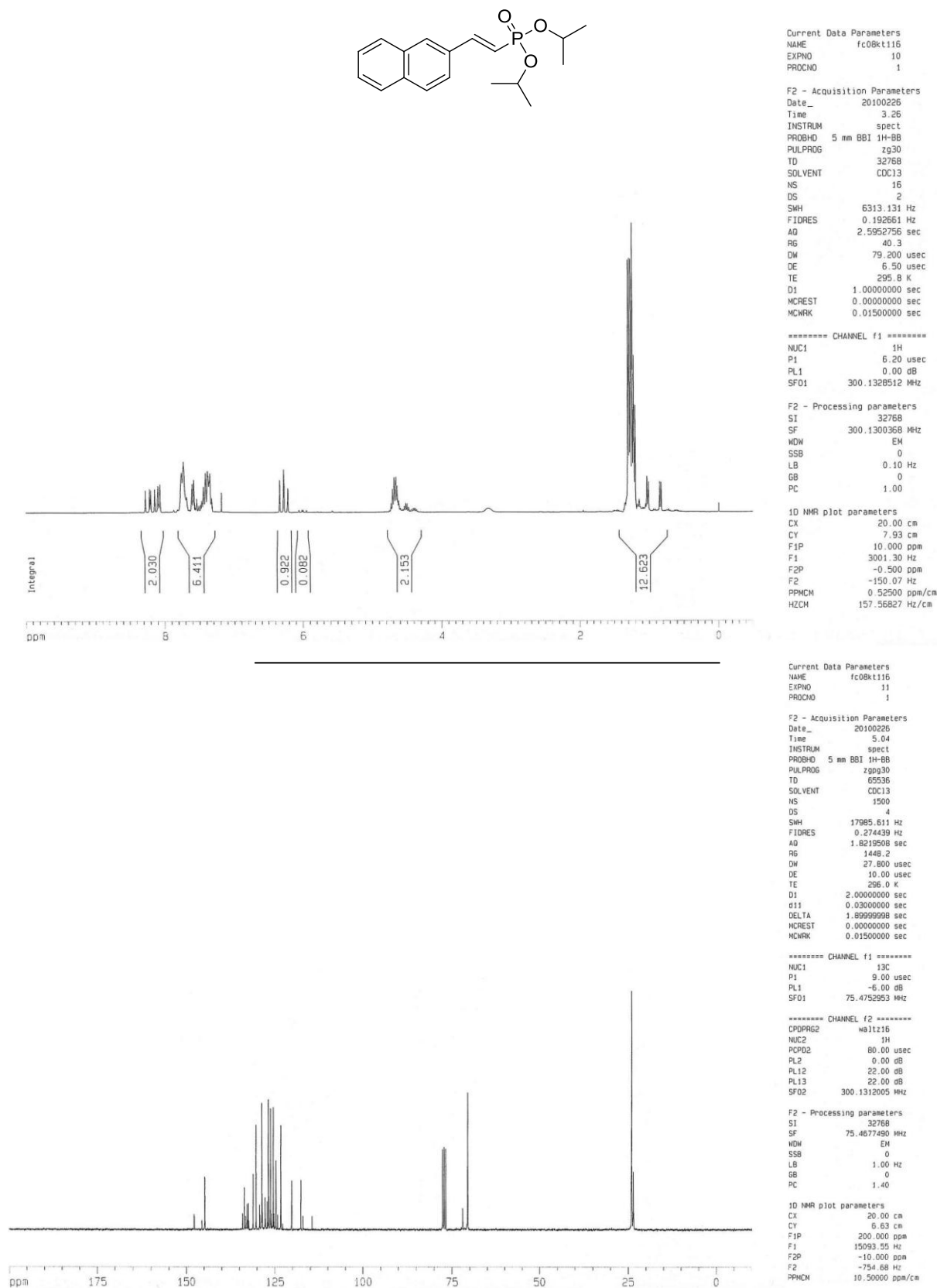
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SF02 300.1312005 MHz

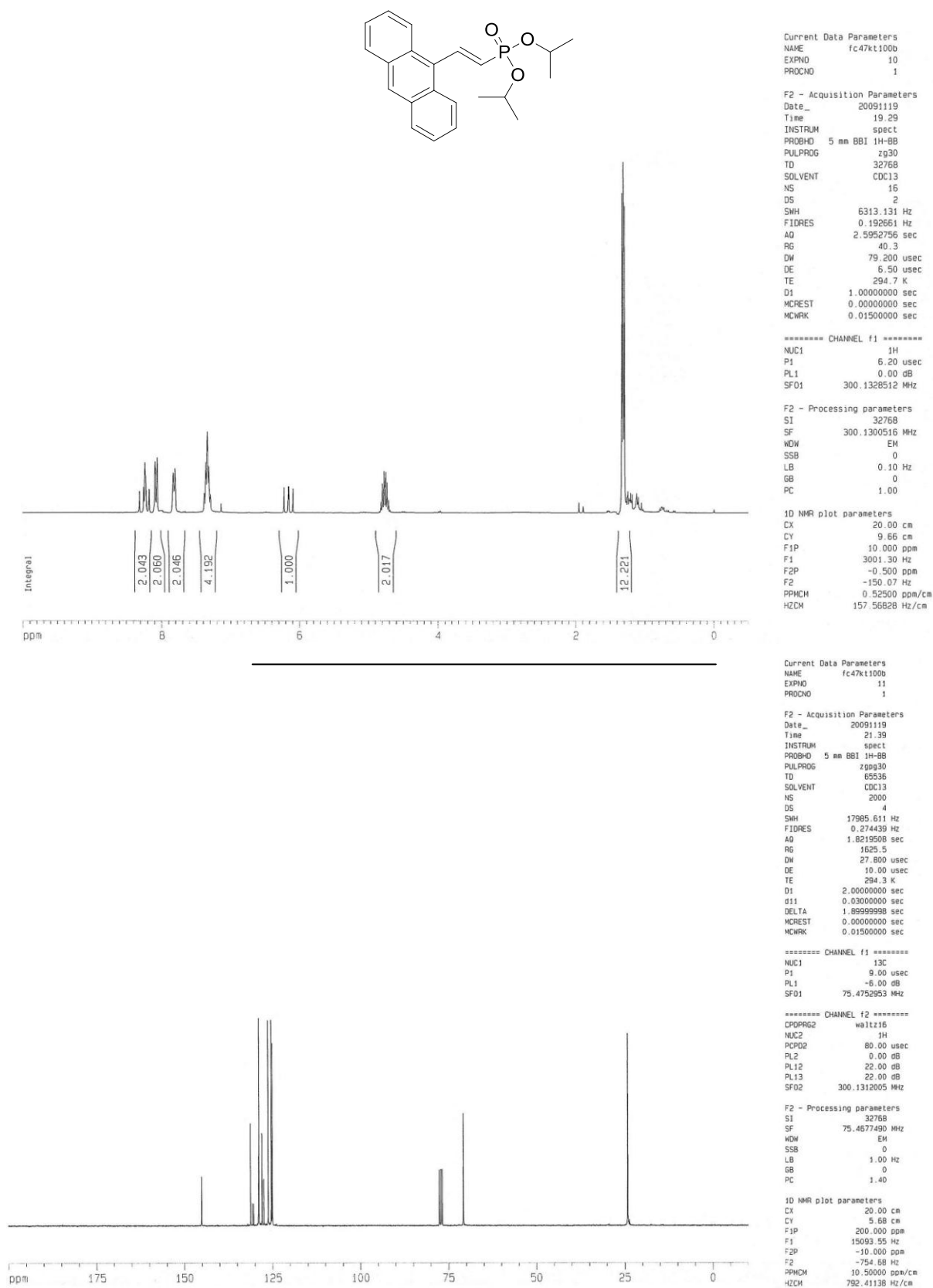
F2 - Processing parameters
S1 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

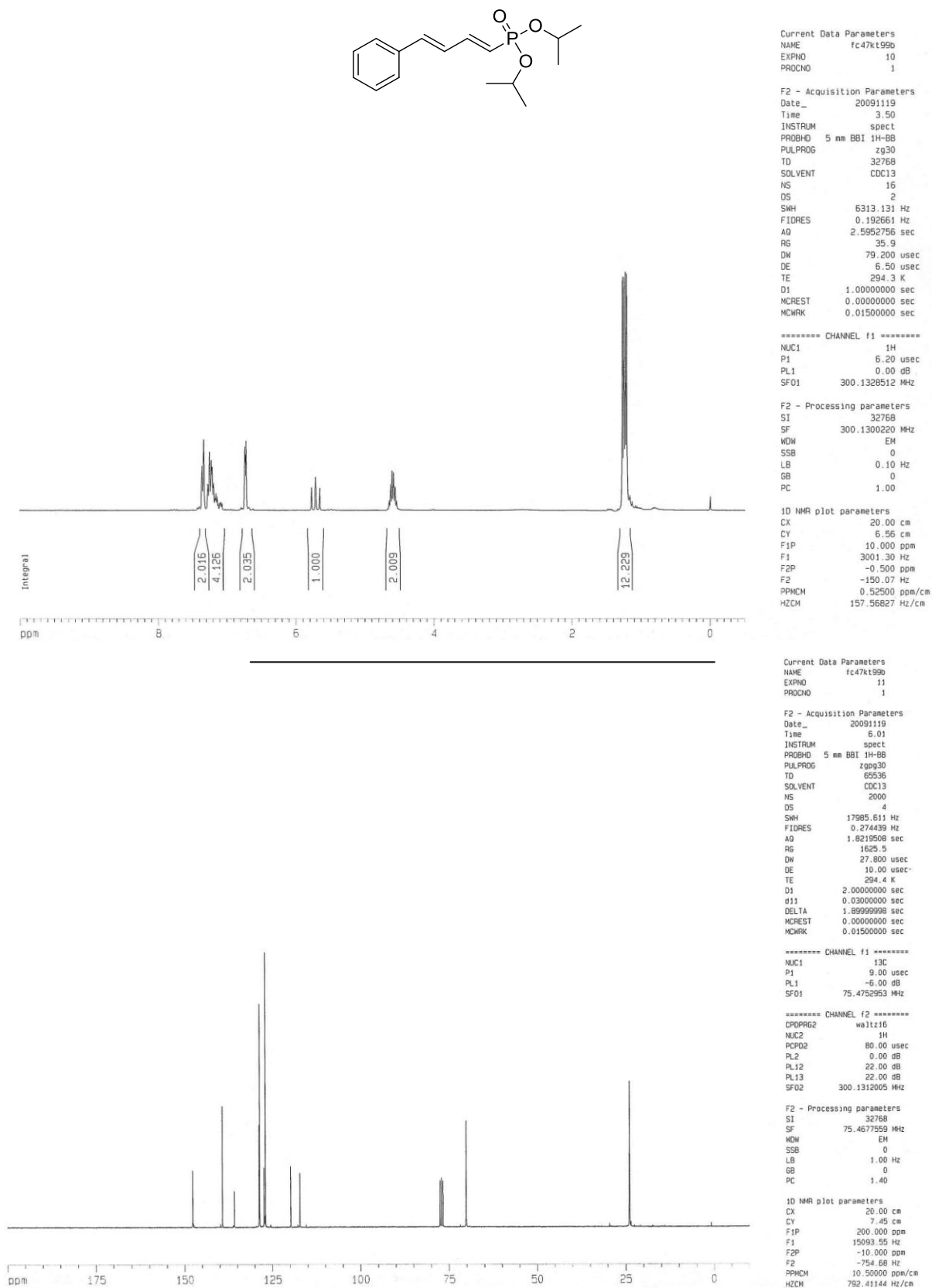
1D NMR plot parameters
CX 20.00 cm
CY 6.71 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41138 Hz/cm

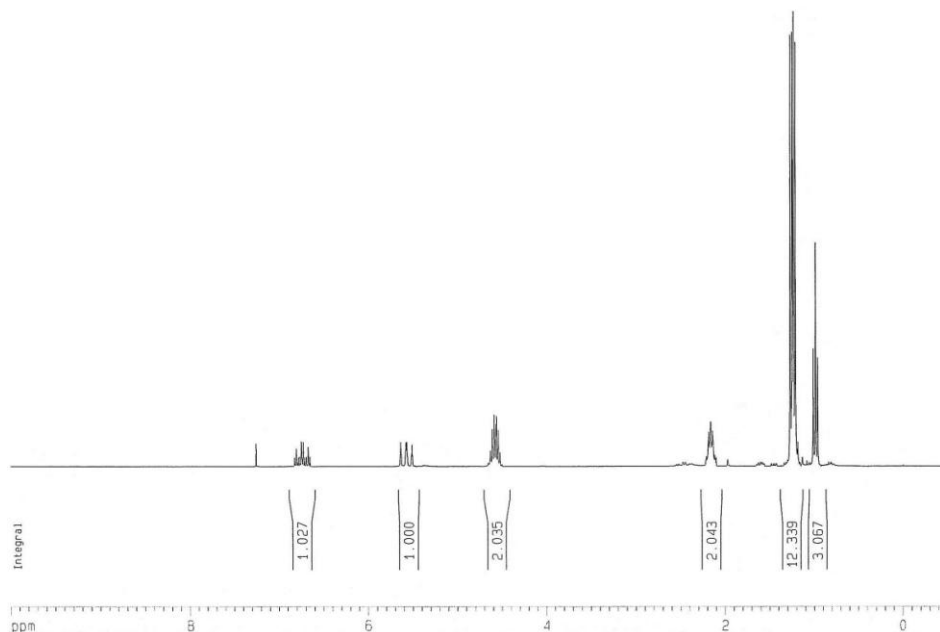
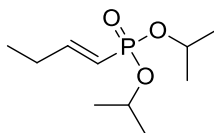












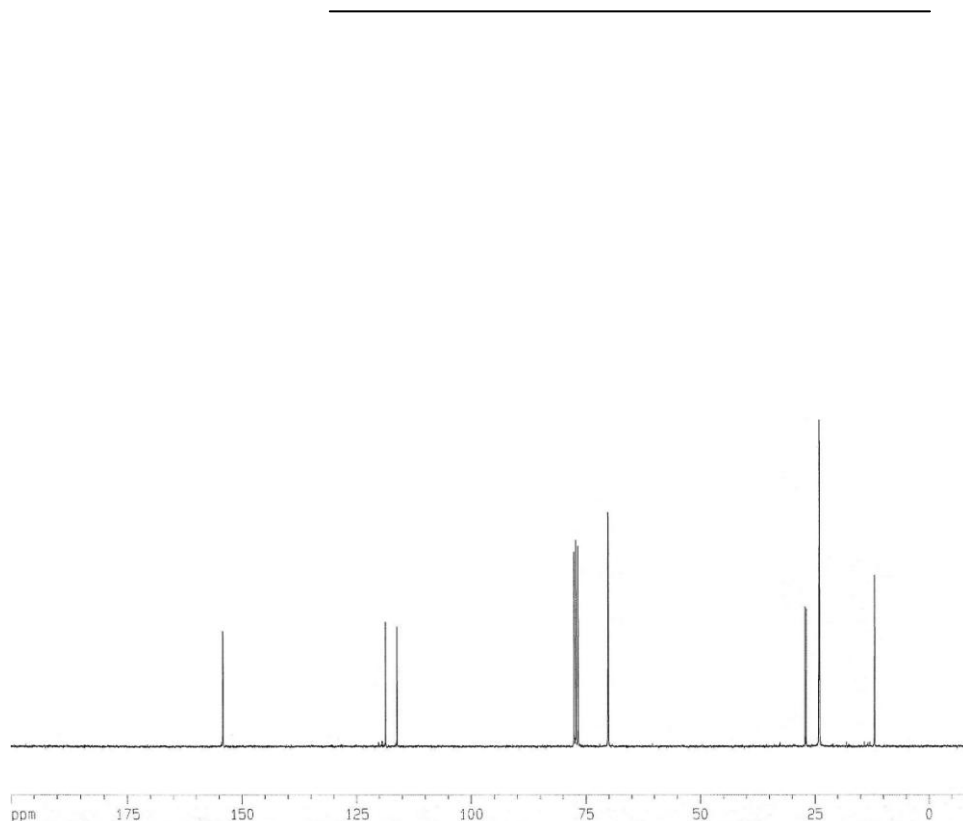
Current Data Parameters
NAME fc47kt104
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091121
Time 18.46
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 57
DW 79.200 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SF01 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300169 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 9.66 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME fc47kt104
EXPNO 12
PROCNO 1

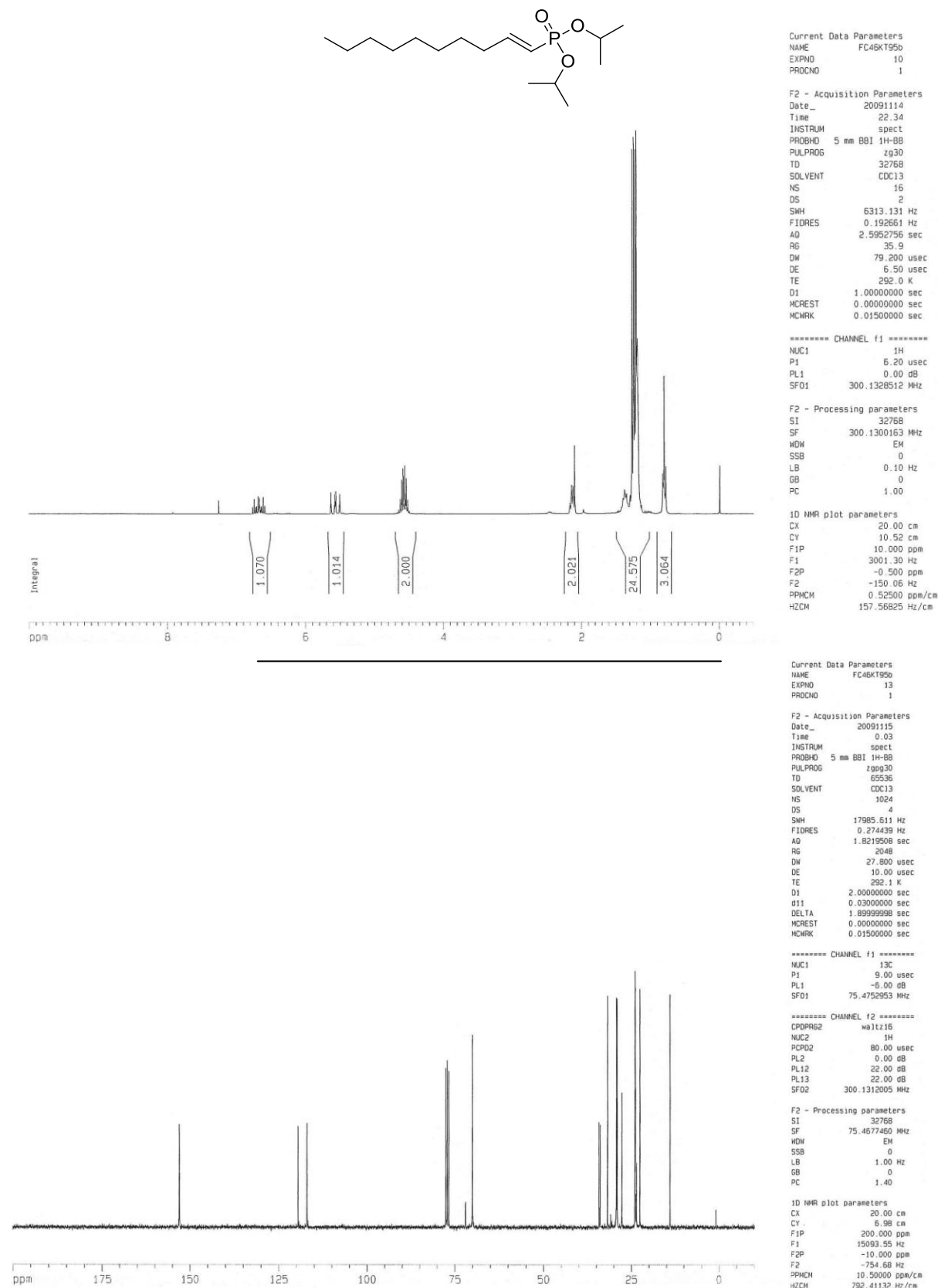
F2 - Acquisition Parameters
Date_ 20091121
Time 23.11
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 10.00 usec
TE 294.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

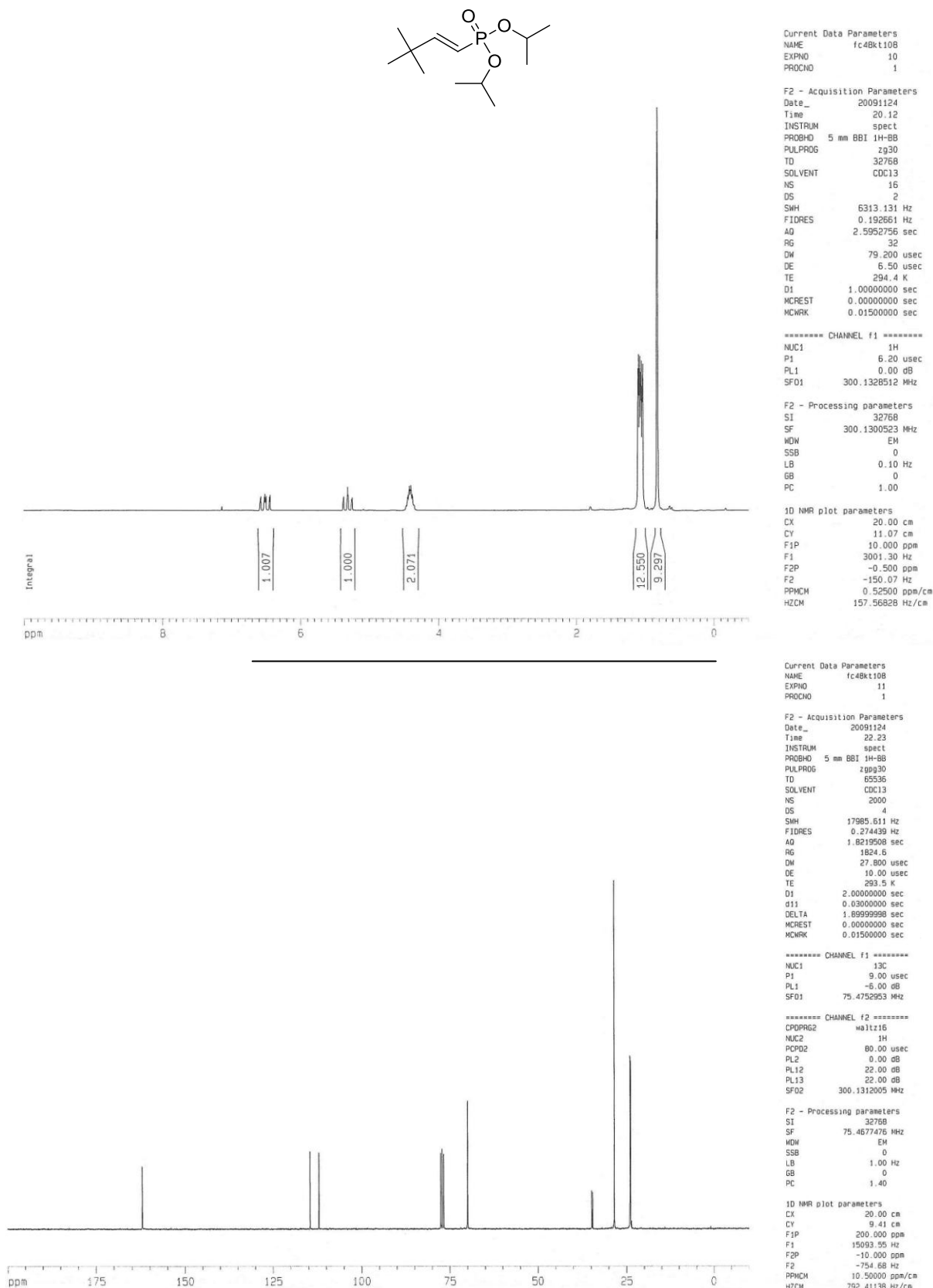
***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SF01 75.4752953 MHz

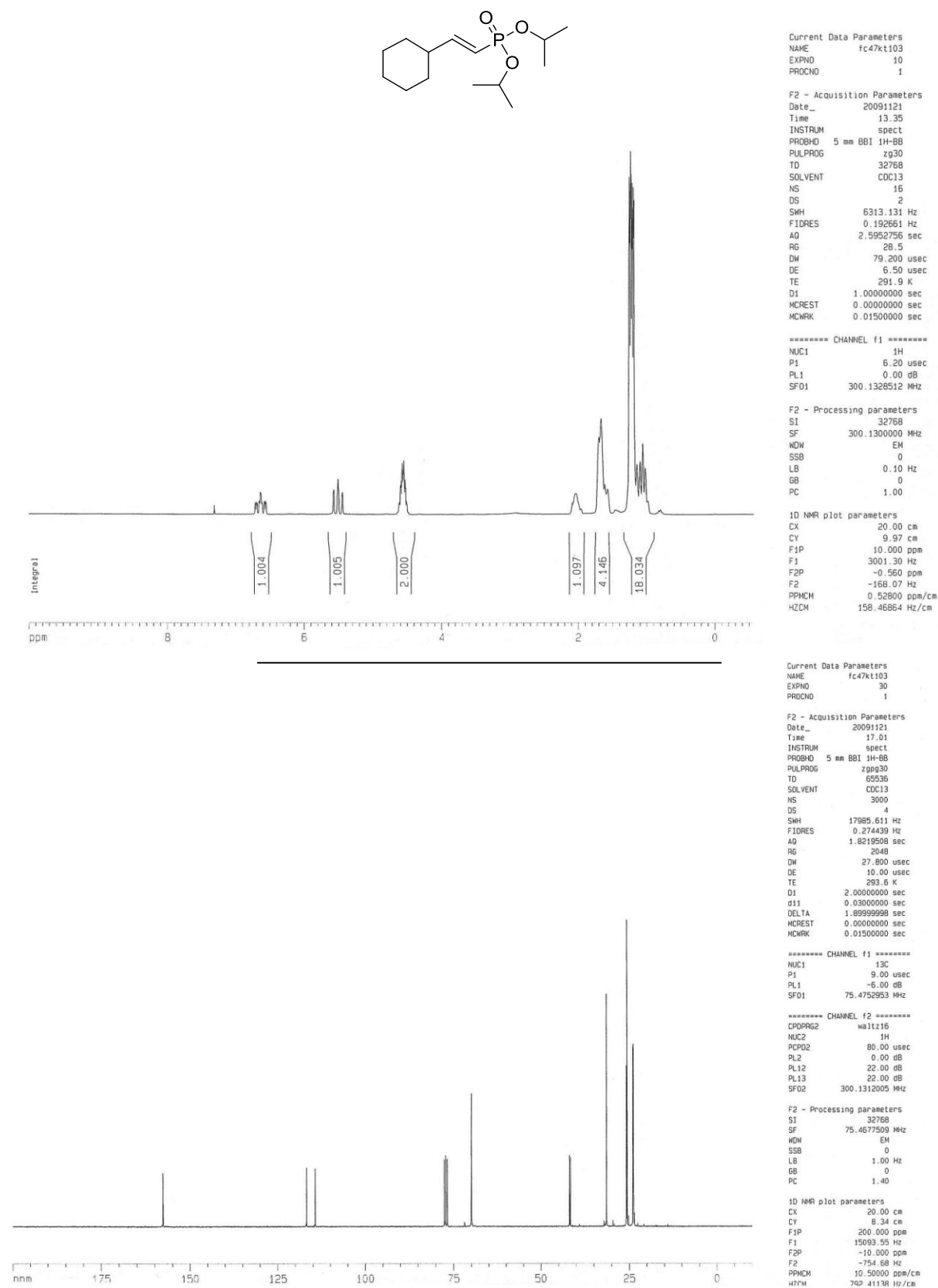
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SF02 300.1312005 MHz

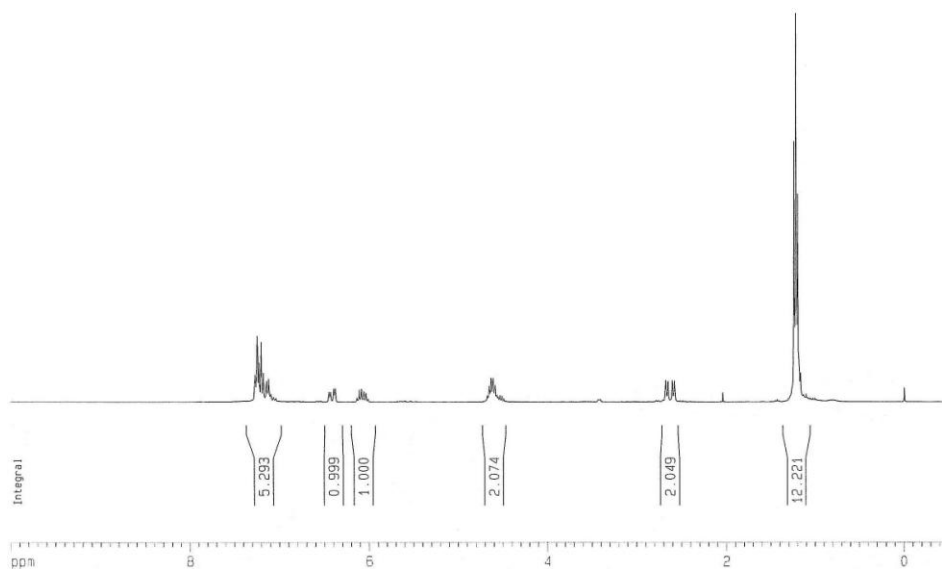
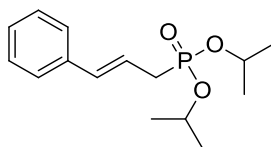
F2 - Processing parameters
SI 32768
SF 75.4677449 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.79 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -75.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41132 Hz/cm









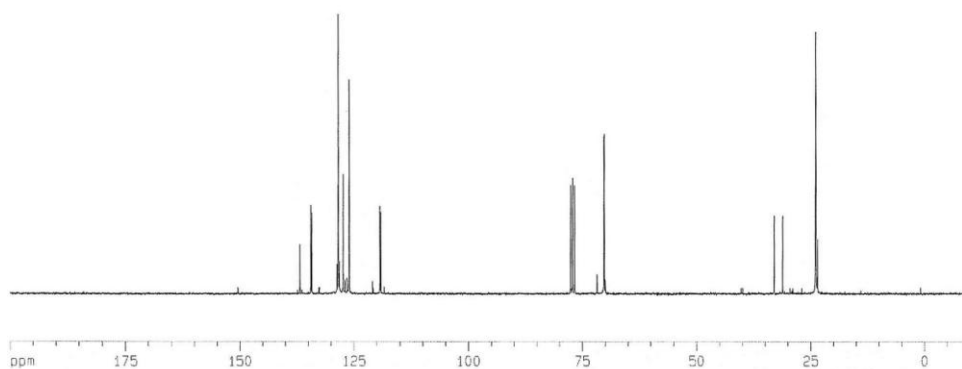
Current Data Parameters
NAME f09k1193
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100303
Time 19.49
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 32
DM 79.200 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SFO1 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300237 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 8.29 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56827 Hz/cm



Current Data Parameters
NAME f09k1193
EXPNO 30
PROCNO 1

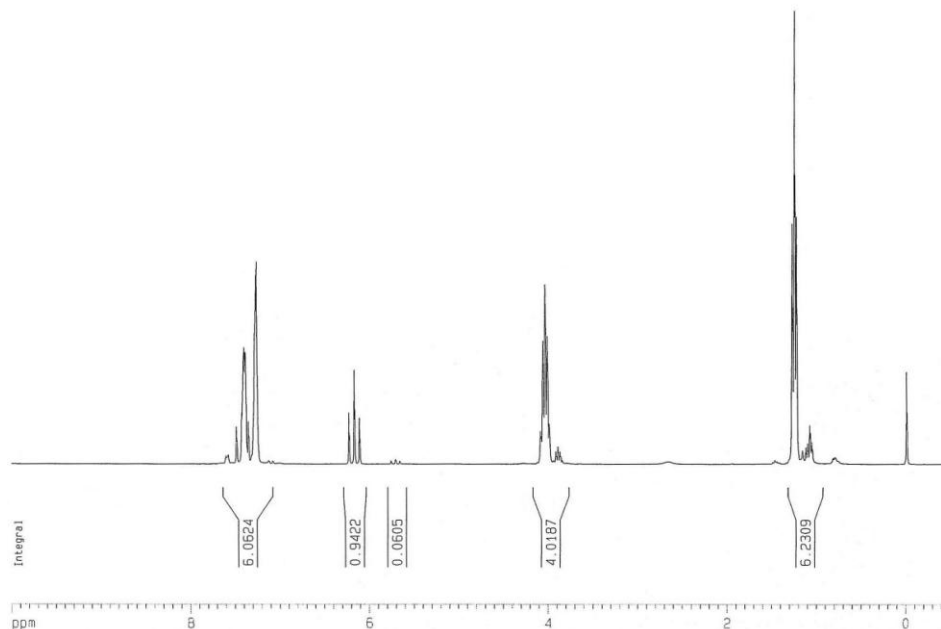
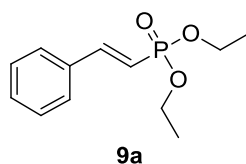
F2 - Acquisition Parameters
Date_ 20100303
Time 21.07
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SMH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1448.2
DM 27.800 usec
DE 10.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -5.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41144 Hz/cm



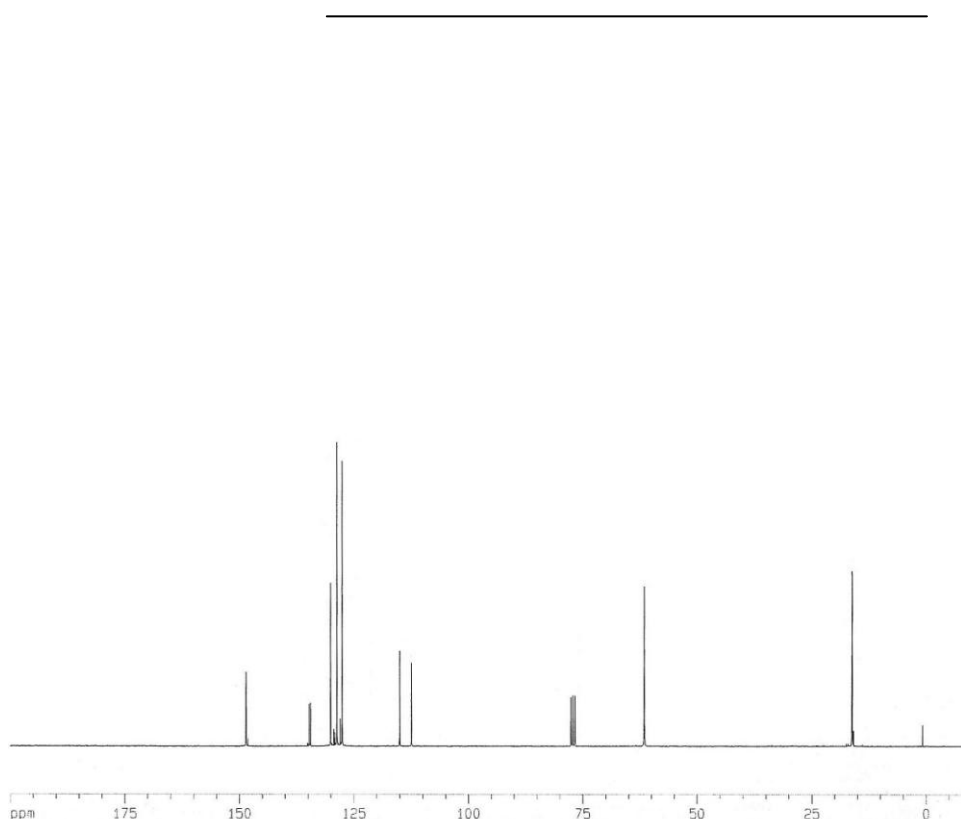
Current Data Parameters
NAME FCD1KT141
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100108
Time 18.19
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 35.9
DW 79.200 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SFO1 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300133 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 9.64 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56824 Hz/cm



Current Data Parameters
NAME FCD1KT141
EXPNO 11
PROCNO 1

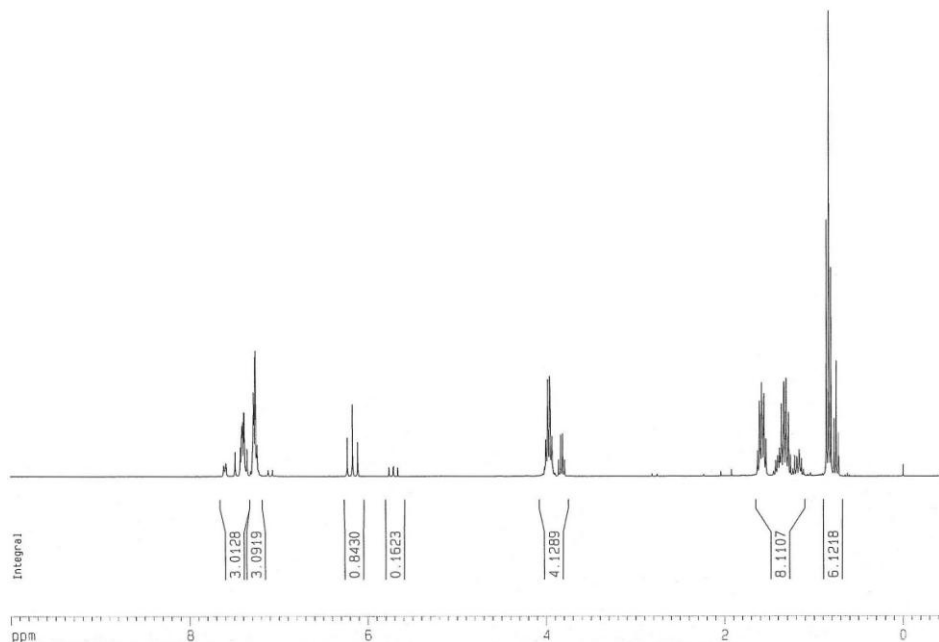
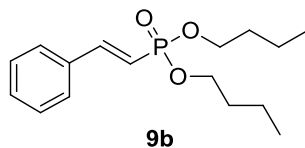
F2 - Acquisition Parameters
Date_ 20100108
Time 20.30
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 10.00 usec
TE 292.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677586 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.33 cm
F1P 200.000 ppm
F1 150093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41144 Hz/cm



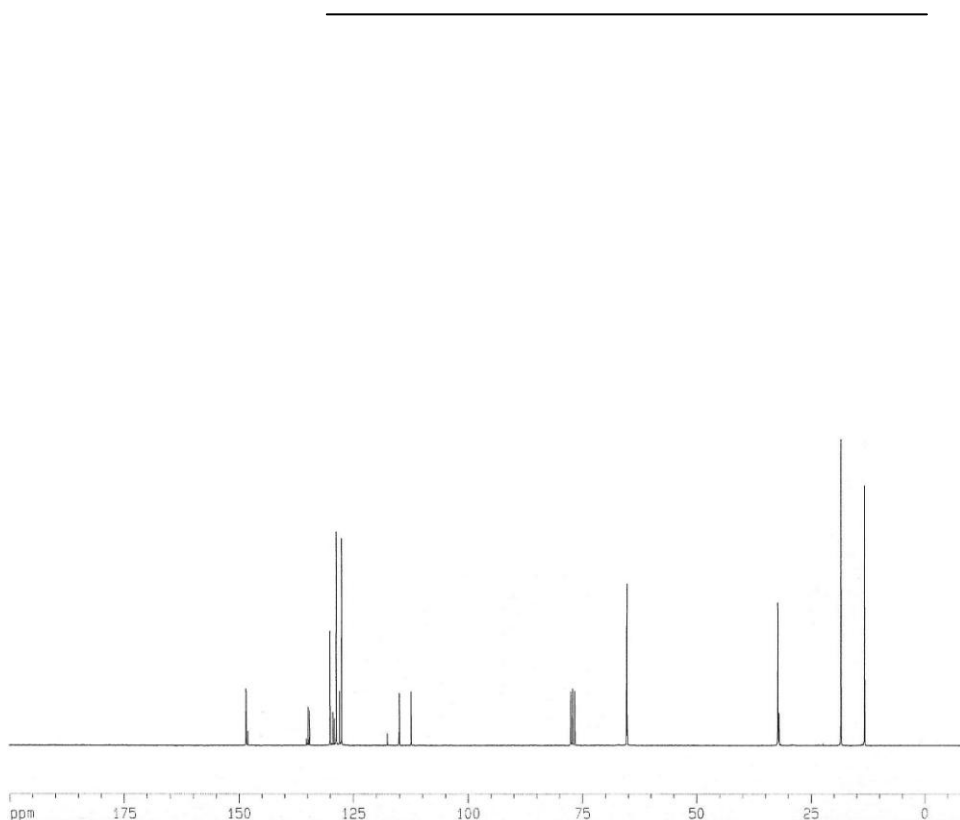
Current Data Parameters
NAME fc5ikt134
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091215
Time 21.53
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 28.5
DW 79.200 usec
DE 6.50 usec
TE 293.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SF01 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300126 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.06 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPHCH 0.52500 ppm/cm
HZCH 157.56825 Hz/cm



Current Data Parameters
NAME fc5ikt134
EXPNO 12
PROCNO 1

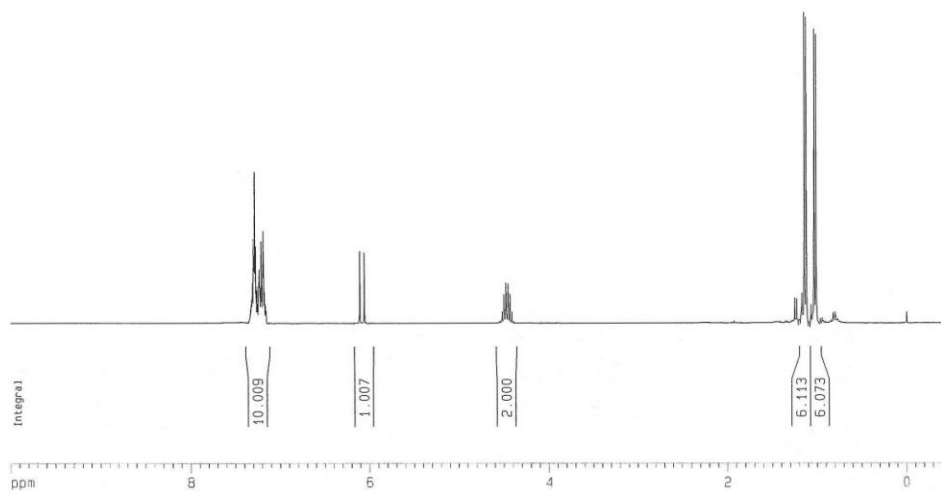
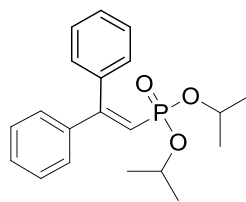
F2 - Acquisition Parameters
Date_ 20091216
Time 0.09
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1448.2
DW 27.800 usec
DE 10.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -5.00 dB
SF01 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677586 MHz
WDW EH
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.39 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPHCH 10.50000 ppm/cm
HZCH 792.41144 Hz/cm



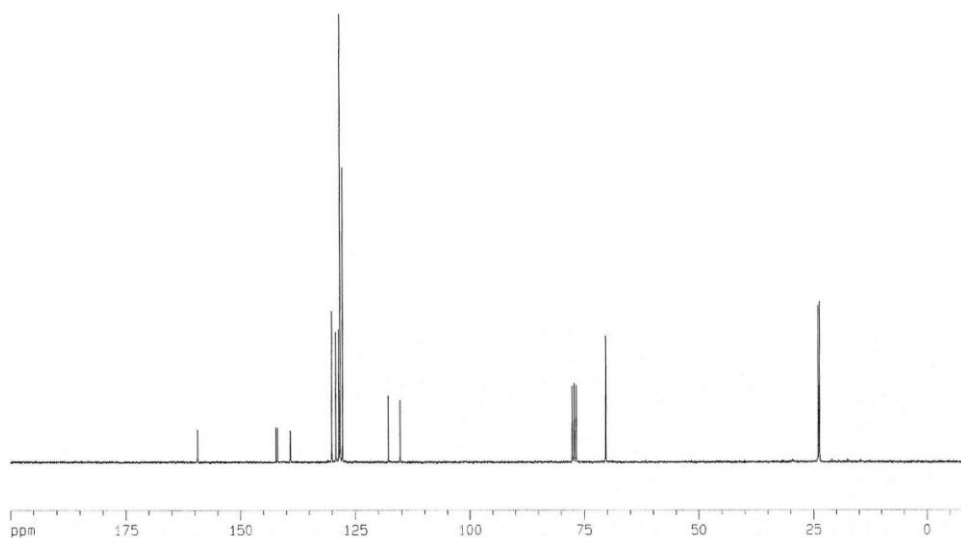
Current Data Parameters
NAME f03kt144
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100123
Time 11.24
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 50.8
DM 79.200 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SFO1 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300342 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 6.62 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56827 Hz/cm



Current Data Parameters
NAME f03kt144
EXPNO 11
PROCNO 1

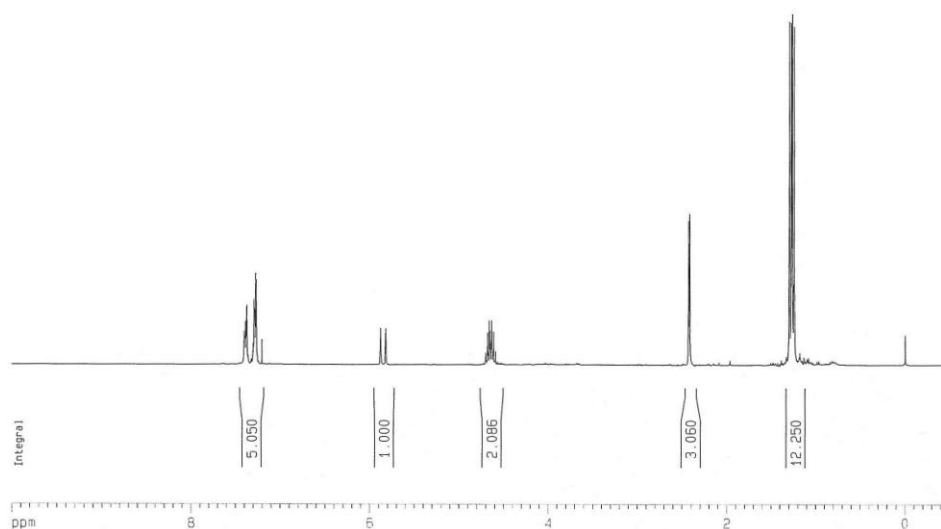
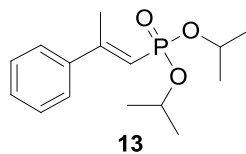
F2 - Acquisition Parameters
Date_ 20100123
Time 12.32
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DM 27.800 usec
DE 10.00 usec
TE 295.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677493 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 9.43 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41138 Hz/cm



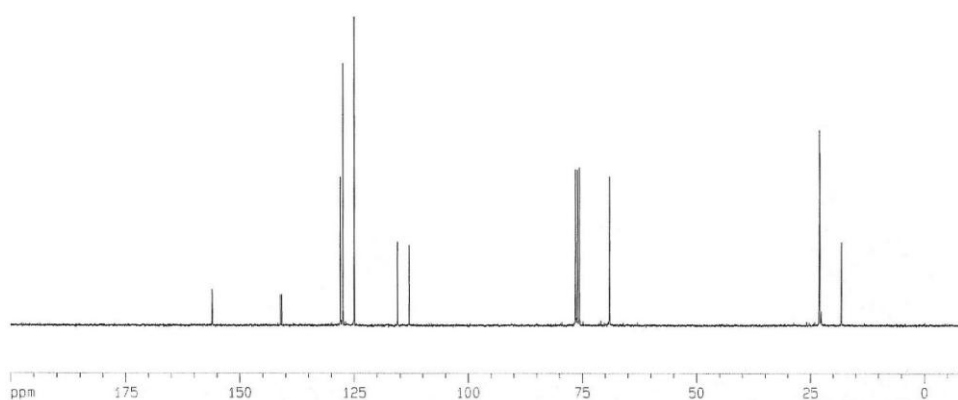
Current Data Parameters
NAME fc5ikt136a
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091219
Time 14.27
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 64
DW 79.200 usec
DE 6.50 usec
TE 293.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SF01 300.1328512 MHz

F2 - Processing parameters
SI 32768
SF 300.1300314 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 7.56 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56827 Hz/cm



Current Data Parameters
NAME fc5ikt136a
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091219
Time 16.37
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 10.00 usec
TE 293.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SF01 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4678288 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.58 cm
F1P 200.000 ppm
F1 15093.57 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41217 Hz/cm

