

The Acid Promoted Addition of Organotrifluoroborates to Unactivated Electrophiles

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Supporting Information

General Information. Commercially available organotrifluoroborates and trifluoroacetic acid were used without any further purification. Aromatic imines were prepared according to the procedure of Jacobsen¹ while enamines were prepared according to the method of Terada². All solvents were purified according to the method of Grubbs.³ Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator. Chromatographic purification of products was accomplished using flash chromatography on Silicycle 230-400 mesh silica gel. Thin-layer chromatography (TLC) was carried out on Silicycle 0.25mm silica gel plates. Visualization of the developed chromatogram was performed by fluorescence quenching or CAM staining.

¹H and ¹³C NMR spectra were recorded on Varian Mercury 300, 400 and Bruker 500 Spectrometers, and are internally referenced to residual protic solvent signals (CHCl₃ = 7.24 ppm). Data for ¹H are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), integration, coupling constant (Hz) and assignment. Data for ¹³C NMR are reported in terms of chemical shift. IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in terms of frequency of absorption (cm⁻¹). Mass spectra were obtained from the Princeton Mass Spectroscopy Facility. Gas liquid chromatography (GLC) was carried out on a Hewlett-Packard 6850 Series gas chromatograph equipped with a splitmode capillary injection system and flame ionization detectors using a Varian HP-1 column (30

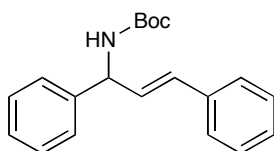
¹ Jacobsen, E. N; Wenzel, A. G. *J. Am. Chem. Soc.* **2002**, *124*, 12964.

² Terada, M.; Sorimachi, K. *J. Am. Chem. Soc.* **2007**, *129*, 292.

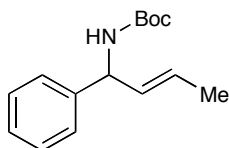
³ Pangborn, A. B; Giardello, M.A.; Grubbs, R. H.; Rosen, R.K.; Timmers, F.J. *Organometallics* **1996**, *15*, 1518.

m x 0.25 mm).

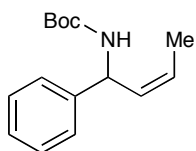
General Procedure: A 20 mL oven dried vial equipped with a magnetic stir bar was charged with imine (0.952 mmol, 2.0 equiv) and acetonitrile (4.8mL, 0.1M) was added. Potassium organotrifluoroborate (0.476 mmol, 1.0 equiv) was added followed by dropwise addition of either trifluoroacetic or dichloroacetic acid (0.476 mmol, 1.0 equiv) and the reaction was stirred at room temperature (23 °C) until completion as determined by TLC monitoring. The crude reaction mixture was poured into a separatory funnel containing NaHCO₃ (sat. aq.) and extracted with CH₂Cl₂ (3 × 25 mL). The combined organic phase was dried (Na₂SO₄) and concentrated *in vacuo*. The product was purified by silica gel chromatography (solvents noted) to yield the title compounds.



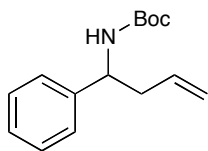
(E)-tert-butyl 1,3-diphenylallylcarbamate (Table 2, entry 1): Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (195 mg, 0.952 mmol) at 23 °C for 4 h to provide the title compound as a white solid (139 mg, 94% yield) following silica gel chromatography (5% Et₂O/petroleum ether, R_f = 0.22 in 5% Et₂O/petroleum ether). IR (film) 3376, 2976, 2926, 1680, 1502, 1456, 1362, 1295, 1234, 1163, 1156, 1025, 1039, 1012, 967, 886, 767, 738, 705 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.46 (s, 9H, -C(CH₃)₃), 4.98 (brs, 1H, -CHNH), 5.48 (brs, 1H, -CHNH), 6.33 (dd, 1H *J* = 6.4, 16 Hz, -CHCHC₆H₅), 6.55 (d, 1H, *J* = 15.6 Hz, -CHCHC₆H₅), 7.39-7.24 (m, 10H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 27.27, 28.40, 126.54, 127.01, 127.58, 127.72, 128.56, 128.75, 129.59, 130.93; HRMS (EI) exact mass calculated for (C₂₀H₂₃NO₂) requires *m/z* 332.16265, found *m/z* 332.16222 [M+Na]⁺.



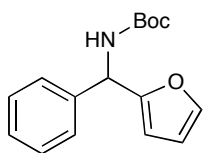
(E)-tert-butyl 1-phenylbut-2-enylcarbamate (Table 2, entry 2): Prepared according to the general procedure from potassium *trans*-methylvinyltrifluoroborate (100 mg, 0.676 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (277 mg, 1.35 mmol) at 23 °C for 4 h to provide the title compound as a white solid (105 mg, 63% yield) following silica gel chromatography (3% Et₂O/petroleum ether, R_f = 0.28 in 5% Et₂O/petroleum ether). IR (film) 3354, 2979, 2934, 1682, 1513, 1494, 1449, 1389, 1364, 1293, 1246, 1165, 1041, 1016, 966, 744 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.44 (s, 9H, -C(CH₃)₃), 1.78 (d, 3H, *J* = 4.8 Hz, -CH₃), 4.86 (brs, 1H, -CHNH), 5.22 (brs, 1H, -CHNH), 5.64-5.60 (m, 2H, -CHCHCH₃), 7.36-7.24 (m, 5H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 17.74, 18.00, 28.17, 28.39, 56.19, 125.43, 126.79, 127.25, 128.55, 131.13; HRMS (EI) exact mass calculated for (C₁₅H₂₁NO₂) requires *m/z* 270.14700 found *m/z* 270.14649 [M+Na]⁺.



(Z)-tert-butyl 1-phenylbut-2-enylcarbamate (Table 2, entry 3): Prepared according to the general procedure from potassium *cis*-methylvinyltrifluoroborate (57.0 mg, 0.385 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (158 mg, 0.770 mmol) at 23 °C for 4 h to provide the title compound as a white solid (64.9 mg, 68% yield) following silica gel chromatography (3% Et₂O/petroleum ether, R_f = 0.28 in 5% Et₂O/petroleum ether). IR (film) 3356, 2976, 2920, 1688, 1510, 1389, 1360, 1230, 1165, 1040, 1018, 799, 755, 697 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 1.44 (s, 9H, -C(CH₃)₃), 1.77 (d, 3H, *J* = 6.3 Hz, -CH₃), 4.83 (brs, 1H, -CHNH), 5.46-5.70 (m, 3H, -CHNH, -CHCHCH₃), 7.22-7.36 (m, 5H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 13.40, 17.74, 28.41, 126.34, 126.80, 127.14, 128.62, 130.74; HRMS (EI) exact mass calculated for (C₁₅H₂₁NO₂) requires *m/z* 270.14700 found *m/z* 270.14636 [M+Na]⁺.

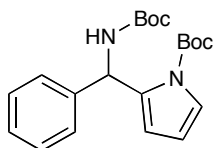


tert-butyl 1-phenylbut-3-enylcarbamate (Table 2, entry 4): To a solution of potassium allyltrifluoroborate (60.0 mg, 0.405 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (108 mg, 0.527 mmol) in acetonitrile (8.1mL, 0.05M) at 0 °C was added dropwise trifluoroacetic acid (30 μ L, .405 mmol). The reaction was let come to 23 °C and stirred for 1 h to provide the title compound as a white solid (85.0 mg, 85% yield) following standard workup procedure and silica gel chromatography (5% Et₂O/hexanes, R_f = 0.35 in 5% Et₂O/petroleum ether). ¹H NMR and ¹³C NMR data in accord with literature values.⁴



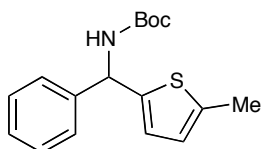
tert-butyl furan-2-yl(phenyl)methylcarbamate (Table 2, entry 5): Prepared according to the general procedure from potassium 2-furantrifluoroborate (127.1 mg, 0.731 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (100 mg, 0.487 mmol) in acetonitrile (1.9mL, 0.25M) at 23 °C for 24 h to provide the title compound as a white solid (109 mg, 82% yield) following silica gel chromatography (5% Et₂O/petroleum ether, R_f = 0.26 in 5% Et₂O/petroleum ether). IR (film) 3367, 2979, 2932, 1687, 1515, 1499, 1457, 1389, 1368, 1299, 1241, 1168, 1149, 1130, 1076, 1043, 1021, 1006, 946, 872, 753 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.41 (s, 9H, -C(CH₃)₃), 5.96 (brd, 1H, *J* = 8.5 Hz, -CHNH), 6.17 (d, 1H, *J* = 3.0 Hz, ArH), 6.35 (t, 1H, *J* = 3.0 Hz, ArH), 6.90 (brs, 1H, -CHNH), 7.30 (m, 1H, ArH), 7.33-7.38 (m, 4H, ArH), 7.47 (s, 1H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.38, 52.66, 107.32, 110.24, 126.93, 127.74, 128.62, 142.39; HRMS (EI) exact mass calculated for (C₁₆H₁₉NO₃) requires *m/z* 273.13649 found *m/z* 273.13719 [M+Na]⁺.

⁴ Vilaivan, T.; Winotapan, C.; Banphavichit, V.; Shinada, T.; Ohfune, Y. *J. Org. Chem.* **2005**, *70*, 3464.



***tert*-butyl-2[(*tert*-butyl-carbonylamino)-phenylmethyl]-1H-pyrrole-1-carboxylate**

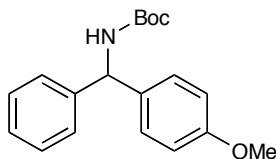
(Table 2, entry 6): Prepared according to the general procedure from potassium 2-*N*-Boc-pyrroletetrafluoroborate (96.1 mg, 0.352 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (108 mg, 0.528 mmol) in acetonitrile (2.3 mL, 0.15M) at $-20\text{ }^{\circ}\text{C}$ for 24 h to provide the title compound as a white solid (95.4 mg, 73% yield) following silica gel chromatography (5% Et₂O/petroleum ether, R_f = 0.20 in 5% Et₂O/petroleum ether). IR (film) 3434, 2976, 2933, 1738, 1714, 1486, 1368, 1332, 1258, 1164, 1142, 1113, 1066, 1044, 1018, 882, 847, 770, 728, 700 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.39 (s, 9H, -C(CH₃)₃), 1.48 (s, 9H, -C(CH₃)₃), 5.75 (brs, 1H, -CHNH), 6.15 (t, 1H, *J* = 3.2 Hz, ArH), 6.26 (brs, 1H, -CHNH), 6.38 (brd, 1H, *J* = 9.2 Hz, ArH), 7.12-7.29 (m, 6H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 27.69, 27.97, 28.48, 52.26, 109.95, 114.26, 122.56, 126.52, 126.93, 128.16, 132.15; HRMS (EI) exact mass calculated for (C₂₁H₂₈N₂O₂) requires *m/z* 411.16861 found *m/z* 411.18898 [M+K]⁺.



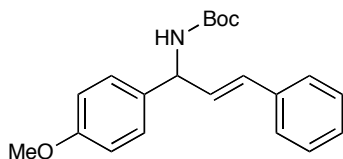
***tert*-butyl(5-methylthiophen-2-yl)(phenyl)methylcarbamate**

(Table 2, entry 7): Prepared according to the general procedure from potassium 2-(5-methyl)thiophenetrifluoroborate (102.8 mg, 0.501 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (154.2 mg, 0.751 mmol) at $-20\text{ }^{\circ}\text{C}$ for 24 h to provide the title compound as a white solid (120.4 mg, 79% yield) following silica gel chromatography (5% Et₂O/petroleum ether, R_f = 0.32 in 5% Et₂O/petroleum ether). IR (film) 3383, 2976, 2933, 1679, 1510, 1456, 1366, 1295, 1249, 1165, 1153, 1011, 869, 842, 757, 712 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.45 (s, 9H, -C(CH₃)₃), 2.43 (s, 3H, -CH₃), 5.22 (brs, 1H, -CHNH), 6.03 (brs, 1H, -CHNH), 6.57 (m, 2H, ArH), 7.27-7.37 (m, 5H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 15.36, 28.37, 54.57, 119.72, 124.74, 125.10, 125.30, 126.82,

126.97, 127.40, 127.65, 128.59; HRMS (EI) exact mass calculated for (C₁₇H₂₁NO₂S) requires m/z 326.11907 found m/z 326.11847 [M+Na]⁺.



tert-butyl (4-methoxyphenyl)(phenyl)-1-methylcarbamate (Table 2, entry 8): To a solution of potassium 4-methoxyphenyltrifluoroborate (192.0 mg, 0.934 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (100 mg, 0.467 mmol) at 23 °C was added dropwise dichloroacetic acid (115 μL, 1.40 mmol). The reaction was stirred for 24 h to provide the title compound as a white solid (115.7 mg, 79% yield) following standard workup procedure and silica gel chromatography (5% Et₂O/hexanes, R_f = 0.22 in 5% Et₂O/petroleum ether). ¹H NMR and ¹³C NMR data in accord with literature values.⁵

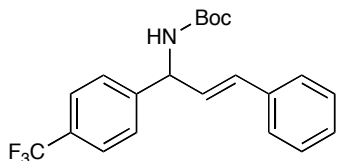


(E)-tert-butyl 1-(4-methoxyphenyl)-3-phenylallylcarbamate (Table 3, entry 1):

Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and 4-methoxybenzaldehyde *N*-(*tert*-butoxycarbonyl)imine (224 mg, 0.952 mmol) at 23 °C for 24 h to provide the title compound as a white solid (126.6 mg, 78% yield) following silica gel chromatography (5–10% Et₂O/petroleum ether, R_f = 0.22 5% Et₂O/petroleum ether). IR (film) 3362, 2978, 2932, 2834, 1682, 1608, 1507, 1461, 1447, 1364, 1298, 1161, 1030, 965, 885, 857, 830, 748, 727, 694 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.46 (s, 9H, -C(CH₃)₃), 3.82 (s, 3H, -OCH₃), 4.93 (brs, 1H, -CHNH), 5.43 (brs, 1H, -CHNH), 6.32 (dd, 1H *J* = 5, 15 Hz, -CHCHC₆H₅), 6.53 (d, 1H, *J* = 15 Hz, -CHCHC₆H₅), 6.90 (d, 2H, *J* = 5Hz, ArH), 7.31-7.23 (m, 5H, ArH), 7.37-7.39 (m, 2H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.45, 55.36, 114.12, 114.39, 126.55, 127.67, 128.25, 128.58, 129.82, 130.66, 132.05, 133.55, 136.67, 159.03; HRMS (EI)

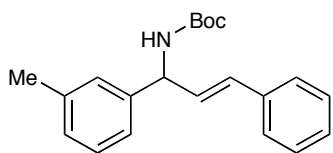
⁵ Ellman, J. A.; Nakagawa, H.; Rech, J. C.; Sindelar, R. W. *Org. Lett.* **2007**, *9*, 5155.

exact mass calculated for (C₂₁H₂₅NO₃) requires *m/z* 339.18344, found *m/z* 362.17285 [M+Na]⁺.



(E)-tert-butyl 1-(4-trifluoromethylphenyl)-3-phenylallylcarbamate (Table 3, entry 2):

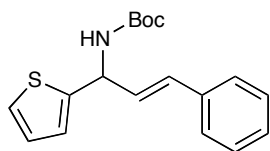
Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and 4-trifluoromethylbenzaldehyde *N*-(*tert*-butoxycarbonyl)imine (260 mg, 0.952 mmol) at 23 °C for 4 h to provide the title compound as a white solid (148.9 mg, 83% yield) following silica gel chromatography (5–10% Et₂O/petroleum ether, R_f = 0.24 5% Et₂O/petroleum ether). IR (film) 3363, 2980, 1683, 1619, 1513, 1448, 1421, 1390, 1366, 1323, 1296, 1158, 1120, 1108, 1067, 1017, 961, 896, 867, 840, 780, 767, 689 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.48 (s, 9H, -C(CH₃)₃), 5.05 (brs, 1H, -CHNH), 5.54 (brs, 1H, -CHNH), 6.33 (dd, 1H *J* = 20, 10 Hz, -CHCHC₆H₅), 6.55 (d, 1H, *J* = 20 Hz, -CHCHC₆H₅), 7.28 (m, 1H, ArH), 7.34 (m, 2H, ArH), 7.39 (m, 2H, ArH), 7.50 (d, 2H, *J* = 10 Hz, ArH), 7.65 (d, 2H, *J* = 10 Hz, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.38, 56.10, 65.92, 125.71, 125.74, 126.58, 127.23, 128.11, 128.40, 128.68, 132.13; HRMS (EI) exact mass calculated for (C₂₁H₂₂F₃NO₂) requires *m/z* 377.16026, found *m/z* 400.14942 [M+Na]⁺.



(E)-tert-butyl 1-(3-methylphenyl)-3-phenylallylcarbamate (Table 3, entry 3):

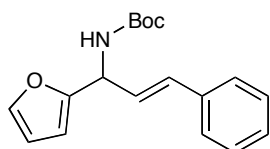
Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and 3-methylbenzaldehyde *N*-(*tert*-butoxycarbonyl)imine (209 mg, 0.952 mmol) at 23 °C for 4 h to provide the title compound as a white solid (145.6 mg, 95% yield) following silica gel chromatography (5–10% Et₂O/petroleum ether, R_f =

0.25 in 5% Et₂O/petroleum ether). IR (film) 3343, 2978, 2933, 1678, 1606, 1511, 1448, 1391, 1366, 1333, 1281, 1252, 1164, 1040, 1017, 968, 865, 836, 780, 765, 736, 694 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.46 (s, 9H, -C(CH₃)₃), 2.36 (s, 3H, -CH₃), 4.96 (brs, 1H, -CHNH), 5.43 (brs, 1H, -CHNH), 6.32 (dd, 1H *J* = 5, 15 Hz, -CHCHC₆H₅), 6.55 (d, 1H, *J* = 15 Hz, -CHCHC₆H₅), 7.11 (m, 1H, ArH), 7.15 (m, 2H, ArH), 7.25 (m, 2H, ArH), 7.32 (m, 2H, ArH), 7.39 (m, 2H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 21.52, 28.43, 56.31, 65.92, 124.02, 126.56, 127.69, 127.77, 128.37, 128.57, 128.68, 129.72, 130.66; HRMS (EI) exact mass calculated for (C₂₁H₂₅NO₂) requires *m/z* 323.18853, found *m/z* 346.17758 [M+Na]⁺.



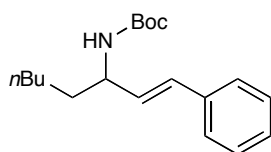
(E)-tert-butyl 1-(2-thiophenyl)-3-phenylallylcarbamate (Table 3, entry 4):

Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and thiophene-2-carboxaldehyde *N*-(*tert*-butoxycarbonyl)imine (203 mg, 0.952 mmol) at 23 °C for 24 h to provide the title compound as a white solid (134.1 mg, 99% yield) following silica gel chromatography (3–10% Et₂O/petroleum ether, R_f = 0.30 in 5% Et₂O/petroleum ether). IR (film) 3348, 2979, 2932, 1692, 1493, 1390, 1366, 1300, 1234, 1155, 1045, 1017, 964, 878, 846, 827, 765, 752, 742 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.48 (s, 9H, -C(CH₃)₃), 5.02 (brs, 1H, -CHNH), 5.71 (brs, 1H, -CHNH), 6.36 (dd, 1H *J* = 6, 16 Hz, -CHCHC₆H₅), 6.65 (d, 1H, *J* = 16 Hz, -CHCHC₆H₅), 6.98 (m, 2H, ArH), 7.25 (m, 2H, ArH), 7.33 (m, 2H, ArH), 7.40 (m, 2H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.41, 52.12, 124.77, 124.93, 126.68, 127.03, 127.91, 128.62, 128.67, 131.31, 136.37, 145.45, 154.79; HRMS (EI) exact mass calculated for (C₁₈H₂₁NO₂S) requires *m/z* 315.1293, found *m/z* 315.12976 [M+Na]⁺.

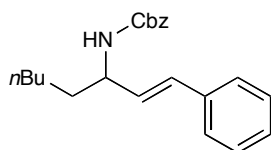


(E)-tert-butyl 1-(2-furanyl)-3-phenylallylcarbamate (Table 3, entry 5):

Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (53.8 mg, 0.256 mmol) and furfural *N*-(*tert*-butoxycarbonyl)imine (75.0 mg, 0.384 mmol) at $-10\text{ }^{\circ}\text{C}$ for 5 h to provide the title compound as a white solid (49.9 mg, 65% yield) following silica gel chromatography (10–20% Et₂O/petroleum ether, $R_f = 0.20$ in 10% Et₂O/petroleum ether). IR (film) 3360, 2975, 2933, 1685, 1513, 1499, 1389, 1364, 1308, 1240, 1163, 1152, 1076, 1044, 1011, 962, 950, 877, 862, 692 cm^{-1} ; ¹H NMR (500 MHz, CDCl₃) δ 1.48 (s, 9H, -C(CH₃)₃), 5.05 (brs, 1H, -CHNH), 5.59 (brs, 1H, -CHNH), 6.25 (d, 1H, $J = 4$ Hz, ArH) 6.30-6.35 (m, 2H, ArH, -CHCHC₆H₅), 6.58 (d, 1H, $J = 16$ Hz, -CHCHC₆H₅), 7.25 (d, 1H, $J = 7$ Hz, ArH), 7.32 (t, 2H, $J = 7$ Hz, ArH), 7.39 (m, 3H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.42, 106.63, 110.34, 126.64, 126.97, 127.86, 128.59, 131.35, 136.49, 142.33, 153.53, 154.91; HRMS (EI) exact mass calculated for (C₁₈H₂₁NO₃) requires m/z 299.15214, found m/z 299.15316 [M+Na]⁺.

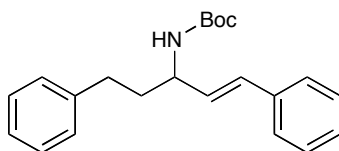
**(E)-tert-butyl 1-phenyloct-1-en-3-ylcarbamate (Table 3, entry 6):**

Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and *tert*-butyl hex-1-enylcarbamate (mixture of *E* & *Z* isomers) (190.0 mg, 0.952 mmol) at $23\text{ }^{\circ}\text{C}$ for 4 h to provide the title compound as a clear oil (137.1 mg, 94% yield) following silica gel chromatography (2.5% EtOAc/petroleum ether, $R_f = 0.34$ in 5% Et₂O/petroleum ether). IR (film) 3363, 2955, 2929, 2858, 1683, 1512, 1453, 1390, 1369, 1303, 1277, 1246, 1165, 1116, 1042, 1017, 964, 864, 777, 747, 693 cm^{-1} ; ¹H NMR (400 MHz, CDCl₃) δ 0.89 (t, 3H, $J = 6$ Hz, -CH₃), 1.46 (s, 9H, -C(CH₃)₃), 1.31-1.50 (m, 8H, -(CH₂)₄), 4.26 (brs, 1H, -CHNH), 4.53 (brs, 1H, -CHNH), 6.09 (dd, $J = 10$ Hz, 16Hz, -CHCHC₆H₅) 6.51 (d, 1H, $J = 16$ Hz, -CHCHC₆H₅), 7.21-7.38 (m, 5H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 14.01, 22.61, 24.45, 25.53, 28.44, 31.65, 35.63, 126.37, 127.44, 128.54, 129.81, 136.91, 195.53; HRMS (EI) exact mass calculated for (C₁₉H₂₉NO₂) requires m/z 303.21983, found m/z 303.2205 [M+Na]⁺.



(E)-benzyl 1-phenyloct-1-en-3-ylcarbamate (Table 3, entry 7):

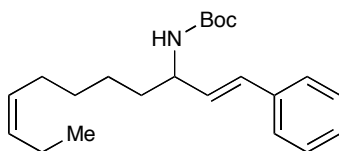
Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and benzyl hex-1-enylcarbamate (mixture of *E* & *Z* isomers) (222.0 mg, 0.952 mmol) at 23 °C for 6 h to provide the title compound as a clear oil (142.4 mg, 89% yield) following silica gel chromatography (5–10% Et₂O/petroleum ether, R_f = 0.23 in 5% Et₂O/petroleum ether). IR (film) 3304, 3032, 2950, 2926, 2856, 1682, 1553, 1495, 1466, 1454, 1300, 1247, 1114, 1044, 1027, 964, 909, 832, 748, 727, 693 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.83–0.90 (m, 5H, -CH₂CH₃), 1.30–1.38 (m, 6H, -(CH₂)₃), 4.34 (brs, 1H, -CHNH), 4.76 (brs, 1H, -CHNH), 5.13 (brs, 2H, -OCH₂Ph), 6.10 (dd, *J* = 8, 20 Hz, -CHCHC₆H₅) 6.55 (d, 1H, *J* = 20 Hz, -CHCHC₆H₅), 7.21–7.37 (m, 10H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 14.09, 22.58, 25.47, 31.61, 35.50, 53.10, 66.77, 126.41, 127.57, 128.56, 130.22, 136.50, 136.70, 195.54; HRMS (EI) exact mass calculated for (C₂₂H₂₇NO₂) requires *m/z* 337.20418, found *m/z* 337.20465 [M+Na]⁺.



(E)-tert-butyl 1,5-diphenylpent-1-en-3-ylcarbamate (Table 3, entry 8):

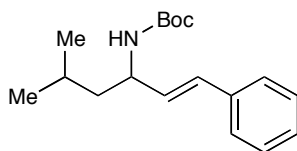
Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and *tert*-butyl 3-phenylprop-1-enylcarbamate (mixture of *E* & *Z* isomers) (222.0 mg, 0.952 mmol) at 23 °C for 4 h to provide the title compound as a white solid (149.9 mg, 94% yield) following silica gel chromatography (5% Et₂O/petroleum ether, R_f = 0.27 in 5% Et₂O/petroleum ether). IR (film) 3318, 2980, 2932, 1679, 1524, 1494, 1453, 1433, 1391, 1365, 1246, 1163, 1045, 1026, 969, 866, 742, 693 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.47 (s, 9H, -C(CH₃)₃), 1.92 (brs, 2H, -CH₂CH₂Ph), 2.72 (m, 2H, -CH₂CH₂Ph), 4.34 (brs, 1H, -CHNH), 4.59 (brs, 1H, -CHNH), 6.13 (dd, *J* = 10, 15 Hz, -CHCHC₆H₅) 6.55 (d, 1H, *J* = 15 Hz, -CHCHC₆H₅), 7.20–7.22 (m, 3H, ArH), 7.24–7.32 (m, 5H, ArH), 7.37–7.38 (m, 2H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.33,

28.37, 28.45, 32.29, 37.17, 37.34, 52.35, 125.99, 126.08, 126.42, 127.59, 128.44, 128.51, 128.58, 128.78, 130.36, 136.80, 141.60; HRMS (EI) exact mass calculated for (C₂₂H₂₇NO₂) requires m/z 337.20418, found m/z 337.20447 [M+Na]⁺.



***tert*-butyl (1*E*, 8*Z*)-1-phenylundeca-1,8-dien-3-ylcarbamate (Table 3, entry 9):**

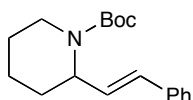
Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and *tert*-butyl nona-1,8-dienylcarbamate (mixture of *E* & *Z* isomers) (228.0 mg, 0.952 mmol) at 23 °C for 4 h to provide the title compound as a clear oil (161.0 mg, 98% yield) following silica gel chromatography (1–5% Et₂O/petroleum ether, R_f = 0.32 in 5% Et₂O/petroleum ether). IR (film) 3377, 2978, 2965, 2934, 2856, 1687, 1504, 1460, 1447, 1388, 1364, 1304, 1235, 1165, 1053, 1003, 985, 914, 862, 750, 693 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.94–0.97 (m, 3H, -CH₃), 1.39 (m, 4H, -(CH₂)₂), 1.45 (s, 9H, -C(CH₃)₃), 2.00–2.04 (m, 6H, -(CH₂)₃), 4.26 (brs, 1H, -CHNH), 4.54 (brs, 1H, -CHNH), 5.34 (m, 2H, -CH₂CHCHCH₂-), 6.09 (dd, *J* = 5Hz, 15Hz, -CHCHC₆H₅), 6.51 (d, 1H, *J* = 15 Hz, -CHCHC₆H₅), 7.23 (dd, 1H, *J* = 10 Hz, ArH), 7.30 (dd, 2H, *J* = 5, 10 Hz, ArH), 7.37 (d, 2H, *J* = 10 Hz, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 14.39, 20.54, 25.49, 26.99, 28.31, 28.45, 29.55, 35.58, 126.39, 127.46, 128.54, 128.88, 129.91, 130.76, 131.88, 136.93; HRMS (EI) exact mass calculated for (C₂₂H₃₃NO₂) requires m/z 343.25113, found m/z 343.25176 [M+Na]⁺.



(*E*)-*tert*-butyl 5-methyl-1-phenylhex-1-en-3-ylcarbamate (Table 3, entry 10):

Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (100 mg, 0.476 mmol) and *tert*-butyl 4-methylpent-1-enylcarbamate (mixture of *E* & *Z* isomers) (176.0 mg, 0.952 mmol) at 23 °C for 4 h to provide the title compound as a clear oil (123.8.0 mg, 90% yield) following silica gel chromatography (3% Et₂O/petroleum

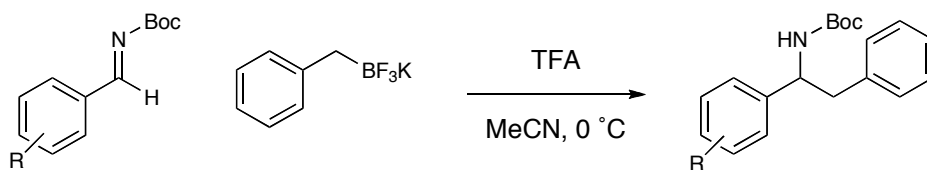
ether, $R_f = 0.29$ in 5% Et₂O/petroleum ether). IR (film) 3321, 2976, 2951, 2934, 1685, 1534, 1494, 1389, 1364, 1273, 1249, 1166, 1124, 1049, 1027, 966, 877, 741, 690 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.956 (dd, 6H, $J = 2, 25$ Hz, -CH(CH₃)₂), 1.06 (dd, 2H, $J = 5, 25$, Hz, -CH₂CH(CH₃)₂), 1.46 (s, 9H, -C(CH₃)₃), 1.71 (m, 1H, -CH₂CH(CH₃)₂), 4.33 (brs, 1H, -CHNH), 4.49 (brs, 1H, -CHNH), 6.07 (dd, $J = 15$ Hz, 10Hz, -CHCHC₆H₅) 6.52 (d, 1H, $J = 15$ Hz, -CHCHC₆H₅), 7.23 (m, 1H, ArH), 7.31 (m, 2H, ArH), 7.37 (d, 2H, $J = 10$ Hz, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 22.63, 22.92, 24.78, 24.97, 25.34, 27.19, 28.36, 28.38, 28.44, 44.81, 50.76, 126.37, 127.44, 128.53, 129.71, 130.97; HRMS (EI) exact mass calculated for (C₁₈H₂₇NO₂) requires m/z 289.20418, found m/z 289.20432 [M+Na]⁺.



tert-butyl 2-styrylpiperidine-1-carboxylate: Prepared according to the general procedure from potassium *trans*-styryltrifluoroborate (15.0 mg, 0.0714 mmol) and *N*-(*tert*-butoxycarbonyl)-1,2,3,4-tetrahydropyridine⁶ (26.2 mg, 0.143 mmol) at -20 °C for 19 h. Reaction conversion determined by achiral GLC analysis on a Varian HP-1 column relative to 4,4'-dimethylbiphenyl as internal standard. IR (film) 2940, 2920, 2855, 1681, 1449, 1398, 1364, 1326, 1275, 1252, 1240, 1157, 1117, 1090, 1039, 1012, 969, 961, 929, 870, 769, 757, 748, 708 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.49 (s, 9H, -C(CH₃)₃), 1.52-1.63 (m, 4H, -CH(CH₂)₃CH₂NH-), 1.74-1.86 (m, 2H, -CH(CH₂)₃CH₂NH-), 2.92 (t, 1H, $J = 13$ Hz, -CH(CH₂)₃CH₂NH-), 4.01 (brd, 1H, $J = 12$ Hz, -CH(CH₂)₃CH₂NH), 4.97 (brs, 1H, -CHNH), 6.19 (dd, $J = 16$ Hz, 5Hz, -CHCHC₆H₅) 6.40 (d, 1H, $J = 16$ Hz, -CHCHC₆H₅), 7.22-7.25 (m, 1H, ArH), 7.31-7.38 (m, 4H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 19.71, 25.58, 28.50, 29.54, 79.49, 126.25, 127.39, 128.58, 128.74, 130.70; HRMS (EI) exact mass calculated for (C₁₈H₂₇NO₂) requires m/z 287.18853, found m/z 310.17767 [M+Na]⁺.

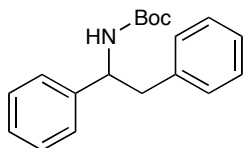
Reactions with Potassium Benzyltrifluoroborate

⁶ Prepared according to: Dieter, K. R.; Sharma, R. R. *J. Org. Chem.* **1996**, *61*, 4180.



General Procedure: To a rigorously degassed solution of potassium benzyltrifluoroborate (50.0 mg, 0.252 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (104 mg, 0.505 mmol) in acetonitrile (2.5 mL, 0.1 M) at 23 °C was added dropwise trifluoroacetic acid (19 μ L, 0.252 mmol). The reaction was stirred at 23 °C for 4 h, then the crude reaction mixture was poured into a separatory funnel containing NaHCO_3 (sat. aq.) and extracted with CH_2Cl_2 (3 \times 25 mL). The combined organic phase was dried (Na_2SO_4), concentrated *in vacuo* and purified via silica gel chromatography (5% Et_2O /hexanes) to give the desired product as a white solid (39.6 mg, 50% yield).

Determination of Initial Reaction Rates: To an oven dried vial was added standardized solutions of benzyl trifluoroborate (0.300 mmol, 0.74 mL), 4,4'-dimethylbiphenyl (0.075 mmol, 0.88 mL) and imine (0.450 mmol, 0.62 mL) in acetonitrile. The reaction was diluted to a total volume of 3.0 mL with acetonitrile, sealed with a septa cap, cooled to 0 °C and purged with argon for 20 minutes. Trifluoroacetic acid (0.300 mmol, 22.3 μ L) was added dropwise and the reaction was sampled at the indicated time periods. Sampling was accomplished by withdrawing 0.10 mL of crude reaction mixture via syringe and filtering through a plug of silica gel with Et_2O . The amount of product present was determined via GLC analysis relative to the internal standard on a Varian HP-1 column.



(*N*)-*tert*-butoxycarbonyl α -benzyl-benzylamine: ^1H NMR and ^{13}C NMR data in accord

with literature values.⁷ Conversion factor for initial rate determination: 0.6182833 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 1.57 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	989.206	95.6969	1.49533553	0.00448601
4	2830.88	492.046	2.68665419	0.00805996
6	1634.67	408.59	3.8635378	0.01159061
8	1888.07	593.887	4.86198094	0.01458594
10	1858.37	672.771	5.59580538	0.01678742
12	2901.9	1189.1	6.33378711	0.01900136
14	1847.43	845.485	7.07400627	0.02122202
16	2192.12	1076.99	7.59407481	0.02278222
20	1495.66	836.098	8.64075777	0.02592227
25	2666.8	869.129	5.03757262	0.01511272
30	2194.6	1403.8	9.88729263	0.02966188

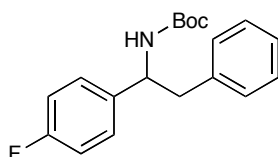
Run #2: $k_{\text{initial}} = 1.59 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1431.19	155.688	1.68145547	0.00504437
4	1440.64	251.977	2.70354098	0.00811062
6	1622.65	406.255	3.86991468	0.01160974
8	1831.56	550.617	4.6468215	0.01394046
10	1966.86	686.274	5.39326329	0.01617979
12	2207.34	838.733	5.8732978	0.01761989
14	2132.35	882.219	6.39507204	0.01918522
16	2404.74	1034.01	6.64636421	0.01993909
20	2268.55	1053.62	7.17898713	0.02153696
25	1648.72	826.923	7.75256989	0.02325771
30	1558.63	825.312	8.18469789	0.02455409

Run #3: $k_{\text{initial}} = 1.53 \times 10^{-3} \text{ M/min}^{-1}$

⁷ Beak, P.; Park, Y. S.; Boys, M.L. *J. Am. Chem. Soc.* **1996**, *118*, 3757.

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1325.6	138.062	1.609864	0.00482959
4	1674.45	295.69	2.72955581	0.00818867
6	1185.44	286.882	3.74068594	0.01122206
8	1608.4	467.73	4.49498955	0.01348497
10	1493.23	504.836	5.22578016	0.01567734
12	1800.9	668.496	5.7376855	0.01721306
14	1724.49	689.1	6.17659456	0.01852978
16	1962.18	836.461	6.5892256	0.01976768
20	2573.9	1164.89	6.9955324	0.0209866
25	2383.14	1154.78	7.48992075	0.02246976
30	2024.87	1041.908	7.95352685	0.02386058



(N)-tert-butoxycarbonyl α -benzyl-4-fluorobenzylamine: IR (film) 3383, 2976, 2908, 1675, 1604, 1506, 1455, 1442, 1391, 1365, 1350, 1296, 1266, 1250, 1223, 1168, 1158, 1097, 1081, 1044, 1014, 876, 837, 819, 730, 699 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.38 (s, 9H, $-\text{C}(\text{CH}_3)_3$), 3.00 (brs, 2H, $-\text{CH}_2\text{Ph}$), 4.88 (brs, 2H, $-\text{CHNH}$), 6.96-6.99 (m, 2H, ArH), 7.01-7.03 (m, 2H, ArH), 7.12-7.15 (m, 2H, ArH), 7.18-7.25 (m, 3H, ArH); ^{13}C NMR (125 MHz, CDCl_3) δ 28.32, 43.35, 115.18, 115.34, 126.62, 127.95, 128.01, 128.38, 129.40, 137.04; HRMS (EI) exact mass calculated for ($\text{C}_{19}\text{H}_{22}\text{FNO}_2$) requires m/z 315.16346, found m/z 338.15347 [$\text{M}+\text{Na}$] $^+$. Conversion factor for initial rate determination: 0.818235 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 1.62 \times 10^{-3} \text{ M/min}^{-1}$

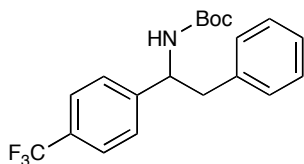
Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1472.79	106.221	1.47532472	0.00442597
4	1571.33	207.437	2.70045461	0.00810136
6	1310.42	249.41	3.89333174	0.01168
8	2074.01	483.968	4.7733564	0.01432007
10	1926.55	534.701	5.67739058	0.01703217
12	2016.9	631.417	6.40397998	0.01921194
14	2087.06	726.588	7.12149785	0.02136449
16	2692.8	985.214	7.4841854	0.02245256
18	1342.05	554.635	8.45389086	0.02536167
20	1632.04	706.03	8.84933055	0.02654799

Run #2: $k_{\text{initial}} = 1.59 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1226.71	93.9261	1.56625491	0.00469876
4	2262.88	299.328	2.70585102	0.00811755
6	2446.21	451.769	3.77781556	0.01133345
8	2203.03	509.159	4.72771268	0.01418314
10	1470.71	422.736	5.8797688	0.01763931
12	2299.83	731.335	6.50487094	0.01951461
14	2239.32	788.038	7.19861691	0.02159585
16	2948.97	916.794	6.35944871	0.01907835
18	1448.46	587.945	8.30325271	0.02490976
20	2089.22	887.642	8.69103962	0.02607312

Run #3: $k_{\text{initial}} = 1.63 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1114.47	87.5502	1.60696649	0.0048209
4	2062.88	281.093	2.78736682	0.0083621
6	2128.29	405.899	3.90126308	0.01170379
8	2520.53	611.233	4.96058601	0.01488176
10	2407.91	683.659	5.80787614	0.01742363
12	1951.32	633.944	6.64569585	0.01993709
14	2036.95	730.884	7.33983247	0.0220195
16	2178	854.954	8.02976683	0.0240893



(N)-tert-butoxycarbonyl α -benzyl-4-trifluoromethylbenzylamine: IR (film) 3380, 2979, 1675, 1619, 1519, 1496, 1443, 1367, 1325, 1267, 1251, 1162 1123, 1108, 1067, 1046, 1016, 850, 839, 779, 752, 732, 699 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.38 (s, 9H, $-\text{C}(\text{CH}_3)_3$), 3.03 (brs, 2H, $-\text{CH}_2\text{Ph}$), 4.96 (brs, 2H, $-\text{CHNH}$), 7.02-7.04 (m, 2H, ArH), 7.21-7.24 (m, 3H, ArH), 7.29-7.31 (m, 2H, ArH), 7.55-7.56 (m, 2H, ArH); ^{13}C NMR (125 MHz, CDCl_3) δ 28.04, 28.29, 125.42, 126.68, 126.86, 128.54, 129.33, 155.01; HRMS (EI) exact mass calculated for $(\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_2)$ requires m/z 364.12863, found m/z 365.13562 $[\text{M}+\text{H}]^+$. Conversion factor for initial rate determination: 0.8526336 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 2.29 \times 10^{-3} \text{ M/min}^{-1}$

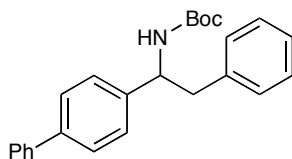
Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1615.58	174.818	2.30653543	0.00691961
4	1627.38	343.351	4.49729933	0.0134919
6	1852.71	504.142	5.80026567	0.0174008
8	3474	1046.13	6.41886577	0.0192566
10	2257.12	844.279	7.97322078	0.02391966
12	1790.91	758.101	9.02309978	0.0270693
14	1116.73	525.649	10.0334458	0.03010034
16	2144.88	1052.02	10.4549858	0.03136496
18	2162.61	1114.35	10.9836292	0.03295089
20	2948.02	1574.72	11.3861099	0.03415833

Run #2: $k_{\text{initial}} = 2.40 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1990.79	267.38	2.8628983	0.00858869
4	1927.93	426.558	4.71616816	0.0141485
6	2222.73	639.109	6.12901485	0.01838704
8	2211.4	755.08	7.27826918	0.02183481
10	2444.55	950.011	8.28384876	0.02485155
12	1819.7	790.147	9.25572733	0.02776718
14	1385.19	660.172	10.1589823	0.03047695
16	1369.3	699.906	10.8954096	0.03268623
18	1562.3	842.2	11.4908791	0.03447264
20	1923.3	1080.86	11.9791186	0.03593736

Run #3: $k_{\text{initial}} = 2.26 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	2086.27	297.532	3.03994426	0.00911983
4	1613.24	363.3	4.80030539	0.01440092
6	2410.13	666.702	5.89649237	0.01768948
8	2343.86	775.736	7.0548004	0.0211644
10	2023.92	751.613	7.91595639	0.02374787
12	738.867	308.725	8.90651863	0.02671956
14	1864.3	824.316	9.42497879	0.02827494
16	1648.38	776.833	10.0455283	0.03013658
18	1313.37	650.461	10.5569052	0.03167072
20	2304.33	1161.54	10.744642	0.03223393



(N)-tert-butoxycarbonyl α -benzyl-4-phenylbenzylamine: IR (film) 3382, 2874, 2923, 1679, 1602, 1514, 1488, 1453, 1442, 1390, 1364, 1290, 1265, 1247, 1167, 1081, 1043, 1021, 1007, 851, 838, 760, 728, 719, 692 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.39 (s, 9H, $-\text{C}(\text{CH}_3)_3$), 3.10 (brs, 2H, $-\text{CH}_2\text{Ph}$), 4.94 (brs, 2H, $-\text{CHNH}$), 7.08-7.09 (m, 2H, ArH), 7.20-7.28 (m, 5H, ArH), 7.33-7.36 (m, 1H, ArH), 7.44-7.47 (m, 2H, ArH), 7.53-7.54 (m, 2H, ArH), 7.58-7.59 (m, 2H, ArH); ^{13}C NMR (125 MHz, CDCl_3) δ 28.35, 43.31, 126.55, 126.84, 127.06, 127.20, 127.26, 127.37, 128.36, 128.78, 129.45, 137.33, 140.04; HRMS (EI) exact mass calculated for ($\text{C}_{25}\text{H}_{27}\text{NO}_2$) requires m/z 373.20418, found m/z 396.19325 $[\text{M}+\text{Na}]^+$. Conversion factor for initial rate determination: 1.0763331 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 1.50 \times 10^{-3} \text{ M/min}^{-1}$

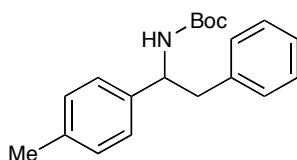
Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1648.68	74.3869	1.21407858	0.00364224
4	2736.1	206.199	2.02787552	0.00608363
6	2791.96	314.332	3.02946618	0.0090884
8	2328.09	341.801	3.95057461	0.01185172
10	1777.07	341.015	5.16363638	0.01549091
12	2310.38	468.956	5.46179487	0.01638538
14	2292.65	529.962	6.22004713	0.01866014
16	2659.42	855.03	8.65129512	0.02595389
18	2952.53	758.271	6.91061713	0.02073185
20	3090.43	891.881	7.76559444	0.02329678

Run #2: $k_{\text{initial}} = 1.46 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	2447.28	101.731	1.11855246	0.00335566
4	2719.02	209.548	2.0737568	0.00622127
6	2679.84	311.412	3.12689417	0.00938068
8	2145.71	332.021	4.16371728	0.01249115
10	2859.69	506.79	4.7686537	0.01430596
12	2780.41	567.492	5.49208951	0.01647627
14	3030.12	677.673	6.017929	0.01805379
16	3345.04	819.382	6.59131108	0.01977393
18	3117.09	808.698	6.98109796	0.02094329
20	2883.57	789.472	7.36703847	0.02210112

Run #3: $k_{\text{initial}} = 1.47 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	2163.21	94.0562	1.16997196	0.00350992
4	2203.65	180.588	2.20512379	0.00661537
6	1735.09	218.012	3.38099943	0.010143
8	2735.37	412.761	4.06040432	0.01218121
10	2560.26	465.694	4.89444301	0.01468333
12	3047.31	801.505	7.07744175	0.02123233
14	3052.27	670.319	5.90942583	0.01772828
16	2059.69	556.528	7.27062698	0.02181188
18	2171.77	626.935	7.76775271	0.02330326
20	3060.91	856.993	7.53378842	0.02260137



(N)-tert-butoxycarbonyl α -benzyl-4-methylbenzylamine: IR (film) 3384, 2979, 2911, 1681, 1512, 1455, 1440, 1389, 1362, 1314, 1293, 1265, 1248, 1167, 1043, 1020, 850, 804, 778, 730, 719, 697 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.37 (s, 9H, $-\text{C}(\text{CH}_3)_3$), 2.32 (s, 3H, $-\text{CH}_3$), 3.04 (brs, 2H, $-\text{CH}_2\text{Ph}$), 4.89 (brs, 2H, $-\text{CHNH}$), 7.05-7.11 (m, 6H, ArH), 7.18-7.24 (m, 3H, ArH); ^{13}C NMR (125 MHz, CDCl_3) δ 21.12, 28.33, 126.33, 126.42, 128.27, 129.15, 129.44, 136.81, 137.57; HRMS (EI) exact mass calculated for ($\text{C}_{20}\text{H}_{25}\text{NO}_2$) requires m/z 311.18853, found m/z 334.17781 $[\text{M}+\text{Na}]^+$. Conversion factor for initial rate determination: 0.8700065 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 1.17 \times 10^{-3} \text{ M}/\text{min}^{-1}$

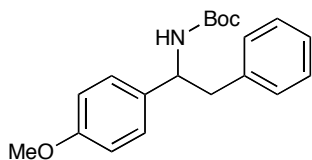
Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
5	2492.3	284.427	2.4821785	0.00744654
10	1876.84	395.861	4.58752002	0.01376256
20	1626.6	628.623	8.40566359	0.02521699
25	1587.23	712.389	9.76202347	0.02928607
30	2614.38	1302.04	10.8322362	0.03249671
35	2209.4	1246.58	12.2718012	0.0368154
40	1815.63	1120.13	13.4184881	0.04025546
45	2280.5	1472.3	14.0419927	0.04212598
50	2910.38	1922.13	14.3646671	0.043094
55	1700.85	1225.64	15.6732629	0.04701979
60	2925.5	2127.56	15.8177323	0.0474532

Run #2: $k_{\text{initial}} = 1.19 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
5	2159.92	236.97	2.38626246	0.00715879
10	2633.02	559.128	4.61869825	0.01385609
15	1622.31	502.634	6.73876829	0.0202163
20	1476.88	577.566	8.50587343	0.02551762
25	2497.3	1126.83	9.81409346	0.02944228
30	2375.97	1222.73	11.1931448	0.03357943
35	2964.36	1696.67	12.4488416	0.03734652
40	2159.99	1327.7	13.3693632	0.04010809
45	1770.4	1183.81	14.5436398	0.04363092
50	3434.54	2357.2	14.9276127	0.04478284
55	2539.18	1828.07	15.6589212	0.04697676
60	1692.6	1287.29	16.5418685	0.04962561

Run #3: $k_{\text{initial}} = 1.18 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
5	2174.41	268.353	2.6842782	0.00805283
10	1264.53	294.549	5.06630022	0.0151989
15	1469.87	467.972	6.92473963	0.02077422
20	2596	996.933	8.35264051	0.02505792
25	2400	1096.9	9.94073052	0.02982219
30	2406.15	1263.35	11.4199313	0.03425979
35	1780.08	1050.47	12.8353182	0.03850595
40	1722.7	1129.62	14.2621574	0.04278647
45	1982.96	1355.61	14.8690532	0.04460716
50	1099.23	812.575	16.0781986	0.0482346
55	2206.14	1686.1	16.6231286	0.04986939
60	1982.11	1577.81	17.3136828	0.05194105



(N)-tert-butoxycarbonyl α -benzyl-4-methoxybenzylamine: IR (film) 3383, 2975, 2910, 2838, 1679, 1613, 1508, 1455, 1441, 1390, 1364, 1303, 1242, 1166, 1109, 1082, 1023, 971, 879, 849, 834, 808, 778, 750, 729, 698 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.38 (s, 9H, $-\text{C}(\text{CH}_3)_3$), 3.02 (brs, 2H, $-\text{CH}_2\text{Ph}$), 3.79 (s, 3H, $-\text{OCH}_3$), 4.84 (brs, 2H, $-\text{CHNH}$), 6.83-6.82 (m, 2H, ArH), 7.03-7.05 (m, 2H, ArH), 7.09-7.11 (m, 2H, ArH), 7.18-7.24 (m, 3H, ArH); ^{13}C NMR (125 MHz, CDCl_3) δ 28.32, 55.27, 113.77, 113.92,

126.41, 127.58, 127.85, 128.26, 128.39, 129.46, 137.54; HRMS (EI) exact mass calculated for (C₂₀H₂₅NO₃) requires *m/z* 327.18344, found *m/z* 350.1726 [M+Na]⁺. Conversion factor for initial rate determination: 0.930085 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 6.89 \times 10^{-5} \text{ M/min}^{-1}$

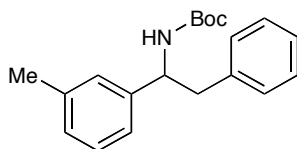
Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
10	2614.04	27.3369	0.24316423	0.00072949
15	2268.21	34.1327	0.34990491	0.00104971
20	2577.82	52.4179	0.47281329	0.00141844
25	1733.99	46.4324	0.62264025	0.00186792
30	2480.12	73.5296	0.68936965	0.00206811
35	2727.74	98.8583	0.84269965	0.0025281
40	1061.26	49.9256	1.09386606	0.0032816
45	2101.74	97.8424	1.08245726	0.00324737
50	2591.82	122.639	1.10023742	0.00330071
55	2773.27	150.958	1.2656879	0.00379706
60	2960.67	173.504	1.36264315	0.00408793

Run #2: $k_{\text{initial}} = 6.86 \times 10^{-5} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
5	2711.22	17.7384	0.15212911	0.00045639
10	2170.09	21.7364	0.23290163	0.0006987
15	1503.12	24.6404	0.38116828	0.0011435
20	2536.31	49.1952	0.45100675	0.00135302
25	2638.28	63.5088	0.55972624	0.00167918
30	1497.03	46.8724	0.7280301	0.00218409
35	2241.67	75.0817	0.77879843	0.0023364
40	1194.23	50.0488	0.9744697	0.00292341
45	2486.65	107.969	1.00959471	0.00302878
50	2530.91	123.648	1.13598617	0.00340796
55	1164.16	67.946	1.35710631	0.00407132
60	2897.52	166.27	1.33428961	0.00400287

Run #3: $k_{\text{initial}} = 7.50 \times 10^{-5} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
5	1675.5	9.45473	0.13121012	0.00039363
10	2230.82	21.438	0.22345104	0.00067035
15	2120.44	33.2527	0.36463938	0.00109392
20	2842.6	55.4533	0.4536013	0.0013608
25	2791.64	69.4631	0.57857198	0.00173572
30	1757.26	58.9961	0.78063843	0.00234192
35	2993.95	104.693	0.81308463	0.00243925
40	3227.72	128.679	0.92698877	0.00278097
45	3125.1	145	1.07851438	0.00323554
50	2679.38	141.206	1.22541019	0.00367623
55	2217.13	137.066	1.43747808	0.00431243
60	1683.32	125.807	1.73780392	0.00521341



(N)-tert-butoxycarbonyl α -benzyl-3-methylbenzylamine: IR (film) 3383, 2980, 2920, 1682, 1606, 1517, 1495, 1443, 1389, 1363, 1349, 1264, 1244, 1159, 1083, 1044, 1027, 1012, 886, 853, 786, 776, 752, 730, 699 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.37 (s, 9H, $-\text{C}(\text{CH}_3)_3$), 2.32 (s, 3H, $-\text{CH}_3$), 3.04 (brs, 2H, $-\text{CH}_2\text{Ph}$), 4.86 (brs, 2H, $-\text{CHNH}$), 6.98-7.00 (m, 2H, ArH), 7.04-7.07 (m, 3H, ArH), 7.17-7.20 (m, 2H, ArH), 7.22-7.24 (m, 2H, ArH); ^{13}C NMR (125 MHz, CDCl_3) δ 21.50, 28.33, 123.40, 126.45, 127.22, 127.98, 128.27, 128.35, 129.43, 137.54, 138.07; HRMS (EI) exact mass calculated for ($\text{C}_{20}\text{H}_{25}\text{NO}_3$) requires m/z 311.18853, found m/z 334.17782 $[\text{M}+\text{Na}]^+$. Conversion factor for initial rate determination: 0.8563388 mmol product / 4,4'-dimethylbiphenyl.

Run #1: $k_{\text{initial}} = 1.50 \times 10^{-3} \text{ M}/\text{min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1157.99	94.9457	1.75531928	0.00526596
4	2554.1	361.433	3.02953194	0.0090886
6	1796.97	341.093	4.06366231	0.01219099
8	3099.82	763.281	5.27149266	0.01581448
10	1929.3	584.892	6.49025182	0.01947076
12	1863.59	638.089	7.3302117	0.02199064
14	2883.28	1086.83	8.06975648	0.02420927
16	2592.99	1070.42	8.83769488	0.02651308
18	1930.5	865.177	9.5944656	0.0287834
20	2113.16	1010.31	10.2354726	0.03070642

Run #2: $k_{\text{initial}} = 1.15 \times 10^{-3} \text{ M/min}^{-1}$

Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	2691.31	207.22	1.64836572	0.0049451
4	1805.57	259.94	3.0820836	0.00924625
6	2823.89	533.886	4.04749562	0.01214249
8	1837.17	464.138	5.40858192	0.01622575
10	1951.35	569.904	6.25247787	0.01875743
12	1780.19	603.841	7.26175966	0.02178528
14	2466.7	894.27	7.76136233	0.02328409
16	3105.7	1202.7	8.29055185	0.02487166
18	3172.01	1336.41	9.01967314	0.02705902
20	2075.12	911.109	9.39967312	0.02819902

Run #3: $k_{\text{initial}} = 1.38 \times 10^{-3} \text{ M/min}^{-1}$

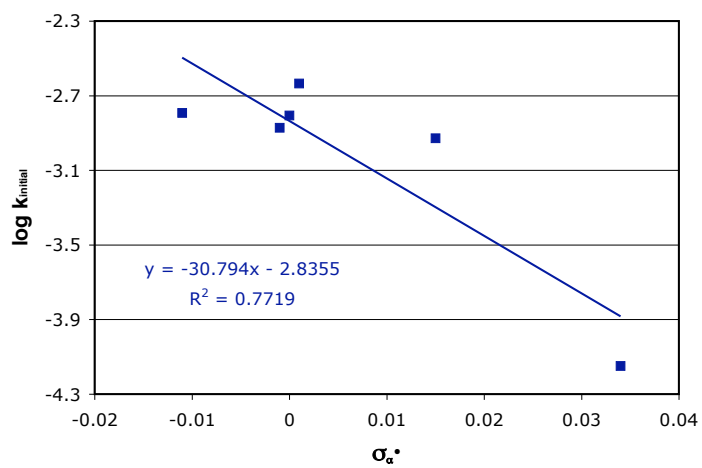
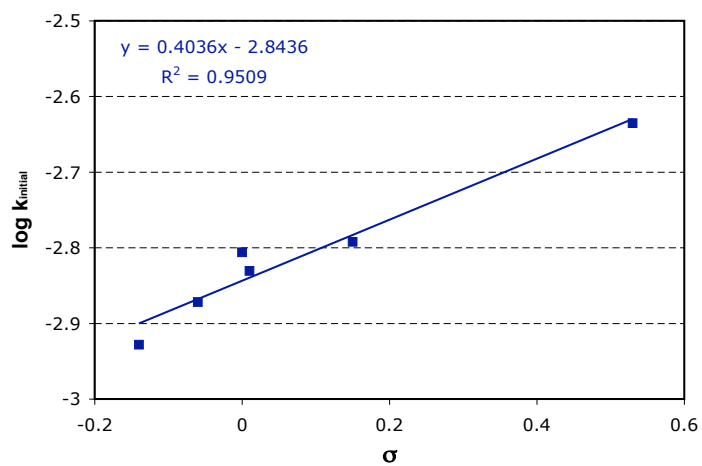
Time (min)	Area std	Area pdt	% pdt	mmol pdt
0	0	0	0	0
2	1907.59	139.714	1.56798	0.00470394
4	2632.65	315.678	2.56706474	0.00770119
6	1689.91	285.036	3.61095245	0.01083286
8	2951.1	602.181	4.36846392	0.01310539
10	2666.82	640.402	5.14096452	0.01542289
12	2170.8	588.227	5.80110562	0.01740332
14	1259.91	397.512	6.75454892	0.02026365
16	1721.94	564.335	7.01624268	0.02104873
18	911.633	329.203	7.73088792	0.02319266
20	2045.28	741.064	7.75690684	0.02327072

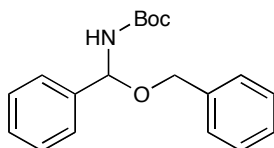
Hammett Plots: Values for the initial rate were averaged over the three runs and plotted versus σ^{δ} and $\sigma_{\alpha}^{\bullet 9}$.

⁸ Values for σ taken from: *Modern Physical Organic Chemistry*; Anslyn, R. A.; Dougherty, D. A.; Publisher: City, Year.

⁹ Value for $\sigma_{\alpha}^{\bullet}$ taken from: Arnold, D. R.; Dust, J. M. *J. Am. Chem. Soc.* **1983**, *105*, 1221.

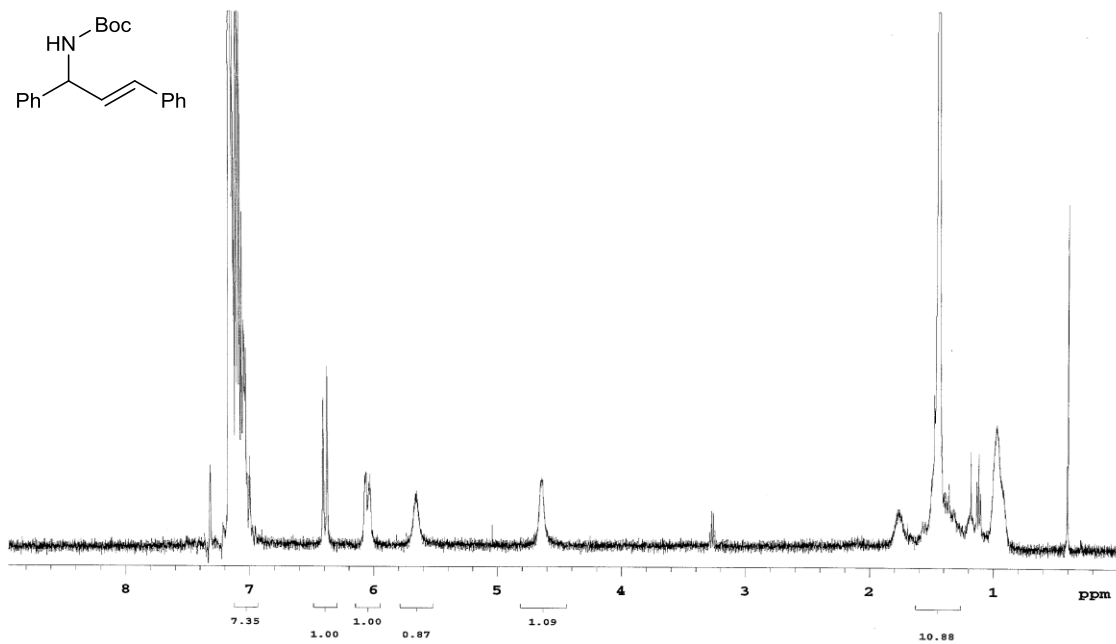
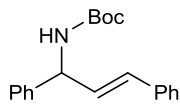
R	Initial Rate	log (Initial Rate)	σ	$\sigma_{\alpha^{\bullet}}$
4-OMe	7.08333E-05	-4.14976232	-0.12	0.034
4-Me	0.00118	-2.928117993	-0.14	0.015
3-Me	0.001343333	-2.871816209	-0.06	-0.001
H	0.001563333	-2.805948412	0	0
4-Ph	0.001476667	-2.830717528	0.01	
4-F	0.001613333	-2.792275893	0.15	-0.011
4-CF3	0.002316667	-2.63513645	0.53	0.001



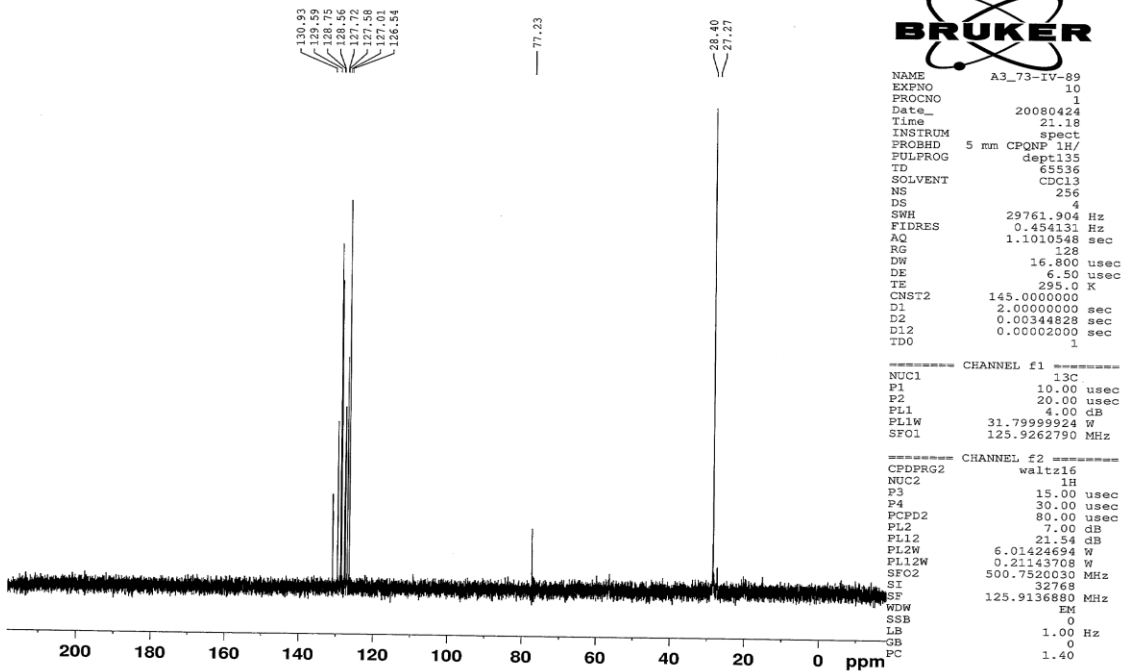


(N)-tert-butoxycarbonyl α -benzyl-benzylamine: To a solution of potassium benzyltrifluoroborate (50.0 mg, 0.252 mmol) and benzaldehyde *N*-(*tert*-butoxycarbonyl)imine (104 mg, 0.505 mmol) in acetonitrile (2.5 mL, 0.1M) at 23 °C was added an O containing balloon and oxygen was bubbled through the solution for 20 minutes. The balloon was removed and 19 μ L (0.252 mmol) of trifluoroacetic acid was added in a dropwise fashion. The reaction was stirred at 23 °C for 4 h to provide the title compound as a white solid (54.6 mg, 69% yield) following standard workup procedure and silica gel chromatography (5% Et₂O/hexanes, R_f = 0.20 in 5% Et₂O/hexanes). IR (film) 3387, 3270, 3064, 2973, 1720, 1689, 1541, 1518, 1497, 1454, 1362, 1266, 1248, 1170, 1053, 1026, 963, 914, 888, 856, 845, 750, 696 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 1.48 (s, 9H, -C(CH₃)₃), 5.07 (AB, 2H, *J* = 5, 11 Hz, -OCH₂Ph), 5.31 (brs, 1H, -CHNH), 6.52 (brs, 1H, -CHNH), 7.33-7.36 (m, 10H, ArH); ¹³C NMR (125 MHz, CDCl₃) δ 28.31, 85.13, 126.51, 128.41, 128.61, 128.99, 129.24, 135.66, 136.32; HRMS (EI) exact mass calculated for (C₁₉H₂₃NO₃) requires *m/z* 352.13150 found *m/z* 352.15219 [M+K]⁺.

¹HNMR and ¹³CNMR Spectra

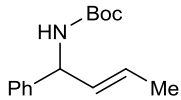


decIV-89-1

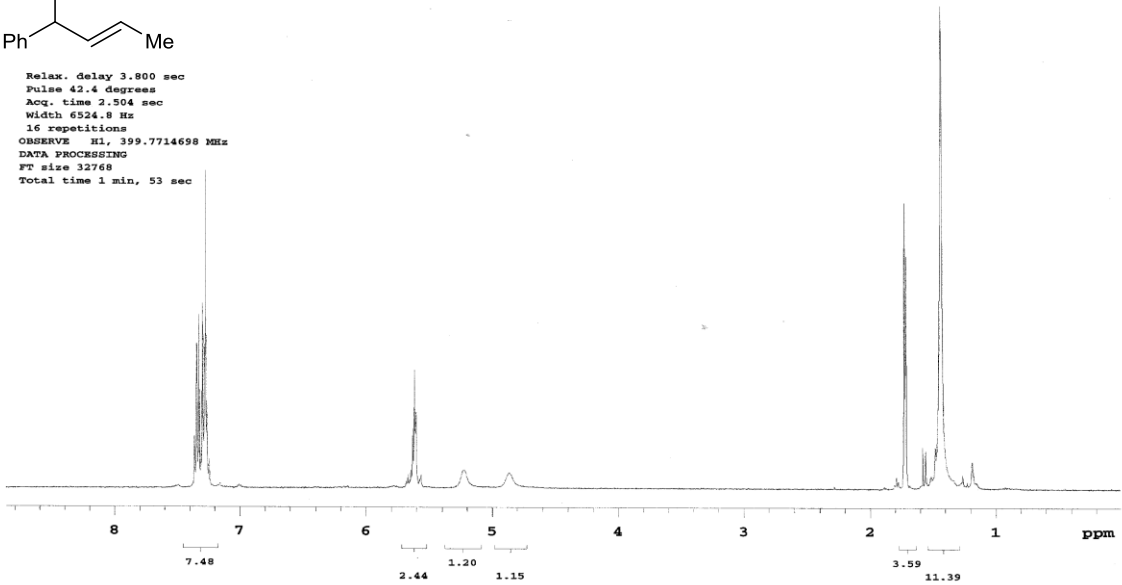


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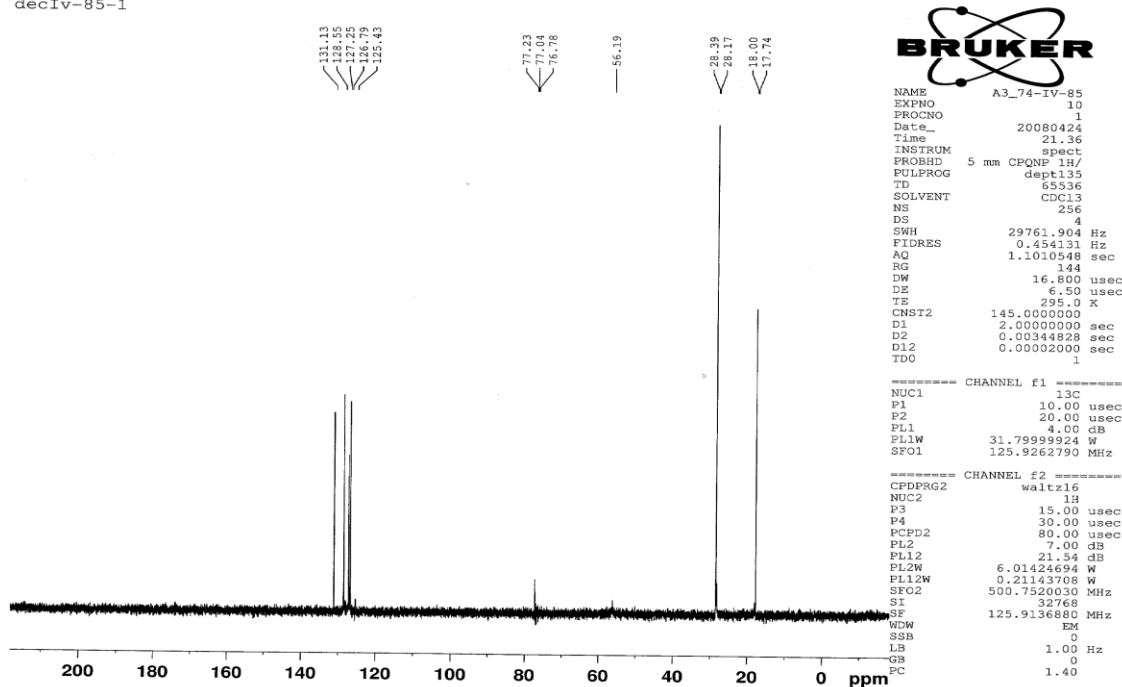
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PROCNO    1
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Time      21.18
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PROBHD    5 mm CPQNP 1H/
FULPROG   dept135
TD         65536
SOLVENT   CDCl3
NS         256
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         128
DW         16.800 usec
DE         6.50 usec
TE         295.0 K
CNST2     145.000000
D1         2.00000000 sec
D2         0.00344828 sec
D12        0.00002000 sec
TD0        1
----- CHANNEL f1 -----
NUC1       13C
P1         10.00 usec
P2         20.00 usec
PL1        4.00 dB
PL1W       31.79999924 W
SFO1       125.9262790 MHz
----- CHANNEL f2 -----
CPDPRG2    waltz16
NUC2       1H
P3         15.00 usec
P4         30.00 usec
PCPD2      80.00 usec
PL2        7.00 dB
PL12       21.54 dB
PL2W       6.01424694 W
PL12W      0.21143708 W
SFO2       500.7520030 MHz
SI         32768
SF         125.9136880 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
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Relax. delay 3.800 sec
 Pulse 42.4 degrees
 Acq. time 2.504 sec
 Width 6524.8 Hz
 16 repetitions
 OBSERVE E1, 399.7714698 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 53 sec



deciv-85-1

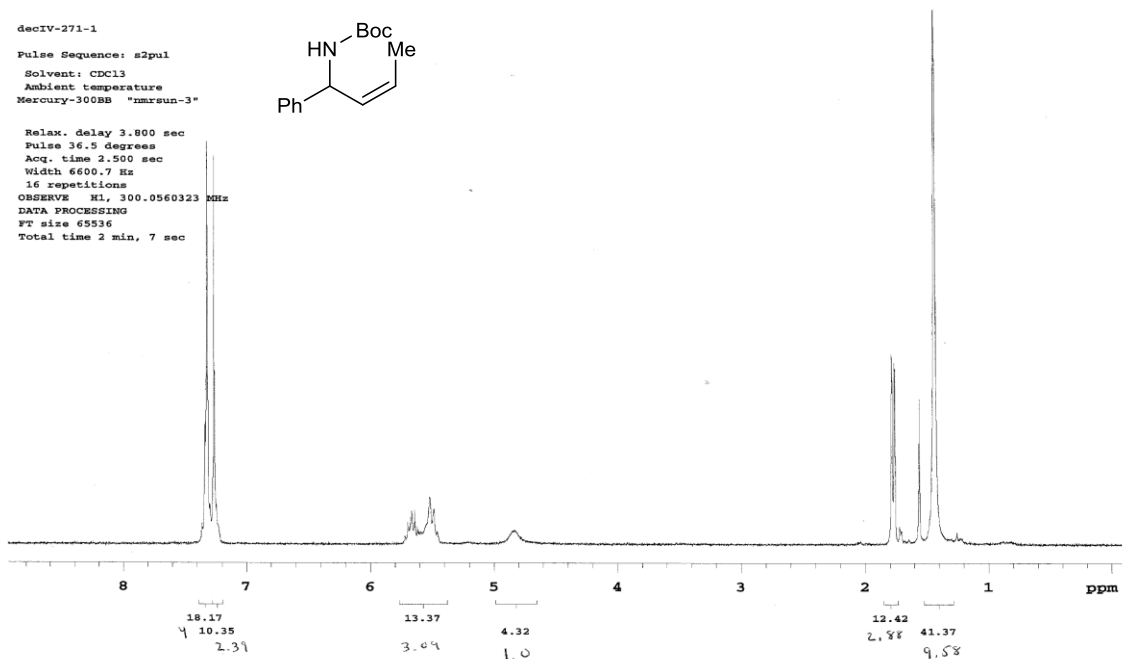
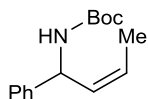


NAME A3_74-IV-85
 EXPNO 10
 PROCNO 1
 Date_ 20080424
 Time 21.36
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG dept135
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 144
 DW 16.800 usec
 DE 6.50 usec
 TE 295.0 K
 CNST2 145.0000000
 D1 2.0000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 10.00 usec
 P2 20.00 usec
 PL1 4.00 dB
 FL1W 31.79999924 W
 SFO1 125.9262790 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 P3 15.00 usec
 P4 30.00 usec
 PCPD2 80.00 usec
 PL2 7.00 dB
 PL12 21.54 dB
 PL2W 6.01424694 W
 FL12W 0.21143708 W
 SFO2 500.7520030 MHz
 SI 32768
 SF 125.9136680 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

decIV-271-1
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-300BB "mrsun-3"
 Relax. delay 3.800 sec
 Pulse 16.5 degrees
 Acq. time 2.500 sec
 Width 6600.7 Hz
 16 repetitions
 OBSERVE M1, 300.0560323 MHz
 DATA PROCESSING
 FT size 65536
 Total time 2 min, 7 sec



decIV-271-1

130.74
 128.62
 126.80
 126.34

77.23

28.41

17.74

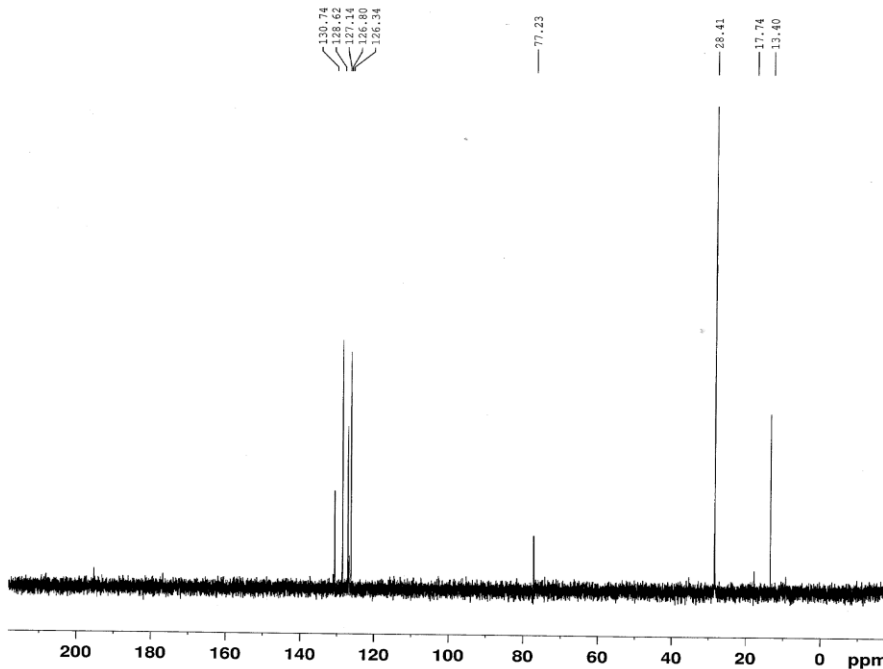
13.40



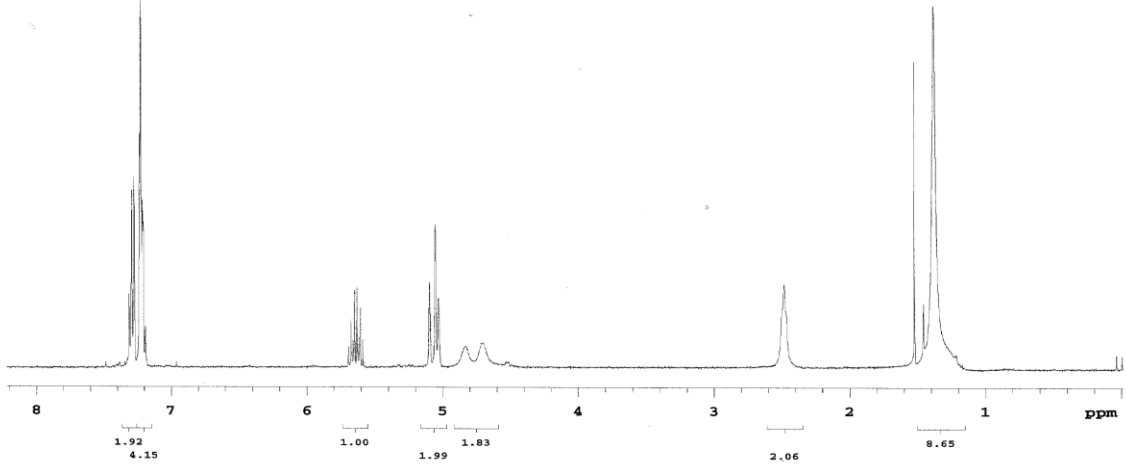
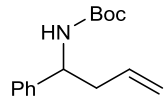
NAME A3_66-IV-271
 EXPNO 10
 PROCNO 1
 Date_ 20080424
 Time 18.10
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 FULPROG dept135
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 101
 DW 16.800 usec
 DE 6.50 usec
 TE 295.0 K
 CNST2 145.0000000
 D1 2.00000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 10.00 usec
 P2 20.00 usec
 PL1 4.00 dB
 PL1W 31.79999924 W
 SFO1 125.9262790 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 P3 15.00 usec
 P4 30.00 usec
 PCPD2 80.00 usec
 PL2 7.00 dB
 PL12 21.54 dB
 PL2W 6.01424694 W
 PL12W 0.21143708 W
 SFO2 500.7520030 MHz
 SI 32768
 SF 125.9136880 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



decIV-161-1
Pulse Sequence: #2pul



decIV-161-1carbon

133.89
129.81
127.15
126.22
118.23

77.24

41.26

28.38

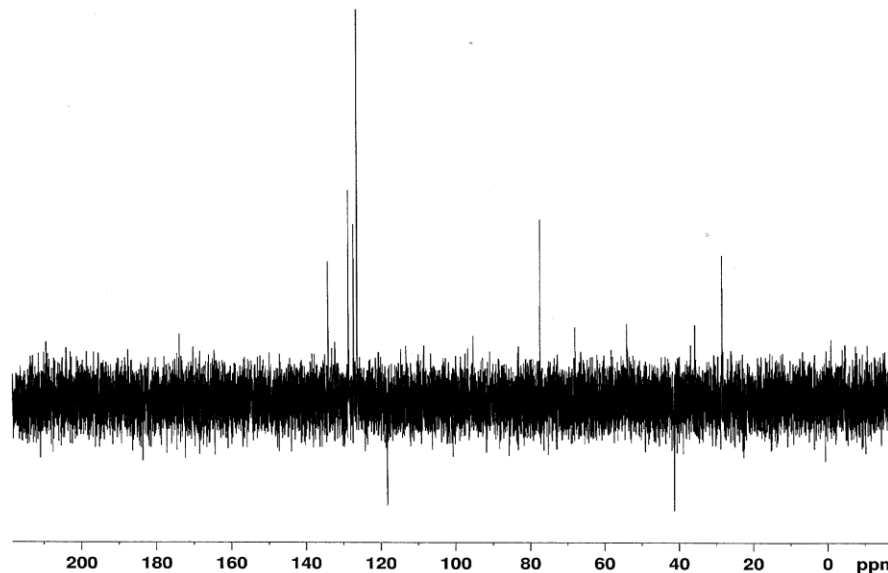


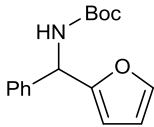
Current Data Parameters
NAME IV-161-1
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080130
Time 12.25
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG dept135
TD 65536
SOLVENT cdc13
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 16400
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.000000
D1 2.0000000 sec
d12 0.00002000 sec
d2 0.00344828 sec
DELTA 0.00001655 sec
TD0 1

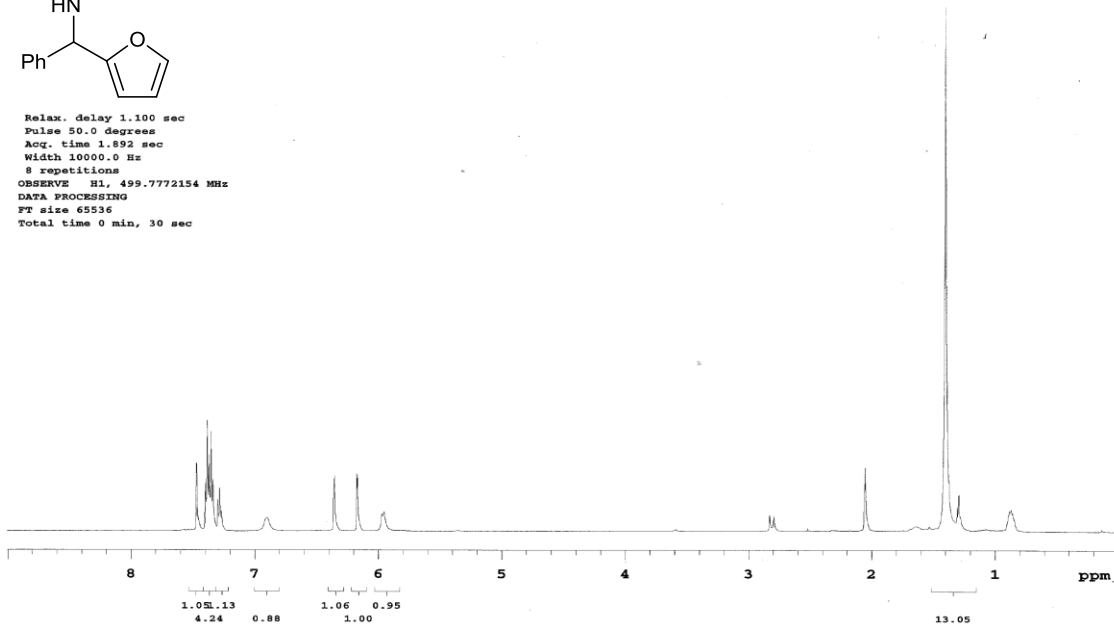
===== CHANNEL f1 =====
NUC1 13C
P1 13.00 usec
P2 26.00 usec
PL1 -0.90 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 7.00 usec
P4 14.00 usec
PCPD2 70.00 usec
PL12 26.10 dB
PL2 6.10 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

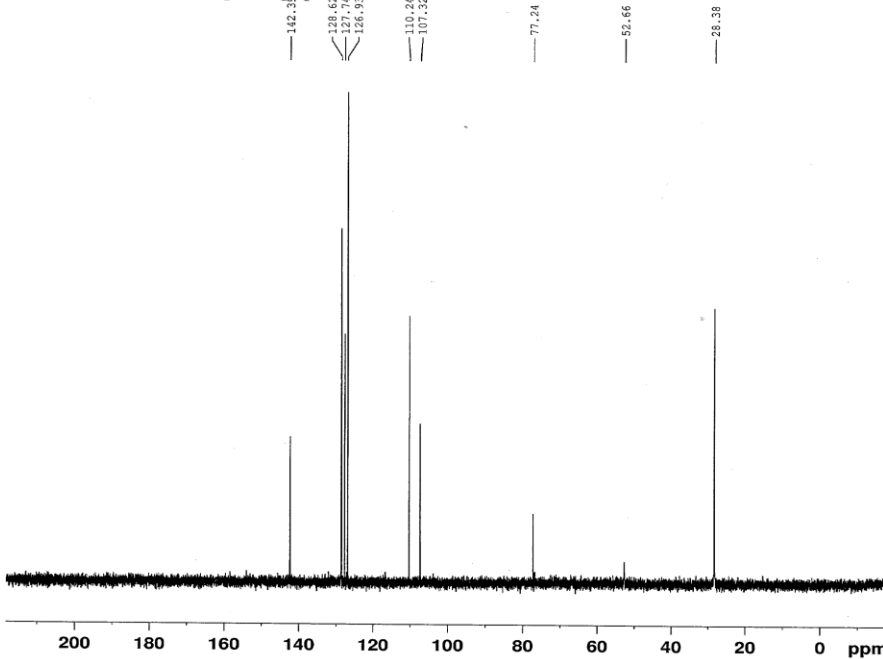




Relax. delay 1.100 sec
 Pulse 50.0 degrees
 Acq. time 1.892 sec
 Width 10000.0 Hz
 8 repetitions
 OBSERVE H1, 499.7772154 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 30 sec



decIV-221-1
 C13DEPT135 CDC13 /opt/topspin/carrera 117



```

NAME      A2_117-decIV-221
EXPNO     1
PROCNO    1
Date_     20080429
Time      23.01
INSTRUM   spect
PROBHD    5 mm CPDCH 13C
PULPROG   dept135
TD         65536
SOLVENT   CDC13
NS         256
DS         4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ        1.1010548 sec
RG         512
DW        16.800 usec
DE        6.50 usec
TE        295.0 K
CNST2     145.000000
D1        2.00000000 sec
D2        0.00344828 sec
D12       0.00002000 sec
TD0       1

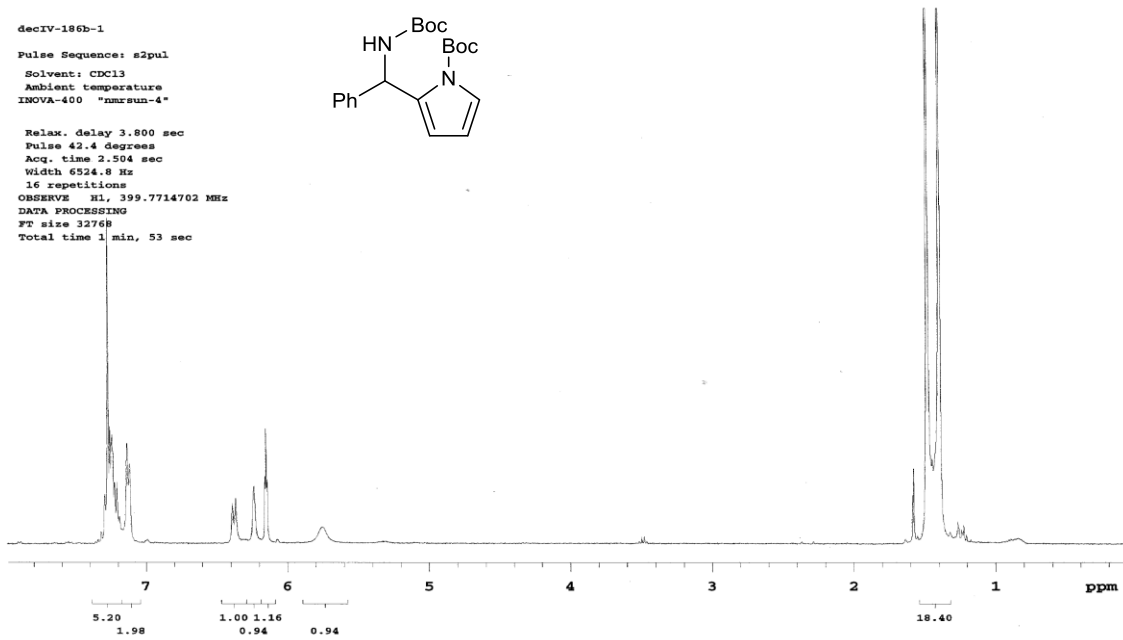
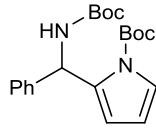
----- CHANNEL f1 -----
NUC1      13C
P1        8.20 usec
P2        16.40 usec
PL1       2.50 dB
PL1W      36.44347000 W
SF01      125.8515908 MHz

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
P3        12.50 usec
P4        25.00 usec
PCPD2     80.00 usec
PL2       3.80 dB
PL12      19.92 dB
PL2W      11.83255291 W
PL12W     0.28912020 W
SF02      500.4550018 MHz
SI        32768
SF        125.8390070 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

decIV-186b-1

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-400 "nmrsum-4"

Relax. delay 3.800 sec
Pulse 42.4 degrees
Acq. time 2.504 sec
Width 6524.8 Hz
16 repetitions
OBSERVE F1, 399.7714702 MHz
DATA PROCESSING
F2 size 32768
Total time 1 min, 53 sec



decIV-186b-1

132.15
128.16
128.52
122.56
114.26
109.95

77.25

52.26

28.48
27.97
27.89



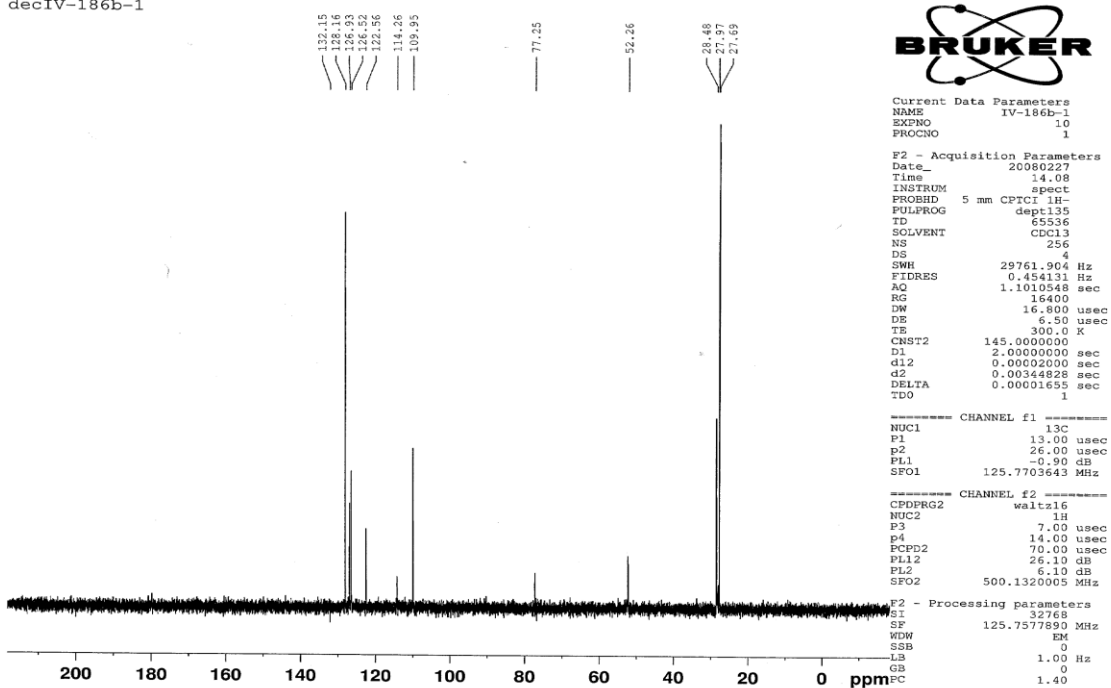
Current Data Parameters
NAME IV-186b-1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080227
Time 14.08
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
FULPROG dept135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 16400
DW 16.800 usec
DE 5.50 usec
TE 300.0 K
CNST2 145.000000
SI 2.0000000 sec
d12 0.0002000 sec
d2 0.00344828 sec
DELTA 0.0001655 sec
TDO 1

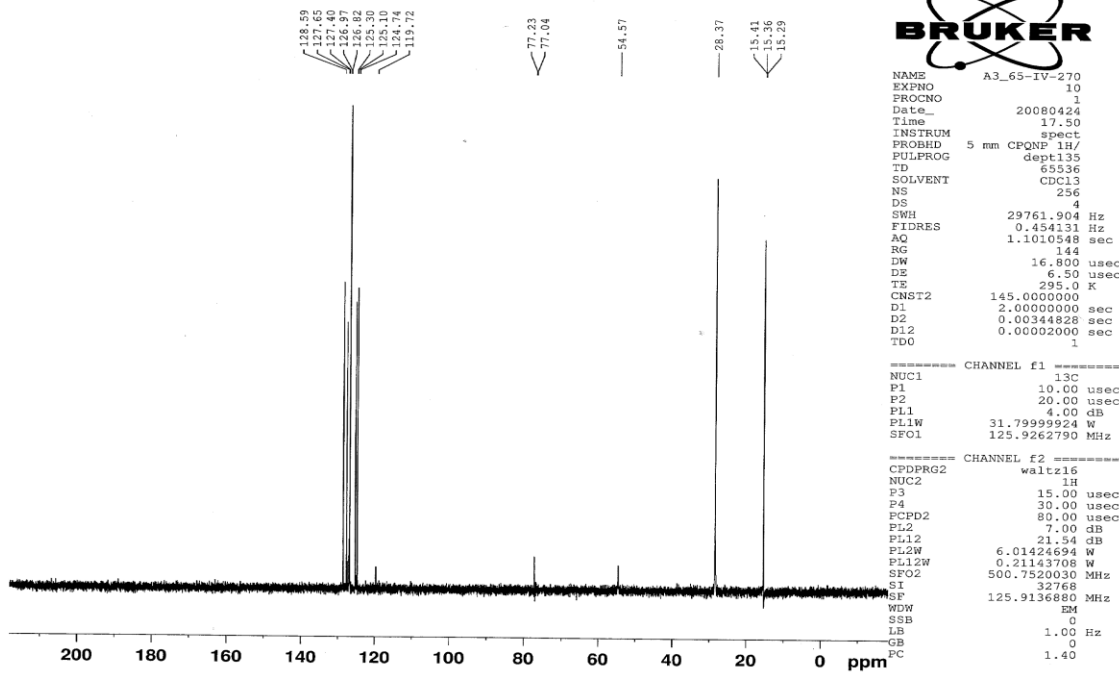
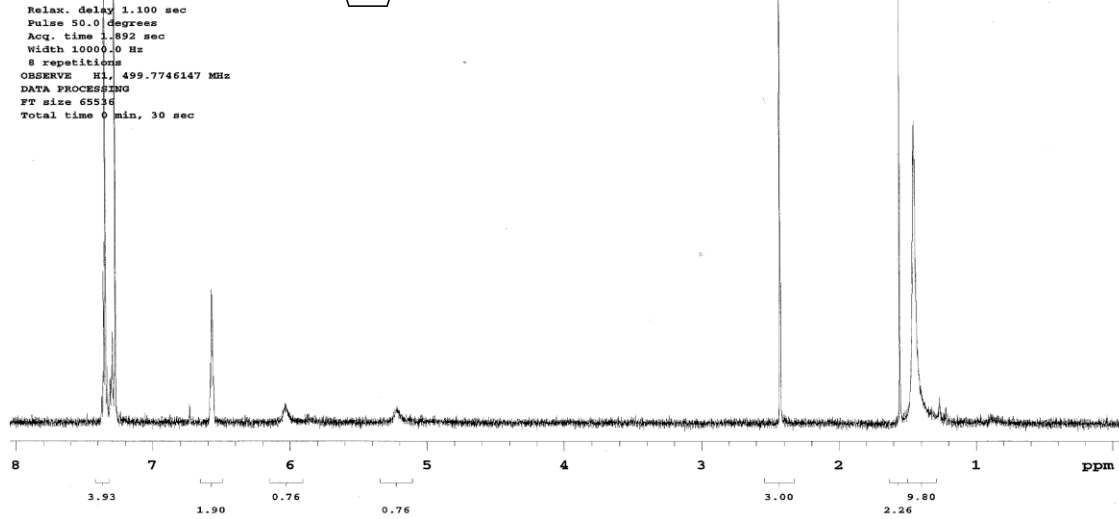
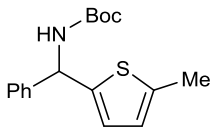
----- CHANNEL f1 -----
NUC1 13C
P1 13.00 usec
P2 26.00 usec
PL1 -0.90 dB
SFO1 125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
P3 7.00 usec
P4 14.00 usec
PCPD2 70.00 usec
PL12 26.10 dB
PL2 6.10 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



decIV-238a-1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INNOVA-500 "mrsun-5"



```

NAME      A3_65-IV-270
EXPNO     10
PROCNO    1
Date_     20080424
Time      17.50
INSTRUM   spect
PROBHD    5 mm CPQNP 1H/
PULPROG   dept135
TD         65536
SOLVENT   CDCl3
NS         256
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         144
DW         16.800 usec
DE         6.50 usec
TE         295.0 K
CNST2     145.0000000
D1         2.0000000 sec
D2         0.00344828 sec
D12        0.00002000 sec
TD0        1

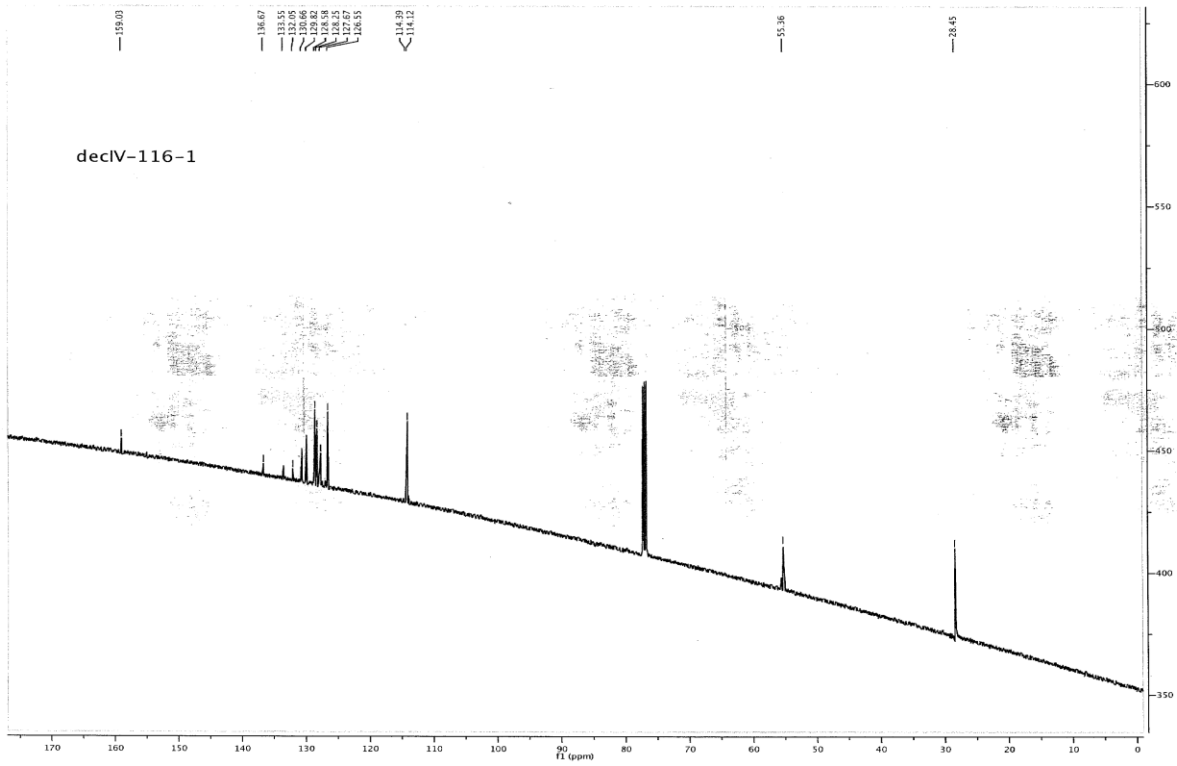
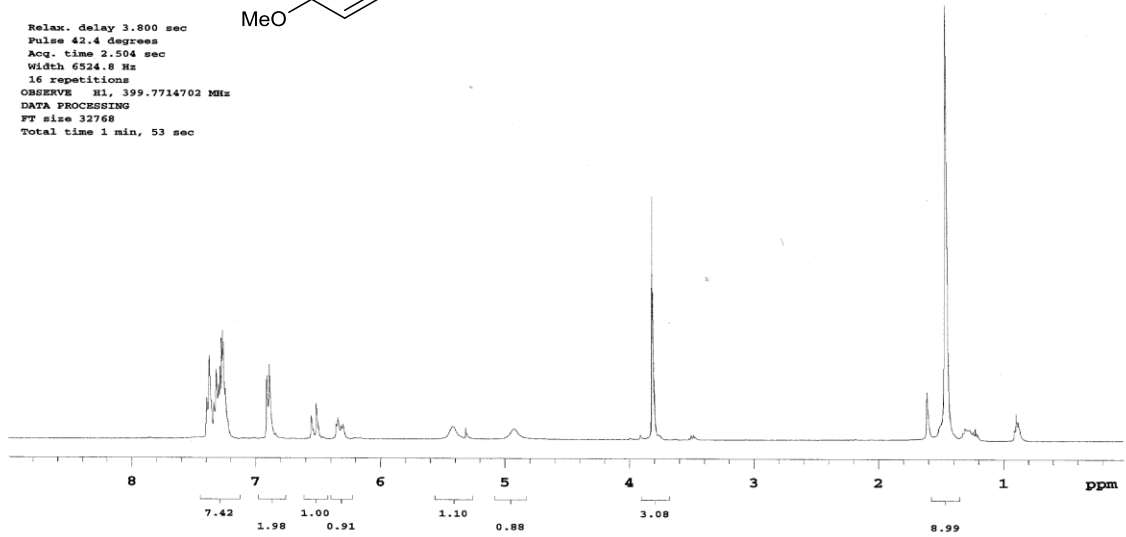
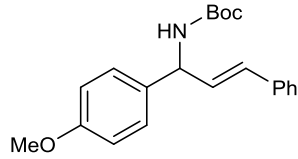
----- CHANNEL f1 -----
NUC1       13C
P1         10.00 usec
P2         20.00 usec
PL1        4.00 dB
PL1W       31.79999924 W
SFO1       125.9262790 MHz

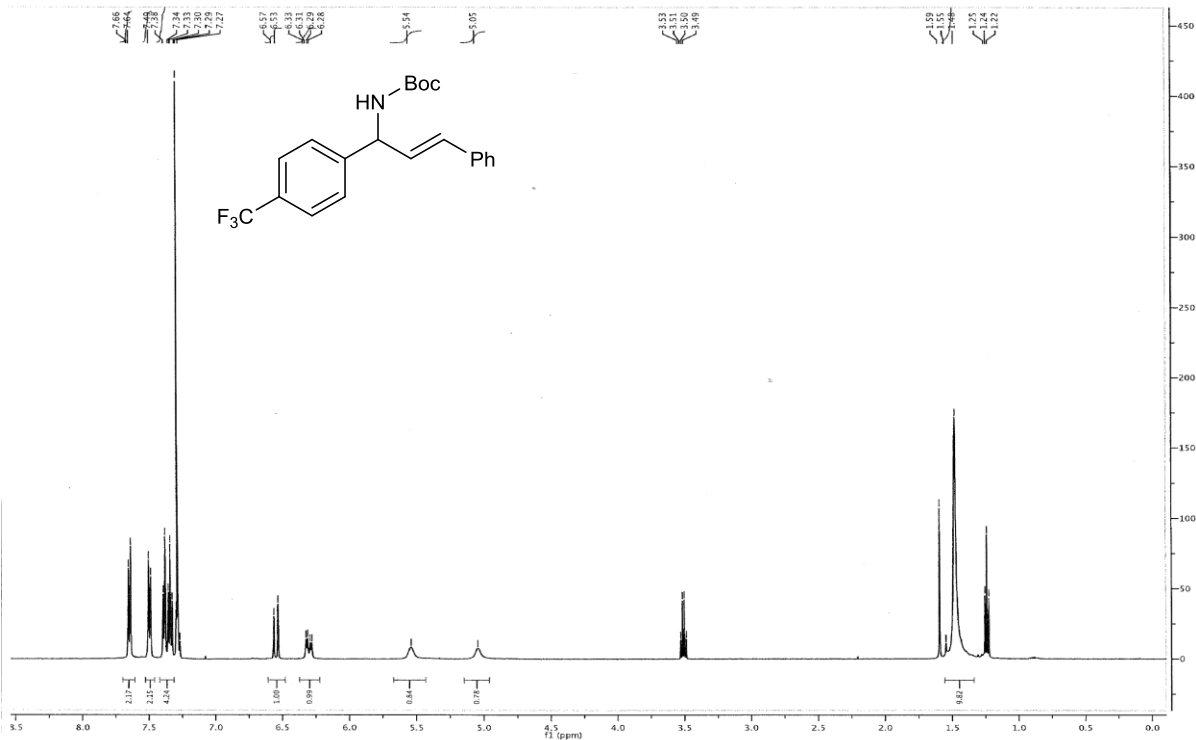
----- CHANNEL f2 -----
CPDPRG2    waltz16
NUC2       1H
P3         15.00 usec
P4         30.00 usec
PCPD2      80.00 usec
PL2        7.00 dB
PL12       21.54 dB
PL2W       6.01424694 W
PL12W      0.21143708 W
SFO2       500.7520030 MHz
SI         32768
SF         125.9136880 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

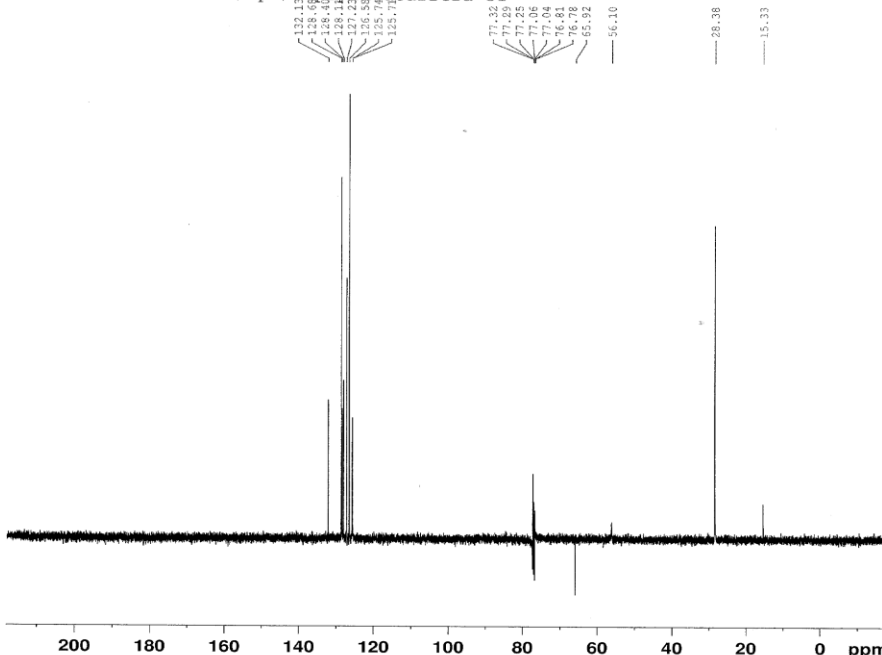

decIV-116m
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INNOVA-400 "nmrsun-4"

Relax. delay 3.800 sec
Pulse 42.4 degrees
Acq. time 2.504 sec
Width 6524.8 Hz
16 repetitions
OBSERVE F1: 399.7714702 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 53 sec





decVI-96-1c
 C13DEPT135.PU CDC13 /opt/topspin dcarrera 31

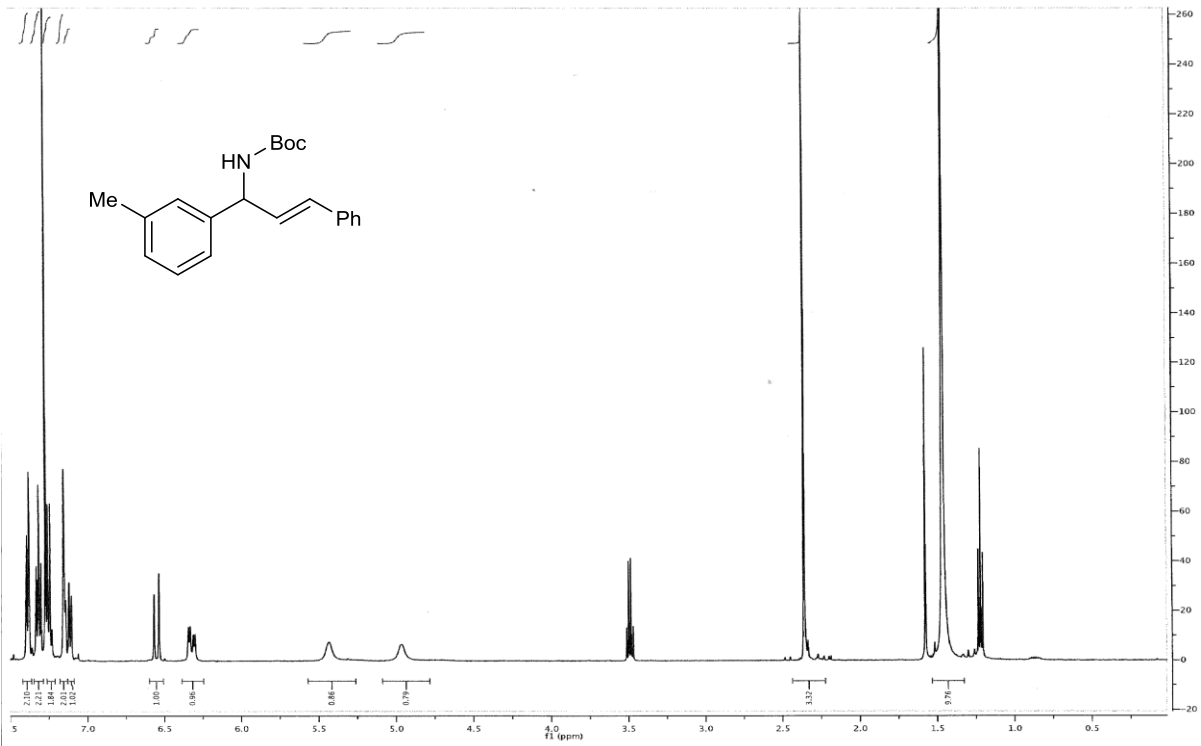


```

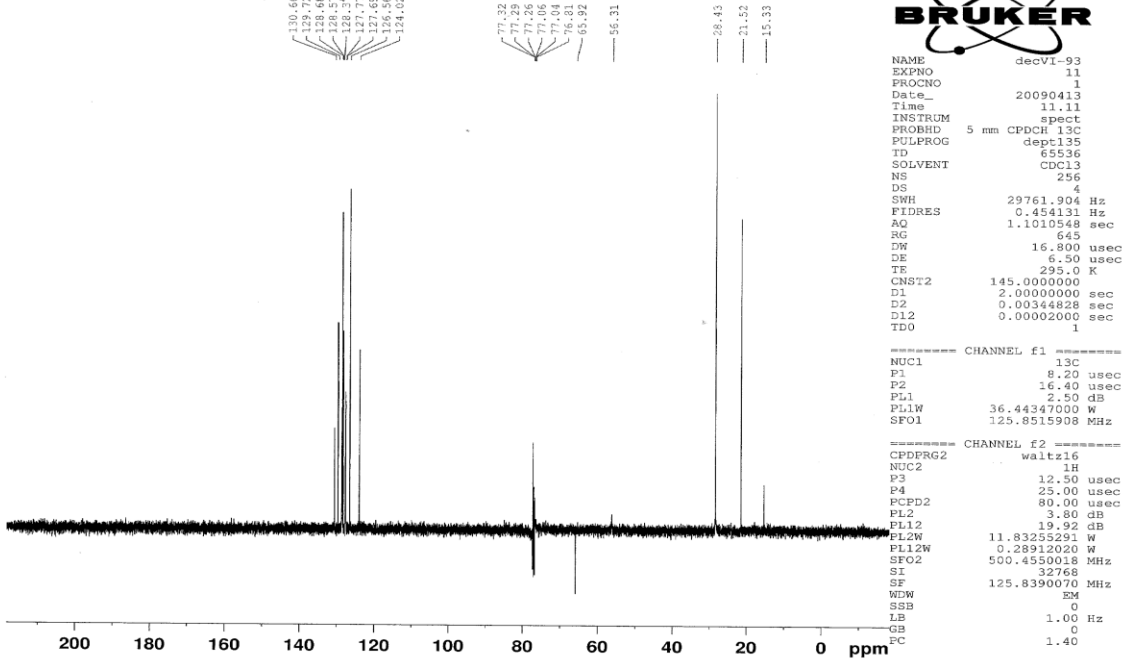
NAME          decVI-96
EXPNO         11
PROCNO        1
Date_         20090413
Time          11.36
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       dept135
TD            65536
SOLVENT       CDC13
NS            256
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            724
DW            16.800 usec
DE            6.50 usec
TE            295.0 K
CNST2         145.0000000
D1            2.0000000 sec
D2            0.00344828 sec
D12           0.00002000 sec
TDO           1

----- CHANNEL f1 -----
NUC1          13C
P1            8.20 usec
P2            16.40 usec
PL1           2.50 dB
PL1W          36.44347000 W
SFO1         125.8515908 MHz

----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
P3            12.50 usec
P4            25.00 usec
PCPD2         80.00 usec
PL2           3.80 dB
PL12          19.92 dB
PL2W          11.83255291 W
PL12W         0.28912020 W
SFO2         500.4550018 MHz
SI            32768
SF           125.8390070 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



decVI-93-1c
 C13DEPT135.PU CDCl3 /opt/topspin dcarrera 30



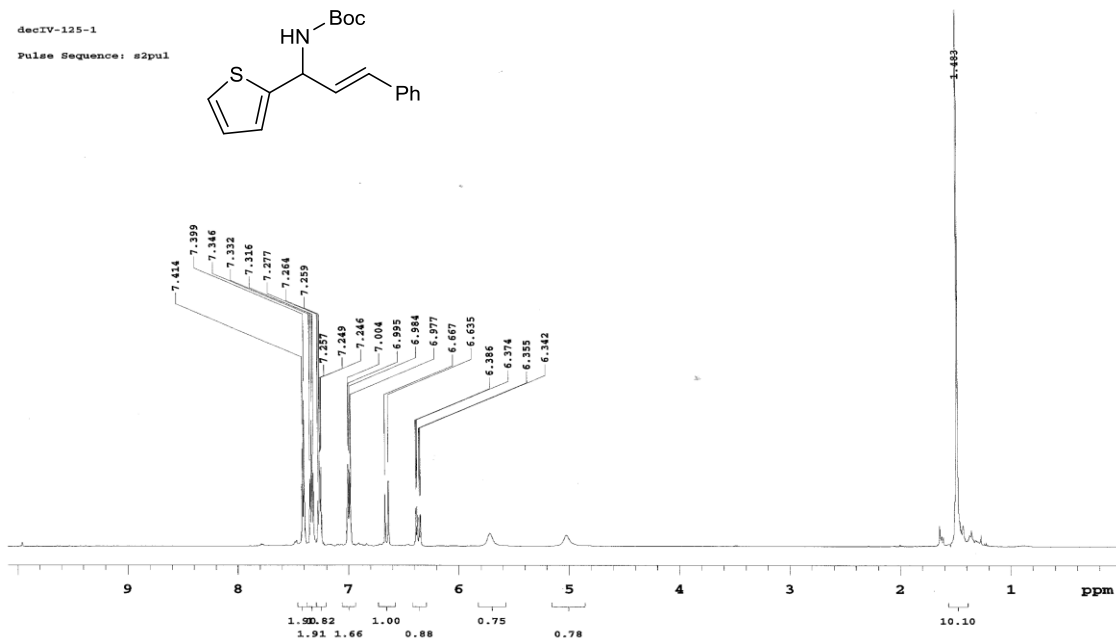
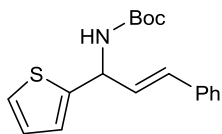
```

NAME          decVI-93
EXPNO         11
PROCNO        1
Date_         20090413
Time          11.11
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       dept135
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            645
DW            16.800 usec
DE            6.50 usec
TE            295.0 K
CNST2         145.0000000
D1            2.00000000 sec
D2            0.00344828 sec
D12           0.00002000 sec
TDO           1

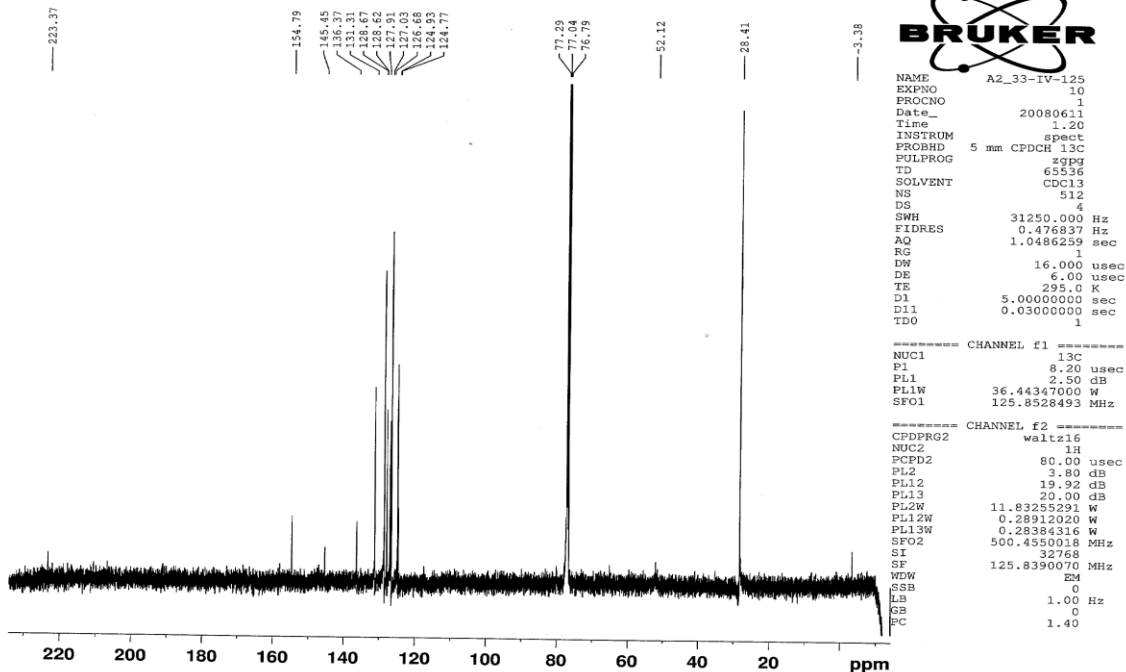
===== CHANNEL f1 =====
NUC1          13C
P1            8.20 usec
P2            16.40 usec
PL1           2.50 dB
PL1W          36.44347000 W
SF01          125.8515908 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
P3            12.50 usec
P4            25.00 usec
PCPD2         80.00 usec
PL2           3.80 dB
PL12          19.2 dB
PL2W          11.83255291 W
PL12W         0.28912020 W
SF02          500.4550018 MHz
SI            32768
SF            125.8390070 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

decIV-125-1
Pulse Sequence: s2pul



C13CPDp1.PU CDCl3 /opt/topspin dcarrera 33



```

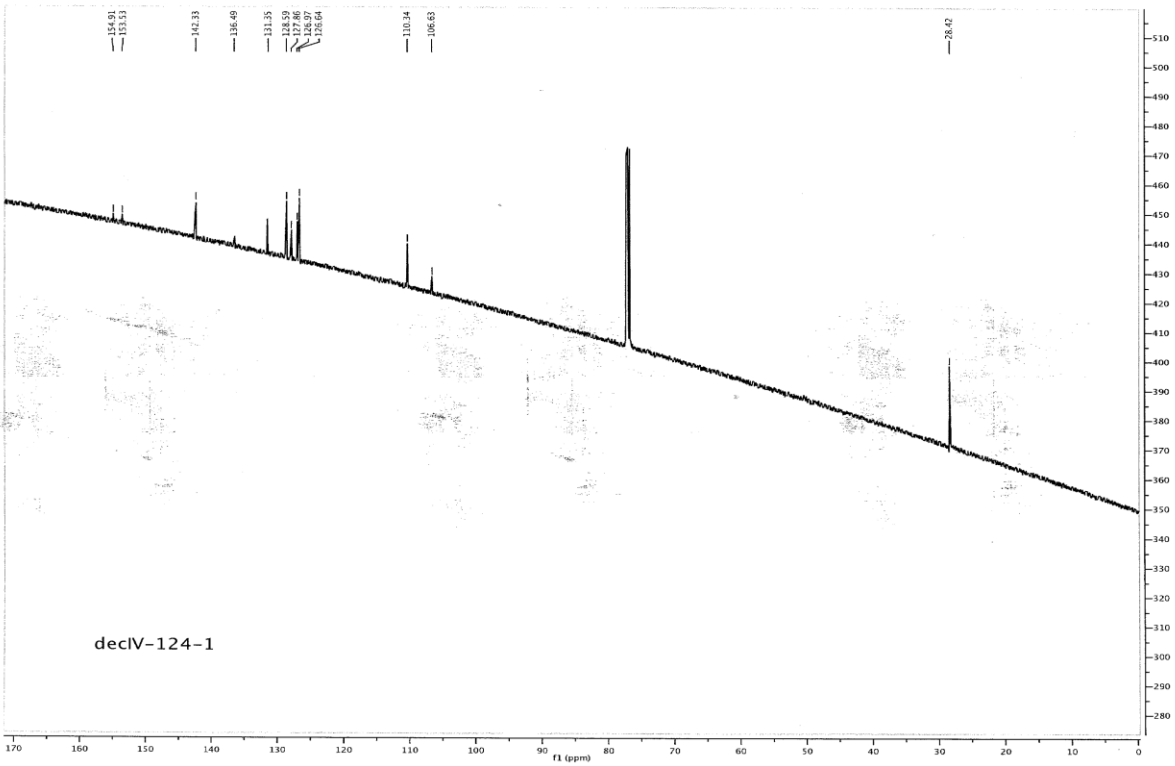
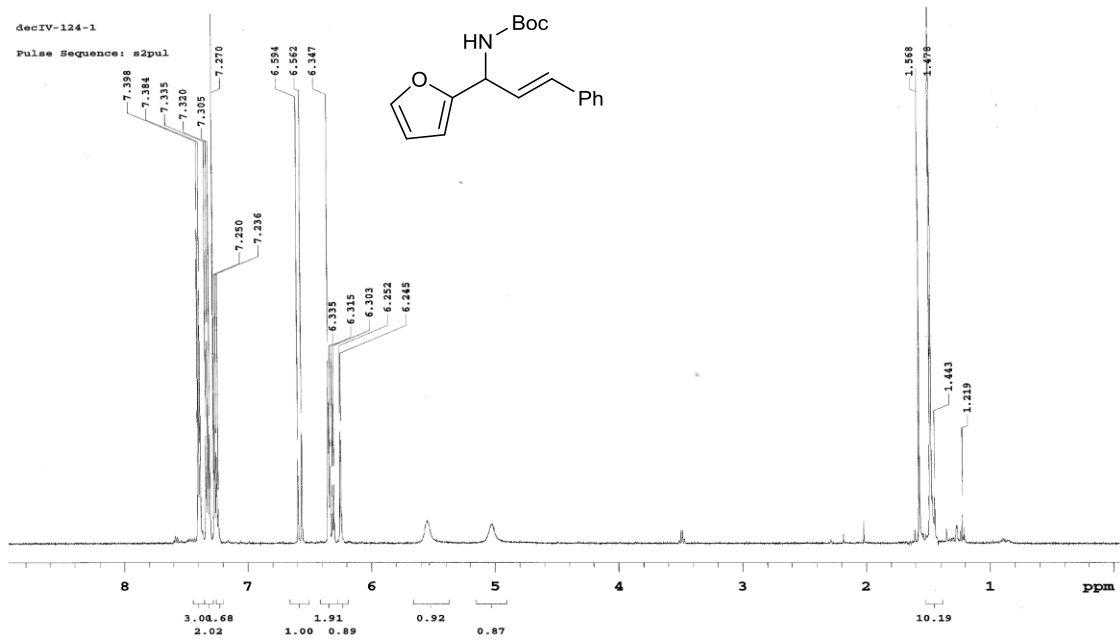
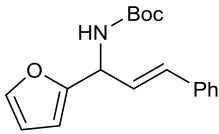
NAME      A2_33-IV-125
EXPNO     10
PROCNO    1
Date_     20080611
Time      1.20
INSTRUM   spect
PROBHD    5 mm CPDCH 13C
PULPROG   zgpg
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH        31250.000 Hz
FIDRES     0.476837 Hz
AQ         1.0486259 sec
RG         1
DW         16.000 usec
DE         6.00 usec
TE         295.0 K
D1         5.0000000 sec
D11        0.0300000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.20 usec
PL1        2.50 dB
PL1W       36.44347000 W
SF01       125.8528493 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        3.80 dB
PL12       19.92 dB
PL13       20.00 dB
PL2W       11.83255291 W
PL12W      0.28912020 W
PL13W      0.28384316 W
SF02       500.4550018 MHz
SI         32768
SF         125.8390070 MHz
WDW        EM
GB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

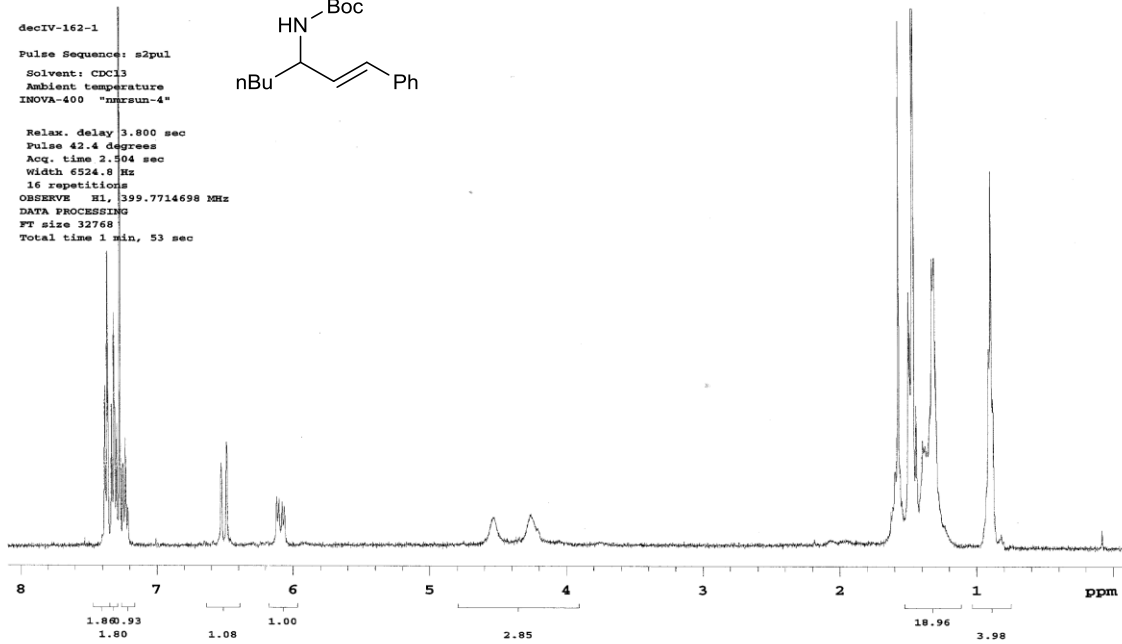
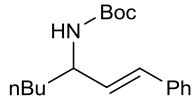
decIV-124-1

Pulse Sequence: s2pul

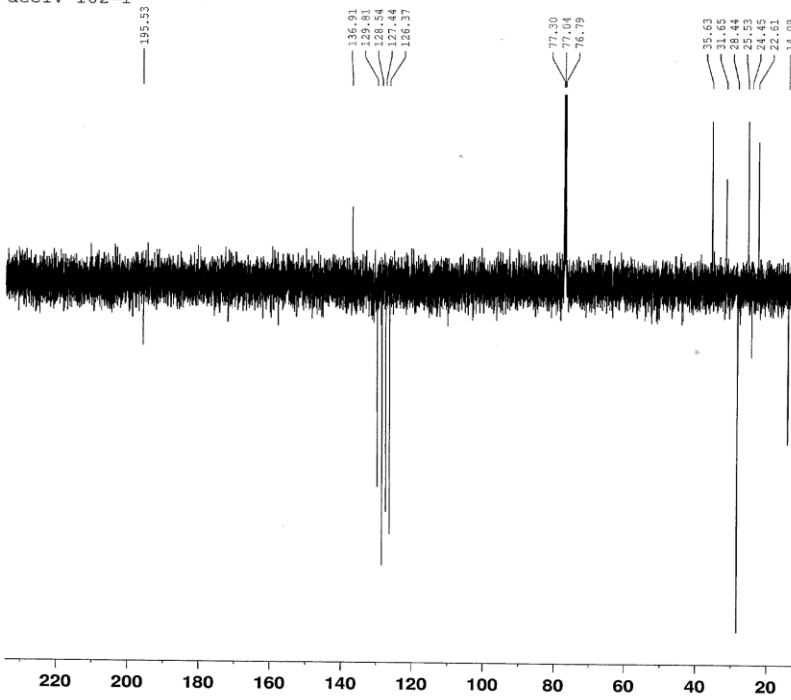


decIV-124-1

decIV-162-1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INNOVA-400 "mrsun-4"
Relax. delay 3.800 sec
Pulse 42.4 degrees
Acq. time 2.904 sec
Width 6524.8 Hz
16 repetitions
OBSERVE F1, 399.7714698 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 53 sec



decIV-162-1



Current Data Parameters
NAME al_2
EXPNO 10
PROCNO 1

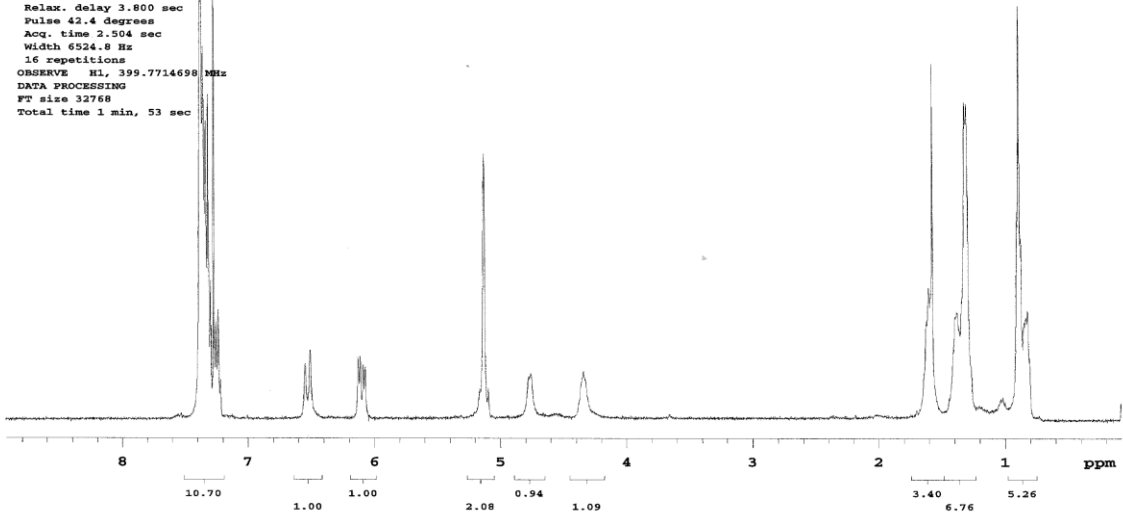
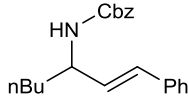
F2 - Acquisition Parameters
Date_ 20080201
Time 11.50
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT cdcl3
NS 256
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 16400
FW 16.000 usec
DE 6.00 usec
TE 295.0 K
CNST11 1.0000000
CNST2 145.0000000
D1 3.0000000 sec
d20 0.00689655 sec
DELTA 0.00001655 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 13.00 usec
P2 26.00 usec
PL1 -0.30 dB
SFO1 125.7716218 MHz

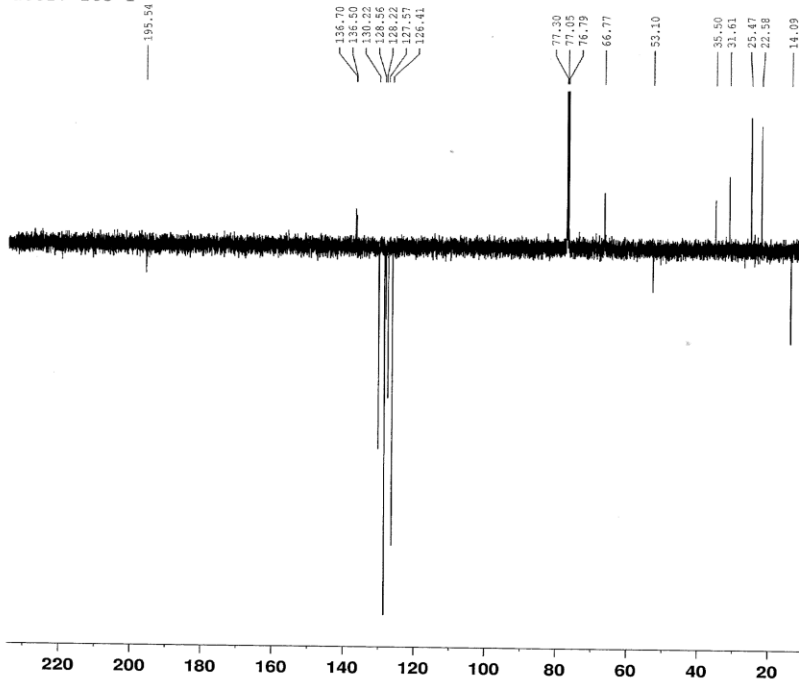
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL12 26.10 dB
PL2 6.10 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

decIV-163-1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INNOVA-400 "nmrsum-4"
Relax. delay 3.800 sec
Pulse 42.4 degrees
Acq. time 2.504 sec
Width 6524.8 Hz
16 repetitions
OBSERVE F1, 399.7714698 MHz
DATA PROCESSING
F2 size 32768
Total time 1 min, 53 sec



decIV-163-1



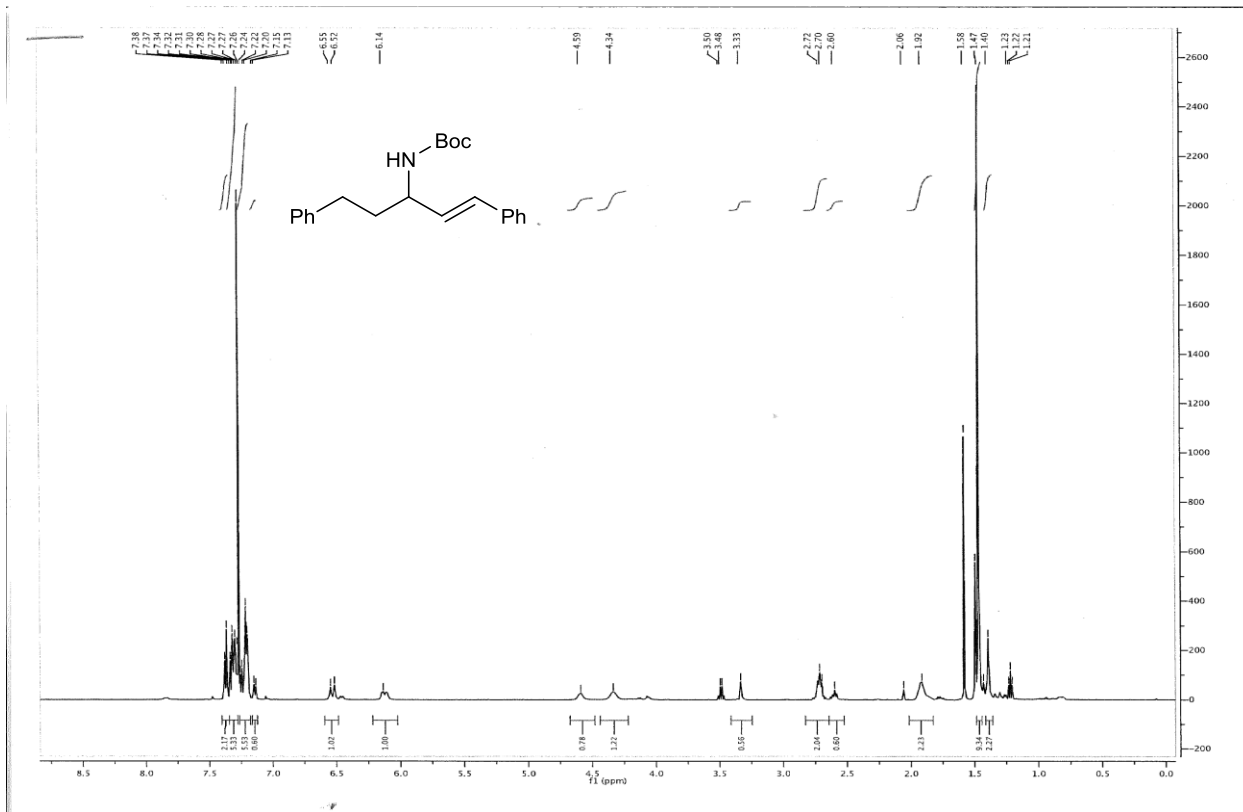
Current Data Parameters
NAME Al_3-
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080201
Time 12.10
INSTRUM spect
PROBHD 5 mm CPTCL 1H-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 16400
DW 16.000 usec
DE 6.00 usec
TE 295.0 K
CNST11 1.0000000
CNST2 145.0000000
D1 3.0000000 sec
d20 0.00689655 sec
DELTA 0.00001655 sec
TDD 1

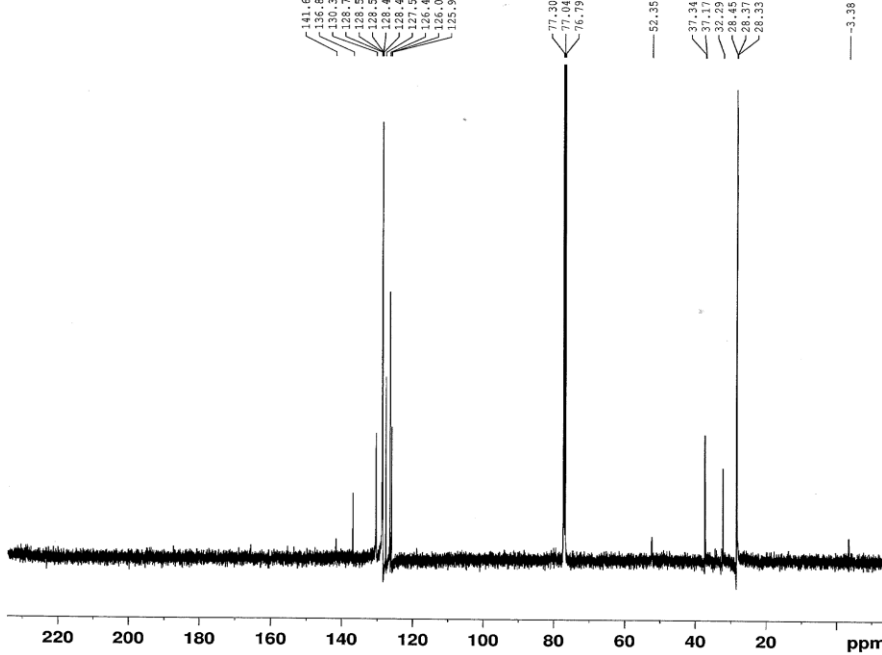
----- CHANNEL f1 -----
NUC1 13C
P1 13.00 usec
P2 26.00 usec
PL1 -0.90 dB
SFO1 125.7716218 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 26.10 dB
PL2 6.10 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



decIV-76-1
 C13CPDpl.PU CDCl3 /opt/topspin dcr 92

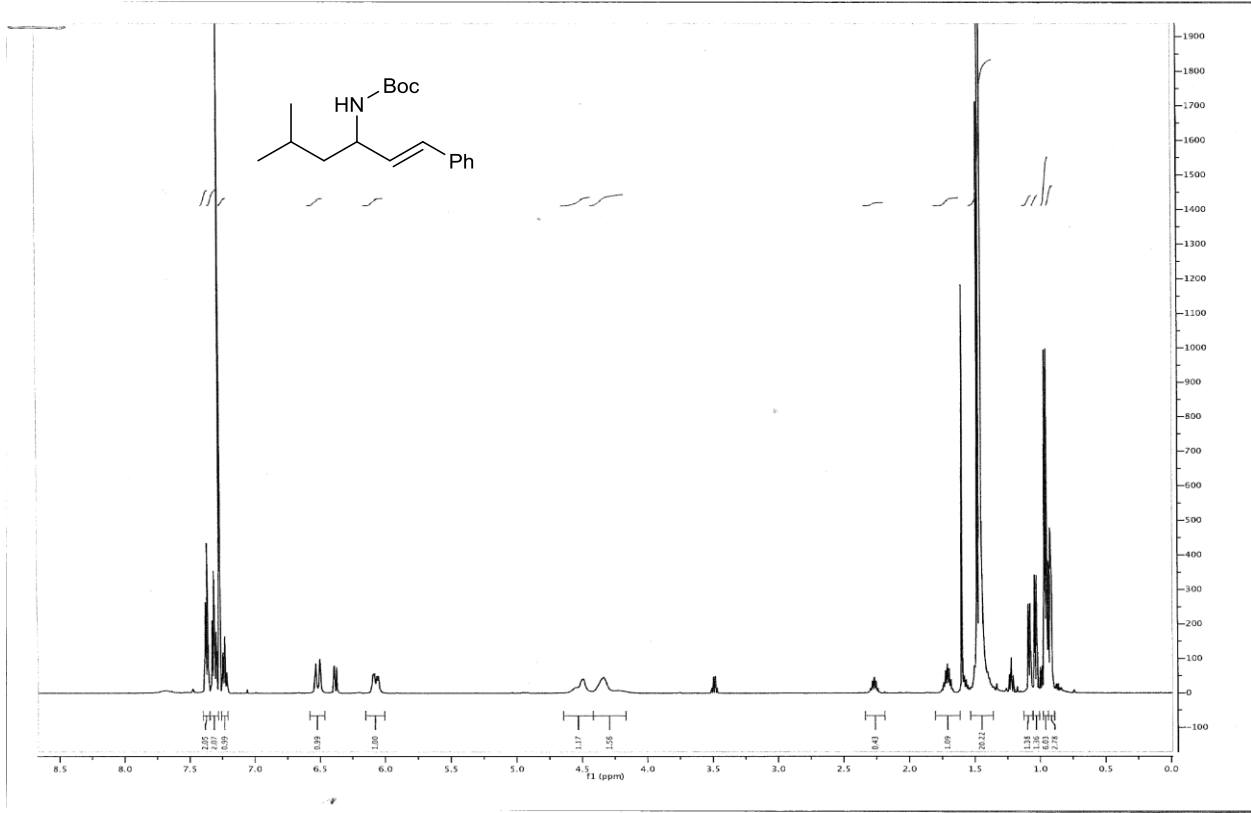


BRUKER

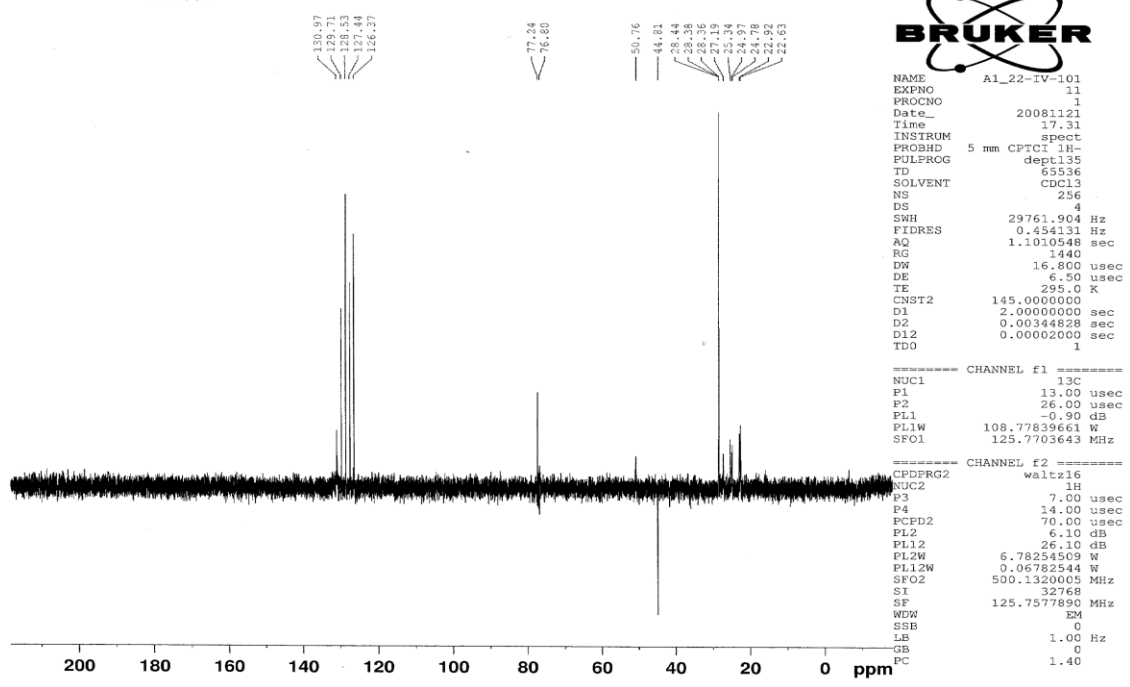
NAME A2_92-decIV-76
 EXPNO 10
 PROCNO 1
 Date_ 20080610
 Time 0.42
 INSTRUM spect
 PROBHD 5 mm CPDCH 13C
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1
 DW 16.000 usec
 DE 6.00 usec
 TE 295.0 K
 D1 5.0000000 sec
 D11 0.0300000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.20 usec
 PL1 2.50 dB
 PL1W 36.44347000 W
 SF01 125.8528493 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 3.80 dB
 PL12 19.92 dB
 PL13 20.00 dB
 PL2W 11.83255291 W
 PL12W 0.28912020 W
 PL13W 0.28364316 W
 SF02 500.4550018 MHz
 SI 32768
 SF 125.8390070 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



decIV-101-1 carbon

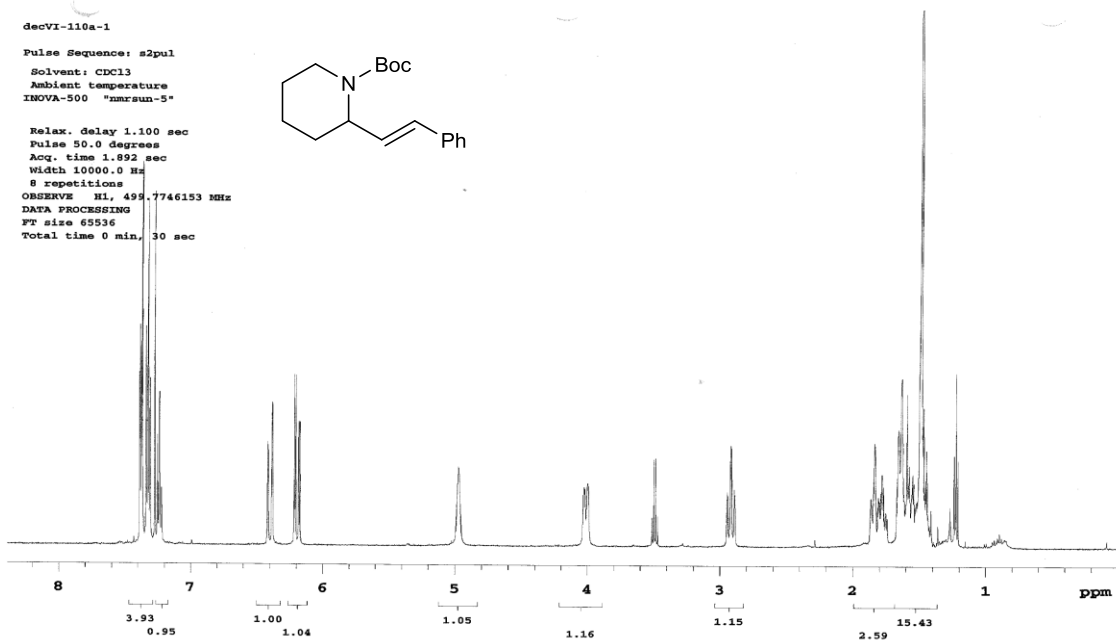
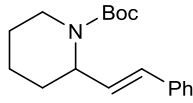


decVI-110a-1

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INNOVA-500 "nmrsm-5"

Relax. delay 1.100 sec
Pulse 50.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
8 repetitions

OBSERVE F1, 499.7746153 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 30 sec



decVI-110a-1carbon

130.70
128.74
128.58
127.39
126.25

79.49
77.30
77.04
76.75

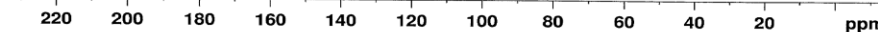
29.54
28.50
25.58
19.71

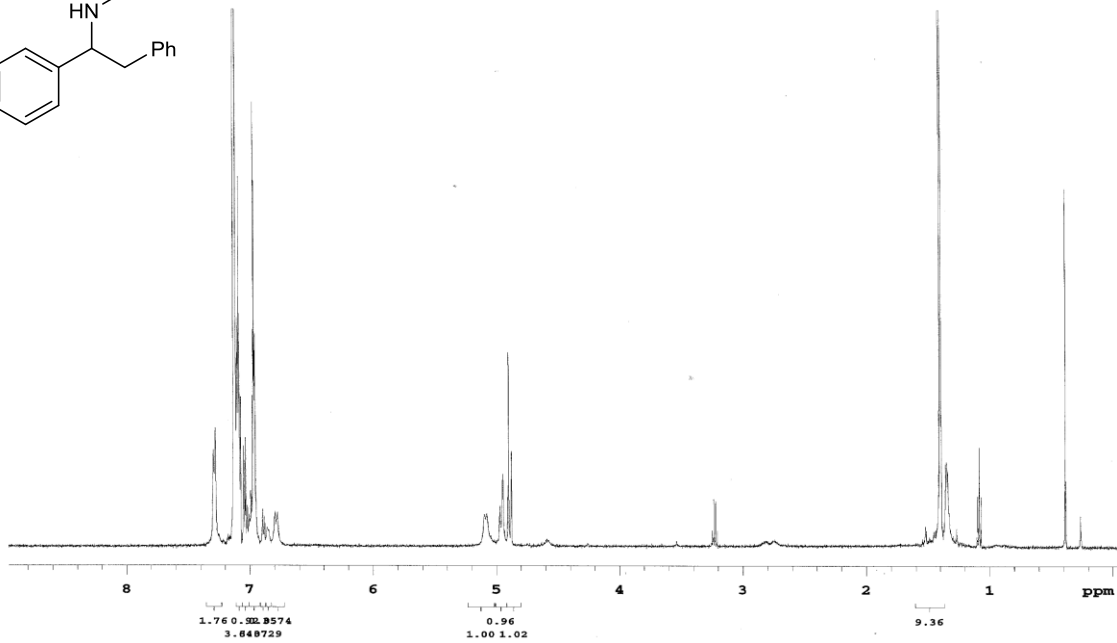
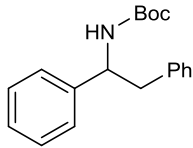


NAME decVI-110-1
EXPNO 10
PROCNO 1
Date_ 20090608
Time 19.00
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 144
DM 16.000 usec
DE 6.50 usec
TE 295.0 K
CNST2 145.000000
CNST11 1.000000
D1 2.0000000 sec
D20 0.00689655 sec
TD0 1

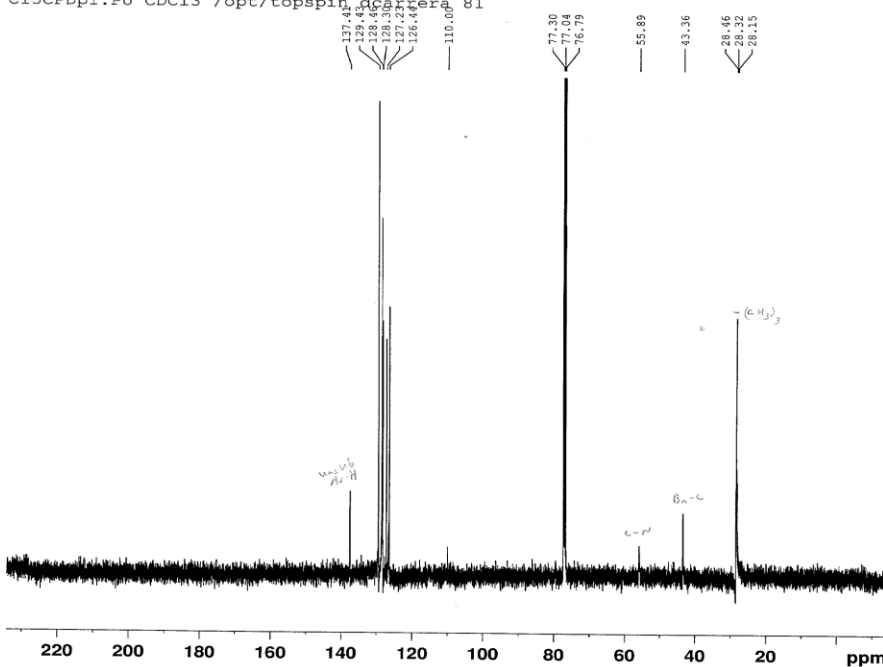
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 4.00 dB
PL1W 31.79999924 W
SFO1 125.9275385 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 21.54 dB
PL2W 6.01424694 W
PL12W 0.21143708 W
SFO2 500.7520030 MHz
SI 32768
SF 125.9136890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





decV-20-3
C13CPDpl.PU CDC13 /opt/topspin/decv-20-3

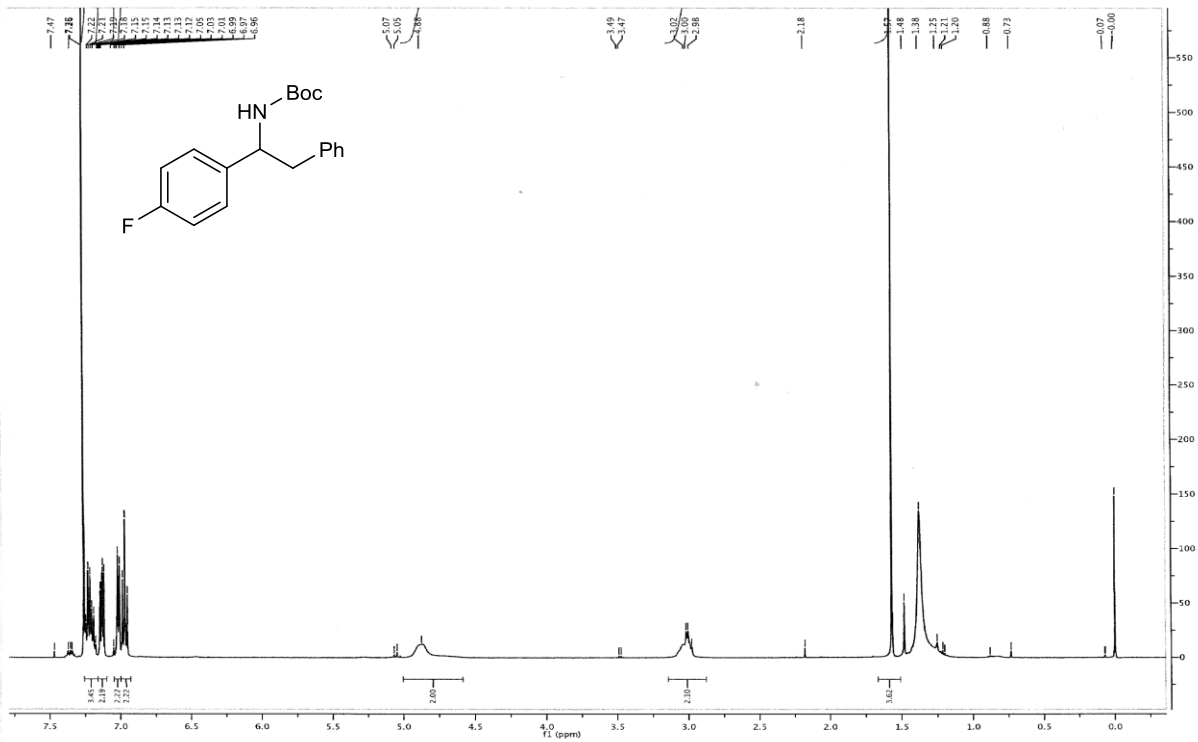


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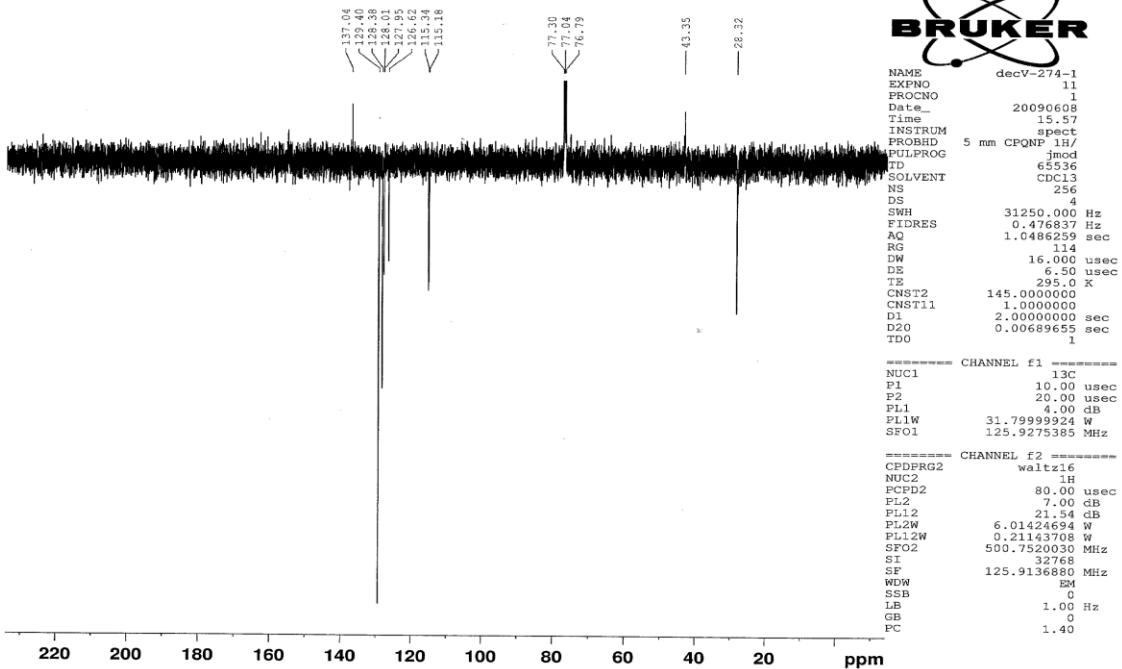
NAME      A2_81-decv-20
EXPNO     10
PROCNO    1
Date_     20080531
Time      16.59
INSTRUM   spect
PROBHD    5 mm CPDCH 13C
PULPROG   zgpg
TD         65536
SOLVENT   CDC13
NS         1024
DS         4
SWH       31250.000 Hz
FIDRES    0.476837 Hz
AQ         1.0486259 sec
RG         1
DW         16.000 usec
DE         6.00 usec
TE         295.0 K
D1         2.0000000 sec
D11        0.0300000 sec
TDO        1

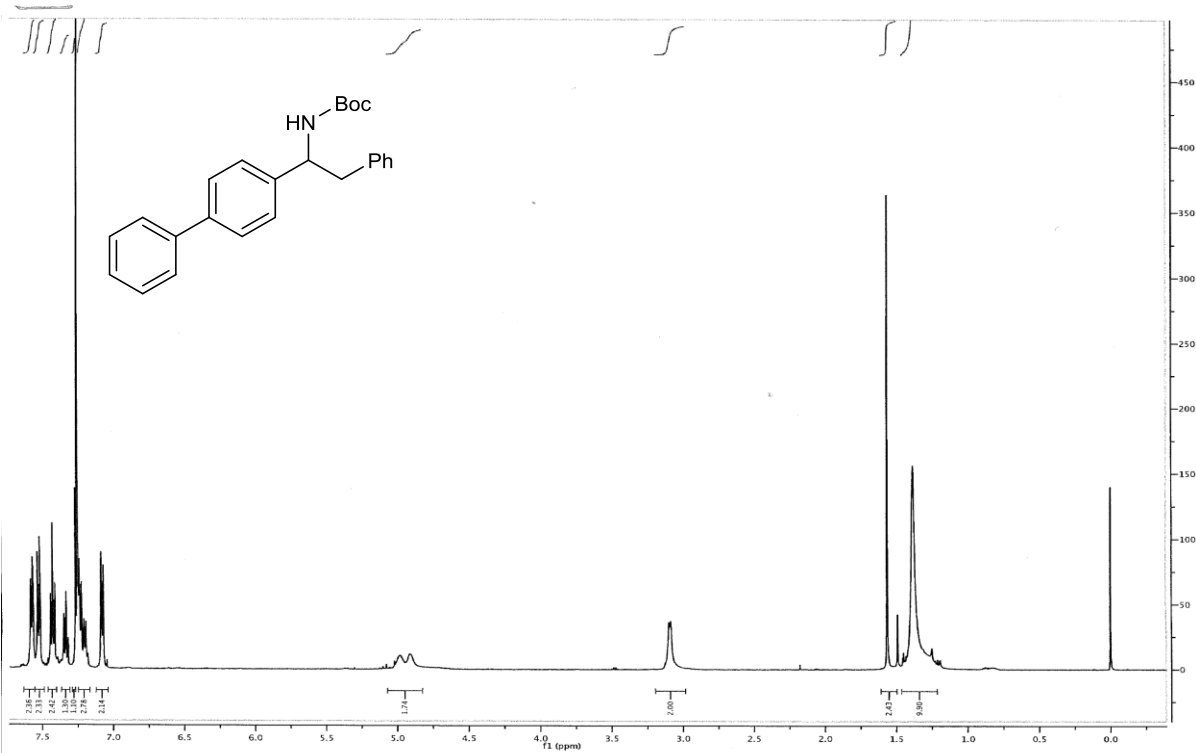
===== CHANNEL f1 =====
NUC1       13C
P1         8.20 usec
PL1        2.50 dB
PL1W       36.44347000 W
SF01       125.8528493 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        3.80 dB
PL12       19.92 dB
PL13       20.00 dB
PL2W       11.83255291 W
PL12W      0.28912020 W
PL13W      0.28364316 W
SF02       500.4550018 MHz
SI         32768
SF         125.8390070 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

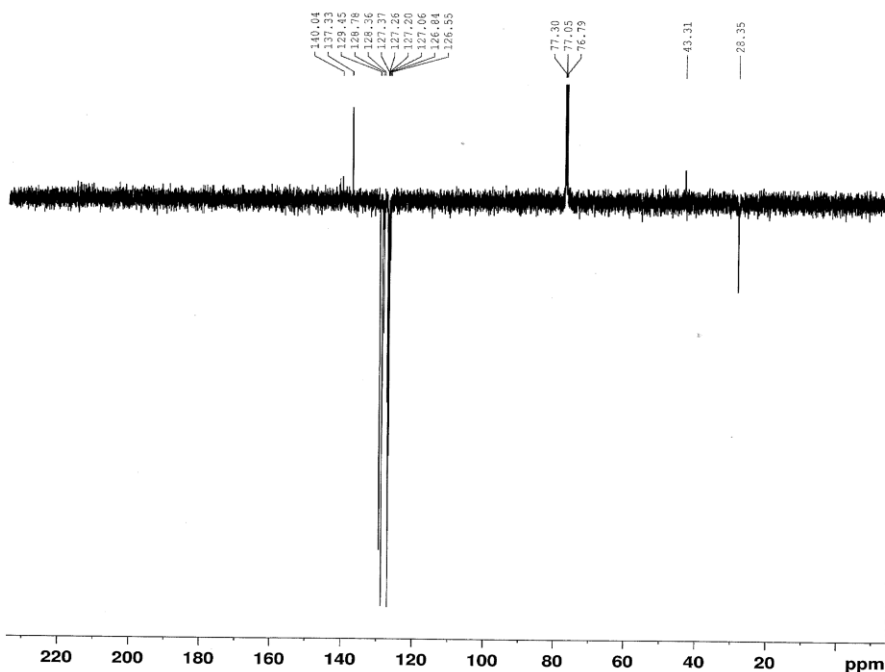


decV-274-1carbon





decV-270-1carbon



```

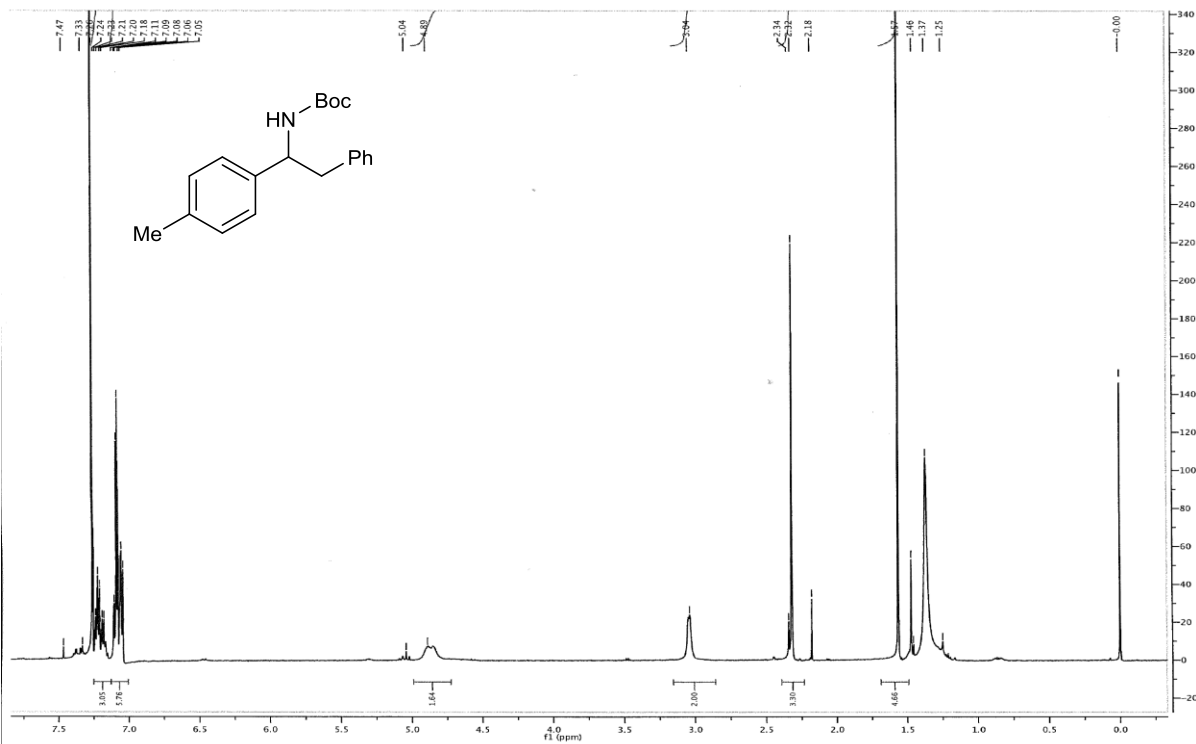
NAME      decV-270-1
EXPNO     11
PROCNO    1
Date_     20090608
Time      15.35
INSTRUM   spect
PROBHD    5 mm CPQNP 1H/
PULPROG   3mod
TD         65536
SOLVENT   CDCl3
NS         256
DS         4
SWH       31250.000 Hz
FIDRES    0.476837 Hz
AQ         1.0486259 sec
RG         114
DW         16.000 usec
DE         6.50 usec
TE         295.0 K
CNST2     145.0000000
CNST11    1.0000000
D1         2.0000000 sec
D20        0.00689655 sec
TD0        1
  
```

```

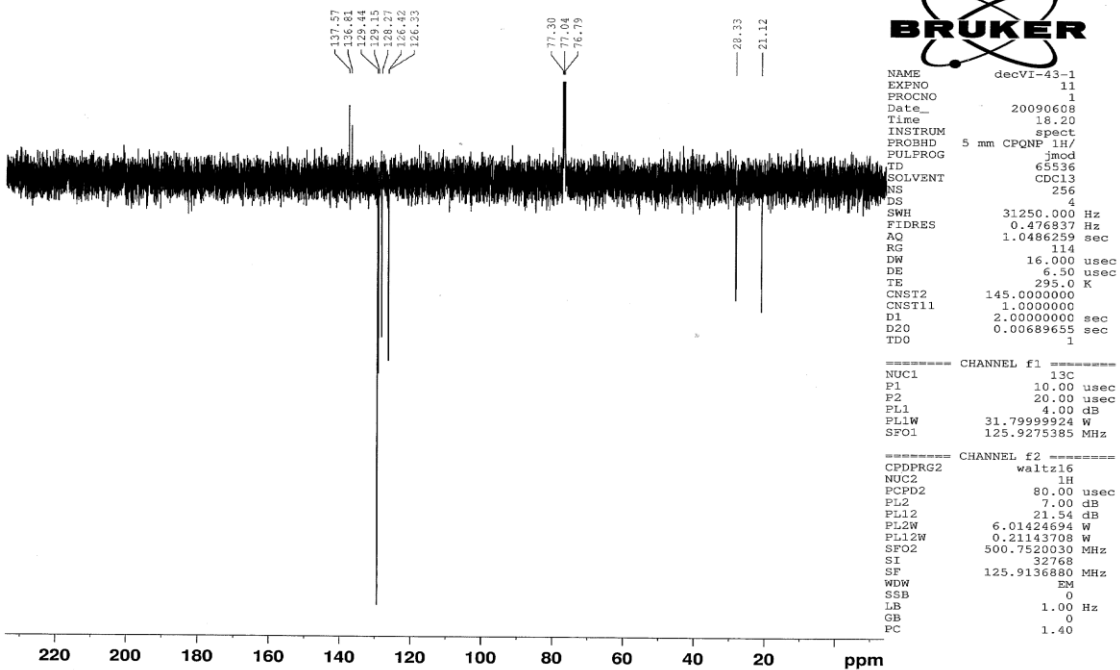
----- CHANNEL f1 -----
NUC1      13C
P1        10.00 usec
P2        20.00 usec
PL1       4.00 dB
PL1W      31.79999924 W
SFO1      125.9275385 MHz
  
```

```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       7.00 dB
PL12      21.54 dB
PL2W      6.01424694 W
PL12W     0.21143708 W
SFO2      500.7520030 MHz
SI         32768
SF         125.9136880 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```



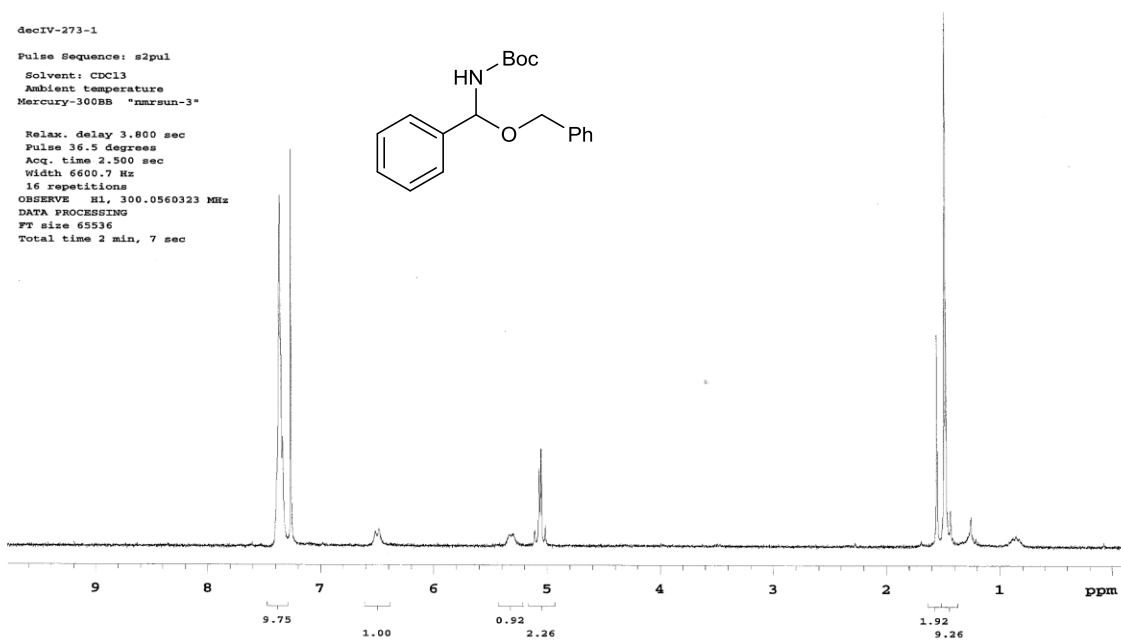
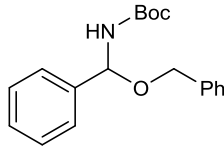
decVI-43-1carbon



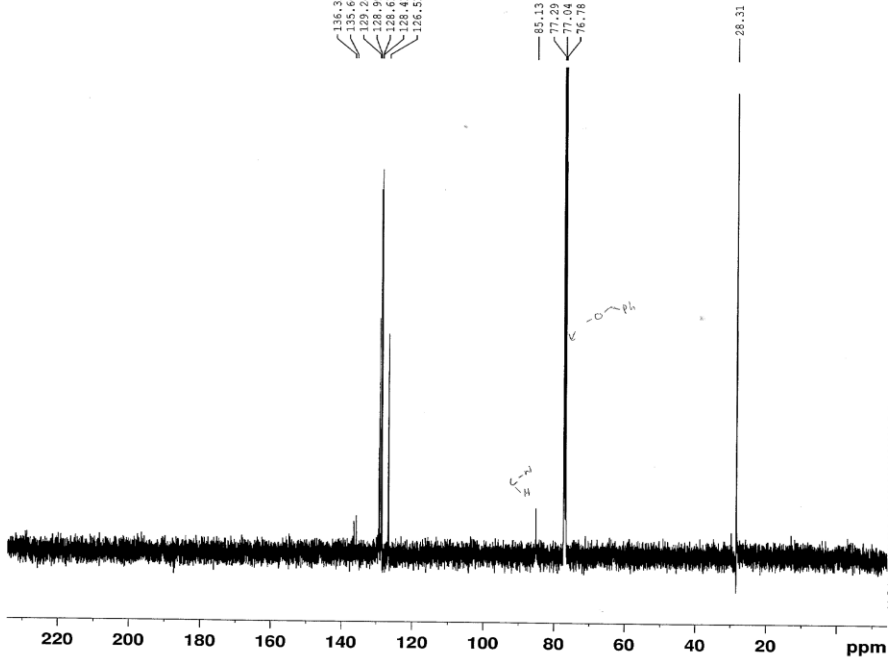
decIV-273-1

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mmsun-3"

Relax. delay 3.800 sec
Pulse 16.5 degrees
Acq. time 2.500 sec
Width 6600.7 Hz
16 repetitions
OBSERVE H1, 300.0560323 MHz
DATA PROCESSING
F2 size 65536
Total time 2 min, 7 sec



decIV-273-1
C13CPDpl.PU CDCl3 /opt/topspin dcarrera 87



NAME A2_87-DECIV-273
EXPNO 10
PROCNO 1
Date_ 20080531
Time 15.01
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1
DW 16.000 usec
DE 6.00 usec
TE 295.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 2.50 dB
PL1W 36.44347000 W
SF01 125.8528493 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.80 dB
PL12 19.92 dB
PL13 20.00 dB
PL2W 11.83256291 W
PL12W 0.28912020 W
PL13W 0.28364316 W
SF02 500.4550018 MHz
SI 32768
SF 125.8390070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40