Supporting Information

Aerobic Oxygenative Cleavage of Electron-Deficient Triple Bonds in the Gold-Catalyzed Cyclization of 1,6-Enynes

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1. General Information

All solvents were dried and distilled according to the standard methods before use.[1] Au(SPhos)Cl, Au(JohnPhos)Cl, Au(*t*Bu-XPhos)Cl and Au(*t*Bu-DavePhos)Cl were prepared from $Au(SMe_2)Cl$ and the corresponding phosphine ligand following the standard protocol.[2] $Au(PPh_3)Cl$, $AuCl_3$, AgOTf, $AgSbF_6$ and $AgNTf_2$ were purchased from Strem Chemicals or Aldrich Chemicals and was stored in a dry-keeper before use. Propiolamide substrates were prepared via standard DCC coupling[3] followed by their *N*-allylation. Propiolamides with aryl substituted alkynes (**1a**, **1c**, **1d**, **1g** and **1h**) were prepared by coupling of the propiolamide precursors with boronic acids.[4] (2-(bromomethyl)allyloxy)(tert-butyl)dimethyl silane,[5] (3-bromoprop-1-en-2-yl)benzene[6] for the preparation of substrates **1k**, **1l** and **1m**, respectively, were prepared according to the literature.

TLC (Thin-layer chromatography) analysis was carried out on Merck silica gel 60 F254 TLC plate and was visualized with UV lamp and potassium permanganate solution. Flash column chromatography was performed on Kieselgel 60 (230-400 mesh). ^1H, ^{13}C NMR and COSY spectra were recorded on a Varian (Mercury, 300 MHz and 400 MHz) spectrometer with TMS as an internal standard. High resolution mass spectra (HRMS) were obtained from Korea Basic Science Institute (KBSI) at Seoul and Daegu. X-ray crystallographic analysis was conducted at Gyeongsang National University.

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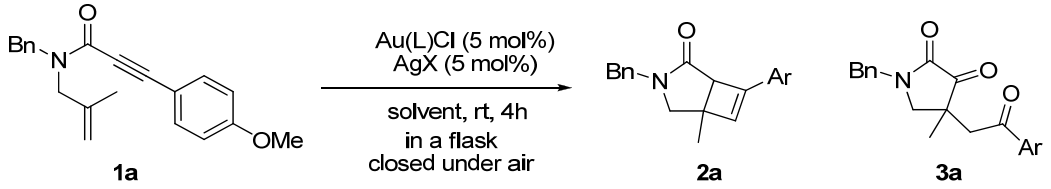
[1] Armarego, W. L. F.; Chai, C. L. L. *Purification of Laboratory Chemicals;* Elsevier: Oxford, 2009.
[2] (a) Mézailles, N.; Ricard, L.; Gagosz, F. *Org. Lett.* **2005**, *7*, 4133; (b) de Frémont, P.; Scott, N. M.; Stevens, E. D.; Nolan, S. P. *Organometallics*, **2005**, *24*, 2411.
[3] Srinivasachari, S.; Fichter, K. M.; Reineke, T. M. *J. Am. Chem. Soc.* **2008**, *130*, 4618.
[4] M. -B. Zhou, W. -T. Wei, Y. -X. Xie, Y. Lei, and J. -H. Li, *J. Org. Chem* . **2010**, 75, 5635
[5] Couladouros, E. A.; Dakanali, M.; Demadis, K. D.; Vidali, V. P. *Org. Lett.* **2009**, *11*, 4430.
[6] Geoghegan K.; Evans, P.; Rozas, I.; Alkorta, I. *Chem. Eur. J.* **2012**, *18*, 13379.

2. Screening of Reaction Conditions

The following reactions were conducted using the substrate **1a** inside a flask closed under air, *i.e.* without rigorous exclusion of air. The catalyst were generated *in-situ*: Au(L)Cl (5 mol %) and AgX (5 mol %) was added to a solution of **1a** (0.1 M) in a vial closed under air.

Table S1. Screening of reaction conditions employing **1a**[a]



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

Entry	Ligand (L)	Ag salt (X)	Solvent	**2a** (%)[b]	**3a** (%)[b]
1	SPhos	$AgSbF_6$	1,4 dioxane	-	75
2	JohnPhos	$AgSbF_6$	1,4 dioxane	-	66
3	*t*Bu-XPhos	$AgSbF_6$	1,4 dioxane	-	36
4	*t*Bu-DavePhos	$AgSbF_6$	1,4 dioxane	-	55
5	PPh_3	$AgSbF_6$	1,4 dioxane	-	37
6	IPr	$AgSbF_6$	1,4 dioxane	-	74
7	$AuCl_3$	-	1,4 dioxane	-	-
8	SPhos	AgOTf	1,4 dioxane	-	51
9	SPhos	$AgNTf_2$	1,4 dioxane	-	45
10	SPhos	$AgBF_4$	1,4 dioxane	24	52
11	SPhos	$AgSbF_6$	$CHCl_3$	74	<5
12	SPhos	$AgSbF_6$	toluene	25	45
13	SPhos	$AgSbF_6$	DCE	66	<5
14	SPhos	$AgSbF_6$	THF	-	54
15	SPhos	$AgSbF_6$	diethyl ether	7	63
16	**SPhos**	**$AgSbF_6$**	**CF_3CH_2OH**	**-**	**86**

[a] Au(I)-complex were generated *in-situ*. [b] Crude yield (^{1}H-NMR, 1,3,5-trimethoxy-benzene as an internal standard).

3. Incorporation of ^{18}O into 3b

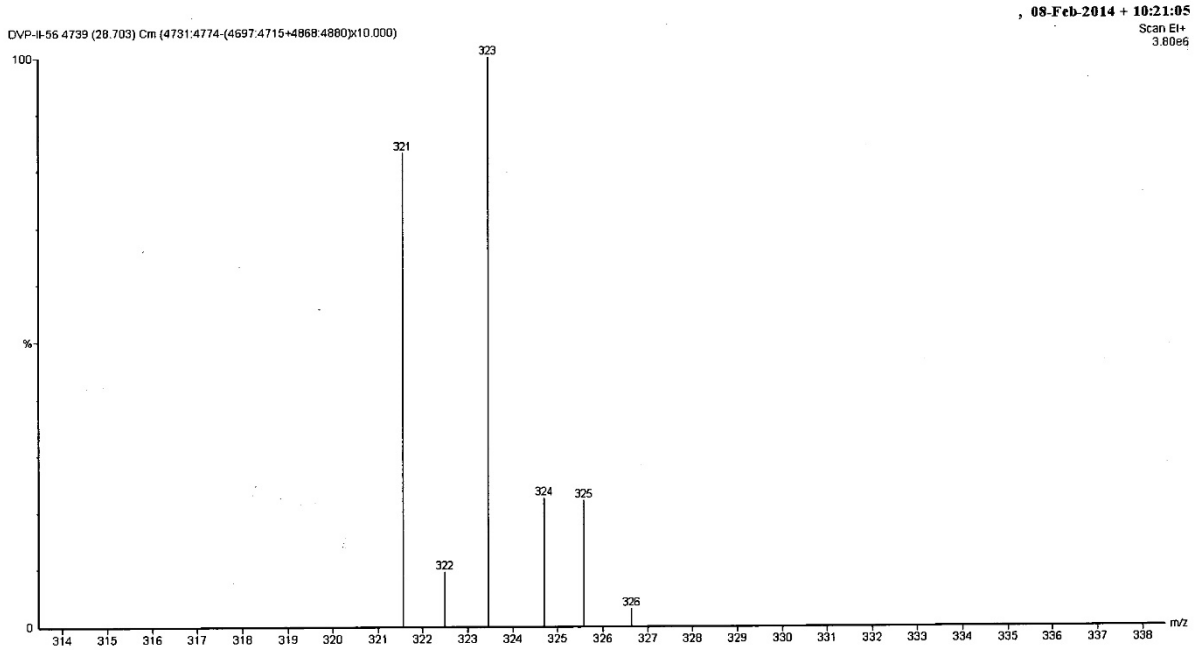
3.1. Exchange with $^{18}OH_2$

To test whether oxygen from $^{18}OH_2$ (99 atom %) is incorporated into the product (**3b**), the reaction of **1b** was conducted in the presence of 15 equiv. of $^{18}OH_2$ in 1,4-dioxane under otherwise standard conditions. To remove the possibility of carbonyl oxygen exchange with adventitious water via a hydrate after the formation of 1,2-dicarbonyl **3b**, the reaction mixture was treated with aqueous 1 N HCl before usual workup. An aliquot was taken from organic layer for GC-MS analysis, which indicated no ^{18}O incorporation in the product **3b**.

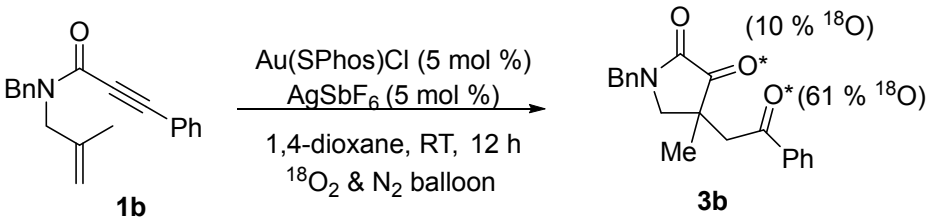
[mass spectrum of normal isotopomer **3b**]

3.2. Exchange with $^{18}O_2$

For $^{18}O_2$ incorporation experiment, a gas mixture of $^{18}O_2$ (90 atom %) and N_2 (ratio of partial pressure = 1:4) in a balloon was used. A mixture of Au(SPhos)Cl (3.2 mg, 0.005 mmol) and $AgSbF_6$ (1.7 mg, 0.005 mmol) was dissolved in anhydrous 1,4-dioxane and the mixture was stirred for 10 min at RT. Substrate **1b** (29.8 mg, 0.10 mmol) was placed in a separate test tube and capped with a septum. The tube was evacuated and then filled with N_2 gas. This evacuation and re-filling was repeated twice before a balloon of $^{18}O_2$ and N_2 mixture (1:4) was connected through the septum. To the test tube was added above solution of pre-catalyst in dioxane via a syringe. The reaction mixture was allowed to stir at rt for 12 h. An aliquot was taken directly from the crude reaction mixture for GC-MS analysis to reduce the oxygen exchange of 1,2-dicarbonyl group with trace amount of water.



[mass spectrum of **3b** from the reaction under $^{18}O_2$]



[Calculation]

For normal oxygen isotopomer **3b** (no ^{18}O), only m/z 321 and 322 was observed for molecular peaks (please see the mass spectrum in **3.1**).

Total relative peak heights of all **3b** species (m/z 321, 322, 323, 324, 325 and 326) from above mass spectrum = 83 + 10 + 100 + 23 + 22 + 3 = 241. Therefore,

3b (%): peak height (m/z 321 & 322)/241 x 100 = 38.6 %

^{18}O-**3b** (%): peak height (m/z 323 & 324)/241 x 100 = 51.0 %

$^{18}O_2$-**3b** (%): peak height (m/z 325 & 326)/241 x 100 = 10.4 %

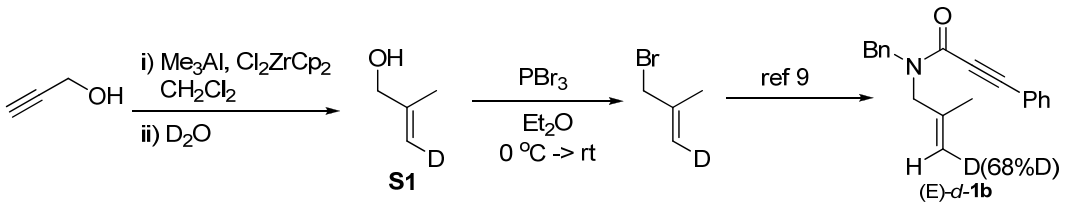
Assuming that all ^{18}O-**3b** resulted from oxygen exchange of $^{18}O_2$-**3b** with extraneous water (H_2O) via a hydrate, mono ^{18}O-**3b** should have ^{18}O atom in the phenyl ketone, not α-ketocarbonyl. Therefore,

^{18}O content at α-ketocarbonyl (%): 10.4 % (only from $^{18}O_2$-**3b**)

^{18}O content at phenylketone carbonyl (%): 10.4 % (from $^{18}O_2$-**3b**) + 51.0 % (from ^{18}O-**3b**) = 61.4 %

4. Preparation of *d*-labeled substrates

Preparation of **S1** for (*E*)-*d*-**1b**[7]

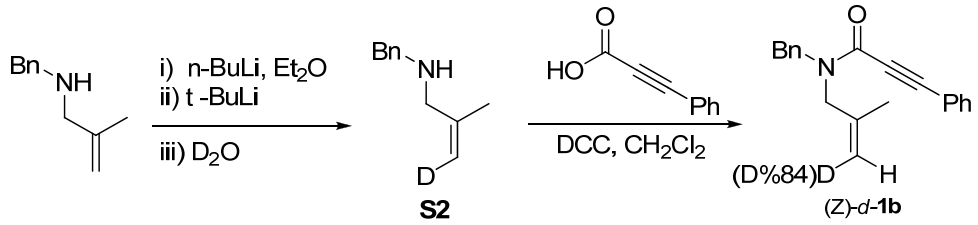


To a solution of Me_3A1 (pyrophoric!) (1.5 mL, 3.0 mmol) and Cp_2ZrCl_2 (292.3 mg, 1.0 mmol) in 1,2-dichloroethane (10 mL) was added dropwise 3-butyn-1-ol (58 μL, 1.0 mmol) in 1,2-dichloroethane (10 mL) at 0 °C. A rapid evolution of methane occurs. After the reaction mixture is stirred for 12 h at room temperature, it was treated with D_2O (1.0 mL) and then with saturated aqueous K_2CO_3 at 0 °C. The heterogeneous mixture was extracted with hexane and ether. The organic layers were combined, dried over $MgSO_4$, and concentrated *in vacuo*. Flash column chromatography (EtOAc:HEX=30:70) provided the corresponding allylic alcohol **S1** (24.9 mg, 34 %) as a colorless oil.

The allylic alcohol **S1** was brominated with PBr_3 and coupled to the amides as described before[8] to provide (*E*)-*d*-**1b**: 89 % yield (68 % D incorporation).

(*E*)-*d*-**1b:** ^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.58-7.46 (m, 2H, major/minor), 7.44-7.24 (m, 8H, major/minor), 5.01 (s, H(D), major), 4.96 (s, H(D), minor), 4.90 (s, H, major), 4.81 (s, 2H, minor), 4.80 (s, H, minor), 4.63 (s, 2H, major), 4.11 (s, 2H, major), 3.95 (s, 2H, minor), 1.78 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 155.3, 154.9, 139.9, 139.6, 136.5, 136.4, 132.6, 132.5, 130.24, 130.19, 128.9, 128.8, 128.7, 128.64, 128.58, 128.0, 127.8, 127.7, 120.6, 120.5, 114.1, 114.0, 113.8, 113.5, 113.2 , 90.7, 90.6, 81.8, 81.7, 53.9, 51.4, 48.7, 46.4, 20.1, 19.9.

Preparation of **S2** for (Z)-*d*-**1b**[9]



To a solution of corresponding amine (161.2 mg, 1.0 mmol) in diethyl ether (5 mL) was added n-BuLi (2.5M in hexane, 0.4 mL, 1.0 mmol) at -50 °C under Ar and the mixture was stirred for 20 min at -50 °C. To the resulting mixture was added *t*-BuLi (1.7M in pentane,

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[7] C. L. Rand, D. E. Van Horn, M. W. Moore, E. -i. Negishi, *J. Org. Chem.* **1981**, 46, 4093.

[8] Koo, J.; Park, H. –S.; Shin, S. *Tetrahedron Lett.* **2013**, *54*, 834.

[9] J. Baluenga, R. Gonzáles, F. J. Fañanás, *Tetrahedron Lett*, **1992**, 33, 7573

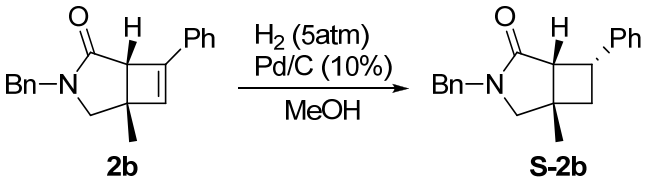
0.71 mL, 1.2 mmol) at -30 °C and stirred 2 h. The mixture was treated with D_2O (22 μL, 1.2 mmol) at -50 °C and stirred overnight. The mixture was quenched with saturated NH_4Cl and extracted with diethyl ether. The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (EtOAc:Hex = 20:80) to give corresponding amine **S2** (100.1 mg, 62 %).

The secondary amine was then coupled with phenylpropiolic acid by DCC,[3] to give (Z)-*d*-**1b**: 76 % yield (84 % D incorporation).

(Z)-*d*-**1b:** ^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.58-7.46 (m, 2H, major/minor), 7.44-7.24 (m, 8H, major/minor), 5.00 (s, H, major), 4.94 (s, H, minor), 4.90 (s, H(D), major), 4.81 (s, 2H, minor), 4.63 (s, 2H, major), 4.11 (s, 2H, major), 3.95 (s, 2H, minor), 1.78 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3, 154.9, 139.9, 139.6, 136.5, 136.4, 132.6, 132.5, 130.23, 130.20, 128.9, 128.8, 128.7, 128.65, 128.63, 128.56, 128.0, 127.8, 127.7, 120.6, 120.5, 114.1, 114.0, 113.8, 113.6, 113.4, 113.1, 112.9, 90.7, 90.6, 81.8, 81.7, 539, 51.4, 48.6, 46.4, 20.2, 20.1, 20.0, 19.9.

5. Structural Determination of 2b

A confirmation of the structure of **2b** was conducted by the following hydrogenation reaction.

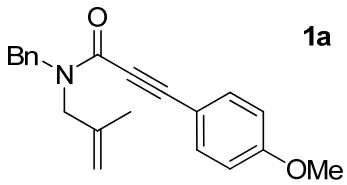


2b **S-2b**

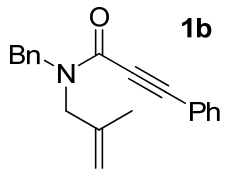
To a solution of **2b** (13.9 mg, 0.065 mmol) in MeOH (2 mL) was added Pd/C (6.9 mg of 10 % Pd/C). The mixture was hydrogenated at 5 atm of H_2 for 48 h at rt. The mixture was filtered off through a Celite and separated by silica gel column chromatography.

S-2b : ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.18 (m, 10H), 4.65 (d of ABq, *J* = 14.4 Hz, H), 4.13 (d of ABq, *J* = 14.5 Hz, H), 4.01 (q, *J* = 9.8 Hz, H), 3.15 (d of ABq, *J* = 9.8 Hz, H), 3.09-3.01 (m, 2H), 2.33 (t, *J* = 11.0 Hz, H), 2.25 (ddd, *J* = 2.8, 9.4, 12.1 Hz, H), 1.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.8, 139.6, 136.9, 128.7, 128.6, 128.1, 127.7, 127.6, 59.6, 51.9, 46.8, 37.9, 37.3, 33.7, 22.9.

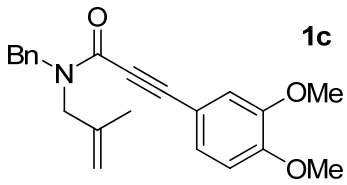
6. Characterization of Substrates



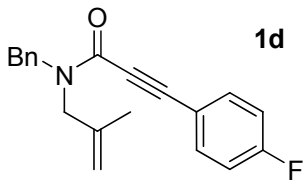
^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.1:1): δ 7.46 (app t, *J* = 9.0 Hz, 2H, major/minor), 7.44-7.22 (m, 5H, major/minor), 6.86 (app t, *J* = 9.0 Hz, 2H, major/minor), 5.01 (s, H, major), 4.95 (s, H, minor), 4.89 (s, H, major), 4.81 (s, 2H, minor), 4.79 (s, H, minor), 4.62 (s, 2H, major), 4.10 (s, 2H, major), 3.94 (s, 2H, minor), 3.83 (s, 3H, major), 3.82 (s, 3H, minor), 1.77 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 161.05, 161.03, 155.5, 155.1, 140.0, 139.7, 136.5, 136.4, 134.3, 134.2, 128.8, 128.6, 128.4, 127.8, 127.7, 127.5, 114.22, 114.19, 113.9, 113.2, 112.4, 112.3, 91.19, 91.14, 80.99, 80.85, 55.4, 53.8, 51.3, 48.5, 46.3, 20.0, 19.9.



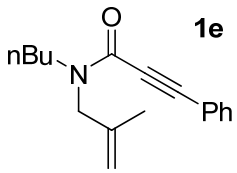
^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.58-7.46 (m, 2H, major/minor), 7.44-7.24 (m, 8H, major/minor), 5.01 (s, H, major), 4.96 (s, H, minor), 4.90 (s, H, major), 4.81 (s, 2H, minor), 4.80 (s, H, minor), 4.63 (s, 2H, major), 4.11 (s, 2H, major), 3.95 (s, 2H, minor), 1.78 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 155.2, 154.8, 139.9, 139.6, 136.4, 136.2, 132.5, 132.4, 130.13, 130.10, 128.8, 128.7, 128.55, 128.52, 128.4, 127.9, 127.7, 127.6, 120.5, 120.4, 114.0, 113.3, 90.6, 90.5, 81.7, 81.6, 53.8, 51.3, 48.6, 46.4, 20.1, 19.9.



^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.1:1): δ 7.42-7.24 (m, 5H, major/minor), 7.20-7.10 (m, H, major/minor), 7.06-6.96 (m, H, major/minor), 6.86-6.78 (m, H, major/minor) 5.02 (s, H, major), 4.95 (s, H, minor), 4.91 (s, H, major), 4.82 (s, 2H, minor), 4.80 (s, H, minor), 4.63 (s, 2H, major), 4.11 (s, 2H, major), 3.95 (s, 2H, minor), 3.93-3.80 (m, 6H, major/minor), 1.78 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 155.4, 155.0, 151.0, 150.9, 148.72, 148.68, 139.9, 139.6, 136.5, 136.4, 128.8, 128.6, 128.4, 127.8, 127.7, 127.5, 126.4, 126.3, 114.83, 114.79, 113.9, 113.2, 112.3, 112.2, 110.97, 110.94, 91.3, 91.2, 80.7, 80.6, 55.96, 55.93, 53.8, 51.3, 48.6, 46.3, 20.0, 19.9.

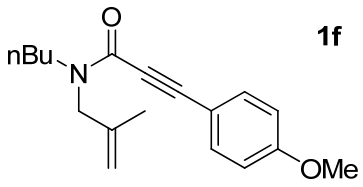


^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.58-7.44 (m, 2H, major/minor), 7.42-7.20 (m, 5H, major/minor), 7.04 (app q, *J* = 9.0 Hz, 2H, major/minor), 5.02 (s, H, major), 4.96 (s, H, minor), 4.90 (s, H, major), 4.80 (s, 3H, major/minor), 4.62 (s, 2H, major), 4.09 (s, 2H, major), 3.95 (s, 2H, minor), 1.77 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.5 (d, *J* = 254.3 Hz), 155.0, 154.7, 139.8, 139.5, 136.4, 136.2, 134.6 (d, *J* = 8.1 Hz), 134.5 (d, *J* = 8.8 Hz), 128.8, 128.7, 128.4, 127.9, 127.6, 116.6 (d, *J* = 3.7 Hz), 116.5 (d, *J* = 3.7 Hz), 116.1 (d, *J* = 22.8 Hz), 116.0 (d, *J* = 22.1 Hz), 114.0, 113.3, 89.6, 89.5, 81.6, 81.5, 81.4, 53.8, 51.3, 48.6, 46.4, 20.0, 19.9.



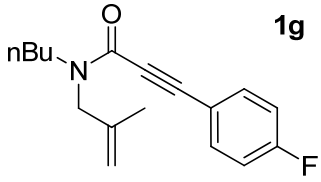
1e

^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1:1): δ 7.60-7.48 (m, 2H), 7.48-7.32 (m, 3H), 4.96 (s, H), 4.91 (s, 2H), 4.83 (s, H), 4.18 (s,2H), 4.04 (s, 2H), 3.57 (t, *J* = 7.4 Hz, 2H), 3.37 (t, *J* = 7.4 Hz, 2H), 1.77 (s, 3H), 1.72 (s, 2H), 1.70-1.48 (m, 2H), 1.46-1.28 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 154.8, 154.6, 140.4, 140.1, 132.33, 132.29, 129.97, 129.91, 128.53, 128.48, 120.66, 120.65, 113.4, 112.7, 89.8, 89.6, 81.9, 81.8, 55.0, 49.5, 47.9, 43.9, 30.5, 29.1, 20.17, 20.02, 19.99, 19.86, 13.8.



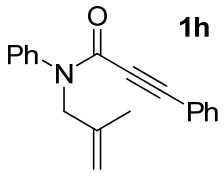
1f

^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1:1): δ 7.49 (d, *J* = 8.9 Hz, 2H), 7.46 (d, *J* = 9.0 Hz, 2H), 6.88 (d, *J* = 9.0 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 4.96 (s, H), 4.90 (s, 2H), 4.82 (s, H), 4.18 (s, 2H), 4.03 (s, 2H), 3.83 (s, 3H), 3.82 (s, 3H), 3.56 (t, *J* = 7.4 Hz, 2H), 3.37 (t, *J* = 7.5 Hz, 2H), 1.77 (s, 3H), 1.71 (s, 3H), 1.64 (quintet, *J* = 7.4 Hz, 2H), 1.54 (quintet, *J* = 7.4 Hz, 2H), 1.46-1.28 (m, 2H), 0.97 (t, *J* = 7.5 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.95, 160.90, 155.1, 154.9, 140.6, 140.2, 134.11, 134.06, 114.22, 114.16, 113.3, 112.61, 112.56, 112.54, 90.3, 90.2, 81.2, 81.1, 55.3, 55.0, 49.5, 47.9, 43.9, 30.5, 29.1, 20.2, 20.03, 19.96, 19.8, 13.8.



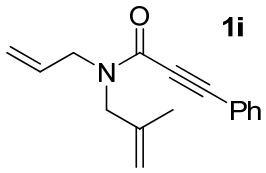
1g

^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1:1): δ 7.60-7.44 (m, 2H), 7.14-6.98 (m, 2H), 4.97 (s, H), 4.90 (s, 2H), 4.83 (s, H), 4.17 (s, 2H), 4.04 (s, 2H), 3.55 (t, *J* = 7.4 Hz, 2H), 3.37 (t, *J* = 7.4 Hz, 2H), 1.76 (s, 3H), 1.71 (s, 3H), 1.70-1.46 (m, 2H), 1.44-1.24 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.5 (d, *J* = 252.9 Hz), 163.4 (d, *J* = 252.8 Hz), 154.6, 154.5, 140.3, 140.0, 134.48 (d, *J* = 8.8 Hz), 134.44 (d, *J* = 8.8 Hz), 116.76 (d, *J* = 3.7 Hz), 116.73 (d, *J* = 3.7 Hz), 115.98 (d, *J* = 22.9 Hz), 115.96 (d, *J* = 22.8 Hz), 113.4, 112.7, 88.8, 88.6, 81.7, 81.6, 54.9, 49.5, 47.8, 43.9, 30.5, 29.1, 20.1, 20.01, 19.97, 19.86.



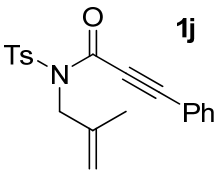
1h

^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 5.5:1): δ 7.57 (d, *J* = 7.1 Hz, 2H, minor), 7.50-7.18 (m, 8H, major/minor), 7.12 (d, *J* = 7.4 Hz, 2H, major), 4.95 (s, H, minor), 4.92 (s, H, minor), 4.88 (s, H, major), 4.80 (s, H, major), 4.59 (s, 2H, minor), 4.40 (s, 2H, major), 1.83 (s, 3H, minor), 1.70 (s, 3H, major); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 154.6, 142.1, 140.2, 132.7, 130.5, 130.2, 129.3, 129.2, 128.8, 128.6, 128.4, 128.3, 127.3, 126.4, 120.6, 113.9, 91.5, 82.8, 54.6, 20.6, 20.4.

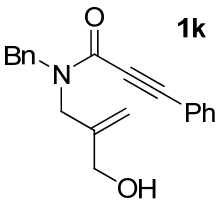


1i

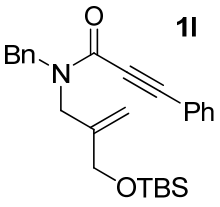
^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.60-7.48 (m, 2H, major/minor), 7.48-7.30 (m, 3H, major/minor), 5.92-5.69 (m, H, major/minor), 5.30-5.12 (m, 2H, major/minor), 4.98 (s, H, major), 4.93 (s, H, minor), 4.90 (s, H, major), 4.82 (s, H, minor), 4.20 (d, *J* = 5.9 Hz, 2H, major/minor), 4.17 (s, 2H, major/minor), 4.03 (s, 2H, major/minor), 1.77 (s, 3H, major), 1.72 (s, 3H, minor); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.71, 154.67, 140.0, 139.8, 132.6, 132.4, 132.3, 131.9, 130.06, 130.05, 128.5, 120.5, 118.2, 118.1, 113.6, 113.0, 90.06, 89.99, 81.6, 81.5, 54.0, 50.4, 49.0, 46.0, 20.0, 19.9.



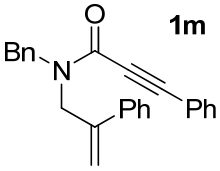
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.49 (d, *J* = 7.1 Hz, 2H), 7.44 (d, *J* = 7.4 Hz, H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 2H), 5.09 (s, H), 4.99 (s, 1H), 4.63 (s, 2H), 2.42 (s, 3H), 1.81(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.0, 145.2, 139.9, 135.8, 132.8, 131.0, 129.4, 129.0, 128.8, 119.5, 112.9, 92.7, 81.6, 52.5, 21.7, 20.3.



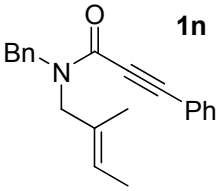
^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 2.2:1): δ 7.58-7.48 (m, 2H, major/minor), 7.46-7.26 (m, 8H, major/minor), 5.32 (s, H, minor), 5.18 (s, H, major), 5.10 (s, H, minor), 4.81 (s, 2H, major), 4.66 (s, 2H, minor), 4.25 (s, 2H, minor), 4.16 (s, 2H, minor), 4.04 (s, 4H, major); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6, 155.3, 143.2, 143.0, 136.3, 135.7, 132.53, 132.49, 130.4, 130.2, 129.0, 128.7, 128.6, 128.5, 128.4, 128.1, 127.8, 127.7, 120.3 120.0, 115.0, 113.6, 91.5, 90.8, 81.4, 81.2, 63.6, 63.5, 51.8, 50.2, 46.7, 45.7.



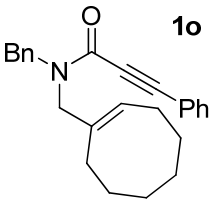
^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1:1): δ 7.56-7.48 (m, 2H), 7.44-7.26 (m, 8H), 5.30 (s, 1H), 5.29 (s, H), 5.03 (s, H), 4.95 (s, H), 4.84 (s, 2H), 4.65 (s, 2H, minor), 4.19 (s, 2H), 4.15 (s, 2H) 4.11 (s, 2H), 4.00 (s, 2H), 0.90 (s, 9H), 0.88 (s, 9H), 0.07 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.2, 154.8, 142.9, 142.6, 136.4, 136.2, 132.5, 132.4, 130.2, 130.1, 128.8, 128.7, 128.6, 128.52, 128.51, 128.4, 127.9, 127.7, 127.6, 120.5, 120.4, 112.4, 112.2, 90.6, 90.5, 81.6, 81.5.



^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.60-7.20 (m, 15H, major/minor), 5.54 (s, H, major), 5.52 (s, H, minor), 5.21 (s, H, minor), 5.12 (s, H, major), 4.75 (s, 2H, major), 4.65 (s, 2H, minor), 4.59 (s, 2H, minor), 4.48 (s, 2H, major); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5, 154.9, 143.1, 142.6, 138.9, 138.2, 136.5, 136.2, 132.6, 130.2, 129.0, 128.8, 128.7, 128.67, 128.64, 128.61, 128.5, 128.34, 128.29, 128.0, 127.8, 127.7, 126.5, 126.4, 120.6, 120.5, 115.7, 115.0, 90.8, 90.6, 81.8, 51.7, 51.3, 46.9, 46.3.

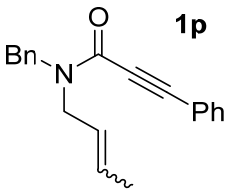


^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.2:1): δ 7.60-7.42 (m, 2H, major/minor), 7.42-7.20 (m, 8H, major/minor), 5.46-5.36 (m, H, major), 5.36-5.26 (m, H, minor), 4.76 (s, 2H, minor), 4.57 (s, 2H, major), 4.10 (s, 2H, major), 3.93 (s, 2H, minor), 1.70-1.56 (m, 6H, major/minor); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6, 155.59, 155.5, 137.2, 137.0, 136.8, 136.6, 133.05, 133.02, 132.9, 131.0, 130.8, 130.7, 130.6, 129.5, 129.34, 129.3, 129.2, 129.13, 129.1, 129.0, 128.6, 128.4, 128.3, 128.2, 128.1, 124.5, 123.7, 121.1, 121.0, 120.9, 91.4, 91.3, 91.0, 82.4, 82.3, 82.2, 56.3, 52.0, 51.5, 50.7, 46.9, 46.4, 14.4, 14.2, 14.0, 13.95.



^{1}H NMR (400 MHz, $CDCl_3$, rotameric mixture, 1.5:1): δ 7.60-7.20 (m, 10H), 5.53 (t, *J* = 8.2 Hz H, major), 5.44 (t, *J* = 8.2 Hz H, minor), 4.80 (s, 2H, minor), 4.62 (s, 2H, major), 4.09 (s, 2H, major), 3.94 (s, 2H, minor), 2.19-2.09 (m, 4H, major/minor), 1.54-1.49 (m,

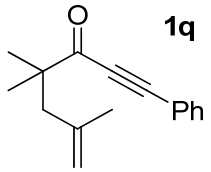
8H, major/minor); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.1, 154.9, 136.7, 136.5, 135.2, 134.9, 132.5, 132.3, 130.1, 130.0, 128.8, 128.6, 128.58, 128.53, 128.3, 127.9, 127.8, 127.7, 127.5, 120.6, 120.5, 90.4 , 90.3 , 81.9 , 81.8 , 53.4 , 51.2, 48.2 , 46.3, 29.7, 29.6, 28.58, 28.55, 26.77, 26.57, 26.50, 26.44, 26.33, 26.25, 26.22.



1p

(E/Z = 3:1)

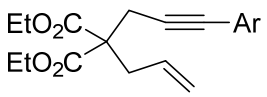
^{1}H NMR (400 MHz, $CDCl_3$ rotameric mixture, 1:1): δ 7.61-7.20 (m, 10H, major/minor), 5.77-5.52 (m, H, major/minor), 5.52-5.33 (m, H, major/minor) 4.82 (s, 2H, minor), 4.64 (s, 2H, major), 4.21 (d, *J* = 6.6 Hz, H, cis), 4.10 (d, *J* = 5.8 Hz, H, trans), 4.00 (d, *J* = 7.0 Hz, H, cis), 3.91 (d, *J* = 5.8 Hz, H, trans) 1.71, (dd *J* = 3.5, 12.9 Hz, 3H, trans), 1.58, (dd *J* = 6.3, 18.3 Hz, 3H, cis) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.5, 154.3, 136.5, 136.3, 132.3, 132.2, 130.1, 129.97, 129.91,128.78, 128.70, 128.6, 128.5, 128.45, 128.41, 128.21, 127.79, 127.74, 127.50, 127.4, 125.2, 124.9, 124.5. 124.1, 120.4, 120.3, 90.4, 90.2, 90.1, 90.0, 81.8, 81.7, 81.6, 51.7, 51.4, 49.9, 46.7, 46.5, 45.3, 44.6, 39.8, 17.6.



1q

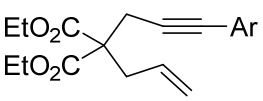
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.1 Hz, 2H), δ 7.45 (t, *J* = 7.4 Hz, H) δ 7.38 (t, *J* = 7.8 Hz, 2H), 4.80 (s, H), 4.72 (s, H,), 2.49 (s, 2H,), 1.71 (s, 3H), 1.25 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.1, 142.2, 133.1, 130.7, 128.7, 120.3, 114.9, 92.5, 86.4, 48.5, 47.8, 24.6, 24.1.

1r is a known compound and our material matched the ^{1}H and ^{13}C spectra in the literature (D. A. Candito, M. Lautens, *Synlett*, **2011**, 1987-1992).



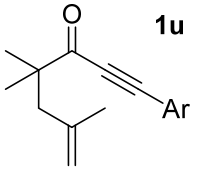
1s (Ar = 3,5-di-Me-C_6H_3)

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.99 (s, 2H), δ 6.91 (s, H), 5.73-5.63 (m, H), 5.20 (d, *J* = 17.2 Hz, H), 5.13 (d, *J* = 10.2 Hz, H), 4.20 (q, *J* = 7.0 Hz, 4H), 2.99 (s, 2H), 2.86 (d, *J* = 7.5 Hz, 2H), 2.26 (s, 6H), 1.26 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.9, 137.7, 132.0, 129.8, 129.3, 122.8, 119.7, 83.7, 83.4, 61.5, 57.0, 36.5, 23.4, 21.1, 20.9, 14.1.



1t (Ar = 4-F-C_6H_4)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (dt, *J* = 1.9, 3.5 Hz, 2H), δ 6.96 (t, *J* = 8.6 Hz, 2H), 5.74-5.63 (m, H), 5.20 (d, *J* = 16.8 Hz, H), 5.15 (d, *J* = 9.8 Hz, H), 4.22 (q, *J* = 7.0 Hz, 4H), 2.99 (s, 2H,), 2.85 (d, *J* = 7.4 Hz, 2H), 1.26 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.7, 163.6 (d, *J* = 247.8 Hz), 133.5 (d, *J* = 8.8 Hz), 131.9, 119.8, 119.4 (d, *J* = 3.3 Hz), 115.6 (d, *J* = 22.0 Hz), 84.2, 82.5, 61.7, 57.0, 36.7, 23.5, 14.1.

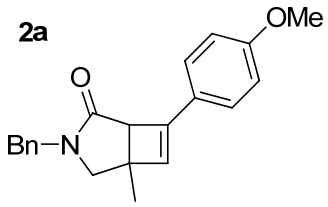


1u

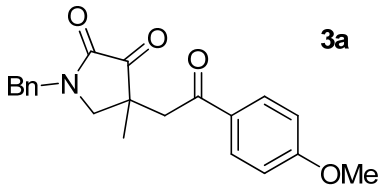
(Ar = 4-MeO-C_6H_4)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 9.0 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 4.83 (s, H), 4.70 (s, 1H,), 3.84 (s, 3H,), 2.48 (s, 2H), 1.70 (s, 3H) 1.25 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.0, 161.5, 142.2, 134.9, 114.6, 114.3, 112.0, 93.5, 86.1, 55.4, 48.1, 47.7, 24.5, 24.0.

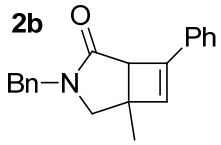
7. Characterization of Products



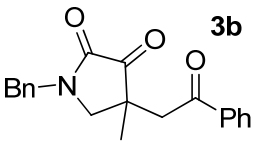
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.5 Hz, 2H), 7.38-7.22 (m, 3H), 7.17 (d, *J* = 7.1 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 6.28 (s, H), 4.45 (d of ABq, *J* = 14.8 Hz, H), 4.38 (d of ABq, *J* = 14.9 Hz, H), 3.81 (s, 3H), 3.47 (s, H), 3.23 (d of ABq, *J* = 9.8 Hz, H) 3.07 (d of ABq, 10.6 Hz, H), 1.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 173.7, 159.8, 147.3, 136.5, 130.8, 128.6, 128.0, 127.4, 127.0, 125.9, 113.9, 55.3, 55.0, 54.4, 46.8, 40.2, 22.1; HRMS(EI+) Calcd for $C_{21}H_{21}O_2N$ $[M]^+$ 319.1567, found 319.1568.



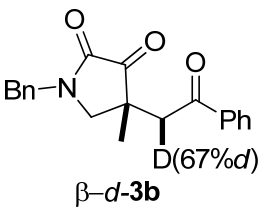
IR (neat): 1763, 1710, 1674 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 9.0 Hz, 2H), 7.42-7.20 (m, 5H), 6.90 (d, *J* = 8.9 Hz, 2H), 4.81 (d of ABq, *J* = 14.5 Hz, H), 4.68 (d of ABq, *J* = 14.4 Hz, H), 3.86 (s, 3H), 3.57 (d of ABq, *J* = 18.4 Hz, H), 3.44 (d of ABq, *J* = 18.4 Hz, H), 3.39 (d of ABq, *J* = 10.5 Hz, H), 3.17 (d of ABq, *J* = 10.5 Hz, H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 202.3, 194.8, 164.1, 159.7, 134.8, 130.5, 128.9, 128.36 128.33, 128.1, 113.9, 55.5, 53.6, 48.3, 47.4, 40.0, 23.5; HRMS(FAB+) Calcd for $C_{21}H_{22}O_4N$ $[M+1]^+$ 352.1543, found 352.1544; white solid; mp 178-182 °C.



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 7.1 Hz, 2H), 7.37 (t, *J* = 7.0 Hz, 2H), 7.34-7.22 (m, 4H), 7.17 (d, *J* = 6.6 Hz, 2H), 6.44 (s, H), 4.45 (d of ABq, *J* = 14.8 Hz, H), 4.39 (d of ABq, *J* = 14.5 Hz, H), 3.51 (s, H), 3.25 (dd, *J* = 1.6, 10.5 Hz, H) 3.08 (d, 10.2 Hz, H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 173.7, 148.0, 136.6, 133.6, 133.0, 128.8, 128.6, 128.2, 127.6, 125.6, 55.2, 54.3, 47.0, 40.6, 22.2; HRMS(FAB+) Calcd for $C_{20}H_{20}ON$ $[M+1]^+$ 290.1539, found 290.1543.



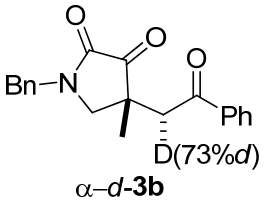
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.8 Hz, H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.40-7.28 (m, 5H), 4.80 (d of ABq, *J* = 14.4 Hz, H), 4.72 (d of ABq, *J* = 14.4 Hz, H), 3.62 (d of ABq, *J* = 18.4 Hz, H), 3.48 (d of ABq, *J* = 18.4 Hz, H), 3.40 (d of ABq, *J* = 10.5 Hz, H), 3.19 (d of ABq, *J* = 10.5 Hz, H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.4, 196.5, 159.6, 135.2, 134.7, 133.9, 128.9, 128.8, 128.3, 128.2, 128.1, 53.5, 48.2, 47.6, 40.0, 23.5; HRMS(FAB+) Calcd for $C_{20}H_{20}O_3N$ $[M+1]^+$ 322.1438, found 322.1438; white solid; mp 148-152 °C.



β–*d*-**3b**

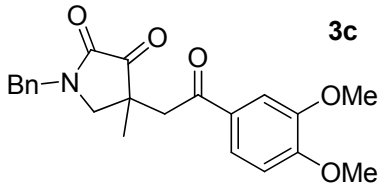
(Obtained from (*E*)-**1b**) ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.40-7.28 (m, 5H), 4.80 (d of ABq, *J* = 14.8 Hz, H), 4.72 (d of ABq, *J* = 14.8 Hz, H), 3.62 (d of ABq, *J* = 18.4 Hz, 0.32H of **3b**), 3.48 (d of ABq, *J* = 18.4 Hz, 0.32H of **3b**), 3.47 (s, 0.68H of *d*-**3b**), 3.39 (d of ABq, *J* = 10.5 Hz, H), 3.19 (d of ABq, *J* = 10.5 Hz, H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.4, 196.6, 159.7, 135.4, 134.8, 134.1, 129.0, 128.9, 128.5, 128.3, 128.2, 53.6, 48.4, 47.5(**3b**), 47.3

(t, J_{CD} = 19.1 Hz, *d*-**3b**), 40.0, 23.6.



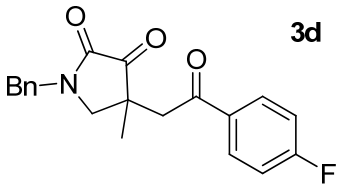
α–*d*-**3b**

(Obtained from (*Z*)-**1b**) ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.40-7.28 (m, 5H), 4.80 (d of ABq, *J* = 14.5 Hz, H), 4.72 (d of ABq, *J* = 14.5 Hz, H), 3.62 (d of ABq, *J* = 18.3 Hz, 0.26H of **3b**), 3.59 (s, 0.69H of *d*-**3b**), 3.48 (d of ABq, *J* = 18.4 Hz, 0.26H of **3b**), 3.39 (d of ABq, *J* = 10.5 Hz, H), 3.19 (d of ABq, *J* = 10.5 Hz, H), 1.23 (s, 3H).



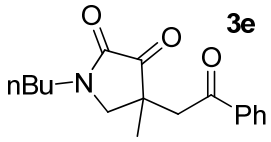
3c

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54-7.46 (m, H), 7.44-7.28 (m, 6H), 6.85 (d, *J* = 8.6 Hz, H), 4.86 (d of ABq, *J* = 14.4 Hz, H), 4.65 (d of ABq, *J* = 14.8 Hz, H), 3.95 (s, 3H), 3.91 (s, 3H), 3.58 (d of ABq, *J* = 18.3 Hz, H), 3.46 (d of ABq, *J* = 18.4 Hz, H), 3.40 (d of ABq, *J* = 10.5 Hz, H), 3.19 (d of ABq, *J* = 10.5 Hz, H), 1.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.3, 195.1, 159.6, 153.9, 149.1, 134.8, 128.9, 128.5, 128.3, 128.1, 123.1, 110.02, 110.00, 56.1, 56.0, 53.6, 48.3, 47.3, 40.0, 23.5; HRMS(FAB+) Calcd for $C_{22}H_{24}O_5N$ $[M+1]^+$ 382.1649, found 382.1654; sticky oil.



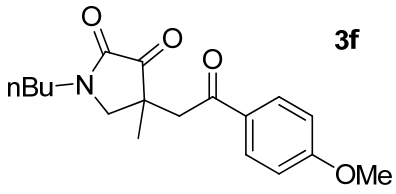
3d

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 5.5, 8.6 Hz, 2H), 7.44-7.22 (m, 5H), 7.12 (t, *J* = 8.6 Hz, 2H), 4.79 (d of ABq, *J* = 14.4 Hz, H), 4.73 (d of ABq, *J* = 14.8 Hz, H), 3.58 (d of ABq, *J* = 18.3 Hz, H), 3.44 (d of ABq, *J* = 18.8 Hz, H), 3.39 (d of ABq, *J* = 10.9 Hz, H), 3.19 (d of ABq, *J* = 10.5 Hz, H), 1.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.2, 194.8, 166.2 (d, *J* = 255.8 Hz), 159.5, 134.7, 131.7, (d, *J* = 2.9 Hz), 130.9 (d, *J* = 9.5 Hz), 128.9, 128.3, 128.1, 115.9 (d, *J* = 22.1 Hz), 54.5, 48.3, 47.3, 40.0, 23.6; HRMS(FAB+) Calcd for $C_{20}H_{19}O_3NF$ $[M+1]^+$ 340.1343, found 340.1342; white solid; mp 186-190 °C.



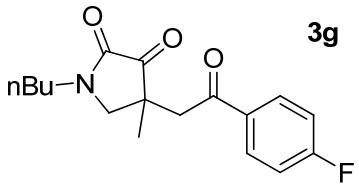
3e

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.4 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, H), 7.46 (t, *J* = 7.8 Hz, 2H), 3.37-3.47 (m, 5H), 3.35 (d of ABq, *J* = 10.5 Hz, H), 1.74-1.54 (m, 2H), 1.40 (sextet, *J* = 7.4 Hz, 2H) 1.31 (s, 3H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 202.5, 196.5, 159.5, 135.4, 133.9, 128.8, 128.2, 54.1, 47.1, 44.2, 40.1, 28.8, 23.7, 19.9, 13.7; HRMS(FAB+) Calcd for $C_{17}H_{22}O_3N$ $[M+1]^+$ 288.1594, found 288.1594; white solid; mp 123-125 °C.

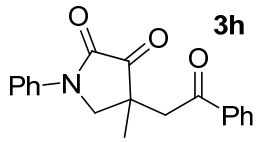


3f

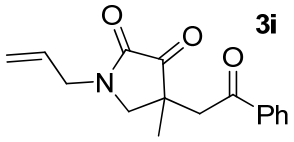
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.6 Hz, 2H), 6.94 (d, *J* = 8.6 Hz, 2H), 3.87 (s, 3H), 3.62-3.47 (m, 5H), 3.33 (d, *J* = 10.5Hz, H), 1.74-1.54 (m, 2H), 1.48-1.34 (m, 2H), 1.30 (s, 3H), 0.96 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 202.6, 194.9, 164.1, 159.6, 130.5, 128.5, 113.9, 55.5, 54.2, 46.9, 44.2, 40.1, 28.8, 23.7, 19.9, 13.7; HRMS(FAB+) Calcd for $C_{18}H_{24}O_4N$ $[M+1]^+$ 318.1700, found 318.1699; white solid; mp 158-162 °C.



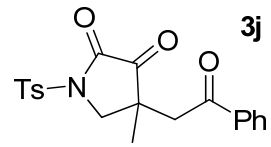
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (dd, *J* = 5.1, 8.6 Hz, 2H), 7.13 (t, *J* = 8.6 Hz, 2H), 3.66-3.46 (m, 5H) 3.35 (d of ABq, *J* = 10.6 Hz, H), 1.74-1.54 (m, 2H), 1.41 (sextet, *J* = 7.0 Hz, 2H), 1.31 (s, 3H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 202.5, 194.9, 166.2 (d, *J* = 255.8 Hz), 159.4, 131.8 (d, *J* = 3.0 Hz), 130.9 (d, *J* = 9.6 Hz), 115.9 (d, *J* = 22.1 Hz), 54.1, 47.0, 44.2, 40.1, 28.8, 23.7, 19.9, 13.7; HRMS(FAB+) Calcd for $C_{17}H_{21}O_3NF$ $[M+1]^+$ 306.1500, found 306.1501; white solid; mp 164-165 °C.



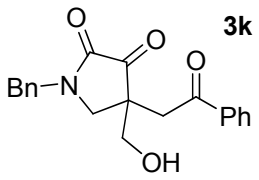
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 7.8 Hz, 2H), 7.86 (d, *J* = 7.8 Hz, 2H), 7.60 (t, *J* = 7.5 Hz, H), 7.47 (t, *J* = 7.9 Hz, 2H), 7.46 (t, *J* = 8.2 Hz, 2H), 7.28 (t, *J* = 7.4 Hz, H), 4.03 (d of ABq, *J* = 10.1 Hz, H), 3.83 (d of ABq, *J* = 10.1 Hz, H), 3.74 (d of ABq, *J* = 18.7 Hz, H), 3.67 (d of ABq, *J* = 18.8 Hz, H), 1.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.6, 196.5, 158.1, 138.9, 135.2, 134.1, 129.2, 128.8, 128.3, 119.5, 126.5, 55.0, 47.7, 39.8, 23.8; HRMS(FAB+) Calcd for $C_{19}H_{18}O_3N$ $[M+1]^+$ 308.1281, found 308.1283; white solid; mp 162-164 °C.



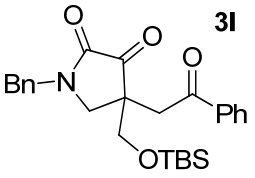
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.4 Hz, 2H), 7.60 (t, *J* = 7.4Hz, H), 7.46 (t, *J* = 7.4 Hz, 2H), 5.81 (ddd, *J* = 5.8, 10.6, 17.2 Hz, H), 5.33 (d, *J* = 17.5 Hz, H), 5.29 (d, *J* = 10.6 Hz, H), 4.19 (d, *J* = 6.2 Hz, 2H), 3.65 (d of ABq, *J* = 18.8 Hz, H), 3.55 (d of ABq, *J* = 18.7 Hz, H), 3.53 (d of ABq, *J* = 10.5 Hz, H), 3.32 (d of ABq, *J* = 10.5 Hz, H), 1.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 202.4, 196.7, 159.4, 135.4, 134.1, 130.9, 128.9, 128.3, 119.6, 53.9, 47.6, 47.1, 40.2, 23.8; HRMS(FAB+) Calcd for $C_{16}H_{18}O_3N$ $[M+1]^+$ 272.1281, found 272.1281; sticky oil.



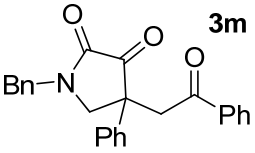
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.77 (d, *J* = 7.0 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, H), 7.44 (t, *J* = 7.8 Hz, 2H), 7.37 (d, *J* = 8.2 Hz, 2H), 3.92 (d of ABq, *J* = 10.6 Hz, H), 3.72 (d of ABq, *J* = 10.2 Hz, H), 3.68 (d of ABq, *J* = 18.7 Hz, H), 3.60 (d of ABq, *J* = 18.7 Hz, H), 2.47 (s, 3H), 1.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 196.5, 156.7, 146.1, 134.7, 134.4, 133.9, 130.0, 128.9, 128.6, 128.4, 53.5, 49.8, 40.7, 23.2, 21.9; HRMS(FAB+) Calcd for $C_{20}H_{20}NO_5S$ $[M+1]^+$ 386.1057, found 386.1062; sticky oil.



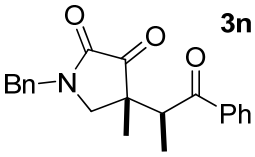
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.4 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, H), 7.42 (t, *J* = 7.4 Hz, 2H), 7.38-7.28 (m, 5H), 4.82 (d of ABq, *J* = 14.8 Hz, H), 4.69 (d of ABq, *J* = 14.9 Hz, H), 3.88-3.62 (m, 3H), 3.52 (d of ABq, *J* = 19.1 Hz, 1H), 3.47 (d of ABq, *J* = 11.3 Hz, H), 3.28 (d of ABq, *J* = 10.9 Hz, H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.2, 196.7, 160.4, 135.3, 134.7, 134.1, 129.0, 128.9, 128.6, 128.4, 128.2, 66.0, 49.8, 48.5, 46.1, 44.4; HRMS(FAB+) Calcd for $C_{20}H_{20}O_4N$ $[M+1]^+$ 338.1387, found 338.1392; colorless oil.



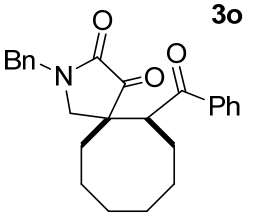
3l ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.40-7.26 (m, 5H), 4.79 (d of ABq, *J* = 14.8 Hz, H), 4.74 (d of ABq, *J* = 14.8 Hz, H), 3.73 (d of ABq, *J* = 9.0 Hz, H), 3.60 (d of ABq, *J* = 9.0 Hz, 1H), 3.58 (d of ABq, *J* = 9.3 Hz, H), 3.51 (s, 2H), 3.28 (d of ABq, *J* = 10.6 Hz, H), 0.81 (s, 9H), 0.00 (s,3H), -0.04 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.2, 196.2, 160.1, 135.4, 134.9, 134.1, 128.95, 128.90, 128.6, 128.3, 128.1, 67.4, 49.8, 48.5, 46.1, 44.0, 25.8, 18.3, -5.4; HRMS(FAB+) Calcd for $C_{26}H_{34}O_4NSi$ $[M+1]^+$ 452.2252, found 452.2257; colorless oil.



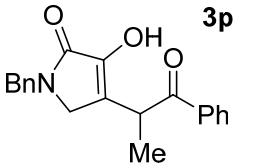
3m ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.4 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.40-7.24 (m, 8H), 7.24-7.18 (m, 2H), 4.82 (d of ABq, *J* = 14.5 Hz, H), 4.77 (d of ABq, *J* = 14.5 Hz, H), 3.97 (d of ABq, *J* = 18.3 Hz, H), 3.93 (d of ABq, *J* = 10.9 Hz, H), 3.90 (d of ABq, *J* = 18.7 Hz, H), 3.71 (d of ABq, *J* = 10.9 Hz, H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 196.4, 159.7, 138.1, 135.5, 134.6, 134.1, 129.4, 129.0, 128.9, 128.7, 128.3, 128.2, 126.2, 53.6, 48.6, 48.3, 48.1; HRMS(EI+) Calcd for $C_{25}H_{21}O_3N$ $[M+1]^+$ 383.1516, found 383.1518; sticky oil.



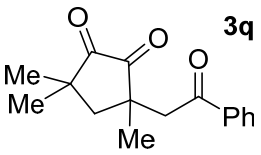
3n ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.4 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.40-7.24 (m, 5H), 4.73 (d of ABq, *J* = 14.9 Hz, H), 4.69 (d of ABq, *J* = 14.9 Hz, H), 3.93 (q, *J* = 7.4 Hz, 1H), 3.64 (d of ABq, *J* = 11.7 Hz, H), 3.31 (d of ABq, *J* = 11.7 Hz, H), 1.23 (d, *J* = 7.4 Hz, 3H), 1.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.7, 201.6, 159.4, 135.0, 134.6, 133.9, 128.9, 128.6, 128.4, 128.3, 128.2, 52.5, 48.8, 48.4, 43.8, 22.7, 14.0; HRMS(FAB+) Calcd for $C_{21}H_{22}O_3N$ $[M+1]^+$ 336.1594, found 336.1596; sticky oil.



3o ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.28-7.21 (m, 5H), 4.73 (d of ABq, *J* = 14.9 Hz, H), 4.59 (d of ABq, *J* = 14.9 Hz, H), 3.90 (d, *J* = 5.4 Hz, H), 3.25 (d of ABq, *J* = 11.0 Hz, H), 2.81 (d of ABq, *J* = 11.0 Hz, H), 2.65-2.57 (m, H), 2.00 (t, *J* = 13.1 Hz, H), 1.86-1,67 (m, 8H), 1.60-1.52 (m, 2H), 1.47-1.43 (m, H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.1, 200.9, 160.6, 135.0, 134.8, 133.8, 128.97, 128.93, 128.90, 128.2, 55.3, 48.1, 44.9, 31.2, 28.1, 27.6, 25.7, 24.9, 23.2; HRMS(FAB+) Calcd for $C_{25}H_{28}O_3N$ $[M+1]^+$ 390.2064, found 390.2069; sticky oil.

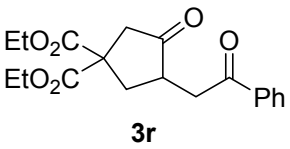


3p ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (d, *J* = 7.8 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.35-7.27 (m, 3H), 7.17 (d, *J* = 6.6 Hz, 2H), 4.88 (q, *J* = 7.0 Hz, H) 4.69 (d of ABq, *J* = 14.9 Hz, H), 4.46 (d of ABq, *J* = 14.9 Hz, H), 3.72 (d of ABq, *J* = 18.4 Hz, H), 3.55 (d of ABq, *J* = 18.4 Hz, H) 1.35 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.9, 167.3, 142.0, 136.4, 135.7, 133.6, 128.8, 128.7, 128.6, 128.0, 127.7, 119.9, 47.08, 37.9, 30.1, 16.2; HRMS(FAB+) Calcd for $C_{20}H_{20}NO_3$ $[M+1]^+$ 322.1438, found 322.1443; thick oil.



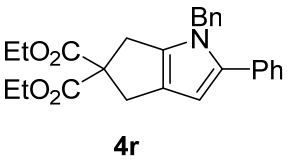
3q

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, H), 7.44 (d, *J* = 7.8 Hz, 2H), 3.75 (d of ABq, *J* = 18.7 Hz, H), 3.64 (d of ABq, *J* = 18.4 Hz, H), 2.00 (d, *J* = 13.3 Hz, H), 1.91 (d, *J* = 13.3 Hz, H), 1.32 (s, 3H), 1.29 (s, 3H), 1.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.5, 206.5, 198.0, 134.9, 134.0, 128.7, 128.4, 54.8, 46.0, 41.2, 40.4, 27.1, 25.24, 25.23; HRMS(FAB+) Calcd for $C_{16}H_{19}O_3$ $[M+1]^+$ 259.1329, found 259.1334; yellow solid, mp 135-137 °C.



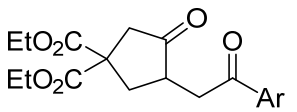
3r

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 7.4 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, H), 7.46 (t, *J* = 7.4 Hz, 2H), 4.28-4.19 (m, 4H), 3.51 (dd, *J* = 2.8, 18.3 Hz, H), 3.24 (dd, *J* = 6.6, 18.7 Hz, H), 3.05 (d of ABq, *J* = 18.7 Hz, H), 2.98 (d of ABq, *J* = 18.7 Hz, H), 2.92-2.84 (m, 2H), 2.24 (t, *J* = 15.6 Hz, H), 1.27 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 215.1, 197.1, 171.3, 170.7, 136.3, 133.5, 128.7, 128.1, 62.2, 62.1, 55.2, 44.7, 43.3, 38.6, 35.7, 14.1. HRMS (EI+) Calcd for $C_{19}H_{22}O_6$ $[M]^+$ 346.1411, found 346.1414; colorless oil.



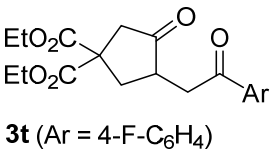
4r

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.21 (m, 8H), 6.99 (d, *J* = 7.0 Hz, 2H), 6.04 (s, H), 5.04 (s, 2H), 4.17 (q, *J* = 7.0 Hz, 4H), 3.37 (s, 2H), 3.20 (s, 2H), 1.22 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 138.8, 138.4, 135.6, 133.6, 128.8, 128.6, 128.5, 127.3, 126.8, 126.3, 122.6, 103.8, 64.8, 61.7, 49.2, 34.7, 33.8, 14.1. HRMS (EI+) Calcd for $C_{26}H_{27}NO_4$ $[M]^+$ 417.1935, found 417.1939; sticky oil.



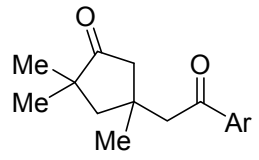
3s (Ar = 3,5-di-Me-C_6H_3)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (s, 2H), 7.20 (s, H), 6.84 (d, *J* = 20.7 Hz, H), 4.27- 4.18 (m, 4H), 3.48 (dd, *J* = 2.7, 18.3 Hz, H), 3.19 (dd, *J* = 6.6, 18.3 Hz, H), 3.05 (d of ABq, *J* = 18.7 Hz, H), 2.97 (d of ABq, *J* = 18.7 Hz, H), 2.92-2.82 (m, 2H), 2.68 (d, *J* = 16.0 Hz, H), 2.35 (s, 3H), 2.27 (s, 3H), 1.36 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 215.1, 204.4, 197.6, 171.3, 170.7, 170.3, 170.1, 139.2, 138.4, 137.7, 136.5, 135.1, 129.2, 128.0, 125.9, 62.2, 62.15, 62.10, 57.8, 55.3, 44.7, 43.4, 41.6, 38.7, 38.3, 35.8, 30.1, 25.7, 24.9, 21.39, 21.33, 14.1, 14.0. HRMS (EI+) Calcd for $C_{21}H_{26}O_6$ $[M]^+$ 374.1724, found 374.1728; colorless oil.



3t (Ar = 4-F-C_6H_4)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98-7.94 (m, 2H), 7.13 (t, *J* = 8.6 Hz, 2H), 4.28-4.21 (m, 4H), 3.46 (dd, *J* = 3.2, 18.3 Hz, H), 3.20 (dd, *J* = 7.0, 18.7 Hz, H), 3.05 (d of ABq, *J* = 18.7 Hz, H), 2.97 (d of ABq, *J* = 18.7 Hz, H), 2.92-2.84 (m, 2H), 2.23 (t, *J* = 15.6 Hz, H), 1.27 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 214.9, 195.6, 171.2, 170.7, 166.0 (d, *J* = 260 Hz), 132.9 (d, *J* = 8.8 Hz), 130.8 (d, *J* = 3.7 Hz), 116.0 (d, *J* = 22.0 Hz), 62.2, 62.1, 55.3, 44.6, 43.3, 38.5, 35.7, 29.8, 14.1; HRMS (EI+) Calcd for $C_{19}H_{21}FO_6$ $[M]^+$ 364.1317, found 364.1321; colorless oil.



5u (Ar = 4-MeO-C_6H_4)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 9.0 Hz, 2H), 6.91 (d, *J* = 8.2 Hz, 2H), 3.87 (s, 3H), 3.05 (s, 2H), 2.60 (d of ABq, *J* = 17.5 Hz, H), 2.33 (d of ABq, *J* = 17.5 Hz, H), 2.06 (d of ABq, *J* = 13.3 Hz, H), 1.93 (d of ABq, *J* = 13.7 Hz, H), 1.22 (s, 3H), 1.19 (s, 3H), 1.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 223.3, 197.4, 163.6, 131.0, 130.4, 113.8, 55.6, 51.2, 51.0, 49.2, 45.0, 35.4, 28.6, 27.8, 27.3. HRMS (EI+) Calcd for $C_{17}H_{22}O_3$ $[M]^+$ 274.1563, found 274.1568; colorless oil.

8. X-ray crystallographic analysis data for compound 3e

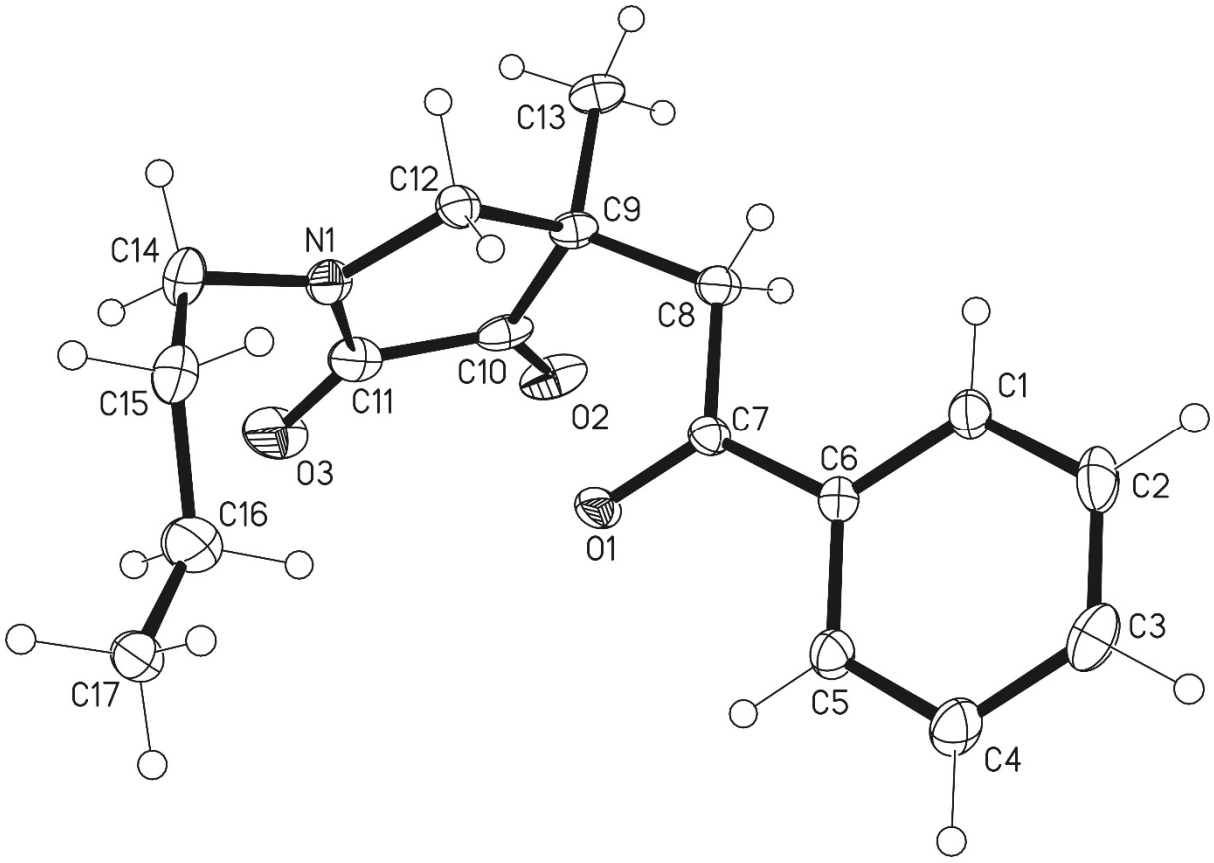


Table S2. Crystal data and structure refinement for **3e**

Identification code	**3e**	
Empirical formula	C17 H21 N O3	
Formula weight	287.35	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Hexagonal	
Space group	P6(5)	
Unit cell dimensions	a = 11.2648(8) Å	α= 90°.
	b = 11.2648(8) Å	β= 90°.
	c = 21.2304(15) Å	γ = 120°.
Volume	2333.1(3) $Å^3$	
Z	6	
Density (calculated)	1.227 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	924	
Crystal size	0.32 x 0.09 x 0.08 mm^3	
Theta range for data collection	2.09 to 27.58°.	
Index ranges	-14<=h<=7, -13<=k<=13, -27<=l<=27	
Reflections collected	14031	
Independent reflections	3606 [R(int) = 0.0604]	
Completeness to theta = 27.58°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9933 and 0.9737	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3606 / 1 / 199	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.0563, wR2 = 0.1160	
R indices (all data)	R1 = 0.1063, wR2 = 0.1336	
Absolute structure parameter	-1.6(17)	
Largest diff. peak and hole	0.159 and -0.203 $e.Å^{-3}$	

Table S3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3e**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	9040(2)	7767(2)	1488(1)	48(1)
O(2)	8741(3)	5159(2)	2171(1)	66(1)
O(3)	6414(2)	4074(2)	1317(2)	83(1)
N(1)	8133(2)	5151(2)	586(1)	46(1)
C(1)	12487(3)	10650(3)	1556(1)	43(1)
C(2)	13153(3)	12061(3)	1598(1)	56(1)
C(3)	12434(4)	12722(3)	1668(1)	58(1)
C(4)	11017(3)	11986(3)	1703(2)	53(1)
C(5)	10347(3)	10586(2)	1660(1)	42(1)
C(6)	11066(2)	9893(2)	1582(1)	32(1)
C(7)	10290(3)	8376(2)	1517(1)	34(1)
C(8)	11029(3)	7581(3)	1476(1)	40(1)
C(9)	10130(3)	6126(3)	1236(1)	37(1)
C(10)	8828(3)	5358(3)	1609(1)	46(1)
C(11)	7621(3)	4774(3)	1161(2)	55(1)
C(12)	9609(2)	6082(3)	562(1)	40(1)
C(13)	10918(3)	5339(3)	1277(2)	53(1)
C(14)	7317(3)	4811(3)	13(2)	66(1)
C(15)	7165(4)	5993(4)	-240(2)	71(1)
C(16)	6353(5)	6391(5)	181(2)	94(1)
C(17)	5866(7)	7223(7)	-125(3)	50(2)
C(17')	5131(13)	5940(20)	139(7)	199(9)

Table S4. Bond lengths [Å] and angles [°] for **3e**.

O(1)-C(7)	1.221(3)
O(2)-C(10)	1.209(3)
O(3)-C(11)	1.228(3)
N(1)-C(11)	1.326(4)
N(1)-C(14)	1.456(4)
N(1)-C(12)	1.457(3)
C(1)-C(2)	1.380(4)
C(1)-C(6)	1.388(3)
C(2)-C(3)	1.355(5)
C(3)-C(4)	1.384(5)
C(4)-C(5)	1.370(4)
C(5)-C(6)	1.388(4)
C(6)-C(7)	1.486(3)
C(7)-C(8)	1.499(3)
C(8)-C(9)	1.520(4)
C(9)-C(10)	1.502(4)
C(9)-C(12)	1.539(4)
C(9)-C(13)	1.539(4)
C(10)-C(11)	1.514(5)
C(14)-C(15)	1.522(5)
C(15)-C(16)	1.498(5)
C(16)-C(17')	1.210(12)
C(16)-C(17)	1.455(7)

C(11)-N(1)-C(14)	124.6(3)
C(11)-N(1)-C(12)	114.8(2)
C(14)-N(1)-C(12)	120.2(2)
C(2)-C(1)-C(6)	120.0(3)
C(3)-C(2)-C(1)	120.7(3)
C(2)-C(3)-C(4)	120.3(3)
C(5)-C(4)-C(3)	119.5(3)
C(4)-C(5)-C(6)	121.0(3)
C(5)-C(6)-C(1)	118.5(2)
C(5)-C(6)-C(7)	119.0(2)
C(1)-C(6)-C(7)	122.5(2)
O(1)-C(7)-C(6)	120.1(2)

O(1)-C(7)-C(8)	119.3(2)
C(6)-C(7)-C(8)	120.6(2)
C(7)-C(8)-C(9)	113.2(2)
C(10)-C(9)-C(8)	112.1(2)
C(10)-C(9)-C(12)	103.0(2)
C(8)-C(9)-C(12)	112.6(2)
C(10)-C(9)-C(13)	108.5(2)
C(8)-C(9)-C(13)	109.7(2)
C(12)-C(9)-C(13)	110.7(2)
O(2)-C(10)-C(9)	126.3(3)
O(2)-C(10)-C(11)	124.6(3)
C(9)-C(10)-C(11)	109.0(2)
O(3)-C(11)-N(1)	128.3(3)
O(3)-C(11)-C(10)	125.0(3)
N(1)-C(11)-C(10)	106.7(2)
N(1)-C(12)-C(9)	105.6(2)
N(1)-C(14)-C(15)	113.4(3)
C(16)-C(15)-C(14)	113.8(4)
C(17')-C(16)-C(17)	61.2(10)
C(17')-C(16)-C(15)	124.8(7)
C(17)-C(16)-C(15)	114.2(4)

Symmetry transformations used to generate equivalent atoms:

Table S5. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3e**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

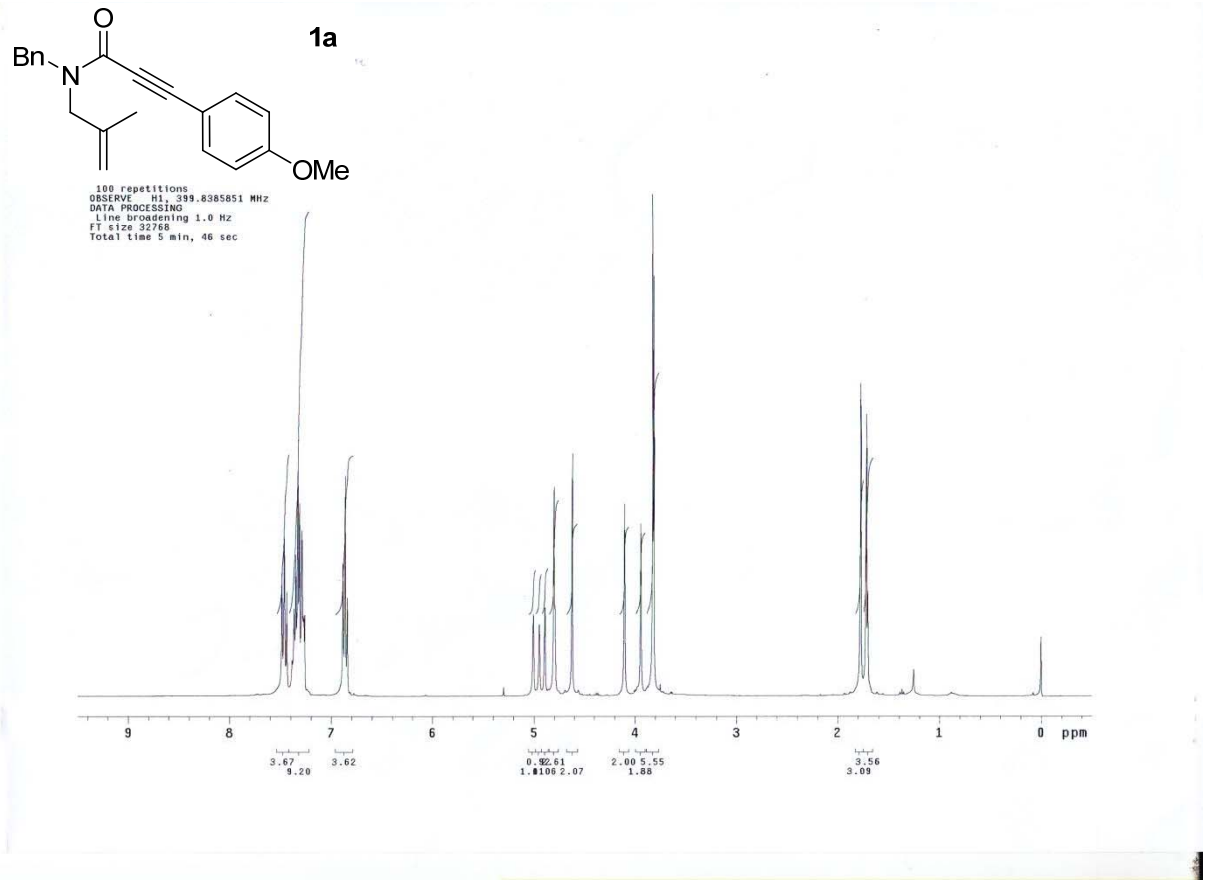
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	28(1)	32(1)	78(1)	-3(1)	9(1)	12(1)
O(2)	104(2)	54(1)	53(1)	19(1)	35(1)	48(1)
O(3)	47(1)	46(1)	131(2)	9(1)	40(1)	6(1)
N(1)	37(1)	29(1)	63(2)	-4(1)	8(1)	9(1)
C(1)	35(2)	41(2)	44(2)	-6(1)	0(1)	12(1)
C(2)	40(2)	45(2)	53(2)	-4(1)	3(1)	-2(1)
C(3)	73(2)	31(2)	56(2)	-1(1)	-4(2)	16(2)
C(4)	60(2)	34(2)	67(2)	0(1)	-11(2)	24(2)
C(5)	41(2)	33(1)	51(2)	0(1)	-5(1)	16(1)
C(6)	33(1)	32(1)	28(1)	2(1)	0(1)	12(1)
C(7)	30(1)	34(1)	36(1)	3(1)	5(1)	15(1)
C(8)	39(2)	40(2)	43(2)	0(1)	4(1)	22(1)
C(9)	46(2)	34(1)	37(1)	6(1)	9(1)	25(1)
C(10)	66(2)	30(1)	49(2)	12(1)	27(2)	29(1)
C(11)	44(2)	25(1)	87(2)	5(2)	20(2)	10(1)
C(12)	33(1)	33(1)	48(2)	2(1)	8(1)	12(1)
C(13)	67(2)	52(2)	57(2)	0(1)	7(2)	43(2)
C(14)	52(2)	55(2)	75(2)	-29(2)	-14(2)	14(2)
C(15)	67(2)	84(3)	68(2)	-36(2)	-28(2)	42(2)
C(16)	89(3)	143(4)	83(3)	-45(3)	-30(2)	83(3)
C(17)	44(4)	62(4)	48(3)	-5(3)	-2(3)	31(3)
C(17')	112(10)	350(20)	159(13)	-157(15)	-79(10)	132(14)

Table S6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3e**.

	x	y	z	U(eq)
H(1A)	13002	10196	1510	52
H(2A)	14125	12574	1577	68
H(3A)	12904	13694	1694	69
H(4A)	10514	12450	1757	64
H(5A)	9375	10081	1684	51
H(8A)	11380	7545	1899	48
H(8B)	11826	8069	1192	48
H(12A)	9817	7007	425	47
H(12B)	10046	5739	264	47
H(13A)	10335	4402	1123	79
H(13B)	11172	5312	1716	79
H(13C)	11748	5802	1018	79
H(14A)	6394	4022	99	79
H(14B)	7749	4527	-315	79
H(15A)	8089	6800	-301	86
H(15B)	6714	5733	-657	86
H(16A)	5554	5547	345	113
H(16B)	6930	6905	545	113
H(16C)	6519	6194	616	113
H(16D)	6787	7400	148	113
H(17A)	5355	7450	179	60
H(17B)	5264	6711	-477	60
H(17C)	6650	8070	-283	60
H(17D)	4843	6376	459	238
H(17E)	4645	4951	205	238
H(17F)	4914	6146	-280	238

Appendix

Copy of ^{1}H and ^{13}C NMR Spectra of
Substrates and Products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Bn
N
O
OMe
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DATA PROCESSING
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FT size 32768
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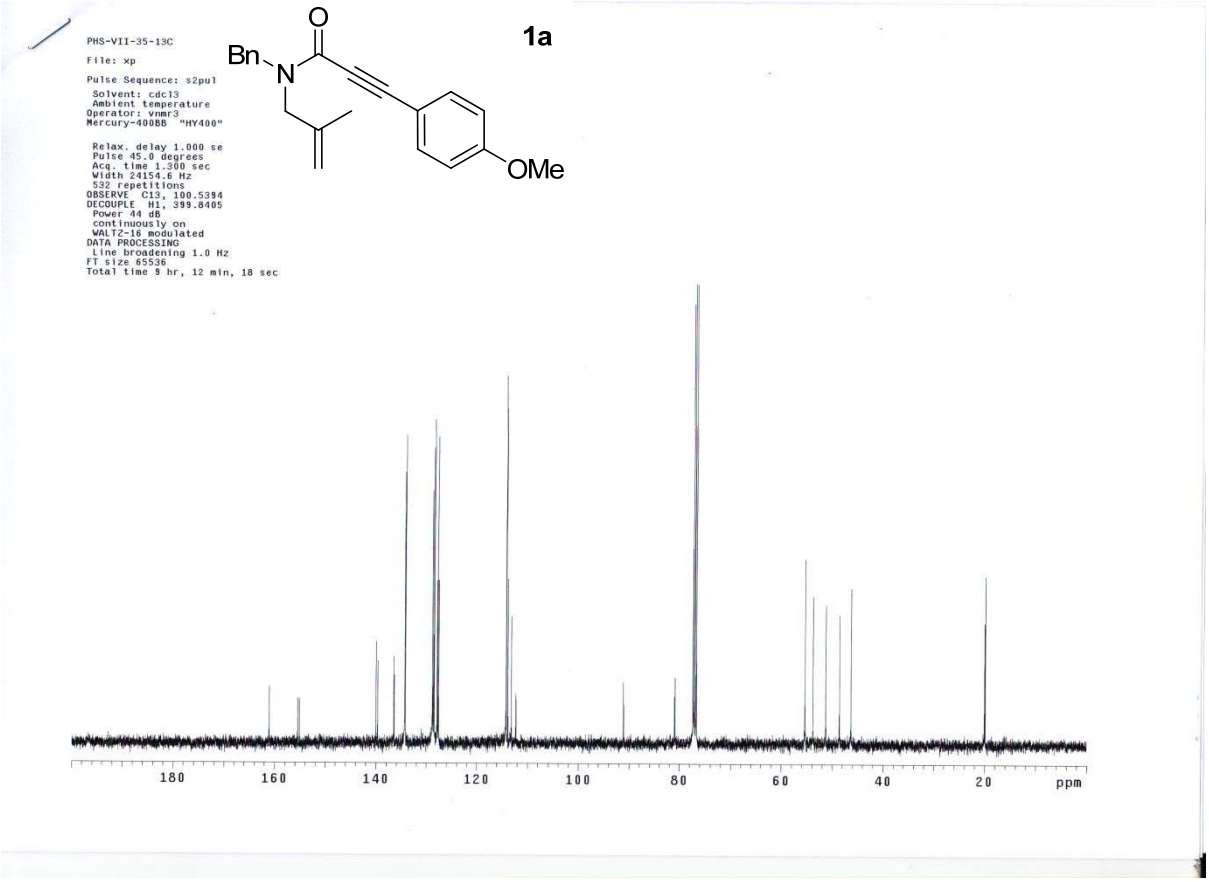
9
8
7
6
5
4
3
2
1
0 ppm
3.67
9.20
3.62
0.92
1.00
2.61
2.07
2.00
5.55
1.88
3.56
3.09


1a
Bn
N
O
OMe
PHS-VII-35-13C
File: xp
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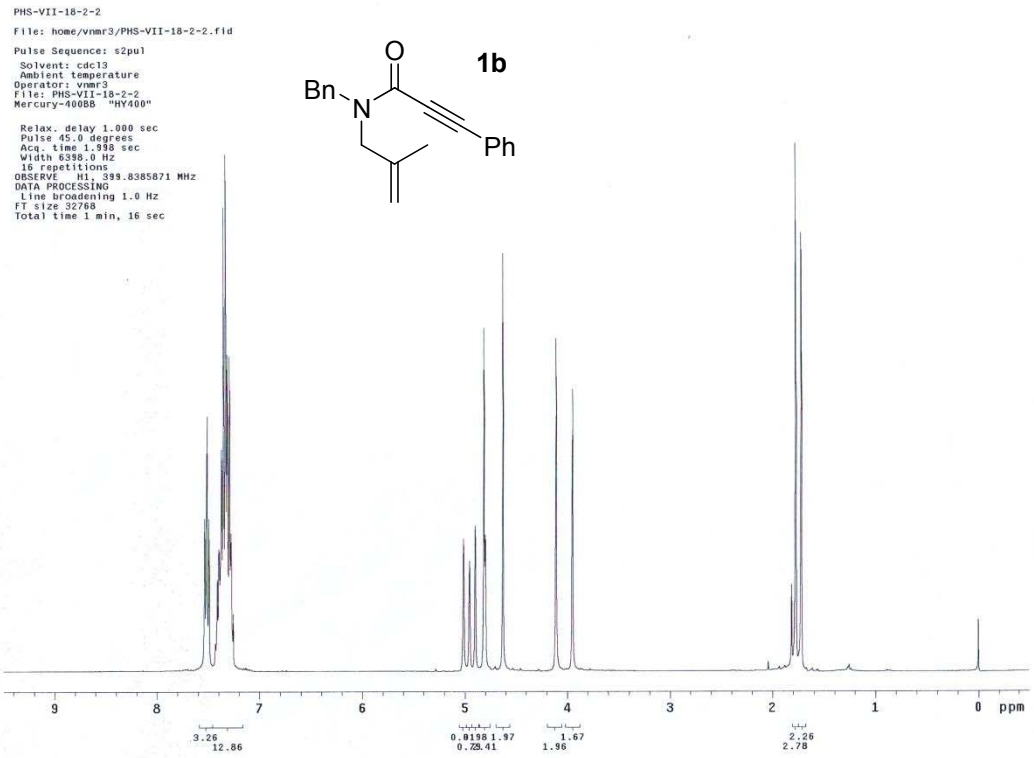
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 se
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
532 repetitions
OBSERVE C13, 100.5394
DECOUPLE H1, 399.8405
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
180
160
140
120
100
80
60
40
20
ppm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File: home/vnmr3/PHS-VII-18-2-2.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VII-18-2-2
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385871 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 min, 16 sec
1b
Bn
N
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
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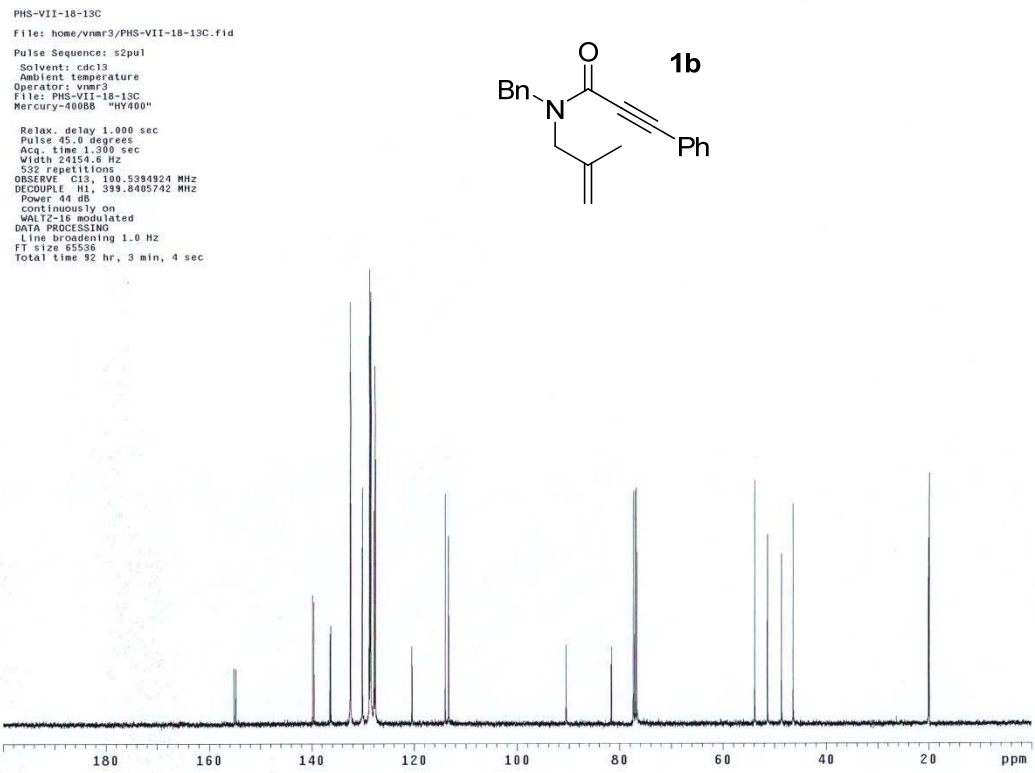
12.86
0.81
0.79
1.97
1.41
1.67
1.96
2.26
2.78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Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VII-18-13C
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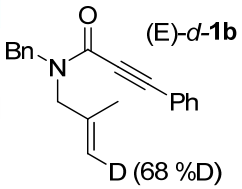
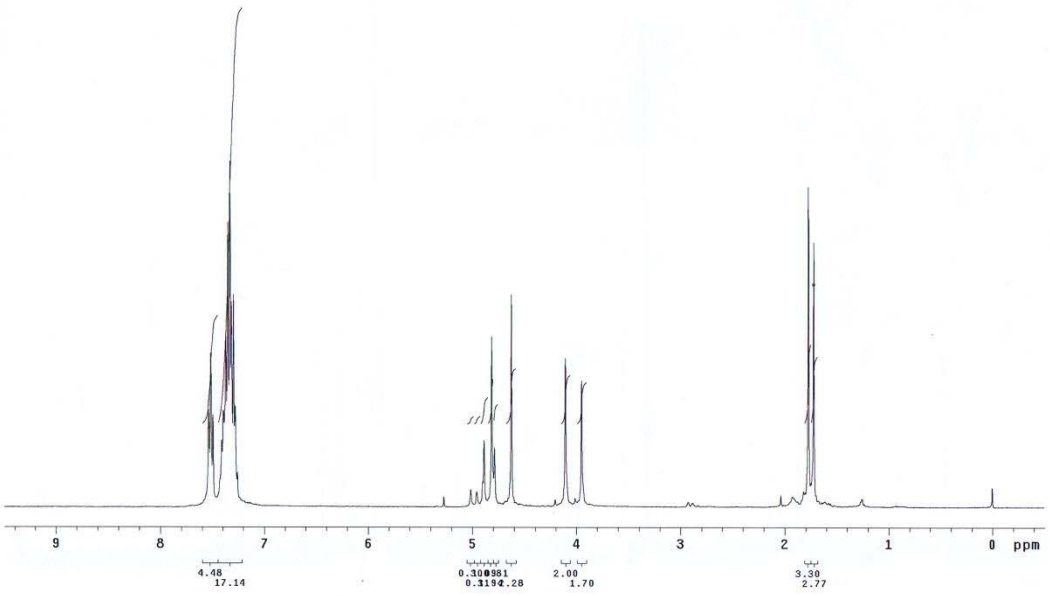
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
532 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 92 hr, 3 min, 4 sec
1b
Bn
N
O
Ph
180
160
140
120
100
80
60
40
20
ppm

KJY-VI-32
File: xp
Pulse Sequence: s2pul



(E)-*d*-**1b**



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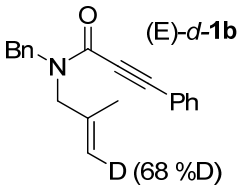
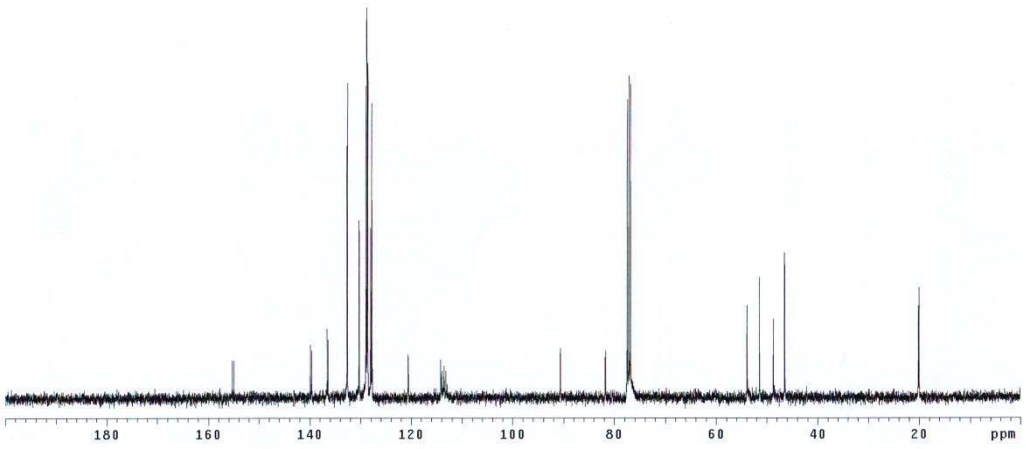
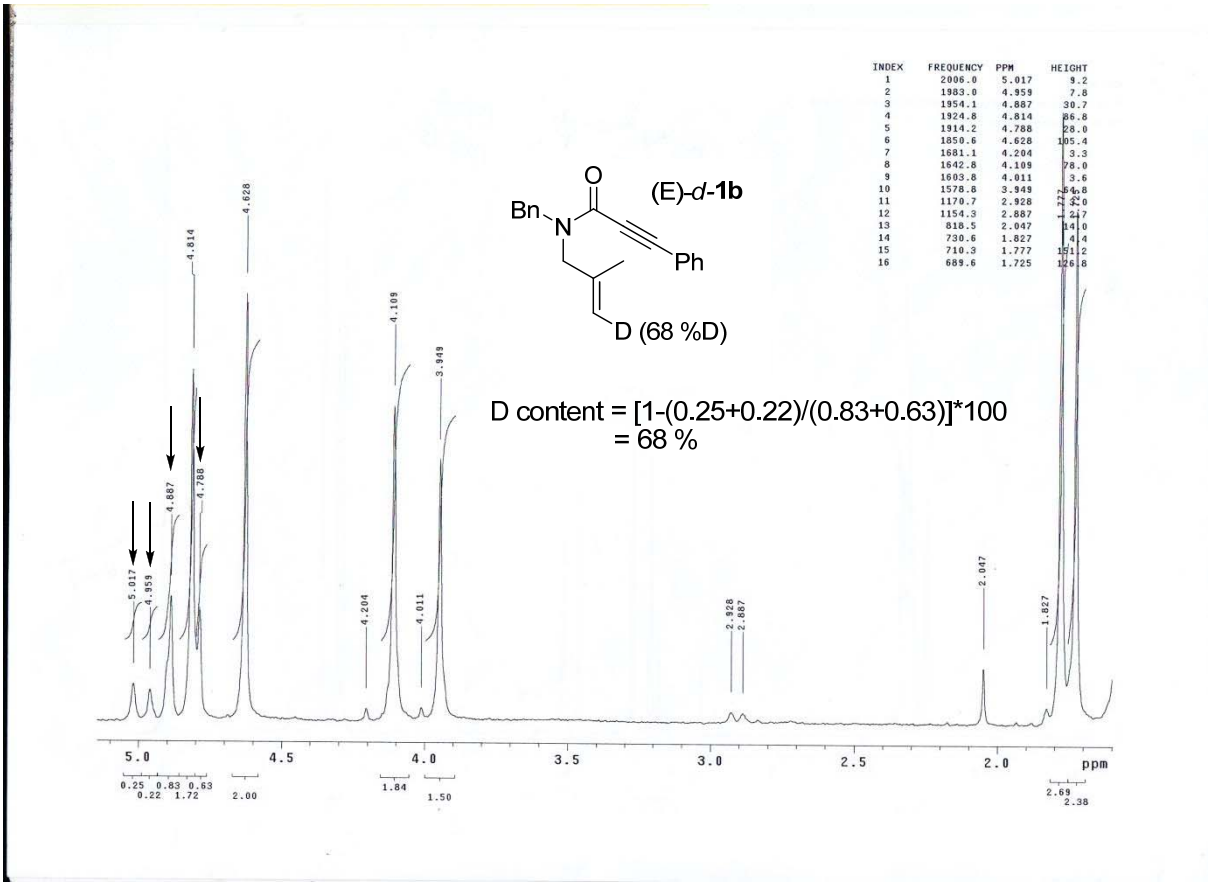
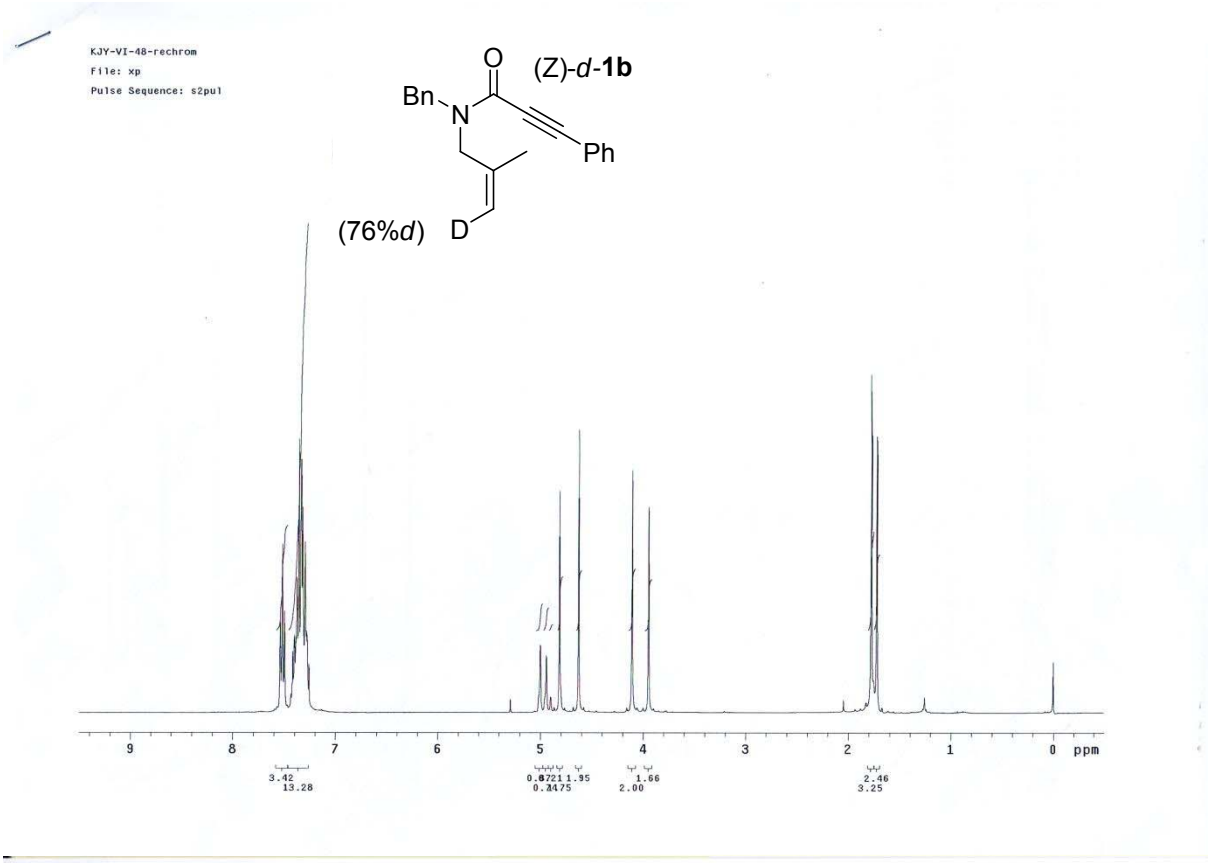
Pulse Sequence: s2pul

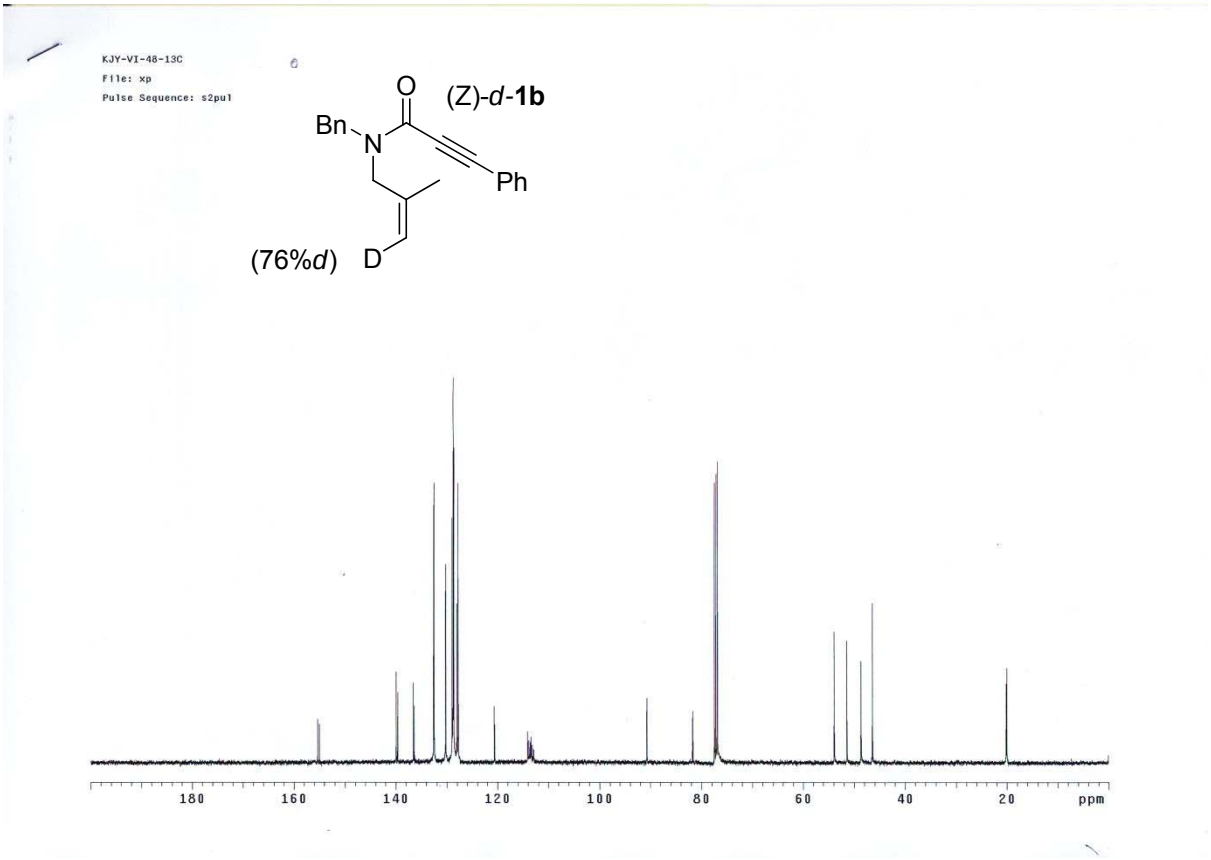


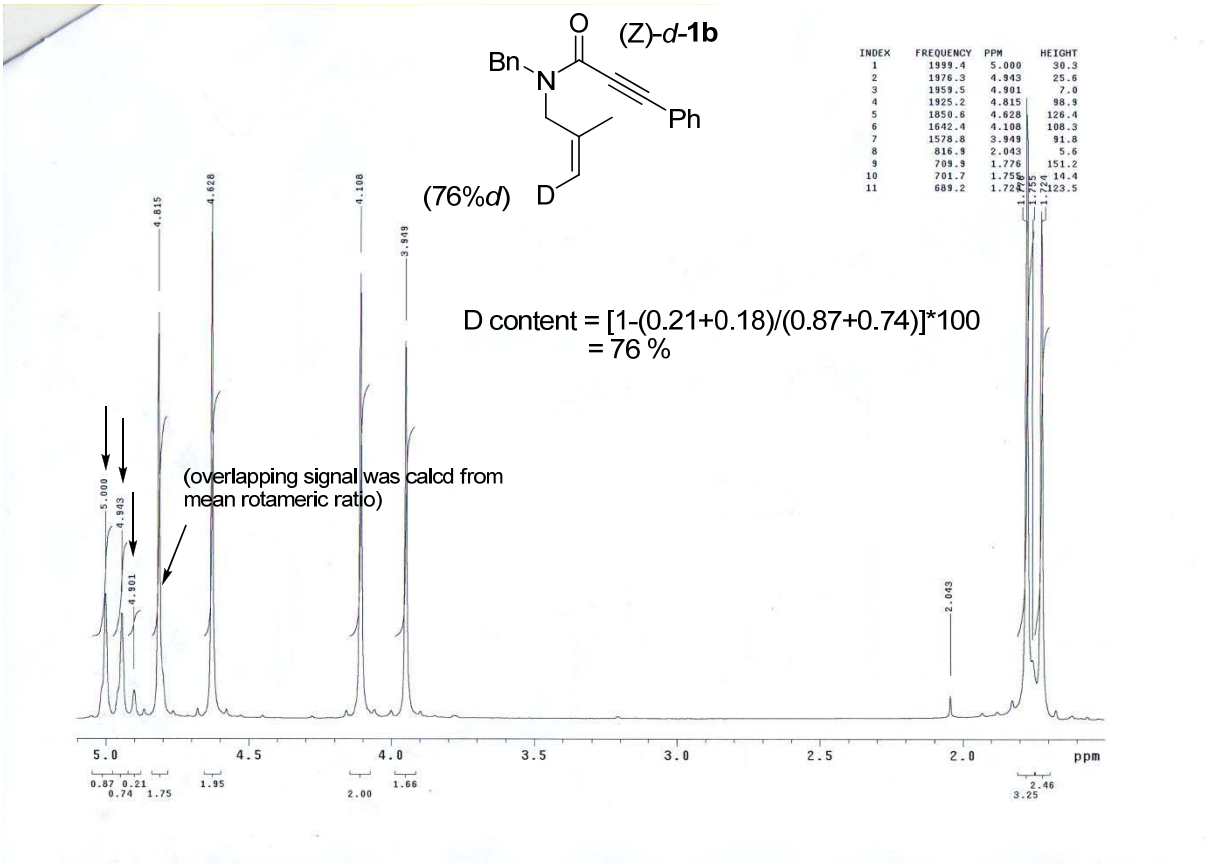
(E)-*d*-**1b**

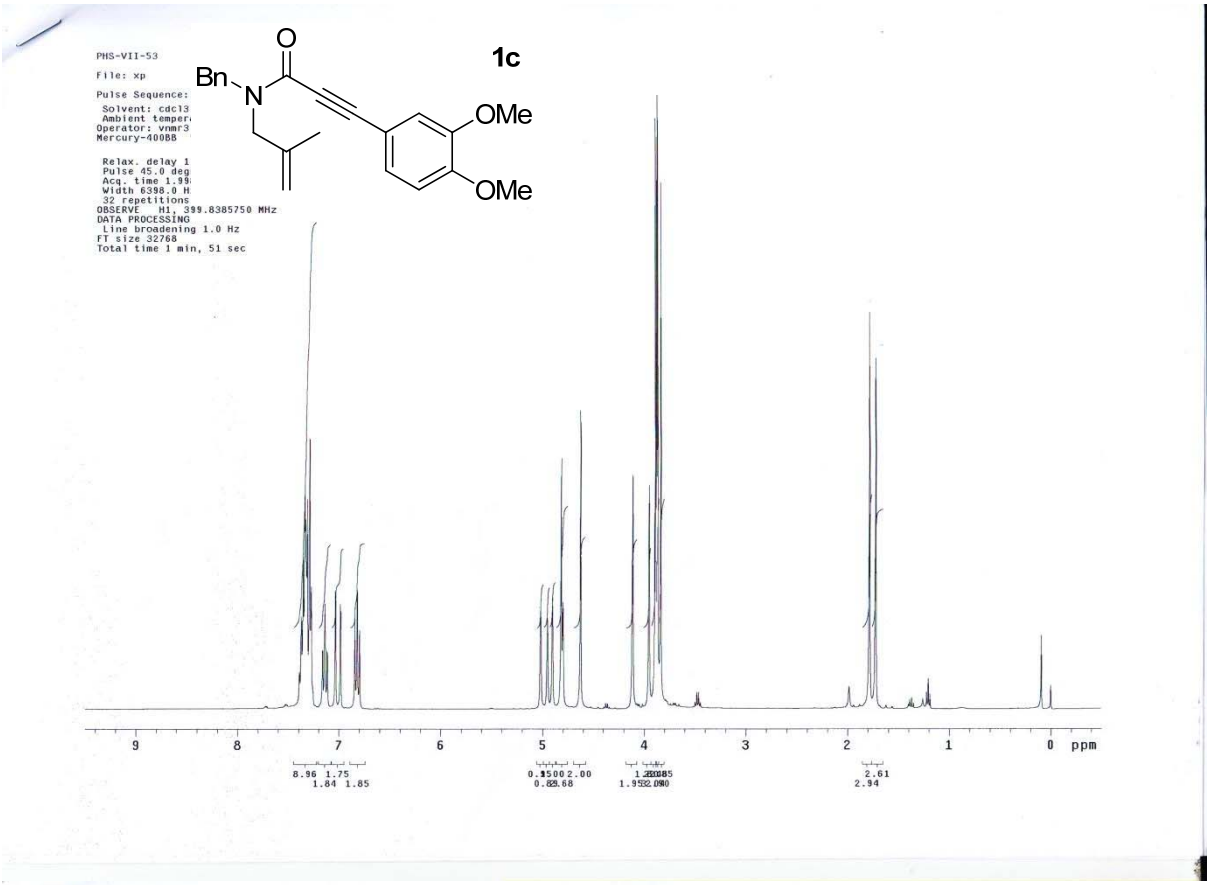


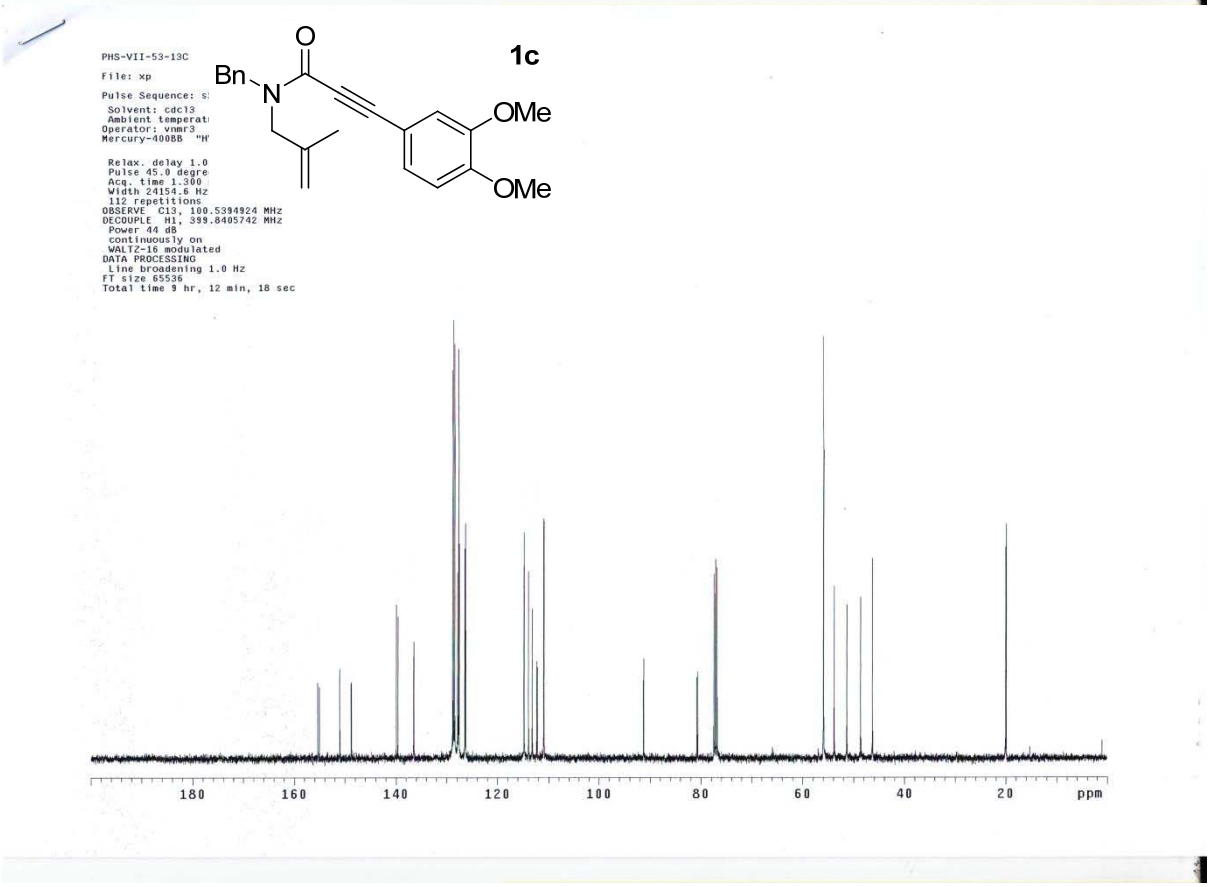

INDEX FREQUENCY PPM HEIGHT
1 2006.0 5.017 9.2
2 1983.0 4.959 7.8
3 1954.1 4.887 30.7
4 1924.8 4.814 86.8
5 1914.2 4.788 28.0
6 1850.6 4.628 105.4
7 1681.1 4.204 3.3
8 1642.8 4.109 78.0
9 1603.8 4.011 3.6
10 1578.8 3.948 54.8
11 1170.7 2.928 2.0
12 1154.3 2.887 2.7
13 818.5 2.047 14.0
14 730.6 1.827 4.4
15 710.3 1.777 151.2
16 689.6 1.725 126.8
(E)-d-1b
Bn
N
O
Ph
D (68 %D)
D content = [1-(0.25+0.22)/(0.83+0.63)]*100
= 68 %
5.017
4.959
4.887
4.814
4.788
4.628
4.204
4.109
4.011
3.948
2.928
2.887
2.047
1.827
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
0.25 0.83 0.63
0.22 1.72
2.00
1.84
1.50
2.69
2.38

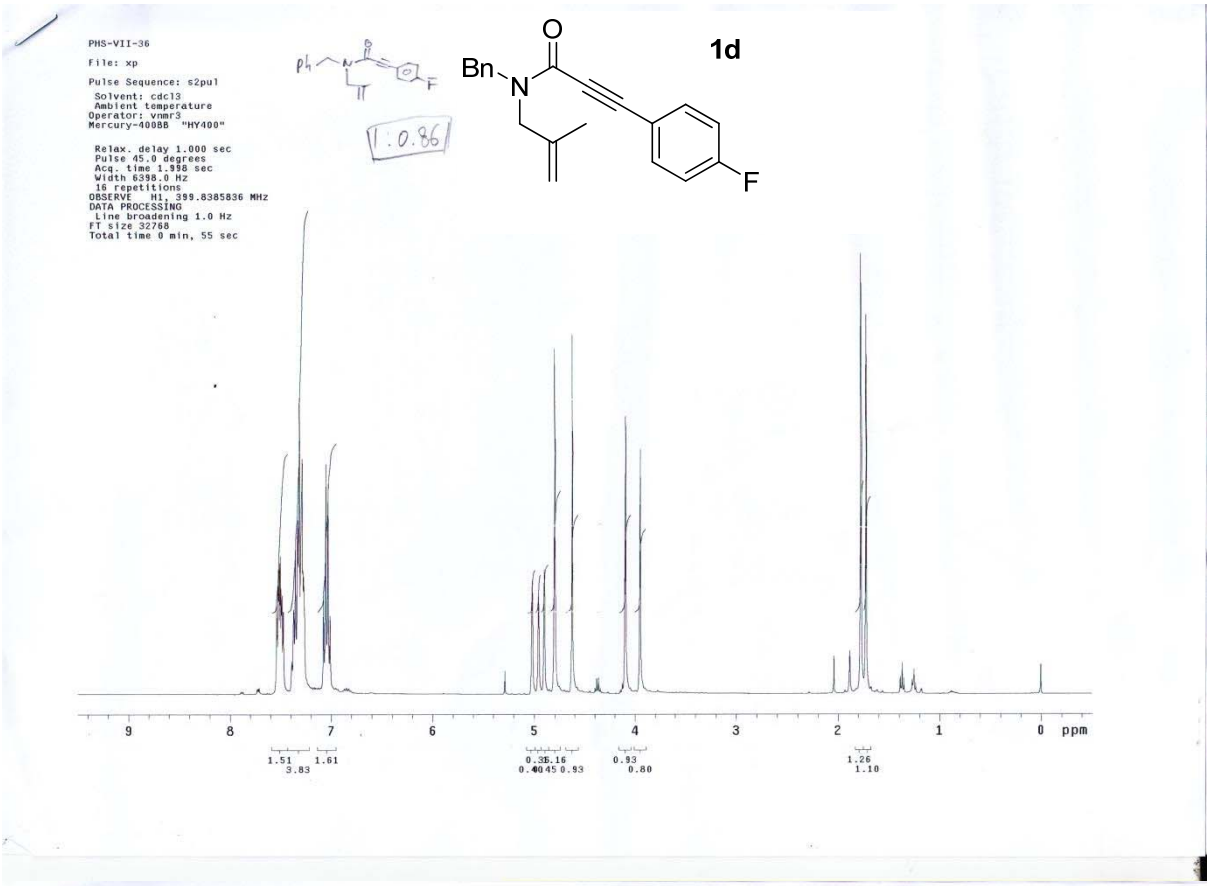

KJY-VI-48-rechrom
File: xp
Pulse Sequence: s2pul
(Z)-*d*-**1b**
Bn
N
O
Ph
(76%*d*)
D
9
8
7
6
5
4
3
2
1
0
ppm
3.42
13.28
1.95
1.66
2.00
2.46
3.25

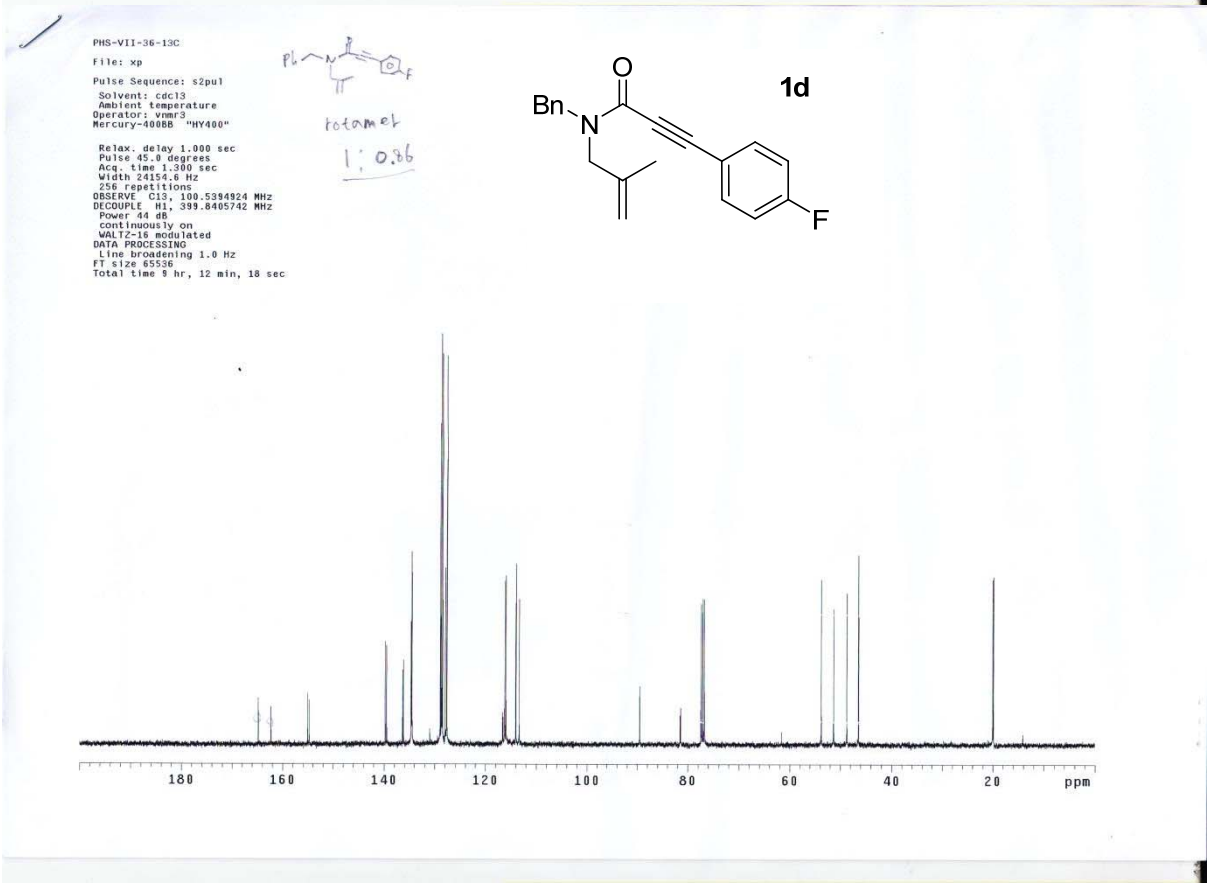

KJY-VI-48-13C
File: xp
Pulse Sequence: s2pul
(Z)-*d*-**1b**
Bn
N
O
Ph
(76%*d*)
D
180
160
140
120
100
80
60
40
20
ppm

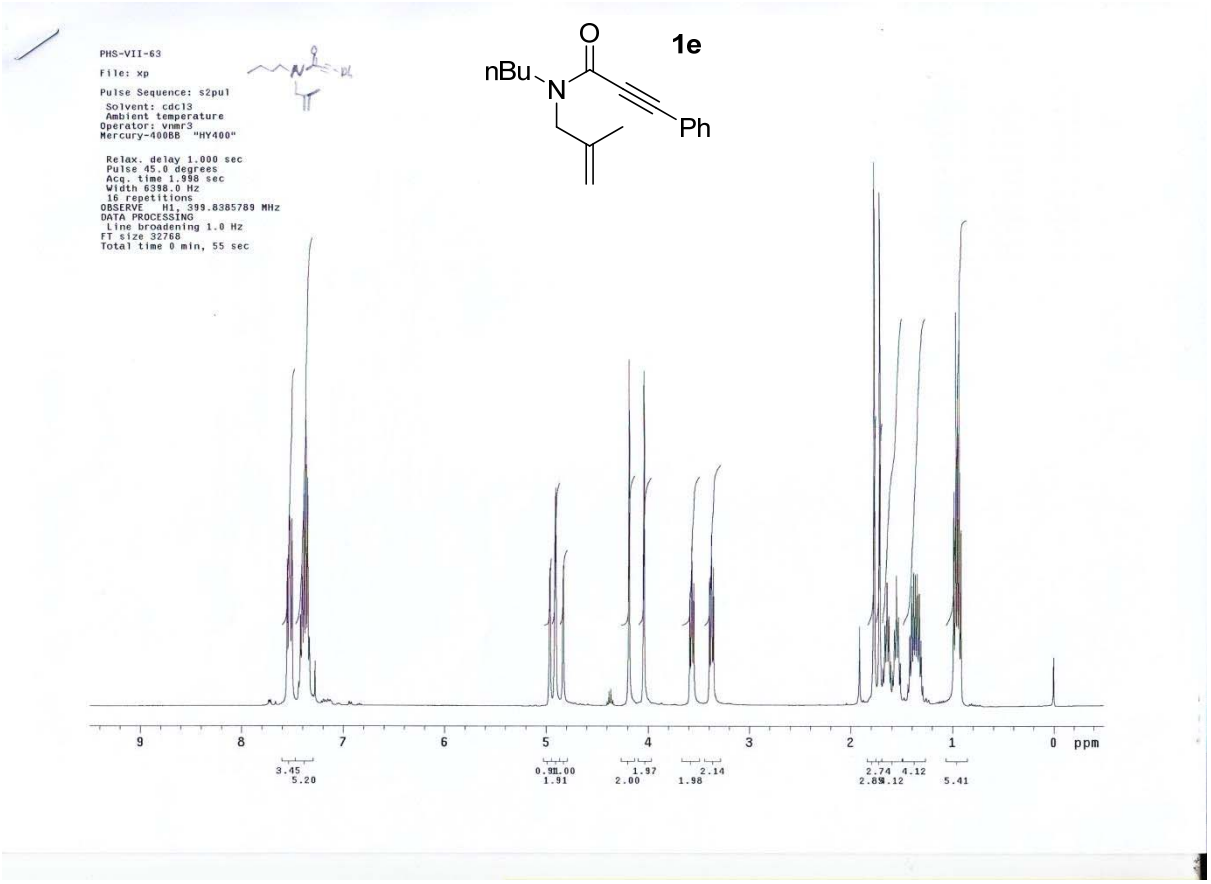

(Z)-d-1b
Bn
N
O
Ph
D
(76%d)
INDEX FREQUENCY PPM HEIGHT
1 1999.4 5.000 30.3
2 1976.3 4.943 25.6
3 1959.5 4.901 7.0
4 1925.2 4.815 98.9
5 1850.6 4.628 126.4
6 1642.4 4.108 108.3
7 1578.8 3.949 91.8
8 816.9 2.043 5.6
9 709.9 1.776 151.2
10 701.7 1.756 14.4
11 689.2 1.724 123.5
D content = [1-(0.21+0.18)/(0.87+0.74)]*100
= 76 %
(overlapping signal was calcd from
mean rotameric ratio)
5.000
4.943
4.901
4.815
4.628
4.108
3.949
2.043
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
0.87
0.74
0.21
1.75
1.95
2.00
1.66
3.25
2.46

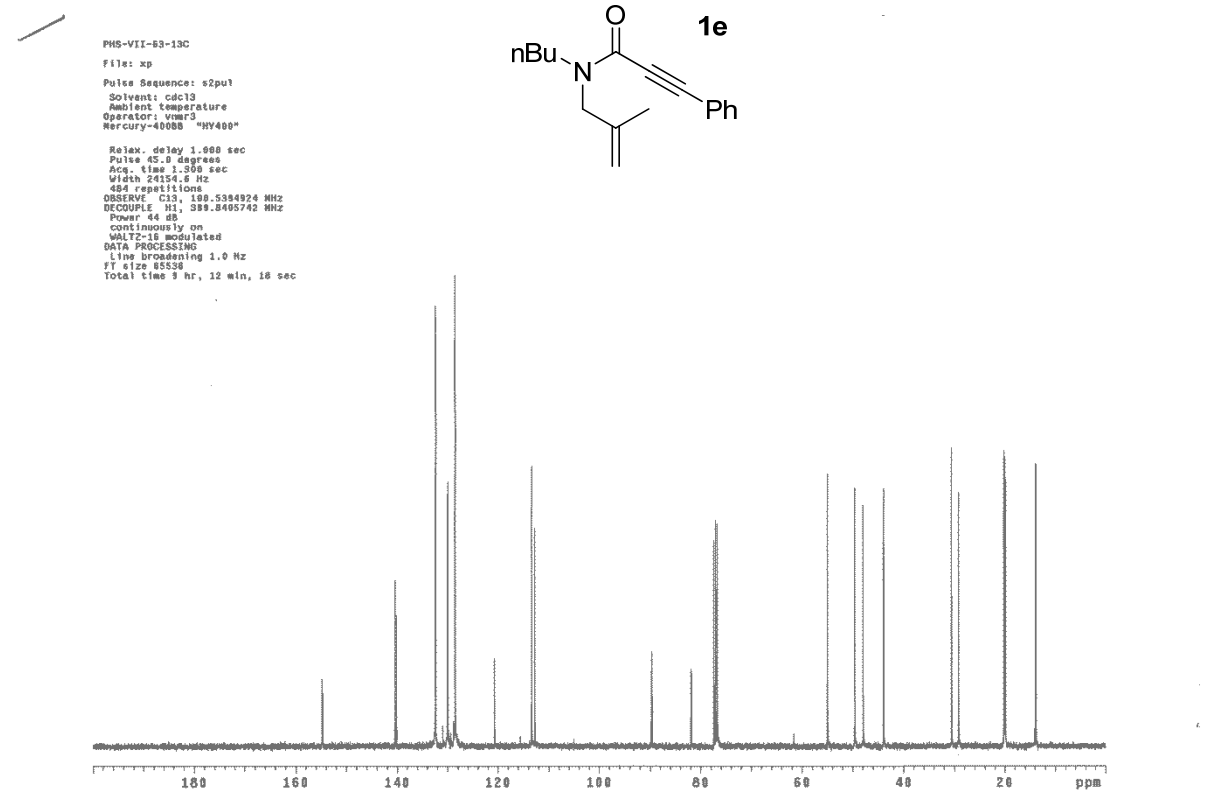

PHS-VII-53
File: xp
Pulse Sequence:
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB
Relax. delay 1
Pulse 45.0 deg
Acq. time 1.99
Width 6398.0 H
32 repetitions
OBSERVE H1, 399.8385750 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 min, 51 sec
1c
Bn
N
O
OMe
OMe
9
8
7
6
5
4
3
2
1
0 ppm
8.96 1.75
1.84 1.85
2.00
2.61
2.94

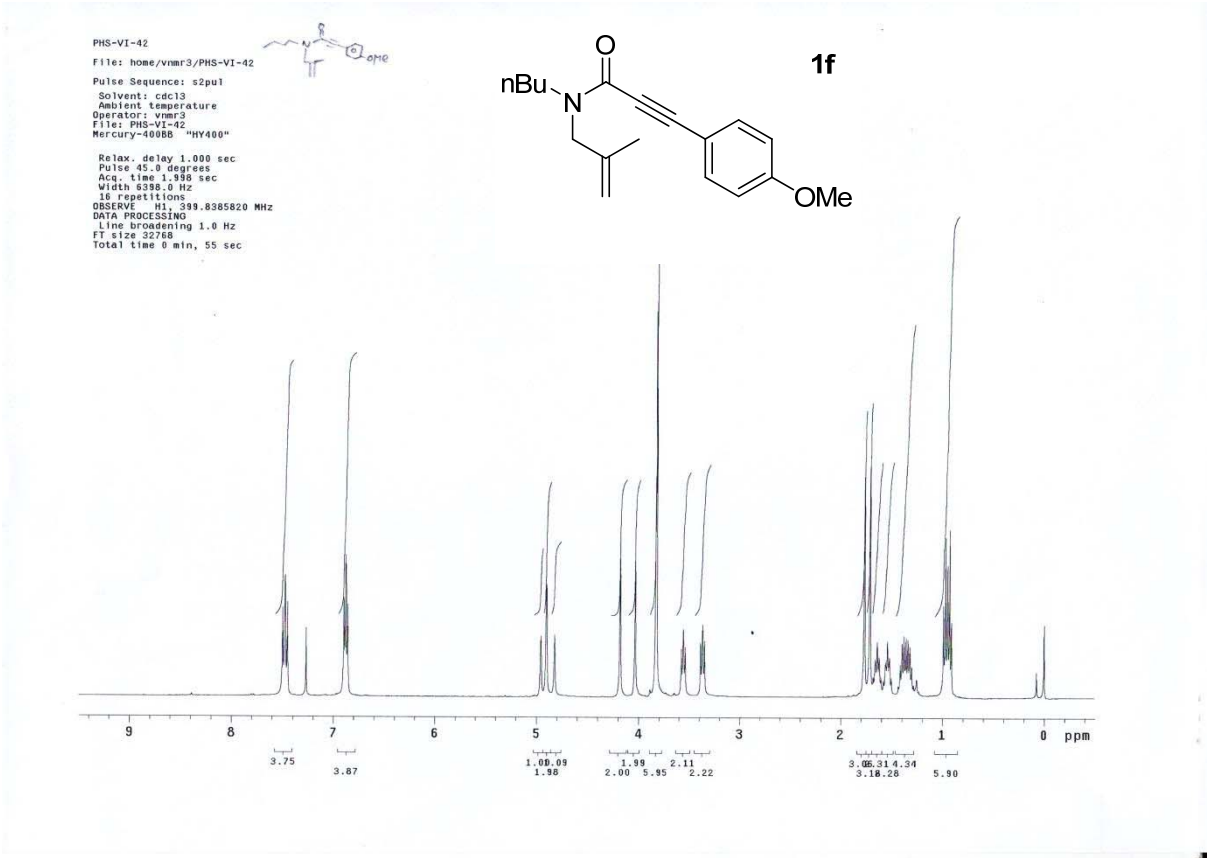

PHS-VII-53-13C
File: xp
Pulse Sequence:
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB
Relax. delay 1.0
Pulse 45.0 degre
Acq. time 1.300
Width 24154.6 Hz
112 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
1c
Bn
N
O
OMe
OMe
180
160
140
120
100
80
60
40
20
ppm

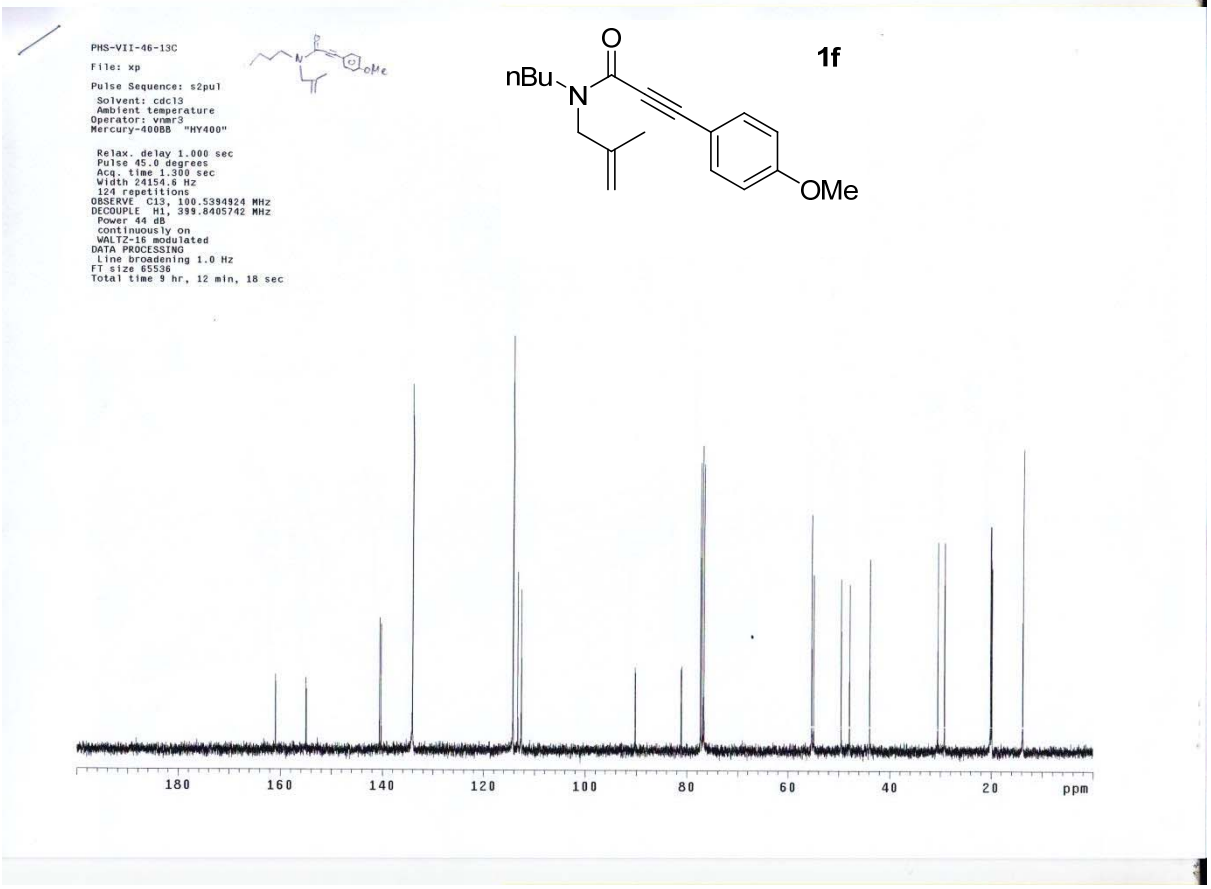

PHS-VII-36
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385836 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
1:0.86
1d
Bn
N
O
F
9
8
7
6
5
4
3
2
1
0
ppm
1.51
3.83
1.61
0.40
0.45
0.93
0.93
0.80
1.26
1.10

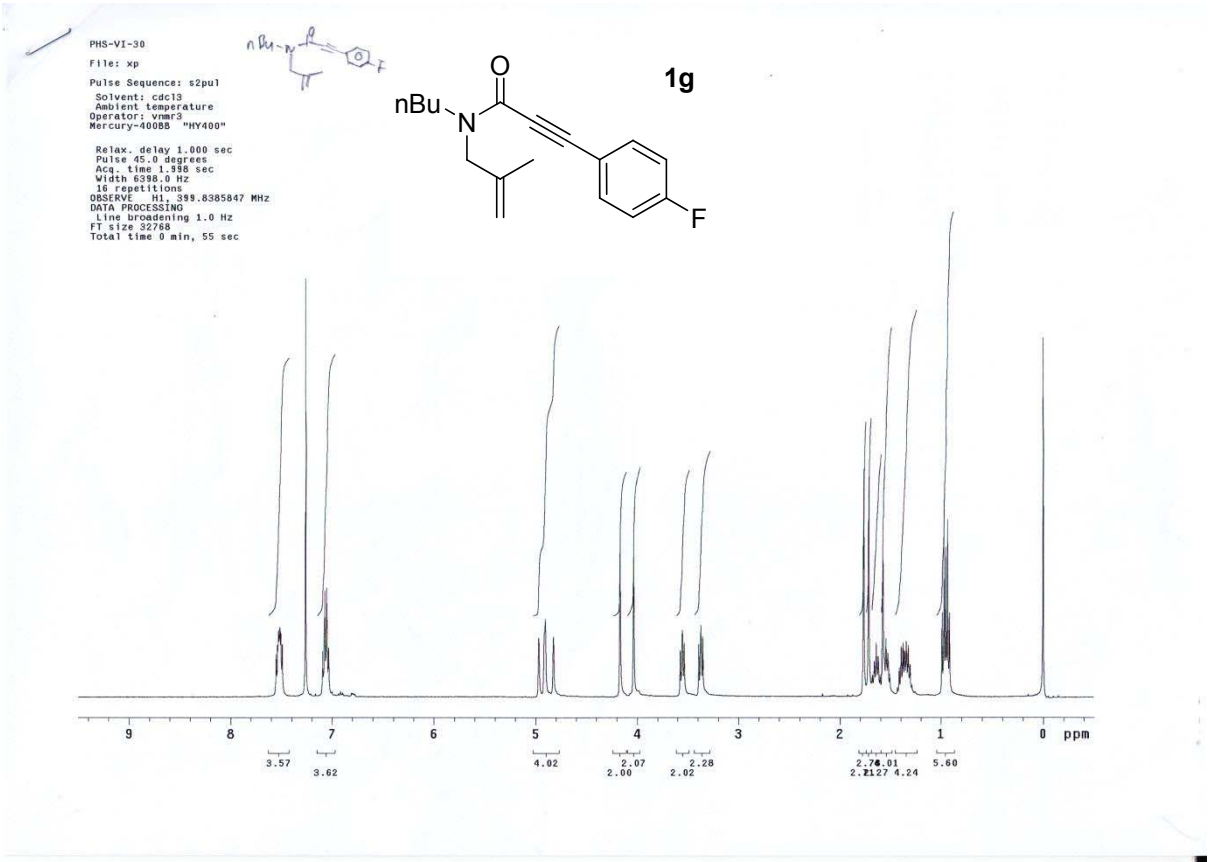

PHS-VII-36-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
256 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
rotamer
1:0.86
1d
Bn
N
O
F
180
160
140
120
100
80
60
40
20
ppm

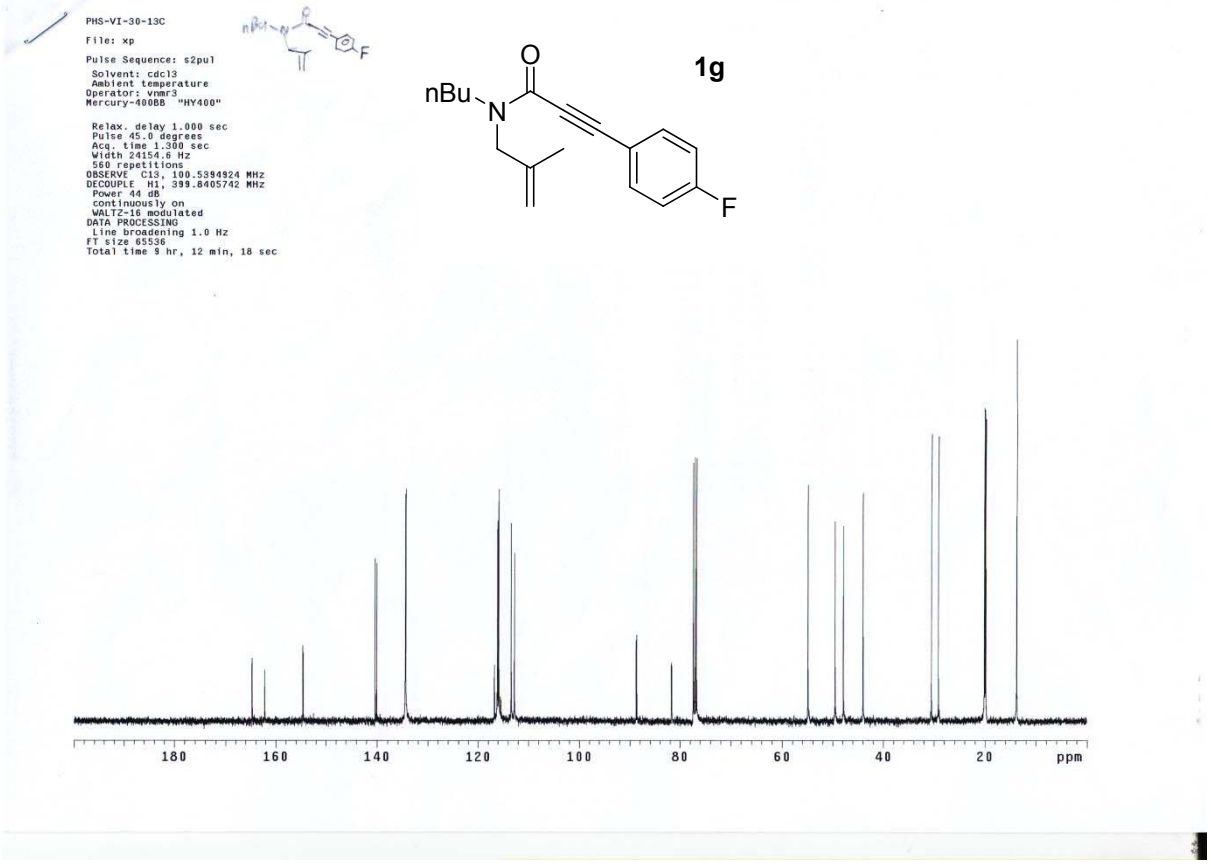

PHS-VII-63
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385789 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
nBu
N
O
Ph
1e
9
8
7
6
5
4
3
2
1
0
ppm
3.45
5.20
0.91
1.00
1.91
1.97
2.00
2.14
1.98
2.74
2.69
4.12
5.12
5.41

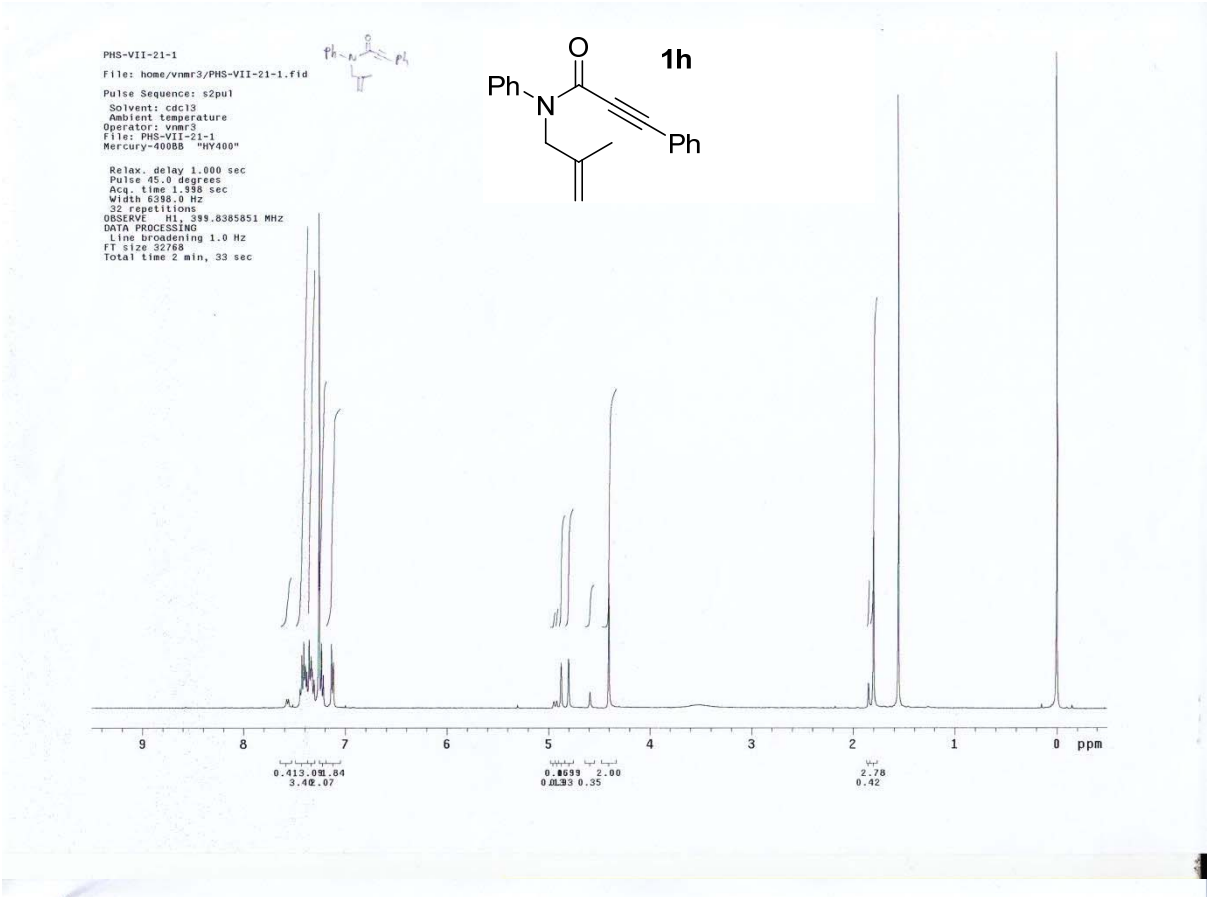

PHS-VII-63-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.8 Hz
484 repetitions
OBSERVE C13, 100.5384924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 16 sec
nBu
N
O
Ph
1e
180
160
140
120
100
80
60
40
20
ppm

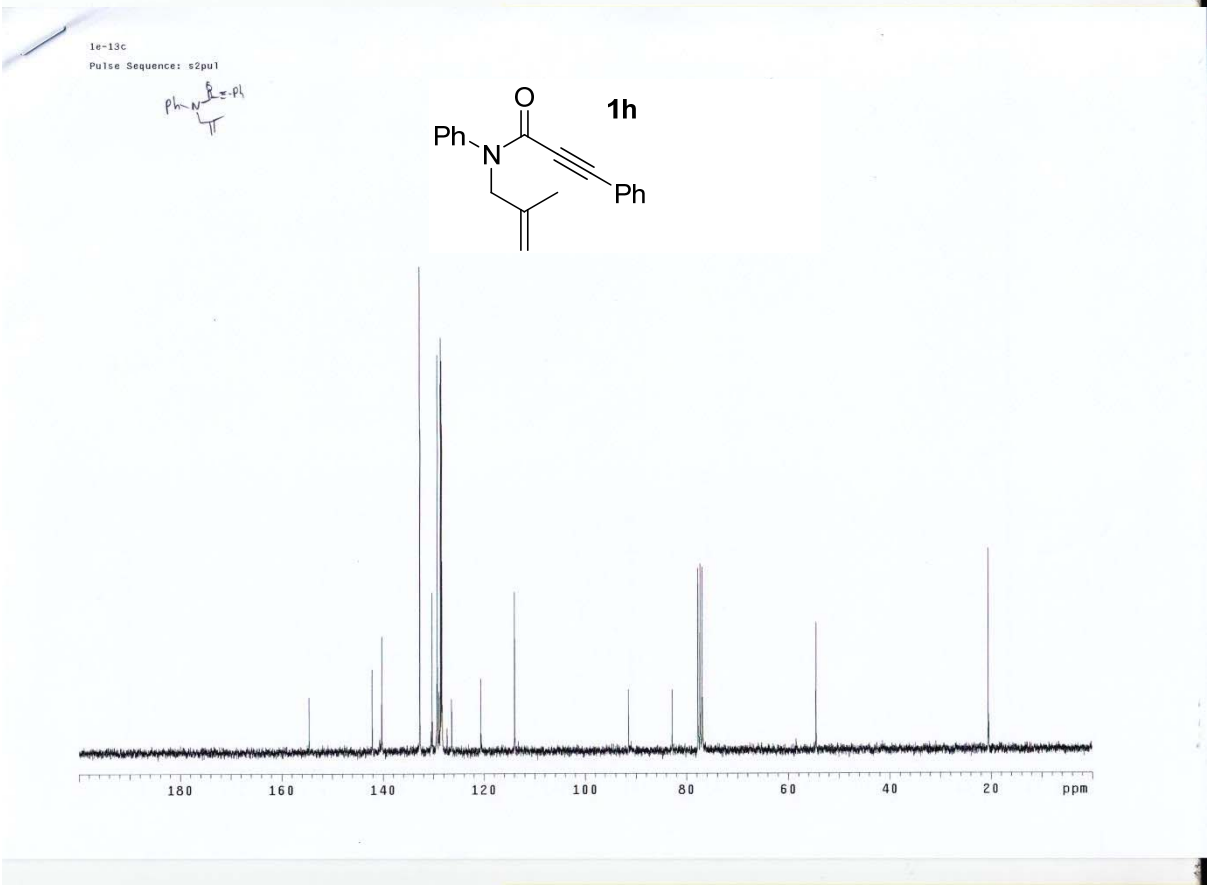

PHS-VI-42
File: home/vnmr3/PHS-VI-42
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VI-42
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385820 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
nBu
N
O
OMe
1f
9
8
7
6
5
4
3
2
1
0
ppm
3.75
3.87
1.00
0.09
1.98
1.99
2.00
5.95
2.11
2.22
3.06
3.18
1.31
4.34
28
5.90

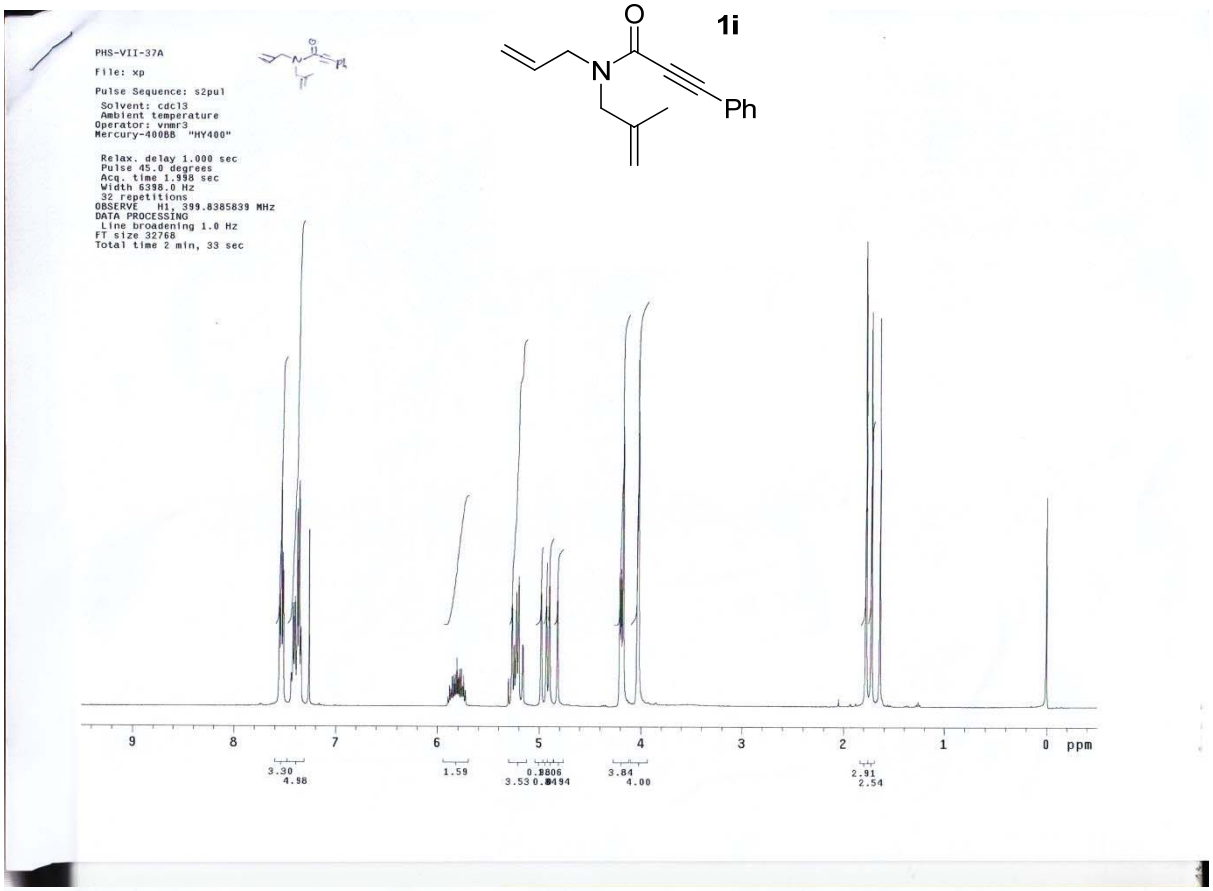

PHS-VII-46-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
124 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
nBu
N
O
OMe
1f
180
160
140
120
100
80
60
40
20
ppm

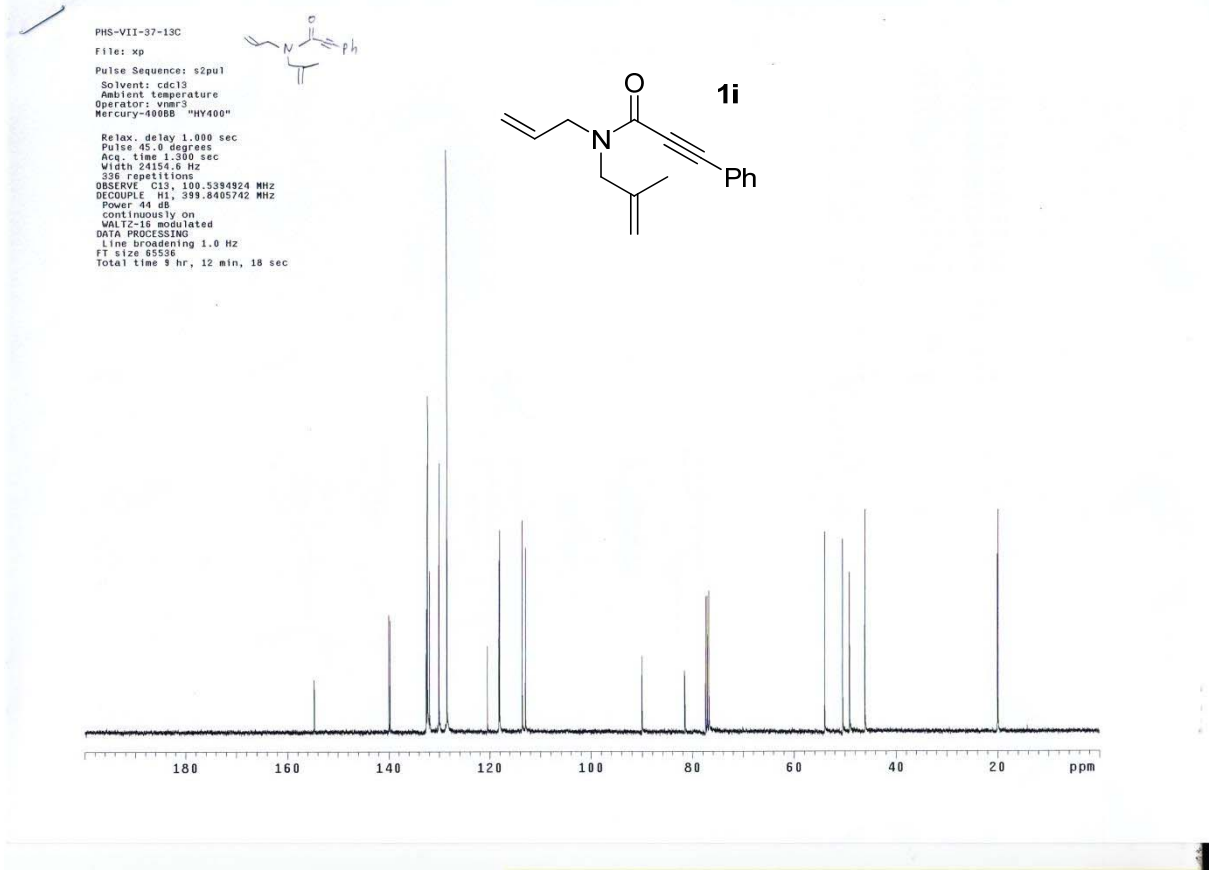

PHS-VI-30
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385847 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
nBu
N
O
F
1g
9
8
7
6
5
4
3
2
1
0
ppm
3.57
3.62
4.02
2.07
2.00
2.28
2.02
2.76
2.01
2.27
4.24
5.60

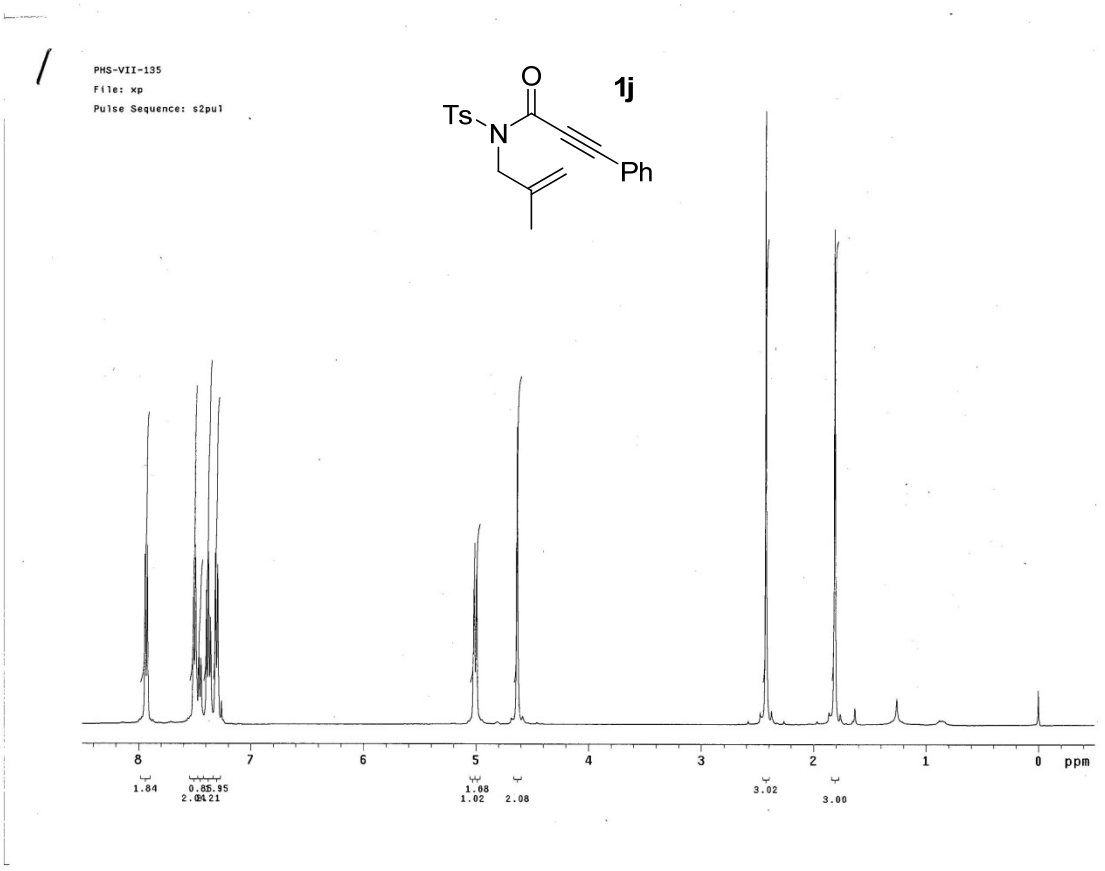

PHS-VI-30-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
560 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
nBu
N
O
F
1g
180
160
140
120
100
80
60
40
20
ppm

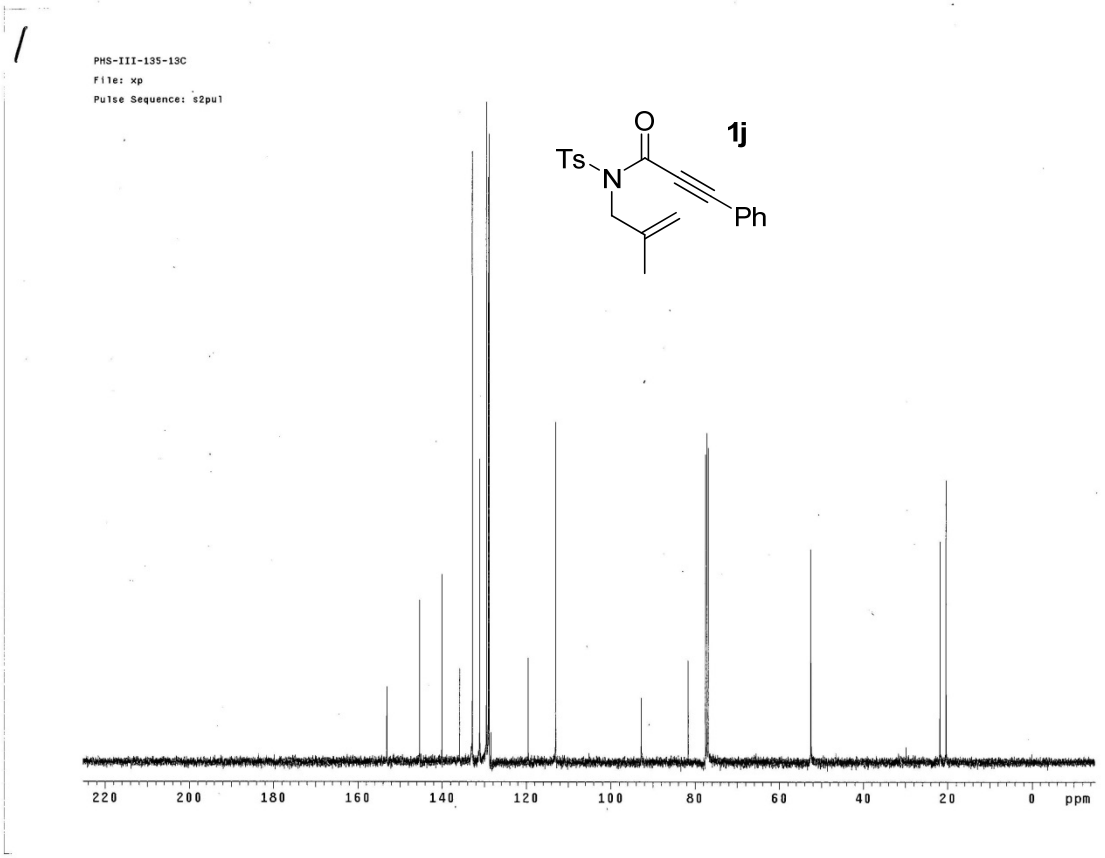

PHS-VII-21-1
File: home/vnmr3/PHS-VII-21-1.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VII-21-1
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8385851 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 2 min, 33 sec
1h
Ph
N
O
Ph
9
8
7
6
5
4
3
2
1
0 ppm
0.41
3.40
3.07
1.84
2.00
0.35
2.78
0.42

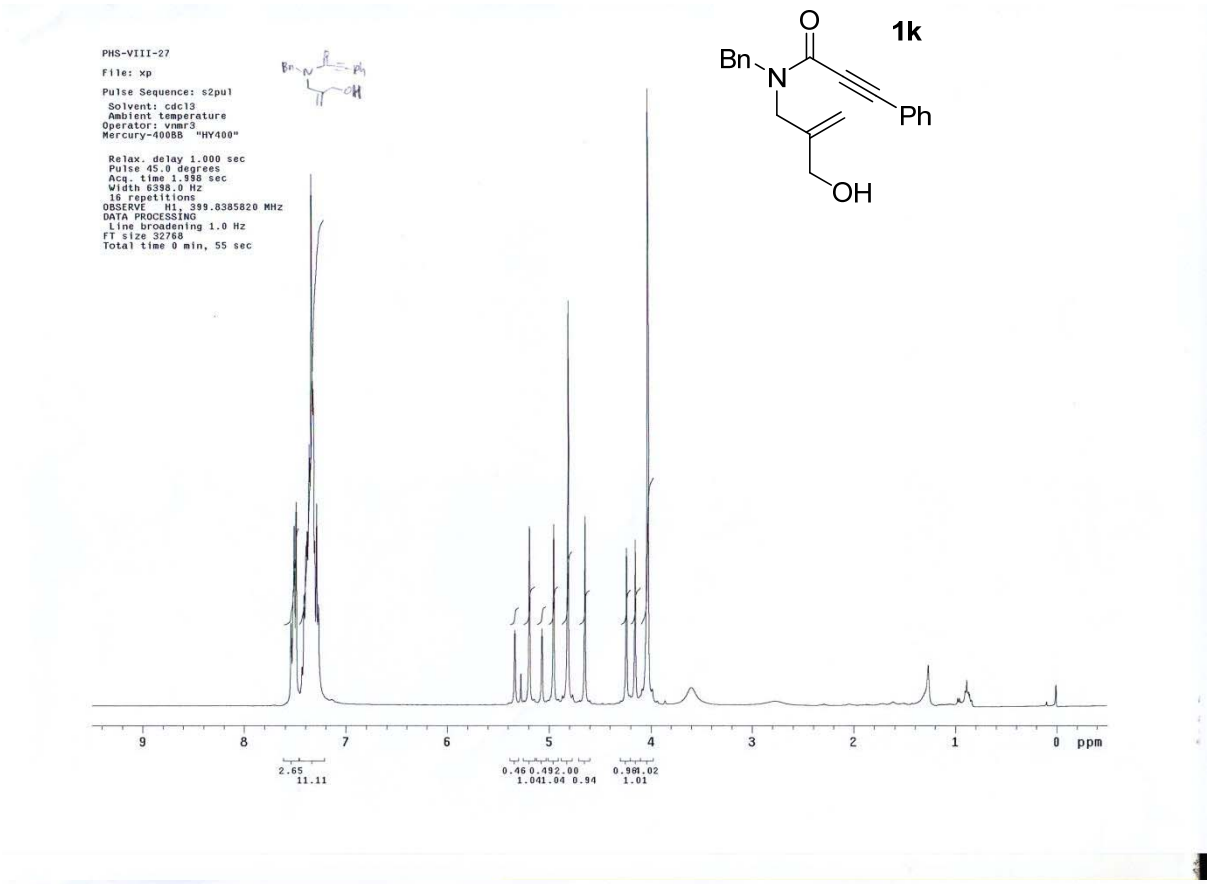

1e-13c
Pulse Sequence: s2pul
1h
Ph
N
O
Ph
180
160
140
120
100
80
60
40
20
ppm

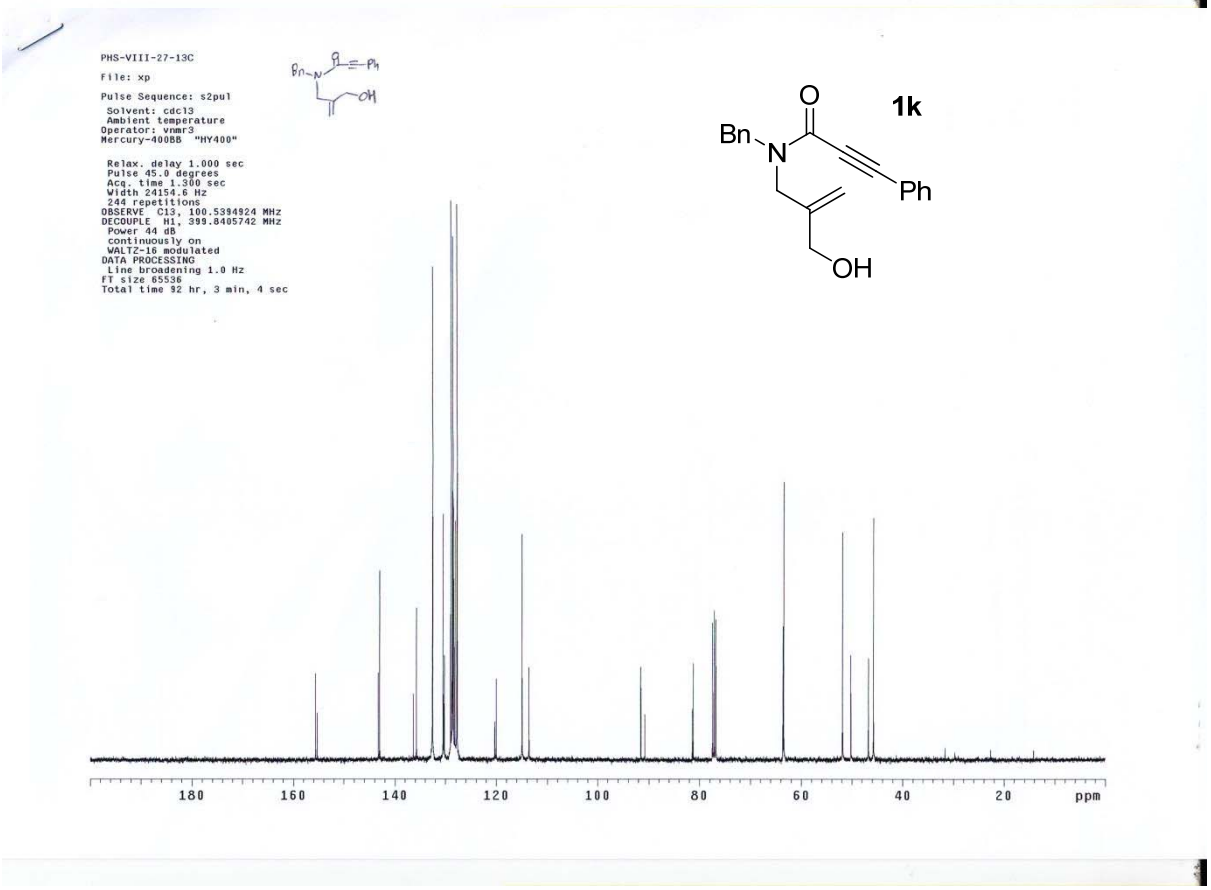

PHS-VII-37A
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8385839 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 2 min, 33 sec
1i
O
N
Ph
9
8
7
6
5
4
3
2
1
0 ppm
3.30
4.98
1.59
0.8806
3.53
4.00
2.91
2.54

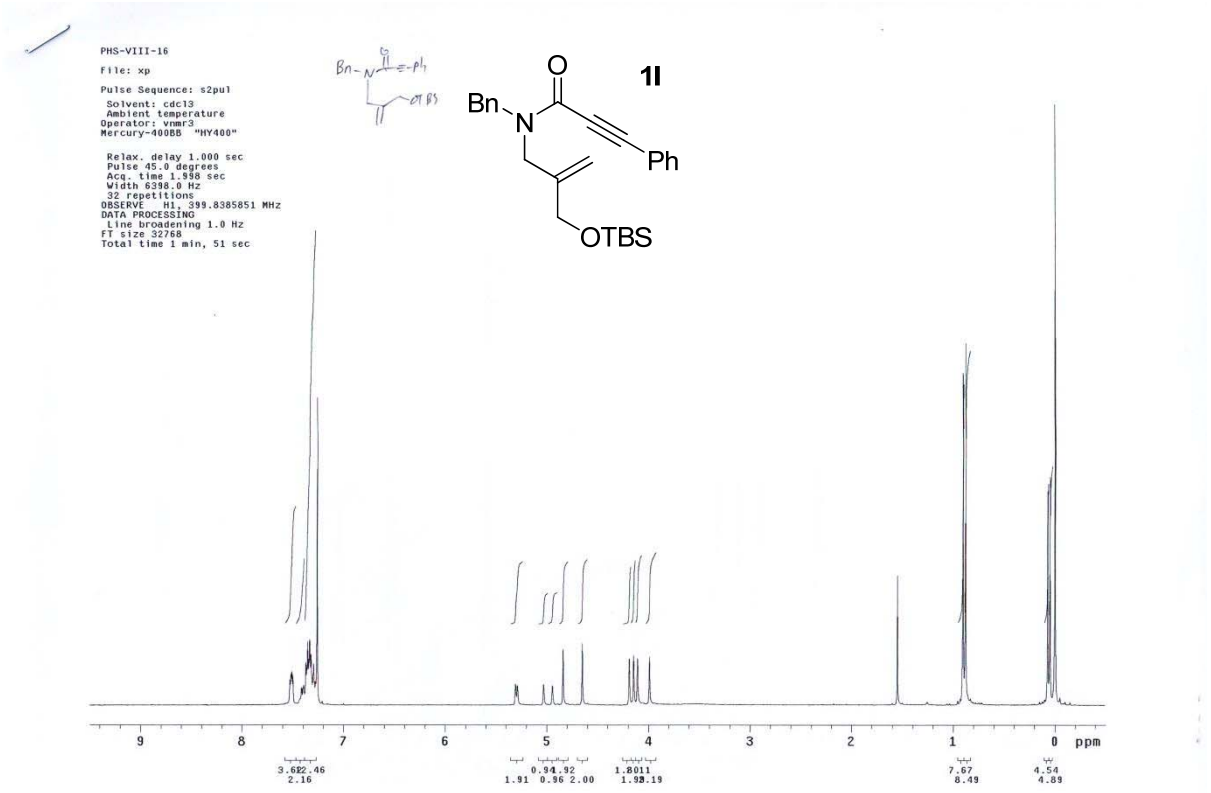

PHS-VII-37-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
336 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
1i
O
N
Ph
180
160
140
120
100
80
60
40
20
ppm

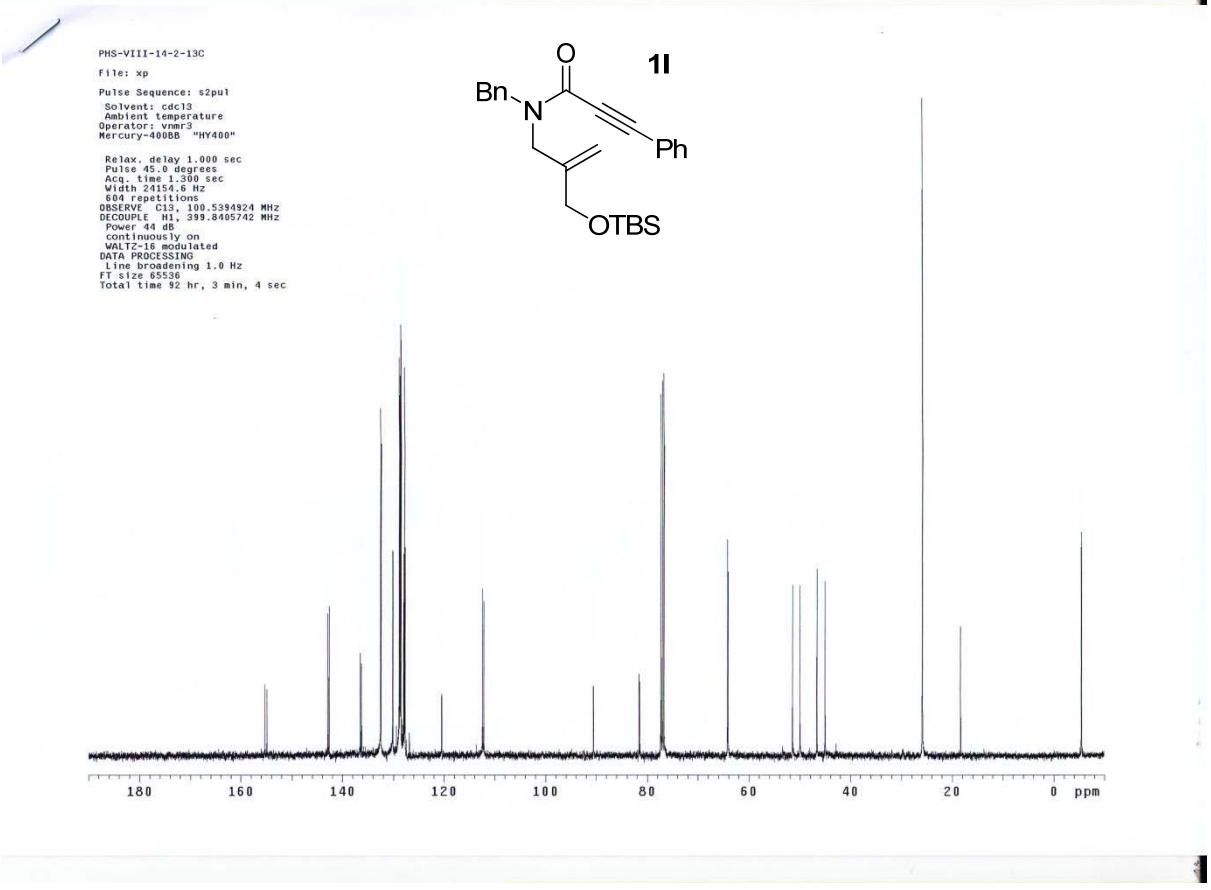

PHS-VII-135
File: xp
Pulse Sequence: s2pul
1j
Ts
N
O
Ph
8
7
6
5
4
3
2
1
0
ppm
1.84
0.85
0.95
2.04
4.21
1.08
1.02
2.08
3.02
3.00


PHS-III-135-13C
File: xp
Pulse Sequence: s2pul
1j
Ts
N
O
Ph
220
200
180
160
140
120
100
80
60
40
20
0
ppm

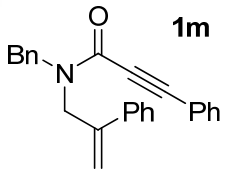

PHS-VIII-27
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385820 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
1k
Bn
N
O
Ph
OH
9
8
7
6
5
4
3
2
1
0 ppm
2.65
11.11
0.46
0.49
2.00
1.04
1.04
0.94
0.96
1.02
1.01


PHS-VIII-27-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
244 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 92 hr, 3 min, 4 sec
1k
Bn
N
O
Ph
OH
180
160
140
120
100
80
60
40
20
ppm

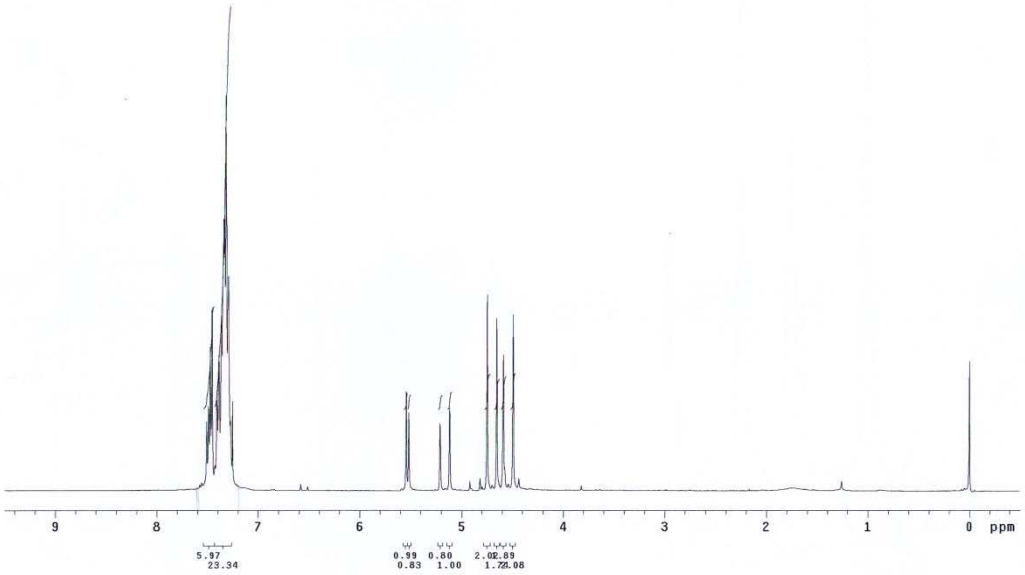

PHS-VIII-16
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8385851 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 min, 51 sec
1l
Bn
N
O
Ph
OTBS
9
8
7
6
5
4
3
2
1
0 ppm
3.62
2.46
2.16
0.94
0.92
1.91
0.96
2.00
1.80
11
1.98
3.19
7.67
8.49
4.54
4.89


PHS-VIII-14-2-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
604 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 92 hr, 3 min, 4 sec
1l
Bn
N
O
Ph
OTBS
180
160
140
120
100
80
60
40
20
0 ppm

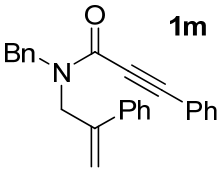
KJY-VI-74-1H
File: xp
Pulse Sequence: s2pul



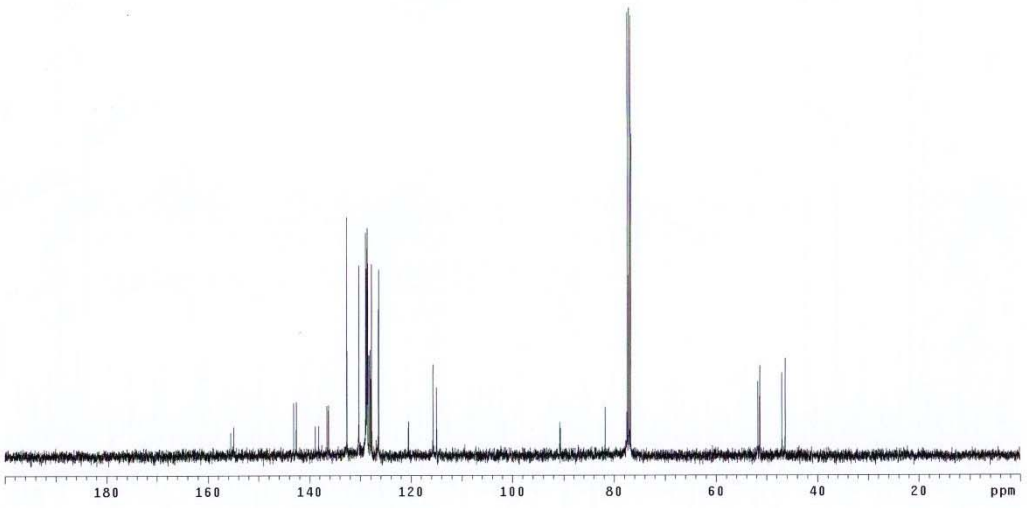
1m

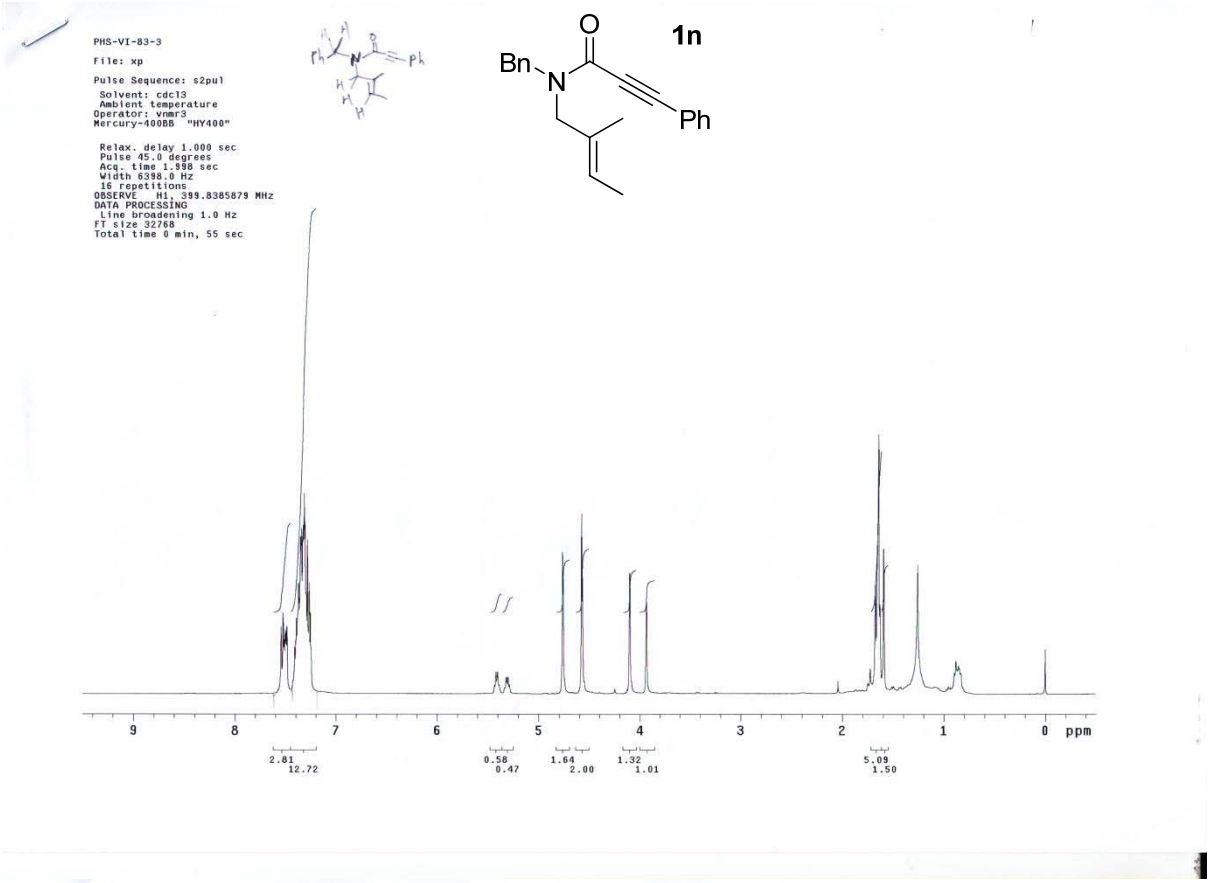


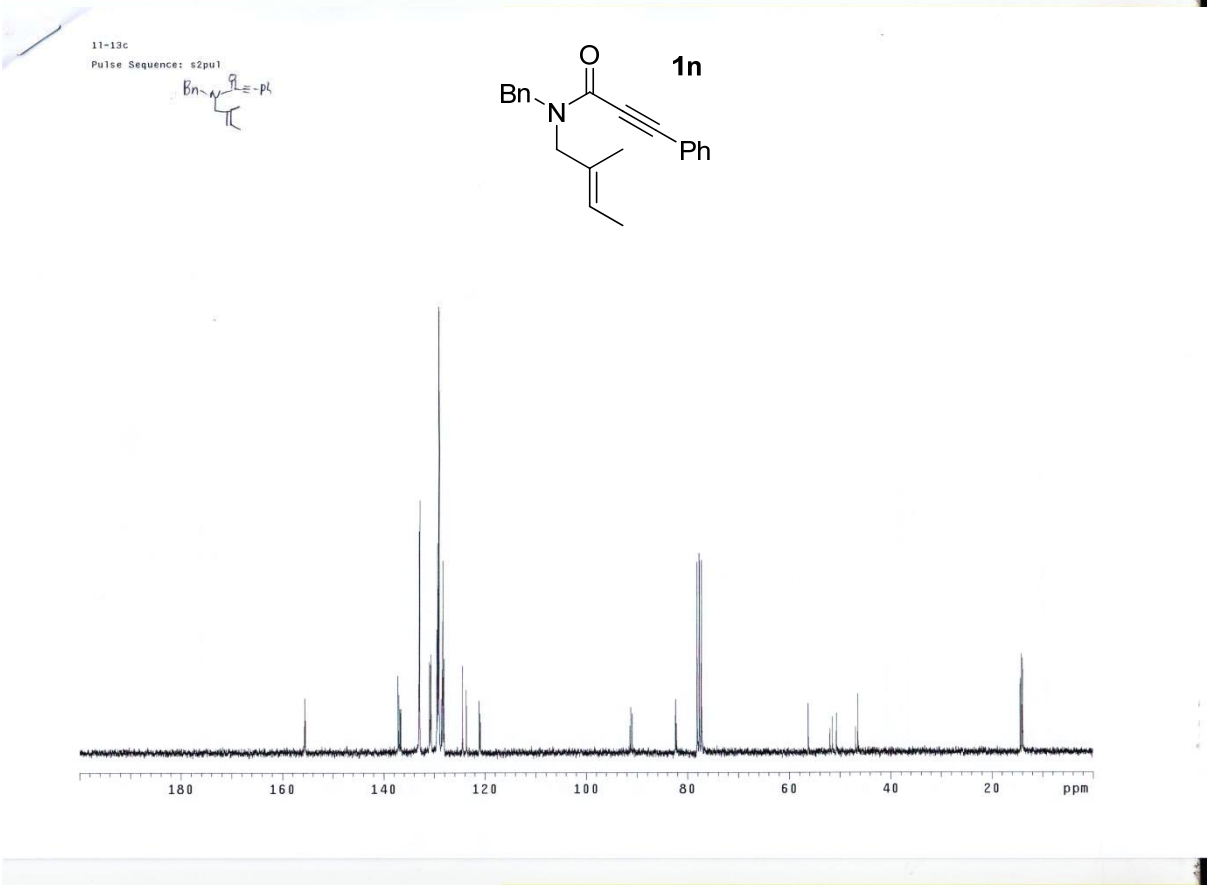
KJY-VI-74-13C
File: xp
Pulse Sequence: s2pul

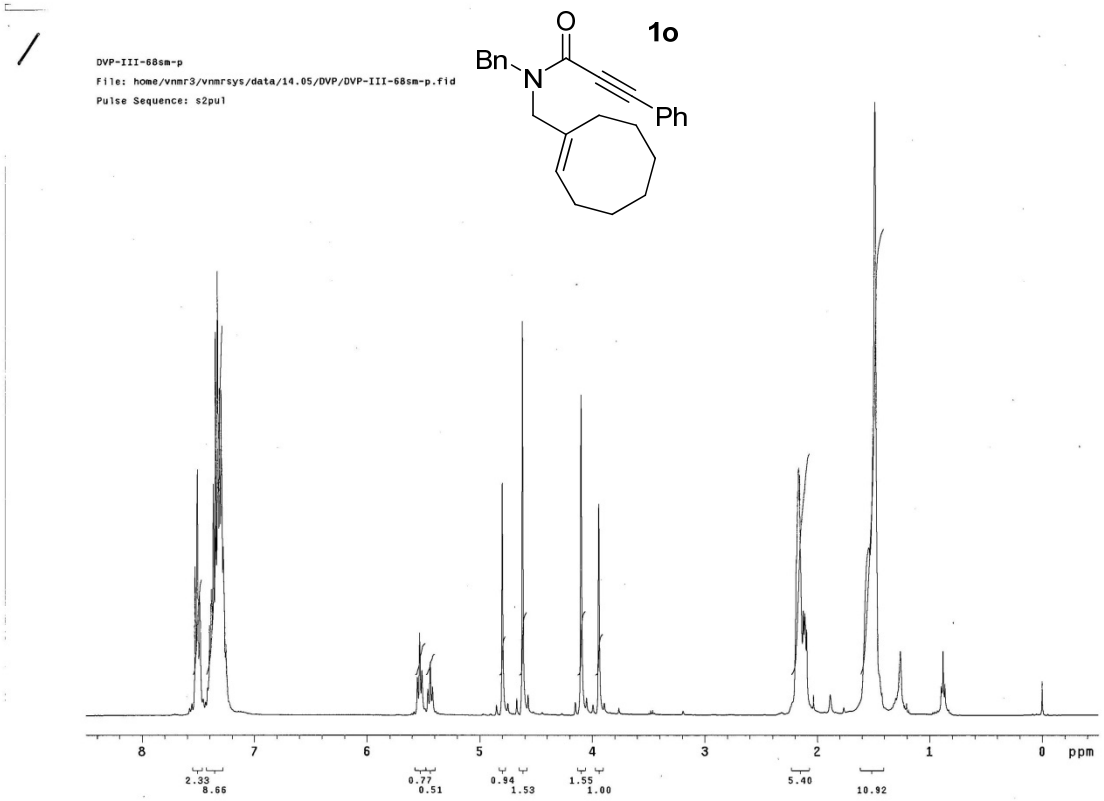


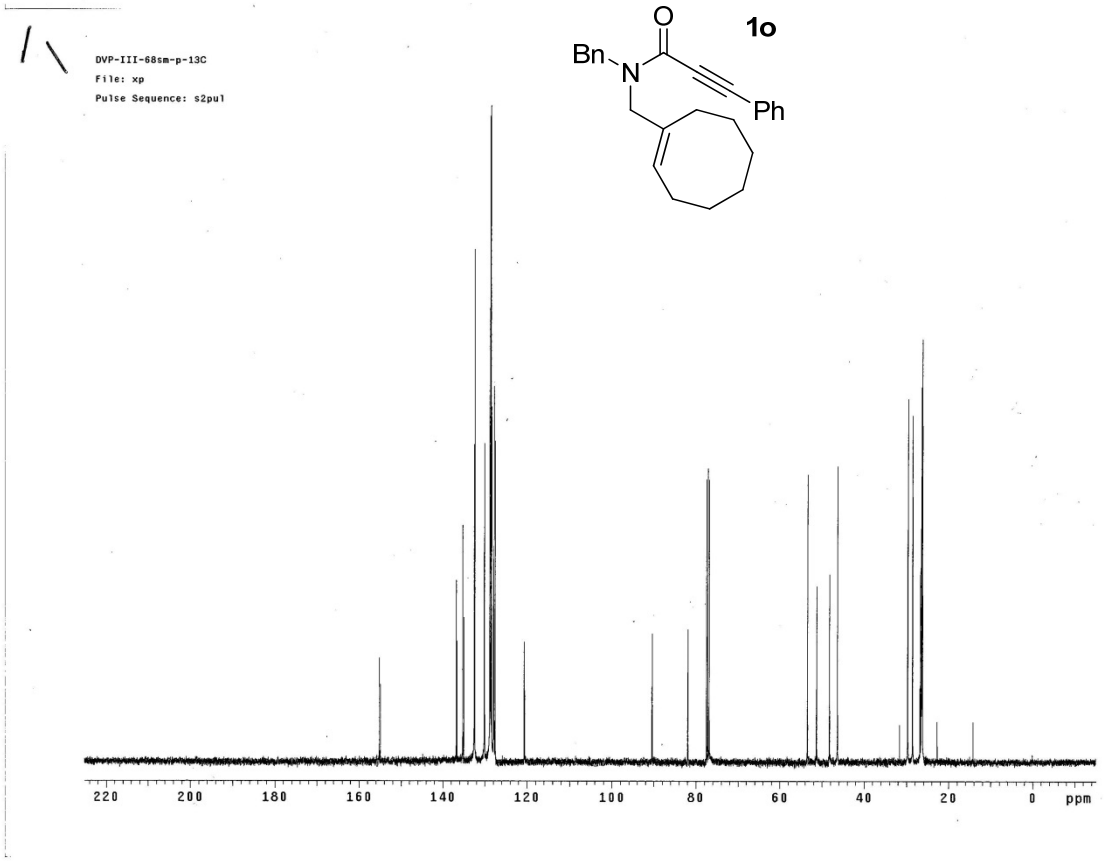
1m

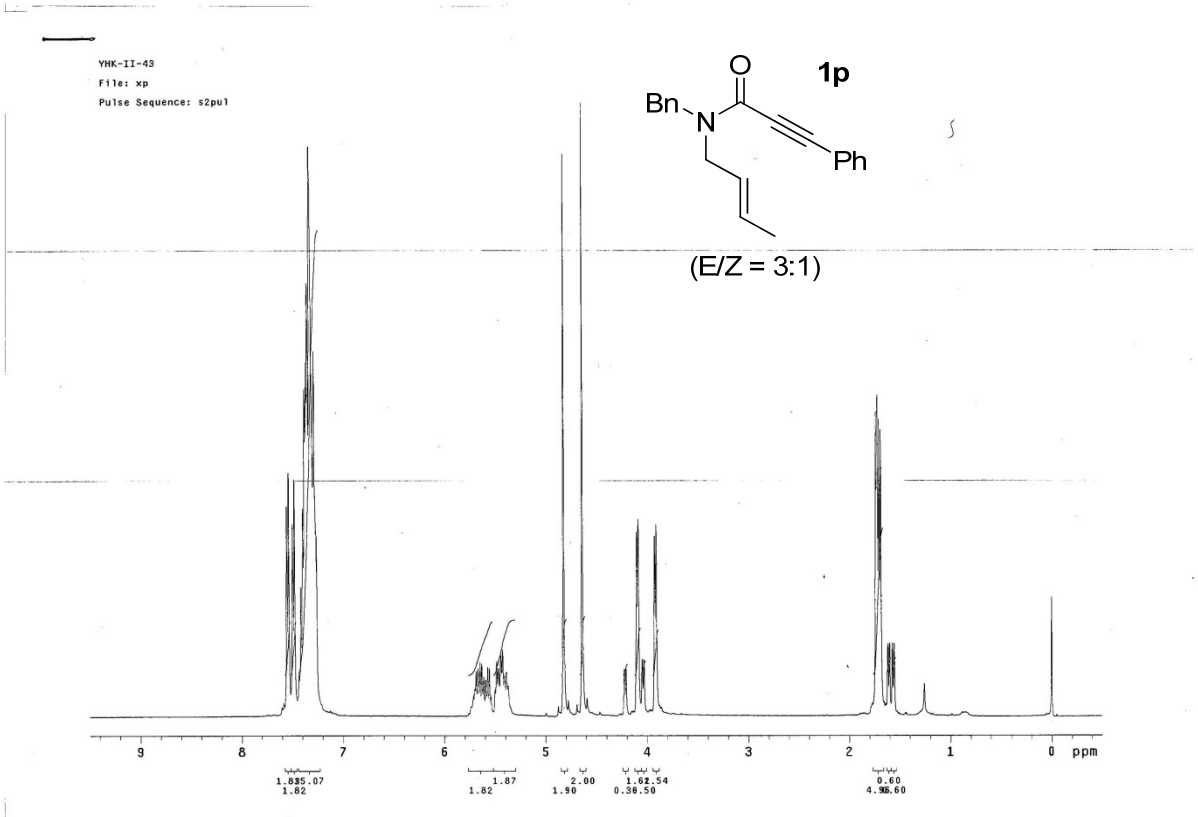


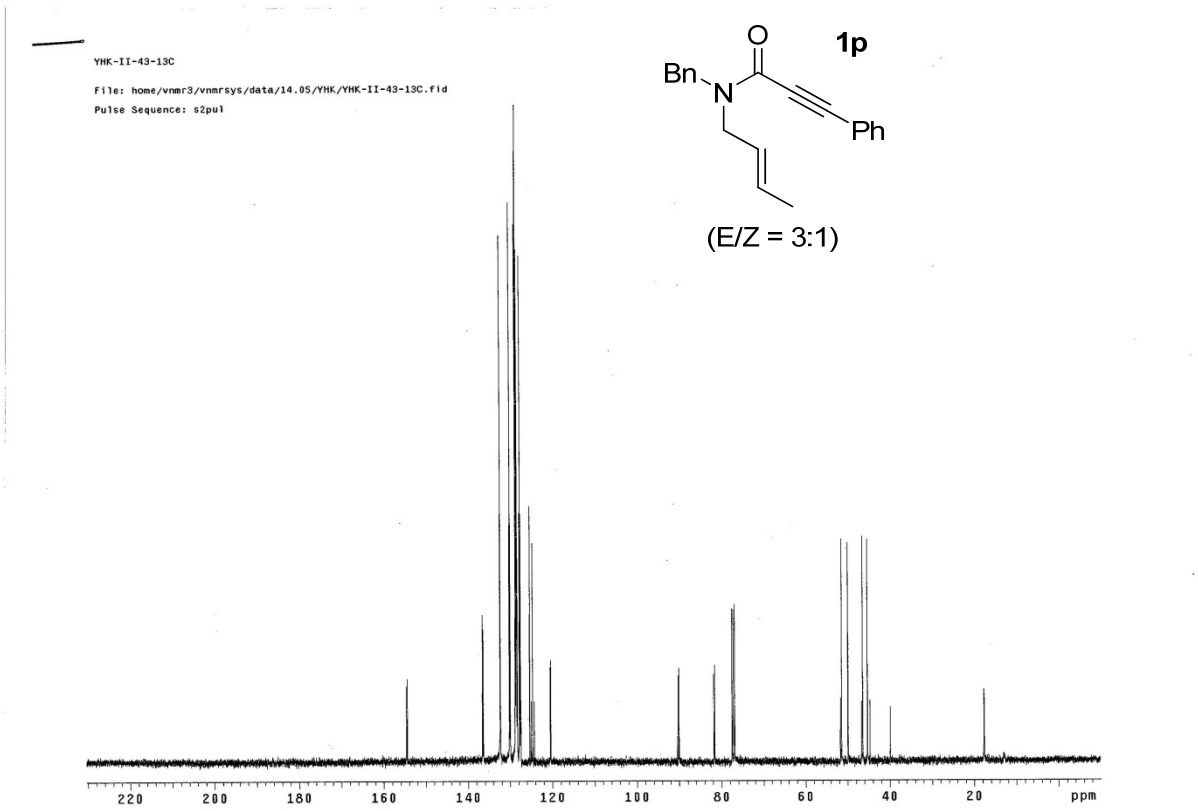

PHS-VI-83-3
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385879 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
1n
Bn
N
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
2.81
12.72
0.58
0.47
1.64
2.00
1.32
1.01
5.09
1.50

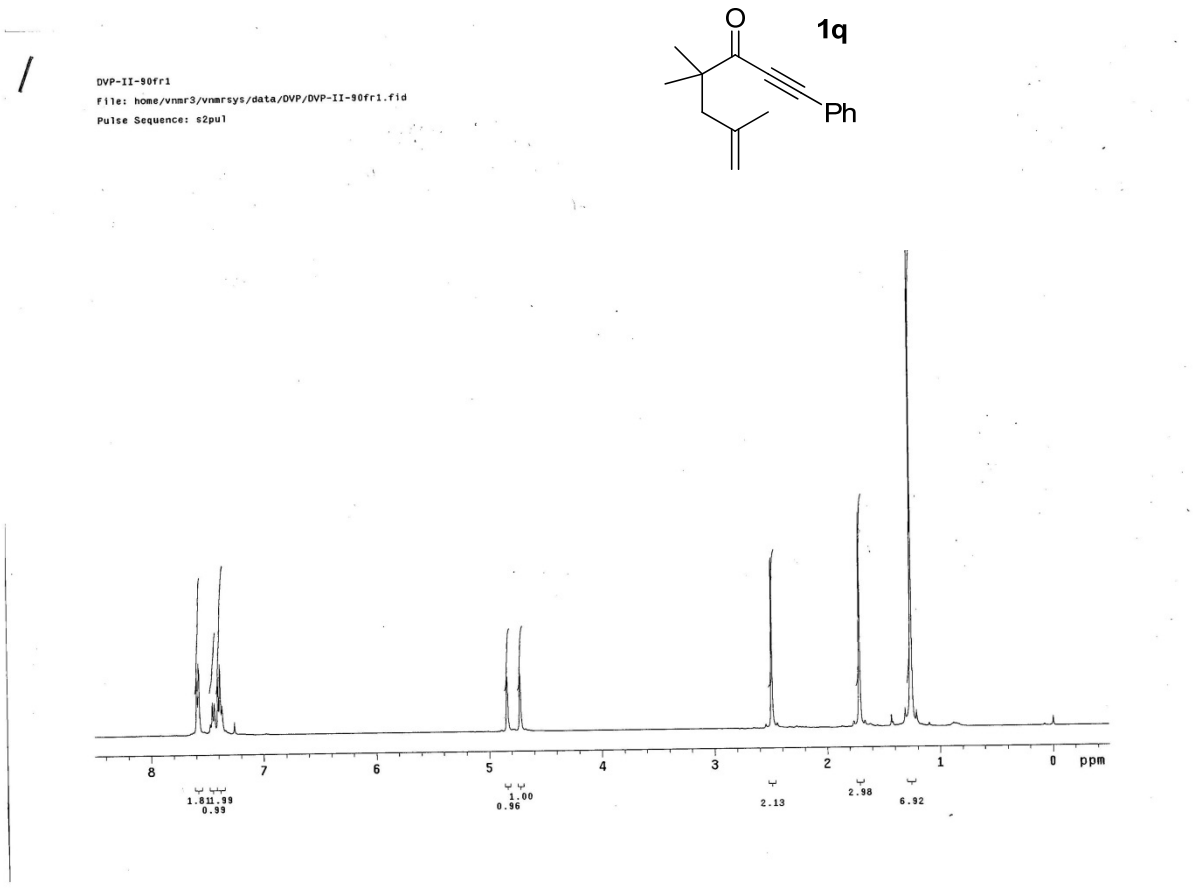

11-13c
Pulse Sequence: s2pul
1n
Bn
N
O
Ph
180
160
140
120
100
80
60
40
20
ppm

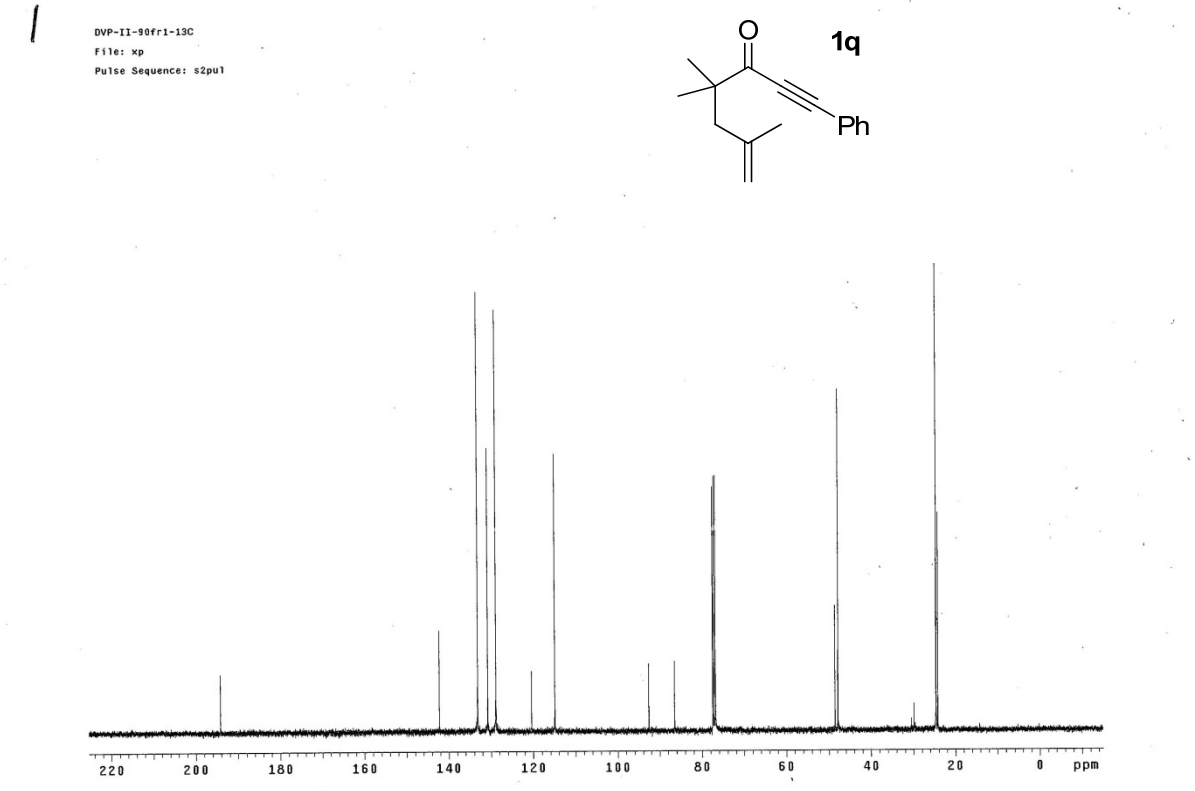

1o
Bn
N
O
Ph
DVP-III-68sm-p
File: home/vnmr3/vnmrsys/data/14.05/DVP/DVP-III-68sm-p.fid
Pulse Sequence: s2pul
8
7
6
5
4
3
2
1
0
ppm
2.33
8.66
0.77
0.51
0.94
1.53
1.55
1.00
5.40
10.92


1o
Bn
N
O
Ph
DVP-III-68sm-p-13C
File: xp
Pulse Sequence: s2pul
220
200
180
160
140
120
100
80
60
40
20
0
ppm

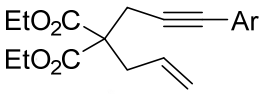

YHK-II-43
File: xp
Pulse Sequence: s2pul
1p
Bn
N
O
Ph
(E/Z = 3:1)
9
8
7
6
5
4
3
2
1
0
ppm


YHK-II-43-13C
File: home/vnmr3/vnmrsys/data/14.05/YHK/YHK-II-43-13C.fid
Pulse Sequence: s2pul
1p
Bn
N
O
Ph
(E/Z = 3:1)
220
200
180
160
140
120
100
80
60
40
20
ppm

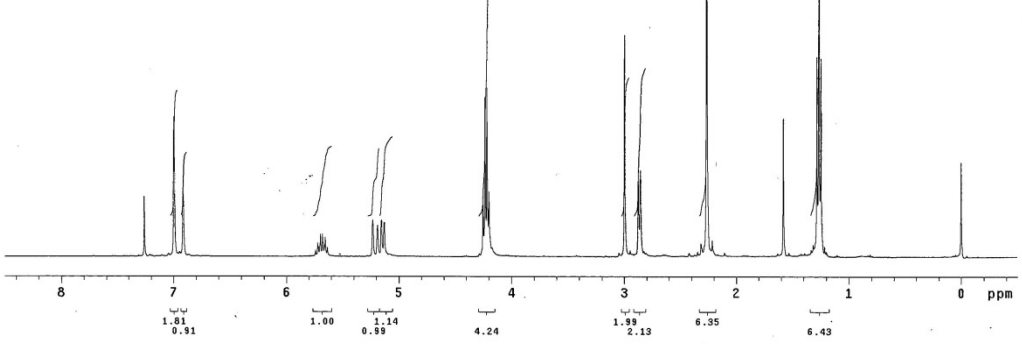

DVP-II-90fr1
File: home/vnmr3/vnmrsys/data/DVP/DVP-II-90fr1.fid
Pulse Sequence: s2pul
O
1q
Ph
8
7
6
5
4
3
2
1
0
ppm
1.81
1.99
0.99
1.00
0.96
2.13
2.98
6.92


DVP-II-90fr1-13C
File: xp
Pulse Sequence: s2pul
O
1q
Ph
220
200
180
160
140
120
100
80
60
40
20
0
ppm

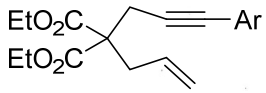
DVP-IV-41
File: xp
Pulse Sequence: s2pul



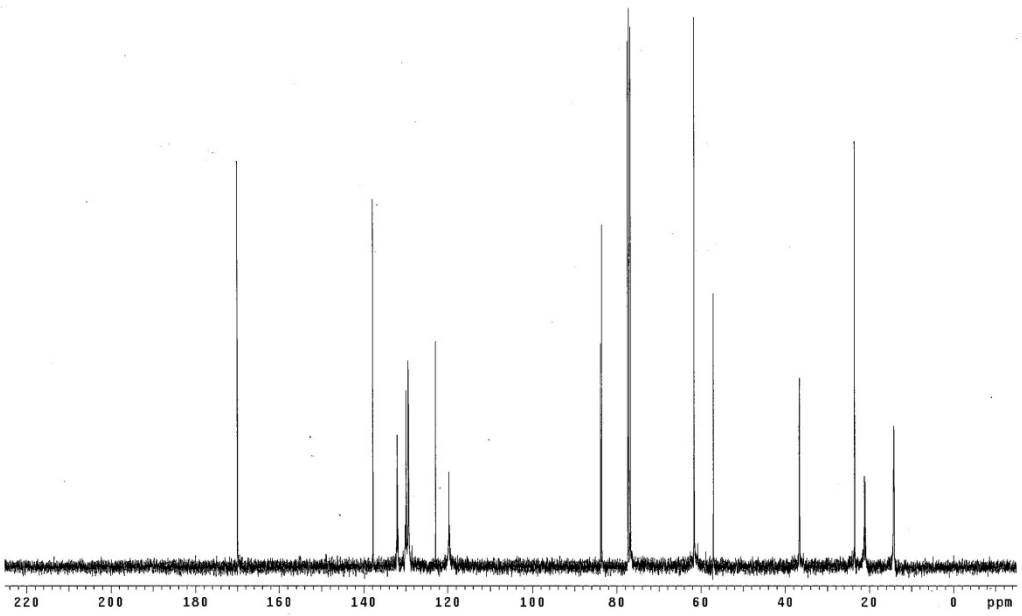
1s (Ar = 3,5-di-Me-C_6H_3)



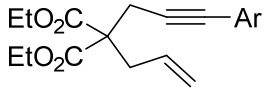
DVP-IV-41-13C
File: home/vnmr3/vnmrsys/data/14.07/DVP/DVP-IV-41-13C.fid
Pulse Sequence: s2pul



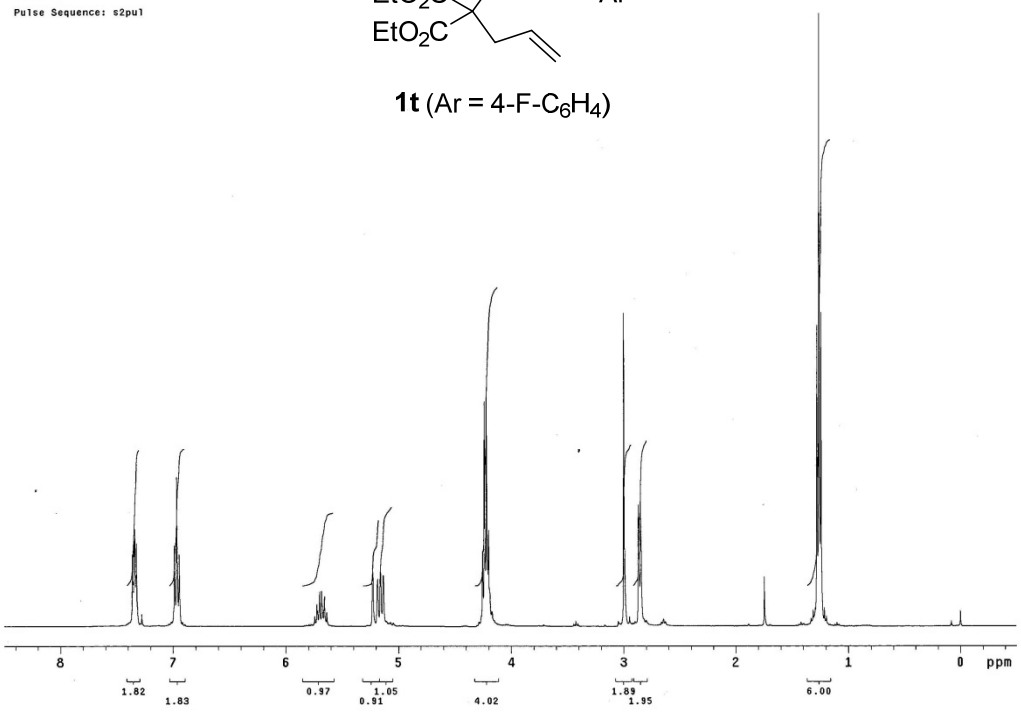
1s (Ar = 3,5-di-Me-C_6H_3)



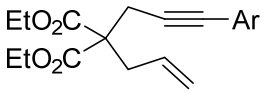
DVP-IV-44
File: xp
Pulse Sequence: s2pul



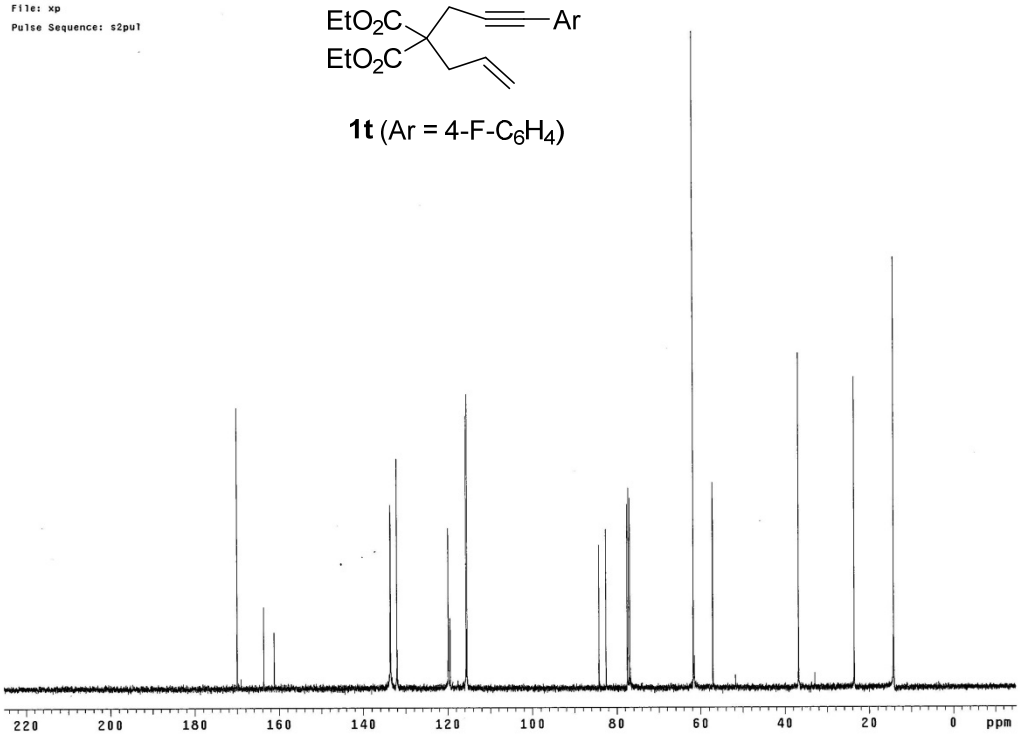
1t (Ar = 4-F-C_6H_4)

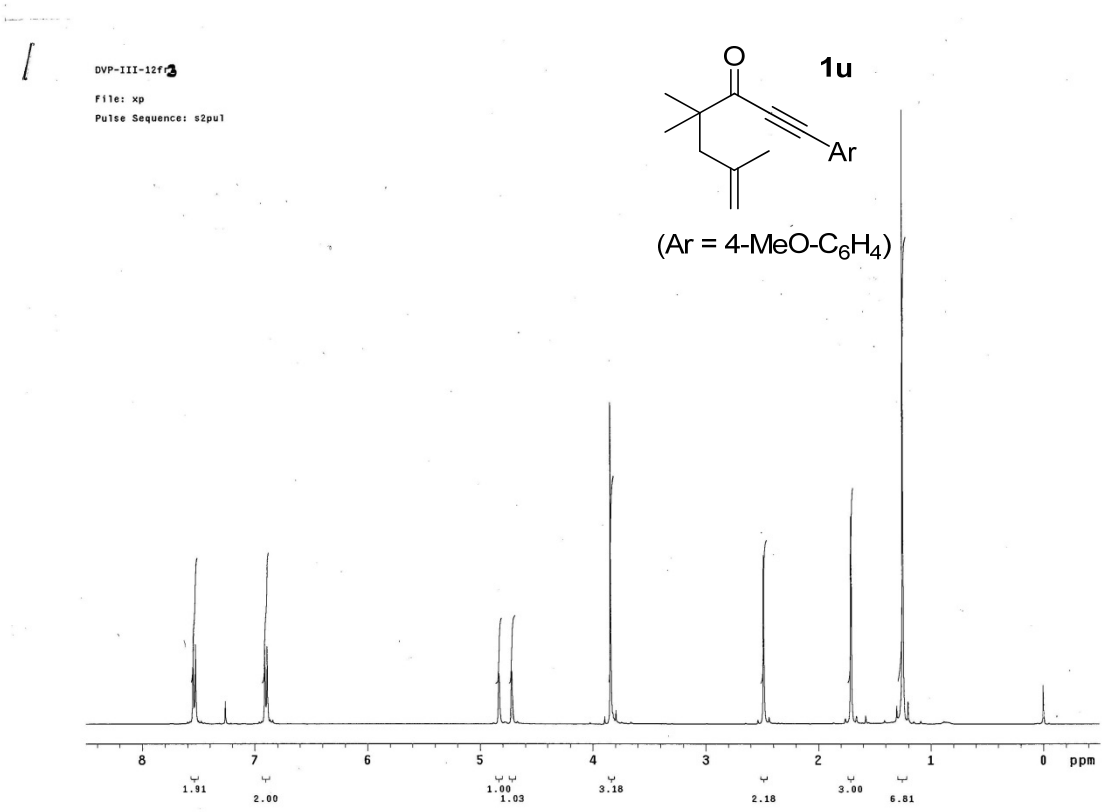


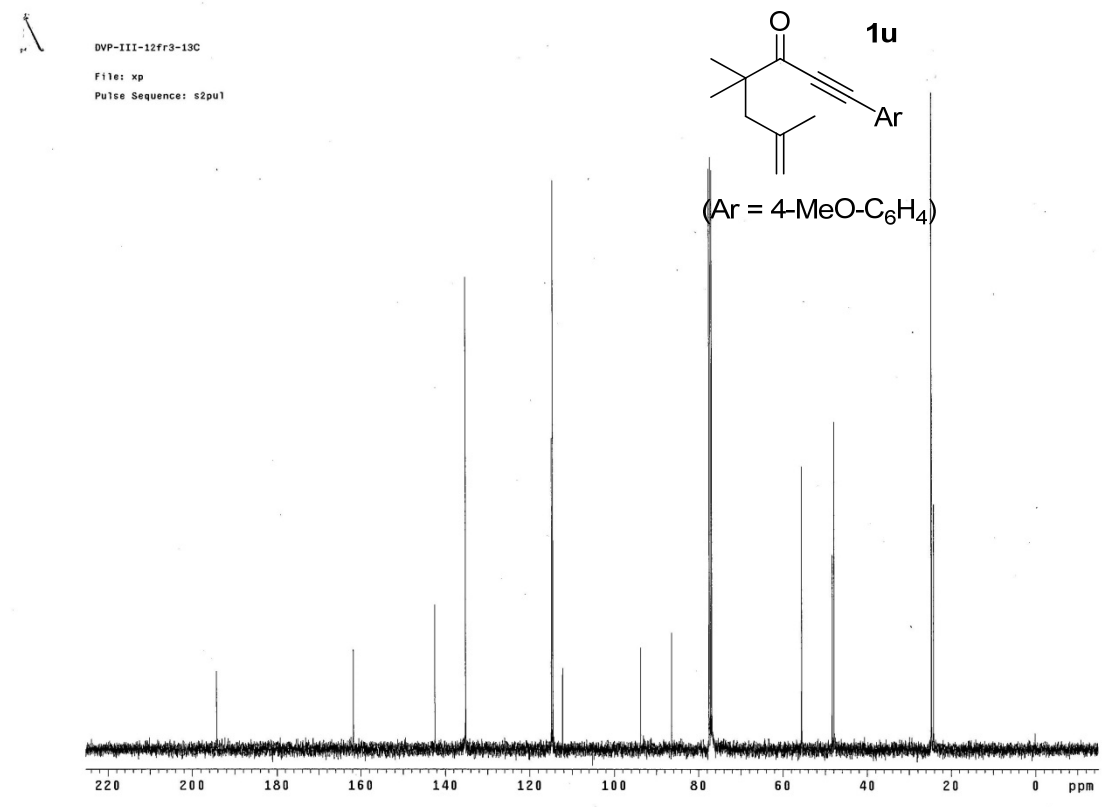
DVP-IV-44-13C
File: xp
Pulse Sequence: s2pul

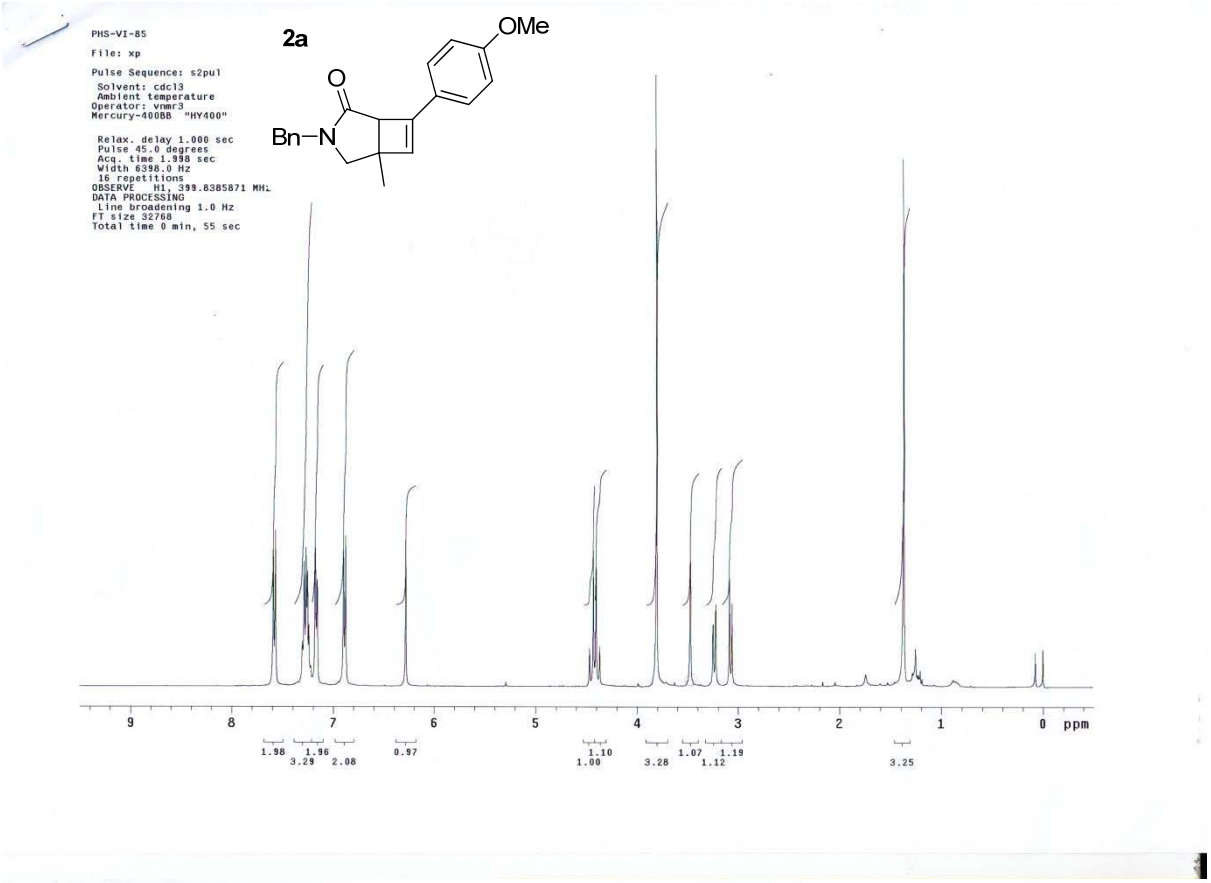


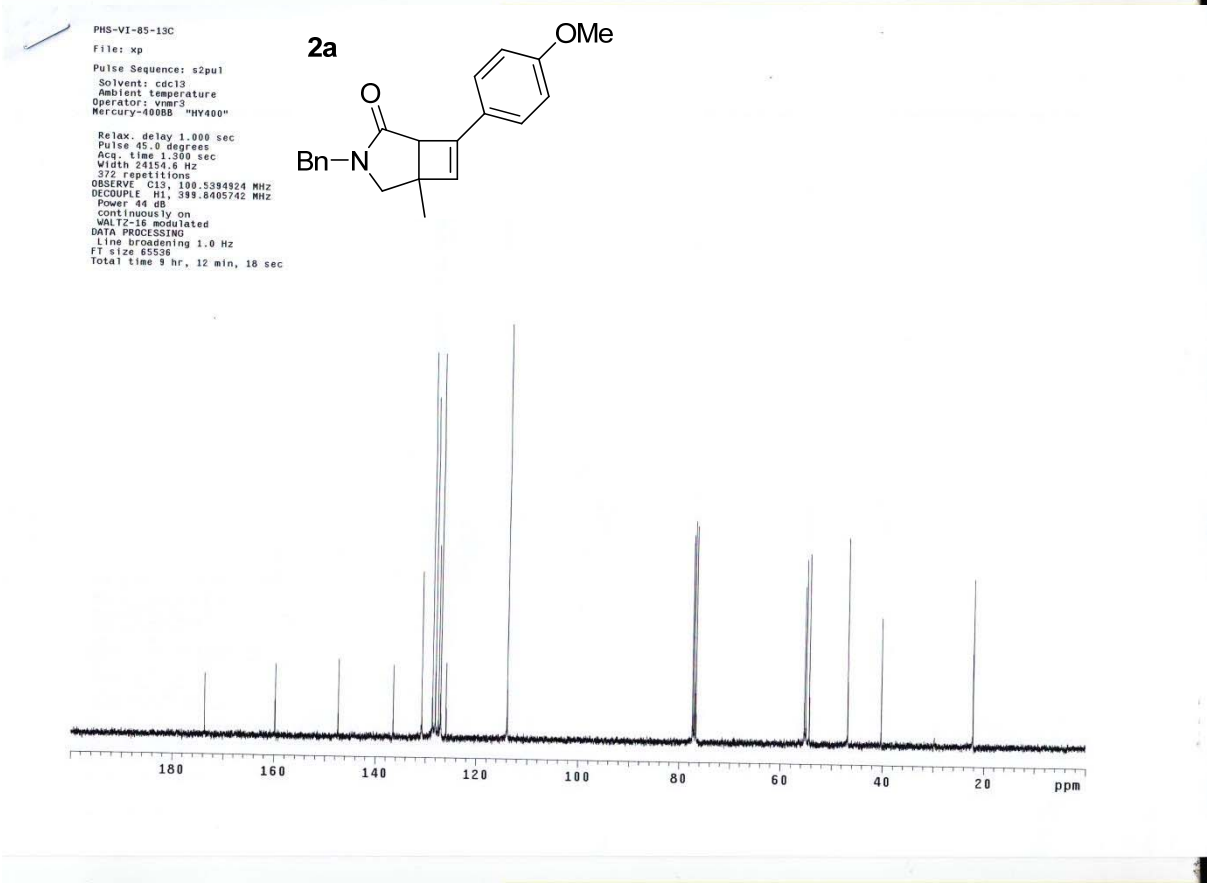
1t (Ar = 4-F-C_6H_4)

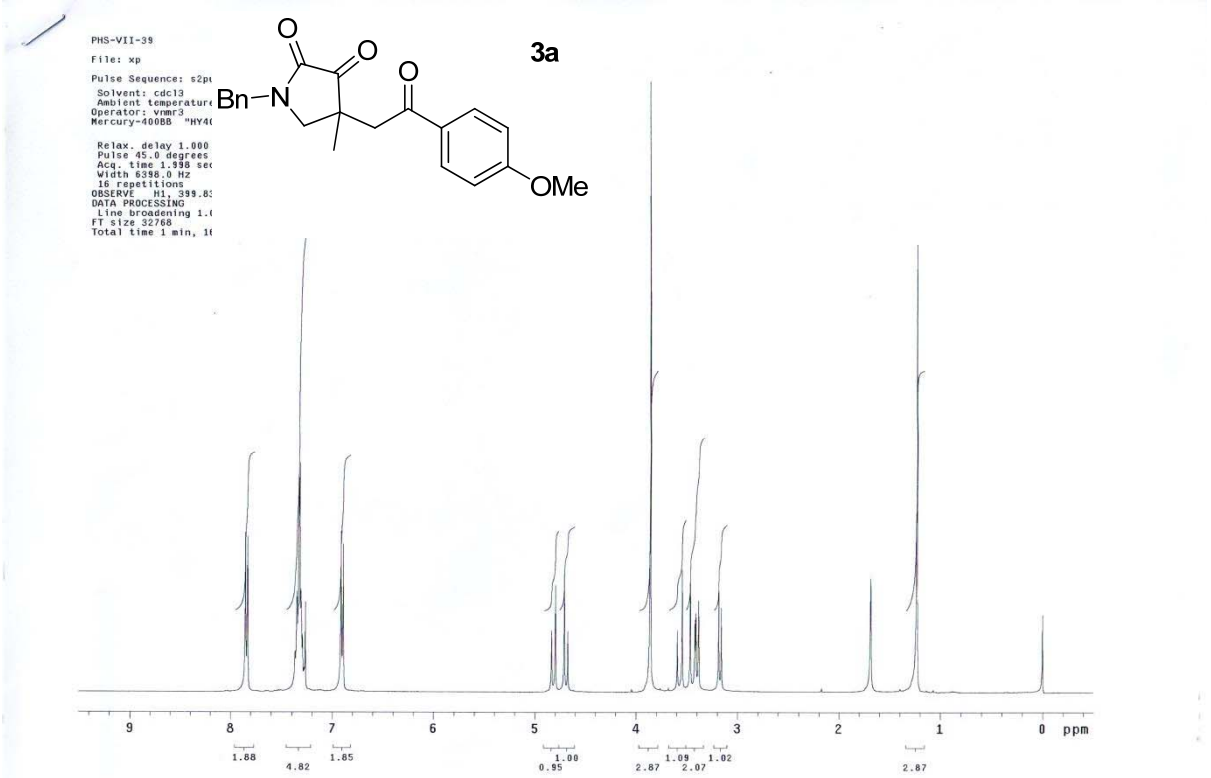


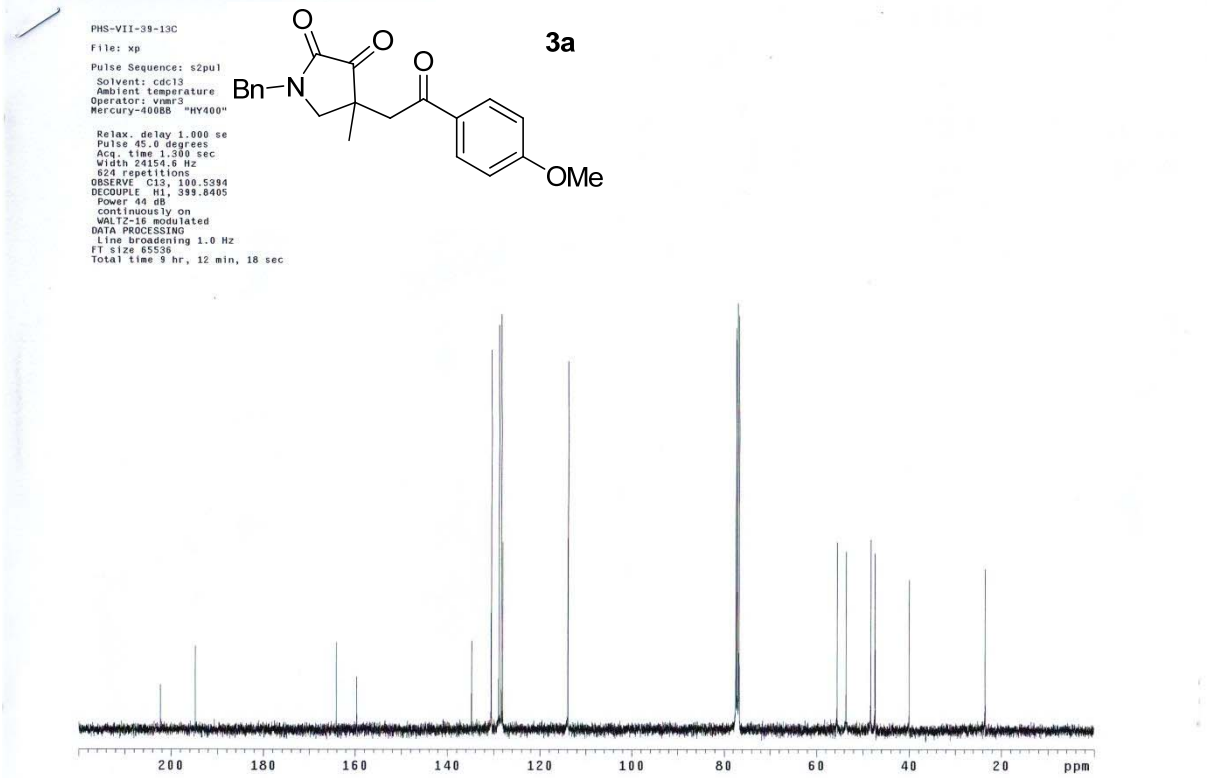

DVP-III-12fr3
File: xp
Pulse Sequence: s2pul
O
1u
Ar
(Ar = 4-MeO-C6H4)
8
7
6
5
4
3
2
1
0
ppm
1.91
2.00
1.00
1.03
3.18
2.18
3.00
6.81

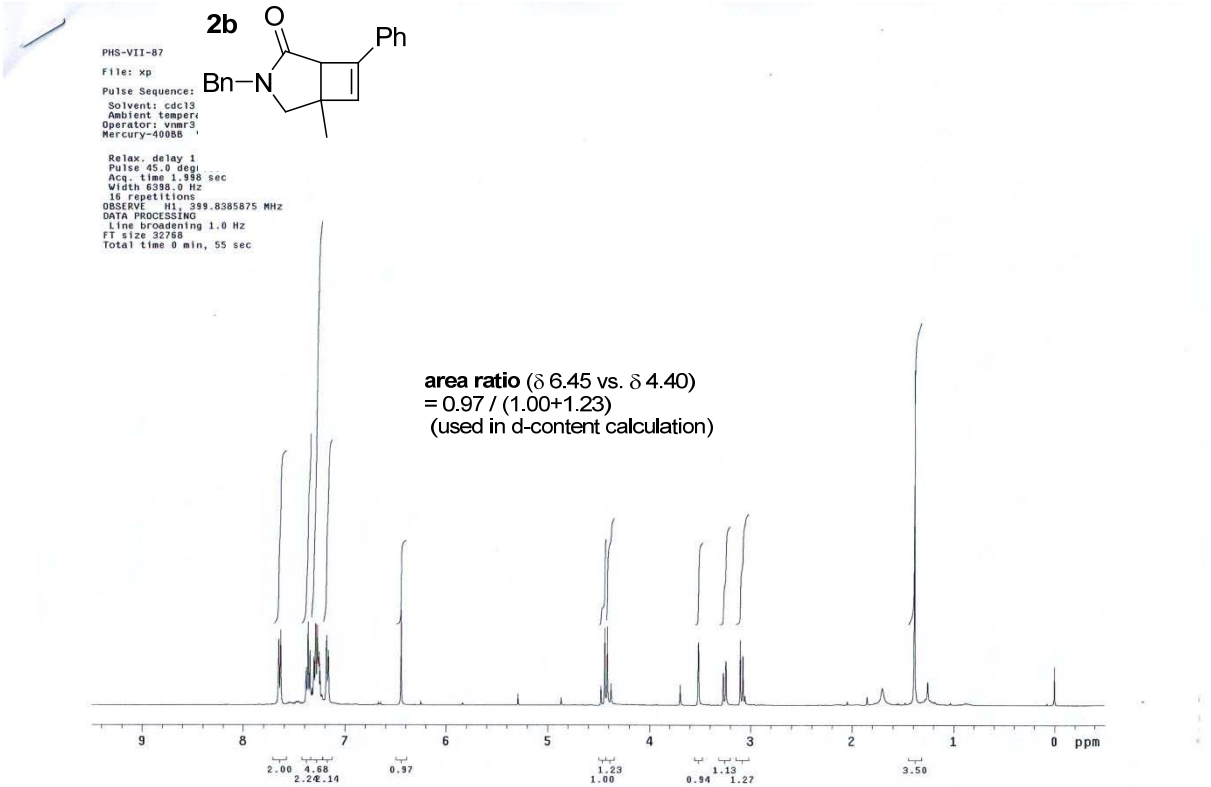

DVP-III-12fr3-13C
File: xp
Pulse Sequence: s2pul
O
1u
Ar
(Ar = 4-MeO-C6H4)
220
200
180
160
140
120
100
80
60
40
20
0
ppm

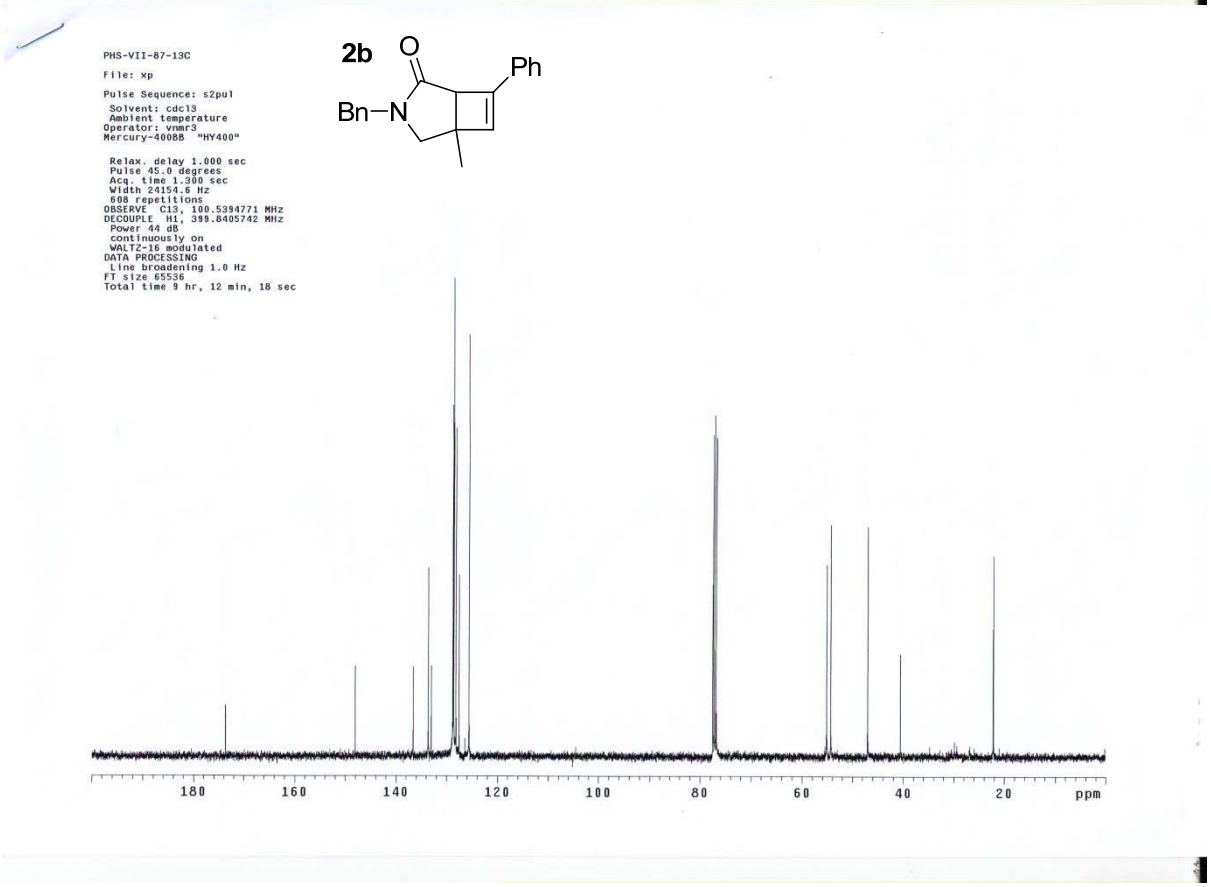

PHS-VI-85
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385871 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
2a
OMe
O
Bn–N
9
8
7
6
5
4
3
2
1
0 ppm
1.98
1.96
3.29
2.08
0.97
1.10
1.00
3.28
1.07
1.19
1.12
3.25

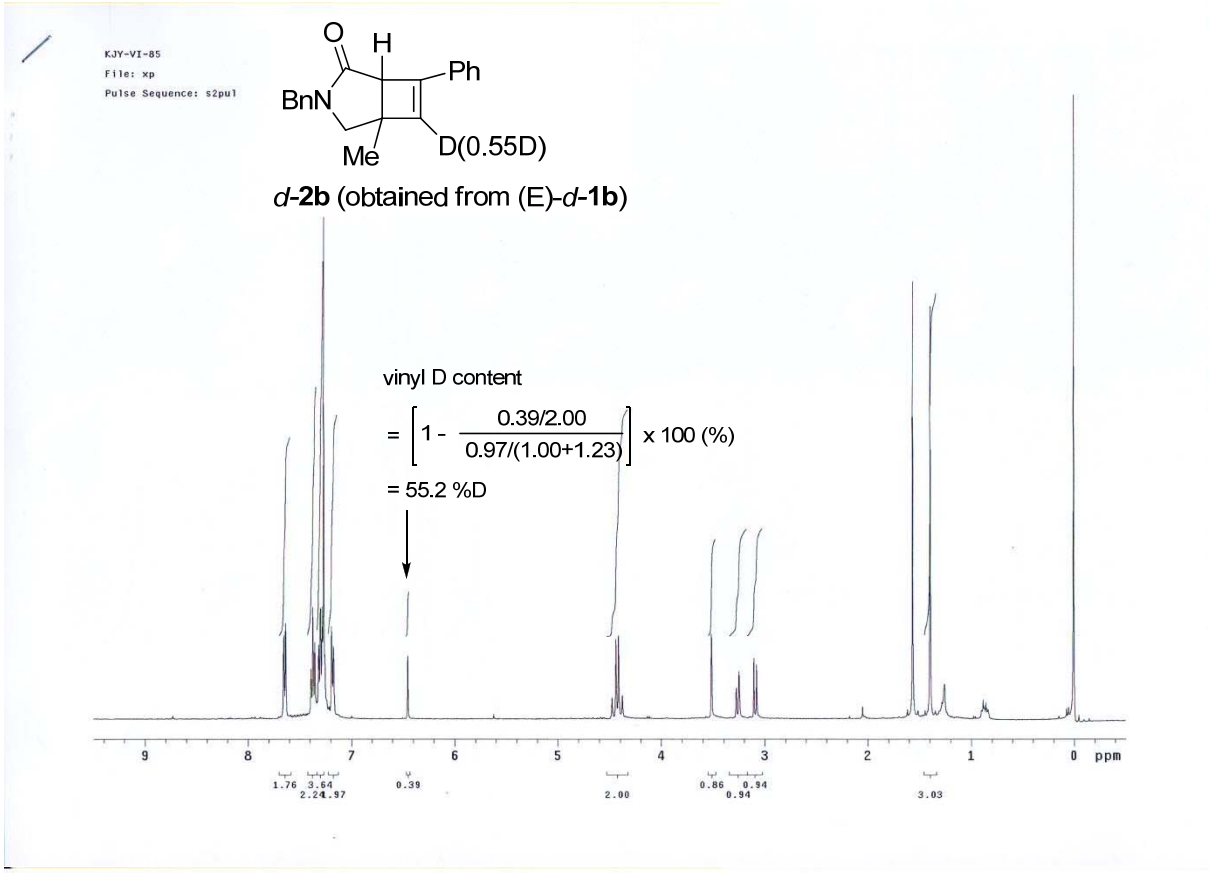

PHS-VI-85-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
372 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
2a
OMe
O
Bn–N
180
160
140
120
100
80
60
40
20
ppm

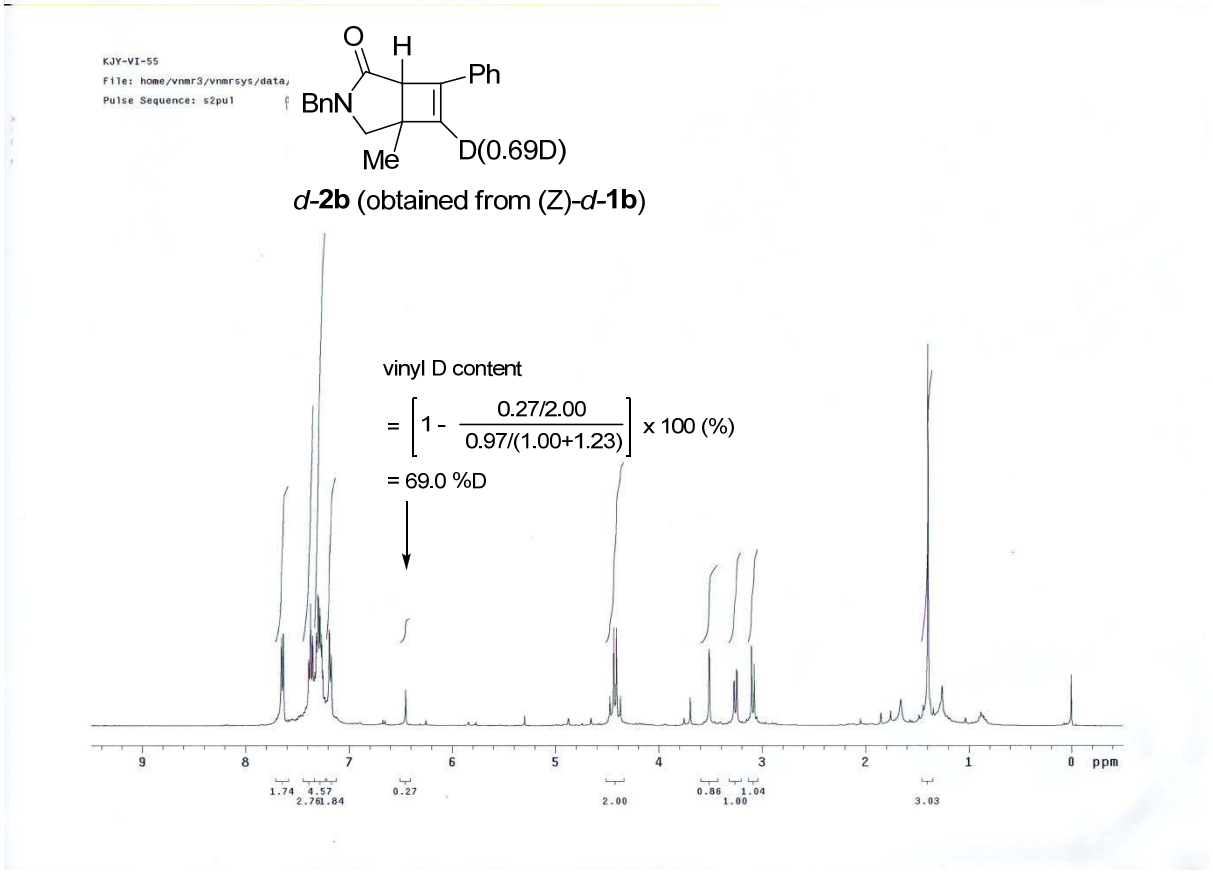

PHS-VII-39
File: xp
Pulse Sequence: s2pu
Solvent: cdcl3
Ambient temperatur
Operator: vnmr3
Mercury-400BB "HY4
Relax. delay 1.000
Pulse 45.0 degrees
Acq. time 1.998 se
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8
DATA PROCESSING
Line broadening 1.
FT size 32768
Total time 1 min, 1
3a
Bn
N
O
O
O
OMe
9
8
7
6
5
4
3
2
1
0
ppm
1.88
4.82
1.85
0.95
1.00
2.87
2.07
1.09
1.02
2.87

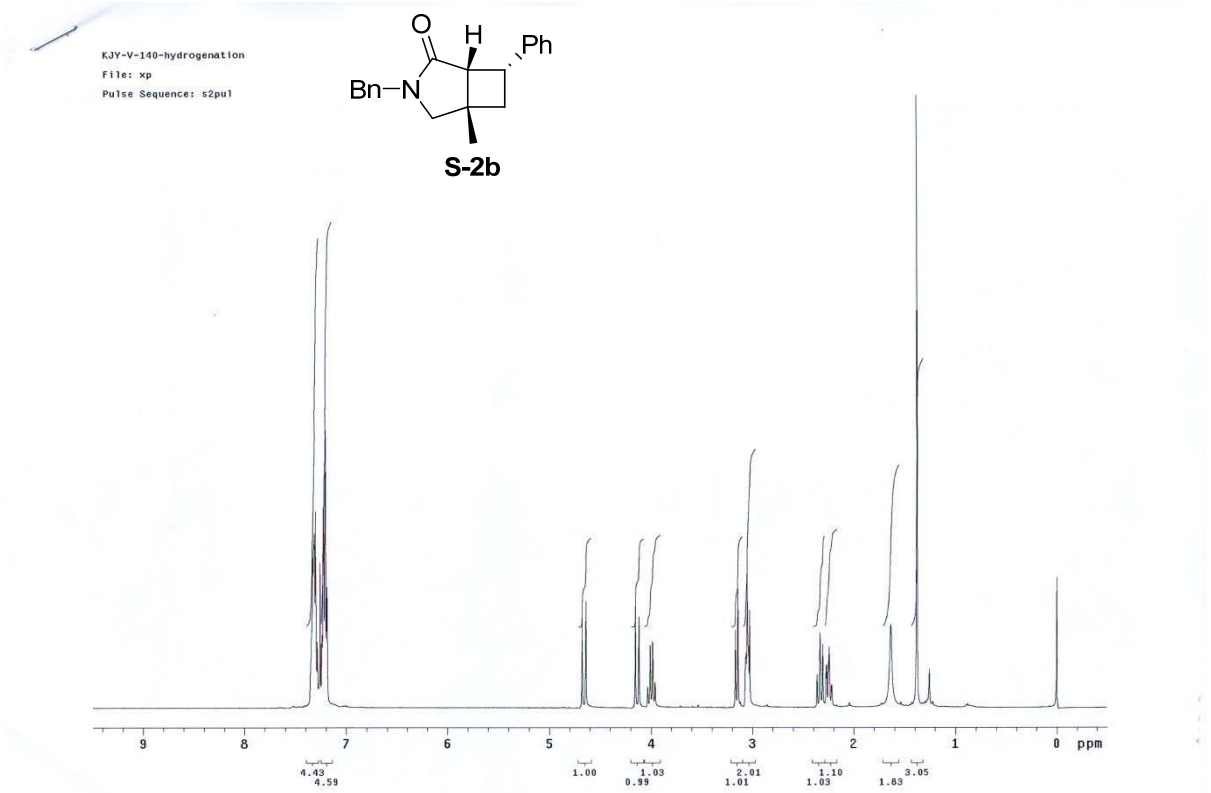

PHS-VII-39-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 se
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
624 repetitions
OBSERVE C13, 100.5394
DECOUPLE H1, 399.8405
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
3a
Bn
N
O
O
O
OMe
200
180
160
140
120
100
80
60
40
20
ppm

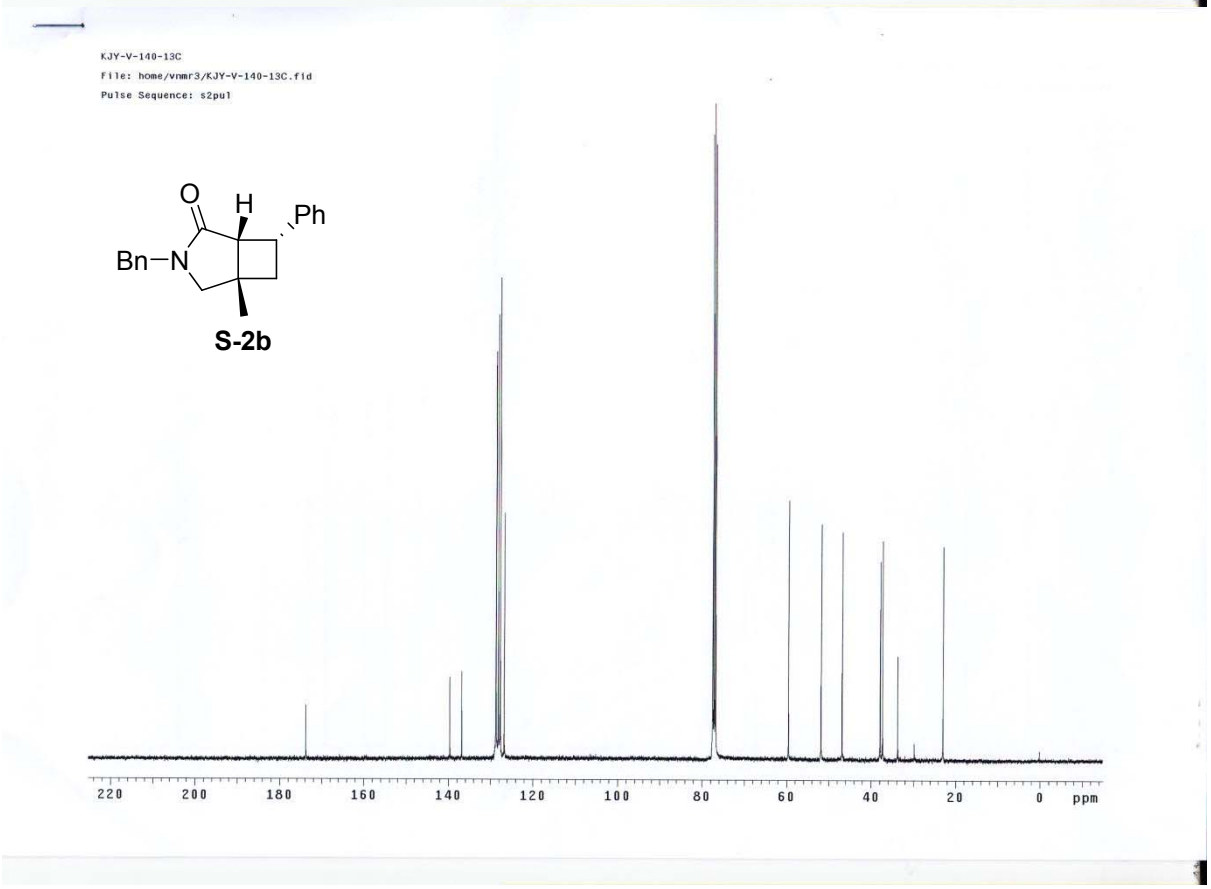

2b
O
Ph
Bn—N
PHS-VII-87
File: xp
Pulse Sequence:
Solvent: cdcl3
Ambient temper
Operator: vnmr3
Mercury-400BB
Relax. delay 1
Pulse 45.0 deg
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385875 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
area ratio (δ 6.45 vs. δ 4.40)
= 0.97 / (1.00+1.23)
(used in d-content calculation)
9
8
7
6
5
4
3
2
1
0 ppm
2.00
4.68
2.24
2.14
0.97
1.23
1.00
0.94
1.13
1.27
3.50

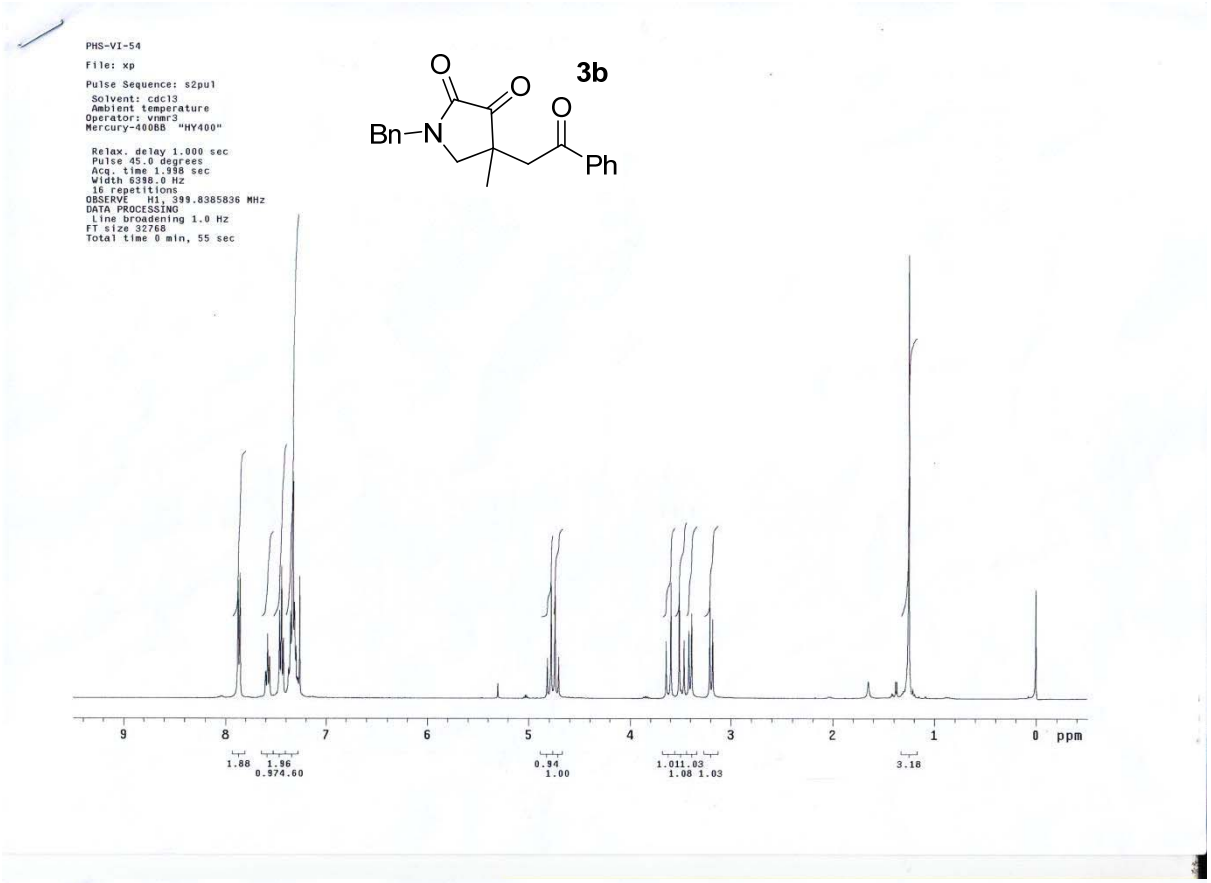

2b
O
Ph
Bn—N
PHS-VII-87-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
608 repetitions
OBSERVE C13, 100.5394771 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
180
160
140
120
100
80
60
40
20
ppm

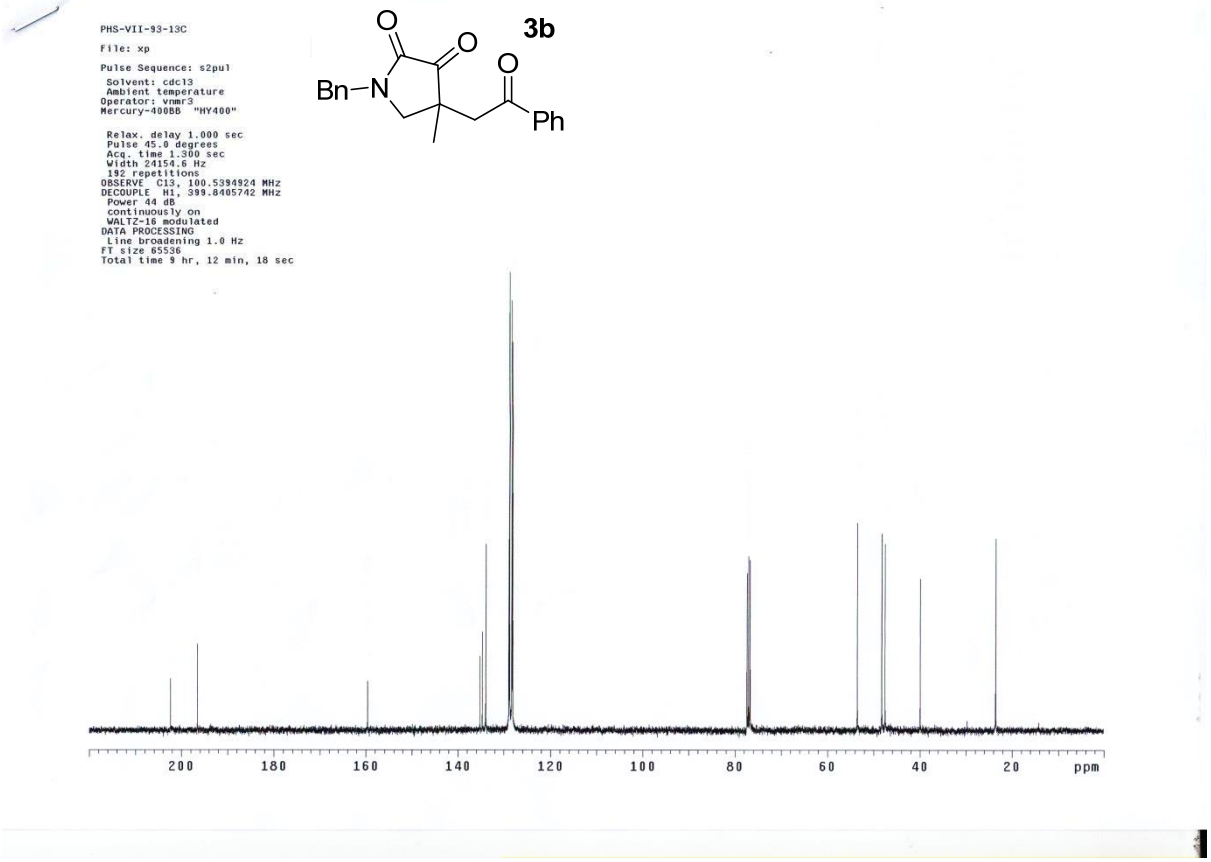

KJY-VI-85
File: xp
Pulse Sequence: s2pul
O
H
Ph
BnN
Me
D(0.55D)
d-2b (obtained from (E)-d-1b)
vinyl D content
= [1 - 0.39/2.00 / 0.97/(1.00+1.23)] x 100 (%)
= 55.2 %D
9 8 7 6 5 4 3 2 1 0 ppm
1.76 3.64 2.24 1.97 0.39 2.00 0.86 0.94 0.94 3.03


KJY-VI-55
File: home/vnmr3/vnmrsys/data/
Pulse Sequence: s2pul
O
H
Ph
BnN
Me
D(0.69D)
d-2b (obtained from (Z)-d-1b)
vinyl D content
= [1 - 0.27/2.00 / 0.97/(1.00+1.23)] x 100 (%)
= 69.0 %D
9 8 7 6 5 4 3 2 1 0 ppm
1.74 4.57 2.78 1.84 0.27 2.00 0.86 1.04 1.00 3.03

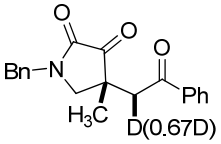

KJY-V-140-hydrogenation
File: xp
Pulse Sequence: s2pul
O
H
Ph
Bn—N
S-2b
9
8
7
6
5
4
3
2
1
0 ppm
4.43
4.59
1.00
1.03
0.99
2.01
1.01
1.10
1.03
1.83
3.05


KJY-V-140-13C
File: home/vnmr3/KJY-V-140-13C.fid
Pulse Sequence: s2pul
O
H
Ph
Bn—N
S-2b
220
200
180
160
140
120
100
80
60
40
20
0 ppm


PHS-VI-54
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385836 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
3b
Bn
N
O
O
O
Ph
9
8
7
6
5
4
3
2
1
0 ppm
1.88
1.96
0.974.60
0.94
1.00
1.011.03
1.08 1.03
3.18

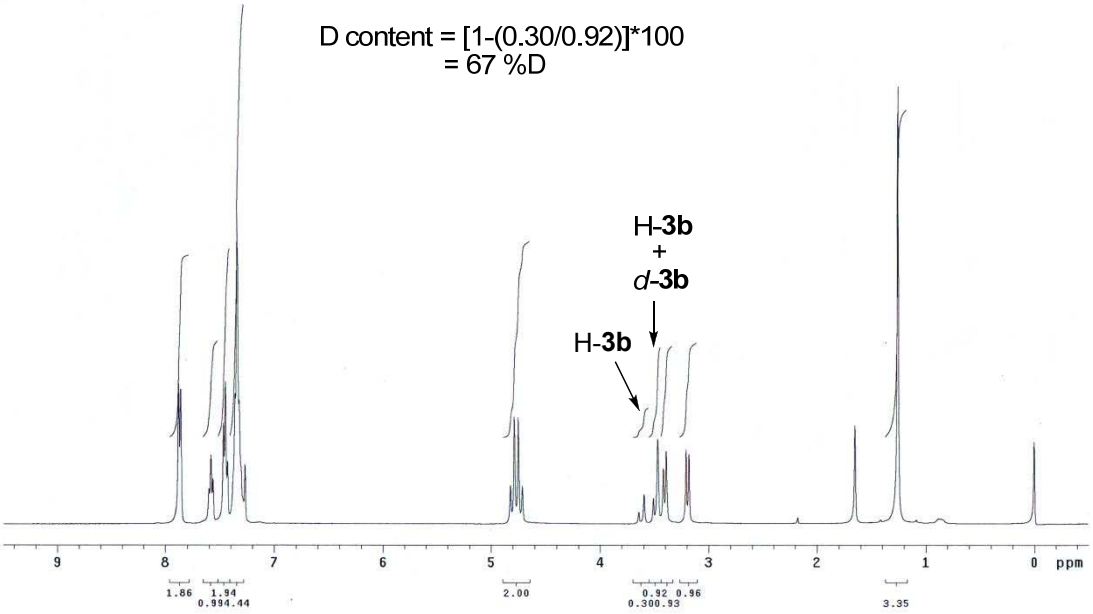

PHS-VII-93-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
192 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 12 min, 18 sec
3b
Bn
N
O
O
O
Ph
200
180
160
140
120
100
80
60
40
20
ppm

KJY-VI-33-recryst
File: xp
Pulse Sequence: s2pul

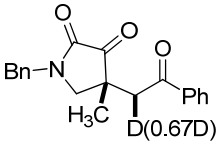


d-**3b** (Eq 3)

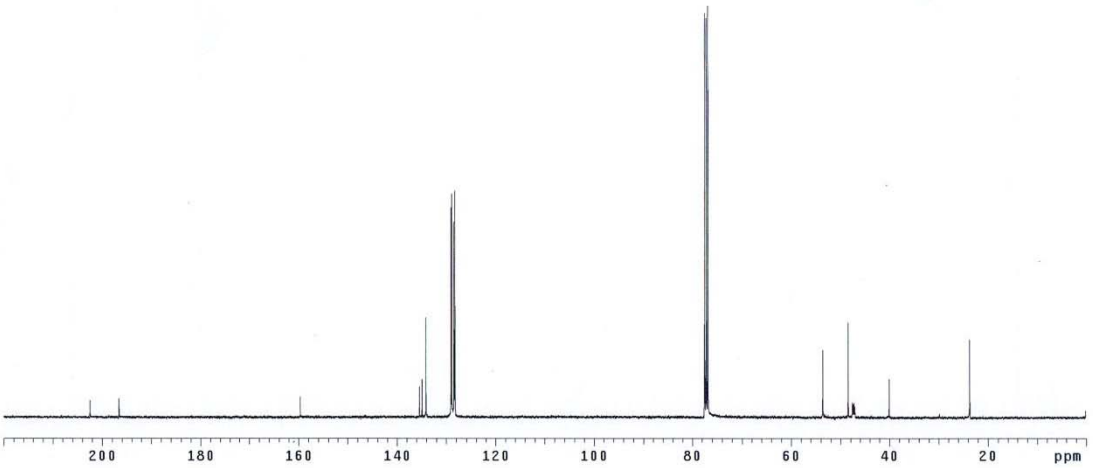
D content = [1-(0.30/0.92)]*100
= 67 %D

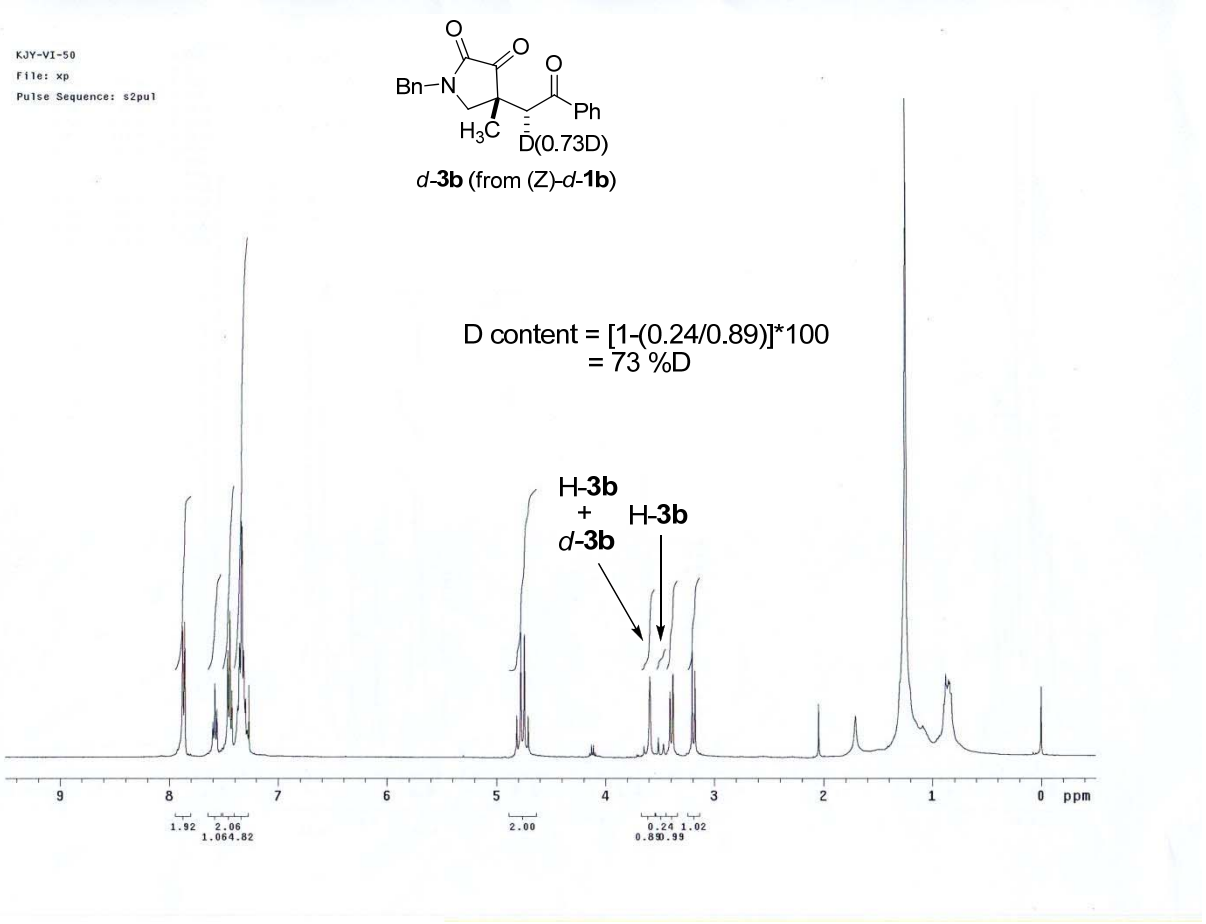


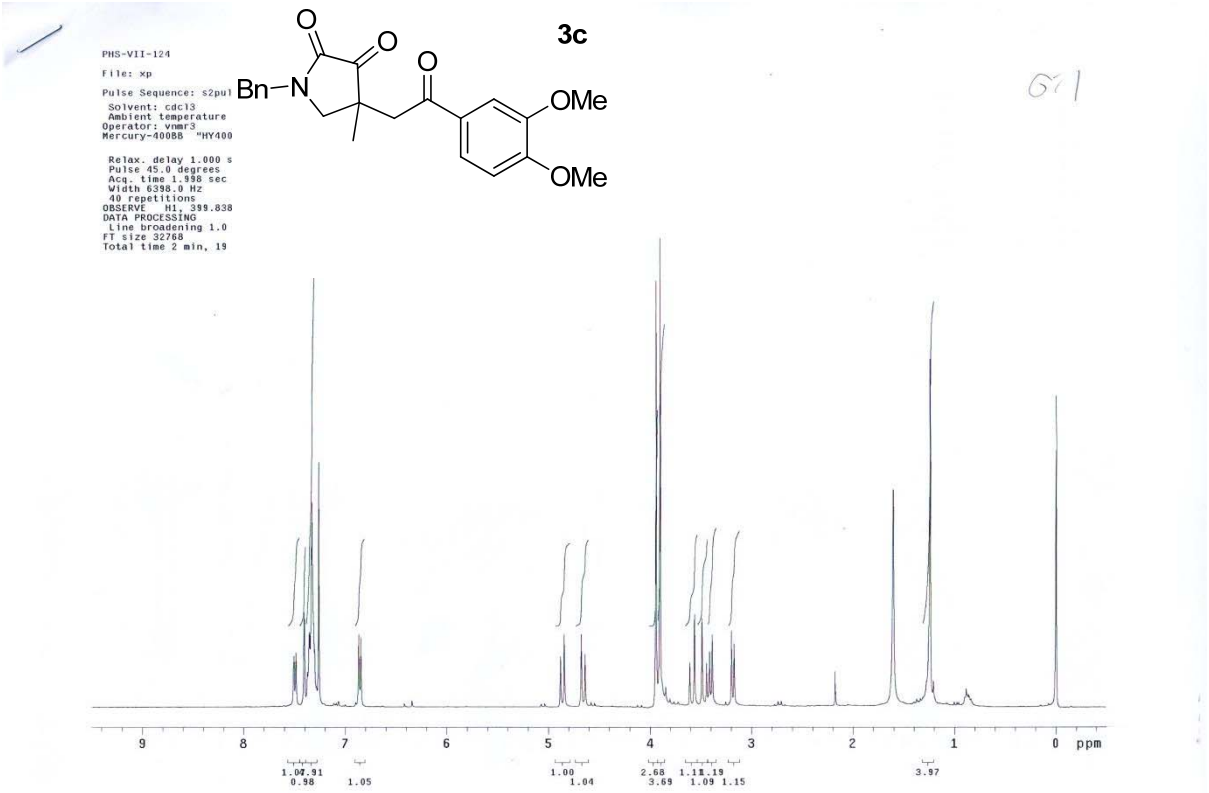
Std Carbon experiment
File: home/vnmr3/vnmrsys/data/KJY/KJY-VI-33-13C.fid
Pulse Sequence: s2pul

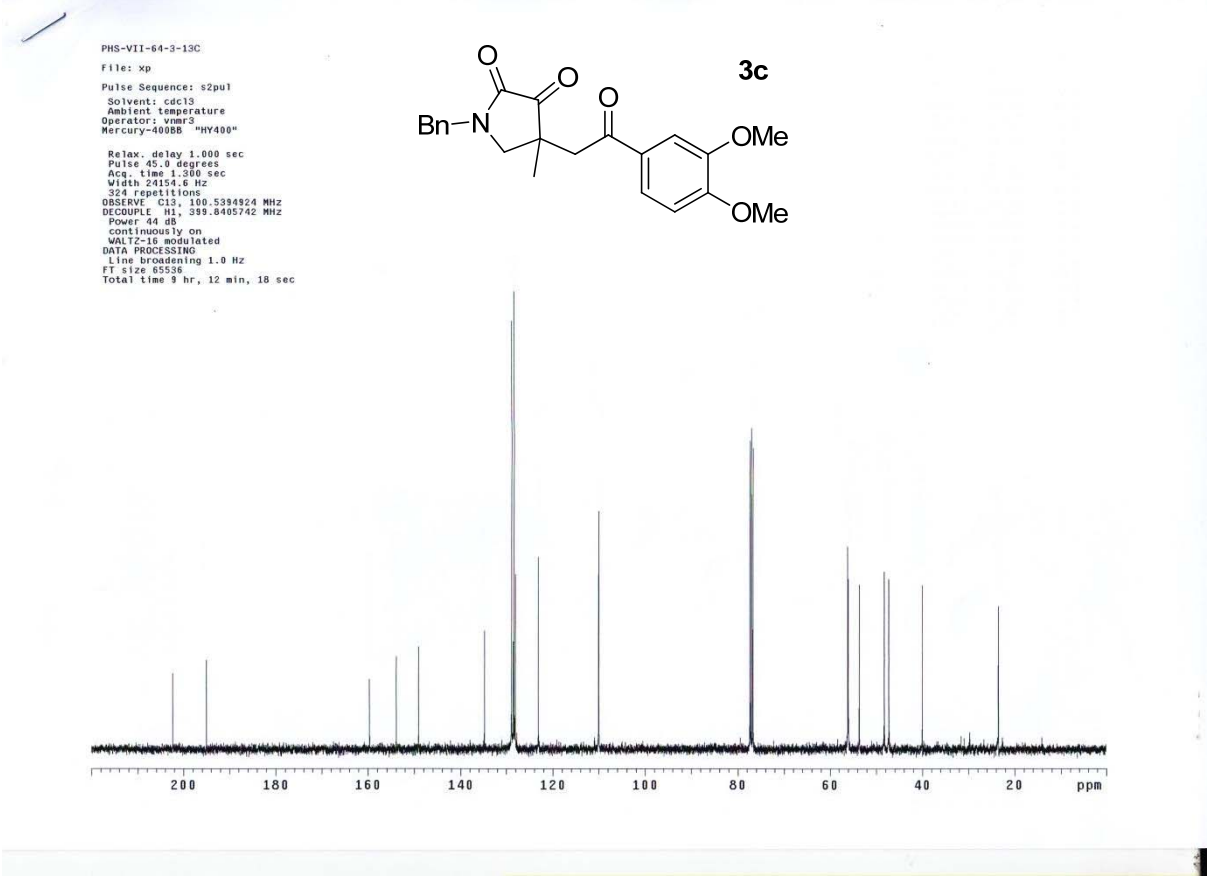


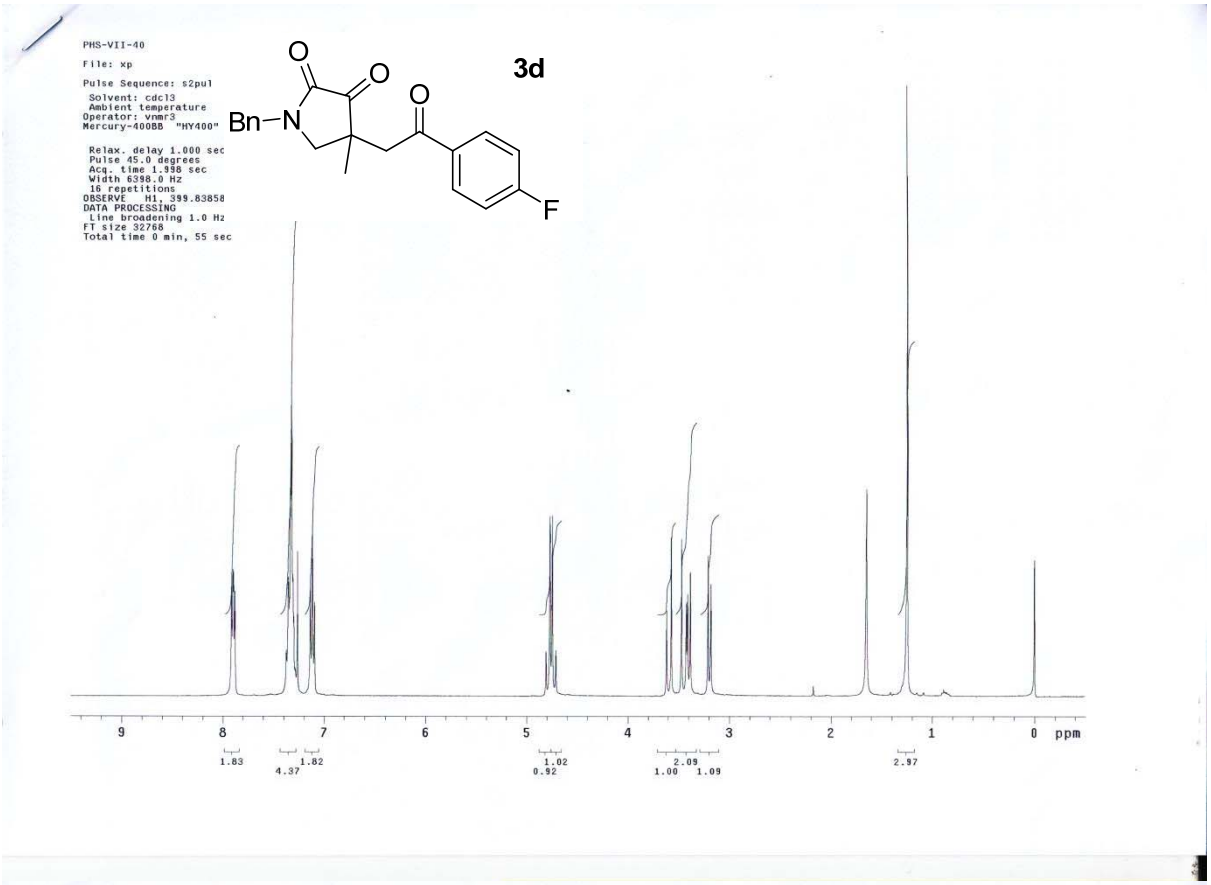
d-**3b** (Eq 3)

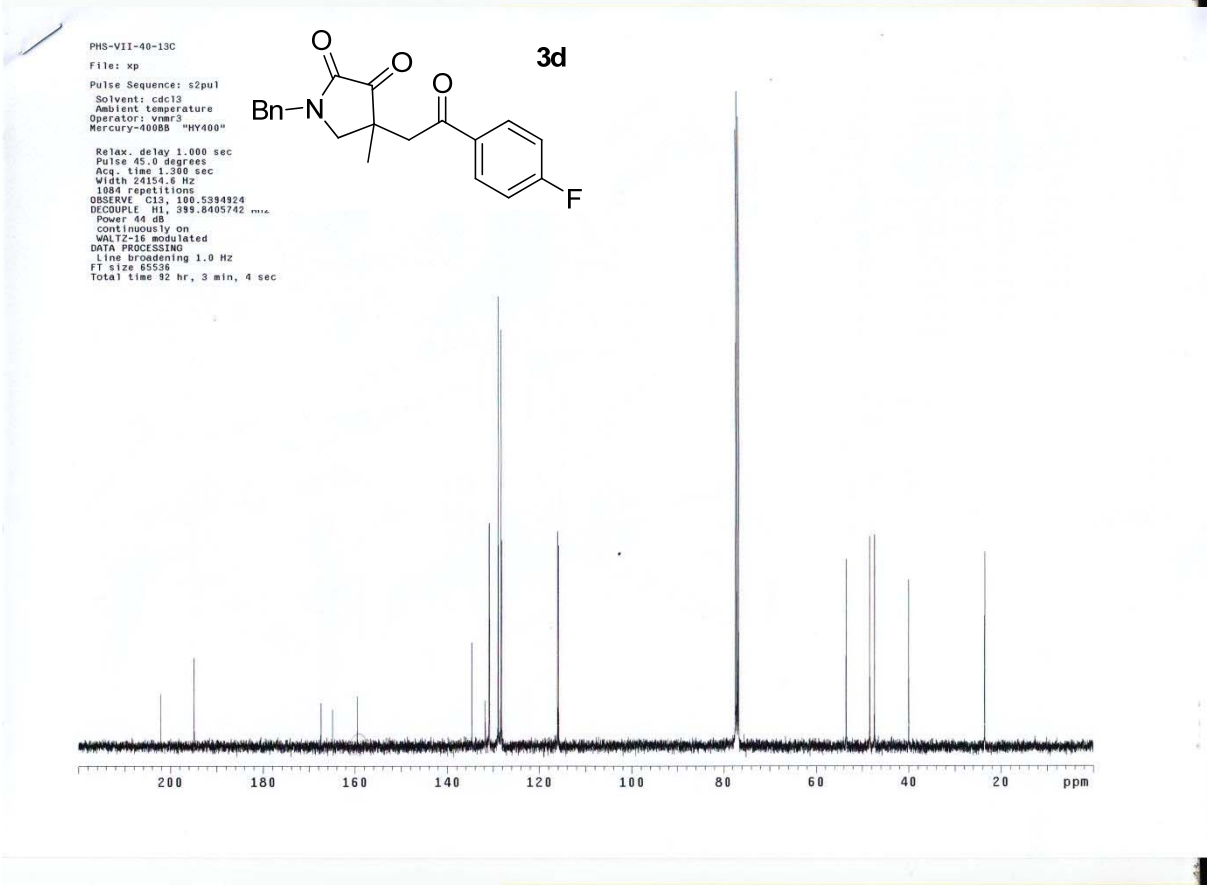


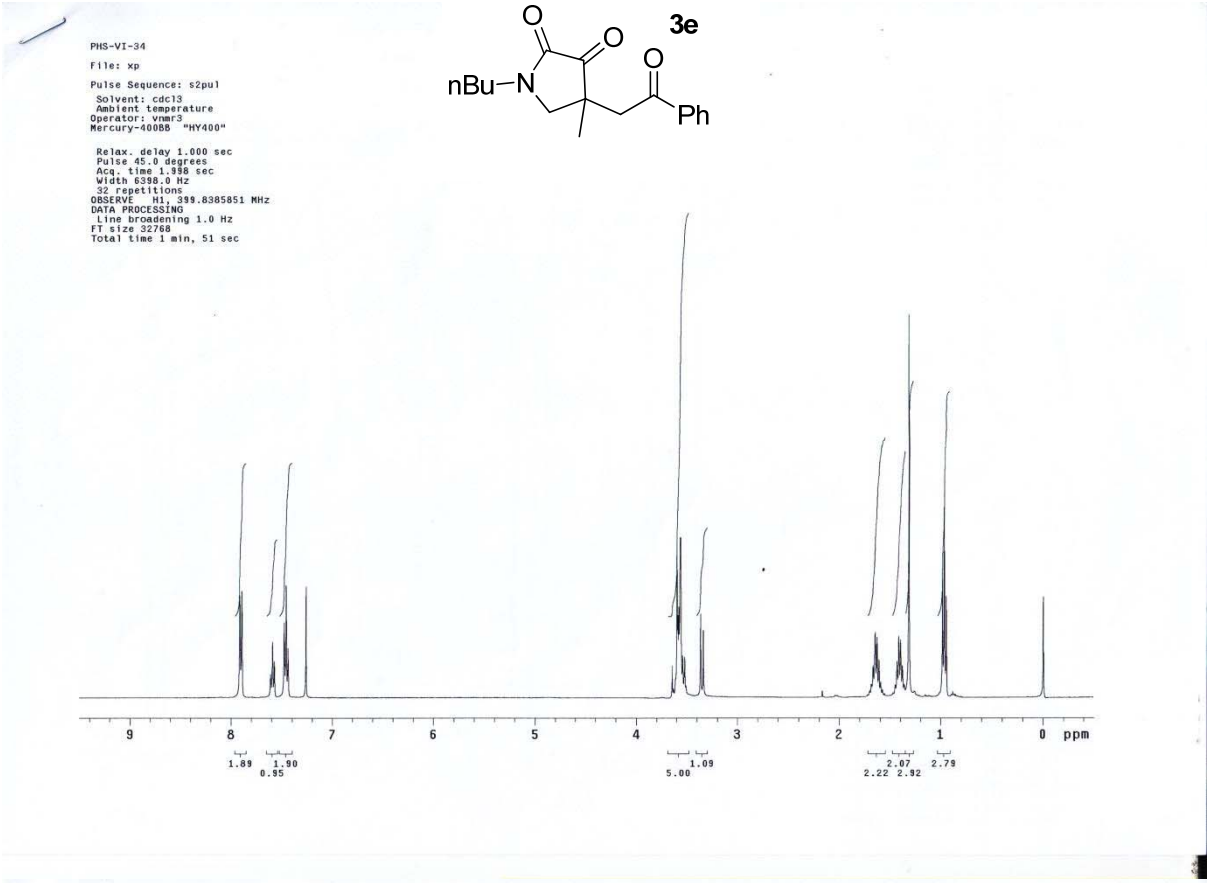

KJY-VI-50
File: xp
Pulse Sequence: s2pul
Bn–N
O
O
O
Ph
H3C
D(0.73D)
d-3b (from (Z)-d-1b)
D content = [1-(0.24/0.89)]*100
= 73 %D
H-3b
+
d-3b
H-3b
9
8
7
6
5
4
3
2
1
0
ppm
1.92
2.06
1.06
4.82
2.00
0.24
0.89
1.02
0.99

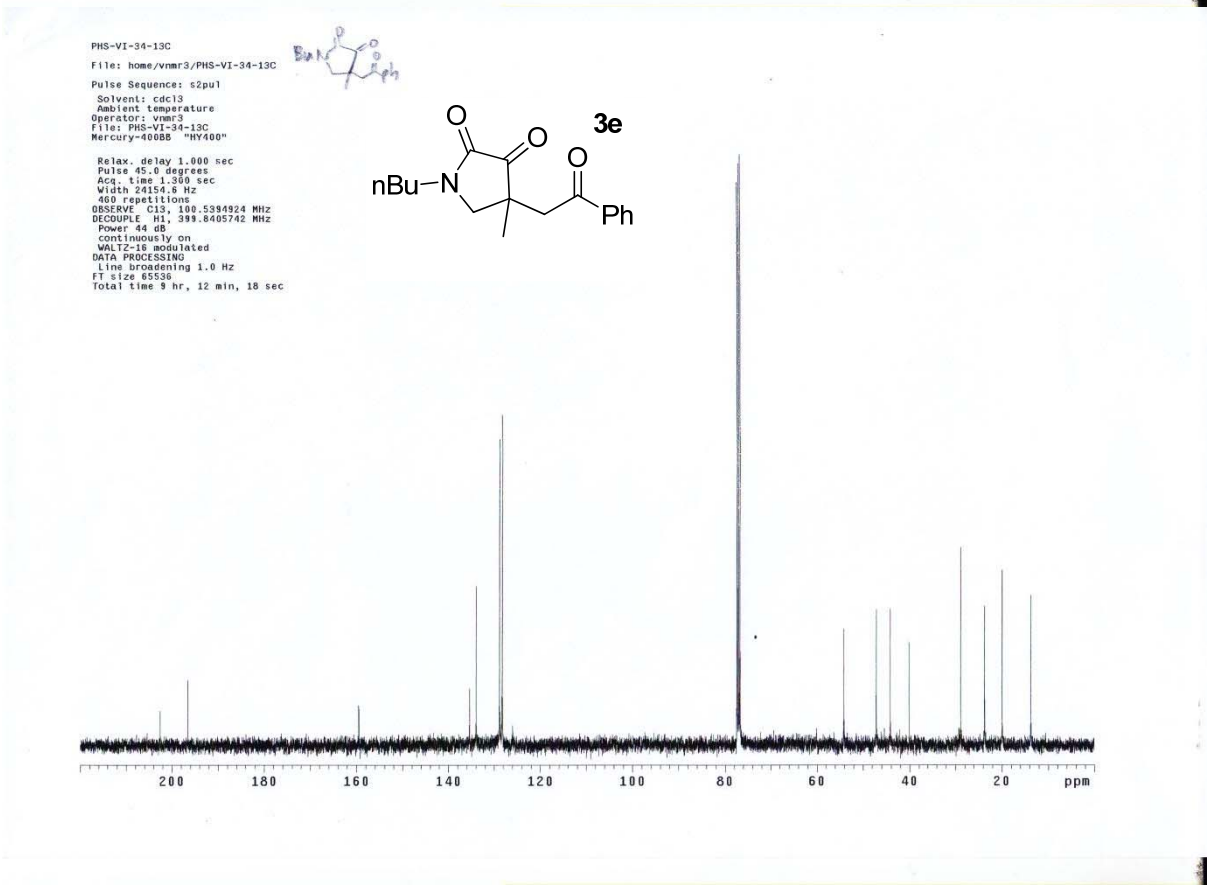

3c
PHS-VII-124
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400
Relax. delay 1.000 s
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
40 repetitions
OBSERVE H1, 399.838
DATA PROCESSING
Line broadening 1.0
FT size 32768
Total time 2 min, 19
Bn
N
O
O
O
OMe
OMe
9
8
7
6
5
4
3
2
1
0 ppm
1.07
0.98
1.05
1.00
1.04
2.68
3.69
1.11
1.09
1.19
1.15
3.97

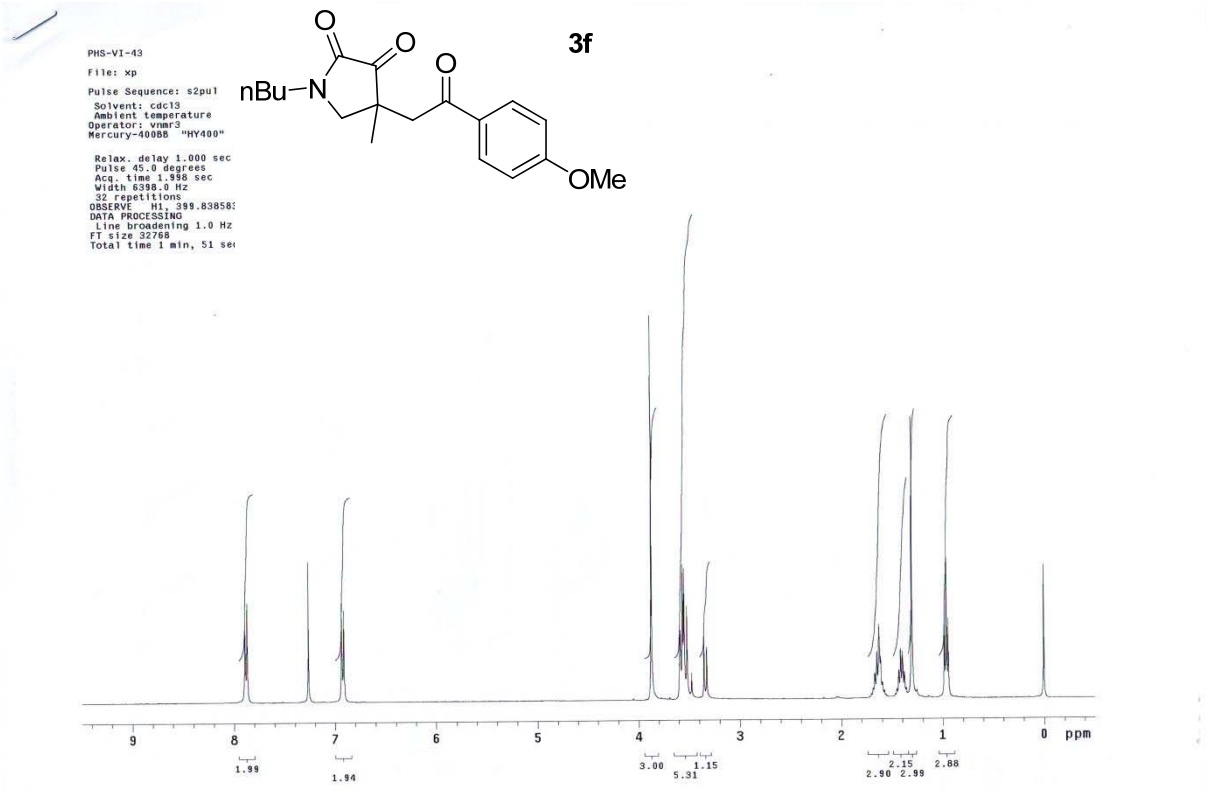

3c
PHS-VII-64-3-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
324 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
Bn
N
O
O
O
OMe
OMe
200
180
160
140
120
100
80
60
40
20
ppm

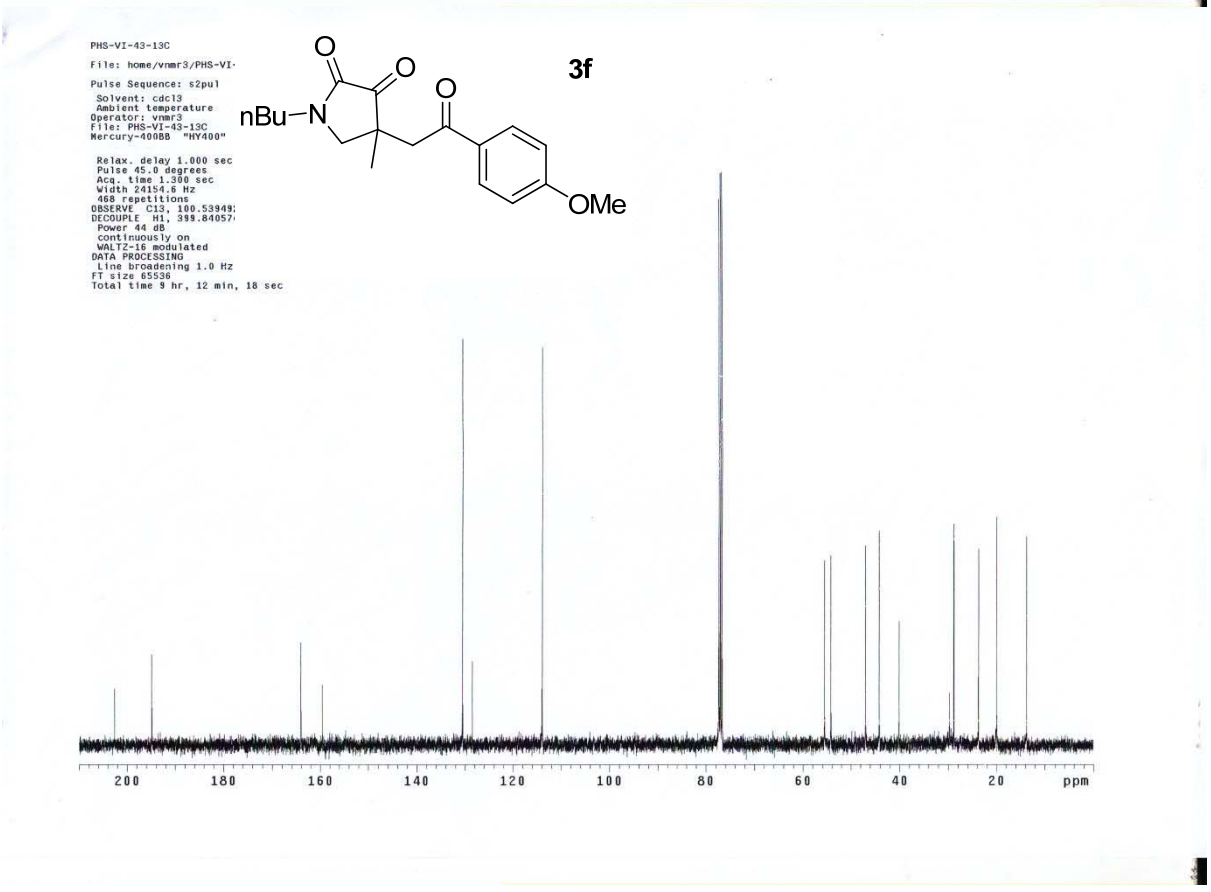

PHS-VII-40
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.83858
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
3d
Bn
N
O
O
O
F
9
8
7
6
5
4
3
2
1
0
ppm
1.83
4.37
1.82
1.02
0.92
2.09
1.00
1.09
2.97

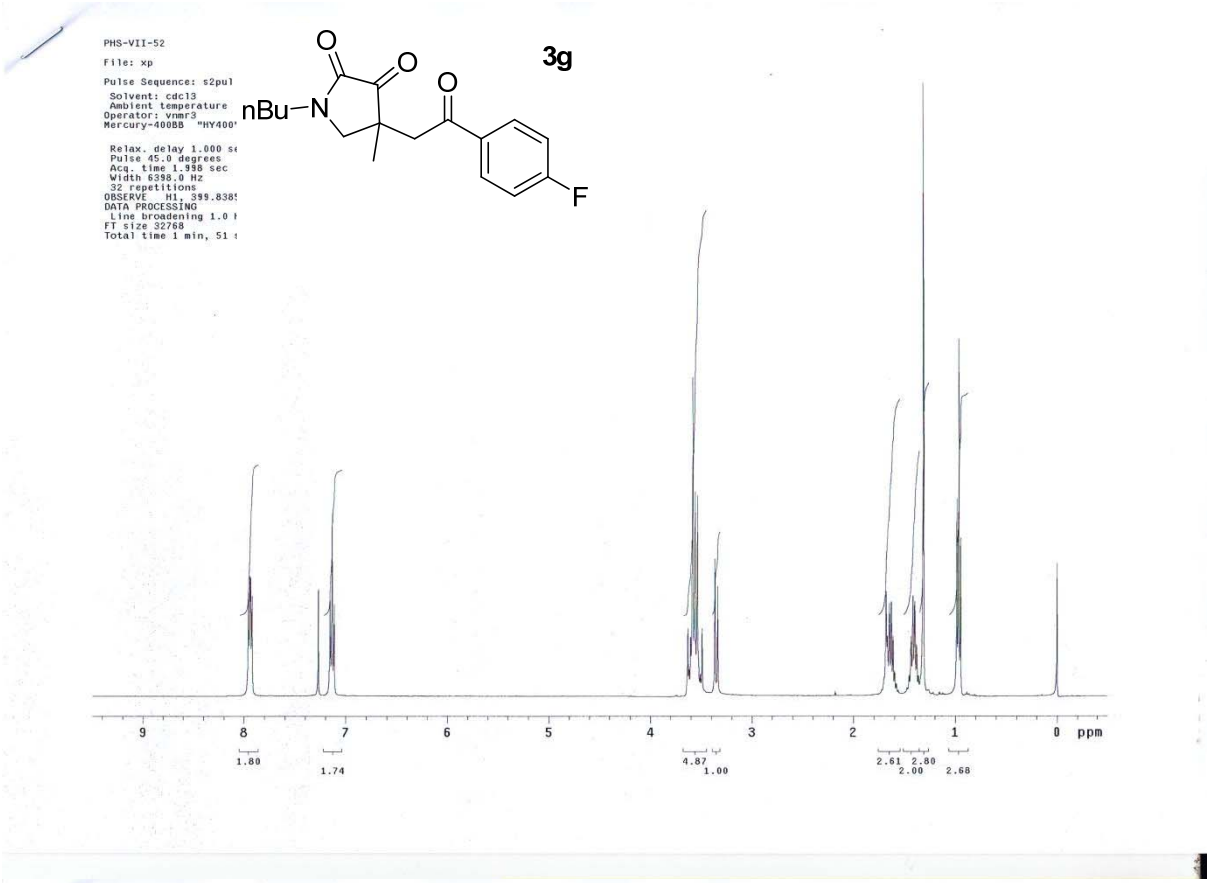

PHS-VII-40-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1084 repetitions
OBSERVE C13, 100.5394924
DECOUPLE H1, 399.8405742
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 92 hr, 3 min, 4 sec
3d
Bn
N
O
O
O
F
200
180
160
140
120
100
80
60
40
20
ppm

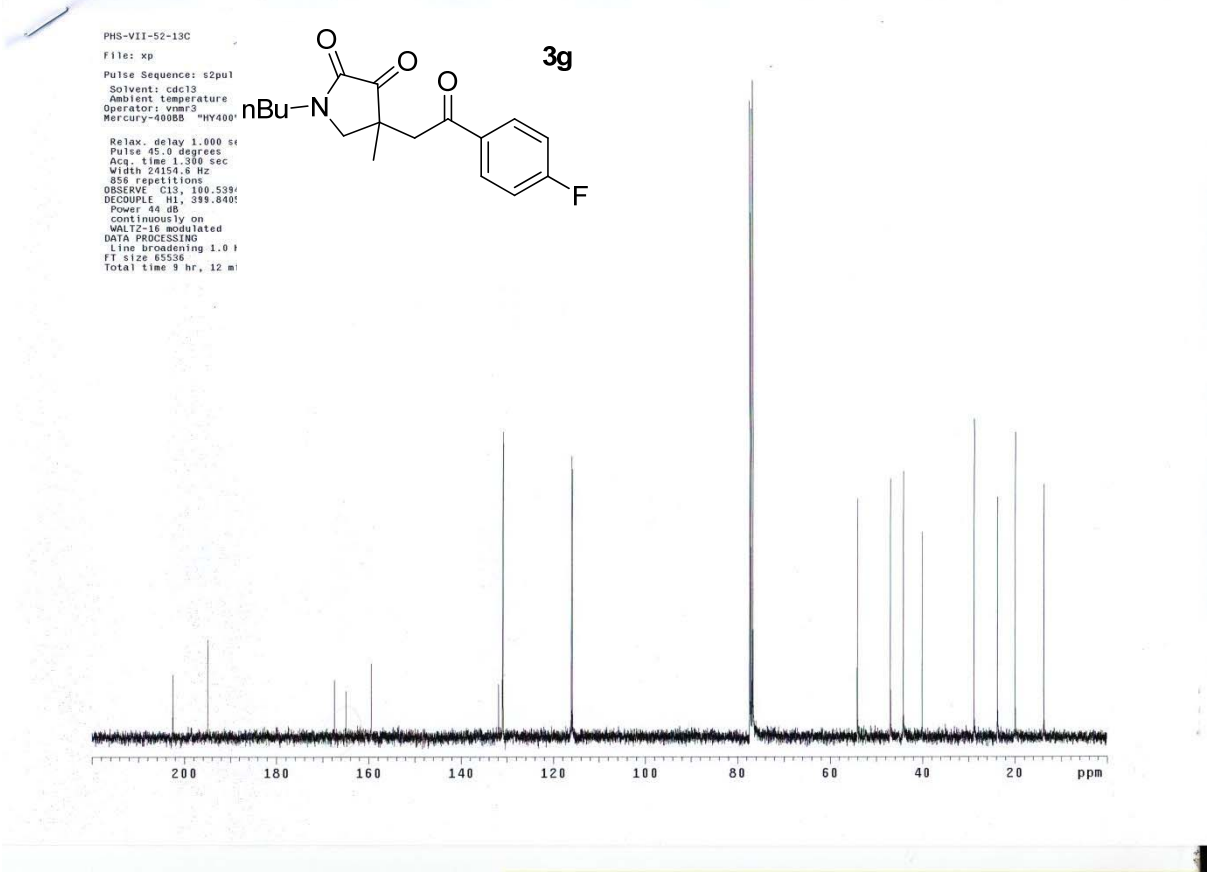

PHS-VI-34
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.8385851 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 min, 51 sec
3e
nBu
N
O
O
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
1.89
1.90
0.95
5.00
1.09
2.22
2.07
2.92
2.79

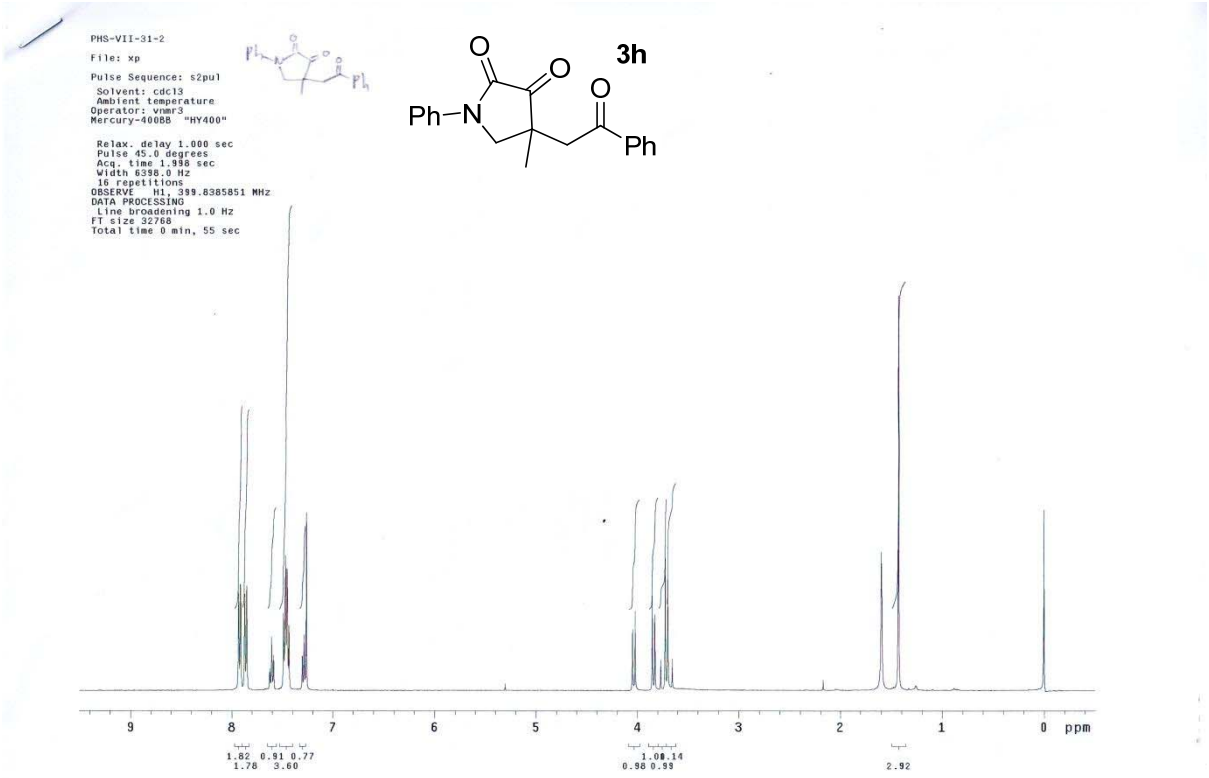

PHS-VI-34-13C
File: home/vnmr3/PHS-VI-34-13C
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VI-34-13C
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
460 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
3e
nBu
N
O
O
O
Ph
200
180
160
140
120
100
80
60
40
20
ppm

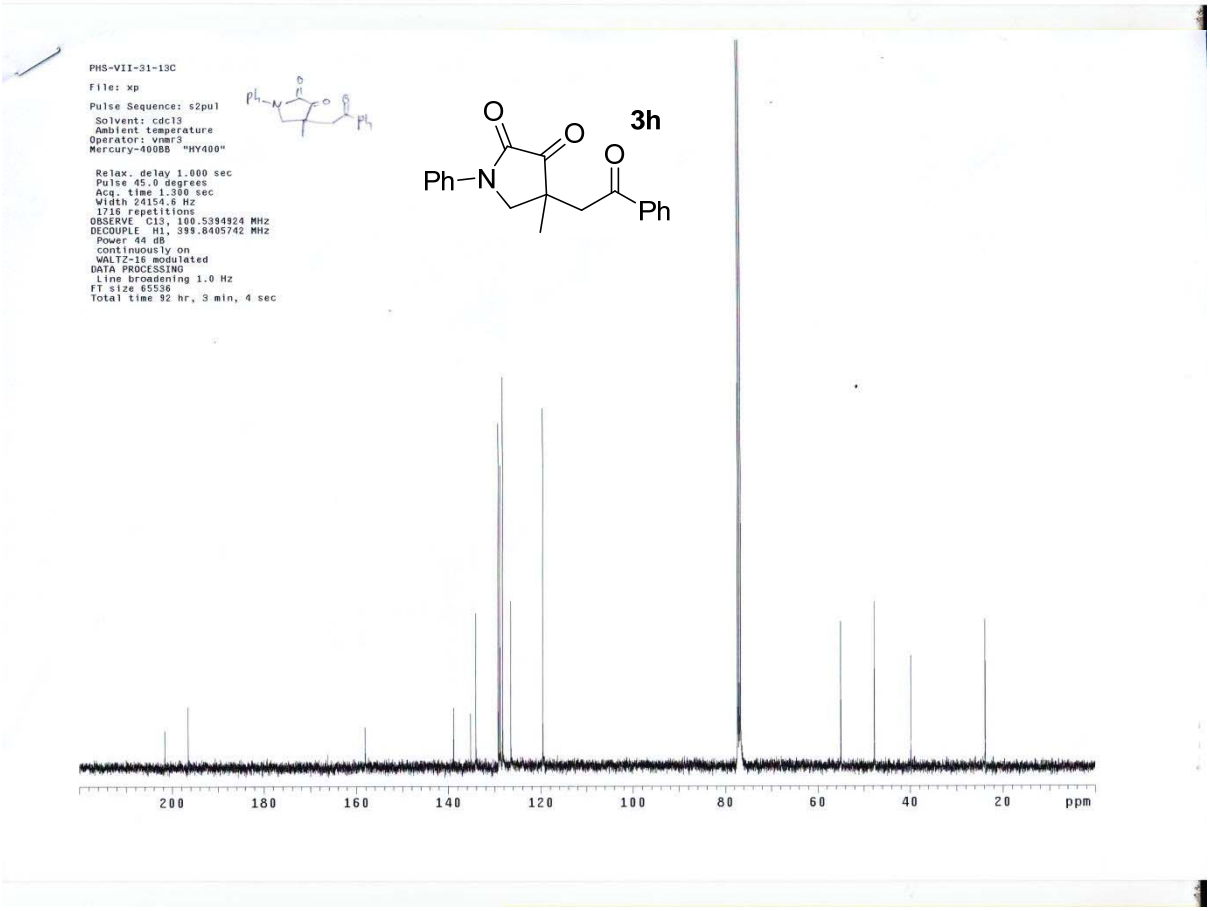

PHS-VI-43
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.83858
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 min, 51 se
3f
nBu
N
O
O
O
OMe
9
8
7
6
5
4
3
2
1
0 ppm
1.99
1.94
3.00
1.15
5.31
2.15
2.90
2.99
2.88

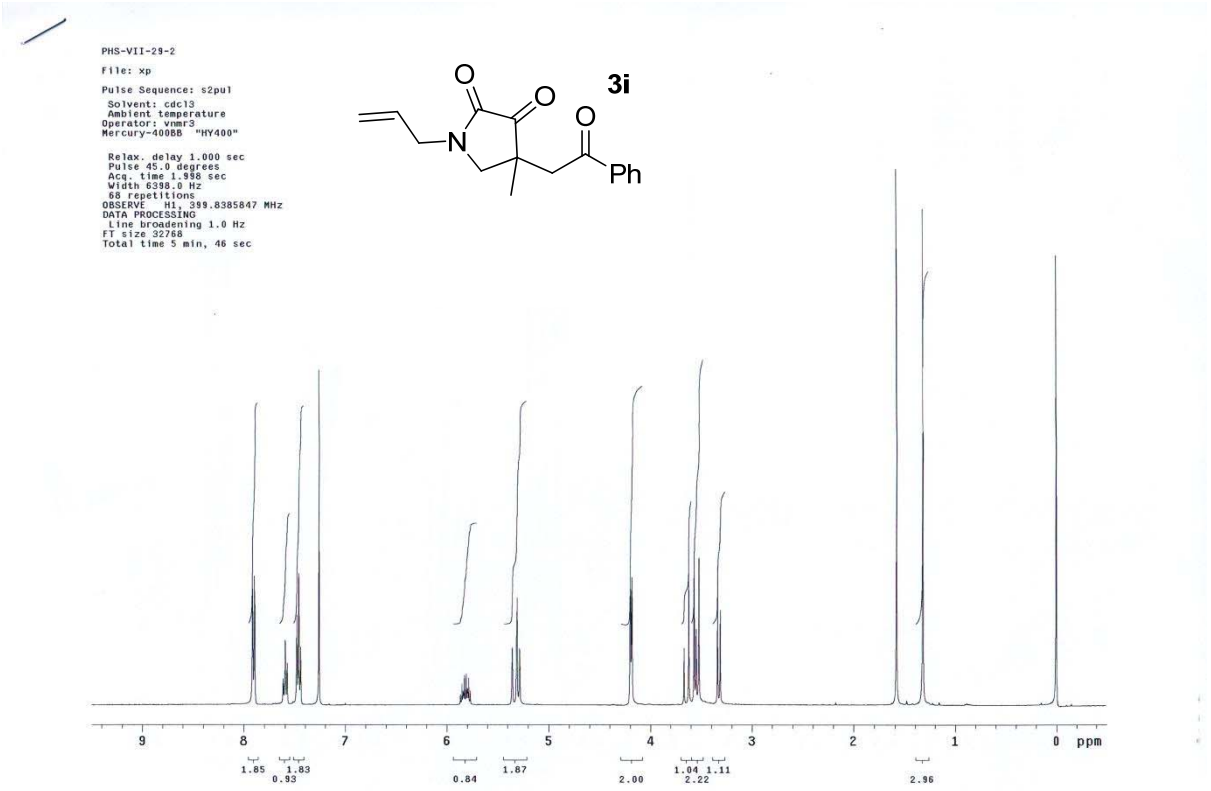

PHS-VI-43-13C
File: home/vnmr3/PHS-VI-
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VI-43-13C
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
468 repetitions
OBSERVE C13, 100.53949
DECOUPLE H1, 399.84057
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
3f
nBu
N
O
O
O
OMe
200
180
160
140
120
100
80
60
40
20
ppm

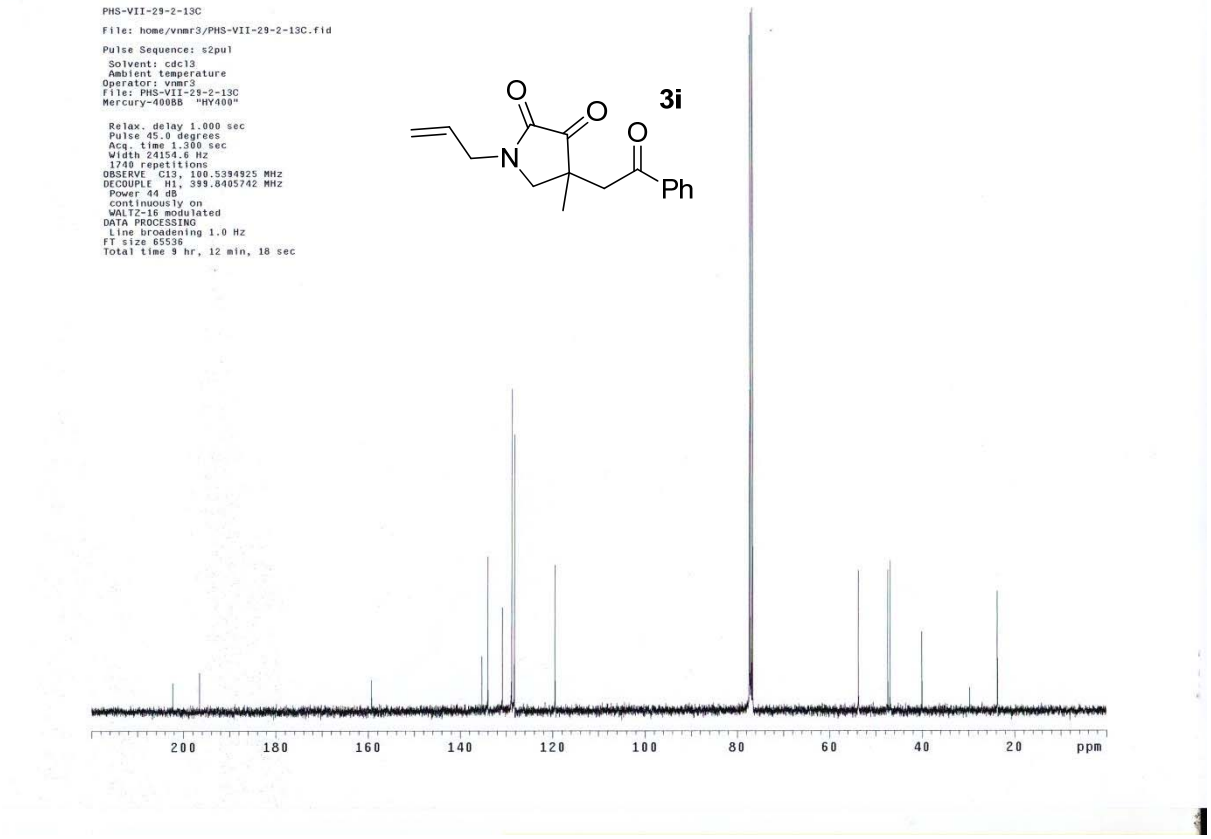

PHS-VII-52
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 s
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.838
DATA PROCESSING
Line broadening 1.0
FT size 32768
Total time 1 min, 51
3g
nBu
N
O
O
O
F
9
8
7
6
5
4
3
2
1
0
ppm
1.80
1.74
4.87
1.00
2.61
2.80
2.00
2.68


PHS-VII-52-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 s
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
856 repetitions
OBSERVE C13, 100.539
DECOUPLE H1, 399.840
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0
FT size 65536
Total time 9 hr, 12 m
3g
nBu
N
O
O
O
F
200
180
160
140
120
100
80
60
40
20
ppm


PHS-VII-31-2
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385851 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
3h
Ph
N
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
1.82
1.78
0.91
3.60
0.77
1.08
0.98
0.14
0.89
2.92

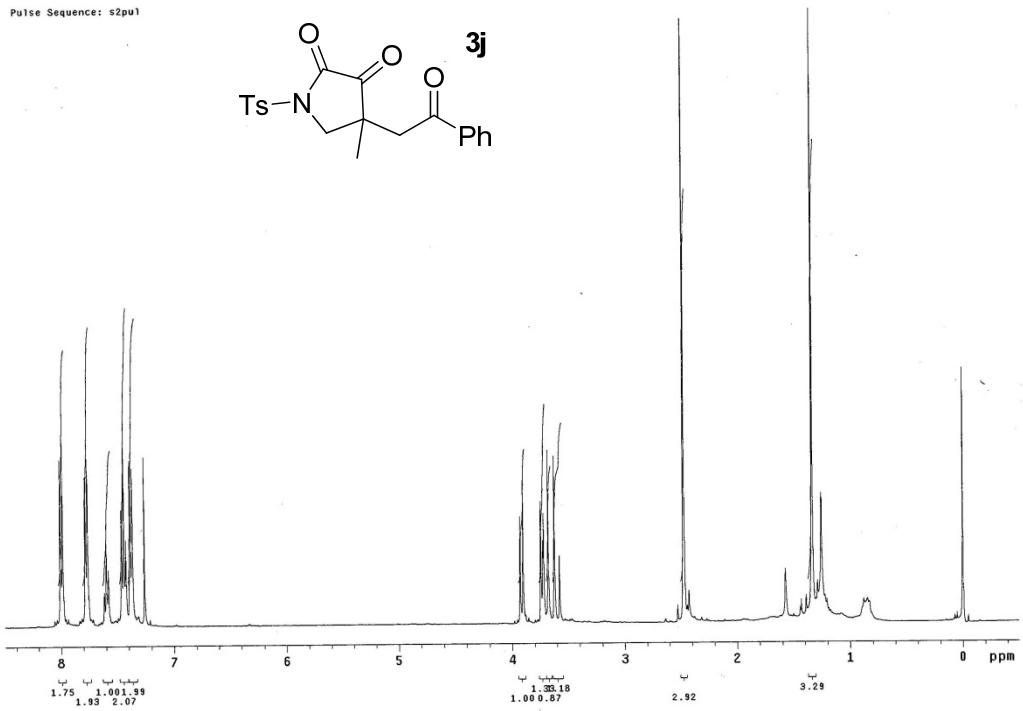

PHS-VII-31-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1716 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 92 hr, 3 min, 4 sec
3h
Ph
N
O
Ph
200
180
160
140
120
100
80
60
40
20
ppm


PHS-VII-29-2
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
68 repetitions
OBSERVE H1, 399.8385847 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 5 min, 46 sec
3i
O
O
O
N
Ph
9
8
7
6
5
4
3
2
1
0 ppm
1.85
1.83
0.93
0.84
1.87
2.00
1.04
1.11
2.22
2.96


PHS-VII-29-2-13C
File: home/vnmr3/PHS-VII-29-2-13C.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
File: PHS-VII-29-2-13C
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1740 repetitions
OBSERVE C13, 100.5394925 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
3i
O
O
O
N
Ph
200
180
160
140
120
100
80
60
40
20
ppm

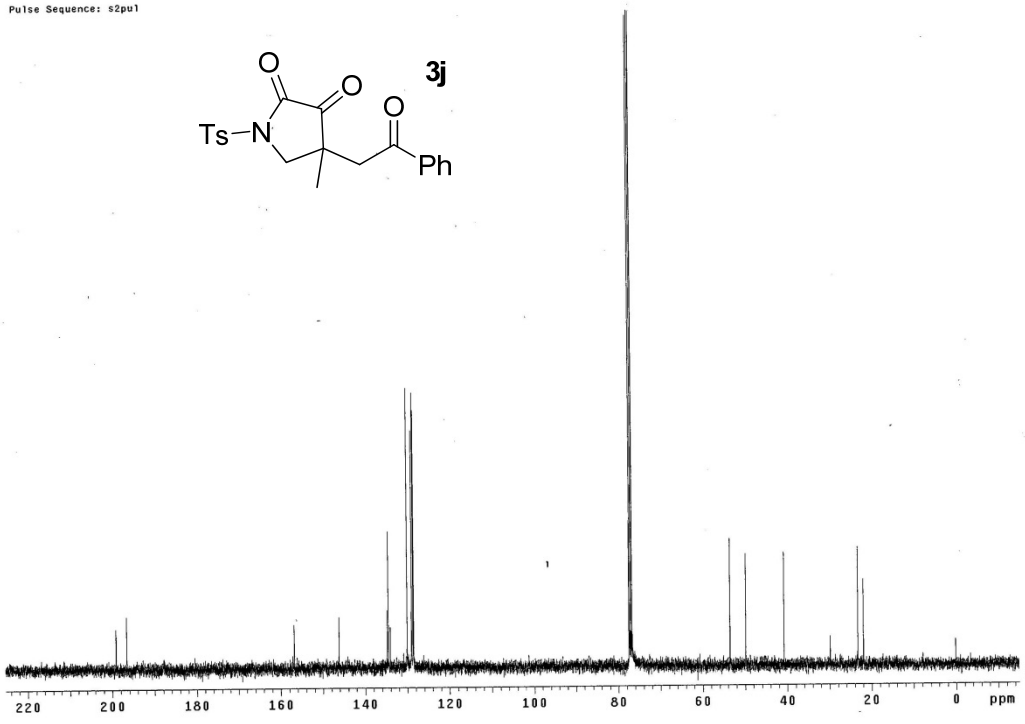
DVP-III-44fr2-p
File: xp
Pulse Sequence: s2pul

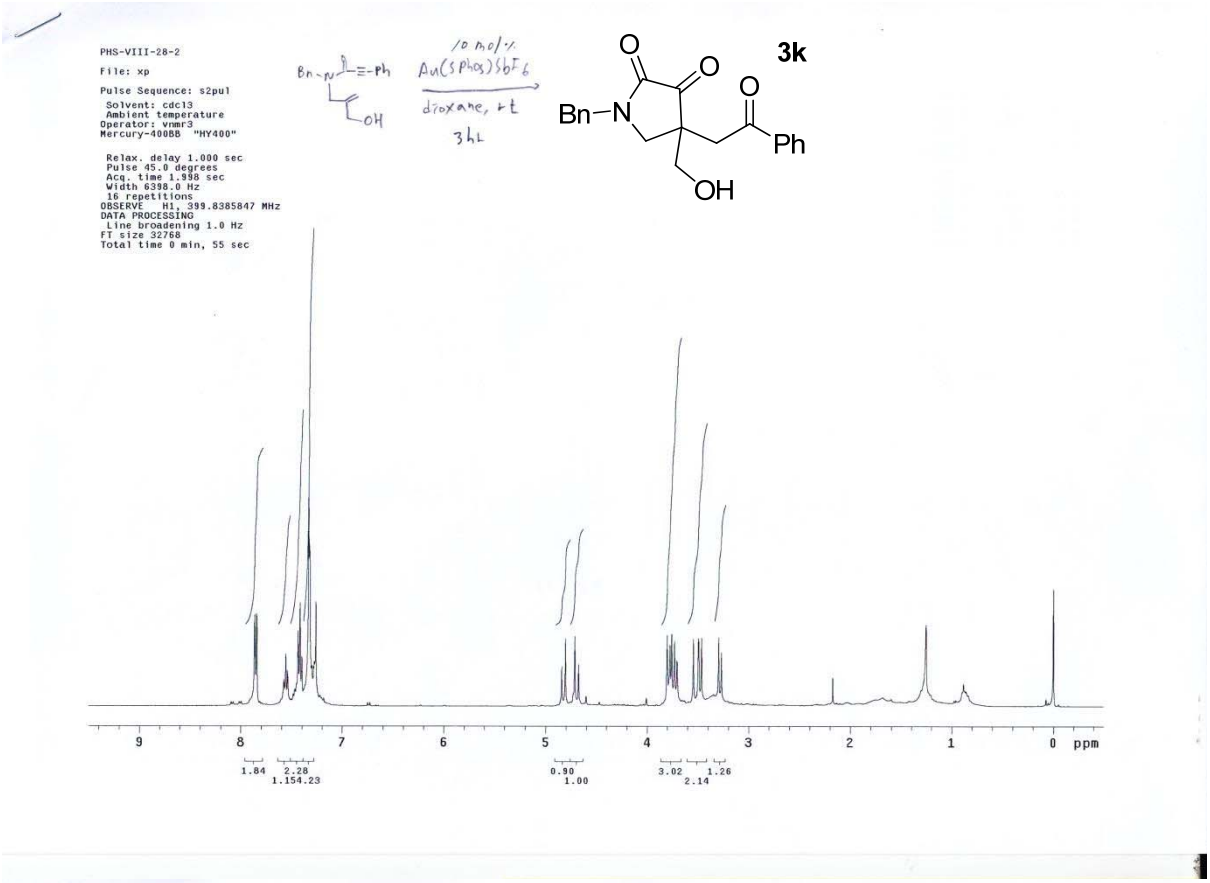
3j

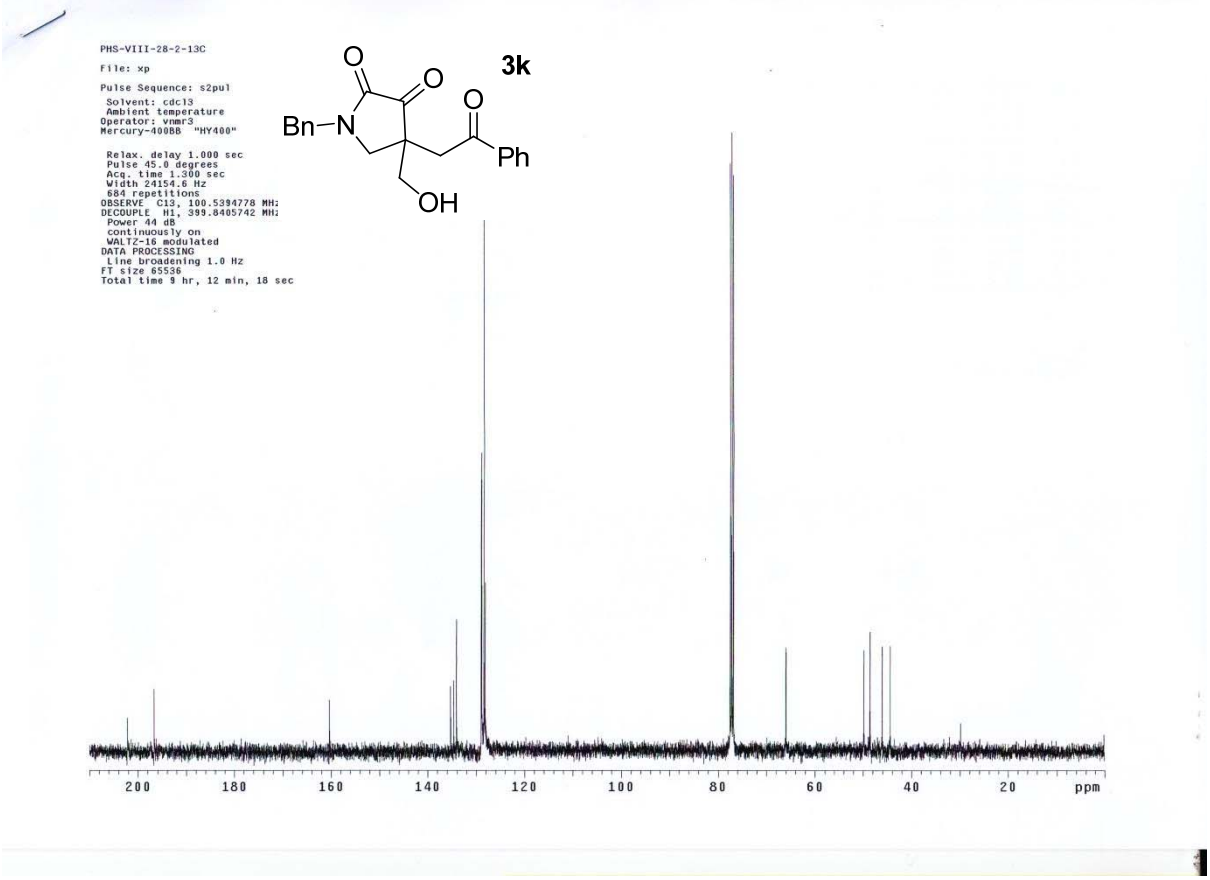


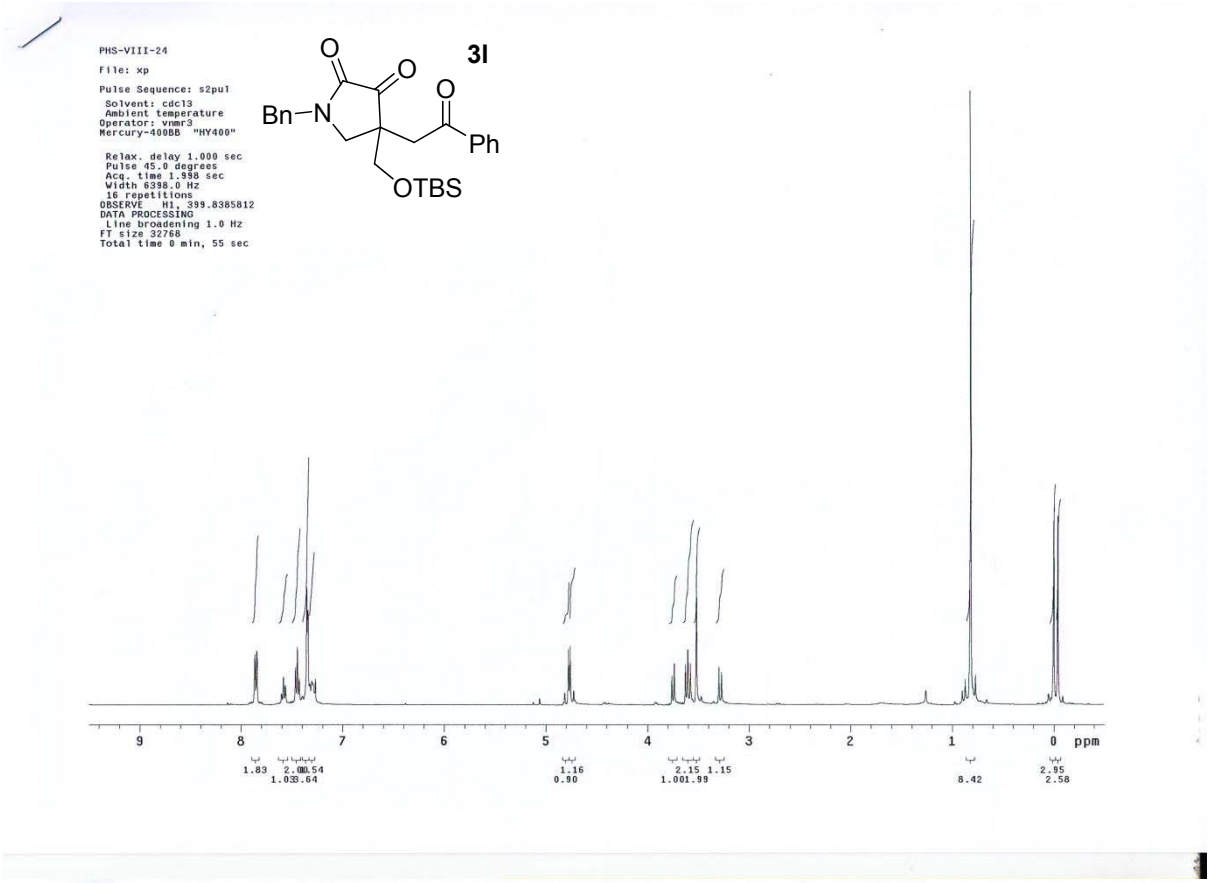
DVP-III-44fr2-p-13C
File: xp
Pulse Sequence: s2pul

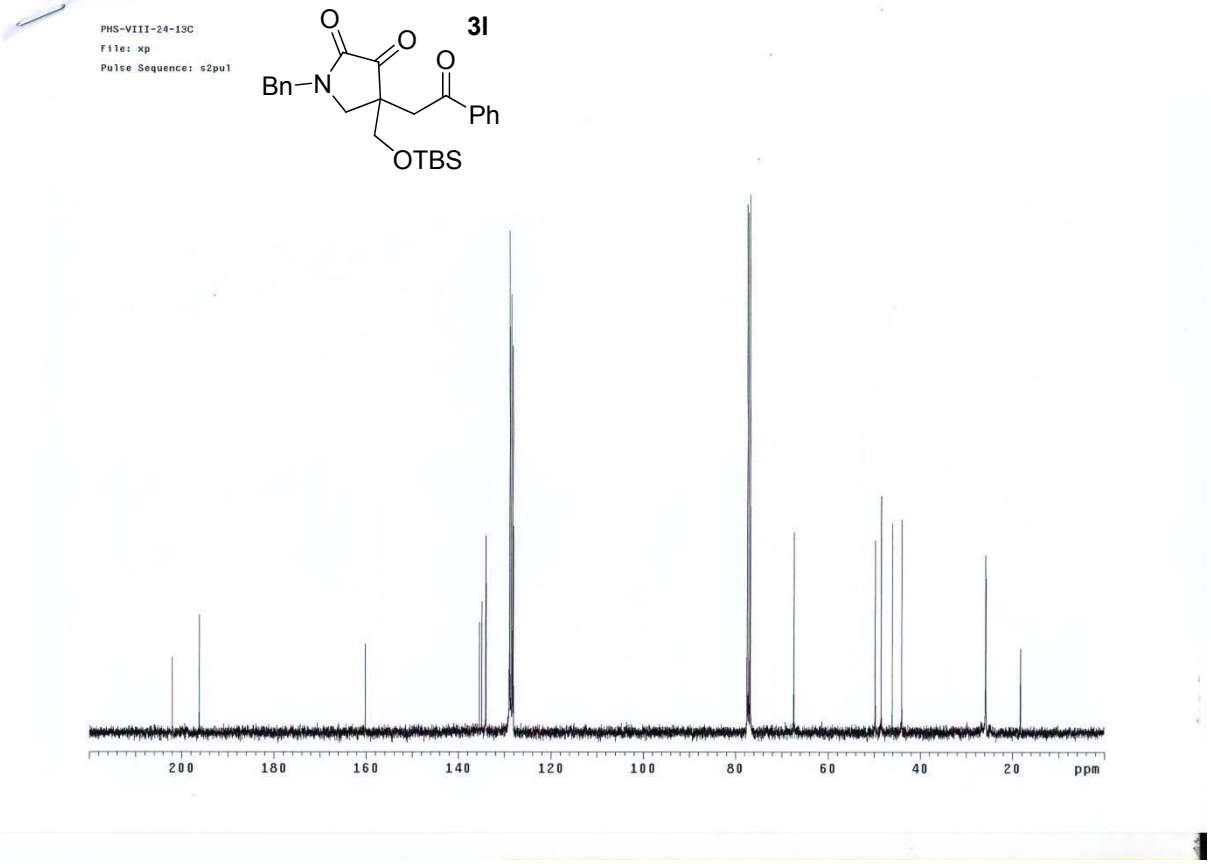
3j



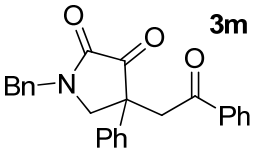

PHS-VIII-28-2
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385847 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
10 mol%
Au(SPhos)SbF6
dioxane, rt
3h
3k
Bn
N
Ph
OH
9
8
7
6
5
4
3
2
1
0
ppm
1.84
2.28
1.154.23
0.90
1.00
3.02
1.26
2.14

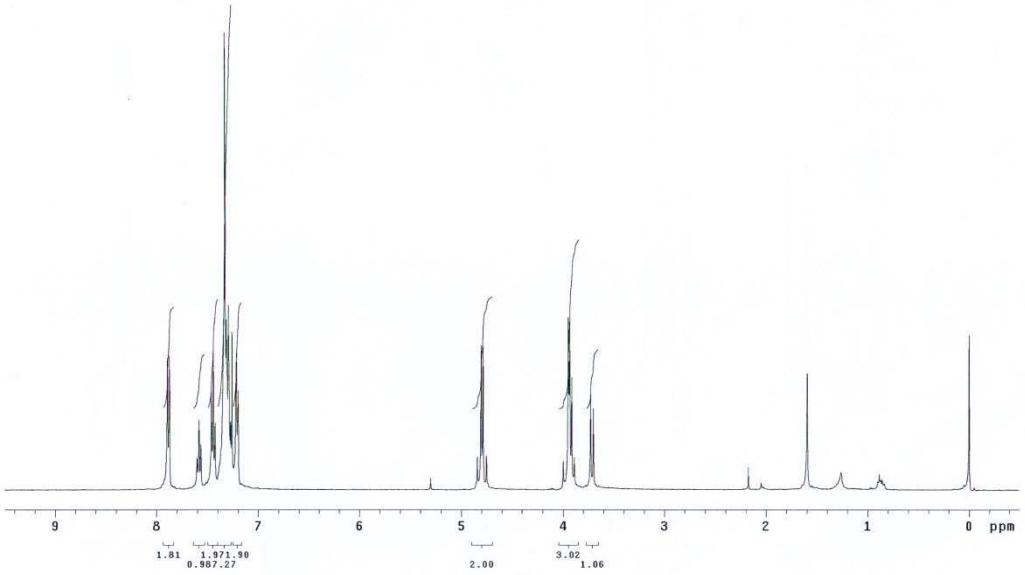

PHS-VIII-28-2-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
684 repetitions
OBSERVE C13, 100.5394778 MH:
DECOUPLE H1, 399.8405742 MH:
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
3k
Bn
N
Ph
OH
200
180
160
140
120
100
80
60
40
20
ppm


PHS-VIII-24
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.8385812
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 55 sec
3l
Bn
N
O
O
O
Ph
OTBS
9
8
7
6
5
4
3
2
1
0
ppm
1.83
1.03
3.64
1.16
0.90
2.15
1.15
1.00
1.99
8.42
2.95
2.58

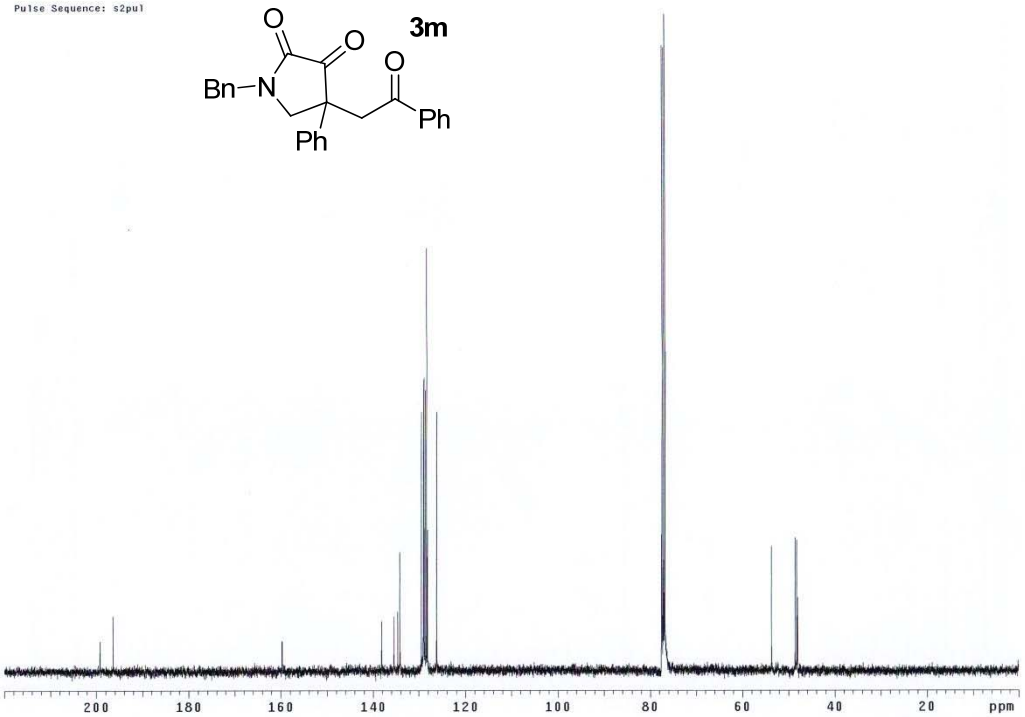

PHS-VIII-24-13C
File: xp
Pulse Sequence: s2pul
3l
Bn
N
O
O
O
Ph
OTBS
200
180
160
140
120
100
80
60
40
20
ppm

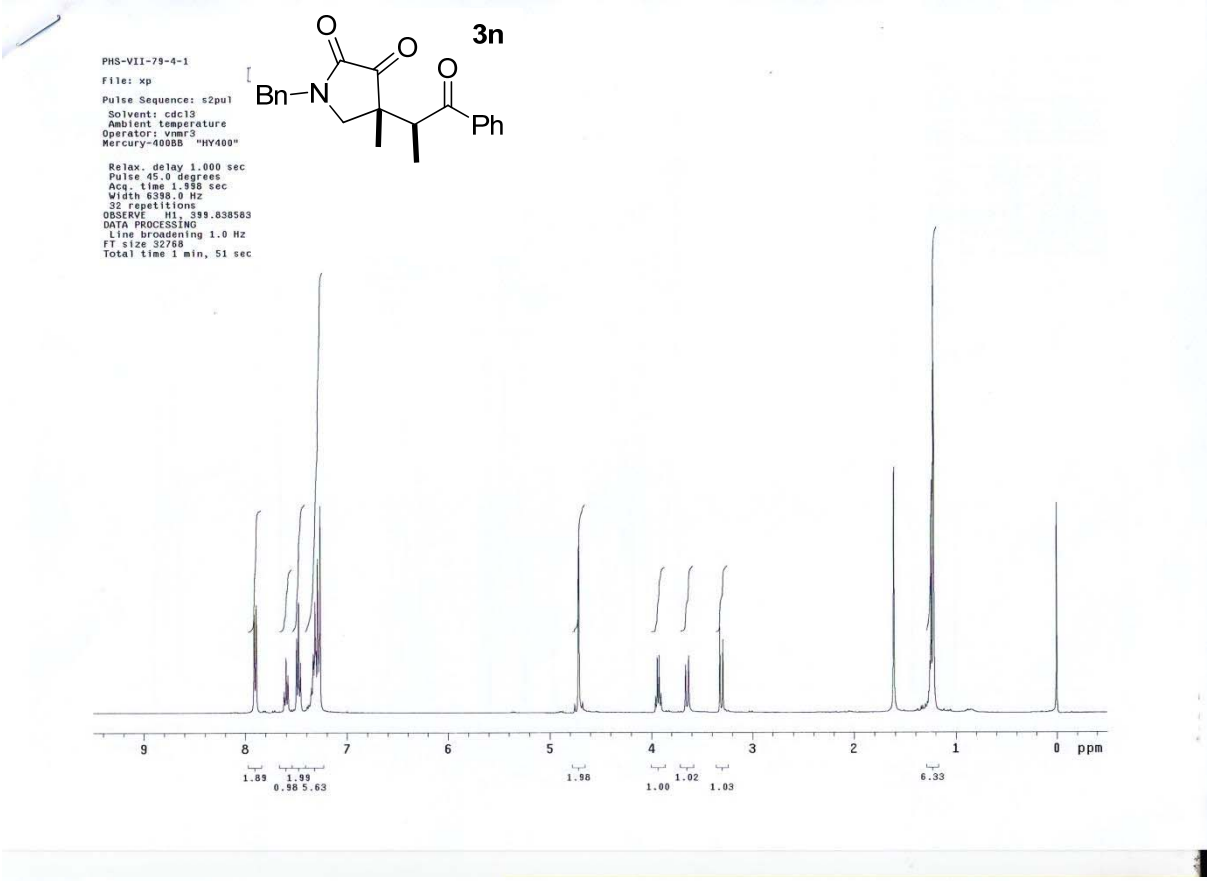
KJY-VI-81-recrystalliz-1H
File: xp
Pulse Sequence: s2pul

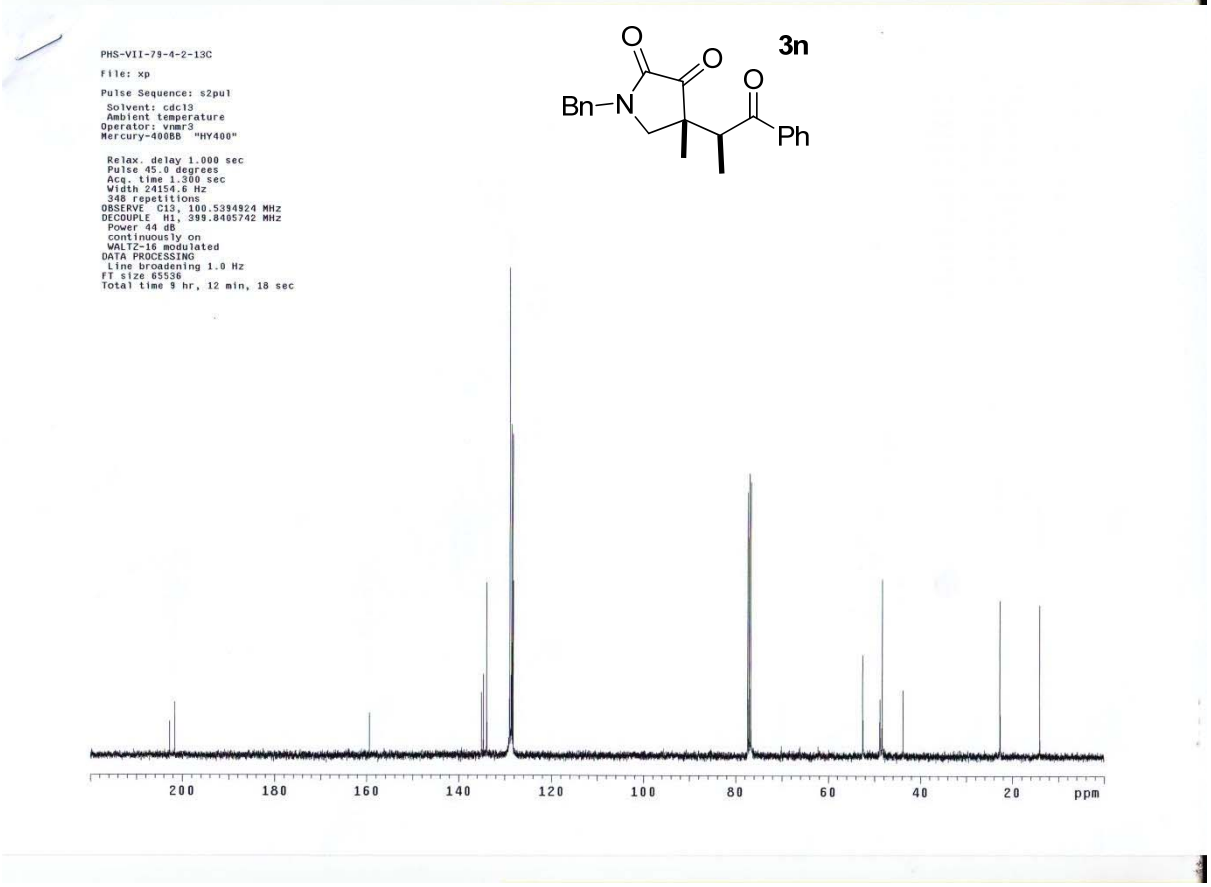


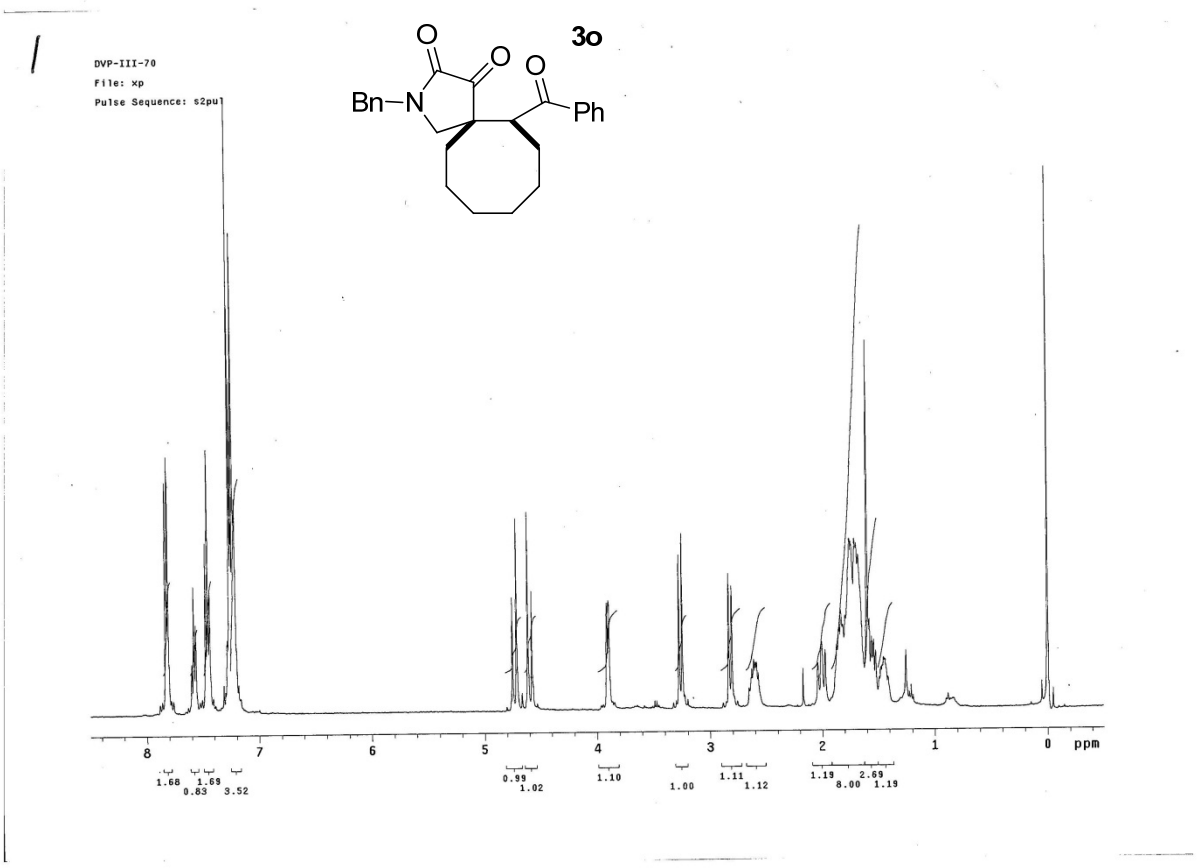


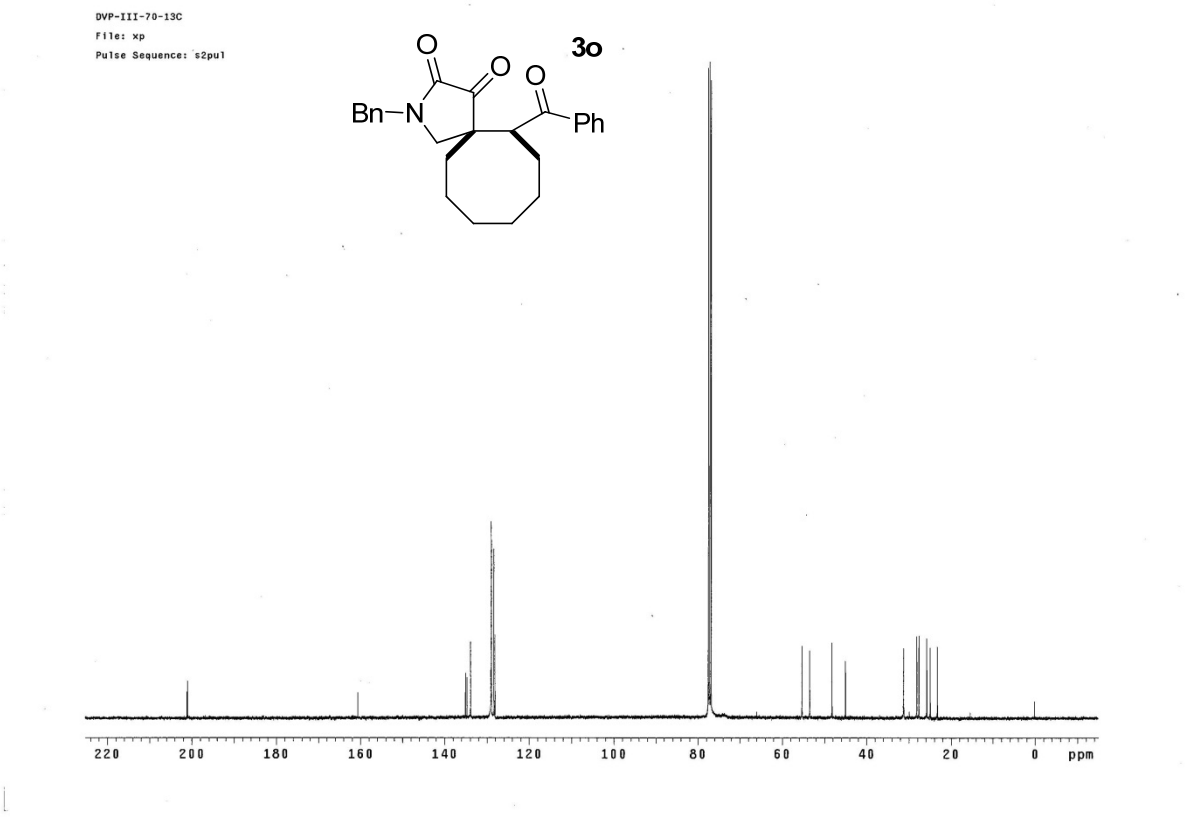
KJY-VI-81-recrystalliz-13C
File: xp
Pulse Sequence: s2pul

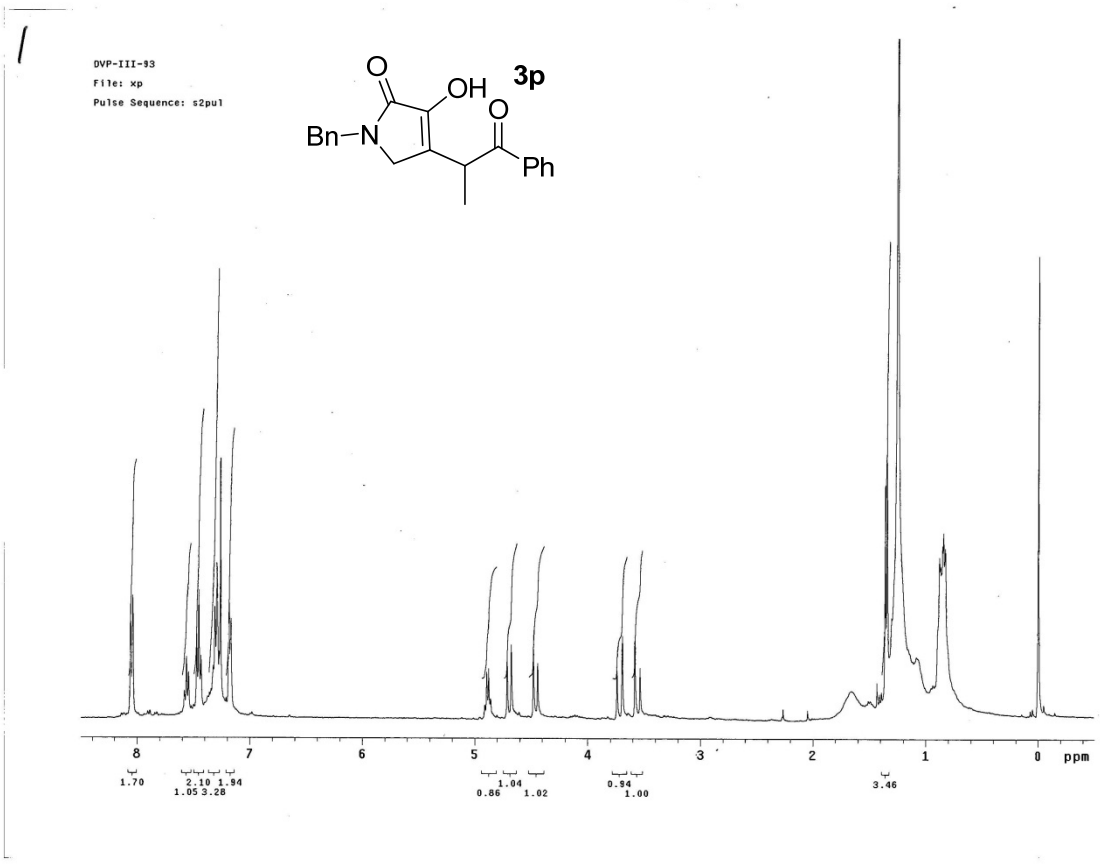


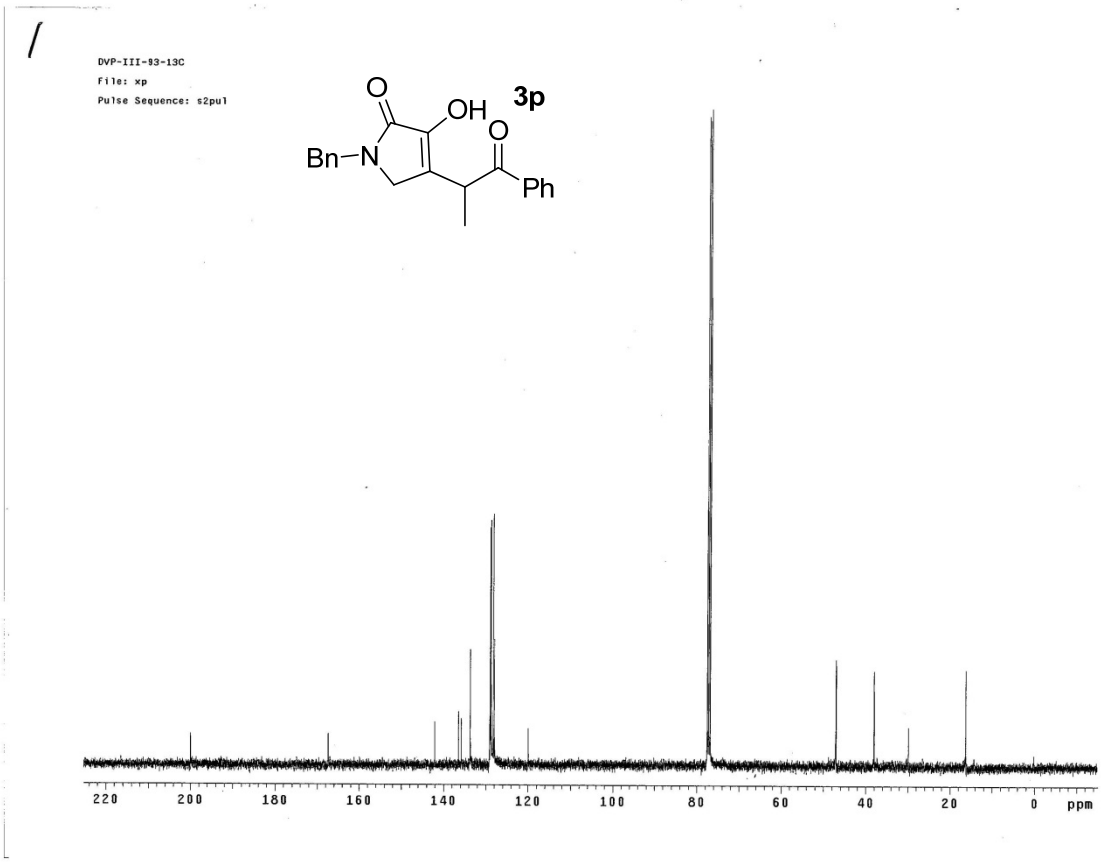

3n
PHS-VII-79-4-1
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.838583
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 1 min, 51 sec
Bn
N
O
O
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
1.89
1.99
0.98
5.63
1.98
1.00
1.02
1.03
6.33


3n
PHS-VII-79-4-2-13C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: vnmr3
Mercury-400BB "HY400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
348 repetitions
OBSERVE C13, 100.5394924 MHz
DECOUPLE H1, 399.8405742 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 18 sec
Bn
N
O
O
O
Ph
200
180
160
140
120
100
80
60
40
20
ppm

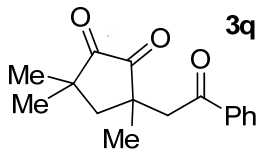

DVP-III-70
File: xp
Pulse Sequence: s2pul
3o
Bn
N
O
O
O
Ph
8
7
6
5
4
3
2
1
0
ppm
1.68
1.69
0.83
3.52
0.99
1.02
1.10
1.00
1.11
1.12
1.19
2.69
8.00
1.19


DVP-III-70-13C
File: xp
Pulse Sequence: s2pul
3o
Bn
N
O
O
O
Ph
220
200
180
160
140
120
100
80
60
40
20
0
ppm

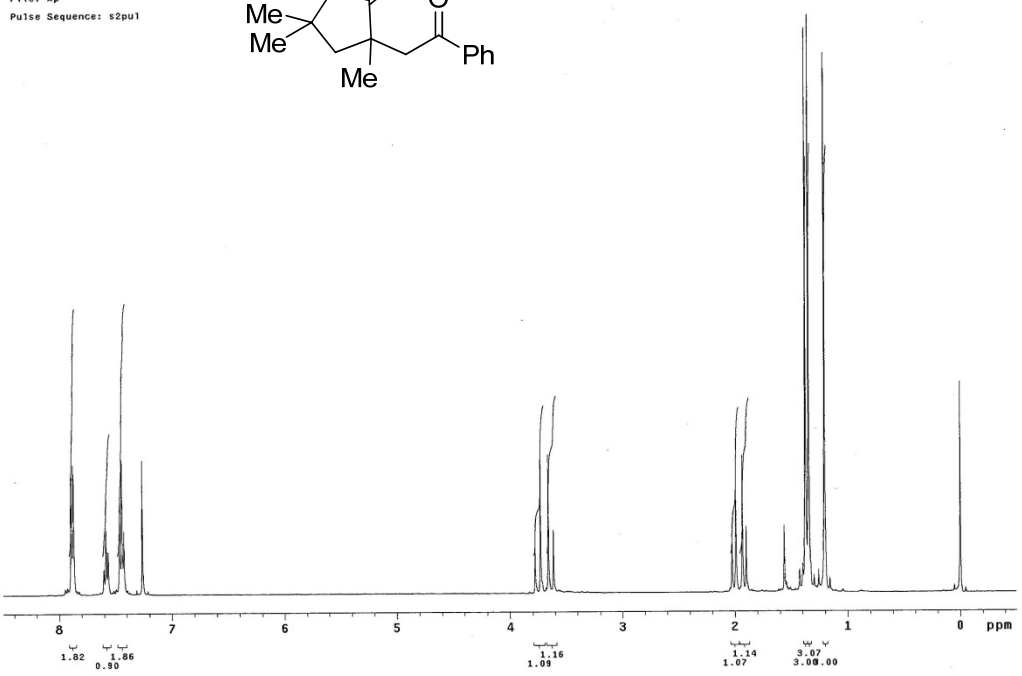

DVP-III-93
File: xp
Pulse Sequence: s2pul
3p
Bn
N
OH
O
O
Ph
8
7
6
5
4
3
2
1
0
ppm
1.70
2.10
1.05
1.94
3.28
1.04
0.86
1.02
0.94
1.00
3.46


DVP-III-93-13C
File: xp
Pulse Sequence: s2pul
3p
Bn
N
OH
O
O
Ph
220
200
180
160
140
120
100
80
60
40
20
0
ppm

DVP-III-49
File: xp
Pulse Sequence: s2pul

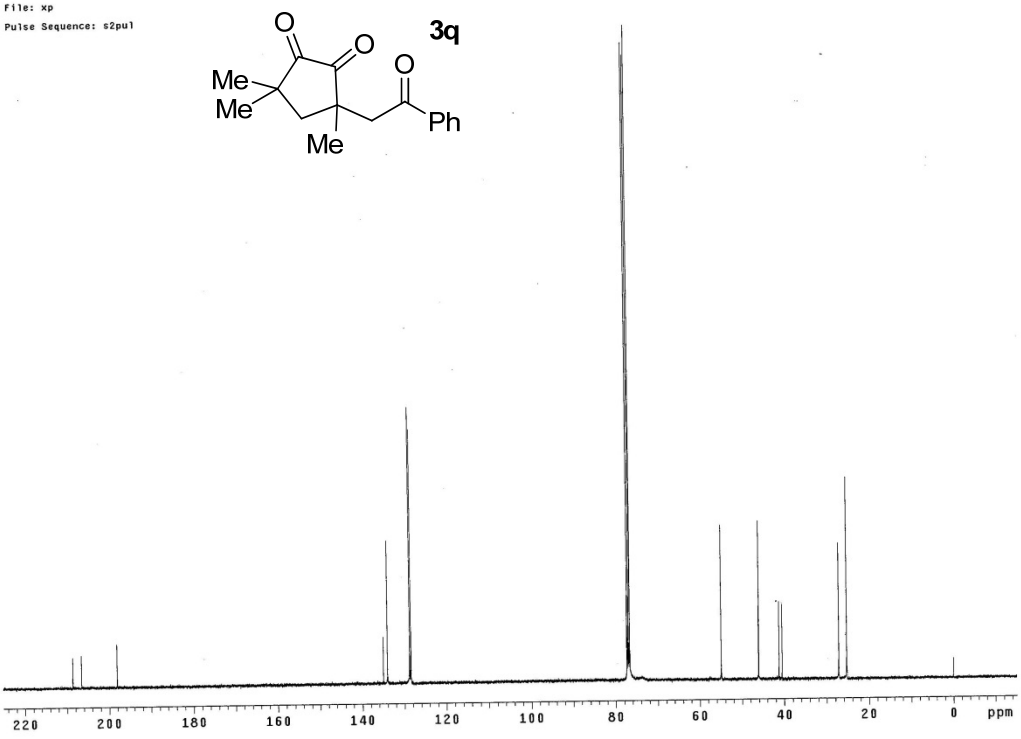


3q

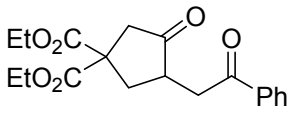


DVP-III-49-13C
File: xp
Pulse Sequence: s2pul

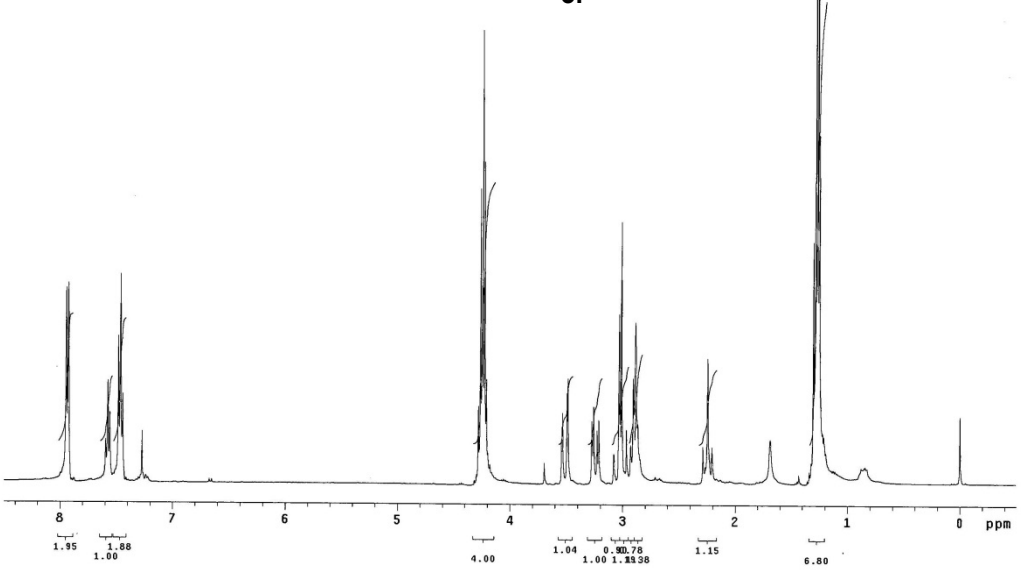
3q



DVP-IV-30p
File: home/vnmr3/vnmrsys/data/14.07/DVP/DVP-IV-30p.fid
Pulse Sequence: s2pul

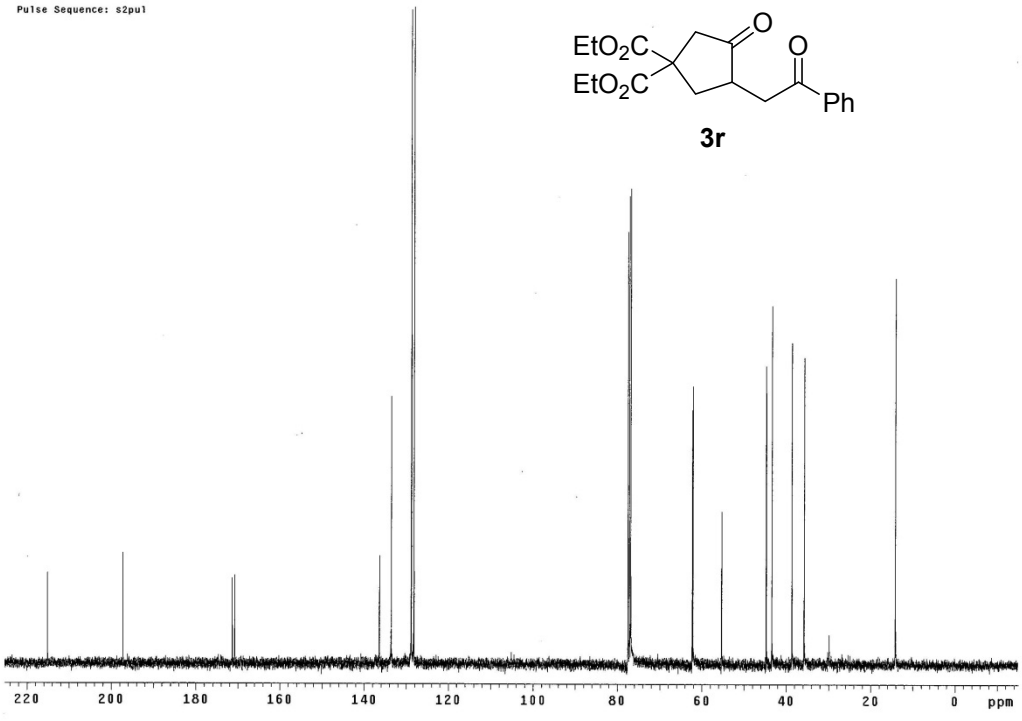


3r

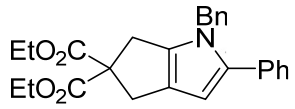


DVP-IV-30p-13C
File: xp
Pulse Sequence: s2pul

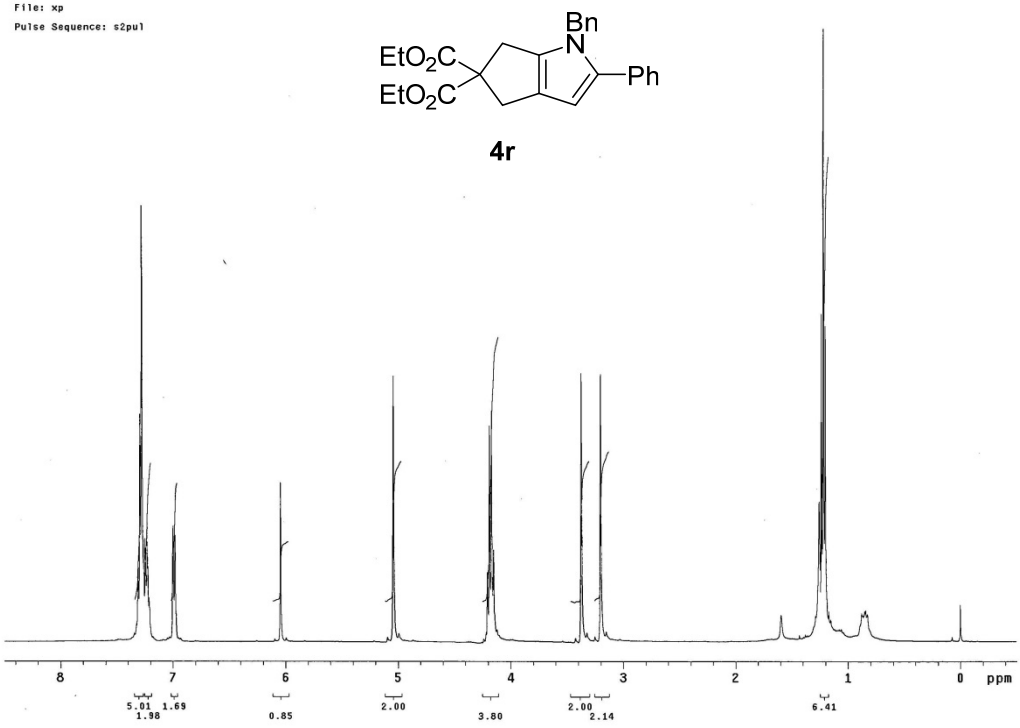
3r



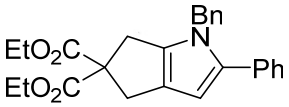
DVP-IV-39
File: xp
Pulse Sequence: s2pul



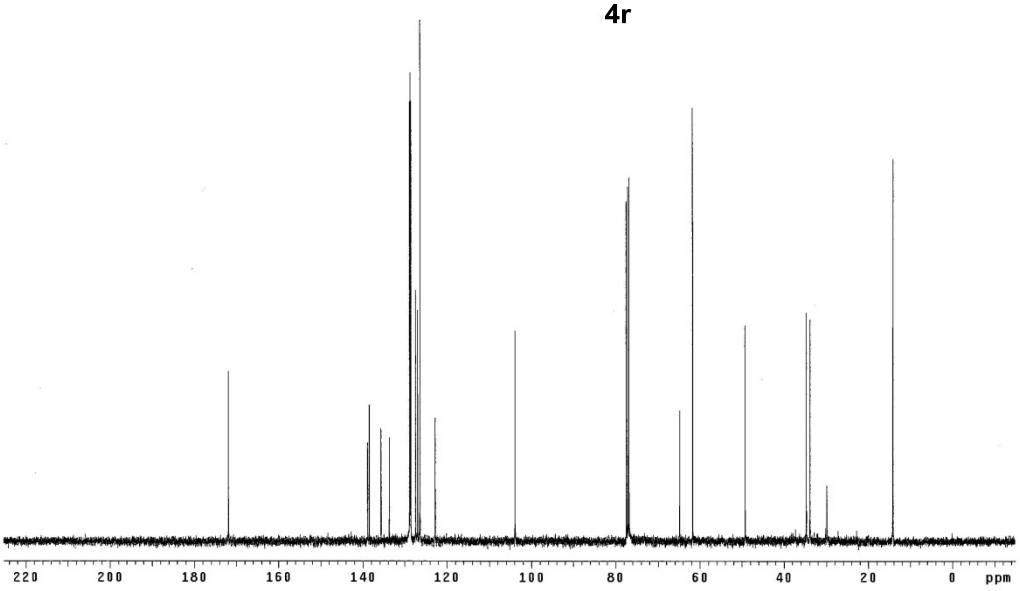
4r



DVP-IV-39-13C
File: xp
Pulse Sequence: s2pul

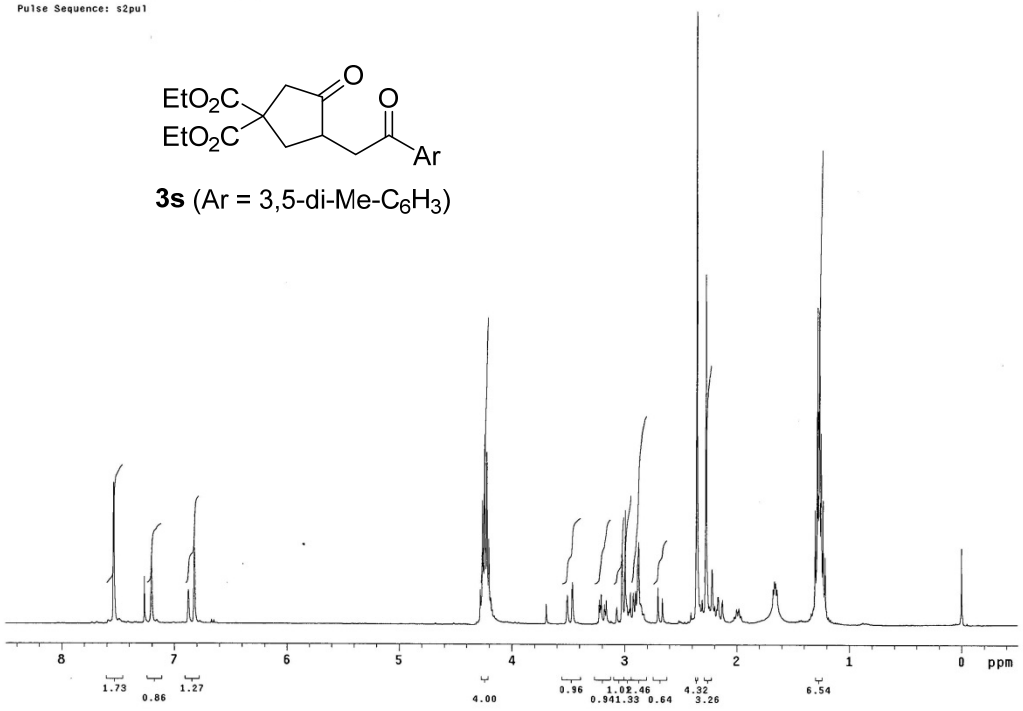


4r



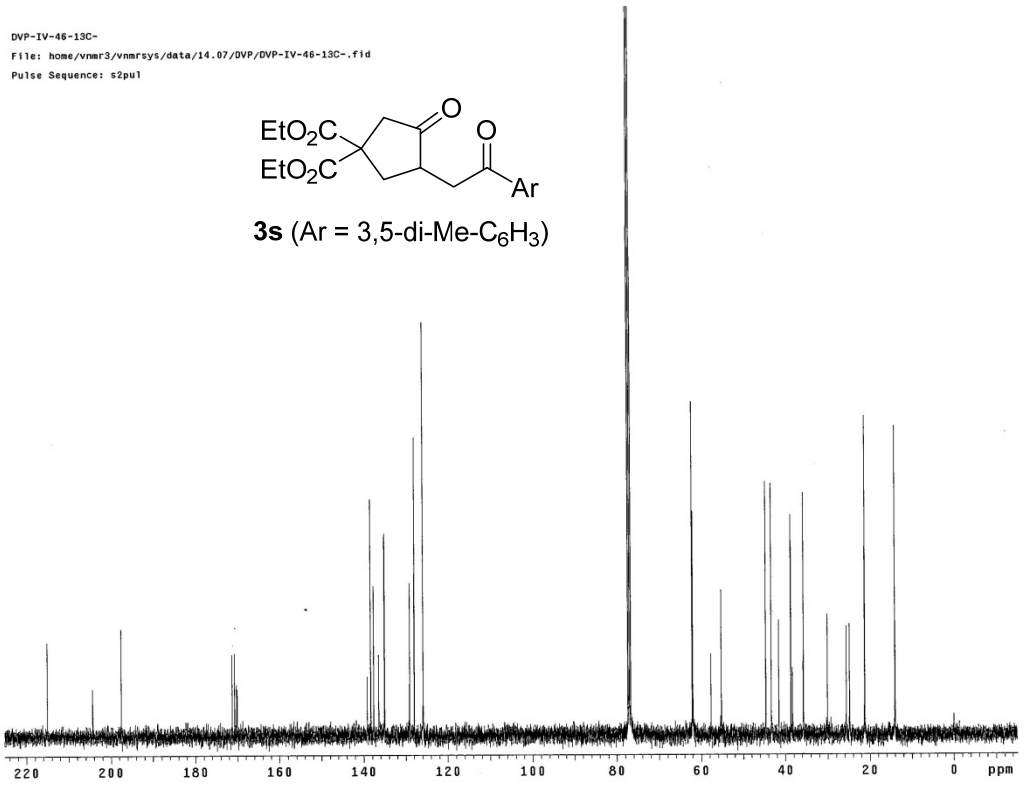
DVP-IV-46
File: home/vnmr3/vnmrsys/data/14.07/DVP/DVP-IV-46.fid
Pulse Sequence: s2pul

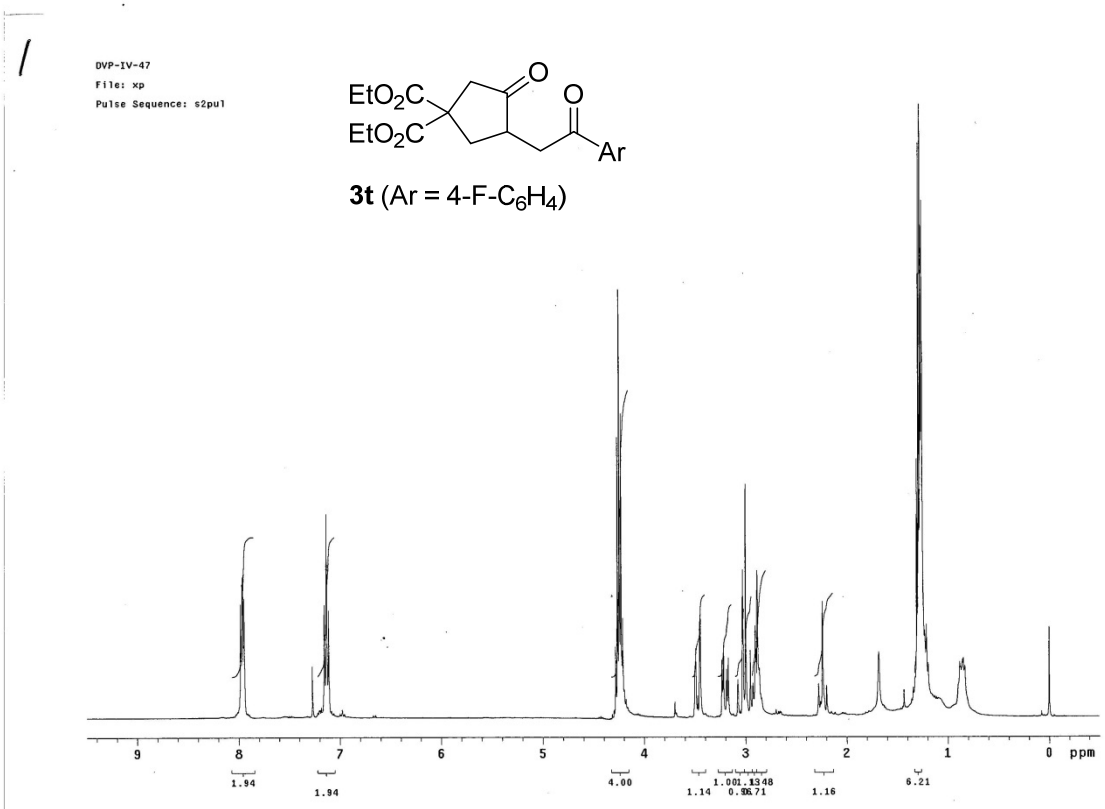
3s (Ar = 3,5-di-Me-C_6H_3)

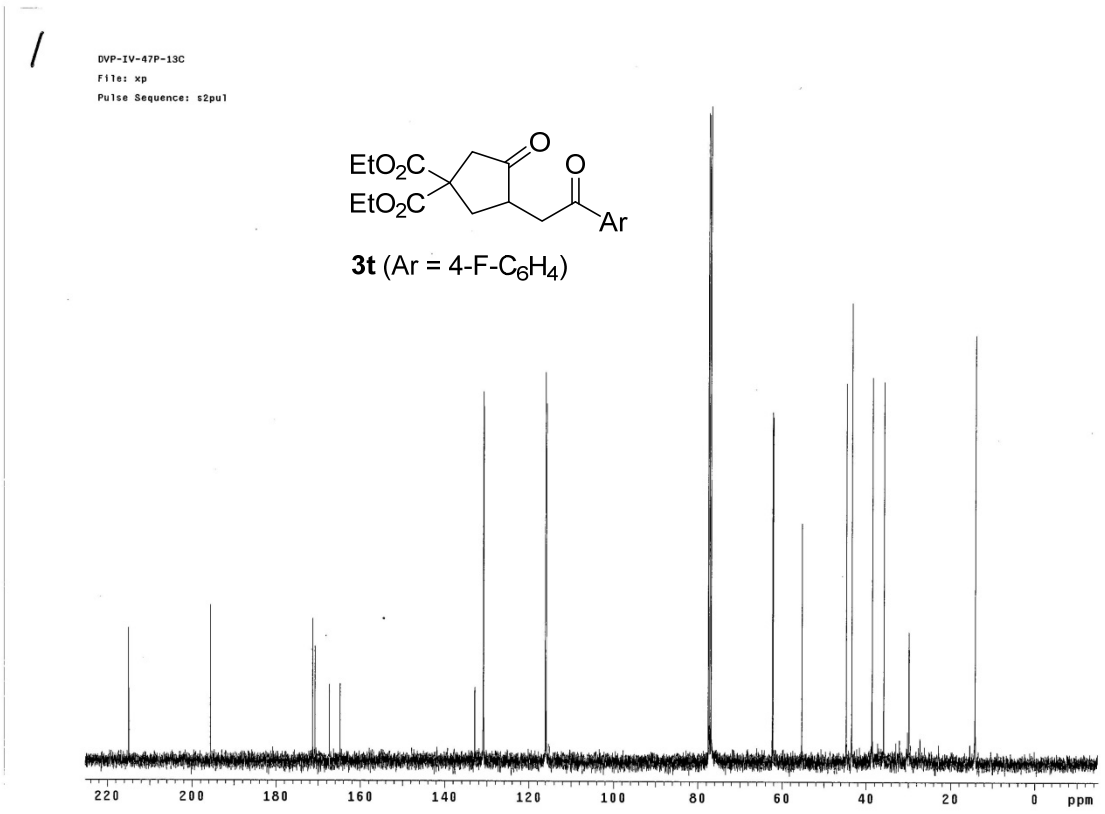


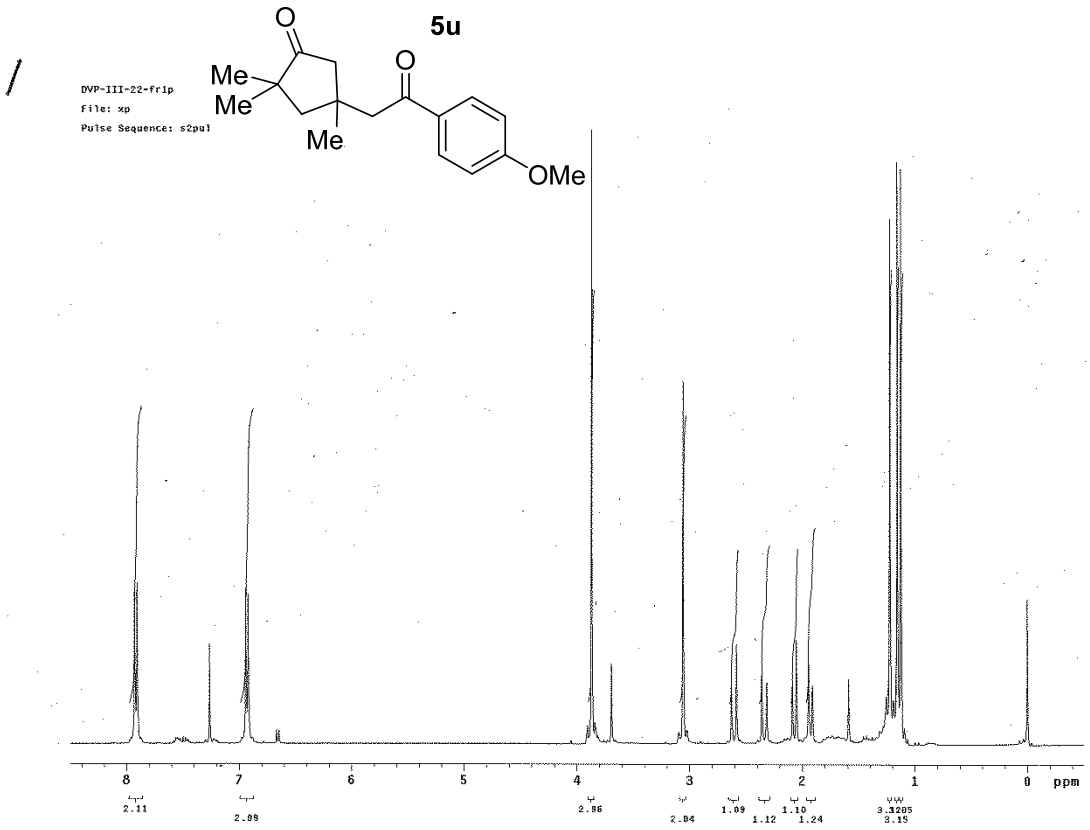
DVP-IV-46-13C-
File: home/vnmr3/vnmrsys/data/14.07/DVP/DVP-IV-46-13C-.fid
Pulse Sequence: s2pul

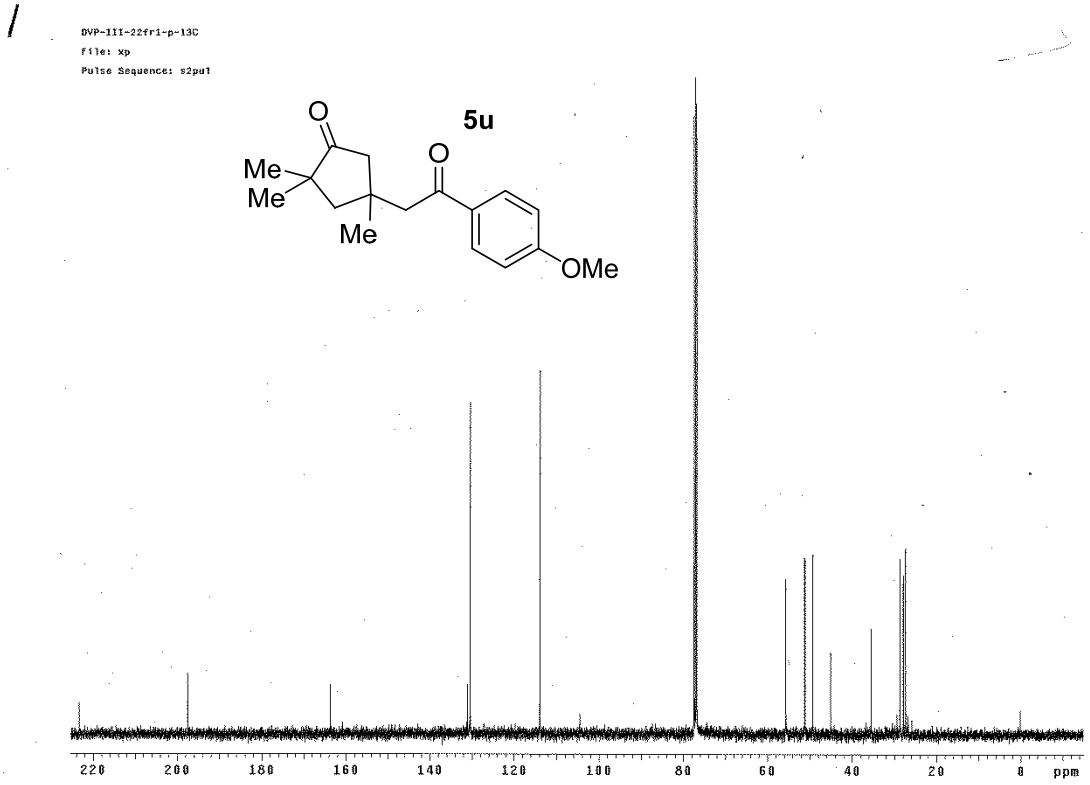
3s (Ar = 3,5-di-Me-C_6H_3)




DVP-IV-47
File: xp
Pulse Sequence: s2pul
EtO2C
EtO2C
O
O
Ar
3t (Ar = 4-F-C6H4)
9
8
7
6
5
4
3
2
1
0 ppm
1.94
1.94
4.00
1.14
1.16
6.21


DVP-IV-47P-13C
File: xp
Pulse Sequence: s2pul
EtO2C
EtO2C
O
O
Ar
3t (Ar = 4-F-C6H4)
220
200
180
160
140
120
100
80
60
40
20
0 ppm


5u
DVP-III-22-fr1p
file: xp
Pulse Sequence: s2pul
Me
Me
Me
O
O
OMe
8
7
6
5
4
3
2
1
0
ppm
2.11
2.09
2.86
2.84
1.09
1.12
1.10
1.24
3.1885
3.15


DVP-III-22fr1-p-13C
file: xp
Pulse Sequence: s2pul
5u
Me
Me
Me
O
O
OMe
220
200
180
160
140
120
100
80
60
40
20
0
ppm