One-Pot Synthesis of Aza-Diketopiperazines Enabled by Controlled Reactivity of *N*-Isocyanate Precursors

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General Information

Purification of reaction products was carried out by flash column chromatography using Silicycle silica gel (40-63 μm), unless otherwise noted. Analytical thin layer chromatography (TLC) was performed on aluminum, cut to size. Visualization was accomplished with UV light followed by staining with a potassium permanganate solution and heating.

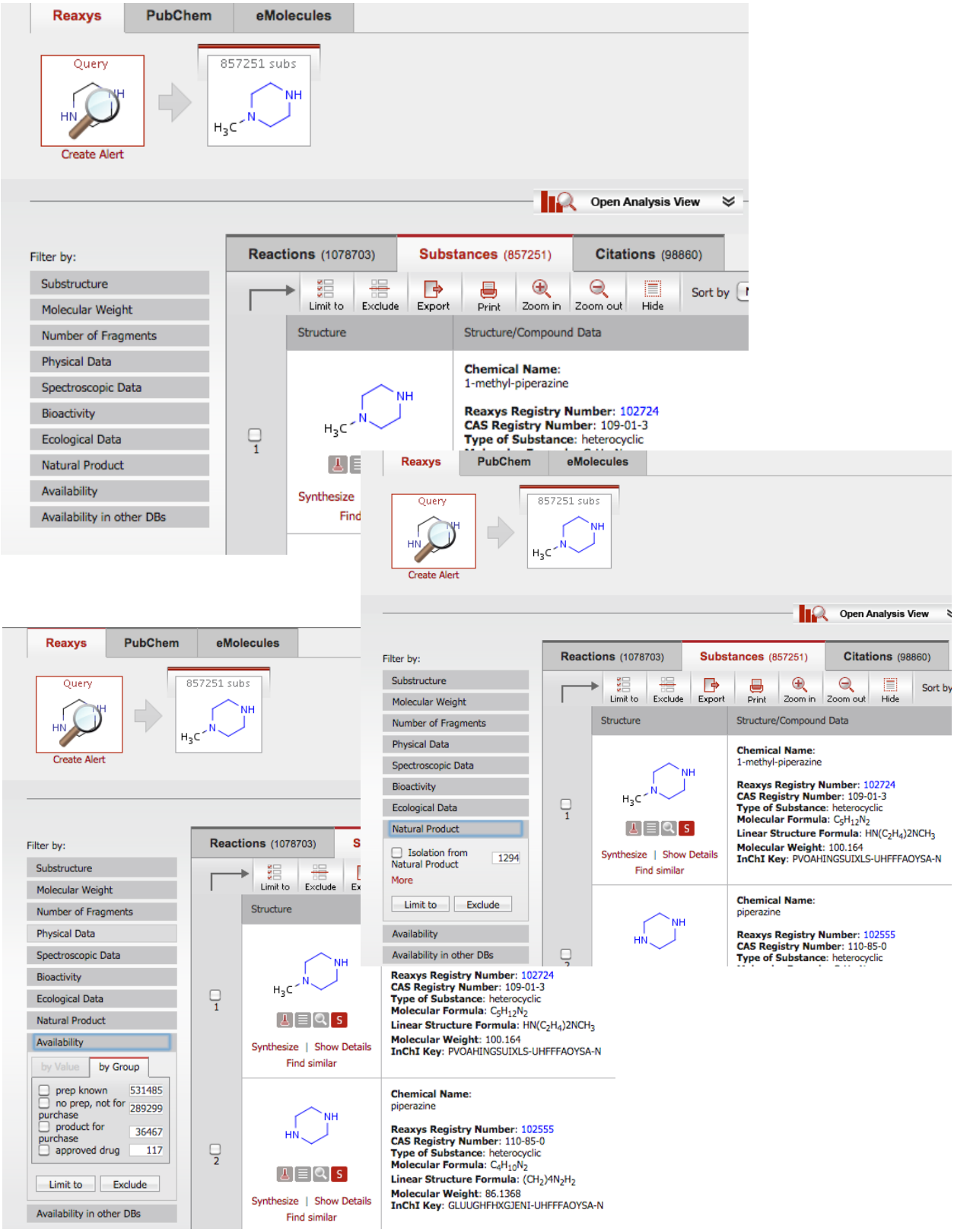
^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker AVANCE 300 MHz and 400 MHz spectrometers at ambient temperature, unless otherwise indicated. Spectral data was reported in ppm using solvent as the reference ($CDCl_3$ at 7.26 ppm, C_6D_6 at 7.15 ppm, or DMSO-d_6 at 2.50 ppm for ^{1}H NMR and $CDCl_3$ at 77.0 ppm or DMSO-d_6 at 39.43 for ^{13}C NMR). ^{1}H NMR data was reported as: multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, quint. = quintet, sext. = sextuplet, sept. = septuplet, m = multiplet), integration, and coupling constant (s) in Hz. Infrared (IR) spectra were obtained with neat thin films on a sodium chloride disk and were recorded on a Bomem Michelson 100 Fourier transform infrared spectrometer (FTIR). High-resolution mass spectroscopy (HRMS) was performed on a Kratos Concept-11A mass spectrometer with an electron beam of 70 eV at the Ottawa-Carleton Mass Spectrometry Centre.

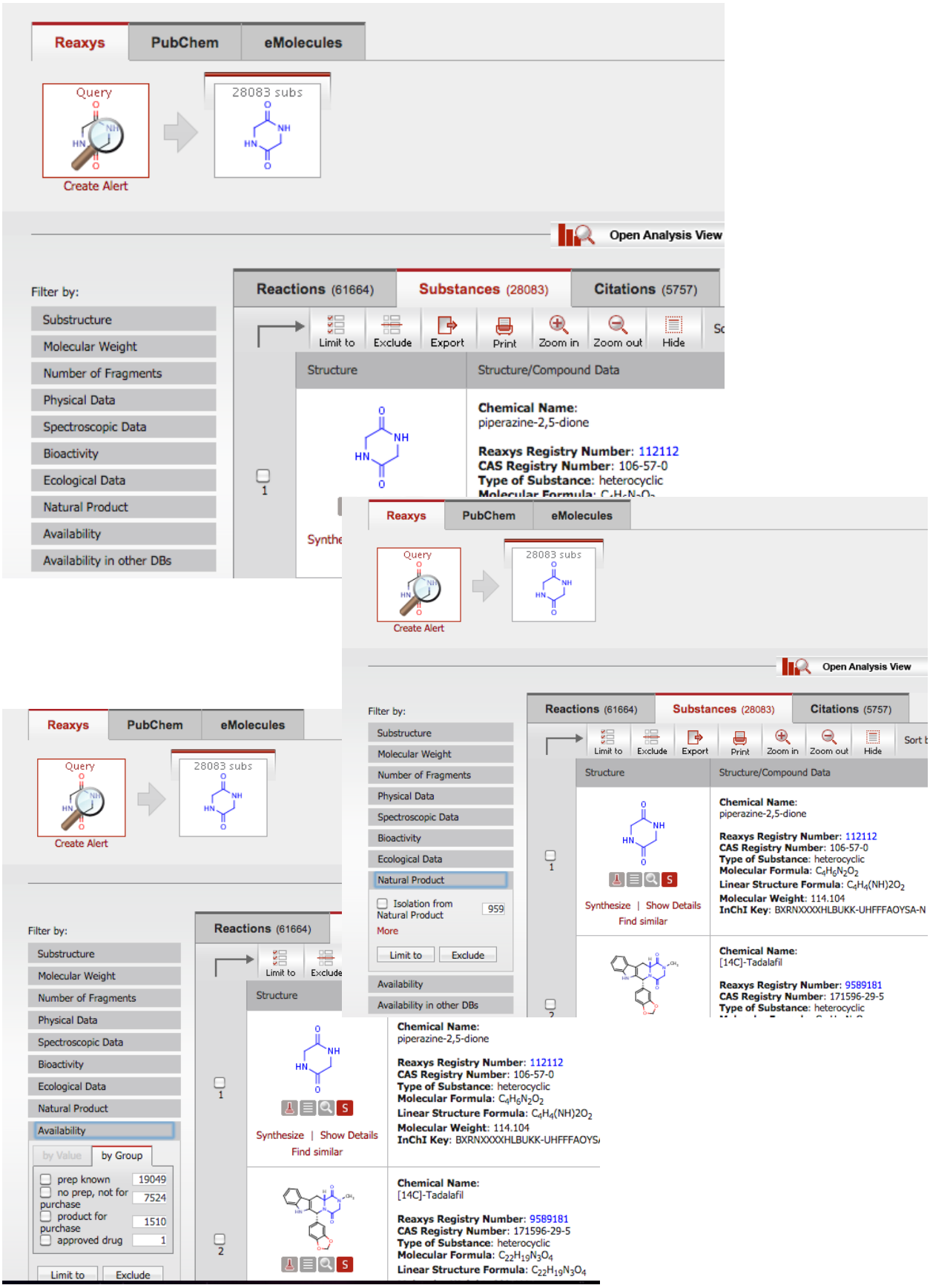
Materials

Unless otherwise noted, all commercially available materials were purchased from commercial sources and used without further purification.

Structural search (Figure 1)

Performed August 21, 2015 using Reaxys as a search engine.

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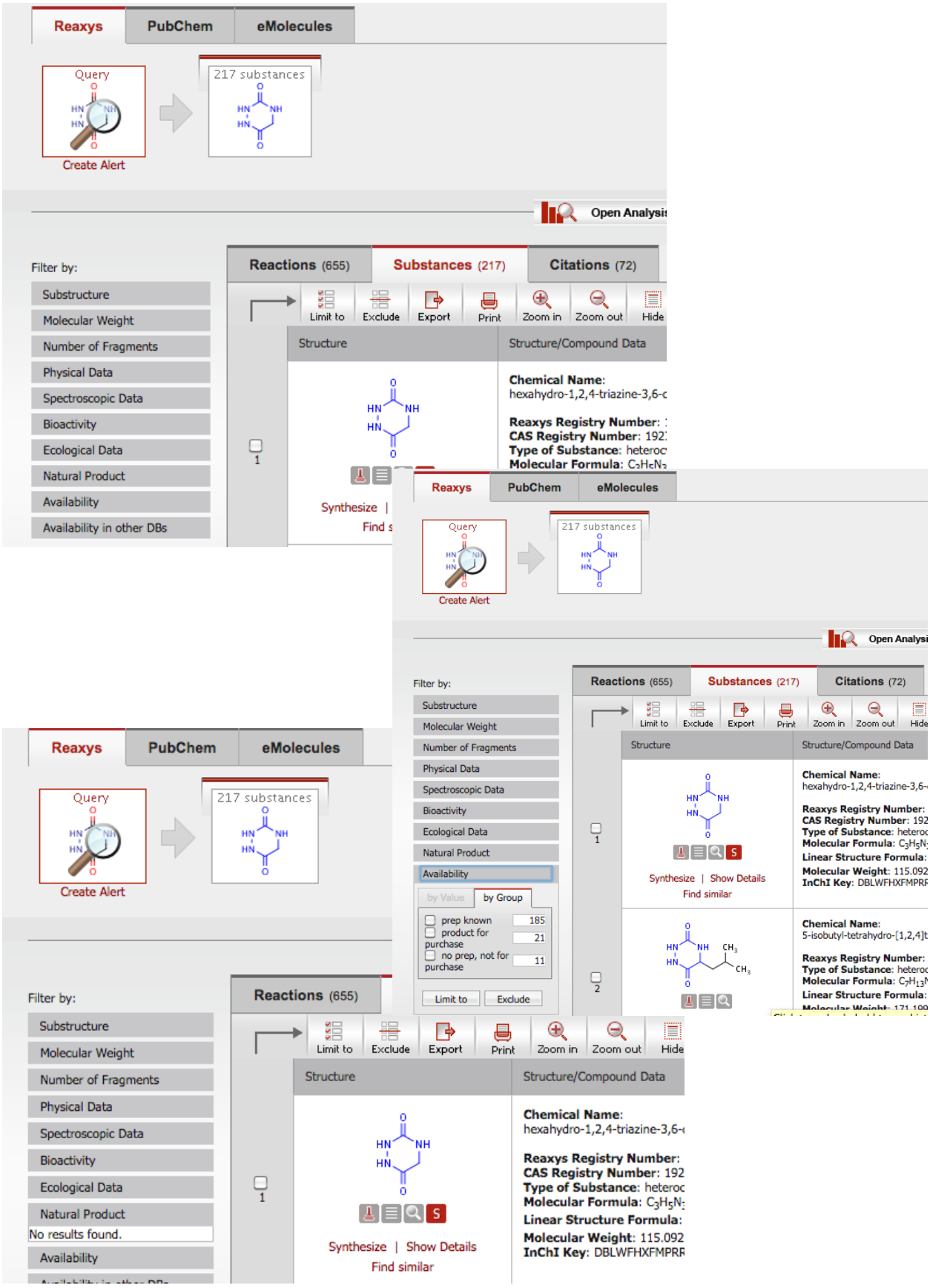
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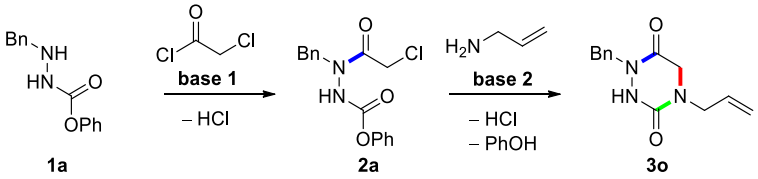
Table 1. Complete Reaction Optimization for One-Pot Sequence[a]

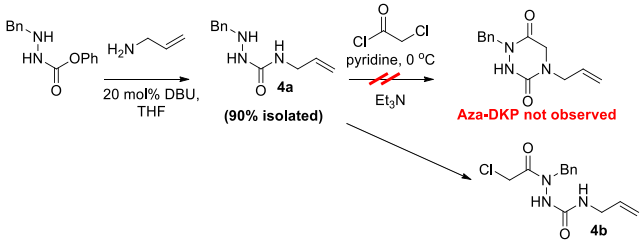


solvent	amine equiv	base 1	base 2	yield (%)
THF	1.1	Pyridine	Et_3N	44
MeCN	1.1	Pyridine	Et_3N	60
DCM	1.1	Pyridine	Et_3N	38
DMSO	1.1	Pyridine	Et_3N	35
DMF	1.1	Pyridine	Et_3N	33
THF	1.1	Pyridine	Et_3N	40[b]
MeCN	1.1	Pyridine	Et_3N	59[b]
MeCN	1.5	Pyridine	Et_3N	68
MeCN	**2.0**	**Pyridine**	**Et_3N**	**75[c]**
MeCN	2.0	DMAP	DMAP	11
MeCN	2.0	Pyridine	DMAP	37
MeCN	2.0	NMI	NMI	21
MeCN	2.0	Pyridine	NMI	46

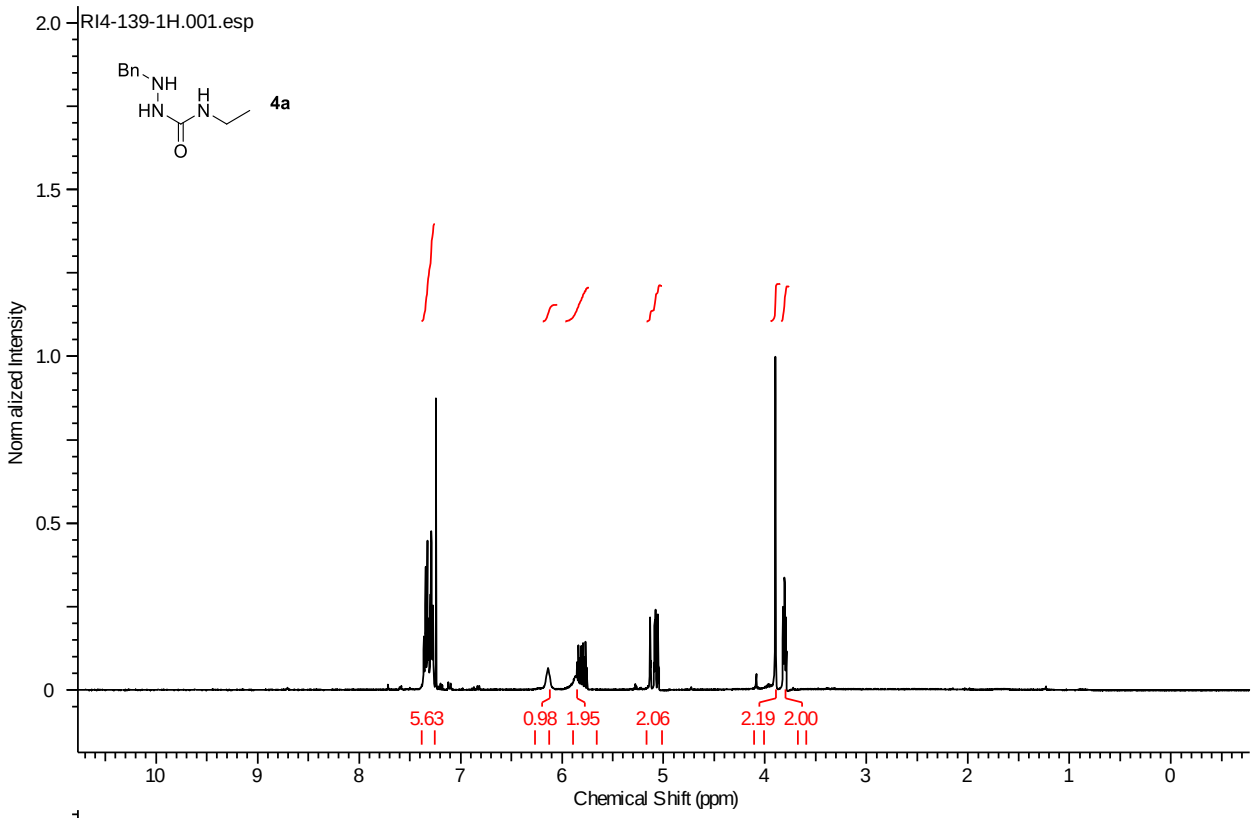
[a]Conditions: carbazate (1 equiv) stirring at 0 °C in MeCN (0.6 M); then **base 1** (1.1 equiv), then chloroacetyl chloride (1.05 equiv). The solution was warmed to room temp. and stirred for 2 hours. A solution of amine (1.1-2.0 equiv) and **base 2** (2.5 equiv) in MeCN was added to the reaction (0.3 M) and stirred at room temp. for 24 h. [b]1.0 equiv of NaI was added to the reaction before addition of amine/ **base 2**. Yields are NMR yields using 1,3,5-trimethoxybenzene as internal standard. [c]Isolated yield.

Control Experiment (Equation 1)

Scheme 1. Control Experiment for Path A vs. Path B



We inquired if intramolecular cyclization could occur from the resulting semi-carbazide following substitution of an amine on the *N*-isocyanate (see **Scheme 2** in communication, path B). To test this, we decided to implement known *N*-isocyanate substitution technology, 20 mol% DBU in THF, to afford the desired semi-carbazide. It should be noted that when this reaction was attempted in a one-pot fashion, extruded phenol from the generation of the *N*-isocyanate sequestered the chloroacetyl chloride reagent, thus impeding aza-DKP formation. Therefore, the semi-carbazide was isolated and then subjected to the reaction conditions — chloroacetyl chloride and pyridine for 2 h, then Et_3N for 24 h. No aza-DKP product was observed, instead the crude reaction mixture yielded only βN-acyl semicarbazide **4b**.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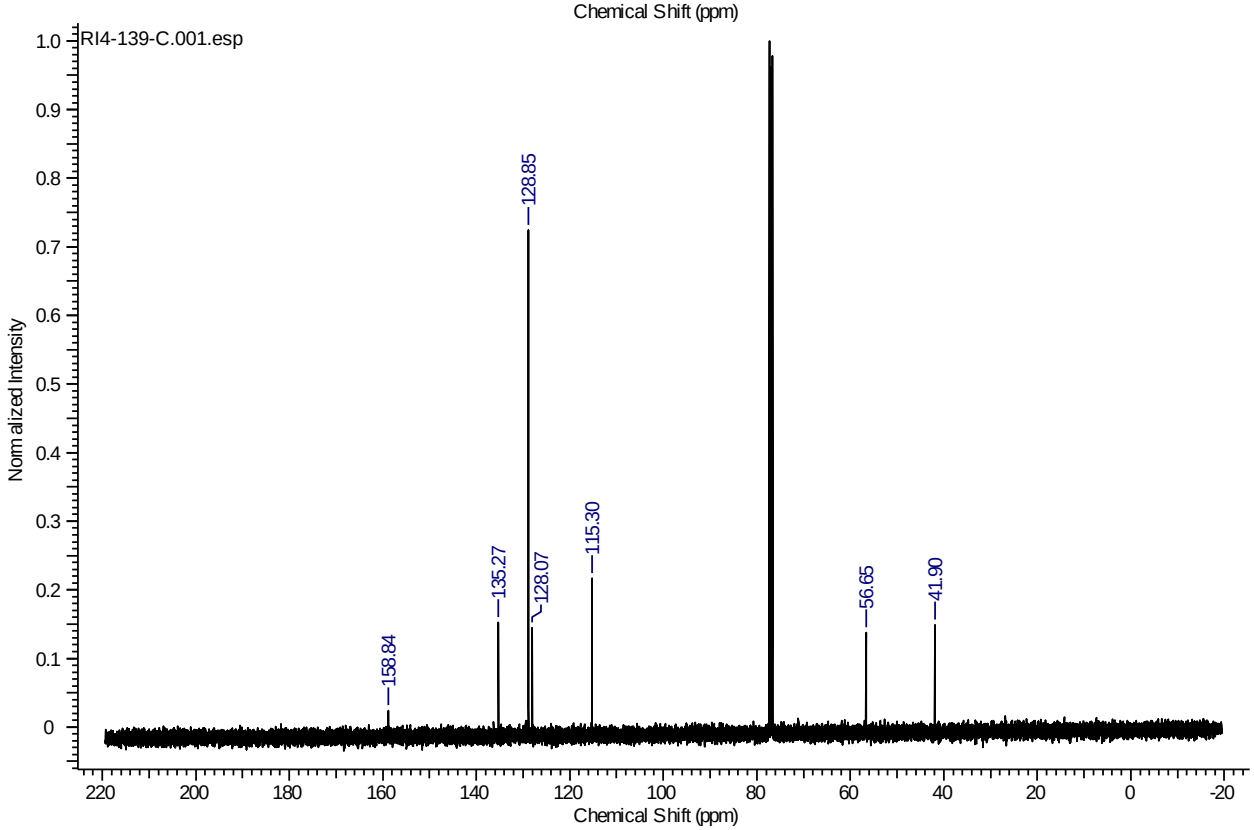
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One-Pot Aza-DKP Sequence: Amine Scope (Table 2)

General procedure 1A: To an oven-dried 10 mL glass vial were added benzyl carbazate **1a**[1] (1.0 equiv, 0.0968 g, 0.0400 mmol,) and MeCN (1.4 mL, 0.60 M). The solution was allowed to stir in an ice bath until reaching 0 ºC. To this solution was added pyridine (1.1 equiv, 0.0348 g, 0.0420 mmol), then chloroacetyl chloride (1.05 equiv, 0.0474 g, 0.0440 mmol). Immediately following the addition of these reagents, the reaction was removed from the ice bath and allowed to stir for 2 h. Subsequently, a solution of amine (2.0 equiv, 0.0800 mmol), and triethylamine (2.5 equiv, 0.102 g, 1.00 mmol) in MeCN were added to reach a final concentration of 0.30 M. The reaction was allowed to stir overnight at room temperature for 24 h. The reaction contents were concentrated under reduced pressure, and then an aqueous extraction with CH_2Cl_2 and 1.0 M HCl was used to remove excess amine and salts. The combined organic layers were dried with Na_2SO_4, concentrated under reduced pressure and purified by silica gel chromatography to give the corresponding aza-DKP.

General procedure 1B: To an oven-dried 10 mL glass vial were added benzyl carbazate **1a**[1] (1.0 equiv, 0.0968 g, 0.0400 mmol,) and CH_2Cl_2 (1.4 mL, 0.60 M). The solution was allowed to stir in an ice bath until reaching 0 ºC. To this solution was added pyridine (1.1 equiv, 0.0348 g, 0.0420 mmol), then chloroacetyl chloride (1.05 equiv, 0.0474 g, 0.0440 mmol). Immediately following the addition of these reagents, the reaction was removed from the ice bath and allowed to stir for 2 h. The reaction contents were concentrated under reduced pressure, and then an aqueous extraction with CH_2Cl_2 and 1.0 M HCl was used to remove salts. The combined organic layers were dried with Na_2SO_4, then concentrated under reduced pressure. The residue was dissolved in THF (0.30 M), then amine (1.1 equiv, 0.0440 mmol), and triethylamine (1.3 equiv, 0.0526 g, 0.520 mmol) were added. The reaction was refluxed in THF overnight for 16 h. The reaction contents were concentrated under reduced pressure and purified by silica gel chromatography to give the corresponding aza-DKP.

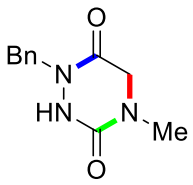


Table 2, entry 3a: 1-Benzyl-4-methyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using methylamine (2.0 M in THF solution, 0.400 mL, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2) to yield a white crystalline solid (0.0361 g, 41%). TLC Rf = 0.61 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.37 (br s, 1H), 7.26-7.38 (m, 5H), 4.66 (s, 2H), 3.89 (s, 2H), 2.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2 154.8 (C), 135.8 (C), 128.4 (CH), 127.6 (CH), 127.5 (CH), 50.3 (CH_2), 48.0 (CH_2), 33.1 (CH_3). IR (film): 3759, 3600, 3033, 1698, 1654, 1647, 1506, 1488, 1446, 1265 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{11}H_{13}N_3O_2$ $[M]^+$: 219.1008. Found: 219.1015.

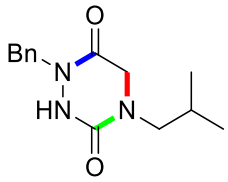


Table 2, entry 3b: 1-Benzyl-4-isobutyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using isobutylamine (0.0585 g, 0.800 mmol). The title compound was purified by column chromatography (30% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0634 g, 60%). TLC Rf = 0.52 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.28 (br s, 1H), 7.19-7.45 (m, 5H), 4.67 (s, 2H), 3.88 (s, 2H), 3.00 (d, *J* = 7.3 Hz, 2H), 1.83 (dt, *J* = 13.6, 6.8 Hz, 1H), 0.79 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7 (C), 155.2 (C), 135.9 (C), 128.4 (CH), 127.5 (CH), 127.4 (CH), 52.4 (CH_2), 49.2 (CH_2), 48.2 (CH_2), 26.1 (CH), 19.7 (CH_3). IR (film): 3710, 2968, 1695, 1650, 1500, 1466, 1423, 1267 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{14}H_{19}N_3O_2$ $[M]^+$: 261.1477. Found: 261.1481.

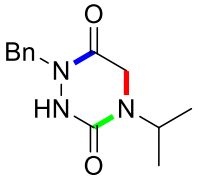


Table 2, entry 3c: 1-Benzyl-4-isopropyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using isopropylamine (0.0473 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0346 g, 35%). TLC Rf = 0.48 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 8.52 (br s, 1H), 7.28-7.42 (m, 1H), 7.02-7.81 (m, 6H), 4.76 (s, 2H), 4.43 (sept, *J* = 6.8 Hz, 1H), 3.78 (s, 2H), 1.15 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.5 (C), 155.5 (C), 134.3 (C), 128.8 (CH), 128.5 (CH), 128.3 (CH), 49.4 (CH), 45.0 (CH_2), 43.1 (CH_2), 19.1 (CH_3). IR (film): 3861, 3749, 2993, 1730, 1683, 1654, 1647, 1506, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{13}H_{17}N_3O_2$ $[M]^+$: 247.1321. Found: 247.1316.

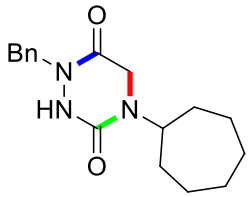


Table 2, entry 3d: 1-Benzyl-4-cycloheptyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using cycloheptylamine (0.0906 g, 0.800 mmol). The title compound was purified by column chromatography (30% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0253 g, 21%). TLC Rf = 0.62 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56 (br s, 1H), 7.29-7.39 (m, 5H), 4.77 (s, 2H), 4.16 (tt, *J* = 10.3, 4.0 Hz, 1H), 3.83 (s, 2H), 1.48-1.81 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 160.9 (C), 155.1 (C), 133.9 (C), 128.9 (CH), 128.5 (CH), 55.2 (CH), 49.5 (CH_2), 44.4 (CH_2), 32.0 (CH_2), 27.5 (CH_2), 24.9 (CH_2). IR (film): 3735, 2921, 2916, 2896, 1772, 1733, 1716, 1683, 1650, 1647, 1506, 1455, 1423, 1299, 1263, 1205, 1180 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{17}H_{23}N_3O_2$ $[M]^+$: 301.1790. Found: 301.1835.

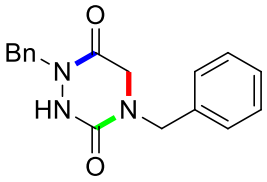


Table 2, entry 3e: 1,4-Dibenzyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using benzylamine (0.0857 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0850 g, 72%). TLC Rf = 0.25 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.28-7.39 (m, 10H), 4.81 (s, 2H), 4.51 (s, 2H), 3.88 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.7 (C), 155.3 (C), 135.2 (C), 134.2 (C), 128.9 (CH), 128.8 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 49.9 (CH_2), 49.5 (CH_2), 48.5 (CH_2). IR (film): 3834, 3735, 3687, 3049, 2322, 1663, 1662, 1642, 1506, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{17}H_{17}N_3O_2$ $[M]^+$: 295.1321. Found: 295.1300.

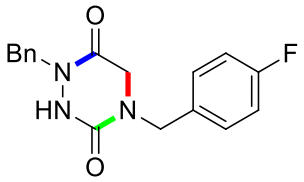


Table 2, entry 3f: 1-Benzyl-4-(4-fluorobenzyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using 4-fluorobenzylamine (0.100 g, 0.800 mmol). The title compound was purified by column chromatography (100% CH_2Cl_2 then 50% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0526 g, 42%). TLC Rf = 0.80 in 100% EtOAc. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.50 (br s, 1H), 7.21-7.35 (m, 7H), 7.11-7.17 (m, 2H), 4.64 (s, 2H), 4.34 (s, 2H), 3.76 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4 (C), 160.3 (C), 154.8 (C), 135.8 (C), 132.95 (C), 130.0 (CH), 129.9 (CH), 128.5 (CH), 127.4 (CH), 115.4 (CH), 115.2 (CH), 48.2 (CH_2), 48.2 (CH_2), 47.9 (CH_2). IR (film): 3735, 3649, 3004, 2354, 1733, 1683, 1642, 1506, 1455, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{17}H_{16}FN_3O_2$ $[M]^+$: 313.1227. Found: 313.1206.

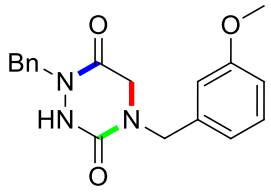


Table 2, entry 3g: 1-Benzyl-4-(3-methoxybenzyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using 3-methoxybenzylamine (0.102 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2 then 2% MeOH/CH_2Cl_2) to yield an amorphous white solid (0.0482 g, 37%). TLC Rf = 0.55 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.53 (br s, 1H), 7.26-7.35 (m, 6H), 6.87-6.80 (m, 3H), 4.68 (s, 2H), 4.37 (s, 2H), 3.79 (s, 2H), 3.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4 (C), 159.4 (C), 154.9 (C), 138.3 (C), 135.8 (C), 129.7 (CH), 128.5 (CH), 127.5 (CH), 127.3 (CH), 120.0 (CH), 113.5 (CH), 112.8 (CH), 55.0 (CH_3), 48.5 (CH_2), 48.3 (CH_2), 48.2 (CH_2). IR (film): 3726, 1791, 1772, 1733, 1684, 1662, 1647, 1488, 1455, 1212 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{18}H_{19}N_3O_3$ $[M]^+$: 325.1426. Found: 325.1442

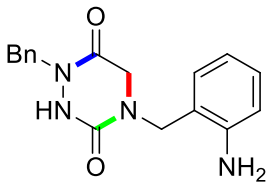


Table 2, entry 3h: 4-(2-Aminobenzyl)-1-benzyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using 2-aminobenzylamine (0.0977 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2 then 2% MeOH/CH_2Cl_2) to yield an amorphous white solid with minor impurities (presumably phenol). TLC Rf = 0.32 in 50% EtOAc/CH_2Cl_2. 50% yield determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as internal standard.

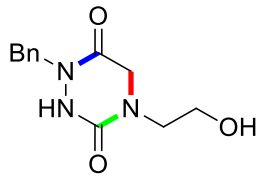


Table 2, entry 3i: 1-Benzyl-4-(2-hydroxyethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using ethanolamine (0.0489 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2 then 10% MeOH/CH_2Cl_2) to yield an amorphous white solid (0.0430 g, 43%). TLC Rf = 0.45 in 10% CH_3OH/CH_2Cl_2. ^{1}H NMR (300 MHz , DMSO-d_6,): δ ppm 9.30 (br s, 1H), 7.24-7.39 (m, 6H), 4.73 (t, *J* = 5.4 Hz, 1H), 4.67 (s, 2H), 3.98 (s, 2H), 3.51 (q, *J* = 5.5 Hz, 2H), 3.25 (t, *J* = 5.8 Hz, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.1 (C), 156.0 (C), 133.9 (C), 128.9 (CH), 128.5 (CH), 127.4 (CH), 60.5 (CH_2), 50.3 (CH_2), 49.5 (CH_2), 49.4 (CH). IR (film): 3683, 3010, 1868, 1739, 1683, 1652, 1533, 1458, 1419, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{15}N_3O_3$ $[M]^+$: 249.1113. Found: 249.1129.

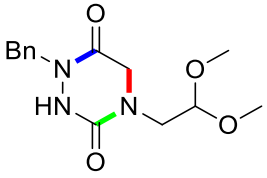


Table 2, entry 3j: 1-Benzyl-4-(2,2-dimethoxyethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using aminoacetaldehyde dimethyl acetal (0.0841 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0730 g, 62%). TLC Rf = 0.38 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ ppm 9.45 (br s, 1H), 7.22-7.39 (m, 5H), 4.67 (s, 2H), 4.48 (t, *J* = 5.5 Hz, 1H), 3.96 (s, 2H), 3.23-3.32 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7 (C), 155.2 (C), 135.9 (C), 128.4 (CH), 127.5 (CH), 127.4 (CH), 52.4 (CH_2), 49.2 (CH_2), 48.2 (CH_2), 26.1 (CH), 19.7 (CH_3). IR (film): 3735, 2935, 1683, 1662, 1647, 1506, 1456, 1265, 1191, 1118, 1047, 1051 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{14}H_{19}N_3O_4$ $[M]^+$: 293.1376. Found: 293.1379.

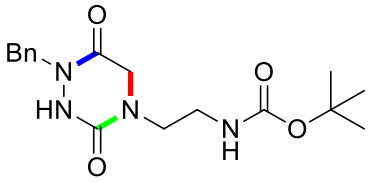


Table 2, entry 3k: *tert*-Butyl (2-(1-benzyl-3,6-dioxo-1,2,4-triazinan-4-yl)ethyl)carbamate: Synthesized according to general procedure **1A** using *N*-Boc-ethylenediamine (0.128 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0850 g, 61%). TLC Rf = 0.18 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.49 (br s, 1H), 7.31-7.39 (m, 5H), 4.89 (br s, 1H), 4.78 (s, 2H), 4.00 (s, 2H), 3.40-3.43 (m, 2H), 3.30-3.32 (m, 2H), 1.43 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7 (C), 155.2 (C), 135.9 (C), 128.4 (CH), 127.5 (CH), 127.4 (CH), 52.4 (CH_2), 49.2 (CH_2), 48.2 (CH_2), 26.1 (CH), 19.7 (CH_3). IR (film): 3735, 3666, 3461, 1718, 1683, 1588, 1647, 1506, 1436, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{17}H_{24}N_4O_4$ $[M]^+$: 348.1798. Found: 348.1790.

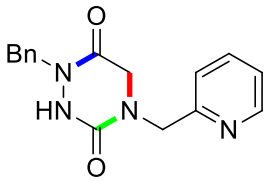


Table 2, entry 3l: 1-Benzyl-4-(pyridin-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using 2-picolylamine (0.0865 g, 0.800 mmol). The title compound was purified by column chromatography (100% EtOAc then 5% CH_3OH/CH_2Cl_2) to yield an amorphous white solid (0.0870 g, 73%). TLC Rf = 0.18 in 5% CH_3OH/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.52 (br s, 1H), 8.46-8.58 (m, 1H), 7.76 (td, *J* = 7.69, 1.86 Hz, 1H), 7.26-7.43 (m, 6H), 7.22 (d, *J* = 7.8 Hz, 1H), 4.70 (s, 2H), 4.51 (s, 2H), 3.99 (s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.4 (C), 156.7 (C), 154.8 (C), 136.8 (C), 135.8 (CH), 128.5 (CH), 127.6 (CH), 127.5 (CH), 122.4 (CH), 121.5 (CH), 50.5 (CH_2), 49.4 (CH_2), 48.2 (CH_2). IR (film): 3735, 3670, 3035, 2349, 1733, 1683, 1652, 1647, 1525, 1506, 1455, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{16}H_{16}N_4O_2$ $[M]^+$: 296.1273. Found: 296.1266.

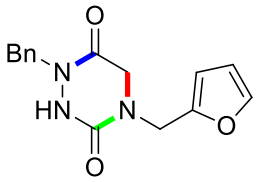


Table 2, entry 3m: 1-Benzyl-4-(furan-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using furfurylamine (0.0777 g, 0.800 mmol). The title compound was purified by column chromatography (20% EtOAc/CH_2Cl_2 to 50% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0719 g, 63%). TLC Rf = 0.62 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.62 (dd, *J* = 1.86, 0.9 Hz, 1H), 7.20-7.40 (m, 5H), 6.42 (dd, *J* = 3.14, 2.0 Hz, 1H), 6.35 (dd, *J* = 3.23, 0.5 Hz, 1H), 4.67 (s, 2H), 4.41 (s, 2H), 3.83 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7 (C), 155.2 (C), 135.9 (C), 128.4 (CH), 127.5 (CH), 127.4 (CH), 52.4 (CH_2), 49.2 (CH_2), 48.2 (CH_2), 26.1 (CH), 19.7 (CH_3). IR (film): 3861, 3736, 3649, 1739, 1683, 1652, 1647, 1506, 1456, 1265 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{15}N_3O_3$ $[M]^+$: 285.1113. Found: 285.1115.

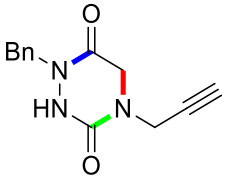


Table 2, entry 3n: 1-Benzyl-4-(prop-2-yn-1-yl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1B** using propargylamine (0.0242 g, 0.440 mmol). The title compound was purified by column chromatography (30% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0691 g, 71%). TLC Rf = 0.30 in 30% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm 8.95 (br s, 1H), 7.21-7.29 (m, 5H), 4.70 (s, 2H), 4.02 (d, *J* = 3.5 Hz, 2H), 3.93 (s, 2H), 2.27 (t, *J* = 2.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.6 (C), 154.7 (C), 134.1 (C), 128.7 (CH), 128.6 (CH), 128.3 (CH), 76.7 (C), 72.4 (CH), 49.4 (CH_2), 48.2 (CH_2), 35.5 (CH_2). IR (film): 3722, 3687, 3629, 3056, 2221, 1868, 1772, 1730, 1683, 1647, 1506, 1395. HRMS (EI): Exact mass calcd for $C_{13}H_{13}N_3O_2$ $[M]^+$: 243.1008. Found: 243.1023.

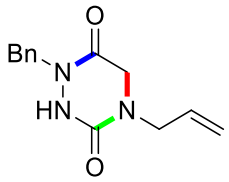


Table 2, entry 3o: 4-Allyl-1-benzyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using allylamine (0.0460 g, 0.800 mmol). The title compound was purified by column chromatography (30% EtOAc/CH_2Cl_2) to yield an amorphous white solid (0.0736 g, 75%). TLC Rf = 0.40 in 30% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 8.80 (s, 1H), 7.26-7.39 (m, 5H), 5.73 (ddt, *J* = 16.8, 10.5, 6.1 Hz, 1H), 5.14-5.31 (m, 2H), 4.76 (s, 2H), 3.88 (d, *J* = 6.1 Hz, 2H), 3.83 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.8 (C), 155.2 (C), 134.2 (C), 131.4 (CH), 128.7 (CH), 128.5 (CH), 128.3 (CH), 119.3 (CH_2), 49.4 (CH_2), 48.7 (CH_2), 48.4 (CH_2). IR (film): 3157, 3056, 2939, 1695, 1643, 1608, 1456, 1419, 1303, 1267, 1207, 1186 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{13}H_{15}N_3O_2$ $[M]^+$: 245.1164. Found: 245.1190.

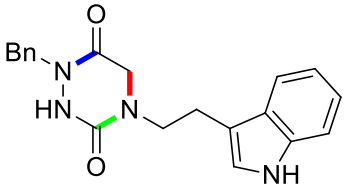


Table 2, entry 3p: 4-(2-(1H-Indol-3-yl)ethyl)-1-benzyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using tryptamine (0.128 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/CH_2Cl_2 then 2% CH_3OH/CH_2Cl_2) to yield an amorphous white solid (0.0795 g, 57%). TLC Rf = 0.25 in 2% CH_3OH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 8.03 (br s, 1H), 7.73 (br s, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.26-7.37 (m, 6H), 7.18 (td, *J* = 7.6, 1.1 Hz, 1H), 7.09 (td, *J* = 7.6, 1.1 Hz, 1H), 6.97 (d, *J* = 2.3 Hz, 1H), 4.70 (s, 2H), 3.78 (s, 2H), 3.59 (t, *J* = 7.3 Hz, 2H), 3.02 (t, *J* = 7.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.5 (C), 154.7 (C), 136.2 (C), 135.9 (C), 128.4 (CH), 127.4 (CH), 127.1 (C), 122.7 (C), 120.9 (C), 118.3 (C), 118.3 (C), 111.4 (C), 111.1 (CH), 48.7 (CH_2), 48.0 (CH_2), 46.3 (CH_2), 22.6 (CH_2), 19.7 (CH_3). IR (film): 3735, 3670, 1739, 1683, 1644, 1539, 1506, 1456, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{20}H_{20}N_4O_2$ $[M]^+$: 348.1586. Found: 348.1598.

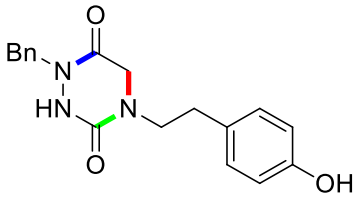


Table 2, entry 3q: 1-Benzyl-4-(4-hydroxyphenethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using tyramine (0.109 g, 0.800 mmol). The title compound precipitated out of solution in 100% CH_2Cl_2 as a white solid to give pure product after aqueous extraction (0.0547 g, 42%). TLC Rf = 0.33 in 5% CH_3OH/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm 9.31 (br s, 1H), 9.20 (br s, 1H), 7.32 (s, 3H), 7.22 (s, 2H), 6.98 (d, J = 8.4 Hz, 2H), 6.66 (d, J = 8.4 Hz, 2H), 4.64 (s, 2H), 3.86 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4 (C), 155.7 (C), 154.6 (C), 135.8 (C), 129.5 (CH), 128.9 (C), 128.4 (CH), 127.5 (CH), 127.4 (CH), 115.2 (CH), 48.7 (CH_2), 48.0 (CH_2), 47.2 (CH_2), 31.8 (CH_2). IR (film): 3853, 3735, 3020, 2347, 1739, 1683, 1648, 1525, 1506, 1456, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{18}H_{19}N_3O_3$ $[M]^+$: 325.1426. Found: 325.1428.

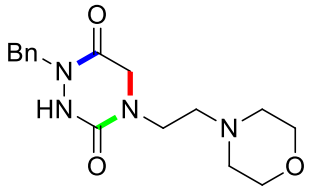
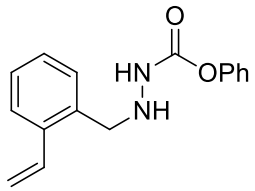


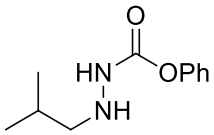
Table 2, entry 3r: 1-Benzyl-4-(2-morpholinoethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using 4-(2-aminoethyl)morpholine (0.104 g, 0.800 mmol). The title compound was purified by column chromatography (100% EtOAc/CH_2Cl_2 then 2% CH_3OH/CH_2Cl_2) to yield an amorphous white solid (0.0980 g, 77%). TLC Rf = 0.12 in 2% CH_3OH/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.31 (br s, 1H), 7.22-7.37 (m, 5H), 4.66 (s, 2H), 3.97 (s, 2H), 3.53 (t, J = 4.5 Hz, 4H), 3.31 (t, J = 6.4 Hz, 2H), 2.29-2.45 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.5 (C), 154.7 (C), 135.9 (C), 128.4 (CH), 127.5 (CH), 66.3 (CH_2), 55.4 (CH_2), 53.3 (CH_2), 49.1 (CH_2), 48.1 (CH_2), 42.3 (CH_2). IR (film): 3735, 2943, 1772, 1733, 1716, 1683, 1647, 1515, 1488, 1456, 1338, 1263, 1244, 1188, 1114, 1070 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{16}H_{22}N_4O_3$ $[M]^+$: 318.1692. Found: 318.1688.

Carbazate Synthesis (S.M. for Table 3)

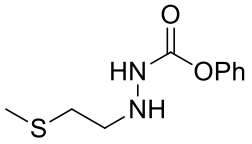
General Procedure 2 (Preparation of substrates): *Carbazate Condensation*: Prepared according to a modified procedure from the Leighton group.[2] To a solution of the corresponding aldehyde or ketone in MeOH (0.3 M) was added *O*-phenyl carbazate (1.00 equiv). The reaction was carried out at room temperature (aldehyde) or refluxed in MeOH (ketone) until consumption of the aldehyde or ketone was judged complete by TLC. The corresponding crude hydrazones were directly reduced without further purification. *Hydrazone Reduction*: Prepared from a modified procedure by Lane.[3] The corresponding hydrazone was diluted in 2:1 MeOH:AcOH (0.2 M) and combined with $NaBH_3CN$ (3 equiv). The reaction mixture was stirred for 16 h. Methanol and excess acetic acid were evaporated and the mixture was dissolved in EtOAc. The pH was increased using saturated sodium bicarbonate solution to reach a pH of 8. The solution was extracted three times with EtOAc and then brine and dried over Na_2SO_4. Volatiles were removed *in-vacuo* and the crude product was purified by column chromatography or filtration.



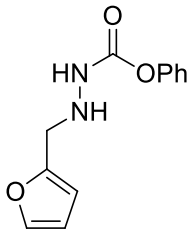
Carbazate 1b: Phenyl 2-(2-vinylbenzyl)hydrazinecarboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (1.33 g, 8.80 mmol) and 2-vinylbenzaldehyde[4] (1.22 g, 9.23 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (1.66 g, 26.4 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (2.5% EtOAc/CH_2Cl_2) to yield an amorphous white solid (1.64 g, 70%). TLC Rf = 0.41 in 2.5% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm 7.55-7.62 (m, 1H), 7.13-7.45 (m, 10H), 5.74 (dd, *J* = 17.5, 1.2 Hz, 1H), 5.39 (dd, *J* = 10.9, 1.2 Hz, 1H), 4.20 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5 (C), 150.6 (C), 137.6 (C), 133.9 (CH), 130.6 (CH), 129.4 (CH), 128.2 (CH), 127.8 (CH), 125.8 (CH), 125.6 (CH), 121.4 (CH), 116.4 (CH_2), 83.8 (C), 53.0 (CH_2). IR (film): 3188, 1683, 1662, 1555, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{16}H_{16}N_2O_2$ $[M]^+$: 268.1212. Found: 268.1257.



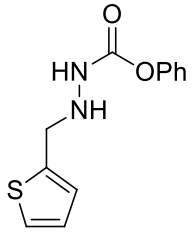
Carbazate 1c: Phenyl 2-isobutylhydrazinecarboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (3.13 g, 20.6 mmol) and isobutyraldehyde (1.49 g, 20.6 mmol) at room temperature in MeOH (0.3M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.88 g, 61.8 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (2.5% EtOAc/CH_2Cl_2 then 10% EtOAc/CH_2Cl_2) to yield an amorphous white solid (3.24 g, 76%). TLC Rf = 0.15 in 100% CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm 7.32-7.42 (m, 2H), 7.18-7.25 (m, 1H), 7.11-7.18 (m, 2H), 2.77 (d, *J* = 6.9 Hz, 2H), 1.71-1.89 (m, 1H), 0.97 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.6 (C), 129.6 (C), 129.2 (CH), 125.4 (CH), 121.3 (CH), 59.5 (CH_2), 26.7 (CH), 20.4 (CH_3). IR (film): 3350, 2970, 1718, 1652, 1533, 1490, 1455, 1266, 1238, 1203, 1163 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{11}H_{16}N_2O_2$ $[M]^+$: 208.1212. Found: 208.1225.



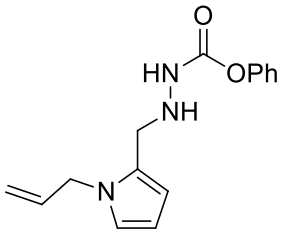
Carbazate 1e: Phenyl 2-(3-(methylthio)propyl)hydrazine-1-carboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (2.68 g, 17.6 mmol) and 3 (methylthio)propanal (1.83 g, 17.6 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.31 g, 52.8 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (80% Hexane/EtOAc) to yield an amorphous white solid (0.200 g, 5%). TLC Rf = 0.59 in 50% Hexane/EtOAc. ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm 7.33-7.43 (m, 2H), 7.19-7.26 (m, 1H), 7.12-7.19 (m, 2H), 3.13 (t, *J* = 6.9 Hz, 2H), 2.61 (t, *J* = 7.1 Hz, 2H), 2.13 (s, 3H), 1.88 (quin, *J* = 7.02 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5 (C), 150.6 (C), 129.3 (CH), 125.5 (CH), 121.3 (CH), 50.5 (CH_2), 31.7 (CH_2), 27.0 (CH_2), 15.5 (CH_3). IR (film): 3853, 3836, 3735, 3722, 3712, 3670, 3627, 1874, 1801, 1772, 1733, 1699, 1662, 1647, 1506, 1456, 1361, 1263, 1201, 1163 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{11}H_{16}N_2O_2S$ $[M]^+$: 240.0932. Found: 111.09289.



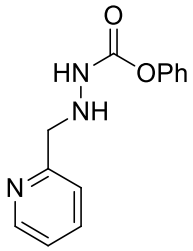
Carbazate 1f: Phenyl 2-(furan-2-ylmethyl)hydrazine-1-carboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (2.60 g, 17.1 mmol) and furan-2-carbaldehyde (1.64 g, 17.1 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.22 g, 51.3 mmol) was added and allowed to stir for 16 h.The title compound was purified by column chromatography (100% CH_2Cl_2) to yield an amorphous white solid (3.48 g, 87%). TLC Rf = 0.20 in 100% CH_2Cl_2. ^1H NMR (400 MHz, $CDCl_3$) δ ppm 7.43 (dd, *J* = 1.76, 0.8 Hz, 1H), 7.33-7.41 (m, 2H), 7.19-7.26 (m, 1H), 7.11-7.17 (m, 2H), 6.36 (dd, *J* = 3.1, 2.0 Hz, 1H), 6.31 (d, *J* = 2.7 Hz, 1H), 4.11 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6 (C), 151.3 (C), 150.9 (C), 142.9 (CH), 129.6 (CH), 125.8 (CH), 121.6 (CH), 110.6 (CH), 109.1 (CH), 48.3 (CH_2). IR (film): 3674, 2364, 1733, 1655, 1506, 1490, 1455, 1265, 1203 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{12}N_2O_3$ $[M]^+$: 232.0848. Found: 232.08262.



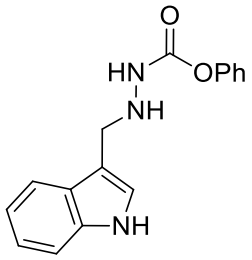
Carbazate 1g: Phenyl 2-(thiophen-2-ylmethyl)hydrazine-1-carboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (2.57 g, 16.9 mmol) and thiophene-2-carbaldehyde (1.89 g, 16.9 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.18 g, 50.7 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (80% Hexane/EtOAc) to yield an amorphous white solid (2.63 g, 63%). TLC Rf = 0.78 in 50% Hexane/EtOAc. ^1H NMR (400 MHz, $CDCl_3$) δ ppm 7.35-7.42 (m, 2H), 7.30 (dd, *J* = 5.00, 1.3 Hz, 1H), 7.20-7.26 (m, 1H), 7.12-7.17 (m, 2H), 7.02-7.05 (m, 1H), 6.98-7.02 (m, 1H), 4.32 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3 (C), 150.5 (C), 140.0 (C), 129.3 (CH), 126.9 (CH), 126.7 (CH), 125.6 (CH), 125.5 (CH), 121.3 (CH), 49.9 (CH_2). IR (film): 3751, 3687, 1743, 1731, 1696, 1652, 1558, 1533, 1506, 1456, 1266, 1205, 1164 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{12}N_2O_2S$ $[M]^+$: 248.0619. Found: 248.06037.



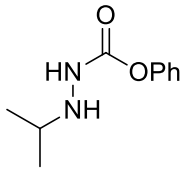
Carbazate 1h: Phenyl 2-((1-allyl-1*H*-pyrrol-2-yl)methyl)hydrazinecarboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (2.99 g, 19.6 mmol) and 1-allyl-1*H*-pyrrole-2-carbaldehyde (2.65 g, 19.6 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.70 g, 58.9 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (100% CH_2Cl_2 then 5% EtOAc/CH_2Cl_2) to yield an amorphous white solid (3.75 g, 74%). TLC Rf = 0.33 in 2.5% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm 7.36-7.45 (m, 2H), 7.21-7.29 (m, 1H), 7.12-7.21 (m, 2H), 6.70 (dd, *J* = 2.8, 1.9 Hz, 1H), 6.17-6.22 (m, 1H), 6.12-6.17 (m, 1H), 6.02 (ddt, *J* = 17.1, 10.3, 5.2, Hz, 1H), 5.19 (dq, *J* = 10.3, 1.6 Hz, 1H), 4.98 (dq, *J* = 17.1, 1.6 Hz, 1H), 4.64 (dt, *J* = 5.0, 1.7 Hz, 2H), 4.07 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4 (C), 150.5 (C), 134.9 (CH), 129.3 (CH), 127.2 (C), 125.5 (CH), 122.2 (CH), 121.3 (CH), 116.3 (CH_2), 110.0 (CH), 107.1 (CH), 49.0 (CH_2), 46.7 (CH_2). IR (film): 3729, 3654, 2981, 1731, 1699, 1539, 1515, 1490, 1460, 1444, 1265, 1203 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{17}N_3O_2$ $[M]^+$: 271.1321. Found: 271.1257.



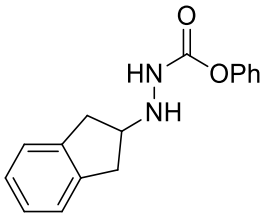
Carbazate i: Phenyl 2-(pyridin-2-ylmethyl)hydrazinecarboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (1.89 g, 17.6 mmol) and picolinaldehyde (2.64 g, 17.6 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.32 g, 52.8 mmol) was added and allowed to stir for 16 h. The title compound was filtered after reduction, washed with EtOAc and dried under vacuum to yield an amorphous white solid (2.57 g, 60%). TLC Rf = 0.20 in 2% CH_3OH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm 8.58 (d, *J* = 4.8 Hz, 1H), 7.66 (td, *J* = 7.7, 1.8 Hz, 1H), 7.25-7.38 (m, 3H), 7.17-7.23 (m, 2H), 7.11 (d, *J* = 7.8 Hz, 2H), 4.24 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.2 (C), 154.7 (C), 148.4 (CH), 136.1 (CH), 129.0 (CH), 124.7 (CH), 121.8 (CH), 121.7 (CH), 121.2 (CH), 112.2 (C), 55.5 (CH_2). IR (film): 3555, 3024, 2333, 1728, 1683, 1666, 1541, 1498, 1140, 1266, 1201 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{13}H_{13}N_3O_2$ $[M]^+$: 243.1008. Found: 243.0947.



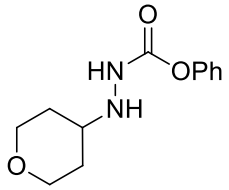
Carbazate 1j: Phenyl 2-((1*H*-indol-3-yl)methyl)hydrazine-1-carboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (2.27 g, 14.9 mmol) and 1*H*-indole-2-carbaldehyde (2.17 g, 14.9 mmol) at room temperature in MeOH (0.3 M) overnight. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (2.81 g, 44.7 mmol) was added and allowed to stir for 16 h. The title compound was filtered after reduction, washed with methanol and dried under vacuum to yield an amorphous white solid (1.48 g, 35%). TLC Rf = 0.47 in 50% Hexane/EtOAc. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm 9.14 (d, *J* = 3.9 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.33-7.43 (m, 3H), 7.29 (d, *J* = 2.3 Hz, 1H), 7.17-7.25 (m, 1H), 7.03-7.15 (m, 3H), 6.94-7.03 (m, 1H), 4.74 (d, *J* = 5.0 Hz, 1H), 4.11 (d, *J* = 5.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.1 (C), 150.9 (C), 136.2 (C), 129.2 (CH), 127.1 (C), 124.9 (CH_2), 124.1 (CH), 121.6 (CH), 120.9 (CH), 118.7 (CH), 118.3 (CH), 115.1 (CH), 111.3 (C), 111.0 (CH), 46.0 (CH_2). IR (film): 3726, 3640, 2981, 1676, 1644, 1506, 1456, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{16}H_{15}N_3O_2$ $[M]^+$: 281.1164. Found: 281.1161.



Carbazate 1k: Phenyl 2-isopropylhydrazine-1-carboxylate: Synthesized according to general procedure **2** by refluxing *O*-phenyl carbazate (1.14 g, 19.7 mmol) in acetone (0.3 M) overnight. The crude reaction was concentrated under reduced pressure then diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.71 g, 59.1 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (40% Hexane/EtOAc) to yield an amorphous white solid (1.75 g, 45%). TLC Rf = 0.63 in 50% Hexane/EtOAc.^{1}H NMR (300 MHz, DMSO-d_6) δ ppm 9.03 (d, *J* = 4.1 Hz, 1H), 7.32-7.43 (m, 2H), 7.16-7.24 (m, 1H), 7.04-7.12 (m, 2H), 4.53 (br s, 1H), 3.01-3.14 (m, 1H), 0.96 (d, *J* = 6.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 129.3 (C), 129.2 (C), 124.9 (CH), 121.5 (CH), 115.2 (CH), 49.5 (CH), 20.6 (CH_3). IR (film): 3735, 2970, 2430, 1731, 1705, 1645, 1490, 1456, 1266, 1207, 1164 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{10}H_{14}N_2O_2Na$ $[M]^+$: 217.0953. Found: 217.0953.



Carbazate 1l: Phenyl 2-(2,3-dihydro-1*H*-inden-2-yl)hydrazine-1-carboxylate: Synthesized according to general procedure **2** by stirring *O*-phenyl carbazate (2.50 g, 16.4 mmol) and 1,3-dihydro-2*H*-inden-2-one (2.17 g, 16.4 mmol) at room temperature in MeOH (0.3 M) for 1 h. The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.10 g, 49.2 mmol) was added and allowed to stir for 16 h. The title compound was filtered after reduction, washed with methanol and dried under vaccum to yield an amorphous white solid (3.69 g, 83%). TLC Rf = 0.21 in 80% Hexane/EtOAc. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.18 (d, *J* = 3.9 Hz, 1H), 7.37-7.41 (m, 2H), 7.18-7.24 (m, 3H), 7.09-7.13 (m, 4H), 4.88 (br s, 1H), 3.93 (td, *J* = 6.6, 3.2 Hz, 1H), 2.99 (dd, *J* = 16.4, 6.8 Hz, 2H), 2.80 (dd, *J* = 16.4, 3.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.2 (C), 150.9 (C), 141.8 (C), 129.3 (CH), 126.0 (CH), 124.9 (CH), 124.5 (CH), 121.6 (CH), 58.9 (CH), 37.4 (CH_2). IR (film): 3687, 2989, 2380, 1716, 1683, 1643, 1539, 1521, 1456, 1263, 1201 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{16}H_{16}N_2O_2$ $[M]^+$: 268.1212. Found: 268.12376.



Carbazate 1m: Phenyl 2-(tetrahydro-2*H*-pyran-4-yl)hydrazine-1-carboxylate: Synthesized according to general procedure **2** by refluxing *O*-phenyl carbazate (2.70 g, 17.7 mmol) and tetrahydro-4*H*-pyran-4-one (1.78 g, 17.7 mmol). The crude reaction was diluted in 2:1 MeOH:AcOH (0.2 M) and $NaBH_3CN$ (3.34 g, 53.1 mmol) was added and allowed to stir for 16 h. The title compound was purified by column chromatography (50% Hexane/EtOAc) to yield an amorphous white solid (1.95 g, 47%). TLC Rf = 0.21 in 50% Hexane/EtOAc. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.38 (t, *J* = 7.9 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.12-7.17 (m, 2H), 4.00 (dt, *J* = 11.6, 3.5 Hz, 2H), 3.42 (td, *J* = 11.4, 2.2 Hz, 2H), 3.14-3.28 (m, 1H), 1.84 (d, *J* = 12.4 Hz, 2H), 1.42-1.58 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4 (C), 150.3 (C), 129.0 (CH), 125.2 (CH), 120.9 (CH), 85.7 (CH_2), 55.0 (CH), 30.8 (CH_2). IR (film): 3753, 2846, 1731, 1718, 1662, 1539, 1456, 1369, 1265, 1240, 1203, 1163, 1093, 1012 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{16}N_2O_3$ $[M]^+$: 236.1161. Found: 236.11563.

One-Pot Aza-DKP Sequence: Carbazate Scope (Table 3)

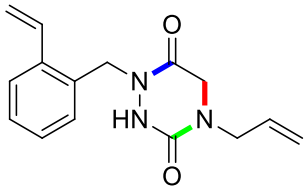


Table 3, entry 3s: 4-Allyl-1-(2-vinylbenzyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1b** (0.107 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0597 g, 55%). TLC Rf = 0.18 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm 9.38 (s, 1H), 7.56 (s, 1H), 7.26-7.34 (m, 2H), 7.19 (s, 1H), 7.01 (s, 1H), 5.72 (dd, *J* = 17.4, 1.4 Hz, 2H), 5.34 (dd, *J* = 11.1, 1.3 Hz, 1H), 5.16-5.24 (m, 2H), 4.76 (s, 2H), 3.78-3.86 (m, 4H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.6 (C), 154.4 (C), 136.0 (C), 133.6 (CH), 132.7 (C), 132.4 (CH), 127.8 (CH), 127.7 (CH), 127.4 (CH), 125.7 (CH), 117.9 (CH_2), 116.7 (CH_2), 48.0 (CH_2), 47.6 (CH_2), 45.9 (CH_2). IR (film): 3718, 3336, 2993, 1695, 1683, 1662, 1653, 1647, 1636, 1506, 1456, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{17}N_3O_2$ $[M]^+$: 271.1321. Found: 271.1307.

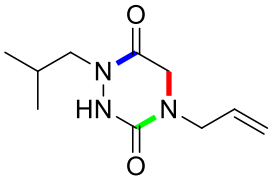


Table 3, entry 3t: 4-Allyl-1-isopropyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1c** (0.0830 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0610 g, 72%). TLC Rf = 0.25 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.30 (br s, 1H), 5.75 (ddt, *J* = 17.6, 9.8, 5.8 Hz, 1H), 5.23-5.16 (m, 2H), 3.82 (dt, *J* = 5.8, 1.4 Hz, 1H), 3.73 (s, 2H), 3.33 (d, *J* = 7.5 Hz, 2H), 2.02 (dq, *J* = 13.6, 7.0 Hz, 1H), 0.82 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.3 (C), 154.8 (C), 132.8 (CH), 117.8 (CH_2), 51.6 (CH_2), 48.2 (CH_2), 47.5 (CH_2), 25.7 (CH), 19.5 (CH_3). IR (film): 3749, 3710, 2960, 2358, 1747, 1733, 1683, 1647, 1489, 1417, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{10}H_{17}N_3O_2$ $[M]^+$: 211.1321. Found: 211.1327.

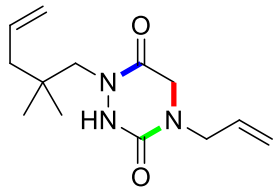


Table 3, entry 3u: 4-Allyl-1-(2,2-dimethylpent-4-en-1-yl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1d**[1] (0.0993 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 30% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0704 g, 70%). TLC Rf = 0.62 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 9.17 (s, 1H), 5.69-5.90 (m, 2H), 5.14-5.27 (m, 2H), 4.97-5.10 (m, 2H), 3.82 (d, J = 5.7 Hz, 2H), 3.73 (s, 2H), 1.90-2.02 (m, 2H), 0.87 (s, 6H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 161.7 (C), 155.0 (C), 134.8 (CH), 132.9 (CH), 117.6 (CH_2), 54.4 (CH_2), 48.4 (CH_2), 47.5 (CH_2), 44.6 (CH_2), 35.9 (C), 25.1 (CH_3). IR (film): 3735, 3639, 2966, 1733, 1689, 1647, 1515, 1466, 1267 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{13}H_{21}N_3O_2$ $[M]^+$: 251.1634. Found: 251.1622.

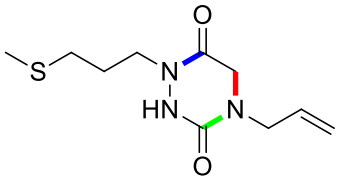


Table 3, entry 3v: 4-Allyl-1-(3-(methylthio)propyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1e** (0.0960 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 60% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0570 g, 62%). TLC Rf = 0.22 in 60% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.33 (br s, 1H), 5.82-5.68 (m, 1H), 5.23-5.16 (m, 2H), 3.81 (dt, J = 5.9, 1.3 Hz, 2H), 3.73 (s, 2H), 3.56 (t, J = 7.0 Hz, 2H), 2.43-2.38 (m, 2H), 2.03 (s, 3H), 1.80 (quint, J = 7.2 Hz, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.3 (C), 154.7 (C), 132.8 (CH), 117.9 (CH_2), 48.1 (CH_2), 47.6 (CH_2), 44.1 (CH_2), 30.1 (CH_2), 25.9 (CH_2), 14.6 (CH_3). IR (film): 3853, 3735, 1747, 1733, 1718, 1683, 1662, 1647, 1506, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{10}H_{17}N_3O_2S$ $[M]^+$: 243.1041. Found: 243.1041.

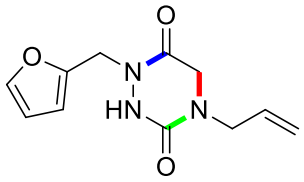


Table 3, entry 3w: 4-Allyl-1-(furan-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1f** (0.0930 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0560 g, 60%). TLC Rf = 0.21 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.38 (br s, 1H), 7.61 (dd, *J* = 1.8, 0.9 Hz, 1H), 6.42 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.36 (dd, *J* = 3.3, 1.8 Hz, 1H), 5.80.-5.66 (m, 1H), 5.19-5.12 (m, 2H), 4.66 (s, 2H), 3.80-3.77 (m, 4H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.6 (C), 154.6 (C), 148.8 (C), 142.9 (CH), 132.7 (CH), 117.8 (CH_2), 110.5 (CH), 109.1 (CH), 48.1 (CH_2), 47.6 (CH_2), 41.5 (CH_2). IR (film): 3735, 2993, 1733, 1683, 1662, 1651, 1647, 1521, 1506, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{11}H_{13}N_3O_3$ $[M]^+$: 235.0957. Found: 235.0956.

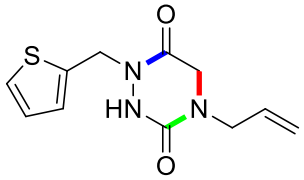


Table 3, entry 3x: 4-Allyl-1-(thiophen-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1g** (0.0990 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0610 g, 61%). TLC Rf = 0.33 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.14 (br s, 1H), 7.46 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.08 (dd, *J* = 3.4, 1.1 Hz, 1H), 6.98 (dd, *J* = 5.0, 3.4 Hz, 1H), 5.78-5.65 (m, 1H), 5.17-5.10 (m, 2H), 4.81 (s, 2H), 3.79-3.74 (m, 4H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.3 (C), 154.6 (C), 137.4 (C), 132.7 (CH), 127.6 (CH), 126.6 (CH), 126.4 (CH), 117.8 (CH_2), 48.1 (CH_2), 47.6 (CH_2), 43.3 (CH_2). IR (film): 3745, 3735, 3666, 2358, 1733, 1683, 1662, 1655, 1647, 1506, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{11}H_{13}N_3O_2S$ $[M]^+$: 251.0728. Found: 251.0732.

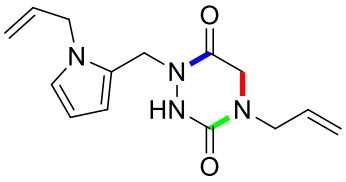


Table 3, entry 3y: 4-Allyl-1-((1-allyl-1*H*-pyrrol-2-yl)methyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1h** (0.109 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0735 g, 67%). TLC Rf = 0.45 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm 9.19 (s, 1H), 6.70 (dd, *J* = 2.6, 1.9 Hz, 1H), 6.14 (dd, *J* = 3.5, 1.8 Hz, 1H), 5.97 (dd, *J* = 3.4, 2.8 Hz, 1H), 5.83-5.94 (m, 1H), 5.66-5.77 (m, 1H), 5.19 (d, *J* = 0.8 Hz, 1H), 5.13-5.17 (m, 1H), 5.05 (dd, *J* = 10.3, 1.7 Hz, 1H), 4.78 (dd, *J* = 17.0, 1.6 Hz, 1H), 4.60 (s, 2H), 4.55 (dt, *J* = 5.0, 1.6 Hz, 2H), 3.77 (dt, *J* = 6.0, 1.2 Hz, 2H), 3.74 (s, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 159.7 (C), 154.2 (C), 135.6 (CH), 132.7 (CH), 125.5 (C), 122.3 (CH), 118.0 (CH_2), 115.5 (CH_2), 110.2 (CH), 106.9 (CH), 48.5 (CH_2), 48.0 (CH_2), 47.6 (CH_2), 40.3 (CH_2). IR (film): 3733, 3020, 1747, 1716, 1683, 1657, 1647, 1506, 1456, 1398, 1263, 1244, 1186 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{14}H_{18}N_4O_2$ $[M]^+$: 274.1430. Found: 274.1418.

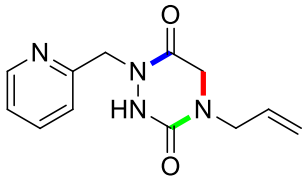


Table 3, entry 3z: 4-Allyl-1-(pyridin-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1i** (0.0970 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 10% MeOH/CH_2Cl_2 to afford the pure compound as a colorless oil (0.0550 g, 56%). TLC Rf = 0.17 in 10% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.38 (br s, 1H), 8.50-8.47 (m, 1H), 7.76 (td, *J* = 7.7, 1.8 Hz, 1H), 7.27 (dd, *J* = 7.6, 4.8 Hz, 1H), 7.20 (d, *J* = 7.8 Hz 1H), 5.81-5.68 (m, 1H), 5.25-5.15 (m, 2H), 4.75 (s, 2H), 3.82-3.80 (m, 4H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.9 (C), 155.1 (C), 154.5 (C), 149.2 (CH), 136.9 (CH), 132.8 (CH), 122.6 (CH), 121.0 (CH), 117.8 (CH_2), 49.8 (CH_2), 48.1 (CH_2), 47.7 (CH_2). IR (film): 3733, 3610, 3033, 1731, 1706, 1680, 1649, 1642, 1506, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{14}N_4O_2$ $[M]^+$: 246.1117. Found: 246.1115.

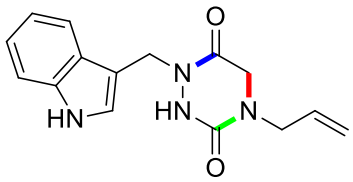


Table 3, entry 3aa: 1-((1H-Indol-3-yl)methyl)-4-allyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1j** (0.113 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The title compound was purified by column chromatography (50% EtOAc/ CH_2Cl_2) to yield an amorphous white solid (0.0705 g, 62%). TLC Rf = 0.37 in 50% EtOAc/ CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.99 (br s, 1H), 9.21 (br s, 1H), 7.59 (d, J = 8.0 Hz, 1H), 7.27-7.39 (m, 2H), 7.01-7.11 (m, 1H), 6.89-6.99 (m, 1H), 5.55-5.69 (m, 1H), 4.98-5.13 (m, 2H), 4.75 (s, 2H), 3.56-3.75 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.1 (C), 155.0 (C), 136.7 (C), 133.1 (CH), 126.8 (C), 126.5 (CH), 121.8 (CH), 119.4 (CH), 119.3 (CH), 118.2 (CH_2), 112.0 (CH), 109.1 (C), 48.6 (CH_2), 47.9 (CH_2), 40.5 (CH), 19.7 (CH_3). IR (film): 3861, 3735, 3082, 1772, 1739, 1683, 1653, 1533, 1506, 1456, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{16}N_4O_2$ $[M]^+$: 284.1273. Found: 284.1263.

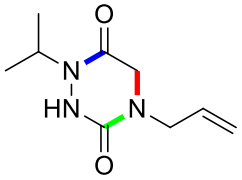


Table 3, entry 3bb: 4-Allyl-1-isopropyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1k** (0.0780 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 16 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0520 g, 66%). TLC Rf = 0.18 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.07 (br s, 1H), 5.82-5.69 (m, 1H), 5.22-5.14 (m, 2H), 4.60 (sept, J = 6.8 Hz, 1H), 3.80 (d, J = 5.8 Hz, 2H), 3.68 (s, 2H), 1.14 (d, J = 6.9 Hz, 6H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.8 (C), 155.5 (C), 132.9 (CH), 117.8 (CH_2), 49.4 (CH_2), 47.6 (CH_2), 46.0 (CH_2), 18.5 (CH_3). IR (film): 3849, 3715, 2946, 2356, 1757, 1744, 1683, 1647, 1489, 1447, 1266 cm^{-1}. HRMS (EI): Exact mass calcd for $C_9H_{15}N_3O_2$ $[M]^+$: 197.1164. Found: 197.1162.

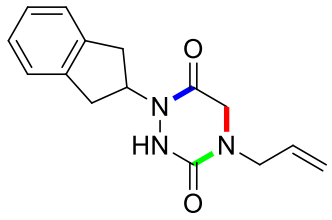


Table 3, entry 3cc: 4-Allyl-1-(2,3-dihydro-1*H*-inden-2-yl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1l** (0.107 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 72 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 30% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0600 g, 55%). TLC Rf = 0.36 in 30% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.07 (br s, 1H), 5.82-5.69 (m, 1H), 5.22-5.14 (m, 2H), 4.60 (sept, *J* = 6.8 Hz, 1H), 3.80 (d, *J* = 5.8 Hz, 2H), 3.68 (s, 2H), 1.14 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 162.2 (C), 155.4 (C), 140.4 (C), 132.8 (CH), 126.5 (CH), 124.4 (CH), 118.0 (CH), 54.2 (CH), 49.0 (CH_2), 47.8 (CH_2), 34.2 (CH_2). IR (film): 3851, 3751, 3735, 3649, 3072, 1747, 1733, 1688, 1662, 1647, 1533, 1506, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{17}N_3O_2$ $[M]^+$: 271.1321. Found: 271.1322.

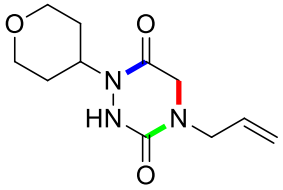


Table 3, entry 3dd: 4-Allyl-1-(tetrahydro-2H-pyran-4-yl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1m** (0.0960 g, 0.400 mmol), allylamine (0.0460 g, 0.800 mmol). The acylation was allowed to stir for 16 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 100% EtOAc to afford the pure compound as a white solid (0.0570 g, 60%). TLC Rf = 0.14 in 100% EtOAc. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.14 (br s, 1H), 5.82-5.69 (m, 1H), 5.22-5.14 (m, 2H), 4.44 (tt, *J* = 12.1, 4.2 Hz, 1H), 3.89 (dd, *J* = 11.2, 4.4 Hz, 2H), 3.80 (dt, *J* = 5.9, 1.3 Hz, 2H), 3.72 (s, 2H), 3.38-3.30 (m, 2H), 1.95 (qd, *J* = 12.4, 4.7 Hz, 2H), 1.43 (dd, *J* = 12.2, 2.3 Hz, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 161.7 (C), 155.4 (C), 132.8 (CH), 117.9 (CH_2), 66.2 (CH_2), 51.5 (CH_2), 48.5 (CH_2), 47.7 (CH_2), 28.6 (CH_2). IR (film): 3851, 3735, 1730, 1714, 1683, 1655, 1647, 1506, 1265 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{11}H_{17}N_3O_3$ $[M]^+$: 239.1270. Found: 239.1279.

Mixed Aza-DKP Synthesis (Table 4)

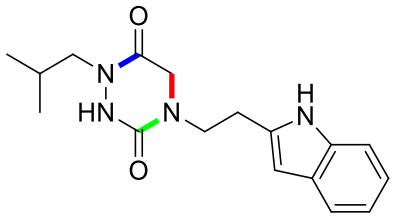


Table 4, entry 3ee: 4-(2-(1H-Indol-2-yl)ethyl)-1-isobutyl-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1c** (0.0830 g, 0.400 mmol), tryptamine (0.128 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 3% MeOH/CH_2Cl_2 to afford the pure compound as a white solid (0.0654 g, 52%). TLC Rf = 0.10 in 3% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 8.55 (br s, 1H), 8.08 (br s, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.37 (dt, *J* = 8.0, 0.9 Hz, 1H), 7.21 (td, *J* = 7.6, 1.2 Hz, 1H), 7.13 (td, *J* = 7.6, 1.2 Hz, 1H), 7.05 (d, *J* = 2.2 Hz, 1H), 3.80 (s, 2H), 3.65 (t, *J* = 7.3 Hz, 2H), 3.44 (d, *J* = 7.5 Hz, 2H), 3.07 (t, *J* = 7.3 Hz, 2H), 2.00-2.21 (sept, 1H), 0.93 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.7 (C), 155.8 (C), 136.3 (C), 127.2 (C), 122.3 (CH), 121.9 (CH), 119.6 (CH), 118.5 (CH), 112.4 (C), 111.3 (CH), 53.1 (CH_2), 49.9 (CH_2), 47.43 (CH_2), 26.3 (CH), 23.4 (CH_2), 19.7 (CH_3). IR (film): 3865, 3735, 2993, 1733, 1683, 1652, 1647, 1506, 1456, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{17}H_{22}N_4O_2$ $[M]^+$: 314.1743. Found: 314.1753.

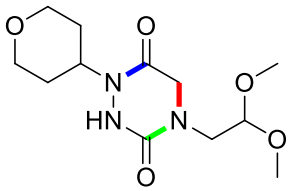


Table 4, entry 3ff: 4-(2,2-Dimethoxyethyl)-1-(tetrahydro-2H-pyran-4-yl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1m** (0.0960 g, 0.400 mmol), aminoacetaldehyde dimethyl acetal (0.0840 g, 0.800 mmol). The acylation was allowed to stir for 16 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 3% MeOH/CH_2Cl_2 to afford the pure compound as a white solid (0.0400 g, 35%). TLC Rf = 0.10 in 3% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.12 (br s, 1H), 4.49 (t, *J* = 5.5 Hz, 1H), 4.45-4.37 (m, 1H), 3.92-3.84 (m, 4H), 3.38-3.26 (m, 10H), 2.04-1.87 (m, 2H), 1.42 (dd, *J* = 12.5, 2.7 Hz, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 161.7 (C), 155.6 (C), 102.0 (CH), 66.2 (CH_2), 53.7 (CH_3), 51.5 (CH), 50.3 (CH_2), 47.0 (CH_2), 28.6 (CH_2). IR (film): 3735, 3004, 2354, 1730, 1718, 1683, 1653, 1647, 1506, 1423, 1386, 1299, 1266, 1203, 1147, 1126, 1085, 1070, 1051, 1008 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{21}N_3O_5$ $[M]^+$: 287.1481. Found: 287.1456.

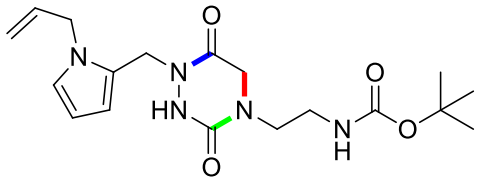


Table 4, entry 3gg: *tert*-Butyl (2-(1-((1-allyl-1*H*-pyrrol-2-yl)methyl)-3,6-dioxo-1,2,4-triazinan-4-yl)ethyl)carbamate: Synthesized according to general procedure **1A** using carbazate **1h** (0.109 g, 0.400 mmol), *N*-Boc ethylenediamine (0.128 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 100% EtOAc to afford the pure compound as a white solid (0.0700 g, 46%). TLC Rf = 0.38 in 100% EtOAc. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.03 (br s, 1H), 6.81 (t, J = 5.6 Hz, 1H), 6.69 (dd, J = 2.7, 1.8 Hz, 1H), 6.15 (dd, J = 3.5, 1.8 Hz, 4H), 5.96 (dd, J = 3.4, 2.8 Hz, 1H), 5.93-5.83 (m, 1H), 5.06 (dd, J = 10.3, 1.6 Hz, 1H), 4.77 (dd, J = 17.1, 1.6 Hz), 4.59-4.55 (m, 4H), 3.86 (s, 2H), 3.22-3.18 (m, 2H), 3.07-3.03 (m, 2H), 1.36 (s, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 159.4 (C), 154.3 (C), 135.6 (CH), 125.5 (C), 122.3 (CH), 115.4 (CH_2), 100.3 (CH), 47.0 (CH_2), 106.9 (CH), 77.7 (C), 48.9 (CH_2), 48.5 (CH_2), 45.3 (CH_2), 40.3 (CH_2), 38.7 (CH_2), 28.2 (CH_3). IR (film): 3853, 3751, 3735, 3629, 1733, 1683, 1647, 1521, 1394, 1265, 1232, 1151 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{18}H_{27}N_5O_4$ $[M]^+$: 377.2063. Found: 377.2047.

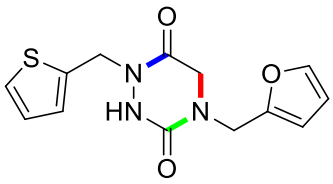


Table 4, entry 3hh: 4-(Furan-2-ylmethyl)-1-(thiophen-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1g** (0.0973 g, 0.400 mmol), furfurylamine (0.0780 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0800 g, 69%). TLC Rf = 0.36 in 50% EtOAc/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.56 (br s, 1H), 7.60 (d, J = 0.9 Hz, 1H), 7.46 (dd, J = 5.0, 1.1 Hz, 1H), 7.07 (d, 3.3 Hz, 1H), 6.98 (dd J = 5.1, 3.5 Hz, 1H), 6.50 (dd, J = 3.2, 2.0 Hz, 1H), 6.32 (d, J = 2.8 Hz, 1H), 4.80 (s, 2H), 4.38 (s, 2H), 3,78 (s, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.0 (C), 154.4 (C), 150.0 (C), 142.9 (CH), 137.3 (C), 127.6 (CH), 126.7 (CH), 126.4 (CH), 110.5 (CH), 108.7 (CH), 48.1 (CH_2), 43.3 (CH_2), 41.6 (CH_2). IR (film): 3853, 3735, 3651, 1772, 1743, 1733, 1683, 1662, 1652, 1555, 1506, 1456, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{13}H_{13}N_3O_3S$ $[M]^+$: 291.0678. Found: 291.0684.

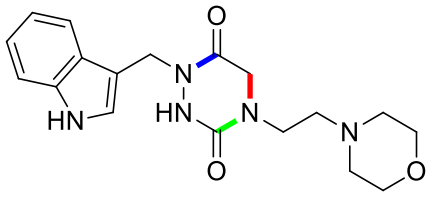


Table 4, entry 3ii: 1-((1H-indol-3-yl)methyl)-4-(2-morpholinoethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1j** (0.113 g, 0.400 mmol), 4-(2-aminoethyl)morpholine (0.104 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 100% EtOAc then 5% CHOH/CH_2Cl_2 to afford the pure compound as a white solid (0.872 g, 61%). TLC Rf = 0.44 in 5% CHOH/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$): δ ppm 8.49 (br s, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 8.2 Hz, 1H), 7.09-7.23 (m, 4H), 4.94 (s, 2H), 3.99 (s, 2H), 3.56-3.74 (m, 4H), 3.37 (t, *J* = 6.2 Hz, 2H), 2.33-2.58 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.6 (C), 154.9 (C), 136.5 (C), 126.3 (C), 125.5 (CH), 123.0 (CH), 120.5 (CH), 119.2 (CH), 111.5 (CH), 108.0 (C), 66.9 (CH_2), 56.1 (CH_2), 53.6 (CH_2), 50.1 (CH_2), 43.2 (CH_2), 41.1 (CH_2). IR (film): 3751, 3735, 3670, 1683, 1647, 1506, 1455, 1266, 1186, 1116 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{18}H_{23}N_5O_3$ $[M]^+$: 357.1801. Found: 357.1828.

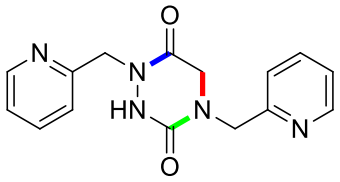


Table 4, entry 3jj: 1,4-Bis(pyridin-2-ylmethyl)-1,2,4-triazinane-3,6-dione: Synthesized according to general procedure **1A** using carbazate **1i** (0.0970 g, 0.400 mmol), 2-picolylamine (0.0865 g, 0.800 mmol). The acylation was allowed to stir for 2 h and the amination for 24 h. The crude mixture was purified by silica gel column chromatography using 50% EtOAc/CH_2Cl_2 to afford the pure compound as a white solid (0.0392 g, 33%). TLC Rf = 0.36 in 50% EtOAc/ CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$): δ ppm 8.72 (br s, 1H), 8.49-8.61 (m, 2H), 7.62-7.77 (m, 2H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.27 (ddd, *J* = 7.5, 4.9, 1.1 Hz, 1H), 7.22 (ddd, *J* = 7.5, 4.9, 1.1 Hz, 1H), 4.90 (s, 2H), 4.63 (s, 2H), 4.00 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.5 (C), 155.7 (C), 154.6 (C), 154.5 (C), 149.4 (CH), 149.3 (CH), 137.5 (CH), 137.0 (CH), 123.3 (CH), 122.8 (CH), 122.6 (CH), 122.5 (CH), 51.6 (CH_2), 49.5 (CH_2), 49.2 (CH_2). IR (film): 3735, 3612, 3043, 1733, 1716, 1683, 1662, 1652, 1506, 1263 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{15}N_5O_2$ $[M]^+$: 297.1226. Found: 297.1213.

References

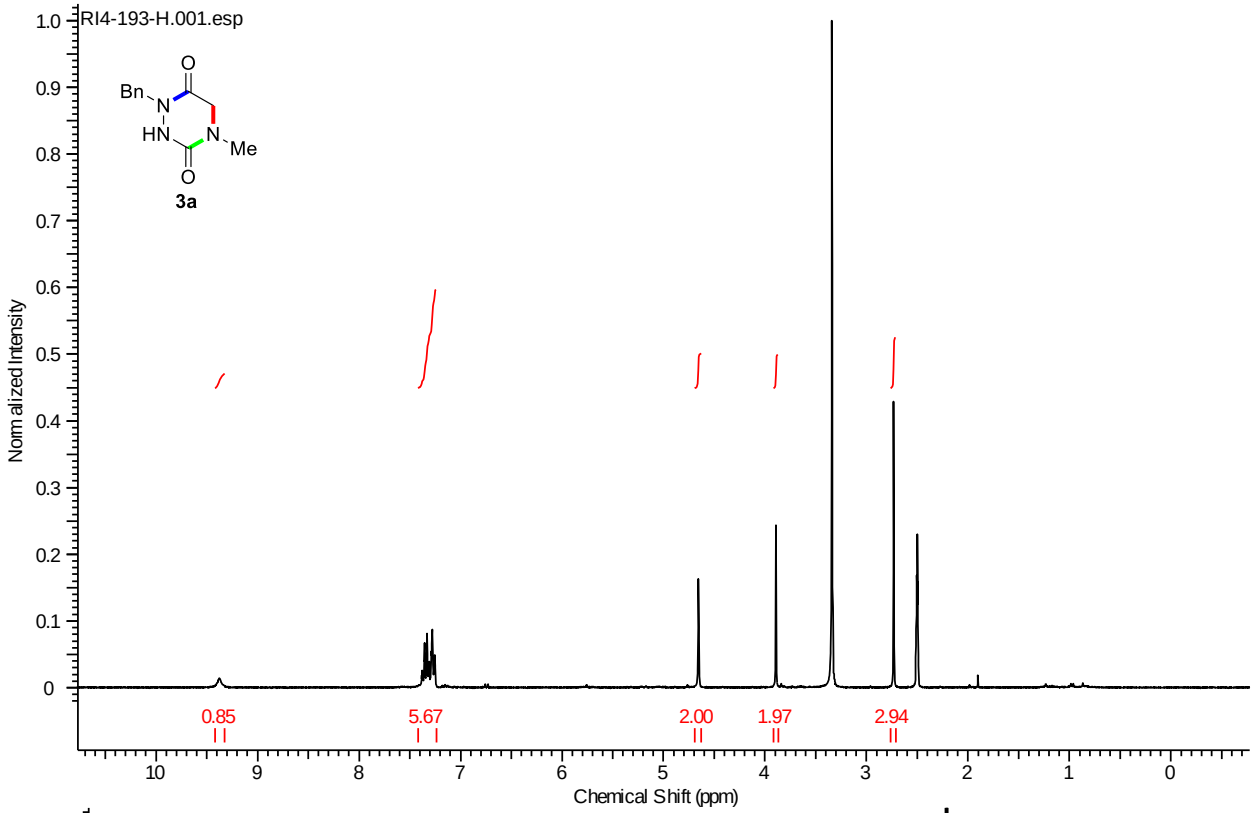
(1) Clavette, C.; Vincent-Rocan, J.-F.; Beauchemin, A. M. *Angew. Chem. Int. Ed.* **2013**, *52*, 12705.

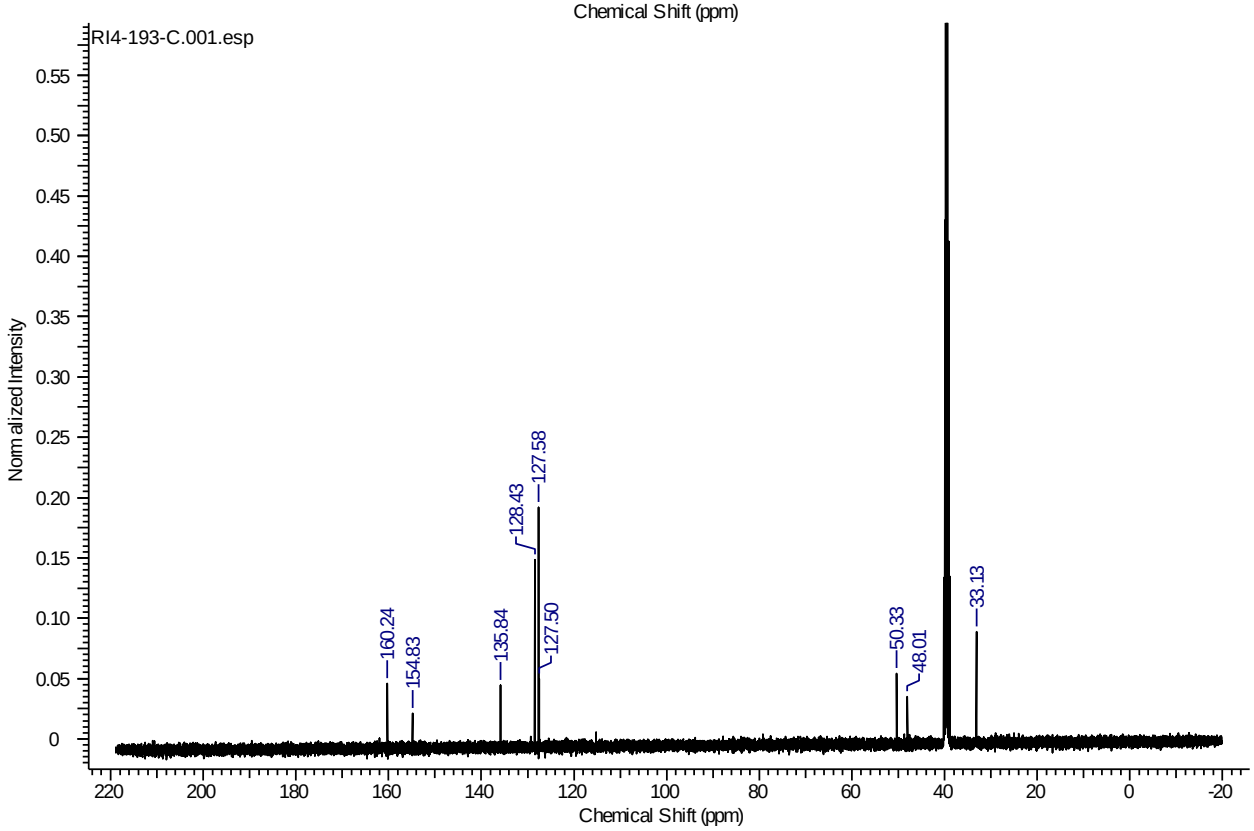
(2) Leighton, J. L.; Berger, R.; Duff, K. *J. Am. Chem. Soc.* **2004**, *126*, 5686.

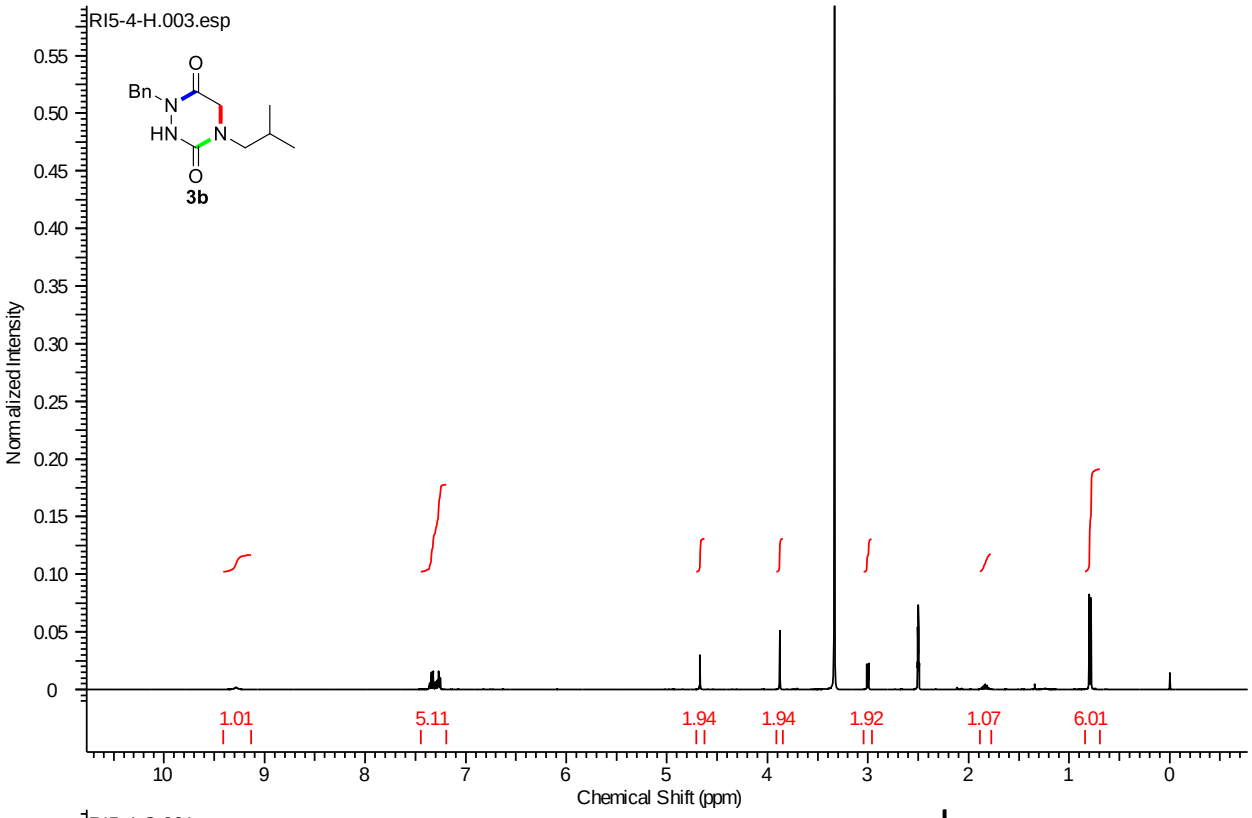
(3) Lane, C. F. *Synthesis* **1975**, 135.

(4) Tillmann, K.; Klaus, B.; Roland, F.; Birgit, W.; Ernst-Ulrich, W. *J. Org. Chem.* **2011**, *76*, 1979.

Spectral Data



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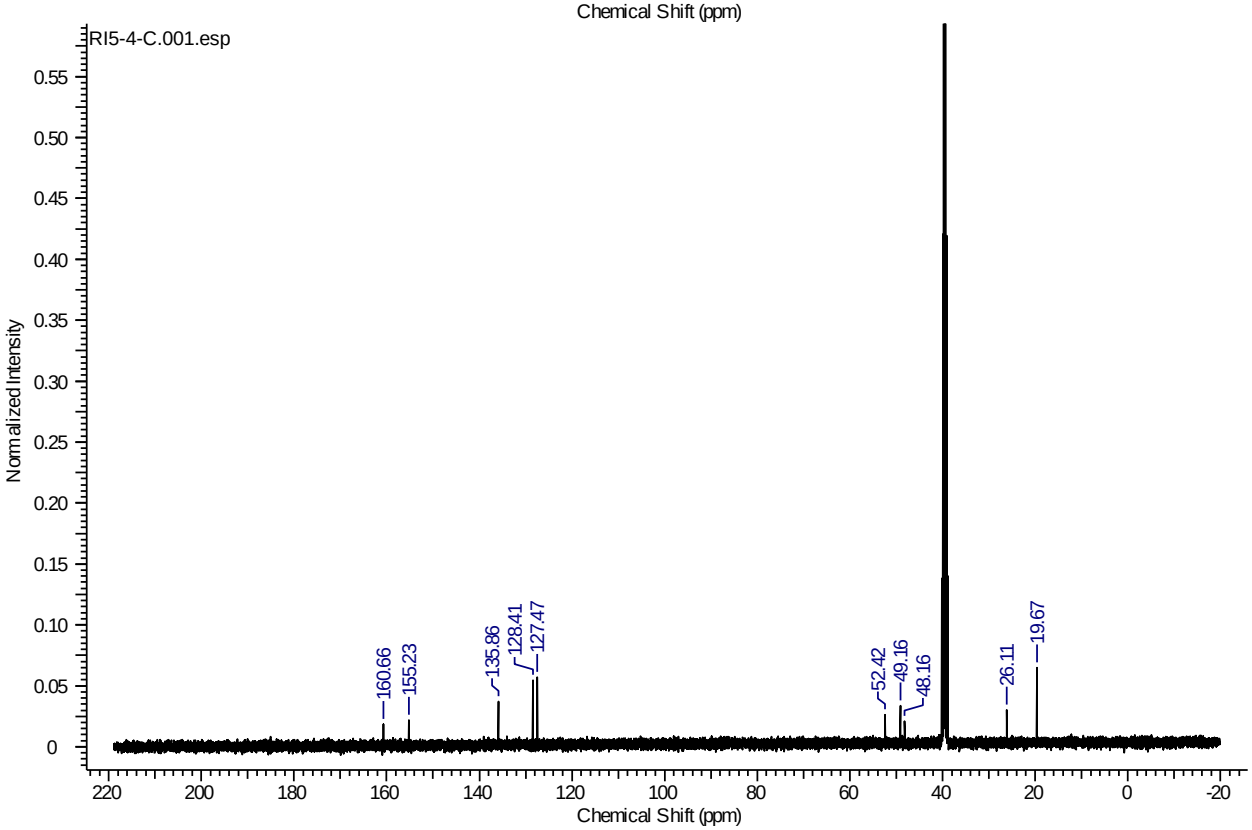
1.92
1.07
6.01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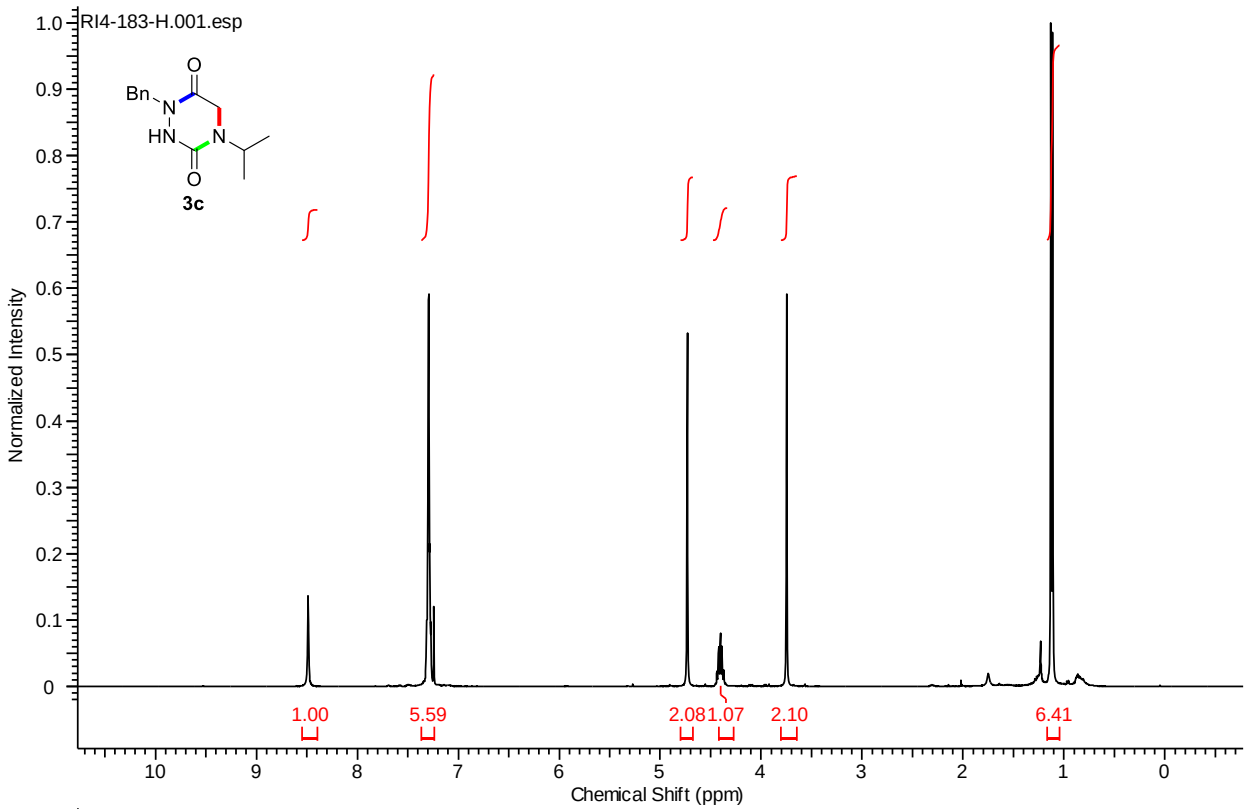
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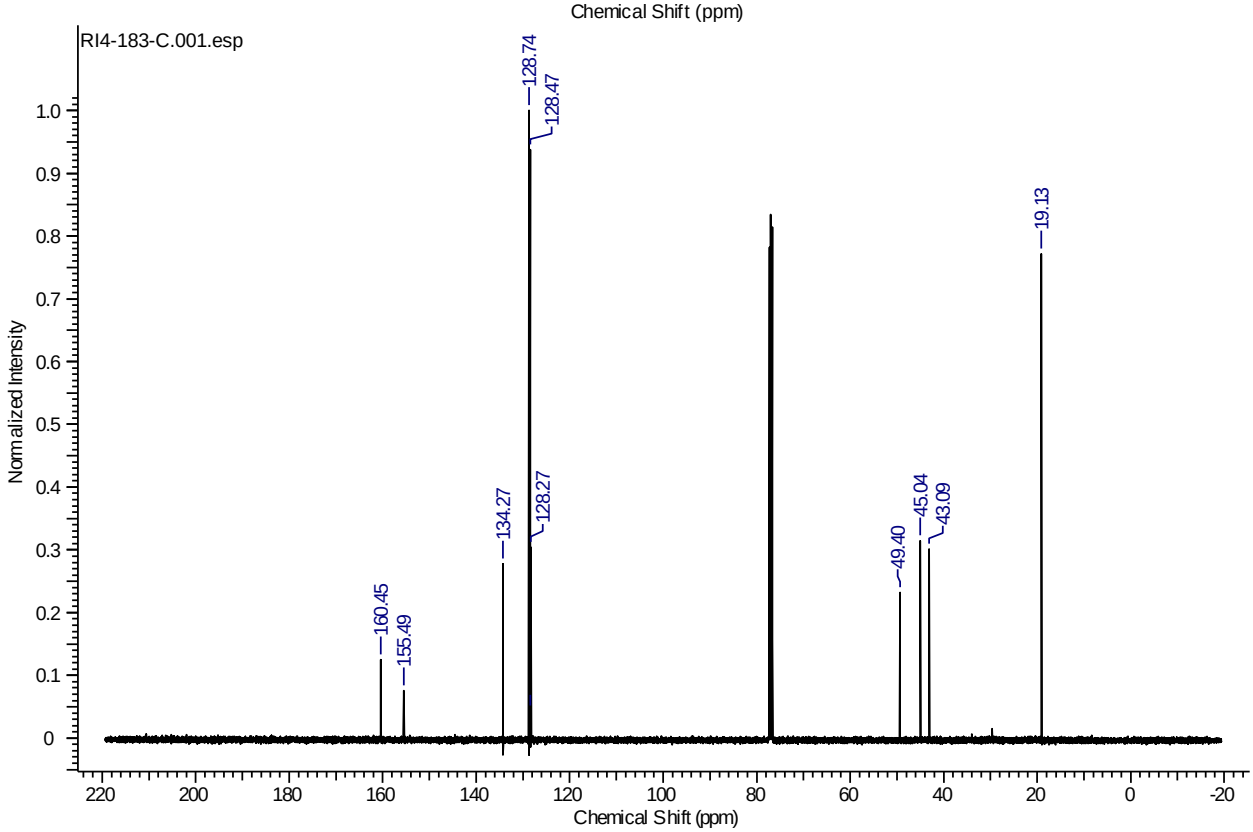
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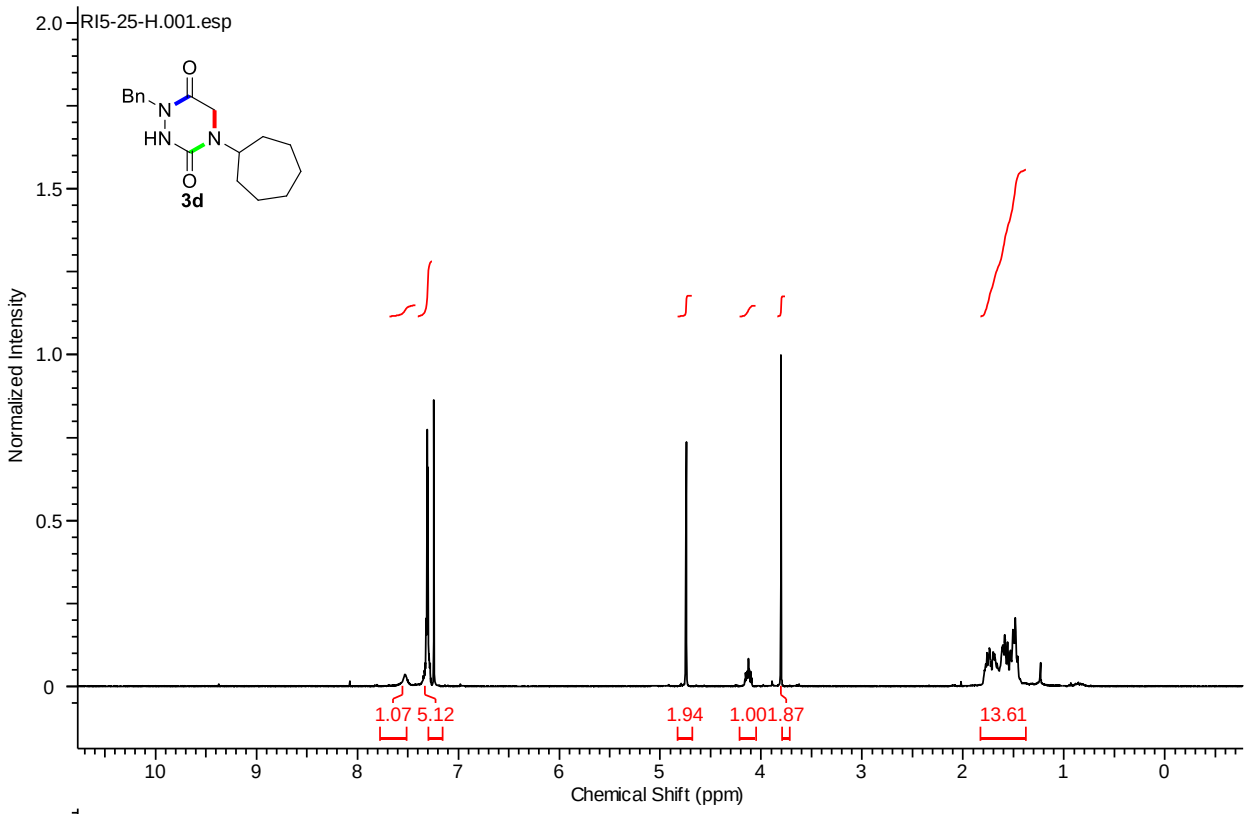
Normalized Intensity
Chemical Shift (ppm)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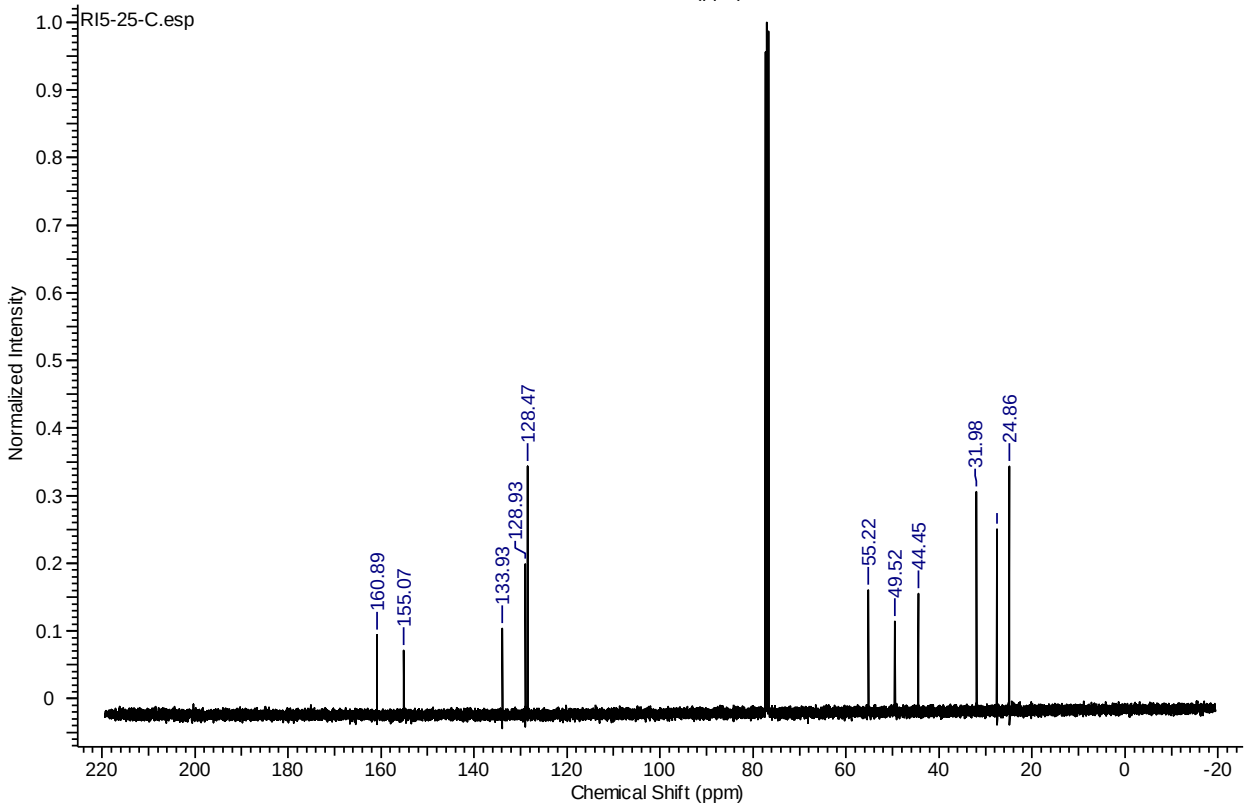
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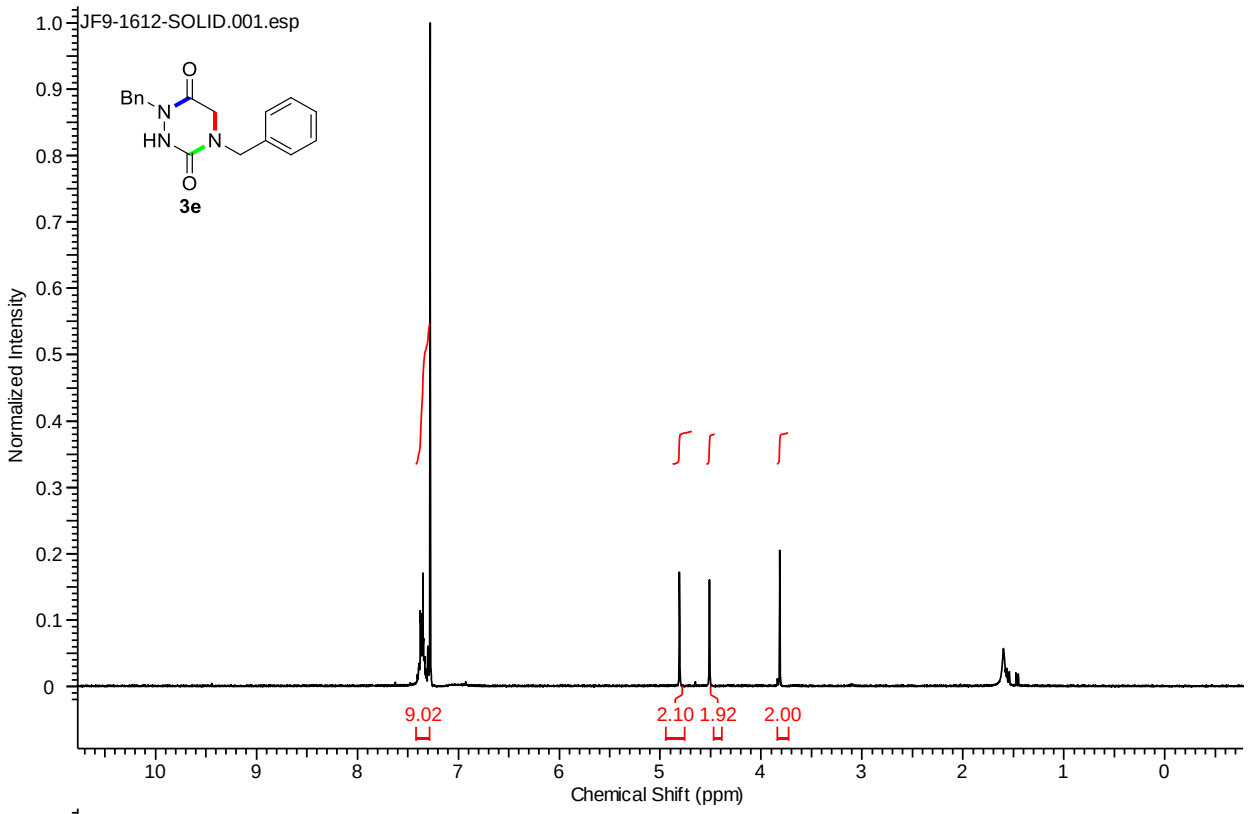
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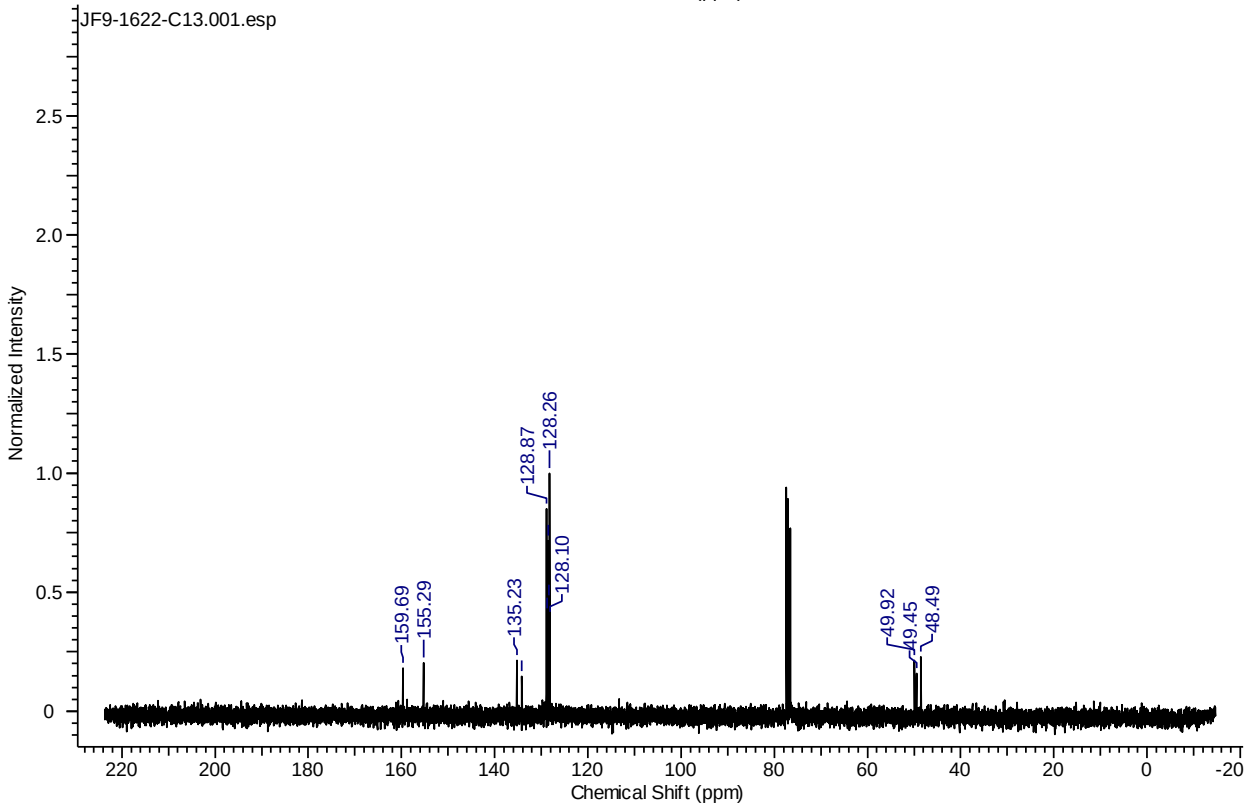
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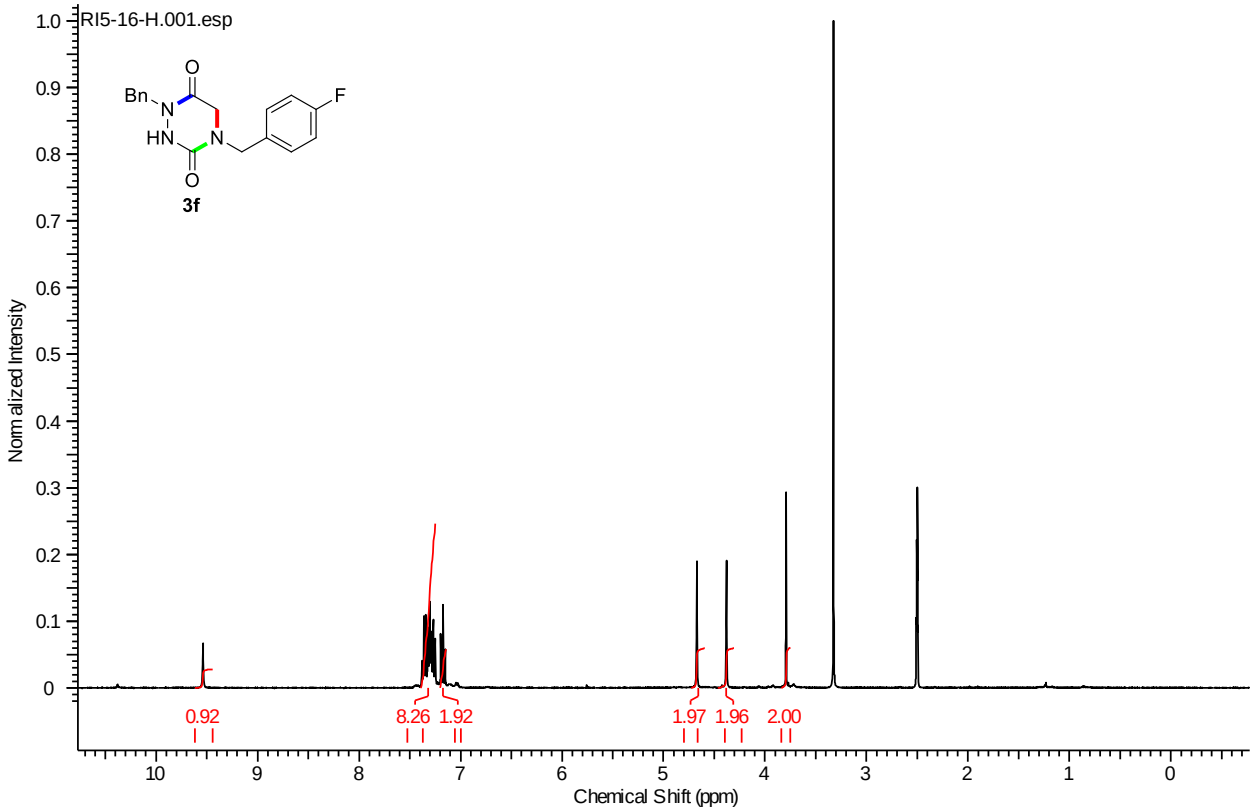
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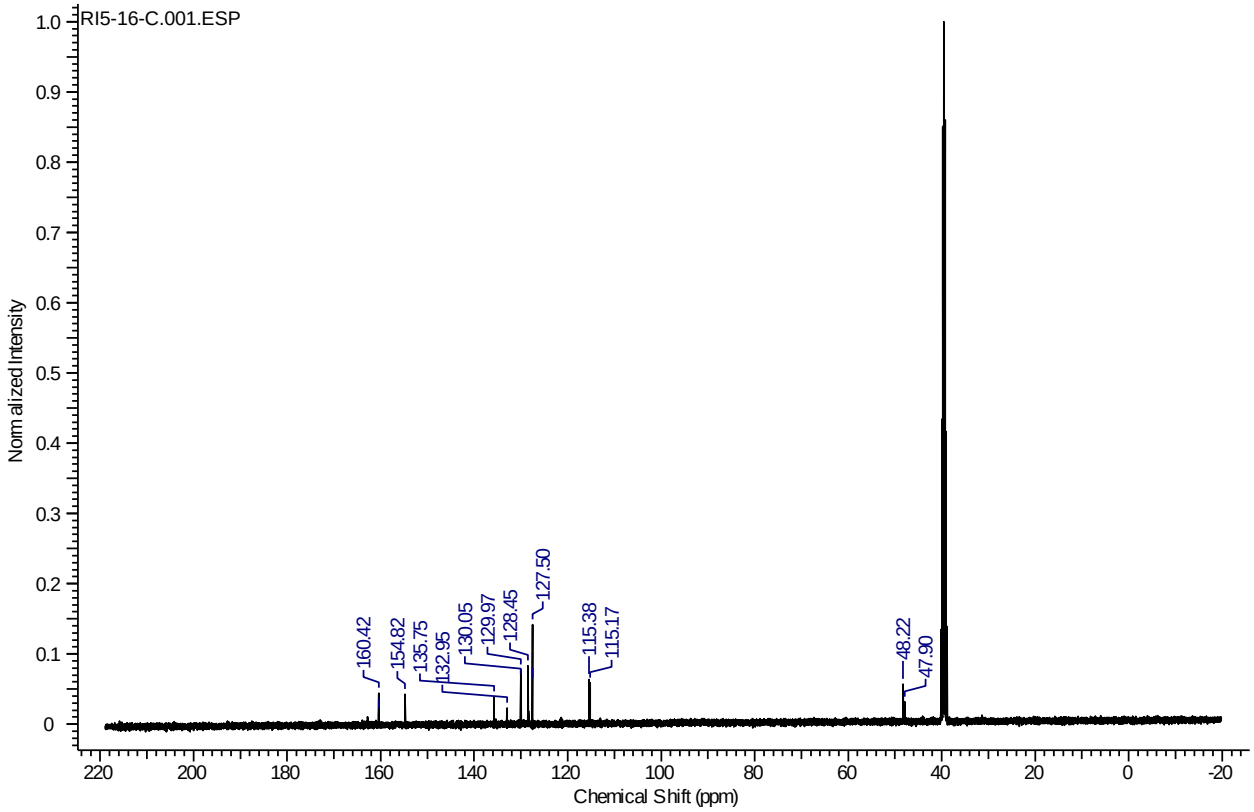
2.00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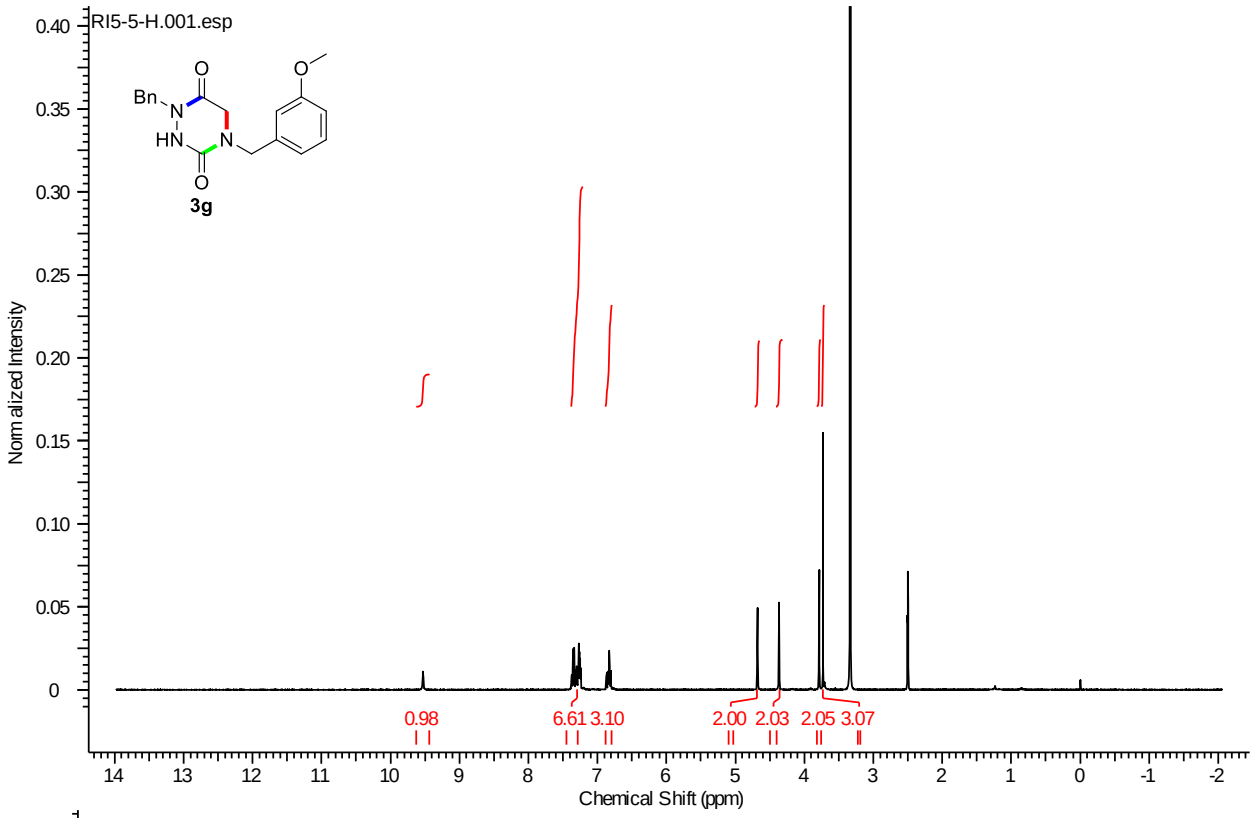
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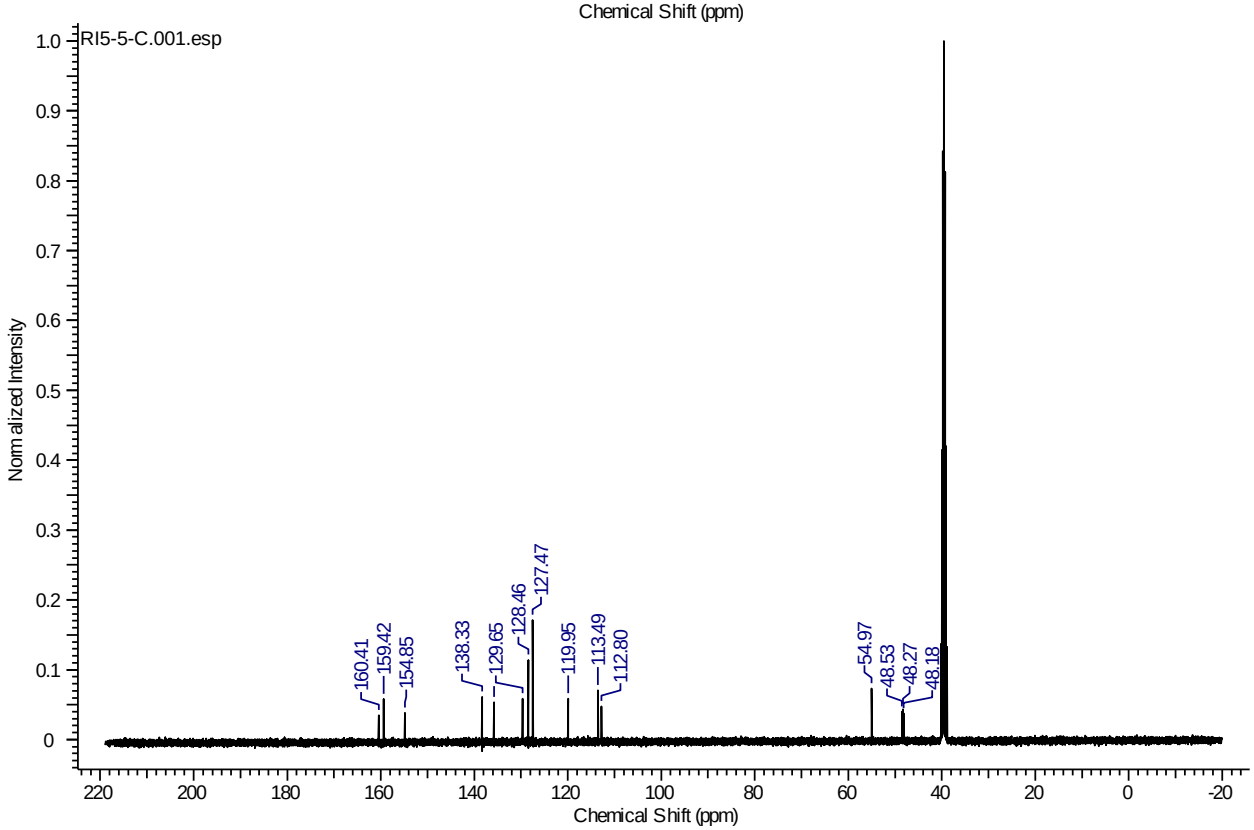
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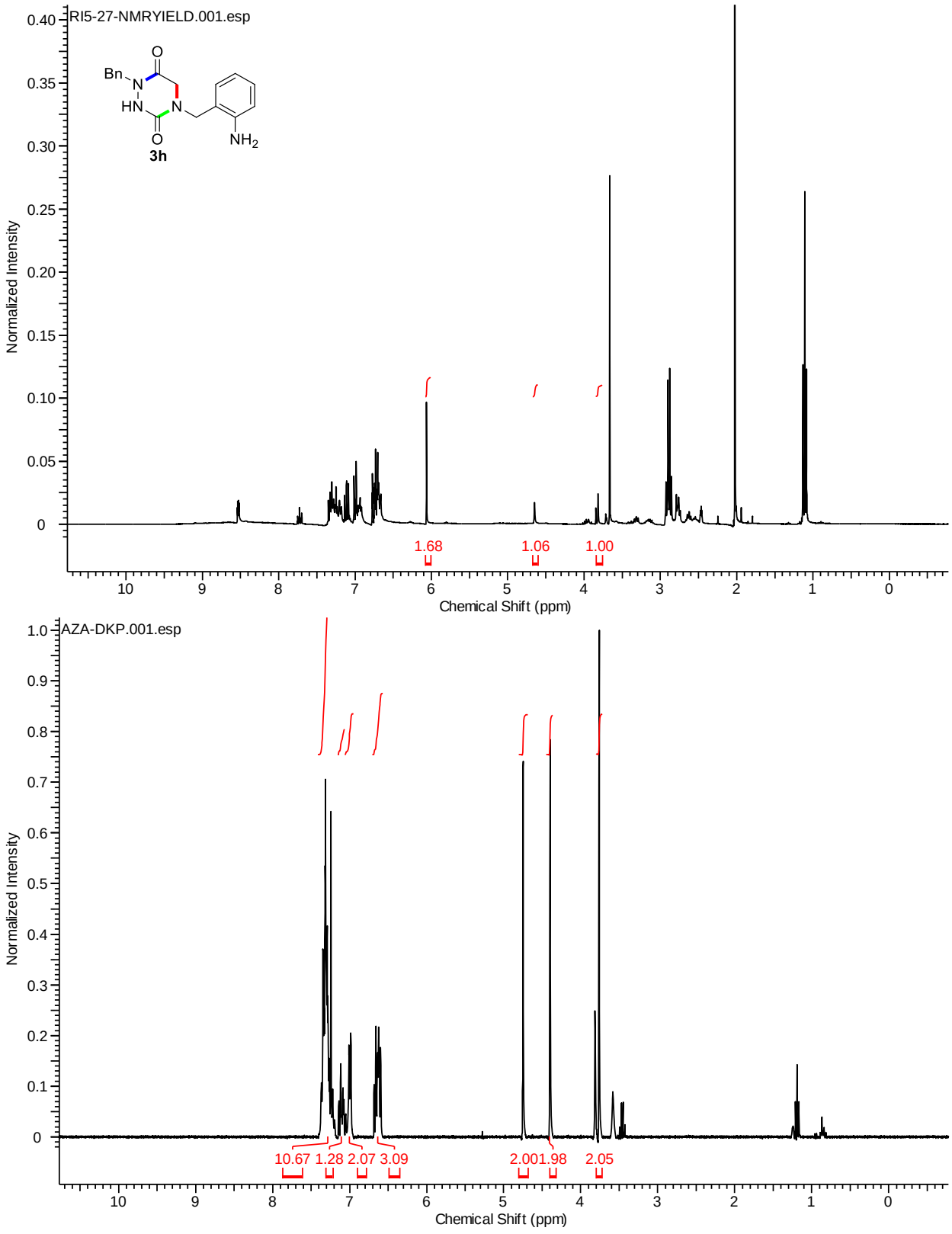
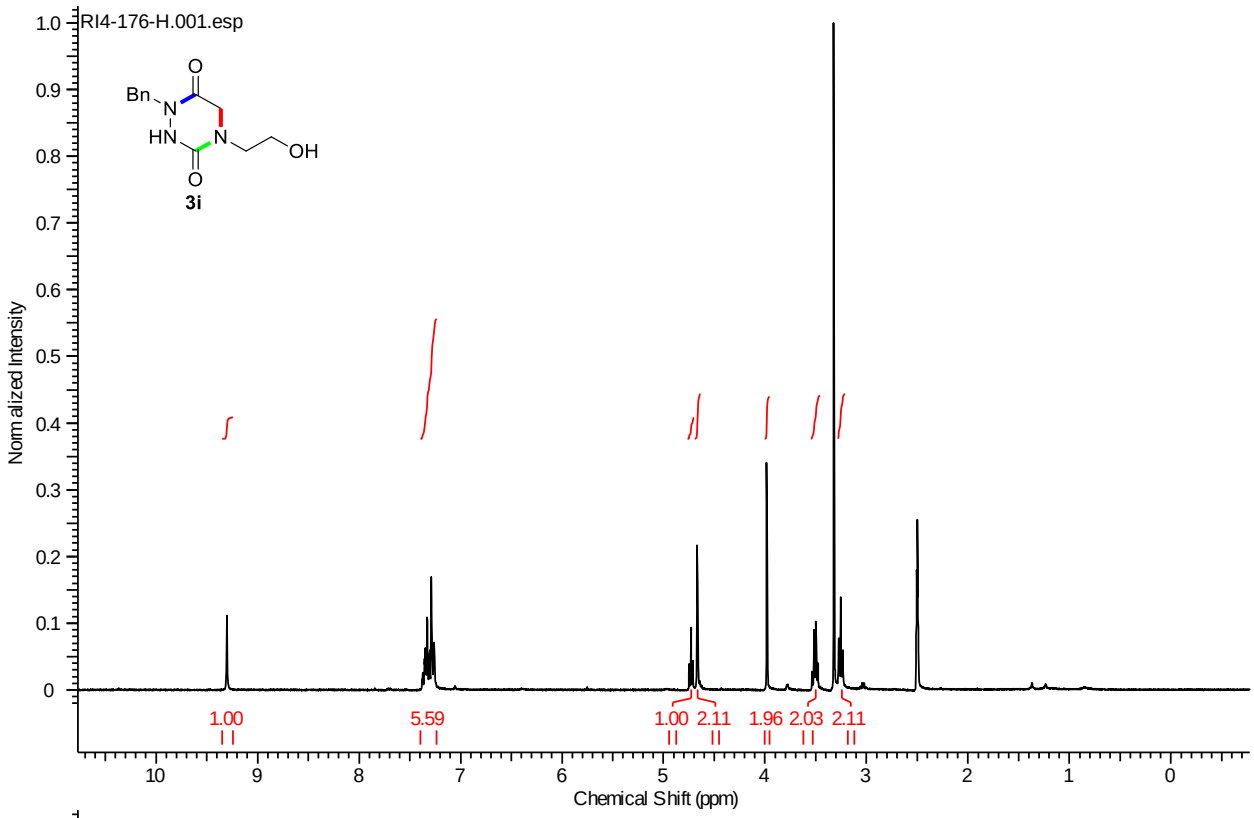


37.8 mg TMB used standard. Top spectra is NMR yield. Bottom spectra is isolation attempt.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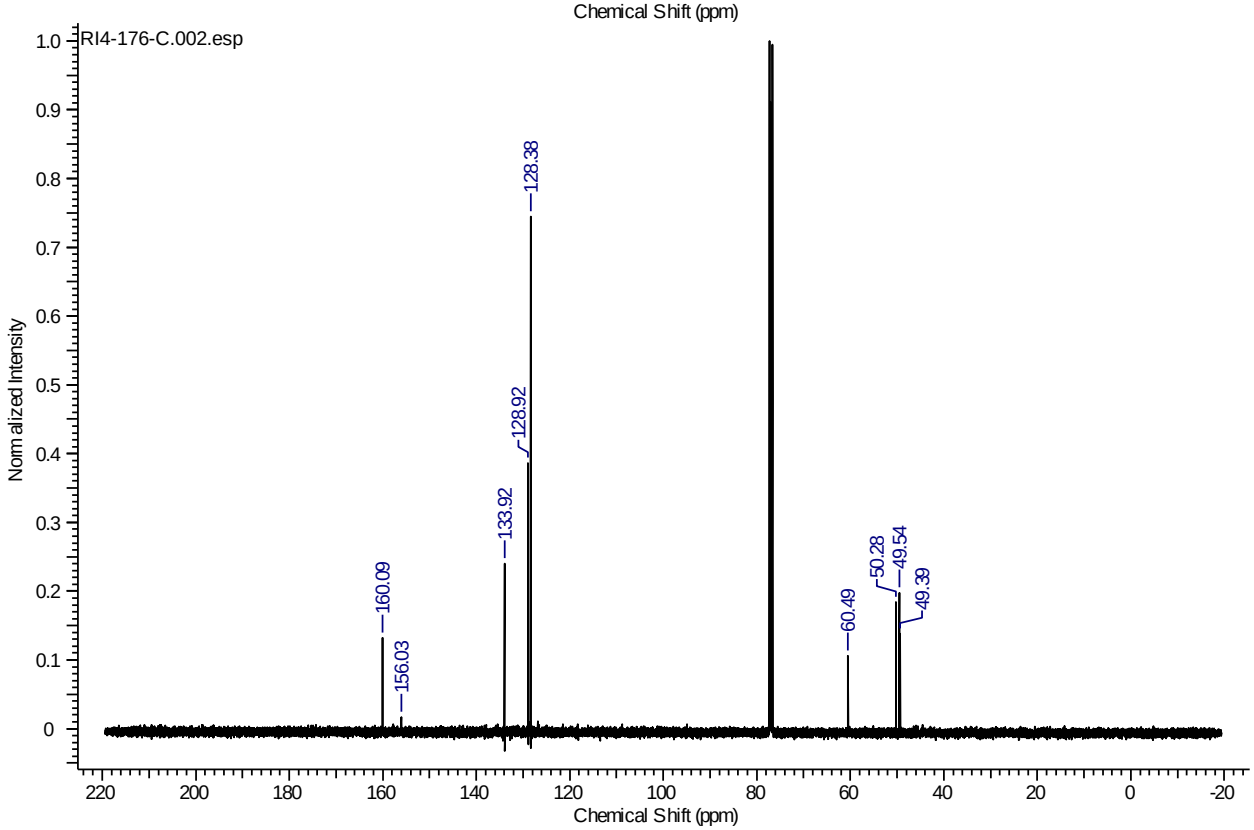
2.03
2.11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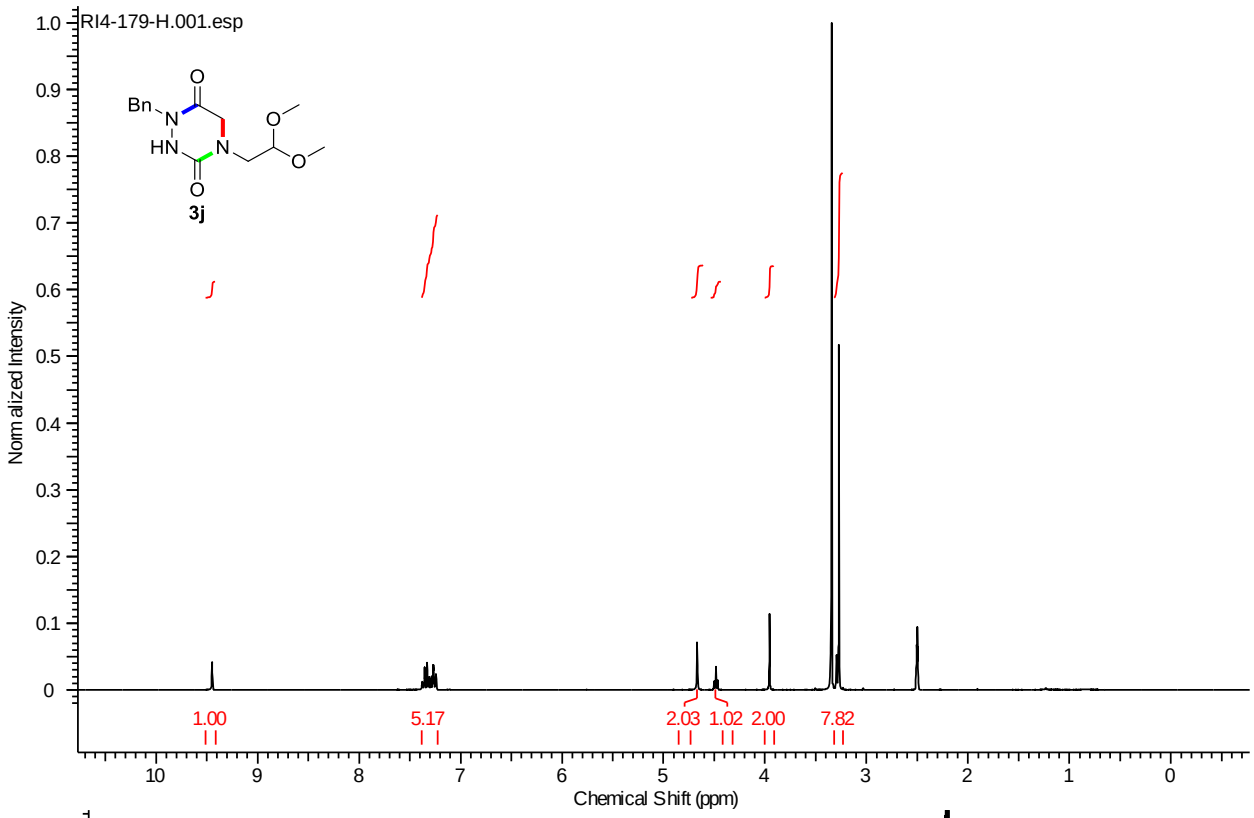
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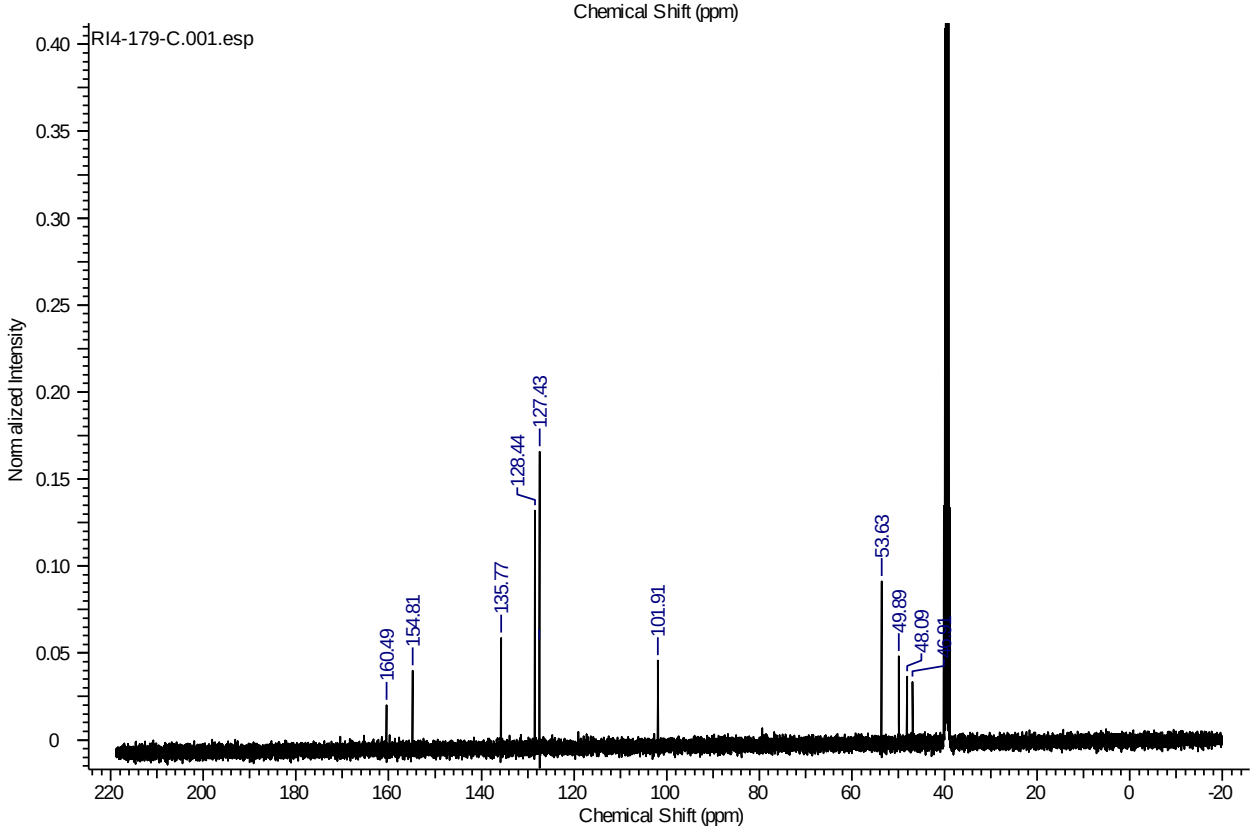
Chemical Shift (ppm)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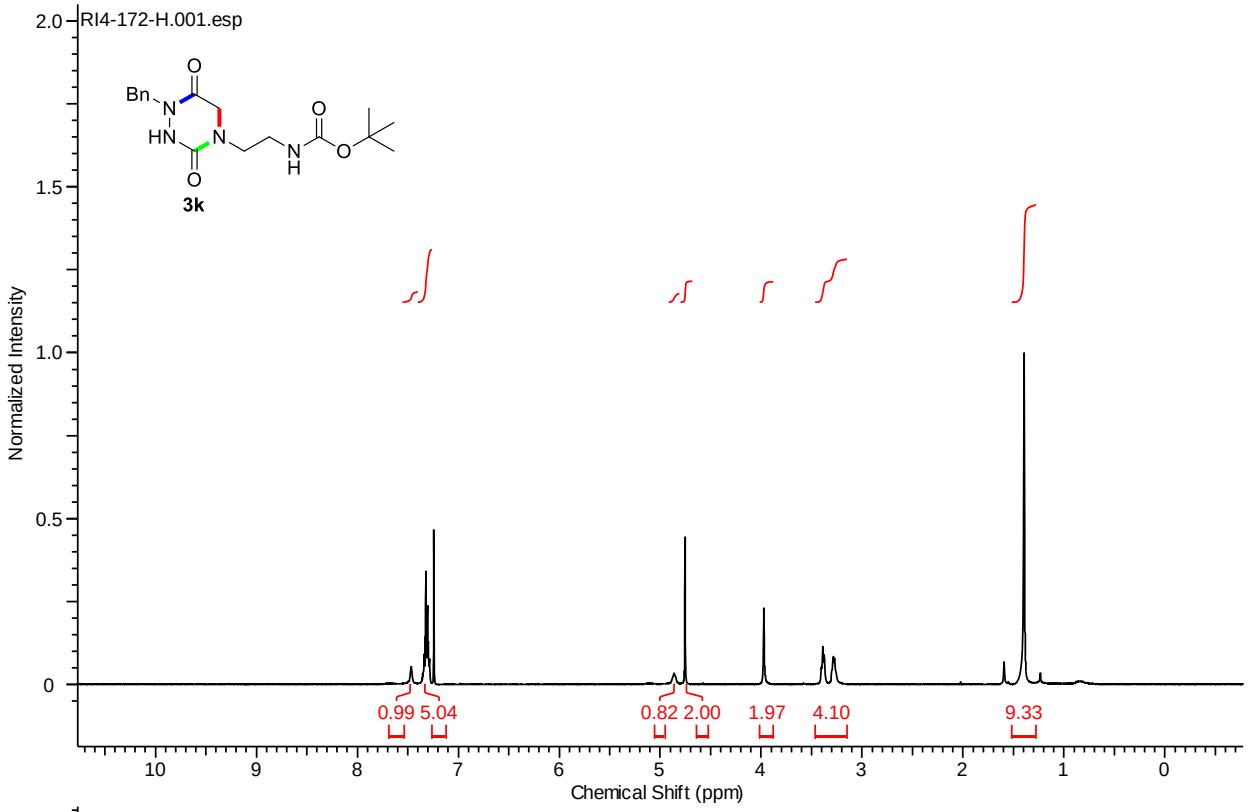
Chemical Shift (ppm)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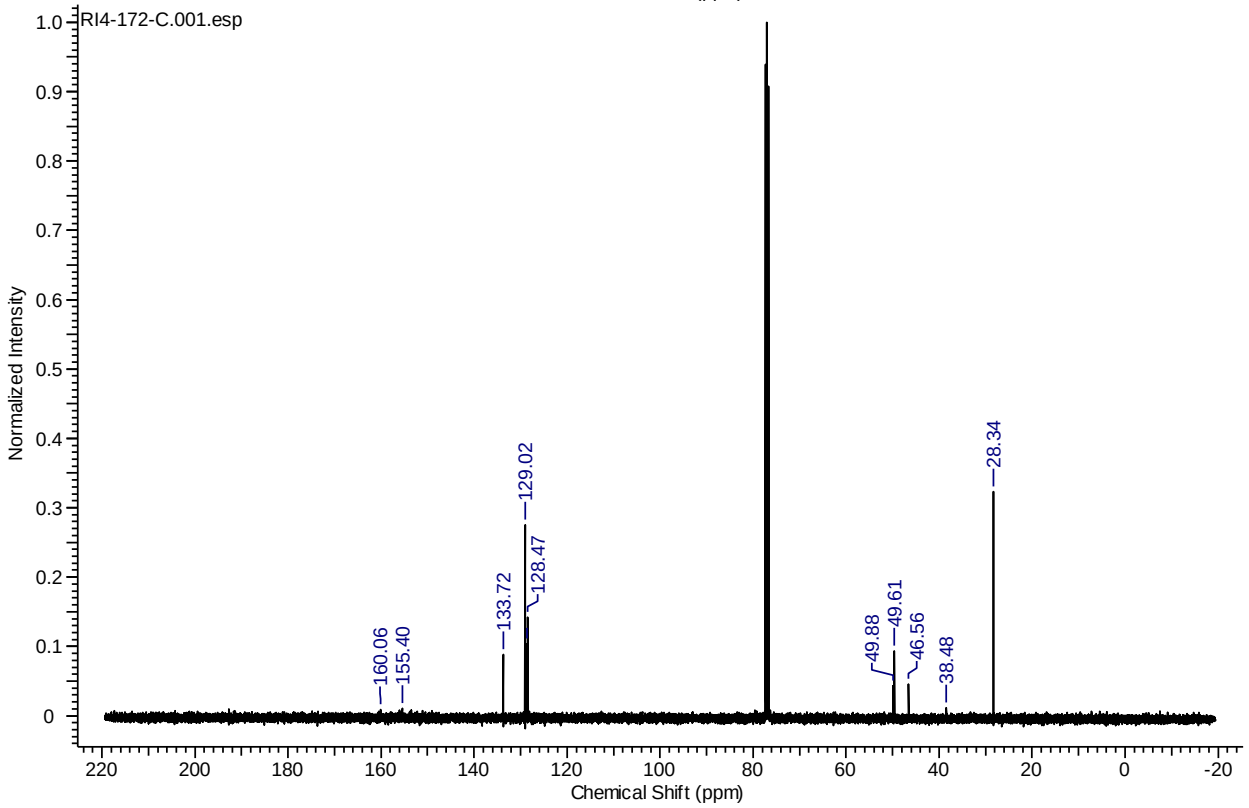
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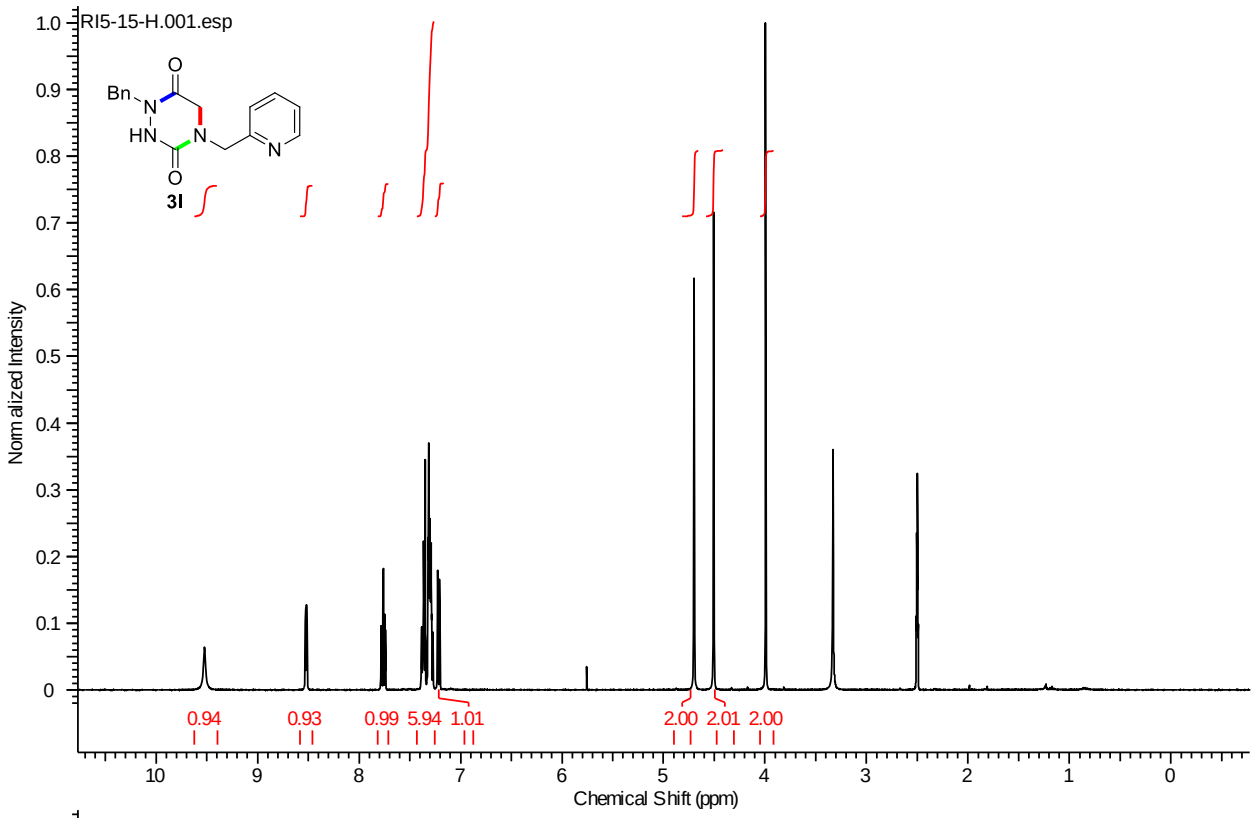
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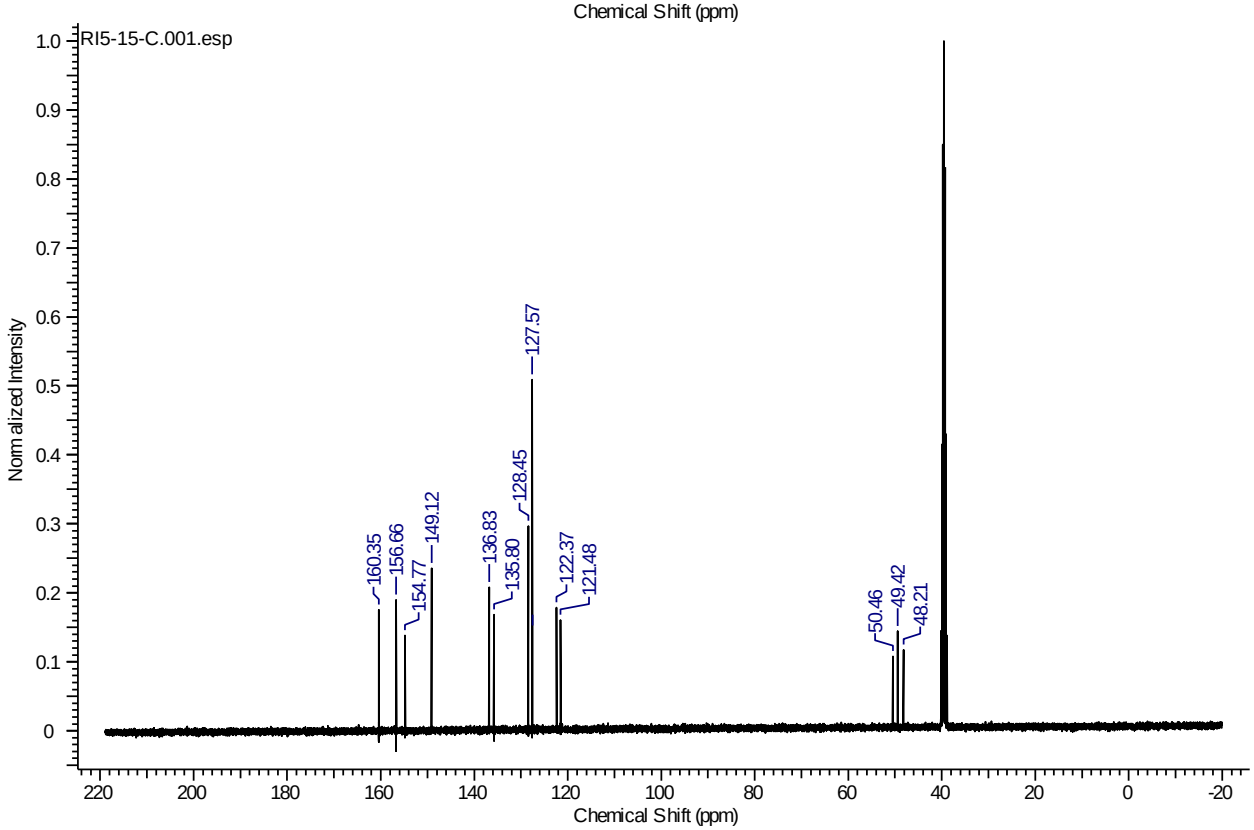
Chemical Shift (ppm)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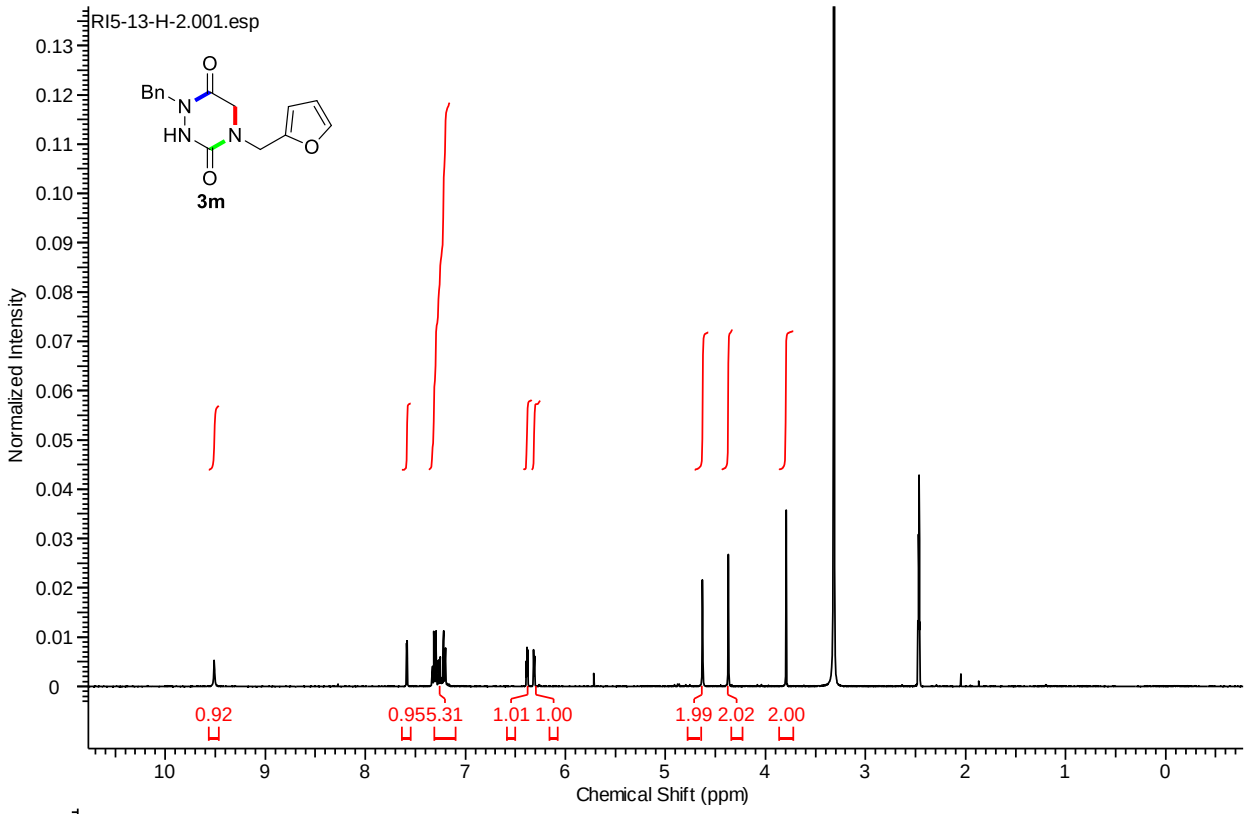
Chemical Shift (ppm)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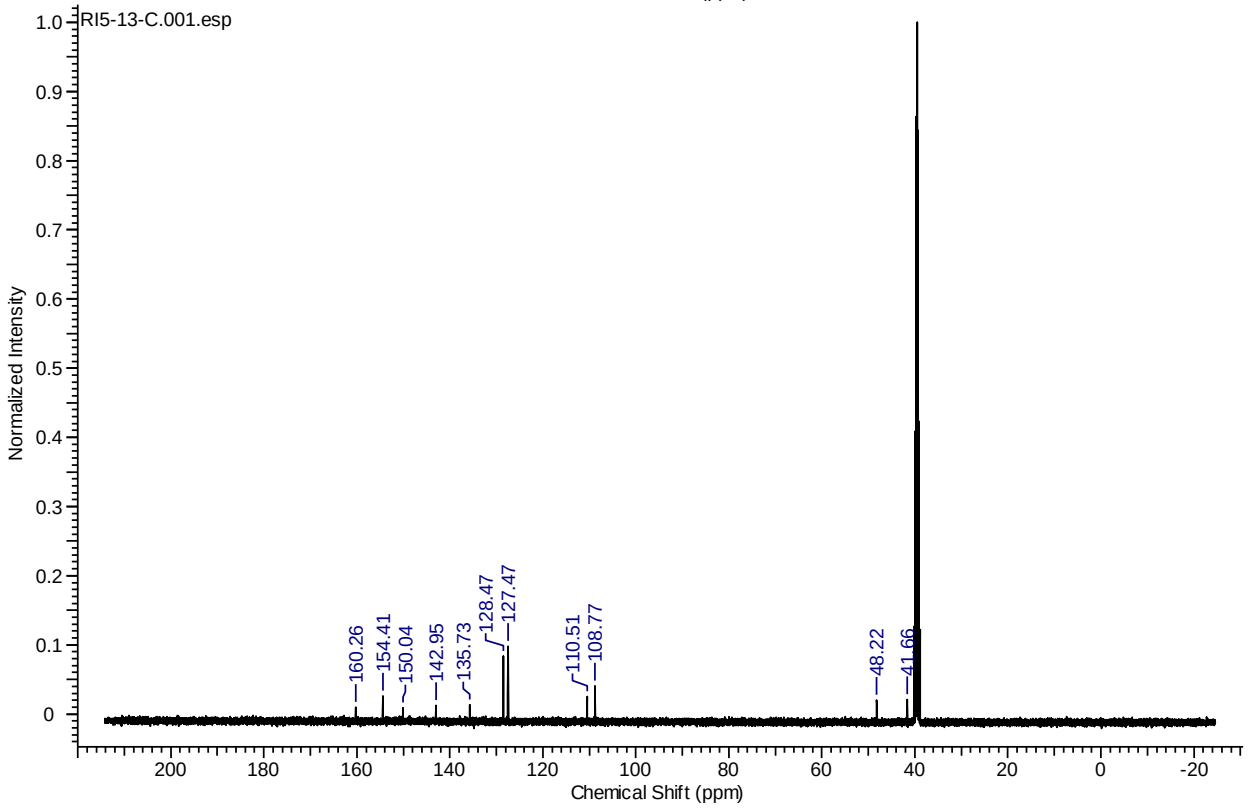
Chemical Shift (ppm)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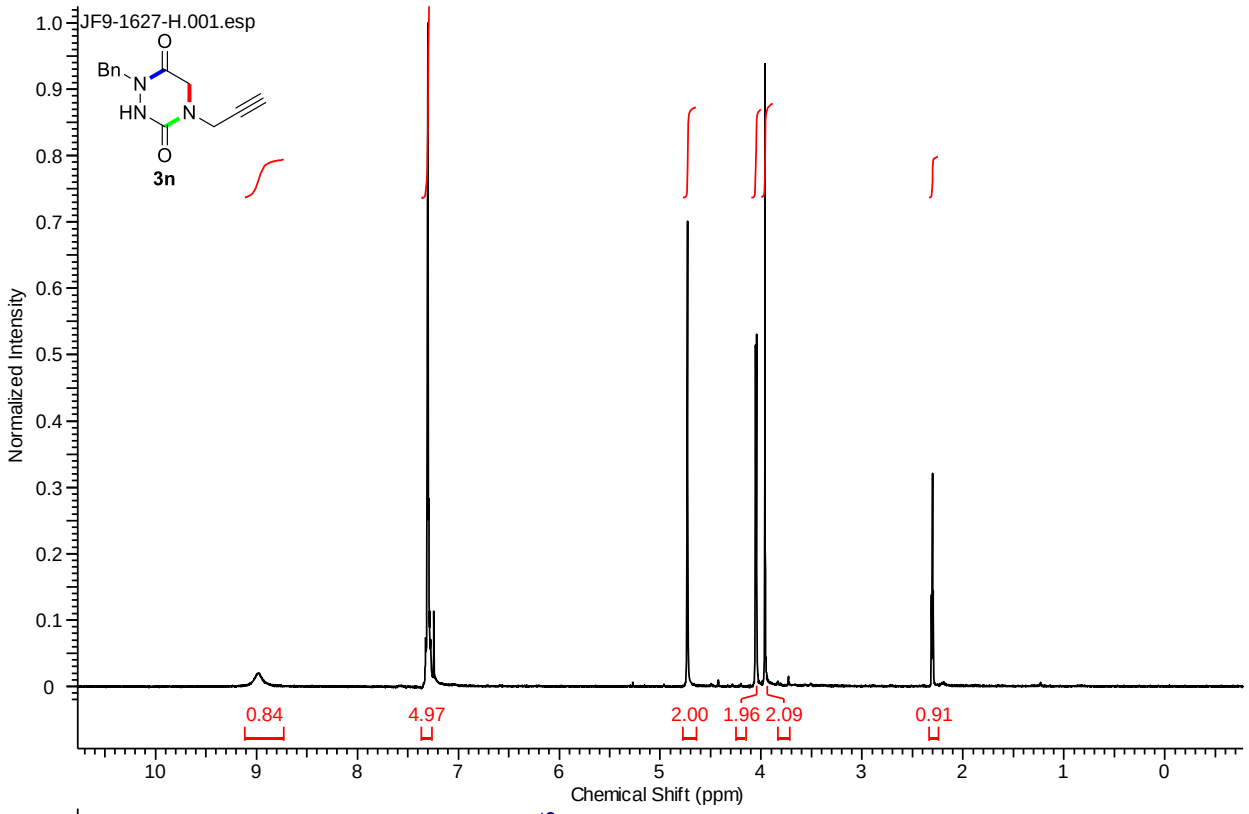
Chemical Shift (ppm)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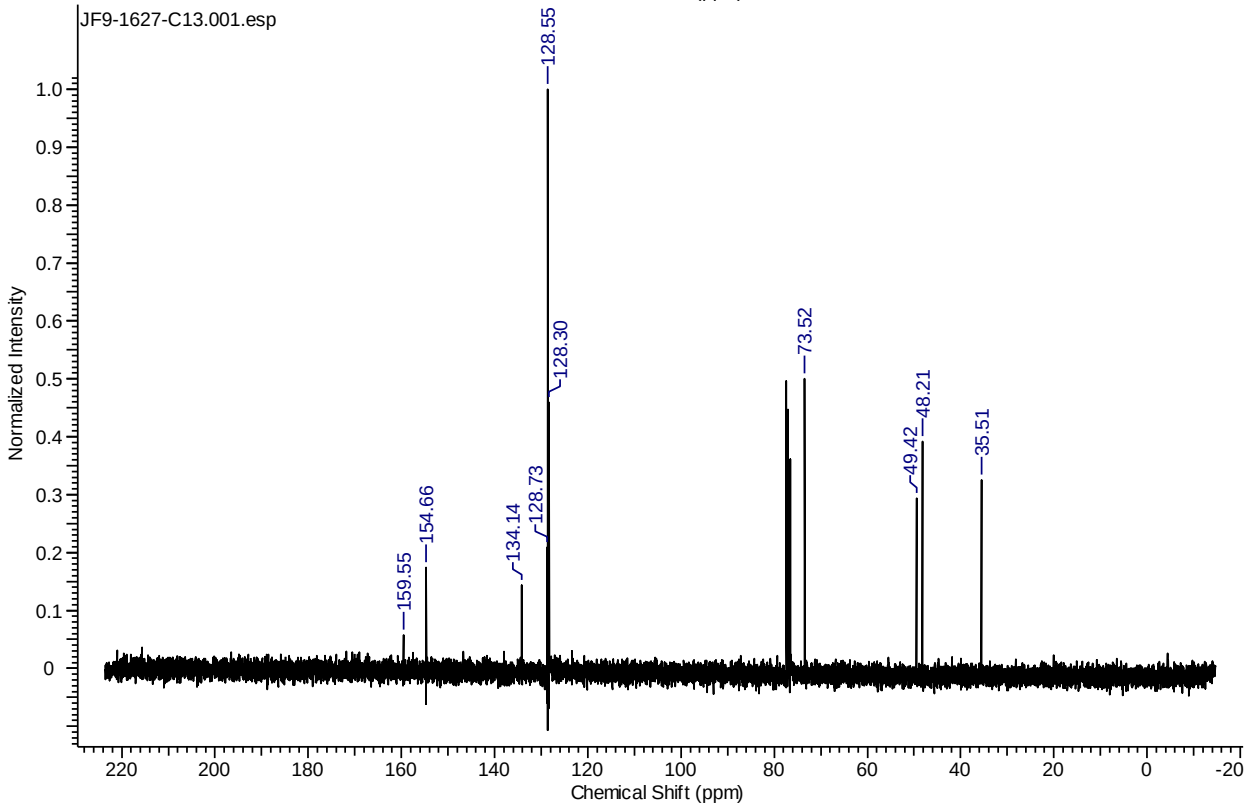
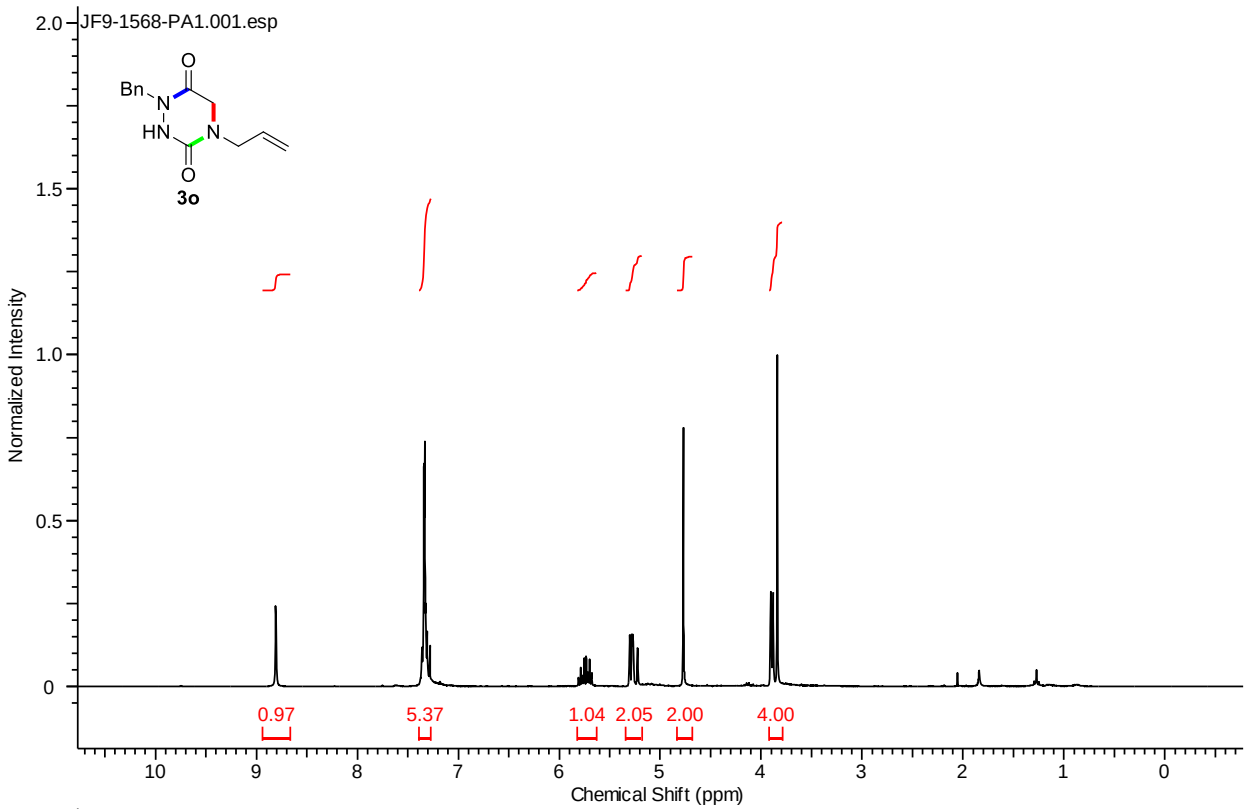
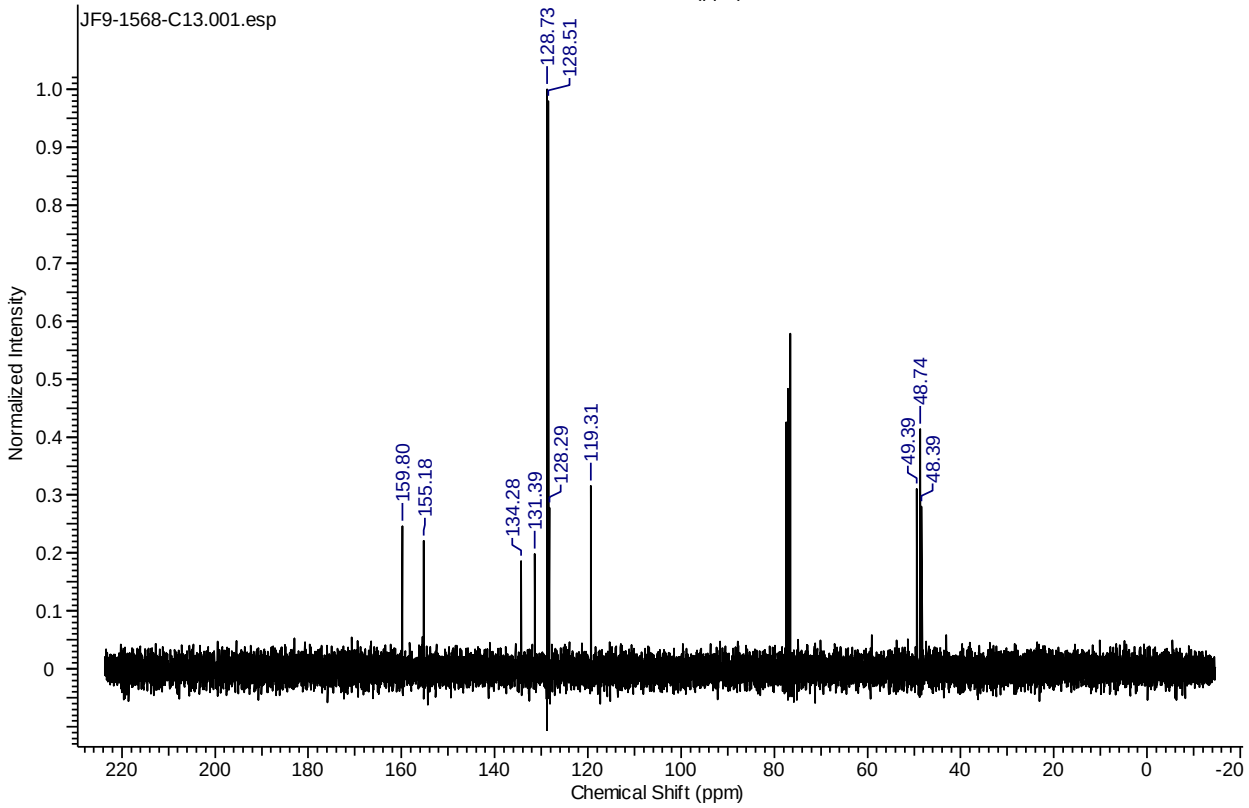
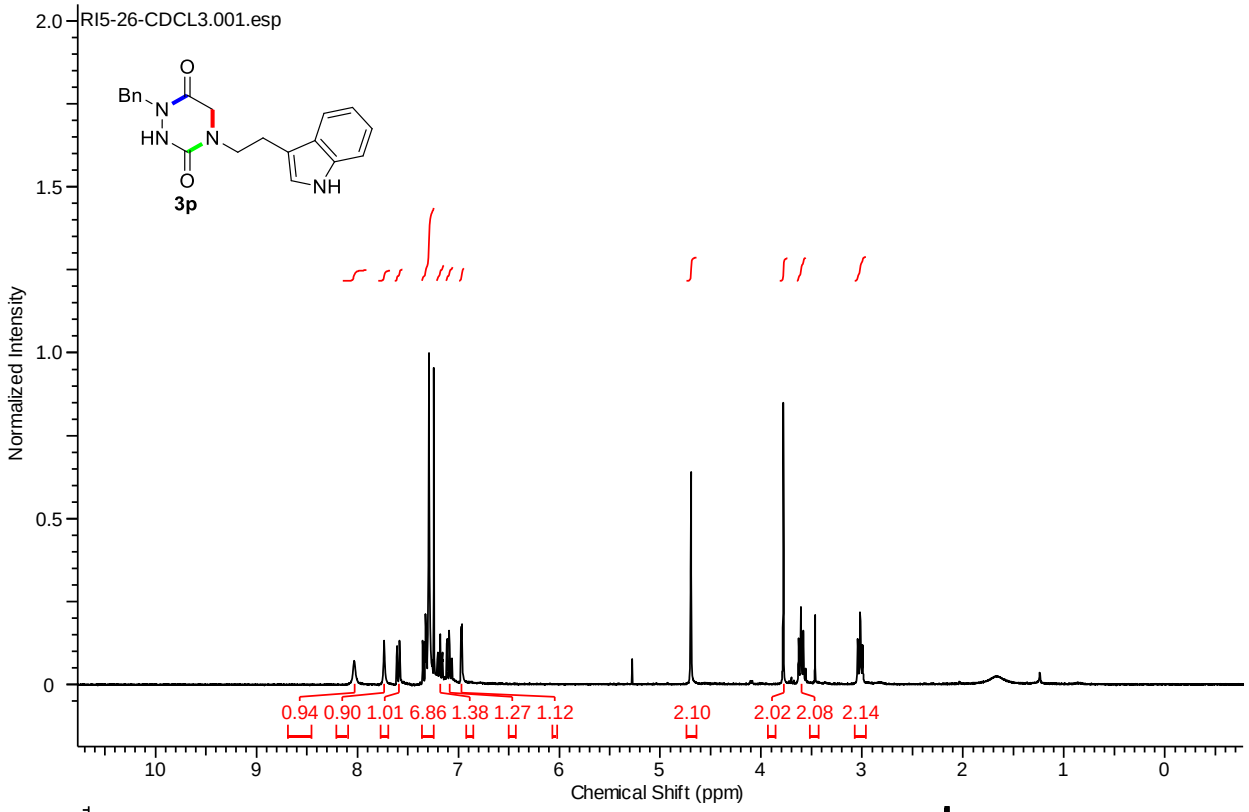
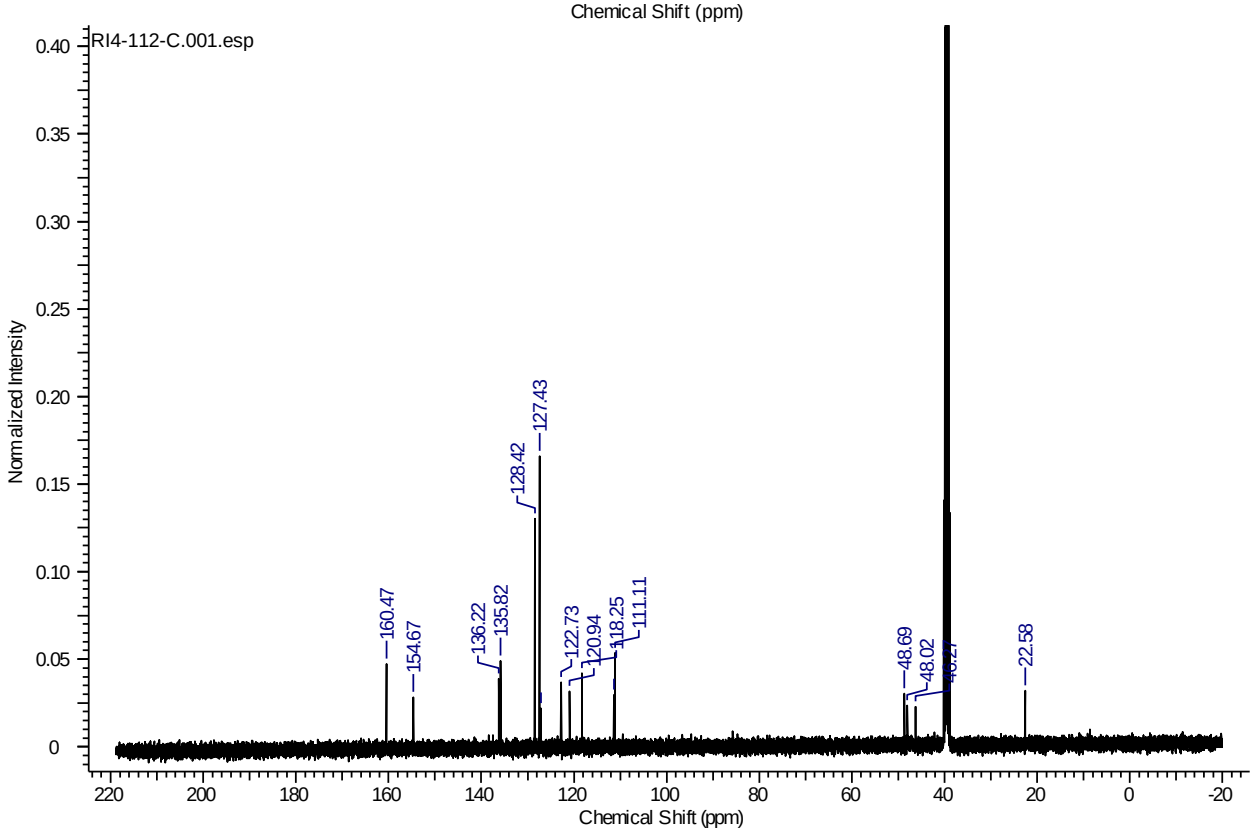
0.91
Chemical Shift (ppm)

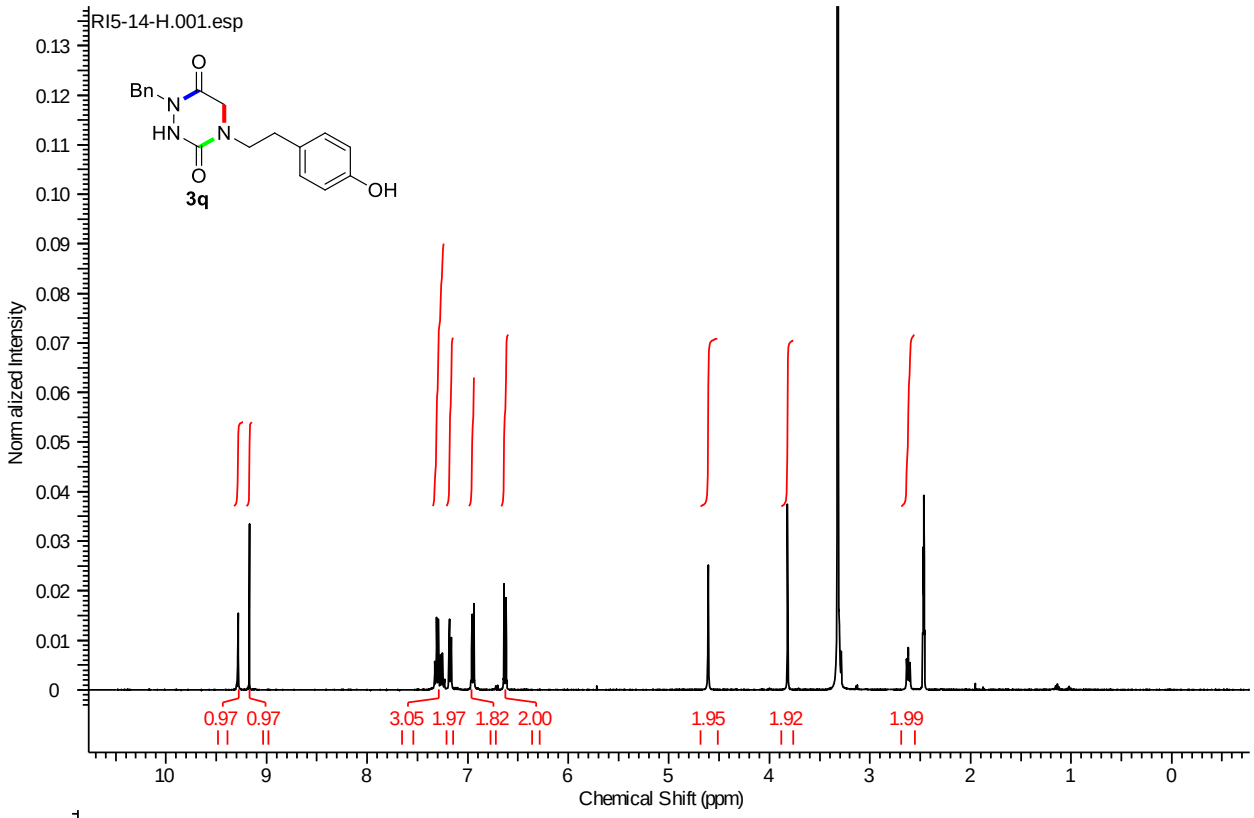

JF9-1627-C13.001.esp
128.55
128.30
73.52
49.42
48.21
35.51
159.55
154.66
134.14
128.73
Normalized Intensity
Chemical Shift (ppm)

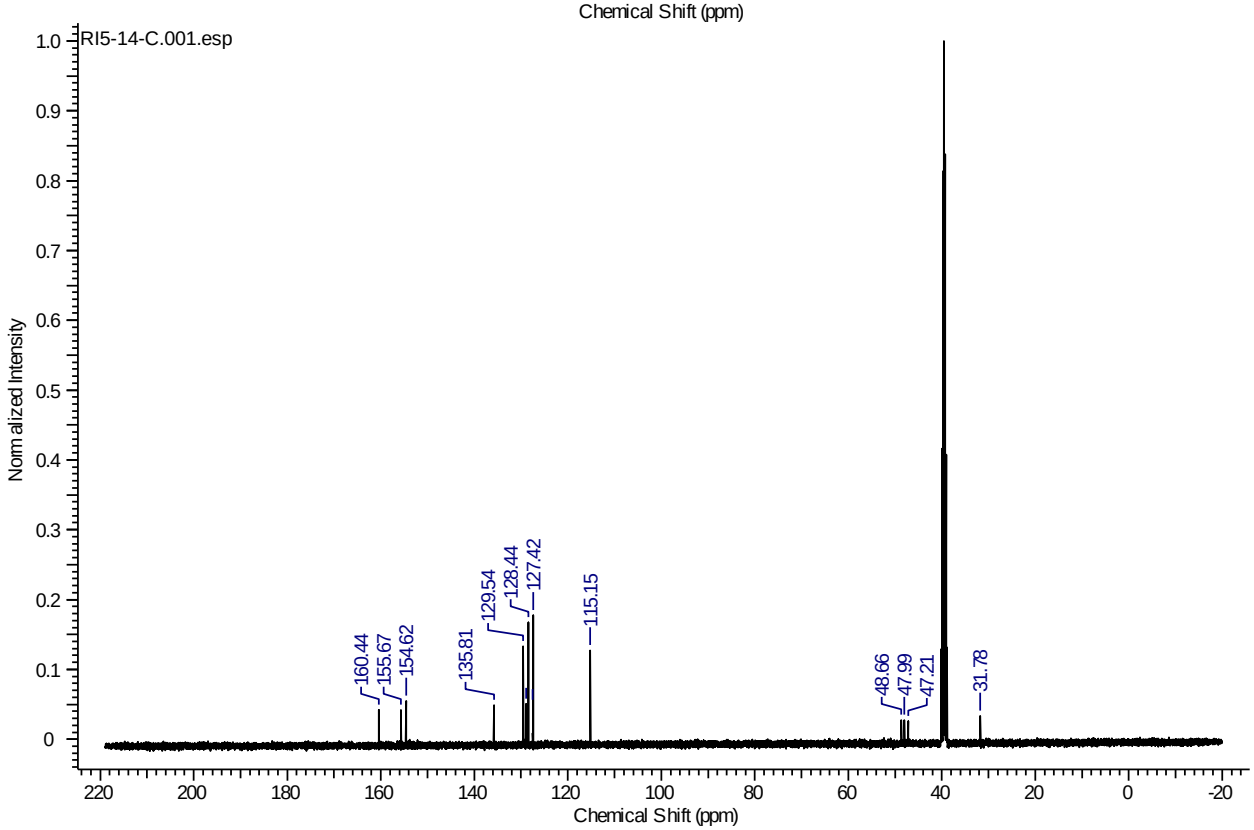

JF9-1568-PA1.001.esp
Bn
N
HN
N
O
O
3o
Normalized Intensity
0.97
5.37
1.04
2.05
2.00
4.00
Chemical Shift (ppm)

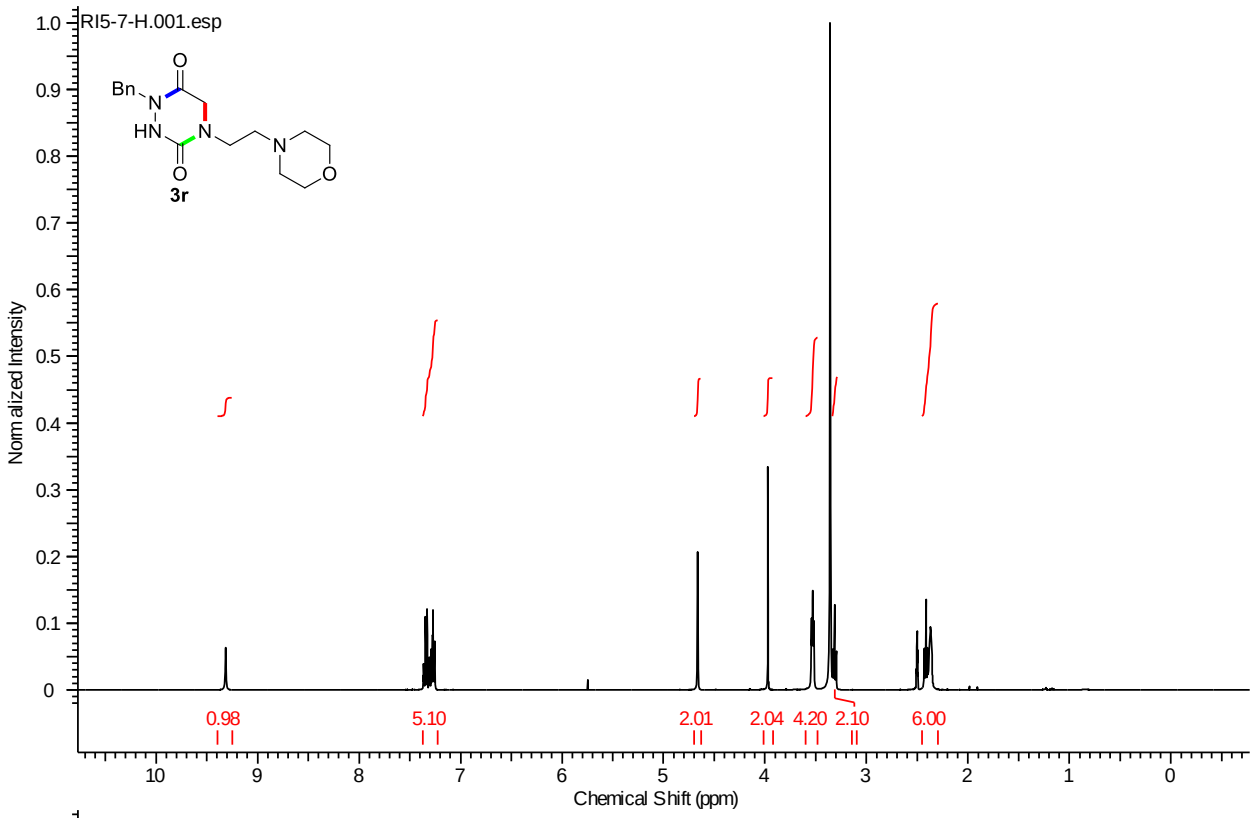

JF9-1568-C13.001.esp
128.73
128.51
159.80
155.18
134.28
131.39
128.29
119.31
49.39
48.74
48.39
Normalized Intensity
Chemical Shift (ppm)

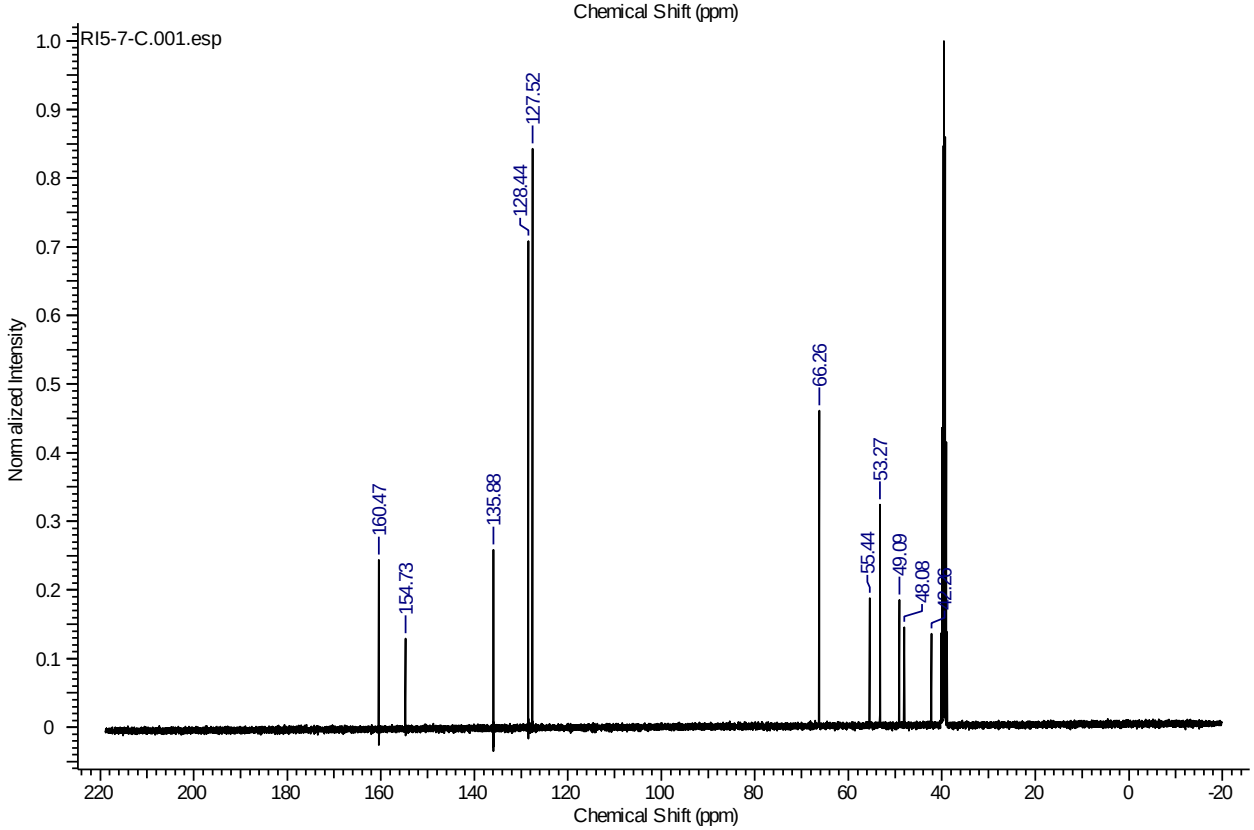

RI5-26-CDCL3.001.esp
Bn
N
HN
N
O
O
NH
3p
Normalized Intensity
Chemical Shift (ppm)
0.94 0.90 1.01 6.86 1.38 1.27 1.12
2.10
2.02 2.08 2.14

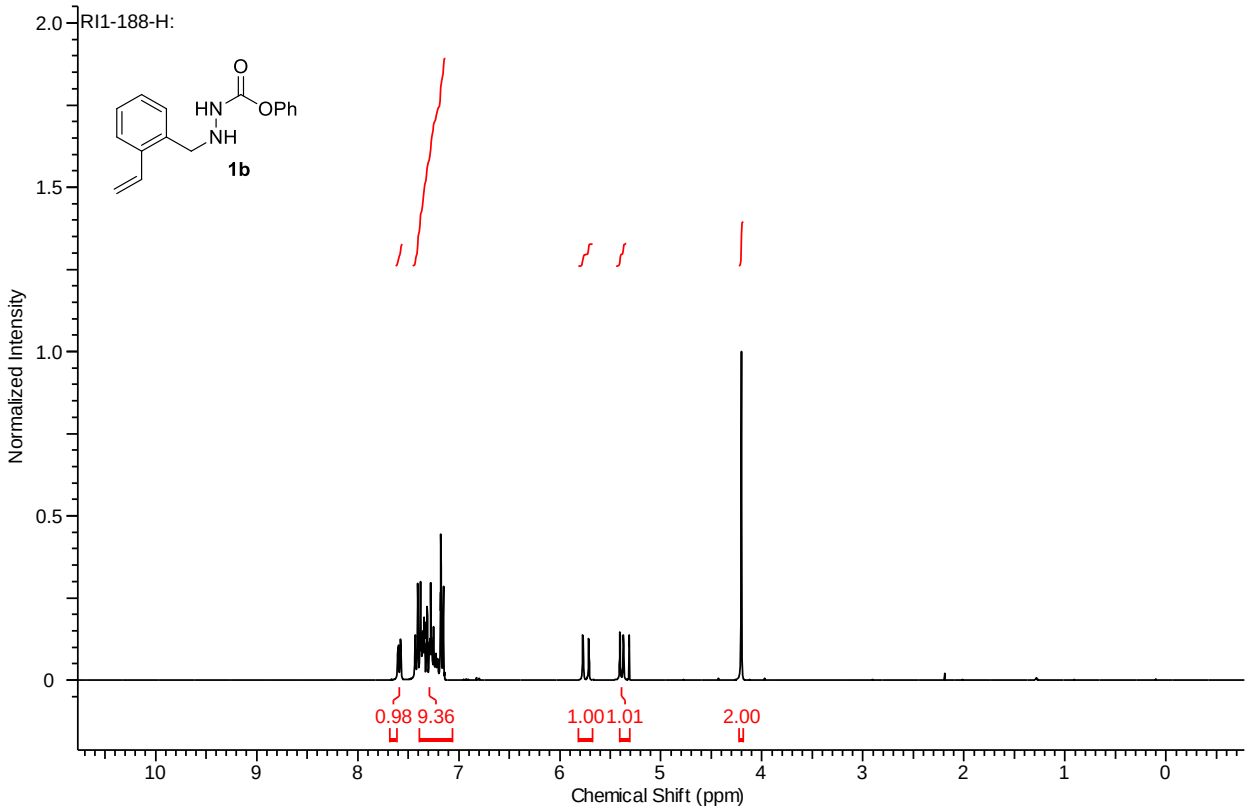

RI4-112-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
160.47
154.67
136.22
135.82
128.42
127.43
122.73
120.94
118.25
111.11
48.69
48.02
40.21
22.58

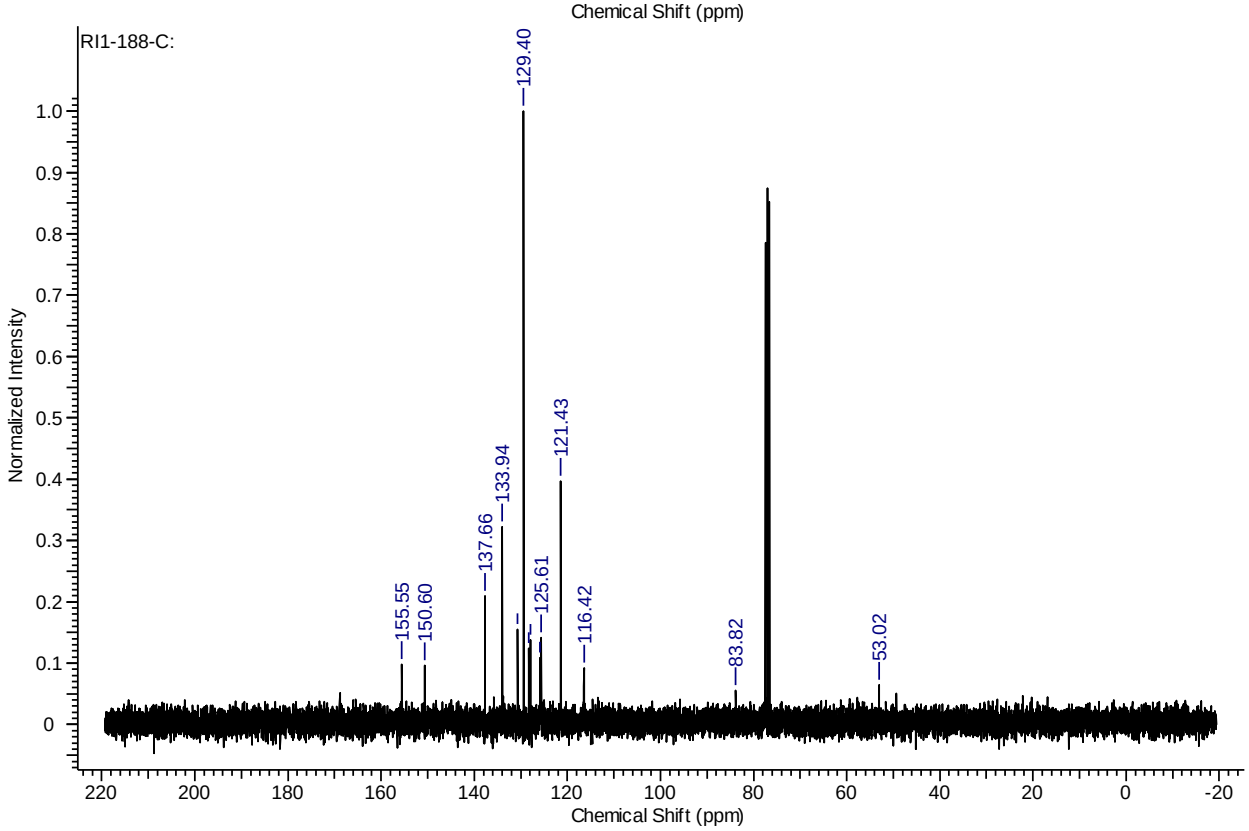

RI5-14-H.001.esp
Bn
N
HN
N
O
O
OH
3q
Normalized Intensity
0.97 0.97
3.05 1.97 1.82 2.00
1.95
1.92
1.99
Chemical Shift (ppm)

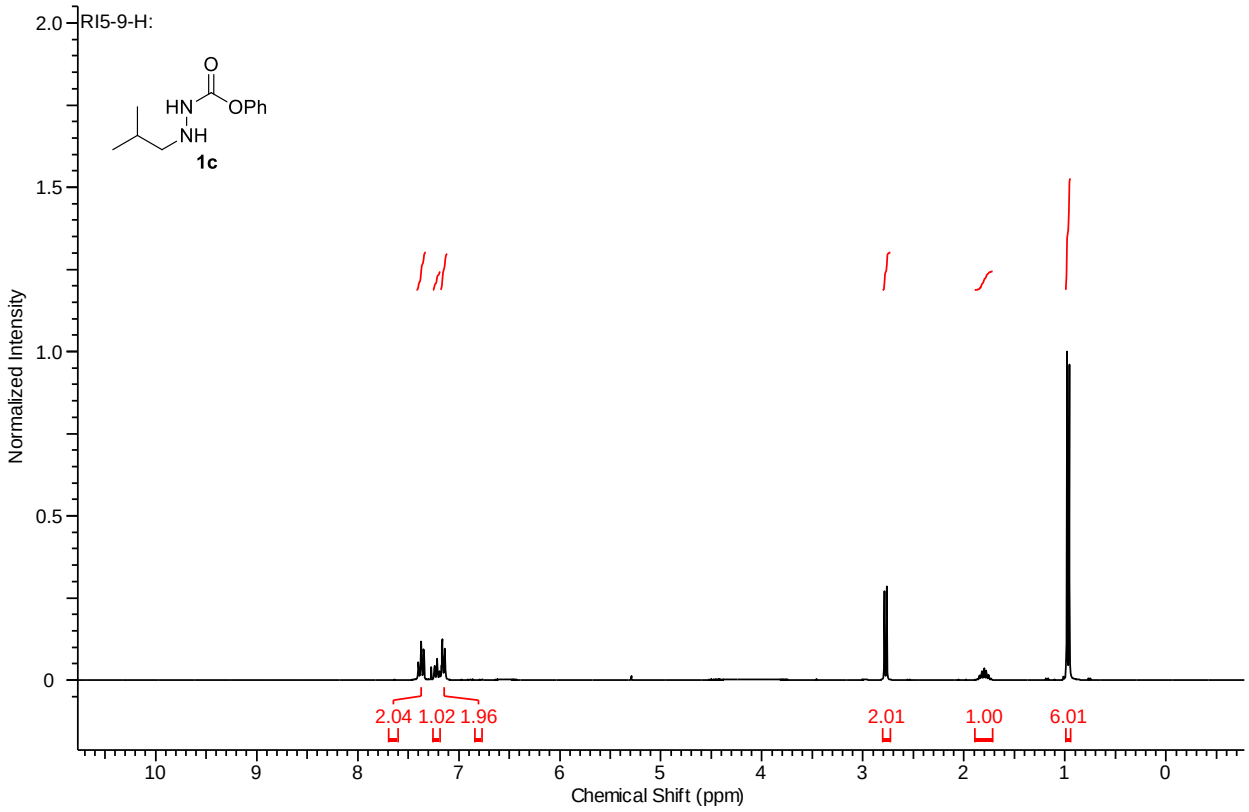

RI5-14-C.001.esp
Normalized Intensity
160.44
155.67
154.62
135.81
129.54
128.44
127.42
115.15
48.66
47.99
47.21
31.78
Chemical Shift (ppm)

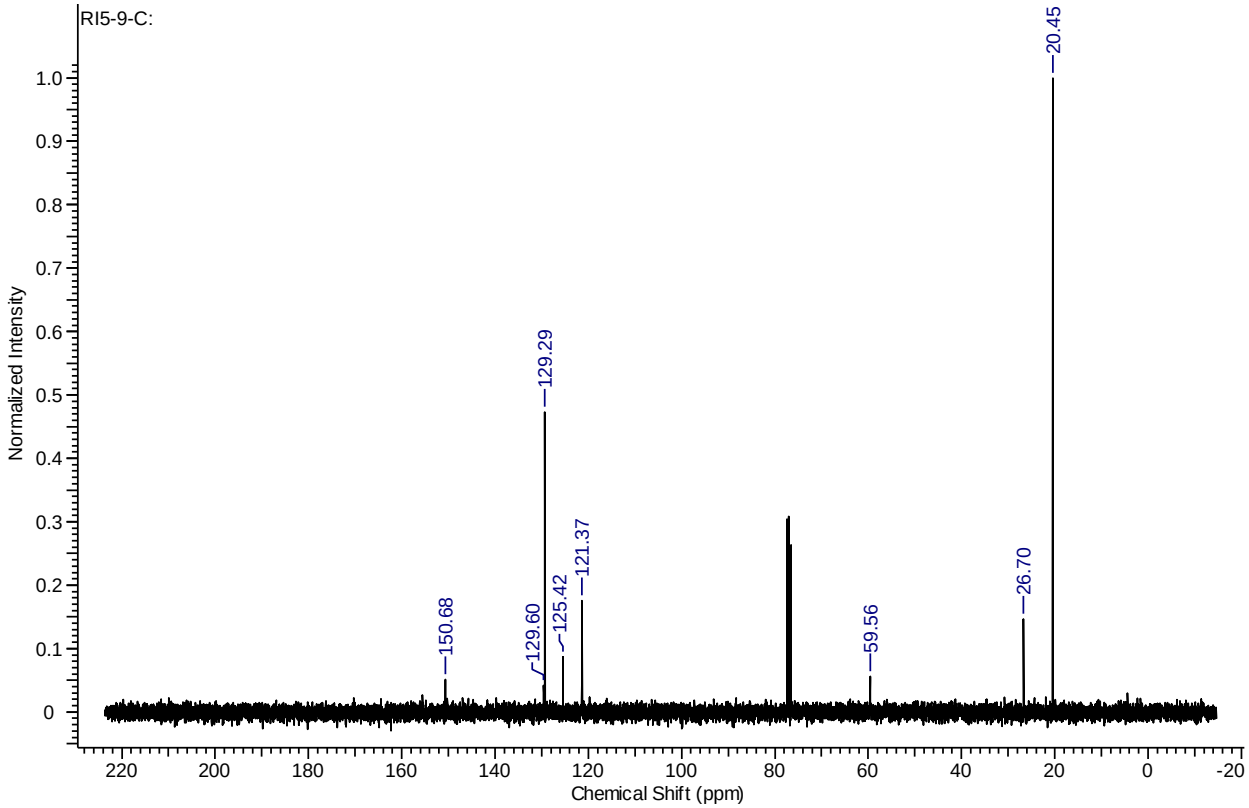

RI5-7-H.001.esp
Bn
N
HN
N
N
O
O
O
3r
Normalized Intensity
0.98
5.10
2.01
2.04
4.20
2.10
6.00
Chemical Shift (ppm)

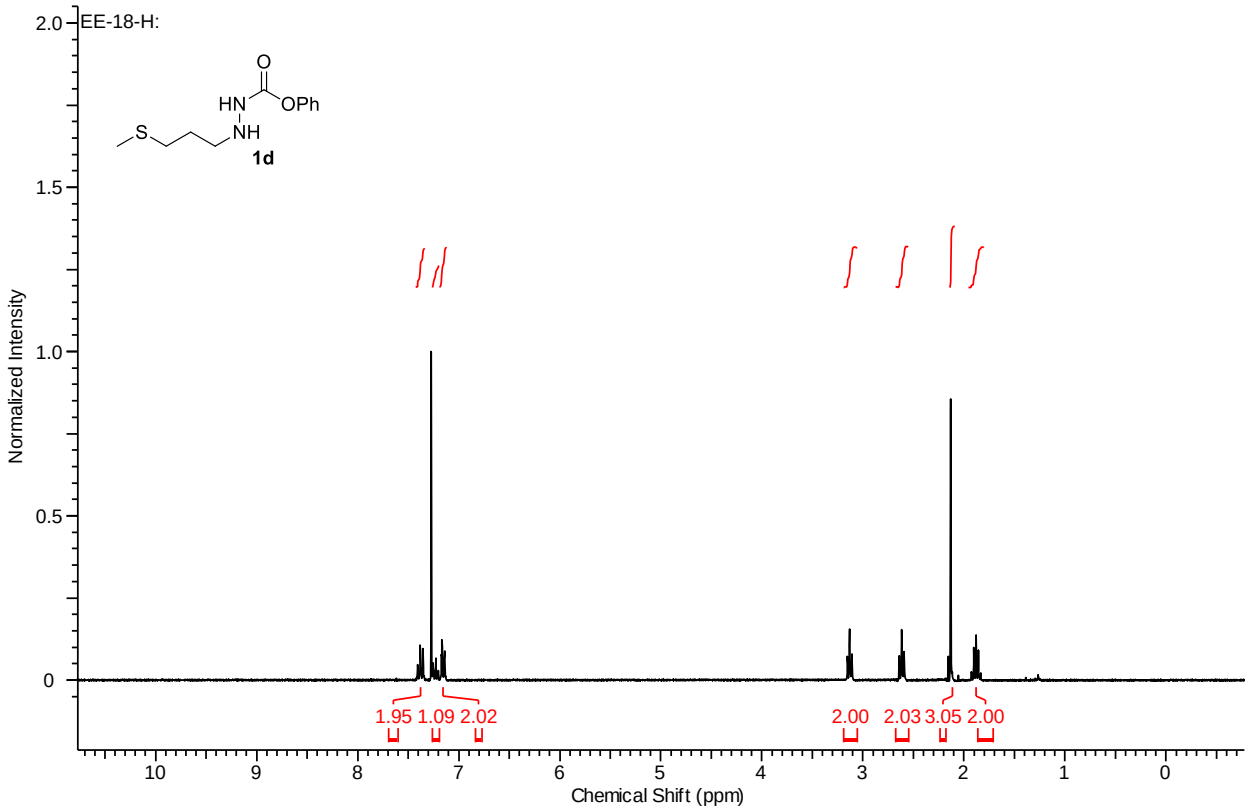

RI5-7-C.001.esp
127.52
128.44
66.26
160.47
154.73
135.88
53.27
55.44
49.09
48.08
Normalized Intensity
Chemical Shift (ppm)

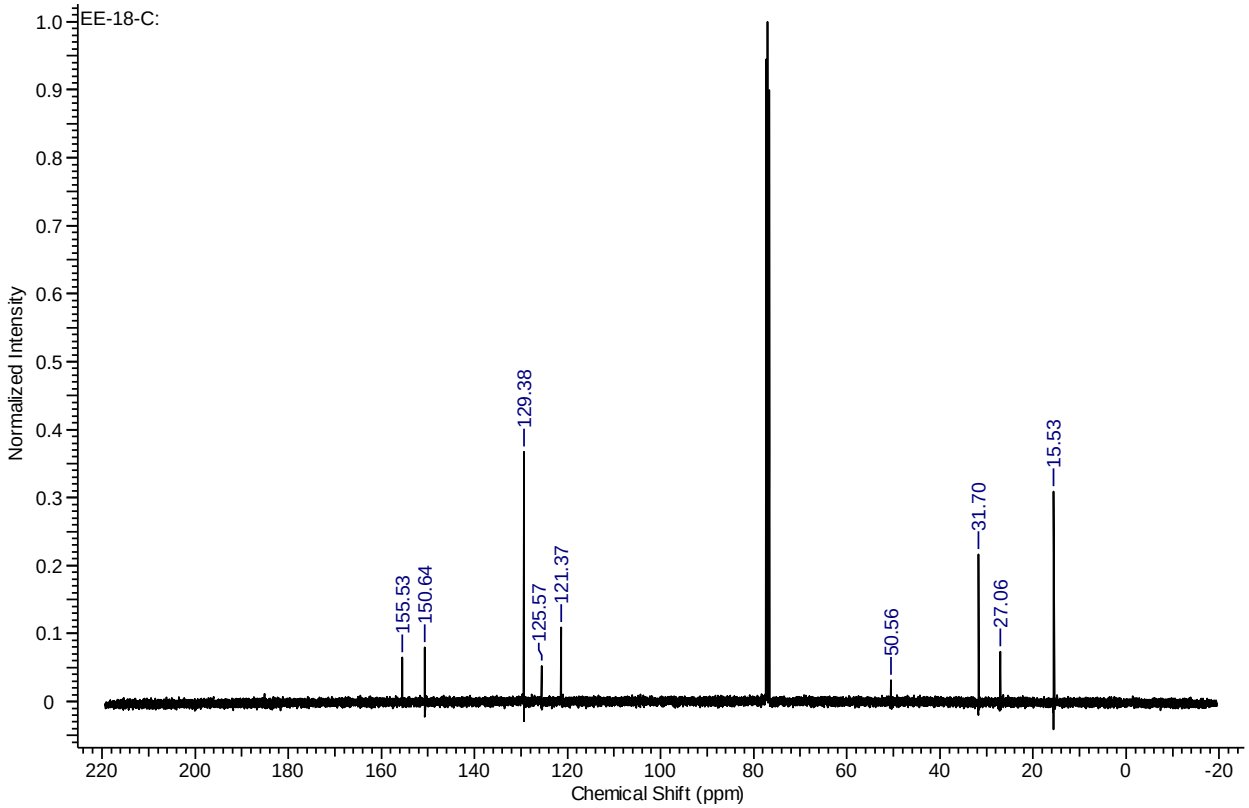

RI1-188-H:
HN
NH
O
OPh
1b
Normalized Intensity
Chemical Shift (ppm)
0.98
9.36
1.00
1.01
2.00

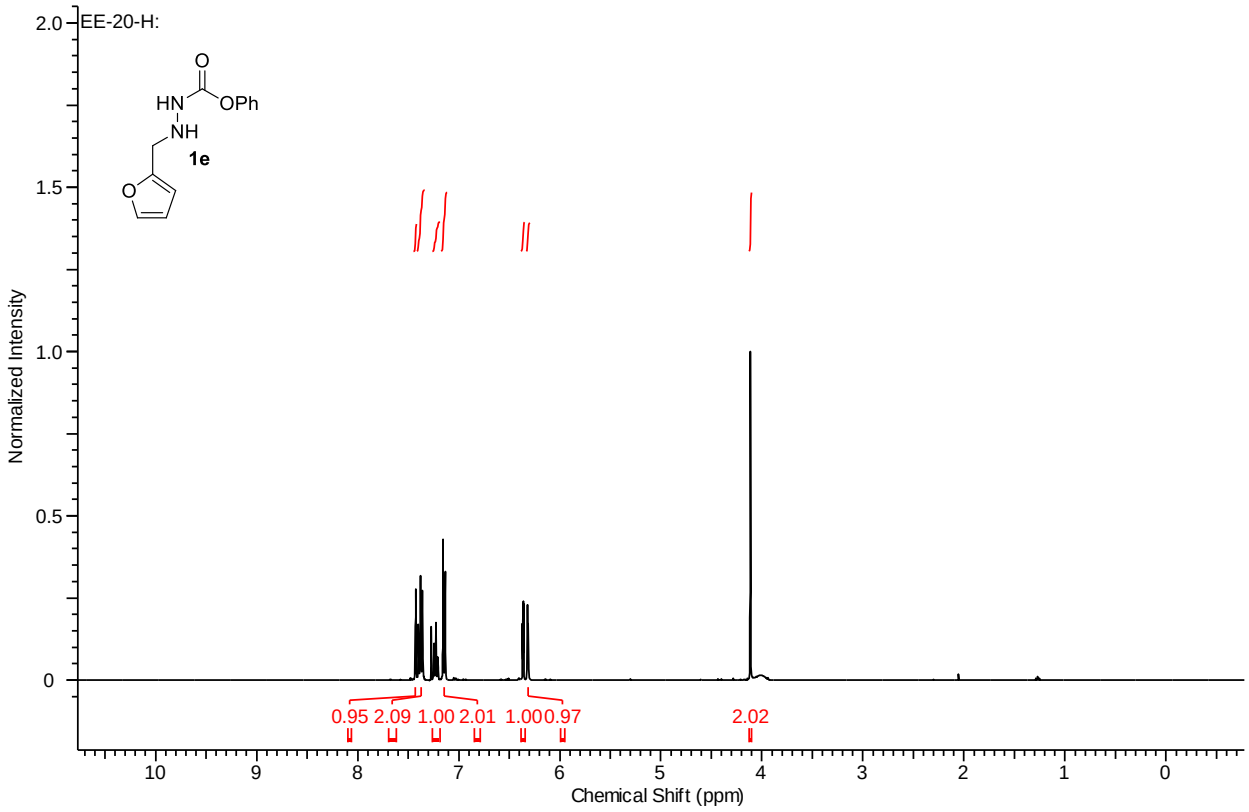

RI1-188-C:
Normalized Intensity
Chemical Shift (ppm)
129.40
133.94
137.66
121.43
155.55
150.60
125.61
116.42
83.82
53.02

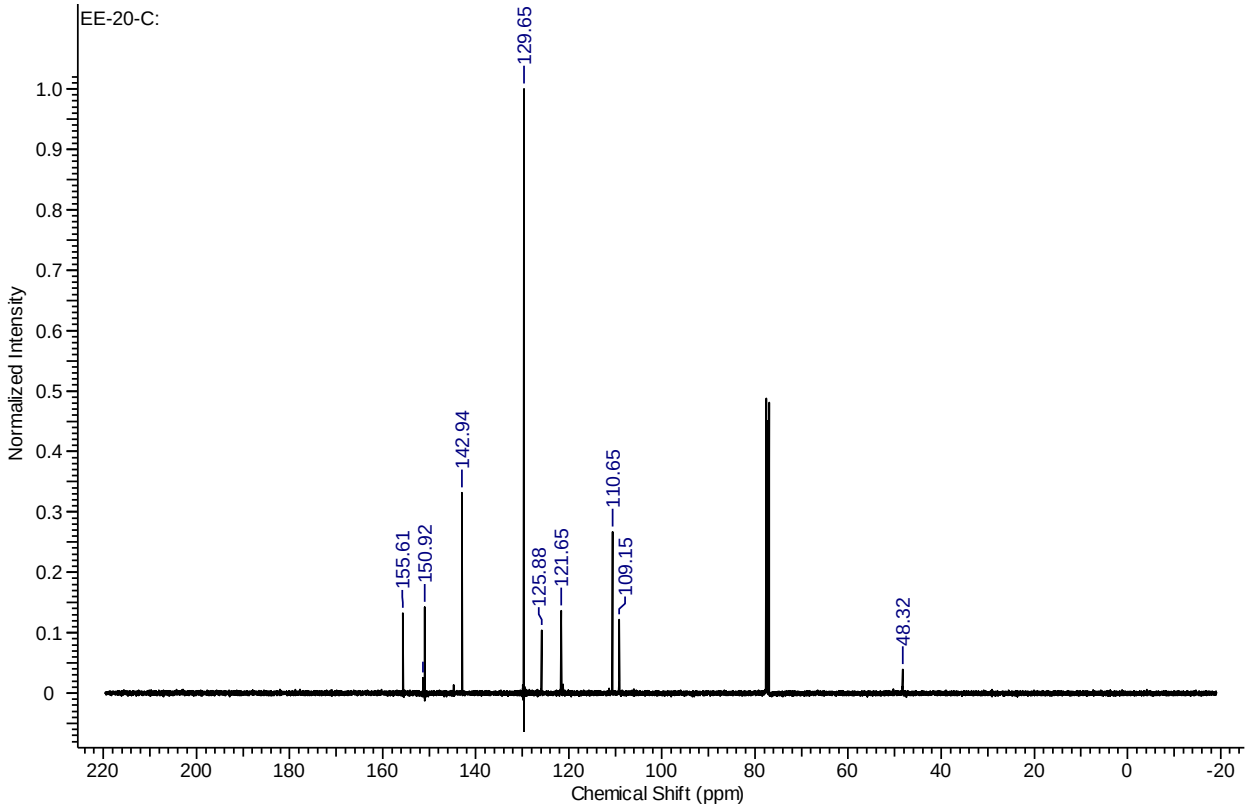

RI5-9-H:
O
HN
OPh
NH
1c
Normalized Intensity
2.04 1.02 1.96
2.01
1.00
6.01
Chemical Shift (ppm)

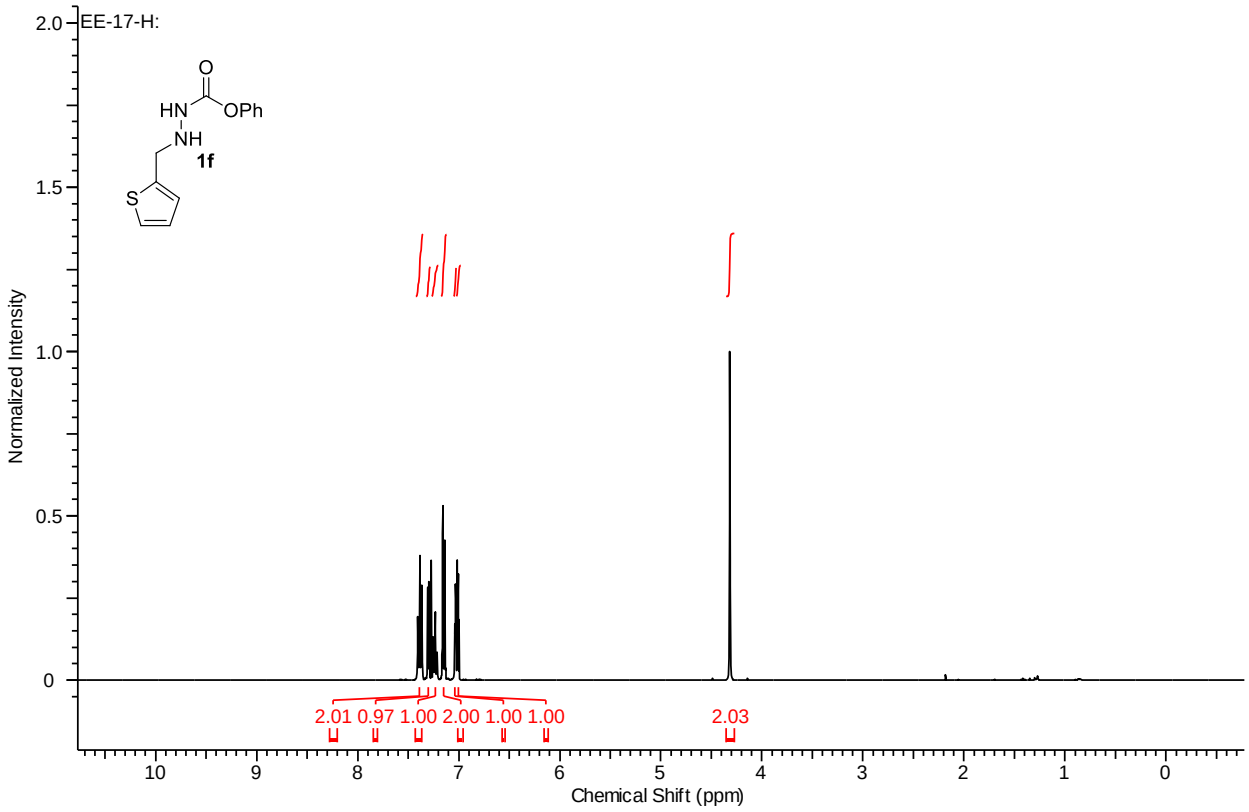

RI5-9-C:
Normalized Intensity
150.68
129.60
129.29
125.42
121.37
59.56
26.70
20.45
Chemical Shift (ppm)

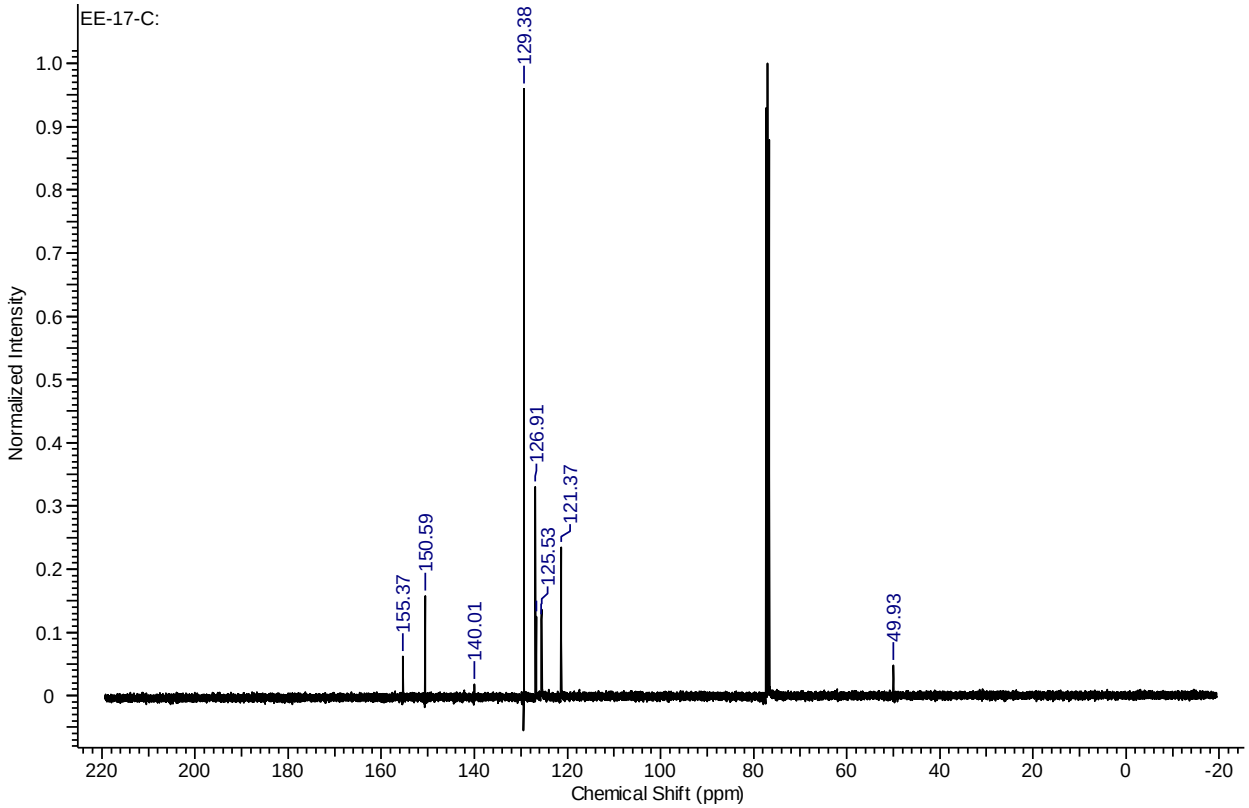

EE-18-H:
HN
O
OPh
NH
S
1d
Normalized Intensity
Chemical Shift (ppm)
1.95
1.09
2.02
2.00
2.03
3.05
2.00

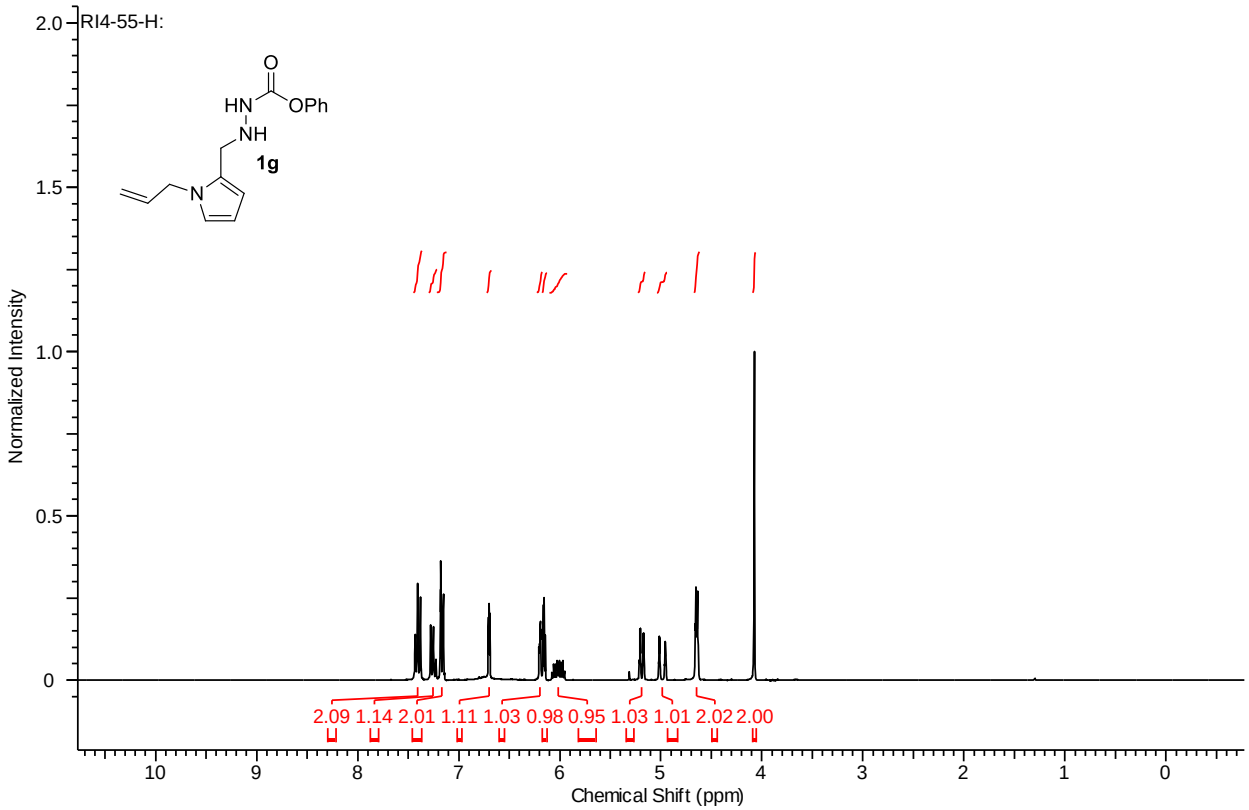

EE-18-C:
Normalized Intensity
Chemical Shift (ppm)
155.53
150.64
129.38
125.57
121.37
50.56
31.70
27.06
15.53

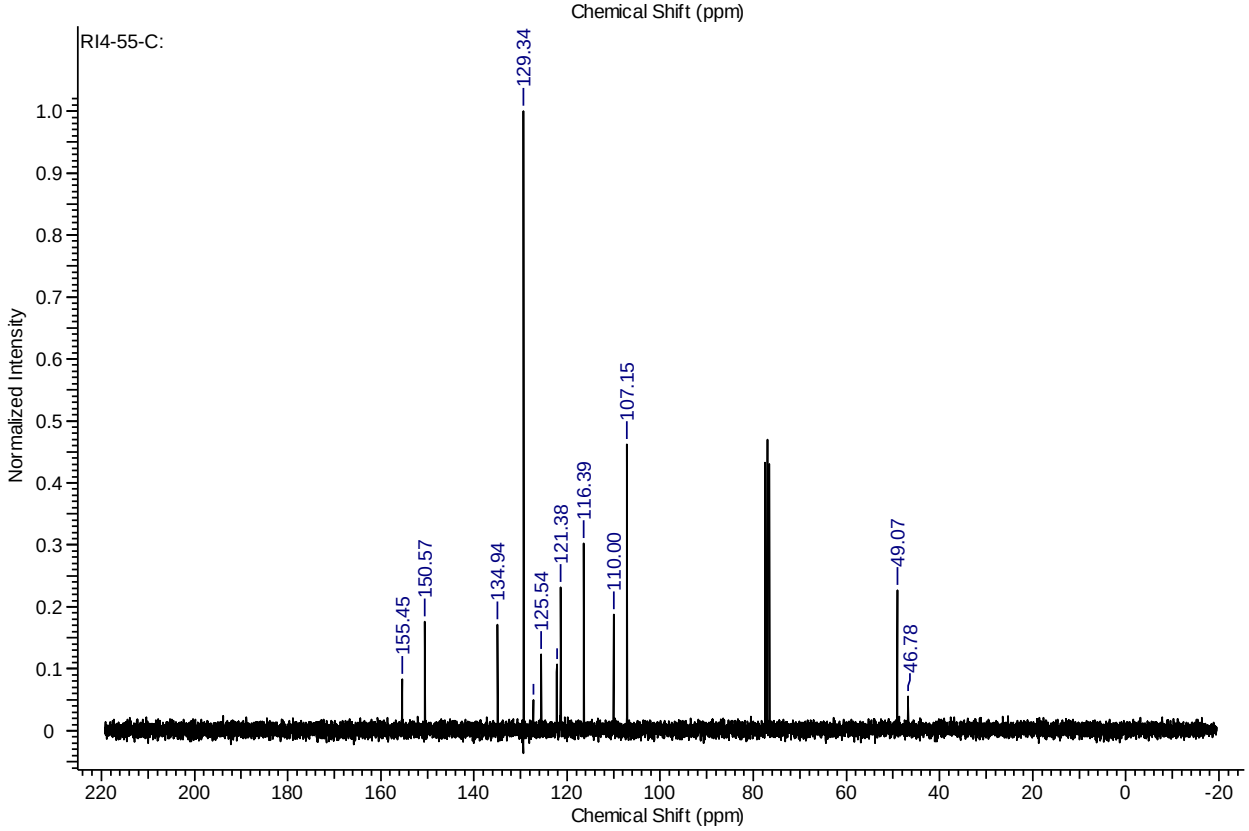

EE-20-H:
HN
OPh
NH
1e
O
Normalized Intensity
0.95 2.09 1.00 2.01 1.00 0.97
2.02
Chemical Shift (ppm)

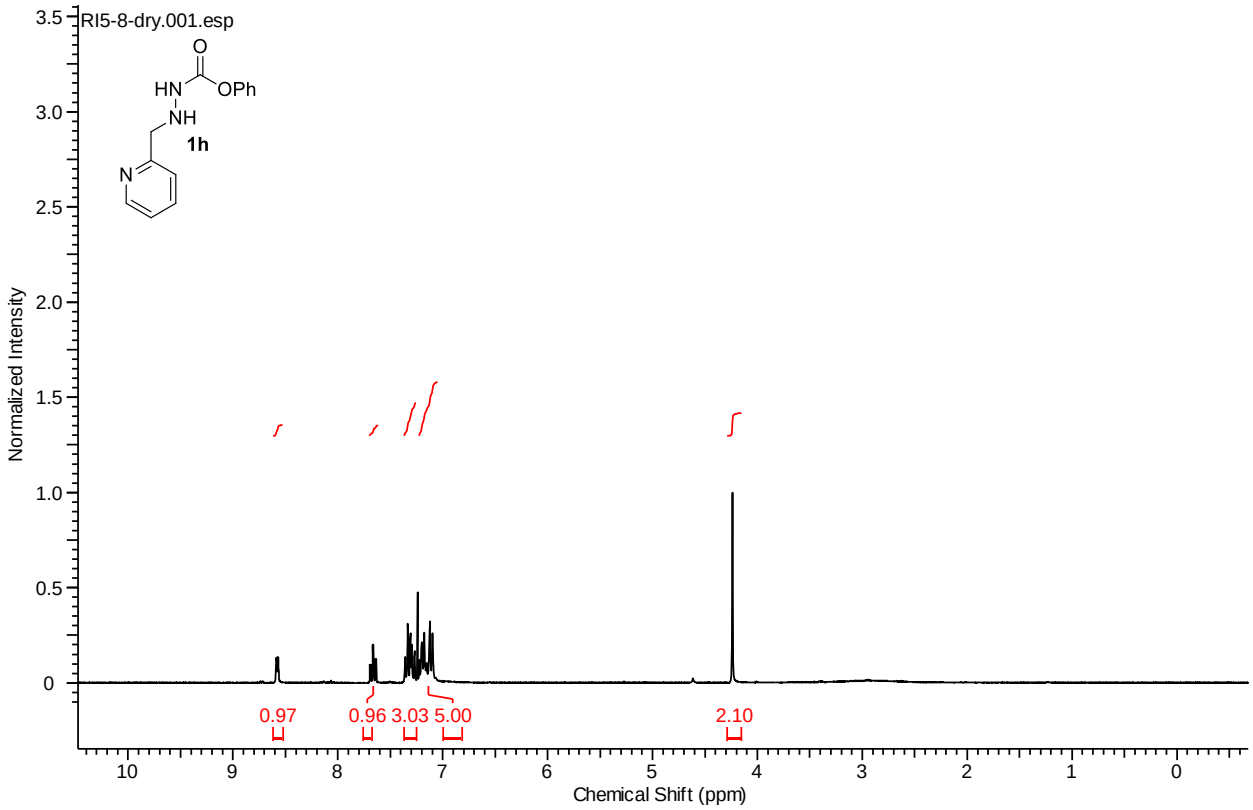

EE-20-C:
129.65
142.94
155.61
150.92
125.88
121.65
110.65
109.15
48.32
Normalized Intensity
Chemical Shift (ppm)

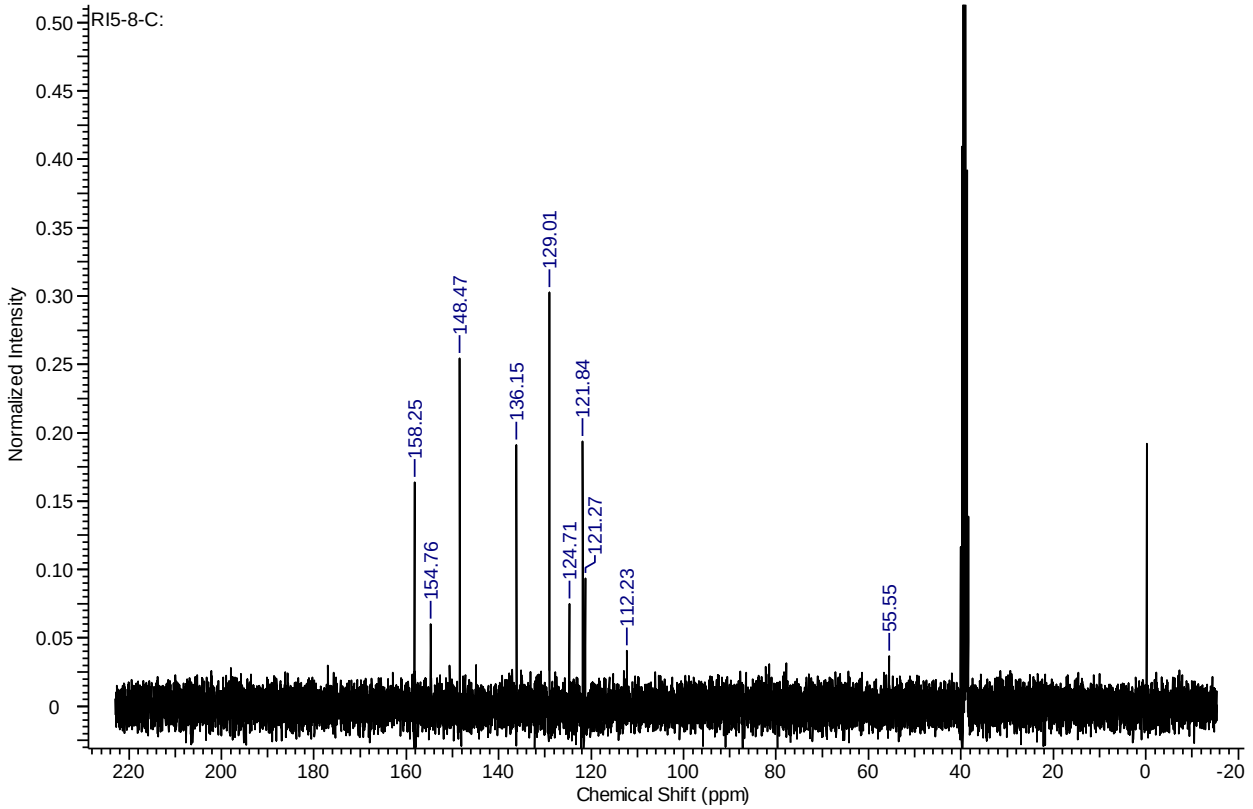

EE-17-H:
O
HN
OPh
NH
1f
S
Normalized Intensity
2.01 0.97 1.00 2.00 1.00 1.00
2.03
Chemical Shift (ppm)

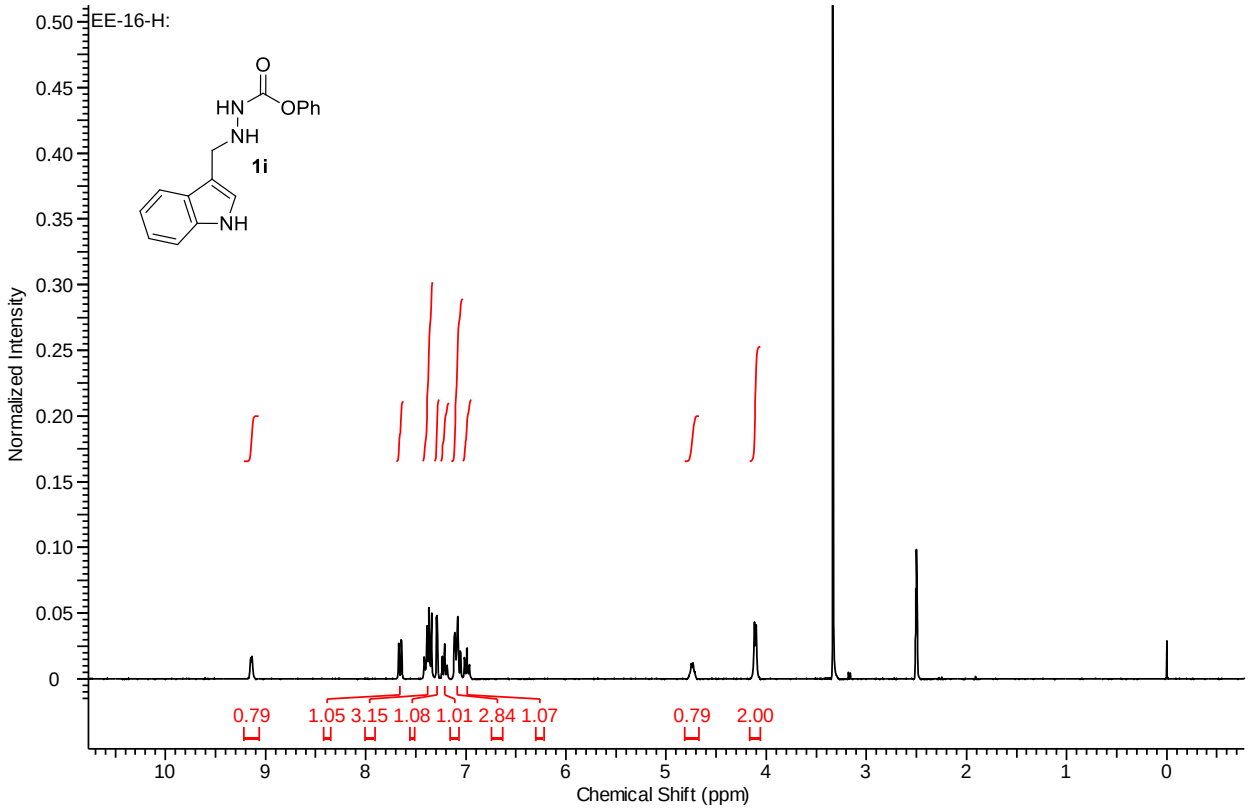

EE-17-C:
129.38
126.91
121.37
125.53
155.37
150.59
140.01
49.93
Normalized Intensity
Chemical Shift (ppm)

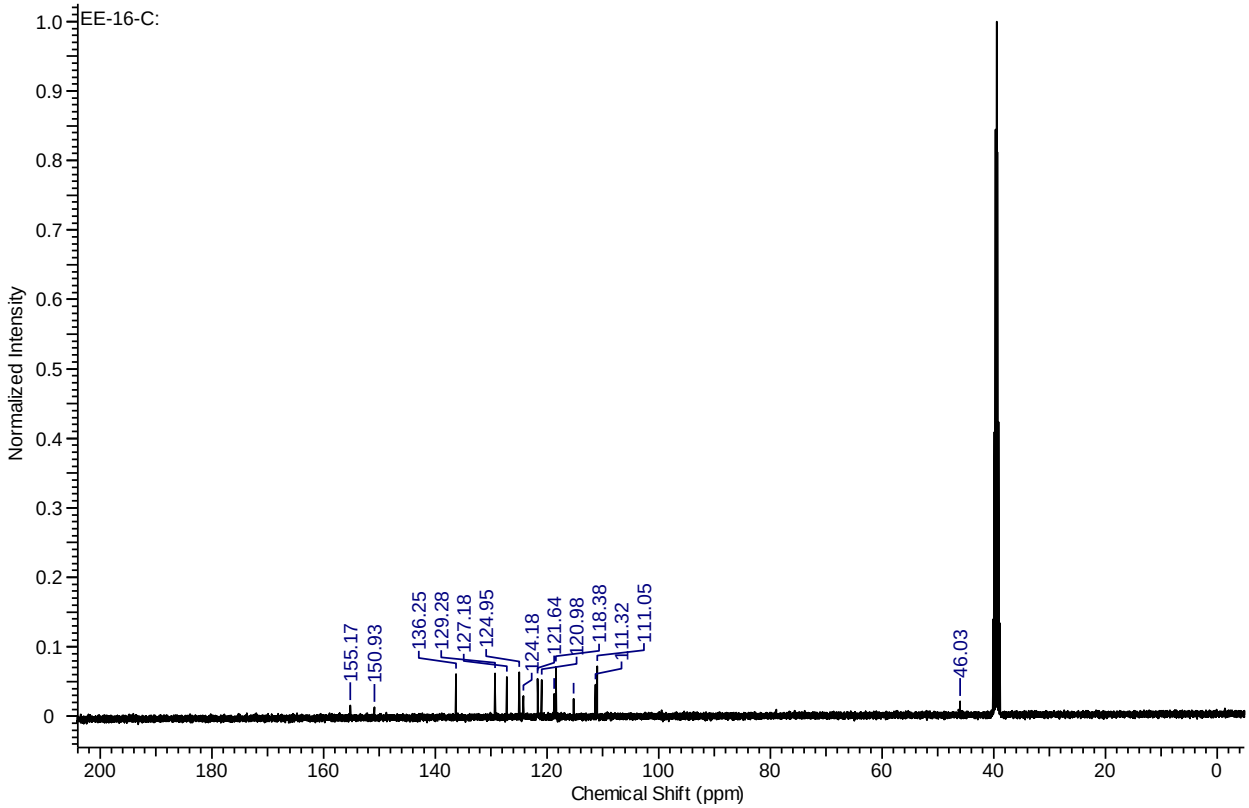

RI4-55-H:
HN
O
OPh
NH
1g
N
Normalized Intensity
2.09 1.14 2.01 1.11 1.03 0.98 0.95 1.03 1.01 2.02 2.00
Chemical Shift (ppm)

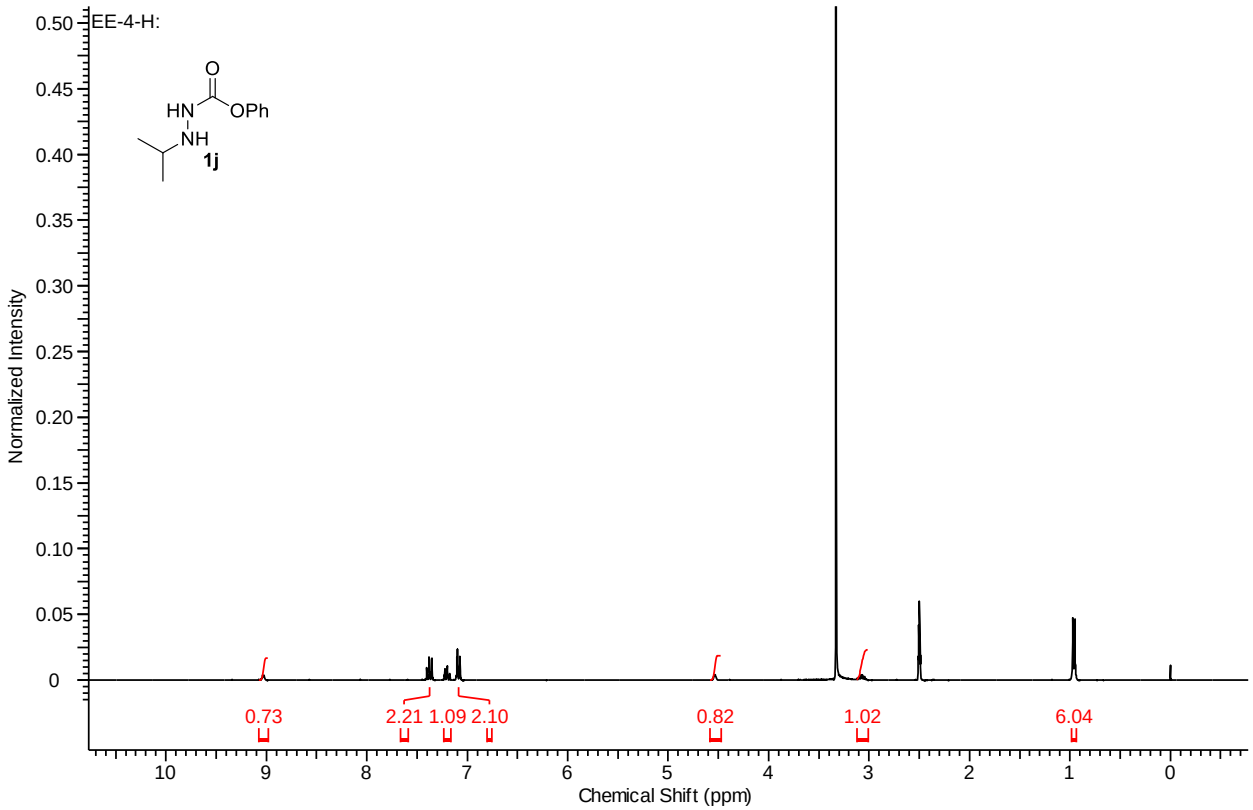

RI4-55-C:
129.34
155.45
150.57
134.94
125.54
121.38
116.39
110.00
107.15
49.07
46.78
Normalized Intensity
Chemical Shift (ppm)

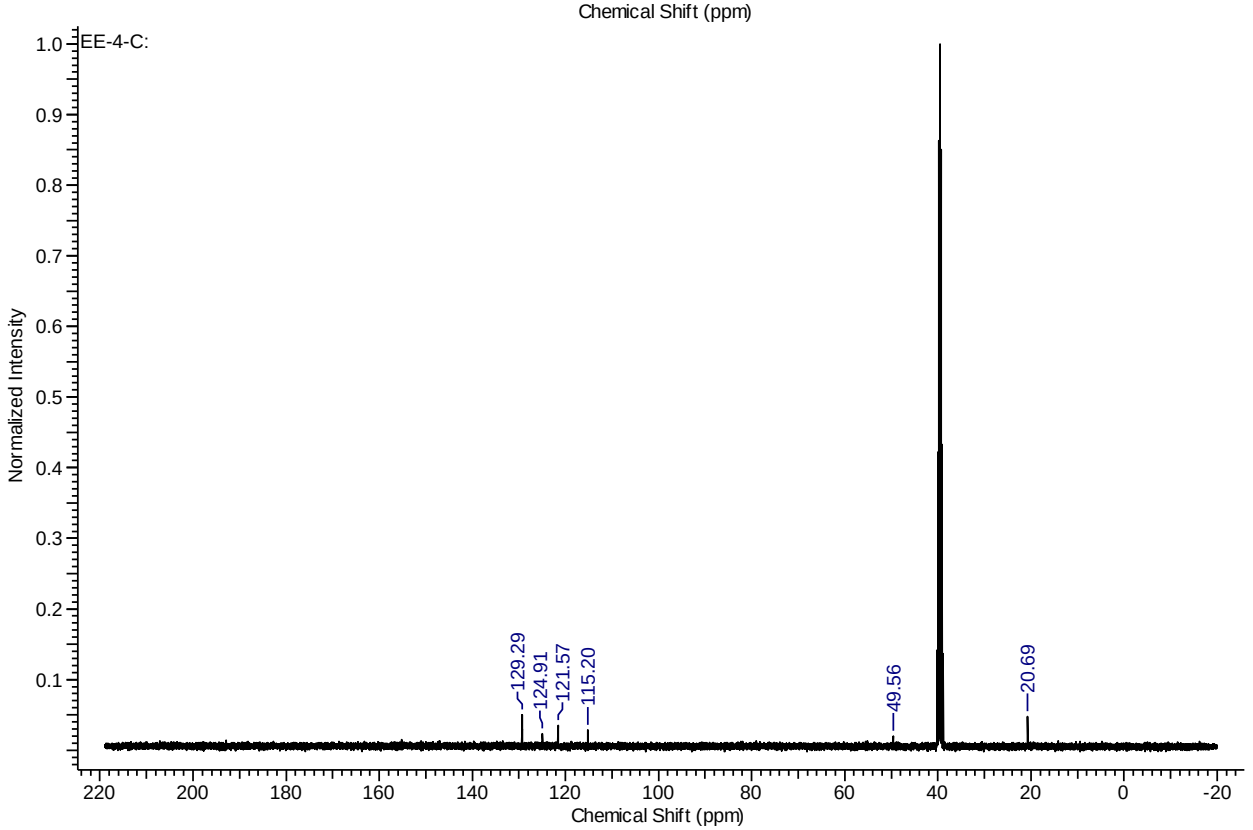

RI5-8-dry.001.esp
O
HN
OPh
NH
1h
N
Normalized Intensity
0.97
0.96
3.03
5.00
2.10
Chemical Shift (ppm)

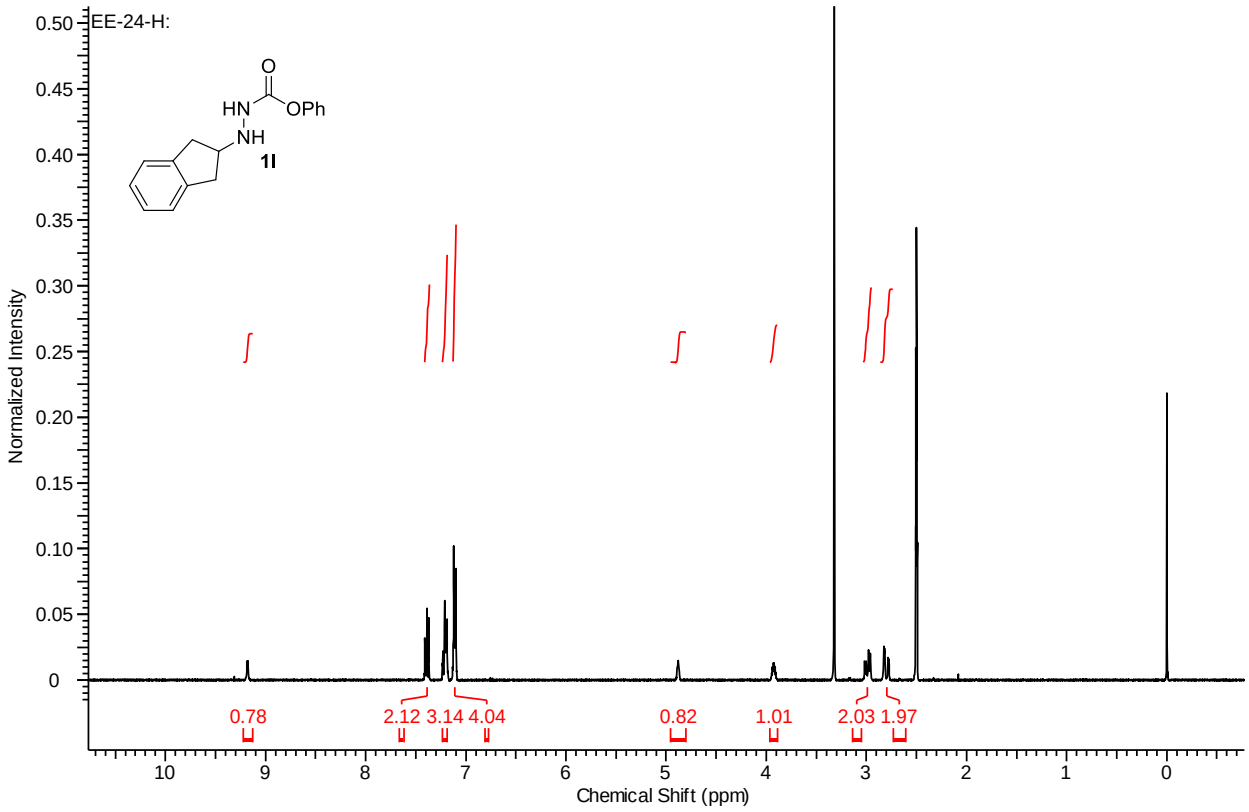

RI5-8-C:
158.25
154.76
148.47
136.15
129.01
124.71
121.84
121.27
112.23
55.55
Normalized Intensity
Chemical Shift (ppm)

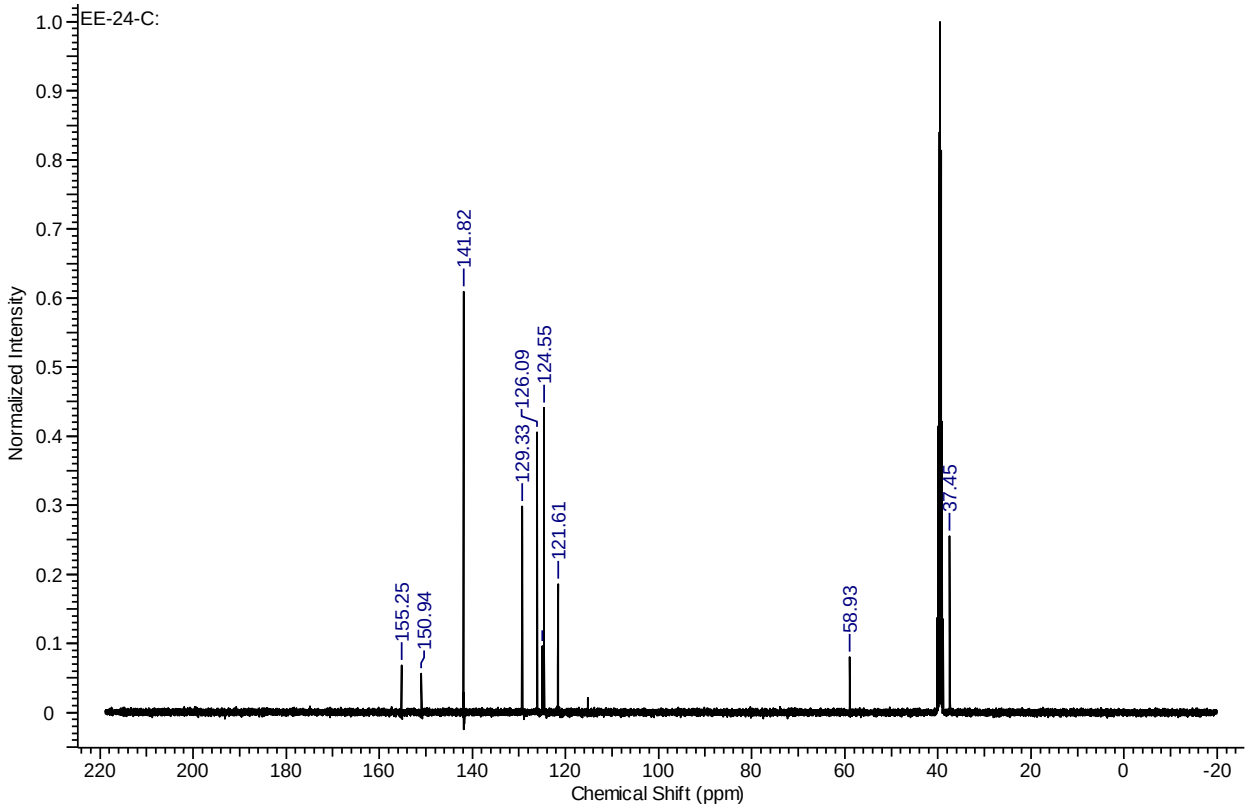

EE-16-H:
HN
O
OPh
NH
1i
NH
Normalized Intensity
0.79
1.05
3.15
1.08
1.01
2.84
1.07
0.79
2.00
Chemical Shift (ppm)

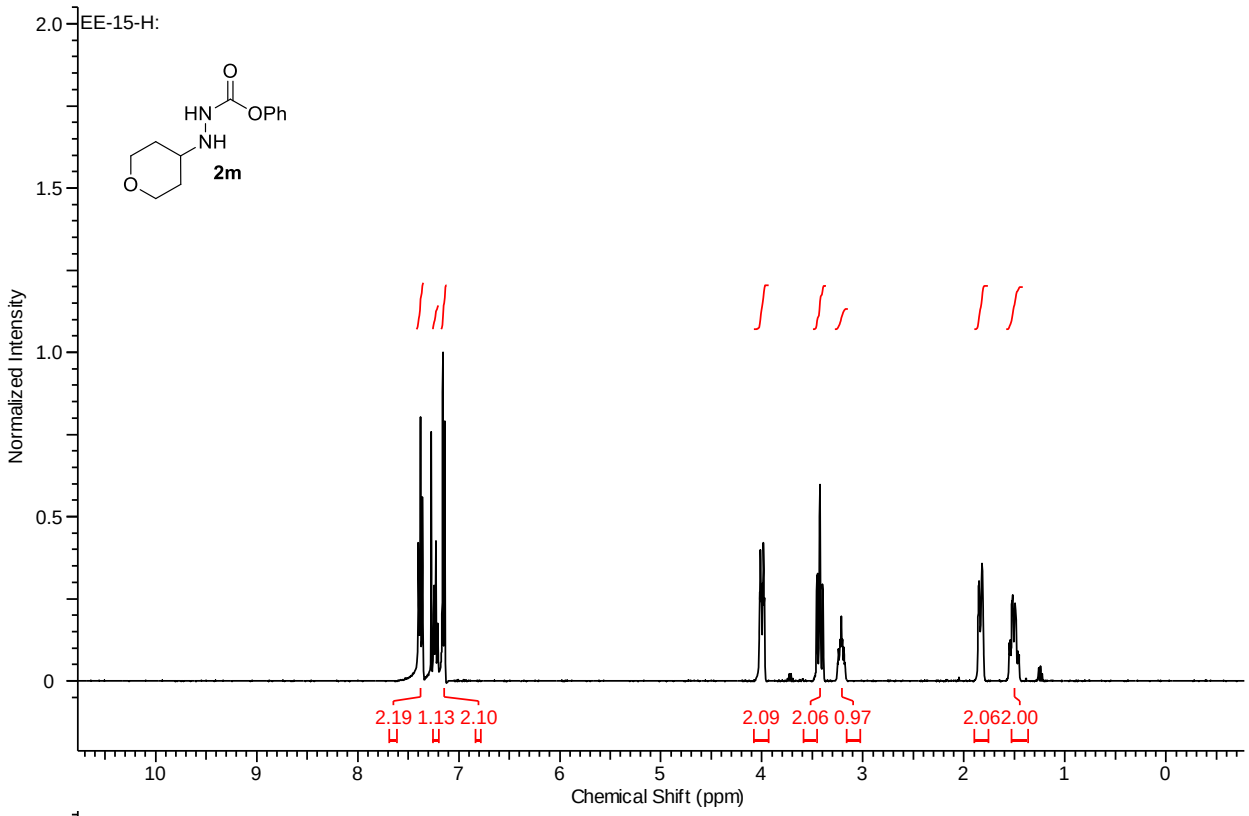

EE-16-C:
Normalized Intensity
155.17
150.93
136.25
129.28
127.18
124.95
124.18
121.64
120.98
118.38
111.32
111.05
46.03
Chemical Shift (ppm)

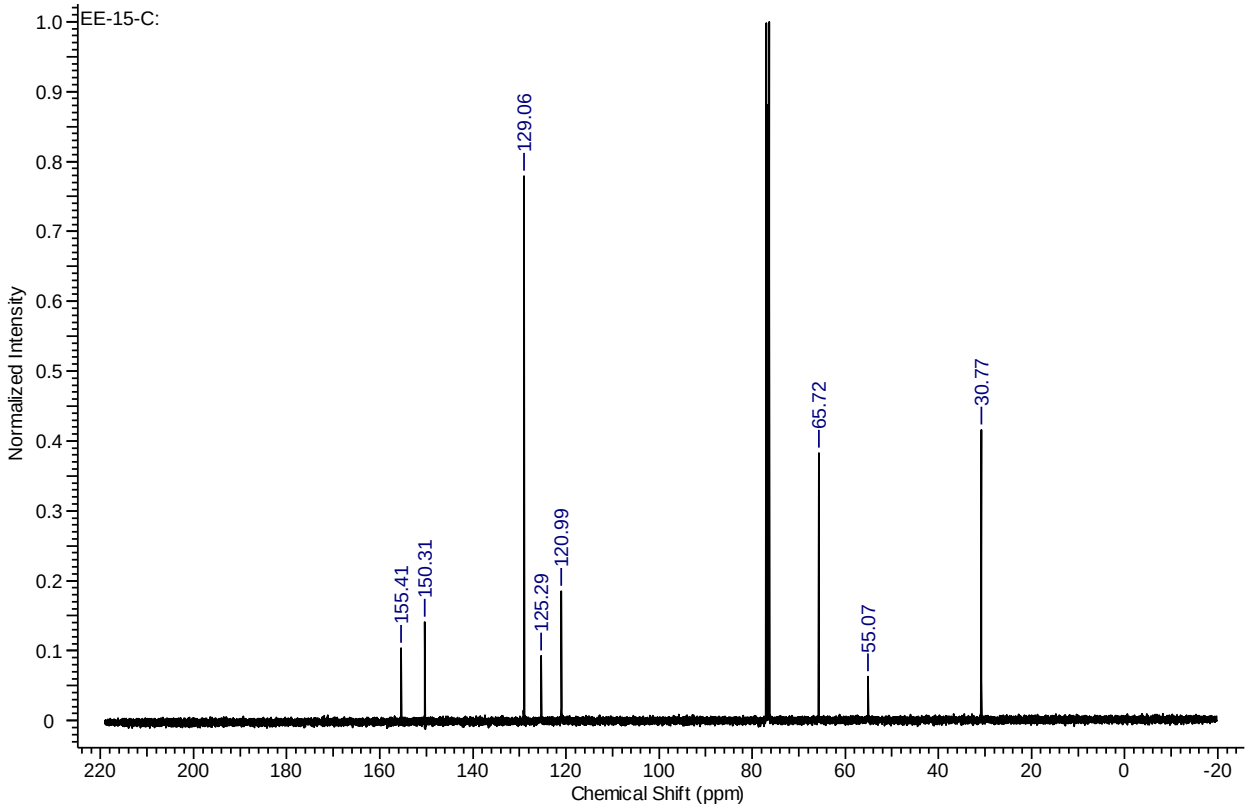

EE-4-H:
O
HN
OPh
NH
1j
Normalized Intensity
0.73
2.21
1.09
2.10
0.82
1.02
6.04
Chemical Shift (ppm)

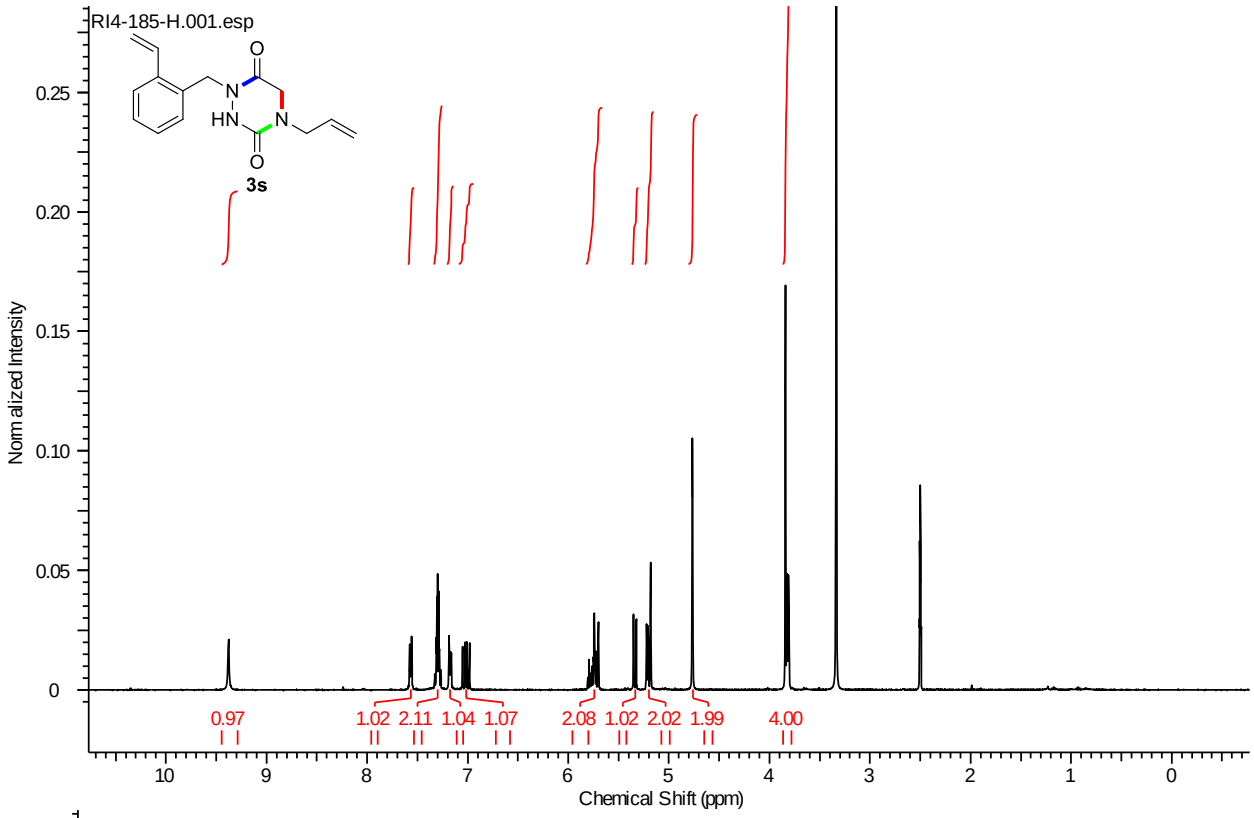

EE-4-C:
Normalized Intensity
129.29
124.91
121.57
115.20
49.56
20.69
Chemical Shift (ppm)

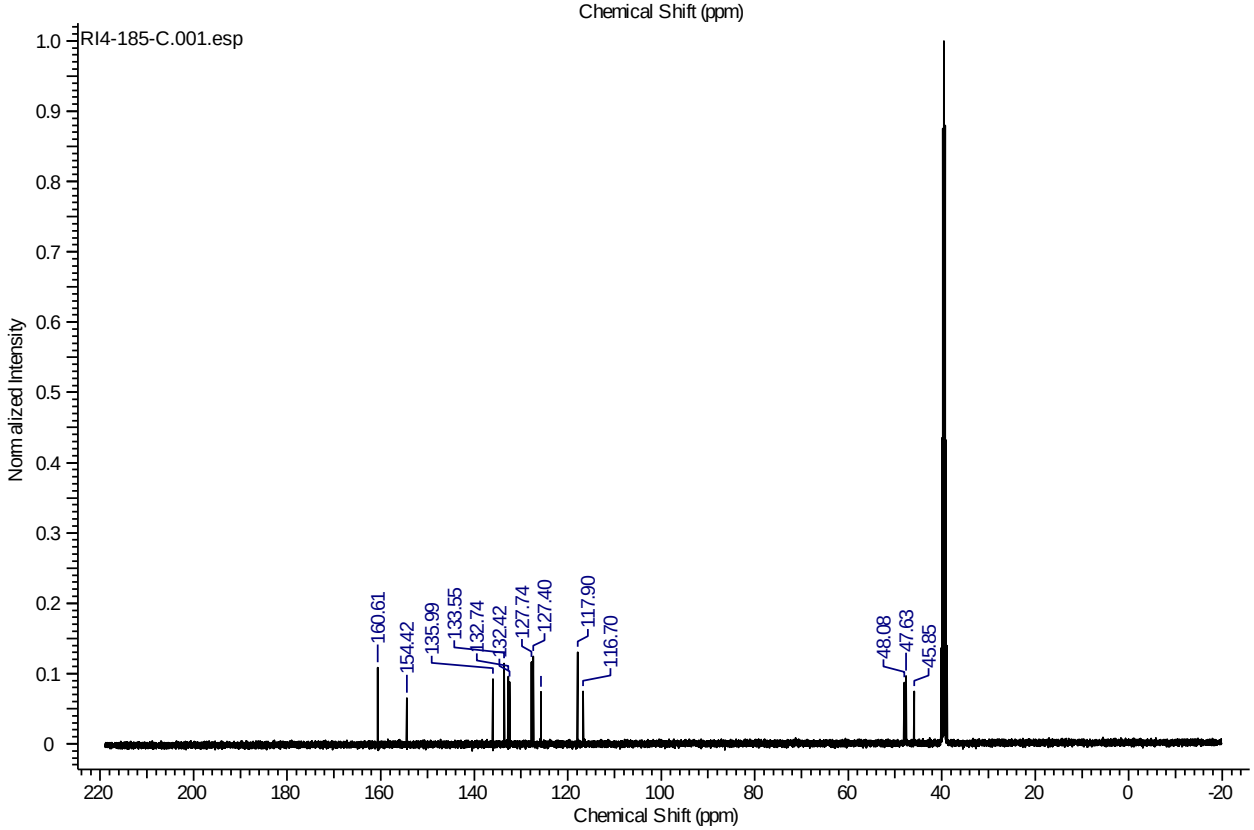

EE-24-H:
O
HN
OPh
NH
1l
Normalized Intensity
0.78
2.12
3.14
4.04
0.82
1.01
2.03
1.97
Chemical Shift (ppm)

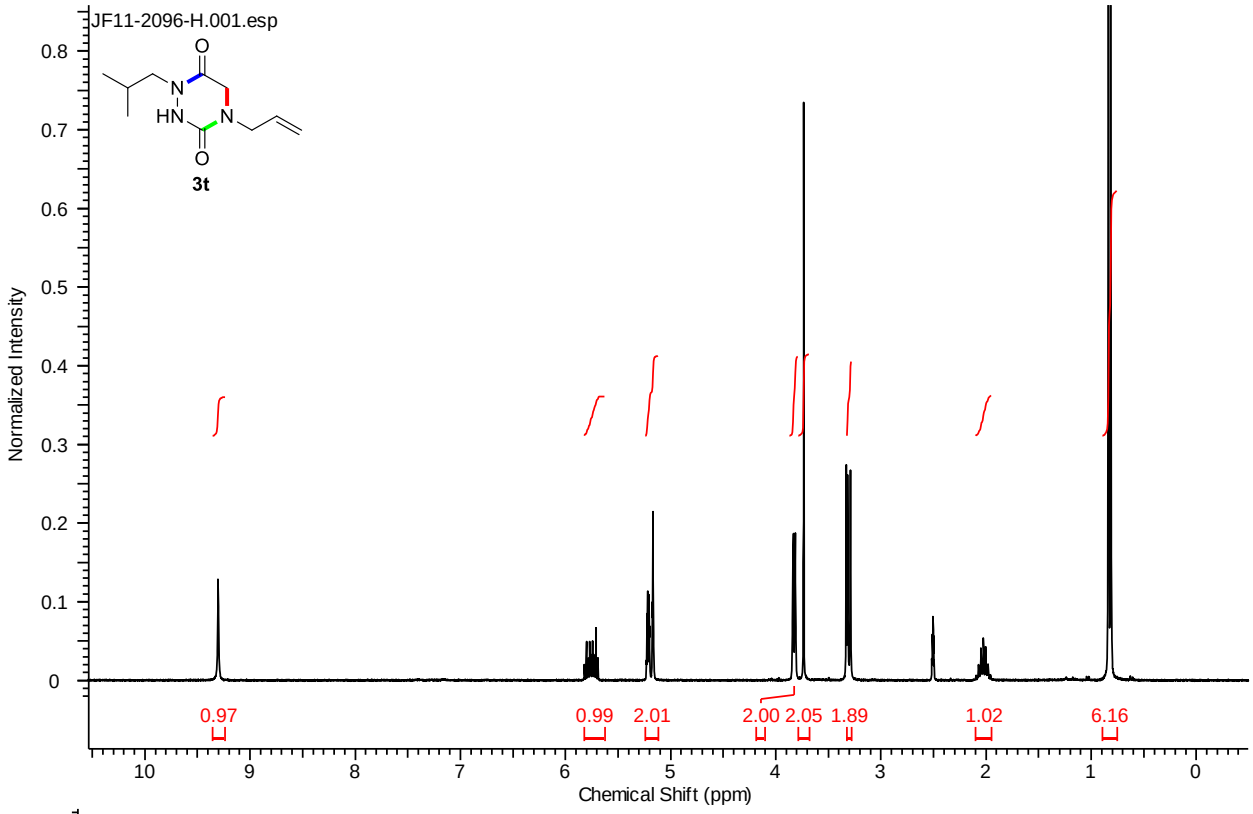

EE-24-C:
Normalized Intensity
141.82
129.33
126.09
124.55
121.61
155.25
150.94
58.93
37.45
Chemical Shift (ppm)

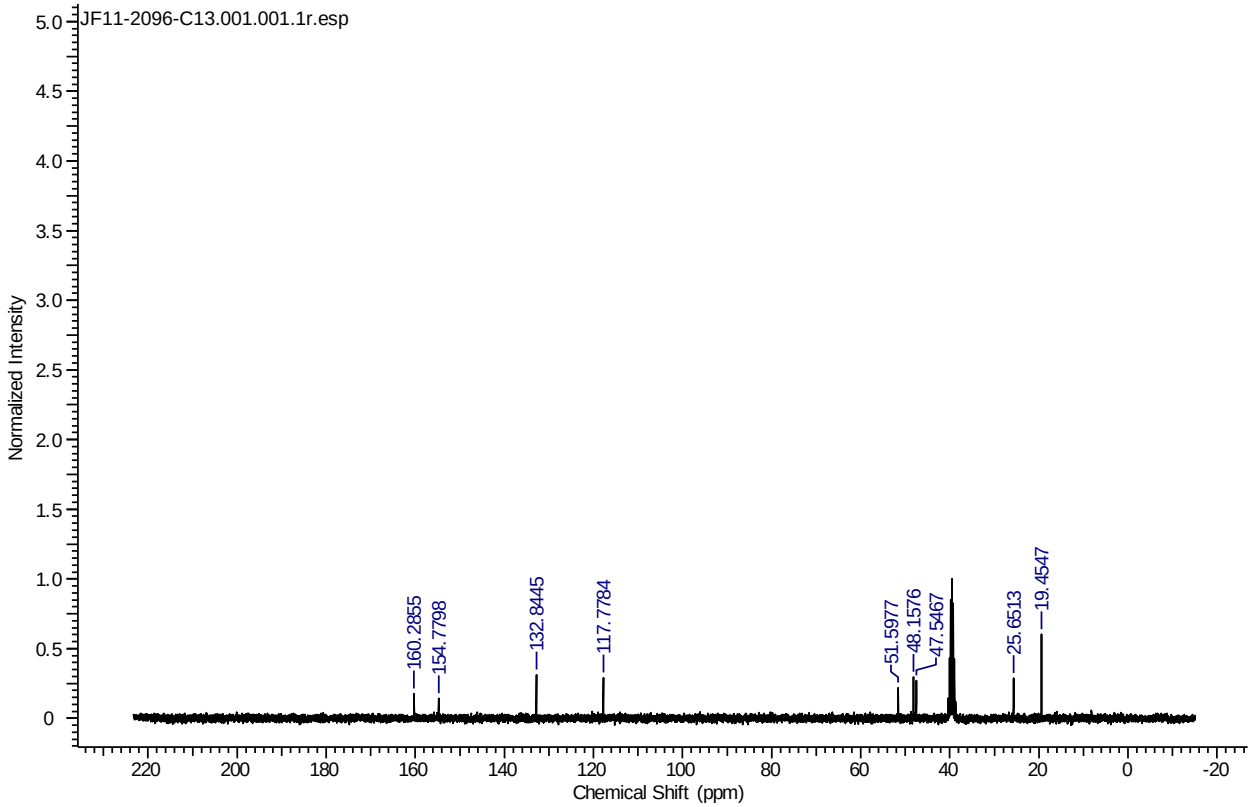

EE-15-H:
HN
O
OPh
NH
O
2m
Normalized Intensity
2.19 1.13 2.10
2.09 2.06 0.97
2.06 2.00
Chemical Shift (ppm)

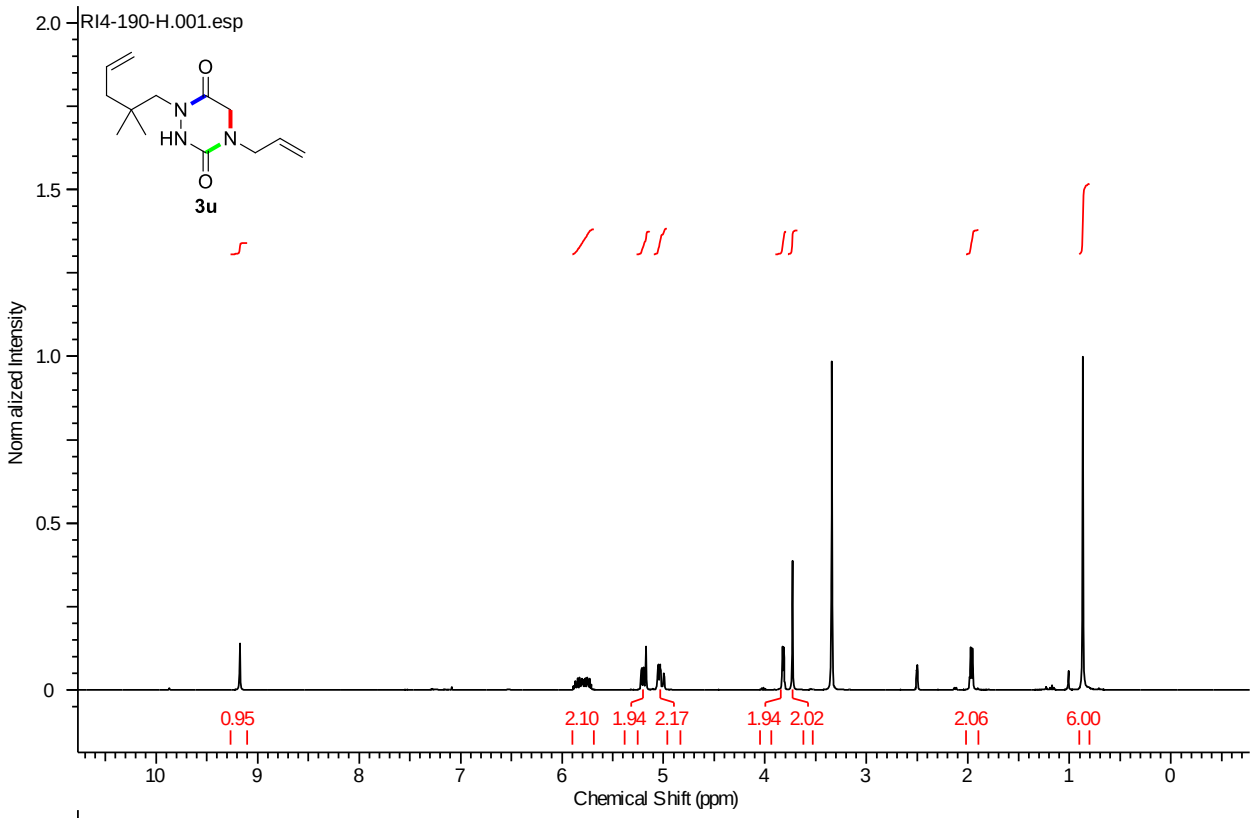

EE-15-C:
129.06
155.41
150.31
125.29
120.99
65.72
55.07
30.77
Normalized Intensity
Chemical Shift (ppm)

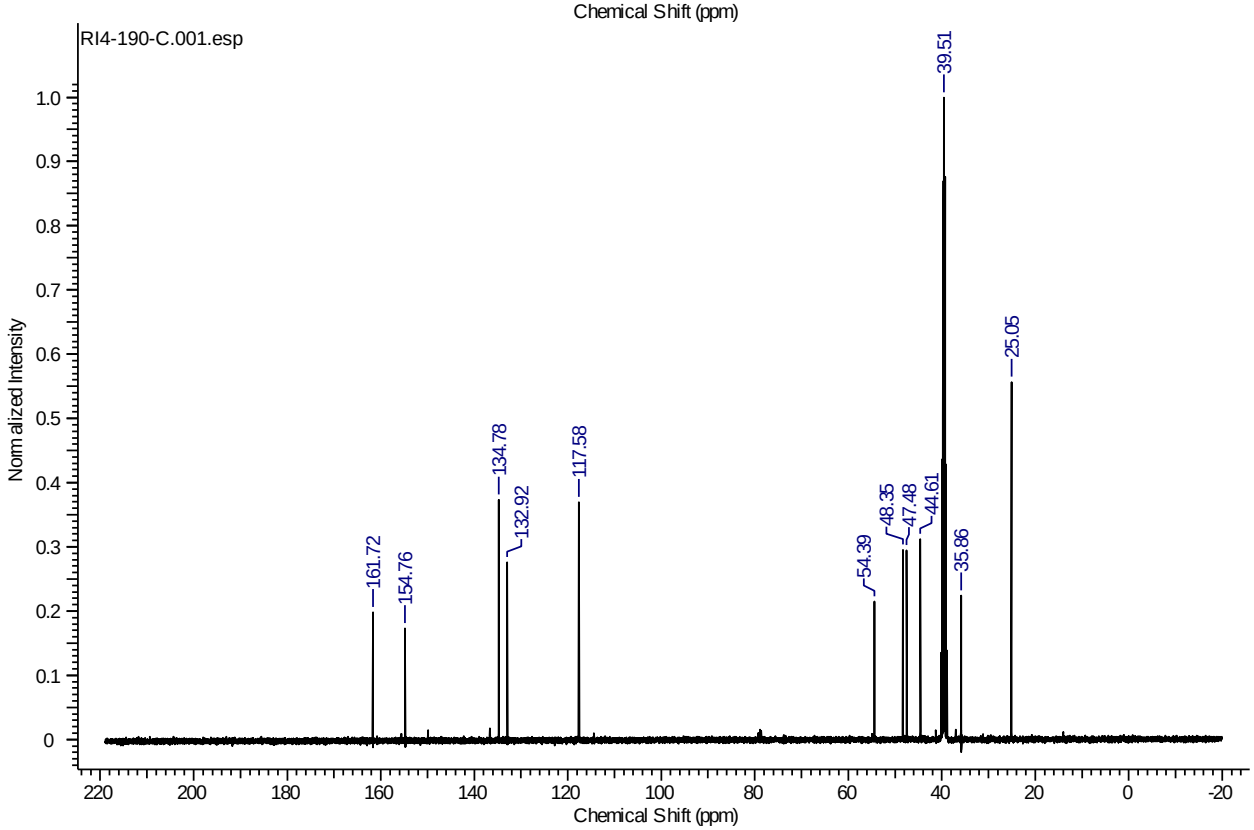

RI4-185-H.001.esp
O
N
HN
N
O
3s
Normalized Intensity
0.25
0.20
0.15
0.10
0.05
0
0.97
1.02
2.11
1.04
1.07
2.08
1.02
2.02
1.99
4.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

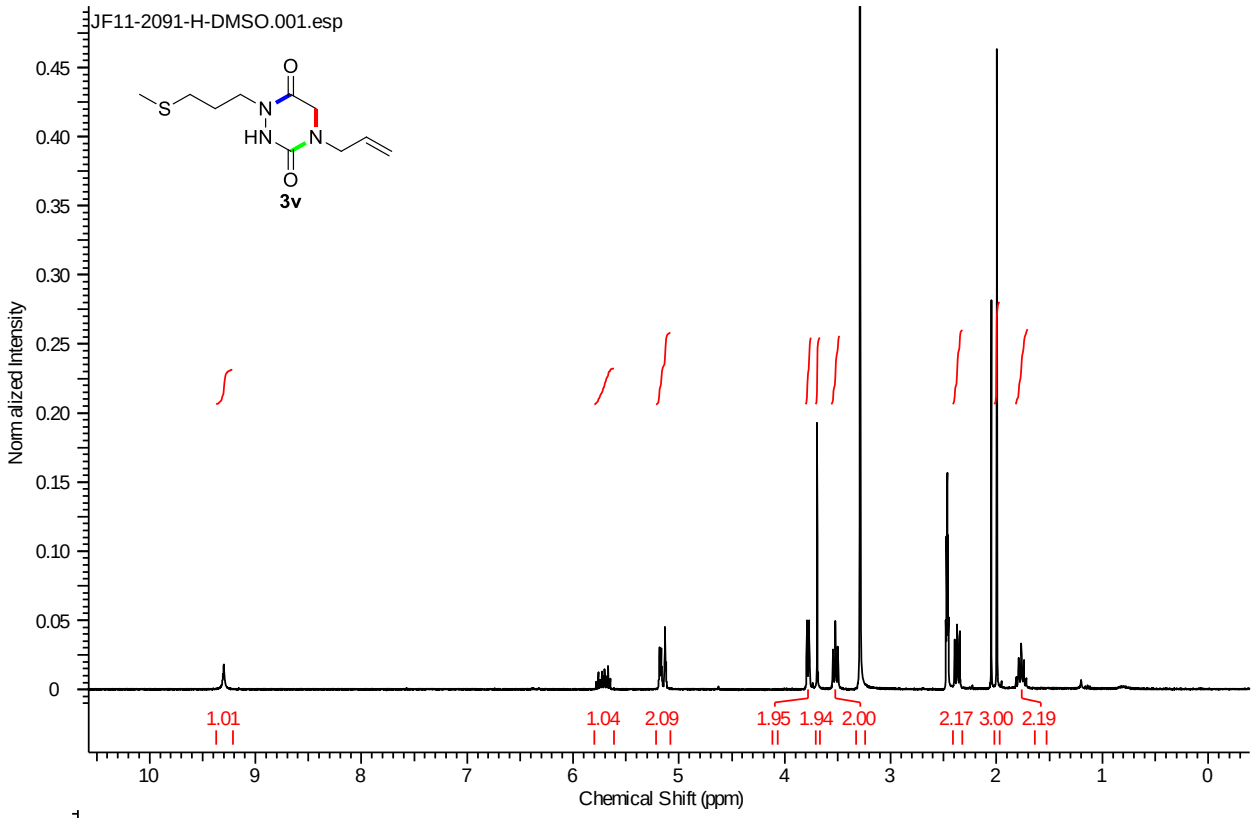

RI4-185-C.001.esp
Normalized Intensity
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
160.61
154.42
135.99
133.55
132.74
132.42
127.74
127.40
117.90
116.70
48.08
47.63
45.85
220
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

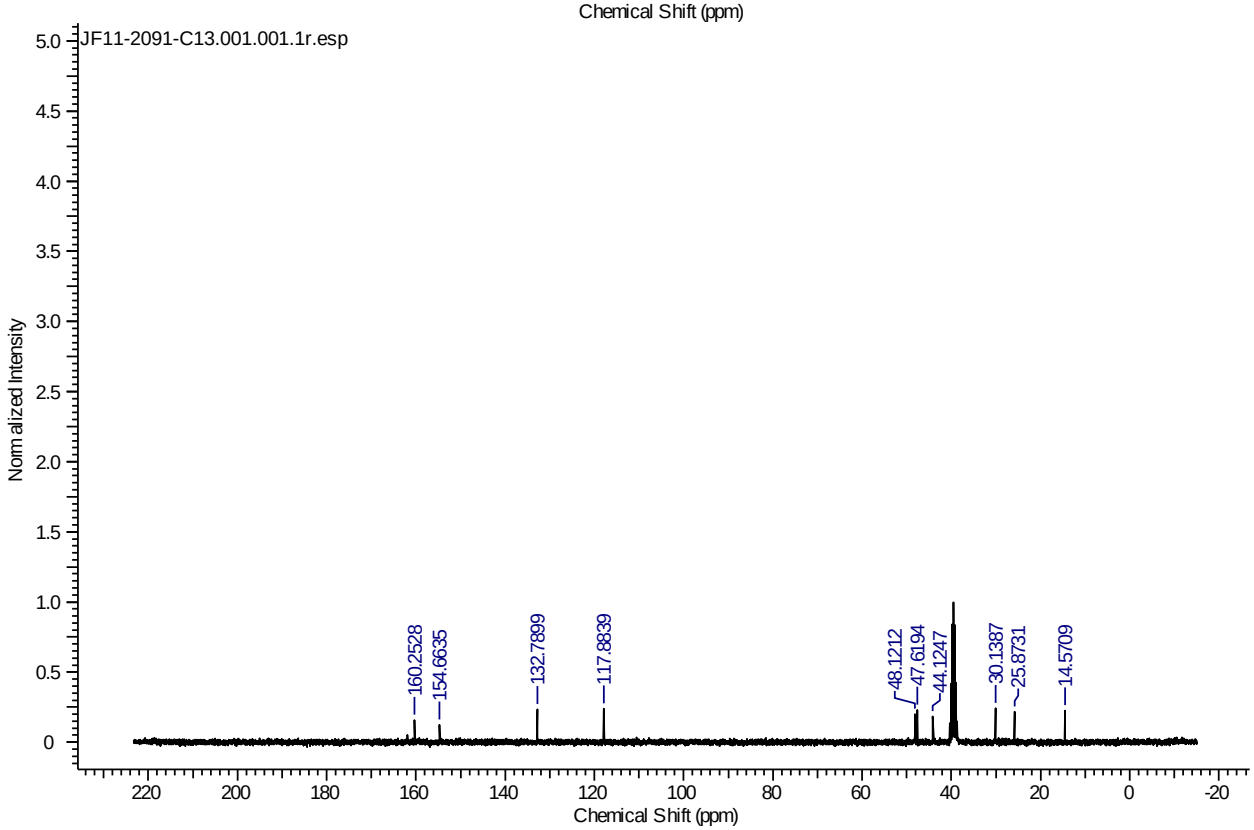

JF11-2096-H.001.esp
3t
Normalized Intensity
Chemical Shift (ppm)
0.97
0.99
2.01
2.00
2.05
1.89
1.02
6.16

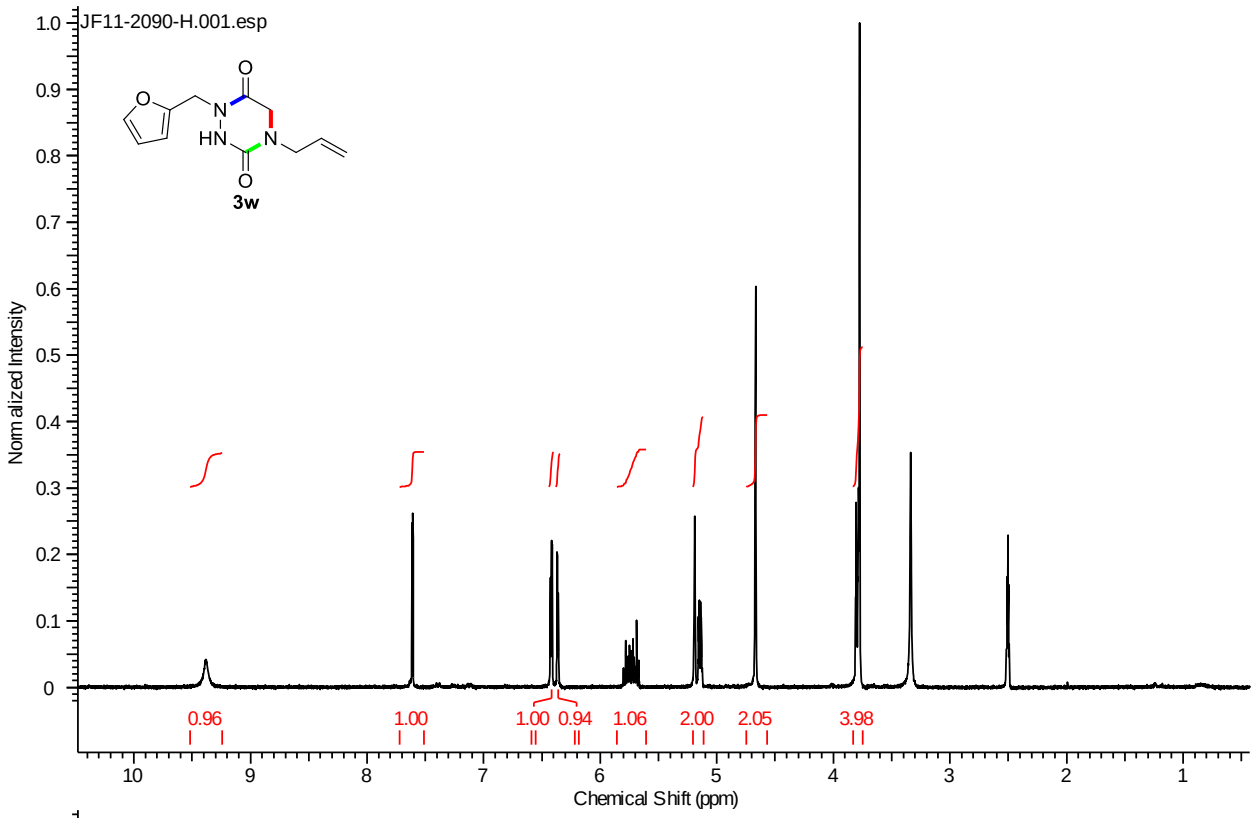

JF11-2096-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.2855
154.7798
132.8445
117.7784
51.5977
48.1576
47.5467
25.6513
19.4547

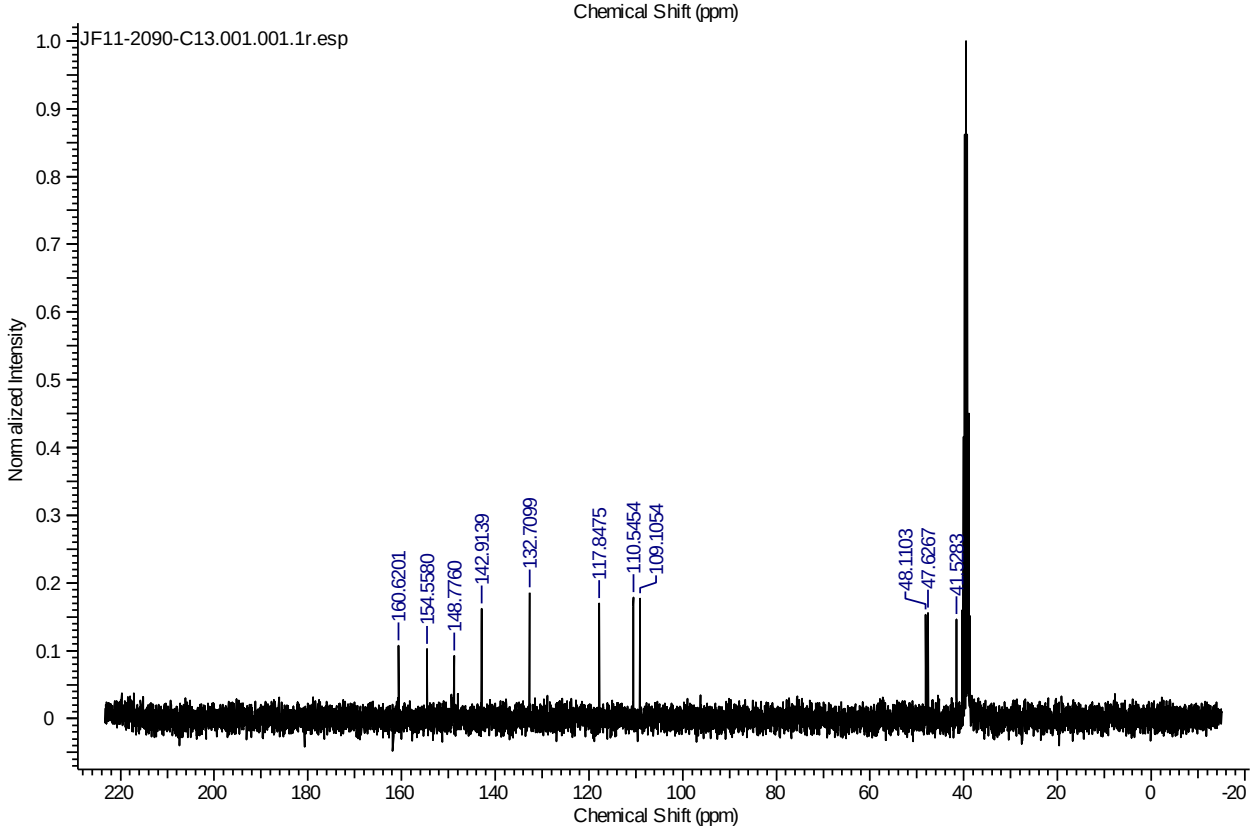

RI4-190-H.001.esp
3u
Normalized Intensity
Chemical Shift (ppm)
0.95
2.10
1.94
2.17
1.94
2.02
2.06
6.00

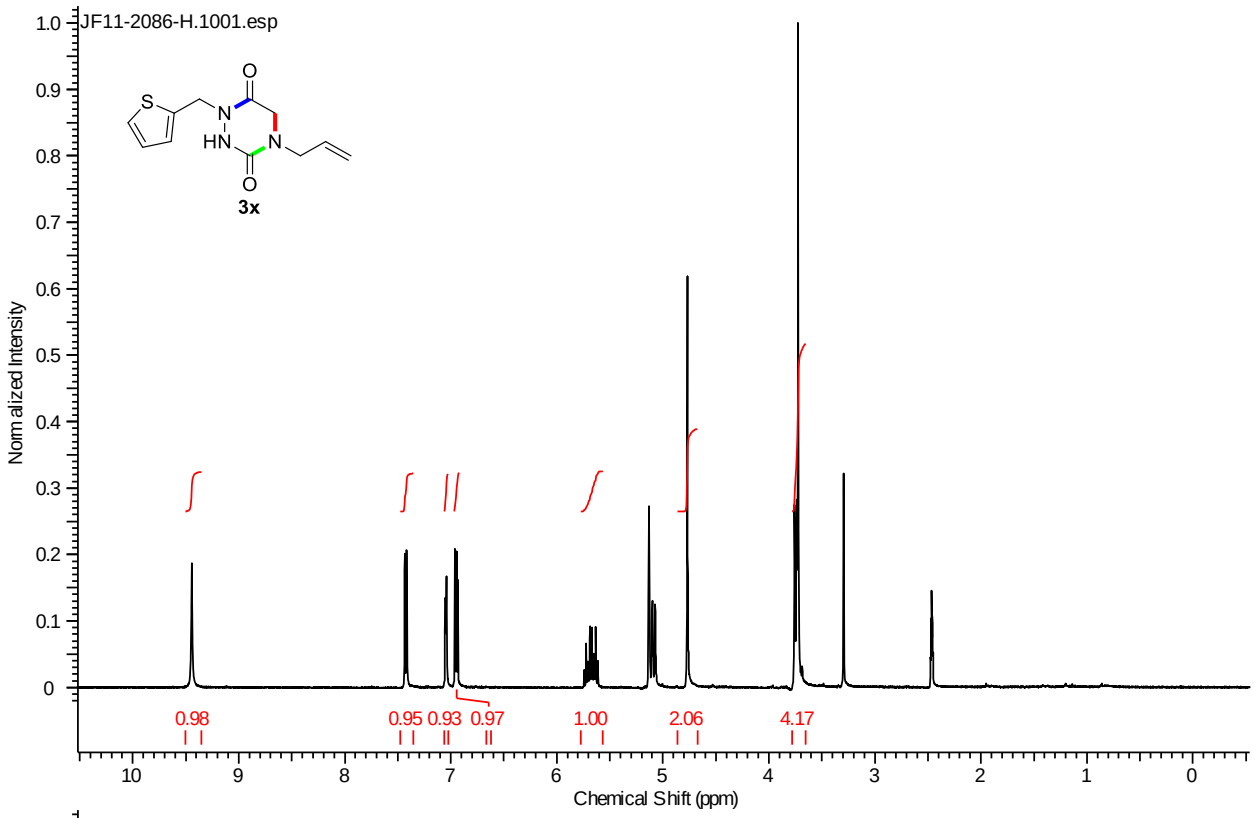

RI4-190-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
161.72
154.76
134.78
132.92
117.58
54.39
48.35
47.48
44.61
39.51
35.86
25.05

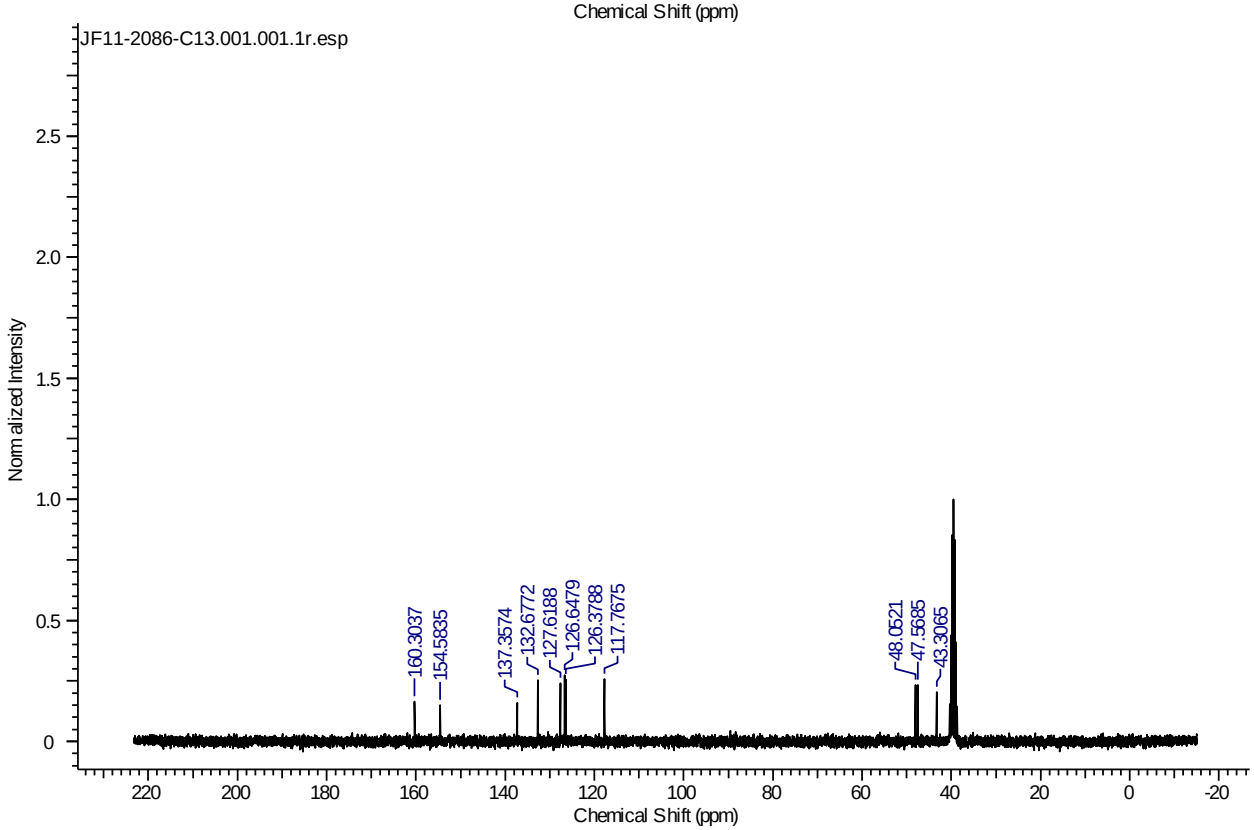

JF11-2091-H-DMSO.001.esp
3v
Normalized Intensity
Chemical Shift (ppm)
1.01
1.04
2.09
1.95
1.94
2.00
2.17
3.00
2.19

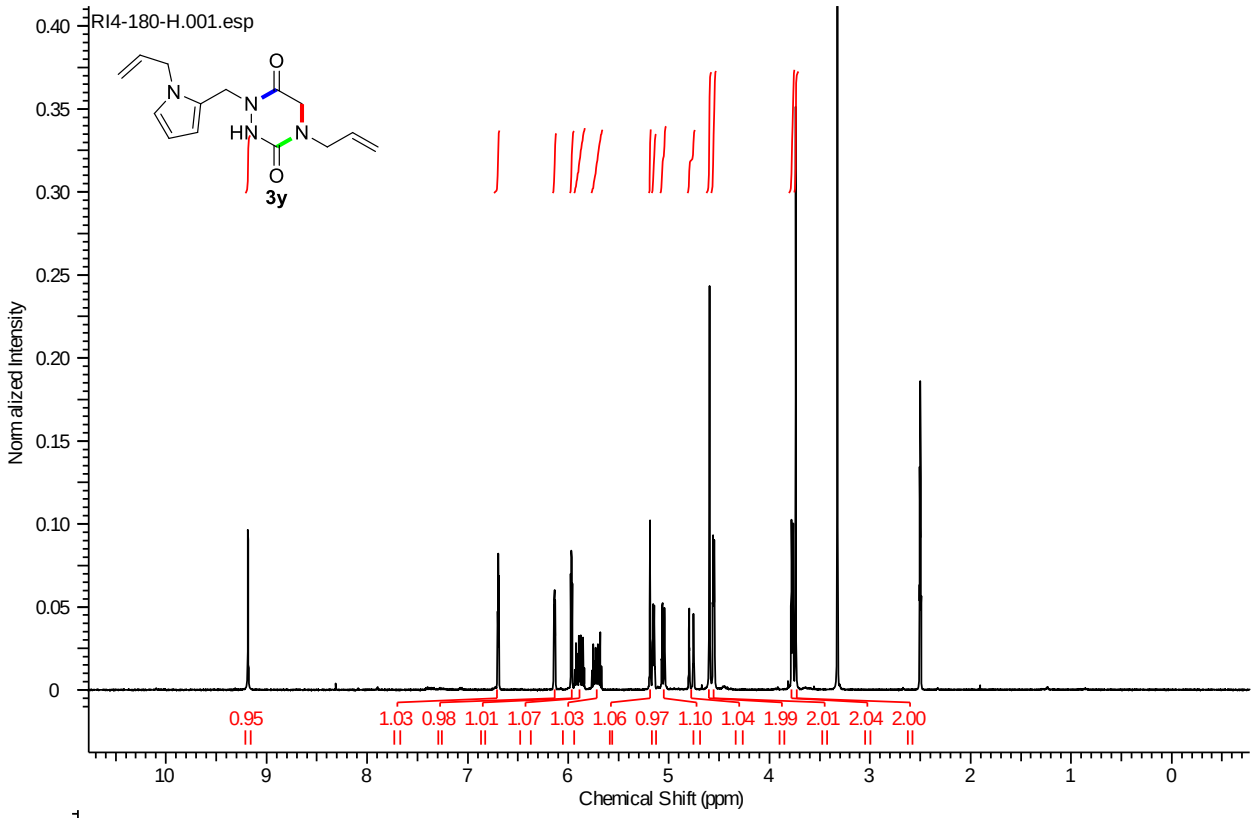

JF11-2091-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.2528
154.6635
132.7899
117.8839
48.1212
47.6194
44.1247
30.1387
25.8731
14.5709

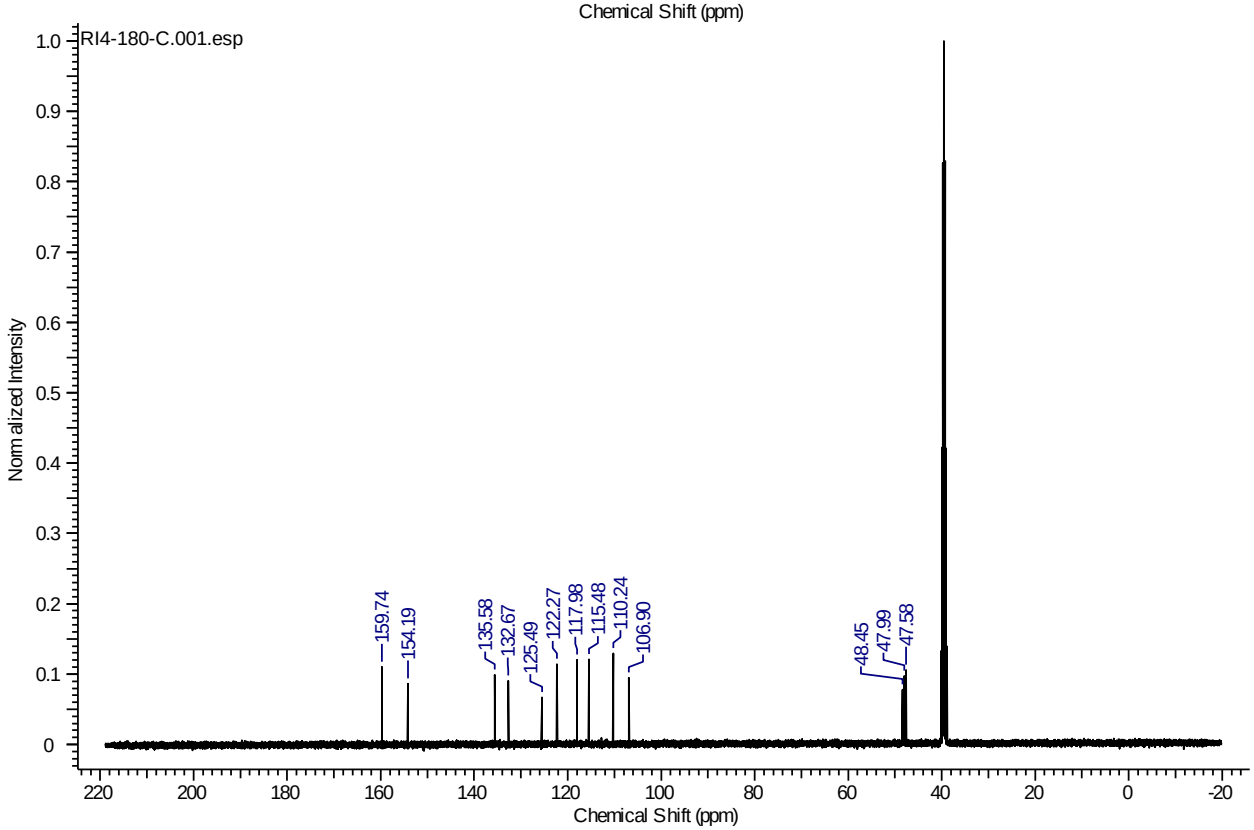

JF11-2090-H.001.esp
3w
Normalized Intensity
Chemical Shift (ppm)
0.96
1.00
1.00
0.94
1.06
2.00
2.05
3.98

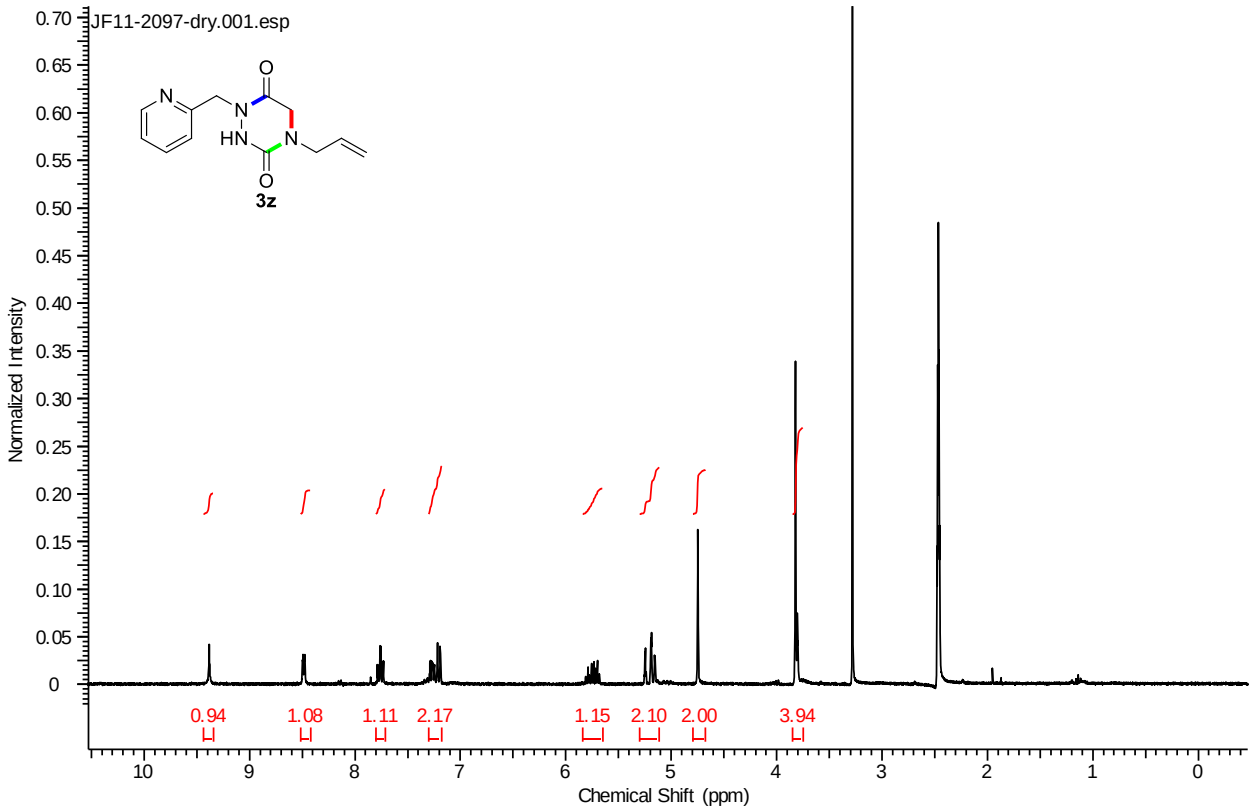

JF11-2090-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.6201
154.5580
148.7760
142.9139
132.7099
117.8475
110.5454
109.1054
48.1103
47.6267
41.5283

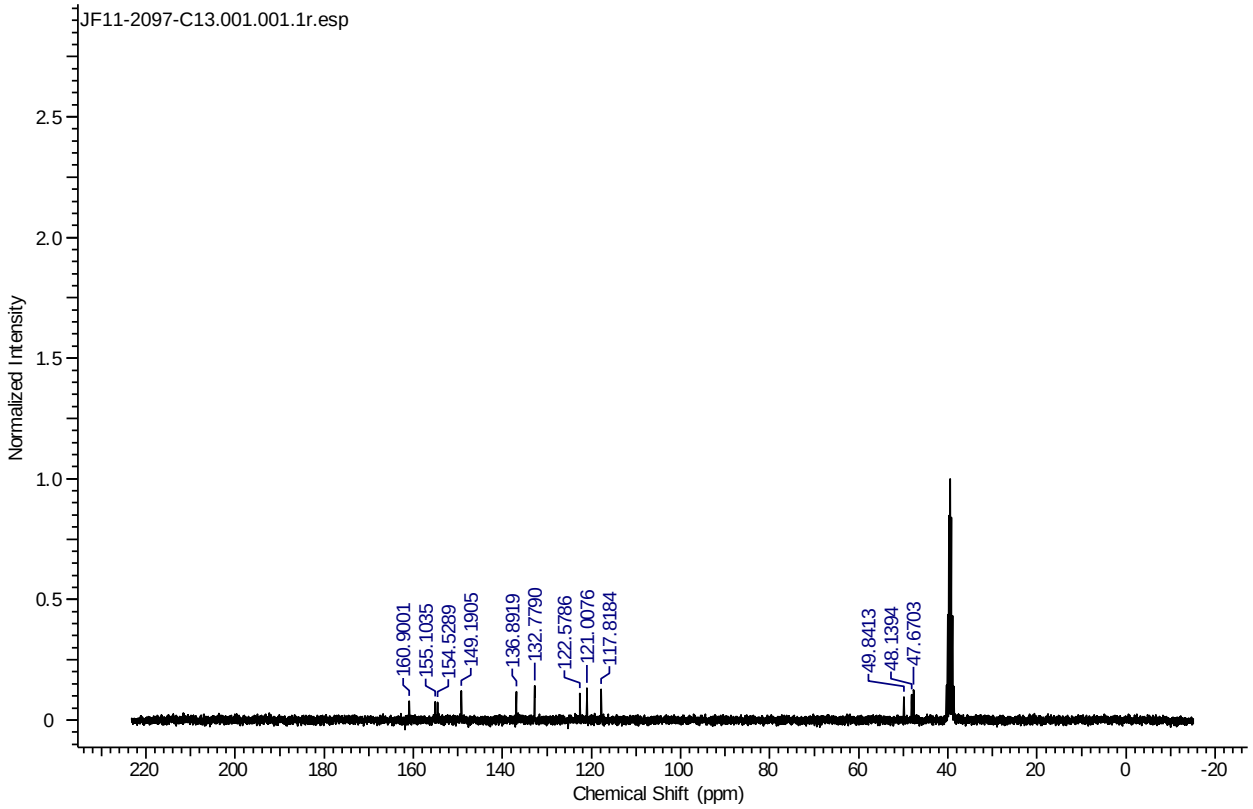

JF11-2086-H.1001.esp
3x
Normalized Intensity
Chemical Shift (ppm)
0.98
0.95
0.93
0.97
1.00
2.06
4.17

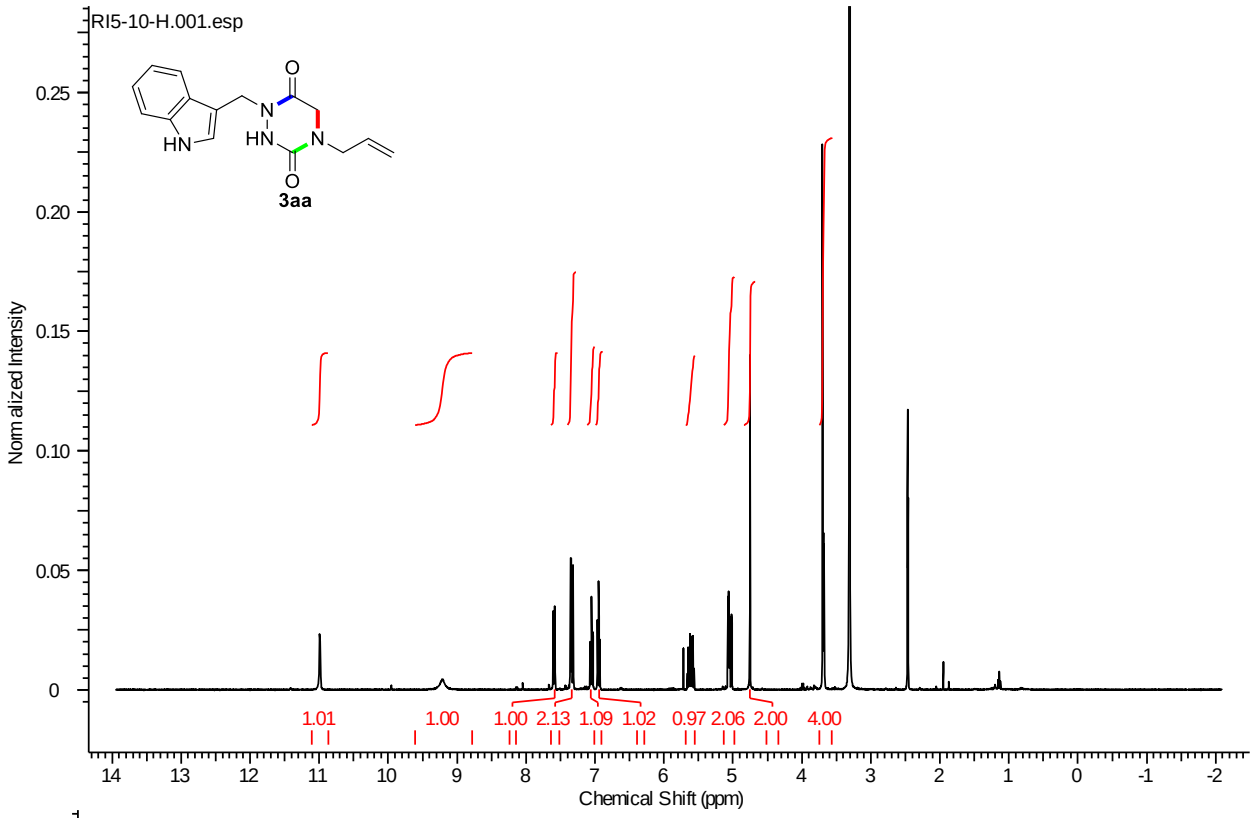

JF11-2086-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.3037
154.5835
137.3574
132.6772
127.6188
126.6479
126.3788
117.7675
48.0521
47.5685
43.3065

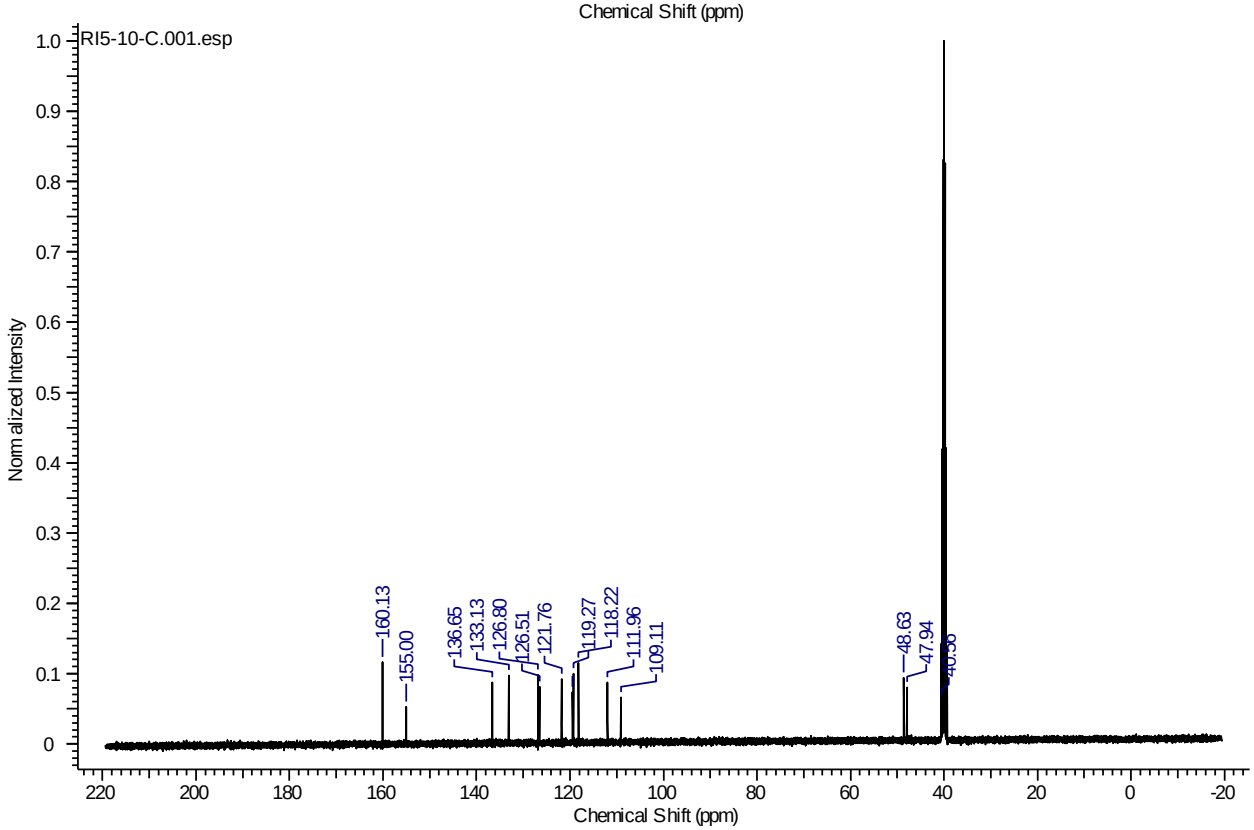

RI4-180-H.001.esp
3y
Normalized Intensity
Chemical Shift (ppm)
0.95
1.03 0.98 1.01 1.07 1.03 1.06 0.97 1.10 1.04 1.99 2.01 2.04 2.00

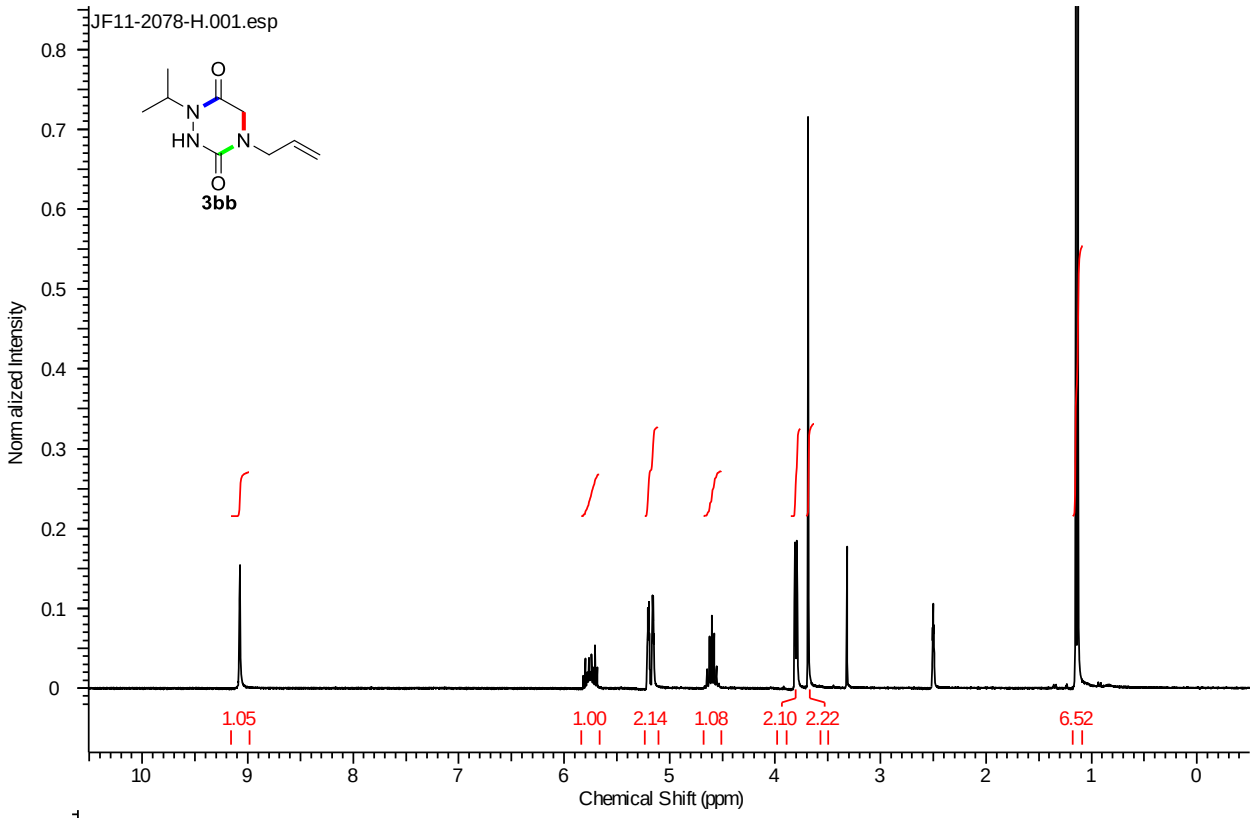

RI4-180-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
159.74
154.19
135.58
132.67
125.49
122.27
117.98
115.48
110.24
106.90
48.45
47.99
47.58

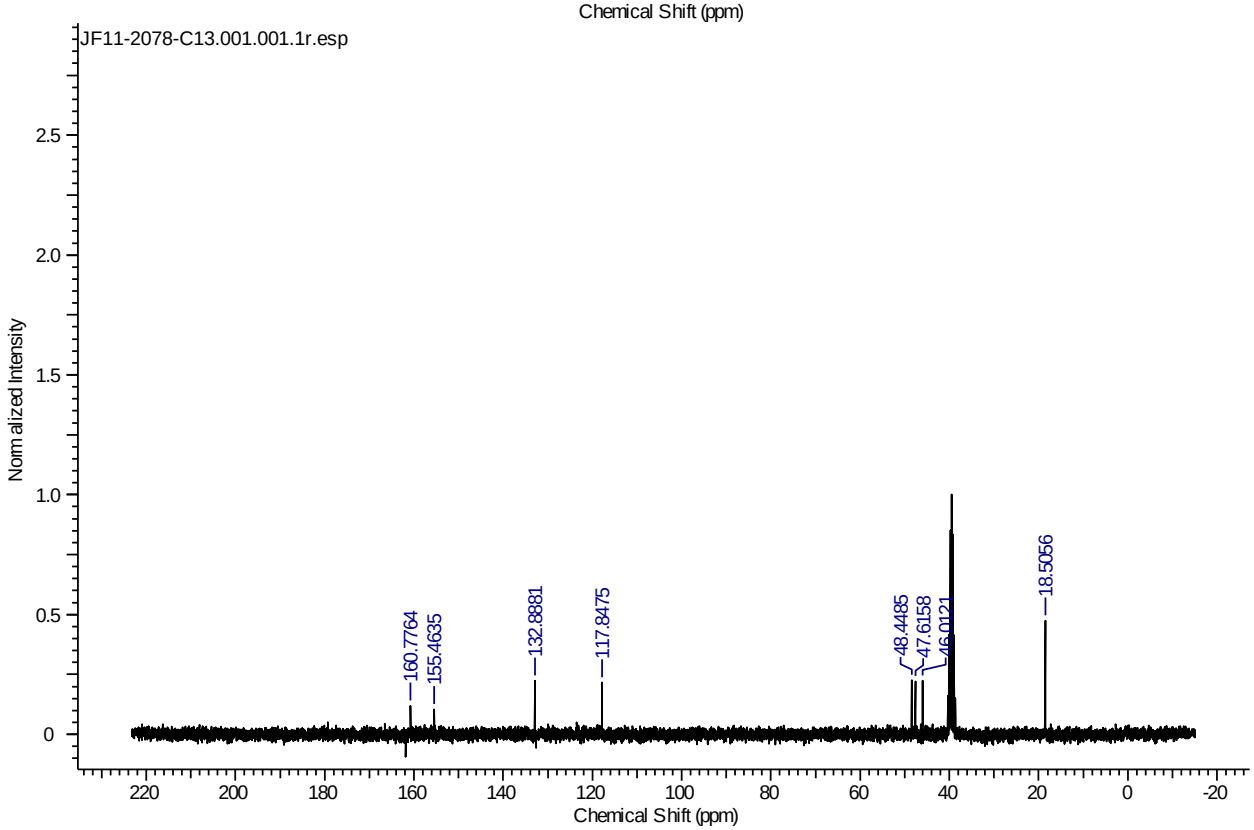

JF11-2097-dry.001.esp
3z
Normalized Intensity
Chemical Shift (ppm)
0.94
1.08
1.11
2.17
1.15
2.10
2.00
3.94

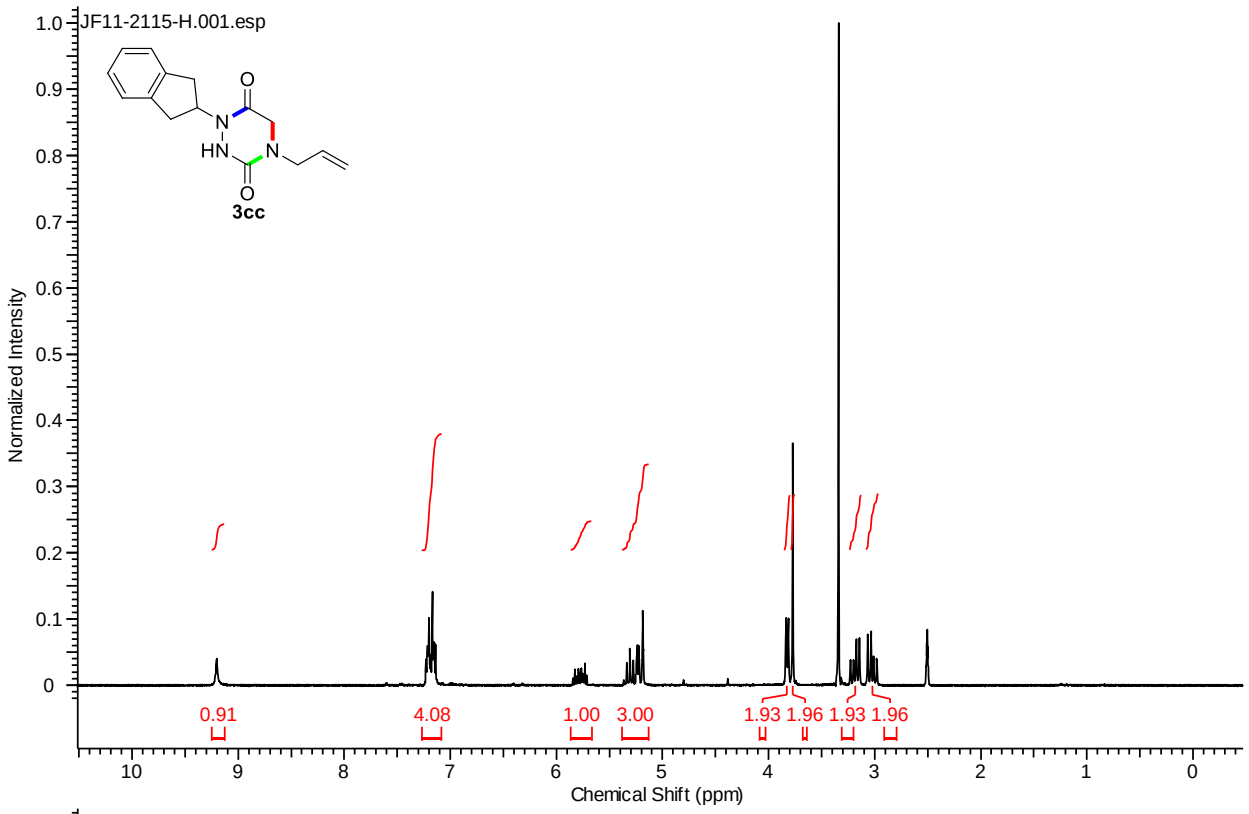

JF11-2097-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.9001
155.1035
154.5289
149.1905
136.8919
132.7790
122.5786
121.0076
117.8184
49.8413
48.1394
47.6703

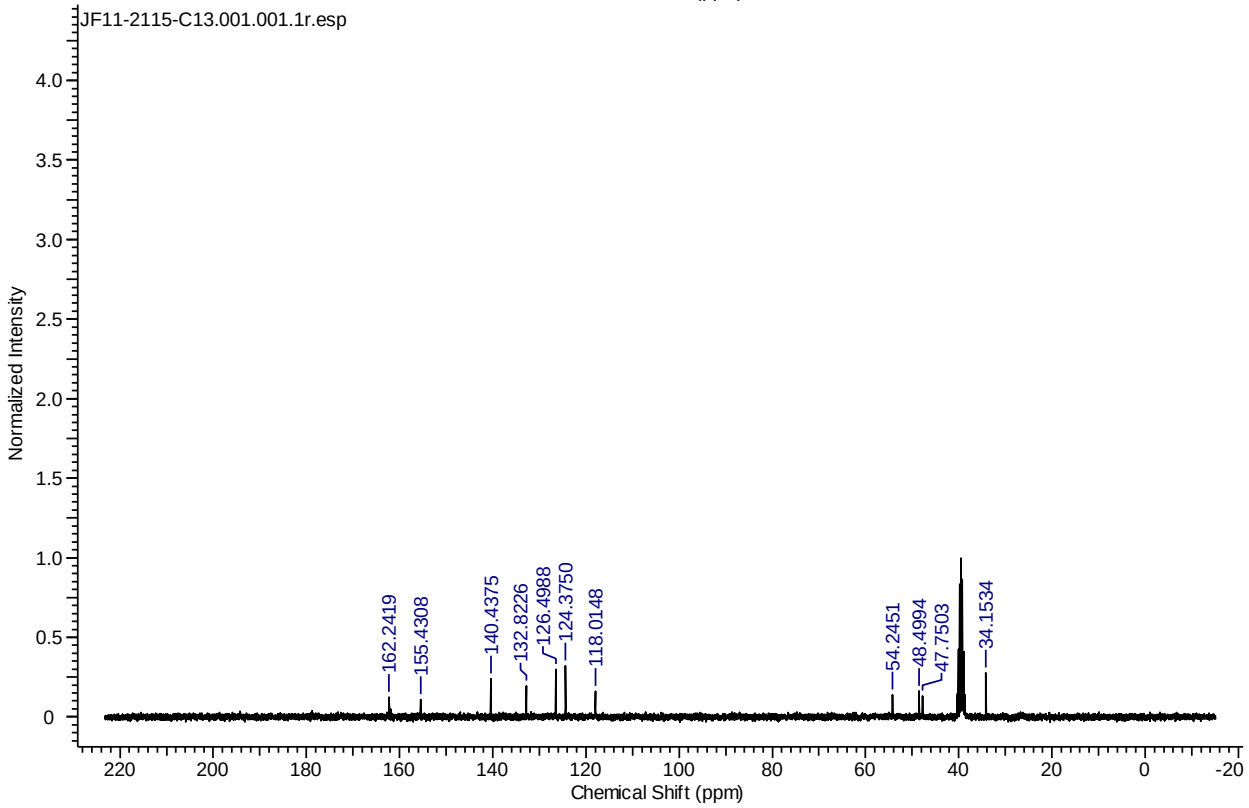

RI5-10-H.001.esp
3aa
Normalized Intensity
Chemical Shift (ppm)
1.01
1.00
1.00
2.13
1.09
1.02
0.97
2.06
2.00
4.00

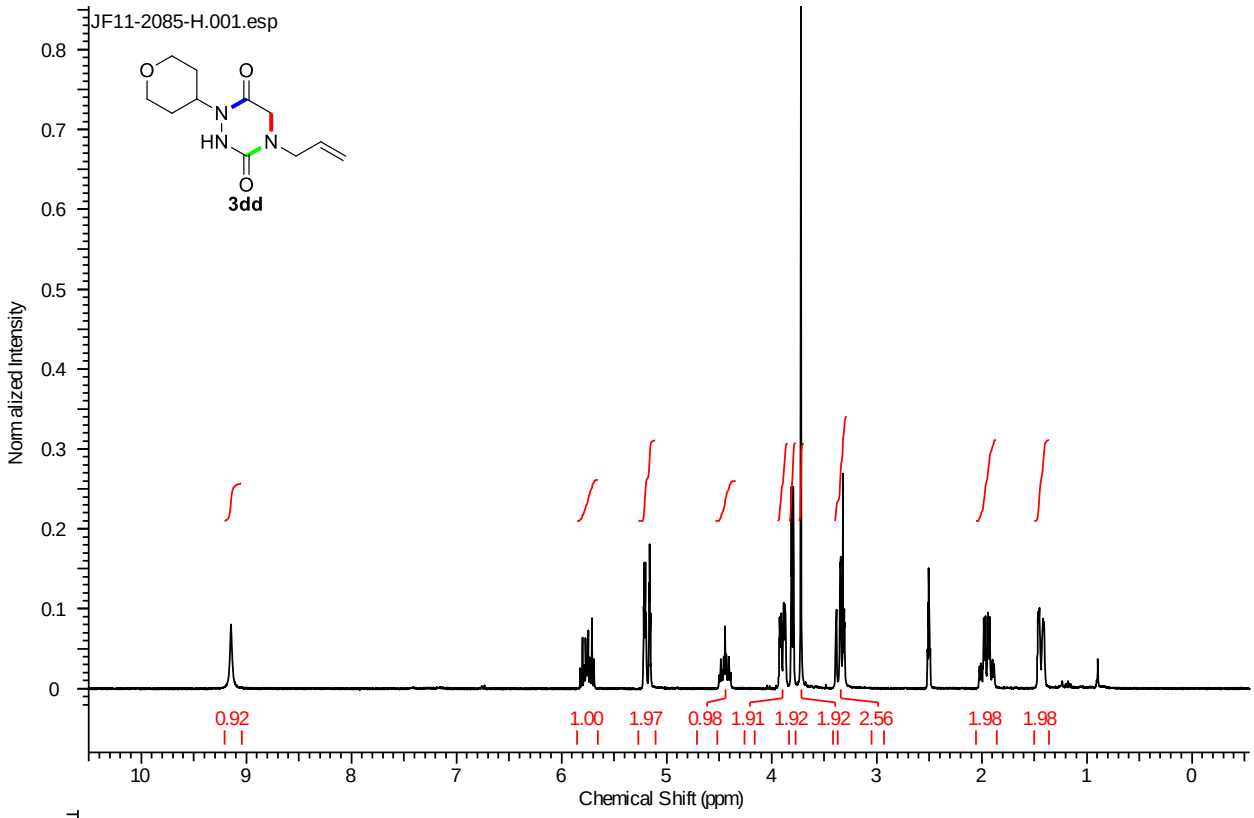

RI5-10-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
160.13
155.00
136.65
133.13
126.80
126.51
121.76
119.27
118.22
111.96
109.11
48.63
47.94
40.58

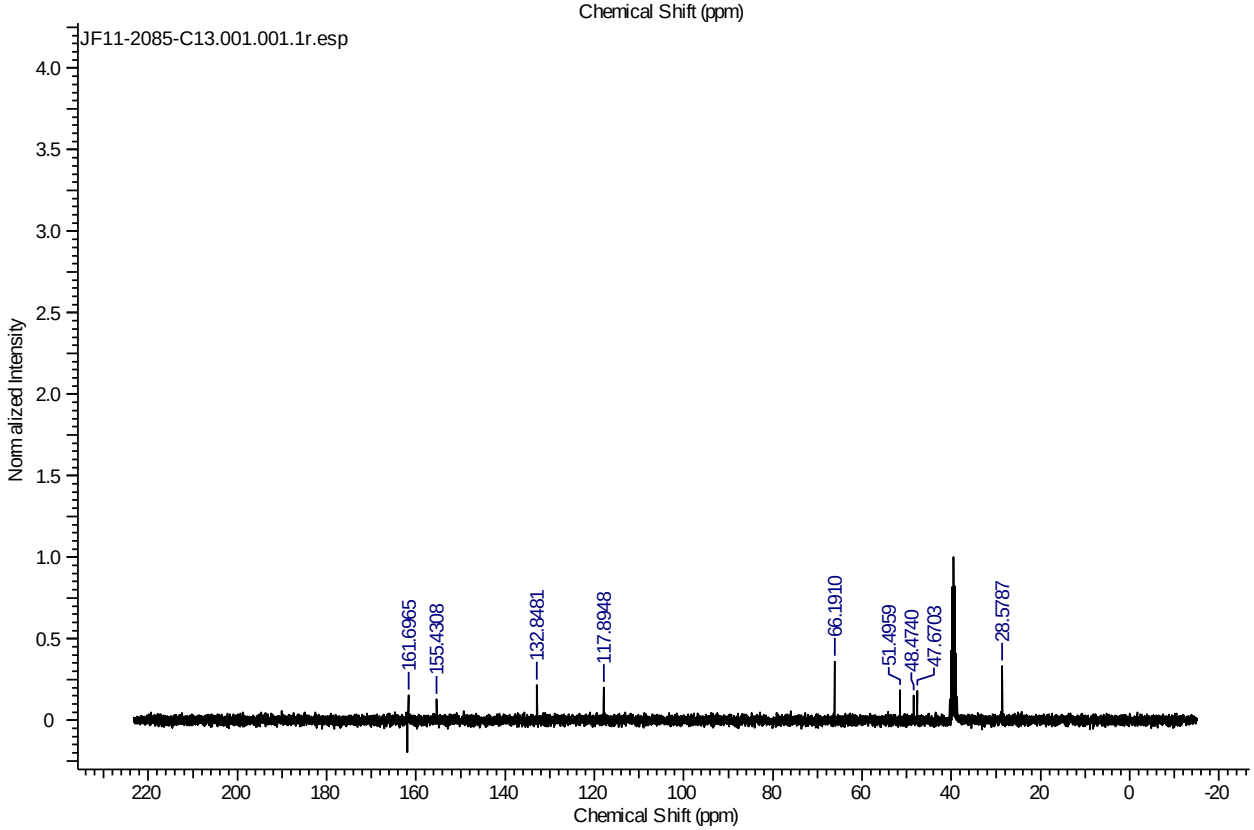

JF11-2078-H.001.esp
3bb
Normalized Intensity
Chemical Shift (ppm)
1.05
1.00
2.14
1.08
2.10
2.22
6.52

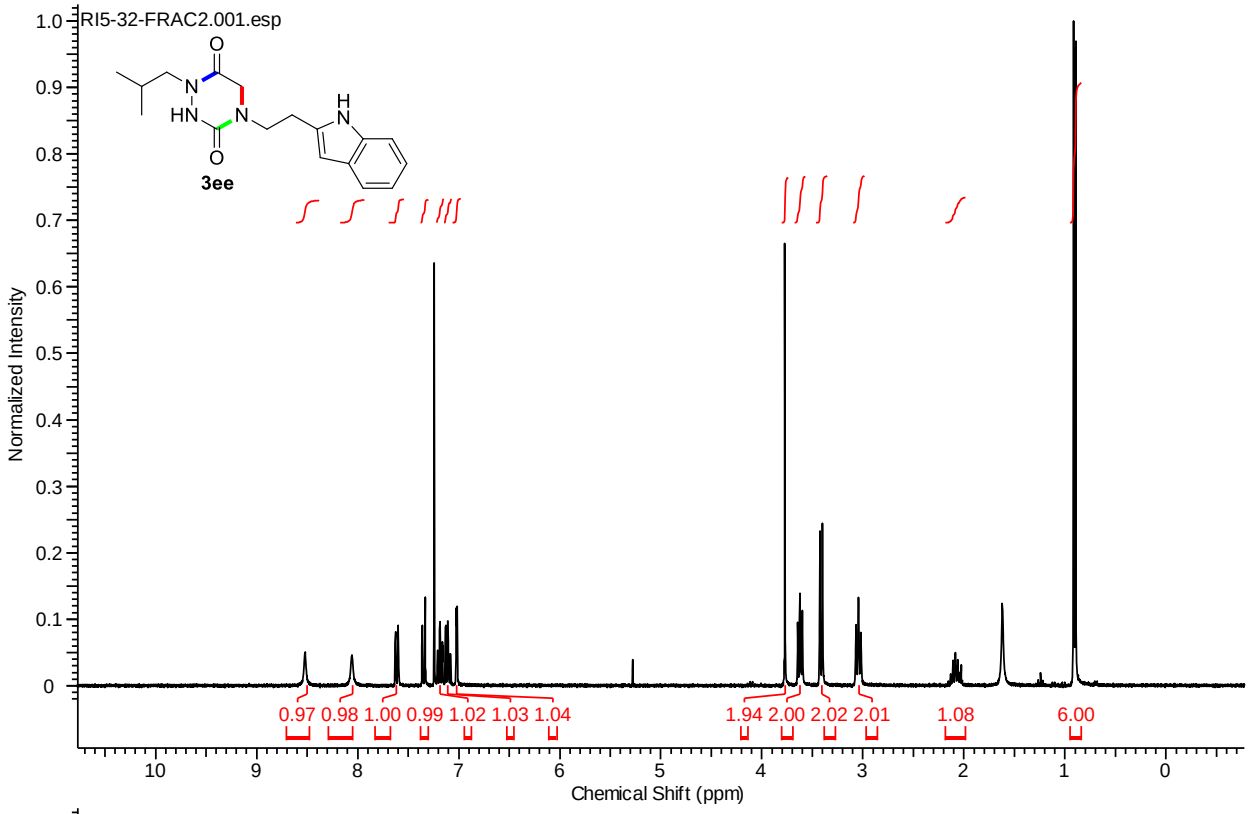

JF11-2078-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.7764
155.4635
132.8881
117.8475
48.4485
47.6158
46.0121
18.5056

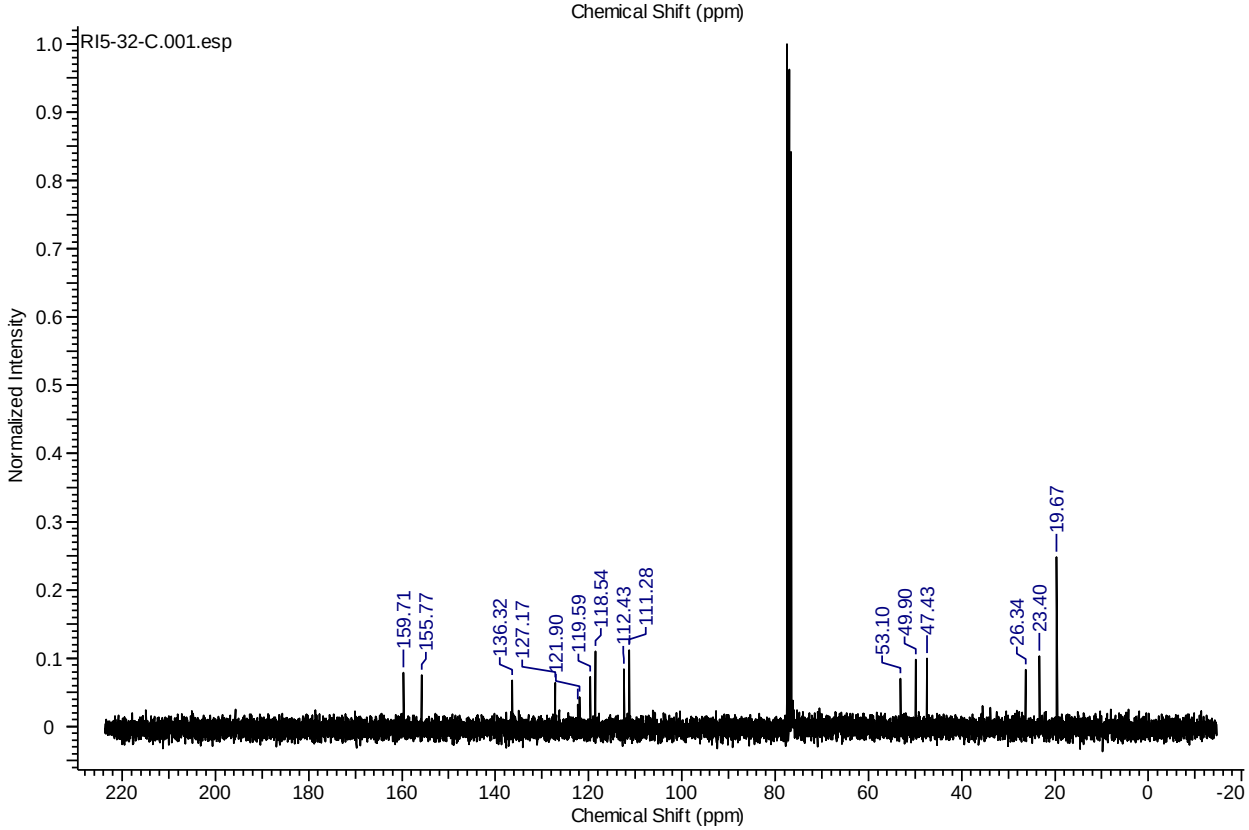

JF11-2115-H.001.esp
3cc
Normalized Intensity
Chemical Shift (ppm)
0.91
4.08
1.00
3.00
1.93
1.96
1.93
1.96

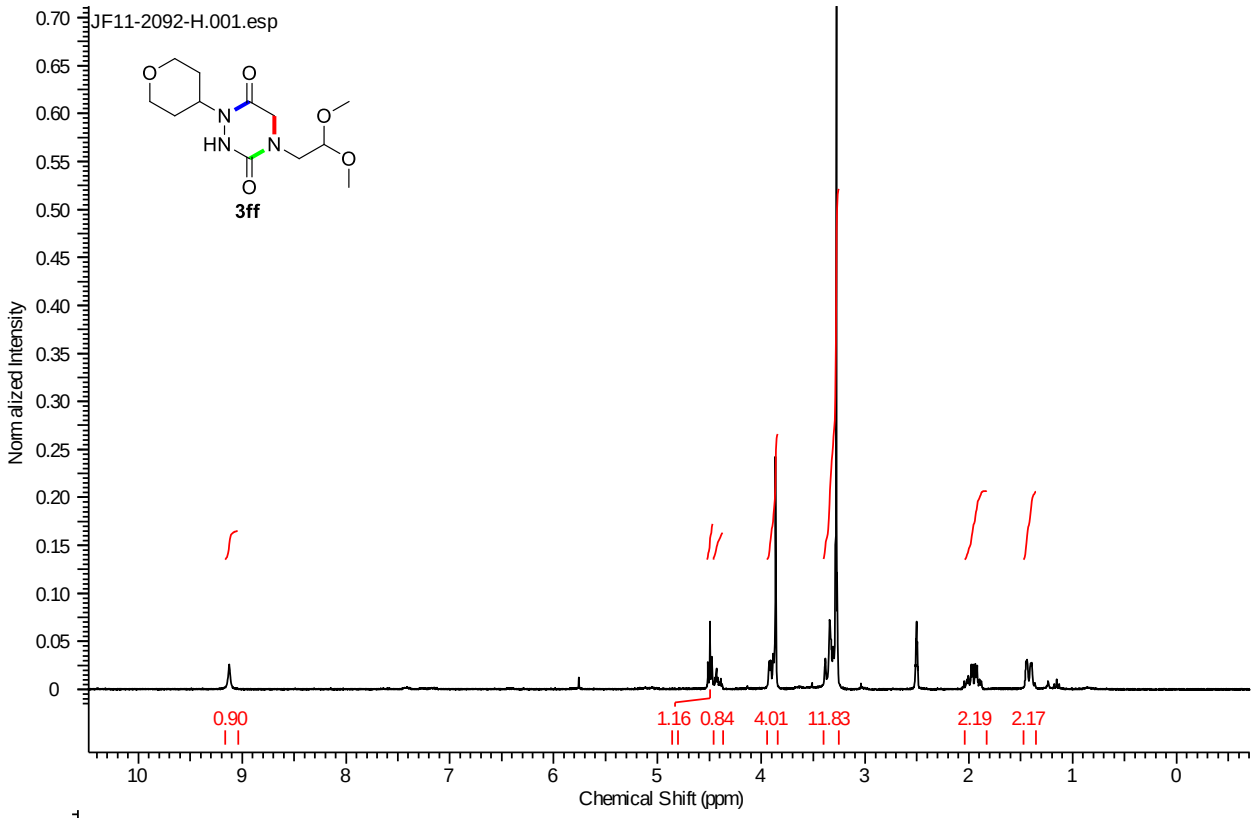

JF11-2115-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
162.2419
155.4308
140.4375
132.8226
126.4988
124.3750
118.0148
54.2451
48.4994
47.7503
34.1534

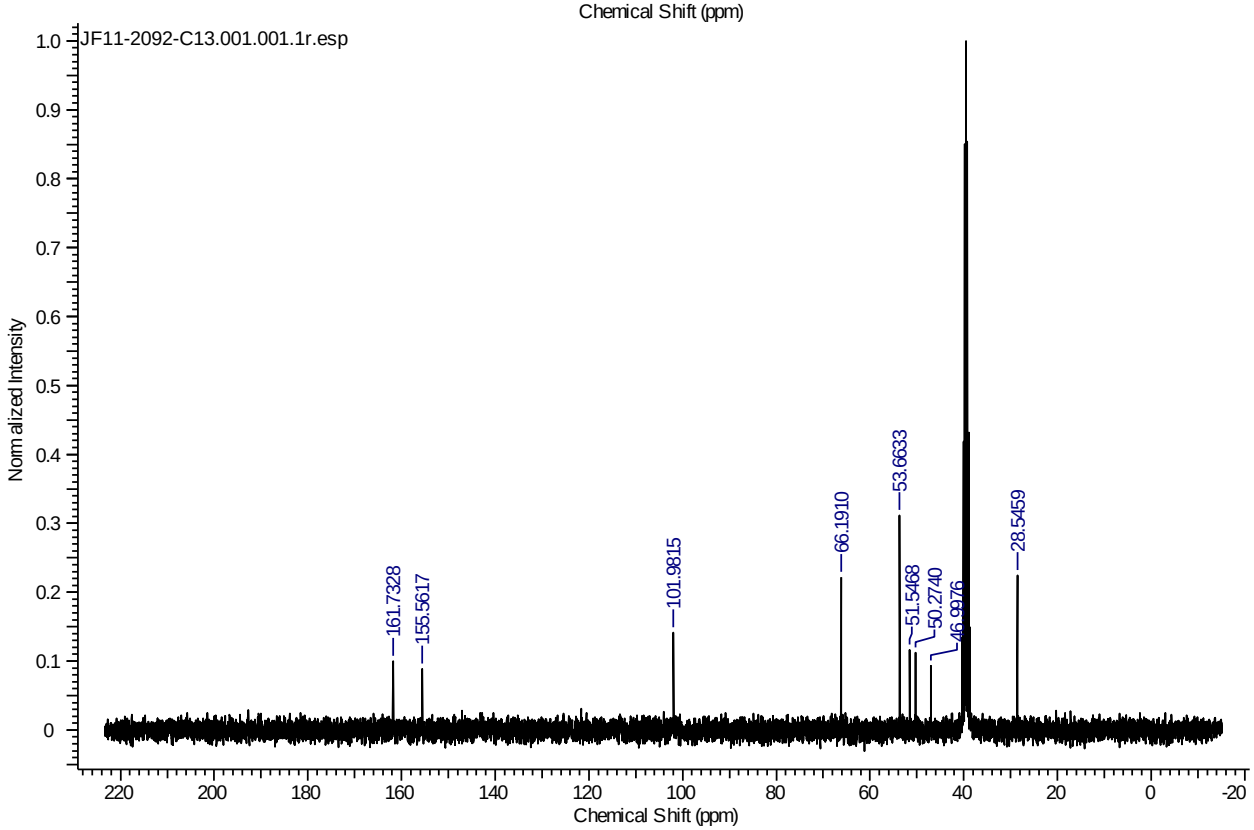

JF11-2085-H.001.esp
3dd
Normalized Intensity
Chemical Shift (ppm)
0.92
1.00
1.97
0.98
1.91
1.92
1.92
2.56
1.98
1.98

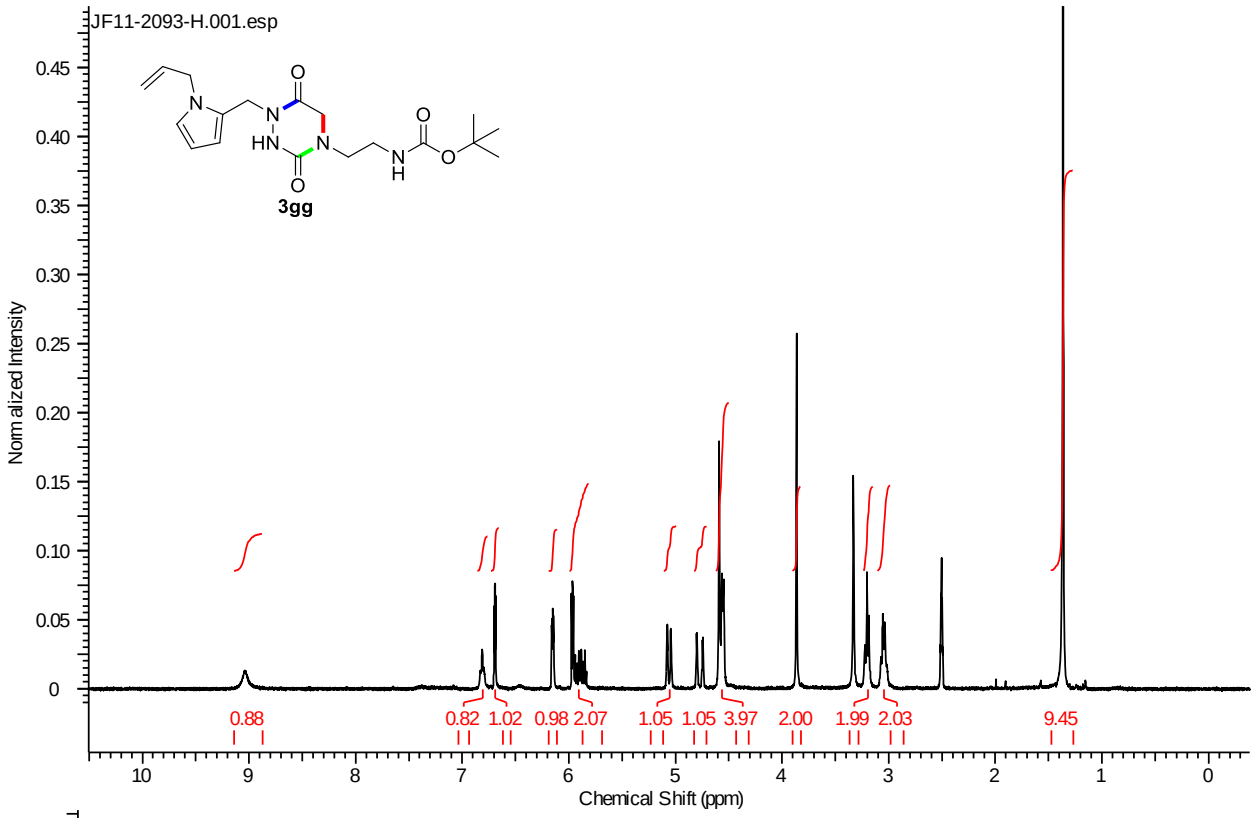

JF11-2085-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
161.6965
155.4308
132.8481
117.8948
66.1910
51.4959
48.4740
47.6703
28.5787

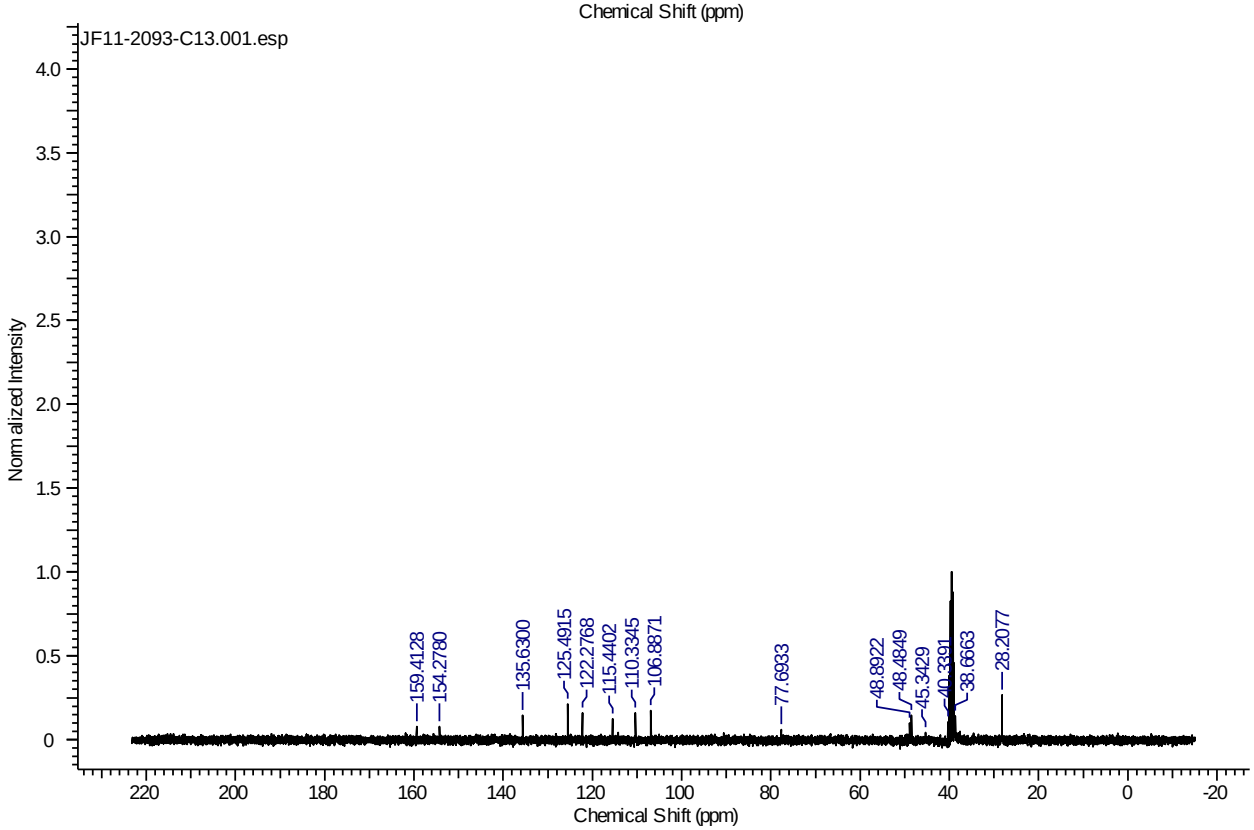

RI5-32-FRAC2.001.esp
3ee
Normalized Intensity
Chemical Shift (ppm)
0.97 0.98 1.00 0.99 1.02 1.03 1.04
1.94 2.00 2.02 2.01
1.08
6.00

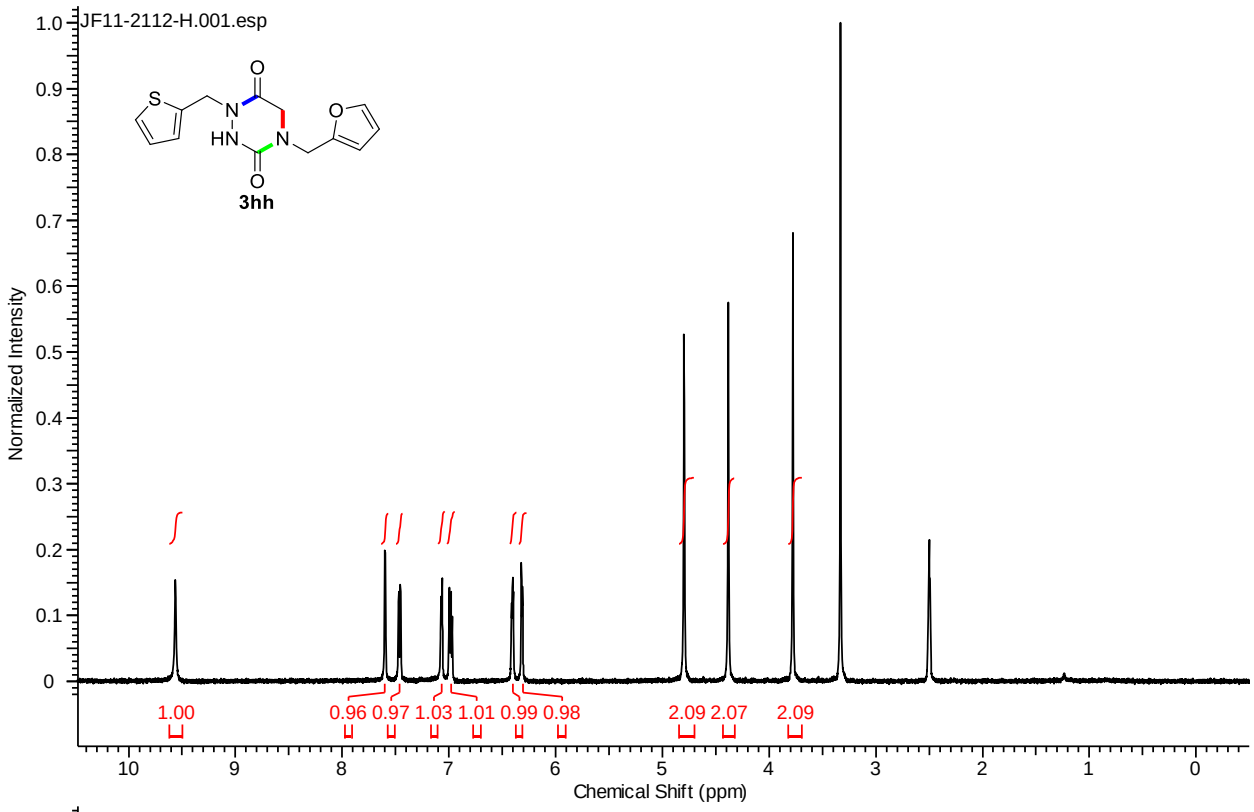

RI5-32-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
159.71
155.77
136.32
127.17
121.90
119.59
118.54
112.43
111.28
53.10
49.90
47.43
26.34
23.40
19.67

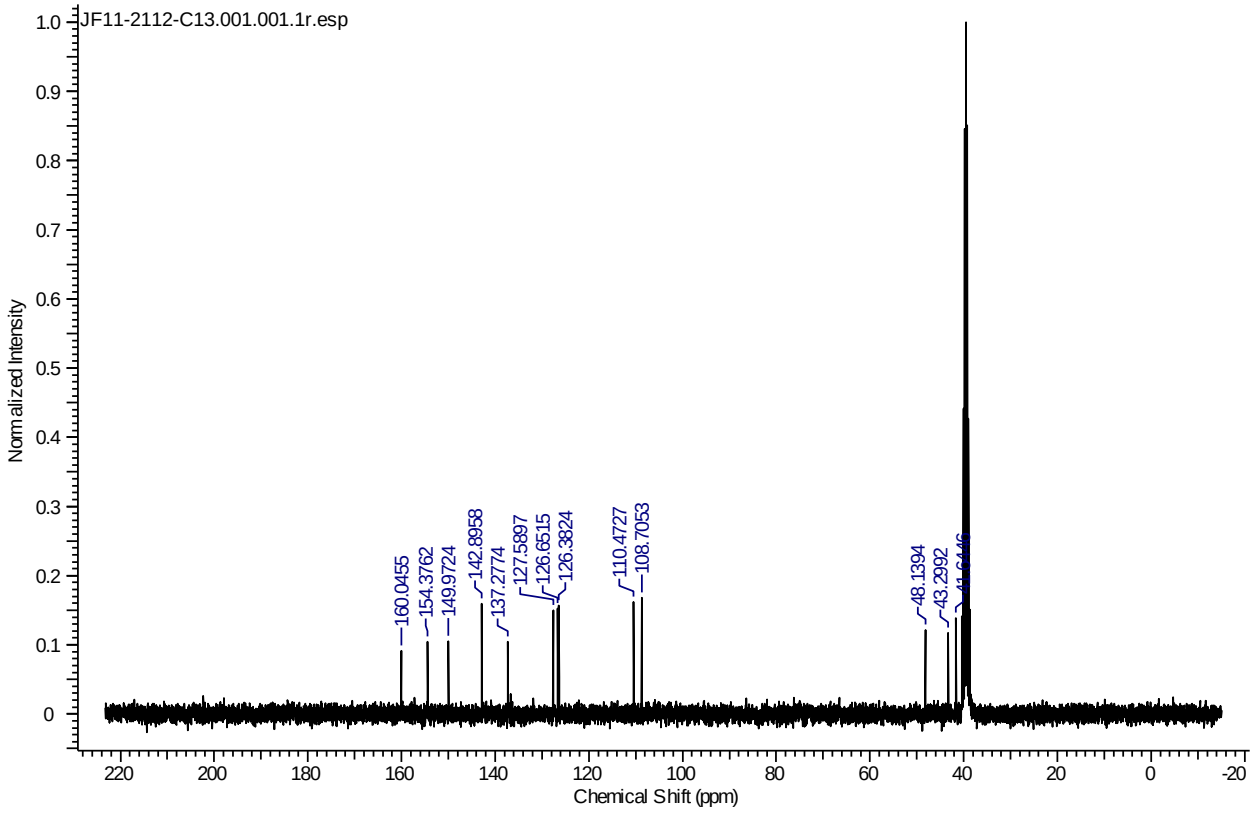

JF11-2092-H.001.esp
3ff
Normalized Intensity
Chemical Shift (ppm)
0.90
1.16
0.84
4.01
11.83
2.19
2.17

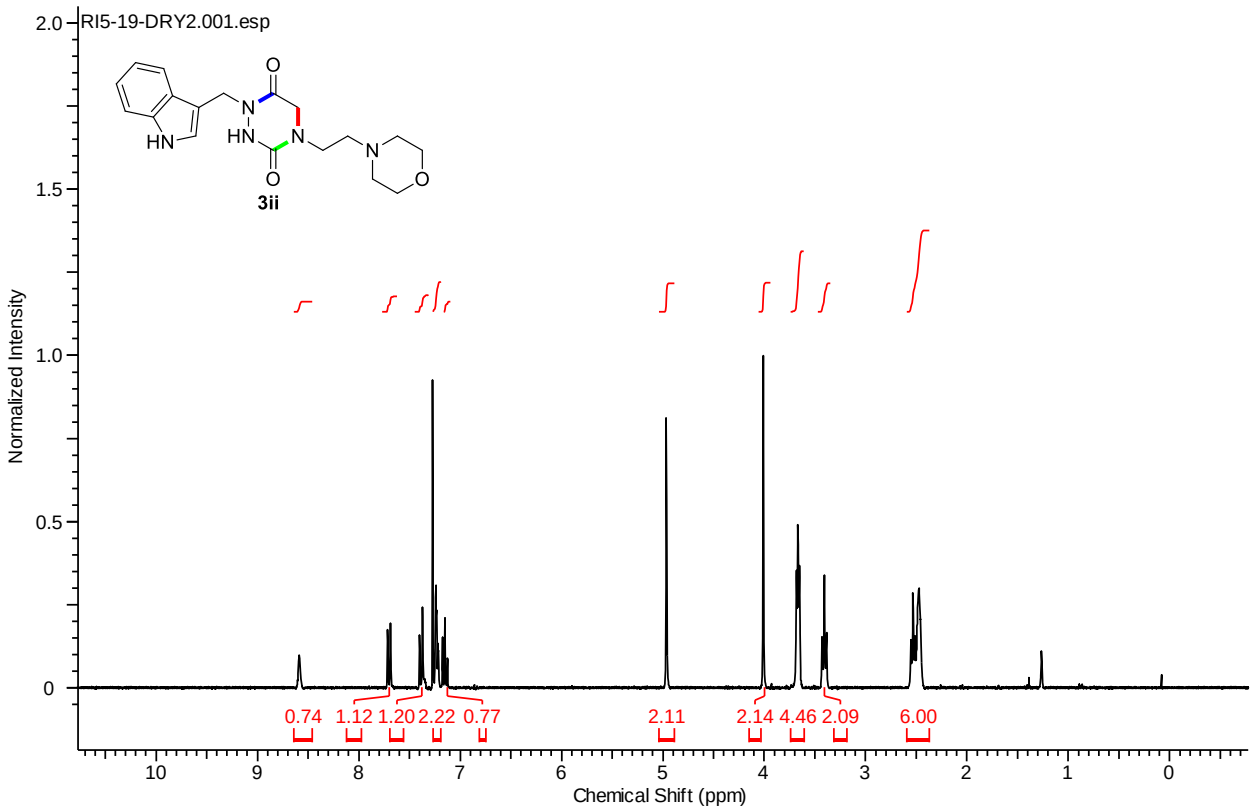

JF11-2092-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
161.7328
155.5617
101.9815
66.1910
53.6633
51.5468
50.2740
46.9976
28.5459

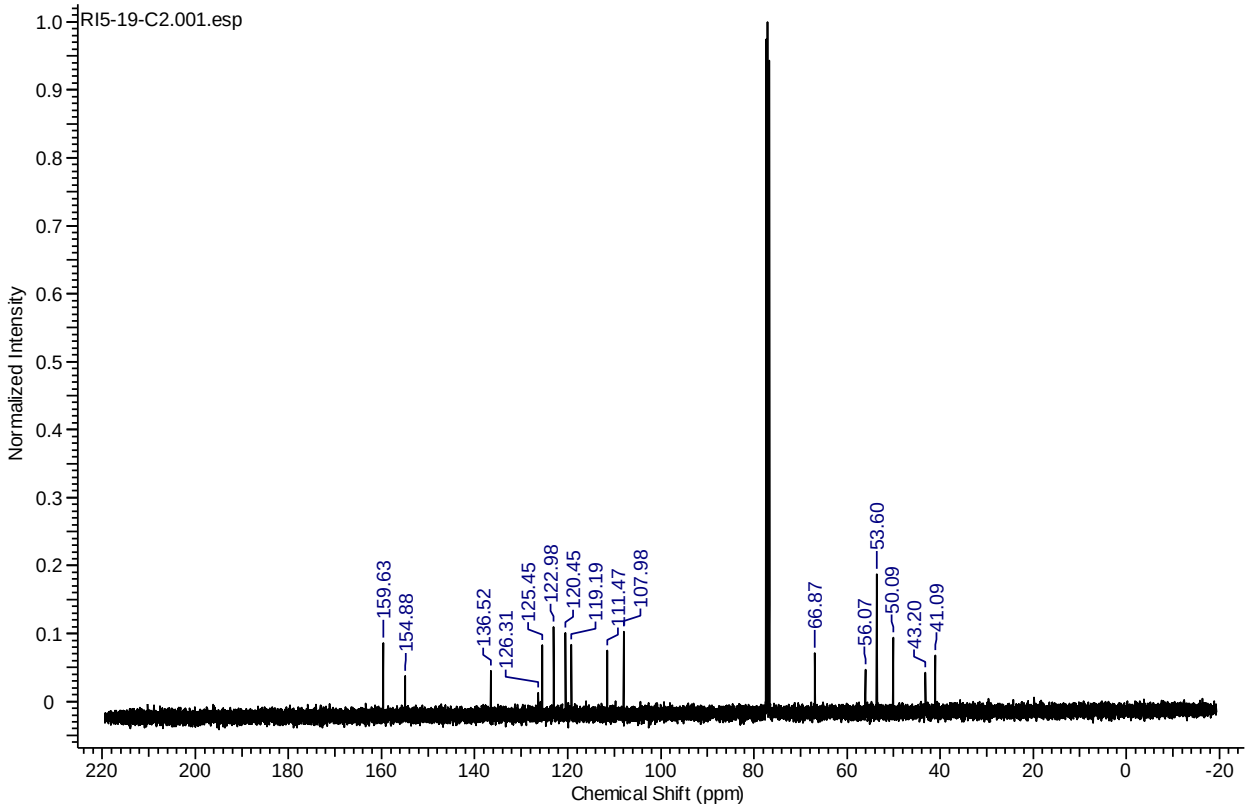

JF11-2093-H.001.esp
3gg
Normalized Intensity
Chemical Shift (ppm)
0.88
0.82
1.02
0.98
2.07
1.05
1.05
3.97
2.00
1.99
2.03
9.45

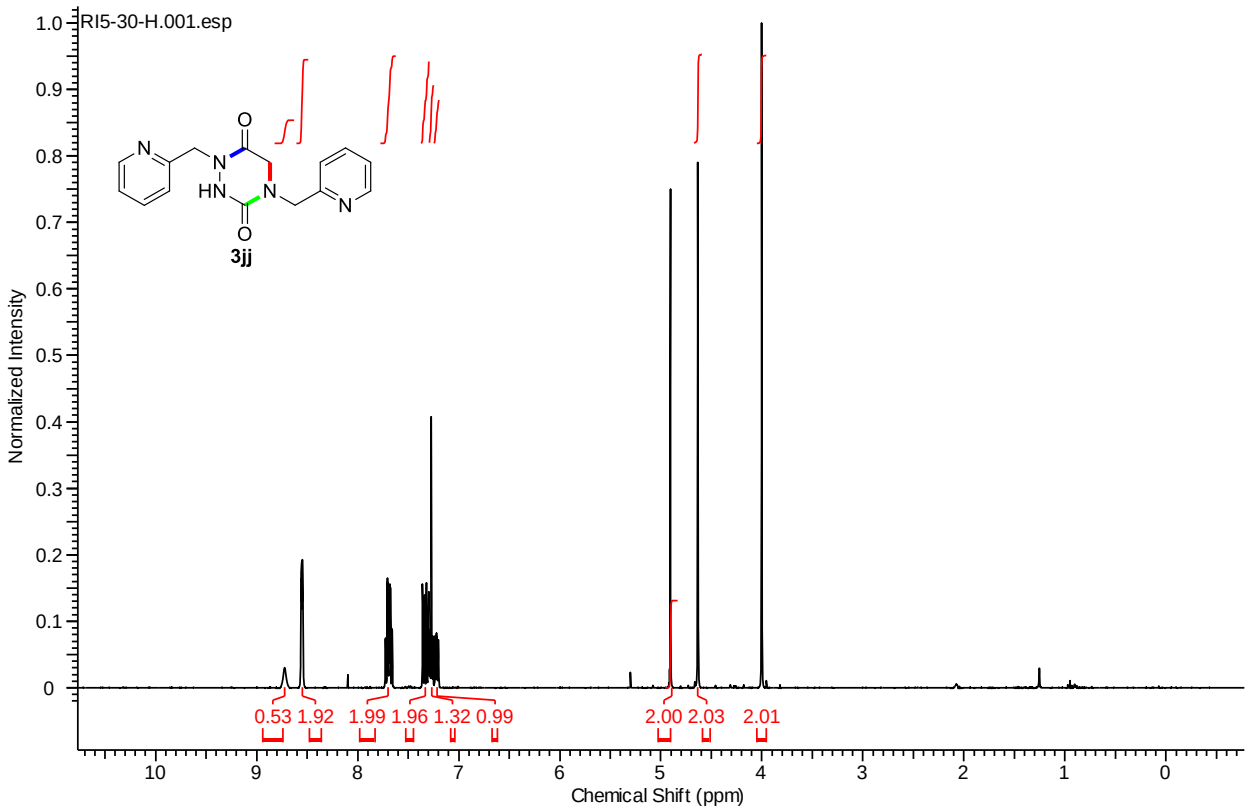

JF11-2093-C13.001.esp
Normalized Intensity
Chemical Shift (ppm)
159.4128
154.2780
135.6300
125.4915
122.2768
115.4402
110.3345
106.8871
77.6933
48.8922
48.4849
45.3429
40.3391
38.6663
28.2077

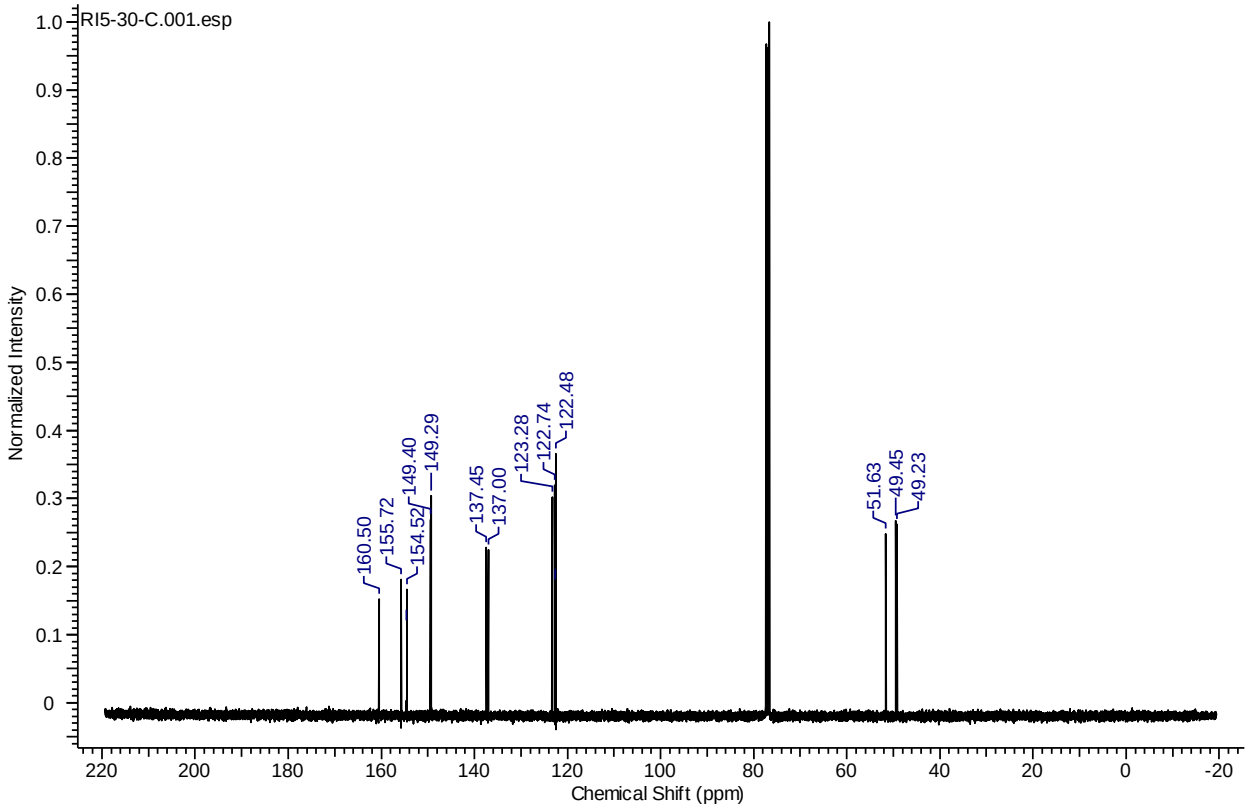

JF11-2112-H.001.esp
3hh
Normalized Intensity
Chemical Shift (ppm)
1.00
0.96 0.97 1.03 1.01 0.99 0.98
2.09 2.07 2.09


JF11-2112-C13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.0455
154.3762
149.9724
142.8958
137.2774
127.5897
126.6515
126.3824
110.4727
108.7053
48.1394
43.2992


RI5-19-DRY2.001.esp
3ii
Normalized Intensity
Chemical Shift (ppm)
0.74
1.12
1.20
2.22
0.77
2.11
2.14
4.46
2.09
6.00


RI5-19-C2.001.esp
Normalized Intensity
Chemical Shift (ppm)
159.63
154.88
136.52
126.31
125.45
122.98
120.45
119.19
111.47
107.98
66.87
56.07
53.60
50.09
43.20
41.09


RI5-30-H.001.esp
3jj
Normalized Intensity
0.53 1.92 1.99 1.96 1.32 0.99 2.00 2.03 2.01
Chemical Shift (ppm)


RI5-30-C.001.esp
Normalized Intensity
160.50
155.72
154.52
149.40
149.29
137.45
137.00
123.28
122.74
122.48
51.63
49.45
49.23
Chemical Shift (ppm)