Synthesis of *N*-Oxyureas by Substitution and Cope-Type Hydroamination Reactions Using *O*-Isocyanate Precursors

Meredith A. Allen,[†] Ryan A. Ivanovich,[†] Dilan E. Polat, and André M. Beauchemin*

Centre for Catalysis Research and Innovation, Department of Chemistry and Biomolecular Sciences, University of Ottawa, 10 Marie-Curie, Ottawa, ON, K1N 6N5, Canada.

andre.beauchemin@uottawa.ca

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General Information. Purification of reaction products was carried out by flash column chromatography using silica gel (40-63 μm), unless otherwise noted. Analytical thin layer chromatography (TLC) was performed on aluminum or glass, cut to size. Visualization

was accomplished with UV light followed by staining with a potassium permanganate solution or cerium molybdate 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 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 (ap = apparent, 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. ^{13}C NMR was reported indicating information from DEPT experiments. Infrared (IR) spectra were obtained were recorded on an Attenuated Total Reflectance Fourier transform infrared spectrometer (ATR-FTIR). High-resolution mass spectroscopy (HRMS) was performed on a mass spectrometer with an electron beam of 70ev (EI) or Micromass Q-TOF I - Time of flight Electrospray Ionisation mass spectrometer (ESI). Compounds that are particularly sensitive to fragmentation were heated to a lower temperature in the EI source. Microwave reactions were performed using a Biotage Initiator Eight microwave reactor using appropriate microwave vials.

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

The syntheses of *N*-oxy-carbamates **1a**, **1b**, **1h**, **1j**, and **1m** were adapted from a literature procedure.[1]

General Procedure 1: Synthesis of *N*-Acyloxycarbamates

To a roundbottom flask charged with a stir bar was added *N*-hydroxycarbamate (1.00 equiv.) then CH_2Cl_2 (0.3 M) and allowed to stir at 0 °C. *Hydroxycarbamates are insoluble and will not always dissolve completely before the next step of the reaction* Upon observing most of the hydroxycarbamate dissolve, Et_3N was added (1.00 equiv.) and finally the acyl chloride was added dropwise (1.00 equiv.). The reaction was allowed to warm gradually to room temperature. Upon completion, H_2O was added and the reaction mixture was extracted with CH_2Cl_2. The organic layer was recollected and washed with a saturated $NaHCO_3$ solution, and then the organic phase was recollected and washed with brine. The organic phase was dried over Na_2SO_4 (stirring for 15 minutes). Solids were filtered and the filtrate was collected and concentrated under reduced pressure to give the corresponding pure acyloxycarbamates.

General Procedure 2: Substitution of *N*-Hydroxycarbamates

To a 5 mL microwave vial charged with a stir bar was added the phenyl *N*-hydroxycarbamate **1a** (1.10 equiv.), then THF (0.3 M), to which was added triethylamine (0.20 equiv.) and the corresponding amine (1.00 equiv.). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via conventional (oil bath) heating. Solvent was removed under reduced pressure and the products were then purified by silica gel chromatography to give the corresponding pure hydroxyureas

General Procedure 3: Substitution of *N*-Alkoxycarbamates

To a 5 mL microwave vial charged with a stir bar was added the phenyl *N*-alkoxycarbamate (1.05 equiv.), then MeCN (0.3 M), to which was added the corresponding amine (1.00 equiv.). The vial was sealed with a microwave cap and heated for 30 min at 120 ^{0}C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH and once with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure to give the corresponding pure *N*-ethoxyureas.

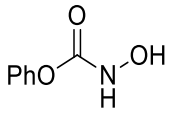
General Procedure 4: Substitution of *N*-Acyloxycarbamates

To a 5 mL microwave vial charged with a stir bar was added the phenyl *N*-acyloxycarbamate (1.00 equiv.), then THF (0.2 M), to which was added the corresponding amine (1.05 equiv.) and then imidazole (0.10 equiv.). The vial was sealed with a microwave cap and heated for 1-3 hours at 100 ^{0}C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH and once with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure to give the corresponding pure acyloxyureas. In some cases, a higher degree of purity was needed after the extraction and the products could easily be recrystallized or purified by silica gel chromatography.

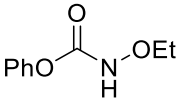
General Procedure 5: Cascade Substitution/Hydroamination of *N*-Oxyureas

To an oven-dried microwave vial was weighed the allylamine (1.00 equiv.). Phenyl hydroxycarbamate **1a** (1.00 equiv.), trifluoromethanesulfonamide (0.50 equiv.), EtOAc (0.2 M), and a stir bar were added. The vial was sealed with a microwave cap, purged with argon, and heated to 175 °C via microwave irradiation for 20 min- 2.5 h. The reaction mixture was then concentrated under reduced pressure. Purification was performed by flash chromatography.

Synthesis of *O*-isocyanate precursors (**Table 1**)**:**

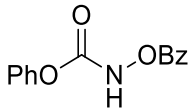


Phenyl *N*-hydroxycarbamate 1a: The title compound was synthesized according to a literature procedure.[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.31 (br s, 1H), 9.08 (br s, 1H), 7.40 (m, 2H), 7.22 (m, 1H), 7.10 (m, 2H).

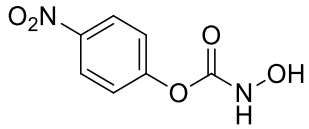


Phenyl *N*-ethoxycarbamate 1b: The title compound was synthesized according to a literature procedure[1] using phenyl chloroformate (12.6 mL, 0.100 mol), *O*-ethoxylamine hydrochloride (11.7 g, 0.120 mol), and sodium bicarbonate (18.5 g, 0.220 mol) in

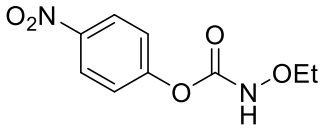
H_2O/CH_2Cl_2 (1:1) (0.4 M). The title compound was obtained as a crystalline white solid (14.9 g, 82%). TLC R*f* = 0.73 in 2% MeOH/CH_2Cl_2. M. p. 60.5-61.6 ^{0}C. ^{1}H (300 MHz, $CDCl_3$) δ 7.64-7.57 (br s, 1H), 7.43-7.34 (m, 2H), 7.27-7.20 (m, 1H), 7.19-7.13 (m, 2H), 4.05 (q, *J* = 7.1 Hz, 2H), 1.32 (t, *J* = 7.0 Hz, 3H). ^{13}C (75 MHz, $CDCl_3$) δ 155.3 (C), 150.4 (C), 129.4 (C), 125.8 (CH), 121.3 (CH), 72.7 (CH_2), 13.4 (CH_3). IR (FTIR): 3204, 2983, 1719, 1588, 1475, 1376, 1251 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_9H_{11}NO_3Na$ [M+Na]+: 204.0637. Found: 204.0647.



Phenyl *N*-benzoyloxycarbamate 1c: The title compound was synthesized according to general procedure **1** using phenyl *N*-hydroxycarbamate **1a** (38.3 g, 0.250 mol), Et_3N (34.9 g, 0.250 mol) and benzoyl chloride (29.0 mL, 0.250 mol) in CH_2Cl_2 (0.83 L, 0.30 M). The reaction was allowed to stir at room temperature overnight. Upon completion, H_2O was added and the reaction mixture was extracted with CH_2Cl_2. The organic layer was recollected and washed with $NaHCO_3$, and then the organic phase was recollected again and washed with brine. The organic phase was dried over Na_2SO_4 (stirring for 15 minutes). Solids were filtered and the filtrate was collected and concentrated under reduced pressure to yield an amorphous white solid (61.2 g, 95%). TLC R*f* = 0.35 in 20% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.66 (s, 1H), 8.11 (d, *J* = 7.9 Hz, 2H), 7.66-7.61 (m, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.39-7.34 (m, 2H), 7.25-7.16 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7 (C), 154.7 (C), 150.1 (C), 134.3 (CH), 129.9 (CH), 129.4 (CH), 128.7 (CH), 126.4 (C), 126.1 (CH), 121.2 (CH). IR (FTIR): 3271, 1770, 1739, 1450, 1229, 1030 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{14}H_{11}NO_4Na$ $[M+Na]^+$: 280.0587. Found 280.0586.

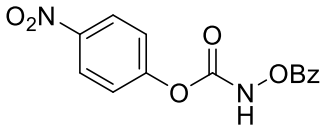


4-Nitrophenyl hydroxycarbamate 1d: The title compound was synthesized according to a literature procedure.[2] ^{1}H-NMR (300 MHz, DMSO-d_6) δ 10.71 (s, 1H), 9.31 (s, 1H), 8.30-8.25 (m, 2H), 7.44-7.38 (m, 2H).

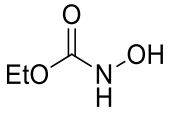


1-(4-Nitrophenol)-N-(O-ethylhydroxy) carbamate 1e: The title compound was synthesized according to a modified literature procedure[1] using *O*-ethyl hydroxylamine hydrochloride (0.610 g, 6.20 mmol), sodium bicarbonate (0.949 g, 11.3 mmol) and 4-nitrophenyl chloroformate (0.629 g, 3.10 mmol) in CH_2Cl_2 (10 mL, 0.30 M) were stirred at room temperature overnight. The reaction mixture was diluted with CH_2Cl_2 and transferred to a separatory funnel, then washed with brine, dried over $MgSO_4$ and filtered.

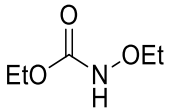
The reaction mixture was concentrated under reduced pressure and recrystallized in diethyl ether. The title compound was obtained as a crystalline white solid (0.356 g, 51%). TLC R*f* = 0.73 in 2% MeOH/CH_2Cl_2. M.p. 128.9-129.9 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31-8.25 (m, 2H), 7.75-7.67 (br s, 1H), 7.40-7.34 (m, 4H), 4.07 (q, *J* = 7.1 Hz, 2H), 1.34 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) 155.1 (C), 153.9 (C), 145.1 (C), 125.2 (CH), 121.9 (CH), 72.9 (CH_2), 13.3 (CH_3). IR (FTIR): 3164, 2930, 1755, 1737, 1615, 1519, 1487, 1344 cm^{-1}. HRMS (EI): Exact mass could not be obtained for the molecular ion. Exact mass calcd for fragment $[C_6H_5NO_3]^+$ (*p*-nitrophenol): 139.0264. Found: 139.0276. Exact mass calcd for fragment $[C_3H_5NO_2]^+$ (ethoxy-isocyanate): 87.0315. Found: 87.0321.



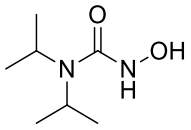
1-(4-Nitrophenol)-N-(O-benzoylhydroxy) carbamate 1f: The title compound was synthesized according to general procedure **1** using 1-(4-nitrophenol)-*N*-hydroxycarbamate (0.198 g, 1.00 mmol), triethylamine (0.14 mL, 1.0 mmol) and benzoyl chloride (0.12 mL, 1.0 mmol) in CH_2Cl_2 (3.3 mL, 0.30 M) were stirred at room temperature overnight. The reaction mixture was diluted with CH_2Cl_2 and transferred to a separatory funnel, then washed with brine, dried over $MgSO_4$ and filtered. The reaction mixture was concentrated under reduced pressure and recrystallized in diethyl ether. The title compound as obtained as a crystalline white solid (0.109 g, 36%). TLC R*f* = 0.77 in 2% MeOH/CH_2Cl_2. M. p. 152.3-153.0 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.69-8.63 (br s, 1H), 8.33-8.27 (m, 2H), 8.17-8.12 (m, 2H), 7.72-7.66 (m, 1H), 7.57-7.50 (m, 2H), 7.45-7.39 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.5 (C), 154.7 (C), 153.2 (C), 145.5 (C), 134.7 (CH), 130.1 (CH), 128.9 (CH), 126.0 (C), 125.3 (CH), 122.0 (CH). IR (FTIR): 3167, 1773, 1755, 1520, 1487, 1344 cm^{-1}. HRMS (EI/ESI): Accurate mass could not be obtained due to the instability of this compound. Exact mass calcd for $C_{24}H_{15}N_3O_9Na$ $[M+Na]^+$ (*O*-benzoyl isocyanurate trimer): 512.0706. Found: 512.0724.



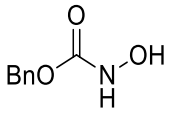
Ethyl hydroxycarbamate 1g: The title compound was synthesized according to a literature procedure using ethyl chloroformate (2.9 mL, 0.030 mol), hydroxylamine hydrochloride (10.4 g, 0.150 mol), and sodium hydroxide (7.20 g, 0.180 mol).[3] The title compound was obtained as a clear and colourless liquid (2.40 g, 76 %) and was used without further purification. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 (br s, 2H), 4.19 (q, *J* = 7.1 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H).



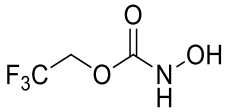
Ethyl ethoxycarbamate 1h: The title compound was synthesized according to a literature procedure[1] using ethyl chloroformate (1.44 mL, 15.0 mmol), *O*-ethoxylamine hydrochloride (1.83 g, 18.9 mmol), and sodium bicarbonate (2.77 g, 33.0 mmol) in CH_2Cl_2/ H_2O (1:1) (0.5 M). The crude reaction mixture was isolated using flash chromatography (25% EtOAc/Hexane). The title compound was obtained as a clear and colourless liquid (1.83 g, 91%).^{1}H NMR (300 MHz, $CDCl_3$) δ 4.21 (q, *J* = 7.2 Hz, 2H), 3.93 (q, *J* = 7.0 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.25 (t, *J* = 7.0 Hz, 3H).



3-Hydroxy-1,1-diisopropylurea 1i: The title compound was synthesized according to a prep. of *N*, *N*-diethyl hydroxyurea using diisopropylcarbamic chloride (1.64 g, 10 mmol), hydroxylamine hydrochloride (0.879 g, 11.5 mmol), and potassium carbonate (1.45 g, 10.5 mmol) in EtOAc/H_2O (60:1) (1.45 M).[4] The crude reaction mixture was isolated using flash chromatography (30% EtOAc/Hexane then 40% EtOAc/Hexane). The title compound was obtained as an amorphous white solid (0.544 g, 34%). TLC R*f* = 0.34 in 40% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.77 (br s, 1H), 6.48 (br s, 1H), 3.77 (sept, *J*= 6.8 Hz, 2H), 1.28 (d, *J* = 6.8 Hz, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6 (C), 45.9 (CH), 20.8 (CH_3). IR (FTIR): 3292, 2964, 2927, 1632, 1493, 1446, 1376, 1367, 1047 cm^{-1}. HRMS (EI): Exact mass calcd for $C_7H_{16}N_2O_2$ $[M]^+$: 160.1212. Found: 160.1208.

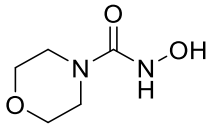


Benzyl hydroxycarbamate 1j: The title compound was synthesized according to a literature procedure[1] using benzyl chloroformate (1.43 mL, 10.0 mmol), hydroxylamine hydrochloride (0.777 g, 11.3 mmol), and sodium bicarbonate (1.85 g, 22.0 mmol) in 1.4 : 1 CH_2Cl_2/H_2O (0.5 M). The crude reaction mixture was isolated using flash chromatography (2% MeOH/CH_2Cl_2, then 5% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous white solid (0.998 g, 60%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 (s, 5H), 7.28 (s, 1H), 6.81 (br s), 5.18 (s, 2H).

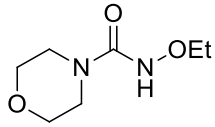


2,2,2-Trifluoroethyl hydroxycarbamate 1k: The title compound was synthesized according to a literature procedure.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (br s, 1H), 6.18 (br s, 1H), 4.56 (q, *J* = 8.3 Hz, 2H).

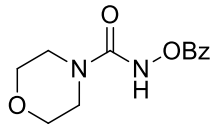
Substitution of *O*-isocyanate precursors (**Table 1**):



1-Morpholin-3-hydroxy-urea 2a: The title compound was synthesized using a modified general procedure **2** using phenyl *N*-hydroxycarbamate **1a** (0.754 g, 4.93 mmol), DIPEA (0.013 mL, 0.20 mmol) and morpholine (0.43 mL, 5.0 mmol) in THF (17 mL, 0.30 M). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via an oil bath. The reaction mixture was concentrated under reduced pressure and recrystallized in Et_2O/Hexane. The title compound was obtained as a crystalline white solid (0.511 g, 72%). TLC R*f* = 0.73 in 2% MeOH/CH_2Cl_2. M. p. 172.0-172.7 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 9.09-9.06 (br s, 1H), 8.07-8.04 (br s, 1H), 3.54-3.49 (m, 4H), 3.24-3.19 (m, 4H). ^{13}C (75 MHz, DMSO-d_6) δ 159.7(C), 65.8 (CH_2), 43.6 (CH_2). IR (FTIR): 3303, 3163, 1773, 1519, 1487 cm^{-1}. HRMS (EI): Exact mass calcd for $C_5H_{10}N_2O_3$ $[M]^+$: 146.0691. Found: 146.0688.



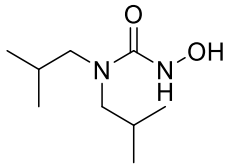
1-Morpholin-3-ethoxy-urea 2b: The title compound was synthesized according to general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.901 g, 1.05 mmol) and morpholine (0.087 g, 1.0 mmol) in MeCN (5.0 mL, 0.20 M). The vial was sealed with a microwave cap and heated for 30 min at 120 °C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH, with brine, dried over $MgSO_4$, filtered. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (3% MeOH/CH_2Cl_2 then 10% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous yellowish solid (0.174 g, 94%). TLC R*f* = 0.35 in 5% MeOH/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15-6.82 (br s, 1H), 3.92 (q, *J* = 7.1 Hz, 2H), 3.73-3.64 (m, 4H), 3.45-3.37 (m, 4H), 1.26 (t, *J* = 7.2 Hz, 3H). ^{13}C (75 MHz, $CDCl_3$) δ 159.2 (C), 71.8 (CH_2), 66.4 (CH_2), 44.0 (CH_2), 13.5 (CH_3). IR (FTIR): 3165, 1773, 1518, 1487, 1451, 1253 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_7H_{14}N_2O_3Na$ $[M+Na]^+$: 197.0902. Found: 197.0902.



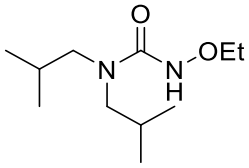
***N*-(Benzoyloxy)morpholine-4-carboxamide 2c:** The title compound was synthesized according to general procedure **4** using phenyl *N*-benzoyloxycarbamate **1c** (0.257 g, 1.00 mmol), and diisobutylamine (0.18 mL, 1.05 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 3 h at 120 °C. Upon completion, a small amount of Hexane was added to the crude reaction

resulting in a precipitate that was filtered and subsequently washed with a small amount of ether. The title compound was obtained as an off-white amorphous solid (0.156 g, 57 %). TLC R*f* = 0.18 in 50% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.67-8.29 (br s, 1H), 8.16-8.08 (m, 2H), 7.69-7.60 (m, 1H), 7.55-7.46 (m, 2H), 3.80-3.69 (m, 4H), 3.59-3.50 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7 (C), 157.2 (C), 134.2 (CH), 129.9 (CH), 128.7 (CH), 126.9 (C), 66.3 (CH_2), 44.2 (CH_2). IR (FTIR): 3162, 3075, 2980, 2960, 2916, 2841, 1752, 1663, 1507, 1424, 1239 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{12}H_{14}N_2O_4Na$ $[M+Na]^+$: 273.0851. Found: 273.0866.

O-Isocyanate Substitution: Amine Scope **(Scheme 3):**

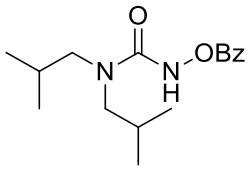


1,1-Diisobutyl-3-hydroxy-urea 3a: The title compound was synthesized according to general procedure **2** using phenyl *N*-hydroxycarbamate **1a** (0.842 g, 5.50 mmol), triethylamine (0.14 mL, 1.0 mmol) and diisobutylamine (0.87 mL, 5.0 mmol) in THF (17 mL, 0.30 M). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via oil bath heating. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (2% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous white solid (0.785 g, 84%). TLC R*f* = 0.54 in 2% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.65-6.61 (br s, 1H), 6.60-6.52 (br s, 1H), 3.07 (d, *J* = 7.6 Hz, 4H), 1.99 (sept, *J* = 7.5 Hz, 2H), 0.92 (d, *J* = 6.7 Hz, 12H). ^{13}C (75 MHz, $CDCl_3$) δ 162.0 (C), 54.4 (CH_2), 27.1 (CH), 20.1 (CH_3). IR (FTIR): 3365, 3295, 1699, 1496, 1276 cm^{-1}. HRMS (EI): Exact mass calcd for $C_9H_{20}N_2O_2$: 188.1525. Found: 188.1515.

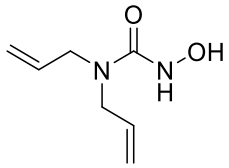


1,1-Diisobutyl-3-ethoxy-urea 3b: The title compound was synthesized according to general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.901 g, 1.05 mmol) and diisobutylamine (0.129 g, 1.00 mmol) in MeCN (5.0 mL, 0.20 M). The vial was sealed with a microwave cap and heated for 30 min at 120 °C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH, with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The title compound was obtained as an amorphous white solid (0.211 g, 98%). TLC R*f* = 0.65 in 5% MeOH/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08-7.05 (br s, 1H), 3.92 (q, *J* = 7.1 Hz, 2H), 3.03 (d, *J* = 7.6 Hz, 4H), 2.04-1.92 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H), 0.90 (d, *J* = 6.7 Hz, 12H). ^{13}C (75 MHz, $CDCl_3$) δ 159.7 (C), 71.8 (CH_2), 55.1 (CH_2), 27.3 (CH), 20.2 (CH), 13.6 (CH). IR (FTIR): 3197, 2957, 1636, 1520,

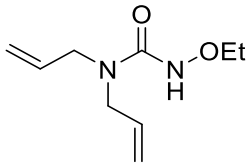
1410, 1383 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{11}H_{24}N_2O_2Na$ $[M+Na]^+$: 239.1736. Found: 239.1729.



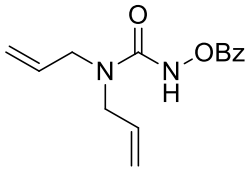
3-(Benzoyloxy)-1,1-diisobutylurea 3c: The title compound was synthesized according to general procedure **4** using phenyl *N*-benzoyloxycarbamate **1c** (0.257 g, 1.00 mmol), and diisobutylamine (0.18 mL, 1.05 mmol) and imidazole (0.007 g, 0.10 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 3 h. The reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.260 g, 89%). TLC R*f* = 0.35 in 40% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (br s, 1H), 8.11 (dd, *J* = 8.4, 1.1 Hz, 2H), 7.62-7.58 (m, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 3.14 (d, *J* = 7.6 Hz, 4H), 2.16-1.97 (m, 2H), 0.95 (d, *J* = 6.7 Hz, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3 (C), 158.1 (C), 134.1 (C), 130.0 (CH), 128.7 (CH), 127.3 (CH), 55.1 (CH_2), 27.3 (CH), 20.3 (CH_3). IR (FTIR): 3125, 2958, 2872, 1760, 1655, 1482, 1417, 1236 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{16}H_{24}N_2O_3Na$ $[M+Na]^+$: 315.1685. Found: 315.1700.



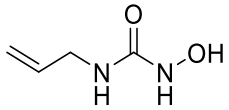
1,1-Diallyl-3-hydroxy-urea 3d: The title compound was synthesized according to general procedure **2** using phenyl *N*-hydroxycarbamate **1a** (0.168 g, 1.00 mmol), triethylamine (0.03 mL, 0.2 mmol) and diallylamine (0.097 mg, 1.0 mmol) in THF (3.3 mL, 0.30 M). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via an oil bath heating. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (2% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous white solid (0.098 g, 63%). TLC R*f* = 0.53 in 5% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.86-6.80 (br s, 1H), 6.69 (d, *J* = 3.1 Hz, 1H), 5.86-5.72 (m, 2H), 5.27-5.18 (m, 4H), 3.89 (dt, *J* = 5.5, 1.5 Hz, 4H). ^{13}C (75 MHz, $CDCl_3$) δ 161.5 (C), 132.8 (CH), 117.5 (CH_2), 49.0 (CH_2). IR (FTIR): 3246, 2915, 1640, 1471, 1414, 1281 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{14}N_2O_2$ $[M]^+$: 156.0899. Found: 156.0914.



1,1-Diallyl-3-ethoxy-urea 3e: The title compound was synthesized according to general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.901 g, 1.05 mmol) and diallylamine (0.097 g, 1.0 mmol) in MeCN (5.0 mL, 0.20 M). The vial was sealed with a microwave cap and heated for 30 min at 120 °C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH, with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The title compound was obtained as a yellow oil (0.158 g, 86%). TLC R*f* = 0.26 in 2% MeOH/CH_2Cl_2. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14-7.11 (br s, 1H), 5.85-5.74 (m, 2H), 5.52-5.18 (m, 4H), 3.91 (q, *J* = 7.1Hz, 2H), 3.86 (dt, *J* = 5.6, 1.4 Hz, 4H), 1.24 (t, *J* = 7.0 Hz, 3H). ^{13}C (75 MHz, $CDCl_3$) δ 159.3 (C), 133.4 (CH), 117.3 (CH_2), 71.8 (CH_2), 49.1 (CH_2), 13.5 (CH_3). IR (FTIR): 3228, 2979, 1643, 1471, 1283 cm^{-1}. HRMS (EI): Exact mass calcd for $C_9H_{16}N_2O_2$ $[M]^+$: 184.1212. Found 184.1240.

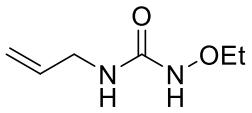


1,1-Diallyl-3-(benzoyloxy)urea 3f: The title compound was synthesized according to general procedure **4** using phenyl *N*-benzoyloxycarbamate **1c** (0.257 g, 1.00 mmol), and diallylamine (0.13 mL, 1.05 mmol) and imidazole (0.007 g, 0.10 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated via microwave irradiation for 3 h. The reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.232 g, 89%). TLC R*f* = 0.35 in 30% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (br s, 1H), 8.11 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.61 (tt, *J* = 7.4, 1.6 Hz, 1H), 7.46 (tt, *J* = 7.6, 1.4 Hz, 2H), 5.86 (ddt, *J* = 17.2, 10.4, 5.3 Hz, 2H), 5.35-5.27 (m, 4H), 3.97 (dt, *J* = 5.4, 1.4 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9 (C), 157.8 (C), 134.1 (C), 132.7 (CH), 130.0 (CH), 128.6 (CH), 127.2 (CH), 117.8 (CH_2), 49.3 (CH_2). IR (FTIR): 3226, 3078, 2959, 1759, 1662, 1600, 1484, 1235 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{14}H_{16}N_2O_3H$ $[M+H]^+$: 261.1239. Found: 261.1214.

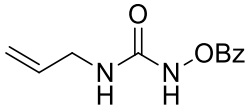


1-Allyl-3-hydroxy-urea 3g: The title compound was synthesized according to a modified general procedure **2** using phenyl *N*-hydroxycarbamate **1a** (0.306 g, 2.00 mmol),

triethylamine (0.006 g, 0.2 mmol) and allylamine (0.75 mL, 10 mmol) in THF (6.7 mL, 0.30 M). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via oil bath heating. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (3% MeOH/CH_2Cl_2 then 10% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous white solid (0.156 g, 67%). TLC R*f* = 0.10 in 2% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.57 (d, *J* = 1.2 Hz, 1H), 8.33-8.31 (br s, 1H), 6.82-6.74 (br s, 1H), 5.88-5.74 (m, 1H), 5.13-4.98 (m, 2H), 3.70-3.63 (m, 2H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 161.3 (C), 136.5 (CH), 114.4 (CH_2), 41.0 (CH_2). IR (FTIR): 3310, 3161, 2853, 1592, 1519, 1488, 1345 cm^{-1}. HRMS (EI): Exact mass calcd for $C_4H_8N_2O_2$ $[M]^+$: 116.0586. Found 116.0607.

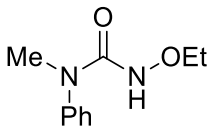


1-Allyl-3-ethoxy-urea 3h: The title compound was synthesized according to a modified general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.901 g, 1.05 mmol) and allylamine (0.37 mL, 5.0 mmol) in MeCN (5.0 mL, 0.20 M). The vial was sealed with a microwave cap and heated for 30 min at 120 °C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH, with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The title compound was obtained as a white solid (0.113 g, 79%). TLC R*f* = 0.47 in 5% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.07-6.96 (br s, 1H), 5.98-5.74 (m, 2H), 5.33-5.12 (m, 2H), 3.98-3.85 (m, 4H), 1.20 (t, *J* = 7.1 Hz, 3H). ^{13}C (75 MHz, $CDCl_3$) δ 160.0 (C), 134.5 (CH), 115.9 (CH_2), 71.9 (CH_2), 41.9 (CH_2), 13.7 (CH_3). IR (FTIR): 3164, 2971, 1669, 1650, 1529, 1441, 1364 cm^{-1}. HRMS (EI): Exact mass calcd for $C_6H_{12}N_2O_2$ $[M]^+$: 144.0899. Found: 144.0900.

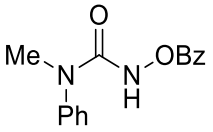


1-Allyl-3-benzoyloxy-urea 3i: The title compound was synthesized according to a modified general procedure **4** using phenyl *N*-benzoylcarbamate **1c** (0.257 g, 1.00 mmol), imidazole (0.007 g, 0.1 mmol) and allylamine (0.37 mL, 5.0 mmol) in THF (3.3 mL, 0.30 M). The vial was sealed with a microwave cap and heated for 1 h at 100 °C via microwave irradiation. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (20% EtOAc/Hexane). The title compound was obtained as a colourless oil (0.129 g, 58%). TLC R*f* = 0.89 in 5% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82-7.77 (m, 2H), 7.55-7.41 (m, 3H), 6.28-6.13 (br s, 1H), 6.03-5.88 (m, 1H), 5.32-5.17 (m, 2H), 4.11 (tt, *J* = 5.8, 1.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4 (C), 134.4 (C), 134.1 (CH), 131.4 (CH), 128.5 (CH), 126.9 (CH), 116.5 (CH_2), 42.4 (CH_2) (one C missing despite multiple attempts, high S/N ratio and long acquisition times). IR (FTIR): 3304, 3065, 1635, 1602, 1576, 1553, 1488, 1305 cm^{-1}. HRMS (EI): Exact mass could not be obtained for the molecular ion. Exact mass calcd for fragment $[C_4H_7N_2O_2]^+$ (*N*-

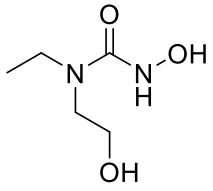
oxyurea): 115.0502. Found: 115.0516, Exact mass calcd for fragment $[C_7H_5O]^+$ (benzoyl): 105.0355. Found: 105.0311.



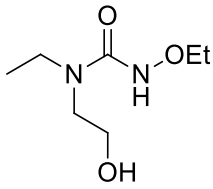
3-Ethoxy-1-methyl-1-phenylurea 3k: The title compound was synthesized according to a modified general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.901 g, 1.05 mmol), *N*-methylaniline (0.107 g, 1.00 mmol) and imidazole (0.006 g, 0.2 mmol) in MeCN (5.0 mL, 0.20 M). The vial was sealed with a microwave cap and heated for 2 h at 120 °C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH, with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (3% MeOH/CH_2Cl_2 then 10% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous yellowish solid (0.078 g, 40%). TLC R*f* = 0.84 in CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.37 (m, 2H), 7.33-7.27 (m, 1H), 7.24-7.19 (m, 2H), 7.00-6.97 (br s, 1H), 3.83 (q, *J* = 6.9 Hz, 2H), 3.24 (s, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.2 (C), 141.9 (C), 129.9 (CH), 127.7 (CH), 126.9 (CH), 71.8 (CH_2), 37.4 (CH_3), 13.3 (CH_3). IR (FTIR): 3409, 2980, 1747, 1669, 1603, 1507, 1202 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{10}H_{14}N_2O_2Na$ $[M+Na]^+$: 217.0953. Found: 217.0943.



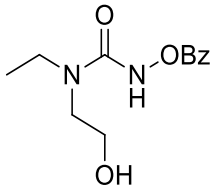
3-(Benzoyloxy)-1-methyl-1-phenylurea 3l: The title compound was synthesized according to general procedure **4** using phenyl *N*-benzoyloxycarbamate **1c** (0.257 g, 1.00 mmol), and *N*-methylaniline (0.13 mL, 1.05 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 6 h at 120 °C. The reaction mixture was concentrated under reduced pressure and purified by column chromatography (10% EtOAc/Hexane then 40% EtOAc/Hexane). The title compound was obtained as an amorphous white solid (0.232 g, 91%). TLC R*f* = 0.40 in 50% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (br s, 1H), 8.07-8.04 (m, 2H), 7.62-7.57 (m, 1H), 7.51-7.35 (m, 7H), 3.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8 (C), 157.1 (C), 141.4 (C), 134.0 (C), 130.4 (CH), 130.0 (CH), 128.7 (CH), 128.6 (CH), 127.2 (CH), 127.1 (CH), 38.0 (CH_3). IR (FTIR): 3125, 2958, 2873, 1760, 1655, 1483, 1452, 1237 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{15}H_{14}N_2O_3Na$ $[M+Na]^+$: 293.0902. Found: 293.0900.



1-Ethyl-3-hydroxy-1-(2-hydroxyethyl)urea 3m: The title compound was synthesized according to general procedure **2** using phenyl *N*-hydroxycarbamate **1a** (0.842 g, 5.50 mmol), triethylamine (0.14 mL, 1.0 mmol) and 2-(ethylamino)ethanol (0.446 g, 5.00 mmol) in THF (17 mL, 0.30 M). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via oil bath heating. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (2% MeOH/CH_2Cl_2 then 5% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous yellowish solid (0.671 g, 91%). TLC R*f* = 0.10 in 2% MeOH/CH_2Cl_2. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.69 (d, *J* = 1.8 Hz, 1H), 7.88 (d, *J* = 2.2 Hz, 1H), 4.87 (t, *J* = 5.1 Hz, 1H), 3.47 (q, *J* = 5.7 Hz, 2H), 3.24-3.16 (m, 4H), 1.00 (t, *J* = 7.1 Hz, 3H). ^{13}C (75 MHz, DMSO-d_6) δ 160.1 (C), 59.9 (CH_2), 48.0 (CH_2), 41.4 (CH_2), 13.3 (CH_3). IR (FTIR): 3164, 2931, 1773, 1615, 1519, 1344 cm^{-1}. HRMS (EI): Exact mass calcd for $C_5H_{12}N_2O_3$ $[M]^+$: 148.0848. Found: 148.0845.

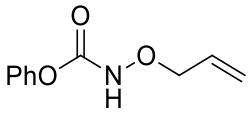


3-Ethoxy-1-ethyl-1-(2-hydroxyethyl)urea 3n: The title compound was synthesized according to general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.901 g, 1.05 mmol) and 2-(ethylamino)ethanol (0.089 g, 1.0 mmol) in MeCN (5.0 mL, 0.20 M). The vial was sealed with a microwave cap and heated for 30 min at 120 °C via microwave irradiation. The reaction mixture was diluted with EtOAc and transferred to a separatory funnel, then washed twice with 1 M NaOH, with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The title compound was obtained as a yellowish solid (0.155 g, 88%). TLC R*f* = 0.26 in 5% MeOH/CH_2Cl_2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.36-8.28 (br s, 1H), 3.87 (q, *J* = 7.0 Hz, 2H), 3.80-3.74 (m, 2H), 3.37-3.26 (m, 4H), 1.23 (t, *J* = 7.0 Hz, 3H), 1.15 (t, *J* = 7.1 Hz, 3H).^{13}C (75 MHz, $CDCl_3$) δ 161.4 (C), 71.4 (CH_2), 61.4 (CH_2), 48.5 (CH_2), 41.8 (CH_2), 13.5 (CH_3), 13.0 (CH_3). IR (FTIR): 3383, 3275, 2976, 1625, 1518, 1487, 1382 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_7H_{16}N_2O_3Na$ $[M+Na]^+$: 199.1059. Found: 199.1064.

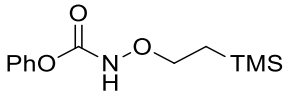


3-(Benzoyloxy)-1-ethyl-1-(2-hydroxyethyl)urea 3o: The title compound was synthesized according to general procedure **4** using phenyl *N*-benzoyloxycarbamate **1c** (0.257 g, 1.00 mmol), and *N*-ethylethanolamine (0.10 mL, 1.05 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 6 h at 120 °C. The reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.159 g, 63%). TLC R*f* = 0.28 in 50% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.66-9.65 (br s, 1H), 8.12-8.08 (m, 2H), 7.62-7.56 (m, 1H), 7.48-7.43 (m, 2H), 3.84 (t, *J* = 4.8 Hz, 2H), 3.47 (t, *J* = 4.8 Hz, 2H), 3.39 (q, *J* = 7.1 Hz, 2H), 3.20 (br s, 1H), 1.18 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0 (C), 159.1 (C), 134.0 (C), 130.0 (CH), 128.7 (CH), 127.6 (CH), 62.2 (CH_2) 48.7 (CH_2) 42.7 (CH_2) 13.2 (CH_3). IR (FTIR): 3355, 3156, 2987, 2916, 1726, 1655, 1464, 1448, 1268, 1236 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{12}H_{16}N_2O_4H$ $[M+H]^+$: 253.1188. Found: 253.1161.

Synthesis of *O*-isocyanate precursors **(Scheme 4):**

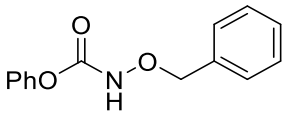


Phenyl allyloxycarbamate 1l: The title compound was synthesized according to a literature procedure.[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (br s, 1H), 7.42-7.35 (m, 2H), 7.26-7.20 (m, 1H), 7.18-7.13 (m, 2H), 6.02 (ddt, *J*= 17.3, 10.3, 6.4 Hz, 1H), 5.44-5.34 (m, 2H), 4.46 (dt, *J*= 6.4, 1.2 Hz, 2H).

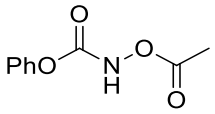


Phenyl 2-(trimethylsilyl)ethoxycarbamate 1m: The title compound was synthesized according to a literature procedure[1] using *O*-(2-(trimethylsilyl)ethyl)hydroxylamine (5.00 g, 37.5 mmol), and phenyl chloroformate (3.93 mL, 31.3 mmol) in 1 : 1 CH_2Cl_2/H_2O (78 mL, 0.40 M). The reaction was allowed to stir at room temperature overnight. Upon completion, the reaction mixture was diluted with CH_2Cl_2 (78 mL) and extracted from the H_2O. The organic phase was recollected and washed three times using saturated $NaHCO_3$ (25 mL), followed by a single brine wash (25 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2

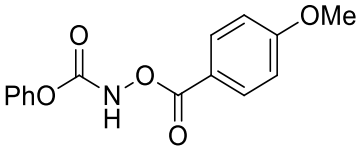
(50 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as a clear oil (7.51 g, 95%). TLC R*f* = 0.75 in 20% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (br s, 1H), 7.40-7.35 (m, 2H), 7.25-7.20 (m, 1H), 7.18-7.15 (m, 2H), 4.06-4.01 (m, 2H), 1.09-1.03 (m, 2H), 0.05 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6 (C), 150.6 (C), 129.5 (CH), 125.9 (CH), 121.5 (CH), 75.0 (CH_2), 16.8 (CH_2), -1.3 (CH_3). IR (FTIR): 3273, 2952, 1733, 1593, 1474, 1370, 1248, 1201 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{12}H_{19}NO_3Si$ [M+Na]$^+$: 276.1032. Found: 276.1034.



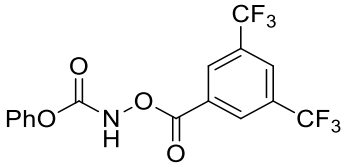
Phenyl benzyloxycarbamate 1n: The title compound was synthesized according to a literature procedure.[7] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (br s, 1H), 7.48-7.37 (m, 7H), 7.28-7.23 (m, 1H), 7.18-7.15 (m, 2H), 4.99 (s, 2H).



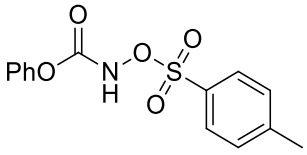
Phenyl acyloxycarbamate 1o: The title compound was synthesized according to a literature procedure.[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.44-8.39 (br s, 1H), 7.41-7.33 (m, 2H), 7.27-7.21 (m, 1H), 7.18-7.10 (m, 2H), 2.23 (s, 3H).



Phenyl (4-methoxybenzoyl)oxycarbamate 1p: The title compound was synthesized according to general procedure **4** using phenyl *N*-hydroxycarbamate **1a** (17.8 g, 0.117 mol), triethylamine (16.4 mL, 0.117 mol) and 4-methoxybenzoyl chloride (20.0 g, 0.117 mol) in CH_2Cl_2 (390 mL, 0.30 M). The reaction was allowed to stir at room temperature overnight. Upon completion the organic phase was washed three times using saturated $NaHCO_3$ (100 mL), followed by a single brine wash (100 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (100 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as a white solid (30.92 g, 92%). TLC R*f* = 0.35 in 20% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.82 (br s, 1H), 8.10 (d, *J* = 8.9 Hz, 2H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.28-7.20 (m, 3H), 6.98 (d, *J* = 8.9 Hz, 2H), 3.89 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.5 (C), 164.6 (C), 154.9 (C), 150.3 (C), 132.3 (CH), 129.6 (CH), 126.2 (CH), 121.3 (CH), 118.6 (C), 114.2 (CH), 55.6 (CH_3). IR (FTIR): 3207, 3020, 2938, 1725, 1605, 1471, 1234 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{15}H_{13}NO_5Na$ [M+Na]$^+$: 310.0692. Found: 310.0698.

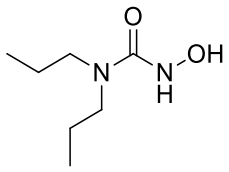


Phenyl (3,5-trifluoromethylbenzoyl)oxycarbamate 1q: The title compound was synthesized according to general procedure **1** using phenyl *N*-hydroxycarbamate **1a** (1.02 g, 6.70 mmol), triethylamine (0.93 mL, 6.7 mmol) and 3,5-trifluoromethylbenzoyl chloride (1.21 mL, 6.70 mmol) in CH_2Cl_2 (22 mL, 0.30 M). The reaction was allowed to stir at room temperature overnight. Upon completion, the reaction mixture was diluted with H_2O (5.0 mL) and extracted three times using CH_2Cl_2 (5.0 mL), followed by a single brine wash (5.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (10.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as a white solid (2.63 g, 99%). TLC R*f* = 0.75 in 20% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (s, 1H), 8.57 (s, 2H), 8.17 (s, 1H), 7.43-7.38 (m, 2H), 7.29-7.18 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4 (C), 154.7 (C), 150.1 (C), 132.8 (q, *J* = 34.5 Hz) (C), 130.2 (m) (CH), 129.7 (CH), 128.9 (C), 127.8 (m) (CH), 126.5 (CH), 122.7 (q, *J* = 273.1 Hz) (CH), 121.2 (CH). IR (FTIR): 3214, 3092, 1780, 1734, 1614, 1591, 1475, 1378, 1279 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{16}H_9F_6NO_4$ $[M]^+$: 393.0436. Found: 393.0450.



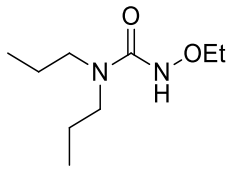
Phenyl tosyloxycarbamate 1r: The title compound was synthesized according to a literature procedure.[9] ^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.07 (s, 1H), 8.00-7.91 (m, 2H), 7.46-7.11 (m, 5H), 6.95-6.84 (m, 2H), 2.48 (s, 3H).

O-Isocyanate Substitution: Oxy-Carbamate Scope (**Scheme 4**):

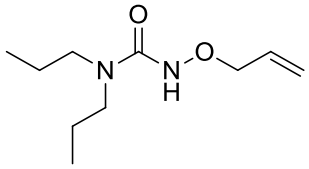


3-Hydroxy-1,1-dipropylurea 4a: The title compound was synthesized according to general procedure **2** using phenyl *N*-hydroxycarbamate **1a** (0.101 g, 0.660 mmol), triethylamine (0.0121 g, 0.120 mmol) and dipropylamine (0.0607 g, 0.060 mmol) in THF (0.30 M). The vial was sealed with a microwave cap and heated for 16 h at 70 °C via oil bath heating. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (3% MeOH/CH_2Cl_2 then 12% MeOH/CH_2Cl_2). The title compound was obtained as an amorphous white solid (0.081 g, 85%). TLC R*f* = 0.18 in 50% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.64 (br s, 1H), 6.58 (br s, 1H), 3.19-

3.15 (m, 4H), 1.63-1.54 (m, 4H), 0.91 (t, J = 7.4 Hz, 6H). ^{13}C (100 MHz, $CDCl_3$) δ 161.4 (C), 48.4 (CH_2), 21.3 (CH_2), 11.2 (CH_3). IR (FTIR): 3274, 2959, 2931, 2875, 1613, 1498, 1376, 1250, 1094 cm^{-1}. HRMS (EI): Exact mass calcd for $C_7H_{16}N_2O_2$ $[M]^+$: 160.1212. Found: 160.1209.

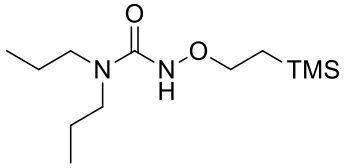


3-Ethoxy-1,1-dipropylurea 4b: The title compound was synthesized according to general procedure **3** using phenyl *N*-ethoxycarbamate **1b** (0.181 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.1 mmol) in MeCN (5.0 mL, 0.20 M). The reaction was heated via microwave irradiation for 20 min at 120 °C. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as a clear oil (0.171 g, 91%). TLC Rf = 0.44 in 20% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (br s, 1H), 3.91 (q, J = 7.0 Hz, 2H), 3.13 (t, J = 7.7 Hz, 4H), 1.57 (ap sext, J = 7.6 Hz, 4H), 1.24 (t, J = 7.0 Hz, 3H), 0.90 (t, J = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4 (C), 71.9 (CH_2), 49.1 (CH_2), 21.7 (CH_2), 13.7 (CH_3), 11.4 (CH_3). IR (FTIR): 3216, 2964, 2933, 2875, 1725, 1644, 1478, 1243 cm^{-1}. HRMS (EI): Exact mass could not be obtained for the molecular ion. Exact mass calcd for fragment $[C_2H_6NO]^+$ (*O*-ethylhydroxylamine): 60.0449. Found: 60.0440, Exact mass calcd for fragment $[C_7H_{14}NO]^+$ (*N,N*-dipropyl carbamoyl): 128.1075. Found: 128.1056.

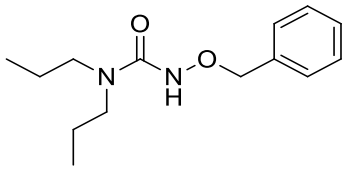


3-(Allyloxy)-1,1-dipropylurea 4c: The title compound was synthesized according to general procedure **3** using phenyl allyloxycarbamate **1l** (0.193 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.1 mmol) in MeCN (5.0 mL, 0.20 M). The reaction was heated via microwave irradiation for 20 min. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.188 g, 94%). TLC Rf = 0.43 in 20% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (br s, 1H), 5.97 (ddt, J = 17.1, 10.5, 6.4 Hz, 1H), 5.33-5.24 (m, 2H), 4.34 (dt, J = 6.3, 1.1 Hz, 2H), 3.11 (t, J = 7.7 Hz, 4H), 1.55 (ap sext, J = 7.6 Hz, 4H), 0.87 (t, J = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1 (C), 133.1 (CH),

119.7 (CH_2), 77.2 (CH_2), 48.9 (CH_2), 21.6 (CH_2), 11.4 (CH_3). IR (FTIR): 3219, 2962, 2927, 2873, 1644, 1481, 1405, 1379, 1244 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{10}H_{20}N_2O_2Na$ $[M+Na]^+$: 223.1423. Found: 223.1394.

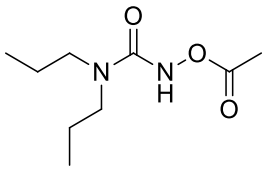


1,1-Dipropyl-3-(2-(trimethylsilyl)ethoxy)urea 4d: The title compound was synthesized according to general procedure **3** using phenyl 2-(trimethylsilyl)ethoxycarbamate **1m** (0.253 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.05 mmol) in MeCN (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 20 min. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.242 g, 93%). TLC R*f* = 0.38 in 20% EtOAc/Hexane. ^1H NMR (400 MHz, $CDCl_3$) δ 6.98 (br s, 1H), 3.91-3.87 (m, 2H), 3.11 (dd, *J* = 8.4, 7.0 Hz, 4H), 1.55 (ap sext, *J* = 7.6 Hz, 4H), 1.01-0.97 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 6H), -0.05 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4 (C), 74.1 (CH_2), 49.1(CH_2), 21.7 (CH_2), 16.9 (CH_2), 11.5 (CH_3), 1.28 (CH_3). IR (FTIR): 3192, 2959, 2929, 2875, 1636, 1518, 1490, 1405, 1426 cm^{-1}. HRMS (ESI): Exact mass calcd for $C_{12}H_{28}N_2O_2SiNa$ $[M+Na]^+$: 283.1818. Found: 283.1821.

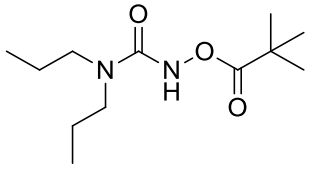


3-(Benzyloxy)-1,1-dipropylurea 4e: The title compound was synthesized according to general procedure **3** using phenyl benzyloxycarbamate **1n** (0.243 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.05 mmol) in MeCN (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 20 min. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.240 g, 96%). TLC R*f* = 0.39 in 20% EtOAc/Hexane. ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.30 (m, 5H), 6.96 (br s, 1H), 4.87 (s, 2H), 3.08 (t, *J* = 7.7 Hz, 4H), 1.51 (ap sext, *J* = 7.6 Hz, 4H), 0.84 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0 (C), 136.3 (C), 129.3 (CH), 128.6 (CH), 128.5 (C), 78.1 (CH_2), 49.0 (CH_2), 21.6 (CH_2), 11.4 (CH_3). IR (FTIR): 3203, 3064, 3033, 2958, 2928, 2872, 1648, 1505, 1473,

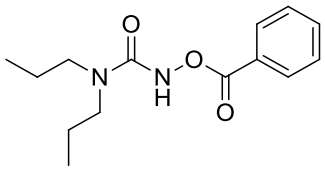
1455, 1413, 1239 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{14}H_{22}N_2O_2$ $[M]^+$: 250.1681. Found: 250.1664.



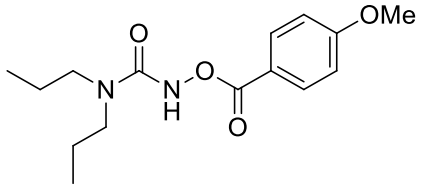
3-Acetoxy-1,1-dipropylurea 4f: The title compound was synthesized according to general procedure **4** using phenyl acetoxycarbamate **1o** (0.195 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.05 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 3 h. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.125 g, 62%). TLC R*f* = 0.42 in 20% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 (br s, 1H), 3.17 (t, *J* = 7.6 Hz, 4H), 2.19 (s, 3H), 1.57 (ap sext, *J* = 7.5, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1 (C), 157.5 (C), 48.9 (CH_2), 21.4 (CH_2), 18.6 (CH_3), 11.3 (CH_3). IR (FTIR): 3162, 2964, 2936, 2877, 1782, 1655, 1478, 1376 cm^{-1}. HRMS (EI): Exact mass calcd for $C_9H_{18}N_2O_3$ $[M]^+$: 202.1317. Found: 202.1324.



3-(Pivaloyloxy)-1,1-dipropylurea 4g: The title compound was synthesized according to general procedure **4** with slight modification, phenyl pivaloyloxycarbamate (0.237 g, 1.00 mmol) was formed *in situ* following general procedure **1** and used without purification, to which was added dipropylamine (0.14 mL, 1.1 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 3 h at 120 °C. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.215 g, 88%) over two steps. TLC R*f* = 0.66 in 20% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.17 (s, 1H), 3.19 (t, *J* = 7.7 Hz, 4H), 1.61 (ap sext, *J* = 7.5 Hz, 4H), 1.31 (s, 9H), 0.91 (t, *J* = 7.4, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0 (C), 157.6 (C), 48.9 (CH_2), 38.4 (C), 27.1 (CH_3), 21.4 (CH_2), 11.3 (CH_3). IR (FTIR): 3231, 2964, 2936, 2874, 1773, 1659, 1478, 1241 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{12}H_{24}N_2O_3$ $[M]^+$: 244.1787. Found: 244.1791.

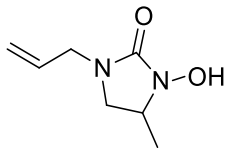


3-(Benzoyloxy)-1,1-dipropylurea 4h: The title compound was synthesized according to general procedure **4** using phenyl *N*-benzoyloxycarbamate **1c** (0.257 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.1 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 3 h at 120 °C. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.230 g, 87%). TLC R*f* = 0.40 in 20% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.57 (br s, 1H), 8.12-8.08 (m, 2H), 7.62-7.56 (m, 1H), 7.44 (tt, *J* = 7.5, 1.3 Hz, 2H), 3.24 (t, *J* = 7.6 Hz, 4H), 1.64 (ap sext, *J* = 7.5 Hz, 4H), 0.92 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2 (C), 157.6 (C), 134.0 (C), 129.9 (CH), 128.6 (CH), 127.3 (C), 49.0 (CH_2), 21.4 (CH_2), 11.3 (CH_3). IR (FTIR): 3213, 2963, 2931, 2874, 1781, 1762, 1647, 1510, 1483, 1378 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{14}H_{20}N_2O_3$ $[M]^+$: 264.1474. Found: 264.1453.

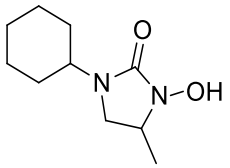


3-((4-Methoxybenzoyl)oxy)-1,1-dipropylurea 4i: The title compound was synthesized according to general procedure **4** using phenyl (4-methoxybenzoyl)oxycarbamate **1p** (0.287 g, 1.00 mmol), and dipropylamine (0.14 mL, 1.1 mmol) and imidazole (0.007 g, 0.1 mmol) in THF (5.0 mL, 0.20 M). The reaction was heated under microwave irradiation for 1 h at 120 °C. Upon completion, the reaction mixture was diluted with EtOAc (5.0 mL) and washed three times using 1 M NaOH (2.0 mL), followed by a single brine wash (2.0 mL). The organic phase was collected and dried over Na_2SO_4 (stirring for 15 min). The solids were filtered and rinsed with CH_2Cl_2 (5.0 mL). The filtrate was collected and concentrated under reduced pressure to yield the title compound as an amorphous white solid (0.280 g, 95%). TLC R*f* = 0.33 in 20% EtOAc/Hexane. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.49 (s, 1H), 8.07-8.04 (m, 2H), 6.93-6.91 (m, 2H), 3.23 (t, *J* = 7.6 Hz, 4H), 1.64 (ap sext, 7.5 Hz, 4H), 0.92 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0 (C), 164.3 (C), 157.8 (C), 132.2 (CH), 119.5 (C), 114.1 (CH), 55.6 (CH_3), 49.1 (CH_2), 21.6 (CH_2), 11.4 (CH_3). IR (FTIR): 3218, 2963, 2933, 2874, 1748, 1666, 1604, 1509, 1463, 1238, 1165 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{15}H_{22}N_2O_4$ $[M]^+$: 294.1580. Found: 294.1595.

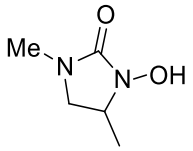
Cascade *O*-Isocyanate Substitution and Cope-type Hydroamination of Hydroxy-Carbamates (**Scheme 5**)**:**



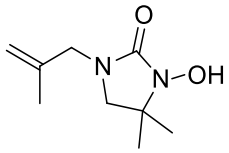
1-Allyl-3-hydroxy-4-methylimidazolidin-2-one 5a: The title compound was synthesized according to general procedure **5** using phenyl *N*-hydroxycarbamate **1a** (0.061 g, 0.400 mmol), diallylamine (0.039 g, 0.400 mmol), and trifluoromethanesulfonamide (0.030 g, 0.200 mmol) in EtOAc (2.0 mL, 0.20 M). The vial was sealed with a microwave cap, purged, then heated for 1 h at 175 °C via microwave irradiation. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (50% EtOAc/Hexane then 100% EtOAc). The title compound was obtained as an amorphous white solid (0.039 g, 62%). TLC R*f* = 0.57 in 100% EtOAc. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (br s, 1H), 5.72 (ddt, *J* = 17.6, 9.8, 6.1 Hz, 1H), 5.23-5.18 (m, 2H), 3.91 (ddt, *J* = 15.4, 5.9, 1.5 Hz, 1H), 3.72 (ddt, *J* = 15.4, 6.3, 1.3 Hz, 1H), 3.59 (ddt, *J* = 9.5, 7.7, 6.1 Hz, 1H), 3.35 (dd, *J* = 8.7, 7.7 Hz, 1H), 2.82 (dd, *J* = 9.5, 8.7 Hz, 1H), 1.33 (d, *J* = 6.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2 (C), 132.2 (CH), 118.4 (CH_2), 55.4 (CH), 47.8 (CH_2), 46.7 (CH_2), 16.7 (CH_3). IR (FTIR): 3198, 2973, 2898, 1690, 1490, 1449, 1258, 977 cm^{-1}. HRMS (EI): Exact mass calcd for $C_7H_{12}N_2O_2$ $[M]^+$: 156.0899. Found: 156.0918.



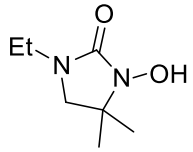
1-Cyclohexyl-3-hydroxy-4-methylimidazolidin-2-one 5b: The title compound was synthesized according to general procedure **5** using phenyl *N*-hydroxycarbamate **1a** (0.061 g, 0.400 mmol), *N*-cyclohexylallylamine (0.056 g, 0.400 mmol), and trifluoromethanesulfonamide (0.030 g, 0.200 mmol) in EtOAc (2.0 mL, 0.20 M). The vial was sealed with a microwave cap, purged, then heated for 20 min at 175 °C via microwave irradiation. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (65% EtOAc/Hexane then 75% EtOAc/Hexane). The title compound was obtained as an amorphous white solid (0.039 g, 49%). TLC R*f* = 0.34 in 75% EtOAc/Hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (br s, 1H), 3.72 (tt, *J* = 11.6, 3.8 Hz, 1H), 3.51 (ddq, *J* = 9.5, 7.7, 6.1 Hz, 1H), 3.33 (dd, *J* = 8.5, 7.7 Hz, 1H), 2.81 (dd, *J* = 9.5, 8.5 Hz, 1H), 1.81-1.64 (m, 5H), 1.39-1.24 (m, 7H), 1.12-1.01 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8 (C), 55.9 (CH), 51.6 (CH), 44.0 (CH_2), 30.2 (CH_2), 29.8 (CH_2), 25.4 (CH_2), 25.4 (CH_2), 25.3 (CH_2), 16.7 (CH_3). IR (FTIR): 3189, 2924, 2853, 1690, 1484, 1436, 1254 cm^{-1}. HRMS (EI): Exact mass calcd for $C_{10}H_{18}N_2O_2$ $[M]^+$: 198.1368. Found: 198.1348.



3-Hydroxy-1,4-dimethylimidazolidin-2-one 5c: The title compound was synthesized according to general procedure **5** using phenyl *N*-hydroxycarbamate **1a** (0.061 g, 0.400 mmol), *N*-methylallylamine (0.028 g, 0.400 mmol), and trifluoromethanesulfonamide (0.030 g, 0.200 mmol) in EtOAc (2.0 mL, 0.20 M). The vial was sealed with a microwave cap, purged, then heated for 2.5 h at 175 °C via microwave irradiation. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (65% EtOAc/Hexane then 100% EtOAc). The title compound was obtained as an amorphous white solid (0.020 g, 38%). TLC R*f* = 0.12 in 100% EtOAc. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (br s, 1H), 3.58 (ddq, *J* = 9.5, 7.7, 6.1 Hz, 1H), 3.38 (dd, *J* = 8.5, 7.7 Hz, 1H), 2.85 (dd, *J* = 9.5, 8.5 Hz, 1H), 2.82 (s, 3H), 1.30 (d, *J* = 6.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7 (C), 55.3 (CH), 50.5 (CH), 31.1 (CH_3), 16.7 (CH_3). IR (FTIR): 3196, 2906, 1694, 1497, 1404, 1261, 1015 cm^{-1}. HRMS (EI): Exact mass calcd for $C_5H_{10}N_2O_2$ $[M]^+$: 130.0742. Found: 130.0758.



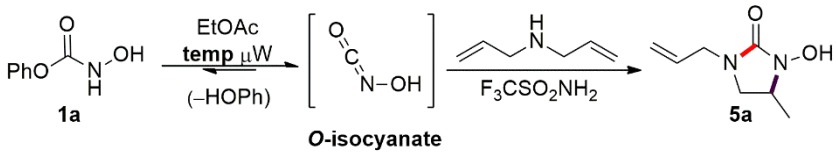
3-Hydroxy-4,4-dimethyl-1-(2-methylallyl)imidazolidin-2-one 5d: The title compound was synthesized according to general procedure **5** using phenyl *N*-hydroxycarbamate **1a** (0.061 g, 0.400 mmol), bis(2-methyl)allylamine (0.050 g, 0.400 mmol), and trifluoromethanesulfonamide (0.030 g, 0.200 mmol) in EtOAc (2.0 mL, 0.20 M). The vial was sealed with a microwave cap, purged, then heated for 1 h at 175 °C via microwave irradiation. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (60% EtOAc/Hexane). The title compound was obtained as an amorphous white solid (0.054 g, 72%). TLC R*f* = 0.62 in 100% EtOAc. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (br s, 1H), 4.90-4.84 (m, 2H), 3.75 (s, 2H), 2.95 (s, 2H), 1.69 (s, 3H), 1.30 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (C), 140.2 (C), 113.4 (CH_2), 59.4 (C), 53.9 (CH_2), 50.1 (CH_2), 22.7 (CH_3), 19.9 (CH_3). IR (FTIR): 3199, 2988, 2969, 2916, 2854, 1686, 1482, 1317, 948 cm^{-1}. HRMS (EI): Exact mass calcd for $C_9H_{16}N_2O_2$ $[M]^+$: 184.1212. Found: 184.1187.



1-Ethyl-3-hydroxy-4,4-dimethylimidazolidin-2-one 5e: The title compound was synthesized according to general procedure **5** using phenyl *N*-hydroxycarbamate **1a** (0.061 g, 0.400 mmol), *N*-ethyl(2-methyl)allylamine (0.040 g, 0.400 mmol), and trifluoromethanesulfonamide (0.030 g, 0.200 mmol) in EtOAc (2.0 mL, 0.20 M). The vial was sealed with a microwave cap, purged, then heated for 1 h at 175 °C via microwave

irradiation. The reaction mixture was concentrated under reduced pressure and isolated using flash chromatography (60% EtOAc/Hexane then 100% EtOAc). The title compound was obtained as an amorphous white solid (0.039 g, 62%). TLC R*f* = 0.43 in 100% EtOAc. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (br s, 1H), 3.27 (q, *J* = 7.3 Hz, 2H), 3.03 (t, *J* = 7.3 Hz, 2H), 1.30 (s, 1H), 1.10 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.7 (C), 59.5 (C), 53.6 (CH_2), 38.4 (CH_2), 22.4 (CH_3), 12.3 (CH_3). IR (FTIR): 3178, 2970, 2930, 2856, 1694, 1451, 1441, 1236, 1089, 796 cm^{-1}. HRMS (EI): Exact mass calcd for $C_7H_{14}N_2O_2$ $[M]^+$: 158.1055. Found: 158.1076.

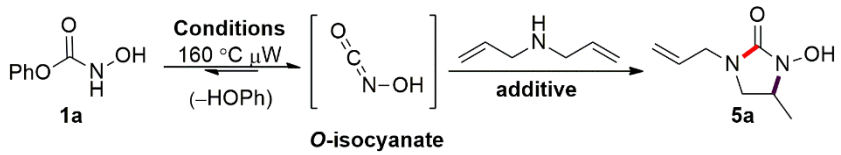
Table S1: Temperature requirements for hydroamination



Entry	Temp (°C)	PhOH formation (%)	^{1}H NMR yield (%)[b]
1	75	25	0
2	100	51	0
3	125	86	trace
4	150	100	28
5	**175**	**90 (no SM)**	**60**

Conditions: Phenyl *N*-hydroxycarbamate **1a** (1.0 equiv.), diallylamine (1.0 equiv.), $TfNH_2$ (0.5 equiv.) in EtOAc (0.2M), 1 h.

Table S2: Additive Survey for Cascade Substitution-Hydroamination Reaction

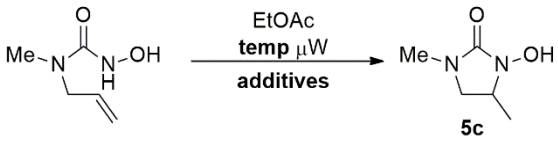


Entry	Conditions	Additive (equiv.)	^{1}H NMR yield vs. PhOH (%)
1	A	-	66
2	A	*i*Pr_2NEt (0.05)	63
3	A	DBU (0.25)	6
4	A	DBU (1.0)	0
5	B	$NaCNBH_3$ (1.0)	4
6	A	Diphenylurea (1.0)	17
7	A	Tf_2NH (1.0)	18
8	A	$TsNH_2$ (1.0)	66
9	A	Nitroaniline (1.0)	64
10	A	$TfNH_2$ (1.0)	77
11	A	$TfNH_2$ (0.1)	71
12	A	$TfNH_2$ (0.25)	73
13	**A**	**$TfNH_2$ (0.5)**	**78**
14	A	$TfNH_2$ (1.5)	70
15	A	$TfNH_2$ (2.0)	65

Conditions A: Phenyl *N*-hydroxycarbamate **1a** (1.0 equiv.), diallylamine (1.0 equiv.) in EtOAc (0.2M), 160 °C, 1 h.

Conditions B: Phenyl *N*-hydroxycarbamate **1a** (1.0 equiv.), diallylamine (1.0 equiv.) in *i*PrOH (0.2M), 160 °C, 1 h.

Table S3: Non-cascade hydroamination optimization



Entry	Additive (equiv.)	temp (°C)	Time (h)	^{1}H NMR yield (%)
1	-	175	1	32
2	PhOH (1.0)	175	1	41
3	$TfNH_2$ (0.5)	175	1	40
4	PhOH (1.0), $TfNH_2$ (0.5)	175	1	36
5	PhOH (1.0), $TfNH_2$ (0.5)	150	2	34
6	PhOH (1.0), $TfNH_2$ (0.5)	175	2	48

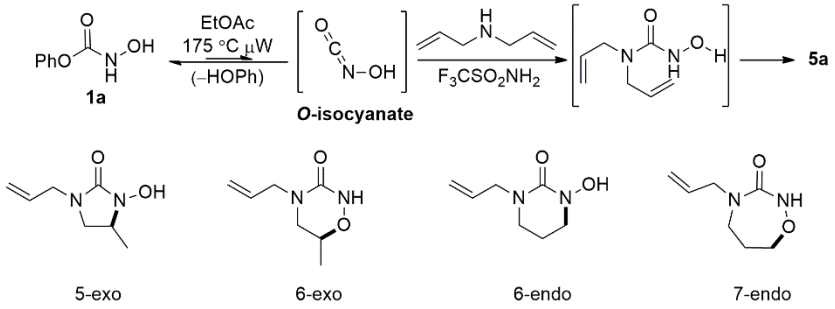
Conditions: Hydroxyurea (1.0 equiv.) in EtOAc (0.2M).

(Isolated yield for cascade hydroamination is 38% after 2.5 h)

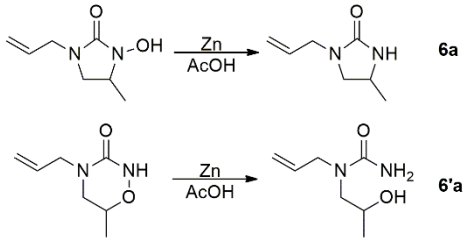
Structural Assignment of Cyclic *N*-Hydroxyureas

The structural assignment of the cyclic products obtained from the cascade substitution and hydroamination of hydroxy-carbamate *O*-isocyanate precursors was completed as follows.

From the given reaction of hydroxy-carbamate **1a**, there are 4 possible cyclic urea products that correspond the to found accurate mass of **5a** (Found: 156.0918); 5-exo, 6-exo, 6-endo, and 7-endo.



The ^{1}H NMR of **5a** definitively removes the 6-endo and 7-endo cyclizations as possible products. The 5-exo and 6-exo are more structurally similar, so further derivatization was performed. The cleavage of the N-O bond using zinc dust and AcOH was used to conclusively determine the product.



The ^{1}H NMR, ^{13}C NMR, and HRMS all suggest that **6a** is the product of the reduction of **5a** rather than **6'a**, allowing the conclusion that the 5-exo cyclic hydroxyurea is the product of the cascade substitution and hydroamination of **1a**.

1-allyl-4-methylimidazolidin-2-one (**6a**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.76 (ddt, J = 17.1, 10.1, 6.1, 1H), 5.23-5.15 (m, 2H), 4.66 (br s, 1H), 3.86-3.71 (m, 3H), 3.50 (dd, J = 8.5, 8.5 Hz, 1H), 2.82 (dd, J = 8.7, 6.5 Hz, 1H), 1.24 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7 (C), 133.3 (CH), 117.6 (CH_2), 51.9 (CH), 46.2 (CH_2), 45.6 (CH_2), 21.4 (CH_3). HRMS (EI): Exact mass calcd for $C_7H_{12}N_2O$ $[M]^+$: 140.0950. Found: 140.0975.

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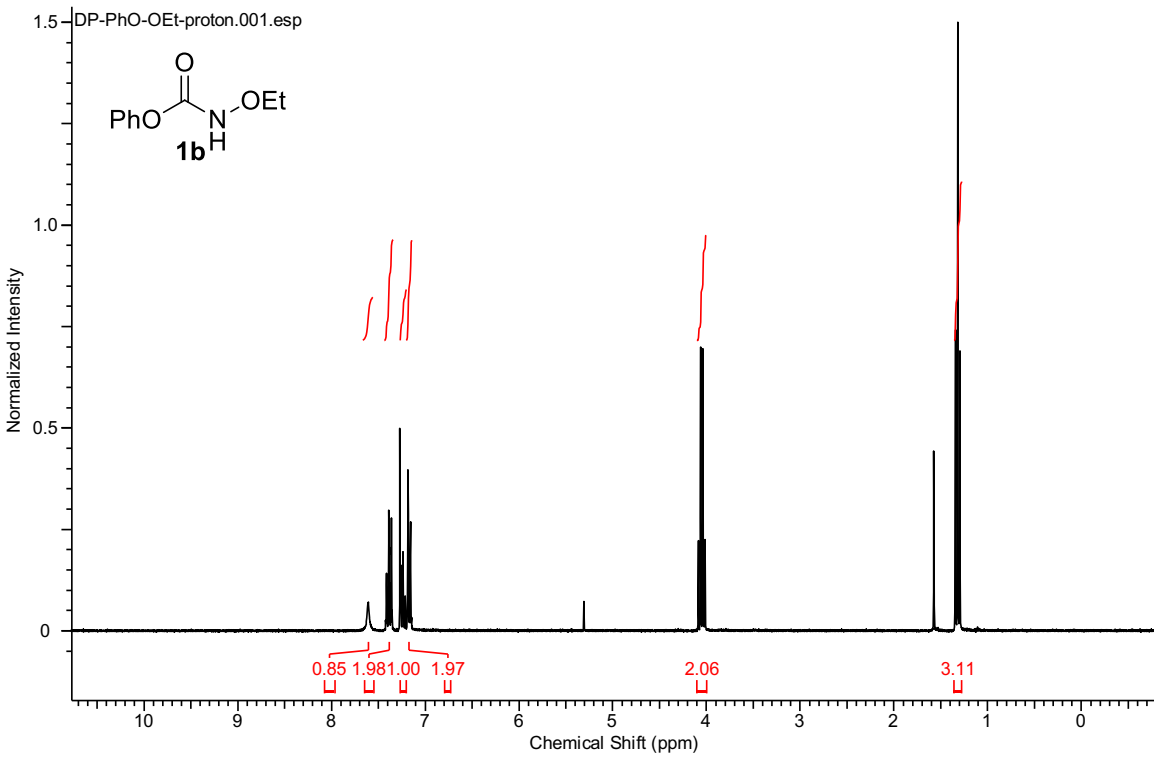
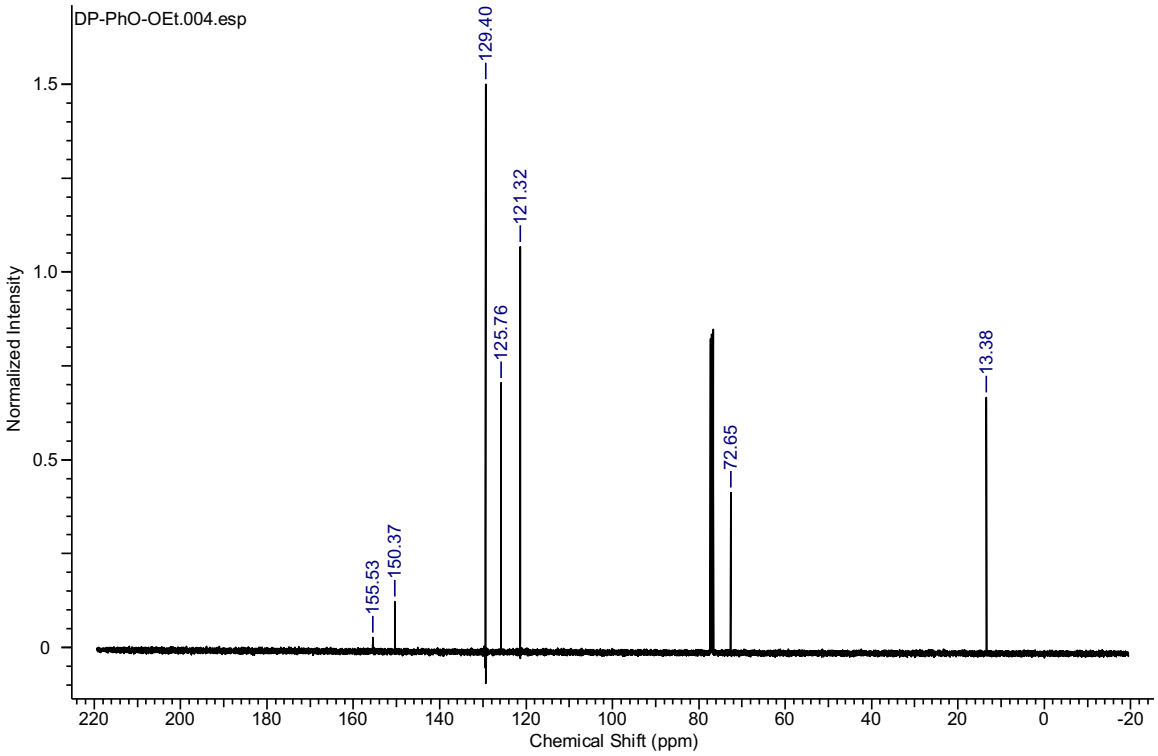
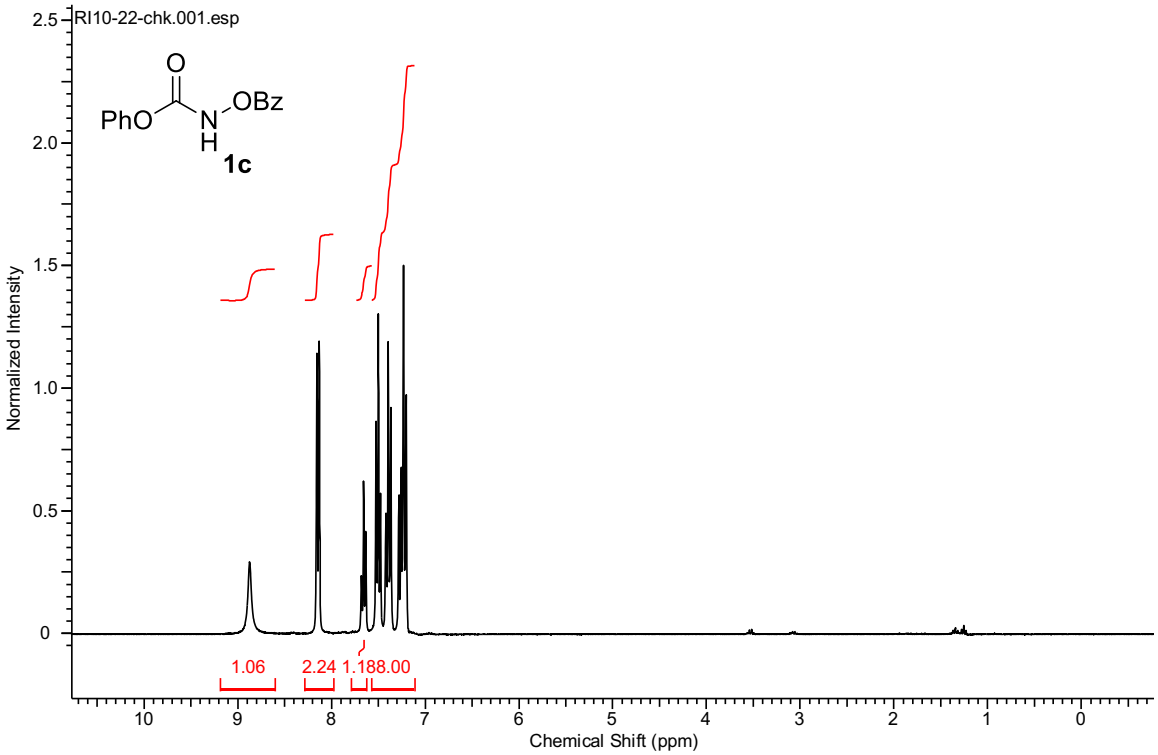
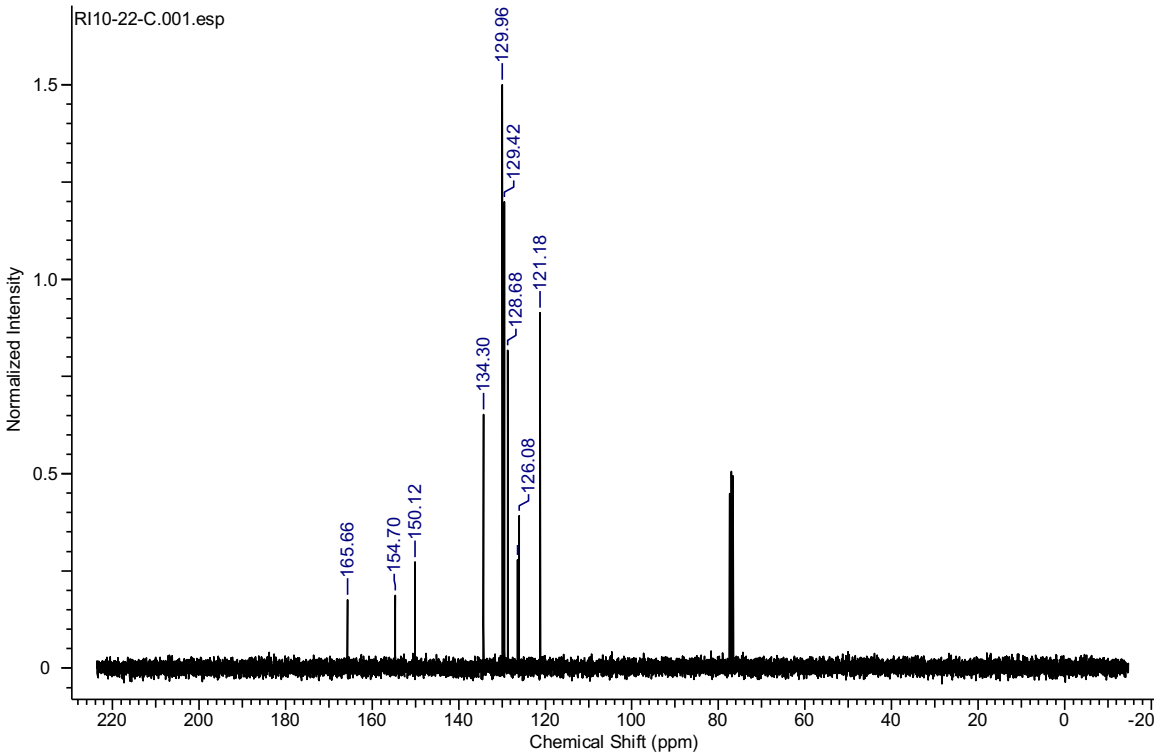
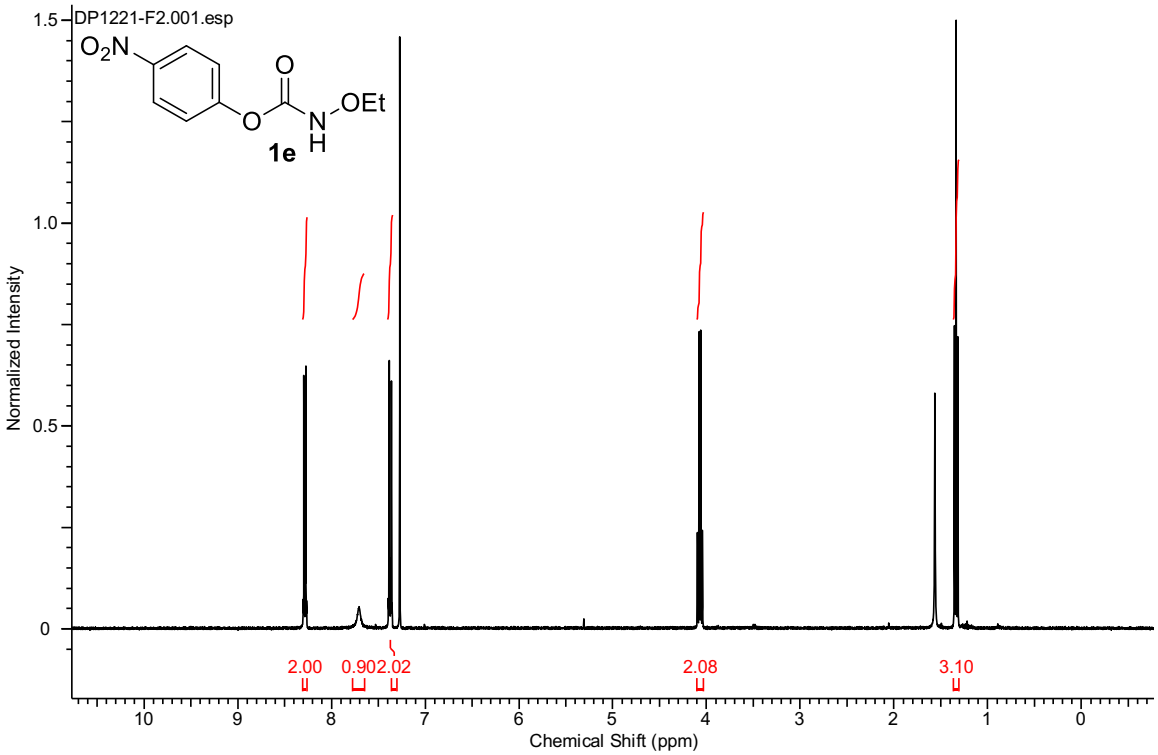
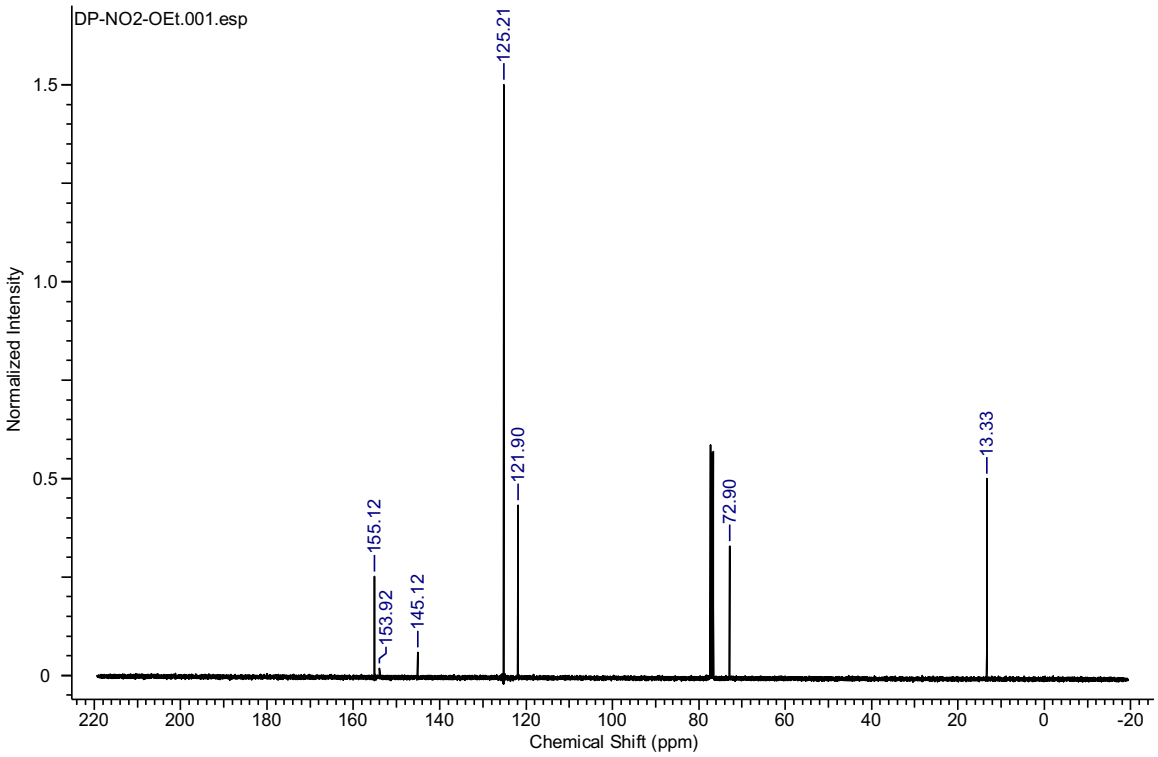

DP-PhO-OEt-proton.001.esp
PhO
O
N
H
OEt
1b
Normalized Intensity
0.85 1.98 1.00 1.97
2.06
3.11
Chemical Shift (ppm)

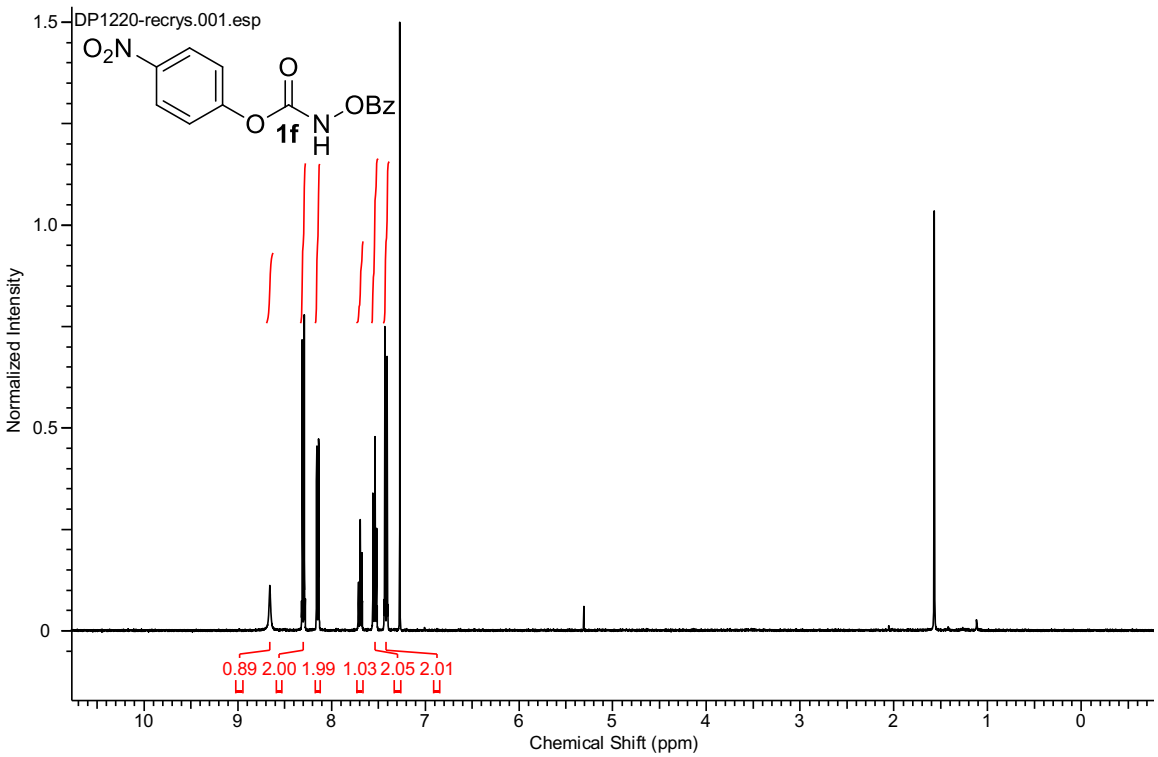

DP-PhO-OEt.004.esp
129.40
121.32
125.76
155.53
150.37
72.65
13.38
Normalized Intensity
Chemical Shift (ppm)

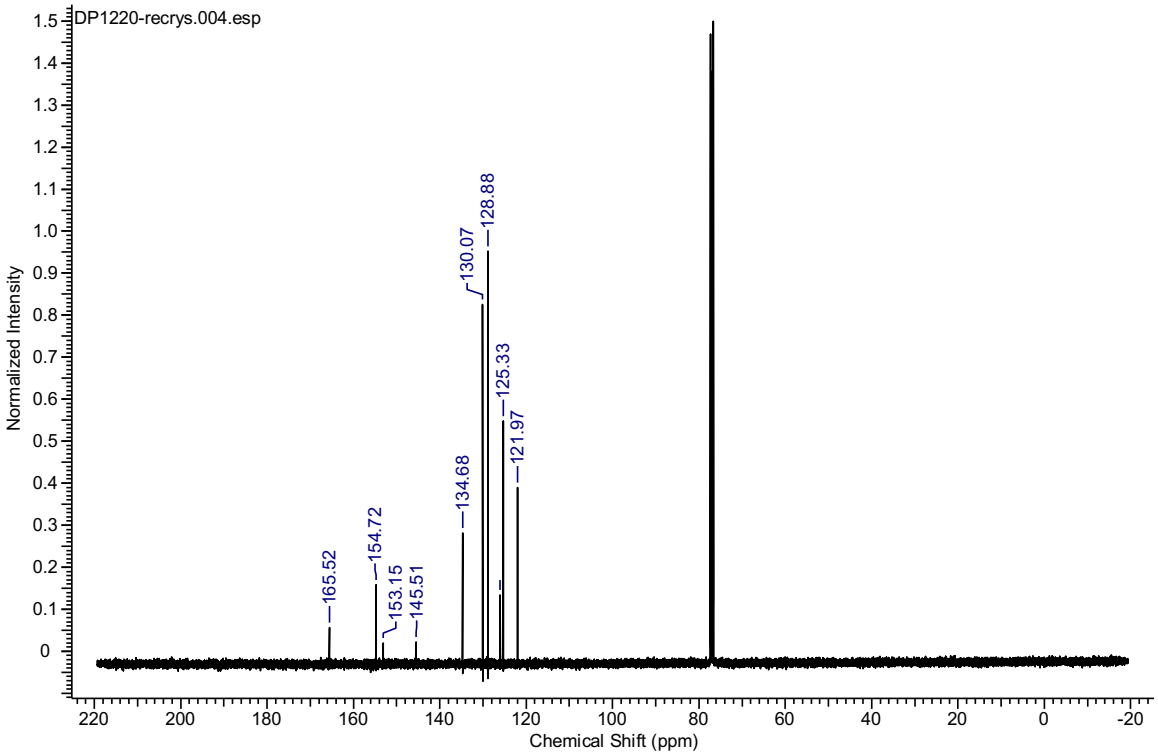

RI10-22-chk.001.esp
O
PhO
N
H
OBz
1c
Normalized Intensity
1.06
2.24
1.18
8.00
Chemical Shift (ppm)

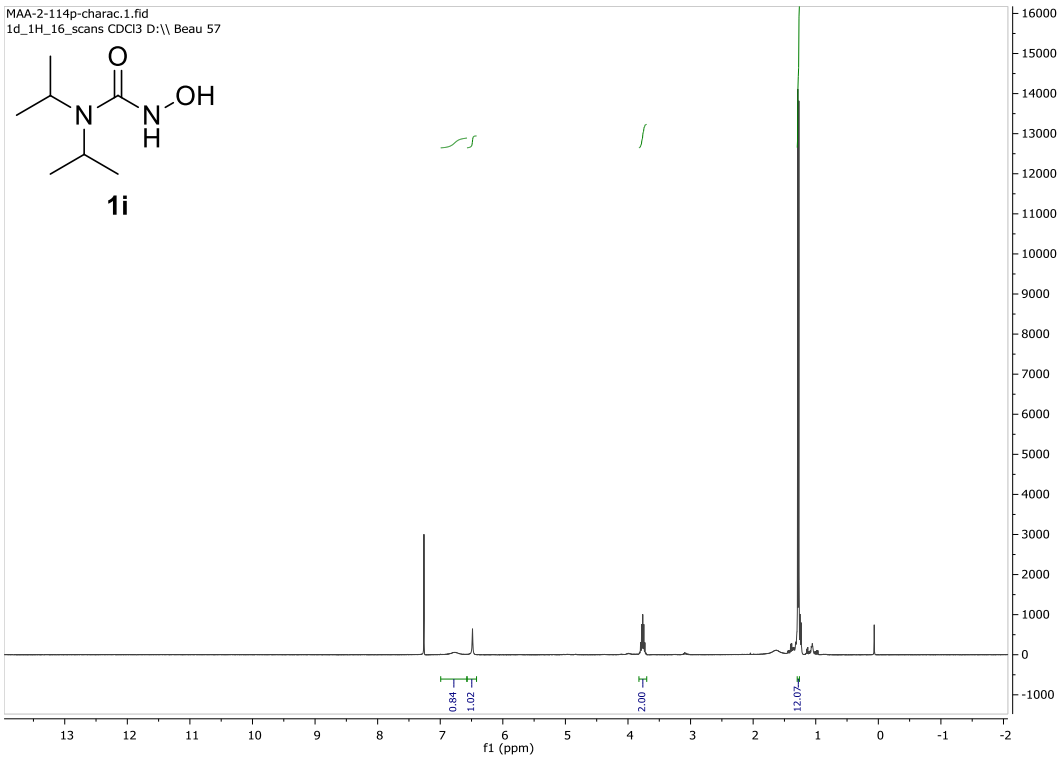

RI10-22-C.001.esp
129.96
129.42
128.68
121.18
134.30
126.08
165.66
154.70
150.12
Normalized Intensity
Chemical Shift (ppm)

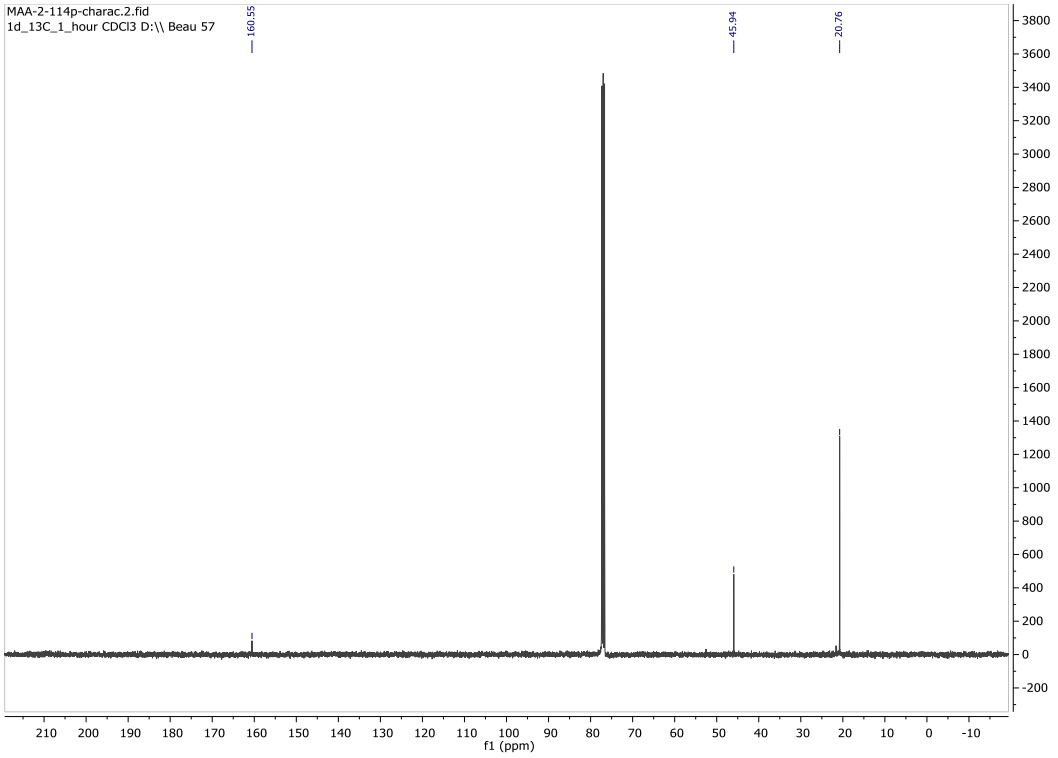

DP1221-F2.001.esp
O_2N
O
O
N
H
OEt
1e
Normalized Intensity
1.5
1.0
0.5
0
2.00
0.90
2.02
2.08
3.10
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

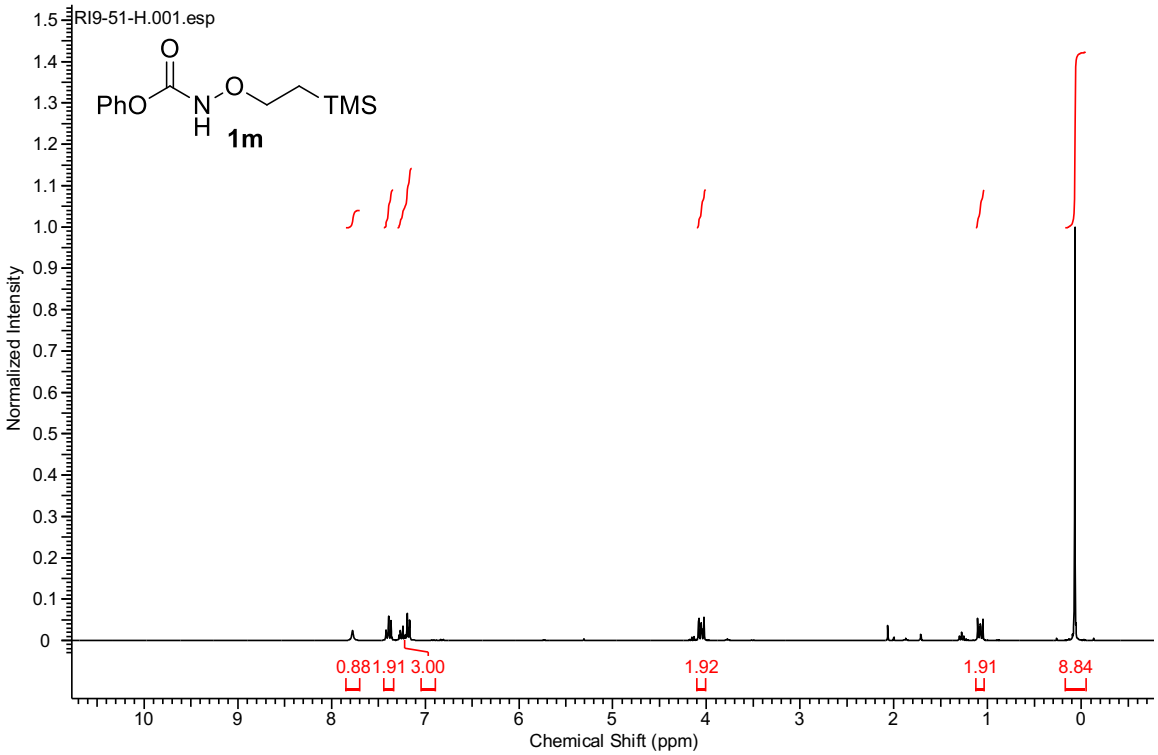

DP-NO2-OEt.001.esp
Normalized Intensity
1.5
1.0
0.5
0
125.21
121.90
155.12
153.92
145.12
72.90
13.33
220
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

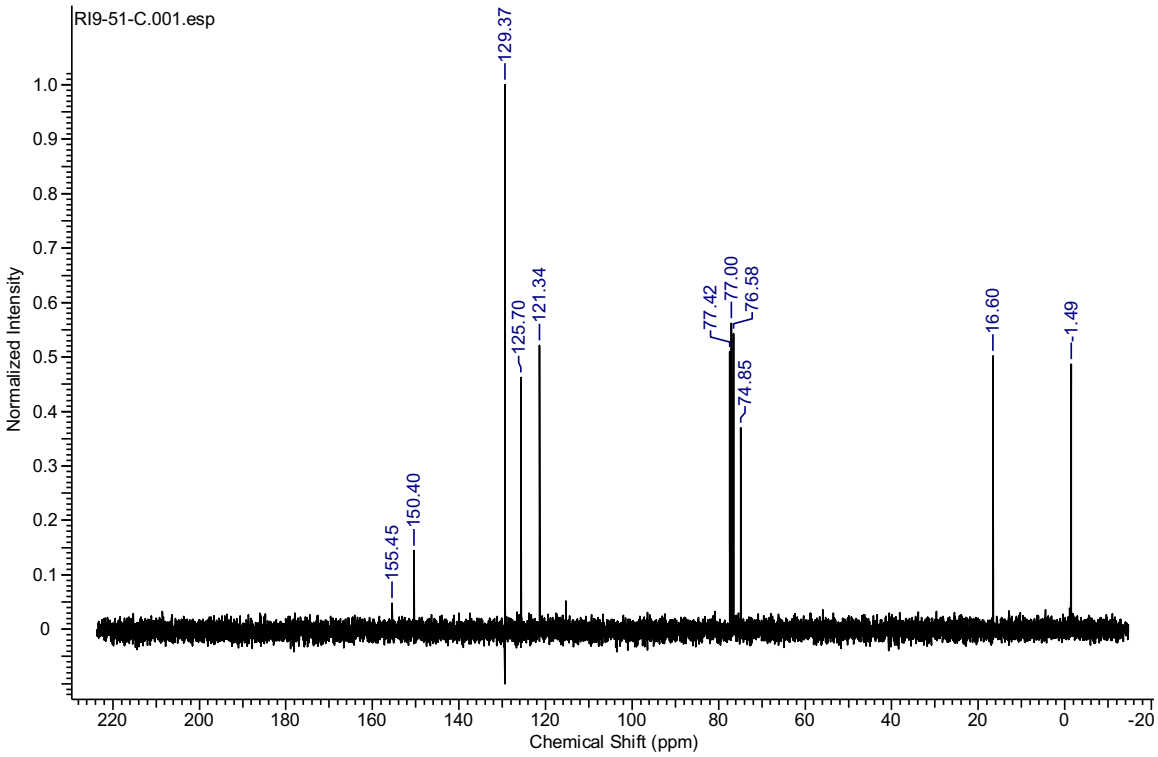

DP1220-recrys.001.esp
O2N
O
O
N
OBz
H
1f
Normalized Intensity
Chemical Shift (ppm)
0.89 2.00 1.99 1.03 2.05 2.01

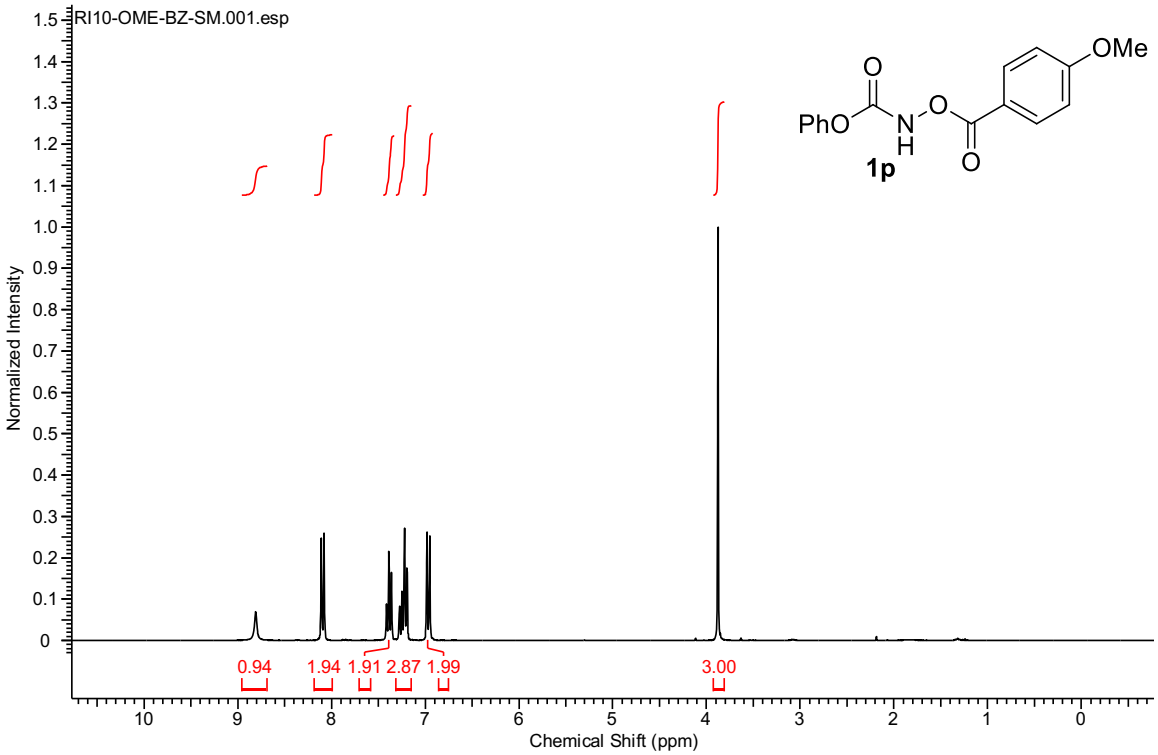

DP1220-recrys.004.esp
Normalized Intensity
Chemical Shift (ppm)
165.52
154.72
153.15
145.51
134.68
130.07
128.88
125.33
121.97

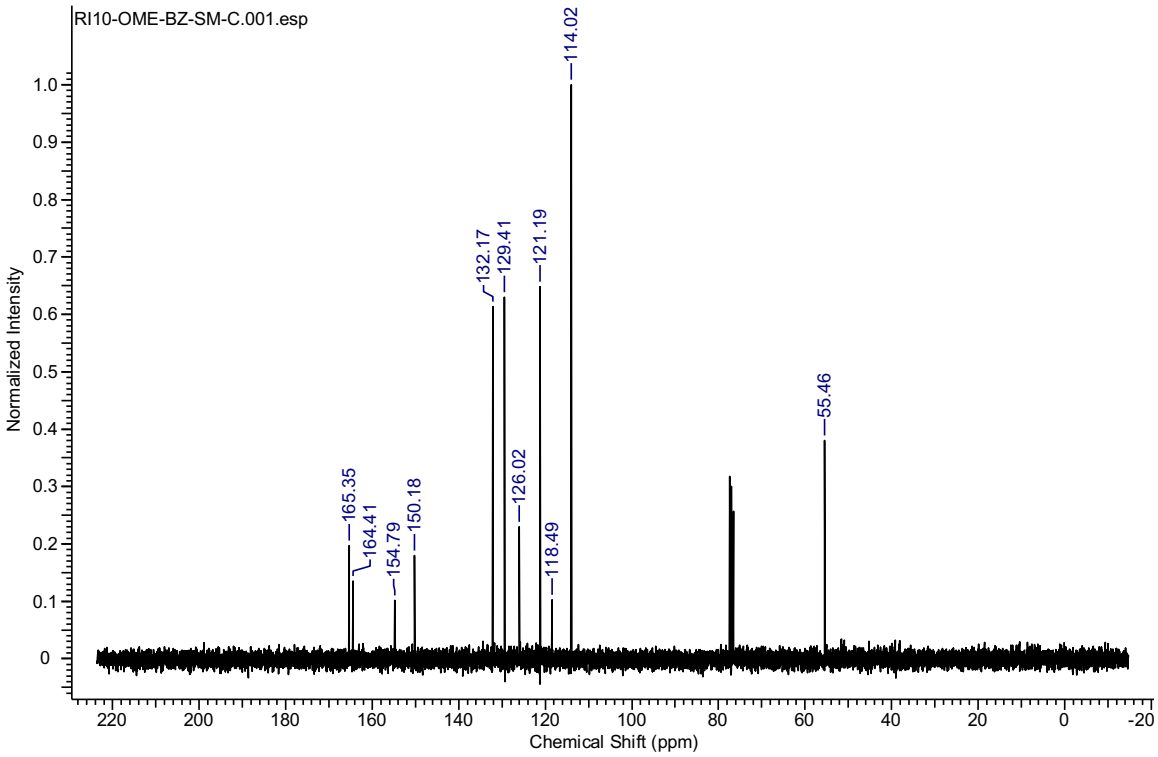

MAA-2-114p-charac.1.fid
1d_1H_16_scans CDCl3 D:\\ Beau 57
O
N
N
OH
H
1i
0.84
1.02
2.00
12.07
f1 (ppm)

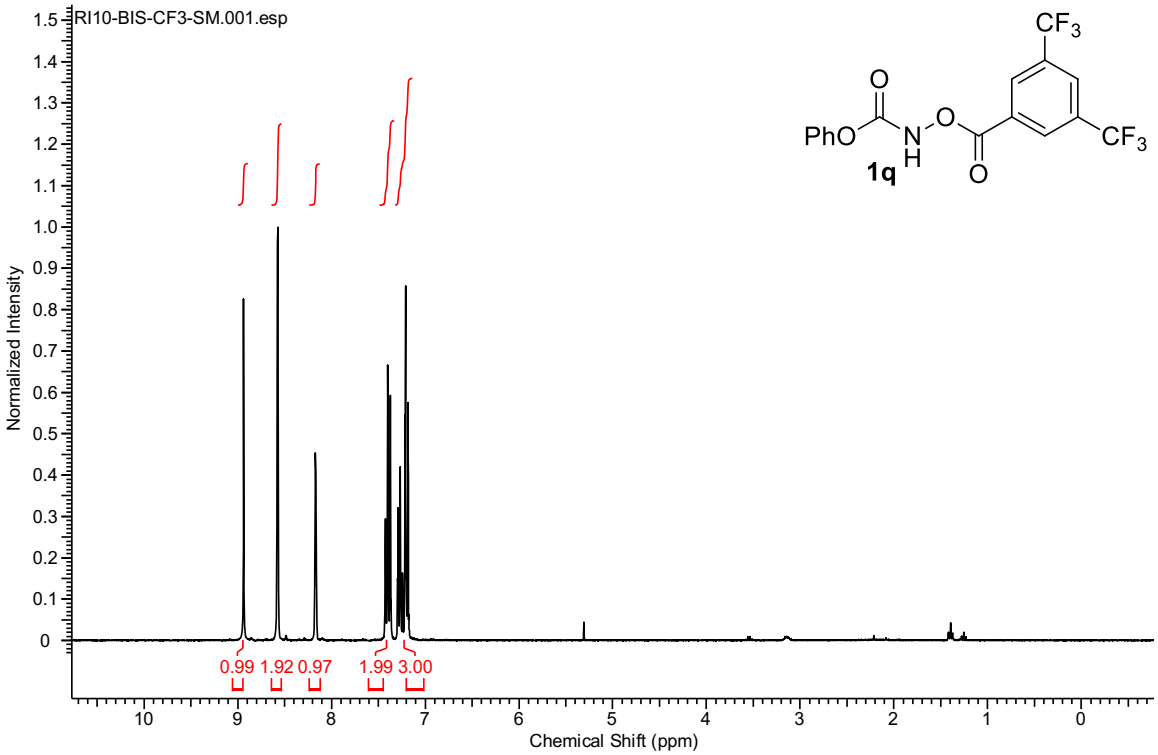

MAA-2-114p-charac.2.fid
1d_13C_1_hour CDCl3 D:\\ Beau 57
160.55
45.94
20.76
f1 (ppm)

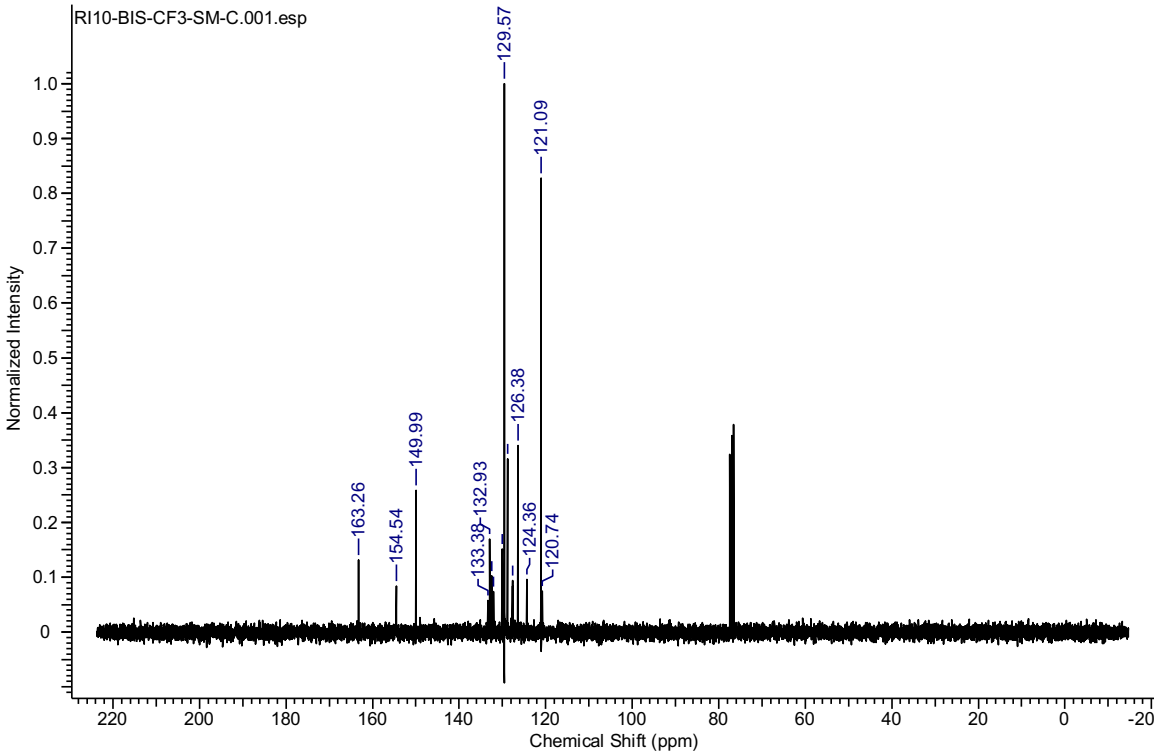

RI9-51-H.001.esp
PhO
O
N
H
O
TMS
1m
Normalized Intensity
0.88
1.91
3.00
1.92
1.91
8.84
Chemical Shift (ppm)

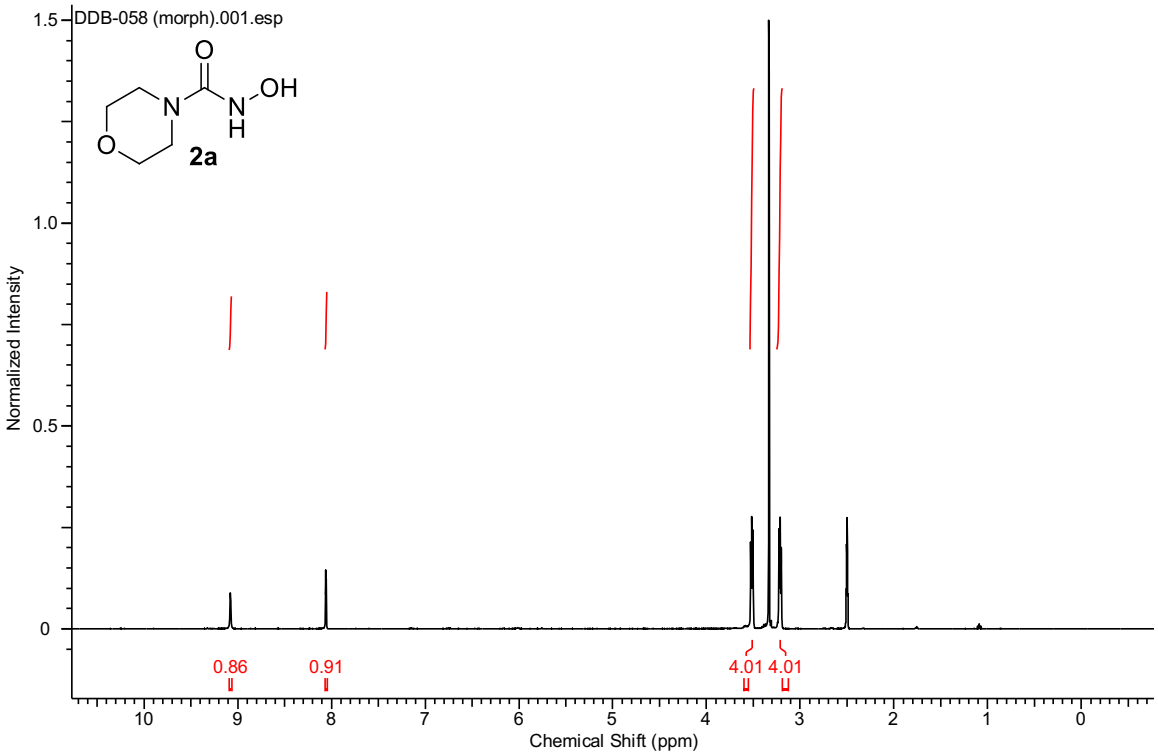

RI9-51-C.001.esp
129.37
125.70
121.34
77.42
77.00
76.58
74.85
155.45
150.40
16.60
1.49
Normalized Intensity
Chemical Shift (ppm)

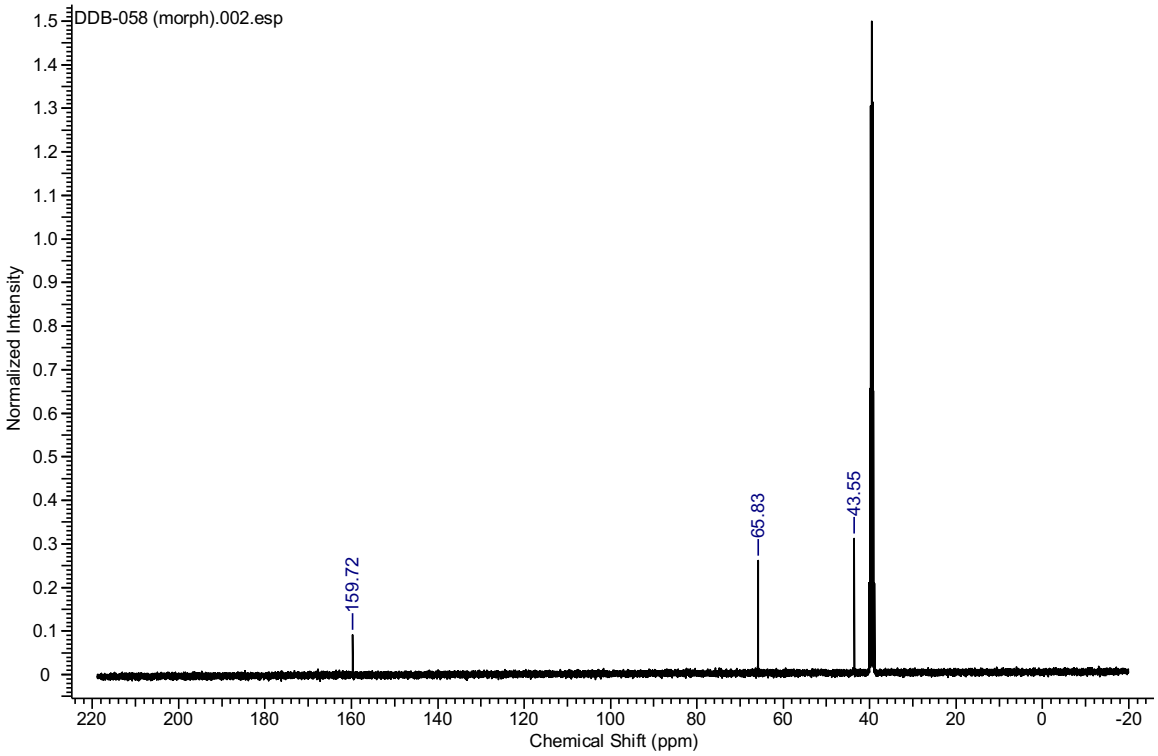

RI10-OME-BZ-SM.001.esp
Normalized Intensity
Chemical Shift (ppm)
0.94
1.94
1.91
2.87
1.99
3.00
PhO
N
H
O
O
O
OMe
1p

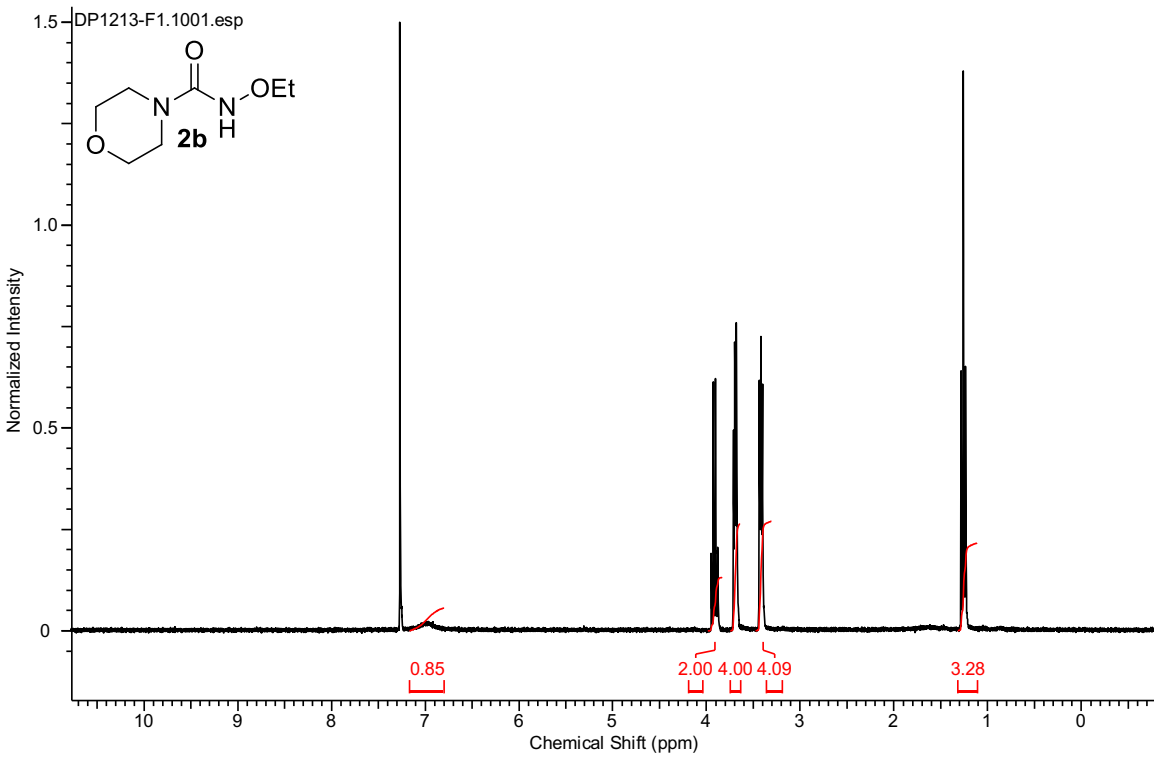

RI10-OME-BZ-SM-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
165.35
164.41
154.79
150.18
132.17
129.41
126.02
121.19
118.49
114.02
55.46

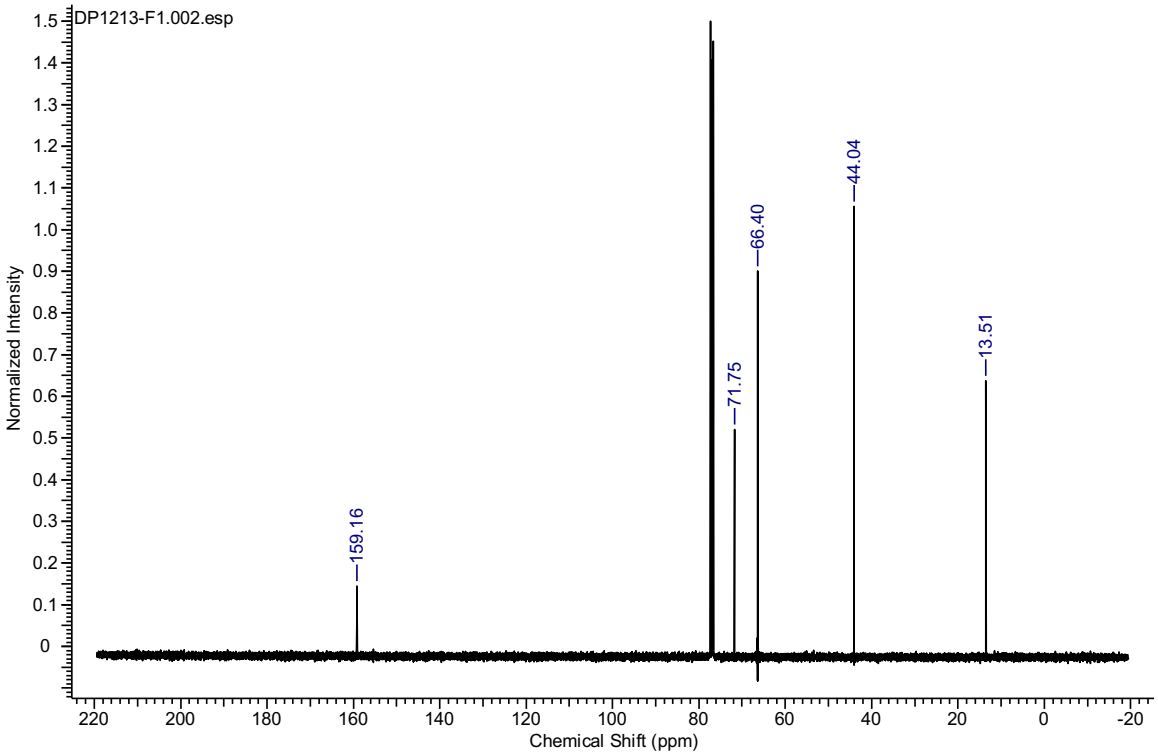

RI10-BIS-CF3-SM.001.esp
Normalized Intensity
Chemical Shift (ppm)
0.99 1.92 0.97 1.99 3.00
PhO
O
N
H
O
O
CF3
CF3
1q

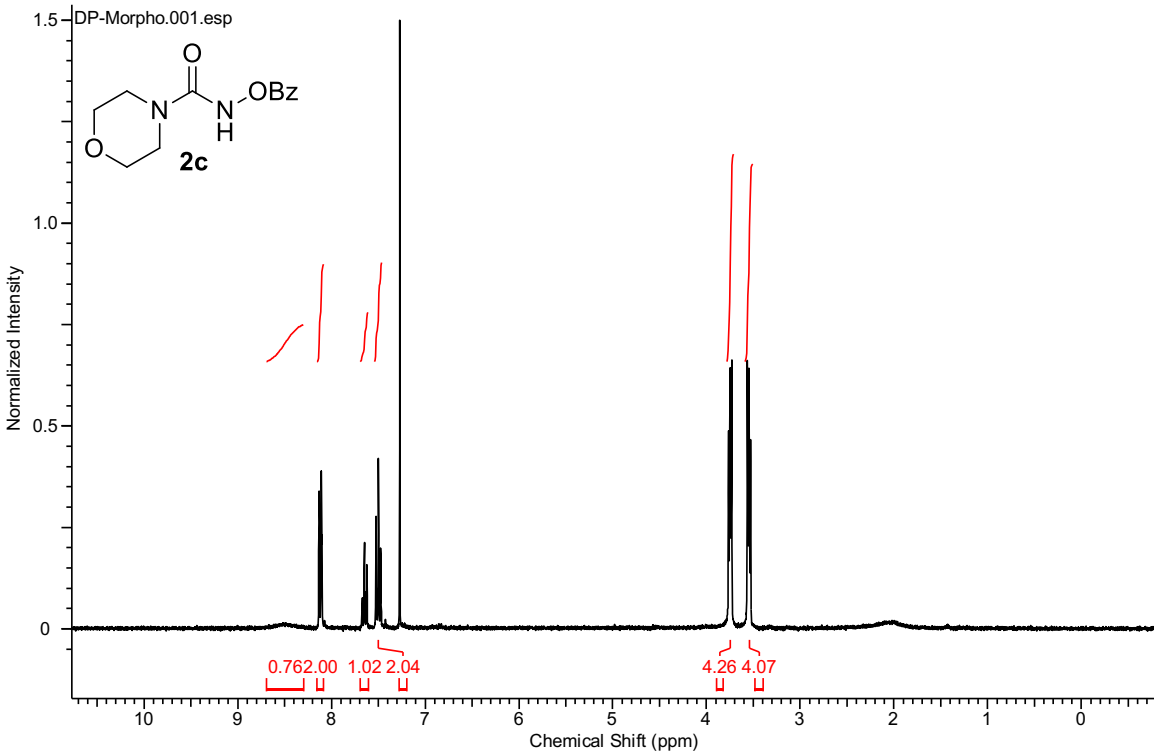

RI10-BIS-CF3-SM-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
163.26
154.54
149.99
133.38
132.93
129.57
126.38
124.36
121.09
120.74

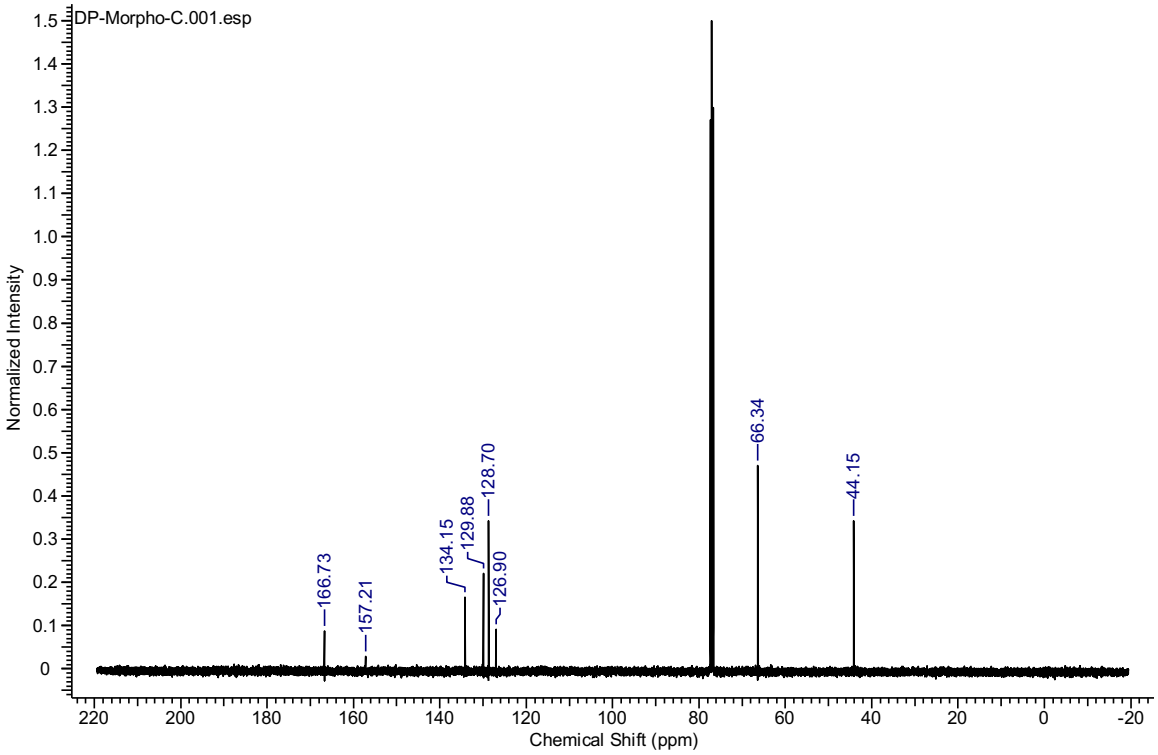

DDB-058 (morph).001.esp
O
N
N
OH
H
O
2a
Normalized Intensity
0.86
0.91
4.01
4.01
Chemical Shift (ppm)

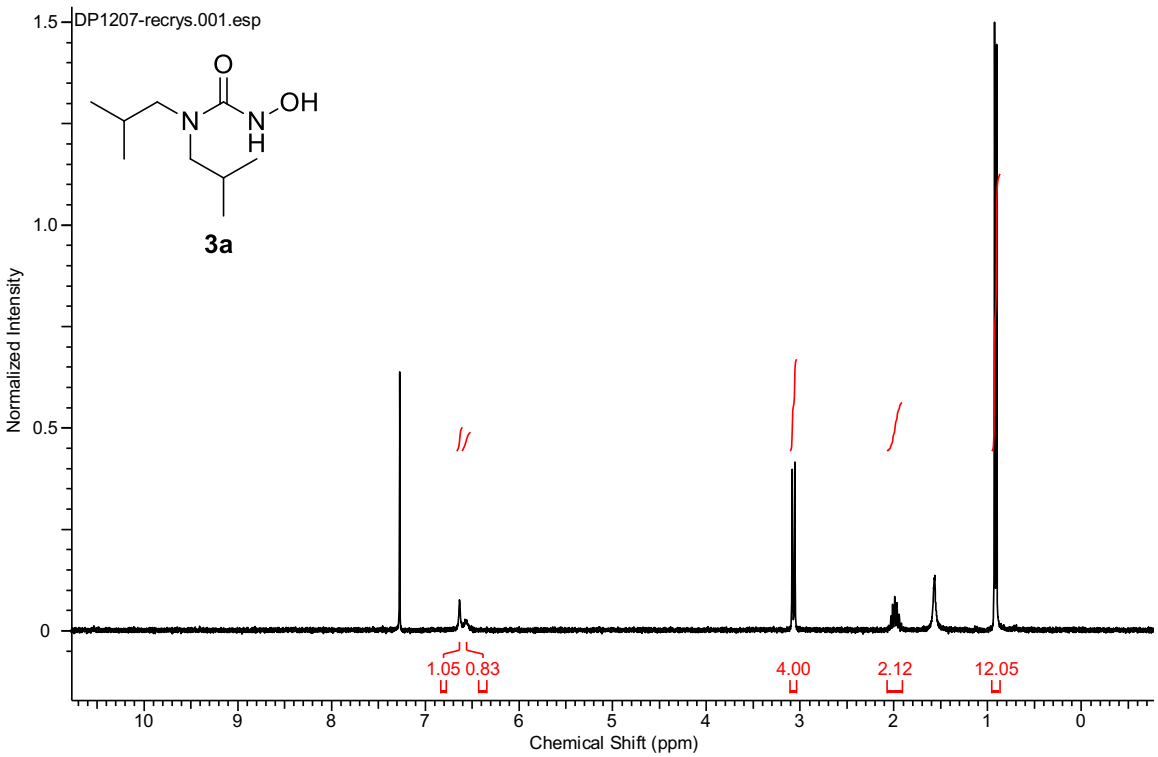

DDB-058 (morph).002.esp
Normalized Intensity
159.72
65.83
43.55
Chemical Shift (ppm)

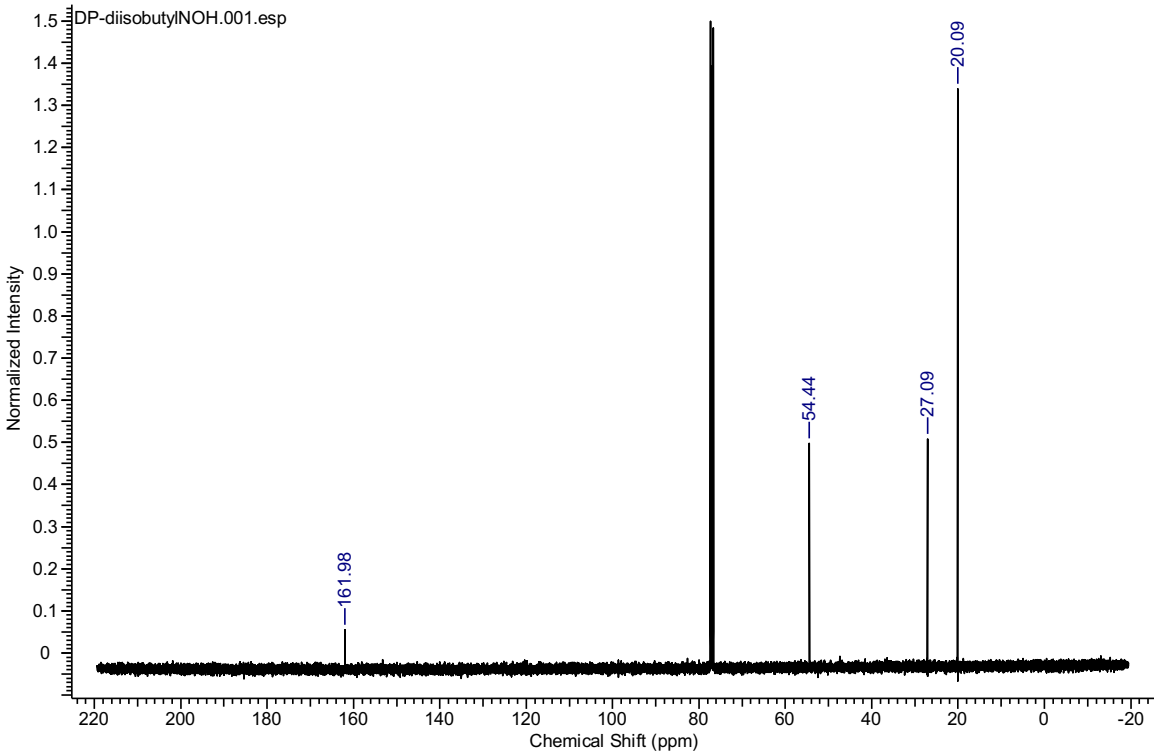

DP1213-F1.1001.esp
O
N
N
OEt
H
O
2b
Normalized Intensity
0.85
2.00
4.00
4.09
3.28
Chemical Shift (ppm)

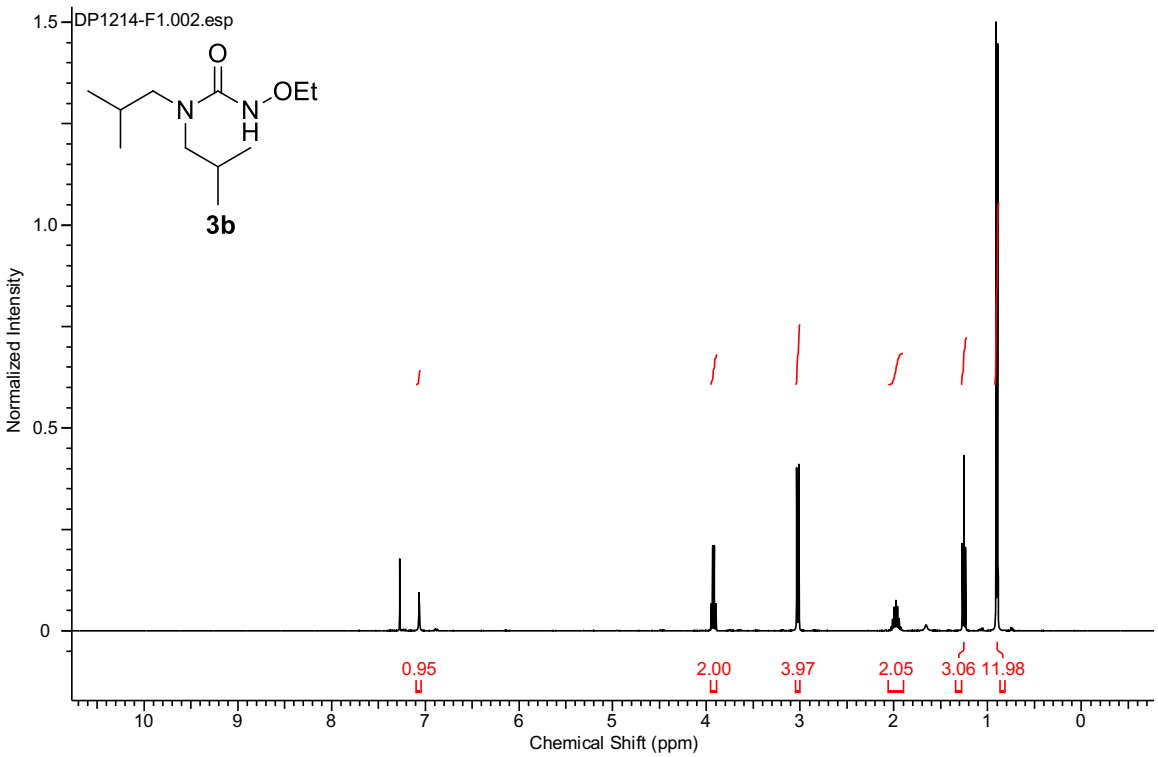

DP1213-F1.002.esp
159.16
71.75
66.40
44.04
13.51
Normalized Intensity
Chemical Shift (ppm)

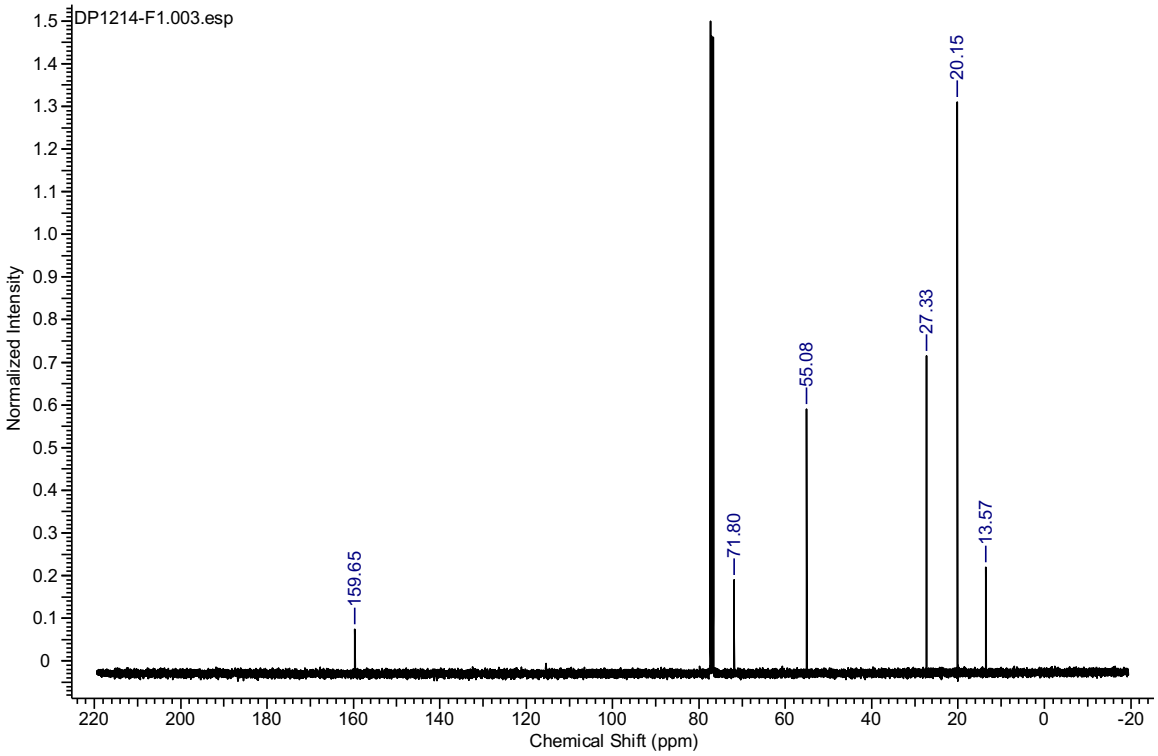

DP-Morpho.001.esp
O
N
O
N
H
OBz
2c
Normalized Intensity
0.76 2.00 1.02 2.04
4.26 4.07
Chemical Shift (ppm)

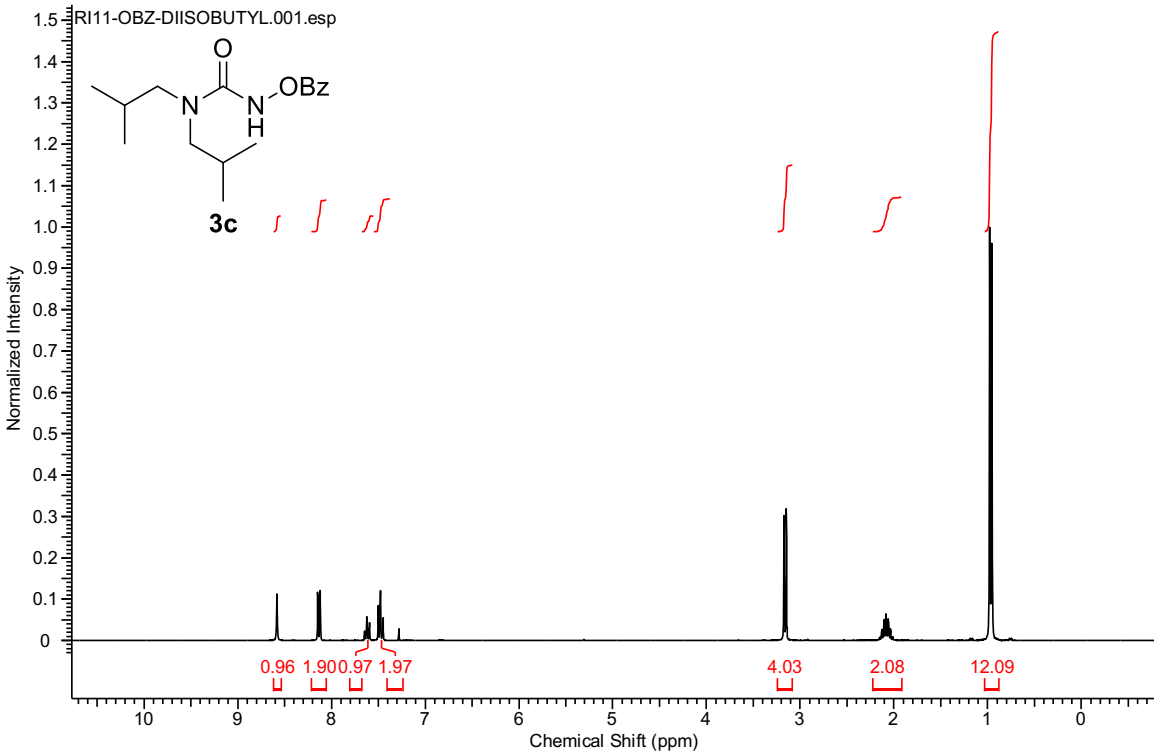

DP-Morpho-C.001.esp
Normalized Intensity
166.73
157.21
134.15
129.88
128.70
126.90
66.34
44.15
Chemical Shift (ppm)

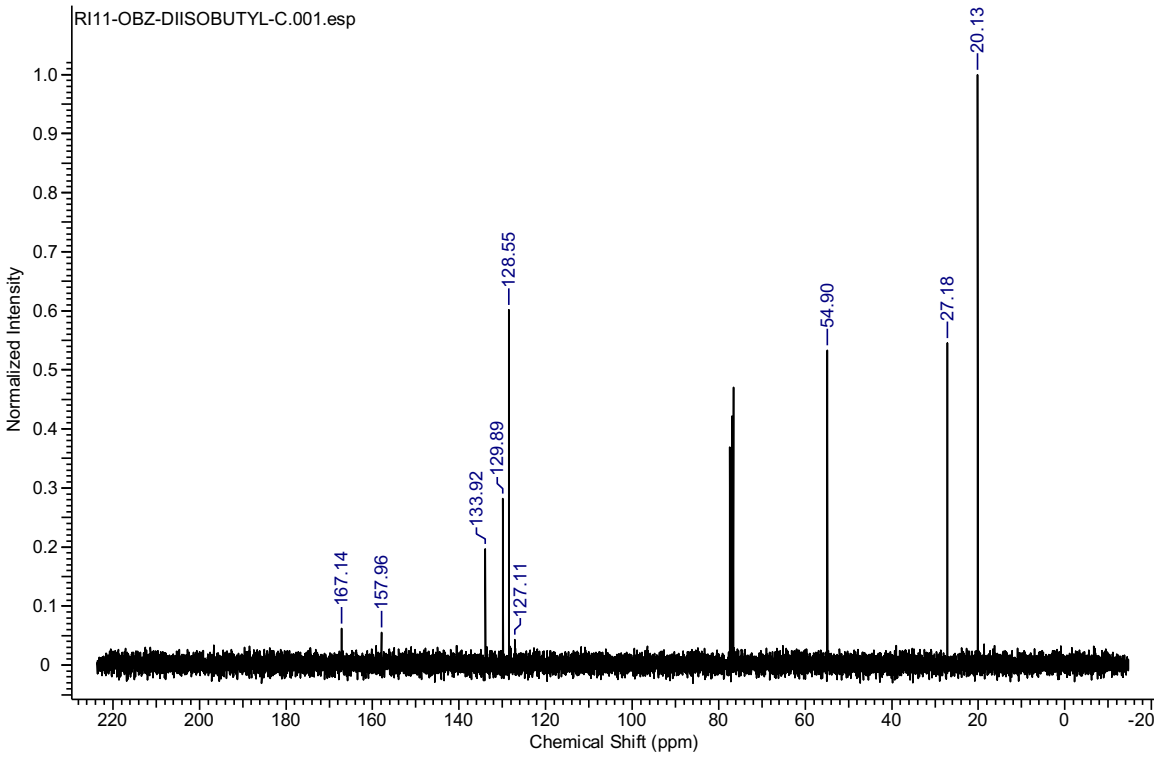

DP1207-recrys.001.esp
O
N
N
OH
H
3a
Normalized Intensity
1.05
0.83
4.00
2.12
12.05
Chemical Shift (ppm)

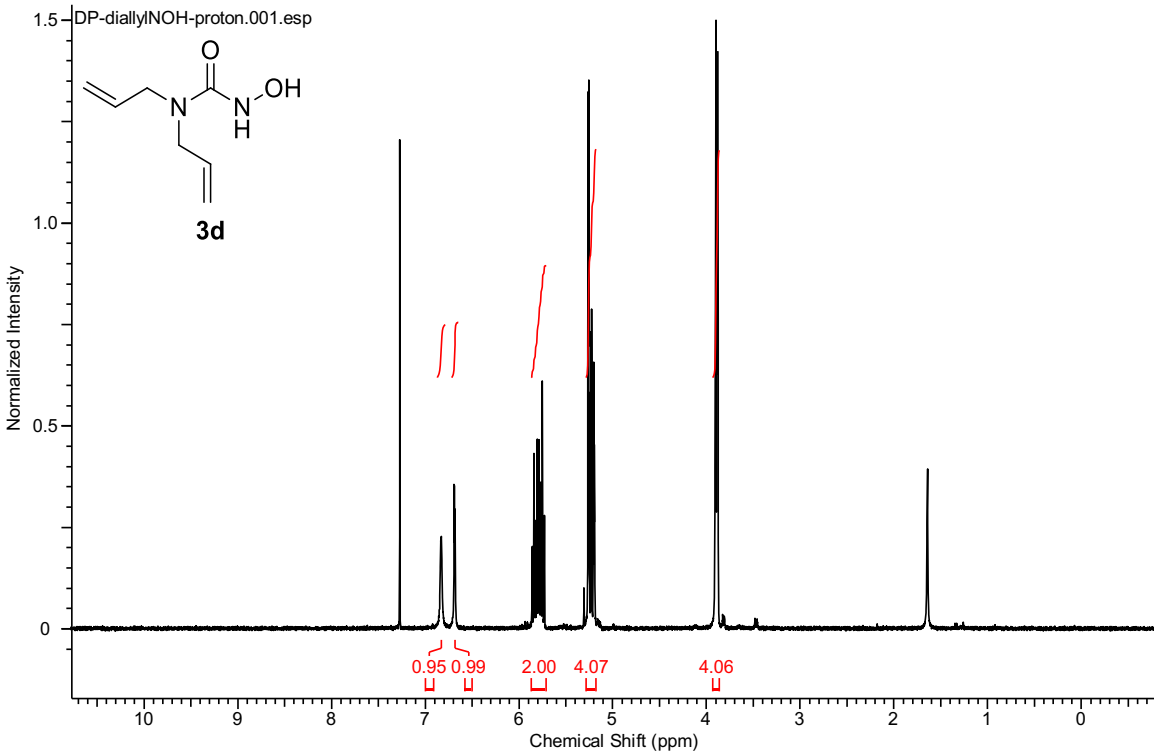

DP-diisobutylNOH.001.esp
161.98
54.44
27.09
20.09
Normalized Intensity
Chemical Shift (ppm)

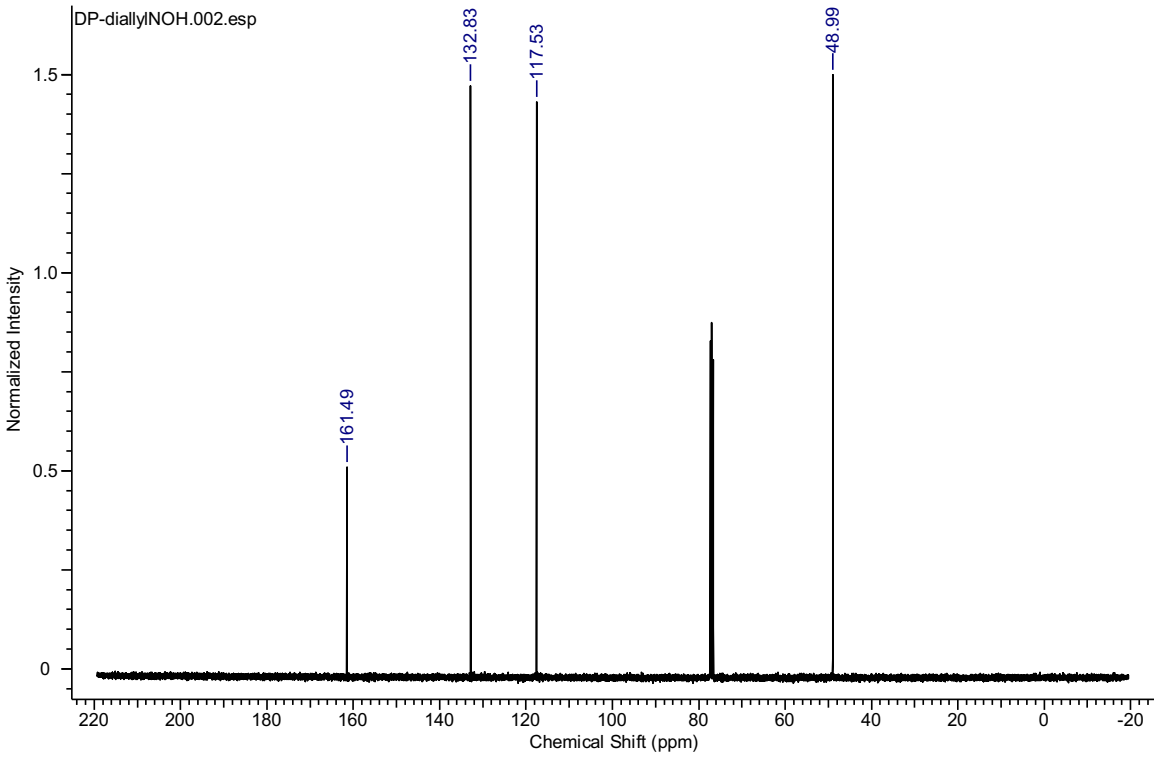

DP1214-F1.002.esp
O
N
N
H
OEt
3b
Normalized Intensity
0.95
2.00
3.97
2.05
3.06
11.98
Chemical Shift (ppm)

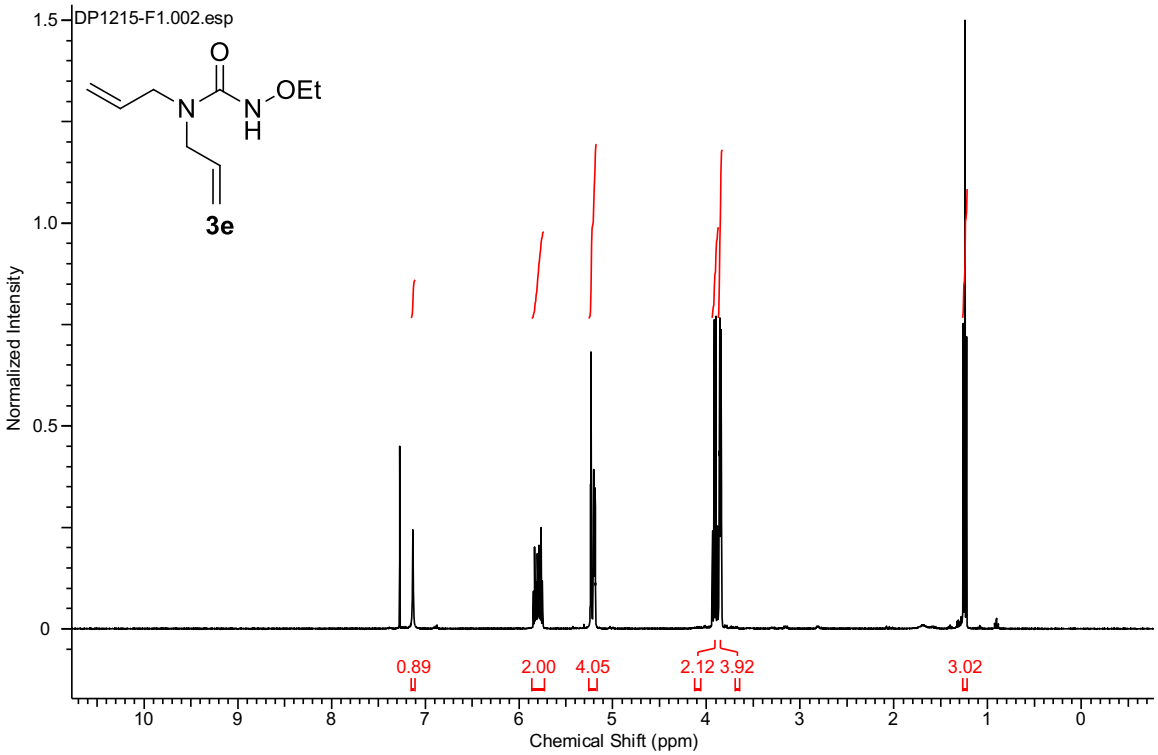

DP1214-F1.003.esp
159.65
71.80
55.08
27.33
20.15
13.57
Normalized Intensity
Chemical Shift (ppm)

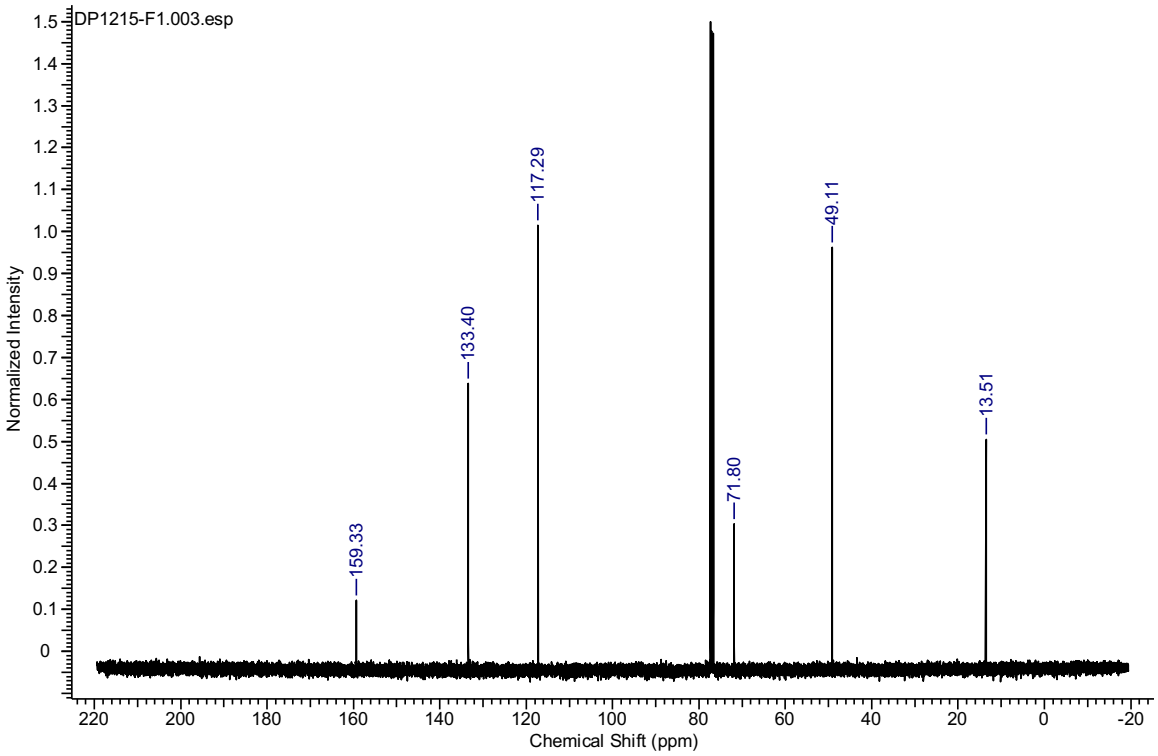

RI11-OBZ-DIISOBUTYL.001.esp
O
N
N
H
OBz
3c
Normalized Intensity
0.96
1.90
0.97
1.97
4.03
2.08
12.09
Chemical Shift (ppm)

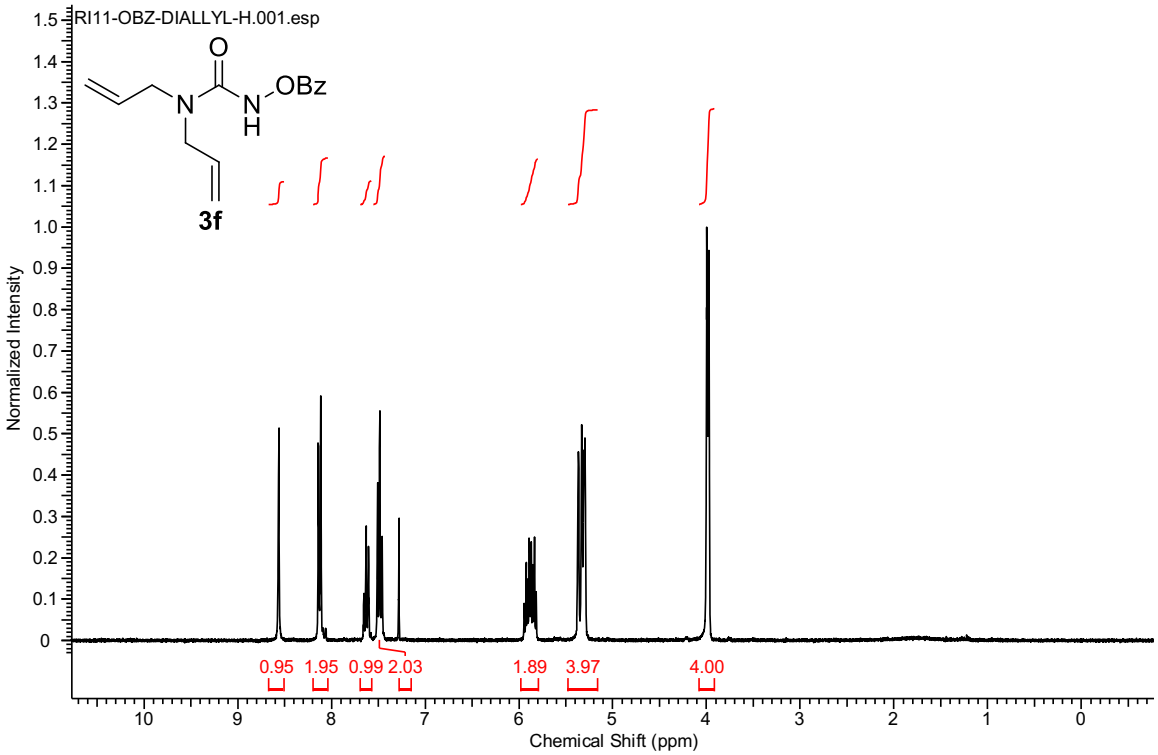

RI11-OBZ-DIISOBUTYL-C.001.esp
Normalized Intensity
167.14
157.96
133.92
129.89
128.55
127.11
54.90
27.18
20.13
Chemical Shift (ppm)

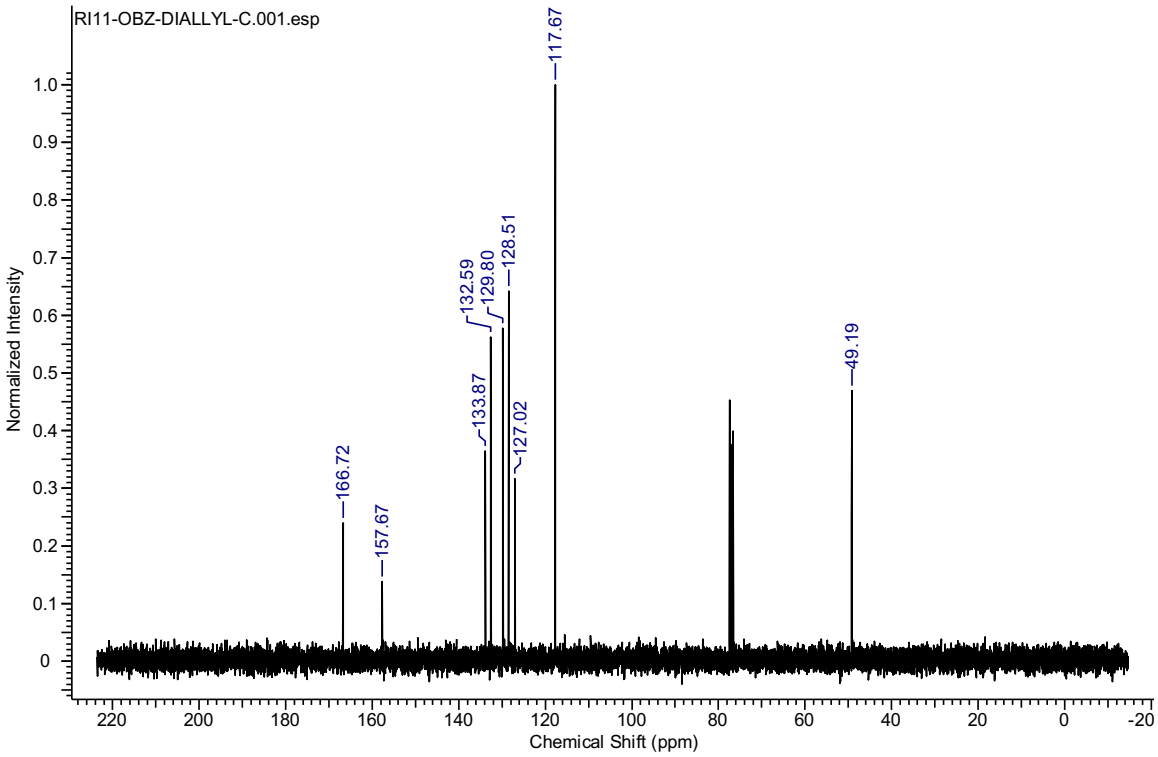

DP-diallylNOH-proton.001.esp
O
N
N
H
OH
3d
Normalized Intensity
1.5
1.0
0.5
0
0.95
0.99
2.00
4.07
4.06
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

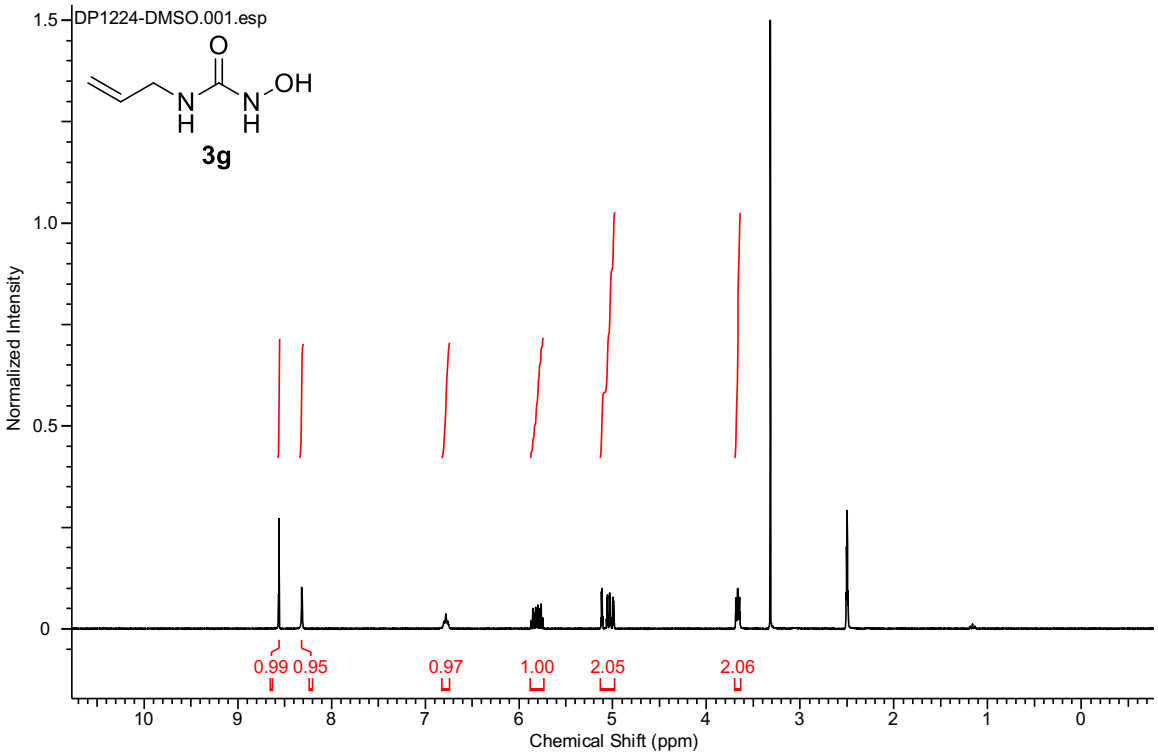

DP-diallylNOH.002.esp
132.83
117.53
48.99
161.49
Normalized Intensity
1.5
1.0
0.5
0
220
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

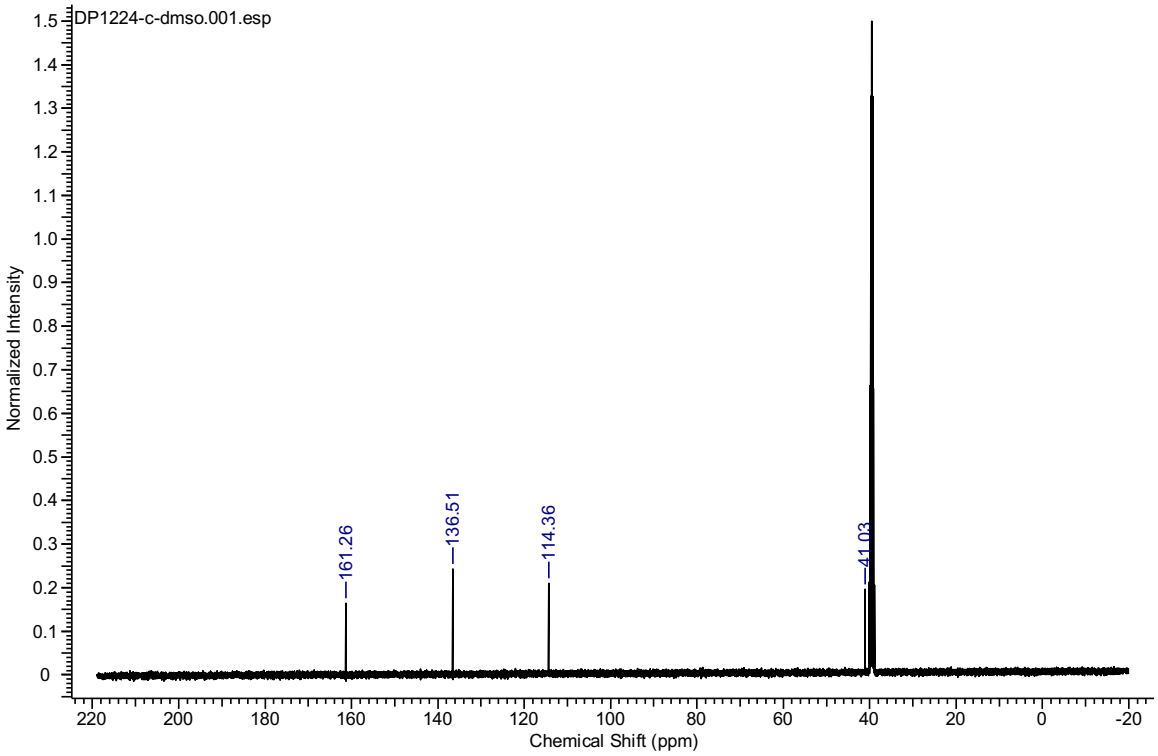

DP1215-F1.002.esp
O
N
N
H
OEt
3e
Normalized Intensity
0.89
2.00
4.05
2.12
3.92
3.02
Chemical Shift (ppm)

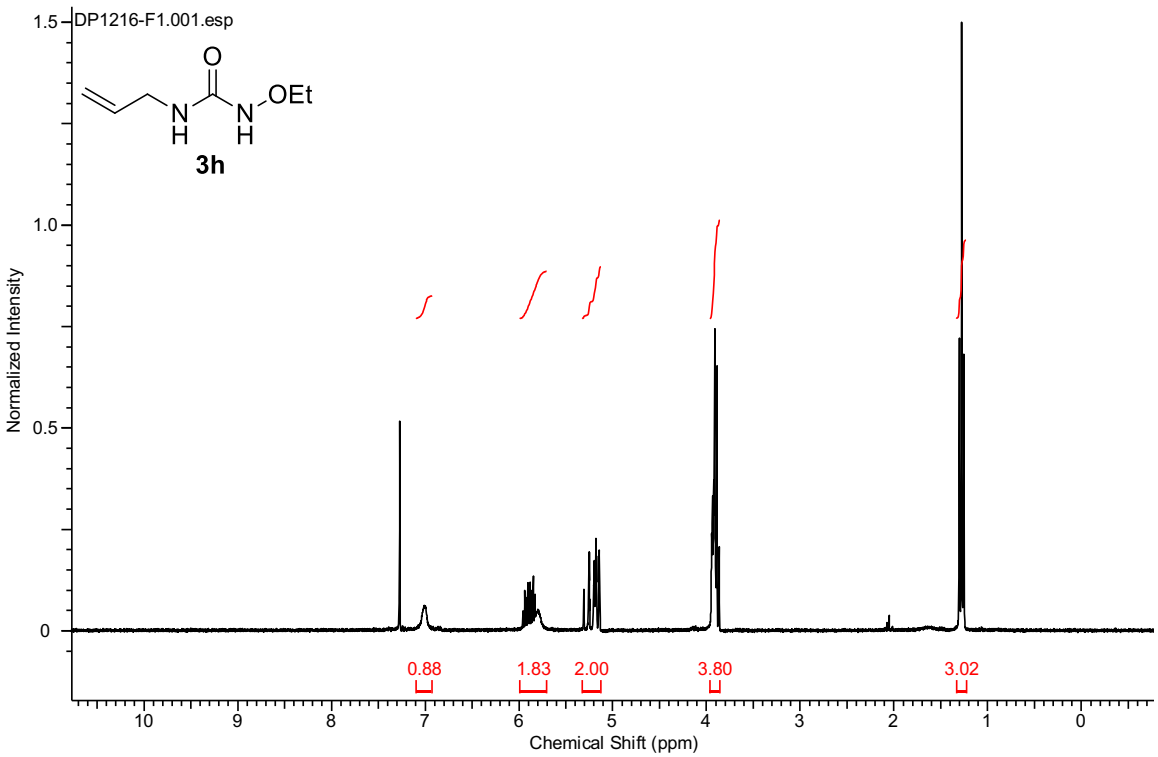

DP1215-F1.003.esp
Normalized Intensity
159.33
133.40
117.29
71.80
49.11
13.51
Chemical Shift (ppm)

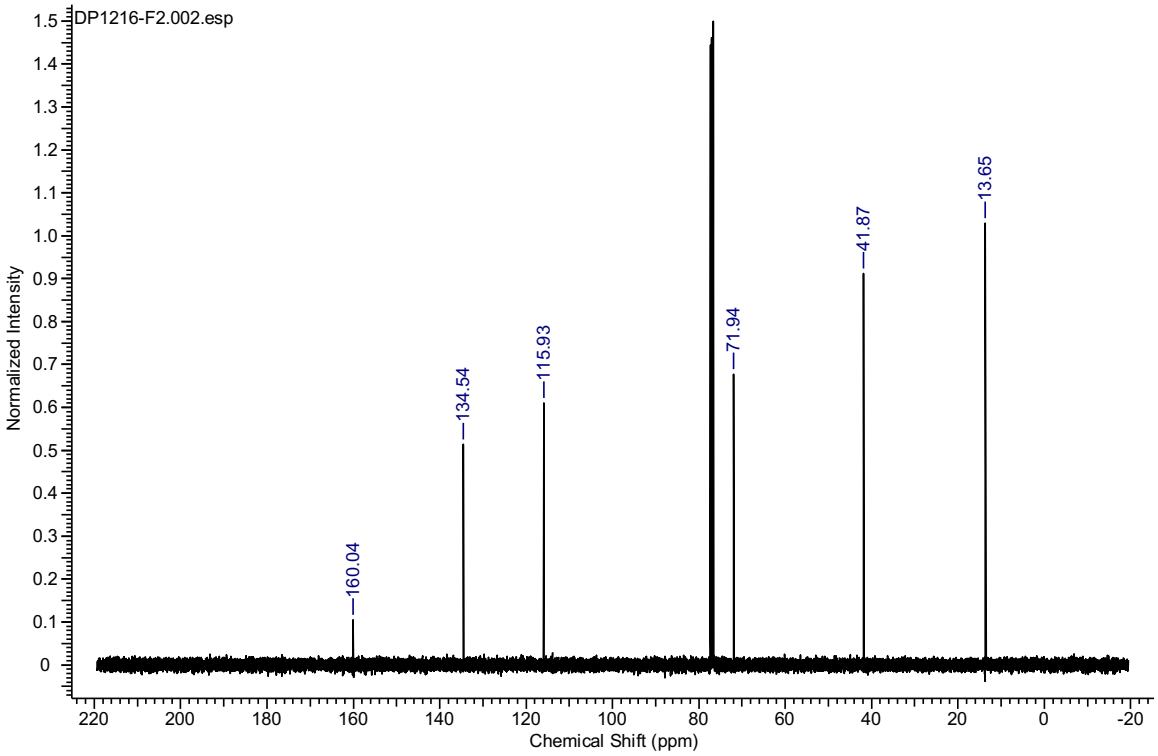

RI11-OBZ-DIALLYL-H.001.esp
O
N
N
H
OBz
3f
Normalized Intensity
0.95
1.95
0.99
2.03
1.89
3.97
4.00
Chemical Shift (ppm)

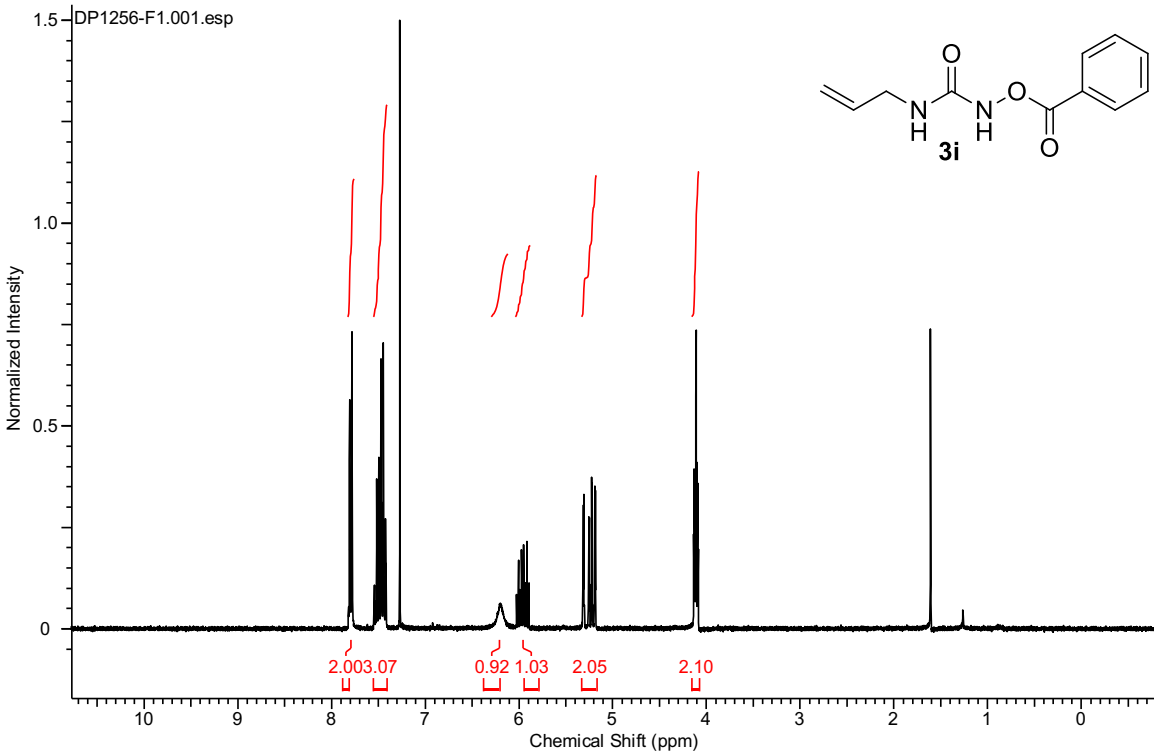

RI11-OBZ-DIALLYL-C.001.esp
Normalized Intensity
166.72
157.67
133.87
132.59
129.80
128.51
127.02
117.67
49.19
Chemical Shift (ppm)

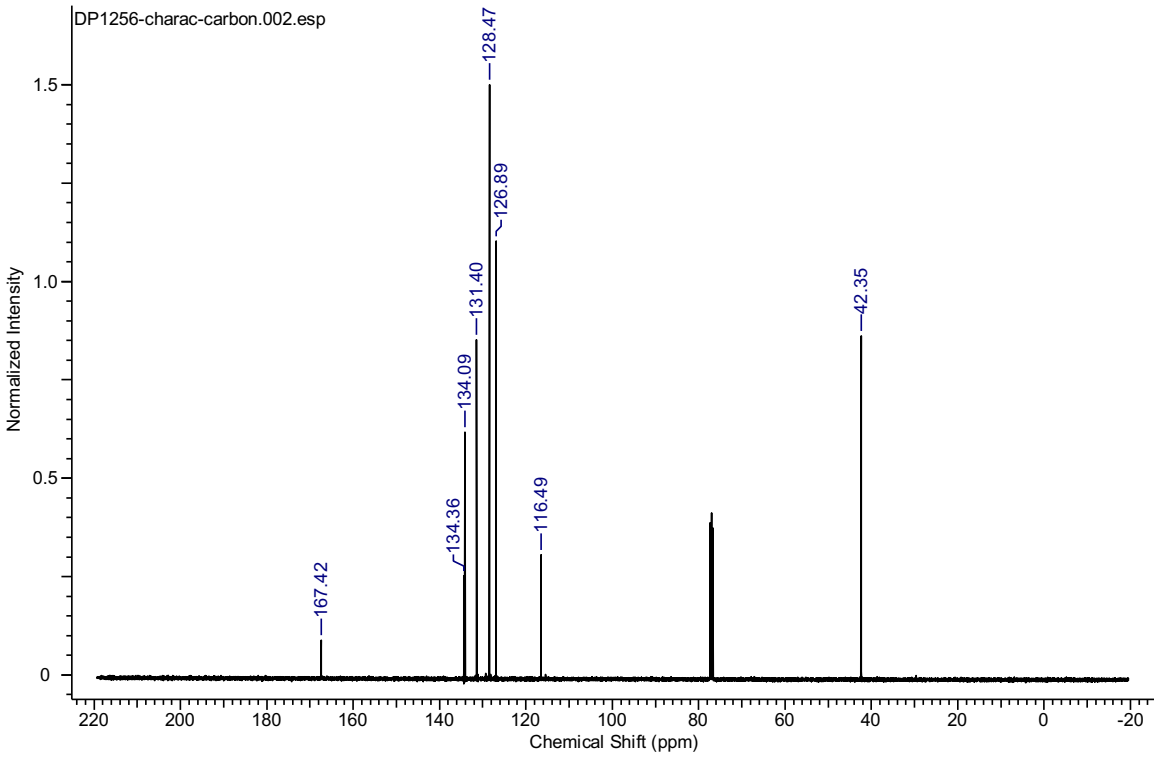

DP1224-DMSO.001.esp
O
N
H
N
H
OH
3g
Normalized Intensity
0.99
0.95
0.97
1.00
2.05
2.06
Chemical Shift (ppm)

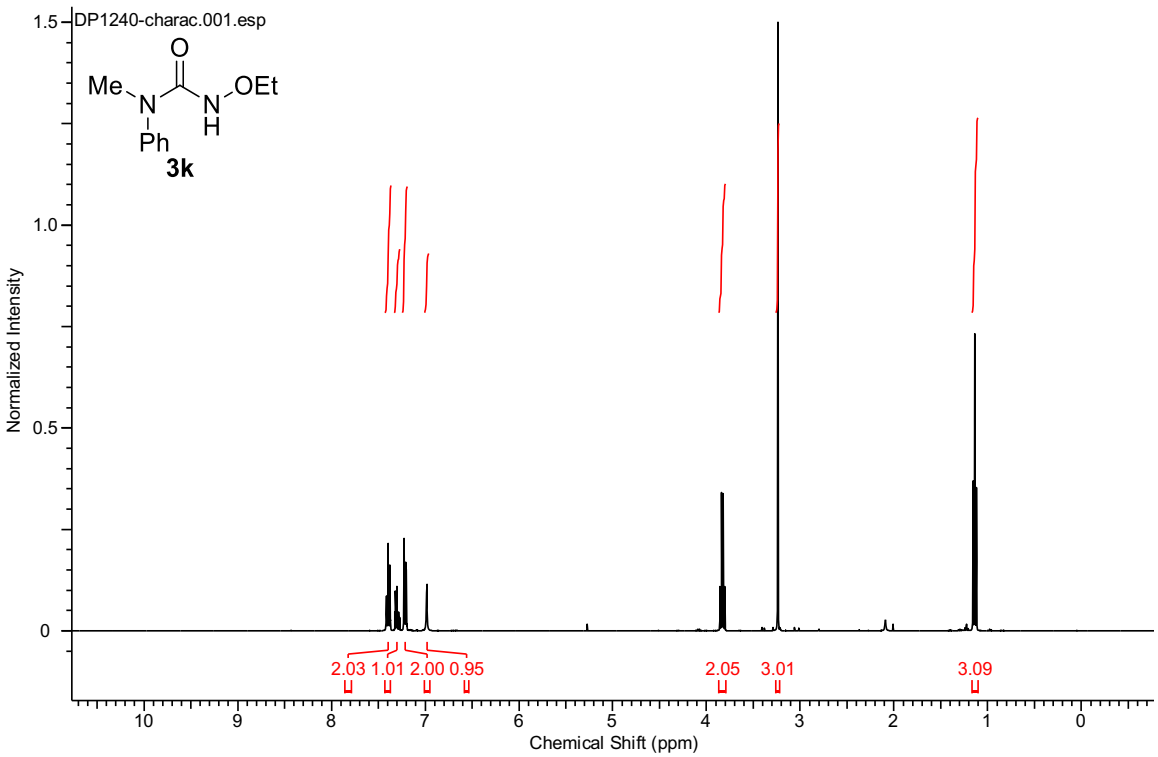

DP1224-c-dmso.001.esp
Normalized Intensity
161.26
136.51
114.36
41.03
Chemical Shift (ppm)

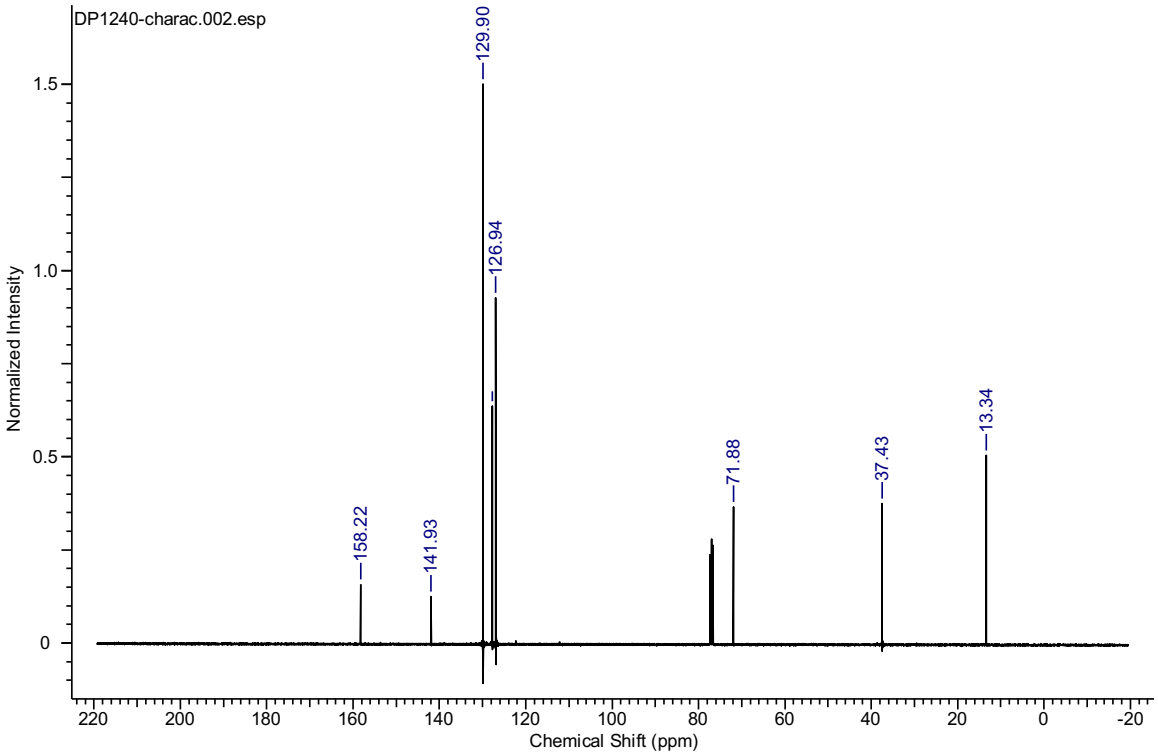

DP1216-F1.001.esp
O
N
H
N
H
OEt
3h
Normalized Intensity
0.88
1.83
2.00
3.80
3.02
Chemical Shift (ppm)

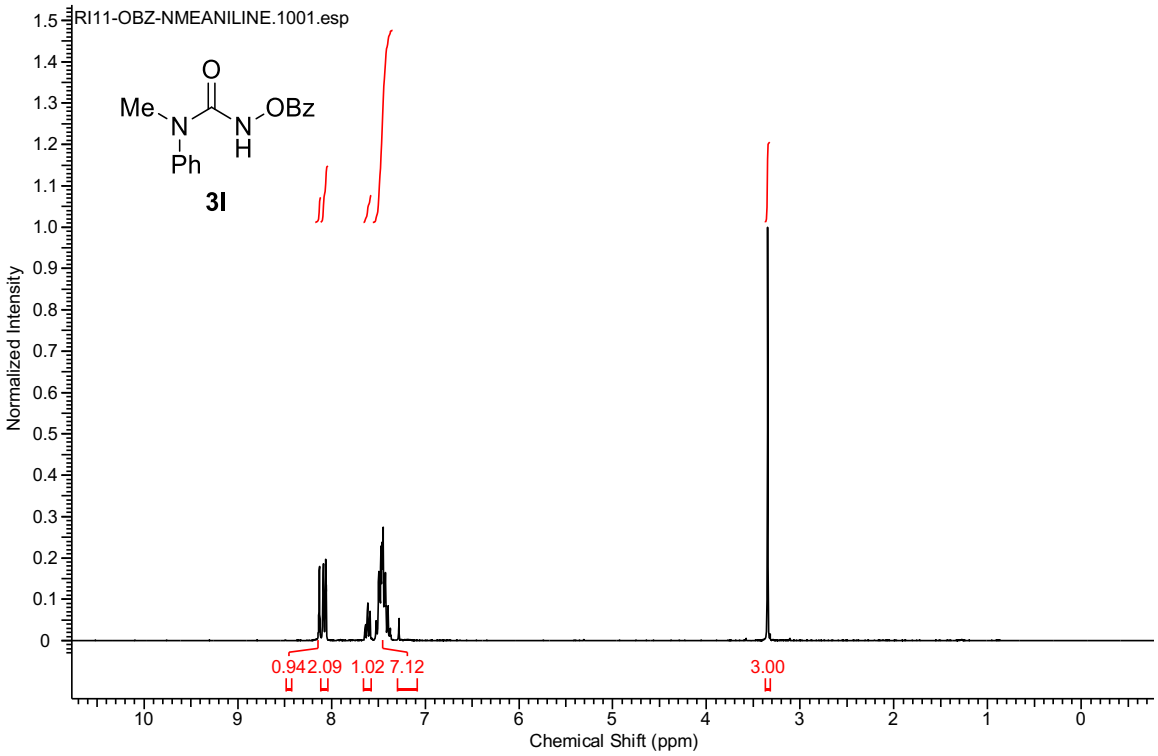

DP1216-F2.002.esp
160.04
134.54
115.93
71.94
41.87
13.65
Normalized Intensity
Chemical Shift (ppm)

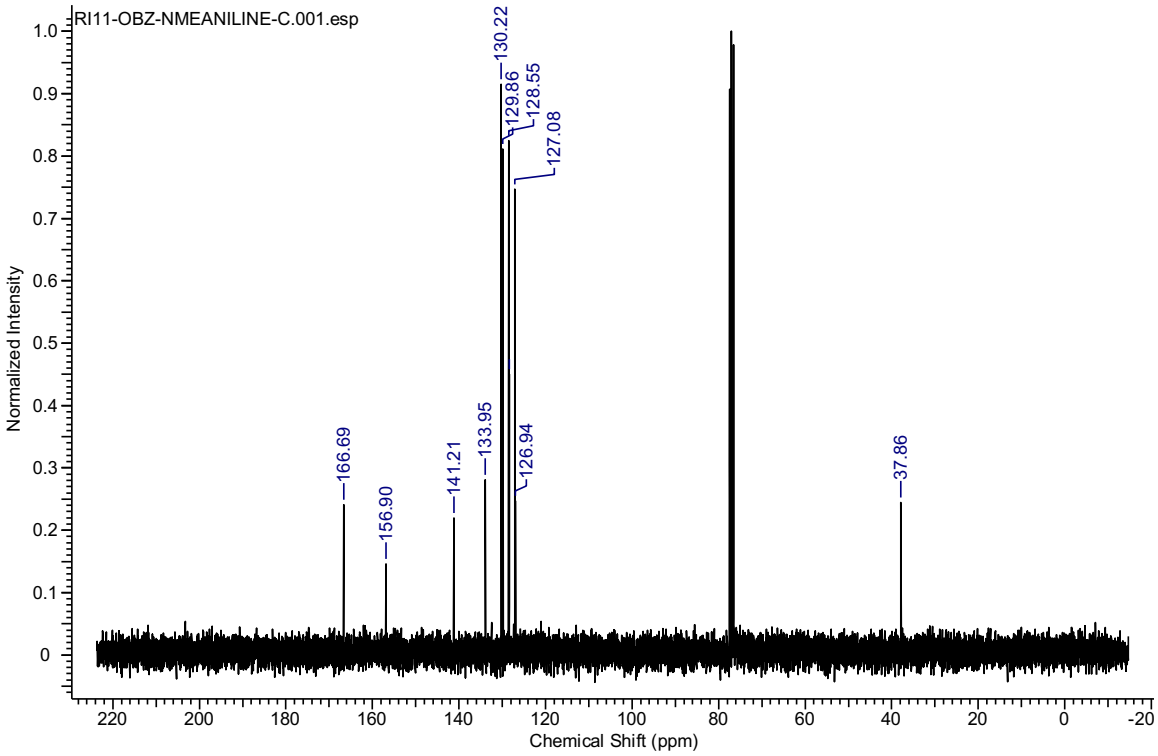

DP1256-F1.001.esp
Normalized Intensity
Chemical Shift (ppm)
2.00
3.07
0.92
1.03
2.05
2.10
3i

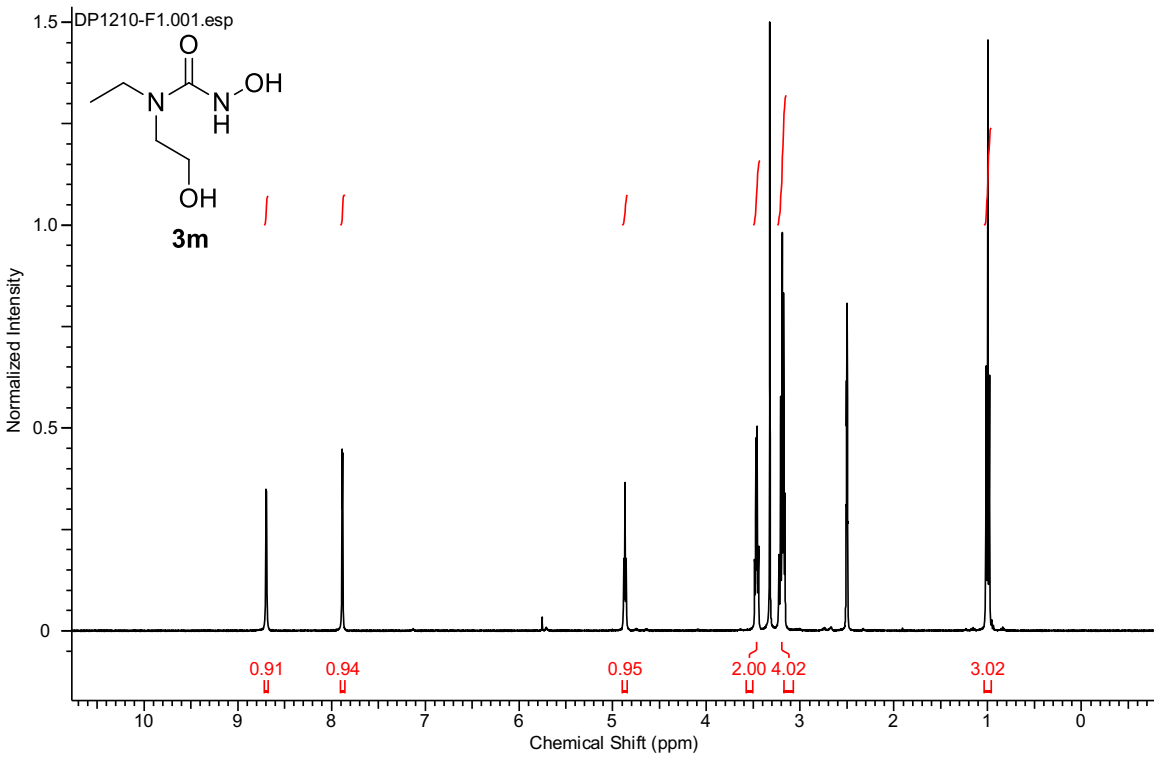

DP1256-charac-carbon.002.esp
Normalized Intensity
Chemical Shift (ppm)
128.47
126.89
131.40
134.09
134.36
116.49
167.42
42.35

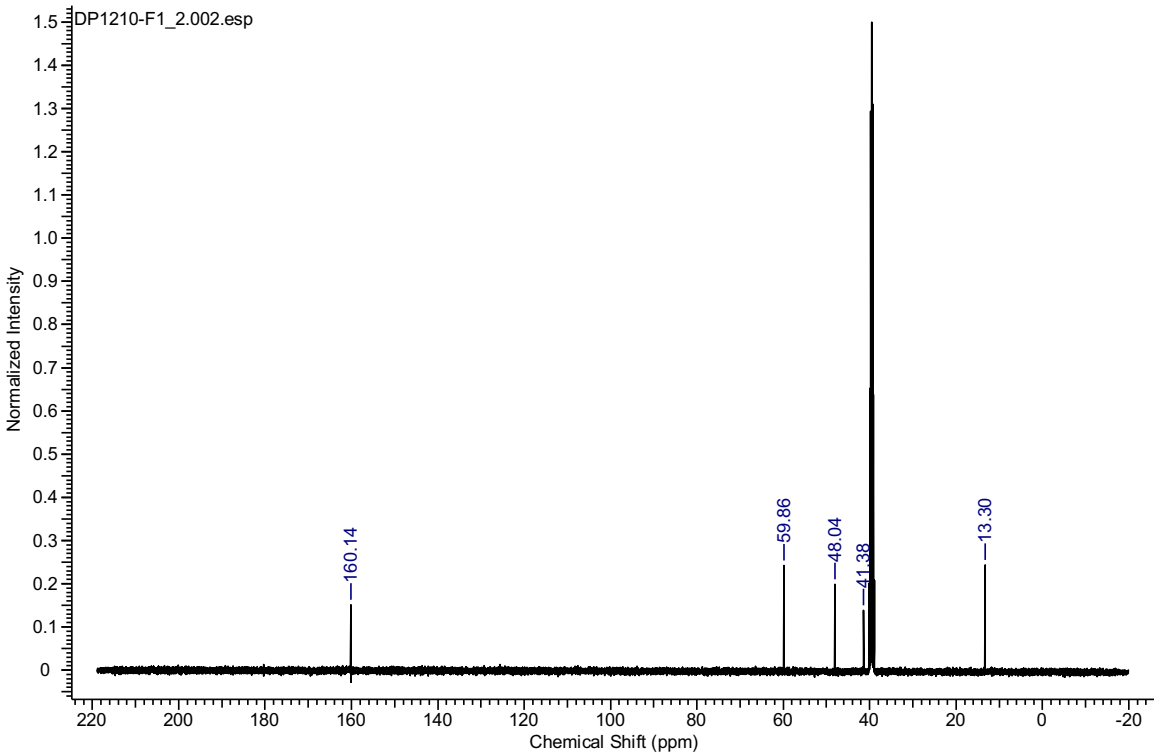

DP1240-charac.001.esp
Me
N
O
N
H
OEt
Ph
3k
Normalized Intensity
Chemical Shift (ppm)
2.03 1.01 2.00 0.95
2.05
3.01
3.09

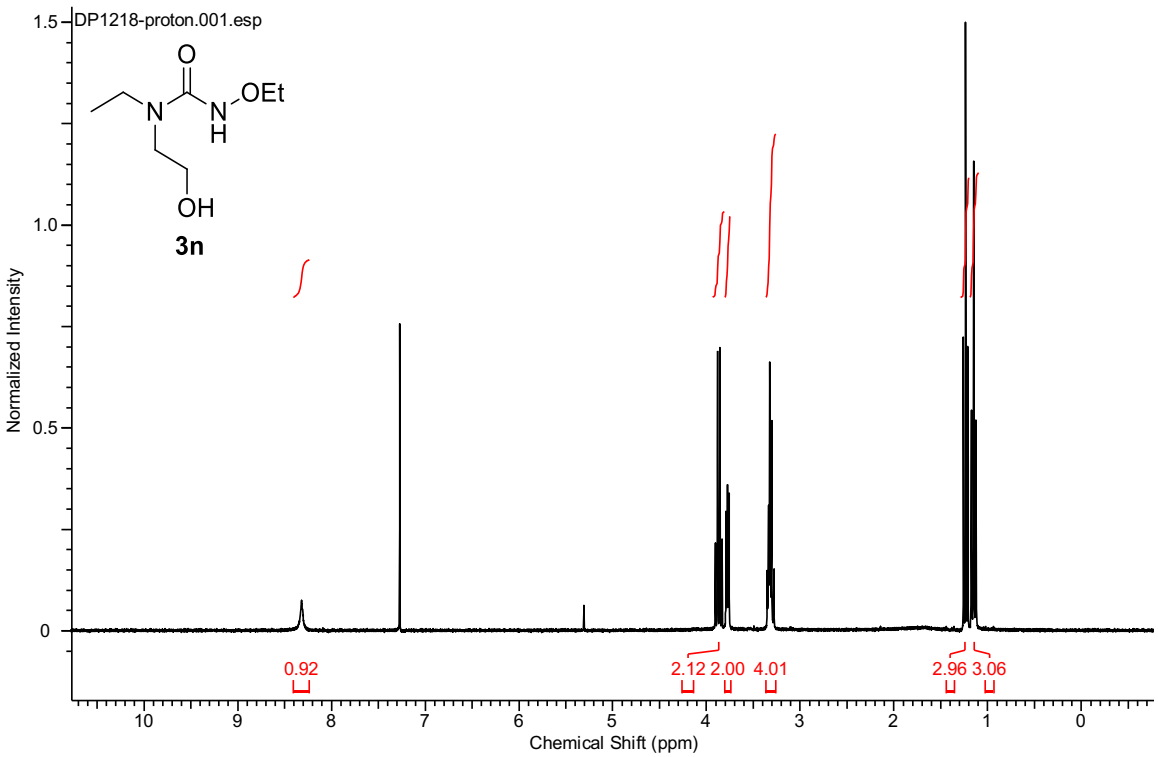

DP1240-charac.002.esp
129.90
126.94
158.22
141.93
71.88
37.43
13.34
Normalized Intensity
Chemical Shift (ppm)

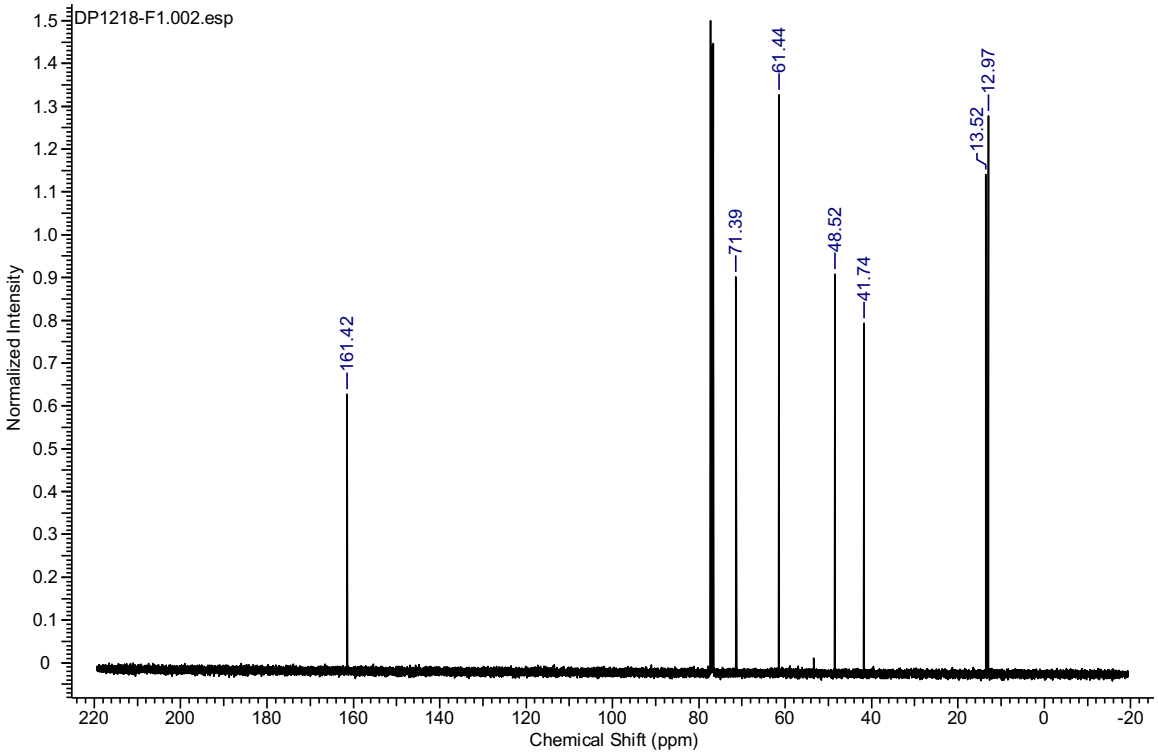

RI11-OBZ-NMEANILINE.1001.esp
Me
N
Ph
O
N
H
OBz
3l
Normalized Intensity
0.94 2.09 1.02 7.12
3.00
Chemical Shift (ppm)

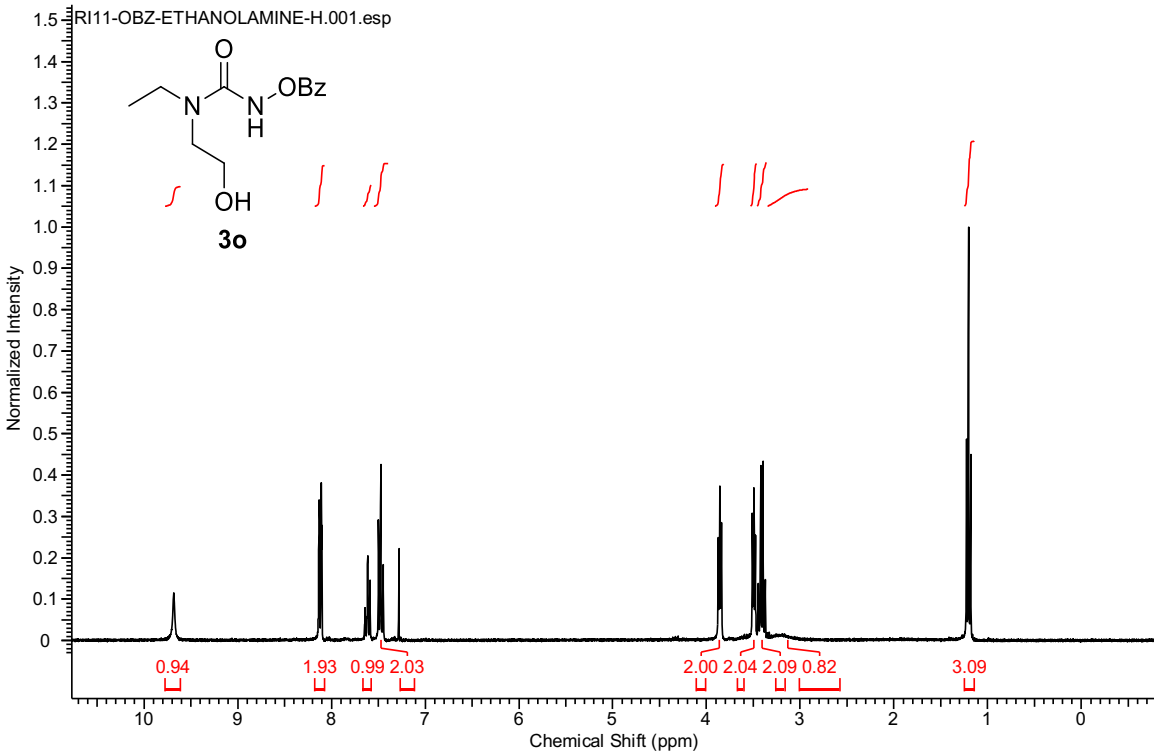

RI11-OBZ-NMEANILINE-C.001.esp
130.22
129.86
128.55
127.08
166.69
156.90
141.21
133.95
126.94
37.86
Normalized Intensity
Chemical Shift (ppm)

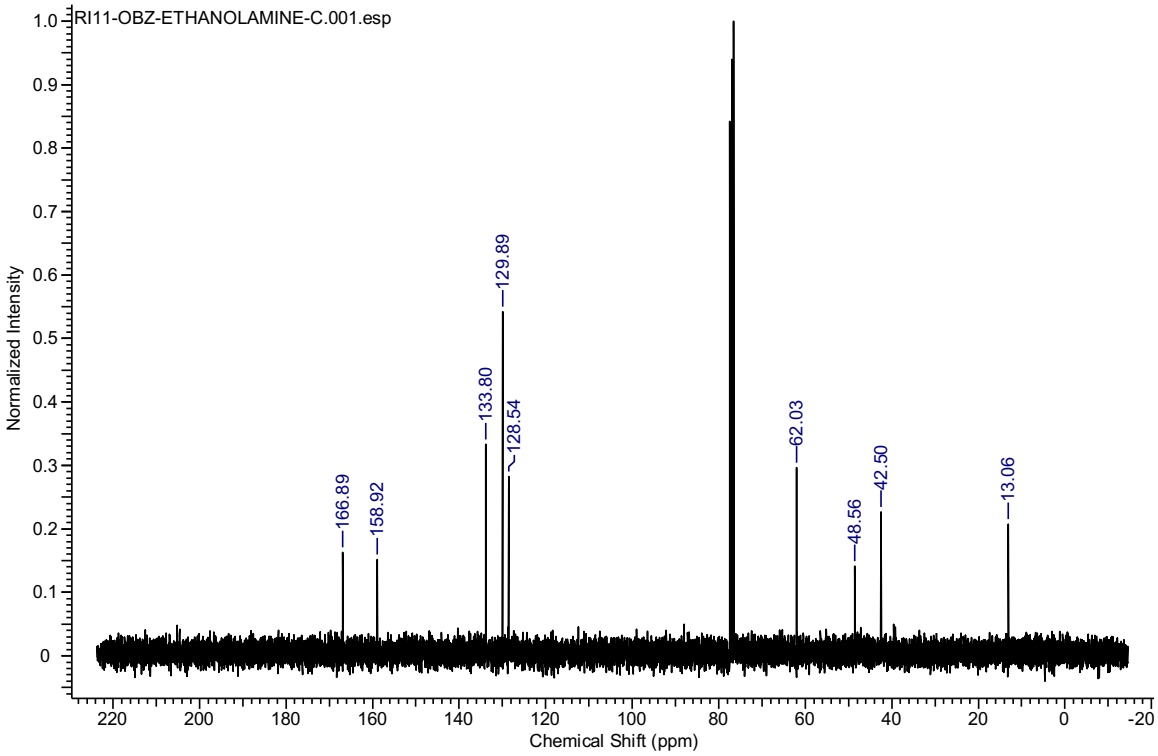

DP1210-F1.001.esp
O
N
OH
N
H
OH
3m
Normalized Intensity
0.91
0.94
0.95
2.00
4.02
3.02
Chemical Shift (ppm)

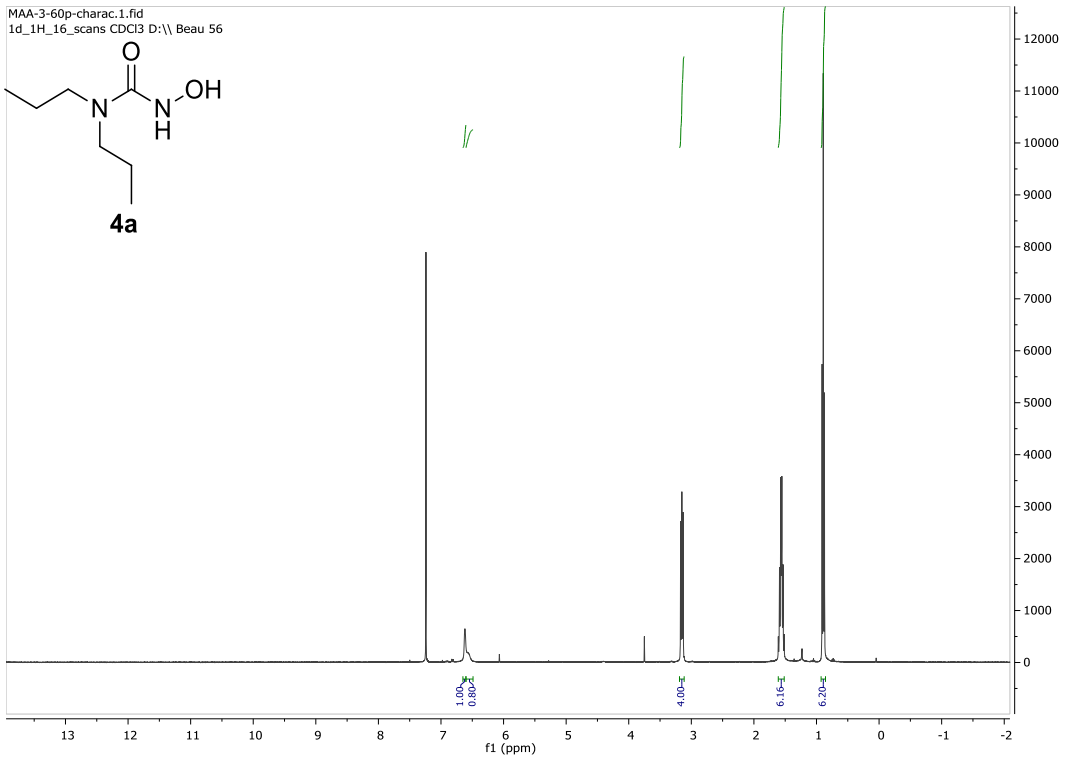

DP1210-F1_2.002.esp
Normalized Intensity
160.14
59.86
48.04
41.38
13.30
Chemical Shift (ppm)

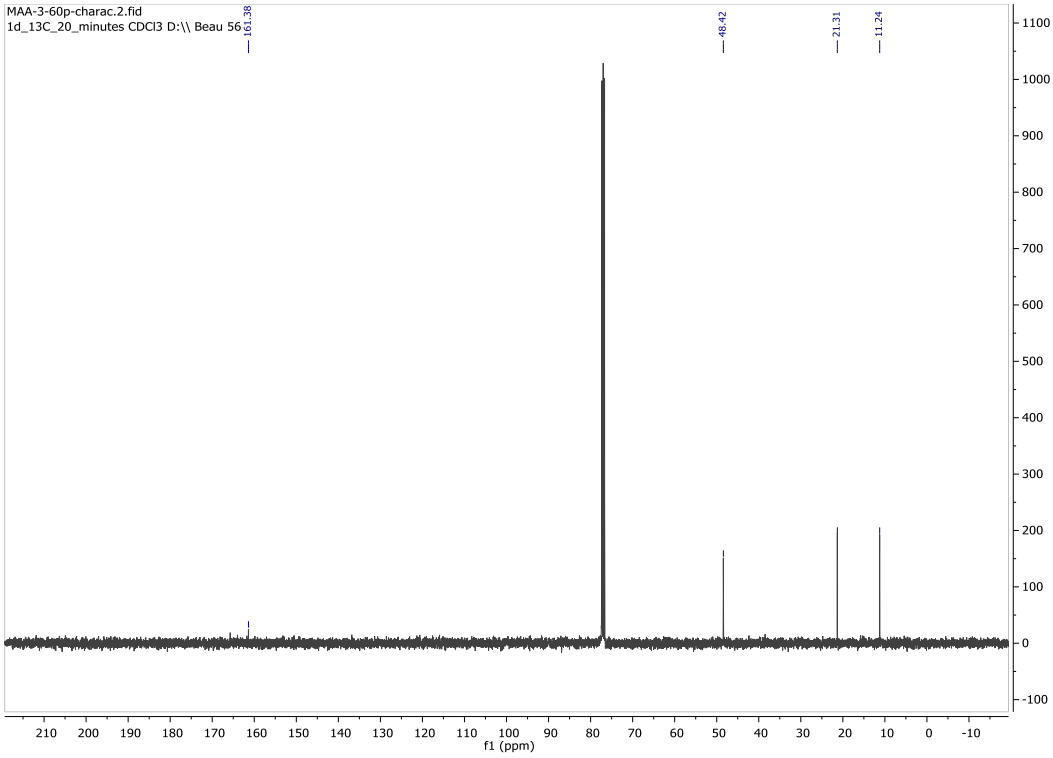

DP1218-proton.001.esp
O
OEt
N
N
H
OH
3n
Normalized Intensity
Chemical Shift (ppm)
0.92
2.12
2.00
4.01
2.96
3.06

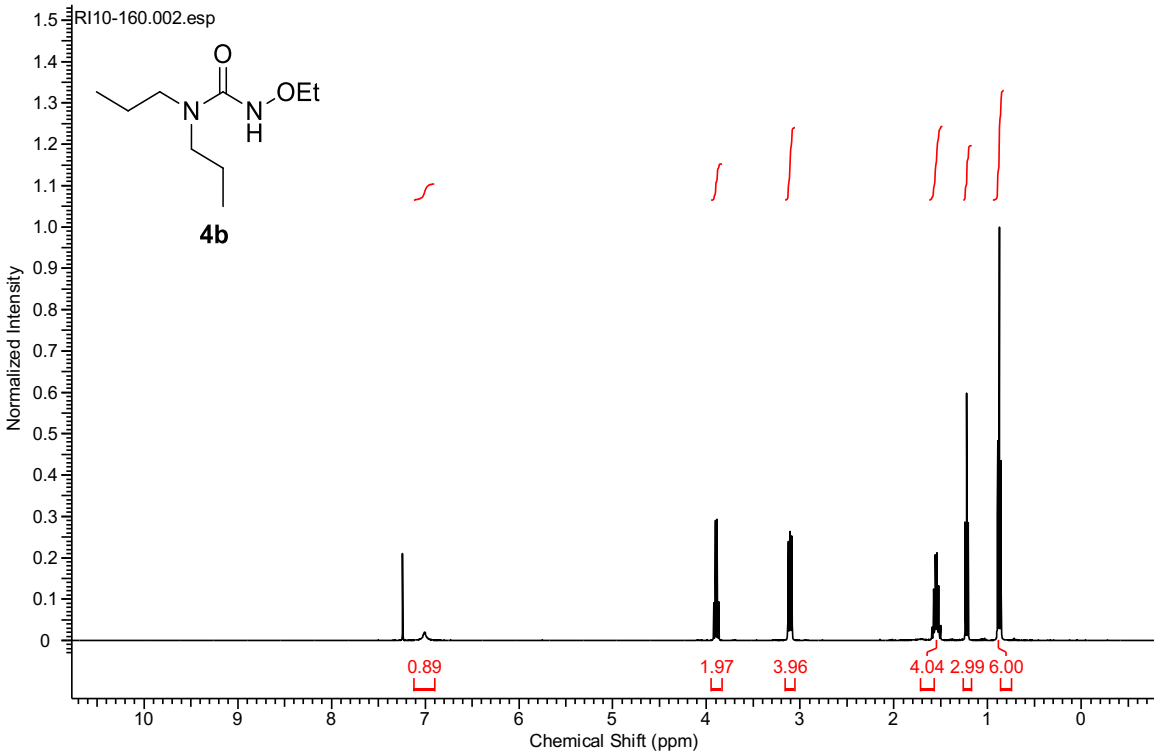

DP1218-F1.002.esp
Normalized Intensity
Chemical Shift (ppm)
161.42
71.39
61.44
48.52
41.74
13.52
12.97

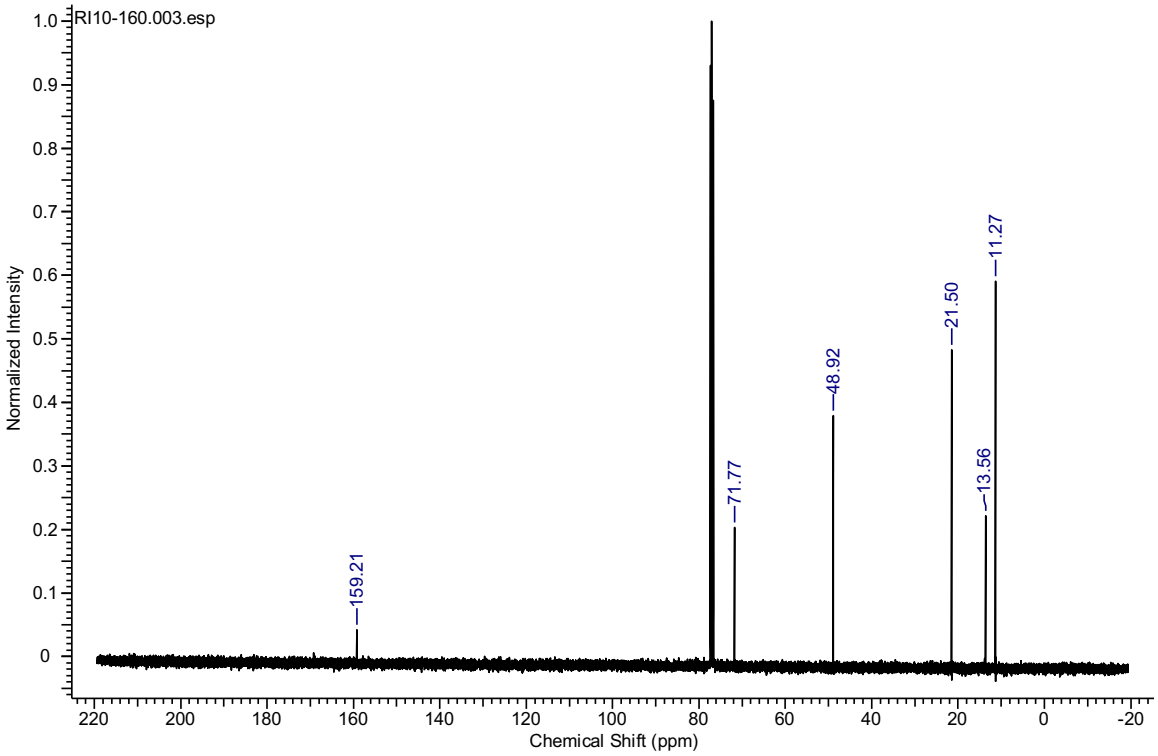

RI11-OBZ-ETHANOLAMINE-H.001.esp
O
N
N
H
OBz
OH
3o
Normalized Intensity
0.94
1.93
0.99
2.03
2.00
2.04
2.09
0.82
3.09
Chemical Shift (ppm)

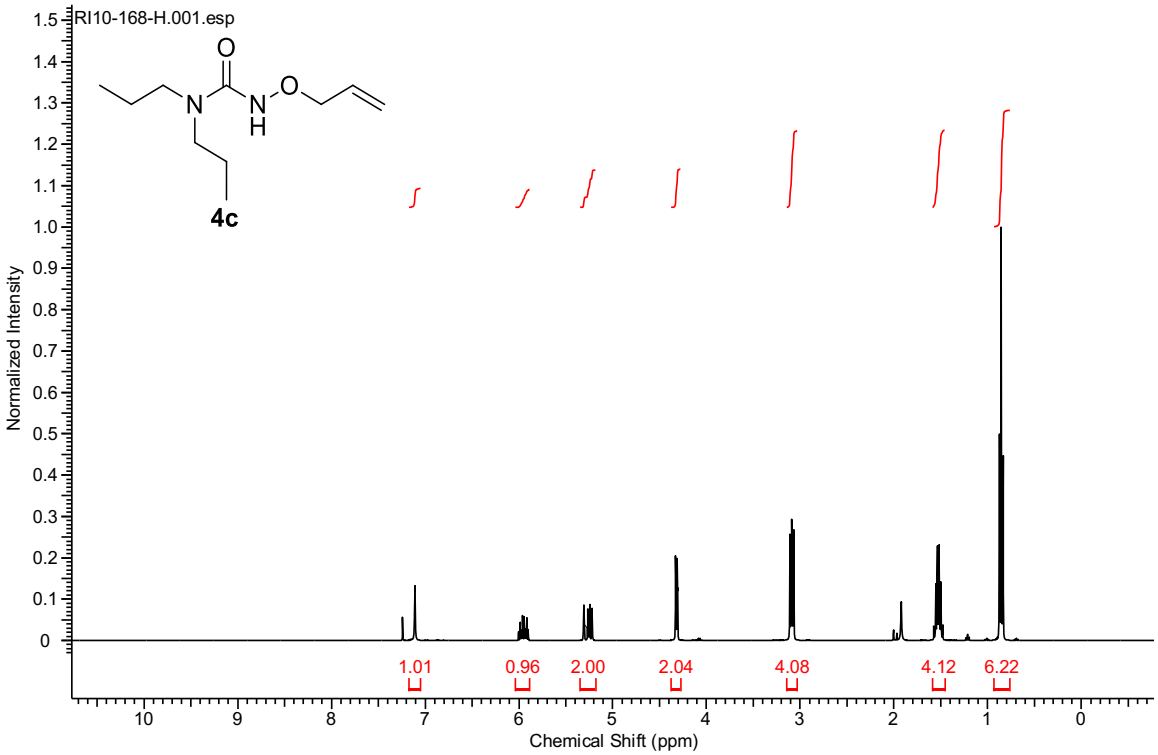

RI11-OBZ-ETHANOLAMINE-C.001.esp
Normalized Intensity
166.89
158.92
133.80
129.89
128.54
62.03
48.56
42.50
13.06
Chemical Shift (ppm)

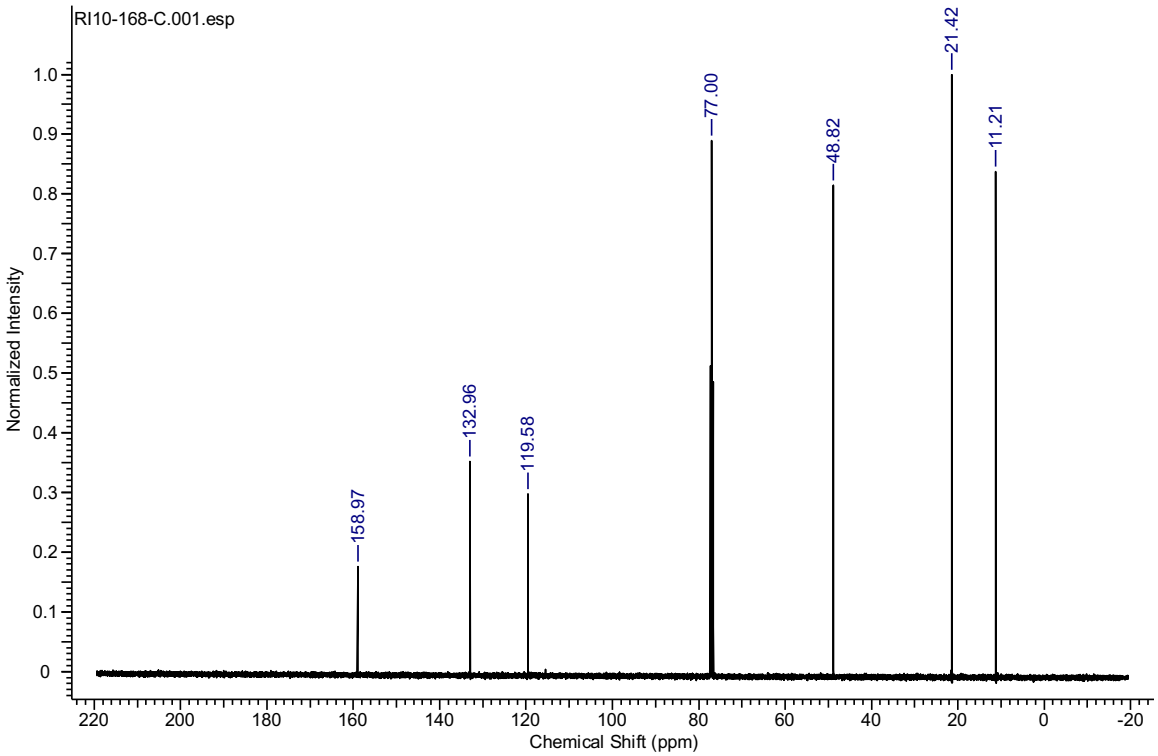

MAA-3-60p-charac.1.fid
1d_1H_16_scans CDCl3 D:\\ Beau 56
O
N
N
H
OH
4a
1.00
0.00
4.00
6.16
6.20
f1 (ppm)

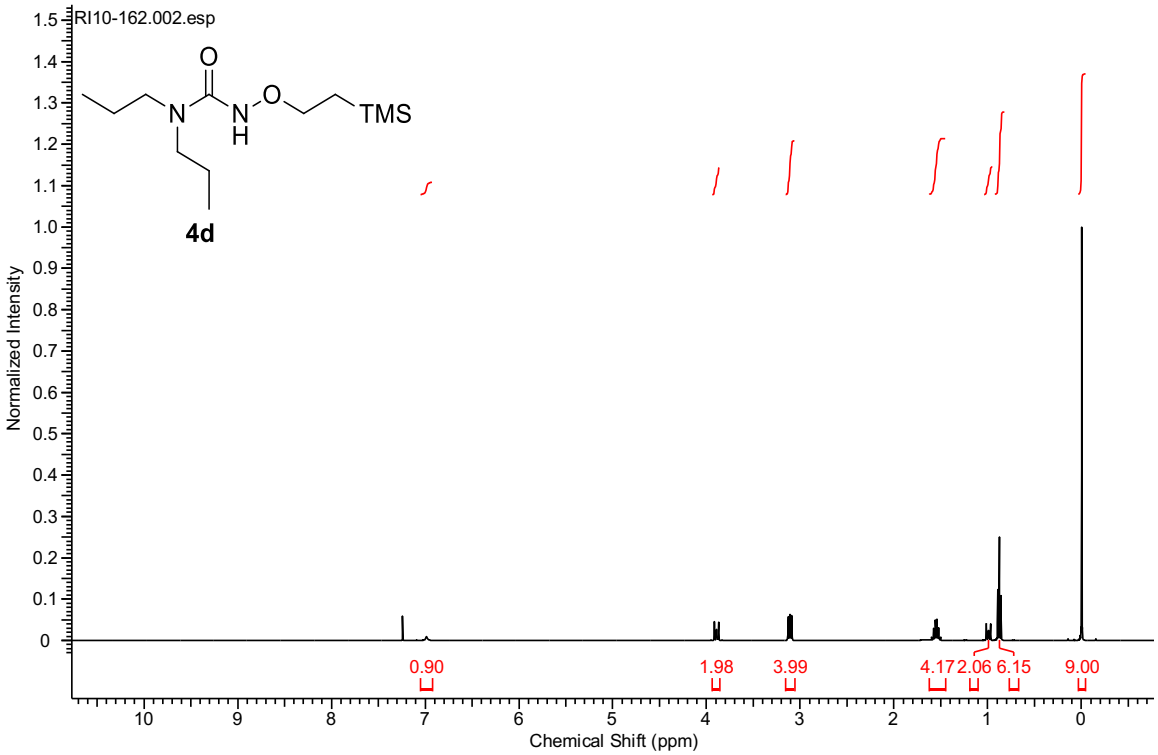

MAA-3-60p-charac.2.fid
1d_13C_20_minutes CDCl3 D:\\ Beau 56
161.38
48.42
21.31
11.24
f1 (ppm)

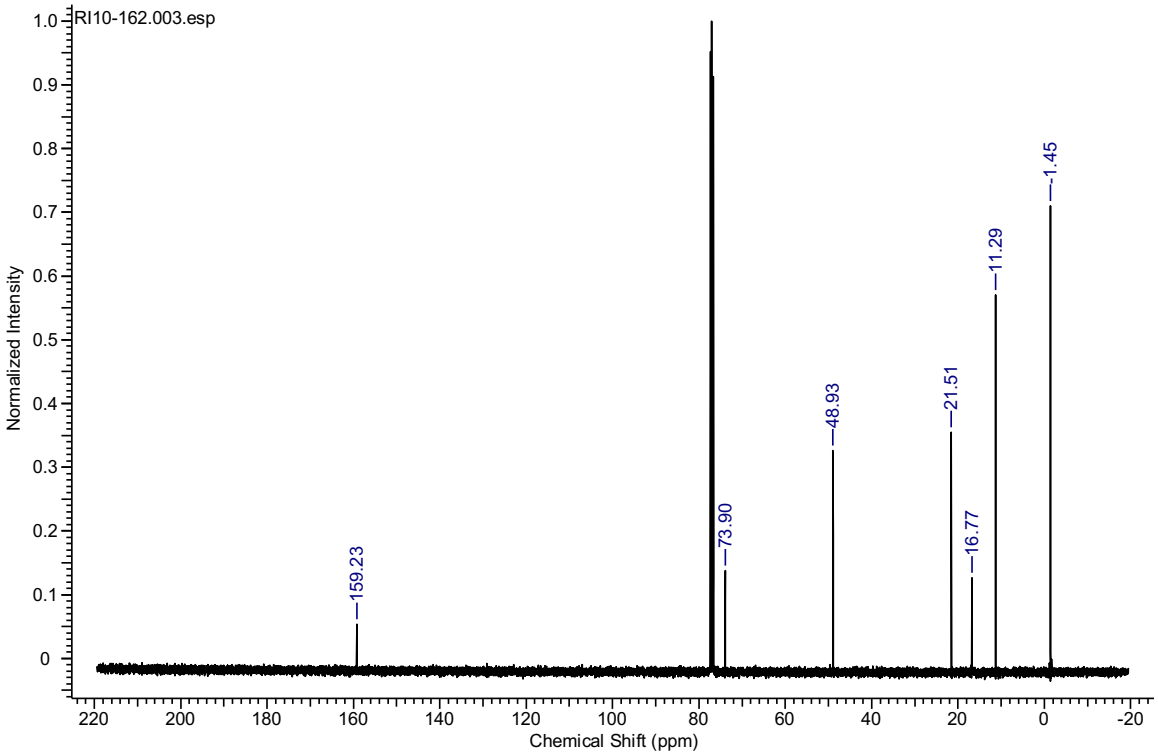

RI10-160.002.esp
O
N
N
H
OEt
4b
Normalized Intensity
0.89
1.97
3.96
4.04
2.99
6.00
Chemical Shift (ppm)

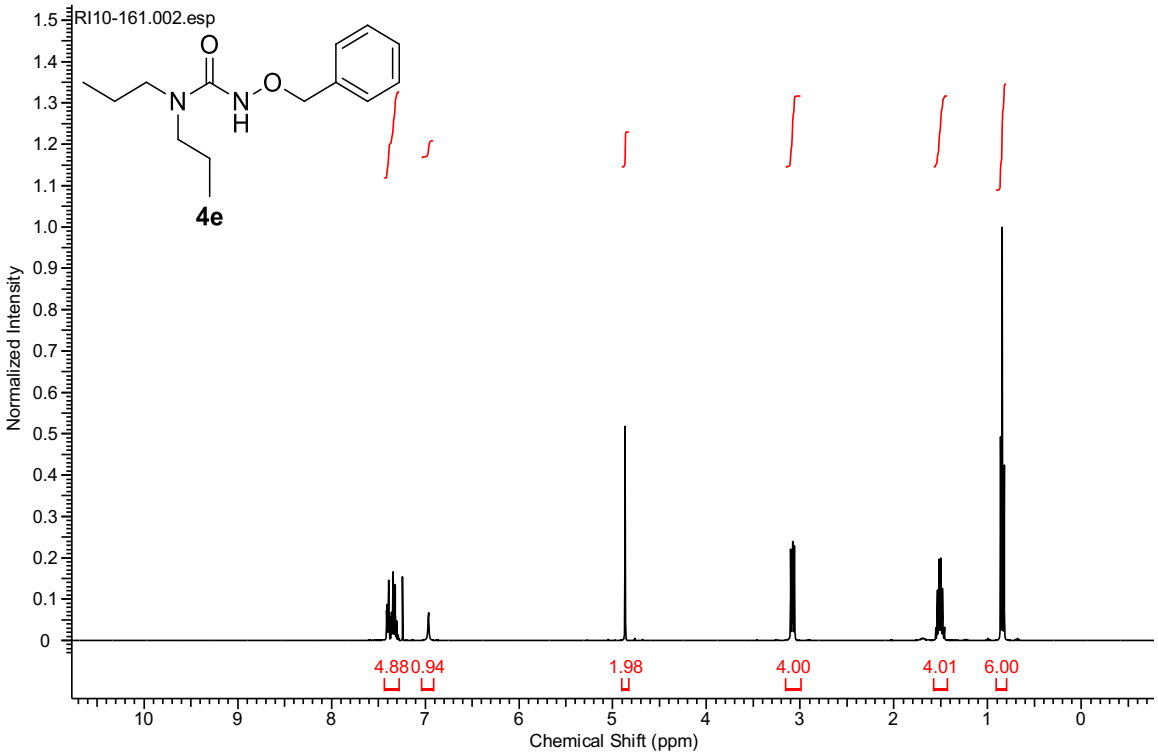

RI10-160.003.esp
Normalized Intensity
159.21
71.77
48.92
21.50
13.56
11.27
Chemical Shift (ppm)

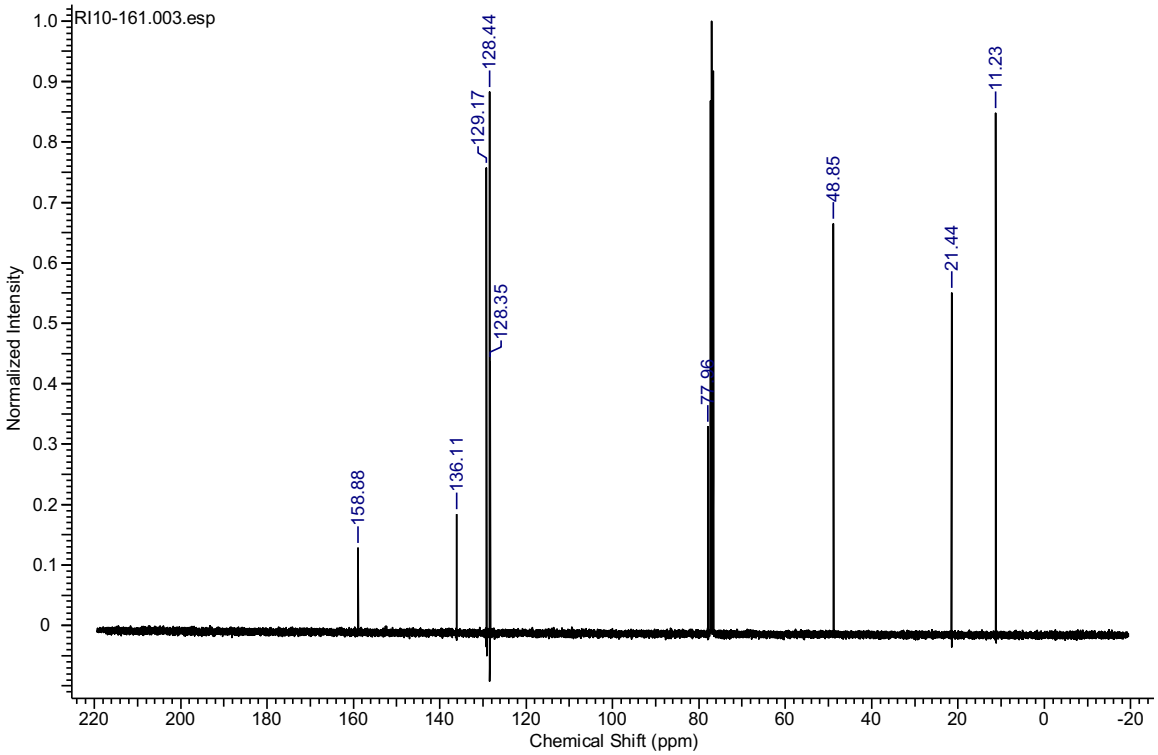

RI10-168-H.001.esp
O
N
N
H
O
4c
Normalized Intensity
1.01
0.96
2.00
2.04
4.08
4.12
6.22
Chemical Shift (ppm)

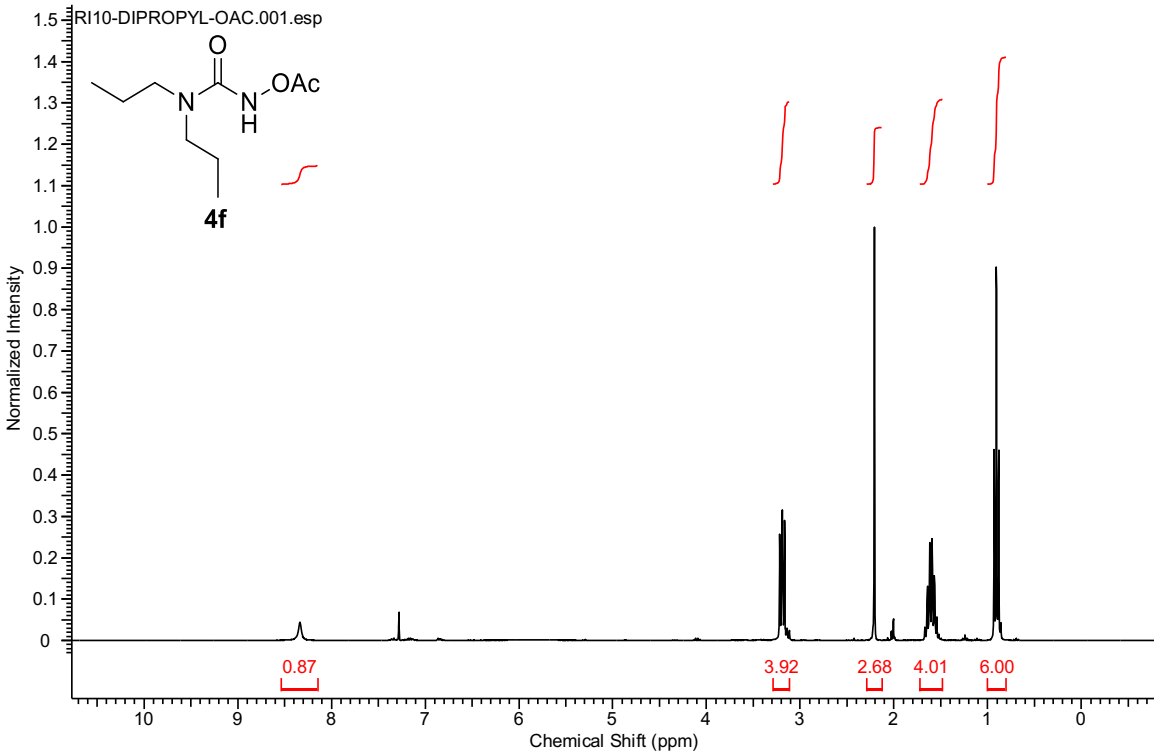

RI10-168-C.001.esp
158.97
132.96
119.58
77.00
48.82
21.42
11.21
Normalized Intensity
Chemical Shift (ppm)

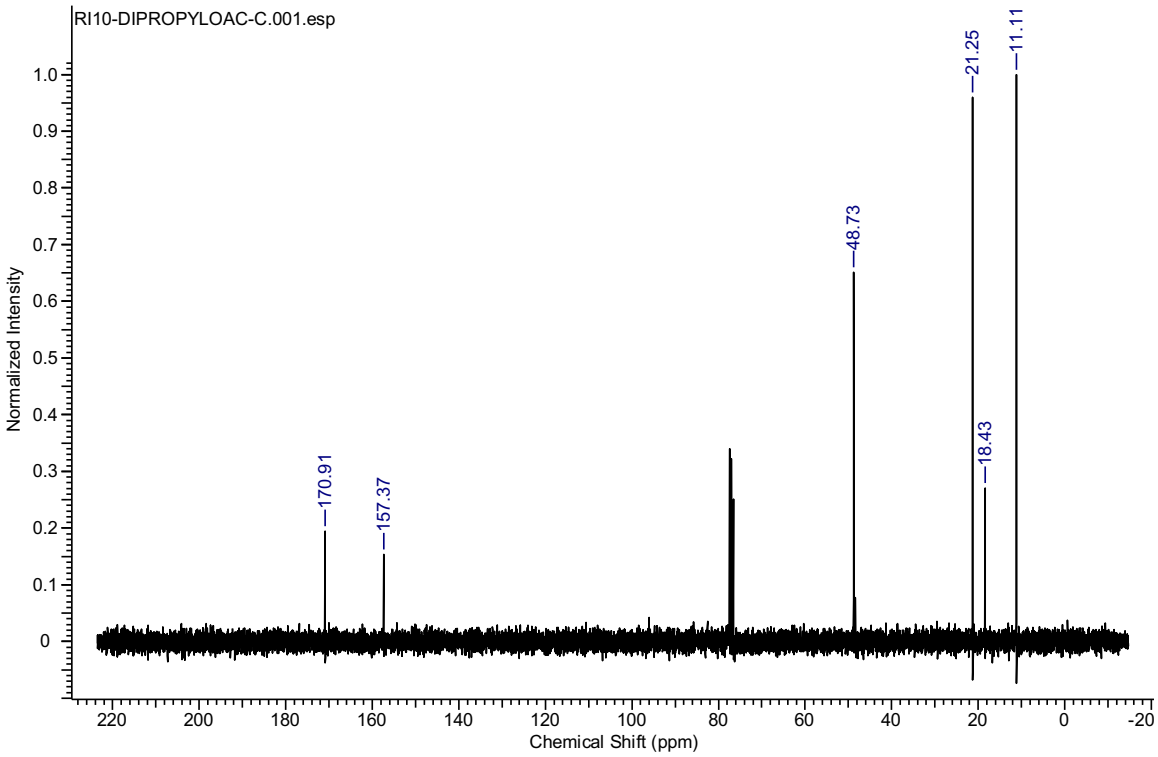

RI10-162.002.esp
O
N
N
H
O
TMS
4d
Normalized Intensity
0.90
1.98
3.99
4.17
2.06
6.15
9.00
Chemical Shift (ppm)

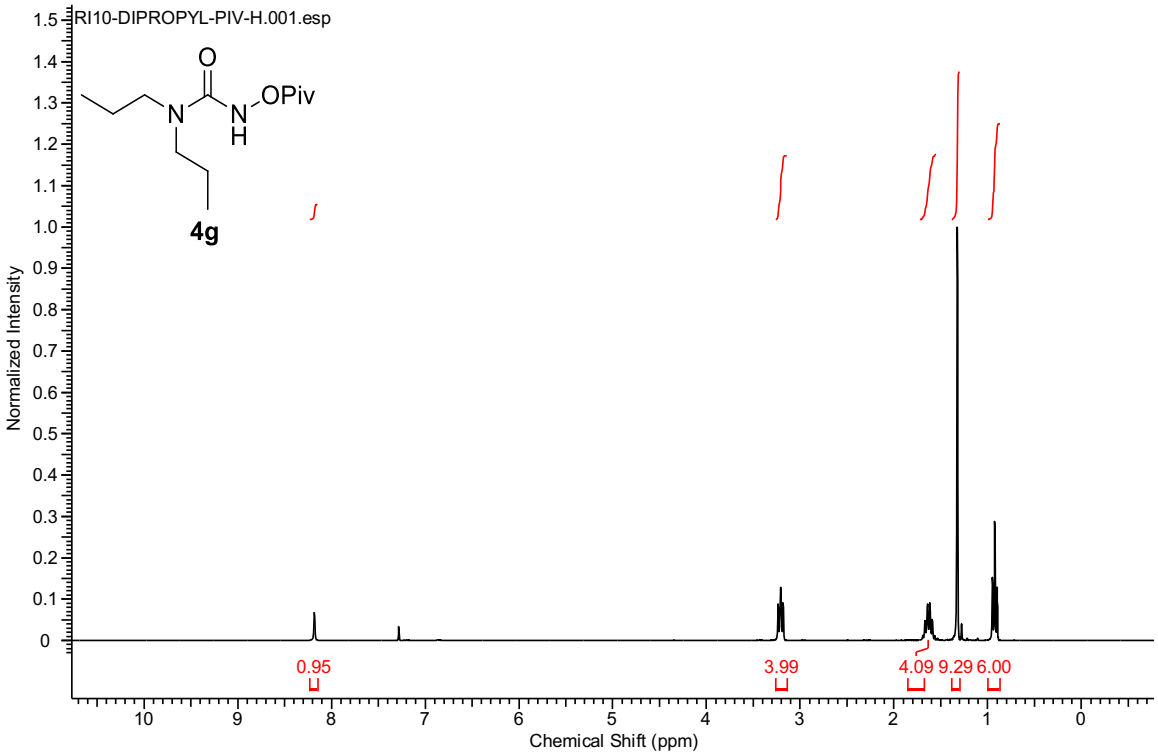

RI10-162.003.esp
Normalized Intensity
159.23
73.90
48.93
21.51
16.77
11.29
1.45
Chemical Shift (ppm)

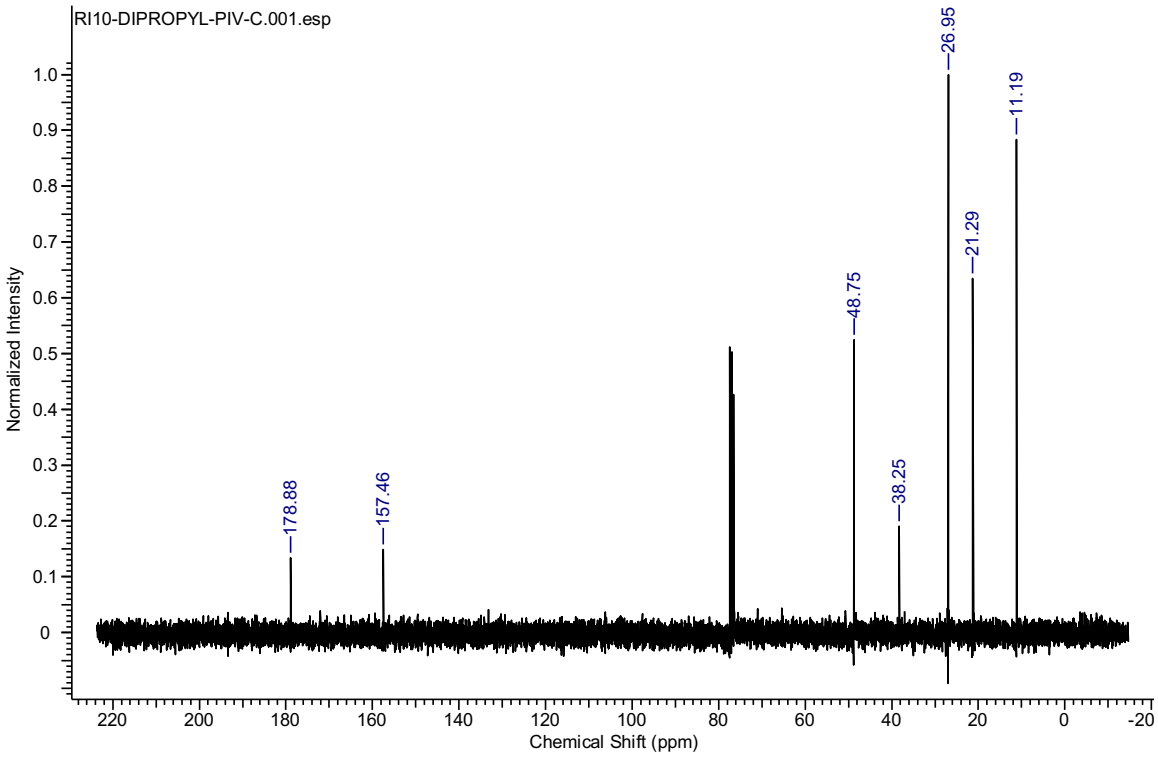

RI10-161.002.esp
O
N
N
H
O
4e
Normalized Intensity
4.88
0.94
1.98
4.00
4.01
6.00
Chemical Shift (ppm)

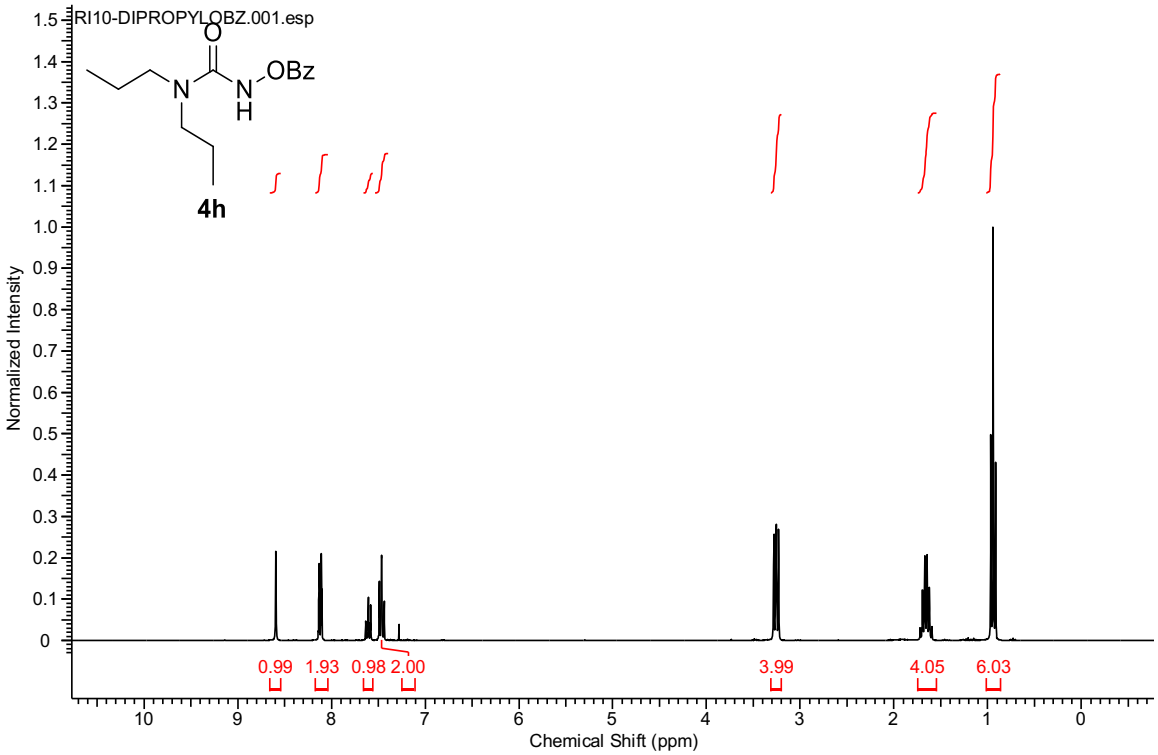

RI10-161.003.esp
158.88
136.11
129.17
128.44
128.35
77.96
48.85
21.44
11.23
Normalized Intensity
Chemical Shift (ppm)

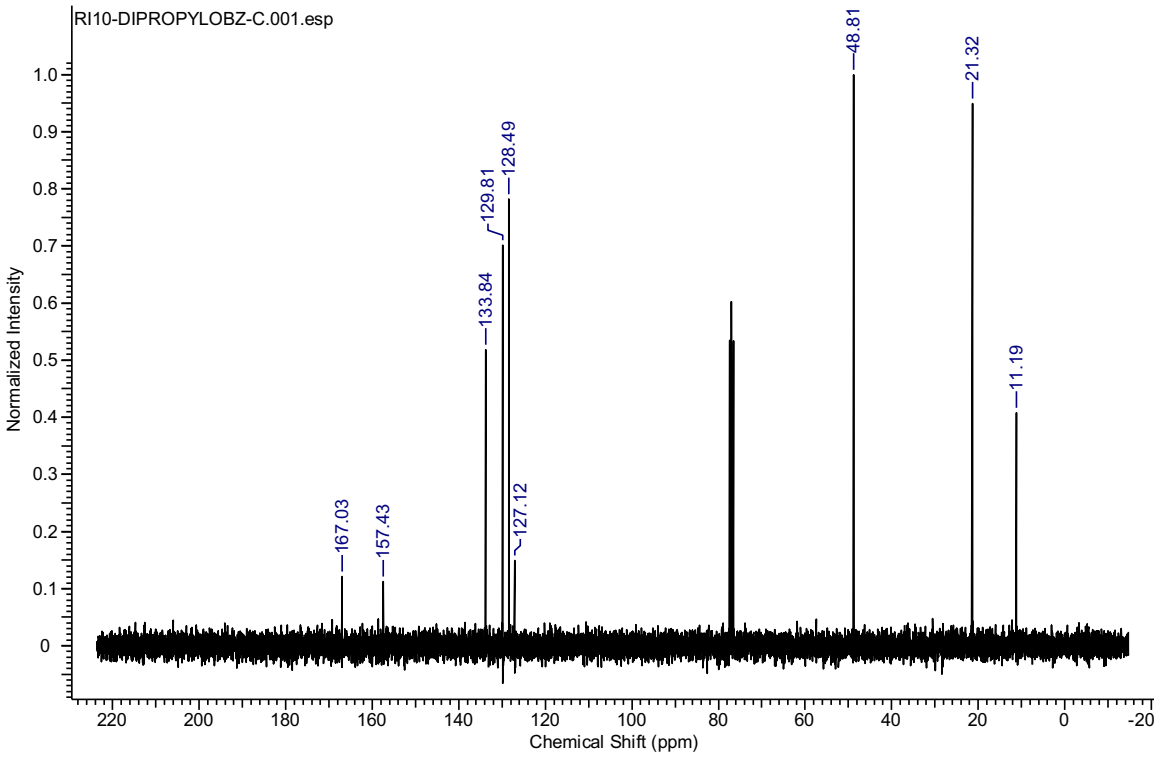

RI10-DIPROPYL-OAC.001.esp
O
N
N
H
OAc
4f
Normalized Intensity
0.87
3.92
2.68
4.01
6.00
Chemical Shift (ppm)

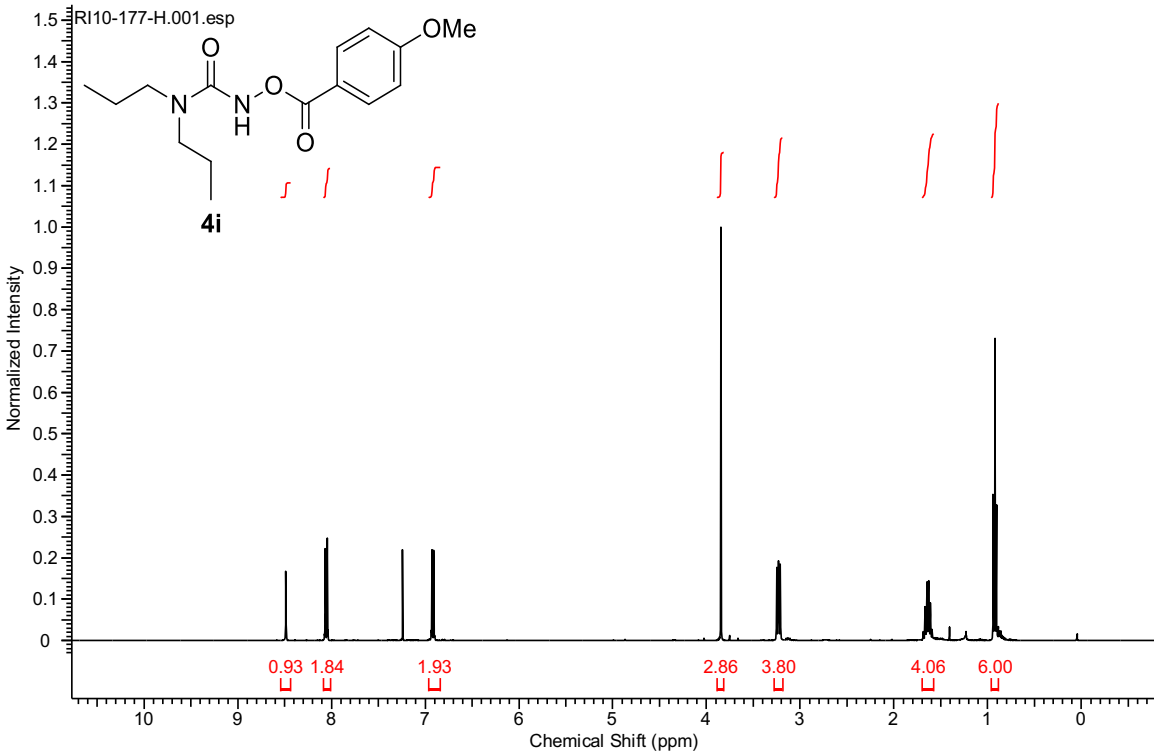

RI10-DIPROPYLOAC-C.001.esp
Normalized Intensity
170.91
157.37
48.73
21.25
18.43
11.11
Chemical Shift (ppm)

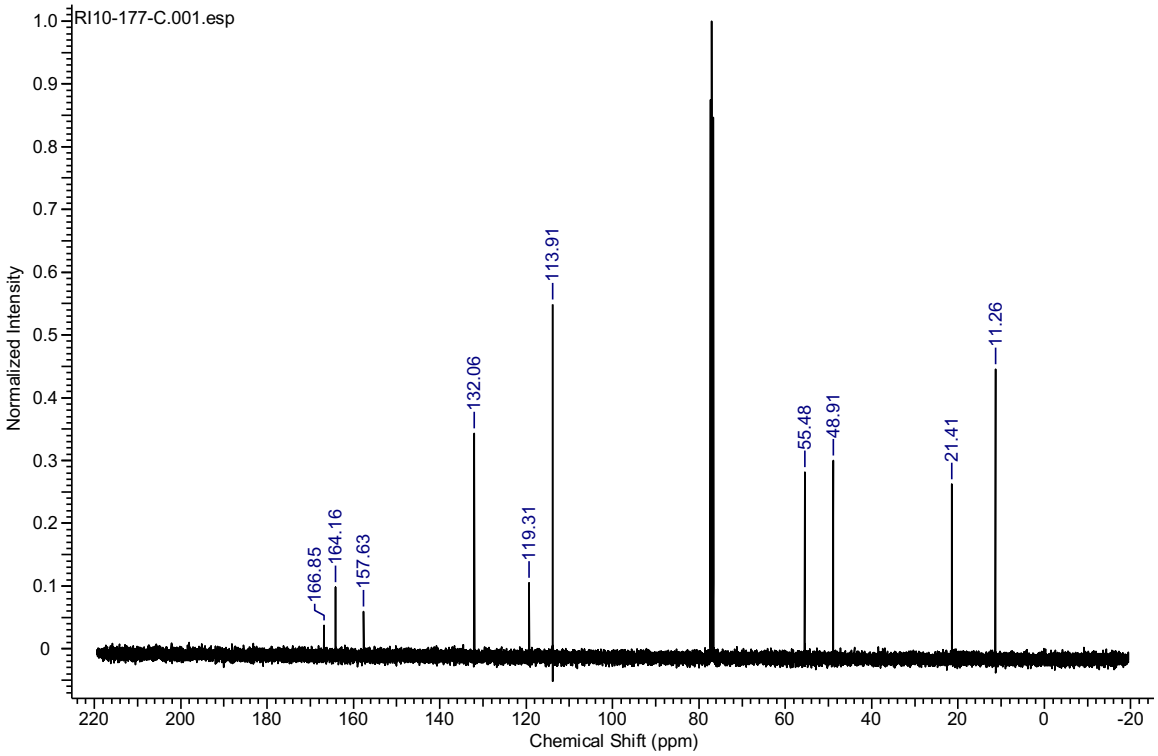

RI10-DIPROPYL-PIV-H.001.esp
O
N
N
H
OPiv
4g
Normalized Intensity
0.95
3.99
4.09
9.29
6.00
Chemical Shift (ppm)

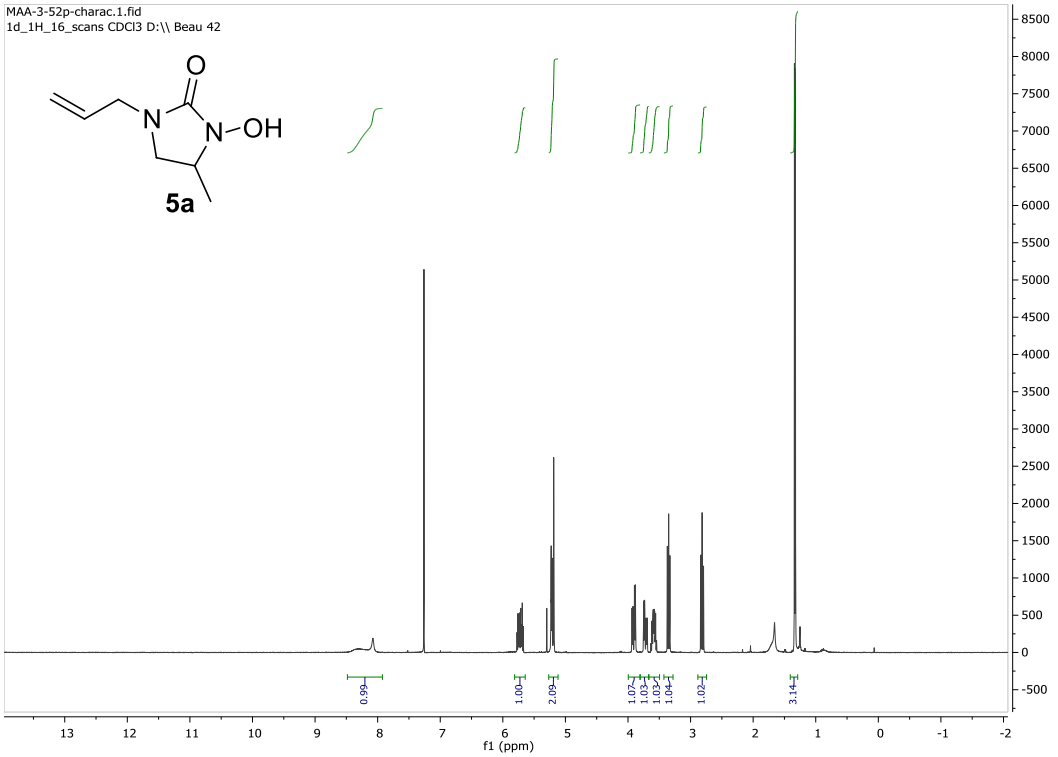

RI10-DIPROPYL-PIV-C.001.esp
178.88
157.46
48.75
38.25
26.95
21.29
11.19
Normalized Intensity
Chemical Shift (ppm)

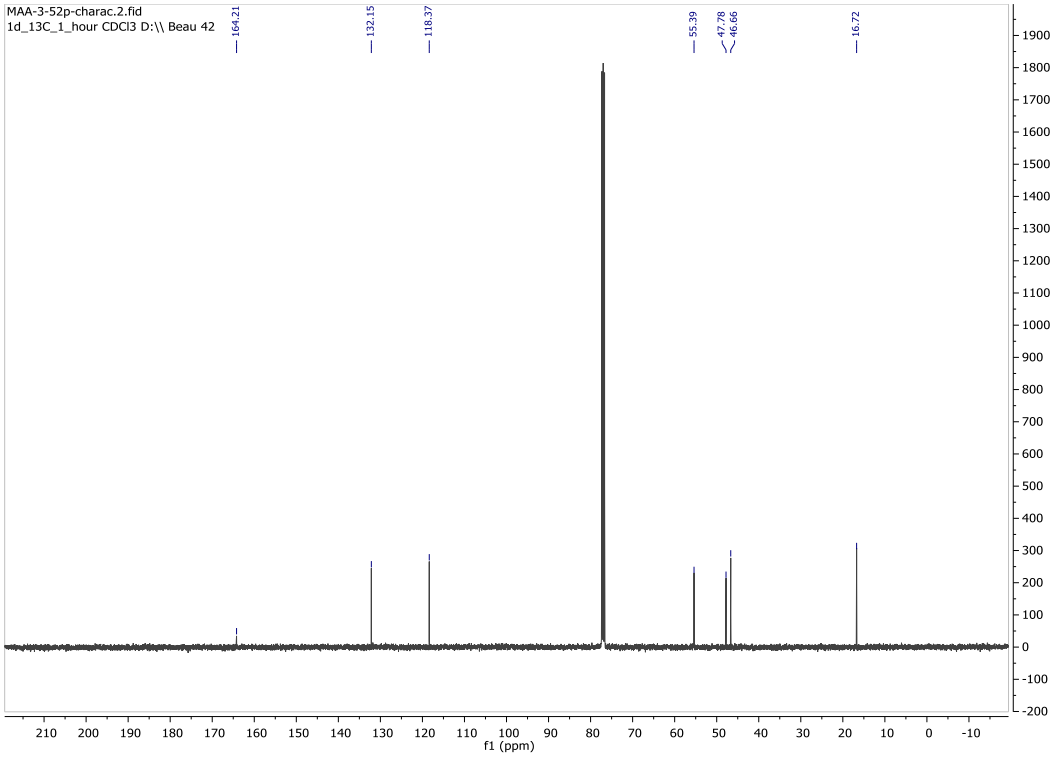

RI10-DIPROPYLOBZ.001.esp
O
N
N
H
OBz
4h
Normalized Intensity
0.99
1.93
0.98
2.00
3.99
4.05
6.03
Chemical Shift (ppm)

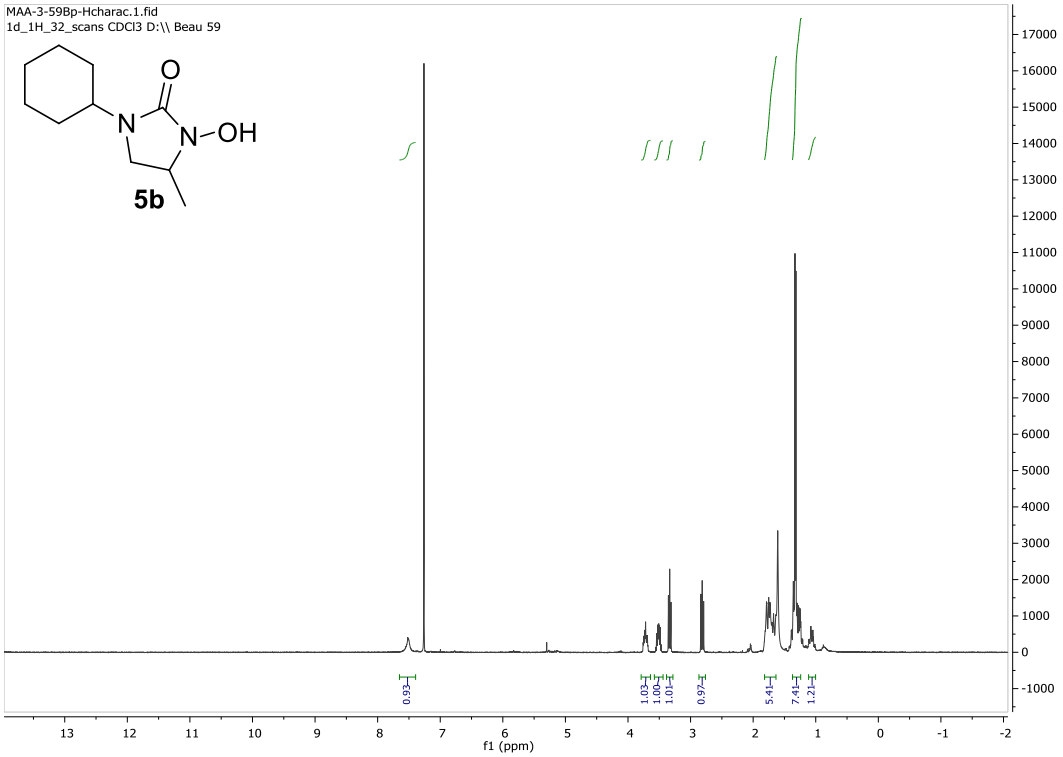

RI10-DIPROPYLOBZ-C.001.esp
48.81
21.32
129.81
128.49
133.84
11.19
167.03
157.43
127.12
Normalized Intensity
Chemical Shift (ppm)

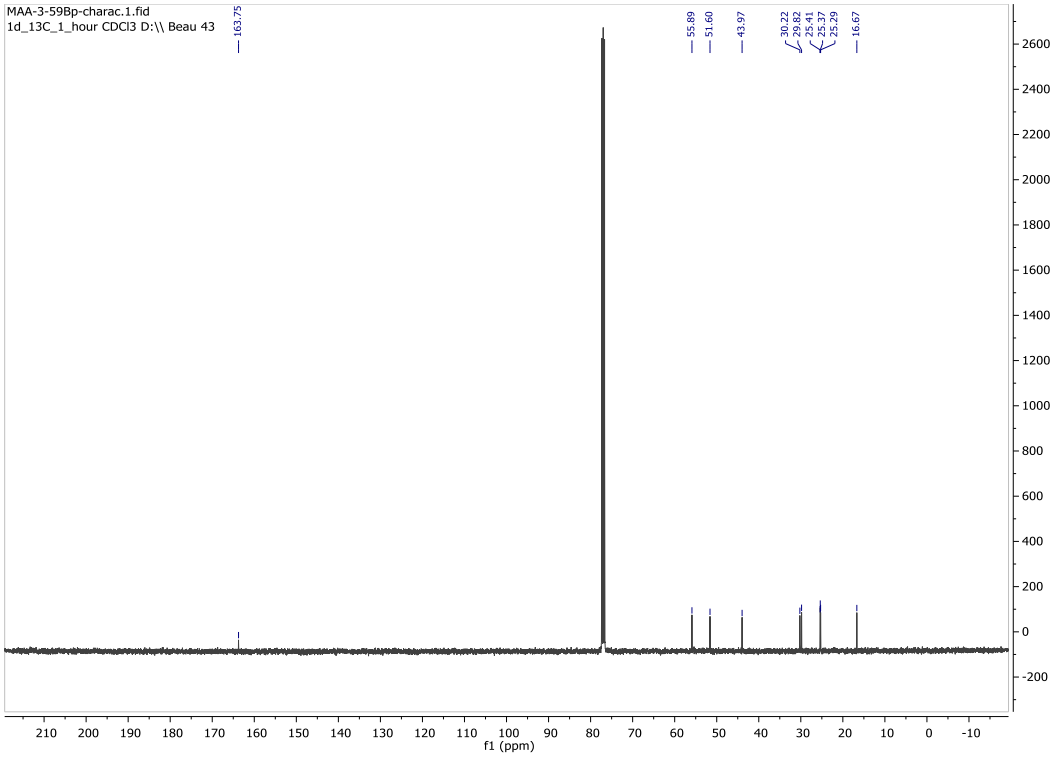

RI10-177-H.001.esp
OMe
O
N
N
H
O
O
4i
Normalized Intensity
Chemical Shift (ppm)
0.93
1.84
1.93
2.86
3.80
4.06
6.00

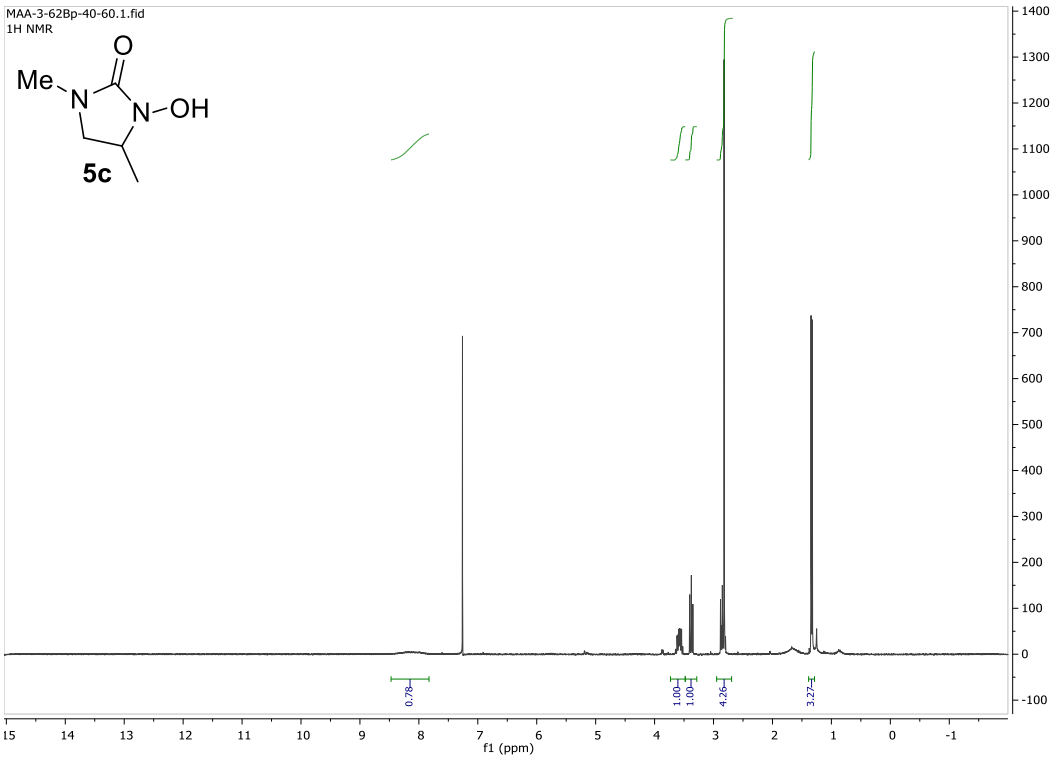

RI10-177-C.001.esp
Normalized Intensity
Chemical Shift (ppm)
166.85
164.16
157.63
132.06
119.31
113.91
55.48
48.91
21.41
11.26

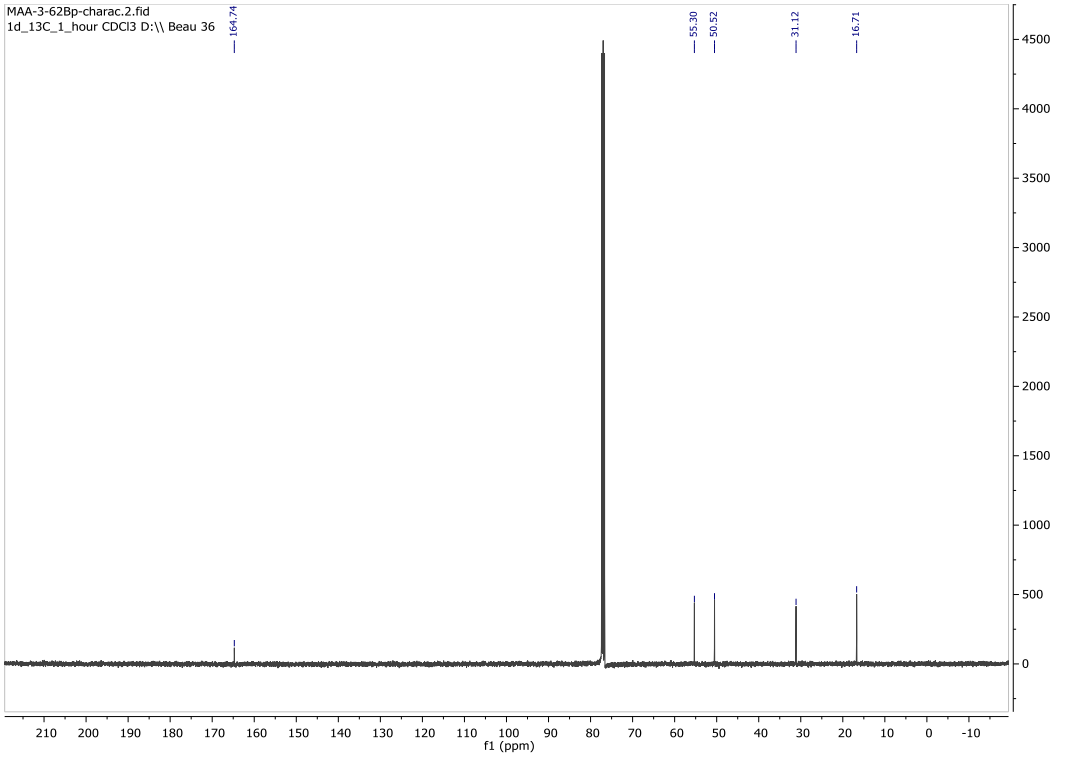

MAA-3-52p-charac.1.fid
1d_1H_16_scans CDCl3 D:\\ Beau 42
O
N
N–OH
5a
0.99
1.00
2.09
1.07
1.03
1.03
1.04
1.02
3.14
f1 (ppm)

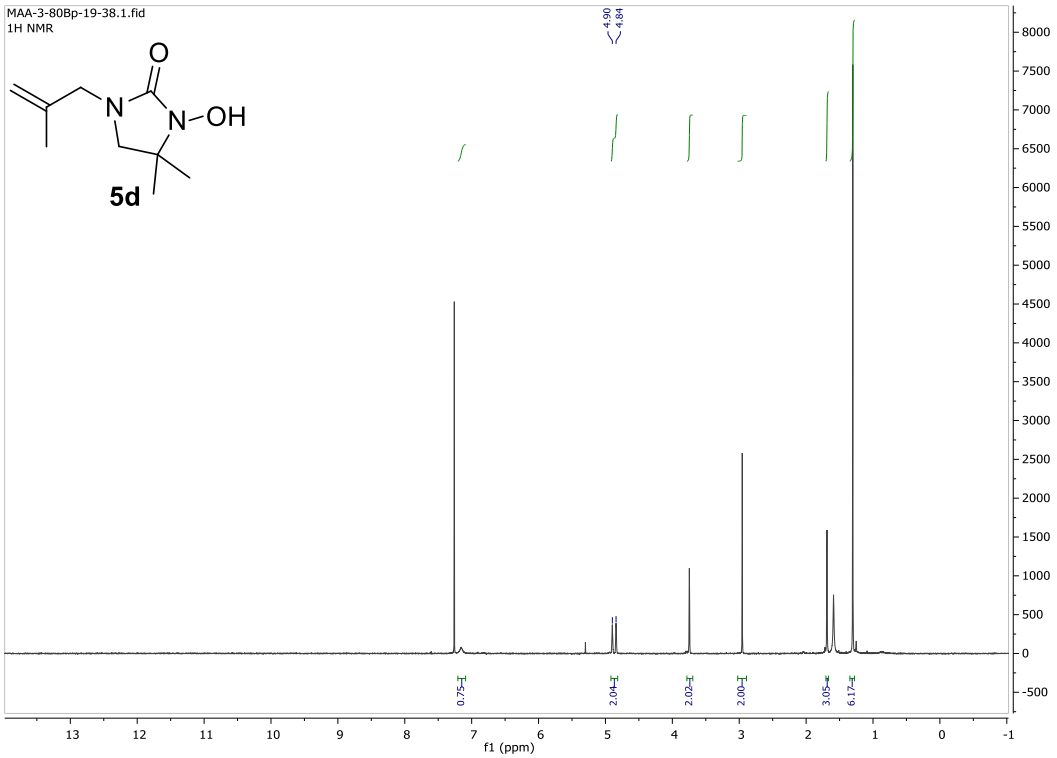

MAA-3-52p-charac.2.fid
1d_13C_1_hour CDCl3 D:\\ Beau 42
164.21
132.15
118.37
55.39
47.78
46.66
16.72
f1 (ppm)

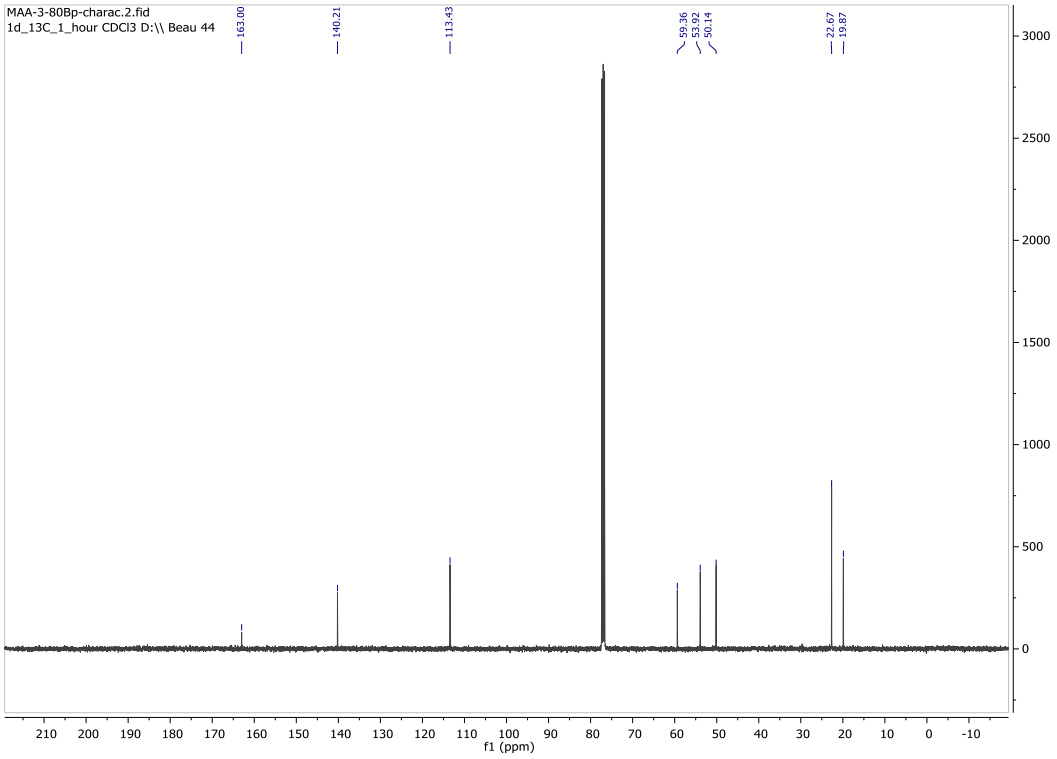

MAA-3-59Bp-Hcharac.1.fid
1d_1H_32_scans CDCl3 D:\\ Beau 59
O
N
N-OH
5b
0.93
1.03
1.00
1.01
0.97
5.41
7.41
1.21
f1 (ppm)

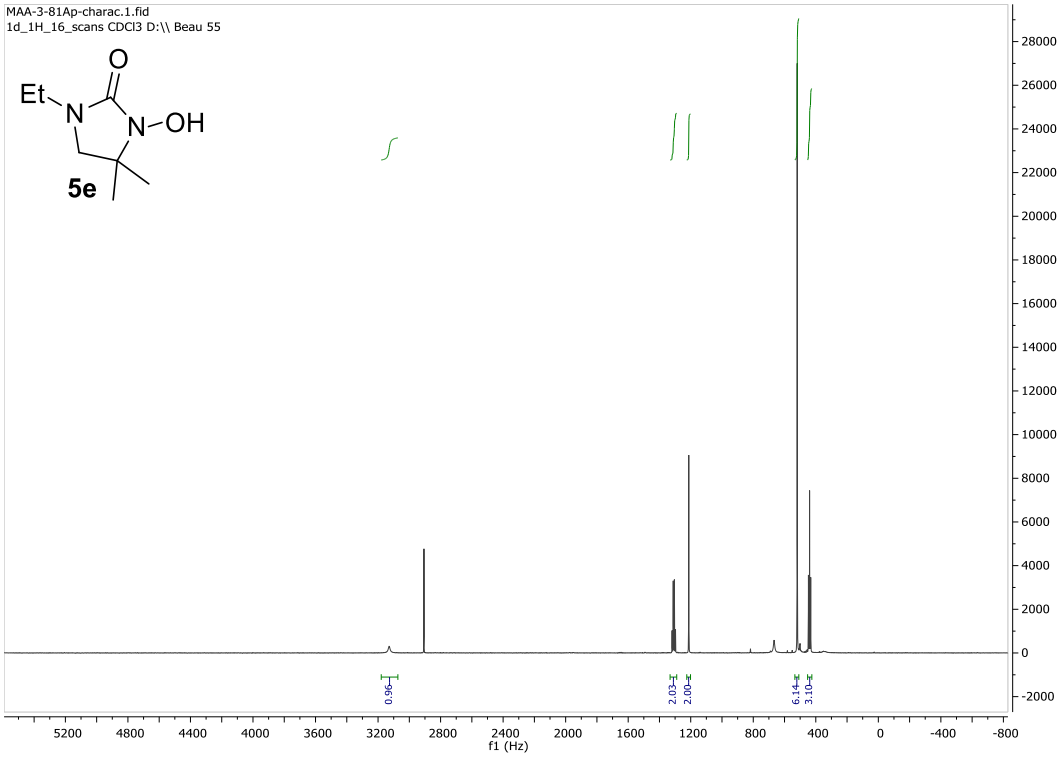

MAA-3-59Bp-charac.1.fid
1d_13C_1_hour CDCl3 D:\\ Beau 43
163.75
55.89
51.60
43.97
30.22
29.82
25.41
25.37
25.29
16.67
f1 (ppm)

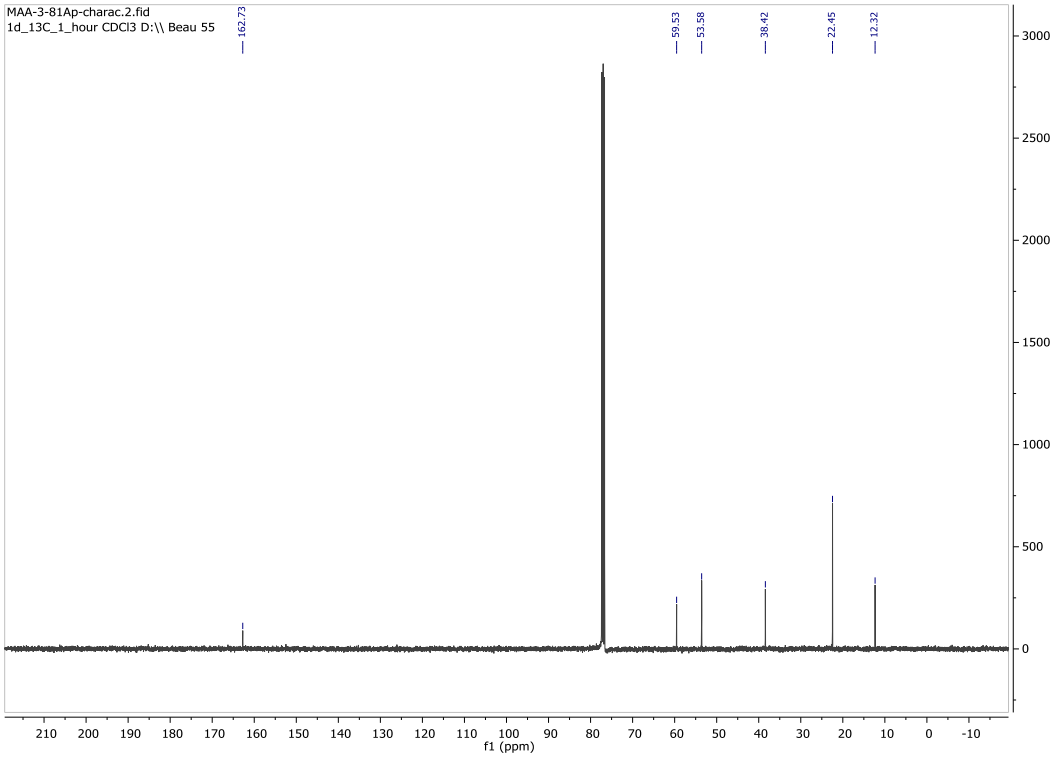

MAA-3-62Bp-40-60.1.fid
1H NMR
Me
N
O
N
OH
5c
0.78
1.00
1.00
4.26
3.27
f1 (ppm)

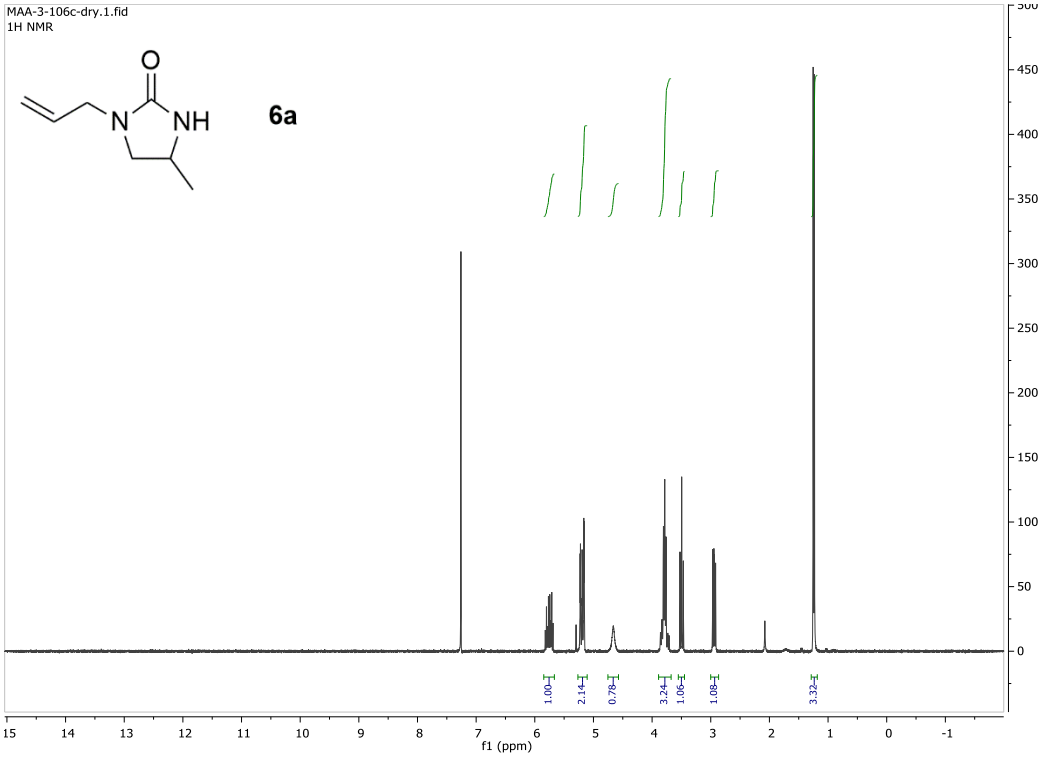

MAA-3-62Bp-charac.2.fid
1d_13C_1_hour CDCl3 D:\\ Beau 36
164.74
55.30
50.52
31.12
16.71
f1 (ppm)

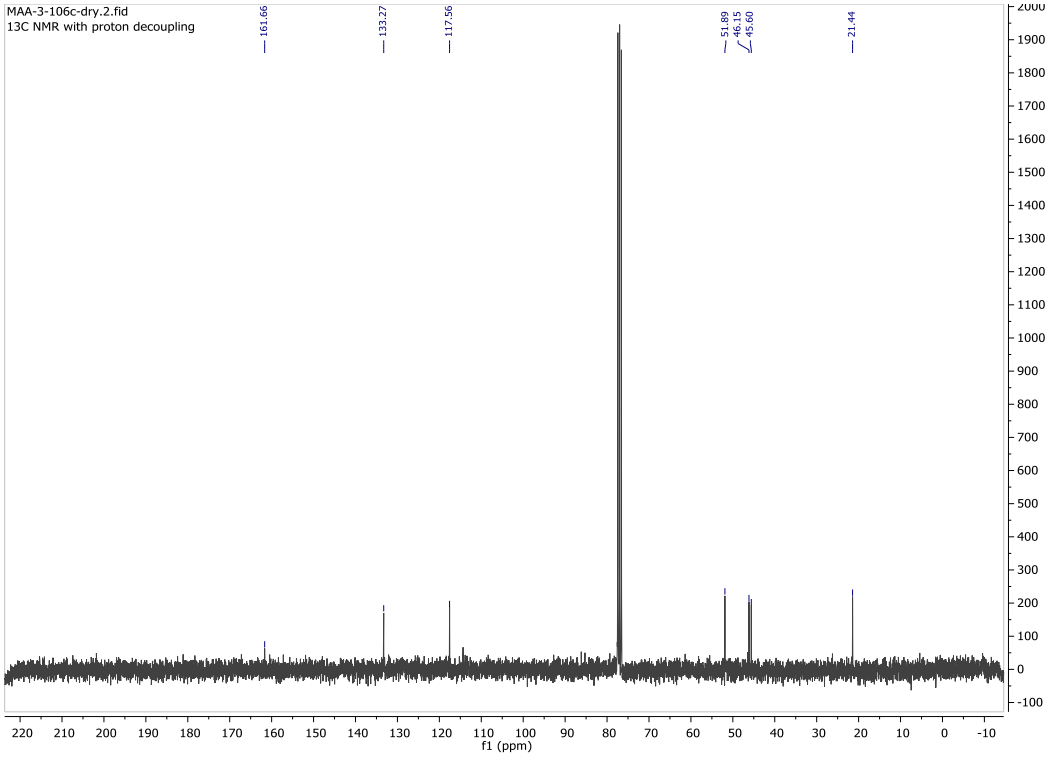

MAA-3-80Bp-19-38.1.fid
1H NMR
O
N
N
OH
5d
4.90
4.84
0.75
2.04
2.02
2.00
3.05
6.17
f1 (ppm)


MAA-3-80Bp-charac.2.fid
1d_13C_1_hour CDCl3 D:\\ Beau 44
163.00
140.21
113.43
59.36
53.92
50.14
22.67
19.87
f1 (ppm)


MAA-3-81Ap-charac.1.fid
1d_1H_16_scans CDCl3 D:\\ Beau 55
Et
N
O
N
OH
5e
0.96
2.03
2.00
6.14
3.10
f1 (Hz)


MAA-3-81Ap-charac.2.fid
1d_13C_1_hour CDCl3 D:\\ Beau 55
162.73
59.53
53.58
38.42
22.45
12.32
f1 (ppm)


MAA-3-106c-dry.1.fid
1H NMR
O
N
NH
6a
1.00
2.14
0.78
3.24
1.06
1.08
3.32
f1 (ppm)


MAA-3-106c-dry.2.fid
13C NMR with proton decoupling
161.66
133.27
117.56
51.89
46.15
45.60
21.44
f1 (ppm)