Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Robust Preparation of Imidazo[5,1-b][1,3,4]oxadiazoles

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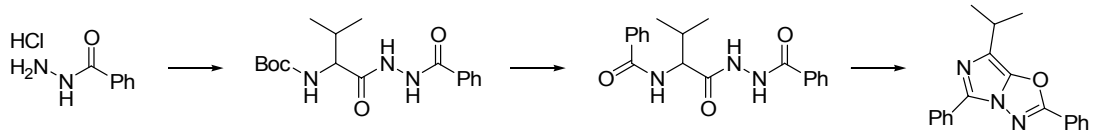
SUPPORTING INFORMATION

1. Experimental Section:

All reagents and solvents were used as purchased from commercial sources. Reactions were carried out under a blanket of nitrogen. Mass spectral data was collected on a Micromass ADM atmospheric pressure chemical ionization instrument (LRMS APCI). NMR spectra were generated on a Varian 400 MHz and 500 MHz instruments. Chemical shifts were recorded in ppm relative to tetramethylsilane (TMS) with multiplicities given as s (singlet), bs (broad singlet), d (doublet), t (triplet), dd (doublet of doublets), dt (double of triplets), and m (multiplet). IR spectra were recorded on a Thermo-Electron/Nicolet Avatar 360 FT-IR Spectrometer. High-resolution mass spectra (HRMS) were measured on an Agilent LC-MS TOF on a Zorbax Eclipse 50 x 4.6 mm 1.8 Micron XDB-C18 column. Low resolution mass spectra were determined on a Waters/Micromass system. GC/MS were determined on an Agilent 6890/5973 GC/MS system in EI mode. The X-ray diffraction measurements were carried out at 298K on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and sealed tube Cu radiation (1.54178 Å) source. Melting points are uncorrected. Compound purity was determined by combustion analysis (Quantitative Technologies Inc.) or high pressure liquid chromatography (HPLC). HPLC conditions utilized are as follows. Gradient: 0 – 1.5 min 5% Acetonitrile (ACN)/water, 1.5 – 10 min 5 – 100% ACN/water, 10 – 11 min 100% ACN, 11 – 12.5 min 100 – 5% ACN/water; UV detector: 254 nM. Retention times (RT) are in minutes and purity is calculated as % total area. (Column: XBridge C18 5 µ (4.6 mm x 150 mm); mobile phase: flow rate of 1.5 mL/min with solvent containing 0.1% TFA.



2b **1b**

7-Isopropyl-2,5-diphenyl-imidazo[5,1-b][1,3,4]oxadiazole 1b

[1-(N'-Benzoyl-hydrazinocarbonyl)-2-methyl-propyl]-carbamic acid tert-butyl ester. To a solution of the benzoic hydrazide HCl (1.46 g, 8.5 mmol), Boc-DL-Val-OH (2.79 g, 12.8 mmol) and TEA (2.2 mL, 16.0 mmol) in THF/DMF (4:1, 25 mL) was added EDCI (2.66 g, 13.9 mmol). After stirring 16 h, the reaction mixture was diluted with CH_2Cl_2 and washed with sat'd $NaHCO_3$ and H_2O. The organic phase was dried ($MgSO_4$), and concentrated. The crude product was triturated with ether/heptane, filtered and dried under vacuum to afford 1.82 g (64%) of [1-(N'-benzoyl-hydrazinocarbonyl)-2-methyl-propyl]-carbamic acid tert-butyl ester as a beige solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.3 Hz, 2 H), 7.60-7.54 (m, 1 H), 7.51-7.44 (m, 2 H), 5.07 (bs, 1 H), 4.12 (app s, 1 H), 2.29-2.22 (m, 1 H), 1.47 (s, 9 H), 1.04-0.98 (dd, *J*= 6.8; 20.4 Hz, 6 H). MS (M-H): 334.

N-[1-(N'-Benzoyl-hydrazinocarbonyl)-2-methyl-propyl]-benzamide 2b. [1-(N'-benzoyl-hydrazinocarbonyl)-2-methyl-propyl]-carbamic acid tert-butyl ester (1.68 g, 5.0

mmol) was stirred in 10% HCl ethanolic solution (20 mL). After 4h, the reaction mixture was concentrated to afford the amine-HCl (1.31 g) as a white solid which was used without further purification. To a solution of the amine (0.50 g), benzoic acid (0.27 g, 2.2 mmol) and TEA (0.38 mL, 2.7 mmol) in THF/DMF (2.5:1, 14 mL) was added EDCI (0.46 g, 2.4 mmol). After 3 h, the reaction mixture was diluted with CH_2Cl_2 and washed with sat'd $NaHCO_3$ and H_2O. The organic phase was concentrated and purified via flash column chromatography (0 → 10% MeOH/EtOAc) to afford 0.49 g (79%) of **2b** as a white solid. Melting point: 231 – 233 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.51 (bs, 1 H), 8.87 (bs, 1 H), 7.79-7.71 (m, 4 H), 7.56-7.44 (m, 3 H), 7.41-7.36 (m, 3 H), 6.88 (d, *J* = 8.7 Hz, 1 H), 4.63 (t, *J* = 8.4 Hz, 1 H), 2.33-2.26 (m, 1 H), 1.05 (apparent t, *J* = 5.4 Hz, 6 H). IR (thin film): 3324, 2945, 2832, 1642, 1449, 1021 cm^{-1}. MS (M+H): 340. HPLC: 6.83 min. Anal. Calcd. for $C_{19}H_{21}N_3O_3$: C, 67.24; H, 6.24; N, 12.38. Found: C, 66.80; H, 6.34; N, 12.38.

7-Isopropyl-2,5-diphenyl-imidazo[5,1-b][1,3,4]oxadiazole 1b. A solution of the hydrazide (0.14 g) in $POCl_3$ (2 mL) and acetonitrile (5 mL) was heated to reflux (110 °C). After heating 16 h, the reaction mixture was cooled to ambient temp. and concentrated. The residue was taken up in CH_2Cl_2 and washed with sat'd Na_2CO_3 and H_2O. The organic phase was concentrated. Purification via flash column chromatography (25 → 50% EtOAc/heptane) afforded 0.13 g (73%) of **1b** as a yellow residue. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (d, *J*= 7.3 Hz, 2 H), 8.15-8.13 (m, 2H), 7.64-7.60 (m, 1 H), 7.58-7.55 (m, 2 H), 7.48-7.44 (m, 2 H), 7.35-7.31 (m, 1 H), 3.15-3.12 (m, 1 H), 1.46 (d, *J* = 7.0 Hz, 6 H). ^{13}C NMR (125 MHz) δ 132.8, 129.3, 128.8, 127.3, 125.1, 27.1, 22.4. MS (M+H): 304. Anal. Calcd. for $C_{19}H_{17}N_3O_1$ (0.26 eq CH_2Cl_2): C, 71.08; H, 5.43; N, 12.91. Found: C, 71.14; H, 5.48; N, 12.75 (in some instances CH_2Cl_2 was used to prepare the samples for combustion analysis, which was reflected in the experimental data obtained and is corrected as such).

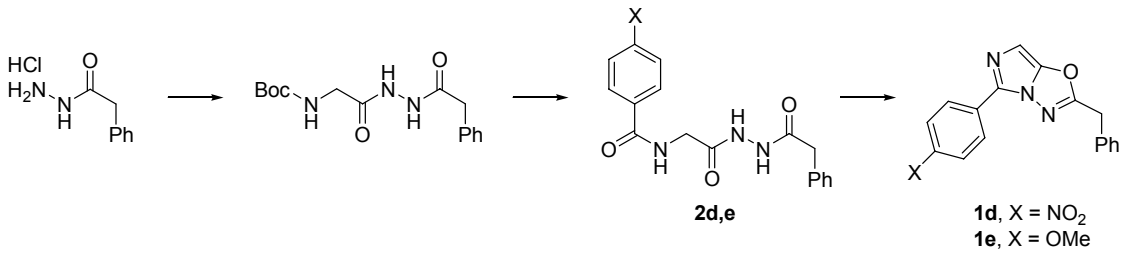
7-Benzyl-2,5-diphenyl-imidazo[5,1-b][1,3,4]oxadiazole 1c (prepared according to the procedure for **1b**).

[2-(N'-Benzoyl-hydrazino)-1-benzyl-2-oxo-ethyl]-carbamic acid tert-butyl ester. Prepared from benzoic hydrazide HCl (0.50 g, 2.9 mmol) and Boc-Phe-OH (1.2 g, 4.4 mmol) to afford 0.96 g (86%) of the carbamate as a white grainy solid. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.41 (bs, 1 H), 10.12 (bs, 1 H), 7.86-7.84 (m, 2 H), 7.55-7.51 (m, 1 H), 7.47-7.45 (m, 2 H), 7.33-7.28 (m, 2 H), 7.26-7.21 (m, 2 H), 7.19-7.16 (m, 1 H), 6.96 (d, *J* = 8.4 Hz, 1 H), 4.27-4.20 (m, 1 H), 3.09-3.03 (m, 1 H), 2.78-2.74 (m, 1 H), 1.04 (s, 9 H). MS (M+H): 384.

N-[2-(N'-Benzoyl-hydrazino)-1-benzyl-2-oxo-ethyl]-benzamide 2c. Prepared from [2-(N'-benzoyl-hydrazino)-1-benzyl-2-oxo-ethyl]-carbamic acid tert-butyl ester (0.40 g, 1.1 mmol) to afford 0.47 g (58%) of **2c** as a white solid. Melting point: 219 – 221 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.41 (bs, 1 H), 10.25 (bs, 1 H), 8.63 (d, *J* = 8.5 Hz, 1 H), 7.89-7.84 (m, 2 H), 7.76-7.73 (m, 2 H), 7.55-7.51 (m, 1 H), 7.47-7.36 (m, 7 H), 7.25-7.21 (m, 2 H), 7.14-7.11 (m, 1 H), 4.84-4.77 (m, 1 H), 3.26-3.21 (m, 1 H), 3.21-3.18 (m, 1 H). IR (thin film): 3246, 1644, 1578, 1535, 697 cm^{-1}. MS (M+H): 388. HPLC: 7.50 min.

Anal. Calcd. for $C_{23}H_{21}N_3O_3$ (0.1 eq. H_2O): C, 70.97; H, 5.49; N, 10.80. Found: C, 70.65; H, 5.57; N, 10.89.

7-Benzyl-2,5-diphenyl-imidazo[5,1-b][1,3,4]oxadiazole 1c. Prepared from **2c** (0.09 g, 0.24 mmol) and purified via flash column chromatography (50 → 75% EtOAc/heptane) to afford 66 mg (80%) of **1c** as a yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24-8.22 (m, 2 H), 8.04-8.02 (m, 2 H), 7.61-7.59 (m, 1 H), 7.54-7.52 (m, 2 H), 7.48-7.43 (m, 2 H), 7.43-7.41 (m, 2 H), 7.37-7.33 (m, 3 H), 7.27-7.24 (m, 1 H), 4.16 (s, 2 H). IR (thin film): 3179, 3063, 1661, 1606, 1451, 1275, 1173, 693 cm^{-1}. MS (M+H): 352. GC-MS: 7.36 min with m/z: 351. HPLC: 8.97 min.



(N'-Phenylacetyl-hydrazinocarbonylmethyl)-carbamic acid tert-butyl ester: To a solution of phenylacetic hydrazide HCl (1.0 g, 6.7 mmol), Boc-Gly-OH (1.40 g, 8.0 mmol), and triethylamine (1.39 mL, 10.0 mmol) in THF/DMF (4:1, 25 mL) was added EDCI (1.30 g, 8.7 mmol). After stirring 3 d, the reaction mixture was diluted with CH_2Cl_2 and washed with sat. $NaHCO_3$ and water. The organic phase was separated, dried ($MgSO_4$), and concentrated. Purification via flash column chromatography (0 → 5% MeOH/EtOAc) afforded 1.03 g (51%) of (N'-phenylacetyl-hydrazinocarbonylmethyl)-carbamic acid tert-butyl ester as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (bs, 1 H), 8.04 (bs, 1 H), 7.31-7.25 (m, 5 H), 5.18 (bs, 1 H), 3.82 (d, J = 6.1 Hz, 2 H), 3.59 (s, 2 H), 1.41 (s, 9 H). MS (M+H): 308.

N-[2-Oxo-2-(N'-phenylacetylhydrazino)-ethyl]-4-nitrobenzamide 2d. N'-Phenylacetylhydrazinocarbonylmethyl)-carbamic acid tert-butyl ester (0.70 g, 2.0 mmol) was stirred in 21% HCl ethanolic solution (20 mL). After 2h, solvent was removed to 50% volume and then ether added and stirred for 10 min. The mixture was then filtered and washed with ether and heptane to afford 0.44 g (75%) of phenylacetic acid N'-(2-aminoacetyl)-hydrazide. To a solution of the crude hydrazide (0.10 g, 0.41 mmol) and 4-nitrobenzoic acid (0.07 g, 0.41 mmol) in DMF (1mL) was added TPTU (0.14 g, 0.45 mmol) followed by DIEA (0.21 mL, 1.23 mmol). After stirring 1 h, the mixture was diluted with water (5 mL) and EtOAc (5 mL), and the precipitate filtered and dried to afford 0.06 g (40%) of **2d** as a pale yellow solid which was carried on to the next step without further purification. Melting point: 267 – 269 ºC. ^{1}H NMR (500 MHz ,DMSO-d_6) δ 9.14 (t, 1H), 8.36 - 8.26 (m, J = 8.8 Hz, 2 H), 8.15 - 8.04 (m, J = 8.8 Hz, 2 H), 7.35 - 7.16 (m, 5 H), 3.96 (d, J = 6.1 Hz, 2 H), 3.46 (s, 2 H). IR (thin film): 3211, 1646, 1593, 1513, 1352, 563 cm^{-1}. MS: 357.0 (M+H), 355.1 (M-H). HRMS Calcd. for $C_{17}H_{17}N_4O_5$,

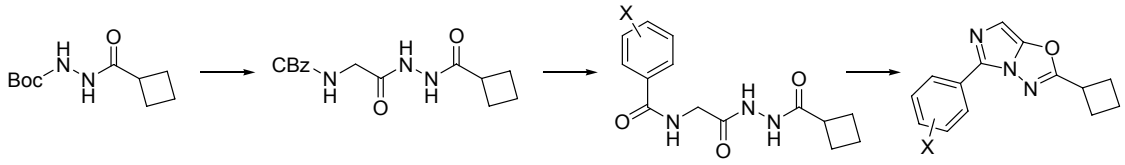
357.1193. Found, 357.1198. HPLC: 6.37 min. Anal. Calcd. for $C_{17}H_{16}N_4O_5$ (0.3 eq. H_2O): C, 56.44; H, 4.63. Found: C, 56.09; H, 4.48.

2-Benzyl-5-(4-nitophenyl)-imidazo[5,1-b][1,3,4]oxadiazole 1d: A solution of N-[2-oxo-2-(N'-phenylacetylhydrazino)-ethyl]-4-nitrobenzamide **2d** (0.06 g) in $POCl_3$ (1 mL) and acetonitrile (2 mL) was heated to reflux (110 °C). After heating 3 h, the mixture was cooled to ambient temp. and concentrated. The residue was taken up in CH_2Cl_2 and washed with sat. Na_2CO_3 (aq) and water. The organic phase was dried (Na_2SO_4) and concentrated. Purification via flash column chromatography (5 → 50% EtOAc/heptane) afforded 0.035 g (65%) of 2-benzyl-5-(4-nitophenyl)-imidazo[5,1-b][1,3,4]oxadiazole **1d** as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (s, 4 H), 7.32 – 7.42 (m, 5H), 6.72 (s, 1H), 4.21 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 147.4, 147.0, 134.8, 132.4, 129.4, 129.2, 128.4, 127.3, 125.2, 124.5, 98.7, 33.6. IR (thin film): 2921, 1506, 1334, 1084, 570 cm^{-1}. MS (M+H): 321. HPLC: 8.97 min. Anal. Calcd. for $C_{17}H_{12}N_4O_3$: C, 63.75; H, 3.78; N, 17.49. Found: C, 63.63; H, 3.51; N, 17.36.

2-Benzyl-5-(4-methoxyphenyl)-imidazo[5,1-b][1,3,4]oxadiazole 1e (prepared according to the procedure for **1d**).

N-[2-Oxo-2-(N'-phenylacetylhydrazino)-ethyl]-4-methoxybenzamide 2e: Prepared from phenylacetic acid N'-(2-aminoacetyl)-hydrazide (0.10 g, 0.41 mmol) and 4-methoxybenzoic acid (0.06 g, 0.41 mmol) to afford 0.075 g (54%) of **2e** as a white solid which was carried on to the next step without further purification, mp = 225 – 227 ºC: ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.07 (s, 1 H), 9.94 (s, 1 H), 8.61 (t, *J* = 5.7 Hz, 1 H), 7.84 (d, *J* = 8.8 Hz, 2 H), 7.33 - 7.18 (m, 5 H), 6.99 (d, *J* = 8.5 Hz, 2 H), 3.90 (d, *J* = 5.9 Hz, 2 H), 3.80 (s, 3 H), 3.45 (s, 2 H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 169.5, 168.8, 166.6, 162.3, 136.5, 129.9, 129.7, 128.9, 127.1, 126.9, 114.1, 56.0, 46.3, 41.7. IR (thin film): 3212, 1634, 1592, 572 cm^{-1}. MS: 342.2 (M+H). HPLC: 6.26 min. HRMS Calcd. for $C_{18}H_{20}N_3O_4$: 342.1448. Found: 342.1442.

2-Benzyl-5-(4-methoxyphenyl)-imidazo[5,1-b][1,3,4]oxadiazole 1e: Prepared from N-[2-oxo-2-(N'-phenylacetylhydrazino)-ethyl]-4-methoxybenzamide **2e** (0.075 g, 0.22 mmol) to yield 0.05 g (76%) of **1e** as a yellow solid, mp = 103 – 105 ºC: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.8 Hz, 2 H), 7.38 (s, 5 H), 6.99 (d, *J* = 8.8 Hz, 2 H), 6.56 (s, 1 H), 4.16 (s, 2 H), 3.85 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.9, 147.0, 146.2, 134.8, 132.9, 129.33, 129.30, 128.2, 126.6, 123.8, 114.4, 95.8, 55.6, 33.5. IR (thin film): 2929, 1732, 1570, 1538, 1252, 1178, 992, 566 cm^{-1}. MS (M+H): 306. HPLC: 7.19 min. Anal. Calcd. for $C_{18}H_{15}N_3O_2$: C, 70.81; H, 4.95; N, 13.76. Found: C, 69.83; H, 5.15; N, 10.94.



N'-Cyclobutanecarbonyl-hydrazinecarboxylic acid tert-butyl ester. Prepared from tert-butyl carbazate (3.0 g, 22.7 mmol) and cyclobutanecarboxylic acid (2.7 g, 27.2 mmol) to afford 4.58 g (94%) of N'-cyclobutanecarbonyl-hydrazinecarboxylic acid tert-butyl ester as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (bs, 1 H), 6.47 (bs, 1 H), 3.06 (quint., *J* = 8.4 Hz, 1 H), 2.39-2.31 (m, 2 H), 2.23-2.11 (m, 2 H), 2.04-1.84 (m, 2 H), 1.47 (s, 9 H). MS (M+H): 115.

4-(N'-Cyclobutanecarbonyl-hydrazino)-4-oxo-butyric acid benzyl ester. (Prepared according to the procedure for **2b**). ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.67 (bs, 2 H), 7.43 (t, *J* = 6.2. 1 H), 7.31-7.25 (m, 5 H), 4.97 (s, 2 H), 3.61 (d, *J* = 6.1 Hz, 2 H), 3.01 (quint., *J* = 8.3 Hz, 1 H), 2.11-1.97 (m, 4 H), 1.88-1.84 (m, 1 H), 1.78-1.71 (m, 1 H). MS (M-H): 303.

Cyclobutanecarboxylic acid N'-(2-amino-acetyl)-hydrazide. A solution of 4-(N'-cyclobutanecarbonyl-hydrazino)-4-oxo-butyric acid benzyl ester (0.55 g, 1.8 mmol) and 10% Pd/C (100mg) in MeOH (25mL) in a Parr shaker was hydrogenated at 50 psi. After 3 h, the mixture was filtered through Celite, washed with MeOH, and concentrated to yield 0.32g (100%) of cyclobutanecarboxylic acid N'-(2-amino-acetyl)-hydrazide as a gum. ^{1}H NMR (500 MHz, DMSO-d_6) δ 3.31 (bs, 2 H), 3.07 (quint., *J* = 8.3 Hz, 1 H), 2.16-2.00 (m, 4 H), 1.95-1.87 (m, 1 H), 1.79-1.75 (m, 1 H). MS (M+H): 172.

N-[2-(N'-Cyclobutanecarbonylhydrazino)-2-oxoethyl]-4-nitrobenzamide 2f. To a solution of cyclobutanecarboxylic acid N'-(2-aminoacetyl)-hydrazide (0.50 g, 2.9 mmol) and 4-nitrobenzoic acid (0.49 g, 2.9 mmol) in DMF (5 mL) was added TPTU (0.98 g, 3.2 mmol) followed by DIEA (1.53 mL, 8.8 mmol). After stirring 16 h, the reaction mixture was diluted with water (10 mL), and pH was adjusted to 10 with 1N NaOH. The solution was extracted with ethyl acetate (3 x 75 mL), and the combined organics were washed with 1N LiCl (50 mL) followed by brine (50 mL), dried (Na_2SO_4), and concentrated. The residual oil was triturated from 10% CH_2Cl_2/ether, filtered, and dried to furnish 0.40 g (42%) of N-[2-(N'-cyclobutanecarbonylhydrazino)-2-oxoethyl]-4-nitrobenzamide **2f** as a white solid, mp = 257 – 260 ºC: ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.85 (s, 1 H), 9.59 (s, 1 H), 9.10 (t, *J* = 6 Hz, 1 H),.8.28 (d, *J* = 8.8 Hz, 2 H), 8.06 (d, *J* = 8.8 Hz, 2 H), 3.91 (d, *J* = 6.1 Hz, 2 H), 3.02 (m, 1 H), 2.08 (m, 2H), 2.0 (m, 2 H), 1.84 (m, 1H), 1.74 (m, 1H). IR (thin film): 3171, 1652, 1606, 1534, 714 cm^{-1}. MS (M+H): 321.1. HPLC: 5.74 min. HRMS Calcd. for $C_{14}H_{17}N_4O_5$: 321.1193. Found: 321.1190. Anal. Calcd. for $C_{14}H_{16}N_4O_5$: C, 52.50; H, 5.03; N, 17.49. Found: C, 52.11; H, 4.86; N, 17.30.

2-Cyclobutyl-5-(4-nitrophenyl)-imidazo[5,1-b][1,3,4]oxadiazole 1f: A solution of N-[2-(N'-cyclobutanecarbonylhydrazino)-2-oxoethyl]-4-nitrobenzamide **2f** (0.10 g, 0.3 mmol) in $POCl_3$ (1 mL) and MeCN (2 mL) was heated to 110 °C. After 2 h, the mixture was cooled and concentrated. The resulting residue was taken up in CH_2Cl_2 and washed with sat. Na_2CO_3 (aq) and water. The organic phase was dried (Na_2SO_4), and concentrated. Purification via flash column chromatography (5 → 50% EtOAc/heptane) afforded 45 mg (50%) of 2-cyclobutyl-5-(4-nitrophenyl)-imidazo[5,1-b][1,3,4]oxadiazole **1f** as a yellow solid, mp = 161-163 °C: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 (s, 4 H), 6.73 (s, 1 H), 3.72 (quint., *J* = 8.3 Hz, 1 H), 2.45 – 2.60 (m, 4 H), 2.19 (m, 1 H), 2.11 (m, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.8, 147.5, 146.8, 134.9, 127.1, 125.0, 124.4, 98.6, 31.8, 26.7, 19.1. MS (M+H): 285. IR: 2992, 2953, 1504, 1329, 1088, 852 cm^{-1}. Anal. Calcd. for $C_{14}H_{12}N_4O_3$ (0.2 eq CH_2Cl_2): C, 56.61; H, 4.15; N, 18.60. Found: C, 56.80; H, 3.96; N, 18.65.*

* Elemental analysis corresponds to proton spectrum for compound **1f** dated Oct. 14, 2008 that contained trace amount of CH_2Cl_2. In order to obtain additional characterization, carbon spectrum was obtained after sample had been dried thoroughly (dated Feb. 5, 2009). This trend holds true for other examples.

2-Cyclobutyl-5-(3,5-dibromophenyl)-imidazo[5,1-b][1,3,4]oxadiazole 1g (prepared according to the procedure for **1f**).

N-[2-(N'-Cyclobutanecarbonylhydrazino)-2-oxoethyl]- 3,5-dibromobenzamide 2g: Prepared from cyclobutanecarboxylic acid N'-(2-aminoacetyl)-hydrazide (0.40 g, 2.3 mmol) and 3,5-dibromobenzoic acid (0.65 g, 2.3 mmol) and purified by trituration from 10% CH_2Cl_2:ether, filtered to afford 0.35 g (25%) of **2g** as a white powder, mp = 229 - 231 °C. ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.86 (bs, 1 H), 9.61 (bs, 1 H), 9.05 (t, *J* = 5.6 Hz, 1 H), 8.05 (s, 3 H), 3.90 (d, *J* = 5.8 Hz, 2 H), 3.05 (quint., *J* = 8.3 Hz, 1 H), 2.12 (m, 2 H), 2.02 (m, 2 H), 1.90 (m, 1 H), 1.77 (m, 1 H). IR (thin film): 3265, 1705, 1654, 1543, 1326, 744, 666, 626 cm^{-1}. MS: 434.0 (M+H). HRMS Calcd. For $C_{14}H_{16}N_3O_3Br_2$: 431.9552. Found: 431.9570. HPLC: 7.09 min, 95% pure.

2-Cyclobutyl-5-(3,5-dibromophenyl)-imidazo[5,1-b][1,3,4]oxadiazole 1g: Prepared from N-[2-(N'-cyclobutanecarbonylhydrazino)-2-oxoethyl]-3,5-dibromobenzamide **2g** (0.10 g, 0.3 mmol) and purified via flash column chromatography (5 → 50% EtOAc/heptane) to afford 80 mg (80%) of **1g** as a pale yellow solid. ^{1}H NMR (500 MHz, CD_3OD) δ 8.31 (s, 2 H), 8.05 (s, 1 H), 7.61, (s, 1 H), 4.01 (m, 1 H), 2.86 – 2.58 (m, 4 H), 2.33 – 2.23 (m, 1 H), 2.19 – 2.10 (m, 1H). ^{13}C NMR (126 MHz , CD_3OD) δ 175.9, 145.2, 137.0, 128.0, 125.4, 124.0, 94.0, 31.6, 26.3, 18.7. MS (M+H): 397.8. HPLC: 10.767 min. IR: 3114, 2953, 1647, 1575, 1553, 1210, 1046, 1010, 939, 860, 744 cm^{-1}.

2,5,7-Triphenyl-imidazo[5,1-b][1,3,4]oxadiazole 1h (prepared according to the procedure for **1b**)

[2-(N'-Benzoyl-hydrazino)-2-oxo-1-phenyl-ethyl]-carbamic acid tert-butyl ester. Prepared from benzoic hydrazide HCl (0.57 g, 3.3 mmol) and Boc-Phg-OH (0.75 g, 3.0 mmol) to afford 0.54g (49%) of the carbamate as a white solid. ^{1}H NMR (500 MHz,

DMSO-d_6) δ 10.34 (bs, 1 H), 7.82-7.79 (m, 2 H), 7.53-7.38 (m, 5 H), 7.32-7.30 (m, 2 H), 7.28-7.22 (m, 1 H), 5.32 (d, *J* = 8.4 Hz, 1 H), 1.35 (s, 9 H). MS (M-H): 368.

N-[2-(N'-Benzoyl-hydrazino)-2-oxo-1-phenyl-ethyl]-benzamide 2h. Prepared from [2-(N'-benzoyl-hydrazino)-2-oxo-1-phenyl-ethyl]-carbamic acid tert-butyl ester (0.47 g, 1.5 mmol) and purified by trituration from ether/heptane, filtered, washed (H_2O) and dried under vacuum to afford 0.33 g (58%) of **2h** as a white powder. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.47 (bs, 1 H), 10.42 (bs, 1 H), 8.92 (d, *J* = 8.5 Hz, 1 H), 7.91-7.86 (m, 2 H), 7.84-7.80 (m, 2 H), 7.61-7.59 (m, 2 H), 7.54-7.22 (m, 9 H), 5.89 (d, *J* = 8.4 Hz, 1 H). MS (M-H): 372. HRMS Calcd. for $C_{22}H_{20}N_3O_3$: 374.1499. Found: 374.1492. Anal. Calcd. for $C_{22}H_{19}N_3O_3$ (0.16 eq. H_2O): C, 70.22; H, 5.18; N, 11.17. Found: C, 69.97; H, 4.90; N, 11.10.

2,5,7-Triphenyl-imidazo[5,1-b][1,3,4]oxadiazole 1h. Prepared from **2h** (0.16 g, 0.42 mmol) and purified via flash column chromatography (20 → 40% EtOAc/CH_2Cl_2) to afford 87 mg (62%) of **1h** as a yellow solid, mp = 268 – 270 ºC: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.35-8.33 (m, 2 H), 8.24-8.21 (m, 2 H), 7.98 (dd, *J*= 1.1, 8.3 Hz, 2 H), 7.68-7.59 (m, 3 H), 7.51-7.46 (m, 3 H), 7.43-7.37 (m, 1 H), 7.28-7.22 (m, 2 H). ^{13}C NMR (125 MHz) δ 133.1, 129.5, 129.2, 129.0, 128.9, 127.5, 125.4, 124.7. MS (M+H): 338. HPLC: 11.11 min. Anal. Calcd for $C_{22}H_{15}N_3O_1$: C, 78.32; H, 4.48; N, 12.46. Found: C, 78.53; H, 4.36; N, 12.43.

5-Ethyl-7-methyl-2-phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1j (prepared according to the procedure for **1a**)

[2-(N'-Benzoyl-hydrazino)-1-methyl-2-oxo-ethyl]-carbamic acid benzyl ester. Prepared from benzoic hydrazide HCl (0.52 g, 3.8 mmol), CBz-Ala-OH (0.94 g, 4.2 mmol) to afford 0.75 g (73%) of the carbamate as a white solid. ^{1}H NMR (500 MHz, CD_3OD) δ 7.88-7.86 (m, 2 H), 7.60-7.57 (m, 1 H), 7.48-7.45 (m, 2 H), 7.37-7.26 (m, 5), 5.11 (s, 2 H), 4.32 (q, *J* = 7.2 Hz, 1 H), 1.45 (d, *J* = 7.2 Hz, 3 H). MS (M-H): 340.

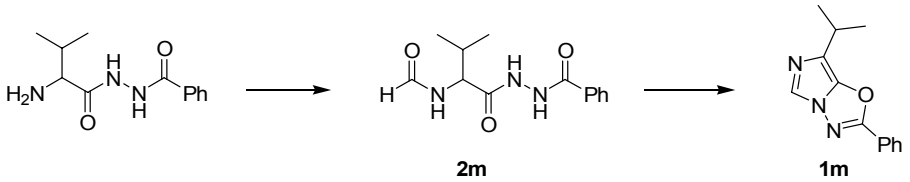
N-[2-(N'-Benzoyl-hydrazino)-1-methyl-2-oxo-ethyl]-propionamide 2j. Prepared from [2-(N'-benzoyl-hydrazino)-1-methyl-2-oxo-ethyl]-carbamic acid benzyl ester (0.37 g, 1.1 mmol) to afford 0.17 g (60%) of **2j** as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J*= 7.2 Hz, 2 H), 7.57-7.54 (m, 1 H), 7.47-7.44 (m, 2 H), 5.99-5.93 (m, 1 H), 4.68-4.65 (m, 1 H), 2.30 (q, *J* = 7.6 Hz, 2 H), 1.47 (d, *J* = 7.1 Hz, 3 H), 1.19 (t, *J* = 7.6 Hz, 3 H). MS: (M+H): 264 and (M-H): 262. HRMS [M + Na] Calcd. for $C_{13}H_{17}N_3O_3Na$: 286.1162. Found: 286.1165.

5-Ethyl-7-methyl-2-phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1j. Prepared from **2j** (93 mg, 0.35 mmol) and purified via flash column chromatography (0 → 7% MeOH/CH_2Cl_2) to afford 61 mg (76%) of **1j** as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06-8.04 (m, 2 H), 7.60-7.57 (m, 1 H), 7.54-7.51 (m, 2 H), 2.91 (q, *J* = 7.7 Hz, 2 H), 2.34 (s, 3 H), 1.40 (t, *J* = 7.6 Hz, 3 H). ^{13}C NMR (125 MHz) δ 132.6, 129.3, 127.1, 124.7, 20.5, 12.2, 11.9. IR (thin film): 3210, 2976, 1659, 1558, 1450, 1419, 1281, 1164, 772, 688 cm^{-1}. MS (M+H): 228. GC-MS: 4.45 min with m/z: 227. Anal. Calcd. for $C_{13}H_{13}N_3O_1$ (0.28 eq CH_2Cl_2): C, 63.54; H, 5.44; N, 16.74. Found: C, 63.41; H, 5.27; N, 16.96.

7-Benzyl-5-ethyl-2-phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1k (prepared according to the procedure for **1b**)

N-[2-(N'-Benzoyl-hydrazino)-1-benzyl-2-oxo-ethyl]-propionamide 2k. Prepared from [2-(N'-benzoyl-hydrazino)-1-benzyl-2-oxo-ethyl]-carbamic acid tert-butyl ester (0.40g, 1.1 mmol, see prep for **1c** above) to afford 0.32 g (65%) of **2k** as a white solid, mp = 196 – 198 ºC: ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.36 (bs, 1 H), 10.13 (bs, 1 H), 8.05 (d, = 8.8 Hz, 1 H), 7.85-7.83 (m, 2 H), 7.55-7.52 (m, 1 H), 7.47-7.43 (m, 2 H), 7.27-7.20 (m, 3 H), 7.17-7.14 (m, 1 H), 4.64-4.58 (m, 1 H), 3.08-3.03 (dd, *J* = 3.8, 13.8 Hz, 1 H), 2.78-2.72 (m, 1 H), 2.02-1.93 (m, 2 H), 0.82 (t, *J* = 7.6 Hz, 3 H). IR (thin film): 3179, 3063, 1661, 1606, 1451, 1275, 1173, 693 cm^{-1}. MS (M+H): 340. HPLC: 6.56 min. Anal. Calcd. for $C_{19}H_{21}N_3O_3$: C, 67.24; H, 6.24; N, 12.38. Found: C, 67.01; H, 6.25; N, 12.33.

7-Benzyl-5-ethyl-2-phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1k. Prepared from **2k** (0.10g, 0.30 mmol) and purified via flash column chromatography (50% EtOAc/Hept) to afford 61 mg (67%) of **1k** as a clear, faintly yellow gum. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 1.2, 8.1 Hz, 2 H), 7.58-7.55 (m, 1 H), 7.54-7.48 (m, 2 H), 7.38-7.36 (m, 2 H), 7.34-7.31 (m, 2 H), 7.25-7.22 (m, 1 H), 4.05 (s, 2 H), 2.92 (q, *J* = 7.6 Hz, 2 H), 1.41 (t, *J* = 7.7 Hz, 3 H). ^{13}C NMR (125 MHz) δ 132.6, 131.0, 129.2, 128.9, 128.6, 127.1, 126.5, 124.5, 33.5, 21.6, 12.2. IR (thin film): 3238, 1663, 1604, 1451, 1275, 1072, 690 cm^{-1}. MS (M+H): 304. HPLC: 7.00 min. Anal. Calcd. for $C_{19}H_{17}N_3O_1$ (0.32 eq CH_2Cl_2): C, 70.20; H, 5.38; N, 12.71. Found: C, 70.06; H, 5.28; N, 12.85.

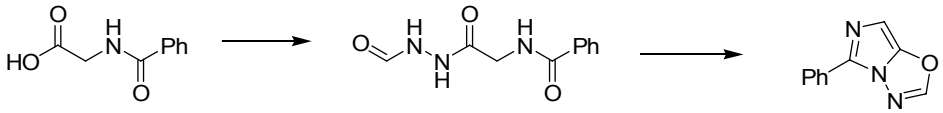


2m **1m**

7-Isopropyl-2-phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1m

N-[1-(N'-Benzoyl-hydrazinocarbonyl)-2-methyl-propyl]-formamide 2m. Benzoic acid N'-(2-amino-3-methyl-butyryl)-hydrazide was prepared from [1-(N'-benzoyl-hydrazinocarbonyl)-2-methyl-propyl]-carbamic acid tert-butyl ester according to the procedure for **1b**. To a solution of the amine (0.37 g, 1.3 mmol), formic acid (0.31 g, 6.7 mmol) and TEA (0.28 mL, 2.0 mmol) in THF/DMF (2.5:1, 14 mL) was added EDCI (0.33 g, 1.7 mmol). After stirring 16 h, the reaction mixture was diluted with CH_2Cl_2 and washed with sat'd $NaHCO_3$ and H_2O. The organic phase was concentrated and the residue triturated with ether/heptane, filtered, and dried under vacuum to afford 61 mg (17%) of **2m** as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.11 (s, 1 H), 7.86-7.83 (m, 2 H), 7.57-7.51 (m, 3 H), 6.18-6.14 (m, 1 H), 4.51-4.46 (m, 1 H), 2.24-2.18 (m, 1 H), 1.01 (apparent t, *J* = 7.80 Hz, 6 H). MS (M-H): 262.

7-Isopropyl-2-phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1m. The formamide **2m** (49 mg, 0.19 mmol) was taken up in acetonitrile (2 mL) and $POCl_3$ (1 mL) and heated to reflux. After 90 min, the mixture was cooled to ambient temp. and concentrated. The residue was taken up in CH_2Cl_2 and washed with sat'd $NaHCO_3$ and H_2O. The organic phase was concentrated and purified via flash column chromatography (25 → 50% EtOAc/heptane) to afford 17 mg (40%) of **1m** as white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06-8.04 (m, 2 H), 7.75 (s, 1 H), 7.61-7.57 (m, 1 H), 7.54-7.49 (m, 2 H), 3.09 (app quint., J = 6.9 Hz, 1 H), 1.39 (d, J = 6.9 Hz, 6 H). ^{13}C NMR (125 MHz) δ 133.1, 129.4, 127.3, 116.2, 26.6, 22.1. MS (M+H): 228.

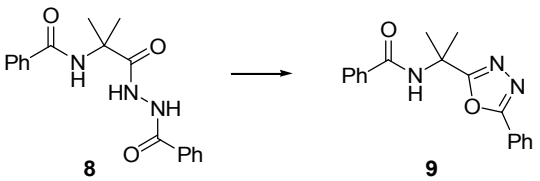


2n **1n**

5-Phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1n

N-[2-(N'-Formyl-hydrazino)-2-oxo-ethyl]-benzamide 2n. To a solution of hippuric acid (0.50 g, 2.8 mmol) and formic hydrazide (0.22 g, 3.6 mmol) in CH_2Cl_2 (10 mL) was added EDCI (0.70 g, 3.6 mmol). After stirring 16 h, sat'd $NaHCO_3$ was added and stirred vigorously. The precipitate was filtered, washed with ether and H_2O and dried under vacuum to afford 0.36 g (58%) of **2n** as a faint pink solid. ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.97 (bs, 1 H), 8.78-8.72 (m, 1 H), 7.84-7.81 (m, 2 H), 7.51-7.47 (m, 1 H), 7.44-7.40 (m, 2 H), 3.90-3.88 (m, 2 H). MS (M+H): 222.

5-Phenyl-imidazo[5,1-b][1,3,4]oxadiazole 1n. A solution of the hydrazide **2n** (0.20 g, 0.92 mmol) in acetonitrile (2 mL) and $POCl_3$ (1 mL) was heated to 100 °C. After 4 h, the reaction was concentrated. Purification via flash column chromatography (30 → 50% EtOAc/heptane) afforded 39 mg (23%) of **1n** as a light brown solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19-8.17 (m, 2 H), 8.04 (s, 1 H), 7.49-7.46 (m, 2 H), 7.39-7.37 (m, 1 H), 6.75 (s, 1 H). ^{13}C NMR (125 MHz) δ 154.1, 129.0, 128.8, 125.2, 96.4. MS (M+H): 186.

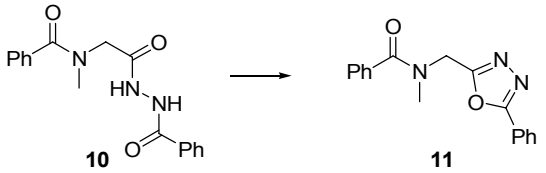


8 **9**

N-[2-(N'-Benzoyl-hydrazino)-1,1-dimethyl-2-oxo-ethyl]-benzamide 8. Prepared from Boc-Aib-OH according to the procedure for **2b**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.49 (d, J = 4.39 Hz, 1 H), 9.16 (d, J = 4.39 Hz, 1 H), 7.81-7.75 (m, 4 H), 7.50-7.45 (m, 2 H), 7.39-

7.35 (m, 4 H), 6.85 (s, 1 H), 1.68 (s, 6 H). MS (M+H): 326. HRMS Calcd. for $C_{18}H_{20}N_3O_3$: 326.1499. Found: 326.1503.

N-[1-Methyl-1-(5-phenyl-[1,3,4]oxadiazol-2-yl)-ethyl]-benzamide 9. A solution of hydrazide **8** (0.101 g, 0.31 mmol) in $POCl_3$ (1 mL) and MeCN (2 mL) was heated to 110 °C. After 16h, the reaction mixture was cooled to room temp and concentrated. The resulting residue was taken up in CH_2Cl_2 and washed with sat'd $NaHCO_3$ and H_2O. The organic phase was concentrated. Purification via flash column chromatography (EtOAc) afforded 62 mg (65%) of **9** as a gum. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10-8.08 (m, 4 H), 7.57-7.43 (m, 6 H), 6.79 (bs, 1 H), 2.28 (app t, *J* = 1.2 Hz, 6 H). MS (M+H): 308. HRMS Calcd. for $C_{18}H_{18}N_3O_2$: 308.1393. Found: 308.1392.

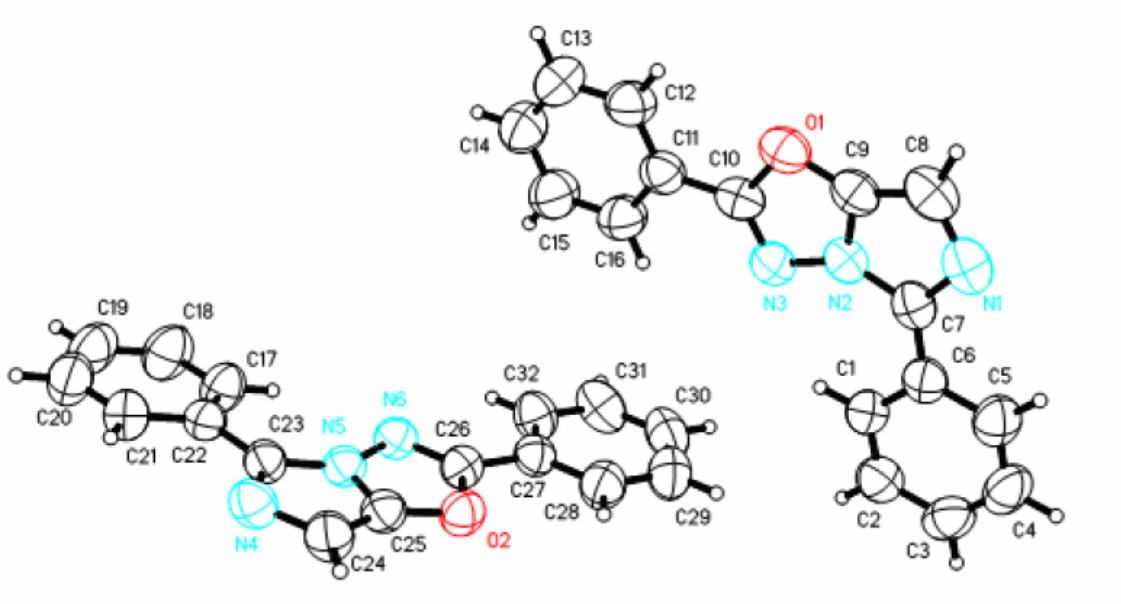


N-[2-(N'-Benzoyl-hydrazino)-2-oxo-ethyl]-N-methyl-benzamide 10. Prepared from Boc-Sar-OH according to the procedure for **2b**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89-7.83 (m, 4 H), 7.60-7.44 (m, 6 H), 4.29 (s, 2 H), 3.17 (s, 3 H). MS (M+H): 312. HRMS [M+Na] Calcd. for $C_{17}H_{17}N_3O_3Na$: 334.1162. Found: 334.1159.

N-Methyl-N-(5-phenyl-[1,3,4]oxadiazol-2-ylmethyl)-benzamide 11. A solution of the hydrazide (0.073 g, 0.23 mmol) in $POCl_3$ (1 mL) and acetonitrile (2 mL) was heated to reflux (110 °C). After heating 5 h, the reaction mixture was cooled to ambient temp.and concentrated. The residue was taken up in CH_2Cl_2 and washed with sat'd Na_2CO_3 and H_2O. The organic phase was concentrated. Purification via flash column chromatography (50 → 100% EtOAc/heptane) afforded 22 mg (32%) of **11** as an off-white residue. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.12-7.98 (m, 2 H), 7.57-7.38 (m, 8 H), 5.02 (s, 2 H), 3.11 (s, 3 H). MS (M+H): 294. HPLC: 7.489 min. IR: 1640, 1552, 1395, 1267, 1069, 717, 539 cm^{-1}. HRMS Calcd. for $C_{17}H_{16}N_3O_2$: 294.1237. Found: 294.1241.

2. X-ray Crystallography

ORTEP Structure of $C_{16}H_{11}N_3O$ **1a** with ellipsoids drawn at 50% probability: M = 261.28, orthorhombic, a = 5.6393(2), b = 11.9631(4), c = 38.0483(13) Å, U = 2566.87(15) Å^3, T = 298 K, space group $P2_12_12_1$, Z = 8, 8654 reflections measured, 3574 unique (R_{int} = 0.0263) which were used in all calculations. The final R1 = 0.0385 with wR2 = 0.0943.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C11
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C10
C9
C8
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C16
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N2
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C7
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C17
C6
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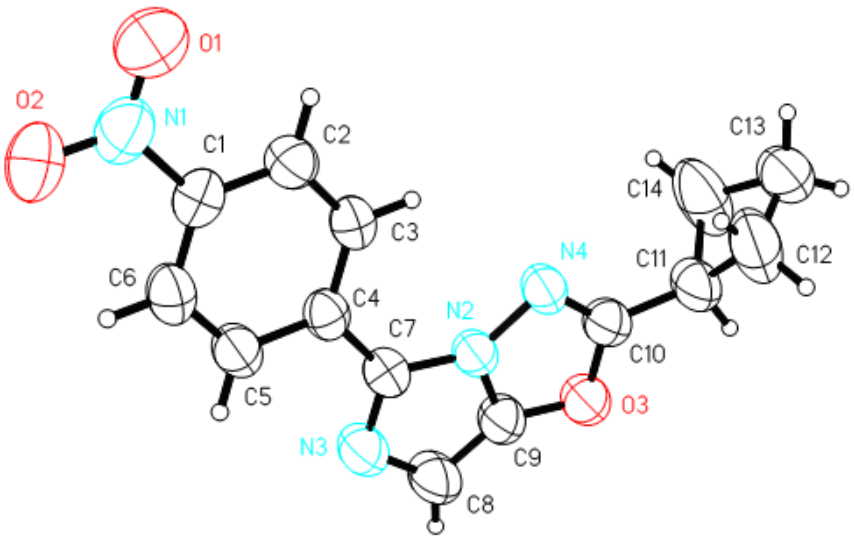
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C11
C12
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O3

3. Spectra

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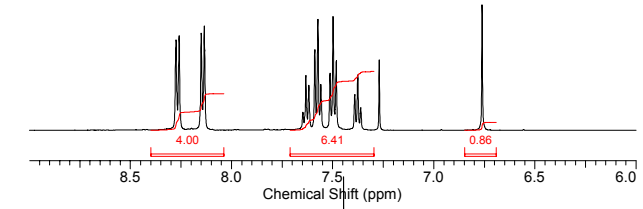
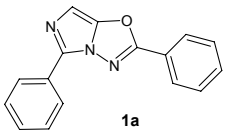
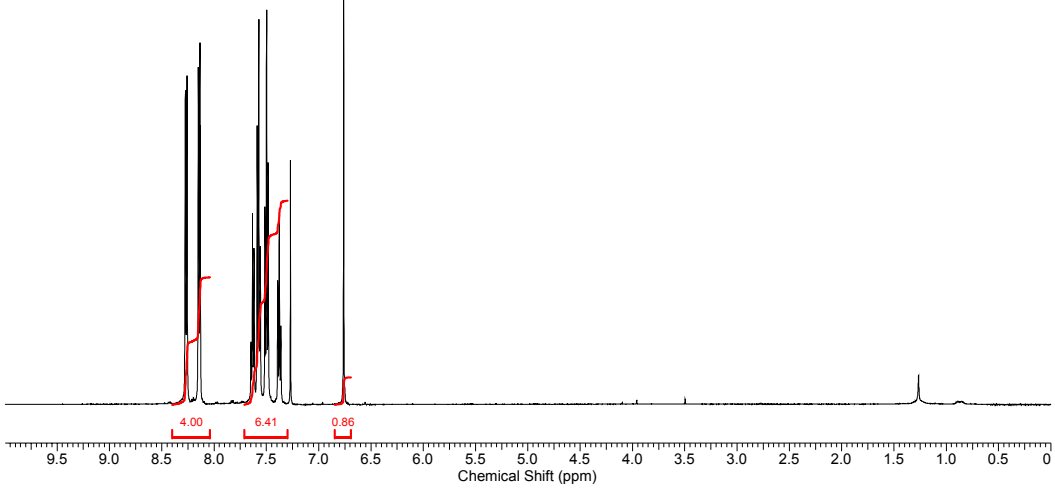




1a



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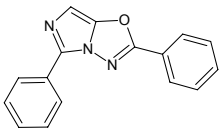

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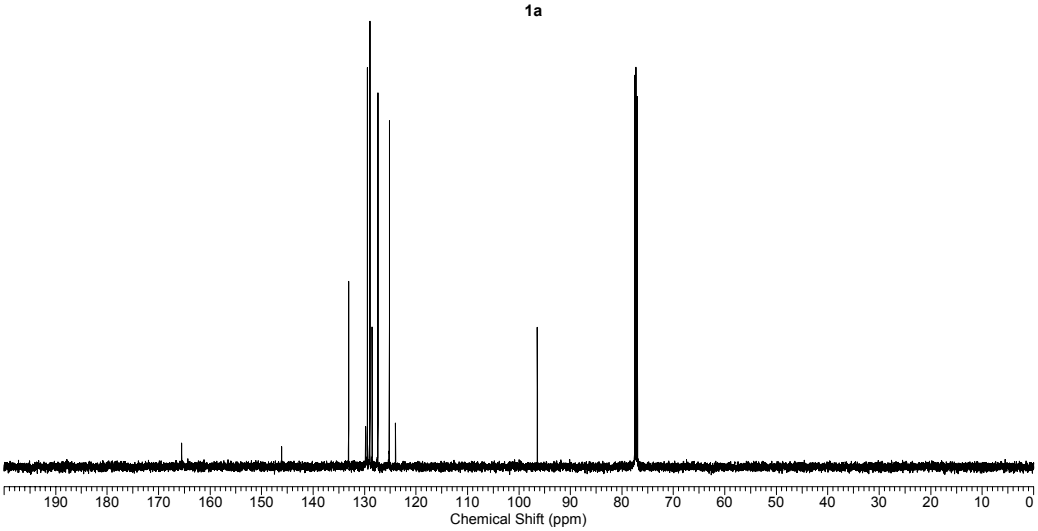

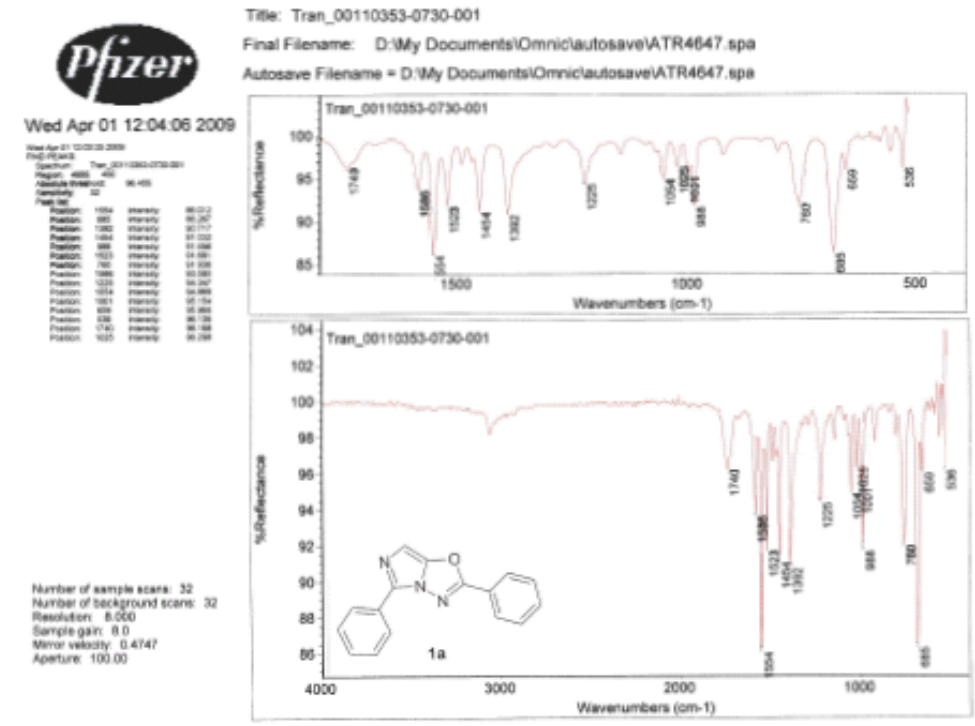

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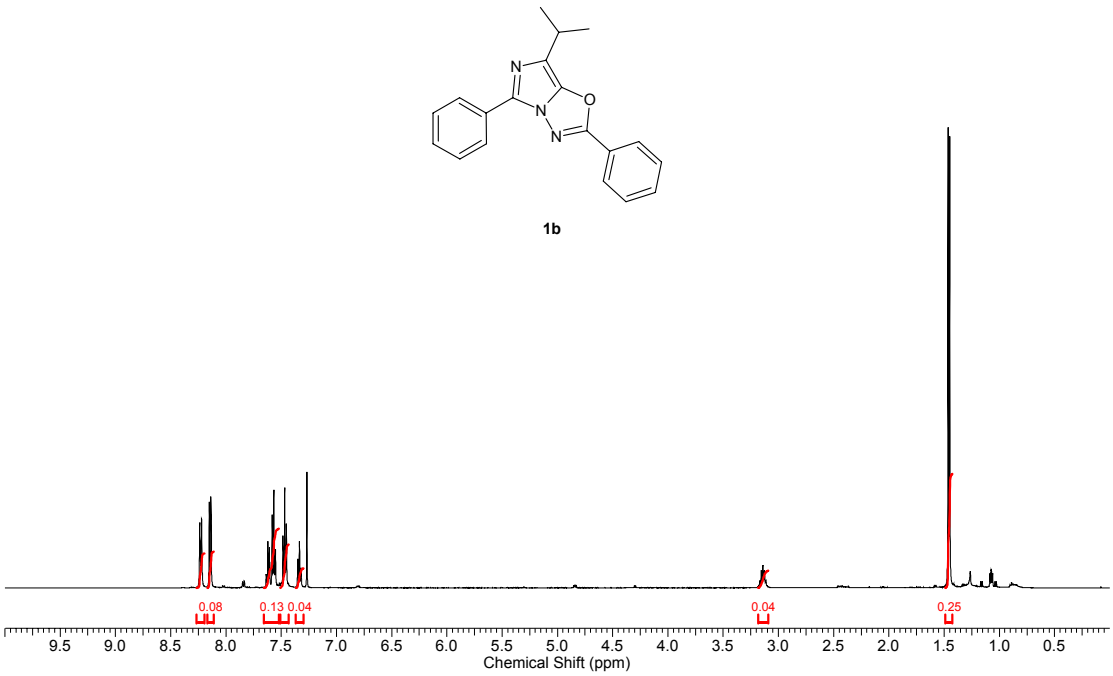

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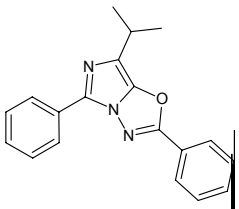
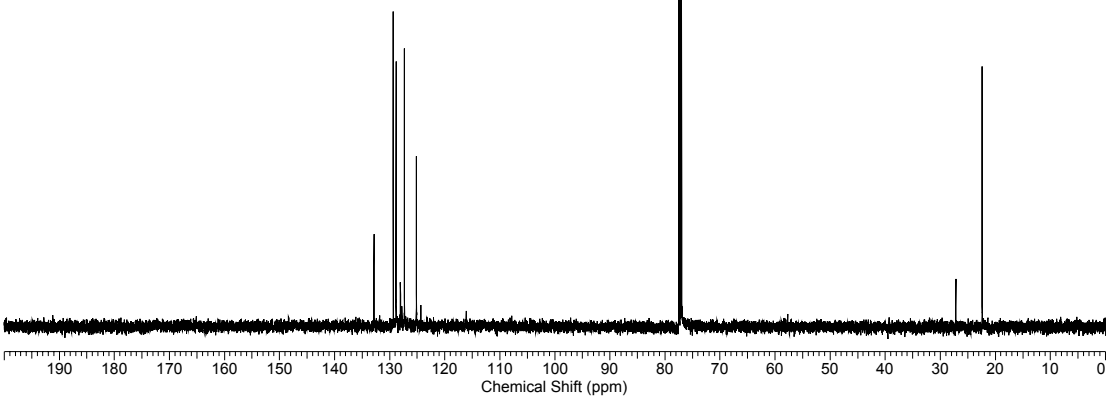


1b



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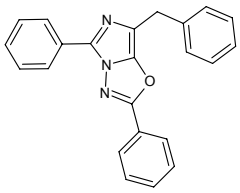
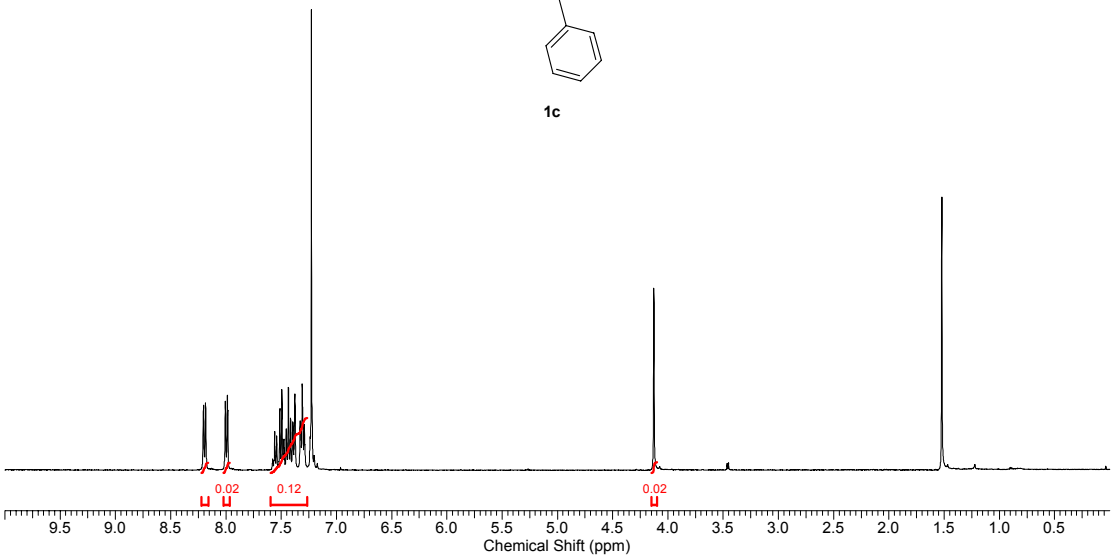
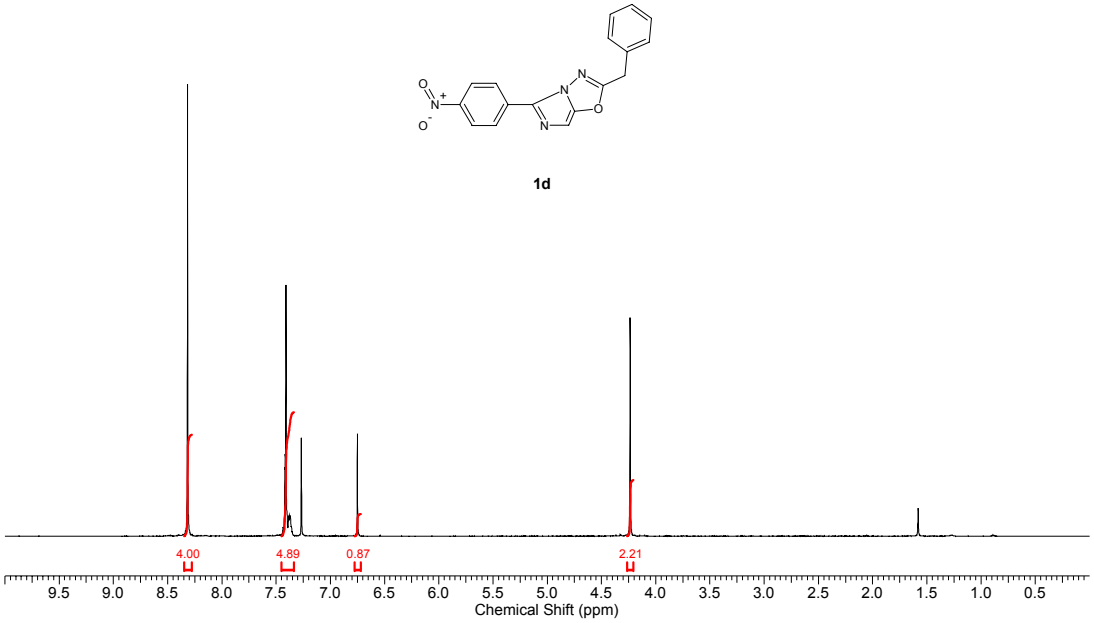
Frequency (MHz)	399.67	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	56.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2398.0217	***Sweep Width (Hz)***	6394.88	***Temperature (degree C)***	25.000		



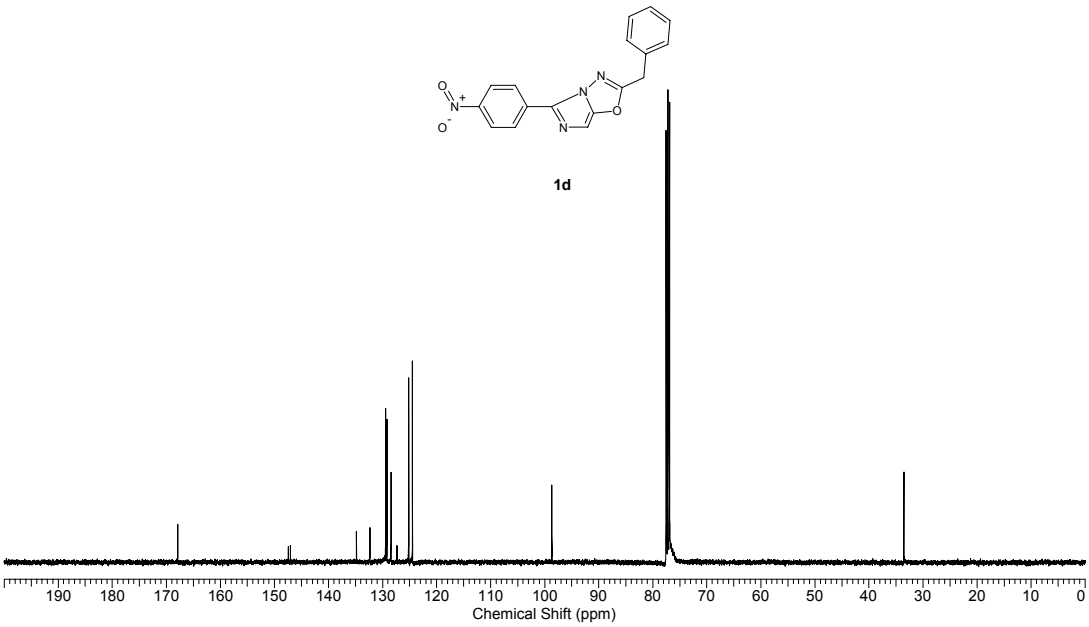
1c



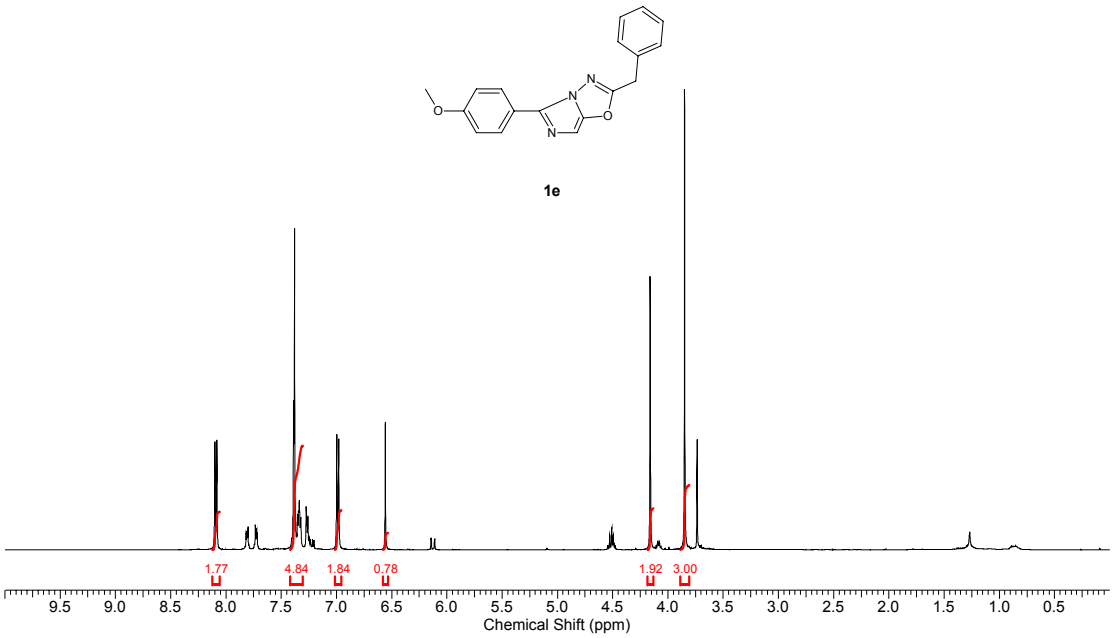
Acquisition Time (sec)	2.9452	***Date***	Feb 4 2009	***Date Stamp***	Feb 4 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34694.TMP\PRODUCTION\UNITYP\PATELNC\00110021-0994-011						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	48.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



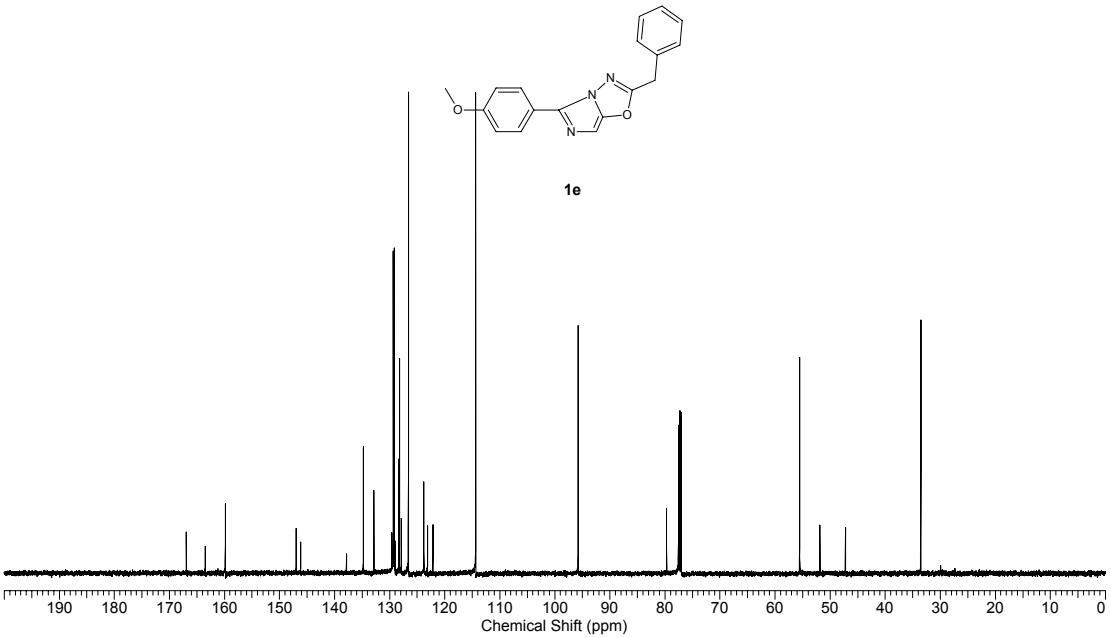
Acquisition Time (sec)	1.3582	***Date***	Feb 4 2009	***Date Stamp***	Feb 4 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34699.TMP\PRODUCTION\UNITYO\PATELNC\00110021-0994-011						
Frequency (MHz)	100.52	***Nucleus***	13C	***Number of Transients***	8192	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	10051.0752	***Sweep Width (Hz)***	24125.45	***Temperature (degree C)***	25.000		

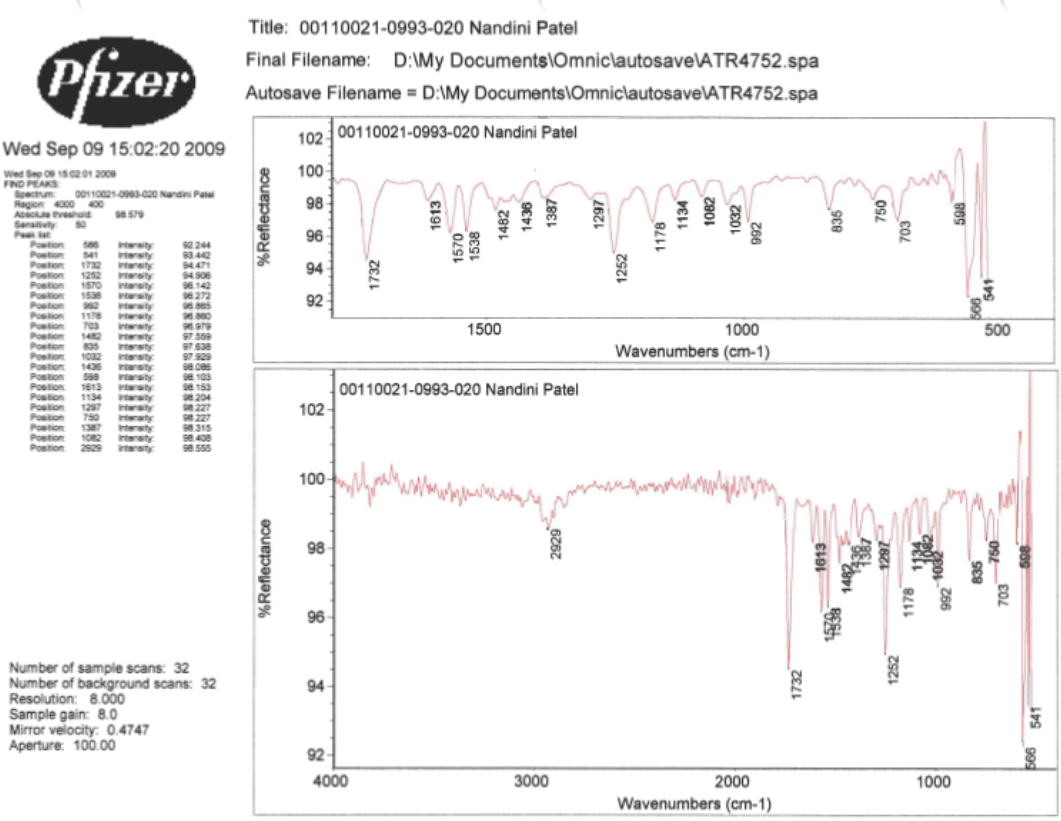


Acquisition Time (sec)	2.9452	***Date***	Feb 5 2009	***Date Stamp***	Feb 5 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34700.TMP\PRODUCTION\UNITYP\PATELNC\00110021-0993-020						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	30.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

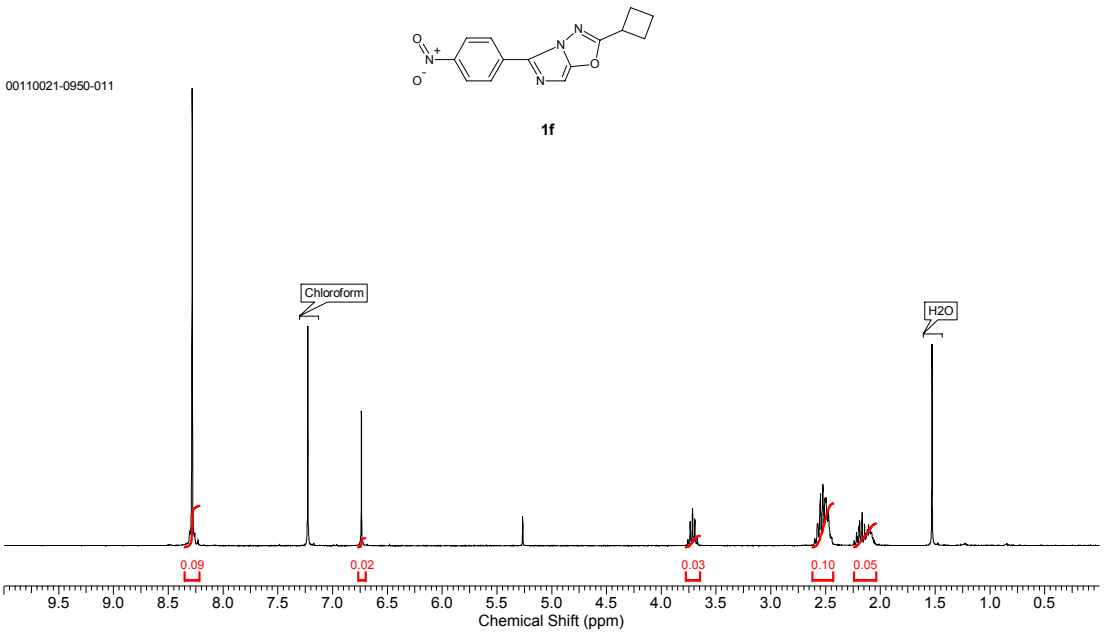


Acquisition Time (sec)	1.0863	***Date***	Feb 5 2009	***Date Stamp***	Feb 5 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34704.TMP\PRODUCTION\UNITYP\PATELNC\00110021-0993-020						
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	12567.9639	***Sweep Width (Hz)***	30165.91	***Temperature (degree C)***	25.000		



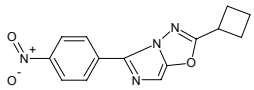


Acquisition Time (sec)	3.6829	***Date***	Oct 14 2008	***Date Stamp***	Oct 14 2008
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS59311.TMP\PRODUCTION\UNITYN\PATELNC\00110021-0950-011.2008288155111.FID\FID				
Frequency (MHz)	399.67	***Nucleus***	1H	***Number of Transients***	16
Original Points Count	23552	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	54.00	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2398.0217
Sweep Width (Hz)	6394.88	***Temperature (degree C)***	25.000		

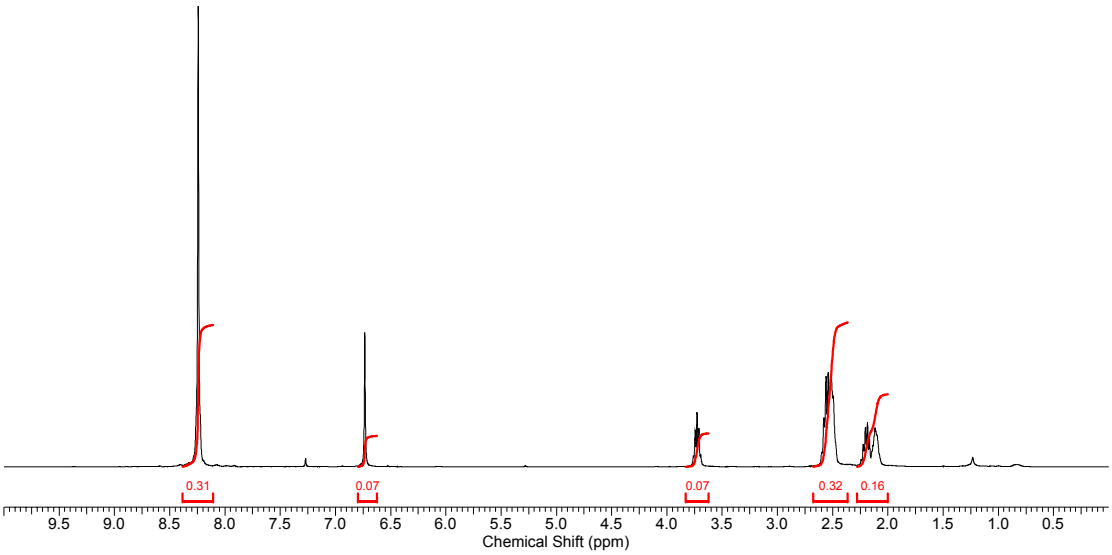


1f

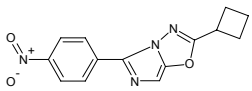
Acquisition Time (sec)	2.9452	***Date***	Feb 5 2009	***Date Stamp***	Feb 5 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS9625.TMP\PRODUCTION\UNITYP\PATELNC\00110021-0950-011						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	26.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	3000.5186	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



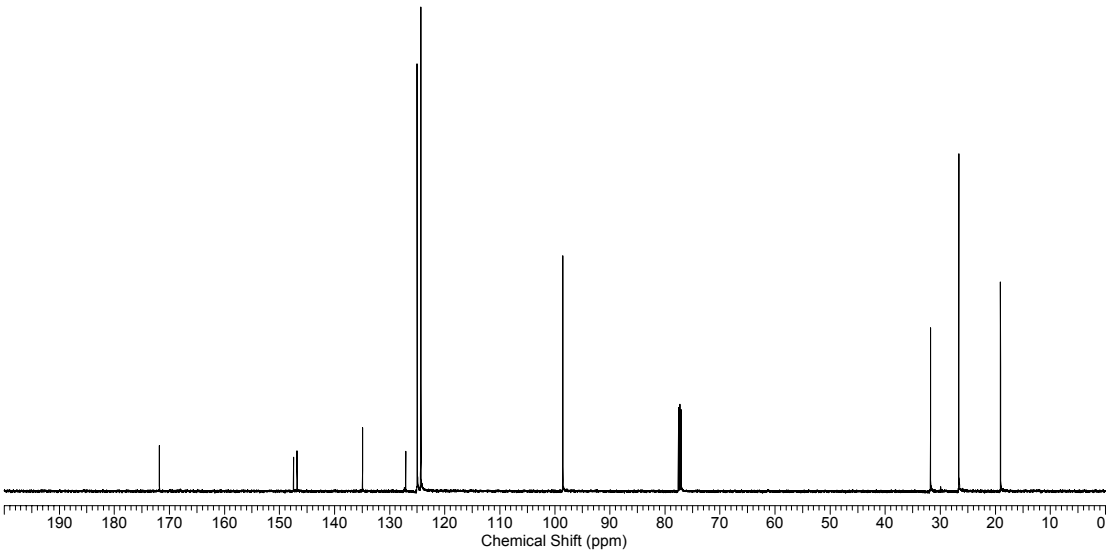
1f

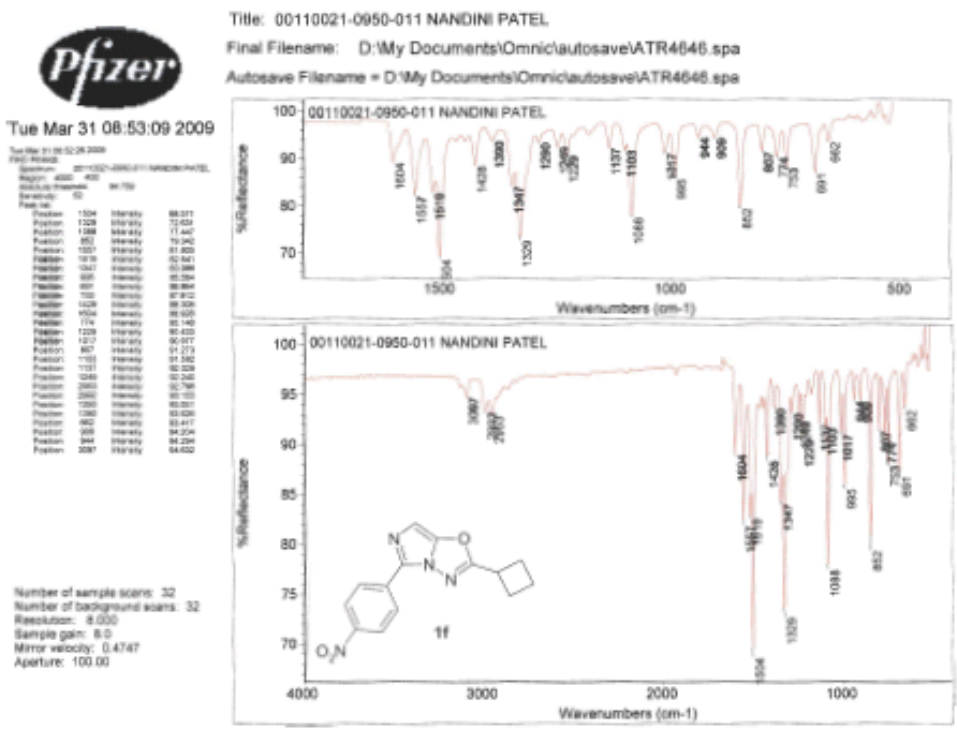


Acquisition Time (sec)	1.0863	***Date***	Feb 5 2009	***Date Stamp***	Feb 5 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS9618.TMP\PRODUCTION\UNITYP\PATELNC\00110021-0950-011						
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	12567.9639	***Sweep Width (Hz)***	30165.91	***Temperature (degree C)***	25.000		

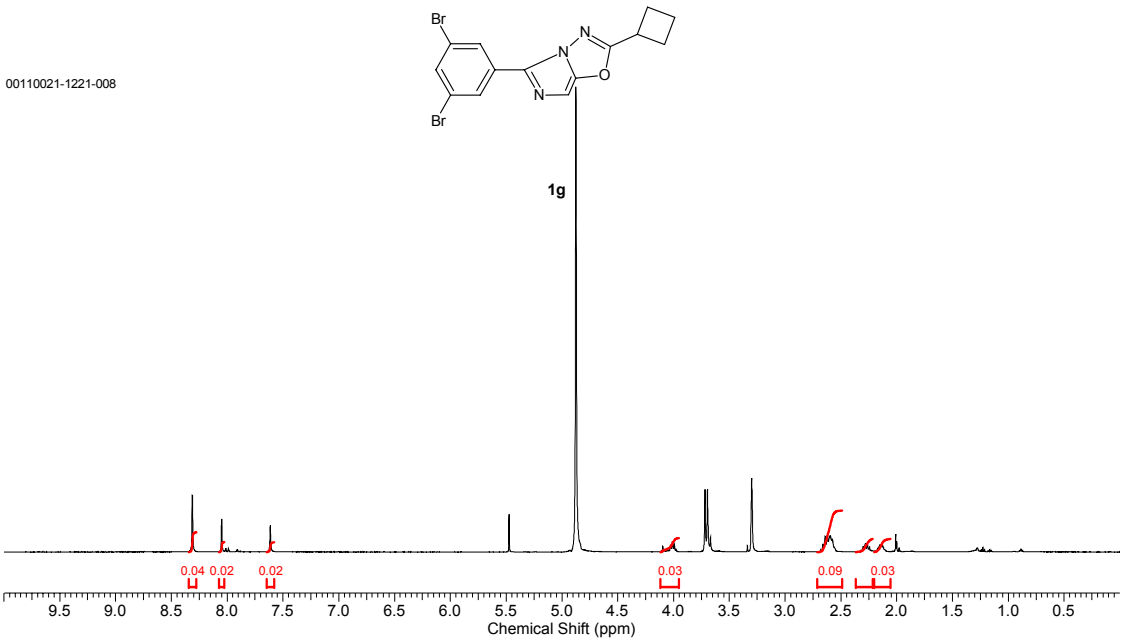


1f

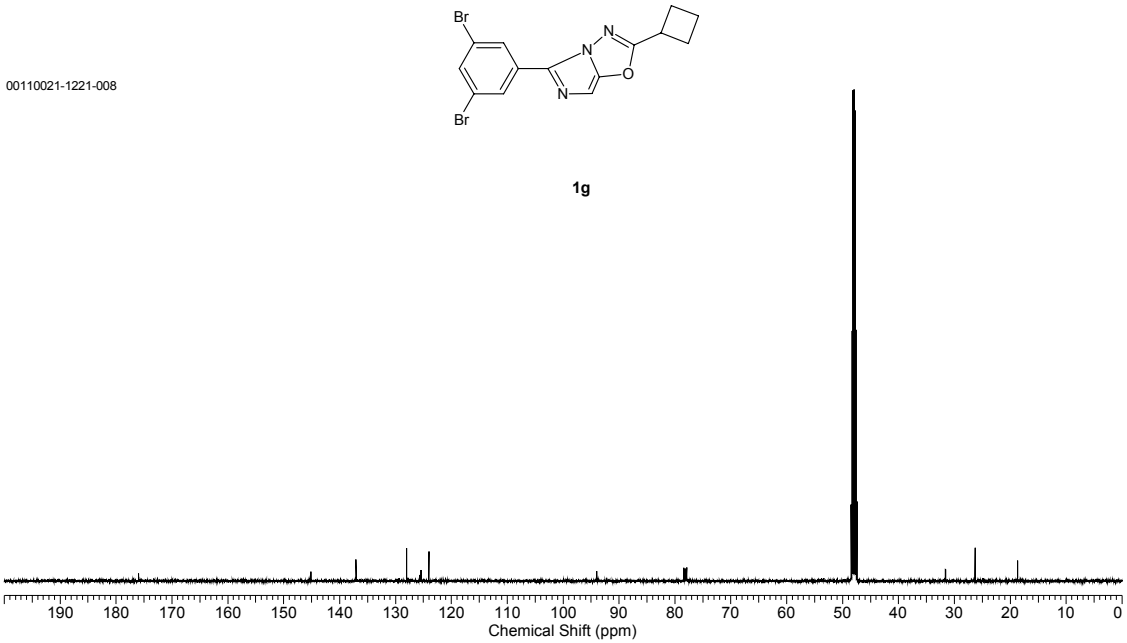




Acquisition Time (sec)	2.9452	***Date***	Sep 30 2009	***Date Stamp***	Sep 30 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS593.TMP\PRODUCTION\UNITYP\PATELNC\00110021-1221-008.2009273152117.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	42.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

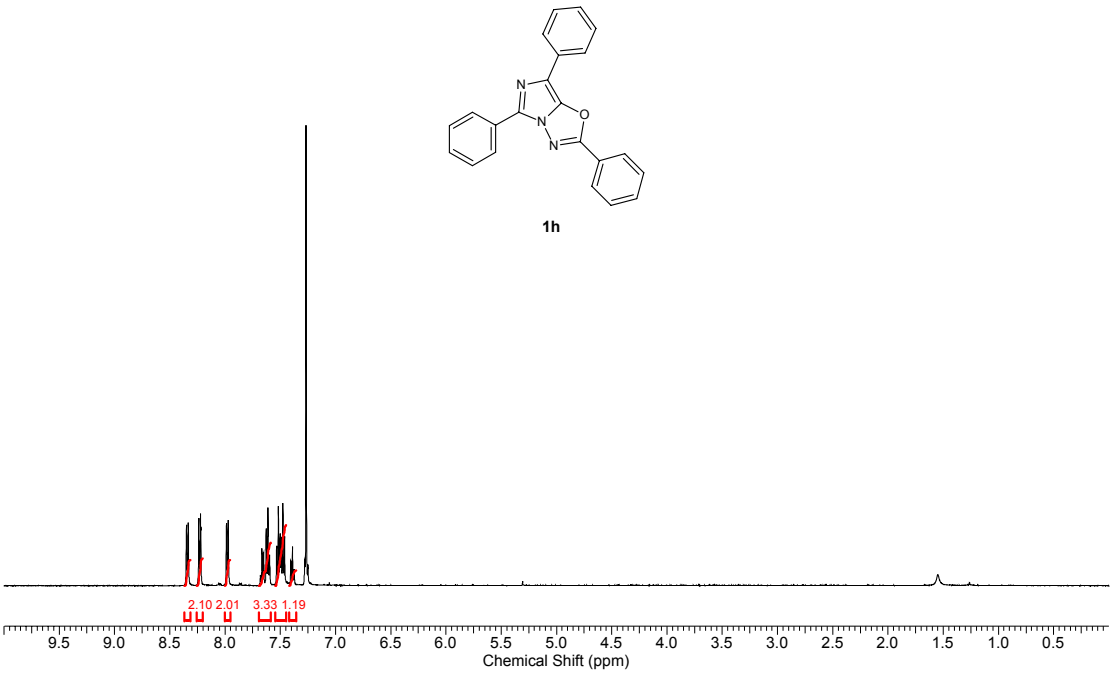


Acquisition Time (sec)	1.0863	***Date***	Sep 30 2009	***Date Stamp***	Sep 30 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS591.TMP\PRODUCTION\UNITYP\PATELNC\00110021-1221-008.2009273143155.FID\FID				
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512
Original Points Count	32768	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	60.00	***Solvent***	METHANOL-d4	***Spectrum Offset (Hz)***	12568.0879
Sweep Width (Hz)	30165.91	***Temperature (degree C)***	25.000		

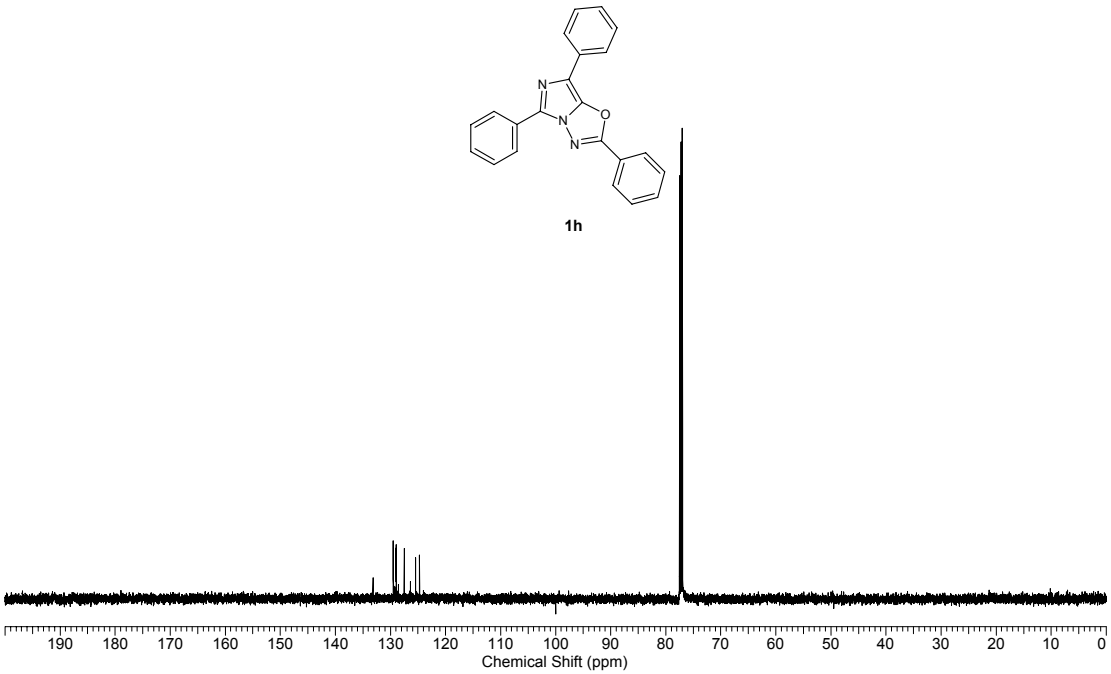


1g

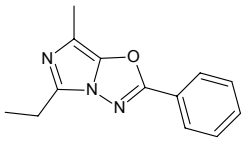
Acquisition Time (sec)	2.9452	***Date***	Dec 9 2008	***Date Stamp***	Dec 9 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34714.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0683-002						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	54.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



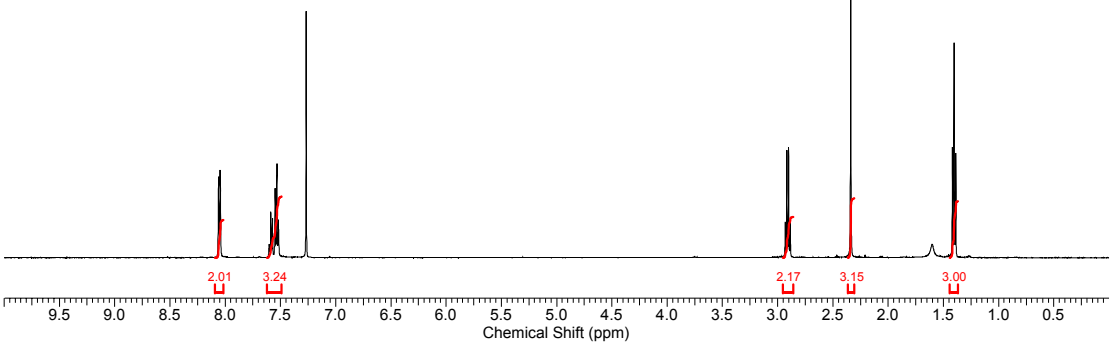
Acquisition Time (sec)	1.0863	***Date***	Dec 9 2008	***Date Stamp***	Dec 9 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34715.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0683-002						
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	12567.9639	***Sweep Width (Hz)***	30165.91	***Temperature (degree C)***	25.000		



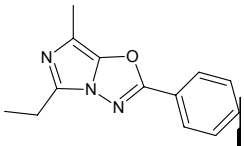
Acquisition Time (sec)	2.9452	***Date***	Oct 13 2008	***Date Stamp***	Oct 13 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34716.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0608-002						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	54.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



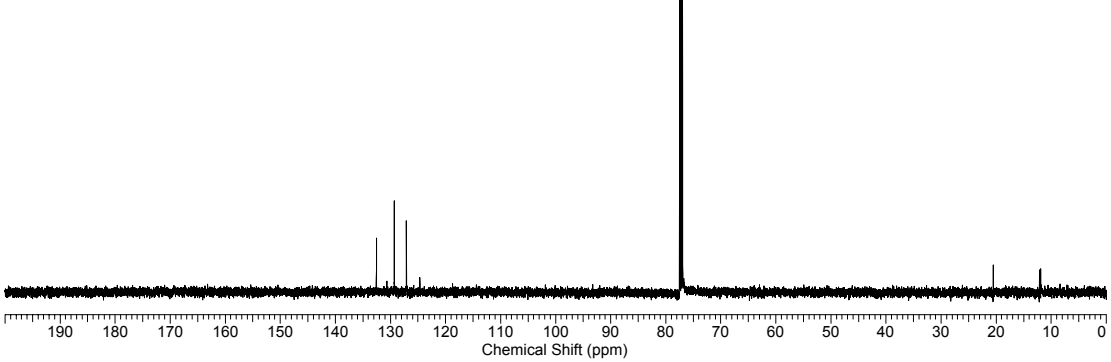
1j



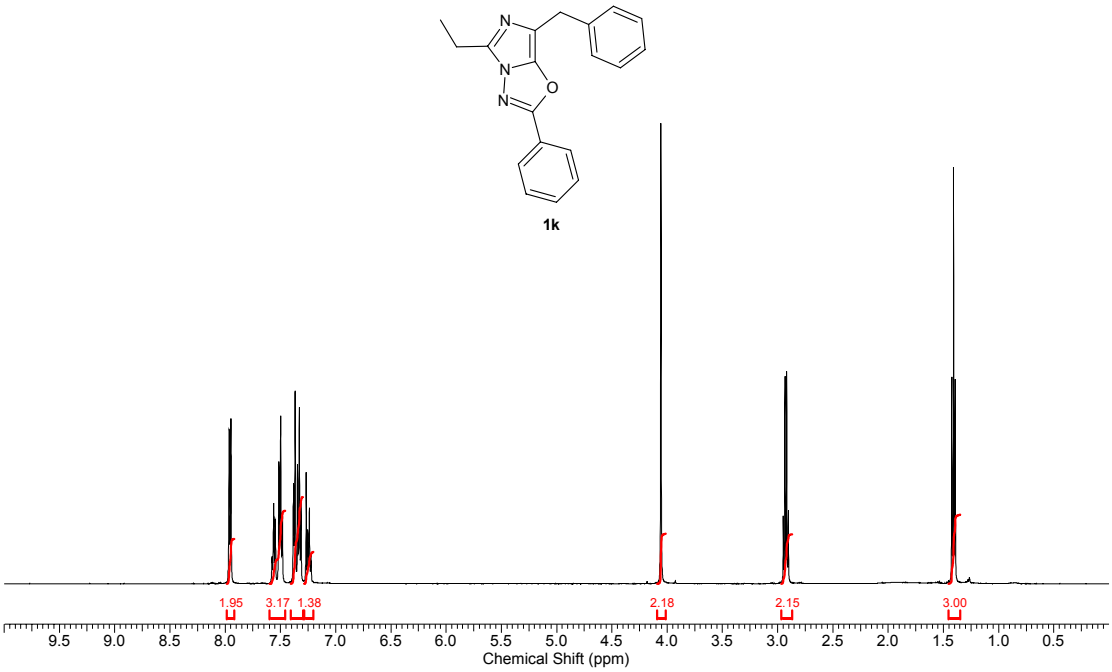
Acquisition Time (sec)	1.0863	***Date***	Oct 13 2008	***Date Stamp***	Oct 13 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34717.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0608-002						
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	12567.9639	***Sweep Width (Hz)***	30165.91	***Temperature (degree C)***	25.000		



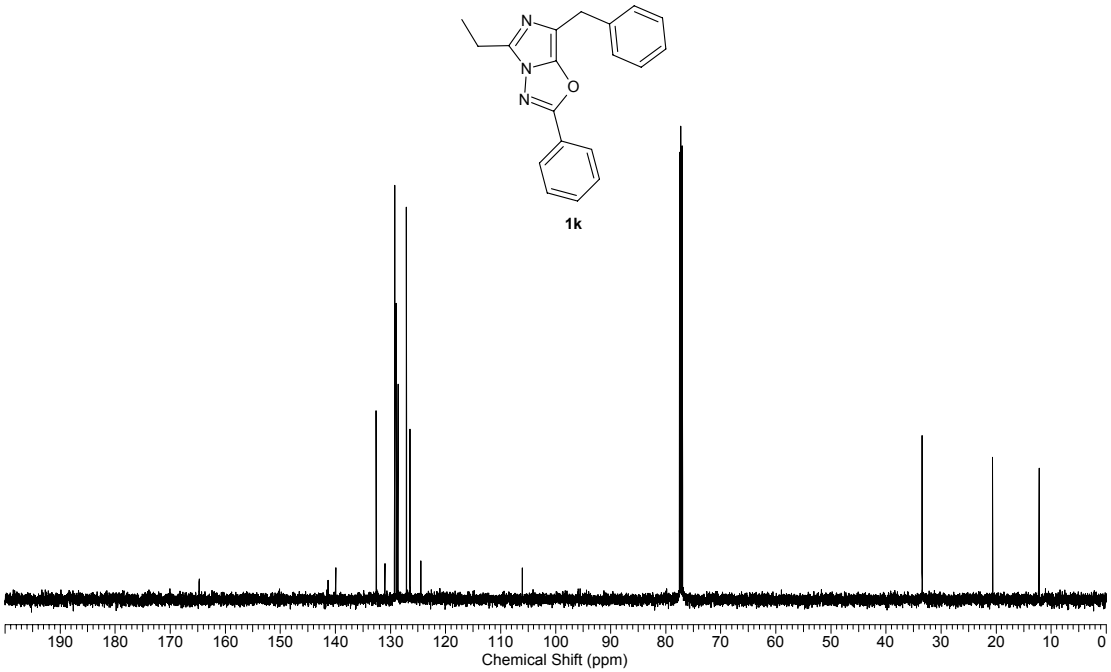
1j



Acquisition Time (sec)	2.9452	***Date***	Oct 27 2008	***Date Stamp***	Oct 27 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34718.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0630-002						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	42.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

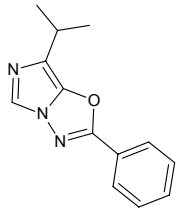


Acquisition Time (sec)	1.0863	***Date***	Oct 27 2008	***Date Stamp***	Oct 27 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS34719.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0630-002						
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	12567.9639	***Sweep Width (Hz)***	30165.91	***Temperature (degree C)***	25.000		

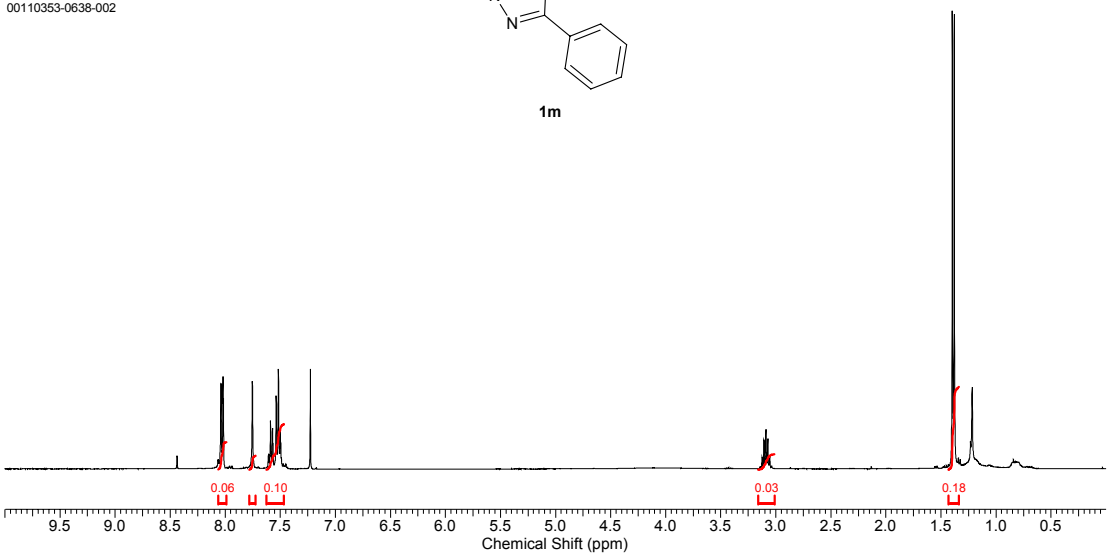


Acquisition Time (sec)	3.6829	***Date***	Nov 3 2008	***Date Stamp***	Nov 3 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS53730.TMP\PRODUCTION\UNITYN\TRAN02\00110353-0638-002.2008308111102.FID\FID						
Frequency (MHz)	399.67	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	50.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2398.0217	***Sweep Width (Hz)***	6394.88	***Temperature (degree C)***	25.000		

00110353-0638-002

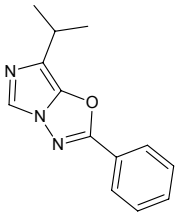


1m

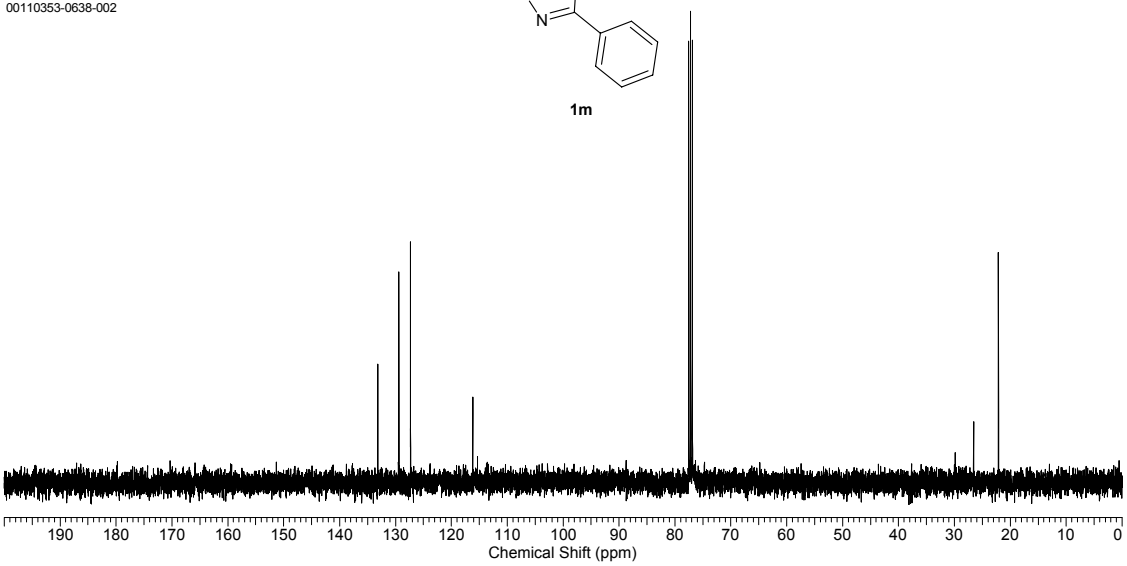


Acquisition Time (sec)	1.3586	***Date***	Nov 3 2008	***Date Stamp***	Nov 3 2008
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS53731.TMP\PRODUCTION\UNITYN\TRAN02\00110353-0638-002.2008308111756.FID\FID				
Frequency (MHz)	100.51	***Nucleus***	13C	***Number of Transients***	256
Original Points Count	32768	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	60.00	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	10049.6934
Sweep Width (Hz)	24118.18	***Temperature (degree C)***	25.000		

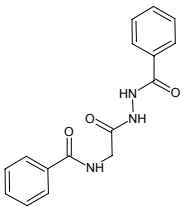
00110353-0638-002



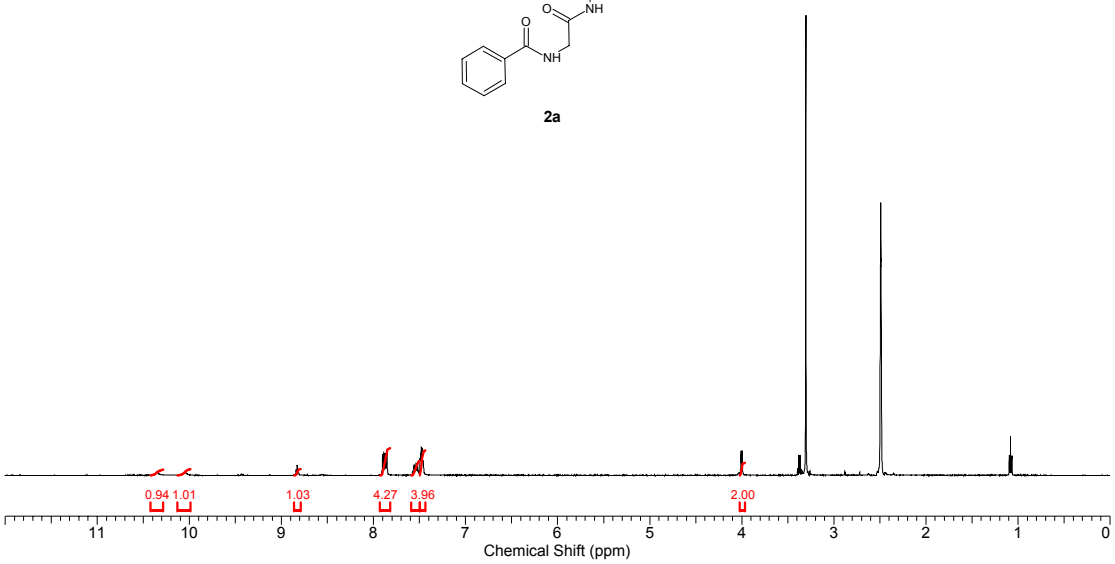
1m



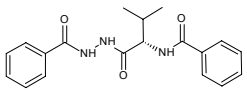
Acquisition Time (sec)	2.9452	***Date***	Oct 10 2008	***Date Stamp***	Oct 10 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS52608.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0604-001						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	50.00	***Solvent***	DMSO-d6
Spectrum Offset (Hz)	2998.9763	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



2a

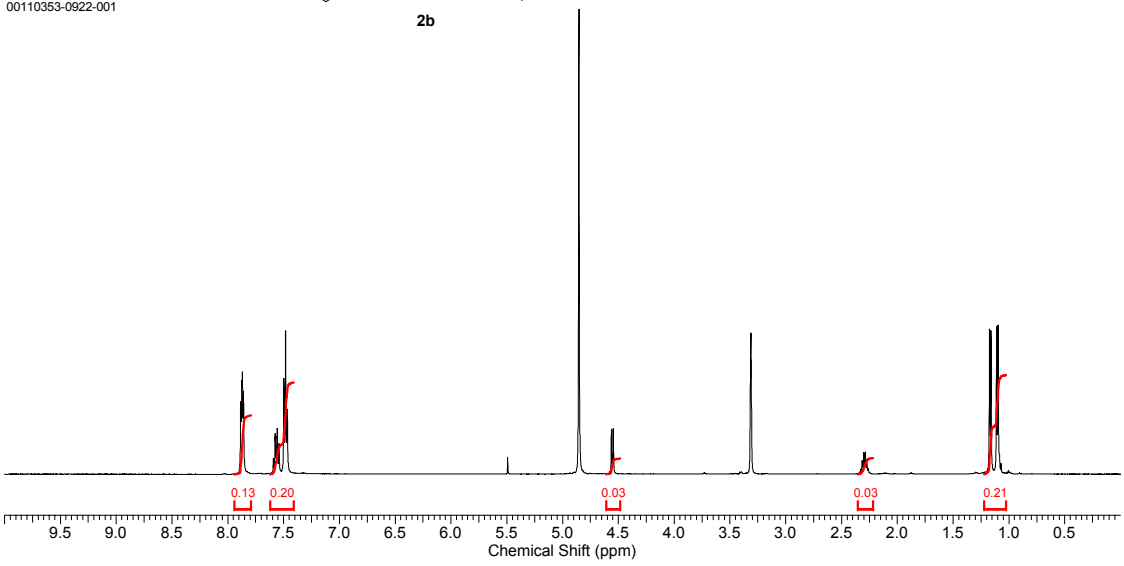


Acquisition Time (sec)	2.9452	***Date***	Jun 2 2009	***Date Stamp***	Jun 2 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43828.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0922-001.2009153171907.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	44.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

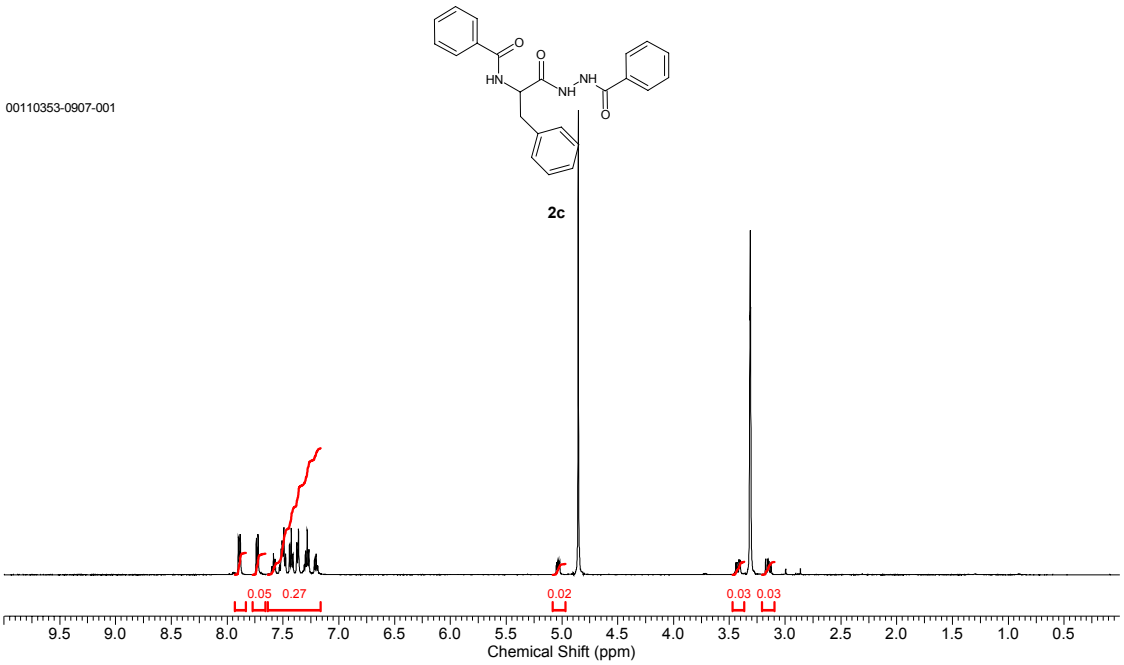


2b

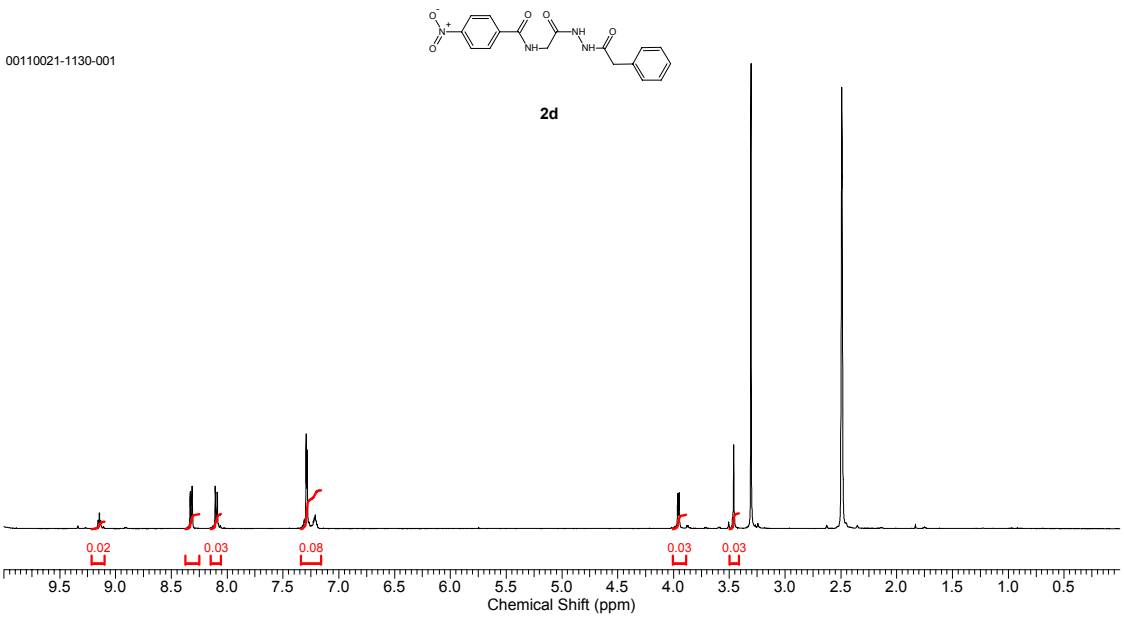
00110353-0922-001



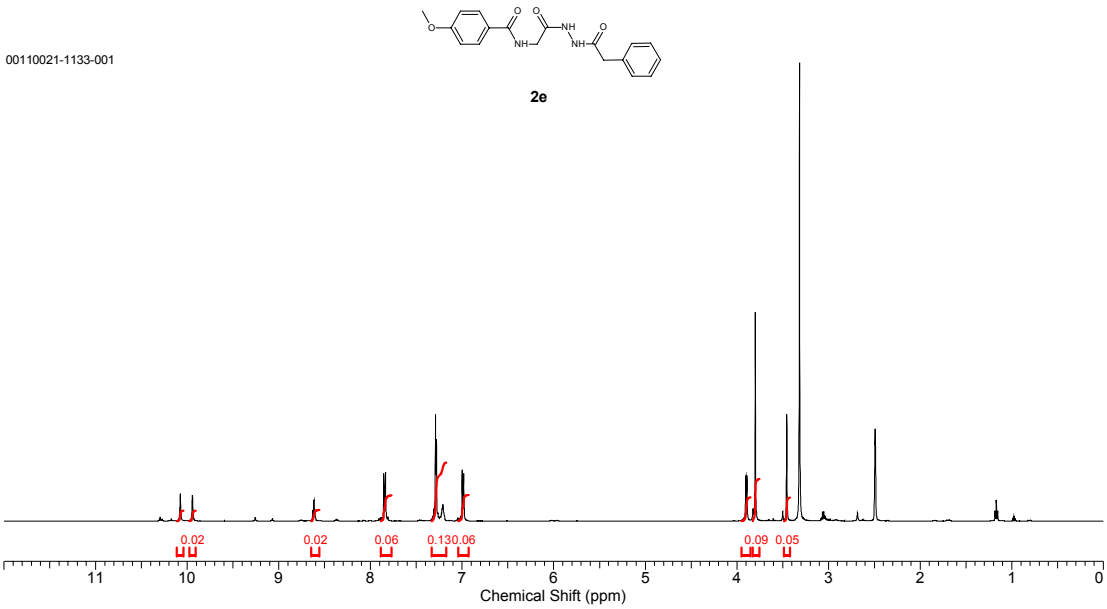
Acquisition Time (sec)	2.9452	***Date***	May 28 2009	***Date Stamp***	May 28 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43830.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0907-001.2009148082006.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	42.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



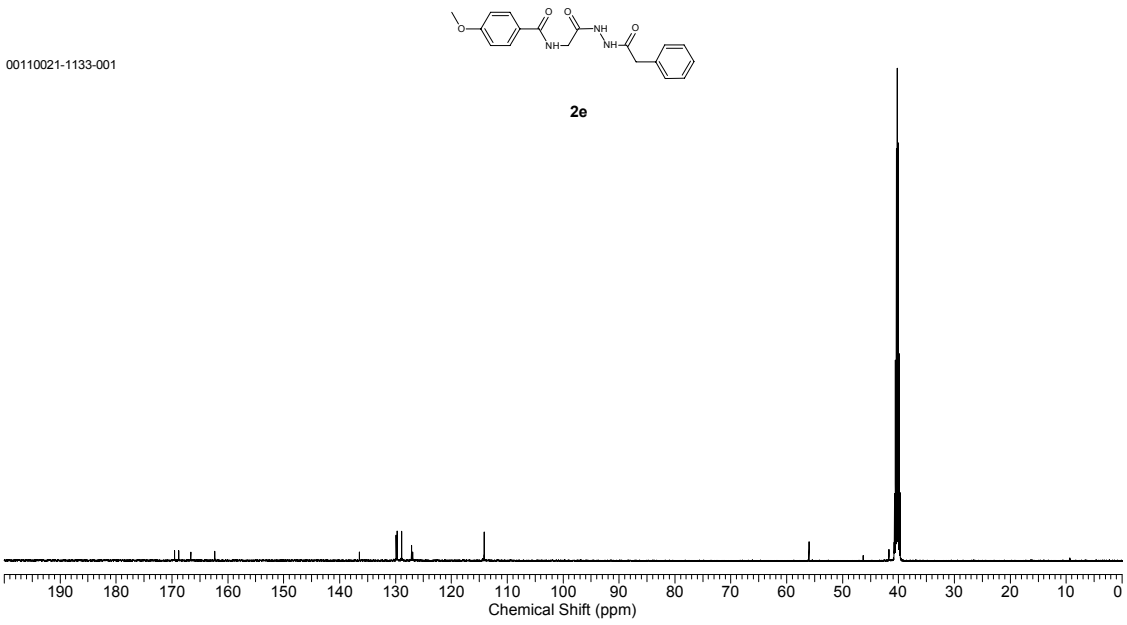
Acquisition Time (sec)	4.0976	***Date***	Jun 29 2009	***Date Stamp***	Jun 29 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43833.TMP\PRODUCTION\UNITYP\PATELNC\00110021-1130-001.2009180164700.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	64	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	50.00	***Solvent***	DMSO-d6
Spectrum Offset (Hz)	2998.9763	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



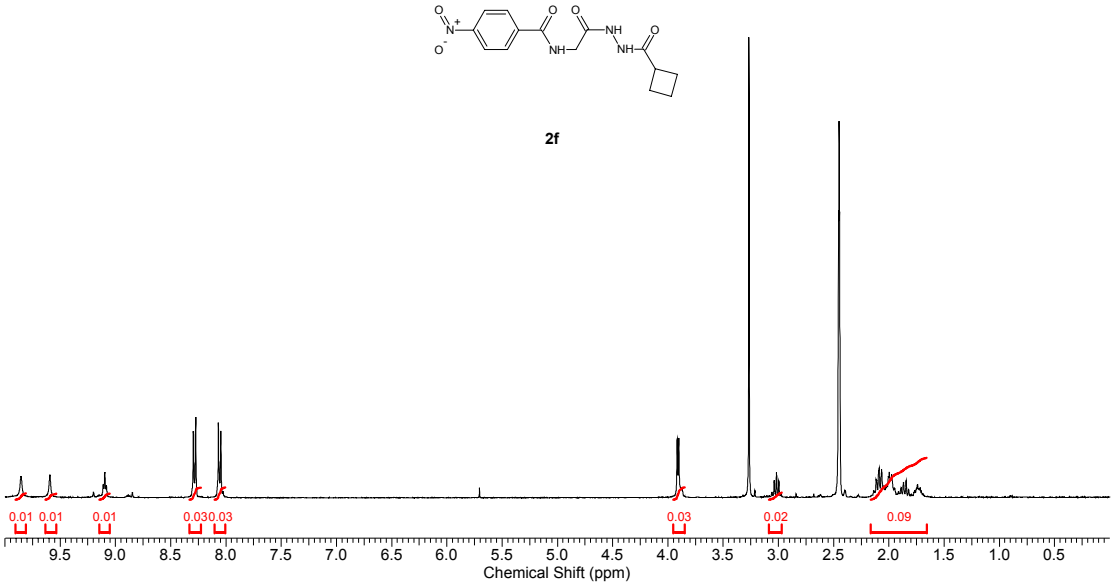
Acquisition Time (sec)	2.0488	***Comment***	200 mg white solids	***Date***	Jul 3 2009	***Date Stamp***	Jul 3 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43850.TMP\PRODUCTION\UNITYP\PATELNC\00110021-1133-001.2009184192617.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	256	***Original Points Count***	16384
Points Count	16384	***Pulse Sequence***	s2pul	***Receiver Gain***	42.00	***Solvent***	DMSO-d6
Spectrum Offset (Hz)	2998.9763	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



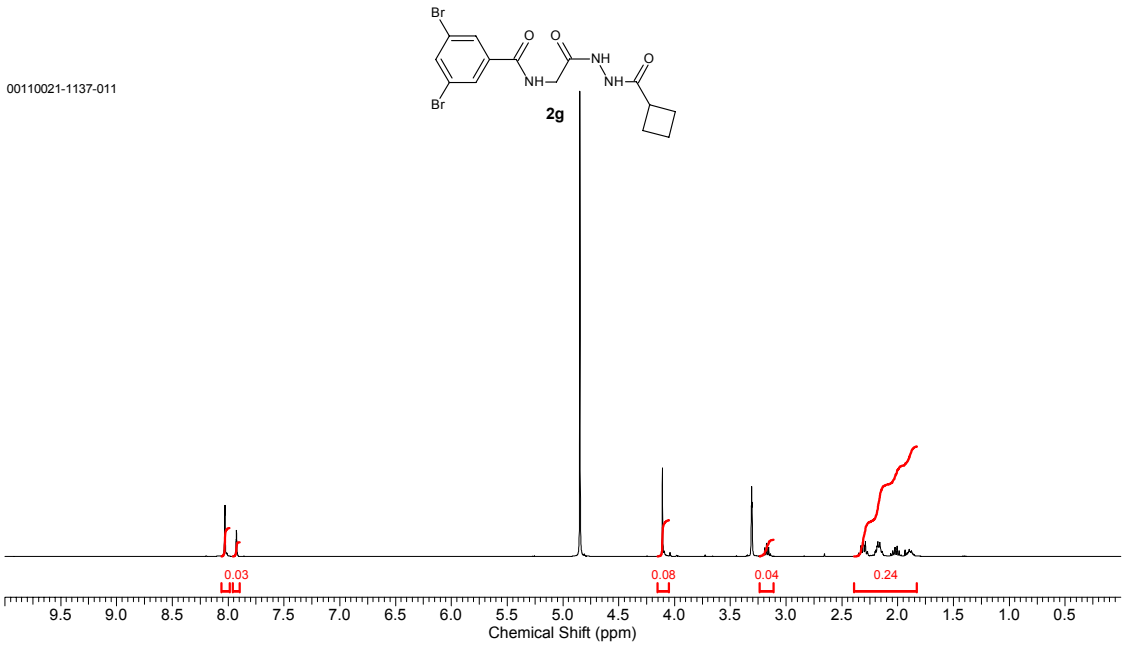
Acquisition Time (sec)	1.0863	***Comment***	200 mg white solids	***Date***	Jul 3 2009	***Date Stamp***	Jul 3 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43851.TMP\PRODUCTION\UNITYP\PATELNC\00110021-1133-001.2009184235113.FID\FID						
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	8192	***Original Points Count***	32768
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	60.00	***Solvent***	DMSO-d6
Spectrum Offset (Hz)	12568.0879	***Sweep Width (Hz)***	30165.91	***Temperature (degree C)***	25.000		



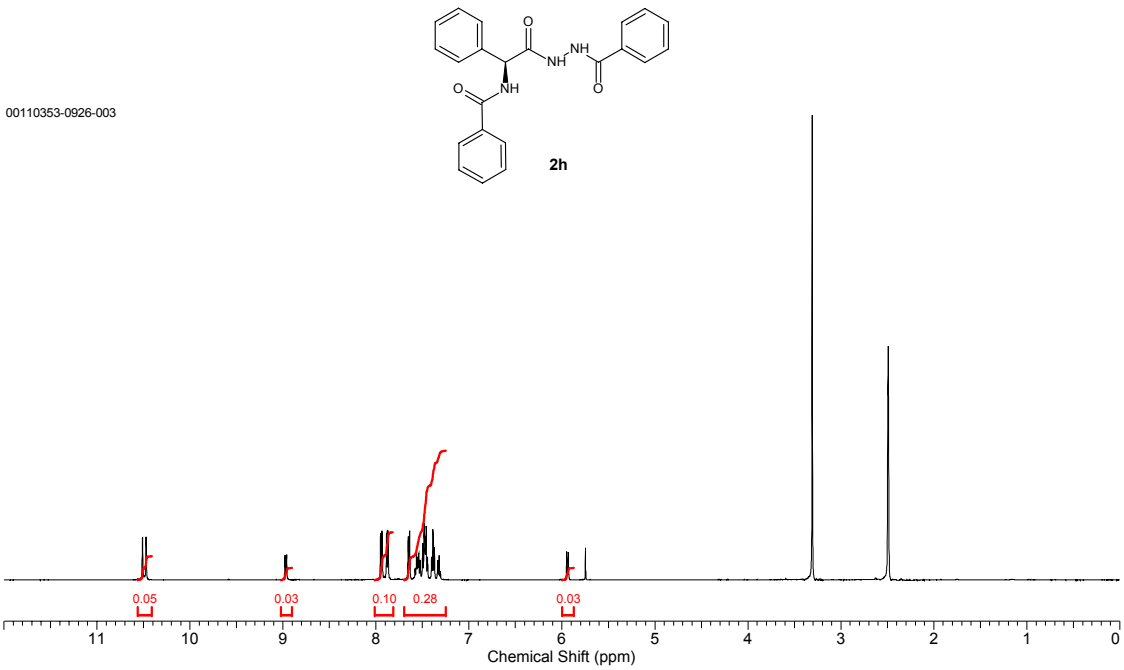
Acquisition Time (sec)	3.6829	***Comment***	340 mg solids	***Date***	Oct 10 2008	***Date Stamp***	Oct 10 2008
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS52624.TMP\PRODUCTION\UNITYN\PATELNC\00110021-0946-002						
Frequency (MHz)	399.67	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	54.00	***Solvent***	DMSO-d6
Spectrum Offset (Hz)	2398.0010	***Sweep Width (Hz)***	6394.88	***Temperature (degree C)***	25.000		



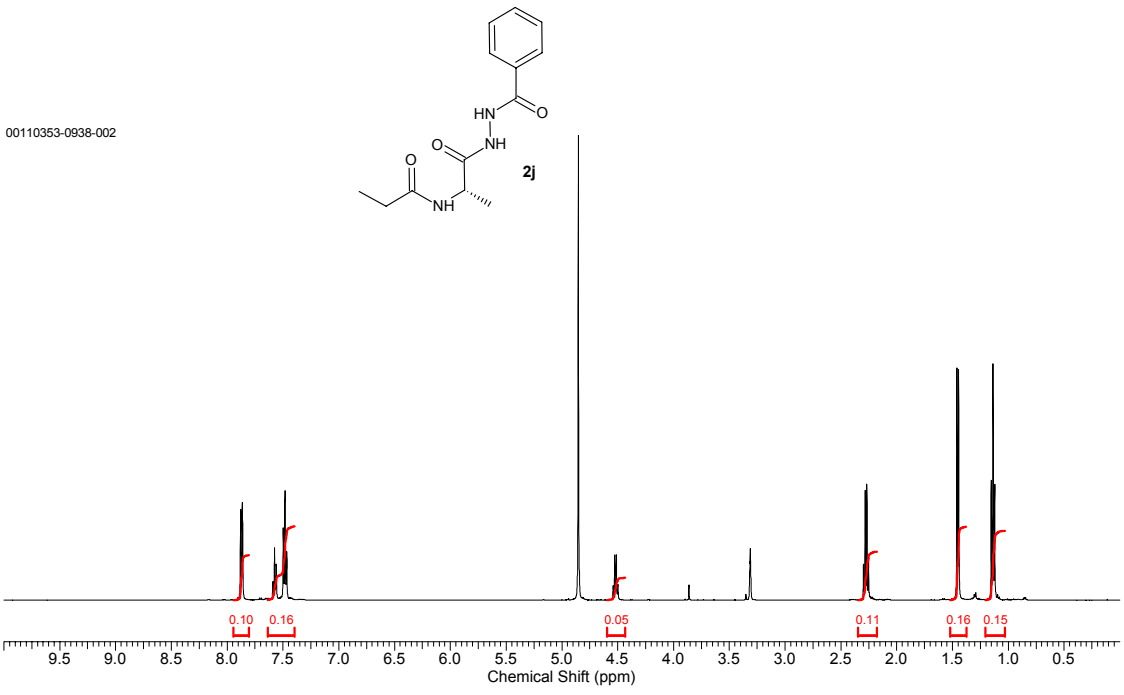
Acquisition Time (sec)	2.0488	***Date***	Jul 8 2009	***Date Stamp***	Jul 8 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43857.TMP\PRODUCTION\UNITYP\PATELNC\00110021-1137-011.2009189192607.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	256	***Original Points Count***	16384
Points Count	16384	***Pulse Sequence***	s2pul	***Receiver Gain***	44.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



Acquisition Time (sec)	2.9452	***Date***	Jun 17 2009	***Date Stamp***	Jun 17 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43859.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0926-003.2009168101340.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	48.00	***Solvent***	DMSO-d6
Spectrum Offset (Hz)	2998.9763	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



Acquisition Time (sec)	2.9452	***Date***	Jun 17 2009	***Date Stamp***	Jun 17 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43864.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0938-002.2009168133930.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	36.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



Acquisition Time (sec)	1.0863	***Date***	Jun 17 2009	***Date Stamp***	Jun 17 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43865.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0938-002.2009168135340.FID\FID				
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512
Original Points Count	32768	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	60.00	***Solvent***	METHANOL-d4	***Spectrum Offset (Hz)***	12568.0879
Sweep Width (Hz)	30165.91	***Temperature (degree C)***	25.000		

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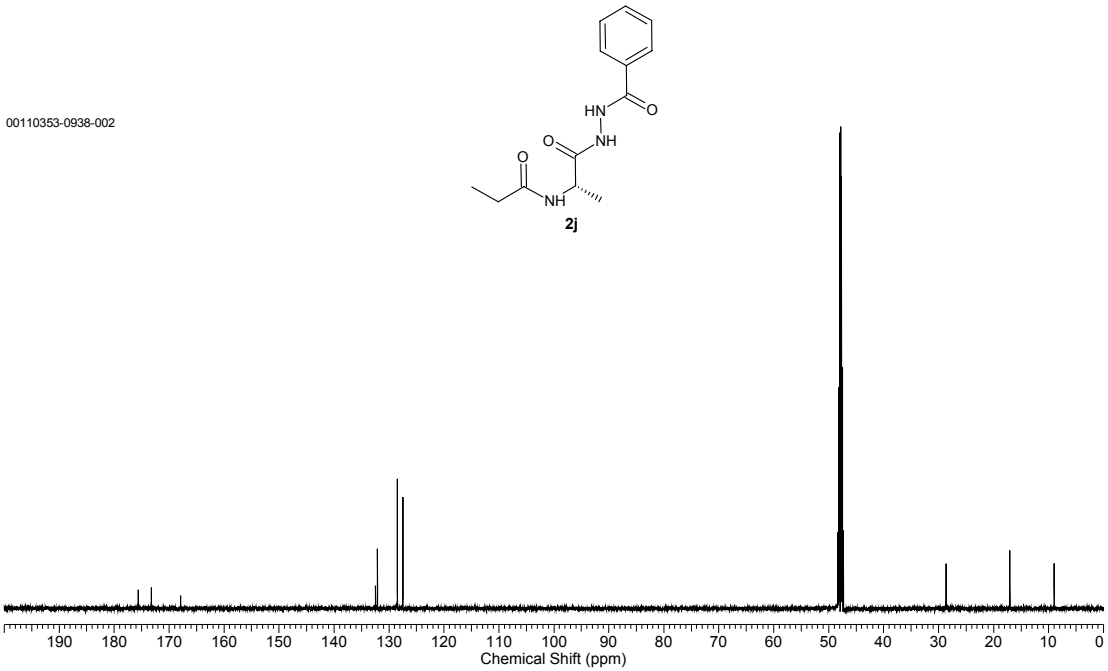
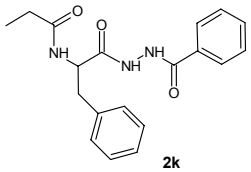


2j

Acquisition Time (sec)	2.9452	***Date***	May 26 2009	***Date Stamp***	May 26 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43866.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0903-001.2009146150540.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	38.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



2k

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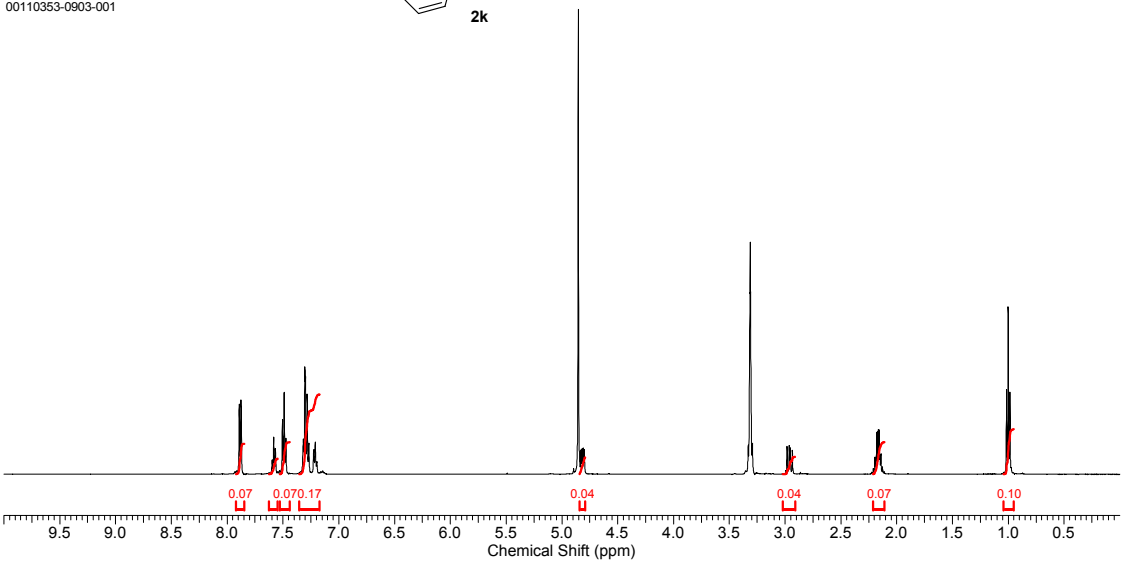
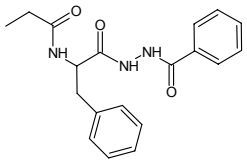


Acquisition Time (sec)	1.0863	***Date***	May 26 2009	***Date Stamp***	May 26 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43867.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0903-001.2009146151949.FID\FID				
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512
Original Points Count	32768	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	60.00	***Solvent***	METHANOL-d4	***Spectrum Offset (Hz)***	12568.0879
Sweep Width (Hz)	30165.91	***Temperature (degree C)***	25.000		



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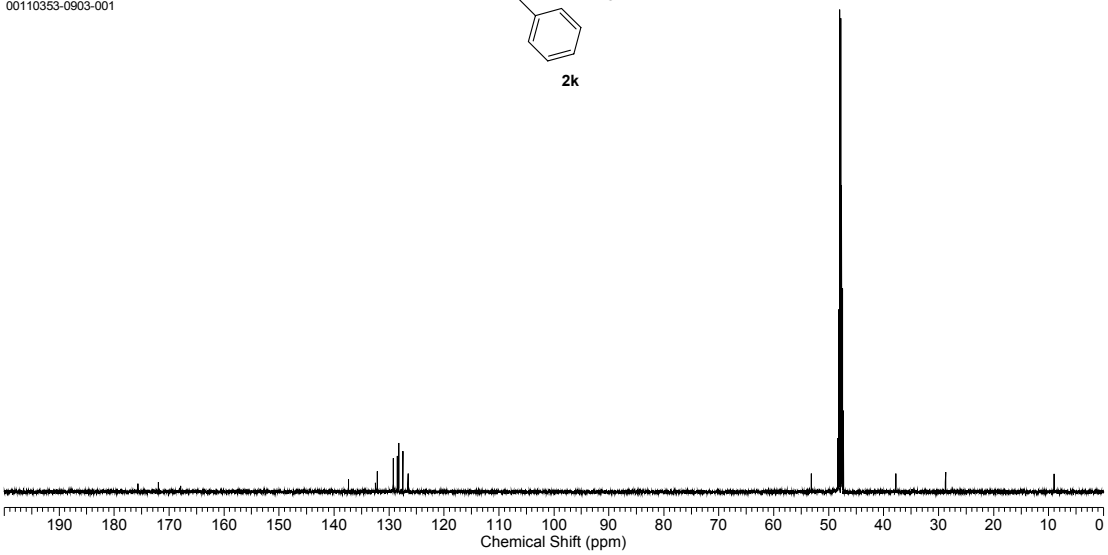
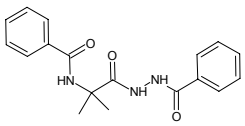
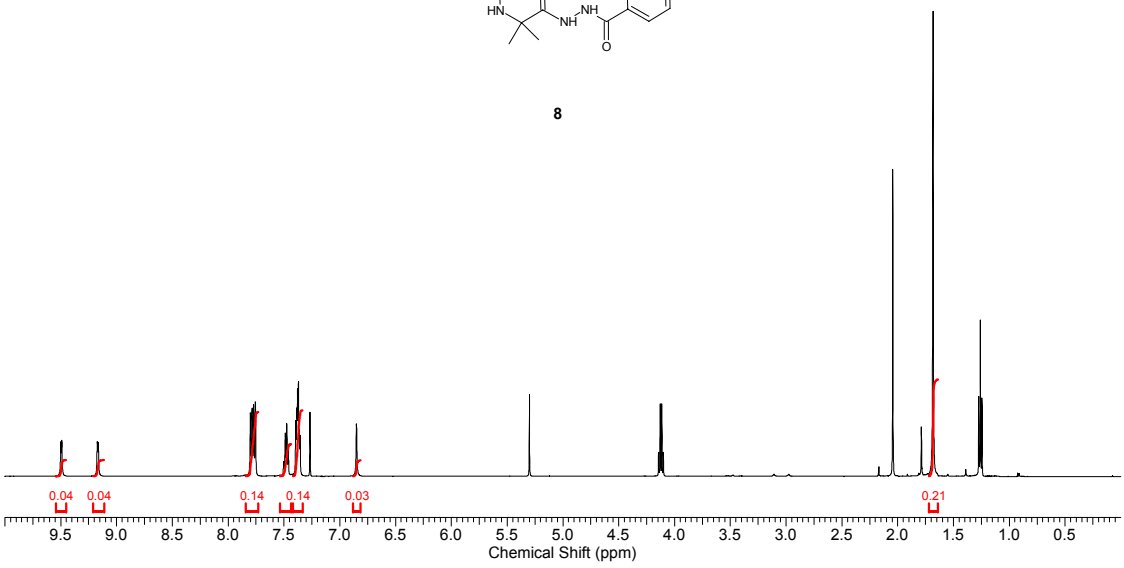
2k



Acquisition Time (sec)	2.9452	***Date***	Oct 14 2008	***Date Stamp***	Oct 14 2008		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS13455.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0610-001						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	38.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		



8



Acquisition Time (sec)	1.0863	***Date***	Jun 18 2009	***Date Stamp***	Jun 18 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS43873.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0939-002.2009169093137.FID\FID				
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512
Original Points Count	32768	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	60.00	***Solvent***	METHANOL-d4	***Spectrum Offset (Hz)***	12568.0879
Sweep Width (Hz)	30165.91	***Temperature (degree C)***	25.000		

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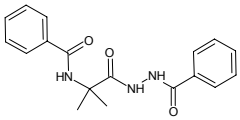

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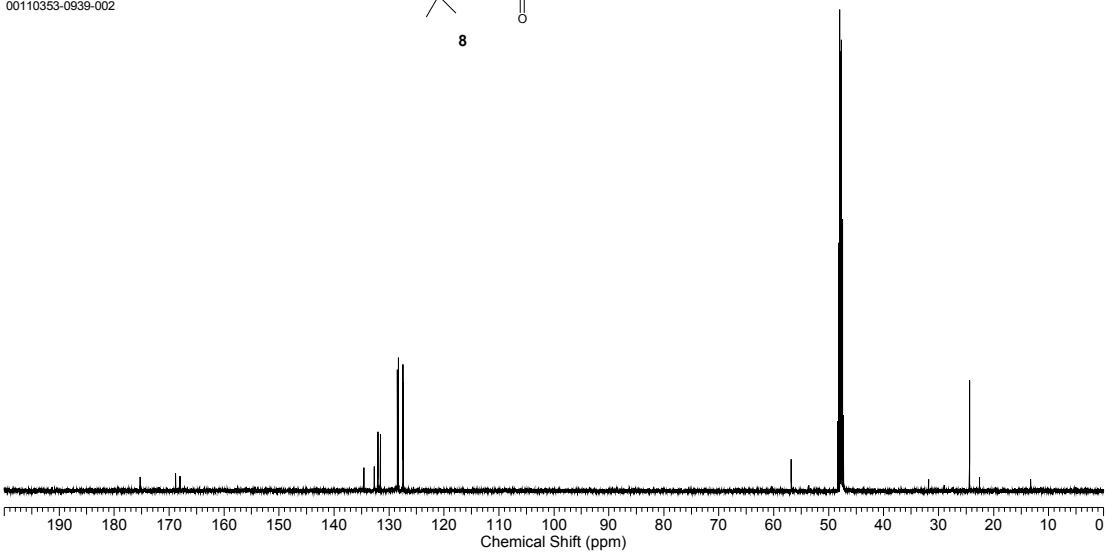

Acquisition Time (sec)	2.9452	***Date***	Jun 29 2009	***Date Stamp***	Jun 29 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS3101.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0940-001.2009180144825.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	48.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

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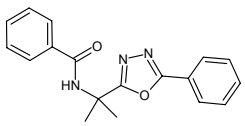
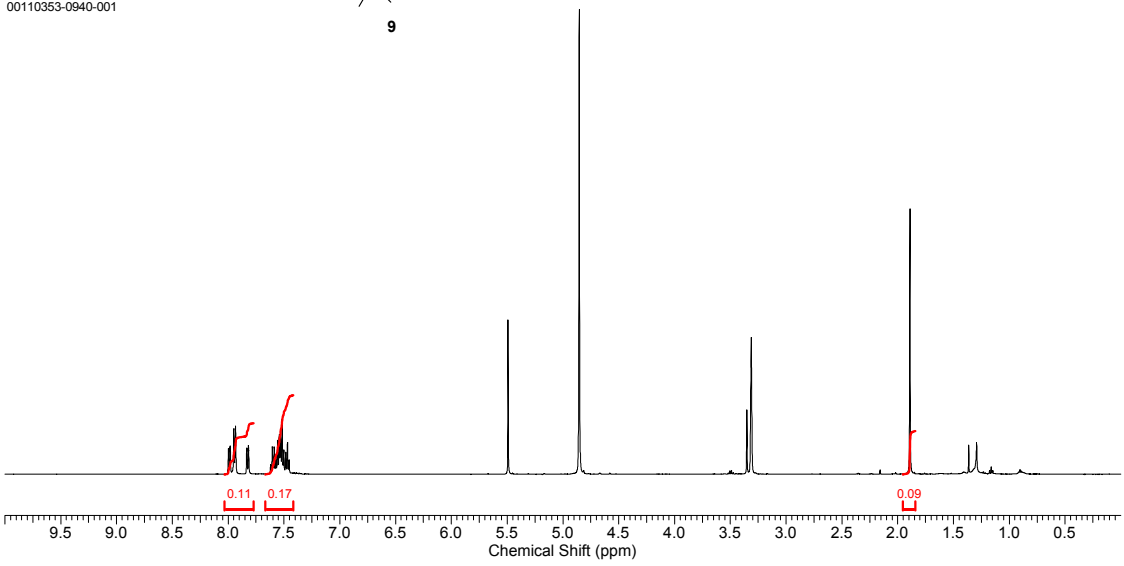


9



Acquisition Time (sec)	2.9452	***Date***	Jun 10 2009	***Date Stamp***	Jun 10 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS3104.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0934-001.2009161145639.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	44.00	***Solvent***	METHANOL-d4
Spectrum Offset (Hz)	2998.9304	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

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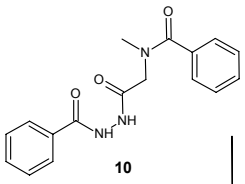
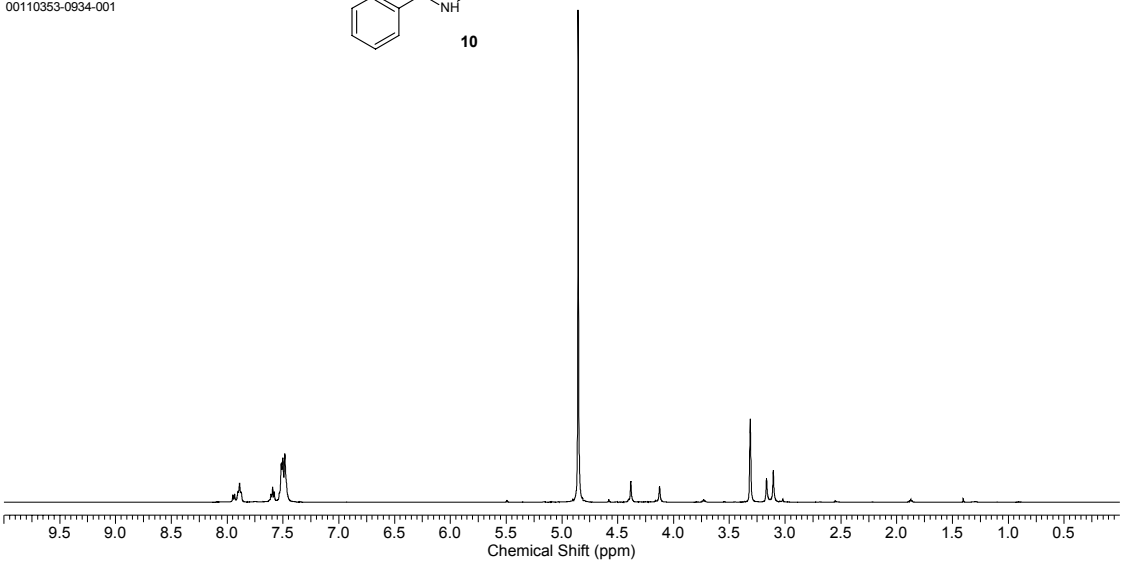


10



Acquisition Time (sec)	2.9452	***Date***	Jun 16 2009	***Date Stamp***	Jun 16 2009		
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS3110.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0935-001.2009167142259.FID\FID						
Frequency (MHz)	499.82	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	23552
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	38.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2998.9116	***Sweep Width (Hz)***	7996.80	***Temperature (degree C)***	25.000		

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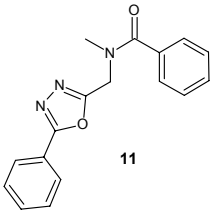
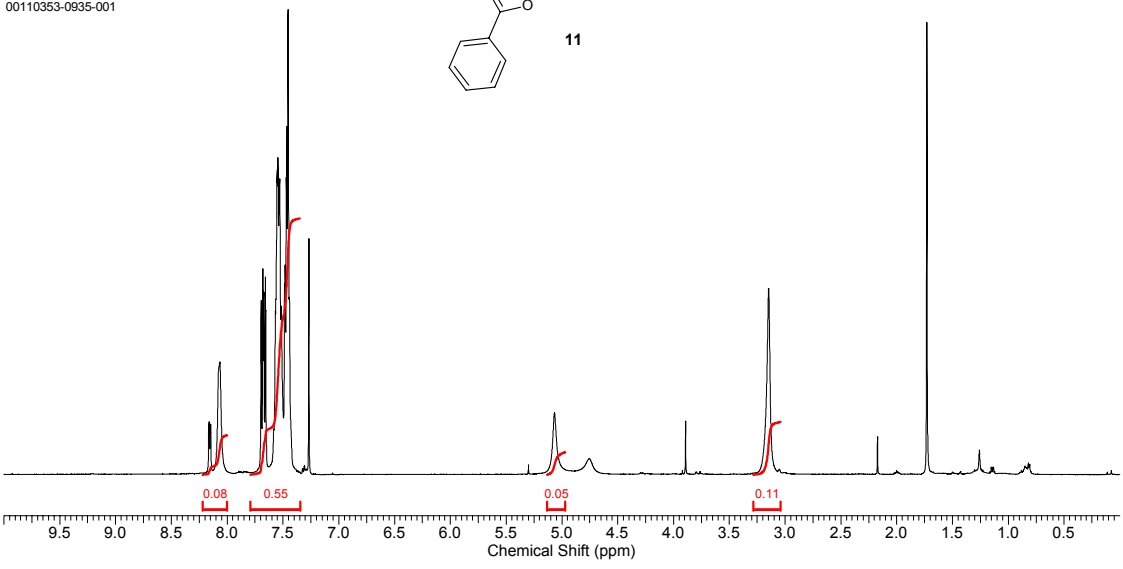


11



Acquisition Time (sec)	1.0863	***Date***	Jun 16 2009	***Date Stamp***	Jun 16 2009
File Name	C:\DOCUME~1\PATELNC\LOCALS~1\TEMP\GAINS3113.TMP\PRODUCTION\UNITYP\TRAN02\00110353-0935-001.2009167143707.FID\FID				
Frequency (MHz)	125.69	***Nucleus***	13C	***Number of Transients***	512
Original Points Count	32768	***Points Count***	32768	***Pulse Sequence***	s2pul
Receiver Gain	60.00	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	12567.9639
Sweep Width (Hz)	30165.91	***Temperature (degree C)***	25.000