Supplementary Material

Access to 2,5-disubstituted furans through a Passerini-Smiles/furyl rearrangement pathway

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General Experimental Details

Methanol was distilled from CaH_2 under N_2 immediately before use. All other reagents and solvents were commercial grade (Aldrich or Acros) and used without purification.

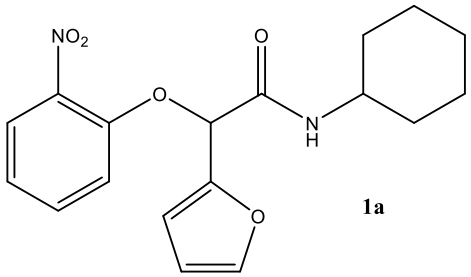
Thin layer chromatography (TLC) was performed using plastic-backed silica gel (225 μm) plates. Most of the compounds were purified via manual flash column chromatography that utilized 230–400 mesh silica gel from Sigma-Aldrich. Some compounds were purified via automated chromatography on the Biotage Isolera™ Flash Purification system using a gradient method with SNAP Ultra cartridges. Products were visualized by UV light, and/or the use of ceric ammonium molybdate, *p*-anisaldehyde, and potassium iodoplatinate solutions.

IR spectra were recorded on a Nicolet™ iS5 FT-IR Spectrometer and are reported in wavenumbers (cm^{-1}). Liquids and oils were analyzed as neat films on a NaCl plate (transmission), whereas solids were applied to a diamond plate (ATR). Nuclear magnetic resonance spectra (NMR) were acquired on a Bruker Ascend (400 MHz) Spectrometer and processed with TopSpin 3.2. Chemical shifts are measured relative to residual solvent peaks as an internal standard set to □ 7.26 and □ 77.1 ($CDCl_3$). HRMS FAB data was collected from a JEOL MStation [JMS-700] Mass Spectrometer at the University of Missouri-St Louis. Direct infusion mass spectrometry (DIMS) was used to verify accurate masses for new synthetic compounds, using a Triversa Nanomate nanospray direct infusion robot (Advion) attached to a Q-Exactive Mass Spectrometer. These samples were analyzed at the Proteomics & Mass Spectrometry Facility at the Danforth Plant Science Center (St Louis, MO). A Biotage Initiator+ microwave synthesizer was used for irradiation studies. 2-Allyl-6-nitrophenol was prepared from 2-nitrophenol at 175°C.[1,2]

[1] Bromidge, S. M.; Bertani, B.; Borriello, M.; Bozzoli, A.; Faedo, S.; Gianotti, M.; Gordon, L. J.; Hill, M.; Zucchelli, V.; Watson, J. M.; Zonzini, L. 8-[2-(4-Aryl-1-piperazinyl)ethyl]-2H-1,4-benzoxazin-3(4H)-ones: Dual-acting 5-HT1 receptor antagonists and serotonin reuptake inhibitors—Part II. *Bioorg. Med. Chem. Lett.*, **2009**, 19, 2338-2342.

[2] Rao, K.; Sirohi, R.; Shorey, M.; Kishore, D. Microwave Induced Lewis Acid Catalyzed Claisen Rearrangement of O-Allylaryl Ethers. *Int. J. Chem. Sci.* **2009**, *7*, 1667.

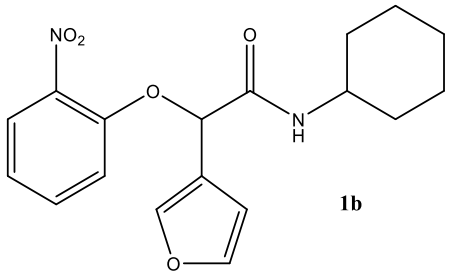
Synthesis of *N*-cyclohexyl-2-(furan-2-yl)-2-(2-nitrophenoxy)acetamide (1a).



1a

To a solution of 2-nitrophenol (27.8 mg, 0.2 mmol, 1 equiv) in acetonitrile (0.50 mL), 2-furaldehyde (22.5 µL, 0.26 mmol, 1.3 equiv), cyclohexyl isocyanide (37.3 µL, 0.3 mmol, 1.5 equiv), and i-Pr_2NEt (4 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 5% ethyl acetate in hexanes) providing compound **1a** (51.3 mg, 74%): R_f = 0.50 (50:50 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.2 Hz, 1H), 7.56-7.51 (m, 2H), 7.41 (d, *J* = 1.0 Hz, 1H), 7.22 (d, *J* = 8.3 Hz, 1H), 7.12-7.08 (m, 1H), 6.52 (d, *J* = 3.1 Hz, 1H), 6.36 (dd, *J* = 3.1, 1.7 Hz, 1H), 5.76 (s, 1H), 3.85-3.83 (m, 1H), 2.03-1.89 (m, 1H), 1.78-1.66 (m, 2H), 1.62-1.59 (m, 1H), 1.43-1.31 (m, 4H), 1.28-1.22 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 150.4, 148.4, 143.8, 139.6, 135.2, 126.7, 122.2, 116.3, 111.3, 111.0, 74.8, 48.5, 32.8, 25.2; IR: (diamond plate: [cm^{-1}]): 3386 (m) [N-H], 2930 (m) [C-H], 1585 (m) [C=O], 1247 (s) [C-N]; HR-FAB MS $[M+Na]^+$ calcd for $C_{18}H_{20}N_2O_5Na^+$ 367.12644; found 367.12698.

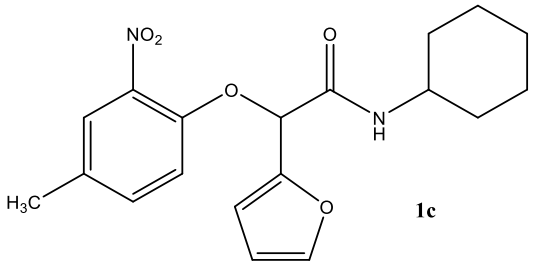
Synthesis of *N*-cyclohexyl-2-(furan-3-yl)-2-(2-nitrophenoxy)acetamide (1b).



1b

To a solution of 2-nitrophenol (140.3 mg, 1.0 mmol, 1 equiv) in acetonitrile (1.0 mL), 3-furaldehyde (113 µL, 1.3 mmol, 1.3 equiv), cyclohexyl isocyanide (188 µL, 1.5 mmol, 1.5 equiv), and i-Pr_2NEt (17.6 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 10% ethyl acetate in hexanes) providing compound **1b** (178.7 mg, 52%); R_f = 0.50 (50:50 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 1.4 Hz, 1H), 7.57 (s, 1H), 7.51 (dd, *J* = 15.9, 7.8Hz, 1H) 7.38-7.36 (m, 2H), 7.12-7.07 (m, 2H), 6.47 (d, *J* = 1.0 Hz, 1H), 5.68 (s, 1H), 3.81-3.74 (m, 1H) 1.91-1.86 (m, 2H), 1.72-1.69 (m, 2H), 1.61-1.58 (m, 1H), 1.40-1.31 (m, 3H), 1.30-1.20 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 150.4, 143.9, 141.0, 139.5, 135.1, 126.6, 121.9, 120.9, 116.0, 108.7, 74.9, 48.3, 32.8, 32.7, 25.5, 24.7; IR: (diamond plate: [cm^{-1}]): 3389 (m) [N-H], 2931 (m) [C-H], 1585 (s) [C=O], 1248 (m) [C-N]; HR-FAB MS $[M+Na]^+$ calcd for $C_{18}H_{20}N_2O_5Na^+$ 367.12644; found 367.12698.

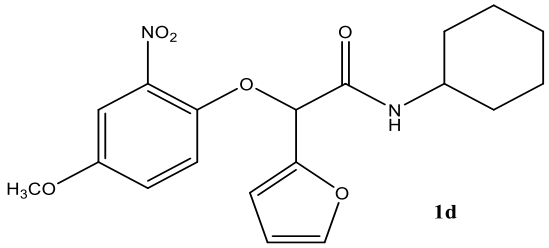
Synthesis of *N*-cyclohexyl-2-(furan-2-yl)-2-(4-methyl-2-nitrophenoxy)acetamide (1c).



1c

To a solution of 4-methyl-2-nitrophenol (79.0 mg, 0.5 mmol, 1 equiv) in acetonitrile (0.50 mL), 2-furaldehyde (55.6 µL, 0.7 mmol, 1.3 equiv), cyclohexyl isocyanide (96.4 µL, 0.8 mmol, 1.5 equiv), and i-Pr_2NEt (9 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing compound **1c** (101.2 mg, 50%); R_f = 0.30 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (s, 1H), 7.54 (br s, NH, 1H), 7.37 (d, *J* = 1.0 Hz, 1H), 7.30 (d, *J* = 8.5 Hz, 1H), 7.06 (d, *J* = 8.6 Hz, 1H), 6.48 (d, *J* = 3.0 Hz, 1H), 6.32 (d, *J* = 2.72, 1H), 5.69 (s, 1H), 3.82-3.80 (m, 1H), 2.30 (s, 3H), 1.96-1.86 (m, 3H), 1.72-1.56 (m, 4H), 1.35-1.19 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 148.5, 148.2, 143.7, 135.7, 132.3, 126.5, 116.4, 111.1, 110.8, 74.9, 48.4, 32.8, 31.0, 25.5, 24.7, 20.3; IR: (diamond plate: [cm^{-1}]): 3387 (m) [N-H], 2930 (m) [C-H], 1526 (s) [C=O], 1251 (s) [C-N]; HR-FAB MS $[M+Na]^+$ calcd for $C_{19}H_{22}N_2O_4Na^+$ 381.14209; found 381.14264.

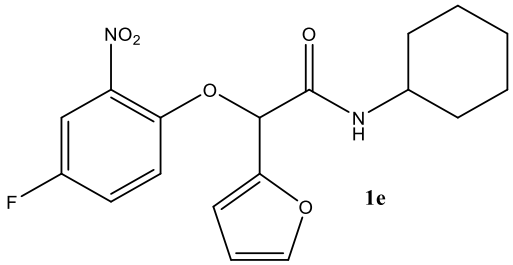
Synthesis of *N*-cyclohexyl-2-(furan-2-yl)-2-(4-methoxy-2-nitrophenoxy)acetamide (1d).



1d

To a solution of 4-methoxy-2-nitrophenol (85.5 mg, 0.5 mmol, 1 equiv) in acetonitrile (0.50 mL), 2-furaldehyde (54.4 µL, 0.7 mmol, 1.3 equiv), cyclohexyl isocyanide (94.3 µL, 0.8 mmol, 1.5 equiv), and i-Pr_2NEt (8.8 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 10% ethyl acetate in hexanes) providing compound **1d** (65.3 mg, 31%); R_f = 0.41 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (br s, NH, 1H), 7.44 (d, *J* = 2.1 Hz, 1H), 7.38 (s, 1H), 7.09-7.04 (m, 2H), 6.46 (d, *J* = 3.0 Hz, 1H), 6.33 (dd, *J* = 3.0, 1.7 Hz, 1H), 5.62 (s, 1H), 3.83-3.76 (m, 4H), 2.01-1.88 (m, 3H), 1.73-1.57 (m, 4H), 1.36-1.23 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4, 154.3, 148.7, 144.5, 143.8, 140.0, 121.7, 118.7, 111.3, 110.9, 110.4, 75.7, 56.2, 48.4, 32.8, 25.6, 24.8; IR: (diamond plate: [cm^{-1}]): 3388 (m) [N-H], 2930 (m) [C-H], 1524 (s) [C=O], 1218 (s) [C-N]; HR-FAB MS $[M+Na]^+$ calcd for $C_{19}H_{22}N_2O_6Na^+$ 397.13701; found 397.13754.

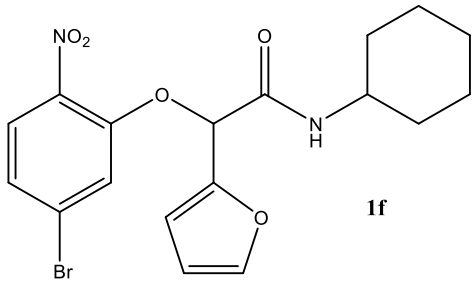
Synthesis of *N*-cyclohexyl-2-(4-fluoro-2-nitrophenoxy)-2-(furan-2-yl)acetamide (1e).



1e

To a solution of 4-fluoro-2-nitrophenol (157.7 mg, 1.0 mmol, 1 equiv) in acetonitrile (1.0 mL), 2-furaldehyde (108 µL, 1.3 mmol, 1.3 equiv), cyclohexyl isocyanide (187 µL, 1.5 mmol, 1.5 equiv), and i-Pr_2NEt (17.5 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing compound **1e** (216.0 mg, 60%); R_f = 0.33 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.69 (m, 1H), 7.46 (br s, NH, 1H), 7.40 (s, 1H), 7.30-7.25 (m, 1H), 7.26-7.19 (m, 1H), 6.52 (d, *J* = 3.0 Hz, 1H), 6.35 (d, *J* = 2.9 Hz, 1H), 5.72 (s, 1H), 3.86-3.84 (m, 1H), 2.00-1.99 (m, 2H), 1.75-1.72 (m, 2H), 1.63-1.60 (m, 1H), 1.38-1.22 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.9, 157.4, 154.9, 148.1, 146.8, 143.6, 139.4, 122.0, 118.3, 113.6, 110.6, 75.4, 48.3, 32.7, 32.6, 26.0, 25.5, 24.6; IR: (diamond plate: [cm^{-1}]): 3393 (m) [N-H], 2931 (m) [C-H], 1526 (s) [C=O], 1266 (s) [C-N].

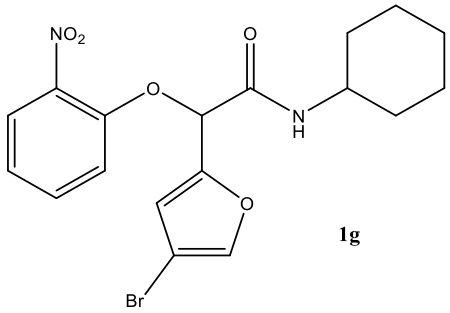
Synthesis of *2*-(5-bromo-2-nitrophenoxy)-*N*-cyclohexyl-2-(furan-2-yl)acetamide (1f).



1f

To a solution of 5-bromo-2-nitrophenol (110.6 mg, 0.51 mmol, 1 equiv) in acetonitrile (0.50 mL), 2-furaldehyde (54 µL, 0.65 mmol, 1.3 equiv), cyclohexyl isocyanide (93 µL, 0.75 mmol, 1.5 equiv), and i-Pr_2NEt (7 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing compound **1f** (165.3 mg, 77%): R_f = 0.60 (50:50 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.7 Hz, 1H), 7.51 (br d, *J* = 7.5 Hz, 1H, NH), 7.47-7.40 (m, 2H), 7.25 (dd, *J* = 8.7, 1.7 Hz, 1H), 6.57 (d, *J* = 2.9 Hz, 1H), 6.42-6.37 (m, 1H), 5.76 (s, 1H), 3.93-3.79 (m, 1H), 2.07-1.87 (m, 2H), 1.84-1.69 (m, 2H), 1.67-1.57 (m, 1H), 1.46-1.20 (m, 5H) ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 150.7, 147.7, 143.9, 138.3, 129.4, 127.6, 125.3, 119.9, 111.7, 110.9, 75.0, 48.4, 32.7, 32.6, 25.4, 24.6 (2C).

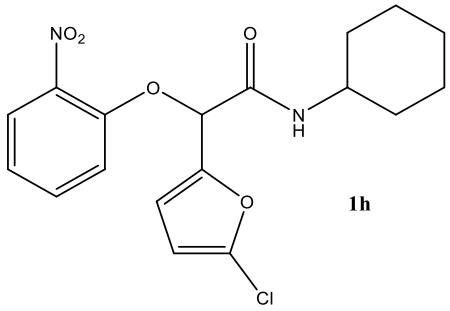
Synthesis of 2-(4-bromofuran-2-yl)-*N*-cyclohexyl-2-(2-nitrophenoxy)acetamide (1g).



1g

To a solution of 2-nitrophenol (140 mg, 1.0 mmol, 1 equiv) in acetonitrile (1.0 mL), 4-bromo-2-furaldehyde (230.4 mg, 1.3 mmol, 1.3 equiv), cyclohexyl isocyanide (189 µL, 1.5 mmol, 1.5 equiv), and i-Pr_2NEt (17.6 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 30% ethyl acetate in hexanes) providing compound **1g** (276.2 mg, 64%); R_f = 0.53 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.56 (m, 2H), 7.41 (s, 1H), 7.17-7.11 (m, 1H), 6.58 (s, 1H), 5.71 (s, 1H), 3.84-3.82 (m, 1H), 1.99-1.88 (m, 2H), 1.75-1.59 (m, 4H), 1.40-1.22 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.6, 150.2, 149.5, 142.0, 139.6, 135.3, 126.8, 122.5, 116.1, 114.3, 100.7, 74.4, 48.6, 32.9, 25.5, 24.8; IR: (diamond plate: [cm^{-1}]): 3382 (m) [N-H], 2930 (m) [C-H], 1585 (s) [C=O], 1247 (s) [C-N], 735 (w) [C-Br]; HR-FAB MS $[M+Na]^+$ calcd for $C_{18}H_{19}BrN_2O_5Na^+$ 445.03696; found 445.0315.

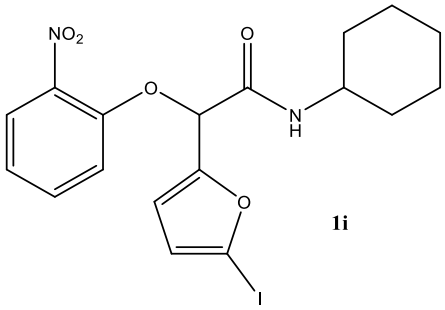
Synthesis of 2-(5-chlorofuran-2-yl)-*N*-cyclohexyl-2-(2-nitrophenoxy)acetamide (1h).



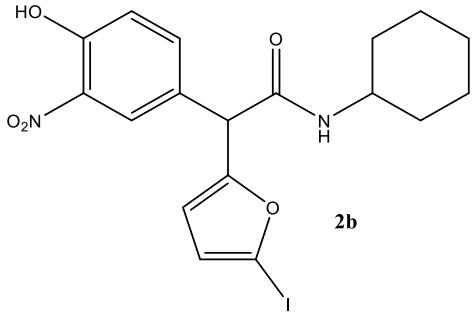
1h

To a solution of 2-nitrophenol (130.0 mg, 0.9 mmol, 1 equiv) in acetonitrile (935 µL), 5-chloro-2-furaldehyde (158.5 mg, 1.2 mmol, 1.3 equiv), cyclohexyl isocyanide (174.3 µL, 1.4 mmol, 1.5 equiv), and i-Pr_2NEt (16.3 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 20% ethyl acetate in hexanes) providing compound **1h** (108.5 mg, 31%); R_f = 0.33 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.2 Hz, 1H), 7.57 (m, 2H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.14 (dd, *J* = 9.2, 6.4 Hz, 1H), 6.52 (d, *J* = 3.2 Hz, 1H), 6.16 (d, *J* = 3.3 Hz, 1H), 5.68 (s, 1H), 3.85-3.83 (m, 1H), 2.01-1.89 (m, 2H), 1.79-1.60 (m, 4H), 1.42-1.23 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.6, 150.3, 148.0, 139.7, 138.1, 135.2, 126.8, 122.4, 116.2, 113.7, 107.7, 74.6, 48.7, 32.8, 25.6, 24.8; IR: (diamond plate: [cm^{-1}]): 3386 (m) [N-H], 2931 (m) [C-H], 1585 (s) [C=O], 1247 (s) [C-N], 740 (w) [C-Cl].

Synthesis of *N*-cyclohexyl-2-(5-iodofuran-2-yl)-2-(2-nitrophenoxy)acetamide (1i) and *N*-cyclohexyl-2-(4-hydroxy-3-nitrophenyl)-2-(5-iodofuran-2-yl)acetamide (2b).



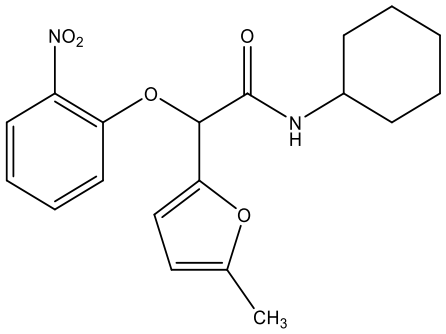
1i



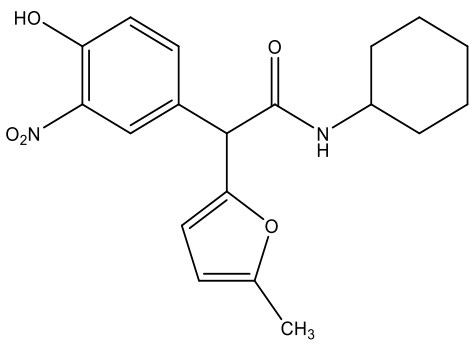
2b

To a solution of 2-nitrophenol (130.0 mg, 0.9 mmol, 1 equiv) in acetonitrile (935 µL), 5-iodo-2-furaldehyde (158.5 mg, 1.2 mmol, 1.3 equiv), cyclohexyl isocyanide (174 µL, 1.4 mmol, 1.5 equiv), and i-Pr_2NEt (16.3 µL, 10 mol%) were added. The reaction proceeds to stir for 72 h at 80 °C. Product was isolated with flash chromatography (SiO_2, 20% ethyl acetate in hexanes) providing compound **1i** and **2b** (105.8 mg, 25%). **1i:** R_f = 0.41 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.2 Hz, 1H), 7.59-7.55 (m, 2H), 7.20 (d, *J* = 8.2 Hz, 1H), 7.13 (dd, *J* = 7.4, 7.4 Hz, 1H) 6.52 (d, *J* = 3.3 Hz, 1H), 6.4 (d, *J* = 3.3 Hz, 1H), 5.73 (s, 1H), 3.85-3.83 (m, 1H), 2.00-1.88 (m, 2H), 1.79-1.72 (m, 3H), 1.62-1.60 (m, 2H), 1.41-1.22 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.8, 153.9, 150.3, 139.7, 135.2, 126.7, 122.4, 121.6, 116.3, 114.1, 89.9, 74.4, 48.6, 32.9, 32.7, 25.6, 24.8. Compound **2b** observed as inseparable mixture of **1i** and **2b**. **2b:** R_f = 0.51 (40:60 EtOAc:Hex).

Synthesis of *N*-cyclohexyl-2-(5-methylfuran-2-yl)-2-(2-nitrophenoxy)acetamide (1j) and *N*-cyclohexyl-2-(4-hydroxy-3-nitrophenyl)-2-(5-methylfuran-2-yl)acetamide (2c).



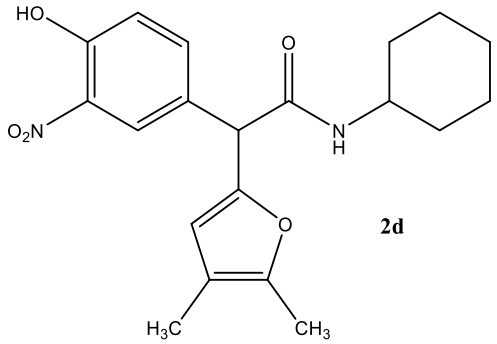
1j



2c

To a solution of 2-nitrophenol (418.6 mg, 3.0 mmol, 1 equiv) in acetonitrile (3.0 mL), 5-methyl-2-furaldehyde (389.1 µL, 3.9 mmol, 1.3 equiv), cyclohexyl isocyanide (561.2 µL, 4.5 mmol, 1.5 equiv), and i-Pr_2NEt (52.4 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing **1j** and **2c**. Compound **1j** observed as inseparable mixture of **1j** and **2c** (186.3 mg, 18%); **1j:** R_f = 0.41 (40:60 EtOAc:Hex). **2c**: R_f = 0.78 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.55 (s, 1H), 8.03 (s, 1H), 7.59 (d, *J* = 8.7 Hz, 1H), 7.11 (d, *J* = 8.7, 1H), 6.06 (d, *J* = 2.8 Hz, 1H), 5.92 (d, *J* = 2.6 Hz, 1H), 5.79 (br s, NH, 1H), 4.77 (s, 1H), 3.82-3.75 (m, 1H), 2.26 (s, 3H), 1.93-1.84 (m, 2H), 1.66-1.57 (m, 3H), 1.40-1.29 (m, 2H), 1.19-1.10 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.2, 154.6, 153.0, 149.6, 138.3, 133.5, 130.2, 124.8, 120.3, 109.9, 106.8, 52.0, 48.8, 33.0, 25.6, 24.8, 13.8. HR-FAB MS $[M+H]^+$ calcd for $C_{19}H_{23}N_2O_4^+$ 359.1605; found 359.16071.

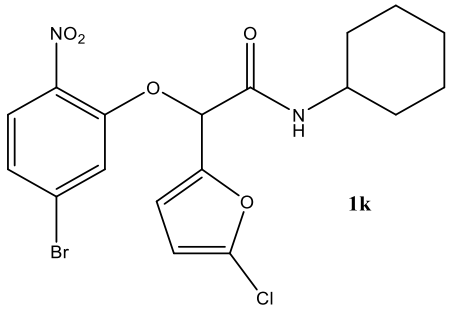
Synthesis of *N*-cyclohexyl-2-(4,5-dimethylfuran-2-yl)-2-(4-hydroxy-3-nitrophenyl)acetamide (2d).



2d

To a solution of 2-nitrophenol (140.5 mg, 1.0 mmol, 1 equiv) in acetonitrile (1.0 mL), 4,5-dimethyl-2-furaldehyde (160.4 µL, 1.3 mmol, 1.3 equiv), cyclohexyl isocyanide (188.4 µL, 1.5 mmol, 1.5 equiv), and i-Pr_2NEt (17.6 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing compound **2d** (103.2 mg, 27%); R_f = 0.74 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.54 (s, 1H), 8.03 (s, 1H), 7.58 (d, *J* = 8.7 Hz, 1H), 7.10 (d, *J* = 8.7 Hz, 1H), 5.94 (s, 1H), 5.86 (br s, NH, 1H), 4.72 (s, 1H), 3.78-3.77 (m, 1H), 2.15 (s, 3H), 2.04-1.89 (m, 5H), 1.84-1.66 (m, 3H), 1.65-1.39 (m, 2H), 1.37-1.18 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 154.5, 148.2, 138.2, 133.4, 130.4, 124.8, 120.2, 115.2, 112.4, 51.9, 48.8, 33.0, 32.8, 25.6, 24.8, 11.5, 10.0.

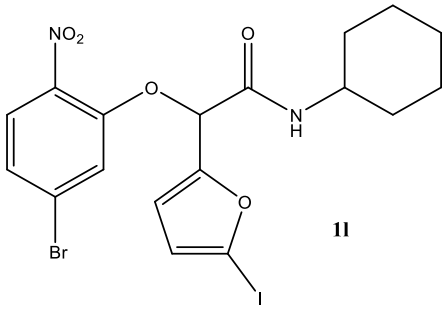
Synthesis of 2-(5-bromo-2-nitrophenoxy)-2-(5-chlorofuran-2-yl)-*N*-cyclohexylacetamide (1k).



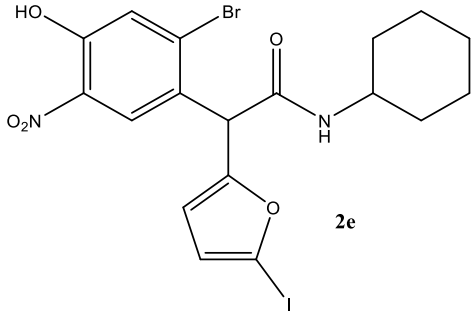
1k

To a solution of 5-bromo-2-nitrophenol (109.0 mg, 0.50 mmol, 1 equiv) in acetonitrile (0.50 mL), 5-chloro-2-furaldehyde (84.0 mg, 0.65 mmol, 1.3 equiv), cyclohexyl isocyanide (93 µL, 0.75 mmol, 1.5 equiv), and i-Pr_2NEt (9 µL, 10 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing compound **1k** (16.1 mg, 7%); R_f = 0.56 (33:67 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.8 Hz, 1H), 7.50 (br d, *J* = 8.0 Hz, NH, 1H), 7.39 (d, *J* = 1.8 Hz, 1H), 7.29 (dd, *J* = 8.7, 1.8 Hz, 1H), 6.58 (d, *J* = 3.4 Hz, 1H), 6.19 (d, *J* = 3.4 Hz, 1H), 5.68 (s, 1H), 3.91-3.81 (m, 1H), 2.05-1.98 (m, 1H), 1.96-1.89 (m, 1H), 1.83-1.71 (m, 2H), 1.67-1.58 (m, 1H), 1.44-1.22 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.0, 150.6, 147.2, 138.4, 138.3, 129.5, 127.7, 125.6, 119.7, 114.1, 107.1, 74.7, 48.6, 32.7, 32.6, 25.4, 24.6 (2C).

Synthesis of 2-(5-bromo-2-nitrophenoxy)-*N*-cyclohexyl-2-(5-iodofuran-2-yl)acetamide (1l) and 2-(2-bromo-4-hydroxy-5-nitrophenyl)-*N*-cyclohexyl-2-(5-iodofuran-2-yl)acetamide (2e).



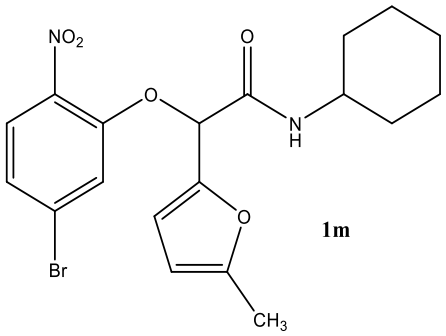
1l



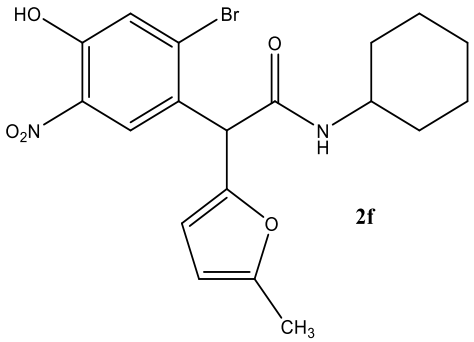
2e

To a solution of 5-bromo-2-nitrophenol (109.9 mg, 0.50 mmol, 1 equiv) in acetonitrile (0.50 mL), 5-iodo-2-furaldehyde (145.1 mg, 0.65 mmol, 1.3 equiv), cyclohexyl isocyanide (93 µL, 1.5 mmol, 1.5 equiv), and i-Pr_2NEt (9 µL, 11 mol%) were added. The reaction proceeds to stir for 72 h at 80 °C. Product was isolated with flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing an inseparable mixture of compounds **1l** and **2e** (47.1 mg, 17%)**. 1l:** R_f = 0.56 (33:67 EtOAc:Hex). **2e:** R_f = 0.50 (33:67 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.53 (s, 1H), 8.05 (s, 1H), 7.48 (s, 1H), 6.57 (d, *J* = 3.2 Hz, 1H), 6.27 (d, *J* = 3.5 Hz, 1H), 5.87 (d, *J* = 3.5 Hz, 1H), 5.25 (s, 1H), 3.87-3.77 (m, 1H), 2.00-1.86 (m, 2H), 1.77-1.70 (m, 4H), 1.43-1.31 (m, 2H), 1.24-1.13 (m, 2H).

Synthesis of 2-(5-bromo-2-nitrophenoxy)-*N*-cyclohexyl-2-(5-methylfuran-2-yl)acetamide (1m) and 2-(2-bromo-4-hydroxy-5-nitrophenyl)-*N*-cyclohexyl-2-(5-methylfuran-2-yl) acetamide (2f).



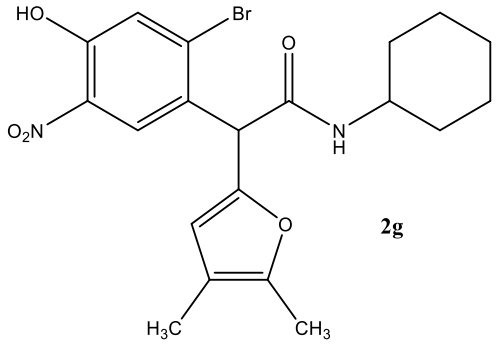
1m



2f

To a solution of 5-bromo-2-nitrophenol (110.0 mg, 0.50 mmol, 1 equiv) in acetonitrile (0.50 mL), 5-methyl-2-furaldehyde (65 µL, 0.65 mmol, 1.3 equiv), cyclohexyl isocyanide (93 µL, 0.75 mmol, 1.5 equiv), and i-Pr_2NEt (9 µL, 12 mol%) were added. The reaction was stirred for 72 h at 80 °C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing **1m** and **2f** (128.0 mg, 58%). Compound **1m** observed as inseparable mixture of **1m** and **2f. 1m:** R_f = 0.40 (33:67 EtOAc:Hex), **2f**: R_f = 0.38 (33:67 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.87 (s, 1H), 8.06 (s, 1H), 7.45 (s, 1H), 6.15 (d, *J* = 3.1 Hz, 1H), 5.97 (dd, *J* = 3.0, 0.9 Hz, 1H), 5.67 (br d, *J* = 7.6 Hz, NH, 1H), 5.18 (s, 1H), 3.88-3.78 (m, 1H), 2.27 (s, 3H), 1.99-1.86 (m, 2H), 1.73-1.57 (m, 4H), 1.39-1.31 (m, 2H), 1.21-1.10 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 153.8, 153.0, 134.4, 130.9, 130.1, 128.8, 126.0, 123.9, 110.4, 106.8, 68.2, 51.5, 48.7, 32.7, 29.7, 25.4, 24.6, 13.6.

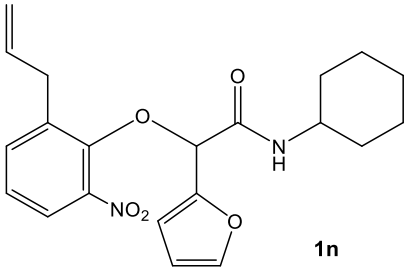
Synthesis of 2-(2-bromo-4-hydroxy-5-nitrophenyl)-*N*-cyclohexyl-2-(4,5-dimethylfuran-2-yl) acetamide (2g).



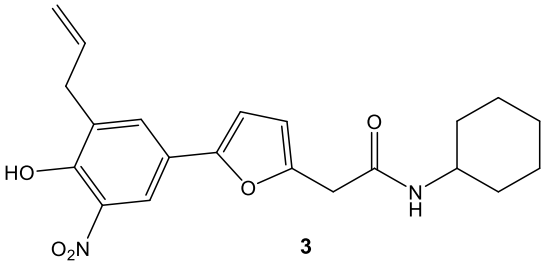
2g

To a solution of 5-bromo-2-nitrophenol (100.4 mg, 0.46 mmol, 1 equiv) in acetonitrile (0.50 mL), 4,5-dimethyl-2-furaldehyde (79 µL, 0.65 mmol, 1.4 equiv), cyclohexyl isocyanide (93 µL, 0.75 mmol, 1.6 equiv), and i-Pr_2NEt (9 µL, 13 mol%) were added. The reaction was stirred for 72 h at 80°C. Product was isolated via flash chromatography (SiO_2, 15% ethyl acetate in hexanes) providing compound **2g** (54.0 mg, 26%); R_f = 0.58 (33:67 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.49 (s, 1H), 8.07 (s, 1H), 7.43 (s, 1H), 6.01 (s, 1H), 5.65 (br s, NH, 1H), 5.12 (s, 1H), 3.87-3.77 (m, 1H), 2.17 (s, 3H), 1.91 (s, 3H), 1.74-1.56 (m, 5H), 1.44-1.32 (m, 3H), 1.21-1.06 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 153.8, 148.2, 147.0, 134.4, 133.0, 130.2, 126.0, 123.8, 115.2, 112.8, 51.5, 48.8, 32.81, 32.76, 29.7, 25.5, 24.7, 11.4, 9.9.

Synthesis of 2-(2-allyl-6-nitrophenoxy)-N-cyclohexyl-2-(furan-2-yl)acetamide (1n) and 2-(5-(3-allyl-4-hydroxy-5-nitrophenyl)furan-2-yl)-*N*-cyclohexylacetamide (3).



1n



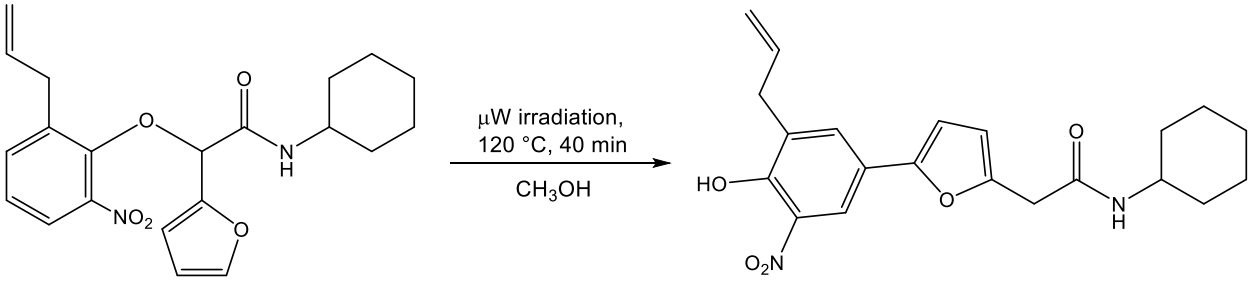
3

To a solution of 2-allyl-6-nitrophenol (86.5 mg, 0.48 mmol) in methanol (0.50 mL), 2-furaldehyde (40.0 µL, 0.48 mmol) and cyclohexyl isocyanide (60.5 µL, 0.49 mmol) were added. The reaction was stirred for 48 h at 65°C. Product was isolated via flash chromatography (SiO_2, 20% ethyl acetate in hexanes) to afford compounds **1n** (12.9 mg, 7%) and **3** (29.5 mg, 16%). Under alternate conditions (50°C for 48h), to afford compounds **1n** (48.0 mg, 26%) and **3** (5.6 mg, 3%).

1n: R_f = 0.51 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.1 Hz, 1H), 7.41 (d, *J* = 7.7 Hz, 1H), 7.32 (s, 1H), 7.20-7.14 (m, 2H), 6.23 (s, 2H), 5.90-5.78 (m, 1H), 5.43 (s, 1H), 5.16 (d, *J* = 10.0 Hz, 1H), 5.08 (d, *J* = 17.0 Hz, 1H), 4.00-3.90 (m, 1H), 3.34 (dd, *J* = 16.0, 6.7 Hz, 1H), 3.17 (dd, *J* = 16.0, 6.0 Hz, 1H), 2.09-2.02 (m, 2H), 1.84-1.76 (m, 2H), 1.69-1.62 (m, 1H), 1.50-1.25 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 146.9, 144.7, 144.0, 143.3, 136.7, 135.4, 135.0, 124.7, 123.8, 119.5, 117.6, 108.9, 77.8, 48.2, 34.0, 33.0, 32.8, 25.5, 24.7 (2C).

3: R_f = 0.29 (40:60 EtOAc:Hex); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.96 (s, 1H), 8.21 (d, *J* = 1.9 Hz, 1H), 7.69 (s, 1H), 6.58 (d, *J* = 3.2 Hz, 1H), 6.31 (d, *J* = 3.1 Hz, 1H), 6.06-5.94 (m, 1H), 5.54 (br s, NH, 1H), 5.17 (s, 1H), 5.14 (d, *J* = 4.6 Hz, 1H), 3.84-3.74 (m, 1H), 3.64 (s, 2H), 3.51 (d, *J* = 6.4 Hz, 2H), 1.94-1.85 (m, 2H), 1.70-1.53 (m, 4H), 1.41-1.28 (m, 2H), 1.16-1.06 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 152.5, 151.3, 149.2, 134.8, 132.6, 132.1, 128.8, 122.9, 117.4, 117.2, 110.7, 106.6, 48.5, 36.7, 33.8, 32.9, 29.7, 25.5, 24.9, 19.2.

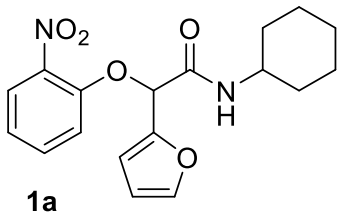
Conversion of 1n to 3: Synthesis of 2-(5-(3-allyl-4-hydroxy-5-nitrophenyl)furan-2-yl)-*N*-cyclohexylacetamide (3).



1n (PS) **3 (PS-R)**

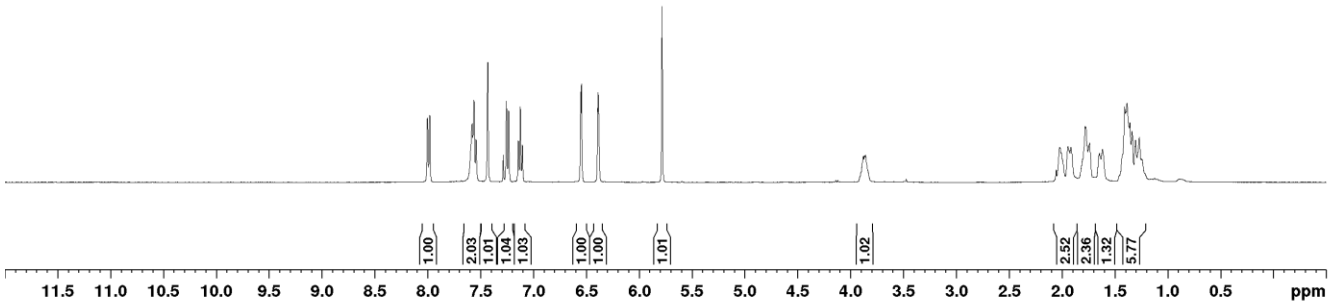
A solution of **1n** (20.5 mg, 0.053 mmol) in 0.50 mL methanol was submitted to microwave irradiation (120°C, 40 minutes). Volatiles were removed *in vacuo* and crude reaction mixture submitted to flash chromatography (SiO_2, 20% ethyl acetate in hexanes) to afford hydroxyphenyl furan **3** (10.7 mg, 52%): R_f = 0.29 (40:60 EtOAc:Hex).

NMR Characterization Data for New Compounds



1a

^{1}H NMR, $CDCl_3$
400 MHz

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RG                    57.34
DW                   62.400 usec
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TE                   292.0 K
D1                1.00000000 sec
TD0                       1

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NUC1                     1H
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PLW1           9.80000019 W

F2 - Processing parameters
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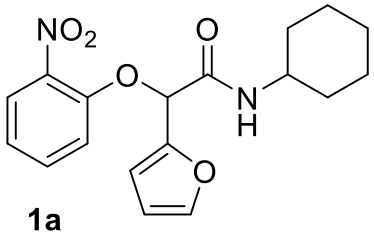
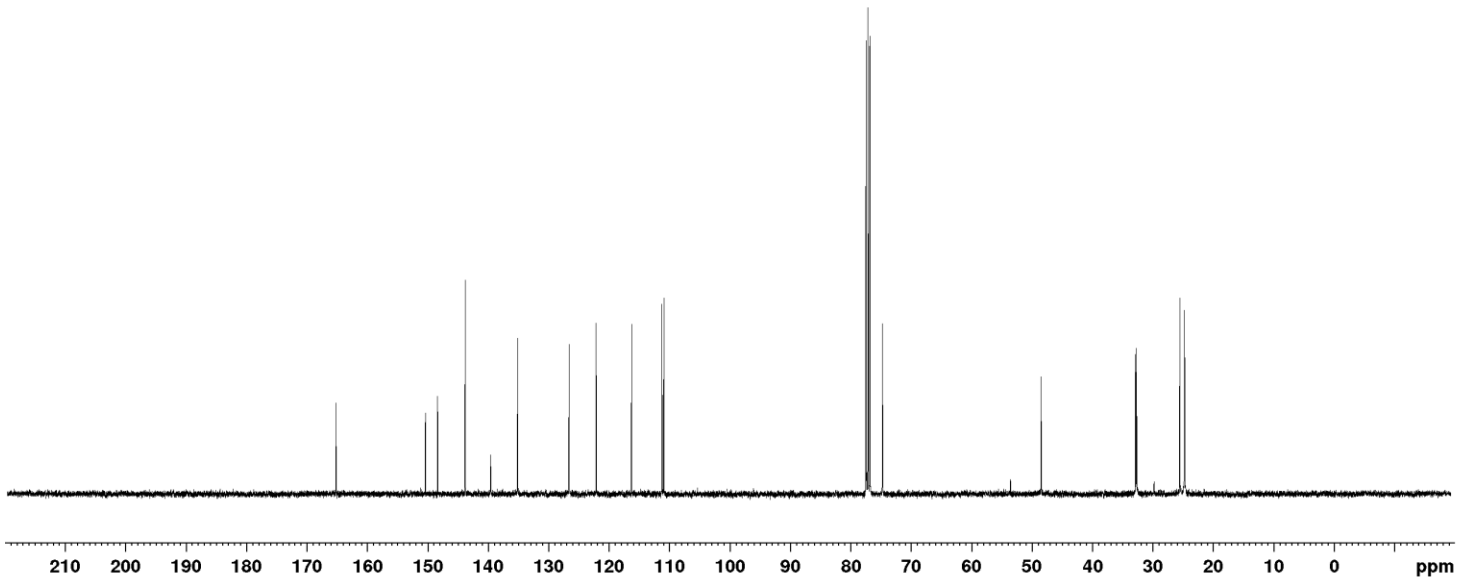


1a

^{13}C NMR, $CDCl_3$
100 MHz

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TD0                   1

======== CHANNEL f1 ========
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NUC1                13C
P1                10.00 usec
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======== CHANNEL f2 ========
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NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

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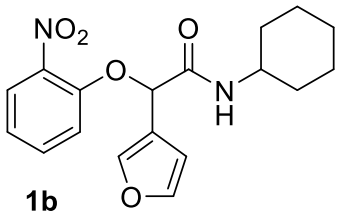

1b

^{1}H NMR, $CDCl_3$
400 MHz


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Current Data Parameters
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EXPNO                   10
PROCNO                   1

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TD                 65536
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NS                    16
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SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  82.91
DW                62.400 usec
DE                 6.50 usec
TE                293.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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NUC1                   1H
P1                  15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
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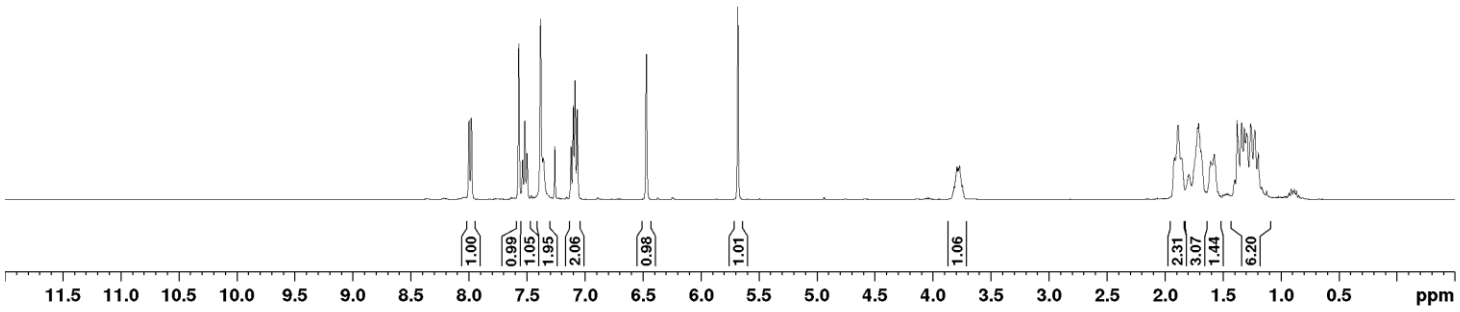
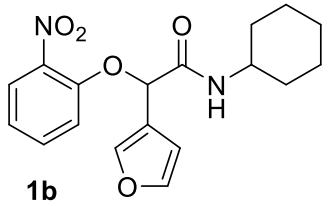
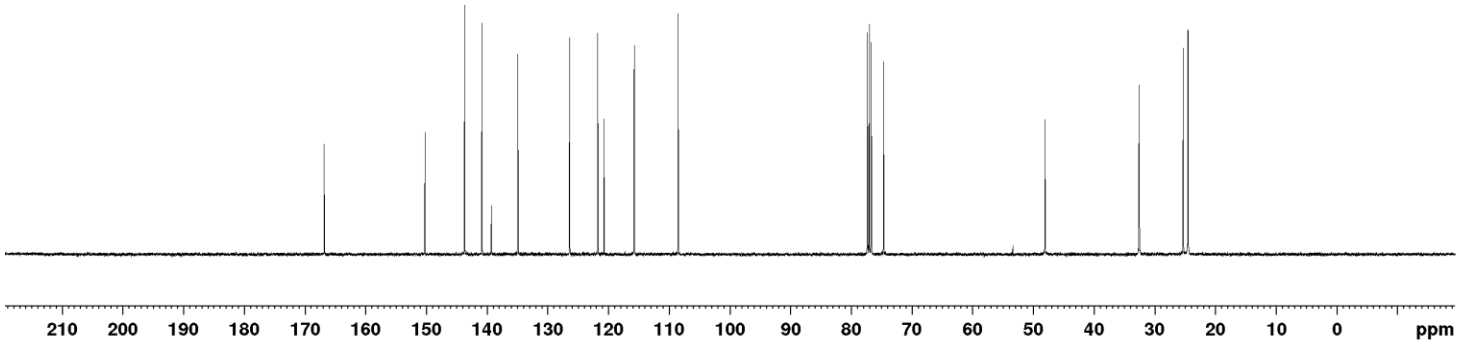




1b

^{13}C NMR, $CDCl_3$
100 MHz

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DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG               210.96
DW               20.800 usec
DE               10.00 usec
TE               294.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1              13C
P1               10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
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PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

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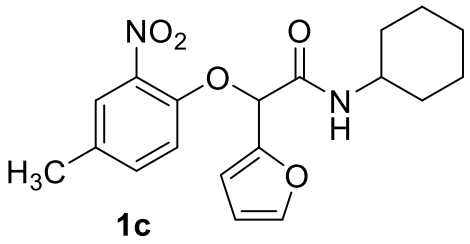
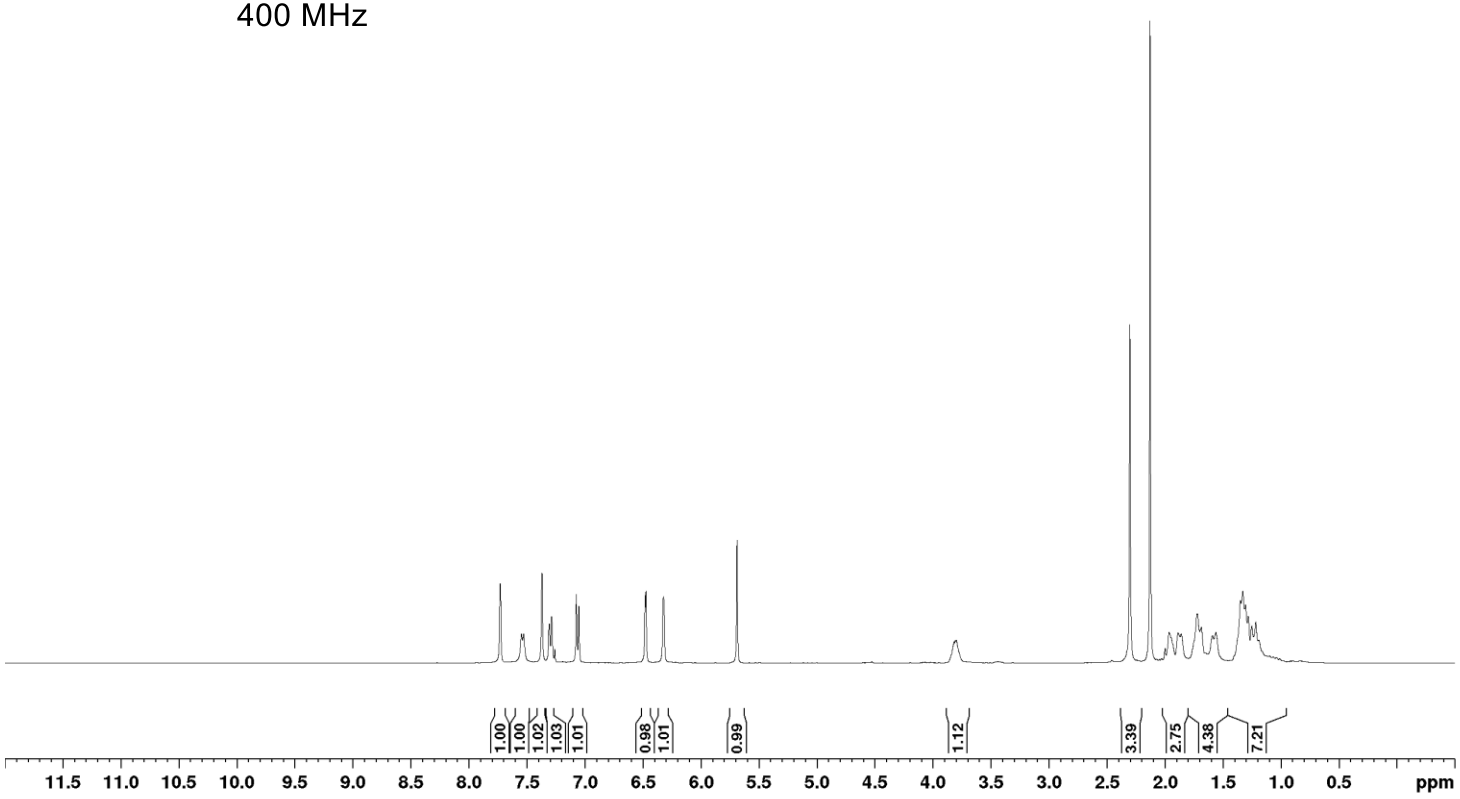


1c

^{1}H NMR, $CDCl_3$
400 MHz

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PROCNO                   1

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DS                     2
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FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  32.72
DW                 62.400 usec
DE                  6.50 usec
TE                292.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                   1H
P1                  15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
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PC                  1.00
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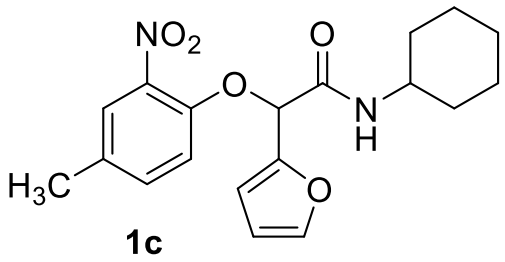
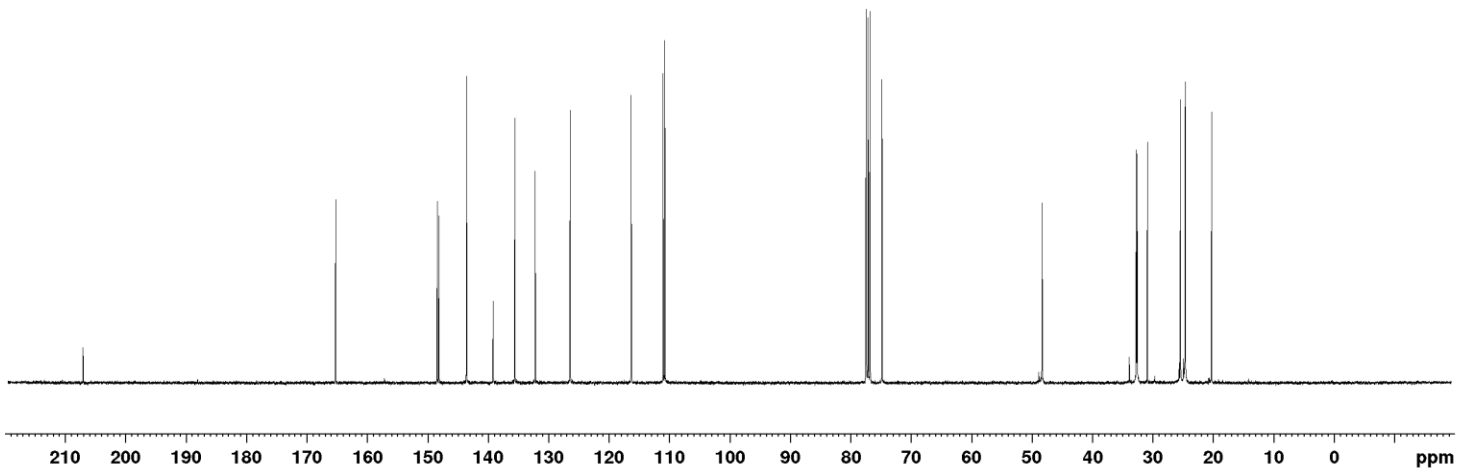
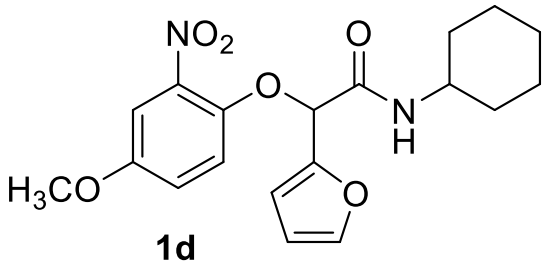


1c

^{13}C NMR, $CDCl_3$
100 MHz



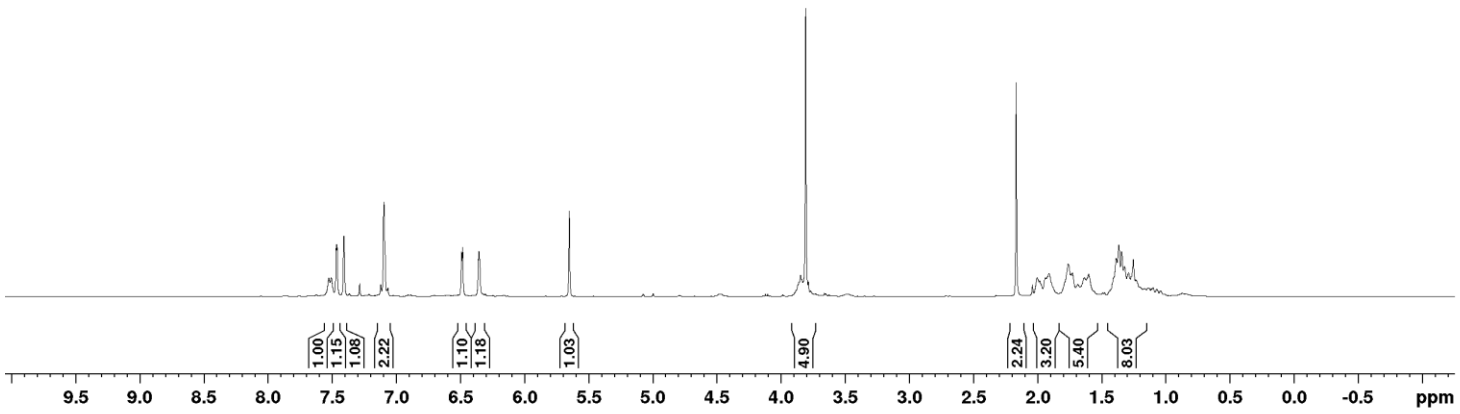

```
Current Data Parameters
NAME      C.S. 2-103-45 Flush
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151209
Time                8.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                294.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                 32768
SF           100.6177912 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

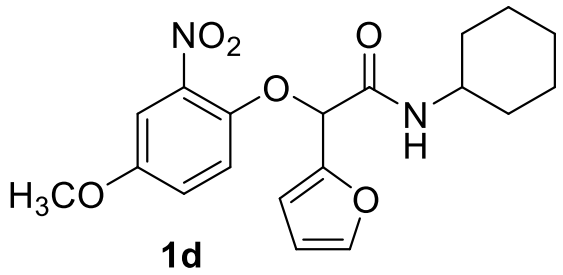


1d

^{1}H NMR, $CDCl_3$
400 MHz

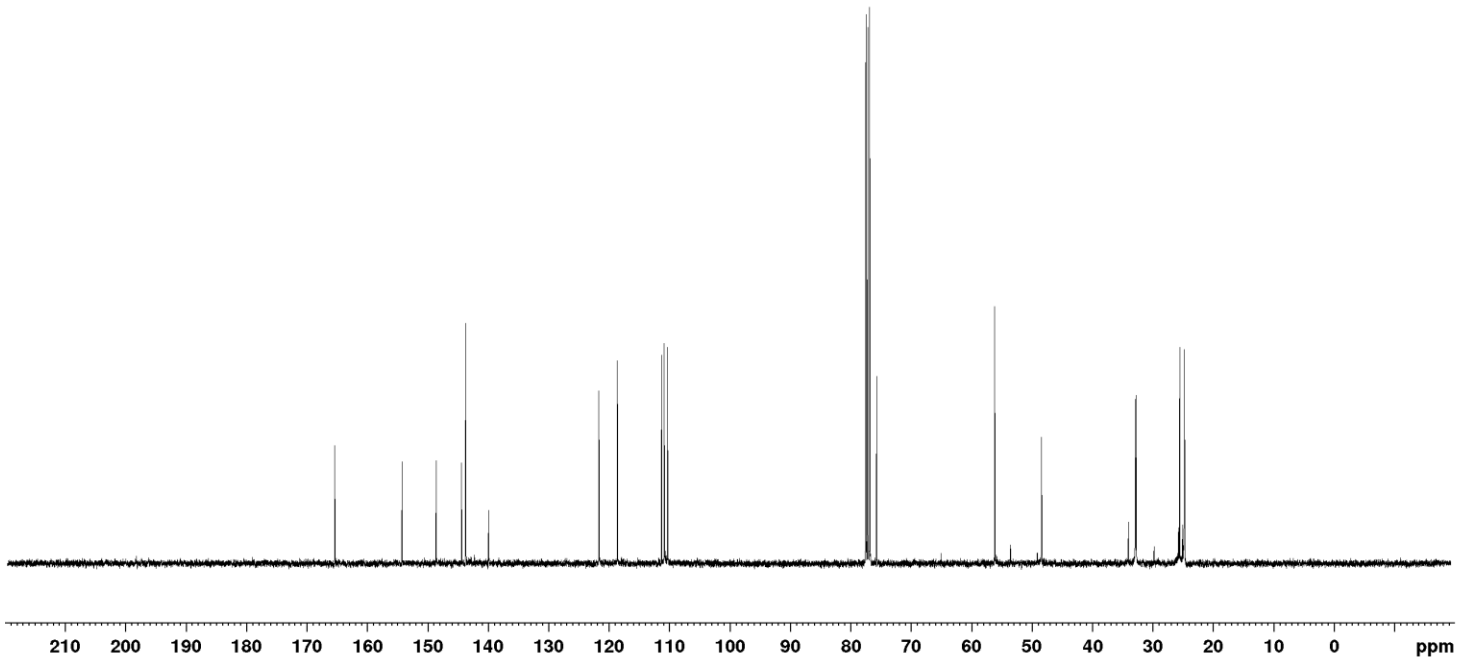

```
Current Data Parameters
NAME      C.S. 2-104-46 f54-6
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20151221
Time              11.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                32.72
DW               62.400 usec
DE                6.50 usec
TE              293.5 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1               1H
P1               15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI               65536
SF           400.1500000 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```



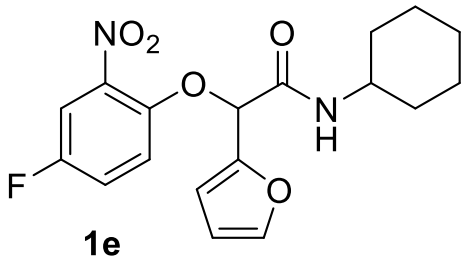


1d

^{13}C NMR, $CDCl_3$
100 MHz



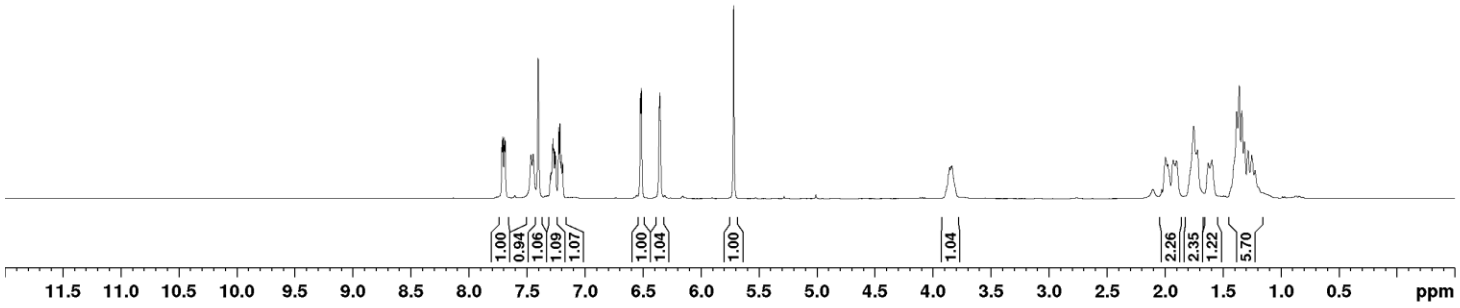

```
Current Data Parameters
NAME      C.S. 2-104-46 f54-6
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160128
Time              14.11
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  288
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG               210.96
DW               20.800 usec
DE               10.00 usec
TE               294.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1               13C
P1               10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                32768
SF           100.6177855 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

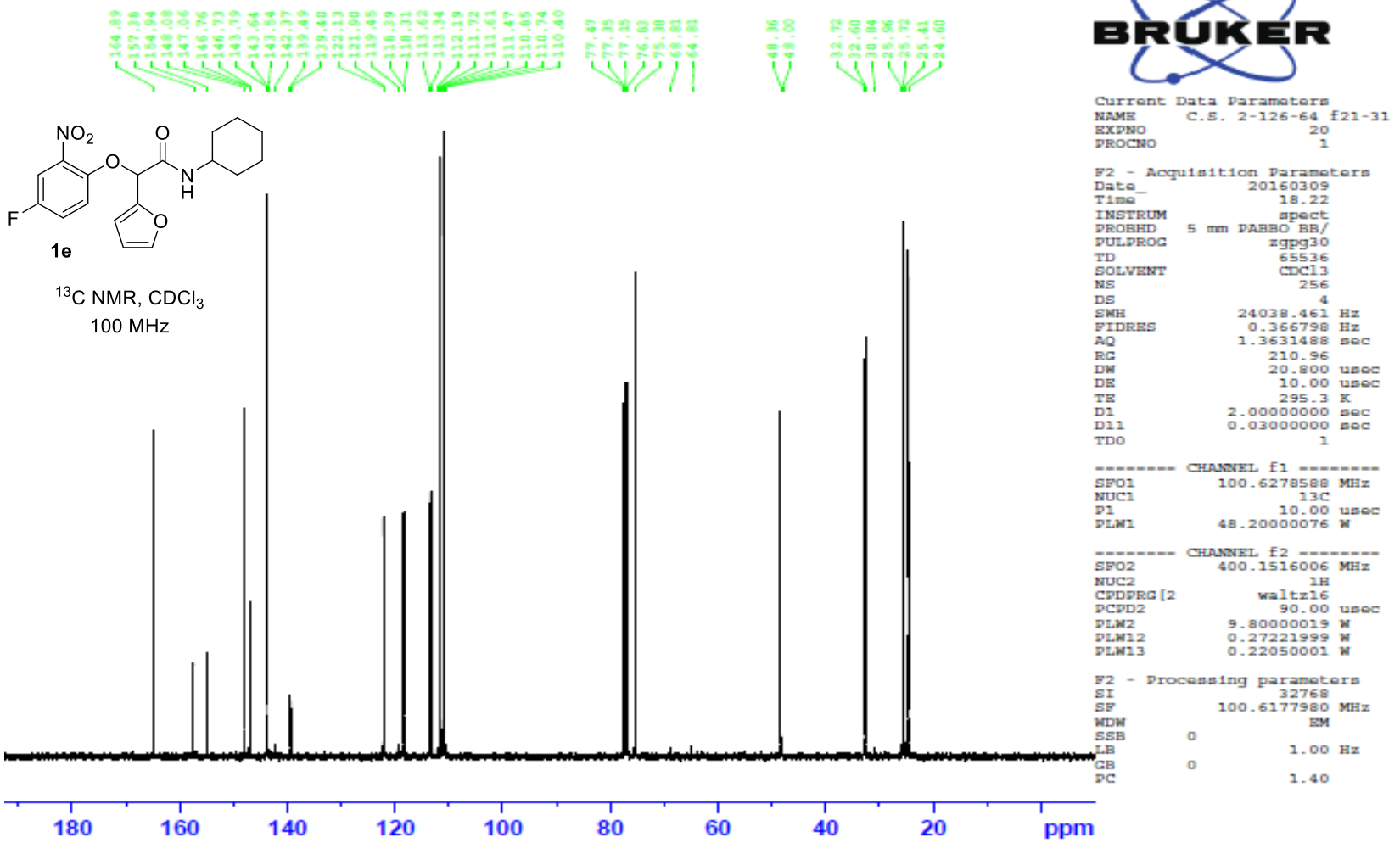


1e

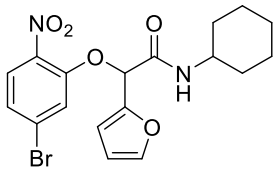
^{1}H NMR, $CDCl_3$
400 MHz


```
Current Data Parameters
NAME      C.S. 2-126-64 f21-3
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160309
Time              17.49
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               32.72
DW               62.400 usec
DE                6.50 usec
TE              293.9 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1               1H
P1               15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI               65536
SF          400.1500000 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```





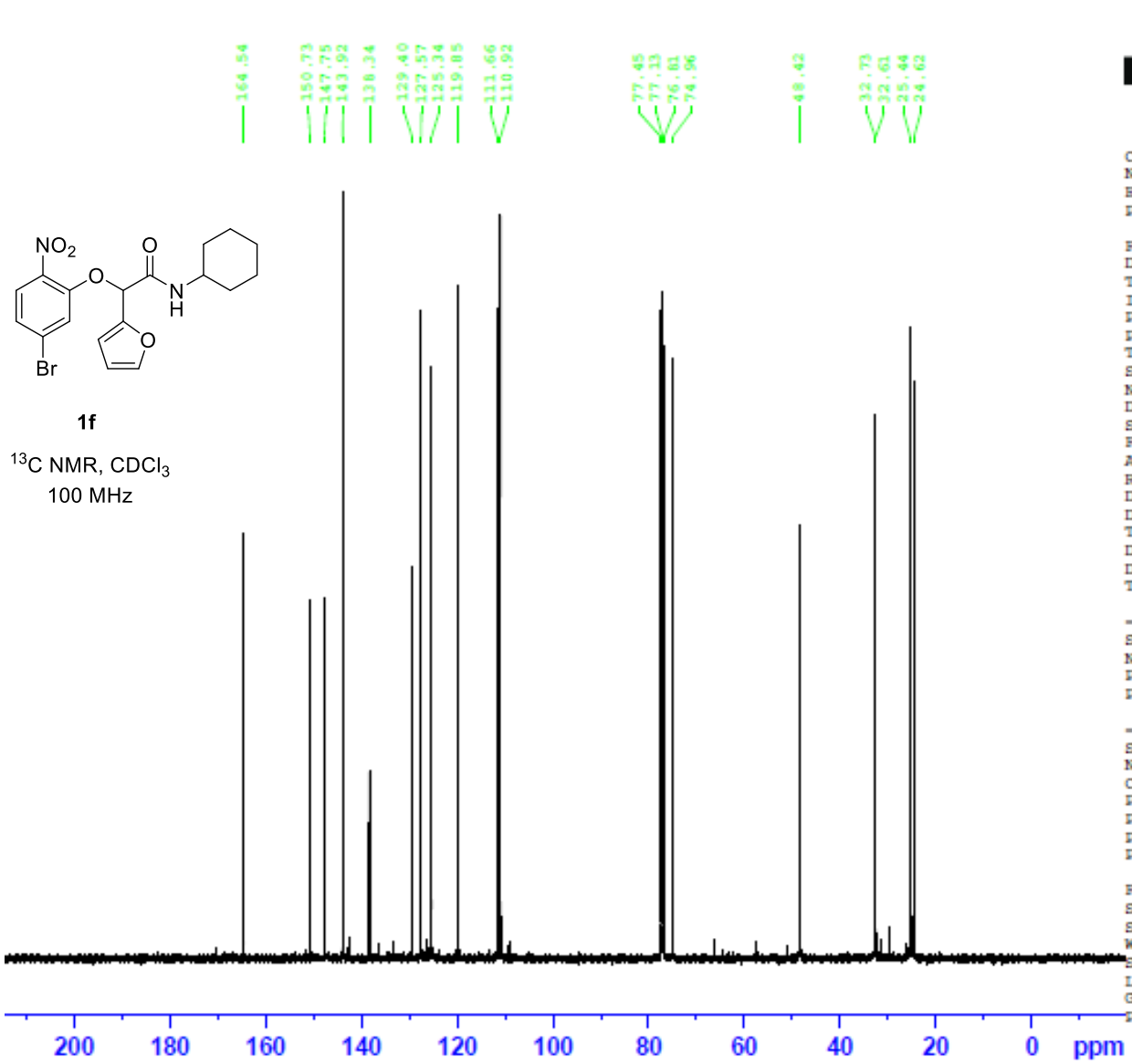

```
Current Data Parameters
NAME      C.S. 2-126-64 f21-31
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_            20160309
Time                18.22
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                 210.96
DW                 20.800 usec
DE                 10.00 usec
TE                 295.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6278588 MHz
NUC1                 13C
P1                 10.00 usec
PLW1          48.20000076 W

======== CHANNEL f2 ========
SFO2          400.1516006 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           9.80000019 W
PLW12          0.27221999 W
PLW13          0.22050001 W

F2 - Processing parameters
SI                   32768
SF            100.6177980 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



1f

^{1}H NMR, $CDCl_3$
400 MHz

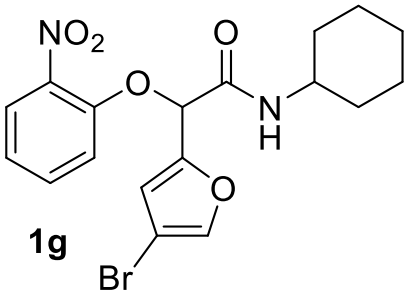

```
Current Data Parameters
NAME        CP 1-24 Fr 13
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190308
Time               17.29
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                32.72
DW               62.400 usec
DE                6.50 usec
TE               293.9 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1               1H
P1                15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI                65536
SF           400.1500059 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



1f

^{13}C NMR, $CDCl_3$
100 MHz

```
Current Data Parameters
NAME          CP-1-24-13C
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20211031
Time               23.04
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                298.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                 32768
SF           100.6177980 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

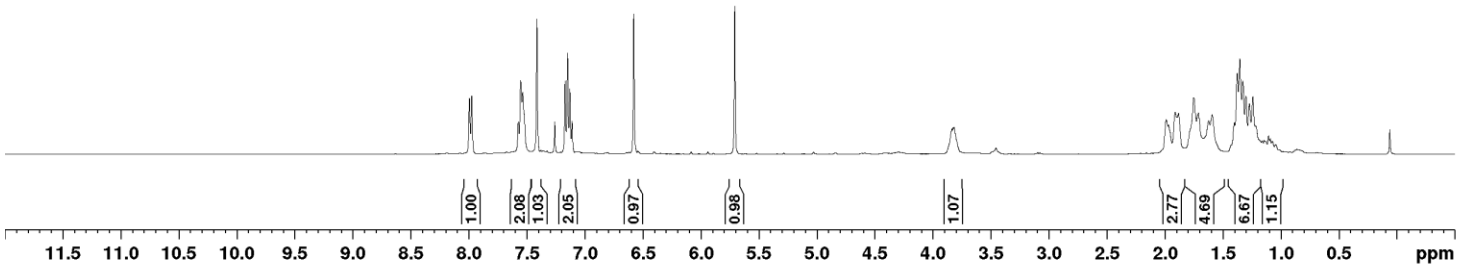


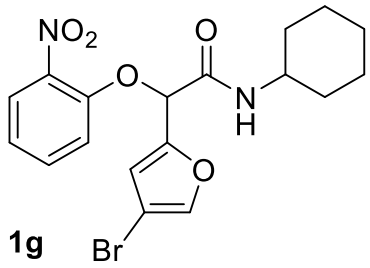
1g

^{1}H NMR, $CDCl_3$

400 MHz

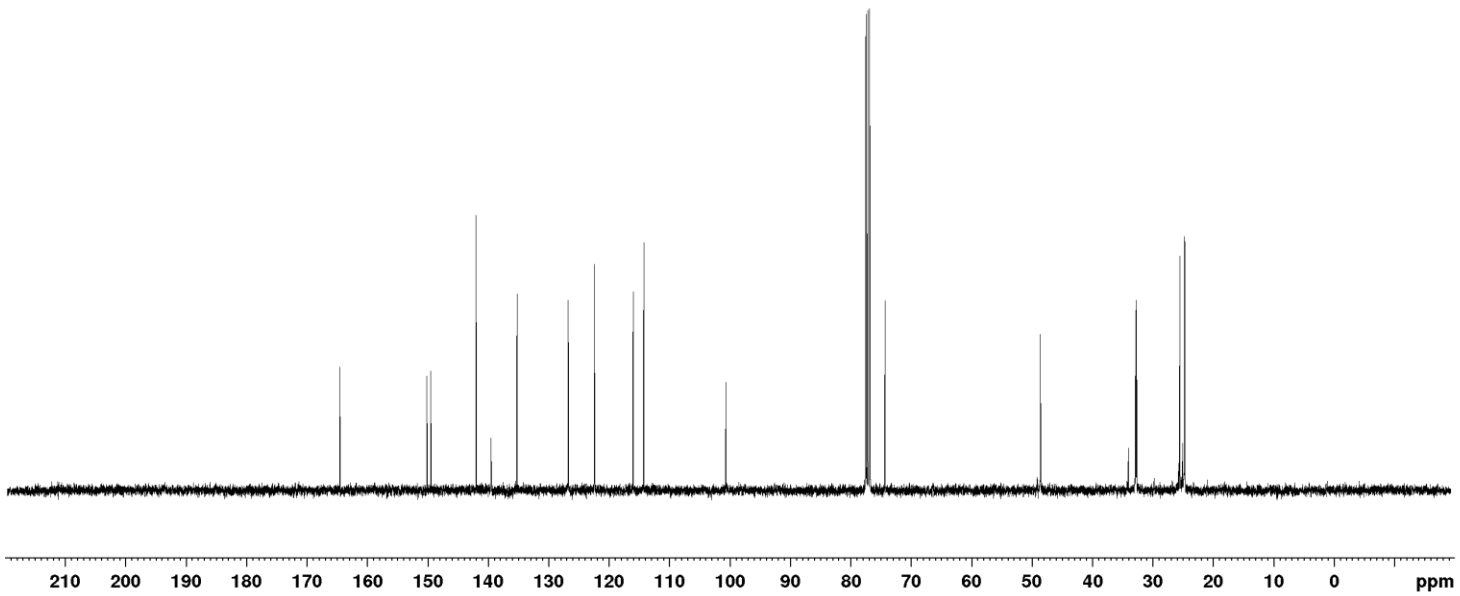
```
Current Data Parameters
NAME      C.S. 2-113-52 f4-9
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160216
Time               7.35
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                66.61
DW                62.400 usec
DE                 6.50 usec
TE               293.8 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1                1H
P1                15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI                65536
SF           400.1500104 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



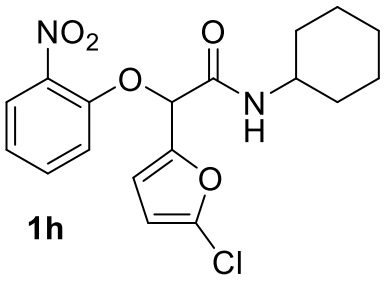


1g

^{13}C NMR, $CDCl_3$
100 MHz



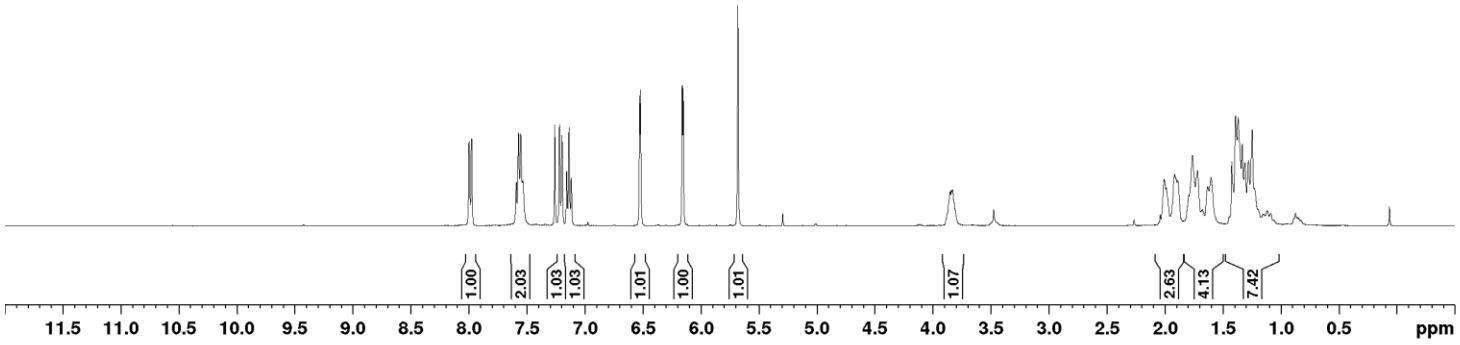

```
Current Data Parameters
NAME      C.S. 2-113-52 f4-9
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160216
Time               7.44
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                294.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                32768
SF           100.6177854 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

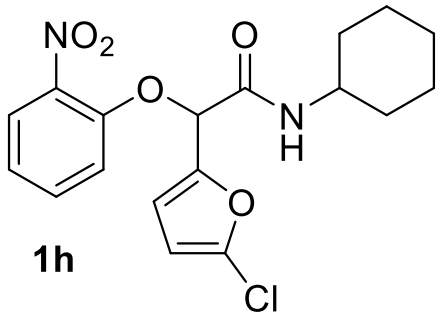


1h

^{1}H NMR, $CDCl_3$
400 MHz

```
Current Data Parameters
NAME       C.S. 2-114r_c
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160206
Time               11.52
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                120.42
DW                62.400 usec
DE                 6.50 usec
TE                293.6 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                  1H
P1                15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1500100 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

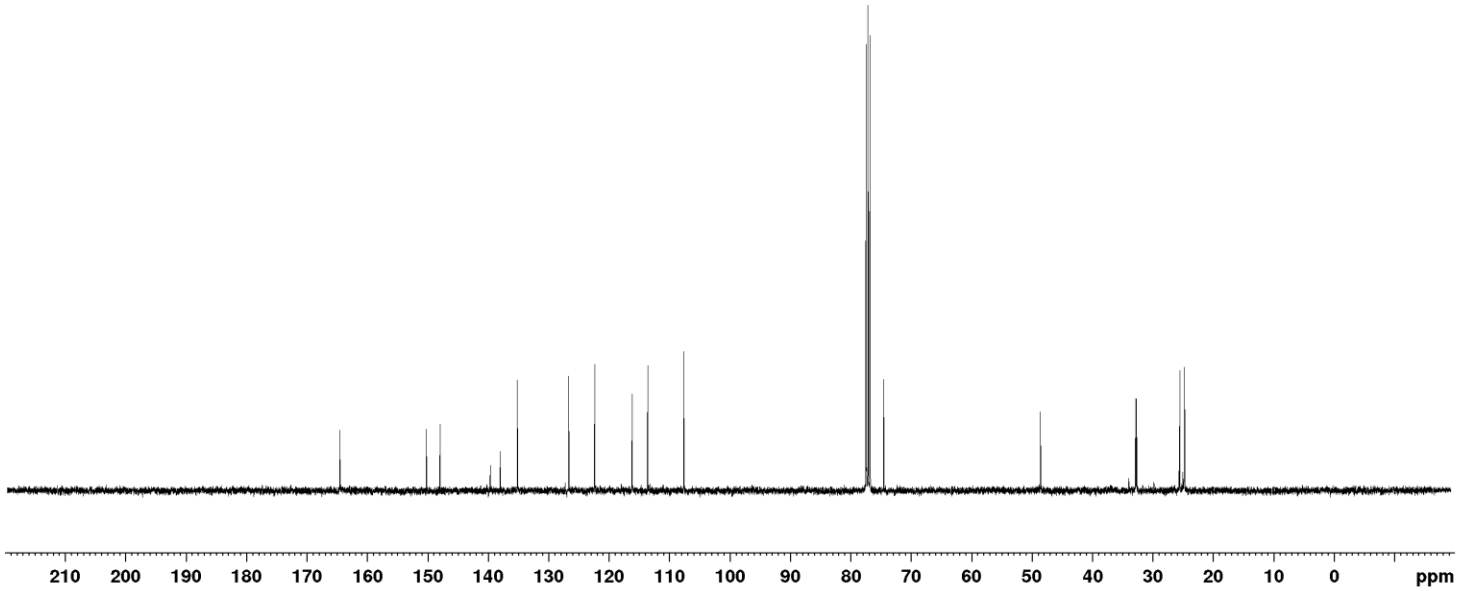




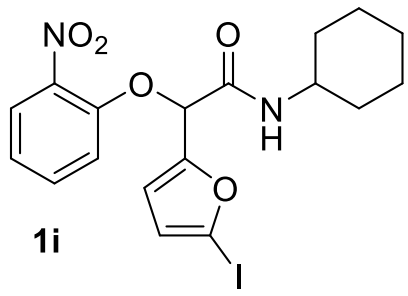
1h

^{13}C NMR, $CDCl_3$

100 MHz



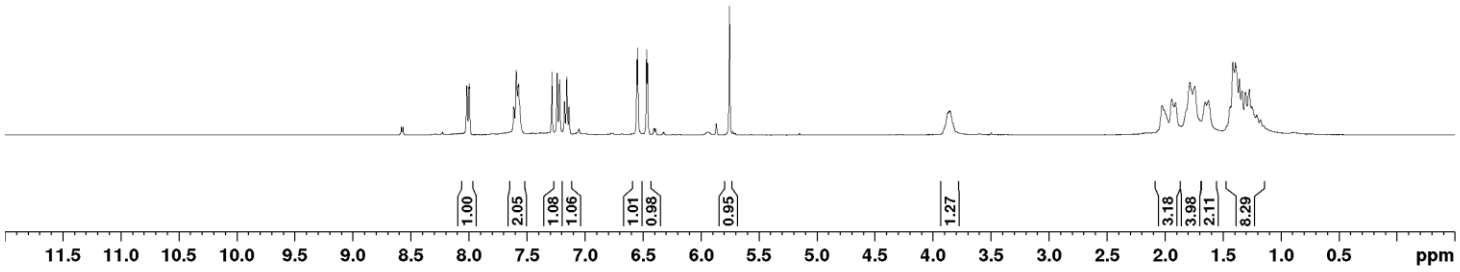

```
Current Data Parameters
NAME       C.S. 2-114r_c
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160208
Time                2.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW               20.800 usec
DE               10.00 usec
TE                294.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                 32768
SF           100.6177821 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

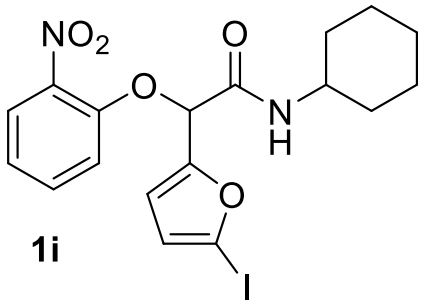


1i

^{1}H NMR, $CDCl_3$
400 MHz


```
Current Data Parameters
NAME      C.S. 2-118-54 f40-4
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160208
Time                1.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 57.34
DW                62.400 usec
DE                 6.50 usec
TE                293.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                  1H
P1                 15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1500000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

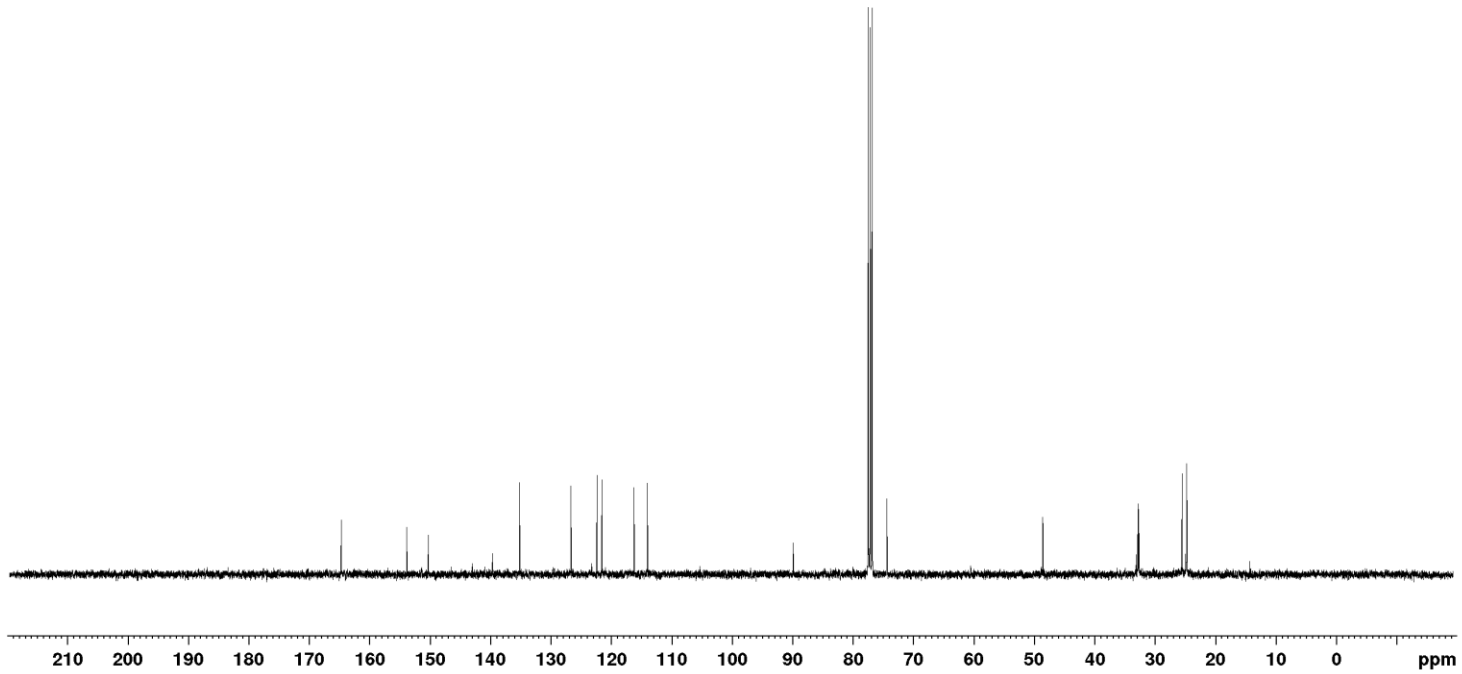




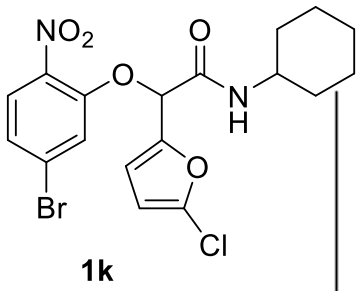
1i

^{13}C NMR, $CDCl_3$

100 MHz




```
Current Data Parameters
NAME      C.S. 2-118-54 f40-4
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160216
Time                8.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                295.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                32768
SF           100.6177836 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

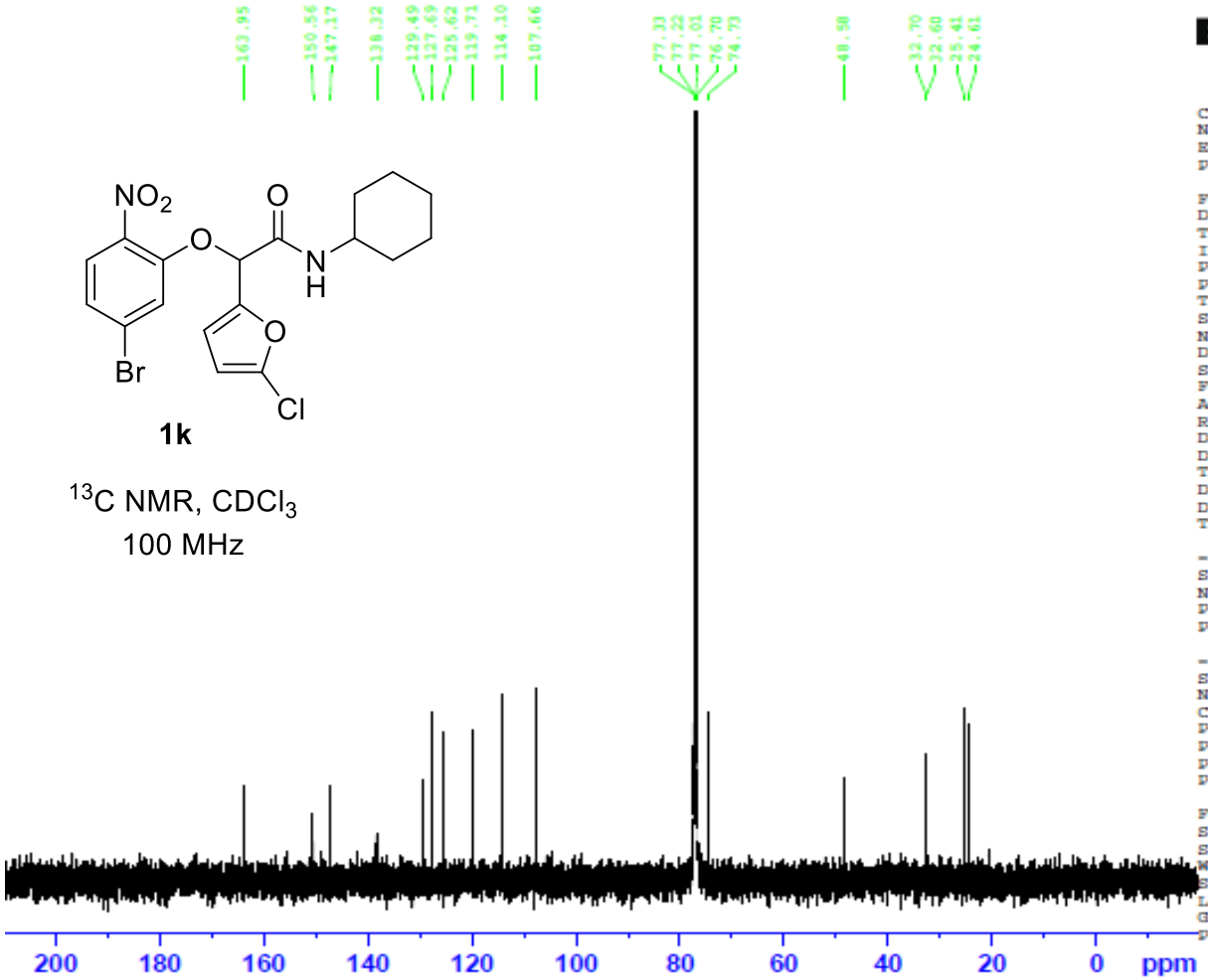


1k

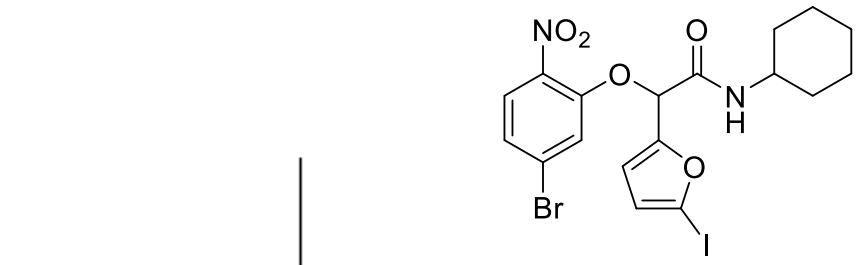
^{1}H NMR, $CDCl_3$
400 MHz

BRUKER

```
Current Data Parameters
NAME      CP-1-8-prep3-re
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211031
Time              16.14
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               152.01
DW               62.400 usec
DE                6.50 usec
TE               298.1 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1               1H
P1               15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI               65536
SF          400.1500069 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```




```
Current Data Parameters
NAME      CP-1-8-prep3-re
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211031
Time              18.29
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG               210.96
DW                20.800 usec
DE                10.00 usec
TE                298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6278588 MHz
NUC1               13C
P1                10.00 usec
PLW1        48.20000076 W

======== CHANNEL f2 ========
SFO2        400.1516006 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2           9.80000019 W
PLW12          0.27221999 W
PLW13          0.22050001 W

F2 - Processing parameters
SI                32768
SF          100.6177980 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

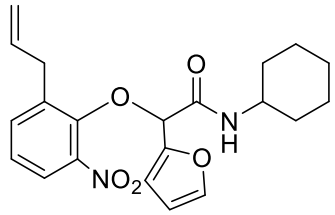


1l (+2e), inseparable

^{1}H NMR, $CDCl_3$

400 MHz

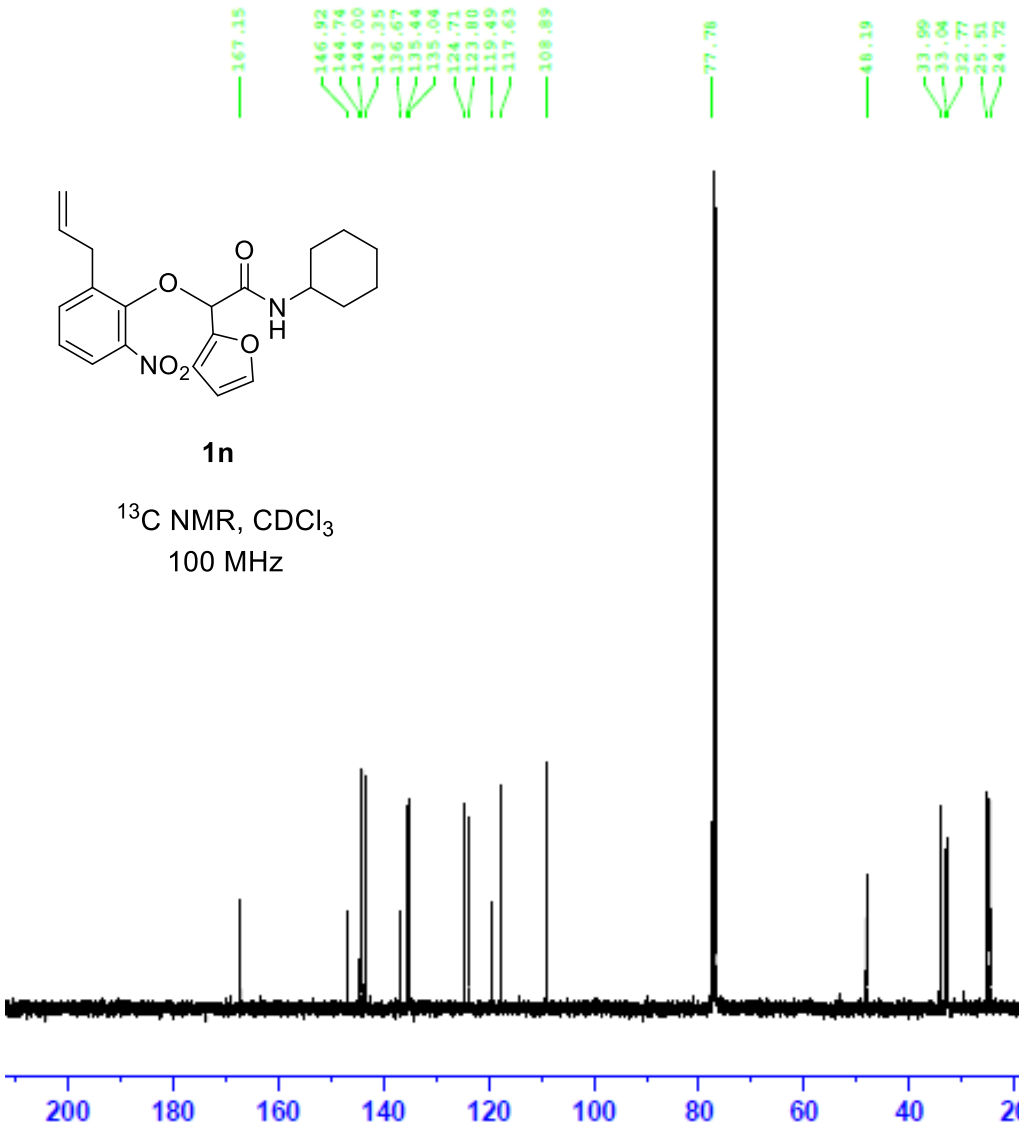

```
Current Data Parameters
NAME      CP 1-9 Fr 35-50
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180321
Time                13.35
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  105.13
DW                  62.400 usec
DE                   6.50 usec
TE                  293.5 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                   1H
P1                  15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                  65536
SF            400.1500128 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



1n

^{1}H NMR, $CDCl_3$
400 MHz

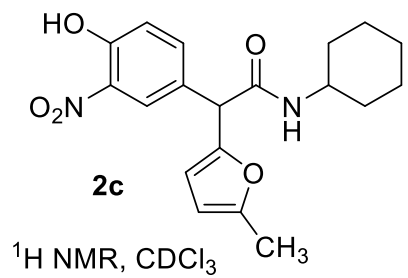

```
Current Data Parameters
NAME      C.S. 2-52-7 f11-16
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141111
Time                 12.34
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  105.13
DW                62.400 usec
DE                 6.50 usec
TE                295.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                   1H
P1                  15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                  65536
SF            400.1500000 MHz
WDW                    EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



1n

^{13}C NMR, $CDCl_3$
100 MHz

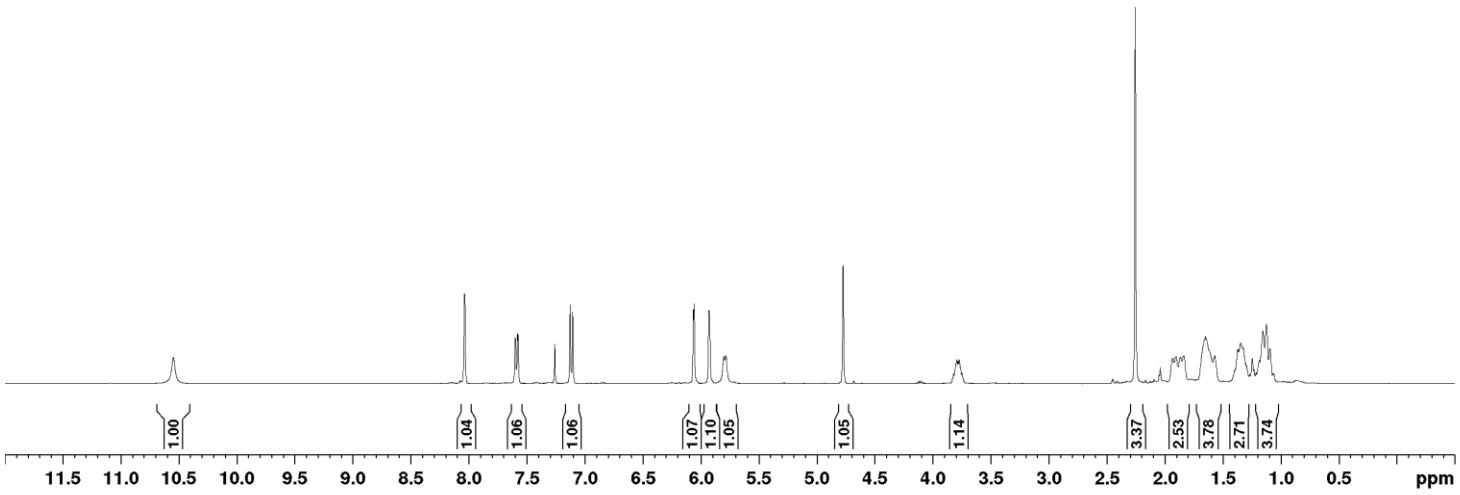
```
Current Data Parameters
NAME       CS-2-92-39-Pr10-18-PS1-13C
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211031
Time              19.48
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW               20.800 usec
DE               10.00 usec
TE               298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1               13C
P1               10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                32768
SF           100.6177980 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

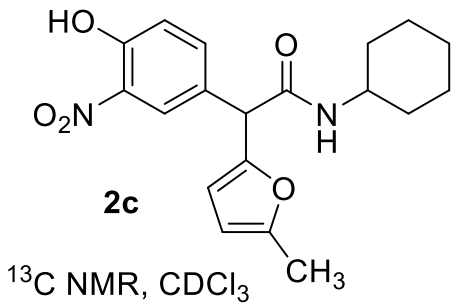


2c

^{1}H NMR, $CDCl_3$
400 MHz

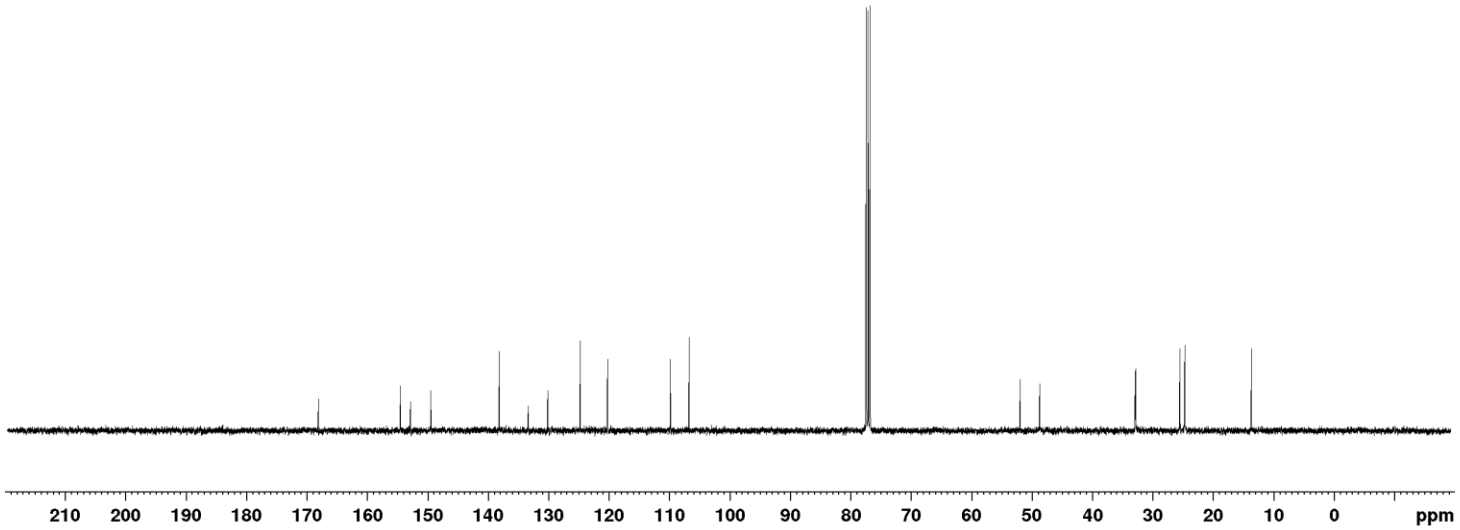
```
Current Data Parameters
NAME      C.S. 2-102-45 f19-2
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151206
Time              18.11
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                82.91
DW              62.400 usec
DE               6.50 usec
TE              292.9 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1                1H
P1               15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI               65536
SF           400.1500104 MHz
WDW                 EM
SSB      0
LB               0.30 Hz
GB       0
PC               1.00
```



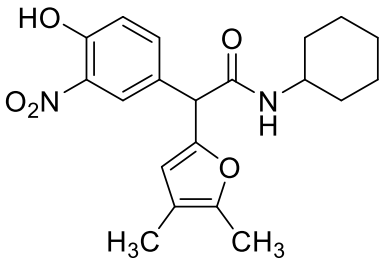


2c

^{13}C NMR, $CDCl_3$
100 MHz




```
Current Data Parameters
NAME      C.S. 2-102-45 f19-2
EXPNO                    20
PROCNO                    1

F2 - Acquisition Parameters
Date_            20160128
Time                13.50
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                 32768
SF           100.6177816 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

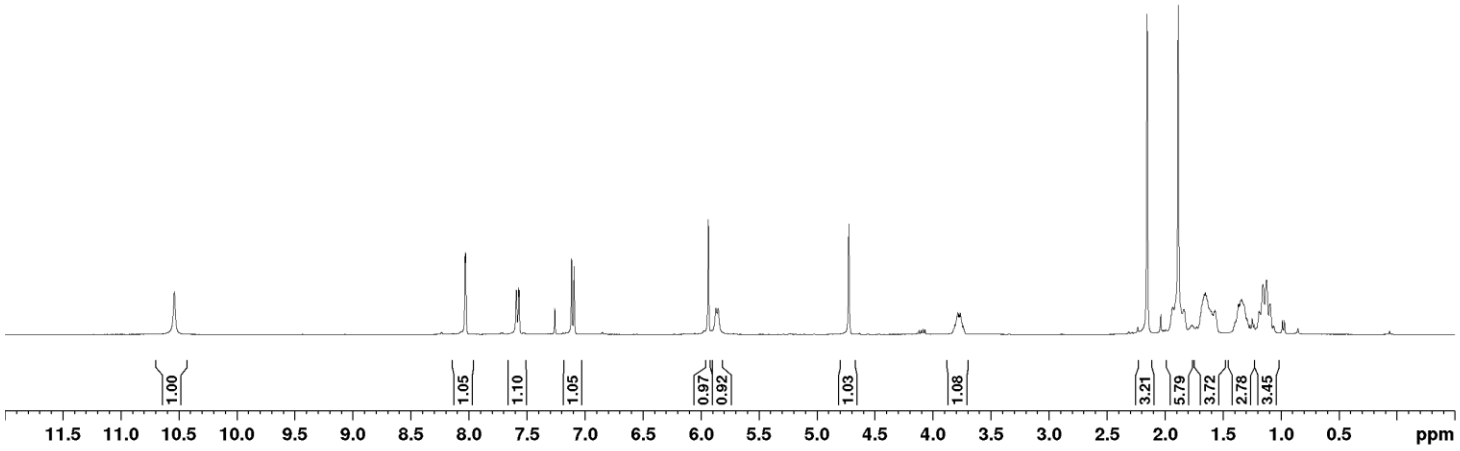


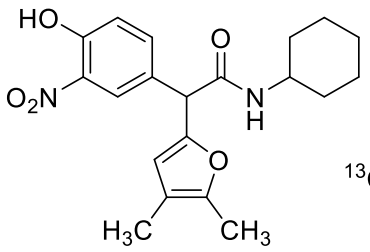
2d

^{1}H NMR, $CDCl_3$

400 MHz

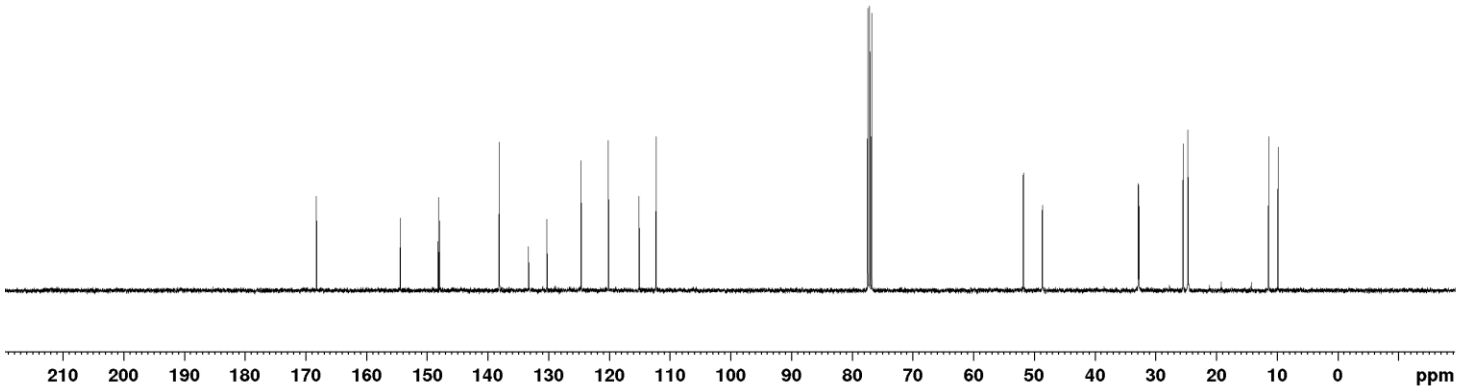

```
Current Data Parameters
NAME      C.S. 2-120-57 f14
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160218
Time                7.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 66.61
DW                62.400 usec
DE                 6.50 usec
TE                293.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                  1H
P1                15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                 65536
SF            400.1500105 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



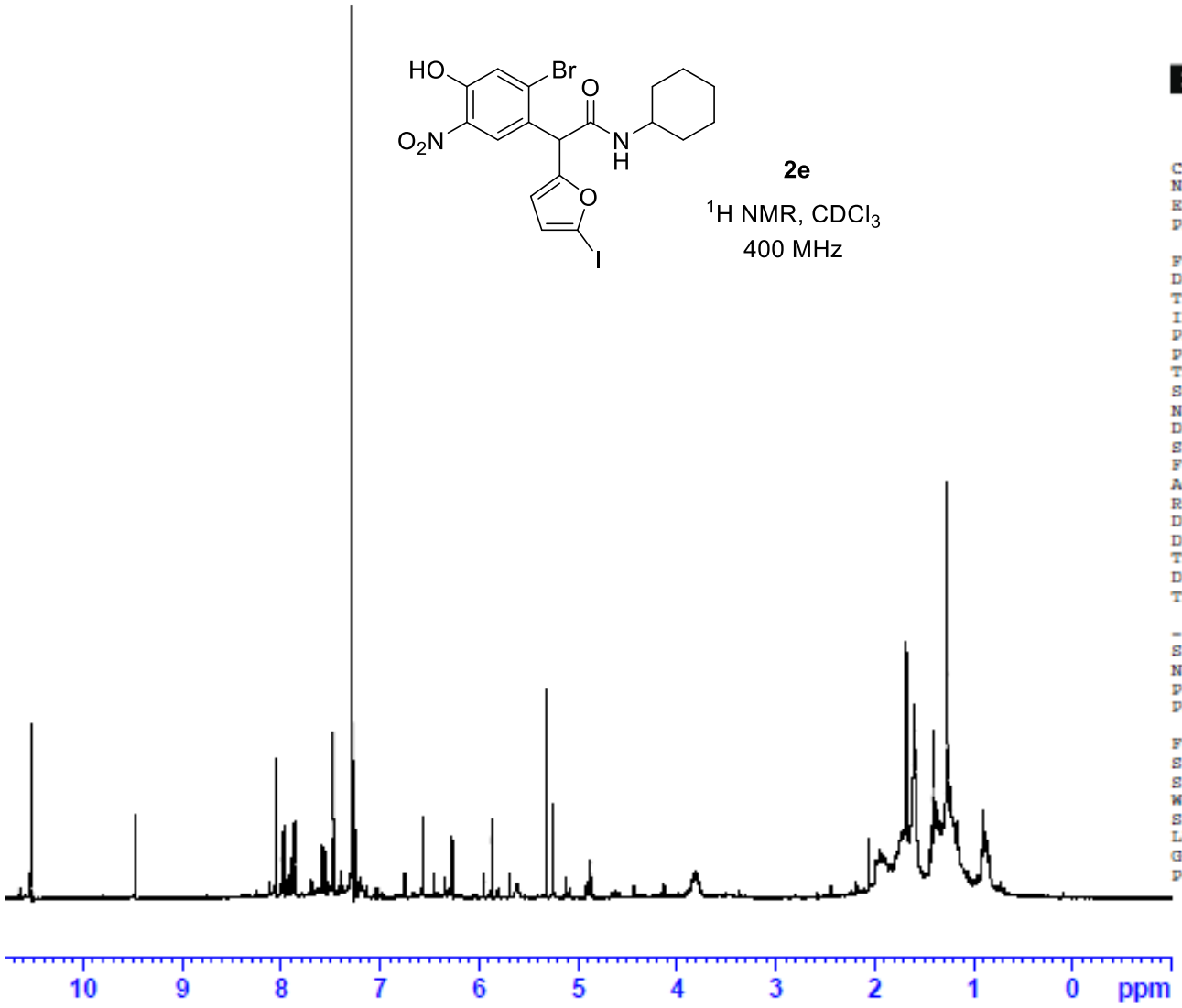


2d

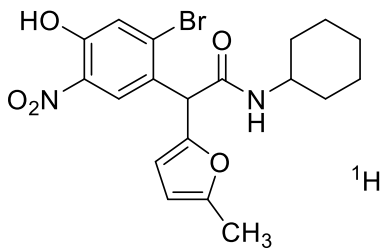
^{13}C NMR, $CDCl_3$
100 MHz




```
Current Data Parameters
NAME      C.S. 2-120-57 f14
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160218
Time              10.53
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                294.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                32768
SF           100.6177857 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```




```
Current Data Parameters
NAME      CP 1-9 Fr 31-34
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180321
Time                13.27
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  152.01
DW                62.400 usec
DE                 6.50 usec
TE                293.4 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                   1H
P1                  15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                  65536
SF            400.1500035 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

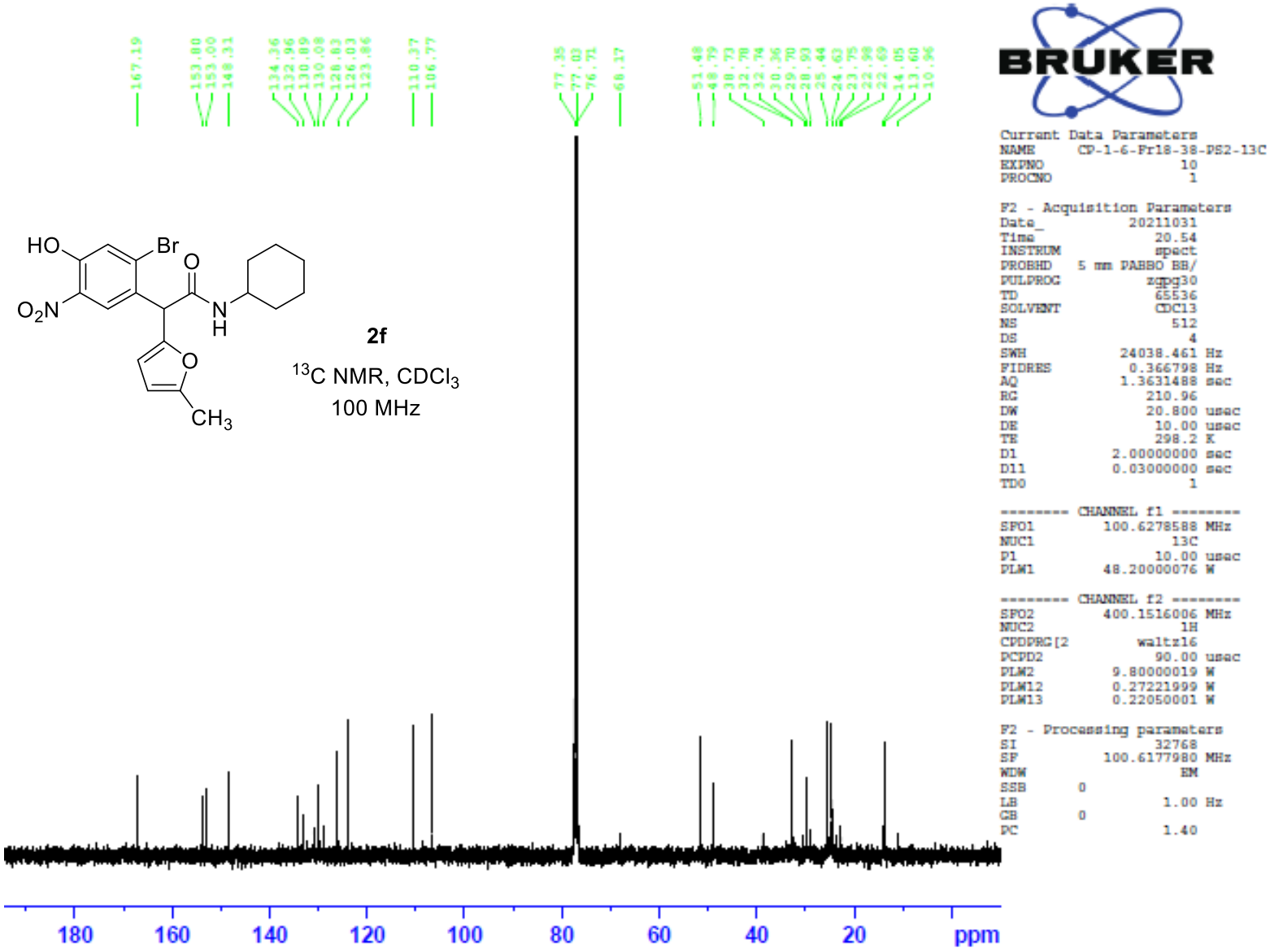


2f

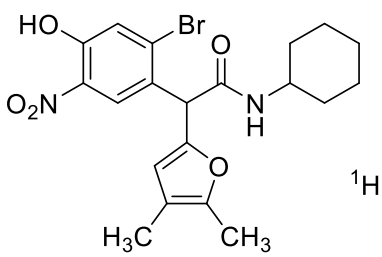
^{1}H NMR, $CDCl_3$
400 MHz

BRUKER

```
Current Data Parameters
NAME      CP 1-6 FR 18-38
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180111
Time                16.17
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  120.42
DW                 62.400 usec
DE                  6.50 usec
TE                 294.8 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                   1H
P1                  15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                  65536
SF            400.1500051 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



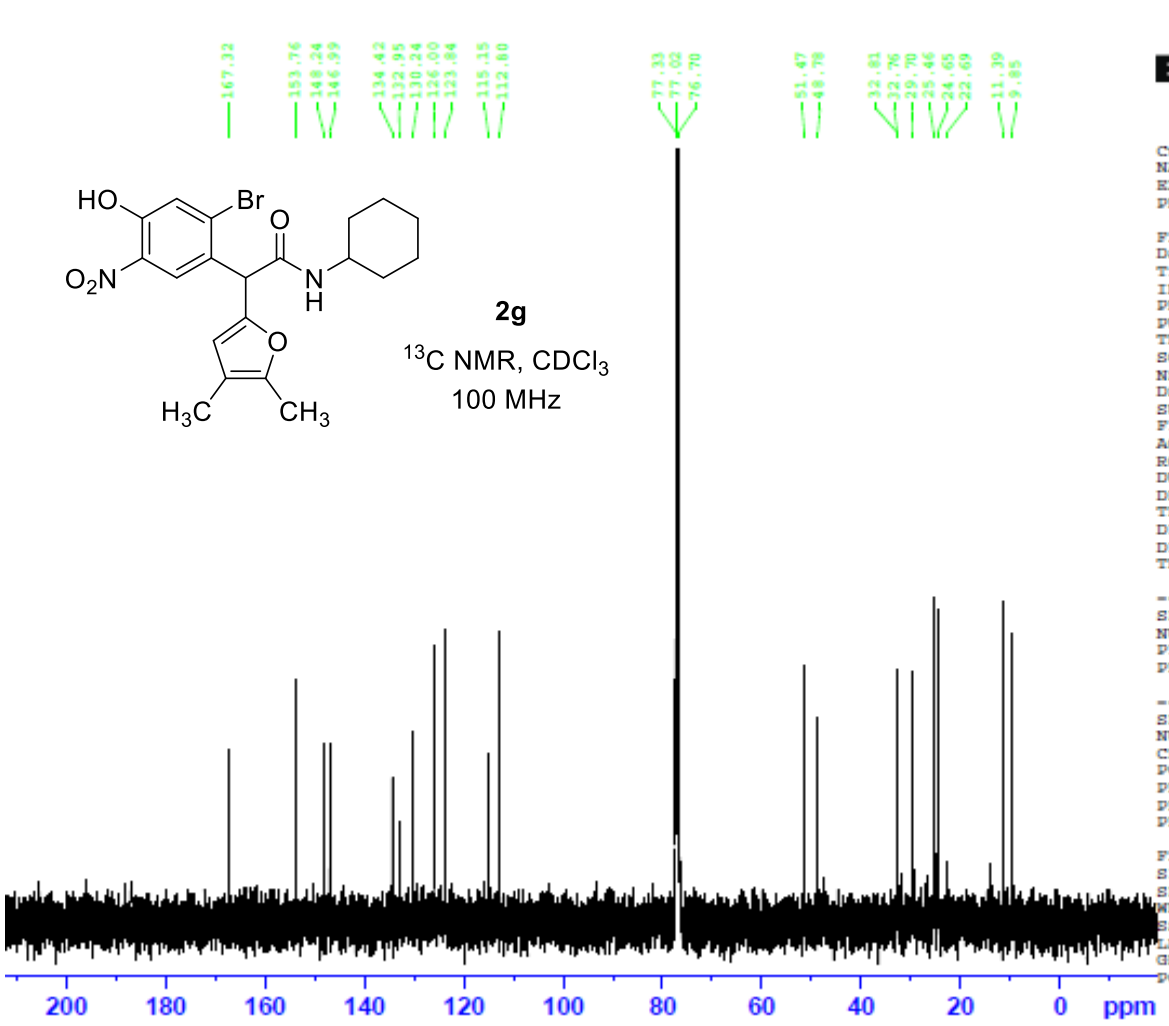

```
Current Data Parameters
NAME      CP-1-6-Fr18-38-PS2-13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211031
Time              20.54
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                210.96
DW                20.800 usec
DE                10.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                 13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          9.80000019 W
PLW12         0.27221999 W
PLW13         0.22050001 W

F2 - Processing parameters
SI                  32768
SF           100.6177980 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



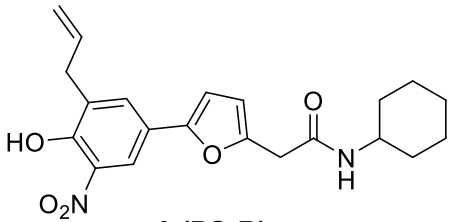
2g

^{1}H NMR, $CDCl_3$
400 MHz


```
Current Data Parameters
NAME      CP-1-7-Fr21-27-re
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20211031
Time               16.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 134.4
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1                1H
P1                15.00 usec
PLW1          9.80000019 W

F2 - Processing parameters
SI                 65536
SF           400.1500100 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



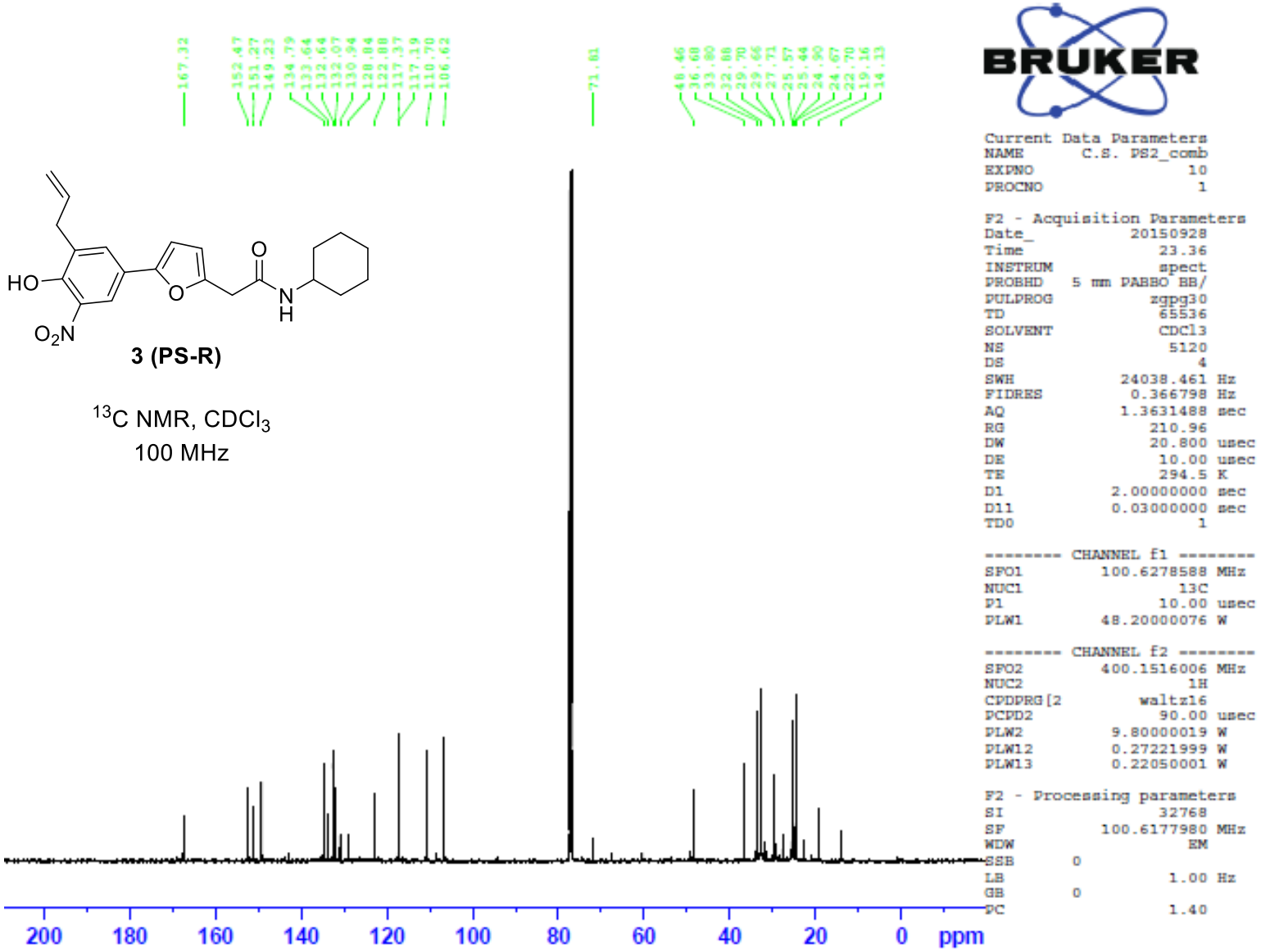

```
Current Data Parameters
NAME      CP-1-7-Fr21-27-re
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20211031
Time              17.27
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG               210.96
DW                20.800 usec
DE                10.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1               13C
P1                10.00 usec
PLW1         48.20000076 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2               1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2           9.80000019 W
PLW12          0.27221999 W
PLW13          0.22050001 W

F2 - Processing parameters
SI                32768
SF           100.6177980 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



3 (PS-R)

^{1}H NMR, $CDCl_3$
400 MHz


```
Current Data Parameters
NAME      C.S. 2-48-5 f40-48
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150506
Time                  9.40
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                   152.01
DW                   62.400 usec
DE                    6.50 usec
TE                   292.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1524711 MHz
NUC1                    1H
P1                   15.00 usec
PLW1           9.80000019 W

F2 - Processing parameters
SI                   65536
SF             400.1500130 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```


167.32
152.47
151.27
149.23
134.79
133.64
132.64
132.07
130.94
120.84
122.80
117.37
117.19
110.70
106.62
71.61
48.46
36.68
33.80
32.88
29.70
29.66
27.71
25.57
25.44
24.90
24.67
22.70
19.16
14.13
HO
O
O
N
H
O_2N
3 (PS-R)
^{13}C NMR, $CDCl_3$
100 MHz
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME C.S. PS2_comb
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150928
Time 23.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5120
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 210.96
DW 20.800 usec
DE 10.00 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
-------- CHANNEL f1 --------
SFO1 100.6278588 MHz
NUC1 13C
P1 10.00 usec
PLW1 48.20000076 W
-------- CHANNEL f2 --------
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 9.80000019 W
PLW12 0.27221999 W
PLW13 0.22050001 W
F2 - Processing parameters
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40