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Electronic Supplementary Information (ESI) for

A visible-light mediated three-component radical process using dithiocarbamate anion catalysis

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A. General Information

The NMR spectra were recorded at 400 MHz and 500 MHz for ^{1}H and 100 or 125 MHz for ^{13}C. The chemical shift (δ) for ^{1}H and ^{13}C are given in ppm relative to residual signals of the solvents ($CHCl_3$ @ 7.26 ppm ^{1}H NMR and 77.16 ppm ^{13}C NMR, and tetramethylsilane @ 0 ppm). Coupling constants are given in Hertz. The following abbreviations are used to indicate the multiplicity: s, singlet; d, doublet; q, quartet; m, multiplet; bs, broad signal; app, apparent.
Infrared (IR) spectra were obtained using a Bruker Alpha FT-IR spectrometer.
High resolution mass spectra (HRMS) were obtained from the ICIQ HRMS unit on MicroTOF Focus and Maxis Impact (Bruker Daltonics) with electrospray ionization. (ESI). X-ray data were obtained from the ICIQ X-Ray unit using a Brucker-Nonius diffractometer equipped with an APPEX 2 4K CCD area detector.
Isolated yields refer to materials of >95% purity as determined by ^{1}H NMR.

The authors are indebted to the team of the Research Support Area at ICIQ, particularly to the NMR, the X-ray Diffraction, and the High Resolution Mass Spectrometry Units.

Grace Fox is thanked for proofreading the manuscript.

General Procedures. All reactions were set up under an argon atmosphere in oven-dried glassware using standard Schlenk techniques, unless otherwise stated. Synthesis grade solvents were used as purchased; anhydrous solvents were taken from a commercial SPS solvent dispenser. Chromatographic purification of products was accomplished using forced-flow chromatography (FC) on silica gel (35-70 mesh). For thin layer chromatography (TLC) analysis throughout this work, Merck pre-coated TLC plates (silica gel 60 GF_{254}, 0.25 mm) were employed, using UV light as the visualizing agent and an acidic mixture of vanillin or basic aqueous potassium permanganate ($KMnO_4$) stain solutions, and heat as developing agents. Organic solutions were concentrated under reduced pressure on a Büchi rotatory evaporator.

Materials. Most of the starting materials used in this study are commercially available and were purchased in the highest purity available from Sigma-Aldrich, Fluka, Alfa Aesar, Fluorochem, and used as received, without further purifications. The synthesis of the DTC catalyst **A** is described in our previous work.[1]

B. Experimental Procedure

B.1. Set-up: Temperature-controlled photoreactor and specifications of the light source

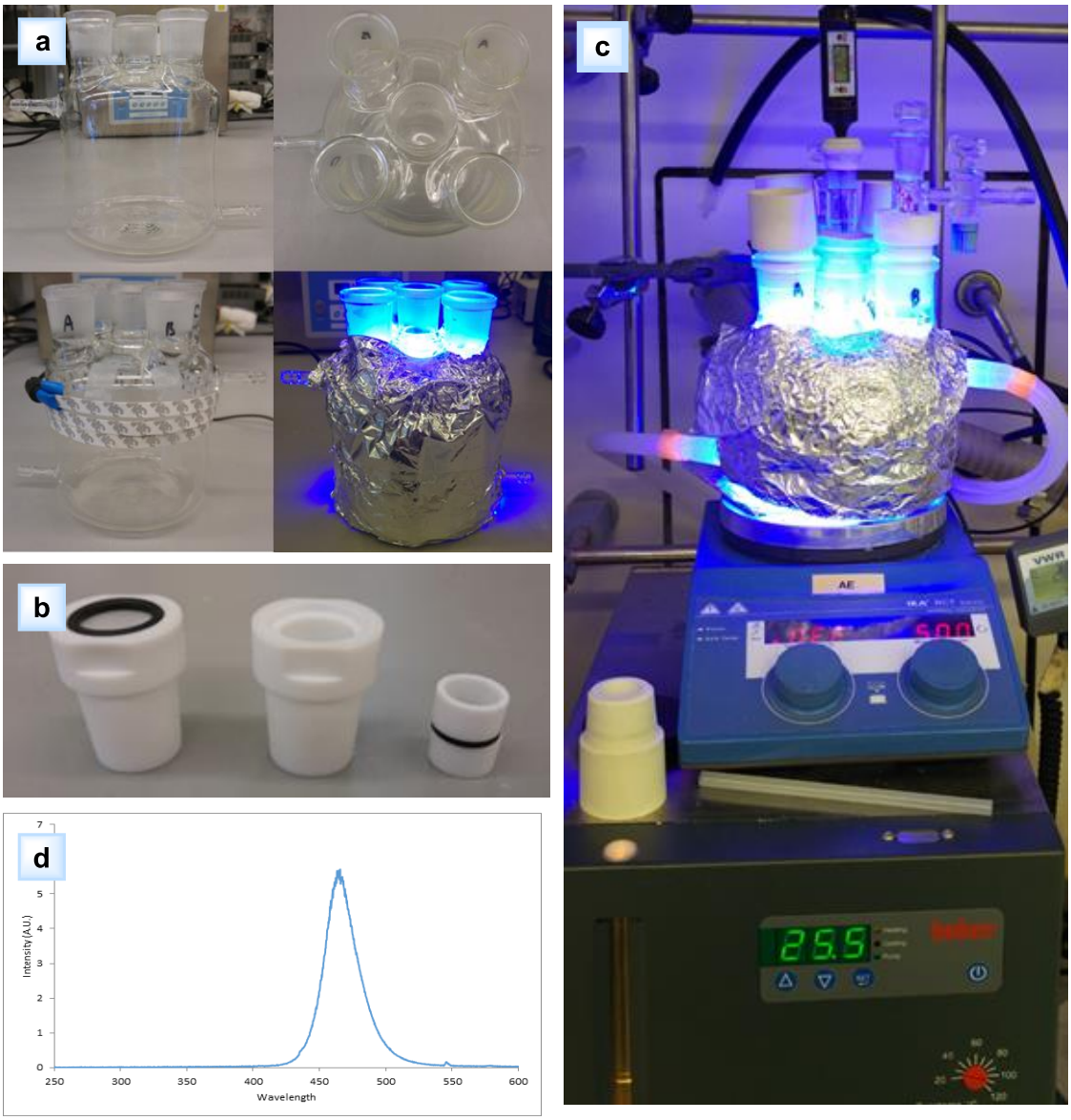
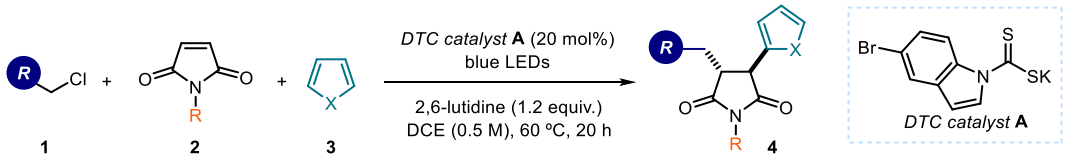


Figure S1. (a) Photoreactor used in this study. (b) Teflon adaptors used to accommodate Schlenk tubes in the photoreactor. (c) Fully assembled photoreactor in operation. (d) Emission spectrum of the 465 nm *blue LED strip* used in this study.

The photoreactor consisted of a 12.5 cm diameter jar, fitted with 4 standard 29 sized ground glass joints arranged in a square and a central 29 sized joint. A commercial 1 meter LED strip was wrapped around the jar, followed by a layer of aluminium foil and cotton for insulation (Figure S1, a). Each of the joints could be used to fit a standard 16 mm or 25 mm diameter Schlenk tube with a Teflon adaptor (Figure S1, b). An inlet and an outlet allow the circulation of liquid from a Huber Minichiller 300 inside the jar. This setup allows to perform reactions at temperatures ranging from -20 ºC to 80 ºC with accurate control of the reaction temperature (± 1ºC) (Figure S1, c). To maintain a consistent illumination during different experiments, only the four external positions were used to perform reactions while the central one was used to monitor the temperature inside a Schlenk tube identical to those used to perform reactions.

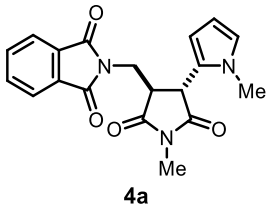
The light source used in this study consisted of a 1 m strip, 14.4W 'LEDXON MODULAR 9009083 LED, SINGLE 5050' purchased from Farnell, catalog number 9009083. The emission spectrum of these LEDs was recorded (Figure S1, d).

B.2. General Procedure for the Three-Component Radical Process



In an oven dried Schlenk tube, the DTC catalyst **A** (31.0 mg, 0.1 mmol, 0.2 equiv.) was dissolved in dichloroethane (0.5 mL), then the alkyl chloride **1** (0.75 mmol, 1.5 equiv.) was added while stirring, followed by 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), maleimide **2** (0.5 mmol, 1.0 equiv.), and pyrrole **3** (5.0 mmol, 10.0 equiv.). An additional volume of dichloroethane (0.5 mL) was added to the reaction vessel, washing the sides from residual solids. The resulting mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup (see section B1, Figure S1), maintained at a temperature of 60 ºC (60-61ºC measured in the central well), and the reaction was stirred for 20 hours under continuous irradiation. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography to afford the corresponding product **4** in the stated yield with >95% purity according to ^{1}H NMR analysis. The exact conditions for chromatography are reported for each compound.

B.3. Characterization of Products



4a

2-((1-methyl-4-(1-methyl-1*H*-pyrrol-2-yl)-2,5-dioxopyrrolidin-3-yl)methyl)isoindoline-1,3-dione (4a): Synthesized according to the general procedure using *N*-(chloromethyl)phthalimide **1a** (147 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4a** was purified by column chromatography (gradient from 20% to 35% AcOEt in hexanes as eluent): 100 mg yellow solid, 57% yield. Crystallization from MeOH afforded crystals suitable for X-ray diffraction analysis (CCDC 1894404, see section E), which revealed a *trans* relative stereochemistry. Product **4a** can be synthesized up to a 5 mmol scale using the same photochemical set-up (see Section B.4. for details).

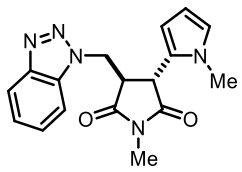
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.65 (dd, *J* = 5.4, 3.1 Hz, 2H), 6.36 (dd, *J* = 2.5, 2.0 Hz, 1H), 5.78 (dd, *J* = 3.6, 1.6 Hz, 1H), 5.72 (dd, *J* = 3.7, 2.8 Hz, 1H), 4.32 (dd, *J* = 14.0, 5.7 Hz, 1H), 4.07-4.02 (m, 2H), 3.62 (s, 3H), 3.61-3.58 (m, 1H), 2.99 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 175.7, 175.4, 168.5, 134.4, 131.9, 125.3, 124.0, 123.6, 107.3, 106.8, 45.2, 43.7, 38.1, 34.4, 25.5.

IR (thin film) ν 2950, 1773, 1694, 1433, 1395, 1379, 1361, 1304, 1271, 1089, 715 cm^{-1}.

HRMS: calculated for $C_{19}H_{17}N_3NaO_4^+$ ($M+Na^+$): 374.1111, found 374.1114.

TLC: 30:70 EtOAc/hexanes, R_f = 0.14.



4b

3-((1*H*-benzo[*d*][1,2,3]triazol-1-yl)methyl)-1-methyl-4-(1-methyl-1*H*-pyrrol-2-yl)pyrrolidine-2,5-dione (4b): Synthesized according to the general procedure using 1-(chloromethyl)-1*H*-benzotriazole **1b** (126 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4b** was purified by column chromatography (gradient from 20% to 35% AcOEt in hexanes as eluent): 91 mg, white foam, 56% yield.

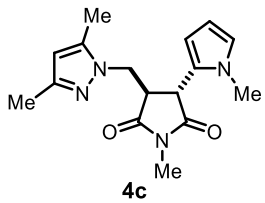
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02-8.00 (m, 1H), 7.62-7.60 (m, 1H), 7.52-7.48 (m, 1H), 7.38-7.35 (m, 1H), 6.60 (dd, *J* = 2.5, 1.9 Hz, 1H), 6.02 (dd, *J* = 3.6, 2.8 Hz, 1H), 5.91 (dd, *J* = 3.7, 1.6 Hz, 1H), 5.21 (dd, *J* = 14.7, 4.6 Hz, 1H), 4.97 (dd, *J* = 14.8, 4.3 Hz, 1H), 4.22 (d, *J* = 6.8 Hz, 1H), 3.65 (s, 3H), 3.64-3.60 (m, 1H), 2.85 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 175.9, 175.2, 146.1, 133.7, 128.4, 125.6, 124.8, 124.4, 120.4, 110.0, 107.6, 106.7, 48.0, 45.5, 41.4, 34.7, 25.6.

IR (thin film) ν 2924, 1698, 1493, 1434, 1383, 1289, 1274, 1162, 1126, 724 cm^{-1}.

HRMS: calculated for $C_{17}H_{17}N_5NaO_2^+$ ($M+Na^+$): 346.1274, found 346.1282.

TLC: 30:70 EtOAc/hexanes, R_f = 0.1.



4c

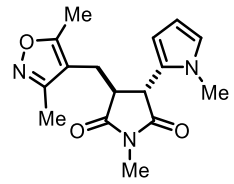
3-((3,5-dimethyl-4,5-dihydro-1*H*-pyrazol-1-yl)methyl)-1-methyl-4-(1-methyl-1*H*-pyrrol-2-yl)pyrrolidine-2,5-dione (4c): Synthesized by a two-step procedure. First, the alkyl chloride **1c** was synthesized via a variation of a reported procedure:[2] in a round bottom flask, (3,5-dimethyl-1H-pyrazol-1-yl)methanol (126 mg, 1 mmol, 1 equiv.) was dissolved in dry chloroform (5 mL) and cooled at 0 ºC. Thionyl chloride (88 µL, 1.2 mmol, 1.2 equiv.) was added dropwise and the reaction was left stirring at ambient temperature for 30 min. Solvent was removed under vacuum at 25 ºC, diethyl ether (5 mL) was added and dried (this was repeated twice) to obtain the crude 1-(chloromethyl)-3,5-dimethyl-1*H*-pyrazole hydrochloride **1c** as a white solid, which was used directly without further purification. The crude product **1c** was dissolved in 1,2-dichloroethane giving a stock solution 0.75 M. 1-(chloromethyl)-3,5-dimethyl-1*H*-pyrazole hydrochloride **1c** (0.75 M, 0.75 mmol, 1.5 equiv.) was added in an oven dried Schlenk tube, then the DTC catalyst **A** (31.0 mg, 0.1 mmol, 0.2 equiv.) was added followed by 2,6-lutidine (70 µL, 0.6 mmol, 1.2 equiv.), *N*-methylmaleimide **2a** (57.0 mg, 0.5 mmol, 1 equiv), and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 20 hours. After cooling to ambient temperature, the solvent was evaporated. Multiple purification by column chromatography (gradient from 10% to 40% AcOEt in hexanes as eluent) resulted in poor separation from several unidentified byproducts, but an analytical amount of the pure major diastereomer was isolated for characterization. The yield (40%) of **4c** and the distereomeric ration (20:1) were inferred by ^{1}H NMR analysis of the crude reaction mixture using trichloroethylene as the internal standard.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.60 (dd, *J* = 2.8, 1.7 Hz, 1H), 6.05 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.90 (dd, *J* = 3.8, 1.1 Hz, 1H), 5.73 (s, 1H), 4.54 (dd, *J* = 14.4, 4.9 Hz, 1H), 4.43 (d, *J* = 5.7 Hz, 1H), 4.27 (dd, *J* = 14.4, 4.3 Hz, 1H), 3.68 (s, 3H), 3.34 (m), 2.96 (s, 3H), 2.20 (s, 3H), 2.12 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 176.7, 176.1, 148.4, 140.0, 126.7, 123.7, 107.3, 106.2, 105.6, 48.5, 45.6, 41.1, 34.4, 25.3, 13.6, 11.0.

HRMS: calculated for $C_{16}H_{20}N_4NaO_2^+$ ($M+Na^+$): 323.1478, found 323.1472.

TLC: 40:60 EtOAc/hexanes, R_f = 0.25.



4d

3-((3,5-dimethylisoxazol-4-yl)methyl)-1-methyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (4d): Synthesized according to the general procedure using 4-(chloromethyl)-3,5-dimethylisoxazole **2d** (109 mg, 93 µL, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4d** was purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent): 96.3 mg pale yellow solid, 64% yield.

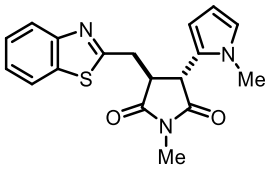
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.61 (dd, *J* = 2.8, 1.7 Hz, 1H), 6.04 (dd, *J* = 3.7, 2.8 Hz, 1H), 5.90 (dd, *J* = 3.8, 1.8, 0.7 Hz, 1H), 3.59 (s, 3H), 3.58 (d, *J* = 6.3 Hz, 1H), 3.26 (app q, *J* = 6.4 Hz, 1H), 2.96 (s, 3H), 2.86 (d, *J* = 6.3 Hz, 2H), 2.23 (s, 3H), 2.08 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.5, 175.3, 166.6, 159.6, 125.3, 124.2, 109.5, 107.4, 106.6, 46.5, 43.2, 34.3, 25.2, 21.9, 11.2, 10.2.

IR (thin film) ν 2924, 1779, 1701, 1429, 1377, 1268, 1110, 980, 698 cm^{-1}.

HRMS: calculated for $C_{16}H_{20}N_3O_3^+$ (M^+): 302.1496, found 302.1499

TLC: 30:70 EtOAc/hexanes, R_f = 0.27.



4e

3-(benzo[d]thiazol-2-ylmethyl)-1-methyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (4e): Synthesized according to the general procedure using 2-(chloromethyl)-1,3-benzothiazole **1e** (138 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4e** was purified by column chromatography (gradient from 10% to 30% EtOAc in hexanes as eluent, two consecutive purifications): 75.9 mg of a pink oil. The isolated material consisted of a mixture containing **4e** an inseparable byproduct in a proportion 6.3:1 (see Figure S2 below), arising from a polar Friedel-Crafts type alkylation of pyrrole with maleimide. Corrected yield of product **4e**: 38%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.1 Hz, 1H), 7.83 (d, *J* = 7.9 Hz, 1H), 7.45 (ddd, *J* = 8.3, 7.2, 1.3 Hz, 1H), 7.37 (ddd, *J* = 8.2, 7.3, 1.2 Hz, 1H), 6.60 (dd, *J* = 2.7, 1.8 Hz, 1H), 6.06 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.98 (dd, *J* = 3.8, 1.7 Hz, 1H), 4.33 (d, *J* = 5.9 Hz, 1H), 3.78 (dd, *J* = 15.9, 5.5 Hz, 1H), 3.66 (s, 3H), 3.56 (dd, *J* = 16.0, 4.6 Hz, 1H), 3.51 (app q, *J* = 5.3 Hz, 1H), 3.04 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.4, 176.1, 166.2, 153.1, 135.4, 126.6, 126.3, 125.4, 123.9, 123.1, 121.7, 107.4, 106.5, 46.6, 42.8, 34.5, 32.4, 25.4.

IR (thin film) ν 2924, 1776, 1695, 1491, 1432, 1381, 1279, 1108, 760, 709 cm^{-1}.

HRMS: calculated for $C_{18}H_{18}N_3O_2S^+$ (M^+): 340.1114, found 340.1113.

TLC: 30:70 EtOAc/hexanes R_f = 0.23.

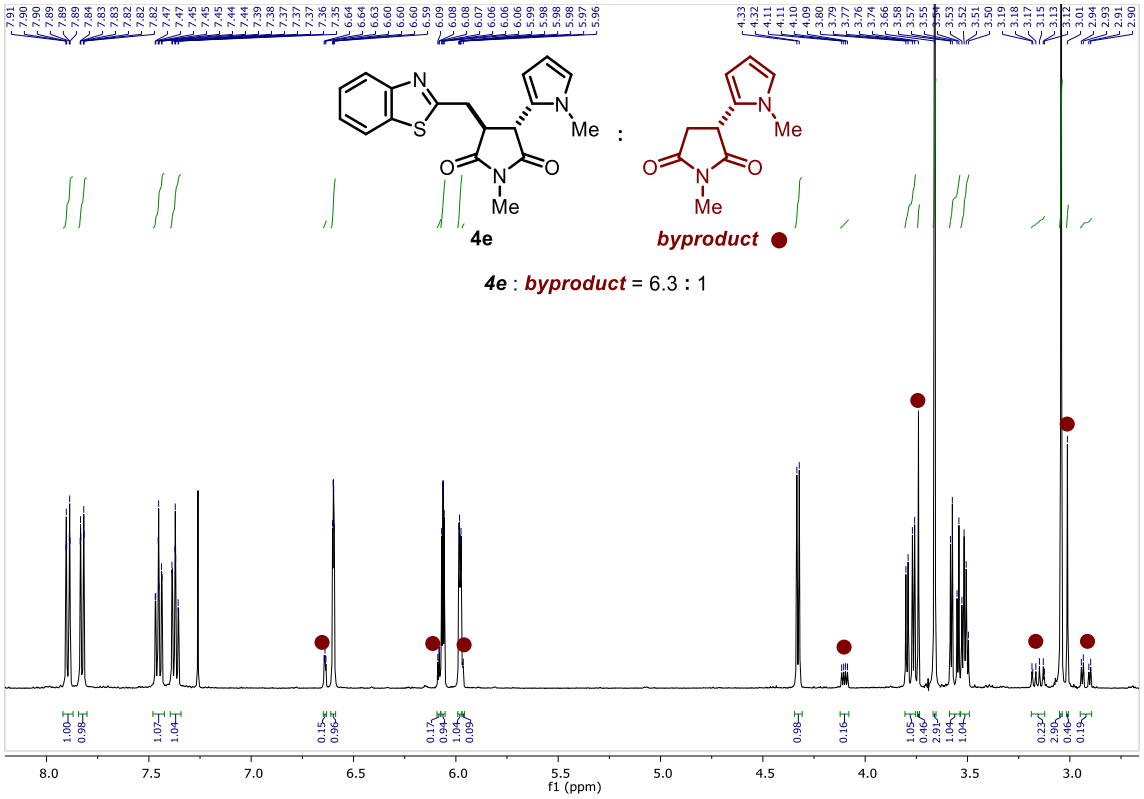
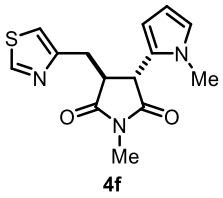


Figure S2. ^{1}H NMR spectrum of the mixture of product **4e** and byproduct (red dots) arising from a polar Friedel-Crafts type alkylation path.



1-methyl-3-(1-methyl-1H-pyrrol-2-yl)-4-(thiazol-4-ylmethyl)pyrrolidine-2,5-dione (4f): Synthesized according to a modification of the general procedure: in an oven dried Schlenk tube, 4-(chloromethyl)thiazole hydrochloride **1f** (128 mg, 0.75 mmol, 1.5 equiv.) was dissolved in 1,2-dichloroethane (1 mL, 0.5 M). Then 2,6-lutidine (157 µL, 1.35 mmol, 2.7 equiv.) was added followed by the DTC catalyst **A** (31 mg, 0.2 equiv., 20 mol%), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup set at a temperature of 60 ºC and irradiated for 20 hours. After cooling to ambient temperature, the solvent was evaporated. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4f** was purified by column chromatography (gradient from 20% to 30% AcOEt in hexanes as eluent): 58.6 mg of a pale yellow solid, 41% yield.

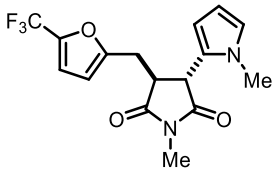
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.69 (d, *J* = 2.0 Hz, 1H), 7.05 (d, *J* = 1.5 Hz, 1H), 6.59 (t, *J* = 2.2 Hz, 1H), 6.05 (dd, *J* = 3.7, 2.8 Hz, 1H), 5.92 (dd, *J* = 3.9, 1.7 Hz, 1H), 4.13 (d, *J* = 5.4 Hz, 1H), 3.64 (s, 3H), 3.47 – 3.39 (m, 2H), 3.36 – 3.27 (m, 1H), 2.95 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.0, 176.3, 153.0, 152.9, 127.0, 123.6, 115.8, 107.3, 106.4, 47.4, 42.6, 34.4, 30.0, 25.2.

IR (thin film) ν 3078, 2921, 1691, 1433, 1290, 1067, 993, 731 cm^{-1}.

HRMS: calculated for $C_{14}H_{15}N_3NaO_2S^+$ ($M+Na^+$): 312.0777, found 312.0773.

TLC: 30:70 EtOAc/hexanes, R_f = 0.13.



4g

1-methyl-3-(1-methyl-1H-pyrrol-2-yl)-4-((5-(trifluoromethyl)furan-2-yl)methyl)pyrrolidine-2,5-dione (4g): Synthesized according to the general procedure using 2-(bromomethyl)-5-(trifluoromethyl)furan **1g** (172 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4g** was purified by column chromatography (gradient from 0% to 2% acetone in toluene as eluent; two consecutive purifications): 85.0 mg of a brown oil, 50% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.66 (dd, J = 3.3, 1.4 Hz, 1H), 6.61 (dd, *J* = 2.7, 1.7 Hz, 1H), 6.15 (d, *J* = 3.2 Hz, 1H), 6.05 (dd, *J* = 3.7, 2.8 Hz, 1H), 5.91 (dd, *J* = 3.8, 1.7 Hz, 1H), 3.81 (d, *J* = 5.9 Hz, 1H), 3.64 (s, 3H), 3.36 (app q, *J* = 5.9 Hz, 1H), 3.24 (d, *J* = 5.9 Hz, 2H), 2.99 (s, 3H).

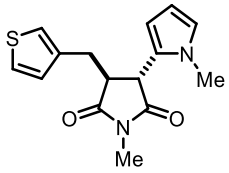
^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 175.4, 154.3 (d, J = 1.5 Hz), 141.4 (q, J = 42.8 Hz), 125.9, 124.0, 119.0 (q, J = 266.8 Hz), 112.6 (q, J = 2.9 Hz), 109.0, 107.3, 106.4, 46.4, 43.2, 34.2, 27.9, 25.2.

^{19}F NMR decoupled ^{1}H (376 MHz, $CDCl_3$) δ: -64.34

IR (thin film) ν 2950, 1779, 1698, 1616, 1560, 1434, 1382, 1321, 1173, 1122, 1103, 712 cm^{-1}.

HRMS: calculated for $C_{16}H_{16}F_3N_2O_3^+$ (M^+): 341.1108, found 341.1105.

TLC: 2:98 acetone/toluene, R_f = 0.35.



4h

1-methyl-3-(1-methyl-1H-pyrrol-2-yl)-4-(thiophen-3-ylmethyl)pyrrolidine-2,5-dione (4h): Synthesized by a two-step procedure. First, the alkyl chloride was synthesized: in an oven dried Schlenk tube, 3-thiophenemethanol (115 mg, 0.75 mmol, 1.5 equiv.) was dissolved in dry dichloromethane (5 mL) and cooled at 0 ºC. Thionyl chloride (88 µL, 1.2 mmol, 1.2 equiv.) was added dropwise and the reaction was left stirring at ambient temperature for 30 min. Solvent was removed under vacuum at 25 ºC, diethyl ether (5 mL) was added and dried (this was repeated twice) to obtain the crude 3-(chloromethyl)thiophene **1h** as a colorless oil in quantitative yield, which was used without further purification. The crude adduct **1h** was dissolved in 1,2-dichloroethane giving a stock solution 0.75 M. 3-(chloromethyl)thiophene **1h** (0.75 M, 0.75 mmol, 1.5 equiv.) was added in an oven dried Schlenk tube, then the DTC catalyst **A** (31 mg, 0.2 equiv.) was added followed by 2,6-lutidine (157 µL, 1.35 mmol, 2.7 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup at a temperature of 60 ºC and irradiated for 20 hours. After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (two consecutive purifications; first purification: gradient from 10% to 20% AcOEt in hexanes as eluent; second purification: gradient from 0% to 2% acetone in toluene as eluent): 100.3 mg of **4h** isolated as a brown oil, 66% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

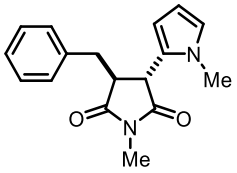
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.25 (m, 1H), 6.98 – 6.96 (m, 1H), 6.86 (dd, *J* = 5.0, 1.3 Hz, 1H), 6.60 (dd, *J* = 2.7, 1.8 Hz, 1H), 6.07 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.95 (ddd, *J* = 3.7, 1.8, 0.6 Hz, 1H), 3.71 (d, *J* = 5.5 Hz, 1H), 3.54 (s, 3H), 3.37 – 3.33 (m, 1H), 3.29 (ddd, *J* = 14.5, 6.3, 0.8 Hz, 1H), 3.12 (dd, *J* = 14.5, 5.1 Hz, 1H), 2.96 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 176.0, 136.8, 128.4, 126.6, 126.5, 123.7, 123.0, 107.3, 106.5, 47.9, 42.5, 34.2, 29.5, 25.1.

IR (thin film) ν 2925, 1776, 1695, 1491, 1432, 1380, 1286, 1119, 1090, 752, 711 cm^{-1}.

HRMS: calculated for $C_{15}H_{17}N_2O_2S^+$ ($M+H^+$):289.1005, found 289.1016.

TLC: 30:70 EtOAc/hexanes, R_f = 0.38.



4i

3-benzyl-1-methyl-4-(1-methyl-1*H*-pyrrol-2-yl)pyrrolidine-2,5-dione (4i): Synthesized according to the general procedure using benzyl bromide (89 µL, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The diastereomeric ratio (7.6:1) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 5.95 (minor diastereomer) and δ 5.87 (major diastereomer). Product **4i** purified by column chromatography (gradient from 15% to 25% AcOEt in hexanes as eluent): 69 mg of a brown oil, 49% yield.

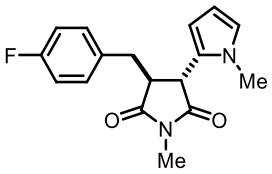
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29-7.20 (m, 3H), 7.14-7.11 (m, 2H), 6.55 (dd, *J* = 2.6, 1.8 Hz, 1H), 6.04 (dd, *J* = 3.6, 2.9 Hz, 1H), 5.91 (dd, *J* = 3.6, 1.6 Hz, 1H), 3.70 (d, *J* = 5.5 Hz, 1H), 3.47 (s, 3H), 3.37 (app q, *J* = 5.7 Hz, 1H), 3.22 (dd, *J* = 14.1, 6.5 Hz, 1H), 3.10 (dd, *J* = 14.1, 5.5 Hz, 1H), 2.94 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.2, 176.0, 136.7, 129.5, 128.9, 127.3, 126.6, 123.6, 107.3, 106.5, 48.4, 42.3, 35.1, 34.2, 25.1.

IR (thin film) ν 2926, 1776, 1695, 1494, 1432, 1382, 1287, 1122, 992, 703 cm^{-1}.

HRMS: calculated for $C_{17}H_{19}N_2O_2^+$ ($M+H^+$): 283.1441, found 283.1437.

TLC: 20:80 EtOAc/hexanes, R_f = 0.19.



4j

3-(4-fluorobenzyl)-1-methyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (4j): Synthesized according to the general procedure using 4-fluorobenzyl bromide (142 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The diastereomeric ratio (5.3:1) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 5.92 (major diastereomer) and δ 5.95 (minor diastereomer). Product **4j** purified by column chromatography (gradient from 10% to 20% AcOEt in hexanes as eluent): 82.2 mg of a brown oil, 55% yield.

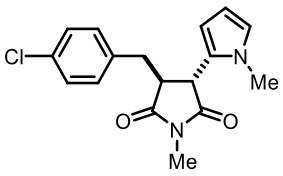
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 – 7.08 (m, 2H), 7.00 – 6.94 (m, 2H), 6.59 (dd, *J* = 2.8, 1.7 Hz, 1H), 6.06 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.93 (dd, *J* = 3.7, 1.6 Hz, 1H), 3.68 (d, *J* = 5.7 Hz, 1H), 3.52 (s, 3H), 3.37 (app q, *J* = 5.8 Hz, 1H), 3.21 (dd, *J* = 14.3, 6.3 Hz, 1H), 3.08 (dd, *J* = 14.3, 5.5 Hz, 1H), 2.96 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.0, 175.8, 162.1 (d, *J* = 246.0 Hz), 132.4 (d, *J* = 3.3 Hz), 131.0 (d, *J* = 8.0 Hz), 126.3, 123.9, 115.8 (d, *J* = 21.3 Hz), 107.3, 106.7, 48.3, 42.4, 34.3, 34.2, 25.2.
^{19}F NMR decoupled ^{1}H (471 MHz, $CDCl_3$) δ: -115.32

IR (thin film) ν 2925, 1696, 1508, 1432, 1381, 1286, 1221, 1120, 711 cm^{-1}.

HRMS: calculated for $C_{17}H_{18}FN_2O_2^+$ (M^+): 301.1347, found 301.1359.

TLC: 20:80 EtOAc/hexanes, R_f = 0.15.



4k

(3*S*,4*R*)-3-(4-chlorobenzyl)-1-methyl-4-(1-methyl-1*H*-pyrrol-2-yl)pyrrolidine-2,5-dione (4k): Synthesized according to the general procedure using 4-chlorobenzyl bromide (154 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The diastereomeric ratio (6.3:1) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 5.82 (major diastereomer) and δ 5.77 (minor diastereomer). Product **4k** was purified by column chromatography (gradient from 15% to 25% AcOEt in hexanes as eluent): 65 mg of a brown oil,

41% yield. DMSO-d_6 was used as a co-solvent in the ^{13}C NMR spectra to prevent overlap of aliphatic peaks.

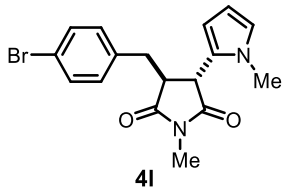
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.5 Hz, 2H), 7.06 (d, *J* = 8.4 Hz, 2H), 6.58 (dd, *J* = 2.6, 1.8 Hz, 1H), 6.04 (d, *J* = 3.6, 2.9 Hz, 1H), 5.91 (dd, *J* = 3.6, 1.6 Hz, 1H), 3.65 (d, *J* = 5.8 Hz, 1H), 3.51 (s, 3H), 3.36 (app q, *J* = 5.9 Hz, 1H), 3.20 (dd, *J* = 14.3, 6.4 Hz, 1H), 3.05 (dd, *J* = 14.2, 5.6 Hz, 1H), 2.94 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$ + DMSO-d_6) δ 177.0, 175.2, 135.7, 131.5, 130.4, 128.0, 126.1, 122.7, 106.4, 106.1, 47.4, 42.2, 33.8, 33.4, 24.4.

IR (thin film) ν 2945, 1776, 1697, 1491, 1433, 1382, 1286, 1092, 1015, 716 cm^{-1}.

HRMS: calculated for $C_{17}H_{18}ClN_2O_2^+$ (M^+): 317.1051, found 317.1054.

TLC: 25:75 EtOAc/hexanes, R_f = 0.26.



4l

(3*S*,4*R*)-3-(4-bromobenzyl)-1-methyl-4-(1-methyl-1*H*-pyrrol-2-yl)pyrrolidine-2,5-dione (4l): Synthesized according to the general procedure using 4-bromobenzyl bromide (187 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). Product **4l** was purified by column chromatography (gradient from 15% to 25% AcOEt in hexanes as eluent): 104 mg browm oil, 57% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

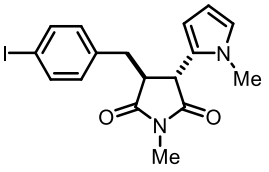
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.02 (d, *J* = 8.4 Hz, 2H), 6.59 (dd, *J* = 2.5, 1.9 Hz, 1H), 6.06 (dd, *J* = 3.6, 2.9 Hz, 1H), 5.93 (dd, *J* = 3.6, 1.6 Hz, 1H), 3.66 (d, *J* = 5.9 Hz, 1H), 3.53 (s, 3H), 3.37 (app q, *J* = 5.9 Hz, 1H), 3.20 (dd, *J* = 14.3, 6.3 Hz, 1H), 3.05 (dd, *J* = 14.3, 5.6 Hz, 1H), 2.95 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.7, 175.6, 135.7, 131.9, 131.1, 126.1, 123.8, 121.2, 107.3, 106.6, 47.9, 42.3, 34.3, 34.2. 25.1.

IR (thin film) ν 2945, 1776, 1694, 1488, 1431, 1381, 1284, 1121, 1090, 714 cm^{-1}.

HRMS: calculated for $C_{17}H_{17}BrN_2NaO_2^+$ ($M+Na^+$): 383.0366, found 383.0360.

TLC: 25:75 EtOAc/hexanes, R_f = 0.26.



4m

3-(4-iodobenzyl)-1-methyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (**4m**): Synthesized according to the general procedure using 4-iodobenzyl bromide (223 mg, 0.75 mmol, 1.5 equiv.), N-methyl maleimide **2a** (56 mg, 0.5 mmol, 1 equiv) and N-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). Product **4m** was purified by column chromatography (gradient from 15% to 25% AcOEt in hexanes as eluent): 128 mg of a pale yellow sticky oil, 63% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

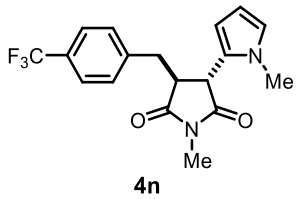
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60-7.55 (m, 2H), 6.89-6.84 (m, 2H), 6.87 (dd, *J* = 2.5, 1.8 Hz, 1H), 6.04 (dd, *J* = 3.6, 2.5 Hz, 1H), 5.9 (dd, *J* = 3.6, 1.8 Hz; 1H), 3.64 (d, *J* = 5.8 Hz; 1H), 3.51 (s, 3H), 3.35 (d, *J* = 5.9 Hz; 1H), 3.17 (dd, *J* = 14.7, 6.2 Hz; 1H), 3.01 (dd, *J* = 14.7, 5.5 Hz; 1H), 2.93 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 178.0, 175.9, 138.2 (x2), 136.6, 131.7 (x2), 126.3, 124.2, 107.6, 106.9, 93.0, 48.2, 42.6, 34.6, 34.6, 25.4.

IR (thin film) ν 2924, 1775, 1694, 1484, 1430, 1380, 1285, 1120, 1089, 1006, 711 cm^{-1}.

HRMS: calculated for $C_{17}H_{17}IN_2O_2^+$ ($M+H^+$): 409.0708, found 409.0387

TLC: 20:80 EtOAc/hexanes, *Rf* = 0.2.



4n

1-methyl-3-(1-methyl-1H-pyrrol-2-yl)-4-(4-(trifluoromethyl)benzyl)pyrrolidine-2,5-dione (4n): Synthesized according to the general procedure using 4-(trifluoromethyl)benzyl bromide (180 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The diastereomeric ratio (4.2:1) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 5.92 (major diastereomer) and δ 5.95 (minor diastereomer). Product **4n** was purified by column chromatography (gradient from 10% to 20% AcOEt in hexanes as eluent): 72.4 mg of a brown oil, 41% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 7.8 Hz, 2H), 6.58 (dd, *J* = 2.7, 1.8 Hz, 1H), 6.04 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.92 (dd, *J* = 3.9, 1.7 Hz, 1H), 3.64 (d, *J* = 5.9 Hz, 1H), 3.51 (s, 3H), 3.42 (app q, *J* = 6.0 Hz, 1H), 3.26 (dd, *J* = 14.2, 6.4 Hz, 1H), 3.17 (dd, *J* = 14.2, 5.7 Hz, 1H), 2.95 (s, 3H).

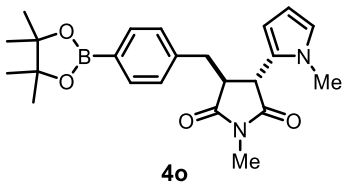
^{13}C NMR (126 MHz, $CDCl_3$) δ 177.6, 175.5, 141.0, 129.9, 129.7 (q, J = 32.5 Hz), 125.9, 125.8 (q, J = 3.8 Hz), 124.1 (q, J = 272.0 Hz), 124.0, 107.4, 106.8, 47.9, 42.6, 34.8, 34.3, 25.2.

^{19}F NMR decoupled ^{1}H (471 MHz, $CDCl_3$) δ: -62.67

IR (thin film) ν 2925, 1778, 1698, 1433, 1382, 1322, 1287, 1162, 1110, 1066, 712 cm^{-1}.

HRMS: calculated for $C_{18}H_{17}F_3N_2NaO_2^+$ ($M+Na^+$): 373.1134, found 373.1119.

TLC: 20:80 EtOAc/hexanes, R_f = 0.23.



4o

1-methyl-3-(1-methyl-1H-pyrrol-2-yl)-4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)pyrrolidine-2,5-dione (4o): Synthesized according to the general procedure using 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl bromide (223 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). Product **4o** was purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent): 39.3 mg pale brown oil, 28% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.58 (dd, *J* = 2.8, 1.8 Hz, 1H), 6.06 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.94 (dd, *J* = 3.6, 1.6 Hz, 1H), 3.68 (d, *J* = 5.5 Hz, 1H), 3.50 (s, 3H), 3.40 (app q, *J* = 5.7 Hz, 1H), 3.30 (dd, *J* = 14.0, 6.0 Hz, 1H), 3.07 (dd, *J* = 14.1, 5.4 Hz, 1H), 2.94 (s, 3H), 1.34 (s, 12H).

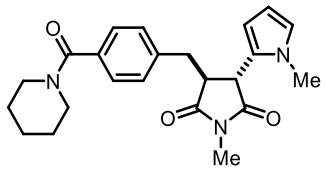
^{13}C NMR (126 MHz, $CDCl_3$) δ 178.1, 176.0, 139.8, 135.4, 129.0, 126.5, 123.8, 107.3, 106.5, 84.0, 48.2, 42.1, 35.0, 34.3, 25.2, 25.0.

^{11}B NMR (160 MHz, $CDCl_3$) δ 30.76.

IR (thin film) ν 3399, 2978, 1696, 1612, 1433, 1359, 1289, 1142, 1090, 1022, 658 cm^{-1}.

HRMS: calculated for $C_{23}H_{29}N_2NaO_4{}^{11}B^+$ ($M+Na^+$): 431.2113, found 431.2122.

TLC: 20:80 EtOAc/hexanes, R_f = 0.31.



4p

2-(5-(4-((1,3-dioxoisoindolin-2-yl)methyl)-1-methyl-2,5-dioxopyrrolidin-3-yl)thiophen-2-yl)acetonitrile (**4p**): Synthesized according to the general procedure using (4-(bromomethyl)phenyl)(piperidin-1-yl)methanone (212 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and N-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). Product **4p** was purified by column chromatography (two consecutive purifications. First purification: gradient from 40% to 60% AcOEt in hexanes as eluent. Second purification: 90:10 tol:acetone): 140.3 mg of a pale yellow solid, 71% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

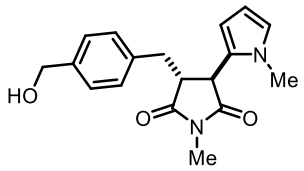
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 – 7.29 (m, 2H), 7.19 – 7.15 (m, 2H), 6.56 (dd, J = 2.8, 1.7 Hz, 1H), 6.03 (dd, J = 3.7, 2.7 Hz, 1H), 5.91 (ddd, J = 3.8, 1.8, 0.6 Hz, 1H), 3.69 (d+bs, J = 6.0 Hz, 1H+2H), 3.52 (s, 3H), 3.41 (app q, J = 5.9 Hz, 1H), 3.28 (s, 2H), 3.24 (dd, J = 14.2, 6.5 Hz, 1H), 3.14 (dd, J = 14.2, 5.4 Hz, 1H), 2.96 (s, 3H), 1.67 (bs, 4H), 1.50 (bs, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.7, 175.5, 169.8, 138.0, 135.4, 129.4 (x2), 127.3 (x2), 125.9, 123.7, 107.1, 106.5, 48.8 (broad), 47.1, 43.2 (broad), 42.3, 34.5, 34.2, 26. (broad), 25.5 (broad), 25.0, 24.5.

HRMS: calculated for $C_{23}H_{28}N_3O_3^+$ ($M+H^+$): 394.2125, found 394.2131.

IR (thin film) ν 2932, 2854, 1776, 1696, 1619, 1430, 1274, 1109, 706 cm^{-1}.

TLC: 60:40 EtOAc/hexanes, *Rf* = 0.16.



4q

3-(4-(hydroxymethyl)benzyl)-1-methyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (**4q**): Synthesized according to the general procedure using 4-hydroxymethylbenzyl bromide (150 mg, 0.75 mmol, 1.5 equiv.), N-methyl maleimide **2a** (56 mg, 0.5 mmol, 1 equiv) and N-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). Product **4q** was purified by column chromatography (gradient from 40% to 60% AcOEt in hexanes as eluent): 77 mg of a yellowish sticky oil, 49% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

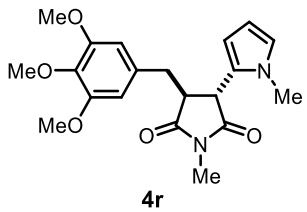
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27-7.23 (m, 2H), 7.12-7.08 (m, 2H), 6.56 (dd, *J* = 2.6, 1.9 Hz, 1H), 6.03 (dd, *J* = 3.4, 2.6 Hz, 1H), 5.9 (dd, *J* = 3.4, 1.9 Hz; 1H), 4.62 (br s, 2H); 3.68 (d, *J* = 5.5 Hz; 1H), 3.49 (s, 3H), 3.36 (q, *J* = 5.7 Hz; 1H), 3.23 (dd, *J* = 14.1, 6.1 Hz; 1H), 3.05 (dd, *J* = 14.1, 5.4 Hz; 1H), 2.92 (s, 3H); 1.89 (br s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 178.4; 176.2; 140.2; 136.2; 129.9 (x2); 127.7 (x2); 126.6;.124.0; 107.5; 106.8; 65.2; 48.5; 42.5; 34.8; 34.5; 25.4.

IR (thin film) ν 3455, 2924, 1693, 1434, 1382, 1288, 1122, 717cm^{-1}.

HRMS: calculated for $C_{18}H_{20}N_2NaO_3^+$ ($M+Na^+$): 335.1366, found 335.1365.

TLC: 50:50 EtOAc/hexanes, *Rf* = 0.25.



4r

1-methyl-3-(1-methyl-1*H*-pyrrol-2-yl)-4-(3,4,5-trimethoxybenzyl)pyrrolidine-2,5-dione (4r): Synthesized according to the general procedure using 5-(chloromethyl)-1,2,3-trimethoxybenzene (162 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Purification by column chromatography (gradient from 20% to 35% AcOEt in hexanes as eluent) resulted in poor separation, but a clean amount (50 mg) of pure **4r** was isolated for

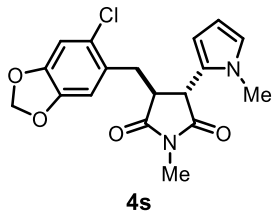
characterization (50 mg). 58% NMR-yield, determined using trichloroethylene as the internal standard. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.57 (dd, *J* = 2.6, 1.9 Hz, 1H), 6.32 (s, 2H), 6.06 (dd, *J* = 3.5, 2.9 Hz, 1H), 5.94 (dd, *J* = 3.6, 1.7 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 6H), 3.71 (d, *J* = 5.7 Hz, 1H), 3.50 (s, 3H), 3.40 (app q, *J* = 5.8 Hz, 1H), 3.15 (dd, *J* = 14.5, 6.8 Hz, 1H), 3.08 (dd, *J* = 14.4, 5.5 Hz, 1H), 2.97 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.5, 176.3, 153.7, 137.3, 132.7, 126.9, 124.0, 107.7, 106.8, 106.5, 61.2, 56.4, 48.4, 42.8, 35.6, 34.5, 25.5.

IR (thin film) ν 2939, 2839, 1697, 1589, 1507, 1458, 1431, 1382,1123, 1006 cm^{-1}.

HRMS: calculated for $C_{20}H_{24}N_2NaO_5^+$ ($M+Na^+$): 395.1577, found 395.1581.

TLC: 30:70 EtOAc/hexanes, R_f = 0.11.



4s

3-((6-chlorobenzo[d][1,3]dioxol-5-yl)methyl)-1-methyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (4s): Synthesized according to the general procedure 6-chloropiperonyl chloride (154 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4s** was purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent): 133 mg of a white foam, 72% yield.

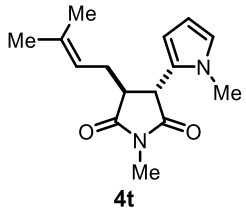
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.8 (s, 1H), 6.7 (s, 1H), 6.6 (dd, *J* = 2.7, 1.7 Hz, 1H), 6.0 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.9 (s, 2H), 5.9 (dd, *J* = 3.6, 1.5 Hz, 1H), 3.8 (d, *J* = 5.4 Hz, 1H), 3.6 (s, 3H), 3.4 (m, 1H), 3.3 (dd, *J* = 14.1, 5.7 Hz, 1H), 3.1 (dd, *J* = 14.2, 7.1 Hz, 1H), 3.0 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 178.0, 175.9, 147.5, 147.1, 127.7, 126.4, 126.0, 123.7, 110.4, 109.9, 107.1, 106.2, 101.9, 47.5, 42.6, 34.2, 32.7, 25.2

IR (thin film) ν 2900, 1776, 1696, 1478, 1433, 1381, 1286, 1232, 1118, 1035, 749, 712 cm^{-1}.

HRMS: calculated for $C_{18}H_{17}ClN_2NaO_4^+$ ($M+Na^+$): 383.0769, found 383.0770.

TLC: 30:70 EtOAc/hexanes, R_f = 0.33



4t

1-methyl-3-(1-methyl-1*H*-pyrrol-2-yl)-4-(3-methylbut-2-en-1-yl)pyrrolidine-2,5-dione (4t): Synthesized according to the general procedure using 1-chloro-3-methylbut-2-ene chloride (170 µL, 1.50 mmol, 3.0 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The diastereomeric ratio (2.9:1) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 5.10 (minor diastereomer) and δ 4.87 (major diastereomer). Product **4t** was purified by column chromatography (gradient from 15% to 20% AcOEt in hexanes as eluent): 62 mg of a brown oil, 47% yield.

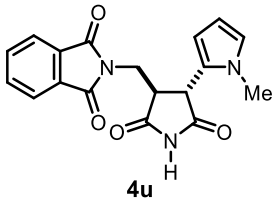
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.62 (dd, *J* = 2.6, 1.9 Hz, 1H), 6.07 (dd, *J* = 3.6, 2.9 Hz, 1H), 5.93 (dd, *J* = 3.6, 1.6 Hz, 1H), 5.05-5.00 (m, 1H), 3.73 (d, *J* = 5.3 Hz, 1H), 3.67 (s, 3H), 3.11 (app q, *J* = 5.5 Hz, 1H), 2.99 (s, 3H), 2.57-2.52 (m, 2H), 1.69 (d, *J* = 0.9 Hz, 1H), 1.62 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 176.4, 136.6, 126.9, 123.8, 118.5, 107.3, 106.7, 47.2, 43.0, 34.4, 28.2, 26.0, 25.1, 18.1.

IR (thin film) ν 2918, 1776, 1696, 1492, 1432, 1380, 1285, 1095, 995, 713 cm^{-1}.

HRMS: calculated for $C_{15}H_{20}N_2NaO_2^+$ ($M+Na^+$): 283.1417, found 283.1415.

TLC: 20:80 EtOAc/hexanes, R_f = 0.28.



4u

2-((4-(1-methyl-1*H*-pyrrol-2-yl)-2,5-dioxopyrrolidin-3-yl)methyl)isoindoline-1,3-dione (4u): Synthesized according to the general procedure using *N*-(chloromethyl)phthalimide chloride **1a** (147 mg, 0.75 mmol, 1.5 equiv.), unprotected maleimide **2b** (48 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). The diastereomeric ratio (1.9:1) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the resonances at δ 5.96 (minor diastereomer) and δ 5.72 (major diastereomer). Purification by column chromatography (gradient from 40% to 55% AcOEt in hexanes as eluent) resulted in very poor separation, but an analytical amount of the major diastereomer was isolated for characterization. NMR yield of the mixture of diastereoisomers (1.9:1) is 77%. DMSO-d_6 was used either as a solvent or co-solvent in the NMR spectra to promote solubility; in the case of ^{13}C NMR, chloroform was required in order to prevent overlap of aromatic peaks.

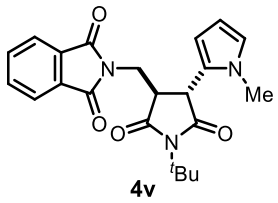
^{1}H NMR (500 MHz, DMSO-d_6) δ 11.30 (s, 1H), 7.77 (s, 4H), 6.48 (app t, *J* = 2.1 Hz, 1H), 5.76 (dd, *J* = 3.5, 1.8 Hz, 1H), 5.60 (dd, *J* = 3.5, 2.8 Hz, 1H), 4.22 (d, *J* = 7.3 Hz, 1H), 4.09 (dd, *J* = 14.0, 6.0 Hz, 1H), 4.02 (dd, *J* = 14.1, 9.0 Hz, 1H), 3.53 (s, 3H), 3.52-3.48 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$ + DMSO-d_6) δ 176.2, 175.9, 168.4, 134.3, 131.9, 125.4, 123.8, 123.5, 107.2, 106.8, 46.2, 45.0, 38.0, 34.3.

IR (thin film) ν 3415, 2924, 2255, 1715, 1399, 1364, 1024, 1003, 823, 722 cm^{-1}.

HRMS: calculated for $C_{18}H_{15}N_3NaO_4^+$ (M+Na$^+$): 360.0955, found 360.0958.

TLC: 50:50 EtOAc/hexanes, R_f = 0.31.



4v

2-((1-(*tert*-butyl)-4-(1-methyl-1*H*-pyrrol-2-yl)-2,5-dioxopyrrolidin-3-yl)methyl)isoindoline-1,3-dione (4v): Synthesized according to the general procedure using *N*-(chloromethyl)phthalimide chloride **1a** (147 mg, 0.75 mmol, 1.5 equiv.), *N*-*tert*-butylmaleimide **2c** (76 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4v** was purified by column chromatography (gradient from 15% to 25% AcOEt in hexanes as eluent): 127 mg of an orange foam, 64% yield.

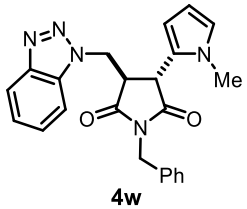
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73-7.69 (m, 2H), 7.65-7.62 (m, 2H), 6.32 (dd, *J* = 2.5, 1.9 Hz, 1H), 5.74 (dd, *J* = 3.6, 1.6 Hz, 1H), 5.68 (dd, *J* = 3.6, 2.8 Hz, 1H), 4.26 (dd, *J* = 14.0, 5.7 Hz, 1H), 3.99 (dd, *J* = 14.0, 9.6 Hz, 1H), 3.88 (d, *J* = 7.3 Hz, 1H), 3.59 (s, 3H), 3.47-3.42 (m, 1H), 1.55 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 176.7, 176.2, 168.5, 134.3, 132.0, 126.1, 123.8, 123.5, 107.3, 106.4, 59.2, 45.0, 43.9, 38.4, 34.3, 28.7.

IR (thin film) ν 2976, 1774, 1697, 1436, 1396, 1330, 1263, 1158, 1106, 714 cm^{-1}.

HRMS: calculated for $C_{22}H_{24}N_3O_4^+$ (M+H$^+$): 394.1761, found 394.1765.

TLC: 20:80 EtOAc/hexanes, R_f = 0.14.



4w

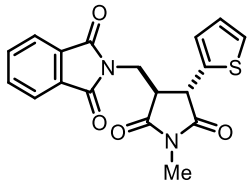
3-((1H-benzo[d][1,2,3]triazol-1-yl)methyl)-1-benzyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidine-2,5-dione (4w): Synthesized according to the general procedure using 1-(chloromethyl)-1*H*-benzotriazole **1b** (126 mg, 0.75 mmol, 1.5 equiv.), *N*-benzylmaleimide **2d** (94 mg, 0.5 mmol, 1 equiv.) and *N*-methylpyrrole **3a** (445 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4w** was purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent): 112.1 mg of a white foam, 56% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dt, *J* = 8.4, 0.9 Hz, 1H), 7.61 (dt, *J* = 8.4, 0.9 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.37 (ddd, *J* = 8.1, 6.9, 1.0 Hz, 1H), 7.20 – 7.15 (m, 1H), 7.14 – 7.09 (m, 2H), 7.03 – 6.98 (m, 2H), 6.62 (dd, *J* = 2.7, 1.7 Hz, 1H), 6.05 (dd, *J* = 3.7, 2.7 Hz, 1H), 5.92 (dd, *J* = 3.5, 1.6 Hz, 1H), 5.24 (dd, *J* = 14.7, 4.3 Hz, 1H), 5.00 (dd, *J* = 14.8, 4.1 Hz, 1H), 4.57 (d, *J* = 14.3 Hz, 1H), 4.48 (d, *J* = 14.3 Hz, 1H), 4.32 (d, *J* = 6.7 Hz, 1H), 3.67 (s, 3H), 3.62 (dt, *J* = 6.7, 4.2 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.4, 174.5, 145.9, 135.0, 133.4, 128.7 (2C), 128.2, 128.1 (2C), 128.0, 125.4, 124.5, 124.2, 120.1, 109.9, 107.4, 106.6, 47.9, 45.3, 42.9, 41.1, 34.6.

HRMS: calculated for $C_{23}H_{22}N_5O_2^+$ ($M+H^+$): 400.1778, found 400.1768.

TLC: 30:70 EtOAc/hexanes, R_f = 0.13.



4x

2-((1-methyl-2,5-dioxo-4-(thiophen-2-yl)pyrrolidin-3-yl)methyl)isoindoline-1,3-dione (4x): Synthesized according to the general procedure using *N*-(chloromethyl)phthalimide chloride **1a** (147 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and thiophene **3b** (400 µL, 5 mmol, 10 equiv.). A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **4x** was purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent): 64.6 mg pale brown solid, 36% yield.

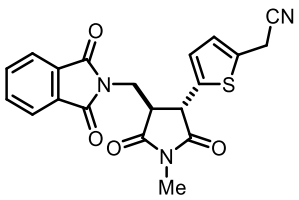
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.73 – 7.69 (m, 2H), 7.08 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.89 (dd, *J* = 3.6, 1.1 Hz, 1H), 6.78 (dd, *J* = 5.2, 3.5 Hz, 1H), 4.32 (dd, *J* = 14.1, 6.2 Hz, 1H), 4.21 (d, *J* = 6.3 Hz, 1H), 4.09 (dd, *J* = 14.1, 9.2 Hz, 1H), 3.66 – 3.56 (m, 1H), 3.07 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 175.0, 175.0, 168.2, 136.9, 134.4, 131.8, 127.1, 126.3, 125.7, 123.6, 47.7, 46.3, 37.9, 25.6.

IR (thin film) ν 2924, 1766, 1693, 1435, 1401, 1365, 1298, 1190, 1107, 1067, 692 cm^{-1}.

HRMS: calculated for $C_{18}H_{14}N_2NaO_4S^+$ ($M+Na^+$): 377.0566, found 377.0558

TLC: 30:70 EtOAc/hexanes, R_f = 0.22.



4y

2-(5-(4-((1,3-dioxoisoindolin-2-yl)methyl)-1-methyl-2,5-dioxopyrrolidin-3-yl)thiophen-2-yl)acetonitrile (**4y**): Synthesized according to the general procedure using *N*-(chloromethyl)phthalimide **1a** (147 mg, 0.75 mmol, 1.5 equiv.), *N*-methylmaleimide **2a** (56 mg, 0.5 mmol, 1 equiv.) and thiophene-2-acetonitrile **3sj** (615 µL, 5 mmol, 10 equiv.). Product **4y** was purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent): 40 mg of a pale yellow solid, 20% yield. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture.

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.83 – 7.80 (m, 4H), 6.82 (dd, *J* = 3.6, 0.9 Hz, 1H), 6.75 (dt, *J* = 3.6, 1.1 Hz, 1H), 4.27 (d, *J* = 6.9 Hz, 1H), 4.14 (dd, *J* = 14.1, 6.4 Hz, 1H), 4.10 (d, *J* = 1.0 Hz, 2H), 4.04 (dd, *J* = 14.1, 8.3 Hz, 1H), 3.54 (dt, *J* = 8.2, 6.6 Hz, 1H), 2.85 (s, 3H).

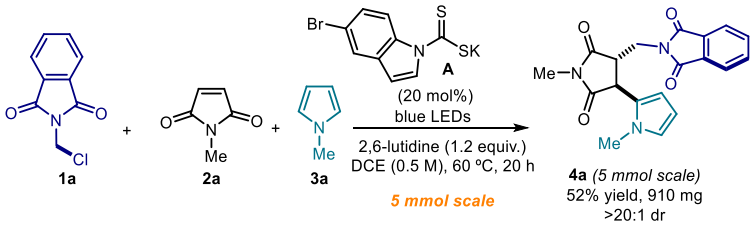
^{13}C NMR (125 MHz, $CDCl_3$) δ 175.2, 175.0, 167.8 (x2), 137.7, 134.5 (x2), 132.0, 131.3, 126.7, 125.9, 123.1 (CN), 123.1(x2), 118.2, 46.2, 45.6, 36.9, 24.9, 17.4.

IR (thin film) ν 2913, 1767, 1693, 1440, 1399, 1363, 1299, 1019, 724 cm^{-1}.

HRMS: calculated for $C_{20}H_{15}N_3NaO_4S^+$ (M+Na+): 416.0669, found 416.0675.

TLC: 50:50 EtOAc/hexanes, *Rf* = 0.43.

B.4. 5 mmol Scale Reaction



The model photoinduced multicomponent reaction can be scaled-up up to 5 mmol scale by using the same experimental set-up described in Figure S1. In an oven dried Schlenk tube (length x diameter = 22 x 2 cm), the DTC catalyst **A** (310 mg, 1 mmol, 0.2 equiv.) was dissolved in dichloroethane (5 mL), then the *N*-(chloromethyl)phthalimide **1a** (1.5 g, 7.5 mmol, 1.5 equiv.) was added, with stirring, followed by 2,6-lutidine (700 µL, 6 mmol, 1.2 equiv.), *N*-methylmaleimide **2a** (556 mg, 5 mmol, 1.0 equiv.), and *N*-methylpyrrole **3a** (4.4 mL, 50 mmol, 10.0 equiv.). An additional volume of dichloroethane (5 mL) was added to the reaction tube, washing the sides from residual solids. The resulting mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup, maintained at a temperature of 60 ºC (60-61ºC measured in the central well), and the reaction was stirred for 30 hours under continuous irradiation.

After cooling to ambient temperature, the solvent was evaporated and the residue purified by column chromatography (gradient from 10% to 30% AcOEt in hexanes as eluent). Peroduct 4a was isolated as a mixture containing about 14% of an inseparable byproduct, arising from a polar Friedel-Crafts type alkylation of pyrrole **3a** with maleimide **2a**: 1.05 g (containing 14% of byproduct). Corrected yield of product **4a**: 52% (906 mg). The NMR yield of the observed single diastereomer was measured as 62%, using 1,1,2-trichloroethene as the internal standard.

C. Product Modifications

C.1. Assembly Line Synthesis of Difunctionalized Pyrroles

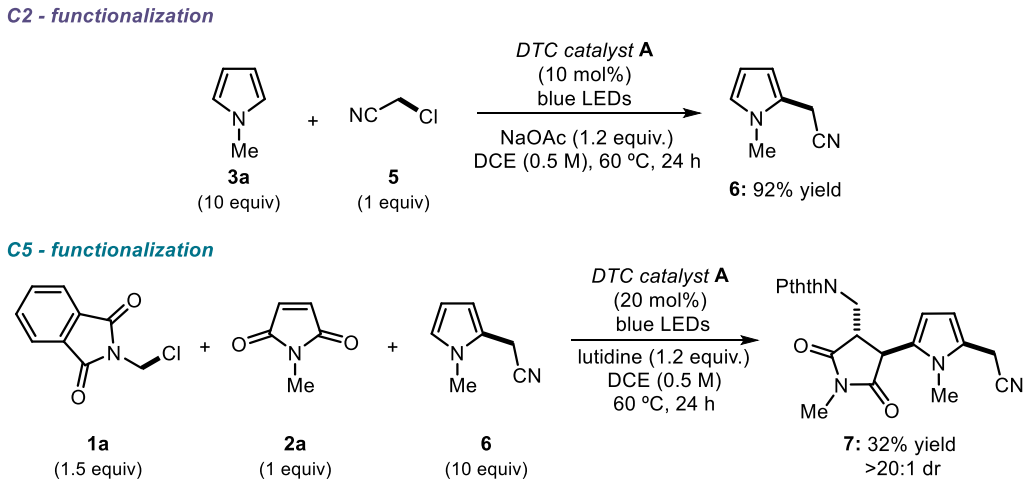
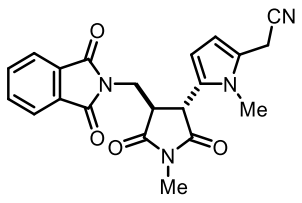


Figure S3. Sequential C2 and C5-functionalization of *N*-methyl pyrrole **3a**

C2-functionalization. In an oven dried Schlenk tube, the DTC catalyst **A** (15.5 mg, 0.05 mmol, 0.1 equiv.) and sodium acetate (49 mg, 0.6 mmol, 1.2 equiv.) were suspended in 1,2-dichloroethane (1 mL), then chloroacetonitrile **5** (32 µL, 0.5 mmol, 1 equiv.) was added followed by *N*-methylpyrrole **3a** (444 µL, 5 mmol, 10 equiv.). The resulting yellow mixture was degassed via three cycles of freeze-pump-thaw. The Schlenk tube was then placed in the irradiation setup (see Figure S1) set at a temperature of 60 ºC (60-61ºC measured in the central well) and irradiated for 24 hours. After cooling to ambient temperature, the volatiles were evaporated and the residue purified by column chromatography on silica gel (toluene as eluent): 55 mg, 92% yield; spectral data matched those reported in the literature.[1]

C5-functionalization. Substrate **7** was synthesized according to the general procedure for the radial MCR reaction using *N*-(chloromethyl)phthalimide **1a** (147 mg, 0.75 mmol, 1.5 equiv.), maleimide **2a** (56 mg, 0.5 mmol, 1.0 equiv.) and the C2-functionalized pyrrole **6** (600 mg, 5.0 mmol, 10.0 equiv.). Irradiation time: 24 hours. A single diastereomer was detected by ^{1}H NMR analysis of the crude reaction mixture. Product **7** was purified by column chromatography (gradient from 10% to 40% AcOEt in hexanes as eluent): 61.8 mg of a pale brown solid, 32% yield.



7

4-((1,3-dioxoisoindolin-2-yl)methyl)-1-methyl-2,5-dioxopyrrolidin-3-yl)-1-methyl-1H-pyrrol-2-yl)acetonitrile (7):

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.68 (dd, *J* = 5.5, 3.0 Hz, 2H), 5.74 (d, *J* = 3.8 Hz, 1H), 5.68 (d, *J* = 3.8 Hz, 1H), 4.38 (dd, *J* = 14.0, 5.3 Hz, 1H), 4.12 – 4.01 (m, 2H), 3.61 (ddd, *J* = 10.2, 7.3, 5.2 Hz, 1H), 3.57 (s, 3H), 3.49 (dd, *J* = 5.6, 0.8 Hz, 2H), 3.02 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 175.0, 174.7, 168.3, 134.4, 131.5, 126.9, 123.5, 121.6, 116.2, 108.3, 106.0, 44.7, 43.9, 37.8, 31.0, 25.3, 16.4.

IR (thin film) ν 2936, 1778, 1765, 1697, 1498, 1400, 1274, 1094, 725 cm^{-1}.

HRMS: calculated for $C_{21}H_{18}N_4NaO_4^+$ ($M+Na^+$): 413.1220, found 413.1215.

TLC: 40:60 EtOAc/hexanes, R_f = 0.23.

C.2. Synthesis of Pyrrolidine 8

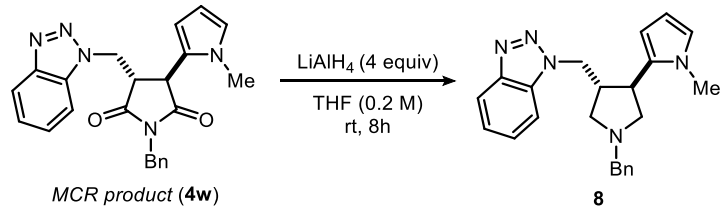
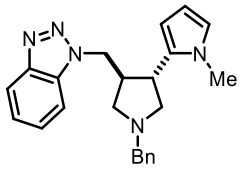


Figure S4. Reduction of the succinimide core of the MCR product **4w**.

A solution of the MCR adduct **4w** (80 mg, 0.2 mmol, 1 equiv.) in dry THF (0.5 mL) was added dropwise to a cooled suspension of $LiAlH_4$ (31 mg, 0.8 mmol, 4 equiv.) in dry THF (0.5 mL) at 0 °C. After completion of the addition, the reaction was stirred at room temperature for 6 h. Complete conversion of **4w** was observed after 6 h, as judged by TLC analysis of the reaction mixture. The reaction mixture was then diluted with Et_2O (1 mL), and quenched successively with water (1 mL) and NaOH (1M, 0.5 mL). The aqueous phase was extracted three times with Et_2O, and the collected organic phase was washed successively with brine, water and dried over magnesium sulfate. The volatiles were evaporated and the residue purified by column chromatography on silica gel (98:2, DCM:MeOH): 68 mg, 92% yield, white solid.



8

1-(((3R,4R)-1-benzyl-4-(1-methyl-1H-pyrrol-2-yl)pyrrolidin-3-yl)methyl)-1H-benzo[d][1,2,3]triazole (8)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (dt, *J* = 8.3, 1.0 Hz, 1H), 7.38 (ddd, *J* = 8.4, 6.7, 1.0 Hz, 1H), 7.34 – 7.19 (m, 8H), 6.47 (dd, *J* = 2.7, 1.8 Hz, 1H), 6.01 (dd, *J* = 3.6, 2.7 Hz, 1H), 5.98 (dd, *J* = 3.6, 1.8 Hz, 1H), 4.71 (s, 1H), 4.70 (d, *J* = 1.7 Hz, 1H), 3.64 (d, *J* = 12.9 Hz, 1H), 3.55 (d, *J* = 12.9 Hz, 1H), 3.24 – 3.13 (m, 2H), 3.06 – 2.96 (m, 1H), 2.76 (dd, *J* = 9.7, 4.7 Hz, 1H), 2.63 (dd, *J* = 9.7, 7.5 Hz, 1H), 2.41 (td, *J* = 7.1, 1.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 146.0, 138.8, 133.7, 133.3, 128.8 (x2C), 128.4 (x2C), 127.4, 127.2, 123.9, 122.3, 120.1, 109.3, 107.0, 105.0, 61.0, 60.1, 56.9, 51.1, 45.1, 39.2, 33.9.

TLC: 2:98 MeOH/DCM, R_f = 0.15.

D. Unsuccessful Substrate Combinations

The optimised conditions of the model MCR reaction were evaluated with a variety of substrates. Those shown in Figure S5 failed to deliver yields higher than the catalyst loading or any product at all.

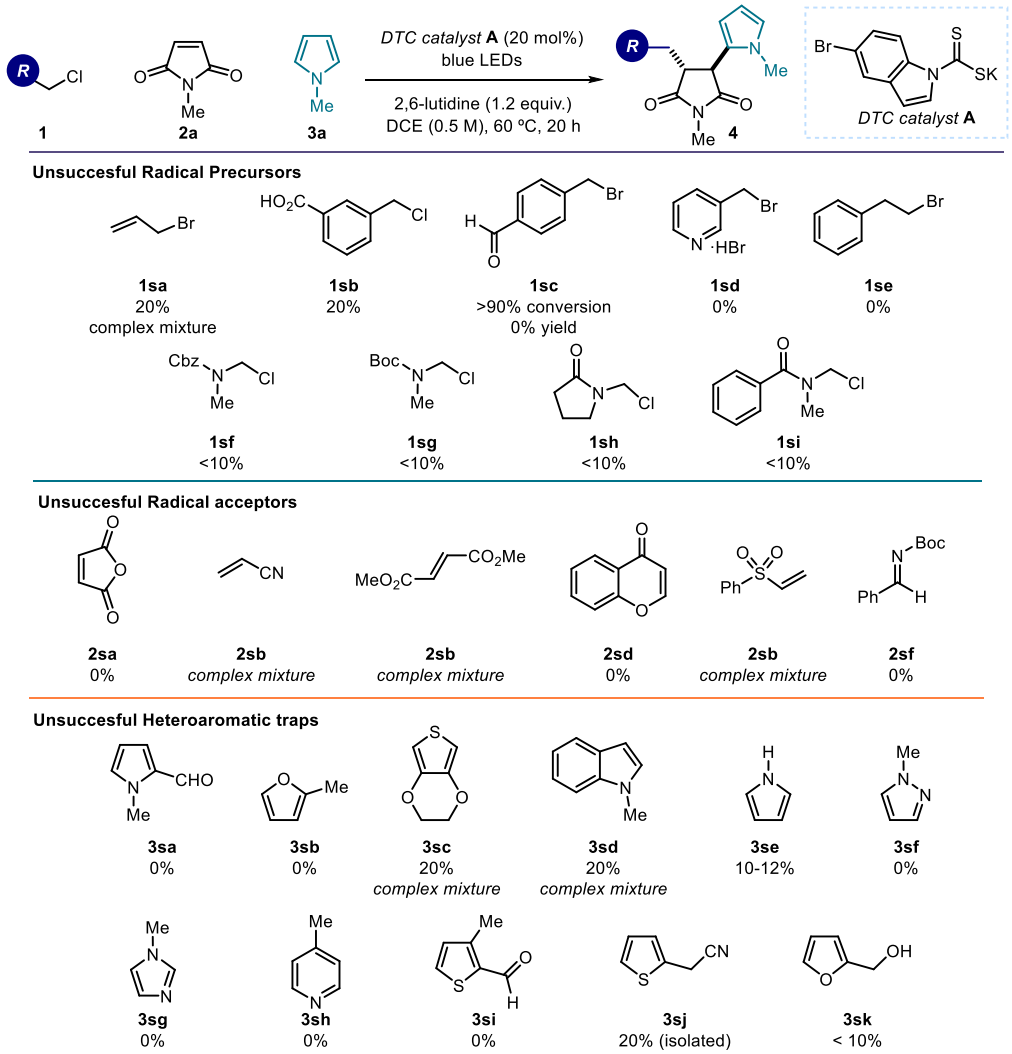


Figure S5. Substrates that failed to provide synthetically useful yields of MCR products. Unless otherwise noted, yields were determined by ^{1}H NMR analysis of the crude mixture using trichloroethylene as the internal standard.

E. Cyclic Voltammetry Measurements

Substrates **1a**, **1b**, **1d**, **1e, 1f, 1g**, **1k**, **1n**, **1o**, **1r** and **1s** were electrochemically characterized. The measured reduction potential values are compiled in Figure S6. The cyclic voltammograms are shown in Figures S7-S17.

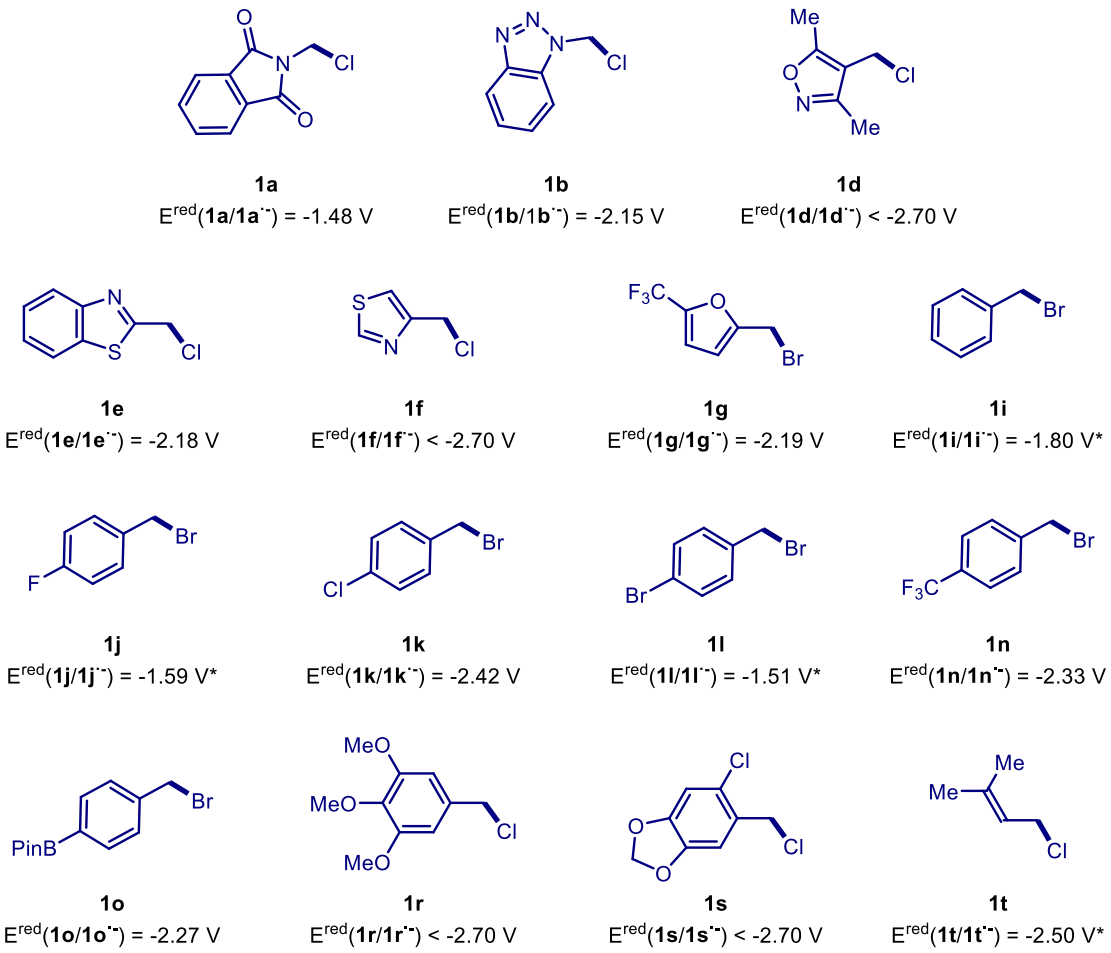


Figure S6. Reduction potentials (E^{red}) measured vs. Ag/AgCl (KCl, 3.5 M). *Reported in literature and referenced to Ag/AgCl (KCl saturated), according to reference 6.

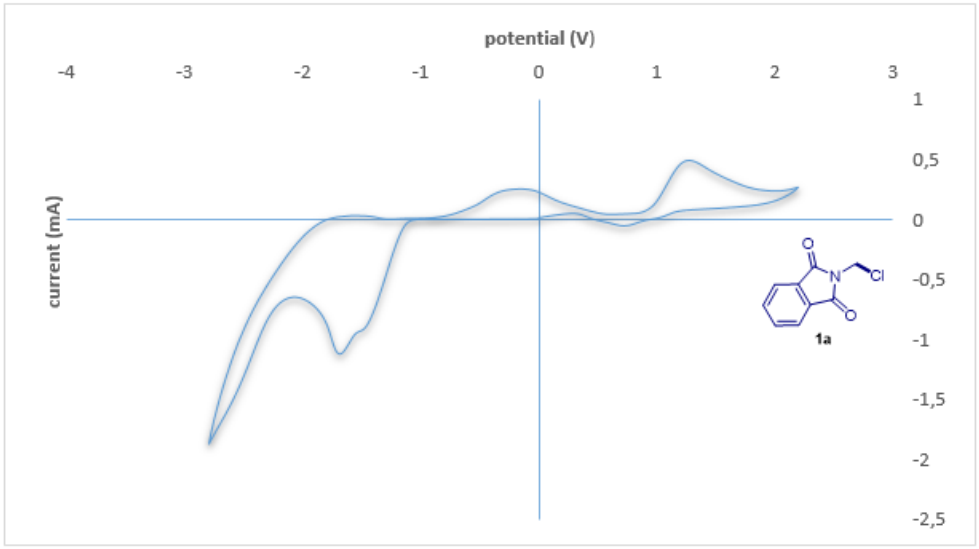


Figure S7. Cyclic voltammogram for *N*-(chloromethyl)phtalamide chloride **1a** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 100 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Irreversible reduction, $E_p^C = E^{red}$(**1a**/**1a**$^{\cdot-}$) = -1.48 V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1a**.

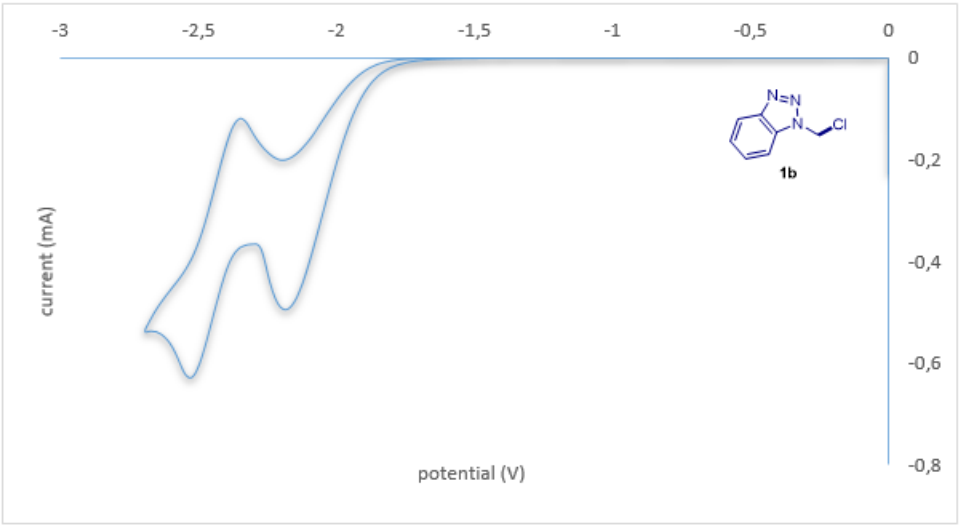


Figure S8. Cyclic voltammogram for 1-(chloromethyl)-1H-benzotriazole **1b** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 100 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. First irreversible reduction, $E_p^C = E^{red}$(**1b**/**1b**$^{\cdot-}$) = -2.15 V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1b**.

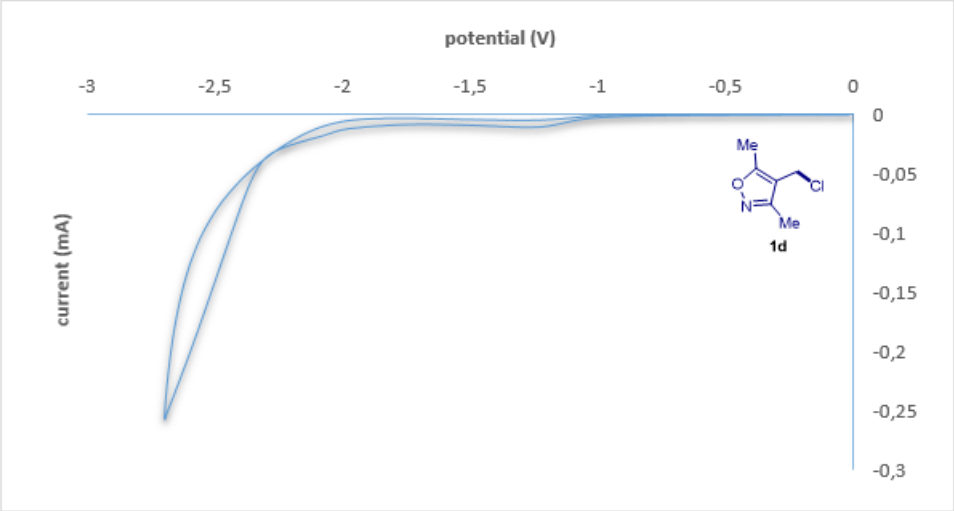


Figure S9. Cyclic voltammogram for 4-(chloromethyl)3,5-dimehtylisoxazole chloride **1d** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 50 mV/s. Glassy carbon electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Reduction of substrate **1d** was not observed in the registered potential window (from 0 to -2.70 V).

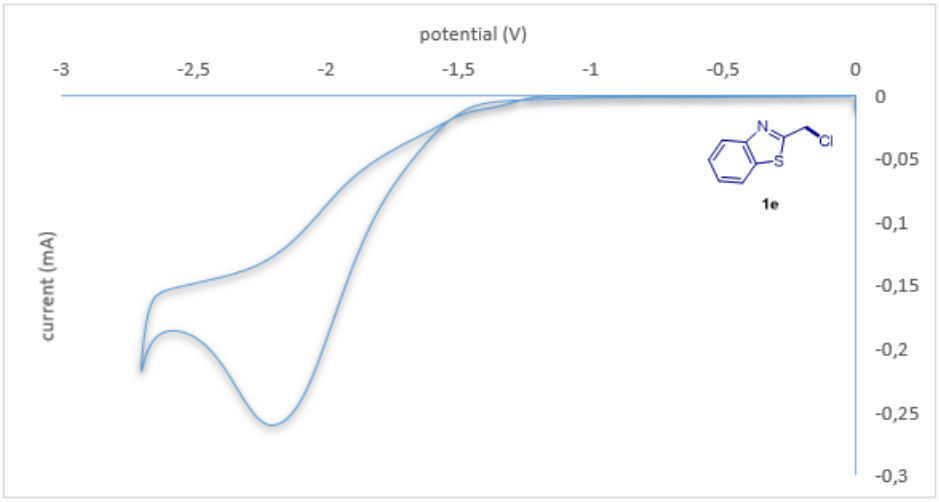


Figure S10. Cyclic voltammogram for 2-(chloromethyl)-1,3-benzothiazole chloride **1e** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 50 mV/s. Glassy carbon electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Irreversible reduction, $E_p^C = E^{red}$(**1e**/**1e**$^{\cdot-}$) = -2.18 V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1e**.

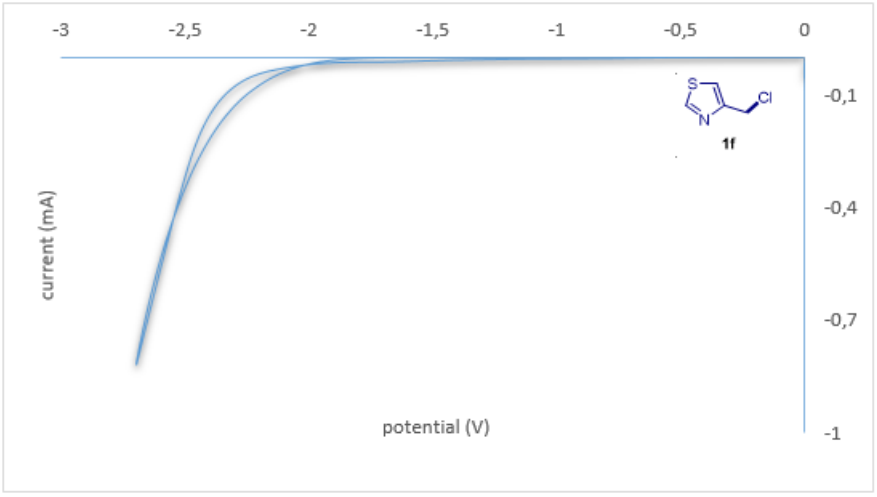


Figure S11. Cyclic voltammogram for 4-(chloromethyl)thiazole **1f** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 500 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Reduction of substrate **1f** was not observed in the registered potential window (from 0 to -2.70 V).

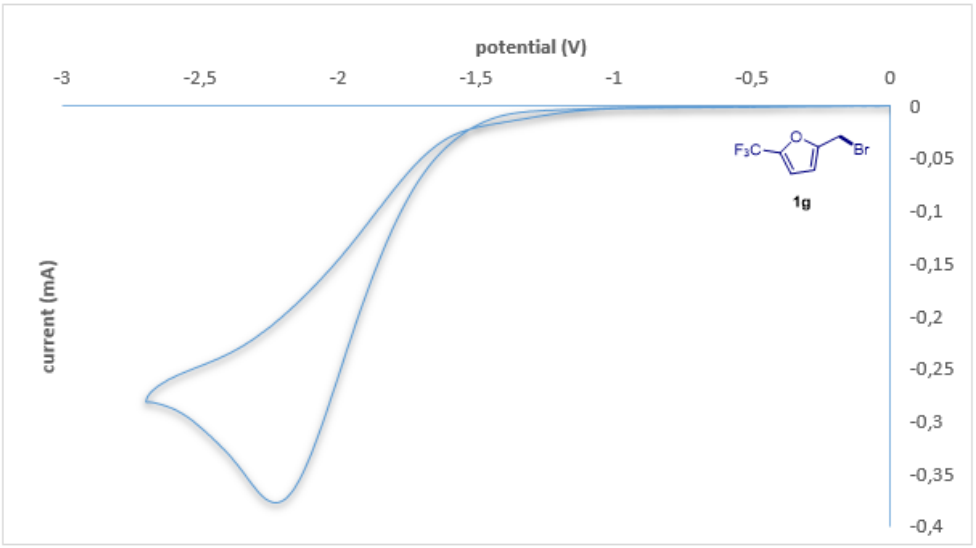


Figure S12. Cyclic voltammogram for 2-(bromomethyl)-5-(trifluoromethyl)furan bromide **1g** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 50 mV/s. Glassy carbon electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Irreversible reduction, $E_p^C = E^{red}(\mathbf{1g}/\mathbf{1g}^{\cdot-})$ = -2.19 V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1g**.

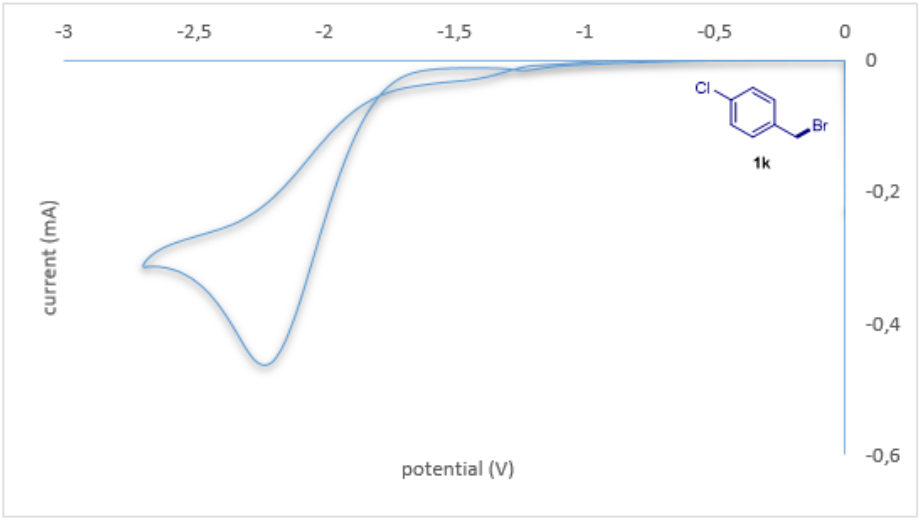


Figure S13. Cyclic voltammogram for 4-chlorobenzyl bromide **1k** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 500 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Irreversible reduction, $E_p^C = E^{red}$(**1k**/**1k**$^{\cdot-}$) = -2.42 V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1k**.

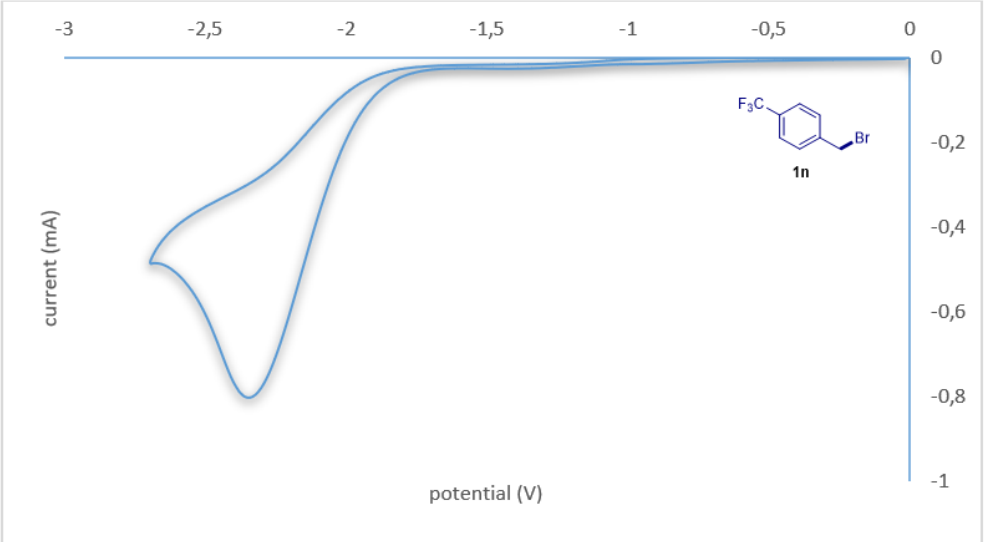


Figure S14. Cyclic voltammogram for 4-(trifluoromethyl)benzyl bromide **1n** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 500 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Irreversible reduction, $E_p^C = E^{red}$(**1n**/**1n**$^{\cdot-}$) = -2.33 V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1n**.

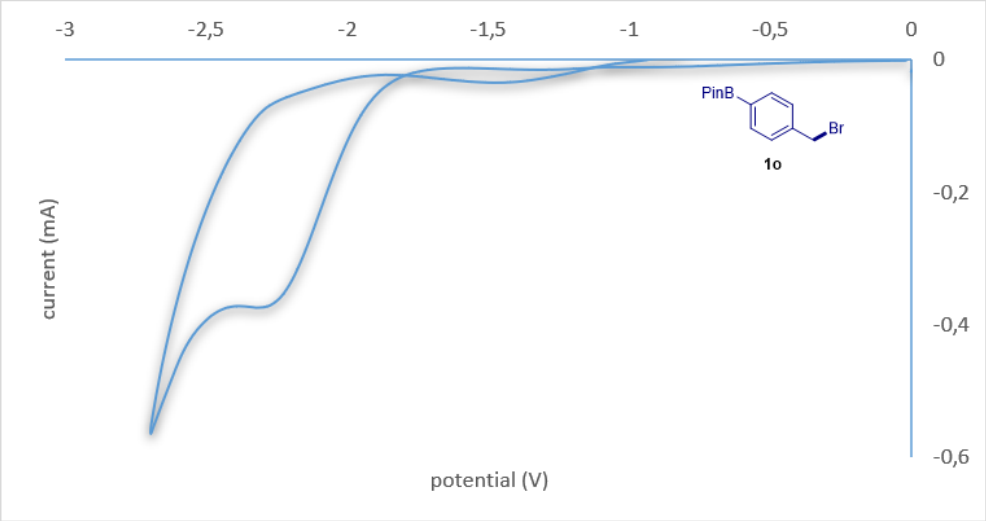


Figure S15. Cyclic voltammogram for 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl bromide **1o** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 500 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Irreversible reduction, $E_p^C = E^{red}(\mathbf{1o}/\mathbf{1o}^{\cdot-}) = -2.27$ V, E_p^C refers to the cathodic peak potential, while the E^{red} value describes the electrochemical properties of **1o.**

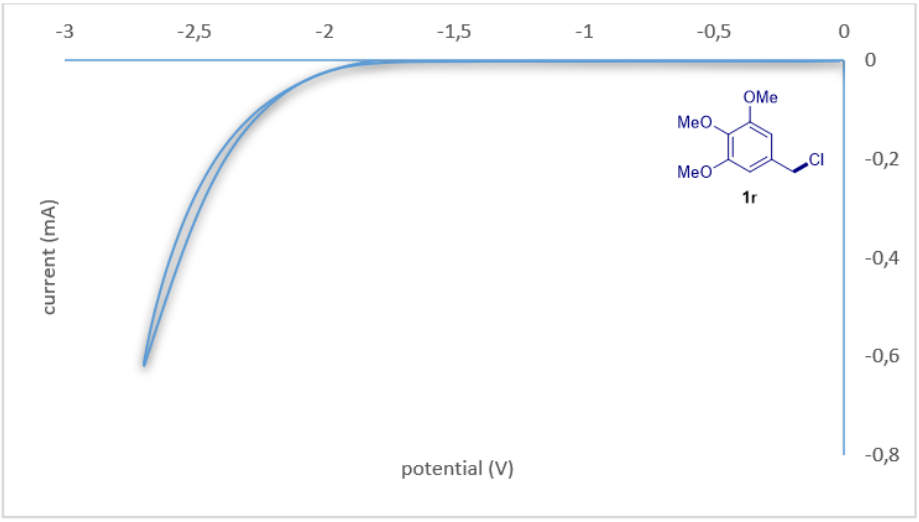


Figure S16. Cyclic voltammogram for 5-(chloromethyl)-1,2,3-trimethoxybenzene **1r** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 100 mV/s. Pt electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Reduction of substrate **1r** was not observed in the registered potential window (from 0 to -2.70 V).

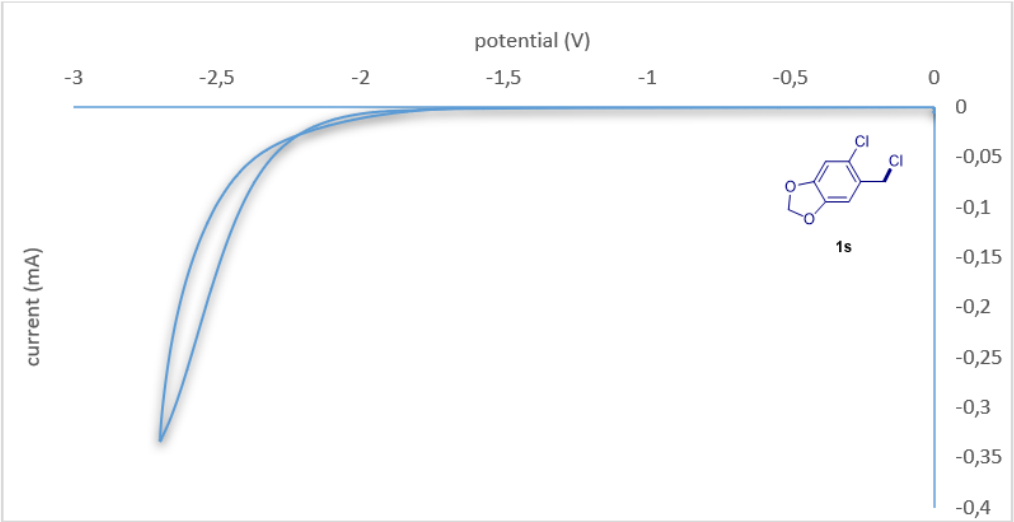


Figure S17. Cyclic voltammogram for 6-chloropiperonyl chloride **1s** [0.02M] in [0.1 M] $TBAPF_6$ in CH_3CN. Sweep rate: 50 mV/s. Glassy carbon electrode working electrode, Ag/AgCl (KCl 3.5 M) reference electrode, Pt wire auxiliary electrode. Reduction of substrate **1s** was not observed in the registered potential window (from 0 to -2.70 V).

The reduction potential of substrates **1i**, **1j**, **1l**, **1t** are reported in literature[3,4,5]. The literature values were referenced to Ag/AgCl. KCl saturated[6], and are the following:

E^{red}(**1i**/**1i**$^{\bullet-}$) = -1.80 V (vs. Ag/AgCl, KCl saturated)

E^{red}(**1j**/**1j**$^{\bullet-}$) = -1.59 V (vs. Ag/AgCl, KCl saturated)

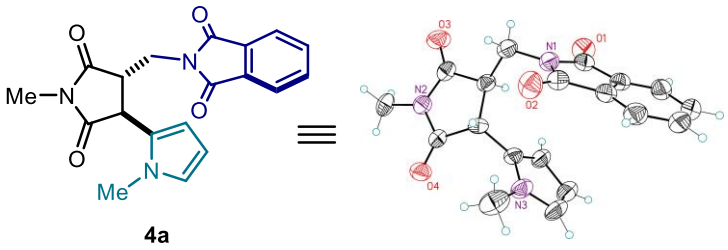
E^{red}(**1l**/**1l**$^{\bullet-}$) = -1.51 V (vs. Ag/AgCl, KCl saturated)

E^{red}(**1t**/**1t**$^{\bullet-}$) = -2.50 V (vs. Ag/AgCl, KCl saturated)

F. X-ray Crystallographic Data

Single Crystal X-ray Diffraction Data for Compound 4a

Crystals of the compound **4a** were obtained by slow evaporation of a methanol solution.
Data Collection. Measurements were made on a Bruker-Nonius diffractometer equipped with an APPEX 2 4K CCD area detector, a FR591 rotating anode with MoKα radiation, Montel mirrors and a Cryostream Plus low temperature device (*T* = 100K). Full-sphere data collection was used with ω and φ scans.



4a CCDC 1894404

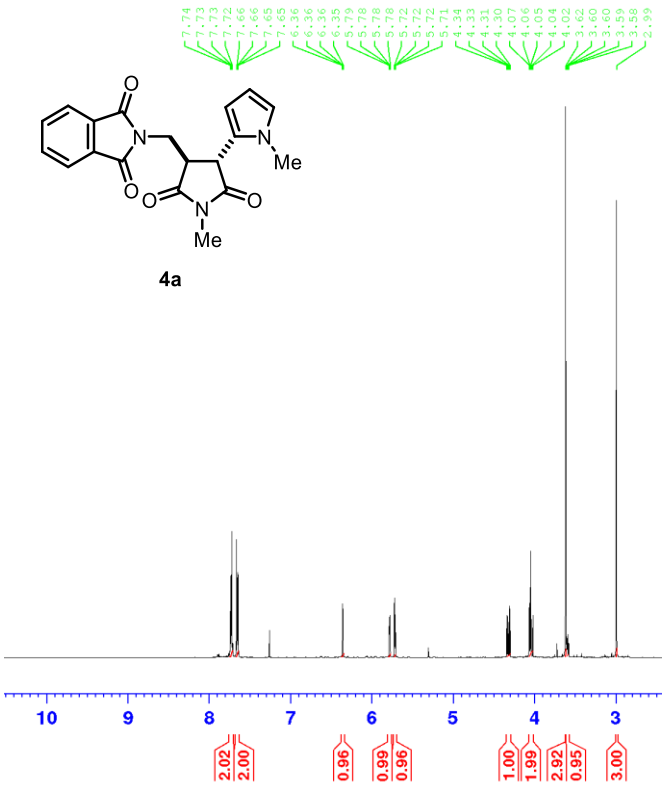
Table S1. Crystal data and structure refinement for **4a**. ***CCDC 1894404***

Empirical formula	C19 H17 N3 O4	
Formula weight	351.35	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 10.5086(4)Å	α= 90°.
b = 12.0160(4)Å	β = 90°.	
c = 27.2839(11)Å	γ = 90°.	
Volume	3445.2(2) $Å^3$	
Z	8	
Density (calculated)	1.355 Mg/m^3	
Absorption coefficient	0.097 mm^{-1}	
F(000)	1472	
Crystal size	0.20 x 0.10 x 0.10 mm^3	
Theta range for data collection	2.446 to 27.116°.	
Index ranges	-8<=h<=13,-15<=k<=13,-35<=l<=35	
Reflections collected	27430	
Independent reflections	3774[R(int) = 0.0358]	
Completeness to theta =27.116°	99.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.990 and 0.898	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3774/ 461/ 365	
Goodness-of-fit on F^2	1.078	
Final R indices [I>2sigma(I)]	R1 = 0.0422, wR2 = 0.1126	
R indices (all data)	R1 = 0.0564, wR2 = 0.1208	
Largest diff. peak and hole	0.179 and -0.205 $e.Å^{-3}$	

G. References

1. Schweitzer-Chaput, B.; Horwitz, M. A.; de Pedro Beato, E.; Melchiorre, P. Photochemical Generation of Radicals from Alkyl Electrophiles Using a Nucleophilic Organic Catalyst. *Nat. Chem.* **2019**, *11*, 129–135.
2. Maksymenko, S.; Parida, K. N.; Pathe, G. K.; More, A. A.; Lipisa, Y. B.; Szpilman, A. M. Transition-Metal-Free Intermolecular α-Arylation of Ketones via Enolonium Species. *Org. Lett.* **2017**, *19*, 6312–6315.
3. Shih, H-W.; Vander Wal, M. N.; Grange, R. L.; MacMillan, D. W. C. Enantioselective α-Benzylation of Aldehydes via Photoredox Organocatalysis. *J. Am. Chem. Soc.* **2010**, *132*, 13600–13603.
4. Zhang, X-M. *J. Chem. Soc., Perkin Trans. 2*, **1993**, 2275–2279.
5. (a) Pavlishchuk, V. V.; Addison, A. W. *Inorganica Chim. Acta* **2000**, *298*, 97–102. (b) "*Handbook of Analytical Chemistry*", L Meites, ed., McGraw Hill, NY (1963).

H. ^{1}H and ^{13}C NMR Spectra



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RG                  80.6
DW                50.000 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

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PL1                 3.00 dB
PL1W          12.58925438 W
SFO1          500.1330008 MHz

F2 - Processing parameters
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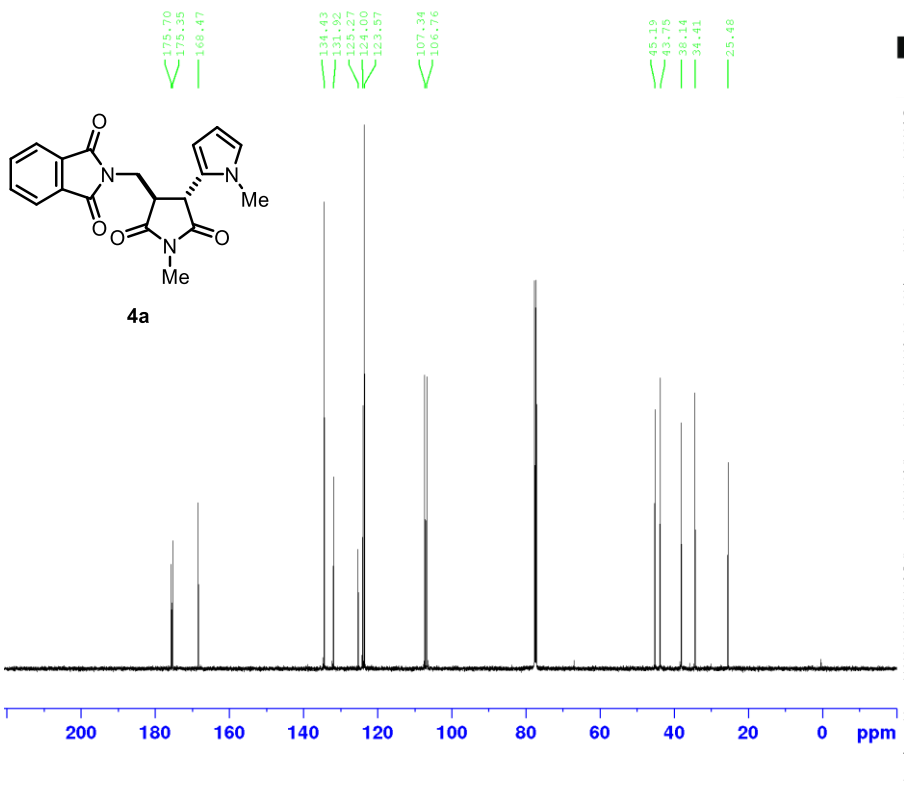

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PL1          0 dB
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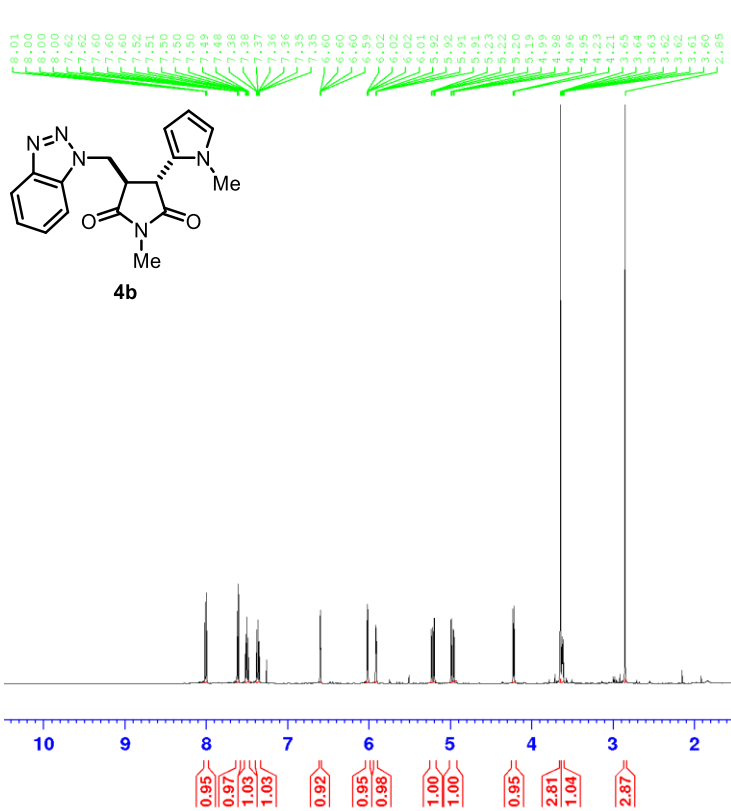

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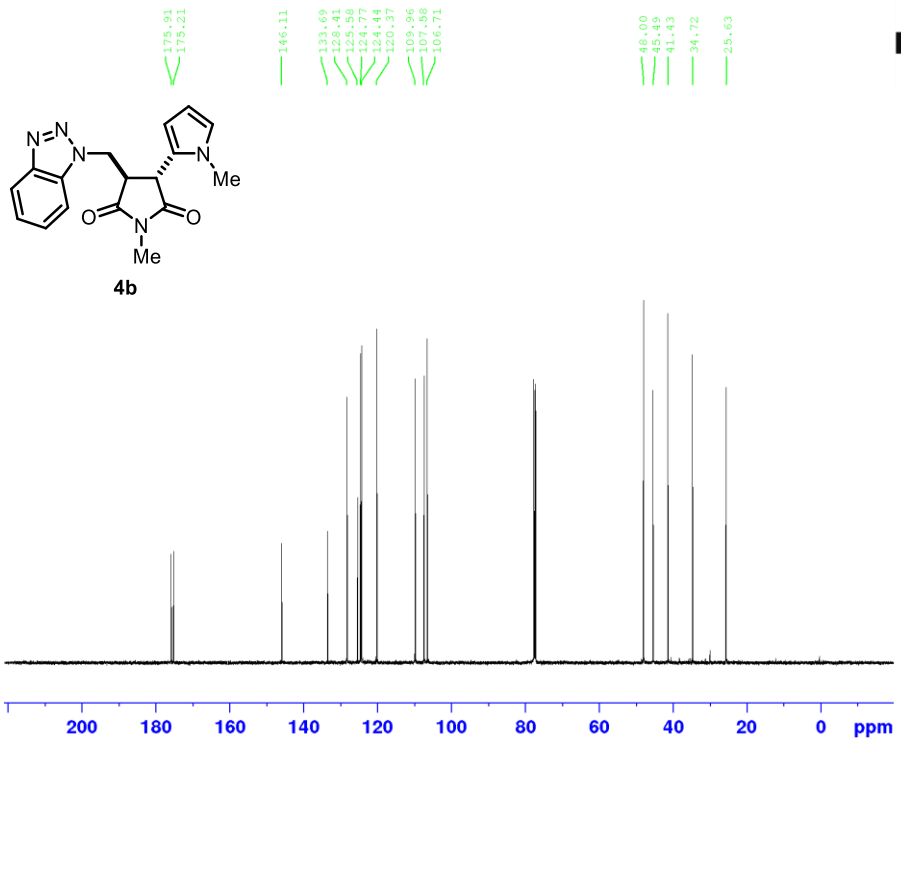

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D11             0.03000000 sec
TD0                    1

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PL1       0 dB
PL1W          63.80191422 W
SFO1          125.7703648 MHz

======== CHANNEL f2 ========
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PCPD2              80.00 usec
PL2                3.00 dB
PL12               18.58 dB
PL13               18.58 dB
PL2W          12.58925438 W
PL12W          0.34833732 W
PL13W          0.34833732 W
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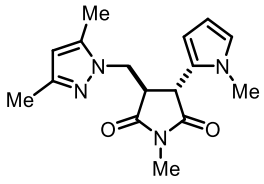

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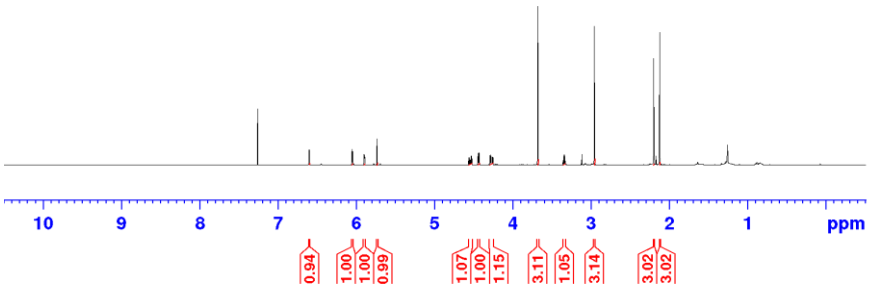

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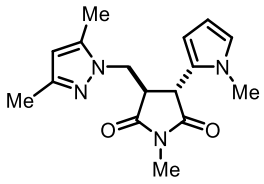
PROBHD    5 mm PATBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                  362
DW               83.200 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -4.08 dB
PL1W        35.02648163 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                   16384
SF           500.1300130 MHz
WDW                     EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



4c

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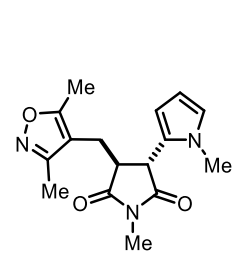
```
Current Data Parameters
NAME      sch998-3FC-final
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20181004
Time               1.34
INSTRUM           spect
PROBHD    5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2000
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                11500
DW               16.500 usec
DE               10.00 usec
TE                299.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.50 usec
PL1          0 dB
PL1W        63.80191422 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.62 dB
PL13              13.62 dB
PL2W        39.81071854 W
PL12W        1.09144032 W
PL13W        1.09144032 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF           125.7577730 MHz
WDW                     EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4d

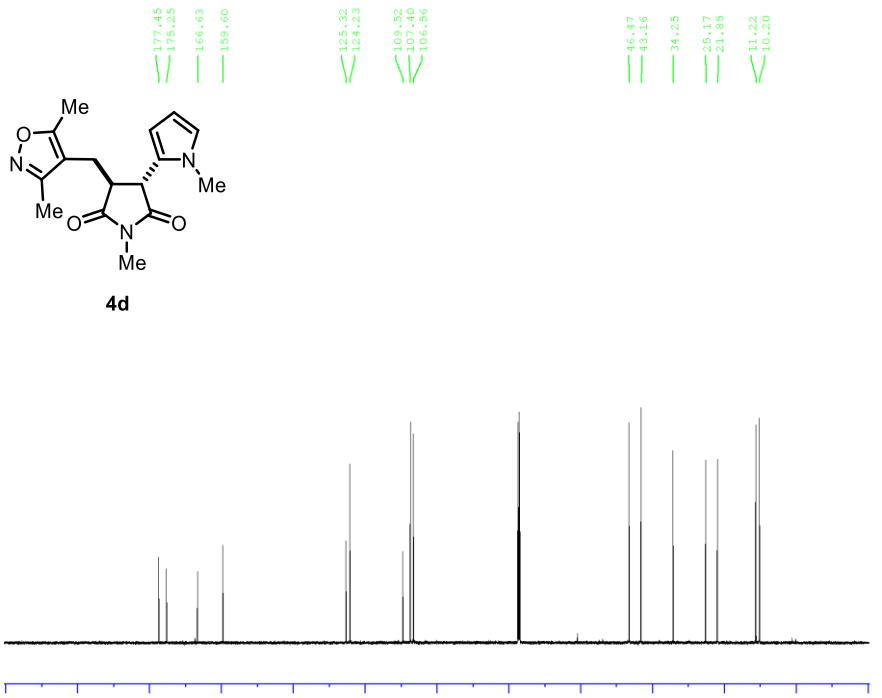
BRUKER

```
Current Data Parameters
NAME         sch1036-ch1
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181108
Time               18.41
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                  71.8
DW                83.200 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -4.08 dB
PL1W         35.02648163 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF           500.1300130 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



4d

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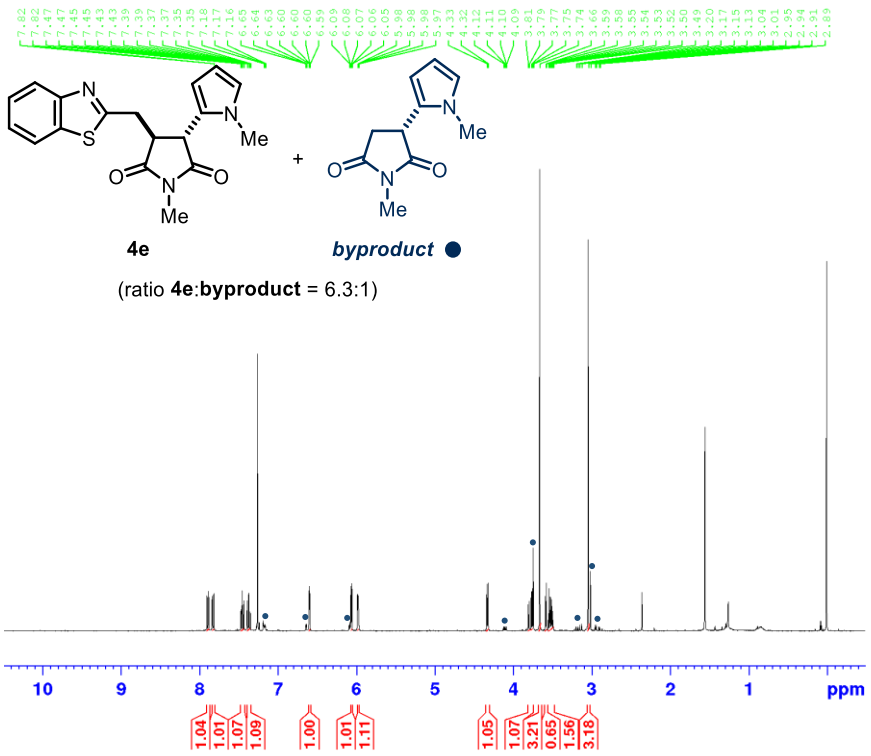
```
Current Data Parameters
NAME       sch1036-ch1-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181109
Time               7.31
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 11500
DW                16.500 usec
DE                10.00 usec
TE                298.2 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.25 usec
PL1        0 dB
PL1W         53.75436783 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.08 dB
PL12               11.54 dB
PL13               11.54 dB
PL2W         35.02648163 W
PL12W         0.96027696 W
PL13W         0.96027696 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577823 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

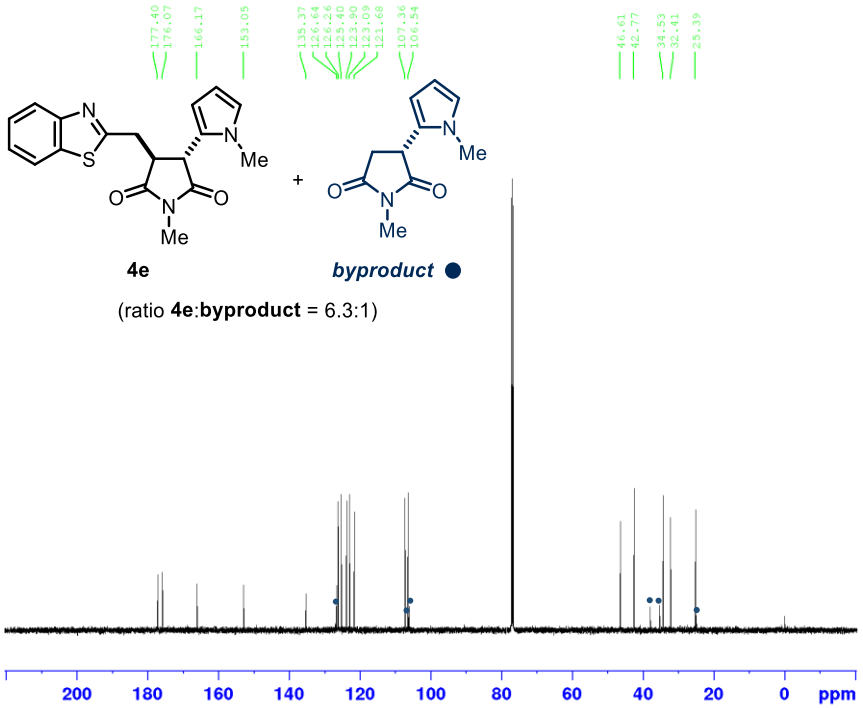



```
Current Data Parameters
NAME      sch1045-2fc-is
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181120
Time               11.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                   362
DW              104.400 usec
DE                6.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



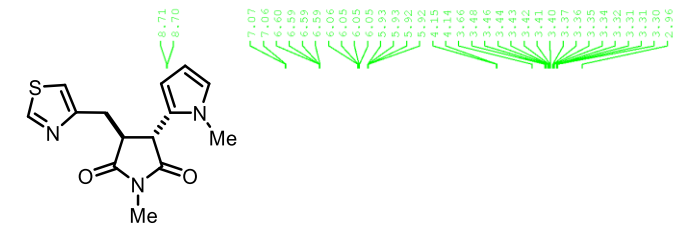

```
Current Data Parameters
NAME      sch1045_1fc-is-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181117
Time               15.03
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 11500
DW                16.500 usec
DE                10.00 usec
TE                298.4 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

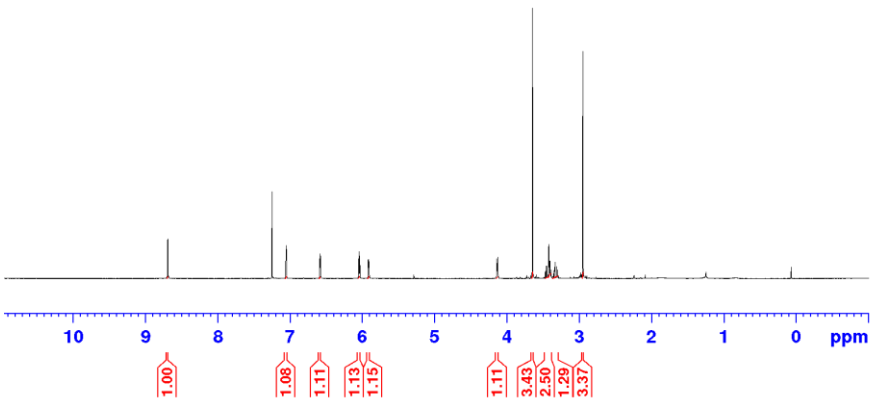
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.25 usec
PL1       0 dB
PL1W         53.75436783 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.08 dB
PL12               11.54 dB
PL13               11.54 dB
PL2W         35.02648163 W
PL12W         0.96027696 W
PL13W         0.96027696 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577742 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4f



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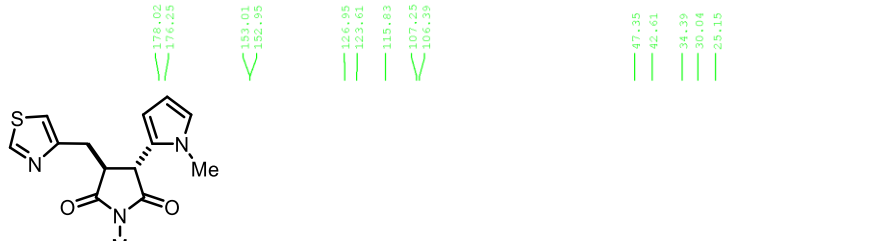
```
Current Data Parameters
NAME         sch1037-rqs
EXPNO                 10
PROCNO                 1

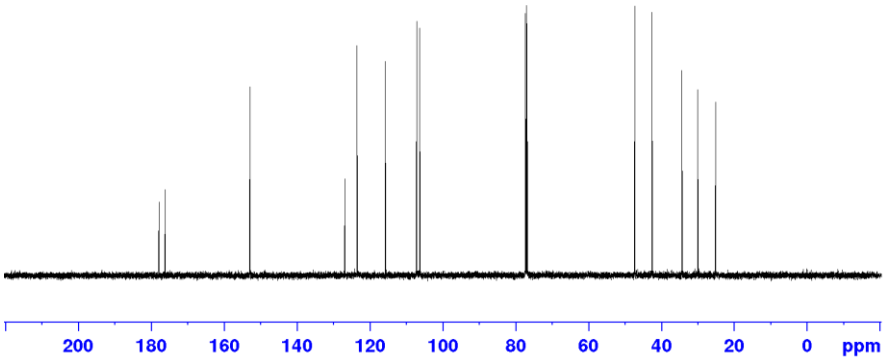
F2 - Acquisition Parameters
Date_           20190130
Time               13.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                  362
DW             104.400 usec
DE               6.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300098 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



4f



BRUKER

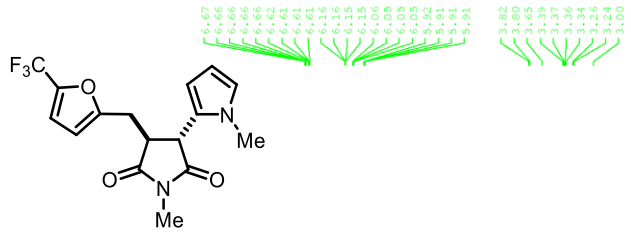
```
Current Data Parameters
NAME       sch1037-ch1-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181109
Time               7.42
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  117
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                13000
DW              16.500 usec
DE              10.00 usec
TE               298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

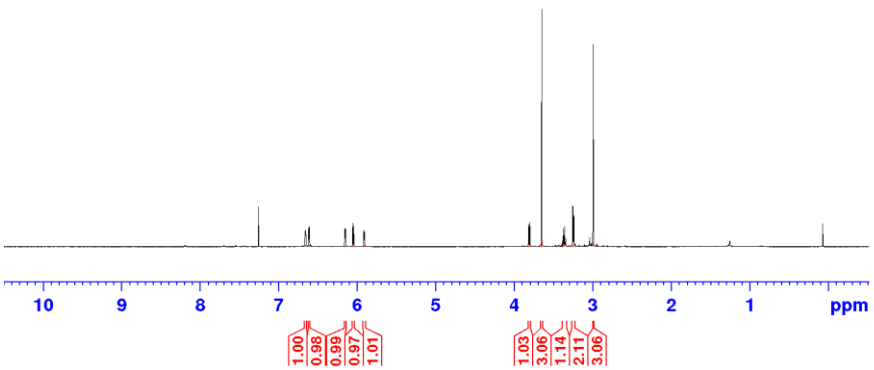
======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1       0 dB
PL1W       53.75436783 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.08 dB
PL12              11.54 dB
PL13              11.54 dB
PL2W        35.02648163 W
PL12W        0.96027696 W
PL13W        0.96027696 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577820 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



4g



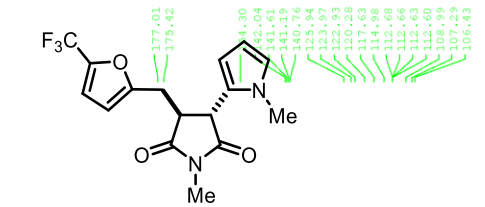
BRUKER

```
Current Data Parameters
NAME         sch1040-rqs
EXPNO                 10
PROCNO                 1

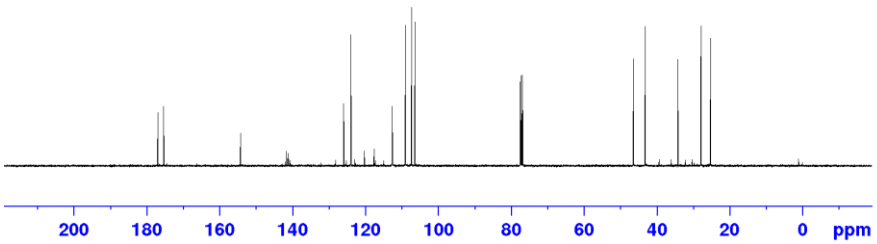
F2 - Acquisition Parameters
Date_           20190130
Time              13.09
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           4789.272 Hz
FIDRES         0.146157 Hz
AQ            3.4209793 sec
RG                 181
DW              104.400 usec
DE                6.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               14.50 usec
PL1              -2.00 dB
PL1W        23.88643074 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300096 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                1.00
```



4g



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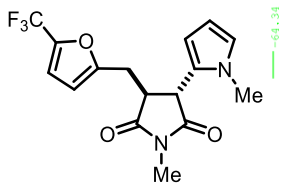
```
Current Data Parameters
NAME      sch1040-fc2-HV-C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181117
Time              1.24
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664256 sec
RG              23170.5
DW               20.850 usec
DE               10.00 usec
TE               298.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                  1

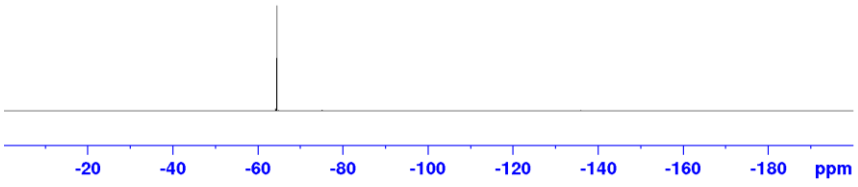
======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PL1              -3.00 dB
PL1W        75.17808533 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             12.83 dB
PL13             12.83 dB
PL2W        23.88643074 W
PL12W        0.78550917 W
PL13W        0.78550917 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127609 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



4g



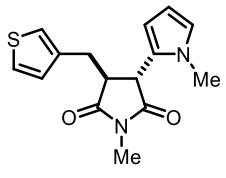

```
Current Data Parameters
NAME      sch1040-rqst-F
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190201
Time              19.04
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD              131072
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           75187.969 Hz
FIDRES        0.573639 Hz
AQ            0.8716288 sec
RG                 2048
DW                6.650 usec
DE                7.14 usec
TE                298.1 K
D1           5.00000000 sec
D11          0.03000000 sec
TD0                   1

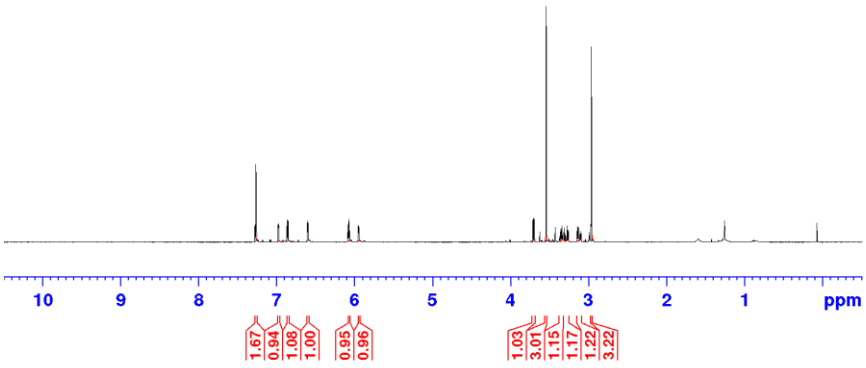
======== CHANNEL f1 ========
NUC1                 19F
P1                12.00 usec
PL1               -3.00 dB
SFO1        376.4607040 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              12.83 dB
PL13              12.83 dB
PL2W        23.88643074 W
PL12W        0.78550917 W
PL13W        0.78550917 W
SFO2        400.1320010 MHz

F2 - Processing parameters
SI                65536
SF          376.4984036 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```



4h

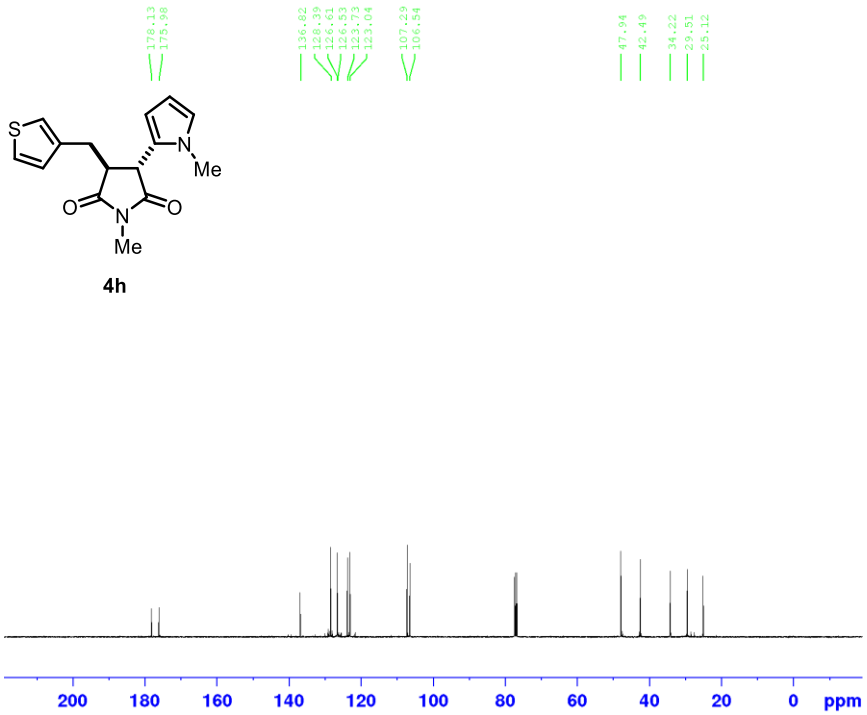



```
Current Data Parameters
NAME       sch1042-rqst
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time              16.36
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                  256
DW              104.400 usec
DE                6.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300097 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



4h

BRUKER

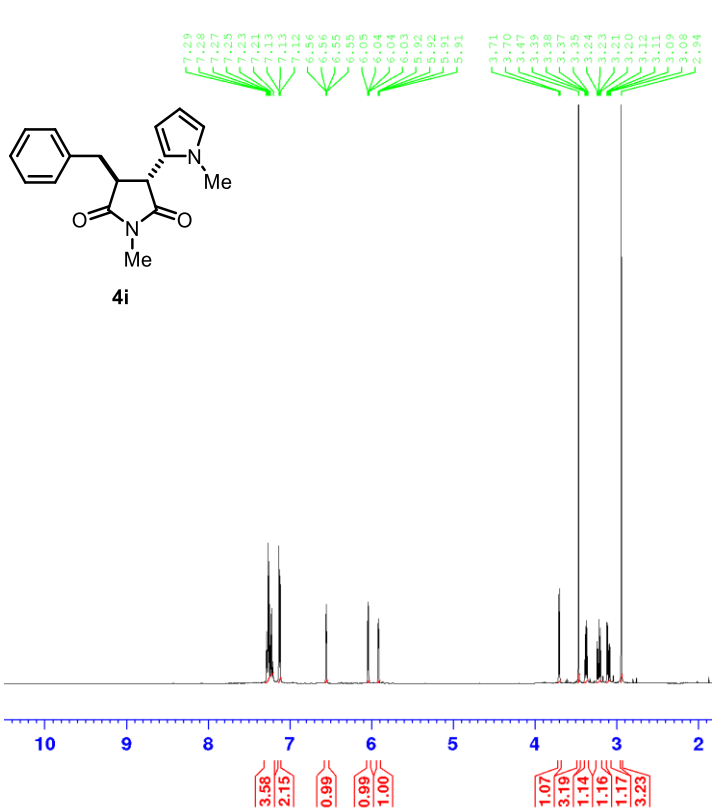
```
Current Data Parameters
NAME      sch1042-fc2-HV-C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20181117
Time                1.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664256 sec
RG              20642.5
DW               20.850 usec
DE               10.00 usec
TE               298.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W        75.17808533 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               12.83 dB
PL13               12.83 dB
PL2W        23.88643074 W
PL12W        0.78550917 W
PL13W        0.78550917 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127649 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



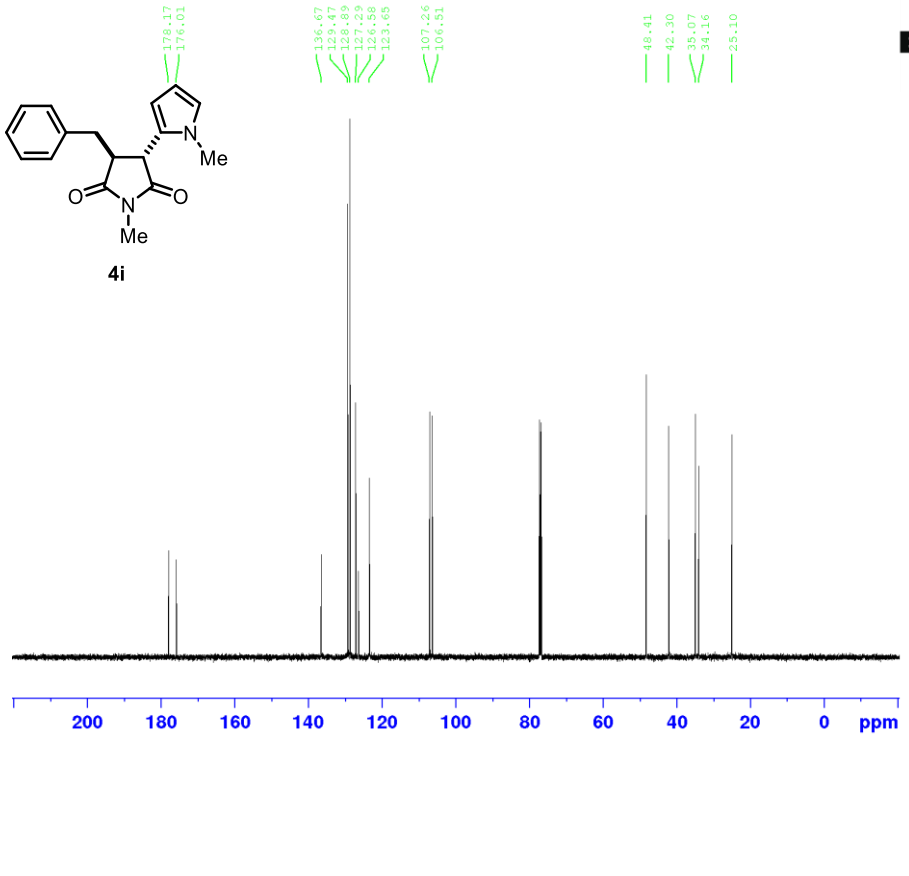
4i

```
Current Data Parameters
NAME       mah-337-1H-re
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20181008
Time              15.50
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ            3.2767999 sec
RG                  64
DW               50.000 usec
DE               10.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               13.25 usec
PL1              -2.00 dB
PL1W        39.81071854 W
SFO1        500.1330008 MHz

F2 - Processing parameters
SI                32768
SF          500.1300210 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



4i

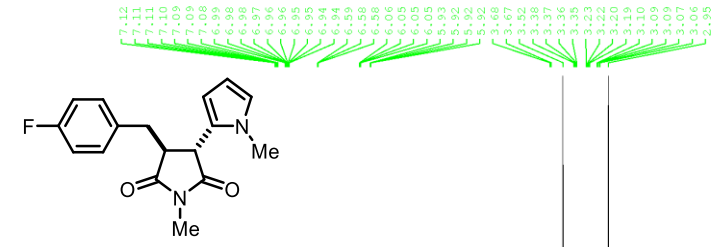
```
Current Data Parameters
NAME       mah-337-13C-re
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20181008
Time              15.55
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 128
DS                   4
SWH          30303.031 Hz
FIDRES           0.462388 Hz
AQ            1.0813440 sec
RG                6500
DW               16.500 usec
DE               10.00 usec
TE               299.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.50 usec
PL1       0 dB
PL1W        63.80191422 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             13.62 dB
PL13             13.62 dB
PL2W        39.81071854 W
PL12W        1.09144032 W
PL13W        1.09144032 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577822 MHz
WDW                  EM
SSB       0
LB               1.00 Hz
GB        0
PC                1.40
```



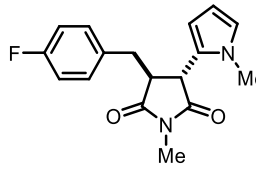
4j

```
Current Data Parameters
NAME      sch1010-is-fc2
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181020
Time                4.58
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                   144
DW                83.200 usec
DE                10.00 usec
TE                297.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.25 usec
PL1                -2.00 dB
PL1W         39.81071854 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF           500.1300142 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



4j

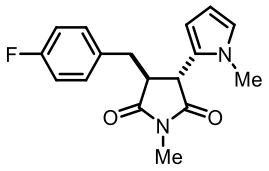
```
Current Data Parameters
NAME      sch1010-is-fc2
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181020
Time                5.35
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 13000
DW                16.500 usec
DE                10.00 usec
TE                299.8 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1        0 dB
PL1W         63.80191422 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.62 dB
PL13               13.62 dB
PL2W         39.81071854 W
PL12W         1.09144032 W
PL13W         1.09144032 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577780 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```



4j

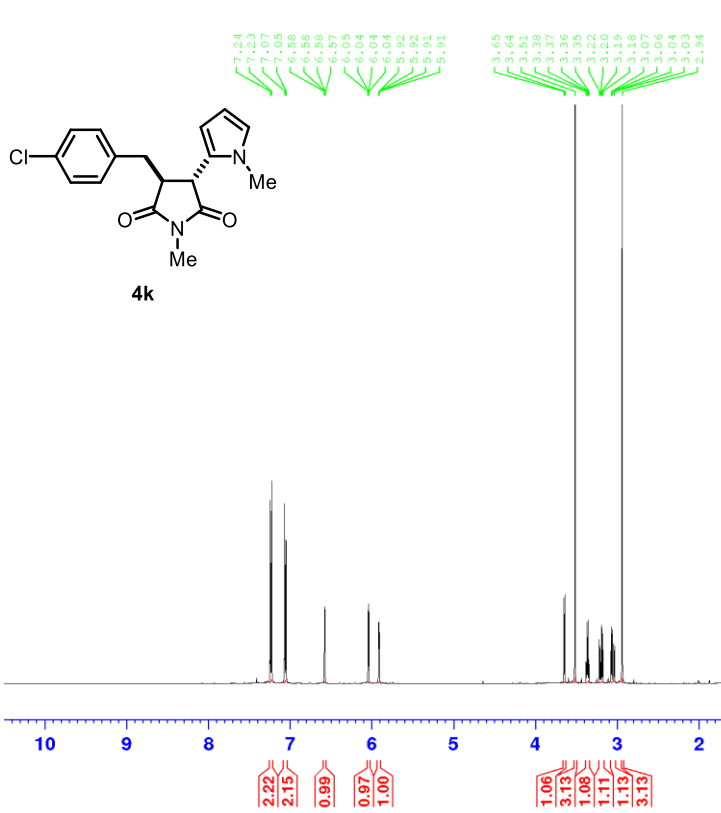

```
Current Data Parameters
NAME      sch1010-is-fc2
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181020
Time                5.37
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG  ICIQ_zgfhigqn.2
TD                131072
SOLVENT             CDC13
NS                      8
DS                      2
SWH          100000.000 Hz
FIDRES         0.762939 Hz
AQ             0.6553600 sec
RG                    406
DW                5.000 usec
DE                 6.50 usec
TE                298.7 K
D1            5.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  19F
P1                 18.75 usec
PL1                 1.50 dB
SFO1         470.5453180 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.62 dB
PL2W         39.81071854 W
PL12W         1.09144032 W
SFO2         500.1325007 MHz

F2 - Processing parameters
SI                    65536
SF           470.5924245 MHz
WDW                     no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



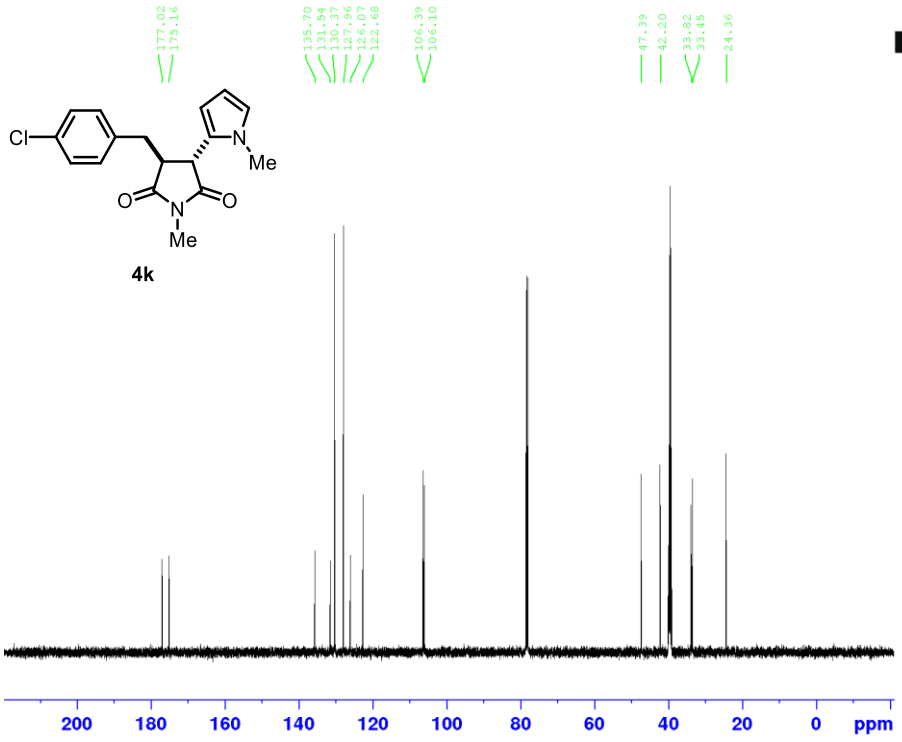
BRUKER

```
Current Data Parameters
NAME                  mah-342-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20181016
Time                      21.34
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           32
DS                            2
SWH                 10000.000 Hz
FIDRES                 0.152588 Hz
AQ                  3.2767999 sec
RG                          128
DW                     50.000 usec
DE                     10.00 usec
TE                     297.9 K
D1                  1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        13.25 usec
PL1                       -2.00 dB
PL1W                39.81071854 W
SFO1                500.1330008 MHz

F2 - Processing parameters
SI                         32768
SF                  500.1300210 MHz
WDW                           EM
SSB                 0
LB                         0.30 Hz
GB                  0
PC                         1.00
```



BRUKER

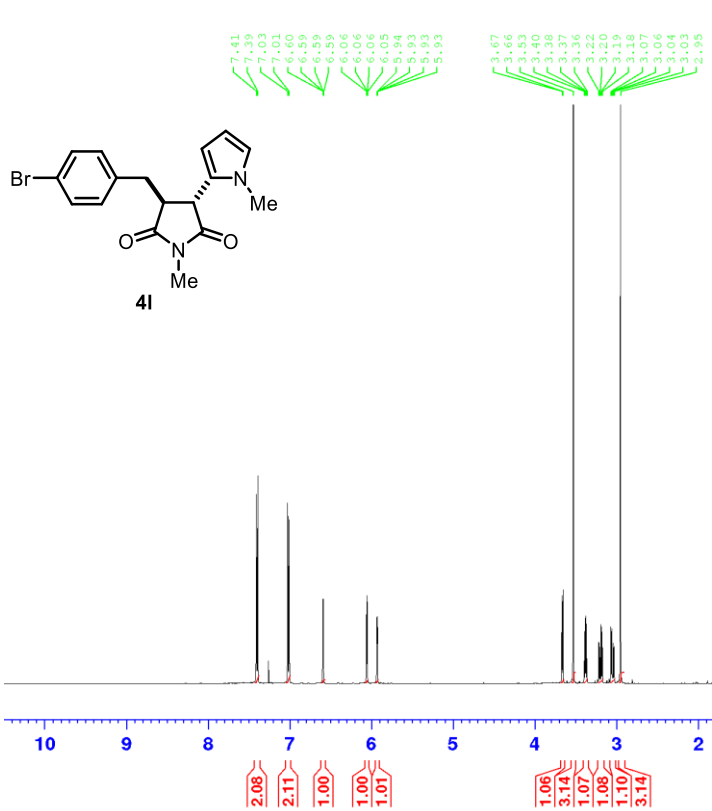
```
Current Data Parameters
NAME      mah-342-13C-cdcl3anddmso
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20181017
Time                      15.07
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                    DMSO
NS                           177
DS                             4
SWH                 30303.031 Hz
FIDRES                 0.462388 Hz
AQ                  1.0813440 sec
RG                         10300
DW                     16.500 usec
DE                     10.00 usec
TE                     299.2 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          13C
P1                         9.50 usec
PL1                 0 dB
PL1W                63.80191422 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                       -2.00 dB
PL12                      13.62 dB
PL13                      13.62 dB
PL2W                39.81071854 W
PL12W                1.09144032 W
PL13W                1.09144032 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7578685 MHz
WDW                           EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
```



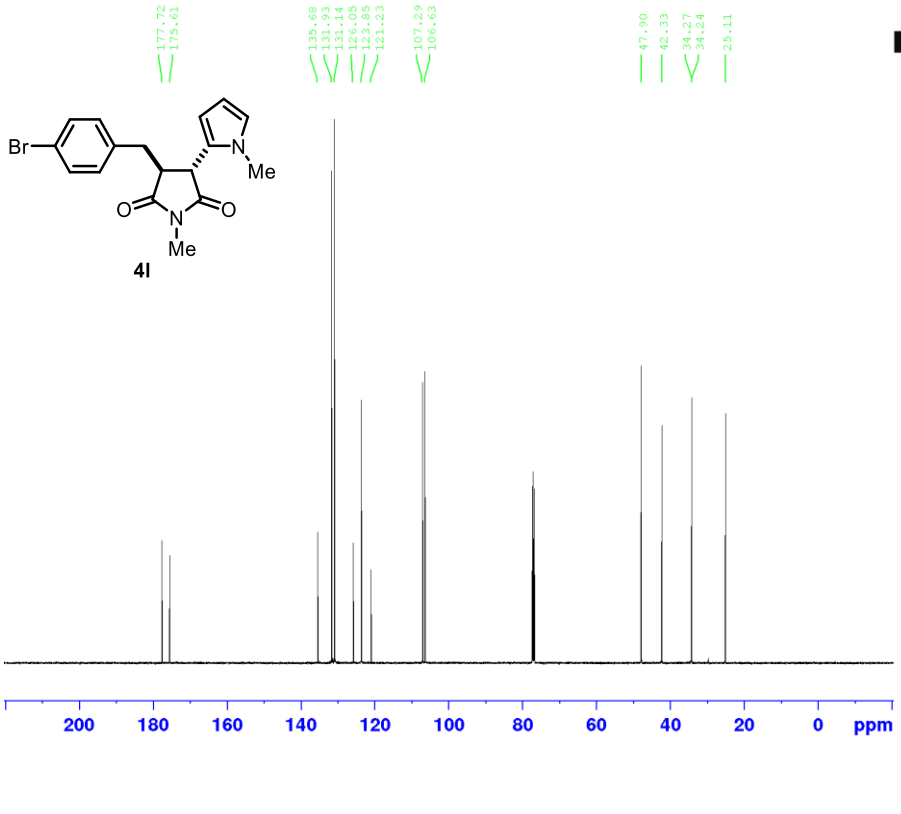
BRUKER

```
Current Data Parameters
NAME                  mah-341-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20181016
Time                       2.36
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                   zg30
TD                        65536
SOLVENT                   CDCl3
NS                           32
DS                            2
SWH                 10000.000 Hz
FIDRES                 0.152588 Hz
AQ                  3.2767999 sec
RG                           57
DW                     50.000 usec
DE                     10.00 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                     13.25 usec
PL1                     -2.00 dB
PL1W                39.81071854 W
SFO1                500.1330008 MHz

F2 - Processing parameters
SI                        32768
SF                  500.1300130 MHz
WDW                          EM
SSB                   0
LB                     0.30 Hz
GB                    0
PC                     1.00
```



BRUKER

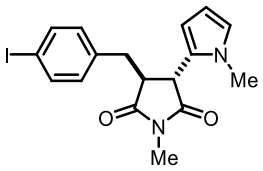
```
Current Data Parameters
NAME                  mah-341-13C
EXPNO                          10
PROCNO                          1

F2 - Acquisition Parameters
Date_                  20181016
Time                       3.13
INSTRUM                   spect
PROBHD    5 mm PATBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                 30303.031 Hz
FIDRES                 0.462388 Hz
AQ                  1.0813440 sec
RG                         10300
DW                     16.500 usec
DE                     10.00 usec
TE                     299.8 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                           1

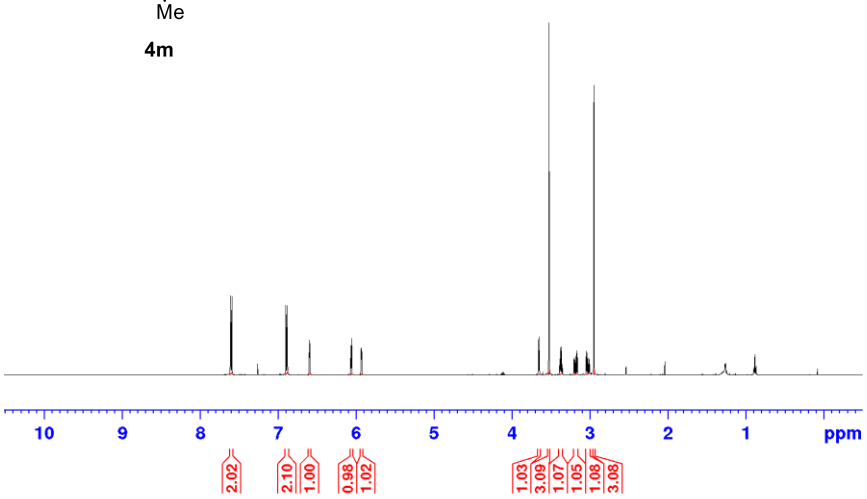
======== CHANNEL f1 ========
NUC1                         13C
P1                      9.50 usec
PL1                0 dB
PL1W                63.80191422 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                  80.00 usec
PL2                     -2.00 dB
PL12                    13.62 dB
PL13                    13.62 dB
PL2W                39.81071854 W
PL12W                1.09144032 W
PL13W                1.09144032 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                  125.7577867 MHz
WDW                          EM
SSB                   0
LB                     1.00 Hz
GB                    0
PC                     1.40
```



4m



```
Current Data Parameters
NAME      BSCB-705 C9-D11
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190411
Time                2.12
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7262976 sec
RG                    57
DW               83.200 usec
DE               10.00 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.50 usec
PL1       0 dB
PL1W        13.68978119 W
SFO1        500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300130 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

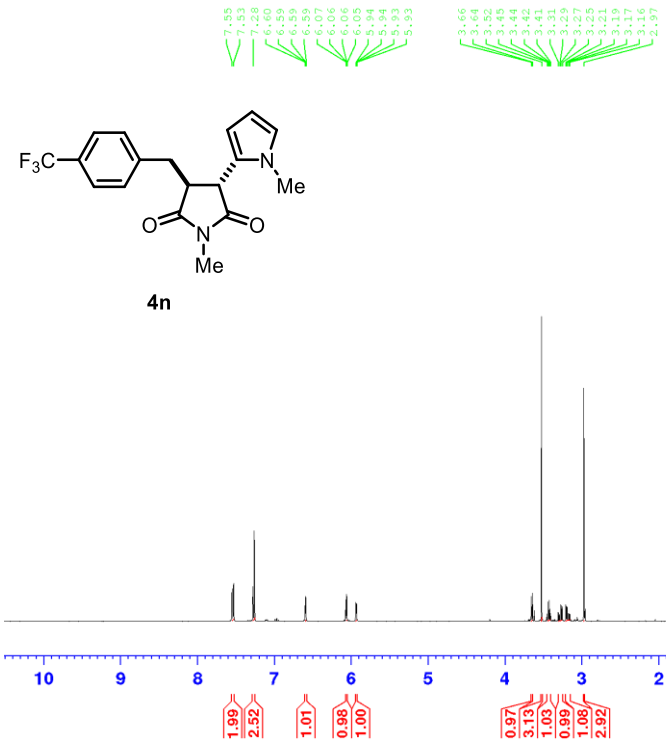

```
Current Data Parameters
NAME      BSCB-705 C9-D11
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190411
Time                2.31
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813440 sec
RG                 10300
DW               16.500 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 8.60 usec
PL1       0 dB
PL1W        53.75436783 W
SFO1        125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2       0 dB
PL12              16.12 dB
PL13              16.12 dB
PL2W        13.68978119 W
PL12W        0.33450022 W
PL13W        0.33450022 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577834 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

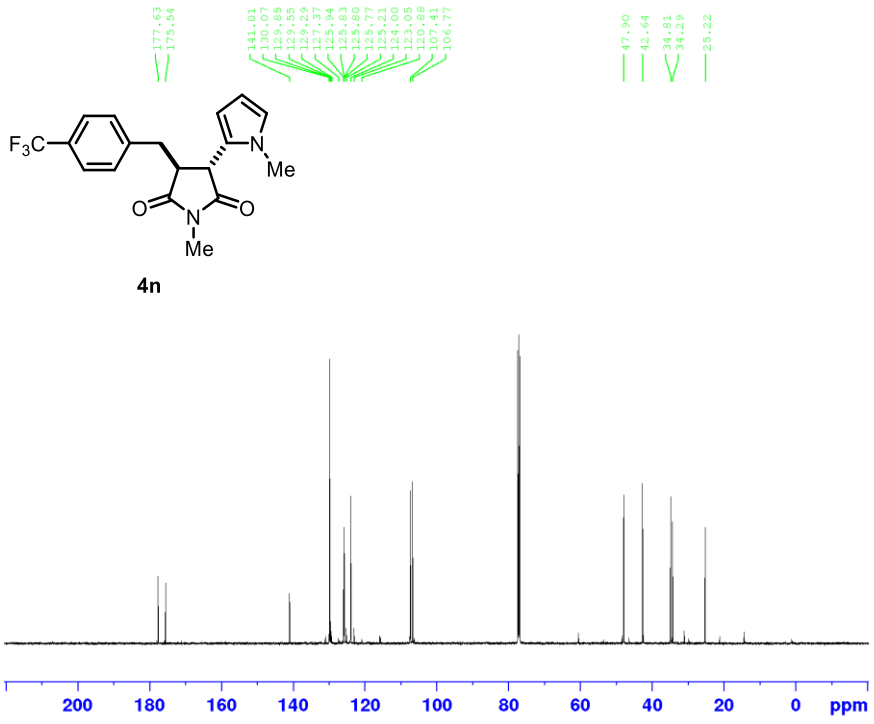


```
Current Data Parameters
NAME        SCH1011-rqst
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time              16.31
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               32768
SOLVENT           CDC13
NS                    8
DS                    2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                322.5
DW              104.400 usec
DE                6.00 usec
TE                298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -2.00 dB
PL1W        23.88643074 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300098 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                1.00
```



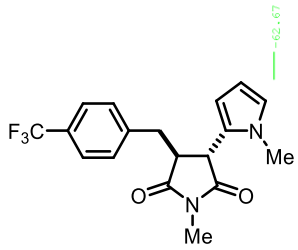
```
Current Data Parameters
NAME      sch1011-fc3-is
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181024
Time               7.38
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD               65536
SOLVENT           CDC13
NS                 1500
DS                    4
SWH          30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813440 sec
RG                13000
DW              16.500 usec
DE                10.00 usec
TE                299.4 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

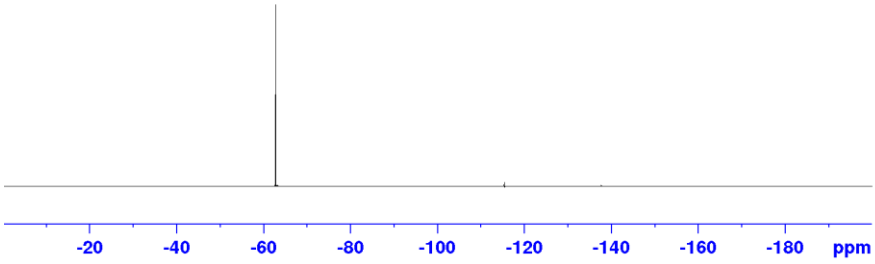
======== CHANNEL f1 ========
NUC1                 13C
P1                9.50 usec
PL1          0 dB
PL1W        63.80191422 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.62 dB
PL13              13.62 dB
PL2W        39.81071854 W
PL12W        1.09144032 W
PL13W        1.09144032 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577752 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.40
```



4n



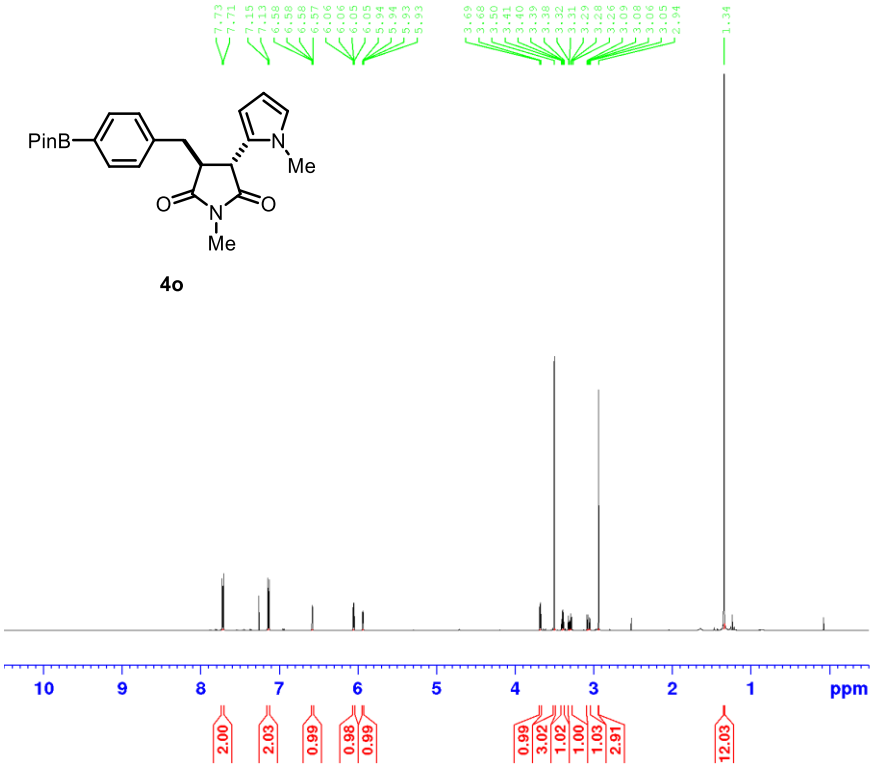

```
Current Data Parameters
NAME      sch1011-is-fc2
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181018
Time               19.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD               131072
SOLVENT             CDCl3
NS                      8
DS                      2
SWH            75187.969 Hz
FIDRES          0.573639 Hz
AQ              0.8716288 sec
RG                 1625.5
DW                 6.650 usec
DE                 7.14 usec
TE                 298.1 K
D1            5.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                 12.00 usec
PL1                -3.00 dB
SFO1         376.4607040 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               12.83 dB
PL13               12.83 dB
PL2W          23.88643074 W
PL12W          0.78550917 W
PL13W          0.78550917 W
SFO2         400.1320010 MHz

F2 - Processing parameters
SI                 65536
SF           376.4984036 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.00
```



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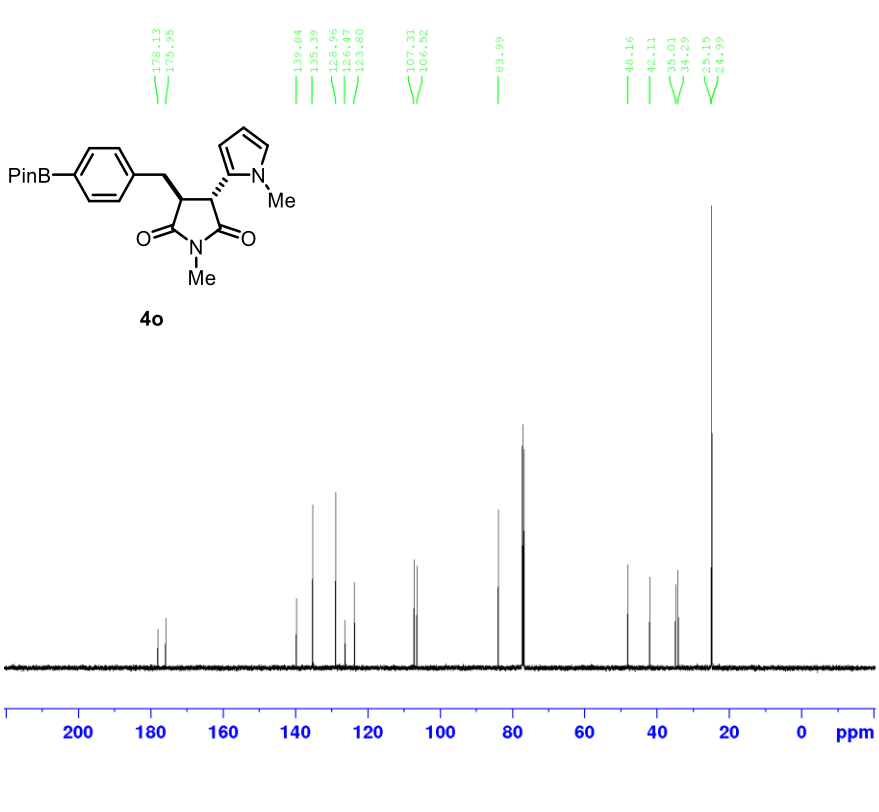
```
Current Data Parameters
NAME      sch1014-is-fc2-2
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20181020
Time                 5.44
INSTRUM             spect
PROBHD    5 mm PATBO BB-
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      2
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ               2.7262976 sec
RG                    128
DW                 83.200 usec
DE                 10.00 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.25 usec
PL1                -2.00 dB
PL1W          39.81071854 W
SFO1          500.1325007 MHz

F2 - Processing parameters
SI                  16384
SF            500.1300131 MHz
WDW                    no
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```



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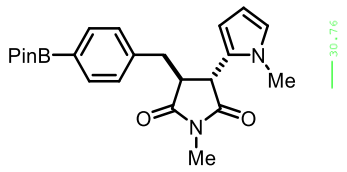
```
Current Data Parameters
NAME      sch1014-is-2fc-CR
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20181022
Time                16.42
INSTRUM             spect
PROBHD    5 mm PATBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    196
DS                      4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                  13000
DW                 16.500 usec
DE                 10.00 usec
TE                 301.2 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                     1

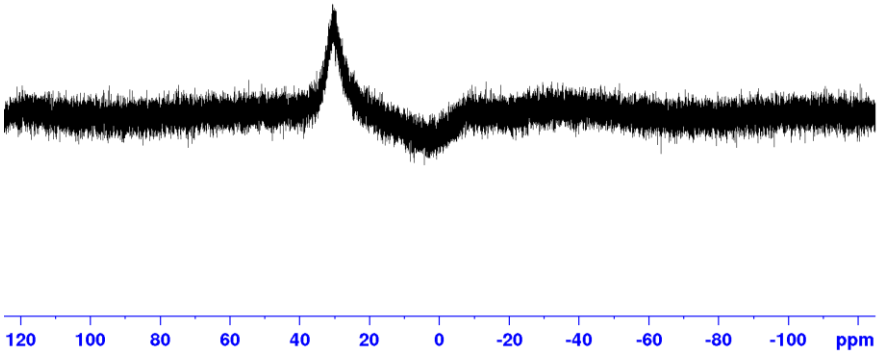
======== CHANNEL f1 ========
NUC1                   13C
P1                  9.50 usec
PL1       0 dB
PL1W          63.80191422 W
SFO1          125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.62 dB
PL13               13.62 dB
PL2W          39.81071854 W
PL12W          1.09144032 W
PL13W          1.09144032 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                  32768
SF            125.7577751 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                   1.40
```



4o



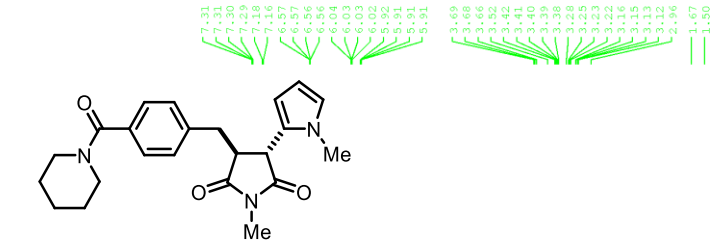

```
Current Data Parameters
NAME      sch1014-is-fc2
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181019
Time                1.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg1g
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ             1.0223616 sec
RG                  512
DW                15.600 usec
DE                 6.00 usec
TE                298.2 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1

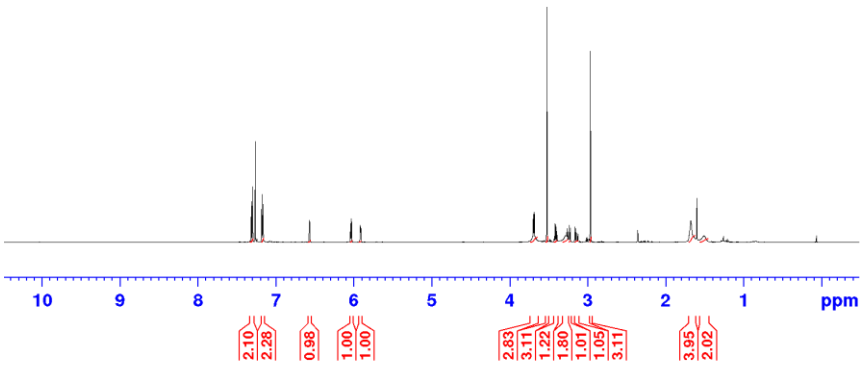
======== CHANNEL f1 ========
NUC1                 11B
P1                  8.00 usec
PL1                 6.00 dB
SFO1         128.3776050 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                12.83 dB
PL2W         23.88643074 W
PL12W         0.78550917 W
SFO2         400.1320010 MHz

F2 - Processing parameters
SI                  32768
SF           128.3776141 MHz
WDW                    no
SSB       0
LB        0 Hz
GB        0
PC                  1.40
```



4p



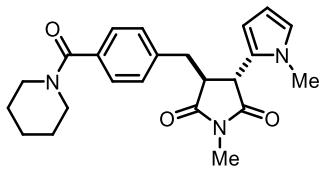
BRUKER

```
Current Data Parameters
NAME      sch1154-2FC-1s
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190414
Time              12.26
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6009.615 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                  256
DW             83.200 usec
DE             10.00 usec
TE               297.8 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.50 usec
PL1       0 dB
PL1W       13.68978119 W
SFO1       500.1325007 MHz

F2 - Processing parameters
SI                16384
SF          500.1300134 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



4p

BRUKER

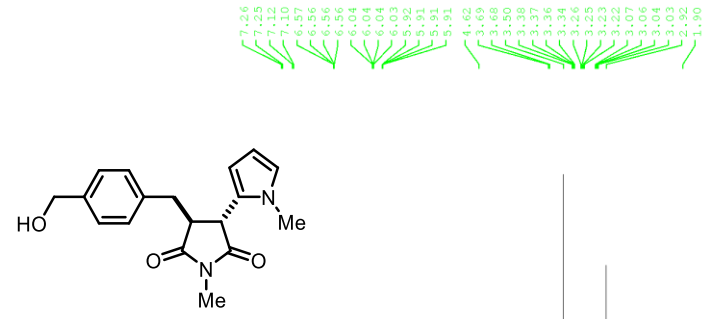
```
Current Data Parameters
NAME       sch1154-Crep
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190416
Time              23.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1600
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG                26008
DW             20.800 usec
DE             10.00 usec
TE               298.1 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1               9.25 usec
PL1              -3.00 dB
PL1W       75.17808533 W
SFO1       100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -3.00 dB
PL12             13.05 dB
PL13             13.05 dB
PL2W       30.07123375 W
PL12W       0.74670875 W
PL13W       0.74670875 W
SFO2       400.1616006 MHz

F2 - Processing parameters
SI                32768
SF          100.6203120 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```



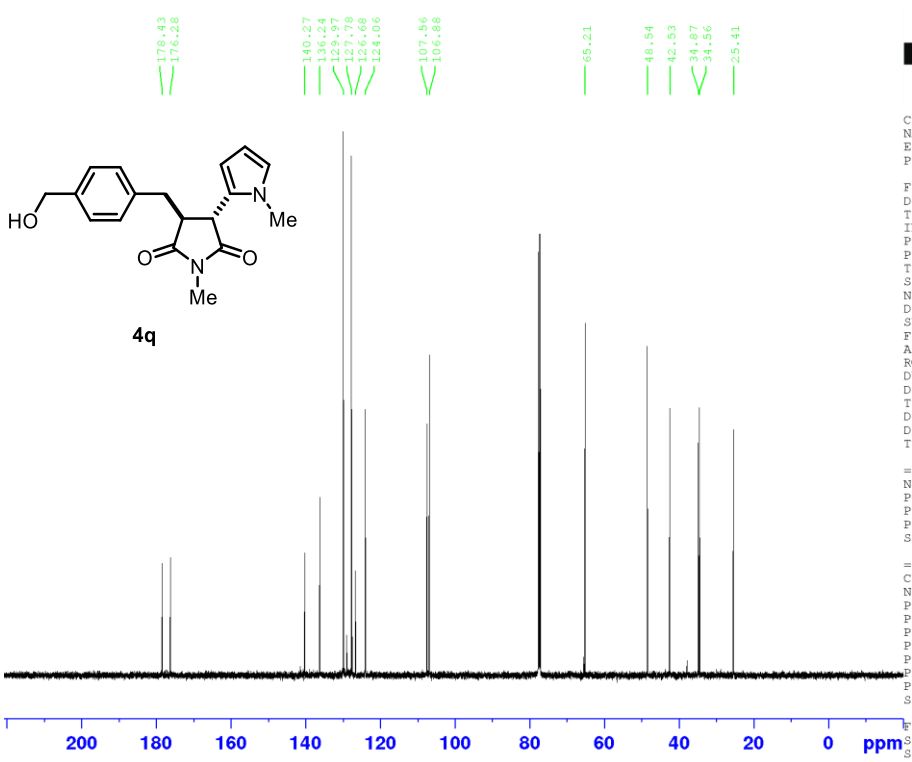
4q


```
Current Data Parameters
NAME      BSCB-SCH-1159 HV
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190417
Time              19.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                 71.8
DW                83.200 usec
DE                10.00 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.50 usec
PL1        0 dB
PL1W       13.68978119 W
SFO1       500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300210 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```



4q

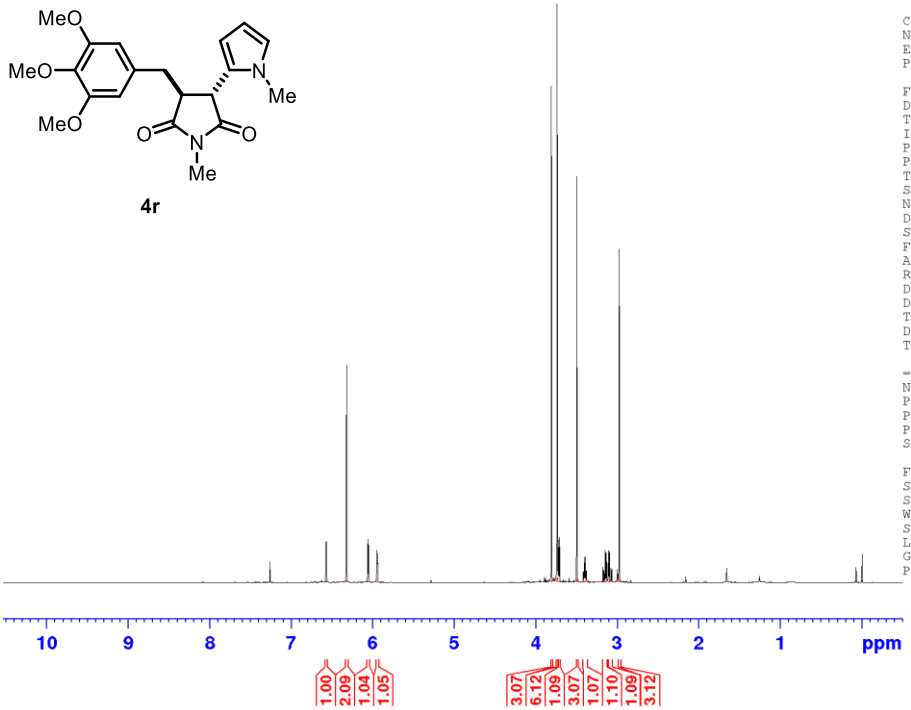
```
Current Data Parameters
NAME      BSCB-SCH-1159 HV
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190417
Time              20.11
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813440 sec
RG                10300
DW                16.500 usec
DE                10.00 usec
TE                298.5 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 8.60 usec
PL1        0 dB
PL1W       53.75436783 W
SFO1       125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2        0 dB
PL12              16.12 dB
PL13              16.12 dB
PL2W       13.68978119 W
PL12W       0.33450022 W
PL13W       0.33450022 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577441 MHz
WDW                   EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```



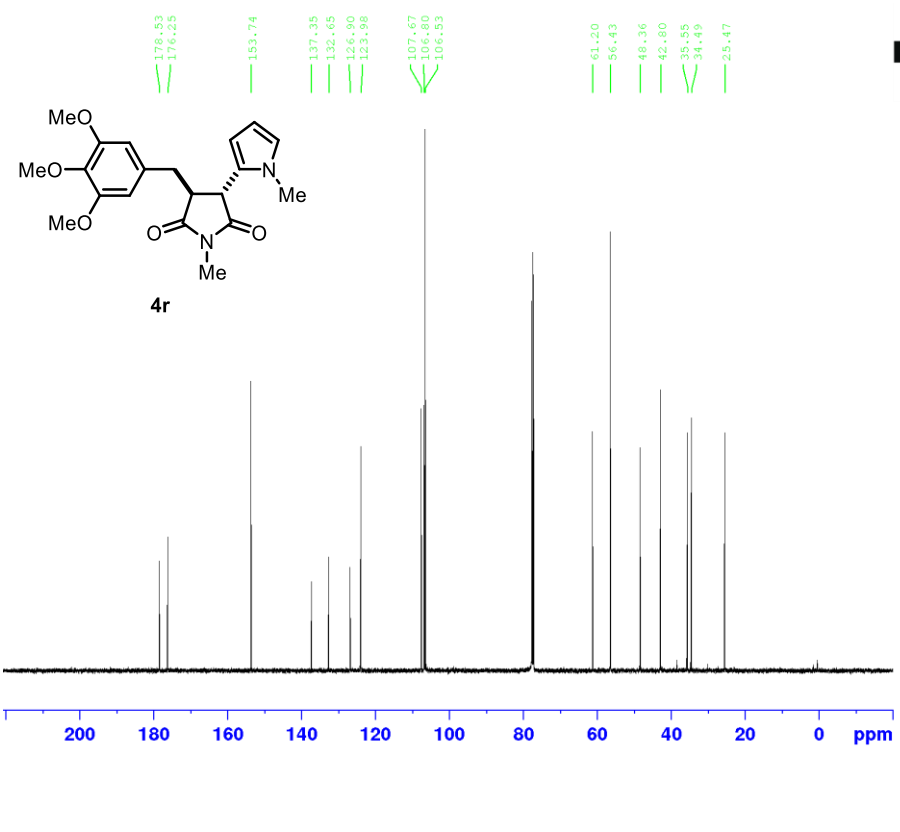
4r

```
Current Data Parameters
NAME                  mah-322-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180908
Time                       5.42
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                            32
DS                             2
SWH                  10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                           64
DW                       50.000 usec
DE                       10.00 usec
TE                       297.9 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                       13.30 usec
PL1                       3.00 dB
PL1W                12.58925438 W
SFO1                500.1330008 MHz

F2 - Processing parameters
SI                           32768
SF                  500.1300131 MHz
WDW                             EM
SSB                0
LB                            0.30 Hz
GB                 0
PC                            1.00
```



4r

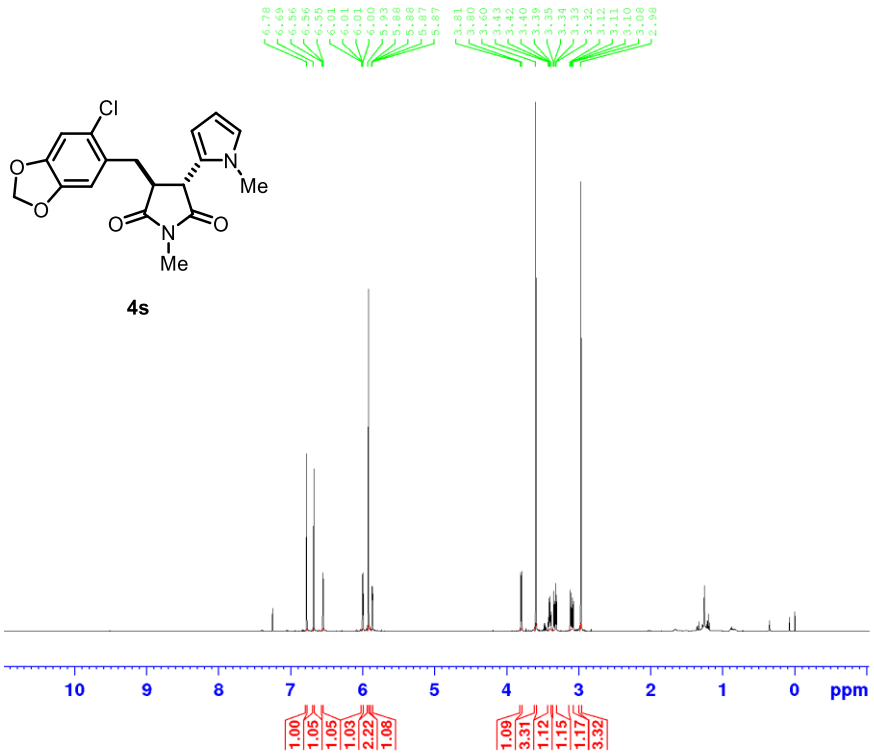
```
Current Data Parameters
NAME                  mah-322-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20180908
Time                       6.19
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         1024
DS                            4
SWH                  30303.031 Hz
FIDRES                0.462388 Hz
AQ                    1.0813440 sec
RG                         11500
DW                       16.500 usec
DE                       10.00 usec
TE                       298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                        13C
P1                        8.60 usec
PL1            0 dB
PL1W                63.80191422 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       3.00 dB
PL12                     18.58 dB
PL13                     18.58 dB
PL2W                12.58925438 W
PL12W                0.34833732 W
PL13W                0.34833732 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                           32768
SF                  125.7577441 MHz
WDW                             EM
SSB                0
LB                            1.00 Hz
GB                 0
PC                            1.40
```

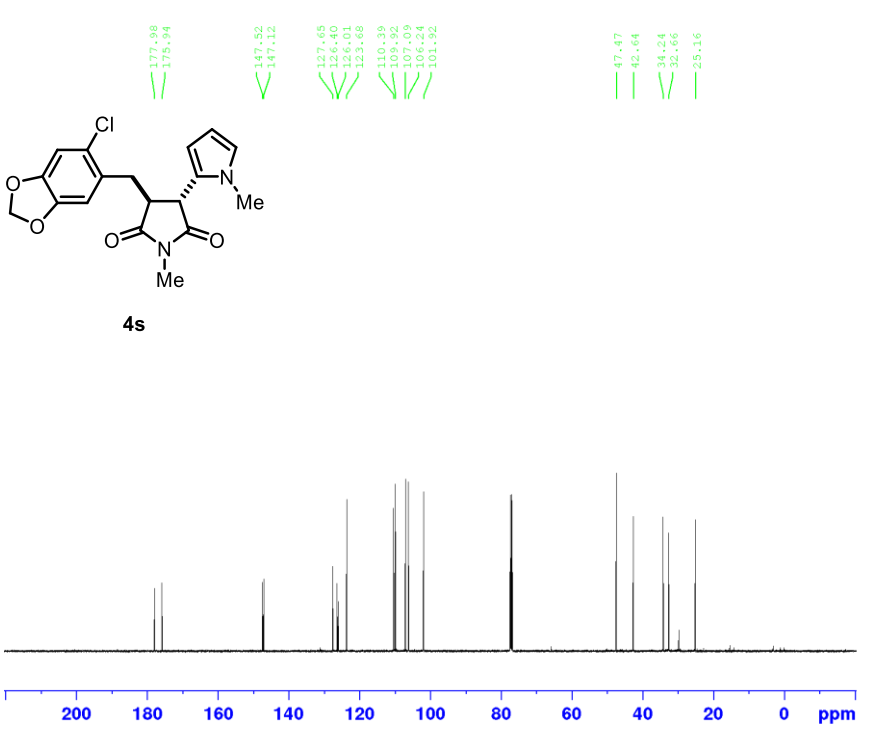



```
Current Data Parameters
NAME      sch1065-fc2-dry4
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20181218
Time                9.49
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                    64
DW                83.200 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -4.08 dB
PL1W         35.02648163 W
SFO1         500.1325007 MHz

F2 - Processing parameters
SI                 16384
SF          500.1300130 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



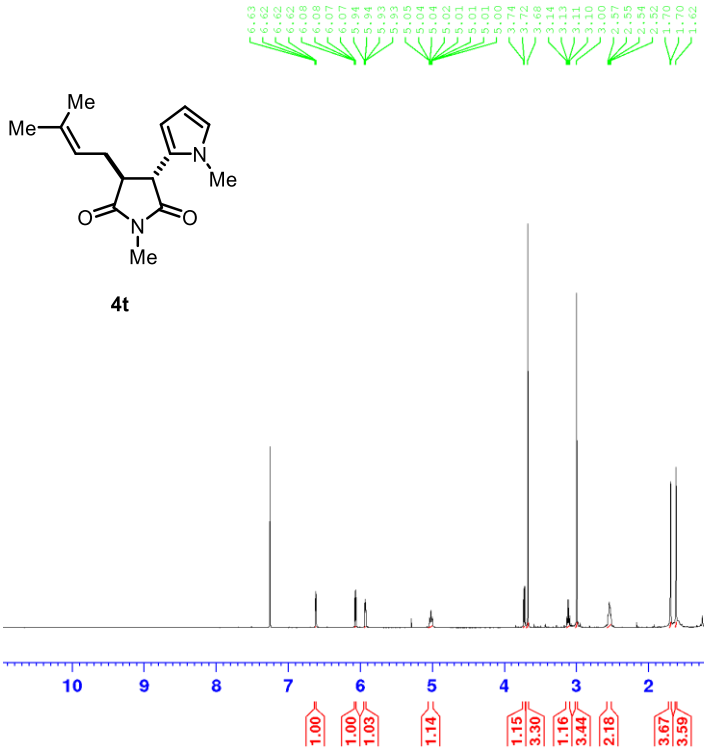

```
Current Data Parameters
NAME      sch1065-fc2-dry2-C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20181218
Time               11.22
INSTRUM            spect
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 10300
DW                16.500 usec
DE                10.00 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.25 usec
PL1          0 dB
PL1W         53.75436783 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.08 dB
PL12               11.54 dB
PL13               11.54 dB
PL2W         35.02648163 W
PL12W         0.96027696 W
PL13W         0.96027696 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577816 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4t

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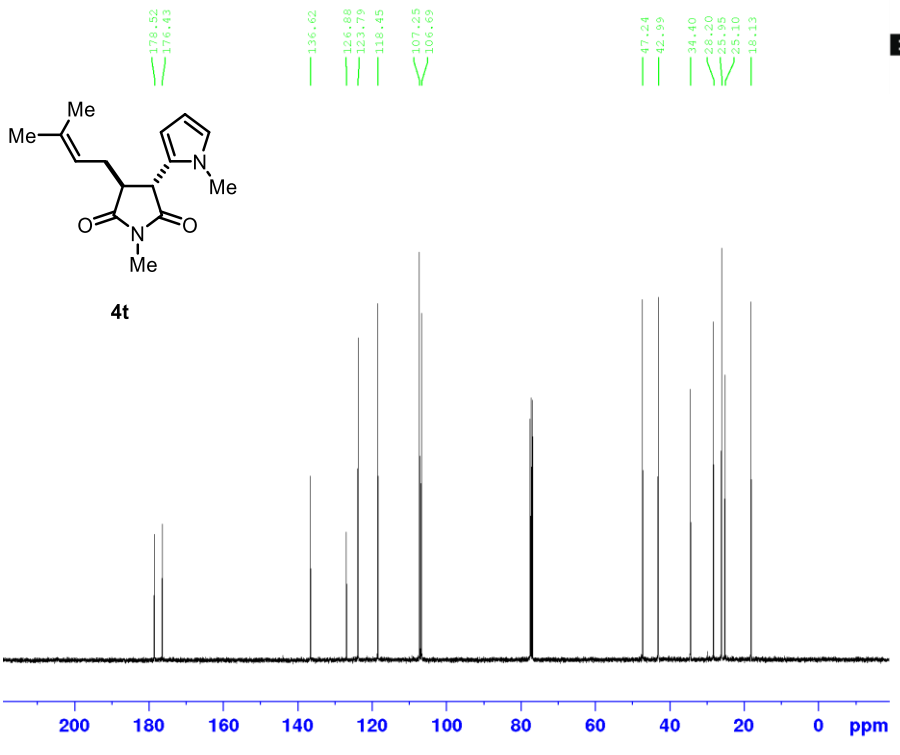
```
Current Data Parameters
NAME         MAH330-rqst
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time               16.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                 322.5
DW                104.400 usec
DE                  6.00 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



4t

BRUKER

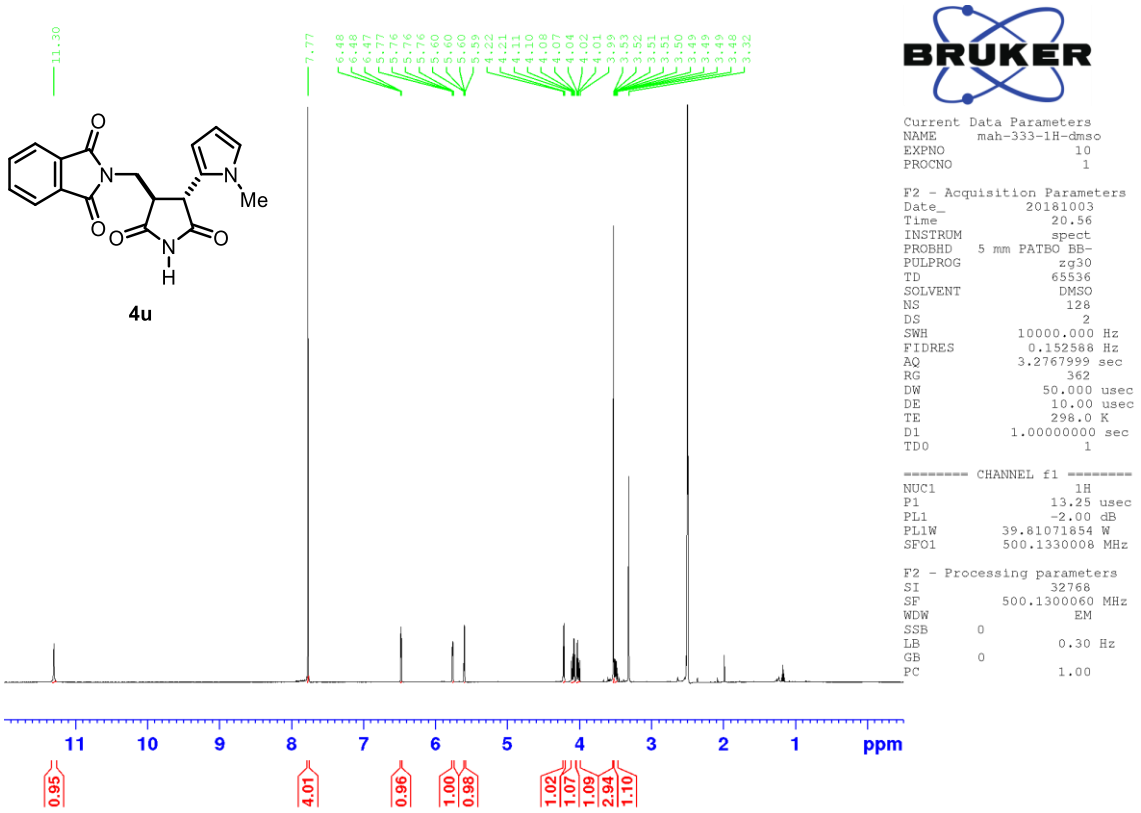
```
Current Data Parameters
NAME      mah-330-13C-400
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20180922
Time                7.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664256 sec
RG                 32768
DW                20.850 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.00 dB
PL13               13.00 dB
PL2W         30.07123375 W
PL12W         0.75535524 W
PL13W         0.75535524 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127600 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



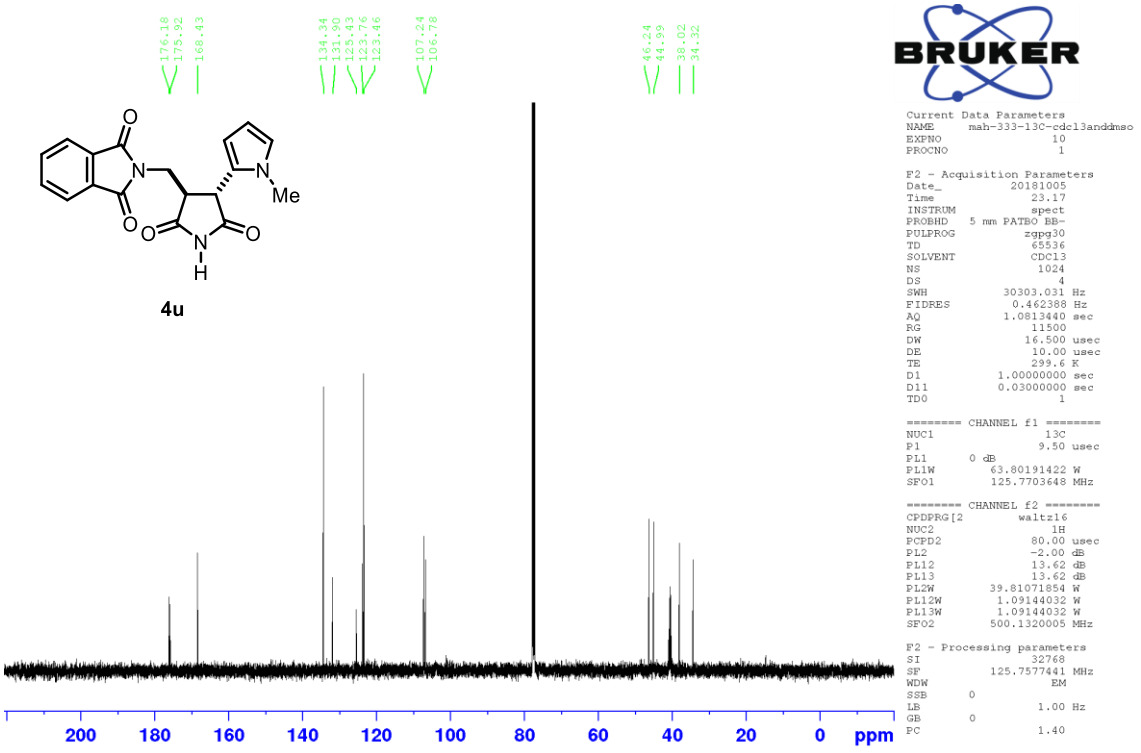
BRUKER

```
Current Data Parameters
NAME      mah-333-1H-dmso
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20181003
Time              20.56
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   128
DS                     2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2767999 sec
RG                   362
DW                50.000 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.25 usec
PL1                -2.00 dB
PL1W         39.81071854 W
SFO1         500.1330008 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300060 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

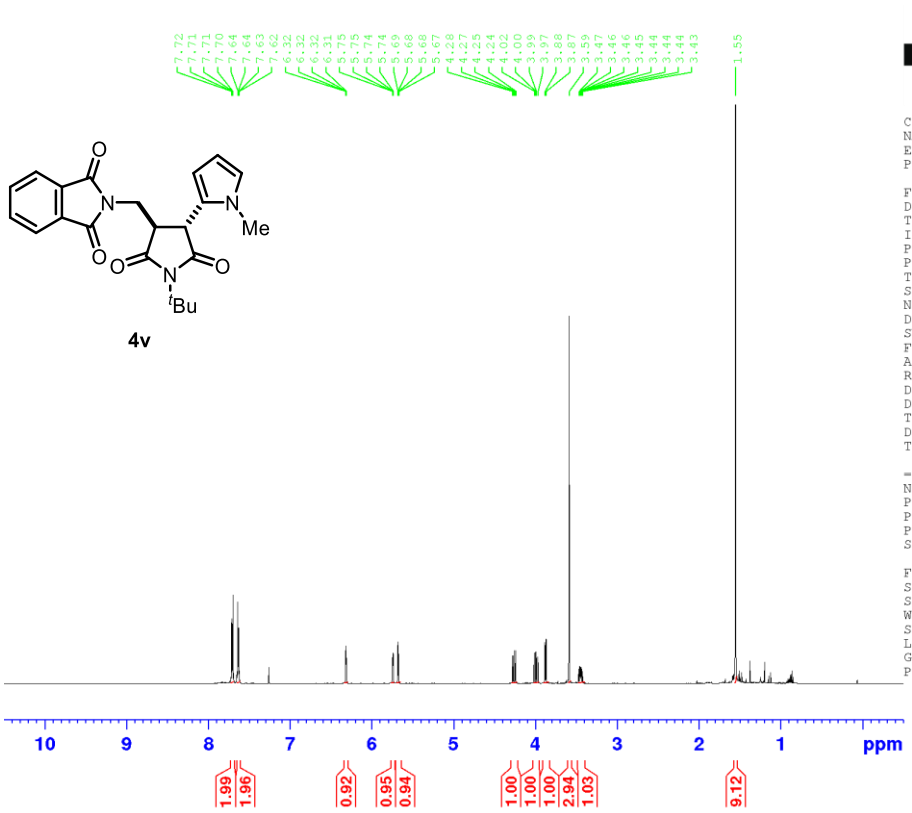
```
Current Data Parameters
NAME      mah-333-13C-cdcl3anddmso
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20181005
Time              23.17
INSTRUM           spect
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30303.031 Hz
FIDRES            0.462388 Hz
AQ             1.0813440 sec
RG                11500
DW                16.500 usec
DE                10.00 usec
TE                299.6 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.50 usec
PL1       0 dB
PL1W         63.80191422 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.62 dB
PL13               13.62 dB
PL2W         39.81071854 W
PL12W         1.09144032 W
PL13W         1.09144032 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577441 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

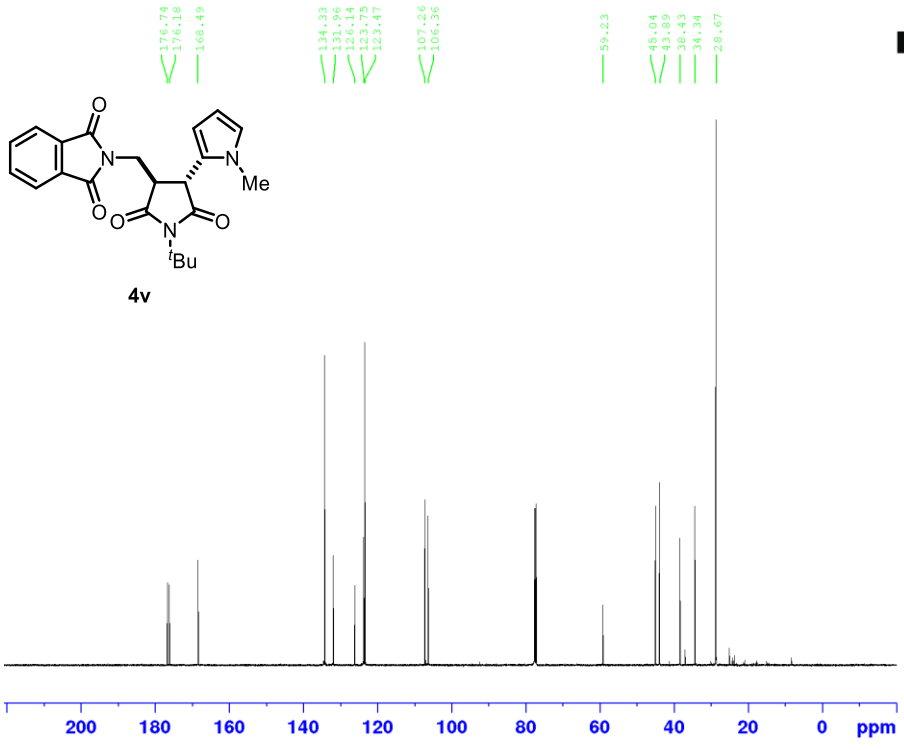


```
Current Data Parameters
NAME                  mah-334-1H
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20181001
Time                      17.53
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                 10000.000 Hz
FIDRES                 0.152588 Hz
AQ                     3.2767999 sec
RG                         50.8
DW                       50.000 usec
DE                       10.00 usec
TE                       298.2 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         13.30 usec
PL1                         3.00 dB
PL1W                12.58925438 W
SFO1                500.1330008 MHz

F2 - Processing parameters
SI                            32768
SF                  500.1300135 MHz
WDW                            EM
SSB                 0
LB                         0.30 Hz
GB                  0
PC                         1.00
```



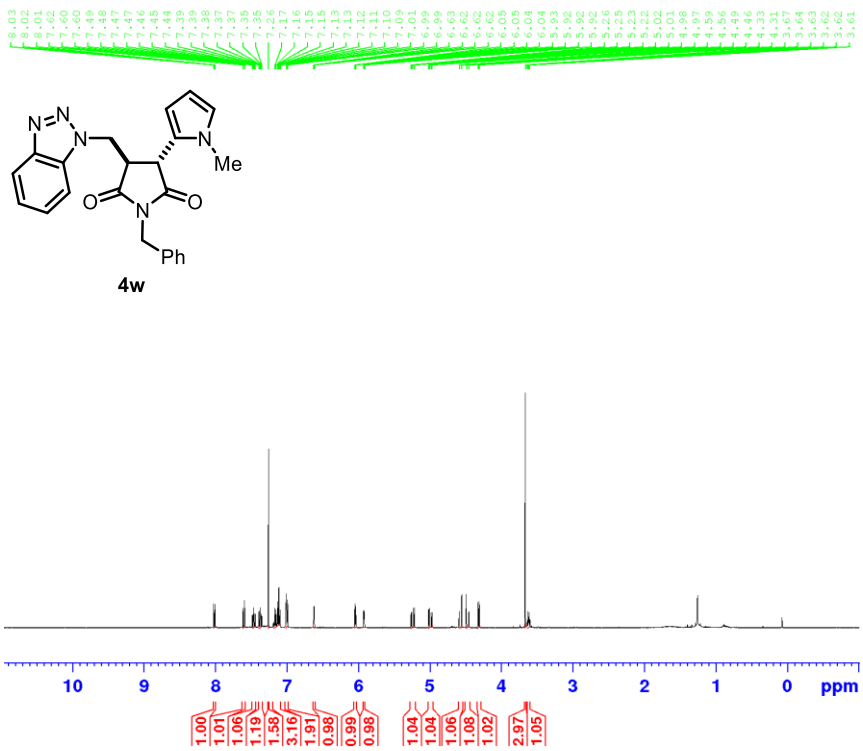
```
Current Data Parameters
NAME                 mah-334-13C
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20181001
Time                      18.31
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                 30303.031 Hz
FIDRES                 0.462388 Hz
AQ                     1.0813440 sec
RG                         11500
DW                       16.500 usec
DE                       10.00 usec
TE                       299.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          13C
P1                          8.60 usec
PL1                 0 dB
PL1W                63.80191422 W
SFO1                125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         3.00 dB
PL12                       18.58 dB
PL13                       18.58 dB
PL2W                12.58925438 W
PL12W                0.34833732 W
PL13W                0.34833732 W
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                            32768
SF                  125.7577441 MHz
WDW                            EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
```



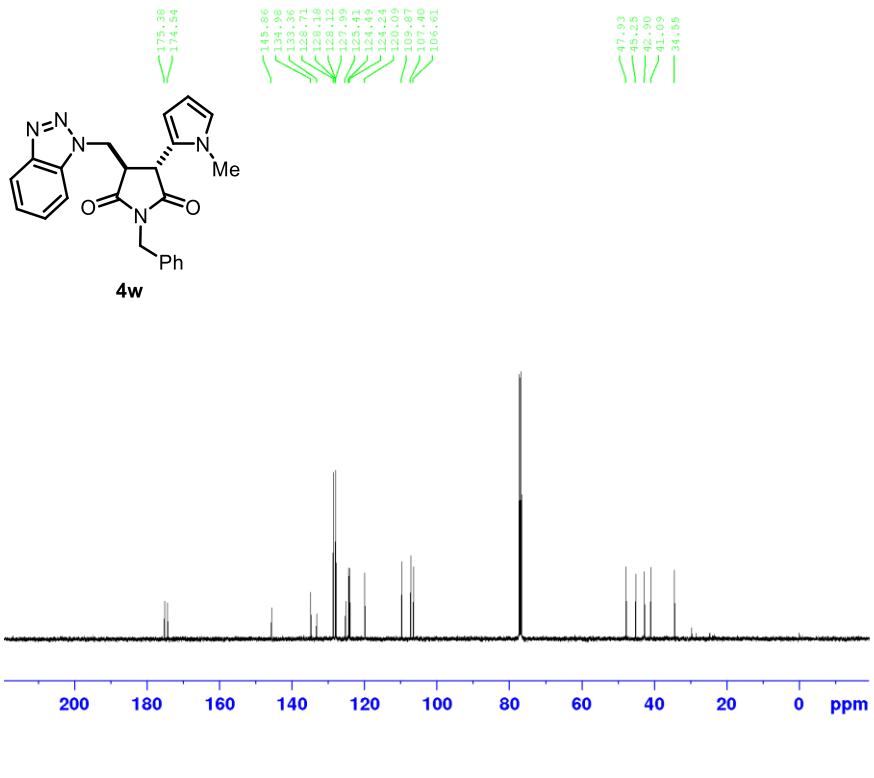
BRUKER

```
Current Data Parameters
NAME         sch1056-rqs
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time              13.14
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4209793 sec
RG                 322.5
DW               104.400 usec
DE                 6.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300097 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



BRUKER

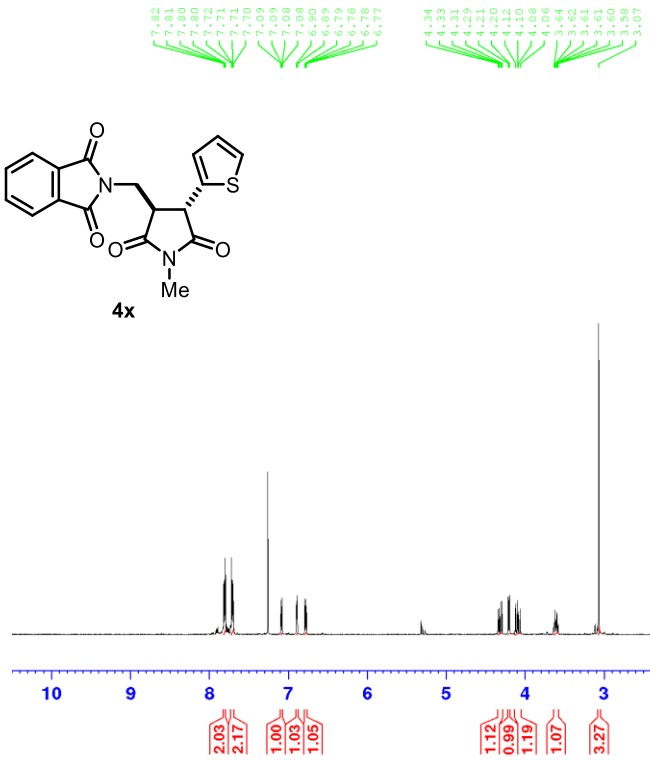
```
Current Data Parameters
NAME      SCH1056-fc1-hexw-cnc
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20181204
Time               5.08
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                26008
DW               20.800 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.25 usec
PL1                -3.00 dB
PL1W         75.17808533 W
SFO1         100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.05 dB
PL13               13.05 dB
PL2W         30.07123375 W
PL12W         0.74670875 W
PL13W         0.74670875 W
SFO2         400.1616006 MHz

F2 - Processing parameters
SI                 32768
SF           100.6203005 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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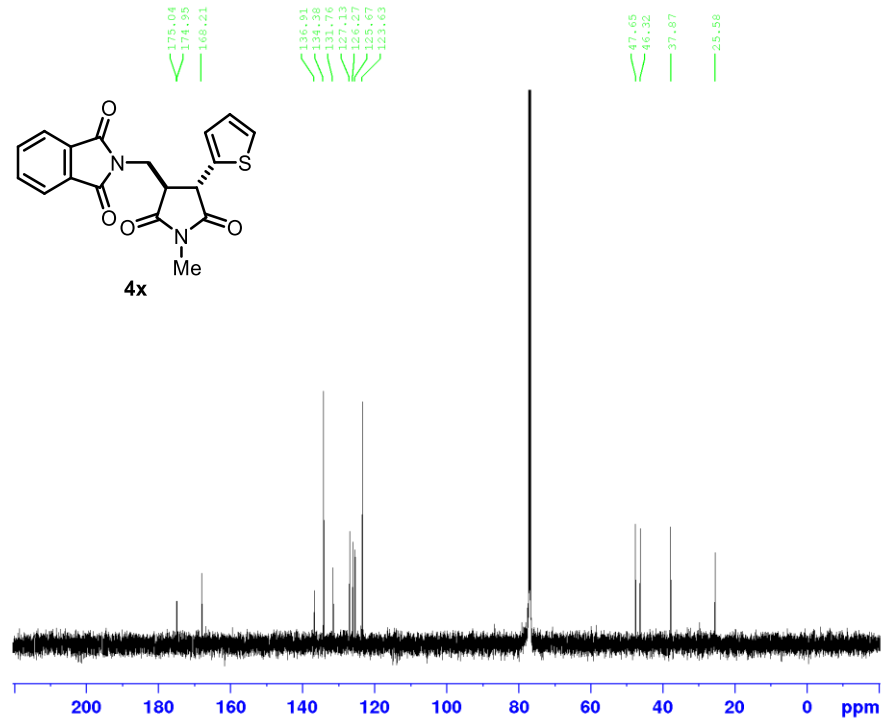
```
Current Data Parameters
NAME         sch990-rqst
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190130
Time               16.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             4789.272 Hz
FIDRES          0.146157 Hz
AQ              3.4209793 sec
RG                   362
DW                104.400 usec
DE                  6.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -2.00 dB
PL1W         23.88643074 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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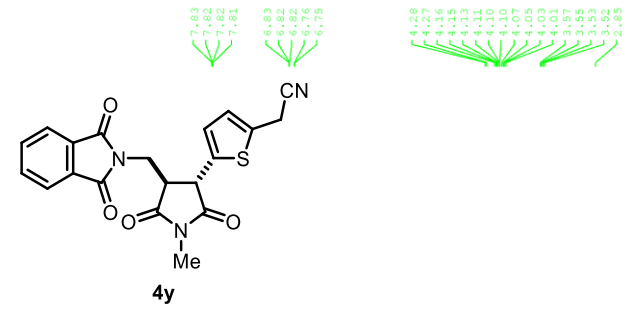
```
Current Data Parameters
NAME      SCH990-is-HV8h
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180926
Time                3.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1500
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813440 sec
RG                 11500
DW                16.500 usec
DE                10.00 usec
TE                298.7 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

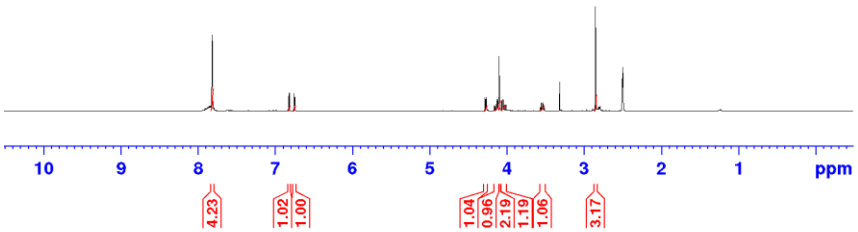
======== CHANNEL f1 ========
NUC1                  13C
P1                 8.60 usec
PL1        0 dB
PL1W         63.80191422 W
SFO1         125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 3.00 dB
PL12               18.58 dB
PL13               18.58 dB
PL2W         12.58925438 W
PL12W         0.34833732 W
PL13W         0.34833732 W
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577715 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4y

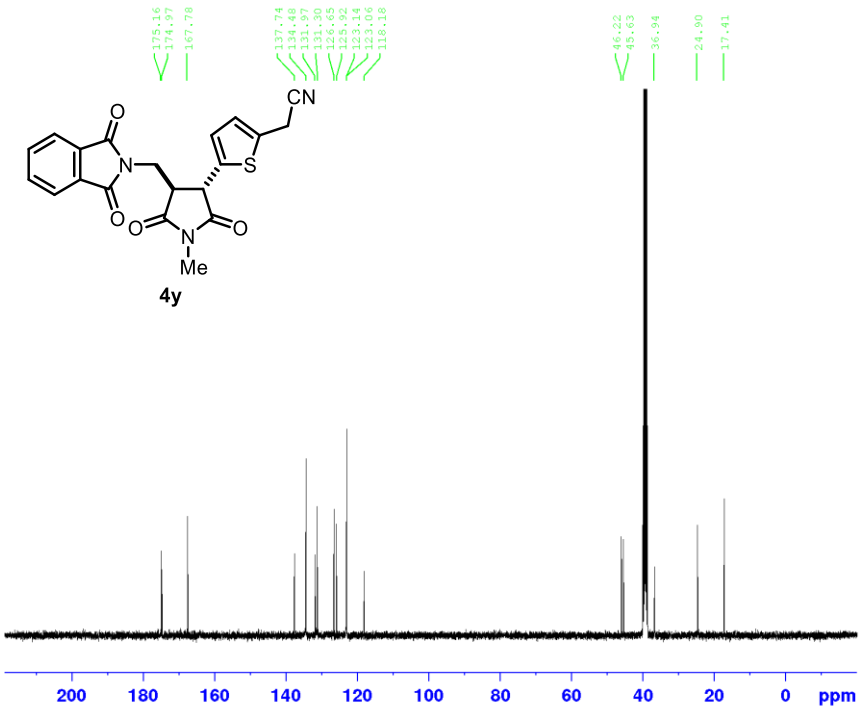



```
Current Data Parameters
NAME      sch1146-2FC-is-dmso
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_            20190415
Time                12.08
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  32768
SOLVENT              DMSO
NS                       8
DS                       2
SWH              4789.272 Hz
FIDRES           0.146157 Hz
AQ               3.4209793 sec
RG                   322.5
DW               104.400 usec
DE                 6.00 usec
TE                 298.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.50 usec
PL1                 -2.00 dB
PL1W          23.88643074 W
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                   16384
SF            400.1300032 MHz
WDW                     EM
SSB         0
LB                   0.30 Hz
GB          0
PC                   1.00
```



4y

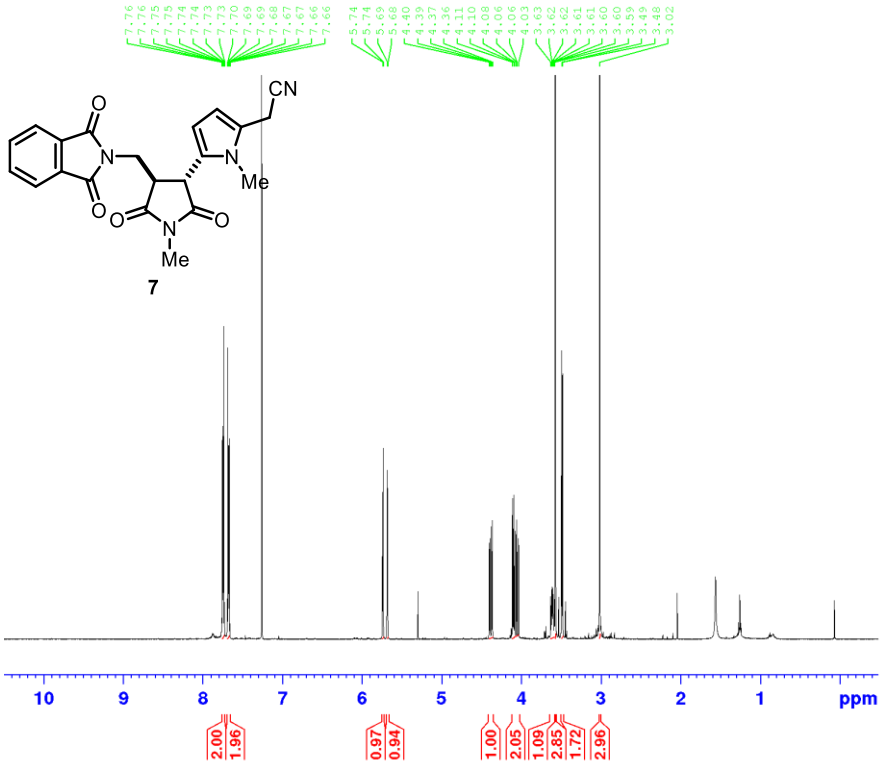

```
Current Data Parameters
NAME      sch1146-2FC-is-dmso
EXPNO                      15
PROCNO                      1

F2 - Acquisition Parameters
Date_            20190415
Time                21.08
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT              DMSO
NS                   1500
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                   26008
DW               20.800 usec
DE                 10.00 usec
TE                 298.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  9.25 usec
PL1                 -3.00 dB
PL1W          75.17808533 W
SFO1          100.6303736 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.05 dB
PL13                13.05 dB
PL2W          30.07123375 W
PL12W          0.74670875 W
PL13W          0.74670875 W
SFO2          400.1616006 MHz

F2 - Processing parameters
SI                   32768
SF            100.6203605 MHz
WDW                     EM
SSB         0
LB                   1.00 Hz
GB          0
PC                   1.40
```

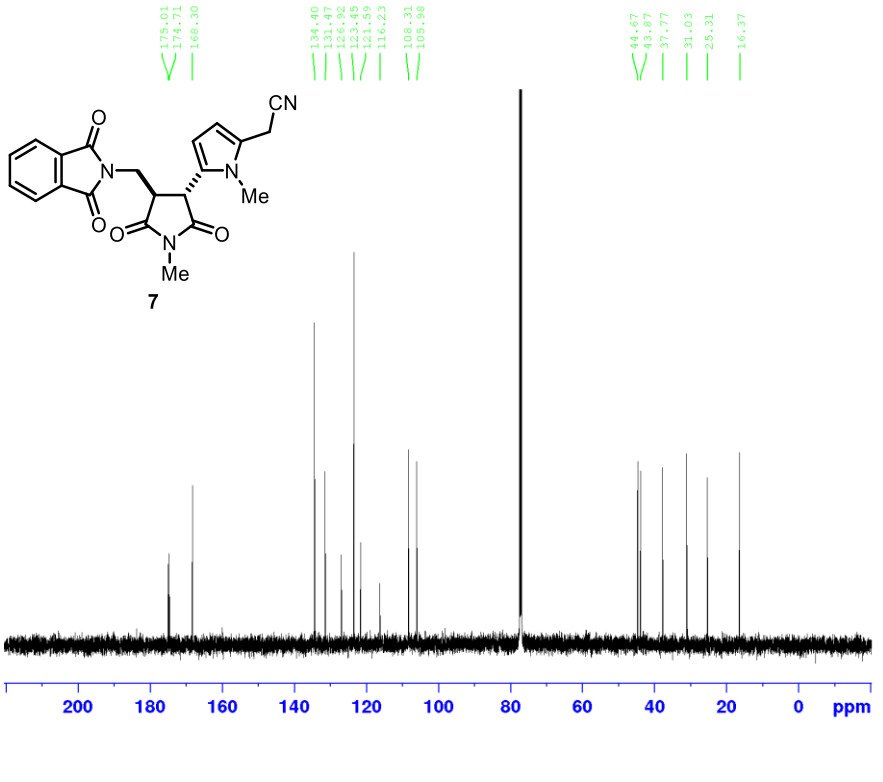



```
Current Data Parameters
NAME      SCH1002-HV-OVN-is
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_               20181009
Time                   21.29
INSTRUM                spect
PROBHD    5 mm PATBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7262976 sec
RG                      575
DW                   83.200 usec
DE                   10.00 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.25 usec
PL1                  -2.00 dB
PL1W          39.81071854 W
SFO1          500.1325007 MHz

F2 - Processing parameters
SI                    16384
SF            500.1300130 MHz
WDW                     EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```



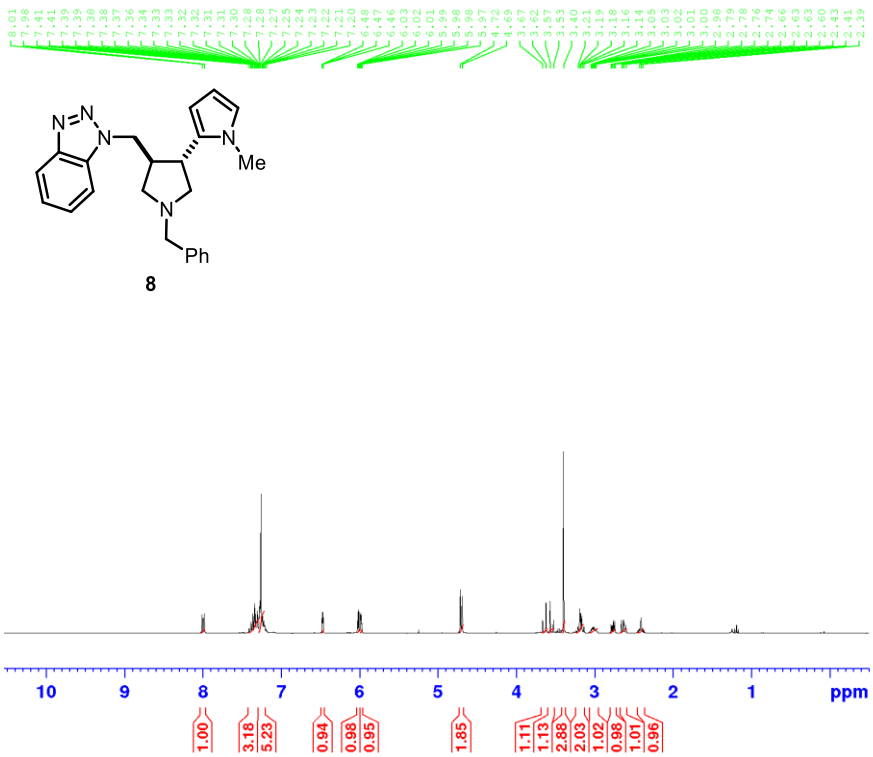

```
Current Data Parameters
NAME      SCH1002-HV-OVN-is
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_               20181009
Time                   22.24
INSTRUM                spect
PROBHD    5 mm PATBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1500
DS                        4
SWH           30303.031 Hz
FIDRES            0.462388 Hz
AQ                1.0813440 sec
RG                    10300
DW                   16.500 usec
DE                   10.00 usec
TE                   300.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.50 usec
PL1        0 dB
PL1W          63.80191422 W
SFO1          125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.62 dB
PL13                 13.62 dB
PL2W          39.81071854 W
PL12W          1.09144032 W
PL13W          1.09144032 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF            125.7577716 MHz
WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```



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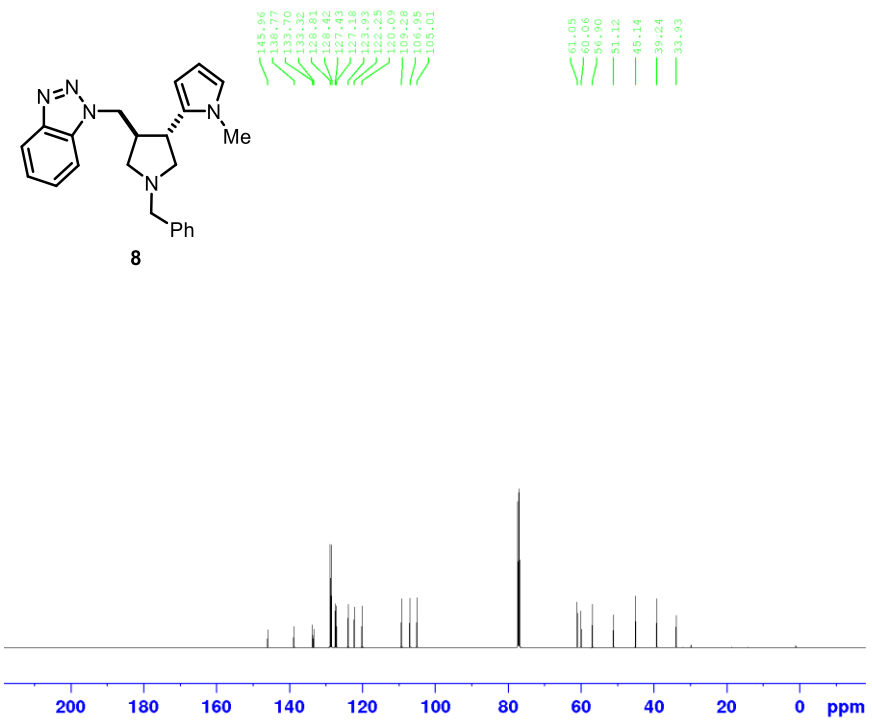
```
Current Data Parameters
NAME        sch1073-is-2
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190109
Time               15.13
INSTRUM       FOURIER300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH            6103.516 Hz
FIDRES          0.093132 Hz
AQ             5.3687091 sec
RG              9.66798
DW                81.920 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1818011 MHz
NUC1                   1H
P1                 11.75 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 32768
SF           300.1800164 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.40
```



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```
Current Data Parameters
NAME        SCH1088AFtfc
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190214
Time               15.01
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1283
DS                     2
SWH           29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                   812
DW                16.800 usec
DE                18.00 usec
TE                298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.8608968 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         63.00000000 W

======== CHANNEL f2 ========
SFO2         500.4925025 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          7.49889994 W
PLW12         0.26363000 W
PLW13         0.16873001 W

F2 - Processing parameters
SI                 32768
SF           125.8482992 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```