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Supplementary Information

Preparation of Pyrrolizinone Derivatives via Sequential Transformations of Cyclic Allyl Imides: Synthesis of Quinolactacide and Marinamide

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General informations

The NMR spectra were recorded on a Bruker Ascend 400 (400 MHz) spectrometer. Chemical shifts are given in parts per million (*δ*) downfield from tetramethylsilane as the internal standard. Deuterochloroform was used as a solvent, unless otherwise stated. Mass spectral data were recorded using Agilent Techologies 6520 Q-TOF spectrometer coupled with Agilent 1200 HPLC or Agilent Technologies 5975C MS coupled with Agilent Technologies 6890N GC. IR spectra were recorded on a IR Termo Scientific NICOLET iS10 (4950) spectrometer. Melting points were determined using Boetius PHMK 05 apparatus without correction. Flash chromatography employed silica gel 60 (230-400 mesh) while thin layer chromatography was carried out using alumina plates with 0.25 mm silica layer (Kieselgel 60 F_{254}, Merck). Compounds were visualized by staining with potassium permanganate solution. The solvents were purified by distillation before use.

General procedures for synthesis of *N*-allyl imides

Procedure A[1]

Anhydride (3 mmol) and allyl amine (513 mg, 9 mmol) were dissolved in toluene (40 mL) and triethylamine (30 mg, 0.3 mmol) was added. The resulting mixture was refluxed for 5-12 h. The solvent was then evaporated under reduced pressure, the solid residue was dissolved in ethyl acetate and washed with water (3 x 10 mL) and brine (2 x 5mL). After drying over anhydrous Na_2SO_4 and removal of the solvent under reduced pressure, the crude imide was purified by flash column chromatography (SiO_2, petroleum ether-ether) to afford the product which was used in next step without further purification.

Procedure B[2]

To a mixture of imide (3 mmol) and potassium carbonate (1.03 g, 7.5 mmol) in acetonitrile (7 mL) allylbromide (0.3 g, 2.5 mmol) was added at ambient temperature. The resulting mixture was stirred at reflux for 13h. After completion of the reaction, the mixture was filtered and the solvent was evaporated under reduced pressure. The crude mixture was purified by column chromatography (SiO_2, petroleum ether-ether) to afford the product which was used in next step without further purification.

6-Allyl-6H-pyrrolo[3,4-b]pyridine-5,7-dione **12h.** The title compound prepared following procedure A was isolated (451 mg, 80%) as a white solid, Mp=107-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.98 (dd, *J* = 5.0, 1.5 Hz, 1H), 8.18 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.63 (dd, *J* = 7.6, 5.0 Hz, 1H), 6.05 – 5.79 (m, 1H), 5.27 (ddd, *J* = 13.7, 11.3, 1.1 Hz, 2H), 4.37 (dt, *J* = 5.8, 1.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 155.3, 151.8, 131.2, 131.0, 127.4, 127.3, 118.5, 40.3.

2-Allyl-4,7-bis(allyloxy)isoindoline-1,3-dione **12i.** The title compound prepared following procedure B from 4,7-dixydroxy-isoindole-1,3-dione with 10 equivalents of allyl bromide was isolated (134 mg, 15%) as a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (s, 2H), 6.06 (ddt, *J* = 17.2, 10.3, 5.0 Hz, 2H), 5.86 (ddt, *J* = 16.0, 10.3, 5.7 Hz, 1H), 5.50 (dd, *J* = 17.3, 1.4 Hz, 2H), 5.33 (dd, *J* = 10.6, 1.3 Hz, 2H), 5.25 (dd, *J* = 17.1, 1.2 Hz, 1H), 5.16 (dd, *J* = 10.2, 1.1 Hz, 1H), 4.72 (dd, *J* = 3.5, 1.5 Hz, 4H), 4.23 (d, *J* = 5.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 149.9, 132.4, 131.7, 122.0, 119.2, 118.2, 117.3, 70.5, 39.8.

2-Allyl-2H-pyrrolo[3,4-b]quinoline-1,3-dione **12l.** The title compound prepared following procedure A was isolated (471 mg, 66%) as a white solid, Mp=182-185 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (d, *J* = 3.9 Hz, 1H), 8.54 – 8.38 (m, 1H), 8.13 – 8.04 (m, 1H), 7.95 (s, 1H), 7.78 (d, *J* = 4.2 Hz, 1H), 6.07 – 5.88 (m, 1H), 5.30 (dd, *J* = 32.0, 13.6 Hz, 2H), 4.46 (d, *J* = 1.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.8, 165.6, 150.9, 132.8, 132.7, 131.6, 130.9, 129.9, 129.6, 128.8, 123.0, 118.57, 40.6.

(3S,4R)-1-Allyl-3,4-bis (benzyloxy)pyrrolidine-2,5-dione **12m.** The title compound prepared following procedure A was isolated (537 mg, 51%) as a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.29 (m, 10H), 5.78 (ddd, *J* = 17.0, 6.0, 4.2 Hz, 1H), 5.23 (t, *J* = 14.6 Hz, 2H), 4.99 (d, *J* = 11.6 Hz, 2H), 4.77 (d, *J* = 11.6 Hz, 2H), 4.40 (d, *J* = 6.0 Hz, 2H), 4.16 – 4.04 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.3, 136.5, 129.9, 128.6, 128.3, 119.3, 78.8, 73.5, 40.7;

Meso-2-allyl-tetrahydrocyclopenta[c]pyrrole-1,3(2H,3aH)-dione **12o.** The title compound prepared following procedure A was isolated (247 mg, 46%) as a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.86 – 5.66 (m, 1H), 5.26 – 5.06 (m, 2H), 4.07 (dt, *J* = 5.8, 1.2 Hz, 2H), 3.17 (d, *J* = 8.9 Hz, 2H), 2.23 – 2.07 (m, 2H), 1.99 – 1.83 (m, 2H), 1.76 (ddd, *J* = 9.6, 8.3, 4.2 Hz, 1H), 1.40 – 1.20 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.8, 130.8, 118.1, 45.2, 40.9, 30.5, 24.8.

*Meso-hexahydro-2-(2-methylallyl)-2*H-*isoindole-1,3-dione* **12r.** The title compound prepared following procedure B was isolated (565 mg, 92%) as a white solid, Mp=47-49 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.87 (s, 1H), 4.76 (s, 1H), 4.02 (s, 2H), 2.89 (t, *J* = 4.5 Hz, 2H), 1.97 – 1.84 (m, 2H), 1.83 – 1.74 (m, 2H), 1.74 (s, 3H), 1.46 (dd, *J* = 12.1, 6.8 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.4, 138.9, 112.1, 43.5, 39.7, 23.8, 21.7, 20.5.

*Meso -3a,4,7,7a-Tetrahydro-2-(2-methylallyl)-2*H*-isoindole-1,3-dione* **12q.** The title compound prepared following procedure B was isolated (498 mg, 81%) as a white solid, Mp=60-62 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.07 – 5.81 (m, 2H), 4.83 (s, 1H), 4.70 (s, 1H), 4.00 (s, 2H), 3.26 – 3.04 (m, 2H), 2.65 (ddd, J = 16.0, 3.7, 1.6 Hz, 2H), 2.36 – 2.14 (m, 2H), 1.69 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.8, 138.4, 127.9, 111.9, 44.2, 39.1, 23.6, 20.3.

General procedure for synthesis of tertiary alcohols

Vinyl magnesium bromide (1.5 mmol, 1*M* solution in THF) was added dropwise to a solution of *N*-allyl imide (1.0 mmol) in dry THF (10 mL) under N_2 atmosphere at –5-0 ˚C. The mixture was stirred at the same temperature until the completion of the reaction, as determined by TLC. Saturated NH_4Cl solution (5 mL) was then added to the mixture and extracted with diethyl ether (2 x 25 mL). The combined organic phases were dried (anhydrous Na_2SO_4), filtered and the solvent removed under reduced pressure. The residue was purified by flash chromatography (SiO_2, petroleum ether-ether) to afford the products.

(5RS)-1-Allyl-5-hydroxy-5-vinylpyrrolidin-2-one **6.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, diethyl ether) afforded the product **6** (67 mg, 40%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.93 – 5.82 (m, 2H), 5.49 (dd, J = 17.2, 0.7 Hz, 1H), 5.35 (dd, J = 10.6, 0.7 Hz, 1H), 5.23 – 5.10 (m, 2H), 4.13 – 4.02 (m, 1H), 3.65 (dd, J = 15.5, 7.0 Hz, 1H), 2.60 (dd, J = 9.0, 8.2 Hz, 1H), 2.42-2.36 (m, 1H), 2.22 – 2.02 (m, 2H), 1.64 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.8, 139.3, 134.5, 117.4, 116.8, 91.4, 42.0, 34.4, 28.9; IR (ATR): ν 3303, 1667, 1403, 989, 924 cm^{-1}; MS (EI): *m/z* 167.0 [M]$^+$, 149.0, 137.0, 111.0, 83.0, 56.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_9H_{13}NO_2$ + H^+]: 168.1025, found 168.1019.

(3RS)-2-Allyl-3-hydroxy-3-vinylisoindolin-1-one **9.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **9** (164 mg, 76%) as a white solid, mp 94-96 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (dd, J = 12.6, 11.4 Hz, 1H), 7.55 (td, J = 7.2, 1.1 Hz, 1H), 7.45 (dd, J = 11.0, 4.1 Hz, 2H), 5.93 – 5.77 (m, 2H), 5.67 (dd, J = 17.0, 10.3 Hz, 1H), 5.42 (dd, J = 10.3, 1.4 Hz, 1H), 5.27 – 5.03 (m, 2H), 4.14 – 3.94 (m, 1H), 3.85 – 3.71 (m, 1H), 3.83 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 146.7, 135.8, 134.01 132.5, 130.7, 129.7, 123.4, 122.6, 118.1, 117.3, 89.7, 41.3; IR (ATR): ν 3273, 1669, 1429, 1394, 927, 772, 698 cm^{-1}; MS (EI): *m/z* 215.0 [M]$^+$, 198.0,

188.0, 159.0, 131.0, 115,0 103.0, 77.0, 56.1; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{13}H_{13}NO_2$ + H^+]: 216.1025, found 216.1019.

(3RS,3aRS,7aSR)-2-Allyl-octahydro-3-hydroxy-3-vinylisoindol-1-one **13a.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **13a** (111 mg, 50%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 5.95 – 5.77 (m, 2H), 5.32 (dd, *J* = 13.6, 7.6 Hz, 2H), 5.21 (dd, *J* = 17.2, 1.5 Hz, 1H), 5.12 (dd, *J* = 10.2, 1.2 Hz, 1H), 4.12 (dd, *J* = 15.5, 5.0 Hz, 1H), 3.56 (dd, *J* = 15.5, 6.9 Hz, 1H), 2.52 (dd, *J* = 13.2, 6.5 Hz, 1H), 2.23-2.21 (m, 2H), 2.01 – 1.91 (m, 1H), 1.68 (dd, *J* = 14.4, 7.8 Hz, 3H), 1.39-1.33 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 138.0, 135.1, 117.0, 116.4, 92.5, 41.9, 41.8, 40.0, 24.2, 23.19, 23.2, 22.3; IR (ATR): *v* 3231, 2936, 1656, 1434, 924, 689 cm^{-1}; MS (EI): *m/z* 221.1 $[M]^+$, 203.1, 188.0, 174.1, 162.0, 139.0, 91.0, 81.0, 55.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{13}H_{19}NO_2$ + H^+]: 222.1494, found 222.1488.

(3RS,3aRS,7aSR)-2-Allyl-2,3,3a,4,7,7a-hexahydro-3-hydroxy-3-vinylisoindol-1-one **13b.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **13b** (183 mg, 91%) as a white solid, mp 57-59 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 5.91 – 5.70 (m, 4H), 5.50 (dd, *J* = 17.1, 1.0 Hz, 1H), 5.35 (dd, *J* = 10.5, 1.0 Hz, 1H), 5.13 (ddd, *J* = 13.7, 11.5, 1.4 Hz, 2H), 3.98 (ddt, *J* = 15.5, 5.7, 1.4 Hz, 1H), 3.71 (dd, *J* = 15.5, 6.3 Hz, 1H), 2.74 (s, 1H), 2.68 (dd, *J* = 15.6, 8.5 Hz, 1H), 2.46 (ddd, *J* = 16.3, 9.5, 4.3 Hz, 2H), 2.37 – 2.20 (m, 1H), 2.19 – 2.07 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.4, 138.6, 134.5, 126.3, 125.8, 117.5, 117.0, 92.4, 42.1, 39.5, 36.9, 23.9, 19.8; IR (ATR): *v* 3343, 1660, 1412, 1095, 929 cm^{-1}; MS (EI): *m/z* 201.1 $[M\text{-}OH]^+$, 186.0, 172.0, 158.0, 115.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$(C_{13}H_{17}NO_2 + H)^+ – H_2O$]: 202.1232, found 202.1229.

*(3RS,3aRS,4SR,,7RS,7aSR)-2-Allyl-3-hydroxy-3-vinyl-2,3,3a,4,7,7a-hexahydro-1*H-*4,7-methanoisoindol-1-one* **13c. C**ompound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **13c** (220 mg, 95%) as a white solid, mp 98-99 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 6.26 (d, *J* = 3.0 Hz, 2H), 5.85 – 5.66 (m, 2H), 5.31 (d, *J* = 17.2 Hz, 1H), 5.25 – 5.11 (m, 2H), 5.05 (dd, *J* = 10.2, 1.3 Hz, 1H), 4.05 – 3.86 (m, 1H), 3.40 (dd, *J* = 15.6, 6.5 Hz, 1H), 3.37 – 3.28 (m, 1H), 3.19 (dd, *J* = 9.0, 4.8 Hz, 1H), 3.12 (d, *J* = 1.2 Hz, 1H), 2.88 (dd, *J* = 9.0, 4.0 Hz, 1H), 1.61 (dd, *J* = 6.5, 1.7 Hz, 2H), 1.41 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.5, 140.8,

136.5, 134.9, 133.7, 116.6, 114.5, 90.4, 51.9, 49.6, 48.6, 45.8, 45.0, 42.0; IR (ATR): *v* 3189, 2986, 1648, 1440, 1406, 1130, 945, 686 cm^{-1}; MS (EI): *m/z* 231.1 [M]$^+$, 213.0, 147.0, 134.0, 91.0, 66.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{14}H_{17}NO_2$ + H$^+$]: 232.1338, found 232.1329.

(3RS)-2-Allyl-5,6-dichloro-3-hydroxy-3-vinylisoindolin-1-one **13d.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 7:3 v/v petroleum ether-diethyl ether) afforded the product **13d** (246 mg, 87%) as a white solid, mp 101-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.52 (s, 1H), 5.95 – 5.76 (m, 2H), 5.62 (ddd, *J* = 16.9, 10.4, 1.1 Hz, 1H), 5.48 (dd, *J* = 10.5, 0.9 Hz, 1H), 5.18 (ddd, *J* = 13.6, 11.3, 1.2 Hz, 2H), 4.06 (dd, *J* = 15.6, 6.1 Hz, 1H), 3.86 (dd, *J* = 15.6, 6.1 Hz, 1H), 3.63 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.0, 145.8, 137.1, 134.8, 134.7, 133.3, 130.3, 125.1, 125.1, 119.2, 117.9, 89.1, 41.7; IR (ATR): *v* 3324, 1674, 1431, 1405, 1279, 931, 892, 773 cm^{-1}; MS (EI): *m/z* 283.0 [M]$^+$, 267.9, 255.9, 226.9, 197.9, 173.0, 165.0, 136.0, 56.1; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{13}H_{11}Cl_2NO_2$ + H$^+$]: 284.0245, found 284.0239.

(3RS)-2-Allyl-4,5,6,7-tetrachloro-3-hydroxy-3-vinylisoindolin-1-one **13e.** Compound **13e** was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 7:3 v/v petroleum ether-diethyl ether) afforded the product **13e** (226 mg, 64%) as a white solid, mp 138-140 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.93 – 5.77 (m, 2H), 5.68 – 5.55 (m, 2H), 5.25 (dd, *J* = 17.1, 1.3 Hz, 1H), 5.18 (dd, *J* = 10.1, 1.1 Hz, 1H), 4.11 (dd, *J* = 15.5, 6.2 Hz, 1H), 3.96 (dd, *J* = 15.5, 6.2 Hz, 1H), 3.64 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.6, 143.1, 137.9, 135.9, 132.9, 132.6, 129.1, 128.5, 127.1, 121.1, 118.4, 88.5, 41.9; IR (ATR): *v* 3320, 1683, 1432, 1403, 1354, 1204, 944, 732 cm^{-1}; MS (EI): *m/z* 352.9 [M]$^+$, 283.8, 267.8, 241.8, 213.8, 69.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{13}H_9Cl_4NO_2$ + H$^+$]: 353.9436, found 353.9430.

(5RS)-1-Allyl-5-hydroxy-3,3-dimethyl-5-vinylpyrrolidin-2-one **13f.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 6:4 v/v petroleum ether-diethyl ether) afforded the product **13f** (142 mg, 73%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.92 – 5.76 (m, 2H), 5.49 (d, *J* = 17.1 Hz, 1H), 5.30 (d, *J* = 10.6 Hz, 1H), 5.23 – 5.03 (m, 2H), 3.97 (dd, *J* = 15.5, 5.5 Hz, 1H), 3.82 – 3.75 (m, 1H), 3.70 (dd, *J* = 15.5, 6.4 Hz, 1H), 2.06 (d, *J* = 3.2 Hz, 2H), 1.30 (s, 3H), 1.18 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.0, 140.5, 134.6, 117.1, 116.3, 88.9, 49.3, 42.1, 39.6, 26.6, 26.3; IR (ATR): *v* 3352, 1663, 1408, 1199, 987, 922 cm^{-1}; MS (EI): *m/z* 195.0 [M]$^+$, 177.1, 162.0, 148.0, 134.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{11}H_{17}NO_2$ + H$^+$]: 196.1338, found 196.1332.

(3RS)-2-Allyl-3-hydroxy-3-vinyl-2,3-dihydro-1H-benzo[de]isoquinolin-1-one **13g.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **13g** (177 mg, 67 %) as a white solid, mp 128-130 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 7.2, 1.1 Hz, 1H), 7.91 (dd, *J* = 8.2, 0.9 Hz, 1H), 7.87 – 7.79 (m, 1H), 7.71 (dd, *J* = 7.3, 0.9 Hz, 1H), 7.60 – 7.51 (m, 1H), 7.42 (dd, *J* = 8.1, 7.4 Hz, 1H), 5.96 (ddd, *J* = 17.2, 5.6, 4.6 Hz, 1H), 5.87 (ddd, *J* = 17.0, 10.4, 1.3 Hz, 1H), 5.67 (dd, *J* = 17.0, 1.1 Hz, 1H), 5.26 (dd, *J* = 10.3, 1.1 Hz, 1H), 5.19 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.09 (dd, *J* = 10.2, 1.4 Hz, 1H), 4.47 – 4.30 (m, 1H), 4.22 – 4.10 (m, 1H), 3.97 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.5, 140.1, 135.3, 133.7, 131.9, 131.8, 128.2, 127.4, 126.6, 126.4, 126.2, 125.5, 116.4, 114.7, 87.7, 45.0; IR (ATR): *v* 3250, 1634, 1607, 1440, 1582, 1396, 988, 781 cm^{-1}; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{17}H_{15}NO_2 + H^+$]: 266.1181, found 266.1176.

(7RS)-6-Allyl-6,7-dihydro-7-hydroxy-7-vinylpyrrolo[3,4-b]pyridin-5-one **13h.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 4:1 v/v diethyl ether-petroleum ether) afforded the product **13h** (160 mg, 74%) as a white solid, mp 125-127 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, *J* = 4.8 Hz, 1H), 8.04 (d, *J* = 7.5 Hz, 1H), 7.37 (dd, *J* = 7.6, 5.0 Hz, 1H), 6.04 – 5.83 (m, 2H), 5.76 (dd, *J* = 17.0, 10.2 Hz, 1H), 5.55 (d, *J* = 10.2 Hz, 1H), 5.27 (d, *J* = 16.5 Hz, 1H), 5.17 (d, *J* = 10.1 Hz, 1H), 4.21 (dd, *J* = 15.6, 5.8 Hz, 2H), 3.99 (dd, *J* = 15.7, 5.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.1, 165.0, 152.9, 134.4, 133.7, 132.0, 125.0, 124.6, 119.9, 117.6, 89.5, 41.6; IR (ATR): *v* 3265, 1667, 1396, 986, 798, 729 cm^{-1}; MS (EI): *m/z* 216.0 [M]$^+$, 189.0, 175.0, 160.0, 104.0, 78.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{12}H_{12}N_2O_2 + H^+$]: 217.0977, found 217.0972.

(3RS)-2-Allyl-4,7-bis(allyloxy)-3-hydroxy-3-vinylisoindolin-1-one **13i.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **13i** (255 mg, 78%) as a pale yellow solid, mp 117-120 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92 (d, *J* = 8.9 Hz, 1H), 6.85 (d, *J* = 8.9 Hz, 1H), 6.14 – 5.87 (m, 3H), 5.80 (dd, *J* = 17.1, 10.3 Hz, 1H), 5.65 (dd, *J* = 17.1, 1.0 Hz, 1H), 5.49 (dd, *J* = 17.3, 1.5 Hz, 1H), 5.44-5.38 (m, 2H), 5.31 – 5.19 (m, 3H), 5.11 (dd, *J* = 10.2, 1.3 Hz, 1H), 4.64 (dt, *J* = 5.0, 1.4 Hz, 2H), 4.59 – 4.48 (m, 2H), 4.13 (dd, *J* = 15.7, 6.0 Hz, 1H), 3.86 (dd, *J* = 15.7, 6.0 Hz, 1H), 3.02 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.3, 150.0, 147.6, 135.7, 135.6, 134.4, 133.1, 132.8, 119.7, 117.7 (2C) 117.5, 116.9, 115.9, 88.3, 70.4, 69.6, 41.3; IR (ATR): *v* 3297, 1672,

1497, 1267, 918, 807 cm^{-1}; MS (EI): *m/z* 327.0 [M]$^+$, 206.9, 54.9; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{19}H_{21}NO_4$ + H$^+$]: 328.1549, found 328.1545.

1-Allyl-5-hydroxy-3-methyl-5-vinylpyrrolidin-2-one **13j.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the major regioisomer **13j** (80 mg, 44%). Stereochemistry of this product could not be determined by standard NMR experiments. Other regioisomer was isolated as a complex mixture and attempts were not made to further purify and characterize it. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.93 – 5.77 (m, 2H), 5.37 (d, *J* = 17.2 Hz, 1H), 5.24 (d, *J* = 10.6 Hz, 1H), 5.17 (dd, *J* = 17.2, 1.4 Hz, 1H), 5.08 (dd, *J* = 10.2, 1.2 Hz, 1H), 4.44 – 4.27 (m, 1H), 3.96 (dd, *J* = 15.5, 5.9 Hz, 1H), 3.67 (dd, *J* = 15.5, 6.0 Hz, 1H), 2.46 (dt, *J* = 17.2, 7.9 Hz, 2H), 1.82 (dd, *J* = 12.3, 6.6 Hz, 1H), 1.36 – 1.22 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.8, 138.9, 134.5, 116.9, 115.5, 89.9, 43.0, 42.1, 35.3, 16.8; IR (ATR): *v* 3320, 1664, 1408, 988, 923 cm^{-1}; MS (EI): *m/z* 181.1 [M]$^+$, 163.0, 148.0, 134.0, 120.0, 107.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{10}H_{15}NO_2$ + H$^+$]: 182,1181, found 182.1173.

1-Allyl-5-hydroxy-3-phenyl-5-vinylpyrrolidin-2-one **13k.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 9:1 v/v diethyl ether-petroleum ether) afforded the alcohol **13k** (73 mg, 30%) as an inseparable mixture of two regioisomers (7.3 : 1, pale yellow oil). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.15 (m, 5H, both regioisomers), 5.99 – 5.88 (m, 2H, major regioisomer), 5.86-5.80 (m, 1H, minor regioisomer), 5.47 (d, J= 17.2 Hz, 1H, major regioisomer), 5.36 (d, *J* = 10.5 Hz, 1H, major regioisomer), 5.25 (d, J=4.0 Hz, 1H, major), 5.23 (m, 2H, major regioisomer), 4.19 – 4.16 (m, 2H, major regioisomer), 4.04 (dd, J = 9.5, 4.7 Hz, 1H, minor), 3.77-3.34 (m, 1H, both regioisomers), 3.23 (dd, J = 18.5, 9.6 Hz, 1H, minor regioisomer), 2.54 (dd, *J* = 13.5, 8.5 Hz, 1H, major regioisomer), 2.18 (dd, *J* = 13.5, 10.1 Hz, 1H, major regioisomer); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.9, 139.0, 138.8, 134.7, 129.2, 128.8, 128.0, 127.3, 127.2, 118.6, 117.5, 116.5, 90.0, 46.4, 43.2, 42.4, 41.2, 37.1, 30.3, 29.7; IR (ATR): *v* 3372, 1698, 1665, 1395, 1334, 925, 697 cm^{-1}; MS (EI): *m/z* [M]$^+$ 243.1, 225.0, 210.0, 188.0, 160.0, 145.0, 131.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{15}H_{17}NO_2$ + H$^+$]: 244,1338, found 244.1327.

(3RS)-2-Allyl-2,3-dihydro-3-hydroxy-3-vinylpyrrolo[3,4-b]quinolin-1-one **13l.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 3:2 v/v diethyl ether-petroleum ether) afforded the product **13l** (146 mg, 55%) as a white solid, mp 146-148 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (s, 1H), 8.07 (d, *J* = 8.5

Hz, 1H), 7.77 (dd, *J* = 16.6, 8.0 Hz, 2H), 7.57 (t, *J* = 7.5 Hz, 1H), 6.14 – 5.92 (m, 2H), 5.84 (dd, *J* = 17.0, 10.2 Hz, 1H), 5.58 (d, *J* = 10.3 Hz, 1H), 5.34 (d, *J* = 17.1 Hz, 1H), 5.22 (d, *J* = 10.1 Hz, 1H), 4.61 (s, 1H), 4.31 (dd, *J* = 15.5, 6.3 Hz, 1H), 4.09 (dd, *J* = 15.5, 5.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 163.5, 149.4, 134.7, 133.8, 132.5, 131.7, 129.4, 129.3, 127.9, 127.7, 122.4, 119.9, 117.8, 89.7, 41.9; IR (ATR): *v* 3179, 1665, 1629, 1431, 1403, 1156, 760 cm^{-1}; MS (EI): *m/z* 266.0 [M]$^+$, 250.0, 239.0, 224.9, 211.0, 128.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{16}H_{14}N_2O_2$ + H$^+$]: 267.1134, found 267.1126.

(3SR,4RS,5RS)-1-Allyl-3,4-bis(benzyloxy)-5-hydroxy-5-vinylpyrrolidin-2-one **13m.** Two products were prepared from *N*-allyl-*meso*-tartrimide following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 6:4 v/v diethyl ether-petroleum ether) afforded two diastereomeric products **13m** (125 mg, 33%) and **13m’** (163 mg, 43%). Compound **13m** was isolated as a white solid, mp 55-57 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 6.97 (m, 10H), 5.85 (tt, *J* = 10.2, 8.6 Hz, 2H), 5.62 – 5.51 (m, 1H), 5.45 (d, *J* = 10.7 Hz, 1H), 5.20 (dd, *J* = 17.2, 1.3 Hz, 1H), 5.13 (dd, *J* = 10.2, 1.2 Hz, 1H), 5.00 (d, *J* = 11.8 Hz, 1H), 4.77 (d, *J* = 11.8 Hz, 1H), 4.68 (d, *J* = 11.9 Hz, 1H), 4.57 (d, *J* = 11.8 Hz, 1H), 4.07 (d, *J* = 5.3 Hz, 1H), 4.00 (dd, *J* = 15.6, 5.6 Hz, 1H), 3.94 (d, *J* = 5.4 Hz, 1H), 3.66 (dd, *J* = 15.5, 6.2 Hz, 1H), 2.80 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 137.3, 137.3, 134.4, 133.9, 128.5, 128.1, 128.0, 127.9, 127.8, 118.7, 117.4, 90.6, 86.9, 78.7, 72.9, 72.6, 41.7; IR (ATR): *v* 3305, 1670, 1068, 1392, 948, 927, 697 cm^{-1}; MS (EI): *m/z*, 361.1 [M-H_2O]$^+$, 324.1, 288.1, 270.1, 182.0, 167.0, 91.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{23}H_{25}NO_4$ + H$^+$]: 380.1862, found 380.1855.

(3SR,4RS,5SR)-1-Allyl-3,4-bis(benzyloxy)-5-hydroxy-5-vinylpyrrolidin-2-one **13 m’** Compound **13m’** was isolated as a white solid, mp 48-50 °C. ^{1}H NMR (400 MHz, CDCl3) δ 7.43 – 7.23 (m, 10H), 5.87 – 5.63 (m, 3H), 5.42 (dd, *J* = 9.8, 1.8 Hz, 1H), 5.21 – 5.05 (m, 3H), 4.79 (d, *J* = 11.7 Hz, 1H), 4.70 – 4.59 (m, 2H), 4.20 (d, *J* = 6.1 Hz, 1H), 3.95 – 3.85 (m, 2H), 3.76 (dd, *J* = 15.6, 5.6 Hz, 1H), 3.54 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 137.6, 137.5, 136.6, 133.5, 128.6, 128.4, 128.3, 128.2, 128.0, 127.9, 119.3, 117.2, 86.8, 82.5, 78.4, 73.2, 72.8, 42.3; IR (ATR): *v* 3408, 1698, 1685, 1452, 1103, 934, 734 cm^{-1}; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{23}H_{25}NO_4$ + H$^+$]: 380.1862, found 380.1852.

(3R,4R,5R)-1-Allyl-3,4-bis(benzyloxy)-5-hydroxy-5-vinylpyrrolidin-2-one **13n.** Compound **13n** was synthesized from *N*-allyl-*L*-tartrimide following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product

13n (261 mg, 69%) as a white solid, mp 37-40 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.18 (m, 10H), 5.88 – 5.63 (m, 3H), 5.42 (dd, *J* = 9.7, 1.9 Hz, 1H), 5.21 – 5.05 (m, 3H), 4.79 (d, *J* = 11.7 Hz, 1H), 4.64 (d, *J* = 5.1 Hz, 2H), 4.20 (d, *J* = 6.1 Hz, 1H), 3.97 – 3.85 (m, 2H), 3.81 – 3.71 (m, 1H), 3.55 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 137.6, 137.5, 136.6, 133.5, 128.6, 128.4, 128.3, 128.2, 128.0, 127.9, 119.3, 117.2, 86.8, 82.5, 78.4, 73.2, 72.8, 42.3; IR (ATR): *ν* 3408, 1698, 1686, 1102, 735, 697 cm^{-1}; MS (EI): *m/z* 361.1 $[M-H_2O]^+$, 324.0, 270.0, 242.0, 167.0, 91.0; HRMS (ESI/Q-TOF) *m/z* calcd for $[C_{23}H_{25}NO_4 + H^+]$: 380.1862, found 380.1855.

(3RS,3aRS,6aSR)-2-Allyl-hexahydro-3-hydroxy-3-vinylcyclopenta[c]pyrrol-1(2H)-one **13o** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **13o** (160 mg, 77%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.85 (dt, *J* = 17.1, 10.3 Hz, 2H), 5.31 (d, *J* = 17.2 Hz, 1H), 5.17 (dd, *J* = 22.9, 5.9 Hz, 2H), 5.08 (dd, *J* = 10.2, 1.3 Hz, 1H), 3.97 (dd, *J* = 15.5, 5.8 Hz, 1H), 3.67 (s, 1H), 3.61 (dd, *J* = 15.6, 6.0 Hz, 1H), 2.92 (td, *J* = 9.1, 3.5 Hz, 1H), 2.74 – 2.62 (m, 1H), 2.11 – 1.99 (m, 1H), 1.94 (dd, *J* = 11.9, 6.2 Hz, 1H), 1.88 – 1.80 (m, 1H), 1.75 – 1.55 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.36, 140.6, 134.6, 116.6, 114.5, 90.9, 48.4, 46.6, 42.1, 29.5, 27.2, 26.1; IR (ATR): *ν* 3322, 2955, 1660, 1408, 989, 928 cm^{-1}; MS (EI): *m/z* 207.1 $[M]^+$, 189.1, 160.0, 132.0, 106.0, 91.0; HRMS (ESI/Q-TOF) *m/z* calcd for $[C_{12}H_{17}NO_2 + H^+]$: 208.1338, found 208.1327.

(3RS)-3-Hydroxy-2-(2-methylallyl)-3-vinylisoindolin-1-one **13p.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 6:4 v/v diethyl ether- petroleum ether) afforded the product **13p** (160 mg, 70%) as a pale yellow solid, mp 115-116 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.5 Hz, 1H), 7.55 (dd, *J* = 7.4, 0.9 Hz, 1H), 7.46 (dd, *J* = 14.7, 7.4 Hz, 2H), 5.87 – 5.58 (m, 2H), 5.41 (dd, *J* = 9.7, 1.9 Hz, 1H), 4.83 (d, *J* = 12.2 Hz, 2H), 4.08 (d, *J* = 15.9 Hz, 1H), 3.73 (d, *J* = 16.0 Hz, 1H), 3.26 (s, 1H), 1.74 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 146.5, 142.1, 135.9, 132.5, 130.7, 129.8, 123.6, 122.6, 118.1, 112.1, 89.9, 44.5, 20.4; IR (ATR): *ν* 3245, 1671, 1431, 1392, 765, 690 cm^{-1}; MS (EI): *m/z* 229.1 $[M]^+$, 212.1, 196.0, 182.0, 159.0, 131.0, 103.0; HRMS (ESI/Q-TOF) *m/z* calcd for $[C_{14}H_{15}NO_2 + H^+]$: 230.1181, found 230.1175.

(3RS,3aRS,7aSR)-2,3,3a,4,7,7a-Hexahydro-3-hydroxy-2-(2-methylallyl)-3-vinylisoindol-1-one **13q.** Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 4:1 v/v diethyl ether- petroleum ether) afforded the product **13q** (158 mg,

68%) as a white solid, mp 57-59 °C. ^{1}H NMR (400 MHz, CDCl$_3$) δ 5.96 – 5.70 (m, 3H), 5.48 (dd, *J* = 17.1, 0.9 Hz, 1H), 5.36 (dd, *J* = 10.5, 0.9 Hz, 1H), 4.82 (d, *J* = 9.3 Hz, 2H), 4.02 (d, *J* = 15.4 Hz, 1H), 3.60 (d, *J* = 15.5 Hz, 1H), 2.69 (dd, *J* = 16.2, 8.2 Hz, 1H), 2.58 – 2.38 (m, 3H), 2.37 – 2.20 (m, 1H), 2.20 – 2.10 (m, 1H), 2.19 – 2.08 (m, 1H), 1.74 (s, 3H); ^{13}C NMR (101 MHz, CDCl$_3$) δ 177.6, 143.1, 138.6, 126.3, 125.6, 117.6, 112.2, 92.6, 45.1, 39.3, 37.0, 24.2, 20.4, 19.7; IR (ATR): *v* 3207, 3024, 1656, 1640, 1423, 1130, 919, 900, 661 cm^{-1}; MS (EI): *m/z* 233.1 [M]$^+$, 215.1, 200.0, 186.0, 160.0, 144.0, 117.0, 91.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{14}H_{19}NO_2$ + H$^+$] : 234.1494, found 234.1487.

(3RS,3aRS,7aSR)-Octahydro-3-hydroxy-2-(2-methylallyl)-3-vinylisoindol-1-one **13r**. Compound was synthesized following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO$_2$, 7:3 v/v diethyl ether- petroleum ether) afforded the product **13r** (160 mg, 68%) as a white solid, mp 77-79 °C. ^{1}H NMR (400 MHz, CDCl$_3$) δ 5.81 (dd, *J* = 17.2, 10.5 Hz, 1H), 5.32 (dd, *J* = 13.9, 6.5 Hz, 2H), 4.85 (d, *J* = 7.5 Hz, 2H), 4.13 (d, *J* = 15.5 Hz, 1H), 3.44 (d, *J* = 15.5 Hz, 1H), 2.52 (q, *J* = 6.8 Hz, 1H), 2.21 (dd, *J* = 13.6, 6.9 Hz, 1H), 2.03 – 1.89 (m, 1H), 1.77 – 1.62 (m, 6H), 1.53 – 1.38 (m, 3H), 1.42 – 1.24 (m, 2H); ^{13}C NMR (101 MHz, CDCl$_3$) δ 176.2, 143.8, 138.0, 116.4, 112.0, 92.9, 45.0, 41.6, 40.1, 24.5, 23.4, 23.1, 22.3, 20.5; IR (ATR): *v* 3206, 2937, 1655, 1639, 1434, 957 cm^{-1}; MS (EI): *m/z* 235.1 [M]$^+$, 217.0, 188.1, 160.0, 134.0, 91.0; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{14}H_{21}NO_2$ + H$^+$]: 236.1651, found 236.1645.

General procedure for hydrolysis of pyrrolizinones

To a solution of pyrrolizinone (0.2 mmol) in THF (2 mL), 1*N* solution of LiOH (0.8 mmol, 4 eq) was added, and the mixture refluxed for 1h. After completion of the reaction (determined by TLC), the mixture was cooled to room temperature, acidified with 1*M* HCl to pH~2 and extracted with ethyl acetate (2 x 20 mL). The combined organic phases were dried with anhydrous Na$_2$SO$_4$, filtered and solvent was removed under reduced pressure. The residue was purified by flash chromatography to afford the products.

3-(1H-pyrrol-2-yl) propanoic acid **15a**[3] Compound was synthesized from pyrrolizinone **7** following the general procedure for pyrrolizinone hydrolysis. Flash chromatography (SiO$_2$, 1:1 v/v petroleum ether-diethyl ether) afforded the product **15a** (21.4 mg, 77%) as a white solid, mp 109-111 °C. ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.36 (s, 1H), 6.68 (dd, *J* = 4.0, 2.6 Hz, 1H), 6.12 (dd, *J* = 5.7, 2.8 Hz, 1H), 5.94 (s, 1H), 2.93 (t, *J* = 6.8 Hz, 2H), 2.72 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (101 MHz, CDCl$_3$) δ

178.2, 130.5, 116.9, 108.2, 105.6, 34.0, 22.4; IR (ATR): *ν* 3355, 2881, 1669, 1407, 1237, 756, 733 cm^{-1}. HRMS (ESI/Q-TOF) *m/z* calculated for [$C_7H_9NO_2$ + H^+]: 140.0712, found 140.0715.

2-(1H-Pyrrol-2-yl)benzoic acid **15b**[4] Compound was synthesized from pyrrolizinone **11** following the general procedure for hydrolysis of pyrrolizinone. Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **15b** (24 mg, 64%) as a pale yellow crystals, mp 138-141 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 7.95 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.70 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.54 (ddd, *J* = 8.8, 7.7, 1.4 Hz, 1H), 7.34 – 7.27 (m, 1H), 6.92 (dd, *J* = 4.2, 2.6 Hz, 1H), 6.59 – 6.50 (m, 1H), 6.31 (dd, *J* = 6.0, 2.6 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 134.1, 132.7, 131.5, 130.3, 126.2, 126.0, 119.4, 110.2, 109.5; IR (ATR): *ν* 3355, 2881,1669, 1407, 1237, 734 cm^{-1}; HRMS (ESI/Q-TOF) *m/z* calcd for [$C_{11}H_9NO_2$ + H^+]: 188.0712, found 188.0714.

4,5-dichloro-2-(1H-pyrrol-2-yl) benzoic acid **15c.** Compound was synthesized from pyrrolizinone **14d** following the general procedure for pyrrolizinone hydrolysis. Flash chromatography (SiO_2, 4:1 v/v diethyl ether-petroleum ether) afforded the product **15**c (37.7 mg, 74%) as a yellow solid, mp 141-146 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.19 (s, 1H), 8.06 (s, 1H), 7.79 (s, 1H), 6.94 (d, *J* = 1.4 Hz, 1H), 6.57 (s, 1H), 6.31 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 137.5, 134.1, 133.5, 131.8, 129.8, 128.0, 124.9, 120.5, 111.6, 110.0; IR (ATR): *ν* 3378, 2849, 2357, 1675, 1308, 1244, 726, 683 cm^{-1}; HRMS (ESI/Q-TOF) *m/z* calculated for [$C_{11}H_7Cl_2NO_2$ + H^+]: 255.9932, found 255.9946.

Derivatization of pyrrolizinones

5H-pyrrolo[2,1-a]isoindole-5-thione **16**. To a solution of pyrrolizinone **11** (20 mg, 0.12 mmol) in dry toluene (2 mL) Lawesson's reagent (48.5 mg, 0.12 mmol) was added under nitrogen atmosphere and the mixture was refluxed overnight. Toluene was removed under reduced pressure and the residue was purified by flash chromatography (SiO_2, 95:5 v/v petroleum ether-diethyl ether) to afford the products **16** (22 mg, 99%) as a brownish red crystals, mp 62-63 ˚C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.12 (dd, *J* = 11.9, 5.1 Hz, 2H), 6.17 (d, *J* = 2.9 Hz, 1H), 6.08 (t, *J* = 3.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.5, 140.4, 138.0, 134.4, 133.9, 127.3, 126.0, 118.9, 117.8, 117.0, 107.9; IR (ATR): *ν* 1615, 1435, 1397, 1288, 1072, 832, 766, 678 cm^{-1}; MS (EI): *m/z* 185.0 [M]$^+$, 158.0, 140.0, 127.0, 114.0.

(Z/E)-Ethyl 2-(5H-pyrrolo[2,1-a]isoindol-5-ylidene)acetate **17.** To a solution of pyrrolizinone **11** (20 mg, 0.12 mmol) in dry toluene (3 mL) (ethyloxycarbonylmethylene)triphenylphosphorane (49.1 mg, 0.14 mmol) was added under nitrogen atmosphere and the mixture was refluxed for four days. Toluene was removed under reduced pressure, and the residue was purified by flash chromatography (SiO_2, 9:1 v/v petroleum ether-diethyl ether) to afford the product **17** (11.7 mg, 41%; *Z/E* ratio: 3:1) as a yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 8.90 (d, *J* = 7.9 Hz, 1H, *E*-isomer), 8.34 (d, *J* = 3.0 Hz, 1H *Z*-isomer), 7.50 (d, *J* = 7.8 Hz, 1H *Z*-isomer), 7.34 (dd, *J* = 8.5, 6.3 Hz, 2H *Z*-isomer +2H *E*-isomer), 7.21 – 7.09 (m, 1H *Z*-isomer +1H *E*-isomer), 6.98 (d, *J* = 2.9 Hz, 1H *E*-isomer), 6.29 – 6.21 (m, 1H *Z*-isomer +2H *E*-isomer), 6.21 – 6.15 (m, 1H, *Z*-isomer), 6.09 (s, 1H *E*-isomer), 5.96 (s, 1H *Z*-isomer), 4.27 (q, *J* = 7.1 Hz, 2H *Z*-isomer +2H *E*-isomer), 1.36 (s, 3H *Z*-isomer +3H *E*-isomer); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.2, 165.6, 143.9, 138.0, 136.9, 134.7, 134.5, 134.0, 133.1, 131.7, 131.3, 129.4, 127.1, 126.5, 125.7, 123.3, 121.6, 118.8, 118.7, 117.2, 115.7, 115.0, 114.2, 107.3, 103.6, 103.1, 99.1, 94.6, 60.3, 14.4; IR (ATR): *ν* 1708, 1644, 1615, 1220, 1156, 752, 724 cm^{-1}; MS (EI): *m/z* 239.0 $[M]^+$, 211.0, 194.0, 167.0, 139.0, 96.9, 69.5; HRMS (ESI/Q-TOF) *m/z* calcd for $[C_{15}H_{13}NO_2 + H^+]$: 240.1025, found 240.1017.

(RS)-5-Vinyl-5H-pyrrolo[2,1-a]isoindol-5-ol **18.** Compound was synthesized from pyrrolizinone **11** (20 mg, 0.12 mmol) and vinyl magnesium bromide (0.36 mmol) following the general procedure for synthesis of tertiary alcohols**.** Flash chromatography (SiO_2, 1:1 v/v petroleum ether-diethyl ether) afforded the product **18** (15.8 mg, 67%) as pale yellow crystals, mp 59-61 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.39-7.31 (m, 3H), 7.16 (td, *J* = 7.5, 0.9 Hz, 1H), 6.96 – 6.84 (m, 1H), 6.32 (t, *J* = 3.1 Hz, 1H), 6.27 (dd, *J* = 3.4, 0.8 Hz, 1H), 6.07 (dd, *J* = 17.0, 10.5 Hz, 1H), 5.43 (d, *J* = 17.0 Hz, 1H), 5.39 – 5.29 (m, 1H), 2.82 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.1, 136.6, 134.6, 132.2, 129.8, 125.7, 123.4, 118.8, 116.9, 115.6, 113.7, 100.3, 89.4; IR (ATR): *ν* 3319, 1089, 939, 749, 71*8* cm^{-1}; MS (EI): *m/z* 197.0 $[M]^+$, 182.0, 168.0, 141.0, 115.0; HRMS (ESI/Q-TOF) *m/z* calcd for $[C_{13}H_{11}NO + H^+]$: 198.0919, found 198.0912.

10H-Pyrrolizino[1,2-b]quinolin-10-one-N-oxide **19.** The mixture of pyrrolizinone **14l** (20 mg, 0.1 mmol) and MCPBA (70 %, 60 mg, 0.24 mmol) in dry acetone (5 mL) was refluxed for 1h. After completion of the reaction, as indicated by TLC, the mixture was cooled to room temperature, and solvent was removed under reduced pressure. The residue was purified by flash chromatography (SiO_2, diethyl ether) to afford the product **19** (15.8 mg, 72%) as a orange solid, mp 206-208 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 8.6 Hz, 1H), 8.04 (s, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.90 – 7.81 (m, 1H), 7.67 (d, *J* = 1.0 Hz, 1H), 7.28 (d, *J* = 2.4 Hz, 1H), 7.07 – 7.01 (m,1H), 6.49 (t, *J* = 3.2

Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.8, 144.4, 136.8, 133.0, 130.7, 129.5, 129.0, 128.6, 126.9, 124.6, 119.9, 119.6, 118.9, 114.0; IR (ATR): *v* 1745, 1392, 1293, 1245, 1218, 1064, 769, 733 cm^{-1}; MS (EI): *m/z* 220.0 $[M]^+$–O, 192.0, 164.0, 137.9; HRMS (ESI/Q-TOF) *m/z* calcd for $[C_{14}H_8N_2O_2 + H^+]$: 237.0664, found 237.0665.

References

1 A. Kamal, S.F. Adil, J. R.Tamboli, B. Siddardha, U. S. N. Murthy, *Lett.Drug Des.Discov.*, 2008, **5***,* 261.

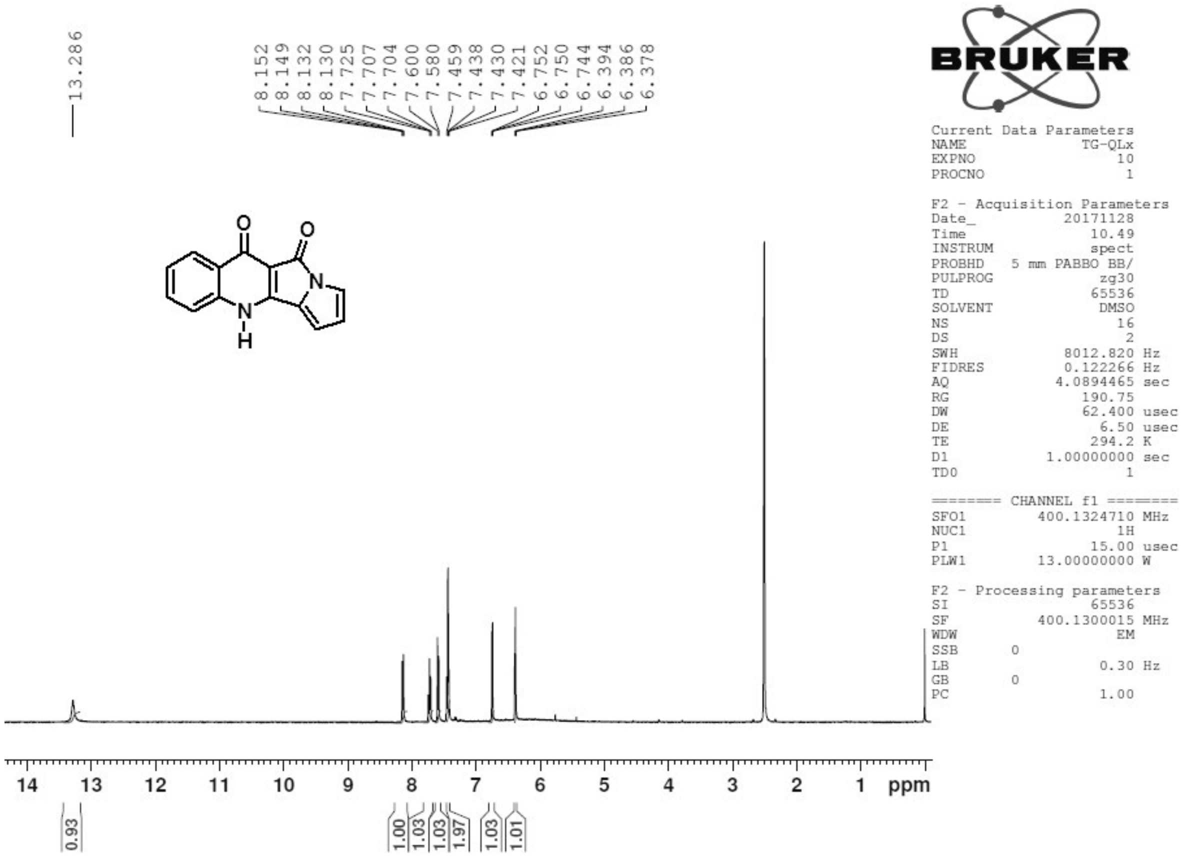
2 X. M. Chen, Y. B. Kang, *Org.Lett.*, 2016, **18***,* 2700.

3 E. Cuevas-Yanez, J. M. Muchowski, R. Cruz-Almanza *Tetrahedron,* 2004, **60**, 1505.

4 T. Itahara*, Fukusokan Kagaki Toronkai Koen Yoshishu 12th*, 1979, 86.

Copies of NMR spectra of synthesized pyrrolizinones and their derivatives

Quinolactacide **1**



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PROCNO                  1

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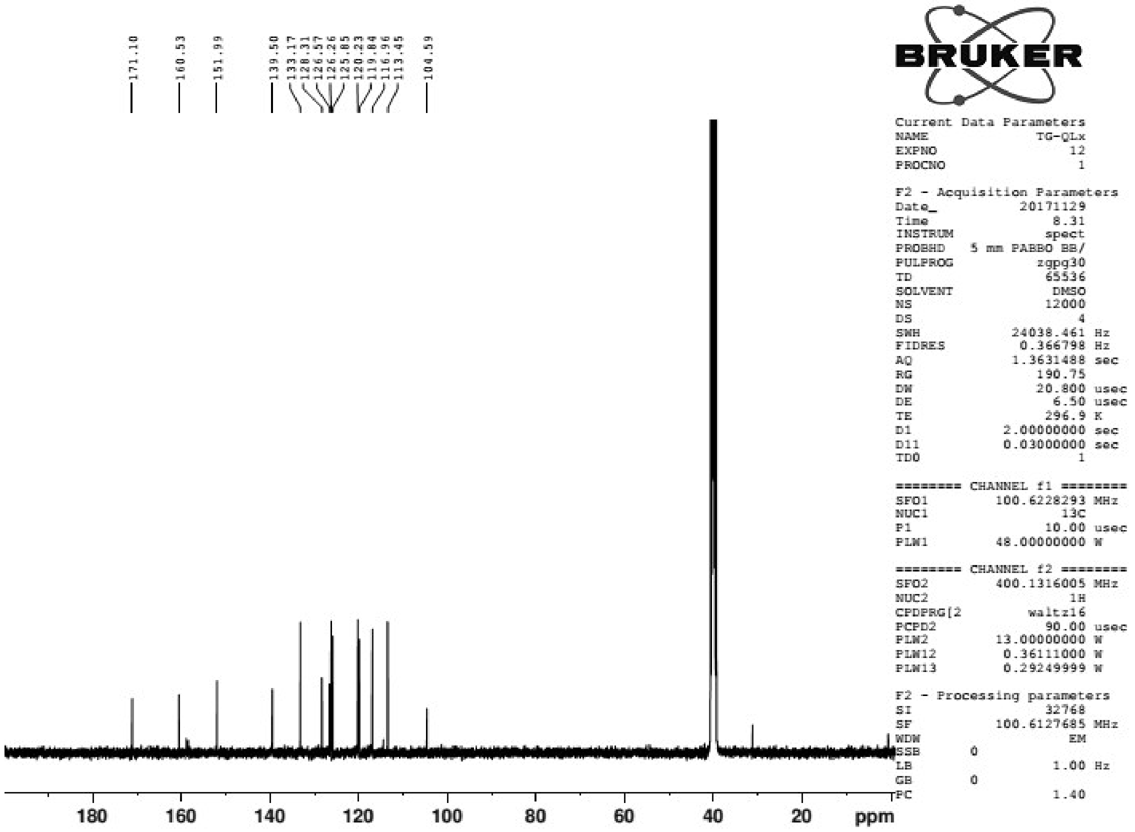

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F2 - Processing parameters
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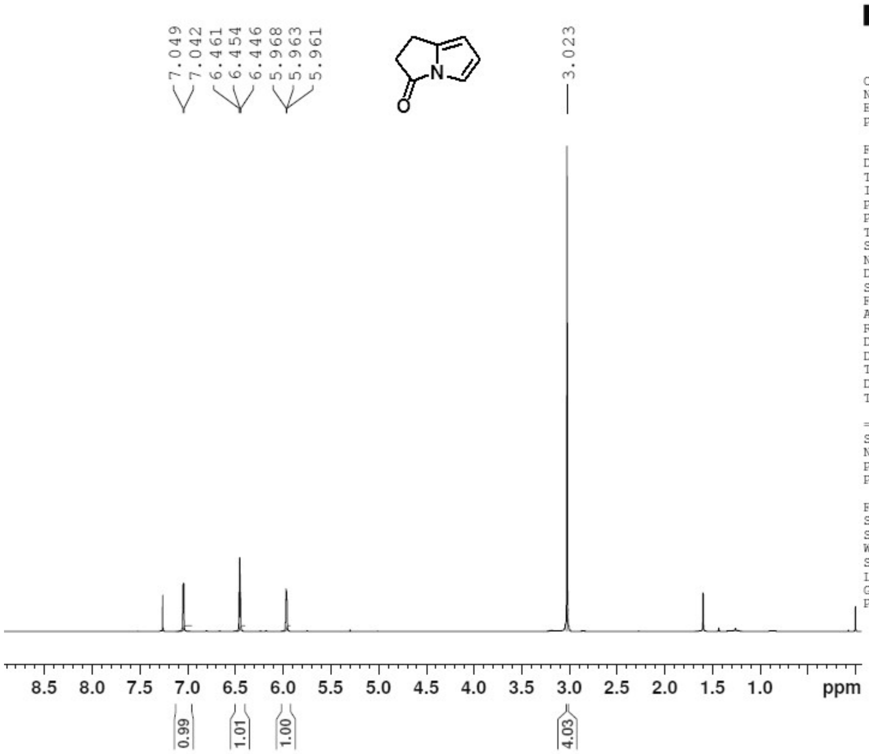
Compound **7**



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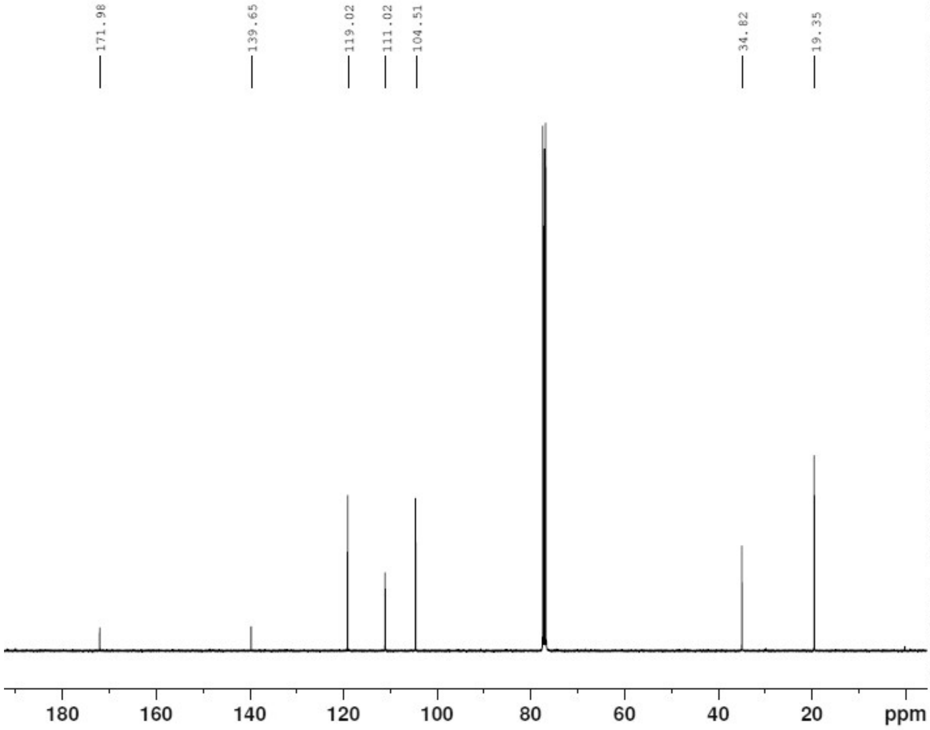

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RG                190.75
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F2 - Processing parameters
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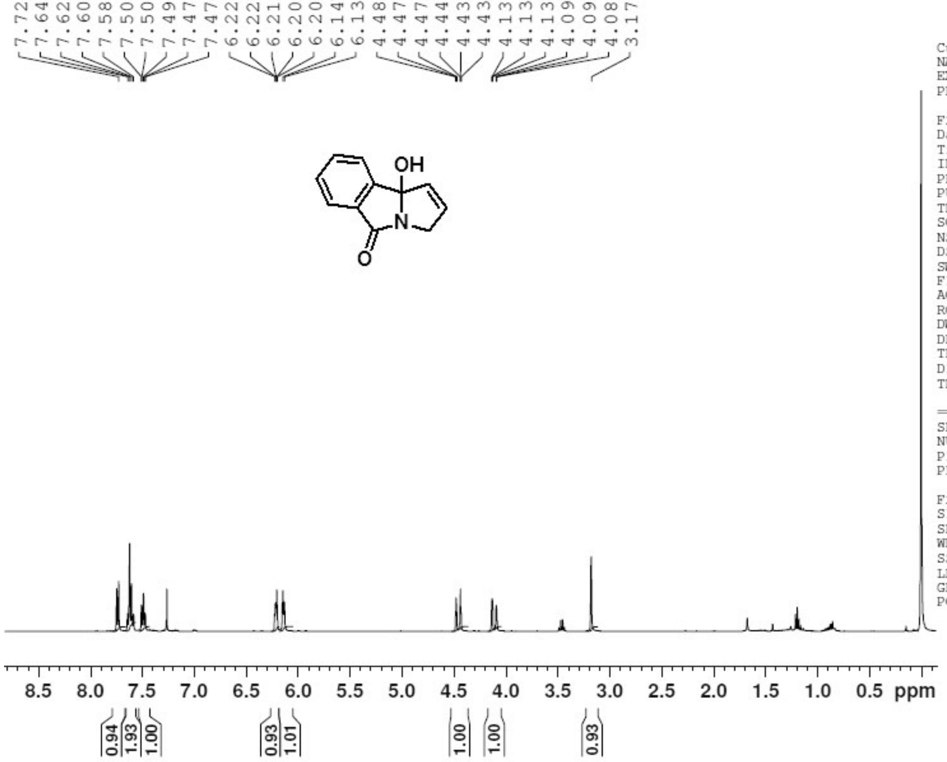
Compound **10**



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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
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RG                190.75
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TE                 297.8 K
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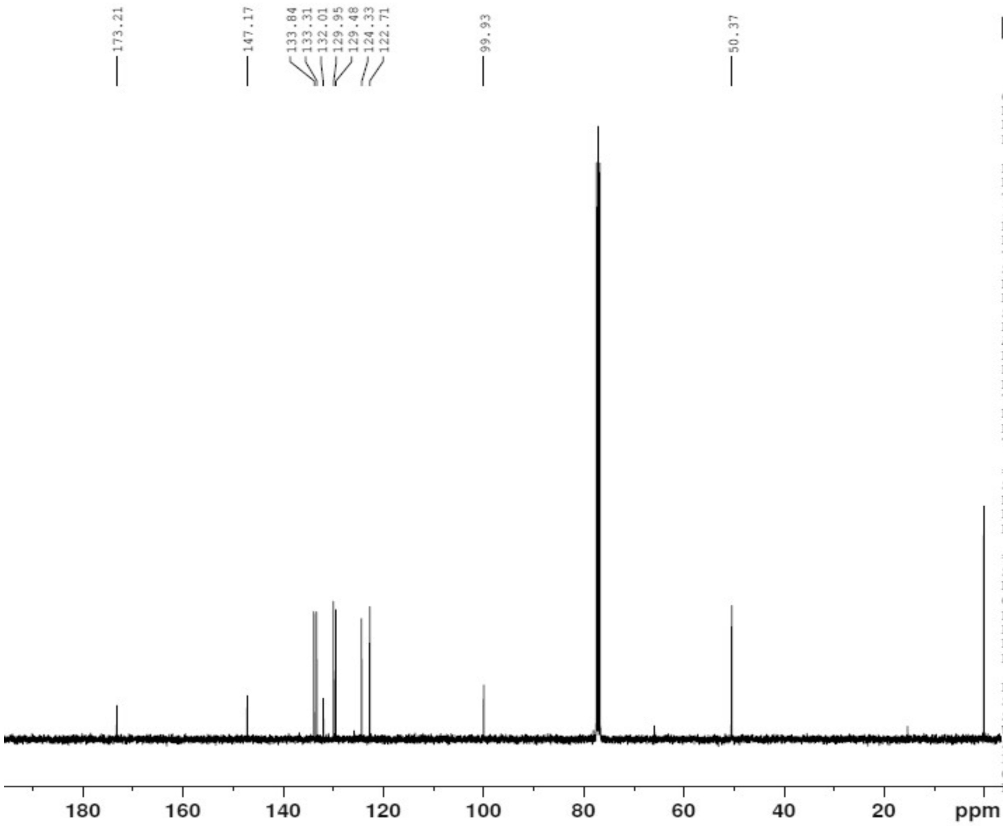

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PROCNO                 1

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PLW12         0.36111000 W
PLW13         0.29249999 W

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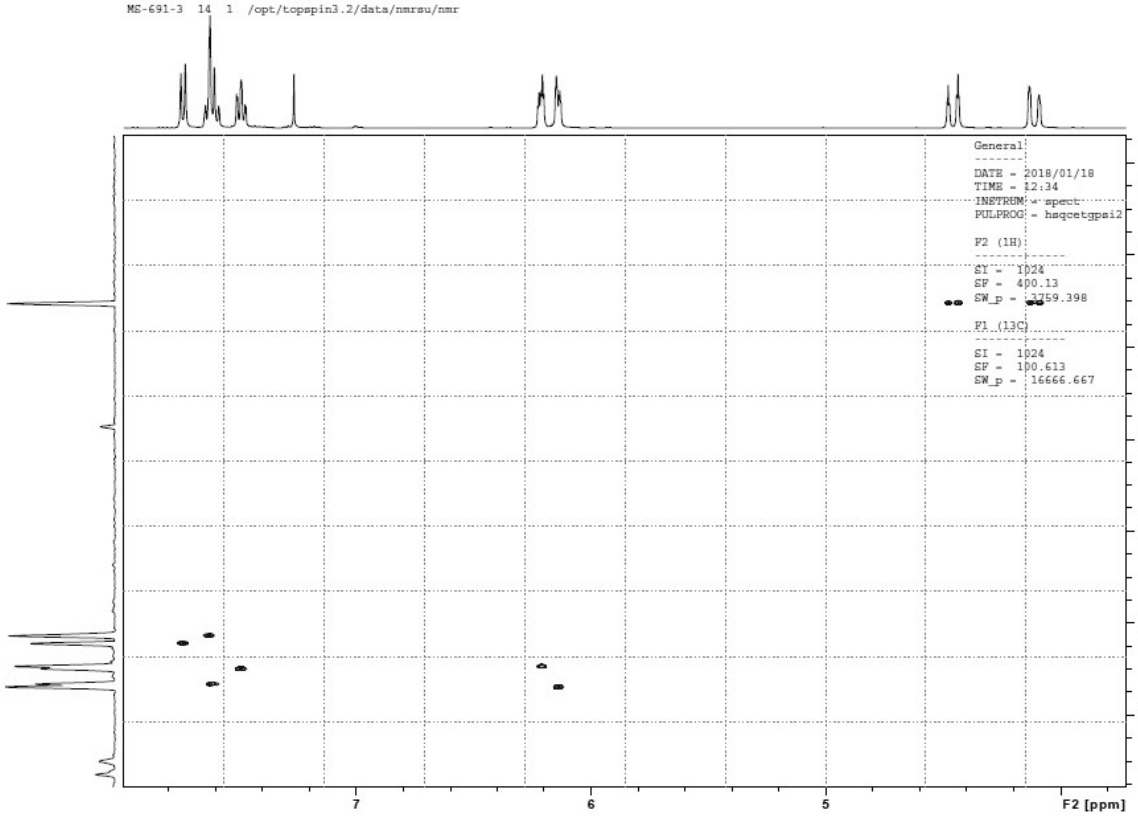
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F1 (13C)
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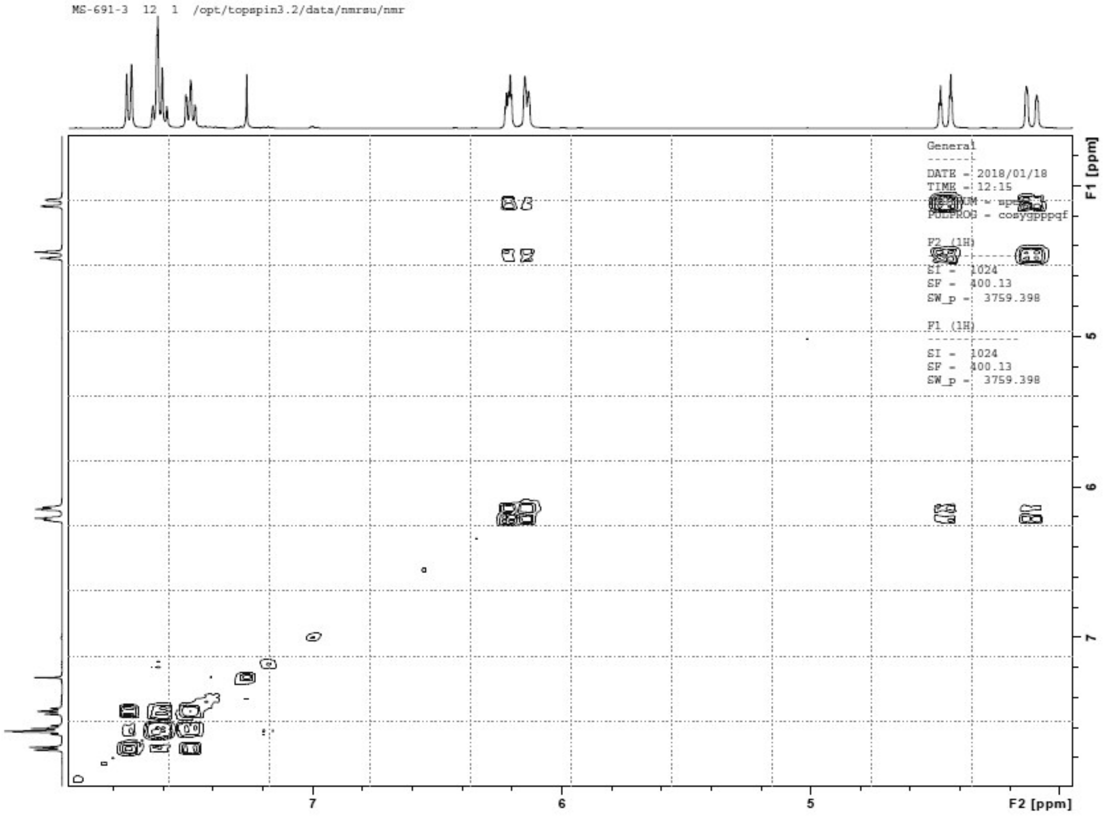
60
80
100
120
140
7
6
5
F2 [ppm]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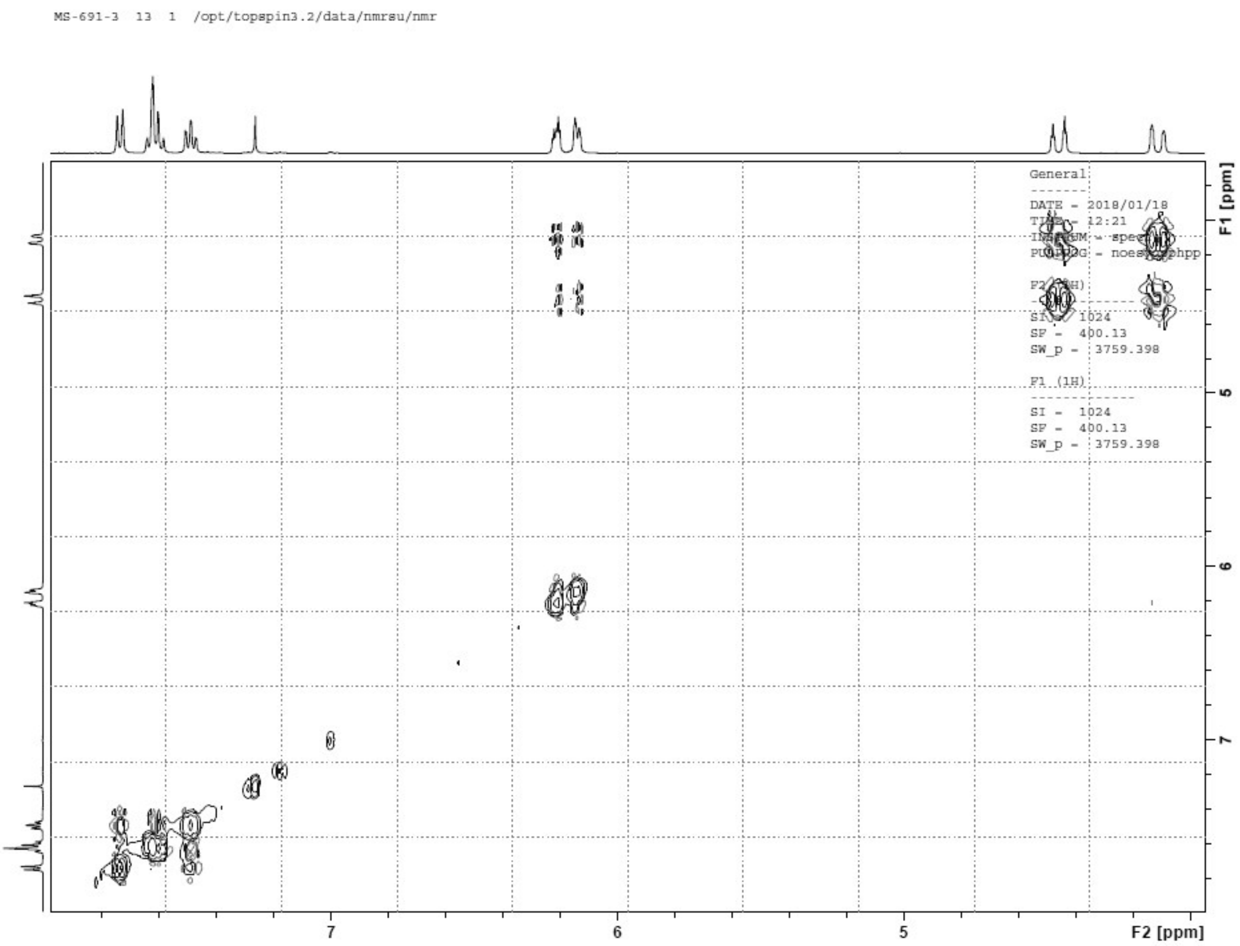
7
7
6
5
F2 [ppm]



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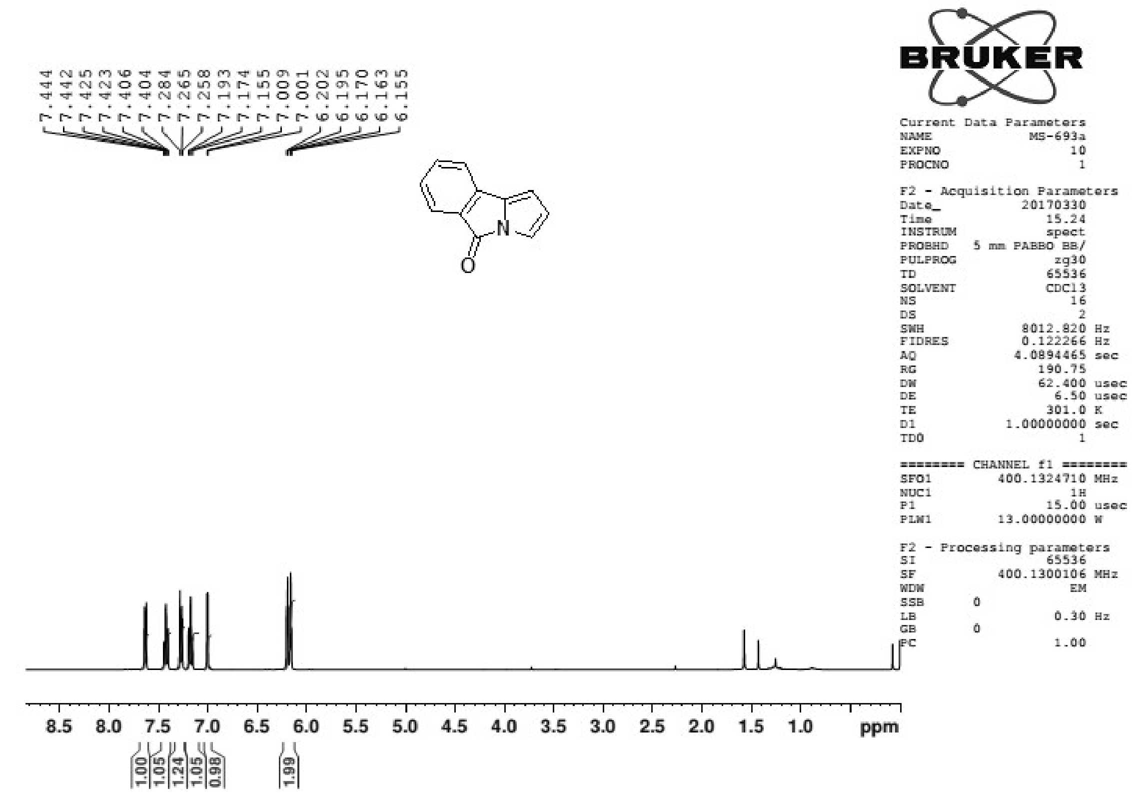
Compound **11**



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PROCNO                    1

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PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                4.0894465 sec
RG                   190.75
DW                   62.400 usec
DE                     6.50 usec
TE                    301.0 K
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TD0                       1

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NUC1                     1H
P1                    15.00 usec
PLW1         13.00000000 W

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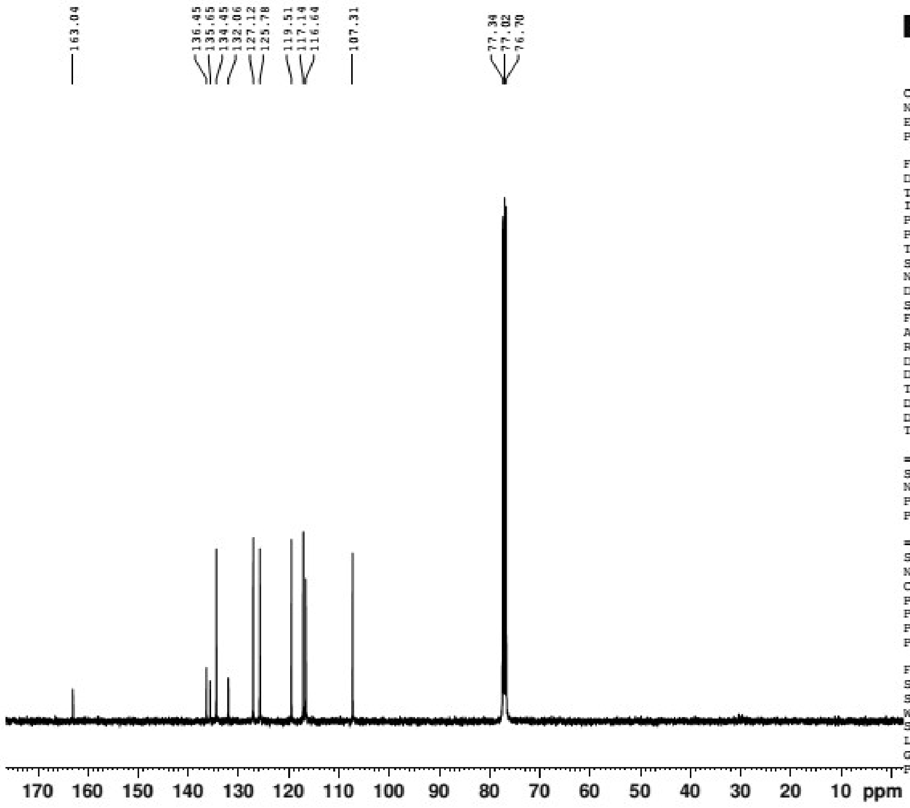

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EXPNO                 11
PROCNO                 1

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SOLVENT           CDCl3
NS                  959
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FIDRES         0.366798 Hz
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RG               190.75
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DE                 6.50 usec
TE                302.8 K
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D11          0.03000000 sec
TD0                   1

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PLW13        0.29249999 W

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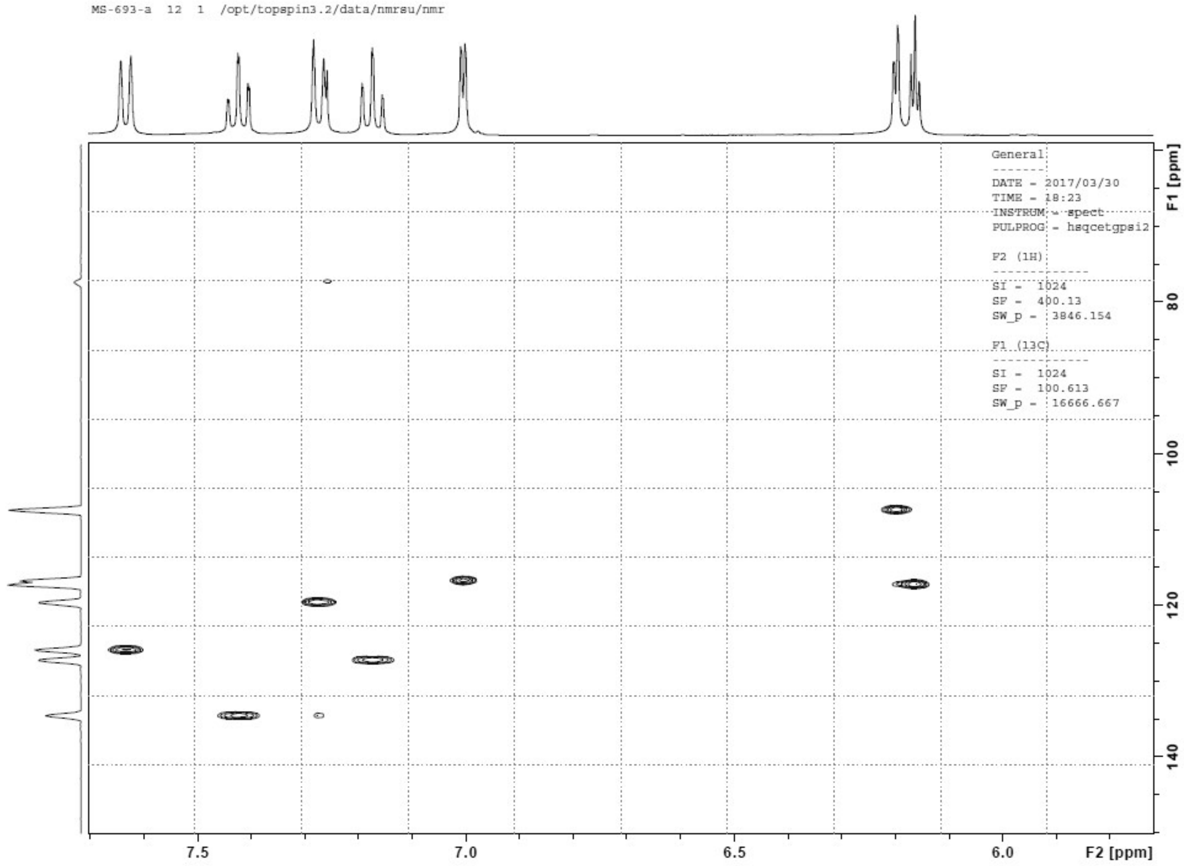
MS-693-a 12 1 /opt/topspin3.2/data/nmrsu/nmr



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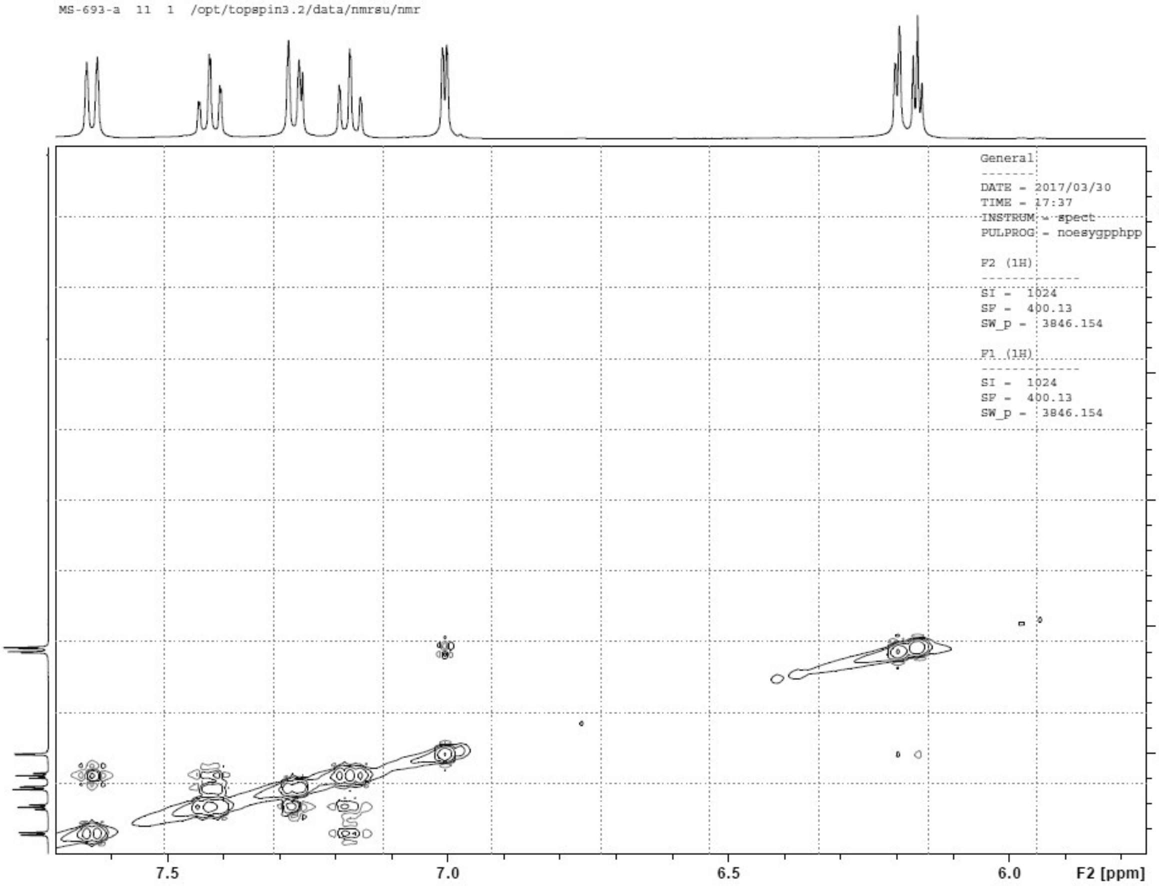

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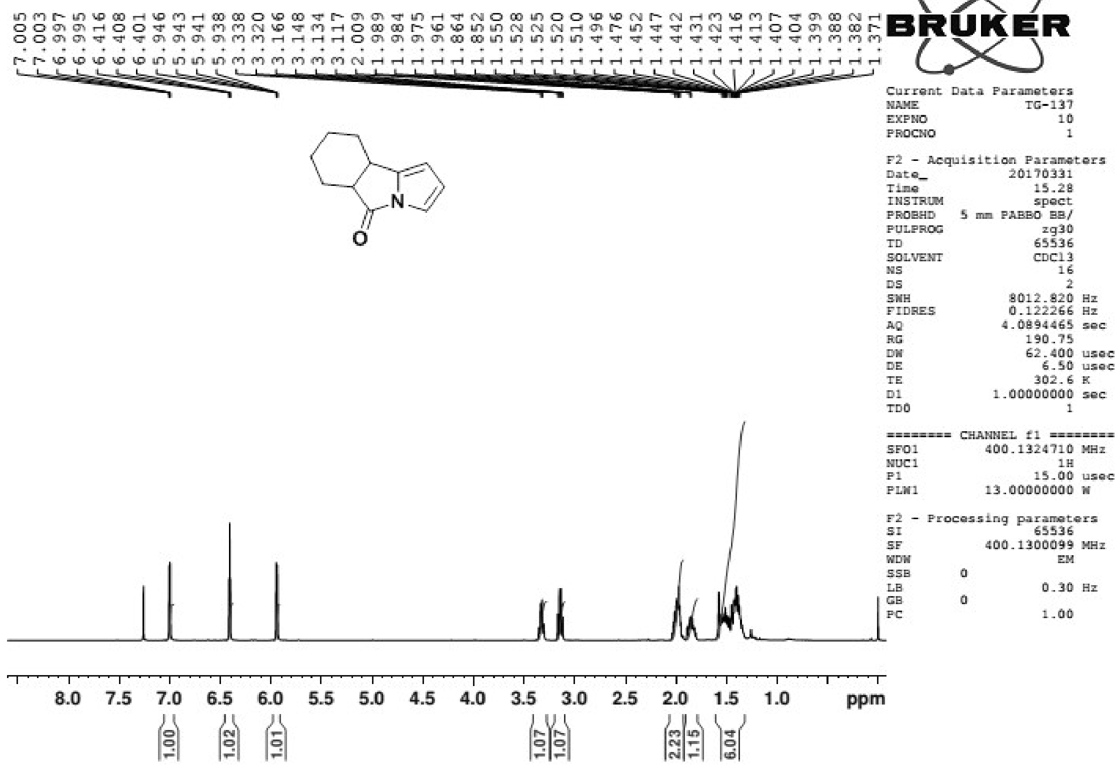
Compound **14a**



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SOLVENT            CDCl3
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FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 302.6 K
D1            1.00000000 sec
TD0                    1

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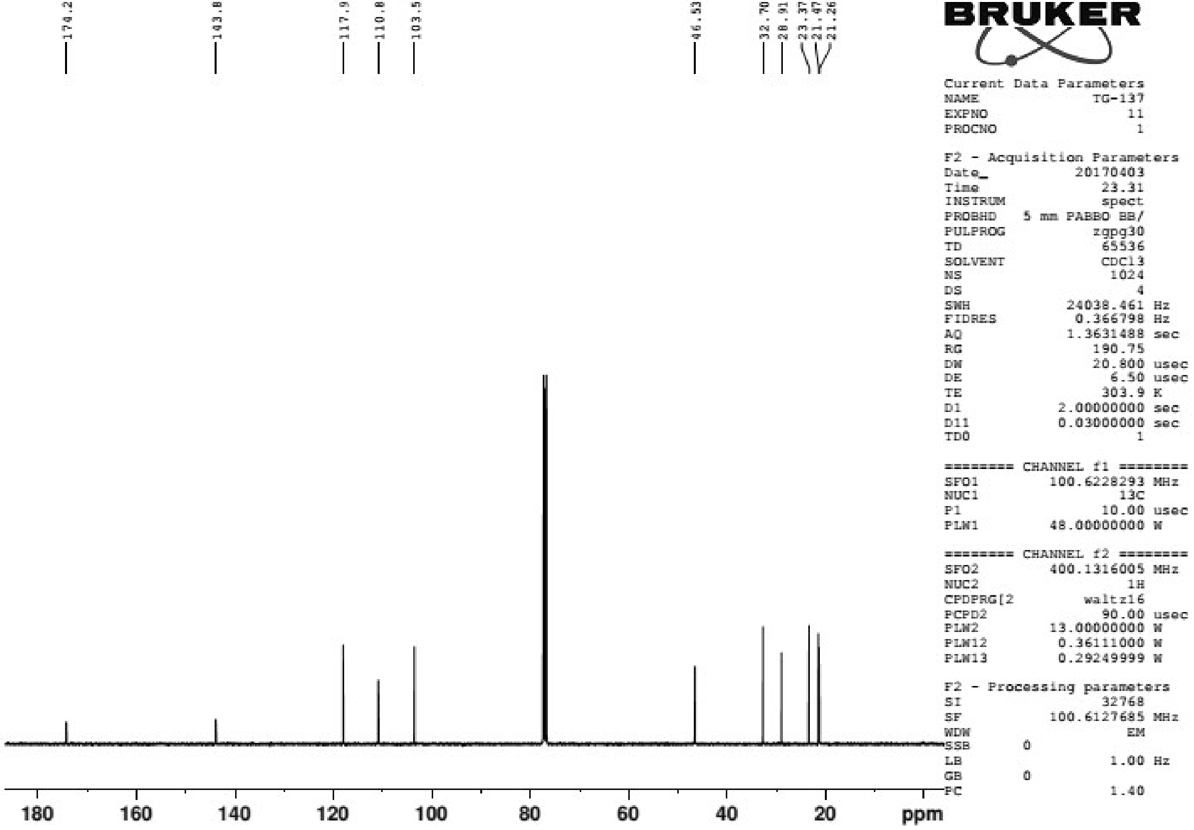

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FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 303.9 K
D1            2.00000000 sec
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PLW12         0.36111000 W
PLW13         0.29249999 W

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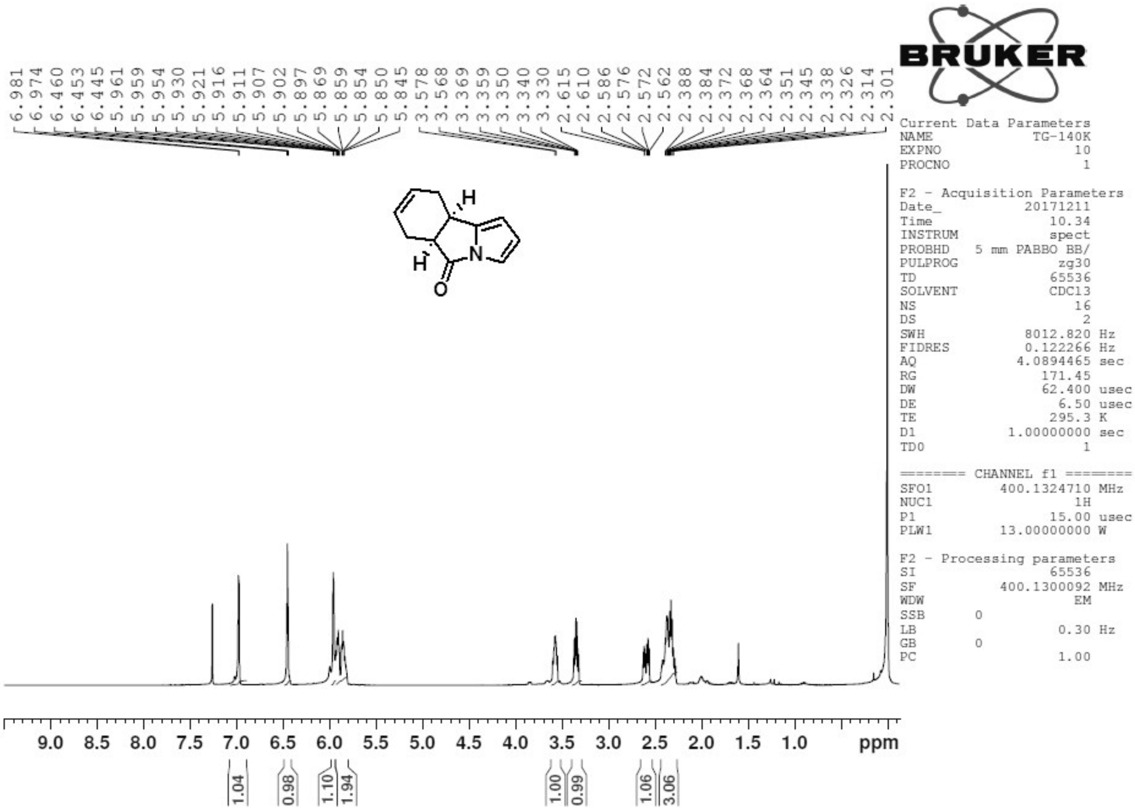
Compound **14b**



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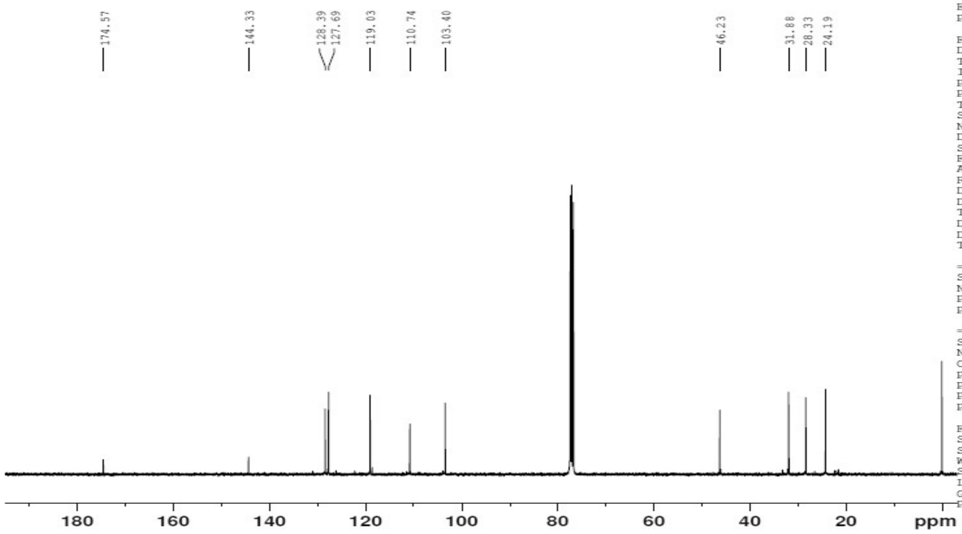
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                171.45
DW                62.400 usec
DE                  6.50 usec
TE                 295.3 K
D1            1.00000000 sec
TD0                    1

======= CHANNEL f1 =======
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300092 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME                TG-140K
EXPNO                    11
PROCNO                    1

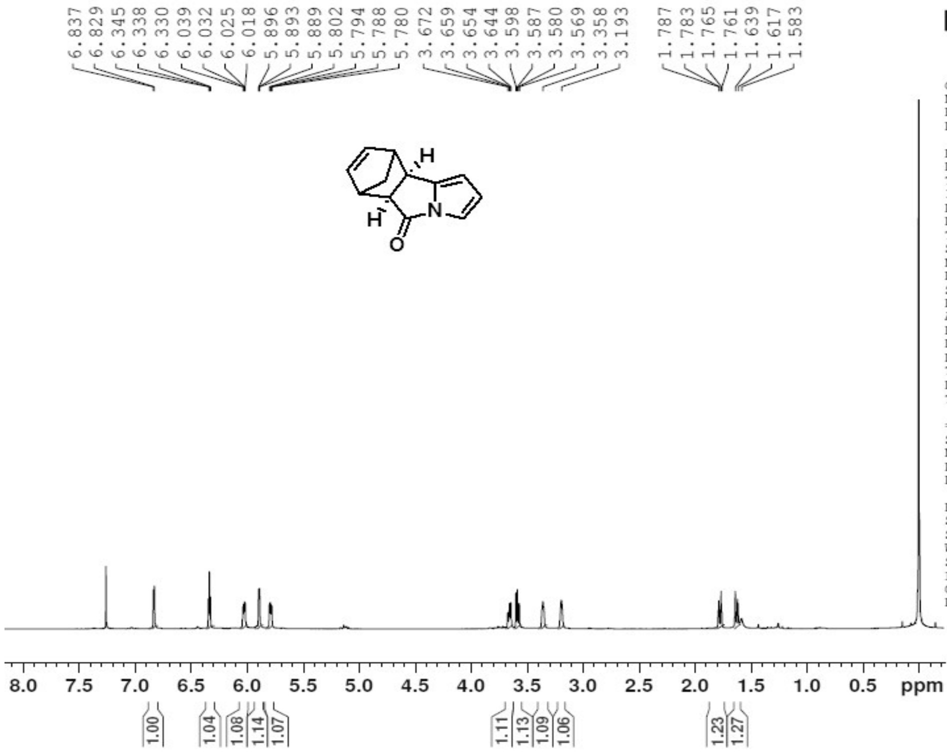
F2 - Acquisition Parameters
Date_              20171211
Time                  11.41
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                   190.75
DW                   20.800 usec
DE                     6.50 usec
TE                    298.4 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6228293 MHz
NUC1                    13C
P1                    10.00 usec
PLW1            48.00000000 W

======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                     1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2            13.00000000 W
PLW12            0.36111000 W
PLW13            0.29249999 W

F2 - Processing parameters
SI                    32768
SF              100.6127687 MHz
WDW                      EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

Compound **14c**

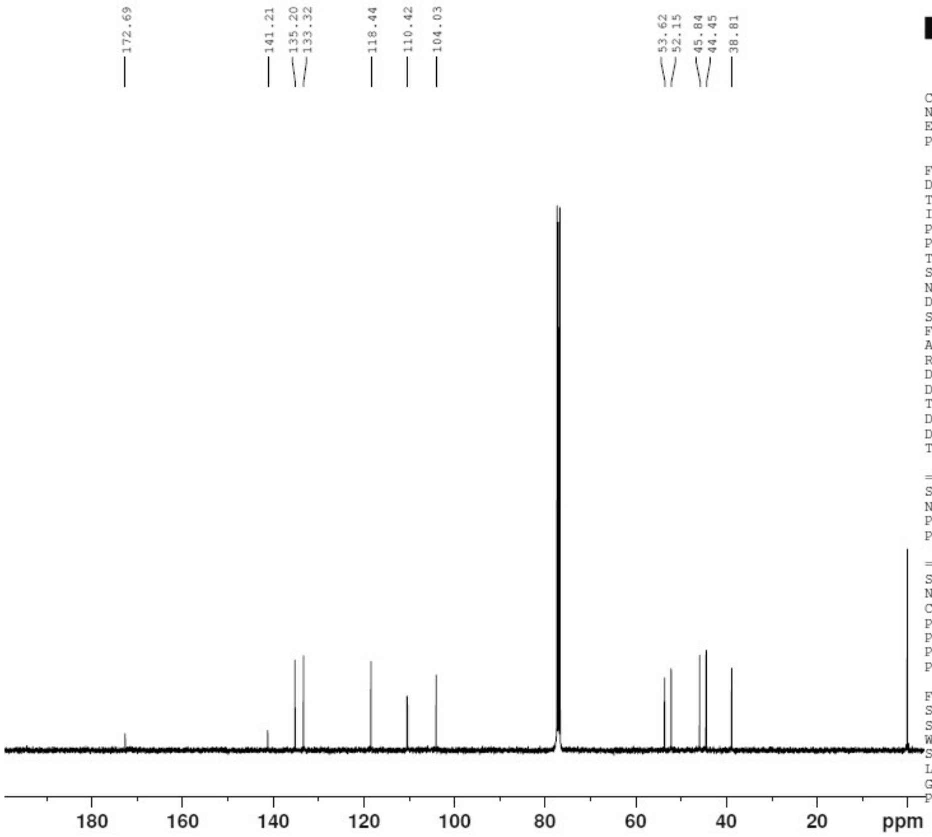


```
Current Data Parameters
NAME              TG-153k
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171211
Time                13.43
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     12
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 190.75
DW                 62.400 usec
DE                   6.50 usec
TE                  296.8 K
D1             1.00000000 sec
TD0                     1

======= CHANNEL f1 =======
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300091 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME              TG-153k
EXPNO                  11
PROCNO                  1

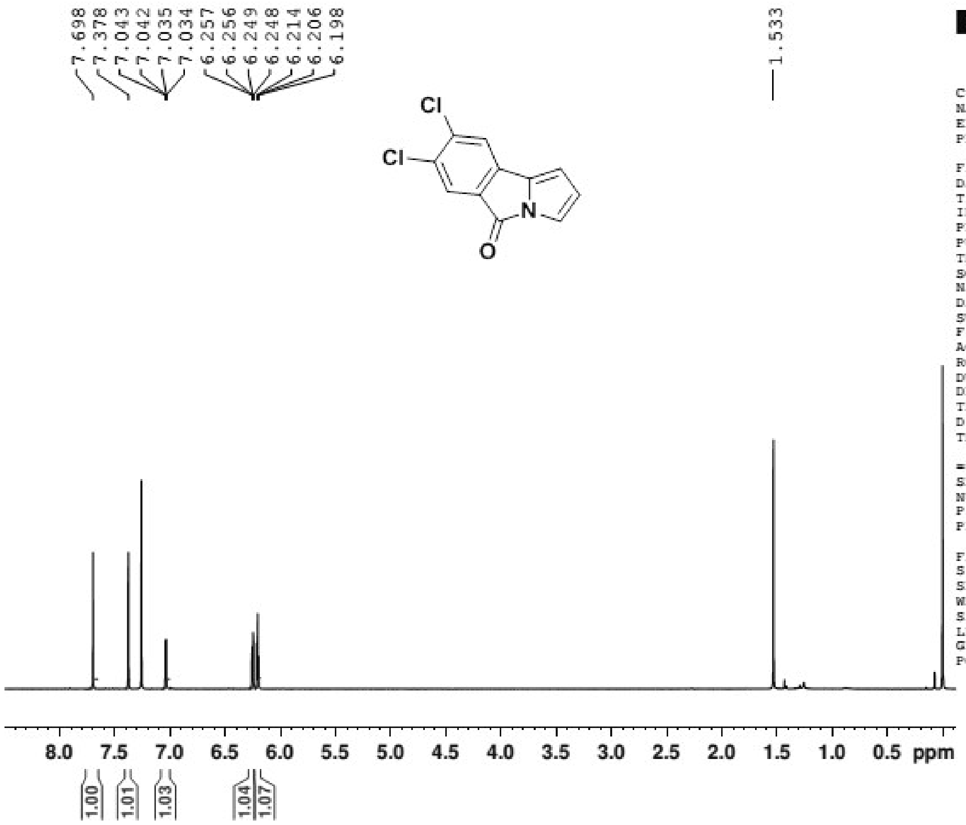
F2 - Acquisition Parameters
Date_            20171211
Time                14.42
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  299.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======= CHANNEL f1 =======
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

======= CHANNEL f2 =======
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

F2 - Processing parameters
SI                  32768
SF            100.6127681 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```

Compound **14d**

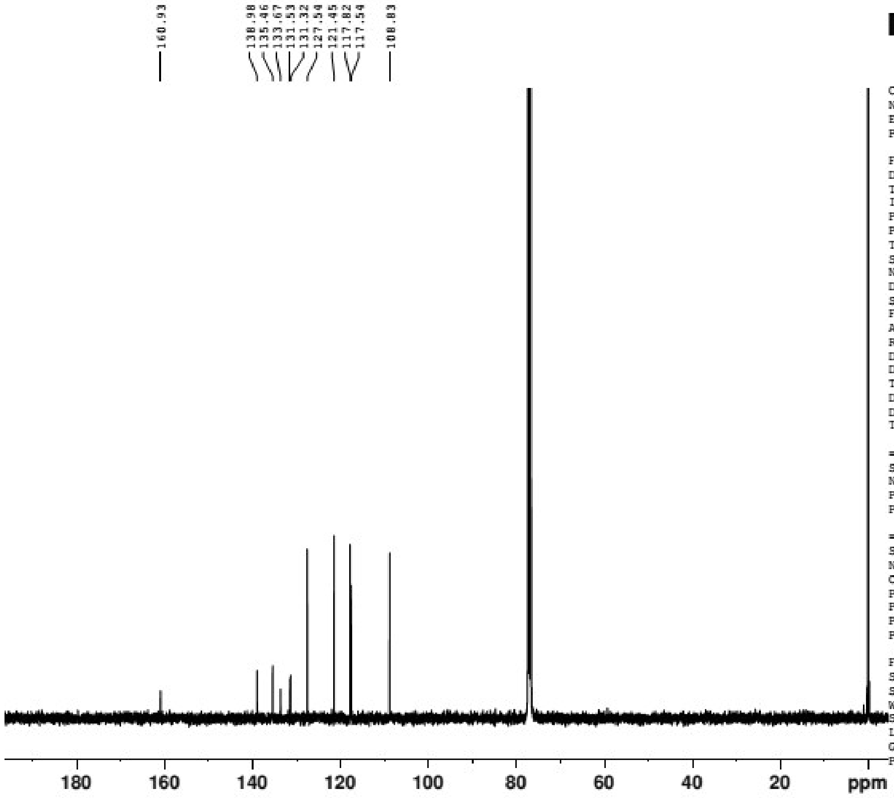


```
Current Data Parameters
NAME               MS-711
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170426
Time                15.07
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 190.75
DW                 62.400 usec
DE                   6.50 usec
TE                  301.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300107 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME             MS-711-2
EXPNO                  10
PROCNO                  1

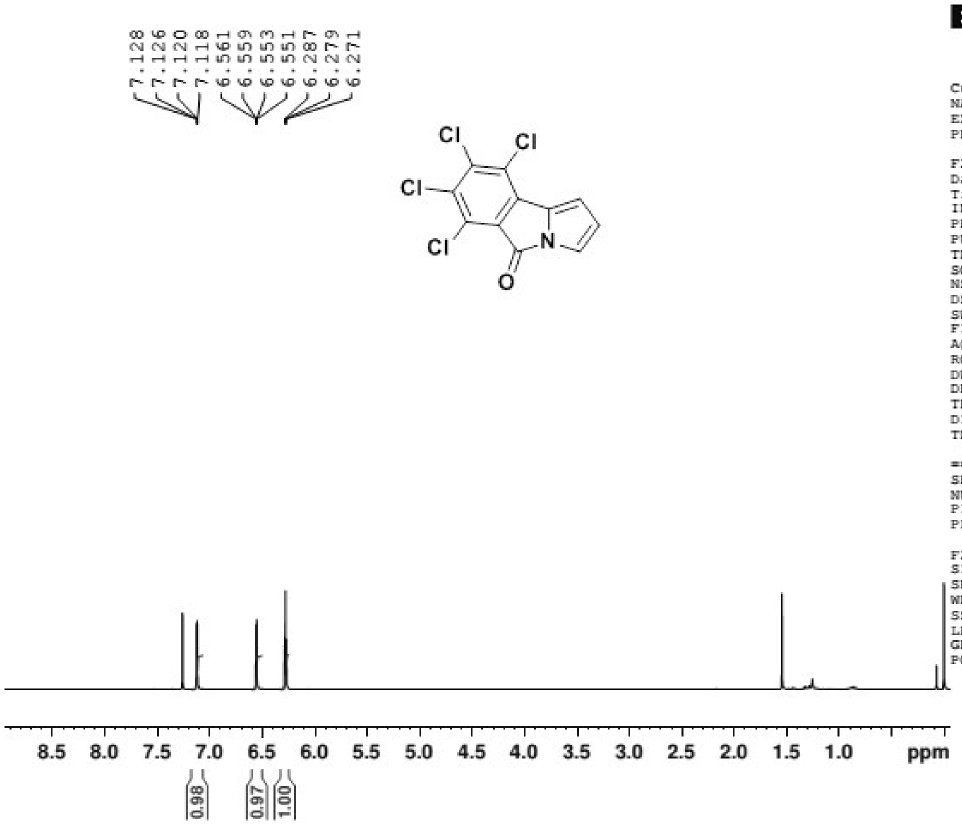
F2 - Acquisition Parameters
Date_            20171122
Time                 4.58
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   4000
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  298.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

F2 - Processing parameters
SI                  32768
SF            100.6127681 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```

Compound **14e**

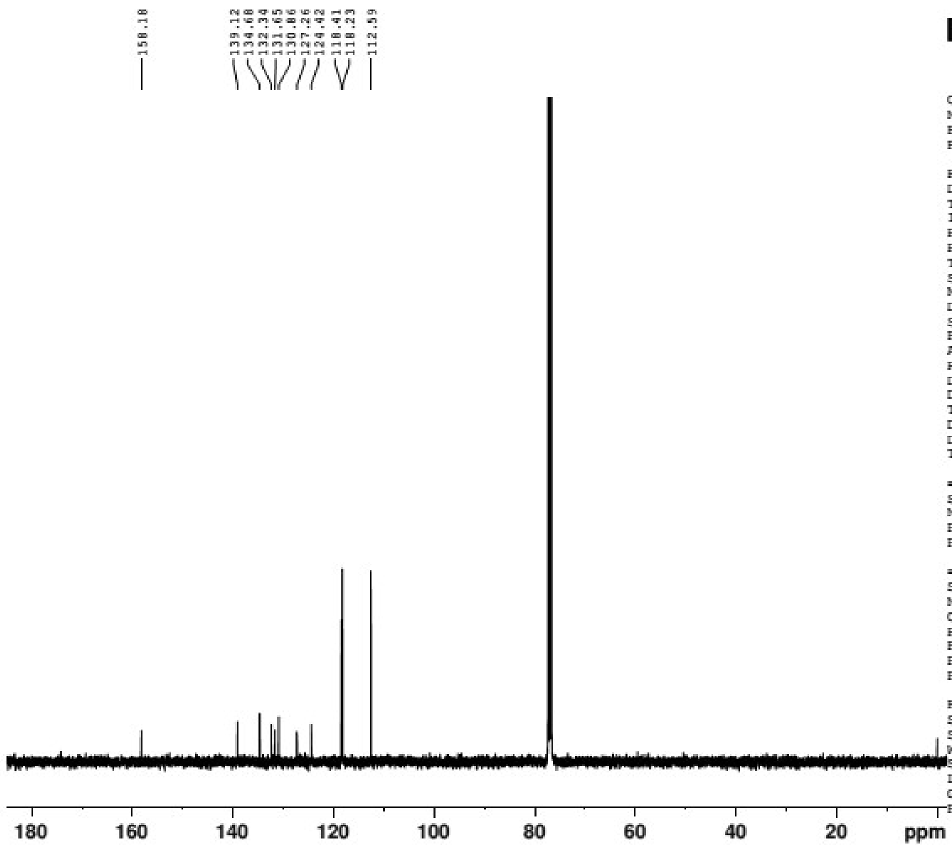


```
Current Data Parameters
NAME              MS-704G
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170413
Time                13.21
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 190.75
DW                 62.400 usec
DE                   6.50 usec
TE                  298.8 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300098 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



```
Current Data Parameters
NAME              MS-704G
EXPNO                  11
PROCNO                  1

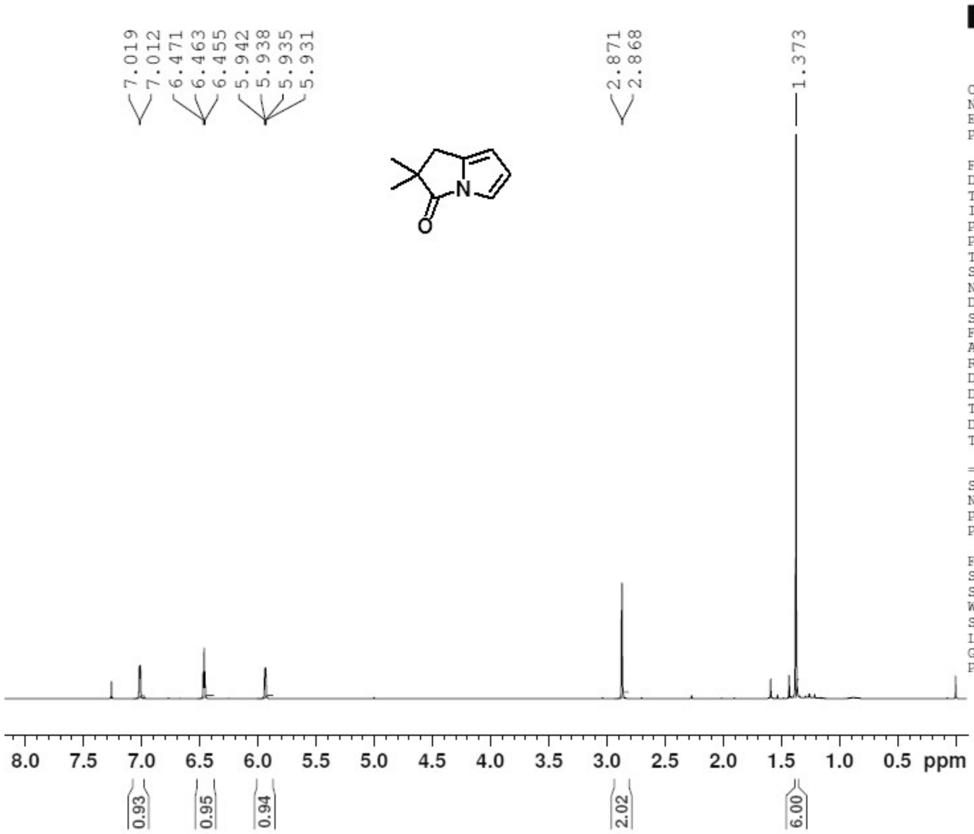
F2 - Acquisition Parameters
Date_            20170413
Time                21.31
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  302.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

Compound **14f**

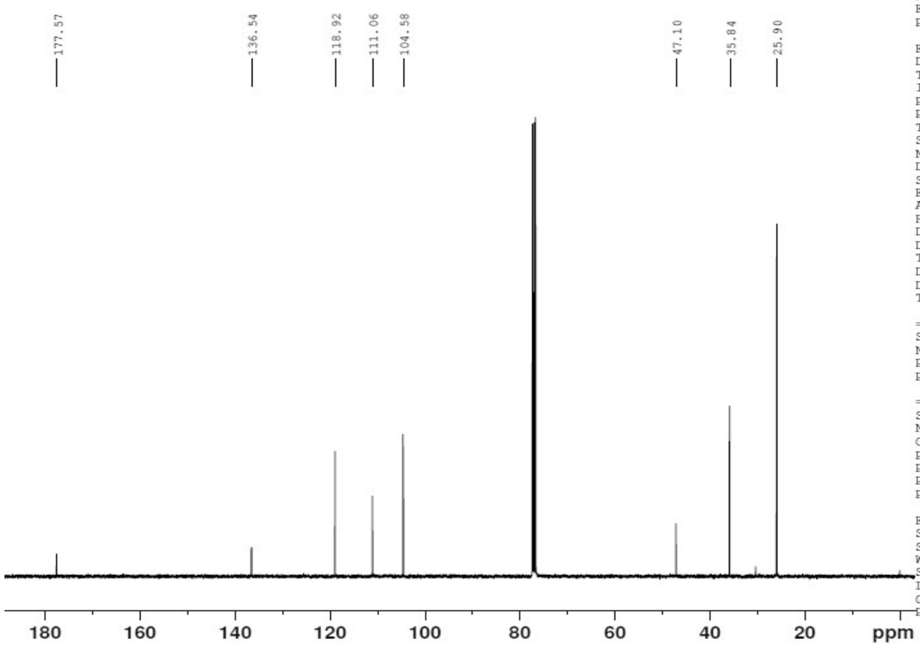


```
Current Data Parameters
NAME              TG-156
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170426
Time               15.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 301.0 K
D1            1.00000000 sec
TD0                    1

======= CHANNEL f1 =======
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300102 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



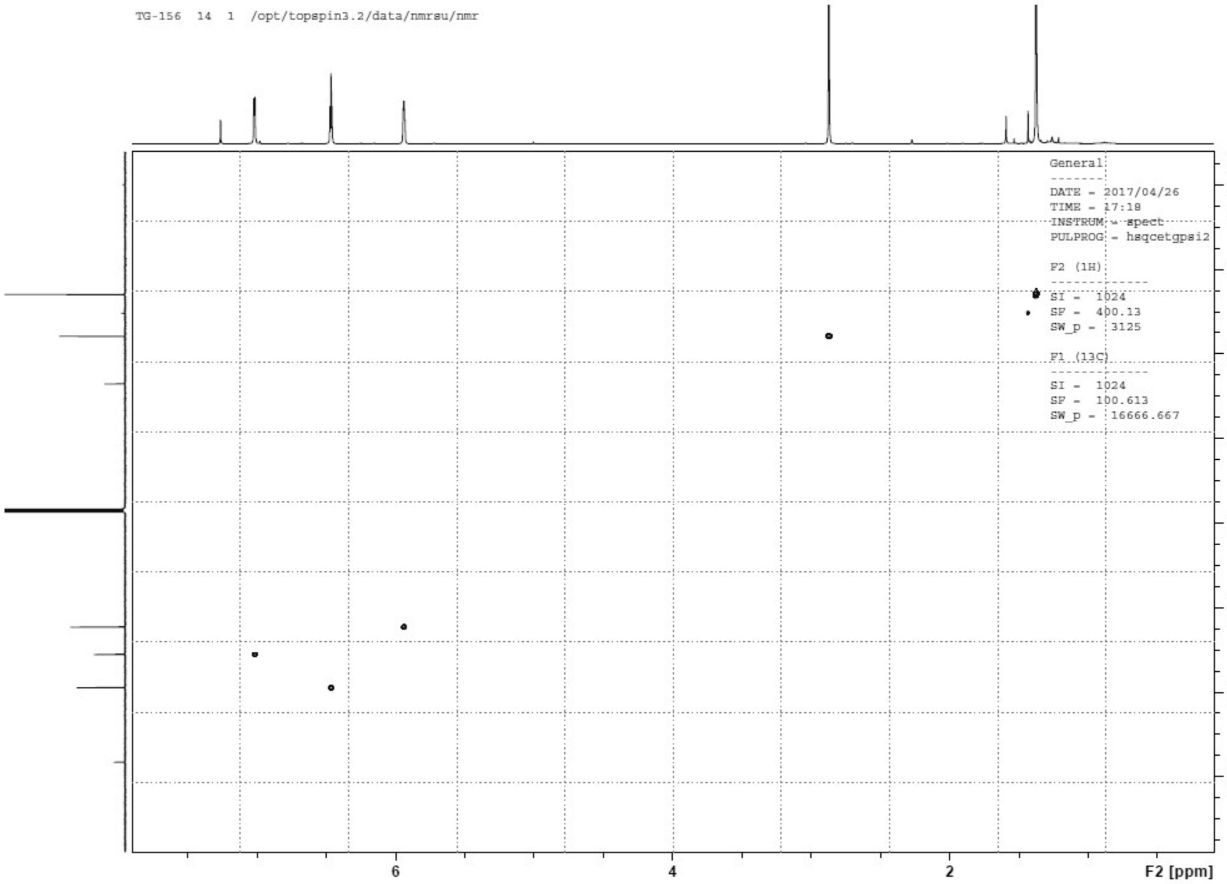
```
Current Data Parameters
NAME              TG-156
EXPNO                 11
PROCNO                 1

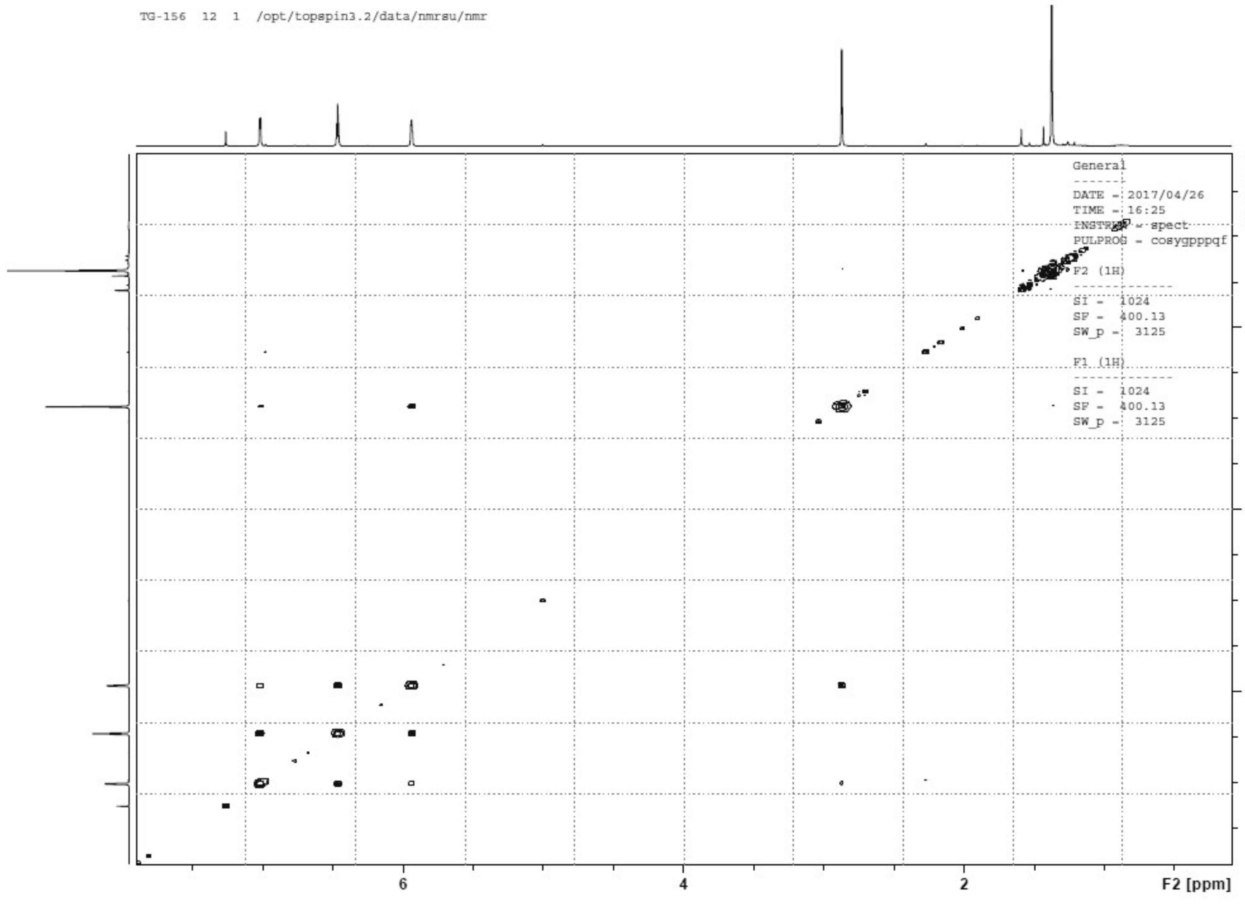
F2 - Acquisition Parameters
Date_           20170426
Time               16.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 303.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

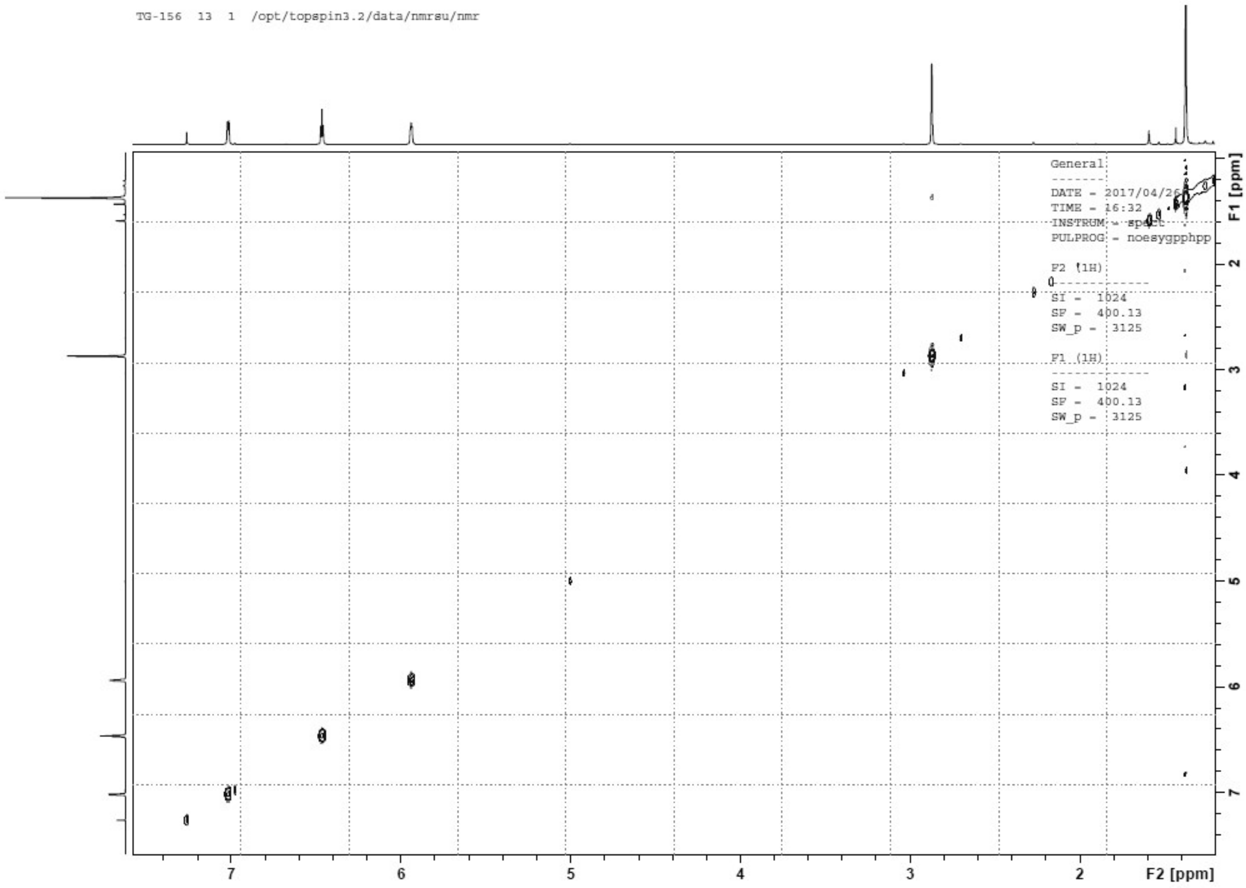
======= CHANNEL f1 =======
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

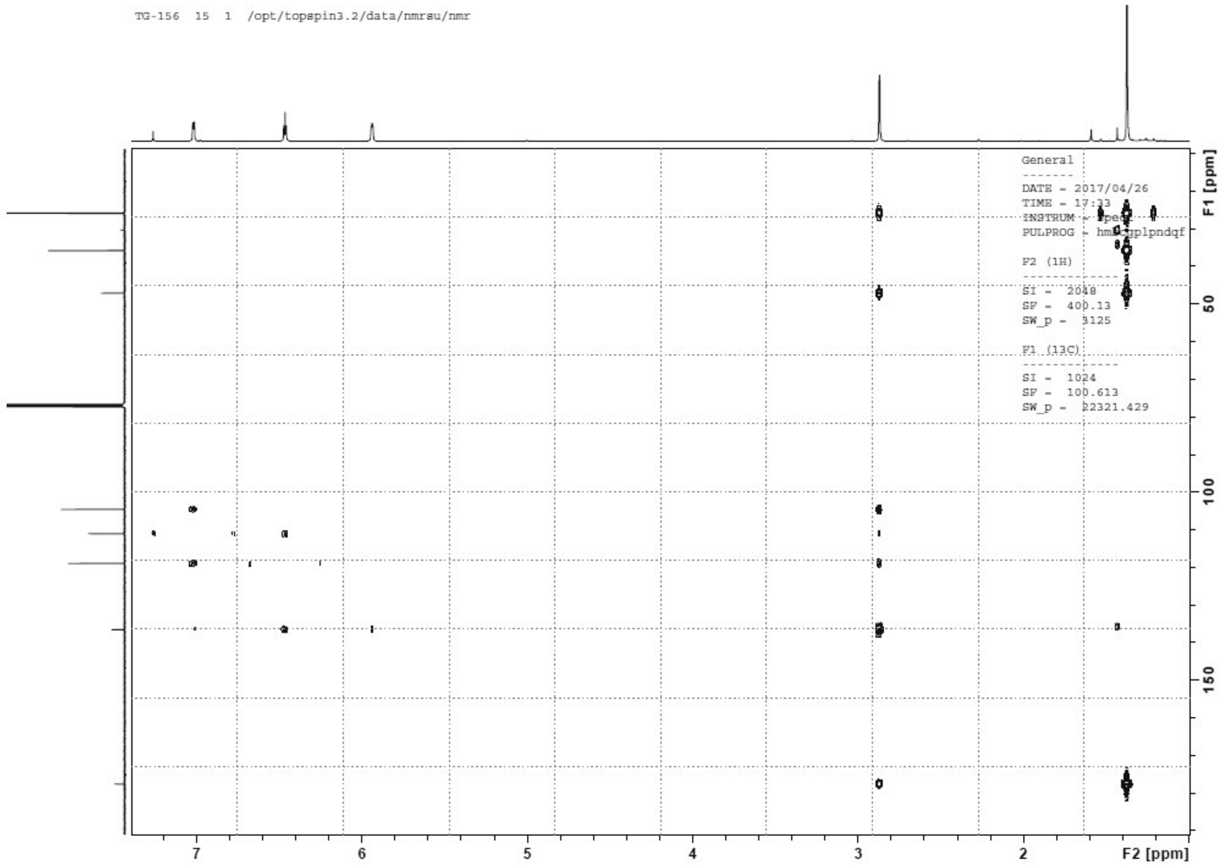
======= CHANNEL f2 =======
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

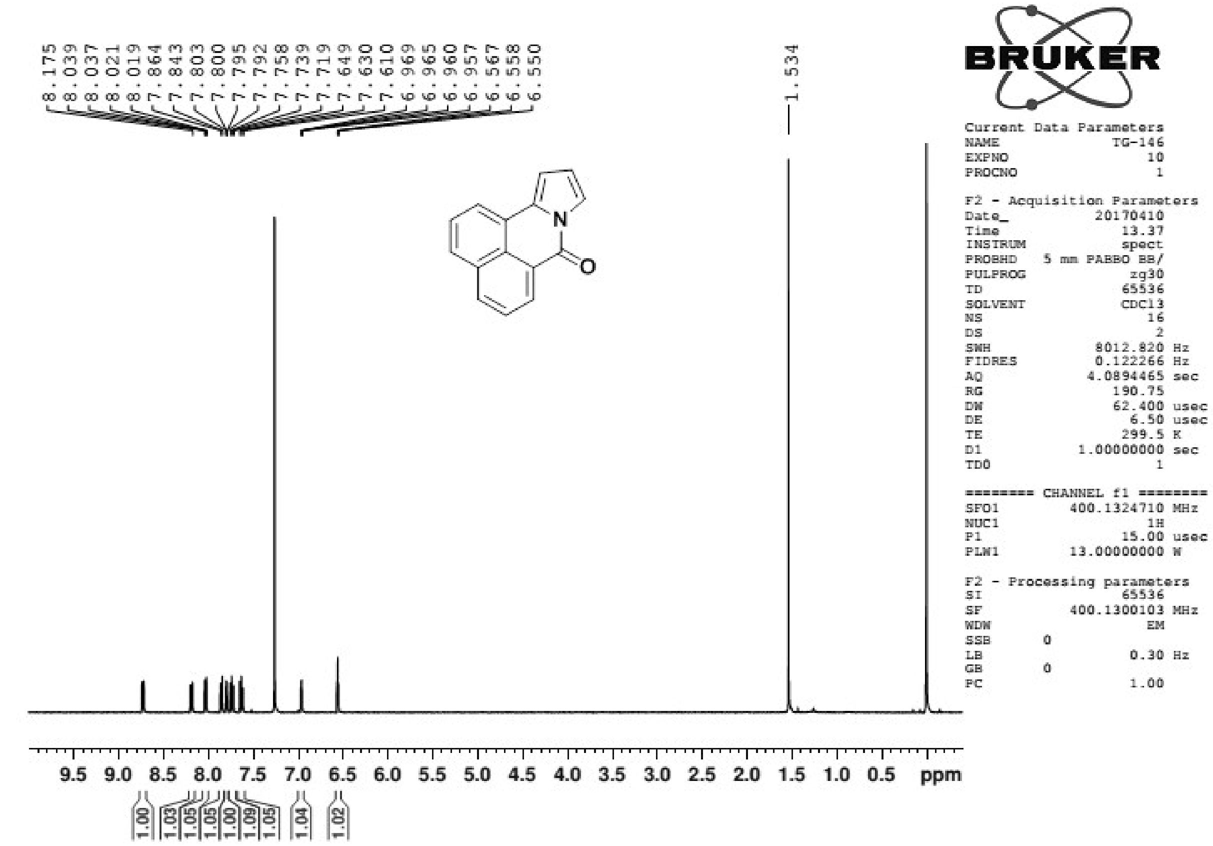

TG-156 14 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/04/26
TIME = 17:18
INSTRUM = spect
PULPROG = hsqcetgpsi2
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3125
F1 (13C)
SI = 1024
SF = 100.613
SW_p = 16666.667
F1 [ppm]
20
40
60
80
100
120
140
6
4
2
F2 [ppm]


TG-156 12 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/04/26
TIME = 16:25
INSTRUM = spect
PULPROG = cosygpppqf
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3125
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 3125
F1 [ppm]
2
4
6
6
4
2
F2 [ppm]


TG-156 13 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/04/26
TIME = 16:32
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3125
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 3125
F1 [ppm]
F2 [ppm]
7
6
5
4
3
2


TG-156 15 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/04/26
TIME = 17:33
INSTRUM = spect
PULPROG = hmbcgplpndqf
F2 (1H)
SI = 2048
SF = 400.13
SW_p = 3125
F1 (13C)
SI = 1024
SF = 100.613
SW_p = 22321.429
F1 [ppm]
F2 [ppm]
50
100
150
7
6
5
4
3
2

Compound **14g**

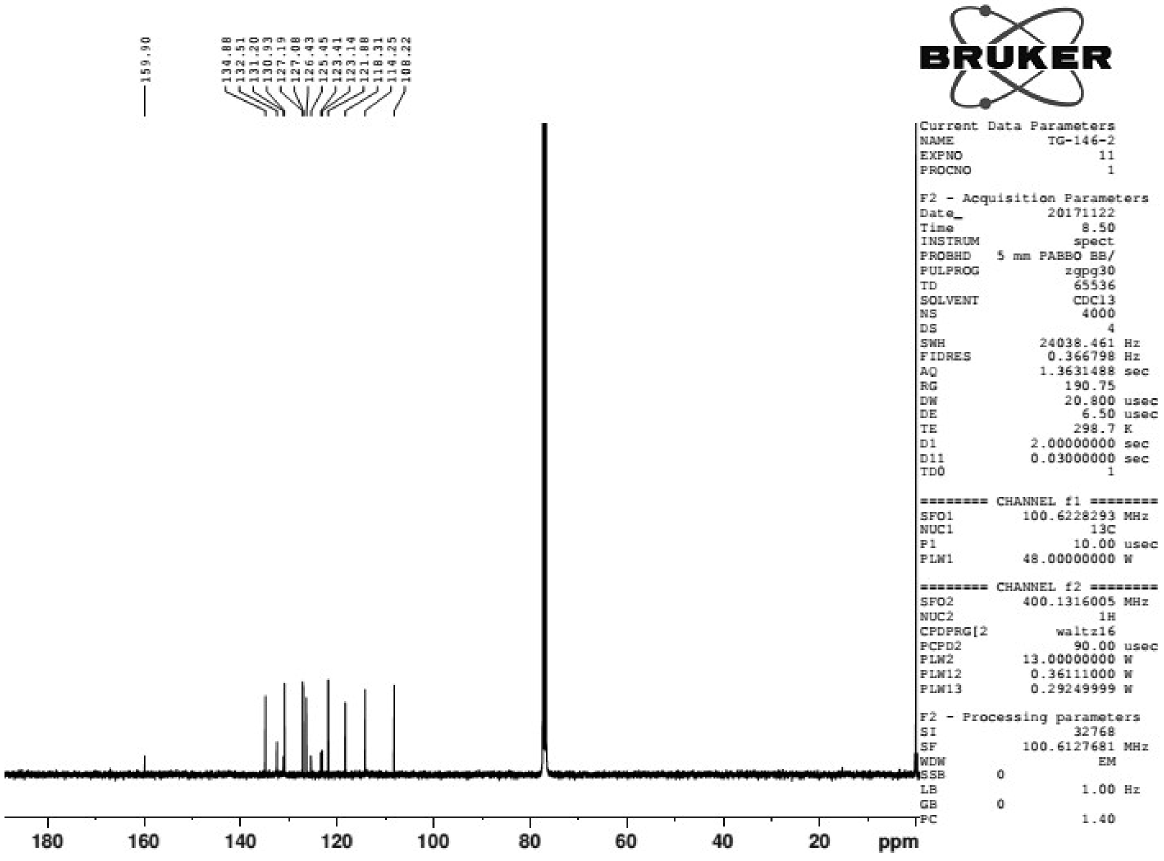


```
Current Data Parameters
NAME              TG-146
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170410
Time               13.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 299.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300103 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



```
Current Data Parameters
NAME            TG-146-2
EXPNO                 11
PROCNO                 1

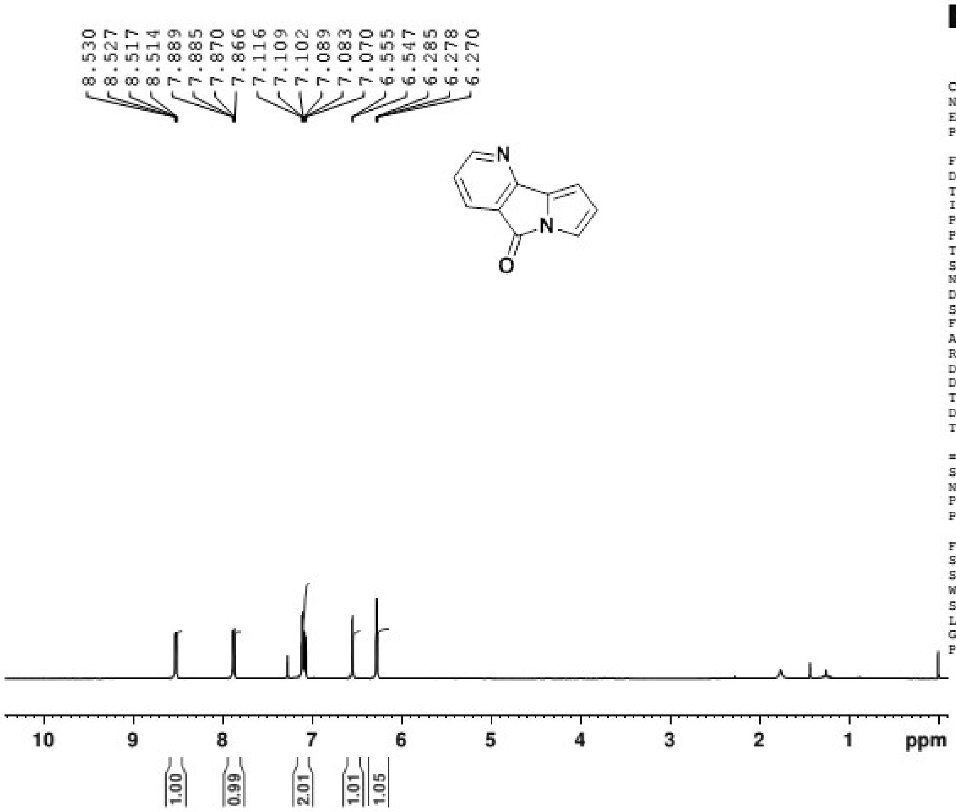
F2 - Acquisition Parameters
Date_           20171122
Time                8.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  4000
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 298.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

F2 - Processing parameters
SI                 32768
SF           100.6127681 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

Compound **14h**

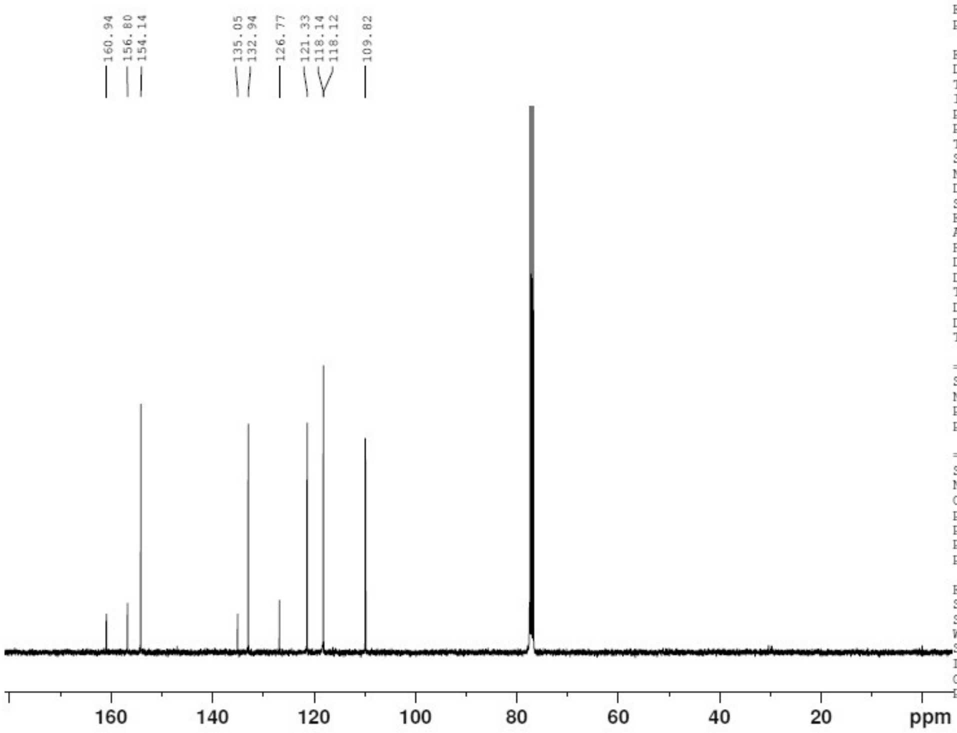


```
Current Data Parameters
NAME              MS-719
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170508
Time                5.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 296.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300051 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME              MS-719
EXPNO                 11
PROCNO                 1

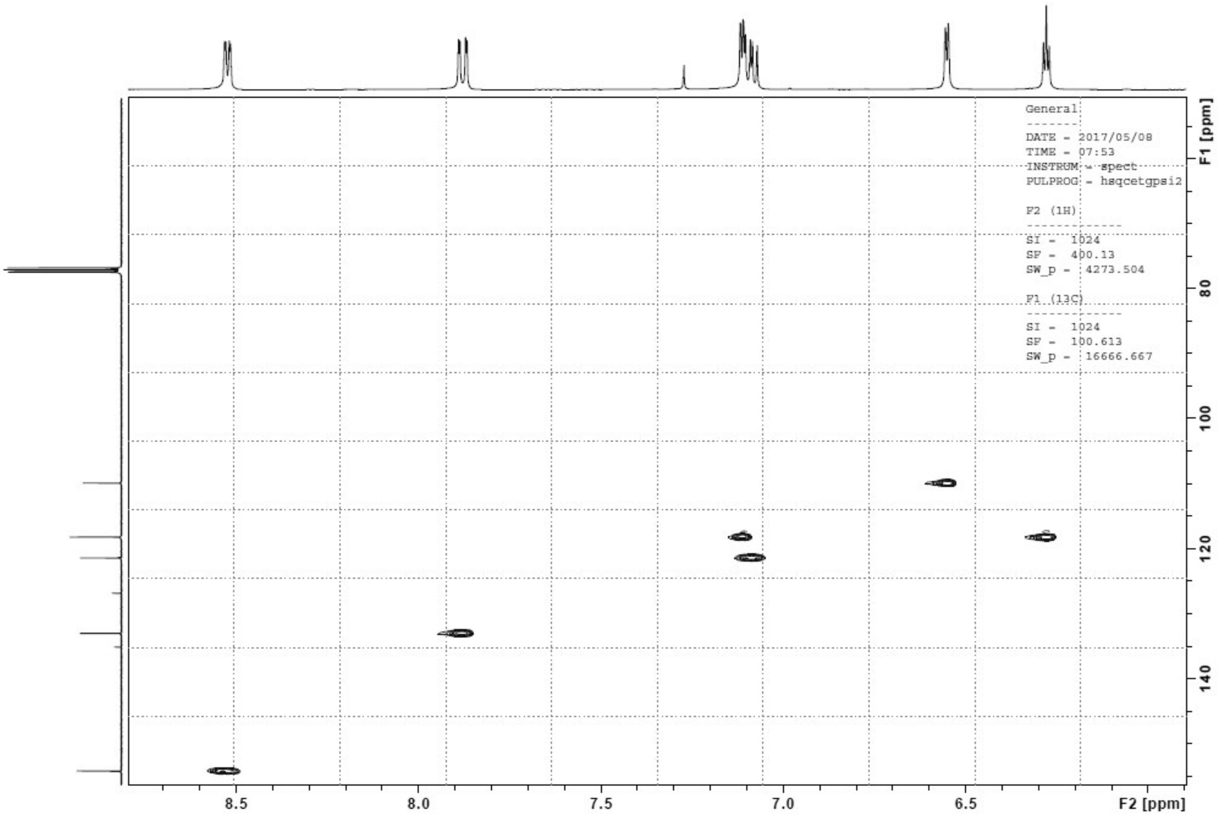
F2 - Acquisition Parameters
Date_           20170508
Time                6.49
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 298.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

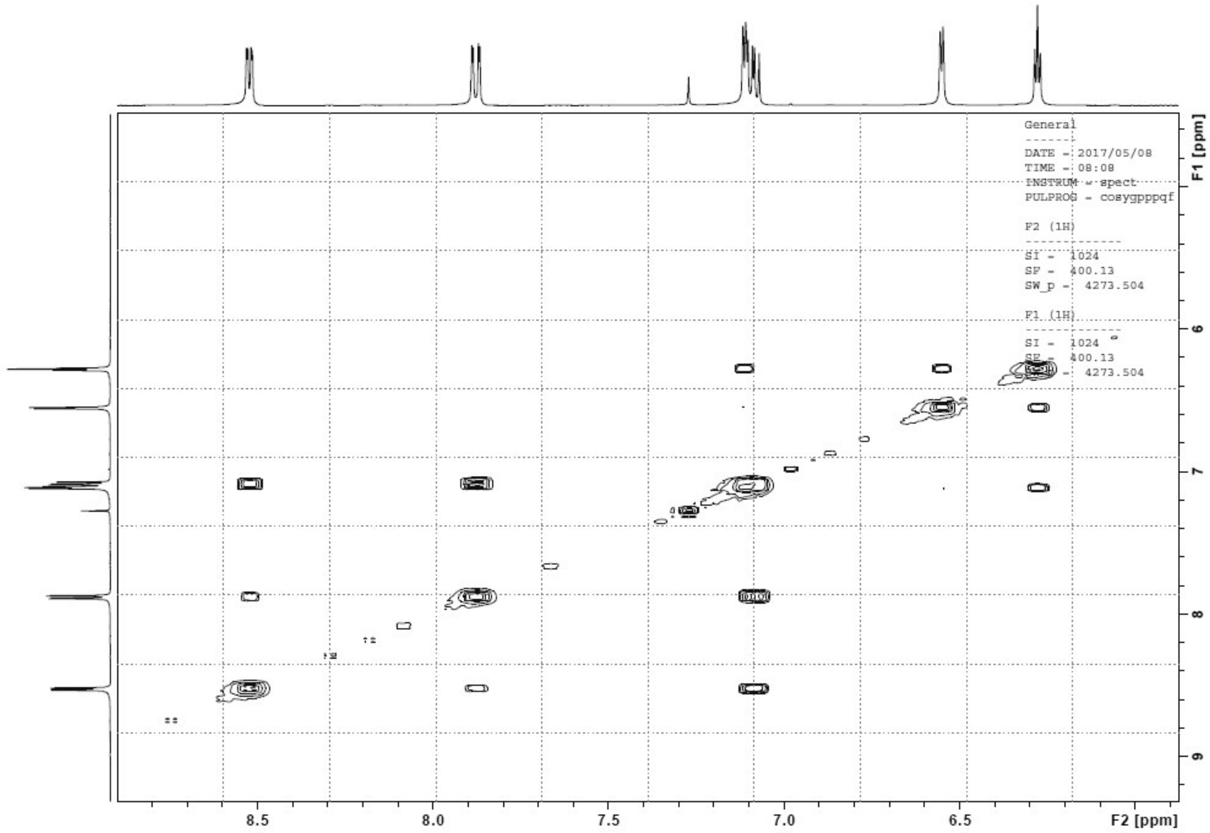
======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

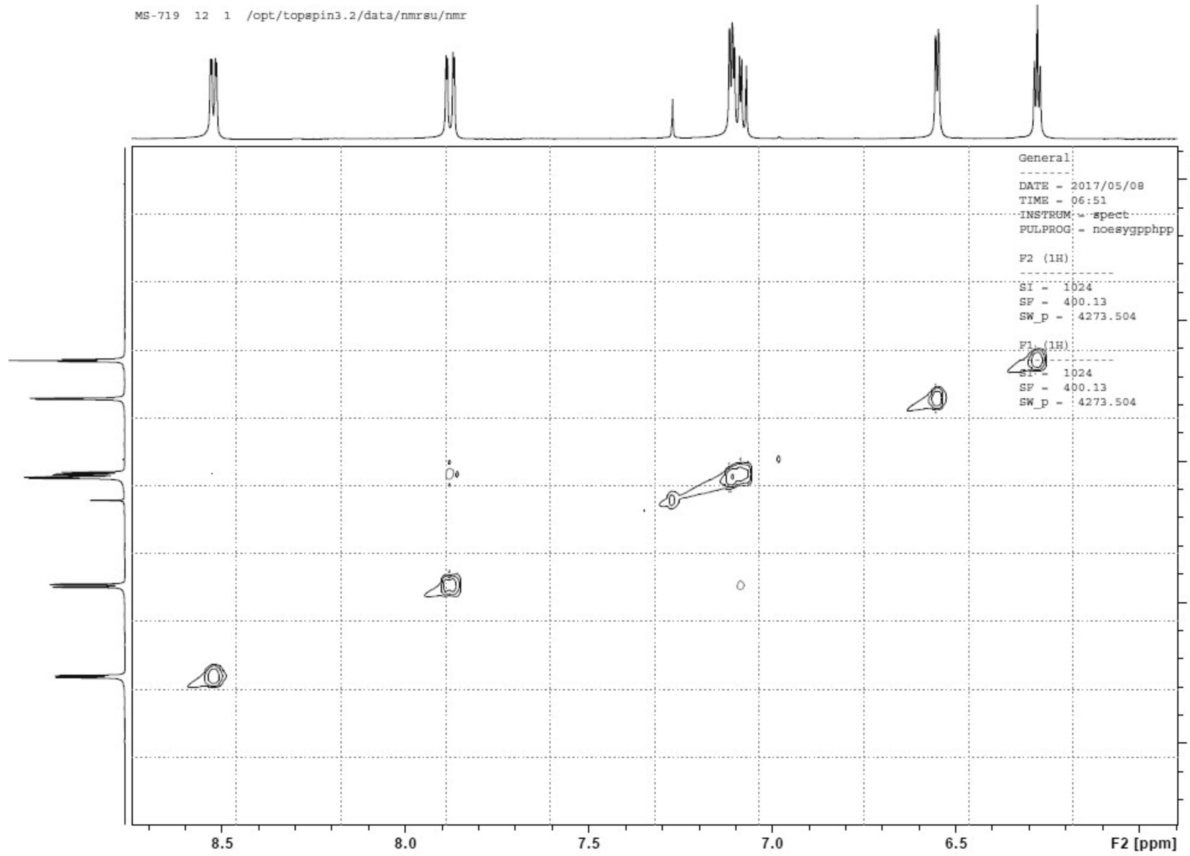
F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

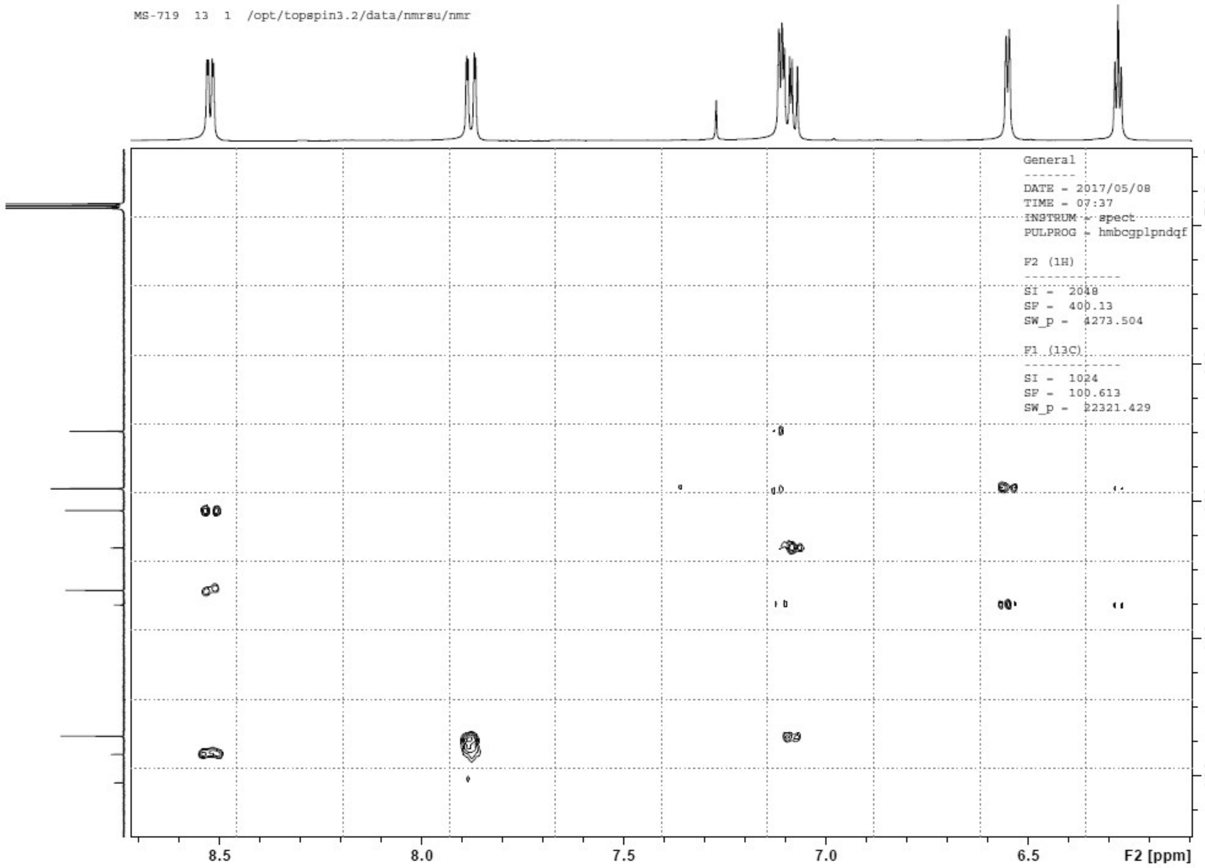
MS-719 14 1 /opt/topspin3.2/data/nmrsu/nmr



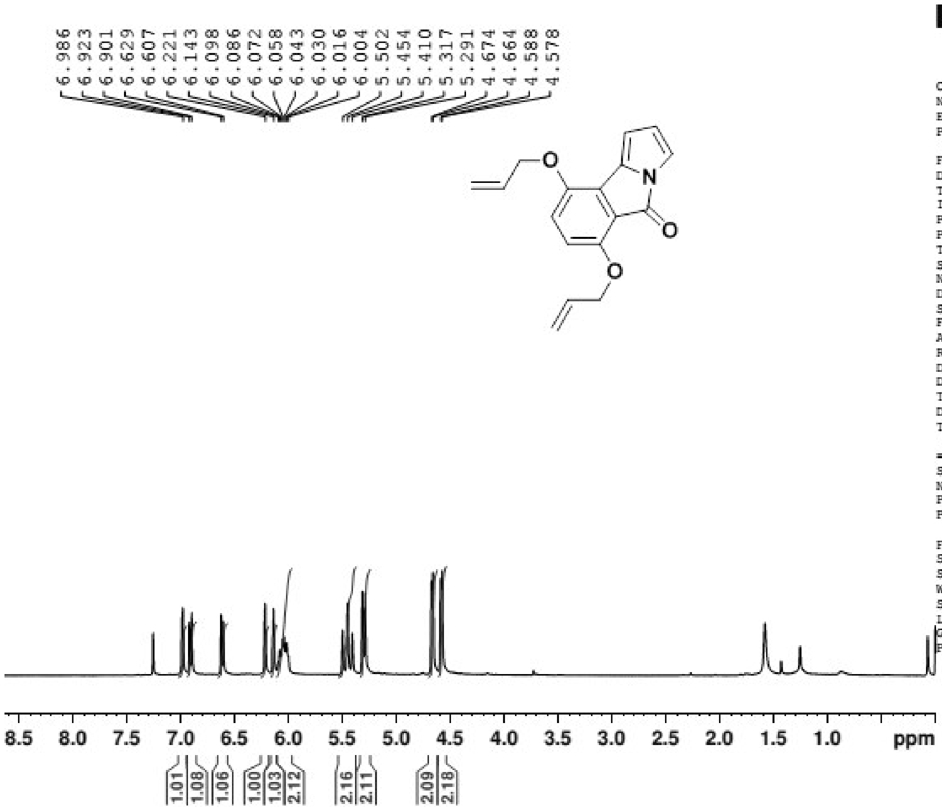
MS-719 15 1 /opt/topspin3.2/data/nmrsu/nmr




MS-719 12 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/05/08
TIME = 06:51
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 4273.504
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 4273.504
F1 [ppm]
6
7
8
9
8.5
8.0
7.5
7.0
6.5
F2 [ppm]


MS-719 13 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/05/08
TIME = 07:37
INSTRUM = spect
PULPROG = hmbcgplpndqf
F2 (1H)
SI = 2048
SF = 400.13
SW_p = 4273.504
F1 (13C)
SI = 1024
SF = 100.613
SW_p = 22321.429
F1 [ppm]
100
120
140
160
8.5
8.0
7.5
7.0
6.5
F2 [ppm]

Compound **14i**

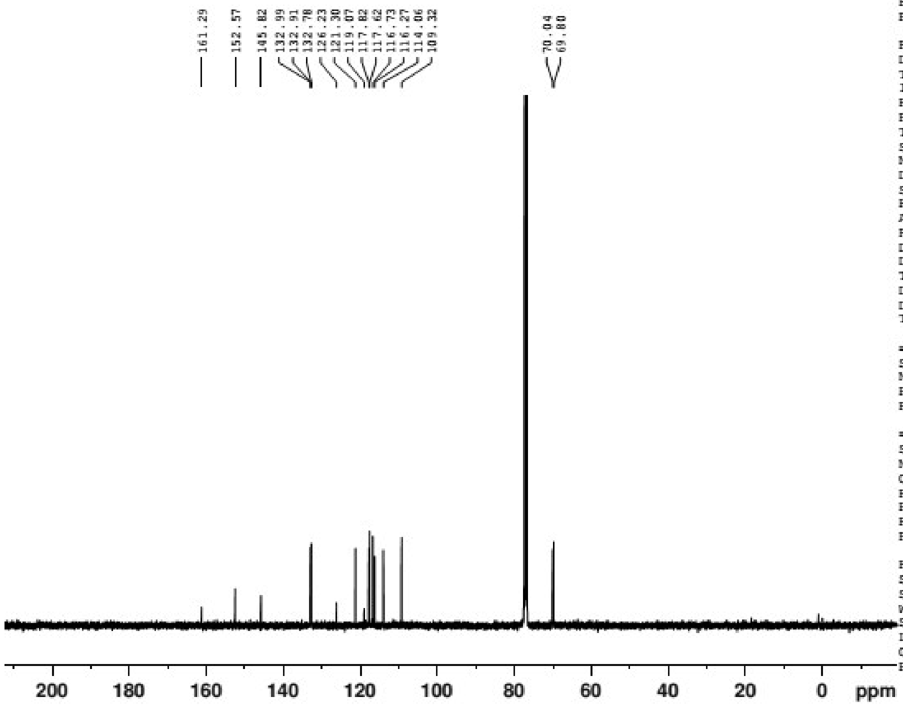


```
Current Data Parameters
NAME              MS-739
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170606
Time               11.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 300.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME              MS-739
EXPNO                 11
PROCNO                 1

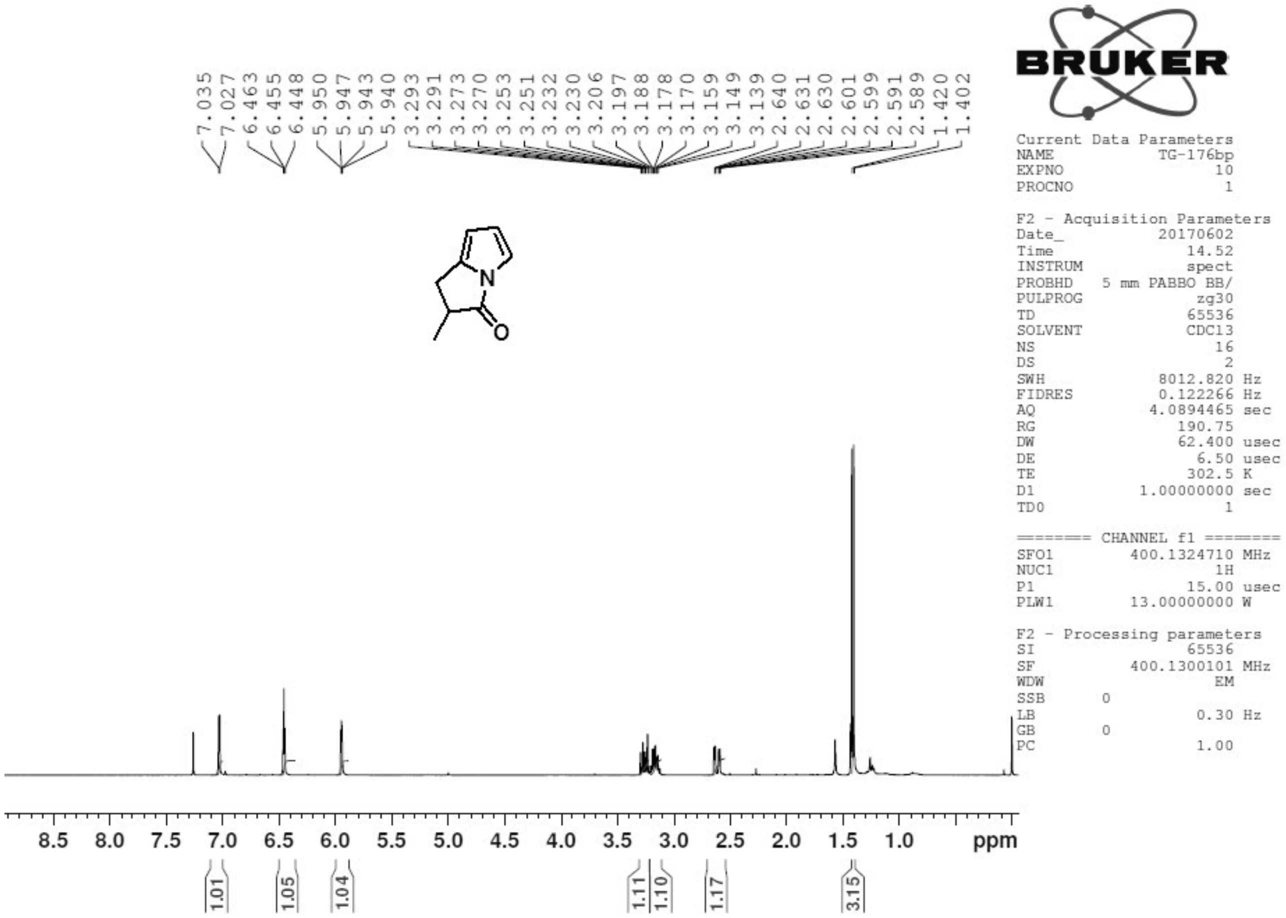
F2 - Acquisition Parameters
Date_           20170606
Time               14.03
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 302.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

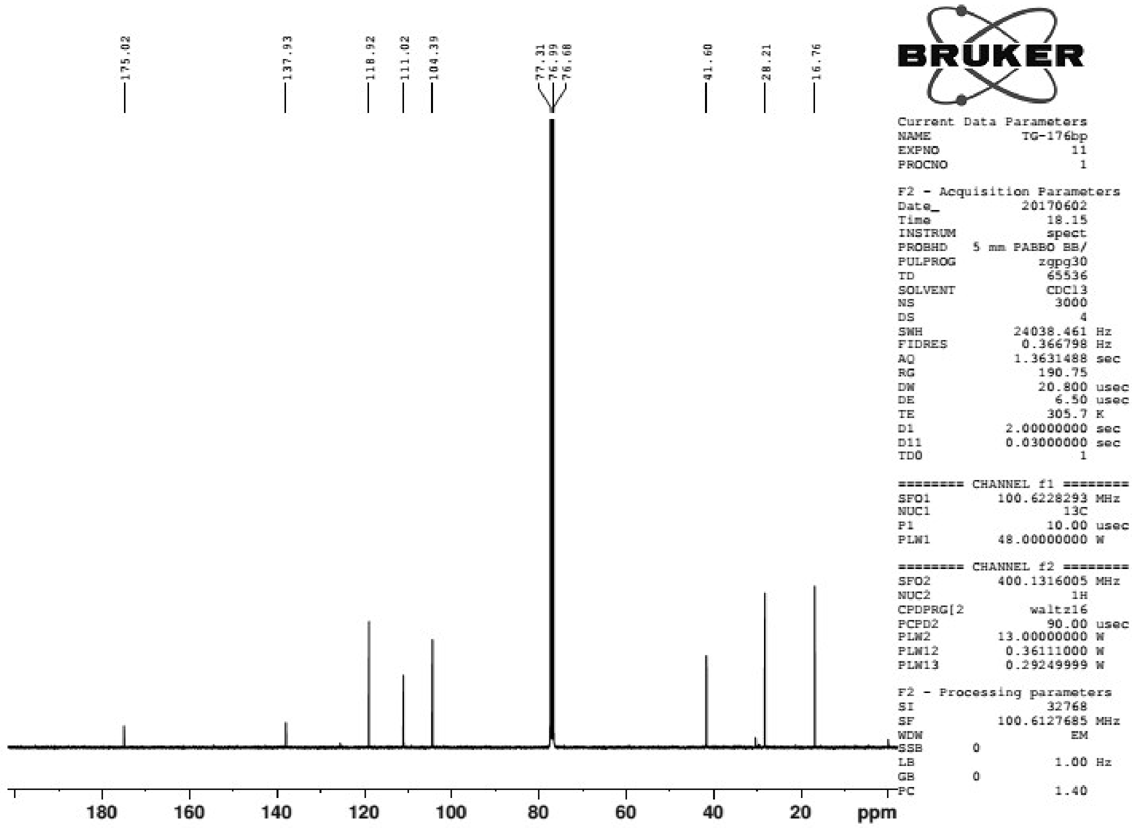
======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

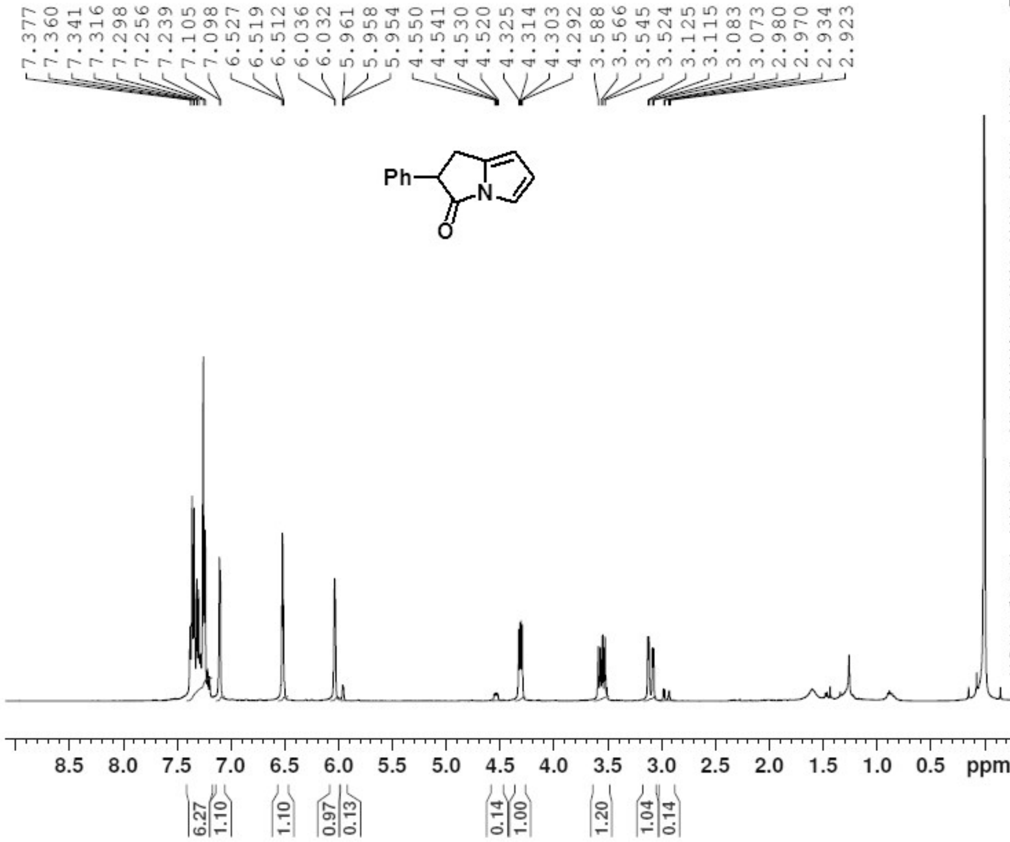
F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

Compound **14j**





Compound **14k**

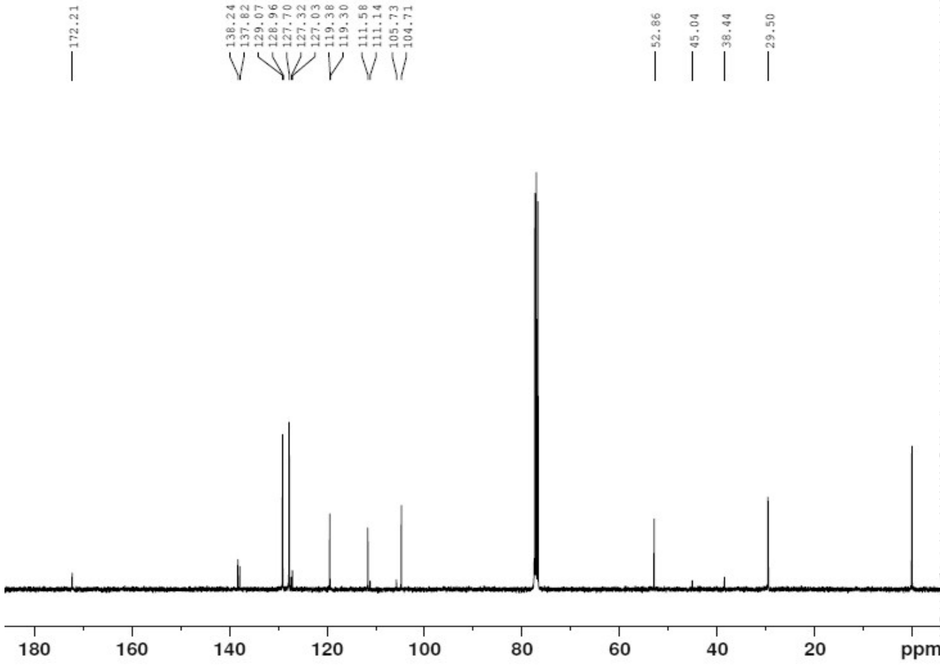


```
Current Data Parameters
NAME             PJ-237K
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171211
Time               12.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 296.0 K
D1            1.00000000 sec
TD0                    1

======= CHANNEL f1 =======
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300117 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME             PJ-237K
EXPNO                 11
PROCNO                 1

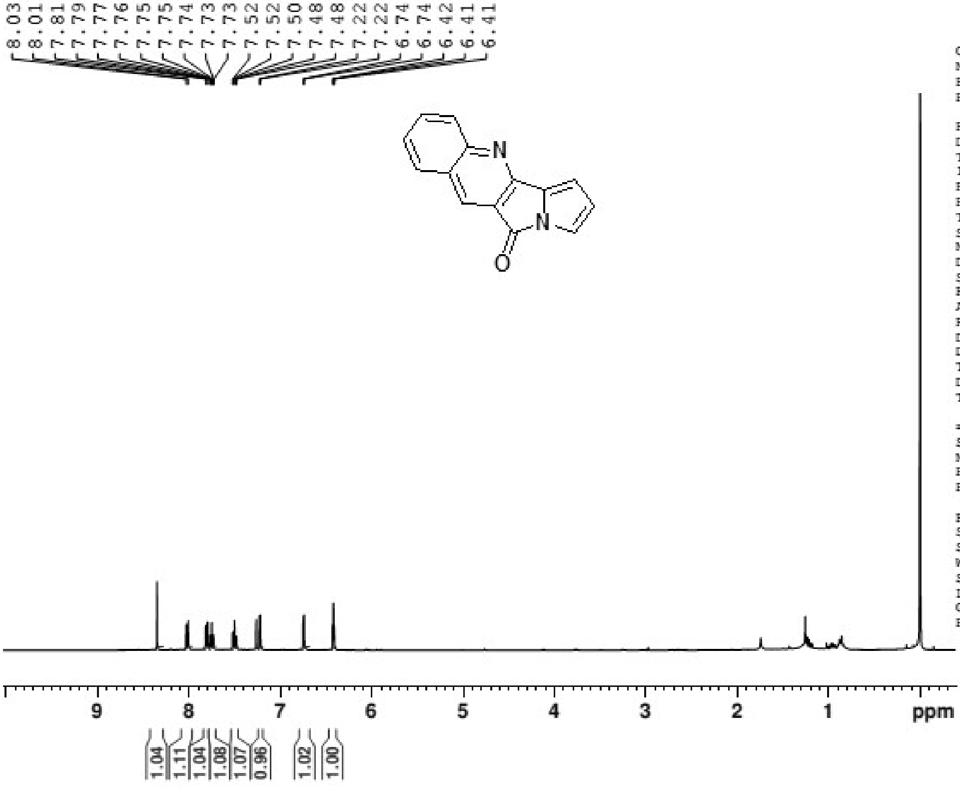
F2 - Acquisition Parameters
Date_           20171211
Time               13.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   931
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======= CHANNEL f1 =======
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======= CHANNEL f2 =======
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

F2 - Processing parameters
SI                 32768
SF           100.6127689 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

Compound **14l**

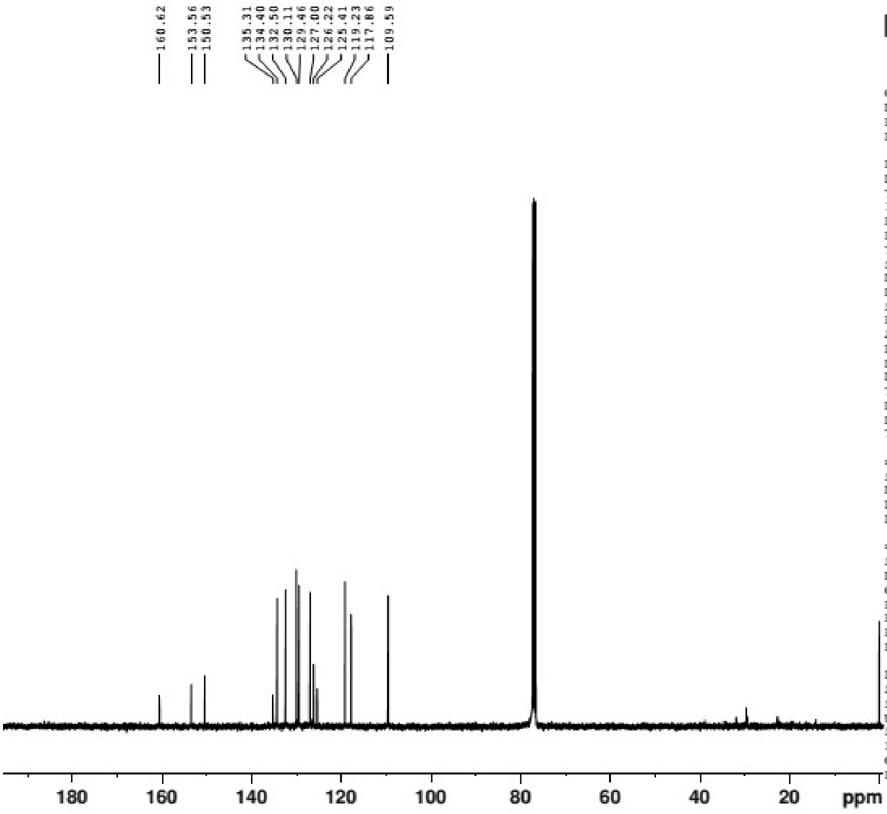


```
Current Data Parameters
NAME               MS-773
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171016
Time                10.53
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 171.45
DW                 62.400 usec
DE                   6.50 usec
TE                  297.4 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1       400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1        13.00000000 W

F2 - Processing parameters
SI                  65536
SF         400.1300078 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME               MS-773
EXPNO                  11
PROCNO                  1

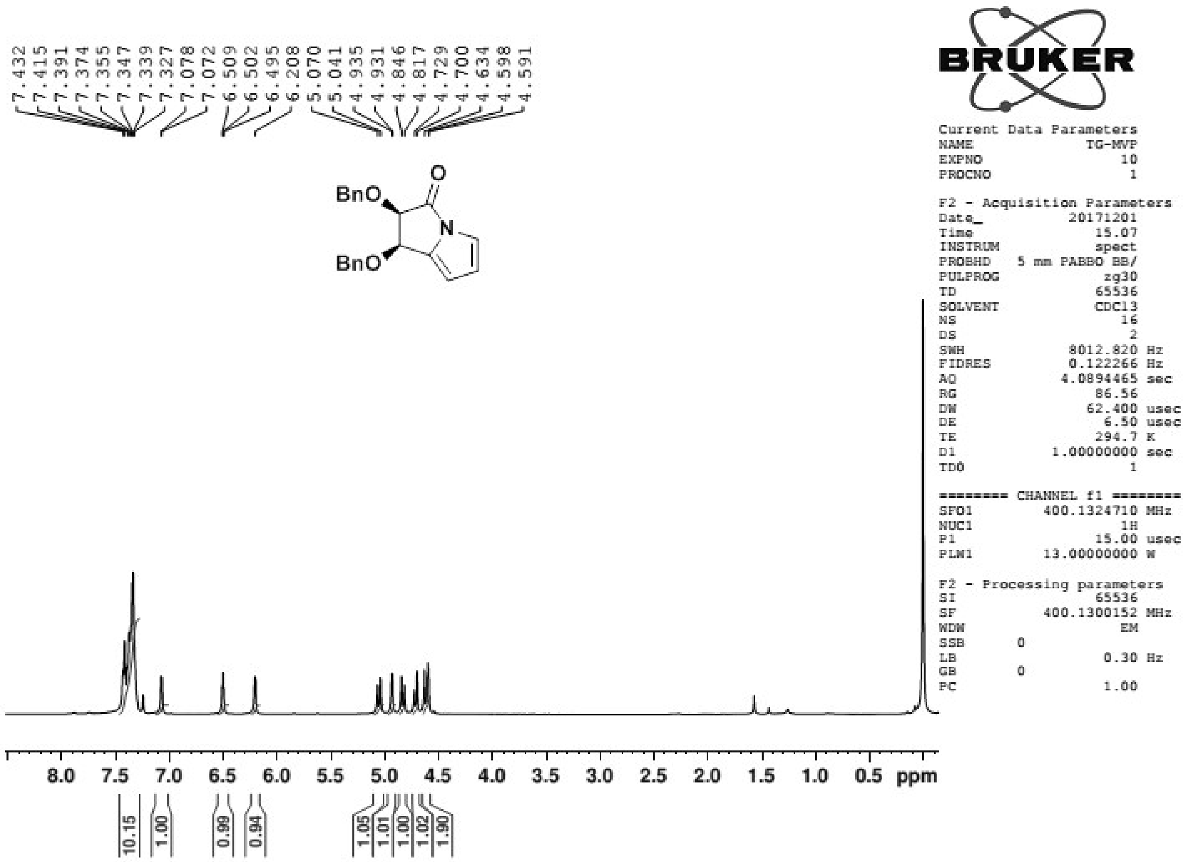
F2 - Acquisition Parameters
Date_            20171017
Time                16.47
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  301.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1       100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2       400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2        13.00000000 W
PLW12        0.36111000 W
PLW13        0.29249999 W

F2 - Processing parameters
SI                  32768
SF         100.6127675 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```

Compound **14m**

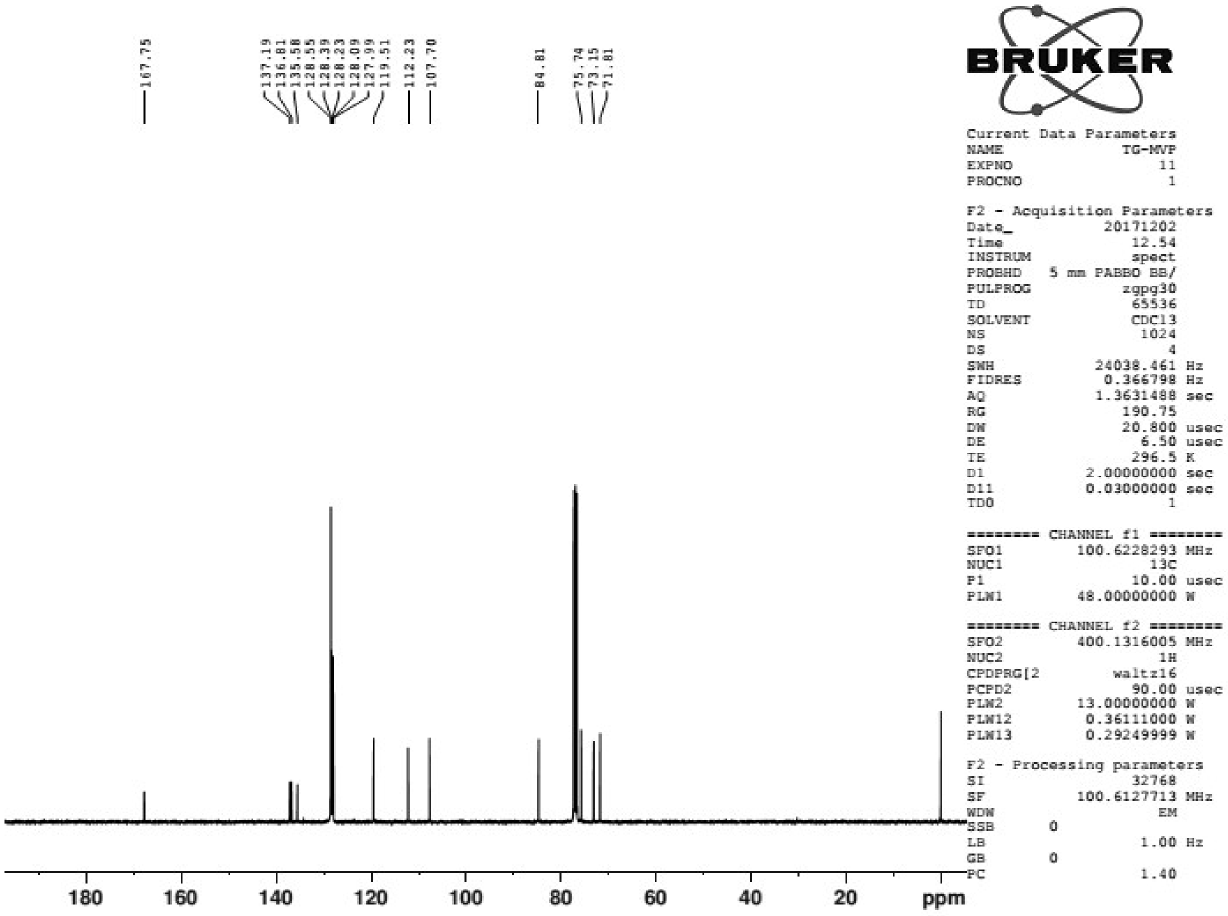


```
Current Data Parameters
NAME               TG-MVP
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171201
Time                15.07
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG                  86.56
DW              62.400 usec
DE                6.50 usec
TE               294.7 K
D1         1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1      400.1324710 MHz
NUC1                   1H
P1              15.00 usec
PLW1       13.00000000 W

F2 - Processing parameters
SI                  65536
SF        400.1300152 MHz
WDW                    EM
SSB      0
LB                0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME               TG-MVP
EXPNO                  11
PROCNO                  1

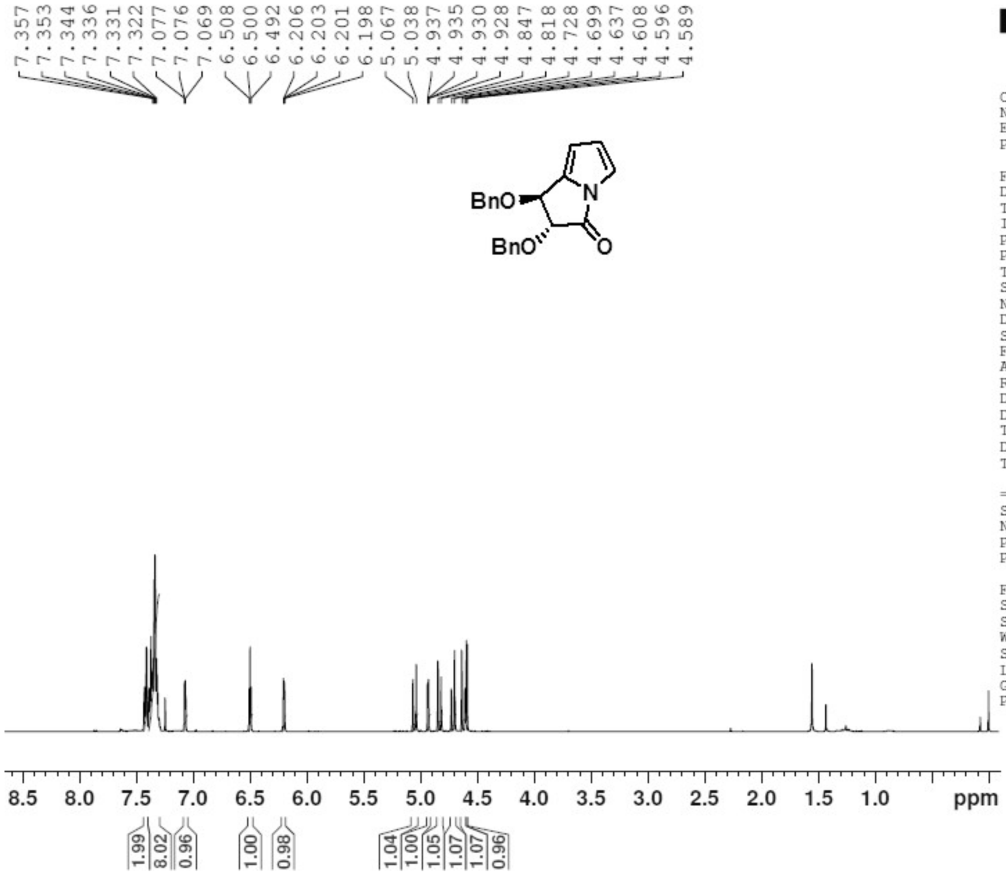
F2 - Acquisition Parameters
Date_            20171202
Time                12.54
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631488 sec
RG                 190.75
DW              20.800 usec
DE                6.50 usec
TE               296.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1      100.6228293 MHz
NUC1                  13C
P1              10.00 usec
PLW1       48.00000000 W

======== CHANNEL f2 ========
SFO2      400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2           90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W
PLW13       0.29249999 W

F2 - Processing parameters
SI                  32768
SF        100.6127713 MHz
WDW                    EM
SSB      0
LB                1.00 Hz
GB       0
PC                   1.40
```

Compound **14n**

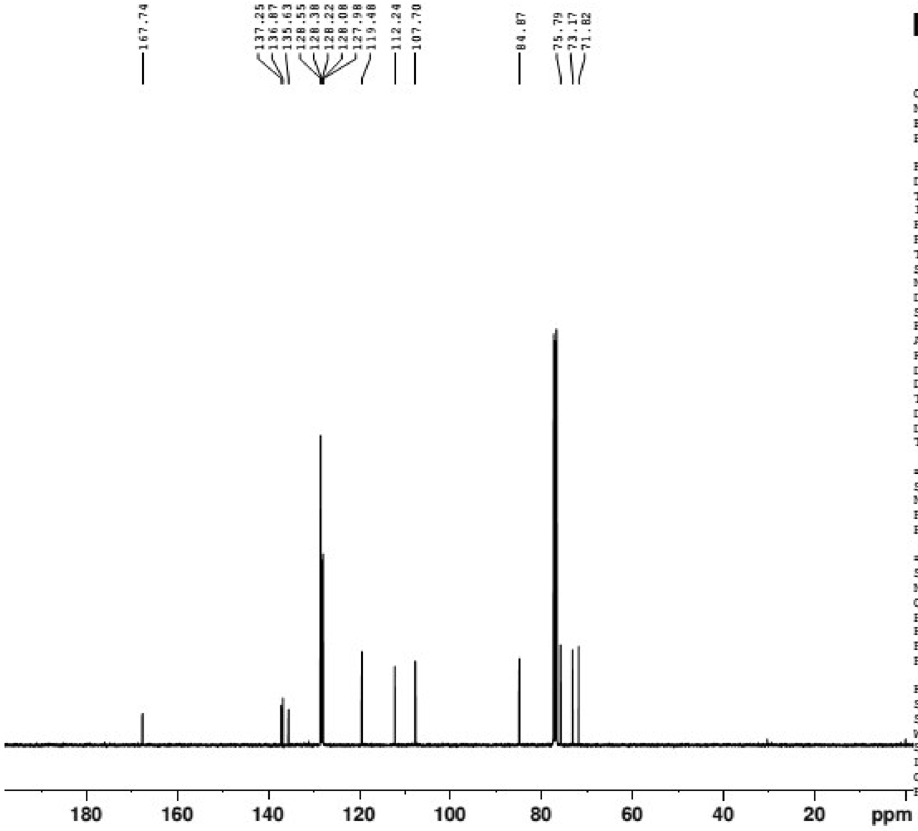


```
Current Data Parameters
NAME              TG-181
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170616
Time                9.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 301.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300147 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME              TG-181
EXPNO                 11
PROCNO                 1

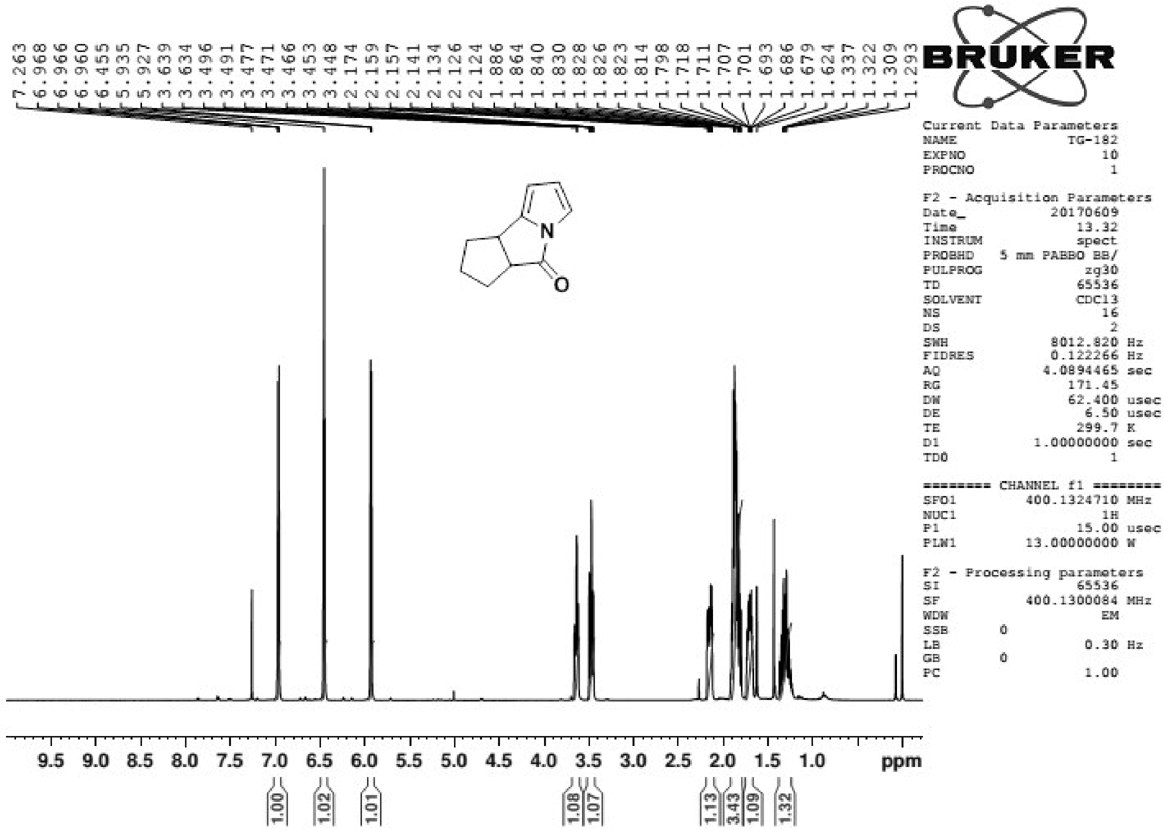
F2 - Acquisition Parameters
Date_           20170612
Time               18.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 304.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

Compound **14o**

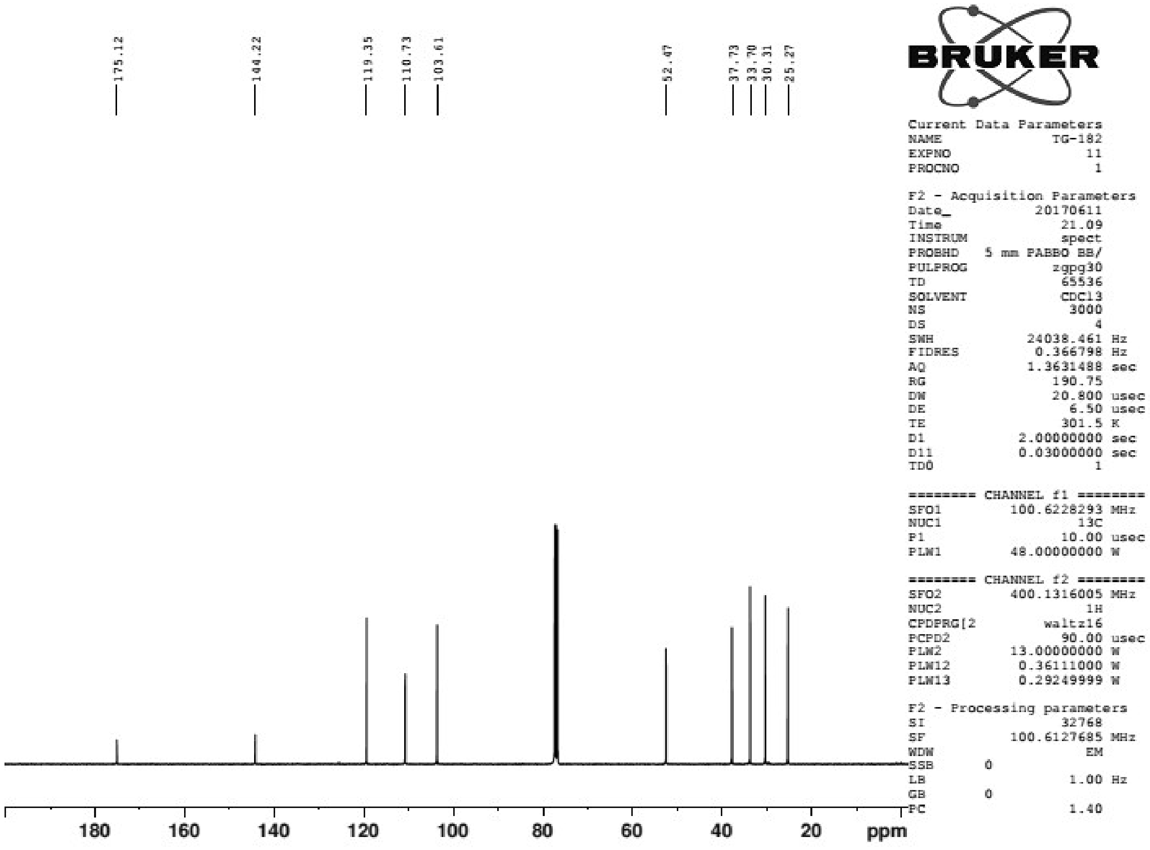


```
Current Data Parameters
NAME                TG-182
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170609
Time                 13.32
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ               4.0894465 sec
RG                  171.45
DW                  62.400 usec
DE                    6.50 usec
TE                   299.7 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                    1H
P1                   15.00 usec
PLW1           13.00000000 W

F2 - Processing parameters
SI                   65536
SF             400.1300084 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



```
Current Data Parameters
NAME                TG-182
EXPNO                   11
PROCNO                   1

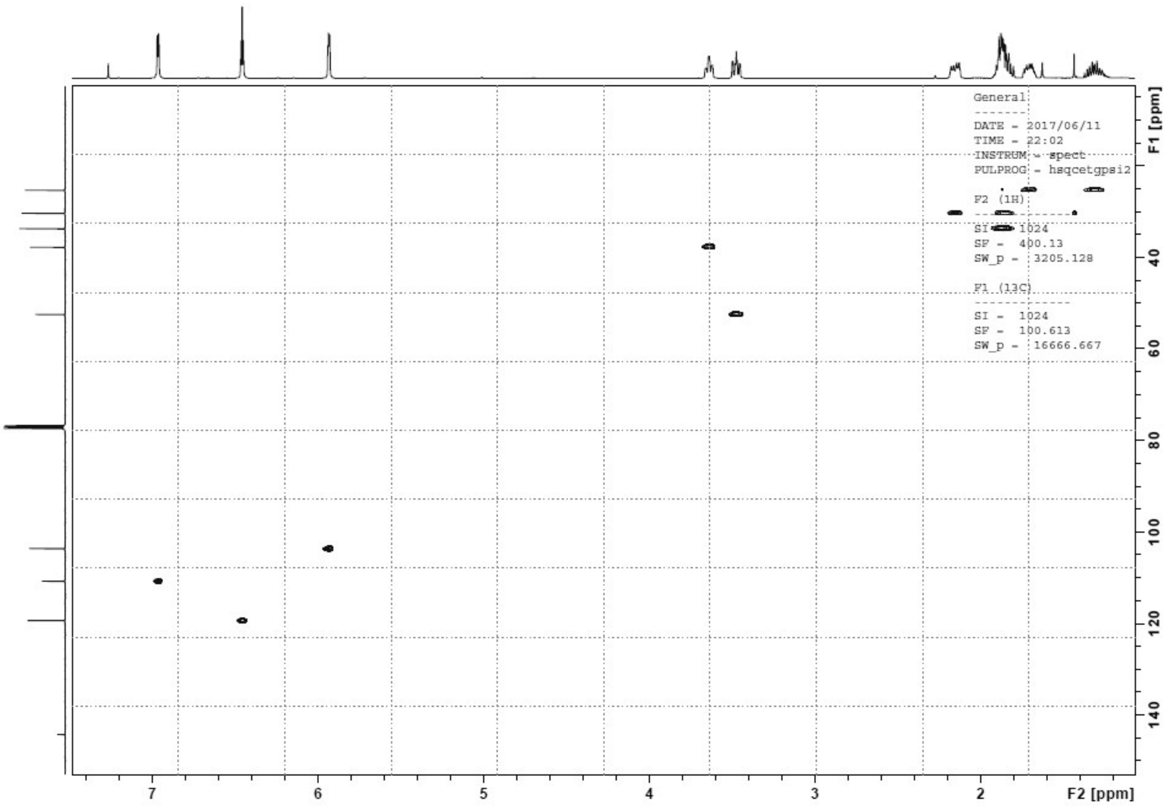
F2 - Acquisition Parameters
Date_             20170611
Time                 21.09
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    3000
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                  190.75
DW                  20.800 usec
DE                    6.50 usec
TE                   301.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                   13C
P1                   10.00 usec
PLW1           48.00000000 W

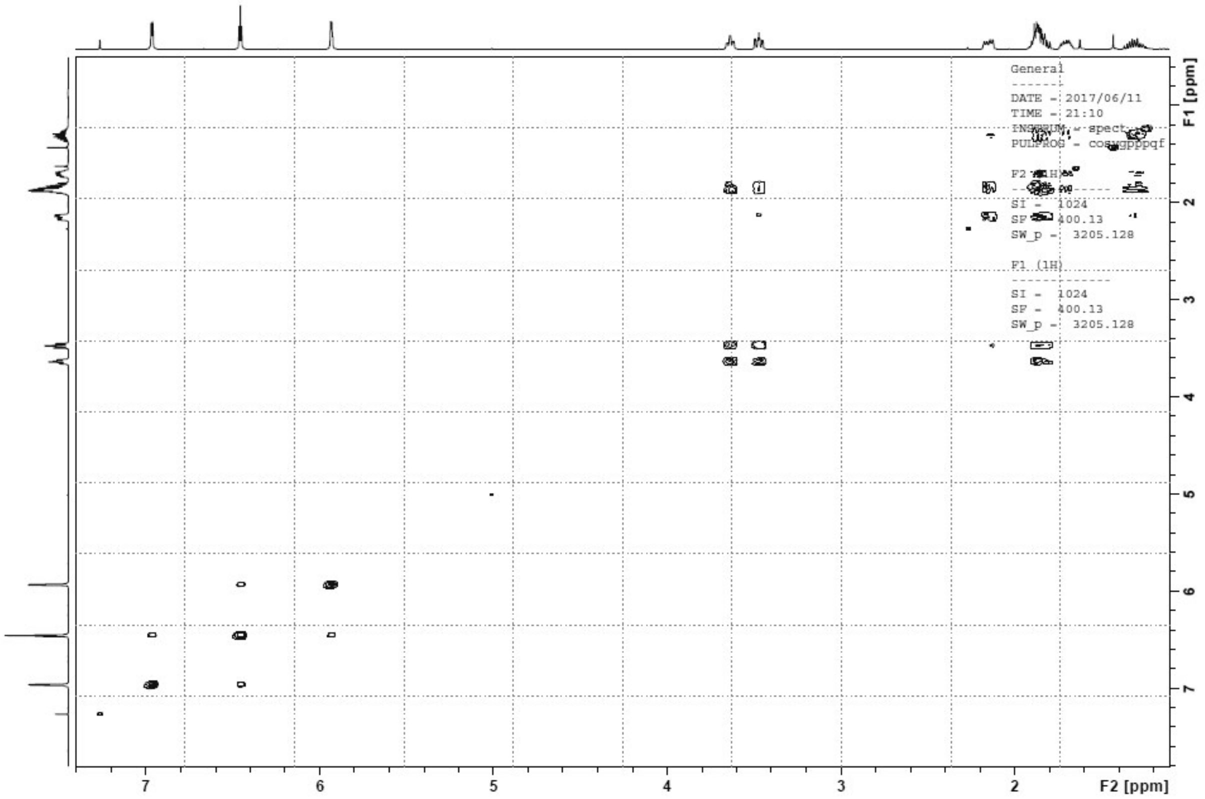
======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2           13.00000000 W
PLW12           0.36111000 W
PLW13           0.29249999 W

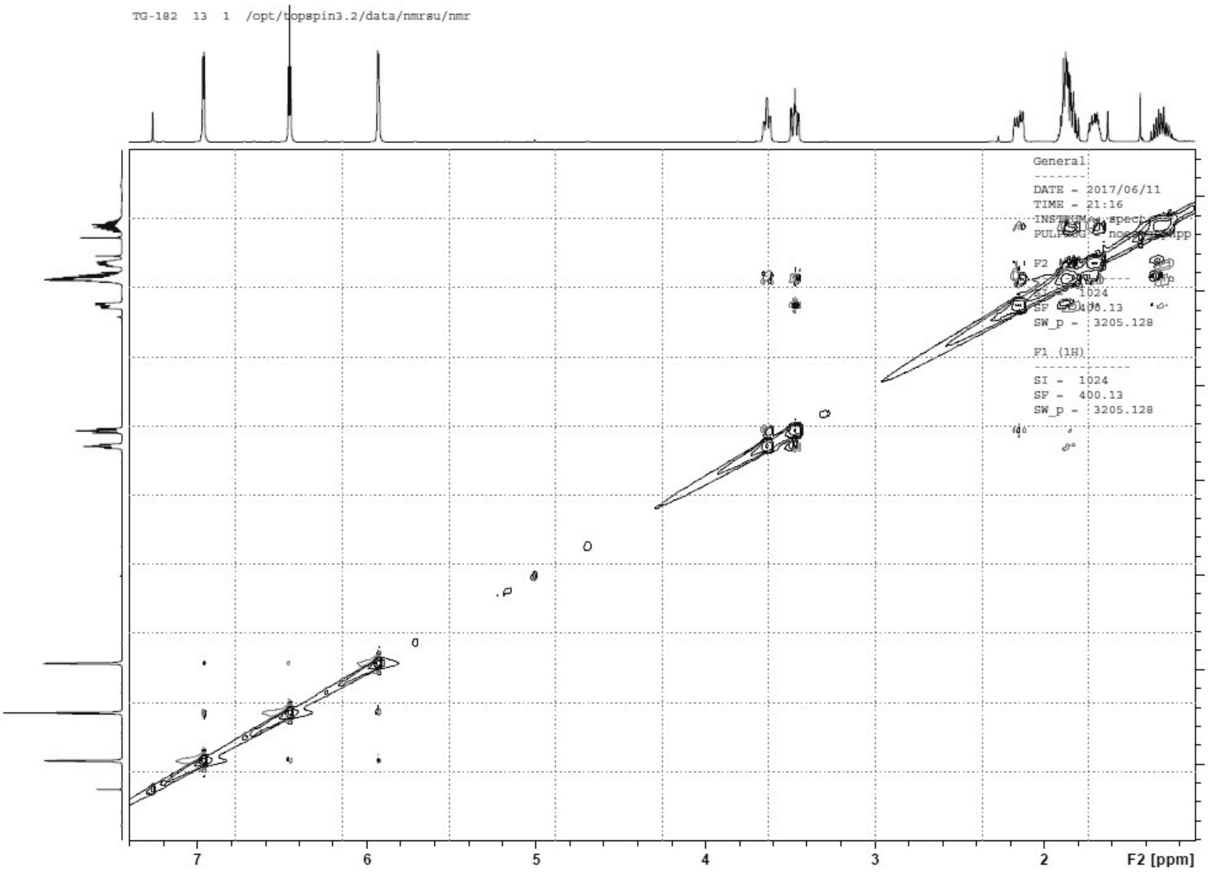
F2 - Processing parameters
SI                   32768
SF             100.6127685 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```

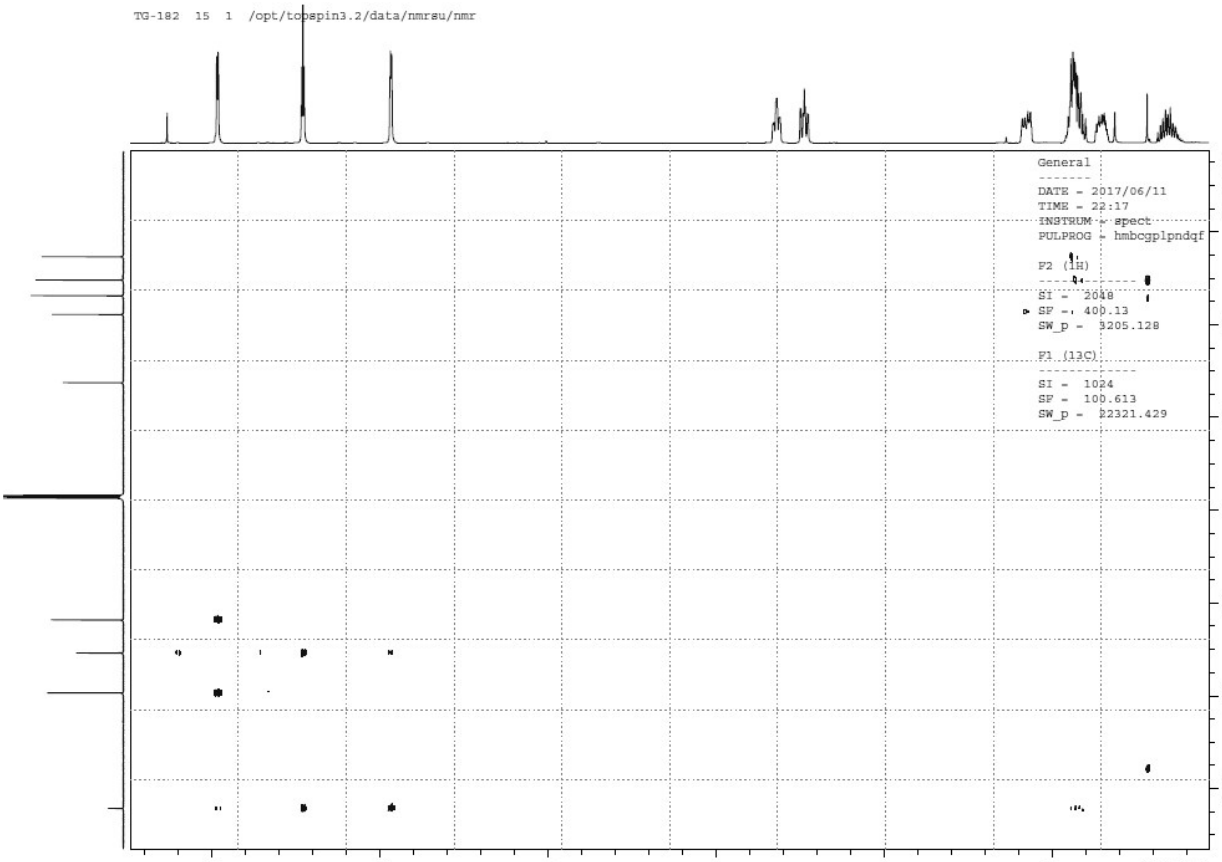
TG-182 14 1 /opt/topspin3.2/data/nmrsu/nmr



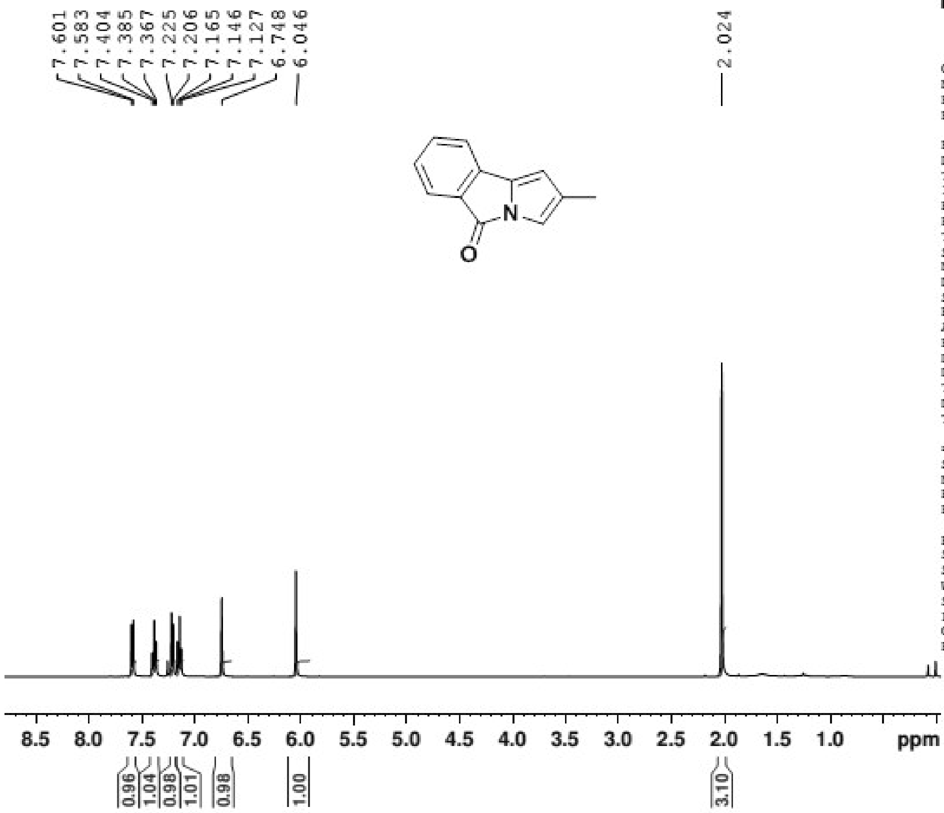
TG-182 12 1 /opt/topspin3.2/data/nmrsu/nmr




TG-182 13 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/06/11
TIME = 21:16
INSTRUM = spect
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3205.128
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 3205.128
F1 [ppm]
F2 [ppm]
2
3
4
5
6
7


TG-182 15 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/06/11
TIME = 22:17
INSTRUM = spect
PULPROG = hmbcgplpndqf
F2 (1H)
SI = 2048
SF = 400.13
SW_p = 3205.128
F1 (13C)
SI = 1024
SF = 100.613
SW_p = 22321.429
F1 [ppm]
F2 [ppm]
40
60
80
100
120
140
7
6
5
4
3
2

Compound **14p**

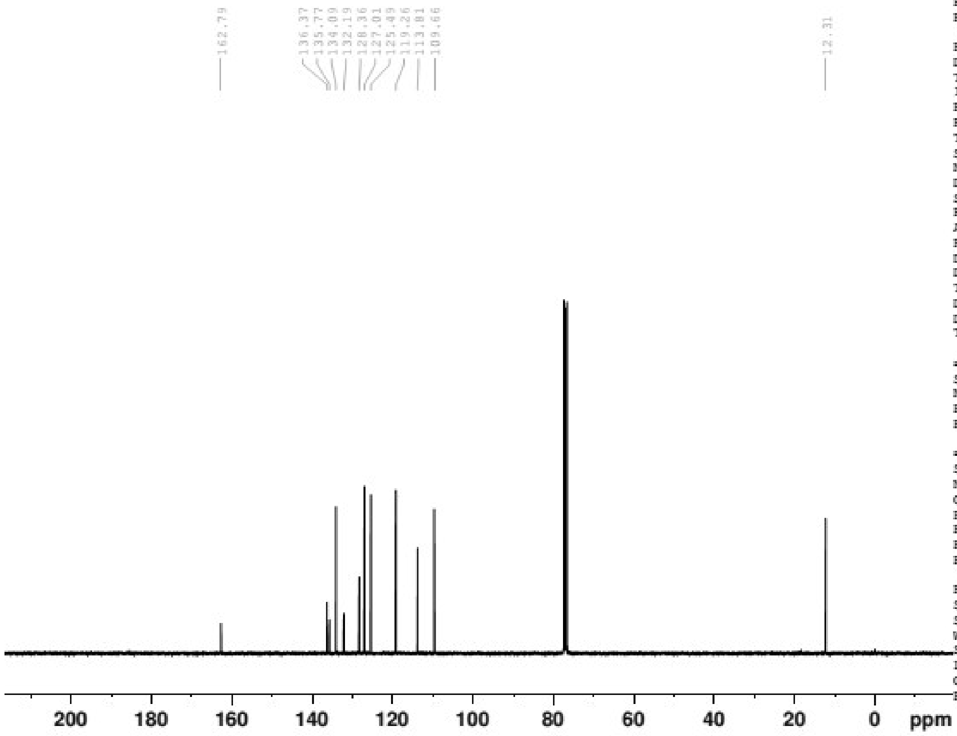


```
Current Data Parameters
NAME              Ms-748
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170623
Time               13.17
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              190.75
DW              62.400 usec
DE                6.50 usec
TE               297.4 K
D1          1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1       400.1324710 MHz
NUC1                  1H
P1               15.00 usec
PLW1       13.00000000 W

F2 - Processing parameters
SI                 65536
SF         400.1300096 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME              Ms-748
EXPNO                 11
PROCNO                 1

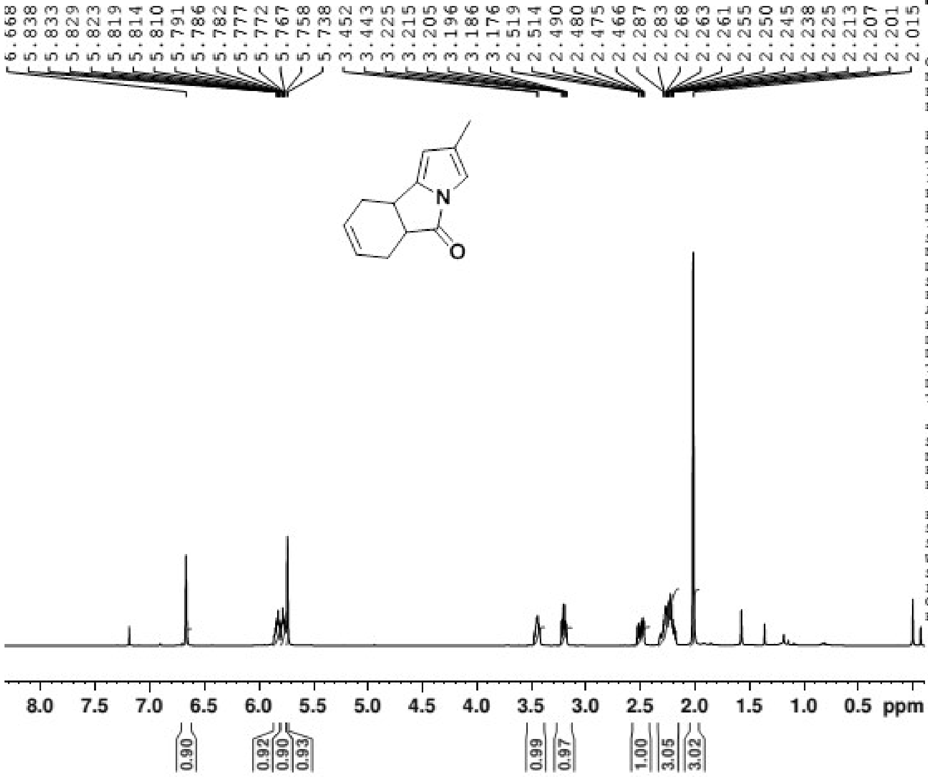
F2 - Acquisition Parameters
Date_           20170625
Time               21.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631488 sec
RG              190.75
DW              20.800 usec
DE                6.50 usec
TE               300.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1       100.6228293 MHz
NUC1                 13C
P1               10.00 usec
PLW1       48.00000000 W

======== CHANNEL f2 ========
SFO2       400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.36111000 W
PLW13       0.29249999 W

F2 - Processing parameters
SI                 32768
SF         100.6127685 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

Compound **14q**

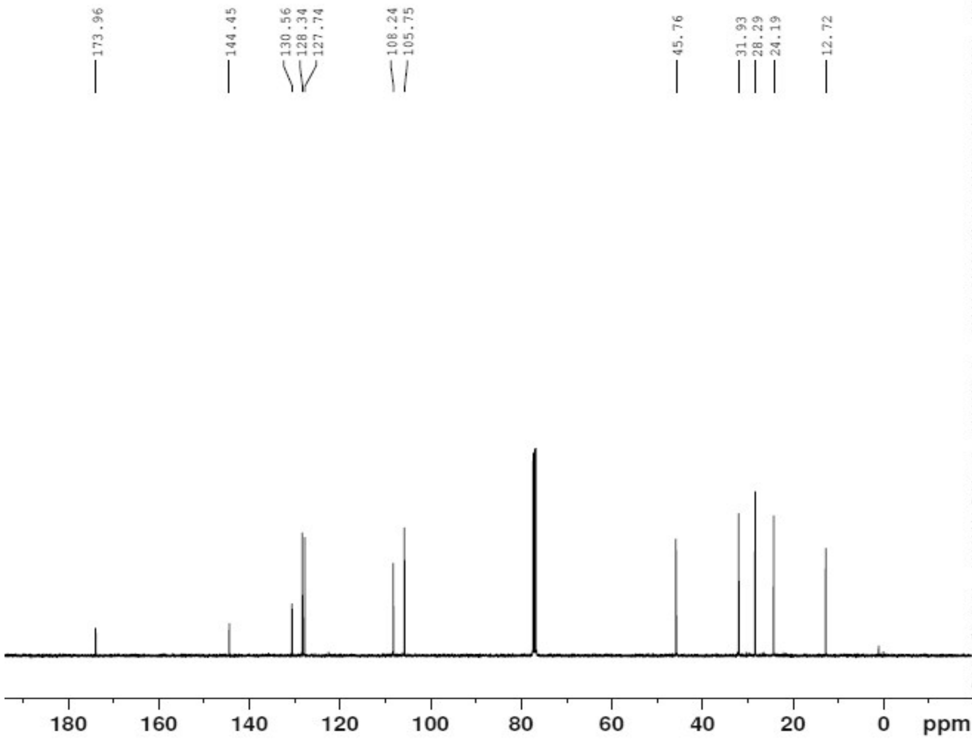


```
Current Data Parameters
NAME              MS-759-2
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_            20170906
Time                15.02
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 109.49
DW                 62.400 usec
DE                   6.50 usec
TE                  299.5 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                     1H
P1                    15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300382 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME              MS-759-2
EXPNO                   11
PROCNO                   1

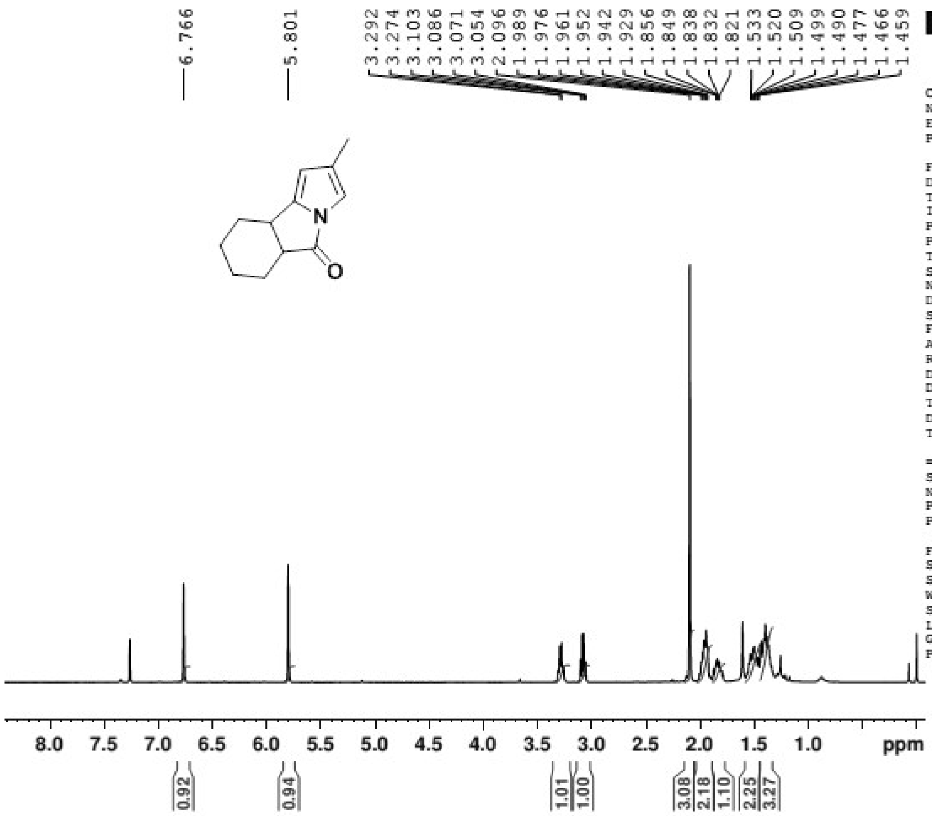
F2 - Acquisition Parameters
Date_            20170906
Time                21.14
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  302.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                    13C
P1                    10.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                     1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```

Compound **14r**

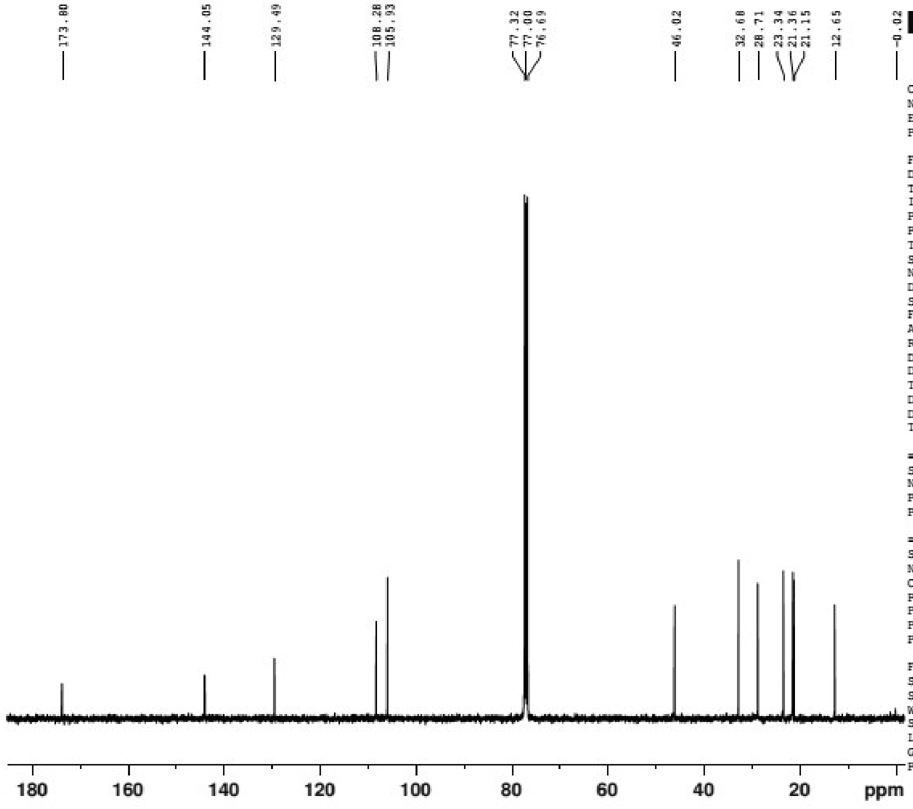


```
Current Data Parameters
NAME             MS-758-2k
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170901
Time                 13.20
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ               4.0894465 sec
RG                  190.75
DW                  62.400 usec
DE                    6.50 usec
TE                   297.6 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                    1H
P1                   15.00 usec
PLW1           13.00000000 W

F2 - Processing parameters
SI                   65536
SF             400.1300086 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



```
Current Data Parameters
NAME             MS-758-2k
EXPNO                   11
PROCNO                   1

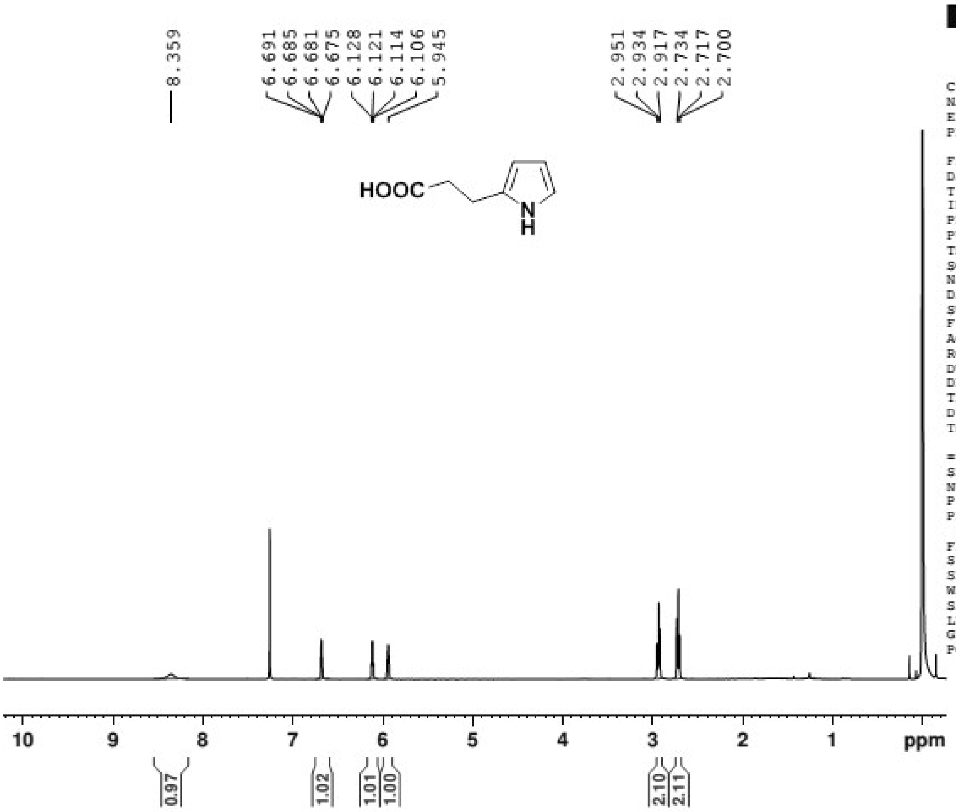
F2 - Acquisition Parameters
Date_             20170901
Time                 21.12
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                  190.75
DW                  20.800 usec
DE                    6.50 usec
TE                   300.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                   13C
P1                   10.00 usec
PLW1           48.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           13.00000000 W
PLW12           0.36111000 W
PLW13           0.29249999 W

F2 - Processing parameters
SI                   32768
SF             100.6127685 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Compound **15a**

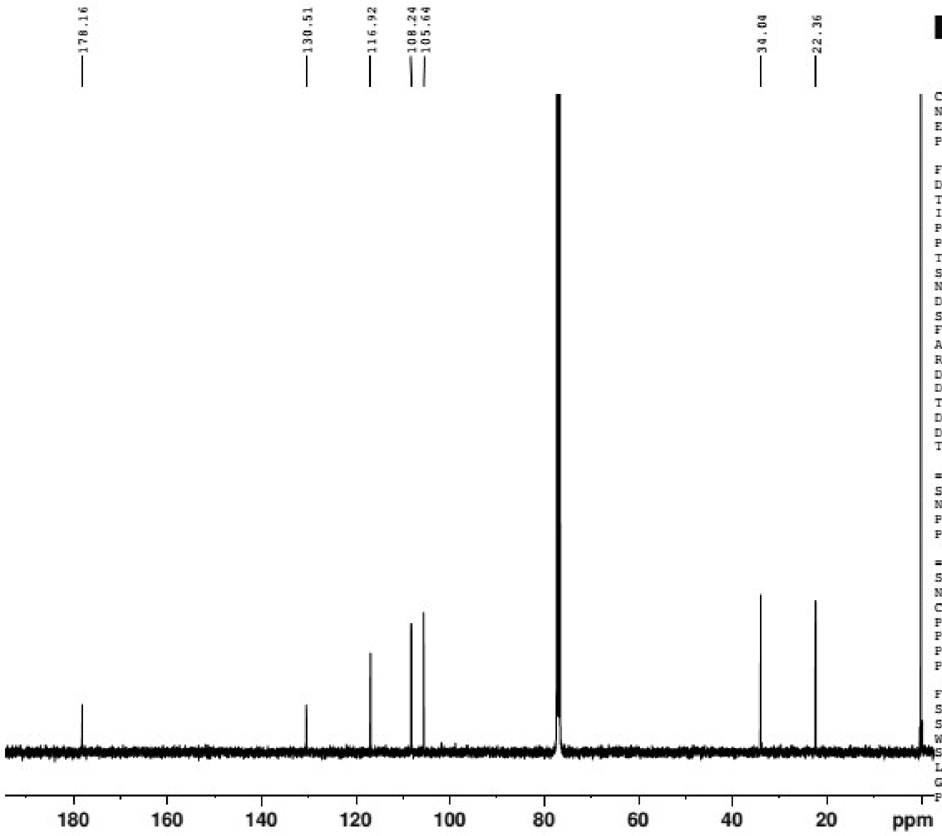


```
Current Data Parameters
NAME              MS-769
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171010
Time               11.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 296.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300104 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



```
Current Data Parameters
NAME              MS-769
EXPNO                 11
PROCNO                 1

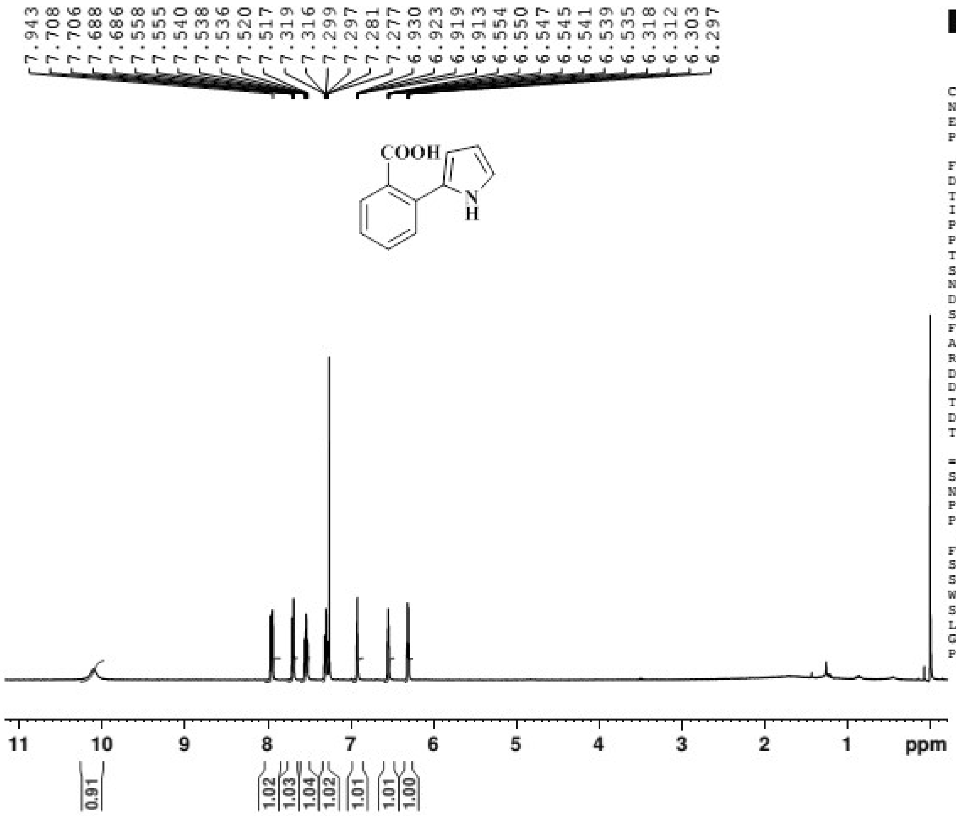
F2 - Acquisition Parameters
Date_           20171011
Time                0.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3000
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 300.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

F2 - Processing parameters
SI                 32768
SF           100.6127674 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

Compound **15b**

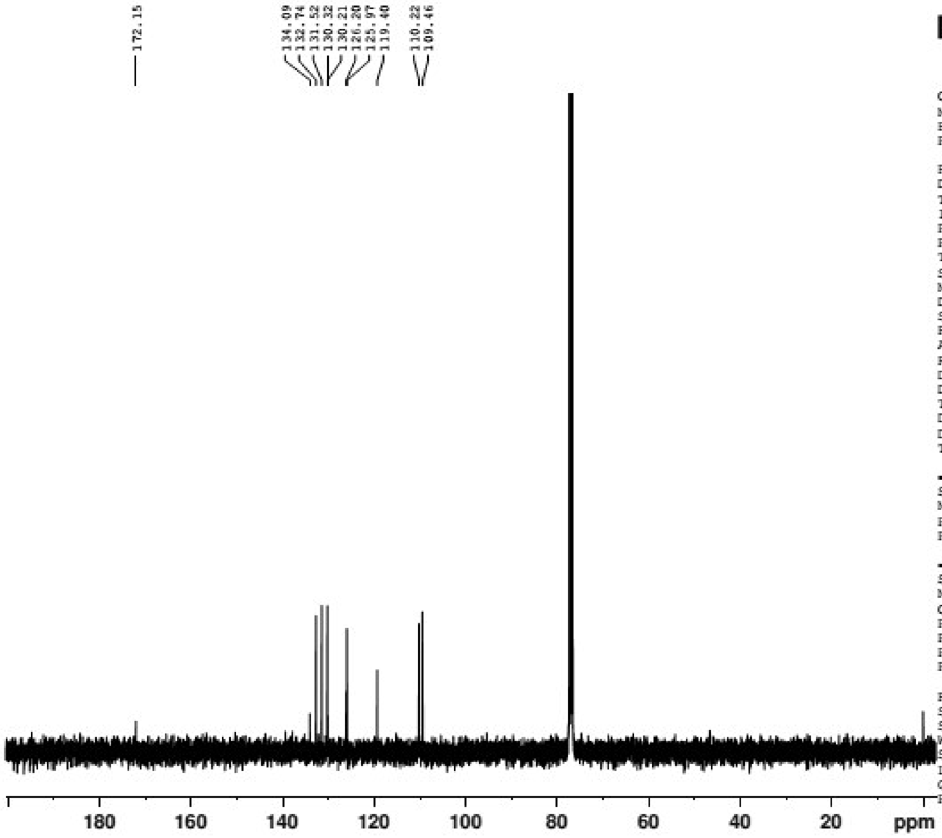


```
Current Data Parameters
NAME                 MS-751
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170908
Time                  10.36
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                4.0894465 sec
RG                   190.75
DW                   62.400 usec
DE                     6.50 usec
TE                    299.4 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                     1H
P1                    15.00 usec
PLW1            13.00000000 W

F2 - Processing parameters
SI                    65536
SF              400.1300103 MHz
WDW                      EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```



```
Current Data Parameters
NAME                 MS-751
EXPNO                    11
PROCNO                    1

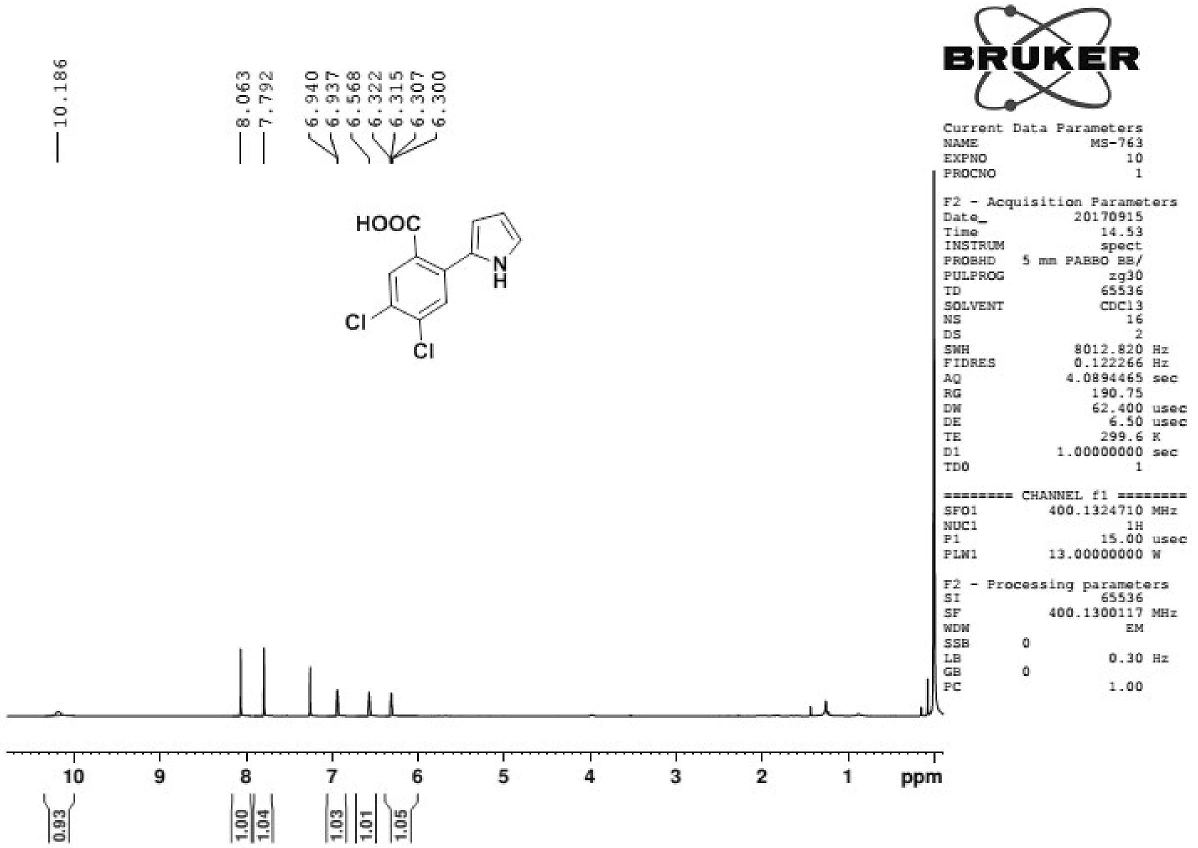
F2 - Acquisition Parameters
Date_              20170909
Time                   2.28
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                   190.75
DW                   20.800 usec
DE                     6.50 usec
TE                    302.8 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6228293 MHz
NUC1                    13C
P1                    10.00 usec
PLW1            48.00000000 W

======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                     1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2            13.00000000 W
PLW12            0.36111000 W
PLW13            0.29249999 W

F2 - Processing parameters
SI                    32768
SF              100.6127685 MHz
WDW                      EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

Compound **15c**

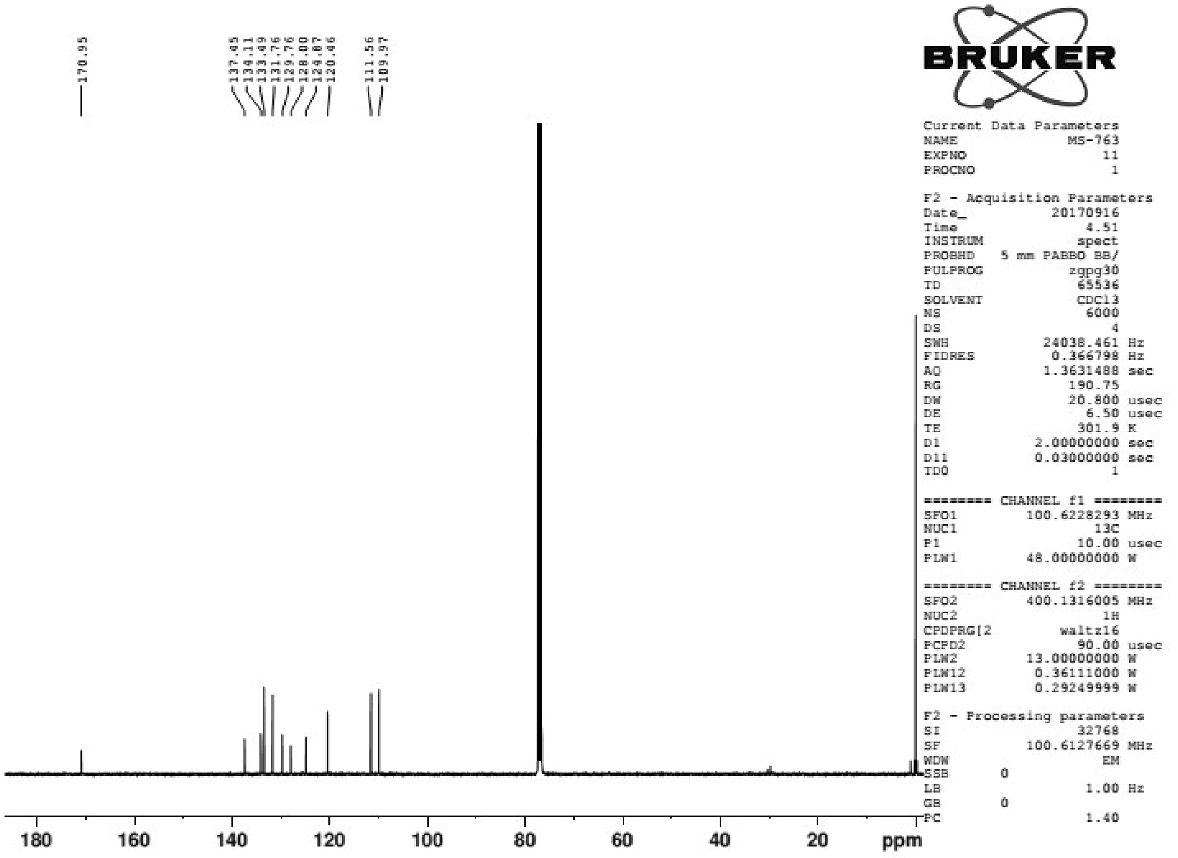


```
Current Data Parameters
NAME               MS-763
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170915
Time                14.53
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 190.75
DW                 62.400 usec
DE                   6.50 usec
TE                  299.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300117 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME               MS-763
EXPNO                  11
PROCNO                  1

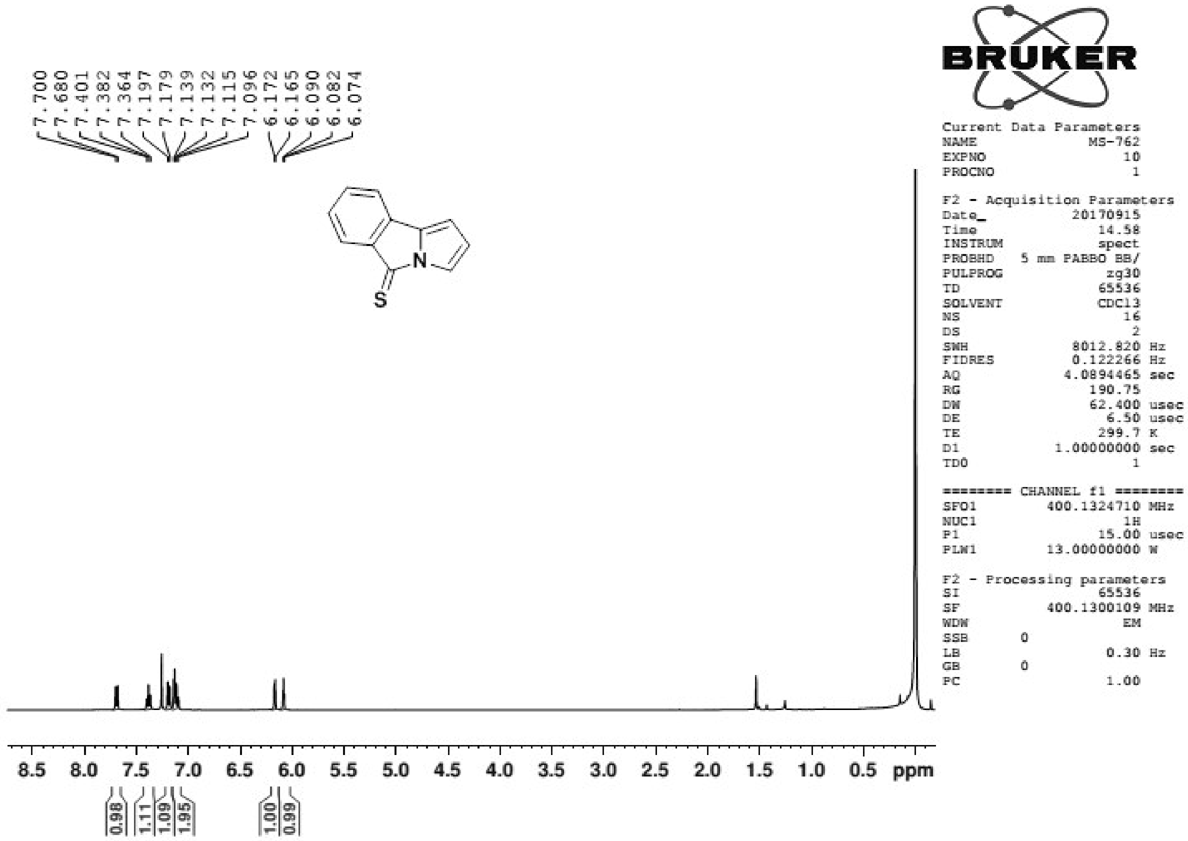
F2 - Acquisition Parameters
Date_            20170916
Time                 4.51
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   6000
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  301.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

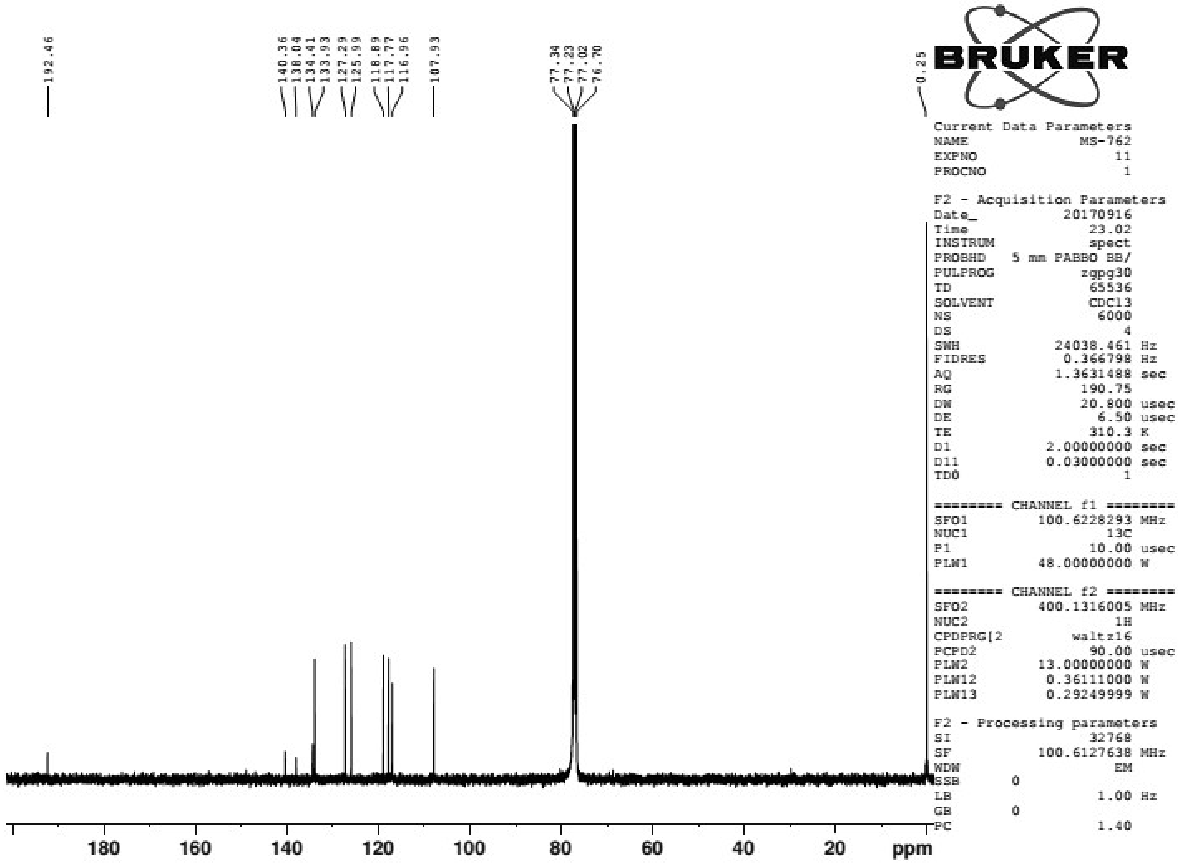
======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

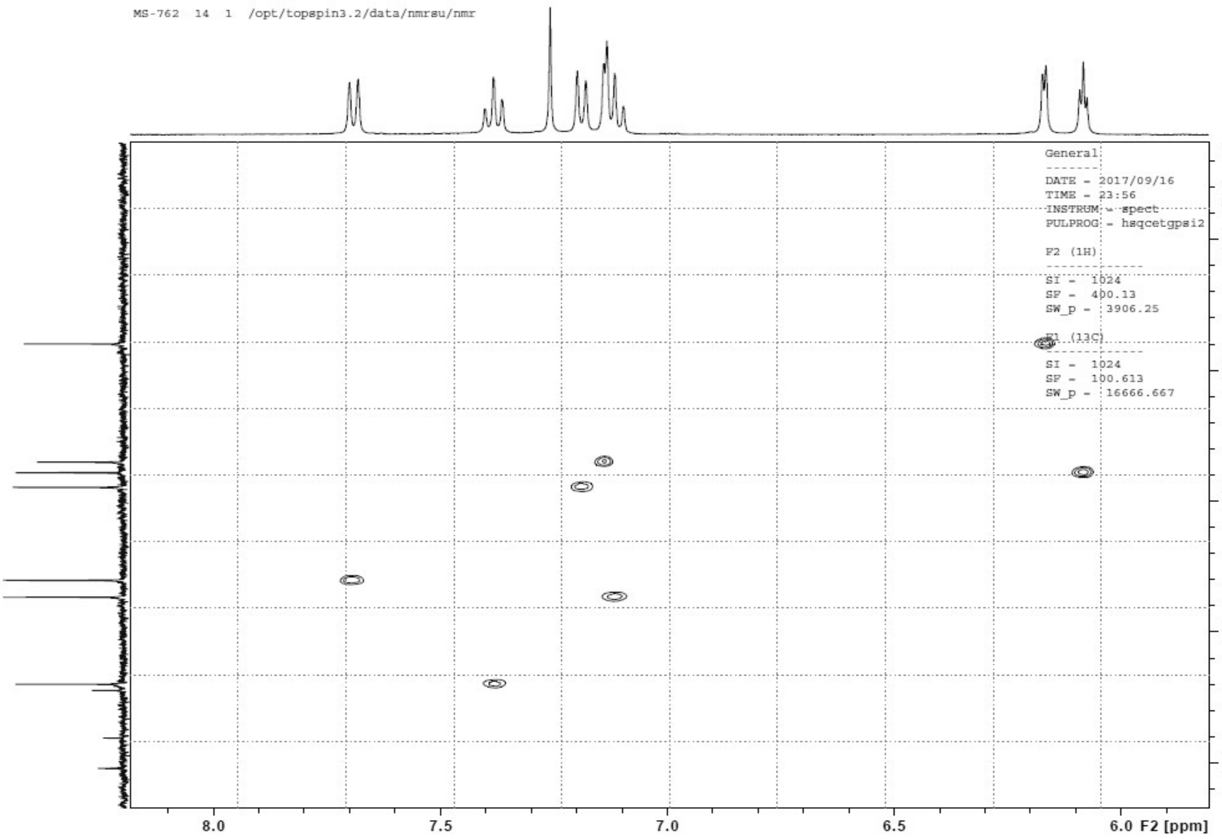
======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

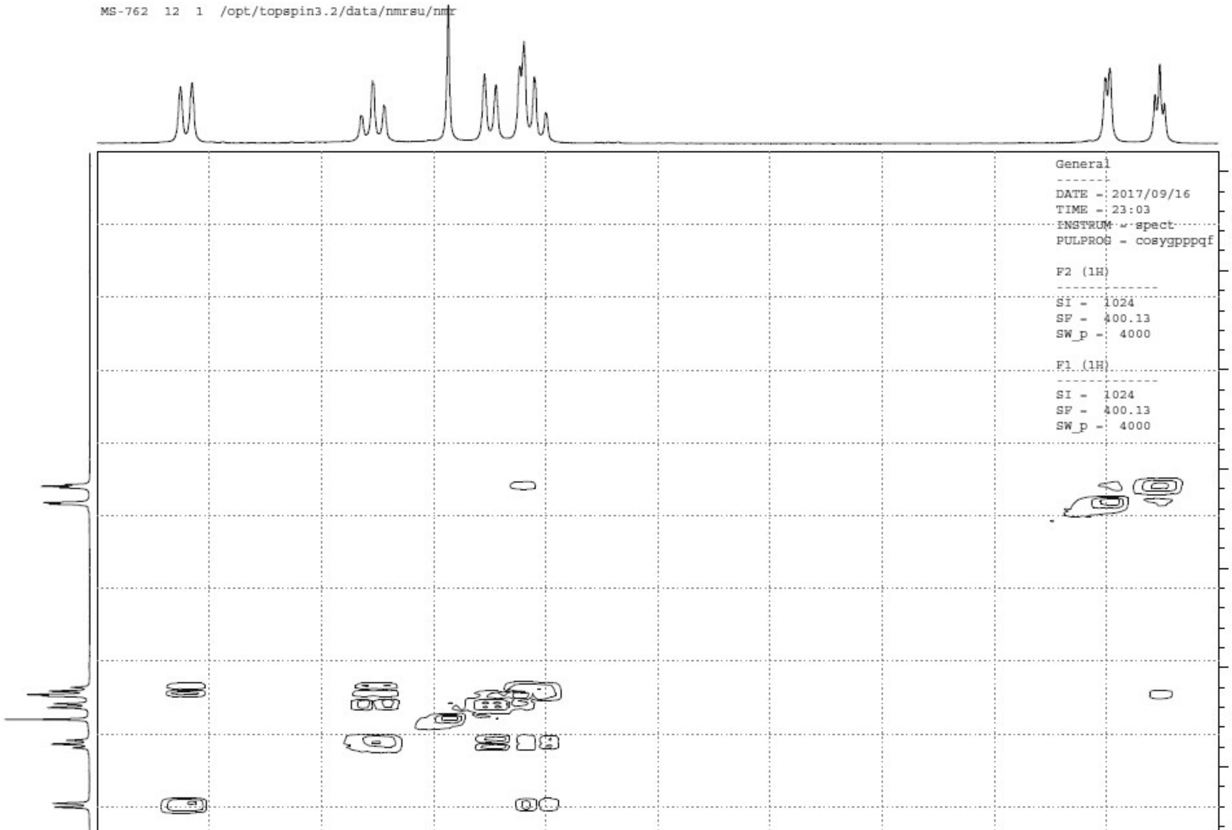
F2 - Processing parameters
SI                  32768
SF            100.6127669 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```

Compound **16**

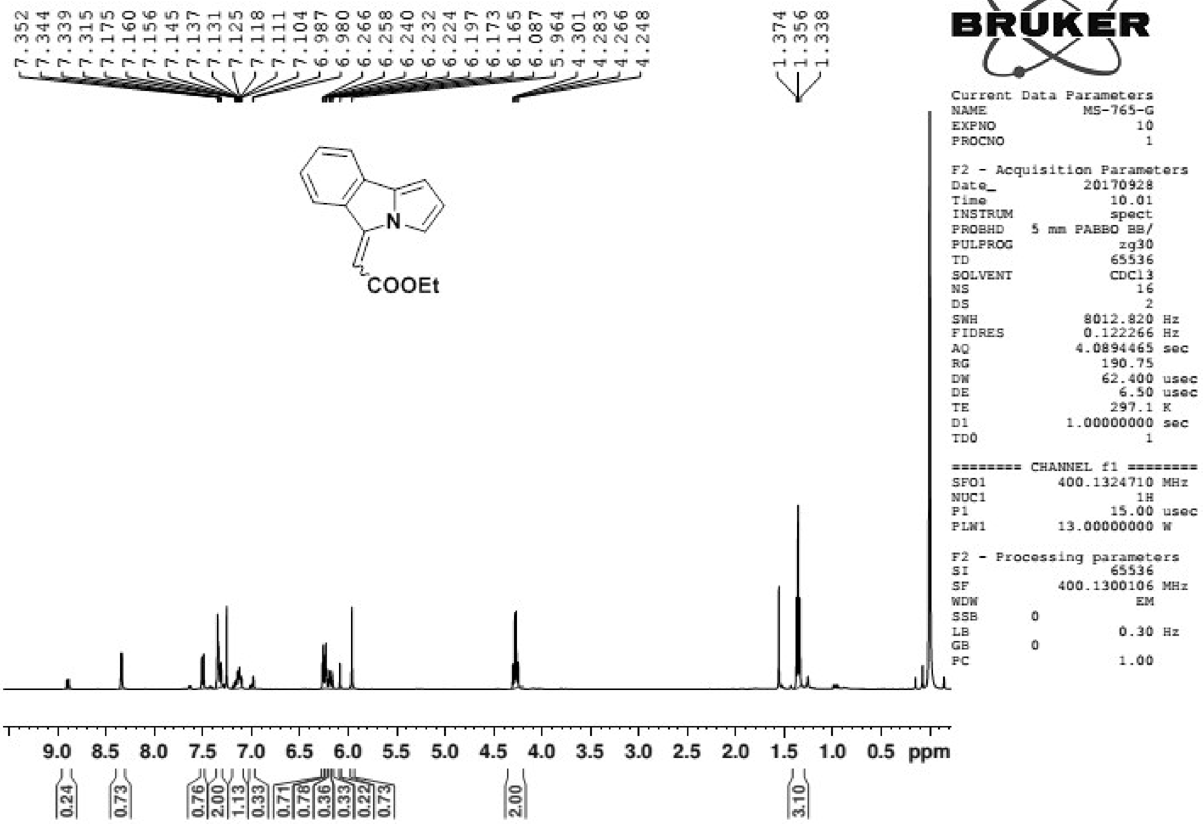

7.700
7.680
7.401
7.382
7.364
7.197
7.179
7.139
7.132
7.115
7.096
6.172
6.165
6.090
6.082
6.074
N
S
BRUKER
Current Data Parameters
NAME MS-762
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170915
Time 14.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 190.75
DW 62.400 usec
DE 6.50 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.98
1.11
1.09
1.95
1.00
0.99


192.46
140.36
138.04
134.41
133.93
127.29
125.99
118.89
117.77
116.96
107.93
77.34
77.23
77.02
76.70
0.25
BRUKER
Current Data Parameters
NAME MS-762
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170916
Time 23.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 190.75
DW 20.800 usec
DE 6.50 usec
TE 310.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PLW1 48.00000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W
PLW13 0.29249999 W
F2 - Processing parameters
SI 32768
SF 100.6127638 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
160
140
120
100
80
60
40
20
ppm


MS-762 14 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/09/16
TIME = 23:56
INSTRUM = spect
PULPROG = hsqcetgpsi2
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3906.25
F1 (13C)
SI = 1024
SF = 100.613
SW_p = 16666.667
F1 [ppm]
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0 F2 [ppm]


MS-762 12 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/09/16
TIME = 23:03
INSTRUM = spect
PULPROG = cosygpppqf
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 4000
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 4000
F1 [ppm]
5.0
5.5
6.0
6.5
7.0
7.5
7.5
7.0
6.5
F2 [ppm]

Compound **17**

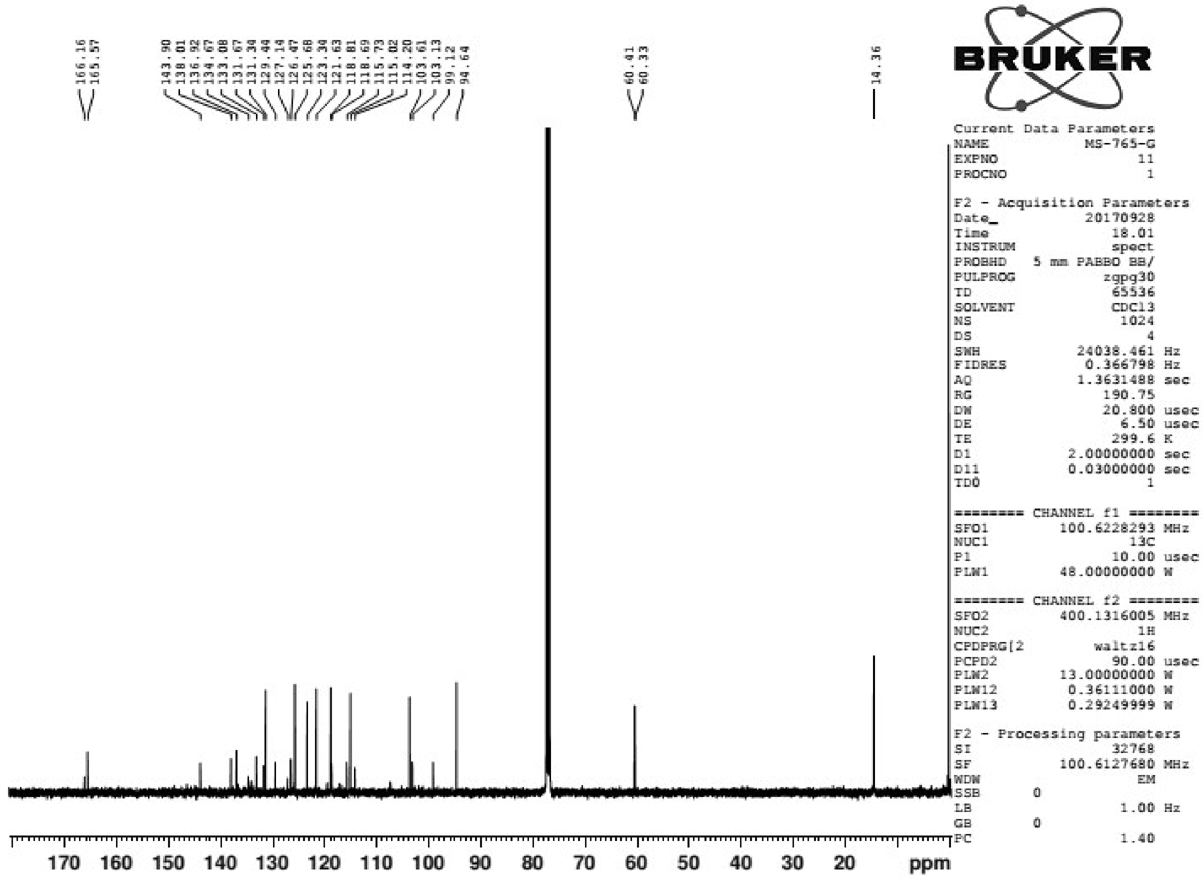


```
Current Data Parameters
NAME               MS-765-G
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170928
Time                  10.01
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                4.0894465 sec
RG                   190.75
DW                   62.400 usec
DE                     6.50 usec
TE                    297.1 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                     1H
P1                    15.00 usec
PLW1            13.00000000 W

F2 - Processing parameters
SI                    65536
SF              400.1300106 MHz
WDW                      EM
SSB      0
LB                     0.30 Hz
GB       0
PC                     1.00
```



```
Current Data Parameters
NAME               MS-765-G
EXPNO                    11
PROCNO                    1

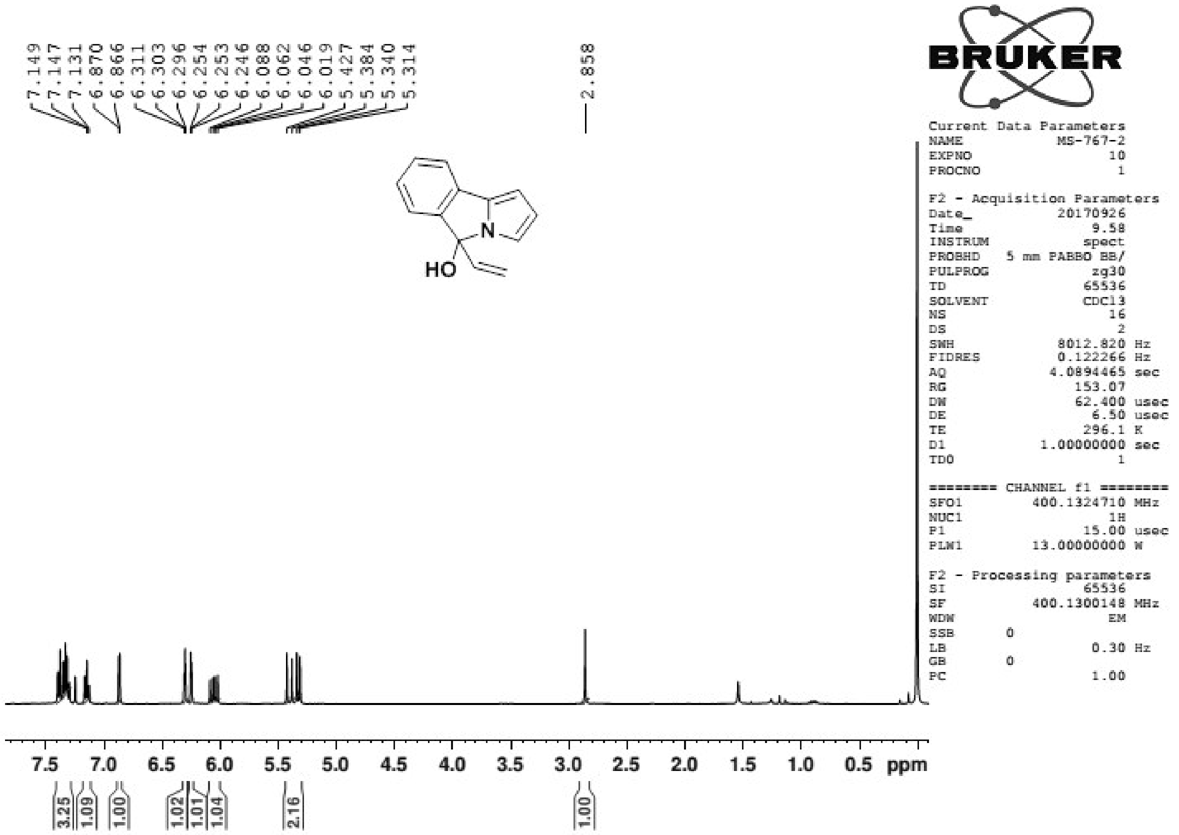
F2 - Acquisition Parameters
Date_              20170928
Time                  18.01
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                   190.75
DW                   20.800 usec
DE                     6.50 usec
TE                    299.6 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6228293 MHz
NUC1                    13C
P1                    10.00 usec
PLW1            48.00000000 W

======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                     1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2            13.00000000 W
PLW12            0.36111000 W
PLW13            0.29249999 W

F2 - Processing parameters
SI                    32768
SF              100.6127680 MHz
WDW                      EM
SSB      0
LB                     1.00 Hz
GB       0
PC                     1.40
```

Compound **18**

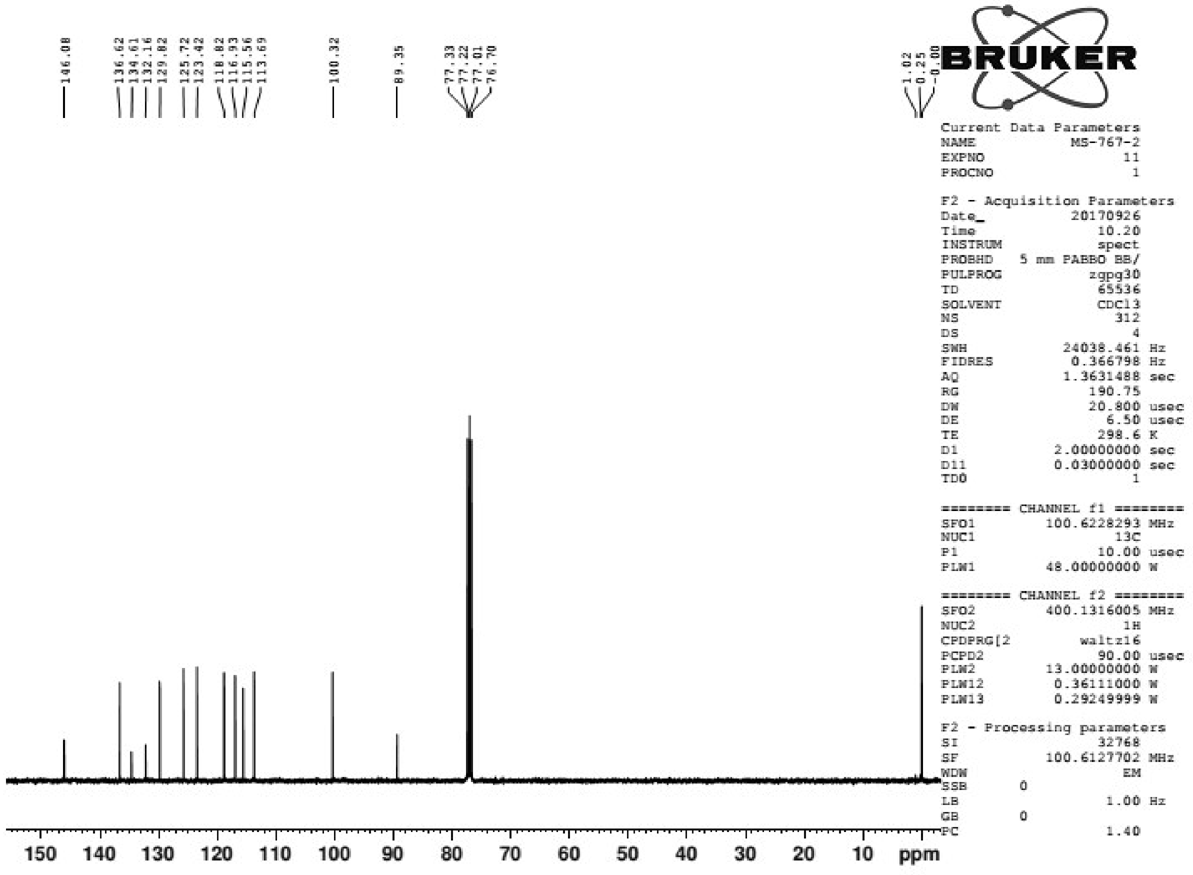


```
Current Data Parameters
NAME             MS-767-2
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170926
Time                 9.58
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 153.07
DW                 62.400 usec
DE                   6.50 usec
TE                  296.1 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300148 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



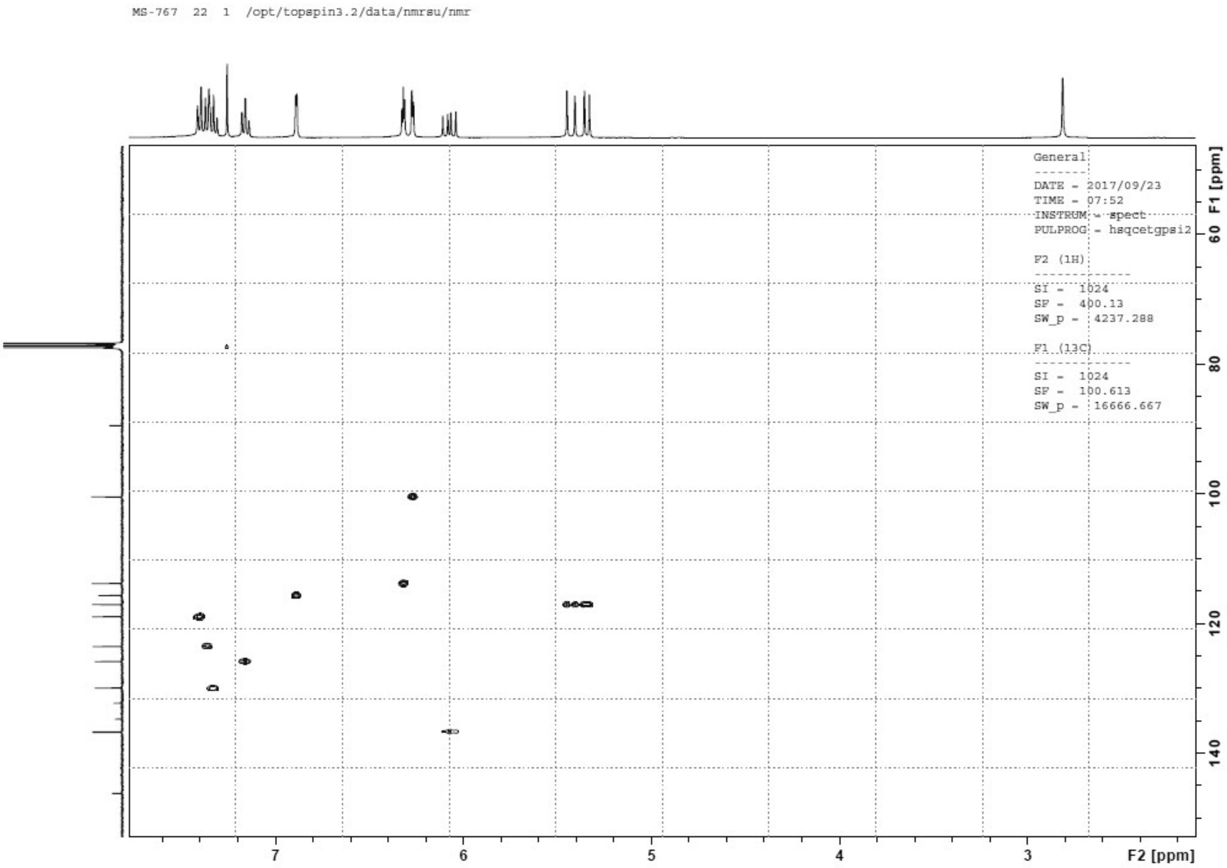
```
Current Data Parameters
NAME             MS-767-2
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170926
Time                10.20
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    312
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  298.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

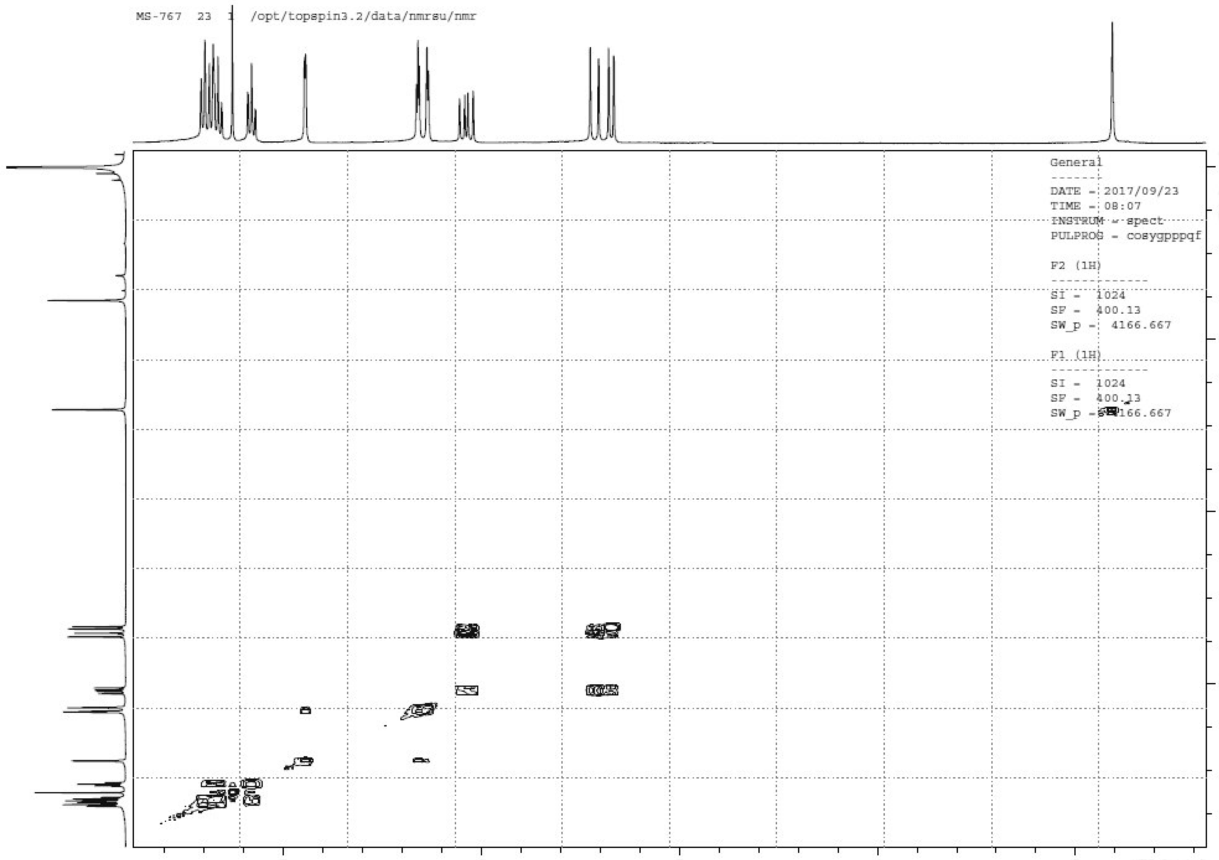
F2 - Processing parameters
SI                  32768
SF            100.6127702 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```



```
General
-------
DATE = 2017/09/23
TIME = 07:52
INSTRUM = spect
PULPROG = hsqcetgpsi2

F2 (1H)
-------------
SI =  1024
SF =  400.13
SW_p =  4237.288

F1 (13C)
-------------
SI =  1024
SF =  100.613
SW_p =  16666.667
```

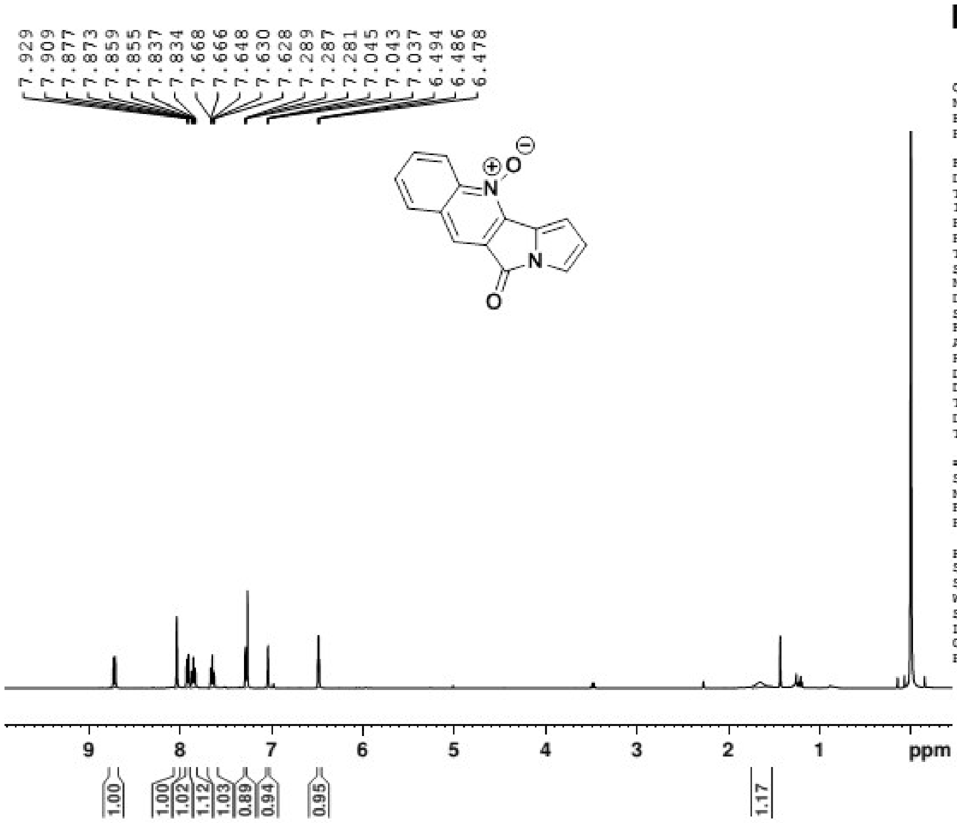


```
General
-------
DATE = 2017/09/23
TIME = 08:07
INSTRUM = spect
PULPROG = cosygpppqf

F2 (1H)
-------------
SI =  1024
SF =  400.13
SW_p =  4166.667

F1 (1H)
-------------
SI =  1024
SF =  400.13
SW_p =  4166.667
```

Compound **19**

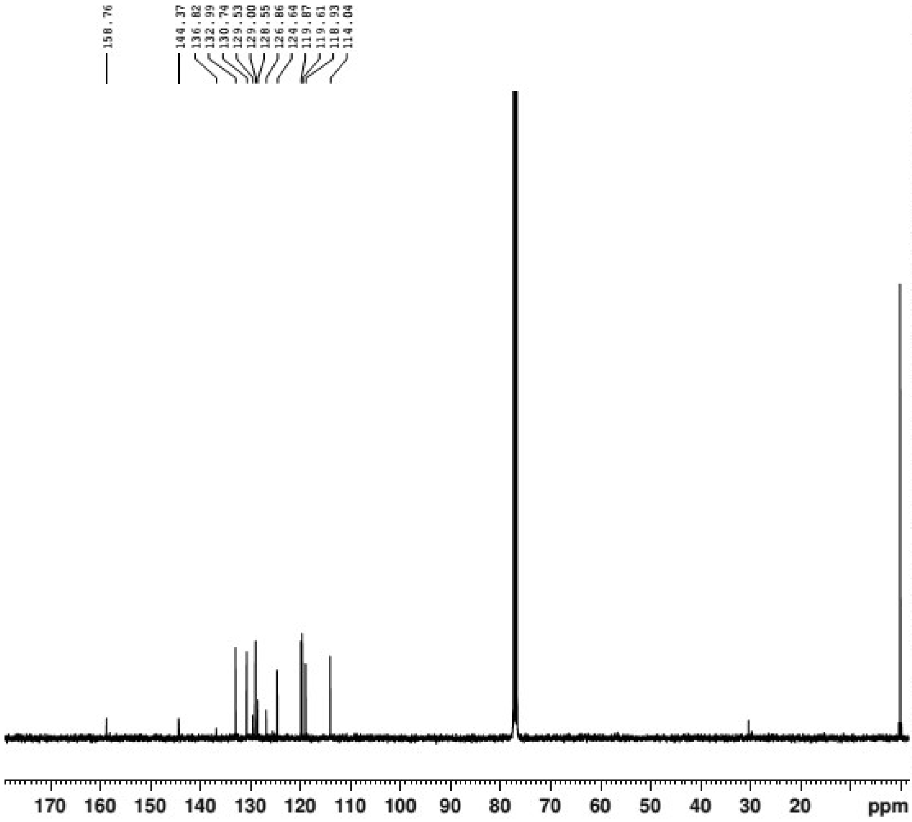


```
Current Data Parameters
NAME               MS-774
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171026
Time                12.15
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 190.75
DW                 62.400 usec
DE                   6.50 usec
TE                  298.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300085 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



```
Current Data Parameters
NAME               MS-774
EXPNO                  11
PROCNO                  1

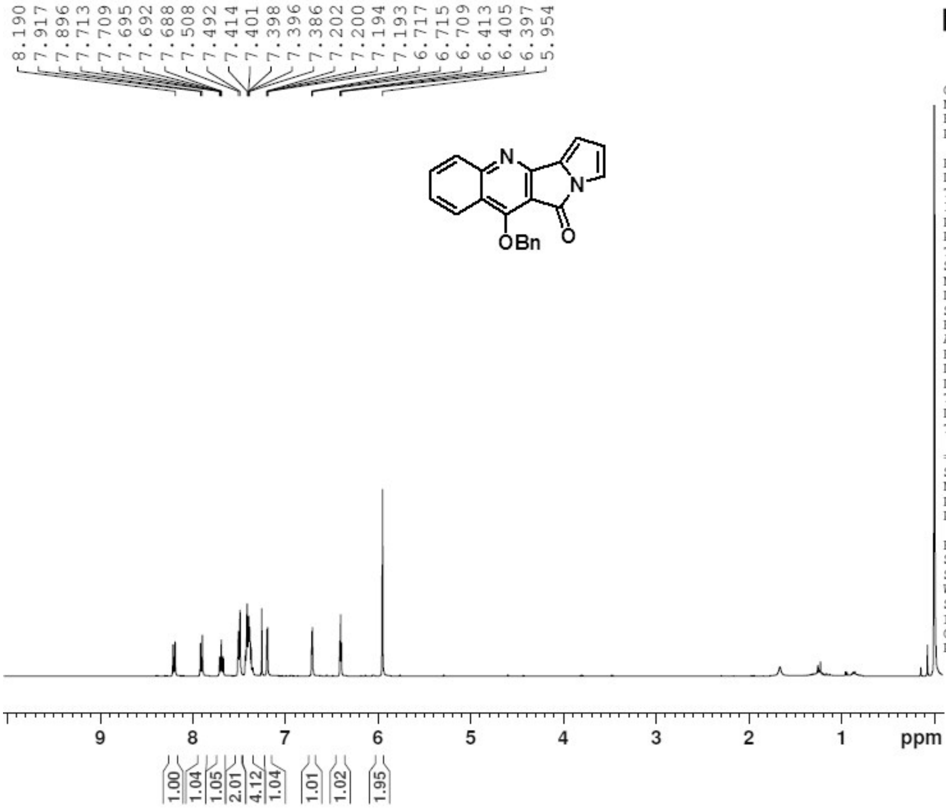
F2 - Acquisition Parameters
Date_            20171026
Time                14.18
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   2000
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  301.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

F2 - Processing parameters
SI                  32768
SF            100.6127669 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

Compound **25**

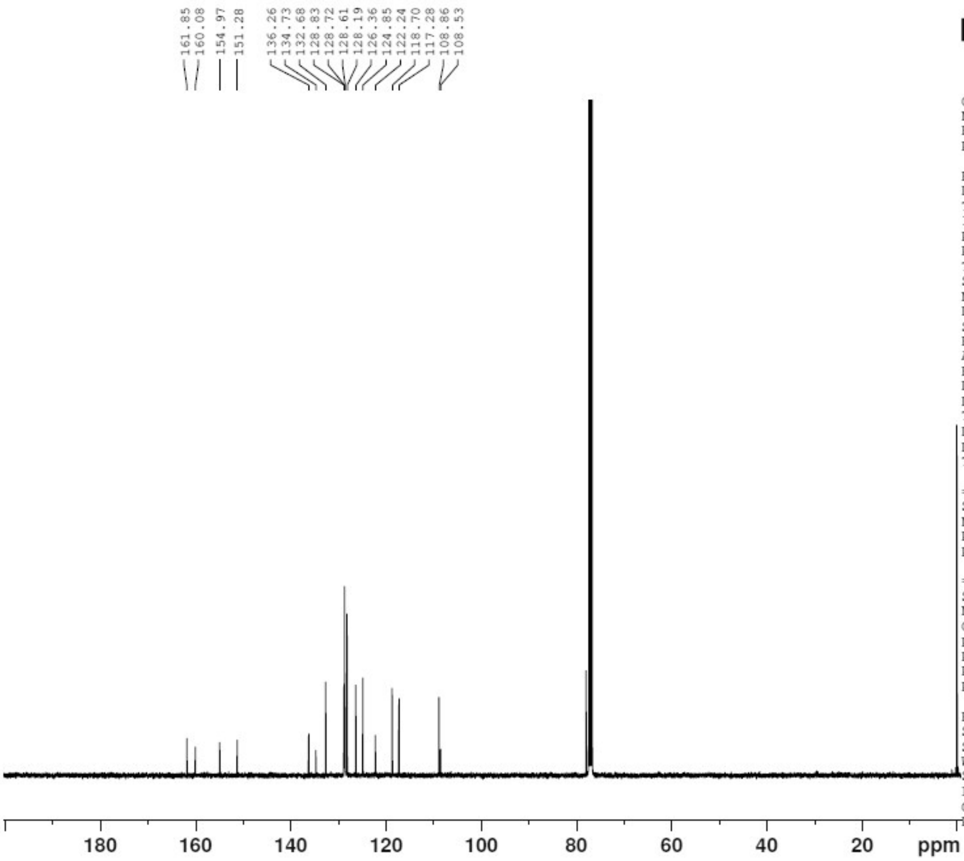


```
Current Data Parameters
NAME              TG-245
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171109
Time               11.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                190.75
DW                62.400 usec
DE                  6.50 usec
TE                 299.5 K
D1            1.00000000 sec
TD0                    1

======= CHANNEL f1 =======
SFO1         400.1324710 MHz
NUC1                  1H
P1                 15.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300111 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



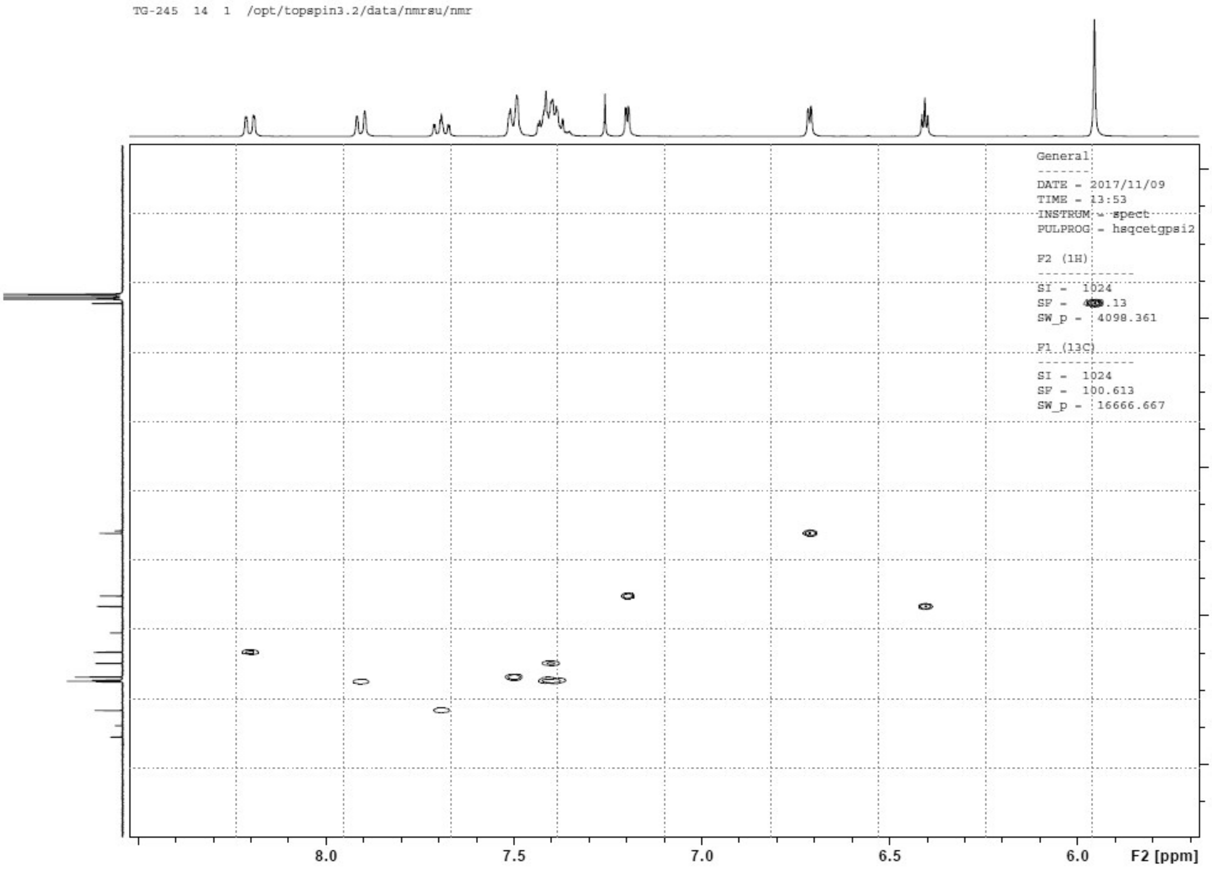
```
Current Data Parameters
NAME              TG-245
EXPNO                 11
PROCNO                 1

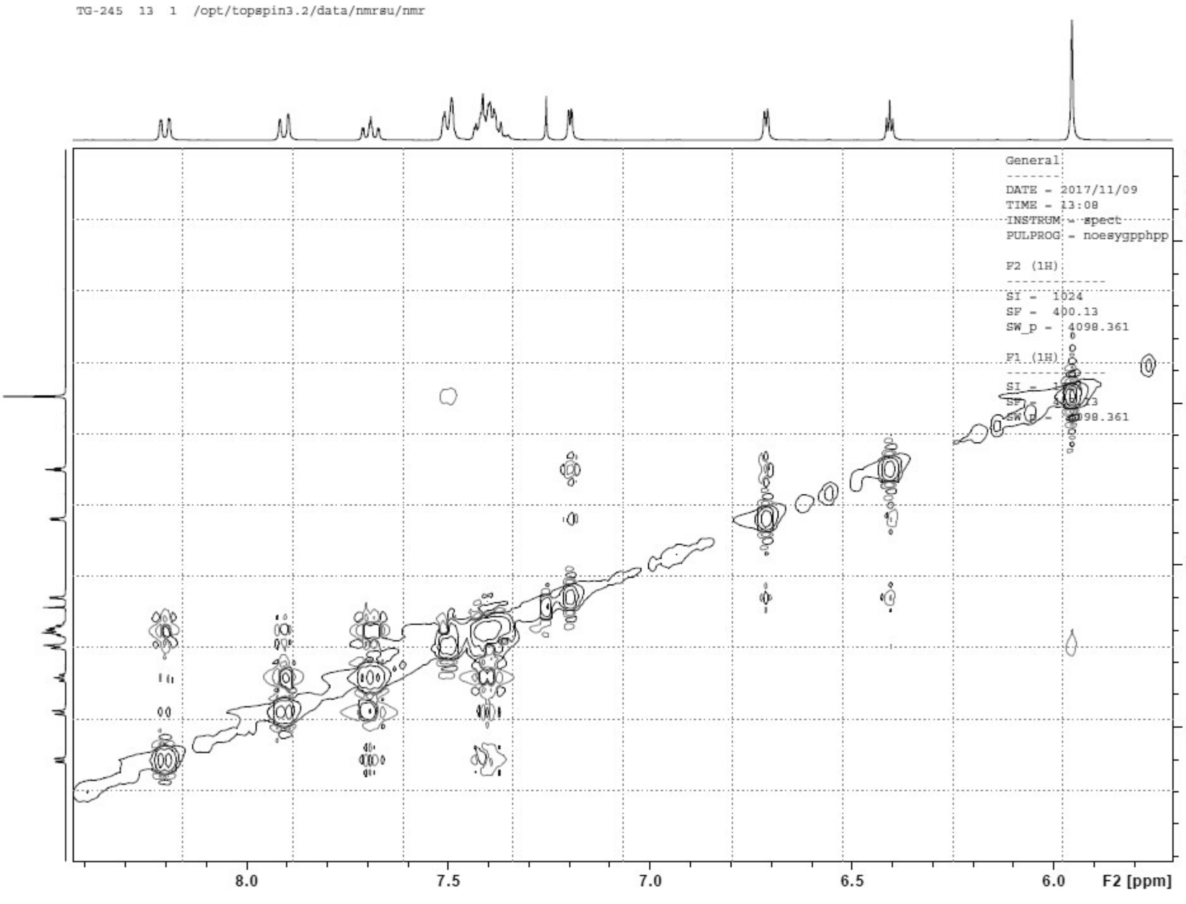
F2 - Acquisition Parameters
Date_           20171109
Time               13.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                190.75
DW                20.800 usec
DE                  6.50 usec
TE                 302.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

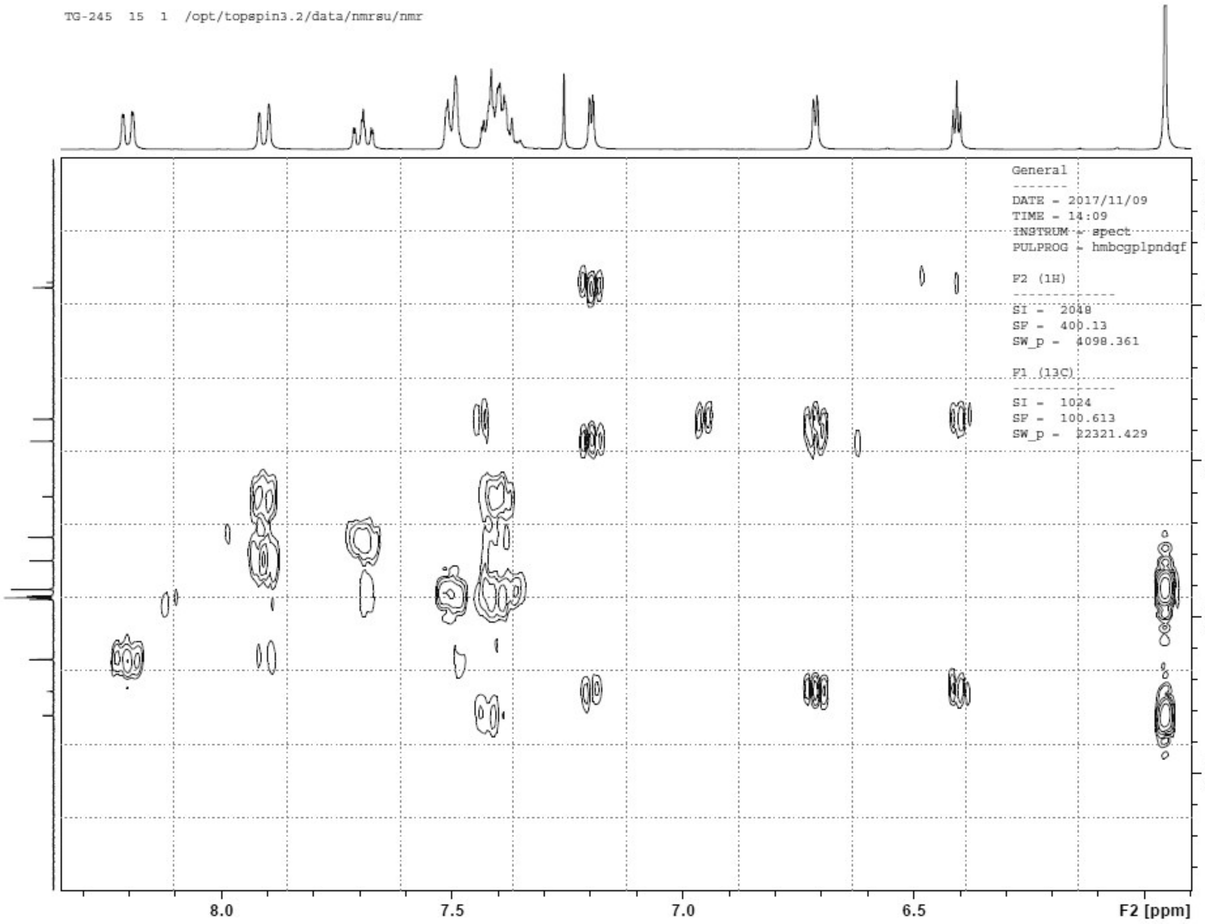
======= CHANNEL f1 =======
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         48.00000000 W

======= CHANNEL f2 =======
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.36111000 W
PLW13         0.29249999 W

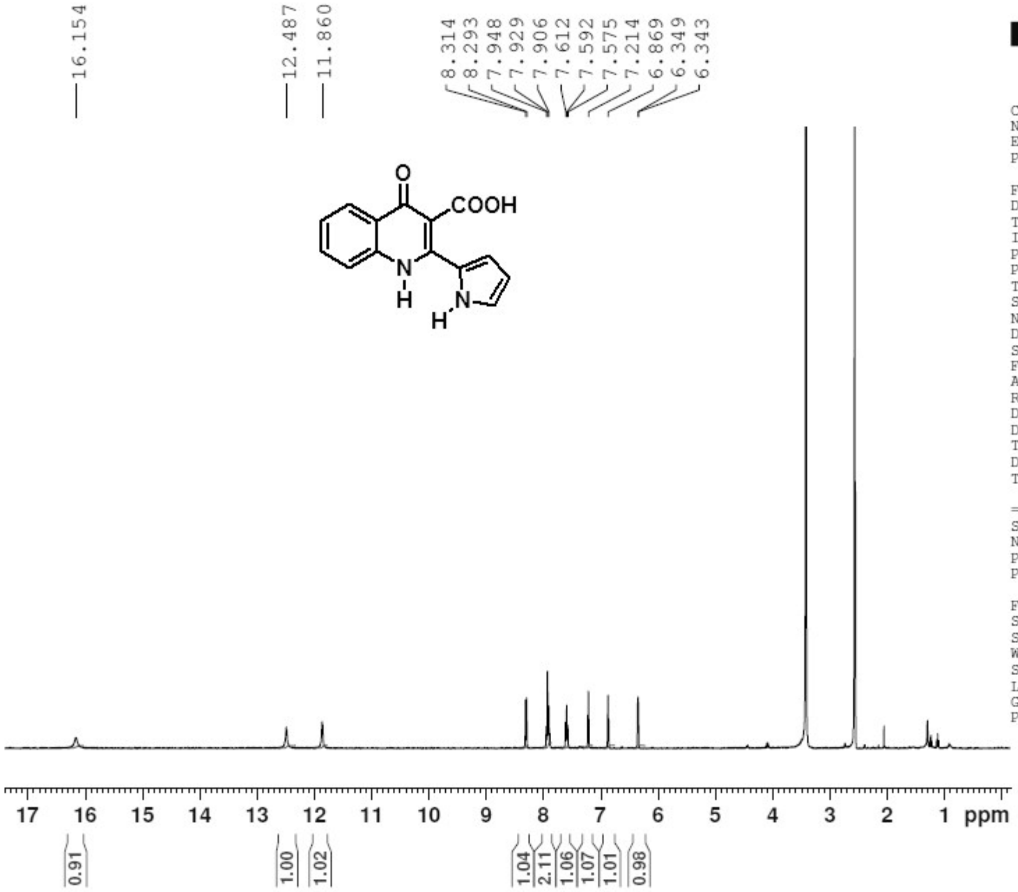
F2 - Processing parameters
SI                 32768
SF           100.6127674 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```


TG-245 14 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/11/09
TIME = 13:53
INSTRUM = spect
PULPROG = hsqcetgpsi2
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 4098.361
F1 (13C)
SI = 1024
SF = 100.613
SW_p = 16666.667
F1 [ppm]
80
100
120
140
8.0
7.5
7.0
6.5
6.0
F2 [ppm]


TG-245 13 1 /opt/topspin3.2/data/nmrsu/nmr
General
DATE = 2017/11/09
TIME = 13:08
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 4098.361
F1 (1H)
SW_p = 4098.361
F1 [ppm]
5
6
7
8
8.0
7.5
7.0
6.5
6.0
F2 [ppm]



Marinamide **26**

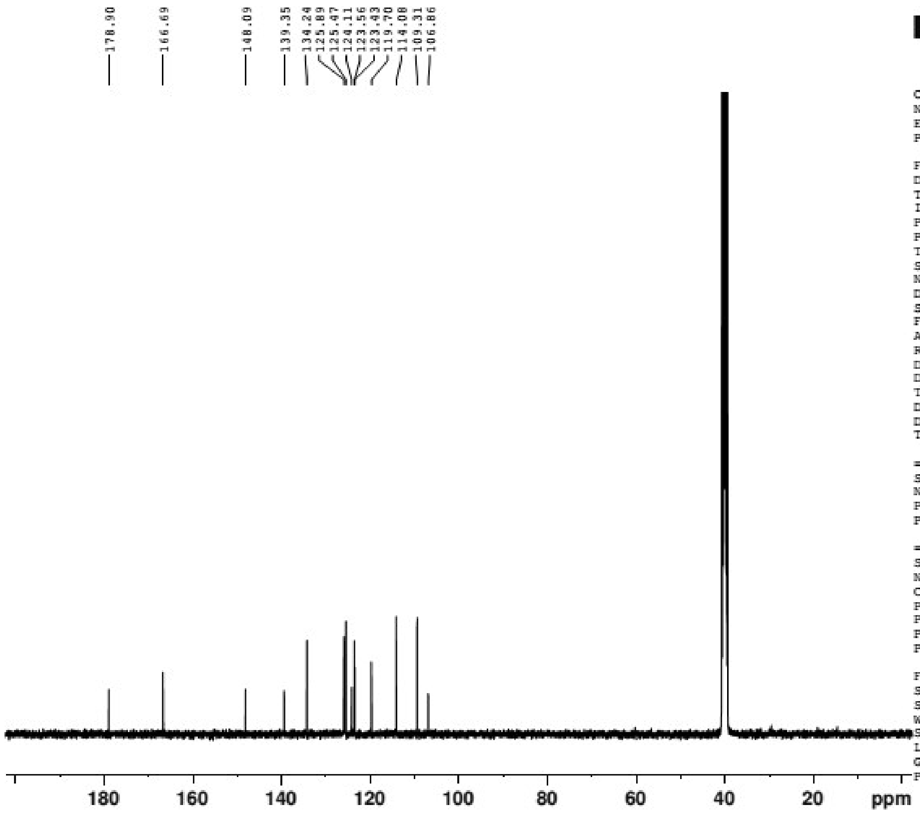


```
Current Data Parameters
NAME             TG-MA_2p
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171205
Time                10.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                      6
DS                      2
SWH              8802.817 Hz
FIDRES           0.134320 Hz
AQ              3.7224448 sec
RG                 190.75
DW                 56.800 usec
DE                   6.50 usec
TE                  292.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1336712 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1299773 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



```
Current Data Parameters
NAME                TG-MA
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171203
Time                 3.59
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                  15000
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631488 sec
RG                 190.75
DW                 20.800 usec
DE                   6.50 usec
TE                  295.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.36111000 W
PLW13          0.29249999 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```