Supporting Information

for

Aza-heterocyclic frameworks through intramolecular π-system trapping of spiro-*N*-acyliminiums generated from isoindolinone

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‡ **Dedicated to the memory of our colleague and friend Dr. Christian Garrault passed away in Le Havre on 22 February 2019.**

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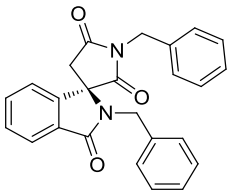
I. General remarks

The main text of the article should appear here with headings as appropriate. Starting materials are commercially available and were used without further purification (suppliers: Carlo Erba Reagents S.A.S., Thermo Fisher Scientific Inc., and Sigma-Aldrich Co.). Melting points were measured on a MPA 100 OptiMelt® apparatus and are uncorrected. Nuclear magnetic resonance (NMR) spectra were acquired at 300 MHz for ^{1}H NMR and at 75 MHz for ^{13}C NMR on a Bruker Advance III 300 MHz spectrometer with tetramethylsilane (TMS) as internal standard, at room temperature (rt). Chemical shifts (δ) are expressed in ppm relative to TMS. Splitting patterns are designed: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; sym m, symmetric multiplet; br s, broaden singlet; br t, broaden triplet. Coupling constants (J) are reported in Hertz (Hz). Thin layer chromatographies (TLC) were realized on Macherey Nagel silica gel plates with fluorescent indicator and were visualized under a UV-lamp at 254 nm and 365 nm. Column chromatographies were performed with a CombiFlash Rf Companion (Teledyne-Isco System) using RediSep packed columns. IR spectra were recorded on a Varian 640-IR FT-IR Spectrometer. Elemental analyses (C, H, N) of new compounds were determined on a Thermo Electron apparatus by 'Pôle Chimie Moléculaire-Welience', Faculté de Sciences Mirande, Université de Bourgogne, Dijon, France. LC-MS was accomplished using an HPLC combined with a Surveyor MSQ (Thermo Electron) equipped with APCI source.

II. General procedure for the preparation of spirosuccinimides (±)-1a-k

To a solution of *N*-alkylated isoindolin-1-one **7** (0.01 mol) in 100 mL of anhydrous CH_3CN were added potassium carbonate (0.015 mol, 2.07 g) and bromo acetamide (0.015 mol) under dry argon atmosphere. The mixture was then refluxed for 24 h. The solution was cooled at room temperature and filtered through a plug of celite. After removal of solvent under reduced pressure, the crude was analysed by ^{1}H NMR spectroscopy then purified by flash chromatography on silica gel column using a mixture of cyclohexane/AcOEt as eluent.

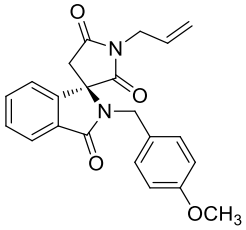
(±)-1',2-Dibenzylspiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1a



Spirosuccinimide (±)-1a: This product was isolated as a white solid in 70% yield, R_f (cyclohexane/AcOEt: 4/1) = 0.42; mp = 178-180 °C; **IR (ν_{max} / cm^{-1})**: 1706, 1687; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.76 (d, *J* = 18.6 Hz, 1H, CH_2), 2.82 (d, *J* = 18.6 Hz, 1H, CH_2), 4.08 (d, *J* = 15.6 Hz, 1H, CH_2), 4.65 (d, *J* = 13.8 Hz, 1H, CH_2), 4.70 (d, *J* = 13.8 Hz, 1H, CH_2), 5.20 (d, *J* = 15.6 Hz, 1H, CH_2), 6.98 (d, *J* = 6 Hz, 1H,H_{aro}), 7.06-7.08 (m, 2H, H_{aro}), 7.23-7.26 (m, 3H, H_{aro}), 7.35-7.40 (m, 5H, H_{aro}), 7.47-7.57 (m, 2H, H_{aro}), 7.92 (d, *J* = 6.9 Hz, 1H,H_{aro})ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.9 (CH_2), 43.3 (CH_2), 44.4 (CH_2), 68.5 (C^q), 119.8 (CH_{aro}), 124.6 (CH_{aro}), 128.1 (2 x CH_{aro}), 128.2 (CH_{aro}), 128.5 (CH_{aro}), 128.8 (2x CH_{aro}), 128.9 (2x CH_{aro}), 129.0 (2 x CH_{aro}), 129.9 (CH_{aro}), 130.7 (C^q_{aro}), 132.9 (CH_{aro}), 135.2 (C^q_{aro}), 135.8

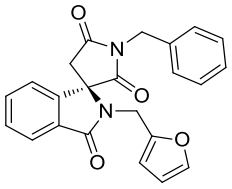
(C^q_{aro}), 144.1 (C^q_{aro}), 168.6 (C=O), 172.5 (C=O), 173.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{21}N_2O_3$ $[M+H]^+$: 397.1507, found 397.1571.

(±)-1'-Allyl-2-(4-methoxybenzyl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1b



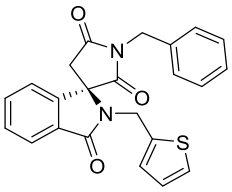
Spirosuccinimide (±)-1b: This compound was isolated as a white solid in 66% yield, R_f (cyclohexane/AcOEt: 4/1) = 0.42; mp = 157-159 °C ; **IR (ν_{max} / cm^{-1})**: 1712, 1684; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.88 (d, *J* = 18.0 Hz, 1H, CH_2), 2.96 (d, *J* = 18.0 Hz, 1H, CH_2), 3.78 (s, 3H, OCH_3), 4,02-4.12 (m, 2H, CH_2), 4.32 (d, *J* = 15.0 Hz, 1H, CH_2), 5.03 (d, *J* = 15.0 Hz, 1H, CH_2), 5.24-5.31 (m, 2H, CH_2=), 5.70-5.83 (m, 1H, CH=), 6.82 (d, *J* = 9.0 Hz, 2H, H_{aro}), 7.16 (d, *J* = 9.0 Hz, 3H, H_{aro}), 7.53-7.61 (m, 2H, H_{aro}), 7.93-7.96 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.87 (CH_2), 41.6 (CH_2), 43.8 (CH_2), 55.2 (OCH_3), 68.1 (C^q), 114.3 (2 x CH_{aro}), 119.7 (CH_2=), 119.7 (CH_{aro}), 124.6 (CH_{aro}), 127.7 (C^q_{aro}), 129.6 (2 x CH_{aro}), 129.8 (CH_{aro}), 129.9 (CH_{aro}), 130.9 (C^q_{aro}), 132.8 (CH_{aro}), 144.2 (C^q_{aro}), 159.5 (C^q_{aro}), 168.5 (C=O), 172.3 (C=O) 173.2 (C=O) ppm. HRMS (+ESI) calculated for $C_{22}H_{21}N_2O_4$ $[M+H]^+$: 377.1457, found 377.1501.

(±)-1'-Benzyl-2-(furan-2-ylmethyl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1c



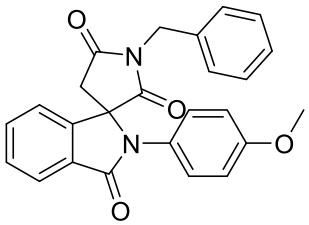
Spirosuccinimide (±)-1c: This product was isolated as a white solid in 60% yield, R_f (cyclohexane/AcOEt: 3/2) = 0.68; mp = 158-160 °C; **IR (ν_{max} / cm^{-1})**: 1691, 1662; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.81 (d, *J* = 18.0 Hz, 1H, CH_2), 2.91 (d, *J* = 18.0 Hz, 1H, CH_2), 4.31 (d, *J* = 15.0 Hz, 1H, CH_2), 4.75 (s, 2H, CH_2), 5.06 (d, *J* = 15.0 Hz, 1H, CH_2), 6.03 (d, *J* = 3.0 Hz, 1H, H_{aro}), 6.22 (t, *J* = 3.0 Hz, 1H, H_{aro}), 6.98 (d, *J* = 9.0 Hz, 1H, H_{aro}), 7.18 (s, 1H, H_{aro}), 7.34-7.54 (m, 7H, H_{aro}), 7.88-7.91 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 36.8 (CH_2), 37.7 (CH_2), 43.3 (CH_2), 68.0 (C^q), 110.2 (CH_{aro}), 111.0 (CH_{aro}), 119.7 (CH_{aro}), 124.5 (CH_{aro}), 128.4 (CH_{aro}), 128.8 (2 x CH_{aro}), 129.1 (2 x CH_{aro}), 129.8 (CH_{aro}), 130.5 (C^q_{aro}), 132.9 (CH_{aro}), 135.2 (C^q_{aro}), 142.7 (CH_{aro}), 144.3 (C^q_{aro}), 148.4(C^q_{aro}), 168.3 (C=O), 172.7 (C=O), 173.2 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{19}N_2O_4$ $[M+H]^+$: 387.1300, found 387.1344.

(±)-1'-Benzyl-2-(furan-2-ylmethyl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1d



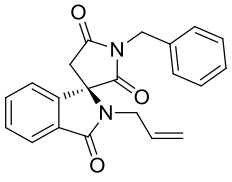
Spirosuccinimide (±)-1d: This product was isolated as a white solid in 77% yield, R_f (cyclohexane/AcOEt: 3/2) = 0.54; mp = 192-194 °C; **IR (ν_{max} / cm^{-1})**: 1688.24 cm^{-1}; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.85 (d, *J* = 18.0 Hz, 1H, CH_2), 2.92 (d, *J* = 18.0 Hz, 1H, CH_2), 4.35 (d, *J* = 18.0 Hz, 1H, CH_2), 4.73 (s, 2H, CH_2), 5.32 (d, *J* = 18.0 Hz, 1H, CH_2), 6.60 (d, *J* = 3.0 Hz, 1H, H_{aro}), 6.80-6.83 (m, 1H, H_{aro}), 6.97 (d, *J* = 9.0 Hz, 1H, H_{aro}), 7.22 (d, *J* = 6.0 Hz, 1H, H_{aro}), 7.35-7.43 (m, 5H, H_{aro}), 7.47-7.56 (m, 2H, H_{aro}), 7.91 (d, *J* = 6.0 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.9 (CH_2), 39.0 (CH_2), 43.3 (CH_2), 68.4 (C^q), 119.8 (CH_{aro}), 124.6 (CH_{aro}), 126.7(CH_{aro}), 127.0 (CH_{aro}), 127.4 (CH_{aro}), 128.5 (CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.9 (CH_{aro}), 130.0 (C^q_{aro}), 133.0 (CH_{aro}), 135.2 (C^q_{aro}), 138.4 (C^q_{aro}), 144.1 (C^q_{aro}), 168.2 (C=O), 172.6 (C=O), 173.3 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{19}N_2O_4S$ $[M+H]^+$: 403.1072, found 403.1122.

(±)-1'-Benzyl-2-(4-methoxyphenyl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1e



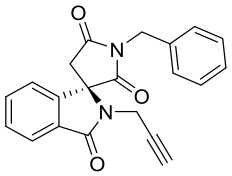
Spirosuccinimide (±)-1e: This product was isolated as a white solid in 43% yield, R_f(cyclohexane/AcOEt: 4/1) = 0.42; mp = 158-160°C; **IR (ν_{max} / cm^{-1})**: 1788, 11699; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 3.08 (d, *J* = 18.4 Hz, 1H, CH_2), 3.18 (d, *J* = 18.4 Hz, 1H, CH_2), 3.80 (s, 1H, CH_3) 4.70 (d, *J* = 13.9 Hz, 1H, CH_2), 4.77 (d, *J* = 13.9 Hz, 1H, CH_2), 6.81 (d, *J* = 8.8 Hz, 2H, H_{aro}), 7.06-7.11 (m, 3H, H_{aro}), 7.27-7.33 (m, 5H, H_{aro}), 7.53-7.60 (m, 2H, H_{aro}), 7.95-7.98 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.5 (CH_2), 43.3 (CH_2), 55.4 (CH_3), 69.9 (C^q), 115.1 (2 x CH_{aro}), 120.1 (CH_{aro}), 124.9 (CH_{aro}), 126.7 (C^q_{aro}), 128.3 (CH_{aro}), 128.7 (2 x CH_{aro}), 128.8 (2 x CH_{aro}), 129.4 (2 x CH_{aro}), 130.0 (CH_{aro}), 131.2 (C^q_{aro}), 133.0 (CH_{aro}), 134.8 (C^q_{aro}), 143.7 (C^q_{aro}), 159.5 (C^q_{aro}), 168.0 (C=O), 172.5 (C=O), 174.1 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{21}N_2O_4$ $[M+H]^+$: 413.1457, found 413.1462.

(±)-2-Allyl-1'-benzylspiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1f



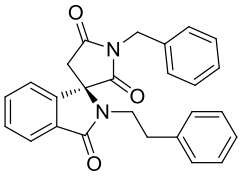
Spirosuccinimide (±)-1f: This product was isolated as a white solid in 95% yield, R_f (cyclohexane/AcOEt: 3/2) = 0.53; mp = 88-90 °C; **IR (ν_{max} / cm^{-1})**: 1748, 1689; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.98 (d, *J* = 18.5 Hz, 1H, CH_2), 3.30 (d, *J* = 18.5 Hz, 1H, CH_2), 4.04 (dd, *J* = 6.2 and 15.7 Hz, 1H, CH_2=), 4.18 (dd, *J* = 6.2 and 15.7 Hz, 1H, CH_2=), 4.72 (d, *J* = 13.8 Hz, 1H, CH_2), 4.78 (d, *J* = 13.8 Hz, 1H, CH_2), 4.89-4.99 (m, 2H, CH_2), 5.69-5.82 (m, 1H, CH=), 7.01-7.04 (m, 1H, H_{aro}), 7.32-7.33 (m, 3H, H_{aro}), 7.39-7.42 (m, 2H, H_{aro}), 7.47-7.54 (m, 2H, H_{aro}), 7.86-788 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.4 (CH_2), 43.3 (CH_2), 43.4 (CH_2), 68.1 (C^q), 119.0 (CH_{aro}), 119.7 (CH_{aro}), 124.4 (CH_{aro}), 128.4 (CH_{aro}), 128.8 (2 x CH_{aro}), 129.1 (2 x CH_{aro}), 129.8 (CH_{aro}), 130.8 (C^q_{aro}), 132.8 (CH_{aro}), 132.9 (CH_{aro}), 135.0 (C^q_{aro}), 144.0 (C^q_{aro}), 168.1 (C=O), 172.5 (C=O), 174.0 (C=O) ppm. HRMS (+ESI) calculated for $C_{21}H_{19}N_2O_3$ $[M+H]^+$: 347.1351, found 347.1395.

(±)-1'-Benzyl-2-(prop-2-yn-1-yl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1g



Spirosuccinimide (±)-1g: This product was isolated as yellow solid in 85% yield, R_f(cyclohexane/AcOEt: 4/1) = 0.51; mp = 135-137 °C; **IR (ν_{max} / cm^{-1})**: 1680, 1674, 1668; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.86 (t, *J* = 2.6 Hz, 1H, CH), 3.07 (d, *J* = 18.5 Hz, 1H, CH_2), 3.74 (d, *J* = 18.5 Hz, 1H, CH_2), 4.15 (dd, *J* = 2.6 and 18.2 Hz, 1H, CH_2), 4.55 (dd, *J* = 2.6 and 18.2 Hz, 1H, CH_2), 4.75 (d, *J* = 13.8 Hz, 1H, CH_2), 4.85 (d, *J* = 13.8 Hz, 1H, CH_2), 7.03-7.06 (m, 1H, H_{aro}), 7.31-7.34 (m, 3H, H_{aro}), 7.41-7.44 (m, 2H, H_{aro}), 7.50-7.53 (m, 2H, H_{aro}), 7.85-7.88 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 29.9 (CH_2), 37.9 (CH_2), 43.4 (CH_2), 68.1 (C^q), 74.6 (CH), 74.6 (C^q), 119.7 (CH_{aro}), 124.6 (CH_{aro}), 128.4 (CH_{aro}), 128.8 (2 x CH_{aro}), 129.2 (2 x CH_{aro}), 129.9 (CH_{aro}), 130.1 (C^q_{aro}), 133.1 (CH_{aro}), 134.9 (C^q_{aro}), 144.3 (C^q_{aro}), 167.8 (C=O), 172.7 (C=O), 173.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{21}H_{16}N_2O_3Na$ $[M+Na]^+$: 367.1053, found 367.1054.

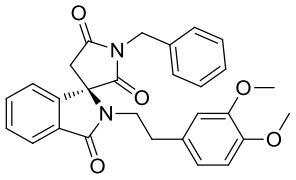
(±)-1'-Benzyl-2-phenethylspiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1h



Spirosuccinimide (±)-1h: This product was isolated as a white solid in 75% yield, R_f (cyclohexane/AcOEt: 3/2) = 0.60; mp = 135-137 °C; **IR (ν_{max} / cm^{-1})**: 1748, 1697; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.51 (d, *J* = 18.0 Hz, 1H, CH_2), 2.69-2.78 (m, 2H, CH_2 + CH_2), 3.04-3.14 (m, 1H, CH_2), 3.20-3.30 (m, 1H, CH_2), 3.59-3.68 (m, 1H, CH_2), 4.54-4.82 (m, 2H, H_{aro}), 6.87 (d, *J* = 7.2 Hz, 1H, H_{aro}), 6.98 (d, *J* = 6.1 Hz, 2H, H_{aro}), 7.13-7.47 (m, 10H, H_{aro}), 7.80 (d, *J* = 7.2 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 34.2 (CH_2), 37.3 (CH_2), 43.4 (CH_2), 43.7 (CH_2), 68.7(C^q), 119.8 (CH_{aro}), 124.2 (CH_{aro}), 126.8 (CH_{aro}), 128.4 (CH_{aro}), 128.7 (2x

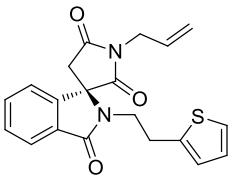
CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.8 (CH_{aro}), 131.2 (C^q_{aro}), 132.7 (CH_{aro}), 138.7 (C^q_{aro}), 143.8 (C^q_{aro}), 168.8 (C=O), 172.6 (C=O), 173.8 (C=O) ppm. HRMS (+ESI) calculated for $C_{26}H_{23}N_2O_3$ $[M+H]^+$: 411.1664, found 411.1696.

(±)-1'-Benzyl-2-(3,4-dimethoxyphenethyl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1i



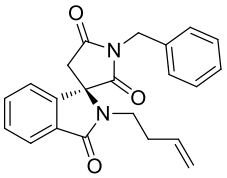
Spirosuccinimide (±)-1i: This product was isolated as a white solid in 76% yield, R_f (cyclohexane/AcOEt: 1/1) = 0.50; mp = 139-141 °C; **IR (ν_{max} / cm^{-1})**: 1699, 1682; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.70 (d, *J* = 18.6 Hz, 1H, CH_2), 2.75-2.81 (m, 1H, CH_2), 2.85 (d, *J* = 18.6 Hz, 1H, CH_2), 3.07-3.17 (m, 1H, CH_2), 3.27-3.37 (m, 1H, CH_2), 3.68-3.76 (m, 1H, CH_2), 3.78 (s, 3H, OCH_3), 3.86 (s, 3H, OCH_3), 4.78 (s, 2H, CH_2), 6.59-6.66 (m, 2H, H_{aro}), 6.76 (d, *J* = 8.1 Hz, 1H, H_{aro}), 6.96 (d, *J* = 7.4 Hz, 1H, H_{aro}), 7.32-7.42 (m, 5H, H_{aro}), 7.45-7.56 (m, 2H, H_{aro}), 7.88-7.91 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 33.7 (CH_2), 37.3 (CH_2), 43.4 (CH_2), 43.6 (CH_2), 55.8 (OCH_3), 55.9 (OCH_3), 68.7 (C^q), 111.4 (CH_{aro}), 112.1 (CH_{aro}), 119.8 (CH_{aro}), 120.6 (CH_{aro}), 124.1 (CH_{aro}), 128.4 (CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.8 (CH_{aro}), 131.1 (C^q_{aro}), 131.2 (C^q_{aro}), 132.7 (CH_{aro}), 135.2 (C^q_{aro}), 143.8 (C^q_{aro}), 147.8 (C^q_{aro}), 149.0 (C^q_{aro}), 168.8 (C=O), 172.6 (C=O), 168.8 (C=O) ppm. HRMS (+ESI) calculated for $C_{28}H_{27}N_2O_5$ $[M+H]^+$: 471.1875, found 471.1920.

(±)-1'-Allyl-2-(2-(thiophen-2-yl)ethyl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1j



Spirosuccinimide (±)-1j: This product was isolated as an oil with a yield of 86%, R_f (cyclohexane/AcOEt: 3/2) = 0.56; **IR (ν_{max} / cm^{-1})**: 1682, 1713; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.57 (d, *J* = 18.0 Hz, 1H, CH_2), 2.84 (d, *J* = 18.0 Hz, 1H, CH_2), 3.08-3.17 (m, 1H, CH_2), 3.28-3.38 (m, 1H, CH_2), 3.46-3.56 (m, 1H, CH_2), 3.83-3.91 (m, 1H, CH_2), 4.20-4.22 (m, 2H, CH_2), 5.25-5.33 (m, 2H, CH_2=), 5.76-5.87 (m, 1H, CH=), 6.81-6.83 (m, 1H, H_{aro}), 6.90-6.93 (m, 1H, H_{aro}), 7.13-7.19 (m, 2H, H_{aro}), 7.54-7.58 (m, 2H, H_{aro}), 7.89-7.92 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 28.2 (CH_2), 37.2 (CH_2), 41.7 (CH_2), 44.1 (CH_2), 69.0 (C^q), 119.8 (CH_{aro}), 119.9 (CH_{aro}), 124.3 (CH_{aro}), 125.9 (CH_{aro}), 127.3 (CH_{aro}), 129.8 (CH_{aro}), 129.9 (CH_{aro}), 131.1 (C^q_{aro}), 132.8 (CH_{aro}), 140.8 (C^q_{aro}), 143.8 (C^q_{aro}), 168.9 (C=O), 172.5 (C=O), 173.5 (C=O) ppm. HRMS (+ESI) calculated for $C_{17}H_{19}N_2O_3S$ $[M+H]^+$: 367.1072, found 367.1110.

(±)-1'-Benzyl-2-(but-3-en-1-yl)spiro[isoindoline-1,3'-pyrrolidine]-2',3,5'-trione (±)-1k

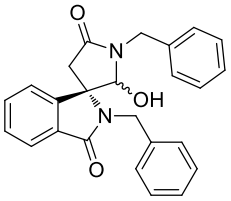


Spirosuccinimide (±)-1k: This product was isolated as an oil with a yield of 91%, R_f (cyclohexane/AcOEt: 3/2) = 0.65; **IR (ν_{max} / cm^{-1})**: 1699, 1688; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.19-2.31 (m, 1H, CH_2), 2.37-2.47 (m, 1H, CH_2), 3.07 (d, *J* = 18.6 Hz, 1H, CH_2), 3.24 (d, *J* = 18.6 Hz, 1H, CH_2), 3.31-3.47 (m, 2H, CH_2), 4.74-4.84 (m, 2H, CH_2), 4.97-5.04 (m, 2H, CH_2=), 5.63-5.77 (m, 1H, CH=), 6.97-7.00 (m, 1H, H_{aro}), 7.29-7.35 (m, 3H, H_{aro}), 7.37-7.41 (m, 2H, H_{aro}), 7.45-7.54 (m, 2H, H_{aro}), 7.84-7.87 (m, 2H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 32.4 (CH_2), 37.7 (CH_2), 40.7 (CH_2), 43.4 (CH_2), 68.4 (C^q), 117.4 (CH_{aro}), 119.8 (CH_{aro}), 124.3 (CH_{aro}), 128.5 (CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.8 (CH_{aro}), 131.2 (C^q_{aro}), 132.7 (CH_{aro}), 134.5 (CH_{aro}), 135.1 (C^q_{aro}),143.8 (C^q_{aro}), 168.5 (C=O), 172.5 (C=O), 173.9 (C=O) ppm. HRMS (+ESI) calculated for $C_{17}H_{19}N_2O_3S$ $[M+H]^+$: 361.1507, found 361.1566.

III. General Procedure for the Preparation of Spiro-hydroxylactams (±)-11(A,B)

To a solution of spirosuccinimide **(±)-1** (2.5 mmol) in a mixture of THF/EtOH (1:1) was added sodium borohydride (10 mmol, 4 equiv.) at 0 °C then the mixture was left under stirring at room temperature for 4 h. The reaction mixture was then cooled to 0 °C and quenched by addition of 1 mL of water then acidified to pH 3 by 2M HCl solution. The mixture was extracted twice by 30 mL of dichloromethane and the organic layer was dried over $MgSO_4$ then evaporated to dryness. The oily residue was purified by flash chromatography on a silica gel column using a mixture of cyclohexane and AcOEt as the eluent.

***N*,*N*'-Dibenzyl-2'-hydroxyspiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11a(A,B)**

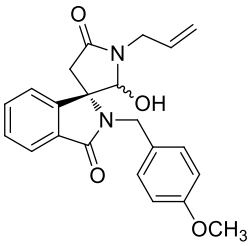


This product was obtained as a mixture of partially separable two diastereoisomers in 81% yield.

Major diastereoisomer (±)-11aA: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 2/3) = 0.30; mp = 226-228 °C; **IR (ν_{max} / cm^{-1}):** 3185, 1699, 1659**; ^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.61 (d, *J* = 17.4 Hz, 1H, CH_2), 2.92 (d, *J* = 17.4 Hz, 1H, CH_2), 3.49-3.51 (m, 1H, OH), 3.87 (d, *J* = 14.3 Hz, 1H, CH_2), 4.68 (d, *J* = 16.0 Hz, 1H, CH_2), 4.92-4.94 (m, 1H, CH_2), 4.97 (d, *J* = 14.3 Hz, 1H, CH_2), 5.07 (d, *J* = 16.0 Hz, 1H, CH_2), 7.09 (d, *J* = 7.3 Hz, 1H, H_{aro}), 7.27-7.64 (m, 12H, H_{aro}), 7.67 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 38.0 (CH_2), 43.6 (CH_2), 44.9 (CH_2), 68.1 (C^q), 86.7 (CH), 120.5 (CH_{aro}), 123.8 (CH_{aro}), 127.5 (2 x CH_{aro}), 127.9 (CH_{aro}), 128.0 (CH_{aro}), 128.8 (2 x CH_{aro}), 129.0 (2 x

CH_{aro}), 129.1 (2 x CH_{aro}), 129.2 (CH_{aro}), 130.1 (C^q_{aro}), 132.6 (CH_{aro}), 135.4 (C^q_{aro}), 137.9 (C^q_{aro}), 146.2 (C^q_{aro}), 169.5 (C=O), 170.1 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{23}N_2O_3$ $[M+H]^+$: 399.1664, found 399.1711.

***N*-Allyl-2'-hydroxy-2-(4-methoxybenzyl)spiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11b(A,B)**

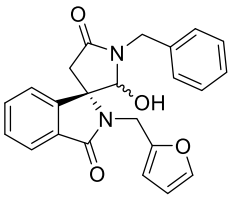


This product was obtained as a mixture of inseparable two diastereoisomers in 100% yield.

Major diastereoisomer (±)-11bA: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.27; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.49 (d, *J* = 17.4 Hz, 1H, CH_2), 2.92 (d, *J* = 17.4 Hz, 1H, CH_2), 3.62 (dd, *J* = 7.8 and 14.9 Hz, 1H, CH_2), 3.74 (s, 3H, OCH_3), 3.79-3.97 (m, 1H, CH_2), 4.23-4.35 (m, 1H, CH_2), 4.52-4.63 (m, 1H, CH_2), 4.76-4.91 (m, 1H, CH), 5.10-5.29 (m, 2H, CH_2=), 5.71-5.85 (m, 1H, CH=), 6.77-6.82 (m, 2H, H_{aro}), 7.10-7.17 (m, 2H, H_{aro}), 7.43 (t, *J* = 7.2 Hz, 1H, H_{aro}), 7.55 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.71 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.4 (CH_2), 42.5 (CH_2), 44.5 (CH_2), 55.2 (OCH_3), 67.8 (C^q), 87.4 (CH), 114.3 (2 x CH_{aro}), 119.5 (CH_2=), 120.4 (CH_{aro}),123.7 (CH_{aro}), 128.7 (2 x CH_{aro}), 129.2 (CH_{aro}), 129.7 (C^q_{aro}), 130.1 (C^q_{aro}), 131.3 (CH_{aro}), 132.9 (CH=), 147.1 (C^q_{aro}),159.0 (C^q_{aro}), 169.5 (C=O), 170.6 (C=O) ppm.

Minor diastereoisomer (±)-11bB: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.27; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.47 (d, *J* = 17.7 Hz, 1H, CH_2), 2.94 (d, *J* = 17.7 Hz, 1H, CH_2), 3.75 (s, 3H, OCH_3), 3.79-3.97 (m, 1H, CH_2), 4.54 (d, *J* = 15.6 Hz, 1H, CH_2), 4.60 (d, *J* = 15.6 Hz, 1H, CH_2), 4.76-4.91 (m, 2H, CH_2), 4.98 (d, *J* = 5.6 Hz, 1H, CH), 5.10-5.29 (m, 2H, CH_2=), 5.57-5.68 (m, 1H, CH=), 6.77-6.82 (m, 2H, H_{aro}), 7.10-7.17 (m, 2H, H_{aro}), 7.46 (t, *J* = 7.2 Hz, 1H, H_{aro}), 7.59 (d, *J* = 9 Hz, 1H, H_{aro}), 7.75 (d, *J* = 9 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 39.2 (CH_2), 42.8 (CH_2), 43.4 (CH_2), 55.2 (OCH_3), 69.1 (C^q), 86.6 (CH), 114.1 (2 x CH_{aro}), 118.9 (CH_2=), 123.5 (CH_{aro}),124.4 (CH_{aro}), 128.7 (2 x CH_{aro}), 129.1 (CH_{aro}), 129.3 (C^q_{aro}), 131.0 (C^q_{aro}), 131.9 (CH_{aro}), 132.0 (CH=), 143.9 (C^q_{aro}),159.1 (C^q_{aro}), 168.5 (C=O), 171.6 (C=O) ppm. HRMS (+ESI) calculated for $C_{22}H_{23}N_2O_4$ $[M+H]^+$: 379.1613, found 379.1670.

(±)-*N*-Benzyl-2-(furan-2-ylmethyl)-2'-hydroxyspiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11c(A,B)

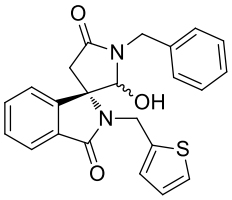


This product was obtained as a mixture of inseparable two diastereoisomers in 90% yield.

Major diastereoisomer (±)-11cA: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.27; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.52 (d, *J* = 17.4 Hz, 1H, CH_2), 3.19 (d, *J* = 17.4 Hz, 1H, CH_2), 4.14-4.29 (m, 2H, CH_2), 4.83 (s, 1H, CH), 4.90-5.06 (m, 2H, CH_2), 5.13 (s, 1H, OH), 6.12-6.28 (m, 2H, H_{aro}), 6.99 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.22 (d, *J* = 6.9 Hz, 2H, H_{aro}), 7.31-7.59 (m, 7H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.0 (CH_2), 38.4 (CH_2), 43.9 (CH_2), 67.0 (C^q), 87.0 (CH), 108.7 (CH_{aro}), 110.6 (CH_{aro}), 120.3 (CH_{aro}),123.3 (CH_{aro}), 127.9 (CH_{aro}), 128.7 (2 x CH_{aro}), 129.0 (CH_{aro}), 129.1 (2 x CH_{aro}), 129.5 (C^q_{aro}), 132.6 (CH_{aro}), 135.7 (C^q_{aro}), 142.1 (CH_{aro}), 147.1 (C^q_{aro}),150.1 (C^q_{aro}), 169.1 (C=O), 170.9 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{21}N_2O_4$ $[M+H]^+$: 389.1457, found 389.1510.

Minor diastereoisomer (±)-11cB: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3)= 0.27; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.50 (d, *J* = 17.8 Hz, 1H, CH_2), 2.90 (d, *J* = 17.8 Hz, 1H, CH_2), 3.97 (d, *J* = 16.0 Hz, 1H, CH_2), 4.60 (d, *J* = 16.0 Hz, 1H, CH_2), 4.83 (s, 1H, CH), 4.90-5.06 (m, 2H, CH_2), 6.12-6.28 (m, 2H, H_{aro}), 7.31-7.59 (m, 9H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 36.0 (CH_2), 39.2 (CH_2), 44.1 (CH_2), 68.7 (C^q), 85.5 (CH), 109.0 (CH_{aro}), 110.7 (CH_{aro}), 123.3 (CH_{aro}), 124.2 (CH_{aro}), 128.1 (CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (CH_{aro}), 129.2 (2 x CH_{aro}), 130.5 (C^q_{aro}), 132.1 (CH_{aro}), 135.8 (C^q_{aro}), 142.3 (CH_{aro}), 144.1 (C^q_{aro}),149.2 (C^q_{aro}), 168.1 (C=O), 171.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{21}N_2O_4$ $[M+H]^+$: 389.1457, found 389.1510.

***N*-Benzyl-2'-hydroxy-2-(thiophen-2-ylmethyl)spiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11d(A,B)**



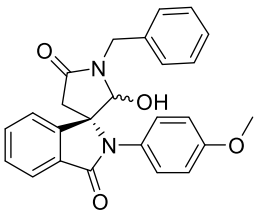
This product was obtained as a mixture of inseparable two diastereoisomers in 86% yield.

Major diastereoisomer (±)-11dA: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.30; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.58 (d, *J* = 17.8 Hz, 1H, CH_2), 2.94 (d, *J* = 17.8 Hz, 1H, CH_2), 3.69 (s, 1H, OH), 4.21 (d, *J* = 16.0 Hz, CH_2), 4.32 (d, *J* = 14.3 Hz, 1H, CH_2), 4.68 (d, *J* = 16.0 Hz, 1H, CH_2), 4.83 (d, *J* = 14.3 Hz, 1H, CH_2), 5.01 (s, 1H, CH), 6.81-6.82 (m, 1H, H_{aro}), 6.86-6.95 (m, 1H, H_{aro}), 7.18 (d, *J* = 5 Hz, 1H, H_{aro}), 7.28-7.42 (m, 6H, H_{aro}), 7.52-7.60 (m, 3H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 38.0 (CH_2), 39.2 (CH_2),44.3 (CH_2), 68.7 (C^q), 85.9 (CH),

123.5 (CH_{aro}), 124.2 (CH_{aro}), 125.9 (CH_{aro}),126.5 (CH_{aro}), 128.2 (CH_{aro}), 128.8 (CH_{aro}), 129.0 (2 x CH_{aro}), 129.2 (2 x CH_{aro}), 130.8 (C^{q}_{aro}), 132.1 (CH_{aro}), 135.8 (C^{q}_{aro}), 139.7 (C^{q}_{aro}), 143.8 (C^{q}_{aro}), 167.9 (C=O), 171.4 (C=O) ppm.

Minor diastereoisomer (±)-11dB: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 1/1) = 0.30. **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.55 (d, *J* = 17.8 Hz, 1H, CH_2), 3.11 (d, *J* = 17.8 Hz, 1H, CH_2), 4.14 (d, *J* = 17.0 Hz, 1H, CH_2), 4.30 (d, *J* = 14.3 Hz, 1H, CH_2), 4.41 (s, 1H, OH), 4.94 (d, *J* = 17.0 Hz, 1H, CH_2), 5.01-5.10 (m, 3H, CH_2), 6.86-6.95 (m, 2H, H_{aro}), 7.04 (d, *J* = 7.3 Hz, 1H, H_{aro}), 7.21 (d, *J* = 5.1 Hz, 1H, H_{aro}), 7.28-7.42 (m, 6H, H_{aro}), 7.52-7.60 (m, 2H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 37.5 (CH_2), 40.2 (CH_2), 43.9 (CH_2), 67.5 (C^{q}), 86.9 (CH), 120.4 (CH_{aro}), 123.6 (CH_{aro}), 125.9 (CH_{aro}), 126.8 (CH_{aro}), 128.0 (CH_{aro}), 128.8 (CH_{aro}), 129.1 (2 x CH_{aro}), 129.2 (2 x CH_{aro}), 129.7 (C^{q}_{aro}), 132.6 (CH_{aro}), 135.6 (C^{q}_{aro}), 140.2 (CH_{aro}), 146.7 (C^{q}_{aro}), 169.6 (C=O), 170.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{21}N_2O_3S$ $[M+H]^+$: 405.1228, found 405.1292.

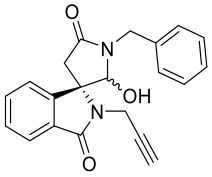
(±)-1'-Benzyl-2'-hydroxy-2-(4-methoxyphenyl)spiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11eA



This product was obtained as a mixture of partially separable two diastereoisomers in 78:22 ratio and 70% global yield. Only the major **(±)-11eA** was described.

Major diastereoisomer (±)-11eA: This product was isolated as a white solid, R_f(cyclohexane/AcOEt: 4/1) = 0.22; mp = 216-218°C; **IR (ν_{max} / cm^{-1})**: 3252, 1788, 11699; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.88 (d, *J* = 17.3 Hz, 1H, CH_2), 3.01 (d, *J* = 17.3 Hz, 1H, CH_2), 3.72 (d, *J* = 5.9 Hz, 1H, CHOH), 3.83 (s, 3H, CH_3), 4.18 (d, *J* = 14.4 Hz, 1H, CH_2), 4.52 (d, *J* = 14.4 Hz, 1H, CH_2), 5.17 (d, *J* = 4.9 Hz, 1H, CHOH), 6.73 (d, *J* = 8.8 Hz, 2H, H_{aro}), 6.90 (d, *J* = 8.8 Hz, 2H, H_{aro}), 7.05-7.08 (m, 2H, H_{aro}), 7.21-7.25 (m, 3H, H_{aro}), 7.37-7.43 (m, 1H, H_{aro}) 7.56-7.58 (m, 2H, H_{aro}), 7.62 (d, *J* = 7.5 Hz, 1H, H_{aro}), ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 39.8 (CH_2), 44.0 (CH_2), 55.4 (CH_3), 70.4 (C^{q}), 85.7 (CH), 115.1 (2 x CH_{aro}), 123.9 (CH_{aro}), 124.0 (CH_{aro}), 126.8 (C^{q}_{aro}), 127.7 (CH_{aro}), 128.7 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.3 (CH_{aro}), 130.3 (2 x CH_{aro}), 131.1 (C^{q}_{aro}), 132.3 (CH_{aro}), 135.4 (C^{q}_{aro}), 143.9 (C^{q}_{aro}), 159.6 (C^{q}_{aro}), 168.6 (C=O), 170.7 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{22}N_2O_4Na$ $[M+Na]^+$: 437.1477, found 437.1480.

1'-Benzyl-2'-hydroxy-2-(prop-2-yn-1-yl)spiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11g(A,B)

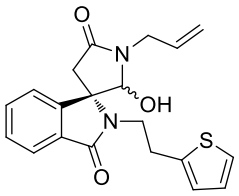


These products, as a mixture of inseparable two diastereoisomers in 63:37 ratio, were obtained as colorless oil in 100% yield according to the general procedure.

Major diastereoisomer (±)-11gA: The NMR characteristics of this product were extracted from the spectrum of the mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.20; **IR (ν_{max} / cm^{-1}):** 3283, 1671, 1659**; ^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.21 (t, *J* = 2.4 Hz, 1H, CH), 2.67 (d, *J* = 17.4 Hz, 1H, CH_2), 3.37 (d, *J* = 17.4 Hz, 1H, CH_2), 4.22 (d, *J* = 14.0 Hz, 1H, CH_2), 4.32 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.52 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.56-4.58 (m, 1H, CHOH), 4.97 (d, *J* = 6.4 Hz, 1H, CHOH), 5.04 (d, *J* = 14.0 Hz, 1H, CH_2), 7.04 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.28-7.43 (m, 5H, H_{aro}), 7.46-7.64 (m, 3H, H_{aro}), ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 30.7 (CH_2), 37.1 (CH_2), 44.0 (CH_2), 67.0 (C^q), 72.1 (CH), 79.3 (C^q), 86.9 (CH), 120.4 (CH_{aro}), 123.7 (CH_{aro}), 128.0 (CH_{aro}), 129.1 (2 x CH_{aro}), 129.2 (3 x CH_{aro}), 129.3 (CH_{aro}), 132.9 (C^q_{aro}), 135.6 (C^q_{aro}), 146.7 (C^q_{aro}), 168.5 (C=O), 170.5 (C=O) ppm.

Minor diastereoisomer (±)-11gB: The NMR characteristics of this product were extracted from the spectrum of the mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.20; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.03 (t, *J* = 2.4 Hz, 1H, CH), 3.01 (d, *J* = 17.4 Hz, 1H, CH_2), 3.08 (d, *J* = 17.4 Hz, 1H, CH_2), 3.70 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 3.77-3.79 (m, 1H, CHOH), 4.21 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.30 (d, *J* = 14.0 Hz, 1H, CH_2), 4.98 (d, *J* = 14.0 Hz, 1H, CH_2), 5.12 (d, *J* = 5.5 Hz, 1H, CHOH), 7.28-7.43 (m, 6H, H_{aro}), 7.46-7.64 (m, 3H, H_{aro}), ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 28.5 (CH_2), 39.4 (CH_2), 44.3 (CH_2), 68.7 (C^q), 72.4 (CH), 78.0 (C^q), 85.6 (CH), 123.6 (CH_{aro}), 124.3 (CH_{aro}), 128.2 (CH_{aro}), 128.8 (3 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 132.3 (C^q_{aro}), 135.8 (C^q_{aro}), 143.7 (C^q_{aro}), 167.4 (C=O), 171.7 (C=O) ppm. HRMS (+ESI) calculated for $C_{21}H_{19}N_2O_3$ $[M+H]^+$: 347.1351, found 347.1349.

***N*-Allyl-2'-hydroxy-2-(2-(thiophen-2-yl)ethyl)spiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11j(A,B)**

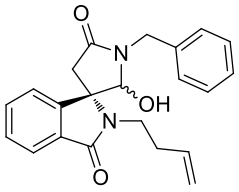


This product was obtained as a mixture of inseparable two diastereoisomers in 82% yield.

Major diastereoisomer (±)-11jA: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.25; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.27 (d, *J* = 17.0 Hz, 1H, CH_2), 2.79 (d, *J* = 17.0 Hz, 1H, CH_2), 3.07-3.31 (m, 2H, CH_2), 3.45-3.91 (m, 3H, CH_2), 4.26-4.38 (m, 1H, CH_2), 5.00 (s, 1H, CH), 5.19-5.35 (m, 2H, CH_2), 5.54-5.62 (m, 1H, OH), 5.73-5.91 (m, 1H, CH=), 6.68 (d, *J* = 3.5 Hz, 1H, H_{aro}), 6.84 (t, *J* = 4.5 Hz, 1H, H_{aro}), 7.05 (d, *J* = 5.1 Hz, 1H, H_{aro}), 7.24 (d, *J* = 5.1 Hz, 1H, H_{aro}), 7.38 (t, *J* = 7.4 Hz, 1H, H_{aro}), 7.50 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.58-7.65 (m, 1H,H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 28.0 (CH_2), 35.7 (CH_2),42.8 (CH_2), 45.1 (CH_2), 67.3 (C^q), 87.4 (CH), 119.6 (CH_2=), 120.2 (CH_{aro}), 123.3 (CH_{aro}), 123.9 (CH_{aro}), 125.5 (CH_{aro}), 127.1 (CH_{aro}), 129.1 (CH_{aro}), 129.9 (C^q_{aro}), 131.6 (CH_{aro}), 132.8 (CH=), 140.7 (C^q_{aro}), 147.7 (C^q_{aro}), 169.4 (C=O), 171.2 (C=O) ppm.

Minor diastereoisomer (±)-11jB: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.25; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.42 (d, *J* = 17.7 Hz, 1H, CH_2), 2.89 (d, *J* = 17.7 Hz, 1H, CH_2), 3.07-3.31 (m, 2H, CH_2), 3.45-3.91 (m, 3H, CH_2), 4.26-4.38 (m, 1H, CH_2), 5.08 (s, 1H, CH), 5.19-5.35 (m, 2H, CH_2), 5.54-5.62 (m, 1H, OH), 5.73-5.91 (m, 1H, CH=), 6.77-6.79 (m, 1H, H_{aro}), 6.90 (t, *J* = 4.4 Hz, 1H, H_{aro}), 7.12 (d, *J* = 5.1 Hz, 1H, H_{aro}), 7.23 (s, 1H, H_{aro}), 7.38 (t, *J* = 7.4 Hz, 1H, H_{aro}), 7.50 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.58-7.65 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 28.7 (CH_2), 39.1 (CH_2),42.8 (CH_2), 43.0 (CH_2), 69.2 (C^q), 85.9 (CH), 120.0 (CH_2=), 123.1 (CH_{aro}), 124.0 (CH_{aro}), 124.2 (CH_{aro}), 125.7 (CH_{aro}), 127.1 (CH_{aro}), 129.1 (CH_{aro}), 131.0 (C^q_{aro}), 131.6 (CH_{aro}), 132.1 (CH=), 140.2 (C^q_{aro}), 144.1 (C^q_{aro}), 168.8 (C=O), 171.2 (C=O) ppm. HRMS (+ESI) calculated for $C_{20}H_{21}N_2O_3S$ $[M+H]^+$: 369.1228, found 369.1285.

***N*-Benzyl-2-(but-3-en-1-yl)-2'-hydroxyspiro[isoindoline-1,3'-pyrrolidine]-3,5'-dione (±)-11k(A,B)**



This product was obtained as a mixture of inseparable two diastereoisomers in 90% yield.

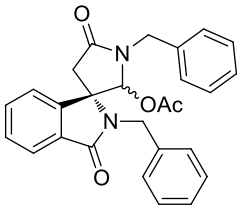
Major diastereoisomer (±)-11kA: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.28; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.24-2.33 (m, 1H, CH_2), 2.40-2.49 (m, 1H, CH_2), 2.59 (d, *J* = 17.0 Hz, 1H, CH_2), 3.16 (d, *J* = 17.0 Hz, 1H, CH_2), 3.63 (t, *J* = 7.3 Hz, 2H, CH_2), 4.21 (d, *J* = 14.2 Hz 1H, CH_2), 4.89-5.49 (m, 4H, CH_2+CH), 5.28 (s, 1H, OH), 5.49-5.85 (m, 1H, CH=), 6.96 (d, *J* = 7.4 Hz, 1H, H_{aro}), 7.18-7.23 (m, 1H, H_{aro}), 7.31-7.63 (m, 7H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 32.6 (CH_2), 36.9 (CH_2), 42.3 (CH_2), 44.0 (CH_2), 67.0 (C^q), 87.0 (CH), 117.3 (CH_2=), 120.1 (CH_{aro}), 123.0 (CH_{aro}), 128.0 (CH_{aro}), 128.8 (2 x CH_{aro}), 129.0 (CH_{aro}), 129.1 (2 x CH_{aro}), 129.9 (C^q_{aro}), 132.3 (CH_{aro}), 134.8 (CH=), 135.7 (C^q_{aro}), 147.2 (C^q_{aro}), 169.2 (C=O), 170.8 (C=O) ppm.

Minor diastereoisomer (±)-11kB: The NMR characteristics of this product were extracted from the spectrum of the purified reaction mixture; R_f (cyclohexane/AcOEt: 2/3) = 0.28; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.98-2.18 (m, 2H, CH_2), 2.71 (d, *J* = 17.7 Hz, 1H, CH_2), 2.85-2.99 (m, 1H, CH_2), 3.08 (d, *J* = 17.1 Hz, 1H, CH_2), 3.21-3.34 (m, 1H, CH_2), 4.89-5.09 (m, 4H, CH_2+CH), 5.49-5.85 (m, 1H, CH=), 7.18-7.23 (m, 1H, H_{aro}), 7.31-7.63 (m, 7H, H_{aro}), 7.70-7.88 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 33.0 (CH_2), 39.6 (CH_2), 39.9 (CH_2), 44.0 (CH_2), 68.7 (C^q), 85.7 (CH), 117.3 (CH_2=), 120.1 (CH_{aro}), 124.2 (CH_{aro}), 128.2 (CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.2 (CH_{aro}), 131.1 (C^q_{aro}), 131.9 (CH_{aro}), 134.4 (CH=), 135.7 (C^q_{aro}), 143.8 (C^q_{aro}), 168.4 (C=O), 171.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{22}H_{23}N_2O_3$ $[M+H]^+$: 363.1664, found 363.1726.

IV. General procedure for the synthesis of α-acetoxylactams (±)-8(A,B)

To a solution of spiro-hydroxylactam **(±)-11(A,B)** (mixture of two diastereoisomers, 0.7 mmol) in 10 mL of CH_2Cl_2 were added acetic anhydride (1.5 equiv.), triethylamine (1.5 equiv.) and dimethylaminopyridine (0.1 equiv.) then the reaction mixture was stirred at room temperature for 1 hour. The reaction mixture was then washed with a saturated aqueous solution of $NaHCO_3$ (10 mL) and extracted twice with dichloromethane. The organic layer was dried over $MgSO_4$ and evaporated under reduced pressure to give an oil residue which was purified by flash column chromatography on silica gel column using cyclohexane-ethyl acetate to afford α-acetoxylactams (±)-**8(A,B)** as following.

(±)-1',2-Dibenzyl-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8a(A,B)

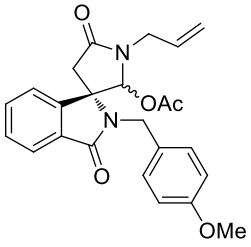


This product was obtained as a mixture of partially separable two diastereoisomers in 91% yield.

Major diastereoisomer (±)-8aA: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 3/2) = 0.48; mp = 166-168 °C; **IR (ν_{max} / cm^{-1})**: 1741, 1705, 1699; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.61 (s, 3H, CH_3), 2.41 (d, *J* = 17.0 Hz, 1H, CH_2), 3.14 (d, *J* = 17.0 Hz, 1H, CH_2), 4.32 (d, *J* = 14.4 Hz, 1H, CH_2=), 4.73 (d, *J* = 16.3 Hz, 1H, CH_2), 4.75 (d, *J* = 14.3Hz, 1H, CH_2), 5.15 (d, *J* = 16.3 Hz, 1H, CH_2), 6.14 (s, 1H, CH), 6.86 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.14 (d, *J* = 6.9 Hz, 2H, H_{ar}), 7.21-7.28 (m, 2H, H_{aro}), 7.31-7.36 (m, 7H, H_{aro}), 7.47 (t, *J* = 7.5 Hz, 1H, H_{aro}), 7.85 (d, *J* = 7.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.2 (CH_3), 36.6 (CH_2), 45.1 (CH_2), 45.2 (CH_2), 65.5 (C^q), 86.2 (CH), 120.1 (CH_{aro}), 124.2 (CH_{aro}), 126.4 (2 x CH_{aro}), 127.2 (CH_{aro}), 128.1 (CH_{aro}),128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 129.6 (C^q_{aro}), 132.8(CH_{aro}), 135.5 (C^q_{aro}), 137.4 (C^q_{aro}), 146.8 (C^q_{aro}), 168.9 (C=O), 169.8 (C=O), 171.5 (C=O) ppm. HRMS (+ESI) calculated for $C_{54}H_{48}N_4O_8Na$ $[2M+Na]^+$: 903.3370, found 903.3412.

Minor diastereoisomer (±)-8aB: The NMR characteristics were extracted from the spectra of the mixture.; R_f (cyclohexane/AcOEt : 1/1) = 0.48; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.49 (s, 3H, CH_3), 2.57 (d, *J* = 17.7 Hz, 1H, CH_2), 3.13 (d, *J* = 17.7 Hz, 1H, CH_2), 3.46 (d, *J* = 14.6 Hz, 1H, CH_2), 4.41 (d, *J* = 16.1 Hz, 1H, CH_2=), 4.55 (d, *J* = 14.6 Hz, 1H, CH_2), 4.68-4.78 (m, 1H, CH_2), 5.97 (s, 1H, CH), 6.86 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.14-7.16 (m, 2H, H_{ar}), 7.22-7.35 (m, 10H, H_{aro}), 7.44-7.54 (m, 2H, H_{aro}), 7.84-7.93 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.1 (CH_3), 39.3 (CH_2), 43.1 (CH_2), 45.6 (CH_2), 67.5 (C^q), 87.3 (CH), 120.1 (CH_{aro}), 123.5 (CH_{aro}), 126.4 (2 x CH_{aro}), 127.4 (CH_{aro}), 127.9 (CH_{aro}), 128.7 (2 x CH_{aro}), 128.8 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 129.6 (C^q_{aro}), 132.8 (CH_{aro}), 135.7 (C^q_{aro}), 137.4 (C^q_{aro}), 146.8 (C^q_{aro}), 167.8 (C=O), 168.5 (C=O), 172.3 (C=O) ppm. HRMS (+ESI) calculated for $C_{27}H_{24}N_2O_4Na$ $[M+Na]^+$: 463.1634, found 463.1658

(±)-1'-Allyl-2-(4-methoxybenzyl)-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8b(A,B)

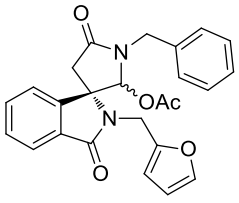


This product was obtained as a mixture of separable two diastereoisomers and were isolated as white solid in 93% global yield;

Major diastereoisomer (±)-8bA: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 4/1) = 0.45; mp = 66-68 °C; **IR (ν_{max} / cm^{-1})**: 1716, 1705, 1698; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.84 (s, 3H, CH_3), 2.35 (d, *J* = 17.1 Hz, 1H, CH_2), 3.04 (d, *J* = 17.1 Hz, 1H, CH_2), 3.66-3.77 (m, 4H, CH_2+ OCH_3), 4.15 (dd, *J* = 14.9, 6.3 Hz, 1H, CH_2), 4.57 (d, *J* = 15.9 Hz, 1H, CH_2), 5.13-5.22 (m, 3H, CH_2+ CH2=), 5.67-5.81 (m, 1H, CH=), 6.09 (s, 1H, CH), 6.75 (d, *J* = 8.1 Hz, 2H, H_{aro}), 7.07 (d, *J* = 8.1 Hz, 2H, H_{aro}), 7.21-7.26 (m, 1H, H_{aro}), 7.49-7.53 (m, 2H, H_{aro}), 7.85 (d, *J* = 6.8 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.5 (CH_3), 36.8 (CH_2), 43.8 (CH_2), 44.6 (CH_2), 55.2 (OCH_3), 65.7 (C^q), 86.6 (CH), 114.2 (2 x CH_{aro}), 119.7 (CH_2=), 120.2 (CH_{aro}), 124.2 (CH_{aro}), 128.0 (2 x CH_{aro}), 129.6 (CH=), 130.0 (C^q_{aro}), 131.1(CH_{aro}), 132.9 (CH_{aro}), 147.0 (C^q_{aro}), 158.9 (C^q_{aro}), 168.8 (C=O), 169.4 (C=O), 171.3 (C=O) ppm. HRMS (+ESI) calculated for $C_{24}H_{25}N_2O_5$ $[M+H]^+$: 421.1719, found 421.1783.

Minor diastereoisomer (±)-8bB: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 4/1) = 0.55; mp = 145-147 °C; **IR (ν_{max} / cm^{-1})**: 1737, 1715, 1688; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.86 (s, 3H, CH_3), 2.55 (d, *J* = 18.0 Hz, 1H, CH_2), 3.09 (d, *J* = 18.0 Hz, 1H, CH_2), 3.40 (dd, *J* = 15.0, *J* = 6.8 Hz, 1H, CH_2=), 3.79 (s, 3H, OCH_3), 3.84 (dd, *J* = 15.0, *J* = 6.8 Hz, 1H, CH_2), 4.45 (d, *J* = 15.8 Hz, 1H, CH_2), 4.83 (d, *J* = 15.8 Hz, 1H, CH_2), 5.07-5.12 (m, 2H, CH_2=), 5.51-5.64 (m, 1H, CH=), 5.93 (s, 1H, CH), 6.85 (d, *J* = 8.7 Hz, 2H, H_{aro}), 7.22 (d, *J* = 8.3 Hz, 2H, H_{ar}), 7.50-7.59 (m, 3H, H_{aro}), 7.92 (d, *J* = 7.2 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.6 (CH_3), 39.5 (CH_2), 42.8 (CH_2), 44.5 (CH_2), 55.3 (OCH_3), 67.3 (C^q), 87.3 (CH), 114.2 (2 x CH_{aro}), 119.2 (CH_2=), 123.4 (CH_{aro}), 124.1 (CH_{aro}), 128.6 (2 x CH_{aro}), 129.3 (C^q_{aro}), 129.6 (CH=), 131.3 (CH_{aro}), 131.7 (C^q_{aro}), 131.8 (CH_{aro}), 142.5 (C^q_{aro}), 159.2 (C^q_{aro}), 167.8 (C=O), 168.6 (C=O), 172.2 (C=O) ppm. HRMS (+ESI) calculated for $C_{24}H_{25}N_2O_5$ $[M+H]^+$: 421.1719, found 421.1785.

(±)-1'-Benzyl-2-(furan-2-ylmethyl)-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8c(A,B)

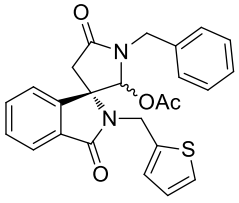


This product was obtained as a mixture of separable two diastereoisomers and were isolated as white solid in 87% global yield.

Major diastereoisomer (±)-8cA: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 4/1) = 0.45; mp = 84-86 °C; **IR (ν_{max} / cm^{-1})**: 1750, 1698, 1616; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.83 (s, 3H, CH_3), 2.46 (d, *J* = 17.3 Hz, 1H, CH_2), 3.35 (d, *J* = 17.3 Hz, 1H, CH_2), 4.30 (d, *J* = 14.4 Hz, 1H, CH_2), 4.56 (d, *J* = 16.2 Hz, 1H, CH_2), 4.70 (d, *J* = 14.4 Hz, 1H, CH_2), 5.06 (d, *J* = 16.2 Hz, 1H, CH_2), 6.02 (s, 1H, CH), 6.22 (s, 2H, H_{aro}), 6.79 (d, *J* = 7.6 Hz, 1H, H_{ar}), 7.19-7.38 (m, 8H, H_{aro}), 7.71 (d, *J* = 7.4 Hz, 1H, H_{aro}), ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.6 (CH_3), 36.4 (CH_2), 38.5 (CH_2), 45.1 (CH_2), 64.9 (C^q), 86.5 (CH), 108.6 (CH_{aro}), 110.8 (CH_{aro}), 120.0 (CH_{aro}), 124.0 (CH_{aro}), 128.1 (CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.4 (CH_{aro}), 129.5 (C^q_{aro}), 132.8 (CH_{aro}), 135.5 (C^q_{aro}), 141.7 (CH_{aro}), 146.7 (C^q_{aro}), 150.1 (C^q_{aro}), 168.4 (C=O), 169.5 (C=O), 171.7 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{22}N_2O_5Na$ $[M+Na]^+$: 453.1426, found 453.1442.

Minor diastereoisomer (±)-8cB: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 4/1) = 0.50; mp = 164-166 °C; **IR (ν_{max} / cm^{-1})**: 1742, 1720, 1701; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.66 (s, 3H, CH_3), 2.54 (d, *J* = 18.1Hz, 1H, CH_2), 3.10 (d, *J* = 18.1 Hz, 1H, CH_2), 4.17 (d, *J* = 17.2 Hz, 1H, CH_2), 4.47 (s, 2H, CH_2), 4.68 (d, *J* = 16.2 Hz, 1H, CH_2), 6.11 (s, 1H, CH), 6.22 (d, *J* = 3.3 Hz, 1H, H_{aro}), 6.31-6.33 (m, 1H, H_{aro}), 7.27-7.37 (m, 6H, H_{aro}), 7.44-7.57 (m,3H, H_{aro}), 7.83 (d, *J* = 7.1 Hz, 1H, H_{aro}), ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 36.0 (CH_2), 39.5 (CH_2), 45.4 (CH_2), 66.8 (C^q), 86.7(CH), 108.7(CH_{aro}), 110.7 (CH_{aro}), 123.4 (CH_{aro}), 124.1 (CH_{aro}), 128.2 (CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.5 (CH_{aro}), 131.4 (C^q_{aro}), 131.7 (CH_{aro}), 135.5 (C^q_{aro}), 142.3 (CH_{aro}), 142.6 (C^q_{aro}), 149.6 (C^q_{aro}), 167.2 (C=O),168.7 (C=O), 172.3 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{23}N_2O_5$ $[M+H]^+$: 431.1562, found 431.1631.

(±)-1'-Benzyl-3,5'-dioxo-2-(thiophen-2-ylmethyl)spiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8d(A,B)



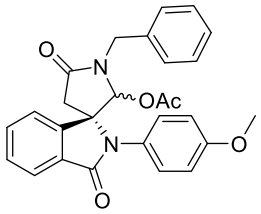
This product was obtained as a mixture of separable two diastereoisomers and were isolated as white solid in 81% global yield.

Major diastereoisomer (±)-8dA: **T**his product was isolated as a white solid, R_f (cyclohexane/AcOEt: 4/1) = 0.42; mp = 82-84 °C; **IR (ν_{max} / cm^{-1})**: 1740, 1705, 1687; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.87 (s, 3H, CH_3), 2.50 (d, *J* = 17.1 Hz, 1H, CH_2), 3.30 (d, *J* = 17.1 Hz, 1H, CH_2), 4.36 (d, *J* = 14.3 Hz, 1H, CH_2), 4.77 (d, *J* = 14.3 Hz, 1H, CH_2), 4.83 (d, *J* = 16.2 Hz, 1H, CH_2), 5.32 (d, *J* = 16.2 Hz, 1H, CH_2), 6.10 (s, 1H, CH), 6.84-6.96 (m, 3H, H_{aro}), 7.17 (d, *J* = 5.0 Hz, 1H, H_{ar}), 7.30-7.47 (m, 7H, H_{aro}), 7.82 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.5 (CH_3), 36.6 (CH_2), 40.5 (CH_2), 45.1 (CH_2), 65.2 (C^q), 86.5 (CH), 120.0 (CH_{aro}), 124.1 (CH_{aro}), 125.2 (CH_{aro}), 126.1 (CH_{aro}), 126.7 (CH_{aro}), 128.1 (CH_{aro}), 128.8 (2 x

CH_{aro}), 128.9 (2 x CH_{aro}), 129.4 (CH_{aro}), 129.5 (C^q_{aro}), 132.8 (CH_{aro}), 135.5 (C^q_{aro}), 140.2 (C^q_{aro}), 146.7 (C^q_{aro}), 168.5 (C=O), 169.5 (C=O), 171.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{22}N_2O_4SNa$ $[M+Na]^+$: 469.1198, found 469.1228.

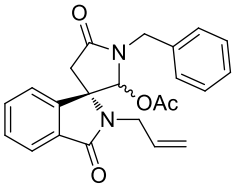
Minor diastereoisomer (±)-8dB: This product was isolated as a white solid, R_f (cyclohexane/AcOEt: 4/1) = 0.52; mp = 78-80 °C; **IR (ν_{max} / cm^{-1})**: 1746, 1715, 1691; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.64 (s, 3H, CH_3), 2.60 (d, *J* = 18.0 Hz, 1H, CH_2), 3.11 (d, *J* = 18.0 Hz, 1H, CH_2), 4.26 (d, *J* = 14.5 Hz, 1H, CH_2), 4.40 (d, *J* = 16.1 Hz, 1H, CH_2), 4.48 (d, *J* = 14.5 Hz, 1H, CH_2), 4.76 (d, *J* = 16.1 Hz, 1H, CH_2), 6.10 (s, 1H, CH), 6.87-6.95 (m, 2H, H_{aro}), 7.22 (d, *J* = 5.0 Hz, 1H, H_{ar}), 7.33 (s, 5H, H_{aro}), 7.44-7.61 (m, 3H, H_{aro}), 7.86 (d, *J* = 6.7 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 38.1 (CH_2), 39.4 (CH_2), 45.4 (CH_2), 67.1 (C^q), 86.9 (CH), 123.5 (CH_{aro}), 124.2 (CH_{aro}), 126.0 (CH_{aro}), 126.4 (CH_{aro}),126.7 (CH_{aro}), 128.2 (CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.6 (CH_{aro}), 131.4 (C^q_{aro}), 131.8 (CH_{aro}), 135.5 (C^q_{aro}), 140.0 (C^q_{aro}), 142.0 (C^q_{aro}), 167.3 (C=O), 168.7 (C=O), 172.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{25}H_{22}N_2O_4SNa$ $[M+Na]^+$: 469.1198, found 469.1230.

(±)-1'-Benzyl-2-(4-methoxyphenyl)-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8eA



This product was prepared from the major diastereoisomer **(±)-11eA** and isolated as a white solid in 81% yield, R_f (cyclohexane/AcOEt: 4/1) = 0.45; mp = 182-184 °C; **IR (ν_{max} / cm^{-1})**: 1738, 1695, 1690; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.70 (s, 3H, CH_3), 2.98 (d, *J* = 17.9 Hz, 1H, CH_2), 3.26 (d, *J* = 17.9 Hz, 1H, CH_2), 3.83 (s, 3H, CH_3), 4.13 (d, *J* = 14.6, Hz, 1H, CH_2), 4.22 (d, *J* = 14.6 Hz, 1H, CH_2), 6.27 (s, 1H, CH), 6.82 (d, *J* = 8.8 Hz, 2H, H_{aro}), 6.97-7.00 (m, 2H, H_{aro}), 7.02 (d, *J* = 8.8 Hz, 2H, H_{aro}), 7.22-7.25 (m, 3H, H_{aro}), 7.51-7.61 (m, 3H, H_{aro}), 7.89 (d, *J* = 7.3 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 40.8 (CH_2), 45.0 (CH_2), 55.4 (OCH_3), 68.2 (C^q), 87.2 (CH), 115.1 (2 x CH_{aro}), 123.5 (CH_{aro}), 124.5 (CH_{aro}), 126.9 (C^q_{aro}), 127.8 (CH_{aro}), 128.6 (2 x CH_{aro}), 128.7 (2 x CH_{aro}), 129.6 (CH_{aro}), 129.8 (2 x CH_{aro}), 131.8 (C^q_{aro}), 132.0 (CH_{aro}), 134.9 (C^q_{aro}), 142.6 (C^q_{aro}), 159.7 (C^q_{aro}), 167.7 (C=O), 168.9 (C=O), 171.9 (C=O) ppm. HRMS (+ESI) calculated for $C_{27}H_{24}N_2O_5Na$[M+Na]$^+$: 479.1583, found 479.1587.

(±)-2-Allyl-1'-benzyl-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8f(A,B)

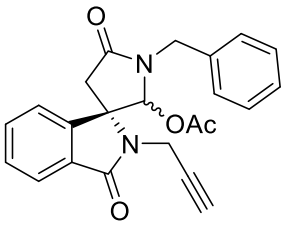


This product was obtained as a mixture of two inseparable diastereoisomers and was isolated as a yellow oil in 93% global yield.

Major diastereoisomer (±)-8fA: The NMR characteristics were extracted from the spectra of the purified reaction mixture; R_f (cyclohexane/AcOEt : 1/1) = 0.48; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.87 (s, 3H, CH_3), 2.48 (d, *J* = 17.1 Hz, 1H, CH_2), 3.24 (d, *J* = 17.1 Hz, 1H, CH_2), 4.01 (dd, *J* = 6.1 and 16.6 Hz, 1H, CH_2), 4.29 (d, *J* = 14.4 Hz, 1H, CH_2), 4.36-4.55 (m, 1H, CH_2), 4.68 (d, *J* = 14.4 Hz, 1H, CH_2), 4.98-5.19 (m, 2H, CH_2), 5.74-5.86 (m, 1H, CH=), 5.99 (s, 1H, CH), 6.79 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.20-7.56 (m, 7H, H_{aro}), 7.70 (d, *J* = 7.5Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.9 (CH_3), 36.4 (CH_2), 44.3 (CH_2), 45.1 (CH_2), 65.1 (C^q), 86.4 (CH), 116.6 (CH_2=), 119.9 (CH_{aro}), 123.9 (CH_{aro}), 128.1 (CH_{aro}),128.8 (2xCH_{aro}), 128.9 (2xCH_{aro}), 129.4 (CH_{aro}), 129.8 (C^q_{aro}), 132.7 (CH_{aro}),133.1 (CH=), 135.5 (C^q_{aro}), 146.8 (C^q_{aro}), 168.3 (C=O), 169.5 (C=O), 171.6 (C=O) ppm.

Minor diastereoisomer (±)-8fB: The NMR characteristics were extracted from the spectra of the purified reaction mixture. R_f (cyclohexane/AcOEt : 1/1) = 0.48; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.61 (s, 3H, CH_3), 2.65 (d, *J* = 17.1 Hz, 1H, CH_2), 3.07 (d, *J* = 17.9 Hz, 1H, CH_2), 3.86 (dd, *J* = 5.6 and 16.2 Hz, 1H, CH_2), 4.14 (d, *J* = 14.6 Hz, 1H, CH_2), 4.36-4.55 (m, 1H, CH_2), 4.77 (d, *J* = 14.5 Hz, 1H, CH_2), 4.98-5.19 (m, 2H, CH_2), 5.58-5.70 (m, 1H, CH=), 6.09 (s, 1H, CH), 6.78 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.20-7.56 (m, 7H, H_{aro}), 7.69 (d, *J* = 7.5Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 39.9 (CH_2), 42.2 (CH_2), 45.3 (CH_2), 65.1 (C^q), 86.9 (CH), 117.9 (CH_2=), 120.1 (CH_{aro}), 123.5 (CH_{aro}), 128.6 (CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 129.8 (C^q_{aro}), 132.4 (CH_{aro}),133.2 (CH=), 135.6 (C^q_{aro}), 146.8 (C^q_{aro}), 167.1 (C=O), 168.6 (C=O), 172.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{22}N_2O_4Na$ $[M+Na]^+$: 413.1477, found 413.1491.

(±)-1',2-Dibenzyl-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8g(A,B)



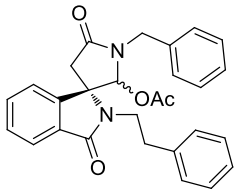
These products, as a mixture of inseparable two diastereoisomers in 63:37 ratio, were obtained as colorless oil in 91% yield according to the general procedure.

Major diastereoisomer (±)-8gA: : The NMR characteristics of this product were extracted from the spectra of the mixture, Rf (cyclohexan/AcOEt: 3/2) = 0.30; **IR (ν_{max} / cm^{-1})**: 1752, 1698, 1665; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.95 (s, 3H, CH_3), 2.21 (t, *J* = 2.4 Hz, 1H, CH), 2.62 (d, *J* = 17.2 Hz, 1H, CH_2), 3.58 (d, *J* = 17.2 Hz, 1H, CH_2), 4.22 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.40 (d, *J* = 14.4 Hz, 1H, CH_2), 4.67 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.75 (d, *J* = 14.4 Hz, 1H, CH_2), 6.09 (s, 1H, CH), 6.85 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.30-7.43 (m, 5H, H_{aro}), 7.46-7.63 (m, 2H, H_{aro}), 7.79-7.84 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.9 (CH_3), 31.0 (CH_2), 36.2 (CH_2), 45.2 (CH_2), 64.9 (C^q), 71.8 (CH), 78.3 (C^q), 86.3 (CH), 120.0 (CH_{aro}), 124.1 (CH_{aro}), 128.1 (CH_{aro}), 128.8 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 132.0

(C^q_{aro}), 133.0 (CH_{aro}), 135.6 (C^q_{aro}), 146.8 (C^q_{aro}), 168.8 (C=O), 169.5 (C=O), 171.5 (C=O) ppm.

Minor diastereoisomer (±)-8gB: The NMR characteristics were extracted from the spectra of the mixture.; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.71 (s, 3H, CH_3), 2.24 (t, *J* = 2.4 Hz, 1H, CH), 3.02 (d, *J* = 18.0 Hz, 1H, CH_2), 3.20 (d, *J* = 18.0 Hz, 1H, CH_2), 3.80 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.30 (dd, *J* = 2.4 and 18.0 Hz, 1H, CH_2), 4.46 (d, *J* = 18.4 Hz, 1H, CH_2), 4.74 (d, *J* = 18.4 Hz, 1H, CH_2), 6.25 (s, 1H, CH), 7.30-7.43 (m, 6H, H_{aro}), 7.46-7.63 (m, 2H, H_{aro}), 7.79-7.84 (m, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 28.4 (CH_2), 39.8 (CH_2), 45.3 (CH_2), 67.1 (C^q), 72.4 (CH), 78.3 (C^q), 86.4 (CH), 123.5 (CH_{aro}), 124.2 (CH_{aro}), 128.4 (CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 131.2 (C^q_{aro}), 133.0 (CH_{aro}), 135.5 (C^q_{aro}), 142.3 (C^q_{aro}), 166.5 (C=O), 168.2 (C=O), 168.8 (C=O) ppm. HRMS (+ESI) calculated for $C_{23}H_{20}N_2O_4Na[M+Na]^+$: 411.1315, found 411.1317

(±)-1'-Benzyl-3,5'-dioxo-2-phenethylspiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8h (A,B)



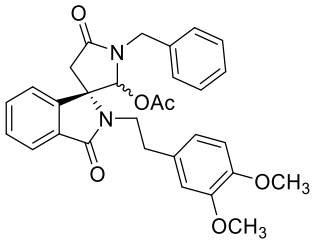
This product was obtained as a mixture of two partially separable diastereoisomers and was isolated as a yellow oil in 91% global yield.

Major diastereoisomer (±)-8hA: This product was isolated as a yellow oil; R_f (cyclohexane/AcOEt: 3/2) = 0.53; **IR (ν_{max} / cm^{-1})**: 1753, 1695, 1679; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.88 (s, 3H, CH_3), 2.16 (d, *J* = 17.1 Hz, 1H, CH_2), 2.75 (d, *J* = 17.1 Hz, 1H, CH_2), 3.00-3.06 (m, 2H, CH_2), 3.50-3.60 (m, 1H, CH_2), 3.99-4.08 (m, 1H, CH_2), 4.31 (d, *J* = 14.4 Hz, 1H, CH_2), 4.65 (d, *J* = 14.4 Hz, 1H, CH_2), 5.93 (s, 1H, CH), 6.80 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.15-7.24 (m, 4H, H_{aro}), 7.28-7.30 (m, 6H, H_{aro}),7.42 (t, *J* = 7.4 Hz, 2H, H_{aro}), 7.78 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.8 (CH_3), 33.9 (CH_2), 35.7 (CH_2), 44.7 (CH_2), 45.1 (CH_2), 65.3 (C^q), 86.6 (CH), 119.9 (CH_{aro}), 123.7 (CH_{aro}), 126.7 (CH_{aro}), 128.1 (CH_{aro}), 128.7 (2 x CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.4 (CH_{aro}), 130.2 (C^q_{aro}), 132.5 (CH_{aro}), 135.5 (C^q_{aro}), 138.8 (C^q_{aro}), 146.6 (C^q_{aro}), 168.6 (C=O), 169.4 (C=O), 171.5 (C=O) ppm. HRMS (+ESI) calculated for $C_{28}H_{27}N_2O_4$ $[M+H]^+$: 455.1926, found 455.1993.

Minor diastereoisomer (±)-8hB: The NMR characteristics were extracted from the spectra of the mixture **(±)-8hB** and **(±)-9hA**. **IR (ν_{max} / cm^{-1})**: 1753, 1695, 1679; R_f (cyclohexane/ AcOEt : 1/1) = 0.50; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.73 (s, 3H, CH_3), 2.46 (d, *J* = 18.0 Hz, 1H, CH_2), 2.75-2.97 (m, 2H, CH_2), 3.03-3.25 (d, *J* = 18.0 Hz, 2H, CH_2), 3.50-3.60 (m, 1H, CH_2), 4.32 (d, *J* = 14.4 Hz, 1H, CH_2), 4.69 (d, *J* = 14.4 Hz, 1H, CH_2), 6.03 (s, 1H, CH), 7.13 (d, *J* = 6.7 Hz, 2H, H_{aro}), 7.20-7.31 (m, 9H, H_{aro}), 7.36-7.49 (m, 2H, H_{aro}), 7.73 (d, *J* = 6.6 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 35.1 (CH_2), 39.8 (CH_2), 42.3 (CH_2), 45.1 (CH_2), 66.9 (C^q), 86.7 (CH), 123.4 (CH_{aro}), 123.7 (CH_{aro}), 126.6 (CH_{aro}), 128.2 (CH_{aro}),

128.6 (2 x CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.5 (CH_{aro}), 131.5 (CH_{aro}), 131.9 (C^{q}_{aro}), 135.2 (C^{q}_{aro}), 138.3 (C^{q}_{aro}), 142.5 (C^{q}_{aro}), 167.7 (C=O), 168.8 (C=O), 172.4 (C=O) ppm. HRMS (+ESI) calculated for $C_{28}H_{27}N_2O_4$ $[M+H]^+$: 455.1926, found 455.1990.

(±)-1'-Benzyl-2-(3,4-dimethoxyphenethyl)-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8i(A,B)

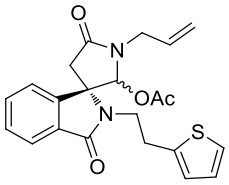


This product was obtained as a mixture of two partially separable diastereoisomers and was isolated as a yellow oil in 93% global yield.

Major diastereoisomer (±)-8iA: This product was isolated as a yellow oil; R_f (cyclohexane/AcOEt: 1/1) = 0.37; **IR (ν_{max} / cm^{-1})**: 1738, 1715, 1698; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.93 (s, 3H, CH_3), 2.21 (d, *J* = 17.1 Hz, 1H, CH_2), 2.84(d, *J* = 17.1 Hz, 1H, CH_2), 2.96-3.11 (m, 2H, CH_2), 3.48-3.58 (m, 1H, CH_2), 3.69 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 4.00-4.09 (m, 1H, CH_2), 4.33 (d, *J* = 14.4 Hz, 1H, CH_2), 4.72 (d, *J* = 14.4 Hz, 1H, CH_2), 5.96 (s, 1H, CH), 6.66 (s, 1H, H_{aro}), 6.74-6.85 (m, 2H, H_{aro}), 7.32-7.35 (m, 6H, H_{aro}),7.46 (t, *J* = 7.5 Hz, 2H, H_{aro}), 7.82 (d, *J* = 7.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.8 (CH_3), 33.3 (CH_2), 35.6 (CH_2), 44.8 (CH_2), 45.1 (CH_2), 55.6 (OCH_3), 55.9 (OCH_3), 65.3 (C^{q}), 86.5 (CH), 111.3 (CH_{aro}), 112.2 (CH_{aro}), 120.0 (CH_{aro}), 120.7 (CH_{aro}), 123.6 (CH_{aro}), 128.1 (CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.4 (CH_{aro}), 130.2 (C^{q}_{aro}), 131.2 (C^{q}_{aro}), 132.5 (CH_{aro}), 135.4 (C^{q}_{aro}), 146.5 (C^{q}_{aro}), 147.8 (C^{q}_{aro}), 148.9 (C^{q}_{aro}), 168.6 (C=O), 169.4 (C=O), 171.5 (C=O) ppm. HRMS (+ESI) calculated for $C_{30}H_{31}N_2O_6$ $[M+H]^+$: 515.2137 found 515.2213.

Minor diastereoisomer (±)-8iA: The NMR characteristics were extracted from the spectra of the purified reaction mixture. R_f (cyclohexane/AcOEt: 1/1) = 0.39; **IR (ν_{max} / cm^{-1}):** 1614.96, 1698.26 (2xC=O); ; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.74 (s, 3H, CH_3), 2.44 (d, *J* = 18.0 Hz, 1H, CH_2), 3.13 (d, *J* = 18.0 Hz, 1H, CH_2), 2.96-3.11 (m, 2H, CH_2), 3.48-3.58 (m, 1H, CH_2), 3.78 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 4.00-4.09 (m, 1H, CH_2), 4.33 (d, *J* = 14.4 Hz, 1H, CH_2), 4.87 (d, *J* = 14.4 Hz, 1H, CH_2), 6.02 (s, 1H, CH), 6.74-6.85 (m, 4H, H_{aro}), 7.32-7.35 (m, 7H, H_{aro}), 7.82 (d, *J* = 7.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.3 (CH_3), 33.9 (CH_2), 35.7 (CH_2), 45.2 (CH_2), 45.8 (CH_2), 55.7 (OCH_3), 55.8 (OCH_3), 67.1 (C^{q}), 86.8 (CH), 111.3 (CH_{aro}), 112.0 (CH_{aro}), 120.1 (CH_{aro}), 120.6 (CH_{aro}), 123.7 (CH_{aro}), 128.4 (CH_{aro}), 128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.3 (CH_{aro}), 131.5 (C^{q}_{aro}), 131.8 (C^{q}_{aro}), 132.4 (CH_{aro}), 135.2 (C^{q}_{aro}), 145.3 (C^{q}_{aro}), 147.7 (C^{q}_{aro}), 149.8 (C^{q}_{aro}), 168.7 (C=O), 171.5 (C=O), 172.5 (C=O) ppm.

(±)-1'-Allyl-3,5'-dioxo-2-(2-(thiophen-2-yl)ethyl)spiro[isoindoline-1,3'-pyrrolidin]-2'-yl acetate (±)-8j(A,B)

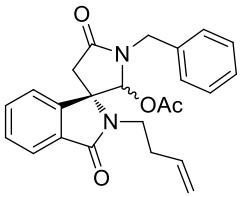


This product was obtained as a mixture of two partially separable diastereoisomers and was isolated as a yellow oil in 89% global yield.

Major diastereoisomer (±)-8jA: This product was isolated as a yellow oil; R_f (cyclohexane/AcOEt: 3/2) = 0.54; **IR (ν_{max} / cm^{-1})**: 1693, 1680, 1668; **^{1}H NMR (300 MHz, CDCl$_3$)**: δ_H 2.10 (s, 3H, CH_3), 2.25 (d, *J* = 17.1 Hz, 1H, CH_2), 2.80 (d, *J* = 17.1 Hz, 1H, CH_2), 3.18-3.27 (m, 1H, CH_2), 3.40-3.50 (m, 1H, CH_2), 3.61-3.71 (m, 1H, CH_2), 3.79 (dd, *J* = 14.1, *J* = 6.8 Hz, 1H, CH_2), 4.05-4.14 (m, 1H, CH_2), 4.18 (dd, *J* = 6.3 and 14.9 Hz, 1H, CH_2), 5.19-5.27 (m, 2H, CH_2=), 5.71-5.84 (m, 1H, CH=), 5.99 (s, 1H, CH), 6.80 (d, *J* = 3.4 Hz, 1H, H_{aro}), 6.91 (t, *J* = 5 Hz, 1H, H_{aro}), 7.13 (d, *J* = 5 Hz, 1H, H_{aro}), 7.24 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.52-7.60 (m, 2H, H_{aro}), 7.88 (d, *J* = 8.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ_C 21.0 (CH_3), 27.8 (CH_2), 35.4 (CH_2), 43.9 (CH_2), 44.9 (CH_2), 65.5 (C^q), 86.6 (CH), 119.7 (CH_2=), 120.7 (CH_{aro}), 123.9 (CH_{aro}), 124.1 (CH_{aro}), 125.7 (CH_{aro}), 127.2 (CH_{aro}), 129.6 (CH_{aro}), 130.3 (C^q_{aro}), 131.0 (CH_{aro}),132.8 (CH=), 140.6 (C^q_{aro}), 146.8 (C^q_{aro}), 168.6 (C=O), 169.3 (C=O), 171.5 (C=O) ppm. HRMS (+ESI) calculated for $C_{22}H_{22}N_2O_4SNa$ $[M+Na]^+$: 433.1198, found 433.1220.

Minor diastereoisomer (±)-8jB: The NMR characteristics were extracted from the spectra of the mixture **(±)-8jB** + **(±)-9jA**. **IR (ν_{max} / cm^{-1})**: 1690, 1689 (2 x C=O); **^{1}H NMR (300 MHz, CDCl$_3$)**: δ_H 2.04 (s, 3H, CH_3), 2.36 (d, *J* = 18.1 Hz, 1H, CH_2), 2.99-3.13 (m, 2H, CH_2), 3.28-3.40 (m, 1H, CH_2), 3.47-3.78 (m, 4H, CH_2), 4.08 (td, *J* = 6.1 and 15.9 Hz, 1H, CH_2), 5.16-5.23 (m, 2H, CH_2=), 5.68-5.82 (m, 1H, CH=), 6.03 (s, 1H, CH), 6.38 (d, *J* = 6.9 Hz, 1H, H_{aro}), 6.80-6.88 (m, 2H, H_{aro}), 7.06-7.13 (m, 2H, H_{aro}), 7.40-7.53 (m, 1H, H_{aro}), 7.76 (t, *J* = 6.1Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ_C 20.6 (CH_3), 29.1 (CH_2), 39.7 (CH_2), 42.9 (CH_2), 44.6 (CH_2), 67.1 (C^q), 86.8 (CH), 119.5 (CH_2=), 120.3 (CH_{aro}), 123.7 (CH_{aro}), 125.7 (CH_{aro}), 127.2 (CH_{aro}), 129.6 (CH_{aro}), 131.1 (CH_{aro}), 131.7 (CH_{aro}), 131.8 (C^q_{aro}),132.5 (CH=), 140.0 (C^q_{aro}), 145.8 (C^q_{aro}), 167.0 (C=O), 170.6 (C=O), 171.3 (C=O) ppm. HRMS (+ESI) calculated for $C_{22}H_{23}N_2O_4S$ $[M+H]^+$: 411.1334, found 411.1402.

(±)-*N*-Benzyl-2-(but-3-en-1-yl)-3,5'-dioxospiro[isoindoline-1,3'-pyrrolidin]-2'-ylacetate (±)-8k(A,B)



This product was obtained as a mixture of two separable diastereoisomers and was isolated as a yellow oil in 91% global yield.

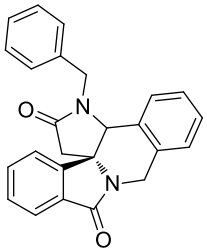
Major diastereoisomer (±)-8kA: This product was isolated as a yellow oil; R_f (cyclohexane/AcOEt: 3/2) = 0.54; **IR (ν_{max} / cm^{-1})**: 1752, 1694 (2 x C=O), **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.91 (s, 3H, CH_3), 2.40-2.52 (m, 2H, CH_2), 2.57 (d, *J* = 16.9 Hz, 1H, CH_2), 3.20 (d, *J* = 16.9 Hz, 1H, CH_2), 3.40-3.51 (m, 1H, CH_2), 3.79-3.89 (m, 1H, CH_2), 4.35 (d, *J* = 14.4 Hz, 1H, CH_2), 4.69 (d, *J* = 14.4 Hz, 1H, CH_2), 5.04-5.11 (m, 2H, CH_2), 5.70-5.84 (m, 1H, CH=), 6.00 (s, 1H, CH), 6.84 (d, *J* = 7.6 Hz, 1H, H_{aro}), 7.26-7.34 (m, 6H, H_{aro}), 7.39 (t, *J* = 7.3 Hz, 1H, H_{aro}), 7.74 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 20.7 (CH_3), 36.4 (CH_2), 36.6 (CH_2), 42.1 (CH_2), 45.2 (CH_2), 65.1 (C^q), 86.5 (CH), 117.4 (CH_2=), 119.9 (CH_{aro}), 123.7 (CH_{aro}), 128.1 (CH_{aro}),128.8 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.4 (CH_{aro}), 130.1(C^q_{aro}), 132.5 (CH_{aro}),134.7 (CH=), 135.5 (C^q_{aro}), 146.5 (C^q_{aro}), 168.5 (C=O), 169.4 (C=O), 171.5 (C=O) ppm. HRMS (+ESI) calculated for $C_{24}H_{24}N_2O_4Na$ $[M+Na]^+$: 427.1634, found 427.1656.

Minor diastereoisomer (±)-8kB: This product was isolated as a yellow oil; R_f (cyclohexane/AcOEt: 3/2) = 0.50; **IR (ν_{max} / cm^{-1})**: 1753.38, 1686.72 (2 x C=O), **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.80 (s, 3H, CH_3), 2.03-2.29 (m, 2H, CH_2), 2.74 (d, *J* = 16.9 Hz, 1H, CH_2), 2.91-3.04 (m, 1H, CH_2), 3.21 (d, *J* = 16.9 Hz, 1H, CH_2), 3.32-3.42 (m, 1H, CH_2), 4.27 (d, *J* = 14.4 Hz, 1H, CH_2), 4.85 (d, *J* = 14.4 Hz, 1H, CH_2), 4.97-5.12 (m, 2H, CH_2), 5.53-5.68 (m, 1H, CH=), 6.11 (s, 1H, CH), 7.26-7.48 (m, 5H, H_{aro}), 7.49-7.58 (m, 3H, H_{aro}), 7.79 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. ^{13}C NMR could not be made because the product is not stable in $CDCl_3$ and it hydrolyzes give the corresponding hydroxylactam under a mixture of two inseparable diastereoisomers.

V. General procedure for π-cationic cyclization of α-acetoxylactams (±)-8

To a solution of α-acetoxylactam **(±)-8** (pure form or mixture of two diastereoisomers 0.7 mmol) in 2 mL of acetonitrile was added 10 mol% of TMSOTf then the reaction mixture was stirred at room temperature for 2 h. The reaction mixture was neutralized with a saturated aqueous solution of $NaHCO_3$ (10 mL) and extracted twice with dichloromethane (20 mL). The organic layer was dried over $MgSO_4$ and evaporated *in vacuo* to give a white product which was purified by flash column chromatography on silica gel column to give compounds **(±)-5** as following.

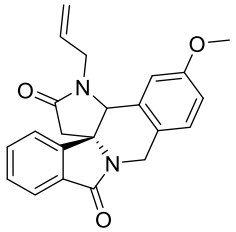
(±)-*N*-Benzyl-1,14b-dihydroisoindolo[2,1-*b*]pyrrolo[3,2-*c*]isoquinoline-2,8(3*H*,10*H*)-dione (±)-5aA



The reaction was carried out on pure α-acetoxylactams (±)-**8aA** or (±)-**8aB**. In both cases the same compound **(±)-5aA** was isolated as sole diastereoisomer in 98% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 3/2) = 0.54; mp = 198-200 °C; **IR (ν_{max} / cm^{-1})**: 1705, 1679; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 3.13 (d, *J* = 18.6 Hz, 1H,

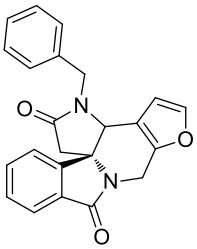
CH_2), 3.19 (d, *J* = 18.6 Hz, 1H, CH_2), 3.67 (d, *J* = 15.1 Hz, 1H, CH_2), 4.25 (d, *J* = 15.7 Hz, 1H, CH_2), 4.84 (s, 1H, CH), 5.12 (d, *J* = 15.1 Hz, 1H, CH_2), 5.22 (d, *J* = 15.7 Hz, 1H, CH_2), 6.99 (d, *J* = 7.4 Hz, 1H, H_{aro}), 7.18-7.23 (m, 3H, H_{aro}), 7.29-7.47 (m, 7H, H_{aro}), 7.55 (t, *J* = 7.5 Hz, 1H, H_{aro}), 7.72 (d, *J* = 7.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 39.9 (CH_2), 41.6 (CH_2), 44.5 (CH_2), 63.4 (CH), 63.9 (C^q), 121.0 (CH_{aro}), 124.0 (CH_{aro}), 127.2 (CH_{aro}), 127.5 (CH_{aro}), 127.8 (CH_{aro}), 128.0 (2 x CH_{aro}), 128.9 (2 x CH_{aro}), 129.1 (CH_{aro}), 130.4 (C^q_{aro}), 130.6 (CH_{aro}), 131.3 (C^q_{aro}), 132.9 (CH_{aro}), 135.6 (C^q_{aro}), 136.0 (C^q_{aro}), 148.3 (C^q_{aro}), 168.2 (C=O), 171.5 (C=O) ppm. HRMS (+ESI) Calculated for $C_{25}H_{21}N_2O_2$ $[M+H]^+$: 381.1558, found: 381.1618.

(±)-*N*-Allyl-13-methoxy-1,14*b*-dihydroisoindolo[2,1-*b*]pyrrolo[3,2-*c*]isoquinoline-2,8-(3*H*,10*H*)-dione (±)-5b(A,B)



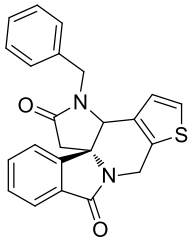
This product was obtained as a mixture of inseparable two diastereoisomers in 70:30 ratio and 88% yield. mp = 76-78 °C; **IR (ν_{max} / cm^{-1})**: 1679, 1611, R_f (cyclohexane/AcOEt : 1/1) = 0.42. **^{1}H NMR (300 MHz, $CDCl_3$; All signals of the spectre were noticed)**: δ_H 2.98-3.14 (m, 4H, CH_2), 3.25-3.42 (m, 2H, CH_2), 3.74 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 4.17 (d, *J* = 7.7 Hz, 1H, CH_2), 4.22 (d, *J* = 7.8 Hz, 1H, CH_2), 4.39-4.54 (m, 2H, CH_2), 4.89 (s, 1H, CH), 4.93 (s, 1H, CH), 5.17-5.29 (m, 6H, CH_2), 5.62-5.79 (s, 2H, CH=), 6.70-6.74 (m, 2H, H_{aro}), 6.81-6.87 (m, 2H, H_{aro}),7.03 (d, *J* = 8.4 Hz, 1H, H_{aro}), 7.24 (s, 1H, H_{aro}),7.47-7.52 (m, 2H, H_{aro}), 7.55-7.58 (m, 2H, H_{aro}), 7.62-7.67 (m, 2H, H_{aro}), 7.77 (d, *J* = 7.6 Hz, 2H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$; All signals of the spectre were noticed):** δ_C 39.1 (CH_2), 40.0 (CH_2), 40.9 (CH_2), 41.3 (CH_2), 43.4(CH_2), 43.6 (CH_2), 55.3 (OCH_3), 55.4 (OCH_3), 63.2 (CH), 63.5 (CH), 63.6 (C^q), 64.0 (C^q), 112.5 (CH_{aro}), 113.0 (CH_{aro}), 113.7 (CH_{aro}), 116.4 (CH_{aro}), 118.4 (CH_{aro}), 119.1 (CH_{aro}), 120.9 (CH_{aro}), 121.0 (CH_{aro}), 122.6 (C^q_{aro}), 124.1 (CH_{aro}), 124.1 (CH_{aro}), 127.4 (C^q_{aro}), 127.4 (CH_{aro}), 129.1 (CH_{aro}), 131.3 (C^q_{aro}), 131.3 (C^q_{aro}), (CH_{aro}), 131.6 (CH_{aro}), 131.7 (CH_{aro}), 131.7 (CH_{aro}), 132.0 (C^q_{aro}), 132.9 (CH_{aro}), 133.0 (CH_{aro}), 137.2 (C^q_{aro}), 148.5 (C^q_{aro}), 148.6 (C^q_{aro}), 158.6 (C^q_{aro}), 159.8 (C^q_{aro}), 167.9 (C=O), 168.2 (C=O), 170.9 (C=O), 171.0 (C=O) ppm. HRMS (+ESI) Calculated for $C_{25}H_{21}N_2O_2$ $[M+H]^+$: 361.1507, found: 361.1569.

(±)-*N*-Benzyl-1,13*b*-dihydrofuro[3',2':4,5]pyrrolo[3',2':2,3]pyrido[2,1-*a*]isoindole-2,8-(3*H*,10*H*)-dione (±)-5cA



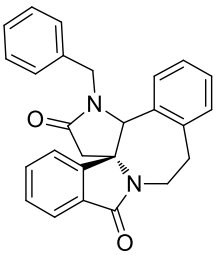
The reaction was carried out on pure α-acetoxylactams **(±)-8cA** or **(±)-8cB**. In both cases the same compound **(±)-5cA** was isolated as sole diastereoisomer in 62% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 2/3) = 0.53; mp = 90-92 °C; **IR (ν_{max} / cm^{-1})**: 1685, 1628; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.47 (d, *J* = 16.5 Hz, 1H, CH_2), 3.08 (d, *J* = 16.5 Hz, 1H, CH_2), 4.19-4.26 (m, 2H, CH_2), 4.39 (s, 1H, CH), 5.28 (d, *J* = 14.6 Hz, 1H, CH_2), 5.31 (d, *J* = 17.0 Hz, 1H, CH_2), 6.27 (d, *J* = 2 Hz, 1H, H_{aro}), 7.13 (d, *J* = 7.3 Hz, 1H, H_{aro}), 7.35 – 7.44 (m, 8H, H_{aro}), 7.78 (d, *J* = 6.6 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 34.9 (CH_2), 37.0 (CH_2), 45.9 (CH_2), 57.8 (CH), 62.6 (C^q), 108.9 (CH_{aro}), 115.6 (C^q_{aro}), 120.1 (CH_{aro}), 123.8 (CH_{aro}), 128.3 (CH_{aro}), 129.0 (2 x CH_{aro}), 129.1 (2 x CH_{aro}), 129.2 (CH_{aro}), 129.5 (C^q_{aro}), 132.67 (CH_{aro}), 135.4 (C^q_{aro}), 143.3 (CH_{aro}), 146.6 (C^q_{aro}), 148.4 (C^q_{aro}), 166.4 (C=O), 170.3 (C=O) ppm. HRMS (+ESI) Calculated for $C_{23}H_{19}N_2O_3$ $[M+H]^+$: 371.1351, found: 371.1417.

(±)-*N*-Benzyl-1,13*b*-dihydropyrrolo[3',2':2,3]thieno[3',2':4,5]pyrido[2,1-a]isoindole-2,8-(3*H*,10*H*)-dione (±)-5dA



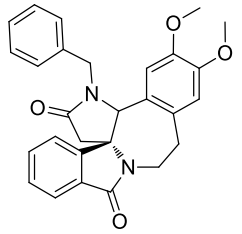
The reaction was carried out on pure α-acetoxylactams **(±)-8dA** or **(±)8dB**. In both cases the same compound **(±)-5dA** was isolated as sole diastereoisomer in 84% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 2/3) = 0.54; mp = 186-188 °C; **IR (ν_{max} / cm^{-1})**: 1681, 1616; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.58 (d, *J* = 16.7 Hz, 1H, CH_2), 3.18 (d, *J* = 16.7 Hz, 1H, CH_2), 4.22 (d, *J* = 14.8 Hz, 1H, CH_2), 4.37 (d, *J* = 17 Hz, 1H, CH_2), 4.51 (s, 1H, CH), 5.43 (d, *J* = 14.8 Hz, 1H, CH_2), 5.51 (d, *J* = 17.0 Hz, 1H, CH_2), 7.02 (d, *J* = 5.1 Hz, 1H, H_{aro}), 7.14 (d, *J* = 7.2 Hz, 1H, H_{aro}), 7.29 (d, *J* = 5.3 Hz, 1H, H_{aro}), 7.34-7.48 (m, 7H, H_{aro}), 7.79 (d, *J* = 7.6 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 35.8 (CH_2), 37.1 (CH_2), 45.7 (CH_2), 59.1 (CH), 62.0 (C^q), 120.2 (CH_{aro}), 123.8 (CH_{aro}), 125.4 (CH_{aro}), 125.7 (CH_{aro}), 128.3 (CH_{aro}), 129.0 (2 x CH_{aro}), 129.1 (2 x CH_{aro}), 129.2 (CH_{aro}), 129.7 (C^q_{aro}), 132.3 (C^q_{aro}), 132.6 (CH_{aro}), 133.9 (C^q_{aro}), 135.3 (C^q_{aro}), 148.7 (C^q_{aro}), 166.0 (C=O), 170.5 (C=O) ppm. HRMS (+ESI) Calculated for $C_{23}H_{19}N_2O_2S$ $[M+H]^+$: 387.1123, found: 387.1180.

(±)-*N*-Benzyl-15,15*a*-dihydro-5*H*-benzo[4,5]pyrrolo[3',2':2,3]azepino[2,1-*a*]isoindole-8,14-(6*H*,13*H*)-dione (±)-5hA



The reaction was carried out on pure α-acetoxylactams **(±)-8hA** or **(±)-8hB**. In both cases the same compound **(±)-5hA** was isolated as sole diastereoisomer in 77% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 3/2) = 0.30; mp = 218-220 °C; **IR (ν_{max} / cm^{-1})**: 1695, 1678; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.85-2.93 (m, 1H, CH_2), 3.09 (d, *J* = 18.4 Hz, 1H, CH_2), 3.18-3.29 (m, 2H, CH_2), 3.37-3.44 (m, 2H, CH_2), 4.50-4.61 (m, 1H, CH_2), 4.79 (s, 1H, CH), 5.18 (d, *J* = 14.3 Hz, 1H, CH_2), 6.51 (d, *J* = 7.4 Hz, 1H, H_{aro}), 7.01-7.10 (m, 3H, H_{aro}), 7.18-7.26 (m, 2H, H_{aro}), 7.31-7.38 (m, 3H, H_{aro}), 7.38-7.44 (m, 2H, H_{aro}), 7.56 -7.66 (m, 2H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 29.9 (CH_2), 35.9 (CH_2), 39.9 (CH_2), 45.0 (CH_2), 64.7(C^q), 72.5 (CH), 120.9 (CH_{aro}), 123.7 (CH_{aro}), 126.9 (CH_{aro}), 127.9 (CH_{aro}), 128.6 (2 x CH_{aro}), 128.7 (2 x CH_{aro}), 128.8 (CH_{aro}), 130.1 (CH_{aro}), 130.5 (C^q_{aro}), 130.9 (CH_{aro}), 131.0 (C^q_{aro}), 131.3 (CH_{aro}), 132.4 (CH_{aro}), 135.2 (C^q_{aro}), 137.4 (C^q_{aro}), 147.3 (C^q_{aro}), 167.2 (C=O), 171.1 (C=O) ppm. HRMS (+ESI) Calculated for $C_{26}H_{23}N_2O_2$ $[M+H]^+$: 395.1715, found: 395.1763.

(±)-*N*-Benzyl-2,3-dimethoxy-15,15*a*-dihydro-5*H*-benzo[4,5]pyrrolo[3',2':2,3]azepino[2,1-*a*]isoindole-8,14(6*H*,13*H*)-dione (±)-5i(A,B)



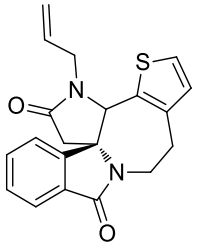
This product was obtained as separable two diastereisomers in 92% yield.

Major diastereoisomer (±)-5iA: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 3/2) = 0.39; mp = 202-204 °C; **IR (ν_{max} / cm^{-1})**: 1682, 1610; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.76-2.84 (m, 1H, CH_2), 3.06-3.23 (m, 3H, CH_2), 3.31-3.40 (m, 2H, CH_2), 3.55 (s, 3H, OCH_3), 3.88 (s, 3H, OCH_3), 4.47-4.58 (m, 1H, CH_2), 4.66 (s, 1H, CH), 5.23 (d, *J* = 14.2 Hz, 1H, CH_2), 5.89 (s, 1H, H_{aro}), 6.70 (s, 1H, H_{aro}), 7.07 (d, *J* = 6.2 Hz, 2H, H_{aro}), 7.30-7.32 (m, 2H, H_{aro}), 7.37-7.44 (m, 2H, H_{aro}), 7.58 (t, *J* = 7.3 Hz, 1H, H_{aro}), 7.66 (d, *J* = 7.2 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 29.5 (CH_2), 36.0 (CH_2), 39.8 (CH_2), 44.9 (CH_2), 55.9 (OCH_3), 55.9 (OCH_3), 64.6 (C^q), 72.1 (CH), 114.1 (CH_{aro}), 115.1 (CH_{aro}), 120.5 (CH_{aro}), 122.1 (C^q_{aro}), 123.8 (CH_{aro}), 127.9 (CH_{aro}), 128.6 (2 x CH_{aro}), 128.7 (2 x CH_{aro}), 128.8 (CH_{aro}), 130.0 (C^q_{aro}), 131.0 (C^q_{aro}), 132.1 (CH_{aro}), 135.3 (C^q_{aro}), 147.0 (C^q_{aro}), 147.5 (C^q_{aro}), 149.6(C^q_{aro}), 167.4 (C=O), 170.9 (C=O) ppm. HRMS (+ESI) Calculated for $C_{28}H_{27}N_2O_4$ $[M+H]^+$: 455.1926, found: 455.1997.

Minor diastereoisomer (±)-5iB: This compound was purified by flash chromatography, R_f (cyclohexane/AcOEt: 3/2) = 0.57; mp = 228-230 °C; **IR (ν_{max} / cm^{-1})**: 1703, 1683; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.48 (d, *J* = 15.0 Hz, 1H, CH_2), 2.99-3.12 (m, 3H, CH_2), 3.35-3.44 (m, 1H, CH_2), 3.57 (d, *J* = 14.5 Hz, 1H, CH_2), 3.85 (s, 3H, OCH_3), 3.92 (s, 3H, OCH_3), 4.52-4.62 (m, 2H, CH+CH_2), 5.11 (d, *J* = 14.5 Hz, 1H, CH_2), 6.53 (s, 1H, H_{aro}), 6.70 (s, 1H, H_{aro}), 7.20 (d, *J* = 7.4 Hz, 2H, H_{aro}), 7.26-7.36 (m, 4H, H_{aro}), 7.52 (t, *J* = 7.4 Hz, 1H, H_{aro}), 7.63 (t, *J* = 7.4 Hz, 1H, H_{aro}), 7.87 (d, *J* = 7.4 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 31.6 (CH_2),

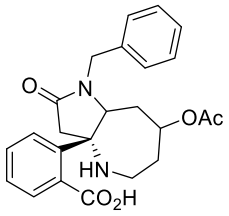
34.9 (CH_2), 41.2 (CH_2), 45.3 (CH_2), 56.0 (OCH_3), 56.1 (OCH_3), 60.2 (CH), 69.4 (C^q), 115.0 (CH_{aro}), 116.9 (CH_{aro}), 120.9 (CH_{aro}), 123.9 (CH_{aro}), 128.0 (CH_{aro}), 128.4 (C^q_{aro}), 128.5(2 x CH_{aro}), 128.8 (2 x CH_{aro}), 129.1 (CH_{aro}), 132.3 (CH_{aro}), 132.4 (C^q_{aro}), 132.5 (C^q_{aro}), 135.3 (C^q_{aro}), 145.7 (C^q_{aro}), 147.3 (C^q_{aro}), 148.6 (C^q_{aro}), 167.8 (C=O), 171.3 (C=O) ppm. HRMS (+ESI) Calculated for $C_{28}H_{27}N_2O_4$ $[M+H]^+$: 455.1926, found: 455.2005.

(±)-*N*-Allyl-14,14*a*-dihydro-4*H*-pyrrolo[3',2':2,3]thieno[2',3':4,5]azepino[2,1-*a*]isoindole-7,13(5*H*,12*H*)-dione (±)-5jA



The reaction was carried out on pure α-acetoxylactams **(±)-8hA** or **(±)-8hB**. In both cases the same compound **(±)-5jA** was isolated as sole diastereoisomer in 90% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 3/2) = 0.51; mp = 186-188 °C; **IR (ν_{max} / cm^{-1})**: 1701, 1675; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.89 (d, *J* = 18.1 Hz, 1H, CH_2), 3.02-3.20 (m, 2H, CH_2), 3.21 (d, *J* = 18.1 Hz, 1H, CH_2), 3.38-3.54 (m, 2H, CH_2), 4.30-4.33 (dd, *J* = 5.1 and 14.9 Hz, 1H, CH_2=), 4.49-4.59 (m, 1H, CH_2=), 4.99 (d, *J* = 17.0 Hz, 1H, H_{aro}), 5.16-5.21(m, 2H, CH+CH_2), 5.63-5.76 (m, 1H, CH=), 6.50 (d, *J* = 5.1Hz, 1H, CH_2), 6.96 (d, *J* = 5.1Hz, 1H, H_{aro}), 7.46 (t, *J* = 7.3 Hz, 1H, H_{aro}), 7.54 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.63 (t, *J* = 7.3 Hz, 1H, H_{aro}) 7.71 (d, *J* = 7.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 25.7 (CH_2), 35.3 (CH_2), 41.2 (CH_2), 43.7 (CH_2), 65.6 (CH), 66.6 (C^q), 119.1 (CH_2=), 120.8 (CH_{aro}), 122.8 (CH_{aro}), 123.8 (CH_{aro}), 128.9 (C^q_{aro}), 129.0 (CH_{aro}), 130.4 (CH_{aro}), 131.0 (CH_{aro}), 131.4 (C^q_{aro}), 132.4 (CH=), 139.0 (C^q_{aro}), 145.9 (C^q_{aro}), 167.3 (C=O), 170.8 (C=O) ppm. HRMS (+ESI) Calculated for $C_{20}H_{19}N_2O_2S$ $[M+H]^+$: 351.1123, found: 351.1186.

(±)-*N*-Benzyl-7-acetoxy-2-oxodecahydropyrrolo[3,2-*b*]azepin-3-yl)benzoic acid (±)-12k



The reaction was carried out on pure α-acetoxylactams **(±)-8kA or (±)-8kB**. In both cases the same compound **(±)-12k**

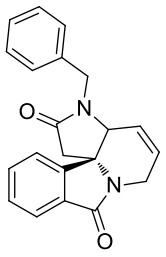
was isolated as sole diastereoisomer in 86% yield: This compound was isolated by flash chromatography, R_f (CH_2Cl_2/EtOH : 4.5/0.5) = 0.35; mp = 189-191 °C; **IR (ν_{max} / cm^{-1})**: 3262, 1679, 1658; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 0.92-1.00 (m, 1H, CH_2), 1.65-1.88 (m, 7H, CH3+CH_2), 2.72-2.84 (m, 2H, CH_2), 3.24 (s, 1H, NH), 3.80-3.87 (m, 2H, CH_2), 3.99 (d, *J* = 14.6 Hz, 1H, CH_2), 4.57 (d, *J* = 14.9 Hz, 1H, CH_2), 5.26 (d, *J* = 15.0 Hz, 1H, CH_2), 7.28-7.42

(m, 6H, H_{aro}), 7.45-7.51 (m, 2H, H_{aro}), 7.59-7.67 (m, 2H, H_{aro}+OH), 7.86 (d, *J* = 7.2 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 23.1 (CH_3), 32.5 (CH_2), 33.9 (CH_2), 37.0 (CH_2), 41.2 (CH_2), 43.8 (CH_2), 44.7 (C^q), 62.1 (CH), 66.6 (CH), 122.6 (CH_{aro}), 122.9 (CH_{aro}), 128.0 (CH_{aro}), 128.3 (2 x CH_{aro}), 129.2 (2 x CH_{aro}), 129.3 (CH_{aro}), 131.1 (C^q_{aro}), 133.1 (CH_{aro}), 135.9 (C^q_{aro}), 148.2 (C^q_{aro}), 166.3 (C=O), 169.1 (C=O), 172.2 (C=O) ppm. HRMS (+ESI) Calculated for $C_{25}H_{21}N_2O_2$ $[M+H]^+$: 423.1875, found: 423.1836.

VI. Procedure for the preparation of compound (±)-4f.

To a solution of α-acetoxylactam **(±)-8** (pure form or mixture of two diastereoisomers 0.7 mmol) in 2 mL of acetonitrile was added 1eq of TMSOTf then the reaction mixture was stirred at room temperature for 24 h. The reaction mixture was neutralized with a saturated aqueous solution of $NaHCO_3$ (10 mL) and extracted twice with dichloromethane (20 mL). The organic layer was dried over $MgSO_4$ and evaporated *in vacuo* to give a white product which was purified by flash column chromatography on silica gel column using cyclohexane-ethyl acetate to give compounds **(±)-4f** or **(±)-4k** as following.

(±)-*N*-Benzyl-3,3a-dihydropyrrolo[3',2':2,3]pyrido[2,1-*a*]isoindole-2,8(1*H*,6*H*)-dione (±)-4f



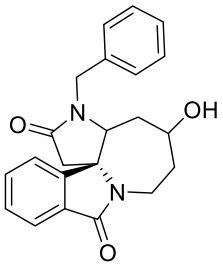
The reaction was carried out on pure α-acetoxylactams **(±)-8fA** or **(±)-8fB**. In both cases the same cyclization product **(±)-4f** was isolated as sole diastereoisomer in 64% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 2/3) = 0.54; mp = 88-90 °C; **IR (ν_{max} / cm^{-1})**: 1681, 1653; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 2.34 (d, *J* = 16.4 Hz, 1H, CH_2), 2.95 (d, *J* = 16.4 Hz, 1H, CH_2), 3.58-3.66 (m, 1H, CH_2), 3.72-3.73 (m, 1H, CH), 4.16 (d, *J* = 14.5 Hz, 1H, CH_2), 4.65-4.73 (m, 1H, CH_2), 4.96 (d, *J* = 14.5 Hz, 1H, CH_2), 5.69 (dd, *J* = 2.2 and 10.3 Hz, 1H, CH=), 5.91 (m, 1H, CH=), 7.12 (d, *J* = 8.1 Hz, 1H, H_{aro}), 7.23-7.30 (m, 5H, H_{aro}), 7.34 (t, *J* = 6.8 Hz, 1H, H_{aro}), 7.74 (d, *J* = 8.5 Hz, 1H, H_{aro}) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C δ 36.6 (CH_2), 36.9 (CH_2), 45.1 (CH_2), 58.7 (CH), 60.8 (C^q), 120.1 (CH_{aro}), 121.7 (CH_{aro}), 123.7(CH=), 125.5 (CH_{aro}), 128.2 (CH=), 128.9 (2 x CH_{aro}), 129.0 (2 x CH_{aro}), 129.1 (CH_{aro}), 130.0 (C^q_{aro}), 132.4 (CH_{aro}), 135.7 (C^q_{aro}), 148.9 (C^q_{aro}), 166.3 (C=O), 170.4 (C=O) ppm. HRMS (+ESI) Calculated for $C_{21}H_{19}N_2O_2$ $[M+H]^+$: 331.1402, found: 331.1453.

VII. Procedure for the preparation of compound (±)-4k.

A solution of α-acetoxylactam **(±)-8** in 2 mL of TFA was stirred at room temperature for 24 h. The reaction mixture was evaporated *in vacuo*, neutralized with a saturated aqueous solution of $NaHCO_3$ (10 mL) and extracted twice with dichloromethane (20 mL). The organic layer was

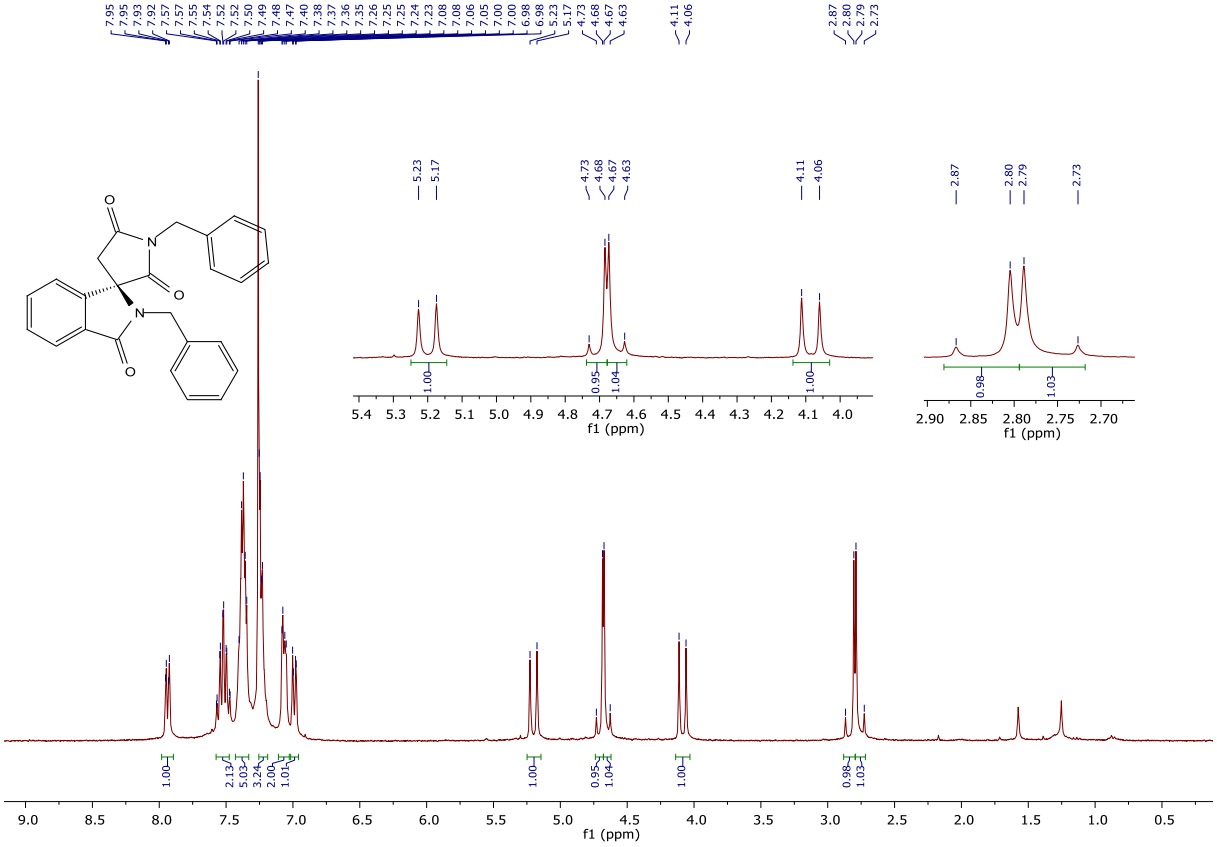
dried over $MgSO_4$ and evaporated *in vacuo* to give a white product which was purified by flash column chromatography on silica gel column using cyclohexane and ethyl acetate to give compound **(±)-4k**.

(±)-3-Benzyl-5-hydroxy-3,3a,4,5,6,7-hexahydro-9*H*-pyrrolo[3',2':2,3]azepino[2,1-*a*]isoindole-2,9(1*H*)-dione (±)-4k.

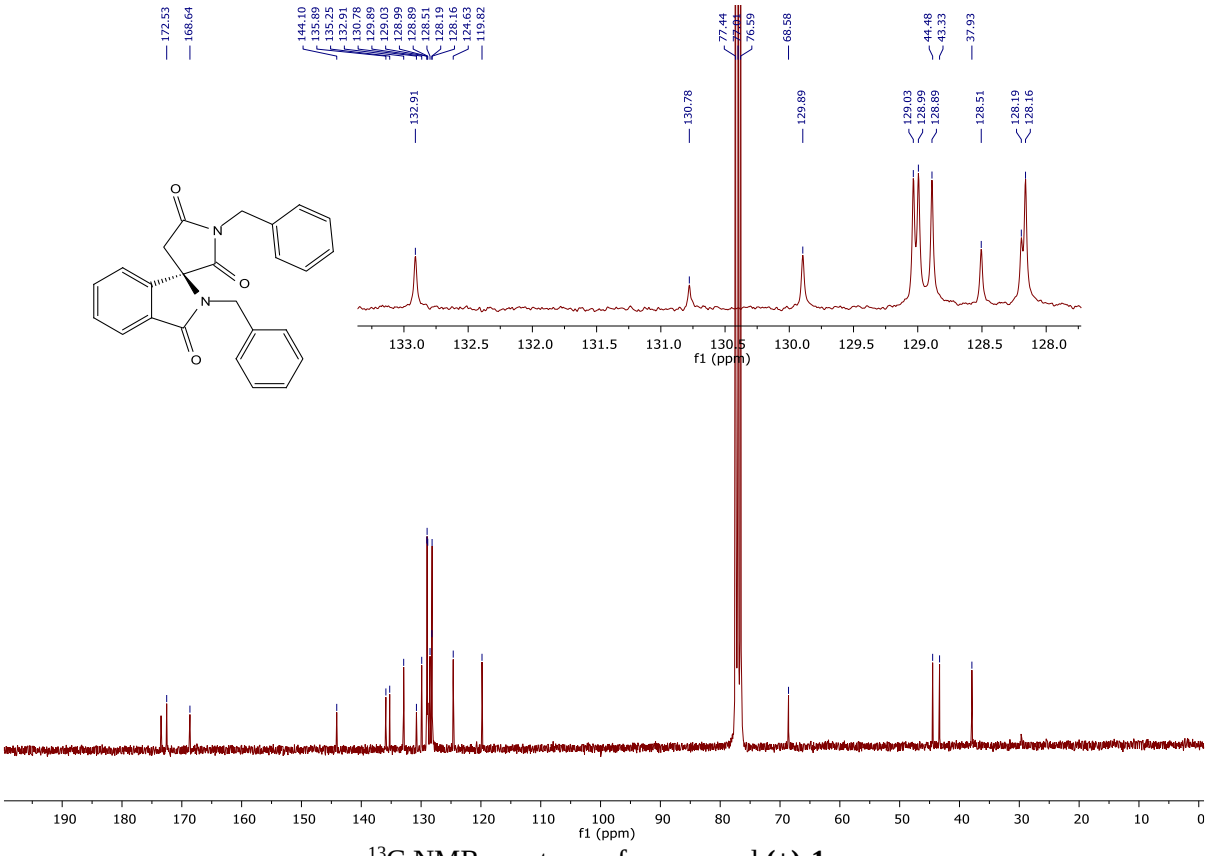


The reaction was carried out on pure α-acetoxylactams (±)-**8kA** or (±)-**8kB**. In both cases the same compound **(±)-4k** was isolated as sole diastereoisomer in 65% yield: This compound was isolated by flash chromatography, R_f (Cyclohexane/AcOEt : 3/2) = 0.34; mp = 202-204 °C; **IR (ν_{max} / cm^{-1})**: 3402, 1704, 1680; **^{1}H NMR (300 MHz, $CDCl_3$)**: δ_H 1.37-1.46 (m, 1H, CH_2), 1.83-1.90 (m, 2H, CH_2), 2.11-2.18 (m, 2H, CH_2), 2.78-2.87 (m, 2H, CH_2), 3.06 (d, *J* = 18.7 Hz, 1H, CH_2), 3.83-3.89 (m, 1H, CH_2), 3.96-4.01 (m, 2H, CH_2), 4.22-4.29 (m, 1H, CH_2), 5.40 (d, *J* = 14.7 Hz, 1H, CH_2), 7.15 (d, *J* = 7.5 Hz, 1H, H_{aro}), 7.28-7.58 (m, 7H, H_{aro}), 7.80 (d, *J* = 7.5 Hz, 1H, H_{aro}), ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ_C 35.1 (CH_2), 35.4 (CH_2), 37.8 (CH_2), 41.5 (CH_2), 45.3 (CH_2), 61.8 (C^q), 66.4 (CH), 66.7 (CH), 121.0 (CH_{aro}), 123.7 (CH_{aro}), 128.2 (2 x CH_{aro}), 128.3 (CH_{aro}), 129.0 (3 x CH_{aro}), 130.9 (C^q_{aro}), 132.8 (CH_{aro}), 135.1 (C^q_{aro}), 148.0 (C^q_{aro}), 167.1 (C=O), 171.9 (C=O) ppm. HRMS (+ESI) Calculated for $C_{22}H_{22}N_2O_3$ $[M+H]^+$: 363.1664, found: 363.1724.

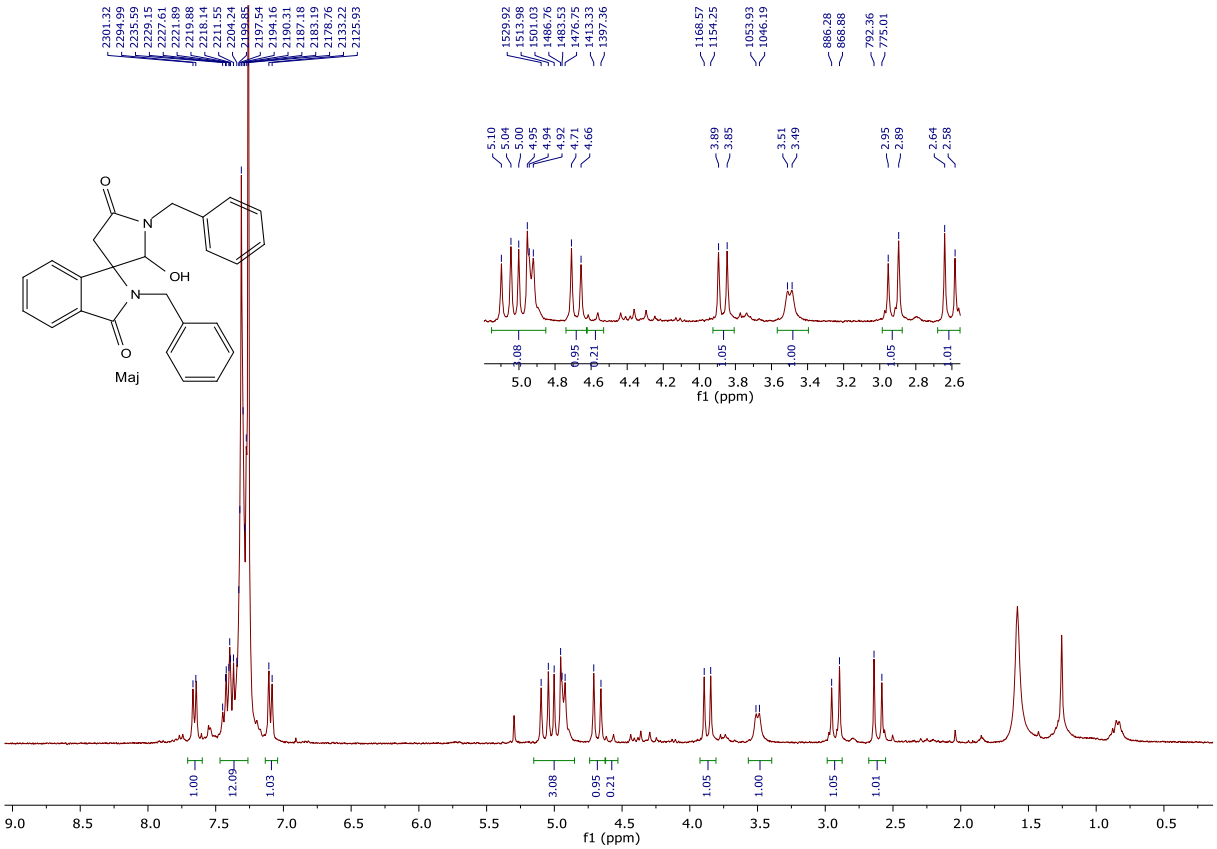
VIII. Copies of ^{1}H and ^{13}C NMR spectra of all new compounds



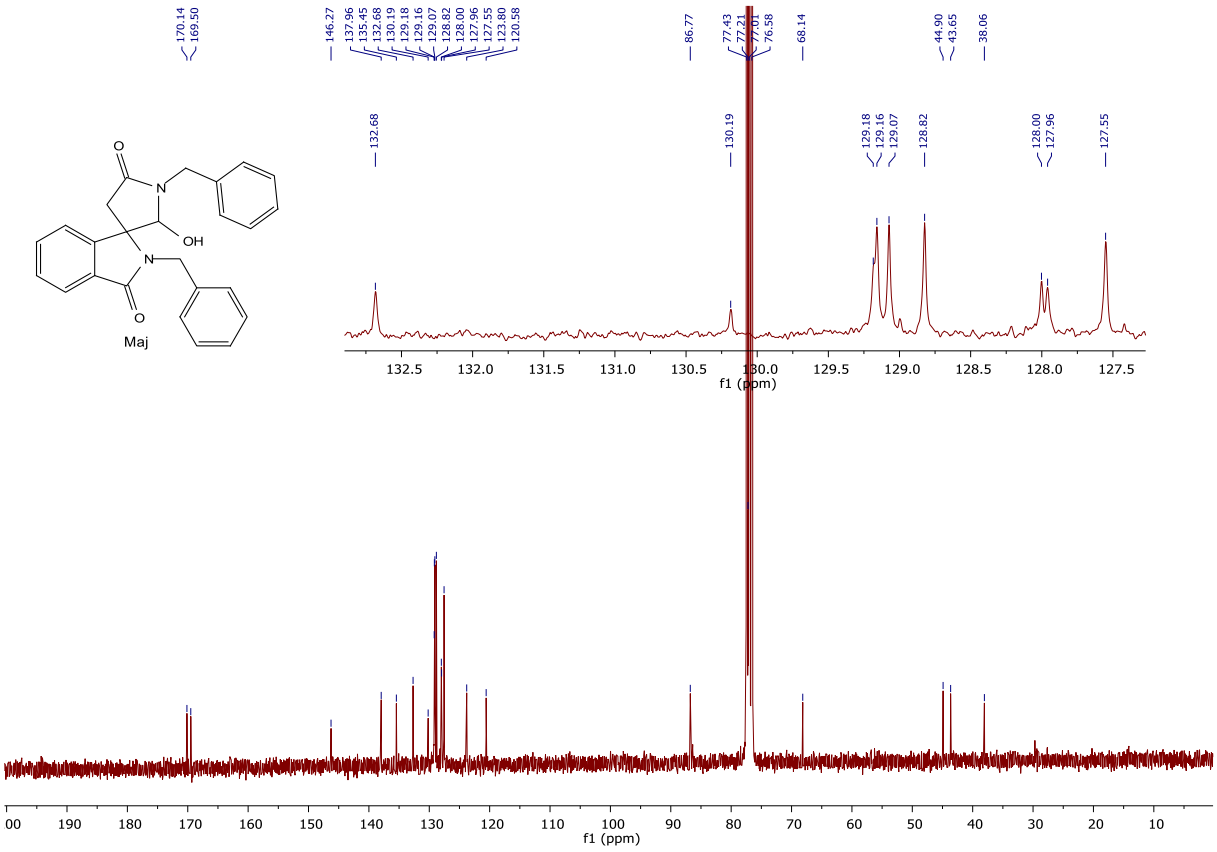
^{1}H NMR spectrum of compound **(±)-1a**



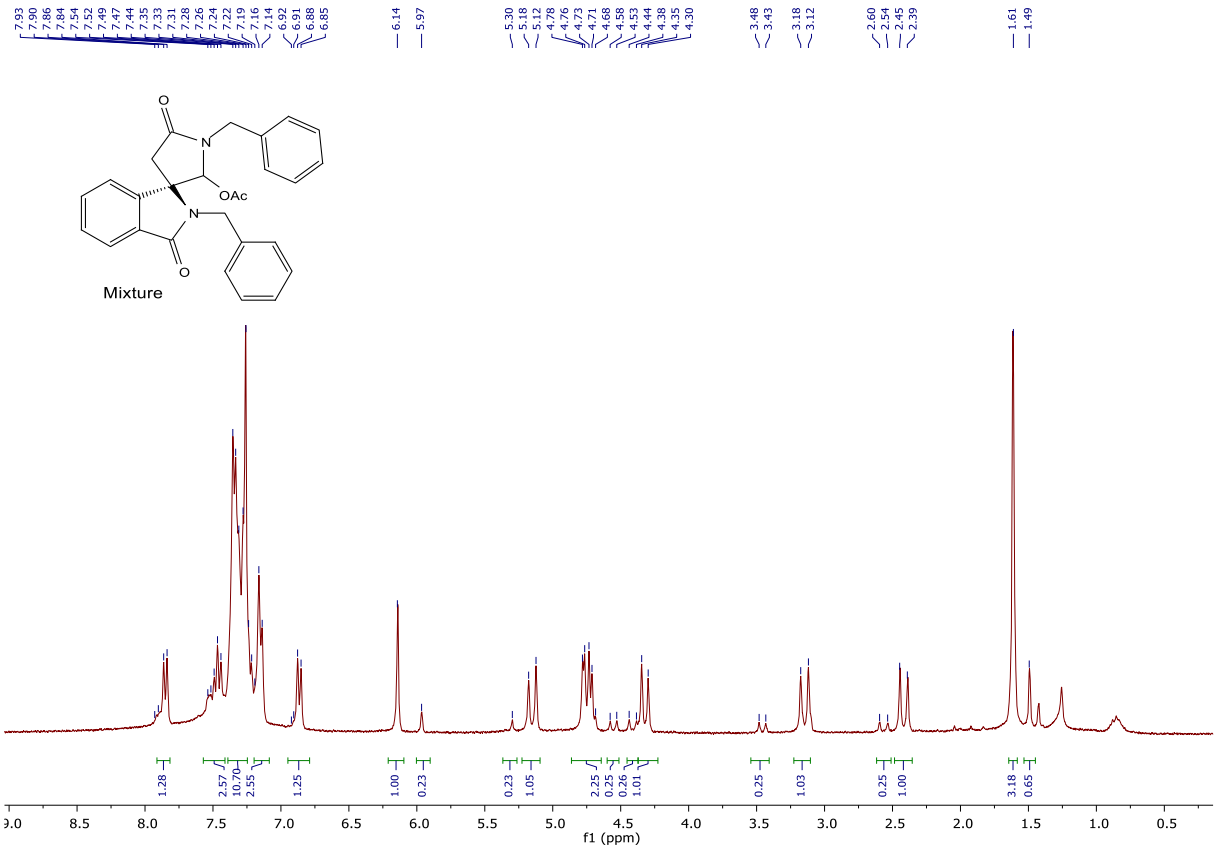
^{13}C NMR spectrum of compound **(±)-1a**



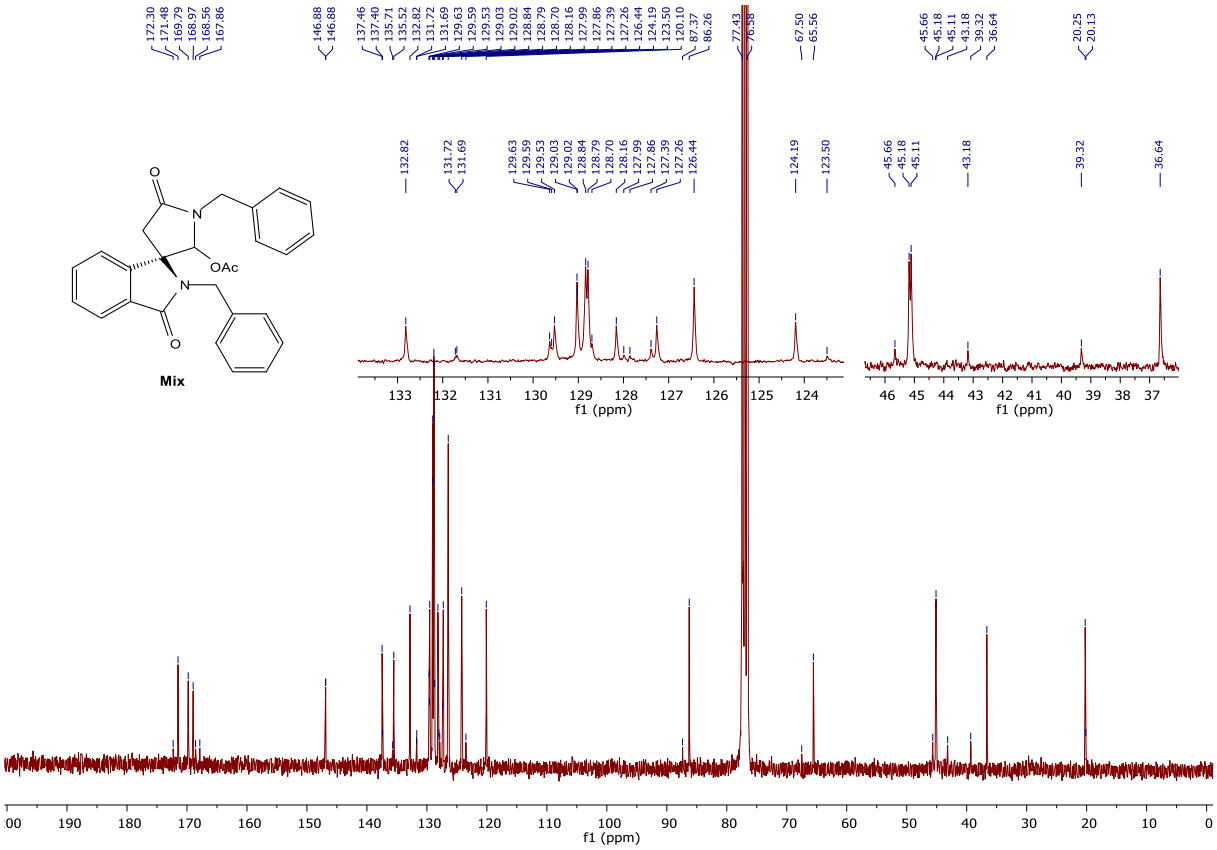
^{1}H NMR spectrum of compound (±)-11aA



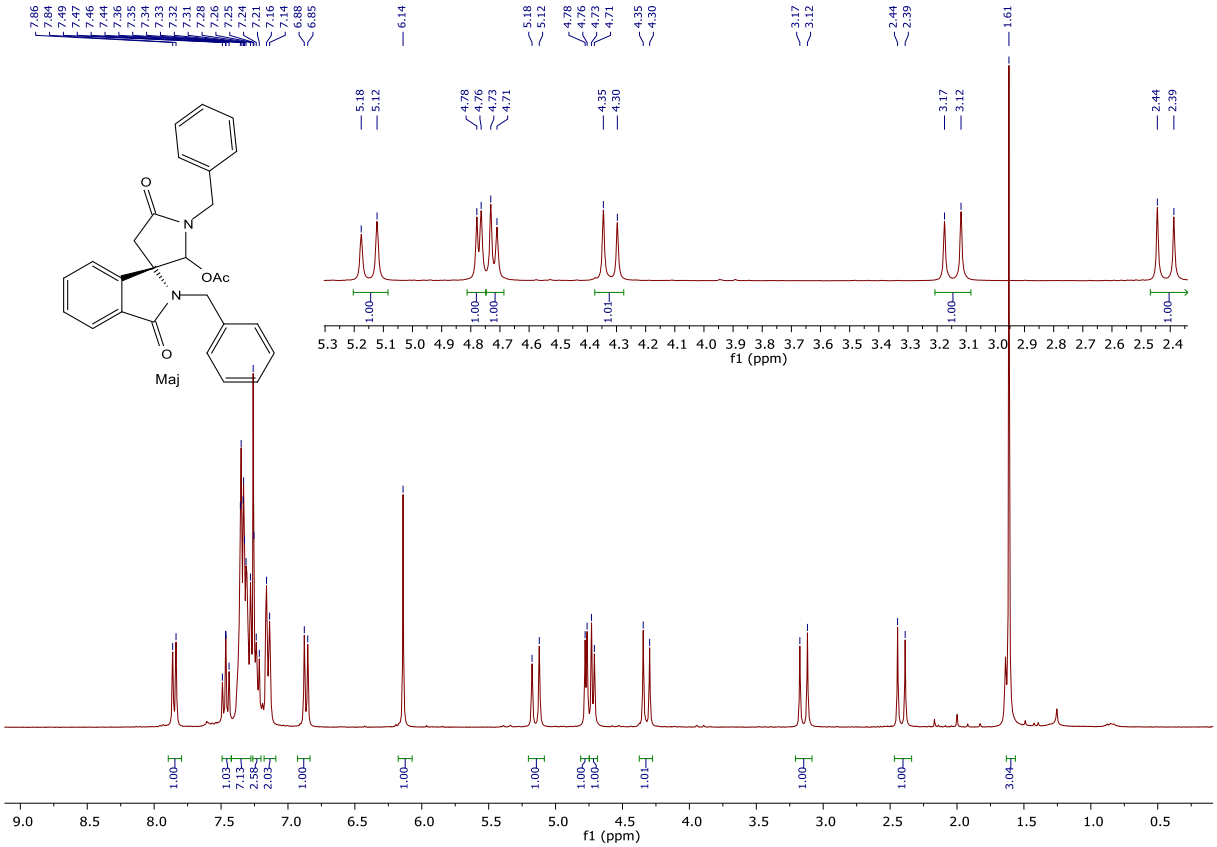
^{13}C NMR spectrum of compound (±)-11Aa



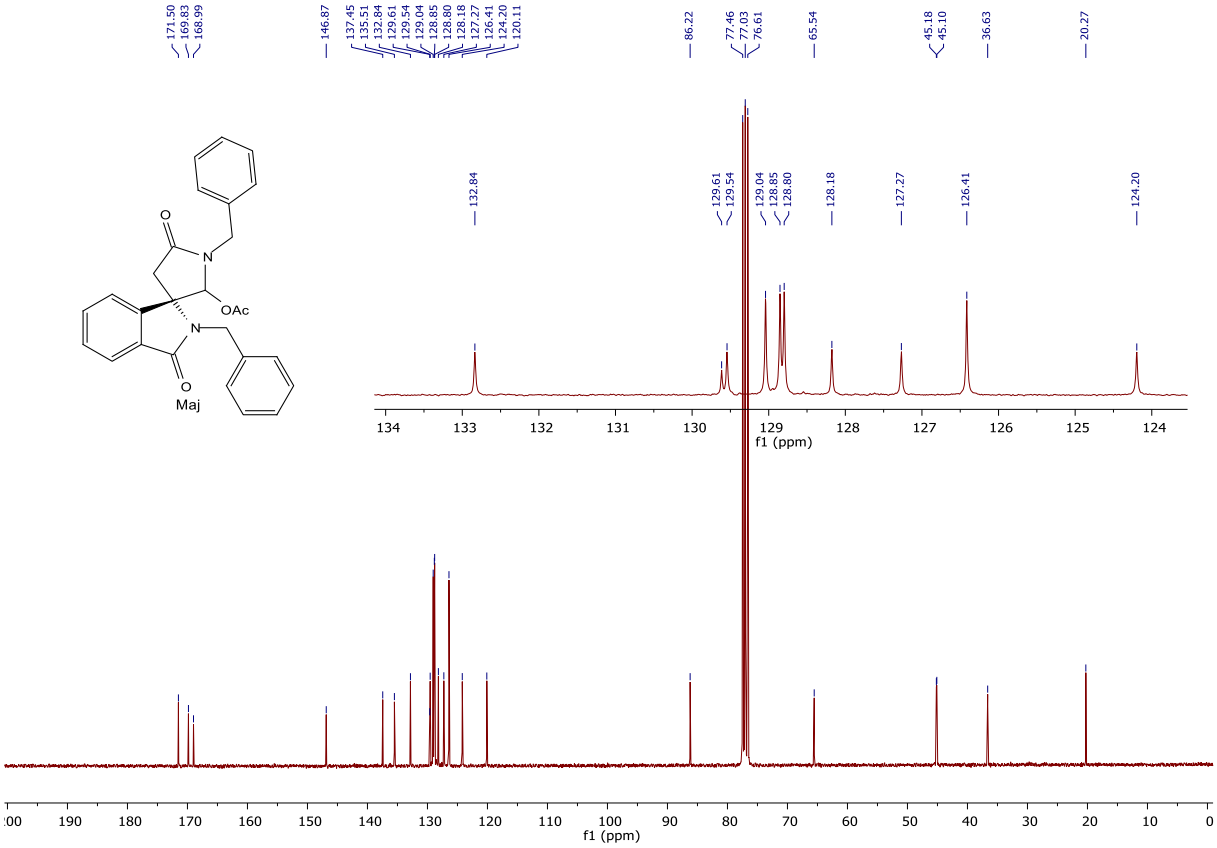
^{1}H NMR spectrum of compound **(±)-8a(A,B)**



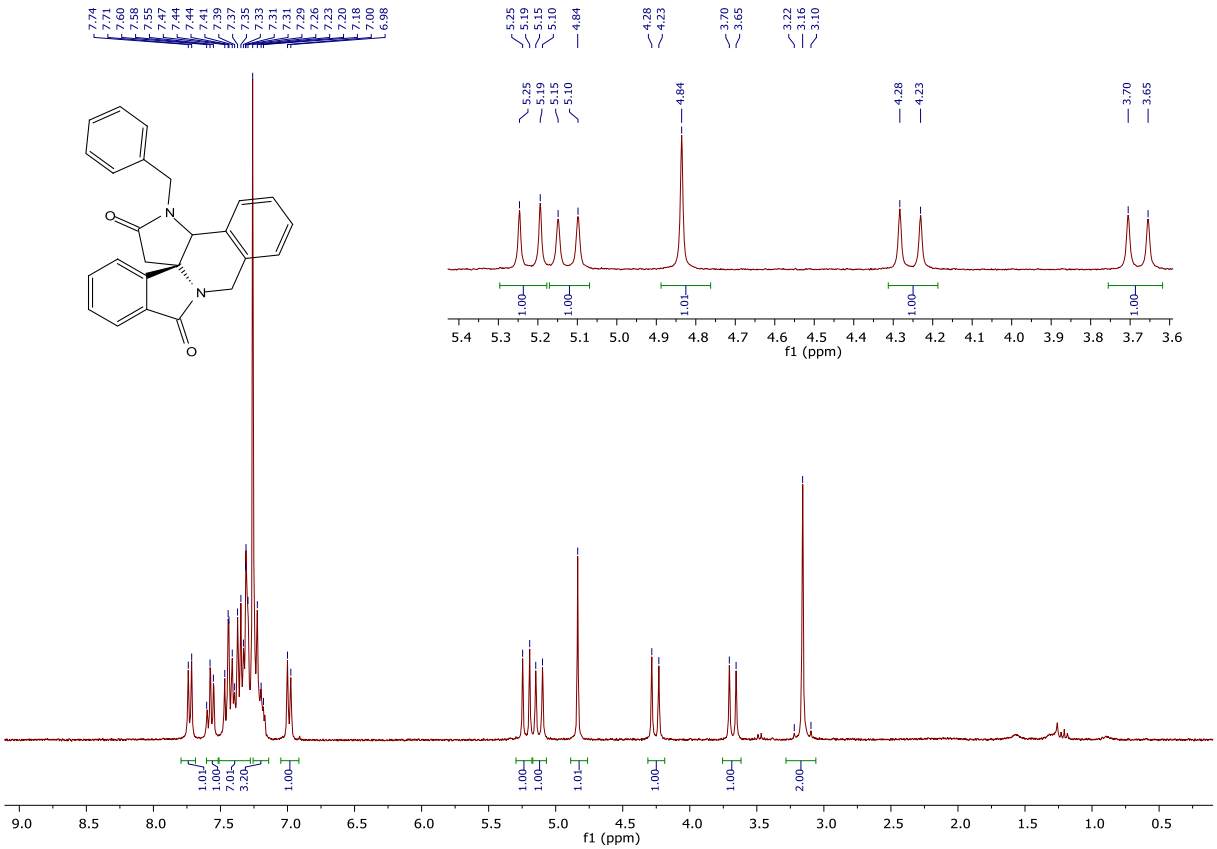
^{13}C NMR spectrum of compound **(±)-8a(A,B)**



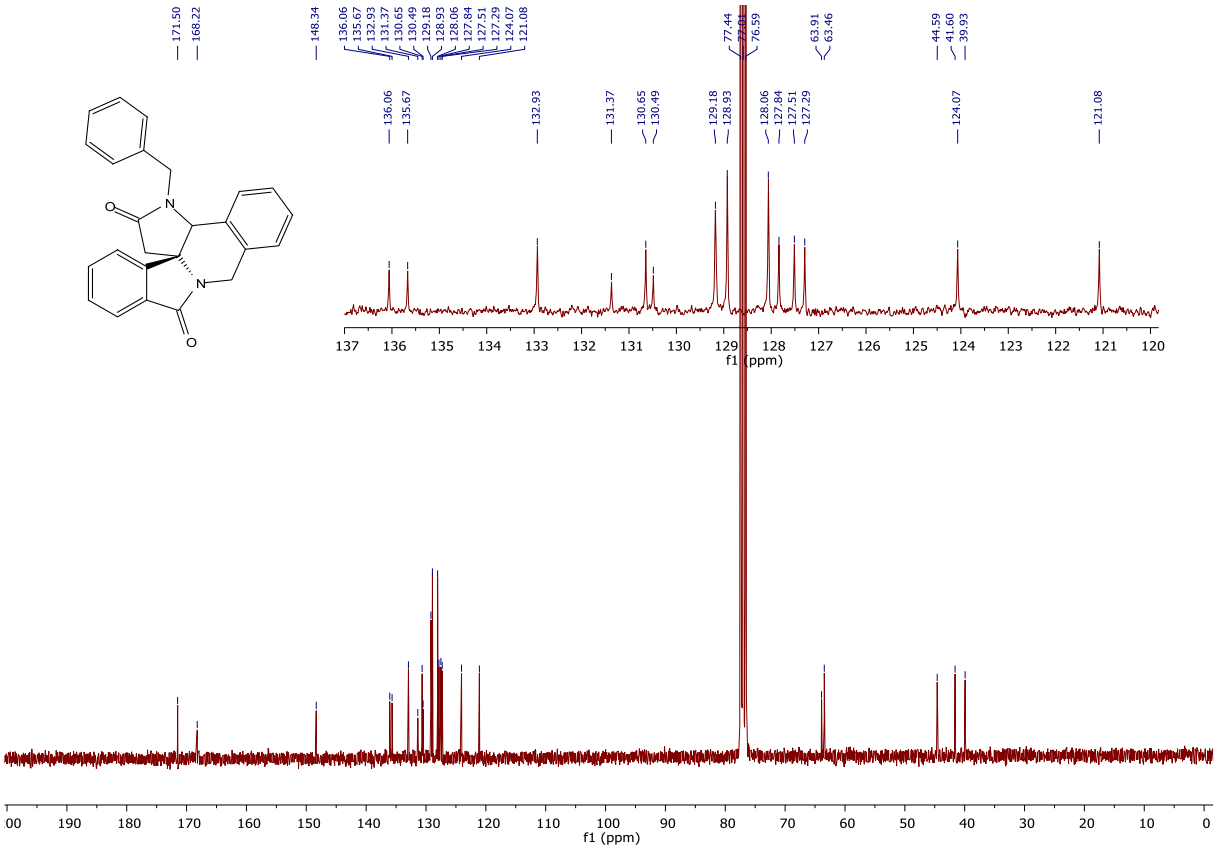
^{1}H NMR spectrum of compound **(±)-8aA**



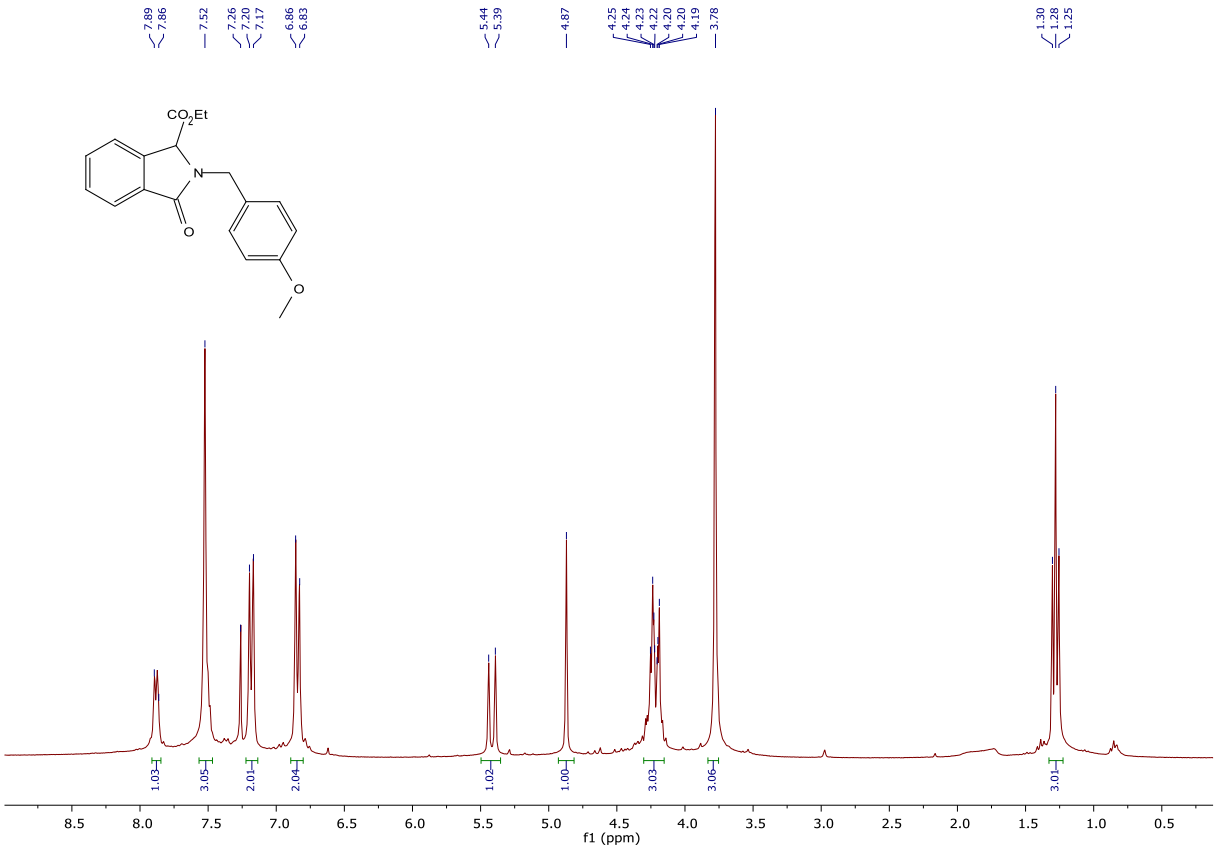
^{13}C NMR spectrum of compound **(±)-8aA**



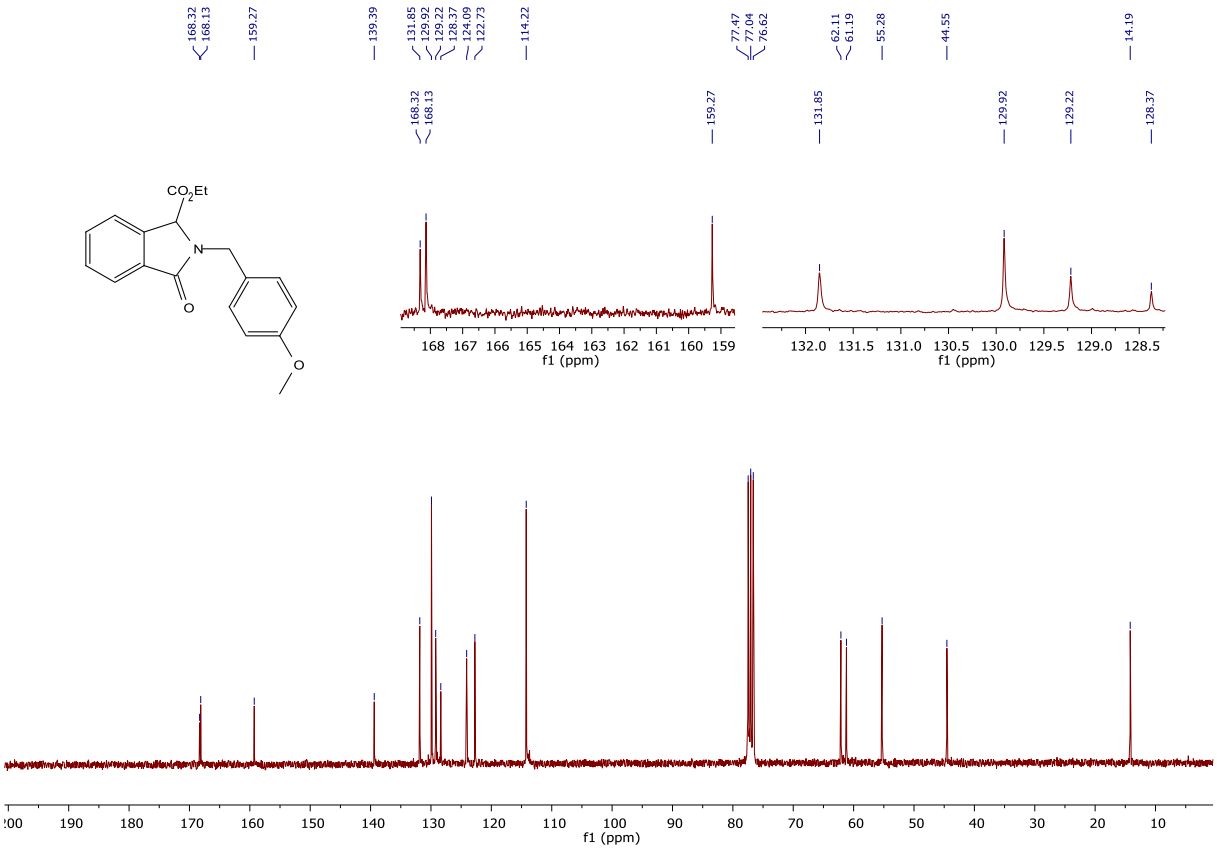
^{1}H NMR spectrum of compound **(±)-5aA**



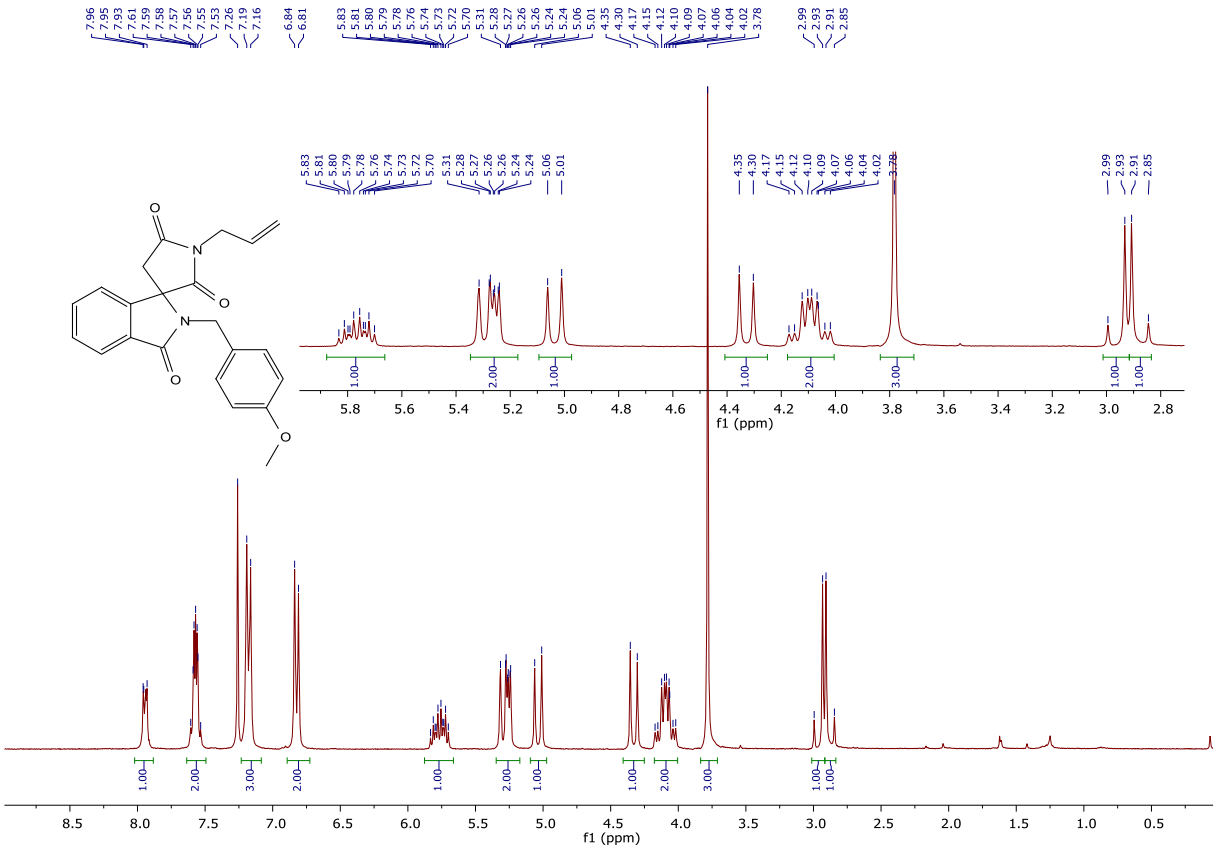
^{13}C NMR spectrum of compound **(±)-5Aa**



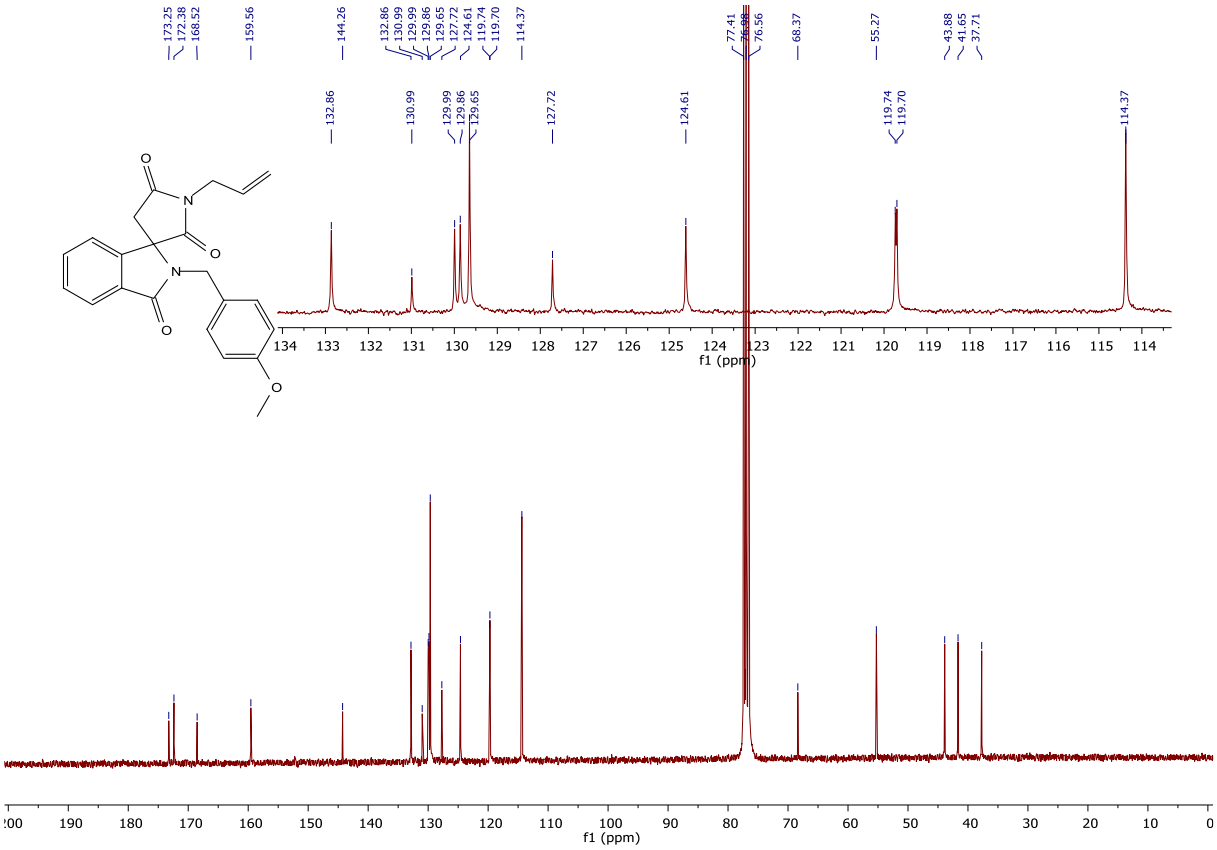
^{1}H NMR spectrum of compound **(±)-7b**



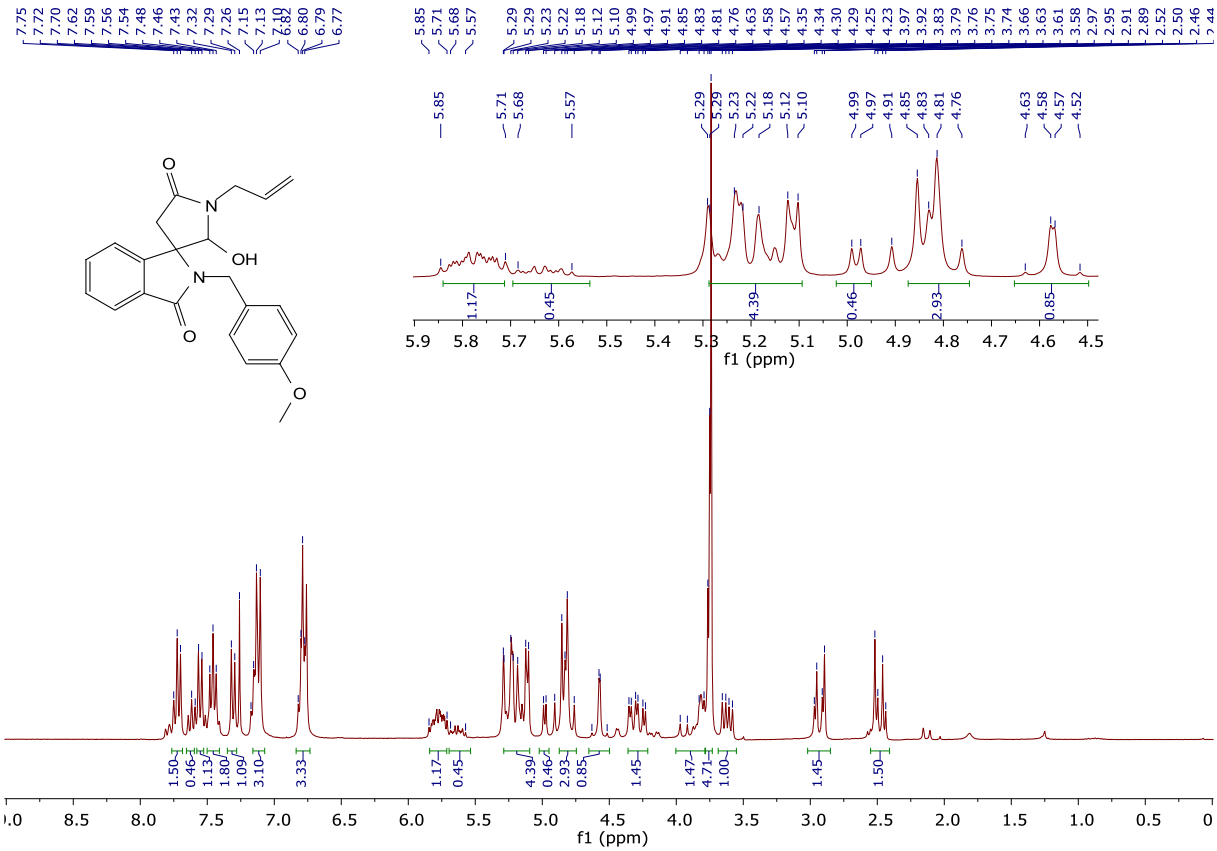
^{13}C NMR spectrum of compound **(±)-7b**



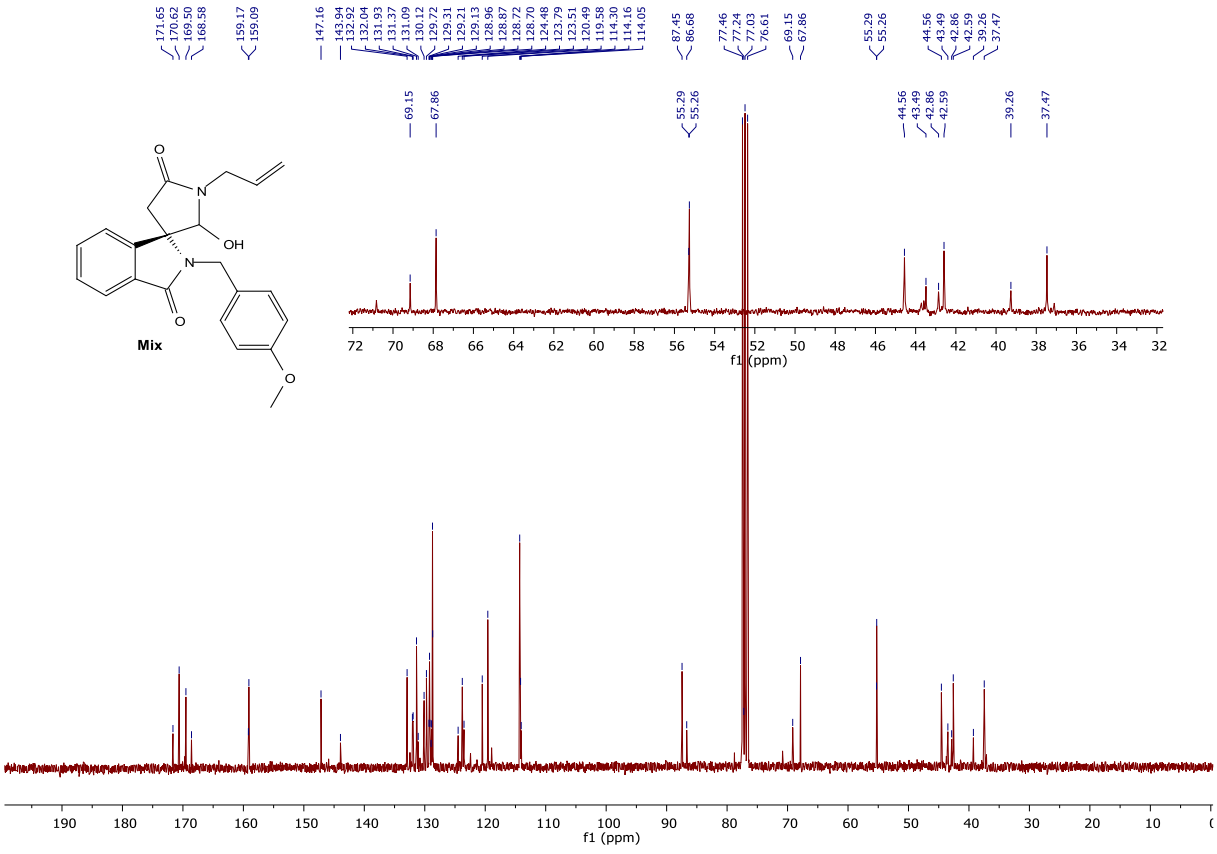
^{1}H NMR spectrum of compound **(±)-1b**



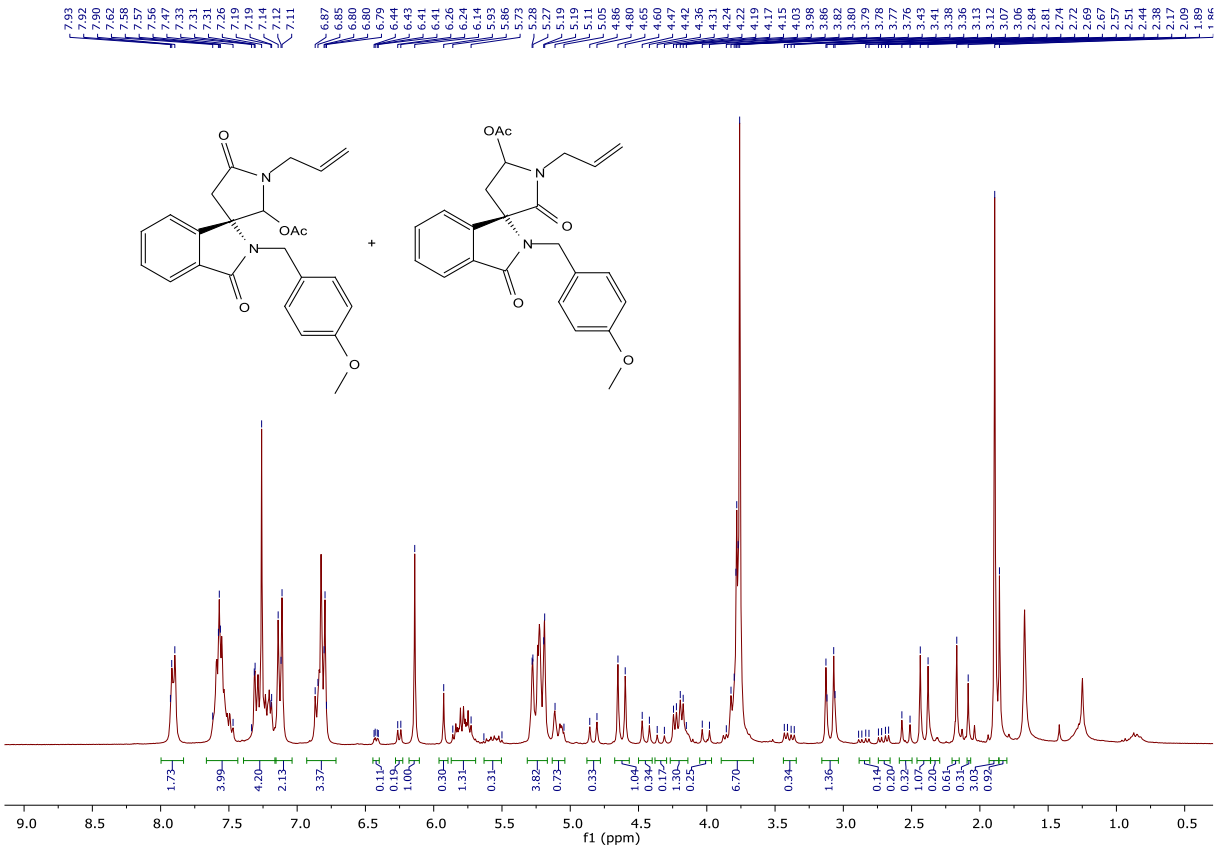
^{13}C NMR spectrum of compound **(±)-1b**



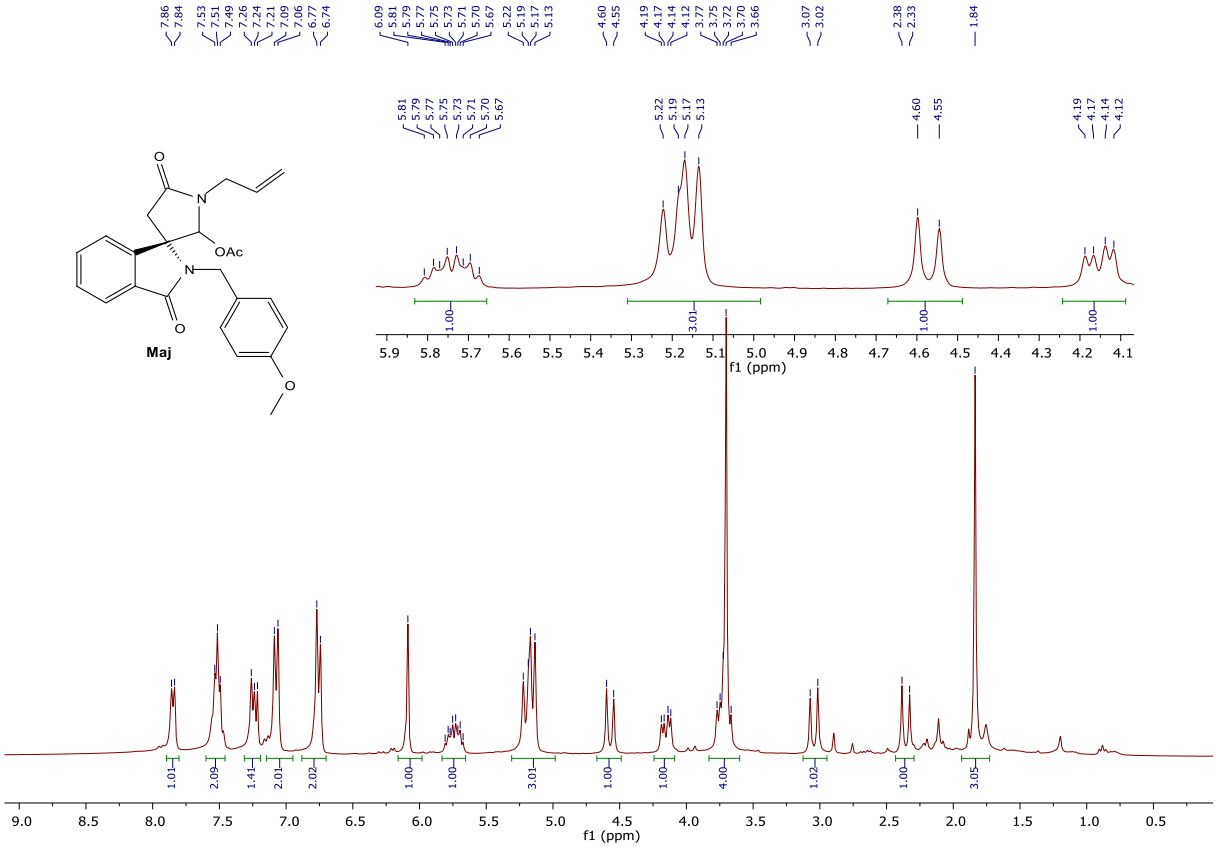
^{1}H NMR spectrum of compound**(±)-11b(A,B)**



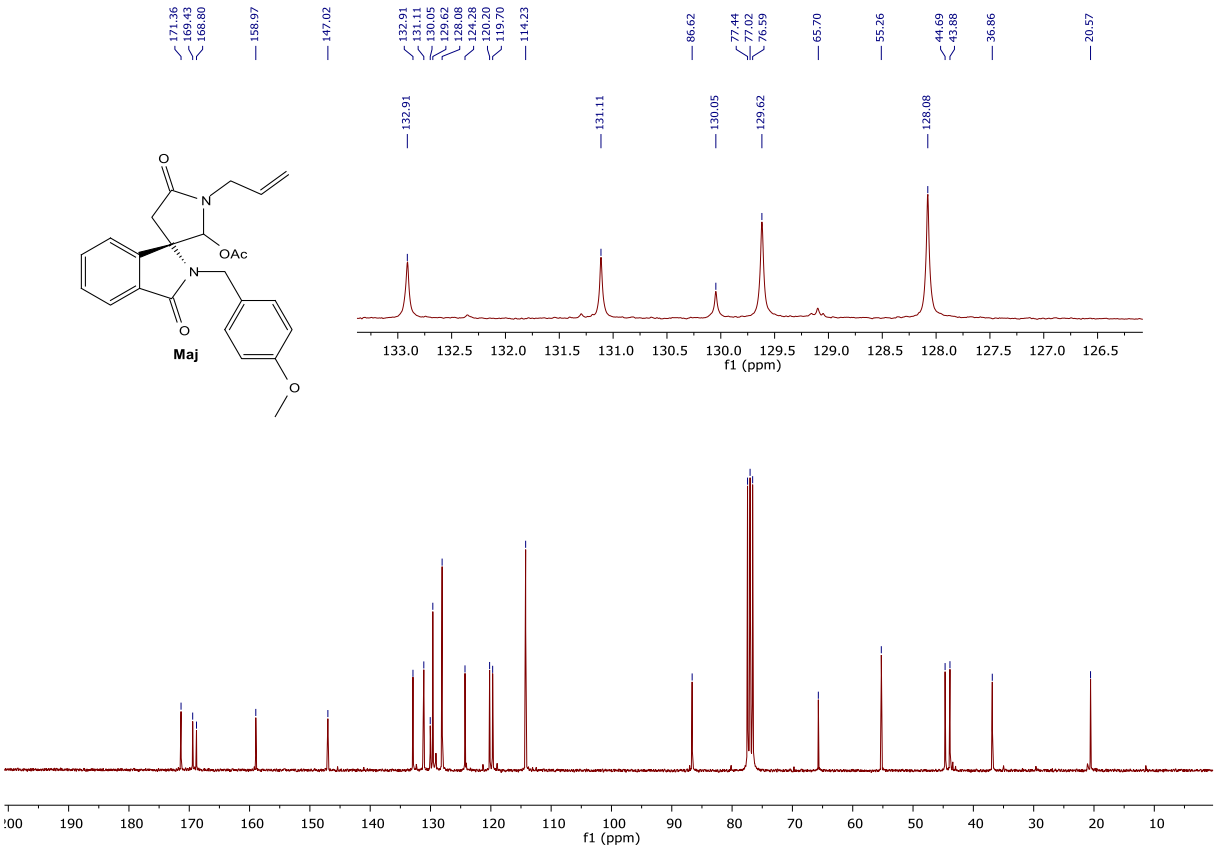
^{13}C NMR spectrum of compound **(±)-11b(A,B)**



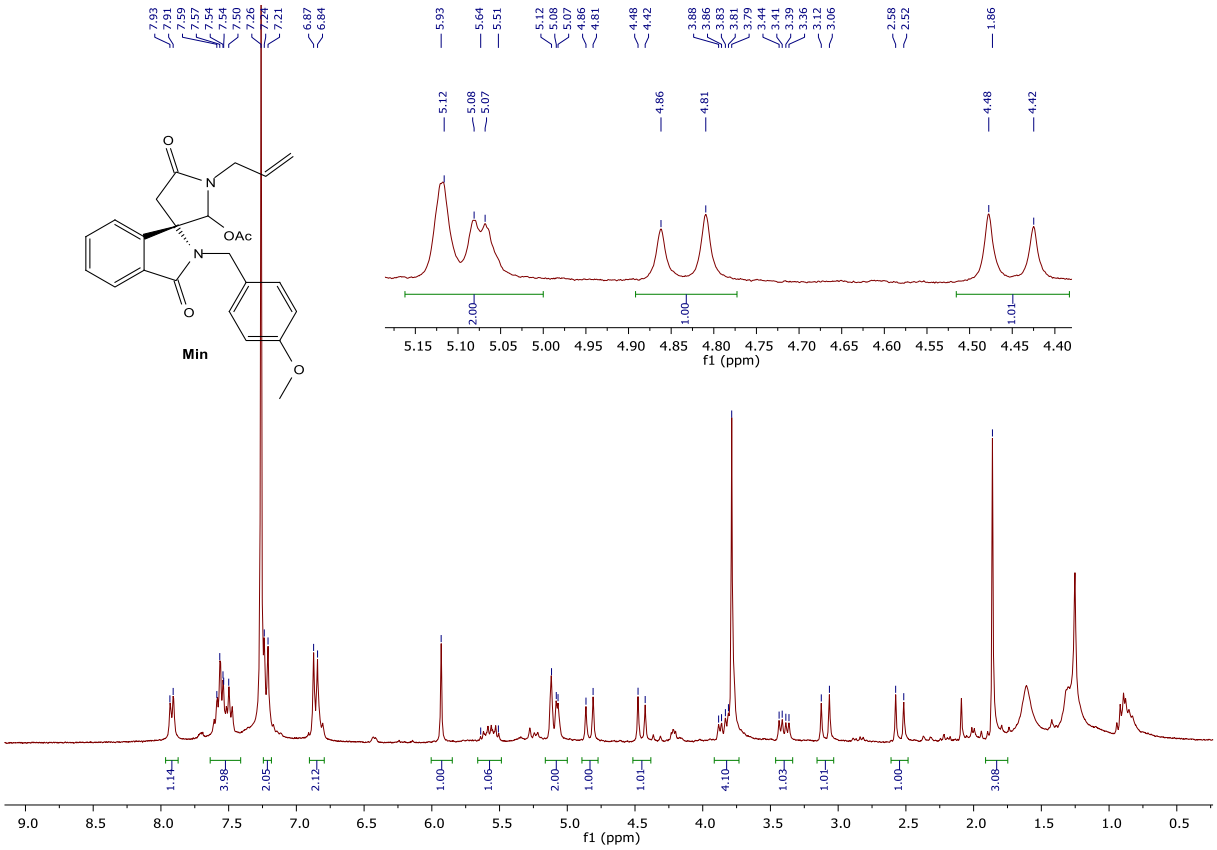
^{1}H NMR spectrum of compound **(±)-8b(A,B)** and **(±)-9b(,A,B)**



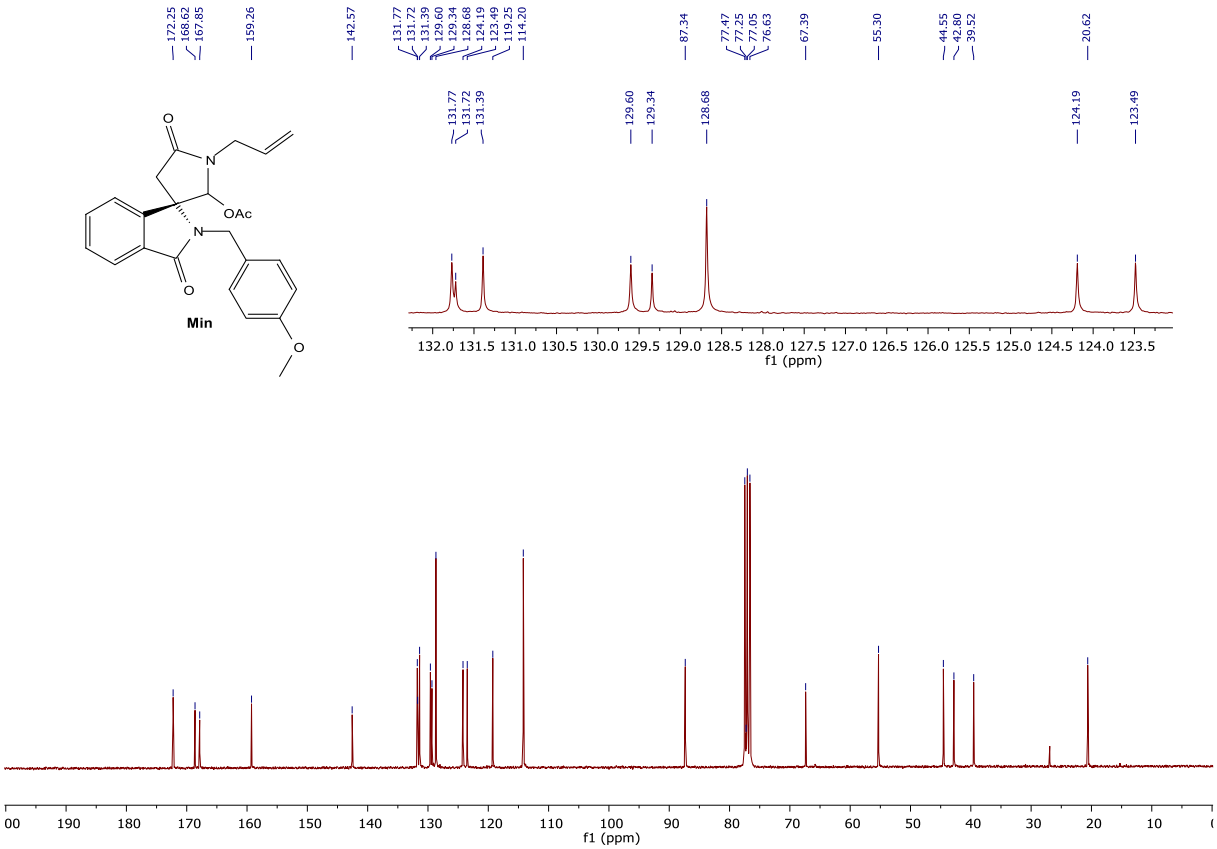
^{1}H NMR spectrum of compound **(±)-8bA**



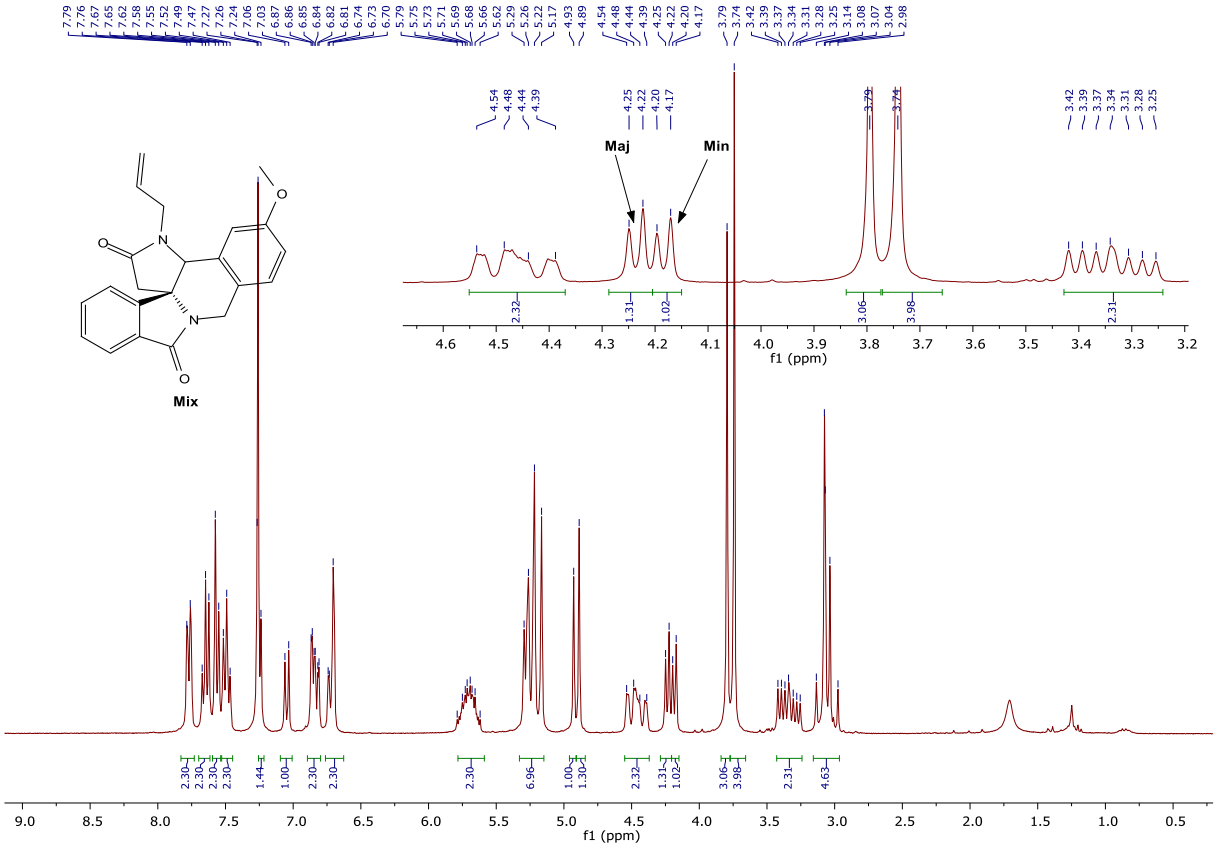
^{13}C NMR spectrum of compound **(±)-8bA**



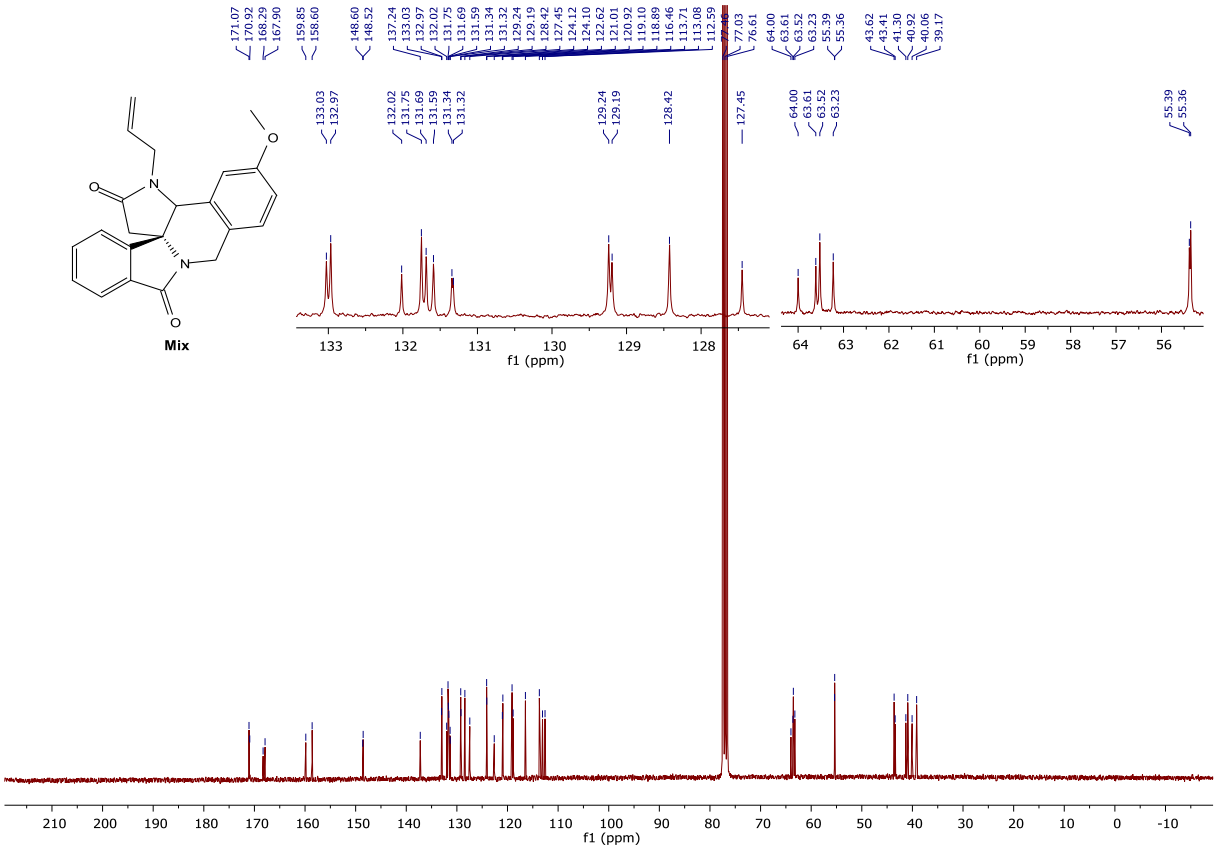
^{1}H NMR spectrum of compound **(±)-8bB**



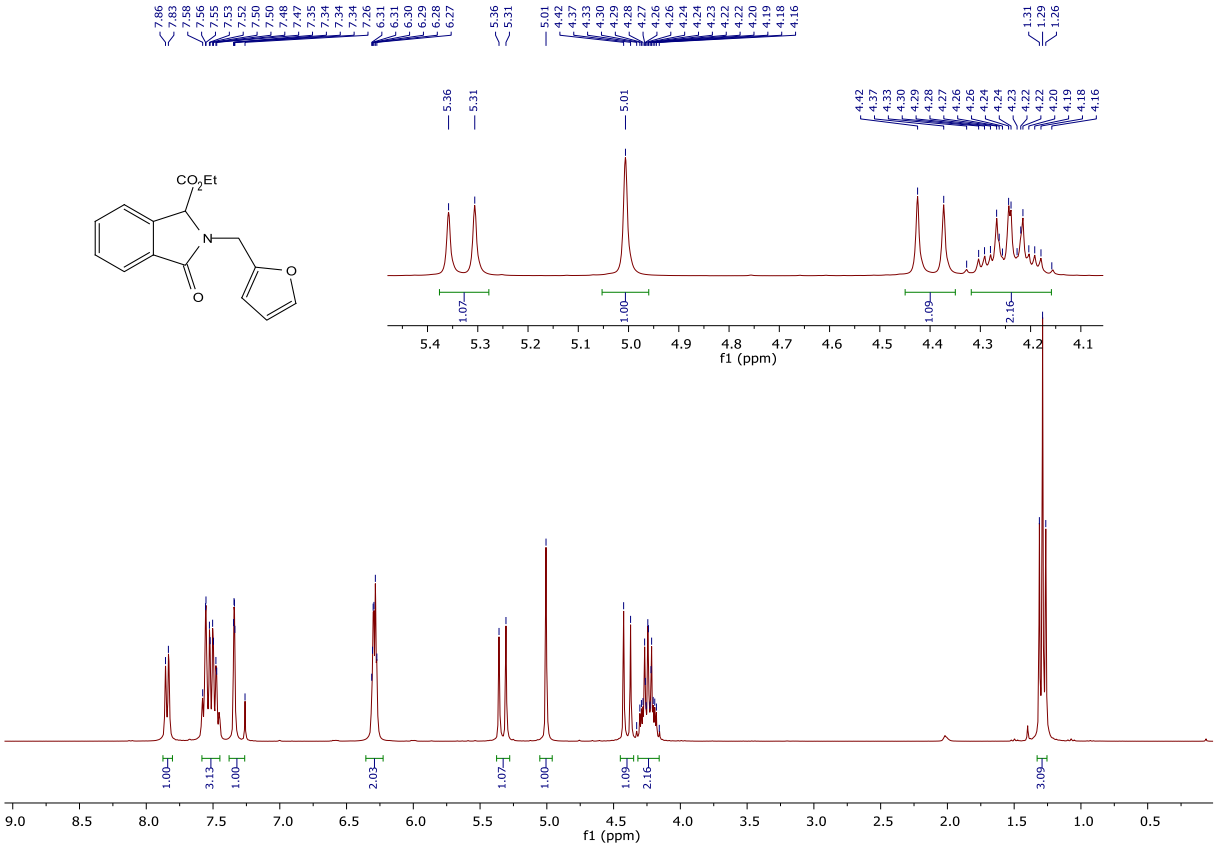
^{13}C NMR spectrum of compound **(±)-8bB**



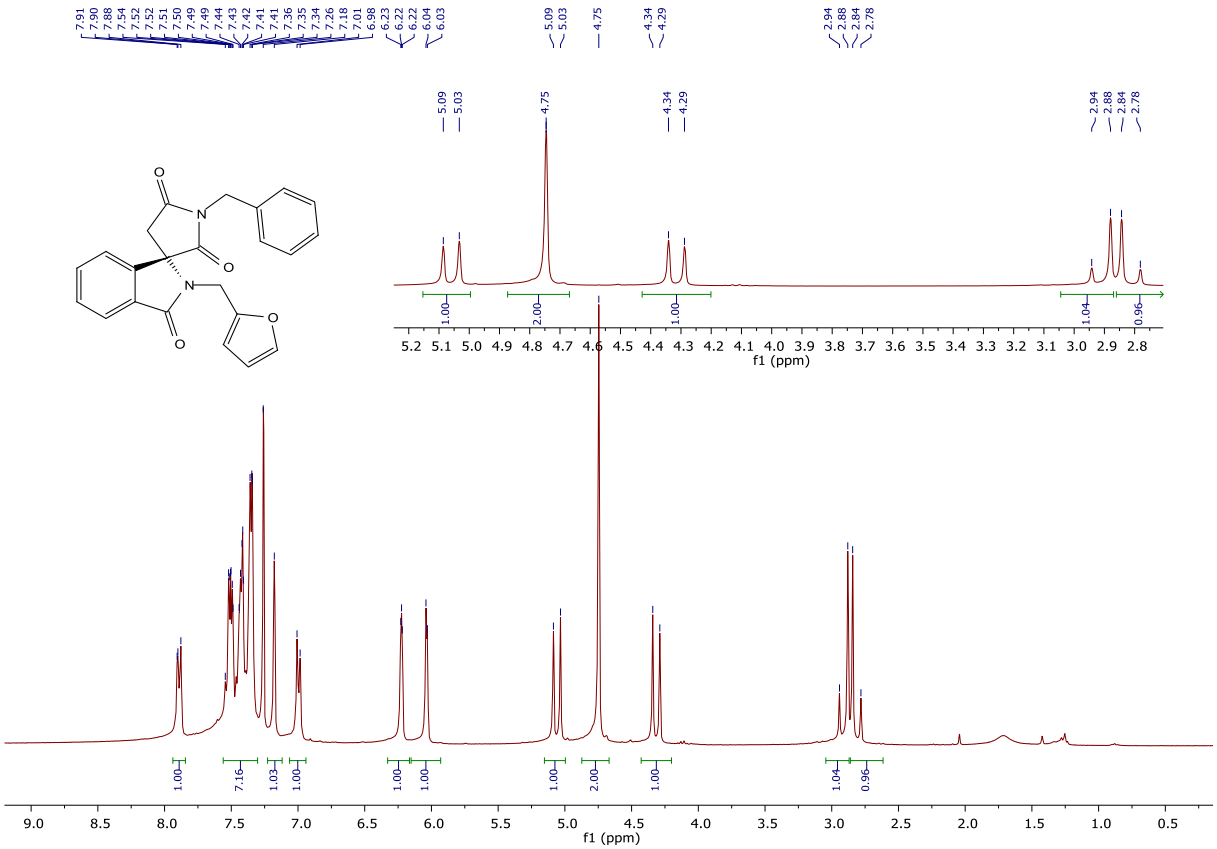
^{1}H NMR spectrum of compound **(±)-5b(A,B)**



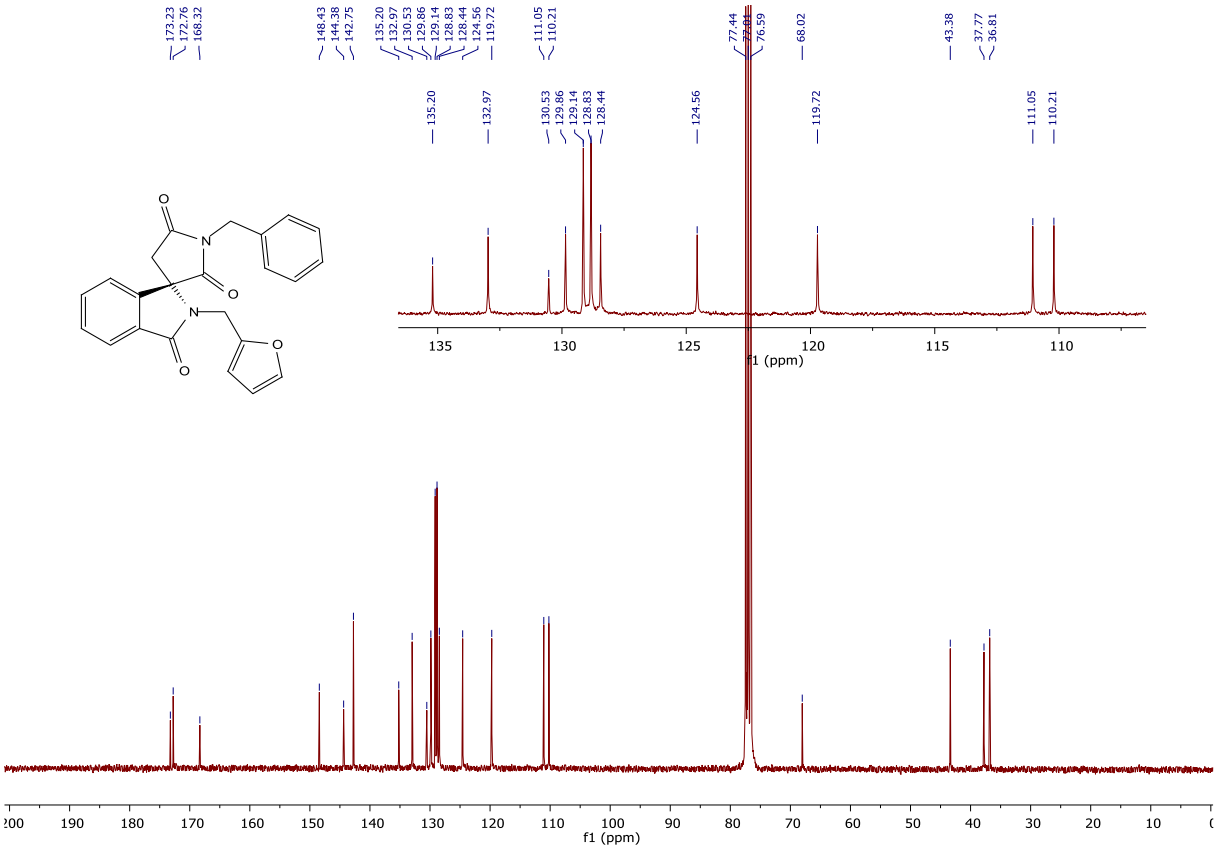
^{13}C NMR spectrum of compound **(±)-5b(A,B)**



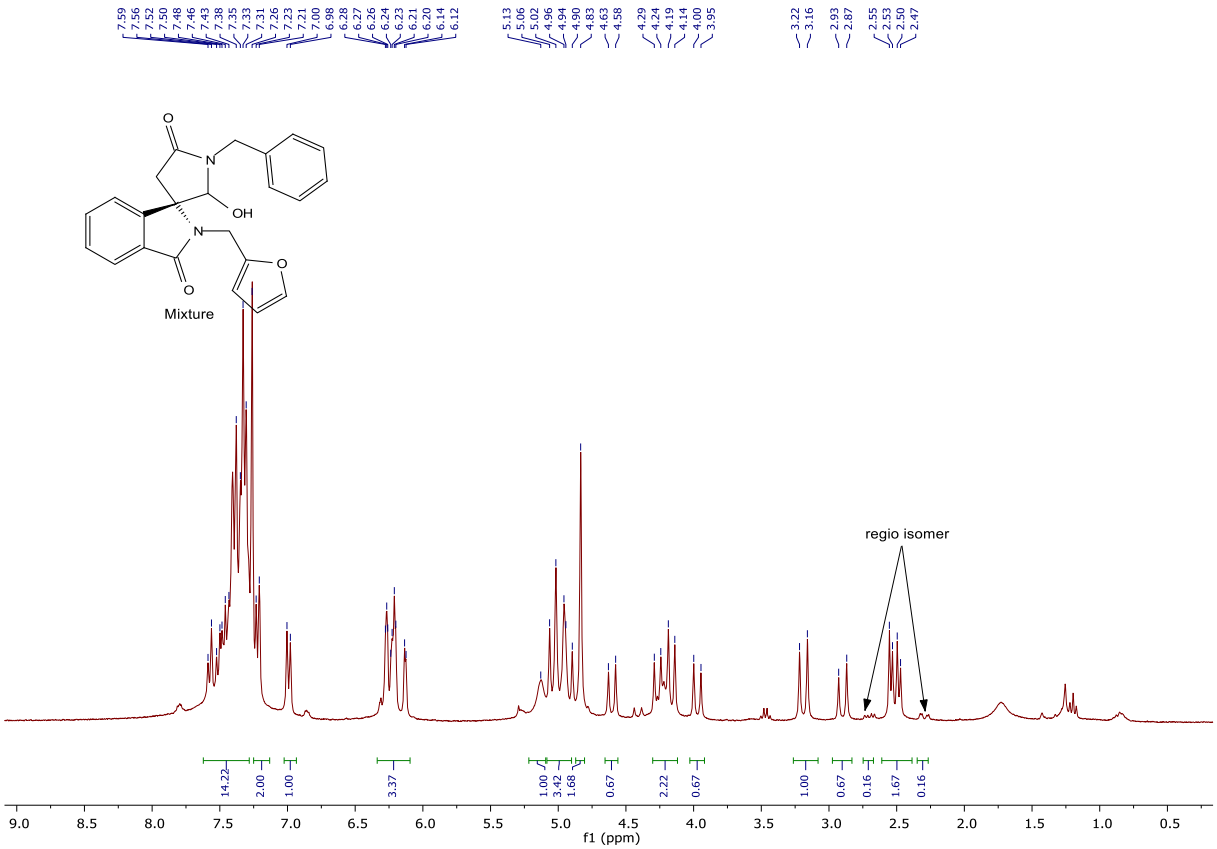
^{1}H NMR spectrum of compound **(±)-7c**



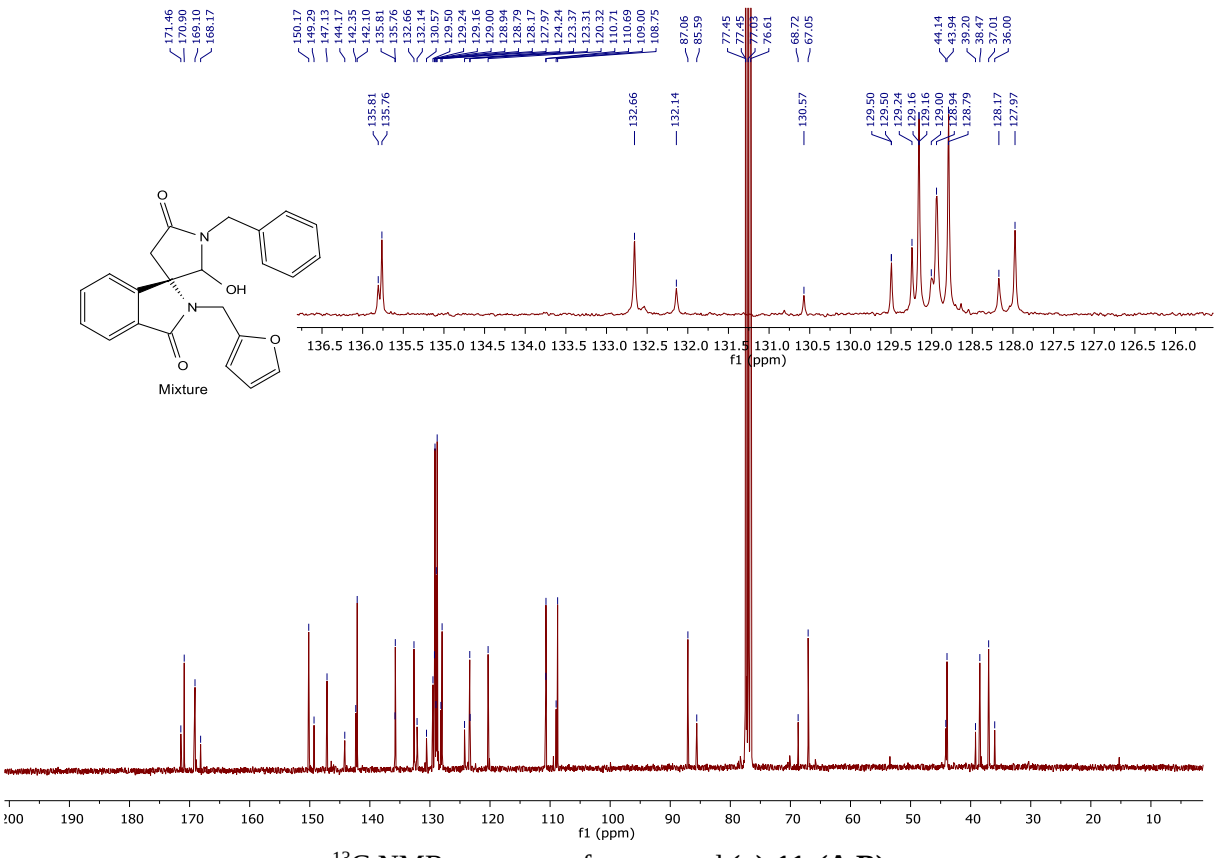
^{1}H NMR spectrum of compound **(±)-1c**



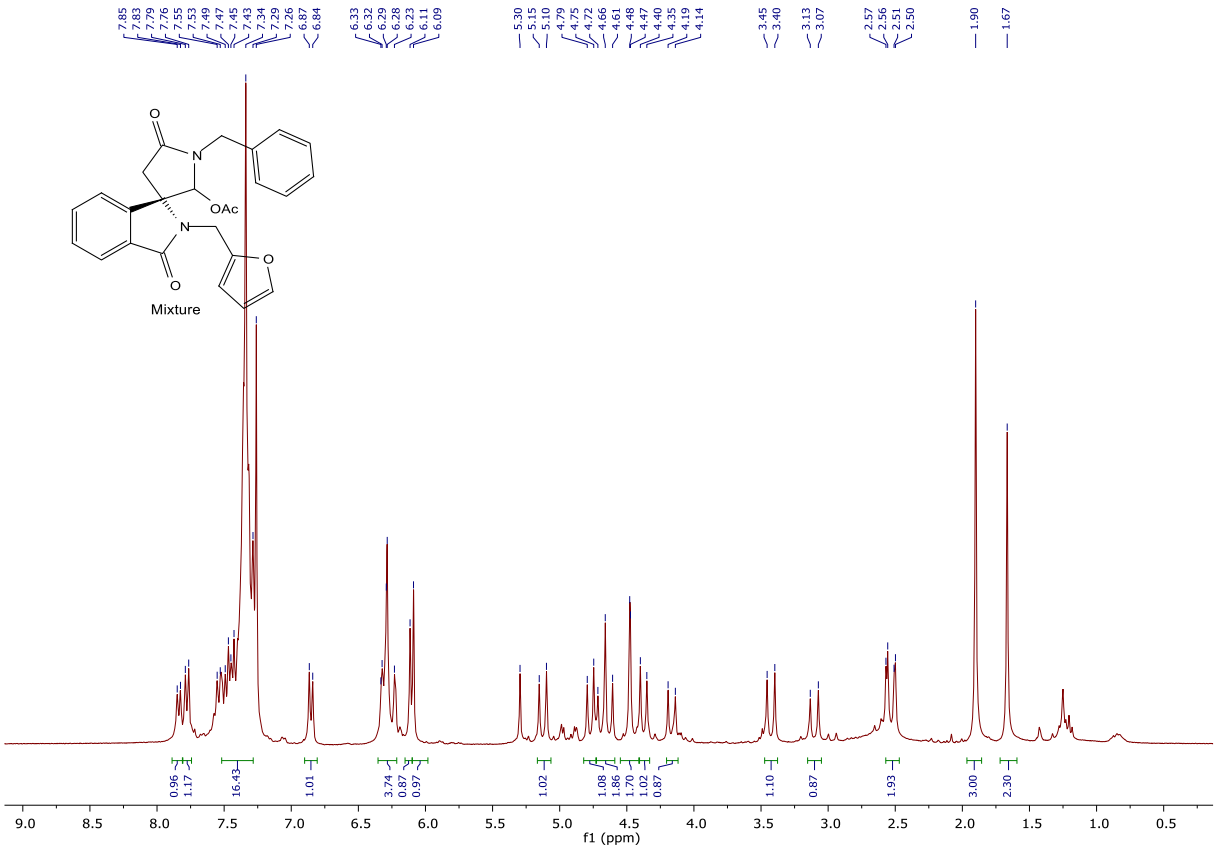
^{13}C NMR spectrum of compound **(±)-1c**



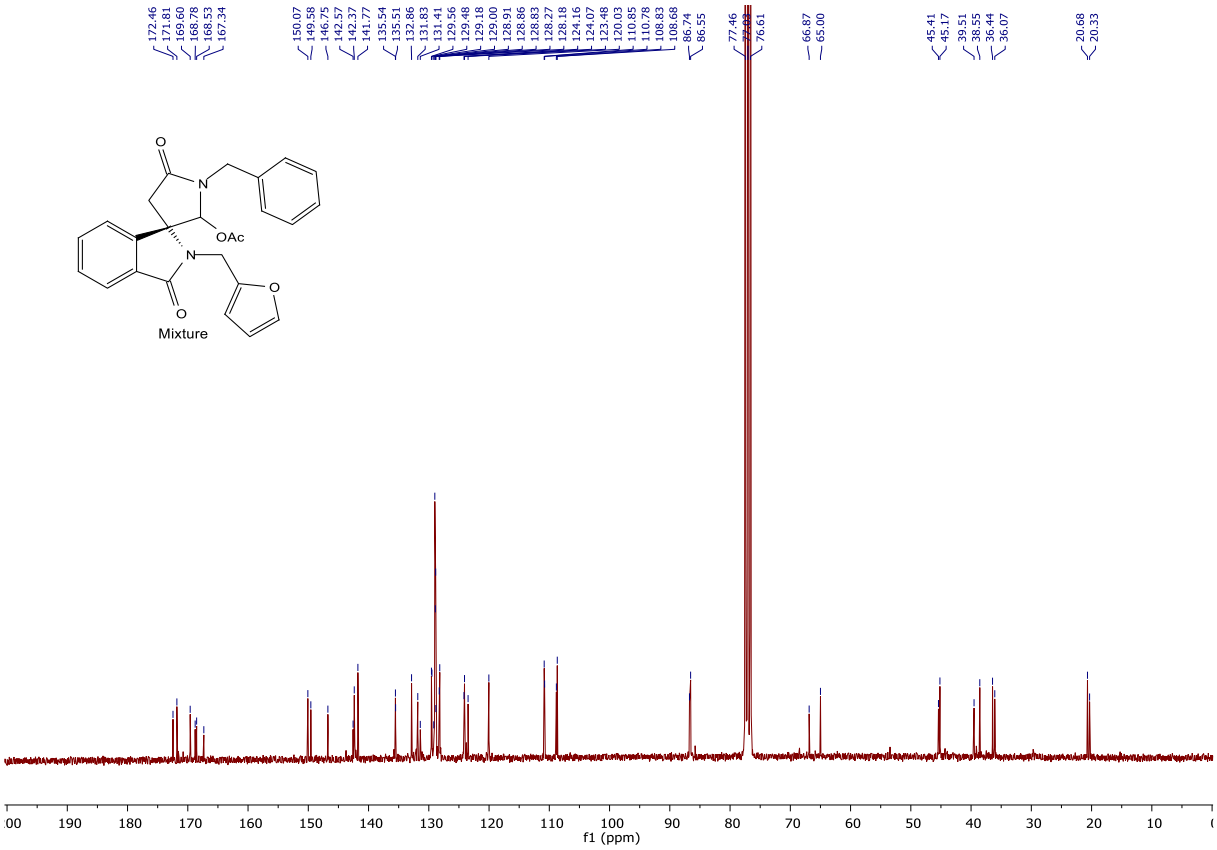
^{1}H NMR spectrum of compound **(±)-11c(A,B)**



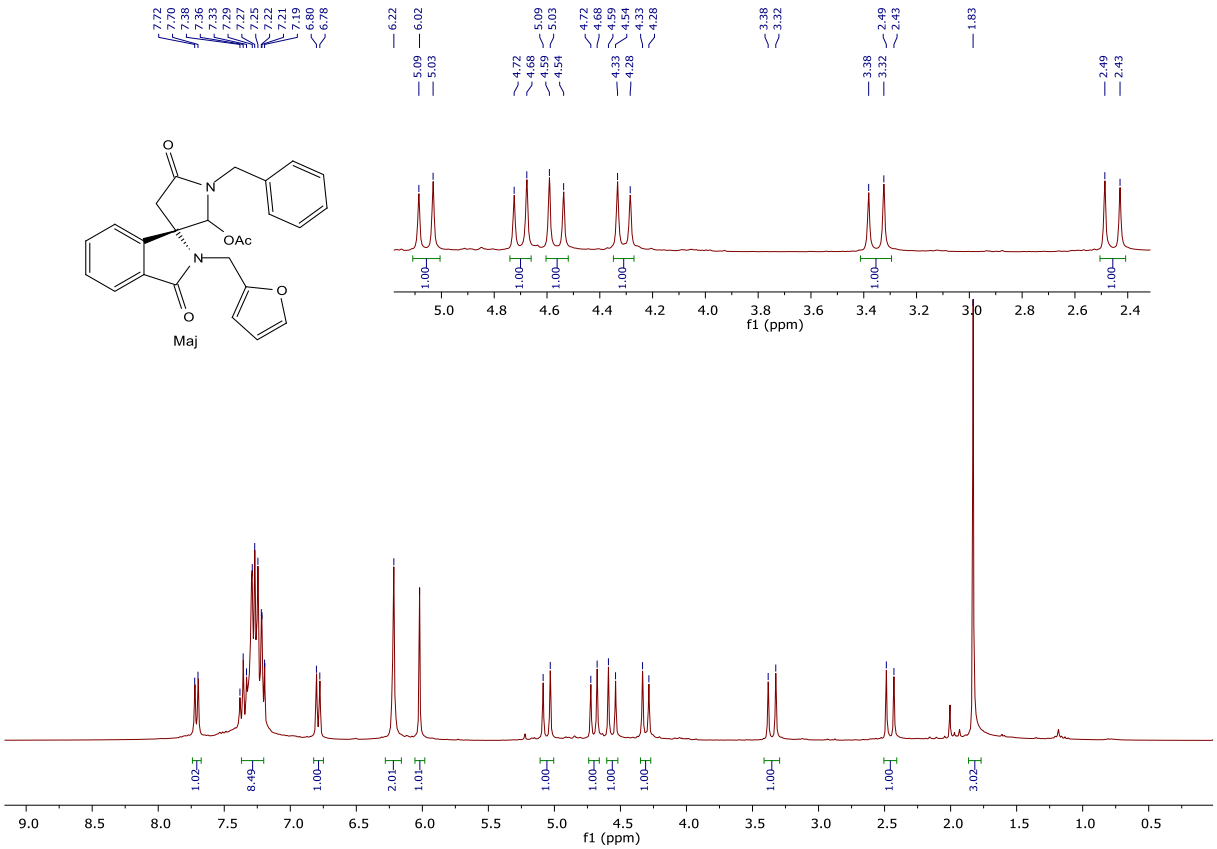
^{13}C NMR spectrum of compound **(±)-11c(A,B)**



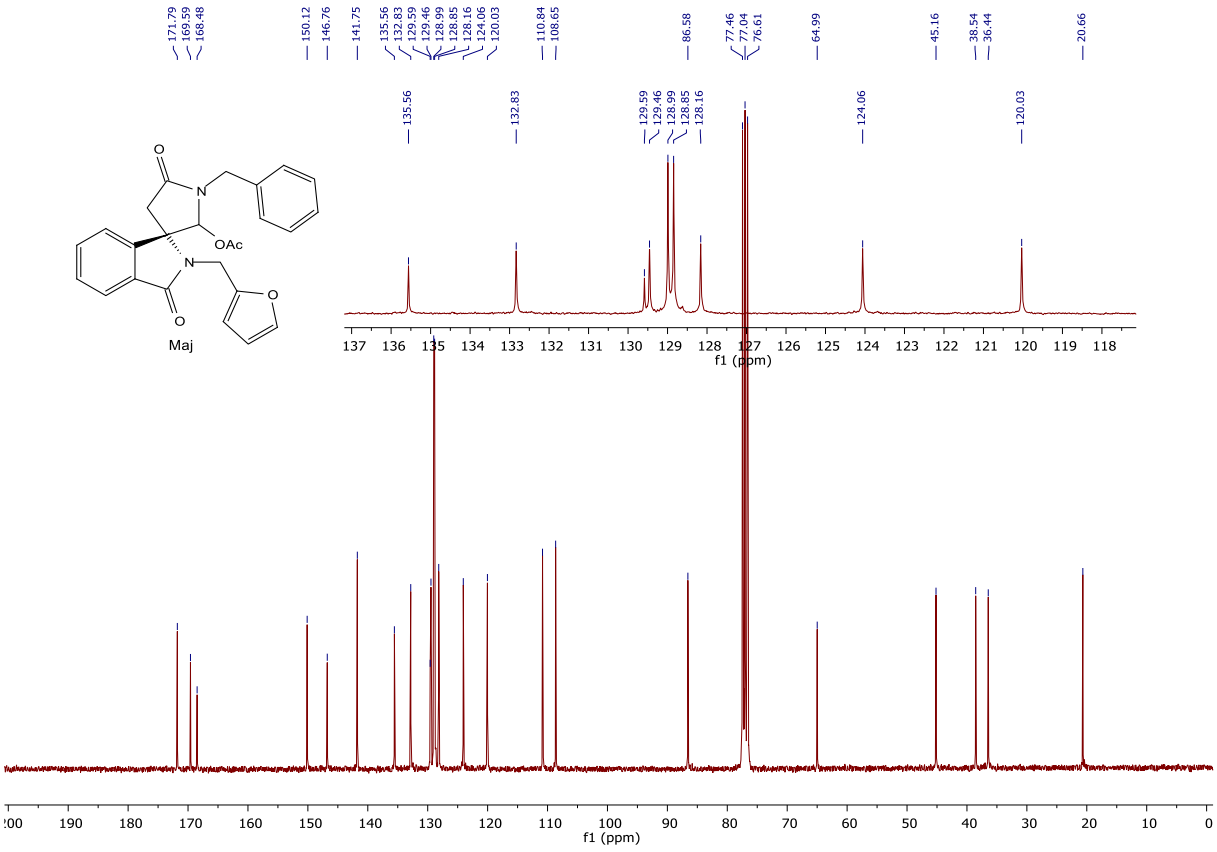
^{1}H NMR spectrum of compound **(±)-8c(A,B)**



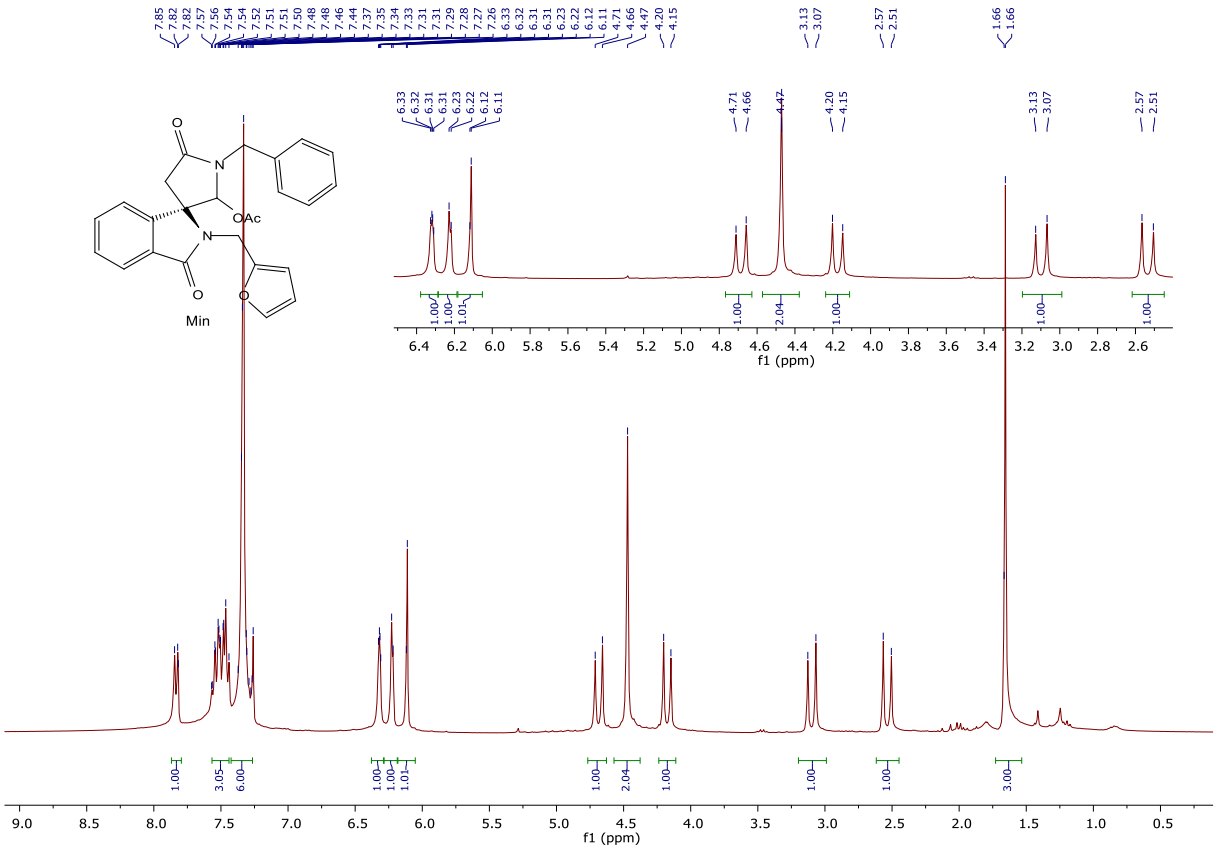
^{13}C NMR spectrum of compound **(±)-8c(A,B)**



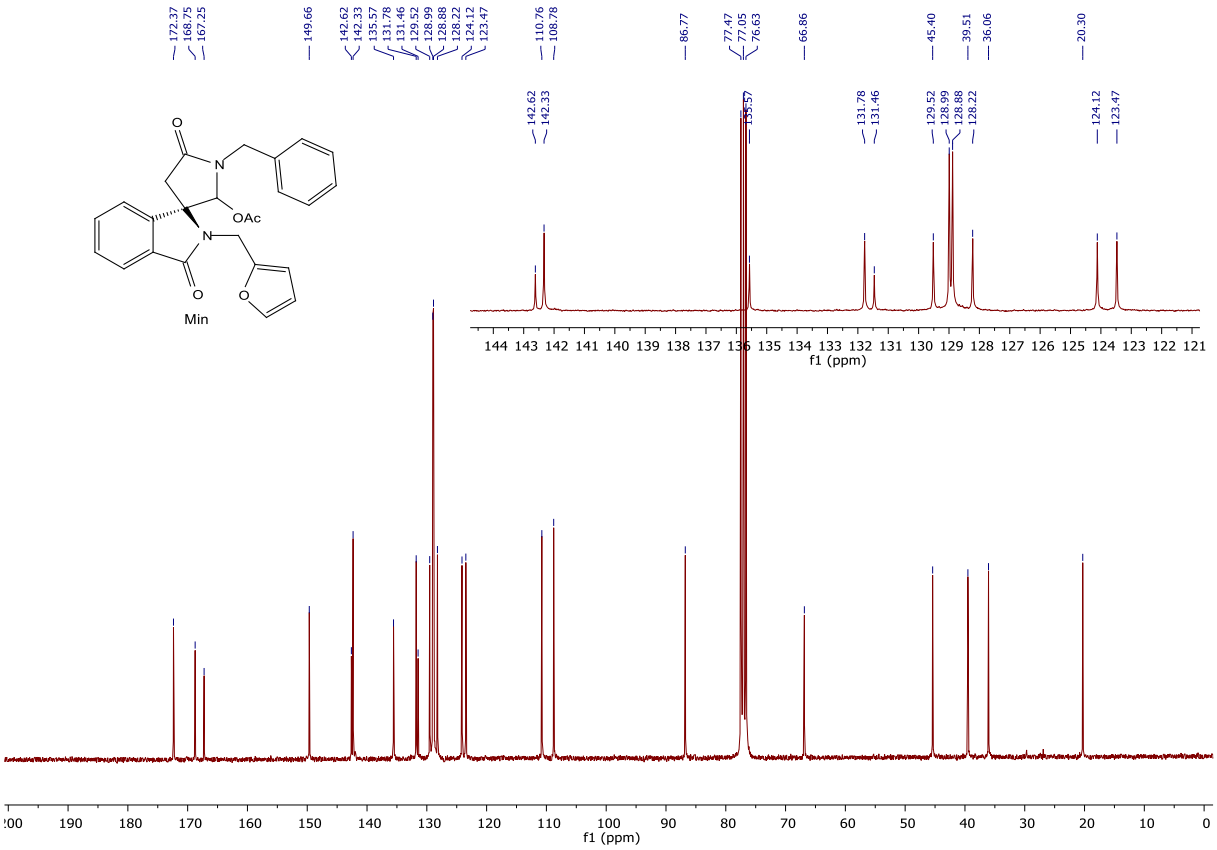
^{1}H NMR spectrum of compound **(±)-8cA**



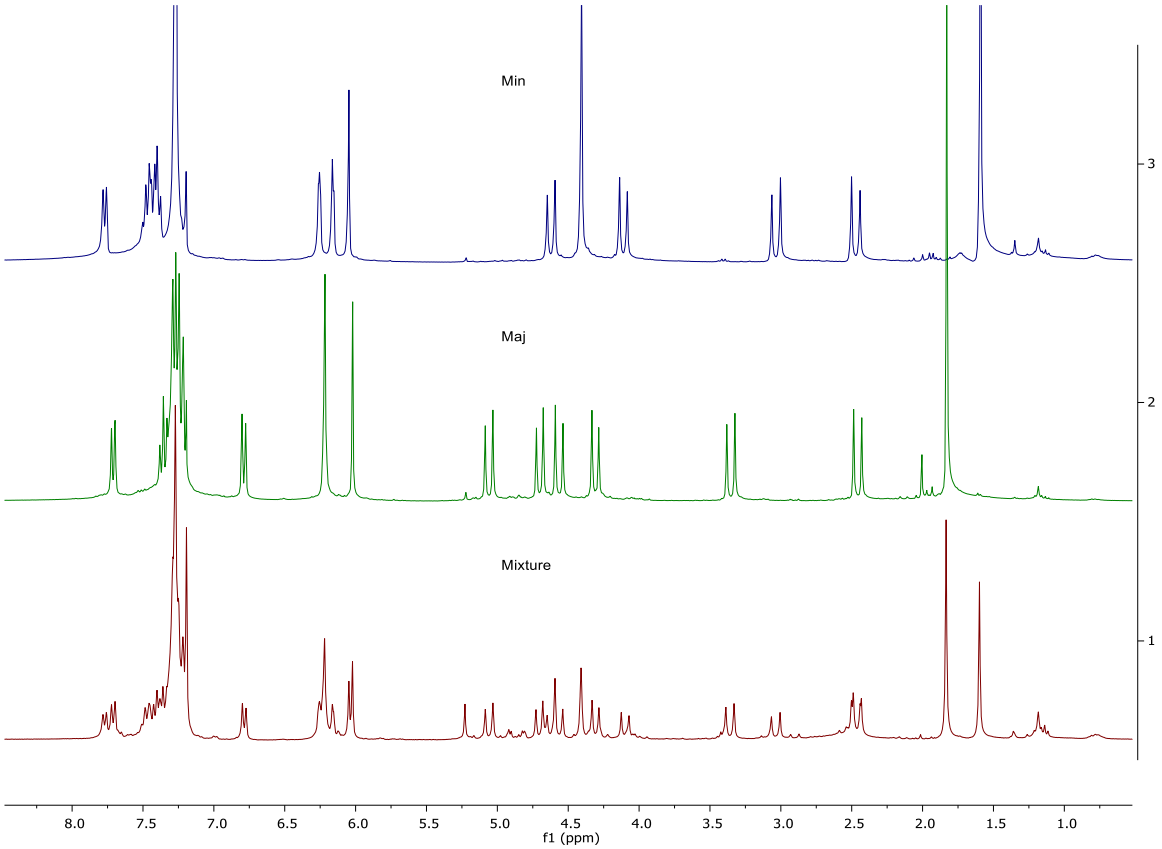
^{13}C NMR spectrum of compound **(±)-8cA**



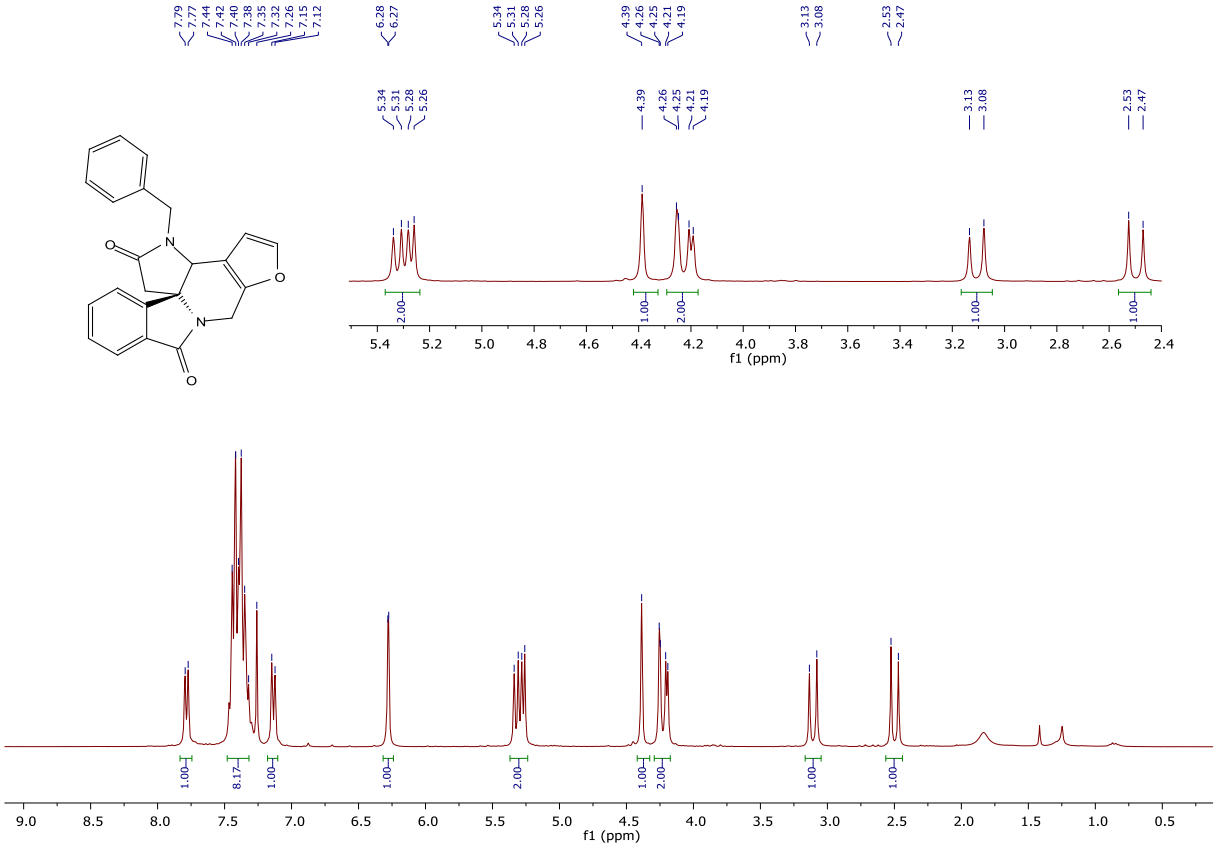
^{1}H NMR spectrum of compound **(±)-8cB**



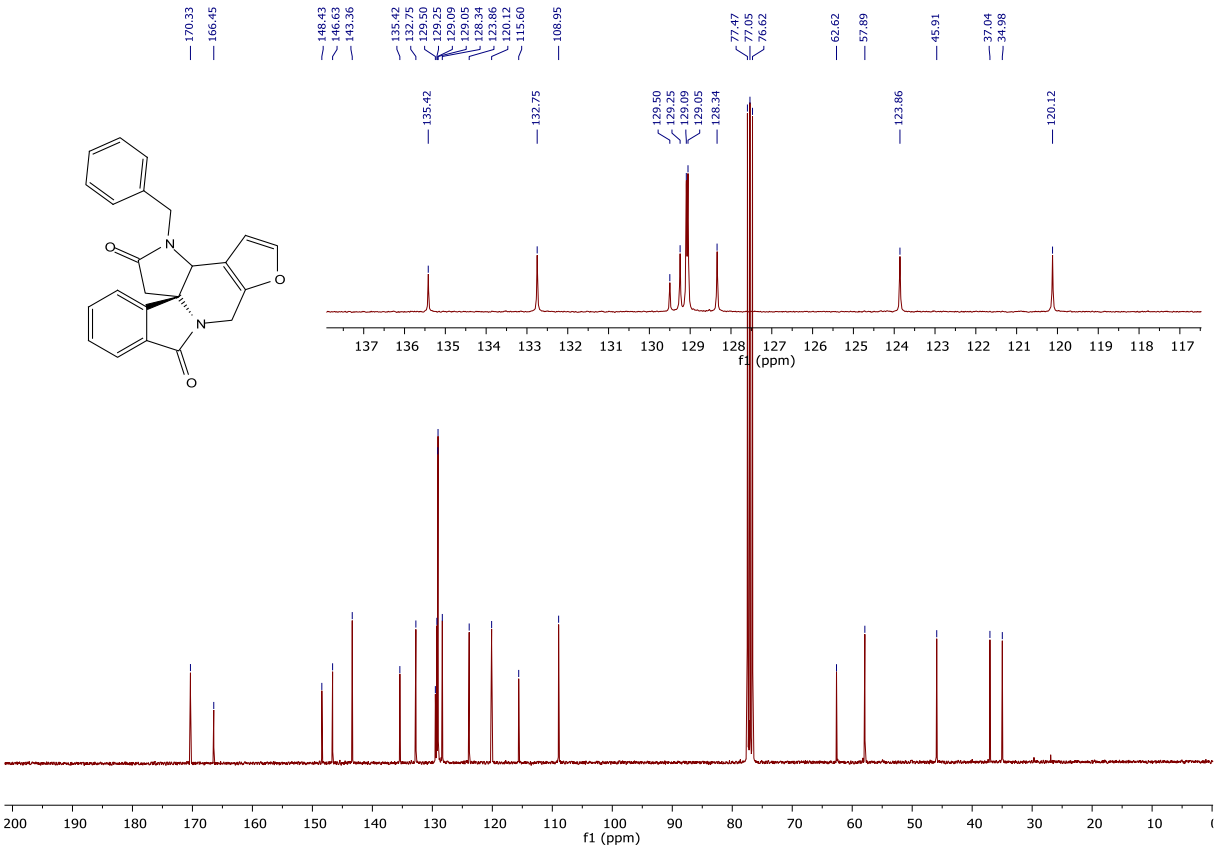
^{13}C NMR spectrum of compound **(±)-8cB**



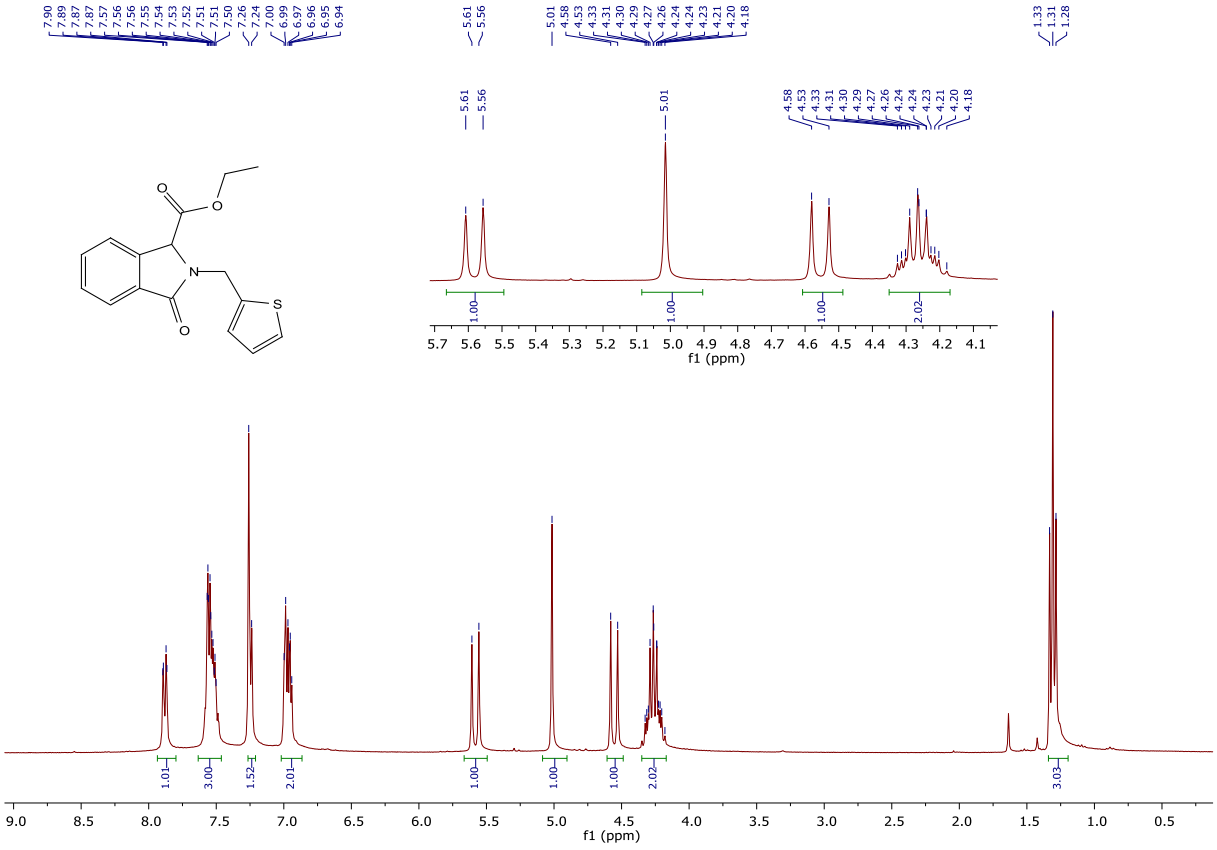
^{1}H NMR spectrum of compounds **(±)-8c(A,B)**, **(±)-8cA** and **(±)-8cB**



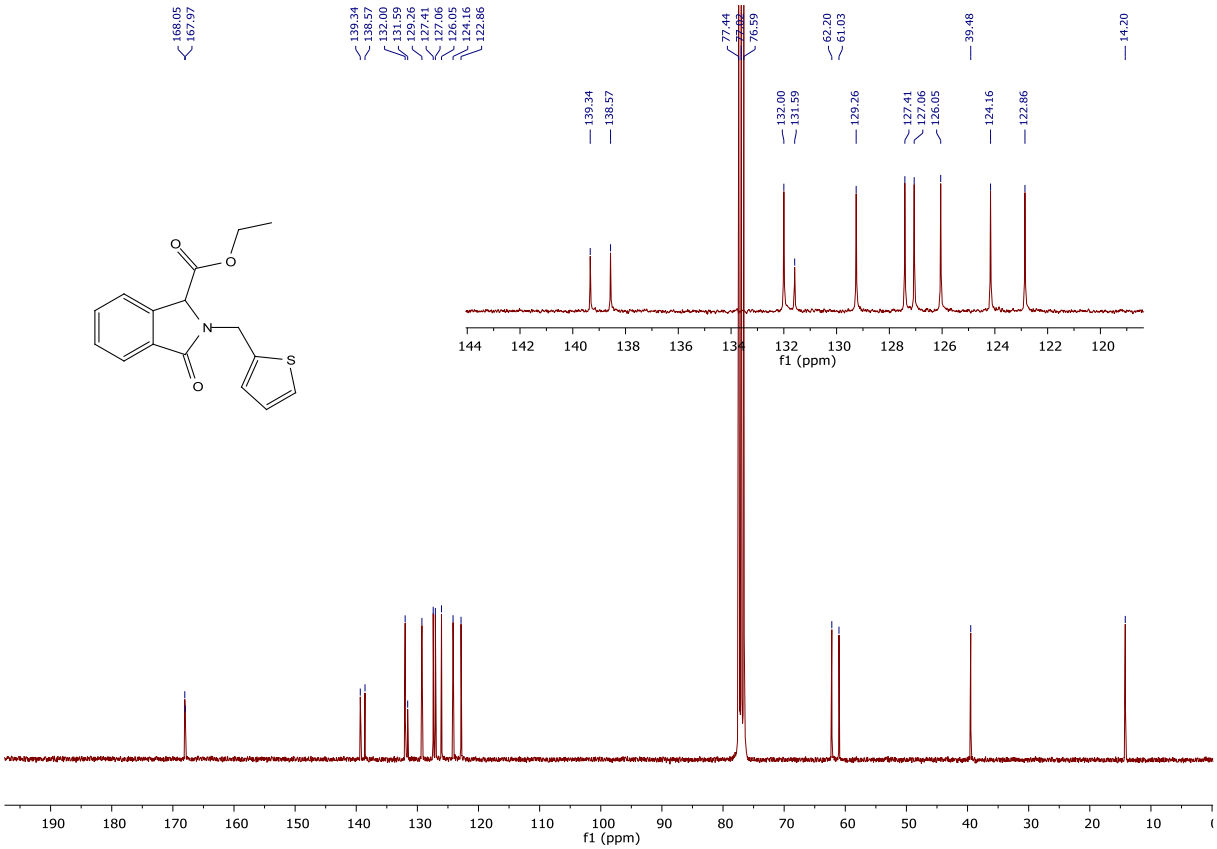
^{1}H NMR spectrum of compound **(±)-5cA**



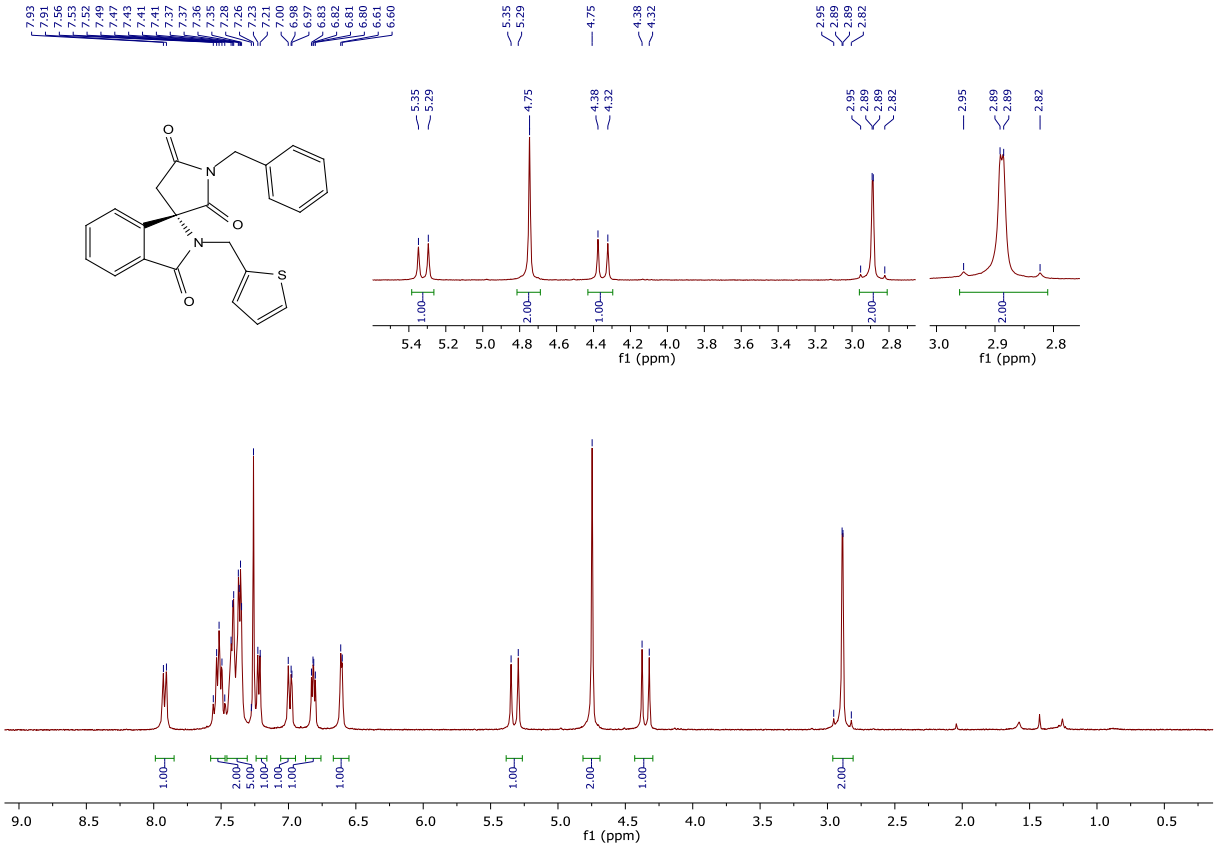
^{13}C NMR spectrum of compound **(±)-5cA**



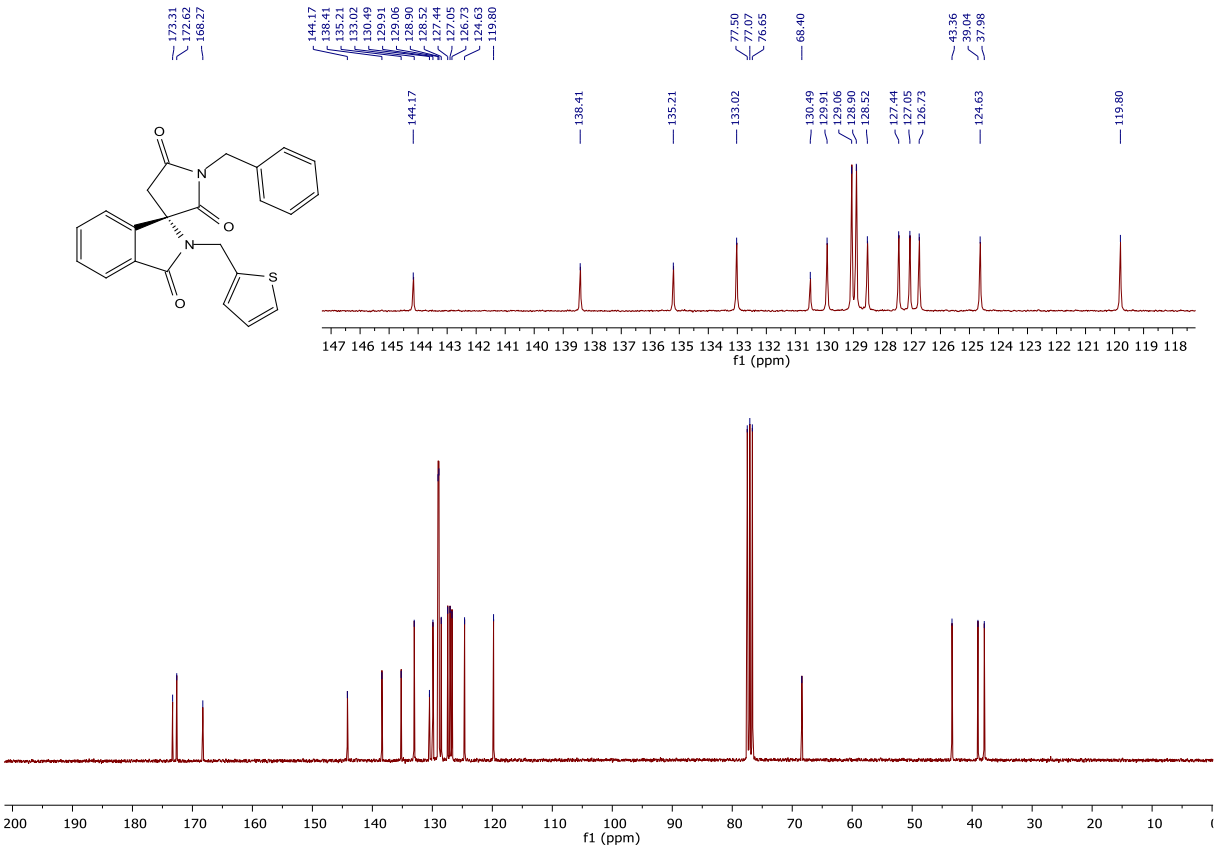
^{1}H NMR spectrum of compound **(±)-7d**



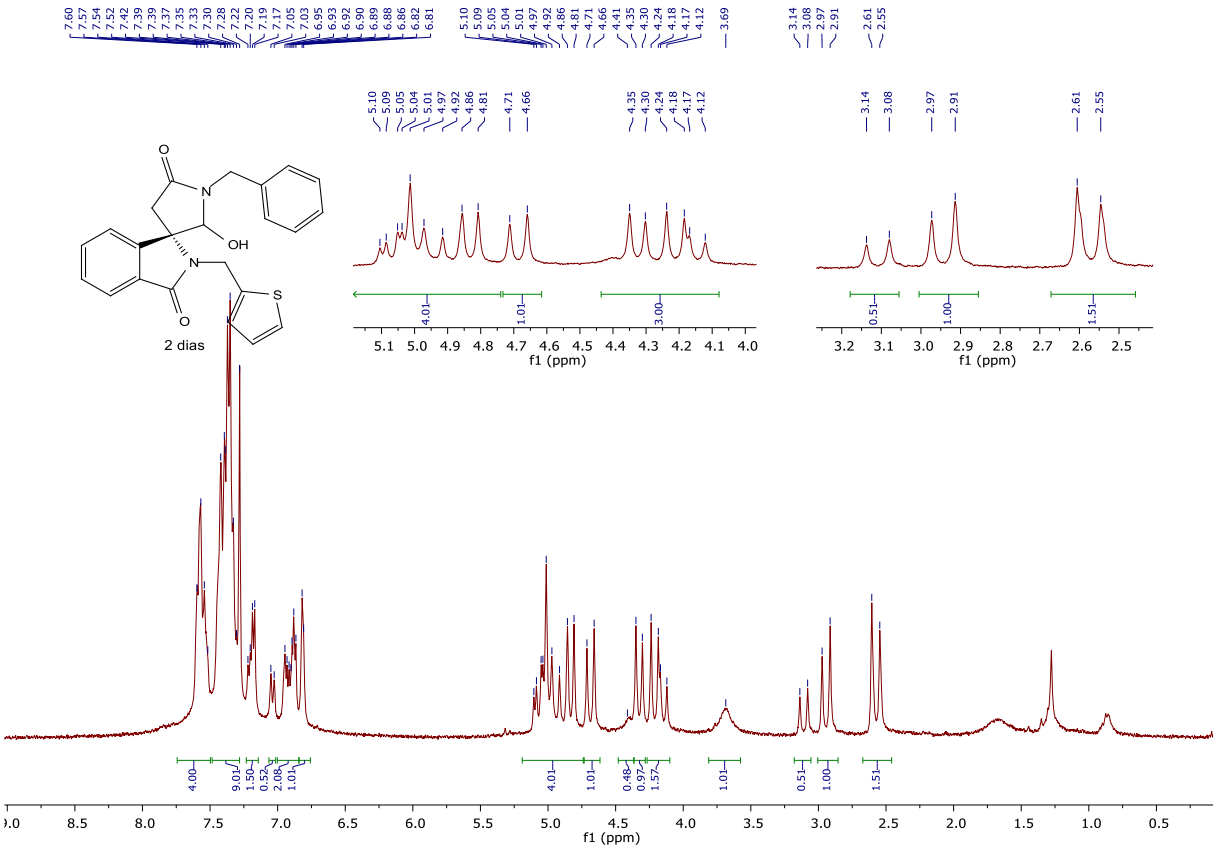
^{13}C NMR spectrum of compound **(±)-7d**



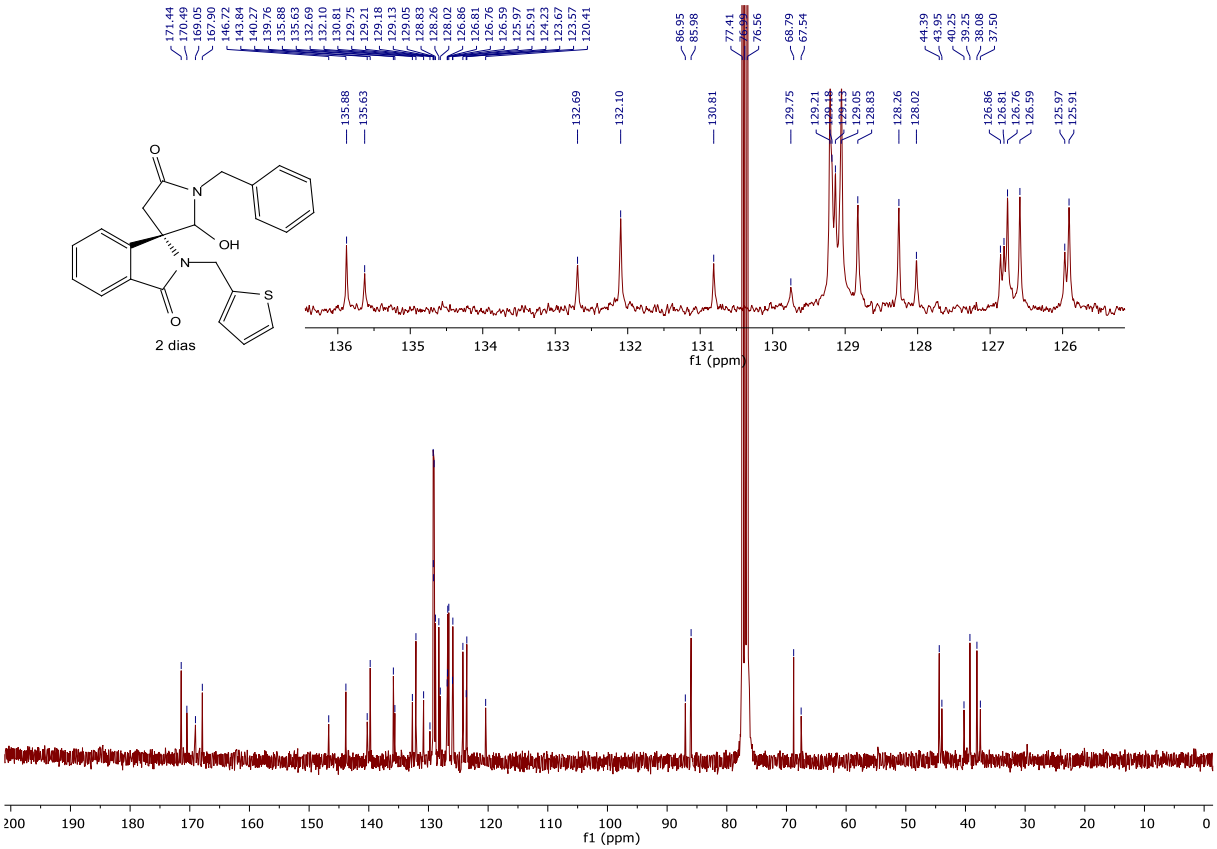
^{1}H NMR spectrum of compound **(±)-1d**



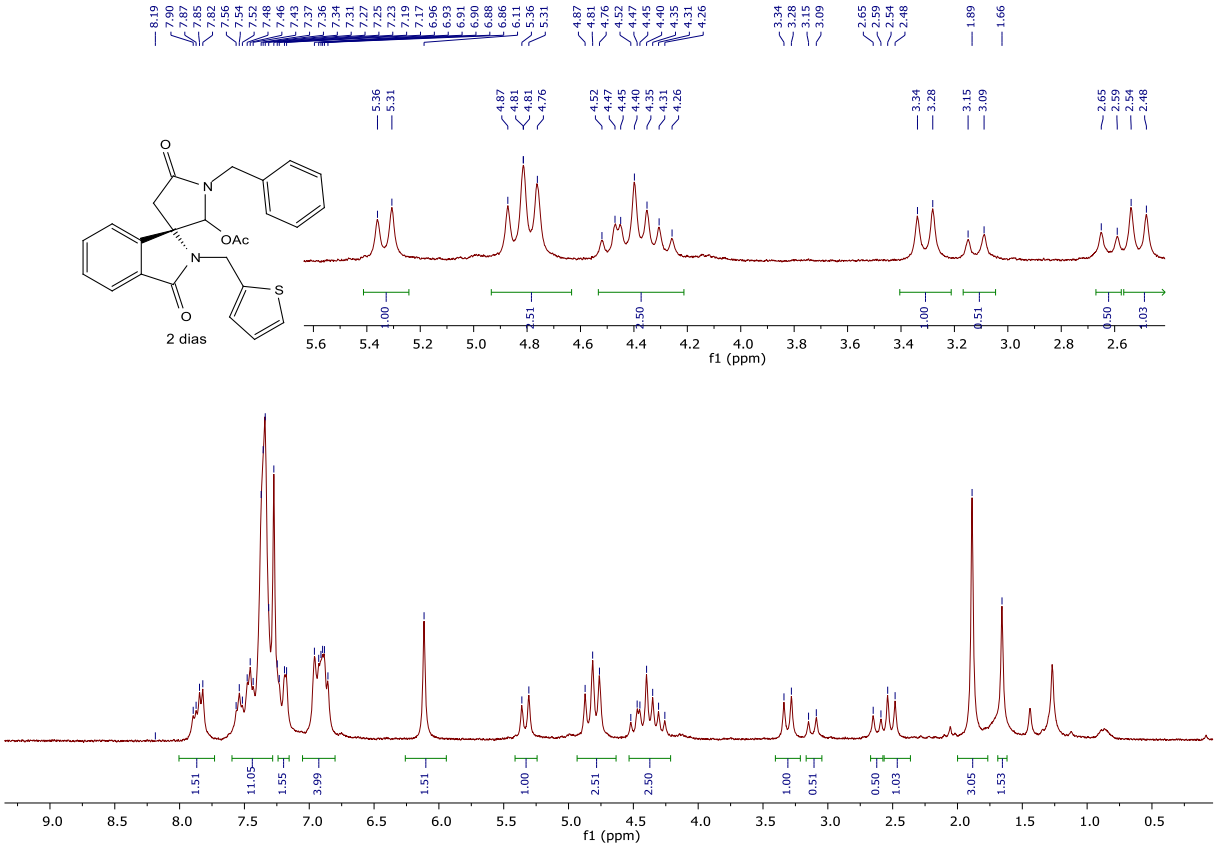
^{13}C NMR spectrum of compound **(±)-1d**



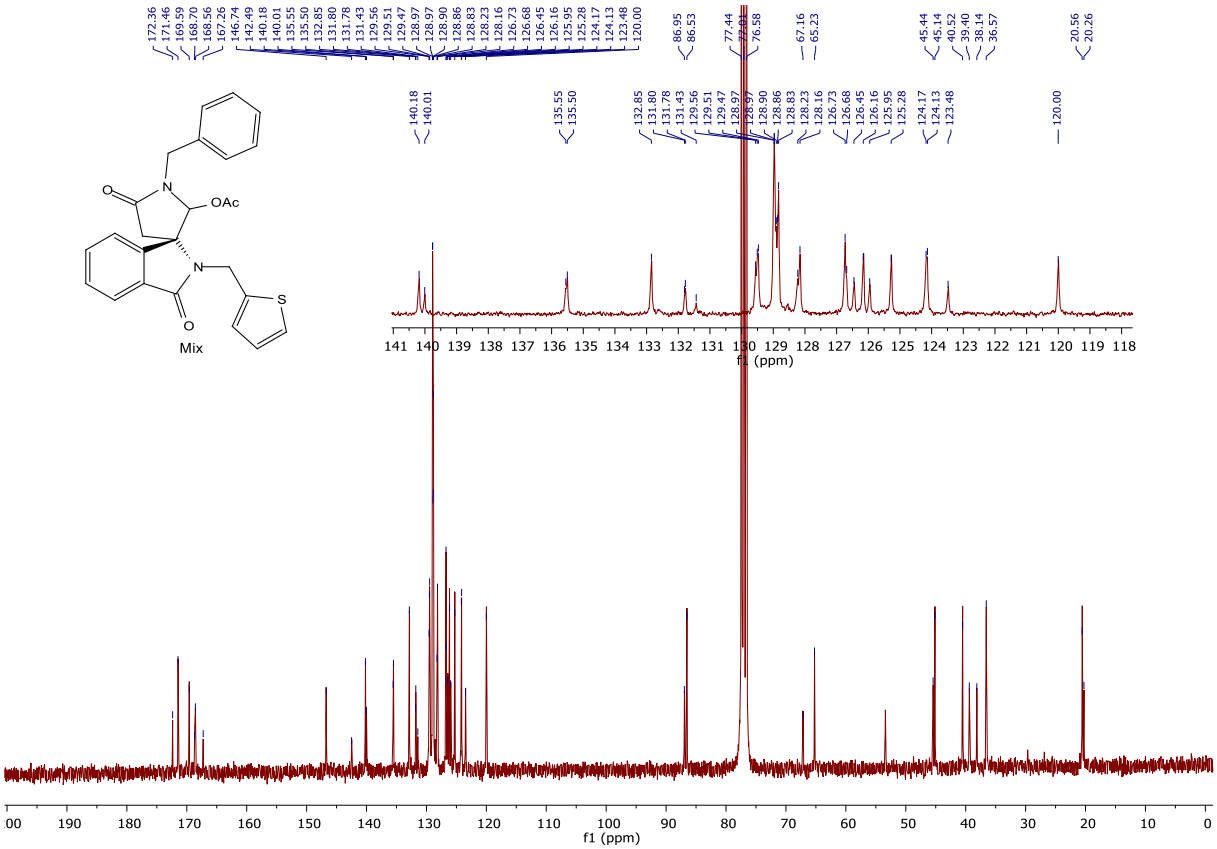
^{1}H NMR spectrum of compound **(±)-11d(A,B)**



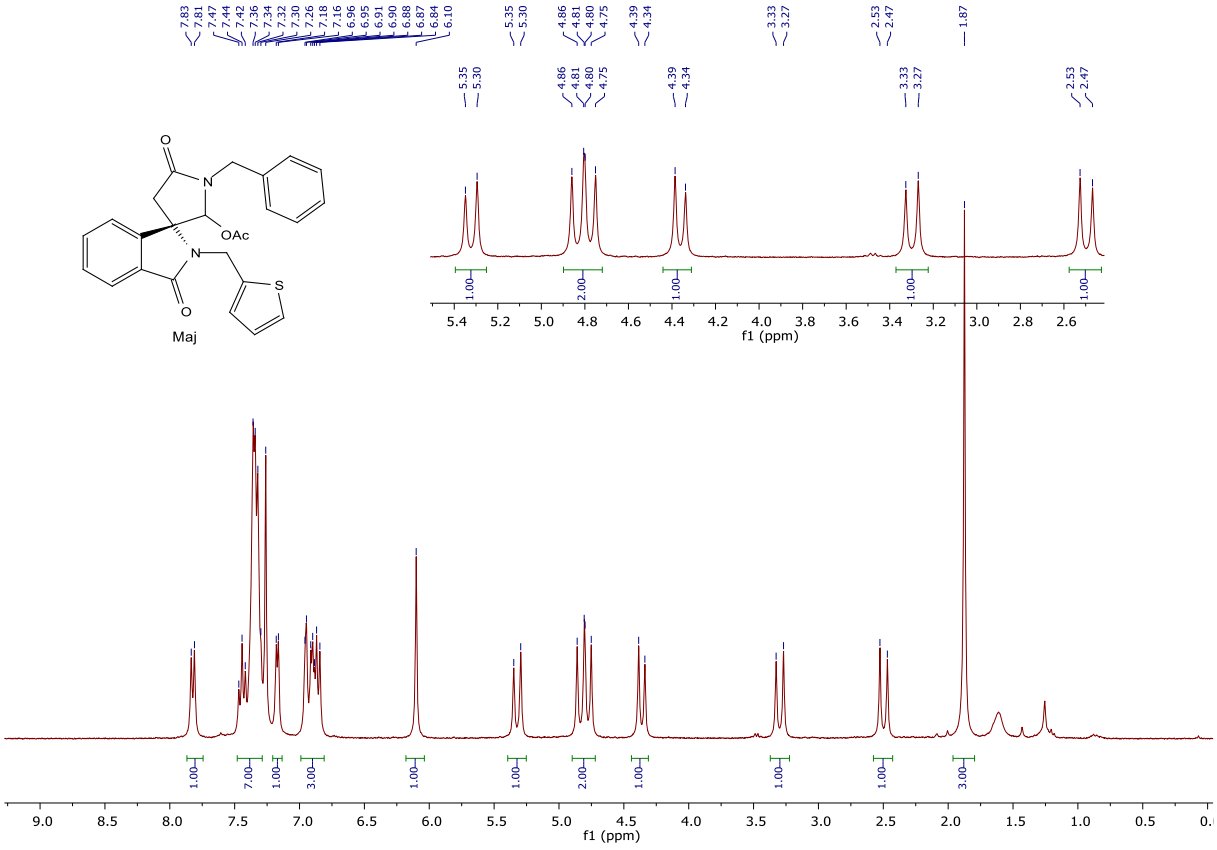
^{13}C NMR spectrum of compound **(±)-11d(A,B)**



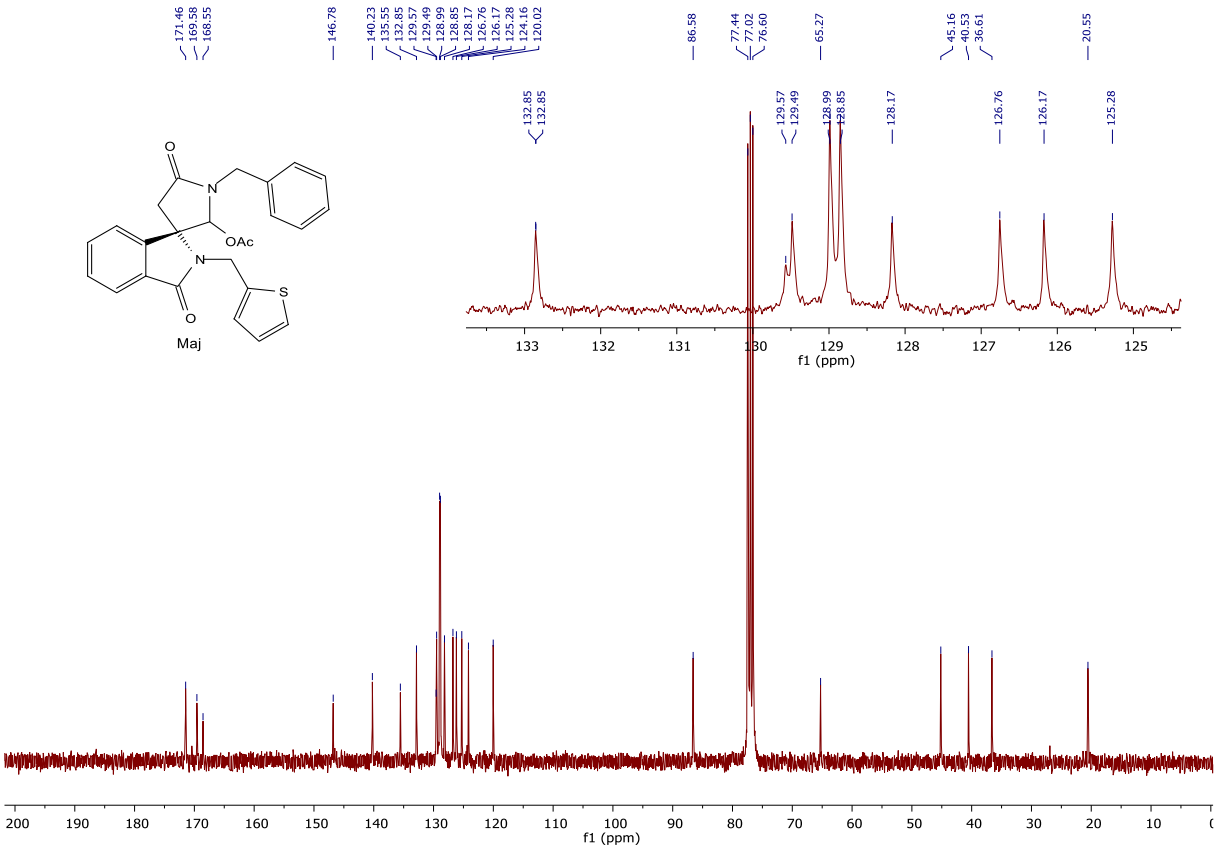
^{1}H NMR spectrum of compound **(±)-8d(A,B)**



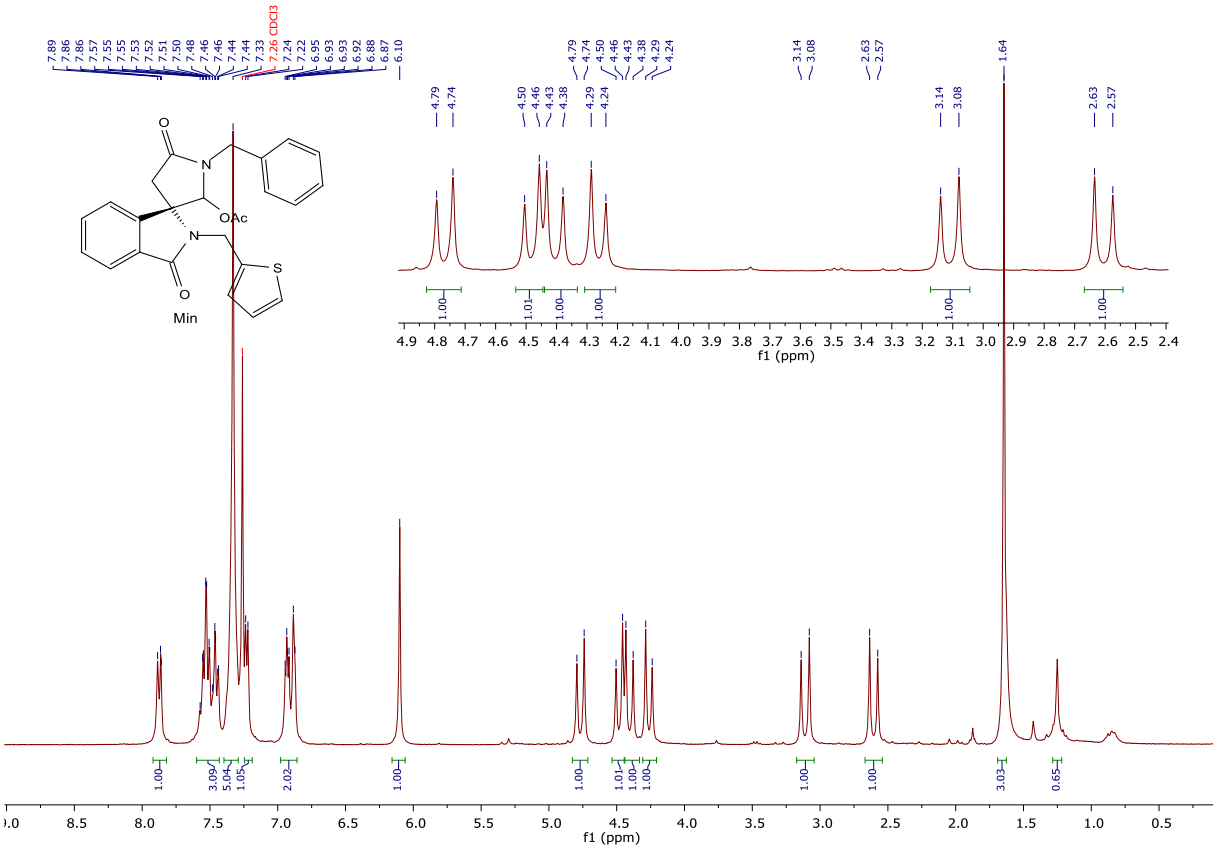
^{13}C NMR spectrum of compound **(±)-8d(A,B)**



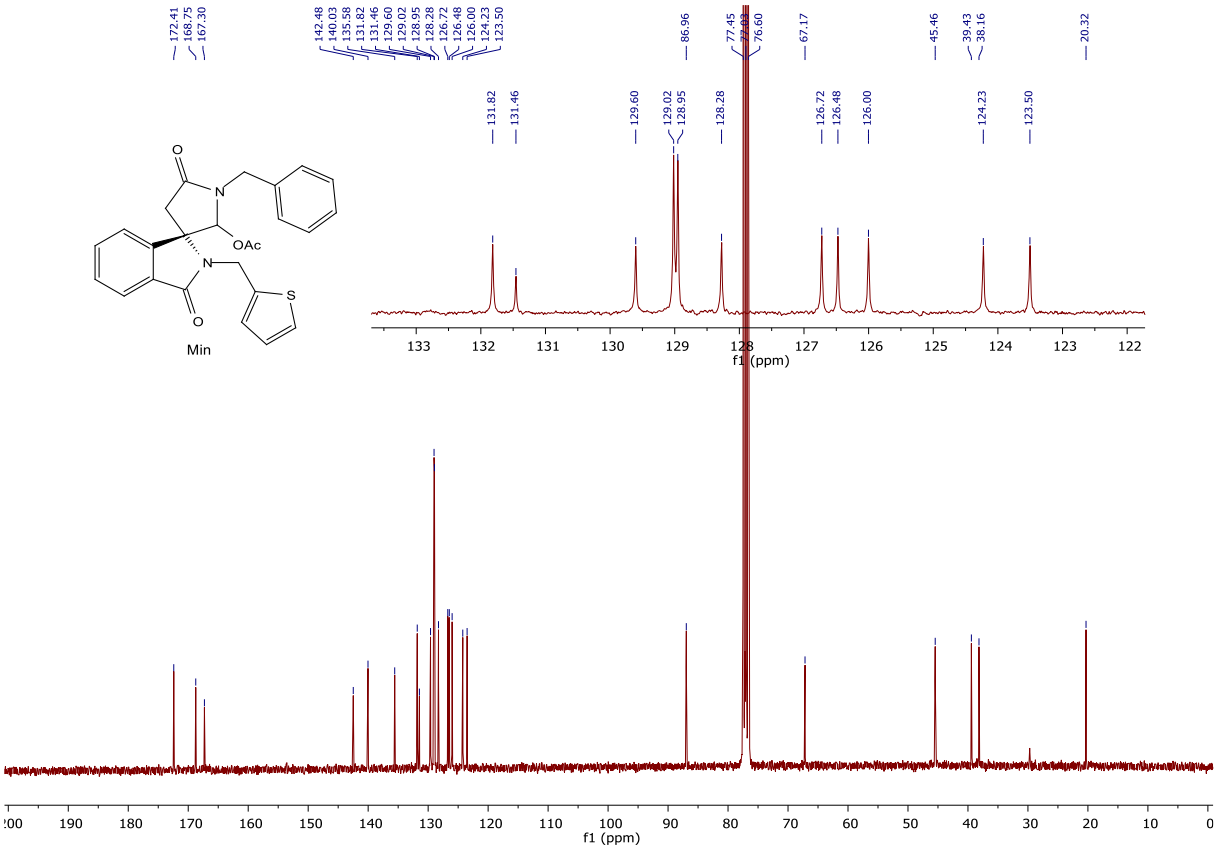
^{1}H NMR spectrum of compound **(±)-8dA**



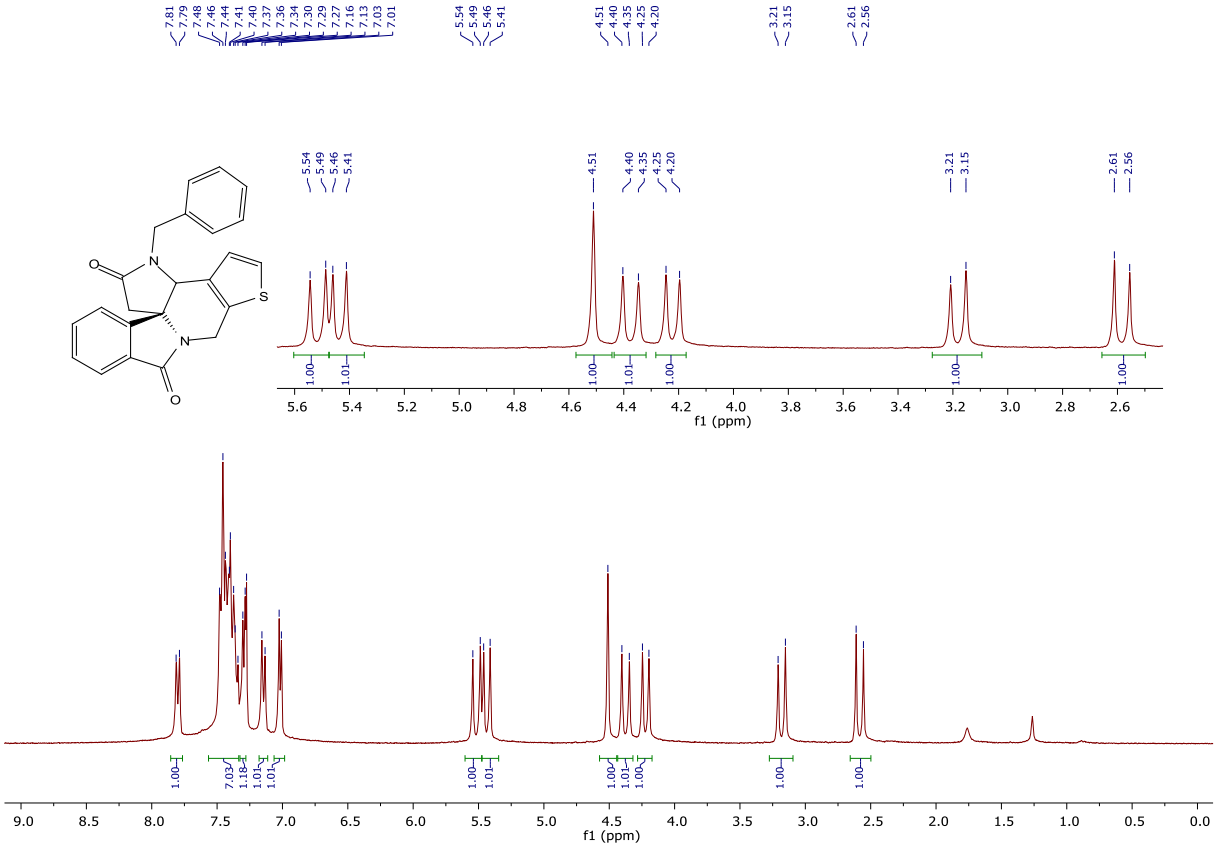
^{13}C NMR spectrum of compound **(±)-8dA**



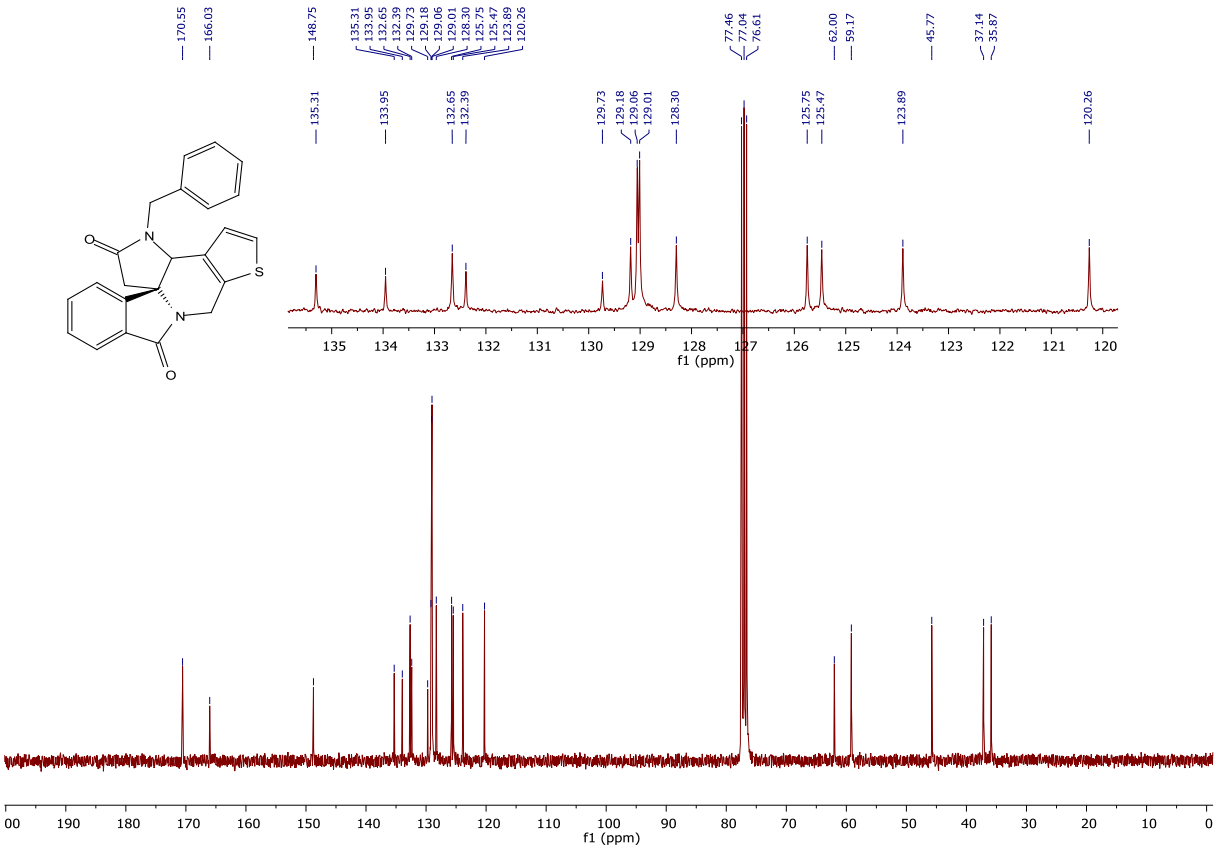
^{1}H NMR spectrum of compound **(±)-8dB**



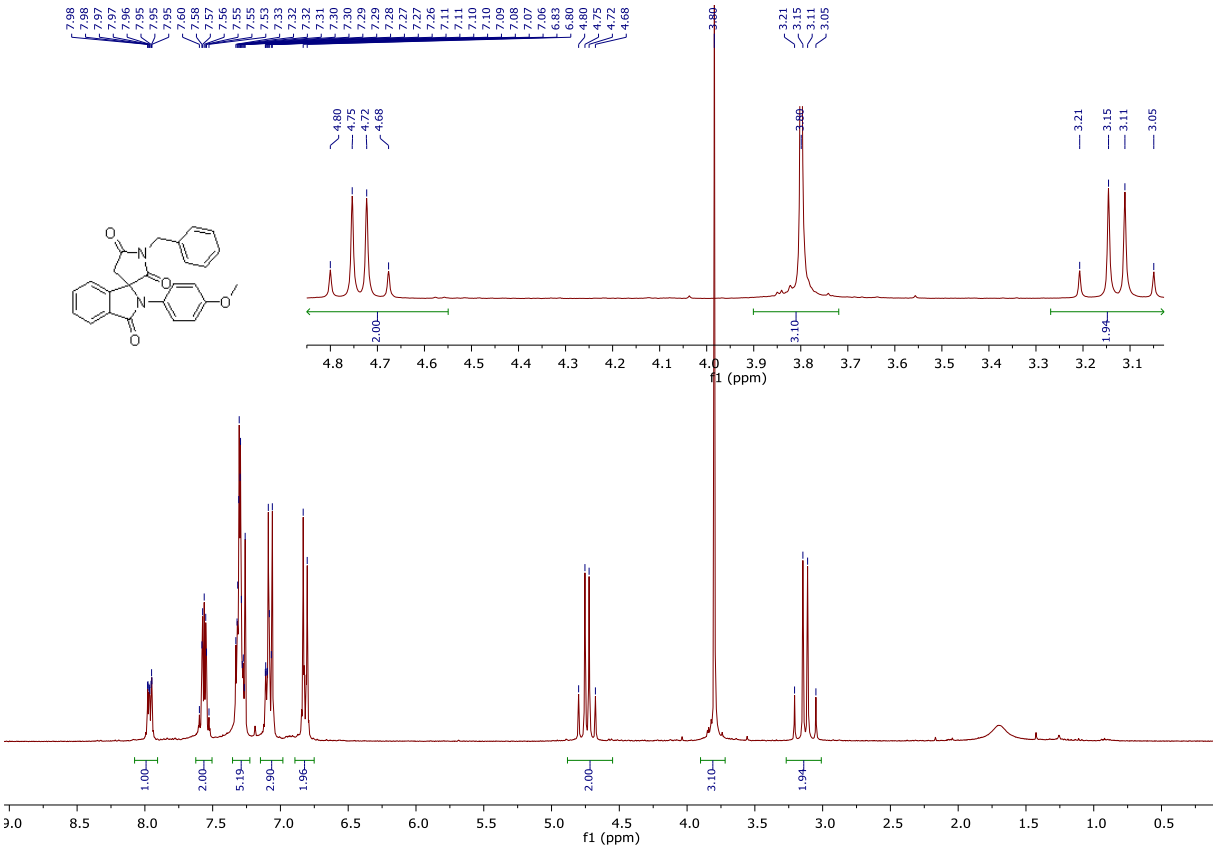
^{13}C NMR spectrum of compound **(±)-8dB**



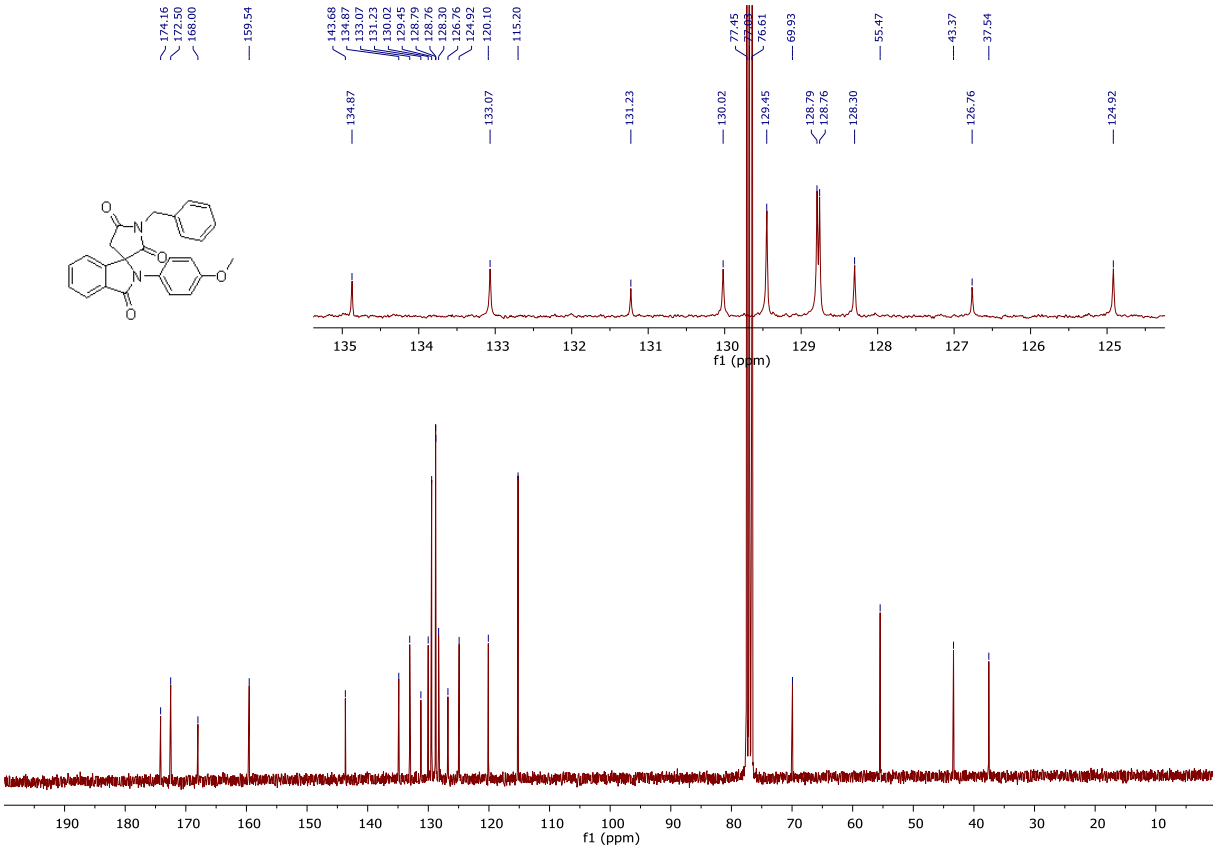
^{1}H NMR spectrum of compound **(±)-5dA**



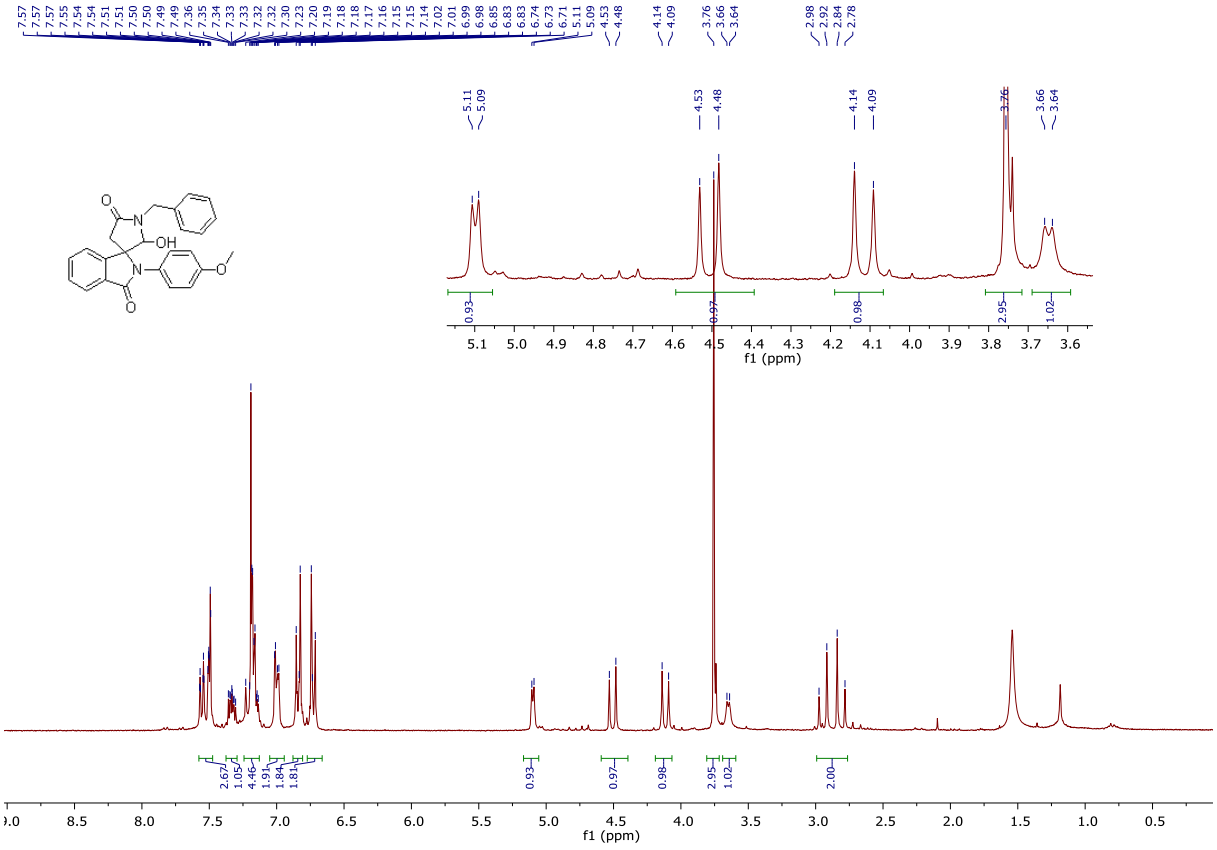
^{13}C NMR spectrum of compound **(±)-5dA**



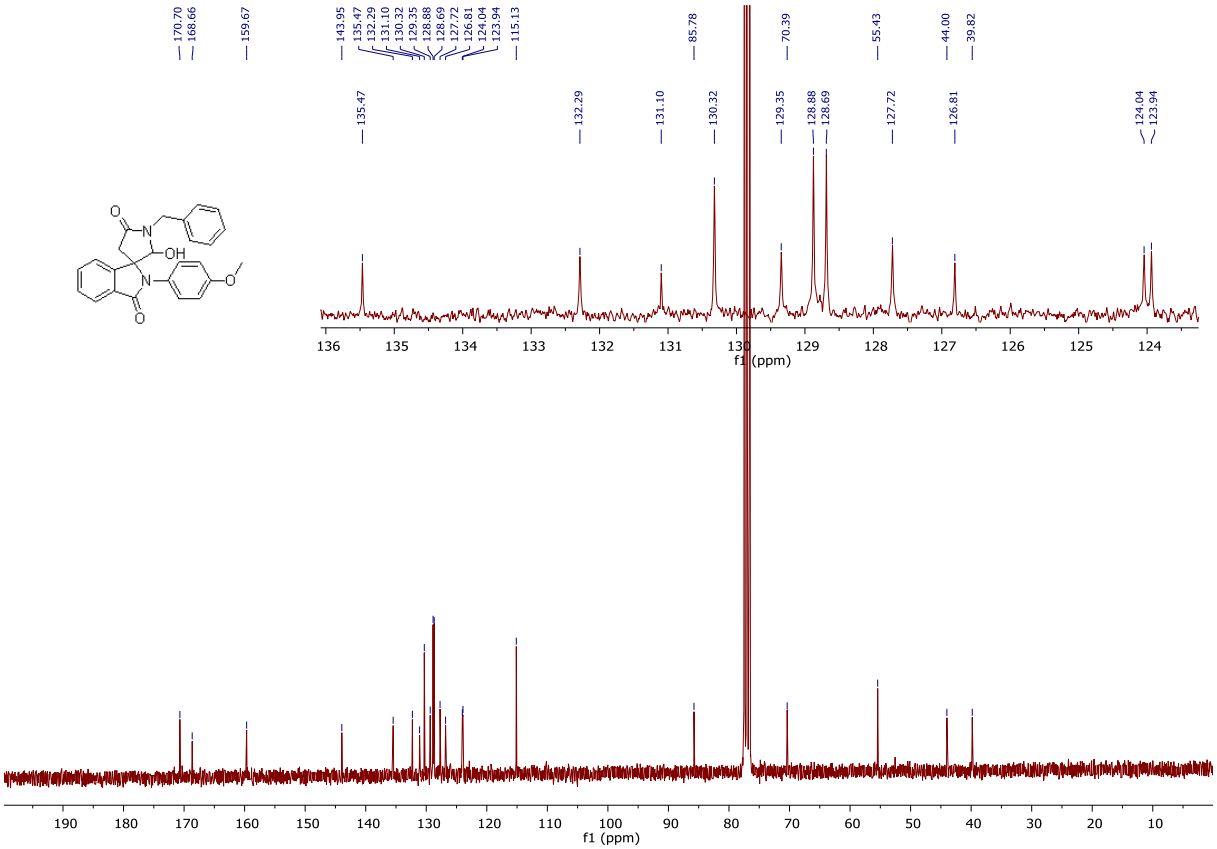
^{1}H NMR spectrum of compound **(±)-1e**



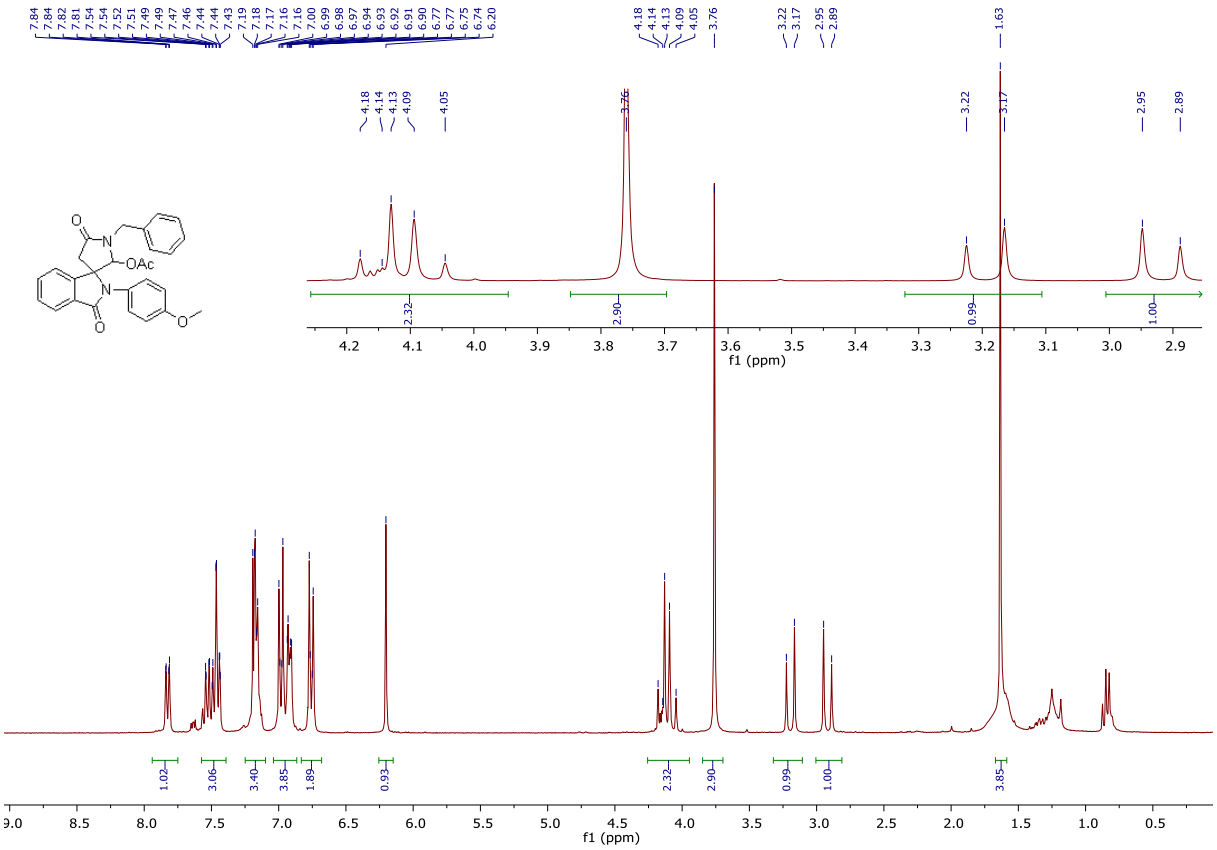
^{13}C NMR spectrum of compound **(±)-1e**



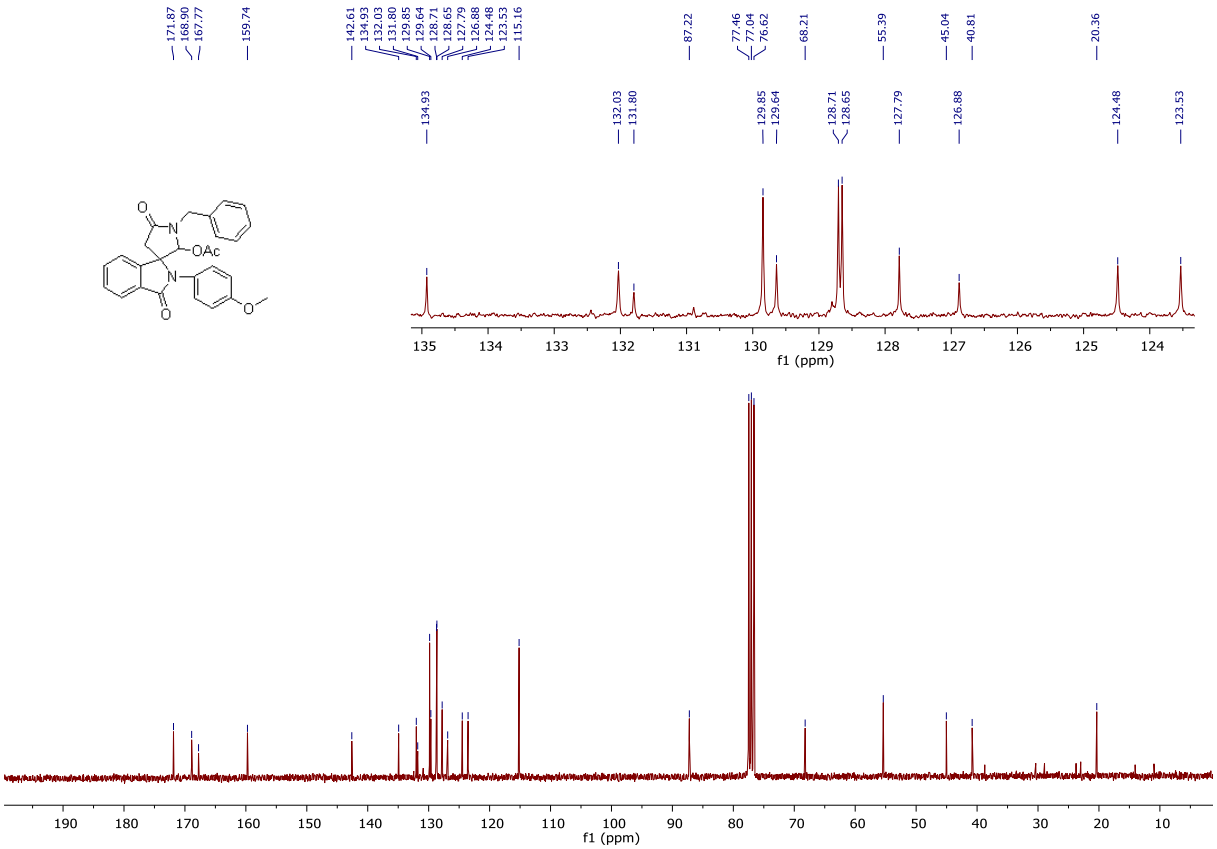
^{1}H NMR spectrum of compound **(±)-11eA**



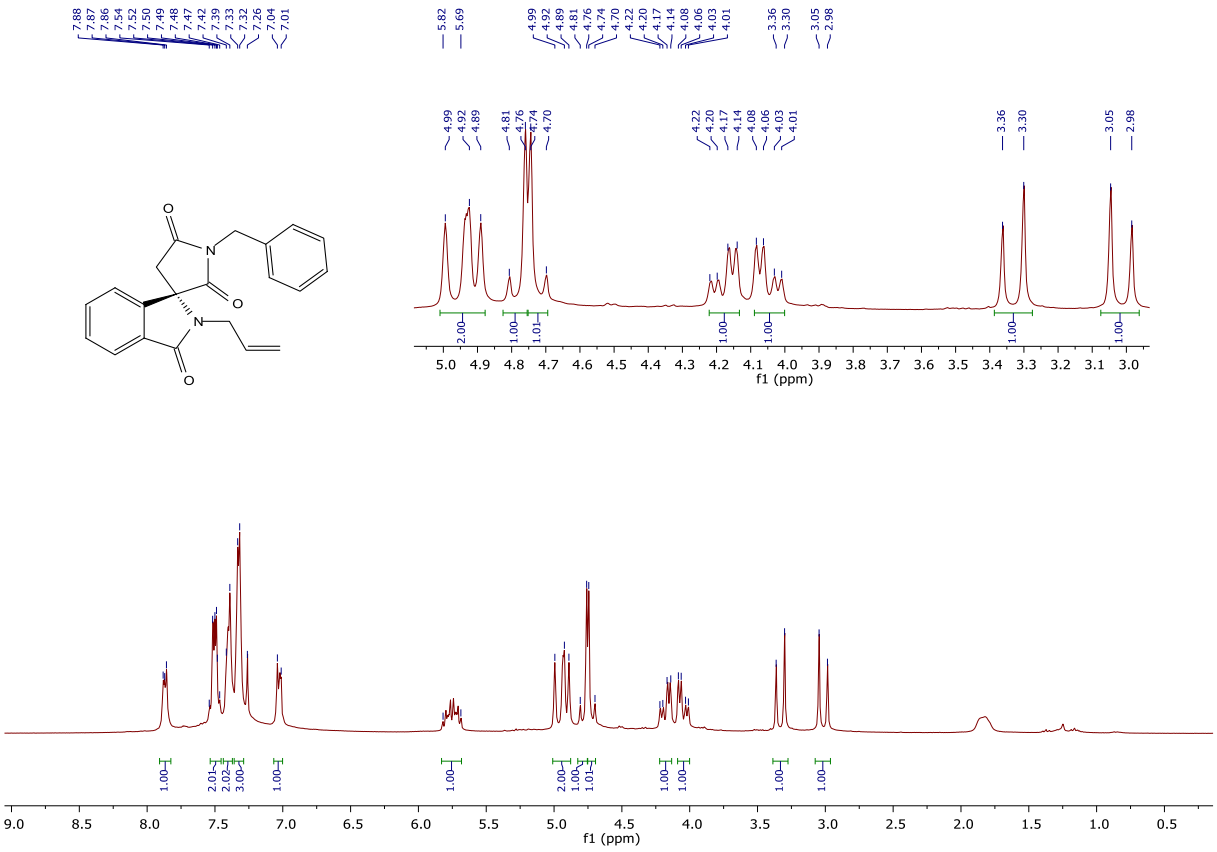
^{13}C NMR spectrum of compound **(±)-11eA**



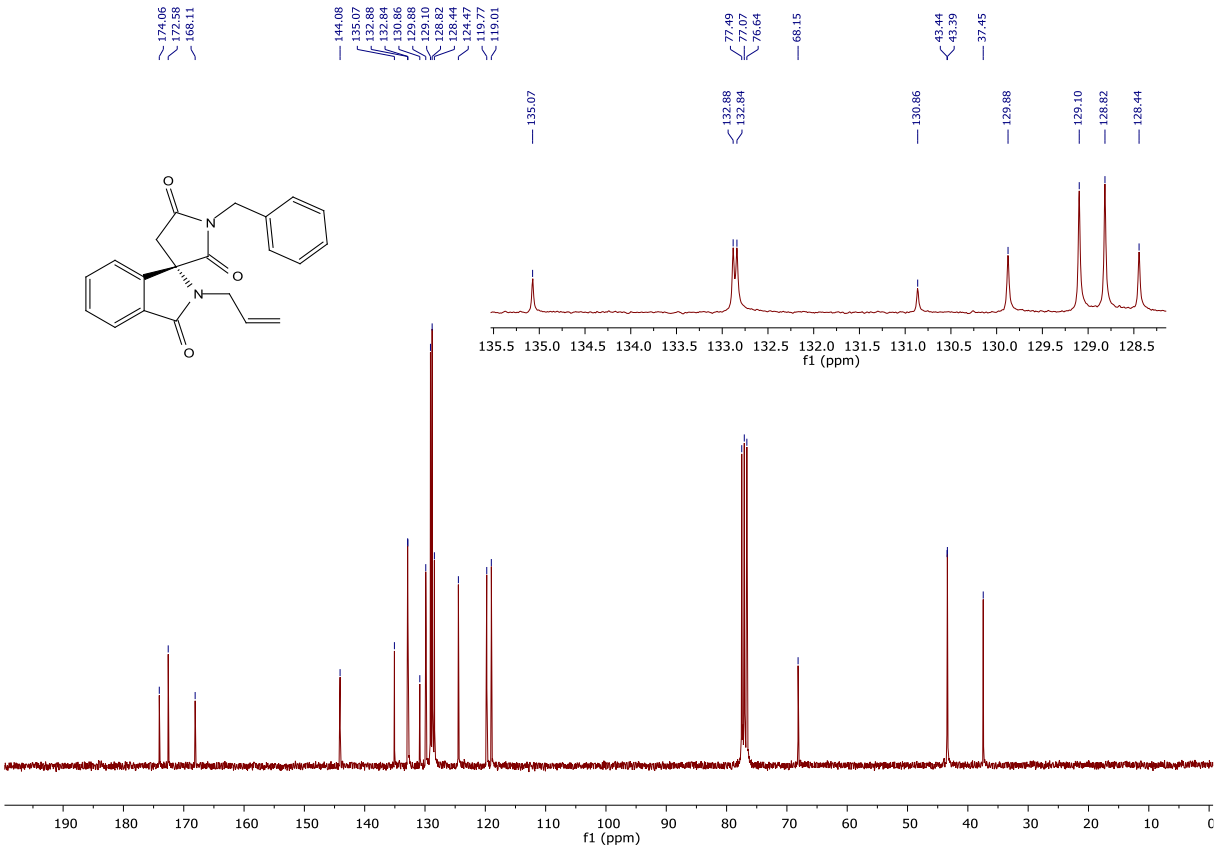
^{1}H NMR spectrum of compound **(±)-8eA**



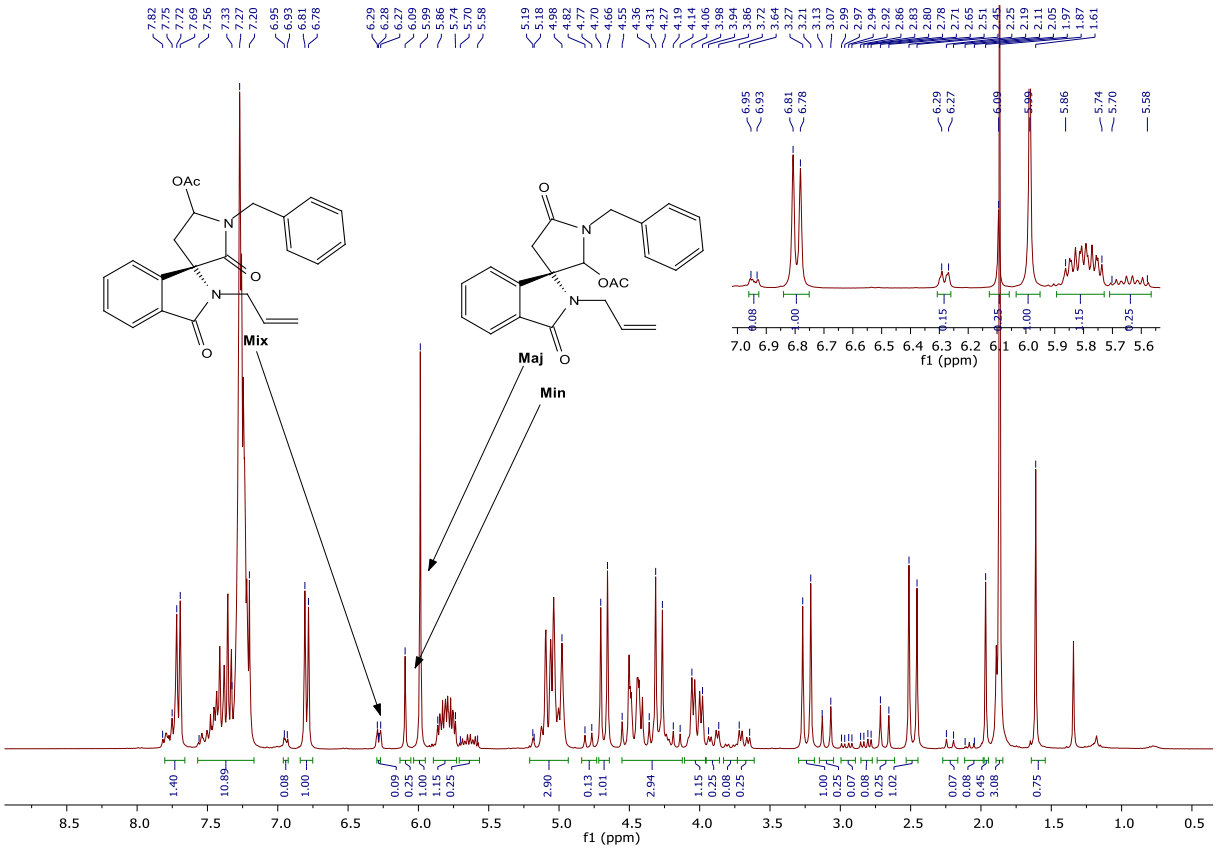
^{13}C NMR spectrum of compound **(±)-8eA**



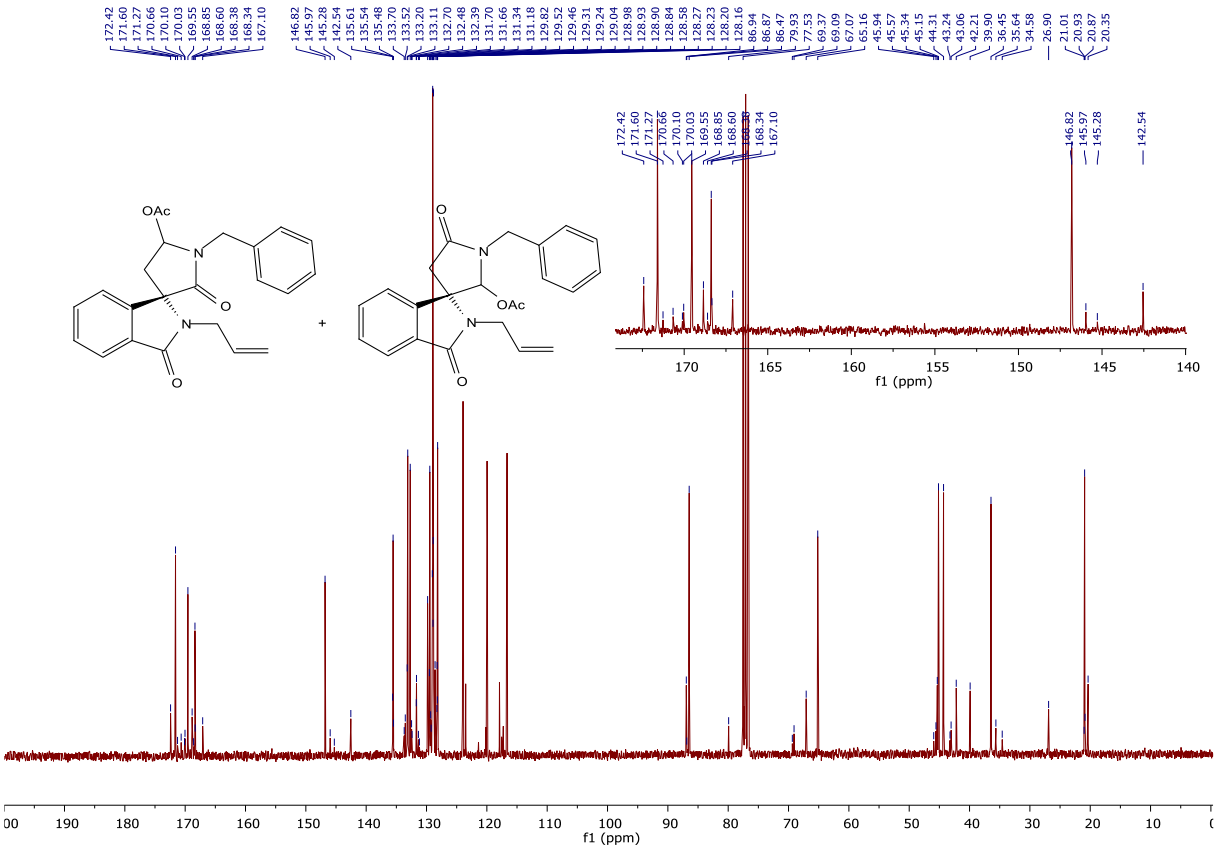
^{1}H NMR spectrum of compound **(±)-1f**



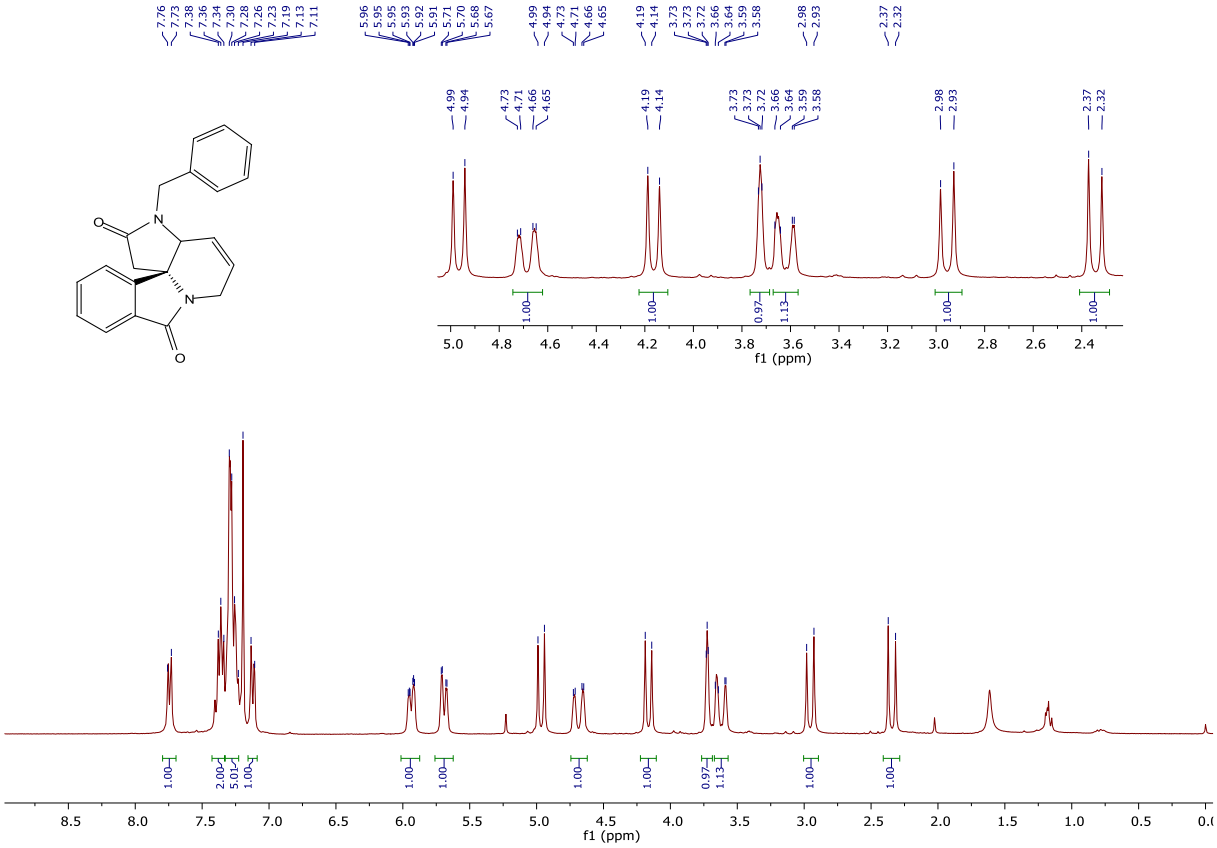
^{13}C NMR spectrum of compound **(±)-1f**



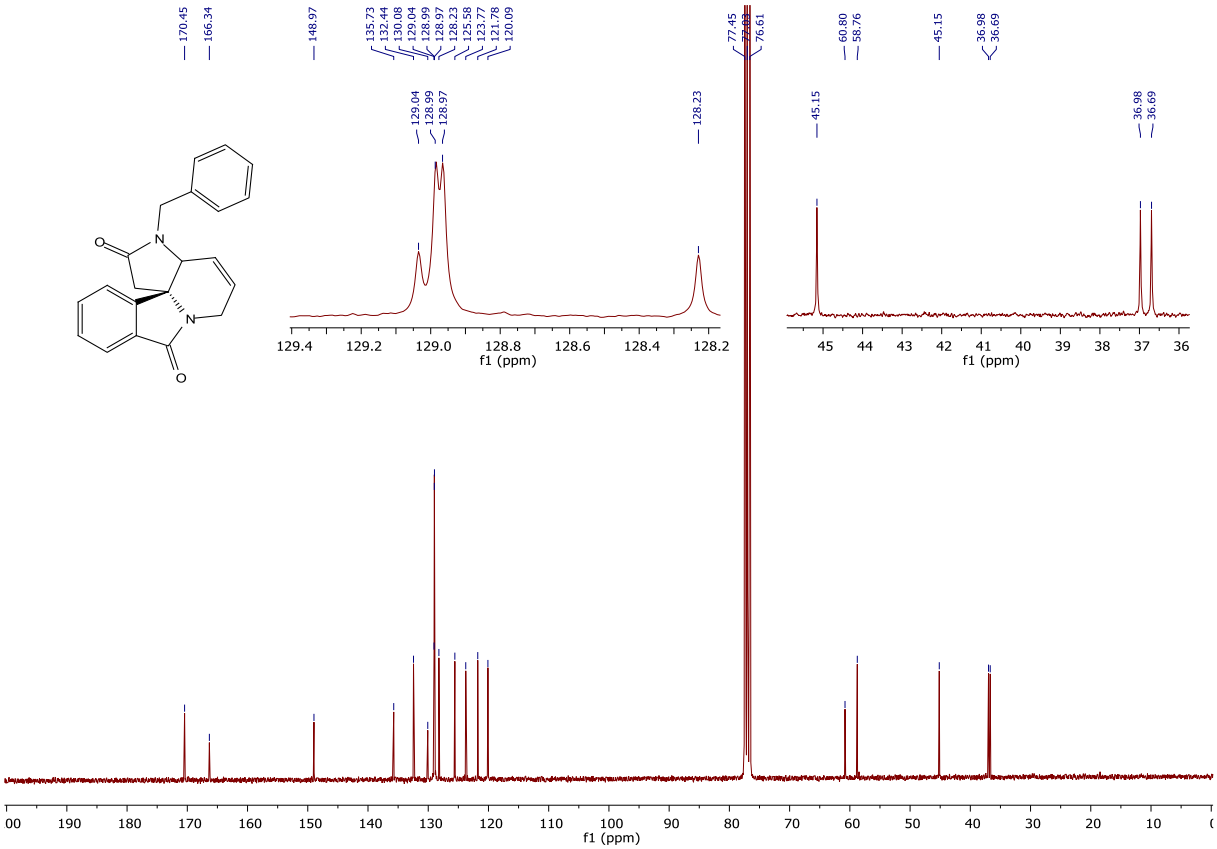
^{1}H NMR spectrum of compounds **(±)-8f(A,B)** and **(±)-9f(A,B)**



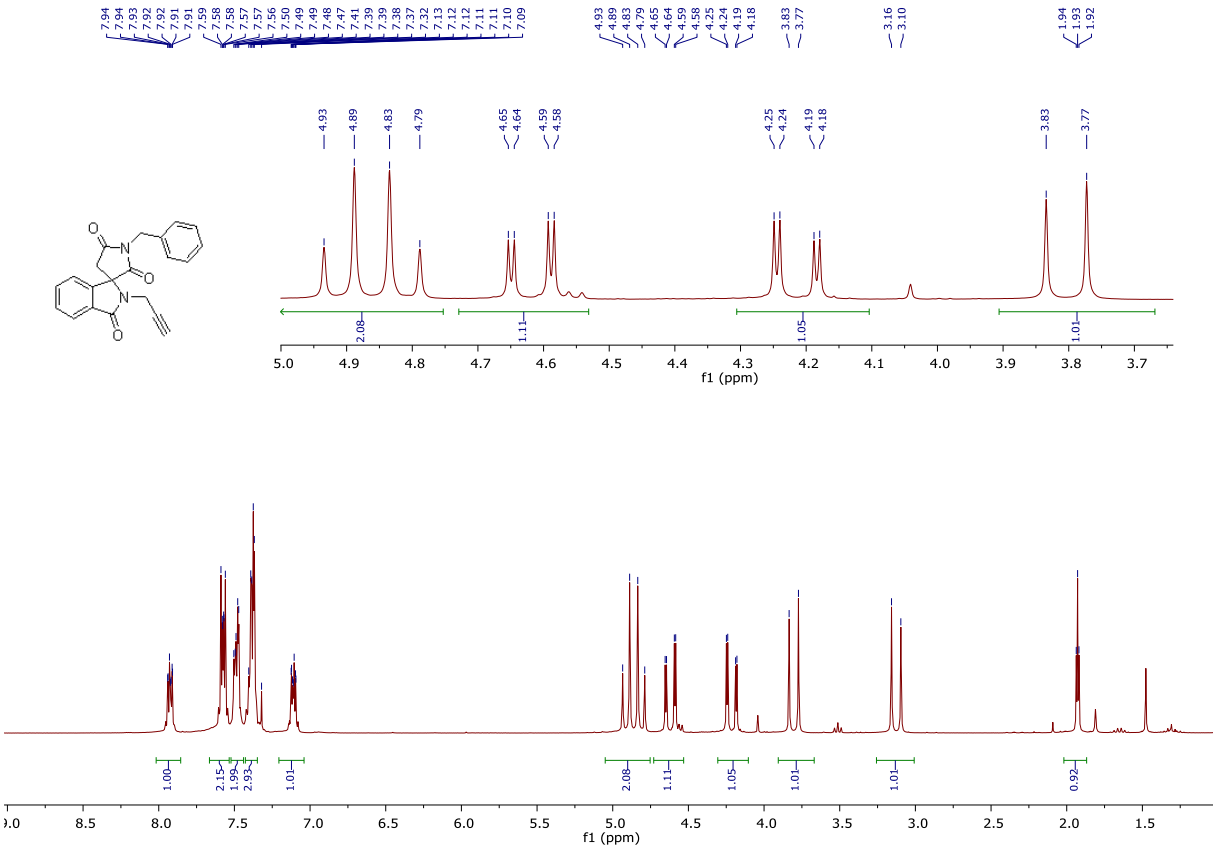
^{13}C NMR spectrum of compounds **(±)-8f(A,B)** and **(±)-9f(A,B)**



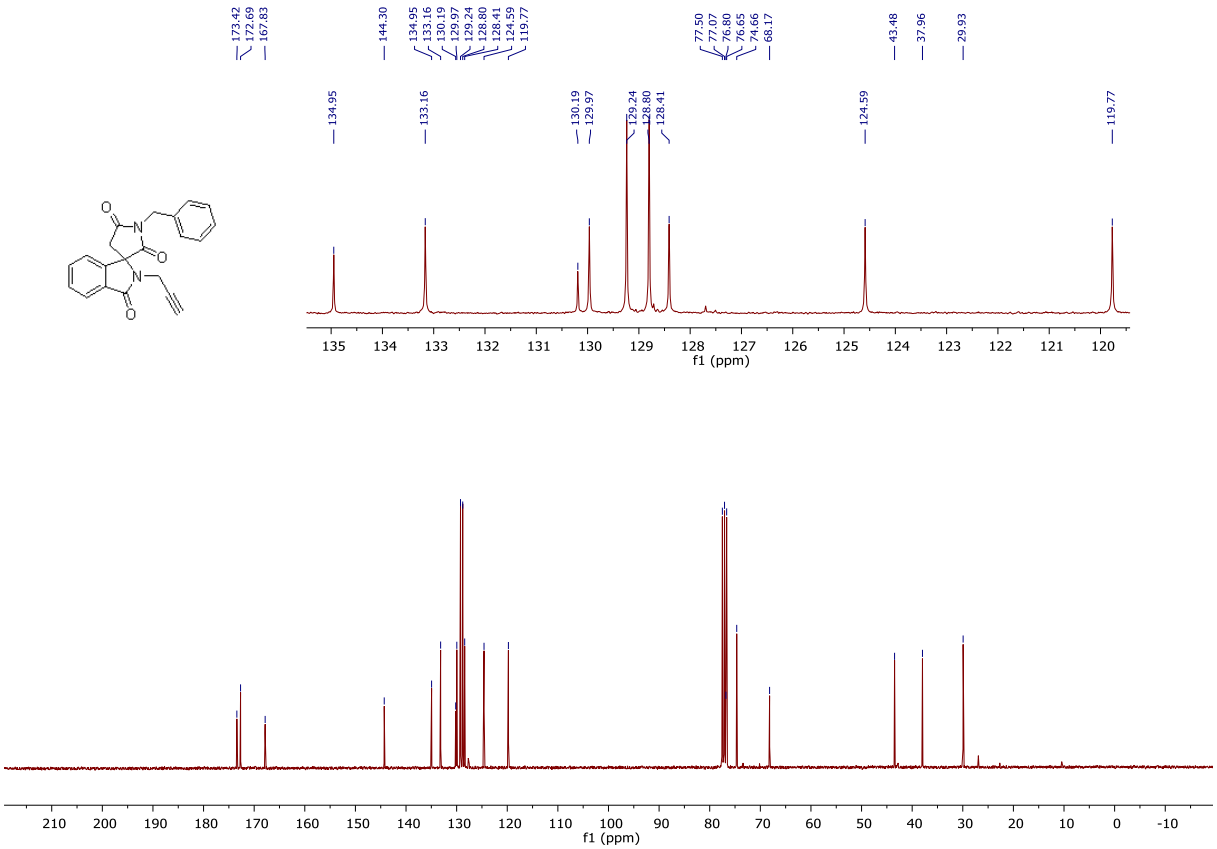
^{1}H NMR spectrum of compound **(±)-4f**



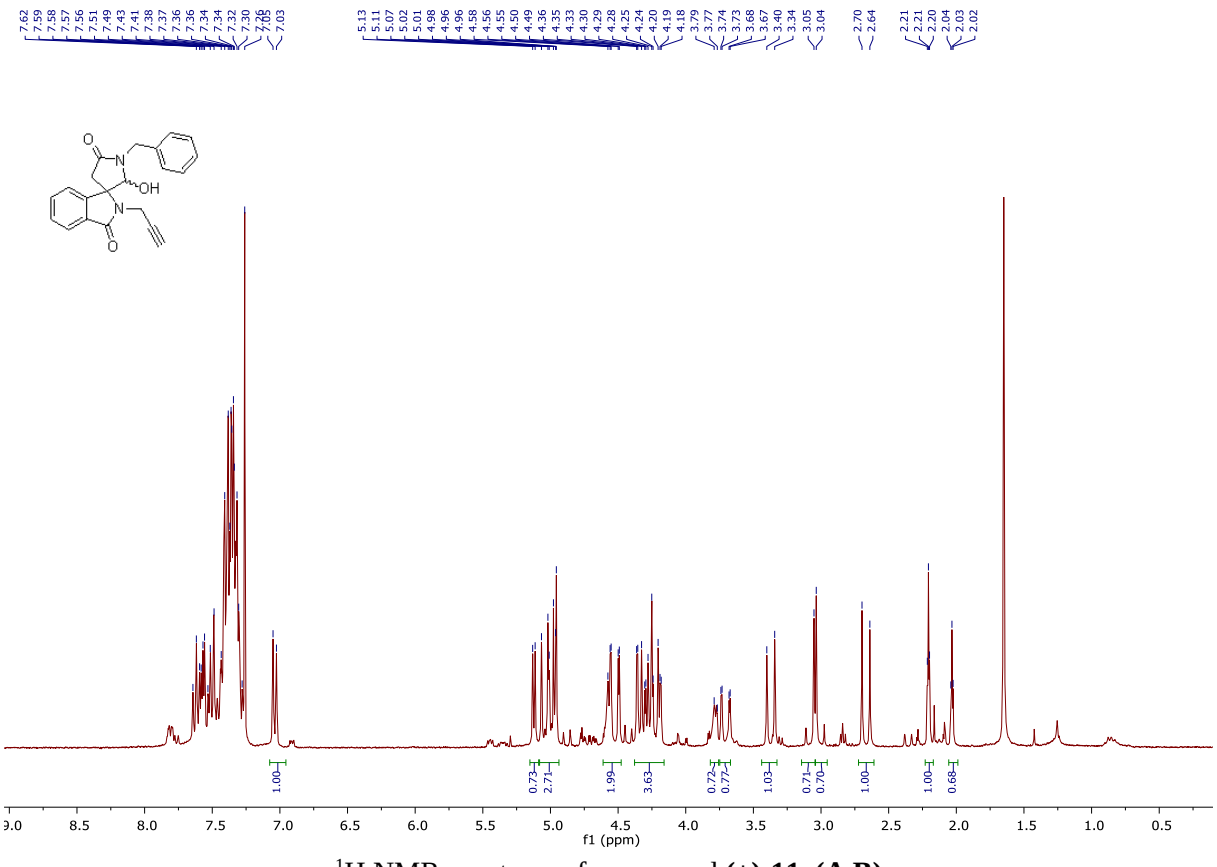
^{13}C NMR spectrum of compound **(±)-4f**



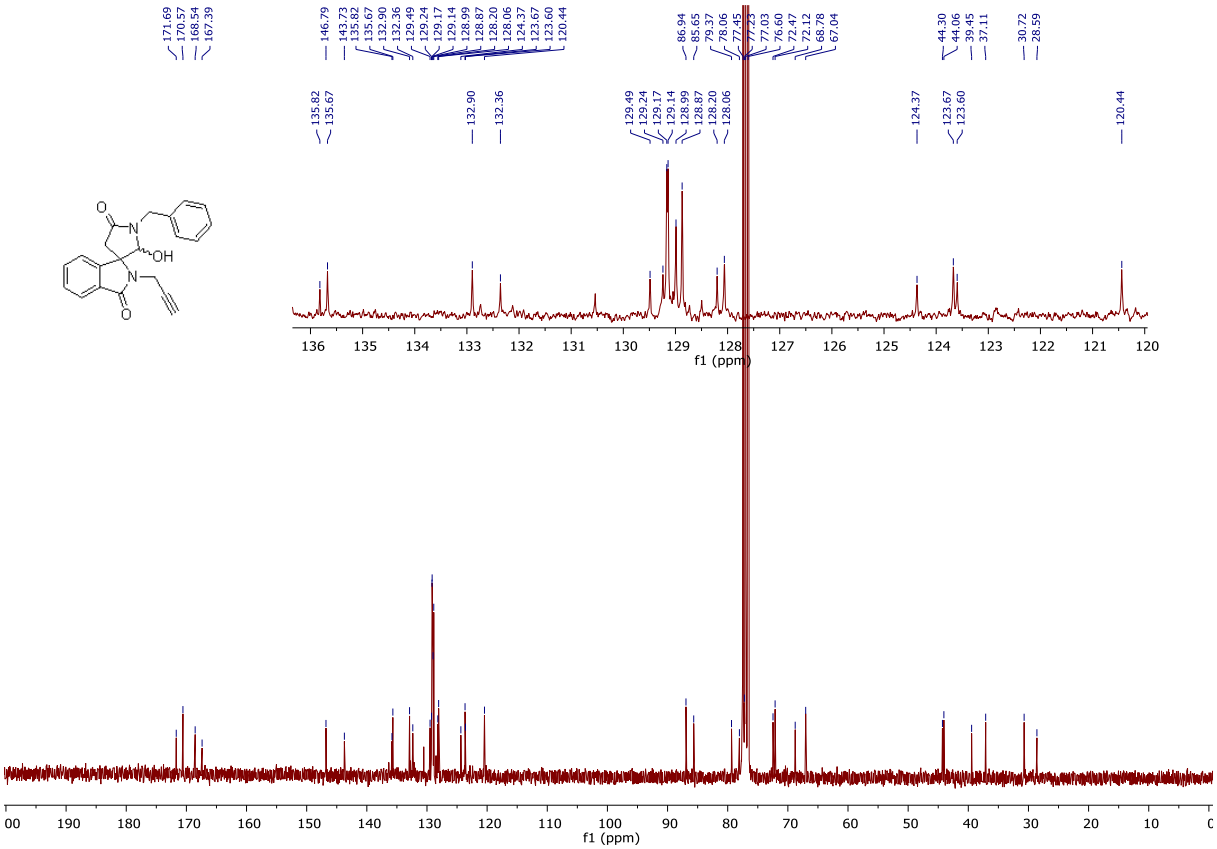
^{1}H NMR spectrum of compound **(±)-1g**



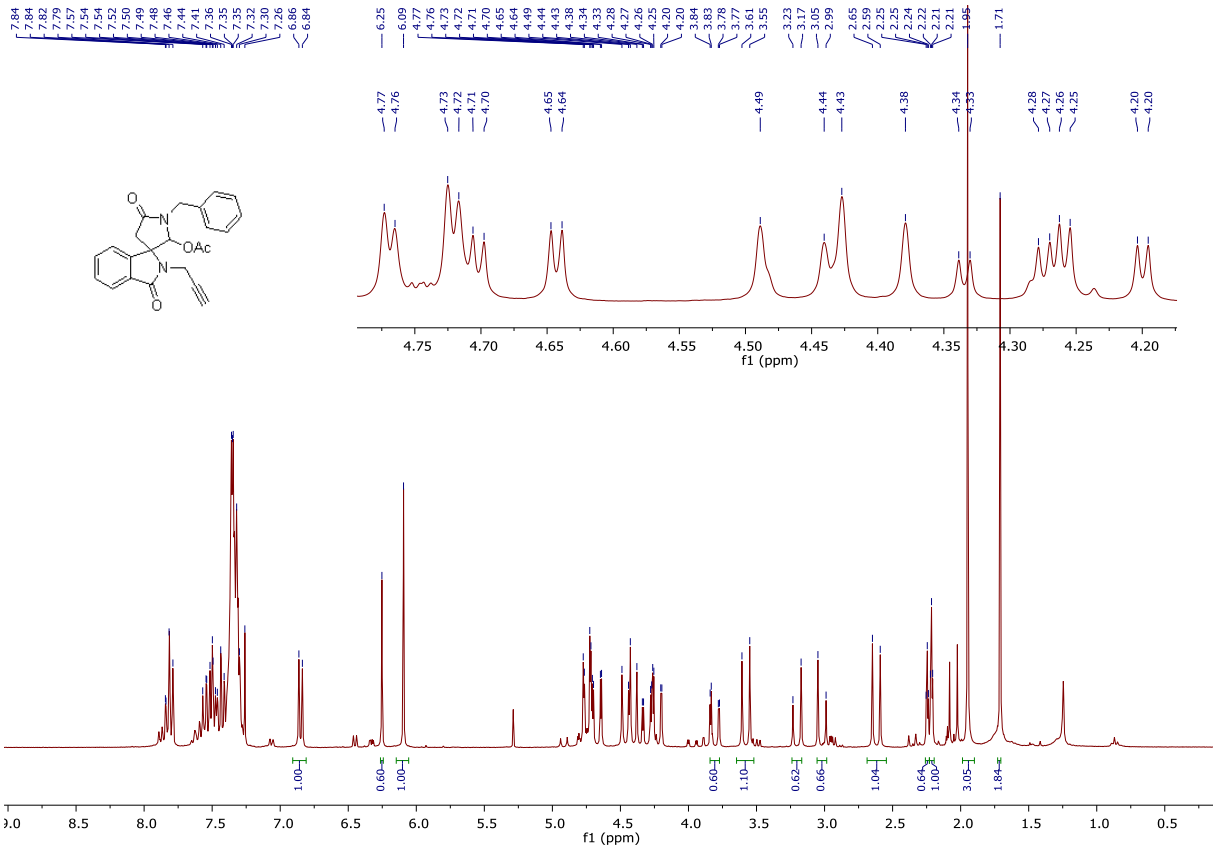
^{13}C NMR spectrum of compound **(±)-1g**



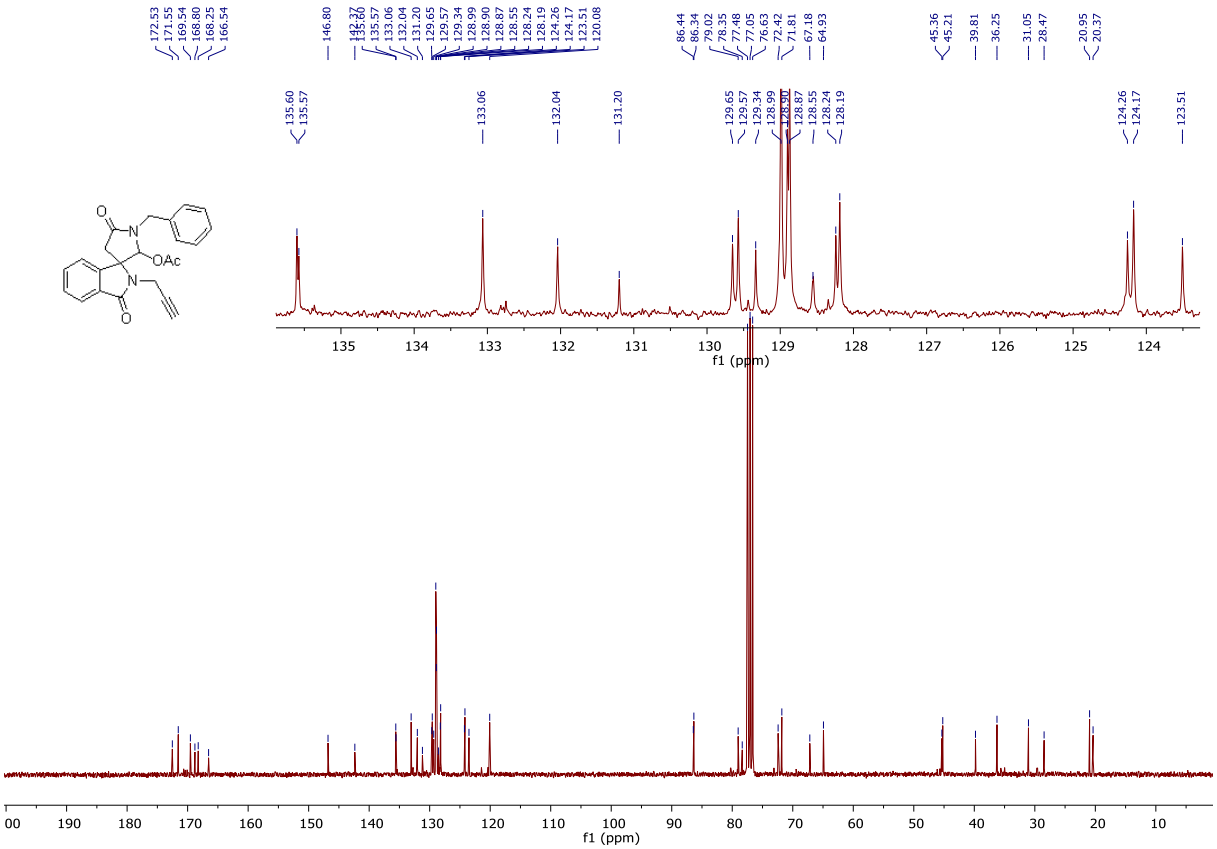
^{1}H NMR spectrum of compound **(±)-11g(A,B)**



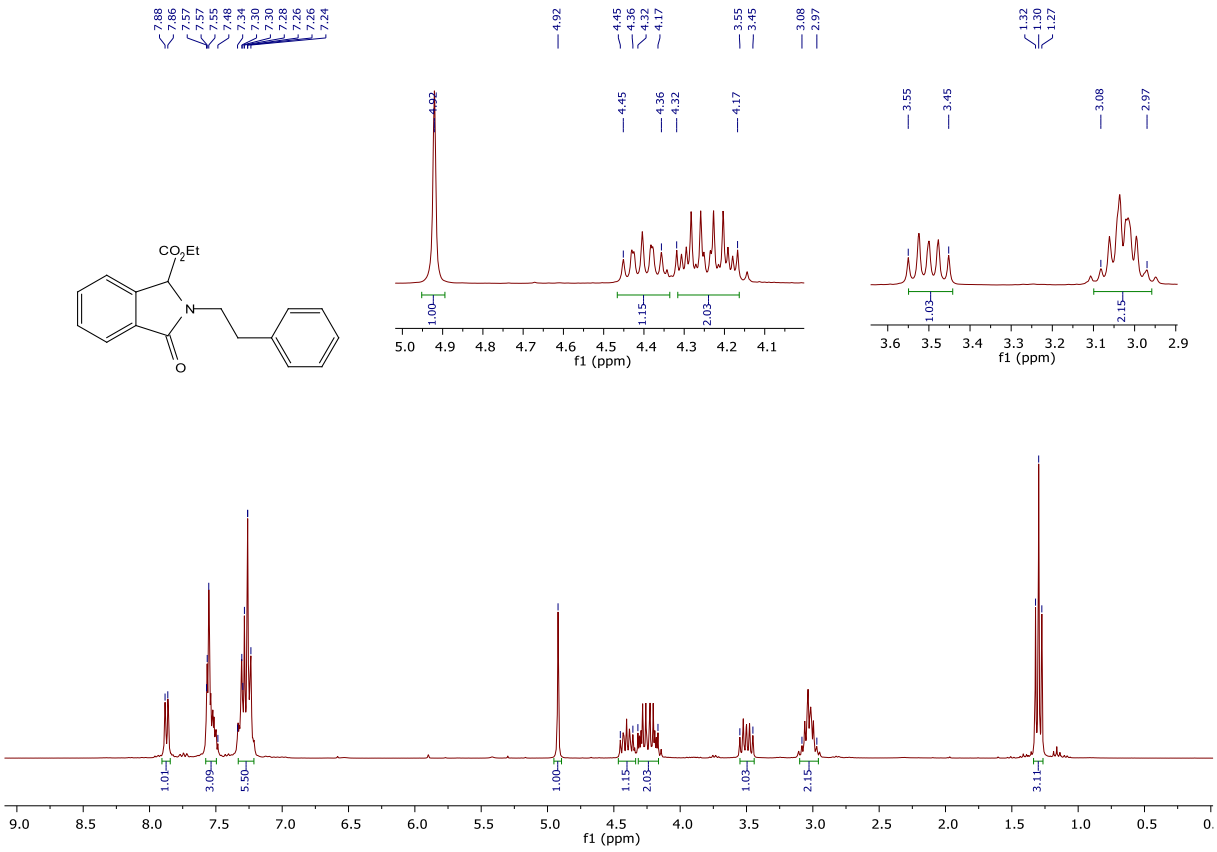
^{13}C NMR spectrum of compound **(±)-11g(A,B)**



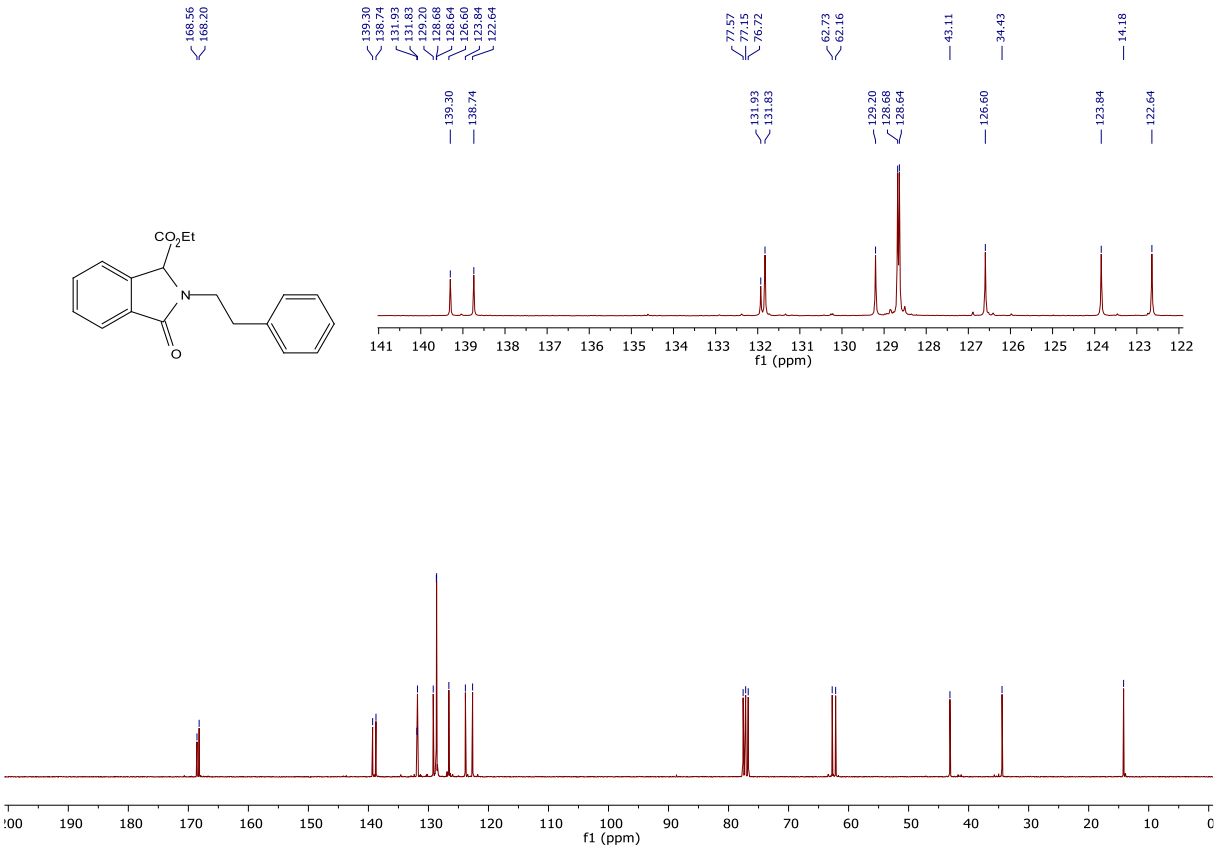
^{1}H NMR spectrum of compound **(±)-8g(A,B)**



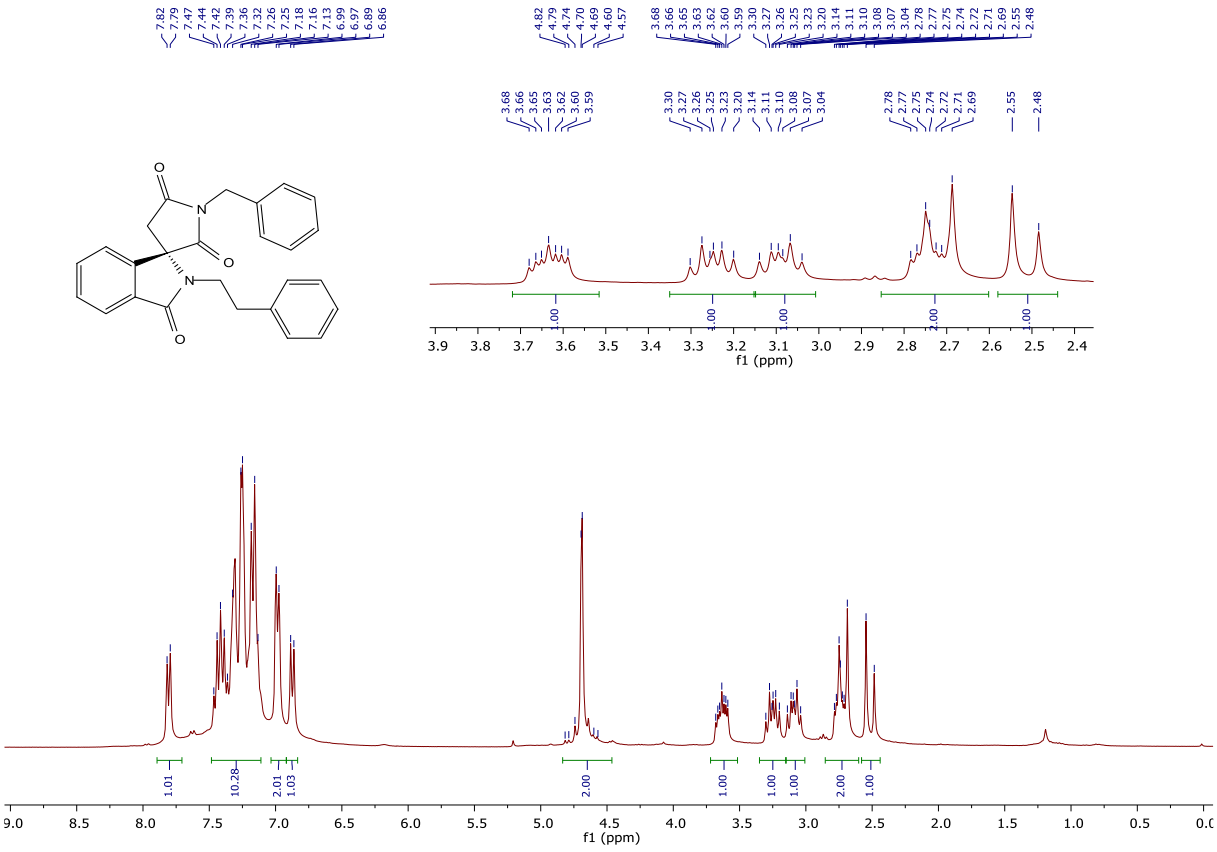
^{13}C NMR spectrum of compound **(±)-8g(A,B)**



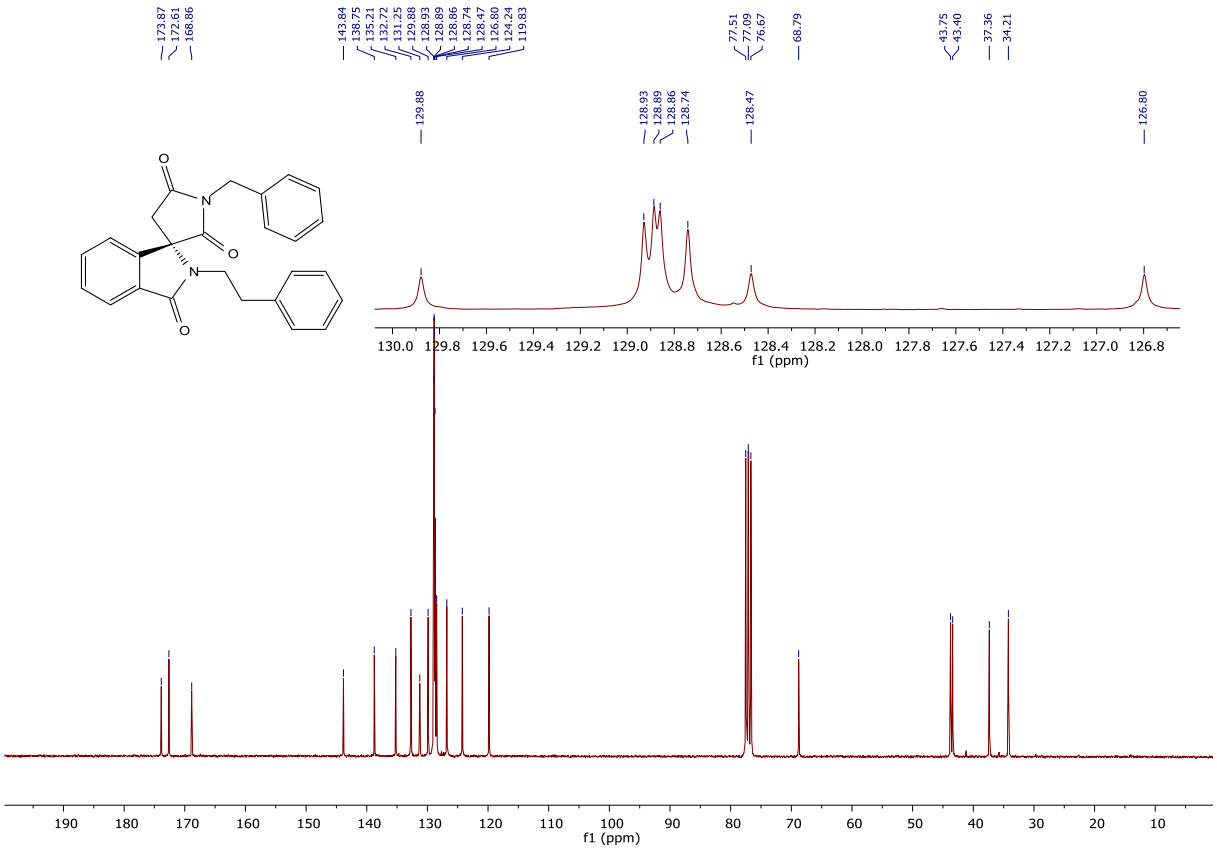
^{1}H NMR spectrum of compound **(±)-7h**



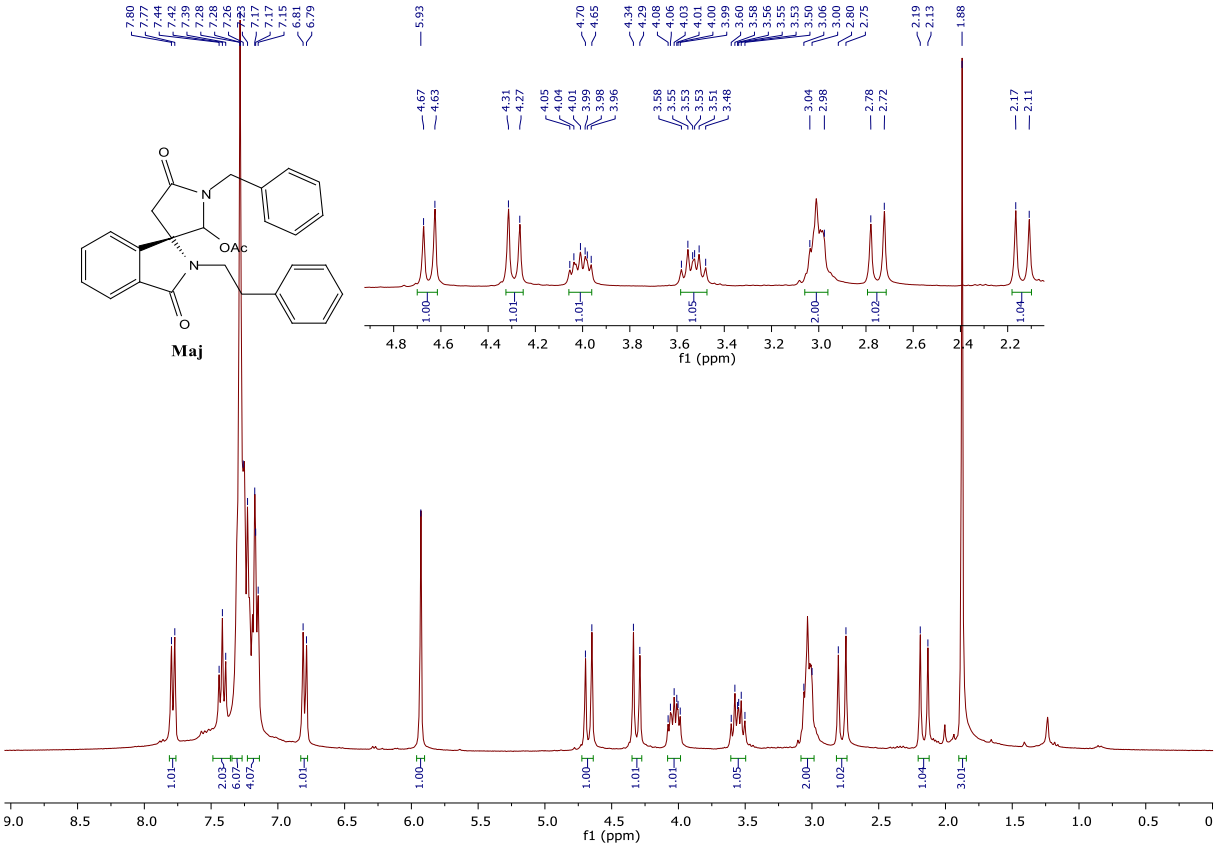
^{13}C NMR spectrum of compound **(±)-7h**



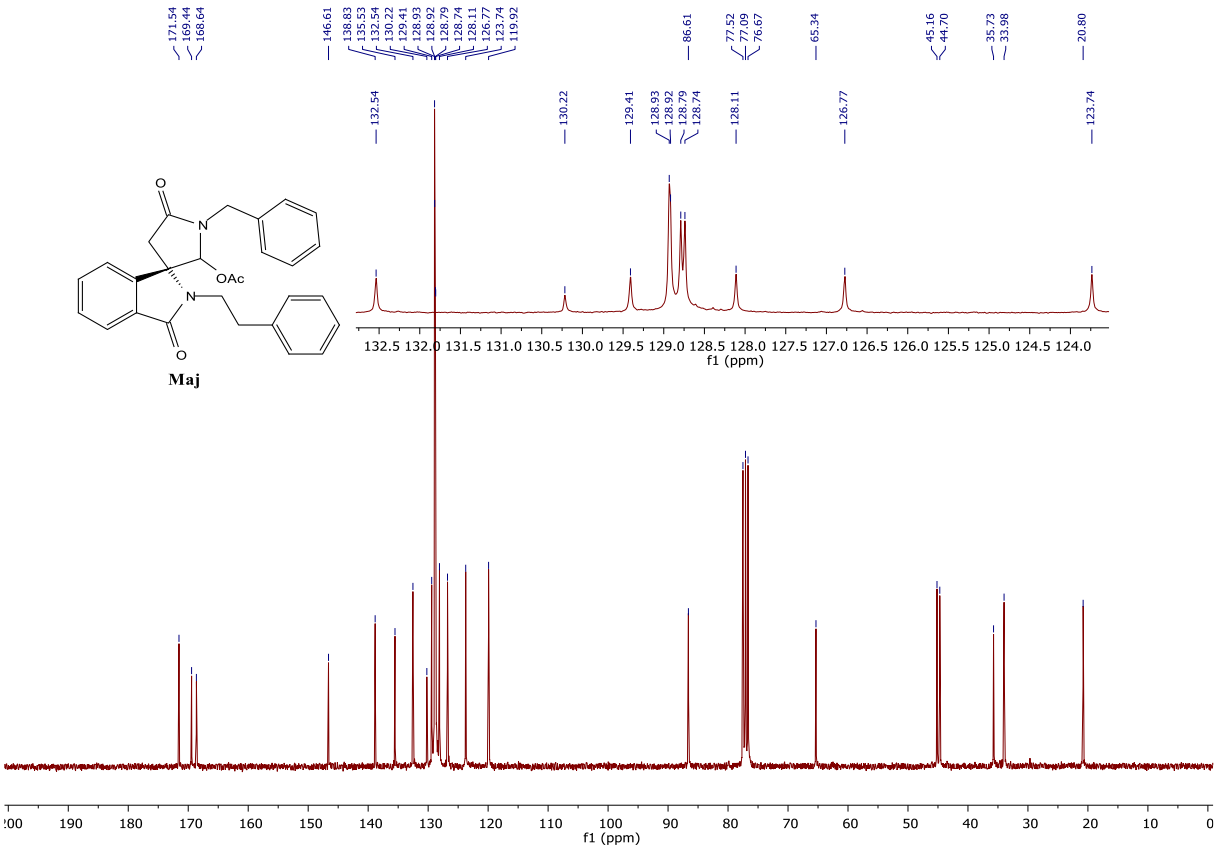
^{1}H NMR spectrum of compound **(±)-1h**



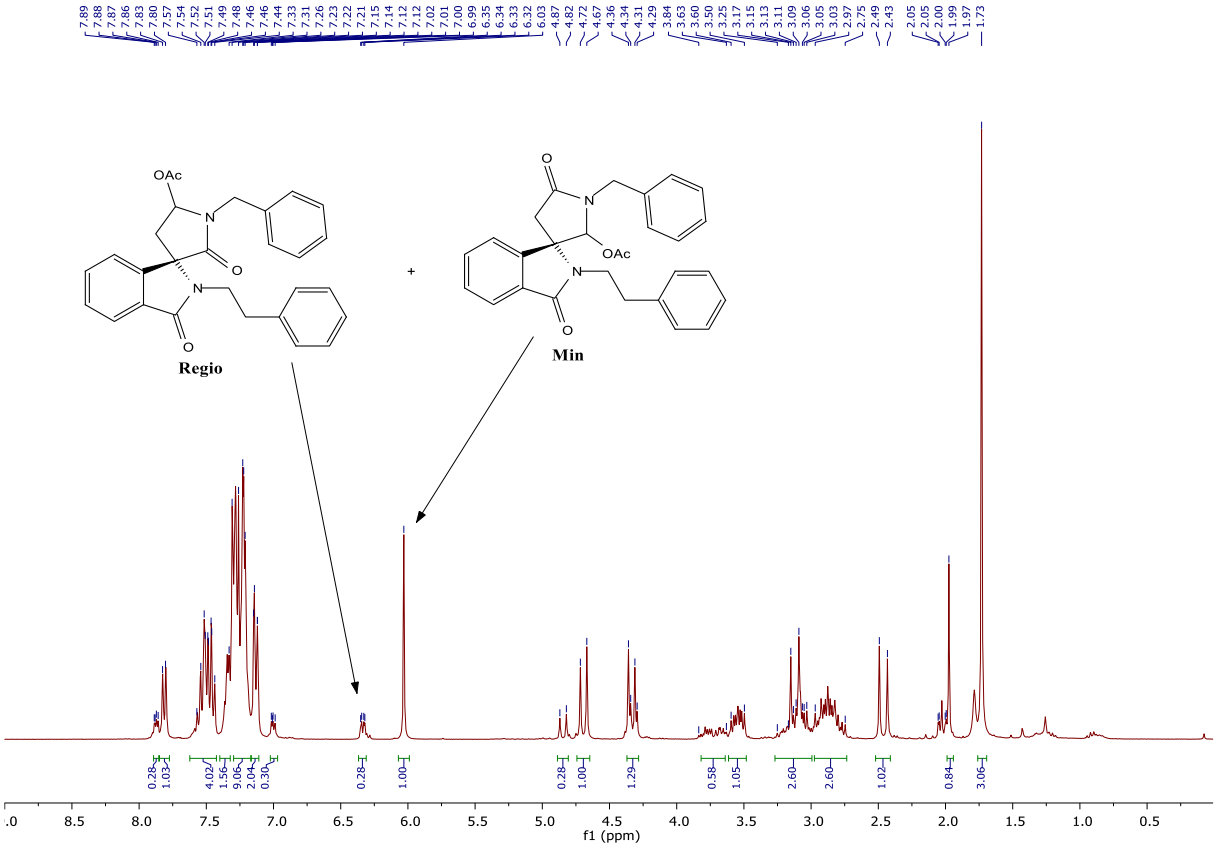
^{13}C NMR spectrum of compound **(±)-1h**



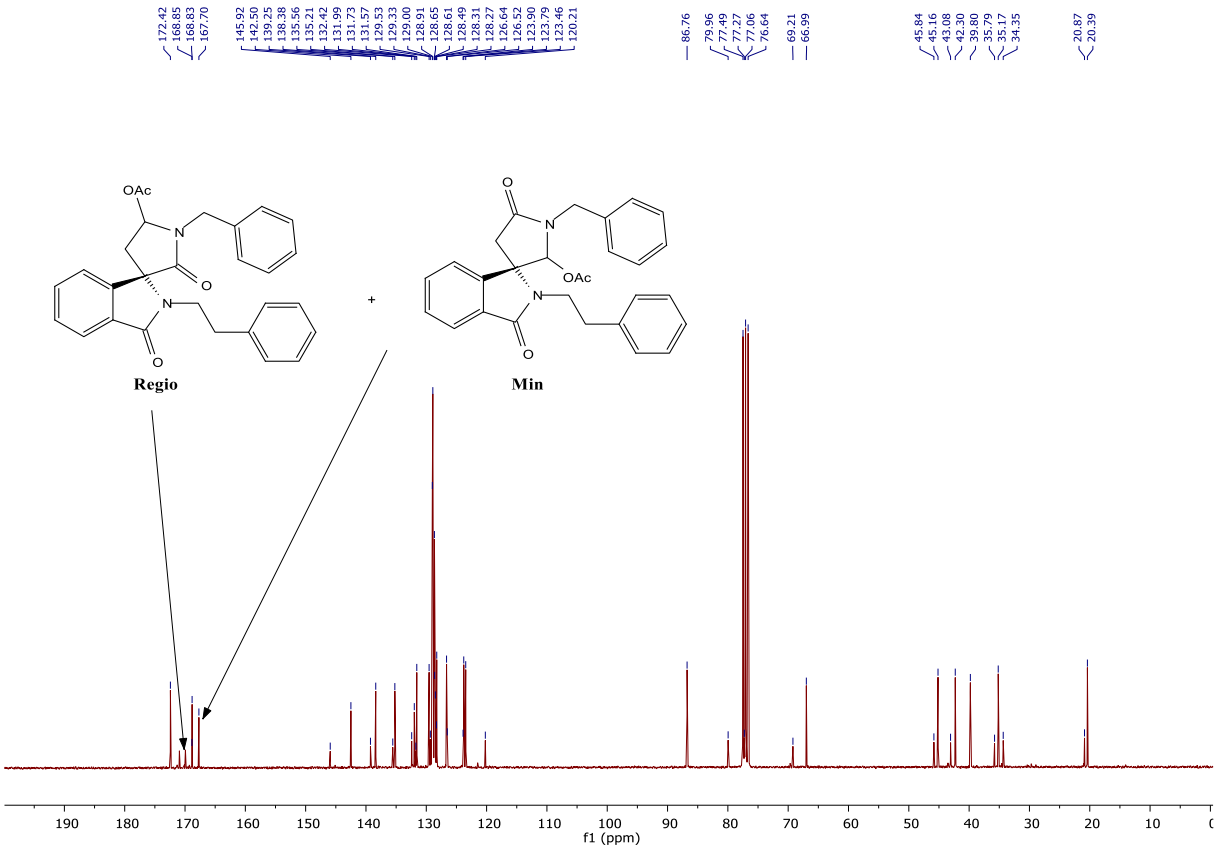
^{1}H NMR spectrum of compound **(±)-8hA**



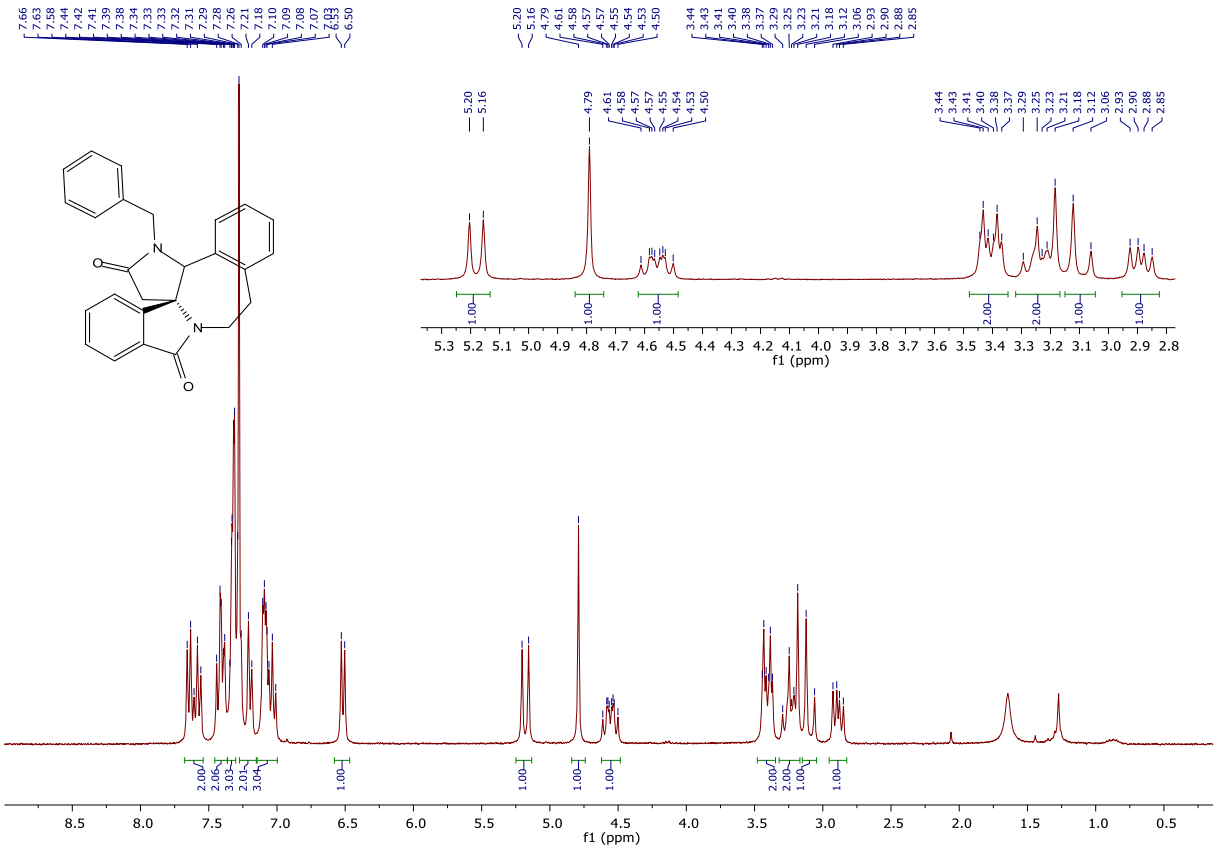
^{13}C NMR spectrum of compound **(±)-8hA**



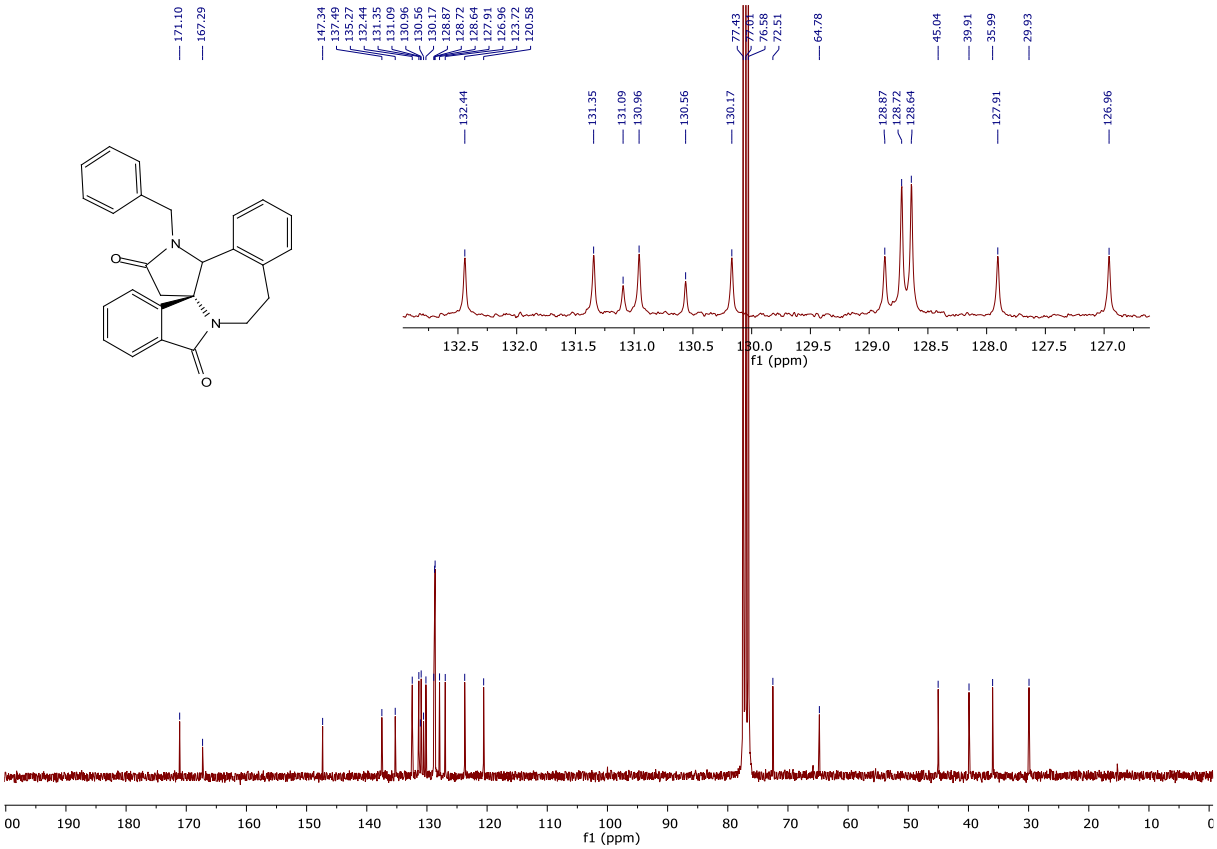
^{1}H NMR spectrum of compounds **(±)-8hB** and **(±)-9h(A,B)**



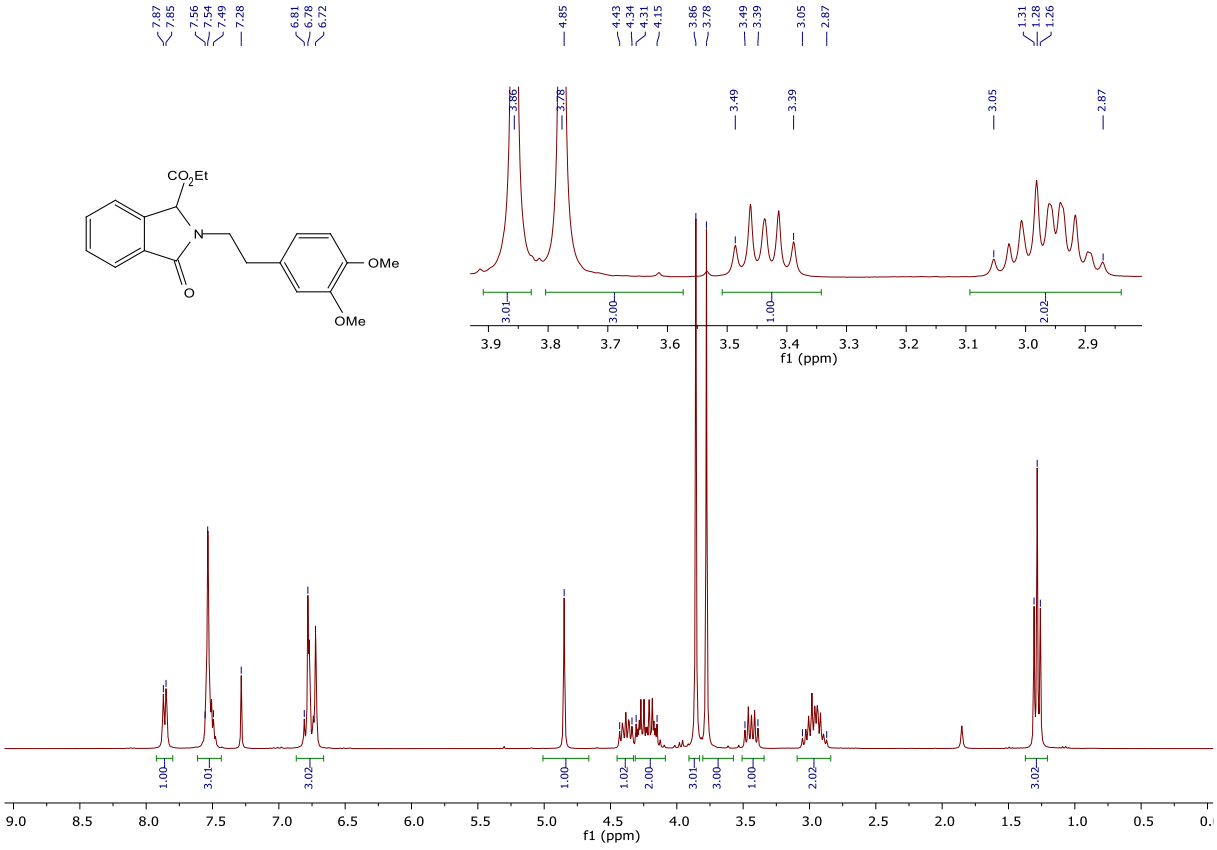
^{13}C NMR spectrum of compounds **(±)-8hB** and **(±)-9h(A,B)**



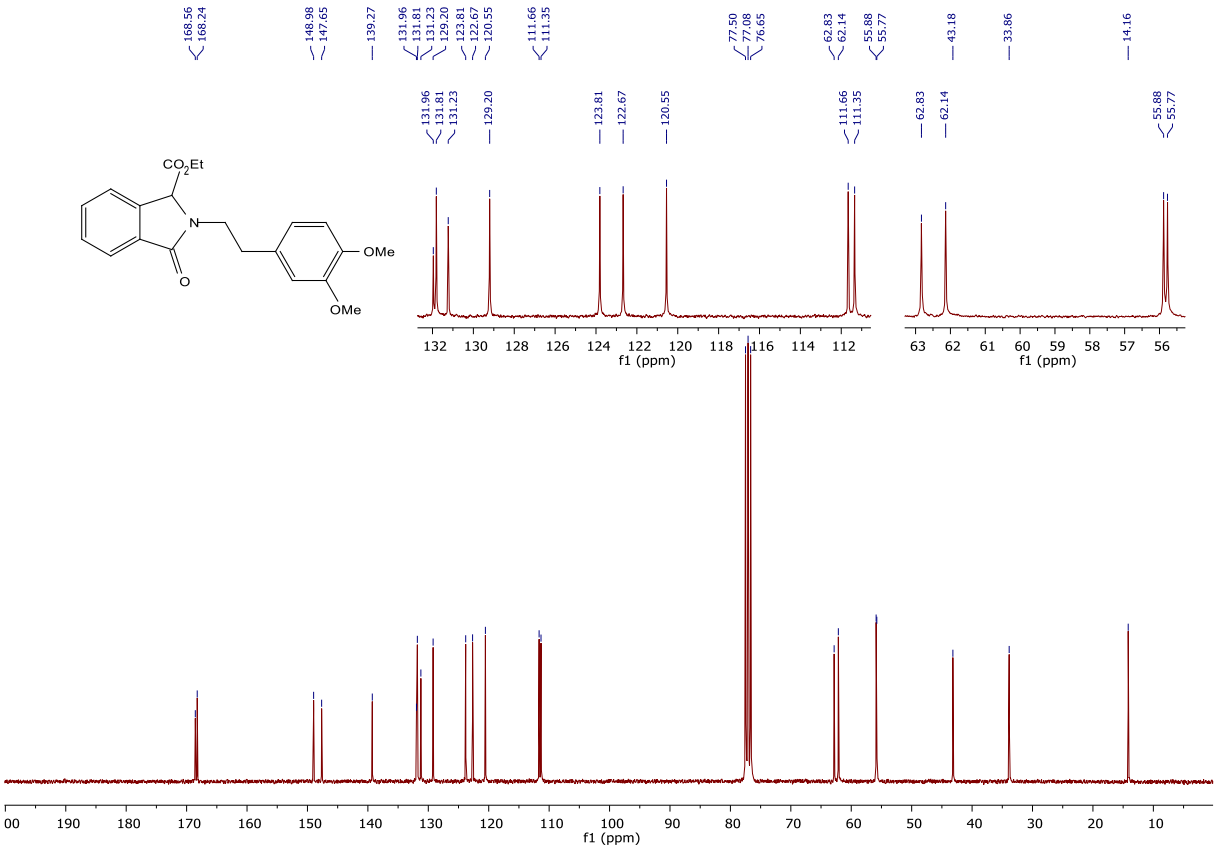
^{1}H NMR spectrum of compound **(±)-5hA**



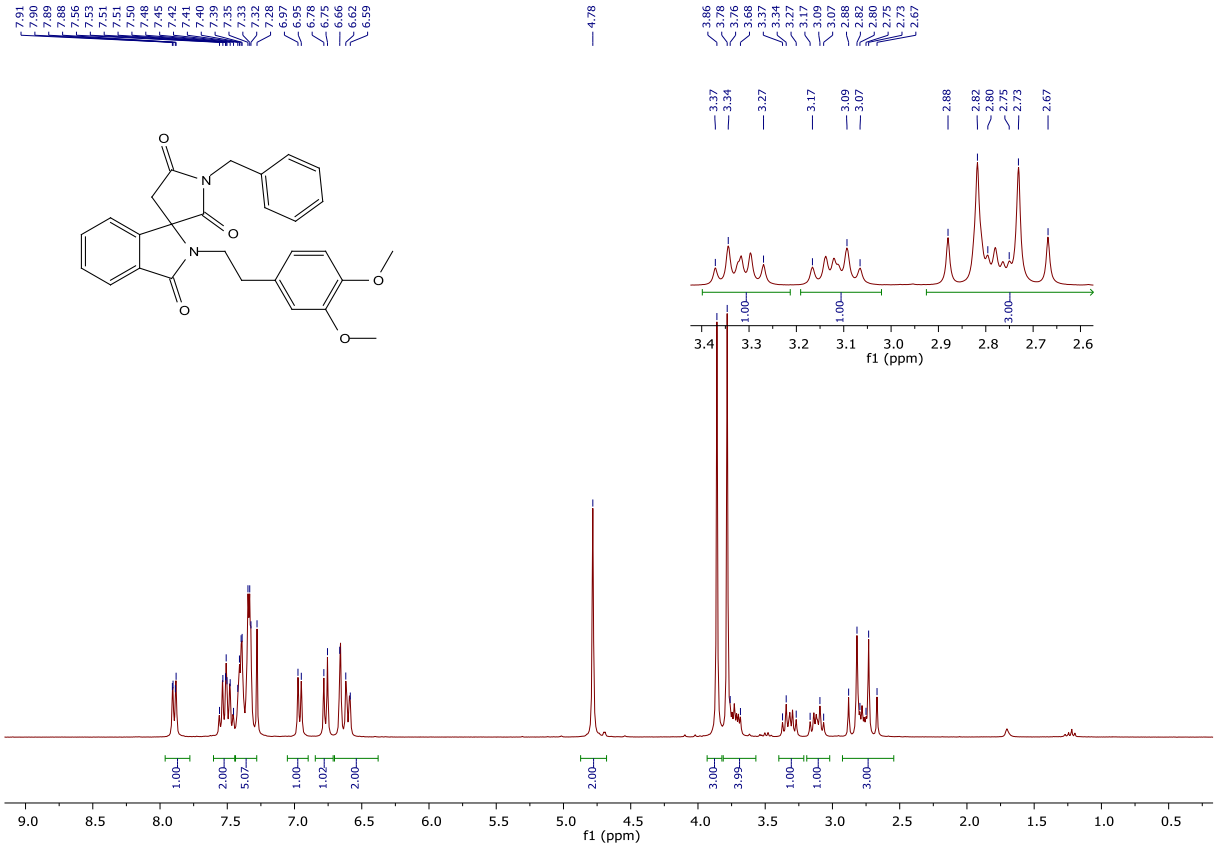
^{13}C NMR spectrum of compound **(±)-5hA**



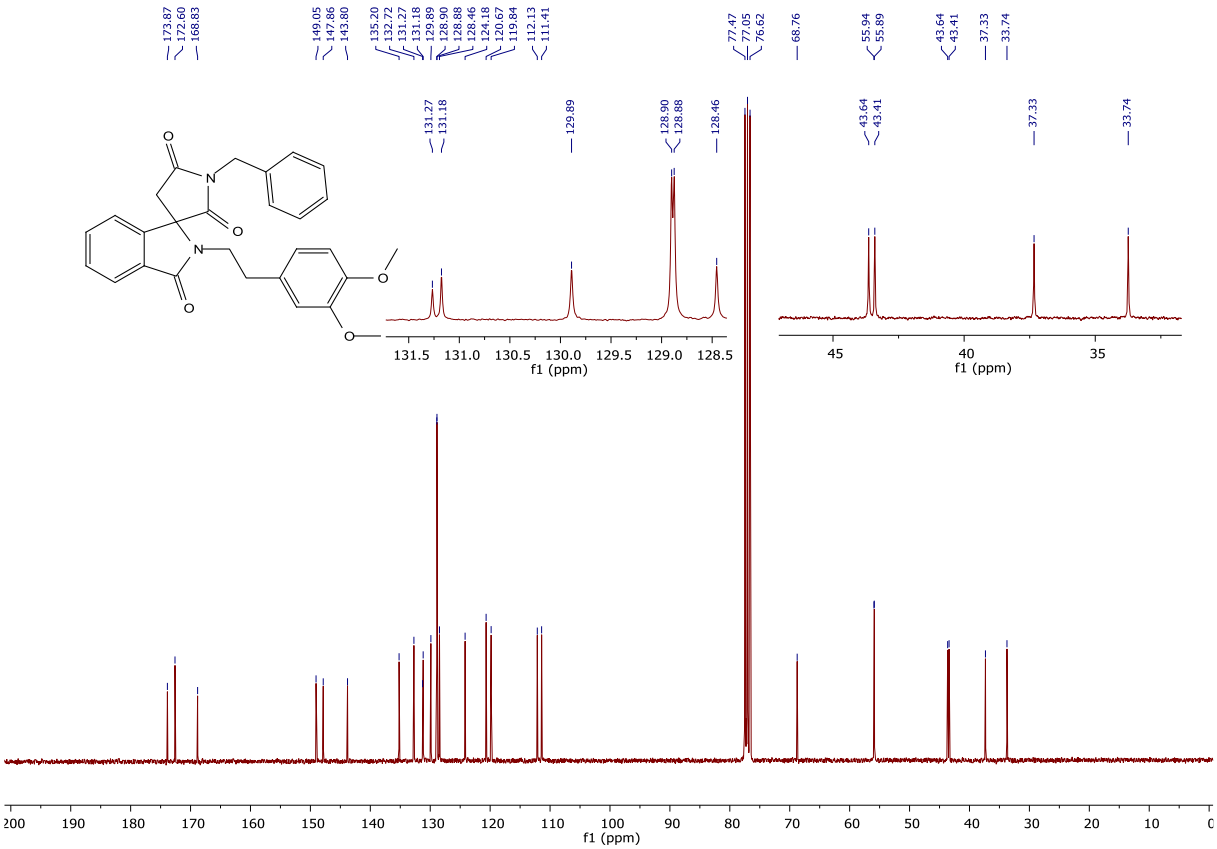
^{1}H NMR spectrum of compound **(±)-7i**



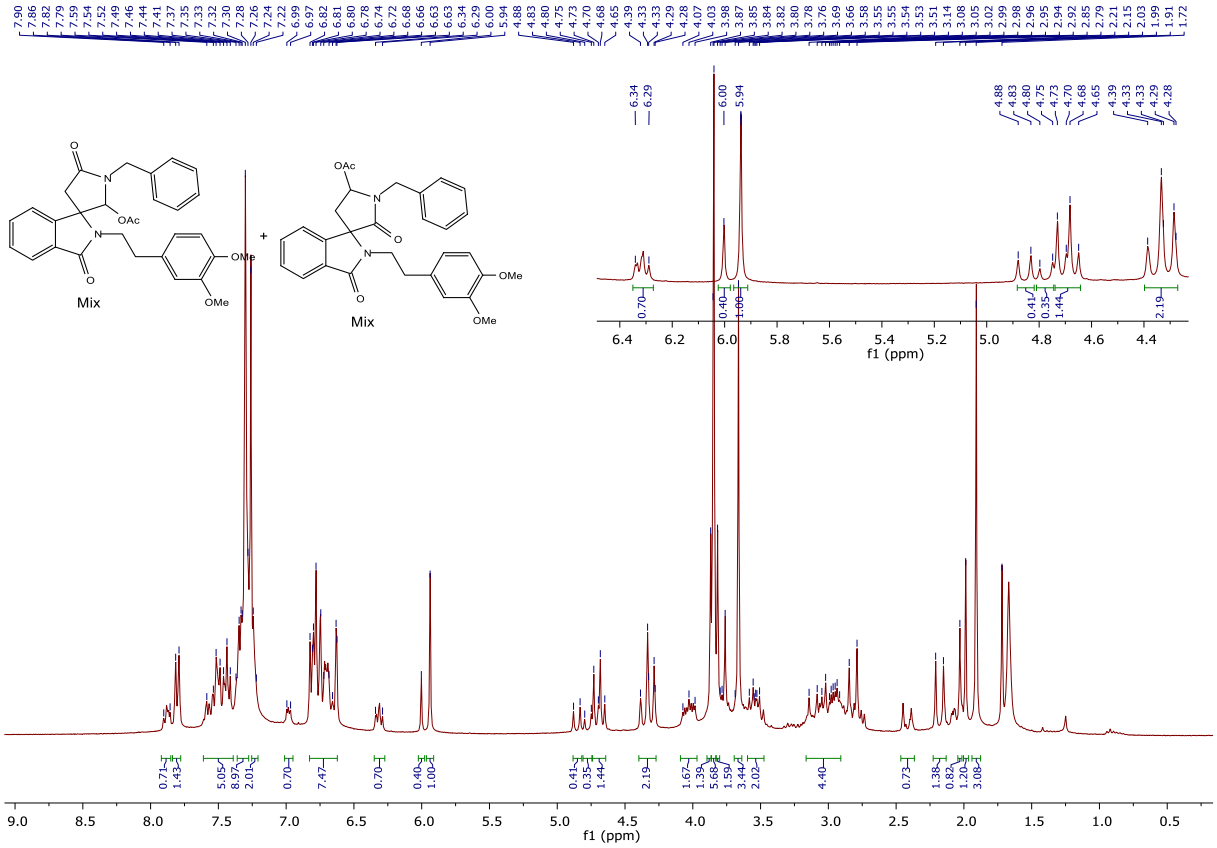
^{13}C NMR spectrum of compound **(±)-7i**



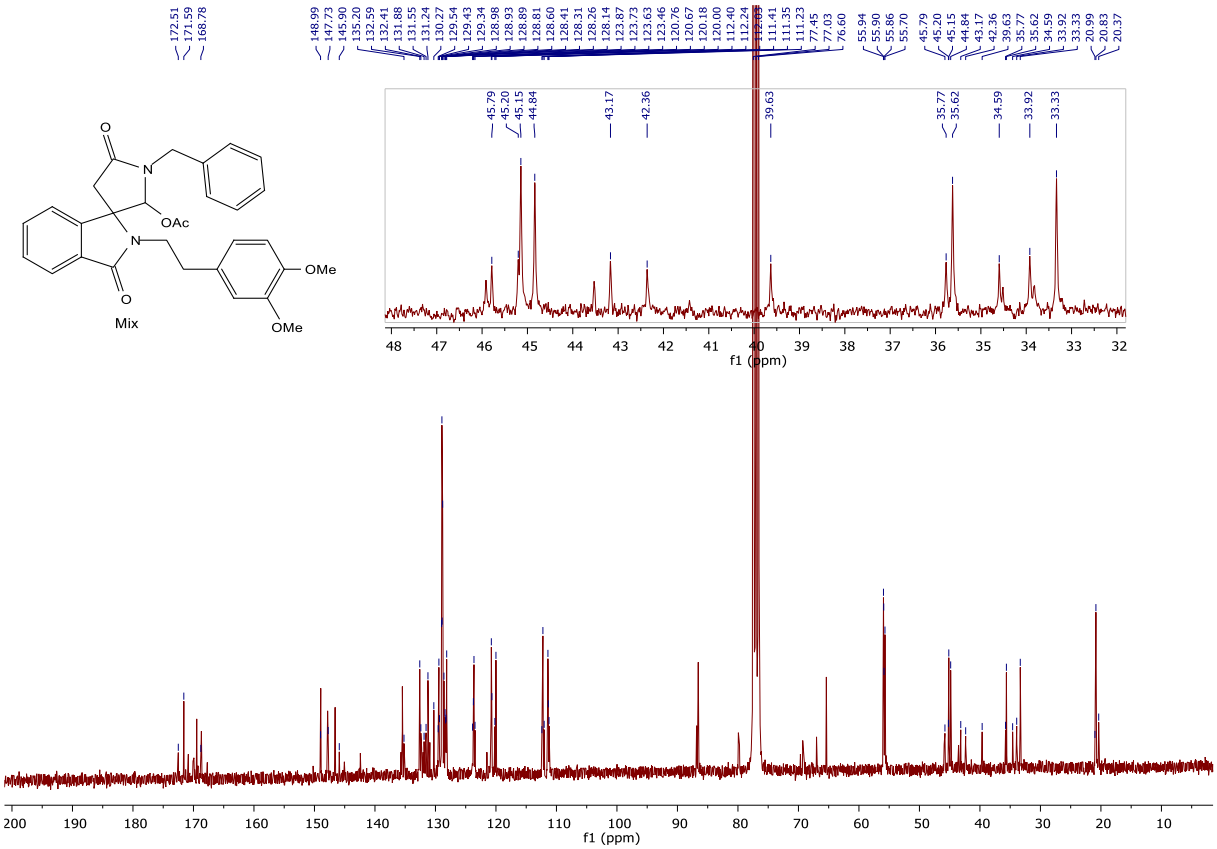
^{1}H NMR spectrum of compound **(±)-1i**



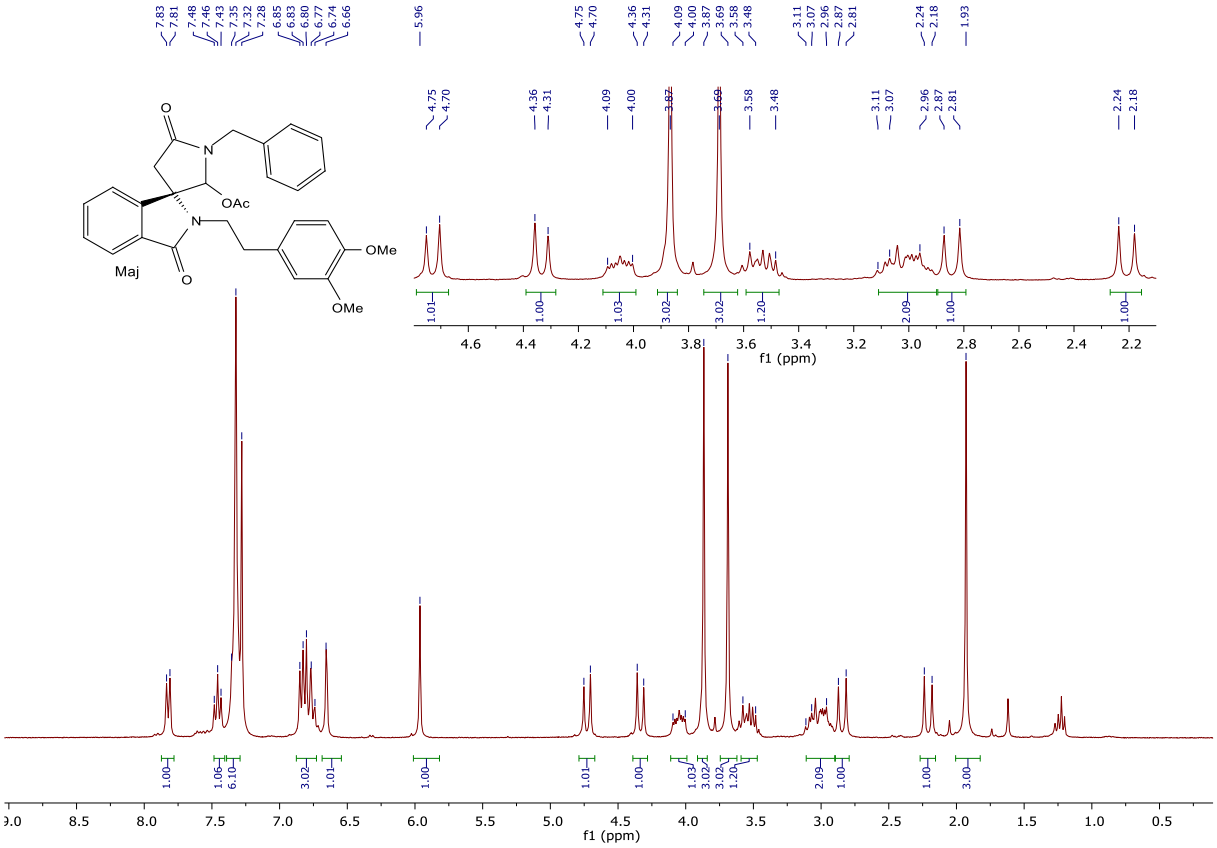
^{13}C NMR spectrum of compound **(±)-1i**



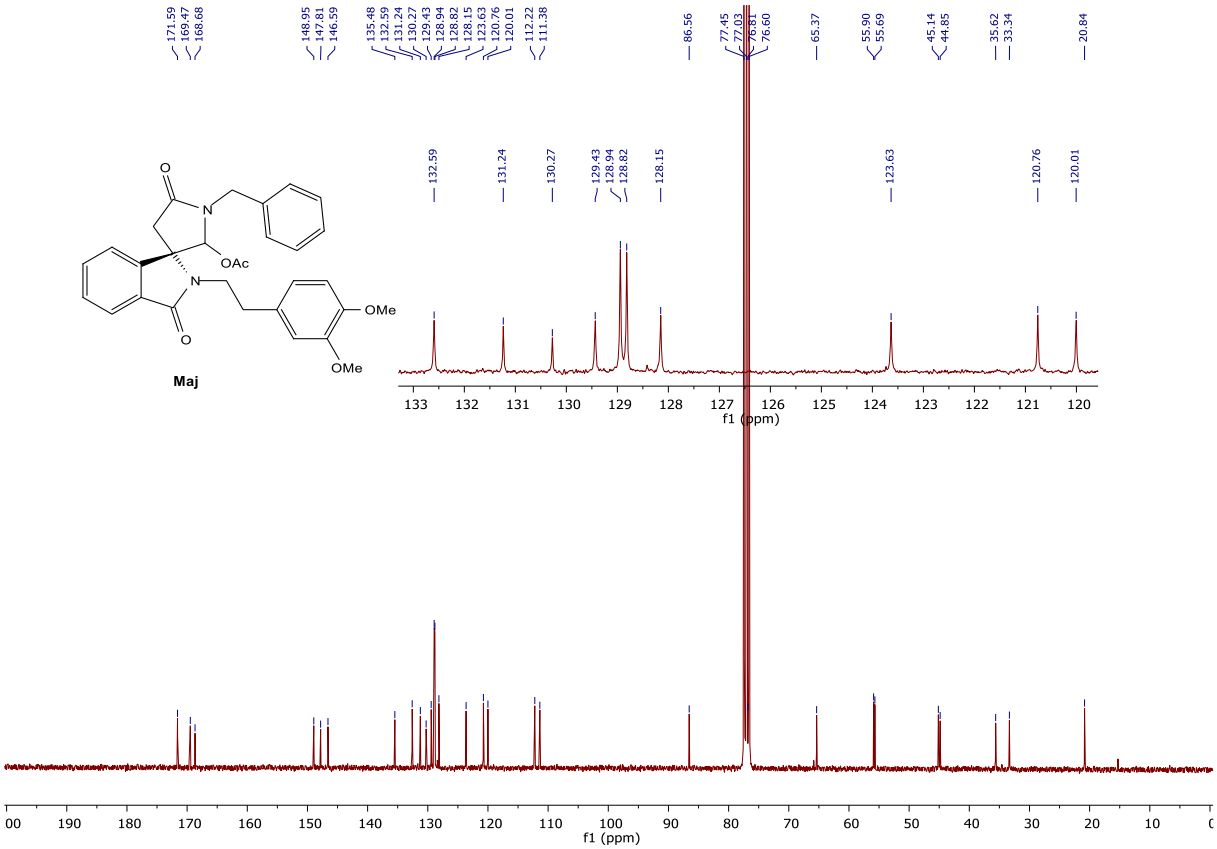
^{1}H NMR spectrum of compounds **(±)-8i(A,B)** and **(±)-9i(A,B)**



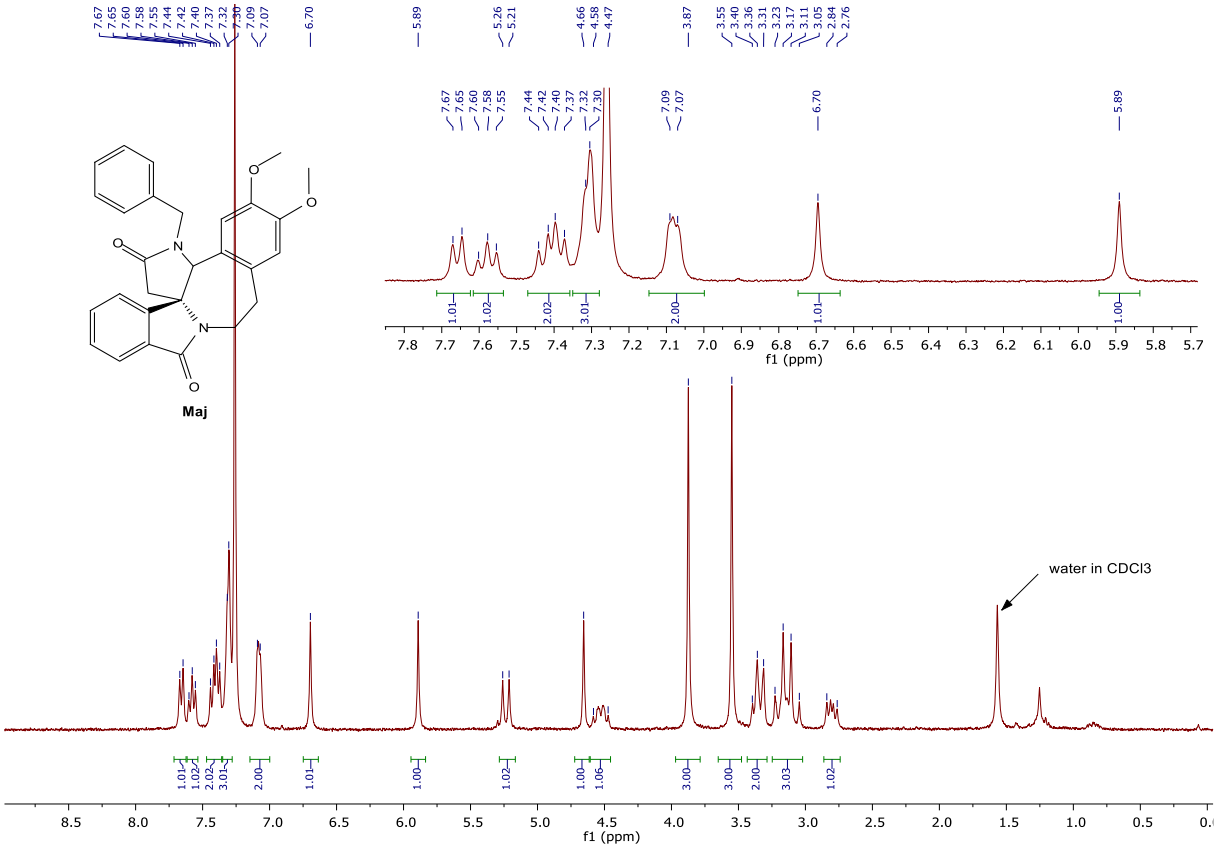
^{13}C NMR spectrum of compound **(±)-8i(A,B)** and **(±)-9i(A,B)**



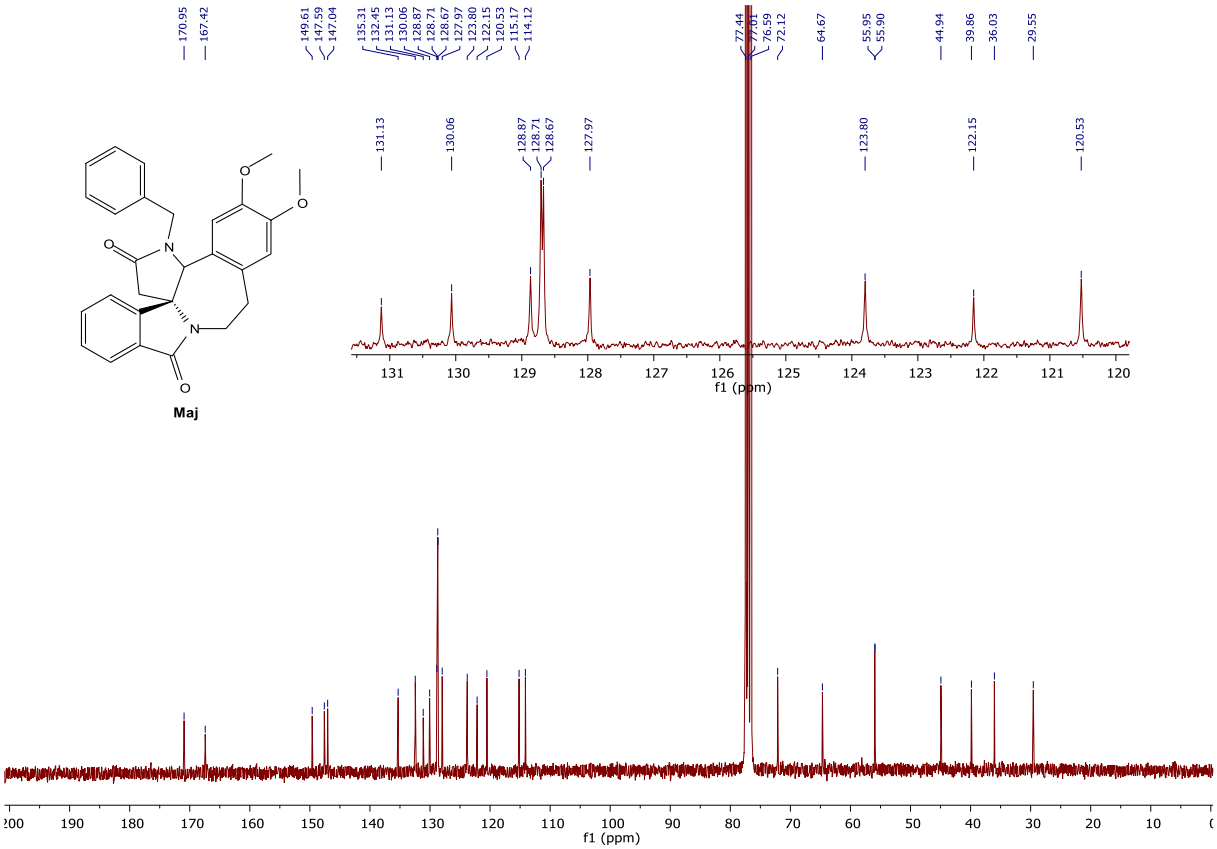
^{1}H NMR spectrum of compound **(±)-8iA**



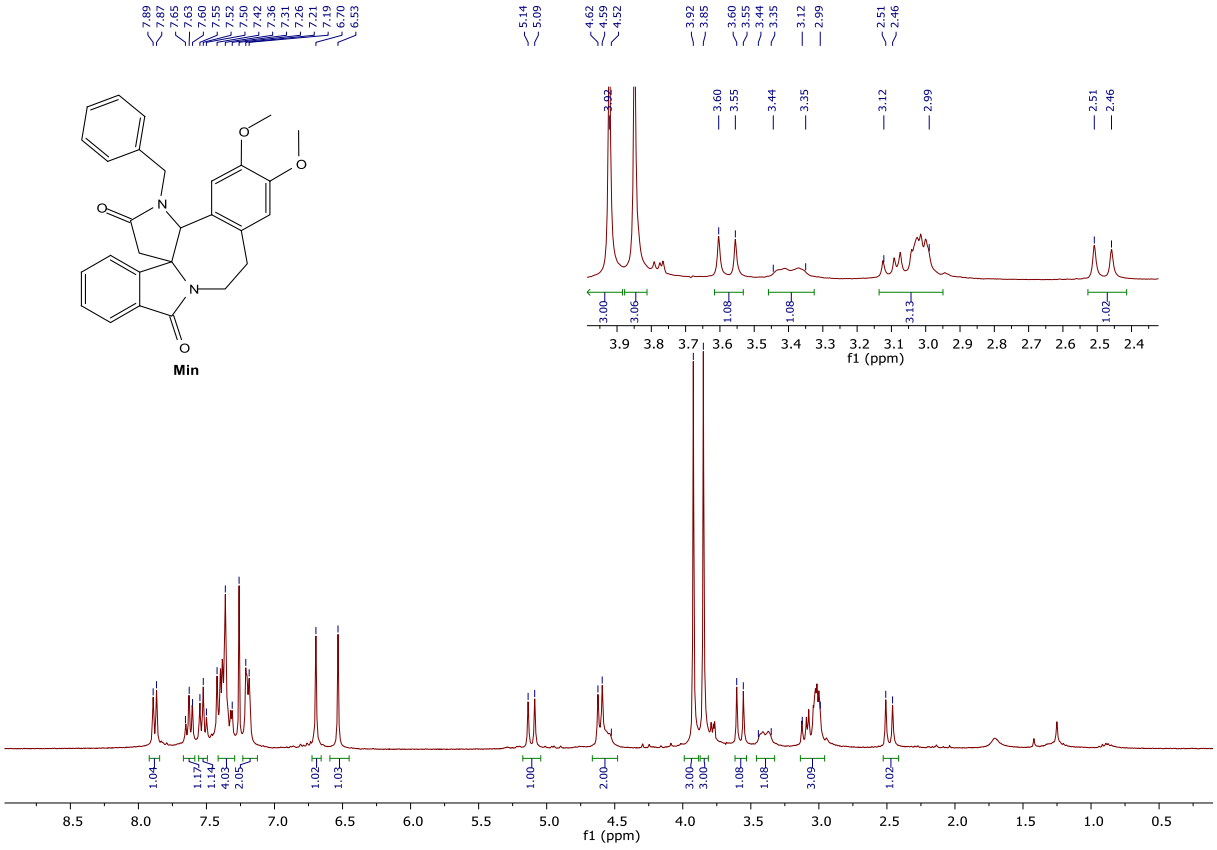
^{13}C NMR spectrum of compound **(±)-8iA**



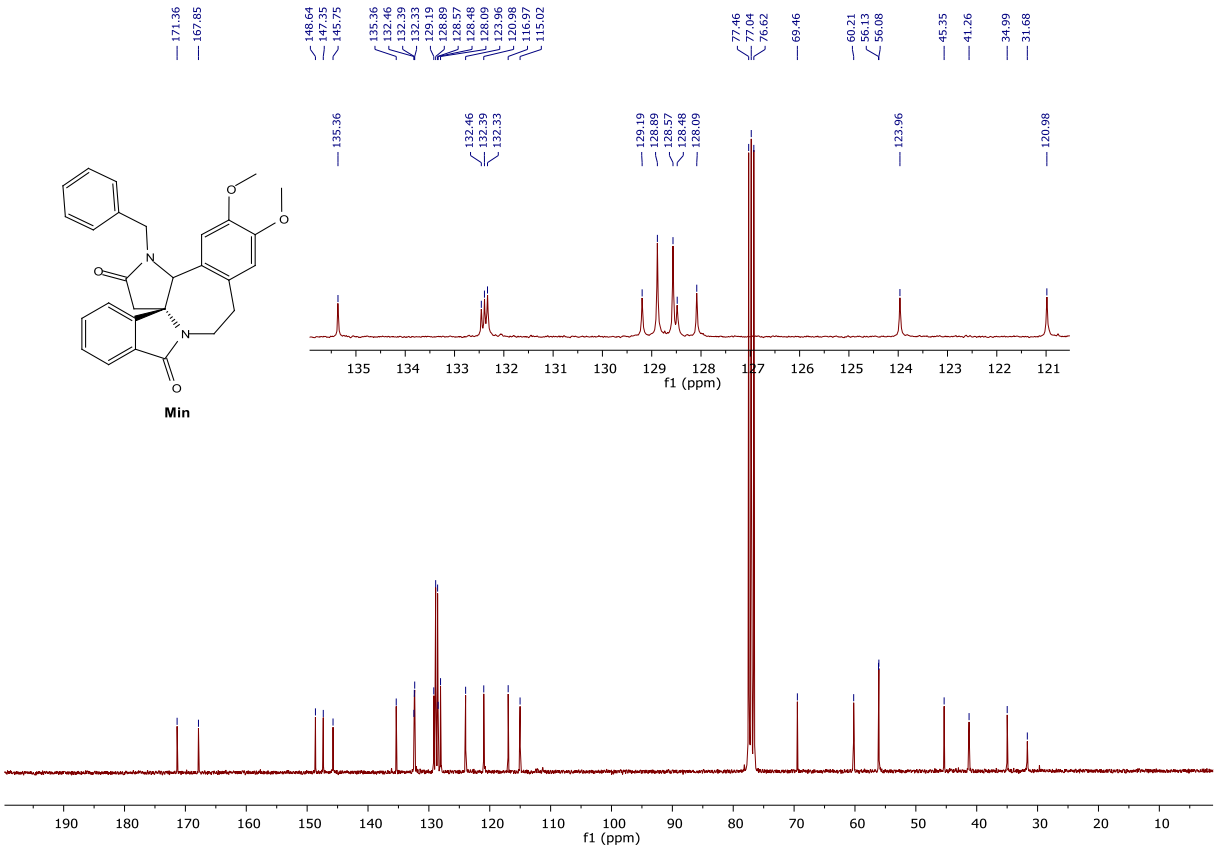
^{1}H NMR spectrum of compound **(±)-5iA**



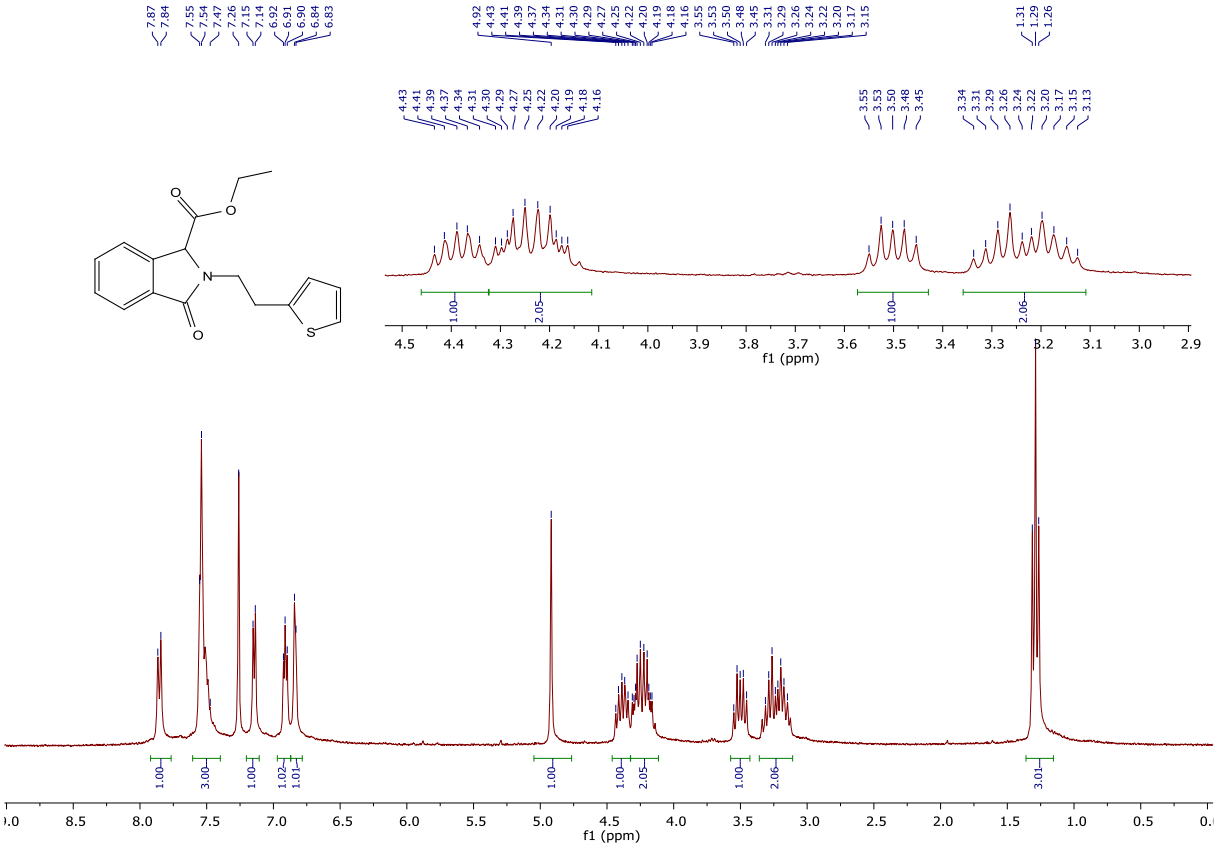
^{13}C NMR spectrum of compound **(±)-5iA**



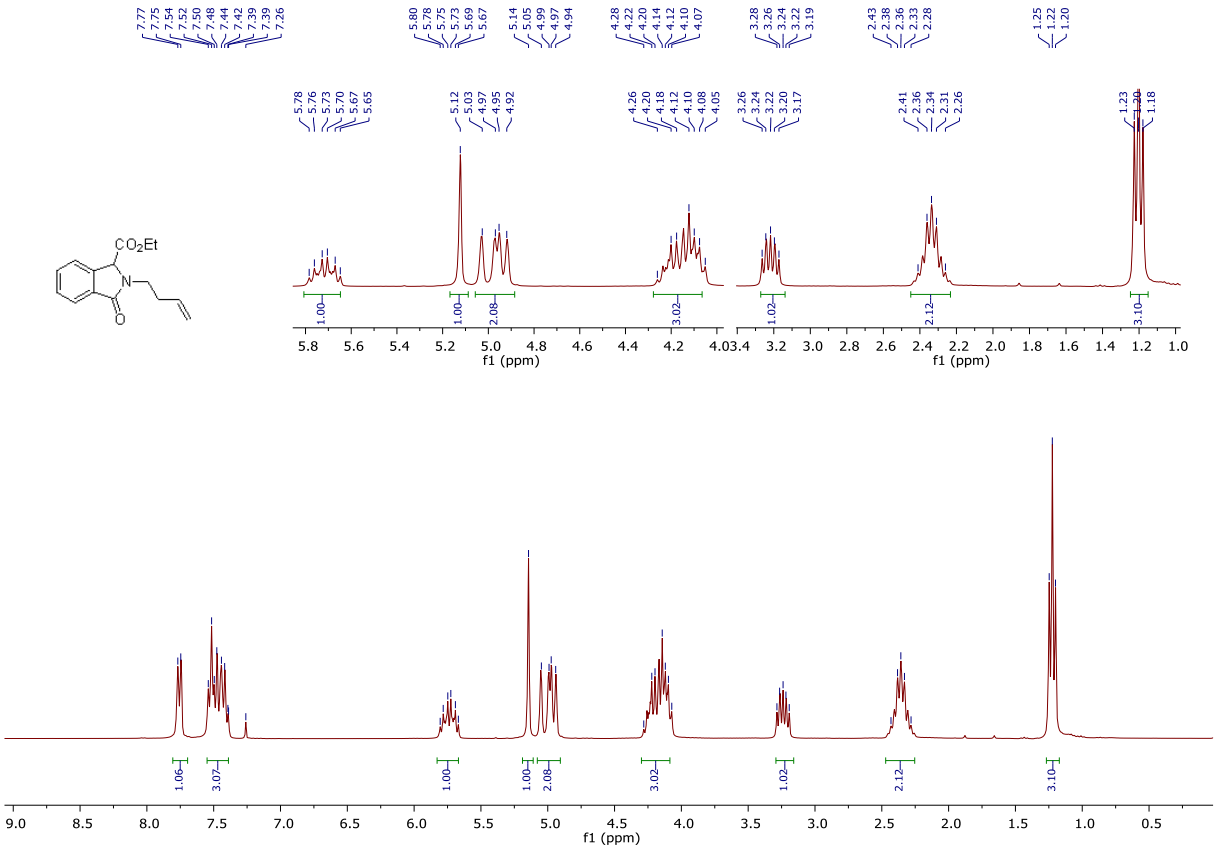
^{1}H NMR spectrum of compound **(±)-5iB**



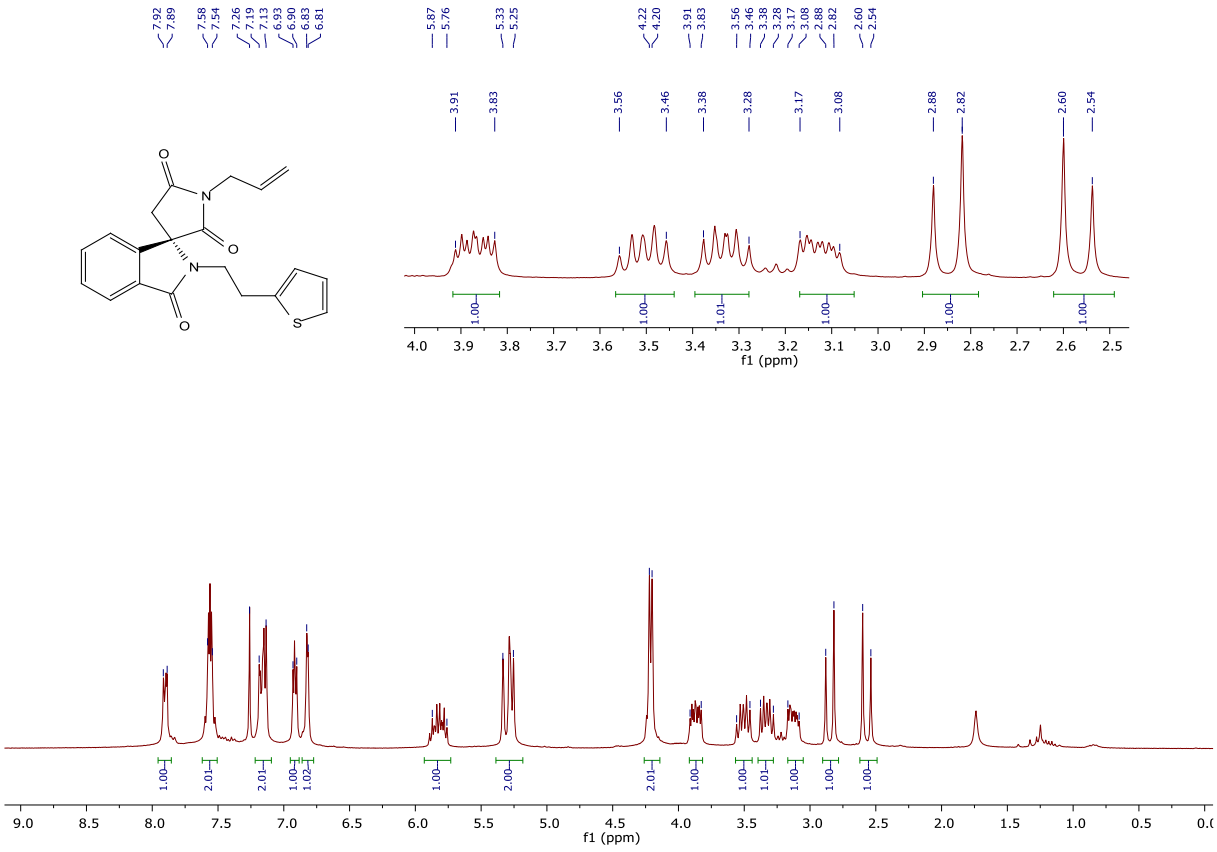
^{13}C NMR spectrum of compound **(±)-5iB**



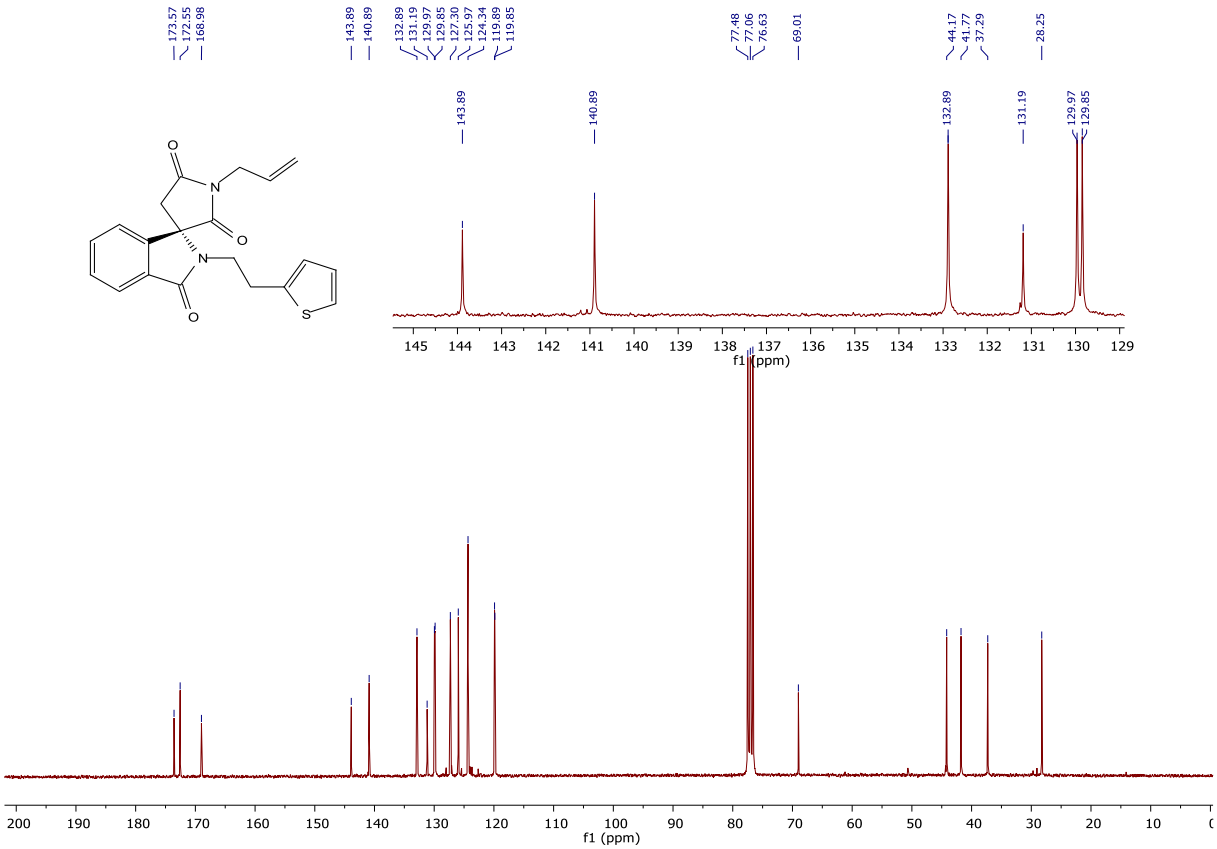
^{1}H NMR spectrum of compound **(±)-7j**



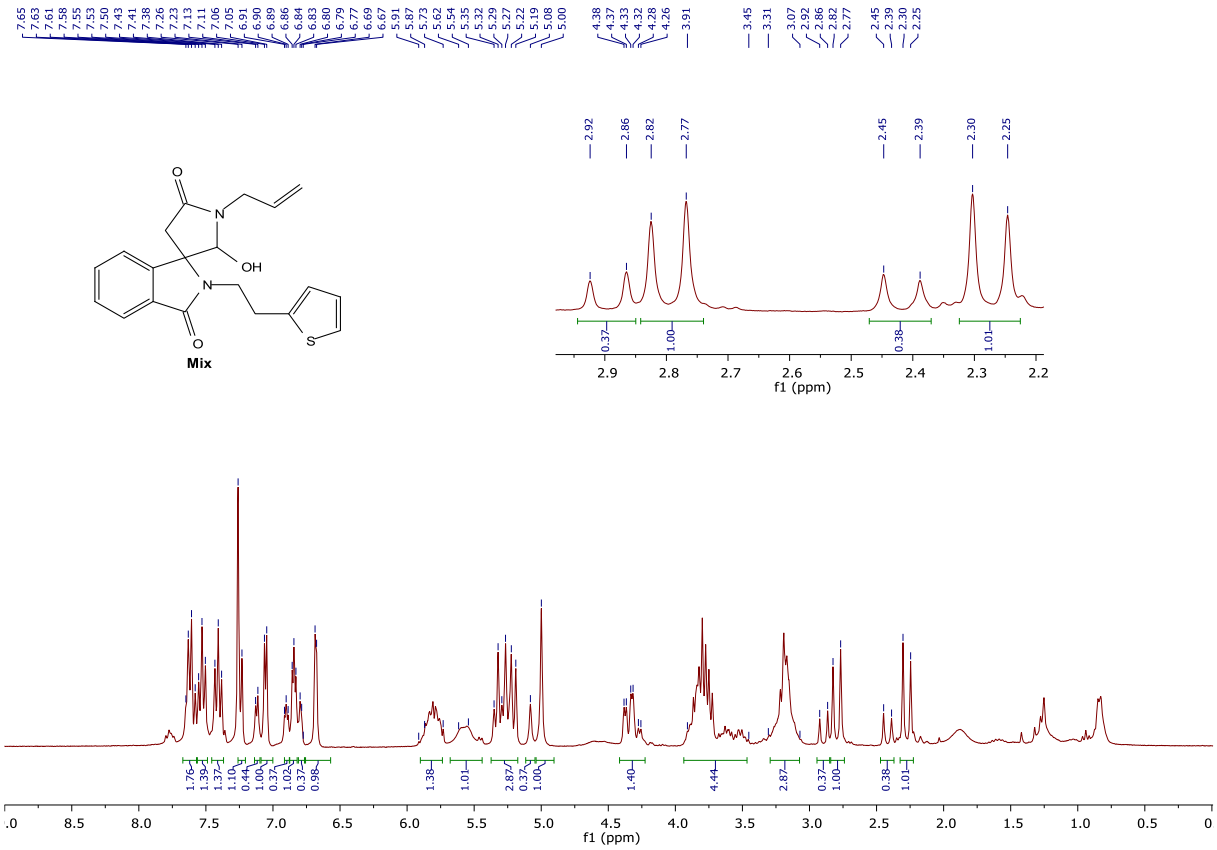
^{13}C NMR spectrum of compound **(±)-7k**



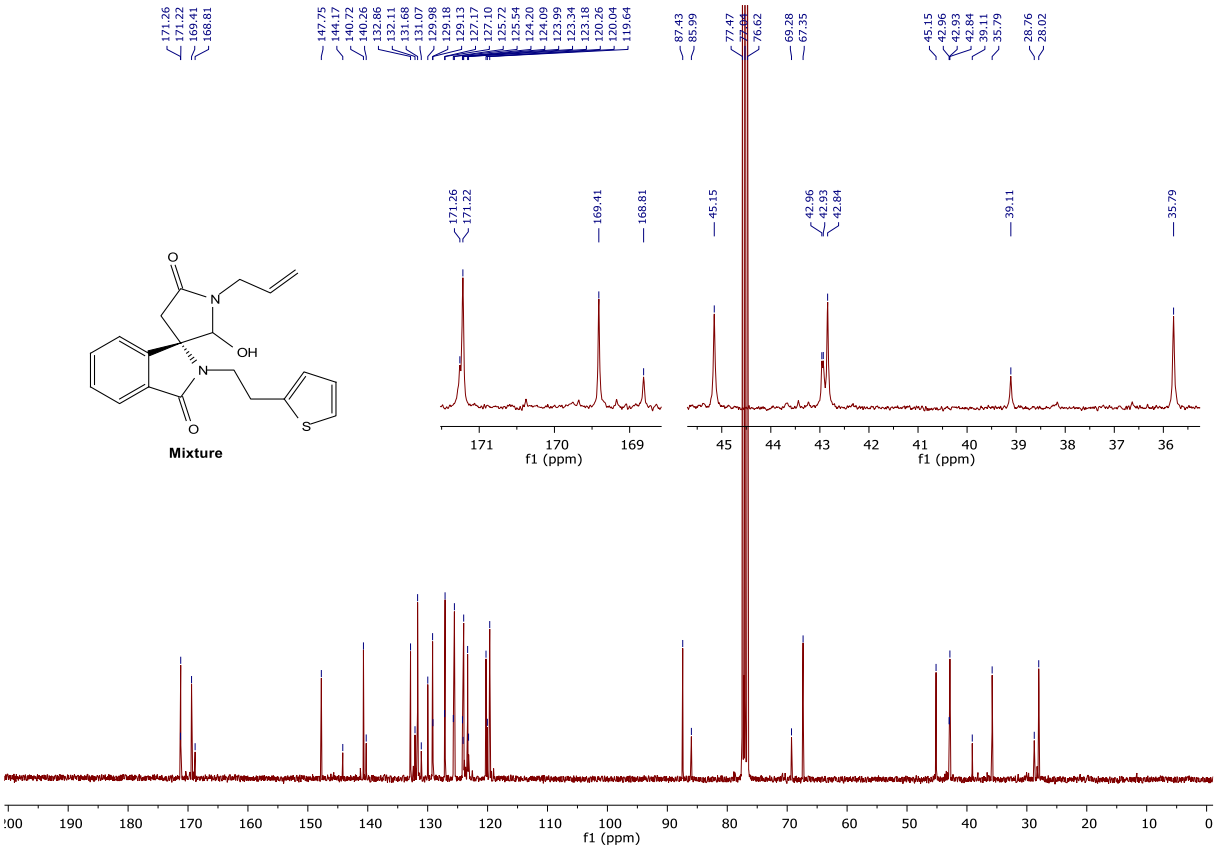
^{1}H NMR spectrum of compound **(±)-1j**



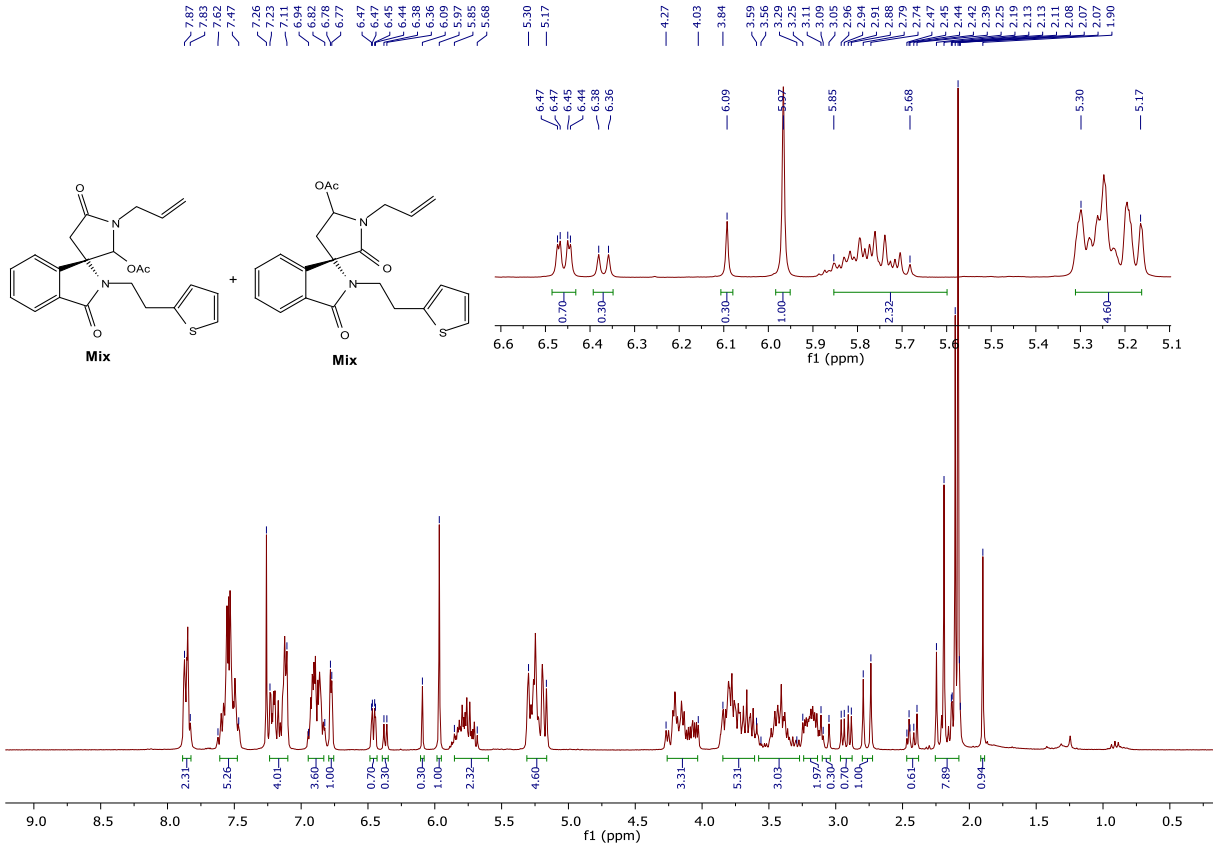
^{13}C NMR spectrum of compound **(±)-1j**



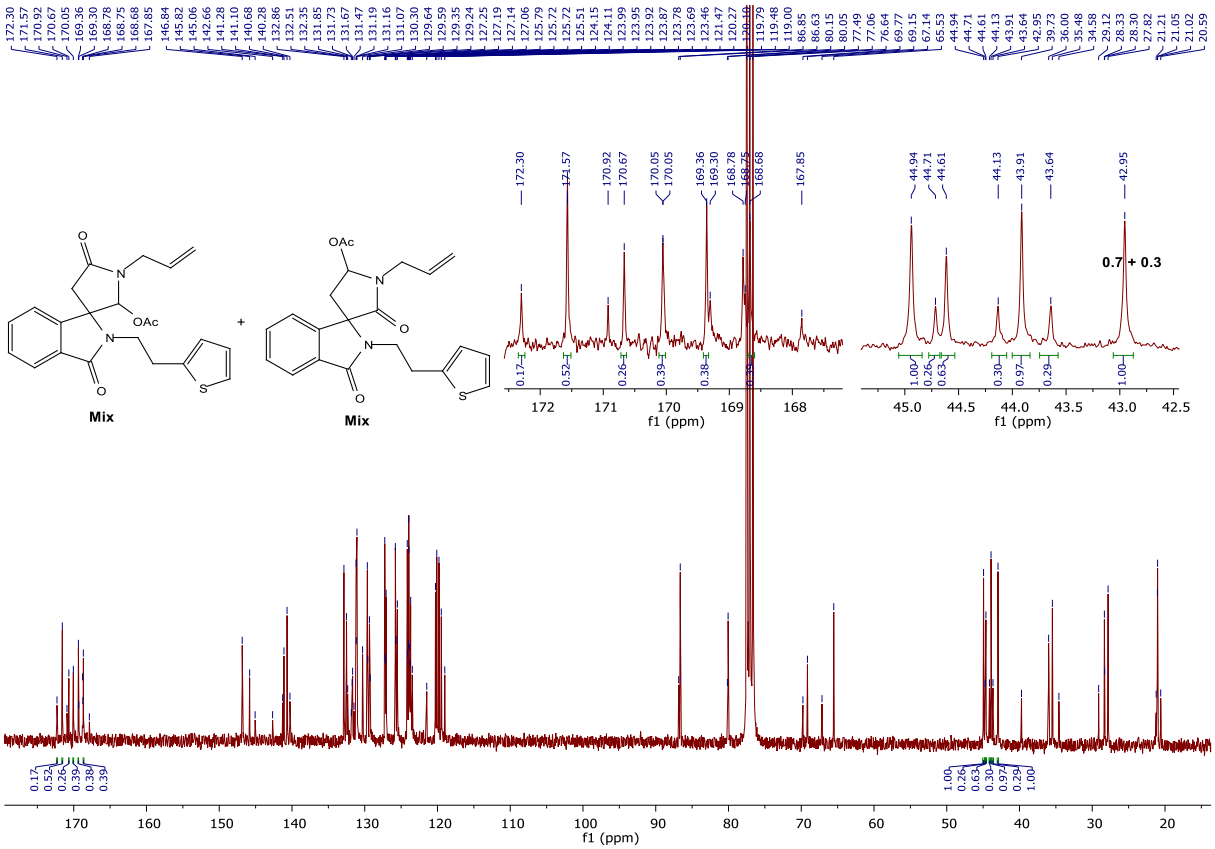
^{1}H NMR spectrum of compound **(±)-11j(A,B)**



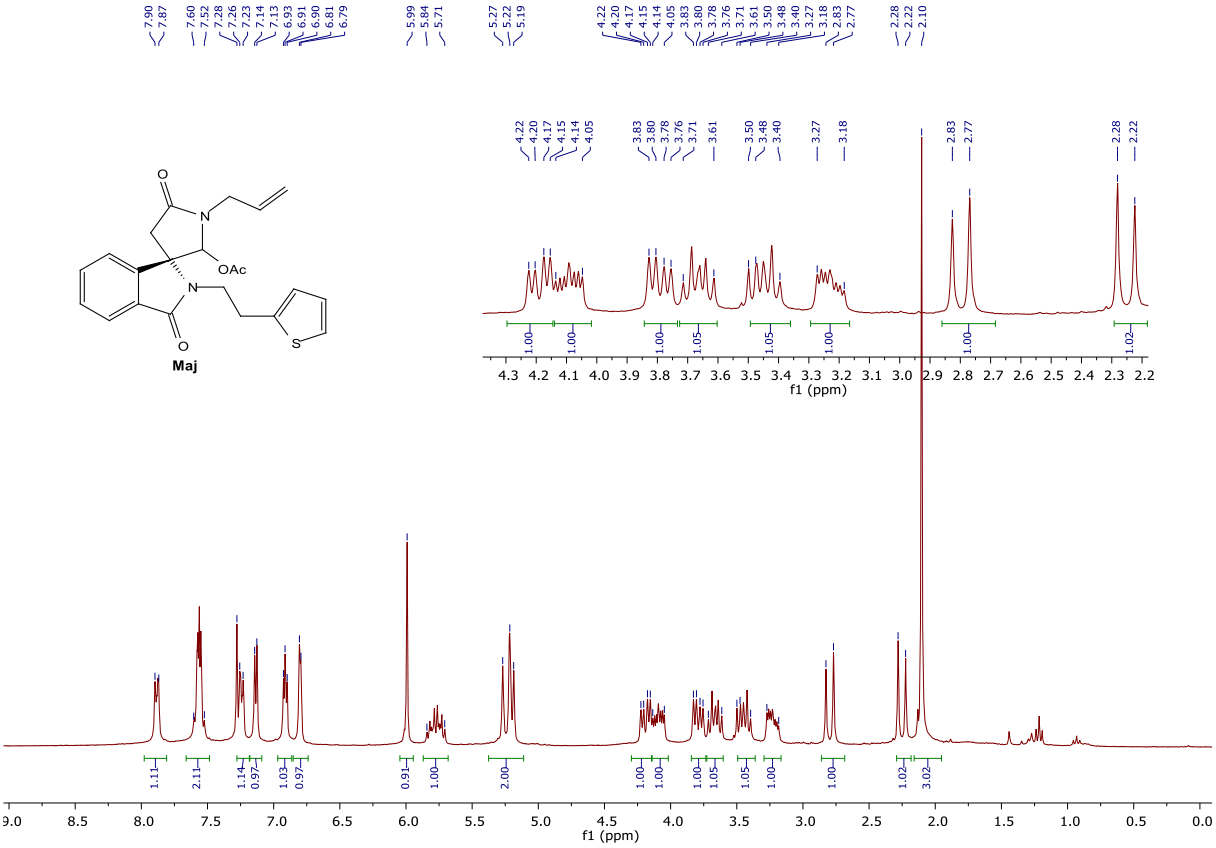
^{13}C NMR spectrum of compound **(±)-11j(A,B)**



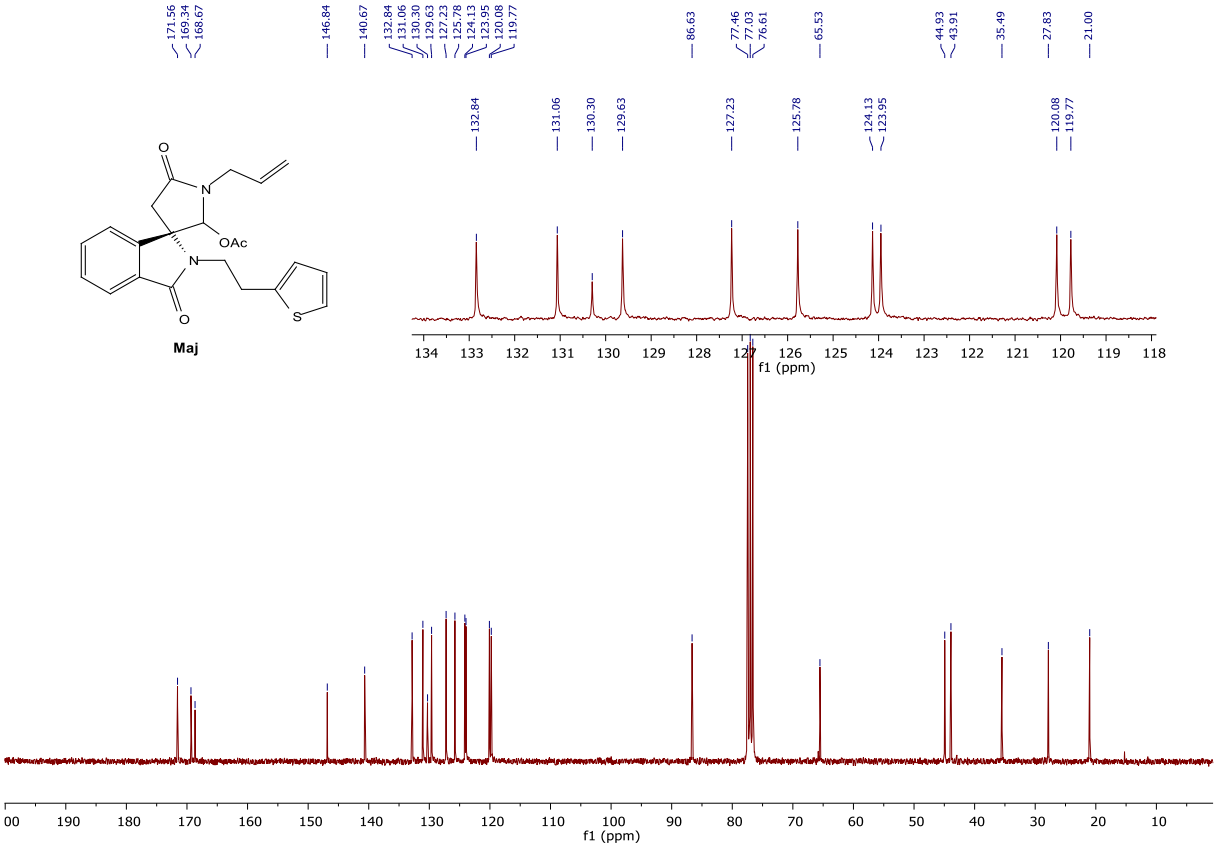
^{1}H NMR spectrum of compounds **(±)-8j(A,B)** and **(±)-9j(A,B)**



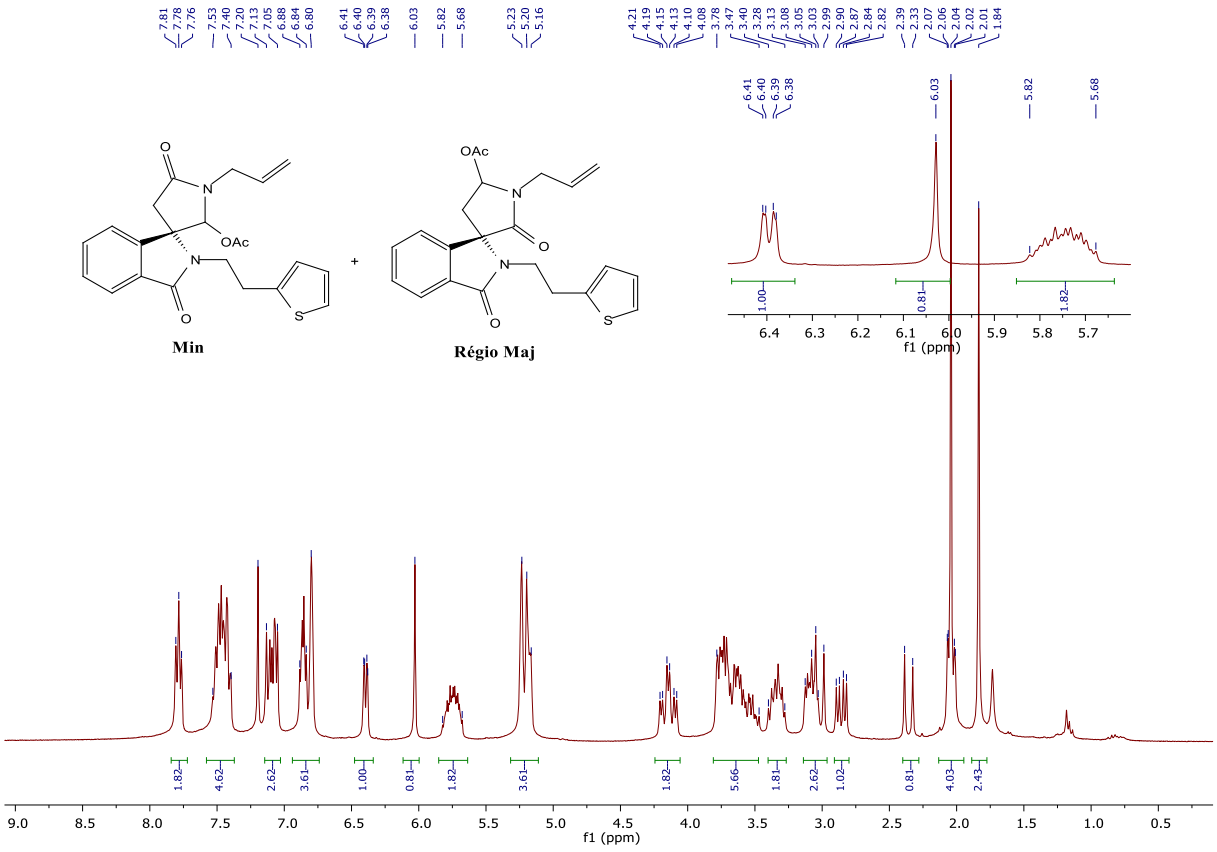
^{13}C NMR spectrum of compounds compounds **(±)-8j(A,B)** and **(±)-9j(A,B)**and **(±)-9j(A,B)**



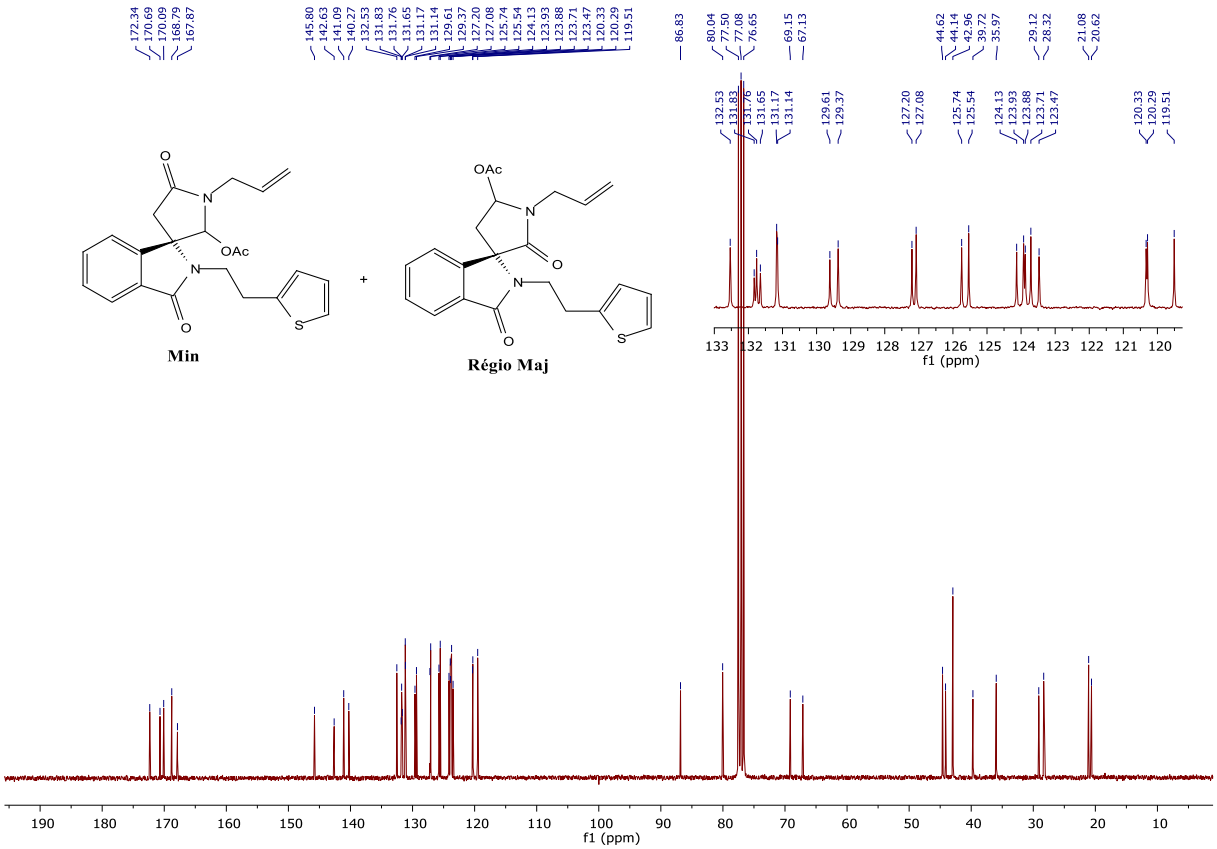
^{1}H NMR spectrum of compound compounds **(±)-8jA**



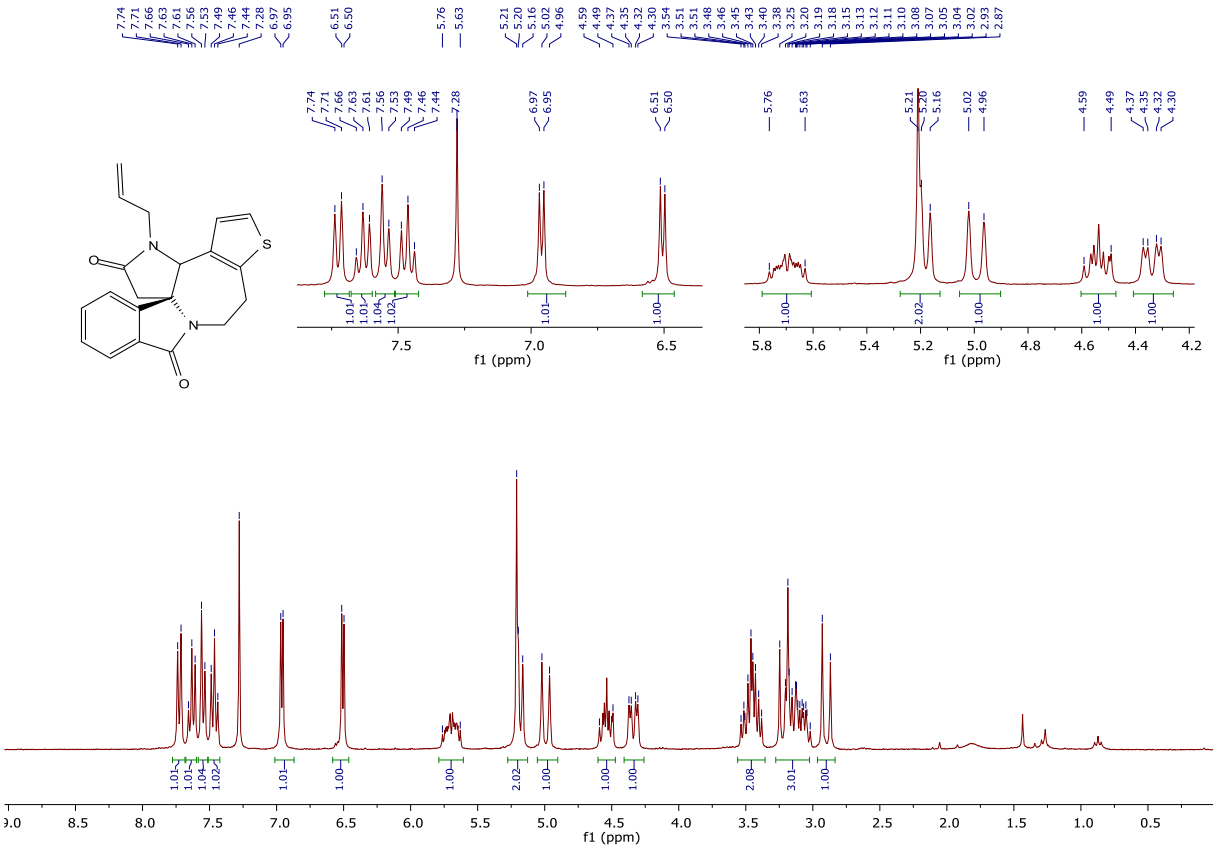
^{13}C NMR spectrum of compound **(±)-8jA**



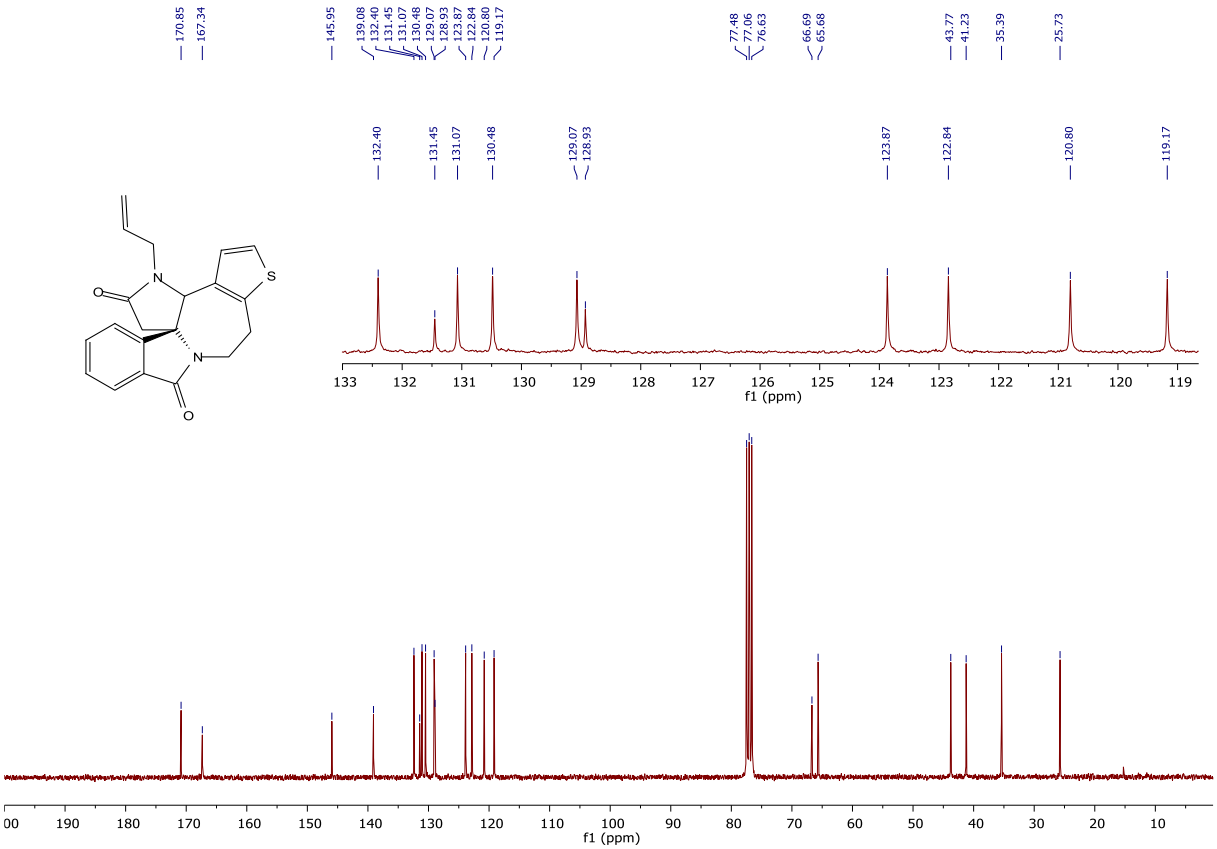
^{1}H NMR spectrum of compounds **(±)-8jB and (±)-9j(A,B)**



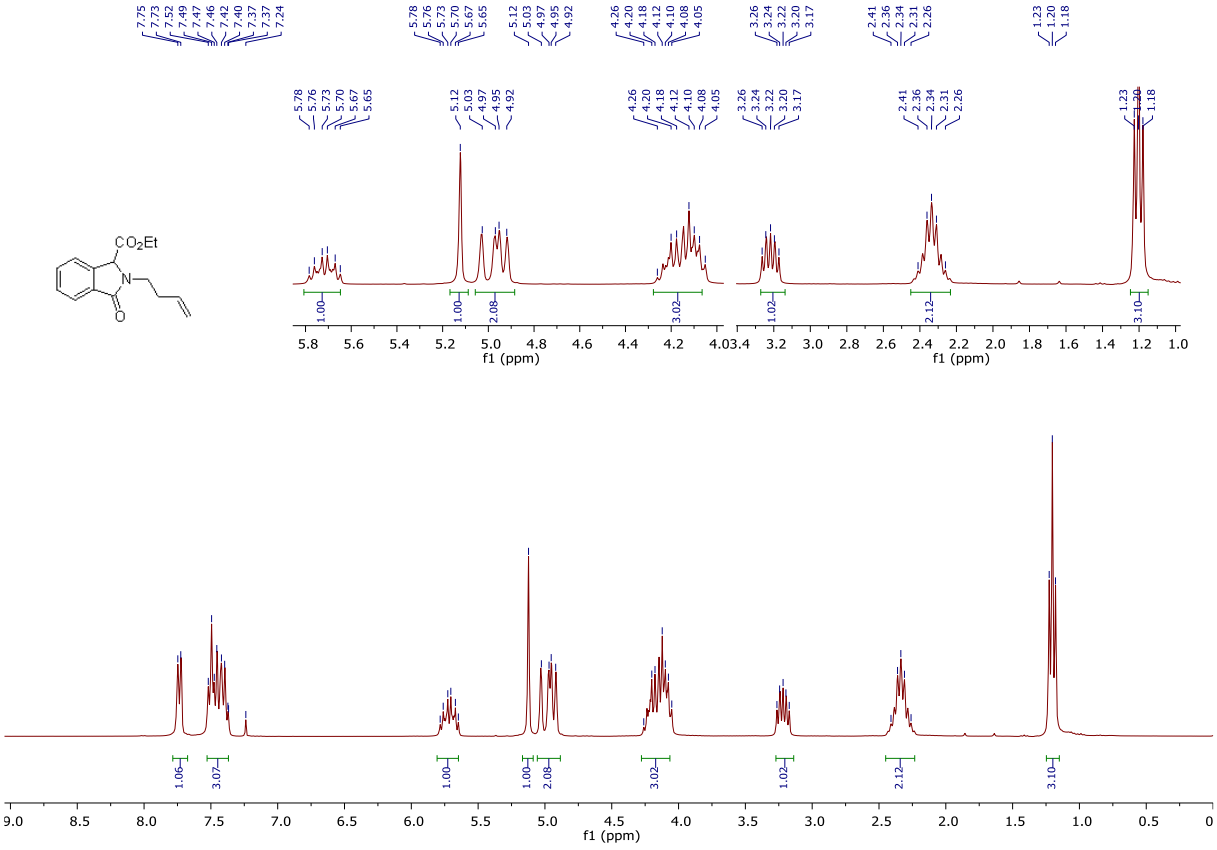
^{13}C NMR spectrum of compounds **(±)-8jB and (±)-9j(A,B)**



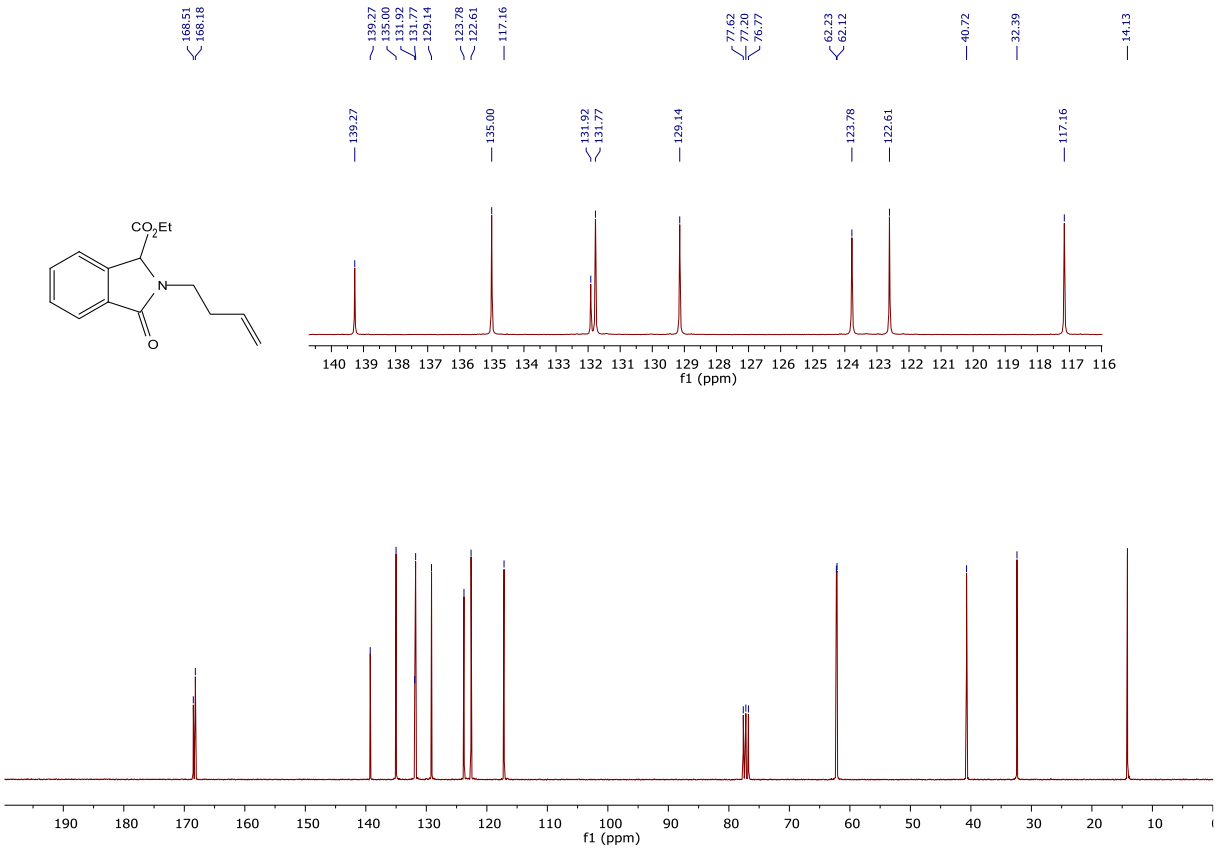
^{1}H NMR spectrum of compound **(±)-5jA**



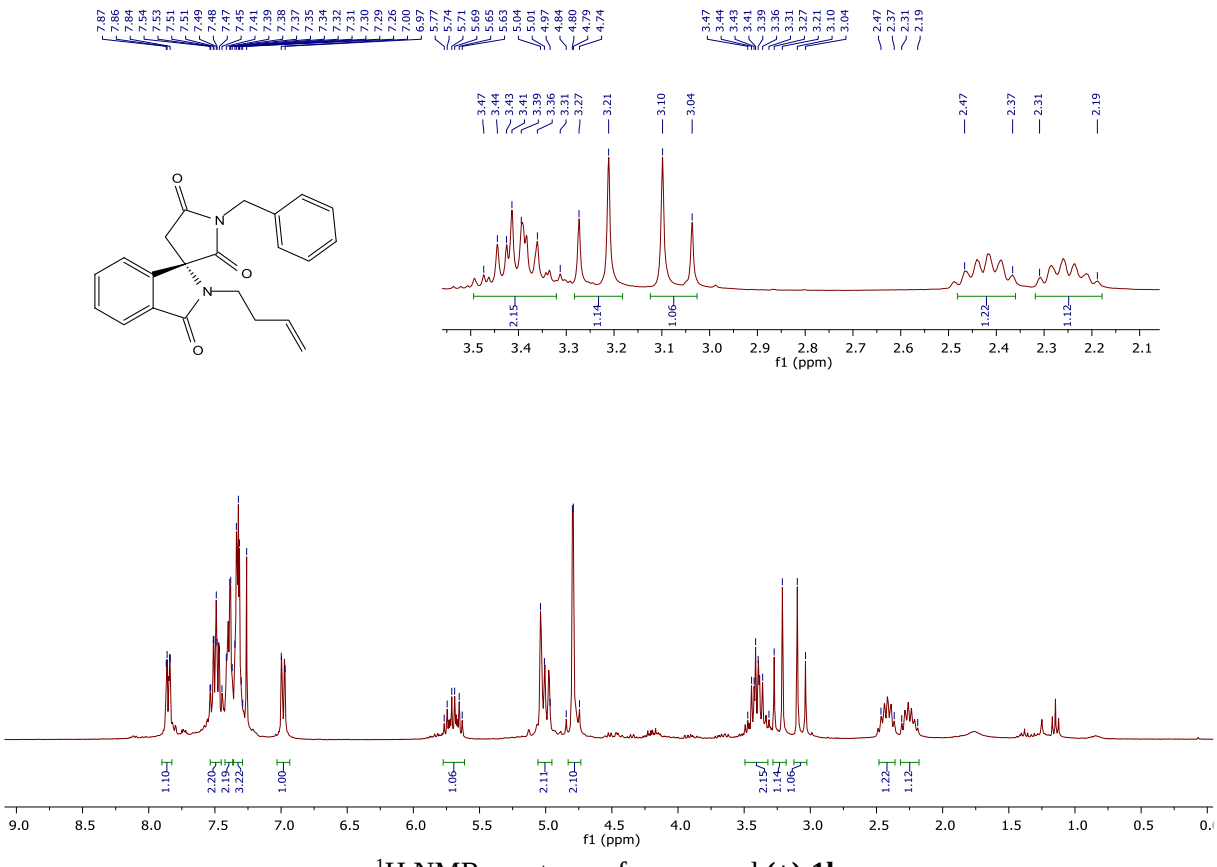
^{13}C NMR spectrum of compound **(±)-5jA**



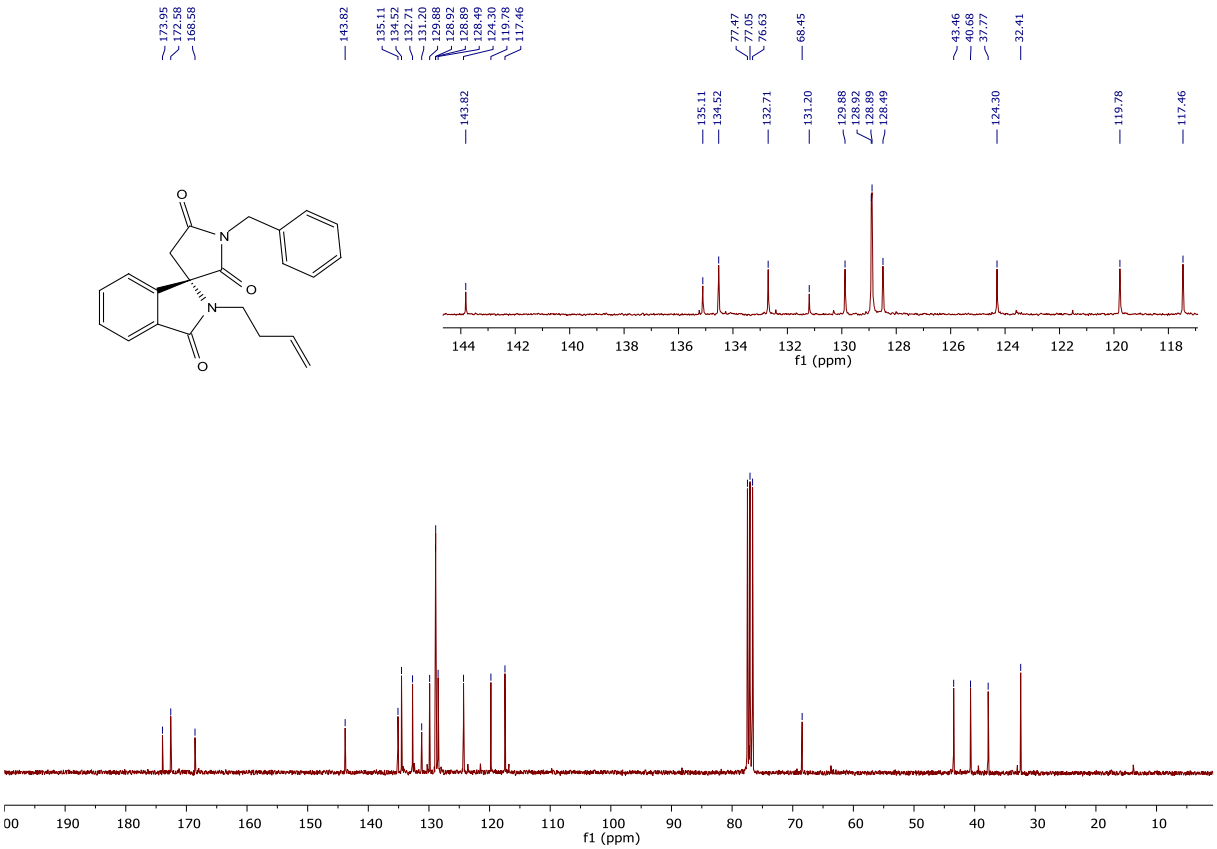
^{1}H NMR spectrum of compound **(±)-7k**



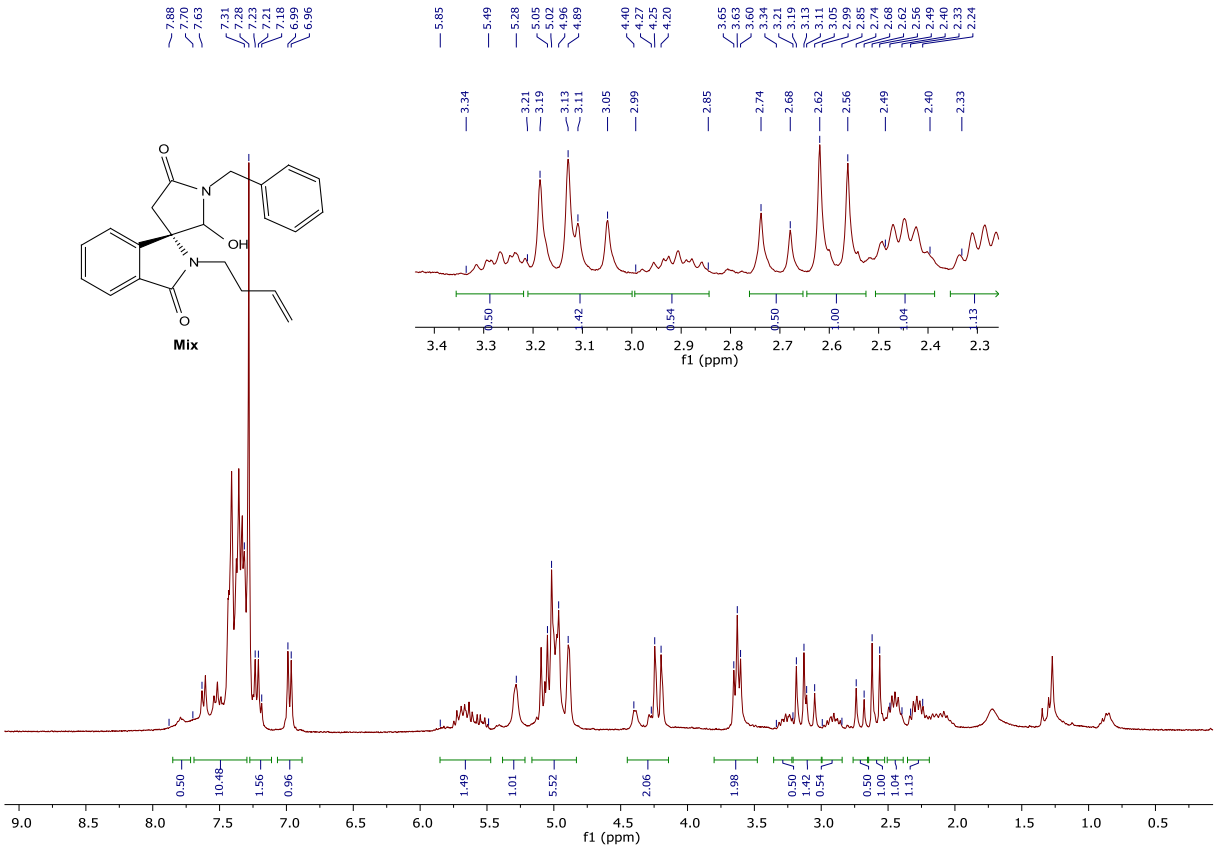
^{13}C NMR spectrum of compound **(±)-7k**



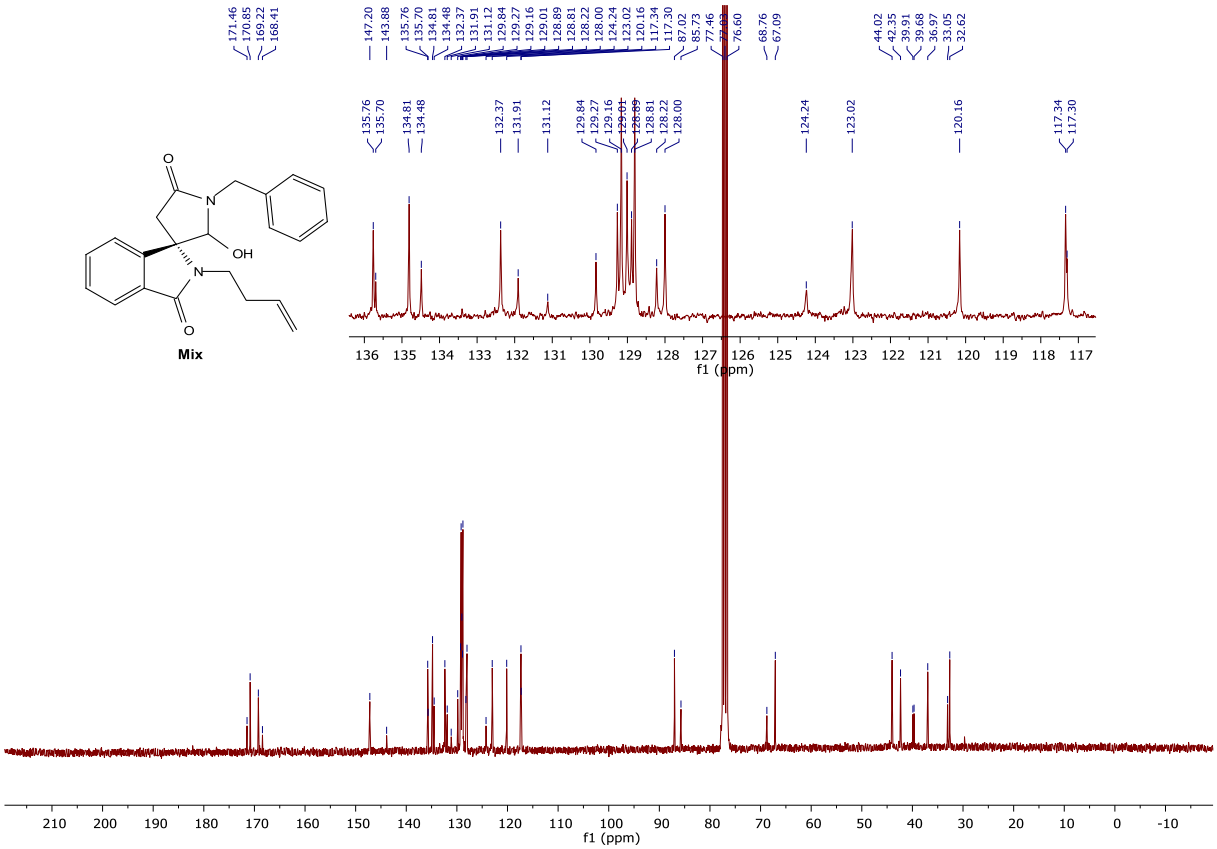
^{1}H NMR spectrum of compound **(±)-1k**



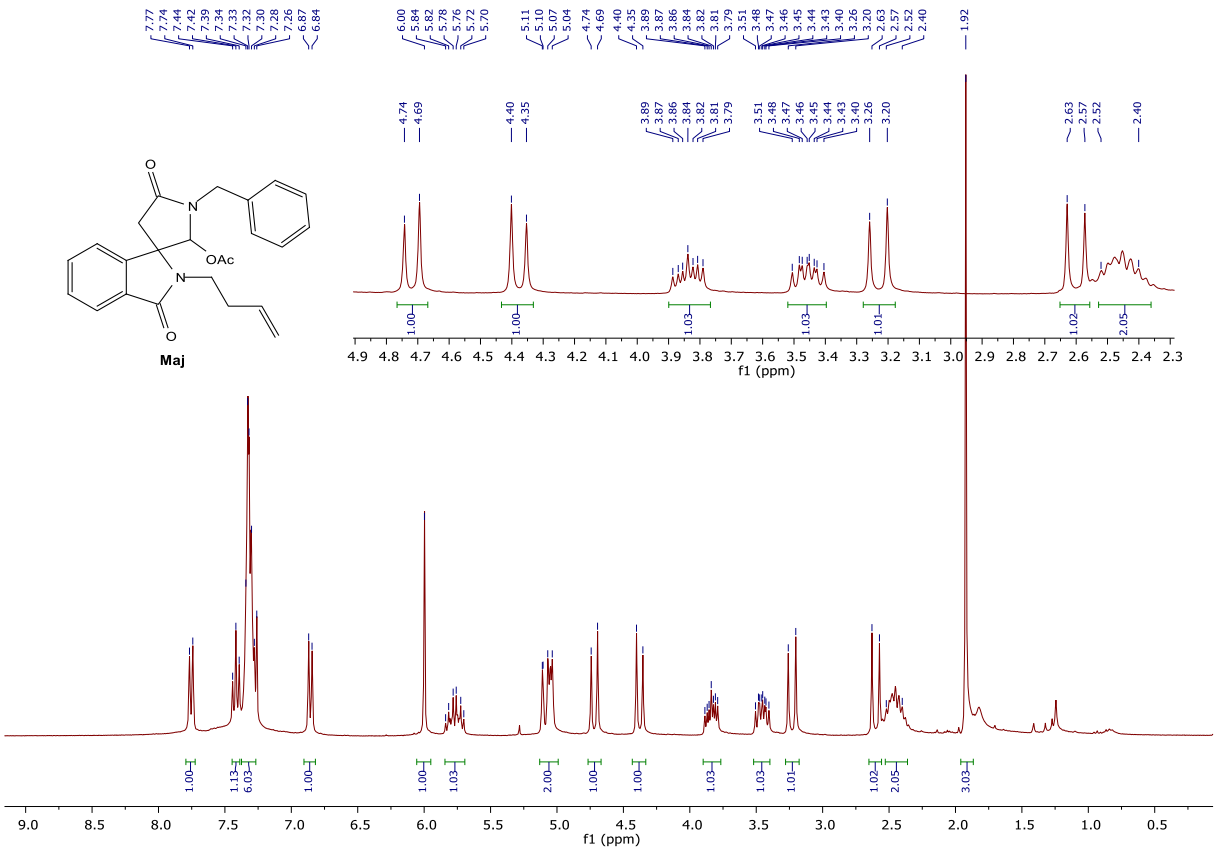
^{13}C NMR spectrum of compound **(±)-1k**



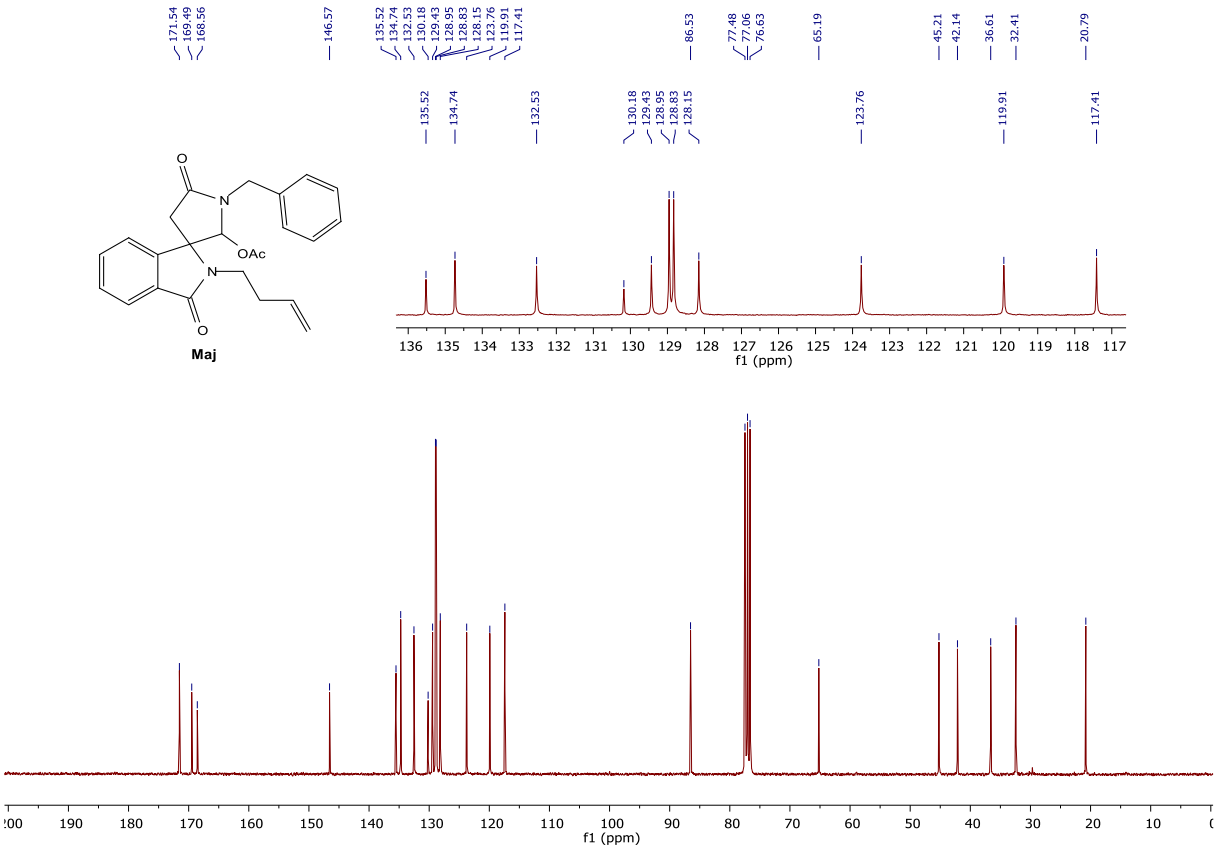
^{1}H NMR spectrum of compound **(±)-11k(A,B)**



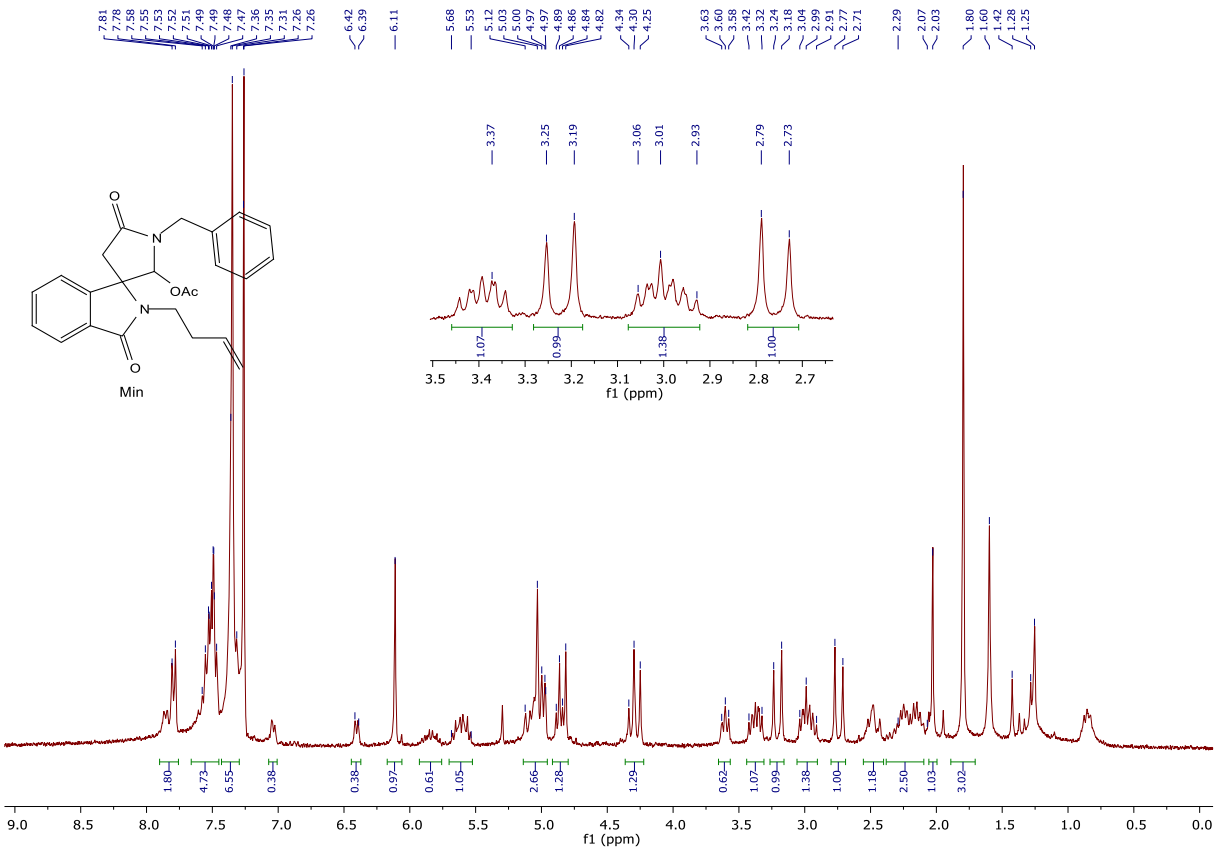
^{13}C NMR spectrum of compound **(±)-11k(A,B)**



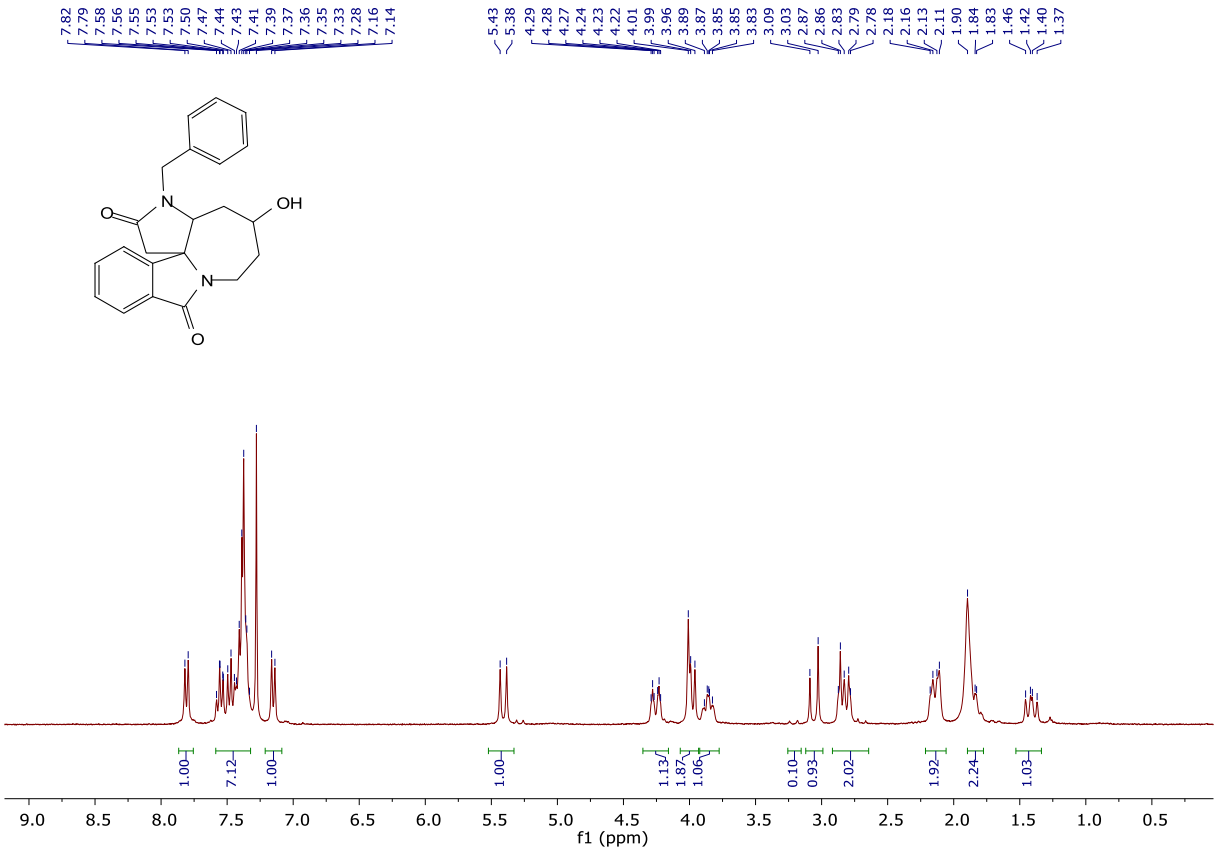
^{1}H NMR spectrum of compound **(±)-8kA**



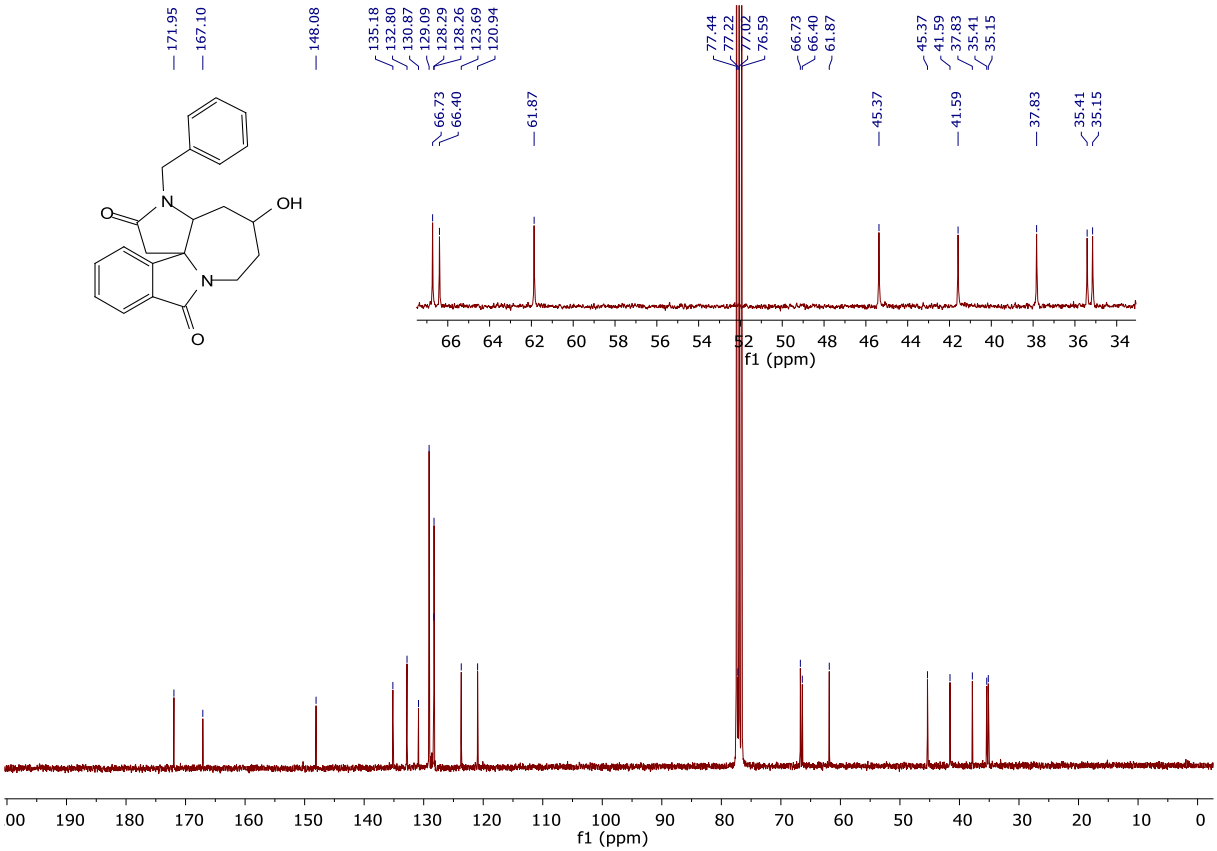
^{13}C NMR spectrum of compound **(±)-8kA**



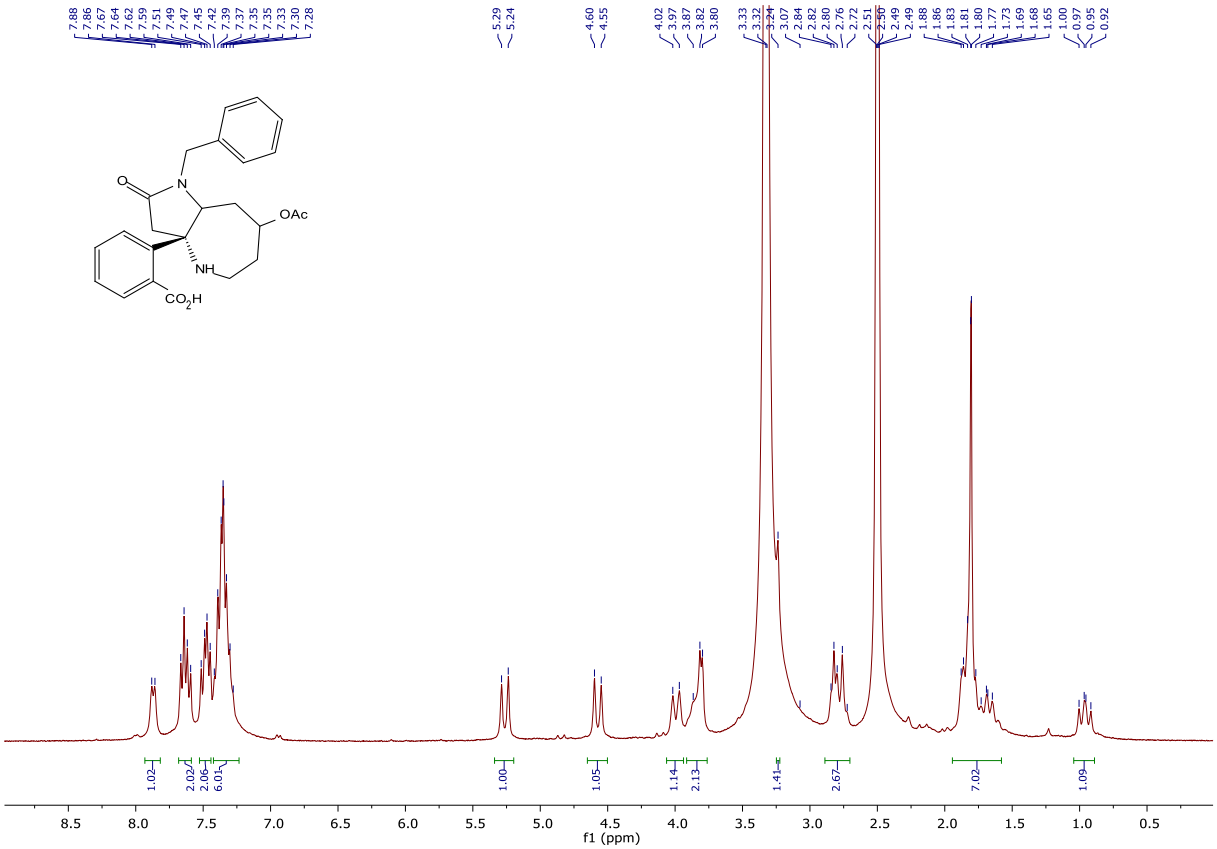
^{1}H NMR spectrum of compound **(±)-8kB**



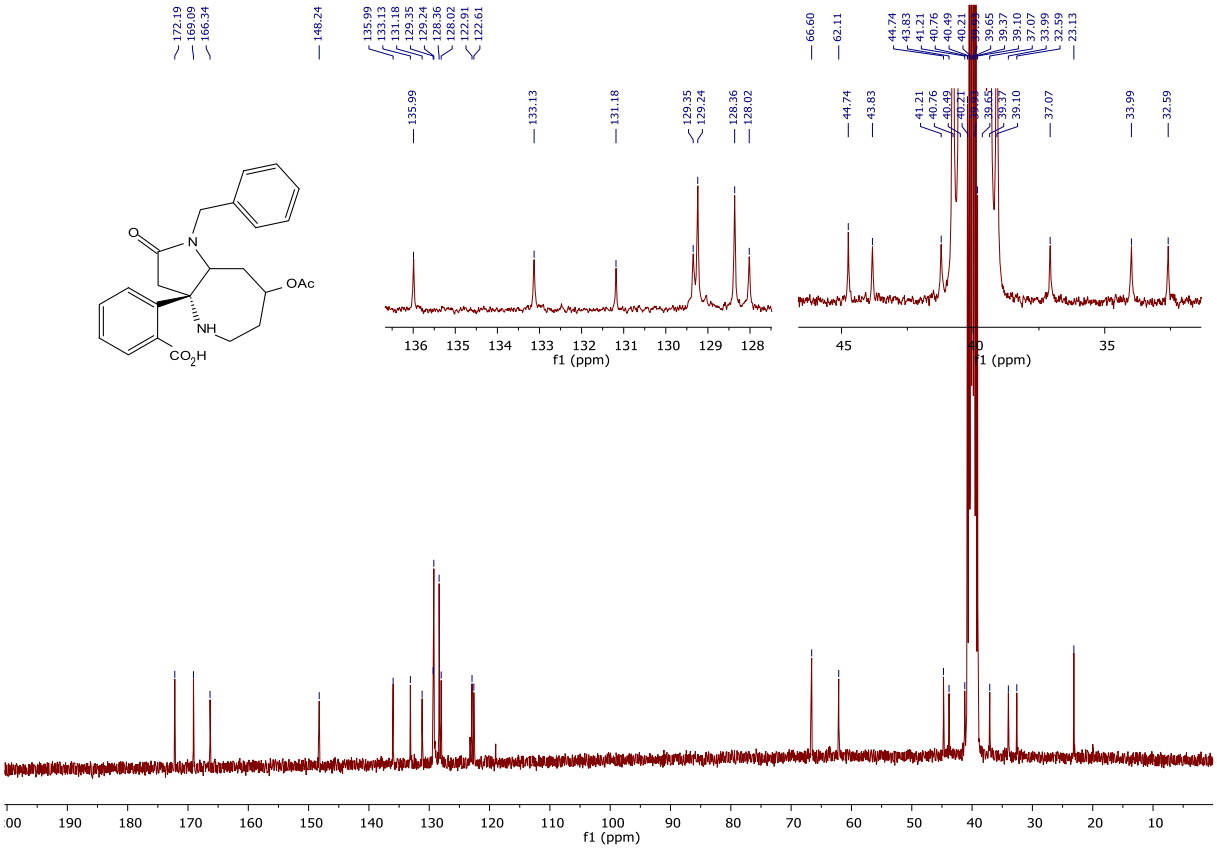
^{1}H NMR spectrum of compound **(±)-4k**



^{13}C NMR spectrum of compound **(±)-4k**



^{1}H NMR spectrum of compound **(±)-12k**



^{13}C NMR spectrum of compound **(±)-12k**

IX. Crystallographic data of compounds **(±)-5aA** and **(±)-5iA**

Table 1. Crystal data collection and structure refinement of products ayoub1 (**5aA**) and mk010707 (**5iA**).

Compound	**(+/-)-5aA**	**(+/-)-5iA**
Deposition Number	1848784	2022086
Formula	$C_{25}H_{20}N_2O_2$	$C_{28}H_{26}N_2O_4$
Formula weight	380.43	454.11
Temperature/K	293	193(2)
Wavelength/Å	0.71073	0.71073
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$
a/Å	10.115(6)	8.4505(17)
b/Å	22.716(8)	19.920(4)
c/Å	8.414(6)	13.650(3)
β	102.49(5)	94.14(3)°
Volume/ Å^3	1887.6(19)	2291.8(8)
Z	4	4
Density (calculated) g/cm^3	1.339	1.317
Absorp. coefficient/mm^{-1}	0.086	0.089
F(000)	800.0	960.0
F(000)’	800.33	960.44
Theta(max)/°	24.990	27.000
H k l_{max}	12 27 9	10 25 17
N_{ref}	3296	5004
R (reflections)	0.0506(2232)	0.0466(3827)
wR_2 (reflections)	0.1352(3292)	0.1308(4679)
S: Goodness-of-fit on F^2	1.031	1.067
Reported T Limits	Tmin=0.930 Tmax=0.960	Tmin=0.962 Tmax=0.965
N_{par}	343	309
Data completeness	0.999	0.935

Figure 1. Molecular structure of the isomer (+/-)-**5aA**.

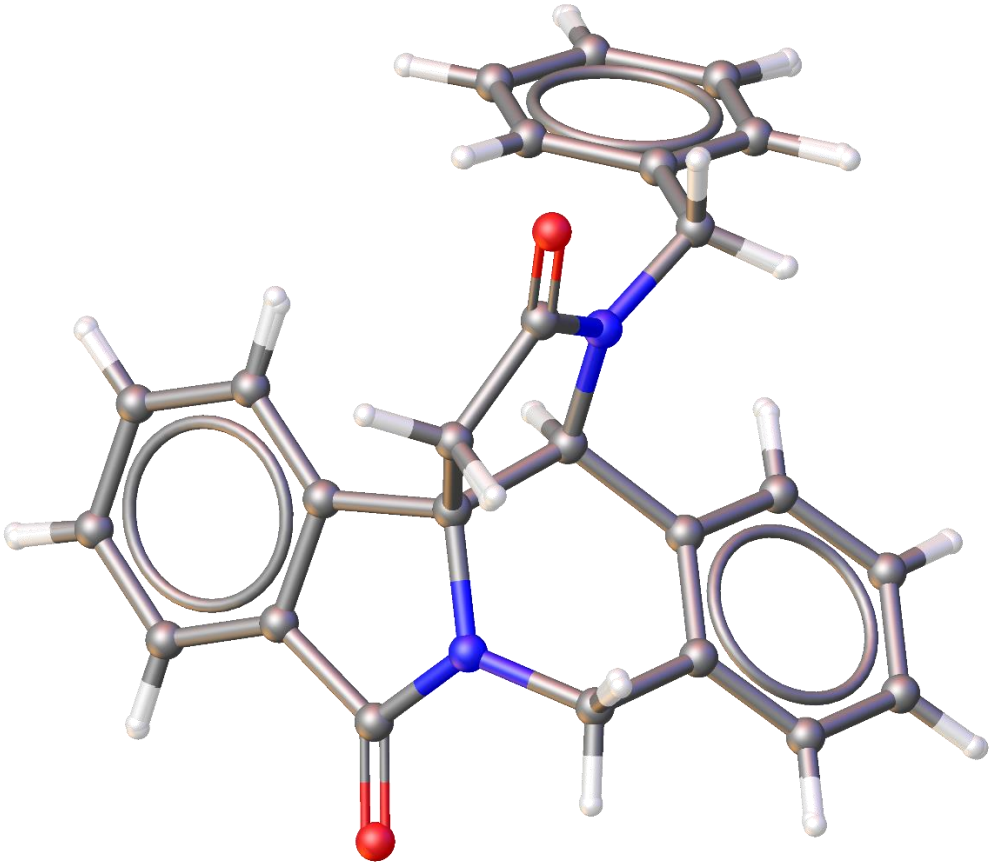
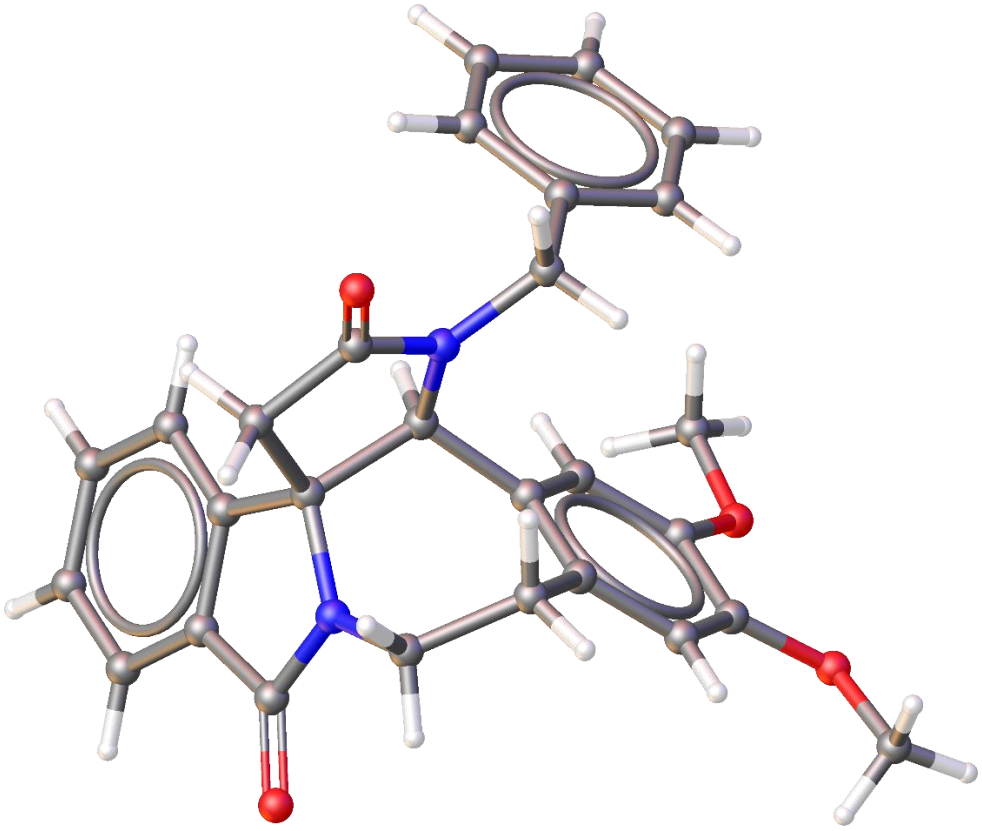


Figure 2. Molecular structure of (+/-)-**5iA** as major isomer.



checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: ayoub1 (Product 5aA)

```
Bond precision:  C-C = 0.0041 A          Wavelength=0.71073

Cell:            a=10.115(6)     b=22.716(8)         c=8.414(6)
                 alpha=90        beta=102.49(5)      gamma=90
Temperature:     293 K

                Calculated                Reported
Volume          1887.6(19)                1887.6(19)
Space group     P 21/c                    P21/c
Hall group      -P 2ybc                   ?
Moiety formula  C25 H20 N2 O2             ?
Sum formula     C25 H20 N2 O2             C25 H20 N2 O2
Mr              380.43                    380.43
Dx,g cm-3       1.339                     1.339
Z               4                         4
Mu (mm-1)       0.086                     0.086
F000            800.0                     660.0
F000'           800.33
h,k,lmax        12,27,9                   12,26,9
Nref            3296                      3292
Tmin,Tmax       0.965,0.974               0.930,0.960
Tmin'           0.960

Correction method= # Reported T Limits: Tmin=0.930 Tmax=0.960
AbsCorr = MULTI-SCAN

Data completeness= 0.999          Theta(max)= 24.990

R(reflections)= 0.0506( 2232)     wR2(reflections)= 0.1352( 3292)

S = 1.031                 Npar= 343
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)...     Please Check
PLAT088_ALERT_3_C Poor Data / Parameter Ratio .....................      9.61 Note
PLAT199_ALERT_1_C Reported _cell_measurement_temperature ..... (K)       293 Check
PLAT200_ALERT_1_C Reported   _diffrn_ambient_temperature ..... (K)       293 Check
PLAT222_ALERT_3_C NonSolvent Resd 1  H   Uiso(max)/Uiso(min) Range       5.2 Ratio
PLAT245_ALERT_2_C U(iso) H18       Smaller than U(eq) C18       by     0.017 Ang**2
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ...............   0.00412 Ang.
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT793_ALERT_4_G Model has Chirality at C3          (Centro SPGR)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C18         (Centro SPGR)          S Verify
PLAT899_ALERT_4_G SHELXL97  is Deprecated and Succeeded by SHELXL/       2018 Note
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   7 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   4 ALERT level G = General information/check it is not something unexpected

   3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

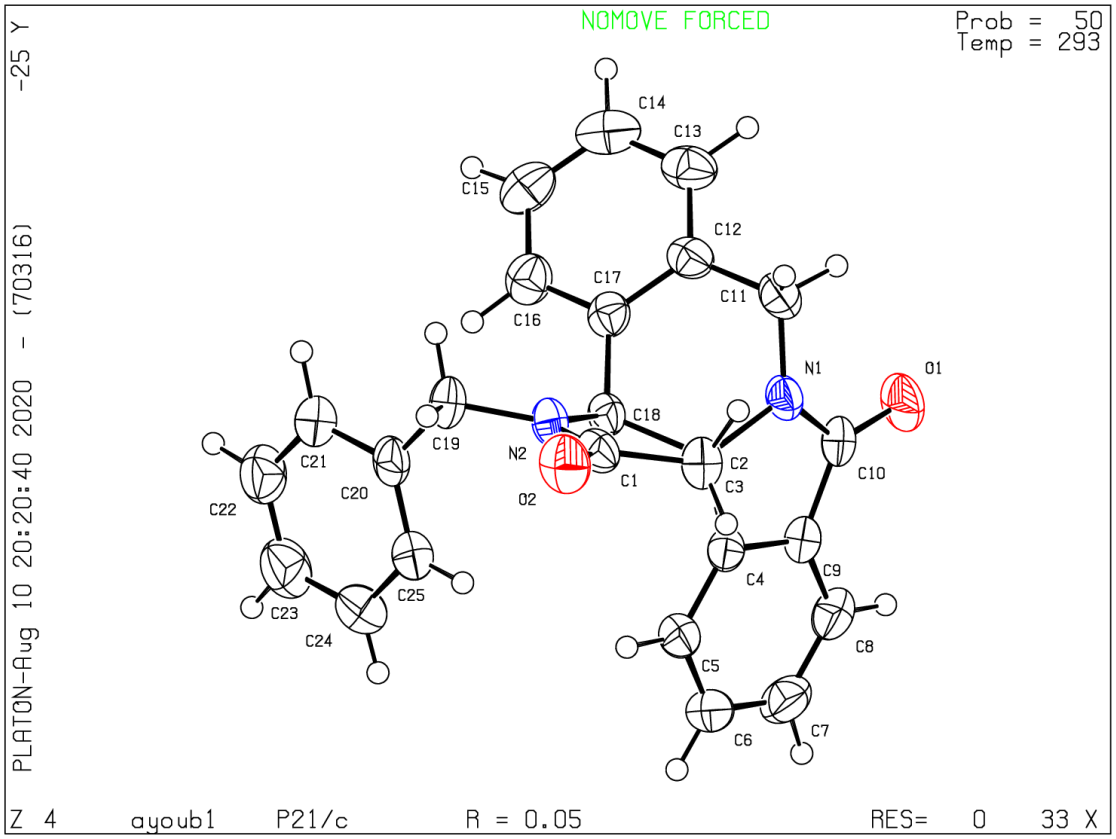
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 16/07/2020; check.def file version of 12/07/2020

Datablock ayoub1 - ellipsoid plot



checkCIF/PLATON report

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: mk010707 (Product 5iA)

```
Bond precision:  C-C = 0.0020 A          Wavelength=0.71073

Cell:            a=8.4505(17)     b=19.920(4)       c=13.650(3)
                 alpha=90         beta=94.14(3)     gamma=90
Temperature:     193 K
```

	Calculated	Reported
Volume	2291.8(8)	2291.7(8)
Space group	P 21/c	P21/c
Hall group	-P 2ybc	?
Moiety formula	C28 H26 N2 O4	?
Sum formula	C28 H26 N2 O4	C28 H26 N2 O4
Mr	454.51	454.51
Dx,g cm-3	1.317	1.317
Z	4	4
Mu (mm-1)	0.089	0.089
F000	960.0	960.0
F000′	960.44	
h,k,lmax	10,25,17	10,25,17
Nref	5004	4679
Tmin,Tmax	0.963,0.966	0.962,0.965
Tmin′	0.963	

```
Correction method= # Reported T Limits: Tmin=0.962 Tmax=0.965
AbsCorr = MULTI-SCAN

Data completeness= 0.935          Theta(max)= 27.000

R(reflections)= 0.0466( 3827)     wR2(reflections)= 0.1308( 4679)

S = 1.067                 Npar= 309
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT018_ALERT_1_C _diffrn_measured_fraction_theta_max .NE. *_full           ! Check
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical          ? Check
PLAT093_ALERT_1_G No s.u.'s on H-positions, Refinement Reported as       mixed Check
PLAT793_ALERT_4_G Model has Chirality at C18         (Centro SPGR)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C19         (Centro SPGR)          S Verify
PLAT899_ALERT_4_G SHELXL97  is Deprecated and Succeeded by SHELXL/       2018 Note
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PLATON version of 16/07/2020; check.def file version of 12/07/2020

Datablock mk010707 - ellipsoid plot