Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

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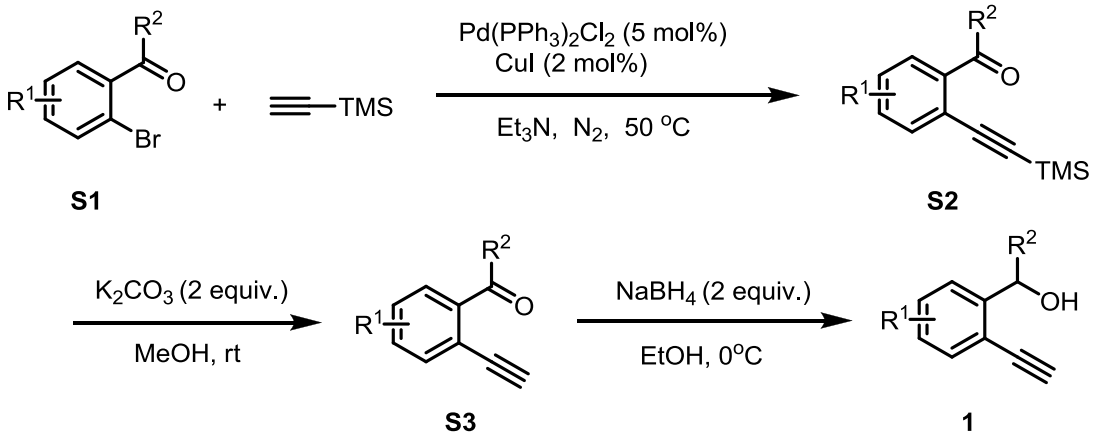
1. General Information

The ^{1}H NMR and ^{13}C NMR spectra were recorded at Bruker AV 400 MHz or 600 MHz. ^{1}H and ^{13}C NMR Chemical shifts were calibrated to tetramethylsilane as an internal reference. Chemical shifts are given in (ppm) and coupling constants (*J*) in Hz. The following abbreviations are used to indicate the multiplicity: s, singlet; d, doublet: t, triplet; q, quartet; m, multiplet; High resolution mass spectrometric (HRMS) analyses spectrum was determined on the Varian 7.0T FTMS instrument.

2. Synthesis of the arylalkynols 1.

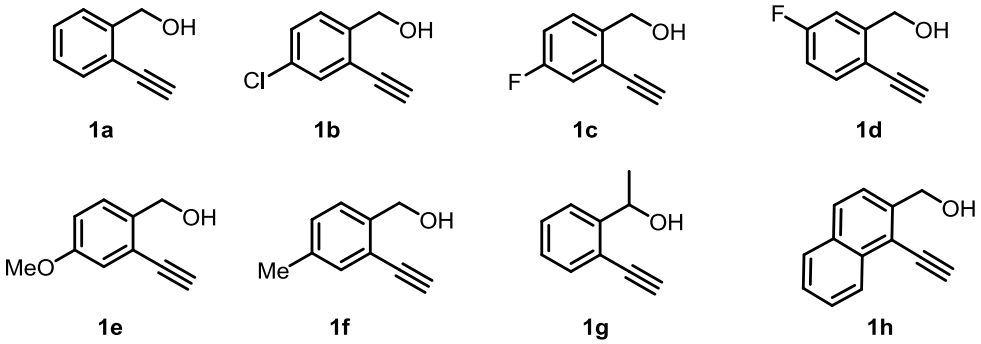


To a solution of 2-bromobenzaldehyde or its derivatives **S1** (20 mmol, 1 equiv.), $PdCl_2(PPh_3)_2$ (702.0 mg, 5 mol %), and CuI (76.0 mg, 2 mol %) in Et_3N (40.0 mL) was added under N_2 atmosphere. After being stirred for 10 mins at room temperature, terminal acetylene (1.5 equiv.) was added to the mixture. The resulting mixture was heated under N_2 atmosphere at 50 ℃ for 6-18 h. After the reaction was completed, the reaction mixture was quenched with distilled water and extracted with CH_2Cl_2 (100 mL × 3). The combined organic layer was washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by column chromatography on silica gel to afford the desired product 2-((trimethylsilyl)ethynyl)benzaldehyde derivatives **S2**.

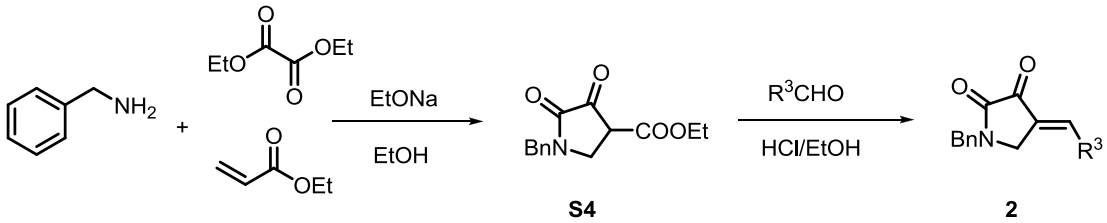
S2 (10 mmol, 1.0 equiv.) was dissolved in the mixture solution of MeOH, and treated with K_2CO_3 (2.764 g, 20 mmol, 2.0 equiv.). After stirring at ambient temperature for 2 h, the mixture was diluted with Et_2O, partitioned with H_2O, dried over Na_2SO_4 and concentrated in vacuo, purified by flash column chromatography to

give the arylalkynyl benzaldehyde derivatives product **S3**.

S3 (8 mmol, 1.0 equiv.) was added to round-bottomed flask and dissolved by MeOH. At 0 ℃, $NaBH_4$ (605.3 mg, 16 mmol, 2.0 equiv.) was added to the flask in batches. After 30 min, the mixture was quenched with saturated NH_4Cl aqueous solution, and then was washed three times with brine, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by column chromatography on silica gel to afford the desired product **1**.



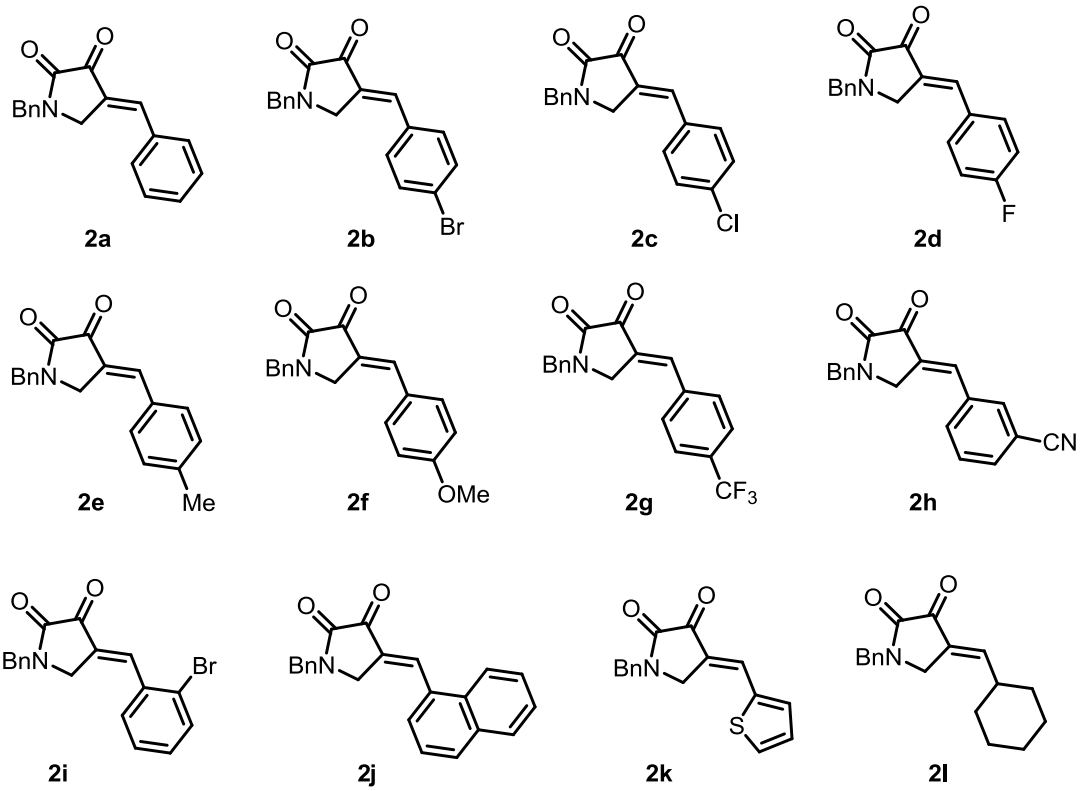
3. Synthesis of the dioxopyrrolidines 2.



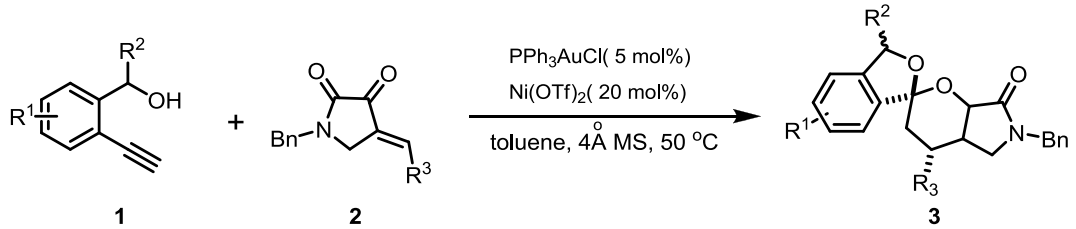
A mixture of benzylamine (7.26 g, 66.0 mmol), ethyl acrylate (7.2 mL, 66.0 mmol) in EtOH (15 mL) was stirred at room temperature for 16 h. Diethyl oxalate (9.0 mL, 66 mmol) and freshly-made sodium ethoxide solution in EtOH (generated from 2.0 g of sodium metal, 80.0 mmol, in 15 mL EtOH) was added. The mixture was heated at reflux for 1 h and solidified. The volatiles were removed in vacuo. The crude product was diluted with H_2O (80 mL) and the pH of the mixture was adjusted to 1 by adding conc.HCl. The mixture was subjected to filtration to afford **S4** as a white solid.

A mixture of **S4** (2.6 g, 9.8 mmol), aromatic aldehyde (9.8 mmol) in EtOH (20 mL)/ 20% aq. HCl (50 mL) was heated at reflux for 4 h. After cooling down to

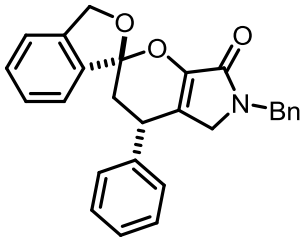
ambient temperature, the aqueous layer was decanted. The obtained chunky solid was collected and further recrystallized fromEtOAc to afford **2** as a bright yellow solid.



4. Synthesis and Characterization of [2+4] annulation products.



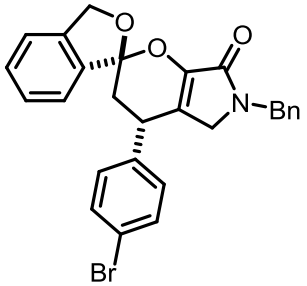
To a shlenck tube, dioxopyrrolidienes **2** (0.2 mmol), $Ni(OTf)_2$ (13 mg, 0.04 mmol, 20% mol), 4Å MS (40 mg) and a magnetic stir was sequentially added under nitrogen atmosphere. The mixture was stirred at room temperature for 2 h, then Ph_3PAuCl (5 mg, 5 mol%) and arylalkynols **1** (0.3 mmol, 1.5 equiv.) was added. The reaction mixture was stirred at 50 ℃ until complete disappearance of **2** observed by TLC. After completion, the mixture was cooled to room temperature, filtered through silica soil and the solvent was removed in vacuo. The product was purified by silica gel column chromatography (petroleum ether/EtOAc = 2 :1) to afford the compound **3**.



3aa

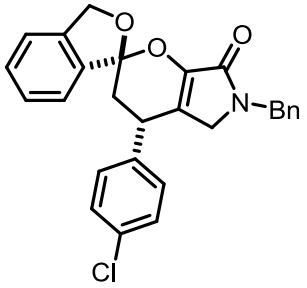
6'-benzyl-4'-phenyl-4',4a',5',6'-tetrahydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(7a'H)-one

White solid, 72 mg, yield 88%, m.p. 164-165 °C, *dr* = 14:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.17 (m, 16H), 5.37 (d, *J* = 12.5 Hz, 1H), 5.11 (d, *J* = 12.6 Hz, 1H), 4.76 (d, *J* = 14.9 Hz, 1H), 4.44 (d, *J* = 14.9 Hz, 1H), 4.10 (dd, *J* = 12.0, 5.8 Hz, 1H), 3.57 (d, *J* = 18.2 Hz, 1.26H), 3.48 (d, *J* = 18.3 Hz, 0.94H), 2.53 (t, *J* = 12.7 Hz, 1H), 2.37 (dd, *J* = 13.3, 5.8 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 144.7, 141.1, 140.2, 138.6, 137.2, 130.0, 129.2, 128.9, 128.3, 128.0, 127.7, 127.6, 123.6, 122.6, 121.3, 110.6, 72.7, 47.7, 46.7, 39.9, 36.7. HRMS (ESI+) calculated for $C_{27}H_{24}NO_3$ ($M+H^+$) 410.1751; found 410.1754.



3ab l-4'-(4-bromophenyl)-4',4a',5',6'-tetrahydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(7a'H)-one

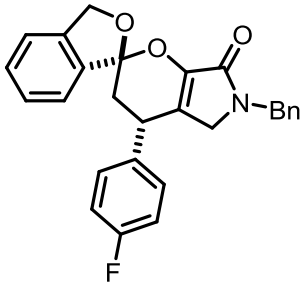
Yellow solid, 74 mg, yield 76%, m.p. 139-140 °C, *dr* = 12:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 – 6.97 (m, 15H), 5.36 (d, *J* = 12.6 Hz, 1H), 5.11 (d, *J* = 12.6 Hz, 1H), 4.74 (d, *J* = 14.9 Hz, 1H), 4.45 (d, *J* = 14.9 Hz, 1H), 4.08 (dd, *J* = 12.0, 5.7 Hz, 1H), 3.56 (dd, *J* = 18.2, 1.7 Hz, 1H), 3.45 (d, *J* = 18.2 Hz, 1H), 2.48 (t, *J* = 12.7 Hz, 1H), 2.35 (dd, *J* = 13.3, 5.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.5, 145.0, 140.14, 140.09, 138.4, 137.1, 132.4, 130.1, 129.7, 128.9, 128.3, 128.0, 127.8, 122.7, 122.5, 121.43, 121.35, 110.5, 72.8, 47.5, 46.7, 39.7, 36.3. HRMS (ESI+) calculated for $C_{27}H_{23}BrNO_3$ ($M+H^+$) 488.0856; found 488.0859.



3ac

6'-benzyl-4'-(4-chlorophenyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

White solid, 74 mg, yield 84%, m.p. 168-169 °C, *dr* = 11:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.28 (m, 3H), 7.25 (dt, *J* = 7.3, 2.8 Hz, 5H), 7.23 – 7.18 (m, 2H), 7.18 – 7.09 (m, 4H), 5.30 (d, *J* = 12.6 Hz, 1H), 5.05 (d, *J* = 12.6 Hz, 1H), 4.69 (d, *J* = 14.9 Hz, 1H), 4.40 (d, *J* = 15.0 Hz, 1H), 4.04 (dd, *J* = 12.1, 5.7 Hz, 1H), 3.51 (dd, *J* = 18.1, 1.7 Hz, 1H), 3.40 (d, *J* = 18.2 Hz, 1H), 2.43 (t, *J* = 12.7 Hz, 1H), 2.30 (dd, *J* = 13.4, 5.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.4, 144.9, 140.1, 139.5, 138.4, 137.1, 133.3, 130.0, 129.4, 129.3, 128.9, 128.3, 128.0, 127.8, 122.8, 122.5, 121.3, 110.5, 72.7, 47.5, 46.7, 39.9, 36.2. HRMS (ESI+) calculated for $C_{27}H_{23}ClNO_3$ ($M+H^+$) 444.1361; found 444.1355.

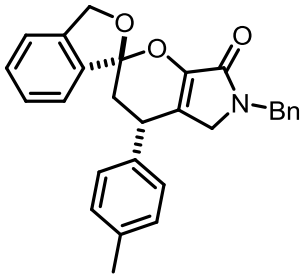


3ad

6'-benzyl-4'-(4-fluorophenyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

Yellow solid, 54 mg, yield 63%, m.p. 143-144 °C, *dr* = 12:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61 – 7.15 (m, 12H), 7.04 (t, *J* = 8.3 Hz, 2H), 5.39 (d, *J* = 12.6 Hz, 1H), 5.13 (d, *J* = 12.6 Hz, 1H), 4.77 (d, *J* = 14.9 Hz, 1H), 4.47 (d, *J* = 14.9 Hz, 1H), 4.12 (dd, *J* = 12.0, 5.7 Hz, 1H), 3.59 (dd, *J* = 18.2, 1.7 Hz, 1H), 3.47 (d, *J* = 18.2 Hz, 1H), 2.51 (t, *J* = 12.7 Hz, 1H), 2.38 (dd, *J* = 13.3, 5.8 Hz, 1H). ^{13}C NMR (101 MHz,

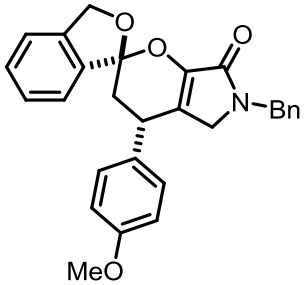
$CDCl_3$) δ 165.6, 163.4, 161.0, 144.8, 140.2, 138.5, 137.2, 136.8, 136.7, 130.1, 129.6, 129.5, 128.9, 128.3, 128.0, 127.8, 123.2, 122.6, 121.4, 116.3, 116.0, 110.6, 72.8, 47.6, 46.7, 39.9, 36.1. HRMS (ESI+) calculated for $C_{27}H_{23}FNO_3$ ($M+H^+$) 428.1656; found 428.1652.



3ae

6'-benzyl-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c] pyrrol]-7'(4'H)-one

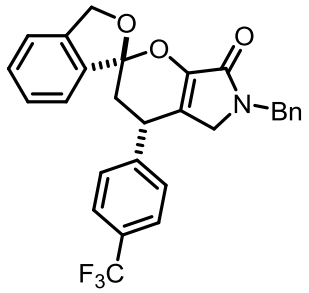
Yellow solid, 79 mg, yield 94%, m.p. 174-175 °C, *dr* = 16:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.32 (m, 3H), 7.32 – 7.22 (m, 4H), 7.23 – 7.16 (m, 2H), 7.16 – 7.07 (m, 4H), 5.36 (d, *J* = 12.6 Hz, 1H), 5.10 (d, *J* = 12.6 Hz, 1H), 4.74 (d, *J* = 15.0 Hz, 1H), 4.44 (d, *J* = 15.0 Hz, 1H), 4.06 (dd, *J* = 12.1, 5.8 Hz, 1H), 3.56 (dd, *J* = 18.2, 1.7 Hz, 1H), 3.48 (dd, *J* = 18.2, 1.2 Hz, 1H), 2.51 (dd, *J* = 13.5, 12.1 Hz, 1H), 2.38 – 2.29 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 144.6, 140.2, 138.6, 137.9, 137.2, 129.93, 129.85, 128.8, 128.3, 128.0, 127.9, 127.7, 123.8, 122.5, 121.3, 110.6, 72.7, 47.6, 46.67, 39.9, 36.3, 21.2. HRMS (ESI+) calculated for $C_{28}H_{26}NO_3$ ($M+H^+$) 424.1907; found 424.1910.



3af

6'-benzyl-4'-(4-methoxyphenyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

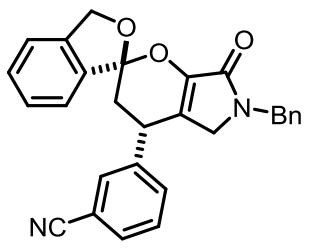
Colorless oil, 79 mg, yield 90%, *dr* = 16:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65 – 7.14 (m, 12H), 7.04 – 6.80 (m, 2H), 5.46 (d, *J* = 12.6 Hz, 1H), 5.21 (d, *J* = 12.6 Hz, 1H), 4.85 (d, *J* = 15.0 Hz, 1H), 4.54 (d, *J* = 15.0 Hz, 1H), 4.16 (dd, *J* = 12.1, 5.7 Hz, 1H), 3.88 (s, 3H), 3.67 (dd, *J* = 18.3, 1.7 Hz, 1H), 3.57 (dd, *J* = 18.2, 1.3 Hz, 1H), 2.60 (dd, *J* = 13.4, 12.1 Hz, 1H), 2.45 (dd, *J* = 13.4, 5.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 158.9, 144.5, 140.2, 138.6, 137.2, 132.9, 130.0, 129.0, 128.8, 128.3, 128.0, 127.7, 124.0, 122.5, 121.3, 114.6, 110.6, 72.7, 55.5, 47.7, 46.7, 40.0, 35.9. HRMS (ESI+) calculated for $C_{28}H_{26}NO_4$ ($M+H^+$) 440.1856; found 424.1853.



3ag

6'-benzyl-4'-(4-(trifluoromethyl)phenyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

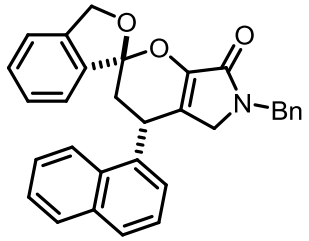
White solid, 81 mg, yield 85%, m.p. 194-195 °C, *dr* = 16:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 8.0 Hz, 2H), 7.39 – 7.25 (m, 5H), 7.25 – 7.16 (m, 5H), 7.16 – 7.11 (m, 2H), 5.29 (d, *J* = 12.6 Hz, 1H), 5.04 (d, *J* = 12.6 Hz, 1H), 4.68 (d, *J* = 15.0 Hz, 1H), 4.37 (d, *J* = 15.0 Hz, 1H), 4.10 (dd, *J* = 12.1, 5.8 Hz, 1H), 3.38 (dd, *J* = 18.2, 1.2 Hz, 1H), 2.44 (dd, *J* = 13.4, 12.1 Hz, 1H), 2.30 (dd, *J* = 13.4, 5.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.4, 145.2, 140.1, 138.2, 137.1, 130.1, 128.9, 128.4, 128.3, 128.1, 127.8, 126.24, 126.20, 122.5, 122.2, 121.4, 110.4, 72.8, 47.5, 46.7, 39.7, 36.7. HRMS (ESI+) calculated for $C_{28}H_{23}F_3NO_3$ ($M+H^+$) 478.1625; found 478.1627.



3ah

3-(6'-benzyl-7'-oxo-4',5',6',7'-tetrahydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-4'-yl)benzonitrile

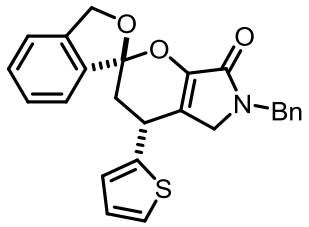
Yellow oil, 63 mg, yield 72.5%, *dr* = 25:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59 – 7.50 (m, 2H), 7.49 – 7.44 (m, 1H), 7.44 – 7.32 (m, 4H), 7.32 – 7.22 (m, 5H), 7.21 – 7.16 (m, 2H), 5.34 (d, *J* = 12.6 Hz, 1H), 5.09 (d, *J* = 12.6 Hz, 1H), 4.75 (d, *J* = 15.0 Hz, 1H), 4.43 (d, *J* = 15.0 Hz, 1H), 4.20 – 4.03 (m, 1H), 3.55 (dd, *J* = 18.2, 1.8 Hz, 1H), 3.39 (dd, *J* = 18.2, 1.2 Hz, 1H), 2.47 (dd, *J* = 13.3, 12.0 Hz, 1H), 2.35 (dd, *J* = 13.4, 5.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.3, 145.4, 142.7, 140.1, 138.1, 137.0, 132.5, 131.6, 131.4, 130.2, 128.9, 128.3, 128.1, 127.9, 122.5, 121.6, 121.4, 118.6, 113.4, 110.4, 72.9, 47.3, 46.7, 39.7, 36.6. HRMS (ESI+) calculated for $C_{28}H_{23}N_2O_3$ ($M+H^+$) 435.1703; found 424.1710.



3aj

6'-benzyl-4'-(naphthalen-1-yl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

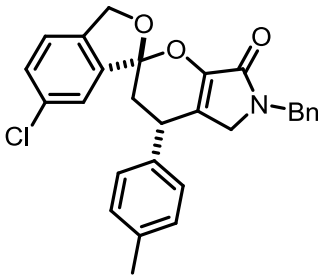
Yellow oil, 41 mg, yield 45%, *dr* = 2:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.18 – 7.98 (m, 1H), 7.89 – 7.74 (m, 3H), 7.52 – 7.42 (m, 5H), 7.41 – 7.24 (m, 10H), 7.23 – 7.08 (m, 5H), 5.41 (d, *J* = 12.6 Hz, 1.41H), 5.17 (d, *J* = 12.5 Hz, 1.55H), 5.07 (dd, *J* = 11.6, 6.2 Hz, 1H), 4.69 (t, *J* = 16.1 Hz, 1.70H), 4.57 – 4.43 (m, 1.91H), 3.68 – 3.52 (m, 2.49H), 3.34 (d, *J* = 18.3 Hz, 0.48H), 3.10 (s, 0.48H), 2.66 – 2.48 (m, 2H), 2.33 (d, *J* = 10.0 Hz, 0.48H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 145.6, 140.2, 138.6, 137.5, 137.3, 137.2, 134.2, 131.8, 130.0, 129.8, 129.3, 129.0, 128.93, 128.85, 128.3, 128.0, 127.9, 127.8, 127.7, 126.7, 126.2, 125.9, 125.7, 124.7, 123.7, 122.6, 122.5, 121.3, 110.7, 72.8, 47.8, 46.7, 39.8, 31.1. HRMS (ESI+) calculated for $C_{31}H_{26}NO_3$ ($M+H^+$) 460.1907; found 460.1907.



3ak

6'-benzyl-4'-(thiophen-2-yl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

White solid, 36 mg, yield 43%, m.p. 184-185 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.39 (m, 2H), 7.37 (dd, *J* = 6.8, 1.1 Hz, 1H), 7.34 – 7.27 (m, 3H), 7.26 (s, 2H), 7.24 – 7.19 (m, 3H), 7.07 – 6.65 (m, 2H), 5.36 (d, *J* = 12.6 Hz, 1H), 5.11 (d, *J* = 12.6 Hz, 1H), 4.75 (d, *J* = 15.0 Hz, 1H), 4.62 – 4.25 (m, 2H), 3.66 (dd, *J* = 18.2, 1.8 Hz, 1H), 3.58 (dd, *J* = 18.2, 1.4 Hz, 1H), 2.61 (dd, *J* = 13.3, 12.0 Hz, 1H), 2.47 (dd, *J* = 13.3, 5.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.5, 144.1, 143.9, 140.2, 138.4, 137.3, 130.1, 128.9, 128.3, 128.1, 127.8, 127.3, 125.5, 124.7, 123.1, 122.7, 121.4, 110.6, 72.8, 47.7, 46.7, 40.2, 32.0. HRMS (ESI+) calculated for $C_{25}H_{22}NO_3S$ ($M+H^+$) 416.1315; found 416.1319.

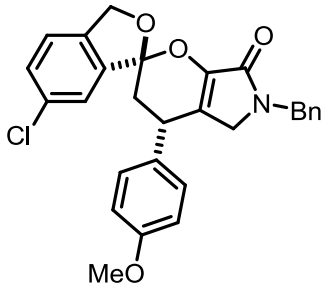


3be

6'-benzyl-6-chloro-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

White solid, 84 mg, yield 92%, m.p. 209-210 °C, *dr* = 16:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (dq, *J* = 3.7, 1.9 Hz, 2H), 7.32 – 7.17 (m, 7H), 7.12 (q, *J* = 8.0 Hz, 4H), 5.31 (d, *J* = 12.8 Hz, 1H), 5.07 (d, *J* = 12.7 Hz, 1H), 4.75 (d, *J* = 15.0 Hz, 1H), 4.43 (d, *J* = 15.0 Hz, 1H), 4.04 (dd, *J* = 12.0, 5.8 Hz, 1H), 3.56 (dd, *J* = 18.3, 1.7 Hz, 1H), 3.48 (d, *J* = 18.4 Hz, 1H), 2.46 (dd, *J* = 13.4, 12.0 Hz, 1H), 2.38 – 2.28 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.5, 144.3, 140.5, 138.6, 137.6, 137.4, 137.2,

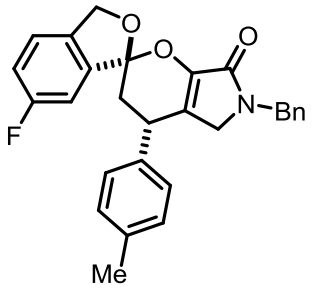
133.9, 130.2, 129.9, 128.9, 128.3, 127.8, 127.7, 124.0, 123.1, 122.6, 110.1, 72.4, 47.6, 46.7, 39.7, 36.2, 21.2. HRMS (ESI+) calculated for $C_{28}H_{25}ClNO_3$ ($M+H^+$) 458.1517; found 458.1509.



3bf

6'-benzyl-6-chloro-4'-(4-methoxyphenyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

White solid, 68 mg, yield 72%, m.p. 179-181 ^{o}C, *dr* = 12:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (dd, *J* = 4.6, 2.6 Hz, 2H), 7.34 – 7.17 (m, 7H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 7.9 Hz, 2H), 5.31 (d, *J* = 12.7 Hz, 1H), 5.07 (d, *J* = 12.7 Hz, 1H), 4.75 (d, *J* = 14.8 Hz, 1H), 4.43 (d, *J* = 14.9 Hz, 1H), 4.03 (dd, *J* = 12.0, 5.7 Hz, 1H), 3.78 (s, 3H), 3.56 (d, *J* = 18.5 Hz, 1H), 3.47 (d, *J* = 18.3 Hz, 1H), 2.45 (t, *J* = 12.7 Hz, 1H), 2.34 (dd, *J* = 13.3, 5.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.5, 159.1, 144.3, 140.5, 138.6, 137.2, 133.9, 132.6, 130.3, 129.0, 128.9, 128.3, 127.8, 124.2, 123.1, 122.6, 114.6, 110.1, 72.4, 55.5, 47.7, 46.7, 39.8, 35.8. HRMS (ESI+) calculated for $C_{28}H_{25}ClNO_4$ ($M+H^+$) 474.1467; found 474.1461.

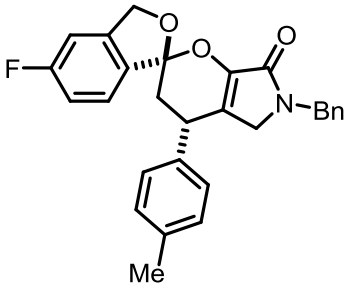


3ce

6'-benzyl-6-fluoro-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

Colorless oil, 57 mg, yield 65%, *dr* = 16:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (dq, *J* = 3.6, 1.8 Hz, 2H), 7.32 – 7.17 (m, 6H), 7.12 (q, *J* = 8.1 Hz, 4H), 5.31 (d, *J* =

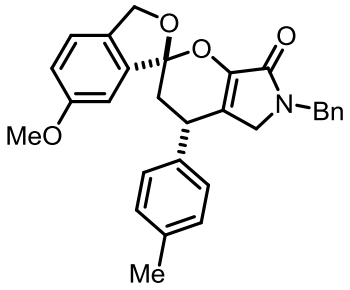
12.8 Hz, 1H), 5.07 (d, *J* = 12.8 Hz, 1H), 4.75 (d, *J* = 15.0 Hz, 1H), 4.43 (d, *J* = 14.9 Hz, 1H), 4.04 (dd, *J* = 12.0, 5.8 Hz, 1H), 3.56 (dd, *J* = 18.2, 1.7 Hz, 1H), 3.51 – 3.43 (dd, *J* = 18.1, 1.5 Hz, 1H), 2.46 (dd, *J* = 13.4, 12.0 Hz, 1H), 2.38 – 2.29 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 165.3, 162.9, 144.5, 142.7, 142.6, 137.8, 137.3, 137.2, 134.5, 129.9, 128.8, 128.2, 127.8, 127.7, 124.2, 124.1, 123.9, 115.5, 115.3, 110.1, 108.7, 108.5, 72.2, 47.6, 46.7, 40.0, 36.3, 21.2. HRMS (ESI+) calculated for $C_{28}H_{25}FNO_3$ ($M+H^+$) 442.1813; found 442.1814.



3de

6'-benzyl-5-fluoro-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano [2,3-c]pyrrol]-7'(4'H)-one

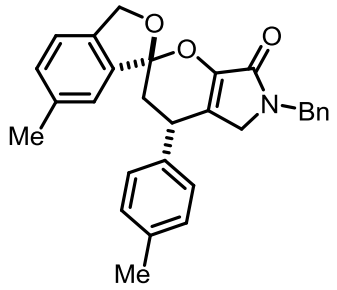
Colorless oil, 63 mg, yield 71%, *dr* = 16:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 – 7.16 (m, 6H), 7.13 (t, *J* = 6.3 Hz, 4H), 7.07 – 6.92 (m, 2H), 5.32 (d, *J* = 12.9 Hz, 1H), 5.06 (d, *J* = 12.9 Hz, 1H), 4.75 (d, *J* = 14.8 Hz, 1H), 4.43 (d, *J* = 14.9 Hz, 1H), 4.05 (dd, *J* = 11.9, 5.6 Hz, 1H), 3.56 (d, *J* = 18.2 Hz, 1H), 3.48 (d, *J* = 18.4 Hz, 1H), 2.47 (t, *J* = 12.7 Hz, 1H), 2.32 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 165.3, 162.9, 144.5, 142.7, 142.6, 137.8, 137.3, 137.2, 134.5, 129.9, 128.8, 128.2, 127.8, 127.7, 124.2, 124.1, 123.9, 115.5, 115.27, 110.1, 108.7, 108.5, 72.1, 47.6, 46.7, 39.9, 36.3, 21.2. HRMS (ESI+) calculated for $C_{28}H_{25}FNO_3$ ($M+H^+$) 442.1813; found 442.1810.



3ee

6'-benzyl-6-methoxy-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

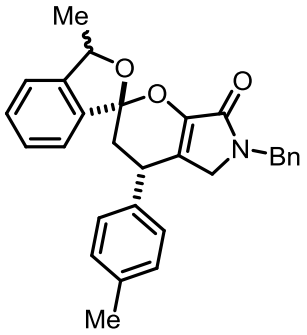
White solid, 75 mg, yield 83%, m.p. 220-221 ^{o}C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.04 (m, 10H), 6.95 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.88 (d, *J* = 2.3 Hz, 1H), 5.30 (d, *J* = 11.9 Hz, 1H), 5.05 (d, *J* = 12.0 Hz, 1H), 4.75 (d, *J* = 15.0 Hz, 1H), 4.44 (d, *J* = 15.0 Hz, 1H), 4.15 – 3.93 (m, 1H), 3.80 (s, 3H), 3.56 (dd, *J* = 18.2, 1.7 Hz, 1H), 3.48 (dd, *J* = 18.2, 1.3 Hz, 1H), 2.47 (dd, *J* = 13.4, 12.0 Hz, 1H), 2.40 – 2.27 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 160.0, 144.6, 140.0, 138.0, 137.3, 132.0, 129.9, 128.8, 128.3, 127.9, 127.7, 123.8, 122.1, 117.2, 110.5, 106.9, 72.5, 55.8, 47.7, 46.7, 40.0, 36.3, 21.2. HRMS (ESI+) calculated for $C_{29}H_{28}NO_4$ ($M+H^+$) 454.2013; found 454.2011.



3fe

6'-benzyl-6-methyl-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

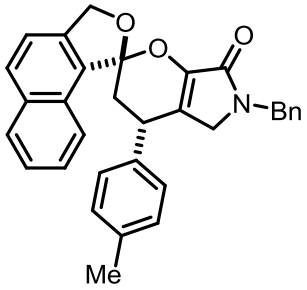
White solid, 62 mg, yield 71%, m.p. 146-147 ^{o}C, *dr* = 11:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 6.81 (m, 13H), 5.31 (d, *J* = 12.3 Hz, 1H), 5.06 (d, *J* = 12.3 Hz, 1H), 4.75 (d, *J* = 15.0 Hz, 1H), 4.43 (d, *J* = 14.9 Hz, 1H), 4.05 (dd, *J* = 12.1, 5.8 Hz, 1H), 3.55 (dd, *J* = 18.2, 1.7 Hz, 1H), 3.47 (d, *J* = 18.2 Hz, 1H), 2.50 (t, *J* = 12.8 Hz, 1H), 2.42 – 2.22 (m, 7H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 144.6, 138.8, 138.0, 137.8, 137.3, 137.2, 137.2, 130.8, 129.8, 128.8, 128.3, 127.9, 127.7, 123.7, 123.0, 121.0, 110.6, 72.6, 47.6, 46.7, 39.9, 36.3, 21.4, 21.2. HRMS (ESI+) calculated for $C_{29}H_{28}NO_3$ ($M+H^+$) 438.2064; found 438.2069.



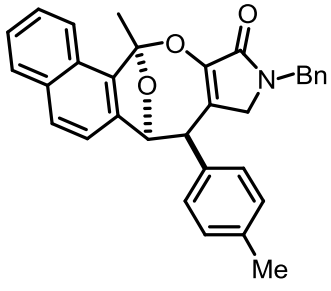
3ge

6'-benzyl-3,6-dimethyl-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[isobenzofuran-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

Colorless oil, 82 mg, yield 91%, *dr* = 11:6.3:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 – 6.80 (m, 22.6H), 5.59 (d, *J* = 6.5 Hz, 0.65H), 5.40 (d, *J* = 6.5 Hz, 1H), 4.73 (dd, *J* = 15.0, 6.0 Hz, 1.64H), 4.45 (dd, *J* = 14.9, 12.6 Hz, 1.66H), 4.06 (dd, *J* = 11.8, 5.7 Hz, 1.66H), 3.56 (dd, *J* = 18.2, 1.7 Hz, 1.58H), 3.47 (d, *J* = 18.2 Hz, 1.50H), 2.68 – 2.37 (m, 1.71H), 2.39 – 2.12 (m, 6.5H), 1.63 (d, *J* =6.6 Hz, 3H), 1.35 (d, *J* = 6.5 Hz, 1.7H), 1.35 (d, *J* = 6.4 Hz, 0.26H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 144.6, 144.4, 138.5, 138.1, 137.3, 137.2, 130.02, 129.97, 129.8, 128.8, 128.3, 128.12, 128.07, 127.9, 127.7, 123.7, 123.6, 122.7, 122.4, 121.2, 121.1, 109.9, 109.4, 81.1, 79.5, 47.70, 47.65, 46.7, 40.4, 40.3, 36.4, 36.1, 23.7, 21.2, 21.1. HRMS (ESI+) calculated for $C_{29}H_{28}NO_3$ ($M+H^+$) 438.2064; found 438.2059.



3he



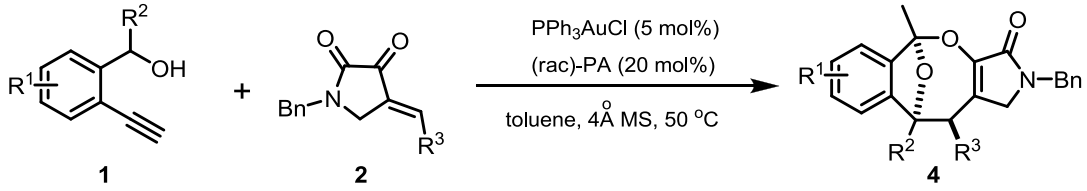
4he

6'-benzyl-4'-(p-tolyl)-5',6'-dihydro-3H,3'H-spiro[naphtho[1,2-c]furan-1,2'-pyrano[2,3-c]pyrrol]-7'(4'H)-one

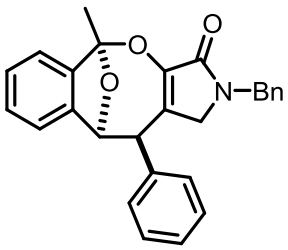
10-benzyl-13-methyl-8-(p-tolyl)-7,8,9,10-tetrahydro-7,13-epoxynaphtho[2',1':6,7]oxocino[2,3-c]pyrrol-11(13H)-one

White solid, 44 mg, yield 46%, m.p. 180-181 °C, **3he** : **4he** = 2.5:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.68 – 8.62 (m, 0.11H), 8.34 (dd, *J* = 8.4, 1.1 Hz, 0.28H), 8.10 – 7.98 (m, 0.76H), 7.90 (dd, *J* = 8.1, 1.8 Hz, 1.75H), 7.81 – 7.67 (m, 0.34H), 7.67 – 7.04 (m, 16H), 6.76 (d, *J* = 7.5 Hz, 0.56H), 6.25 (d, *J* = 8.3 Hz, 0.27H), 5.59 – 5.41 (m, 1H), 5.30 (d, *J* = 12.8 Hz, 0.72H), 5.09 (d, *J* = 6.0 Hz, 0.27H), 4.78 (dd, *J* = 15.1, 2.1 Hz, 1H), 4.59 (d, *J* = 6.0 Hz, 0.28H), 4.45 (d, *J* = 15.0 Hz, 0.73H), 4.16 (dd, *J* = 11.9, 5.7 Hz, 0.72H), 4.03 (d, *J* = 15.0 Hz, 0.28H), 3.60 (dd, *J* = 18.3, 1.7 Hz, 0.77H), 3.54 (dd, *J* = 18.2, 1.3 Hz, 0.76H), 3.32 (d, *J* = 17.8 Hz, 0.28H), 3.19 (dd, *J* = 17.9, 1.1 Hz, 0.28H), 2.98 (dd, *J* = 13.5, 12.0 Hz, 0.73H), 2.44 (dd, *J* = 13.5, 5.7 Hz, 0.74H), 2.36 (s, 0.81H), 2.34 (s, 0.87H), 2.32 (s, 2.11H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 165.7, 144.6, 138.2, 138.0, 137.9, 137.3, 137.1, 134.6, 134.5, 134.4, 133.9, 133.1, 132.6, 132.1, 131.9, 131.4, 130.3, 129.9, 129.5, 129.4, 129.3, 129.1, 128.9, 128.8, 128.4, 128.2, 128.0, 127.8, 127.6, 127.5, 127.4, 126.6, 126.4, 126.0, 125.9, 124.4, 123.9, 123.5, 121.3, 119.1, 118.8, 112.5, 112.4, 85.0, 73.4, 65.6, 55.7, 49.0, 47.8, 46.8, 46.7, 40.9, 36.5, 26.4, 21.4, 21.3. HRMS (ESI+) calculated for $C_{32}H_{28}NO_3$ ($M+H^+$) 474.2064; found 474.2059.

6. Synthesis and Characterization of [4+4] annulation products



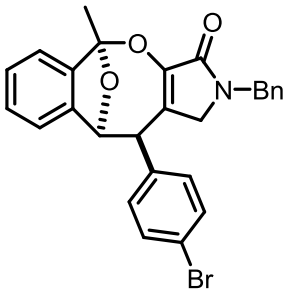
To a shlenck tube, a magnetic stir, dioxopyrrolidiens **2** (0.2 mmol), (*rac*)-PA (12 mg, 20% mol), 4Å MS (40 mg) and toluene (2 mL) were sequentially added under nitrogen atmosphere. The mixture was stirred for 2 h at room temperature, then Ph_3PAuCl (5 mg, 5 mol%) and arylalkynols **1** (0.3 mmol, 1.5 equiv.) was added. The reaction mixture was stirred at 50 ℃ until complete disappearance of **2** observed by TLC. The mixture was cooled to room temperature, filtered through kieselguhr and the solvent was removed in vacuo. The product was purified by silica gel column chromatography (petrol ether/EtOAc = 2 :1) to afford the compound **4**.



4aa

2-benzyl-5-methyl-11-phenyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino [2,3-c]pyrrol-3(5H)-one

Colorless oil, 77 mg, yield 94%, *dr* = 10:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (d, *J* = 7.5 Hz, 1H), 7.39 – 7.28 (m, 5H), 7.27 – 7.09 (m, 7H), 7.08 – 6.91 (m, 2H), 6.14 (d, *J* = 7.6 Hz, 1H), 5.35 (d, *J* = 5.9 Hz, 1H), 4.83 (d, *J* = 15.0 Hz, 1H), 4.58 (d, *J* = 6.0 Hz, 1H), 4.11 (d, *J* = 15.0 Hz, 1H), 3.42 (d, *J* = 17.8 Hz, 1H), 3.24 (d, *J* = 17.8 Hz, 1H), 2.07 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 141.9, 139.2, 138.6, 137.0, 135.0, 130.3, 129.1, 128.8, 128.6, 128.2, 128.13, 128.07, 127.6, 123.7, 122.1, 118.6, 111.3, 85.0, 54.9, 48.9, 46.7, 24.6. HRMS (ESI+) calculated for $C_{27}H_{24}NO_3$ ($M+H^+$) 410.1751; found 410.1755.

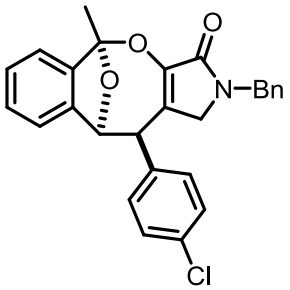


4ab

2-benzyl-11-(4-bromophenyl)-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 78 mg, yield 80%, m.p. 125-126 ^{o}C, *dr* = 6:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65 – 6.99 (m, 13H), 6.84 (d, *J* = 7.9 Hz, 2H), 6.21 (d, *J* = 7.6 Hz, 1H), 5.33 (d, *J* = 6.1 Hz, 1.15H), 4.82 (d, *J* = 14.9 Hz, 1H), 4.54 (d, *J* = 5.9 Hz, 1H), 4.43 (d, *J* = 7.1 Hz, 0.32H), 4.11 (d, *J* = 15.0 Hz, 1H), 3.77 (s, 0.16H), 3.34 (d, *J* = 17.7 Hz, 1H), 3.26 – 3.18 (m, 1.31H), 2.08 (d, *J* = 13.3 Hz, 3.48H).[1] ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 142.1, 138.9, 138.6, 136.9, 134.1, 132.2, 132.0, 131.8, 130.0,

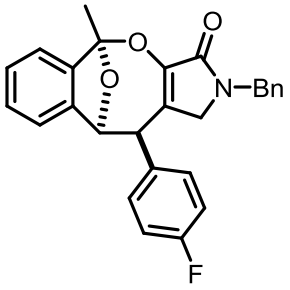
129.3, 128.8, 128.3, 128.2, 128.1, 127.7, 123.5, 122.3, 122.2, 117.8, 111.4, 84.6, 54.4, 48.8, 46.7, 24.6. HRMS (ESI+) calculated for $C_{27}H_{23}BrNO_3$ ($M+H^+$) 488.0856; found 488.0858.



4ac

2-benzyl-11-(4-chlorophenyl)-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

Colorless oil, 74 mg, yield 84%, *dr* = 2.5:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.34 (m, 3H), 7.35 – 7.14 (m, 9H), 7.12 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.10 – 7.01 (m, 0.78H), 6.90 (d, *J* = 7.9 Hz, 2H), 6.21 (d, *J* = 7.5 Hz, 1H), 5.56 – 5.18 (m, 1.39H), 4.81 (d, *J* = 15.0 Hz, 1H), 4.56 (d, *J* = 5.9 Hz, 1H), 4.43 (d, *J* = 5.0 Hz, 0.81H), 4.12 (d, *J* = 15.0 Hz, 1H), 3.78 (s, 0.38H), 3.34 (d, *J* = 17.8 Hz, 1H), 3.27 – 3.16 (m, 1.76H), 2.08 (d, *J* = 13.7 Hz, 4.17H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 142.1, 138.94, 138.6, 136.9, 134.1, 133.6, 131.6, 129.6, 129.28, 129.25, 128.8, 128.24, 128.19, 128.1, 127.7, 123.5, 122.3, 117.9, 111.4, 84.7, 54.4, 48.8, 46.7, 24.6. HRMS (ESI+) calculated for $C_{27}H_{23}ClNO_3$ ($M+H^+$) 444.1361; found 444.1369.

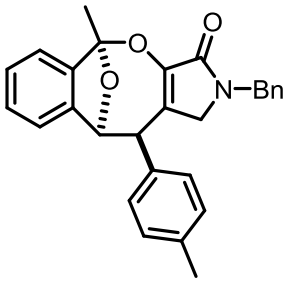


4ad

4ad 2-benzyl-11-(4-fluorophenyl)-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 64 mg, yield 75%, m.p. 128-129 ^{o}C, *dr* = 4:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 7.6 Hz, 1.21H), 7.43 – 7.28 (m, 2.70H), 7.28 – 7.09 (m,

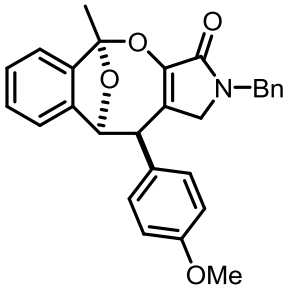
7.47H), 7.08 – 6.97 (m, 3.17H), 6.92 (t, *J* = 6.7 Hz, 1.89H), 6.18 (d, *J* = 7.5 Hz, 1H), 5.52 – 5.32 (m, 1.26H), 4.82 (d, *J* = 15.0 Hz, 1H), 4.57 (d, *J* = 6.0 Hz, 1H), 4.43 (d, *J* = 4.1 Hz, 0.53H), 4.12 (d, *J* = 15.0 Hz, 1H), 3.79 (s, 0.26H), 3.35 (d, *J* = 17.8 Hz, 1H), 3.23 (d, *J* = 17.9 Hz, 1.5H), 2.08 (d, *J* = 14.6 Hz, 3.79H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 163.8, 161.3, 141.9, 139.0, 138.6, 136.9, 131.9, 131.8, 129.2, 128.8, 128.2, 128.1, 127.7, 123.5, 122.3, 118.2, 115.7, 115.5, 111.4, 84.9, 54.2, 48.9, 46.7, 24.7. HRMS (ESI+) calculated for $C_{27}H_{23}FNO_3$ ($M+H^+$) 428.1656; found 428.1653.



4ae

2-benzyl-5-methyl-11-(p-tolyl)-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino [2,3-c]pyrrol-3(5H)-one

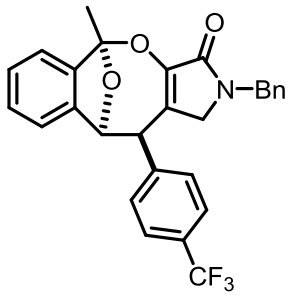
White solid, 67 mg, yield 79%, m.p. 153-154 °C, *dr* = 8:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 – 6.97 (m, 13H), 6.84 (d, *J* = 7.6 Hz, 2H), 6.21 (d, *J* = 7.5 Hz, 1H), 5.32 (d, *J* = 5.8 Hz, 1.13H), 4.82 (d, *J* = 15.0 Hz, 1H), 4.54 (d, *J* = 5.9 Hz, 1H), 4.42 (q, *J* = 15.0 Hz, 0.26H), 4.10 (d, *J* = 15.0 Hz, 1H), 3.79 (s, 0.13H), 3.41 (d, *J* = 17.8 Hz, 1H), 3.32 (d, *J* = 17.5 Hz, 0.14H), 3.22 (d, *J* = 18.0 Hz, 1.12H), 2.36 (s, 3.39H), 2.06 (s, 3.39H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 141.6, 139.2, 138.4, 137.7, 136.9, 131.8, 130.1, 129.7, 129.1, 128.9, 128.6, 127.99, 127.97, 127.9, 127.5, 123.6, 121.9, 118.9, 111.0, 84.9, 54.4, 48.8, 46.5, 24.5, 21.2. HRMS (ESI+) calculated for $C_{28}H_{26}NO_3$ ($M+H^+$) 424.1908; found 424.1909.



4af

2-benzyl-11-(4-methoxyphenyl)-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino[2,3-c]pyrrol-3(5H)-one

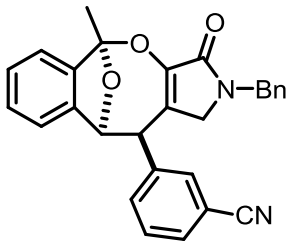
White solid, 76 mg, yield 87%, m.p. 96-97 °C, *dr* = 10:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (d, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 7.4 Hz, 1H), 7.30 – 7.07 (m, 7H), 6.85 (d, *J* = 3.5 Hz, 4H), 6.24 (d, *J* = 7.5 Hz, 1H), 5.32 (d, *J* = 5.9 Hz, 1.10H), 4.83 (d, *J* = 15.0 Hz, 1H), 4.53 (d, *J* = 6.0 Hz, 1.31H), 4.10 (d, *J* = 15.0 Hz, 1H), 3.82 (s, 3.30H), 3.39 (d, *J* = 17.8 Hz, 1.07H), 3.23 (d, *J* = 17.8 Hz, 1.14H), 2.07 (s, 3.30H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 159.4, 141.7, 139.4, 138.6, 137.1, 131.4, 129.0, 128.8, 128.2, 128.1, 127.7, 127.0, 123.7, 122.2, 119.1, 113.9, 111.2, 85.1, 55.5, 54.1, 49.0, 46.7, 24.7. HRMS (ESI+) calculated for $C_{28}H_{26}NO_4$ ($M+H^+$) 440.1856; found 440.1863.



4ag

2-benzyl-5-methyl-11-(4-(trifluoromethyl)phenyl)-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino[2,3-c]pyrrol-3(5H)-one

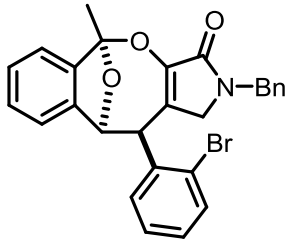
White solid, 76 mg, yield 80%, m.p. 105-106 °C, *dr* = 10:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 7.9 Hz, 2H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.39 (s, 1H), 7.30 – 7.16 (m, 5H), 7.17 – 7.03 (m, 4H), 6.13 (d, *J* = 7.5 Hz, 1H), 5.37 (d, *J* = 5.8 Hz, 1.10H), 4.84 (d, *J* = 15.0 Hz, 1H), 4.66 (d, *J* = 5.9 Hz, 1H), 4.57 – 4.31 (m, 0.20H), 4.11 (d, *J* = 15.0 Hz, 1H), 3.88 (s, 0.11H), 3.35 (d, *J* = 17.7 Hz, 1.04H), 3.23 (d, *J* = 17.8 Hz, 1.14H), 2.07 (s, 3.28H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 142.3, 138.7, 138.6, 136.8, 130.7, 129.4, 128.8, 128.4, 128.2, 127.8, 125.59, 125.55, 123.3, 122.4, 117.3, 111.5, 84.6, 54.8, 48.8, 46.7, 24.6. HRMS (ESI+) calculated for $C_{28}H_{23}F_3NO_3$ ($M+H^+$) 478.1625; found 478.1634.



4ah

3-(2-benzyl-5-methyl-3-oxo-1,2,3,5,10,11-hexahydro-5,10-epoxybenzo[6,7]oxocino [2,3-c]pyrrol-11-yl)benzonitrile

Colorless oil, 56 mg, yield 65%, *dr* = 14:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63 (dd, *J* = 8.2, 3.9 Hz, 1H), 7.57 – 7.26 (m, 3H), 7.32 – 6.91 (m, 9H), 6.05 (dd, *J* = 7.8, 4.0 Hz, 1H), 5.33 (t, *J* = 5.2 Hz, 1.07H), 4.85 (dd, *J* = 15.1, 3.9 Hz, 1H), 4.61 (t, *J* = 5.0 Hz, 1H), 4.43 (d, *J* = 4.0 Hz, 0.14H), 4.09 (dd, *J* = 15.1, 4.0 Hz, 1H), 3.81 (d, *J* = 3.8 Hz, 0.07H), 3.23 (dd, *J* = 7.5, 4.1 Hz, 2.15H), 2.05 (d, *J* = 4.4 Hz, 3.24H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.2, 142.5, 138.6, 138.5, 136.9, 136.8, 134.7, 133.7, 132.0, 129.63, 129.55, 128.9, 128.4, 128.2, 127.8, 123.1, 122.7, 118.4, 116.5, 113.0, 111.7, 84.5, 54.6, 48.7, 46.8, 24.7. HRMS (ESI+) calculated for $C_{28}H_{23}N_2O_3$ ($M+H^+$) 435.1703; found 435.1705.

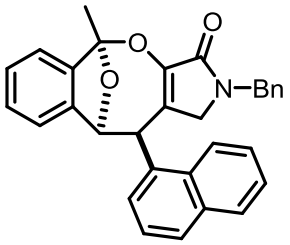


4ai

2-benzyl-11-(2-bromophenyl)-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 71 mg, yield 73%, m.p. 175-176 ^{o}C, *dr* = 6:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 (dd, *J* = 7.7, 1.6 Hz, 1.22H), 7.48 (d, *J* = 7.6 Hz, 1.19H), 7.42 (d, *J* = 9.7 Hz, 0.64H), 7.41 – 7.29 (m, 1.76H), 7.29 – 7.18 (m, 4.70H), 7.20 – 7.02 (m, 5H), 6.50 (dd, *J* = 7.4, 2.0 Hz, 1H), 6.02 (d, *J* = 7.5 Hz, 1H), 5.56 (d, *J* = 6.0 Hz, 1H), 5.44 (s, 0.16H), 5.14 (d, *J* = 6.1 Hz, 1H), 4.83 (d, *J* = 15.0 Hz, 1H), 4.60 – 4.38 (m, 0.49H), 4.14 (d, *J* = 15.0 Hz, 1H), 3.36 (d, *J* = 17.6 Hz, 1H), 3.26 (d, *J* = 18.0 Hz, 1.32H), 2.08 (s, 3.48H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 142.1, 138.9, 138.6,

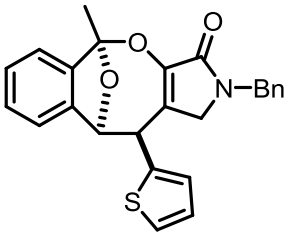
136.7, 134.3, 133.1, 132.6, 129.6, 129.0, 128.7, 128.6, 128.03, 127.98, 127.9, 127.6, 126.9, 125.2, 123.5, 122.1, 117.4, 111.3, 82.0, 53.5, 48.8, 46.6, 24.5. HRMS (ESI+) calculated for $C_{27}H_{23}BrNO_3$ ($M+H^+$) 488.0856; found 488.0858.



4aj

2-benzyl-5-methyl-11-(naphthalen-1-yl)-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 59 mg, yield 64%, m.p. 145-146 °C, *dr* = 4:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 8.4 Hz, 1H), 8.17 (d, *J* = 8.4 Hz, 0.29H), 8.05 – 7.81 (m, 3.11H), 7.74 – 7.48 (m, 5.25H), 7.39 (td, *J* = 7.5, 4.2 Hz, 1.44H), 7.24 (dq, *J* = 8.0, 6.1 Hz, 4.22H), 7.19 – 7.07 (m, 3.20H), 6.73 (d, *J* = 7.0 Hz, 1H), 5.83 (d, *J* = 7.5 Hz, 1H), 5.63 (d, *J* = 5.9 Hz, 1H), 5.53 (d, *J* = 5.6 Hz, 1.27H), 4.89 (d, *J* = 14.9 Hz, 1.27H), 4.49 (s, 0.55H), 4.17 (d, *J* = 14.8 Hz, 1H), 3.60 (d, *J* = 17.7 Hz, 1H), 3.28 (d, *J* = 17.8 Hz, 1.61H), 2.15 (d, *J* = 6.3 Hz, 3.81H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 142.6, 139.5, 138.5, 136.9, 133.9, 131.8, 130.7, 129.5, 129.2, 129.0, 128.8, 128.7, 128.2, 128.1, 127.9, 127.7, 127.3, 126.3, 124.8, 123.9, 122.03, 121.98, 118.7, 111.4, 83.2, 49.8, 49.0, 46.8, 24.7. HRMS (ESI+) calculated for $C_{31}H_{26}NO_3$ ($M+H^+$) 460.1907; found 460.1911.

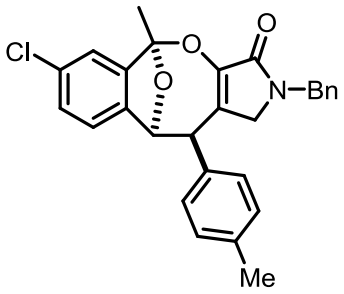


4ak

2-benzyl-5-methyl-11-(thiophen-2-yl)-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 63 mg, yield 76%, m.p. 189-191 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (s, 1H), 7.41 – 7.33 (m, 1H), 7.29 – 7.15 (m, 5H), 7.17 – 7.04 (m, 2H), 6.97 (dd,

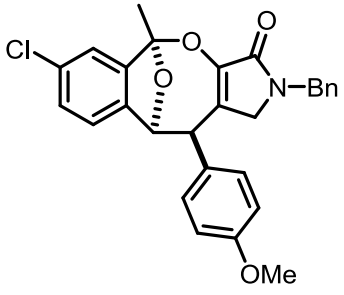
J = 5.2, 3.5 Hz, 1H), 6.59 (d, *J* = 3.4 Hz, 1H), 6.37 (d, *J* = 7.5 Hz, 1H), 5.36 (d, *J* = 5.7 Hz, 1H), 5.02 – 4.65 (m, 2H), 4.11 (d, *J* = 15.0 Hz, 1H), 3.56 (d, *J* = 17.9 Hz, 1H), 3.29 (dd, *J* = 17.8, 1.3 Hz, 1H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 141.4, 139.3, 138.4, 137.3, 137.0, 129.3, 128.8, 128.4, 128.2, 127.9, 127.7, 127.3, 125.2, 123.5, 122.1, 118.2, 111.4, 84.9, 49.6, 49.0, 46.7, 24.5. HRMS (ESI+) calculated for $C_{25}H_{22}NO_3S$ ($M+H^+$) 416.1315; found 416.1315.



4be

2-benzyl-7-chloro-5-methyl-11-(p-tolyl)-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 83 mg, yield 91%, m.p. 200-201 ^{o}C, *dr* = 25:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 1.8 Hz, 1.04H), 7.35 – 7.18 (m, 3.57H), 7.22 – 6.95 (m, 5.05H), 6.83 (d, *J* = 7.6 Hz, 2.10H), 6.11 (d, *J* = 8.0 Hz, 1H), 5.30 (d, *J* = 5.9 Hz, 1.04H), 4.82 (d, *J* = 15.0 Hz, 1H), 4.53 (d, *J* = 6.0 Hz, 1.04H), 4.14 (d, *J* = 15.0 Hz, 1.04H), 3.43 (d, *J* = 17.8 Hz, 1.04H), 3.23 (d, *J* = 17.9 Hz, 1.04H), 2.36 (s, 3.12H), 2.04 (s, 3.12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 141.8, 140.6, 138.1, 137.8, 137.0, 135.0, 131.7, 130.1, 129.4, 128.8, 128.5, 128.2, 127.7, 125.0, 119.0, 110.7, 84.9, 54.4, 49.0, 46.6, 24.5, 21.3. HRMS (ESI+) calculated for $C_{28}H_{25}ClNO_3$ ($M+H^+$) 458.1517; found 458.1526.

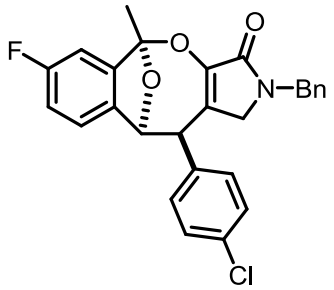


4bf

2-benzyl-7-chloro-11-(4-methoxyphenyl)-5-methyl-1,2,10,11-tetrahydro-5,10-

epoxybenzo[6,7]oxocino[2,3-c]pyrrol-3(5H)-one

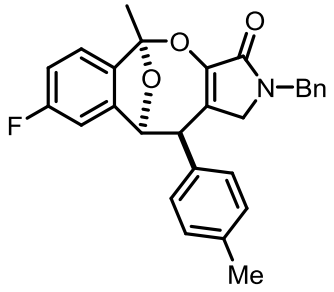
White solid, 68 mg, yield 72%, m.p. 91-92 ^{o}C, *dr* = 33:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 1.8 Hz, 1H), 7.24 – 7.06 (m, 6H), 6.85 (s, 4H), 6.14 (d, *J* = 8.0 Hz, 1H), 5.29 (d, *J* = 5.9 Hz, 1.02H), 4.82 (d, *J* = 15.0 Hz, 1.04H), 4.51 (d, *J* = 5.9 Hz, 1H), 4.14 (d, *J* = 14.9 Hz, 1H), 3.82 (s, 3.08H), 3.41 (d, *J* = 17.8 Hz, 1H), 3.24 (d, *J* = 17.8 Hz, 1.04H), 2.04 (s, 3.09H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 159.5, 141.6, 140.6, 137.8, 137.0, 134.9, 131.3, 128.8, 128.5, 128.2, 127.7, 126.7, 124.9, 122.6, 119.1, 114.0, 110.7, 84.9, 55.5, 53.9, 49.0, 46.7, 24.5. HRMS (ESI+) calculated for $C_{28}H_{25}ClNO_4$ ($M+H^+$) 474.1467; found 474.1470.



4cc

2-benzyl-11-(4-chlorophenyl)-7-fluoro-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino[2,3-c]pyrrol-3(5H)-one

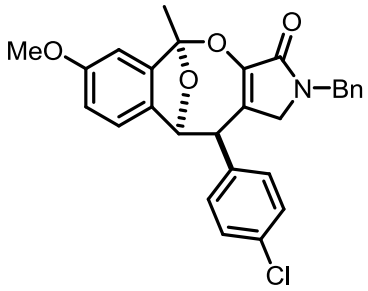
Colorless oil, 71 mg, yield 77%, *dr* = 33:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (dd, *J* = 8.3, 4.7 Hz, 1.03H), 7.37 – 7.29 (m, 2H), 7.29 – 7.19 (m, 3.56H), 7.17 – 7.02 (m, 3.07H), 6.92 (d, *J* = 7.9 Hz, 2H), 5.89 (dd, *J* = 8.3, 2.3 Hz, 1H), 5.28 (d, *J* = 6.0 Hz, 1.03H), 4.81 (d, *J* = 15.0 Hz, 1.03H), 4.56 (d, *J* = 5.9 Hz, 1.03H), 4.14 (d, *J* = 15.0 Hz, 1.03H), 3.37 (d, *J* = 17.7 Hz, 1.02H), 3.23 (dd, *J* = 17.7, 1.2 Hz, 1.04H), 2.05 (s, 3.09H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 162.5, 142.1, 141.5, 136.9, 134.4, 133.2, 131.5, 129.0, 128.9, 128.2, 127.8, 124.0, 117.7, 116.8, 110.9, 110.6, 84.26, 54.2, 48.8, 46.8, 24.7. HRMS (ESI+) calculated for $C_{27}H_{22}ClFNO_3$ ($M+H^+$) 462.1267; found 462.1270.



4de

2-benzyl-8-fluoro-5-methyl-11-(p-tolyl)-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 63 mg, yield 71%, m.p. 144-145 ^{o}C, *dr* = 6:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 (dd, *J* = 8.3, 4.6 Hz, 1.23H), 7.30 – 7.18 (m, 4.22H), 7.13 (dd, *J* = 9.8, 7.3 Hz, 4.45H), 7.10 – 7.01 (m, 1.52H), 6.85 (d, *J* = 7.6 Hz, 1.88H), 5.87 (dd, *J* = 8.4, 2.2 Hz, 1H), 5.28 (d, *J* = 5.8 Hz, 1.17H), 4.82 (d, *J* = 14.9 Hz, 1H), 4.54 (d, *J* = 6.1 Hz, 1.18H), 4.39 (d, *J* = 15.0 Hz, 0.17H), 4.13 (d, *J* = 14.9 Hz, 1H), 3.75 (s, 0.16H), 3.45 (d, *J* = 17.8 Hz, 1H), 3.24 (d, *J* = 17.8 Hz, 1.34H), 2.35 (d, *J* = 15.8 Hz, 3.52H), 2.06 (d, *J* = 10.3 Hz, 3.52H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.50, 162.4, 161.2, 141.8, 138.2, 137.0, 131.6, 130.1, 129.9, 129.4, 128.8, 128.2, 128.0, 127.7, 123.6, 118.8, 116.4, 111.1, 110.8, 84.6, 54.4, 49.0, 46.7, 24.8, 21.3. HRMS (ESI+) calculated for $C_{28}H_{25}FNO_3$ ($M+H^+$) 442.1813; found 442.1820.

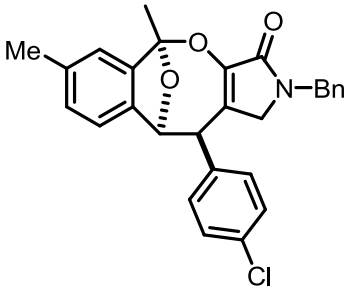


4ec

2-benzyl-11-(4-chlorophenyl)-7-methoxy-5-methyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7]oxocino[2,3-c]pyrrol-3(5H)-one

Colorless oil, 73 mg, yield 77%, *dr* = 14:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.18 (m, 6.19H), 7.17 – 7.07 (m, 2.14H), 7.01 – 6.83 (m, 3.15H), 6.72 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.10 (d, *J* = 8.3 Hz, 1H), 5.27 (d, *J* = 5.8 Hz, 1.07H), 4.81 (d, *J* = 15.0 Hz, 1H), 4.60 (d, *J* = 6.5 Hz, 0.07H), 4.52 (d, *J* = 5.8 Hz, 1H), 4.45 (s, 0.07H), 4.14 (d, *J* =

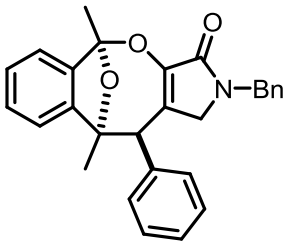
15.0 Hz, 1.06H), 3.82 (s, 3.22H), 3.35 (d, *J* = 17.7 Hz, 1H), 3.22 (d, *J* = 17.8 Hz, 1.14H), 2.05 (s, 3.21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 160.8, 142.0, 140.2, 136.9, 134.0, 133.8, 131.6, 130.8, 128.8, 128.19, 128.16, 127.7, 124.4, 118.1, 115.5, 111.3, 106.4, 84.5, 55.7, 54.5, 48.8, 46.7, 24.5. HRMS (ESI+) calculated for $C_{28}H_{25}ClNO_4$ ($M+H^+$) 474.1467; found 474.1468.



4fc

2-benzyl-11-(4-chlorophenyl)-5,7-dimethyl-1,2,10,11-tetrahydro-5,10-epoxybenzo [6,7]oxocino[2,3-c]pyrrol-3(5H)-one

White solid, 65 mg, yield 71%, m.p. 190-191 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31 – 7.16 (m, 7H), 7.12 – 7.07 (m, 2H), 6.96 (dd, *J* = 7.8, 1.4 Hz, 1H), 6.87 (d, *J* = 7.9 Hz, 2H), 6.08 (d, *J* = 7.7 Hz, 1H), 5.26 (d, *J* = 5.9 Hz, 1H), 4.79 (d, *J* = 15.0 Hz, 1H), 4.51 (d, *J* = 5.8 Hz, 1H), 4.09 (d, *J* = 15.0 Hz, 1H), 3.31 (d, *J* = 17.8 Hz, 1H), 3.19 (d, *J* = 17.8 Hz, 1H), 2.35 (s, 3H), 2.02 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 142.0, 139.3, 138.8, 136.9, 136.0, 134.0, 133.7, 131.7, 129.2, 128.80, 128.76, 128.2, 127.7, 123.2, 122.8, 118.0, 111.3, 84.6, 54.4, 48.8, 46.7, 24.6, 21.5. HRMS (ESI+) calculated for $C_{28}H_{25}ClNO_3$ ($M+H^+$) 458.1517; found 458.1523.

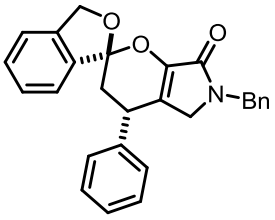


4ga

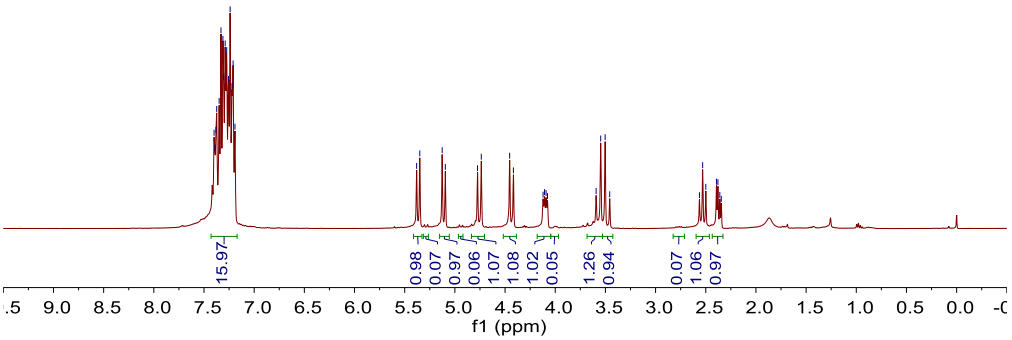
2-benzyl-5,10-dimethyl-11-phenyl-1,2,10,11-tetrahydro-5,10-epoxybenzo[6,7] oxocino[2,3-c]pyrrol-3(5H)-one

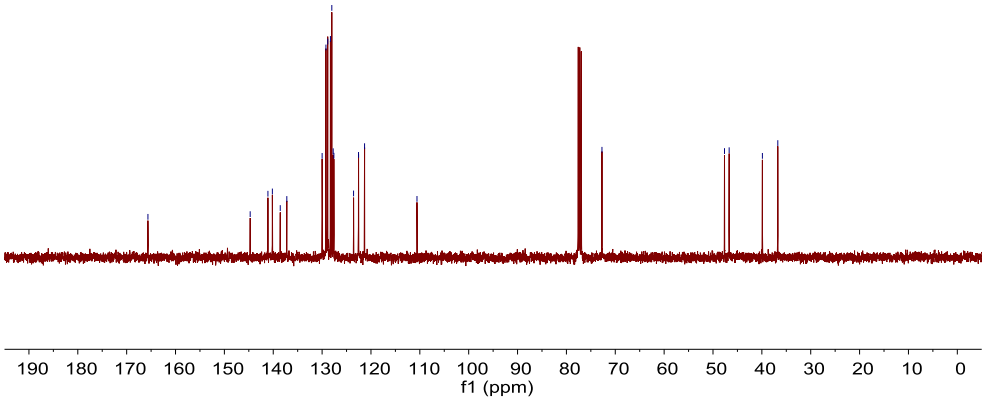
White solid, 73 mg, yield 86%, m.p. 183-184 ^{o}C, *dr* = 33:1, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (dt, *J* = 7.6, 0.9 Hz, 1H), 7.43 – 7.15 (m, 10H), 7.14 – 6.99 (m, 2H), 6.51 (dt, *J* = 7.9, 1.5 Hz, 1H), 6.31 (dt, *J* = 7.6, 0.9 Hz, 1H), 4.83 (d, *J* = 15.1 Hz, 1H), 4.26 (s, 1H), 4.04 (d, *J* = 15.1 Hz, 1H), 3.17 (dd, *J* = 11.0, 1.1 Hz, 2.07H), 2.06 (s, 3.09H), 1.53 (s, 3.09H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 142.4, 141.6, 138.9, 137.1, 136.5, 131.4, 130.5, 129.0, 128.71, 128.69, 128.5, 128.3, 128.1, 128.0, 127.7, 127.6, 123.5, 122.3, 119.8, 110.2, 89.4, 61.0, 49.5, 46.5, 25.4, 25.2. HRMS (ESI+) calculated for $C_{28}H_{26}NO_3$ ($M+H^+$) 424.1907; found 424.1915.

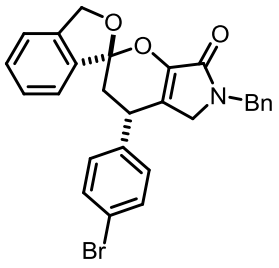
7. Spectra of [2+4] annulation products.



3aa






O
O
O
N
Bn
Br

3ab


7.47
7.45
7.41
7.39
7.37
7.36
7.34
7.32
7.32
7.31
7.30
7.30
7.29
7.28
7.27
7.27
7.27
7.26
7.25
7.22
7.21
7.20
7.13
7.11
5.34
5.12
5.09
4.72
3.58
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3.53
3.47
3.42
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2.45
2.37
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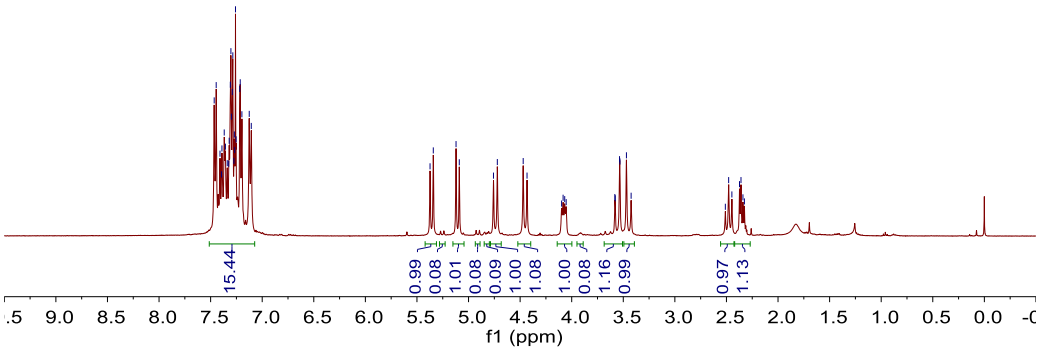
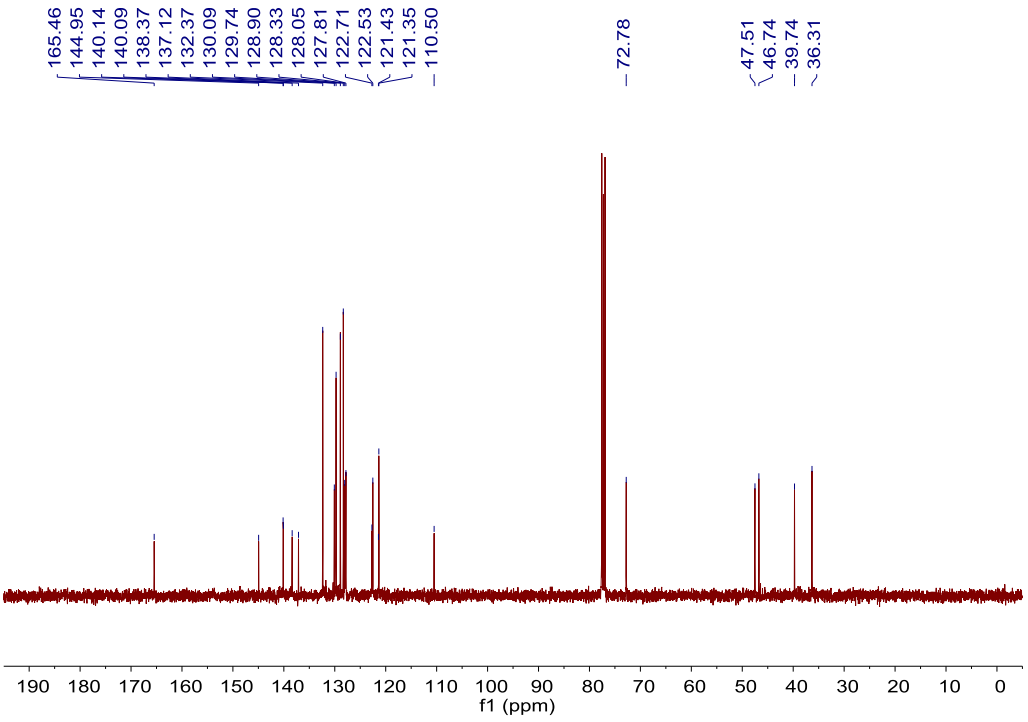
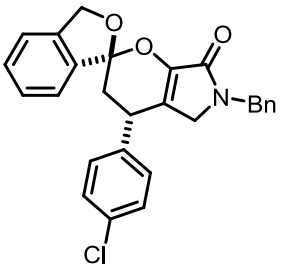
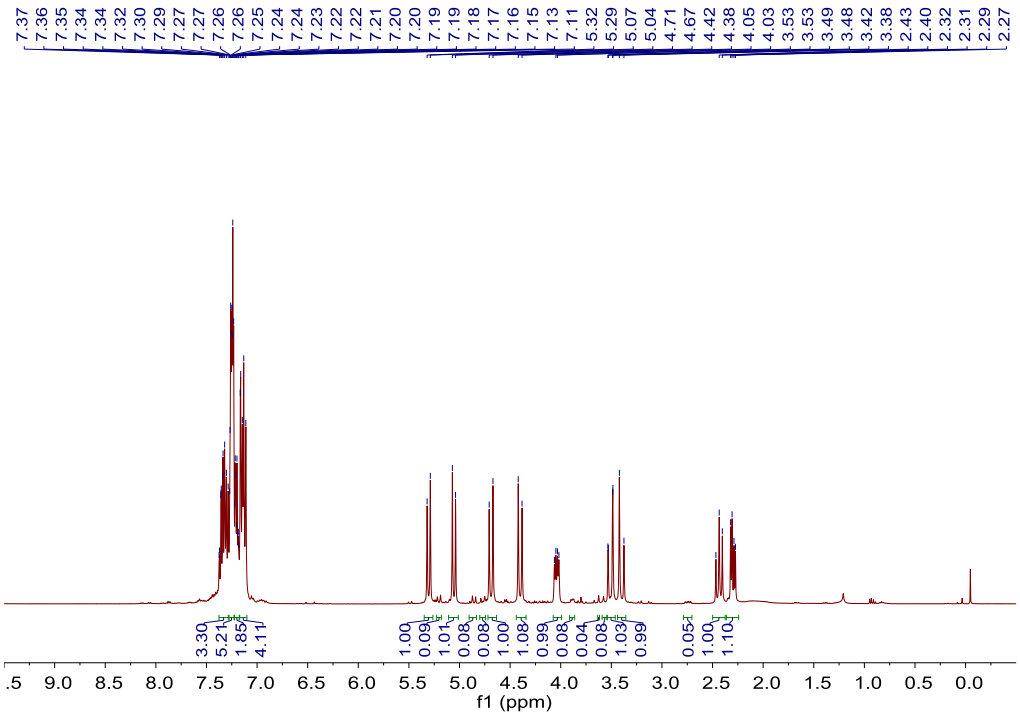
2.34
2.33

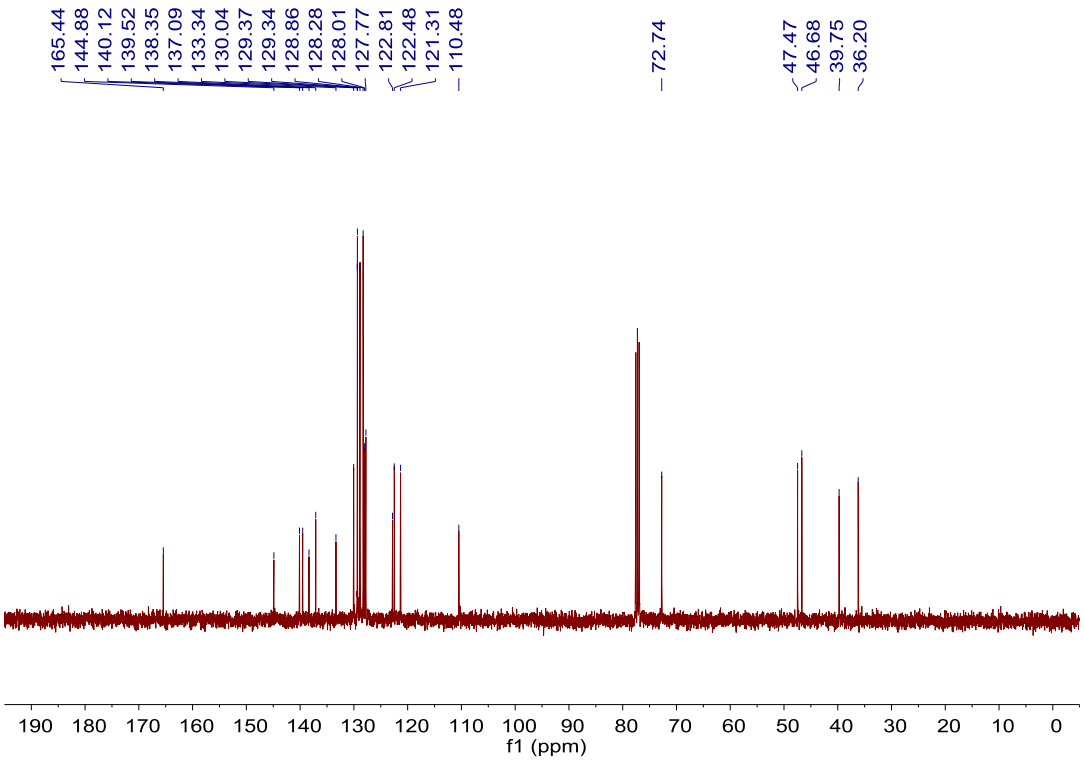

15.44
0.99
0.08
1.01
0.08
0.09
1.00
1.08
1.00
0.08
1.16
0.99
0.97
1.13
f1 (ppm)


165.46
144.95
140.14
140.09
138.37
137.12
132.37
130.09
129.74
128.90
128.33
128.05
127.81
122.71
122.53
121.43
121.35
110.50
72.78
47.51
46.74
39.74
36.31
f1 (ppm)


O
O
O
N
Bn
Cl

3ac


7.37 7.36 7.35 7.34 7.34 7.32 7.30 7.29 7.27 7.27 7.26 7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.19 7.18 7.17 7.16 7.15 7.13 7.11 5.32 5.29 5.07 5.04 4.71 4.67 4.42 4.38 4.05 4.03 3.53 3.53 3.49 3.48 3.42 3.38 2.43 2.40 2.32 2.31 2.29 2.27
3.30 5.21 1.85 4.11 1.00 0.09 1.01 0.08 0.08 1.00 1.08 0.99 0.08 0.04 0.08 1.03 0.99 0.05 1.00 1.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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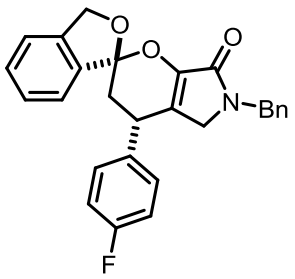
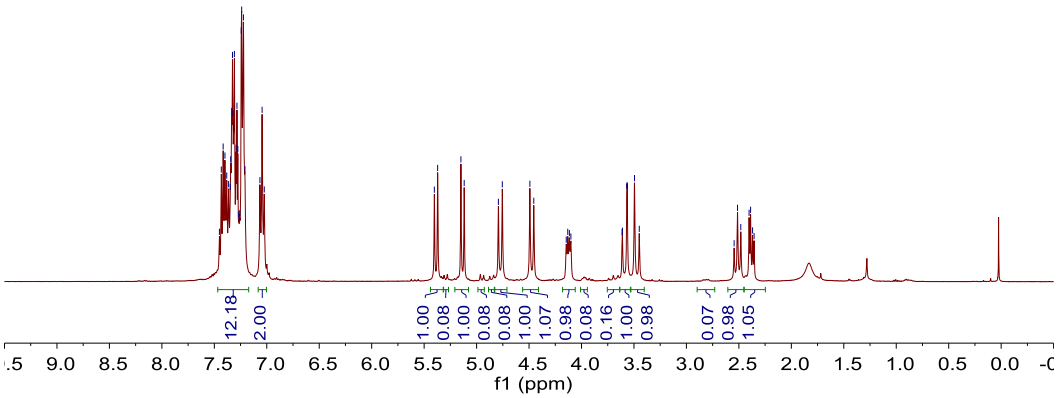
72.74
47.47 46.68 39.75 36.20
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O
O
N
Bn
F

3ad


7.43
7.42
7.40
7.38
7.36
7.35
7.34
7.33
7.33
7.32
7.32
7.31
7.29
7.28
7.28
7.24
7.24
7.22
7.21
7.07
7.04
7.02
5.40
5.37
5.15
5.12
4.75
4.49
4.45
3.61
3.61
3.57
3.56
3.49
3.45
2.55
2.51
2.48
2.40
2.39
2.37
2.35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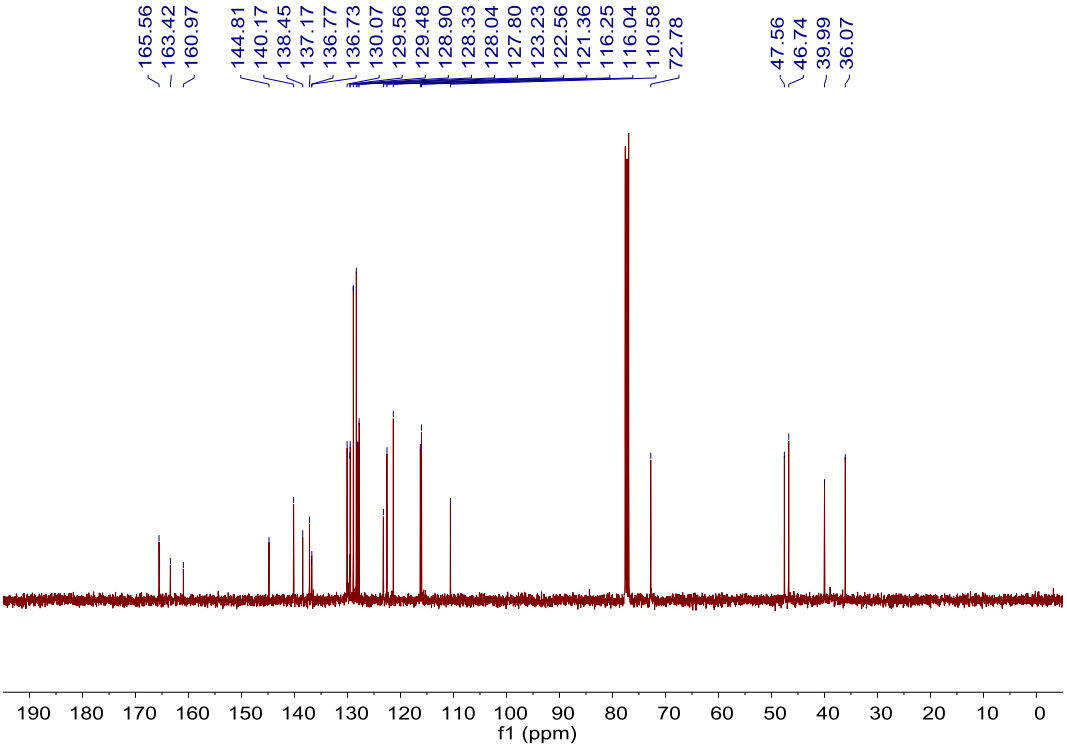
2.00
1.00
0.08
1.00
0.08
0.08
1.00
1.07
0.98
0.08
0.16
1.00
0.98
0.07
0.98
1.05
f1 (ppm)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137.17
136.77
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130.07
129.56
129.48
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128.33
128.04
127.80
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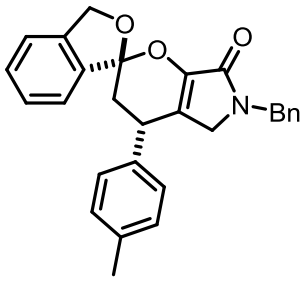
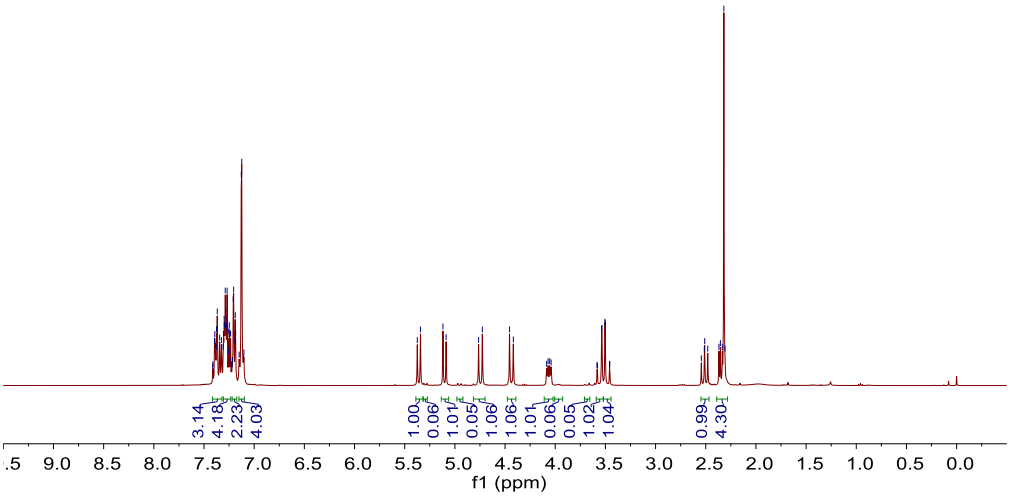
122.56
121.36
116.25
116.04
110.58
72.78
47.56
46.74
39.99
36.07
f1 (ppm)


O
O
O
N
Bn

3ae


7.40 7.39 7.38 7.38 7.37 7.37 7.34 7.33 7.31 7.31 7.30 7.30 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.25 7.24 7.21 7.20 7.19 7.19 7.18 7.15 7.13 7.12 7.11 7.10 5.37 5.34 5.12 5.09 4.76 4.73 4.46 4.42 3.54 3.53 3.50 3.50 2.51 2.51 2.48 2.37 2.35 2.34 2.32 2.31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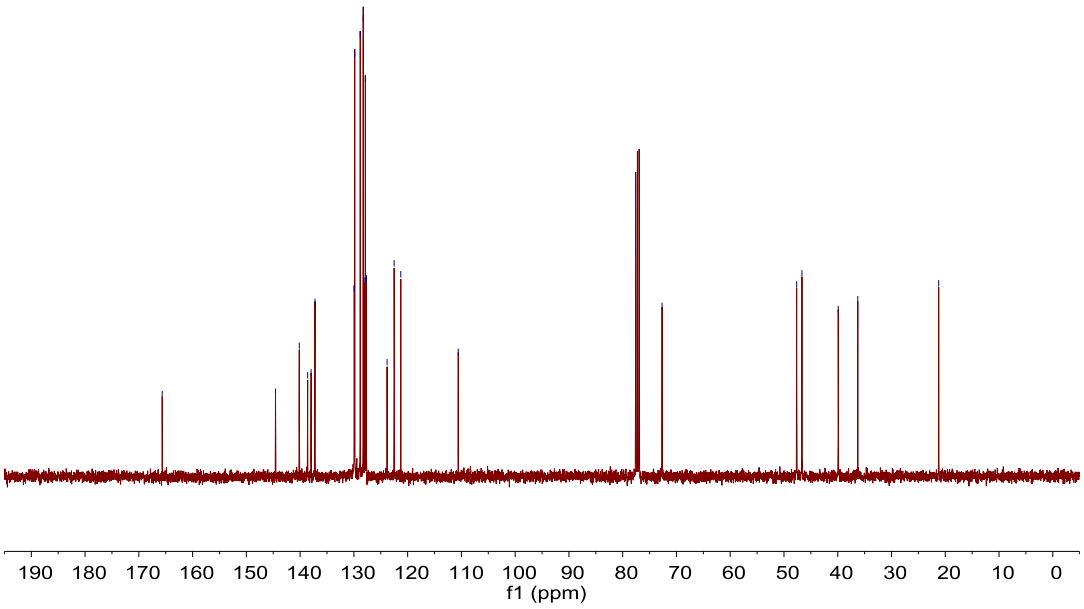
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


165.65 144.58 140.16 138.60 137.96 137.24 129.93 129.85 128.82 128.27 127.96 127.86 127.68 123.82 122.51 121.28 110.59 72.68 47.64 46.66 39.91 36.27 21.23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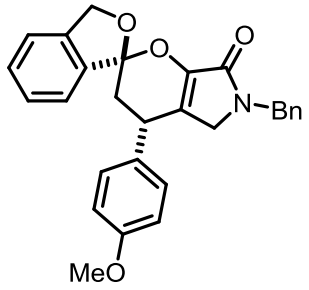
f1 (ppm)


O
O
O
N
Bn
MeO

3af


7.40
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7.39
7.38
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7.35
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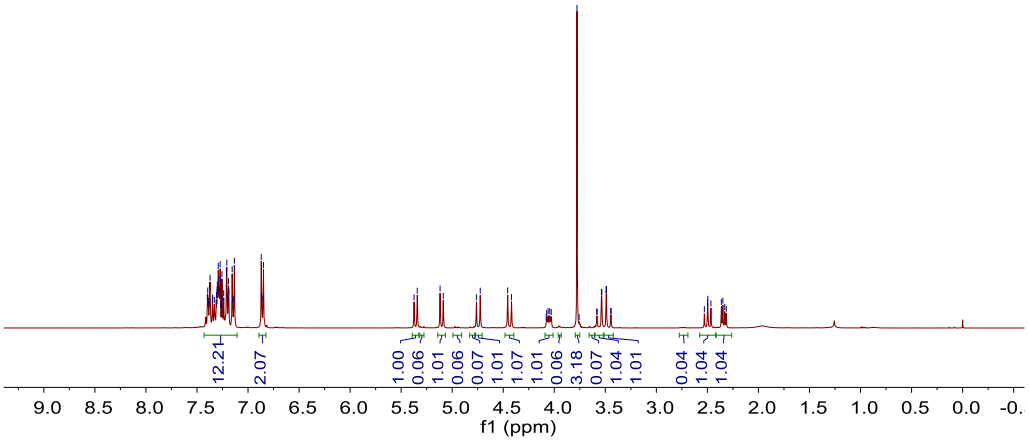
2.36
2.35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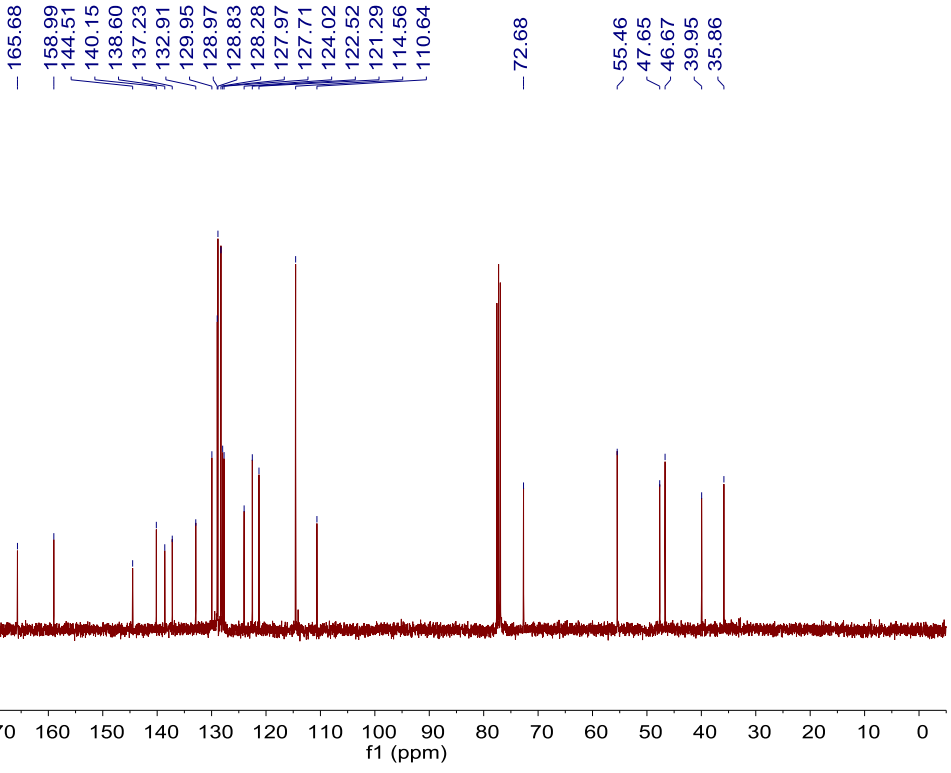
1.01
1.07
1.01
0.06
3.18
0.07
1.04
1.01
0.04
1.04
1.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.83
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127.71
124.02
122.52
121.29
114.56
110.64
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47.65
46.67
39.95
35.86
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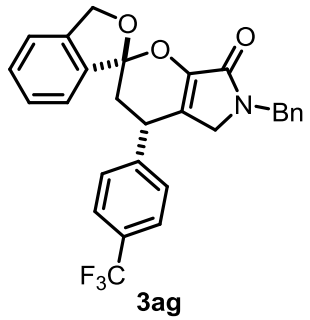
f1 (ppm)


O
O
O
N–Bn
F3C

3ag


7.53
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7.34
7.33
7.32
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7.31
7.30
7.29
7.28
7.26
7.25
7.24
7.24
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7.22
7.22
7.22
7.21
7.20
7.19
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7.18
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7.12
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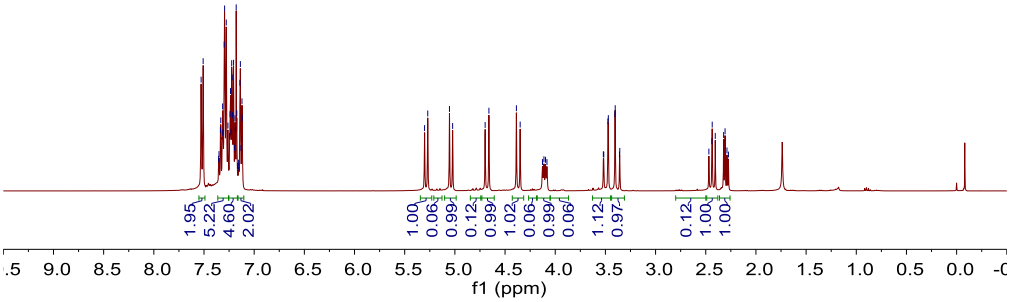
2.29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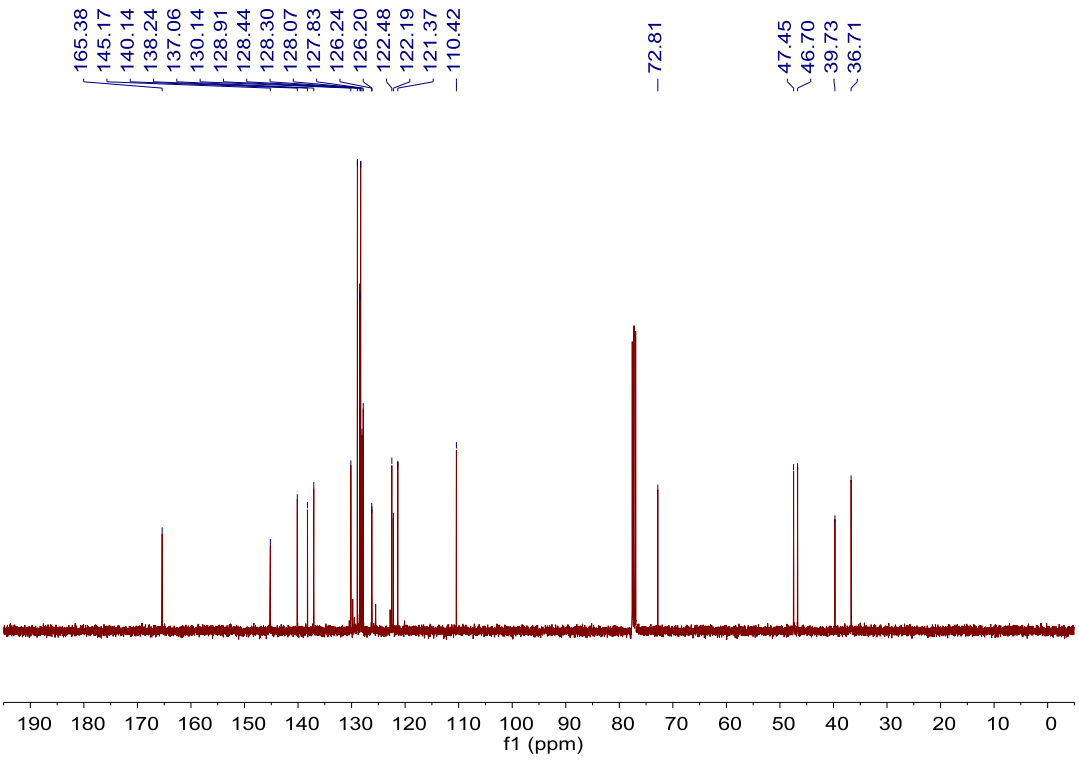
4.60
2.02
1.00
0.06
0.99
0.12
0.99
1.02
0.06
0.99
0.06
1.12
0.97
0.12
1.00
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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122.48
122.19
121.37
110.42
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47.45
46.70
39.73
36.71
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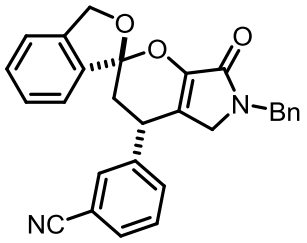
f1 (ppm)


O
O
O
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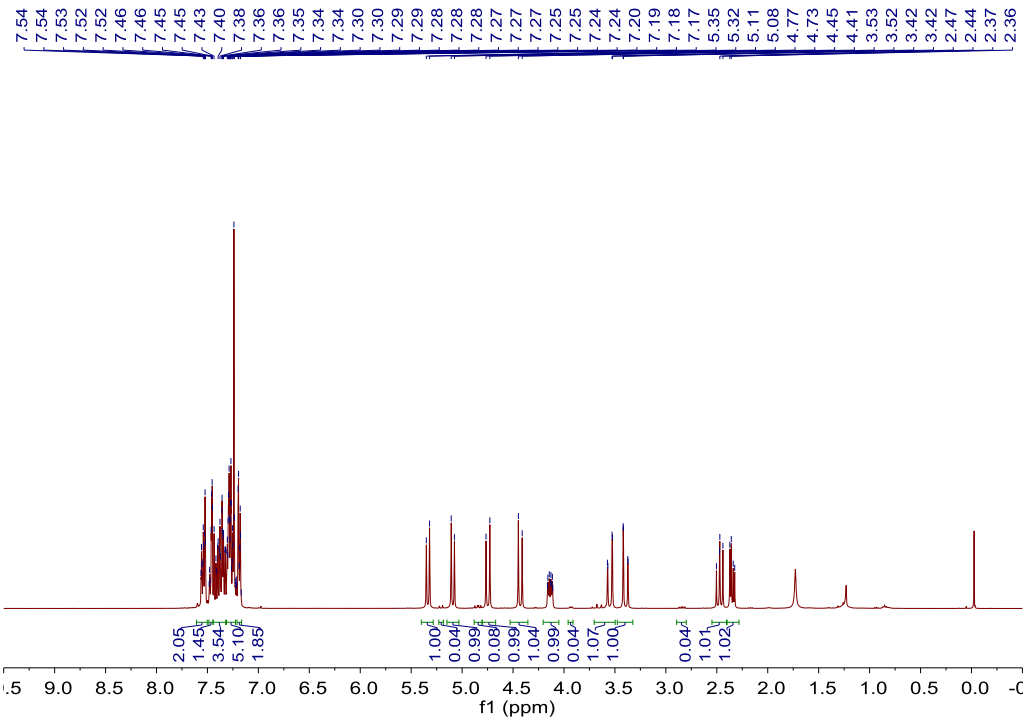
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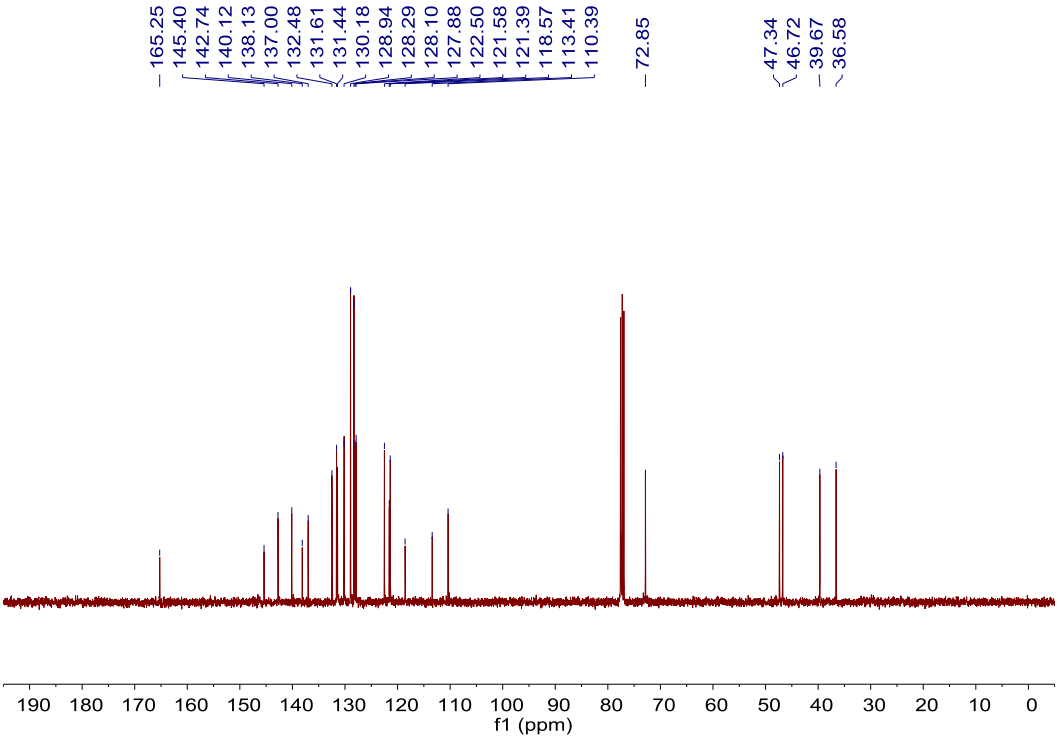
f1 (ppm)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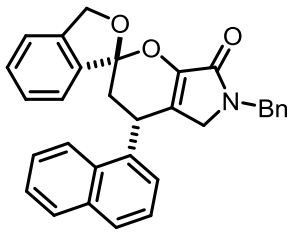
f1 (ppm)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Bn

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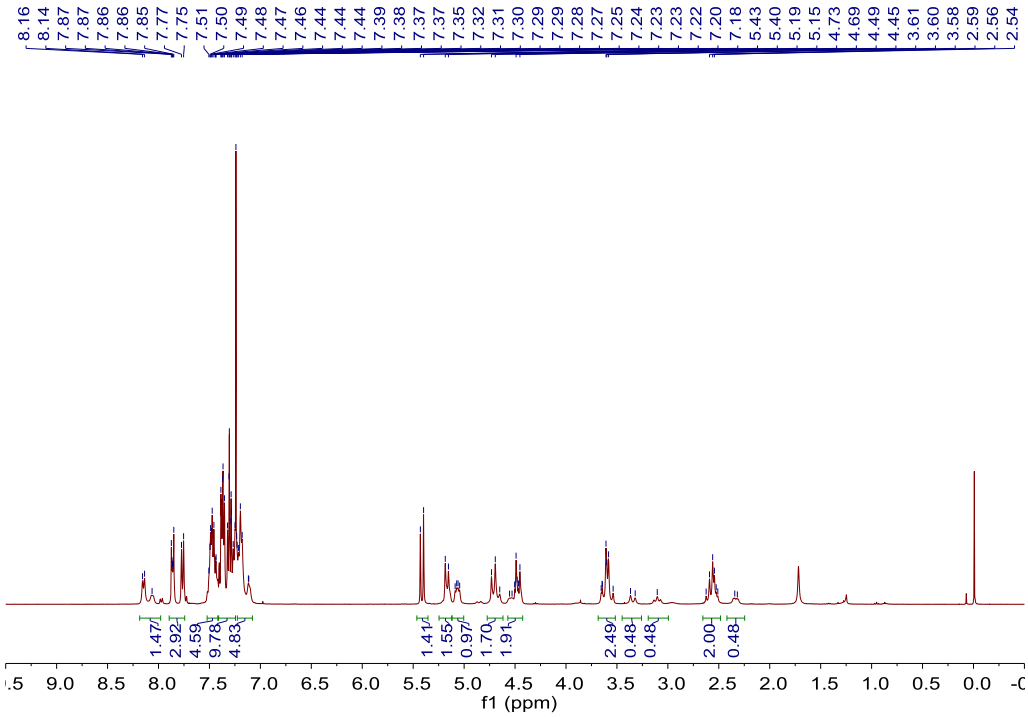
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3.60
3.58
2.59
2.56
2.54
1.47
2.92
4.59
9.78
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1.70
1.91
2.49
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0.48
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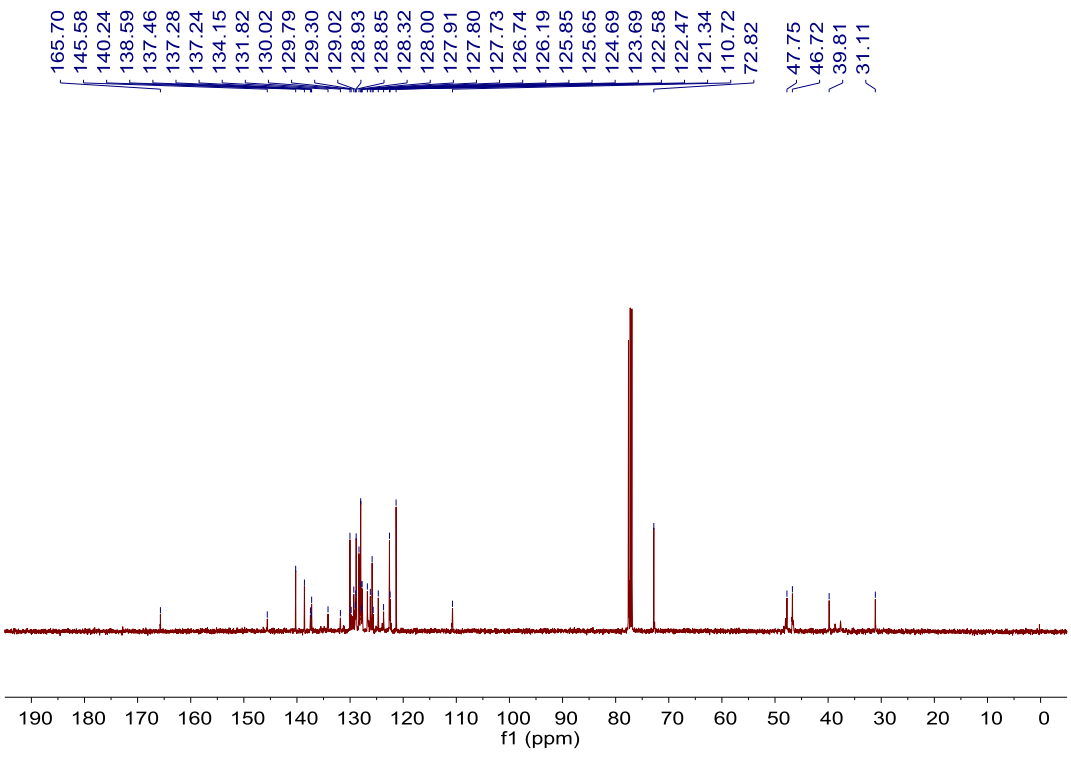
f1 (ppm)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130.02
129.79
129.30
129.02
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128.85
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127.73
126.74
126.19
125.85
125.65
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72.82
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46.72
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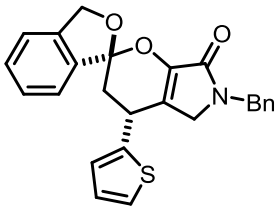
31.11
f1 (ppm)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Bn
S

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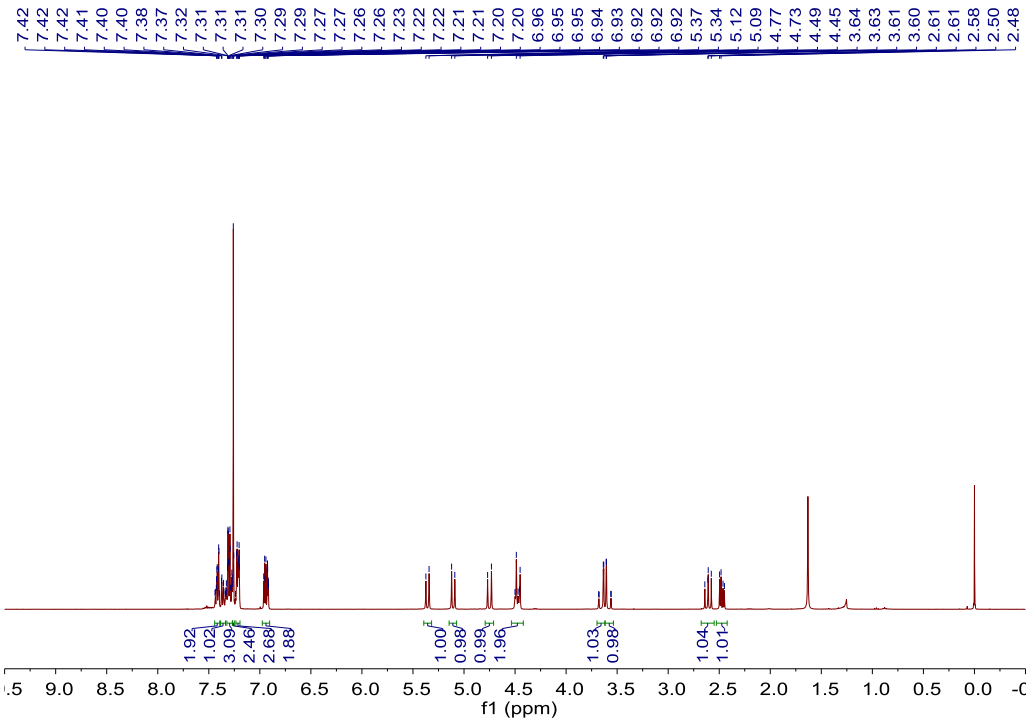
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1.92 1.02 3.09 2.46 2.68 1.88 1.00 0.98 0.99 1.96 1.03 0.98 1.04 1.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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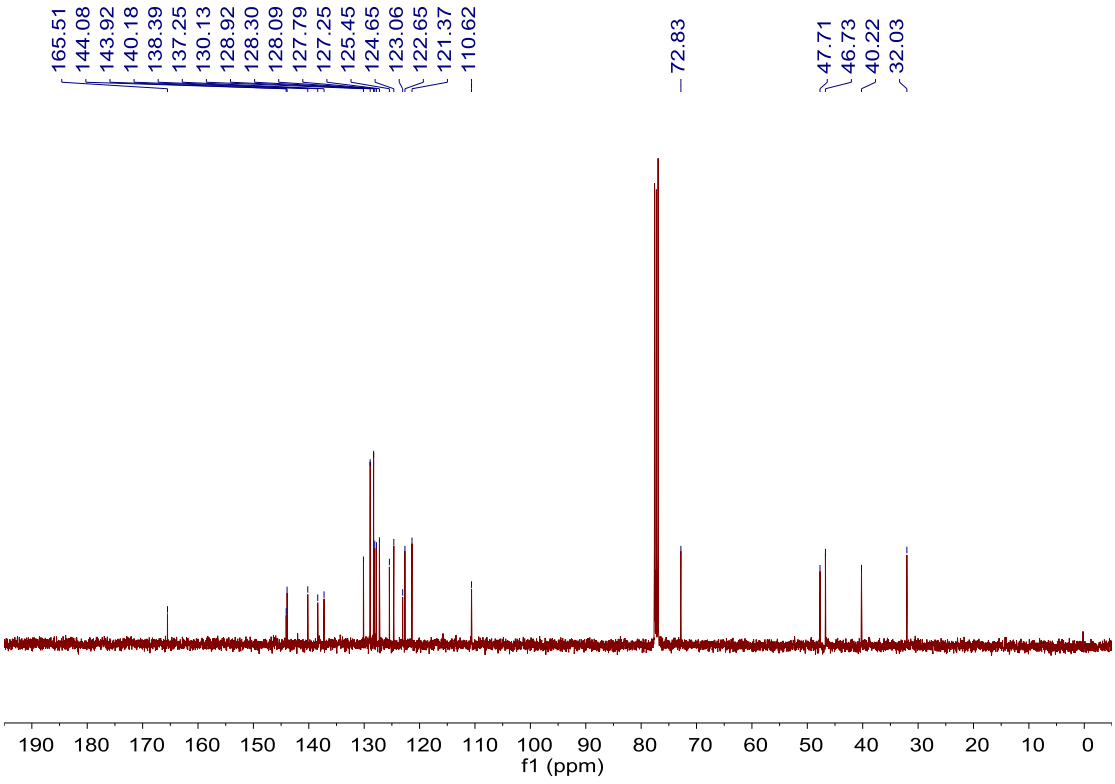
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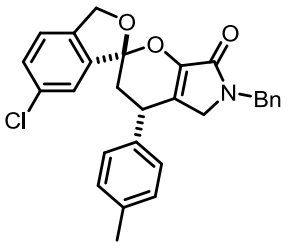
72.83
47.71 46.73 40.22 32.03
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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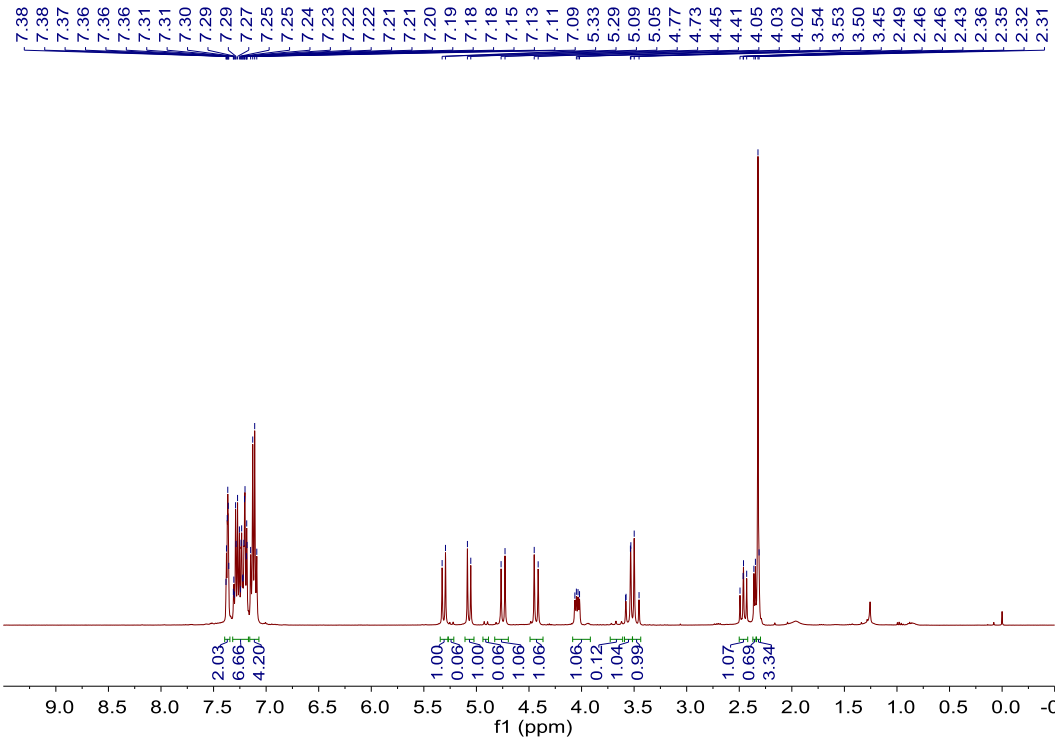
3be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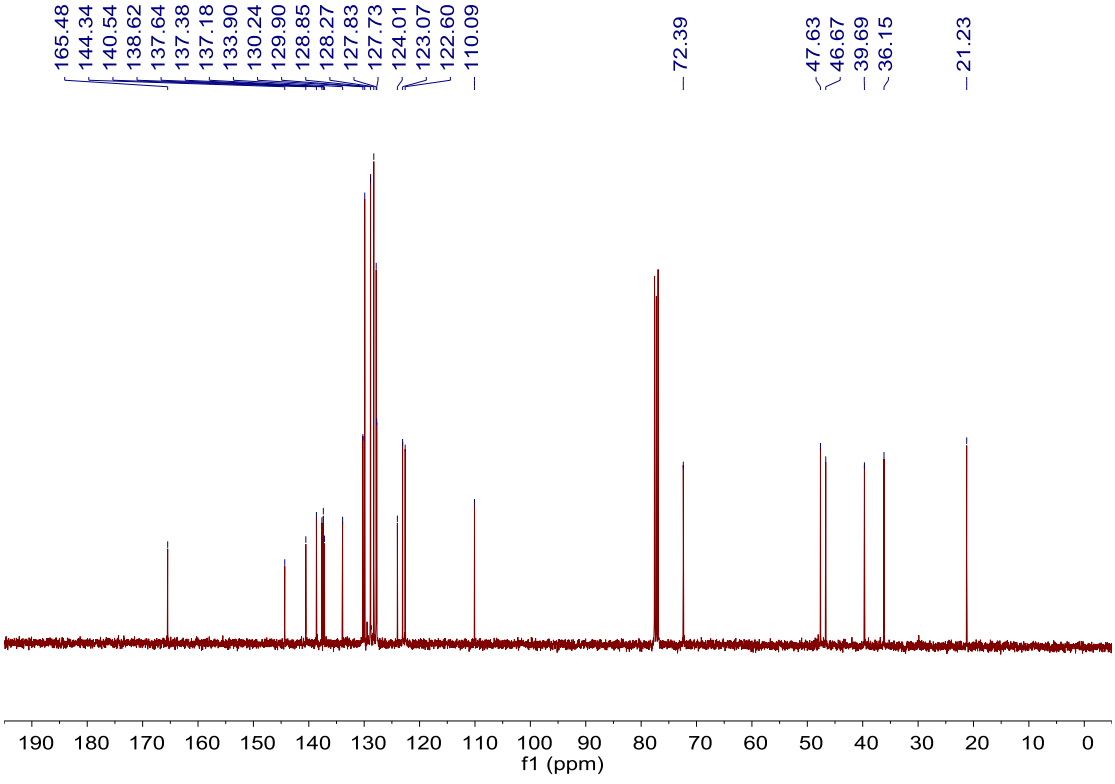
2.03 6.66 4.20 1.00 0.06 1.00 0.06 1.06 1.06 1.06 0.12 1.04 0.99 1.07 0.69 3.34
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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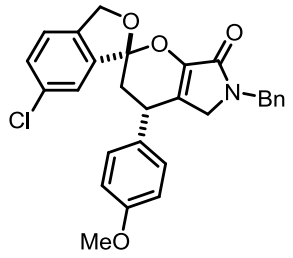
f1 (ppm)


O
O
O
N
Bn
Cl
MeO

3bf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7.29
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4.01
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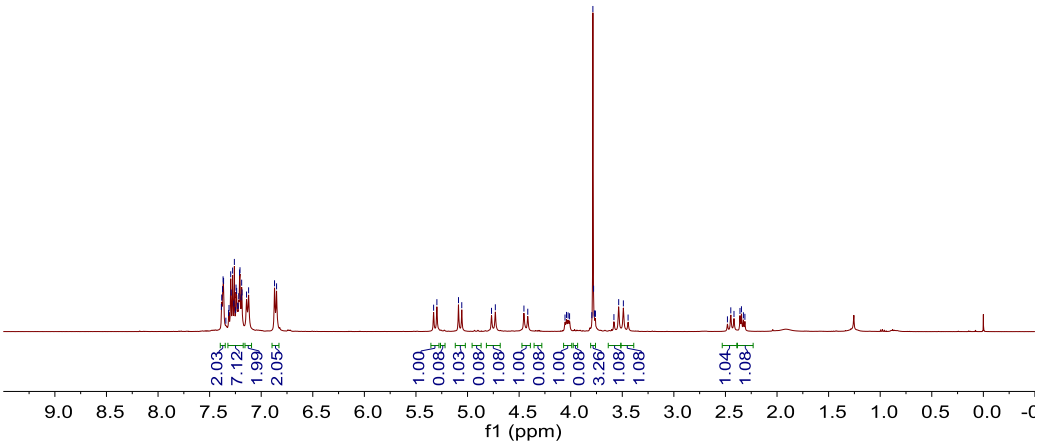
2.33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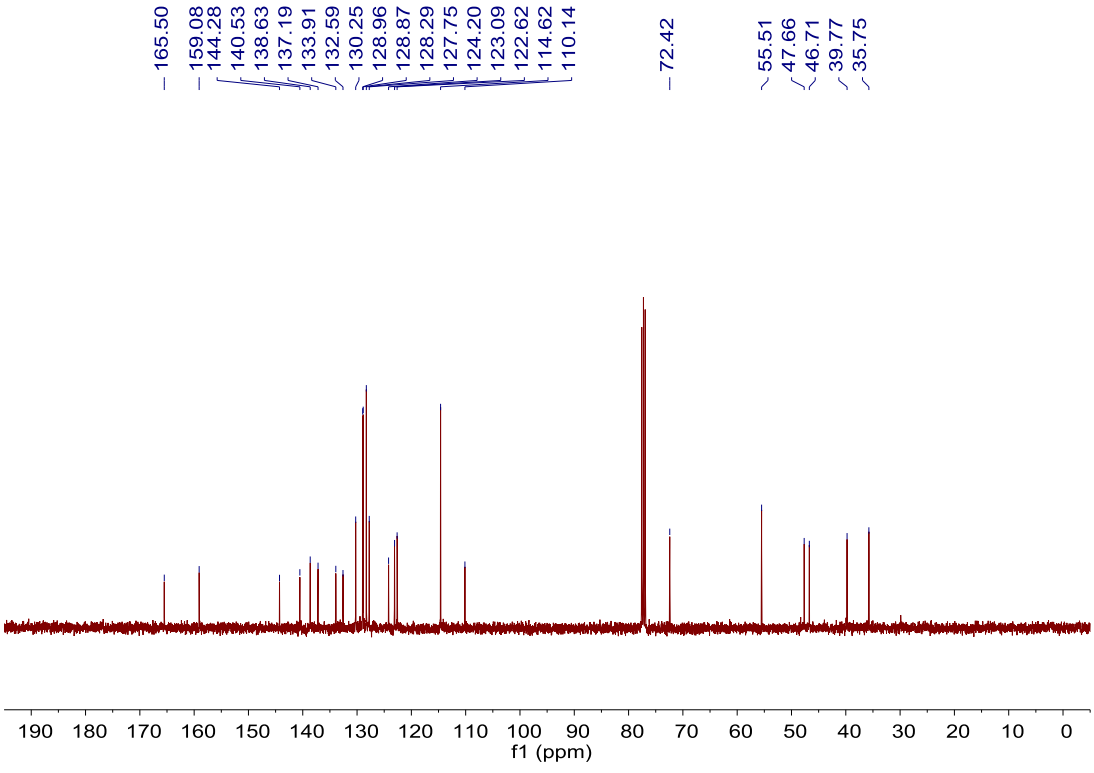
0.08
1.03
0.08
1.08
1.00
0.08
1.00
0.08
3.26
1.08
1.08
1.04
1.08
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.75
124.20
123.09
122.62
114.62
110.14
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47.66
46.71
39.77
35.75
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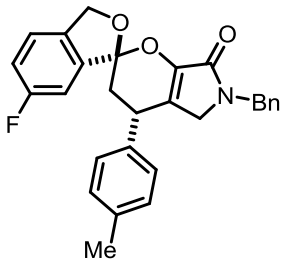
f1 (ppm)


O
O
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Bn
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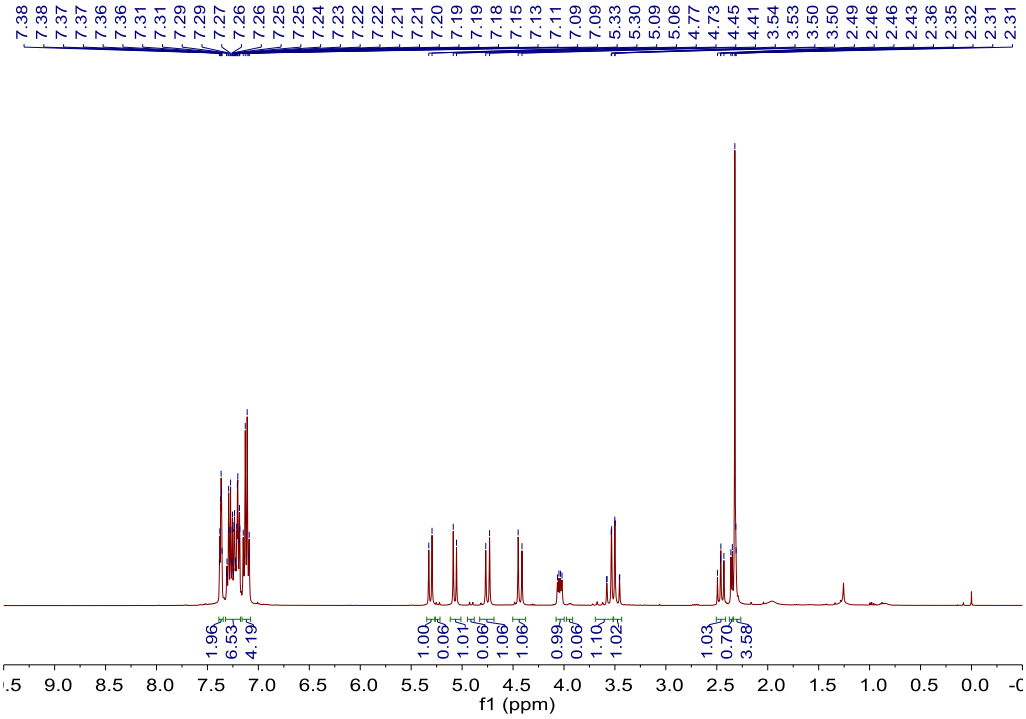
3ce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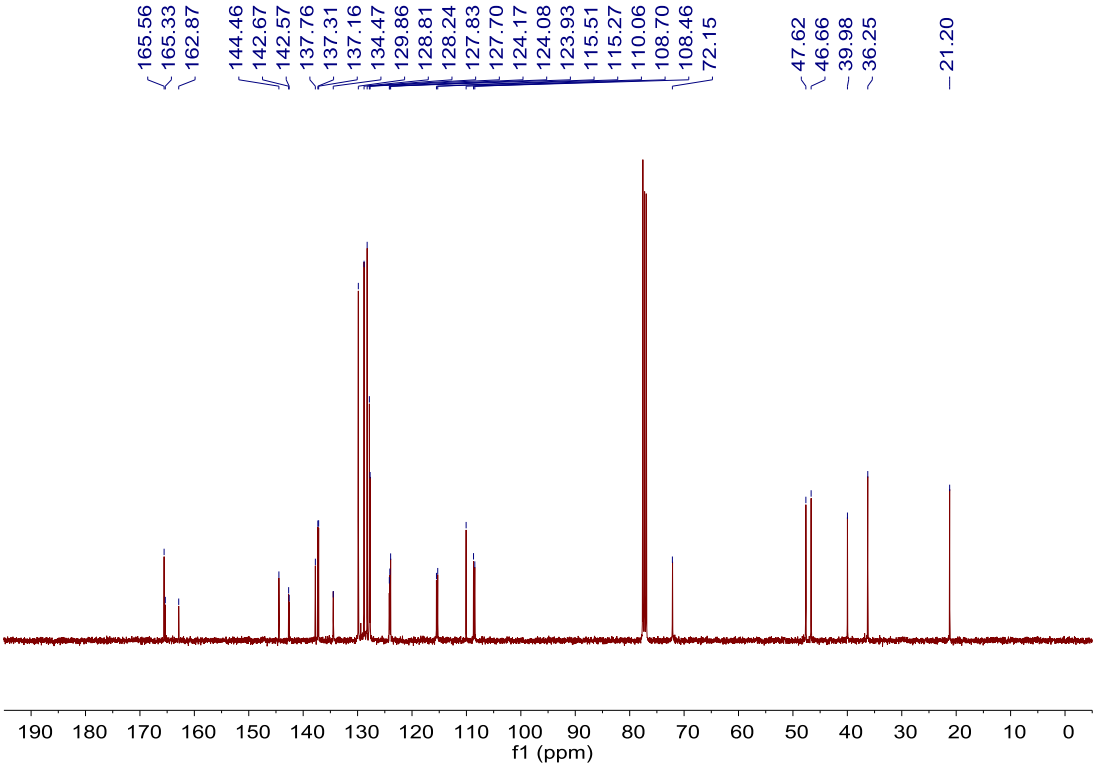
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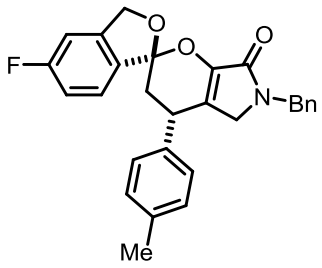
f1 (ppm)


F
O
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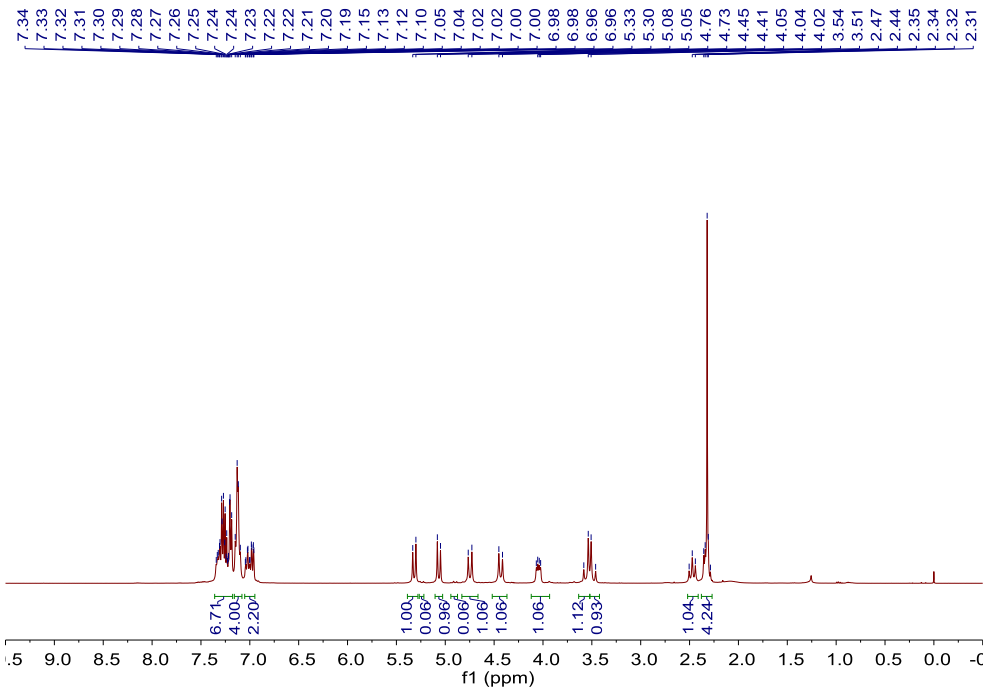
N
Bn
Me

3de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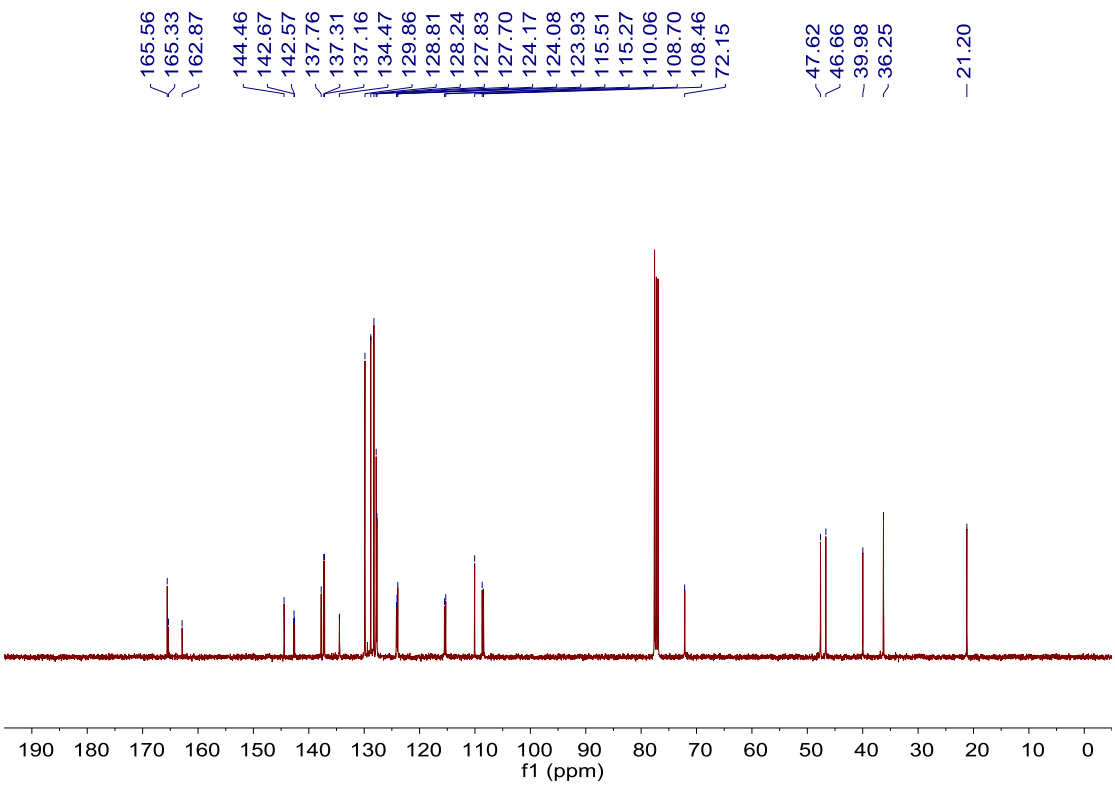
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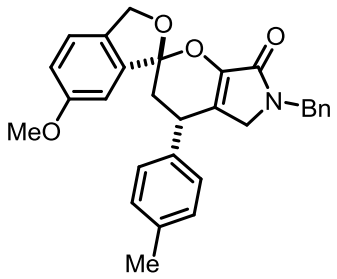
f1 (ppm)


O
O
O
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Bn
MeO
Me

3ee


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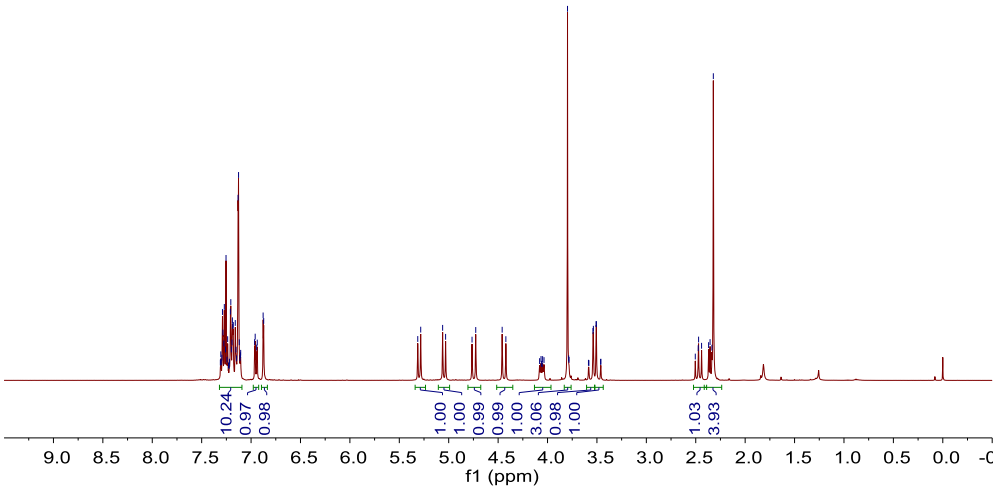
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1.00
3.06
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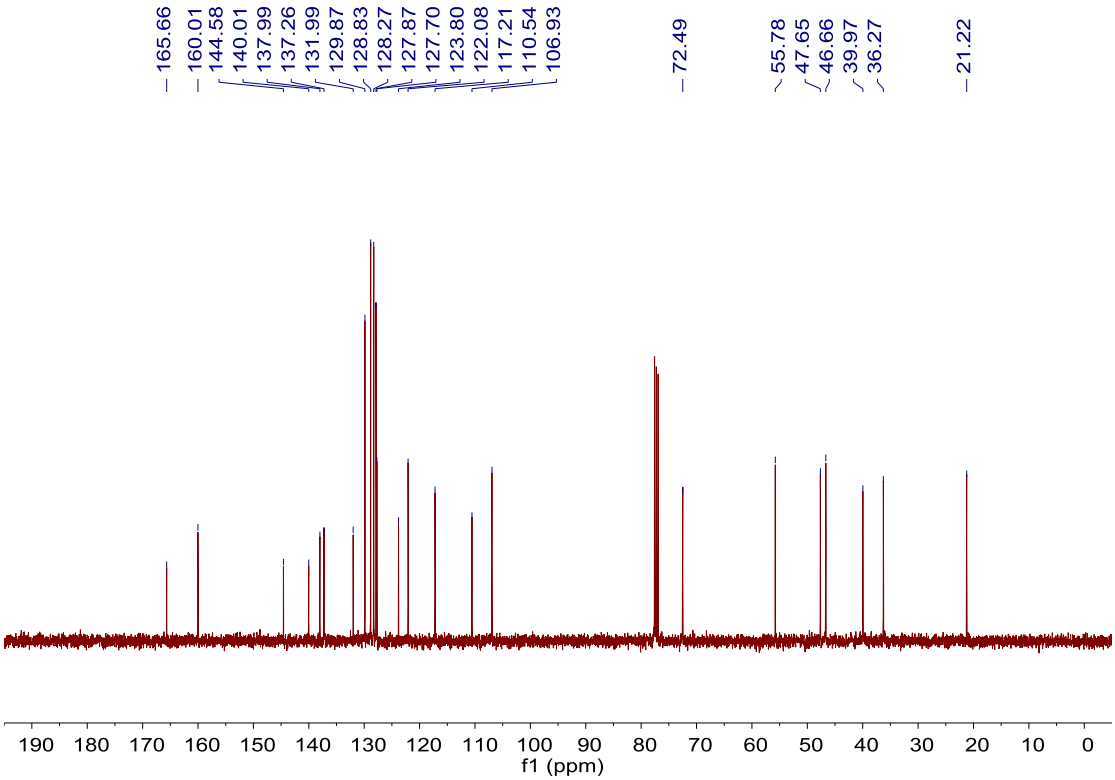
1.00
1.03
3.93
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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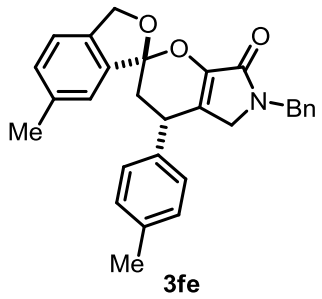
127.87
127.70
123.80
122.08
117.21
110.54
106.93
72.49
55.78
47.65
46.66
39.97
36.27
21.22
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O
O
N
Bn
Me
Me

3fe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5.30
5.08
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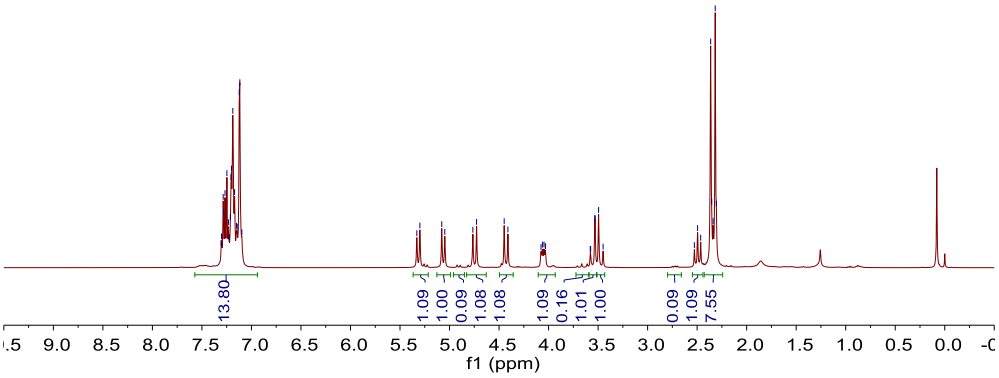
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0.16
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1.00
0.09
1.09
7.55
f1 (ppm)


165.68
144.57
138.83
138.02
137.83
137.26
137.23
137.20
130.83
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128.81
128.26
127.85
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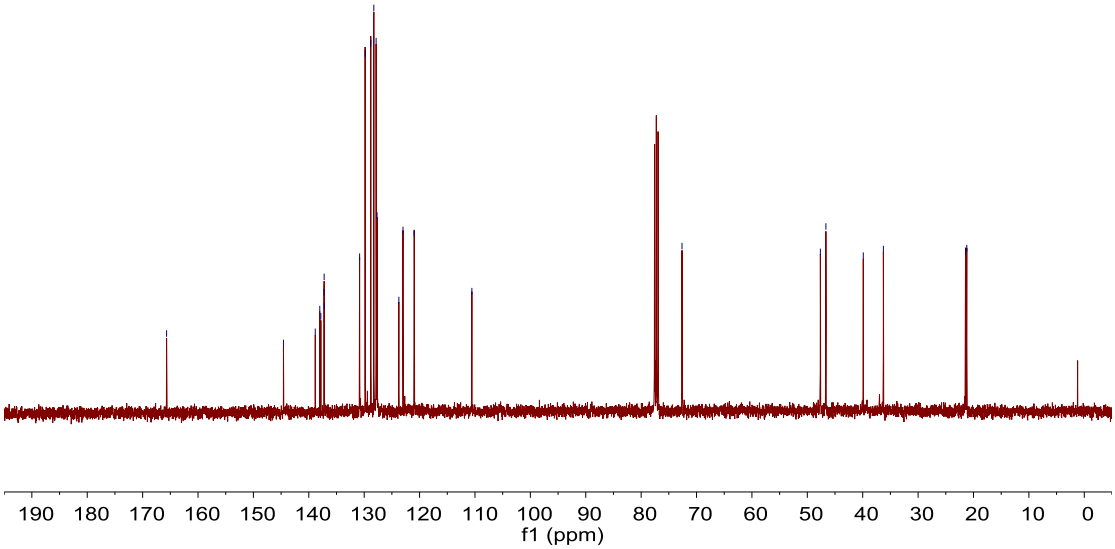
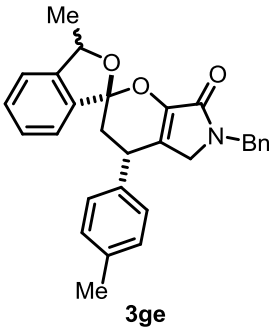
21.44
21.21


f1 (ppm)


Me
O
O
O
N
Bn
Me

3ge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7.31
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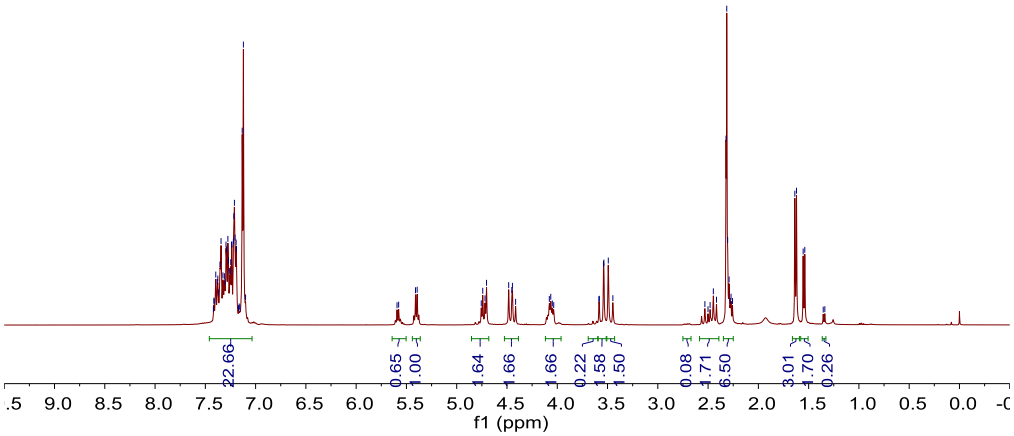
1.54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3.01
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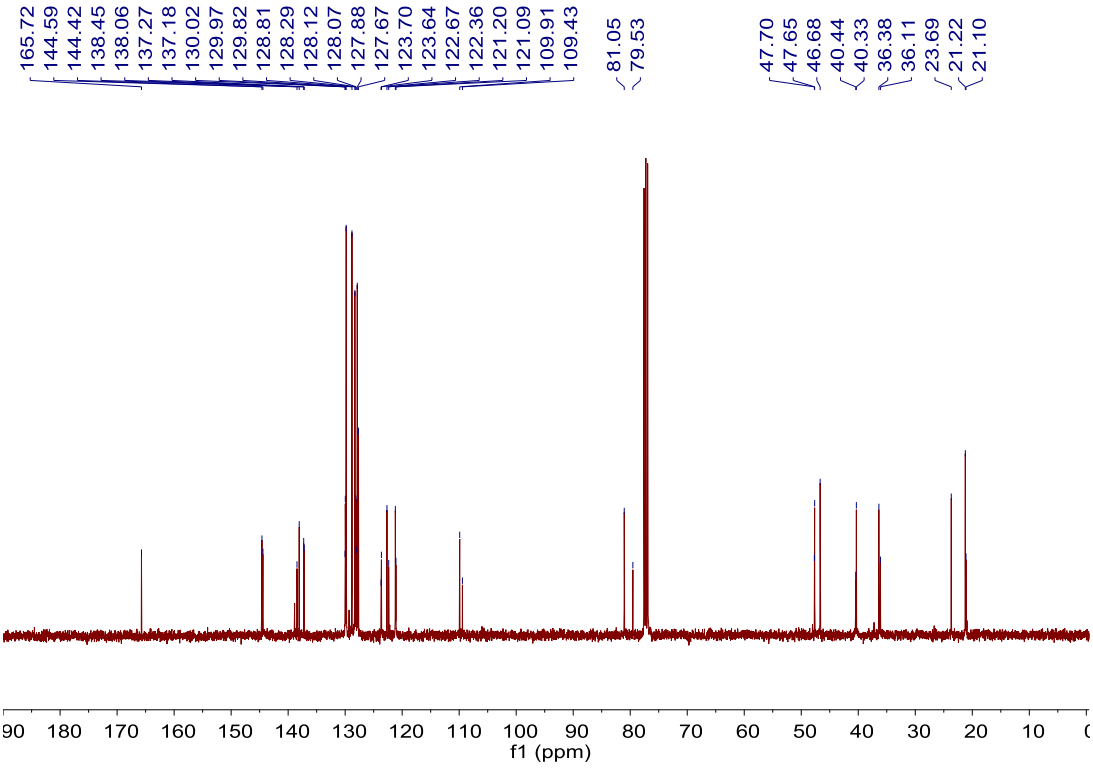
0.26
f1 (ppm)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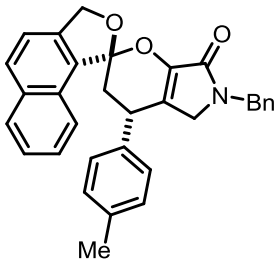
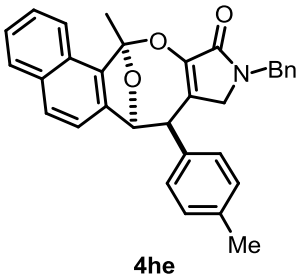
122.36
121.20
121.09
109.91
109.43
81.05
79.53
47.70
47.65
46.68
40.44
40.33
36.38
36.11
23.69
21.22
21.10
f1 (ppm)

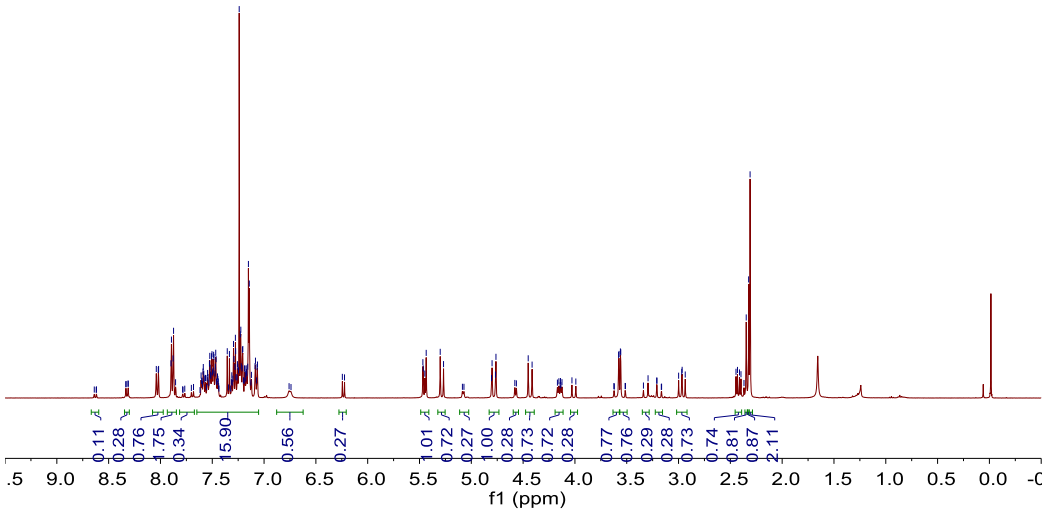

O
O
O
N–Bn
Me

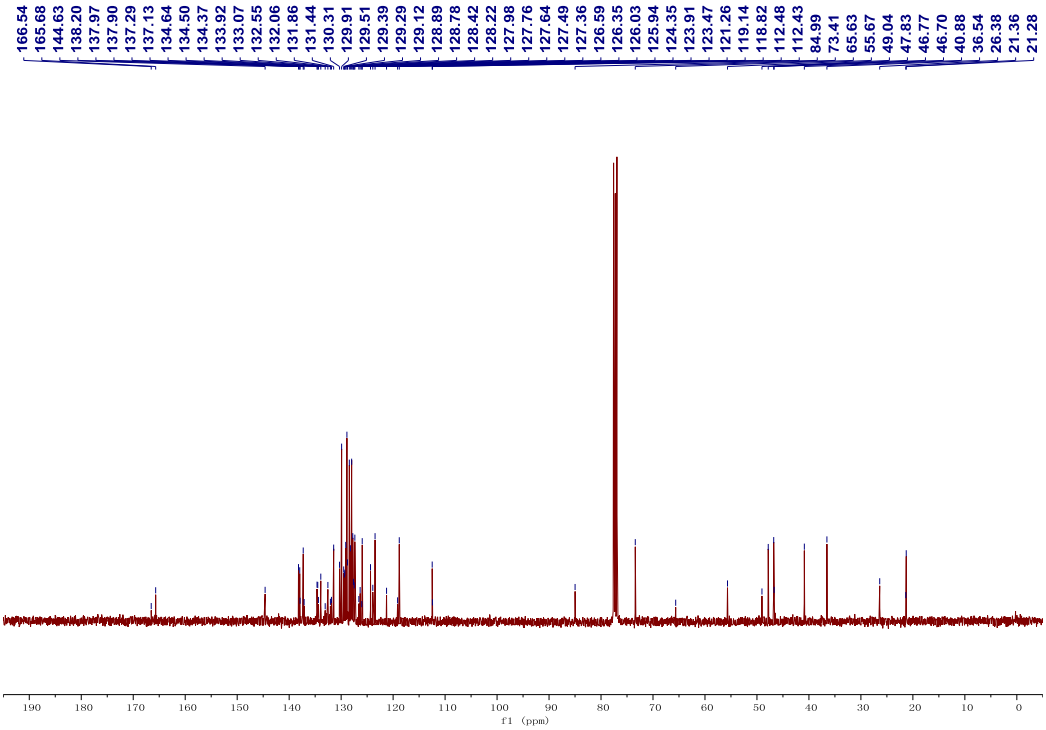
3he


O
O
O
N–Bn
Me

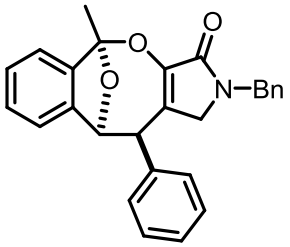
4he


7.90
7.89
7.88
7.87
7.59
7.53
7.52
7.51
7.50
7.49
7.48
7.47
7.47
7.46
7.36
7.33
7.30
7.29
7.28
7.28
7.27
7.26
7.25
7.24
7.23
7.22
7.22
7.21
7.21
7.16
7.15
7.14
7.09
7.08
7.07
7.06
5.47
5.44
5.30
5.27
4.80
4.76
4.45
4.41
3.58
3.57
3.56
3.56
2.35
2.32
2.31

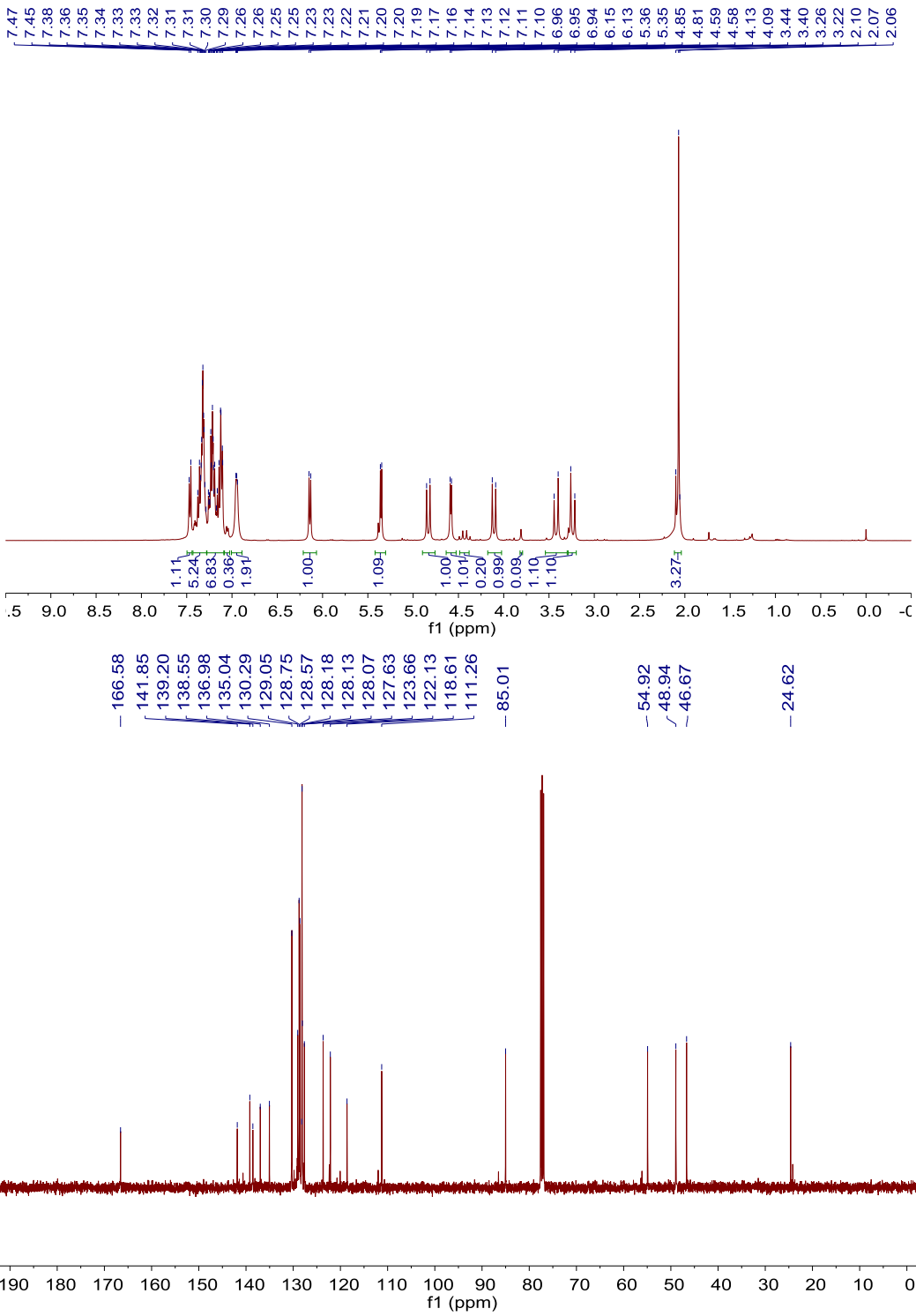

0.11
0.28
0.76
1.75
0.34
15.90
0.56
0.27
1.01
0.72
0.27
1.00
0.28
0.73
0.72
0.28
0.77
0.76
0.29
0.28
0.73
0.74
0.81
0.87
2.11
.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

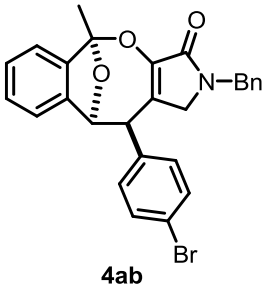

166.54
165.68
144.63
138.20
137.97
137.90
137.29
137.13
134.64
134.50
134.37
133.92
133.07
132.55
132.06
131.86
131.44
130.31
129.91
129.51
129.39
129.29
129.12
128.89
128.78
128.42
128.22
127.98
127.76
127.64
127.49
127.36
126.59
126.35
126.03
125.94
124.35
123.91
123.47
121.26
119.14
118.82
112.48
112.43
84.99
73.41
65.63
55.67
49.04
47.83
46.77
46.70
40.88
36.54
26.38
21.36
21.28
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

8. Spectra of [4+4] annulation products.

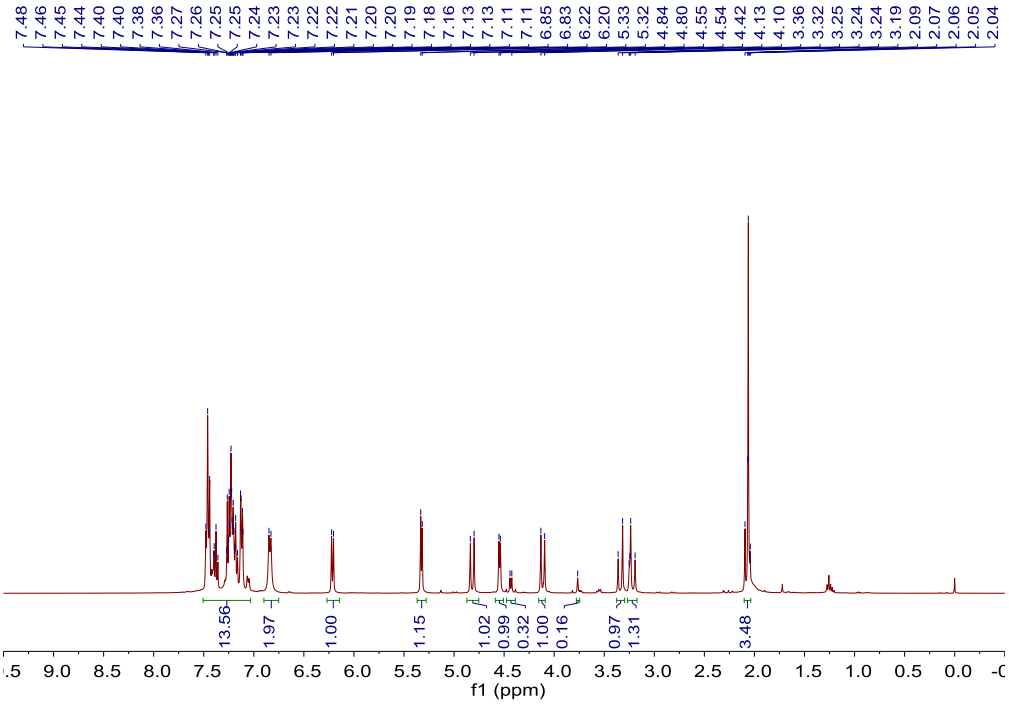


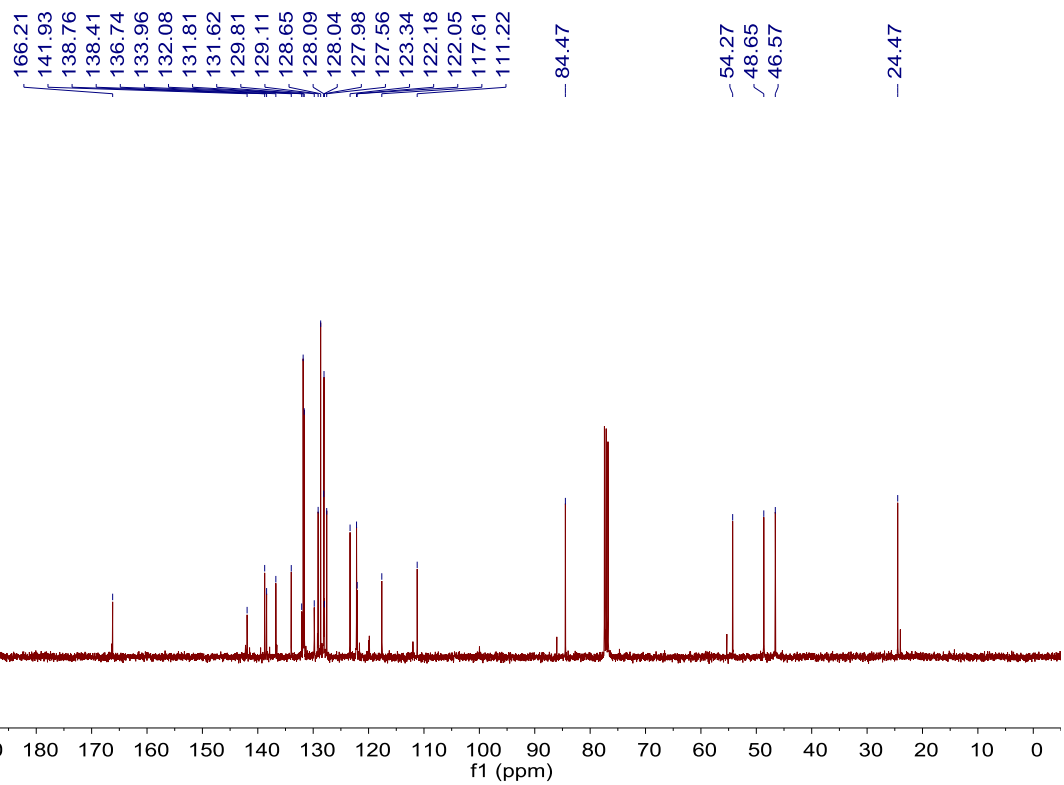
4aa

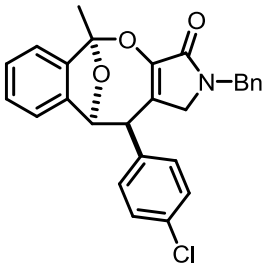



O
O
O
N–Bn
Br

4ab

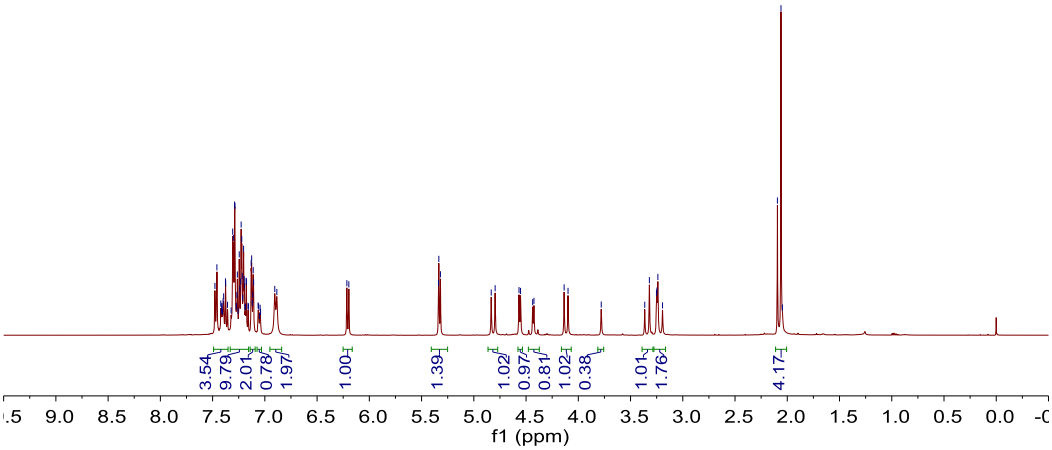

7.48 7.46 7.45 7.44 7.40 7.40 7.38 7.36 7.27 7.26 7.25 7.25 7.24 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.18 7.16 7.13 7.13 7.11 7.11 6.85 6.83 6.22 6.20 5.33 5.32 4.84 4.80 4.55 4.54 4.42 4.13 4.10 3.36 3.32 3.25 3.24 3.24 3.19 2.09 2.07 2.06 2.05 2.04
13.56 1.97 1.00 1.15 1.02 0.99 0.32 1.00 0.16 0.97 1.31 3.48
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

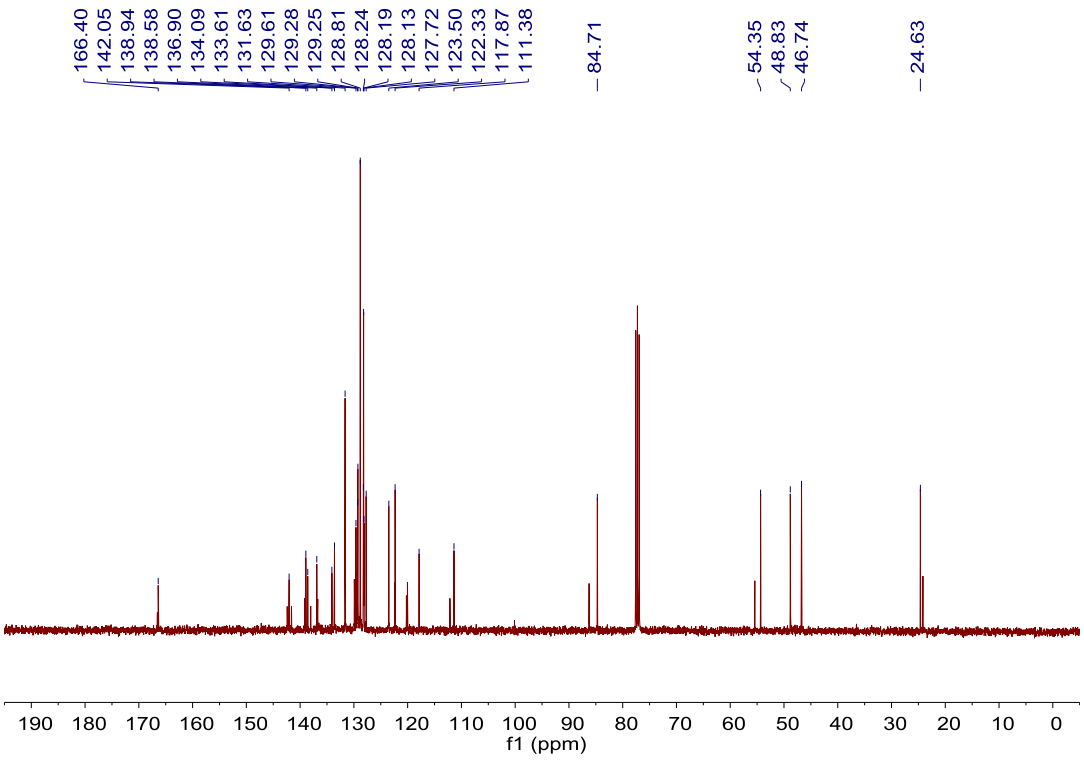

166.21 141.93 138.76 138.41 136.74 133.96 132.08 131.81 131.62 129.81 129.11 128.65 128.09 128.04 127.98 127.56 123.34 122.18 122.05 117.61 111.22
84.47
54.27 48.65 46.57
24.47
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

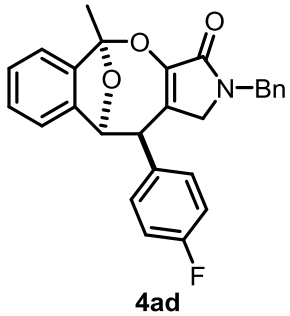

O
O
O
N–Bn
Cl

4ac


7.48
7.46
7.40
7.39
7.38
7.37
7.31
7.30
7.29
7.29
7.27
7.26
7.25
7.24
7.23
7.23
7.22
7.22
7.21
7.20
7.20
7.20
7.18
7.18
7.13
7.13
7.12
7.12
7.11
7.11
6.91
6.89
6.22
6.20
5.34
5.33
5.32
4.83
4.80
4.57
4.55
4.44
4.42
4.14
4.10
3.32
3.25
3.25
3.24
2.09
2.06

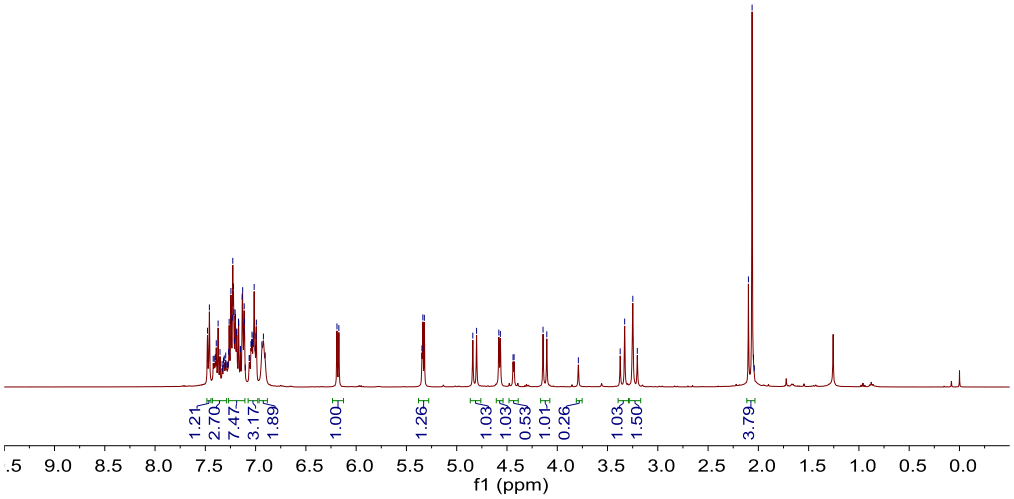

3.54
9.79
2.01
0.78
1.97
1.00
1.39
1.02
0.97
0.81
1.02
0.38
1.01
1.76
4.17
f1 (ppm)

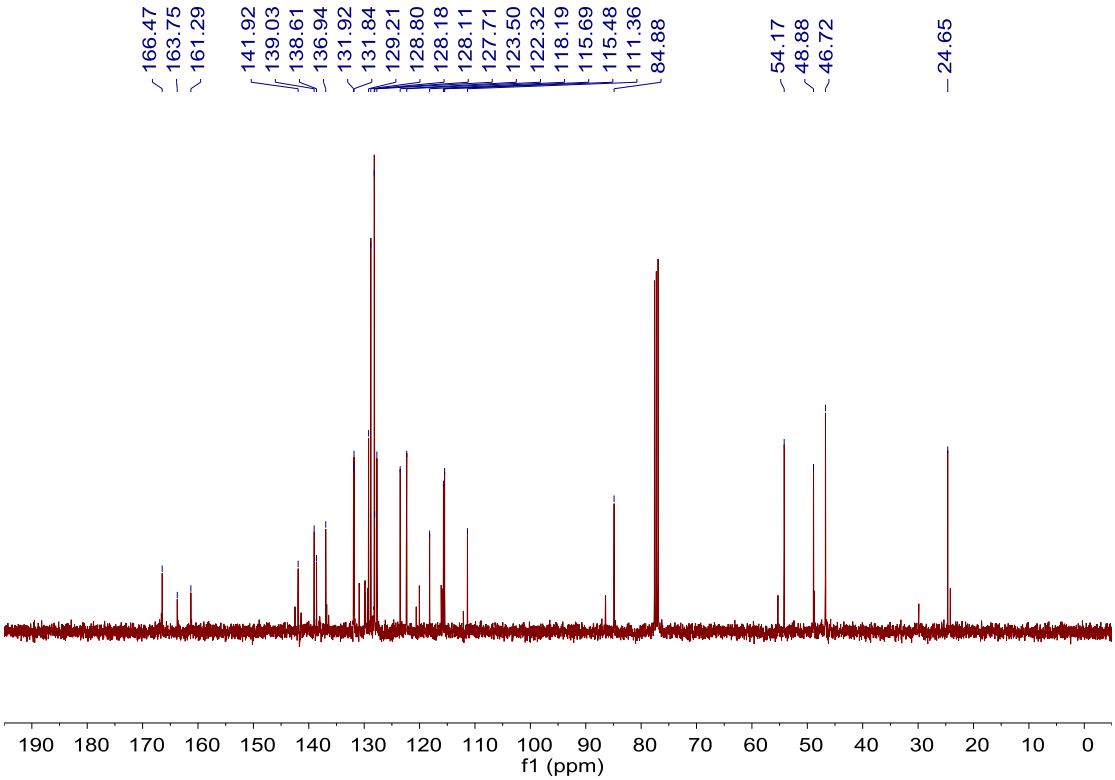

166.40
142.05
138.94
138.58
136.90
134.09
133.61
131.63
129.61
129.28
129.25
128.81
128.24
128.19
128.13
127.72
123.50
122.33
117.87
111.38
84.71
54.35
48.83
46.74
24.63
f1 (ppm)

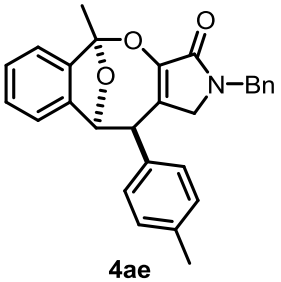

O
O
O
N–Bn
F

4ad

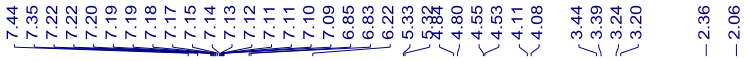

7.48 7.46 7.39 7.37 7.26 7.25 7.24 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.19 7.17 7.17 7.15 7.15 7.13 7.13 7.12 7.11 7.11 7.05 7.04 7.04 7.03 7.03 7.01 7.00 7.00 6.99 6.94 6.92 6.19 6.17 5.35 5.34 5.32 4.84 4.80 4.58 4.57 4.14 4.10 3.33 3.25 2.10 2.06

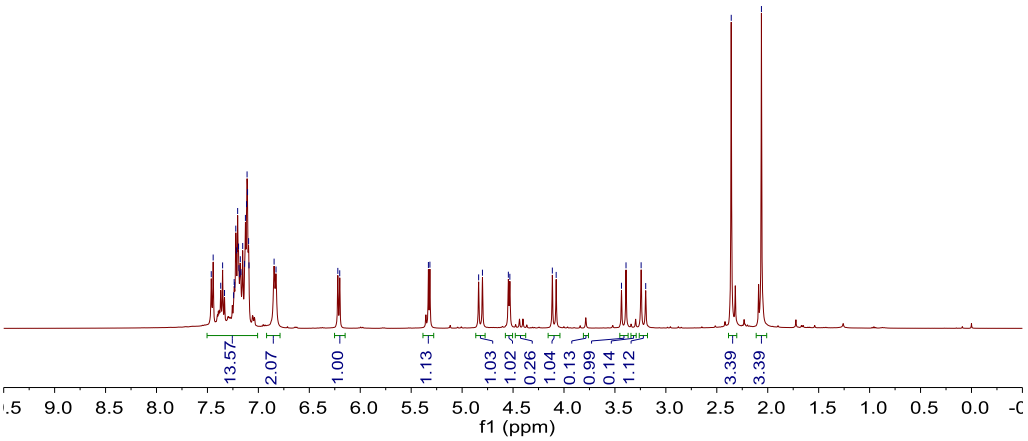

1.21 2.70 7.47 3.17 1.89 1.00 1.26 1.03 1.03 0.53 1.01 0.26 1.03 1.50 3.79
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

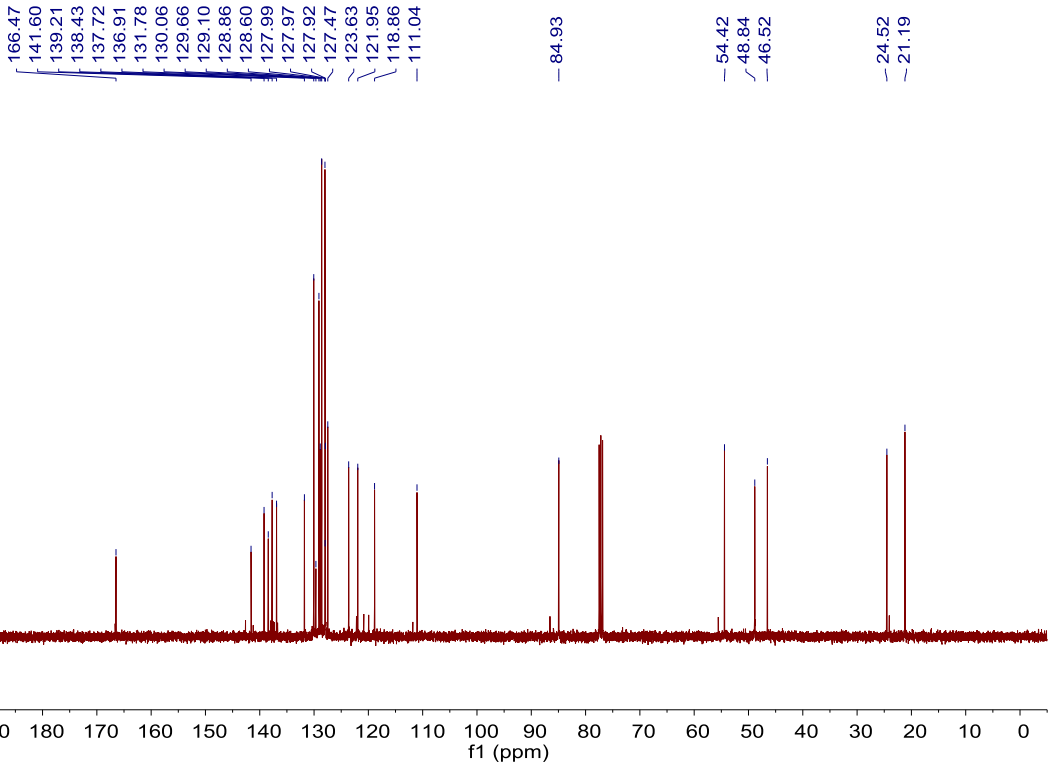

166.47 163.75 161.29 141.92 139.03 138.61 136.94 131.92 131.84 129.21 128.80 128.18 128.11 127.71 123.50 122.32 118.19 115.69 115.48 111.36 84.88 54.17 48.88 46.72 24.65
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

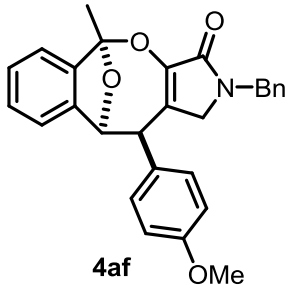

O
O
O
N–Bn

4ae

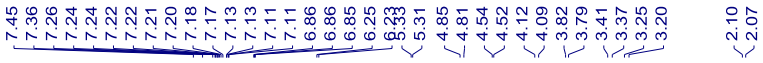

7.44
7.35
7.22
7.22
7.20
7.19
7.19
7.18
7.17
7.15
7.14
7.13
7.12
7.11
7.11
7.10
7.09
6.85
6.83
6.22
5.33
4.84
4.80
4.55
4.53
4.11
4.08
3.44
3.39
3.24
3.20
2.36
2.06

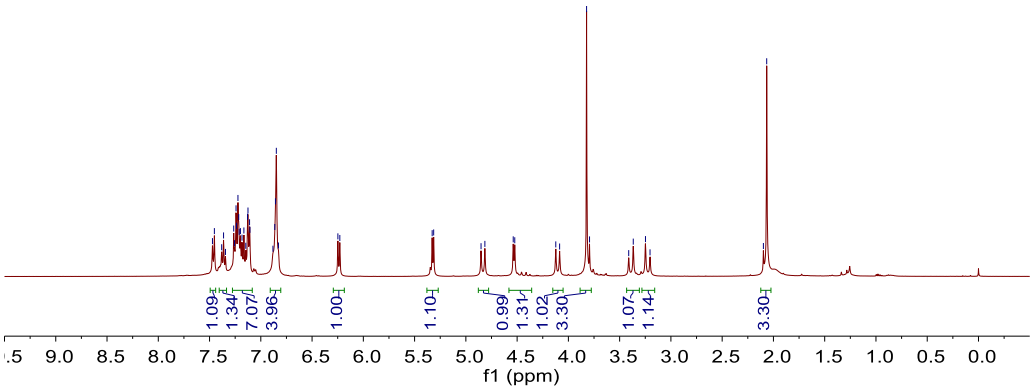

13.57
2.07
1.00
1.13
1.03
1.02
0.26
1.04
0.13
0.99
0.14
1.12
3.39
3.39
f1 (ppm)


166.47
141.60
139.21
138.43
137.72
136.91
131.78
130.06
129.66
129.10
128.86
128.60
127.99
127.97
127.92
127.47
123.63
121.95
118.86
111.04
84.93
54.42
48.84
46.52
24.52
21.19
f1 (ppm)

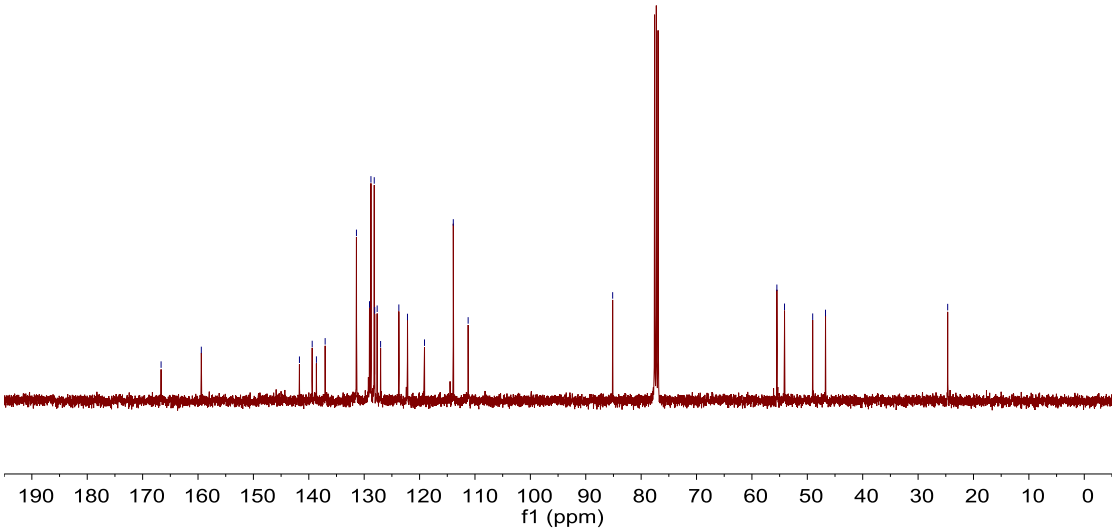

O
O
O
N–Bn
OMe

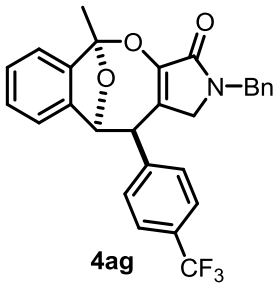
4af


7.45
7.36
7.26
7.24
7.24
7.22
7.22
7.21
7.20
7.18
7.17
7.13
7.13
7.11
7.11
6.86
6.86
6.85
6.25
6.23
5.31
4.85
4.81
4.54
4.52
4.12
4.09
3.82
3.79
3.41
3.37
3.25
3.20
2.10
2.07


1.09
1.34
7.07
3.96
1.00
1.10
0.99
1.31
1.02
3.30
1.07
1.14
3.30
f1 (ppm)

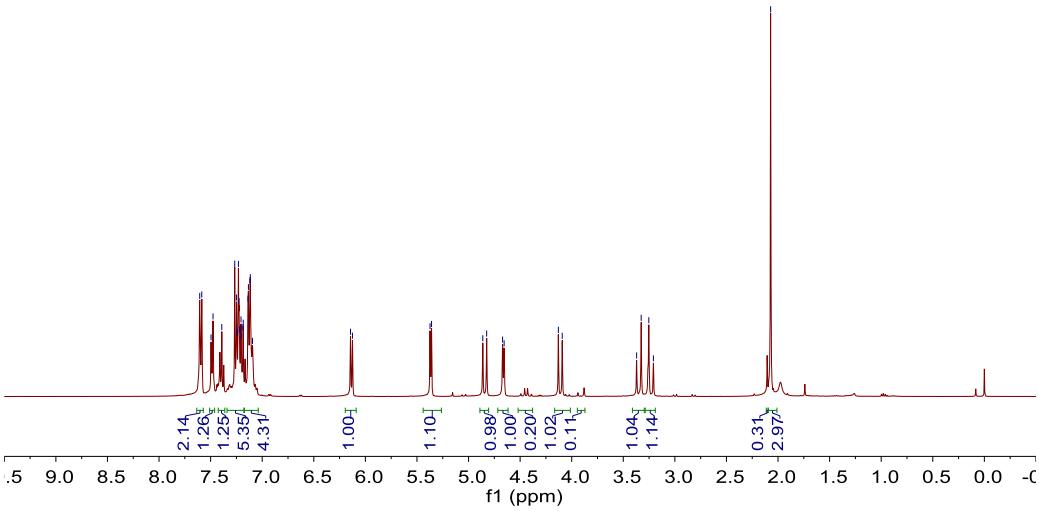

166.67
159.40
141.69
139.39
138.62
137.05
131.40
129.04
128.78
128.18
128.10
127.66
127.04
123.74
122.18
119.11
113.91
111.23
85.14
55.50
54.12
49.03
46.71
24.66

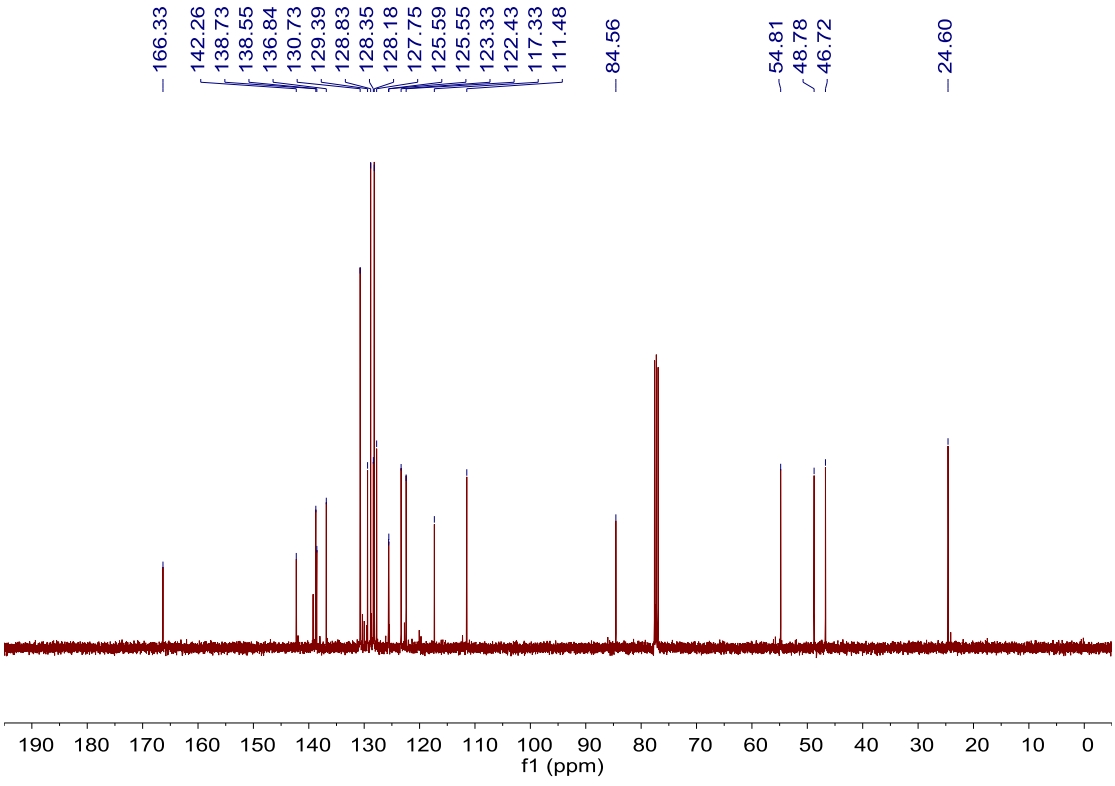

f1 (ppm)

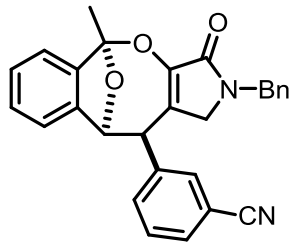

O
O
O
N–Bn
CF3

4ag


7.61
7.59
7.48
7.39
7.27
7.25
7.23
7.22
7.21
7.21
7.19
7.18
7.14
7.13
7.12
7.11
6.13
5.37
5.36
4.86
4.82
4.67
4.65
4.13
4.09
3.37
3.33
3.25
3.21
2.07

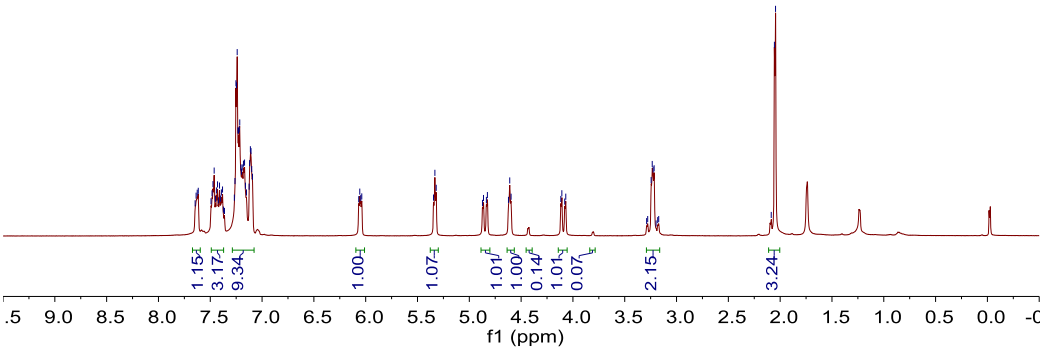

2.14
1.26
1.25
5.35
4.31
1.00
1.10
0.98
1.00
0.20
1.02
0.11
1.04
1.14
0.31
2.97
f1 (ppm)


166.33
142.26
138.73
138.55
136.84
130.73
129.39
128.83
128.35
128.18
127.75
125.59
125.55
123.33
122.43
117.33
111.48
84.56
54.81
48.78
46.72
24.60
f1 (ppm)

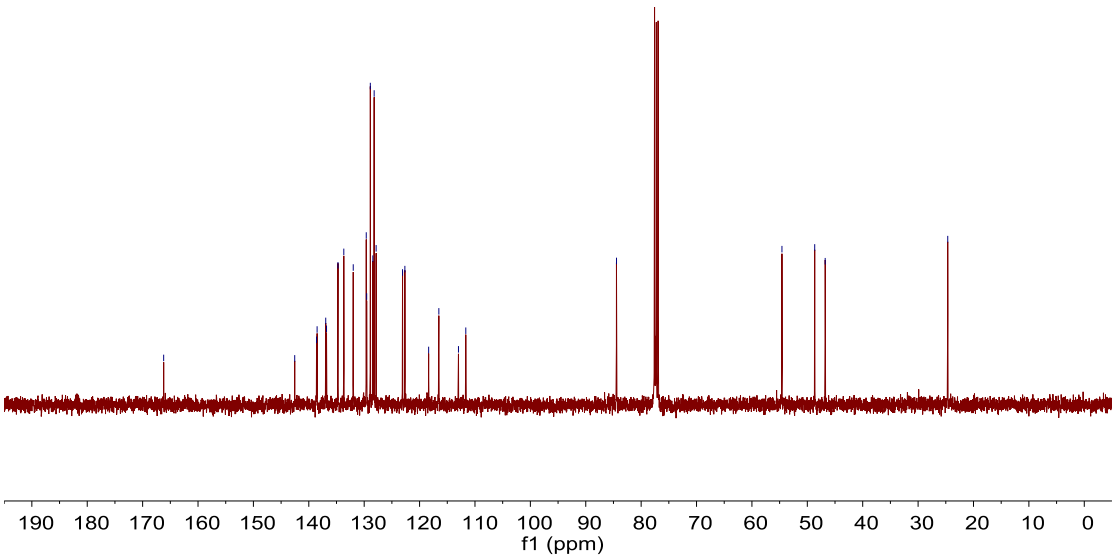

O
O
O
N–Bn
CN

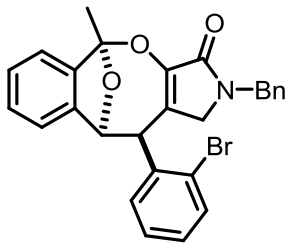
4ah


7.64 7.63 7.62 7.48 7.47 7.46 7.44 7.43 7.41 7.39 7.38 7.27 7.26 7.25 7.25 7.24 7.23 7.23 7.22 7.22 7.20 7.20 7.19 7.18 7.17 7.16 7.15 7.12 7.12 7.11 7.11 7.10 7.09 6.06 6.05 6.04 5.35 5.33 5.32 4.86 4.83 4.61 4.60 4.11 4.07 3.25 3.23 3.23 3.22 2.06 2.04

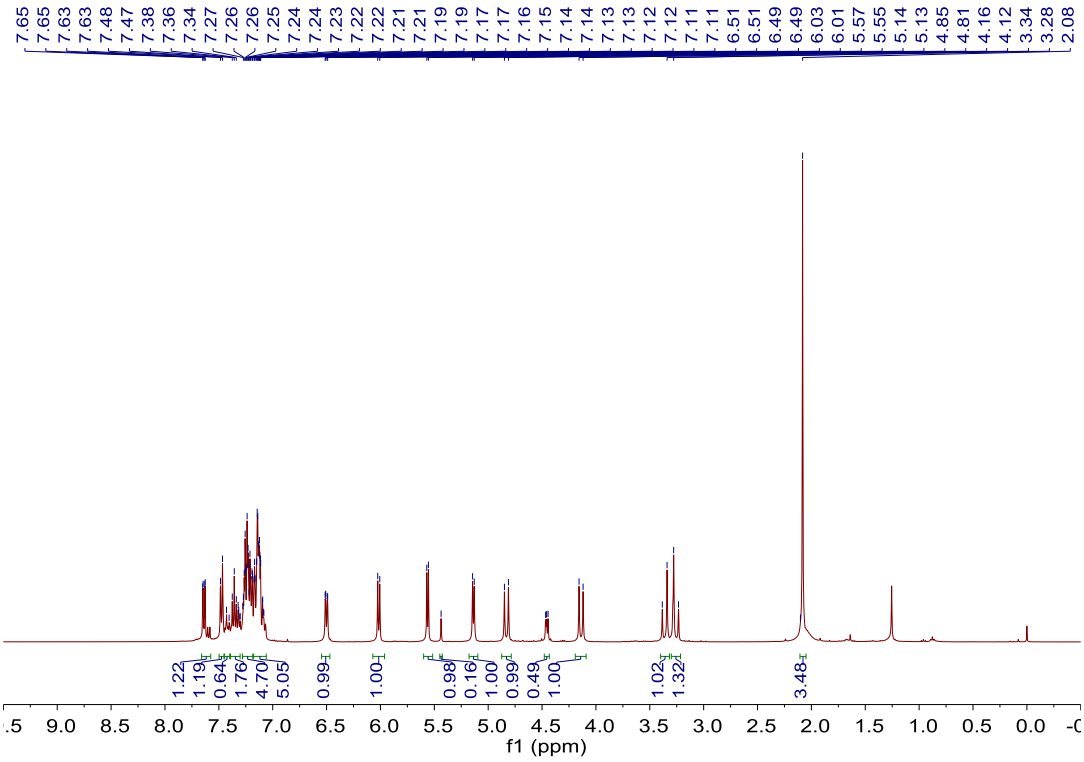

1.15 3.17 9.34 1.00 1.07 1.01 1.00 0.14 1.01 0.07 2.15 3.24
.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

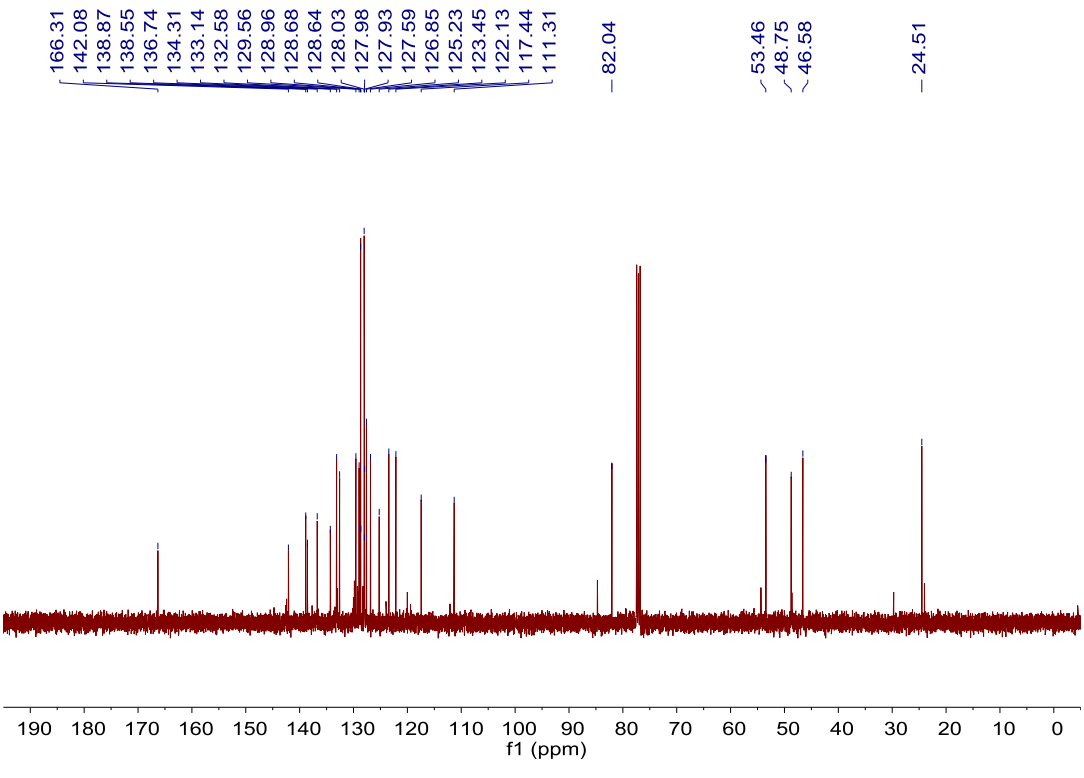

166.20 142.54 138.60 138.50 136.94 136.80 134.73 133.68 131.98 129.63 129.55 128.89 128.44 128.20 127.84 123.08 122.66 118.36 116.52 112.98 111.66
84.45
54.58 48.66 46.77
24.65


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O
O
N–Bn
Br

4ai


7.65 7.65 7.63 7.63 7.48 7.47 7.38 7.36 7.34 7.27 7.26 7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.21 7.21 7.19 7.19 7.17 7.17 7.16 7.15 7.14 7.14 7.13 7.13 7.12 7.12 7.11 7.11 6.51 6.49 6.49 6.03 6.01 5.57 5.55 5.14 5.13 4.85 4.81 4.16 4.12 3.34 3.28 2.08
1.22 1.19 0.64 1.76 4.70 5.05 0.99 1.00 0.98 0.16 1.00 0.99 0.49 1.00 1.02 1.32 3.48
f1 (ppm)


166.31 142.08 138.87 138.55 136.74 134.31 133.14 132.58 129.56 128.96 128.68 128.64 128.03 127.98 127.93 127.59 126.85 125.23 123.45 122.13 117.44 111.31
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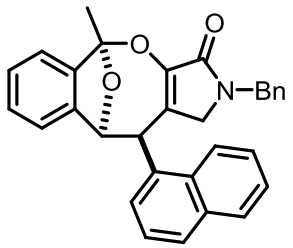
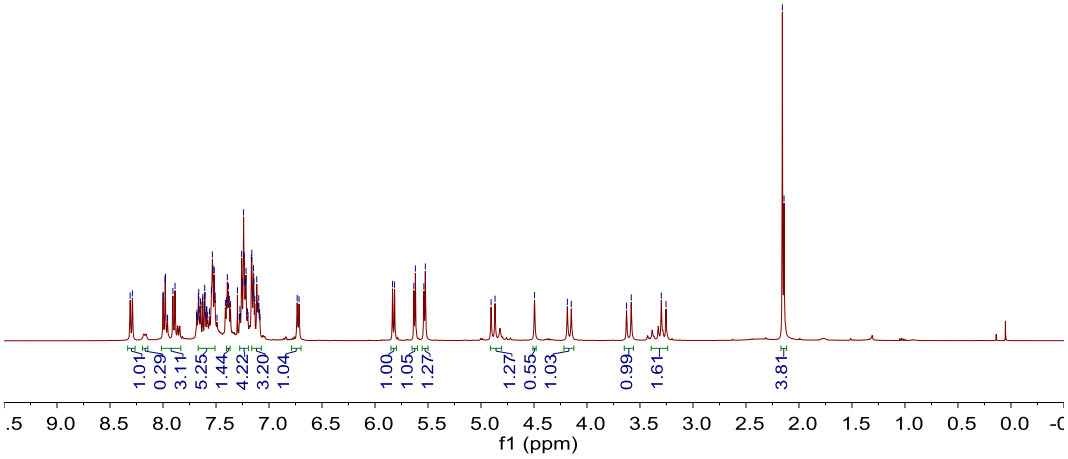
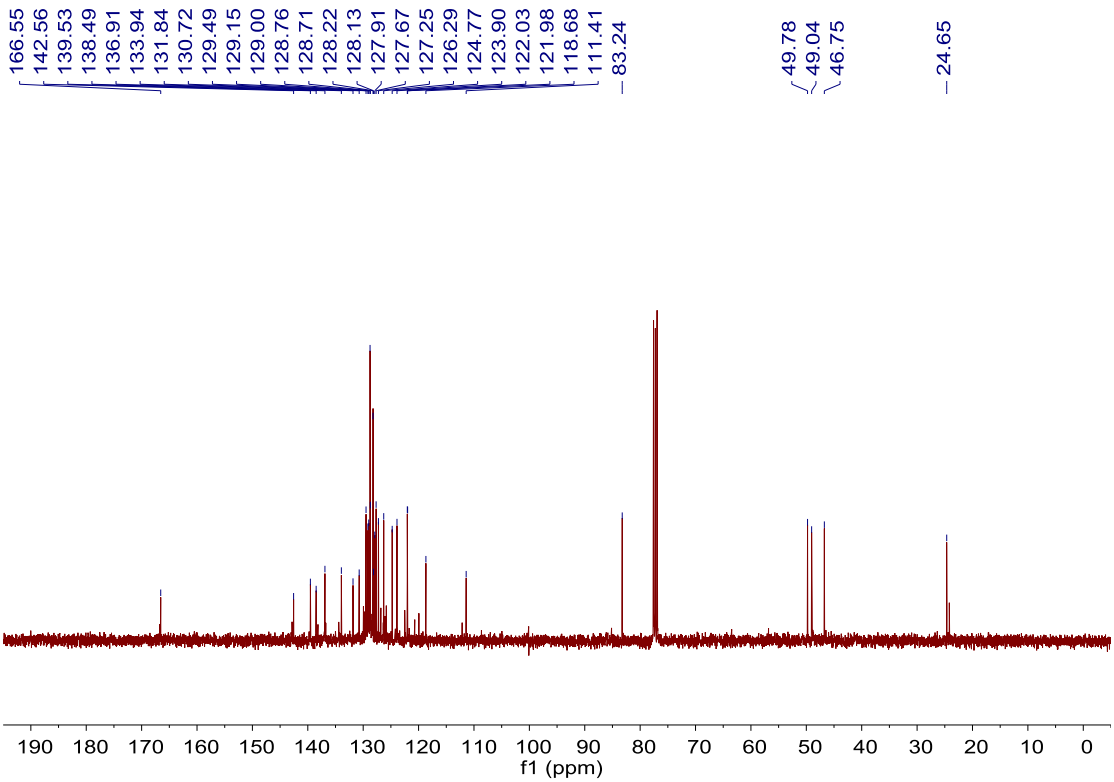
53.46 48.75 46.58
24.51
f1 (ppm)

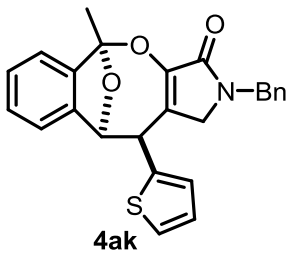

O
O
O
N–Bn

4aj


8.31 8.29 8.00 8.00 7.98 7.98 7.91 7.89 7.67 7.66 7.62 7.61 7.60 7.54 7.53 7.53 7.52 7.51 7.39 7.38 7.37 7.30 7.26 7.25 7.24 7.23 7.23 7.22 7.22 7.17 7.16 7.15 7.15 7.14 7.13 7.12 7.10 6.73 6.72 5.83 5.82 5.63 5.62 5.54 5.52 4.87 4.49 3.58 3.30 2.16 2.14


1.01 0.29 3.11 5.25 1.44 4.22 3.20 1.04 1.00 1.05 1.27 1.27 0.55 1.03 0.99 1.61 3.81
.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.55 142.56 139.53 138.49 136.91 133.94 131.84 130.72 129.49 129.15 129.00 128.76 128.71 128.22 128.13 127.91 127.67 127.25 126.29 124.77 123.90 122.03 121.98 118.68 111.41 83.24 49.78 49.04 46.75 24.65
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O
N–Bn
S

4ak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7.42
7.37
7.37
7.36
7.35
7.34
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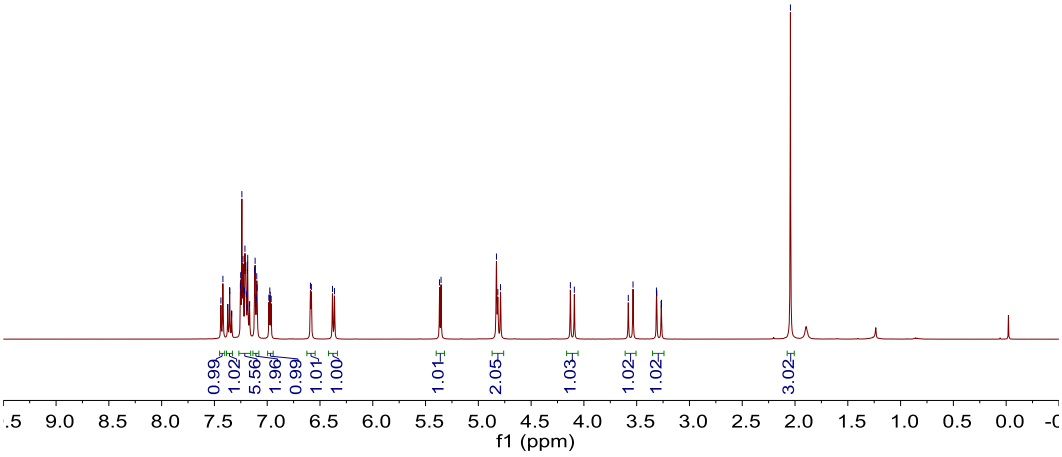
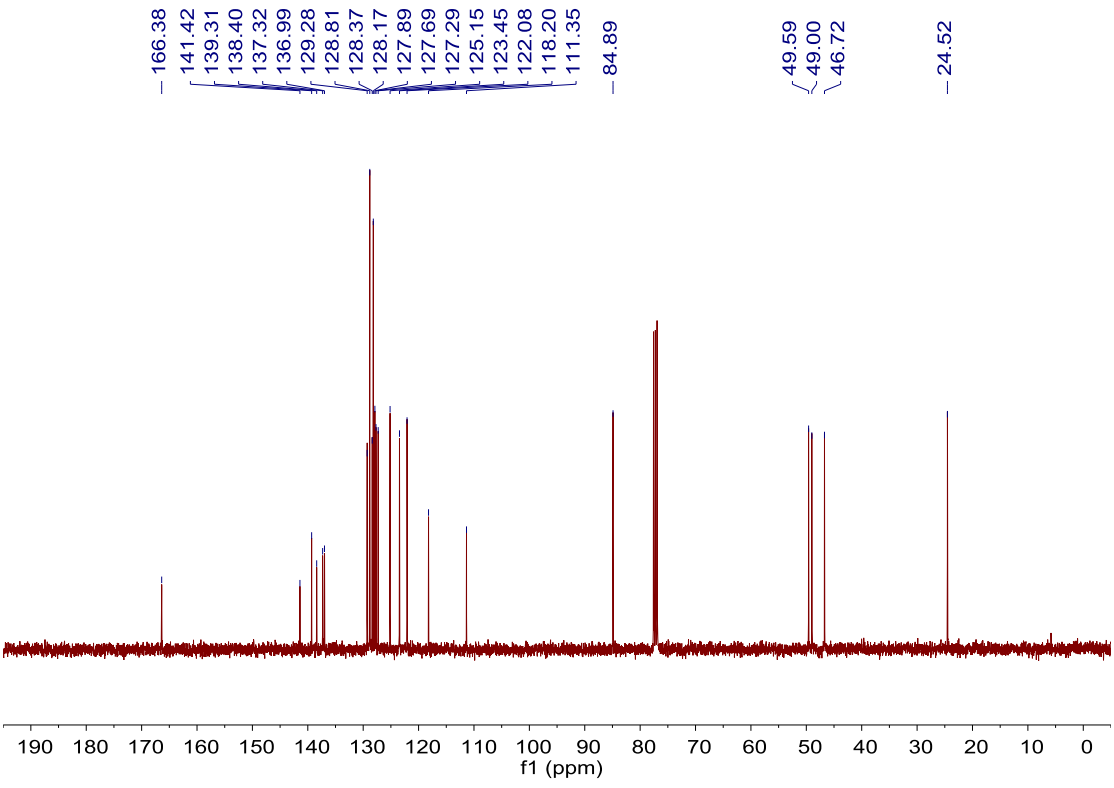
7.25
7.25
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.17
7.12
7.11
7.11
7.10
7.10
6.98
6.98
6.97
6.96
6.59
6.58
6.38
6.36
5.37
5.35
4.83
4.82
4.79
4.13
4.09
3.58
3.53
3.31
3.31
3.27
3.27
2.04


0.99
1.02
5.56
1.96
0.99
1.01
1.00
1.01
2.05
1.03
1.02
1.02
3.02
.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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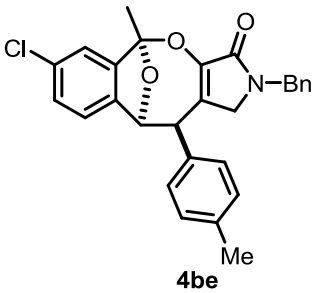
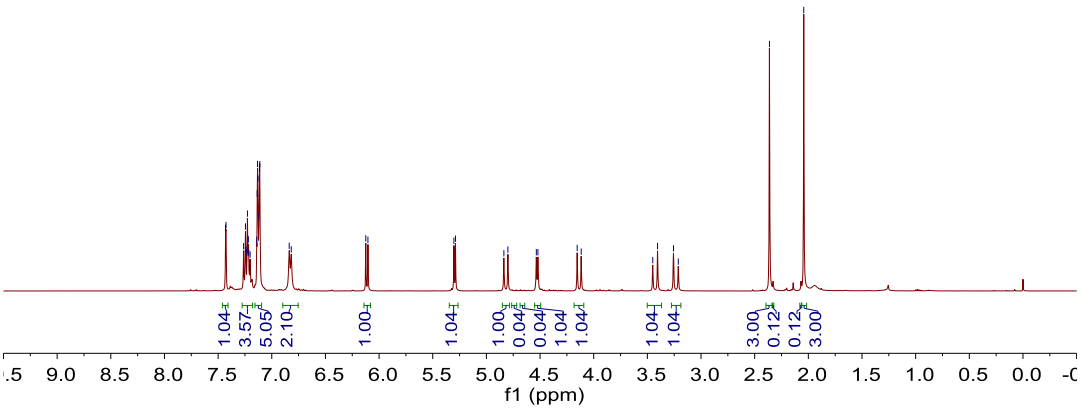
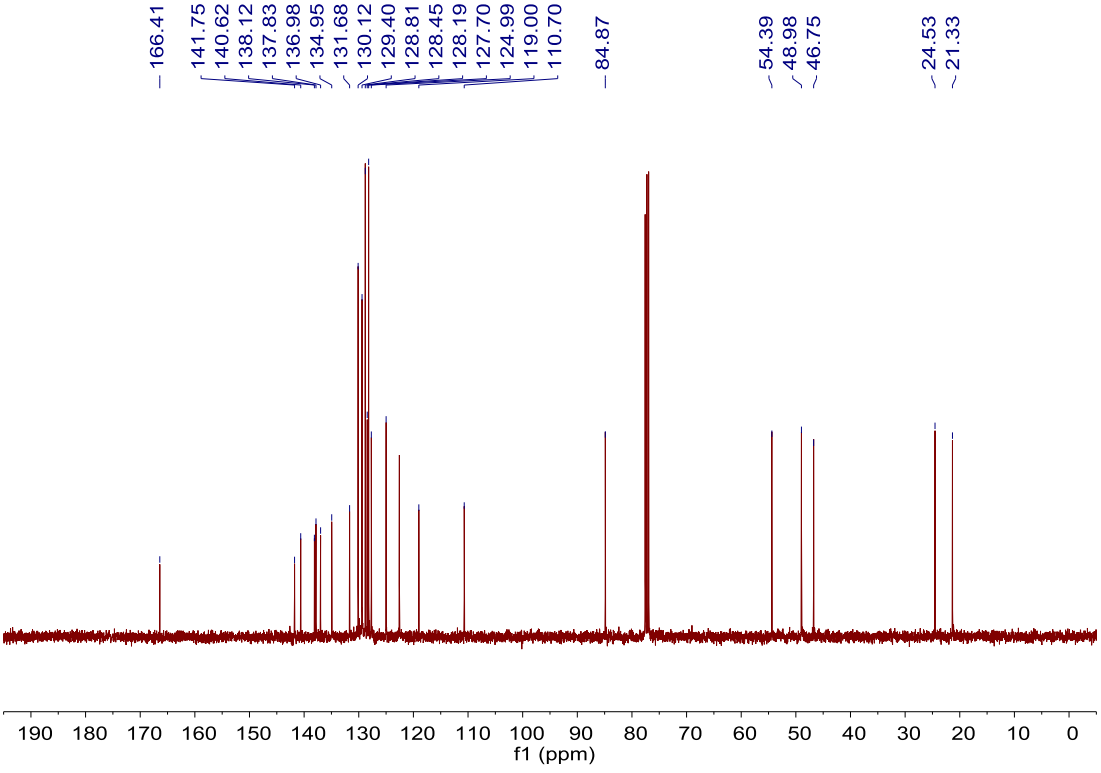
141.42
139.31
138.40
137.32
136.99
129.28
128.81
128.37
128.17
127.89
127.69
127.29
125.15
123.45
122.08
118.20
111.35
84.89
49.59
49.00
46.72
24.52
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Cl
O
O
O
N–Bn
Me

4be


7.43
7.43
7.26
7.25
7.24
7.23
7.22
7.14
7.14
7.13
7.13
7.12
7.12
7.11
6.84
6.82
6.10
5.30
5.29
4.84
4.80
4.53
4.52
4.15
4.12
3.45
3.41
3.26
3.21
2.36
2.04


1.04
3.57
5.05
2.10
1.00
1.04
1.00
0.04
0.04
1.04
1.04
1.04
3.00
0.12
0.12
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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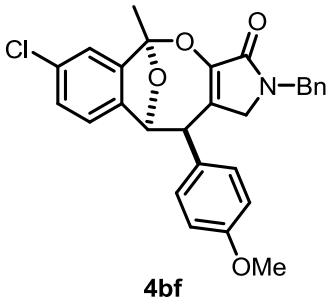
140.62
138.12
137.83
136.98
134.95
131.68
130.12
129.40
128.81
128.45
128.19
127.70
124.99
119.00
110.70
84.87
54.39
48.98
46.75
24.53
21.33
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Cl
O
O
O
N–Bn
OMe

4bf


7.43
7.43
7.27
7.25
7.24
7.23
7.22
7.15
7.14
7.14
7.13
7.13
7.12
7.12
7.11
6.85
6.15
6.13
5.30
5.28
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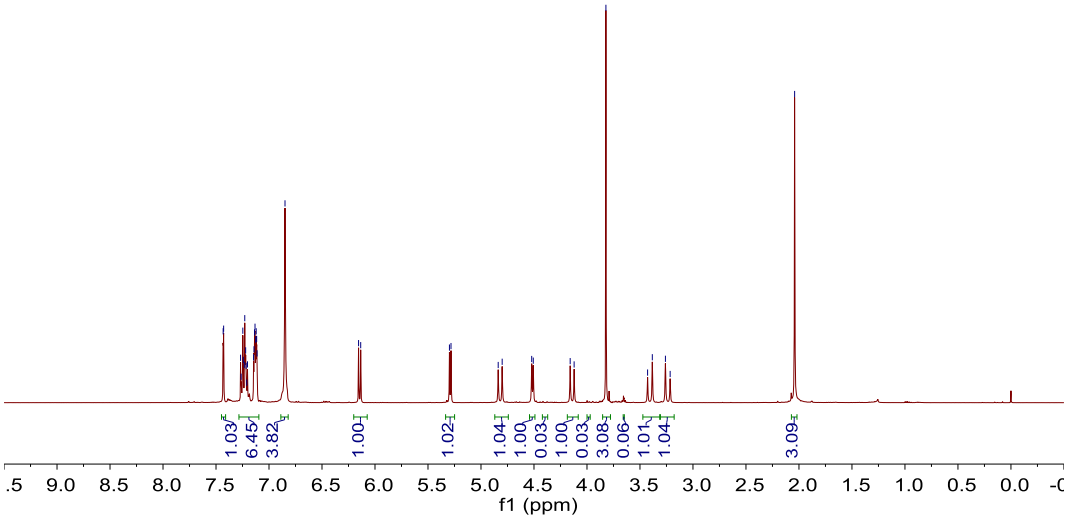
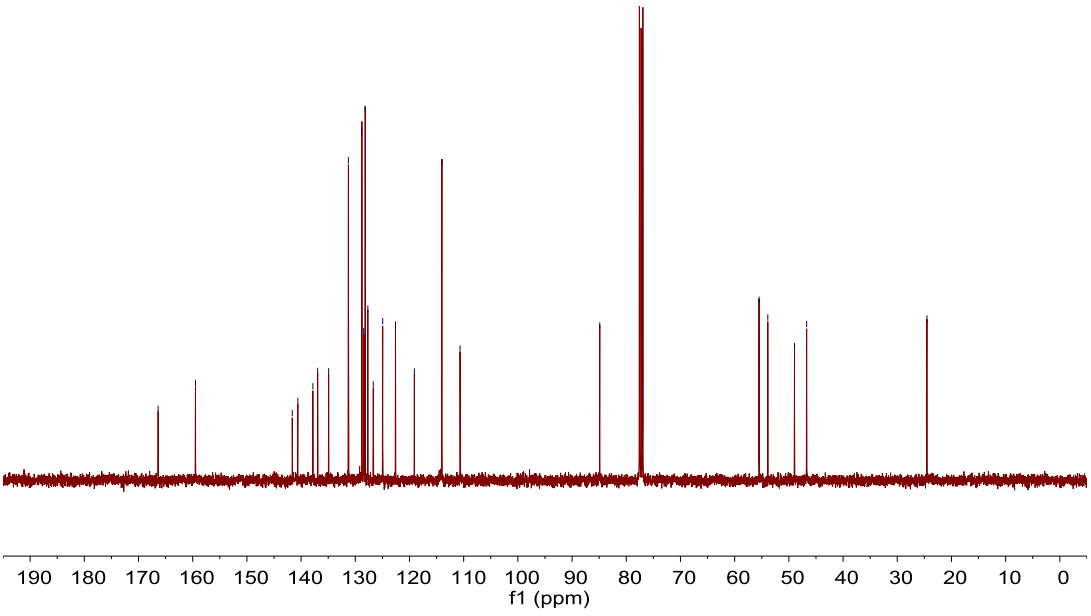
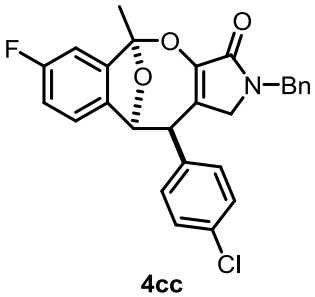
3.82
3.43
3.38
3.26
3.22
2.04


1.03
6.45
3.82
1.00
1.02
1.04
1.00
0.03
1.00
0.03
3.08
0.06
1.01
1.04
3.09
f1 (ppm)

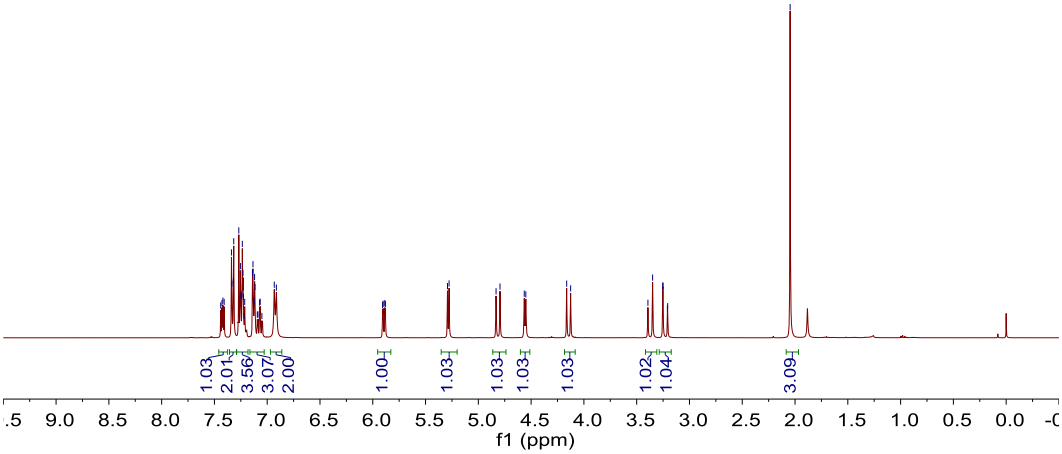

166.38
159.48
141.63
140.60
137.83
136.95
134.92
131.27
128.79
128.45
128.17
127.69
126.68
124.94
122.58
119.10
114.02
110.67
84.89
55.50
53.89
48.96
46.72
24.52

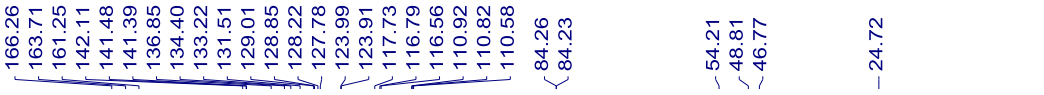

f1 (ppm)

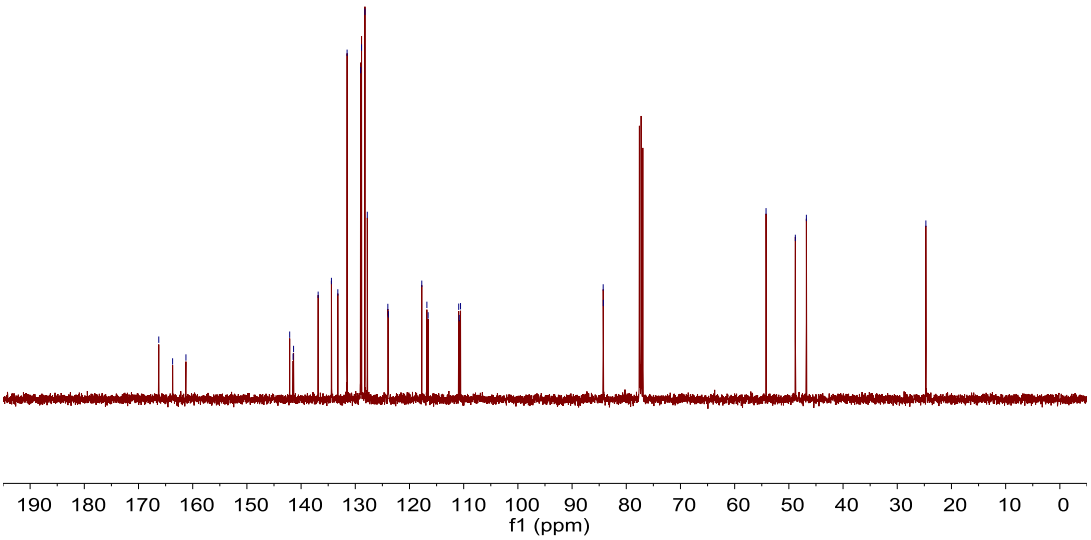

F
O
O
O
N–Bn
Cl

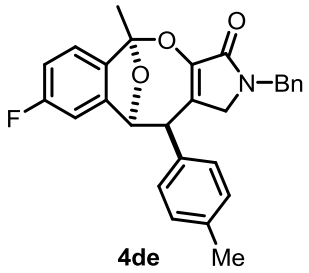
4cc


7.44 7.43 7.42 7.41 7.34 7.33 7.32 7.32 7.27 7.25 7.25 7.24 7.24 7.23 7.23 7.22 7.21 7.14 7.13 7.13 7.12 7.12 7.12 7.09 7.09 7.07 7.07 7.05 6.93 6.91 5.91 5.90 5.89 5.88 5.29 5.28 4.83 4.79 4.56 4.55 4.16 4.13 3.39 3.35 3.25 3.25 3.21 3.21 2.05

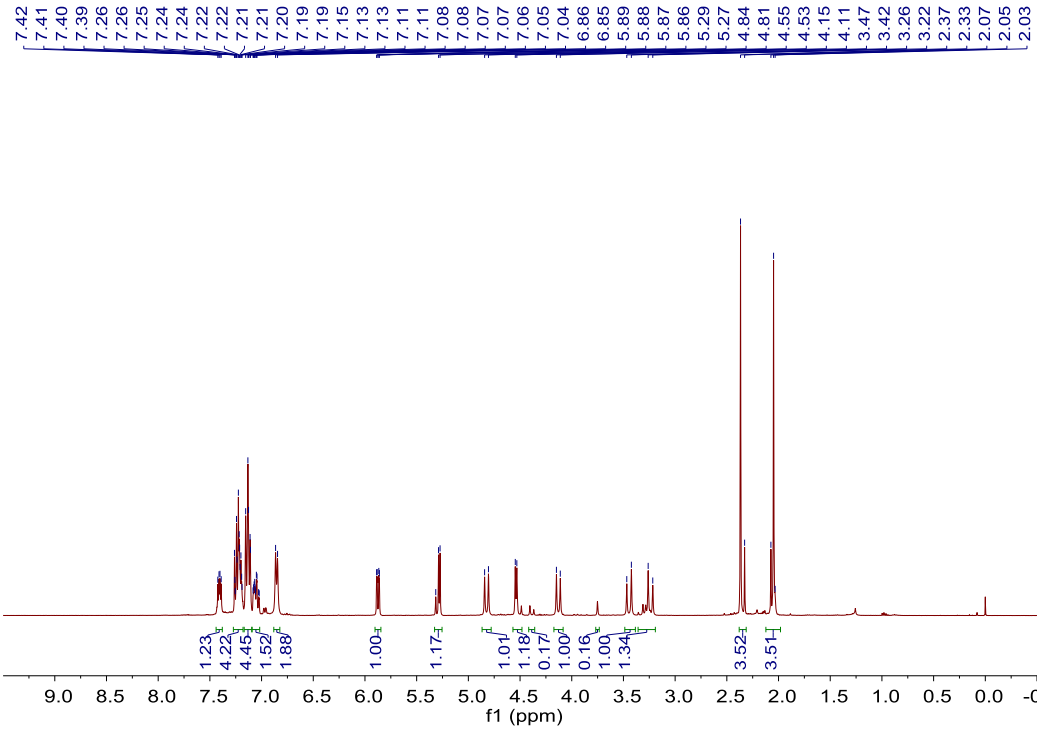

1.03 2.01 3.56 3.07 2.00 1.00 1.03 1.03 1.03 1.03 1.02 1.04 3.09
.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

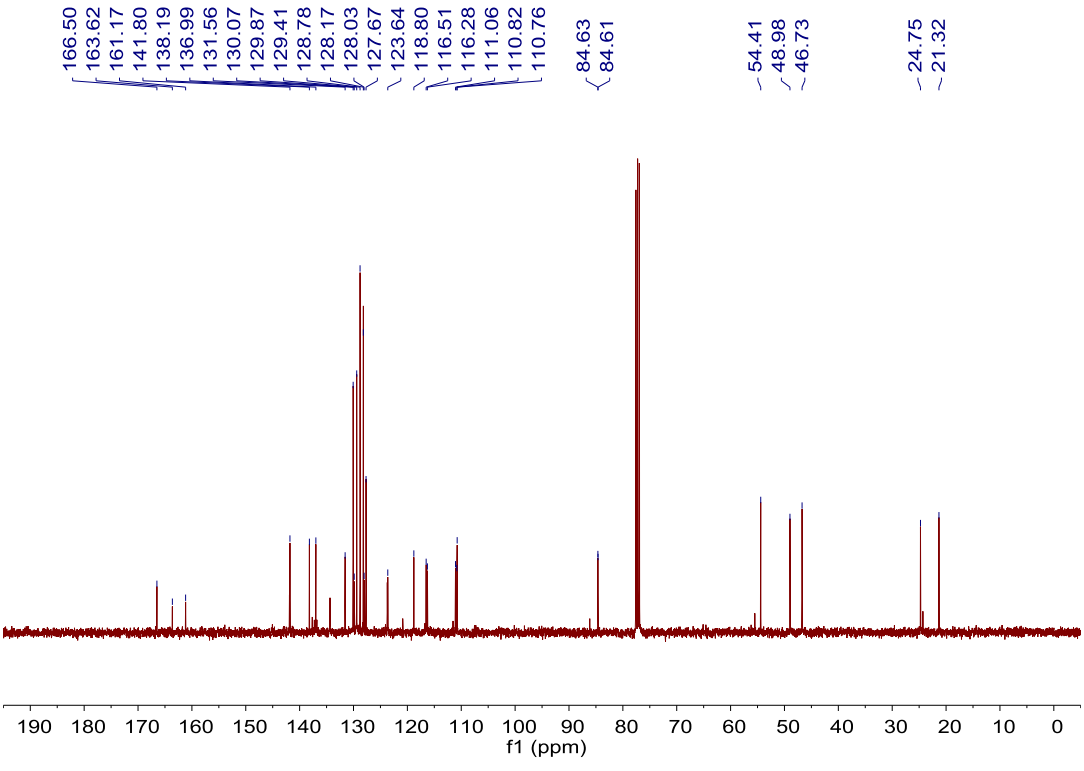

166.26 163.71 161.25 142.11 141.48 141.39 136.85 134.40 133.22 131.51 129.01 128.85 128.22 127.78 123.99 123.91 117.73 116.79 116.56 110.92 110.82 110.58 84.26 84.23 54.21 48.81 46.77 24.72

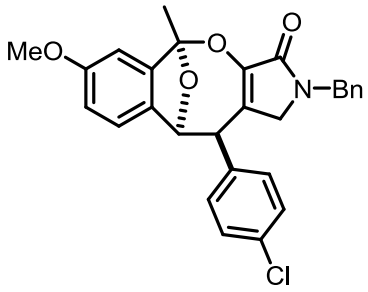

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O
O
N–Bn
F
Me

4de

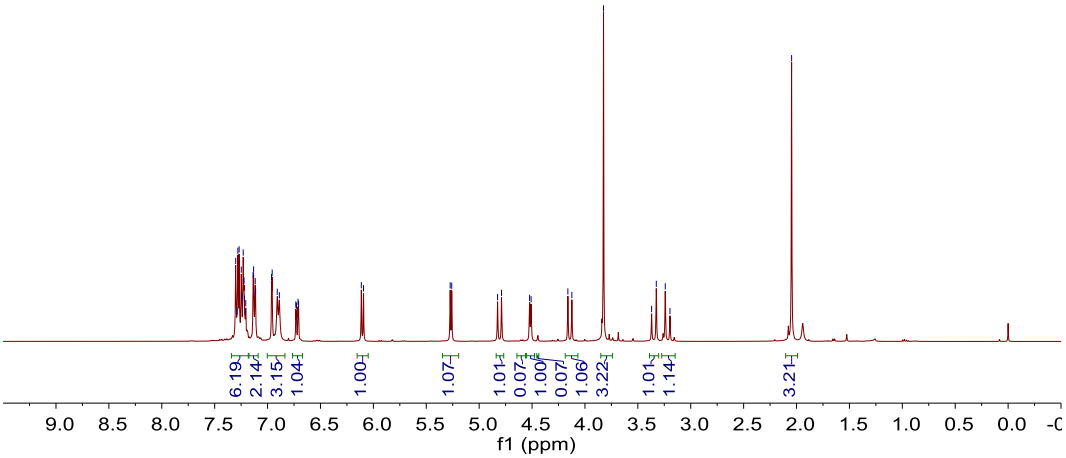

7.42 7.41 7.40 7.39 7.26 7.26 7.25 7.24 7.24 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.15 7.13 7.13 7.11 7.11 7.08 7.08 7.07 7.07 7.06 7.05 7.04 6.86 6.85 5.89 5.88 5.87 5.86 5.29 5.27 4.84 4.81 4.55 4.53 4.15 4.11 3.47 3.42 3.26 3.22 2.37 2.33 2.07 2.05 2.03
1.23 4.22 4.45 1.52 1.88 1.00 1.17 1.01 1.18 0.17 1.00 0.16 1.00 1.34 3.52 3.51
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.50 163.62 161.17 141.80 138.19 136.99 131.56 130.07 129.87 129.41 128.78 128.17 128.03 127.67 123.64 118.80 116.51 116.28 111.06 110.82 110.76
84.63 84.61
54.41 48.98 46.73
24.75 21.32
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


MeO
O
O
O
N–Bn
Cl

4ec


7.30
7.28
7.27
7.25
7.24
7.23
7.22
7.22
7.14
7.13
7.12
7.11
6.96
6.96
6.91
6.89
6.89
6.09
5.27
5.26
4.83
4.79
4.52
4.51
4.16
4.12
3.82
3.37
3.33
3.24
3.20
2.05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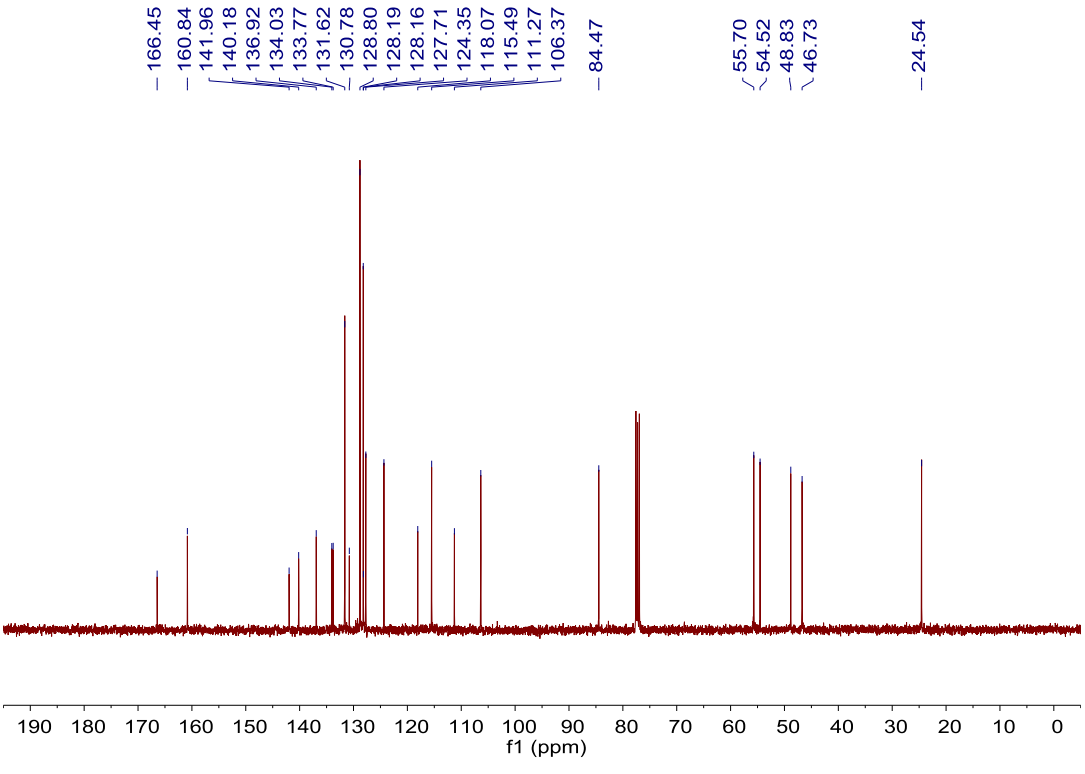
2.14
3.15
1.04
1.00
1.07
1.01
0.07
1.00
0.07
1.06
3.22
1.01
1.14
3.21
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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140.18
136.92
134.03
133.77
131.62
130.78
128.80
128.19
128.16
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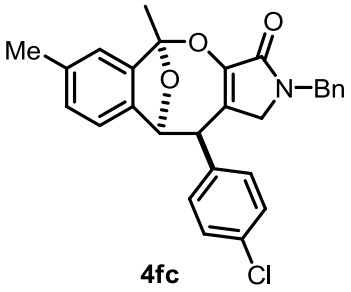
124.35
118.07
115.49
111.27
106.37
84.47
55.70
54.52
48.83
46.73
24.54
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Me
O
O
O
N–Bn
Cl

4fc


7.28
7.26
7.26
7.24
7.24
7.22
7.21
7.20
7.19
7.19
7.11
7.10
7.09
7.09
6.88
6.07
5.26
5.25
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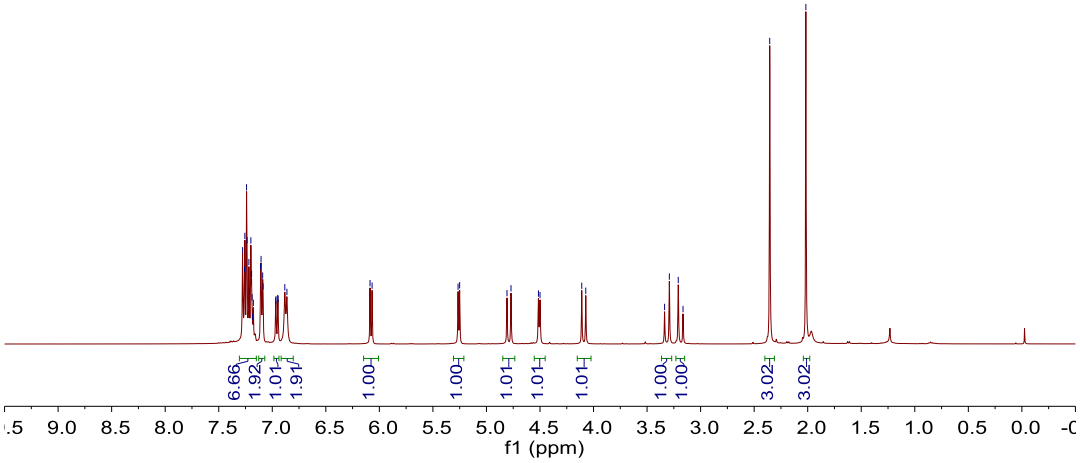
3.29
3.21
3.16
2.35
2.02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1.91
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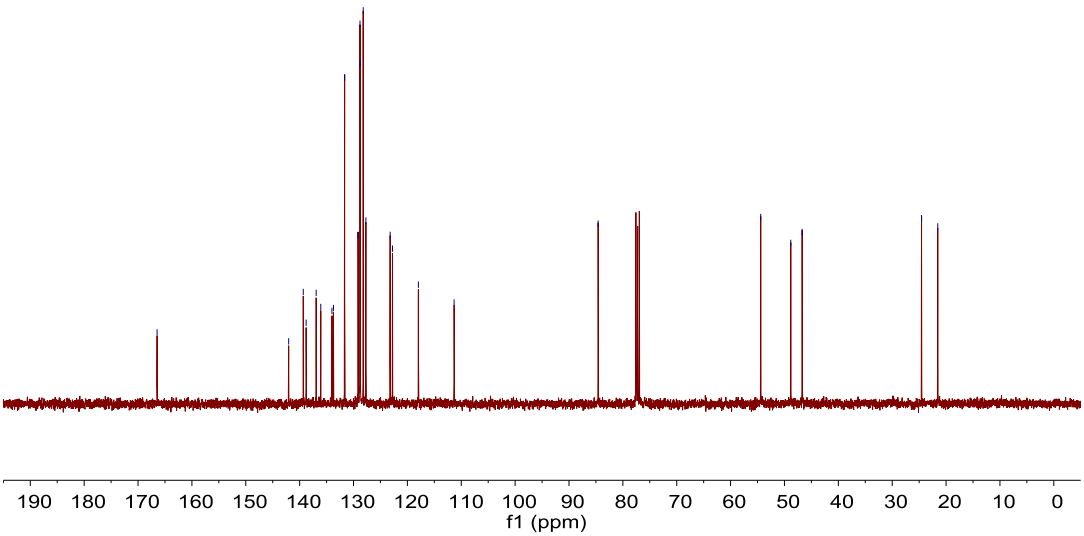
1.00
1.01
1.01
1.01
1.00
1.00
3.02
3.02
f1 (ppm)


166.46
142.03
139.33
138.80
136.93
136.08
134.01
133.73
131.65
129.16
128.80
128.76
128.20
127.70
123.20
122.76
117.95
111.32
84.59
54.41
48.83
46.72
24.55
21.54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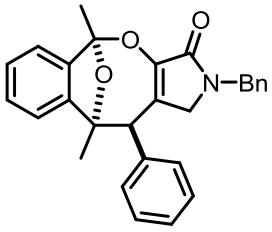
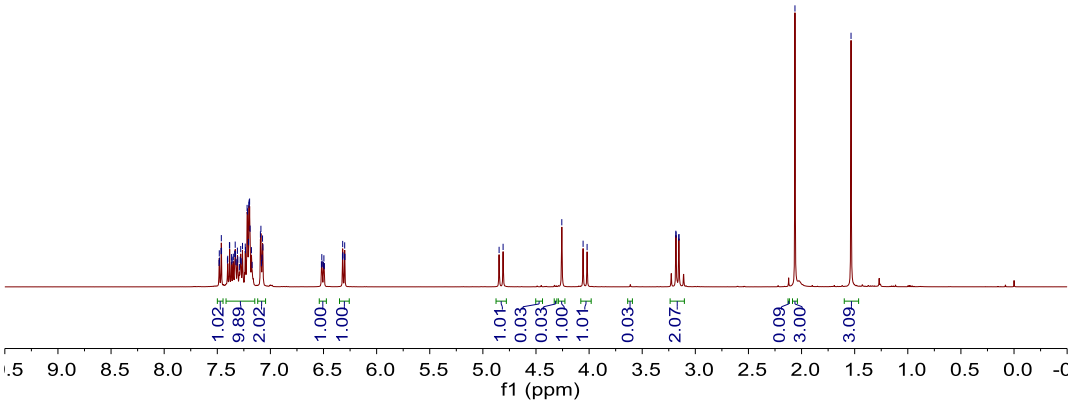
f1 (ppm)


O
O
O
N–Bn

4ga


7.48 7.46 7.46 7.46 7.40 7.39 7.38 7.37 7.35 7.35 7.34 7.33 7.33 7.31 7.31 7.28 7.26 7.24 7.23 7.22 7.22 7.21 7.20 7.20 7.20 7.19 7.18 7.09 7.09 7.08 7.07 7.07 6.52 6.50 6.32 6.32 6.32 6.30 6.30 4.85 4.81 4.26 4.06 4.02 3.18 3.18 3.16 3.15 2.06 1.53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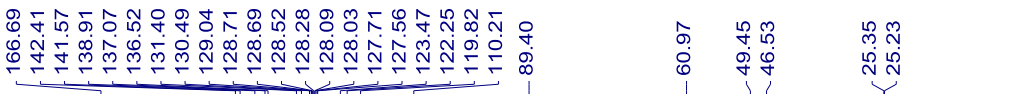
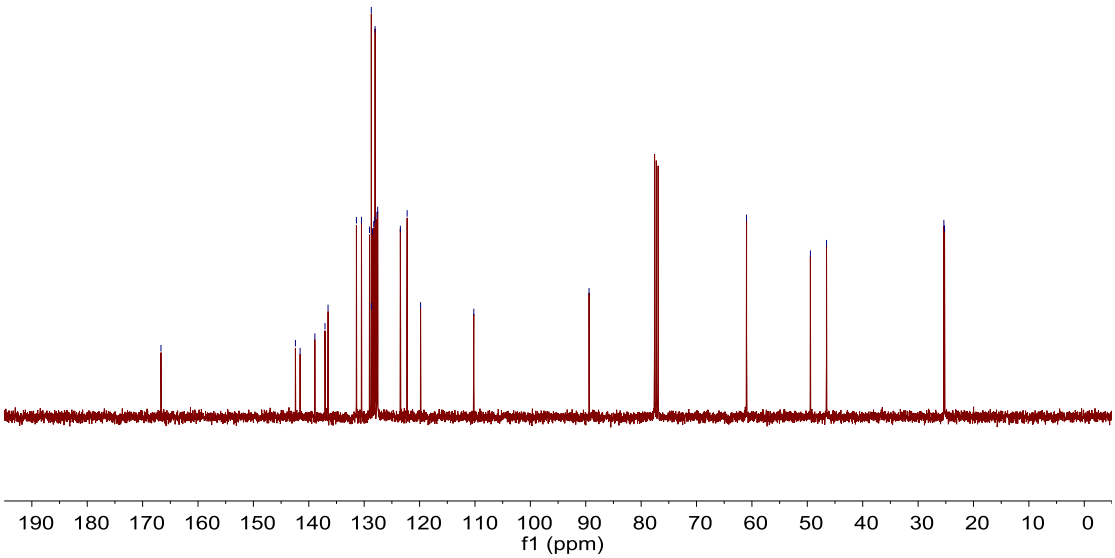
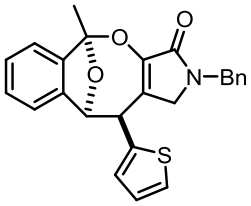
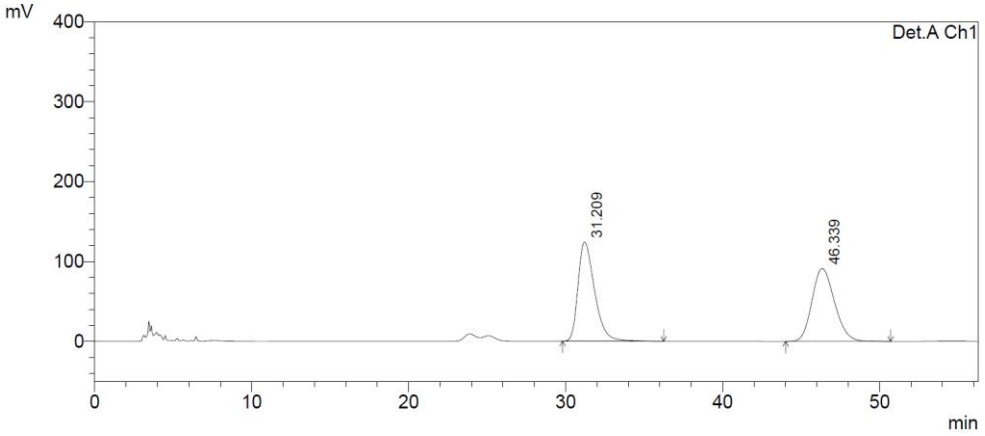
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.69 142.41 141.57 138.91 137.07 136.52 131.40 130.49 129.04 128.71 128.69 128.52 128.28 128.09 128.03 127.71 127.56 123.47 122.25 119.82 110.21 89.40 60.97 49.45 46.53 25.35 25.23


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

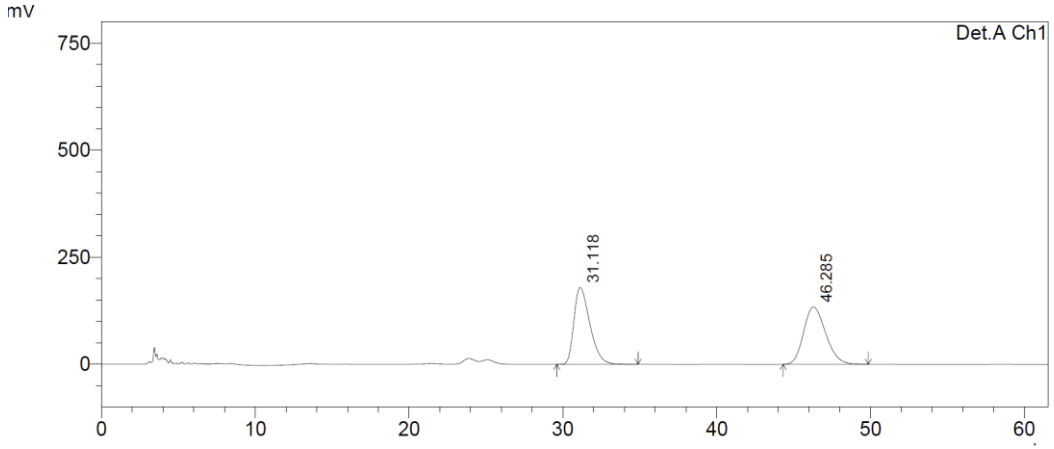


4ak'



Detector A Ch1 254nm

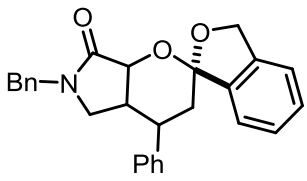
Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.209	9127253	124217	50.029	57.671
2	46.339	9116763	91170	49.971	42.329
Total		18244016	215387	100.000	100.000



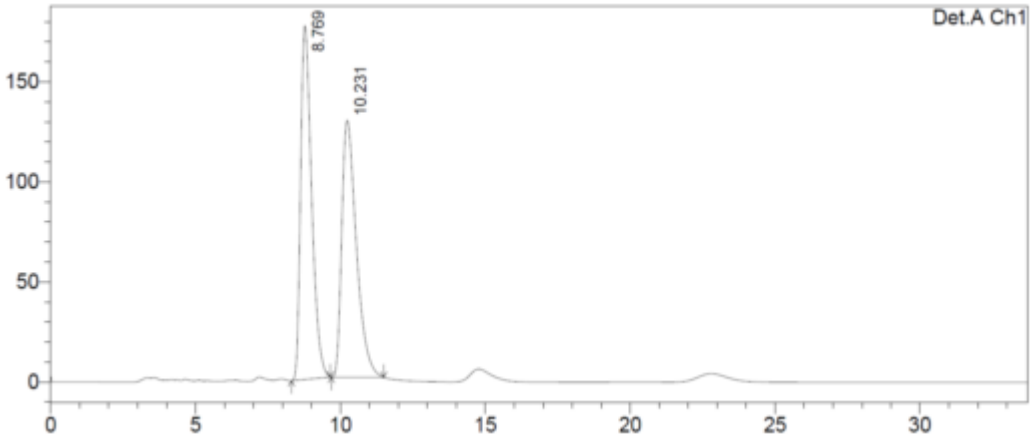
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.118	12947858	179900	49.263	57.328
2	46.285	13335472	133909	50.737	42.672
Total		26283330	313808	100.000	100.000

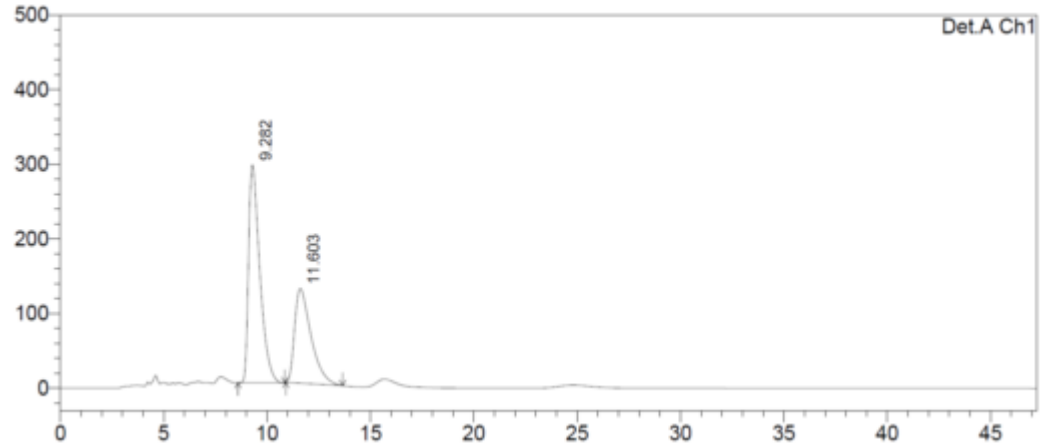
HPLC analysis condition: the *er* value was determined by HPLC analysis using a chiral IC column (2-propanol/n-hexane = 30:70, 1.0 mL/min, λ = 254 nm), retention time t_{minor} = 31.118 min; t_{major} = 46.285 min.



3aa'



Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.769	4652530	176837	50.850	57.902
2	10.231	4497010	128569	49.150	42.098
Total		9149540	305406	100.000	100.000



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.282	10588003	286072	59.666	69.158
2	11.603	7157371	127576	40.334	30.842
Total		17745374	413648	100.000	100.000

HPLC analysis condition: the *er* value was determined by HPLC analysis using a chiral OD-H column (2-propanol/n-hexane = 30:70, 1.0 mL/min, λ = 254 nm), retention time t_{minor} = 9.282 min; t_{major} = 11.603 min.