Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Exploiting an intramolecular Diels–Alder cyclization/dehydro-aromatization sequence for the total syntheses of ellipticines and calothrixin B

Chengdan Deng,† Yuancui Liu,† Mei Xu, Kaiqiang Xie* and Sheng Liu*

State Key Laboratory of Functions and Applications of Medicinal Plants, Guizhou Medical University, Guiyang 550014, China ;

Key Laboratory of Chemistry for Natural Products of Guizhou Province, Chinese Academy of Sciences, Guiyang 550014, China

Email: lsheng@126.com

Table of contents:

1. General description

Starting materials, reagents and solvents were purchased from commercial suppliers and used without further purification. The progress of reactions was monitored by silica gel thin layer chromatography (TLC) plates, visualized under UV. Products were purified by flash column chromatography (FCC) on 200 mesh silica gel. Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on a spectrometer operating at 600 MHz. Data is reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, dd =double doublet, t = triplet, q = quartet, m = multiplet). Carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded on a spectrometer operating at 150 MHz. Fluorine nuclear magnetic resonance spectrum (^{19}F NMR) was recorded on a spectrometer operating at 564 MHz.

2. Procedures and spectroscopic data for compounds 5a-r.

Acid **6** (2.0 mmol) was dissolved in dry tetrahydrofuran, amine **7** (2.4 mmol) and DCC (516 mg, 2.5 mmol) were added. Then the reaction mixture was stirred at room temperature for 12 h. TLC monitored that the acid **6** was completely consumed. The reaction solution was filtered, the filtrate was concentrated under reduced pressure to remove THF and the crude product was purified by flash column chromatography (EA/DCM = 6:1-3:1) to obtain the product (**5a-r**).

*Methyl (Z)-5-((E)-3-(1H-indol-3-yl)but-2-enamido)pent-2-enoate(**5a**).* 362 mg, yield 58%, white solid, IR (KBr) 3349, 3126, 1705, 1647, 1542 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.45 (s, 1H), 8.09 (t, *J* = 5.4 Hz, 1H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.69 (d, *J* = 2.4 Hz, 1H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.16 (t, *J* = 6.6 Hz, 1H), 7.11 (t, *J* = 7.2 Hz, 1H), 6.94-6.90 (m, 1H), 6.41 (s, 1H), 5.96 (d, *J* = 15.6 Hz, 1H), 3.65 (s, 3H), 3.29 (q, *J* = 6.0 Hz, 2H), 2.56 (s, 3H), 2.41 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 167.0, 166.0, 147.3, 143.9, 137.2, 126.9, 124.2, 121.8, 121.7, 120.5, 119.9, 117.3, 115.0, 112.0, 51.2, 37.0, 32.0, 17.0. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_2O_3$ [M + H]$^+$: 313.1552, found: 313.1561.

*Butyl(Z)-3-(2-((E)-3-(1H-indol-3-yl)acrylamido)phenyl)acrylate (**5b**).* 504 mg, yield 65 %, white solid, IR (KBr) 3384, 1658, 1581, 1457, 1072 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.67 (s, 1H), 9.87 (s, 1H), 8.01 (d, *J* = 7.8 Hz, 1 H), 7.89-7.84 (m, 3H), 7.81 (d, *J* = 16.2 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.25-7.20 (m, 3H), 6.95 (d, *J* = 15.6 Hz, 1H). 6.61 (d, *J* = 15.6 Hz, 1H), 4.14 (t, *J* = 6.6 Hz, 2H), 1.63-1.58 (m, 2H), 1.39-1.33 (m, 2H), 0.87 (t, *J* = 7.8 Hz 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.4, 165.5, 140.4, 137.5, 135.2, 131.27, 130.5, 127.0, 126.9, 126.0, 125.3, 124.9, 122.4, 120.5, 120.3, 118.5, 115.2, 112.4, 112.2, 63.7, 30.3, 18.6, 13.5. HRMS (ESI) m/z calcd for $C_{24}H_{24}N_2O_3Na$ [M + Na]$^+$: 411.1679, found: 411.1671.

*Methyl (Z)-5-((E)-3-(1H-indol-3-yl)acrylamido)pent-2-enoate (**5c**).* 597 mg, yield 57%, white solid, IR (KBr) 3373, 3010, 1698, 1522, 1279, 1088, 984 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.55 (s, 1H), 7.98 (t, *J* = 5.4 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.75 (d, *J* = 2.4 Hz, 1H), 7.63 (d, *J* = 16.2 Hz, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.15 (t, *J* = 7.2 Hz, 1H), 6.95-6.90 (m, 1H), 6.61 (d, *J* = 16.2, 1H), 5.96 (d, *J* = 15.6 Hz, 1H), 3.65 (s, 3H), 3.33 (d, *J* = 6.6 Hz, 2H), 2.42 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.4, 166.0, 147.2, 137.3, 133.0, 130.2, 124.9, 122.2, 121.9, 120.3, 119.9, 116.1, 112.2, 112.1, 51.2, 37.2, 32.0. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_3$ [M + H]$^+$: 299.1396, found: 299.1397.

*Methyl (Z)-5-((E)-3-(6-methoxy-1H-indol-3-yl)acrylamido)pent-2-enoate (**5d**).* 131 mg, yield 20%, light brown solid, IR (KBr) 3394, 3138, 1654, 1518, 1087 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.34 (s, 1H), 7.95 (t, *J* = 5.4 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 3.0 Hz, 1H), 7.56 (d, *J* = 15.6 Hz, 1H), 6.94-6.89 (m, 2H), 6.80 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.55 (d, *J* = 16.2 Hz, 1H), 5.96 (d, *J* = 15.6 Hz, 1H), 3.79 (s, 3H), 3.64 (s, 3H), 3.33 (d, *J* = 6.0 Hz, 2H), 2.42 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.4, 166.1, 156.0, 147.2, 138.3, 133.1, 129.3, 121.9, 120.5, 119.0, 115.8, 112.2, 110.2, 95.3, 55.2, 51.2, 37.2, 32.0. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_2O_4$ [M + H]$^+$: 329.1501, found: 329.1501.

*Methyl (Z)-5-((E)-3-(6-methyl-1H-indol-3-yl)acrylamido)pent-2-enoate (**5e**).* 200 mg, isolated yield 32%, white solid, IR (KBr) 3371, 3140, 1655, 1599, 1275, 1041 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.39 (s, 1H), 7.97 (t, *J* = 5.4 Hz, 1H), 7.76 (d, *J* = 7.8 Hz, 1H), 7.66 (d, *J* = 2.4 Hz, 1H), 7.59 (d, *J* = 15.6 Hz, 1H), 7.23 (s, 1H), 6.99 (d, *J* = 7.8 Hz, 1H), 6.95-6.90 (m, 1H), 6.57 (d, *J* = 15.6 Hz, 1H), 5.96 (d, *J* = 15.6 Hz, 1H), 3.65 (s, 3H), 3.35-3.32 (m, 3H), 2.41 (s, 4H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.4, 166.0, 147.2, 137.8, 133.2, 131.4, 129.8, 122.8, 121.9, 121.8, 119.6, 115.8, 112.1, 112.0, 51.2, 37.2, 32.0, 21.2. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_2O_3$ $[M + H]^+$: 313.1552, found: 313.1553.

*Methyl (Z)-5-((E)-3-(6-fluoro-1H-indol-3-yl)acrylamido)pent-2-enoate (**5f**).* 158 mg, isolated yield 25%, yellow solid, IR (KBr) 3371, 3024, 1707, 1606, 1458, 1108, 1074 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.60 (s, 1H), 7.98 (t, *J* = 6.0 Hz, 1H), 7.86-7.84 (m, 1H), 7.76 (d, *J* = 2.4 Hz, 1H), 7.59 (d, *J* = 16.2 Hz, 1H), 7.24 (dd, *J* = 9.6 Hz, 2.4 Hz, 1H), 7.05-7.01 (m, 1H), 6.94-6.89 (m, 1H), 6.60 (d, *J* = 16.2 Hz, 1H), 5.96 (d, *J* = 15.6 Hz, 1H), 3.64 (s 3H), 3.33 (d, *J* = 6.6 Hz,2H), 2.43-2.40 (m, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.2, 166.0, 159.8, 158.3, 147.2, 137.5, 137.4, 132.6, 130.8, 130.8, 121.9, 121.7, 120.9, 120.8, 116.5, 112.2, 108.7, 108.5, 98.5, 98.3, 51.2, 37.2, 32.0. ^{19}F NMR (564 MHz, DMSO-d_6) δ 120.59. HRMS (ESI) m/z calcd for $C_{17}H_{18}N_2O_3F$ $[M + H]^+$: 317.1301, found: 317.1300.

*Methyl (Z)-5-((E)-3-(6-chloro-1H-indol-3-yl)acrylamido)pent-2-enoate (**5g**).* 266 mg, isolated yield 40%, yellow solid, IR (KBr) 3356, 3016, 1666, 1469, 1350, 1063 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.66 (s, 1H), 8.00 (t, *J* = 5.4 Hz, 1H), 7.87 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 2.4 Hz, 1H), 7.60 (d, *J* = 16.2 Hz, 1H), 7.50 (d, *J* = 1.8 Hz, 1H), 7.19 (dd, *J* = 8.4, 1.8 Hz, 1H), 6.94-6.89 (m, 1H), 6.61 (d, *J* = 15.6 Hz, 1H), 5.95 (d, *J* = 15.6 Hz, 1H), 3.64 (s, 3H), 3.33 (m, 2H), 2.42 (q, *J* = 13.2 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.2, 166.0, 147.2, 137.8, 132.4, 131.1, 126.8, 123.7, 121.9, 121.1, 120.5, 116.9, 112.2, 111.9, 51.2, 37.2, 32.0. $C_{17}H_{18}N_2O_3Cl$ $[M + H]^+$: 333.1006, found: 333.1007.

*Methyl (Z)-5-((E)-3-(4-bromo-1H-indol-3-yl)acrylamido)pent-2-enoate (**5h**).* 264 mg, isolated yield 35%, yellow solid, IR (KBr) 3375, 3033, 1653, 1523, 1456, 1093 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.89 (s, 1H), 8.38 (d, *J* = 15.6 Hz, 1H), 7.99 (t, *J* = 6.0 Hz, 1H), 7.92 (s, 1H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.30 (d, *J* = 7.2 Hz, 1H), 7.05 (t, *J* = 7.8 Hz, 1H), 6.93-6.88 (m, 1H), 6.36 (d, *J* = 15.6 Hz, 1H), 5.95 (d, *J* = 15.6 Hz, 1H), 3.65 (s, 3H), 3.33-3.29 (m, 2H), 2.42 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.0, 165.8, 147.2, 137.9, 131.9, 126.2, 124.4, 123.9, 122.8, 121.8, 117.3, 112.8, 112.5, 112.0, 51.2, 37.3, 31.9. HRMS (ESI) m/z calcd for $C_{17}H_{18}N_2O_3Br$ $[M + H]^+$: 377.0501, found: 377.0491.

*Methyl 3-((E)-3-(((Z)-5-methoxy-5-oxopent-3-en-1-yl)amino)-3-oxoprop-1-en-1-yl)-1H-indole-6-carboxylate (**5i**).* 513 mg, isolated yield 72%, gray solid, IR (KBr) 3357, 3024, 2850, 1653, 1504, 1456, 1100 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.93 (s, 1H), 8.10 (s, 1H), 8.04 (t, *J* = 5.4 Hz, 1H), 8.00 (d, *J* = 2.4 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.64 (d, *J* = 15.6 Hz, 1H), 6.95-6.90 (m, 1H), 6.66 (d, *J* = 15.6 Hz, 1H), 5.96 (d, *J* = 15.6 Hz, 1H), 3.87 (s, 3H), 3.64 (s, 3H), 3.34 (d, *J* = 6.0 Hz, 2H), 2.42 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.9, 166.1, 166.0, 147.2, 136.7, 133.3, 132.2, 128.3, 123.1, 121.9, 120.8, 119.6, 117.2, 114.1, 112.4, 52.0, 51.2, 37.2, 32.0. HRMS (ESI) m/z calcd for $C_{19}H_{21}N_2O_5$ $[M + H]^+$: 357.1450, found: 357.1447.

*Methyl (Z)-5-((E)-3-(1-methyl-1H-indol-3-yl)acrylamido)pent-2-enoate (**5j**).* 256 mg, isolated yield 41%, white solid, IR (KBr) 3566, 3016, 1726, 1601, 1531, 1072 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.99 (t, *J* = 6.0 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.74 (s, 1H), 7.58 (d, *J* = 15.6 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.27 (t, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 7.8 Hz, 1H), 6.95-6.90 (m, 1H), 6.60 (d, *J* = 15.6 Hz, 1H), 5.97 (d, *J* = 15.6 Hz, 1H), 3.81 (s, 3H), 3.65 (s, 3H), 3.34 (d, *J* = 6.6 Hz, 2H), 2.43 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.3, 166.0, 147.2, 137.8, 133.8, 132.4, 125.3, 122.3, 121.9, 120.5, 120.0, 116.1, 111.1, 110.6, 51.2, 37.2, 32.7, 32.0. ^{19}F NMR (564 MHz, DMSO-d_6) δ 120.24. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_2O_3$ $[M + H]^+$: 313.1552, found: 313.1548.

*Methyl (Z)-5-((E)-3-(1-benzyl-1H-indol-3-yl)but-2-enamido)pent-2-enoate (**5k**).* 400 mg, isolated yield 26%, light brown solid, IR (KBr) 3334, 3016, 1653, 1076, 744 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.2 Hz, 1H),

7.35 (s, 1H), 7.34-7.30 (m, 4H), 7.23-7.21 (m, 2H), 7.15 (d, *J* = 7.2 Hz, 2H), 7.70-6.95 (m, 1H), 6.30 (s, 1H), 5.95 (d, *J* = 15.6 Hz, 1H), 5.85 (s, 1H), 5.31 (s, 2H), 3.75 (s, 3H), 3.50 (q, *J* = 6.6 Hz, 2H), 2.66 (s, 3H), 2.51 (q, *J* = 6.6 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.7, 166.7, 146.0, 145.9, 137.3, 136.6, 128.8, 127.8, 126.7, 125.5, 122.8, 122.5, 120.8, 120.7, 118.4, 115.6, 110.3, 51.4, 50.2, 37.8, 32.4, 18.1. HRMS (ESI) m/z calcd for $C_{25}H_{27}N_2O_3$ $[M + H]^+$: 403.2022, found: 403.2013.

*Methyl (E)-4-((E)-3-(1H-indol-3-yl)acrylamido)but-2-enoate (**5l**).* 171mg, isolated yield 30%, white solid, IR (KBr) 3315, 3137, 1698, 1602, 1523, 1066 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.58 (s, 1H), 8.20 (t, *J* = 5.4 Hz, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.78 (d, *J* = 2.4 Hz, 1H), 7.68 (d, *J* = 15.6 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.21-7.15 (m, 2H), 6.95-6.91 (m, 1H), 6.68 (d, *J* = 15.6 Hz, 1H), 5.97=5.93 (m, 1H), 4.04-4.02 (m, 2H), 3.66 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.4, 166.0, 146.5, 137.4, 133.6, 130.5, 124.9, 122.2, 120.4, 119.9, 115.6, 112.3, 112.0, 51.4. HRMS (ESI) m/z calcd for $C_{16}H_{17}N_2O_3$ $[M + H]^+$: 285.1239, found: 285.1237.

*Methyl (E)-3-(1-((E)-3-(1H-indol-3-yl)acryloyl)pyrrolidin-2-yl)acrylate (**5m**).* 143 mg, isolated yield 22%, yellow oil, 5m (two rotamer): IR (KBr) 3315, 3010, 1718, 1635, 1487, 1070 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.65 (s, 0.5H), 11.63 (s, 0.5H), 7.91 (s, 0.5H), 7.87 (d, *J* = 2.4 Hz,0.5H), 7.83 (d, *J* = 2.4 Hz, 0.5H), 7.74 (d, *J* = 10.2 Hz, 1H), 7.72 (s, 0.5H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.21-7.15 (m, 1.5H), 7.11 (t, *J* = 7.2 Hz, 0.5H), 7.01 (dd, *J* = 15.6, 6.0 Hz, 0.5H), 6.89 (dd, *J* = 15.6, 5.4 Hz, 0.5H), 6.78 (d, *J* = 15.6 Hz, 0.5H), 6.57 (d, *J* = 15.0 Hz, 0.5H), 5.99 (d, *J* = 5.4 Hz, 0.5H), 5.81 (d, *J* = 16.2 Hz, 0.5H), 4.99 (t, *J* = 6.6 Hz, 0.5H), 4.75 (t, *J* = 4.2 Hz, 0.5H), 3.69-3.51 (m, 5H), 2.22-2.16 (m, 0.5H), 2.05-2.01 (m, 0.5H), 1.96-1.93 (m, 0.5H), 1.90-1.87 (m, 1.5H), 1.85-1.82 (m, 0.5H), 1.80-1.77 (m, 0.5H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.2, 165.8, 165.2, 164.9, 150.0, 149.3, 137.3, 135.4, 135.2, 131.7, 131.5, 130.8, 130.6, 128.7, 125.0, 124.9, 122.3, 120.6, 120.5, 120.3, 120.0, 119.8, 119.3, 113.1, 113.0, 112.3, 57.3, 56.9, 51.5, 51.3, 46.4, 46.2, 31.8, 29.6, 23.5, 21.7. HRMS (ESI) m/z calcd for $C_{18}H_{17}N_2O_4$ $[M + H]^+$:325.1188, found: 325.1187.

*Methyl (Z)-5-((E)-3-(1H-indol-3-yl)acrylamido)hex-2-enoate (**5n**).* 287 mg, isolated yield 46%, light brown solid, IR (KBr) 3307, 3030, 1716, 1653, 1489, 1111, 984 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.20 (s, 1H), 7.88-7.84 (m, 2H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.38 (d, *J* = 3.0 Hz, 1H), 7.25-7.19 (m, 2H), 6.99-6.94 (m, 1H), 6.43 (d, *J* = 15.6 Hz, 1H), 5.93 (d, *J* = 15.6 Hz, 1H), 5.72(d, *J* = 8.4 Hz, 1H), 4.37-4.32 (m, 1H), 3.71 (s, 3H), 2.50-2.47 (m, 2H), 1.24 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.8, 166.7, 144.9, 137.2, 135.0, 128.7, 125.3, 123.6, 122.9, 121.0, 120.2, 115.5, 113.2, 111.9, 51.5, 44.4, 39.1, 20.4. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_2O_3$ $[M + H]^+$: 313.1552, found: 313.1551.

*Methyl (Z)-4-(1-((E)-3-(1H-indol-3-yl)acryloyl)piperidin-2-yl)but-2-enoate (**5o**).* 211 mg, isolated yield 30%, golden yellow oil, IR (KBr) 3346, 3012, 2870, 1717, 1635, 1080 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.58 (s, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.85 (s, 1H), 7.72 (d, *J* = 15.6 Hz, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.14 (t, *J* = 7.2, 1H), 6.93 (d, *J* = 15.0 Hz, 2H), 6.00 (d, *J* = 15.0, 1H), 3.56 (s, 3H), 2.74 (s, 1H), 2.50 (s, 4H), 1.69-1.56 (m, 5H), 1.34 (s, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 165.9, 137.2, 135.9, 129.8, 125.1, 122.1, 120.4, 119.8, 112.5, 112.1, 59.8, 51.2, 40.0, 20.8, 18.6, 14.1. HRMS (ESI) m/z calcd for $C_{21}H_{25}N_2O_3$ $[M + H]^+$: 353.1865, found: 353.1875.

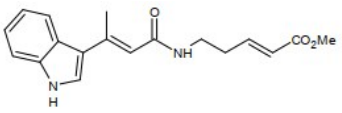
*Butyl (Z)-3-(2-((E)-3-(1H-indol-3-yl)but-2-enamido)phenyl)acrylate (**5p**).* 523 mg, isolated yield 65%, light yellow solid, IR (KBr) 3375, 3219, 2933, 1698, 1327, 1189 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.58 (s, 1H), 9.95 (s, 1H), 8.14 (s, 1H), 7.87-7.84 (m, 2H), 7.80 (s, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 1H), 7.24-7.19 (m, 3H), 6.75 (s, 1H), 6.60 (d, *J* = 15.6 Hz, 1H), 4.14 (t, *J* = 6.0 Hz, 2H), 2.63 (s, 3H), 1.62-1.58 (m, 2H), 1.38-1.34 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.4, 146.8, 140.7, 137.7, 137.4, 130.5, 127.7, 126.8, 125.4, 124.2, 121.9, 120.7, 120.1, 118.2, 117.3, 114.1, 112.2, 63.7, 30.3, 18.7, 17.3, 13.5. HRMS (ESI) m/z calcd for $C_{25}H_{27}N_2O_3$ $[M + H]^+$: 403.2016, found: 403.2004.

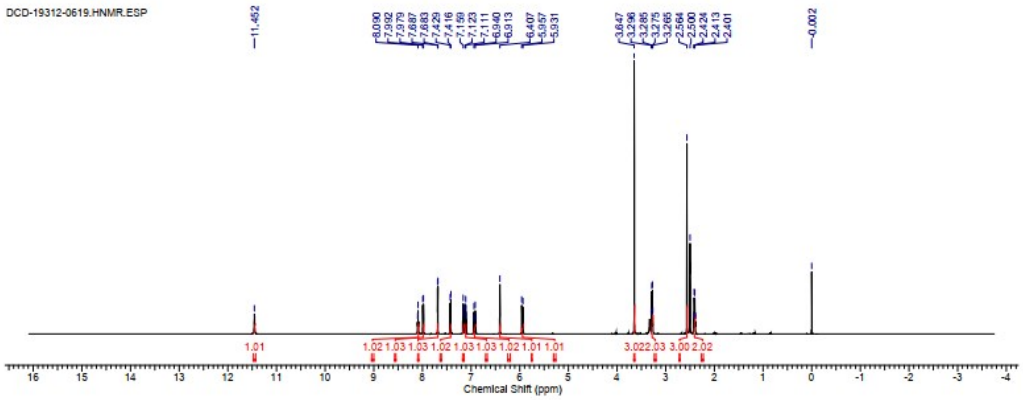
*Tert-butyl (Z)-3-(2-((E)-3-(1H-indol-3-yl)acrylamido)phenyl)acrylate (**5q**).* 202 mg, isolated yield 26%, white solid, IR (KBr) 3238, 2977, 1653, 1576, 1488, 1244, 1151 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.67 (s, 1H), 9.86

(s, 1H), 8.03 (d, *J* = 6.6 Hz, 1H), 7.86-7.81 (m, 4H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.23-7.22 (m, 3H), 6.99 (d, *J* = 16.2 Hz, 1H), 6.52 (d, *J* = 16.2 Hz, 1H), 1.47 (s, 9H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 165.7, 165.5, 139.5, 137.5, 137.4, 135.2, 131.3, 130.4, 127.9, 126.8, 126.0, 125.3, 124.9, 122.4, 120.5, 120.1, 120.0, 115.3, 112.4, 112.2, 80.0, 27.8. HRMS (ESI) m/z calcd for $C_{24}H_{25}N_2O_3$ $[M + H]^+$: 389.1860, found: 389.1847.

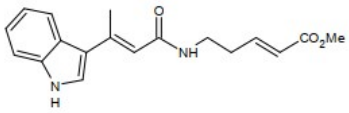
*Benzyl (Z)-3-(2-((E)-3-(1H-indol-3-yl)acrylamido)phenyl)acrylate (**5r**).* 507 mg, isolated yield 60%, white solid, IR (KBr) 3325, 2930, 2850, 2359, 1627, 1244 cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.68 (s, 1H), 9.90 (s, 1H), 8.01 (d, *J* = 6.0 Hz, 1H), 7.95 (d, *J* = 15.6 Hz, 1H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 15.6 Hz, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.45-7.41 (m, 3H), 7.34 (t, *J* = 7.2 Hz, 2H), 7.28 (d, *J* = 7.2Hz, 1H), 7.23 (t, *J* = 7.8 Hz, 3H), 6.97 (d, *J* = 15.6 Hz, 1H), 6.69 (d, *J* = 15.6 Hz, 1H), 5.23 (s, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.2, 165.5, 140.9, 137.6, 137.5, 136.2, 135.3, 131.3, 130.7, 128.4, 128.0, 127.0, 126.0, 125.3, 124.9, 122.4, 120.5, 120.0, 118.2, 115.2, 112.4, 112.2, 65.6, 33.3. HRMS (ESI) m/z calcd for $C_{27}H_{23}N_2O_3$ $[M + H]^+$: 423.1703, found: 423.1691.

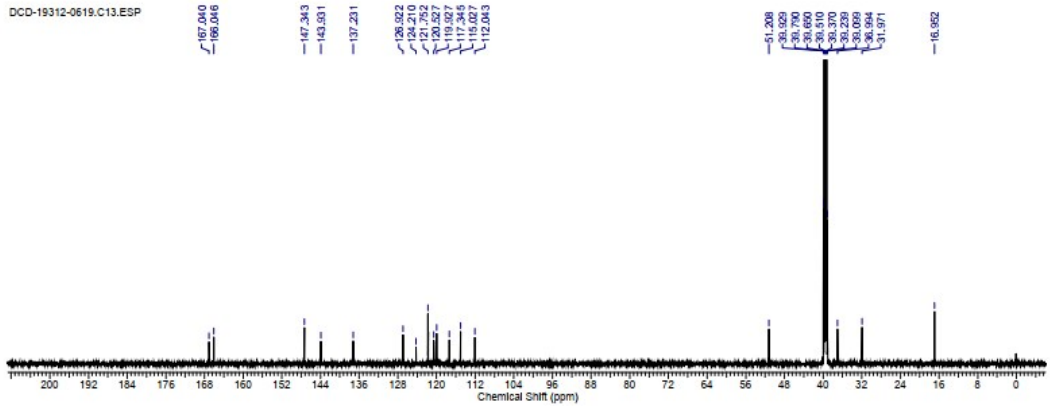
3. NMR and HRMS of compounds 4a-r, 8-12, 1a-c, calothrixin B.



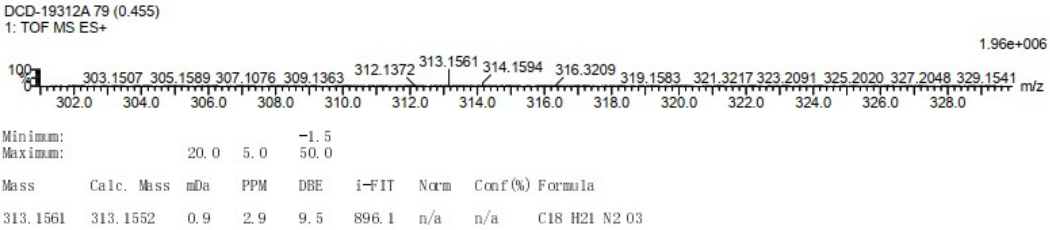


5a

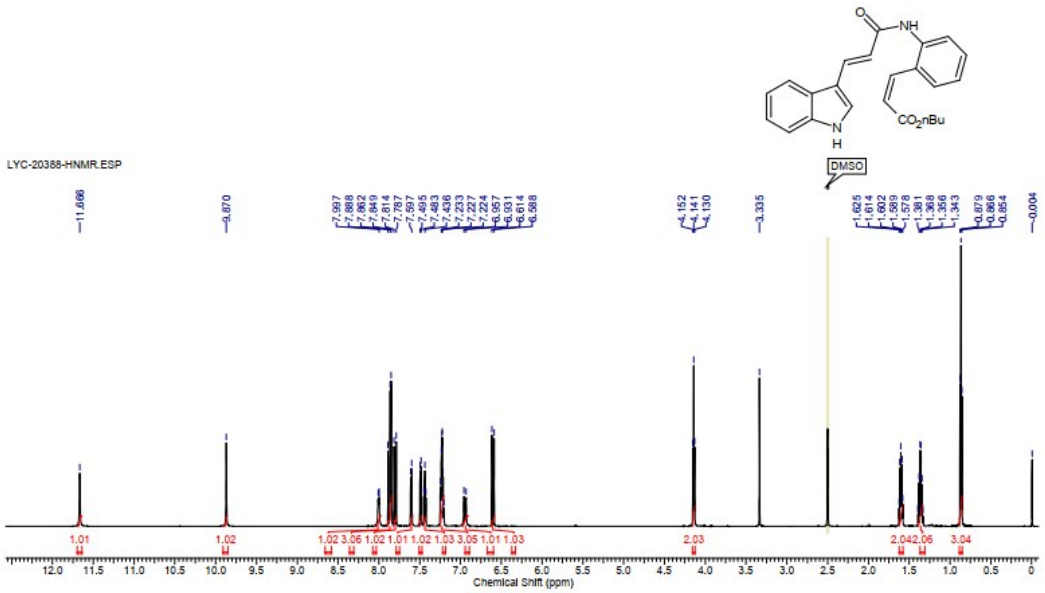




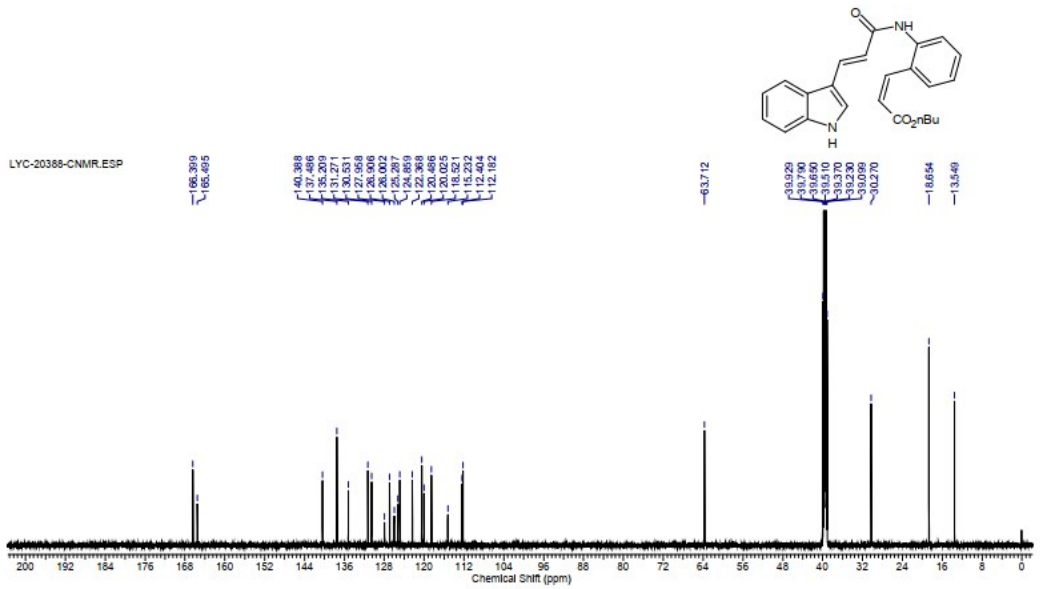
5a



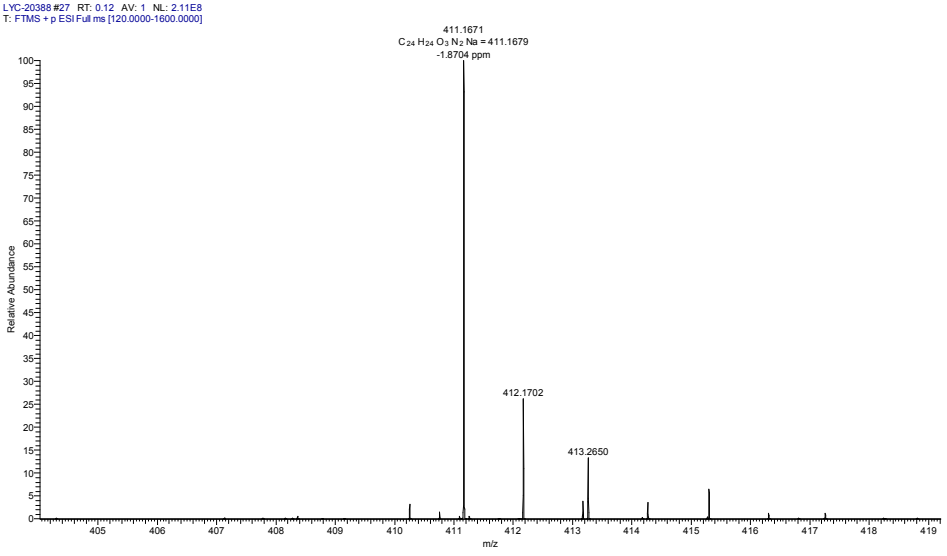
Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
313.1561	313.1552	0.9	2.9	9.5	896.1	n/a	n/a	C18 H21 N2 O3

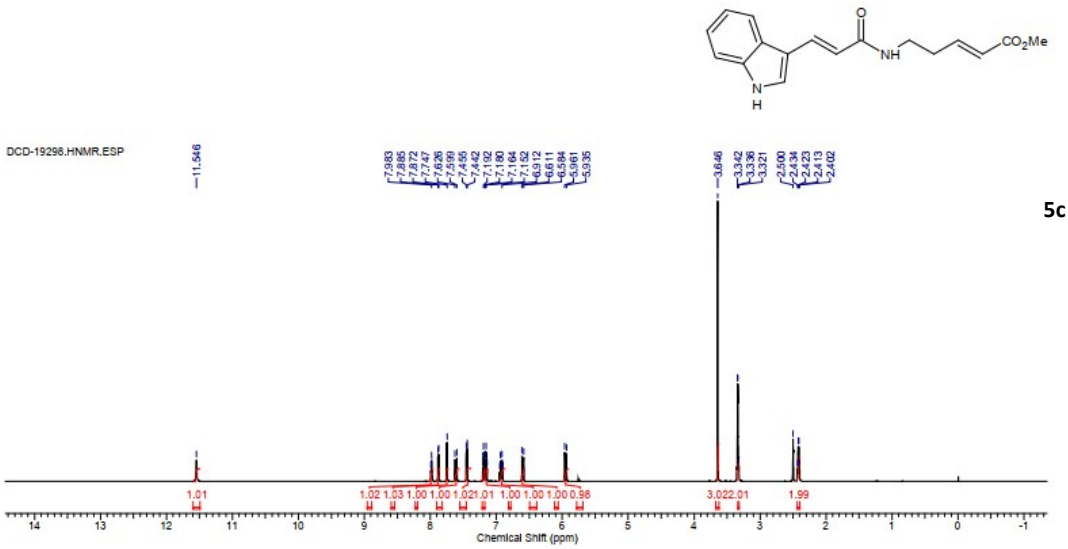

LYC-20388-HNMR.ESP
DMSO
Chemical Shift (ppm)

5b

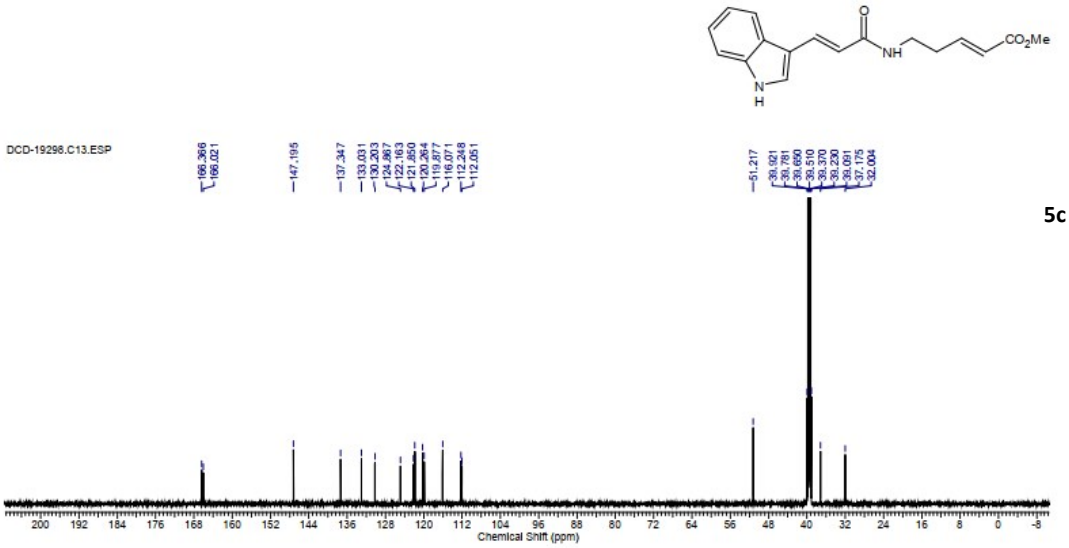

LYC-20388-CNMR.ESP
Chemical Shift (ppm)

5b


LYC-20388 #27 RT: 0.12 AV: 1 NL: 2.11E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
411.1671
C24 H24 O3 N2 Na = 411.1679
-1.8704 ppm
412.1702
413.2650
Relative Abundance
m/z



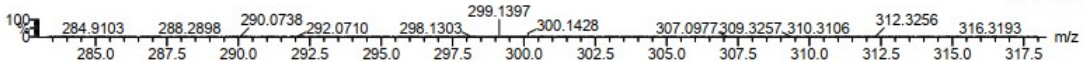
5c



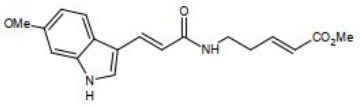
5c

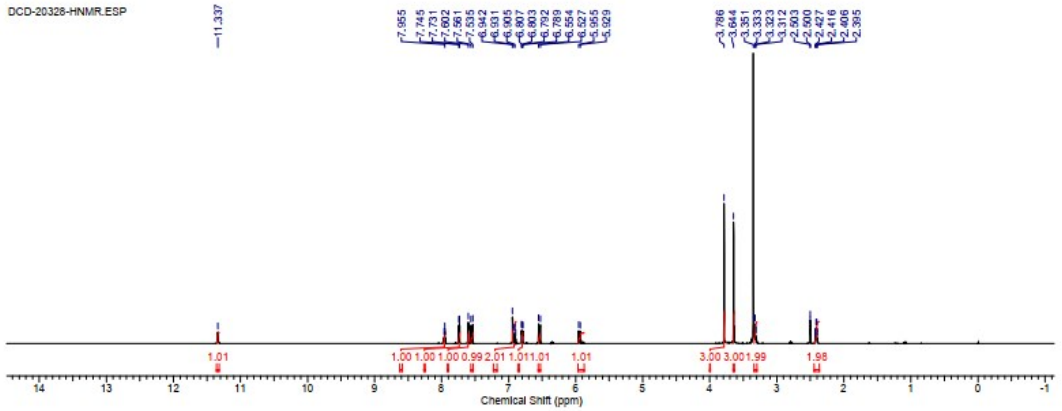
DCD-19298 100 (0.573)
1: TOF MS ES+

4.34e+006

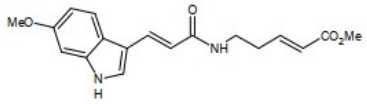


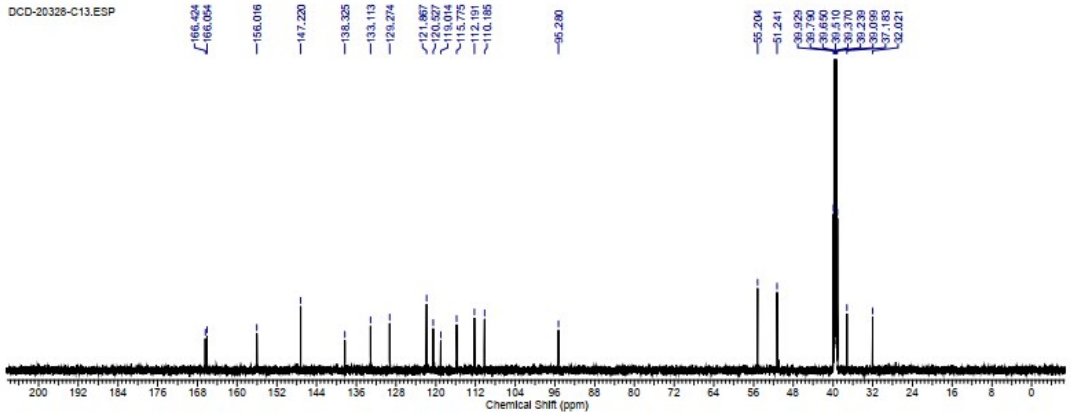
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
299.1397	299.1396	0.1	0.3	9.5	1247.8	n/a	n/a	C17 H19 N2 O3





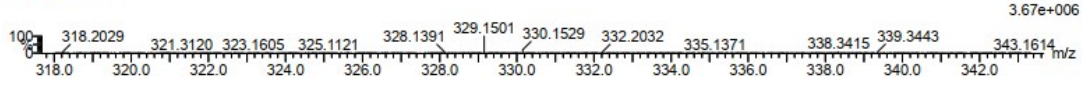
5d



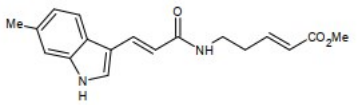


5d

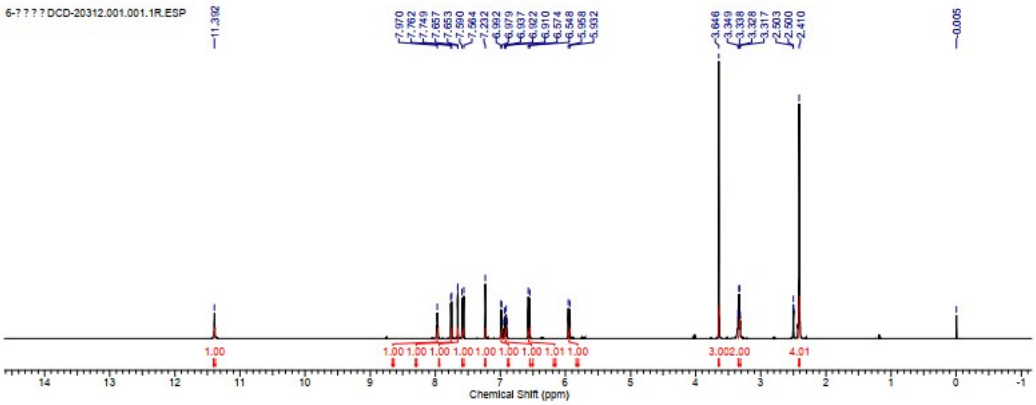
DCD-20328 96 (0.552)
1: TOF MS ES+

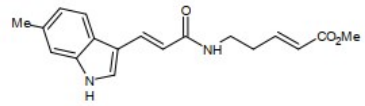


Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
329.1501	329.1501	0.0	0.0	9.5	1167.8	n/a	n/a	C18 H21 N2 O4

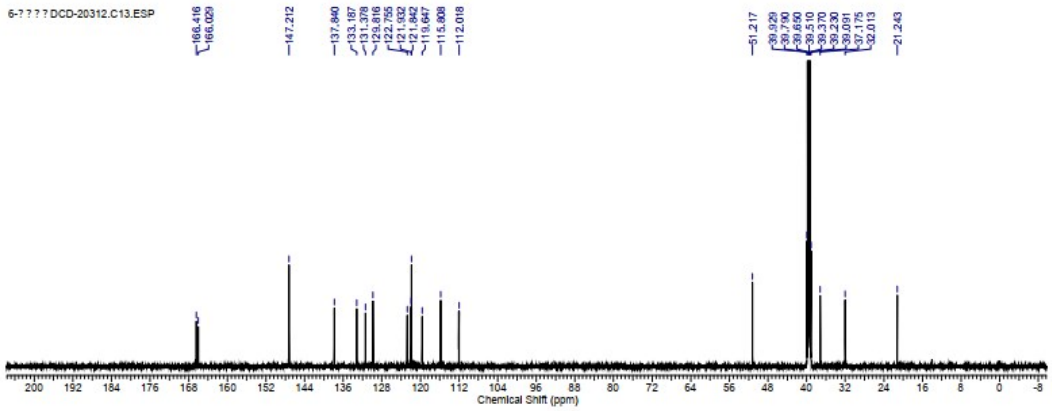


5e



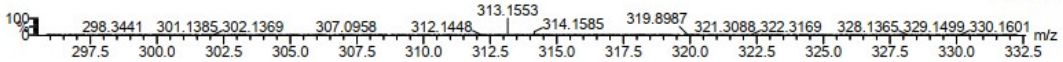


5e

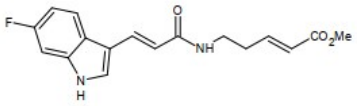


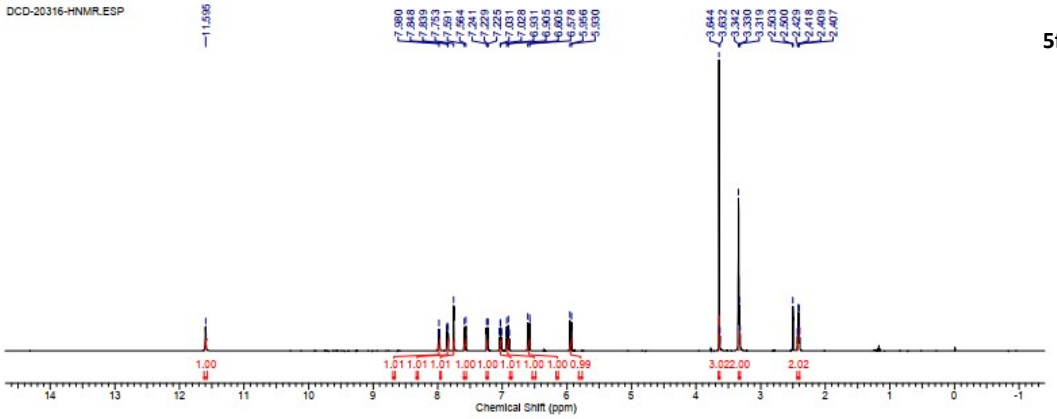
DCD-20312 114 (0.647)
1: TOF MS ES+

2.90e+006

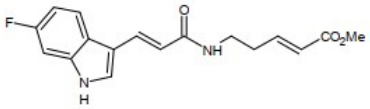


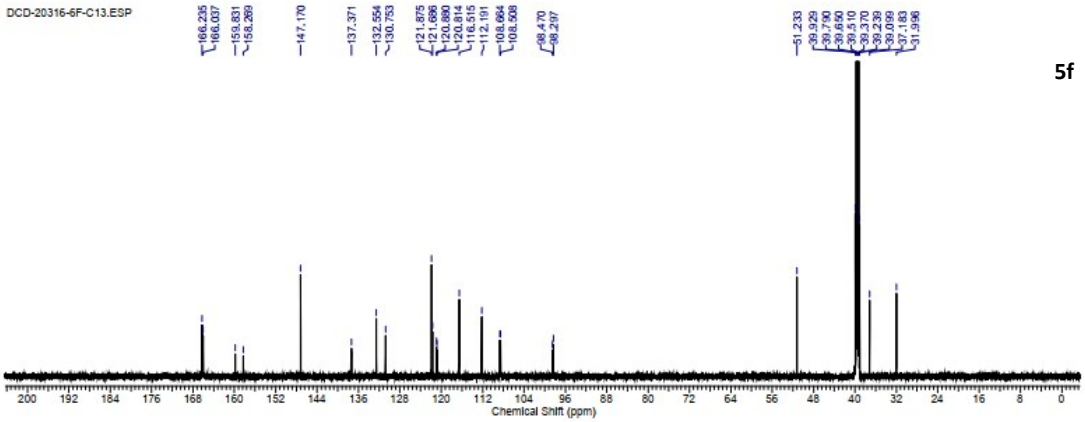
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
313.1553	313.1552	0.1	0.3	9.5	1089.6	n/a	n/a	C18 H21 N2 O3


F
O
NH
CO2Me
N
H

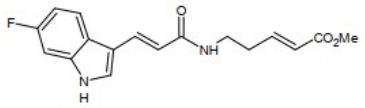

DCD-20316-HNMR.ESP
11.595
7.880
7.848
7.839
7.753
7.591
7.564
7.241
7.229
7.225
7.051
7.028
6.931
6.905
6.605
6.578
5.956
5.930
3.644
3.632
3.342
3.339
3.319
2.503
2.500
2.429
2.418
2.409
2.407
1.00
1.01 1.01 1.01 1.00 1.00 1.01 1.00 1.00 0.99
3.022.00
2.02
Chemical Shift (ppm)

5f

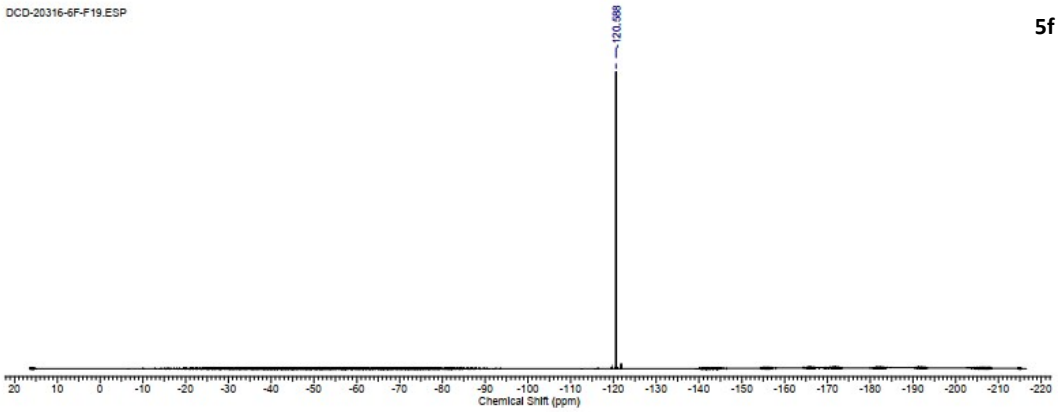

F
O
NH
CO2Me
N
H


DCD-20316-6F-C13.ESP
166.235
166.037
159.831
158.269
147.170
137.371
132.554
130.753
121.875
121.686
120.880
120.814
116.515
112.191
108.664
108.508
98.470
98.297
51.233
39.929
39.790
39.650
39.510
39.370
39.239
39.099
37.183
31.996
Chemical Shift (ppm)

5f

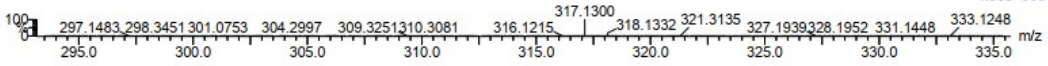


5f

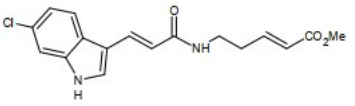


DCD-20316 100 (0.573)
1: TOF MS ES+

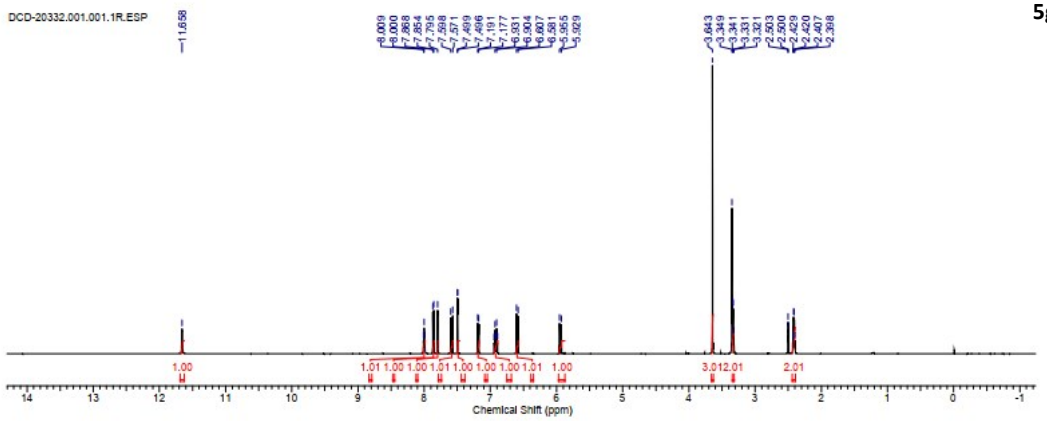
4.39e+006

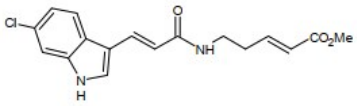


Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
317.1300	317.1301	-0.1	-0.3	9.5	1149.4	n/a	n/a	C17 H18 N2 O3 F

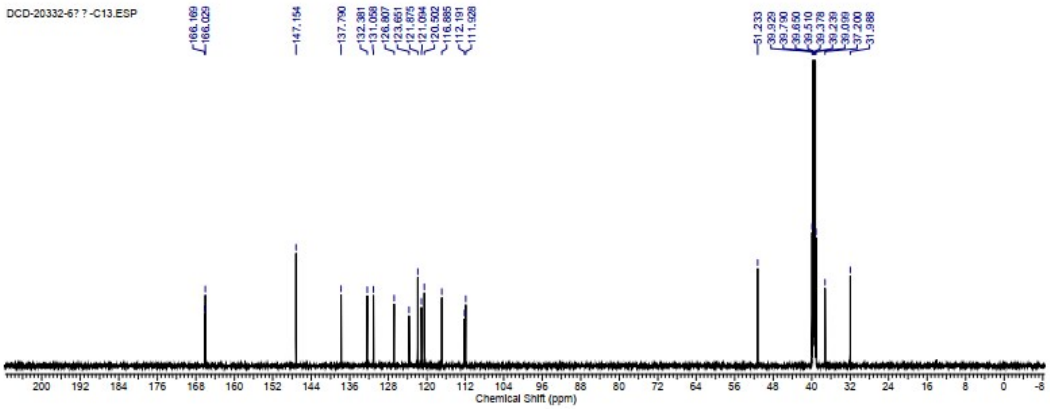


5g

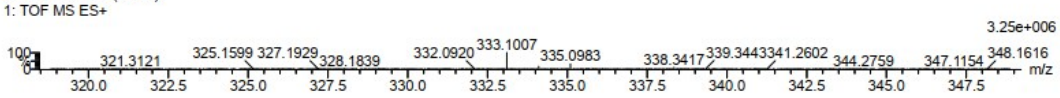




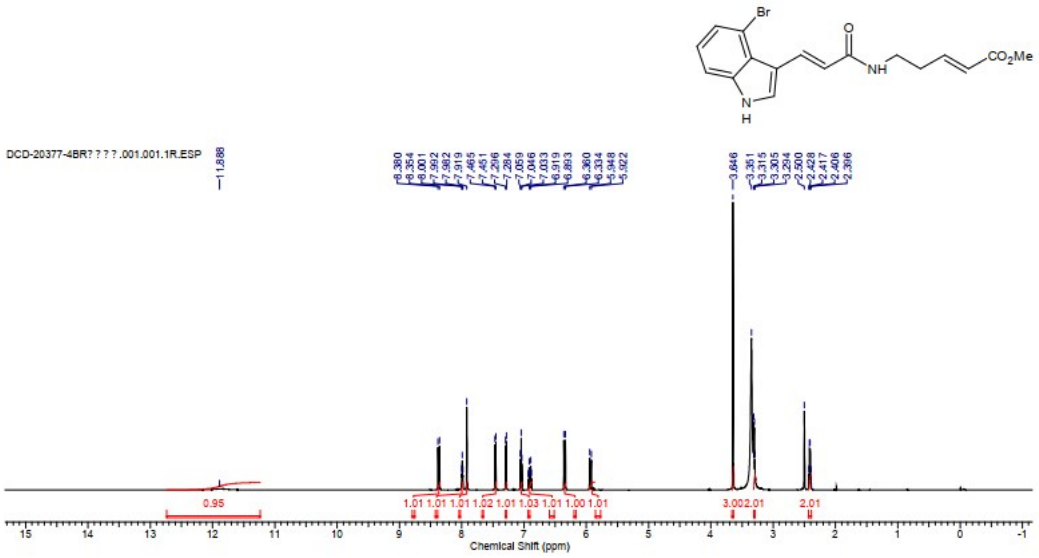
5g



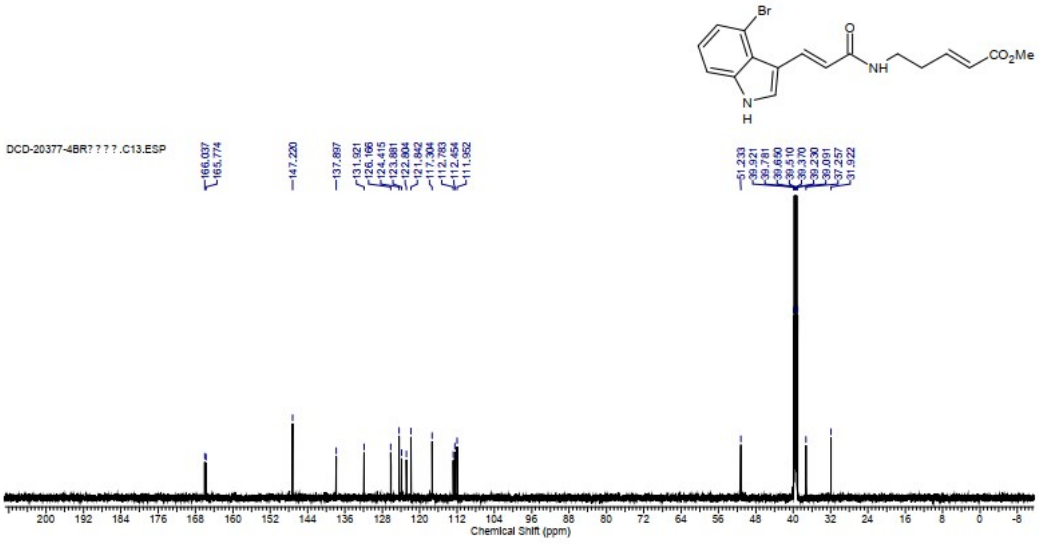
DCD-20332 105 (0.600)
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
333.1007	333.1006	0.1	0.3	9.5	1077.6	n/a	n/a	C17 H18 N2 O3 Cl

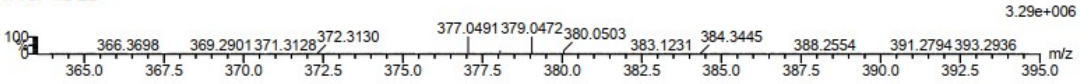


5h

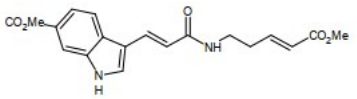


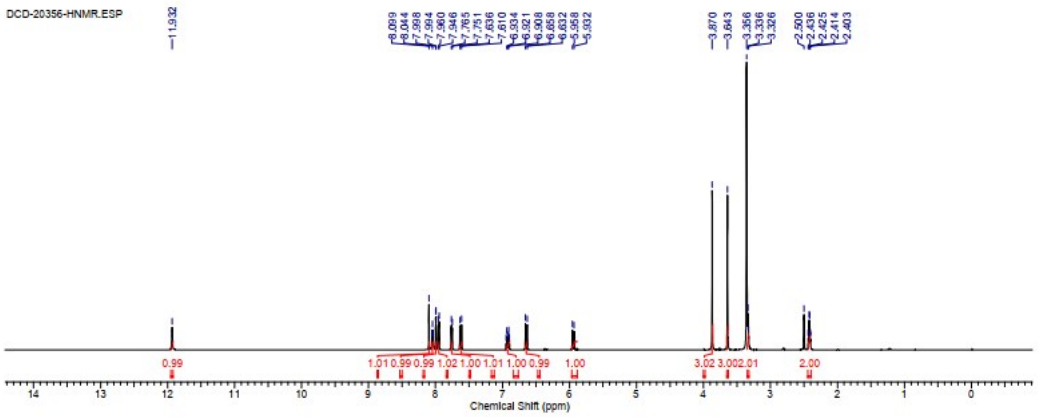
5h

DCD-20377 97 (0.558)
1: TOF MS ES+

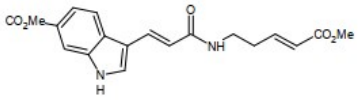


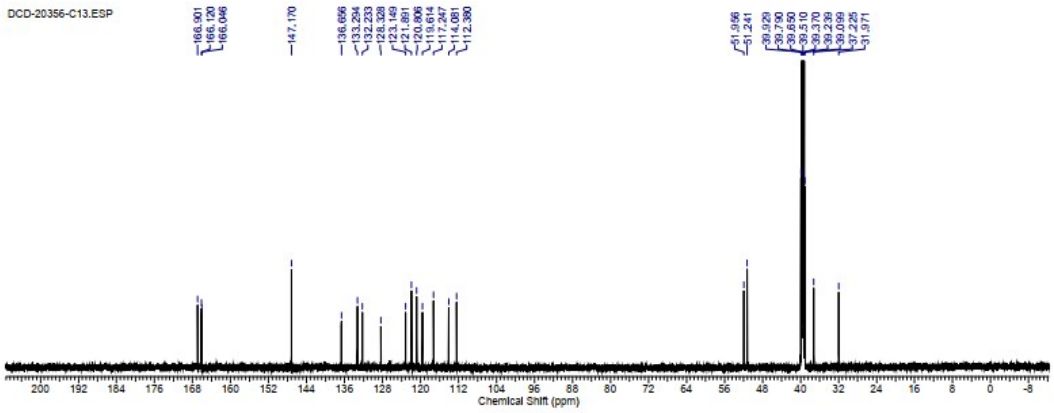
Minimum:				-1.5			
Maximum:		20.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%) Formula
377.0491	377.0501	-1.0	-2.7	9.5	1118.9	n/a	n/a C17 H18 N2 O3 Br





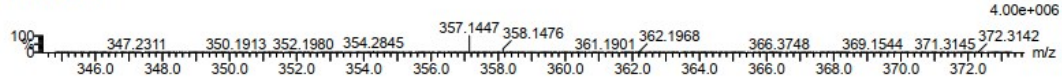
5i



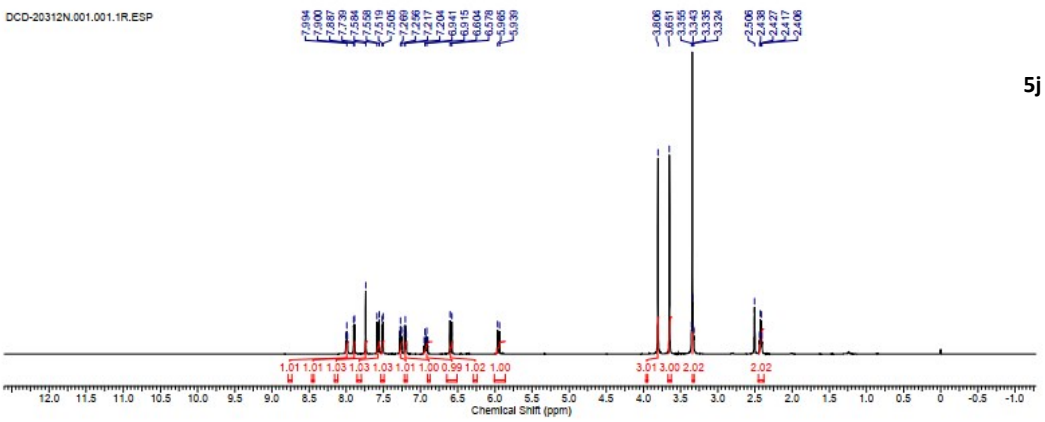


5i

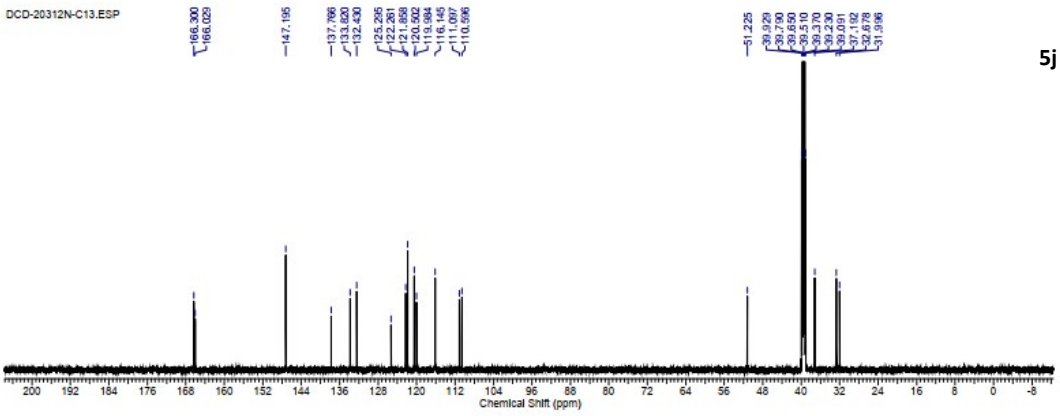
DCD-20356 103 (0.589)
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
357.1447	357.1450	-0.3	-0.8	10.5	1080.0	n/a	n/a	C19 H21 N2 O5

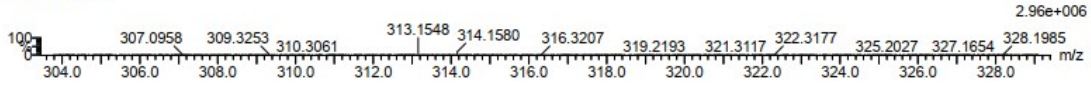


5j

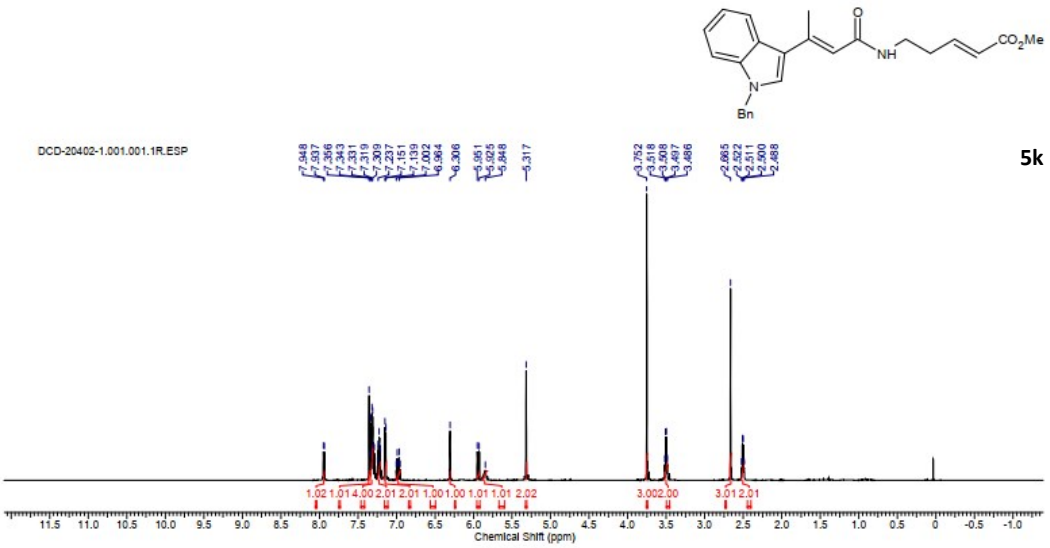


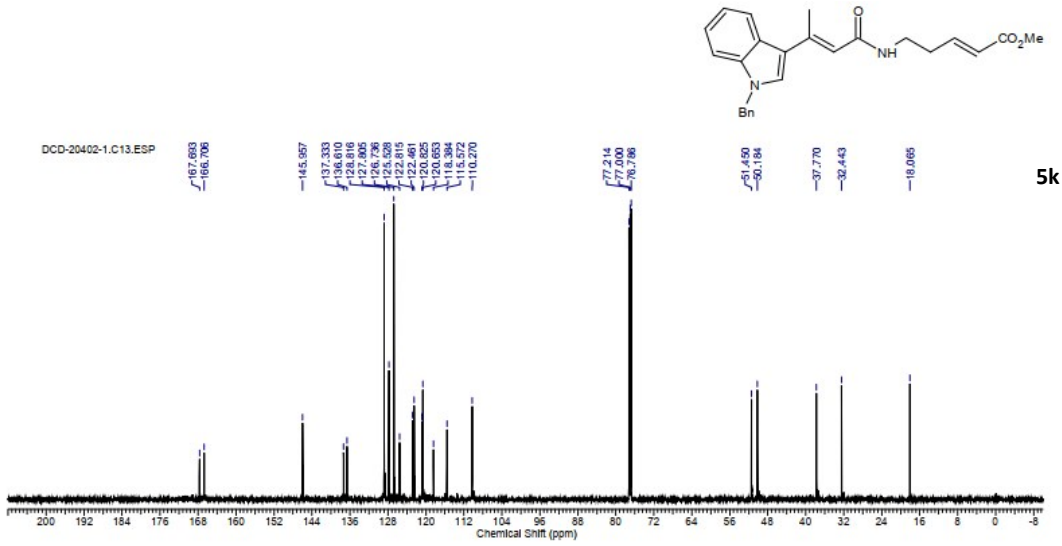
5j

DCD-20312N 101 (0.579)
1: TOF MS ES+



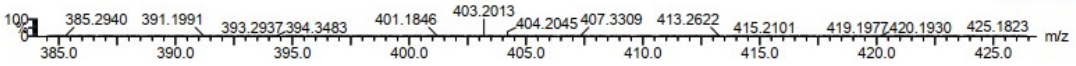
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
313.1548	313.1552	-0.4	-1.3	9.5	1064.6	n/a	n/a	C18 H21 N2 O3



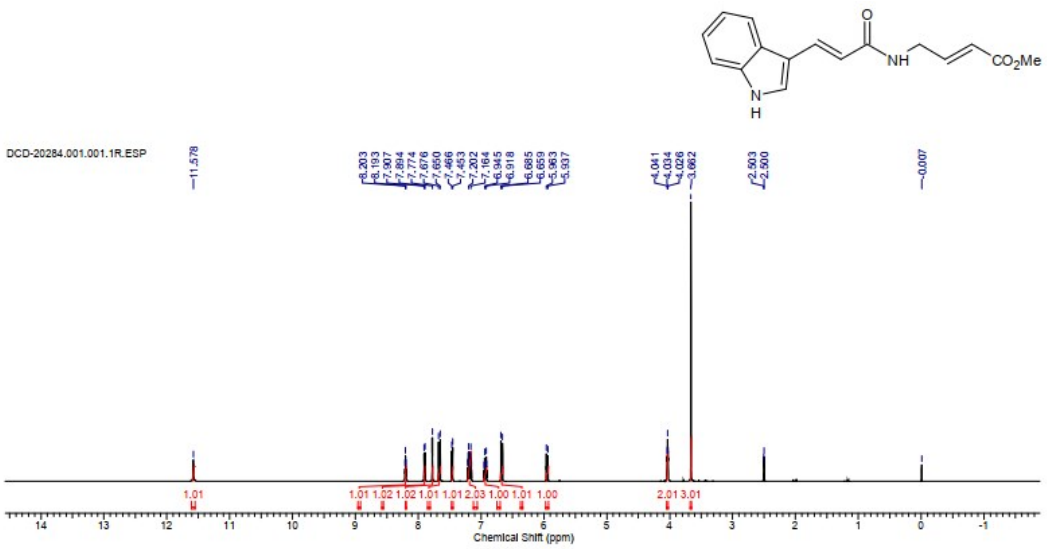


DCD-20402-1 121 (0.691)
1: TOF MS ES+

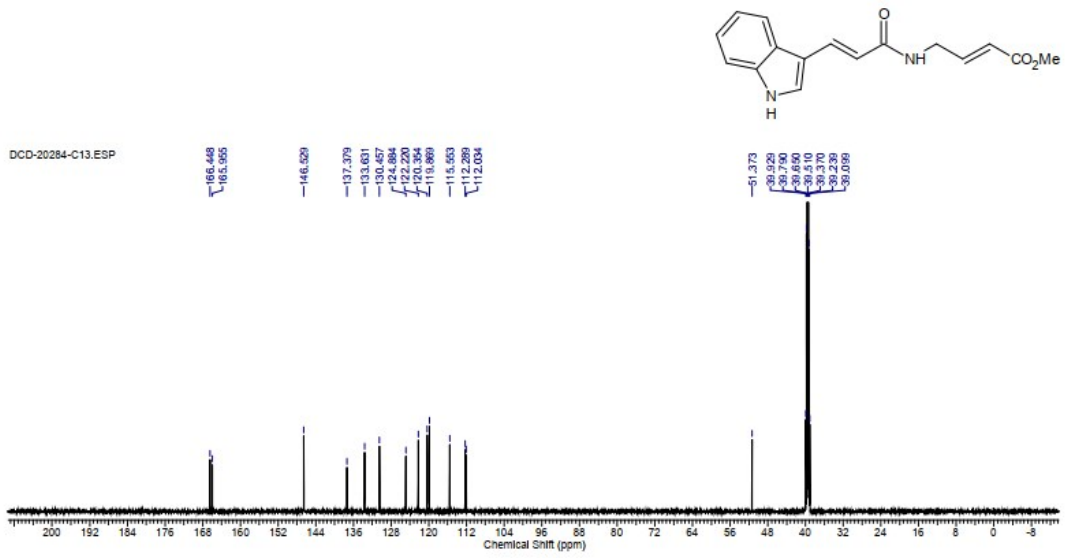
2.78e+006



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
403.2013	403.2022	-0.9	-2.2	13.5	1005.6	n/a	n/a	C25 H27 N2 O3



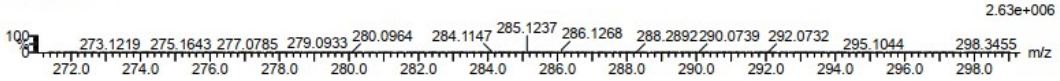
5l



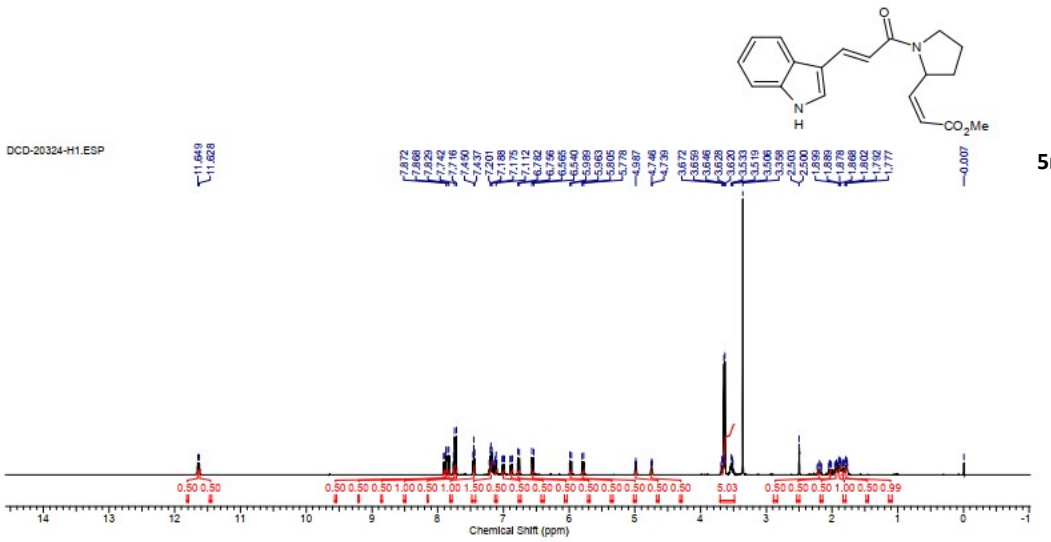
5l

DCD-19284 93 (0.537)
1: TOF MS ES+

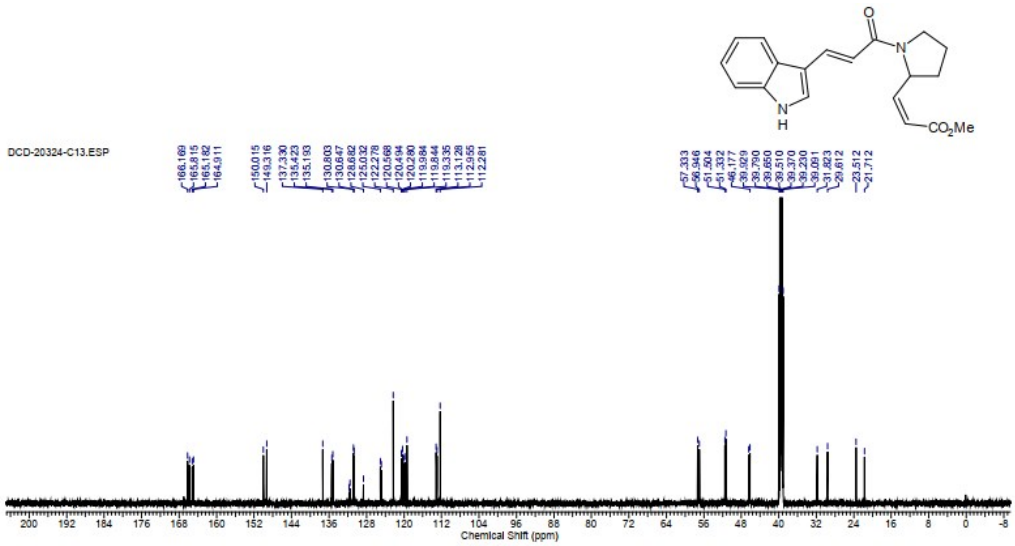
2.63e+006



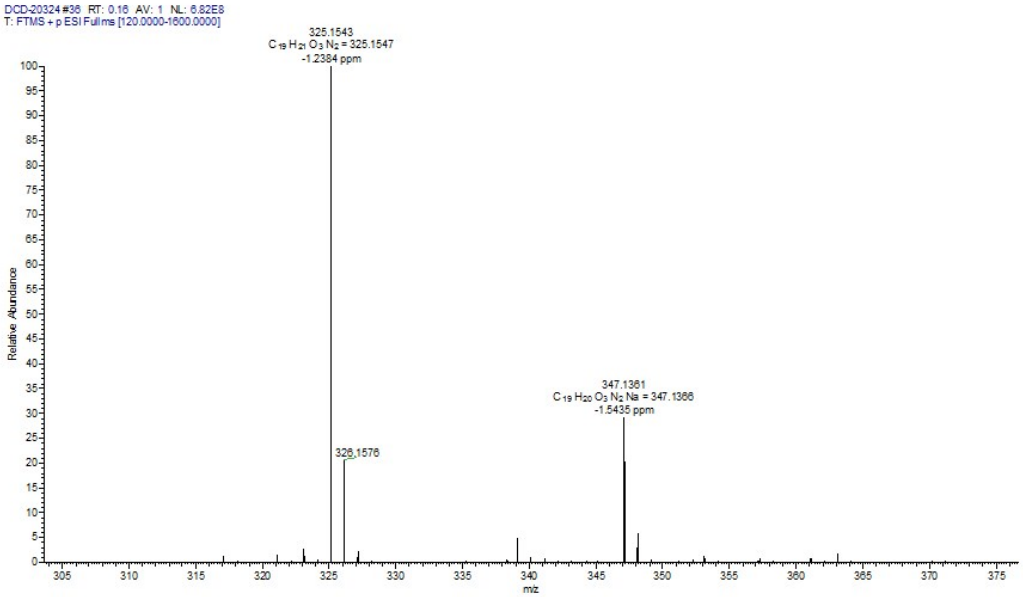
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
285.1237	285.1239	-0.2	-0.7	9.5	1224.1	n/a	n/a	C16 H17 N2 O3

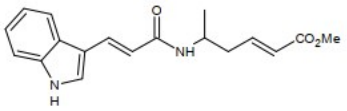

DCD-20324-H1.ESP
Chemical Shift (ppm)

5m

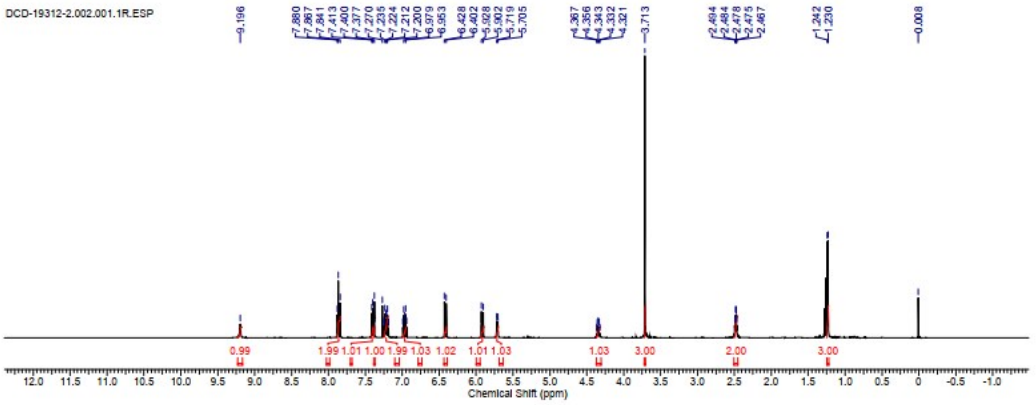

DCD-20324-C13.ESP
Chemical Shift (ppm)

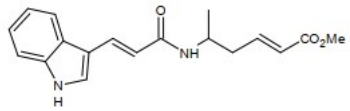
5m


DCD-20324 #36 RT: 0.16 AV: 1 NL: 6.82E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
325.1543
C19 H21 O3 N2 = 325.1547
-1.2384 ppm
326.1576
347.1361
C19 H20 O3 N2 Na = 347.1366
-1.5435 ppm
Relative Abundance
m/z

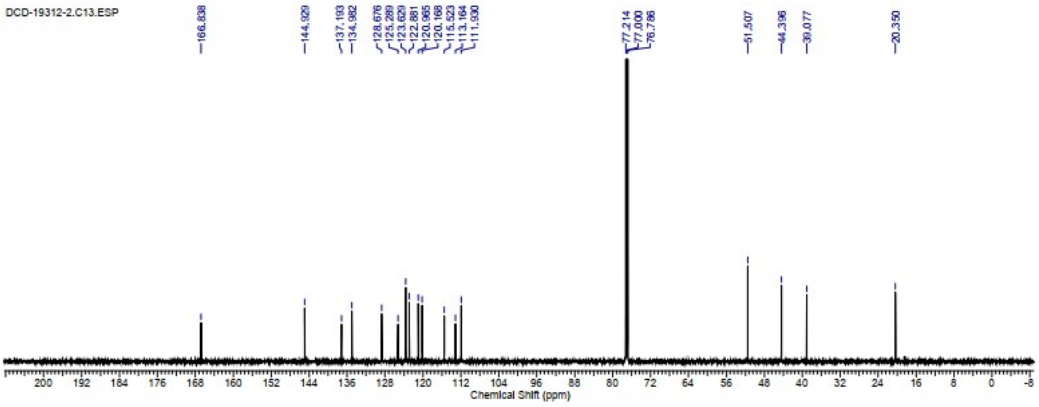


5n

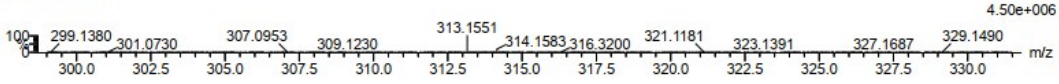




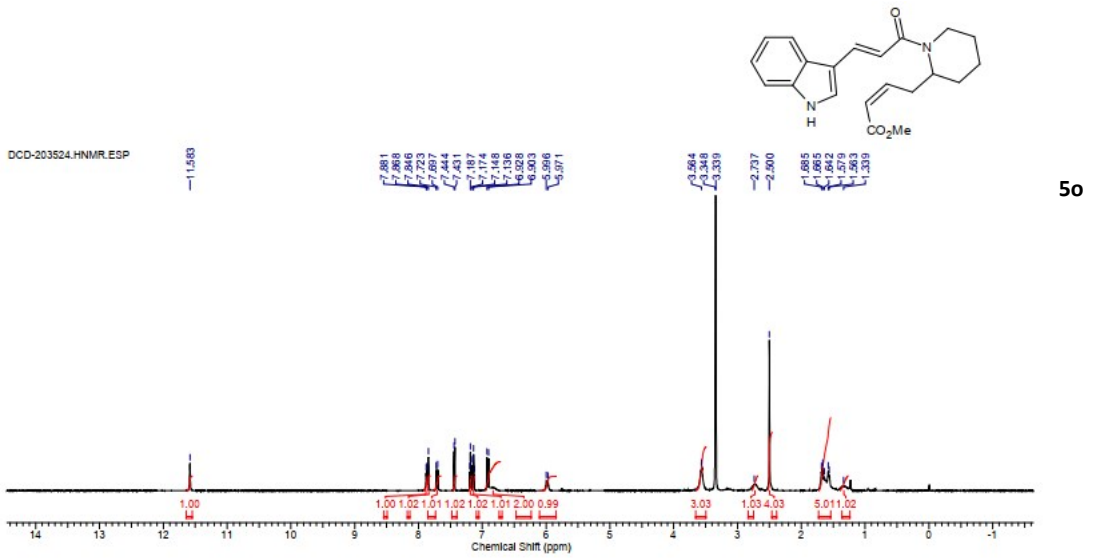
5n



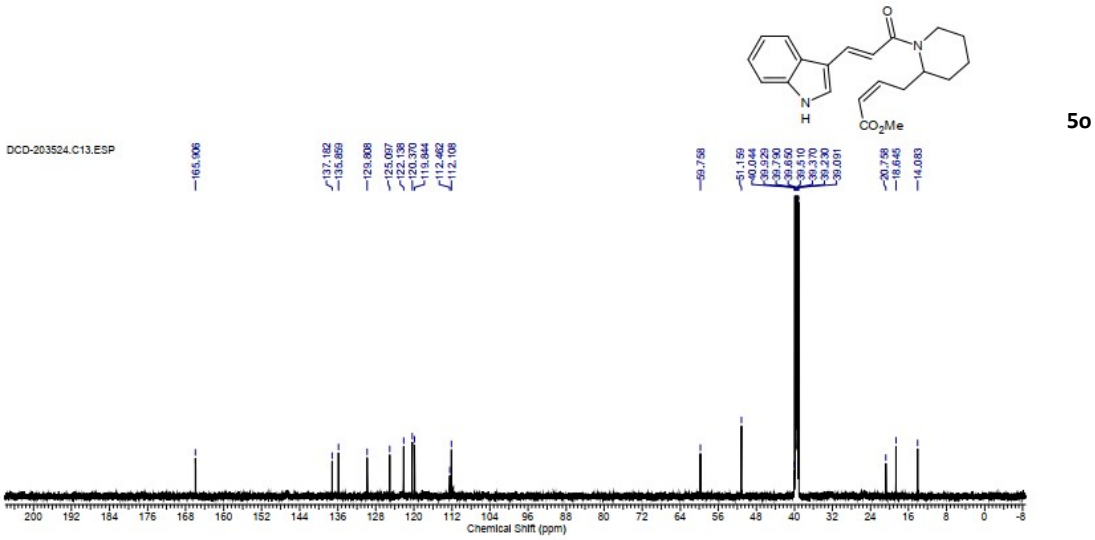
DCD-19312-2 108 (0.615)
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
313.1551	313.1552	-0.1	-0.3	9.5	1149.8	n/a	n/a	C18 H21 N2 O3



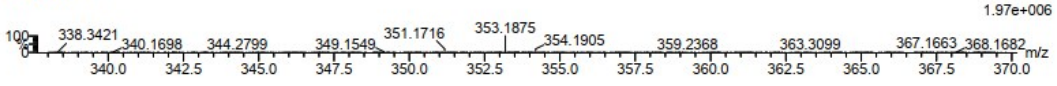
5o



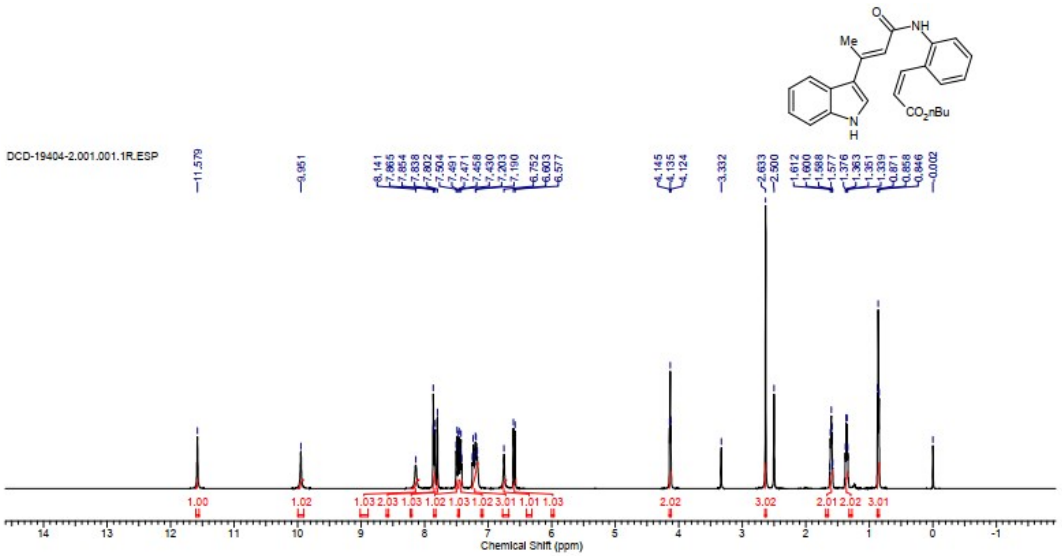
5o

DCD-203524 94 (0.542)
1: TOF MS ES+

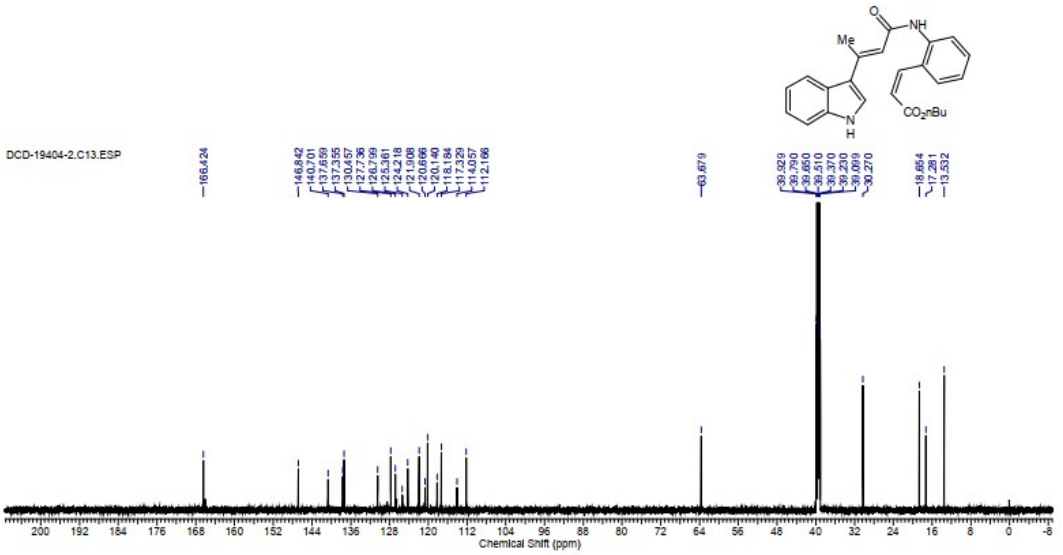
1.97e+006



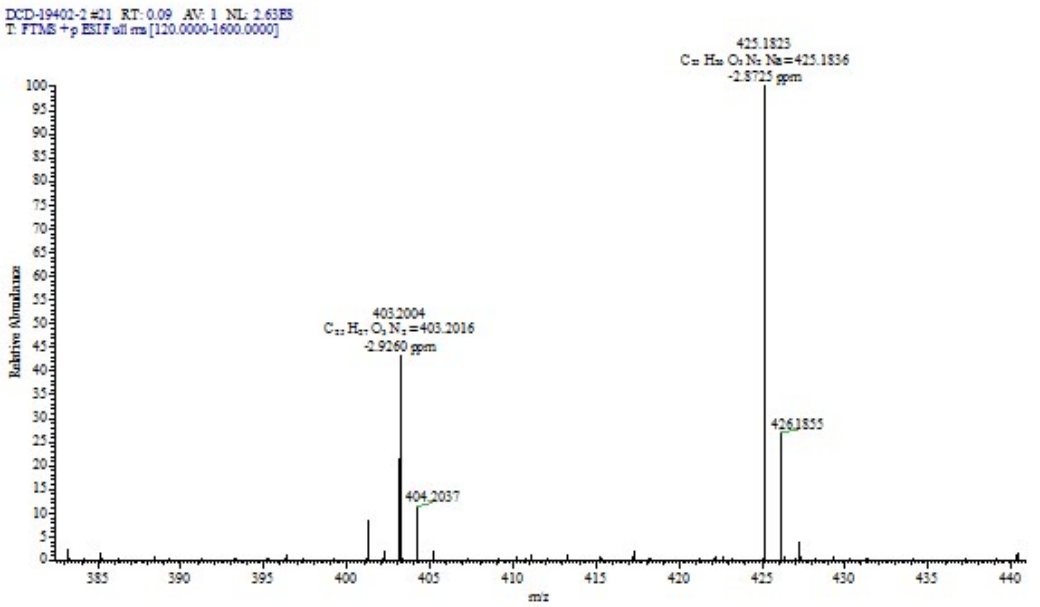
Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
353.1875	353.1865	1.0	2.8	10.5	897.0	n/a	n/a	C21 H25 N2 O3

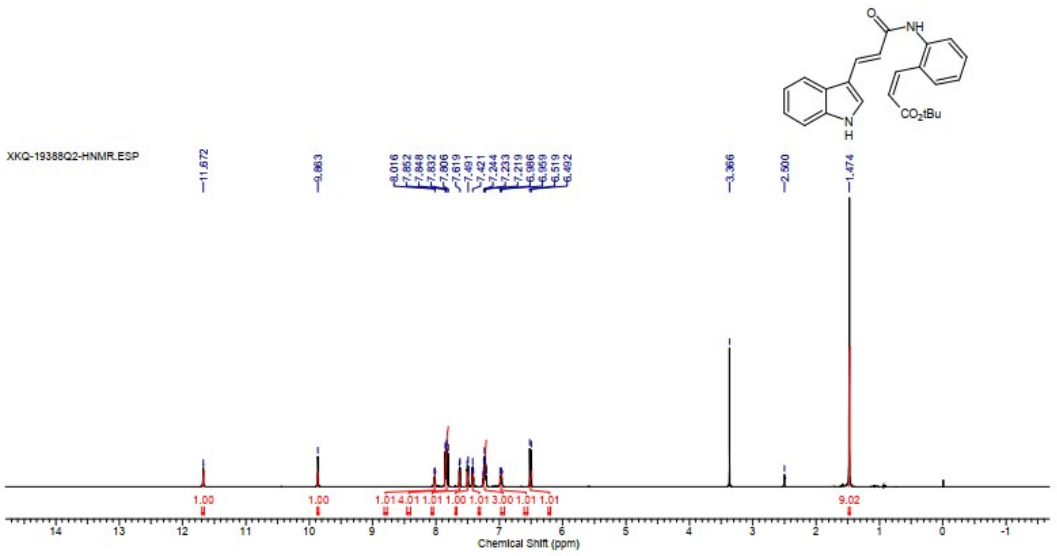

DCD-19404-2.001.001.1R.ESP
Chemical Shift (ppm)

5p

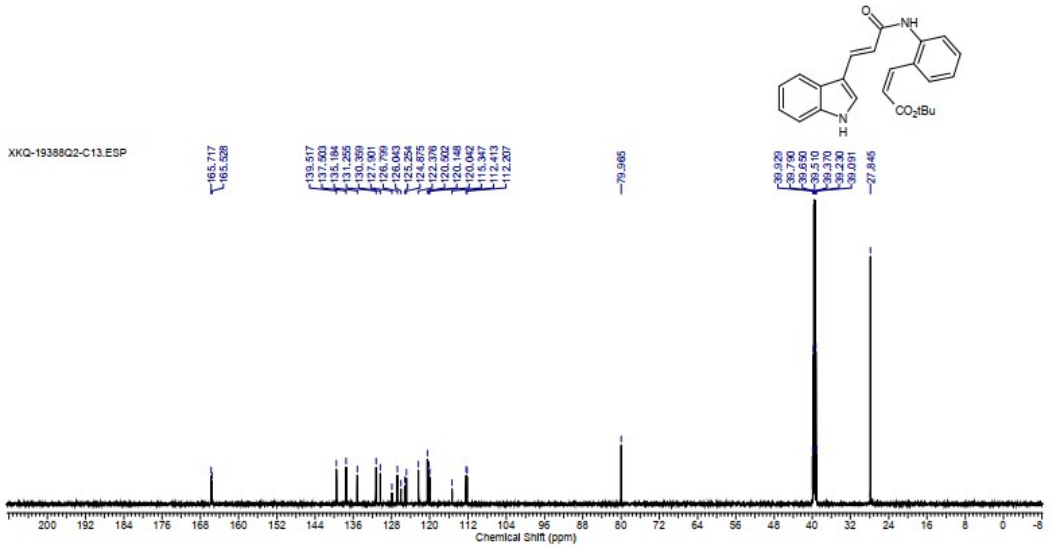

DCD-19404-2.C13.ESP
Chemical Shift (ppm)

5p

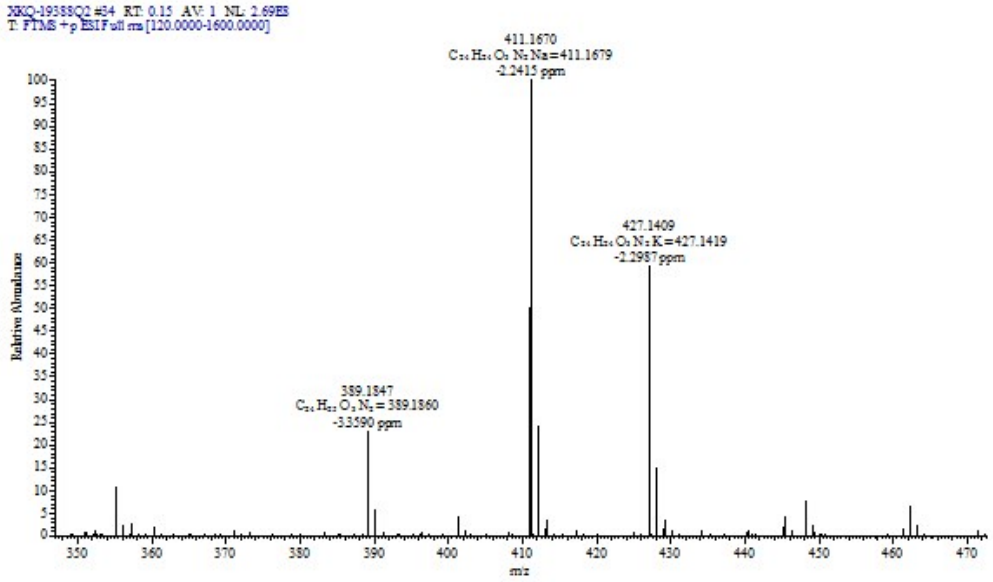

DCD-19402-2 #21 RT: 0.09 AV: 1 NL: 2.63E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
425.1823
C31 H30 O3 N2 Na=425.1836
-2.8725 ppm
403.2004
C31 H27 O3 N2 =403.2016
-2.9260 ppm
426.1855
404.2037
Relative Abundance
m/z

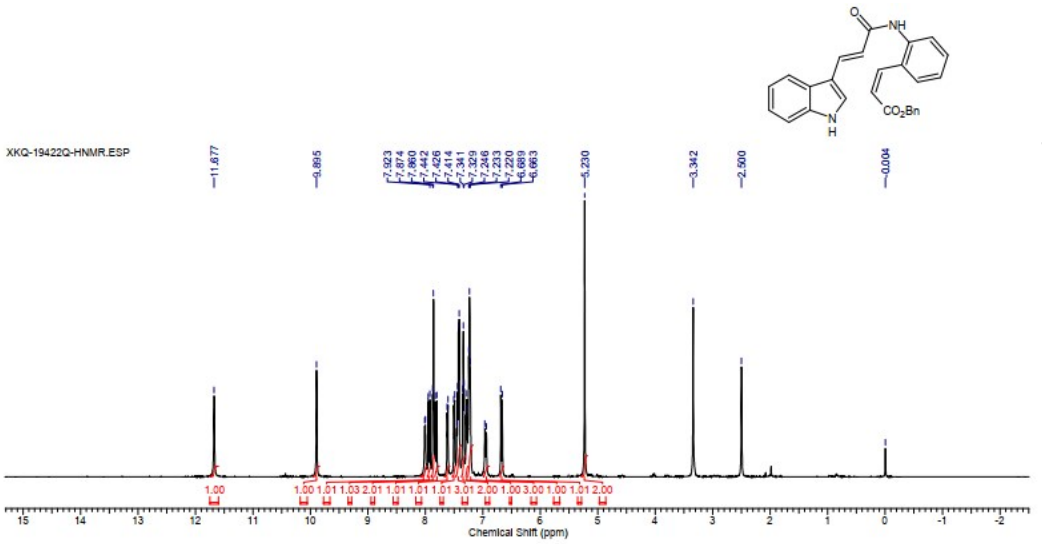

XKQ-19388Q2-HNMR.ESP
Chemical Shift (ppm)

5q

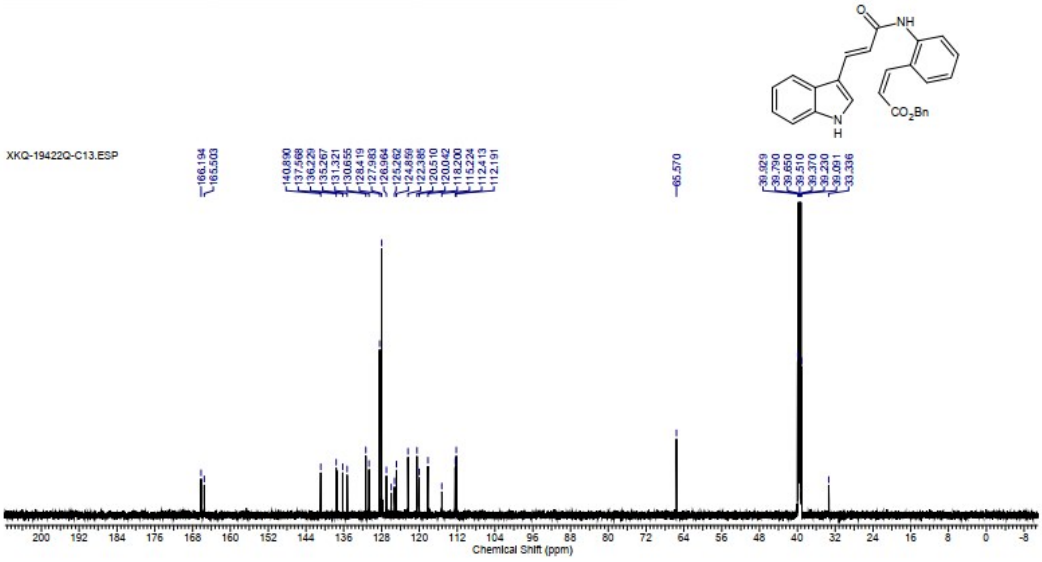

XKQ-19388Q2-C13.ESP
Chemical Shift (ppm)

5q

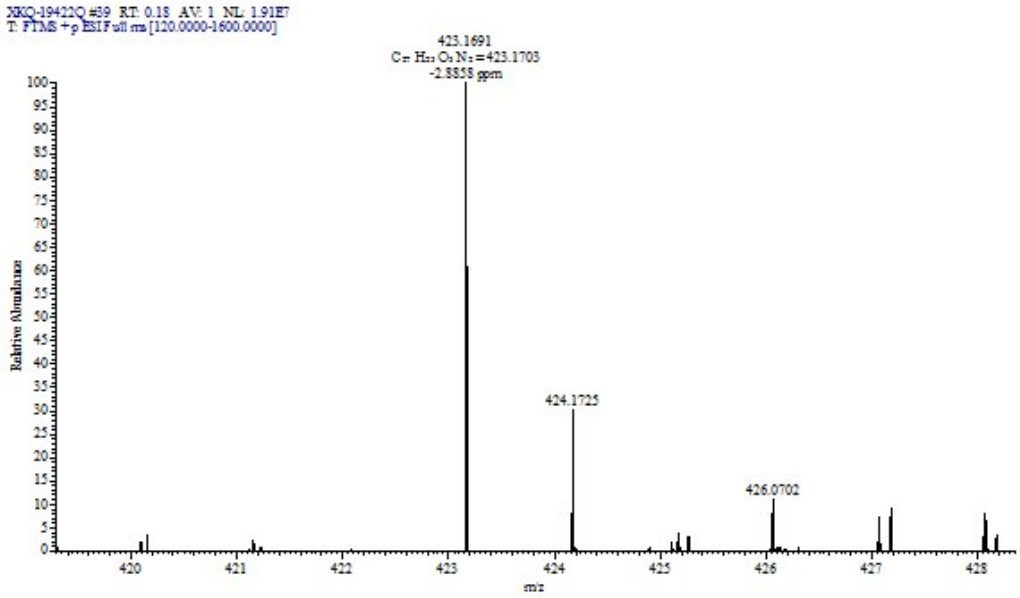

XKQ-19388Q2 #64 RT: 0.15 AV: 1 NL: 2.69E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
411.1670
C24 H24 O3 N2 Na = 411.1679
-2.2415 ppm
427.1409
C24 H24 O3 N2 K = 427.1419
-2.2987 ppm
389.1847
C24 H23 O3 N2 = 389.1860
-3.3590 ppm
Relative Abundance
m/z

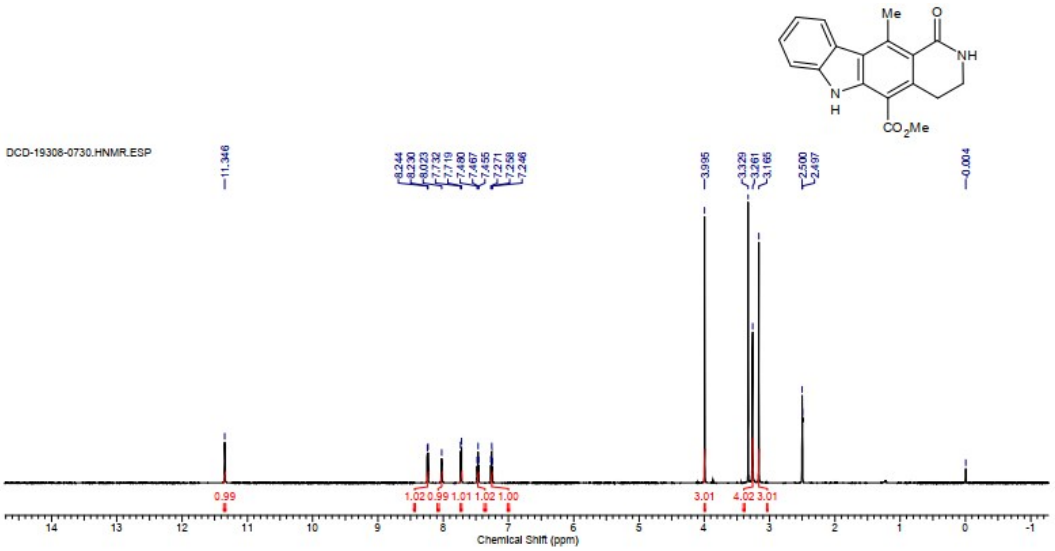

XKQ-19422Q-HNMR.ESP
11.677
8.895
7.923
7.874
7.860
7.422
7.426
7.414
7.341
7.329
7.246
7.233
7.220
6.689
6.663
5.230
3.342
2.500
0.004
1.00
1.00 1.01 1.03 2.01 1.01 1.01 1.01 3.01 2.00 1.00 3.00 1.00 1.01 2.00
Chemical Shift (ppm)

5r

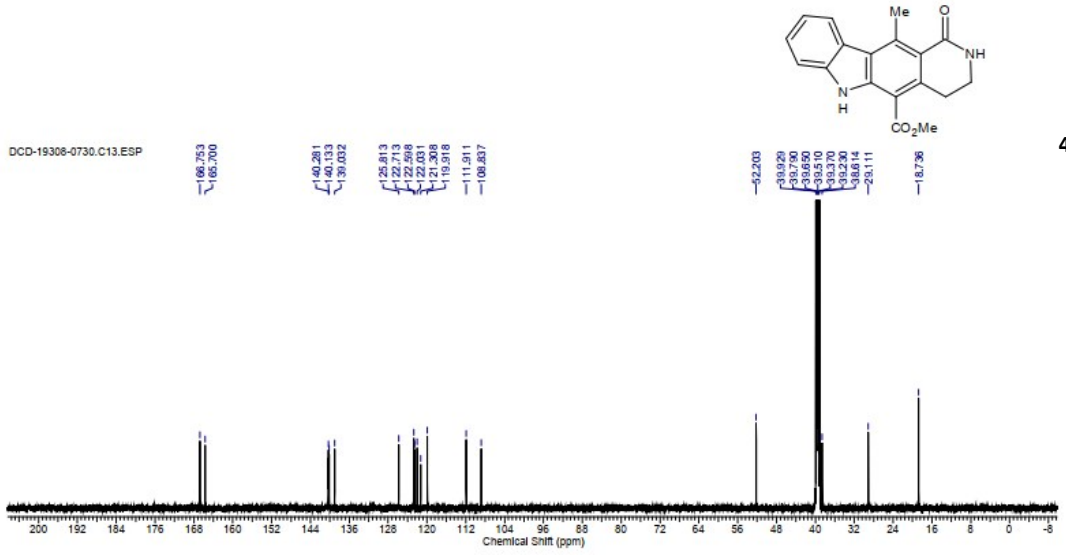

XKQ-19422Q-C13.ESP
166.194
165.503
140.890
137.588
136.229
135.267
131.321
130.655
128.419
127.983
126.964
126.092
124.859
122.385
120.510
120.042
118.200
115.224
112.413
112.191
65.570
39.929
39.780
39.650
39.510
39.370
39.230
39.091
33.336
Chemical Shift (ppm)

5r


XKQ-19422Q #39 RT: 0.18 AV: 1 NL: 1.91E7
T: FTMS + p ESI Full ms [120.0000-1600.0000]
423.1691
C27 H23 O3 N2 = 423.1703
-2.8858 ppm
424.1725
426.0702
Relative Abundance
m/z

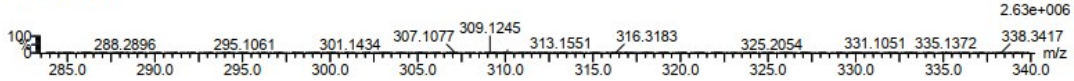


4a

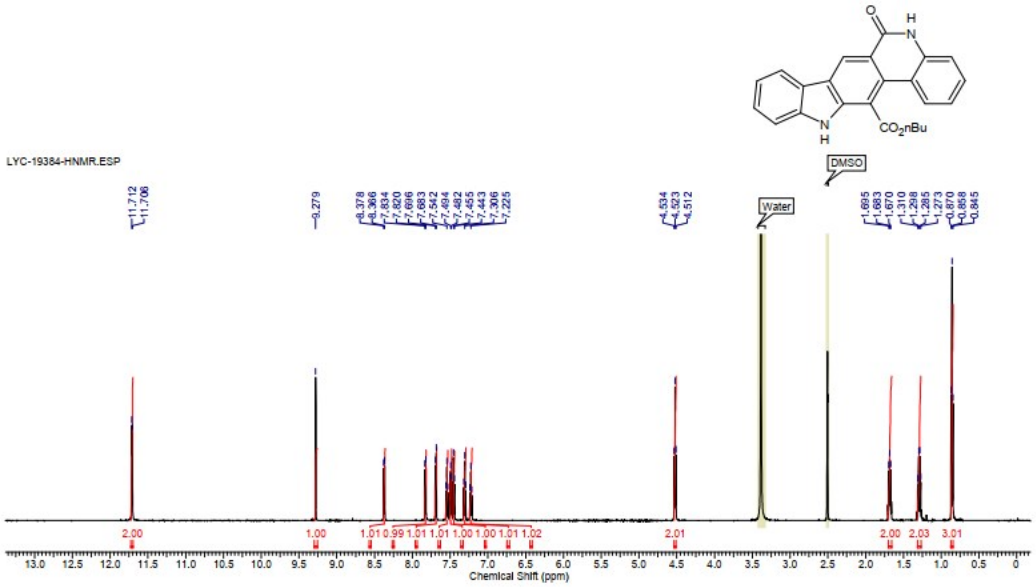


4a

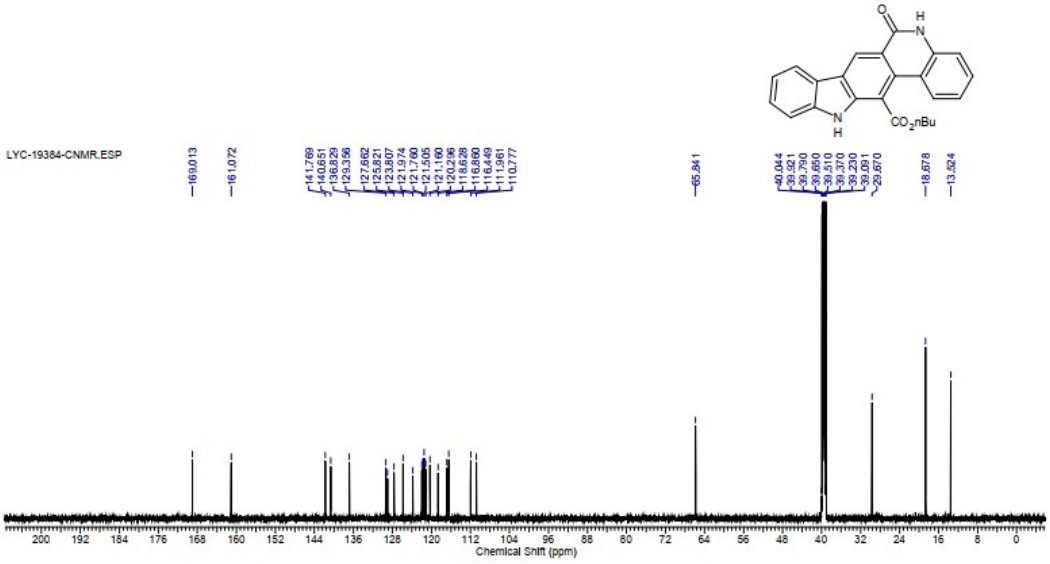
DCD-19308 80 (0.460)
1: TOF MS ES+



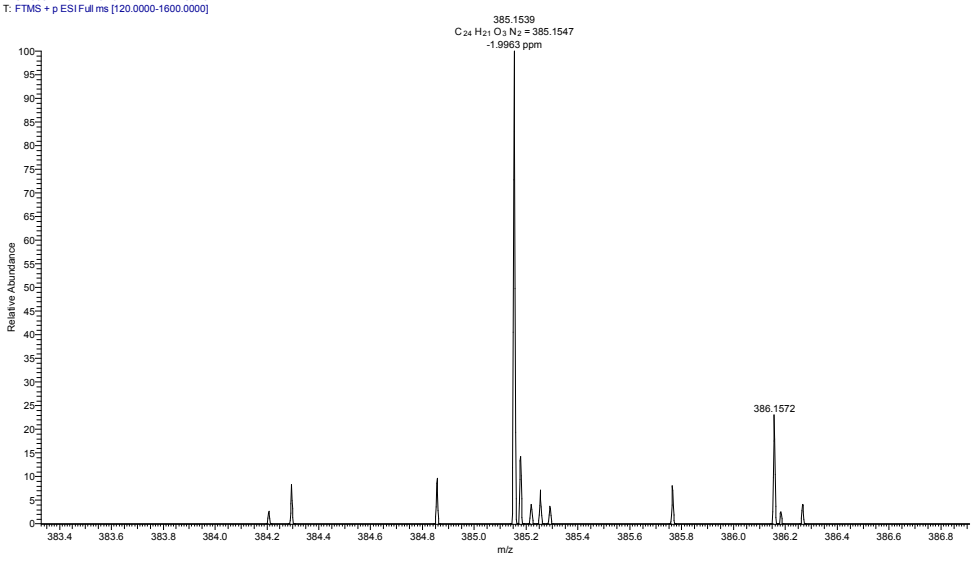
Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
309.1245	309.1239	0.6	1.9	11.5	968.5	n/a	n/a	C18 H17 N2 O3

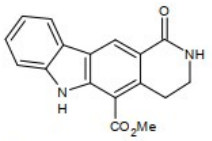

LYC-19384-HNMR.ESP
DMSO
Water
Chemical Shift (ppm)

4b

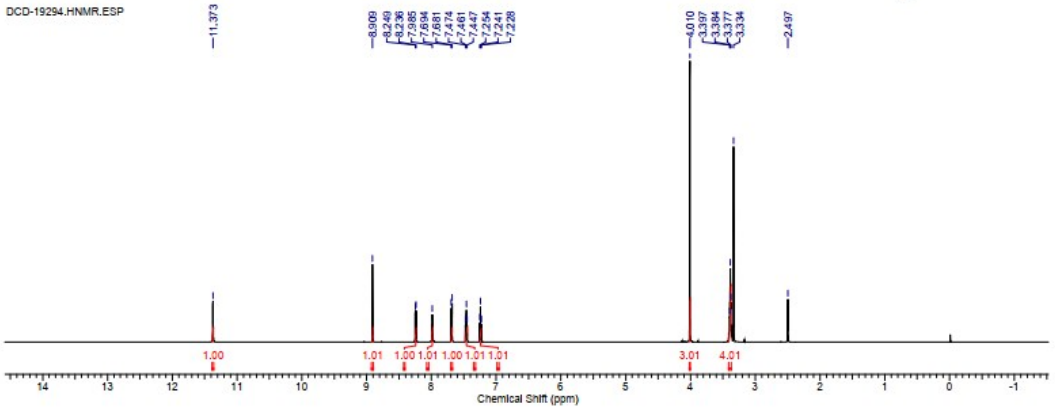

LYC-19384-CNMR.ESP
Chemical Shift (ppm)

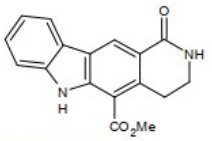
4b


LYC-19384 #45 RT: 0.20 AV: 1 NL: 2.30E6
T: FTMS + p ESI Full ms [120.0000-1600.0000]
385.1539
C24 H21 O3 N2 = 385.1547
-1.9963 ppm
386.1572
Relative Abundance
m/z

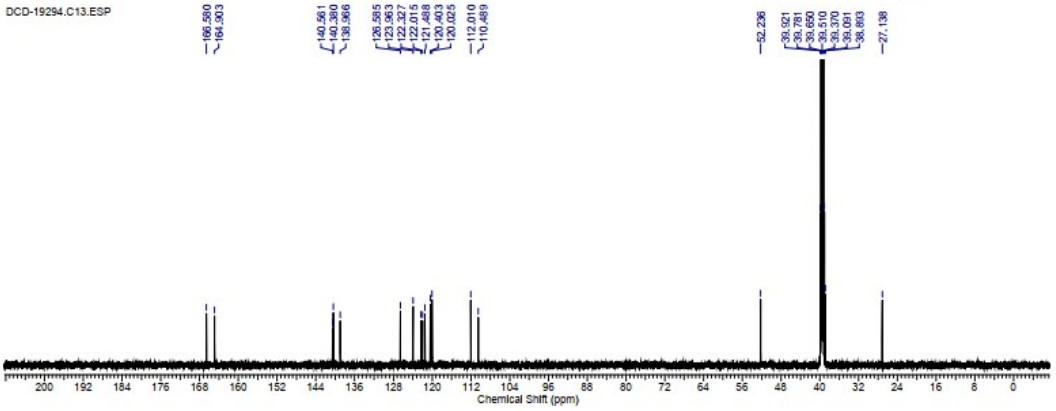


4c



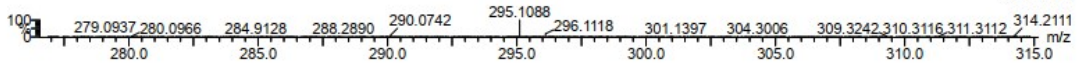


4c

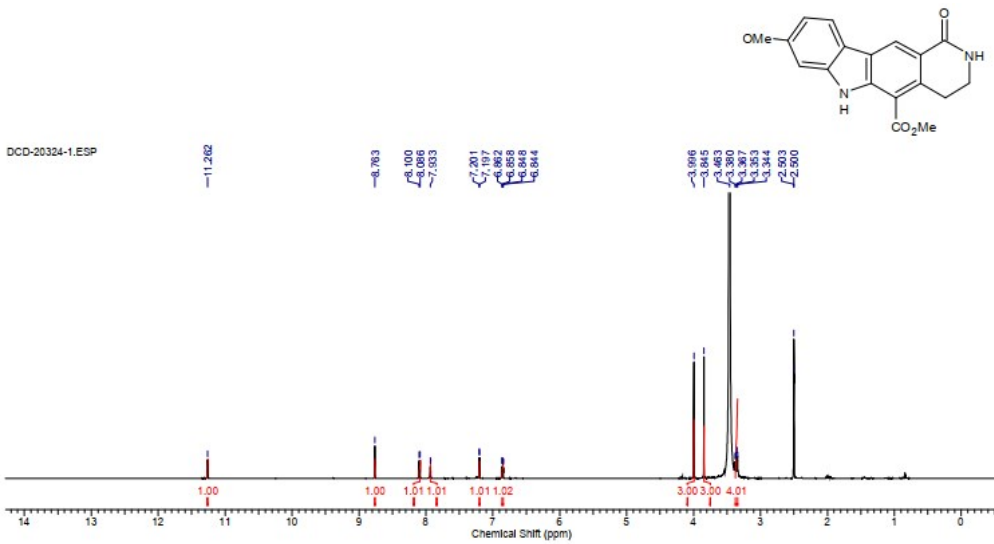


DCD-19294 103 (0.589)
1: TOF MS ES+

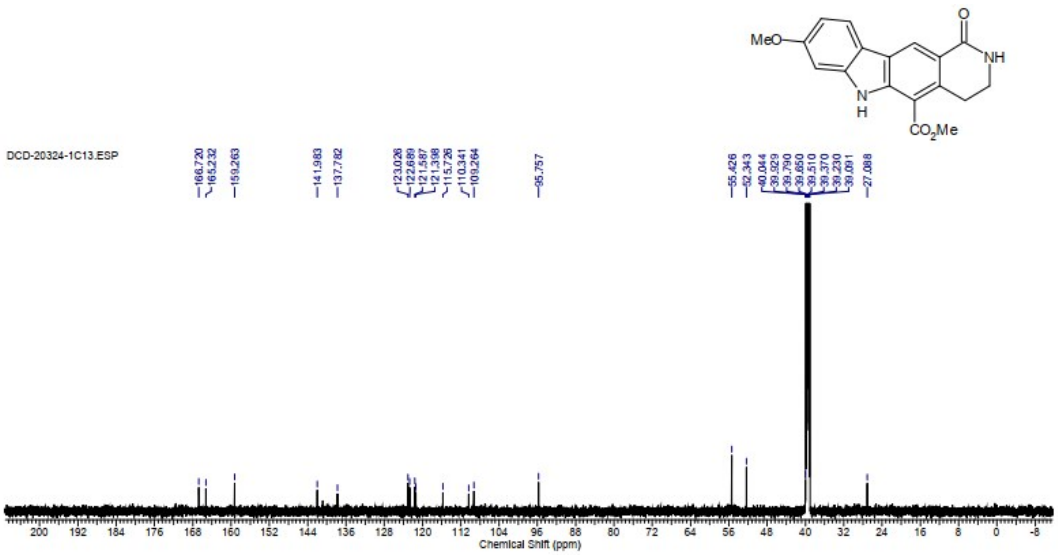
3.55e+006



Minimum:				-1.5			
Maximum:		20.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%) Formula
295.1088	295.1083	0.5	1.7	11.5	1223.8	n/a	n/a C17 H15 N2 O3



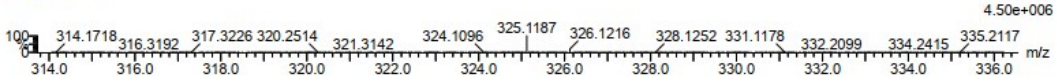
4d



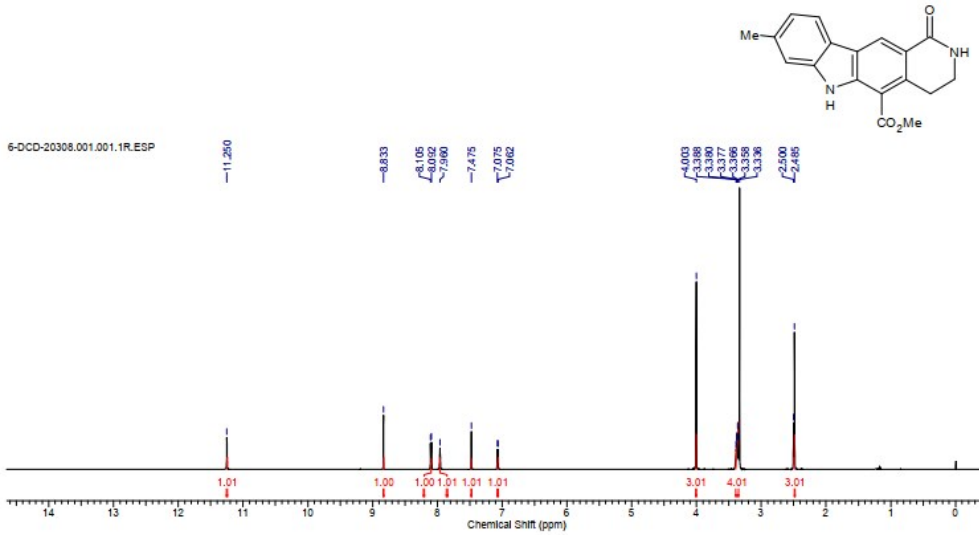
4d

DCD-20324 97 (0.558)
1: TOF MS ES+

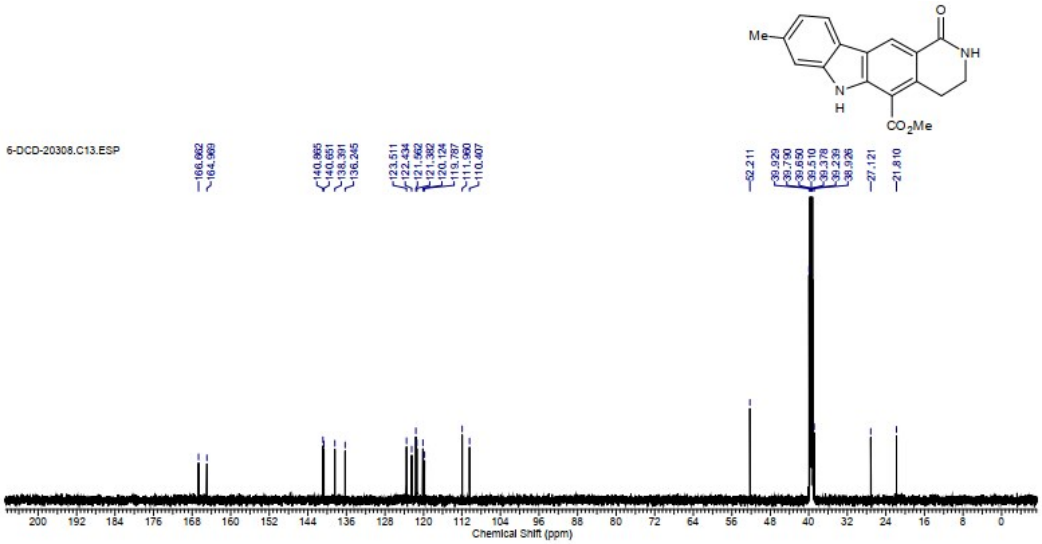
4.50e+006



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
325.1187	325.1188	-0.1	-0.3	11.5	1169.3	n/a	n/a	C18 H17 N2 O4



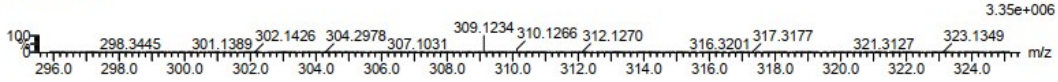
4e



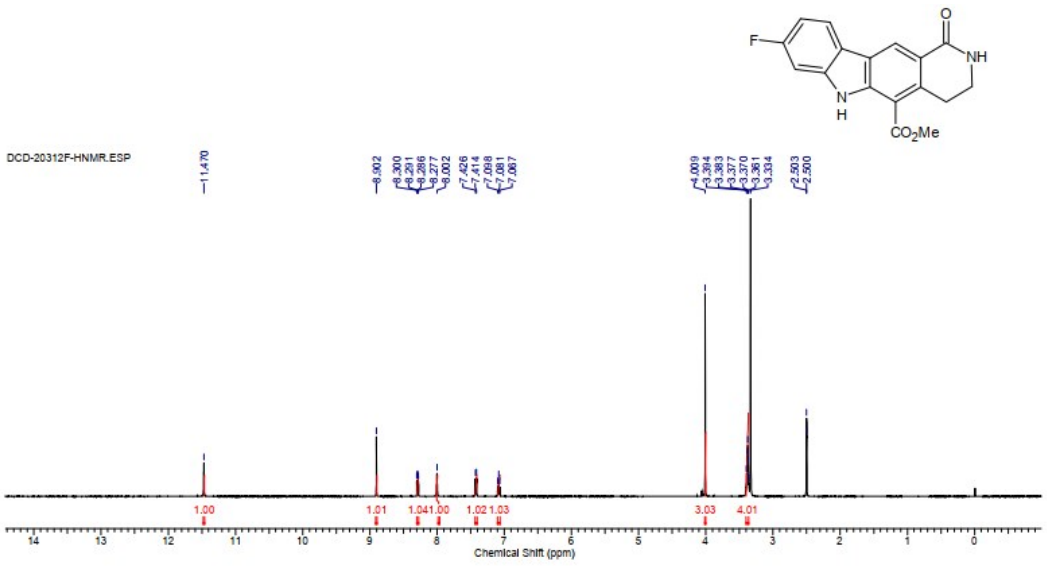
4e

DCD-20308 112 (0.636)
1: TOF MS ES+

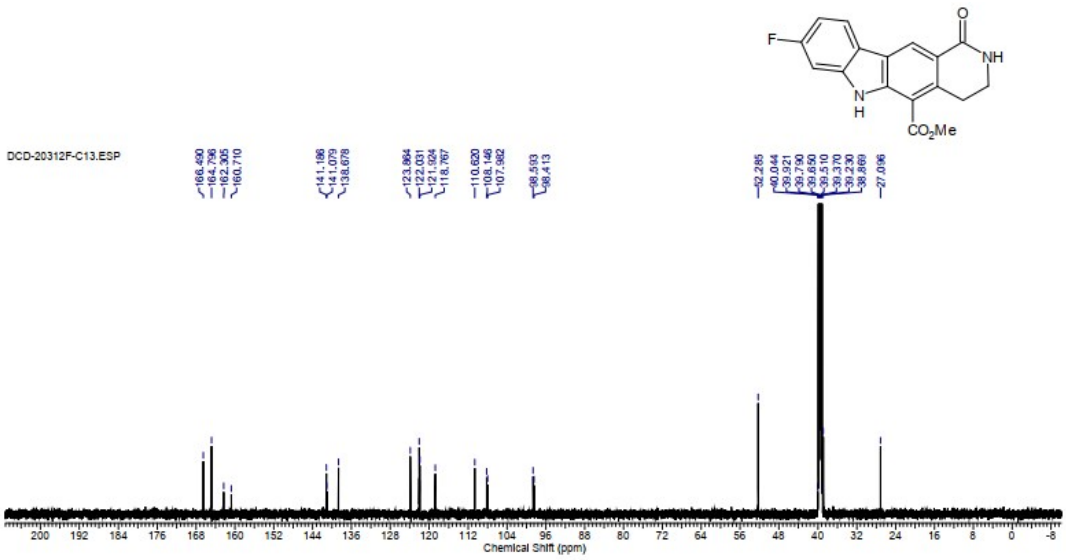
3.35e+006



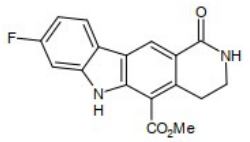
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
309.1234	309.1239	-0.5	-1.6	11.5	1163.0	n/a	n/a	C18 H17 N2 O3


F
O
NH
N
H
CO2Me
DCD-20312F-HNMR.ESP
11.470
8.302
8.300
8.291
8.286
8.277
8.002
7.426
7.414
7.088
7.081
7.067
4.009
3.394
3.383
3.377
3.370
3.361
3.334
2.503
2.500
1.00
1.01
1.04
1.00
1.02
1.03
3.03
4.01
Chemical Shift (ppm)

4f

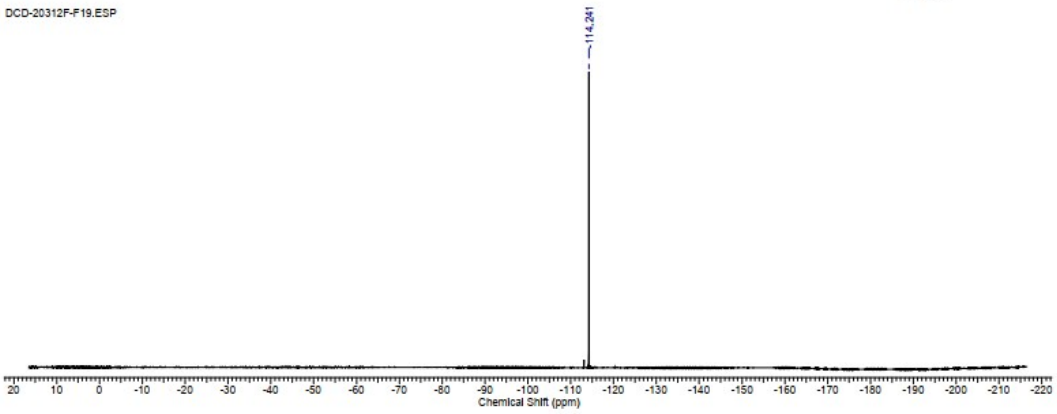

F
O
NH
N
H
CO2Me
DCD-20312F-C13.ESP
166.490
164.796
162.905
160.710
141.186
141.079
138.678
123.864
122.031
121.924
118.767
110.620
108.146
107.962
98.593
98.413
62.285
40.044
39.921
39.790
39.650
39.510
39.370
39.230
39.089
27.096
Chemical Shift (ppm)

4f



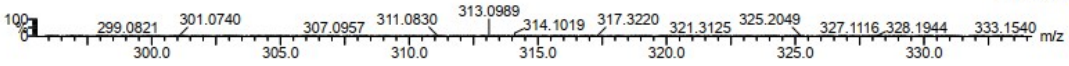
4f

DCD-20312F-F19.ESP

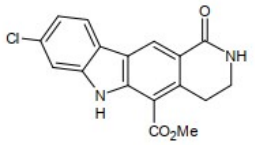


DCD-20312F 99 (0.568)
1: TOF MS ES+

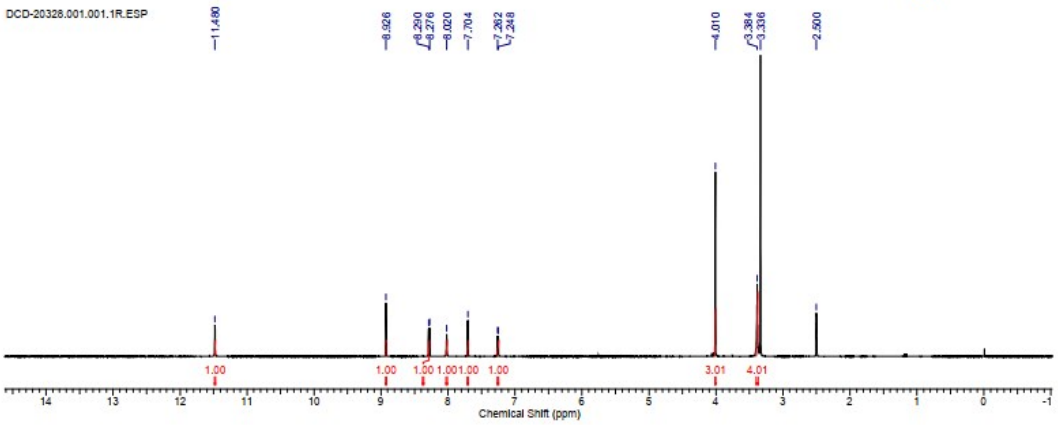
2.45e+006

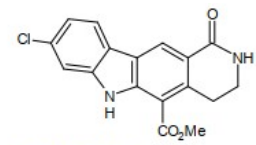


Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
313.0989	313.0988	0.1	0.3	11.5	1110.3	n/a	n/a	C17 H14 N2 O3 F

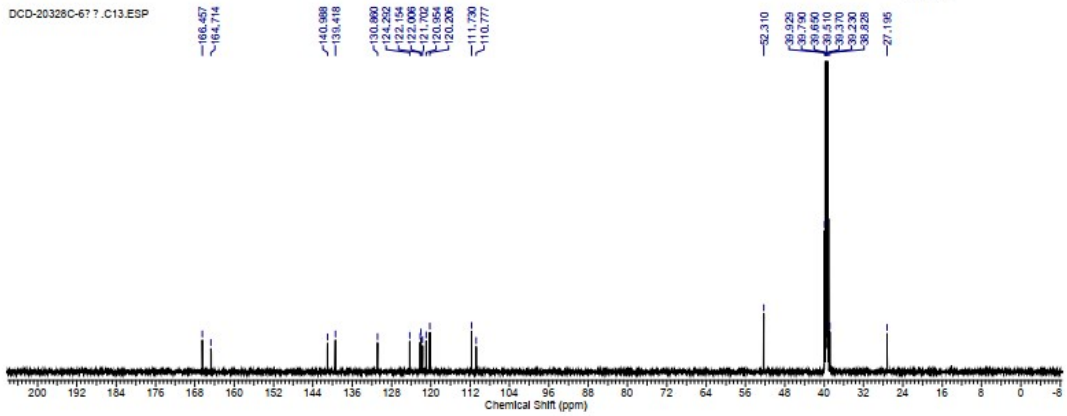


4g

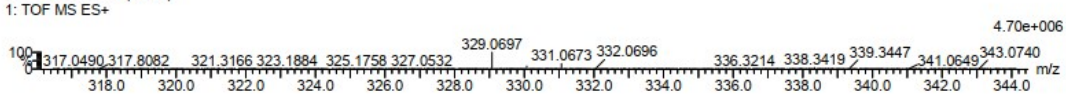




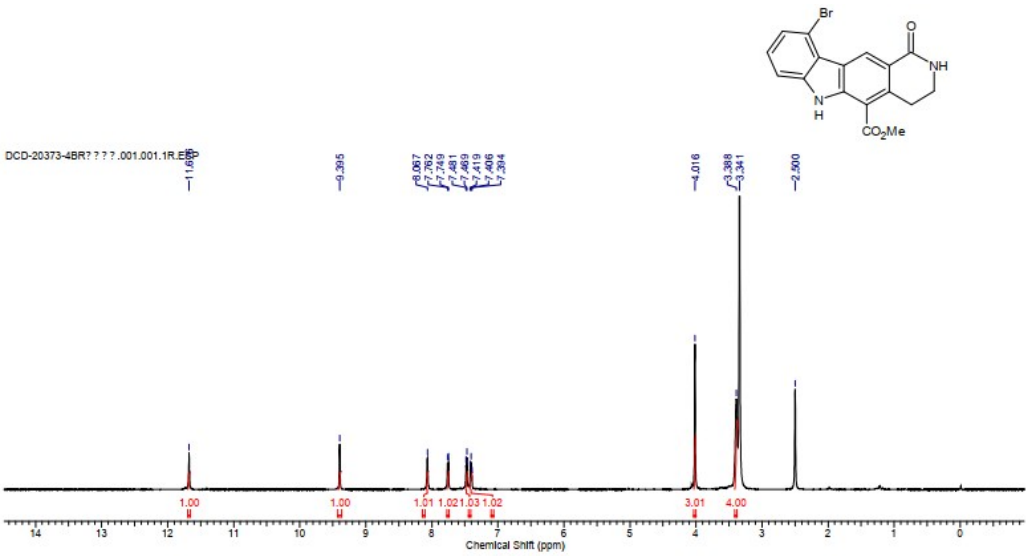
4g



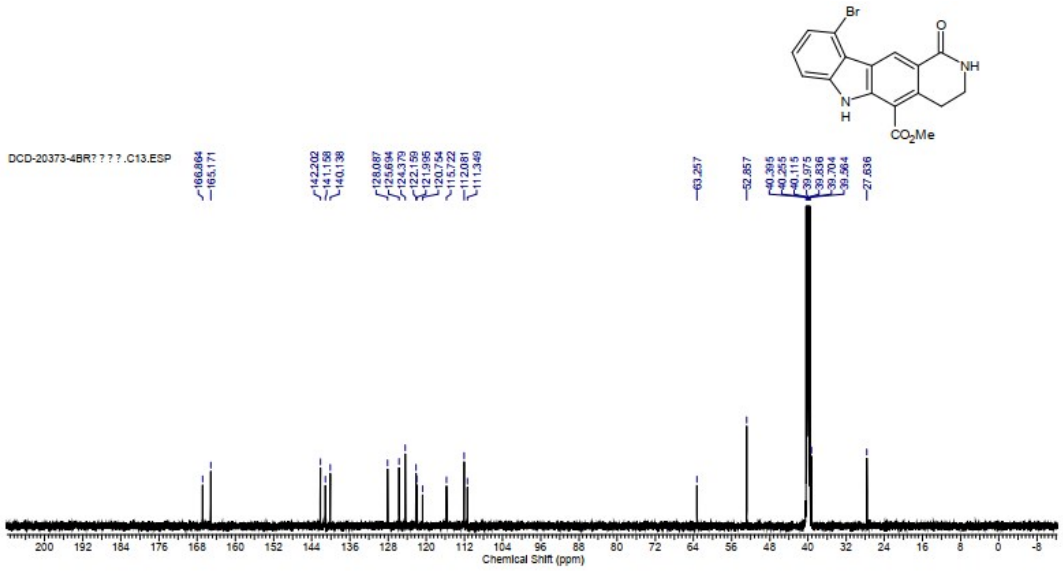
DCD-20328C 105 (0.600)
1: TOF MS ES+



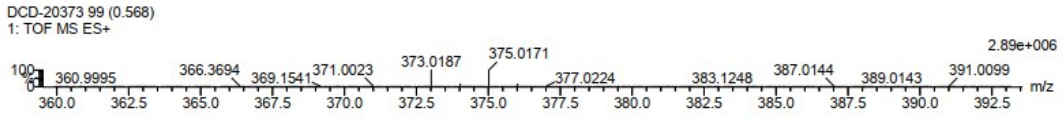
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
329.0697	329.0693	0.4	1.2	11.5	1276.9	n/a	n/a	C17 H14 N2 O3 Cl



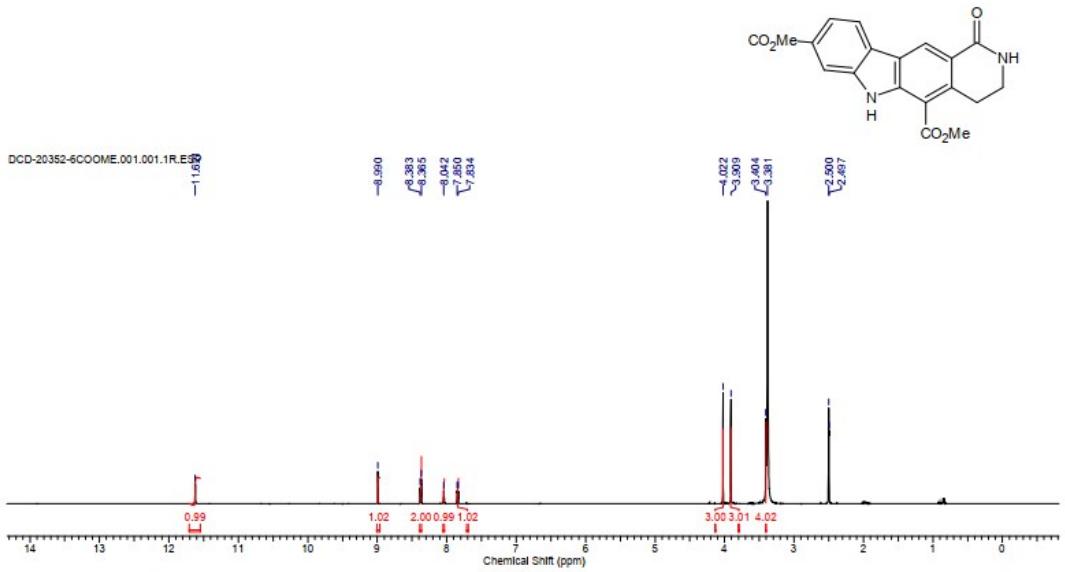
4h



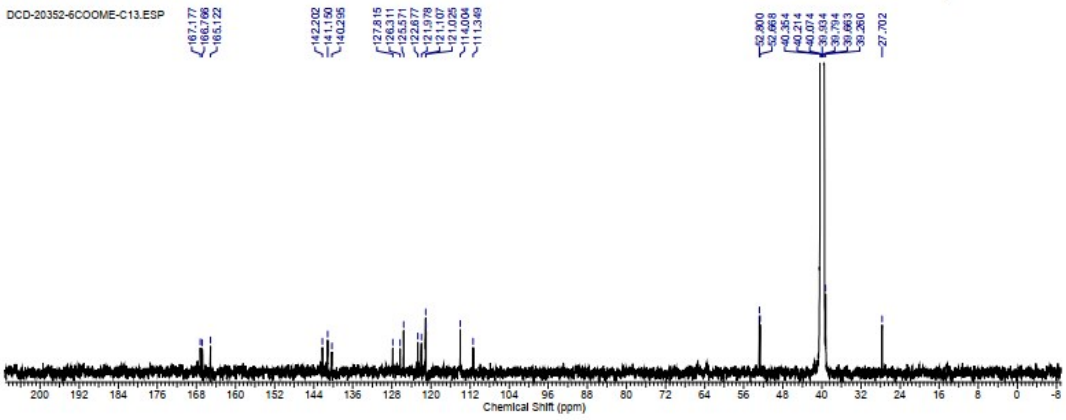
4h



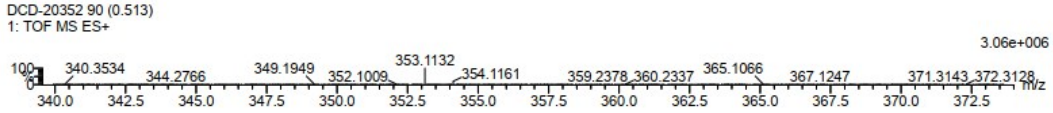
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
373.0187	373.0188	-0.1	-0.3	11.5	1110.0	n/a	n/a	C17 H14 N2 O3 Br



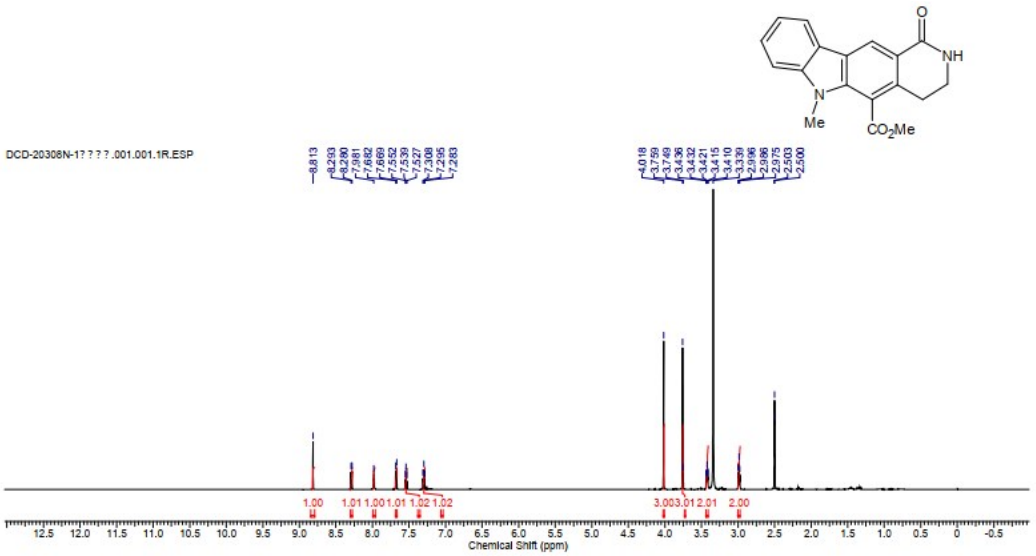
4i



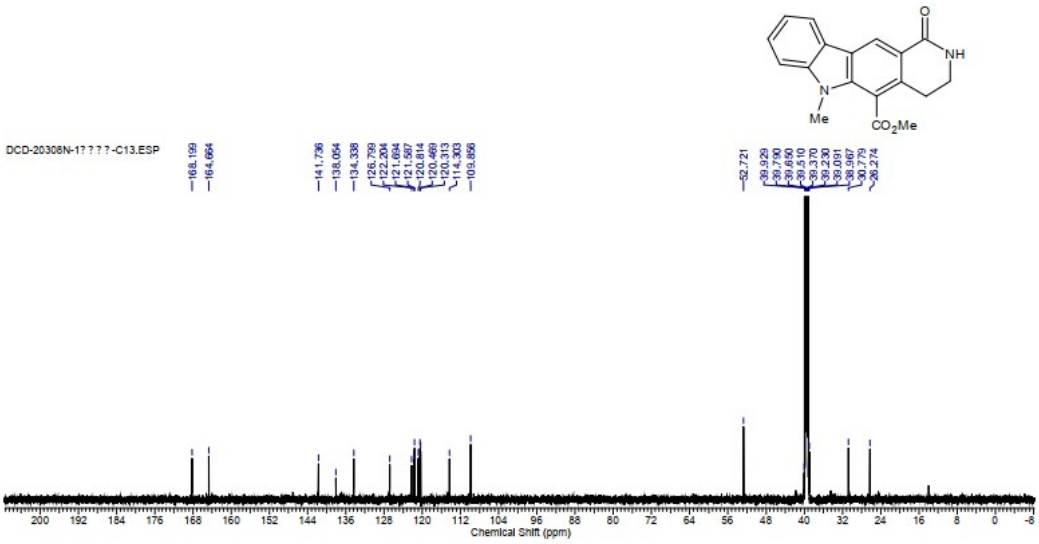
4i



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
353.1132	353.1137	-0.5	-1.4	12.5	1088.8	n/a	n/a	C19 H17 N2 O5

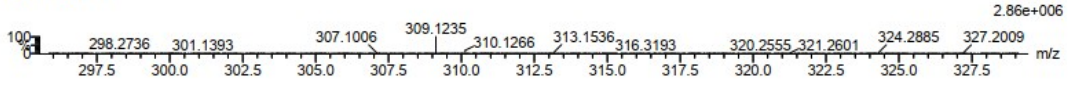


4j

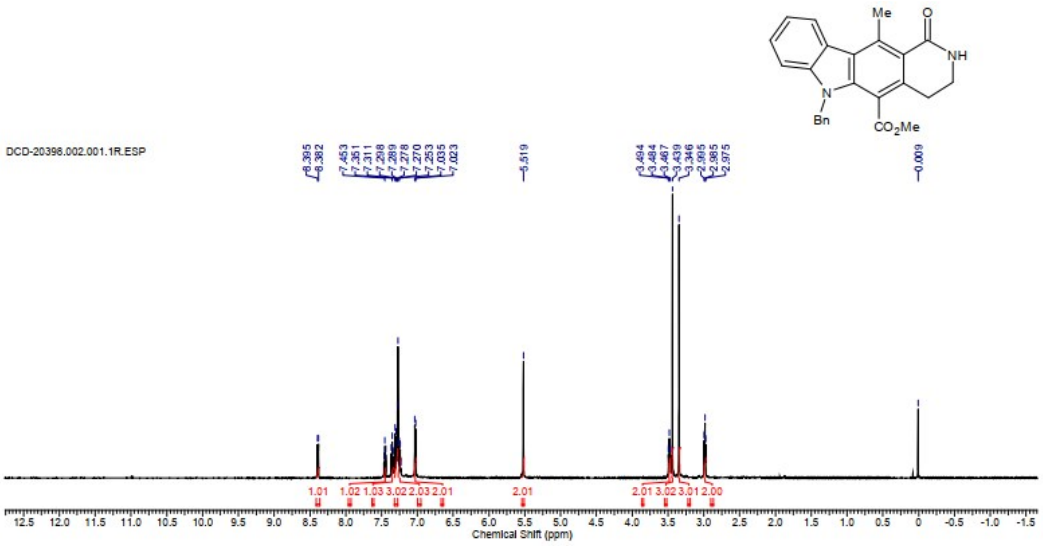


4j

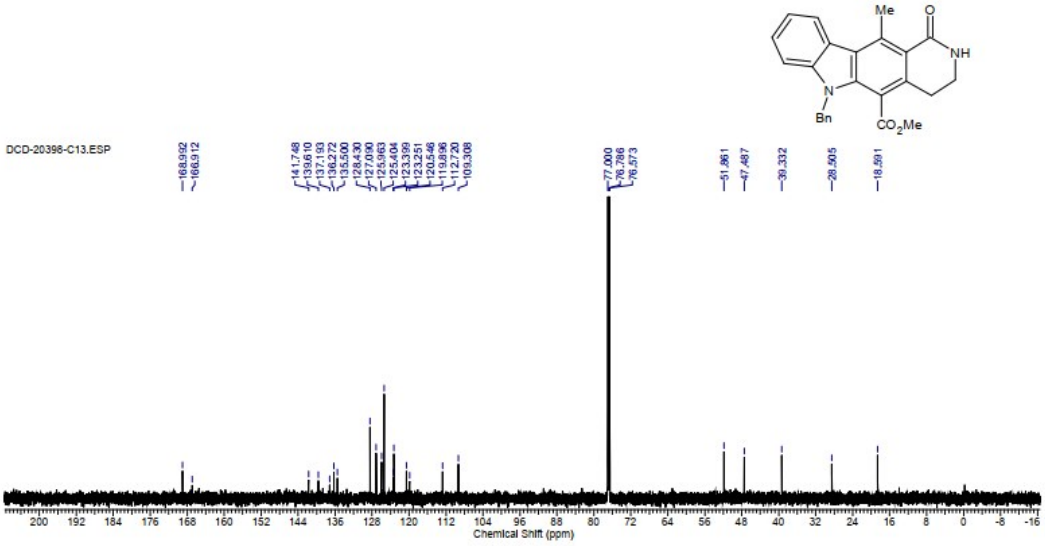
DCD-20308N 124 (0.707)
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
309.1235	309.1239	-0.4	-1.3	11.5	1198.6	n/a	n/a	C18 H17 N2 O3



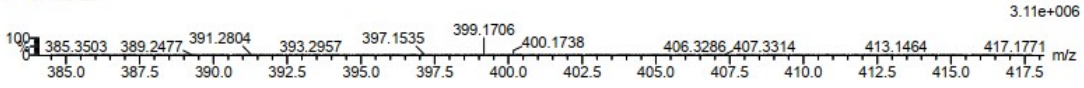
4k



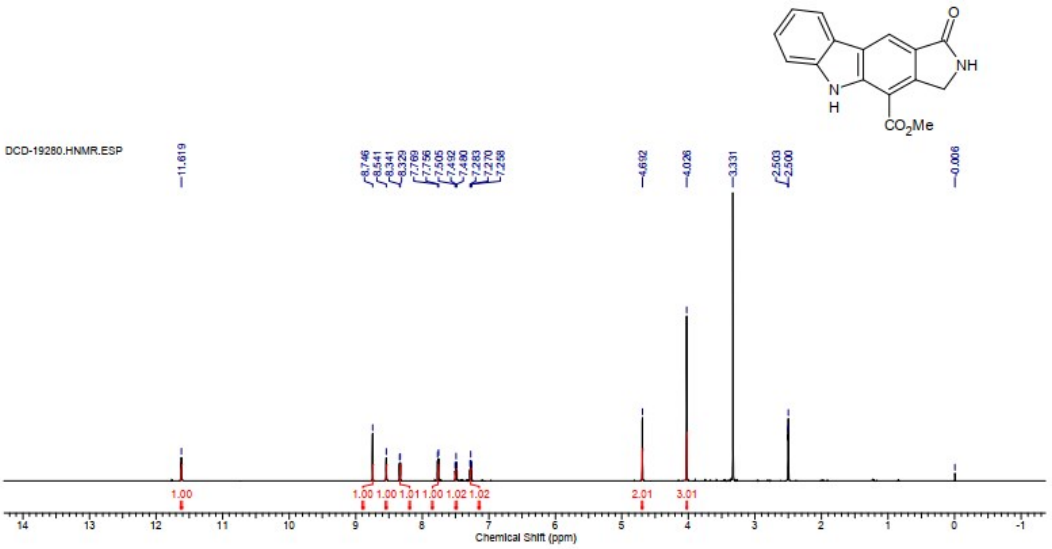
4k

DCD-20398 106 (0.605)
1: TOF MS ES+

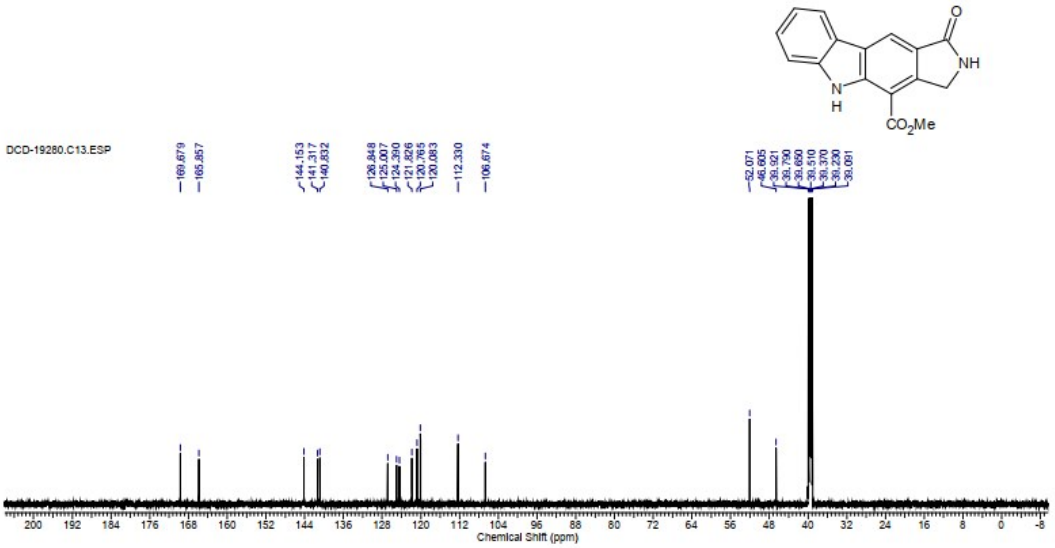
3.11e+006



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
399.1706	399.1709	-0.3	-0.8	15.5	1009.8	n/a	n/a	C25 H23 N2 O3



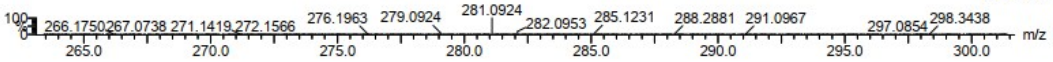
4l



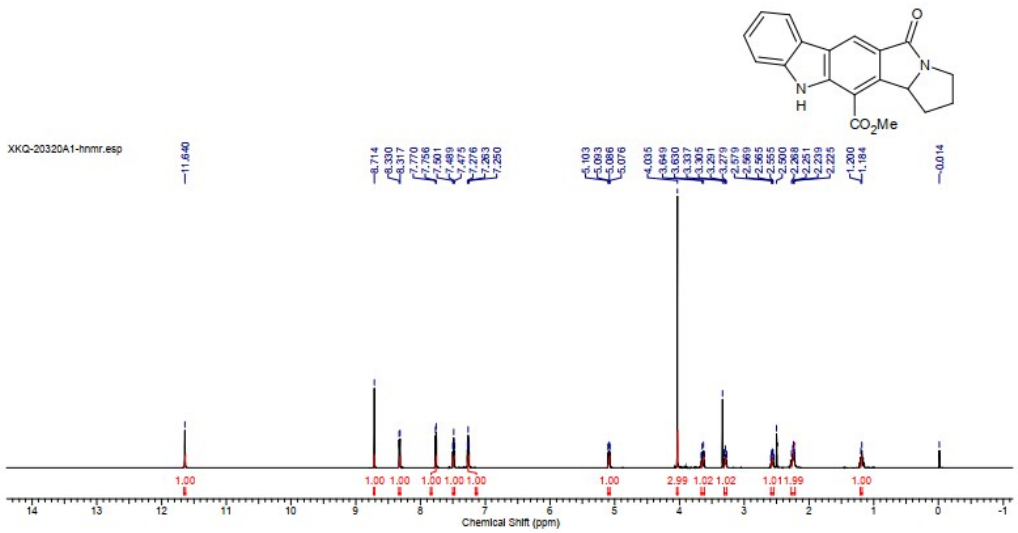
4l

DCD-19280 91 (0.526)
1: TOF MS ES+

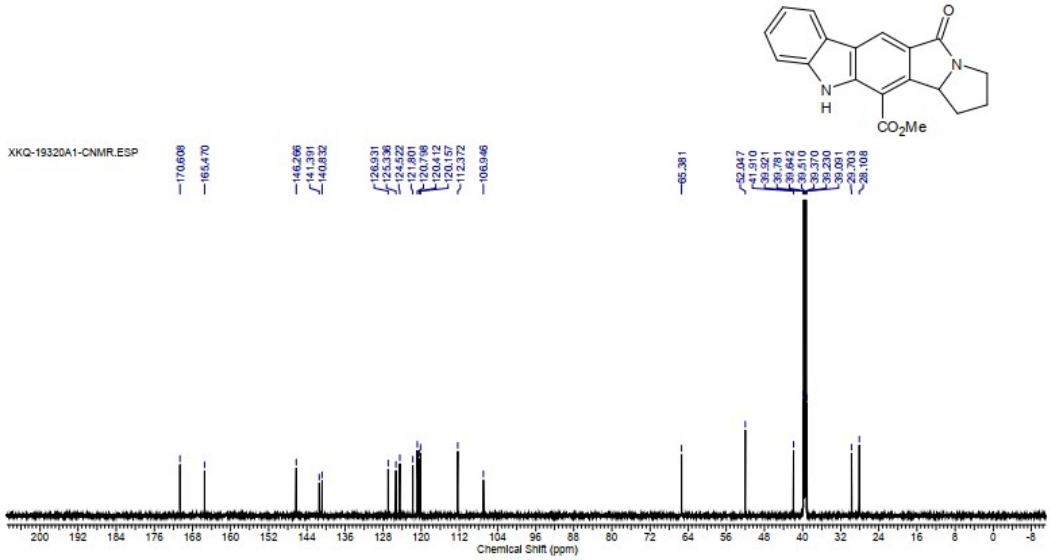
4.49e+006



Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
281.0924	281.0926	-0.2	-0.7	11.5	1306.1	n/a	n/a	C16 H13 N2 O3



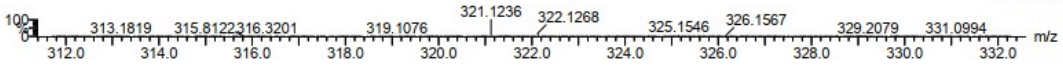
4m



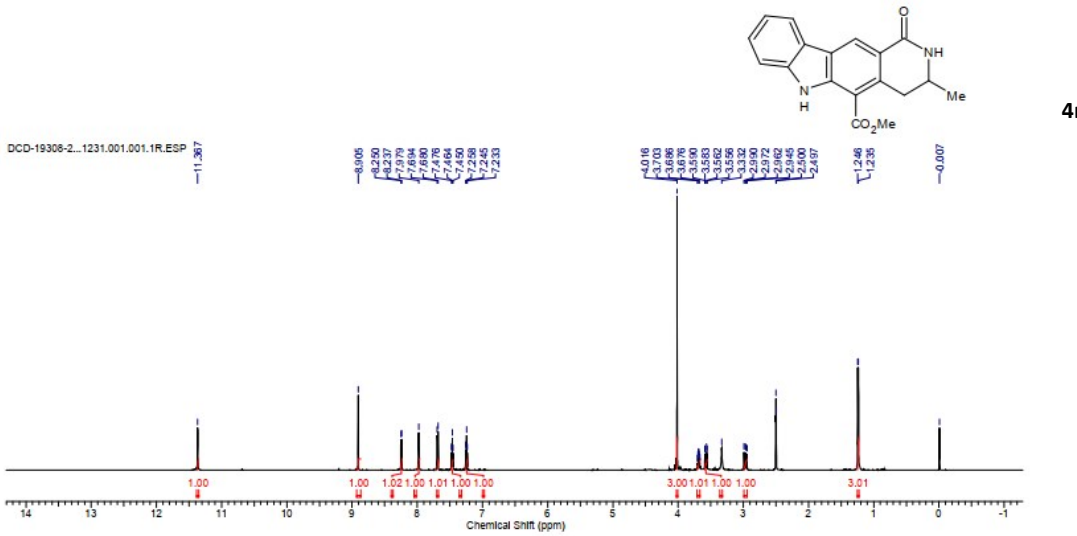
4m

XKQ-19320A1 108 (0.615)
1: TOF MS ES+

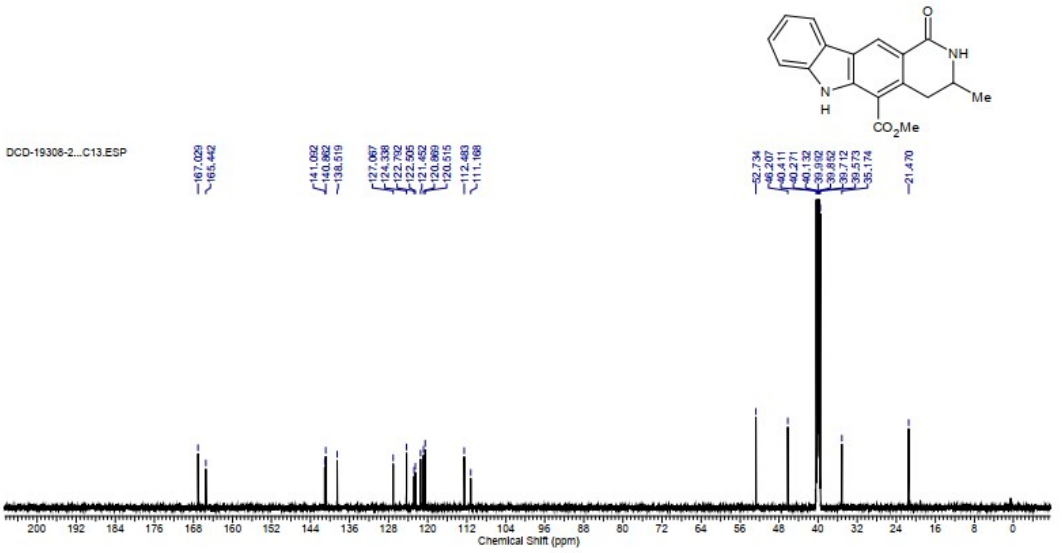
3.49e+006



Minimum:				-1.5				
Maximum:		20.0	20.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
321.1236	321.1239	-0.3	-0.9	12.5	1120.5	n/a	n/a	C19 H17 N2 O3



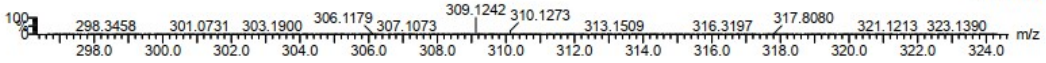
4n



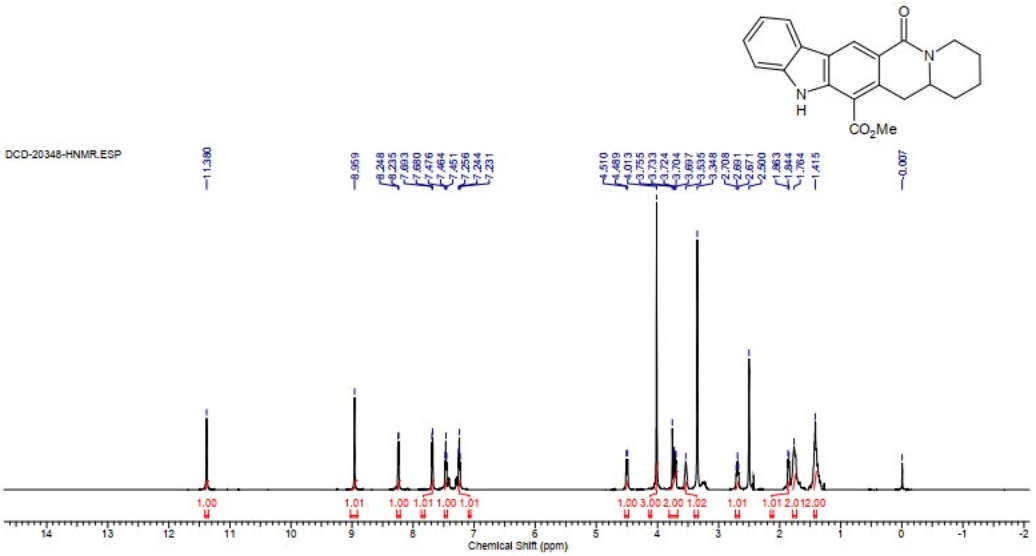
4n

DCD-19308-2 102 (0.584)
1: TOF MS ES+

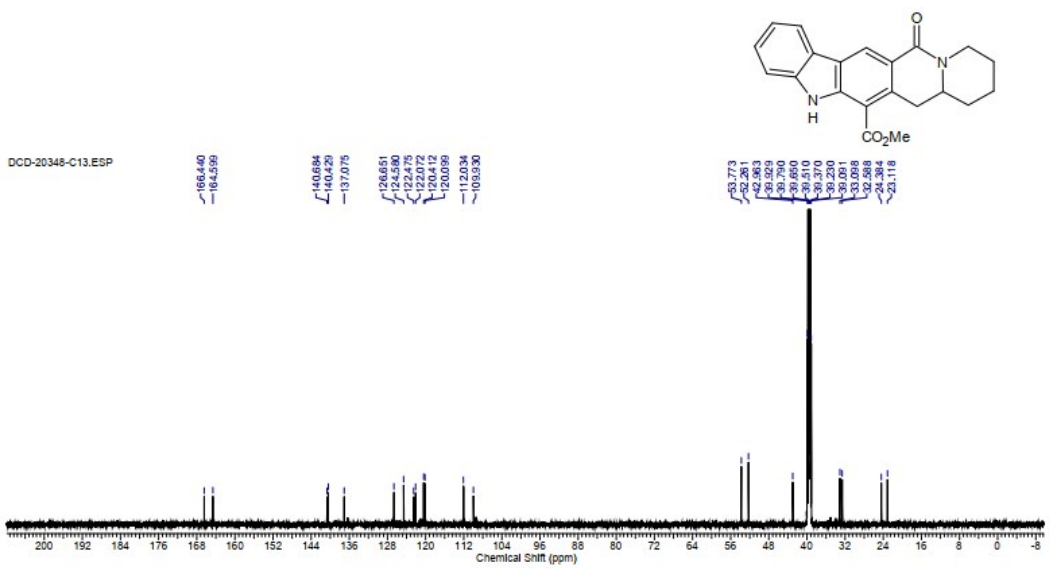
3.96e+006



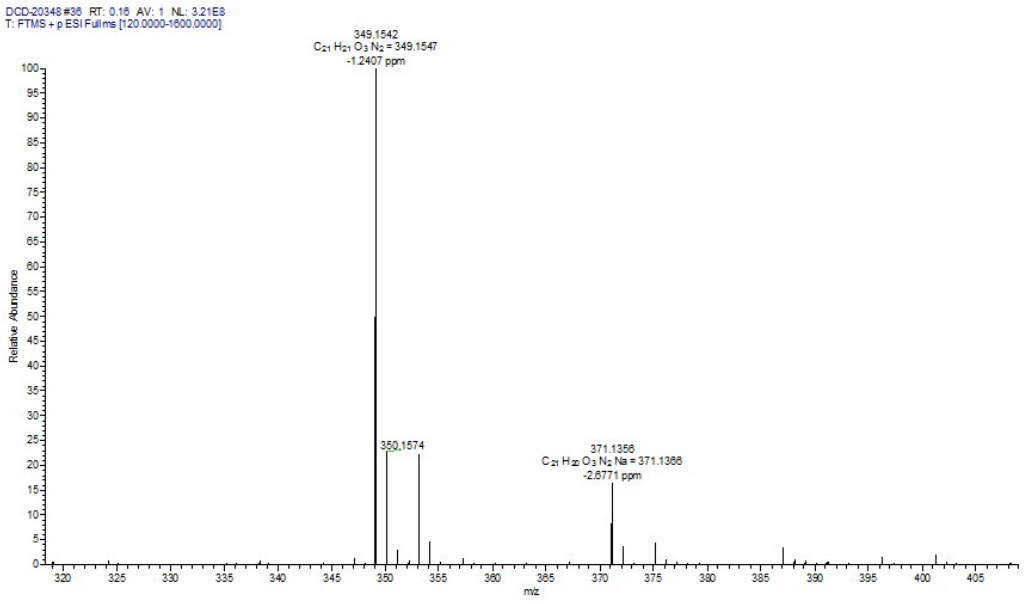
Minimum:				-1.5				
Maximum:		20.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
309.1212	309.1239	0.3	1.0	11.5	1222.2	n/a	n/a	C18 H17 N2 O3

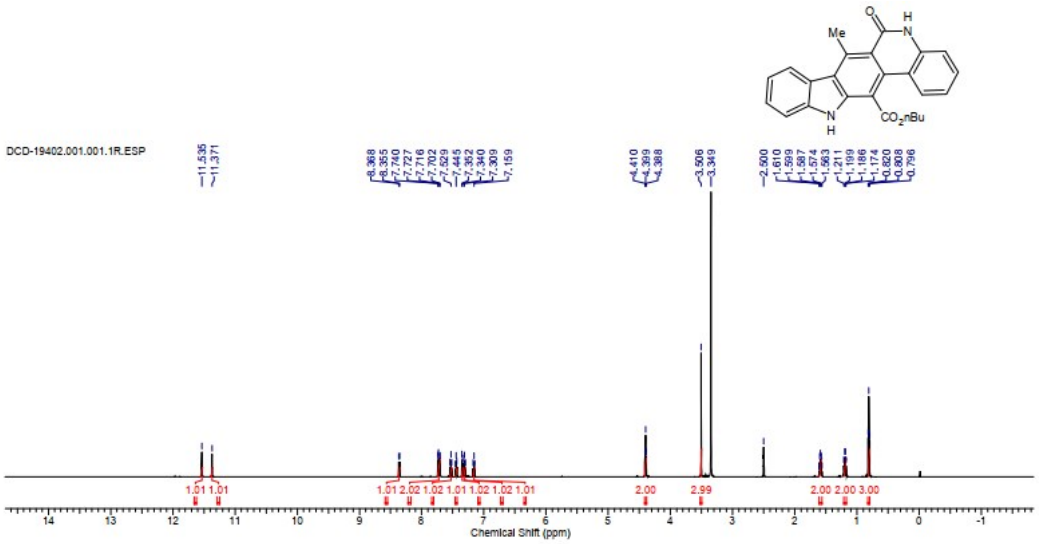

DCD-20348-HNMR.ESP
Chemical Shift (ppm)

4o

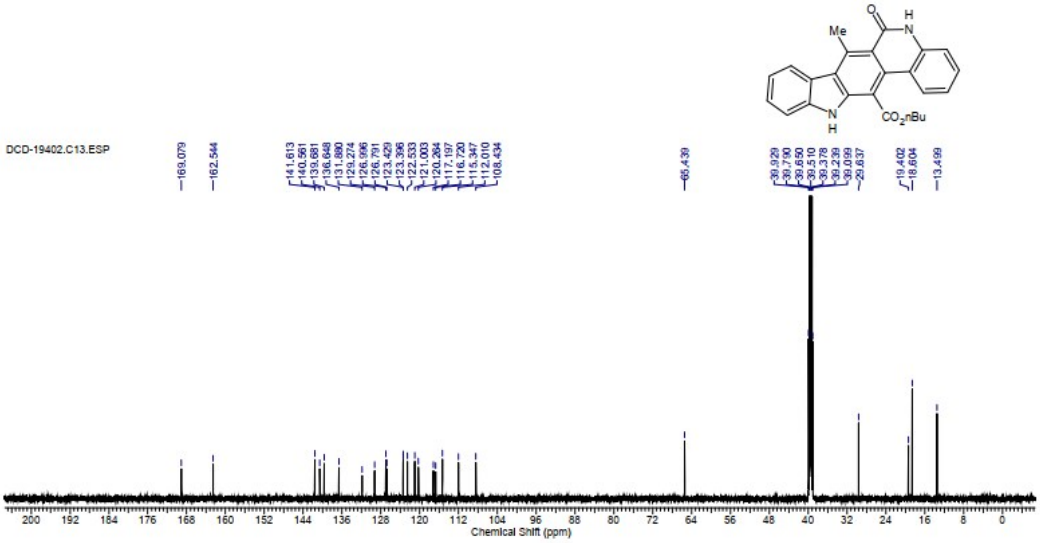

DCD-20348-C13.ESP
Chemical Shift (ppm)

4o

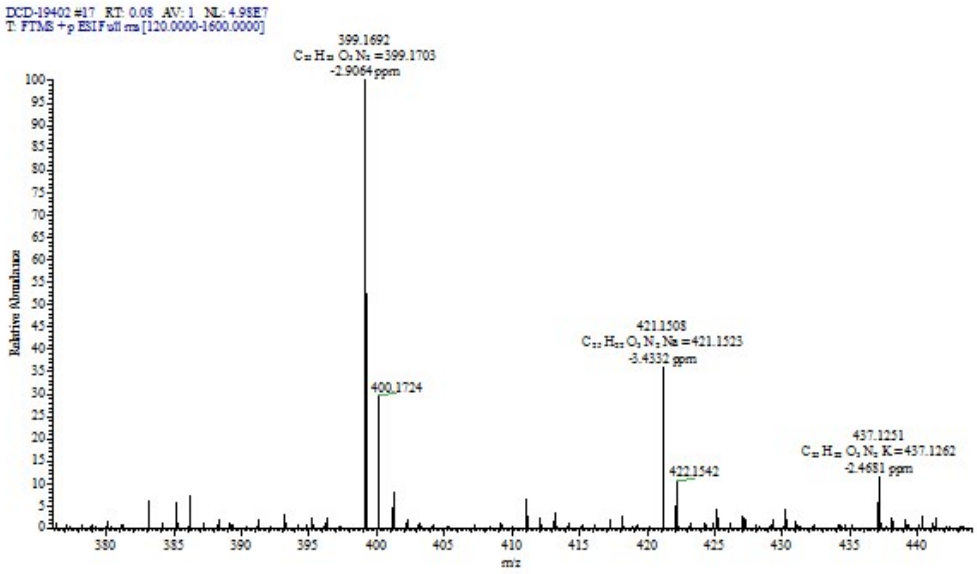

DCD-20348 #36 RT: 0.16 AV: 1 NL: 3.21E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
349.1542
C21 H21 O3 N2 = 349.1547
-1.2407 ppm
350.1574
371.1356
C21 H20 O3 N2 Na = 371.1366
-2.6771 ppm
Relative Abundance
m/z

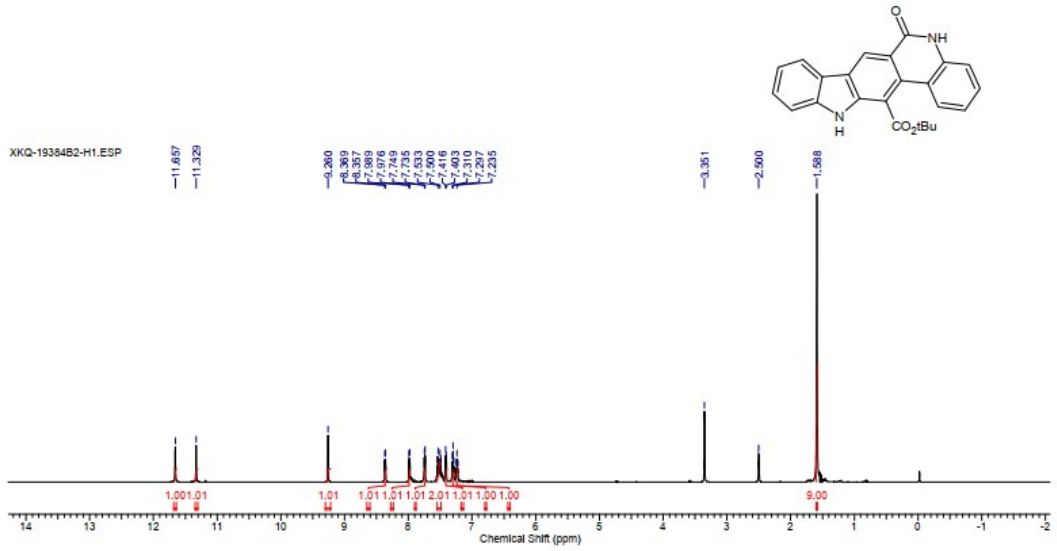

DCD-19402.001.001.1R.ESP
Chemical Shift (ppm)

4p

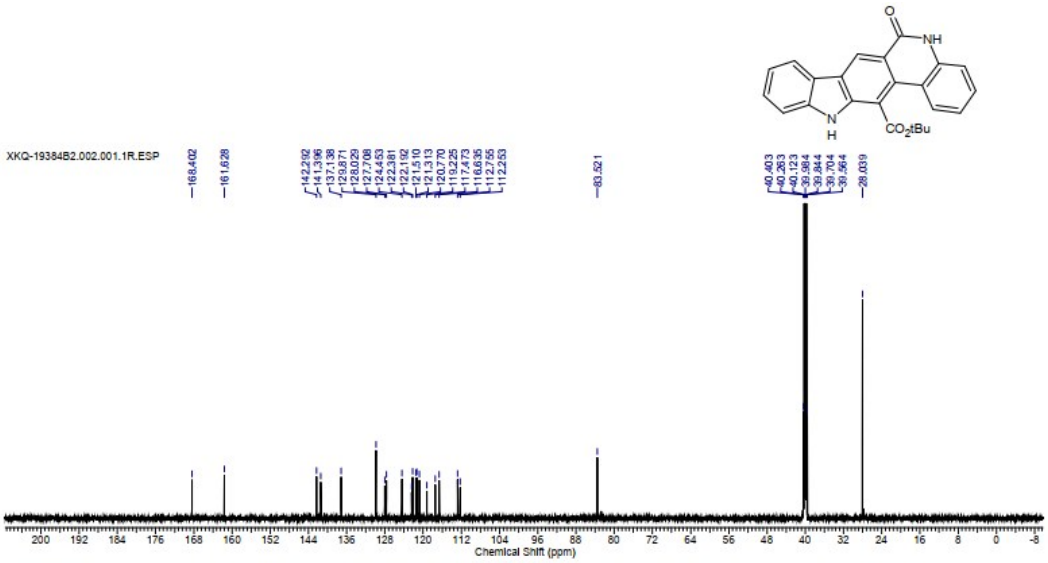

DCD-19402.C13.ESP
Chemical Shift (ppm)

4p

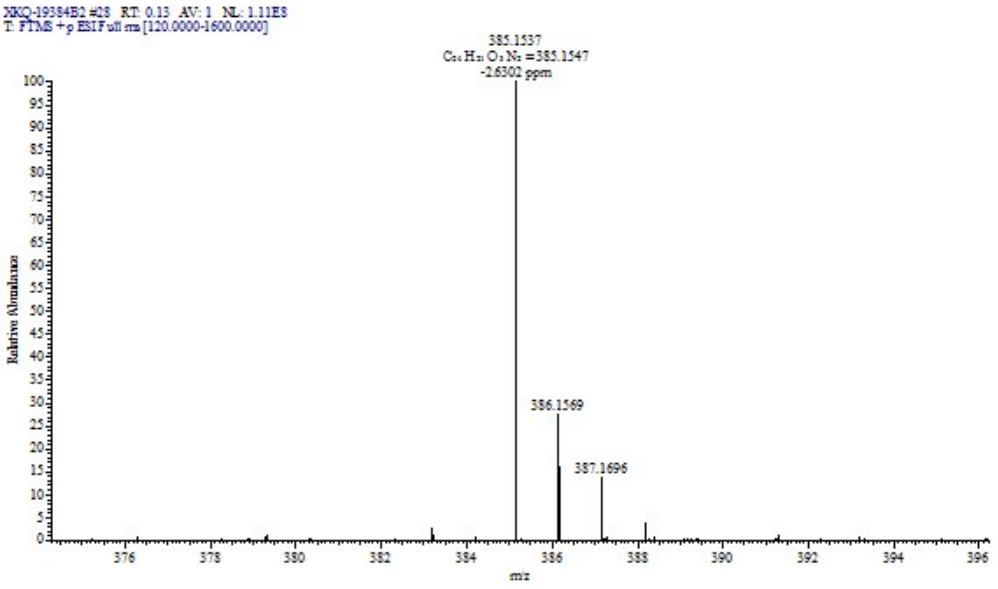

DCD-19402 #17 RT: 0.08 AV: 1 NL: 4.98E7
T: FTMS + p ESI Full ms [120.0000-1600.0000]
399.1692
C32 H24 O3 N2 =399.1703
-2.9064 ppm
400.1724
421.1508
C32 H22 O3 N2 Na =421.1523
-3.4332 ppm
422.1542
437.1251
C32 H24 O3 N2 K=437.1262
-2.4681 ppm
Relative Abundance
m/z

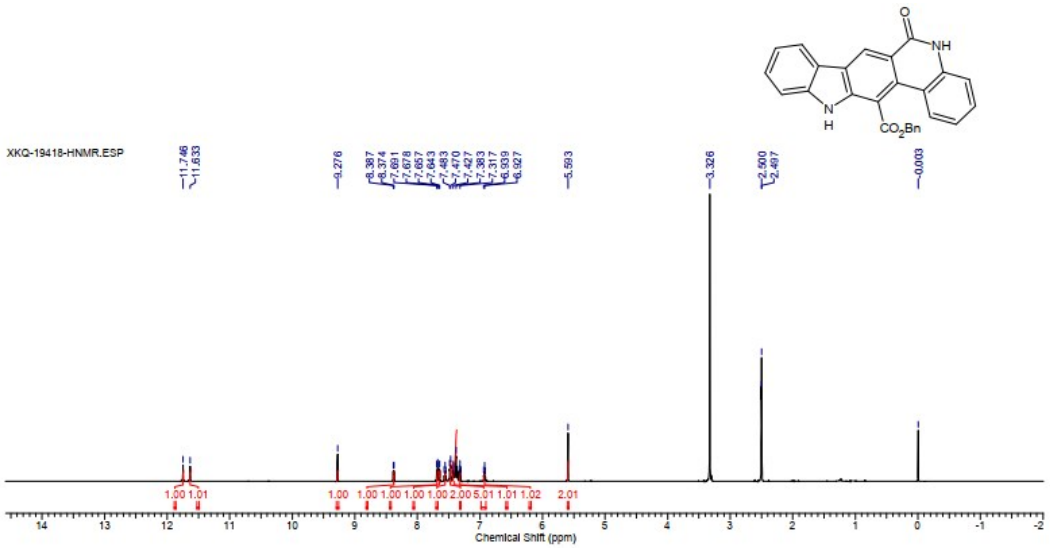

XKQ-19384B2-H1.ESP
11.657
11.329
9.260
8.369
8.357
7.989
7.976
7.749
7.735
7.533
7.500
7.416
7.400
7.310
7.297
7.235
3.351
2.500
1.588
1.00 1.01 1.01
1.01
1.01 1.01 1.01 2.01 1.01 1.00 1.00
9.00
Chemical Shift (ppm)

4q

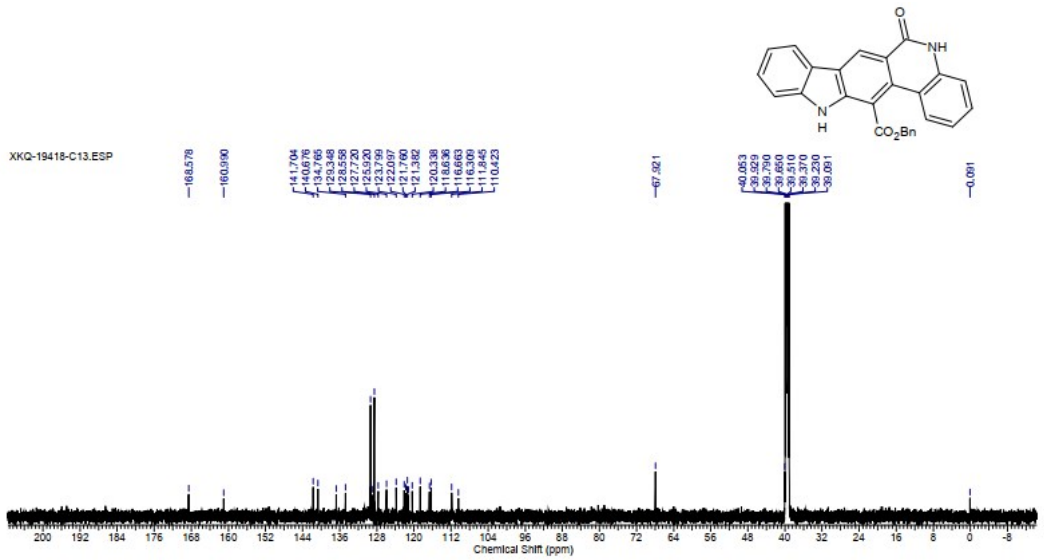

XKQ-19384B2.002.001.1R.ESP
168.402
161.628
142.292
141.396
133.738
129.871
128.029
127.708
124.453
122.381
122.192
121.510
121.313
120.770
119.225
117.473
116.635
112.755
112.253
63.521
40.403
40.263
40.123
39.984
39.844
39.704
39.564
28.039
Chemical Shift (ppm)

4q

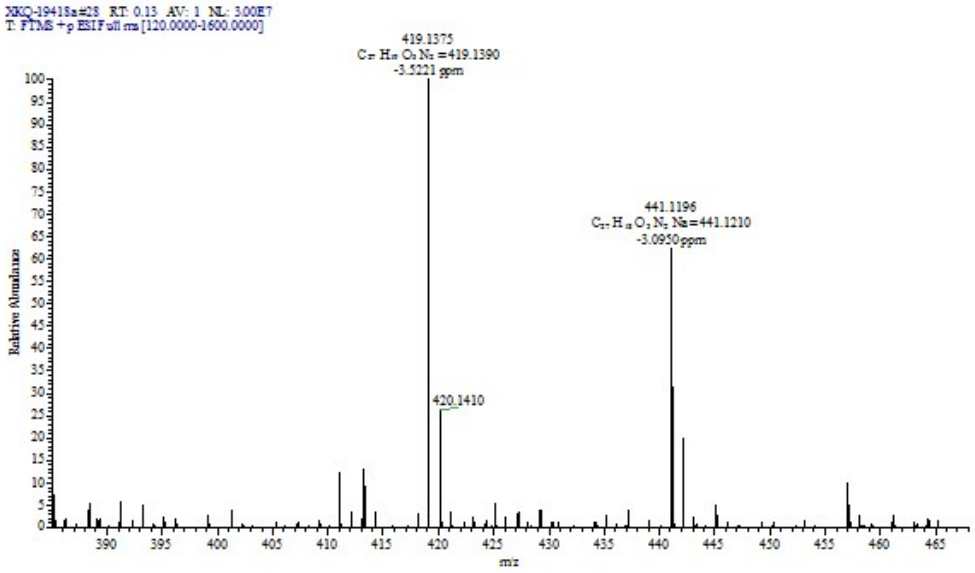

XKQ-19384B2 #28 RT: 0.13 AV: 1 NL: 1.11E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
385.1537
C24 H21 O3 N2 = 385.1547
-2.6302 ppm
386.1569
387.1696
Relative Abundance
m/z

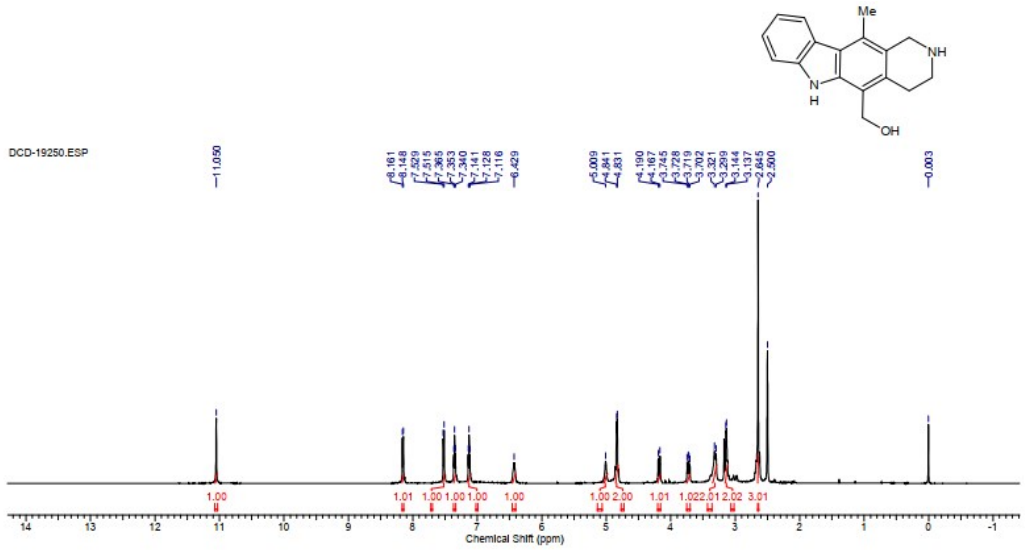

XKQ-19418-HNMR.ESP
Chemical Shift (ppm)

4r

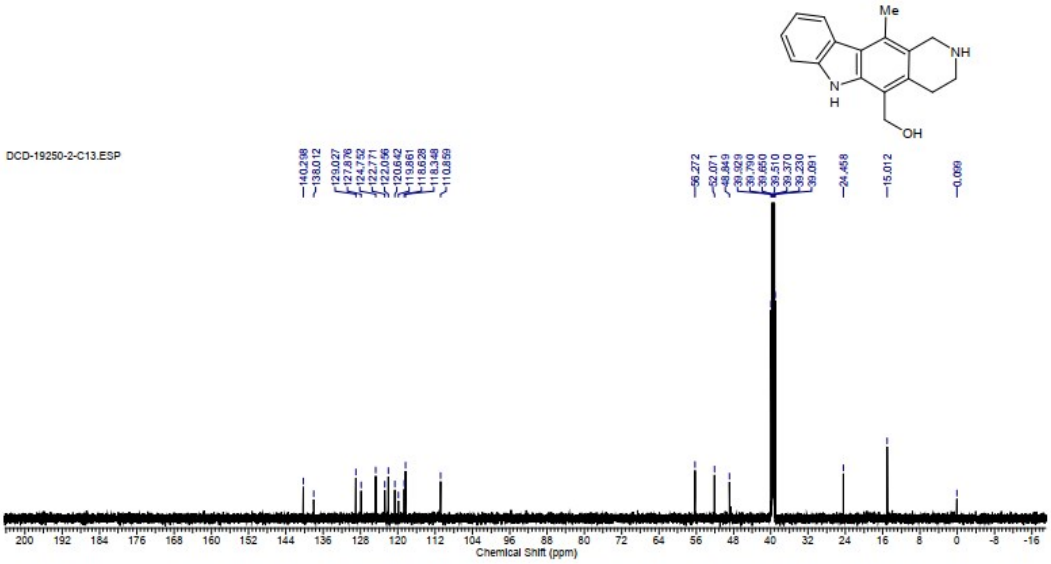

XKQ-19418-C13.ESP
Chemical Shift (ppm)

4r


XKQ-19418a#28 RT: 0.13 AV: 1 NL: 3.00E7
T: FTMS + p ESI Full ms [120.0000-1600.0000]
419.1375
C27 H19 O3 N2 =419.1390
-3.5221 ppm
441.1196
C27 H18 O3 N2 Na=441.1210
-3.0950 ppm
420.1410
Relative Abundance
m/z



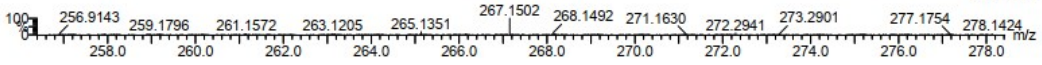
8



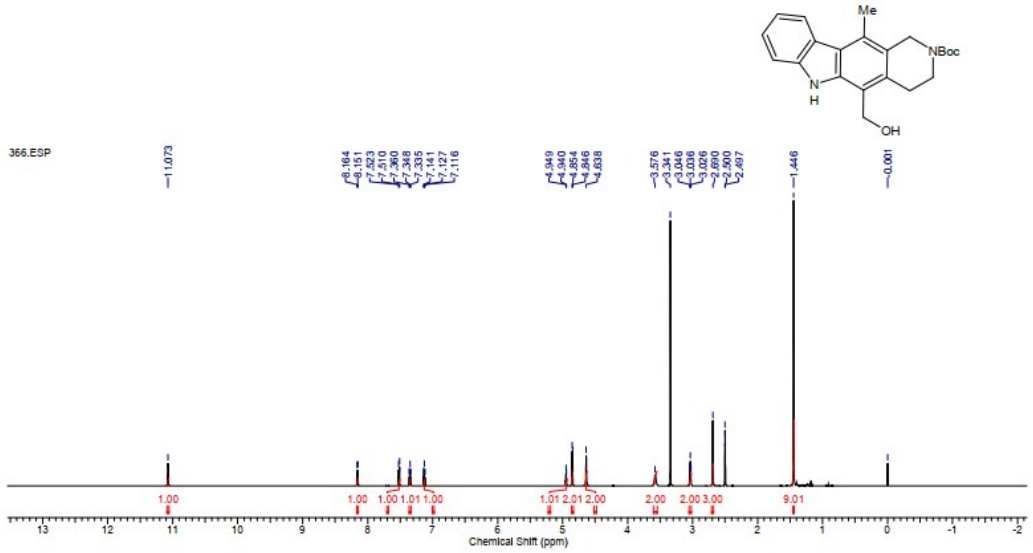
8

DCD-19250-2 90 (0.513)
1: TOF MS ES+

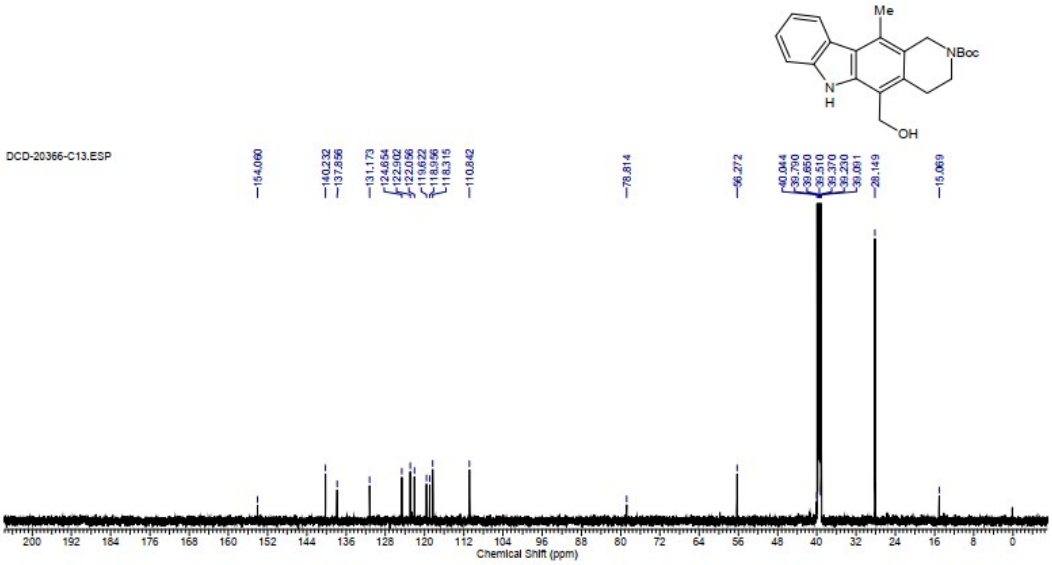
4.04e+004



Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
267.1502	267.1497	0.5	1.9	9.5	632.5	n/a	n/a	C17 H19 N2 O



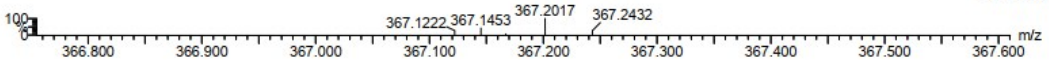
9



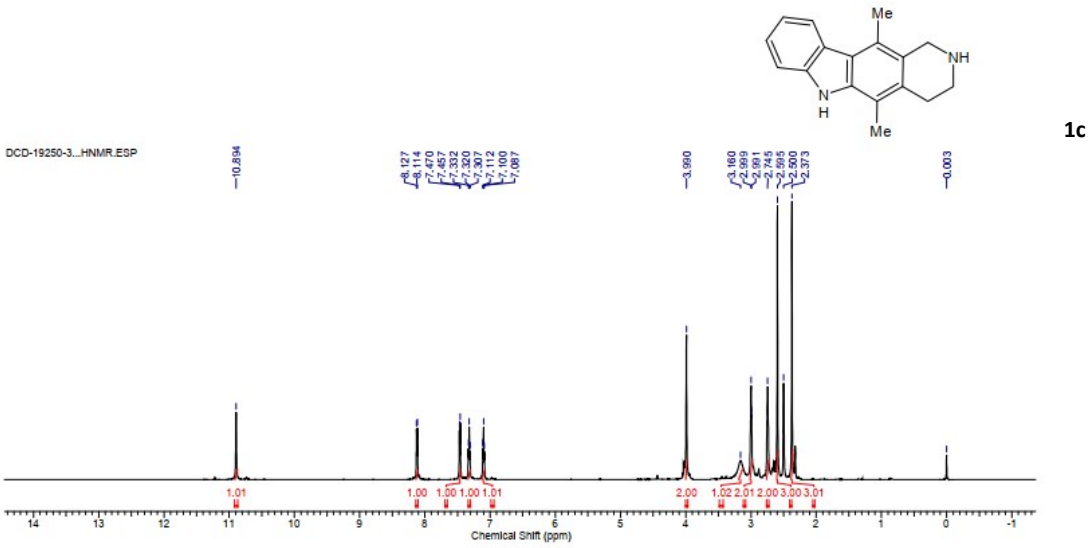
9

DCD-19366 167 (0.941)
1: TOF MS ES+

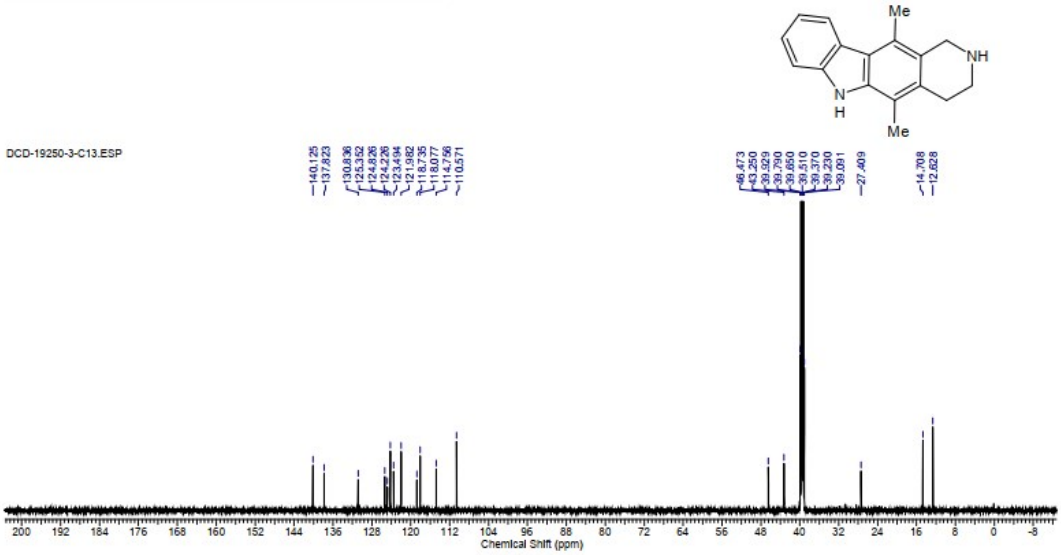
3.91e+002



Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
367.2017	367.2022	-0.5	-1.4	10.5	30.9	n/a	n/a	C22 H27 N2 O3



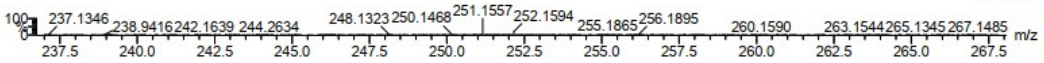
1c



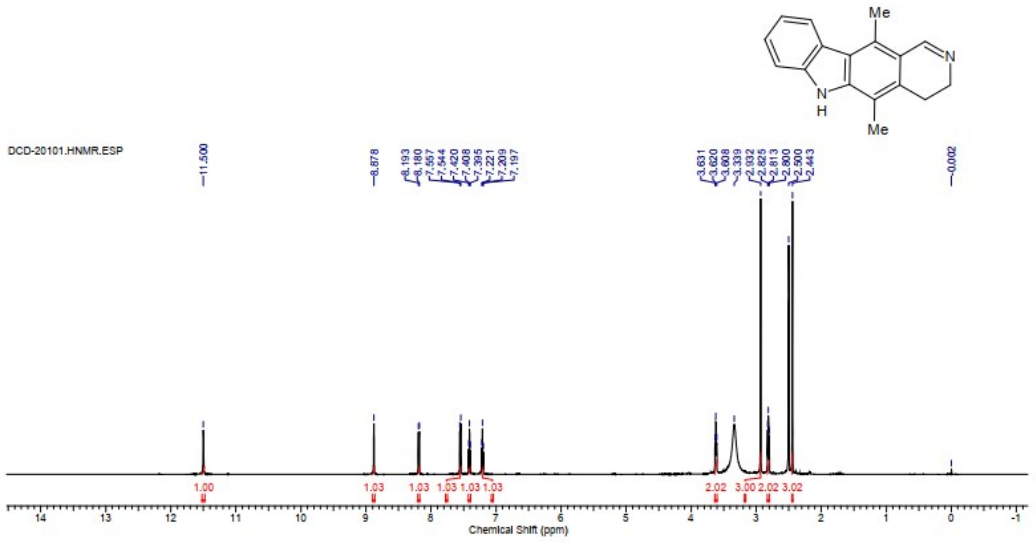
1c

DCD-19250-3 84 (0.481)
1: TOF MS ES+

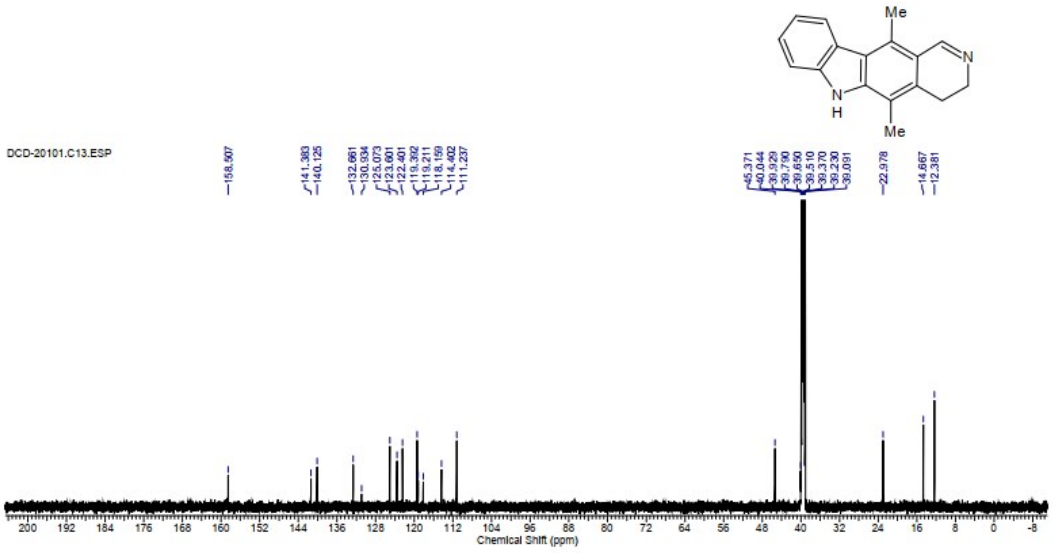
2.00e+006



Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
251.1557	251.1548	0.9	3.6	9.5	1054.6	n/a	n/a	C17 H19 N2



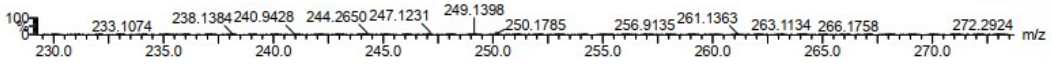
1b



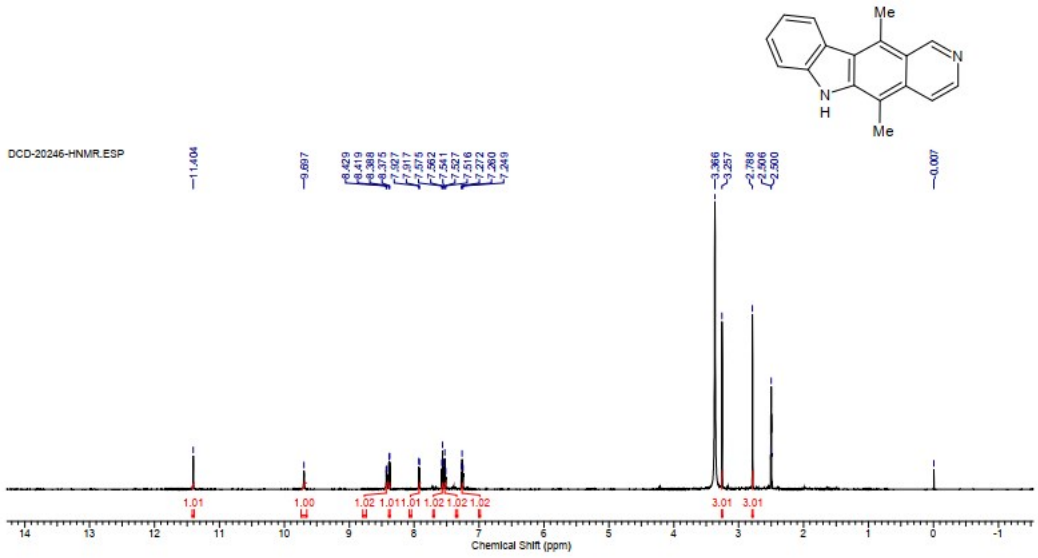
1b

DCD-20101 87 (0.497)
1: TOF MS ES+

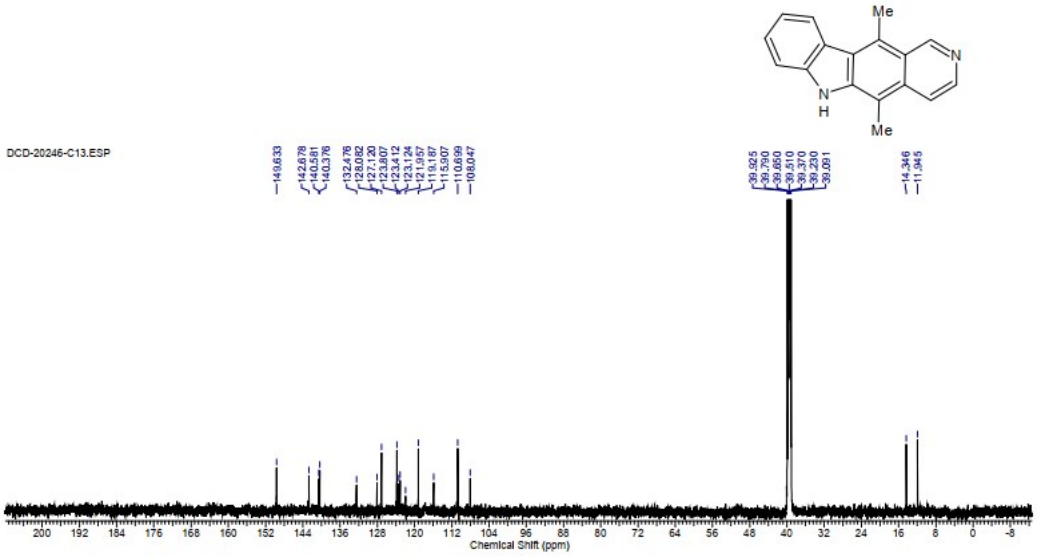
8.88e+004



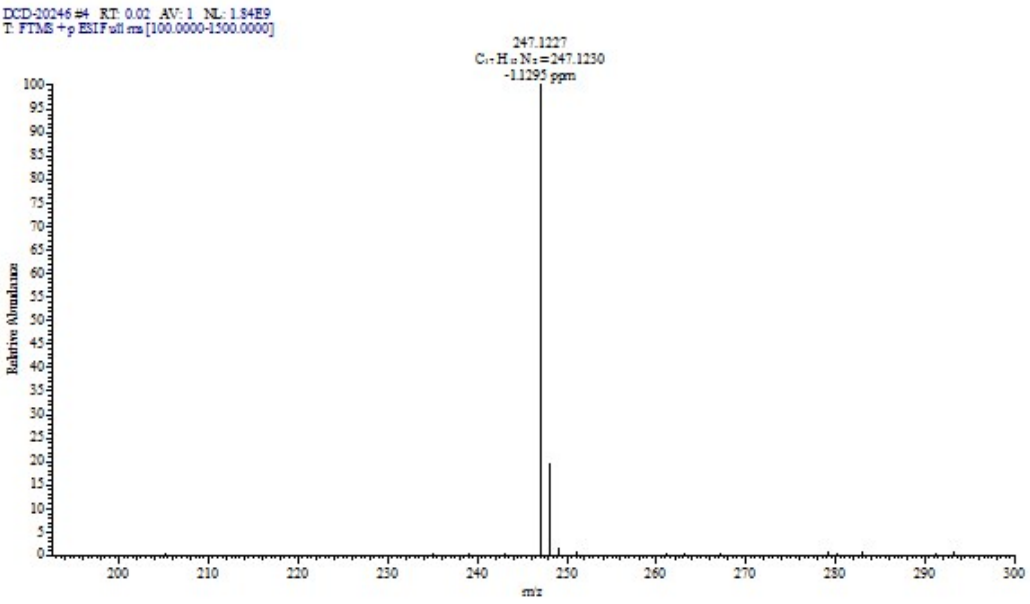
Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
249.1398	249.1392	0.6	2.4	10.5	649.5	n/a	n/a	C17 H17 N2

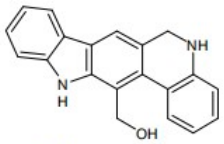


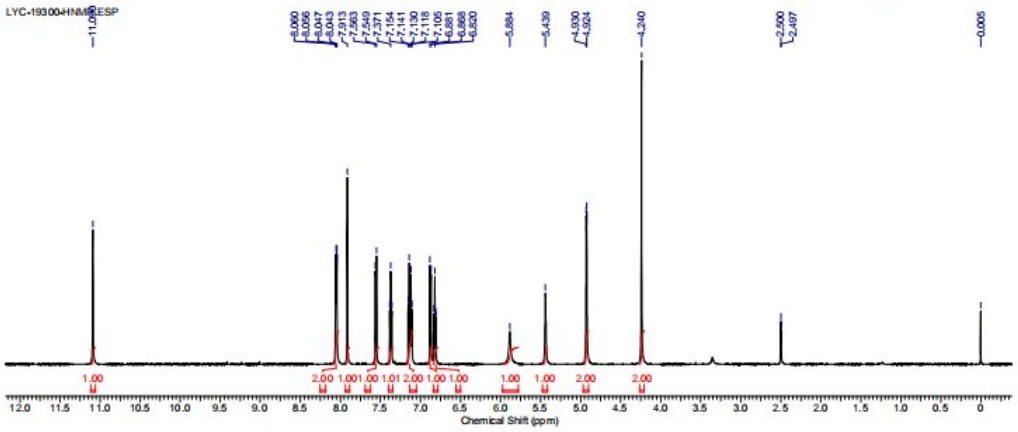
ellipticine



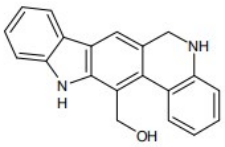
ellipticine



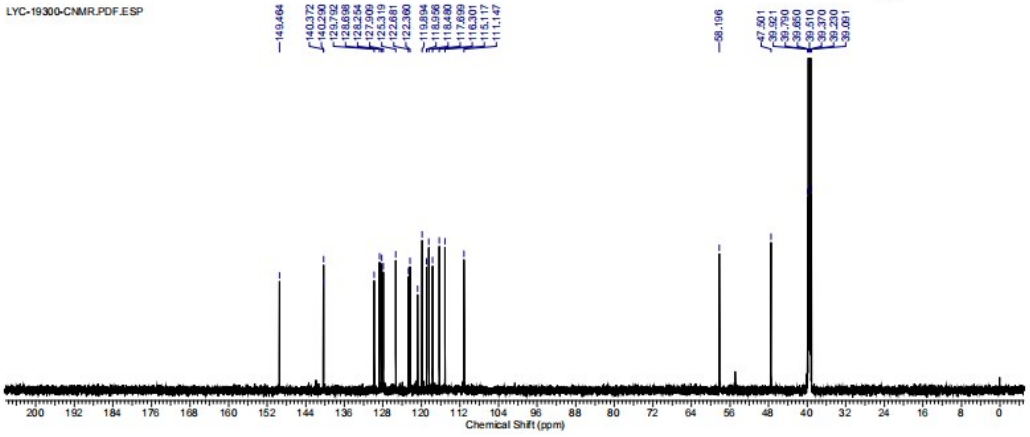

NH
N
H
OH

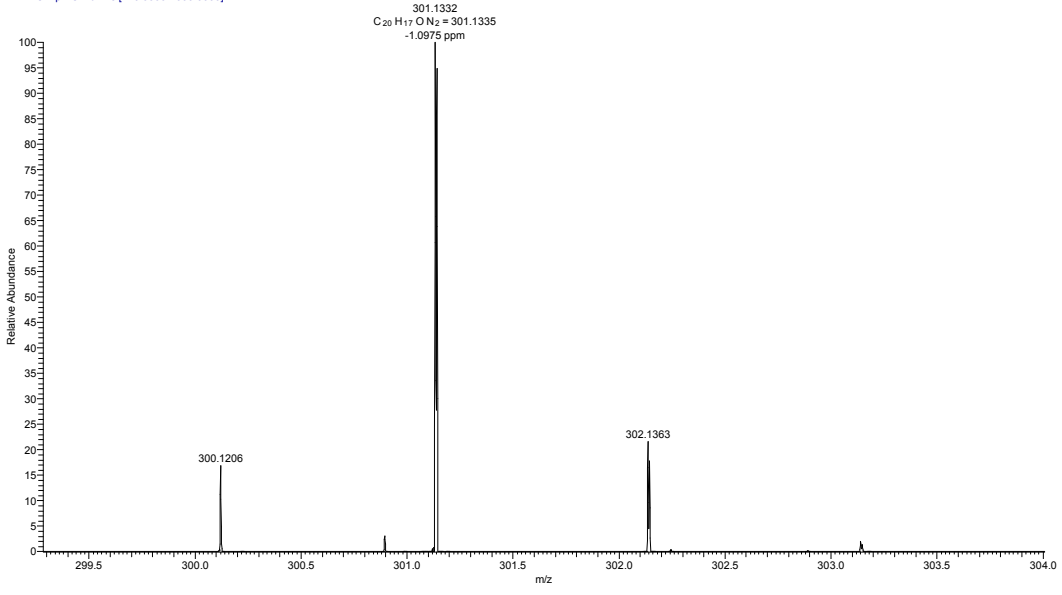

LYC-19300-HNMR.ESP
Chemical Shift (ppm)

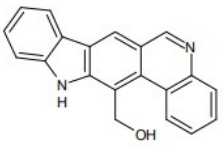
10

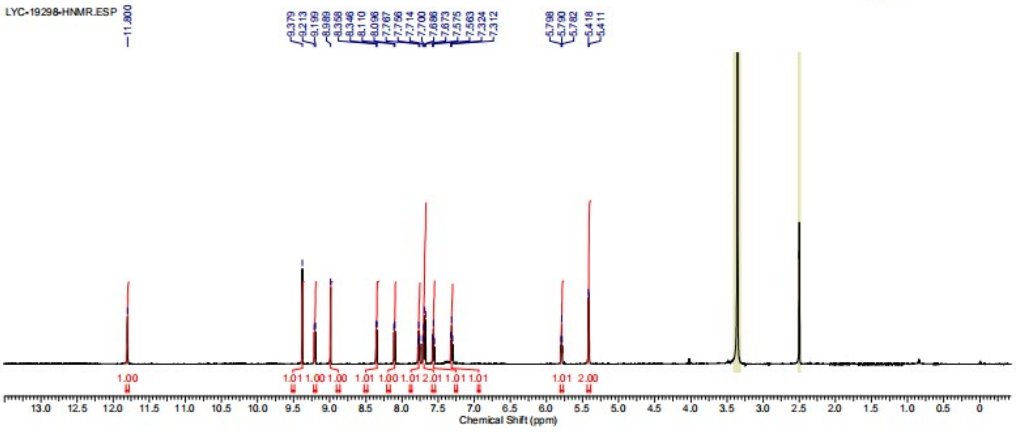

NH
N
H
OH

10

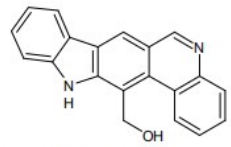

LYC-19300-CNMR.PDF.ESP
Chemical Shift (ppm)

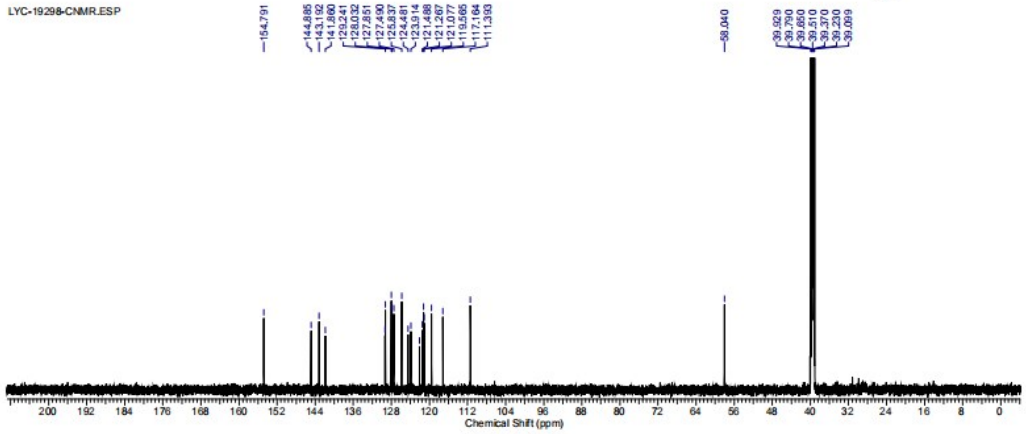

LYC-19300 #28 RT: 0.13 AV: 1 NL: 2.79E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
301.1332
C20 H17 O N2 = 301.1335
-1.0975 ppm
300.1206
302.1363
Relative Abundance
m/z


N
H
OH

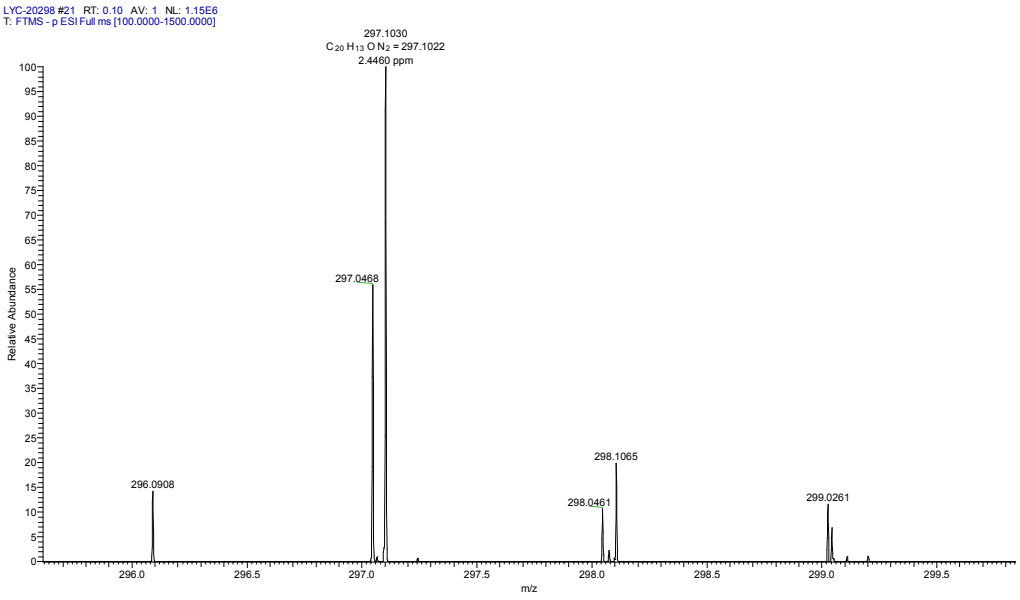

LYC-19298-HNMR.ESP
11.800
9.379
9.213
9.199
8.989
8.358
8.346
8.110
8.090
7.767
7.756
7.714
7.700
7.686
7.673
7.575
7.563
7.324
7.312
5.798
5.790
5.782
5.418
5.411
1.00
1.01
1.00
1.00
1.01
1.00
1.01
2.01
1.01
1.01
1.01
2.00
Chemical Shift (ppm)

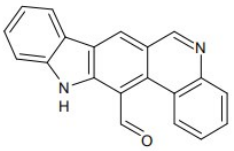
11

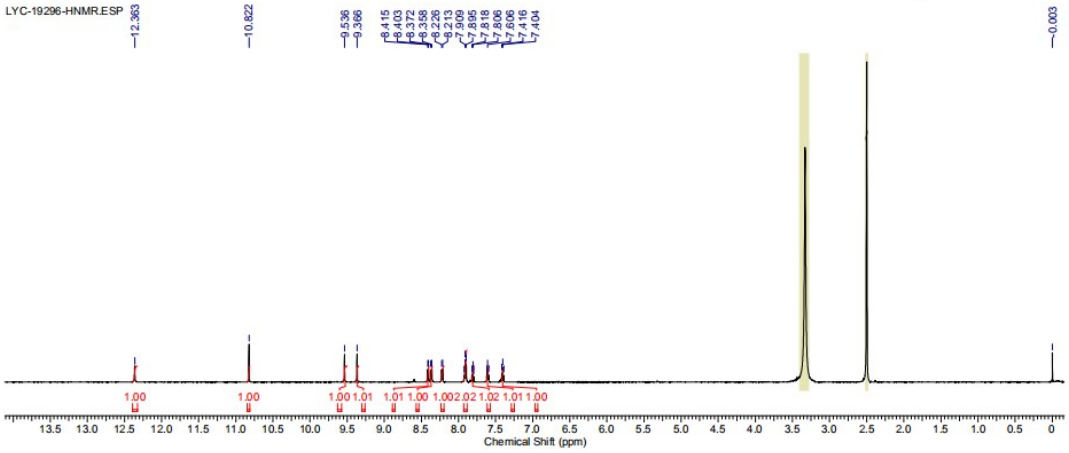

N
H
OH


LYC-19298-CNMR.ESP
154.791
144.885
143.132
141.860
129.241
128.032
127.851
127.490
125.837
124.481
123.914
121.488
121.267
121.077
119.565
117.164
111.393
58.040
39.929
39.790
39.650
39.510
39.370
39.230
39.089
Chemical Shift (ppm)

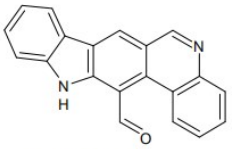
11

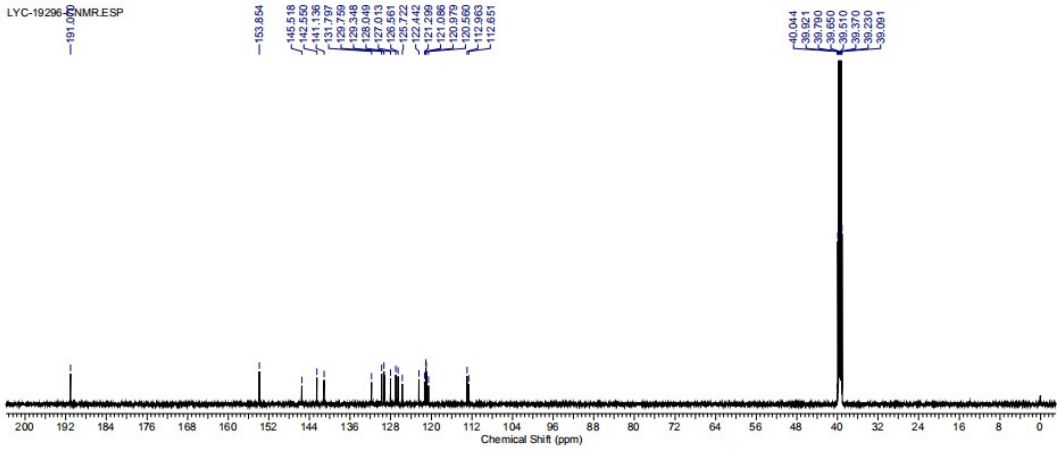

LYC-20298 #21 RT: 0.10 AV: 1 NL: 1.15E6
T: FTMS - p ESI Full ms [100.0000-1500.0000]
297.1030
C20 H13 O N2 = 297.1022
2.4460 ppm
297.0468
296.0908
298.1065
298.0461
299.0261
Relative Abundance
m/z



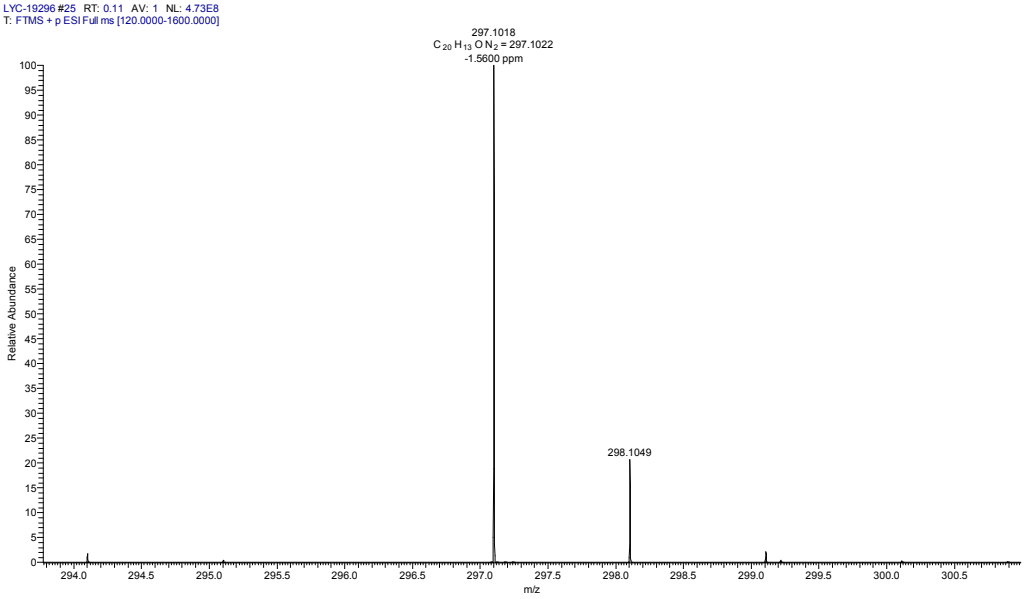

LYC-19296-HNMR.ESP
12.363
10.822
9.536
9.366
8.415
8.403
8.372
8.358
8.226
8.213
7.809
7.795
7.818
7.806
7.606
7.416
7.404
0.003
1.00
1.00
1.00
1.01
1.01
1.00
1.00
2.02
1.02
1.01
1.00
Chemical Shift (ppm)

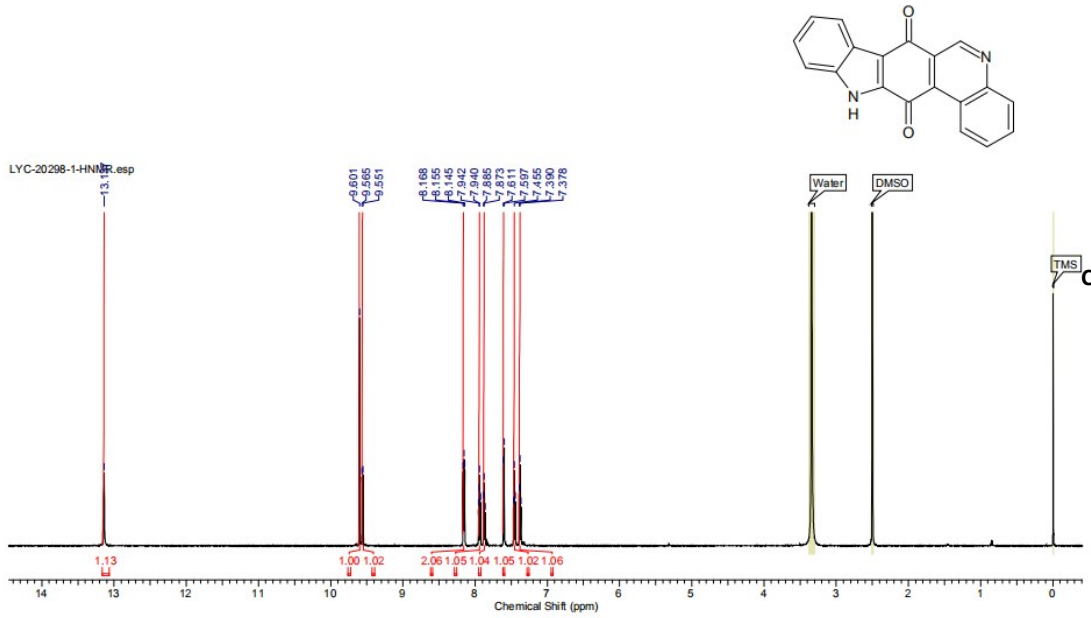
12



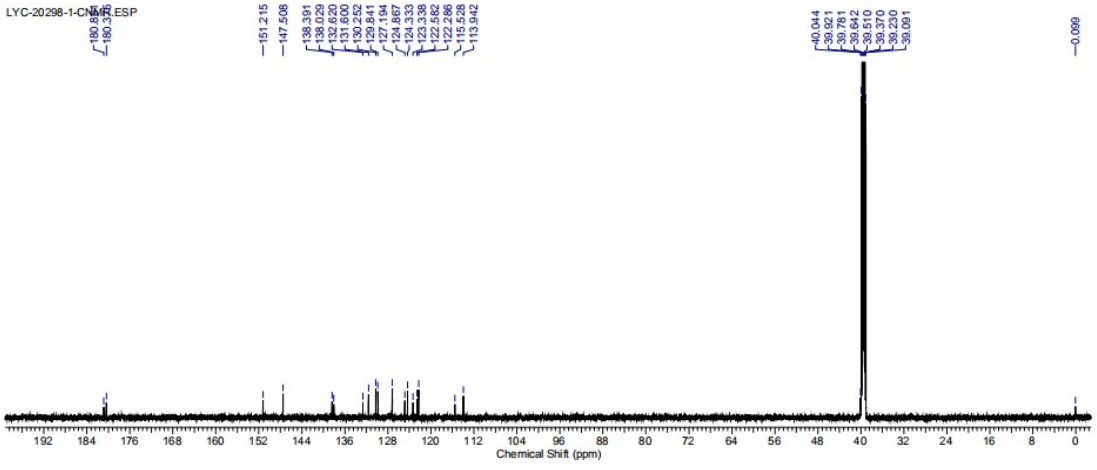

LYC-19296-CNMR.ESP
191.009
153.854
145.518
142.550
141.136
131.797
129.759
129.348
129.049
127.013
126.561
125.722
122.442
121.299
121.086
120.979
120.560
112.883
112.651
40.044
39.921
39.790
39.650
39.510
39.370
39.230
39.091
Chemical Shift (ppm)

12


LYC-19296 #25 RT: 0.11 AV: 1 NL: 4.73E8
T: FTMS + p ESI Full ms [120.0000-1600.0000]
297.1018
C20 H13 O N2 = 297.1022
-1.5600 ppm
298.1049
Relative Abundance
m/z



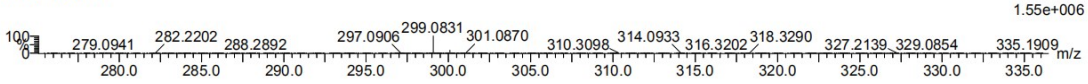
Calothrixin B



Calothrixin B

LYC-19298 106 (0.605)
1: TOF MS ES+

1.55e+006



Minimum:				-1.5				
Maximum:		20.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
299.0831	299.0821	1.0	3.3	15.5	846.9	n/a	n/a	C19 H11 N2 O2