Supporting Information

Catalytic Enantioselective Synthesis of Indolizino[8,7-*b*]indole Alkaloid Derivatives Based on the Tandem Reaction of Tertiary Enamides

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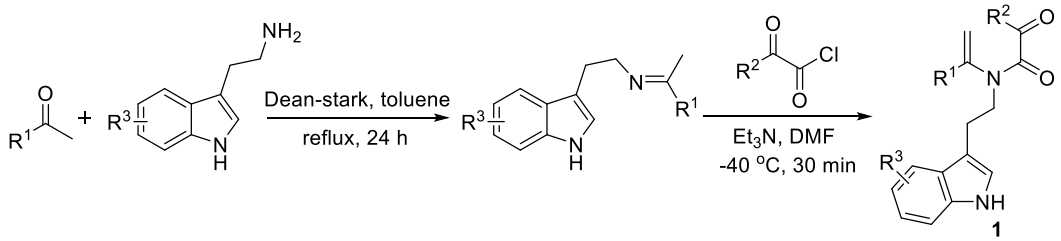
1. General information

All chemicals were dried or purified according to standard procedures prior to use. Flash column chromatography was performed on silica gel (100-200). Reactions were monitored using pre-coated, glass-backed silica gel plates and visualized by means of UV irridation (254 nm) or $KMnO_4$, phosphomolybdic acid, ninhydrine, pancaldi, and *p*-anisaldehyde vanillin. ^1H NMR and ^{13}C NMR spectra were recorded using 400 MHz spectrometers at ambient temperature. ^1H frequency is at 400.13 MHz and ^{13}C frequency is at 100.62 MHz. Chemical shifts are reported in ppm with either tetramethylsilane or the residual solvent resonance used as an internal standard. Abbreviations are used in the description of NMR data as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constant (*J*, Hz). Infrared spectra were recorded using a FT-IR spectrometer with KBr discs in the 4000-400 cm^{-1} region. Mass spectra was measured using mass spectrometers. All yields reported were isolated yields and ee values were determined by HPLC using Daicel ASH or ODH chiral columns eluted with a mixture of isopropanol and hexane at 25 °C.

2. Preparation of Tertiary Enamides



Step 1. The toluene solution of tryptamine (0.5 M) and ketone (0.5 M) was vigorously stirred with Dean-stark at reflux until the conversion of amine to imine was completed. The mixture was concentrated in vacuo to give a crude imine product which was used immediately without further purification.

Step 2. Under argon atmosphere, imine (5 mmol) was dissolved in DMF (10 mL), and then Et_3N (6 mmol) was added. After cooling to -40℃, acyl chloride (6

mmol) was added dropwise during 20 min. The resulting mixture was kept stirring at -40℃ for another 30 min. A saturated aqueous $NaHCO_3$ solution (20 mL) was added to quench the reaction. The mixture was extracted with ethyl acetate (3 × 50 mL), and washed with brine (2 × 50 mL). The organic layer was dried over anhydrous Na_2SO_4, and concentrated under vacuum. The residue was chromatographed on a silica gel column eluted with a mixture of petroleum ether/ethyl acetate (5:1) to give a pure enamide product **1**. The structure of tertiary enamides was fully characterized and the characterization data are listed below.

***N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-phenyl-*N*-(1-phenylvinyl)acetamide (1a)**

White solid (63% yield, 2 steps). m.p. 102-103 ºC; IR (KBr) ν 3399, 1678, 1646, 1626, 1416, 1235, 1205 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.14 (s, 1H), 7.82 (d, *J* = 7.4Hz, 2H), 7.58-7.48 (m, 4H), 7.43-7.33 (m, 6H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.07 (t, *J* = 7.6 Hz, 2H), 5.22 (s, 1H), 4.83 (s, 1H), 3.91 (t, *J* = 7.6 Hz, 2H), 3.10 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 190.4, 167.3, 145.8, 136.2, 134.7, 134.3, 134.0, 129.71, 129.67, 128.89, 128.86, 127.6, 127.4, 122.4, 122.2, 119.5, 118.8, 114.3, 112.3, 111.2, 45.1, 23.3; HRMS (ESI) Calcd. for $C_{26}H_{23}N_2O_2$, $[M+H]^+$ 395.1754. Found: 395.1751.

***N*-(2-(1*H*-indol-3-yl)ethyl)-*N*-(1-(4-bromophenyl)vinyl)-2-oxo-2-phenylacetamid e (1b)**

White solid (66% yield, 2 steps). m.p. 144-146 ºC; IR (KBr) ν 3431, 1678, 1648, 1628, 1421 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 10.10 (s, 1H), 7.85 (d, *J* = 8.2 Hz, 2H), 7.67 (t, *J* = 7.3 Hz, 1H), 7.60 (d, *J* = 8.2 Hz, 2H), 7.54-7.49 (m, 5H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 5.39 (s, 1H), 4.95 (s, 1H), 3.89 (t, *J* = 7.6 Hz, 2H), 3.13 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 191.2, 167.7, 145.6, 137.7, 135.15, 135.09, 134.8, 132.6, 130.2, 130.1, 129.8, 128.5, 123.9, 123.8, 122.2, 119.5, 119.3, 115.6, 112.2, 112.1, 45.7, 24.0; HRMS (ESI) Calcd. for $C_{26}H_{22}N_2O_2Br$, $[M+H]^+$ 473.0859. Found: 473.0858.

***N*-(2-(1H-indol-3-yl)ethyl)-*N*-(1-(4-fluorophenyl)vinyl)-2-oxo-2-phenylacetamide (1c)**

White solid (62% yield, 2 steps). m.p. 116-118 °C; IR (KBr) ν 3409, 3056, 2933, 1680, 1651, 1599, 1506 cm^{-1}; ^{1}H NMR (400MHz, ACETONE-D_6, TMS) δ (ppm) 10.11 (s, 1H), 7.87-7.85 (m, 2H), 7.70-7.65 (m, 1H), 7.64-7.59 (m, 2H), 7.55-7.51 (m, 3H), 7.43-7.40 (m, 1H), 7.25-7.16 (m, 3H), 7.13-7.08 (m, 1H), 7.02-6.98 (m, 1H), 5.32 (s, 1H), 4.92 (s, 1H), 3.88 (t, J = 7.2 Hz, 2H), 3.12 (t, J = 7.6 Hz, 2H); ^{19}F NMR (376MHz, DMSO-d_6) δ (ppm) -113.41 (t, J = 6.0 Hz); ^{13}C NMR (100MHz, ACETONE-D_6, TMS) δ (ppm) 191.2, 167.7, 164.2 (d, J = 246 Hz, 1C), 145.7, 137.6, 135.1, 134.9, 132.2 (d, J = 2.9 Hz, 1C), 130.4 (d, J = 8.6 Hz, 1C), 130.2, 129.7, 128.5, 123.8, 122.2, 119.5, 119.2, 116.3 (d, J = 21.9 Hz, 1C), 114.8, 112.2, 112.1, 45.6, 24.0; HRMS (ESI) Calcd. for $C_{26}H_{21}N_2O_2FNa$, $[M+Na]^+$ 435.1479. Found: 435.1477.

_N_-(2-(1_H_-indol-3-yl)ethyl)-_N_-(1-(4-chlorophenyl)vinyl)-2-oxo-2-phenylacetamide (1d)

White solid (72% yield, 2 steps). m.p. 140-142 °C; IR (KBr) ν 3412, 2930, 1679, 1650, 1488, 1235, 1203 cm^{-1}; ^{1}H NMR (400MHz, ACETONE-D_6, TMS) δ (ppm) 10.11 (s, 1H), 7.86-7.84 (m, 2H), 7.69-7.64 (m, 1H), 7.59-7.49 (m, 5H), 7.46-7.41 (m, 3H), 7.24 (d, J = 2.4Hz, 1H), 7.13-7.09 (m, 1H), 7.02-6.98 (m, 1H), 5.38 (s, 1H), 4.95 (s, 1H), 3.89 (t, J = 7.2 Hz, 2H), 3.12 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100MHz, ACETONE-D_6, TMS) δ (ppm) 191.2, 167.7, 145.4, 137.6, 135.6, 135.1, 134.8, 134.6, 130.2, 129.8, 129.7, 129.5, 128.5, 123.8, 122.2, 119.5, 119.2, 115.5, 112.2, 112.1, 45.7, 24.0; HRMS (ESI) Calcd. for $C_{26}H_{21}N_2O_2ClNa$, $[M+Na]^+$ 451.1184. Found: 451.1186.

_N_-(2-(1_H_-indol-3-yl)ethyl)-_N_-(1-(4-nitrophenyl)vinyl)-2-oxo-2-phenylacetamide (1e)

White solid (79% yield, 2 steps). m.p. 145-147 °C; IR (KBr) ν 3337, 3060, 2940, 1682, 1646, 1595, 1519, 1426 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.21-8.17 (m, 3H), 7.84-7.81 (m, 2H), 7.66-7.58 (m, 3H), 7.50 (d, J = 8.0 Hz, 1H), 7.46-7.42 (m, 2H), 7.37 (d, J = 8.4 Hz, 1H), 7.21-7.17 (m, 1H), 7.10-7.06 (m, 2H), 5.34 (d, J = 0.8 Hz, 1H), 4.98 (d, J = 0.8 Hz, 1H), 3.90 (t, J = 7.2 Hz, 2H), 3.13 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100MHz, ACETONE-D_6, TMS) δ (ppm) 191.2, 167.6,

149.1, 144.8, 142.1, 137.6, 135.3, 134.6, 130.3, 129.8, 129.2, 128.5, 124.5, 123.9, 122.2, 119.5, 119.2, 118.4, 112.2, 112.0, 45.9, 24.0; HRMS (ESI) Calcd. for $C_{26}H_{21}N_3O_4Na$, $[M+Na]^+$ 462.1424. Found: 462.1423.

_N_-(2-(1_H_-indol-3-yl)ethyl)-_N_-(1-(3-bromophenyl)vinyl)-2-oxo-2-phenylacetamide (1f)

White solid (59% yield, 2 steps). m.p. 155-157 °C; IR (KBr) ν 3341, 1670, 1654, 1420, 1238, 1199 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.10 (s, 1H), 7.81 (d, J = 7.8Hz, 2H), 7.62-7.35 (m, 8H), 7.24 (t, J = 7.8 Hz, 1H), 7.19 (t, J = 7.8 Hz, 1H), 7.09 (t, J = 7.1 Hz, 2H), 5.21 (s, 1H), 4.85 (s, 1H), 3.90 (t, J = 7.6 Hz, 2H), 3.10 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 190.3, 167.2, 144.6, 137.0, 136.3, 134.4, 133.8, 132.6, 130.38, 130.36, 129.6, 129.1, 128.9, 127.5, 126.0, 123.0, 122.5, 122.2, 119.6, 118.8, 115.3, 112.1, 111.3, 45.2, 23.3; HRMS (ESI) Calcd. for $C_{26}H_{22}N_2O_2Br$, $[M+H]^+$ 473.0859. Found: 473.0854.

_N_-(2-(1_H_-indol-3-yl)ethyl)-_N_-(1-(3-methoxyphenyl)vinyl)-2-oxo-2-phenylacetamide (1g)

White solid (46% yield, 2 steps). m.p. 109-111 °C; IR (KBr) ν 3345, 3047, 2947, 1674, 1651, 1597, 1425, 1237 cm^{-1}; ^1H NMR (400MHz, ACETONE-D_6, TMS) δ (ppm) 10.08 (s, 1H), 7.85-7.83 (m, 2H), 7.66-7.63 (m, 1H), 7.50 (t, J = 7.6 Hz, 3H), 7.40-7.29 (m, 2H), 7.22 (d, J = 2.4 Hz, 1H), 7.14-7.04 (m, 3H), 7.00-6.91 (m, 2H), 5.33 (s, 1H), 4.91 (s, 1H), 3.88 (t, J = 7.6 Hz, 2H), 3.10 (t, J = 8.0 Hz, 2H); ^{13}C NMR (100MHz, ACETONE-D_6, TMS) δ (ppm) 191.2, 167.7, 160.8, 146.4, 137.6, 137.3, 135.0, 134.9, 130.5, 130.2, 129.7, 128.5, 126.5, 123.7, 122.2, 120.4, 119.5, 119.3, 115.9, 114.9, 113.5, 112.2, 55.6, 45.7, 24.0; HRMS (ESI) Calcd. for $C_{27}H_{24}N_2O_3Na$, $[M+Na]^+$ 447.1679. Found: 447.1676.

_N_-(2-(1_H_-indol-3-yl)ethyl)-2-(3-bromophenyl)-_N_-(1-(4-bromophenyl)vinyl)-2-oxoacetamide (1h)

White solid (68% yield, 2 steps). m.p. 145-147 °C; IR (KBr) ν 3417, 1683, 1644, 1634, 1419, 1226, 1198 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.10 (s, 1H), 7.97 (s, 1H), 7.70 (d, J = 7.8 Hz, 2H), 7.50 (t, J = 7.3 Hz, 3H), 7.36 (t, J = 8.7 Hz, 3H), 7.29 (t, J = 7.8 Hz, 1H), 7.19 (t, J = 7.6 Hz, 1H), 7.13-7.07 (m, 2H), 5.18 (s,

1H), 4.73 (s, 1H), 3.90 (t, *J* = 7.3 Hz, 2H), 3.09 (t, *J* = 7.3 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 188.8, 166.6, 144.9, 137.2, 136.3, 135.6, 133.6, 132.3, 132.1, 132.0, 130.5, 129.0, 128.2, 127.6, 124.1, 123.2, 122.3, 119.6, 118.8, 114.8, 112.1, 111.4, 45.0, 23.3; HRMS (ESI) Calcd. for $C_{26}H_{21}N_2O_2Br_2$, $[M+H]^+$ 552.9944. Found: 552.9941.

_N_-(2-(1_H_-indol-3-yl)ethyl)-2-(4-bromophenyl)-_N_-(1-(4-bromophenyl)vinyl)-2-oxoacetamide (1i)

White solid (66% yield, 2 steps). m.p. 159-160 °C; IR (KBr) ν 3344, 1687, 1650, 1582, 1397, 1220, 1203 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.10 (s, 1H), 7.62 (d, *J* = 8.7 Hz, 2H), 7.55-7.50 (m, 5H), 7.38-7.35 (m, 3H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.11-7.08 (m, 2H), 5.20 (s, 1H), 4.76 (s, 1H), 3.88 (t, *J* = 7.3 Hz, 2H), 3.08 (t, *J* = 7.4 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 189.2, 166.8, 145.0, 136.2, 133.6, 132.7, 132.3, 132.1, 131.0, 129.8, 129.0, 127.5, 124.1, 122.5, 122.4, 119.7, 118.8, 114.7, 112.1, 111.3, 45.1, 23.3; HRMS (ESI) Calcd. For $C_{26}H_{21}N_2O_2Br_2$, $[M+H]^+$ 552.9944. Found: 552.9942.

_N_-(2-(1_H_-indol-3-yl)ethyl)-_N_-(1-(4-bromophenyl)vinyl)-2-oxo-2-(p-tolyl)acetamide (1j)

White solid (54% yield, 2 steps). m.p. 173-175 °C; IR (KBr) ν 3347, 1680, 1647, 1635, 1603, 1425, 1225 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 10.10 (s, 1H), 7.75 (d, *J* = 8.2 Hz, 2H), 7.61-7.58 (m, 2H), 7.51 (t, *J* = 8.7 Hz, 3H), 7.41 (d, *J* = 8.2 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 2.3 Hz, 1H), 7.10 (t, *J* = 7.3 Hz, 1H), 7.00 (t, *J* = 7.3 Hz, 1H), 5.38 (s, 1H), 4.95 (s, 1H), 3.87 (t, *J* = 7.6 Hz, 2H), 3.11 (t, *J* = 7.6 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 190.9, 167.9, 146.2, 145.6, 137.7, 135.2, 132.6, 132.5, 130.4, 130.1, 128.5, 123.9, 123.8, 122.2, 119.5, 119.3, 115.6, 112.2, 112.1, 45.6, 24.0, 21.7; HRMS (ESI) Calcd. For $C_{27}H_{24}N_2O_2Br$, $[M+H]^+$ 487.1016. Found: 487.1013.

_N_-(1-(4-bromophenyl)vinyl)-_N_-(2-(5-chloro-1_H_-indol-3-yl)ethyl)-2-oxo-2-phenylacetamide (1k)

White solid (62% yield, 2 steps). m.p. 158-159 °C; IR (KBr) ν 3420, 1686, 1632, 1448, 1424, 1230, 1204 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.19 (s,

1H), 7.83 (d, *J* = 6.9 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.51 (d, *J* = 8.2 Hz, 2H), 7.46-7.36 (m, 5H), 7.27-7.25 (m, 1H), 7.14 (d, *J* = 1.8 Hz, 1H), 7.12-7.11 (m, 1H), 5.18 (s, 1H), 4.80 (s, 1H), 3.85 (t, *J* = 7.6 Hz, 2H), 3.05 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 190.3, 167.3, 145.0, 134.6, 134.5, 133.9, 133.6, 132.1, 129.7, 129.0, 128.7, 125.4, 124.2, 123.8, 122.6, 118.3, 114.7, 112.3, 112.1, 45.0, 23.2; HRMS (ESI) Calcd. For $C_{26}H_{21}N_2O_2ClBr$, $[M+H]^+$ 507.0469. Found: 507.0466.

_N_-(1-(4-bromophenyl)vinyl)-_N_-(2-(5-methyl-1_H_-indol-3-yl)ethyl)-2-oxo-2-phenyl acetamide (1l)

White solid (70% yield, 2 steps). m.p. 133-135 ^{o}C; IR (KBr) ν 3430, 2942, 1687, 1633, 1424, 1232 cm^{-1}; ^{1}H NMR (400MHz, ACETONE-D_6, TMS) δ (ppm) 9.95 (s, 1H), 7.89-7.87 (m, 2H), 7.70-7.66 (m, 1H), 7.62-7.59 (m, 2H), 7.56-7.49 (m, 4H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.21-7.18 (m, 2H), 6.94-6.92 (m, 1H), 5.37 (s, 1H), 4.96 (s, 1H), 3.86 (t, *J* = 8.0 Hz, 2H), 3.09 (t, *J* = 7.6 Hz, 2H), 2.36 (s, 1H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 191.2, 167.7, 145.6, 136.0, 135.1, 134.8, 132.6, 130.2, 130.1, 129.7, 128.7, 128.2, 123.9, 123.86, 123.80, 118.8, 115.4, 111.9, 111.5, 45.6, 24.0, 21.6; HRMS (ESI) Calcd. For $C_{27}H_{23}N_2O_2BrNa$, $[M+Na]^+$ 509.0835. Found: 509.0834.

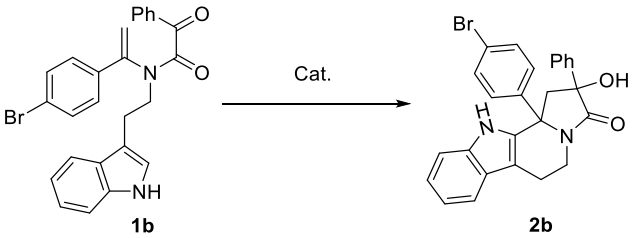
_N_-(1-(4-bromophenyl)vinyl)-_N_-(2-(5-methoxy-1_H_-indol-3-yl)ethyl)-2-oxo-2-phen ylacetamide (1m)

White solid (77% yield, 2 steps). m.p. 143-144 ^{o}C; IR (KBr) ν 3308, 1683, 1640, 1626, 1488, 1218, 1207 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.04 (s, 1H), 7.84 (d, *J* = 8.7 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.49 (d, *J* = 6.9 Hz, 2H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.36 (d, *J* = 6.9 Hz, 2H), 7.23 (d, *J* = 8.7 Hz, 1H), 7.04 (d, *J* = 2.3 Hz, 1H), 6.90 (d, *J* = 2.3 Hz, 1H), 6.85-6.83 (m, 1H), 5.20 (s, 1H), 4.85 (s, 1H), 3.87 (t, *J* = 7.8 Hz, 2H), 3.78 (s, 3H), 3.06 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 190.4, 167.3, 154.1, 145.0, 134.5, 133.9, 133.7, 132.0, 131.4, 129.7, 128.99, 128.97, 127.9, 124.0, 123.2, 114.7, 112.6, 112.1, 111.8, 100.4, 56.0, 45.0, 23.5; HRMS (ESI) Calcd. For $C_{27}H_{24}N_2O_3Br$, $[M+H]^+$ 503.0965. Found: 503.0962.

3. Lewis Acid-Catalyzed Reaction of 1

3.1 Optimization of Reaction Conditions

Table S1. Lewis acid catalyzed reaction of **1b**



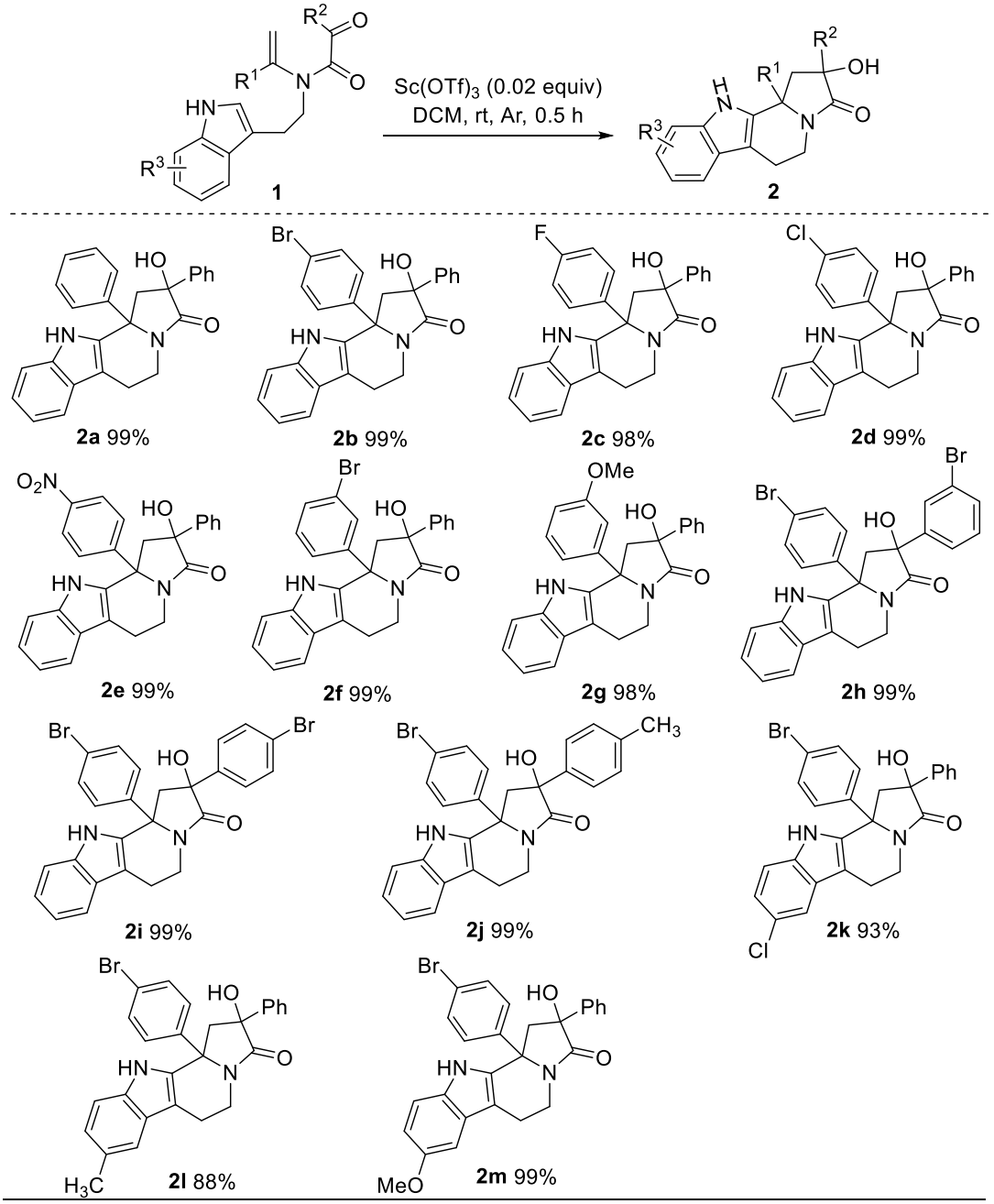
1b **2b**

Entry	Cat. (equiv)	solvent	T (℃)	t (h)	yield(%)[b]
1	$Cu(OTf)_2$ (0.2)	DCM	rt	0.5	69
2	$Sn(OTf)_2$ (0.2)	DCM	rt	0.5	91
3	$Zn(OTf)_2$ (0.2)	DCM	rt	0.5	98
4	$Sc(OTf)_3$ (0.2)	DCM	rt	0.5	99
5	$Sc(OTf)_3$ (0.2)	$CHCl_3$	rt	0.5	86
6	$Sc(OTf)_3$ (0.2)	CH_3CN	rt	0.5	96
7	$Sc(OTf)_3$ (0.2)	Toluene	rt	0.5	99
8	$Sc(OTf)_3$ (0.05)	DCM	rt	0.5	97
9	$Sc(OTf)_3$ (0.02)	DCM	rt	0.5	98
10	$Sc(OTf)_3$ (0.004)	DCM	rt	2	84
11	$Sc(OTf)_3$ (0.001)	DCM	rt	24	mess
12	--	DCM	rt	24	mess

[a] A mixture of **1** (0.5 mmol) and catalyst in dry solvent (5 mL) was stirred under argon protection. [b] Isolated yield.

3.2 Synthesis of Racemic Products 2

Table S2. Lewis Acid Catalyzed Reaction of **1**



3.3 General Procedure for the Synthesis of Racemic Products 2

To a flask (10 mL) equipped with a magnetic stirrer was added **1** (0.5 mmol), $Sc(OTf)_3$ catalyst (2 mol%) and dry CH_2Cl_2 (5 mL) under argon protection. The reaction mixture was stirred at room temperature for 0.5 h and then saturated $NaHCO_3$ aqueous solution (10 mL) was added to quench the reaction. The mixture was extracted with DCM (3×10 mL), and combined organic layer was washed with brine (2×20 mL) and dried over anhydrous Na_2SO_4. The solvents were removed in *vacuo*

and the residue was purified by flash column chromatography (PE:EA = 2:1) to afford products **2**.

4. Optimization of Catalytic Enantioselective Reaction Conditions

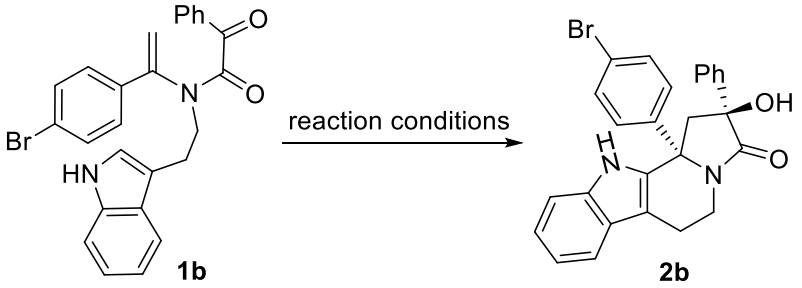


Table S3. Optimization of the reaction catalyzed by different chiral catalyst

Entry	Cat. (0.2 equiv)	Solvent	T (°C)	t (h)	yield[b] (%)	ee[c] (%)
1	**Cat-1**	DCM	35	2	95	9
2	**Cat-2**	DCM	35	2	98	83
3	**Cat-3**	DCM	35	10	76	58
4	**Cat-4**	DCM	35	12	mess	n.d.[d]
5	**L1**/Ti(OiPr)$_4$	DCM	35	2	72	45
6	**L2**/Cu(OTf)$_2$	DCM	35	4	88	-82
7	**L3**/Cu(OTf)$_2$	DCM	35	48	n.d.[d]	0
8	**L4**/Cu(OTf)$_2$	DCM	35	48	n.d.[d]	-55
9	**L5**/Cu(OTf)$_2$	DCM	35	2	94	86
10	**L6**/Cu(OTf)$_2$	DCM	35	12	n.d.[d]	6
11	**L7**/Cu(OTf)$_2$	DCM	35	10	83	80

[a] A mixture of **1b** (0.2 mmol) and catalyst (20 mol%) in dry solvent (5 mL) was stirred at 35 °C under argon protection. [b] Isolated yield. [c] Measured by chiral-phase HPLC. [d] Not determined.

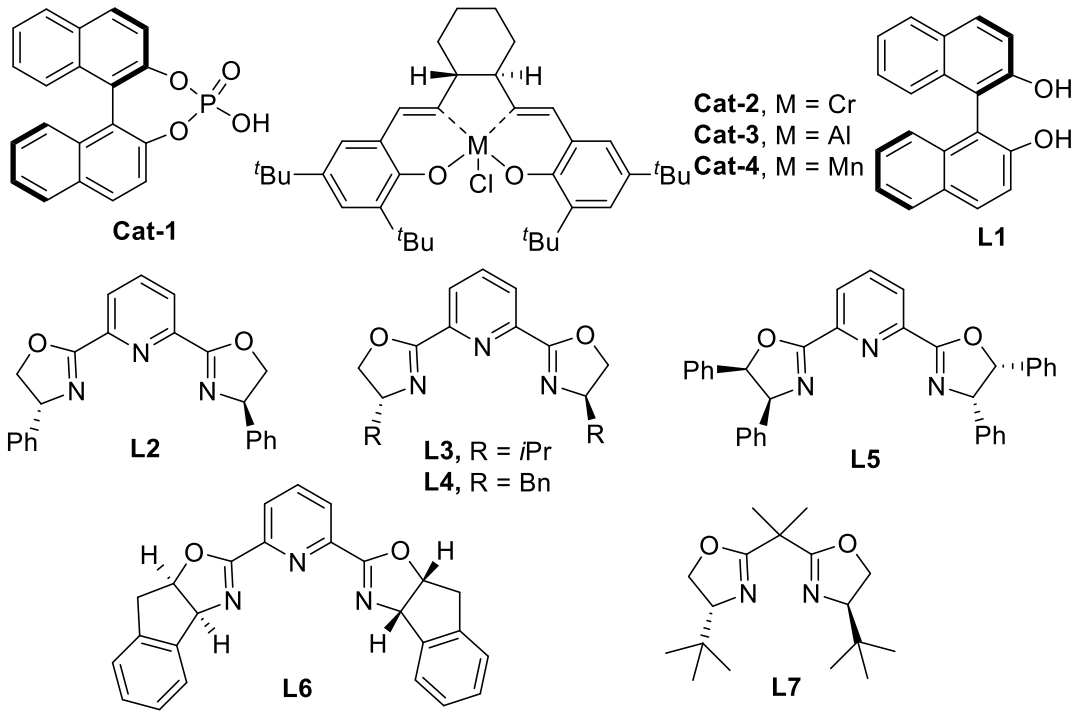


Figure S1. The structure of chiral catalyst

Table S4. Optimization of reaction temperature, solvent, and additive

Entry	Cat. (0.2 equiv)	Additive (2.0 equiv)	Solvent	T (°C)	t (h)	yield[b] (%)	ee[c] (%)
1	**L5**/Cu(OTf)$_2$	--	DCM	rt	10	83	80
2	**L5**/Cu(OTf)$_2$	--	DCM	0	14	76	77
3	**L5**/Cu(OTf)$_2$	HFIP	DCM	35	2	90	83
4	**L5**/Cu(OTf)$_2$	--	toluene	rt	12	93	84
5	**L5**/Cu(OTf)$_2$	--	toluene	35	10	90	87
6	**L5**/Cu(OTf)$_2$	--	toluene	50	8	88	89
7	**L5**/Cu(OTf)$_2$	--	toluene	65	3	86	89
8	**L5**/Cu(OTf)$_2$	--	toluene	80	1	90	87

[a] A mixture of **1b** (0.2 mmol), catalyst (20 mol%) or additive in dry solvent (5 mL) was stirred at different temperature under argon protection.

[b] Isolated yield. [c] Measured by HPLC.

Table S5. Optimization of reaction temperature, solvent, and additive

Entry	Cat. (0.2 equiv)	Additive (equiv)	Solvent	T (^oC)	t (h)	yield[b] (%)	ee[c] (%)
1	**L5**/$Cu(OTf)_2$	--	DCE	50	1	89	76
2	**L5**/$Cu(OTf)_2$	--	p-Xylene	50	10	89	88
3	**L5**/$Cu(OTf)_2$	--	Benzene*	50	10	88	91
4	**L5**/$Cu(OTf)_2$	H_2O (1.0)	Benzene	50	10	77	89
5	**L5**/$Cu(OTf)_2$	H_2O (2.0)	Benzene	50	10	76	94
6	**L5**/$Cu(OTf)_2$	Saturated with H_2O	Benzene	50	10	82	97
7	**L5**/$Cu(OTf)_2$	HFIP (2.0)	Benzene	50	6	80	94
8	**L5**/$Cu(OTf)_2$	--	Benzene	50	10	88	98

[a] A mixture of **1b** (0.2 mmol), catalyst (20 mol%) or additive in dry solvent (5 mL) was stirred at different temperature under argon protection. [b] Isolated yield. [c] Measured by HPLC.

5. Synthesis of Enantioenriched Pyrrolo[2,1-*a*]isoquinoline Derivatives

5.1. General procedure for catalytic enantioselective reaction of tertiary enamides

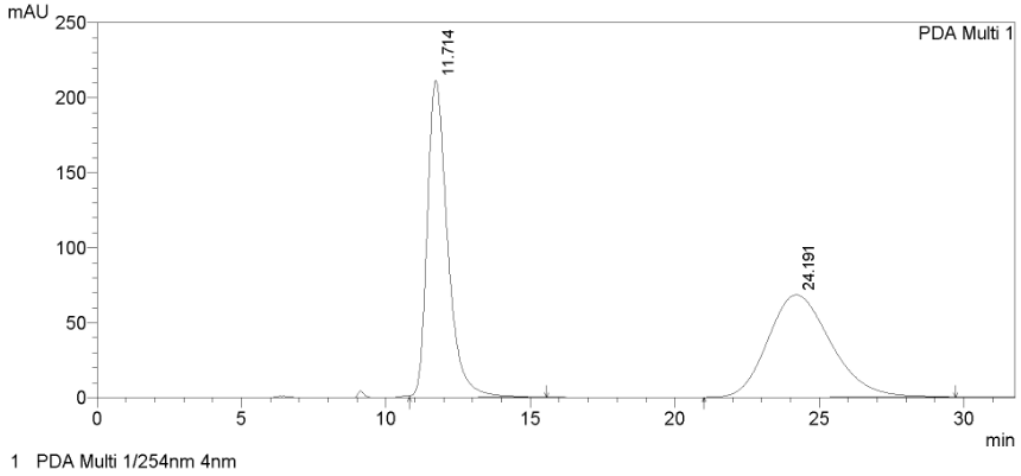
Under argon protection, a mixture of $Cu(OTf)_2$ (14.5 mg, 0.04 mmol) and 2,6-*bis*((4*R*,5*S*)-4,5-diphenyl-4,5-dihydrooxazol-2-yl)pyridine (21.0 mg, 0.04 mmol) in dry benzene (3 mL) was stirred at 50 ^oC for 2 h to give a light blue solution of Cu-Pybox complex. A solution of tertiary enamide **1** (0.2 mmol) in dry benzene (2 mL) was added through a syringe. After stirring for 10 h, the reaction was quenched by adding an aqueous $NaHCO_3$ solution (5%, 10 mL). The mixture was then extracted with CH_2Cl_2 (3×10 mL), and combined organic layer was washed with brine (2 × 20 mL). The organic layer was dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was chromatographed on a silica gel column eluted with a mixture of petroleum ether and ethyl acetate (2:1) to give pure product **2**.

5.3 Characterization Data of Products

(2*S*,11*bS*)-2-hydroxy-2,11b-diphenyl-1,2,5,6,11,11b-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2a)

White solid (68 mg, 86% yield). m.p. 150-152 °C; $[\alpha]_{25}^{D}$ = 112.7° (*c* = 0.5, $CHCl_3$); ee = 97% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3297, 1679, 1655, 1445 cm^{-1}; ^1H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.45 (s, 1H), 7.44-7.39 (m, 4H), 7.34-7.23 (m, 8H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.01 (t, *J* = 7.3 Hz, 1H), 6.03 (s, 1H), 4.31-4.28 (m, 1H), 3.16 (d, *J* = 14.2 Hz, 1H), 3.04-2.98 (m, 1H), 2.95-2.88 (m, 1H), 2.76-2.67 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.6, 144.0, 143.7, 136.3, 136.1, 128.6, 128.0, 127.4, 127.2, 126.3, 125.9, 125.8, 121.4, 118.8, 118.1, 111.3, 106.3, 78.2, 62.8, 51.8, 36.3, 19.9; HRMS (ESI) Calcd. for $C_{26}H_{21}N_2O_2$, $[M-H]^+$ 393.1608. Found: 393.1602.

Chiral HPLC analysis of racemic **2a**.

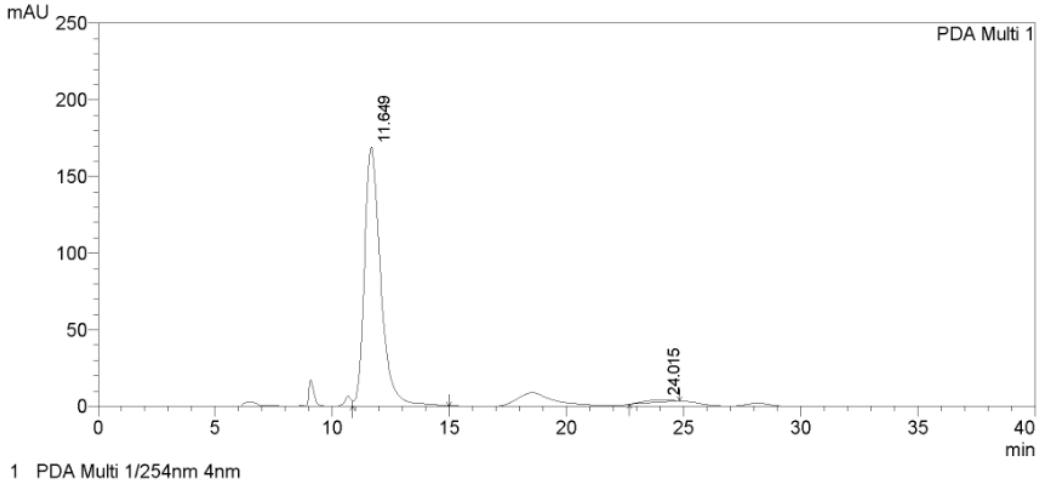


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.714	10352770	211176	50.015	75.581
2	24.191	10346369	68226	49.985	24.419
Total		20699139	279402	100.000	100.000

Chiral HPLC analysis of **2a** from asymmetric reaction.



PeakTable

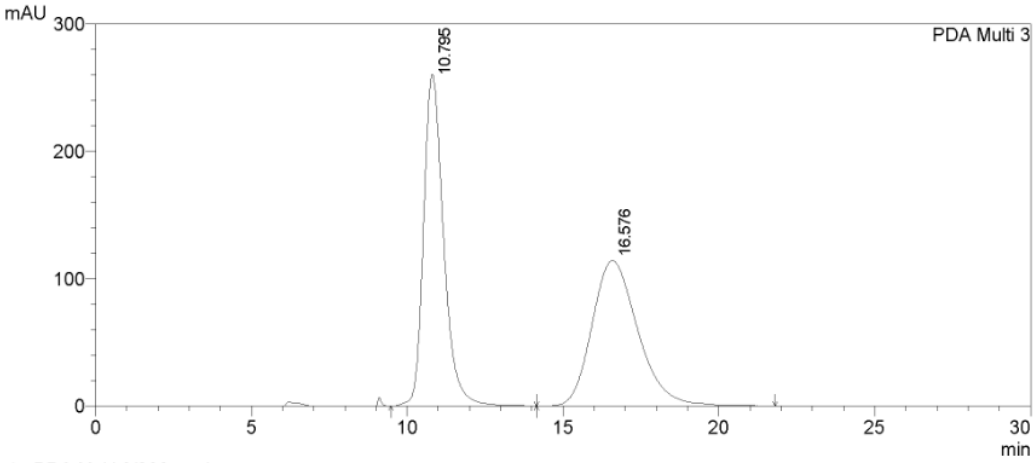
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.649	8192379	169126	98.430	99.065
2	24.015	130688	1596	1.570	0.935
Total		8323068	170723	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-bromophenyl)-2-hydroxy-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2b)

White solid (83 mg, 88% yield). m.p. 161-162 ºC; $[\alpha]_{25}^{D}$ = 164.0° (*c* = 0.5, $CHCl_3$); ee = 99% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3303, 1682, 1489, 1446, 1425, 1396 cm^{-1}; ^{1}H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.46 (s, 1H), 7.52 (d, *J* = 8.7 Hz, 1H), 7.44-7.39 (m, 4H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.28-7.25 (m, 3H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.05 (s, 1H), 3.14 (d, *J* = 14.2 Hz, 1H), 3.04-2.97 (m, 1H), 2.94-2.85 (m, 1H), 2.70 (d, *J* = 14.7 Hz, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.7, 143.5, 143.4, 136.1, 135.7, 131.5, 128.2, 128.0, 127.3, 126.3, 125.8, 121.5, 120.7, 118.8, 118.1, 111.4, 106.5, 78.2, 62.5, 51.5, 36.3, 19.9; HRMS (ESI) Calcd. for $C_{26}H_{20}N_2O_2Br$, $[M-H]^+$ 471.0713. Found: 471.0705.

Chiral HPLC analysis of racemic **2b**.



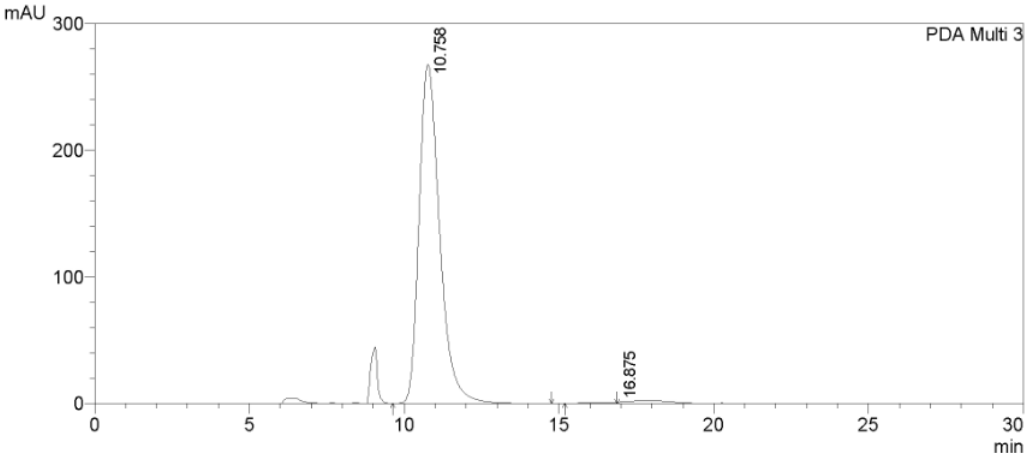
1 PDA Multi 3/280nm 4nm

PeakTable

PDA Ch3 280nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.795	11689112	260564	49.964	69.537
2	16.576	11705805	114149	50.036	30.463
Total		23394917	374713	100.000	100.000

Chiral HPLC analysis of **2b** from asymmetric reaction.



1 PDA Multi 3/280nm 4nm

峰表

PDA Ch3 280nm 4nm

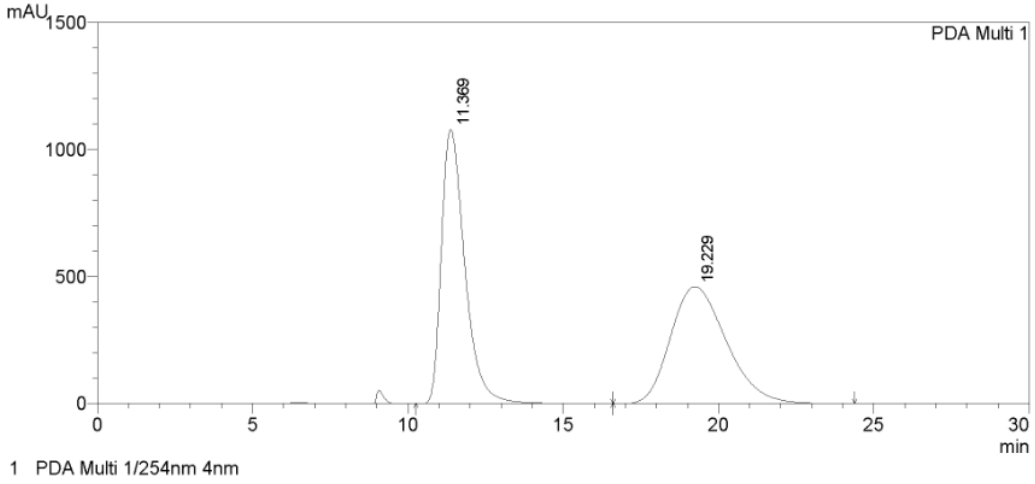
峰#	保留时间	面积	高度	面积 %	高度 %
1	10.758	12434047	268042	99.470	99.616
2	16.875	66192	1033	0.530	0.384
Total		12500239	269075	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-fluorophenyl)-2-hydroxy-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2c)

White solid (78 mg, 94% yield). m.p. 165-167 ^{o}C; $[\alpha]_{25}^{D}$ = 109.3° (*c* = 0.5, $CHCl_3$); ee = 97% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3290, 3053, 2940, 1653, 1509, 1446, 1233 cm^{-1}; ^{1}H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.45 (s, 1H), 7.44-7.39 (m, 4H), 7.35- 7.24 (m, 5H), 7.17-7.11 (m, 3H), 7.02 (t, *J* = 7.2 Hz, 1H), 6.05 (d, *J* = 5.2 Hz, 1H), 4.31-4.28

(m, 1H), 3.17-3.12 (m, 1H), 3.04-2.87 (m, 2H), 2.75-2.68 (m, 2H); ^{19}F NMR (376MHz, DMSO-d_6) δ (ppm) -115.12 (d, J = 0.54 Hz); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.7, 161.2 (d, J = 243 Hz, 1C), 143.6, 140.1, 136.1 (d, J = 4.8 Hz, 1C), 128.1 (d, J = 8.6 Hz, 1C), 128.0, 127.3, 126.3, 125.8, 121.5, 118.8, 118.1, 115.3 (d, J = 21.9 Hz, 1C), 111.3, 106.4, 78.2, 62.4, 51.7, 36.2, 19.9; HRMS (ESI) Calcd. for $C_{26}H_{21}N_2O_2FNa$, $[M+Na]^+$ 435.1479. Found: 435.1477.

Chiral HPLC analysis of racemic **2c**.

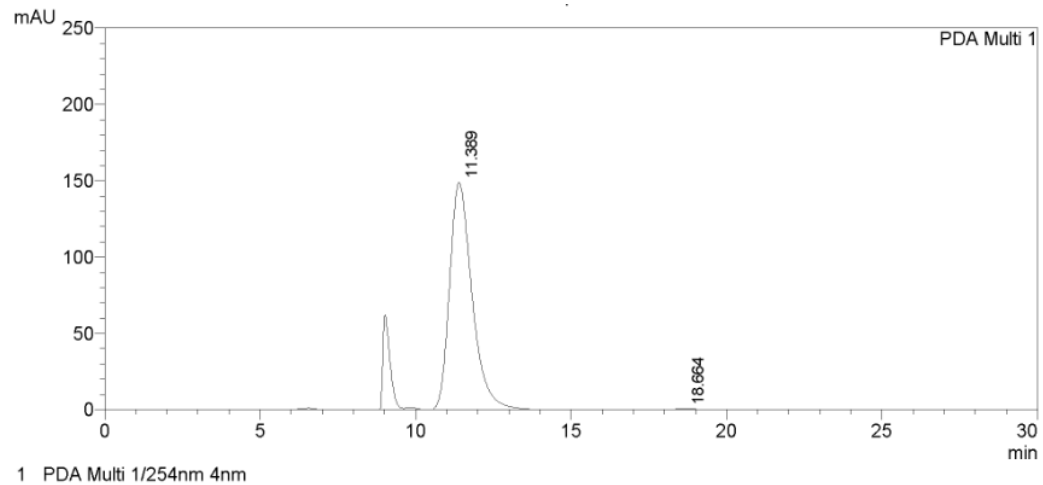


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.369	56939055	1080654	49.567	70.053
2	19.229	57934312	461978	50.433	29.947
Total		114873367	1542632	100.000	100.000

Chiral HPLC analysis of **2c** from asymmetric reaction.



PeakTable

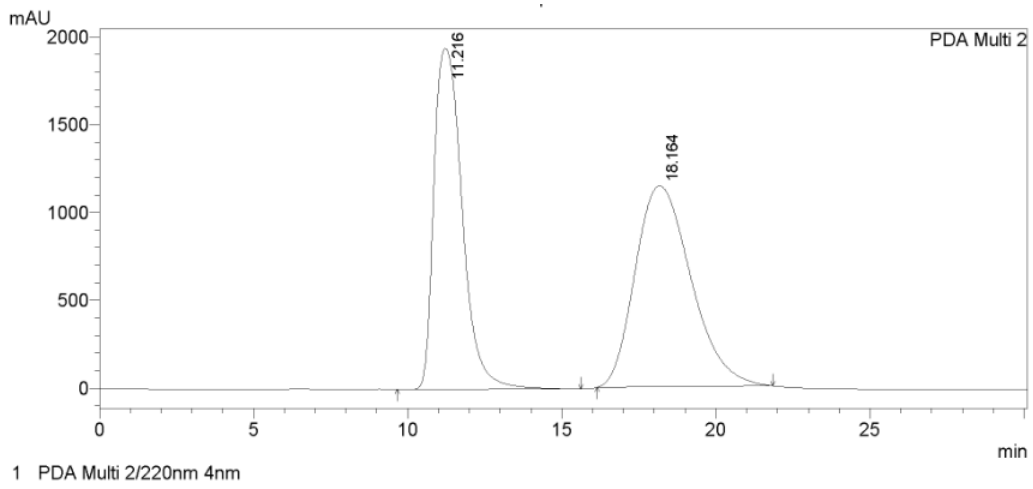
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.389	7707695	149690	98.420	99.148
2	18.664	123747	1286	1.580	0.852
Total		7831442	150976	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-chlorophenyl)-2-hydroxy-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2d)

White solid (79 mg, 92% yield). m.p. 161-163 ^{o}C; $[\alpha]_{25}^{D}$ = 144.0° (*c* = 0.5, $CHCl_3$); ee = 96% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3303, 3053, 2938, 2851, 1651, 1492, 1446, 1432 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 8.17 (s, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.47-7.45 (m, 2H), 7.38- 7.29 (m, 4H), 7.25-7.21 (m, 1H), 7.19-7.14 (m, 3H), 7.04-7.01 (m, 2H), 4.46-4.41 (m, 1H), 3.19-3.00 (m, 3H), 2.97 (d, *J* = 14.0 Hz, 1H), 2.78-2.73 (m, 1H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 174.8, 142.5, 141.4, 136.4, 134.5, 134.2, 128.9, 128.4, 128.3, 126.8, 125.8, 122.9, 120.3, 118.9, 111.5, 109.5, 79.5, 63.1, 50.5, 36.6, 20.4; HRMS (ESI) Calcd. for $C_{26}H_{21}N_2O_2ClNa$, $[M+Na]^+$ 451.1184. Found: 451.1183.

Chiral HPLC analysis of racemic **2d**.

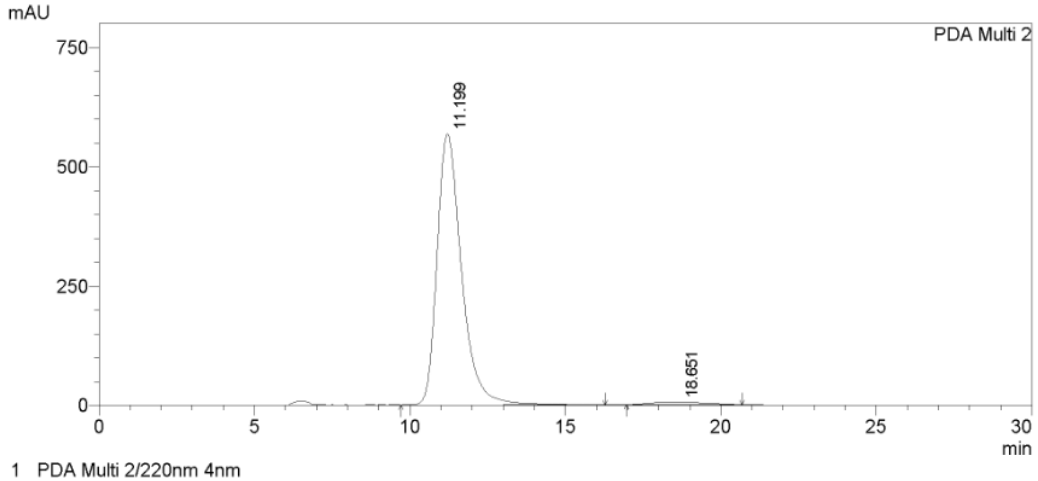


PeakTable

PDA Ch2 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.216	124240103	1943274	46.205	62.909
2	18.164	144645931	1145767	53.795	37.091
Total		268886034	3089041	100.000	100.000

Chiral HPLC analysis of **2d** from asymmetric reaction.



1 PDA Multi 2/220nm 4nm

PeakTable

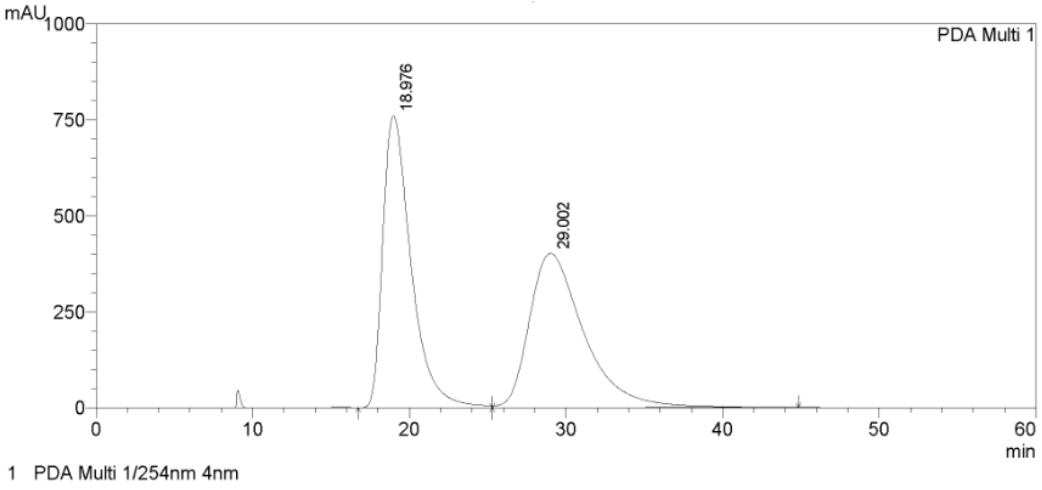
PDA Ch2 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.199	31125337	567758	97.913	98.980
2	18.651	663327	5853	2.087	1.020
Total		31788664	573611	100.000	100.000

(2*S*,11*bS*)-2-hydroxy-11*b*-(4-nitrophenyl)-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2e)

White solid (63 mg, 71% yield). m.p. 182-183 ^{o}C; $[\alpha]_{25}^{D}$ = 64.7° (*c* = 0.5, $CHCl_3$); ee = 99% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3304, 3059, 1683, 1520, 1350 cm^{-1}; ^{1}H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.59 (s, 1H), 8.18 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 2H), 7.46-7.40 (m, 4H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.27-7.23 (m, 1H), 7.18-7.14 (m, 1H), 7.03 (t, *J* = 7.2 Hz, 1H), 6.15 (s, 1H), 4.41-4.36 (m, 1H), 3.20 (d, *J* = 14.0 Hz, 1H), 3.10-3.02 (m, 1H), 2.96-2.88 (m, 1H), 2.77-2.69 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.8, 151.1, 146.6, 143.1, 136.3, 134.9, 128.0, 127.4, 127.3, 126.2, 125.8, 123.8, 121.7, 119.0, 118.2, 111.5, 106.9, 78.1, 62.7, 51.3, 36.5, 19.8; HRMS (ESI) Calcd. for $C_{26}H_{21}N_3O_4Na$, $[M+Na]^+$ 462.1424. Found: 462.1423.

Chiral HPLC analysis of racemic **2e**.

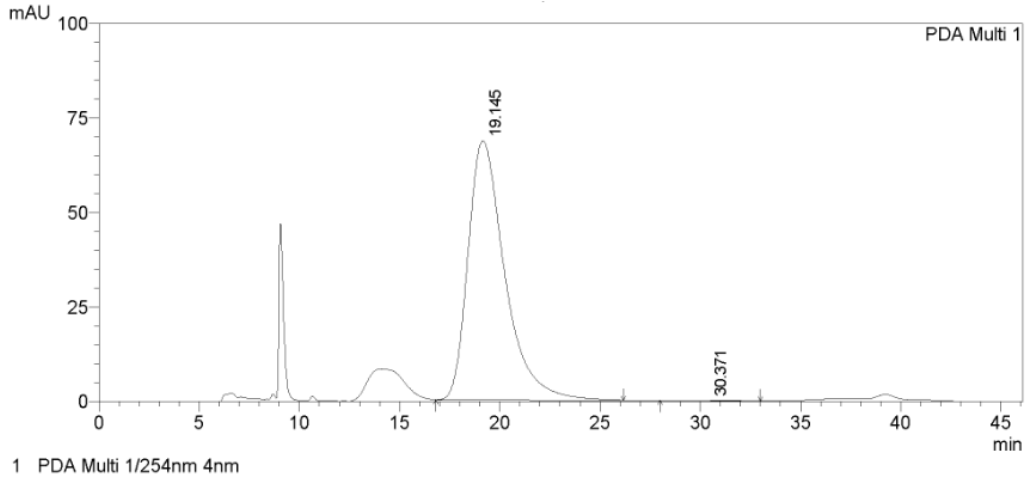


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.976	95658997	761239	49.618	65.469
2	29.002	97132798	401510	50.382	34.531
Total		192791795	1162749	100.000	100.000

Chiral HPLC analysis of **2e** from asymmetric reaction.



PeakTable

PDA Ch1 254nm 4nm

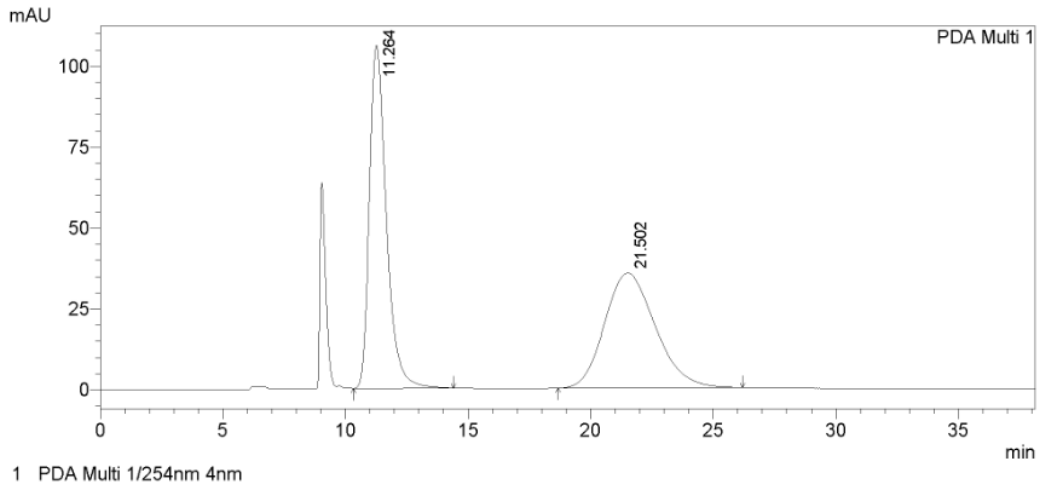
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.145	8742255	68601	99.654	99.739
2	30.371	30371	179	0.346	0.261
Total		8772626	68781	100.000	100.000

(2*S*,11*bS*)-11*b*-(3-bromophenyl)-2-hydroxy-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2f)

White solid (82 mg, 86% yield). m.p. 143-146 ^{o}C; $[\alpha]_{25}^{D}$ = 115.3° (*c* = 0.5, $CHCl_3$); ee = 99% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3301, 1681, 1661, 1446, 1418 cm^{-1}; ^{1}H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.50 (s, 1H), 7.47-7.24 (m, 11H), 7.14 (t, *J* = 7.8 Hz, 1H), 7.03 (t, *J* = 7.6 Hz, 1H), 6.08 (s, 1H), 4.35-4.32 (m, 1H), 3.12 (d, *J* = 13.8 Hz, 1H),

3.07-3.00 (m, 1H), 2.94-2.85 (m, 1H), 2.76-2.70 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.3, 146.2, 143.0, 135.9, 135.2, 130.6, 130.1, 128.5, 127.7, 127.1, 125.9, 125.6, 124.6, 121.6, 121.3, 118.6, 117.9, 111.1, 106.3, 77.8, 62.1, 51.2, 36.0, 19.6; HRMS (ESI) Calcd. for $C_{26}H_{20}N_2O_2Br$, $[M-H]^+$ 473.0688. Found: 473.0684.

Chiral HPLC analysis of racemic **2f**.

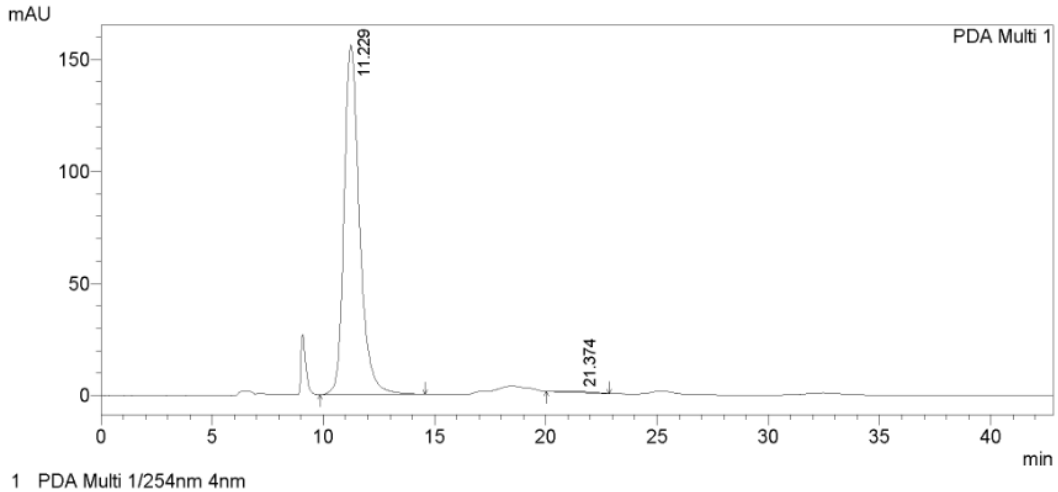


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.264	5069283	105850	49.855	74.901
2	21.502	5098678	35469	50.145	25.099
Total		10167961	141319	100.000	100.000

Chiral HPLC analysis of **2f** from asymmetric reaction.



PeakTable

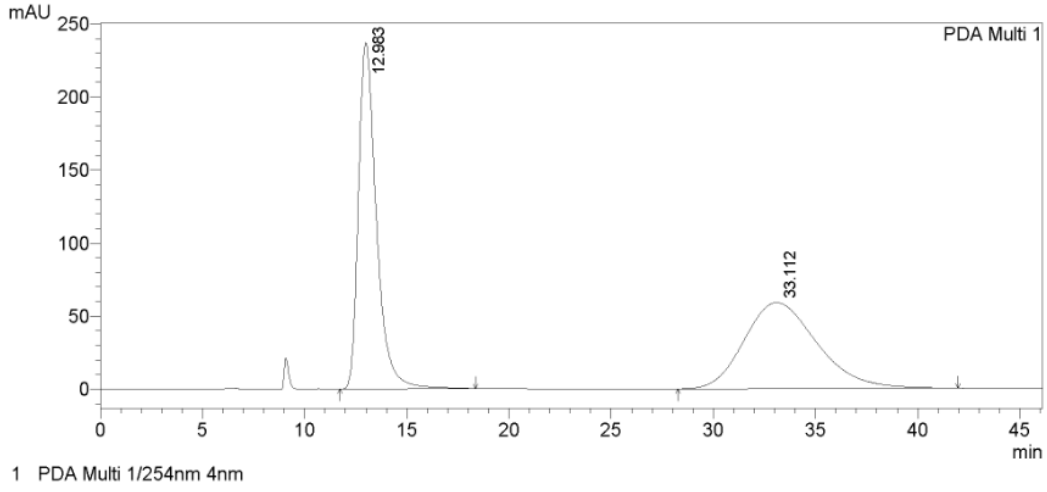
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.229	7516319	155875	99.444	99.670
2	21.374	42030	517	0.556	0.330
Total		7558350	156392	100.000	100.000

(2*S*,11*bS*)-2-hydroxy-11*b*-(3-methoxyphenyl)-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2g)

White solid (71 mg, 83% yield). m.p. 146-148 ^{o}C; $[\alpha]_{25}^{D}$ = 80.0° (*c* = 0.5, $CHCl_3$); ee = 98% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3331, 3058, 2937, 2847, 1655, 1600, 1492, 1446, 1262 cm^{-1}; ^{1}H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.45 (s, 1H), 7.44-7.40 (m, 4H), 7.35-7.32 (m, 2H), 7.28-7.23 (m, 2H), 7.15-7.11 (m, 1H), 7.04-7.00 (m, 1H), 7.89-7.78 (m, 3H), 6.05 (s, 1H), 5.76 (s, 1H), 4.34-4.30 (m, 1H), 3.15-3.02 (m, 2H), 2.95-2.86 (m, 1H), 2.78-2.68 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.5, 159.3, 145.6, 143.8, 136.2, 136.1, 129.7, 128.0, 127.3, 126.2, 125.8, 121.4, 118.8, 118.1, 112.4, 112.2, 111.3, 106.3, 78.2, 62.7, 55.0, 51.6, 36.2, 19.9; HRMS (ESI) Calcd. for $C_{27}H_{24}N_2O_3Na$, $[M+Na]^+$ 447.1679. Found: 447.1681.

Chiral HPLC analysis of racemic **2g**.

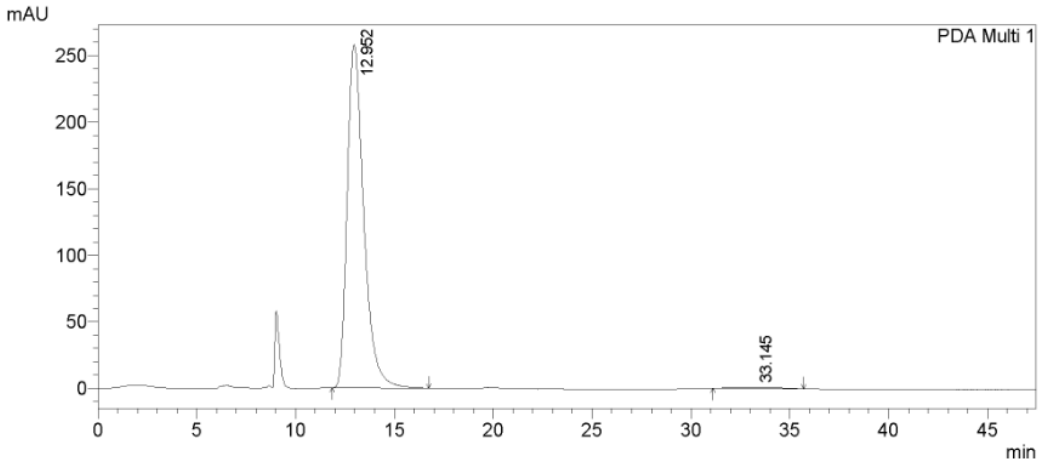


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.983	14495471	236841	49.795	80.098
2	33.112	14614569	58848	50.205	19.902
Total		29110039	295689	100.000	100.000

Chiral HPLC analysis of **2g** from asymmetric reaction.



1 PDA Multi 1/254nm 4nm

PeakTable

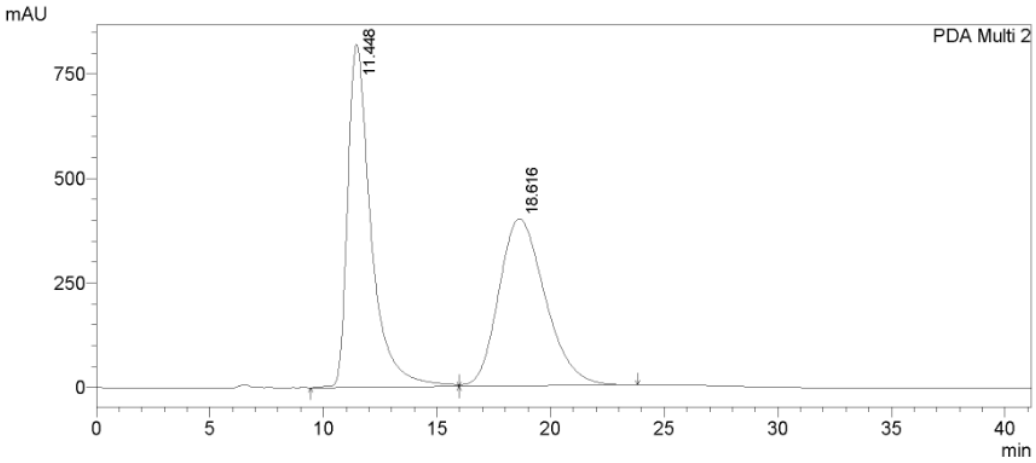
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.952	15043969	258007	99.020	99.635
2	33.145	148966	945	0.980	0.365
Total		15192935	258952	100.000	100.000

(2*S*,11*bS*)-2-(3-bromophenyl)-11*b*-(4-bromophenyl)-2-hydroxy-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2h)

White solid (99 mg, 90% yield). m.p. 184-187 ºC; $[\alpha]_{25}^{D}$ = 118.0° (*c* = 0.5, $CHCl_3$); ee = 99.5% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) *ν* 3322, 1669, 1451, 1433, 1395 cm^{-1}; ^1H NMR (400MHz, DMSO-d_6, TMS) *δ* (ppm) 11.47 (s, 1H), 7.55-7.28 (m, 10H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.02 (t, *J* = 7.3 Hz, 1H), 6.22 (s, 1H), 4.32-4.29 (m, 1H), 3.11 (d, *J* = 14.2 Hz, 1H), 3.07-3.00 (m, 1H), 2.93-2.84 (m, 1H), 2.74-2.68 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) *δ* (ppm) 174.0, 145.8, 143.0, 136.1, 135.5, 131.5, 130.31, 130.26, 128.7, 128.2, 126.2, 125.0, 121.5, 121.4, 120.8, 118.9, 118.2, 111.4, 106.4, 77.7, 62.5, 51.0, 36.4, 19.9; HRMS (ESI) Calcd. for $C_{26}H_{19}N_2O_2Br_2$, $[M-H]^+$ 550.9793. Found: 550.9788.

Chiral HPLC analysis of racemic **2h**.



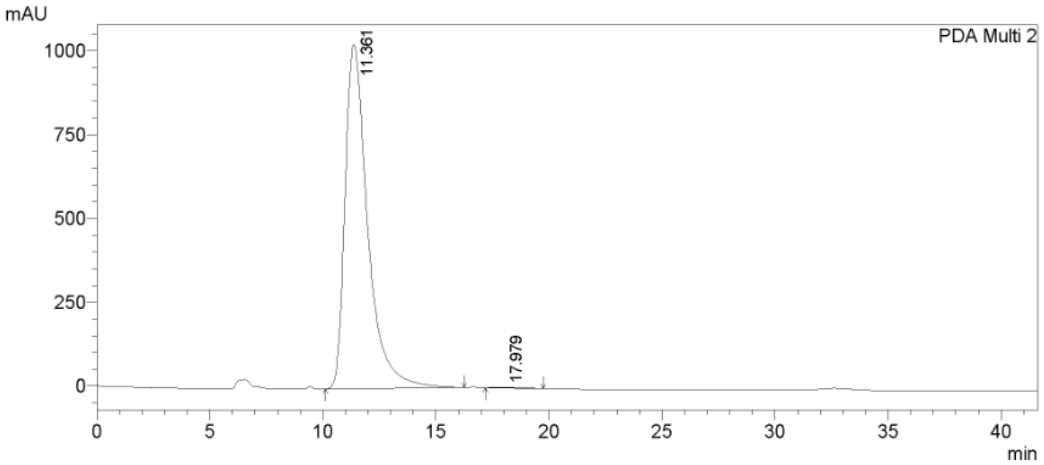
1 PDA Multi 2/220nm 4nm

PeakTable

PDA Ch2 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.448	58570125	819714	50.783	67.221
2	18.616	56763938	399710	49.217	32.779
Total		115334063	1219425	100.000	100.000

Chiral HPLC analysis of **2h** from asymmetric reaction.



1 PDA Multi 2/220nm 4nm

峰表

PDA Ch2 220nm 4nm

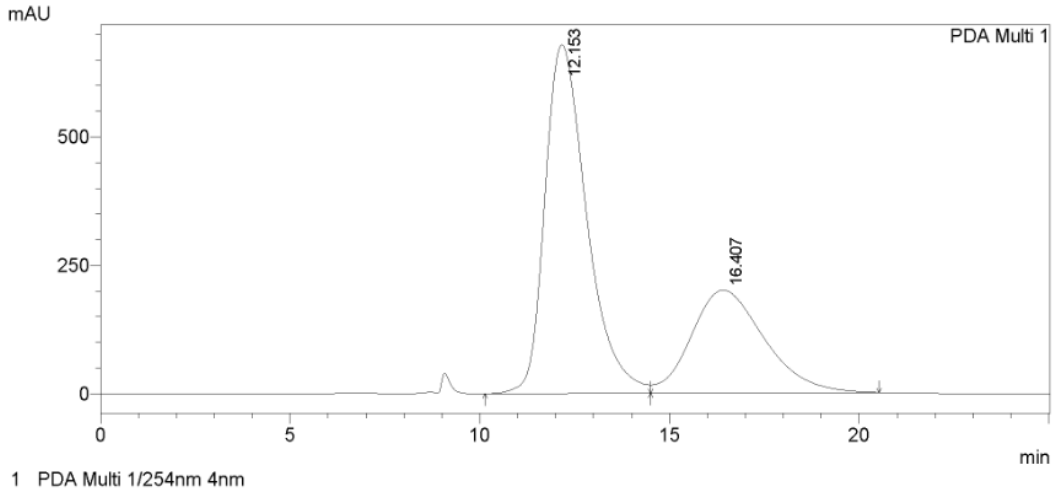
峰#	保留时间	面积	高度	面积 %	高度 %
1	11.361	71639672	1026415	99.735	99.805
2	17.979	190415	2003	0.265	0.195
Total		71830087	1028418	100.000	100.000

(2*S*,11*bS*)-2,11*b*-bis(4-bromophenyl)-2-hydroxy-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2i)

White solid (98 mg, 89% yield). m.p. 286-288 ^{o}C; $[\alpha]_{25}^{D}$ = 145.3° (*c* = 0.5, $CHCl_3$); ee = 97% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3256, 1678, 1487, 1442, 1426, 1395 cm^{-1}; ^{1}H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.46 (s, 1H), 7.53 (d, *J* = 6.9 Hz, 4H), 7.42 (t, *J* = 7.8 Hz, 2H), 7.34 (d, *J* = 8.7 Hz, 2H), 7.25 (d, *J* = 8.7 Hz, 2H), 7.13 (t, *J* = 7.6 Hz,

1H), 7.02 (t, J = 7.6 Hz, 1H), 6.17 (s, 1H), 4.30-4.27 (m, 1H), 3.13 (d, J = 14.2 Hz, 1H), 3.02-2.96 (m, 1H), 2.93-2.86 (m, 1H), 2.72-2.66 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.3, 143.2, 142.8, 136.1, 135.5, 131.5, 130.9, 128.3, 128.2, 126.3, 121.5, 120.8, 120.6, 118.9, 118.2, 111.4, 106.5, 77.9, 62.6, 51.1, 36.4, 19.9; HRMS (ESI) Calcd. for $C_{26}H_{19}N_2O_2Br_2$, $[M-H]^+$ 550.9793. Found: 550.9788.

Chiral HPLC analysis of racemic **2i**.

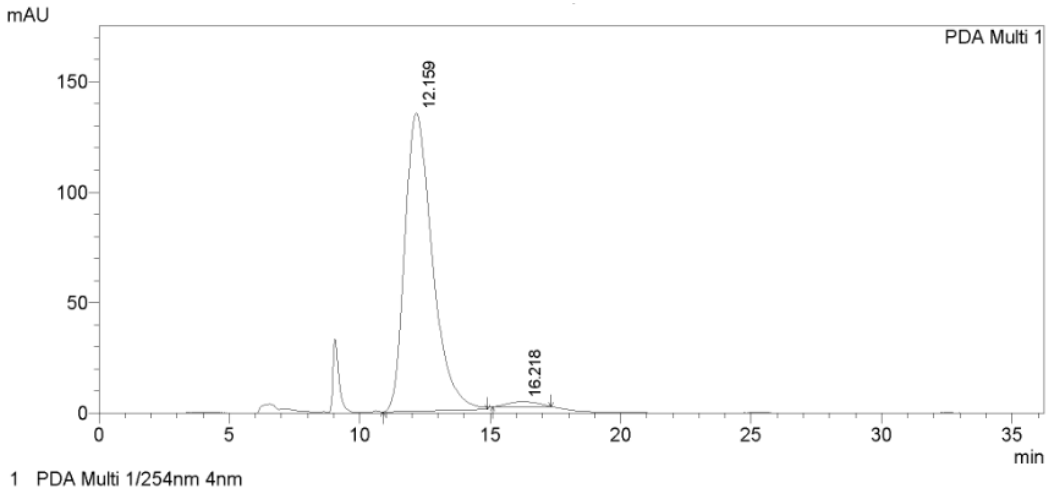


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.153	52186221	679385	66.049	77.229
2	16.407	26824699	200314	33.951	22.771
Total		79010921	879699	100.000	100.000

Chiral HPLC analysis of **2i** from asymmetric reaction.



PeakTable

PDA Ch1 254nm 4nm

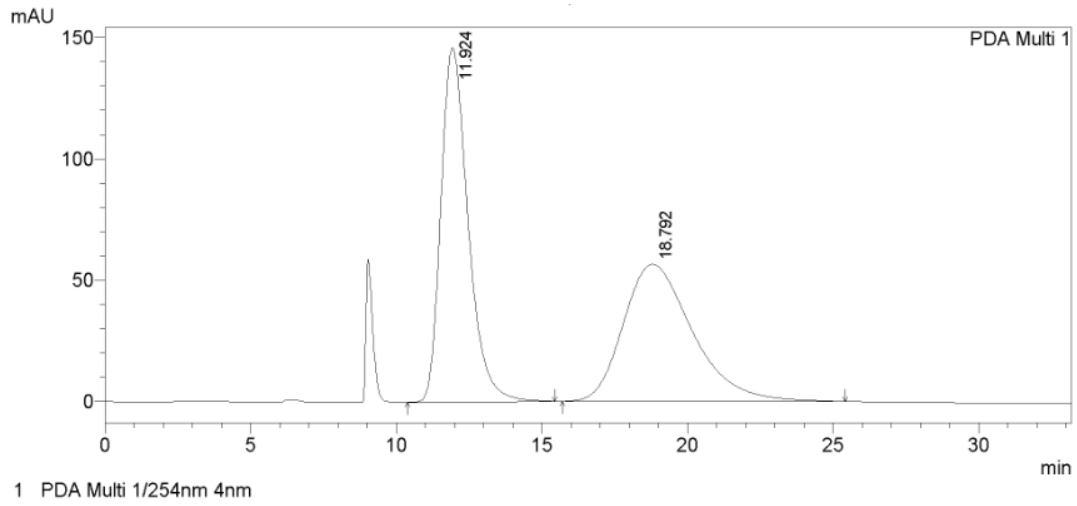
Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.159	9896450	134719	98.258	98.363
2	16.218	175447	2242	1.742	1.637
Total		10071897	136961	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-bromophenyl)-2-hydroxy-2-(p-tolyl)-1,2,5,6,11,11*b*-hexahydro-3

***H*-indolizino[8,7-*b*]indol-3-one (2j)**

White solid (88 mg, 90% yield). m.p. 272-274 ºC; $[\alpha]_{25}^{D}$ = 151.3° (*c* = 0.5, $CHCl_3$); ee = 92% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3358, 3271, 1680, 1441, 1422 cm^{-1}; ^1H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) 11.45 (s, 1H), 7.52 (d, *J* = 8.3 Hz, 2H), 7.42 (t, *J* = 6.6 Hz, 2H), 7.26 (t, *J* = 9.2 Hz, 4H), 7.15-7.11 (m, 3H), 7.02 (t, *J* = 7.6 Hz, 1H), 5.98 (s, 1H), 4.32-4.27 (m, 1H), 3.13 (d, *J* = 14.2 Hz, 2H), 3.01-2.96 (m, 1H), 2.93-2.86 (m, 1H), 2.68 (d, *J* = 13.7 Hz, 2H), 2.28 (s, 3H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.8, 143.4, 140.6, 136.4, 136.1, 135.8, 131.5, 128.5, 128.2, 126.3, 125.7, 121.5, 120.7, 118.8, 118.1, 111.4, 106.5, 78.0, 62.4, 51.5, 36.2, 20.7, 19.9; HRMS (ESI) Calcd. for $C_{27}H_{22}N_2O_2Br$, $[M-H]^+$ 485.0870. Found: 485.0863.

Chiral HPLC analysis of racemic **2j**.

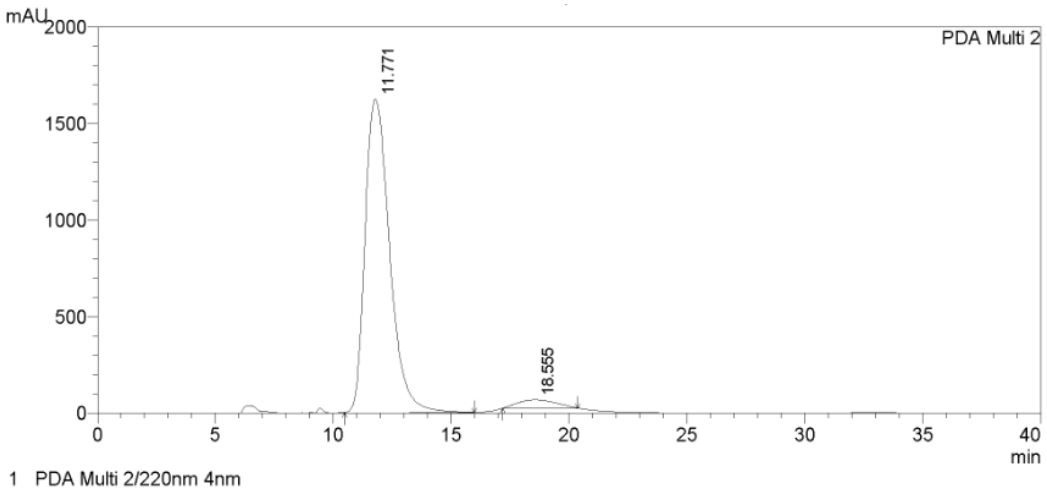


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.924	9475523	145997	50.452	72.105
2	18.792	9305571	56482	49.548	27.895
Total		18781094	202479	100.000	100.000

Chiral HPLC analysis of **2j** from asymmetric reaction.



PeakTable

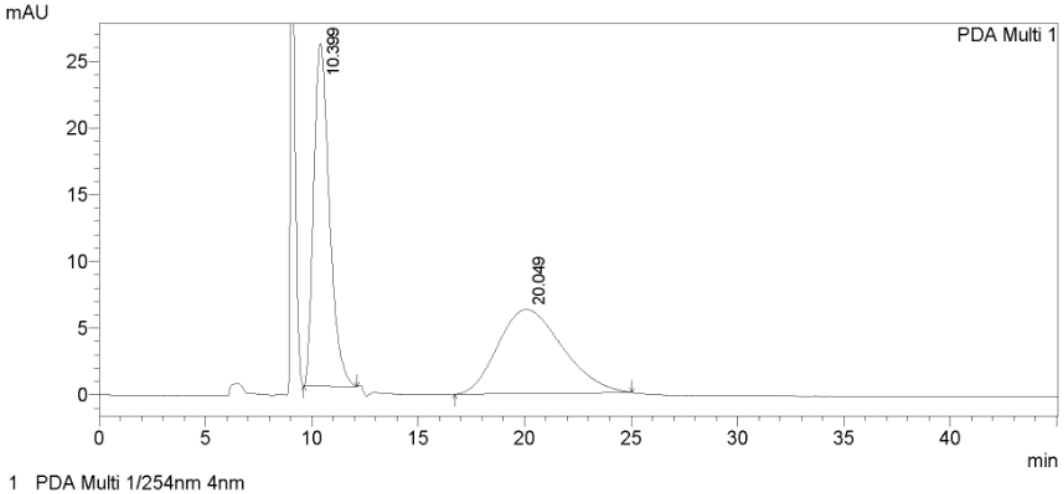
PDA Ch2 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.771	118196578	1626113	96.018	97.423
2	18.555	4902302	43012	3.982	2.577
Total		123098880	1669126	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-bromophenyl)-8-chloro-2-hydroxy-2-phenyl-1,2,5,6,11,11*b*-hexa hydro-3*H*-indolizino[8,7-*b*]indol-3-one (2k)

White solid (87 mg, 86% yield). m.p. 274-277 ºC; $[\alpha]_{25}^{D}$ = 124.0° (*c* = 0.5, $CHCl_3$); ee = 96% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3364, 3267, 1686, 1488, 1446, 1428, 1397 cm^{-1}; ^1H NMR (400MHz, DMSO-d_6, TMS) δ (ppm) δ 11.71 (s, 1H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.49 (d, *J* = 1.8 Hz, 1H), 7.44-7.39 (m, 3H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.28- 7.24 (m, 3H), 7.15-7.12 (m, 1H), 6.08 (s, 1H), 4.30-4.26 (m, 1H), 3.14 (d, *J* = 14.2 Hz, 1H), 3.02-2.95 (m, 1H), 2.91-2.83 (m, 1H), 2.71-2.66 (m, 2H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 174.8, 143.4, 143.1, 137.5, 134.5, 131.6, 128.2, 128.0, 127.5, 127.3, 125.8, 123.5, 121.4, 120.8, 117.6, 112.9, 106.6, 78.1, 62.5, 51.4, 36.2, 19.7; HRMS (ESI) Calcd. for $C_{26}H_{19}N_2O_2ClBr$, $[M-H]^+$ 507.0298. Found: 507.0290.

Chiral HPLC analysis of racemic **2k**.



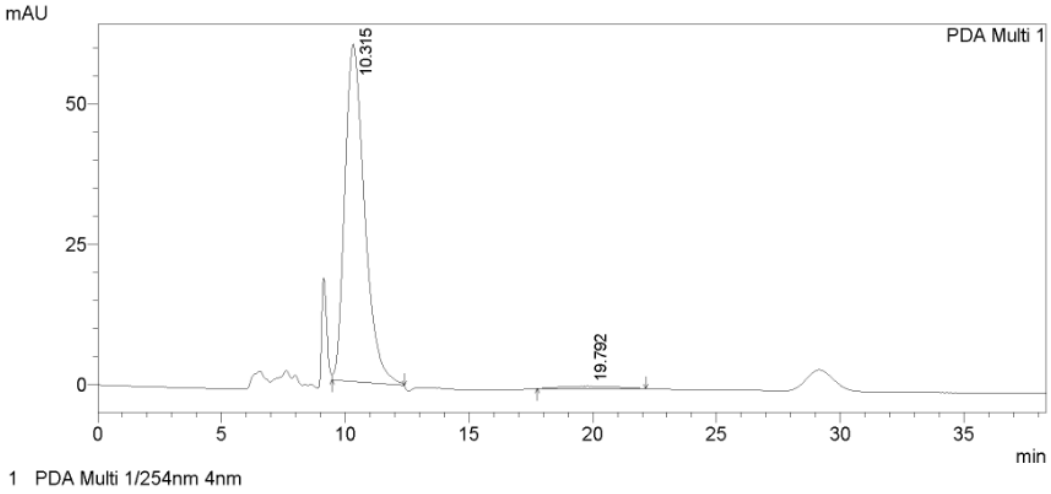
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.399	1319788	25675	49.745	80.274
2	20.049	1333335	6309	50.255	19.726
Total		2653124	31984	100.000	100.000

Chiral HPLC analysis of **2k** from asymmetric reaction.



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

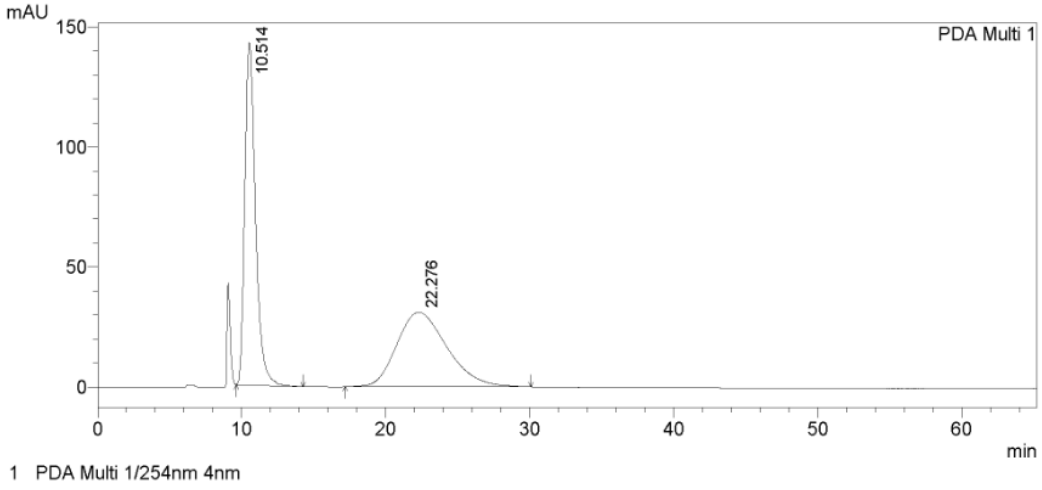
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.315	3339980	59990	98.000	99.274
2	19.792	68157	438	2.000	0.726
Total		3408137	60429	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-bromophenyl)-2-hydroxy-8-methyl-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2l)

White solid (86 mg, 88% yield). m.p. 251-254 ºC; $[\alpha]_{25}^{D}$ = 138.0° (*c* = 0.5, $CHCl_3$); ee = 96% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3406, 3286, 1710, 1679, 1440, 1447, 1412, 1396 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) δ 8.13 (s, 1H), 7.45-7.42 (m, 2H), 7.34-7.29

(m, 6H), 7.23 (d, J = 8.7 Hz, 1H), 7.04 (d, J = 8.2 Hz, 1H), 6.94 (d, J = 8.7 Hz, 2H), 4.42-4.38 (m, 1H), 3.13-2.97 (m, 3H), 2.92 (d, J = 14.2 Hz, 1H), 2.72-2.67 (m, 1H), 2.46 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 174.6, 142.5, 141.8, 134.5, 134.4, 131.7, 129.6, 128.7, 128.5, 128.3, 126.9, 125.7, 124.3, 122.2, 118.5, 111.0, 109.0, 79.4, 63.1, 50.3, 36.5, 21.5, 20.2; HRMS (ESI) Calcd. for $C_{27}H_{22}N_2O_2Br$, $[M-H]^+$ 487.0844. Found: 487.0843.

Chiral HPLC analysis of racemic **2l**.

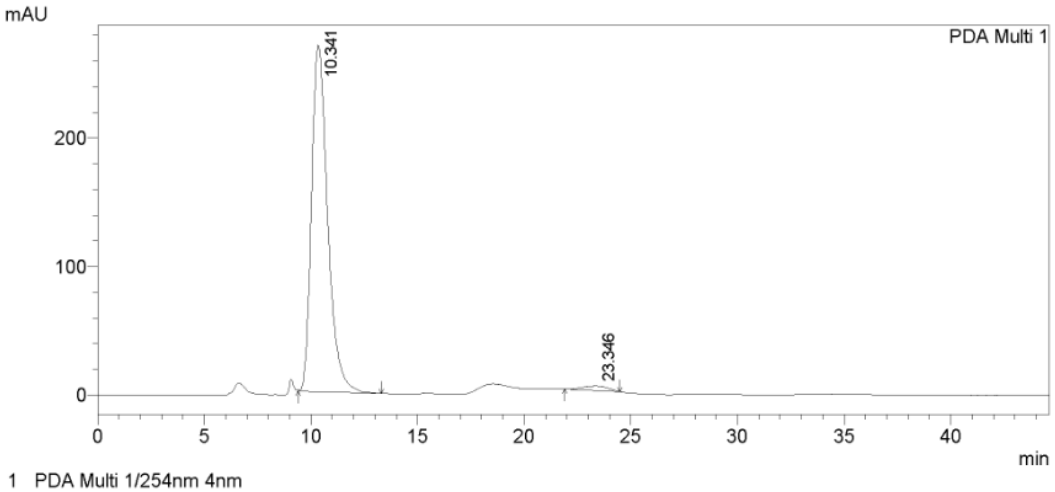


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.514	7371805	142743	49.465	82.117
2	22.276	7531361	31086	50.535	17.883
Total		14903166	173829	100.000	100.000

Chiral HPLC analysis of **2l** from asymmetric reaction.



PeakTable

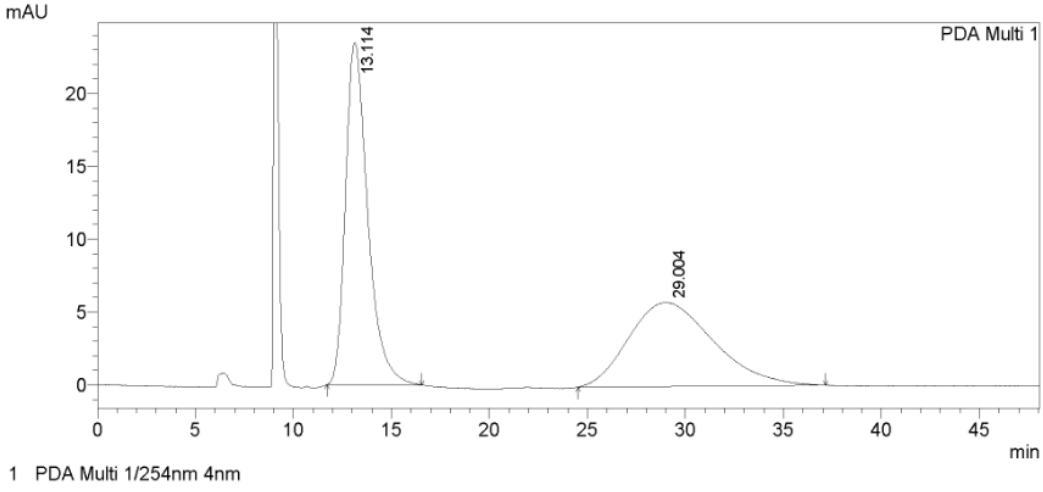
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.341	14234773	269993	97.981	98.708
2	23.346	293285	3535	2.019	1.292
Total		14528058	273527	100.000	100.000

(2*S*,11*bS*)-11*b*-(4-bromophenyl)-2-hydroxy-8-methoxy-2-phenyl-1,2,5,6,11,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (2m)

White solid (87 mg, 86% yield). m.p. 165-167 ^{o}C; $[\alpha]_{25}^{D}$ = 142.0° (*c* = 0.5, $CHCl_3$); ee = 94% (DAICEL Chiralpak AS-H, hexane/2-propanol = 70/30, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3406, 3310, 1693, 1677, 1487, 1436, 1401, 1217 cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 11.29 (s, 1H), 7.52 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 6.9 Hz, 2H), 7.35-7.24 (m, 6H), 6.93 (d, *J* = 2.3 Hz, 1H), 6.78- 6.76 (m, 1H), 6.05 (s, 1H), 4.31-4.28 (m, 1H), 3.76 (s, 3H), 3.13 (d, *J* = 13.8 Hz, 1H), 3.03-2.95 (m, 1H), 2.91-2.83 (m, 1H), 2.70-2.63 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$, TMS) δ (ppm) 174.7, 153.4, 143.5, 143.4, 136.3, 131.5, 131.1, 128.2, 128.0, 127.3, 126.6, 125.8, 120.7, 112.0, 111.4, 106.3, 100.2, 78.2, 62.6, 55.4, 51.5, 36.3, 19.9; HRMS (ESI) Calcd. for $C_{27}H_{22}N_2O_3Br$, $[M-H]^+$ 501.0819. Found: 501.0812.

Chiral HPLC analysis of racemic **2m**.

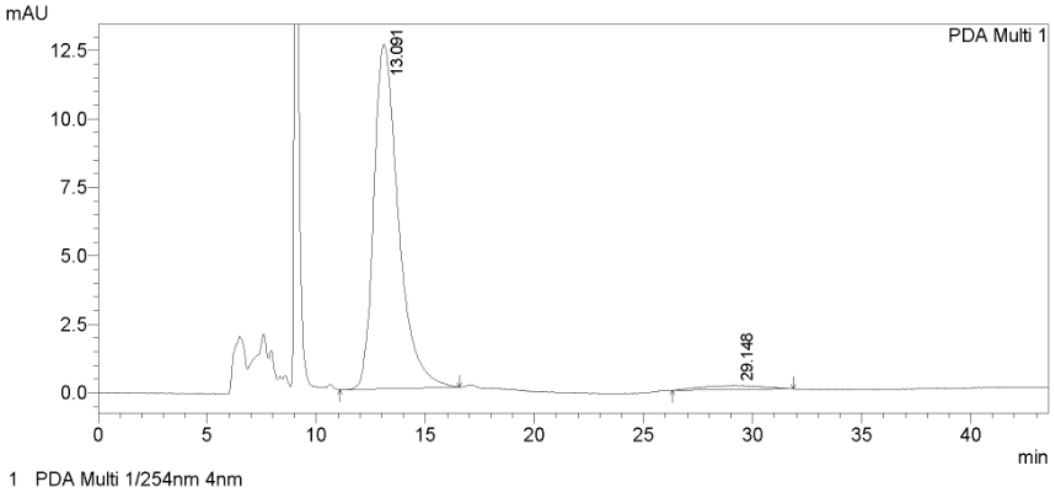


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.114	1860241	23485	51.802	80.290
2	29.004	1730849	5765	48.198	19.710
Total		3591090	29250	100.000	100.000

Chiral HPLC analysis of **2m** from asymmetric reaction.



1 PDA Multi 1/254nm 4nm

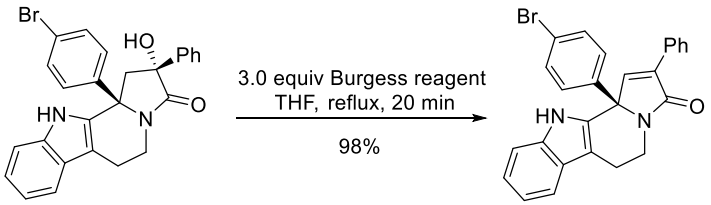
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.091	985823	12580	97.072	98.754
2	29.148	29737	159	2.928	1.246
Total		1015560	12739	100.000	100.000

6. Synthesis of 3, 4 and 5

6.1 Synthesis of 3



2b' **3**

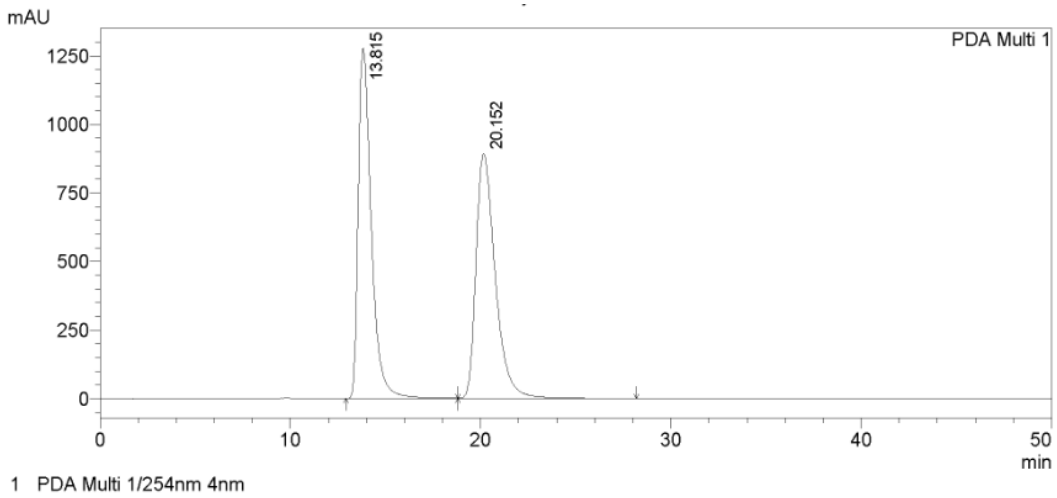
A mixture of **2b'** (94.7 mg, 0.2 mmol) and Burgess reagent (153.8 mg, 0.6 mmol) were refluxed in THF (5 mL) until raw materials disappeared about 20 min. Water was added to quench the reaction and the mixture was extracted with ethyl acetate (3×5 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated in vacuo. Flash chromatography on silica gel (PE:EA = 2:1) afforded **3** as white solid.

(*R*)-11*b*-(4-bromophenyl)-2-phenyl-5,6,11,11*b*-tetrahydro-3*H*-indolizino[8,7-*b*]indol-3-one (3)

White solid (89 mg, 98% yield). m.p. 294-295 ^oC; $[\alpha]_{25}^{D}$ = -122.0° (*c* = 0.5, $CHCl_3$); ee = -97% (DAICEL Chiralpak OD-H, hexane/2-propanol = 80/20, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) *ν* 3230, 3060, 1664, 1485, 1403 cm^{-1}; ^1H NMR

(400MHz, DMSO-d_6, TMS) δ (ppm) 11.51 (s, 1H), 7.97-7.93 (m, 3H), 7.60-7.57 (m, 2H), 7.50-7.37 (m, 5H), 7.17-7.10 (m, 3H), 7.06-7.02 (m, 1H), 4.40-4.36 (m, 1H), 3.03-2.95 (m, 1H), 2.86-2.77 (m, 1H); ^{13}C NMR (100MHz, DMSO-d_6, TMS) δ (ppm) 168.9, 143.7, 138.2, 136.3, 134.0, 131.8, 131.5, 130.8, 129.1, 129.0, 128.6, 127.1, 126.0, 122.1, 121.7, 119.1, 118.7, 111.5, 107.9, 65.8, 35.0, 21.0; HRMS (ESI) Calcd. for $C_{26}H_{19}N_2OBrNa$, $[M+Na]^+$ 477.0573. Found: 477.0572.

Chiral HPLC analysis of racemic **3**.

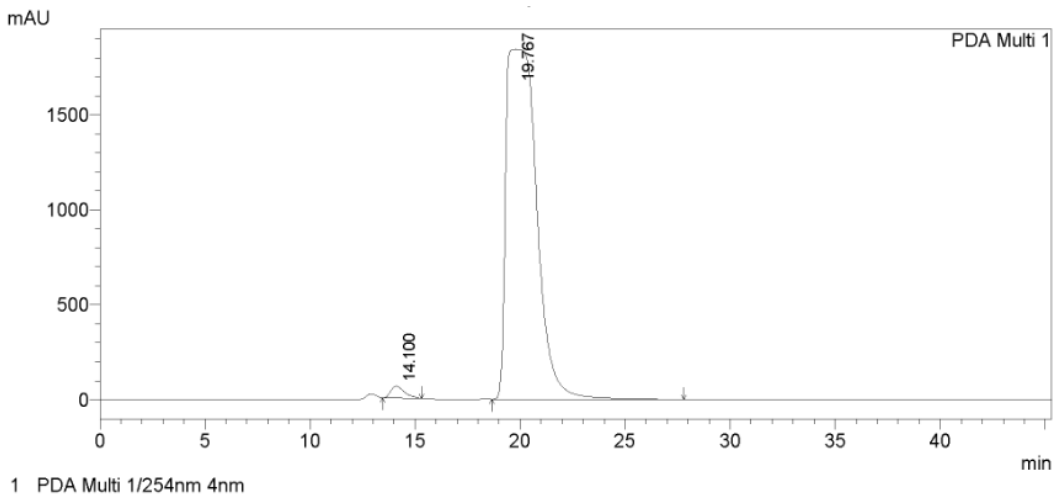


峰表

PDA Ch1 254nm 4nm

峰#	保留时间	面积	高度	面积 %	高度 %
1	13.815	61171762	1276598	49.345	58.866
2	20.152	62795865	892063	50.655	41.134
Total		123967627	2168661	100.000	100.000

Chiral HPLC analysis of **3** from asymmetric reaction.

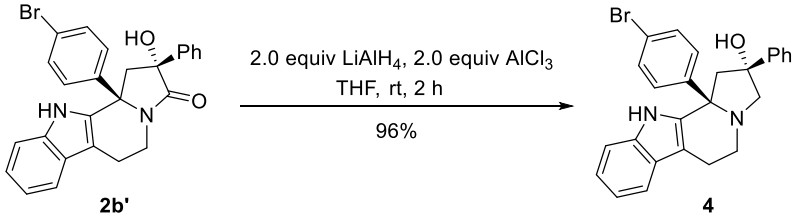


峰表

PDA Ch1 254nm 4nm

峰#	保留时间	面积	高度	面积 %	高度 %
1	14.100	2925145	63488	1.539	3.326
2	19.767	187113253	1845230	98.461	96.674
Total		190038397	1908718	100.000	100.000

6.2 Synthesis of 4



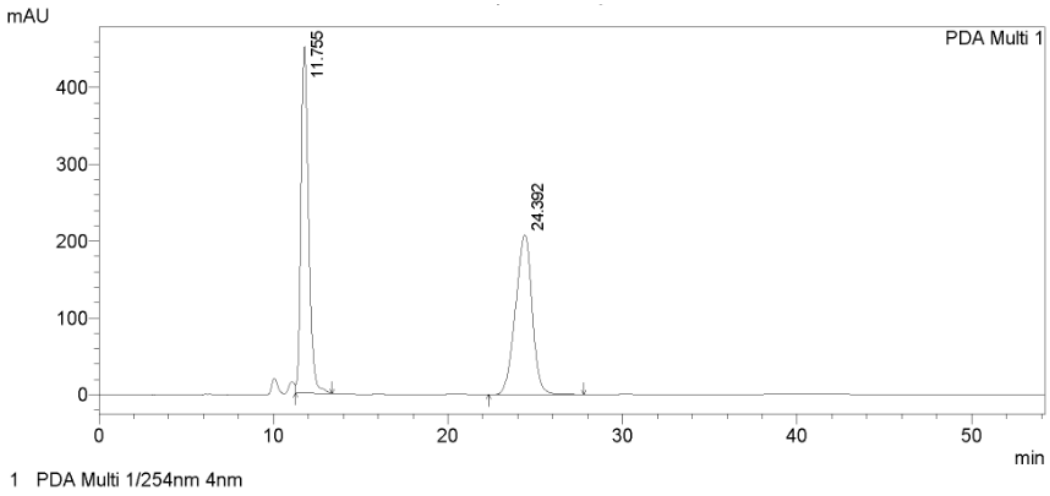
To a flask (10 mL) equipped with a magnetic stirrer was added $LiAlH_4$ (15.3 mg, 0.4 mmol), $AlCl_3$ (52.9 mg, 0.4 mmol), **2b'** (94.7 mg, 0.2 mmol) and dry THF (5 mL) under argon protection. The reaction mixture was stirred at room temperature for 2 h and then saturated NH_4Cl aqueous solution (0.2 mL) was added to quench the reaction. The mixture was extracted with ethyl acetate (3×10 mL), and combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvents were removed in *vacuo* and the residue was purified by flash column chromatography (PE:EA = 4:1) to afford products **4**.

(2*R*,11*bR*)-11*b*-(4-bromophenyl)-2-phenyl-2,3,5,6,11,11*b*-hexahydro-1*H*-indolizino[8,7-*b*]indol-2-ol (4)

White solid (88 mg, 96% yield). m.p. 128-130 °C; $[\alpha]_{25}^{D}$ = 32.7° (*c* = 0.5, $CHCl_3$); ee = -96% (DAICEL Chiralpak OD-H, hexane/2-propanol = 80/20, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3422, 3056, 2921, 2846, 1485, 1460, 1447, 1393 cm^{-1};

^{1}H NMR (400MHz, ACETONE-D_6, TMS) δ (ppm) 10.34 (s, 1H), 7.76-7.74 (m, 2H), 7.50 (d, J = 8.0 Hz, 1H), 7.43-7.40 (m, 3H), 7.38-7.33 (m, 4H), 7.25-7.22 (m, 1H), 7.15-7.11 (m, 1H), 7.07-7.03 (m, 1H), 3.61 (d, J = 9.6 Hz, 1H), 3.47 (d, J = 9.6 Hz, 1H), 3.19 (dd, J = 13.6, 5.6 Hz, 1H), 3.11-3.06 (m, 2H), 2.96-2.86 (m, 2H), 2.63 (d, J = 14.0 Hz, 1H), 2.51 (dd, J = 15.6, 4.4 Hz, 1H); ^{13}C NMR (100MHz, ACETONE-D_6, TMS) δ (ppm) 150.9, 147.1, 137.4, 136.2, 131.7, 130.2, 128.8, 128.4, 127.1, 125.7, 122.1, 121.0, 119.6, 119.0, 111.9, 108.3, 79.6, 68.1, 65.9, 59.4, 41.7, 16.7; HRMS (ESI) Calcd. for $C_{26}H_{24}N_2OBr$, $[M+H]^+$ 459.1067. Found: 459.1063.

Chiral HPLC analysis of racemic **4**.

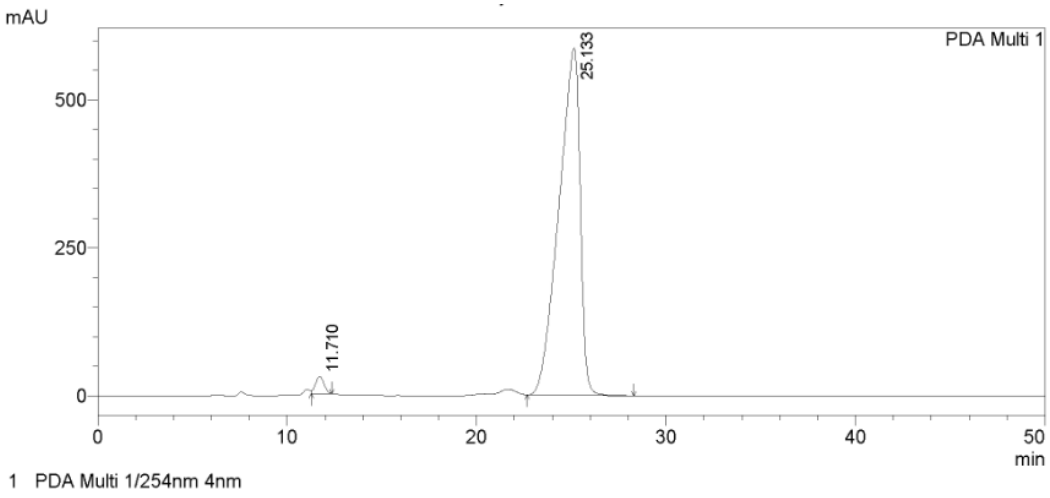


峰表

PDA Ch1 254nm 4nm

峰#	保留时间	面积	高度	面积 %	高度 %
1	11.755	13631891	450569	49.721	68.439
2	24.392	13784753	207786	50.279	31.561
Total		27416644	658355	100.000	100.000

Chiral HPLC analysis of **4** from asymmetric reaction.

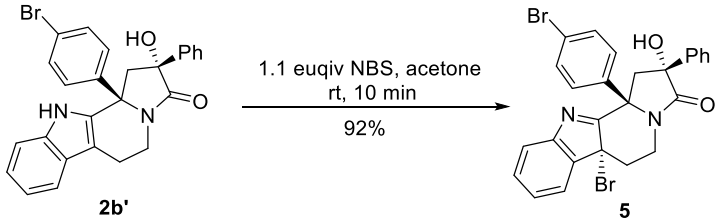


峰表

PDA Ch1 254nm 4nm

峰#	保留时间	面积	高度	面积 %	高度 %
1	11.710	943045	29672	2.016	4.811
2	25.133	45832504	587044	97.984	95.189
Total		46775549	616716	100.000	100.000

6.3 Synthesis of 5



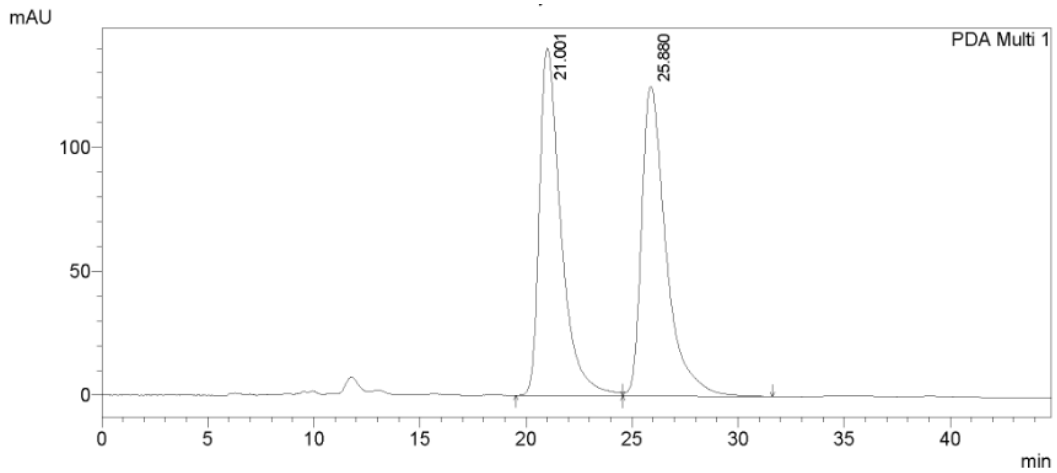
To a mixture of **2b'** (94.7 mg, 0.2 mmol) and dry acetone (5 mL) in flask was added NBS (39.2 mg, 0.22 mmol) in part. The mixture was kept stirring at room temperature until raw materials disappeared about 10 min. The residue was concentrated and purified by flash column chromatography on silica gel (PE:EA = 4:1) to give product **5**.

(2*R*,6*aR*,11*bR*)-6*a*-bromo-11*b*-(4-bromophenyl)-2-hydroxy-2-phenyl-1,2,5,6,6a,11*b*-hexahydro-3*H*-indolizino[8,7-*b*]indol-3-one (5)

White solid (101 mg, 92% yield). m.p. 187-189 ºC; $[\alpha]_{25}^{D}$ = 76.0° (*c* = 0.5, $CHCl_3$); ee = -97.5% (DAICEL Chiralpak OD-H, hexane/2-propanol = 80/20, λ = 254 nm, flow rate = 0.5 mL/min); IR (KBr) ν 3431, 1688, 1424, 1256 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$, TMS) δ (ppm) 7.75 (d, *J* = 7.6 Hz, 1H), 7.48-7.40 (m, 4H), 7.37-7.32 (m, 3H), 7.28-7.23 (m, 3H), 7.21-7.18 (m, 2H), 4.58-4.53 (m, 1H), 3.93 (d, *J* = 14.0 Hz, 1H), 3.66-3.59 (m, 1H), 2.75-2.66 (m, 2H), 1.91-1.83 (m, 1H); ^{13}C NMR (100MHz,

DMSO-d_6, TMS) δ (ppm) 181.3, 173.9, 152.1, 143.2, 141.4, 140.2, 131.4, 129.3, 127.8, 127.3, 127.0, 126.6, 125.8, 122.7, 121.1, 120.2, 80.7, 77.1, 65.8, 48.9, 38.2, 34.1; HRMS (ESI) Calcd. for $C_{26}H_{20}N_2O_2Br_2Na$, $[M+Na]^+$ 572.9784. Found: 572.9783.

Chiral HPLC analysis of racemic **5**.



1 PDA Multi 1/254nm 4nm

峰表

PDA Ch1 254nm 4nm

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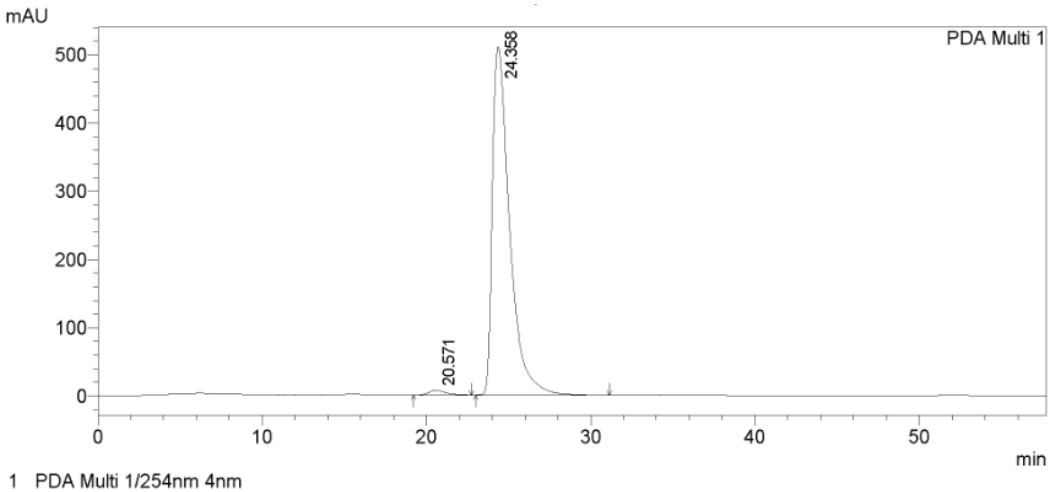
1	21.001	9919477	140151	49.777	52.847
2	25.880	10008340	125051	50.223	47.153
Total		19927817	265202	100.000	100.000

Chiral HPLC analysis of **5** from asymmetric reaction.



1 PDA Multi 1/254nm 4nm

峰表

PDA Ch1 254nm 4nm

峰#	保留时间	面积	高度	面积 %	高度 %
1	20.571	486696	7019	1.295	1.357
2	24.358	37103414	510337	98.705	98.643
Total		37590110	517356	100.000	100.000

7. The X-ray molecular structure of compounds (2*S*, 11b*S*)-2b

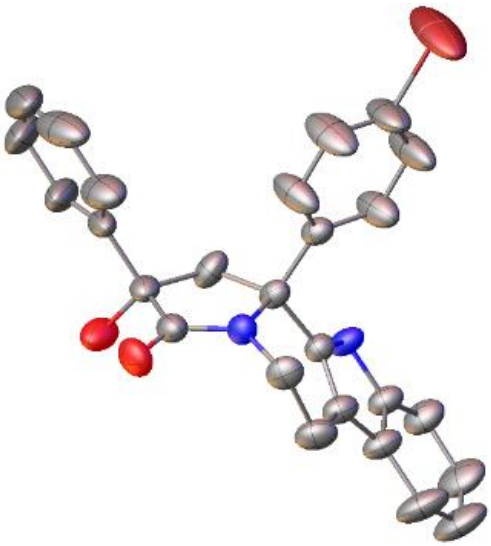


Figure S2. The structure of compound **2b**

7.1 Crystallographic data and structure refinement of (2*S*, 11b*S*)-2b

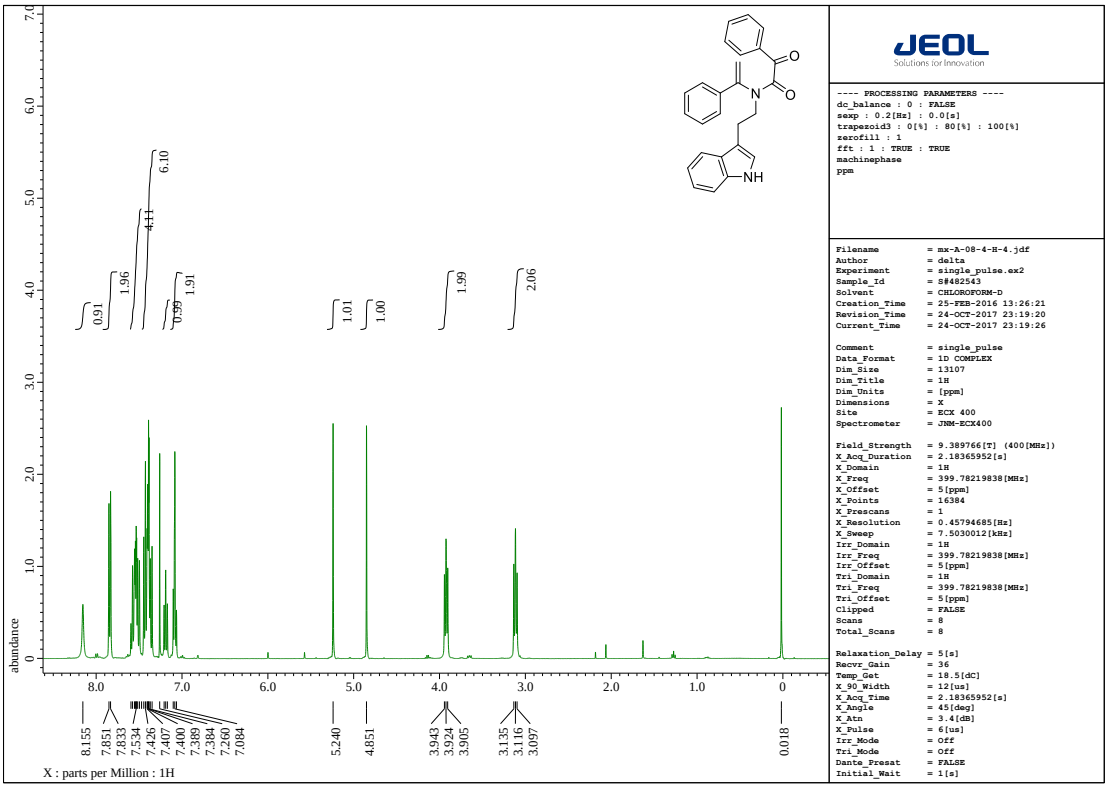
Indentification code	xxm-02
Empirical formula	$C_{30}H_{29}BrN_2O_4$
Formula weight	561.46
Temperature	173.00(10) K
Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	P 1 21 1
Unit cell dimen	a = 9.9350 (2) Å α = 90.00 º b = 12.0944 (2) Å β = 110.004 (2)° c = 12.1297 (2) Å γ = 90.00 º
Volumn	1369.55 (5) $Å^3$
Z	2
Crystal size	0.15 x 0.11 x 0.08 mm^3
CCDC Number	2026743

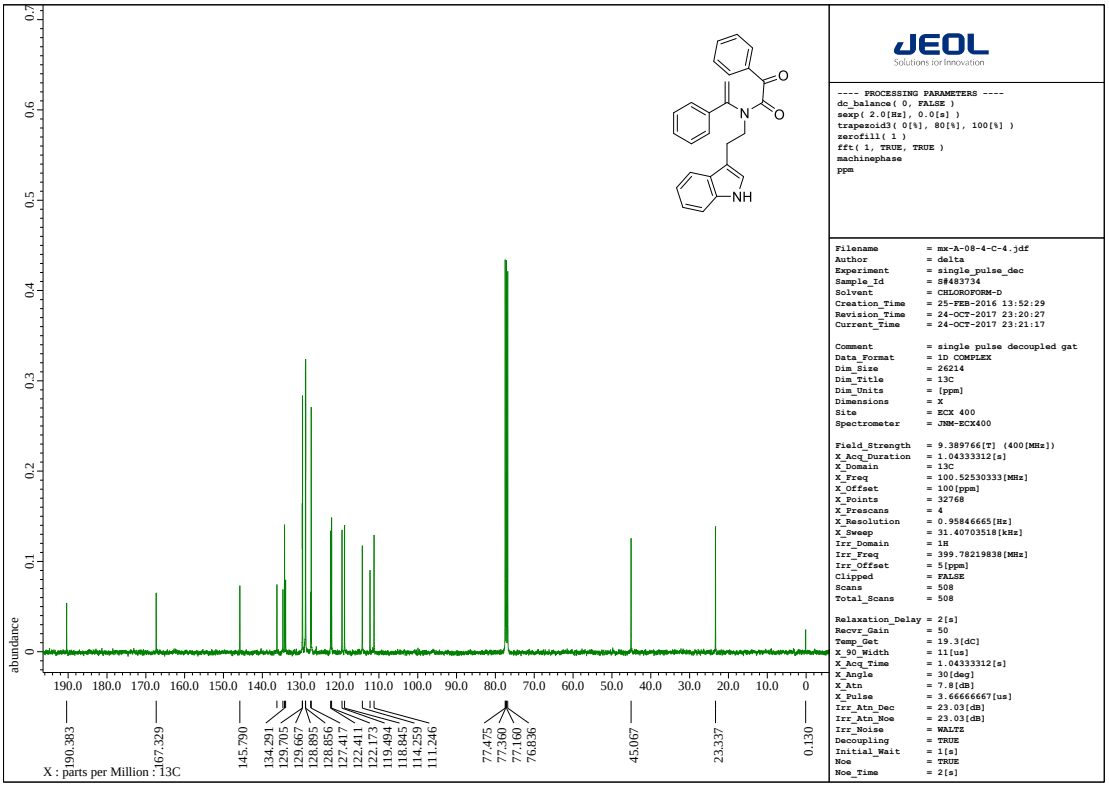
8. References

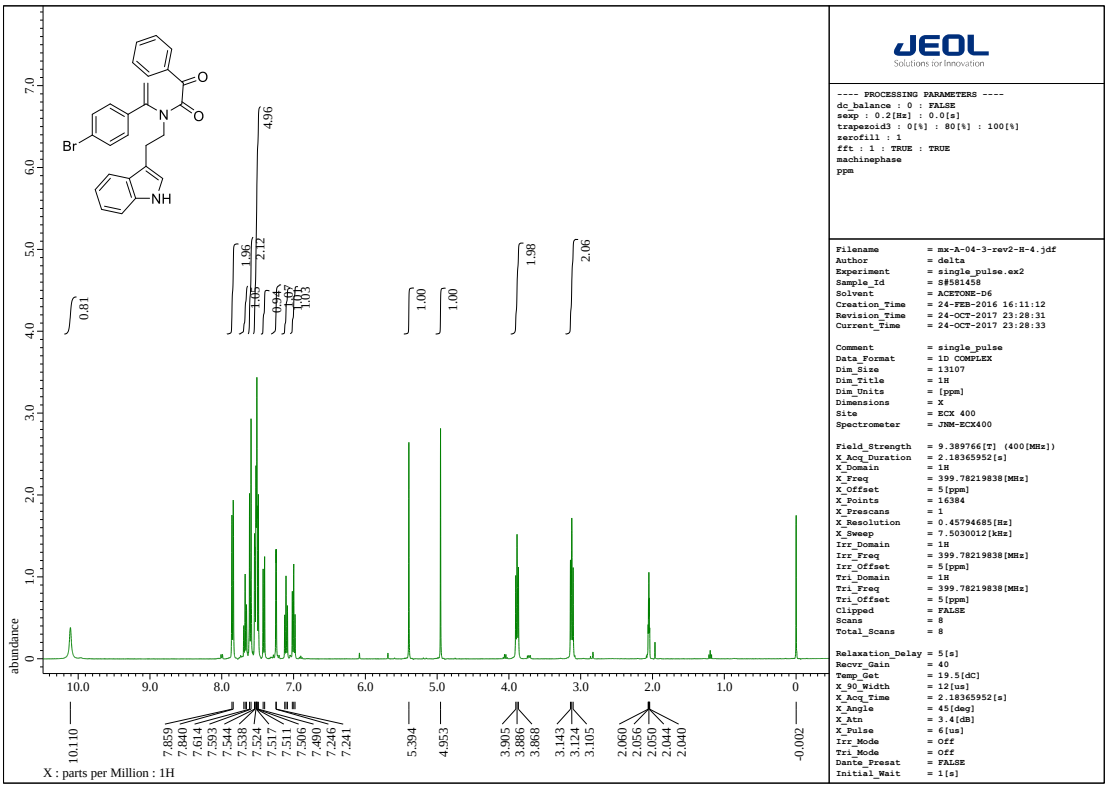
[1] (a) L. Yang, D.-X. Wang, Z.-T. Huang and M.-X. Wang, *J. Am. Chem. Soc.* **2009**, ***131***, 10390. (b) S. Tong, D.-X. Wang, L. Zhao, J. Zhu, M.-X. Wang, *Angew. Chem. Int. Ed.* **2012**, *51*, 4417; *Angew. Chem.* **2012**, *124*, 4493.

[2] (a) L. He, L. Zhao, D.-X. Wang, M.-X. Wang, *Org. Lett.* **2014**, *16*, 5972.(ba) S. Tong, X. Yang, D.-X. Wang, L. Zhao, J. Zhu, M.-X. Wang, *Tetrahedron* **2012**, *68*, 6492. (c) L. Yang, G. Deng, D.-X. Wang, Z.-T. Huang, J. Zhu, M.-X. Wang, *Org. Lett.* **2007**, *9*, 1387.

9. Copies of ^{1}H NMR and ^{13}C NMR Spectra of Products







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fft : 1 : TRUE : TRUE
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ppm
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Revision_Time     = 24-OCT-2017 23:28:31
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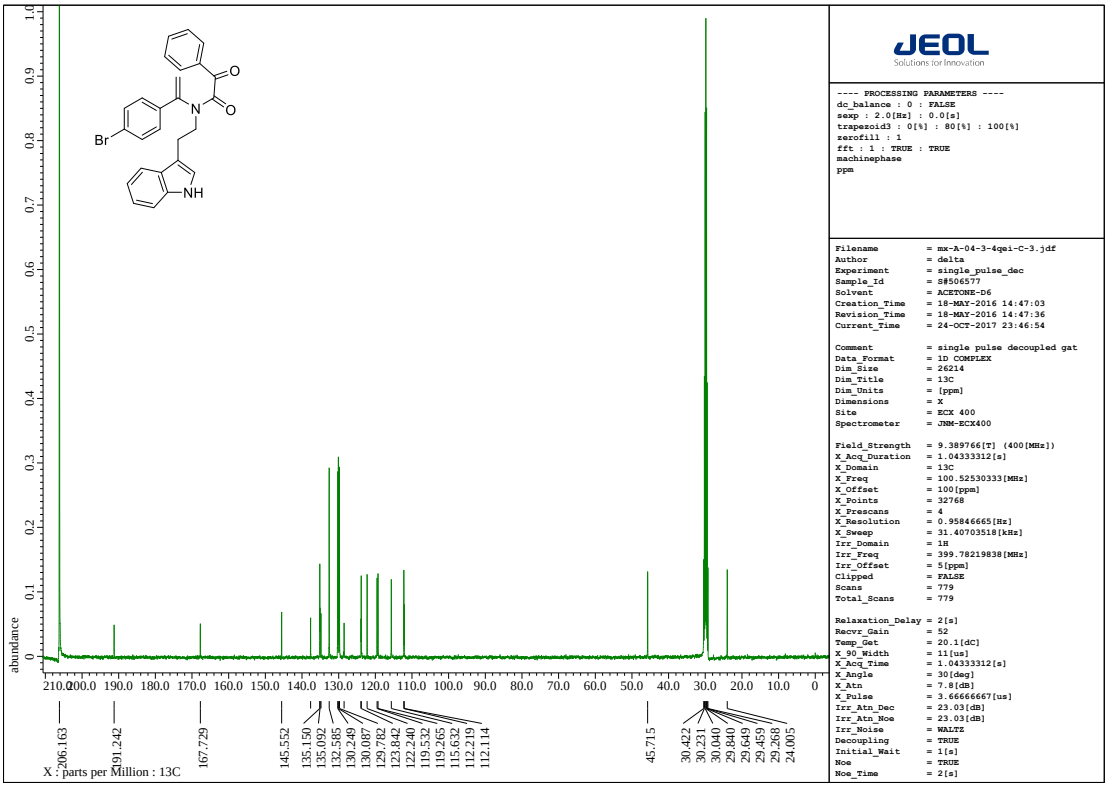

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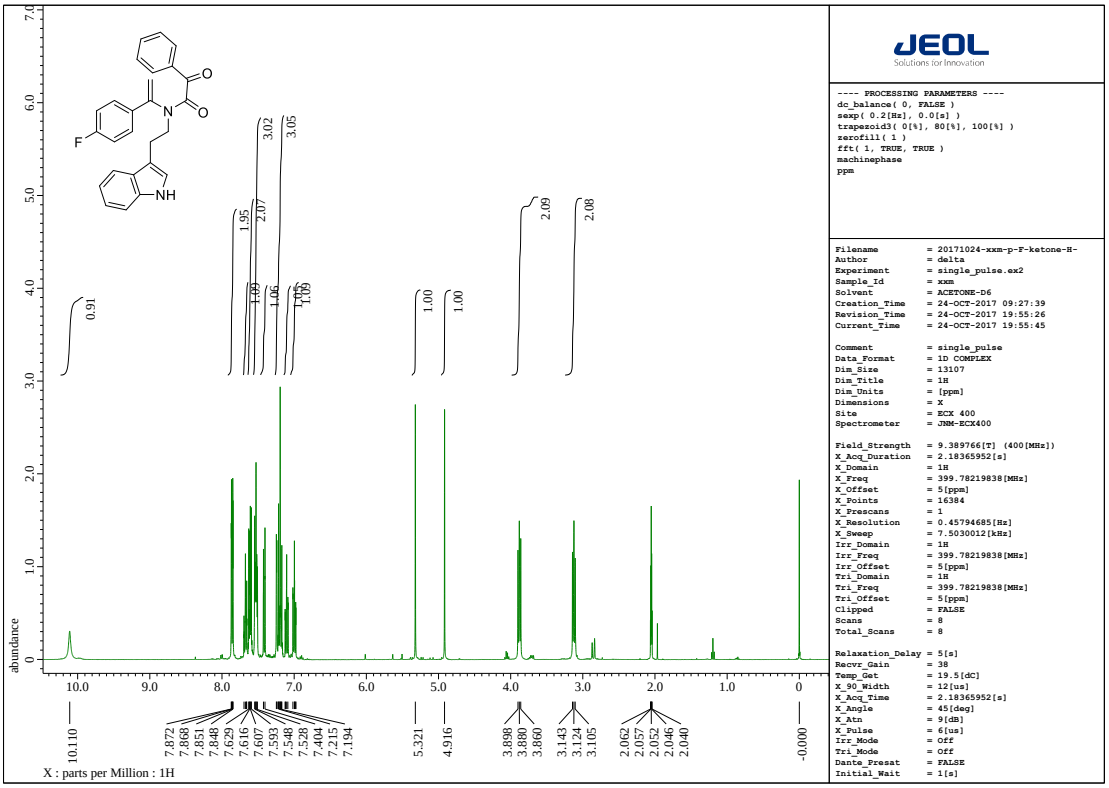
JEOL
Solutions for Innovation
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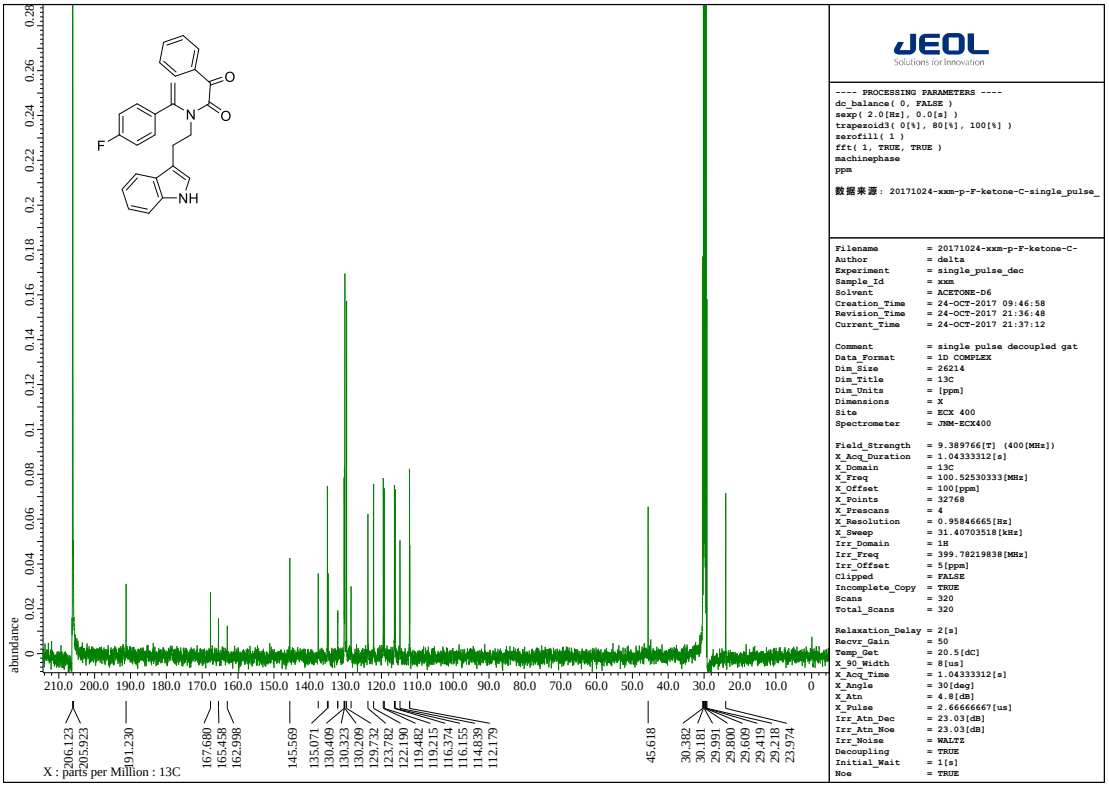
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Solutions for Innovation
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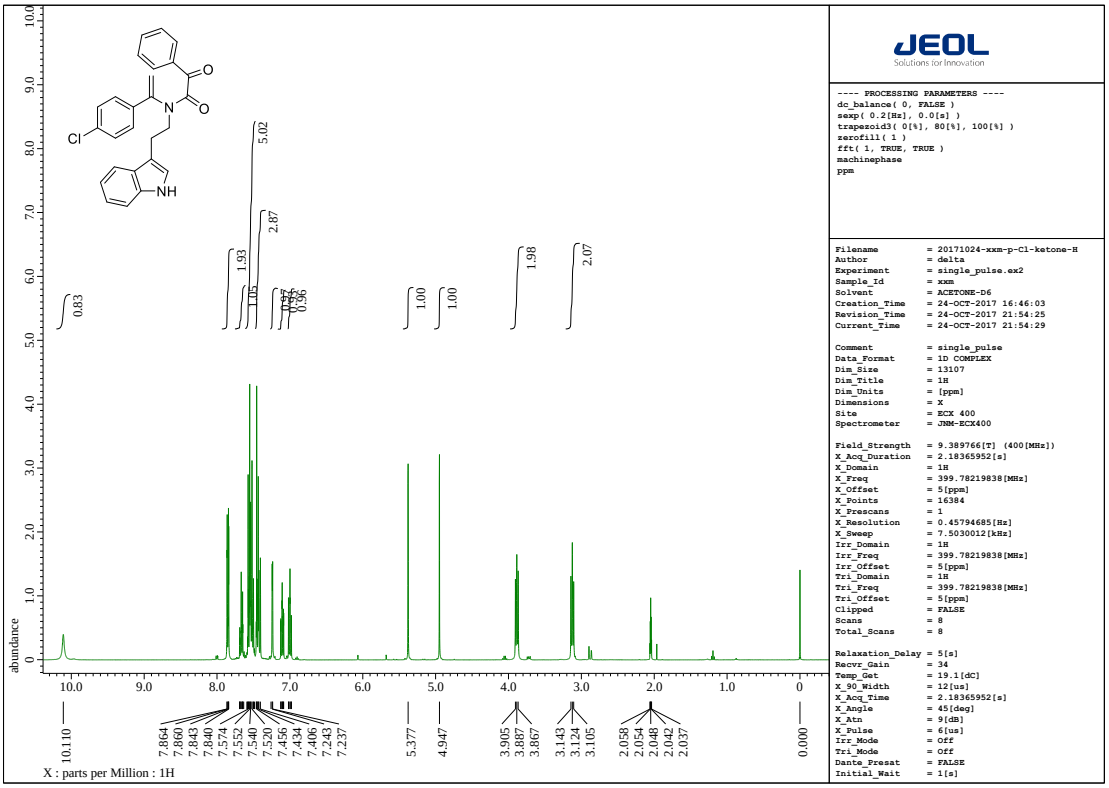
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Solutions for Innovation
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X_Offset = 5[ppm]
X_Points = 16384
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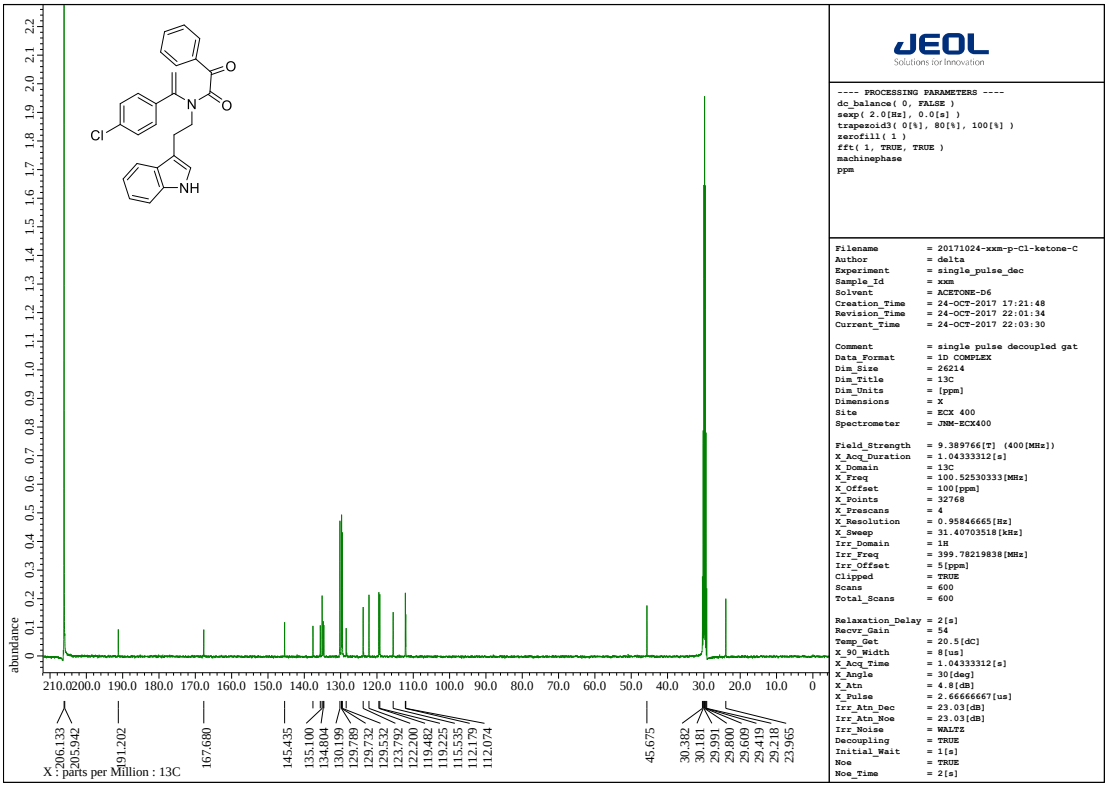
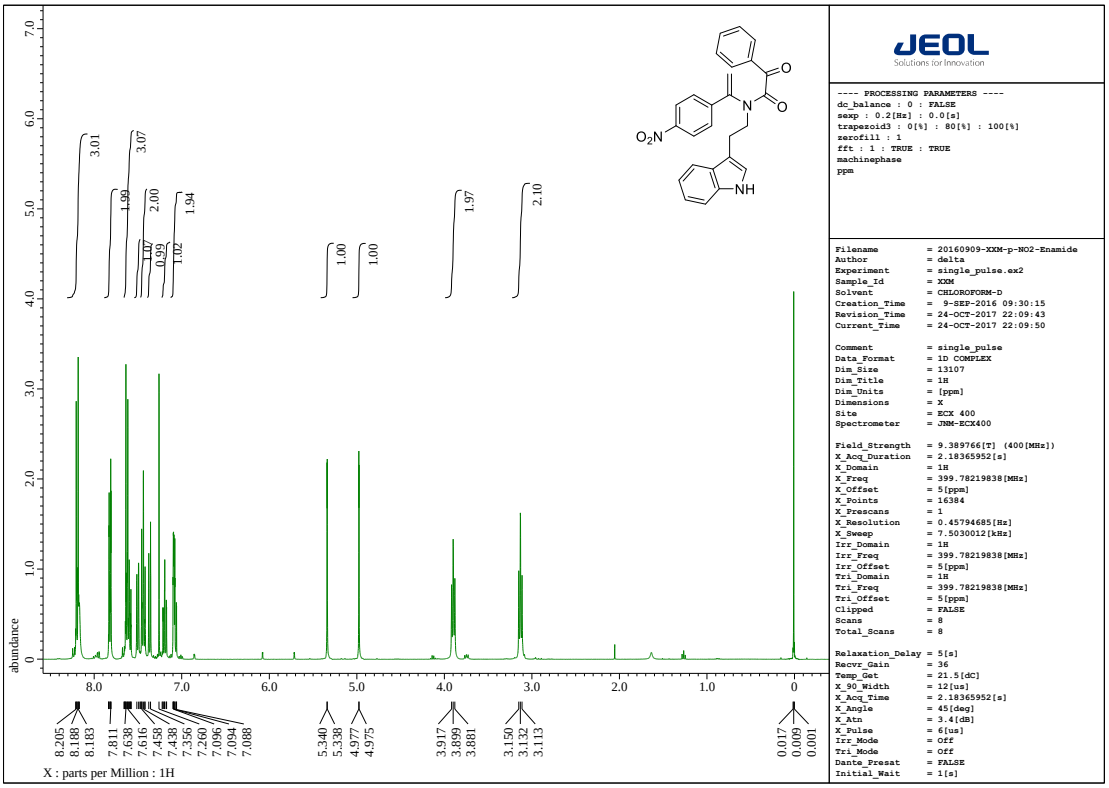
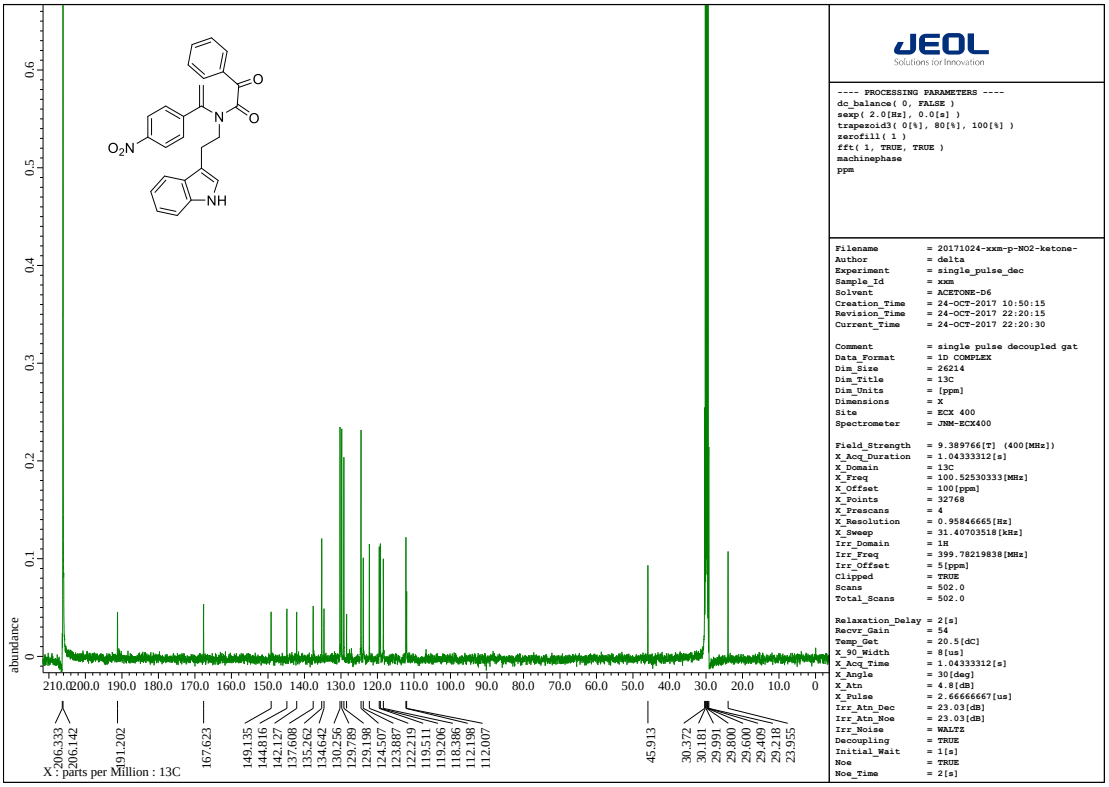
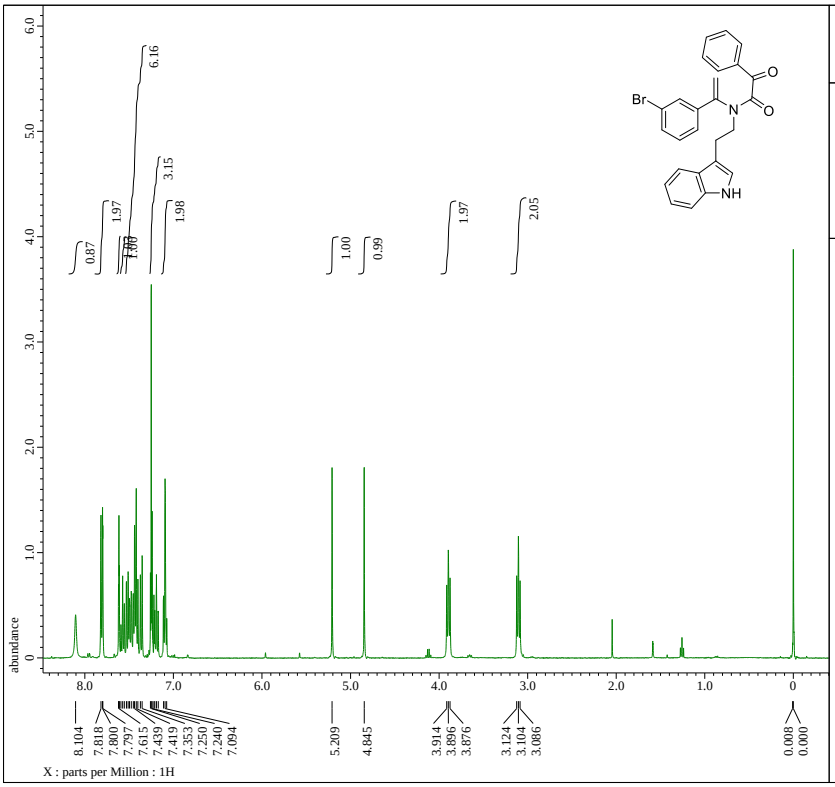
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 19.1[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 9[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
abundance
X : parts per Million : 1H


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20171024-xxm-p-Cl-ketone-C
Author = delta
Experiment = single_pulse_dec
Sample_Id = xxm
Solvent = ACETONE-D6
Creation_Time = 24-OCT-2017 17:21:48
Revision_Time = 24-OCT-2017 22:01:34
Current_Time = 24-OCT-2017 22:03:30
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 600
Total_Scans = 600
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 20.5[dC]
X_90_Width = 8[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4.8[dB]
X_Pulse = 2.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
abundance
X : parts per Million : 13C


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = 20160909-XXM-p-NO2-Enamide
Author = delta
Experiment = single_pulse.ex2
Sample_Id = XXM
Solvent = CHLOROFORM-D
Creation_Time = 9-SEP-2016 09:30:15
Revision_Time = 24-OCT-2017 22:09:43
Current_Time = 24-OCT-2017 22:09:50
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 21.5[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 3.4[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
abundance
X : parts per Million : 1H
3.01
1.99
3.07
2.00
1.07
0.99
1.02
1.94
1.00
1.00
1.97
2.10
8.205
8.188
8.183
7.811
7.638
7.616
7.458
7.438
7.356
7.260
7.096
7.094
7.088
5.340
5.338
4.977
4.975
3.917
3.899
3.881
3.150
3.132
3.113
0.017
0.009
0.001
O2N
NH
O
N


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20171024-xxm-p-NO2-ketone-
Author = delta
Experiment = single_pulse_dec
Sample_Id = xxm
Solvent = ACETONE-D6
Creation_Time = 24-OCT-2017 10:50:15
Revision_Time = 24-OCT-2017 22:20:15
Current_Time = 24-OCT-2017 22:20:30
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 502.0
Total_Scans = 502.0
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 20.5[dC]
X_90_Width = 8[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4.8[dB]
X_Pulse = 2.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
abundance
X : parts per Million : 13C
206.333
206.142
191.202
167.623
149.135
144.816
142.127
137.608
135.262
134.642
130.256
129.789
129.198
124.507
123.887
122.219
119.511
119.206
118.386
112.198
112.007
45.913
30.372
30.181
29.991
29.800
29.600
29.409
29.218
23.955
O2N
NH
O
N



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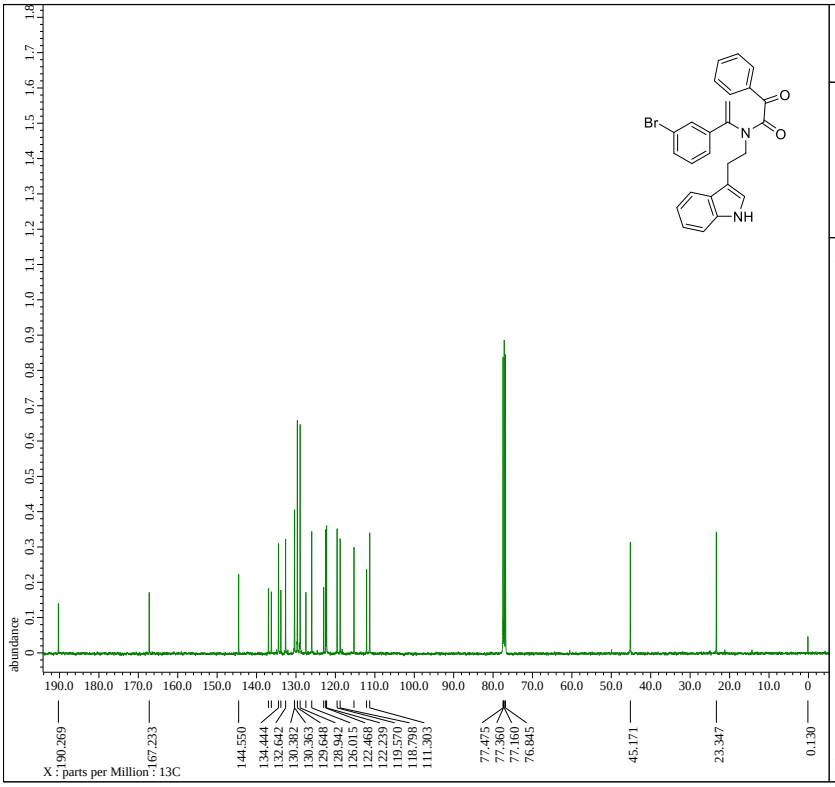
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= mx-A-14-alpha-3-H-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= s#365965
Solvent	= CHLOROFORM-D
Creation_Time	= 1-MAR-2016 10:11:49
Revision_Time	= 24-OCT-2017 23:01:48
Current_Time	= 24-OCT-2017 23:01:52
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 52
Temp_Get	= 20.2[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 3.4[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]



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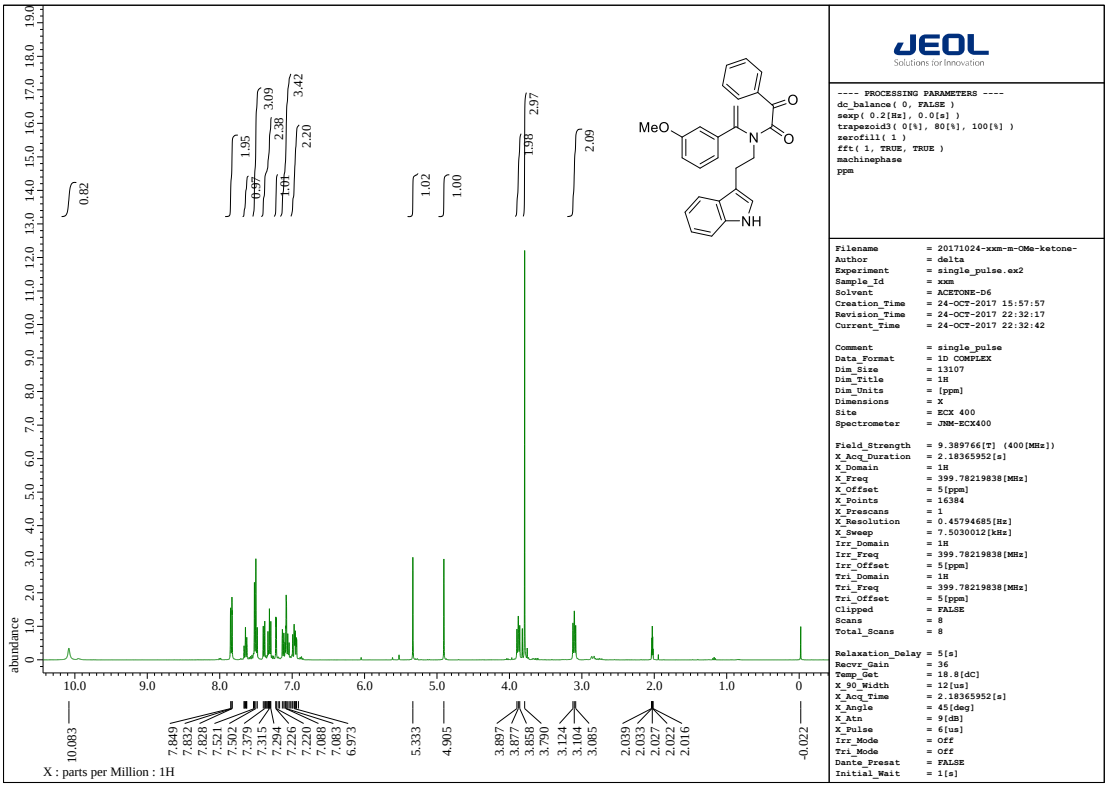
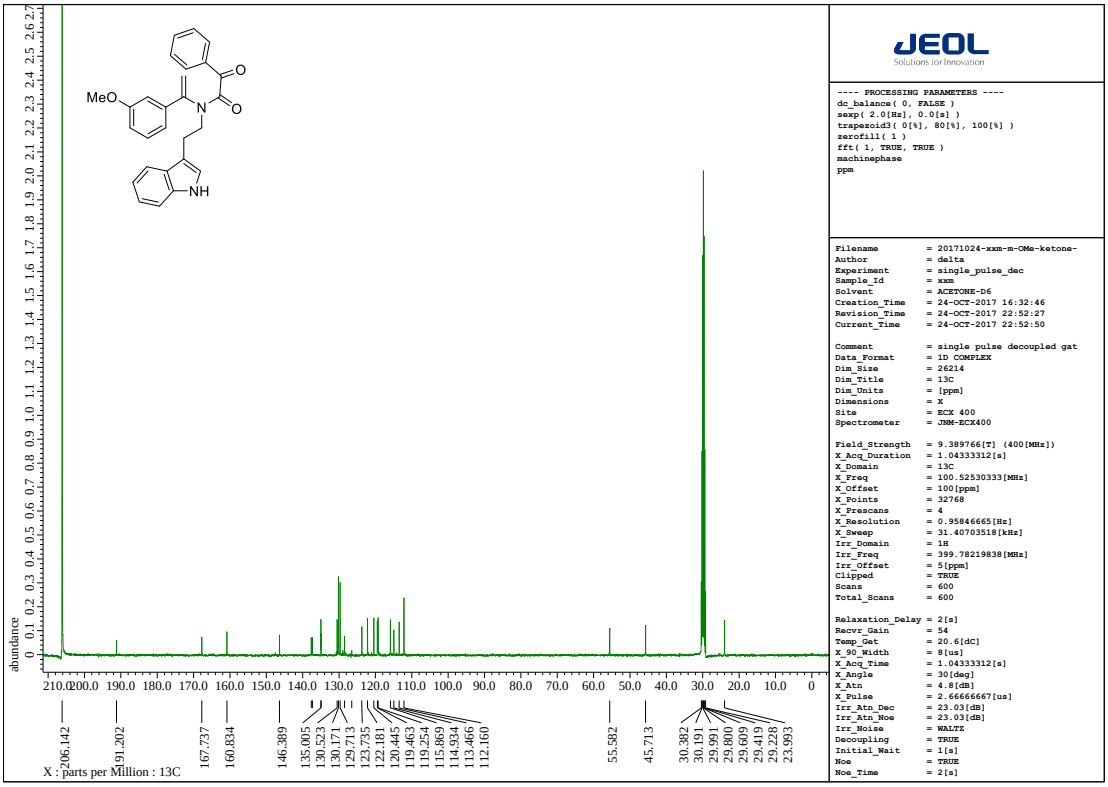
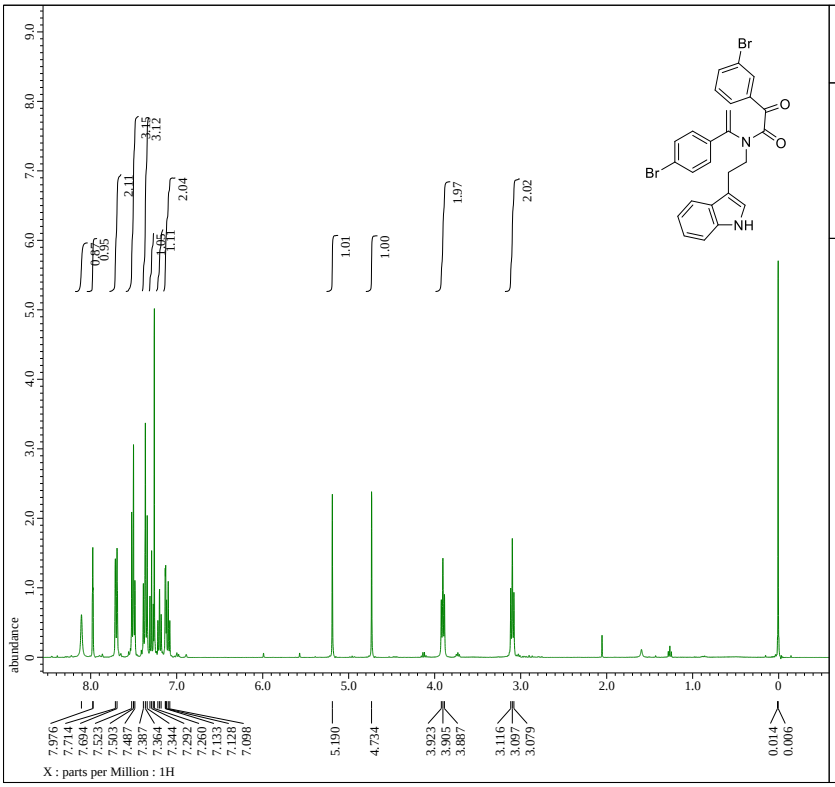
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= mx-A-14-alpha-3-C-rev-5.jd
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= s#596798
Solvent	= CHLOROFORM-D
Creation_Time	= 1-MAR-2016 17:28:07
Revision_Time	= 24-OCT-2017 23:04:32
Current_Time	= 24-OCT-2017 23:05:08
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1000
Total_Scans	= 1000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 56
Temp_Get	= 19.7[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20171024-xxm-m-OMe-ketone-
Author = delta
Experiment = single_pulse.ex2
Sample_Id = xxm
Solvent = ACETONE-D6
Creation_Time = 24-OCT-2017 15:57:57
Revision_Time = 24-OCT-2017 22:32:17
Current_Time = 24-OCT-2017 22:32:42
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 18.8[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 9[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
abundance
X : parts per Million : 1H
MeO
N
O
NH

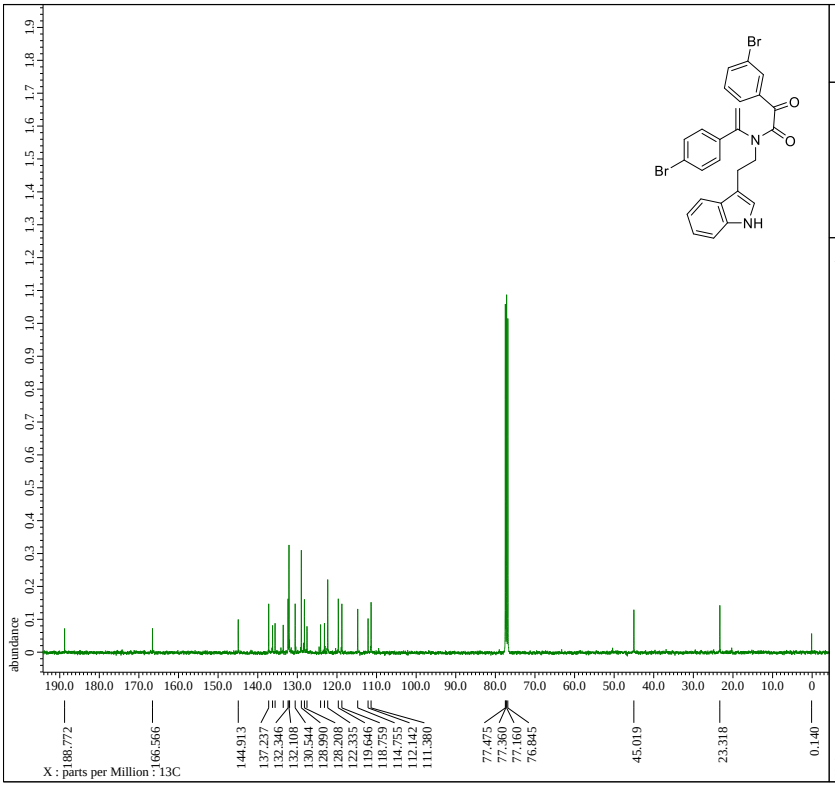

JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20171024-xxm-m-OMe-ketone-
Author = delta
Experiment = single_pulse_dec
Sample_Id = xxm
Solvent = ACETONE-D6
Creation_Time = 24-OCT-2017 16:32:46
Revision_Time = 24-OCT-2017 22:52:27
Current_Time = 24-OCT-2017 22:52:50
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 600
Total_Scans = 600
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 20.6[dC]
X_90_Width = 8[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4.8[dB]
X_Pulse = 2.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
abundance
X : parts per Million : 13C
MeO
N
O
NH



JEOL Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

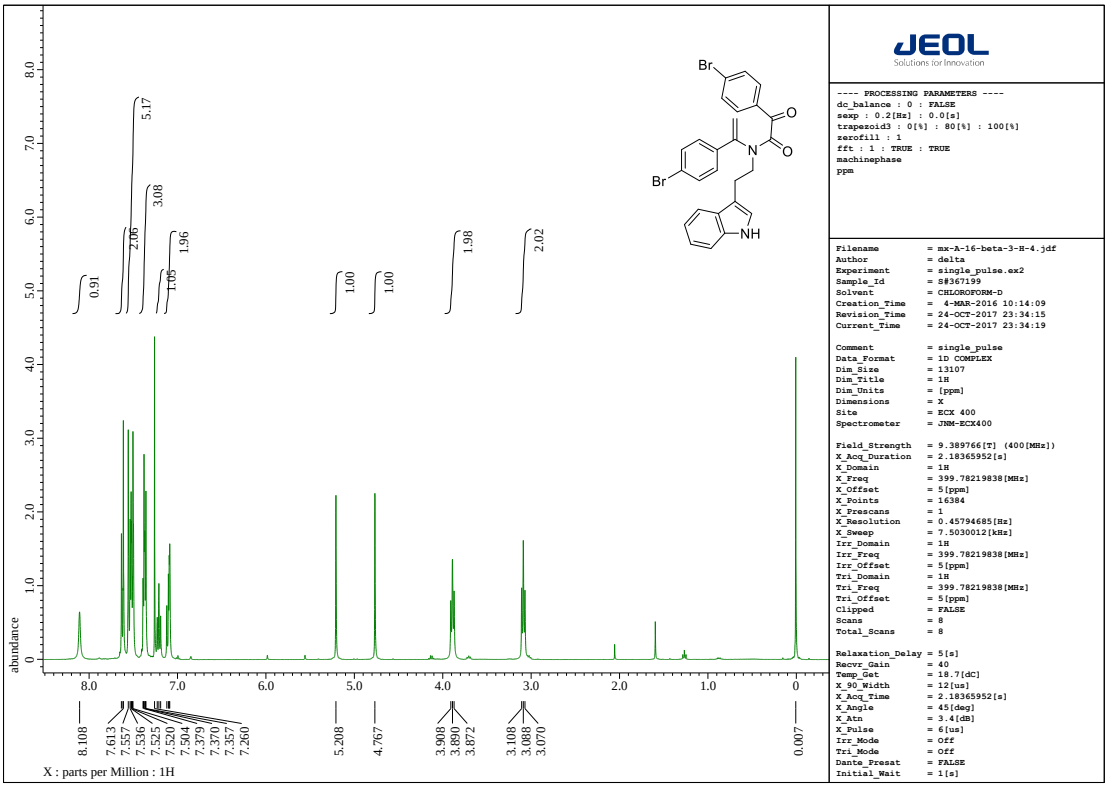
```
Filename          = mx-A-16-alpha-3-H-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#334807
Solvent           = CHLOROFORM-D
Creation_Time     =  4-MAR-2016 09:20:09
Revision_Time     = 24-OCT-2017 23:15:13
Current_Time      = 24-OCT-2017 23:15:17

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 18.8[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```

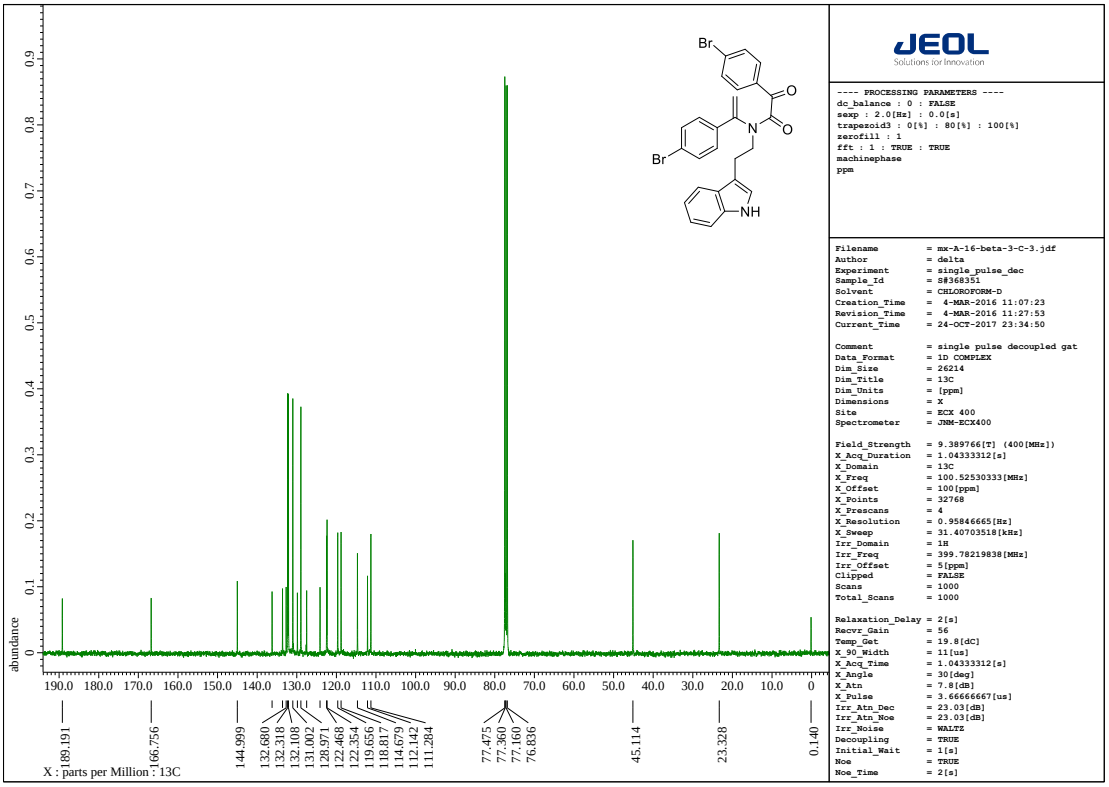


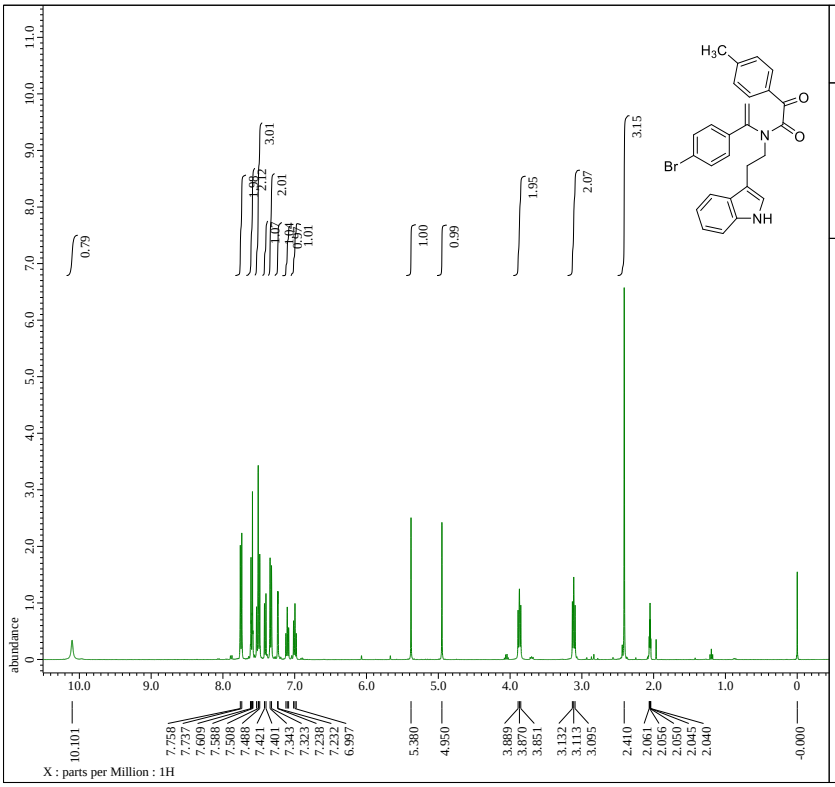
JEOL Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-16-alpha-3-C-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#335981
Solvent           = CHLOROFORM-D
Creation_Time     =  4-MAR-2016 10:05:50
Revision_Time     =  4-MAR-2016 10:10:01
Current_Time      = 24-OCT-2017 23:16:53

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 850
Total_Scans       = 850

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 19.9[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-16-beta-3-H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#367199
Solvent = CHLOROFORM-D
Creation_Time = 4-MAR-2016 10:14:09
Revision_Time = 24-OCT-2017 23:34:15
Current_Time = 24-OCT-2017 23:34:19
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 18.7[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 3.4[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
abundance
X : parts per Million : 1H
Br
Br
O
O
N
NH


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-16-beta-3-C-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#368351
Solvent = CHLOROFORM-D
Creation_Time = 4-MAR-2016 11:07:23
Revision_Time = 4-MAR-2016 11:27:53
Current_Time = 24-OCT-2017 23:34:50
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 1000
Total_Scans = 1000
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 19.8[dC]
X_90_Width = 11[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 7.8[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
abundance
X : parts per Million : 13C
Br
Br
O
O
N
NH



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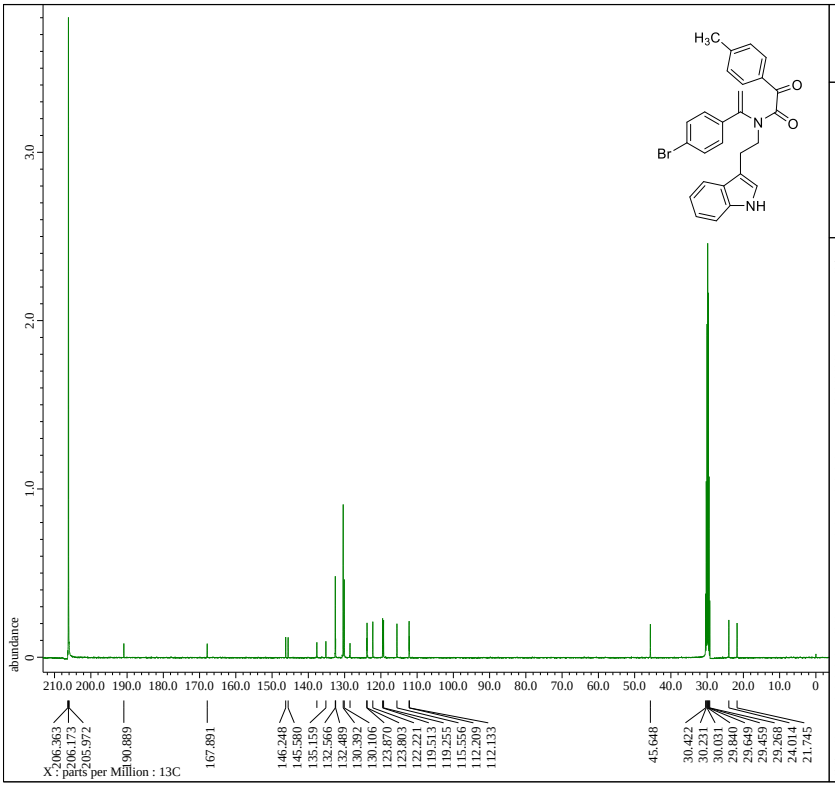
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-20-4-H-7.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#336211
Solvent           = ACETONE-D6
Creation_Time     =  9-MAR-2016 09:22:16
Revision_Time     = 24-OCT-2017 23:32:12
Current_Time      = 24-OCT-2017 23:32:23

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 44
Temp_Get          = 19.3[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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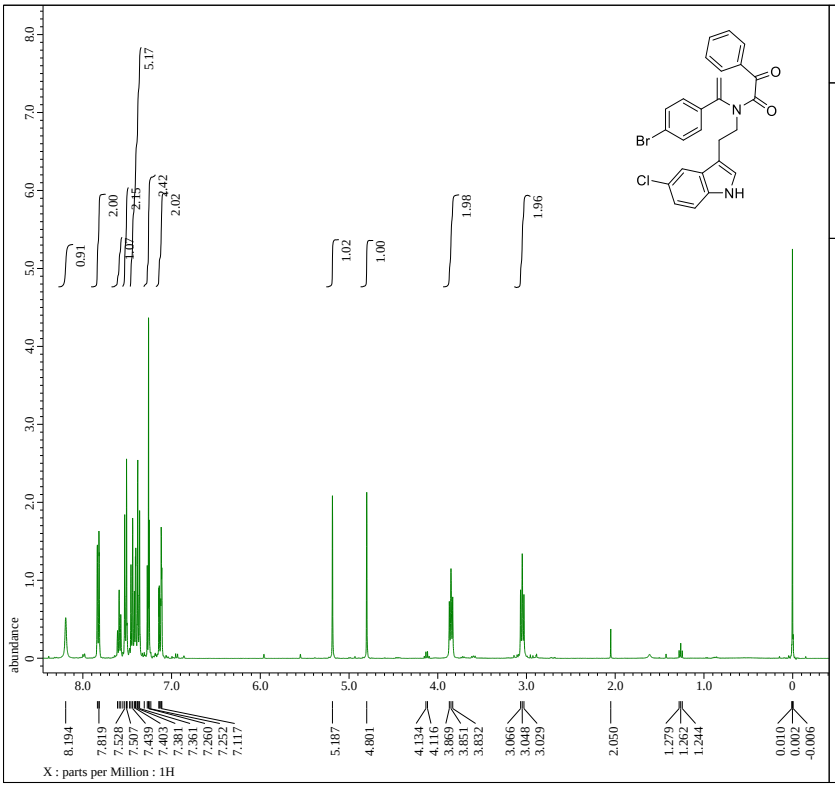
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-20-4-C-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#337511
Solvent           = ACETONE-D6
Creation_Time     =  9-MAR-2016 10:14:03
Revision_Time     =  9-MAR-2016 10:15:48
Current_Time      = 24-OCT-2017 23:32:58

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 962.0
Total_Scans       = 962.0

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 19.6[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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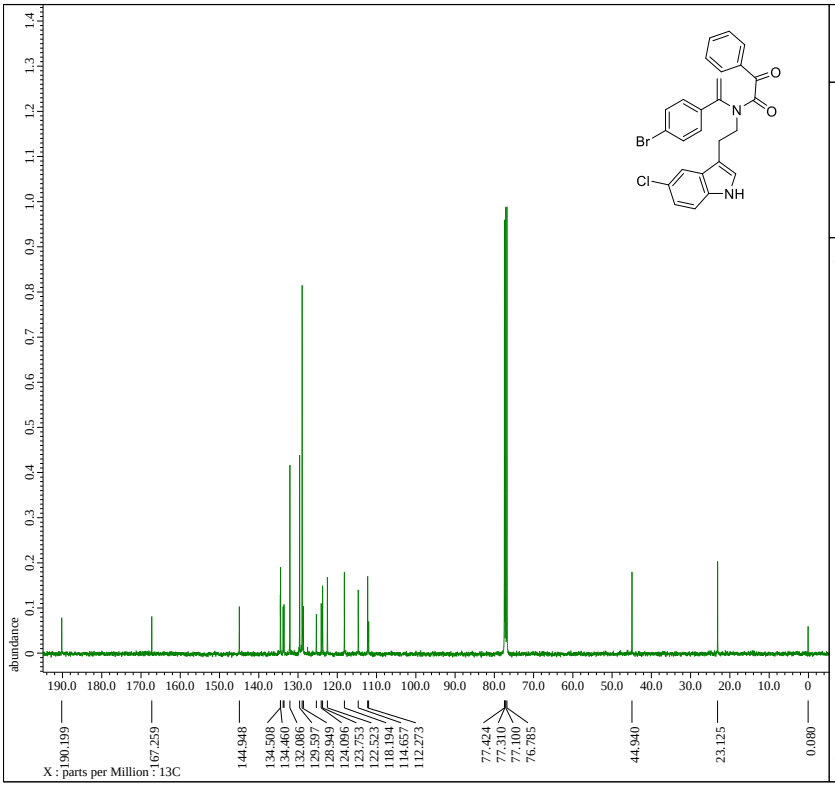
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-27-3-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#806400
Solvent           = CHLOROFORM-D
Creation_Time     = 23-MAR-2016 22:26:14
Revision_Time     = 24-OCT-2017 22:56:18
Current_Time      = 24-OCT-2017 22:56:22

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 18.7[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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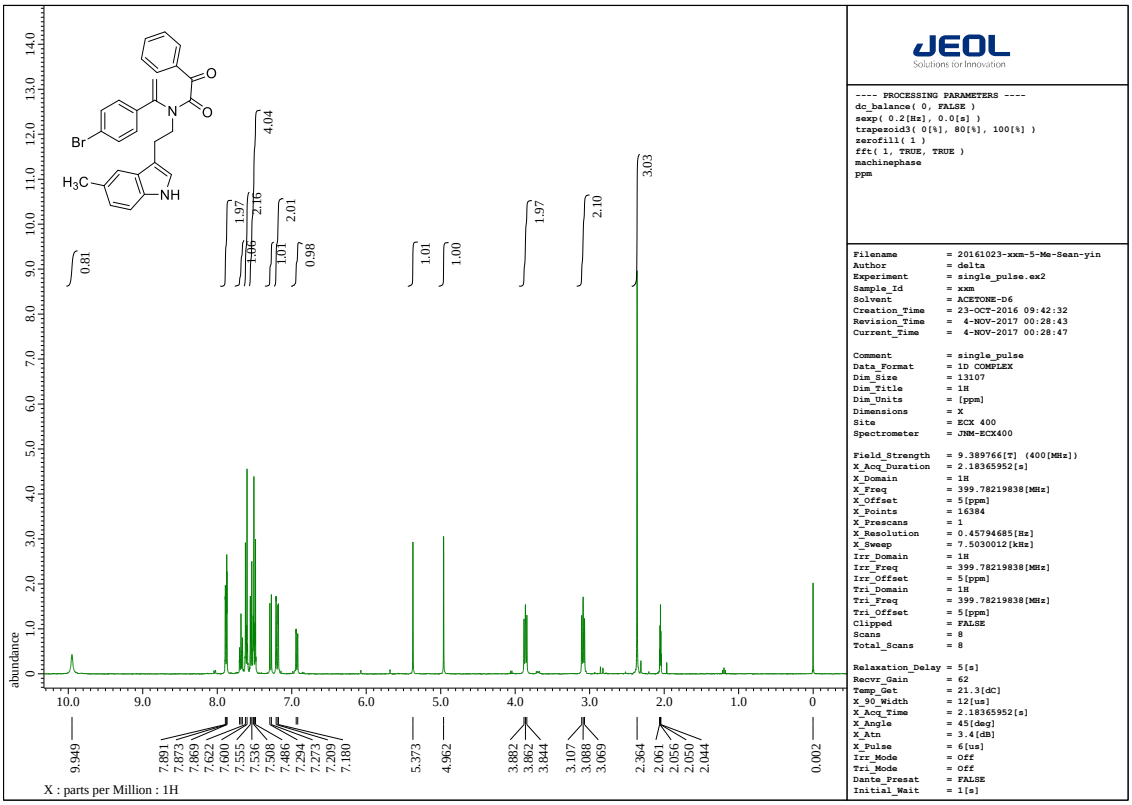
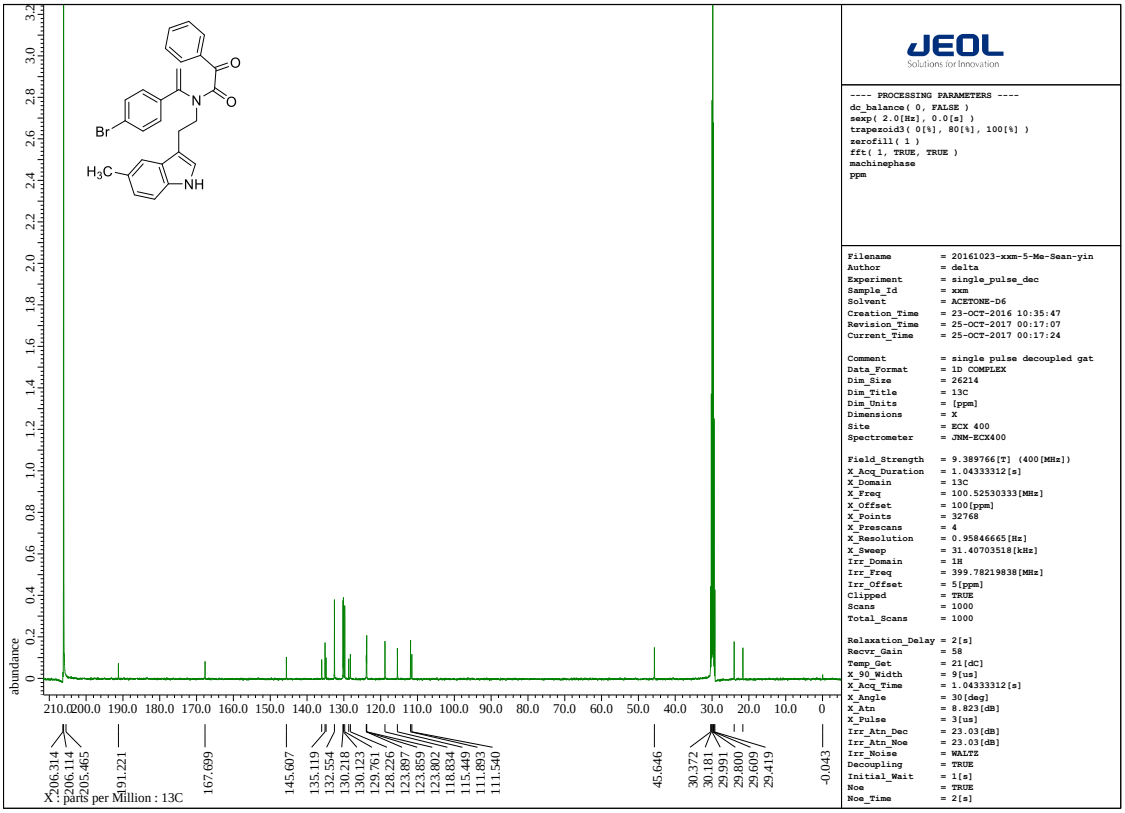
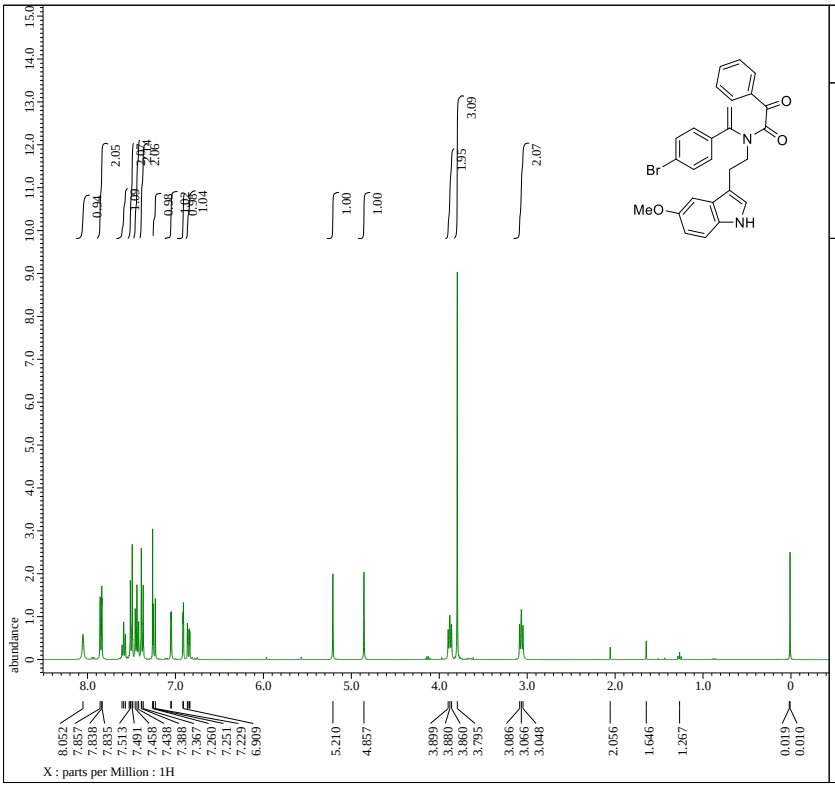
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = mx-A-27-3-C-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#377196
Solvent           = CHLOROFORM-D
Creation_Time     = 25-MAR-2016 11:22:08
Revision_Time     = 24-OCT-2017 22:58:36
Current_Time      = 24-OCT-2017 22:58:54

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 19.6[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20161023-xxm-5-Me-Sean-yin
Author = delta
Experiment = single_pulse.ex2
Sample_Id = xxm
Solvent = ACETONE-D6
Creation_Time = 23-OCT-2016 09:42:32
Revision_Time = 4-NOV-2017 00:28:43
Current_Time = 4-NOV-2017 00:28:47
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 62
Temp_Get = 21.3[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 3.4[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
abundance
X : parts per Million : 1H


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20161023-xxm-5-Me-Sean-yin
Author = delta
Experiment = single_pulse_dec
Sample_Id = xxm
Solvent = ACETONE-D6
Creation_Time = 23-OCT-2016 10:35:47
Revision_Time = 25-OCT-2017 00:17:07
Current_Time = 25-OCT-2017 00:17:24
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 1000
Total_Scans = 1000
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 21[dC]
X_90_Width = 9[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 8.823[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
abundance
X : parts per Million : 13C



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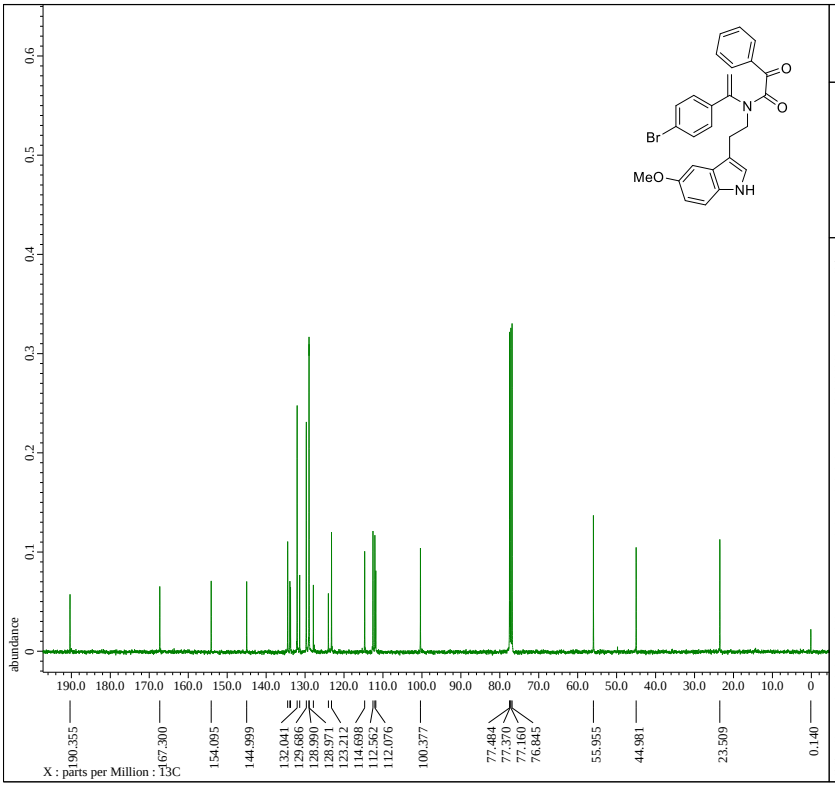
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-22-3-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = s#313973
Solvent           = CHLOROFORM-D
Creation_Time     = 14-MAR-2016 08:45:29
Revision_Time     = 24-OCT-2017 23:10:24
Current_Time      = 24-OCT-2017 23:10:28

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 19.7[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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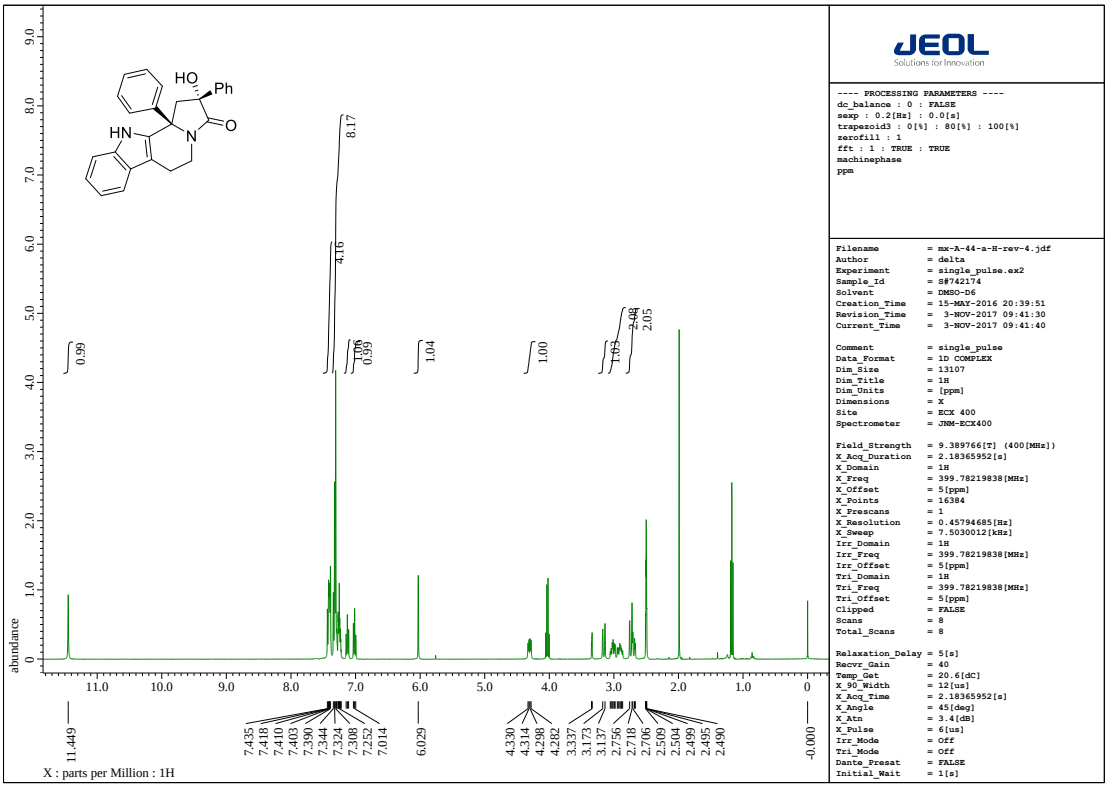
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-22-3-C-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = s#315242
Solvent           = CHLOROFORM-D
Creation_Time     = 14-MAR-2016 09:38:43
Revision_Time     = 14-MAR-2016 09:55:25
Current_Time      = 24-OCT-2017 23:12:34

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.9[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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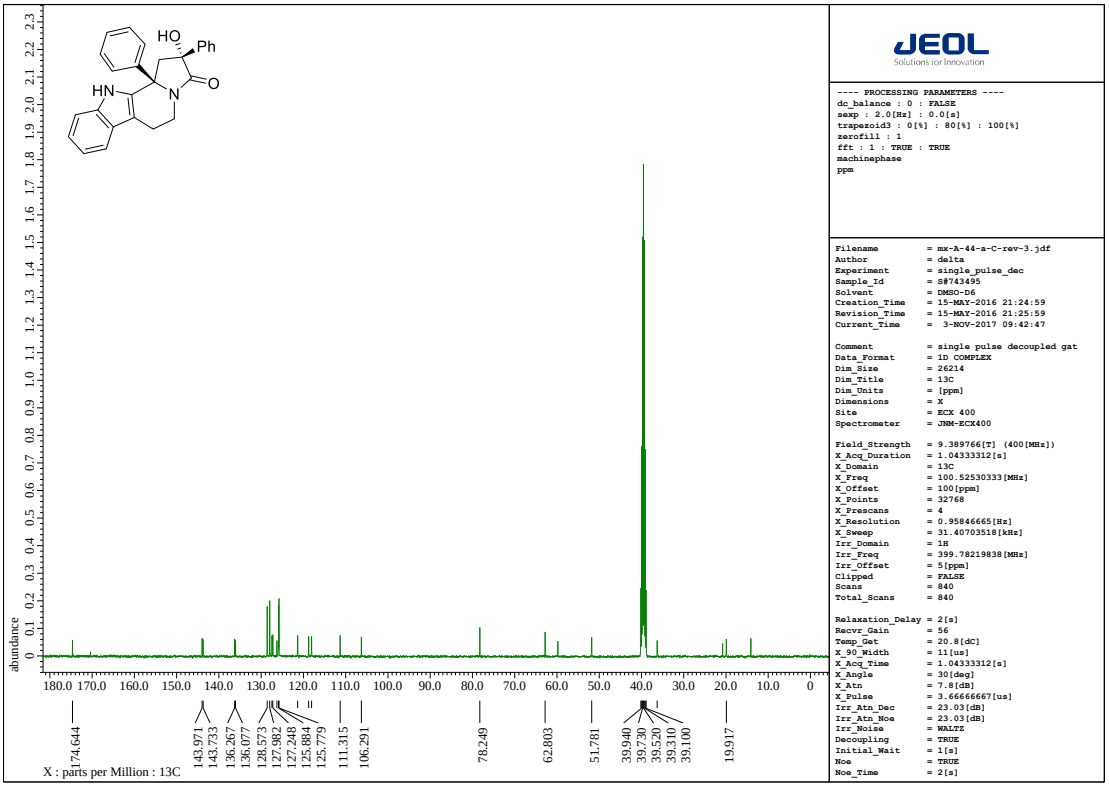
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-44-a-H-rev-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#742174
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 20:39:51
Revision_Time     =  3-NOV-2017 09:41:30
Current_Time      =  3-NOV-2017 09:41:40

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 20.6[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



JEOL Solutions for Innovation

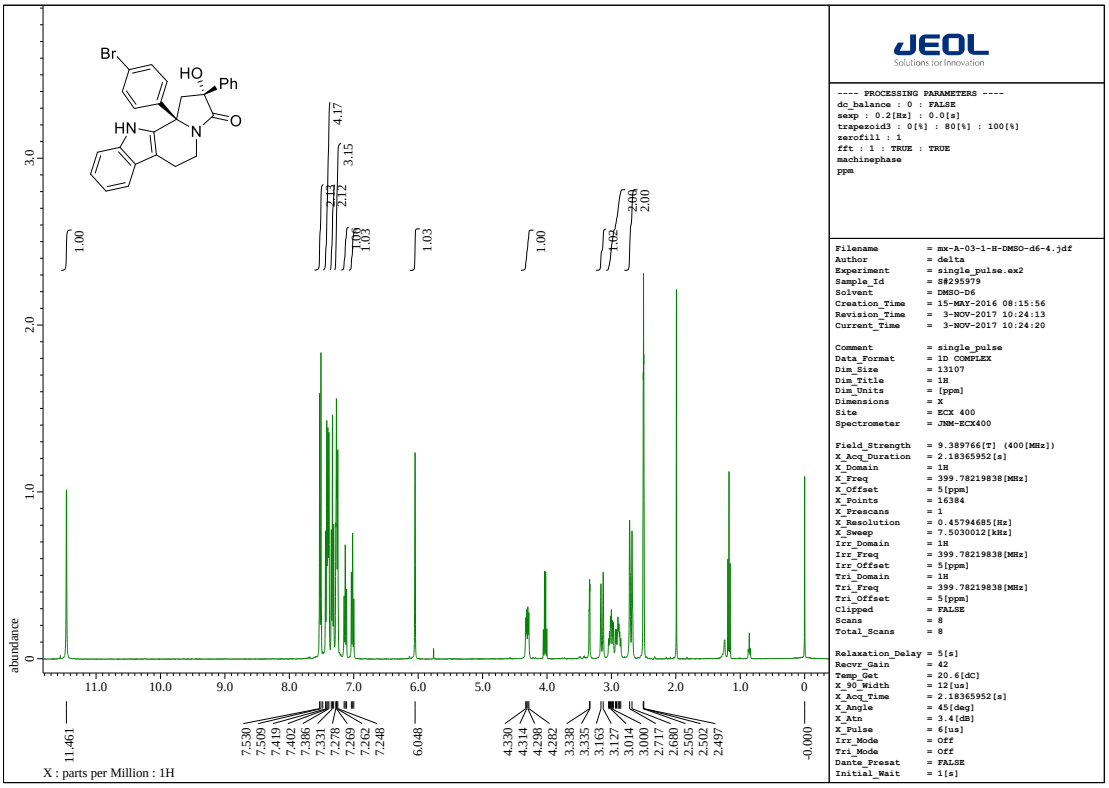
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = mx-A-44-a-C-rev-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#743495
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 21:24:59
Revision_Time     = 15-MAY-2016 21:25:59
Current_Time      =  3-NOV-2017 09:42:47

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 840
Total_Scans       = 840

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 20.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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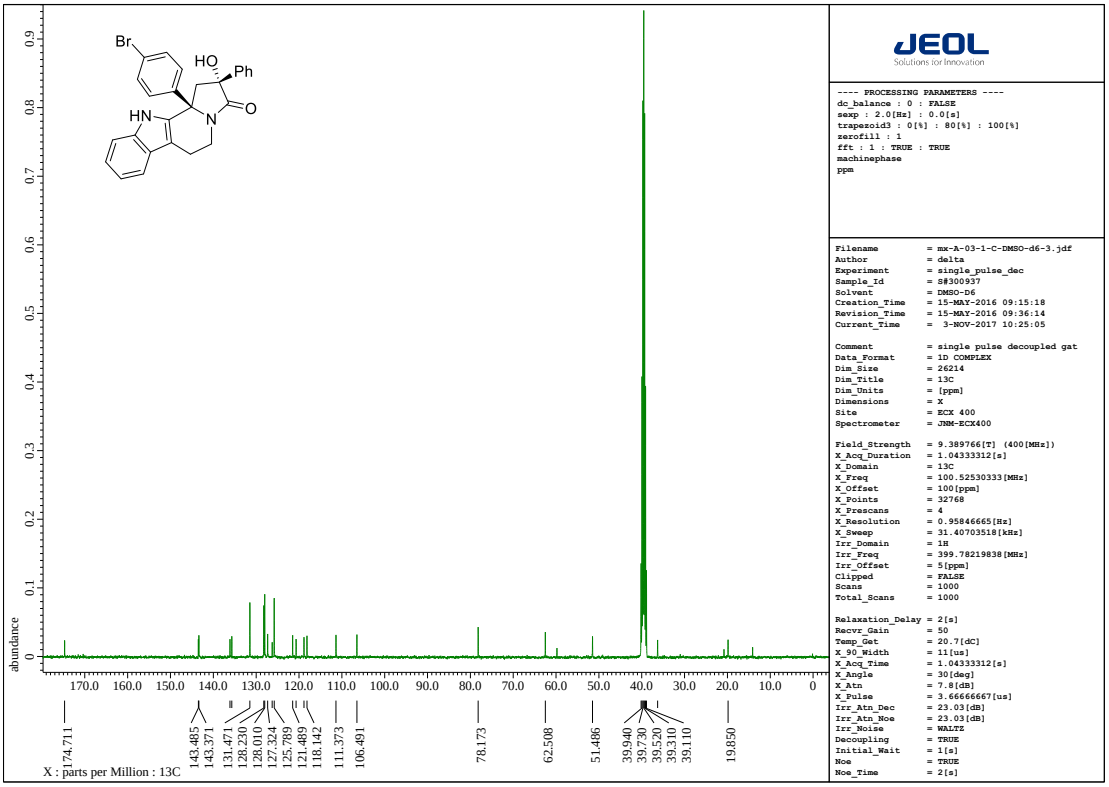
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-03-1-H-DMSO-d6-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = s#295979
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 08:15:56
Revision_Time     =  3-NOV-2017 10:24:13
Current_Time      =  3-NOV-2017 10:24:20

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 20.6[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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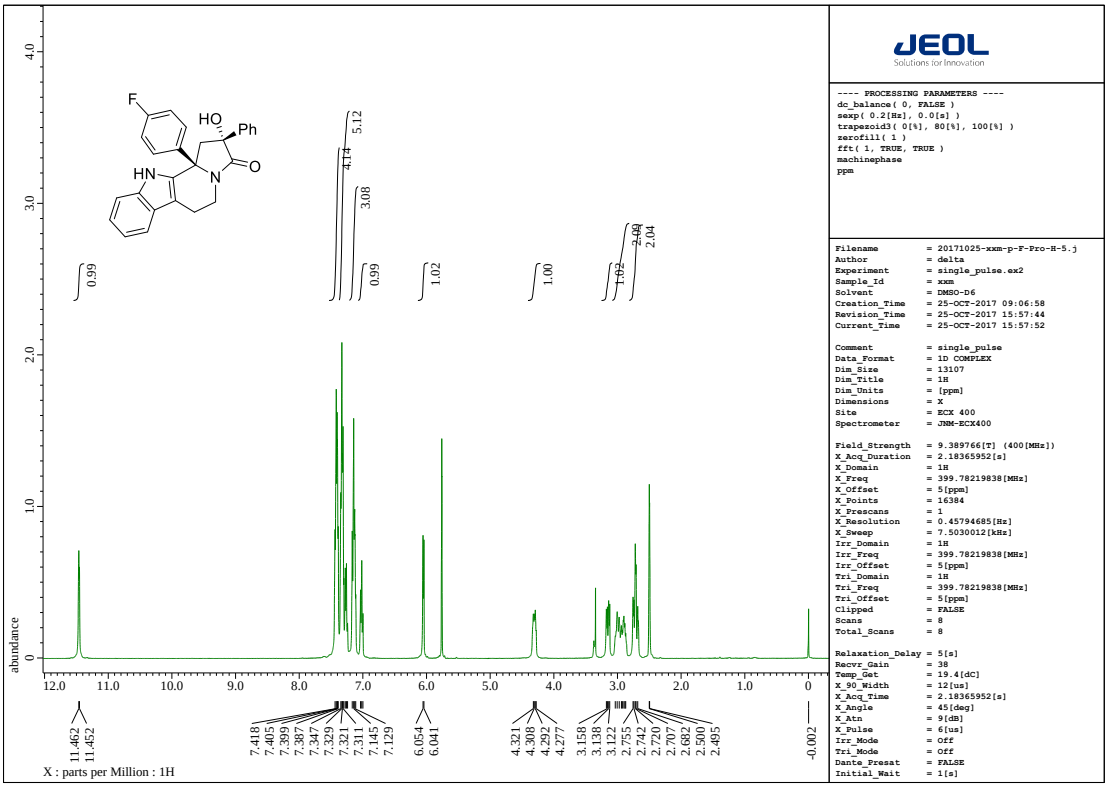
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-03-1-C-DMSO-d6-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = s#300937
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 09:15:18
Revision_Time     = 15-MAY-2016 09:36:14
Current_Time      =  3-NOV-2017 10:25:05

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 20.7[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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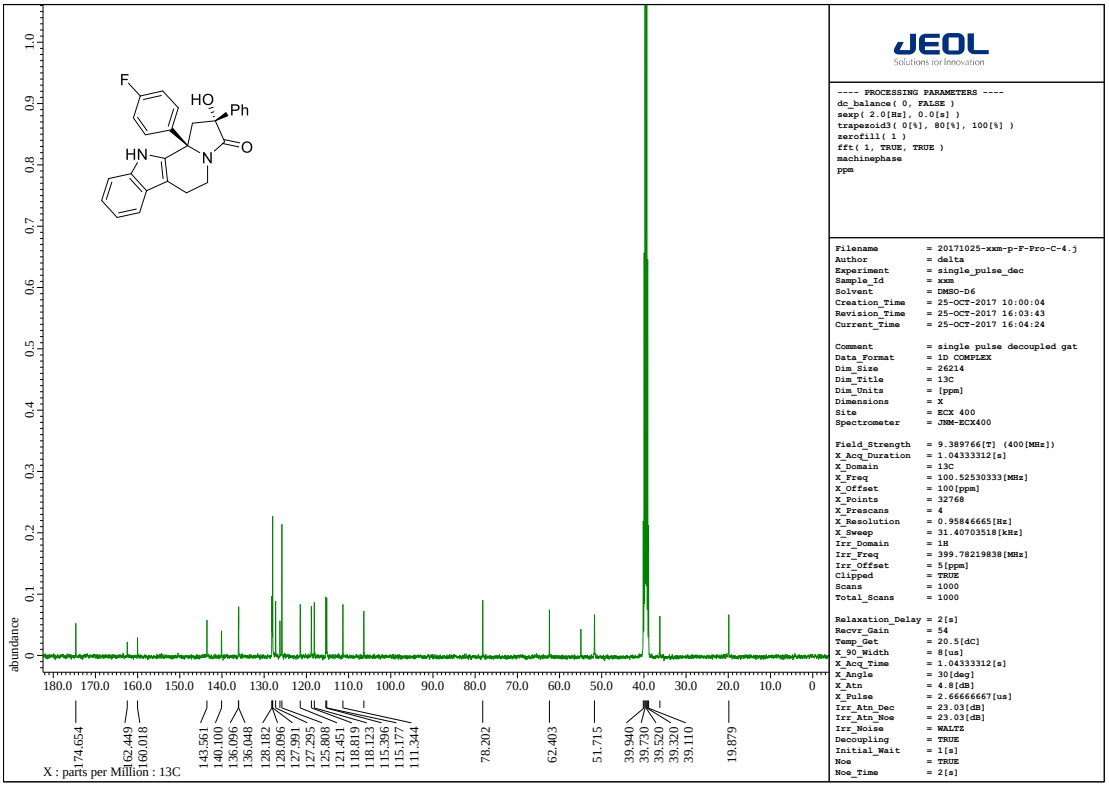
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 20171025-xxm-p-F-Pro-H-5.j
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = xxm
Solvent           = DMSO-D6
Creation_Time     = 25-OCT-2017 09:06:58
Revision_Time     = 25-OCT-2017 15:57:44
Current_Time      = 25-OCT-2017 15:57:52

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 19.4[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 9[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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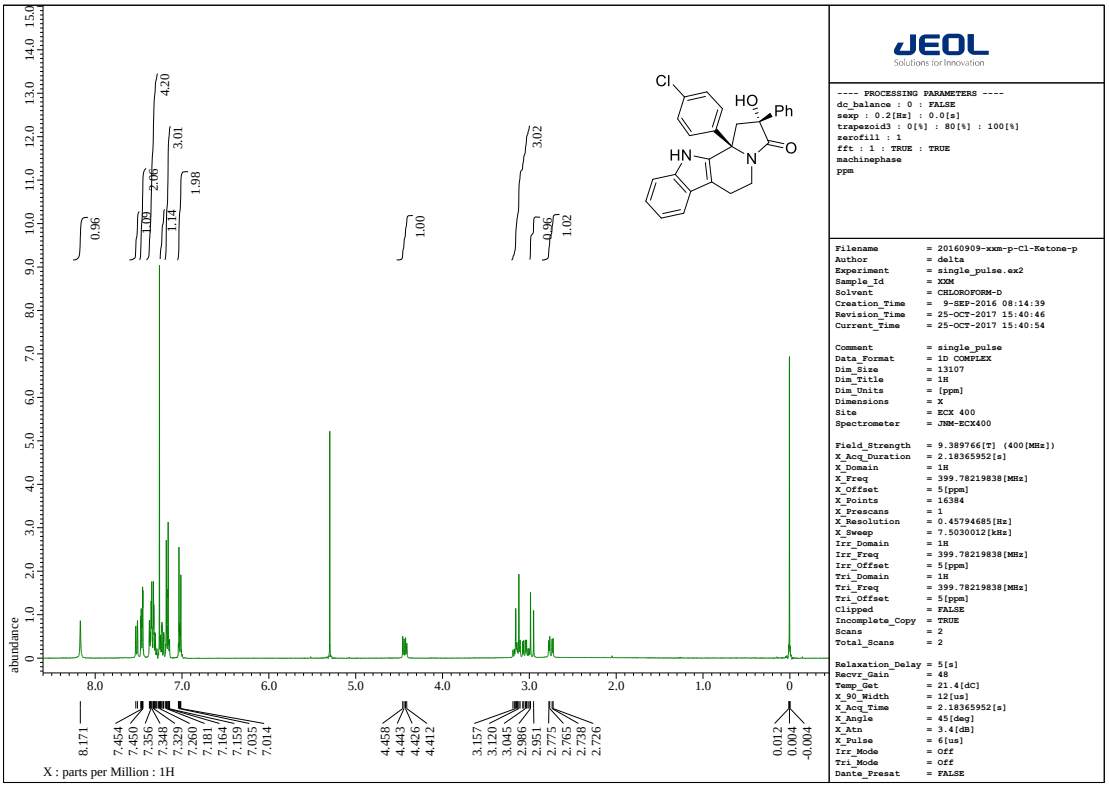
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 20171025-xxm-p-F-Pro-C-4.j
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = xxm
Solvent           = DMSO-D6
Creation_Time     = 25-OCT-2017 10:00:04
Revision_Time     = 25-OCT-2017 16:03:43
Current_Time      = 25-OCT-2017 16:04:24

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 54
Temp_Get          = 20.5[dC]
X_90_Width        = 8[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.8[dB]
X_Pulse           = 2.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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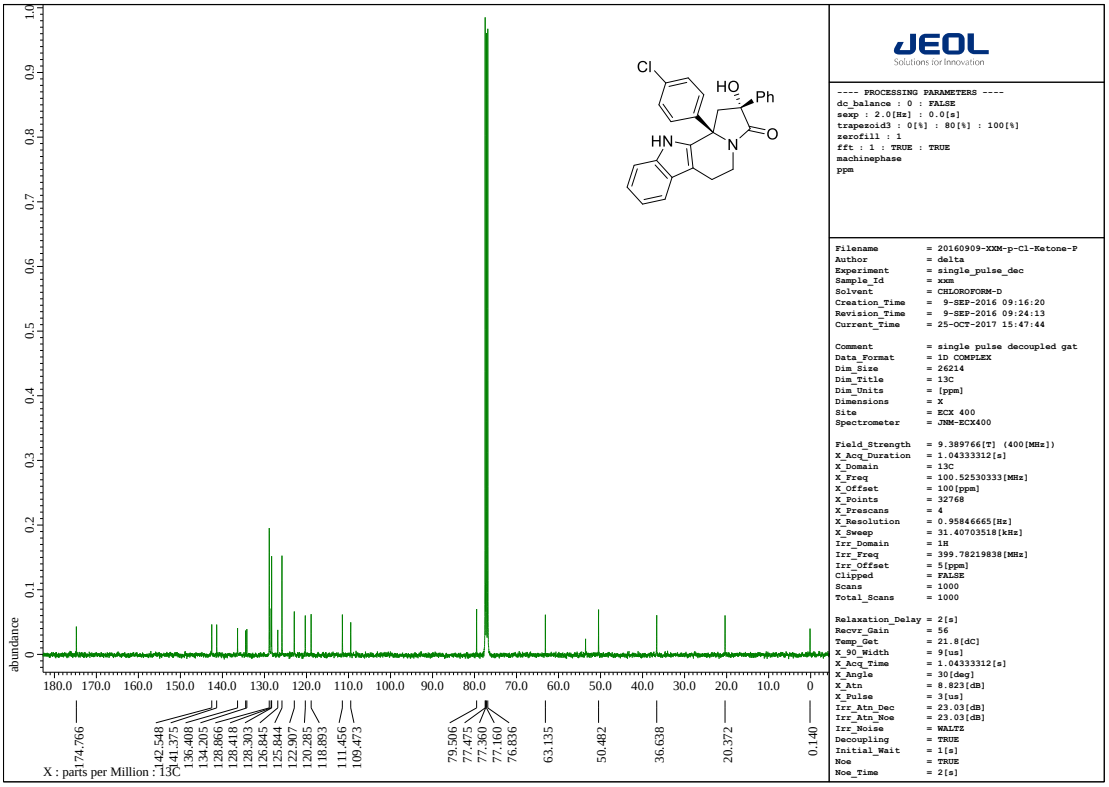
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 20160909-xxm-p-Cl-Ketone-p
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = XXM
Solvent           = CHLOROFORM-D
Creation_Time     =  9-SEP-2016 08:14:39
Revision_Time     = 25-OCT-2017 15:40:46
Current_Time      = 25-OCT-2017 15:40:54

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Incomplete_Copy   = TRUE
Scans             = 2
Total_Scans       = 2

Relaxation_Delay = 5[s]
Recvr_Gain        = 48
Temp_Get          = 21.4[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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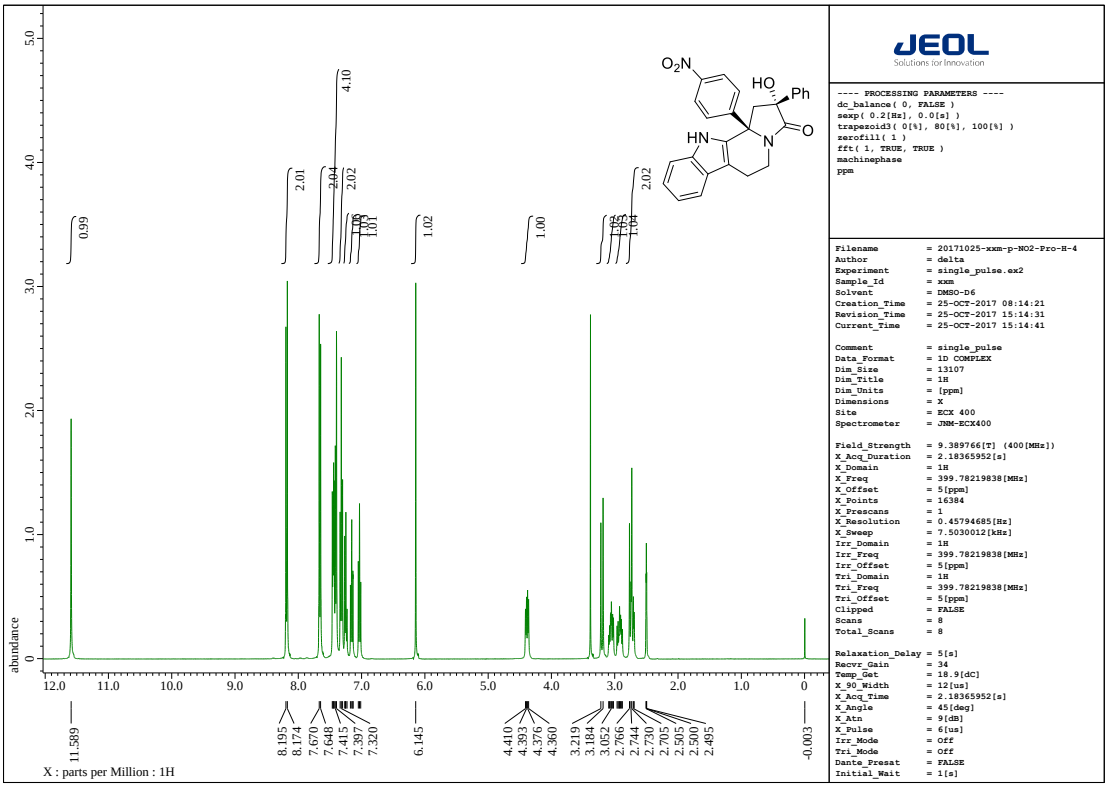
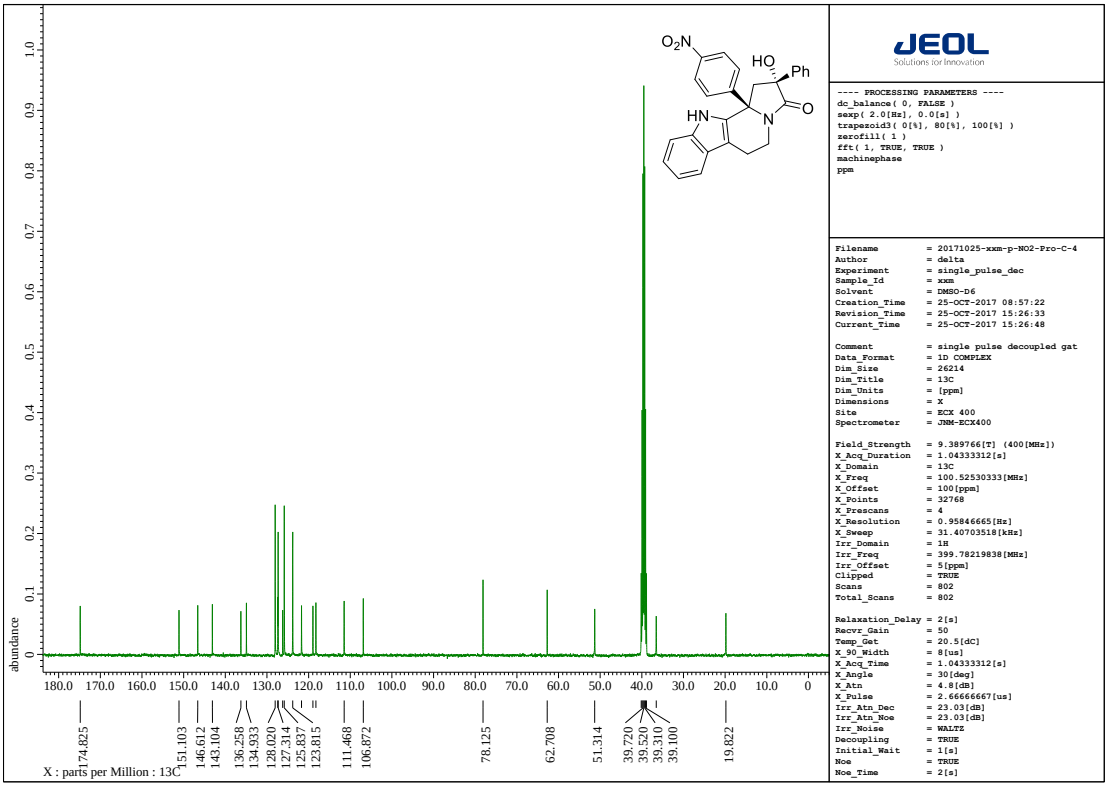
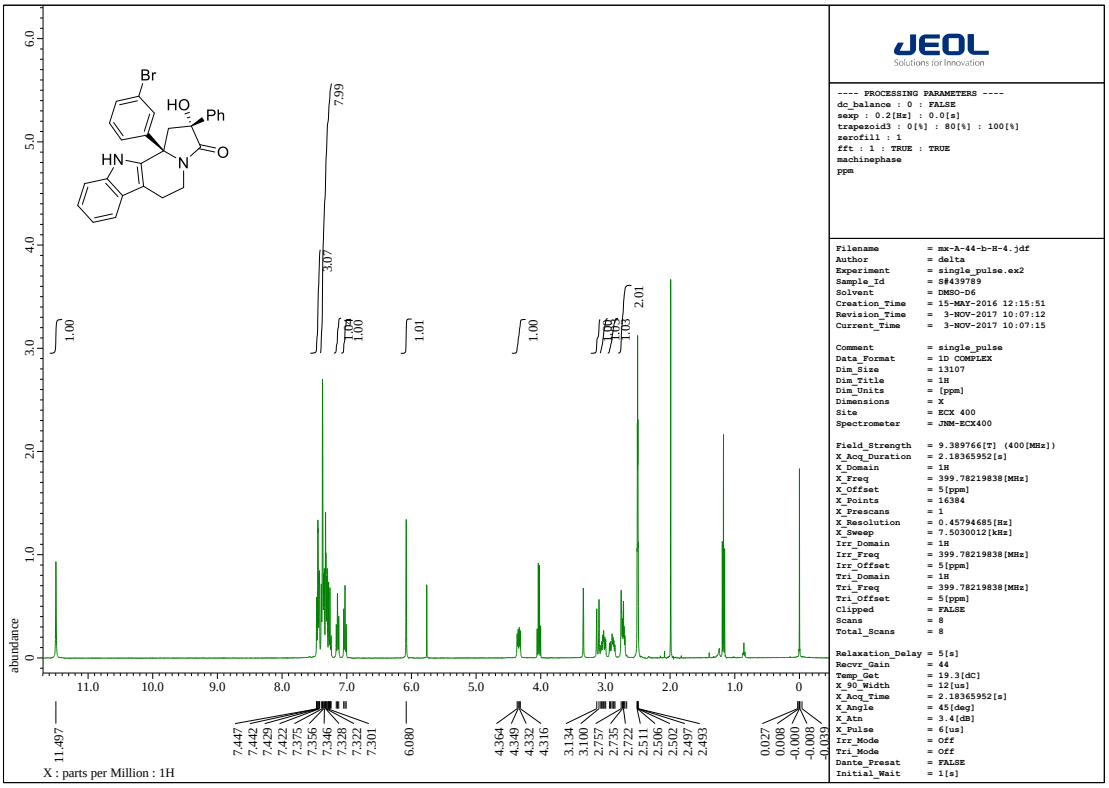
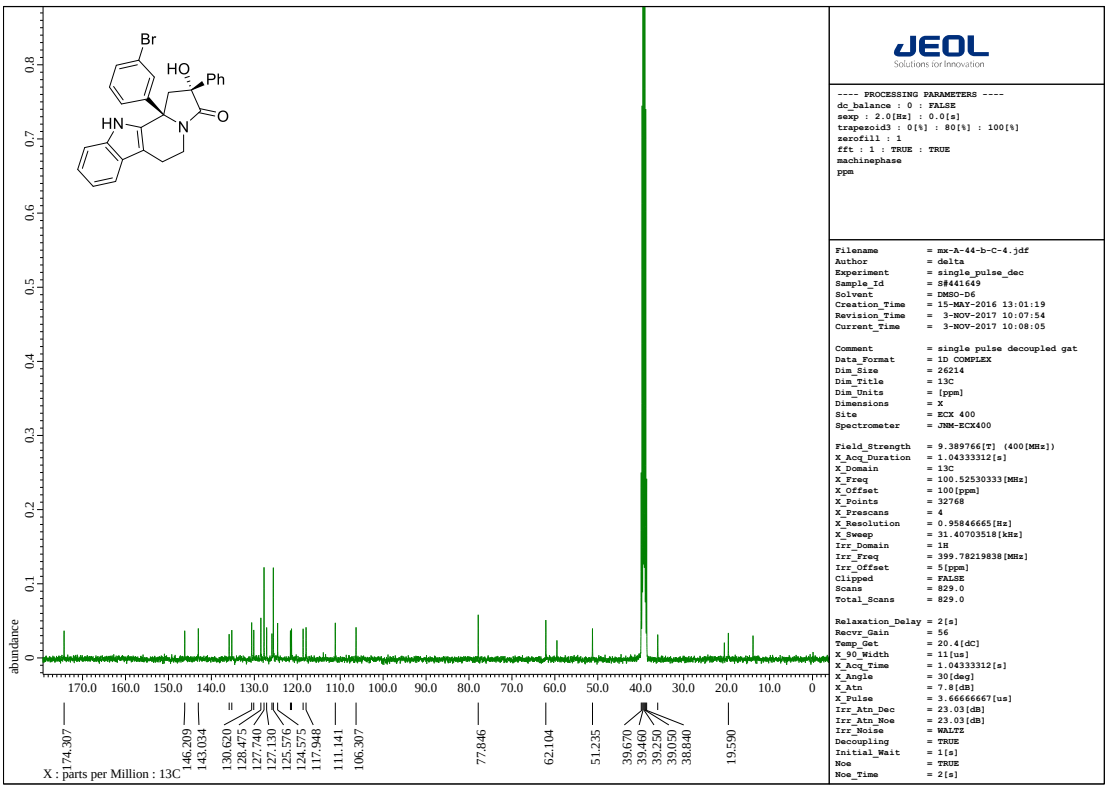
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

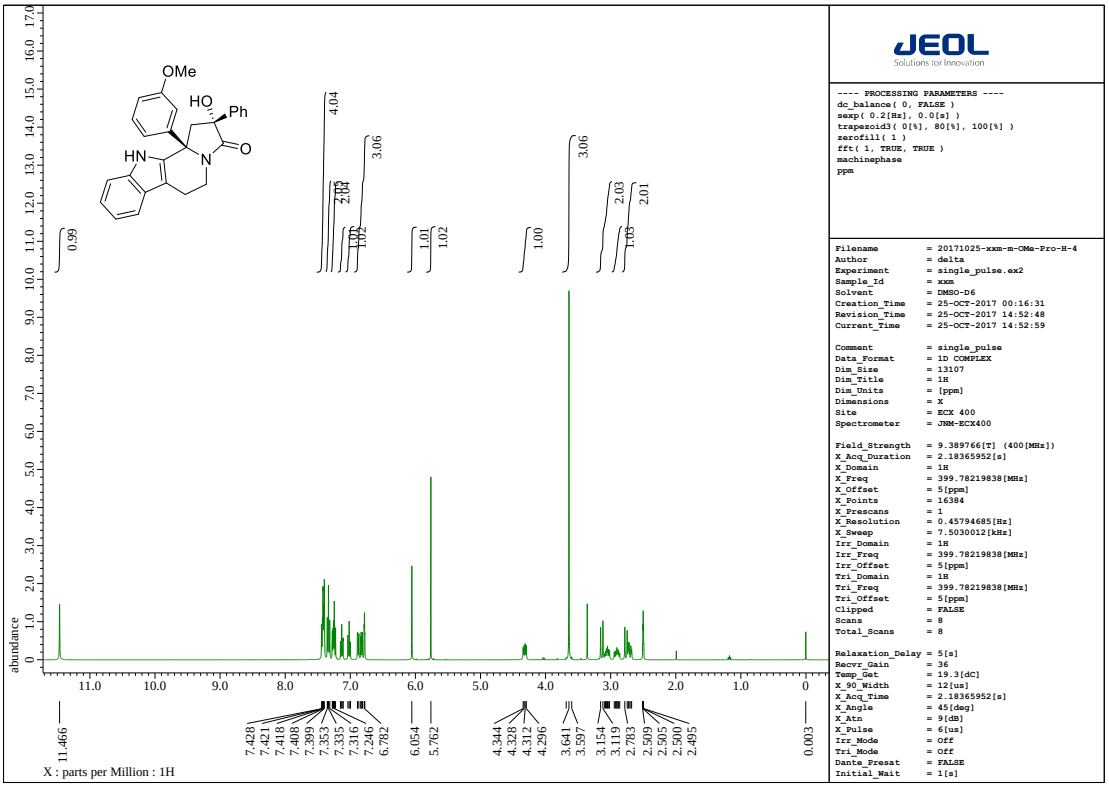
```
Filename          = 20160909-XXM-p-Cl-Ketone-P
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = xxm
Solvent           = CHLOROFORM-D
Creation_Time     =  9-SEP-2016 09:16:20
Revision_Time     =  9-SEP-2016 09:24:13
Current_Time      = 25-OCT-2017 15:47:44

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 21.8[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 8.823[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20171025-xxm-p-NO2-Pro-H-4
Author = delta
Experiment = single_pulse.ex2
Sample_Id = xxm
Solvent = DMSO-D6
Creation_Time = 25-OCT-2017 08:14:21
Revision_Time = 25-OCT-2017 15:14:31
Current_Time = 25-OCT-2017 15:14:41
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 18.9[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 9[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
abundance
X : parts per Million : 1H


JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 20171025-xxm-p-NO2-Pro-C-4
Author = delta
Experiment = single_pulse_dec
Sample_Id = xxm
Solvent = DMSO-D6
Creation_Time = 25-OCT-2017 08:57:22
Revision_Time = 25-OCT-2017 15:26:33
Current_Time = 25-OCT-2017 15:26:48
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 802
Total_Scans = 802
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.5[dC]
X_90_Width = 8[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4.8[dB]
X_Pulse = 2.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
abundance
X : parts per Million : 13C


Br
HO
Ph
HN
N
O
abundance
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-44-b-H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#439789
Solvent = DMSO-D6
Creation_Time = 15-MAY-2016 12:15:51
Revision_Time = 3-NOV-2017 10:07:12
Current_Time = 3-NOV-2017 10:07:15
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 44
Temp_Get = 19.3[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 3.4[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]


Br
HO
Ph
HN
N
O
abundance
X : parts per Million : 13C
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-44-b-C-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#441649
Solvent = DMSO-D6
Creation_Time = 15-MAY-2016 13:01:19
Revision_Time = 3-NOV-2017 10:07:54
Current_Time = 3-NOV-2017 10:08:05
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 829.0
Total_Scans = 829.0
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 20.4[dC]
X_90_Width = 11[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 7.8[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]



JEOL
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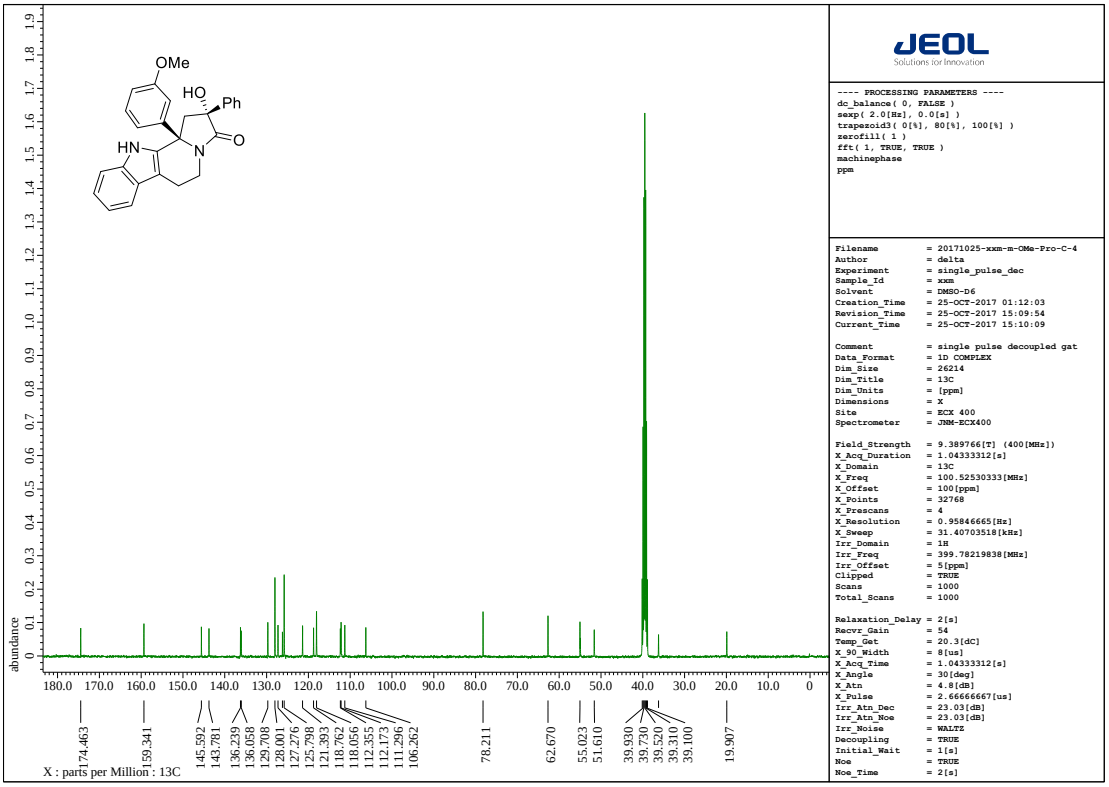
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

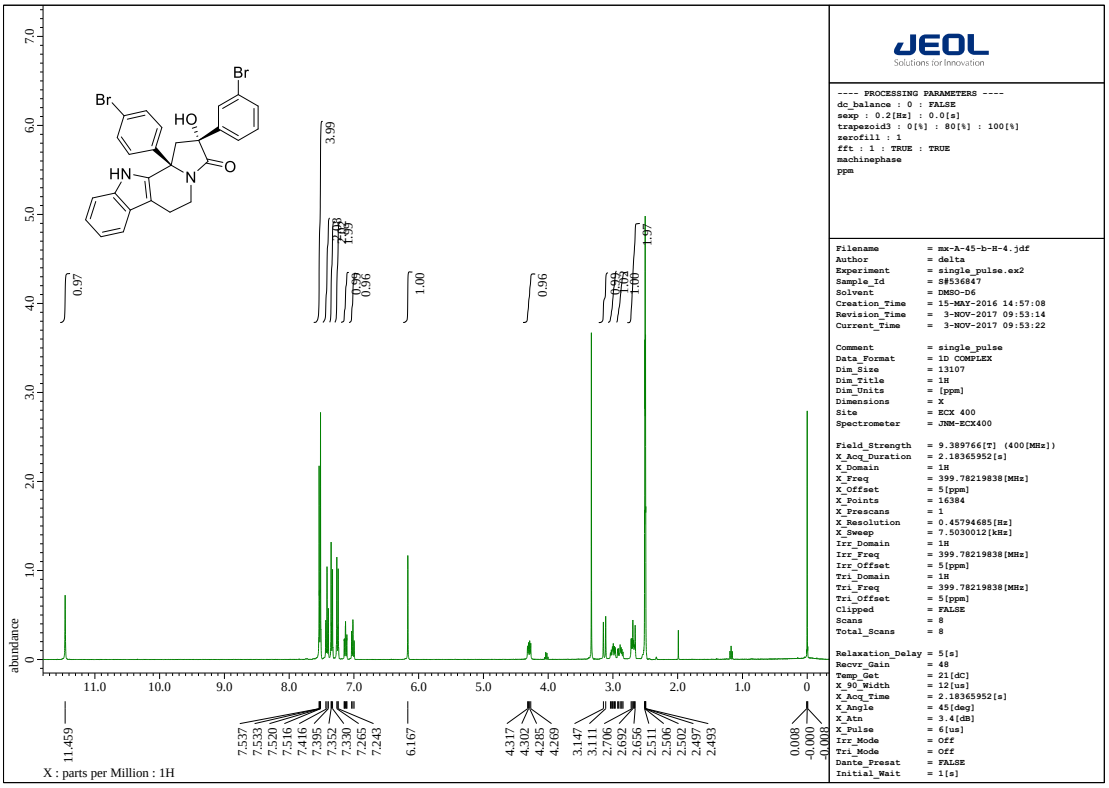
```
Filename          = 20171025-xxm-m-OMe-Pro-H-4
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = xxm
Solvent           = DMSO-D6
Creation_Time     = 25-OCT-2017 00:16:31
Revision_Time     = 25-OCT-2017 14:52:48
Current_Time      = 25-OCT-2017 14:52:59

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 19.3[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 9[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



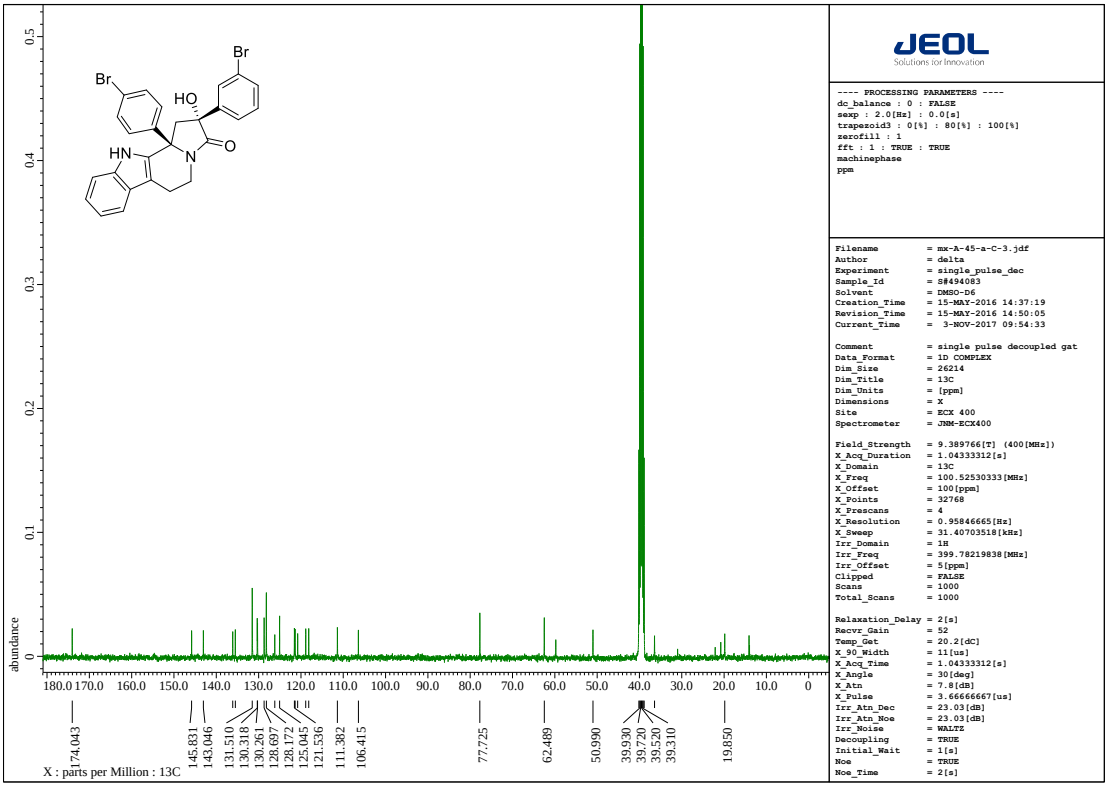
JEOL
Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

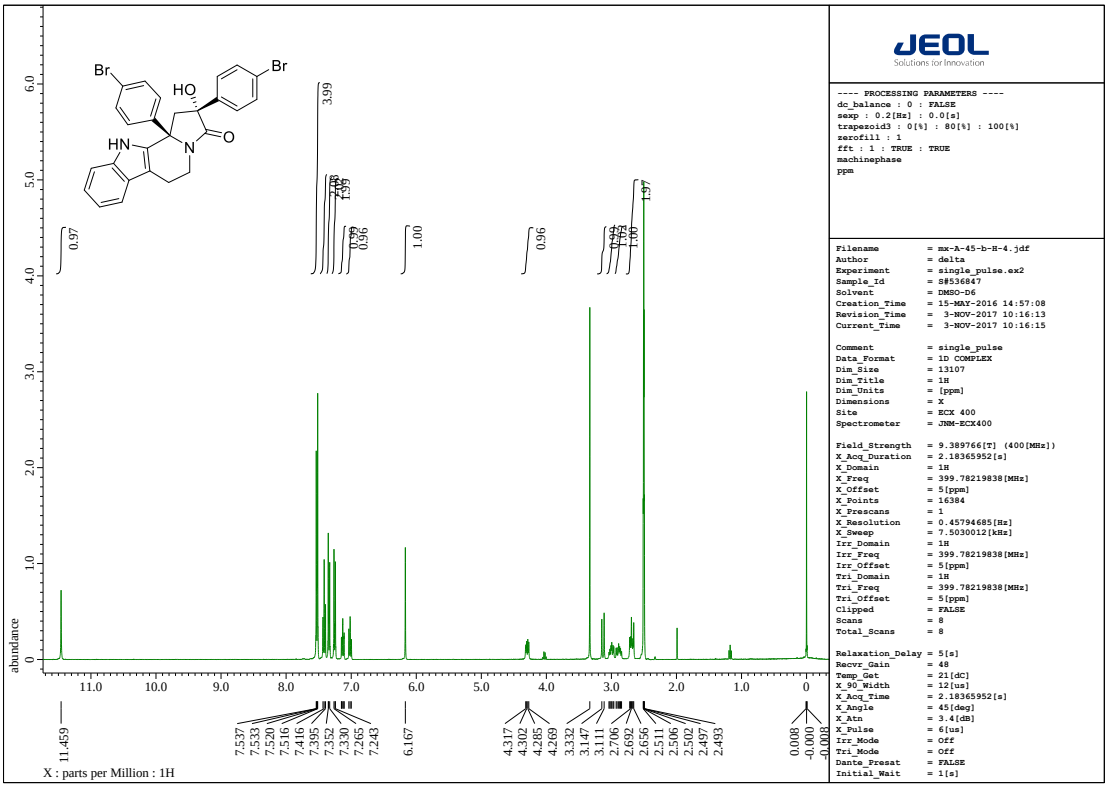
```
Filename          = 20171025-xxm-m-OMe-Pro-C-4
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = xxm
Solvent           = DMSO-D6
Creation_Time     = 25-OCT-2017 01:12:03
Revision_Time     = 25-OCT-2017 15:09:54
Current_Time      = 25-OCT-2017 15:10:09

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 54
Temp_Get          = 20.3[dC]
X_90_Width        = 8[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.8[dB]
X_Pulse           = 2.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```

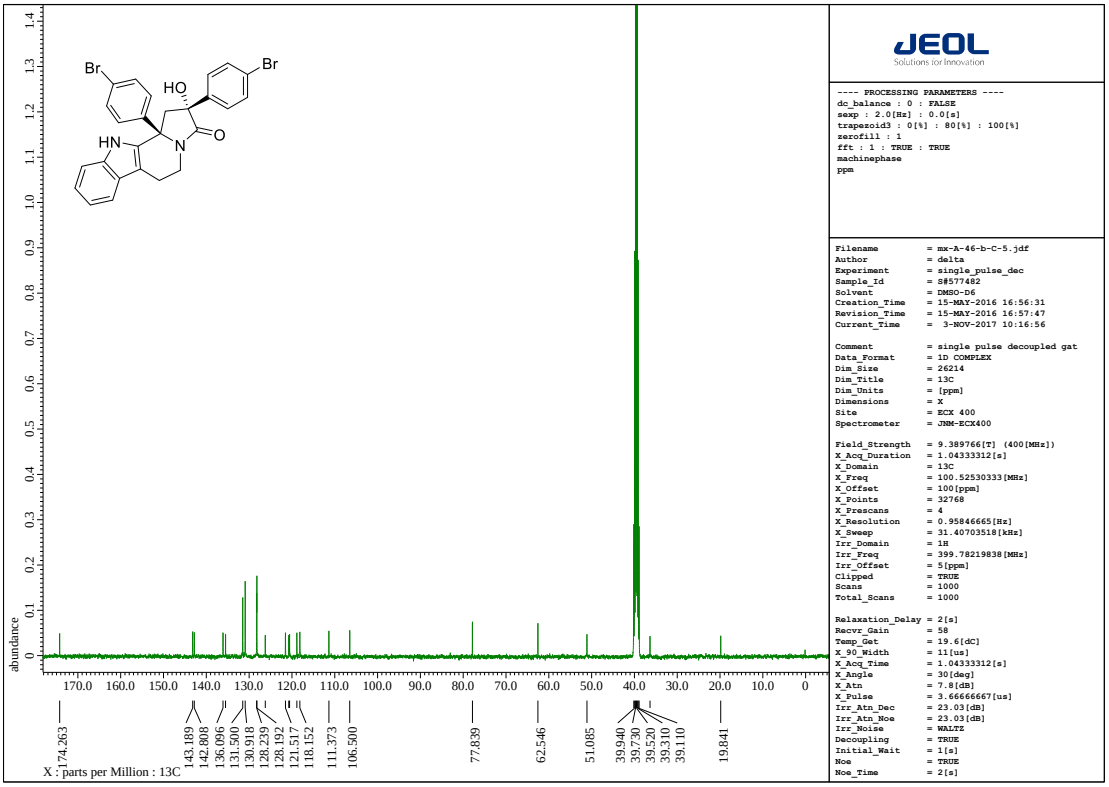


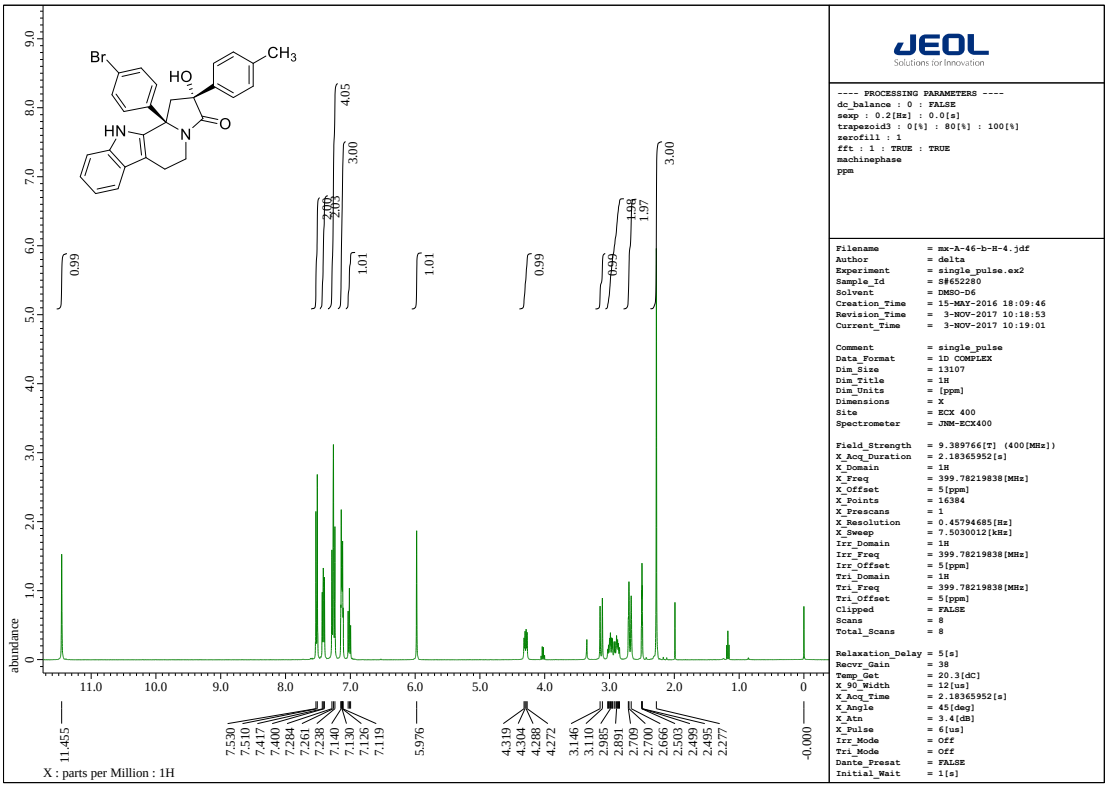

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-45-b-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#536847
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 14:57:08
Revision_Time     =  3-NOV-2017 09:53:14
Current_Time      =  3-NOV-2017 09:53:22

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 48
Temp_Get          = 21[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-45-a-C-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#494083
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 14:37:19
Revision_Time     = 15-MAY-2016 14:50:05
Current_Time      =  3-NOV-2017 09:54:33

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 52
Temp_Get          = 20.2[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```


Br
HO
Br
HN
N
O
abundance
X : parts per Million : 1H
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-45-b-H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = s#536847
Solvent = DMSO-D6
Creation_Time = 15-MAY-2016 14:57:08
Revision_Time = 3-NOV-2017 10:16:13
Current_Time = 3-NOV-2017 10:16:15
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 21[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 3.4[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]


Br
HO
Br
HN
N
O
abundance
X : parts per Million : 13C
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-46-b-C-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = s#577482
Solvent = DMSO-D6
Creation_Time = 15-MAY-2016 16:56:31
Revision_Time = 15-MAY-2016 16:57:47
Current_Time = 3-NOV-2017 10:16:56
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 1000
Total_Scans = 1000
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 19.6[dC]
X_90_Width = 11[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 7.8[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]



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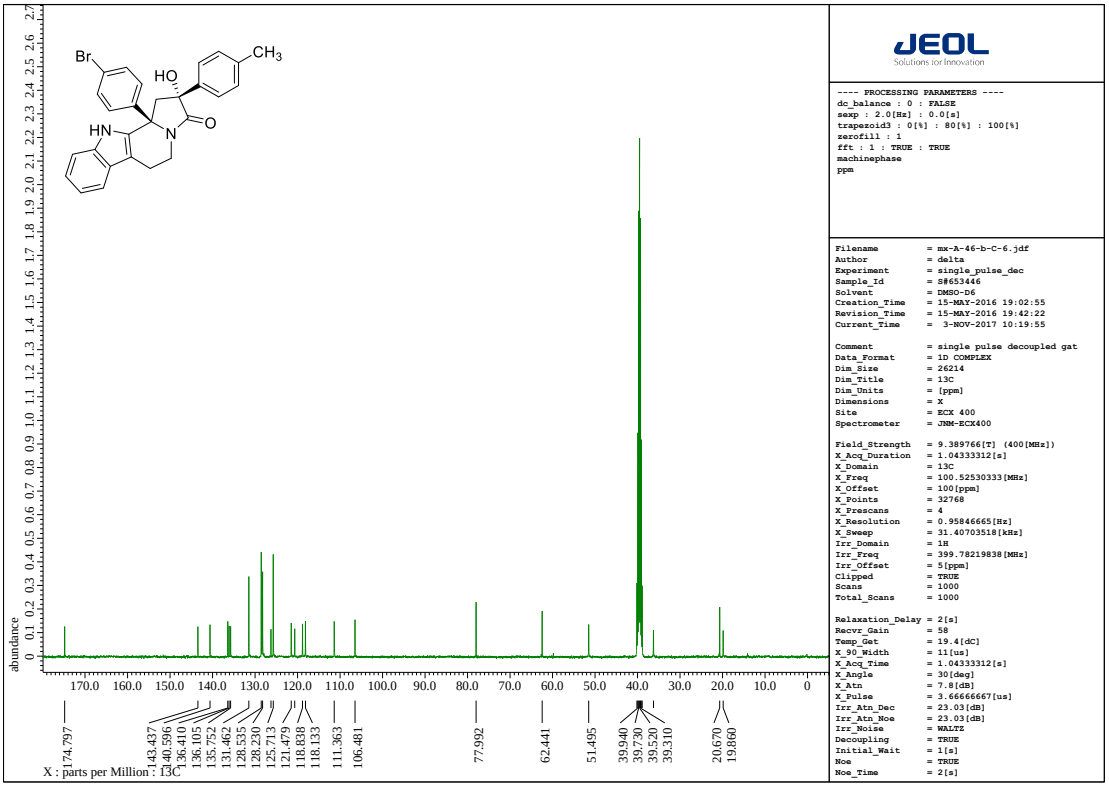
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-46-b-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#652280
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 18:09:46
Revision_Time     =  3-NOV-2017 10:18:53
Current_Time      =  3-NOV-2017 10:19:01

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 38
Temp_Get          = 20.3[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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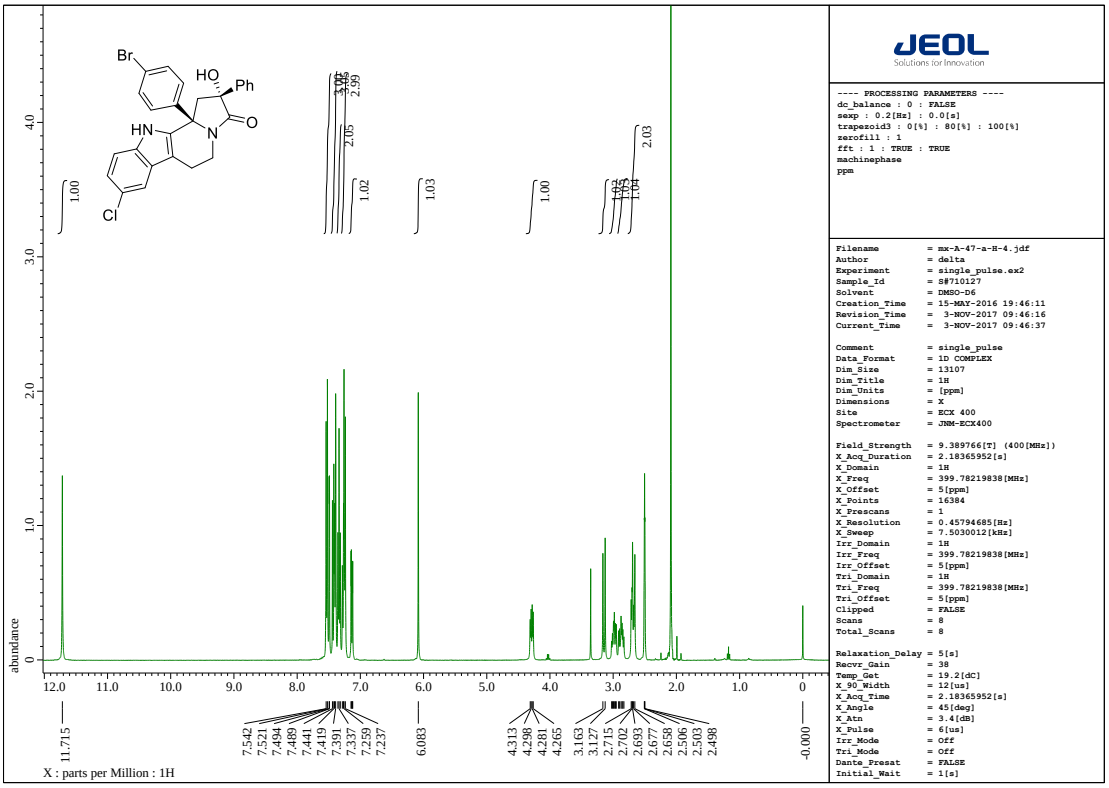
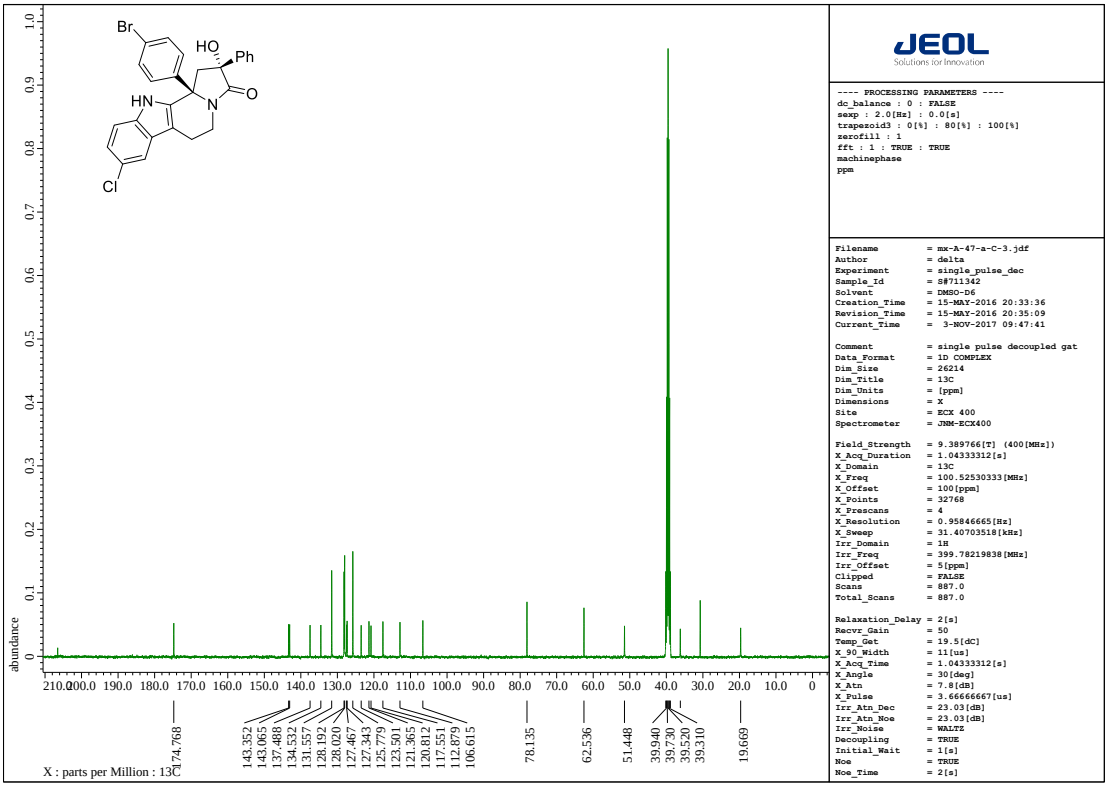
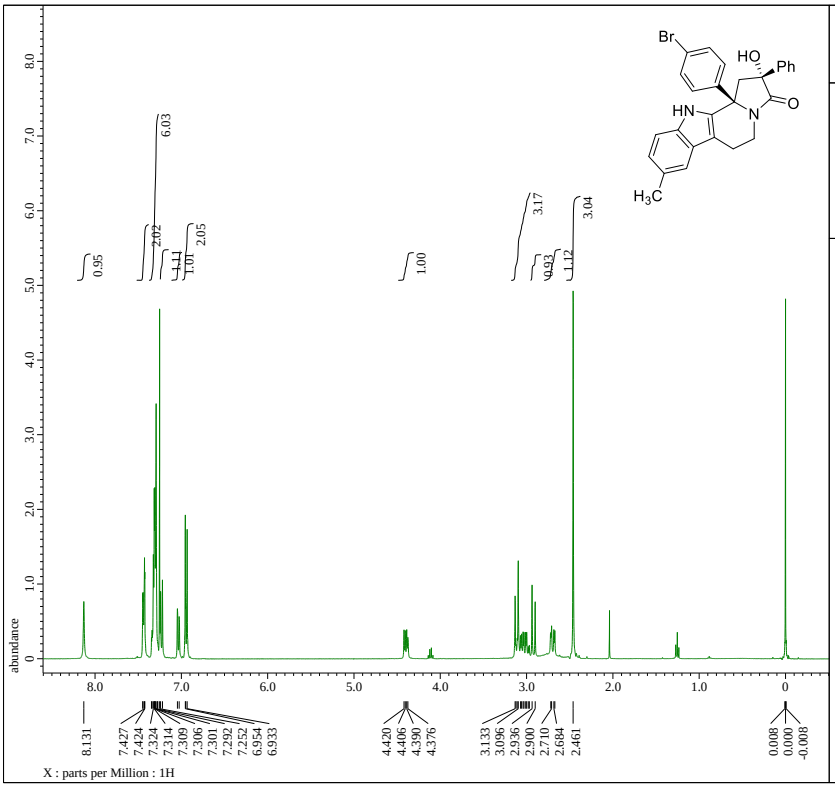
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-46-b-C-6.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#653446
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 19:02:55
Revision_Time     = 15-MAY-2016 19:42:22
Current_Time      =  3-NOV-2017 10:19:55

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 19.4[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```

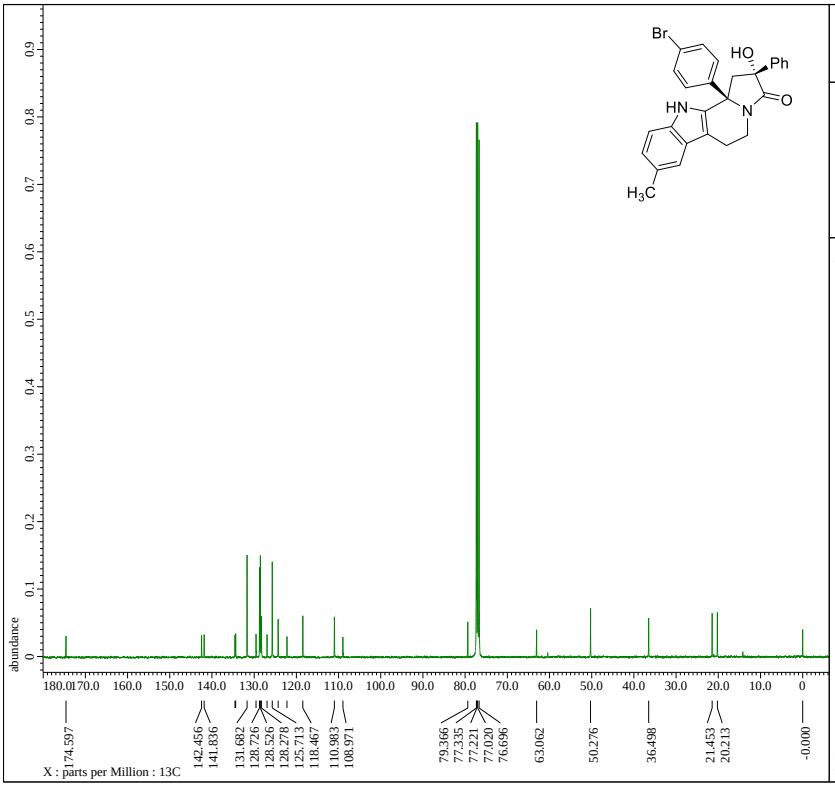

Br
HO
Ph
HN
N
O
Cl
abundance
X : parts per Million : 1H
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-47-a-H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_Id = S#710127
Solvent = DMSO-D6
Creation_Time = 15-MAY-2016 19:46:11
Revision_Time = 3-NOV-2017 09:46:16
Current_Time = 3-NOV-2017 09:46:37
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 19.2[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 3.4[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]


Br
HO
Ph
HN
N
O
Cl
abundance
X : parts per Million : 13C
JEOL
Solutions for Innovation
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Filename = mx-A-47-a-C-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S#711342
Solvent = DMSO-D6
Creation_Time = 15-MAY-2016 20:33:36
Revision_Time = 15-MAY-2016 20:35:09
Current_Time = 3-NOV-2017 09:47:41
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
Irr_Domain = 1H
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 887.0
Total_Scans = 887.0
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.5[dC]
X_90_Width = 11[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 7.8[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 23.03[dB]
Irr_Atn_Noe = 23.03[dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]



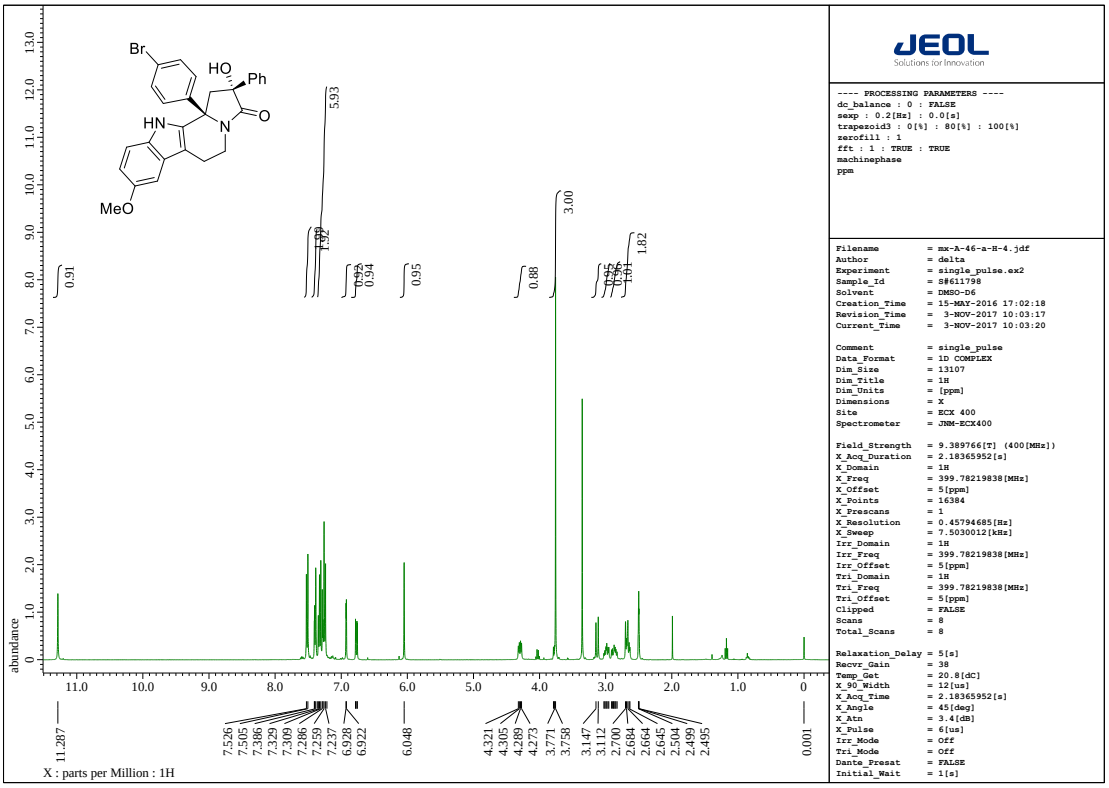
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename	= mx-A-47-b-H-6.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#825445
Solvent	= CHLOROFORM-D
Creation_Time	= 15-MAY-2016 22:58:36
Revision_Time	= 3-NOV-2017 10:11:58
Current_Time	= 3-NOV-2017 10:12:01
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 40
Temp_Get	= 19.3[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 3.4[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]

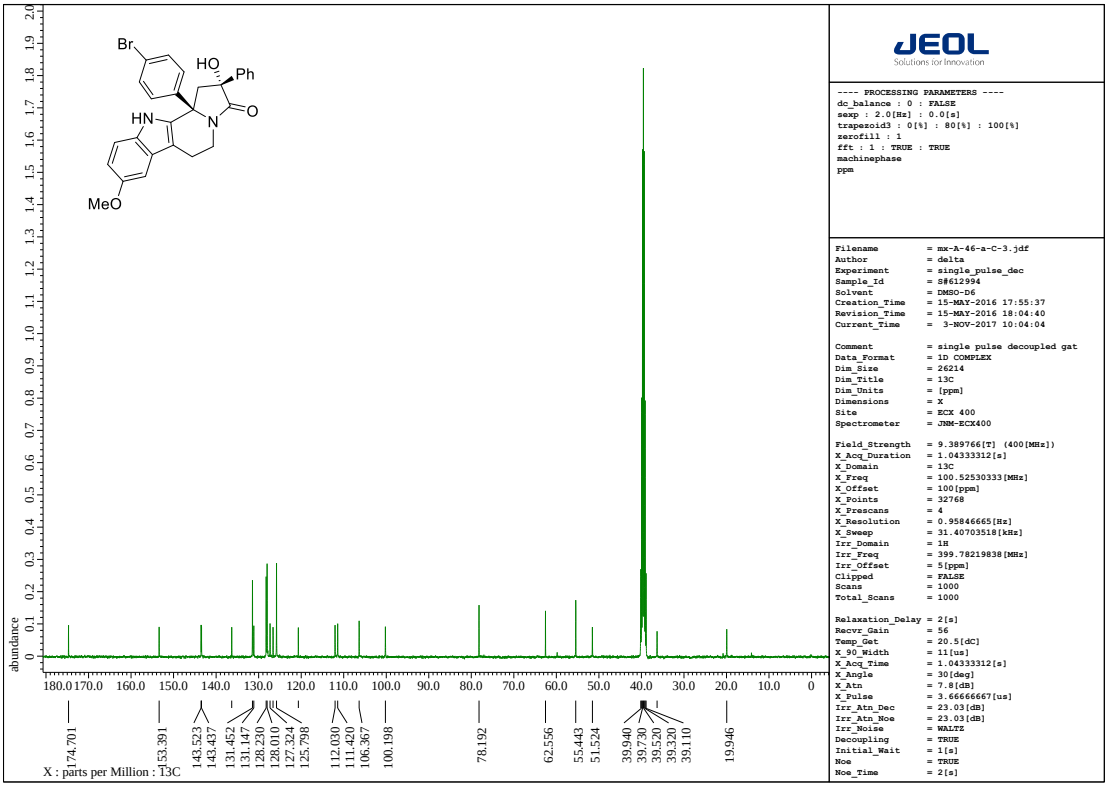


---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

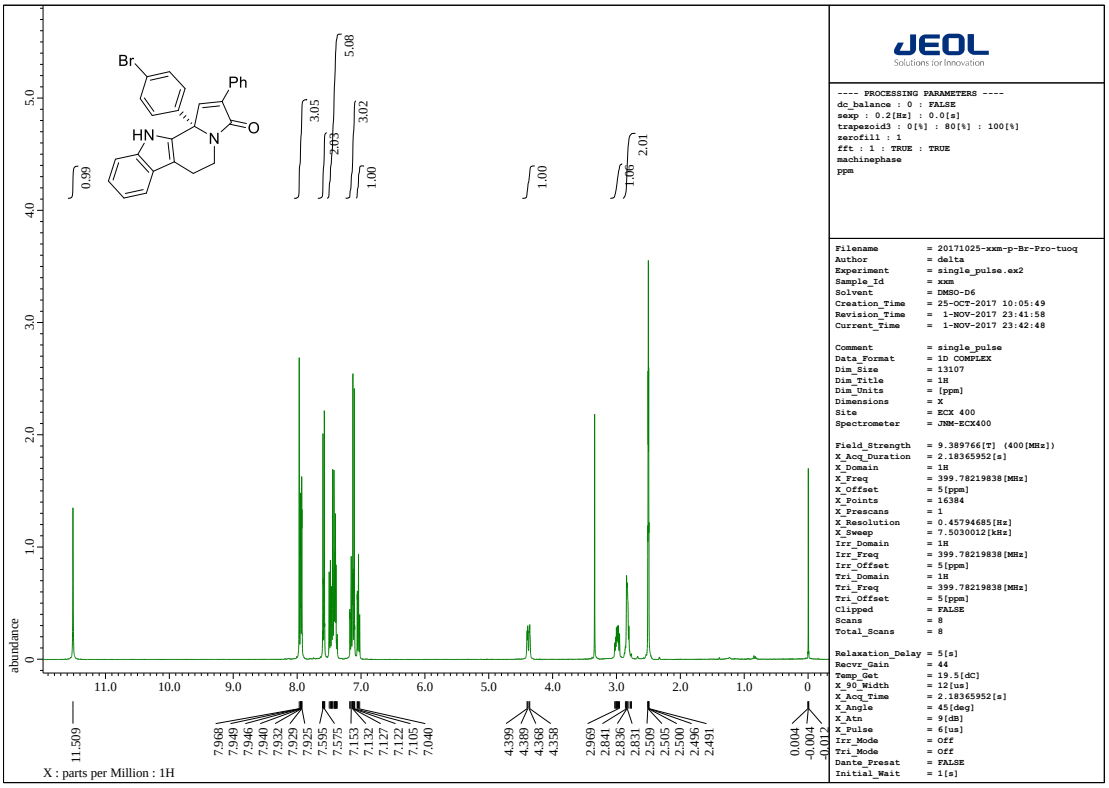
Filename	= mx-A-47-b-C-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#826801
Solvent	= CHLOROFORM-D
Creation_Time	= 16-MAY-2016 07:28:28
Revision_Time	= 18-MAY-2016 13:55:12
Current_Time	= 3-NOV-2017 10:12:38
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 10000
Total_Scans	= 10000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 58
Temp_Get	= 20.9[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-46-a-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#611798
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 17:02:18
Revision_Time     =  3-NOV-2017 10:03:17
Current_Time      =  3-NOV-2017 10:03:20

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 20.8[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename          = mx-A-46-a-C-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#612994
Solvent           = DMSO-D6
Creation_Time     = 15-MAY-2016 17:55:37
Revision_Time     = 15-MAY-2016 18:04:40
Current_Time      =  3-NOV-2017 10:04:04

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 56
Temp_Get          = 20.5[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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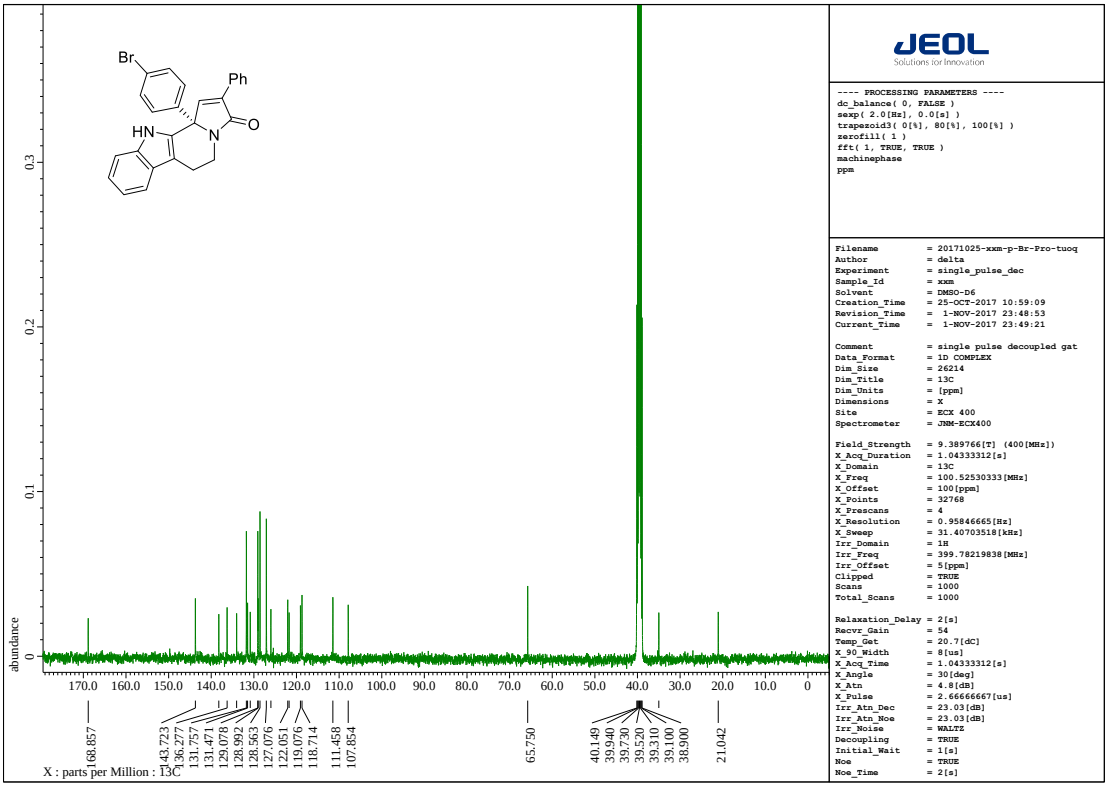
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 20171025-xxm-p-Br-Pro-tuoq
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = xxm
Solvent           = DMSO-D6
Creation_Time     = 25-OCT-2017 10:05:49
Revision_Time     =  1-NOV-2017 23:41:58
Current_Time      =  1-NOV-2017 23:42:48

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 19.5[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 9[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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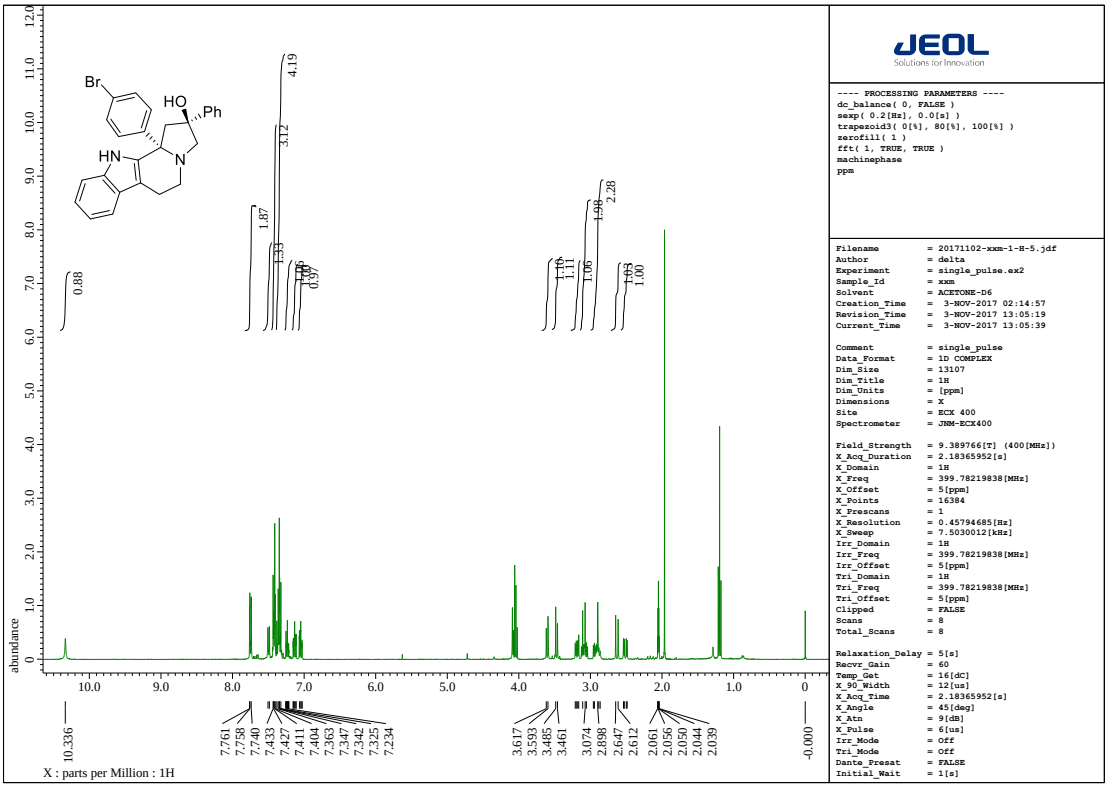
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 20171025-xxm-p-Br-Pro-tuoq
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = xxm
Solvent           = DMSO-D6
Creation_Time     = 25-OCT-2017 10:59:09
Revision_Time     =  1-NOV-2017 23:48:53
Current_Time      =  1-NOV-2017 23:49:21

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 54
Temp_Get          = 20.7[dC]
X_90_Width        = 8[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.8[dB]
X_Pulse           = 2.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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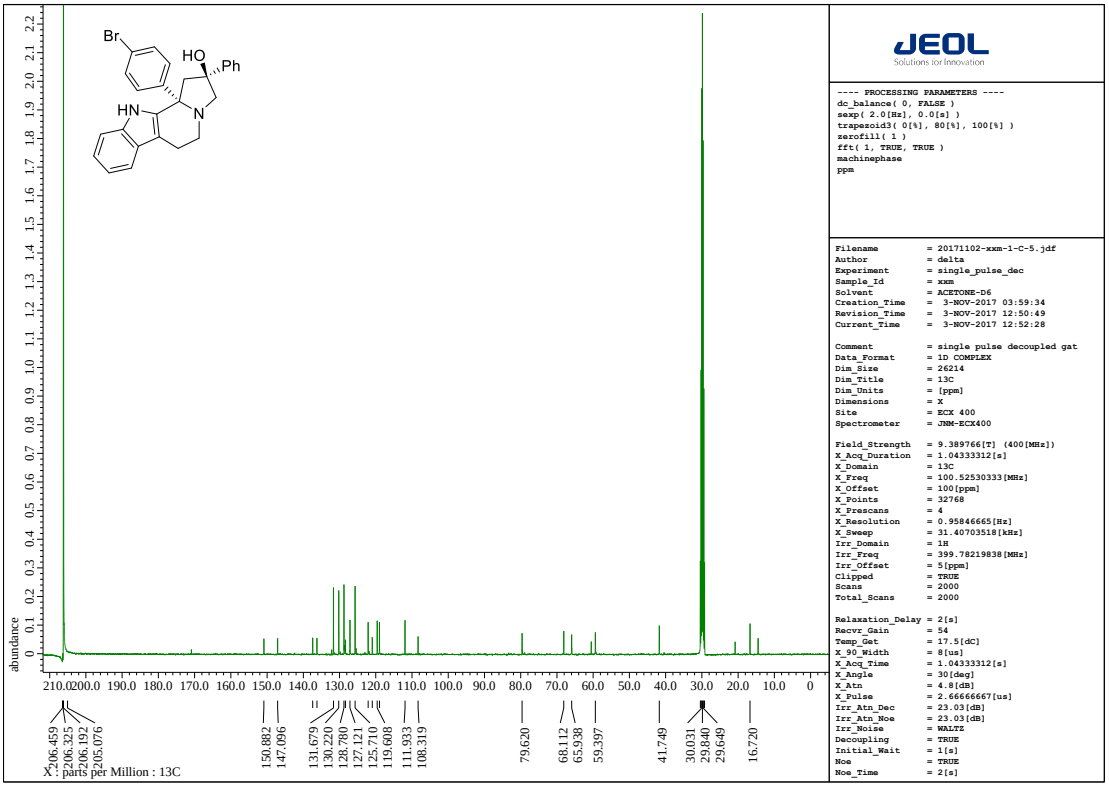
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 20171102-xxm-1-H-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = xxm
Solvent           = ACETONE-D6
Creation_Time     =  3-NOV-2017 02:14:57
Revision_Time     =  3-NOV-2017 13:05:19
Current_Time      =  3-NOV-2017 13:05:39

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 60
Temp_Get          = 16[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 9[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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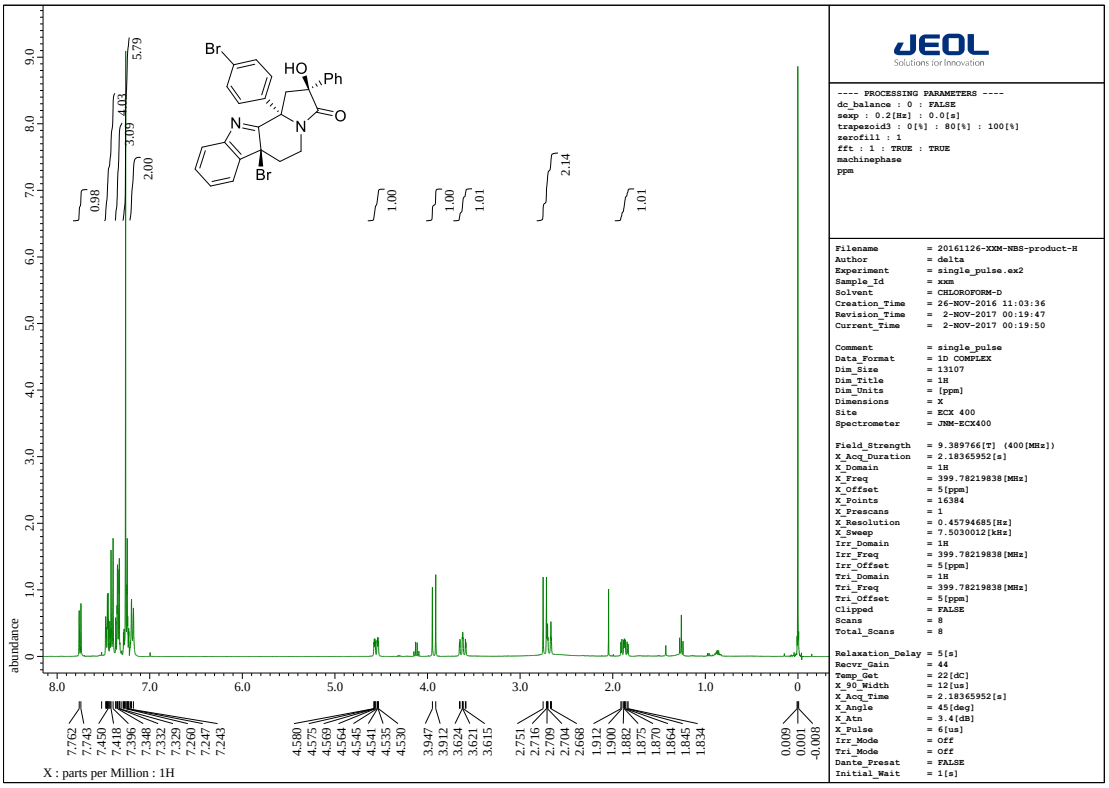
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 20171102-xxm-1-C-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = xxm
Solvent           = ACETONE-D6
Creation_Time     =  3-NOV-2017 03:59:34
Revision_Time     =  3-NOV-2017 12:50:49
Current_Time      =  3-NOV-2017 12:52:28

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 2000
Total_Scans       = 2000

Relaxation_Delay = 2[s]
Recvr_Gain        = 54
Temp_Get          = 17.5[dC]
X_90_Width        = 8[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.8[dB]
X_Pulse           = 2.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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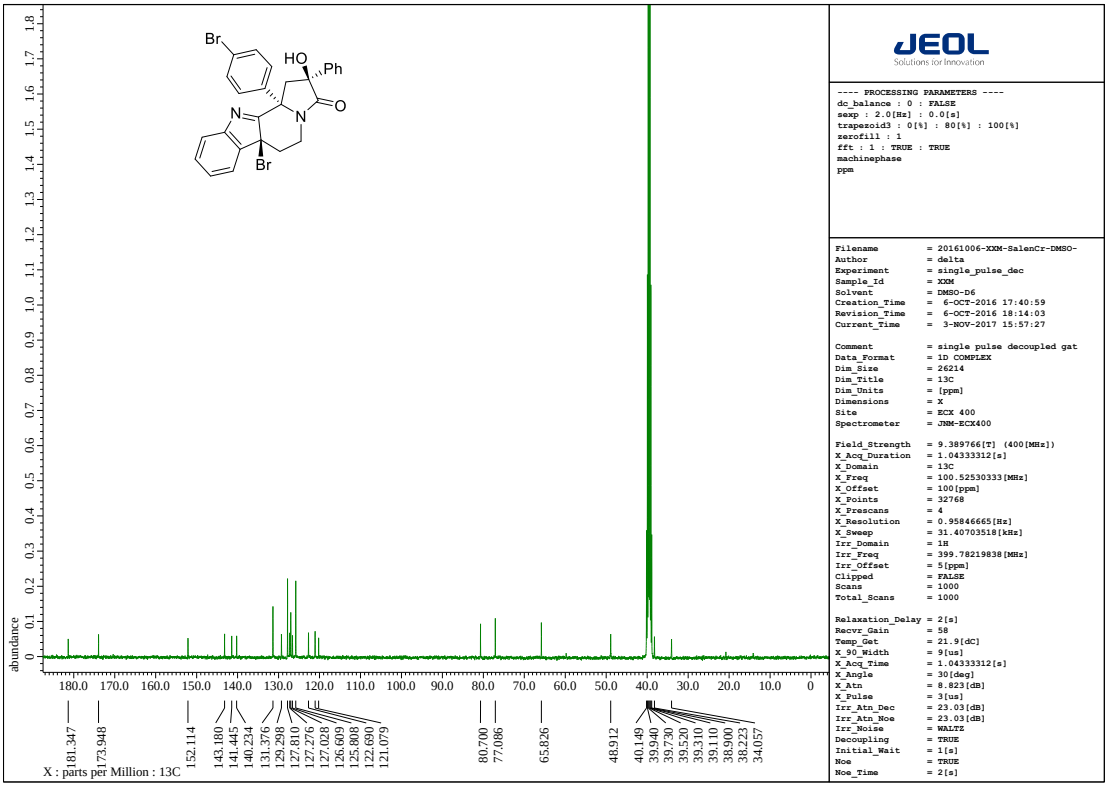
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 20161126-XXM-NBS-product-H
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = xxm
Solvent           = CHLOROFORM-D
Creation_Time     = 26-NOV-2016 11:03:36
Revision_Time     =  2-NOV-2017 00:19:47
Current_Time      =  2-NOV-2017 00:19:50

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 22[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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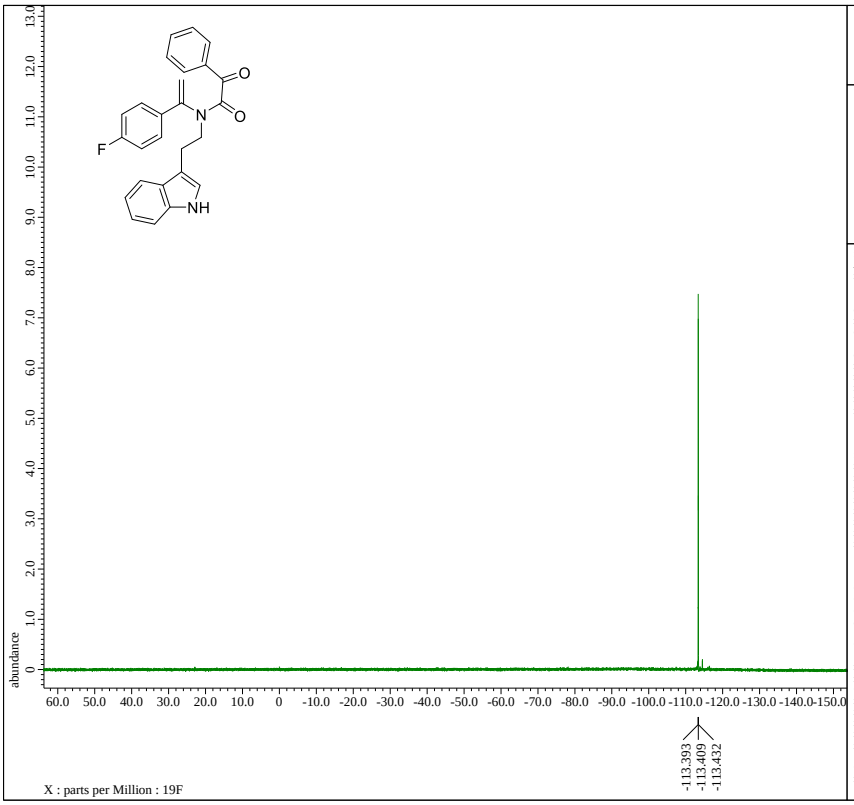
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 20161006-XXM-SalenCr-DMSO-
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = XXM
Solvent           = DMSO-D6
Creation_Time     =  6-OCT-2016 17:40:59
Revision_Time     =  6-OCT-2016 18:14:03
Current_Time      =  3-NOV-2017 15:57:27

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 21.9[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 8.823[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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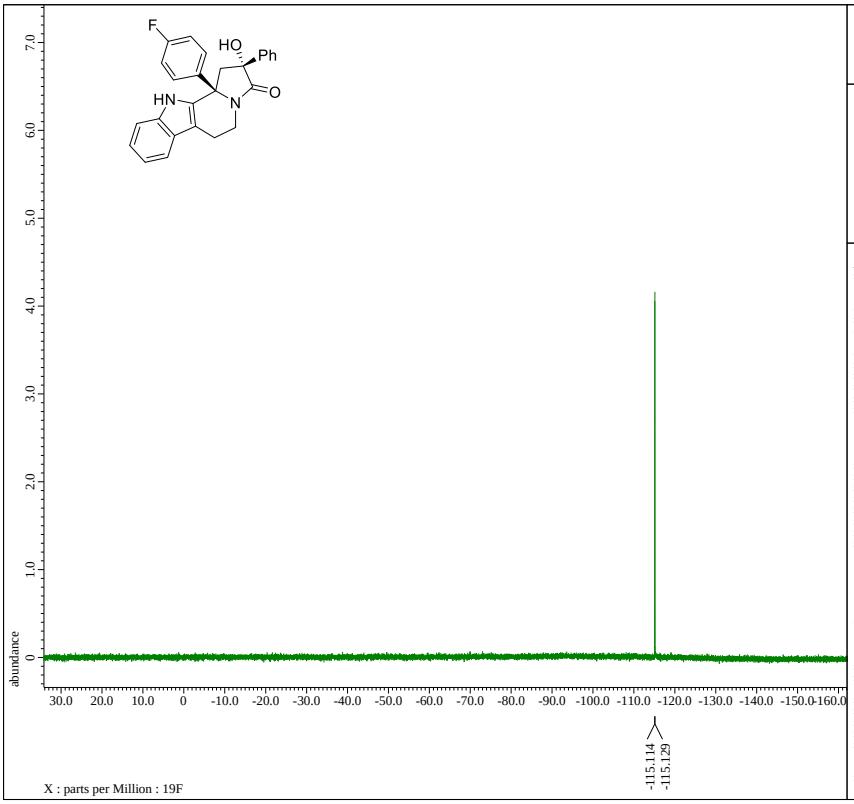
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = single_pulse-154.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#678396
Solvent           = ACETONE-D6
Creation_Time     =  4-NOV-2017 18:38:15
Revision_Time     =  4-NOV-2017 19:50:39
Current_Time      =  4-NOV-2017 19:50:56

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 52428
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.34603008[s]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 65536
X_Prescans        = 1
X_Resolution      = 2.88992217[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 16[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.34603008[s]
X_Angle           = 45[deg]
X_Atn             = 9[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 20171104-xxm-2-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#682242
Solvent           = DMSO-D6
Creation_Time     =  4-NOV-2017 18:43:45
Revision_Time     =  4-NOV-2017 19:44:15
Current_Time      =  4-NOV-2017 19:44:26

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 52428
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.34603008[s]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 65536
X_Prescans        = 1
X_Resolution      = 2.88992217[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 52
Temp_Get          = 15.8[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.34603008[s]
X_Angle           = 45[deg]
X_Atn             = 9[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```