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

A metal-free hydrogenation of 3-substituted 2H-1,4-benzoxazines

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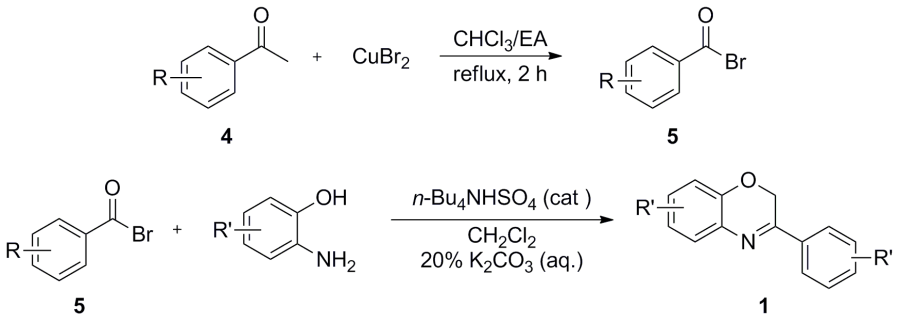
Supporting Information

General consideration: All air-sensitive compounds were handled under an atmosphere of argon or in a nitrogen-filled glovebox. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker AV 400 at ambient temperature with $CDCl_3$ as solvent and TMS as internal standard. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of TMS (0), to the carbon resonance of the $CDCl_3$ (77.23). Coupling constants (*J*) were given in Hertz (Hz). IR spectrums were recorded on Perkin-Elmer-983 spectrometer. Optical rotations were measured with PerkinElmer 341 polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh). All solvents were purified by conventional methods, distilled before use. Commercially available reagents were used without further purification.



Representative procedure for the synthesis of 1,4-benzoxazines 1: To a round-bottomed bottle were added ketone **4** (25 mmol), copper (II) bromide (11.17 g, 50 mmol) and $CHCl_3$/EA = 1/1 (v/v) (25 mL), and the resulting mixture was refluxed for 2 h. The reaction was cooled to room temperature, then the solid was filtered and washed there times by EA. Removal of the solvents *in vacuo*, the residue was used directly without further purification. To a round-bottomed bottle contained 2-aminophenol (10 mmol) and CH_2Cl_2 (40 mL), 20% aqueous K_2CO_3 solution (70 mL) and *n*-Bu_4NHSO_4 (0.0030 g) were added. The solution of compound **5** (10 mmol) in CH_2Cl_2 (30 mL) was added dropwise to the reaction mixture, and the reaction mixture was stirred at room temperature and monitored by TLC. After the

consumption of starting material, organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (10 mL x 2). The combined organics were washed with water and brine, and then dried over anhydrous Na_2SO_4. Removal of the solvents *in vacuo*, the residue was purified by flash column chromatography to obtain the corresponding benzoxazines.

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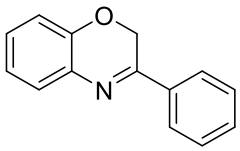
General procedure for the metal-free catalytic hydrogenation (Scheme 2): To a glass test tube (10 mL) were added $B(C_6F_5)_3$ (0.0051 g, 0.010 mmol), 1,4-benzoxazines **1** (0.40 mmol) and dry toluene (0.2 mL). The tube was then moved to a stainless-steel autoclave. After being sealed, the autoclave was purged three times with H_2 and the final pressure of hydrogen was adjusted to 20 bar. The reaction mixture was stirred at 50 °C for 6 h. The reaction mixture was cooled to room temperature, and the solvent was removed under reduced pressure. The crude residue was purified by flash chromatography on silica gel using PE/EA (10/1) as the eluent t to give the desired product **2**.

General procedure for the asymmetric hydrogenation (Scheme 4): To a glass test tube (10 mL) were added $HB(C_6F_5)_2$ (0.0052 g, 0.015 mmol), chiral diene **3i** (0.0091 g, 0.0075 mmol) and dry CH_2Cl_2 (0.6 mL). The resulting mixture was stirred for 5 min at room temperature, and 1,4-benzoxazines **1** (0.30 mmol) was added. The tube was then moved to a stainless-steel autoclave. After being sealed, the autoclave was purged three times with H_2 and the final pressure of hydrogen was adjusted to 20 bar. The reaction mixture was stirred at room temperature for 12 h. The solvent was removed under reduced pressure, and the crude residue was purified by flash chromatography on silica gel using PE/EA (10/1) as the eluent t to give the desired product **2**.



1a: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.96-7.88 (m, 2H), 7.56-7.40 (m, 4H), 7.18-7.11 (m, 1H), 7.05-6.99 (m, 1H), 6.94-6.89 (m, 1H), 5.06 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.9, 146.6, 135.7, 134.0, 131.4, 129.0, 128.9, 128.1, 126.7, 122.6, 115.8, 63.1.

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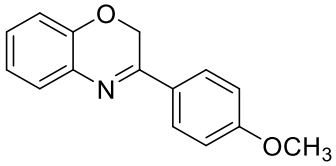

1b: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.90 (d, J = 8.8 Hz, 2H), 7.43-7.38 (m, 1H), 7.15-7.09 (m, 1H), 7.05-7.01 (m, 1H), 6.98 (d, J = 8.8 Hz, 2H), 6.94-6.89 (m, 1H), 5.03 (s, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 162.3, 158.3, 146.5, 134.2, 128.4, 128.33, 128.28, 127.7, 122.5, 115.7, 114.3, 62.9, 55.6.

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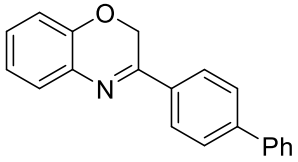

1c: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.01 (d, J = 8.4 Hz, 2H), 7.72 (d, J = 8.4 Hz, 2H), 7.68-7.63 (m, 2H), 7.52-7.44 (m, 3H), 7.43-7.34 (m, 1H), 7.20-7.13 (m, 1H), 7.09-7.01 (m, 1H), 6.97-6.91 (m, 1H), 5.11 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.4, 146.6, 144.0, 140.3, 134.5, 134.1, 129.1, 128.8, 128.2, 128.0, 127.6, 127.4, 127.2,

122.6, 115.8, 63.1.

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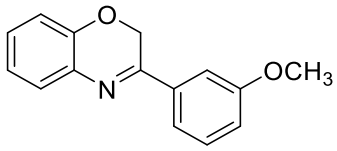
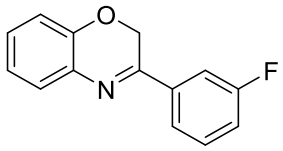


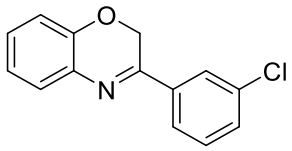
1d: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.60-7.52 (m, 1H), 7.50-7.32 (m, 3H), 7.19-7.12 (m, 1H), 7.08-7.01 (m, 2H), 6.95-6.90 (m, 1H), 5.05 (s, 2H), 3.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 160.2, 158.6, 146.6, 137.0, 133.9, 129.9, 128.9, 128.0, 122.5, 119.1, 117.7, 115.7, 111.4, 63.2, 55.6.

J. Qin, F. Chen, Y.-M. He and Q.-H. Fan, *Org. Chem. Front.,* 2014, **1**, 952.



1e: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.75-7.67 (m, 1H), 7.66-7.58 (m, 1H), 7.51-7.36 (m, 2H), 7.24-7.12 (m, 2H), 7.08-7.00 (m, 1H), 6.95-6.89 (m, 1H), 5.04 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 163.3 (d, J = 245.3 Hz), 157.4, 146.5, 137.9 (d, J = 7.3 Hz), 133.7, 130.5 (d, J = 8.0 Hz), 129.3, 128.2, 122.7, 122.2 (d, J = 2.8 Hz), 118.3 (d, J = 21.4 Hz), 115.8, 113.6 (d, J = 22.7 Hz), 63.0.

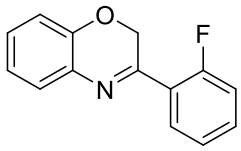
K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



1f: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.98-7.91 (m, 1H), 7.76-7.68 (m, 1H), 7.49-7.34 (m, 3H), 7.20-7.13 (m, 1H), 7.06-6.99 (m, 1H), 6.95-6.88 (m, 1H), 5.01 (s, 2H); ^{13}C

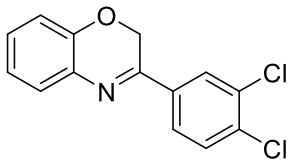
NMR (100 MHz, $CDCl_3$, ppm) δ 157.3, 146.5, 137.3, 135.3, 133.7, 131.2, 130.2, 129.3, 128.2, 126.8, 124.6, 122.7, 115.8, 62.9.

J. Qin, F. Chen, Y.-M. He and Q.-H. Fan, *Org. Chem. Front.,* 2014, **1**, 952.



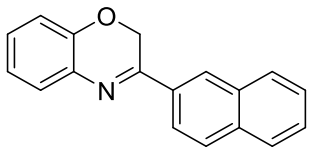
1g: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.06-7.98 (m, 1H), 7.50-7.40 (m, 2H), 7.30-7.21 (m, 1H), 7.20-7.07 (m, 2H), 7.06-6.99 (m, 1H), 6.95-6.89 (m, 1H), 4.98 (d, *J* = 2.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 161.9 (d, *J* = 249.3 Hz), 157.6, 132.8 (d, *J* = 8.6 Hz), 129.8 (d, *J* = 3.6 Hz), 129.2, 128.0, 124.9 (d, *J* = 3.0 Hz), 124.5 (d, *J* = 12.7 Hz), 122.6, 116.3 (d, *J* = 22.3 Hz), 115.9, 65.0 (d, *J* = 13.5 Hz).

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



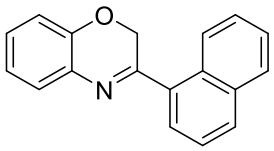
1h: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.08-8.01 (m, 1H), 7.75-7.65 (m, 1H), 7.56-7.49 (m, 1H), 7.45-7.37 (m, 1H), 7.22-7.13 (m, 1H), 7.08-6.99 (m, 1H), 6.95-6.88 (m, 1H), 4.99 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.2, 146.3, 135.5, 135.4, 133.6, 133.5, 130.9, 129.5, 128.5, 128.2, 125.6, 122.7, 115.8, 62.6.

J. Qin, F. Chen, Y.-M. He and Q.-H. Fan, *Org. Chem. Front.,* 2014, **1**, 952.



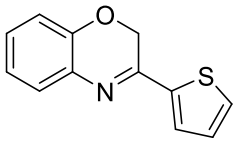
1i: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.28-8.22 (m, 1H), 8.20-8.17 (m, 1H), 7.97-7.85 (m, 3H), 7.60-7.46 (m, 3H), 7.22-7.15 (m, 1H), 7.10-7.03 (m, 1H), 6.99-6.94 (m, 1H), 5.19 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.5, 146.6, 134.9, 134.1, 133.1, 129.1, 128.9, 128.8, 128.1, 128.0, 127.8, 126.95, 126.86, 123.5, 122.6, 115.8, 63.0.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



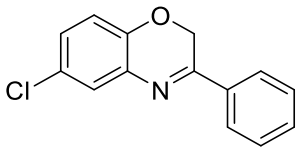
1j: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.50-8.41 (m, 1H), 7.99-7.89 (m, 2H), 7.66-7.49 (m, 5H), 7.25-7.19 (m, 1H), 7.11-7.05 (m, 1H), 7.04-6.97 (m, 1H), 5.02 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 162.1, 146.6, 134.7, 134.22, 134.20, 130.9, 130.7, 129.2, 128.8, 128.1, 127.4, 126.6, 126.3, 125.6, 125.2, 122.7, 116.0, 65.8.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



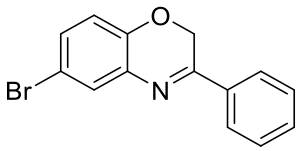
1k: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.57-7.51 (m, 1H), 7.44-7.36 (m, 2H), 7.09-7.17 (m, 2H), 7.05-6.97 (m, 1H), 6.95-6.88 (m, 1H), 5.00 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 153.9, 146.7, 141.6, 134.0, 131.0, 128.5, 128.2, 128.1, 127.7, 122.7, 115.8, 63.1.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



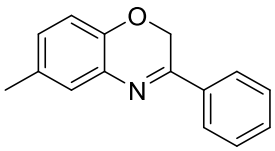
1l: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.91 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.55-7.45 (m, 3H), 7.42 (d, *J* = 2.4 Hz, 1H), 7.10 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 1H), 5.07 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 159.9, 145.2, 135.2, 134.8, 131.8, 129.0, 128.4, 127.7, 127.1, 126.8, 117.2, 116.8, 63.1.

Z.-P. Chen, M.-W. Chen, R.-N. Guo and Y.-G. Zhou, *Org. Lett.,* 2014, **16**, 1406.



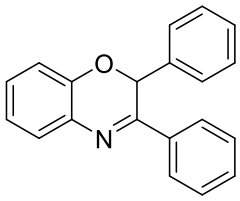
1m: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.89-7.86 (m, 2H), 7.56-7.53 (m 1H), 7.52-7.41 (m, 3H), 7.24-7.18 (m, 1H), 6.80-6.79 (m, 1H), 5.02 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 159.8, 145.6, 135.11, 135.09, 131.7, 131.2, 130.5, 129.0, 126.7, 114.1, 63.0.

Z. Zhang, Y. R. Ji, L. Wojtas, W.-Y. Gao, S. Ma, M. J. Zaworotko and J. C. Antilla, *Chem. Commun.,* 2013, **49**, 7693.



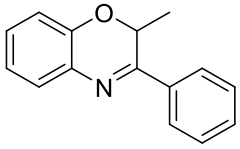
1n: Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.94-7.88 (m, 2H), 7.50-7.43 (m, 3H), 7.27-7.24 (m, 1H), 6.97-6.92 (m, 1H), 6.83-6.79 (m, 1H), 5.02 (s, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.9, 144.3, 135.7, 133.7, 132.0, 131.3, 129.3, 128.9, 128.3, 126.6, 115.3, 63.1, 20.9.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



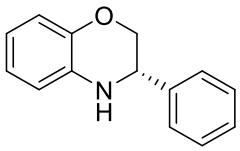
1o: White solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.98-7.83 (m, 2H), 7.50-7.44 (m, 1H), 7.45-7.38 (m, 3H), 7.38-7.31 (m, 2H), 7.30-7.22 (m, 3H), 7.11-7.04 (m, 1H), 7.01-6.95 (m, 1H), 6.85-6.80 (m, 1H), 6.34 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 159.3, 144.7, 136.2, 136.0, 134.1, 131.1, 129.3, 129.1, 129.0, 128.9, 128.3, 127.8, 127.2, 122.4, 116.8, 73.3.

P. G. Baraldi, G. Saponaro, A. R. Moorman, R. Romagnoli, D. Preti, S. Baraldi, E. Ruggiero, K. Varani, M. Targa, F. Vincenzi, P. A. Borea and M. Aghazadeh Tabrizi, *J. Med. Chem.,* 2012, **55**, 6608.



1p: White solid; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.01-7.94 (m, 2H), 7.51-7.42 (m, 4H), 7.20-7.14 (m, 1H), 7.06-7.00 (m, 1H), 6.96-6.91 (m, 1H), 5.53 (q, J = 6.8 Hz, 1H), 1.41 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 161.8, 144.6, 135.4, 133.5, 131.2, 129.0, 128.9, 127.7, 122.3, 116.7, 68.0, 17.1.

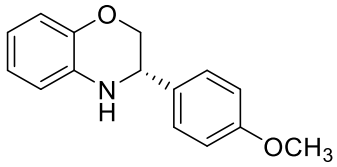
P. G. Baraldi, G. Saponaro, A. R. Moorman, R. Romagnoli, D. Preti, S. Baraldi, E. Ruggiero, K. Varani, M. Targa, F. Vincenzi, P. A. Borea and M. Aghazadeh Tabrizi, *J. Med. Chem.,* 2012, **55**, 6608.



2a: Yellow oil; 95% yield; 33% ee; $[\alpha]_D^{23}$ = +37.2 (*c* 0.60, $CHCl_3$), [lit.: $[\alpha]_D^{20}$ = -118.1 (*c* 1.0,

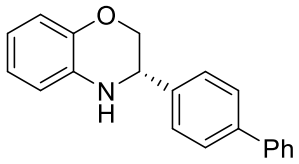
$CHCl_3$) (98% ee for *R*-isomer)]; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.44-7.30 (m, 5H), 6.88-6.78 (m, 2H), 6.75-6.65 (m, 2H), 4.51 (dd, *J* = 8.4, 2.8 Hz, 1H), 4.32-4.25 (m, 1H), 4.04-3.96 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.7, 139.3, 134.1, 129.0, 128.5, 127.4, 121.6, 119.1, 116.8, 115.5, 71.1, 54.3.

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



2b: White solid; 99% yield; 40% ee, $[\alpha]_D^{23}$ = +40.1 (*c* 0.63, $CHCl_3$), [lit.: $[\alpha]_D^{20}$ = -126.5 (*c* 1.0, $CHCl_3$) (98% ee for *R*-isomer)]; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.37-7.31 (m, 2H), 6.99-6.93 (m, 2H), 6.91-6.87 (m, 1H), 6.87-6.81 (m, 1H), 6.77-6.71 (m, 1H), 6.71-6.65 (m, 1H), 4.46 (dd, *J* = 8.8, 2.4 Hz, 1H), 4.27 (dd, *J* = 10.8, 6.4 Hz, 1H), 4.04-3.94 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 159.7, 143.6, 134.2, 131.3, 128.5, 121.6, 119.0, 116.7, 115.5, 114.3, 71.2, 55.4, 53.7.

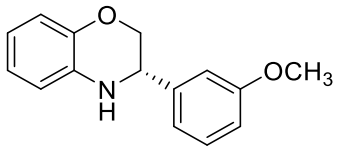
J. Qin, F. Chen, Y.-M. He and Q.-H. Fan, *Org. Chem. Front.,* 2014, **1**, 952.



2c: White solid; 90% yield; 42% ee, $[\alpha]_D^{23}$ = +27.2 (*c* 0.60, $CHCl_3$), [lit.: $[\alpha]_D^{20}$ = +44.8 (*c* 1.04, $CHCl_3$) (86% ee for *S*-isomer)]; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.68-7.60 (m, 4H), 7.55-7.46 (m, 4H), 7.44-7.37 (m, 1H) 6.95-6.90 (m, 1H), 7.90-7.84 (m, 1H), 6.81-6.70 (m, 2H), 4.57 (dd, *J* = 8.4, 2.6 Hz, 1H), 4.30 (dd, *J* = 10.6, 2.6 Hz, 1H), 4.11-4.03 (m, 2H); ^{13}C

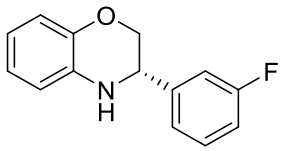
NMR (100 MHz, $CDCl_3$, ppm) δ 143.7, 141.5, 140.7, 138.3, 134.0, 129.0, 127.8, 127.7, 127.6, 127.3, 121.7, 119.1, 116.8, 115.6, 71.1, 54.1.

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



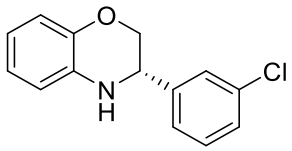
2d: Yellow oil; 92% yield; 33% ee, $[\alpha]_D^{23}$ = +21.5 (*c* 0.63, $CHCl_3$), [lit.: $[\alpha]_D^{20}$ = +84.6 (*c* 0.87, $CHCl_3$) (87% ee for *S*-isomer)]; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.35-7.28 (m, 1H), 7.02-6.95 (m, 2H), 6.92-6.75 (m, 3H), 6.76-6.65 (m, 2H), 4.49 (dd, *J* = 8.8, 2.8 Hz, 1H), 4.30 (dd, *J* = 10.4, 2.8 Hz, 1H), 4.06-3.96 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 160.14, 143.6, 140.9, 134.0, 129.9, 121.6, 119.6, 119.0, 116.7, 115.5, 113.8, 112.8, 71.1, 55.4, 54.3.

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



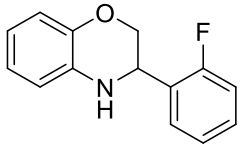
2e: Colorless oil; 92% yield; 31% ee, $[\alpha]_D^{23}$ = +30.2 (*c* 0.60, $CHCl_3$), [lit.: $[\alpha]_D^{20}$ = +58.7 (*c* 1.12, $CHCl_3$) (85% ee for *S*-isomer)]; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.42-7.34 (m, 1H), 7.24-7.18 (m, 1H), 7.18-7.13 (m, 1H), 7.11-7.03 (m, 1H), 7.93-7.83 (m, 2H), 6.79-7.69 (m, 2H), 4.51 (d, *J* = 8.0 Hz, 1H), 4.30 (d, *J* = 10.6 Hz, 1H), 4.05-3.95 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 163.3 (d, *J* = 245.0 Hz), 143.6, 142.1 (d, *J* = 6.9 Hz), 133.7, 130.5 (d, *J* = 8.1 Hz), 122.9 (d, *J* = 2.8 Hz), 121.8, 119.3, 116.8, 115.6, 115.3 (d, *J* = 21.1 Hz), 114.2 (d, *J* = 22.0 Hz), 70.8, 53.8 (d, *J* = 1.3 Hz).

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



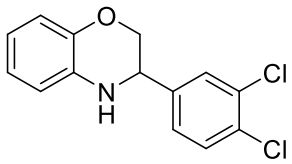
2f: Yellow oil; 97% yield; 30% ee, $[\alpha]_D^{23}$ = +26.2 (*c* 0.61, $CHCl_3$), [lit.: $[\alpha]_D^{20}$ = +71.0 (*c* 1.38, $CHCl_3$) (88% ee for *S*-isomer)]; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.42 (s, 1H), 7.35-7.25 (m, 3H), 6.92-6.80 (m, 2H), 6.79-6.67 (m, 2H), 4.48 (dd, *J* = 8.2, 1.6 Hz, 1H), 4.31-4.25 (m, 1H), 4.03-3.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.6, 141.5, 134.9, 133.6, 130.2, 128.6, 127.5, 125.5, 121.8, 119.3, 116.8, 115.7, 70.8, 53.9.

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



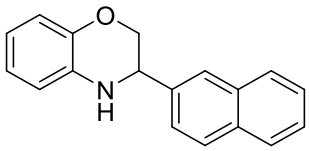
2g: Colorless oil; 98% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.52-7.45 (m, 1H), 7.73-7.26 (m, 1H), 7.20-7.13 (m, 1H), 7.12-7.04 (m, 1H), 6.89-6.79 (m, 2H), 6.75-6.67 (m, 2H), 4.95-4.89 (m, 1H), 4.36 (dd, *J* = 10.8, 2.8 Hz, 1H), 4.04 (dd, *J* = 10.6, 7.2 Hz, 1H), 3.95 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 160.5 (d, *J* = 245.3), 143.7, 133.8, 129.6 (d, *J* = 8.2 Hz), 128.3 (d, *J* = 3.9 Hz), 126.7 (d, *J* = 12.7 Hz), 124.8 (d, *J* = 3.6 Hz), 121.8. 119.2, 116.9, 115.7, 115.6 (d, *J* = 21.4 Hz), 69.2, 47.3 (d, *J* = 3.5 Hz).

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



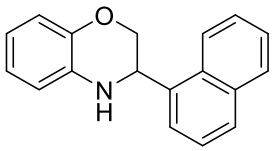
2h: Yellow oil; 96% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.53-7.49 (m, 1H), 7.48-7.43 (m, 1H), 7.27-7.21 (m, 1H), 6.87-6.79 (m, 2H), 6.76-6.67 (m, 2H), 4.52-4.45 (m, 1H), 4.29-4.21 (m, 1H), 4.02-3.90 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.5, 139.7, 133.4, 133.1, 132.3, 130.9, 129.2, 126.6, 121.9, 119.4, 116.8, 115.7, 70.5, 53.3.

K. Gao, C.-B. Yu, D.-S. Wang and Y.-G. Zhou, *Adv. Synth. Catal.,* 2012, **354**, 483.



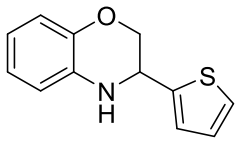
2i: White solid; 90% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.91-7.82 (m, 4H), δ 7.55-7.47 (m, 3H), 6.93-6.82 (m, 2H), 6.77-6.70 (m, 2H), 4.71-4.65 (m, 1H), 4.40-4.34 (m, 1H), 4.15-3.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.7, 136.7, 134.1, 133.5, 133.4, 128.7, 128.1, 127.9, 126.5, 126.4, 126.3, 125.1, 121.7, 119.1, 116.8, 115.6, 71.0, 54.4.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



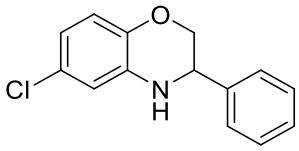
2j: White solid; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.24-8.18 (m, 1H), 8.00-7.94 (m, 1H) 7.92-7.85 (m, 1H) 7.77-7.70 (m, 1H) 7.62-7.49 (m, 3H), 6.96-6.91 (m, 1H), 6.91-6.84 (m, 1H) 6.81-6.73 (m, 2H), 5.36 (d, J = 6.8 Hz, 1H), 4.53 (d, J = 10.8 Hz, 1H), 4.13 (dd, J = 10.8, 6.8 Hz, 1H), 3.42 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.8, 134.6, 134.3, 133.9, 130.9, 129.3, 128.7, 126.7, 125.9, 125.8, 124.6, 122.4, 121.7, 119.1, 116.8, 115.8, 70.3, 50.3.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



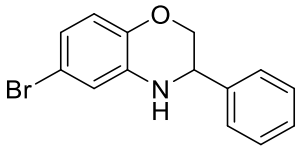
2k: Yellow oil; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.39-7.28 (m, 1H), 7.14-7.04 (m, 1H), 7.03-6.97 (m, 1H), 6.88-6.79 (m, 2H), 6.75-6.68 (m, 1H), 6.68-6.61 (m, 1H), 4.53-4.47 (m, 1H), 4.29-4.22 (m, 1H), 4.02-3.92 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.7, 142.9, 133.2, 127.1, 125.5, 125.0, 121.7, 119.5, 116.8, 115.8, 71.2, 50.3.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



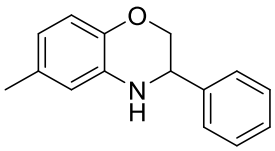
2l: Yellow oil; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.42-7.31 (m, 5H), 6.80-6.71 (m, 1H), 6.69-6.66 (m, 2H), 4.50 (dd, *J* = 8.4, 2.8 Hz, 1H), 4.32-4.24 (m, 1H), 4.08 (brs, 1H), 3.96 (dd, *J* = 11.6, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 142.2, 138.8, 135.0, 120.1, 128.6, 127.3, 126.2, 118.5, 117.6, 114.9, 70.9, 54.1.

Z.-P. Chen, M.-W. Chen, R.-N. Guo and Y.-G. Zhou, *Org. Lett.,* 2014, **16**, 1406.



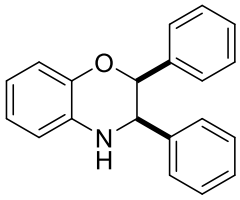
2m: Yellow oil; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.42-7.30 (m, 5H), 6.80-6.74 (m, 2H), 6.72-6.67 (m, 1H), 4.48 (dd, *J* = 8.4, 2.3 Hz, 1H), 4.30-4.23 (m, 1H), 4.06 (brs, 1H), 3.94 (dd, *J* = 10.7, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 142.7, 138.7, 135.4, 129.0, 128.6, 127.3, 121.4, 118.1, 117.7, 113.5, 70.9, 54.0.

Z. Zhang, Y. R. Ji, L. Wojtas, W.-Y. Gao, S. Ma, M. J. Zaworotko and J. C. Antilla, *Chem. Commun.,* 2013, **49**, 7693.



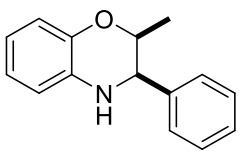
2n: Colorless oil; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.42-7.32 (m, 5H), 6.84-6.79 (m, 1H), 6.60-6.55 (m, 1H), 6.54 (s, 1H), 4.51 (dd, *J* = 8.4, 2.8 Hz, 1H), 4.30 (dd, *J* = 10.6, 2.6 Hz, 1H), 4.05-3.95 (m, 2H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 141.5, 139.5, 133.7, 131.0,128.9, 128.4, 127.3, 119.6, 116.4, 116.1, 71.1, 54.4, 20.9.

X.-W. Liu, C. Wang, Y. Yan, Y.-Q. Wang and J. Sun, *J. Org. Chem.,* 2013, **78**, 6276.



2o: White solid, 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.28-7.09 (m, 6H), 7.04-6.97 (m, 3H), 6.95-6.87 (m, 3H), 6.83-6.71 (m, 2H), 5.46 (d, *J* = 2.8 Hz, 1H), 4.74-4.67 (m, 1H), 4.29 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 144.2, 139.5, 137.8, 133.7, 128.1, 128.0, 127.9, 127.8, 127.7, 127.1, 122.1, 118.8, 117.1, 114.9, 79.4, 59.4.

P. G. Baraldi, G. Saponaro, A. R. Moorman, R. Romagnoli, D. Preti, S. Baraldi, E. Ruggiero, K. Varani, M. Targa, F. Vincenzi, P. A. Borea and M. Aghazadeh Tabrizi, *J. Med. Chem.,* 2012, **55**, 6608.

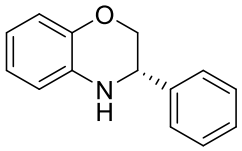


2p: Colorless liquid, 98% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.42-7.30 (m, 5H), 6.96-6.83 (m, 2H), 6.79-6.71 (m, 1H), 6.71-6.66 (m, 1H), 4.57-4.48 (m, 2H), 4.10 (brs, 1H), 1.15 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 143.0, 140.3, 133.4, 128.7,

127.9, 127.6, 121.7, 118.8, 117.1, 115.0, 73.4, 57.8, 15.7.

P. G. Baraldi, G. Saponaro, A. R. Moorman, R. Romagnoli, D. Preti, S. Baraldi, E. Ruggiero, K. Varani, M. Targa, F. Vincenzi, P. A. Borea and M. Aghazadeh Tabrizi, *J. Med. Chem.,* 2012, **55**, 6608.

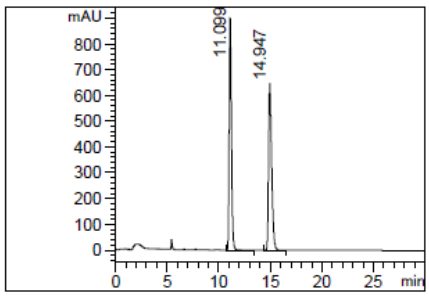
The chromatography for the determination of enantiomeric excess



2a

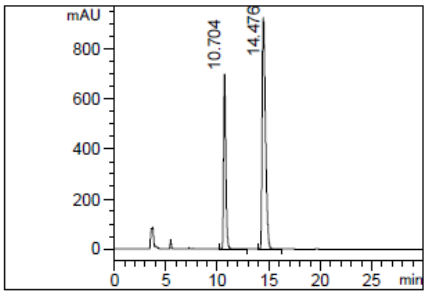
HPLC Conditions: **Column**: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 0.8 mL/min; Detection: UV 254 nm.

Racemic

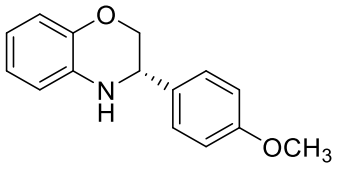


Peak #	RT [min]	Area %	Area
1	11.099	49.820	1.379e4
2	14.947	50.180	1.389e4

Chiral



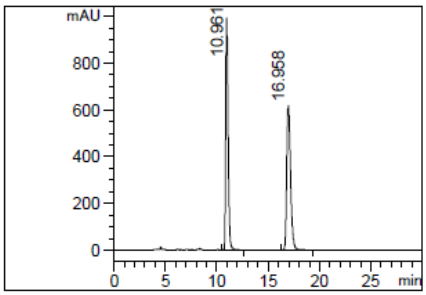
Peak #	RT [min]	Area %	Area
1	10.704	33.596	1.038e4
2	14.476	66.404	2.052e4



2b

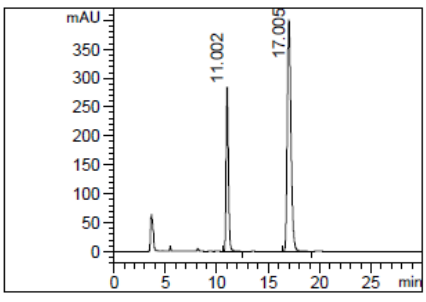
HPLC Conditions: **Column**: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 0.8 mL/min; Detection: UV 254 nm.

Racemic

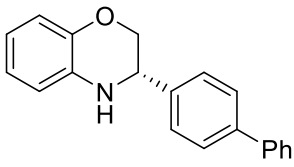


Peak #	RT [min]	Area %	Area
1	10.961	49.795	1.585e4
2	16.958	50.205	1.598e4

Chiral



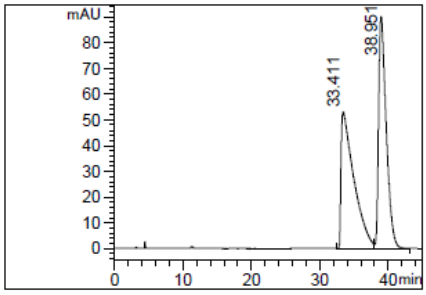
Peak #	RT [min]	Area %	Area
1	11.002	30.095	4.387e3
2	17.005	69.905	1.019e4



2c

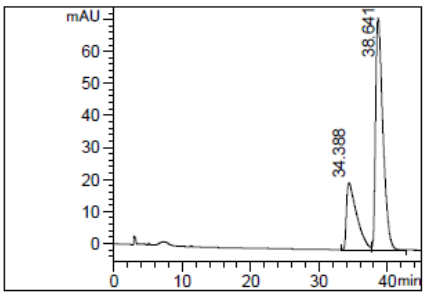
HPLC Conditions: **Column**: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent**: Hexanes/IPA (90/10); **Flow rate**: 1.0 mL/min; Detection: UV 254 nm.

Racemic

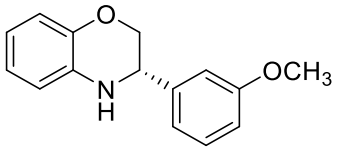


Peak #	RT [min]	Area %	Area
1	33.411	49.705	6.944e3
2	38.951	50.295	7.026e3

Chiral



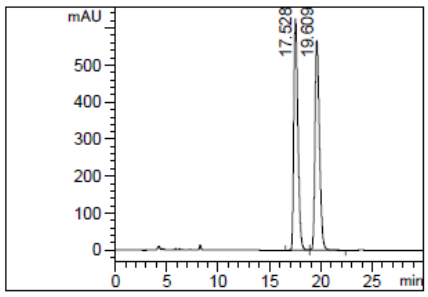
Peak #	RT [min]	Area %	Area
1	34.388	28.790	2.189e3
2	38.641	71.210	5.414e3



2d

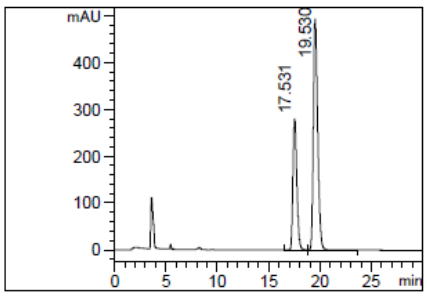
HPLC Conditions: **Column**: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 0.8 mL/min; Detection: UV 254 nm.

Racemic

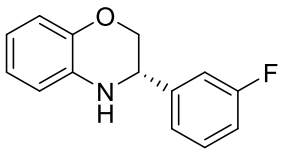


Peak #	RT [min]	Area %	Area
1	17.528	49.932	1.711e4
2	19.609	50.068	1.716e4

Chiral



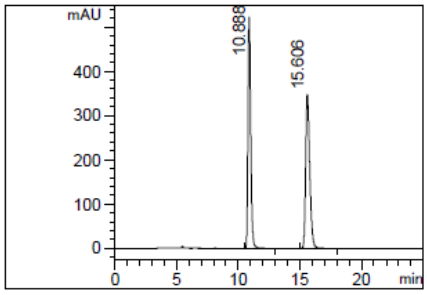
Peak #	RT [min]	Area %	Area
1	17.531	33.519	7.347e3
2	19.530	66.481	1.457e4



2e

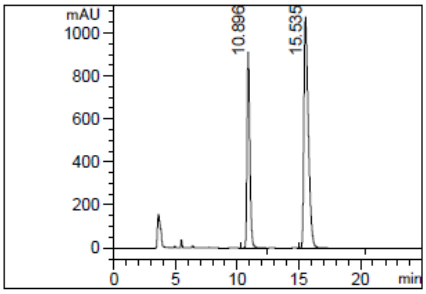
HPLC Conditions: **Column**: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 0.8 mL/min; Detection: UV 254 nm.

Racemic

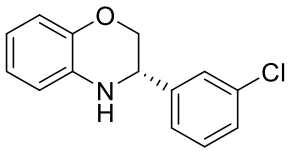


Peak #	RT [min]	Area %	Area
1	10.888	49.824	8.026e3
2	15.606	50.176	8.083e3

Chiral



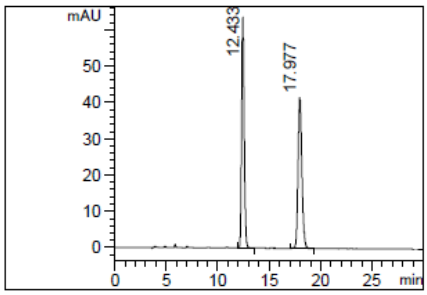
Peak #	RT [min]	Area %	Area
1	10.896	34.429	1.427e4
2	15.535	65.571	2.718e4



2f

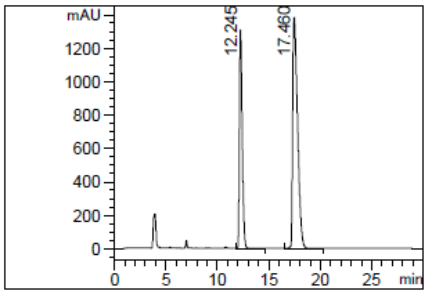
HPLC Conditions: **Column**: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 0.8 mL/min; Detection: UV 254 nm.

Racemic

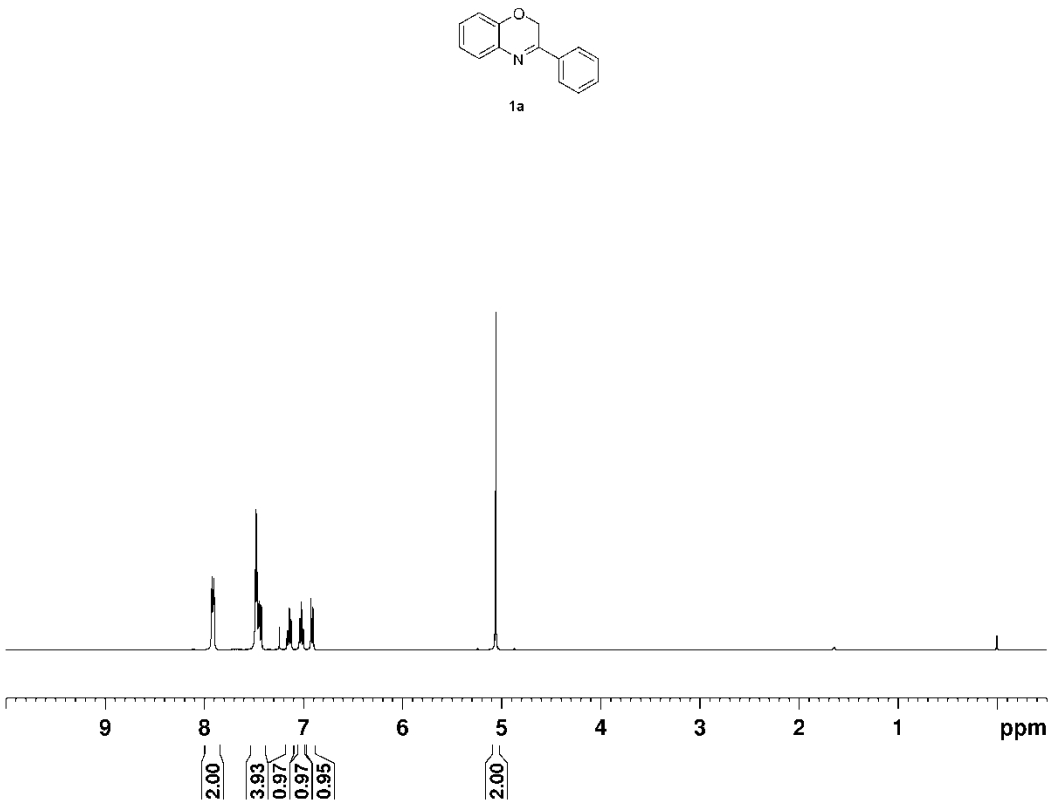


Peak #	RT [min]	Area %	Area
1	12.433	50.061	1.100e3
2	17.977	49.939	1.097e3

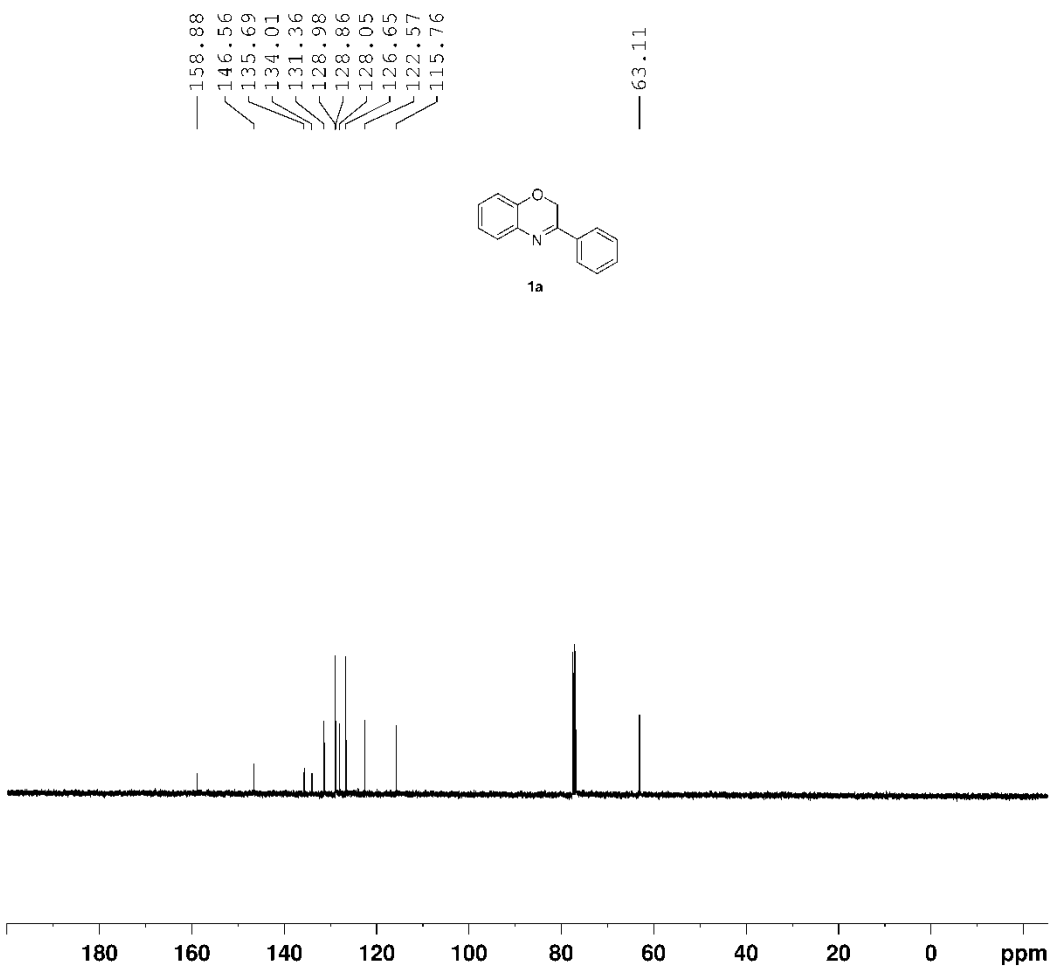
Chiral



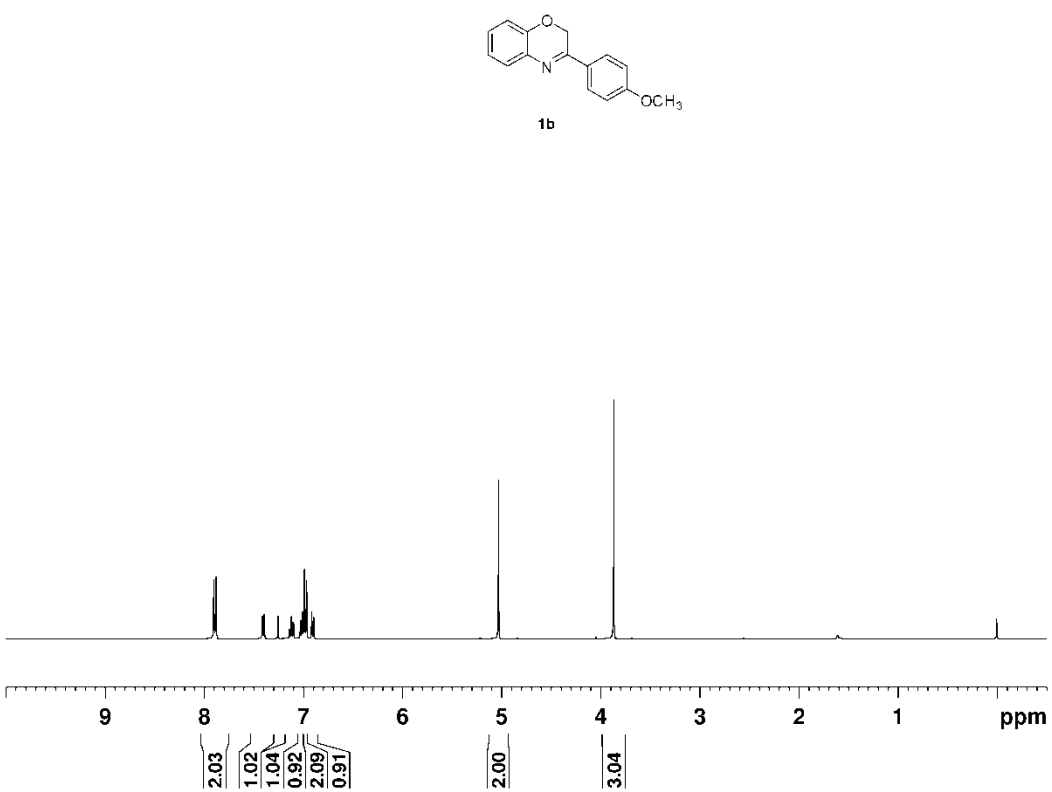
Peak #	RT [min]	Area %	Area
1	12.245	35.148	2.447e4
2	17.460	64.852	4.515e4



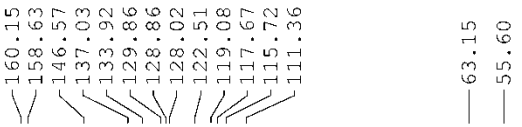
NAME	wsm-2-H-S	
EXPNO	1354	
PROCNO	2	
Date_	20160105	
Time	7.19	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300156	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	

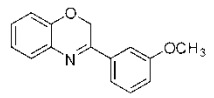


NAME	wsm-2-C-S
EXPNO	1354
PROCNO	1
Date_	20160105
Time	7.21
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	65
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	161
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127494 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



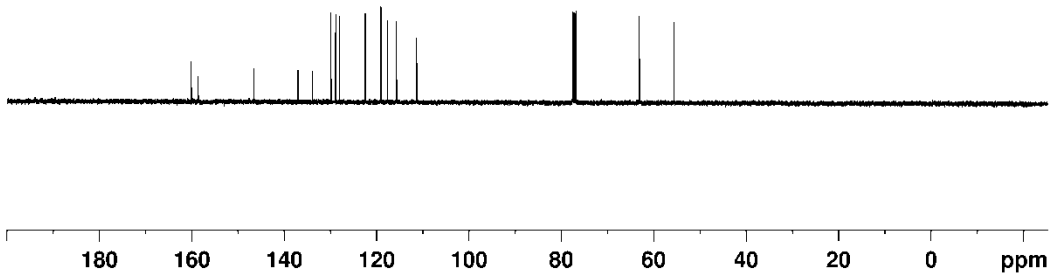
NAME	wsm-2-H-S	
EXPNO	1396	
PROCNO	1	
Date_	20151116	
Time	13.33	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	13	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300100	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	

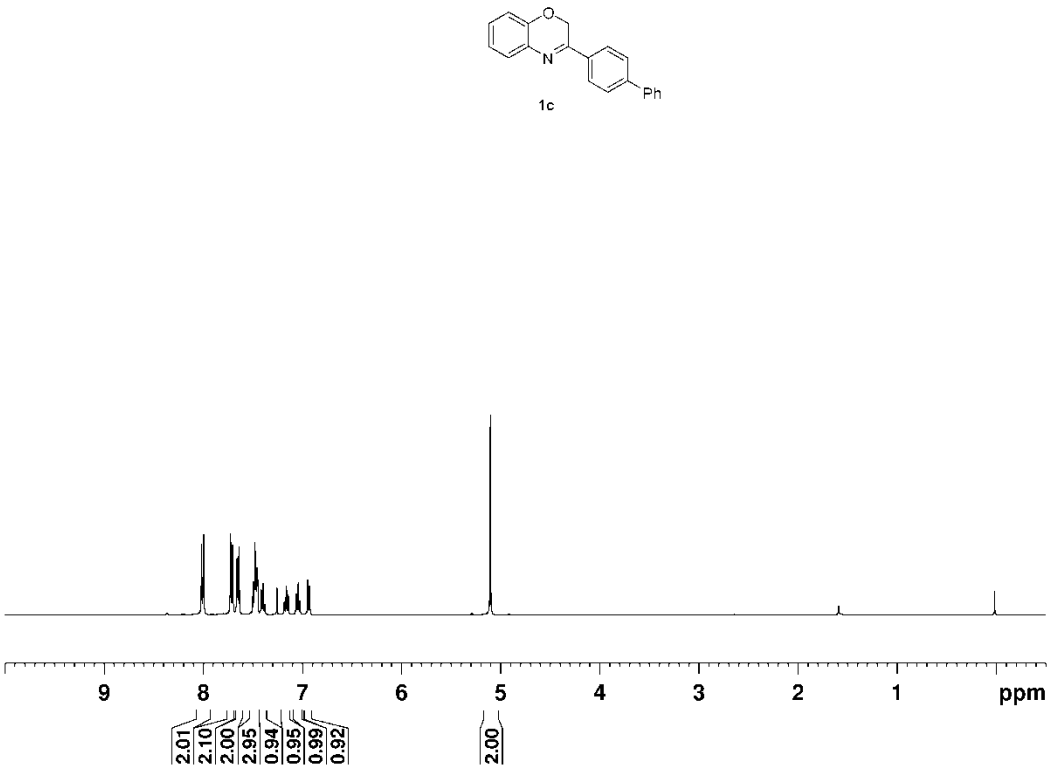




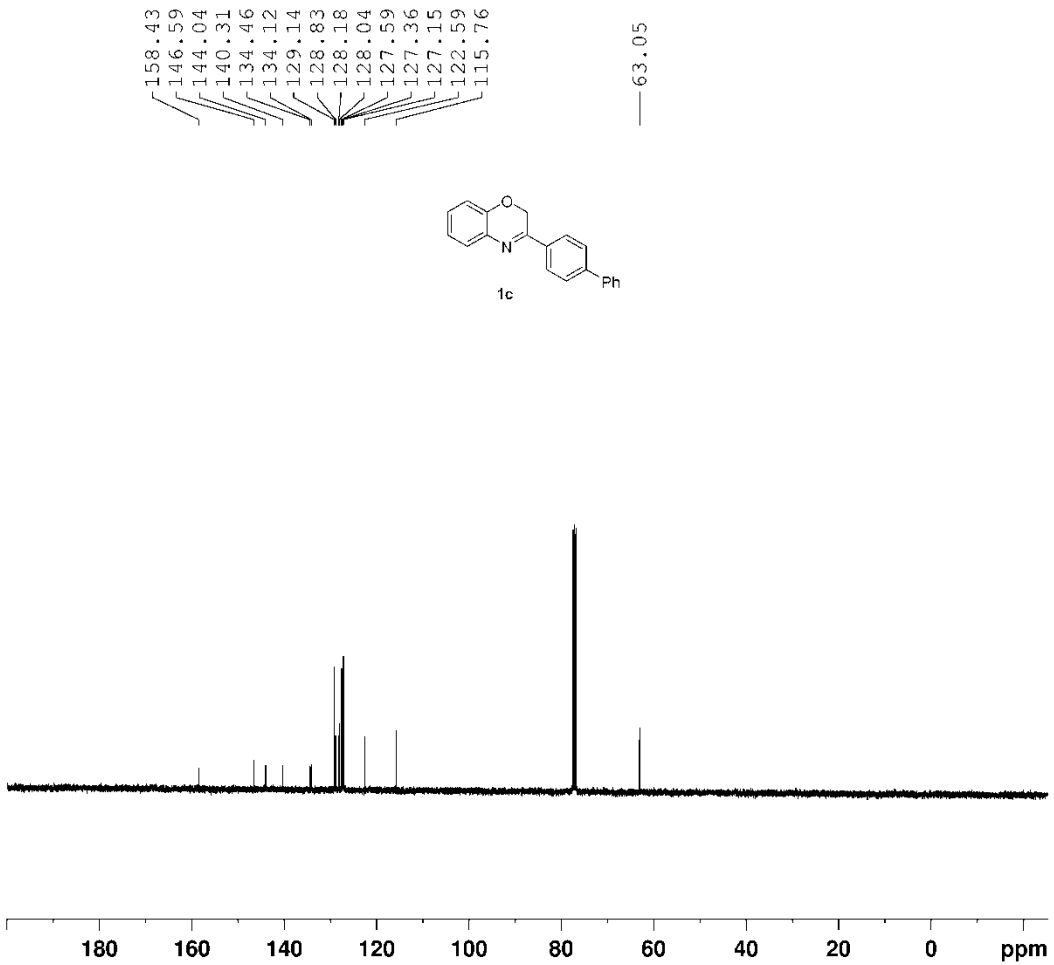
1b

```
NAME            wsm-2-C-S
EXPNO                1379
PROCNO                  1
Date_            20151114
Time                12.27
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG              zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      61
DS                       0
SWH              25252.525 Hz
FIDRES            0.770646 Hz
AQ                0.6488564 sec
RG                     128
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

-------- CHANNEL f1 --------
SFO1            100.6228298 MHz
NUC1                    13C
P1                    9.40 usec
SI                   32768
SF              100.6127538 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

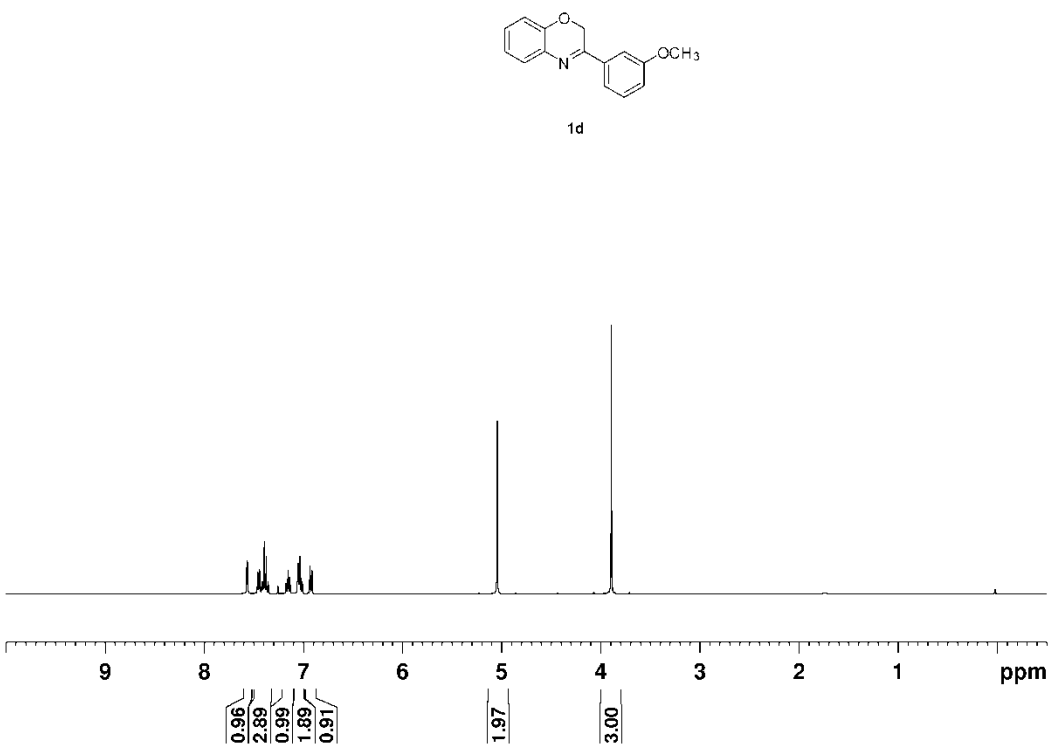




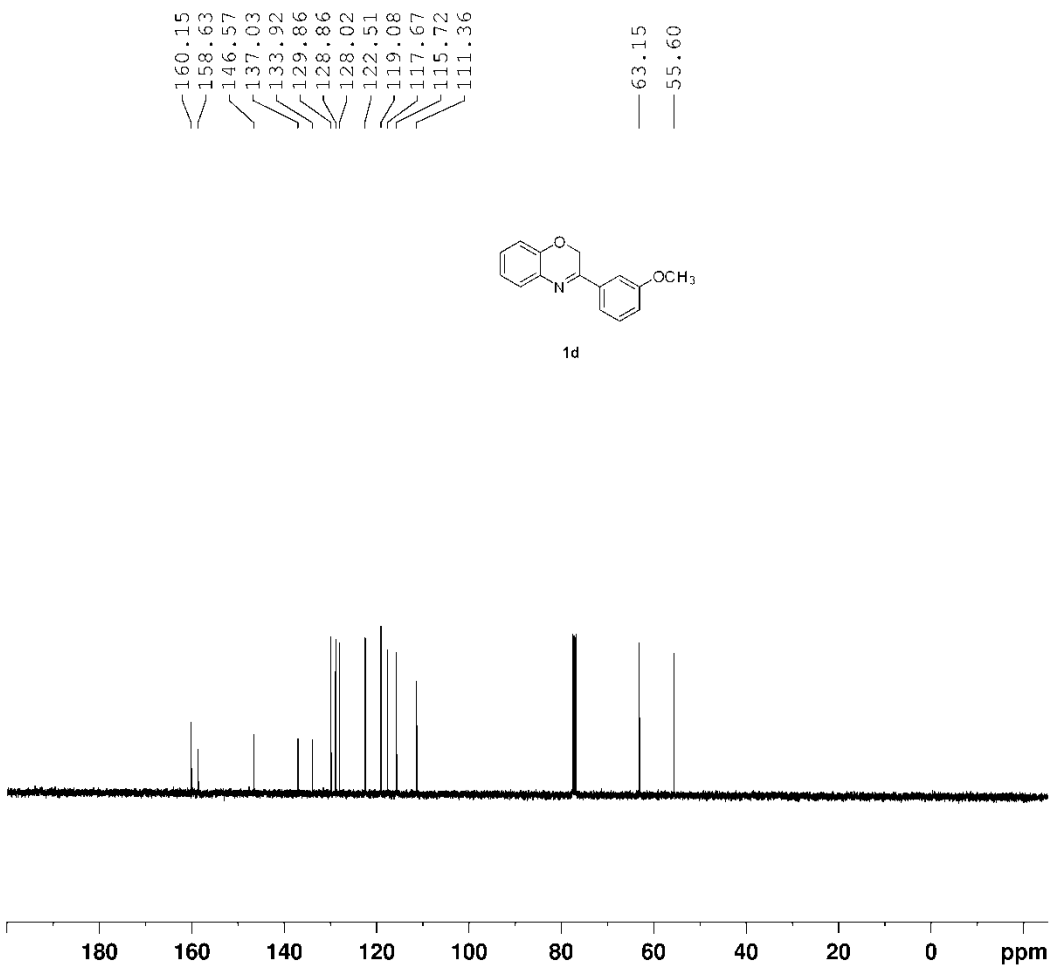
NAME	wsm-2-H-S	
EXPNO	1345	
PROCNO	2	
Date_	20151114	
Time	12.22	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300099	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



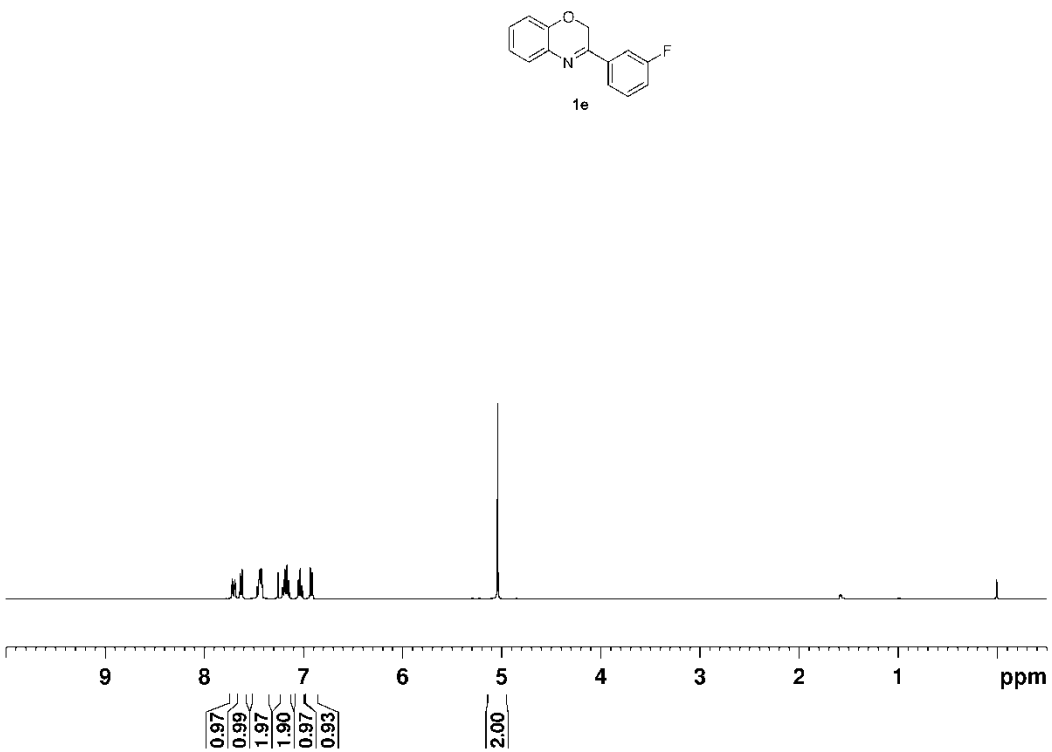
NAME	wsm-2-C-S
EXPNO	1345
PROCNO	1
Date_	20151114
Time	12.15
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	136
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	128
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127495 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



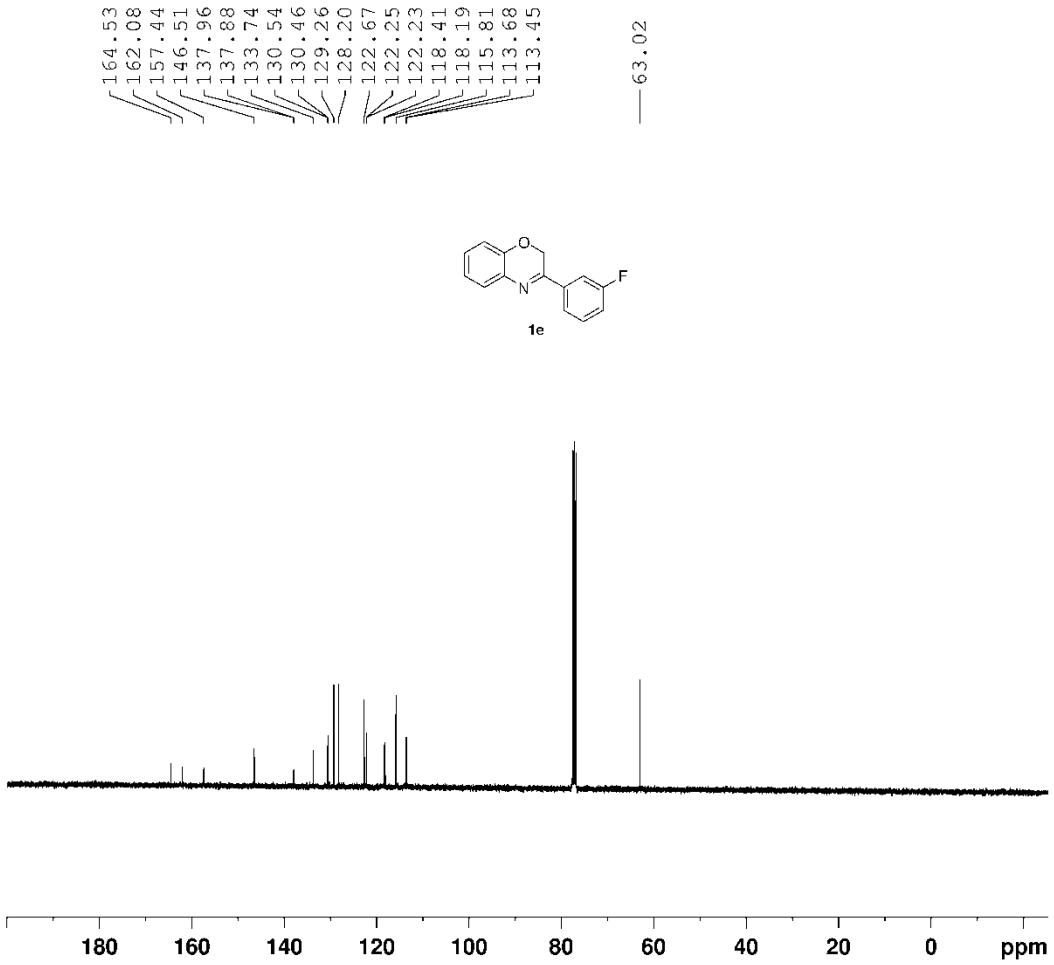
NAME	wsm-2-H-S
EXPNO	1379
PROCNO	1
Date_	20151114
Time	12.25
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	101
DW	41.600 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300100 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00



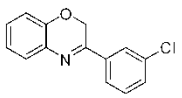

```
NAME            wsm-2-C-S
EXPNO                1379
PROCNO                  1
Date_            20151114
Time                12.27
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG              zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      61
DS                       0
SWH              25252.525 Hz
FIDRES            0.770646 Hz
AQ                0.6488564 sec
RG                     128
DW                  19.800 usec
DE                   6.50 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                     1

-------- CHANNEL f1 --------
SFO1            100.6228298 MHz
NUC1                   13C
P1                   9.40 usec
SI                   32768
SF              100.6127538 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



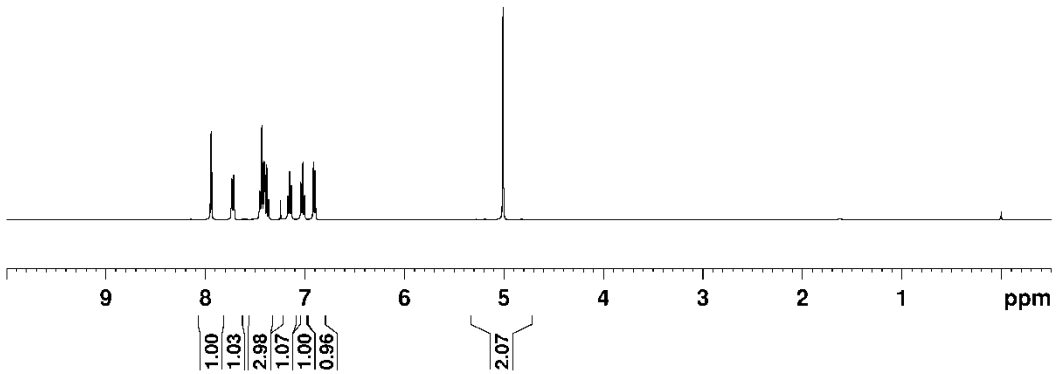
NAME	wsm-2-H-S
EXPNO	1384
PROCNO	1
Date_	20151114
Time	12.31
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	6
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	41.600 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300100 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00



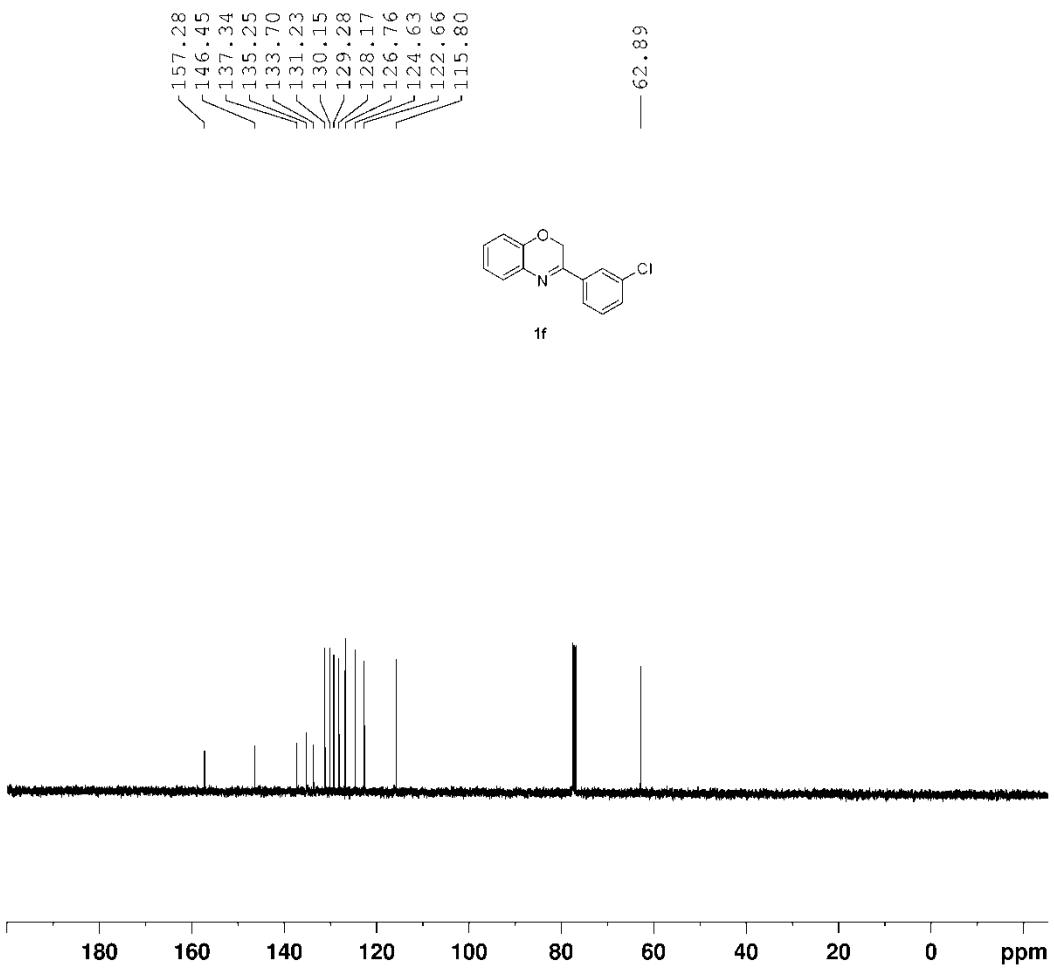
NAME	wsm-2-C-S
EXPNO	1384
PROCNO	1
Date_	20151114
Time	12.32
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	384
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488064 sec
RG	128
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127487 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



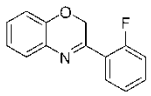
1f



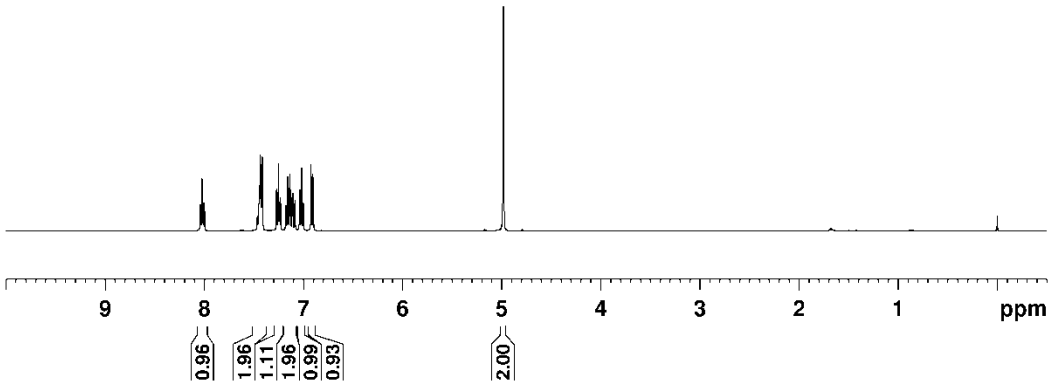
NAME	wsm-2-H-S	
EXPNO	1435	
PROCNO	1	
Date_	20160105	
Time	7.24	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	13	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	128	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300156	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



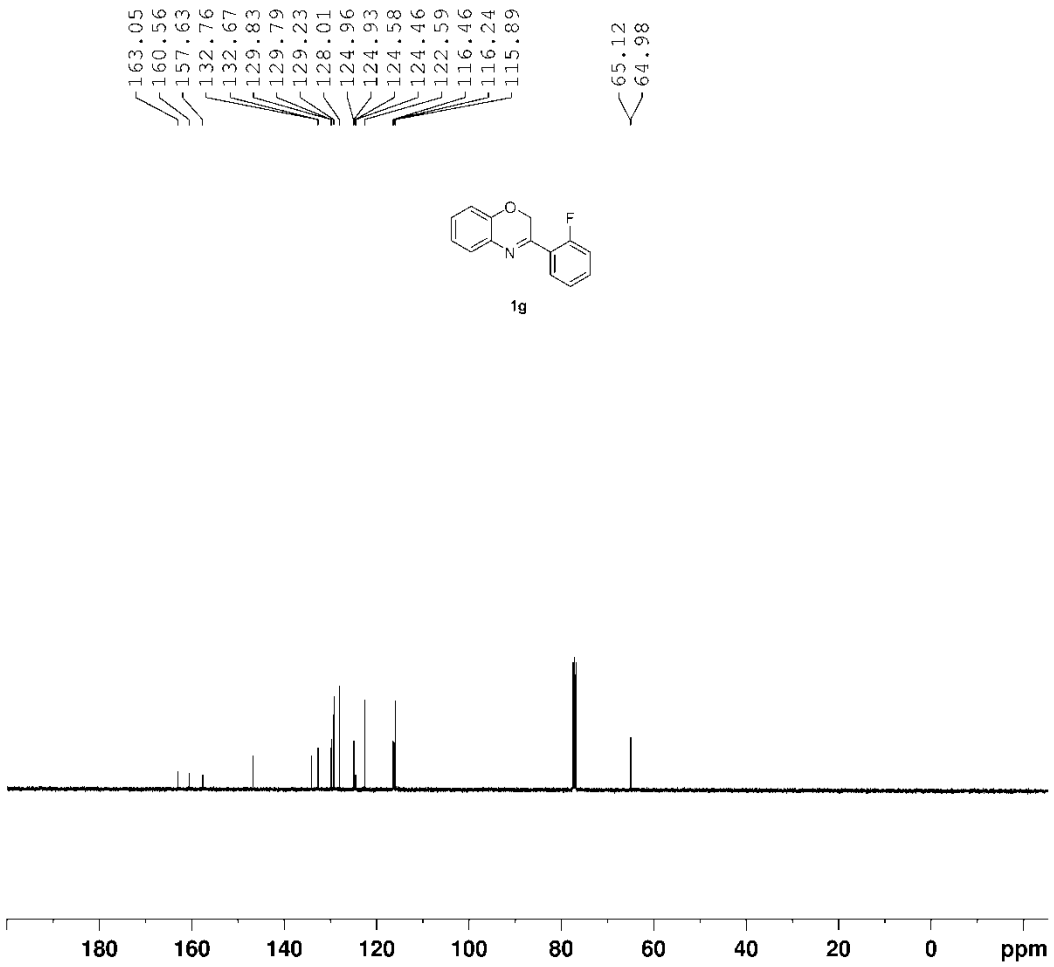

```
NAME           wsm-2-C-S
EXPNO               1435
PROCNO                 1
Date_           20160105
Time                7.26
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    47
DS                     0
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ             0.6488564 sec
RG                   161
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.40 usec
SI                32768
SF          100.6127524 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                 1.40
```



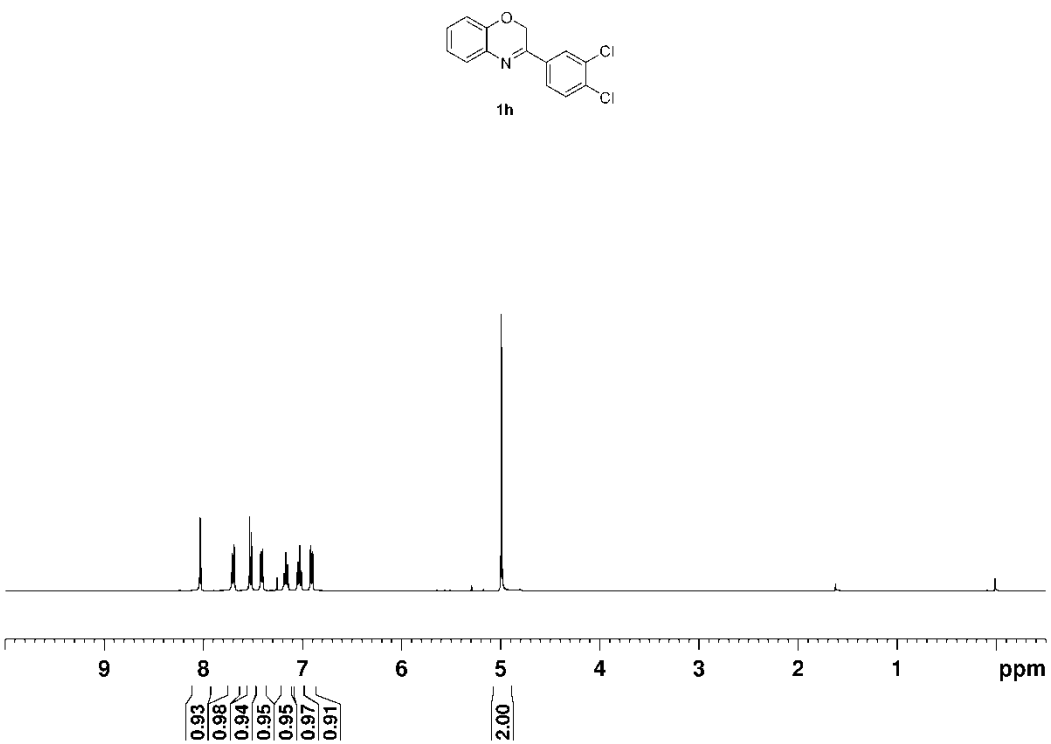
1g



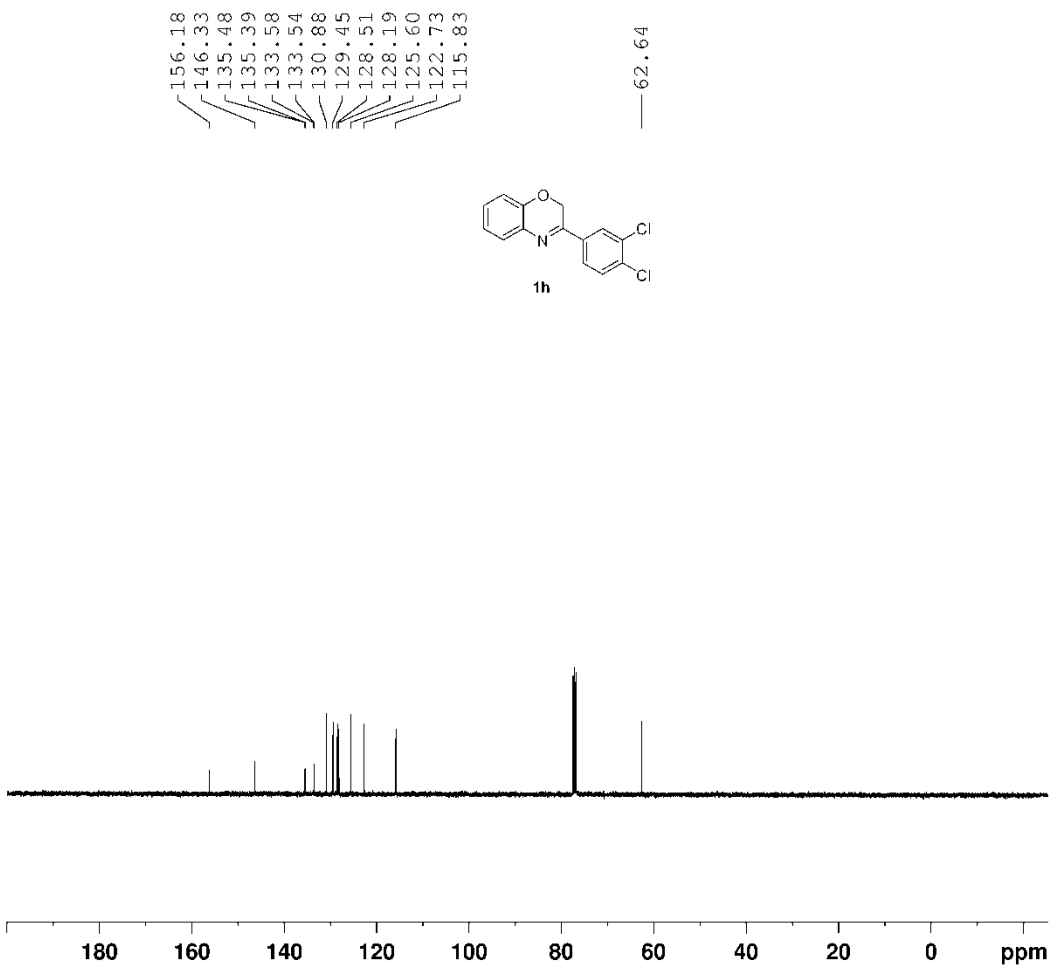
NAME	wsm-2-H-S
EXPNO	1408
PROCNO	1
Date_	20151119
Time	4.37
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	128
DW	41.600 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300169 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00



NAME	wsm-2-C-S
EXPNO	1408
PROCNO	1
Date_	20151119
Time	4.39
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	148
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	128
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127519 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

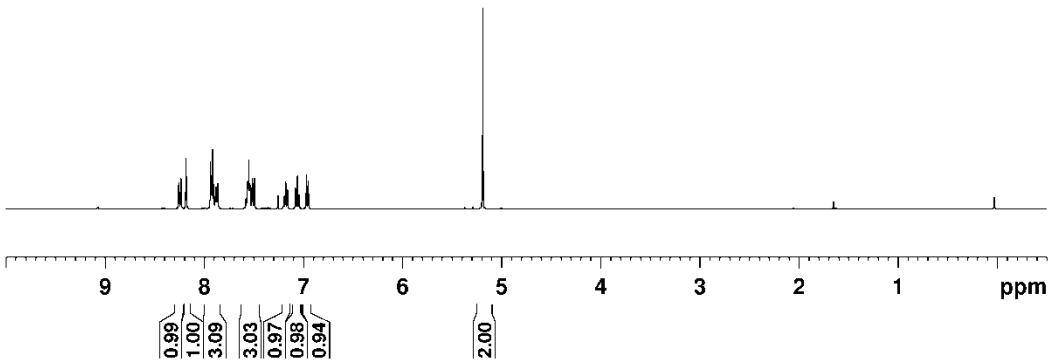


NAME	wsm-2-H-S	
EXPNO	1331	
PROCNO	1	
Date_	20151114	
Time	12.10	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	12	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	144	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300100	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	

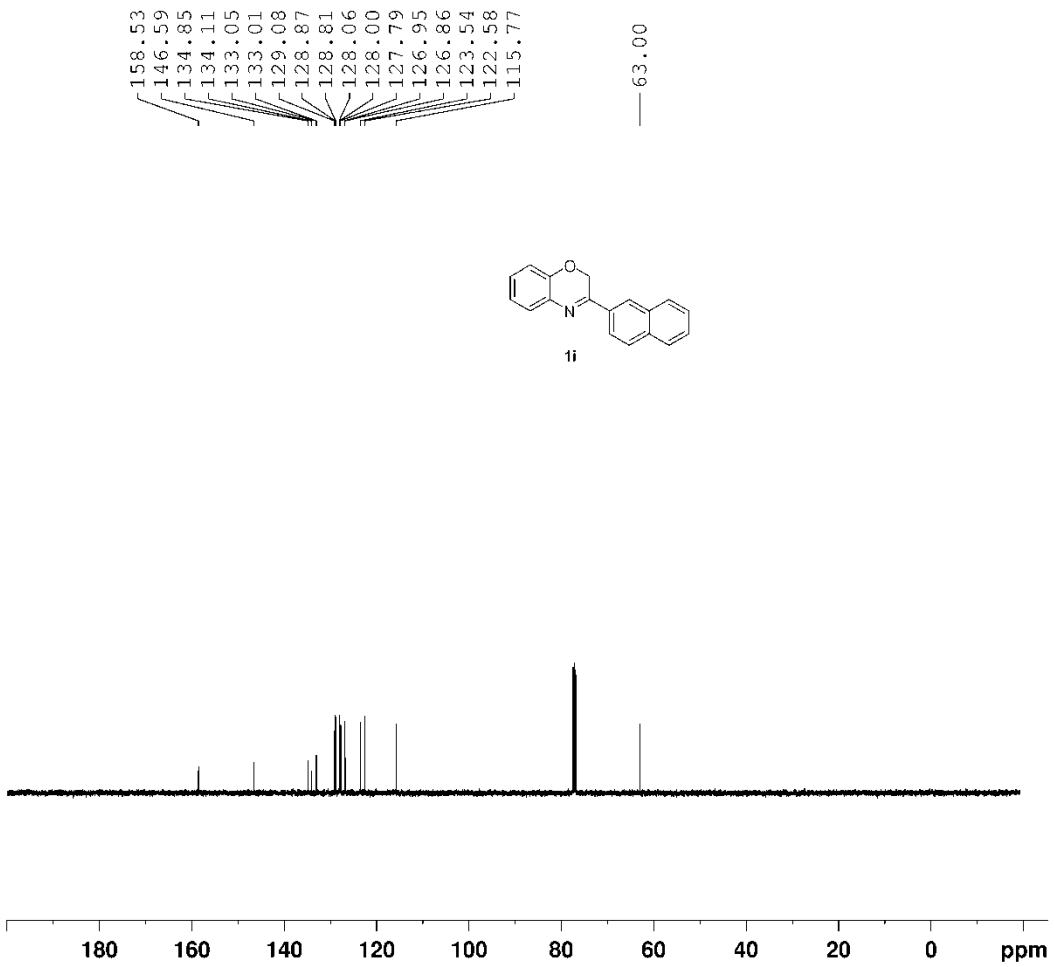



```
NAME           wsm-2-C-S
EXPNO               1331
PROCNO                 1
Date_           20151114
Time               12.10
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG            zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    60
DS                     0
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ             0.6488564 sec
RG                   128
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6228298 MHz
NUC1                  13C
P1                  9.40 usec
SI                 32768
SF          100.6127517 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

1i

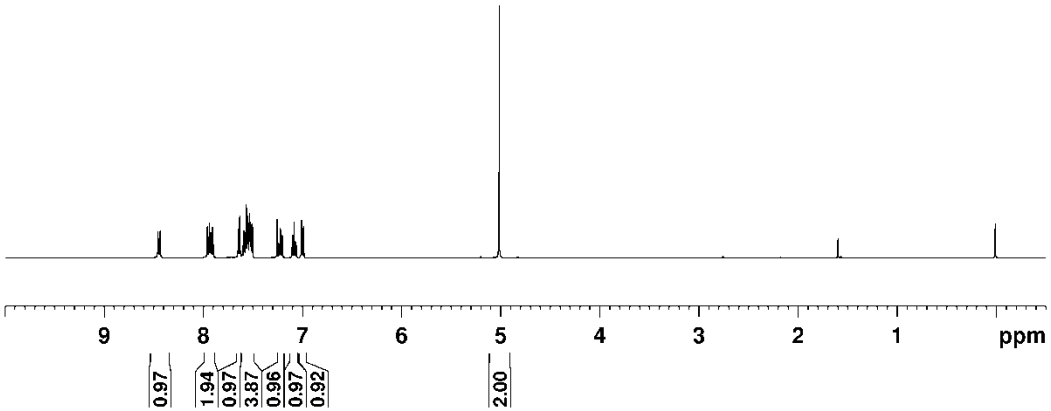


NAME	wsm-2-H-S	
EXPNO	1420	
PROCNO	1	
Date_	20151123	
Time	16.26	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	13	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	116.67	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400098	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



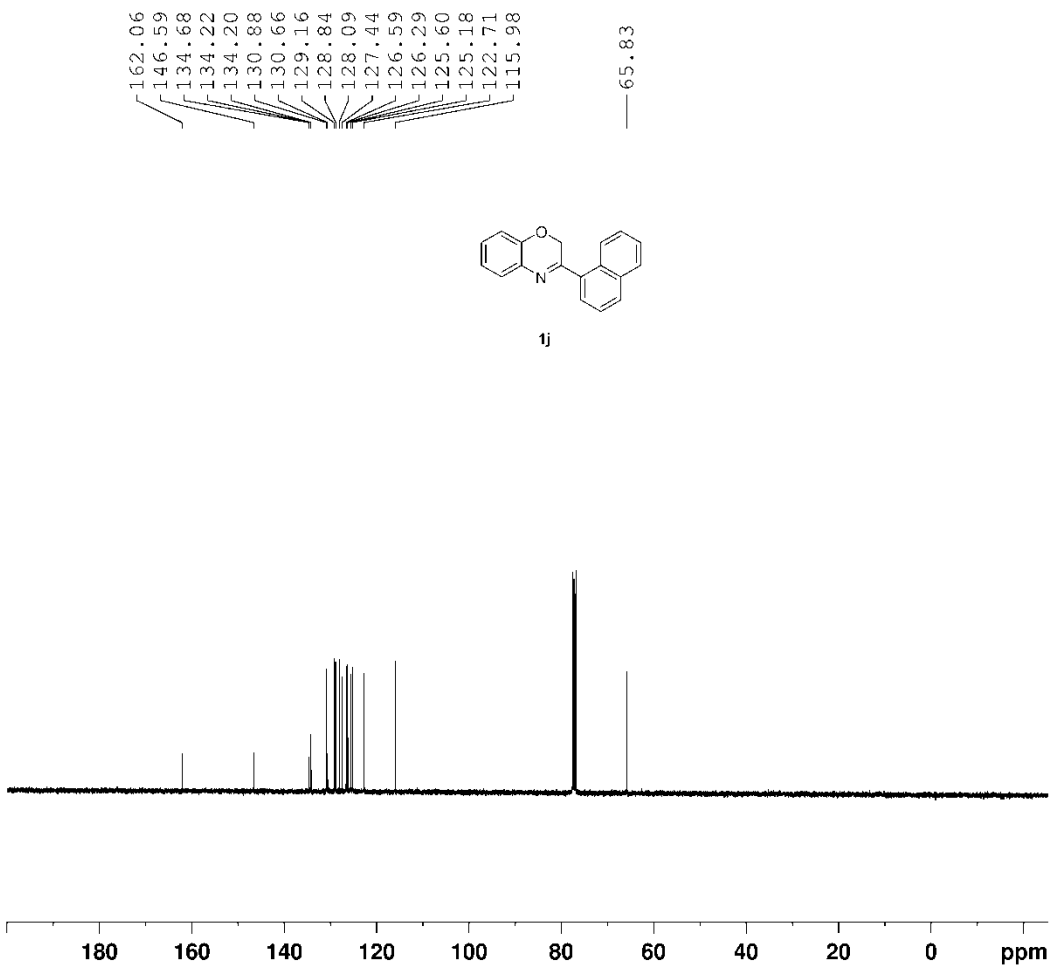

```
NAME           wsm-2-C-S
EXPNO               1420
PROCNO                 1
Date_           20151123
Time               16.29
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    29
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6304916 MHz
NUC1               13C
P1                10.00 usec
SI                32768
SF          100.6404126 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1j

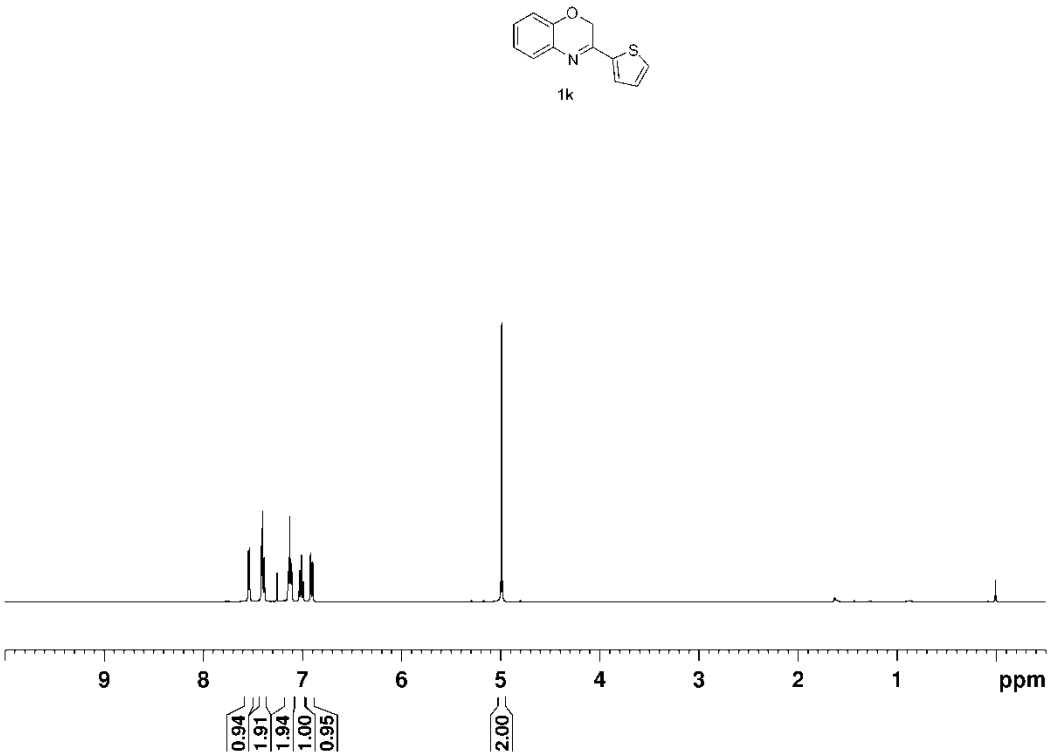


NAME	wsm-2-H-S
EXPNO	1402
PROCNO	1
Date_	20151120
Time	9.42
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	13
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	41.600 usec
DE	6.50 usec
TE	293.0 K
D1	2.00000000 sec
TD0	1

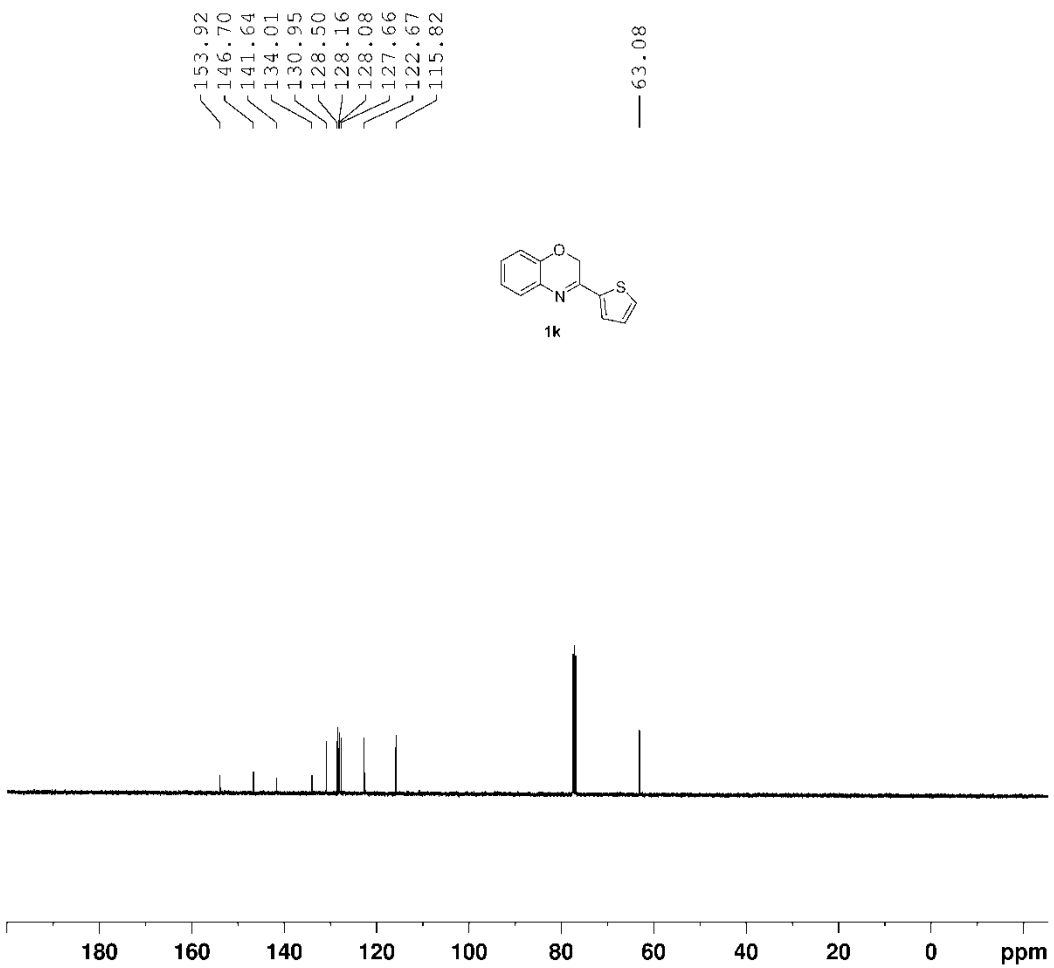
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300102 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00



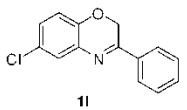

```
NAME            wsm-2-C-S
EXPNO                1402
PROCNO                  1
Date_            20151117
Time                 7.08
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG              zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     303
DS                       0
SWH              25252.525 Hz
FIDRES            0.770646 Hz
AQ                0.6488564 sec
RG                     128
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

-------- CHANNEL f1 --------
SFO1         100.6228298 MHz
NUC1                  13C
P1                  9.40 usec
SI                 32768
SF           100.6127540 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



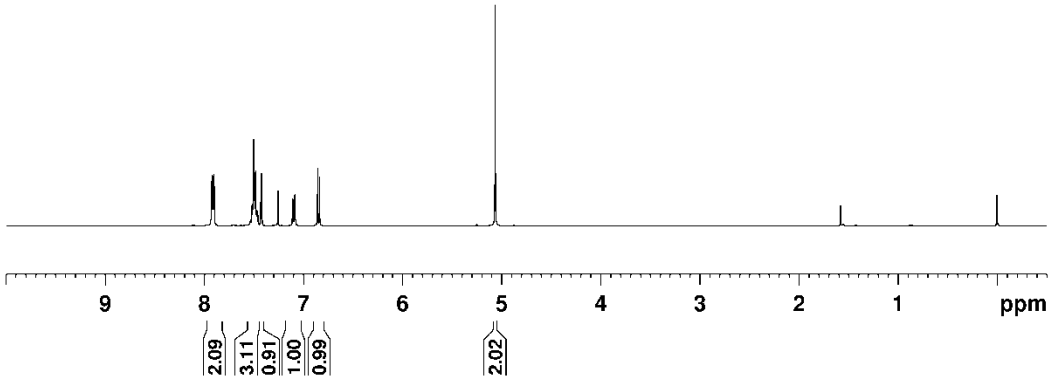
NAME	wsm-2-H-S	
EXPNO	1388	
PROCNO	1	
Date_	20151115	
Time	7.53	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300102	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



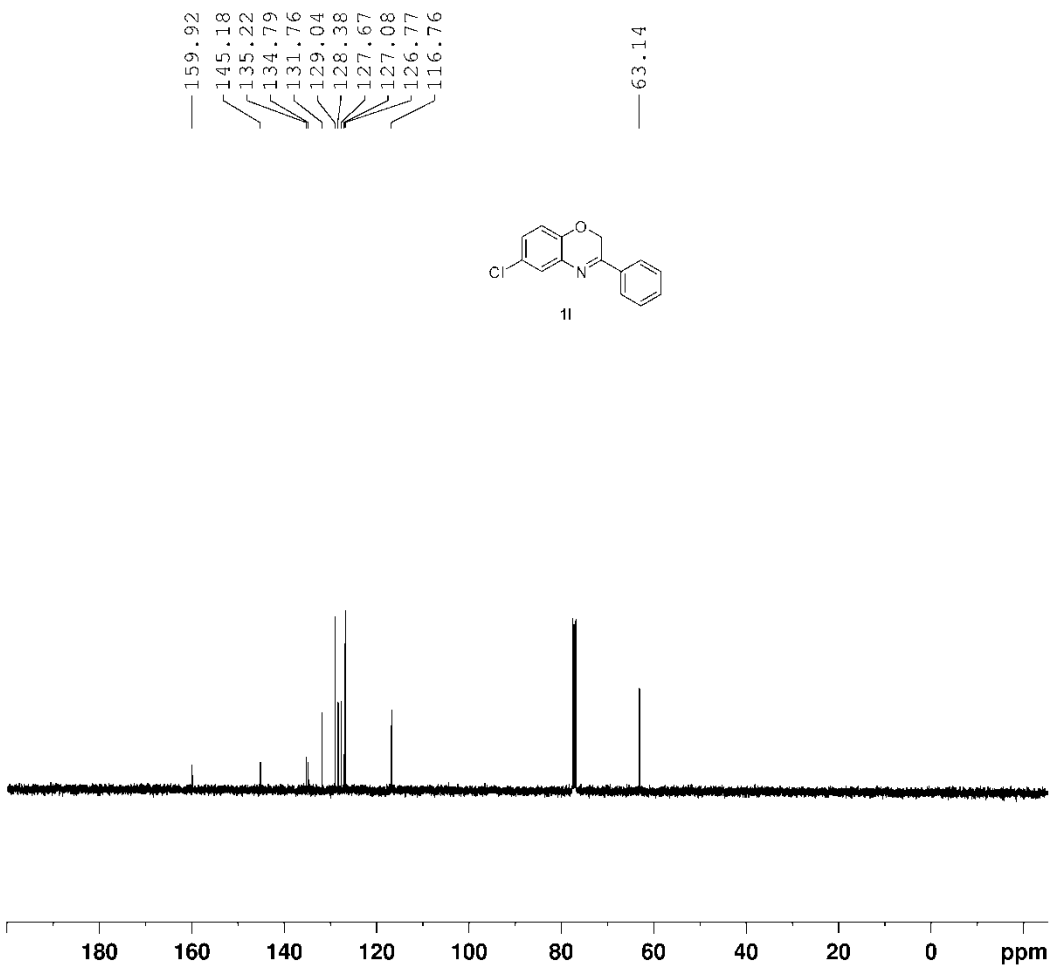
NAME	wsm-2-C-S
EXPNO	1388
PROCNO	1
Date_	20151115
Time	7.57
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	160
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488064 sec
RG	128
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127502 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



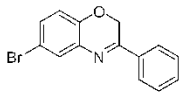
1l



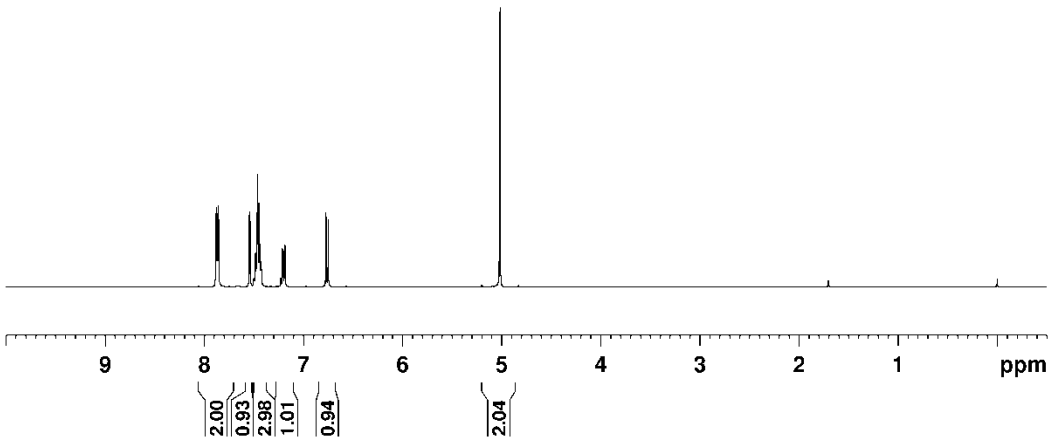
NAME	wsm-2-H-S	
EXPNO	1409	
PROCNO	1	
Date_	20151120	
Time	9.52	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	41.600	usec
DE	6.50	usec
TE	293.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300104	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



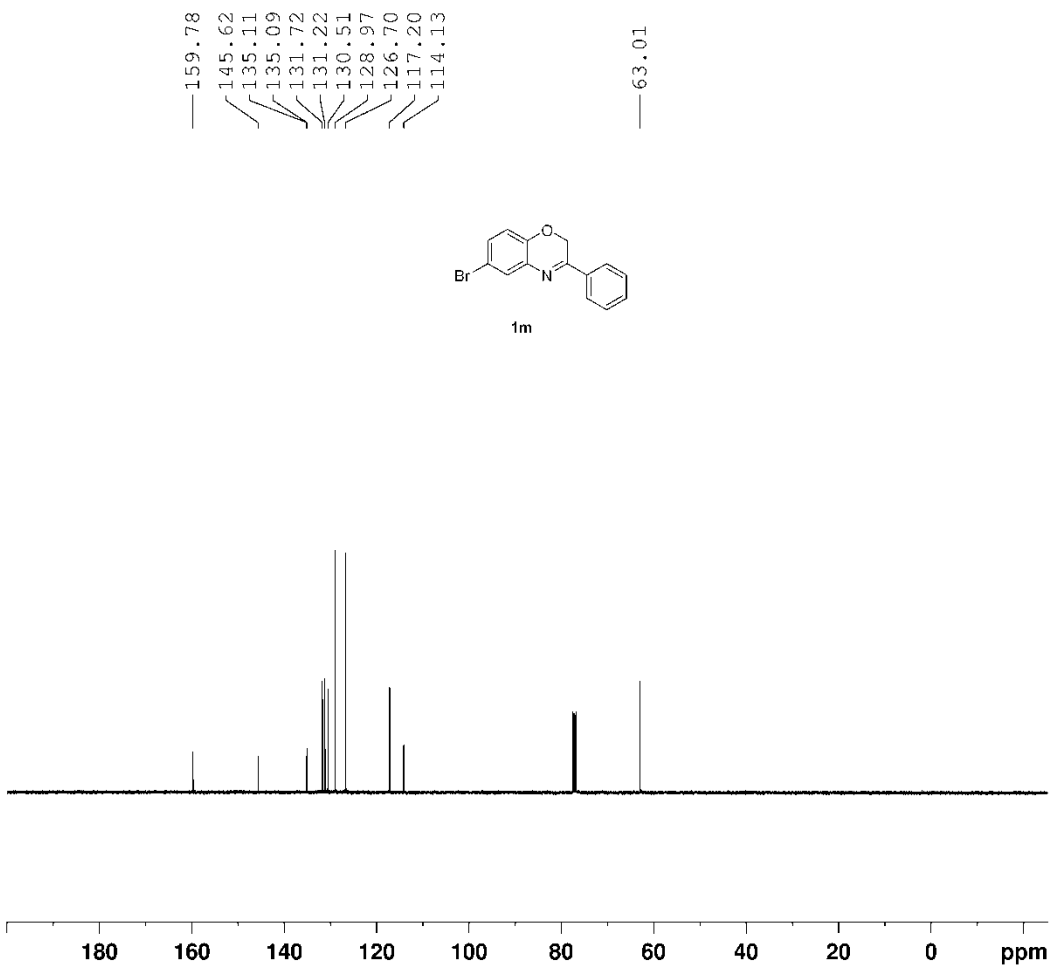

```
NAME            wsm-2-C-S
EXPNO                1409
PROCNO                  1
Date_            20151120
Time                 9.54
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG              zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      44
DS                       0
SWH              25252.525 Hz
FIDRES            0.770646 Hz
AQ                0.6488564 sec
RG                     128
DW                  19.800 usec
DE                   6.50 usec
TE                  295.4 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                   13C
P1                   9.40 usec
SI                   32768
SF              100.6127498 MHz
WDW                    EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



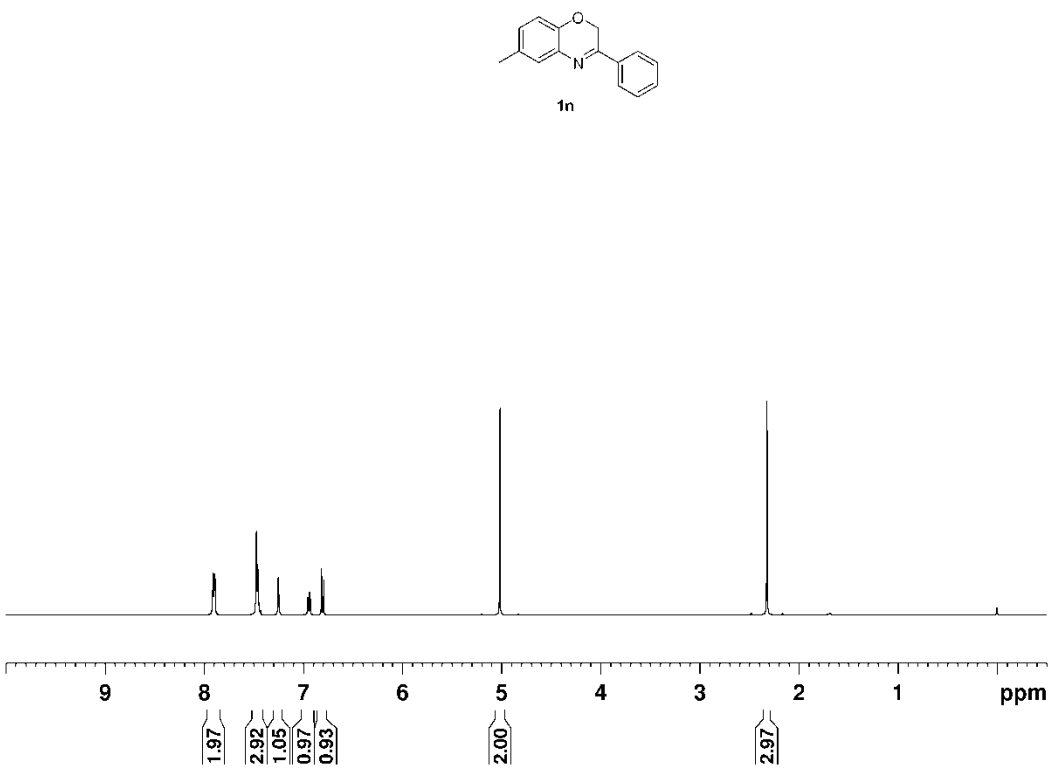
1m



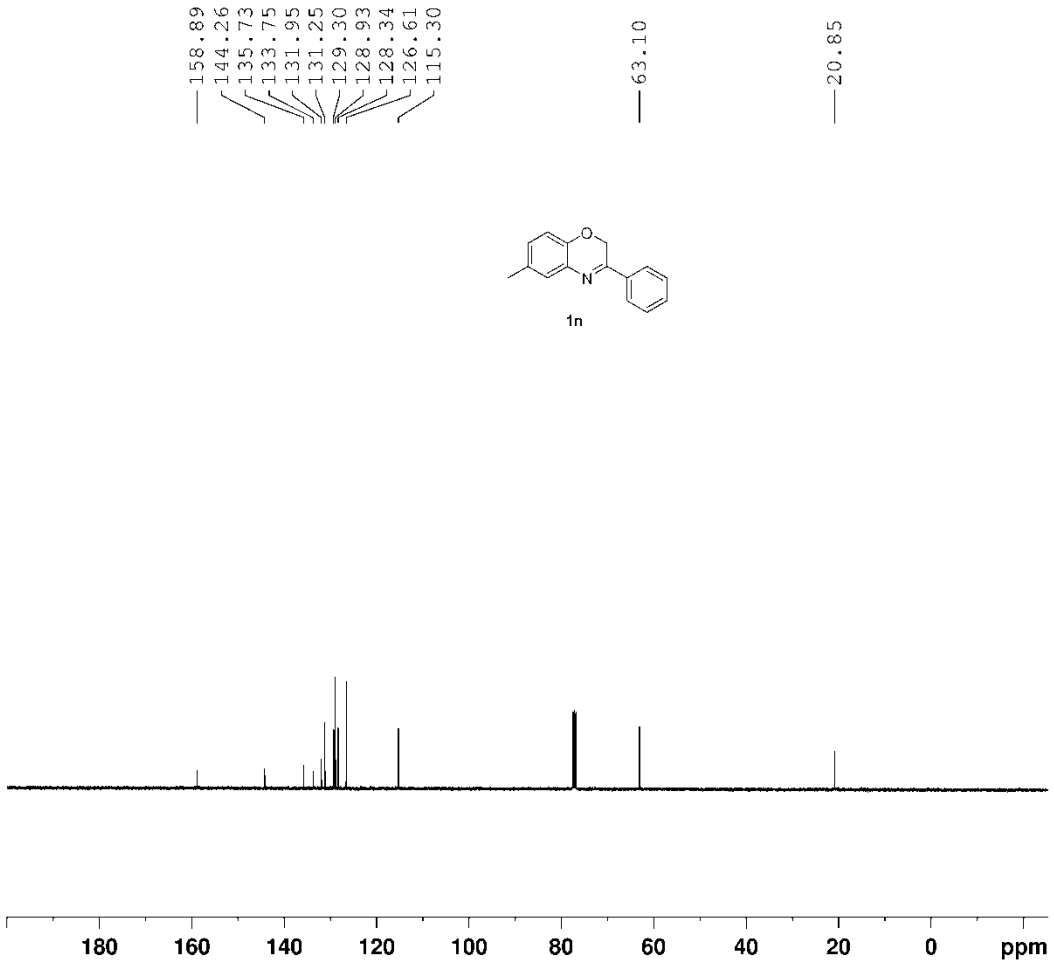
NAME	wsm-2-H-S	
EXPNO	1412	
PROCNO	1	
Date_	20151120	
Time	10.06	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	90.5	
DW	41.600	usec
DE	6.50	usec
TE	294.8	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300213	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



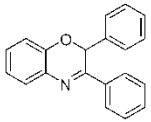

```
NAME          wsm-2-C-S
EXPNO              1412
PROCNO                2
Date_          20151120
Time              10.08
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                32768
SOLVENT           CDCl3
NS                   99
DS                    0
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ             0.6488564 sec
RG                  128
DW                19.800 usec
DE                 6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.40 usec
SI               32768
SF          100.6127571 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



NAME	wsm-2-H-S
EXPNO	1411
PROCNO	1
Date_	20151120
Time	9.58
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	128
DW	41.600 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300169 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

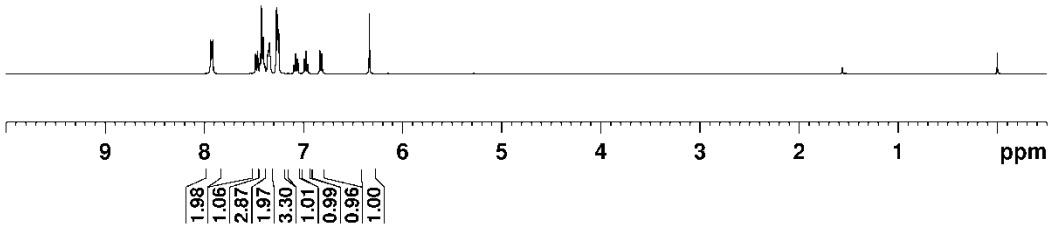


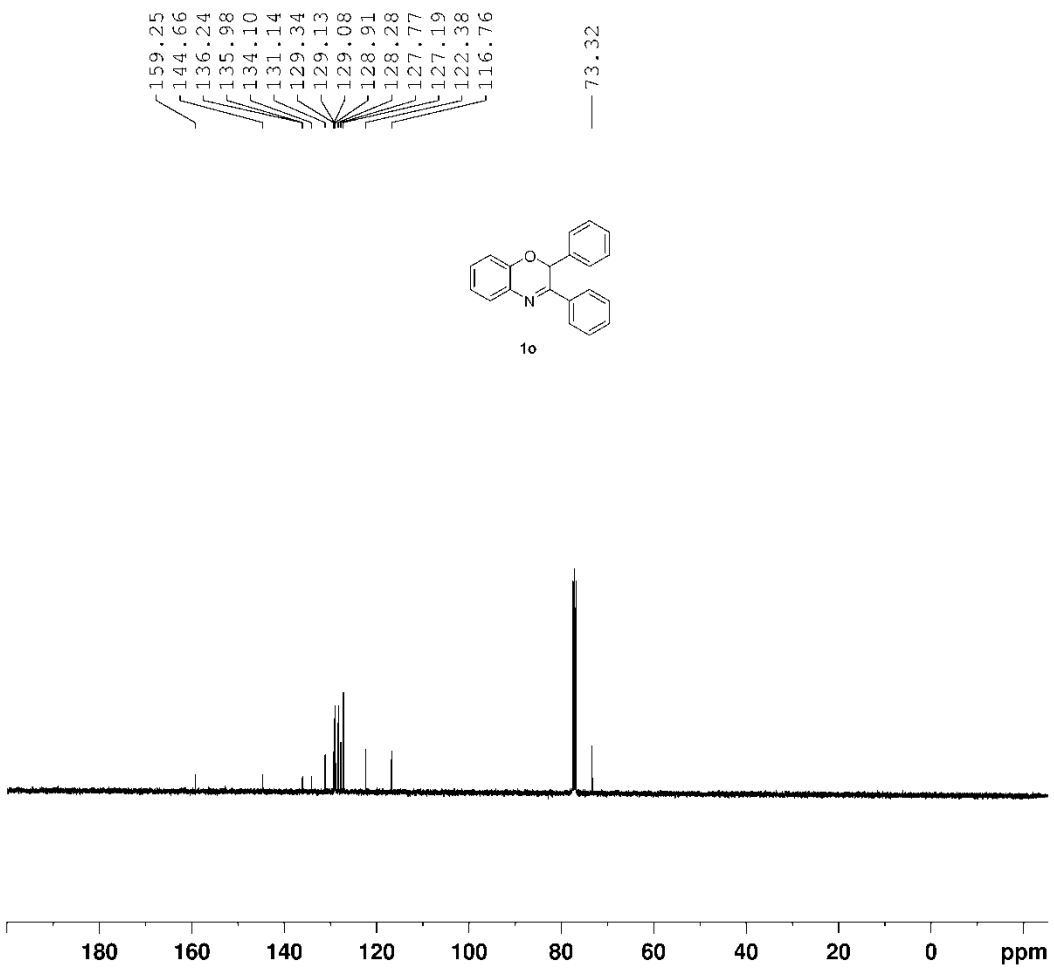
NAME	wsm-2-C-S
EXPNO	1411
PROCNO	1
Date_	20151120
Time	10.01
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	100
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	128
DW	19.800 usec
DE	6.50 usec
TE	295.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127533 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



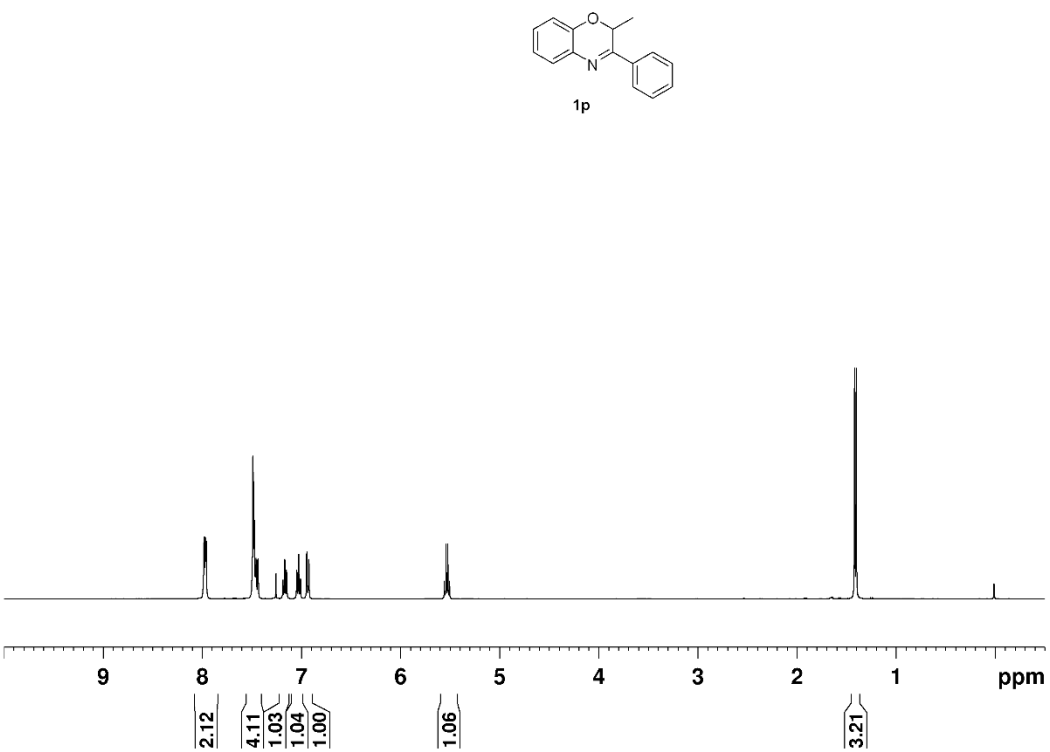
1o

NAME	wsm-2-H-S	
EXPNO	1310	
PROCNO	1	
Date_	20160105	
Time	7.05	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	13	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300142	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	

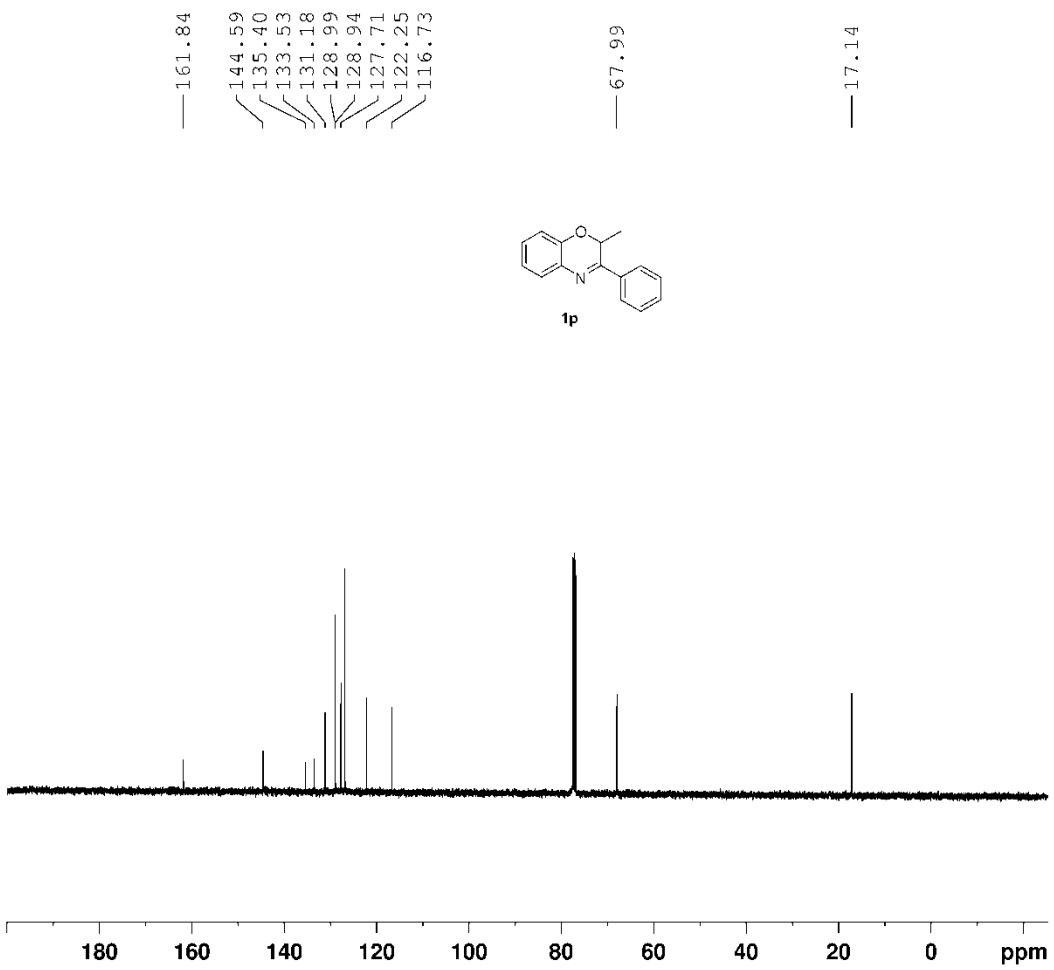




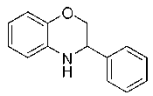
NAME	wsm-2-C-S
EXPNO	1310
PROCNO	1
Date_	20160105
Time	7.07
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	239
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488064 sec
RG	161
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127495 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



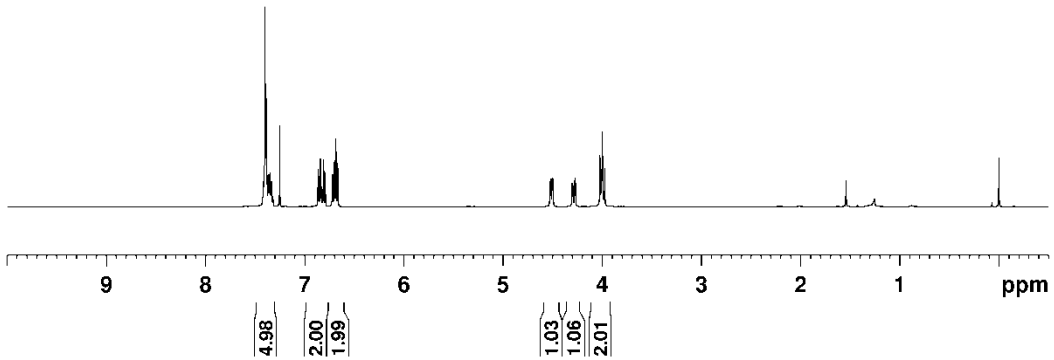

```
NAME           wsm-2-H-S
EXPNO               1482
PROCNO                 1
Date_           20160107
Time               11.14
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                   128
DW                41.600 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                   1H
P1                 12.60 usec
SI                 65536
SF           400.1300100 MHz
WDW                   EM
SSB                    0
LB                  0.50 Hz
GB                     0
PC                  1.00
```



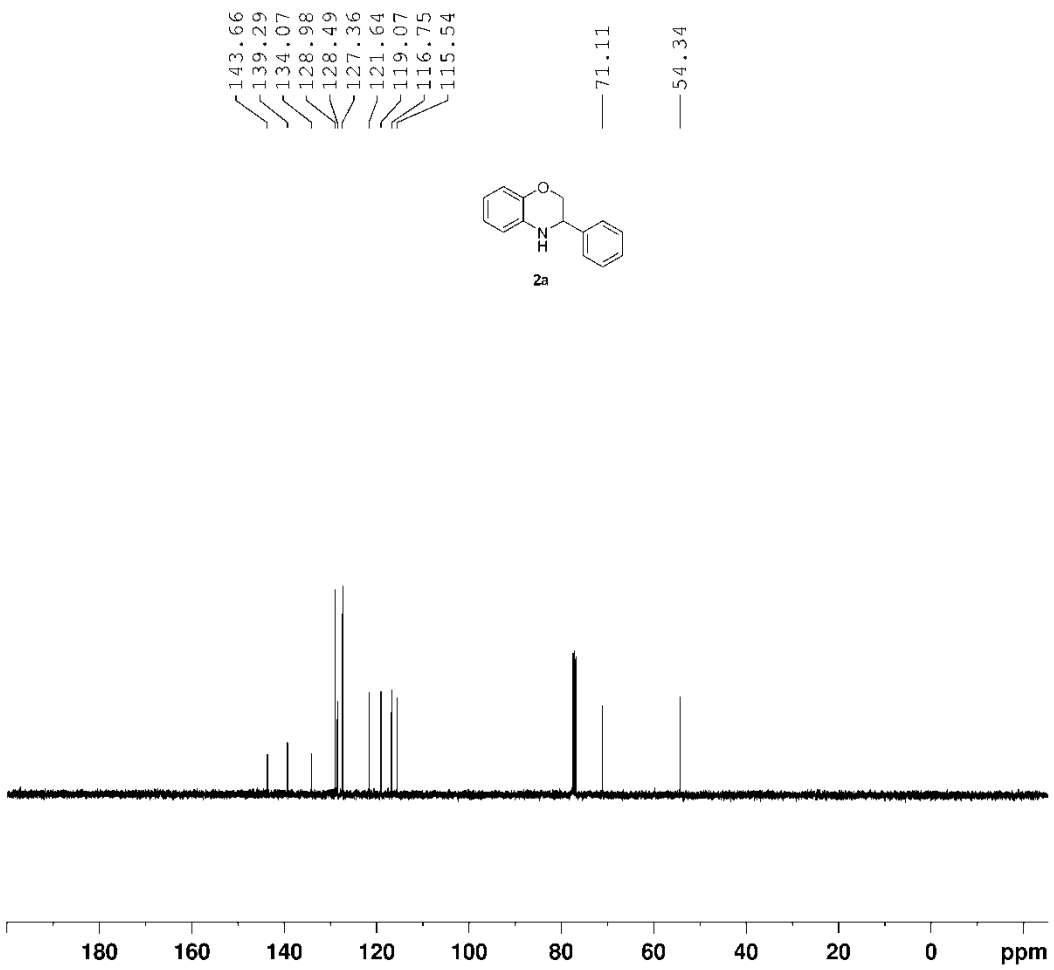

```
NAME           wsm-2-C-S
EXPNO               1482
PROCNO                 1
Date_           20160107
Time               11.16
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   100
DS                     0
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ             0.6488064 sec
RG                   161
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6228298 MHz
NUC1                 13C
P1                  9.40 usec
SI                 32768
SF          100.6127510 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



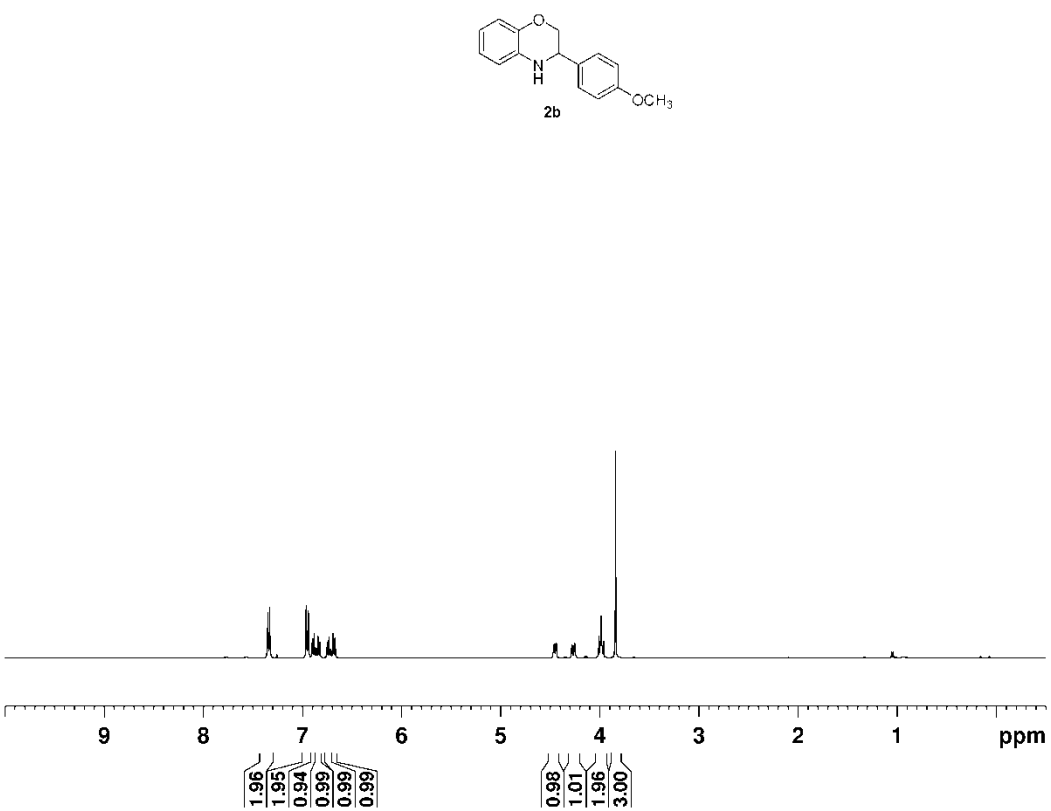
2a



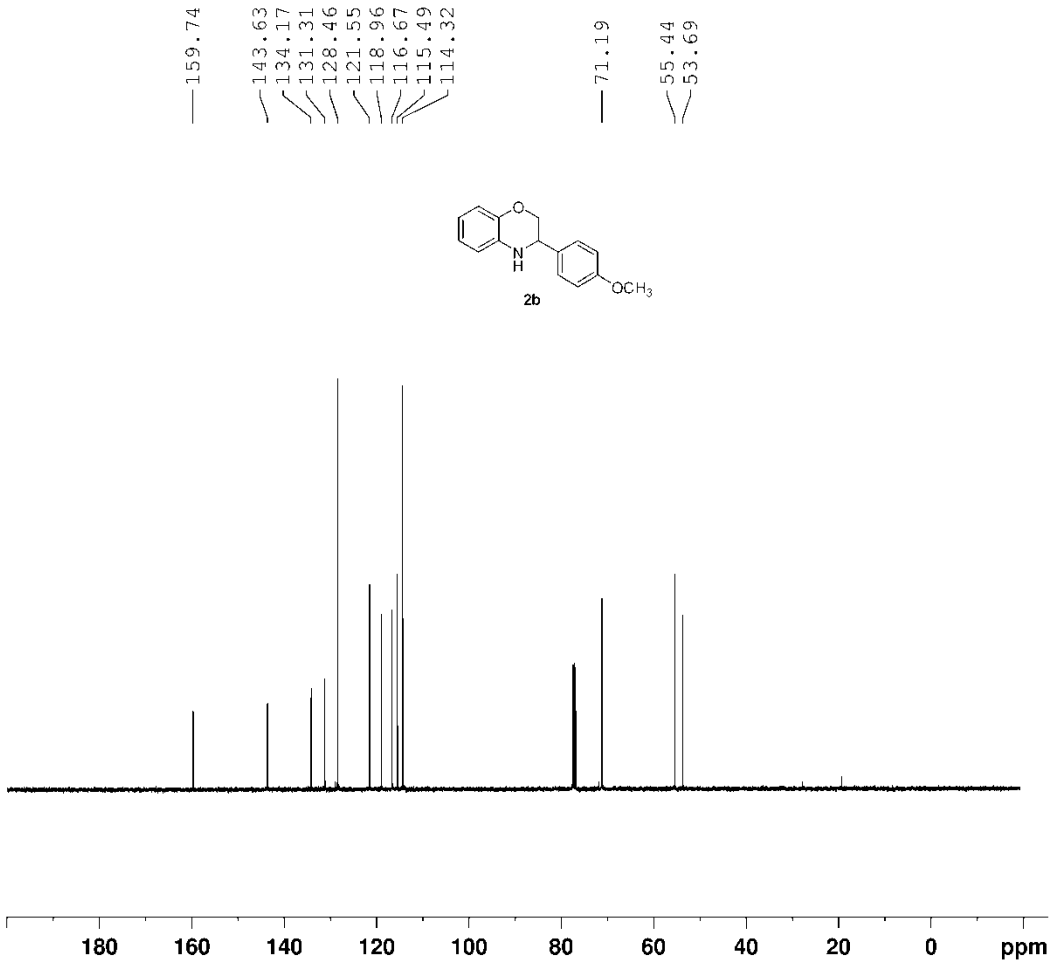
NAME	wsm-2-H-P
EXPNO	1480
PROCNO	1
Date_	20151225
Time	7.48
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	41.600 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300129 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00



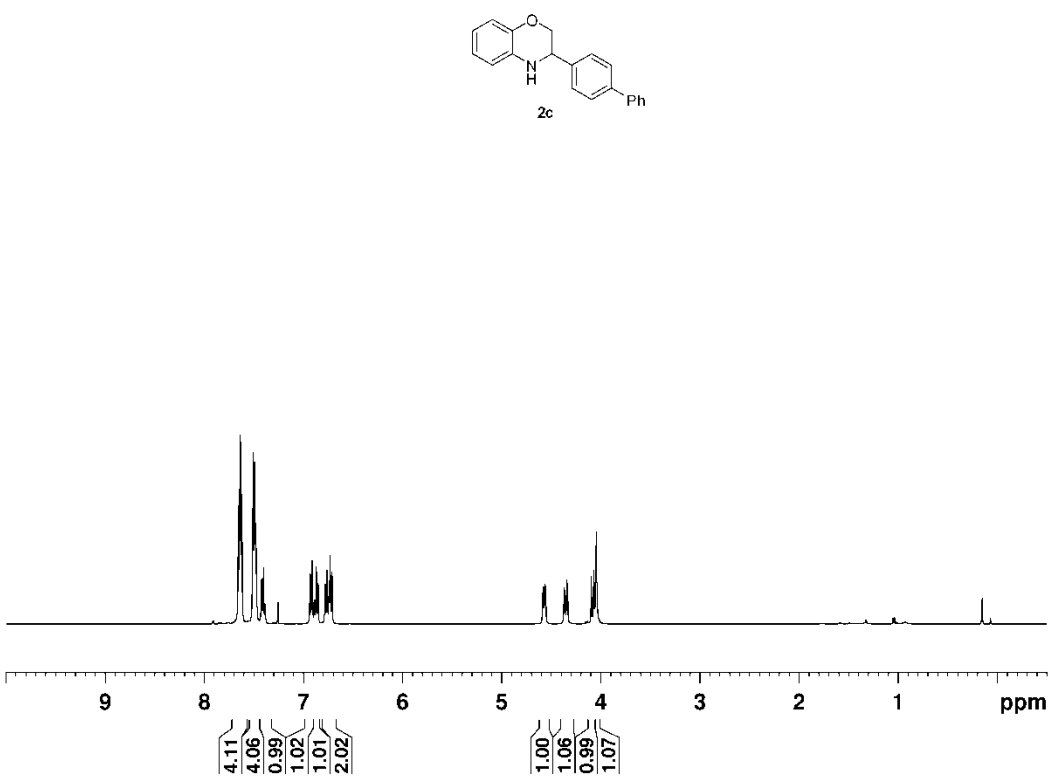

```
NAME           wsm-2-C-P
EXPNO               1440
PROCNO                 1
Date_           20151209
Time                7.22
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    13
DS                     0
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ             0.6488564 sec
RG                   128
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6228298 MHz
NUC1                  13C
P1                  9.40 usec
SI                 32768
SF          100.6127594 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



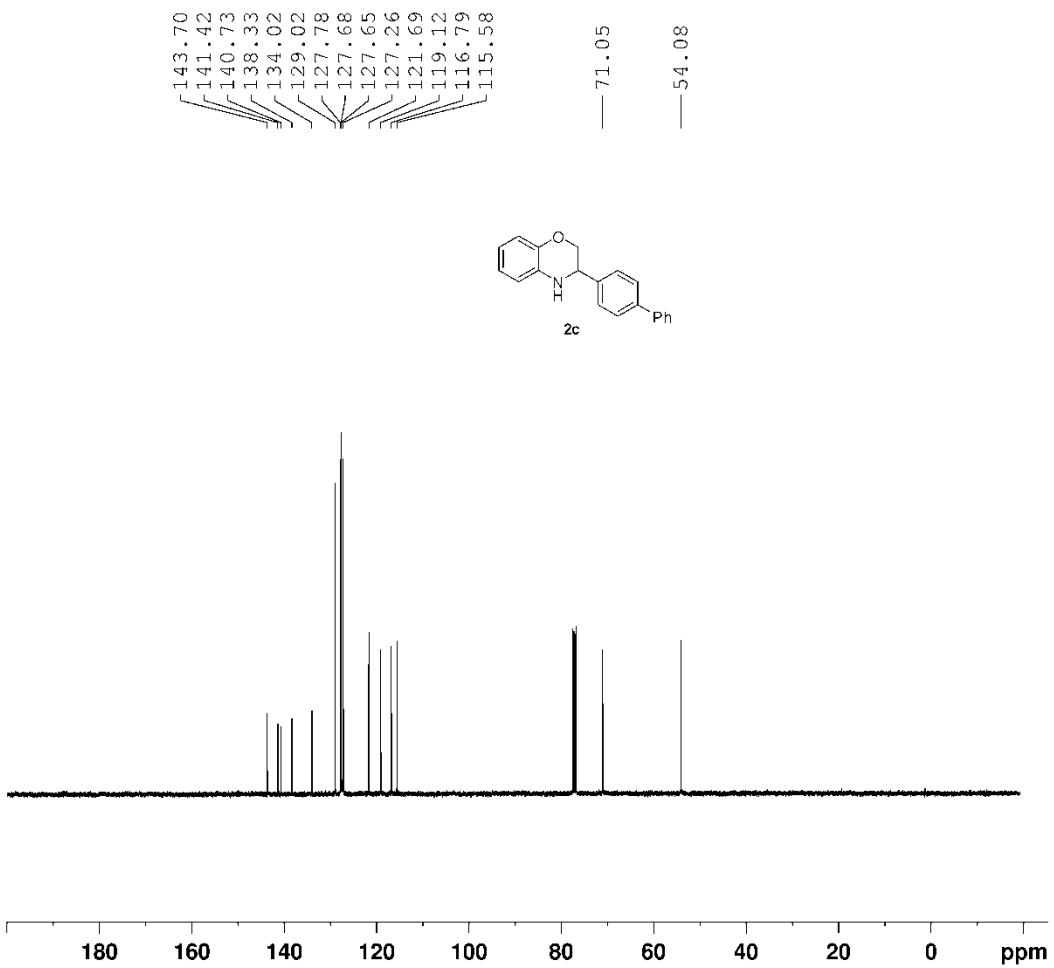
NAME	wsm-2-H-P	
EXPNO	1450	
PROCNO	1	
Date_	20151127	
Time	14.40	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	13	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	125.02	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400093	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	




```
NAME           wsm-2-C-P
EXPNO               1450
PROCNO                 1
Date_           20151127
Time               10.28
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    59
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6304916 MHz
NUC1               13C
P1                10.00 usec
SI                32768
SF          100.6404215 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

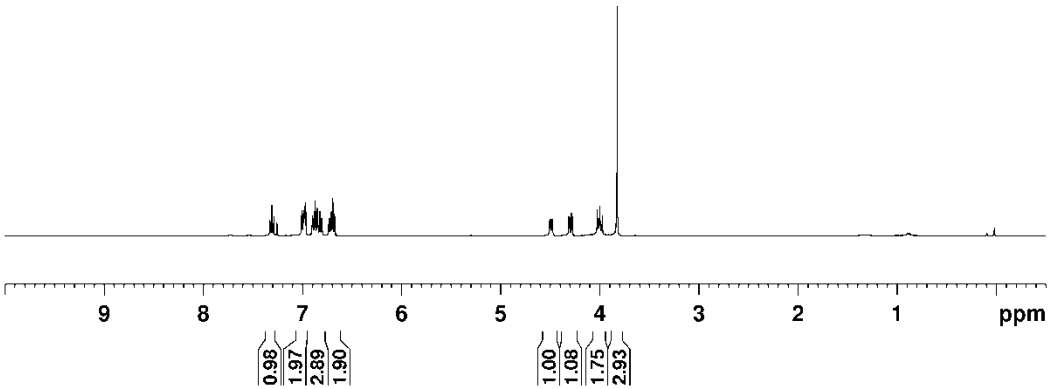


NAME	wsm-2-H-P
EXPNO	1455
PROCNO	1
Date_	20151127
Time	14.53
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	2.0447731 sec
RG	140.59
DW	62.400 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.2424716 MHz
NUC1	1H
P1	14.80 usec
SI	65536
SF	400.2400095 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

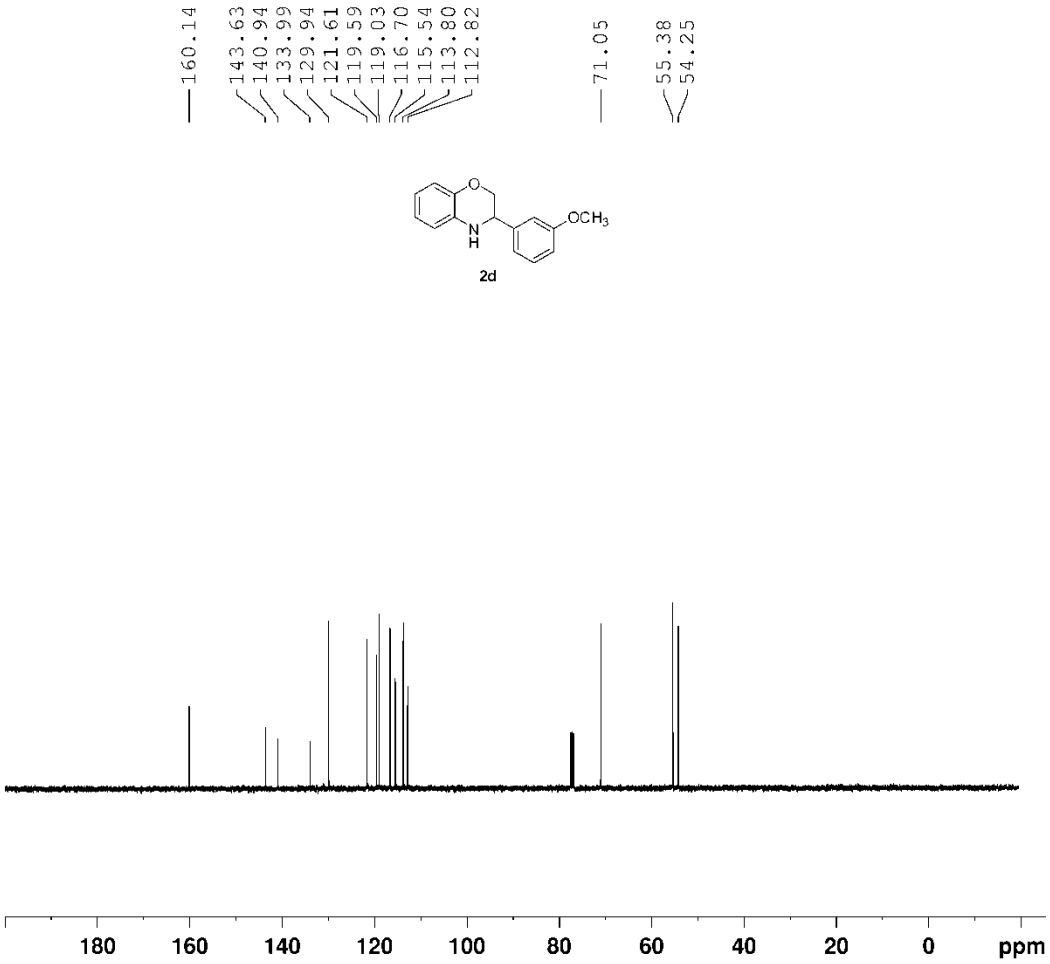


NAME	wsm-2-C-P
EXPNO	1455
PROCNO	1
Date_	20151127
Time	10.50
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	24
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	206.33
DW	20.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6304916 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6404201 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

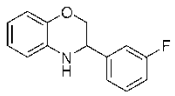
2d



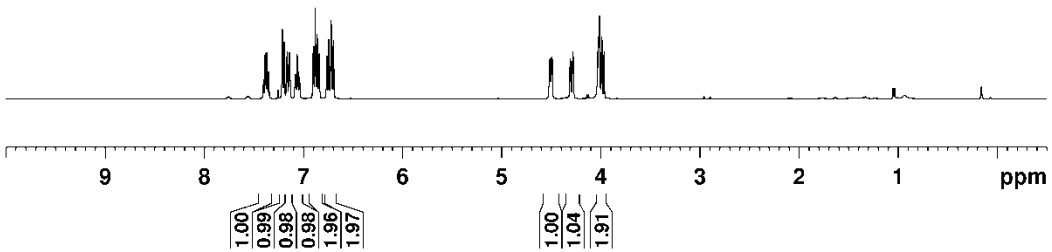
NAME	wsm-2-H-P	
EXPNO	1449	
PROCNO	2	
Date_	20151127	
Time	14.35	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	116.67	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400093	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



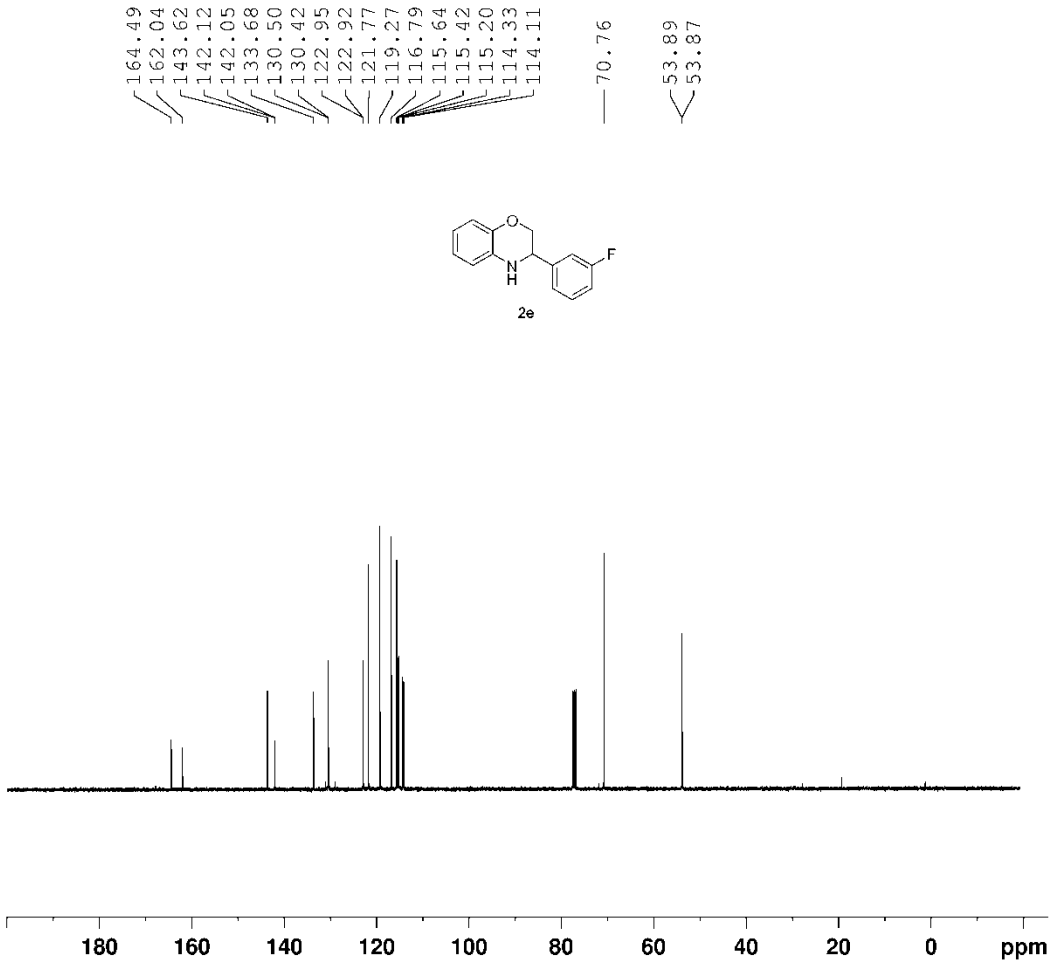

```
NAME           wsm-2-C-P
EXPNO               1449
PROCNO                 1
Date_           20151127
Time               10.20
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    28
DS                     4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG               206.33
DW               20.800 usec
DE                6.50 usec
TE               298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6304916 MHz
NUC1               13C
P1               10.00 usec
SI                32768
SF          100.6404228 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



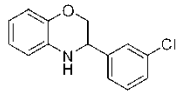
2e



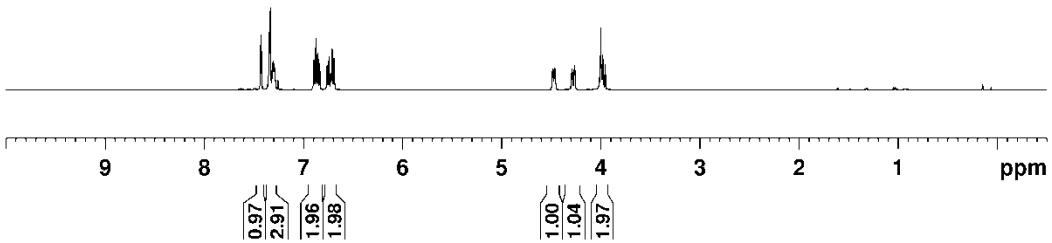
NAME	wsm-2-H-P	
EXPNO	1452	
PROCNO	1	
Date_	20151127	
Time	14.43	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	206.33	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400095	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



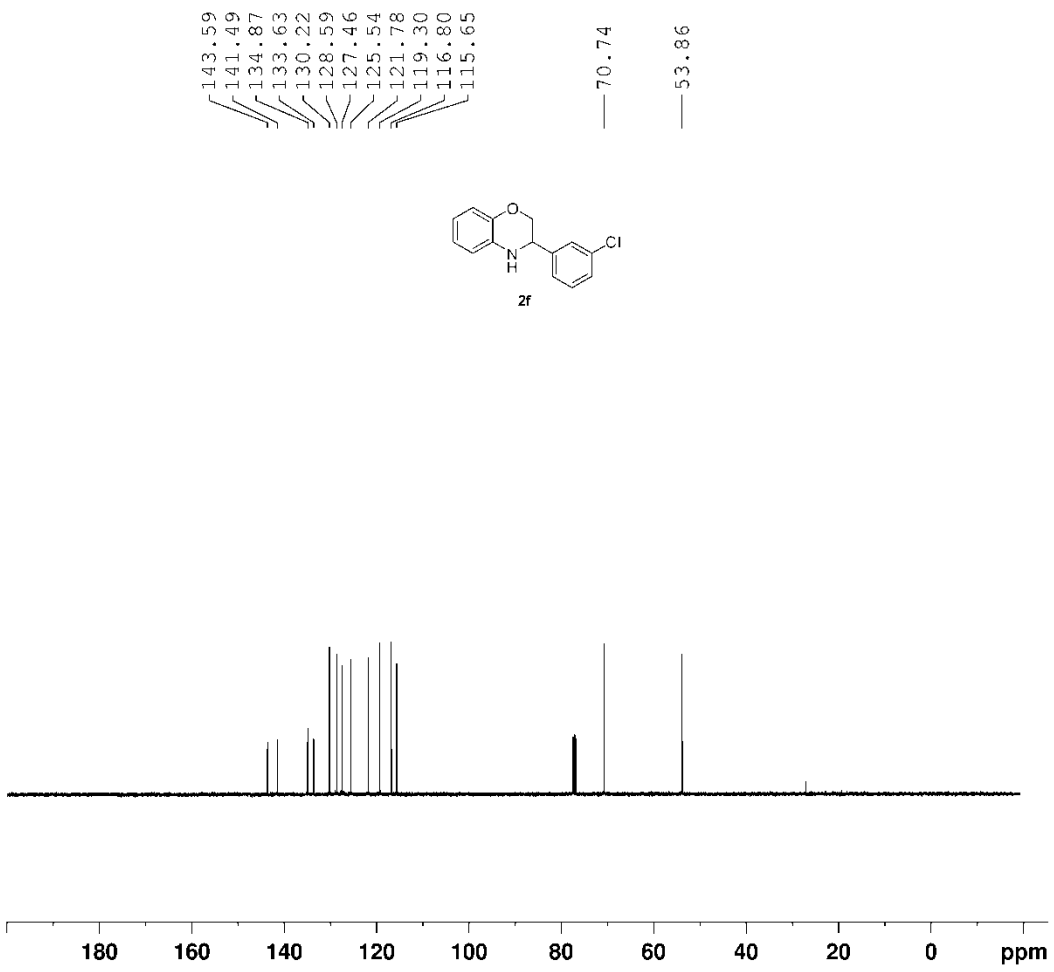

```
NAME            wsm-2-C-P
EXPNO                1452
PROCNO                  1
Date_            20151127
Time                10.34
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     108
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                   206.33
DW                  20.800 usec
DE                   6.50 usec
TE                   298.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

-------- CHANNEL f1 --------
SFO1           100.6304916 MHz
NUC1                    13C
P1                   10.00 usec
SI                    32768
SF             100.6404207 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



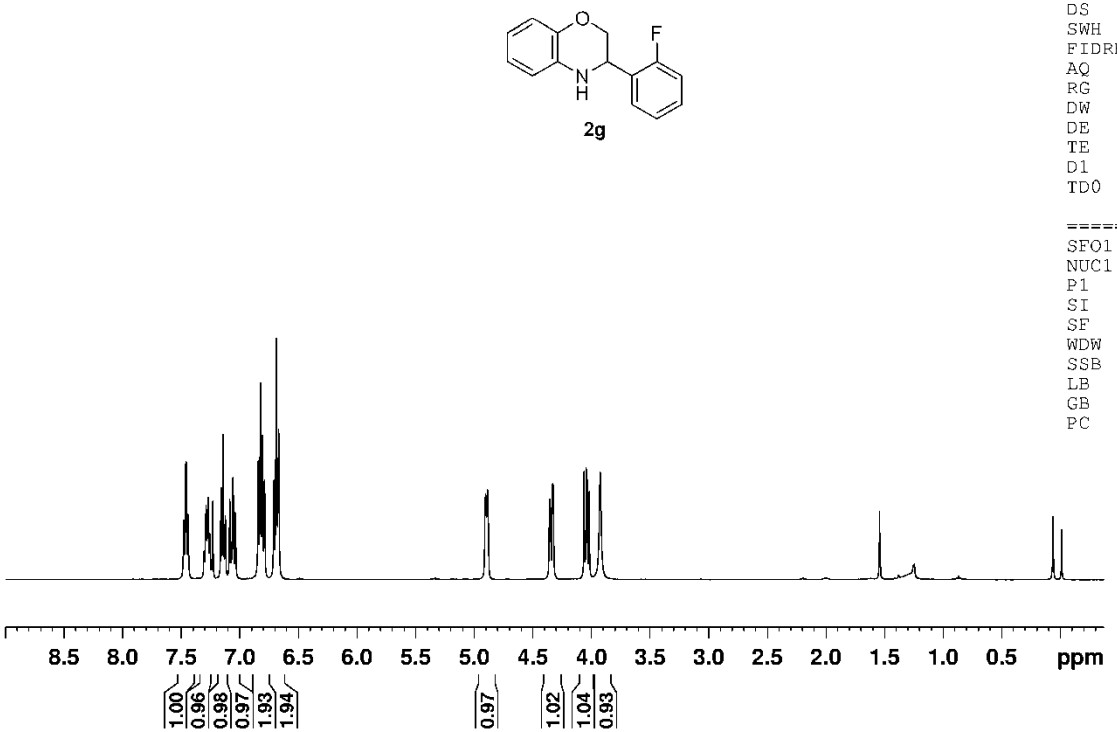
2f



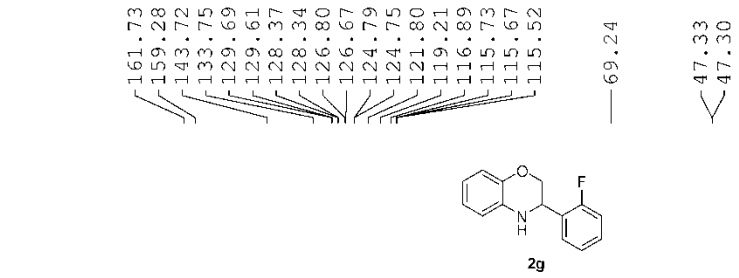
NAME	wsm-2-H-P	
EXPNO	1457	
PROCNO	1	
Date_	20151127	
Time	14.59	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	125.02	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400094	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

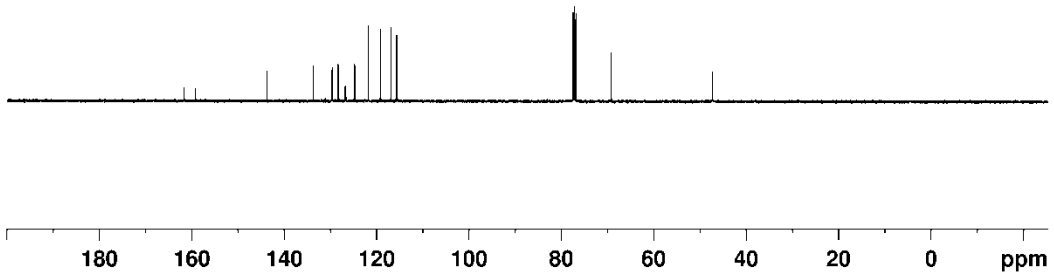



```
NAME            wsm-2-C-P
EXPNO                1457
PROCNO                  1
Date_            20151127
Time                10.55
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      30
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                   206.33
DW                  20.800 usec
DE                   6.50 usec
TE                   298.6 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6304916 MHz
NUC1                   13C
P1                   10.00 usec
SI                   32768
SF             100.6404209 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

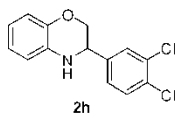


NAME	wsm-2-H-P
EXPNO	1465
PROCNO	2
Date_	20151210
Time	13.52
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	12
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	41.600 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300207 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00





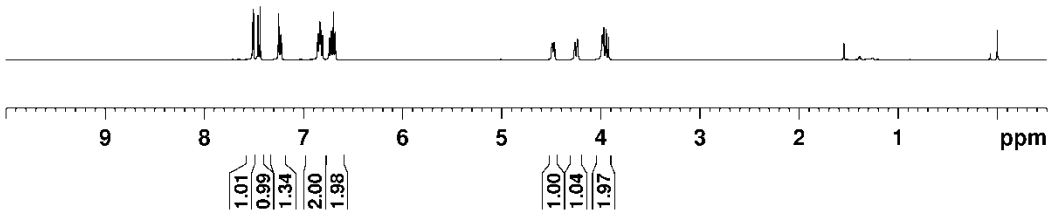
NAME	wsm-2-C-P
EXPNO	1446
PROCNO	1
Date_	20151209
Time	7.36
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	82
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	114
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127564 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

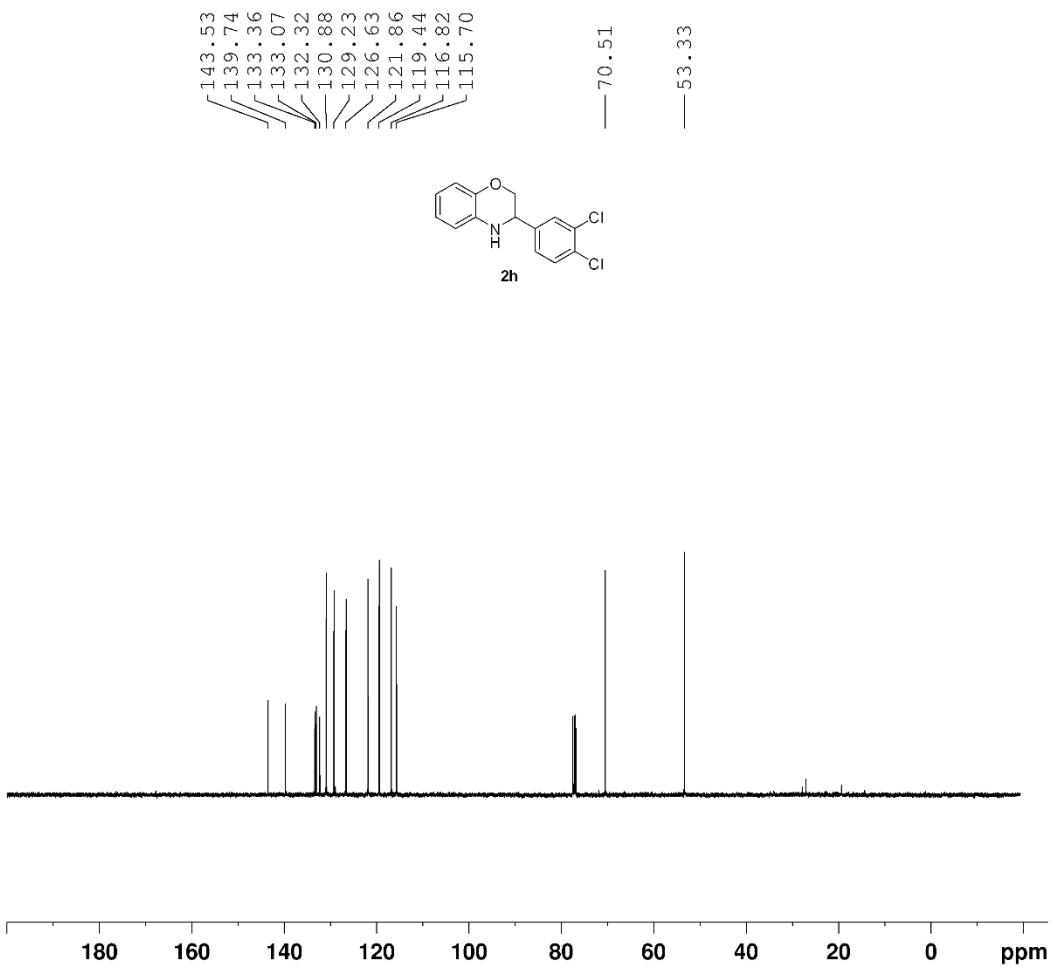


2h

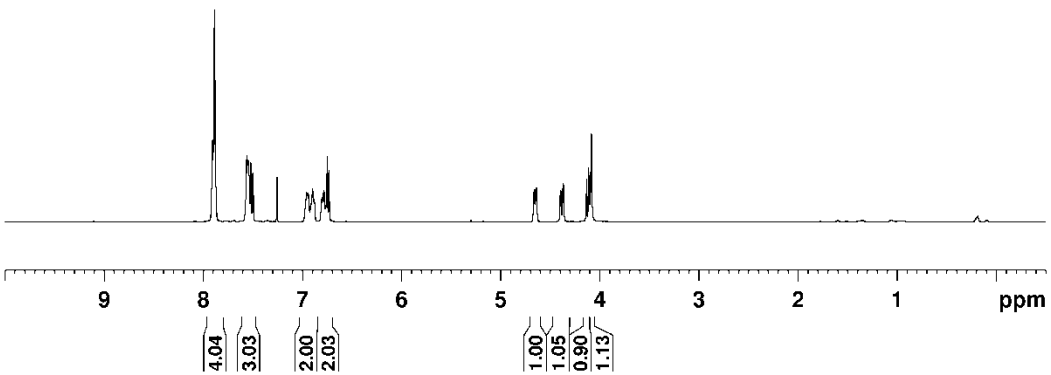
NAME	wsm-2-H-P
EXPNO	1464
PROCNO	1
Date_	20151215
Time	7.37
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	41.600 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1

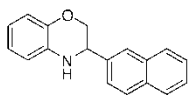
======== CHANNEL f1 ========	
SFO1	400.1320007 MHz
NUC1	1H
P1	12.60 usec
SI	65536
SF	400.1300123 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00





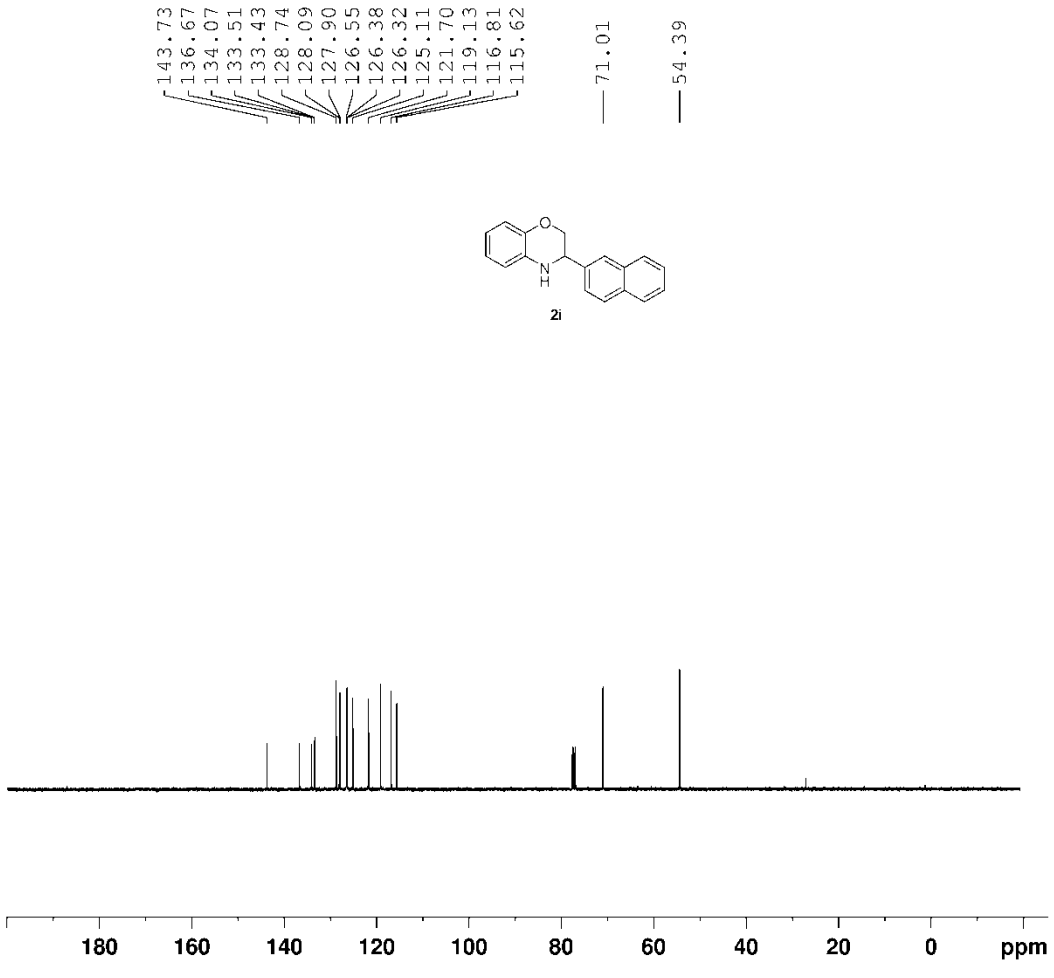
NAME	wsm-2-C-P
EXPNO	1444
PROCNO	1
Date_	20151123
Time	16.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	63
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	206.33
DW	20.800 usec
DE	6.50 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6504916 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6404219 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



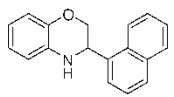


2i

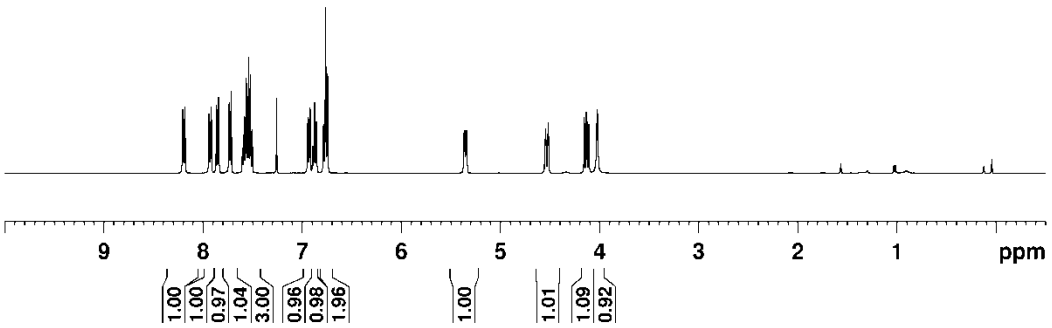
NAME	wsm-2-H-P	
EXPNO	1459	
PROCNO	1	
Date_	20151127	
Time	15.03	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	140.59	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400095	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



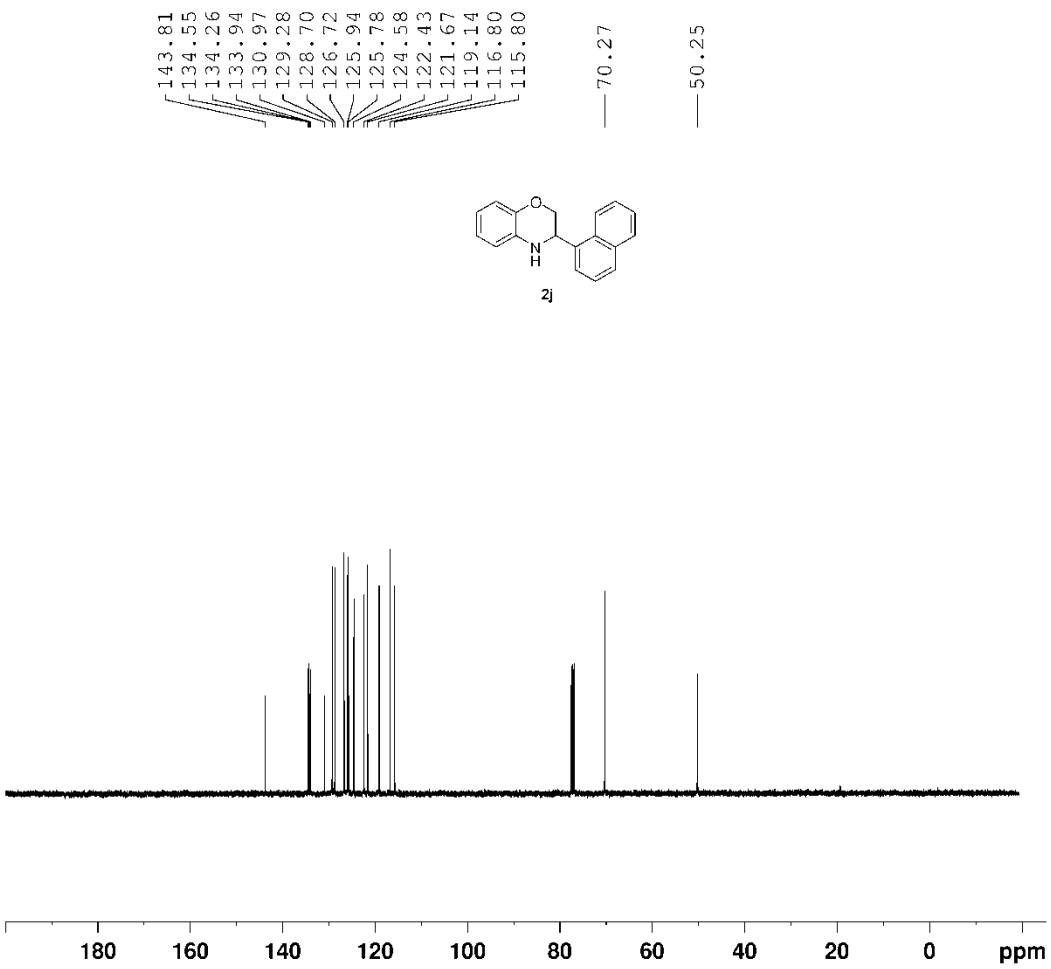
NAME	wsm-2-C-P
EXPNO	1459
PROCNO	1
Date_	20151127
Time	11.00
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	28
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	206.33
DW	20.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6304916 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6404244 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



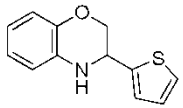
2j



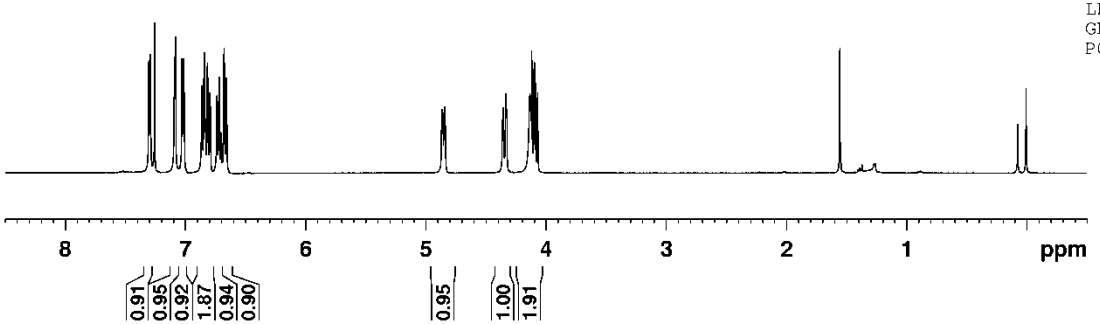
NAME	wsm-2-H-P	
EXPNO	1443	
PROCNO	2	
Date_	20151127	
Time	15.07	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	11	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	102.73	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400092	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



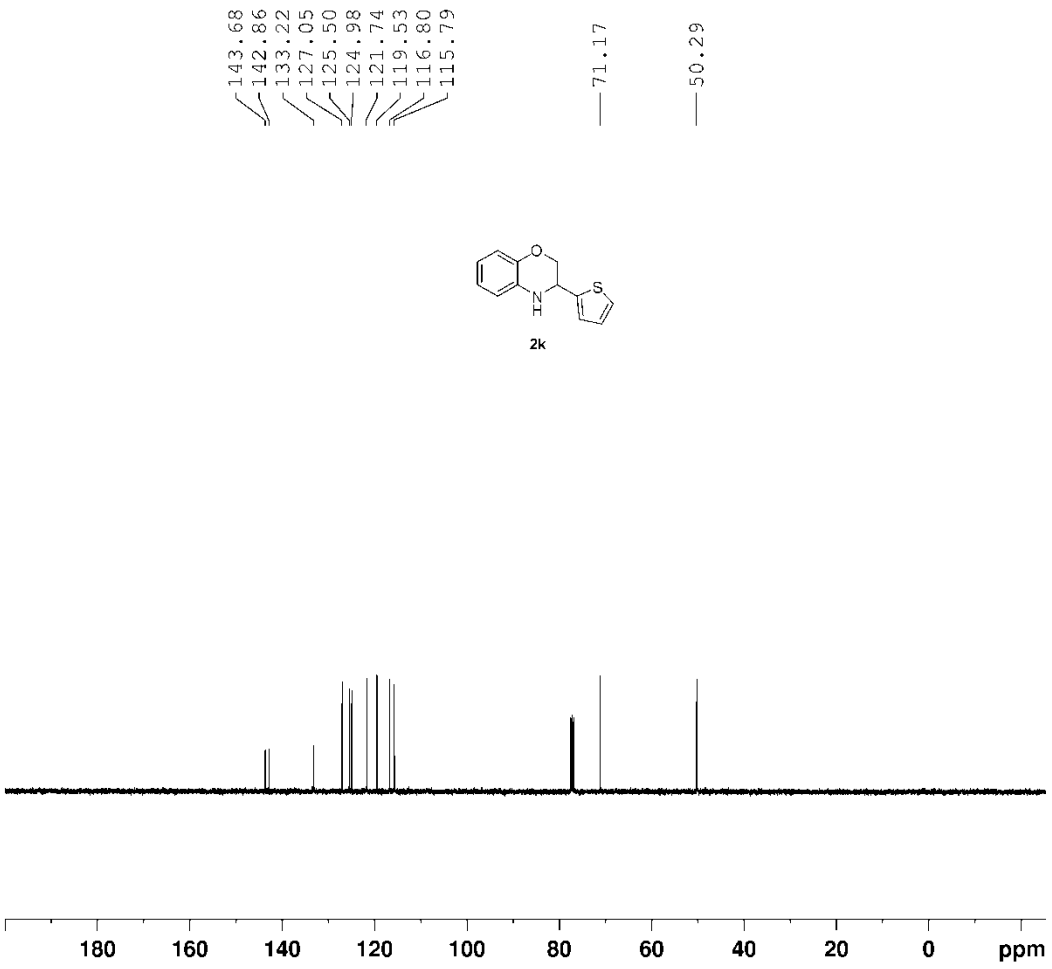
NAME	wsm-2-C-P
EXPNO	1443
PROCNO	1
Date_	20151127
Time	10.14
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	31
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	206.33
DW	20.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6304916 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6404282 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



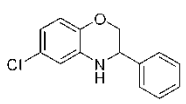
2k



NAME	wsm-2-H-P
EXPNO	1466
PROCNO	3
Date_	20151210
Time	13.56
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 se
RG	203
DW	41.600 us
DE	6.50 us
TE	300.0 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
SFO1	400.1320007 MH
NUC1	1H
P1	12.60 us
SI	65536
SF	400.1300099 MH
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00




```
NAME           wsm-2-C-P
EXPNO               1442
PROCNO                 1
Date_           20151209
Time                7.25
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    26
DS                     0
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ             0.6488564 sec
RG                   114
DW                19.800 usec
DE                 6.50 usec
TE                 300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6228298 MHz
NUC1                  13C
P1                  9.40 usec
SI                 32768
SF          100.6127627 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



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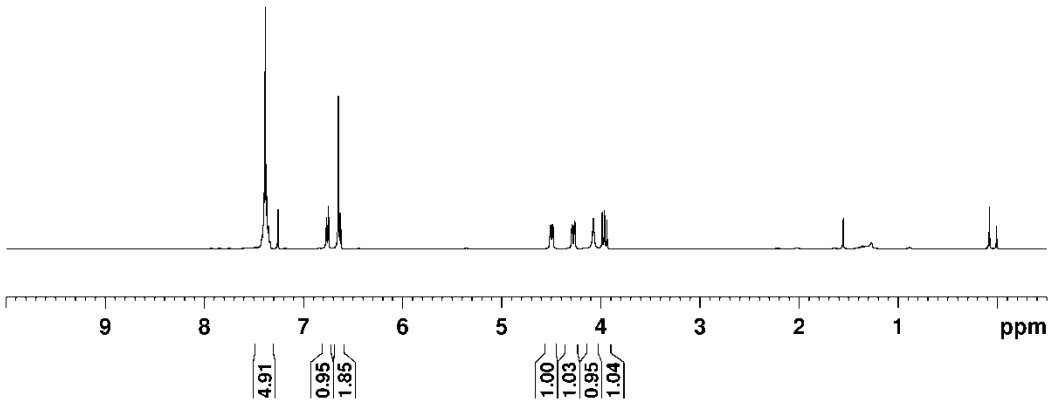
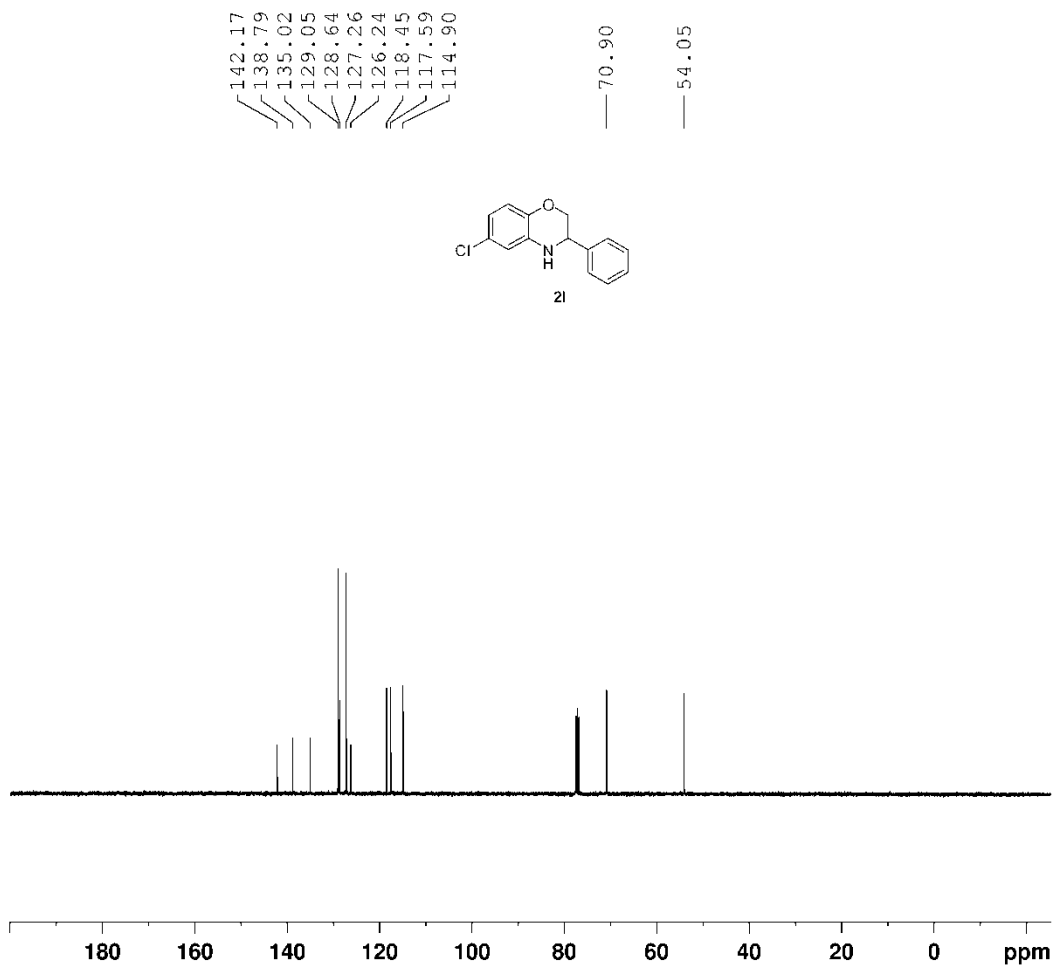
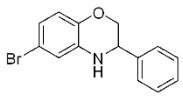


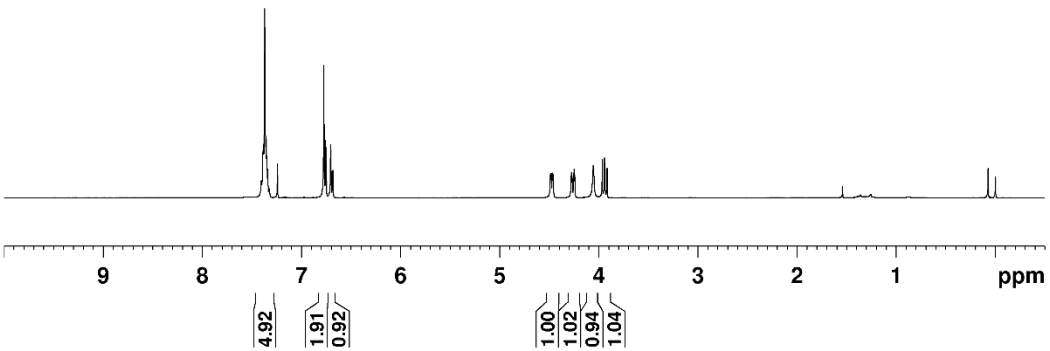
NAME	wsm-2-H-P	
EXPNO	1467	
PROCNO	2	
Date_	20151210	
Time	13.59	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300098	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



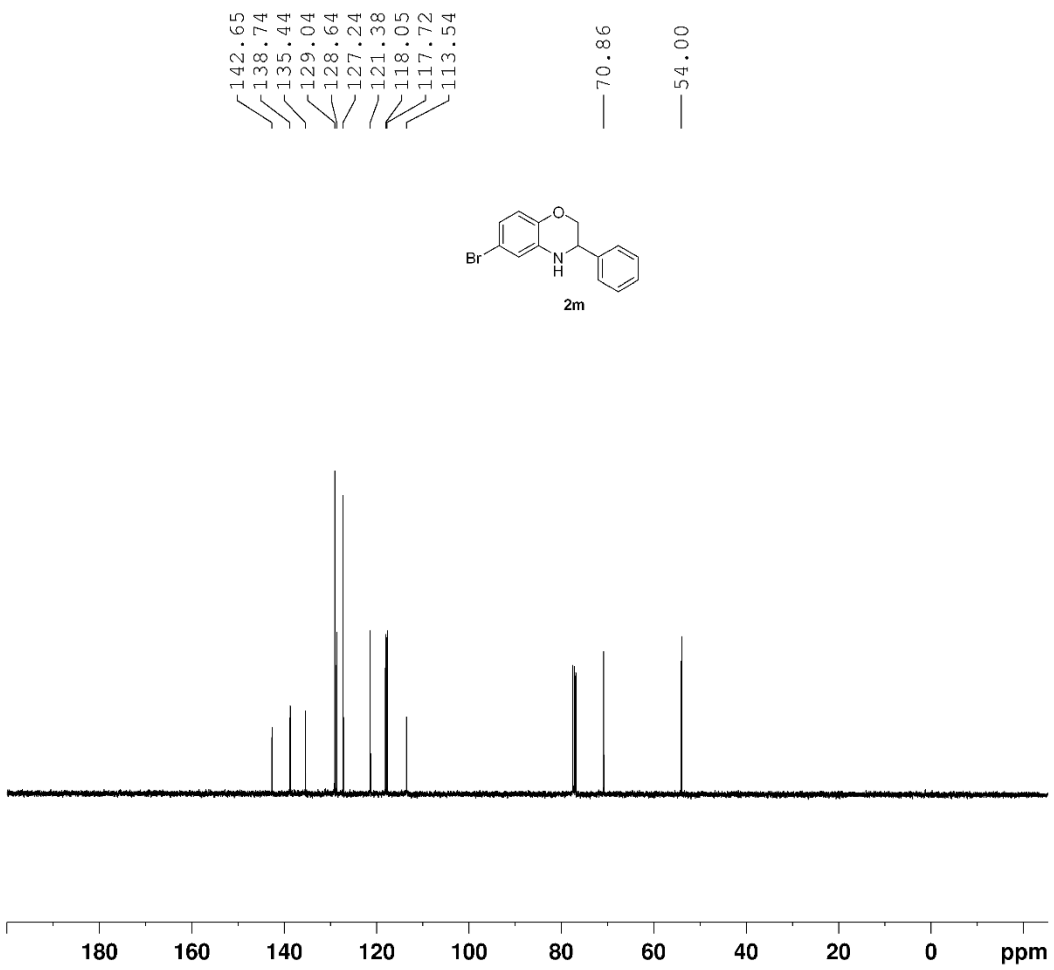

```
NAME            wsm-2-C-P
EXPNO                1447
PROCNO                  1
Date_            20151209
Time                 7.43
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG              zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      38
DS                       0
SWH              25252.525 Hz
FIDRES            0.770646 Hz
AQ                0.6488564 sec
RG                     128
DW                  19.800 usec
DE                   6.50 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                    13C
P1                    9.40 usec
SI                   32768
SF              100.6127609 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



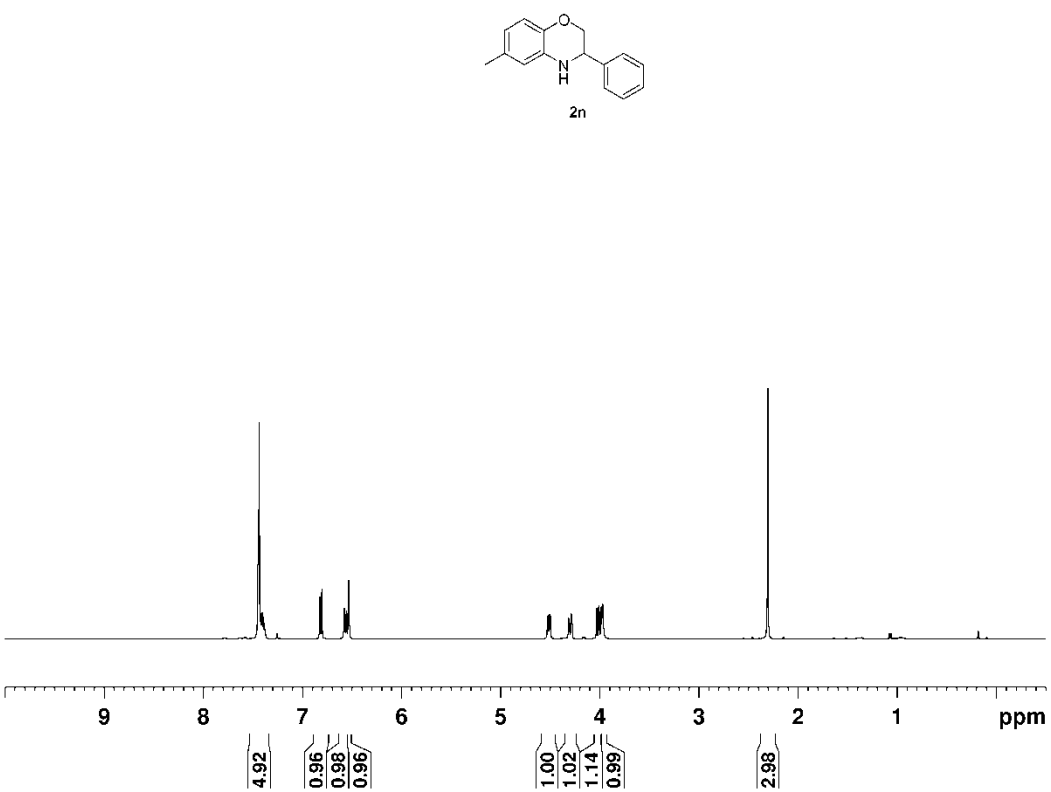
2m



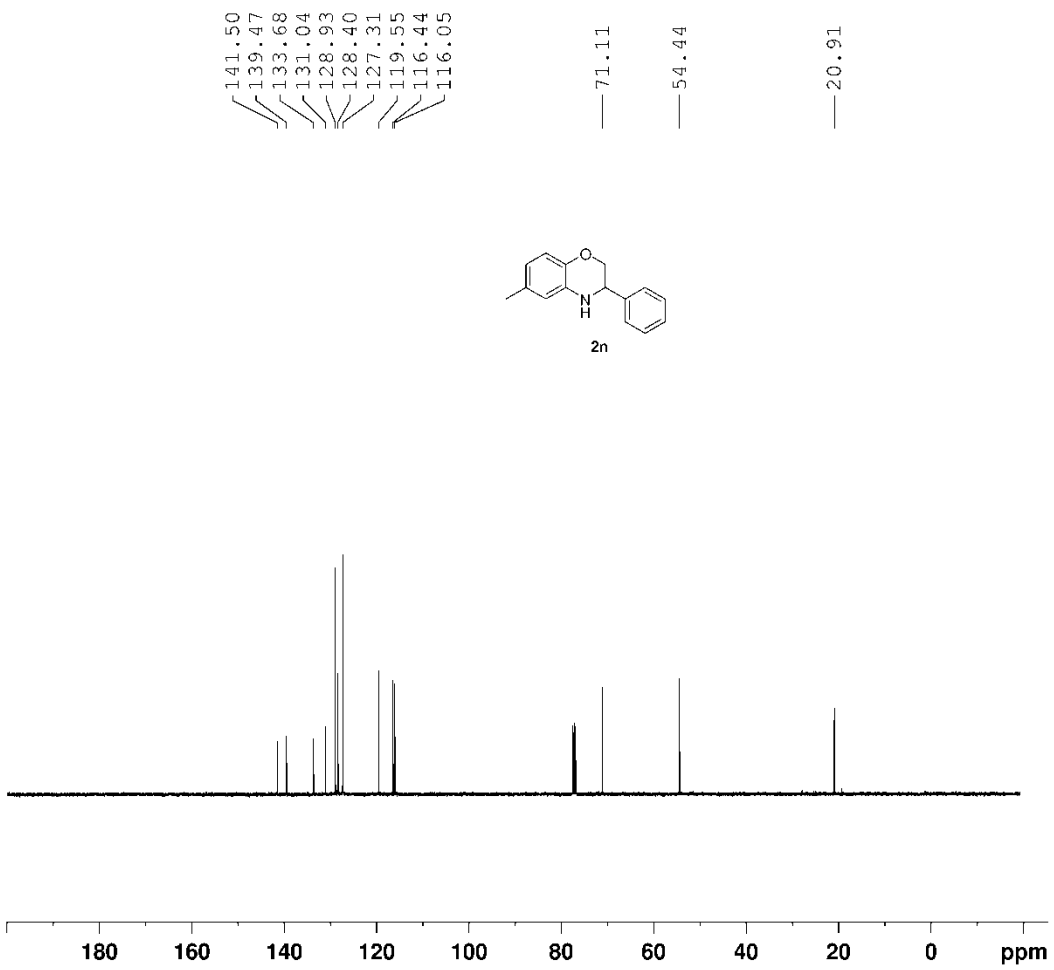
NAME	wsm-2-H-P	
EXPNO	1468	
PROCNO	1	
Date_	20151215	
Time	7.40	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	161	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300160	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



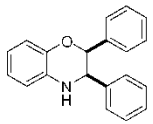
NAME	wsm-2-C-P
EXPNO	1448
PROCNO	1
Date_	20151209
Time	7.46
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	36
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	203
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127625 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



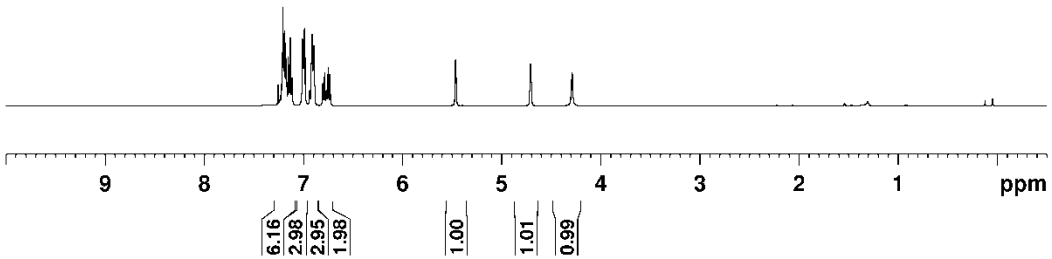
NAME	wsm-2-H-P	
EXPNO	1453	
PROCNO	1	
Date_	20151127	
Time	14.50	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	8012.820	Hz
FIDRES	0.244532	Hz
AQ	2.0447731	sec
RG	80.72	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.2424716	MHz
NUC1	1H	
P1	14.80	usec
SI	65536	
SF	400.2400093	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



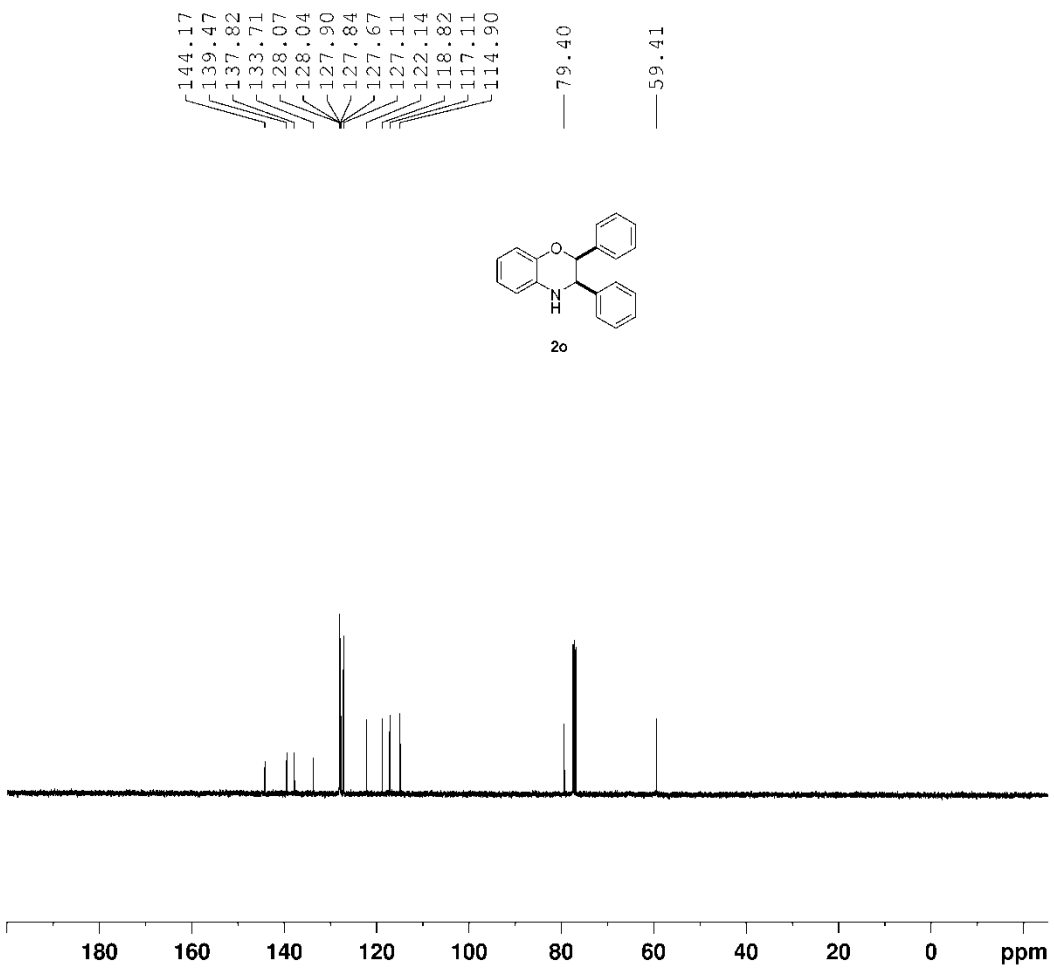

```
NAME           wsm-2-C-P
EXPNO               1453
PROCNO                 2
Date_           20151127
Time               10.44
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    35
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        100.6304916 MHz
NUC1               13C
P1                10.00 usec
SI                32768
SF          100.6404229 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



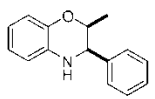
2o



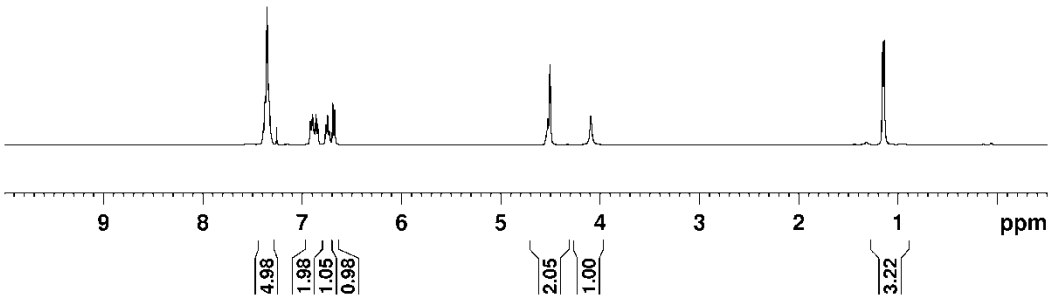
NAME	wsm-2-H-P	
EXPNO	1474	
PROCNO	2	
Date_	20160105	
Time	7.30	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	13	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	101	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300100	MHz
WDW	EM	
SSB	0	
LB	0.50	Hz
GB	0	
PC	1.00	



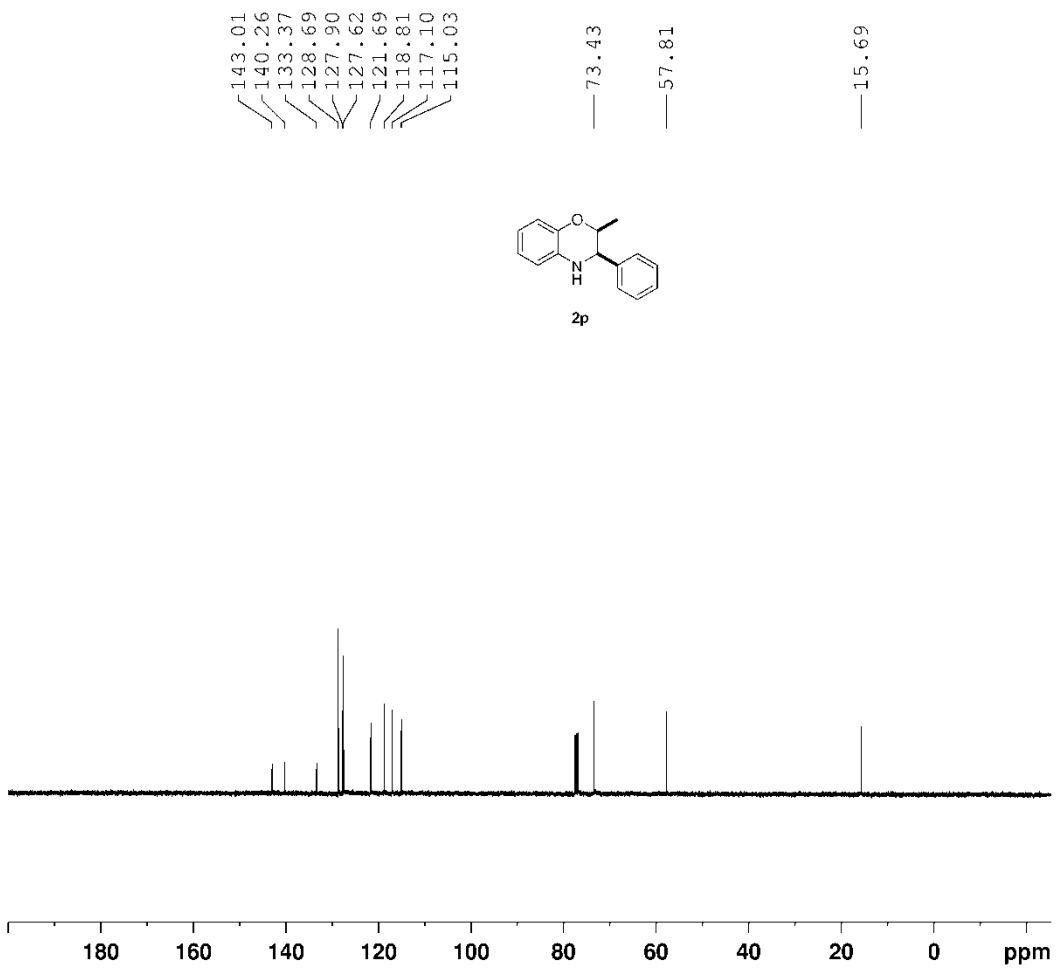
NAME	wsm-2-C-P
EXPNO	1474
PROCNO	1
Date_	20160105
Time	7.31
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	56
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	161
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127556 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



2p



NAME	wsm-2-K-P	
EXPNO	1487	
PROCNO	1	
Date_	20160107	
Time	11.34	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	0	
SWH	12019.230	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	57	
DW	41.600	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	400.1320007	MHz
NUC1	1H	
P1	12.60	usec
SI	65536	
SF	400.1300099	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



NAME	wsm-2-C-P
EXPNO	1487
PROCNO	1
Date_	20160107
Time	11.36
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	38
DS	0
SWH	25252.525 Hz
FIDRES	0.770646 Hz
AQ	0.6488564 sec
RG	161
DW	19.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
-------- CHANNEL f1 --------	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.40 usec
SI	32768
SF	100.6127586 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40