Supporting information

Highly Efficient Ir-Catalyzed Asymmetric Hydrogenation of Benzoxazinones and Derivatives with a Brønsted Acid Cocatalyst

Zhengyu Han,[†] Gang Liu,[†] Rui Wang,[†] Xiu-Qin Dong,*[†] and Xumu Zhang*[§,†]

[†] Key Laboratory of Biomedical Polymers, Engineering Research Center of Organosilicon Compounds & Materials, Ministry of Education, College of Chemistry and Molecular Sciences, Wuhan University, Wuhan, Hubei, 430072, P. R. China.

[§] Department of Chemistry and Shenzhen Grubbs Institute, Southern University of Science and Technology, Shenzhen, Guangdong, 518055, P. R. China.

E-mail: zhangxm@sustc.edu.cn, xiuqindong@whu.edu.cn.

Content

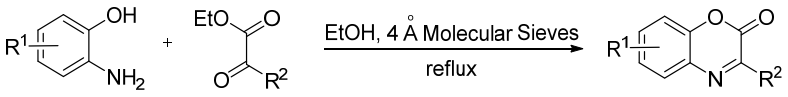
I. General Remarks

All the reactions with air- or moisture-sensitive compounds were carried out in a dry reaction vessel under a positive pressure of nitrogen or in the argon-filled glovebox. Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers without further purification. Anhydrous solvents were purchased from J&K Chemical Technology company and transferred by syringe. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker ADVANCE III (400 MHz) spectrometer with $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to the $CDCl_3$ at 7.26 ppm (for ^{1}H NMR) or 77.0 ppm (for ^{13}C NMR). Data are reported as: multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant in hertz (Hz) and signal area integration in natural numbers. ^{13}C NMR and ^{31}P NMR analyses were run with decoupling. Enantiomeric excess values were determined by Daicel chiral column on an Agilent 1260 Series HPLC instrument. Optical rotations $[\alpha]_D$ were measured on a PERKIN ELMER polarimeter 343 instrument.

All the benzoxazinone or quinoxalinones were prepared according the literature.[1-7] The absolute configuration of products **2a**, **2c**, **2e-2h**, **2j**, **2m**, **4a** and **4c** were determined by comparison of analytical data with the literature (HPLC spectra, optical rotation).[3, 5, 8-9] The absolute configuration of others were assigned by analogy.

II. General Procedure for the Synthesis of Substrates

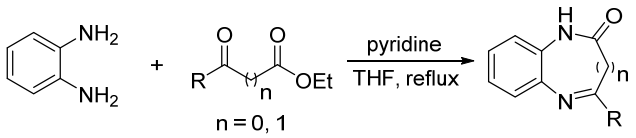
A) Preparation of substrates 1a-1p [1]



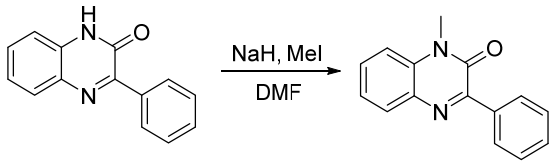
To the solution of appropriate 2-aminophenol derivative (10.0 mmol, 1.0 eq.) and the corresponding α-ketoester (11.0 mmol, 1.1 eq.) in dry ethanol (20 mL) 4Å molecular sieves (2.0 g) was added and the resulting mixture was refluxed for 12 h. Then the solution was cooled to room temperature and passed through a pad of celite, and concentrated in vacuo. The crude product was purified by column chromatography using petroleum ether/EtOAc (20:1) mixtures as the eluent. If

required, the isolated product after chromatography was further recrystallized from EtOH to obtain the corresponding benzoxazinones.

B) Preparation of substrates 3a-3d [1]

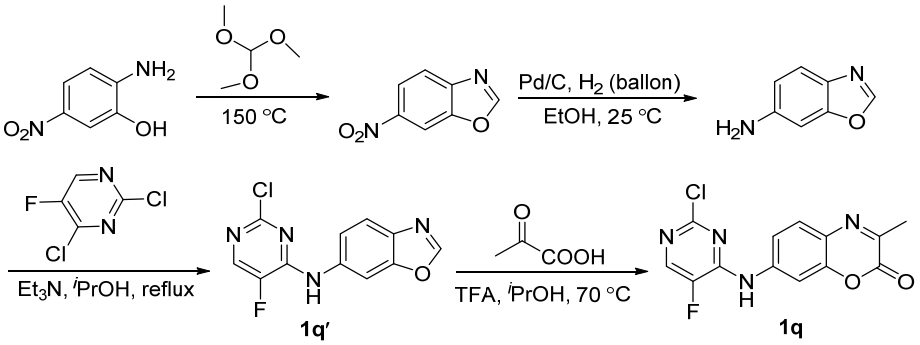


To the solution of 1,2-diaminobenzene (10.0 mmol, 1.0 eq.) in THF (20 mL) the appropriate α-ketoester or ethyl benzoylacetate (10 mmol, 1.0 eq.) was added followed with pyridine (2 mL), the resulting mixture was refluxed for 12 h. Then the solution was cooled to room temperature and concentrated in vacuo. The crude product was purified by column chromatography using petroleum ether/EtOAc (20:1) mixtures as the eluent. If required, the isolated product after chromatography was further recrystallized from EtOH to obtain the corresponding quinoxalinones or 4-phenyl-1H-benzo[*b*][1,4]diazepin-2(3H)-one.



Dissolve the 3-phenylquinoxaline-2(1H)-one (2.0 mmol, 1.0 eq.) in DMF (15 mL) and NaH (6.0 mmol, 3.0 eq.) was added slowly at 0 °C, methyl iodide (6.0 mmol) was then added. The resulting mixture was stirred at 0 °C for another 2 h, and then quenched with water and extracted with EtOAc. The organic layer was washed with saturated $NaHCO_3$ and brine and then dried over Na_2SO_4. The solvent was removed in vacuo to give a solid that was purified by column chromatography using petroleum ether/EtOAc (20:1) mixtures as the eluent to give the corresponding N-methylated 3-substituted quinoxalin-2(1H)-one.

C) Preparation of substrate 1q [2]



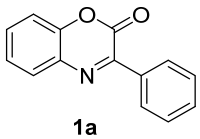
2-amino-5-nitro-phenol (50.0 mmol) was dissolved in trimethylorthoformate (25 mL) and stirred at 150 °C for 12 h. Then the solution was cooled to room temperature and concentrated in vacuo. The crude product was purified by column chromatography using petroleum ether/EtOAc (10:1) mixtures as the eluent to get the desired 6-nitrobenzo[*d*]oxazole.

A solution of 6-nitrobenzo[*d*]oxazole (50.0 mmol) in EtOH (25 mL) was added Pd/C (1.6 g) and stirred under H_2 atmosphere (ballon) at 25 °C for 12 h, then the reaction mixture passed through a column of silica gel to remove the Pd/C and the solution was concentrated under vacuo to get the benzo[*d*]oxazol-6-amine.

2,4-dichloro-5-fluoro-pyrimidine (55.0 mmol) and benzo[*d*]oxazol-6-amine (50.0 mmol) was dissolved in ^{i}PrOH (50 mL) and Et_3N (75 mmol), the resulting mixture was allowed heated to reflux for 12 h. Then the reaction mixture was concentrated under vacuo and purified by column chromatography to get the desired product **1q'**.

1q' (4 mmol) and pyruvic acid (6 mmol) was dissolved in ^{i}PrOH (5 mL) and TFA (0.8 mmol) was added slowly. The resulting mixture was heated to 70 °C and stirred for 18 h. Then the solution was cooled to room temperature and washed with $NaHCO_3$ (aqueous) then extracted with EA (2×20 mL). The organic layer was concentrated under vacuo and purified by column chromatography to get the desired product **1q**.

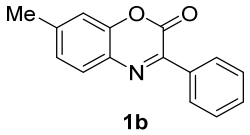
3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1a)



1a

Light yellow solid, 77% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.34-8.32 (m, 2H), 7.87-7.85 (m, 1H), 7.57-7.48 (m, 4H), 7.42-7.38 (m, 1H), 7.35-7.33 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.3, 150.9, 146.4, 134.1, 131.6, 131.4, 131.1, 129.44, 129.41, 128.4, 125.6, 116.2. The characterization data of compound **1a** is in accordance with the reported data in the literature.[2a]

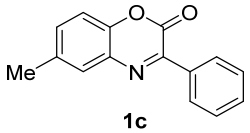
7-methyl-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1b)



1b

Yellow solid, mp = 147-149 °C, 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.32-8.30 (m, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.53-7.47 (m, 3H), 7.22-7.20 (m, 1H) , 7.14 (s, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.5, 149.6, 146.3, 142.5, 134.3, 131.1, 129.7, 129.3, 129.0, 128.3, 126.7, 116.2, 21.8. The characterization data of compound **1b** is in accordance with the reported data in the literature.[1, 2a]

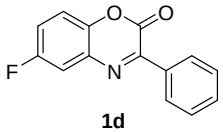
6-methyl-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1c)



1c

Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.32-8.30 (m, 2H), 7.62 (s, 1H), 7.54-7.46 (m, 3H), 7.31-7.28 (m, 1H), 7.20-7.18 (m, 1H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.4, 150.6, 144.3, 135.4, 134.2, 132.1, 131.28, 131.25, 129.3, 129.2, 128.3, 115.7, 20.8. The characterization data of compound **1c** is in accordance with the reported data in the literature.[2a]

6-fluoro-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1d)

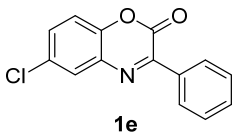


1d

Yellow solid, mp = 143-145 °C, 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.36-8.33 (m, 2H), 7.58-7.49 (m, 4H), 7.34-7.30 (m, 1H), 7.28-7.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ

(ppm) 159.4 (d, *J* = 244.0 Hz), 151.9 (d, *J* = 11.0 Hz), 142.8 (d, *J* = 3.0 Hz), 133.7, 132.0 (d, *J* = 12.0 Hz), 131.8, 129.6, 128.5, 118.5 (d, *J* = 25.0 Hz), 117.2 (d, *J* = 9.0 Hz), 115.0 (d, *J* = 24.0 Hz). HRMS (ESI): [M+H$^+$] Calc. 242.0617, found 242.0611.

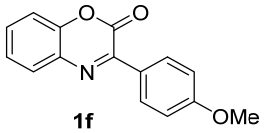
6-chloro-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1e)



1e

Light yellow solid, mp = 149-151 °C, 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.34-8.32 (m, 2H), 7.85 (s, 1H), 7.58-7.46 (m, 4H), 7.29-7.27 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 151.74, 151.68, 145.0, 133.7, 132.1, 131.9, 131.0, 130.7, 129.6, 128.8, 128.5, 117.3. The characterization data of compound **1e** is in accordance with the reported data in the literature. [2a]

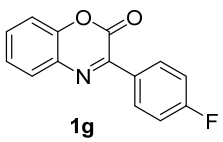
3-(4-methoxyphenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (1f)



1f

Yellow solid, mp = 146-149 °C, 66% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.41-8.39 (m, 2H), 7.84-7.81 (m, 1H), 7.49-7.47 (m, 1H), 7.38-7.36 (m, 1H), 7.34-7.31 (m, 1H), 7.02-7.00 (m, 2H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 162.3, 152.5, 149.8, 146.2, 131.7, 131.4, 130.4, 129.1, 126.8, 125.5, 116.0, 113.8, 55.4. The characterization data of compound **1f** is in accordance with the reported data in the literature.[2a]

3-(4-fluorophenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (1g)

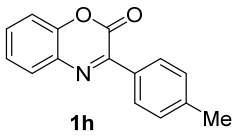


1g

Light brown solid, mp = 166-169 °C, 79% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.43-8.40 (m, 2H), 7.86-7.83 (m, 1H), 7.55-7.51 (m, 1H), 7.43-7.39 (m, 1H), 7.36-7.33 (m, 1H), 7.21-7.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 164.8 (d, *J* = 252.0 Hz), 152.3, 149.5, 146.4,

131.9, 131.8, 131.5, 131.2, 129.4, 125.7, 116.2, 115.5 (d, J = 22.0 Hz). The characterization data of compound **1g** is in accordance with the reported data in the literature.[2a]

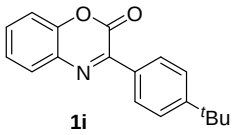
3-(*p*-tolyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (1h)



1h

Yellow solid, 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.26 (d, J = 8.0 Hz, 2H), 7.85-7.83 (m, 1H), 7.52-7.48 (m, 1H), 7.41-7.37 (m, 1H), 7.34-7.30 (m, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.4, 150.7, 146.4, 142.0, 131.7, 131.4, 130.8, 129.4, 129.3, 129.1, 125.5, 116.1, 21.6. The characterization data of compound **1h** is in accordance with the reported data in the literature.[1, 2a]

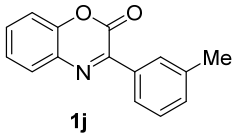
3-(4-(tert-butyl)phenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (1i)



1i

Light yellow solid, mp = 141-144 °C, 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.28-8.26 (m, 2H), 7.86-7.83 (m, 1H), 7.54-7.48 (m, 3H), 7.41-7.32 (m, 2H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 155.0, 152.4, 150.8, 146.4, 131.7, 131.4, 130.8, 129.3, 129.2, 125.5, 125.4, 116.1, 35.0, 31.1.

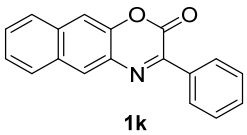
3-(*m*-tolyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (1j)



1j

Yellow solid, mp = 93-96 °C, 53% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.12 (d, J = 8.0 Hz, 2H), 7.87-7.85 (m, 1H), 7.54-7.49 (m, 1H), 7.41-7.32 (m, 4H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.3, 151.1, 146.4, 138.1, 134.0, 132.3, 131.6, 131.0, 129.8, 129.4, 128.3, 126.7, 125.5, 116.1, 21.5. HRMS (ESI): [M+H$^+$] Calc. 238.0868, found 238.0860.

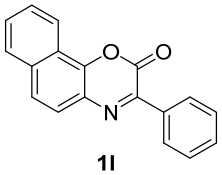
3-phenyl-2*H*-naphtho[2,3-*b*][1,4]oxazin-2-one (1k)



1k

Deep yellow solid, 69% yield; known compound;[4] ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.38-8.35 (m, 3H), 8.00 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.70 (s, 1H), 7.62-7.50 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.2, 151.1, 144.2, 134.2, 133.9, 131.5, 130.9, 130.8, 129.5, 129.2, 128.9, 128.5, 128.4, 127.4, 126.0, 112.3. The characterization data of compound **1k** is in accordance with the reported data in the literature.[4]

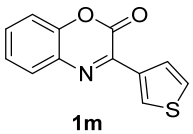
3-phenyl-2*H*-naphtho[1,2-b][1,4]oxazin-2-one (1l)



1l

Deep yellow solid, mp = 157-160 °C, 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.50-8.48 (m, 1H), 8.43-8.41 (m, 2H), 7.92-7.89 (m, 1H), 7.85-7.77 (m, 2H), 7.68-7.63 (m, 2H), 7.56-7.50 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.4, 150.0, 142.6, 134.3, 134.2, 131.3, 129.3, 128.7, 128.4, 128.0, 127.9, 127.3, 125.6, 125.5, 122.5, 122.1.

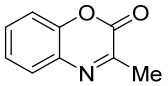
3-(thiophen-3-yl)-2*H*-benzo[*b*][1,4]oxazin-2-one (1m)



1m

Wheat solid, mp = 123-126 °C, 43% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.76 (brs, 1H), 8.00-7.98 (m, 1H), 7.79-7.77 (m, 1H), 7.49-7.45 (m, 1H), 7.38-7.34 (m, 2H), 7.31-7.26 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 152.1, 145.90, 145.87, 136.1, 132.2, 131.5, 130.6, 129.1, 127.8, 125.52, 125.48, 116.1. HRMS (ESI): [M+H^{+}] Calc. 230.0276, found 230.0269.

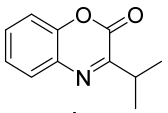
3-methyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1n)



1n

Brown solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.72-7.70 (m, 1H), 7.50-7.46 (m, 1H), 7.38-7.34 (m, 1H), 7.30-7.28 (m, 1H), 2.58 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 155.1, 153.2, 146.5, 131.0, 130.5, 128.6, 125.4, 116.4, 21.3. The characterization data of compound **1n** is in accordance with the reported data in the literature.[2a]

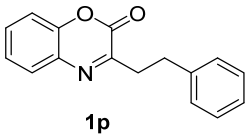
3-isopropyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1o)



1o

White solid; mp = 37-39 °C, 82% yield; known compound;[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.9 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.28- 7.26 (m, 1H), 3.46 (hept, *J* = 6.7 Hz, 1H), 1.33 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.9, 152.5, 146.2, 131.2, 130.3, 128.9, 125.2, 116.2, 31.9, 19.8.

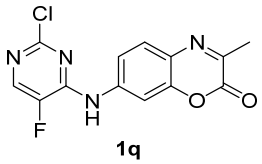
3-phenethyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1p)



1p

Yellow solid; mp = 108-111 °C, 71% yield; known compound;[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.50-7.46 (m, 1H), 7.39-7.35 (m, 1H), 7.31-7.28 (m, 5H), 7.23-7.20 (m, 1H), 3.25-3.20 (m, 2H), 3.16-3.11 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 152.9, 146.4, 140.7, 131.1, 130.6, 128.8, 128.51, 128.47, 126.2, 125.4, 116.4, 35.8, 32.1.

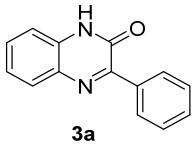
7-((2-chloro-5-fluoropyrimidin-4-yl)amino)-3-methyl-2*H*-benzo[*b*][1,4]oxazin-2-one (1q)



1q

Yellow solid, mp >300 °C, 35% total yield; ^{1}H NMR (400 MHz, C_2D_6SO): δ (ppm) 10.35 (s, 1H), 8.43 (d, *J* = 4.0 Hz, 1H), 7.88 (d, *J* = 4.0 Hz, 1H), 7.72-7.65 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, C_2D_6SO): δ (ppm) 153.7 (d, *J* = 48.0 Hz), 153.1 (d, *J* = 4.0 Hz), 151.0 (d, *J* = 12.0 Hz), 147.1, 147.0, 144.5, 142.8 (d, *J* = 21.0 Hz), 140.1, 128.6, 127.6, 117.9, 107.7, 21.4. HRMS (ESI): [M+H$^+$] Calc. 307.0398, found 307.0391.

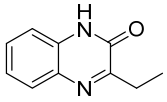
3-phenylquinoxalin-2(1*H*)-one (3a)



3a

Light yellow solid, 84% yield; known compound;[5] ^{1}H NMR (400 MHz, C_2D_6SO): δ (ppm) 12.60 (s, 1H), 8.32-8.29 (m, 2H), 7.84 (t, *J* = 4.0 Hz, 1H), 7.57-7.48 (m, 4H), 7.35-7.31 (m, 2H); ^{13}C NMR (100 MHz, C_2D_6SO): δ (ppm) 155.1, 154.6, 136.1, 132.52, 132.48, 130.8, 130.7, 129.7, 129.3, 128.3, 123.9, 115.6.

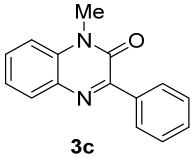
3-ethylquinoxalin-2(1*H*)-one (3b)



3b

Light yellow solid, 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 12.40 (s, 1H), 7.85-7.83 (m, 1H), 7.51-7.47 (m, 1H), 7.38-7.32 (m, 2H), 3.03 (q, *J* = 8.0 Hz, 2H), 1.39 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 162.5, 156.5, 132.8, 130.9, 129.6, 128.7, 124.1, 115.6, 26.8, 10.9.

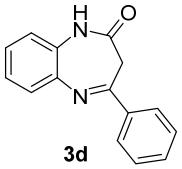
1-methyl-3-phenylquinoxalin-2(1*H*)-one (3c)



3c

Yellow white solid; 57% yield; known compound;[5] ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.31-8.28 (m, 2H), 7.96-7.94 (m, 1H), 7.60-7.54 (m, 1H), 7.50-7.47 (m, 3H), 7.38-7.33 (m, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 154.7, 154.2, 136.1, 133.3, 133.1, 130.4, 130.3, 129.5, 128.1, 123.7, 113.6, 29.3.

4-phenyl-1,3-dihydro-2*H*-benzo[*b*][1,4]diazepin-2-one (3d)



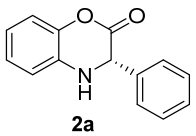
3d

White solid; 81% yield; known compound;[7] ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.88 (s, 1H), 8.12-8.10 (m, 2H), 7.52-7.47 (m, 4H), 7.28-7.24 (m, 2H), 7.12-7.10 (m, 1H), 3.58 (s, 2H);^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 167.7, 158.8, 140.0, 137.6, 131.0, 129.0, 128.7, 128.3, 127.7, 126.5, 125.1, 121.8, 39.8.

III. General Procedure for Asymmetric Hydrogenation

In the argon-filled glovebox, a solution of **L5** (4.9 mg, 0.0055 mmol) and $[Ir(COD)Cl]_2$ (1.7 mg, 0.0025 mmol) in 1.0 mL anhydrous THF was stirred at room temperature for 45 min. 10 μL of the resulting solution transferred by syringe into a vial charged with **1** (0.05 mmol) in 1.0 mL anhydrous THF, then a solution of HCl (1.0 eq.) in dioxane (4 M) was added via syringe. The vials were transferred to an autoclave, which was then charged with 30 atm of H_2 and stirred at room temperature for 16 h. The hydrogen gas was released slowly and the solution was concentrated and passed through a short column of silica gel to remove the metal complex. The product was analyzed by NMR spectroscopy for conversion and chiral HPLC for ee values.

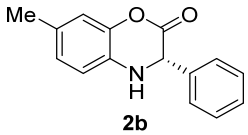
(*S*)-3-phenyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2a)



2a

White solid; >99% conversion; 93% yield, 10.5 mg; 99% ee; known compound;[3] $[\alpha]_D^{25}$ = +78.4 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 16.6 min (major), 11.4 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39-7.35 (m, 5H), 7.05-7.00 (m, 2H), 6.88-6.70 (m, 2H), 5.05 (d, *J* = 4.0 Hz, 1H), 4.27 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.2, 140.8, 136.3, 132.3, 128.97, 128.95, 127.5, 125.2, 120.4, 116.9, 114.8, 59.2.

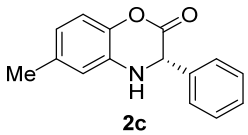
(*S*)-7-methyl-3-phenyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2b)



2b

Off-white solid, mp = 81-84 °C; >99% conversion; 94% yield, 11.2 mg; >99% ee; known compound;[8] $[\alpha]_D^{25}$ = +105.1 (c = 0.7, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 21.0 min (major), 12.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39-7.35 (m, 5H), 6.86-6.82 (m, 2H), 6.71 (d, *J* = 8.0 Hz, 1H), 5.02 (s, 1H), 4.15 (s, 1H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.5, 140.8, 136.4, 130.3, 129.8, 128.9, 127.5, 125.6, 117.3, 114.8, 59.4, 20.6.

(*S*)-6-methyl-3-phenyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2c)

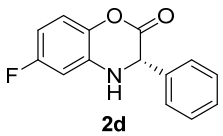


2c

Off-white solid; >99% conversion; 95% yield, 11.4 mg; >99% ee; known compound;[8] $[\alpha]_D^{25}$ = +115.3 (c = 0.7, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 14.5 min (major), 10.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.41-7.36 (m, 5H), 6.93 (d, *J*

= 8.0 Hz, 1H), 6.66 (d, *J* = 8.0 Hz, 1H), 6.62 (s, 1H), 5.04 (s, 1H), 4.17 (s, 1H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.3, 138.8, 136.5, 135.0, 131.9, 128.96, 128.94, 127.4, 121.0, 116.6, 115.3, 59.3, 21.0.

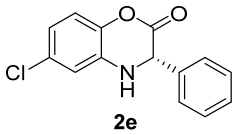
(*S*)-6-fluoro-3-phenyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2d)



2d

Off-white solid, mp = 105-108 °C; >99% conversion; 93% yield, 11.3 mg; >99% ee; $[\alpha]_D^{25}$ = +109.2 (c = 0.7, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 21.0 min (major), 12.7 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.40-7.38 (m, 5H), 7.00-6.96 (m, 1H), 6.58-6.52 (m, 2H), 5.07 (s, 1H), 4.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 164.6, 159.7 (d, *J* = 241.0 Hz), 136.84, 136.81, 136.0, 133.2 (d, *J* = 11.0 Hz), 129.1 (d, *J* = 7.0 Hz), 127.3, 117.8 (d, *J* = 10.0 Hz), 106.5 (d, *J* = 23.0 Hz), 101.8 (d, *J* = 27.0 Hz), 58.7. HRMS (ESI): [M+H$^+$] Calc. 244.0774, found 244.0763.

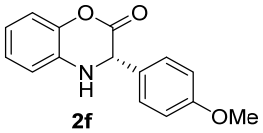
(*S*)-6-chloro-3-phenyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2e)



2e

Off-white solid, mp = 149-151 °C; >99% conversion; 94% yield, 12.2 mg; >99% ee; known compound;[5] $[\alpha]_D^{25}$ = +93.4 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 20.4 min (major), 22.4 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.38-7.37 (m, 5H), 6.96 (d, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 4.0 Hz, 1H), 6.81 (s, 1H), 5.08 (s, 1H), 4.35 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 164.4, 139.3, 135.9, 133.1, 130.2, 129.2, 129.1, 127.3, 120.1, 118.0, 114.6, 58.7.

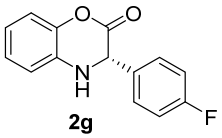
(*S*)-3-(4-methoxyphenyl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2f)



2f

Light yellow solid; >99% conversion; 92% yield, 11.7 mg; >99% ee; known compound;[3] $[\alpha]_D^{25}$ = +87.8 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 36.6 min (major), 13.7 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.32 (d, *J* = 8.0 Hz, 2H), 7.05-7.02 (m, 2H), 6.90-6.86 (m, 3H), 6.81-6.79 (m, 1H), 4.99 (s, 1H), 4.22 (s, 1H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.6, 160.0, 140.9, 132.5, 128.8, 128.3, 125.1, 120.3, 116.9, 114.8, 114.3, 58.8, 55.3.

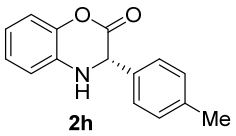
(*S*)-3-(4-fluorophenyl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2g)



2g

Off-white solid; >99% conversion; 92% yield, 11.2 mg; >99% ee; known compound;[3] $[\alpha]_D^{25}$ = +93.1 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 15.3 min (major), 8.6 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.42-7.38 (m, 2H), 7.09-7.04 (m, 4H), 6.89-6.82 (m, 2H), 5.04 (s, 1H), 4.23 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.1, 163.0 (d, *J* = 247.0 Hz), 140.9, 132.2, 132.0 (d, *J* = 3.0 Hz), 129.4 (d, *J* = 9.0 Hz), 125.2, 120.6, 117.0, 116.0 (d, *J* = 21.0 Hz), 114.9, 58.7.

(*S*)-3-(p-tolyl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2h)

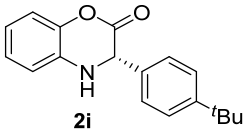


2h

Off-white solid; >99% conversion; 91% yield, 10.9 mg; >99% ee; known compound;[5] $[\alpha]_D^{25}$ = +99.8 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H

column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 33.2 min (major), 10.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.29 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.05-7.02 (m, 2H), 6.88-6.86 (m, 1H), 6.80 (d, *J* = 8.0 Hz, 1H), 5.01 (s, 1H), 4.23 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.4, 140.9, 138.9, 133.3, 132.5, 129.6, 127.3, 125.1, 120.3, 116.9, 114.8, 59.0, 21.2.

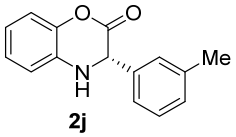
(*S*)-3-(4-(tert-butyl)phenyl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2i)



2i

Off-white solid; >99% conversion; 88% yield, 12.4 mg; >99% ee; known compound;[3] $[\alpha]_D^{25}$ = +98.8 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 8.8 min (major), 7.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.05-7.02 (m, 2H), 6.86-6.84 (m, 1H), 6.80-6.78 (m, 1H), 5.03 (s, 1H), 4.22 (s, 1H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.4, 152.0, 140.9, 133.3, 132.5, 127.2, 125.9, 125.1, 120.3, 116.9, 114.8, 59.0, 34.6, 31.2.

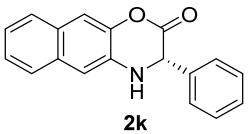
(*S*)-3-(m-tolyl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2j)



2j

Off-white solid, mp = 101-104 °C; >99% conversion; 95% yield, 11.4 mg; >99% ee; known compound;[8] $[\alpha]_D^{25}$ = +87.3 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 15.4 min (major), 10.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.25-7.16 (m, 4H), 7.06-7.00 (m, 2H), 6.88-6.86 (m, 1H), 6.80 (d, *J* = 8.0 Hz, 1H), 5.01 (s, 1H), 4.24 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.4, 140.9, 138.8, 136.2, 132.4, 129.8, 128.8, 128.2, 125.1, 124.5, 120.3, 116.9, 114.8, 59.3, 21.4.

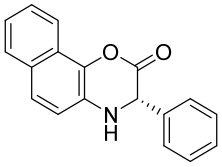
(*S*)-3-phenyl-3,4-dihydro-2*H*-naphtho[2,3-*b*][1,4]oxazin-2-one (2k)



2k

Brown solid; >99% conversion; 95% yield, 13.1 mg; >99% ee; known compound of racemic product;[4] $[\alpha]_D^{25}$ = +165.4 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 254 nm; t_R = 35.3 min (major), 25.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.73 (d, *J* = 8.0 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.48 (s, 1H), 7.43-7.32 (m, 7H), 7.15 (s, 1H), 5.19 (s, 1H), 4.55 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.2, 141.5, 136.4, 131.9, 131.6, 129.1, 128.5, 127.5, 127.3, 126.0, 125.8, 124.0, 113.7, 109.6, 59.2.

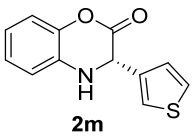
(*S*)-3-phenyl-3,4-dihydro-2*H*-naphtho[1,2-*b*][1,4]oxazin-2-one (2l)



2l

Brown solid; >99% conversion; 91% yield, 12.5 mg; 97% ee; known compound of racemic product;[4] $[\alpha]_D^{25}$ = -22.4 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 26.7 min (major), 22.4 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.09 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.53-7.48 (m, 1H), 7.46-7.43 (m, 2H), 7.37-7.35 (m, 4H), 7.04 (d, *J* = 8.0 Hz, 1H), 5.17 (s, 1H), 4.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 164.9, 136.3, 133.3, 129.0, 128.8, 127.9, 127.7, 127.5, 127.0, 125.1, 124.1, 123.9, 119.3, 116.0, 59.3.

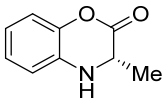
(*S*)-3-(thiophen-3-yl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2m)



2m

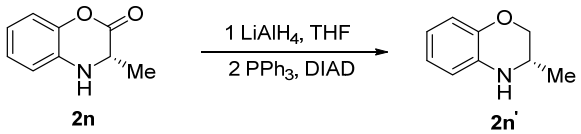
Yellow solid; >99% conversion; 96% yield, 11.1 mg; >99% ee; known compound;[9] $[\alpha]_D^{25}$ = +53.4 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 14.3 min (major), 10.6 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.33-7.31 (m, 1H), 7.27-7.26 (m, 1H), 7.09-7.07 (m, 1H), 7.05-7.01 (m, 2H), 6.87-6.81 (m, 2H), 5.20 (s, 1H), 4.31 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 164.6, 140.8, 137.0, 132.0, 127.0, 126.3, 125.2, 123.6, 120.5, 116.9, 115.0, 55.2.

(*S*)-3-methyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2n)



2n

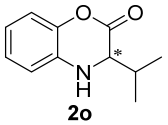
Off-white solid; >99% conversion; 91% yield, 7.4 mg; 97% ee; known compound;[10] $[\alpha]_D^{25}$ = +17.8 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 17.2 min (major), 13.6 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.04-6.98 (m, 2H), 6.88-6.84 (m, 1H), 6.79-6.76 (m, 1H), 3.98 (q, *J* = 4.0 Hz, 1H), 1.55 (d, *J* = 4.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 167.3, 141.4, 132.9, 124.9, 120.4, 116.9, 115.0, 50.5, 17.2.



2n **2n'**

Experimental value for (*S*)-**2n'**: $[\alpha]_D^{20}$ = +15.4 (c = 0.4, $CHCl_3$); Literature data $[\alpha]_D^{20}$ = +19.8 (c = 1.0, $CHCl_3$).[11]

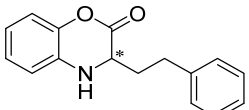
(+)-3-isopropyl-3,4-dihydro-2H-benzo[b][1,4]oxazin-2-one (2o)



2o

White solid; mp = 61-63 °C; >99% conversion; 95% yield, 9.0 mg; 98% ee; known compound;[3] $[\alpha]_D^{25}$ = +24.0 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 254 nm; t_R = 8.2 min (minor), 10.8 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00-6.96 (m, 2H), 6.83-6.75 (m, 2H), 3.98 (s, 1H), 3.77-3.76 (m, 1H), 2.31-2.13 (m, 1H), 1.07 (d, *J* = 6.9 Hz, 3H), 1.01 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 140.7, 132.1, 124.9, 119.8, 116.6, 114.7, 60.4, 29.8, 18.9, 17.6.

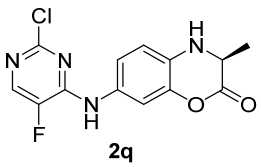
(+)-3-phenethyl-3,4-dihydro-2H-benzo[b][1,4]oxazin-2-one (2p)



2p

Brown solid; >99% conversion; 91% yield, 11.5 mg; 91% ee; known compound;[1] $[\alpha]_D^{25}$ = +6.5 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 13.7 min (minor), 22.4 min (major).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.3 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 3H), 7.02-6.96 (m, 2H), 6.86-6.82 (m, 1H), 6.66-6.64 (m, 1H), 3.93 (ddd, *J* = 7.3, 5.2, 2.1 Hz, 1H), 3.74 (s, 1H), 2.86-2.80 (m, 2H), 2.32-2.27 (m, 1H), 2.11-2.05 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 141.1, 140.3, 132.1, 128.7, 128.4, 126.5, 124.9, 120.4, 116.8, 115.2, 54.4, 32.6, 31.7.

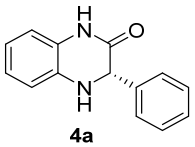
(*S*)-7-((2-chloro-5-fluoropyrimidin-4-yl)amino)-3-methyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (2q)



2q

Off-white solid; >99% conversion; 92% yield, 14.1 mg; 91% ee; $[\alpha]_D^{25}$ = +17.8 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 18.1 min (major), 26.2 min (minor). ^{1}H NMR (400 MHz, C_2D_6SO): δ (ppm) 9.90 (s, 1H), 8.27 (d, *J* = 4.0 Hz, 1H), 7.41 (d, *J* = 4.0 Hz, 1H), 7.26-7.24 (m, 1H), 6.86-6.84 (m, 1H), 6.37 (s, 1H), 4.03-3.98 (m, 1H), 1.38 (d, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, C_2D_6SO): δ (ppm) 167.9, 153.5, 151.5 (d, *J* = 11.0 Hz), 145.6 (d, *J* = 257.0 Hz), 143.2, 141.5 (d, *J* = 20.0 Hz), 140.8, 131.9, 129.7, 119.2, 115.0, 110.8, 50.0, 17.0.

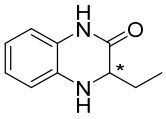
(*S*)-3-phenyl-3,4-dihydroquinoxalin-2(1*H*)-one (4a)



4a

Off-white solid; >99% conversion; 90% yield, 10.1 mg; >99% ee; known compound;[5] $[\alpha]_D^{25}$ = +78.6 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 16.0 min (major), 26.1 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 9.09 (s, 1H), 7.41-7.40 (m, 2H), 7.32-7.28 (m, 3H), 6.92-6.88 (m, 1H), 6.76-6.68 (m, 3H), 5.06 (s, 1H), 4.30 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 167.2, 139.0, 132.8, 128.8, 128.4, 127.1, 124.7, 124.0, 119.3, 115.7, 113.6, 60.6.

(+)-3-ethyl-3,4-dihydroquinoxalin-2(1*H*)-one (4b)

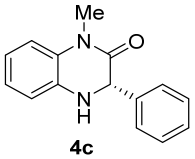


4b

Viscous liquid; >99% conversion; 91% yield, 8.0 mg; 99% ee; $[\alpha]_D^{25}$ = +27.0 (c = 0.5, $CHCl_3$), The enantiomeric excess was determined by HPLC on Chiralcel OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 7.9 min (major), 9.5 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 9.10 (s, 1H), 6.90-6.86 (m, 1H), 6.77-6.72 (m, 2H), 6.68 (d,

J = 8.0 Hz, 1H), 3.99 (s, 1H), 3.89-3.86 (m, 1H), 1.90-1.75 (m, 2H), 1.04 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 169.2, 133.0, 125.2, 123.8, 119.2, 115.4, 113.9, 57.5, 25.0, 9.6.

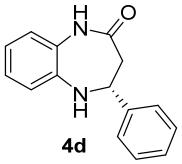
(*S*)-1-methyl-3-phenyl-3,4-dihydroquinoxalin-2(1*H*)-one (4c)



4c

Off-white solid; >99% conversion; 91% yield, 10.8 mg; 98% ee; known compound;[5] $[\alpha]_D^{25}$ = +115.9 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 11.5 min (major), 14.9 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.38-7.36 (m, 2H), 7.31-7.29 (m, 3H), 6.98-6.86 (m, 3H), 6.75-6.73 (m, 1H), 5.05 (s, 1H), 4.36 (s, 1H), 3.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 166.0, 139.0, 134.4, 128.7, 128.3, 127.1, 123.7, 119.5, 114.8, 113.9, 60.8, 29.2.

(*R*)-1,3,4,5-Tetrahydro-4-phenyl-2*H*-1,5-benzodiazepin-2-one (4d)

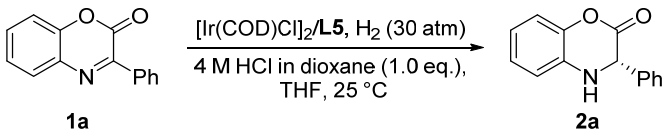


4d

Solid; >99% conversion; 93% yield, 11.1 mg; >99% ee; known compound;[12] $[\alpha]_D^{25}$ = -115.9 (c = 1.0, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralcel OJ-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 32.6 min (major), 18.9 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.18 (s, 1H), 7.38-7.33 (m, 5H), 7.09-7.05 (m, 1H), 6.96-6.94 (m, 2H), 6.85-6.83 (m, 1H), 5.04-5.01 (m, 1H), 3.84 (s, 1H), 2.92-2.86 (m, 1H), 2.76-2.72 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 172.0, 144.3, 138.3, 129.0, 128.1, 127.8, 126.1, 125.9, 122.4, 121.5, 121.1, 63.3, 41.8.

IV. Linear Effect of the Hydrogenation of Substrate 1a

Table S1. Linear effect for Ir/ligand **L5**-catalyzed asymmetric hydrogenation of **1a**.



entry	ee of **L5** (%)	conv. (%)	ee of **2a**(%)
1	0	>99	3
2	10	>99	13
3	20	>99	24
4	40	>99	45
5	60	>99	64
6	80	>99	81
7	90	>99	92
8	>99	>99	99

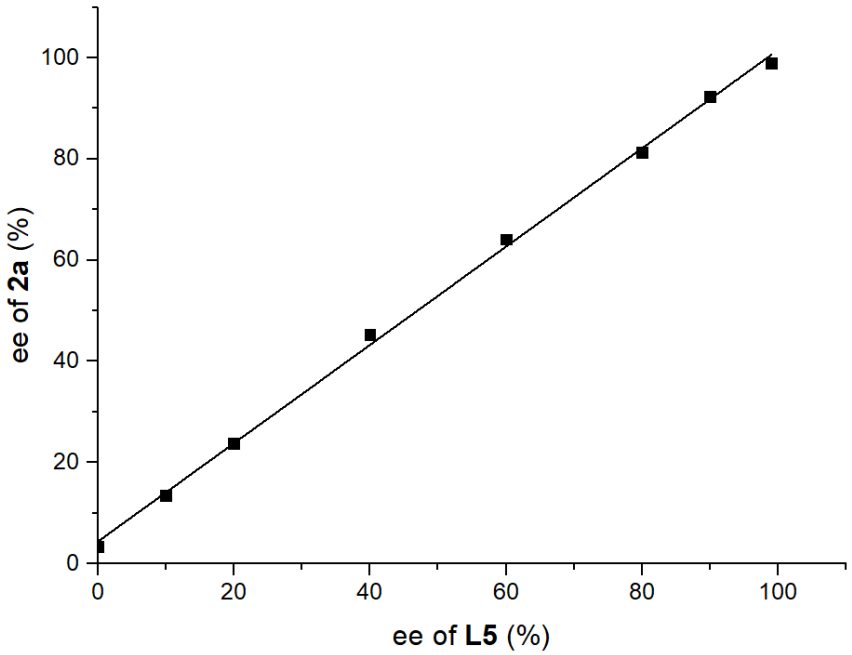
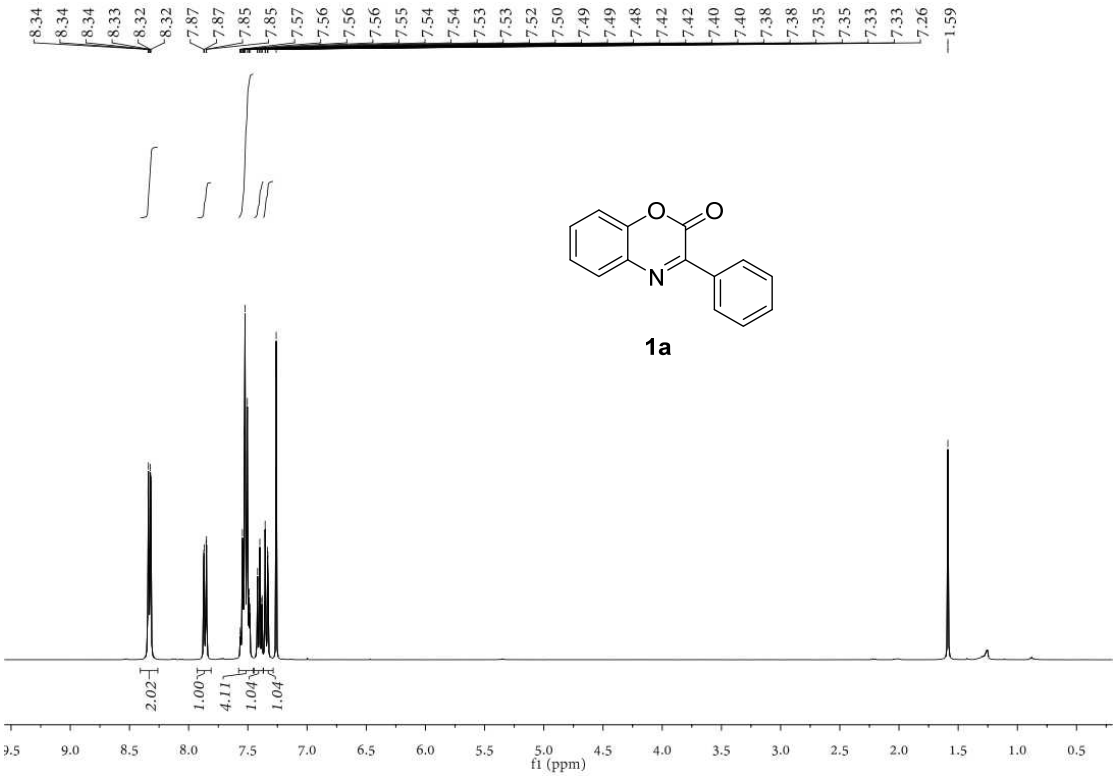


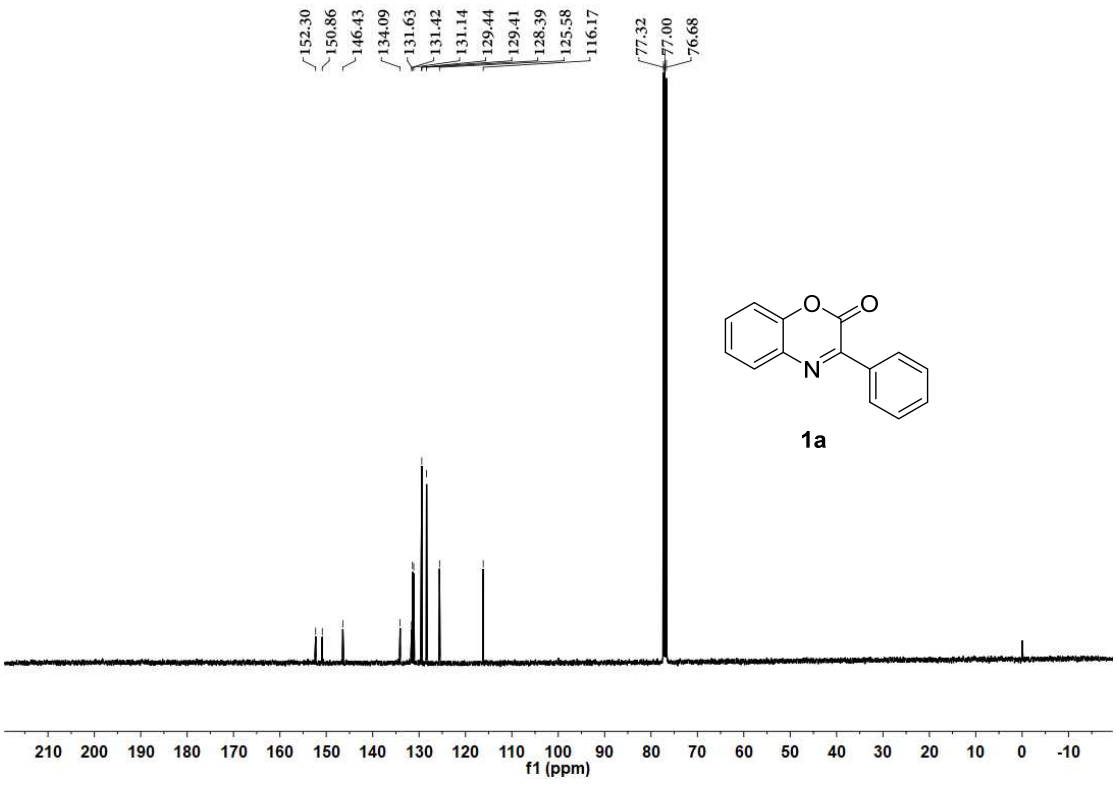
Figure S1. Linear effect of the hydrogenation of substrate **1a** using ligand **L5** with different ee values

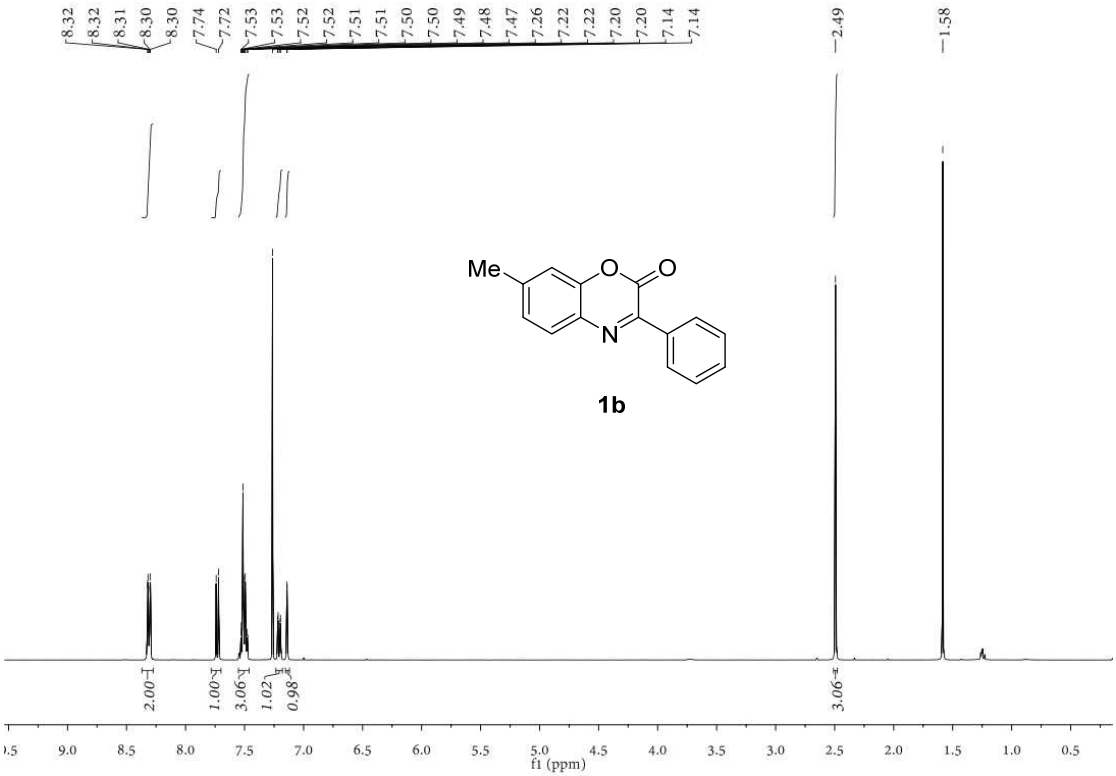
V. Reference

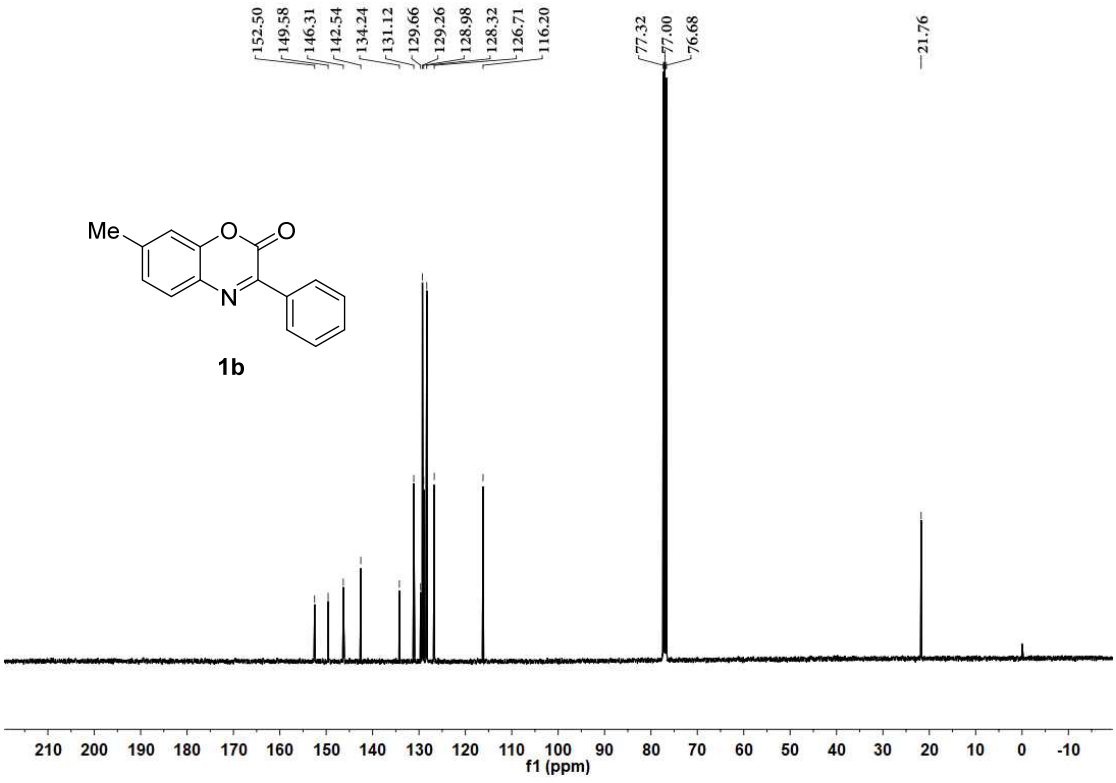
1. Z.-Y. Xue, Y. Jiang, X.-Z. Peng, W.-C. Yuan, X.-M. Zhang, *Adv. Syn. Catal.* **2010**, *352*, 2132-2136.
2. a) S. Yan, L. Ye, M. Liu, J. Chen, J. Ding, W. Gao, X. Huang, H. Wu, *RSC Adv.* **2014**, *4*, 16705-16709. b) D.-J. Zhang, W.-F. Sun, Z.-J. Zhong, R.-M. Gao, H. Yi, Y.-H. Li, Z.-G. Peng, Z.-R. Li, *Molecules* **2014**, *19*, 925-939. c) W.-G. Su, H. Jia, W. Zhang, Y. Cui, X. Yan, Y. Ren, J. Duan, Y. Sai, U.S. Pat. Appl. Publ., 20080255172, 16 Oct 2008. d) Y. C. Teo, S. N. Riduan, Y. Zhang, *Green Chem.* **2013**, *15*, 2365-2368. e) Y. Kamada, N. Sakai, S. Sogabe, K. Ida, H. Oki, K. Sakamoto, W. Lane, G. Snell, M. Iida, Y. Imaeda, J. Sakamoto, J. Matsui, *J. Med. Chem.* **2017**, *60*, 4358-4368.
3. L.-Q. Lu, Y. Li, K. Junge, M. Beller, *J. Am. Chem. Soc.* **2015**, *137*, 2763-2768.
4. S. Nonell, L. R. Ferreras, A. Cañete, E. Lemp, G. Günther, N. Pizarro, A. L. Zanocco, *J. Org. Chem.* **2008**, *73*, 5371-5378.
5. J. L. Núñez-Rico, A. Vidal-Ferran, *Org. Lett.* **2013**, *15*, 2066-2069.
6. a) K. R. Rao, A. Raghunadh, R. Mekala, S. B. Meruva, K. R. Ganesh, T. Krishna, D. Kalita, E. Laxminarayana, M. Palc, *J. Heterocyclic. Chem.* **2016**, *53*, 901-908. b) L. Shi, J. Zhou, J. Wu, J. Cao, Y. Shen, H. Zhou, X. Li, *Bioorg. Med. Chem.* **2016**, *24*, 1840-1852.
7. H. Mtiraoui, R. Gharbi, M. Msaddek, Y. Bretonniere, C. Andraud, C. Sabot, P.-Y. Renard, *J. Org. Chem.* **2016**, 81, 4720-4727.
8. Q.-A. Chen, K. Gao, Y. Duan, Z.-S. Ye, L. Shi, Y. Yang, Y.-G. Zhou, *J. Am. Chem. Soc.* **2012**, *134*, 2442-2448.
9. X. Zhang, B. Xu, M.-H. Xu, *Org. Chem. Front.* **2016**, *3*, 944-948.
10. R. I. Storer, D. E. Carrera, Y. Ni, D. W. C. MacMillan, *J. Am. Chem. Soc.* **2006**, *128*, 84-86.
11. V. N. Charushin, V. P. Krasnov, G. L. Levit, M. A. Korolyova, M. I. Kodess, O. N. Chupakhin, M. H. Kim, H. S. Lee, Y. J. Park, K.-C. Kim, *Tetrahedron: Asymmetry* **1999**, *10*, 2691-2702.
12. C. Fang, J. Cao, K. Sun, J. Zhu, T. Lu, D. Du, *Chem. Eur. J.* **2018**, *24*, 2103-2108.

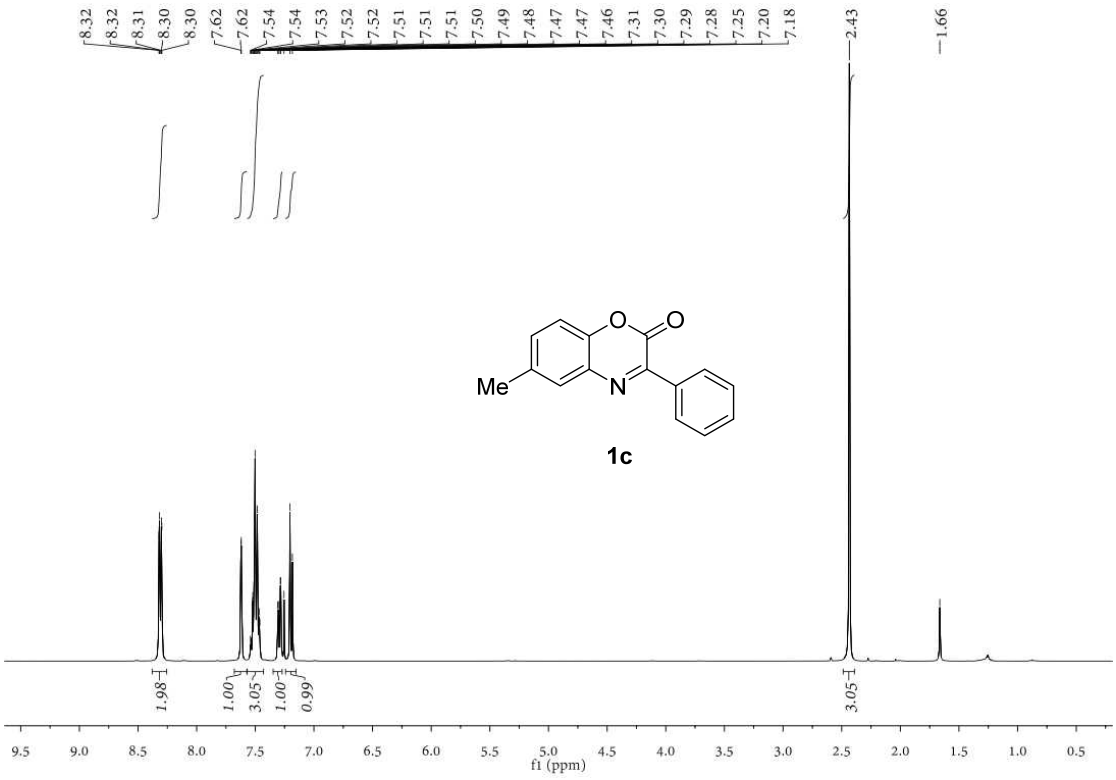
VI. NMR Spectra

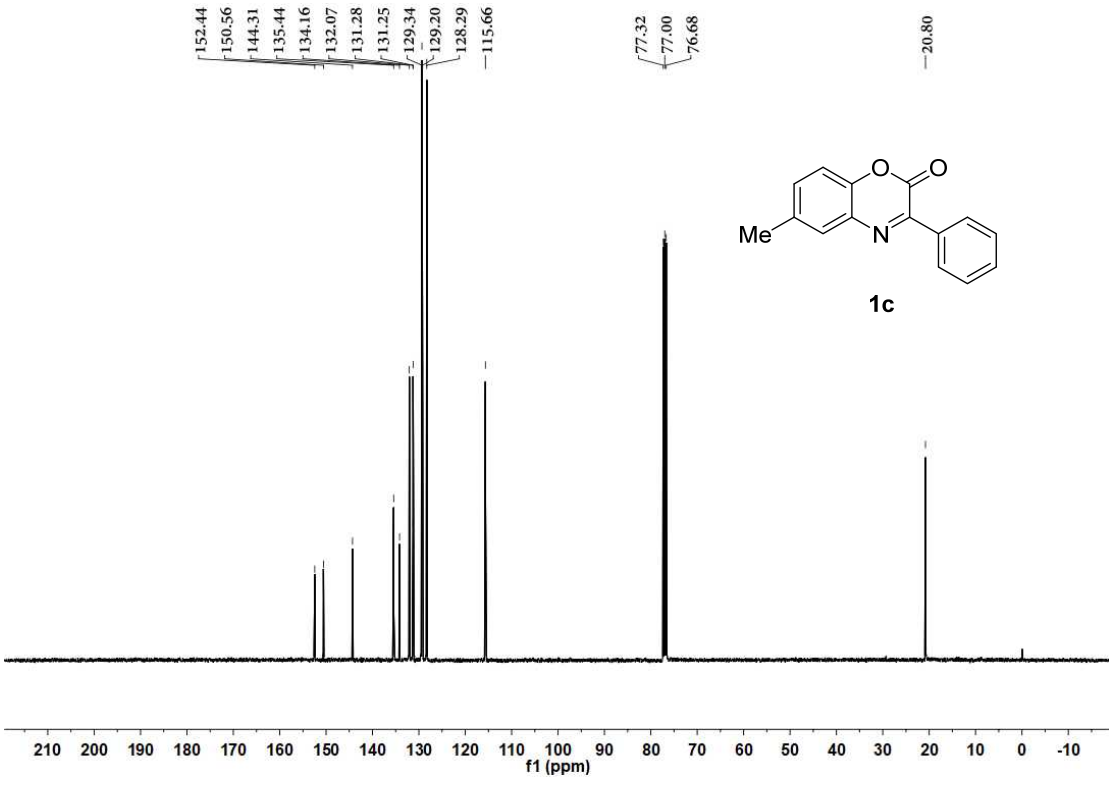


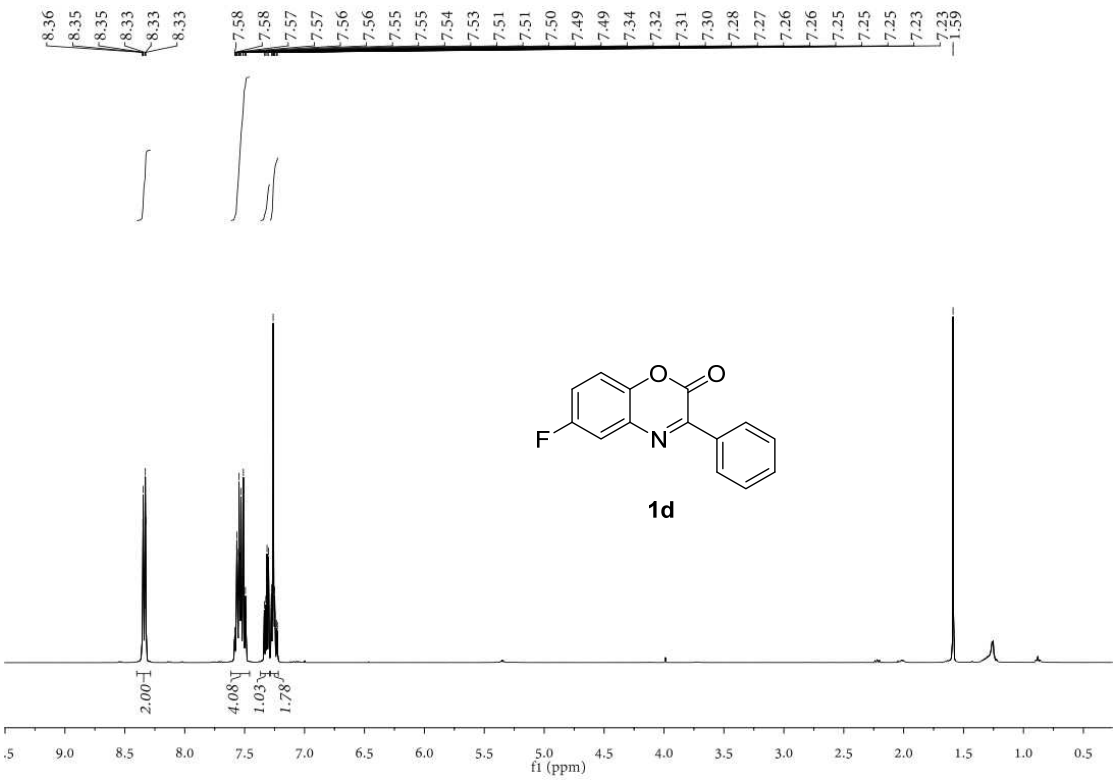


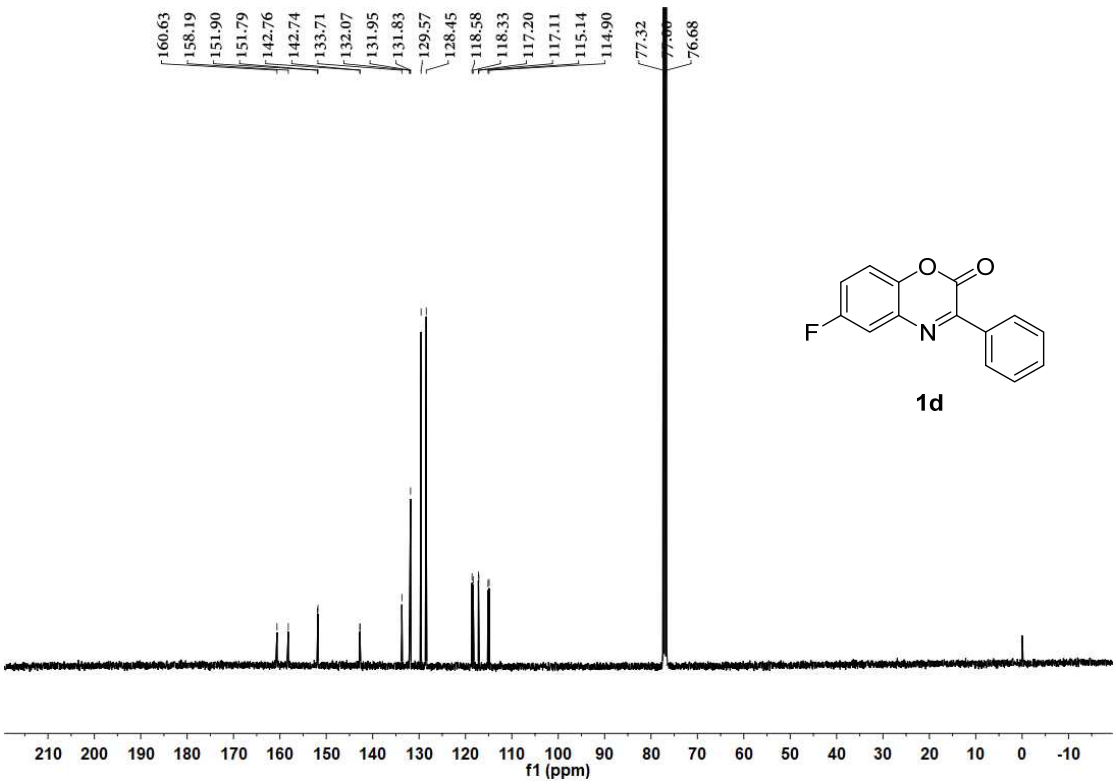

8.32
8.32
8.31
8.30
8.30
7.74
7.72
7.53
7.53
7.52
7.52
7.51
7.51
7.50
7.50
7.49
7.48
7.47
7.26
7.22
7.22
7.20
7.20
7.14
7.14
2.49
1.58
Me
O
O
N
1b
2.00
1.00
3.06
1.02
0.98
3.06
f1 (ppm)

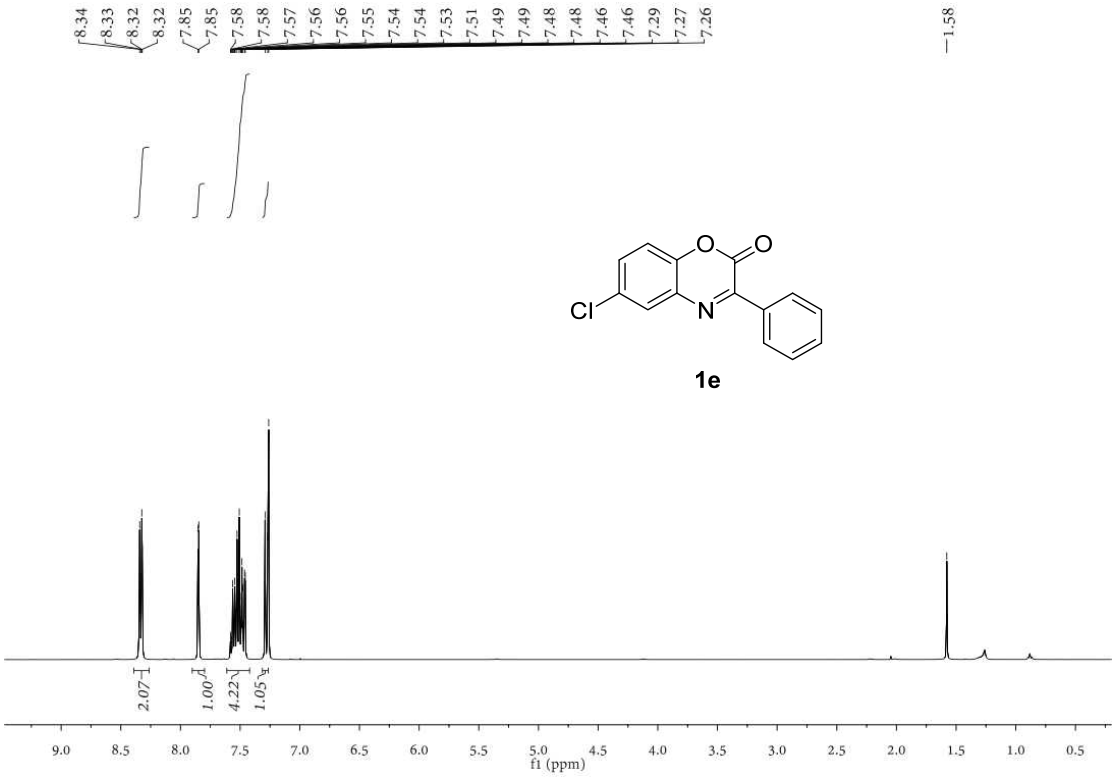

152.50
149.58
146.31
142.54
134.24
131.12
129.66
129.26
128.98
128.32
126.71
116.20
77.32
77.00
76.68
21.76
Me
O
O
N
1b
f1 (ppm)


8.32
8.32
8.31
8.30
8.30
7.62
7.62
7.54
7.54
7.53
7.52
7.52
7.51
7.51
7.51
7.50
7.49
7.48
7.47
7.47
7.46
7.31
7.30
7.29
7.28
7.25
7.20
7.18
2.43
1.66
Me
O
O
N
1c
1.98
1.00
3.05
1.00
0.99
3.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


152.44
150.56
144.31
135.44
134.16
132.07
131.28
131.25
129.34
129.20
128.29
115.66
77.32
77.00
76.68
20.80
Me
O
O
N
1c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

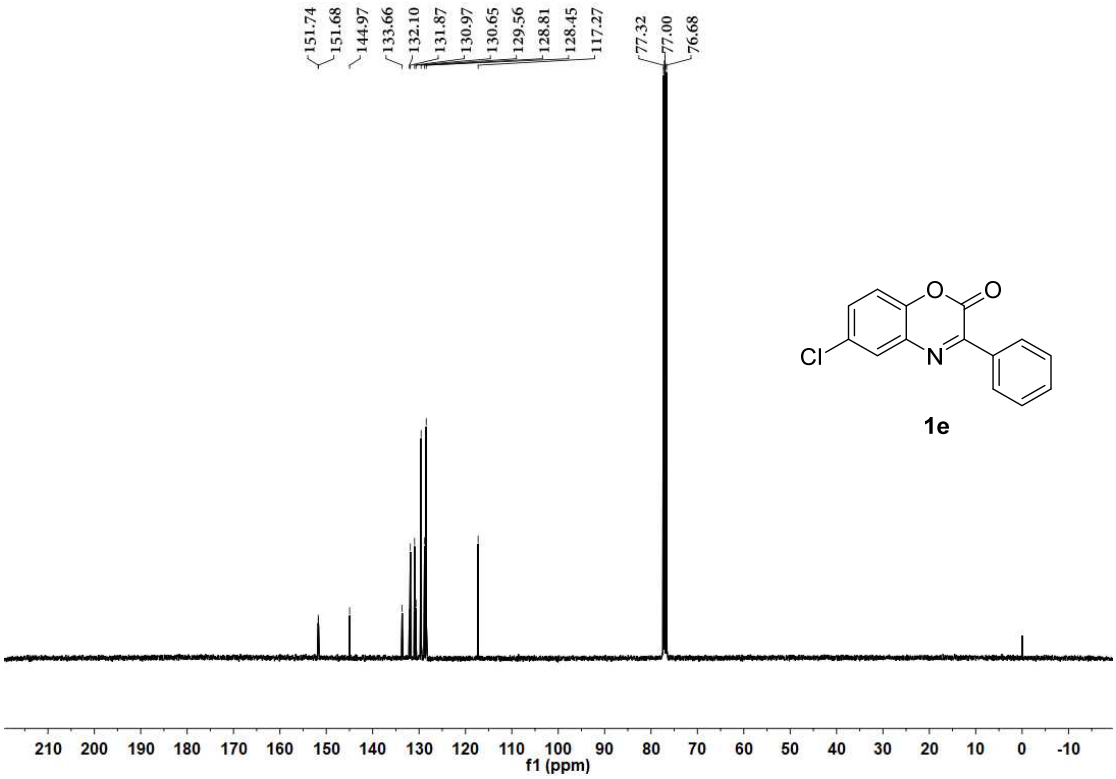

8.36
8.35
8.35
8.33
8.33
8.33
7.58
7.58
7.57
7.57
7.56
7.56
7.55
7.55
7.54
7.53
7.51
7.51
7.50
7.49
7.49
7.34
7.32
7.31
7.30
7.28
7.27
7.26
7.26
7.25
7.25
7.25
7.23
7.23
1.59
O
O
F
N
1d
2.00
4.08
1.03
1.78
f1 (ppm)


160.63
158.19
151.90
151.79
142.76
142.74
133.71
132.07
131.95
131.83
129.57
128.45
118.58
118.33
117.20
117.11
115.14
114.90
77.32
76.68
O
O
F
N
1d
f1 (ppm)


8.34
8.33
8.32
8.32
7.85
7.85
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.54
7.53
7.51
7.49
7.49
7.48
7.48
7.46
7.46
7.29
7.27
7.26
1.58
2.07
1.00
4.22
1.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

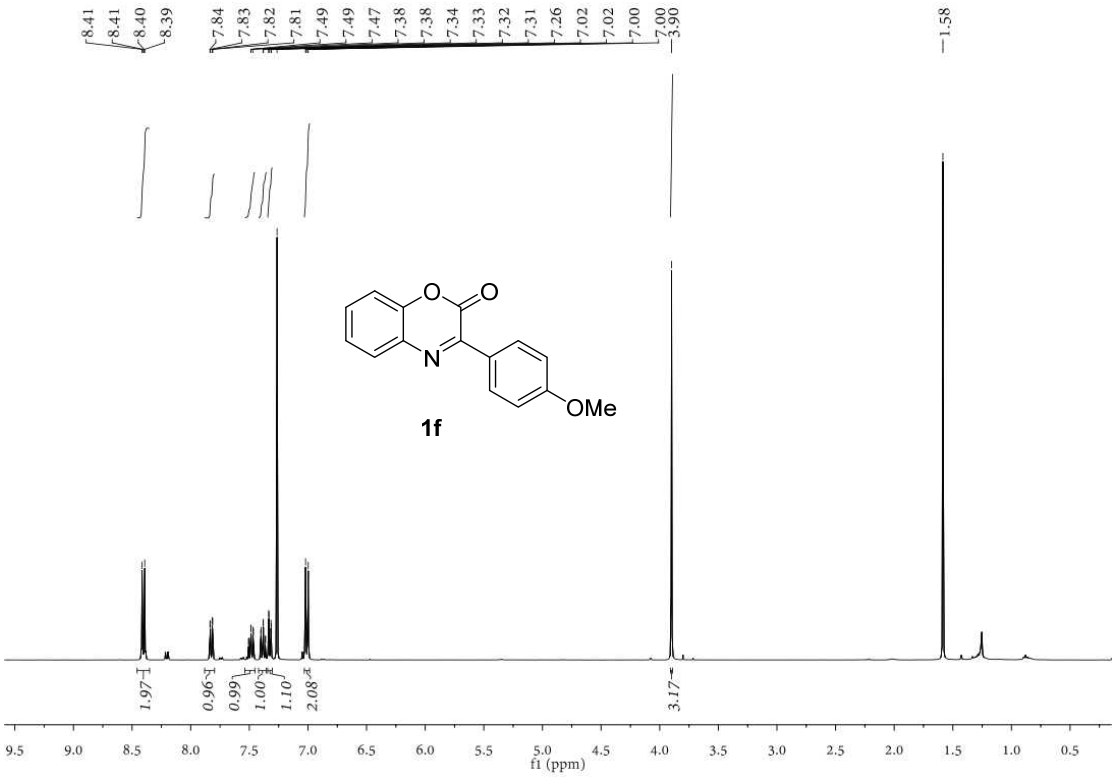
O
O
Cl
N

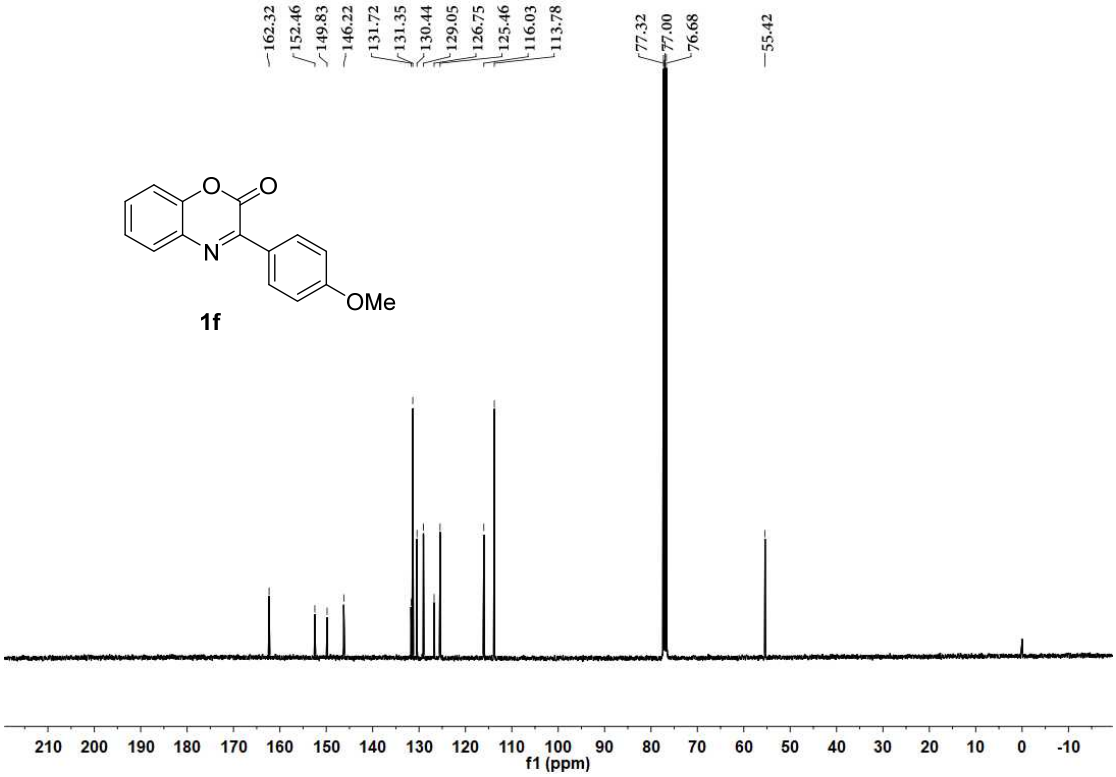
1e

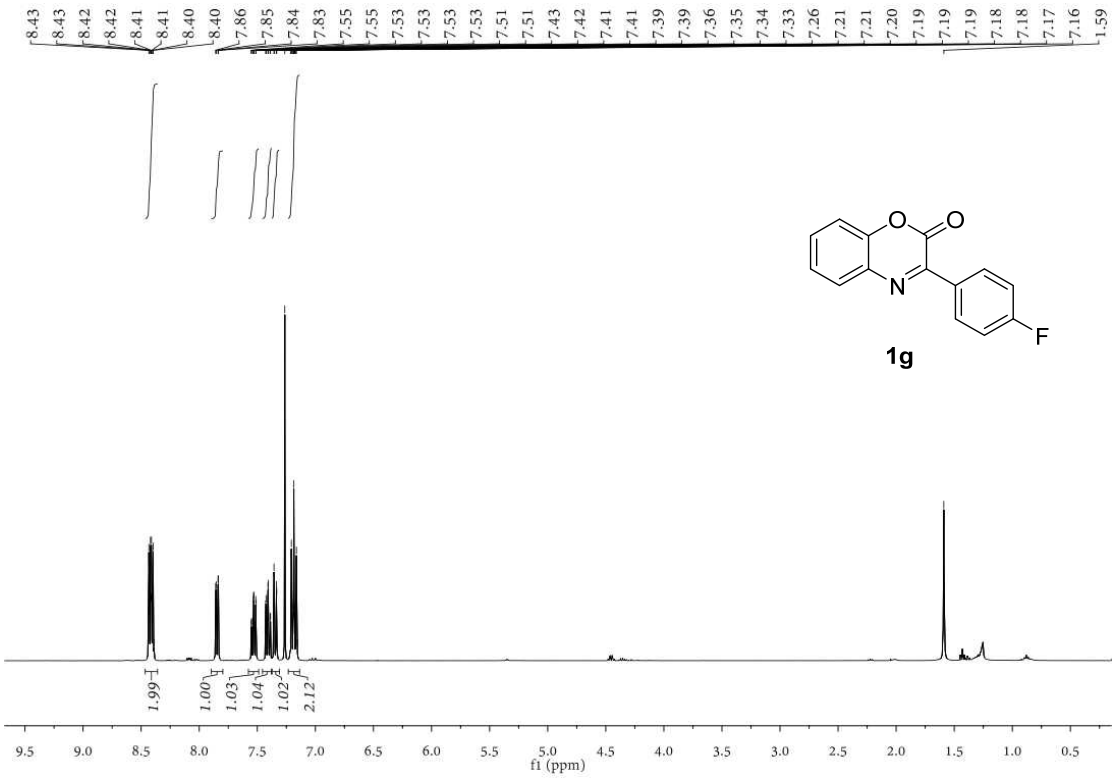

151.74
151.68
144.97
133.66
132.10
131.87
130.97
130.65
129.56
128.81
128.45
117.27
77.32
77.00
76.68
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

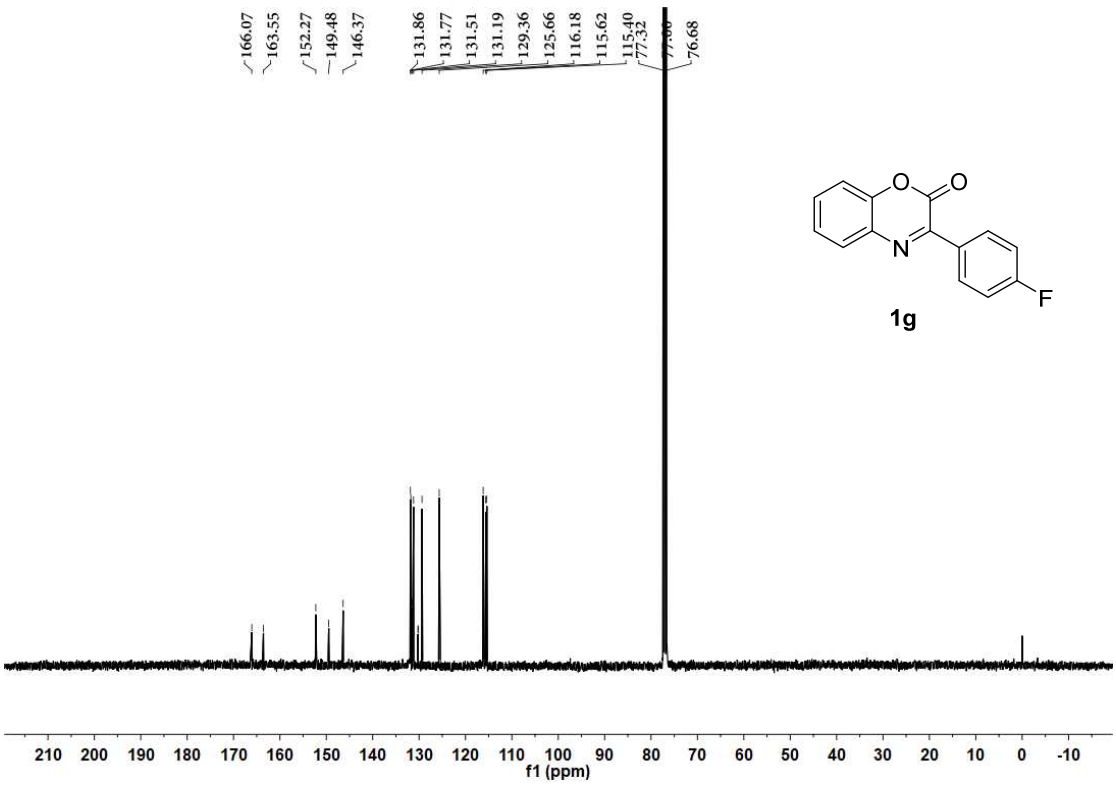
O
O
Cl
N

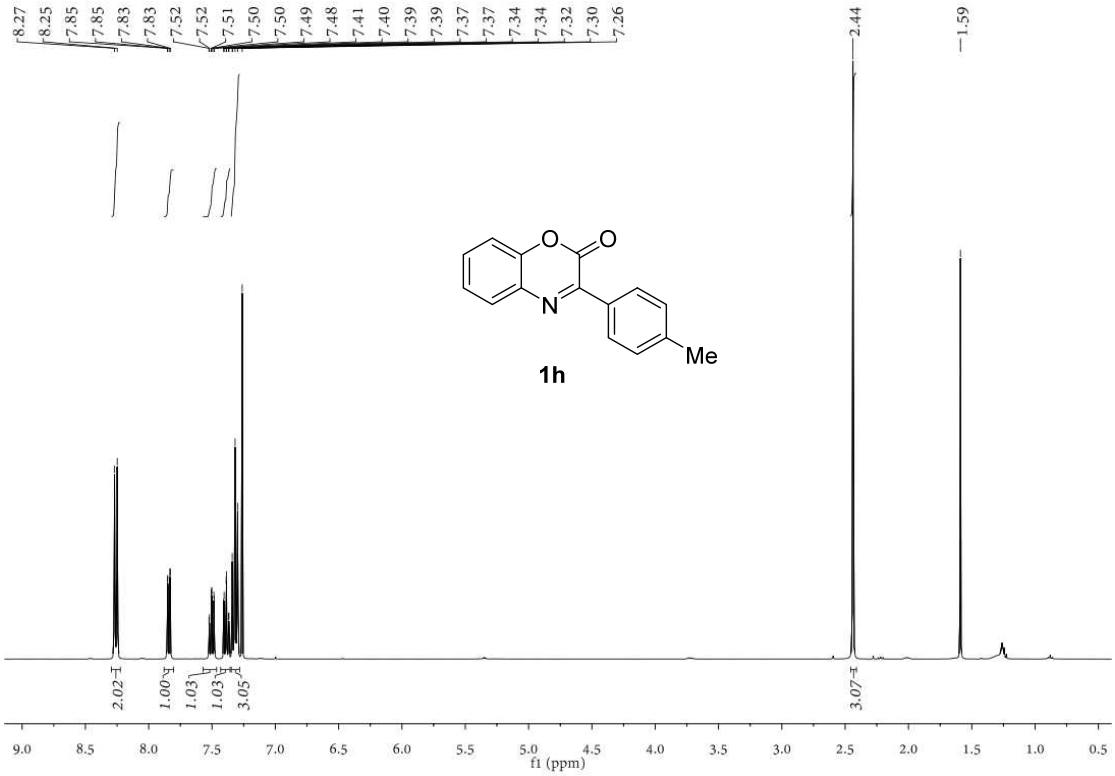
1e

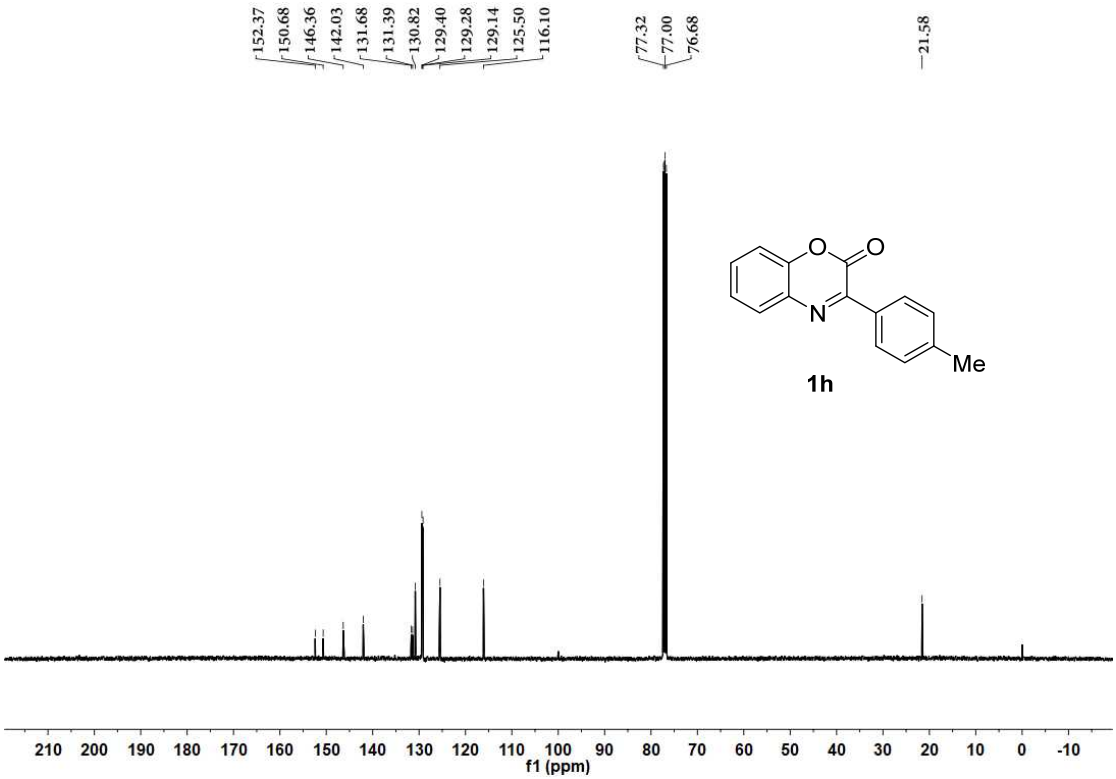

8.41
8.41
8.40
8.39
7.84
7.83
7.82
7.81
7.49
7.49
7.47
7.38
7.38
7.34
7.33
7.32
7.31
7.26
7.02
7.02
7.00
7.00
3.90
1.58
O
O
N
OMe
1f
1.97
0.96
0.99
1.00
1.10
2.08
3.17
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

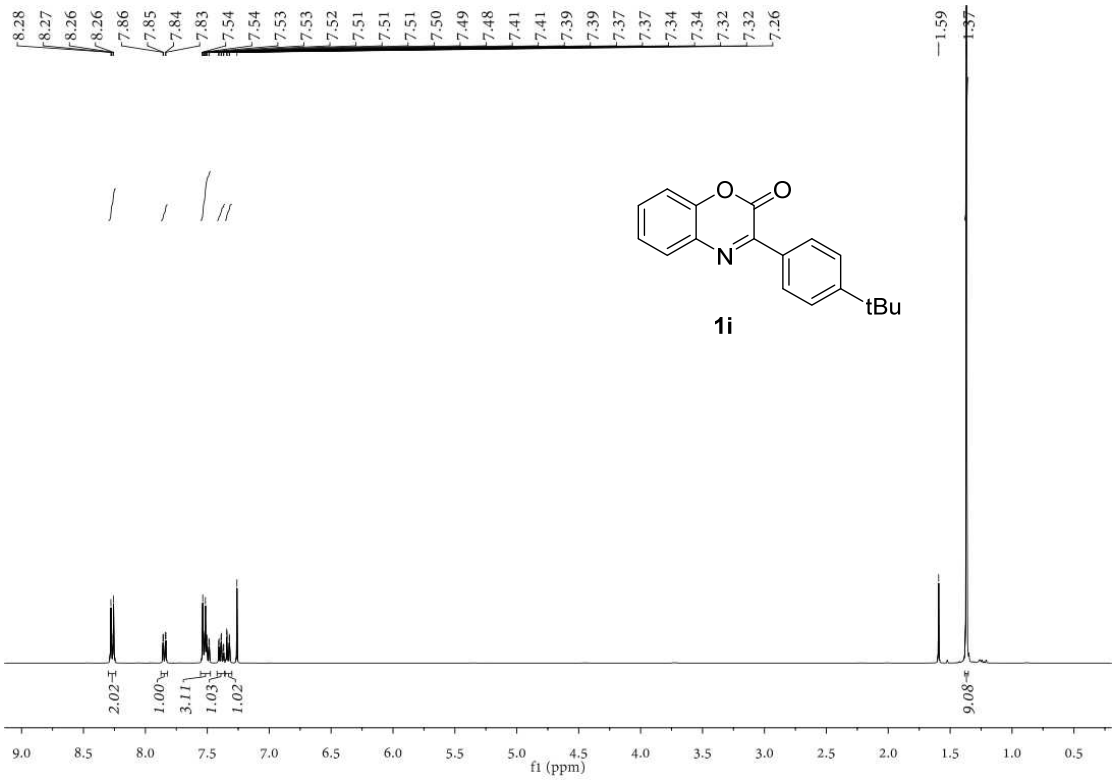

162.32
152.46
149.83
146.22
131.72
131.35
130.44
129.05
126.75
125.46
116.03
113.78
77.32
77.00
76.68
55.42
O
O
N
OMe
1f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

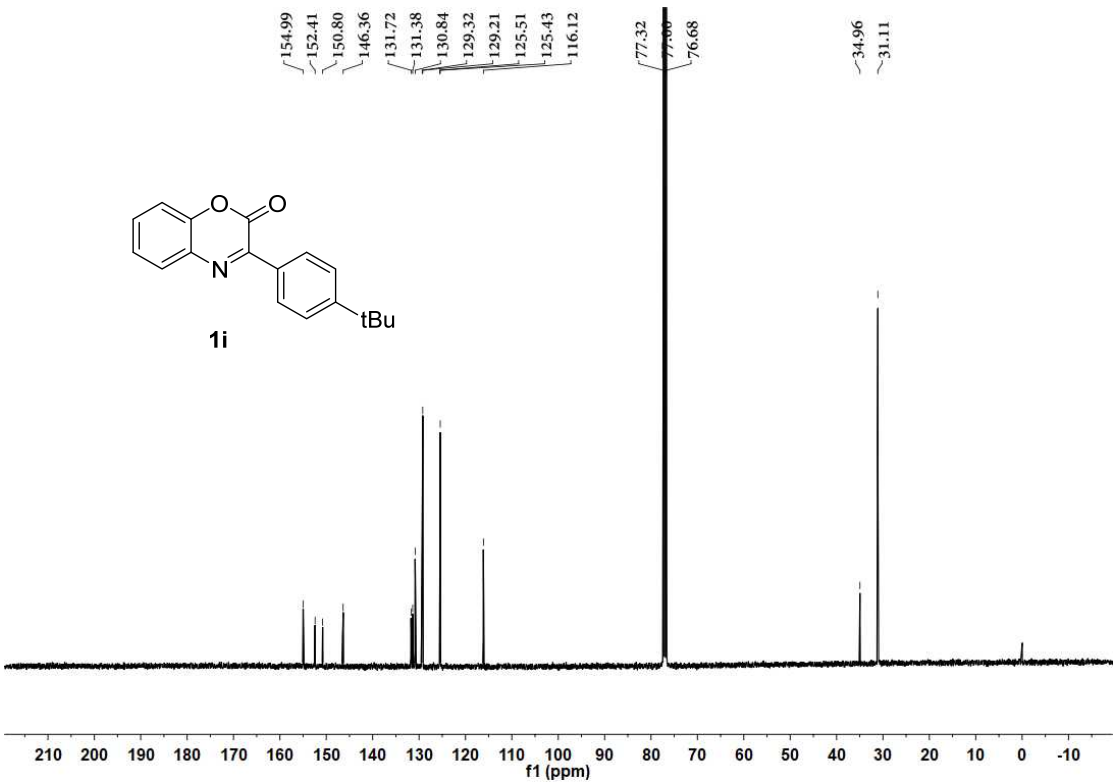

8.43 8.43 8.42 8.42 8.41 8.41 8.40 8.40 7.86 7.85 7.84 7.83 7.55 7.55 7.53 7.53 7.53 7.53 7.51 7.51 7.43 7.42 7.41 7.41 7.39 7.39 7.36 7.35 7.34 7.33 7.26 7.21 7.21 7.20 7.19 7.19 7.19 7.18 7.18 7.17 7.16 1.59
1.99 1.00 1.03 1.04 1.02 2.12
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
O
O
N
F
1g

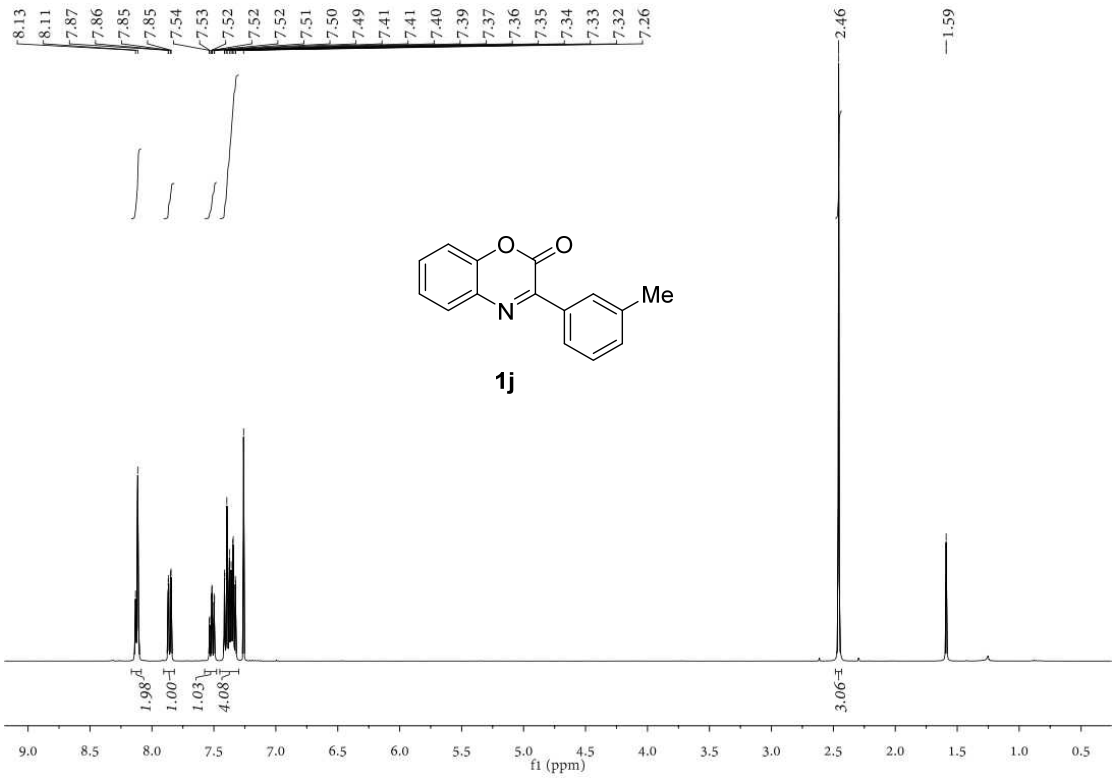

166.07 163.55 152.27 149.48 146.37 131.86 131.77 131.51 131.19 129.36 125.66 116.18 115.62 115.40 77.32 76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
O
O
N
F
1g


8.27
8.25
7.85
7.85
7.83
7.83
7.52
7.52
7.51
7.50
7.50
7.49
7.48
7.41
7.40
7.39
7.39
7.37
7.37
7.34
7.34
7.32
7.30
7.26
2.44
1.59
O
O
N
Me
1h
2.02
1.00
1.03
1.03
3.05
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

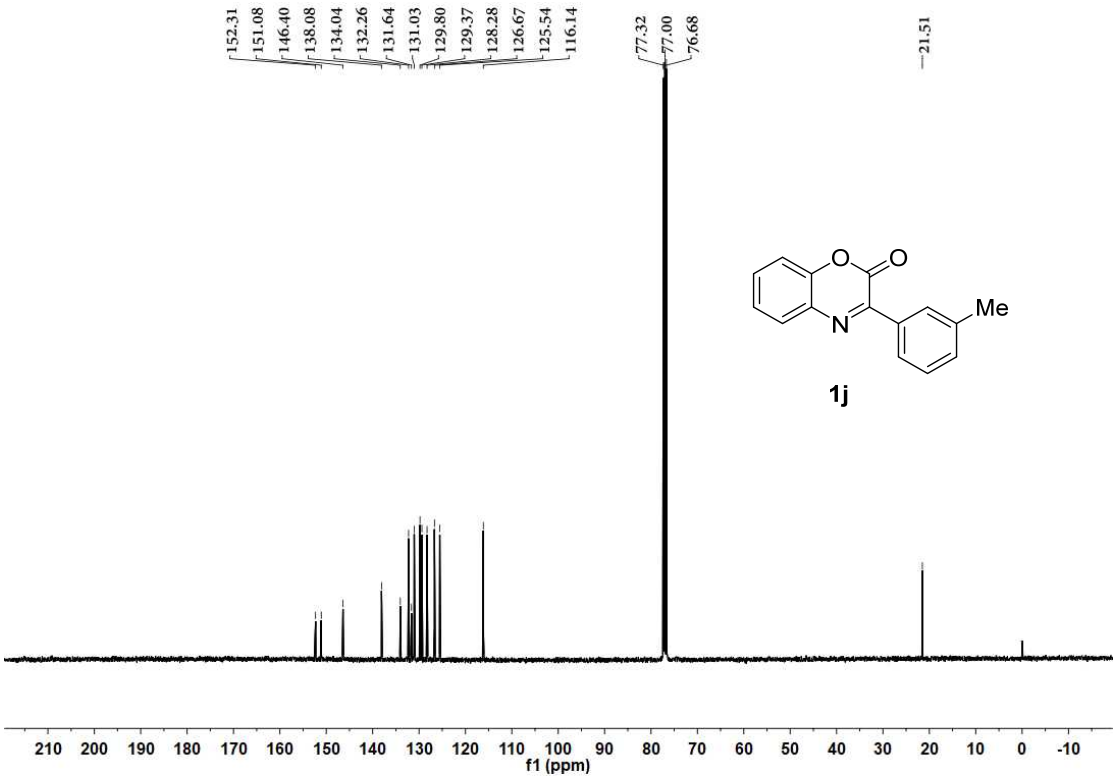

152.37
150.68
146.36
142.03
131.68
131.39
130.82
129.40
129.28
129.14
125.50
116.10
77.32
77.00
76.68
21.58
O
O
N
Me
1h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.28
8.27
8.26
8.26
7.86
7.85
7.84
7.83
7.54
7.54
7.53
7.53
7.52
7.51
7.51
7.51
7.50
7.49
7.48
7.41
7.41
7.39
7.39
7.37
7.37
7.34
7.34
7.32
7.32
7.26
1.59
1.37
O
O
N
tBu
1i
2.02
1.00
3.11
1.03
1.02
9.08
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

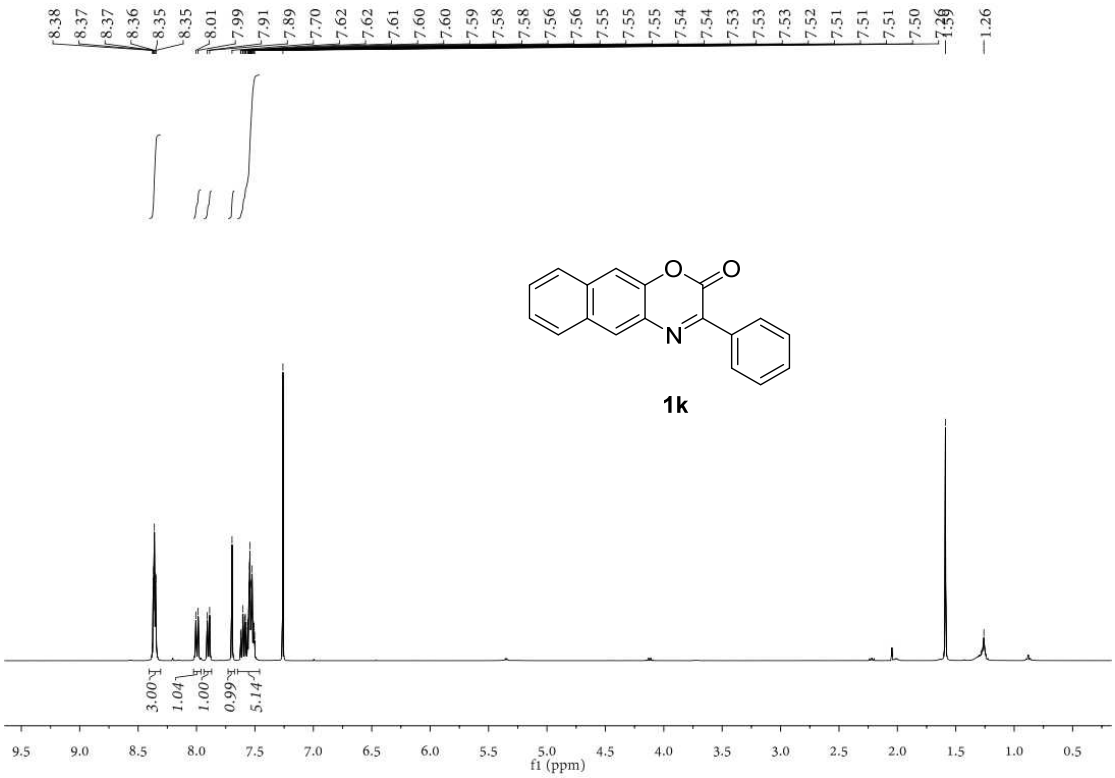

154.99
152.41
150.80
146.36
131.72
131.38
130.84
129.32
129.21
125.51
125.43
116.12
77.32
76.68
34.96
31.11
O
O
N
tBu
1i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

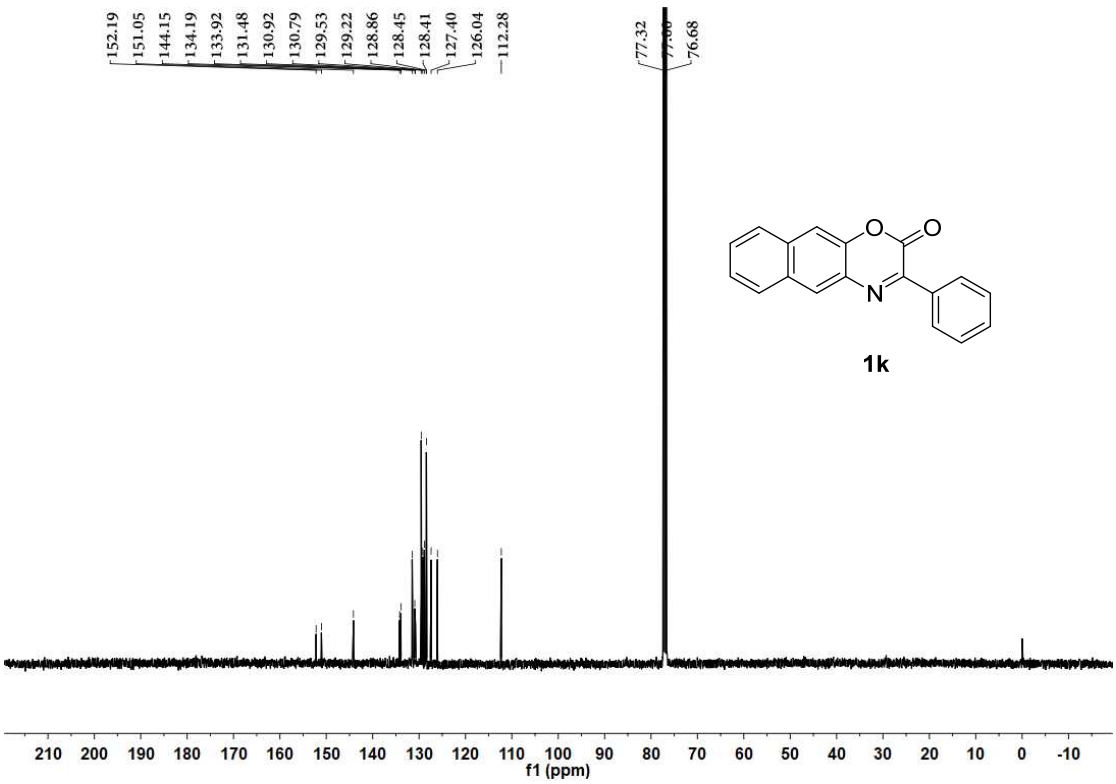

8.13
8.11
7.87
7.86
7.85
7.85
7.54
7.53
7.52
7.52
7.52
7.51
7.50
7.49
7.41
7.41
7.40
7.39
7.37
7.36
7.35
7.34
7.33
7.32
7.26
2.46
1.59
O
O
N
Me
1.98
1.00
1.03
4.08
3.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

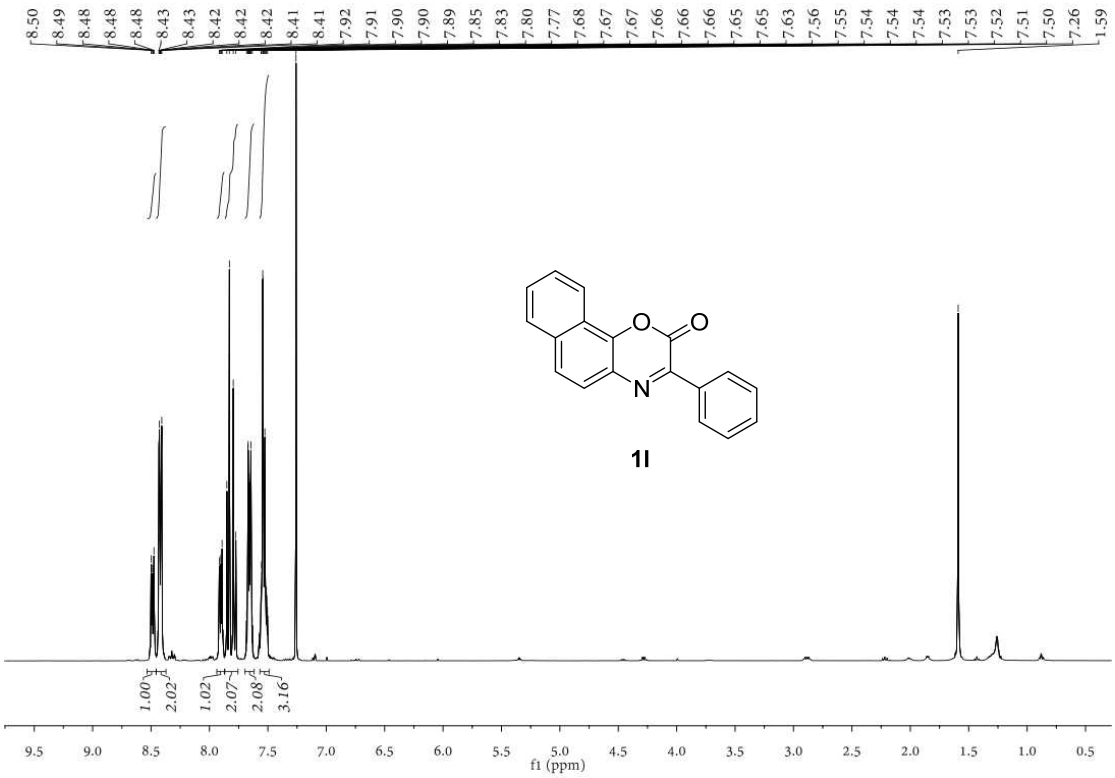
1j

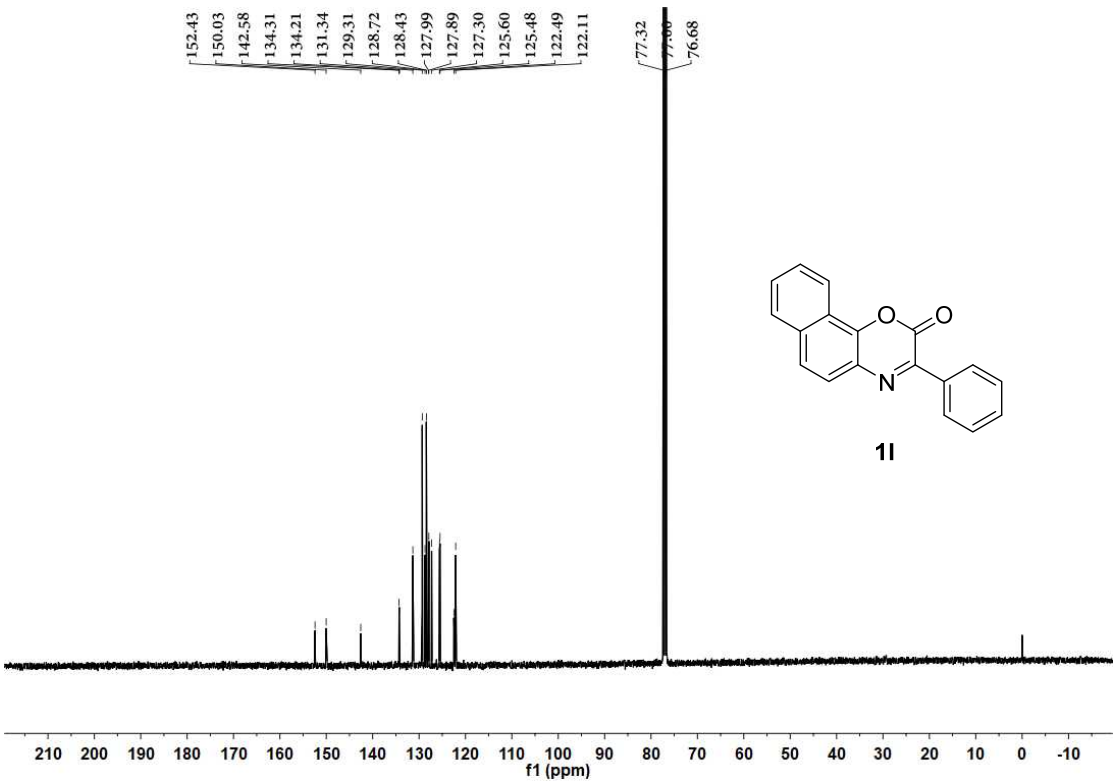

152.31
151.08
146.40
138.08
134.04
132.26
131.64
131.03
129.80
129.37
128.28
126.67
125.54
116.14
77.32
77.00
76.68
21.51
O
O
N
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

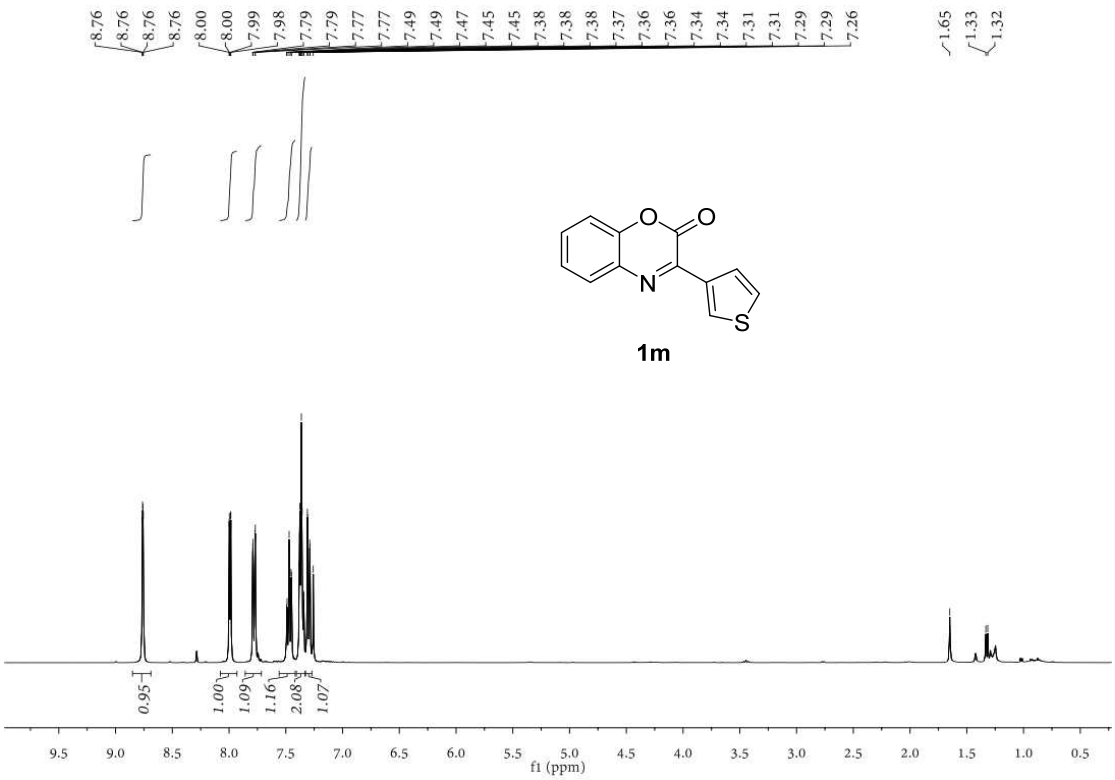
1j

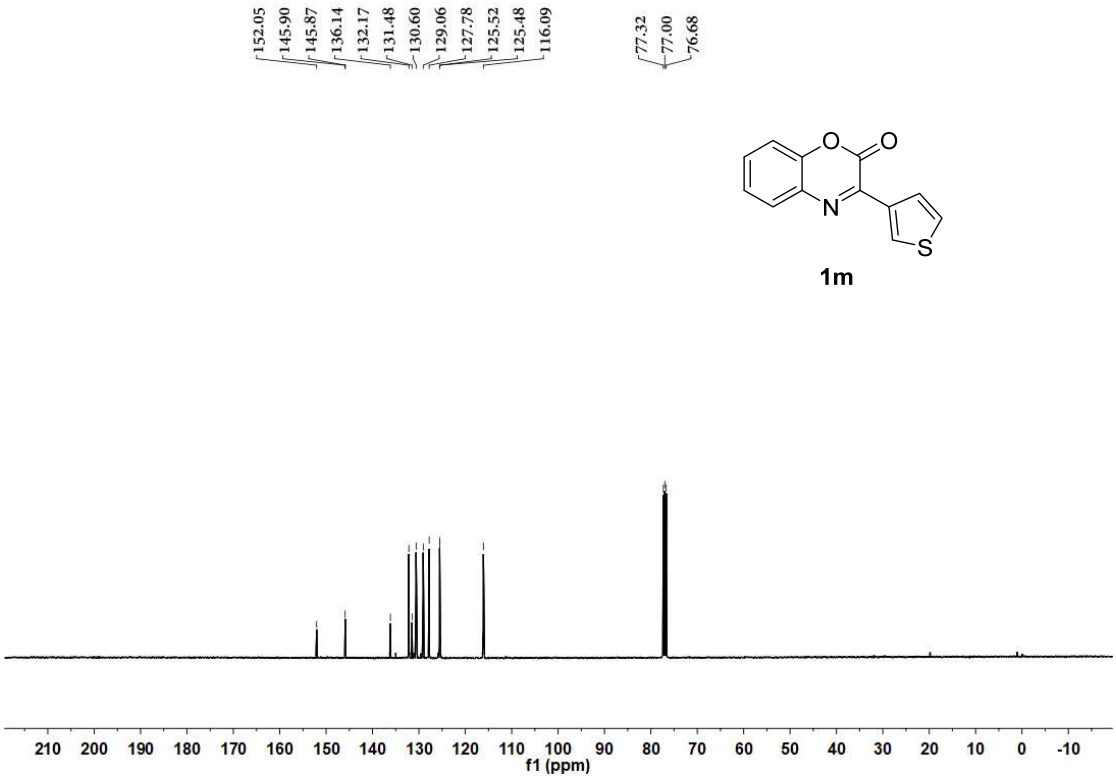

8.38
8.37
8.37
8.36
8.35
8.35
8.01
7.99
7.91
7.89
7.70
7.62
7.62
7.61
7.60
7.60
7.59
7.58
7.58
7.56
7.56
7.55
7.55
7.55
7.54
7.54
7.53
7.53
7.53
7.52
7.51
7.51
7.51
7.50
7.26
1.59
1.26
O
O
N
1k
3.00
1.04
1.00
0.99
5.14
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

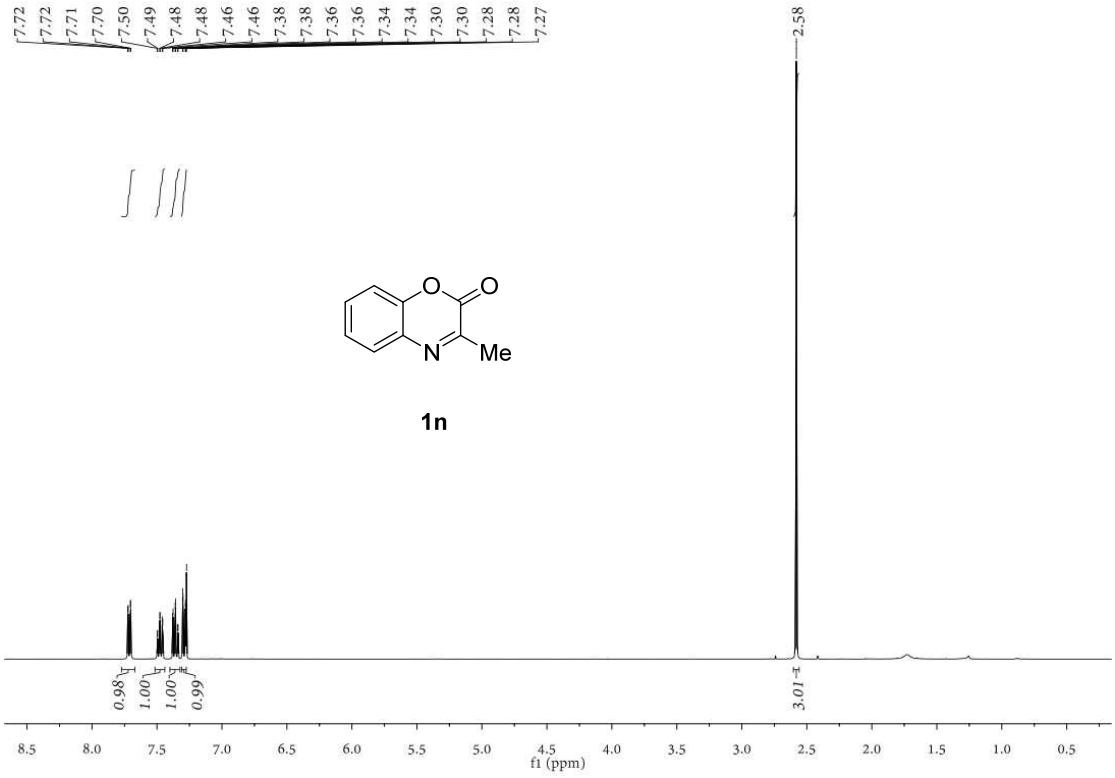

152.19
151.05
144.15
134.19
133.92
131.48
130.92
130.79
129.53
129.22
128.86
128.45
128.41
127.40
126.04
112.28
77.32
76.68
O
O
N
1k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

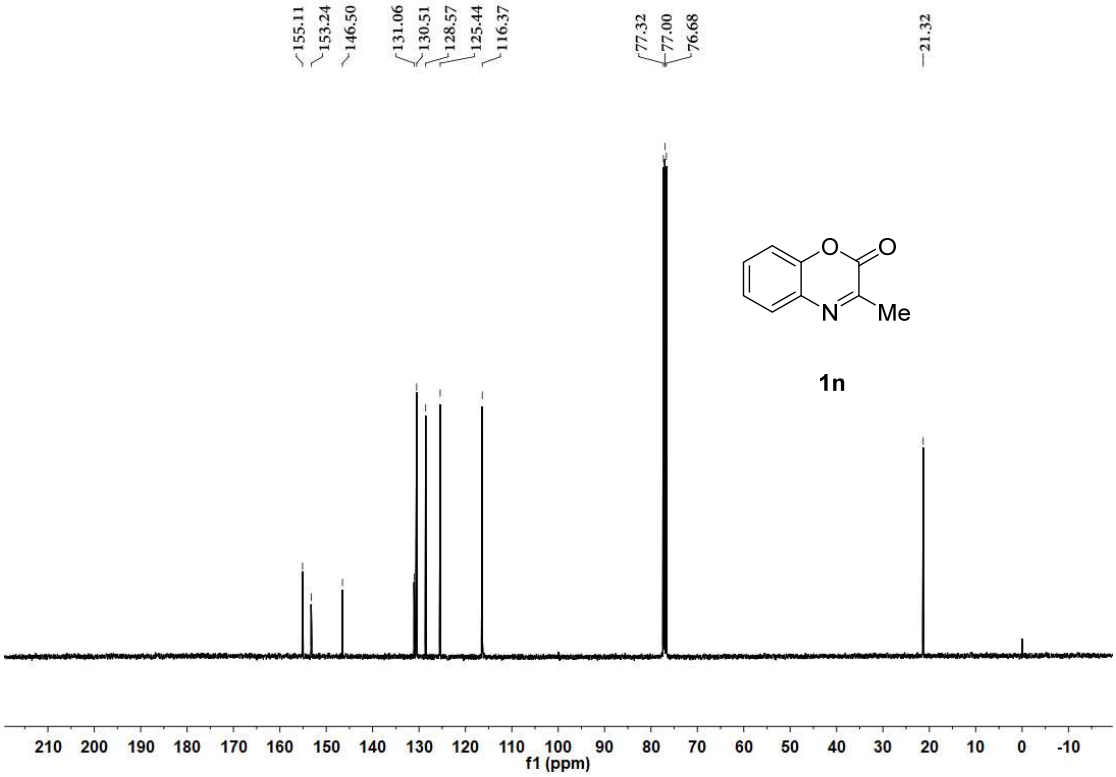

8.50 8.49 8.48 8.48 8.48 8.43 8.43 8.42 8.42 8.42 8.41 8.41 7.92 7.91 7.90 7.90 7.89 7.85 7.83 7.80 7.77 7.68 7.67 7.67 7.66 7.66 7.66 7.65 7.65 7.63 7.56 7.55 7.54 7.54 7.54 7.53 7.53 7.52 7.51 7.50 7.26 1.59
1.00 2.02 1.02 2.07 2.08 3.16
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
O
O
N
1l

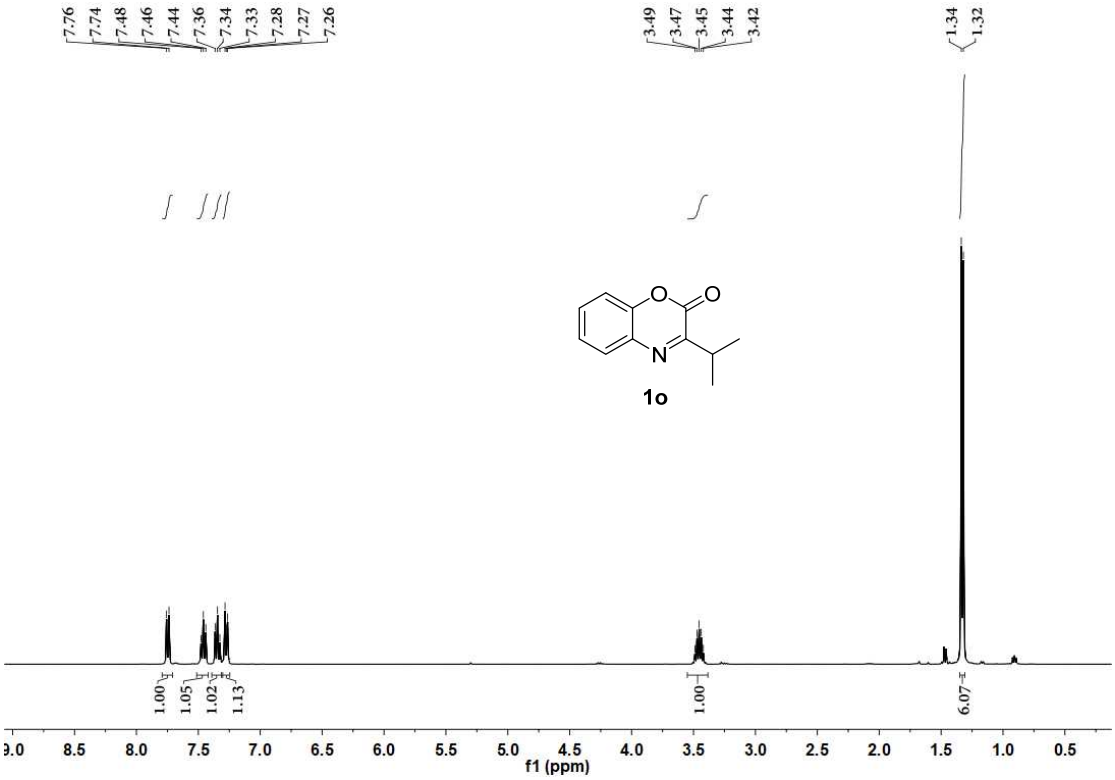

152.43 150.03 142.58 134.31 134.21 131.34 129.31 128.72 128.43 127.99 127.89 127.30 125.60 125.48 122.49 122.11
77.32 76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
O
O
N
1l


8.76
8.76
8.76
8.76
8.00
8.00
7.99
7.98
7.79
7.79
7.77
7.77
7.49
7.49
7.47
7.45
7.45
7.38
7.38
7.38
7.37
7.36
7.36
7.34
7.34
7.31
7.31
7.29
7.29
7.26
1.65
1.33
1.32
O
O
N
S
1m
0.95
1.00
1.09
1.16
2.08
1.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

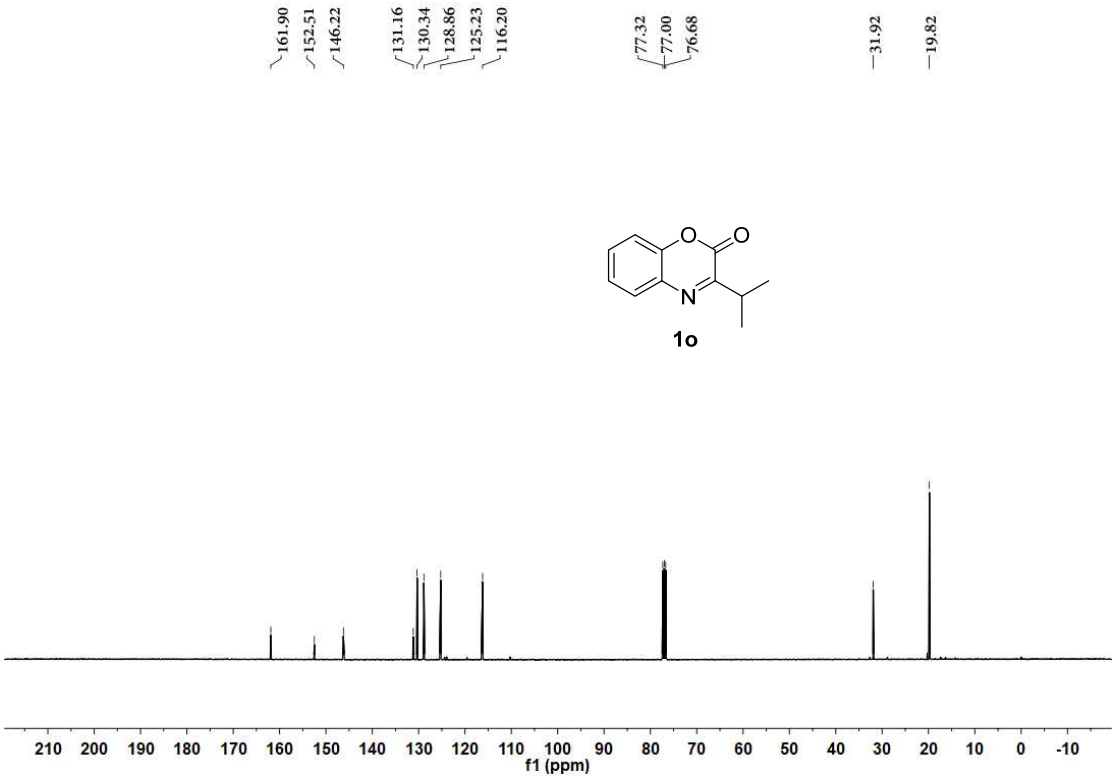

152.05
145.90
145.87
136.14
132.17
131.48
130.60
129.06
127.78
125.52
125.48
116.09
77.32
77.00
76.68
O
O
N
S
1m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.72
7.72
7.71
7.70
7.50
7.49
7.48
7.48
7.46
7.46
7.38
7.38
7.36
7.36
7.34
7.34
7.30
7.30
7.28
7.28
7.27
2.58
O
O
N
Me
1n
0.98
1.00
1.00
0.99
3.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

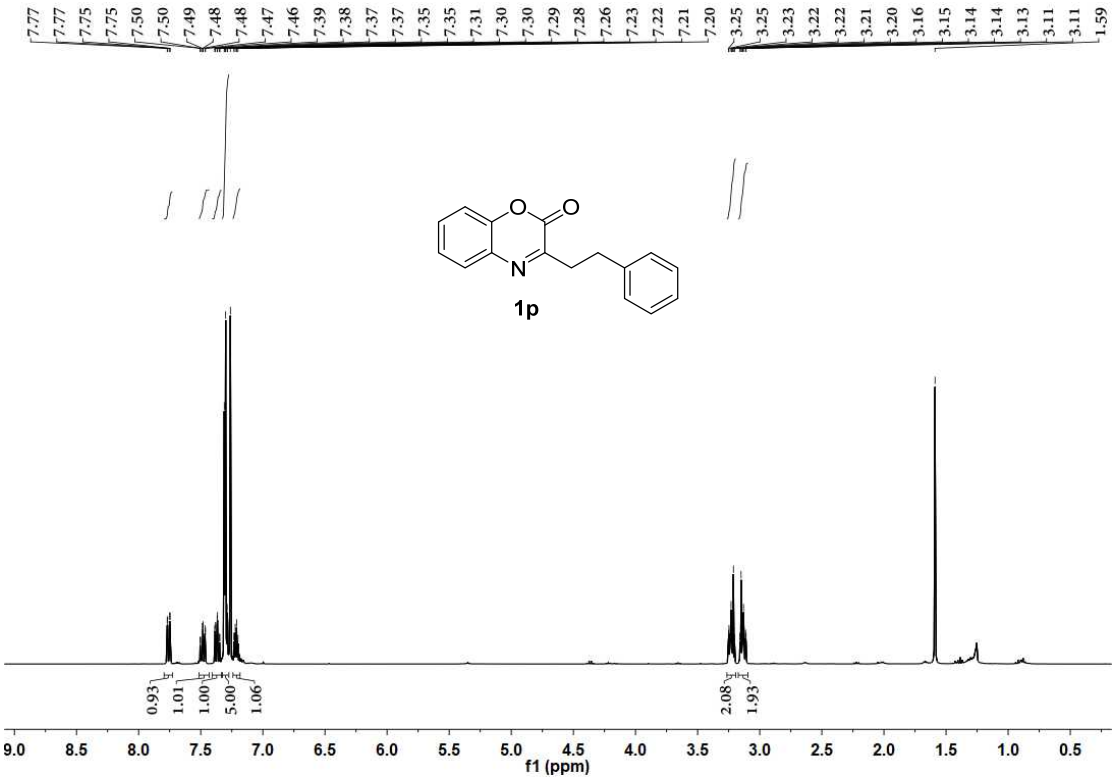

155.11
153.24
146.50
131.06
130.51
128.57
125.44
116.37
77.32
77.00
76.68
21.32
O
O
N
Me
1n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

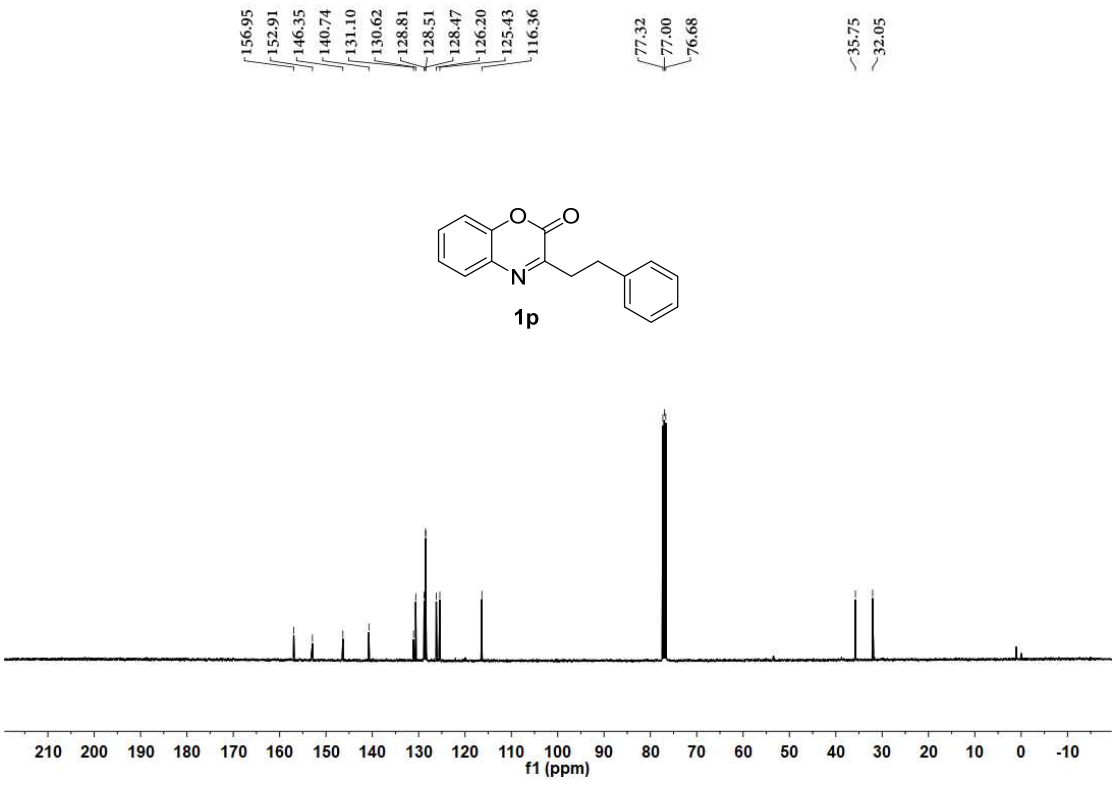

7.76
7.74
7.48
7.46
7.44
7.36
7.34
7.33
7.28
7.27
7.26
3.49
3.47
3.45
3.44
3.42
1.34
1.32
1.00
1.05
1.02
1.13
1.00
6.07
f1 (ppm)

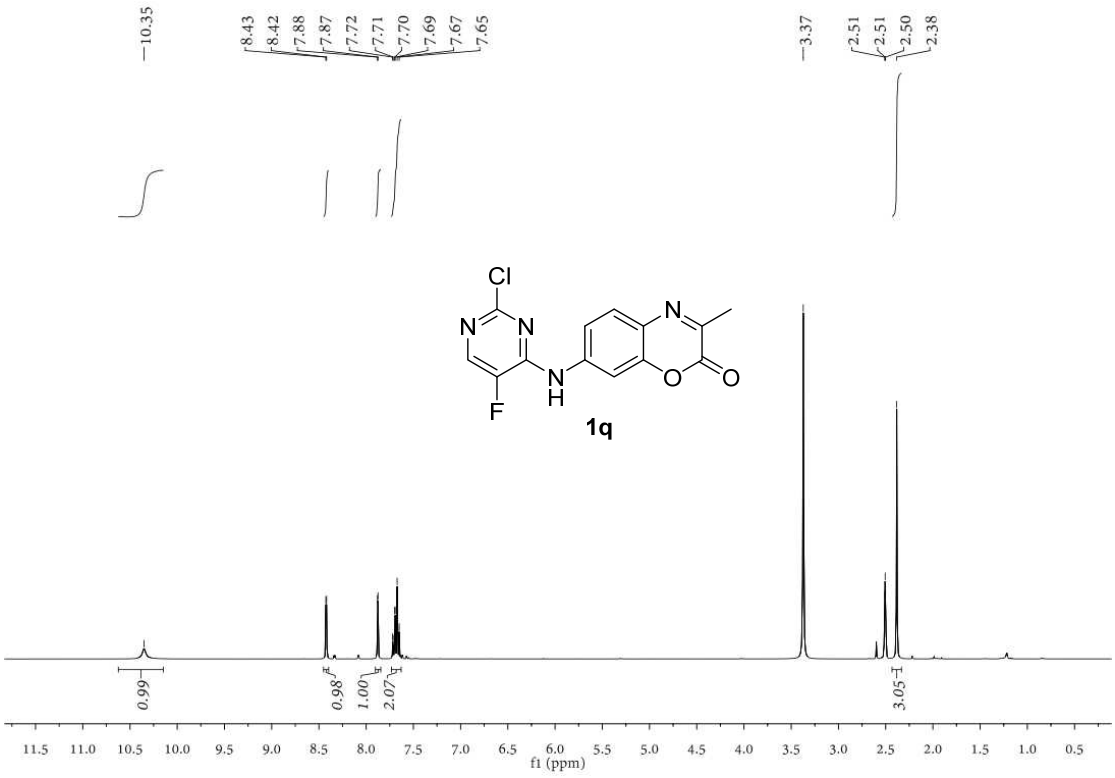
1o

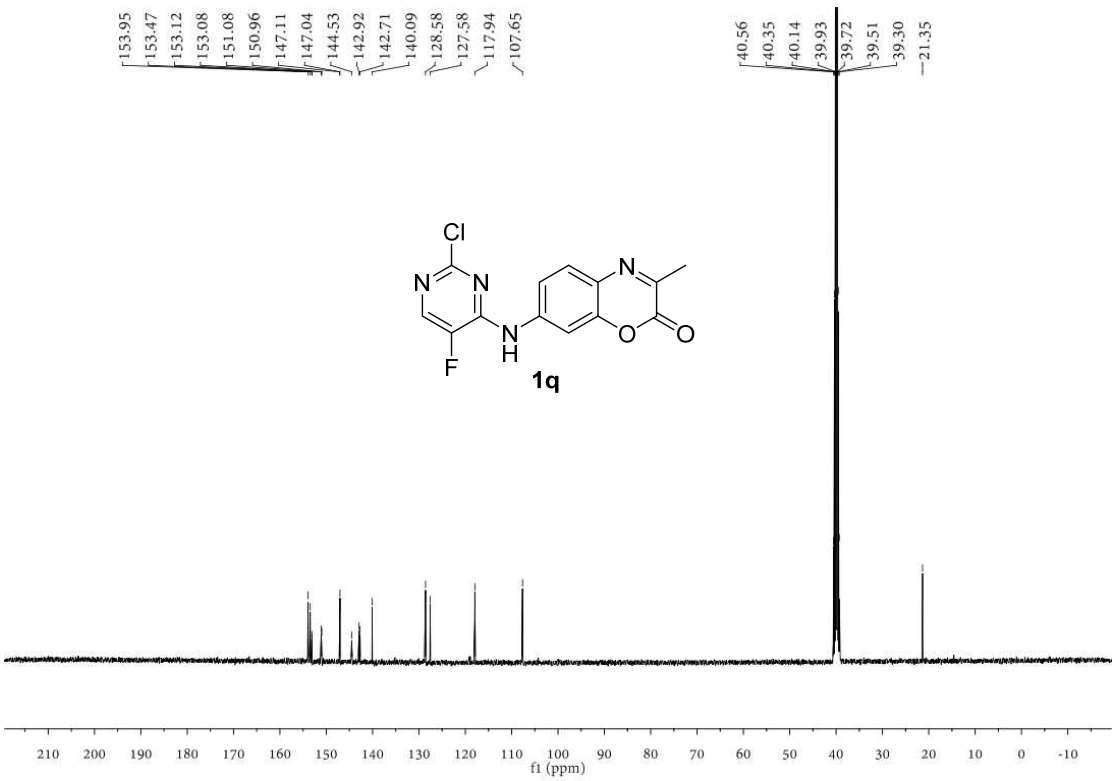

161.90
152.51
146.22
131.16
130.34
128.86
125.23
116.20
77.32
77.00
76.68
31.92
19.82
f1 (ppm)

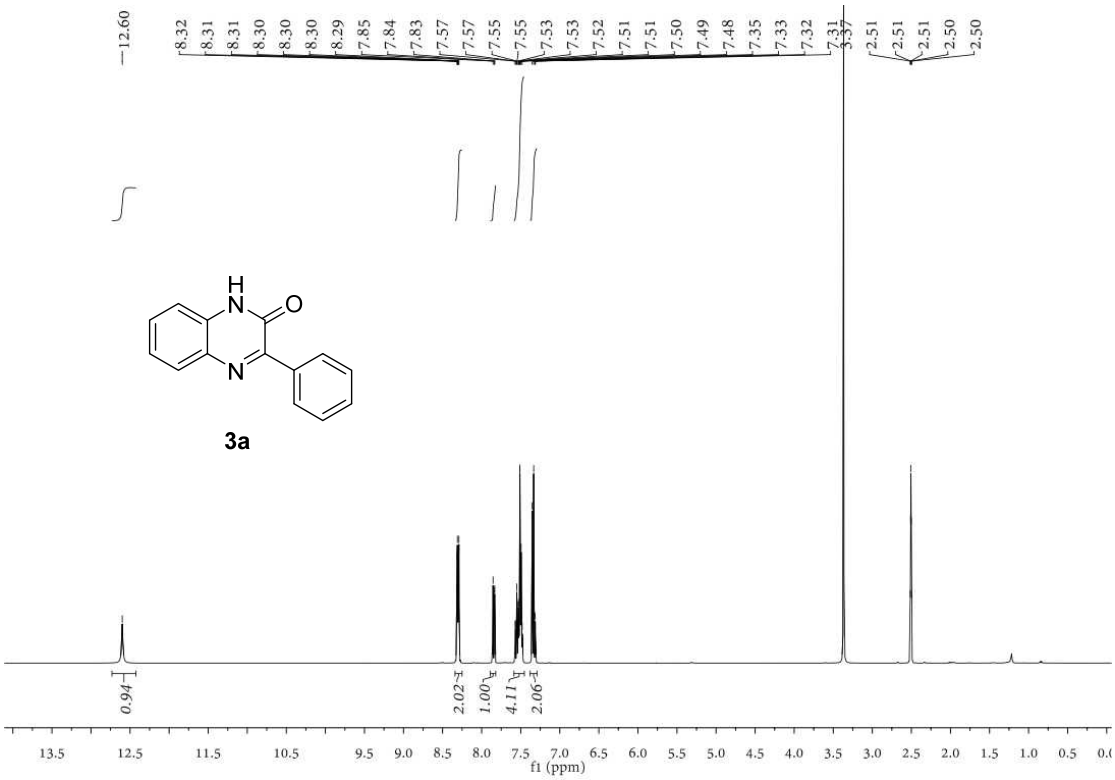
1o


7.77
7.77
7.75
7.75
7.50
7.50
7.49
7.48
7.48
7.47
7.46
7.39
7.38
7.37
7.37
7.35
7.35
7.31
7.30
7.30
7.29
7.28
7.26
7.23
7.22
7.21
7.20
3.25
3.25
3.23
3.22
3.22
3.21
3.20
3.16
3.15
3.14
3.14
3.13
3.11
3.11
1.59
O
O
N
1p
0.93
1.01
1.00
5.00
1.06
2.08
1.93
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

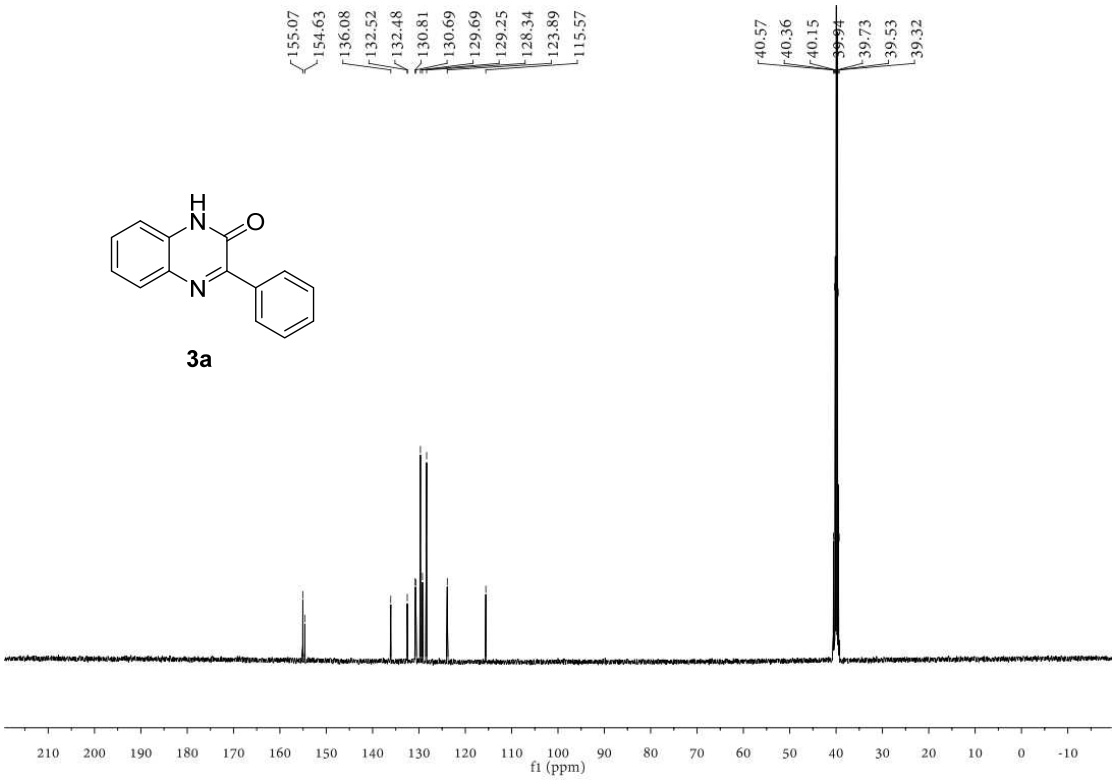

156.95
152.91
146.35
140.74
131.10
130.62
128.81
128.51
128.47
126.20
125.43
116.36
77.32
77.00
76.68
35.75
32.05
O
O
N
1p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


10.35
8.43
8.42
7.88
7.87
7.72
7.71
7.70
7.69
7.67
7.65
3.37
2.51
2.51
2.50
2.38
Cl
N
N
N
H
F
O
O
1q
0.99
0.98
1.00
2.07
3.05
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

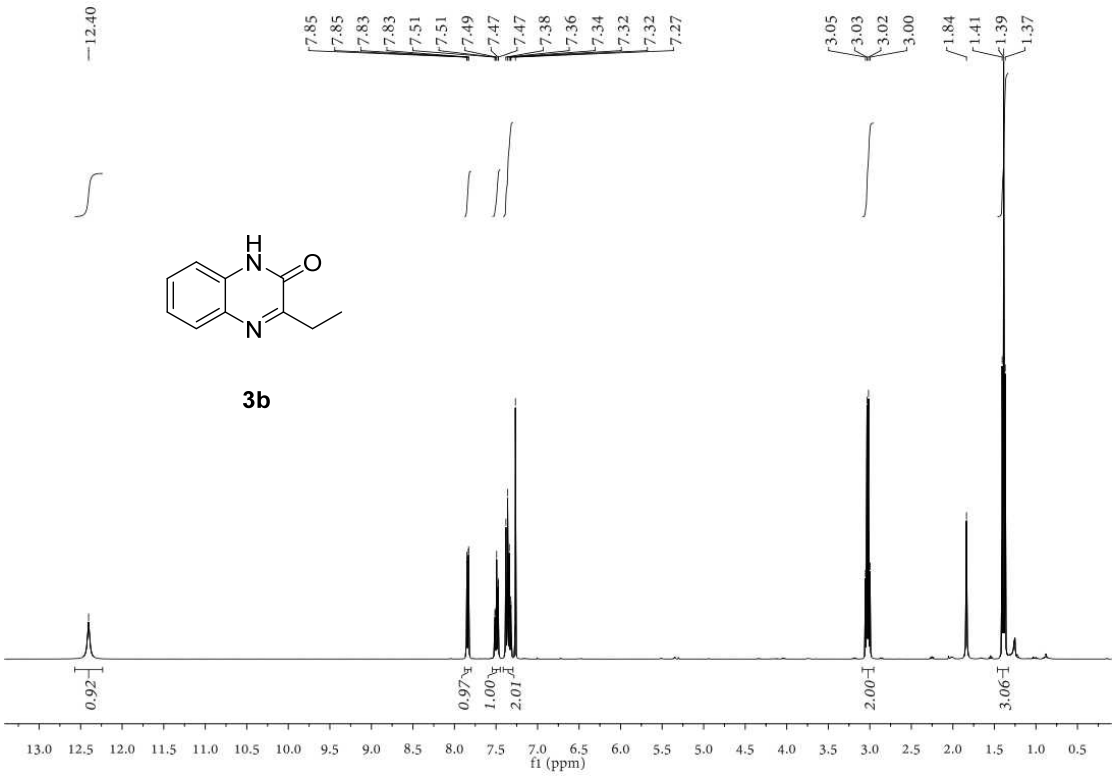

153.95
153.47
153.12
153.08
151.08
150.96
147.11
147.04
144.53
142.92
142.71
140.09
128.58
127.58
117.94
107.65
40.56
40.35
40.14
39.93
39.72
39.51
39.30
21.35
Cl
N
N
N
H
F
O
O
1q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


12.60
8.32
8.31
8.31
8.30
8.30
8.30
8.29
7.85
7.84
7.83
7.57
7.57
7.55
7.55
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.48
7.35
7.33
7.32
7.31
3.37
2.51
2.51
2.51
2.50
2.50
0.94
2.02
1.00
4.11
2.06
f1 (ppm)

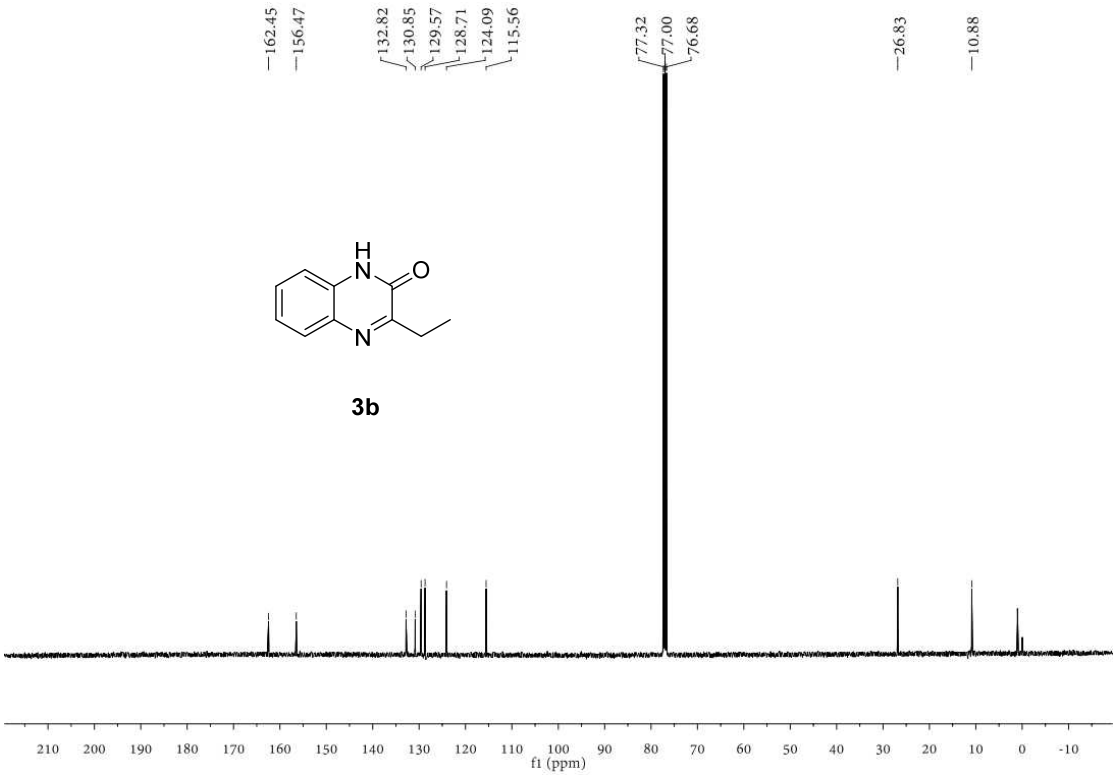
3a


155.07
154.63
136.08
132.52
132.48
130.81
130.69
129.69
129.25
128.34
123.89
115.57
40.57
40.36
40.15
39.94
39.73
39.53
39.32
f1 (ppm)

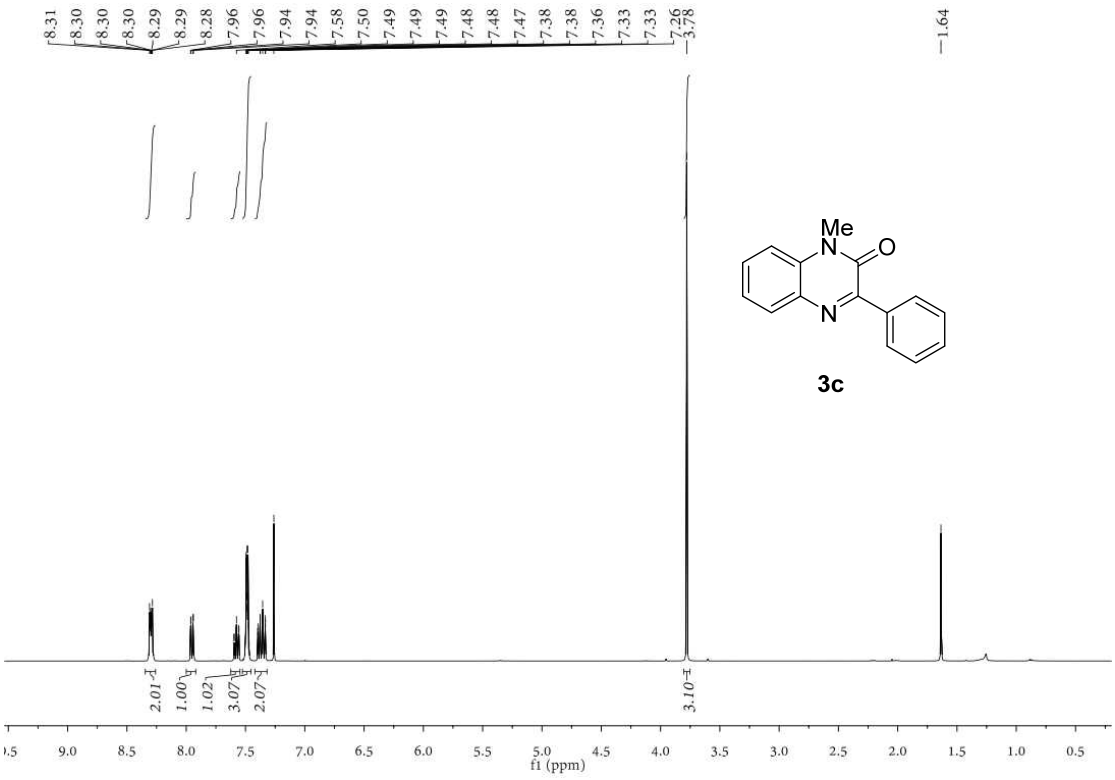
3a

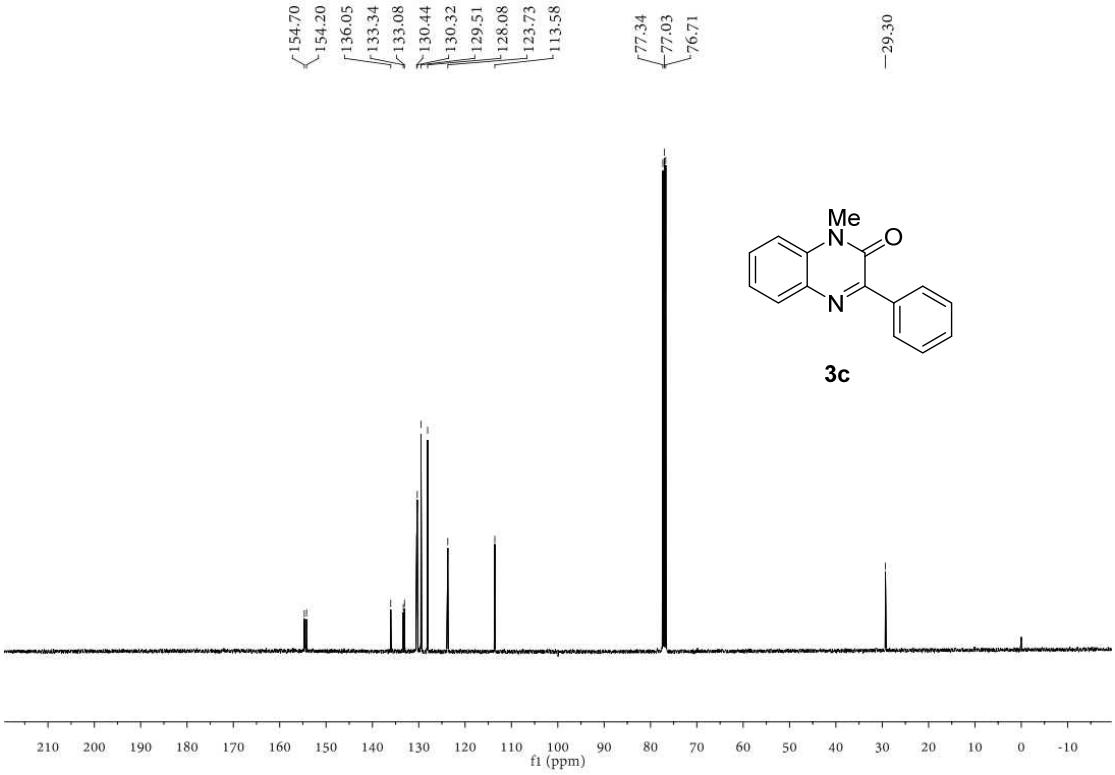

12.40
7.85
7.85
7.83
7.83
7.51
7.51
7.49
7.47
7.47
7.38
7.36
7.34
7.32
7.32
7.27
3.05
3.03
3.02
3.00
1.84
1.41
1.39
1.37
0.92
0.97
1.00
2.01
2.00
3.06
f1 (ppm)

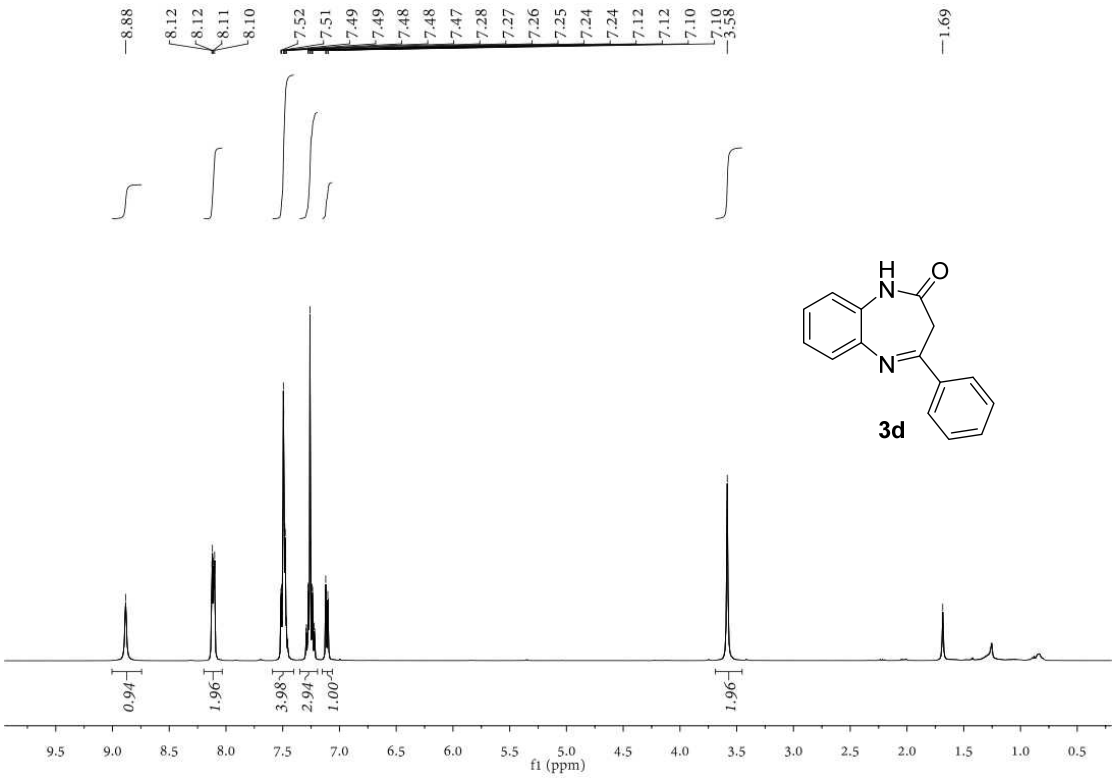
3b

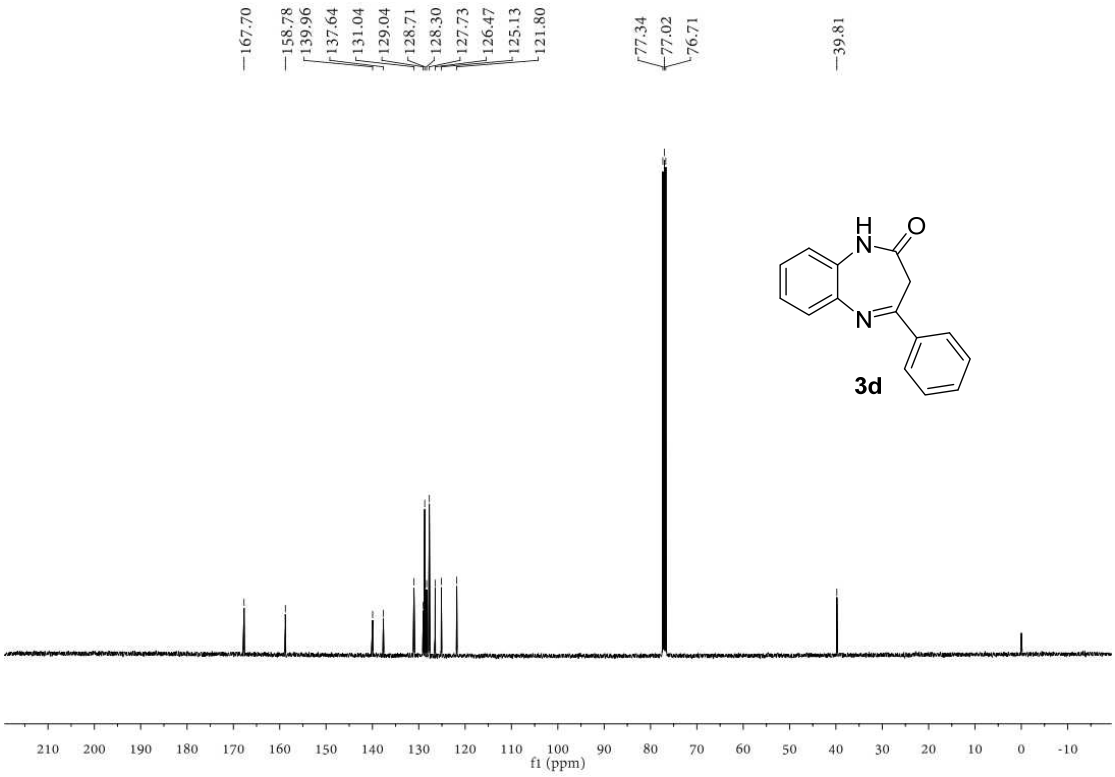

162.45
156.47
132.82
130.85
129.57
128.71
124.09
115.56
77.32
77.00
76.68
26.83
10.88
f1 (ppm)

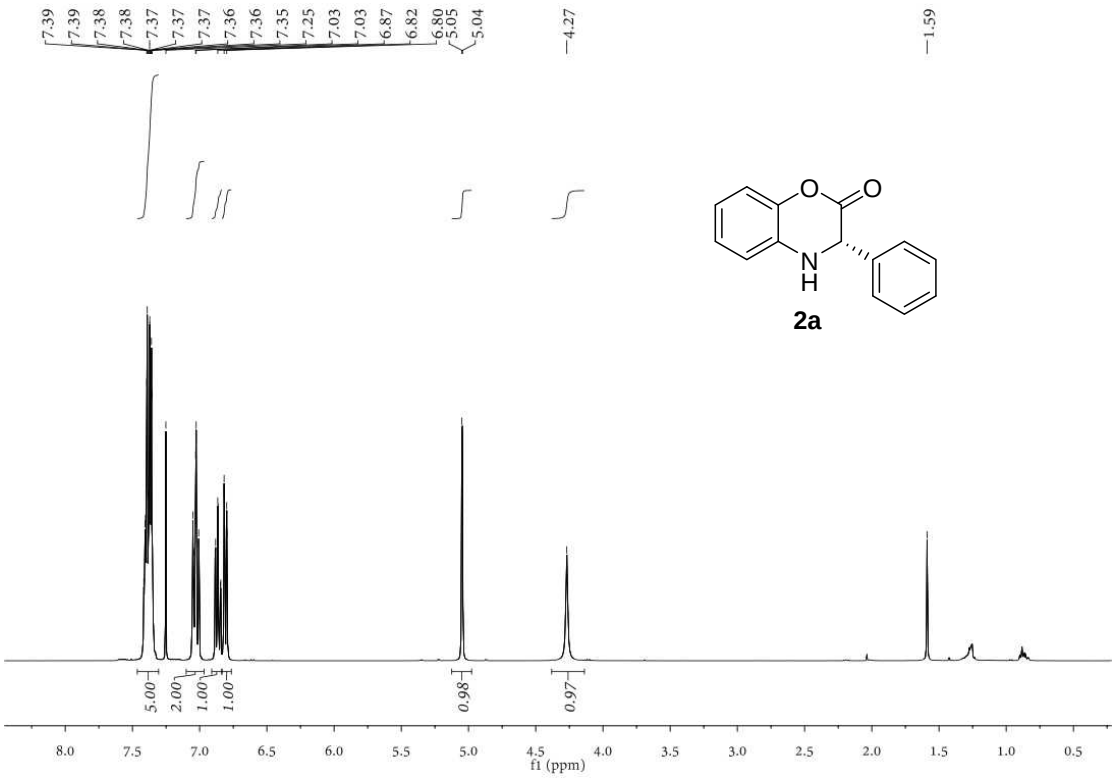
3b

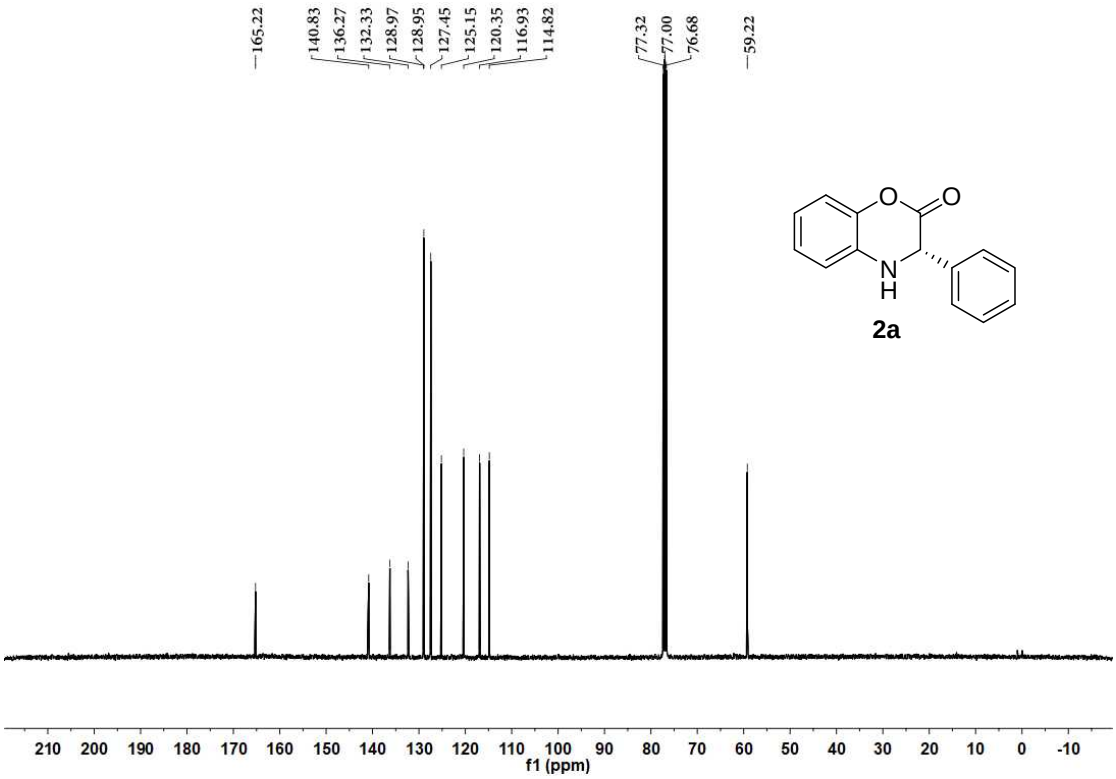

8.31
8.30
8.30
8.30
8.29
8.29
8.28
7.96
7.96
7.94
7.94
7.58
7.50
7.49
7.49
7.49
7.48
7.48
7.47
7.38
7.38
7.36
7.33
7.33
7.26
3.78
1.64
Me
N
O
N
3c
2.01
1.00
1.02
3.07
2.07
3.10
f1 (ppm)

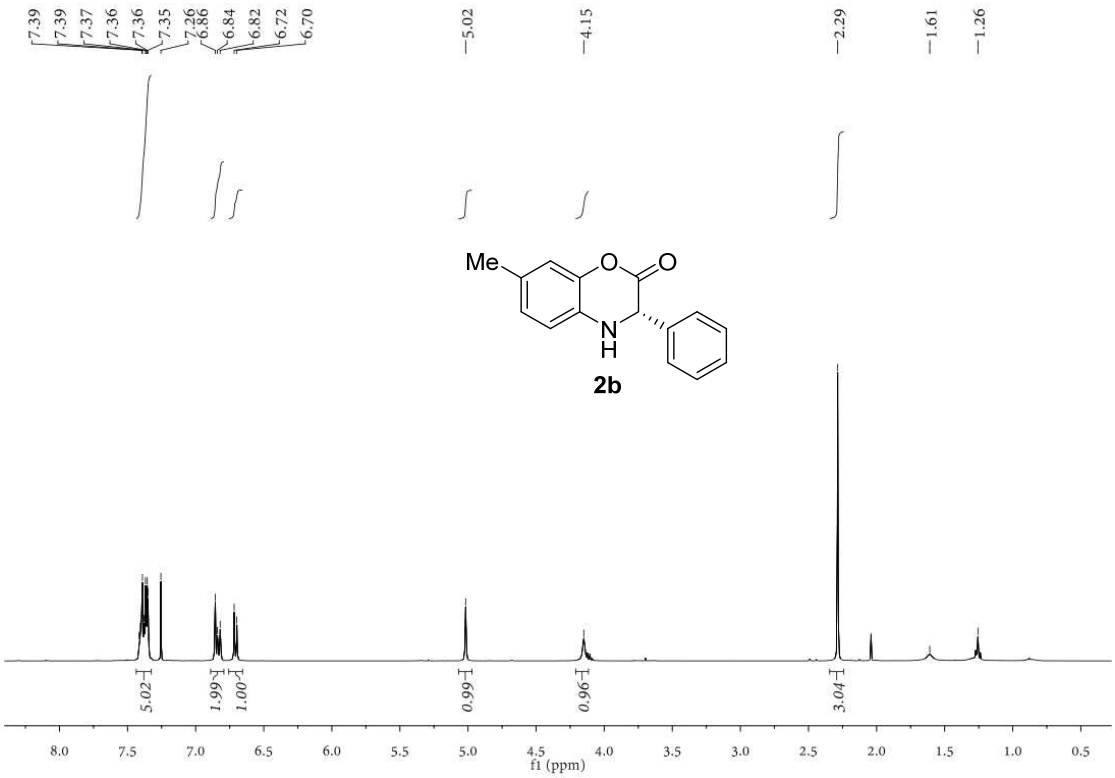

154.70
154.20
136.05
133.34
133.08
130.44
130.32
129.51
128.08
123.73
113.58
77.34
77.03
76.71
29.30
Me
N
O
N
3c
f1 (ppm)

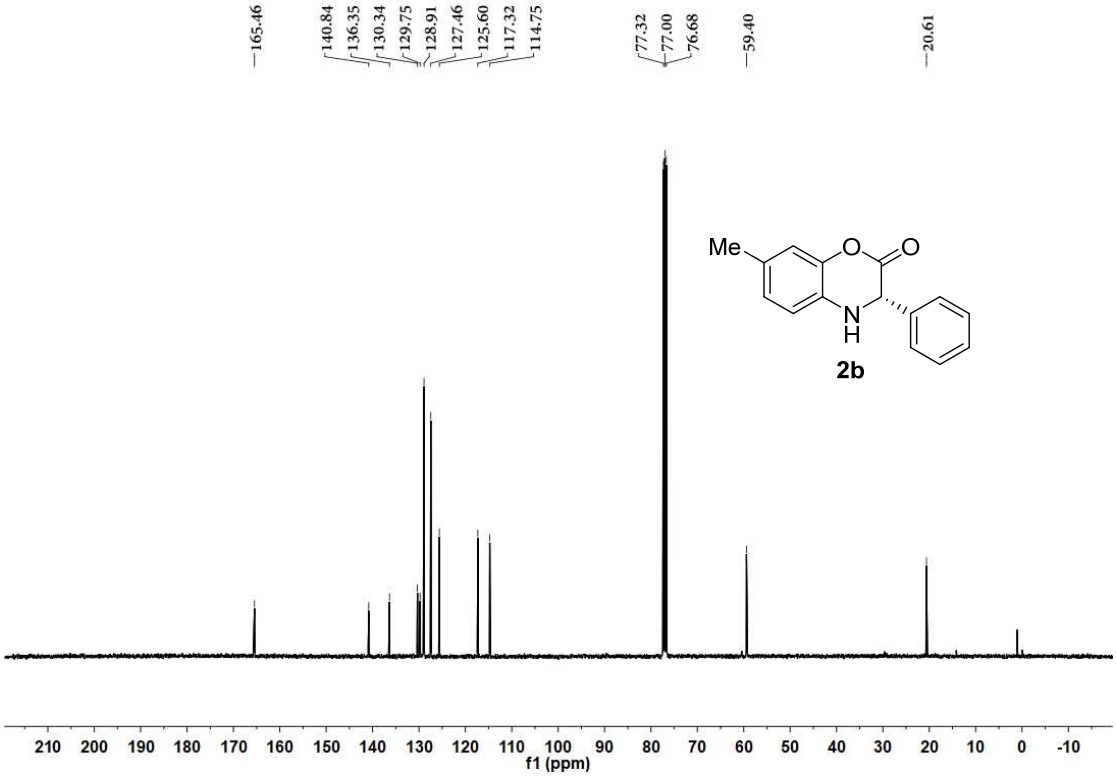

8.88
8.12
8.12
8.11
8.10
7.52
7.51
7.49
7.49
7.48
7.48
7.47
7.28
7.27
7.26
7.25
7.24
7.24
7.12
7.12
7.10
7.10
3.58
1.69
0.94
1.96
3.98
2.94
1.00
1.96
f1 (ppm)
3d

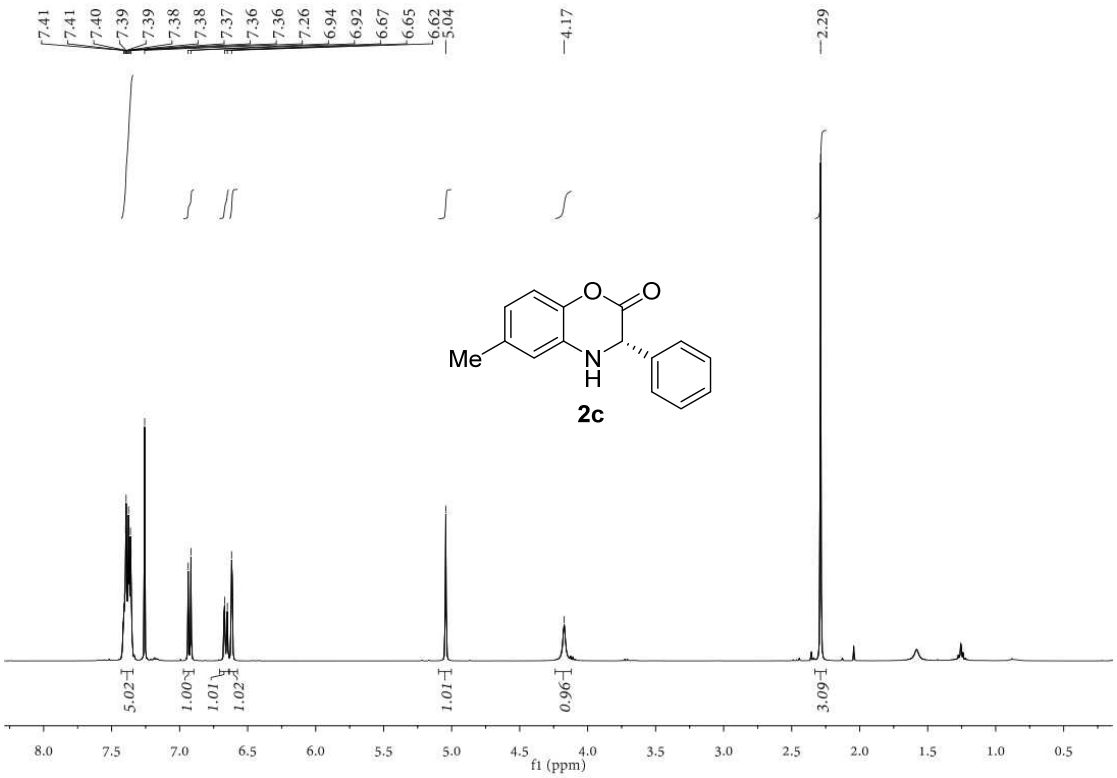

167.70
158.78
139.96
137.64
131.04
129.04
128.71
128.30
127.73
126.47
125.13
121.80
77.34
77.02
76.71
39.81
f1 (ppm)
3d

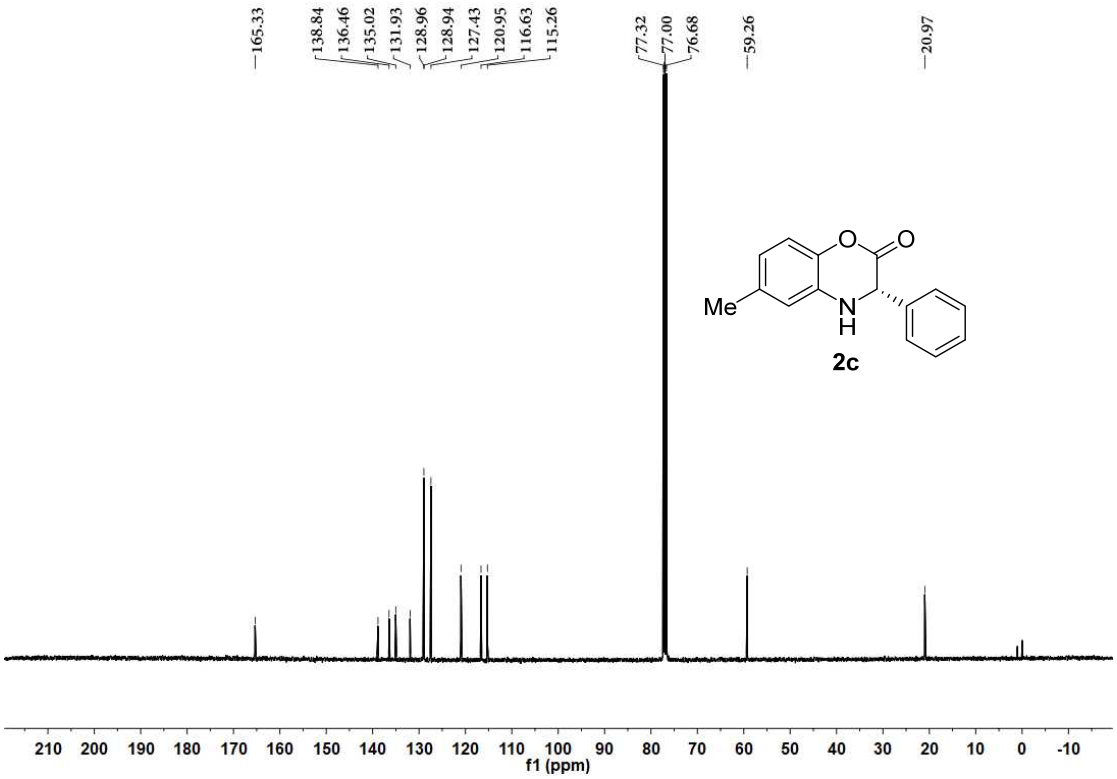

7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
7.25
7.03
7.03
6.87
6.82
6.80
5.05
5.04
4.27
1.59
O
O
N
H
2a
5.00
2.00
1.00
1.00
0.98
0.97
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

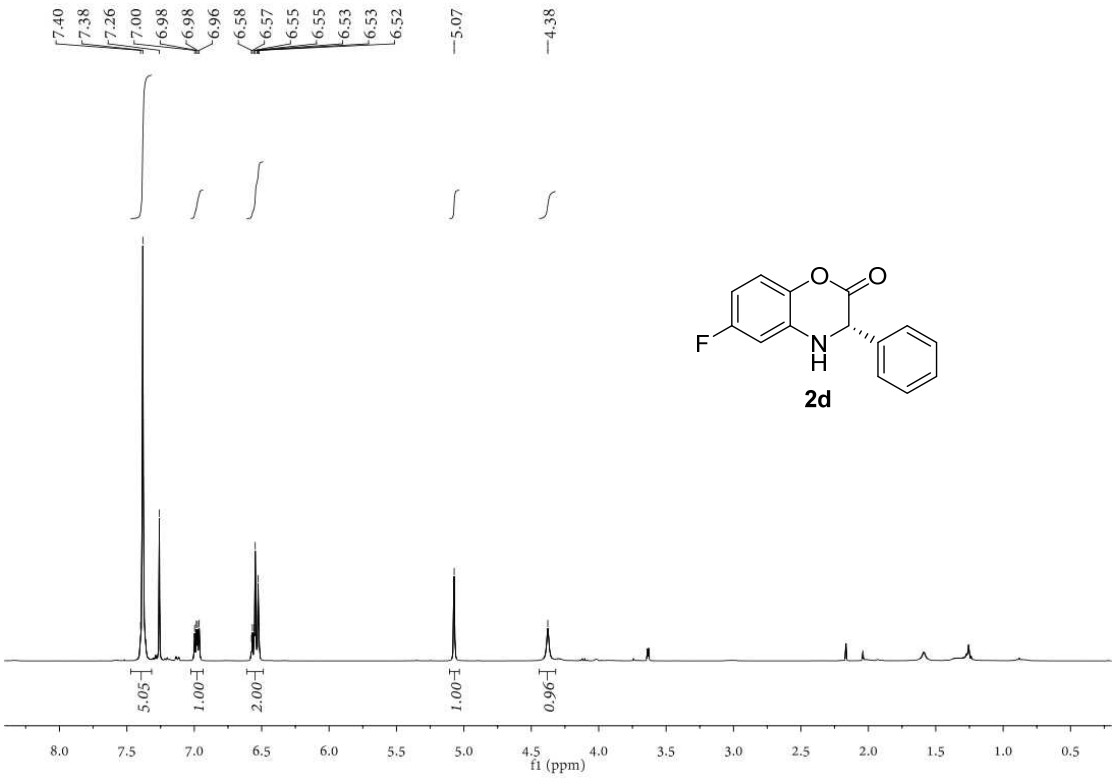

165.22
140.83
136.27
132.33
128.97
128.95
127.45
125.15
120.35
116.93
114.82
77.32
77.00
76.68
59.22
O
O
N
H
2a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.39
7.39
7.37
7.36
7.36
7.35
7.26
6.86
6.84
6.82
6.72
6.70
5.02
4.15
2.29
1.61
1.26
Me
O
O
N
H
2b
5.02
1.99
1.00
0.99
0.96
3.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

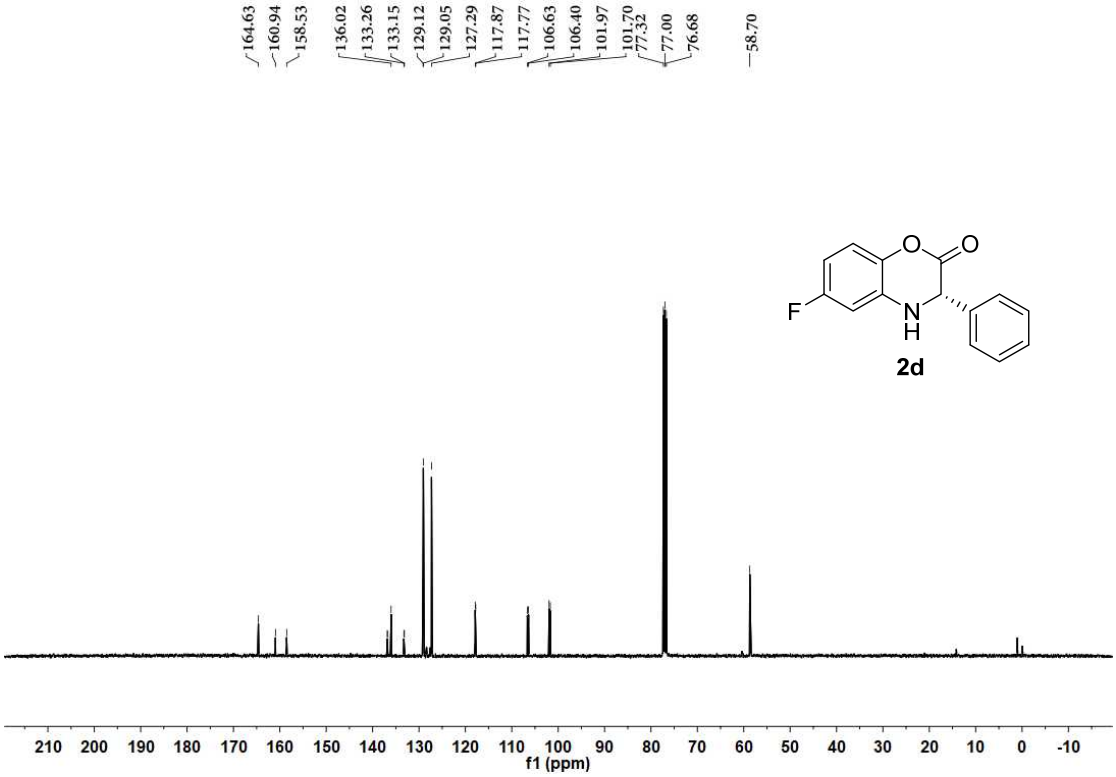

165.46
140.84
136.35
130.34
129.75
128.91
127.46
125.60
117.32
114.75
77.32
77.00
76.68
59.40
20.61
Me
O
O
N
H
2b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.41
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.36
7.36
7.26
6.94
6.92
6.67
6.65
6.62
5.04
4.17
2.29
Me
O
O
N
H
2c
5.02
1.00
1.01
1.02
1.01
0.96
3.09
f1 (ppm)

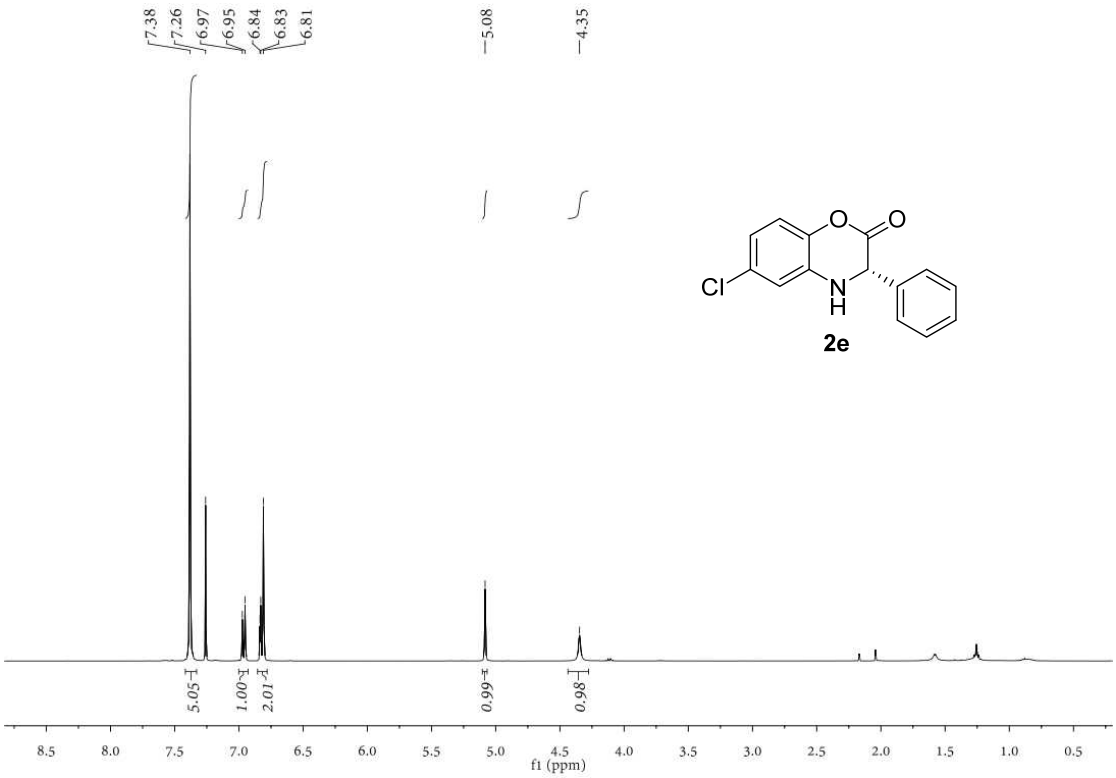

165.33
138.84
136.46
135.02
131.93
128.96
128.94
127.43
120.95
116.63
115.26
77.32
77.00
76.68
59.26
20.97
Me
O
O
N
H
2c
f1 (ppm)


7.40
7.38
7.26
7.00
6.98
6.98
6.96
6.58
6.57
6.55
6.55
6.53
6.53
6.52
5.07
4.38
5.05
1.00
2.00
1.00
0.96
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
F
O
O
N
H

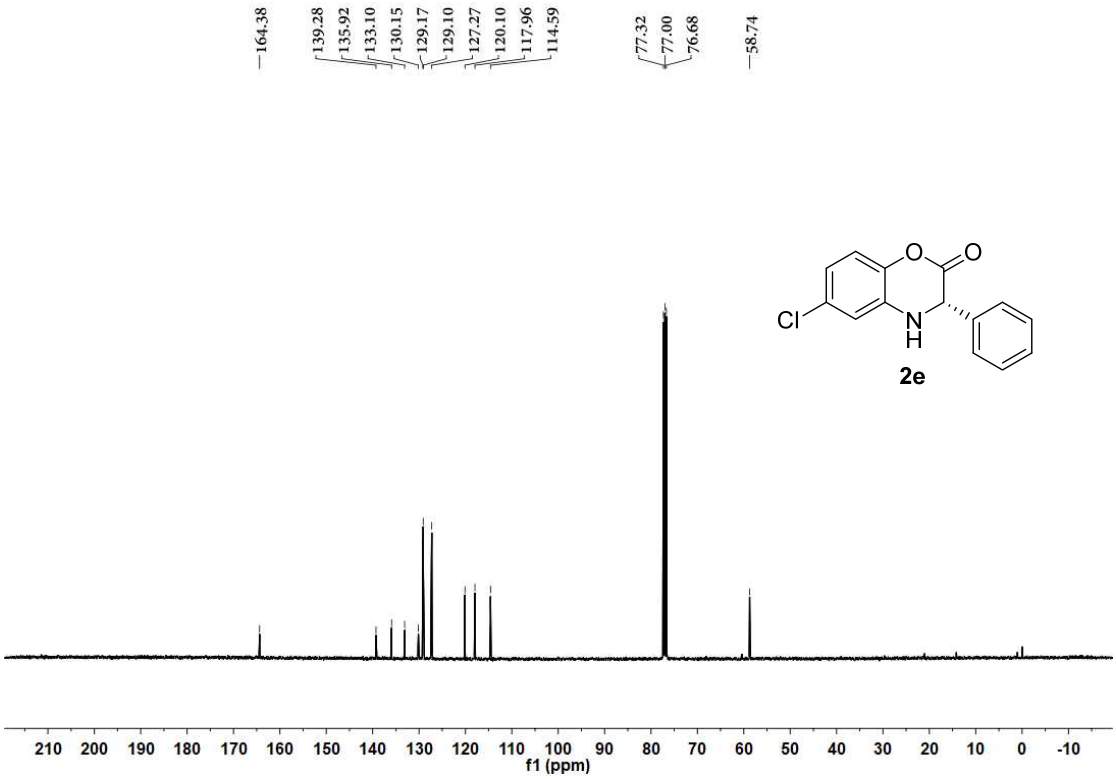
2d


164.63
160.94
158.53
136.02
133.26
133.15
129.12
129.05
127.29
117.87
117.77
106.63
106.40
101.97
101.70
77.32
77.00
76.68
58.70
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
F
O
O
N
H

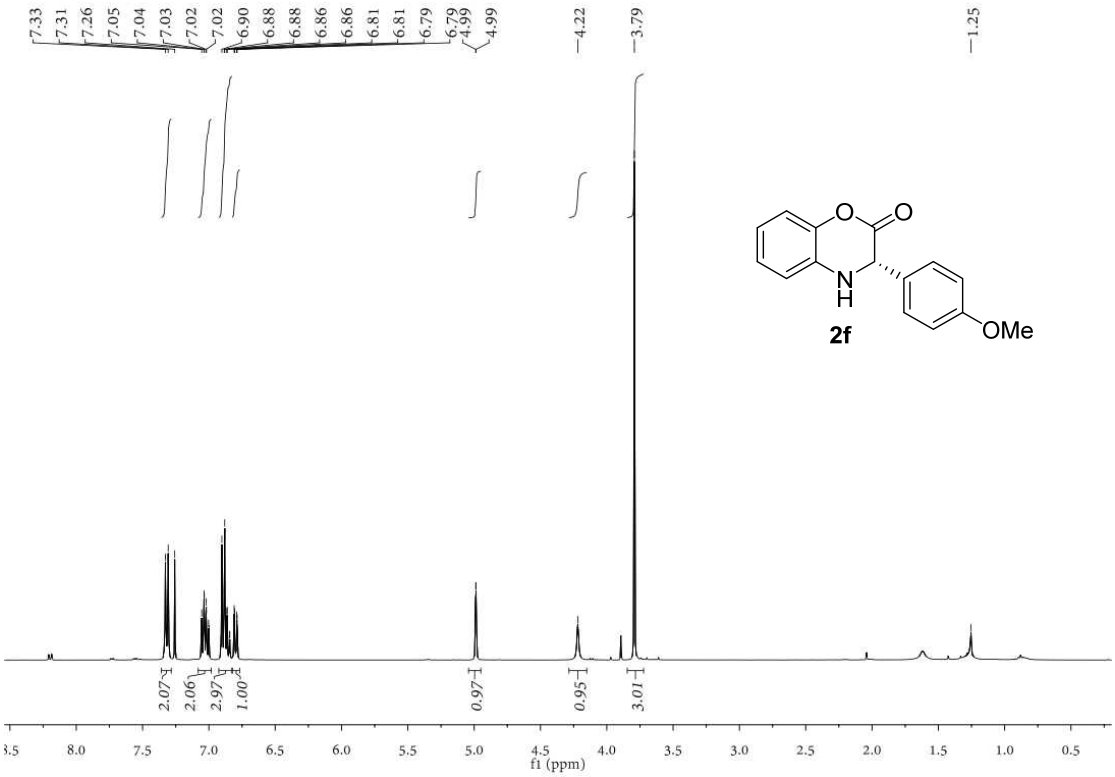
2d

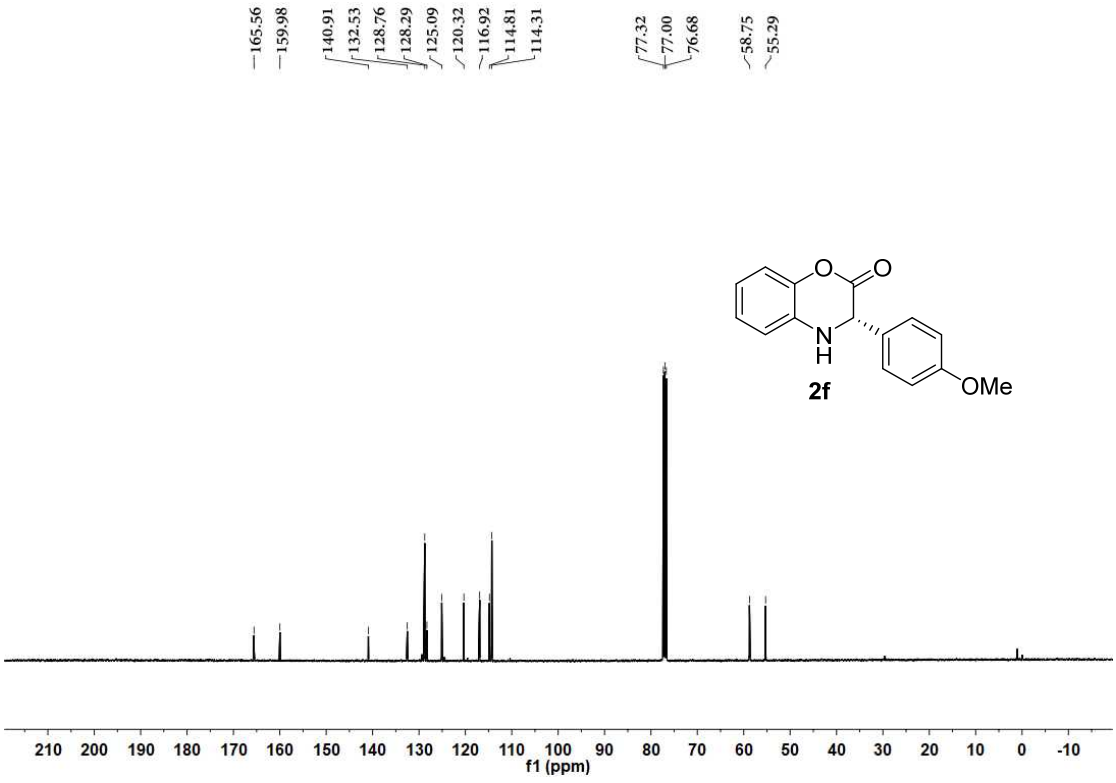

7.38
7.26
6.97
6.95
6.84
6.83
6.81
5.08
4.35
5.05
1.00
2.01
0.99
0.98
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
O
O
Cl
N
H

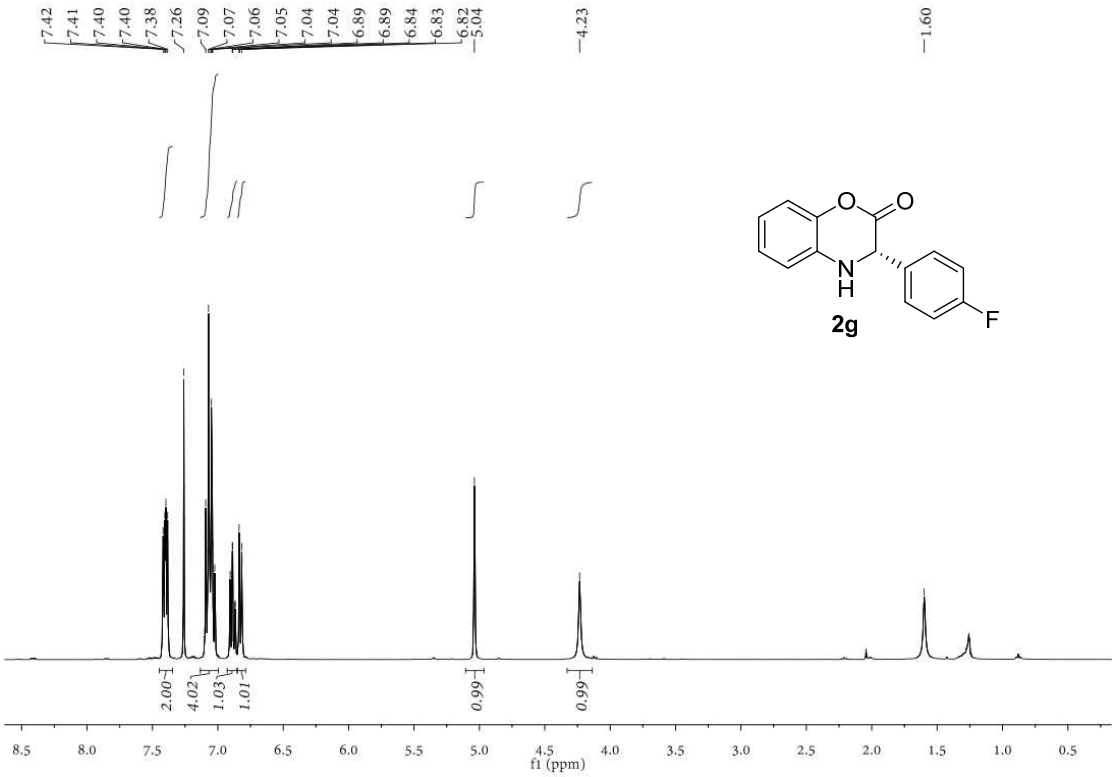
2e

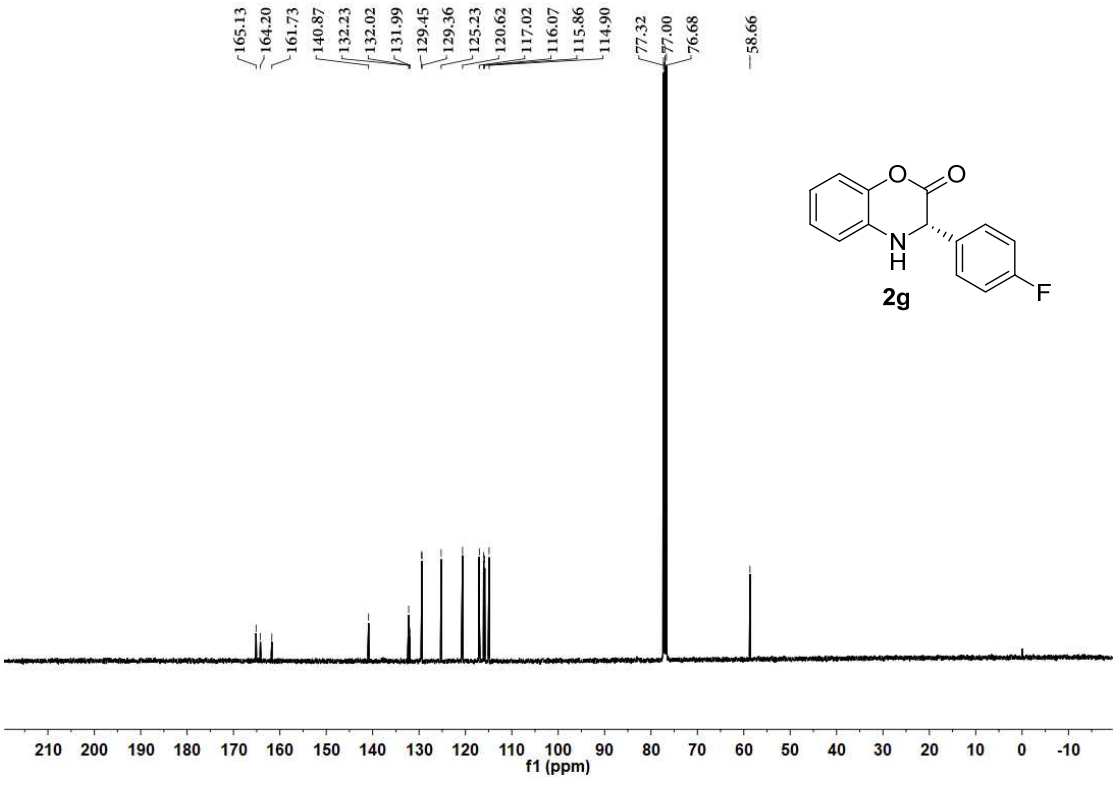

164.38
139.28
135.92
133.10
130.15
129.17
129.10
127.27
120.10
117.96
114.59
77.32
77.00
76.68
58.74
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
O
O
Cl
N
H

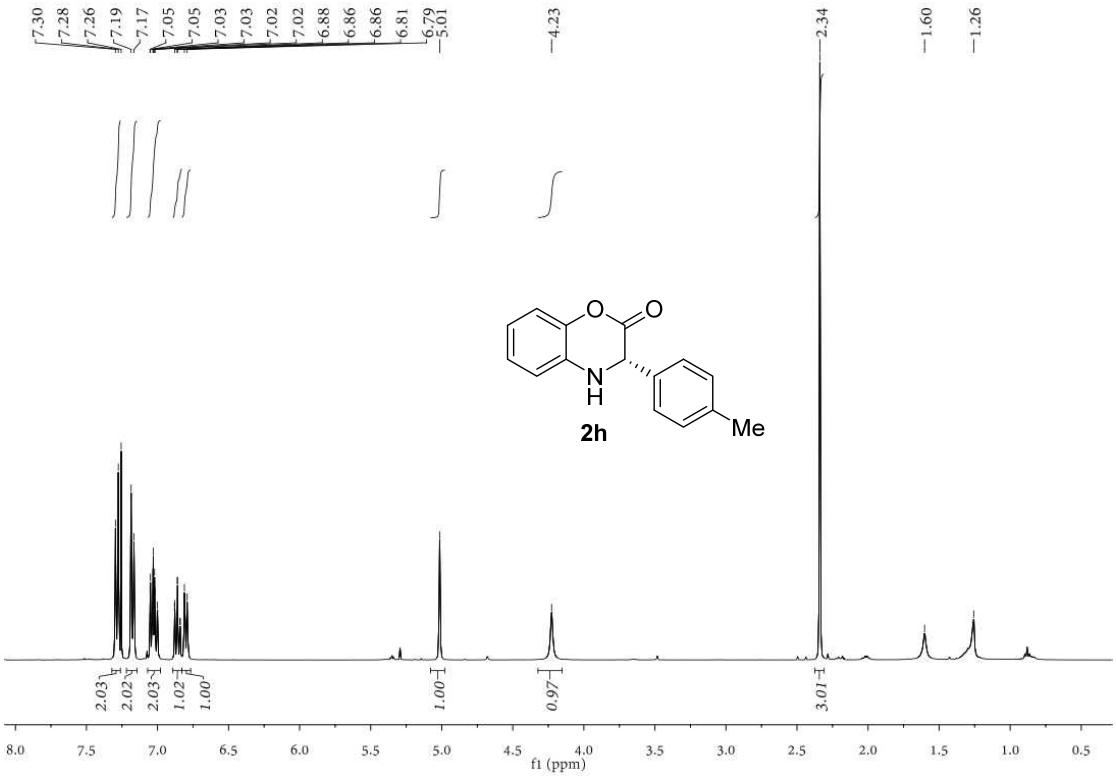
2e

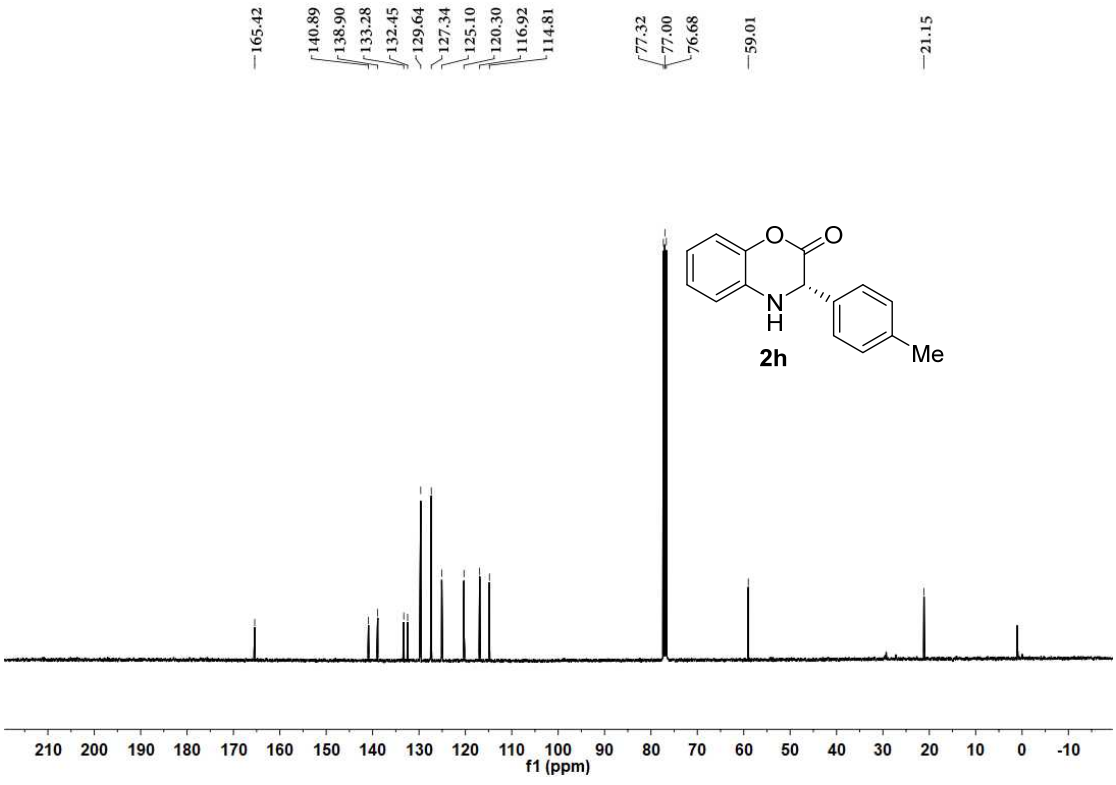

7.33
7.31
7.26
7.05
7.04
7.03
7.02
7.02
6.90
6.88
6.88
6.86
6.86
6.81
6.81
6.79
6.79
4.99
4.99
4.22
3.79
1.25
2.07
2.06
2.97
1.00
0.97
0.95
3.01
f1 (ppm)
2f
OMe

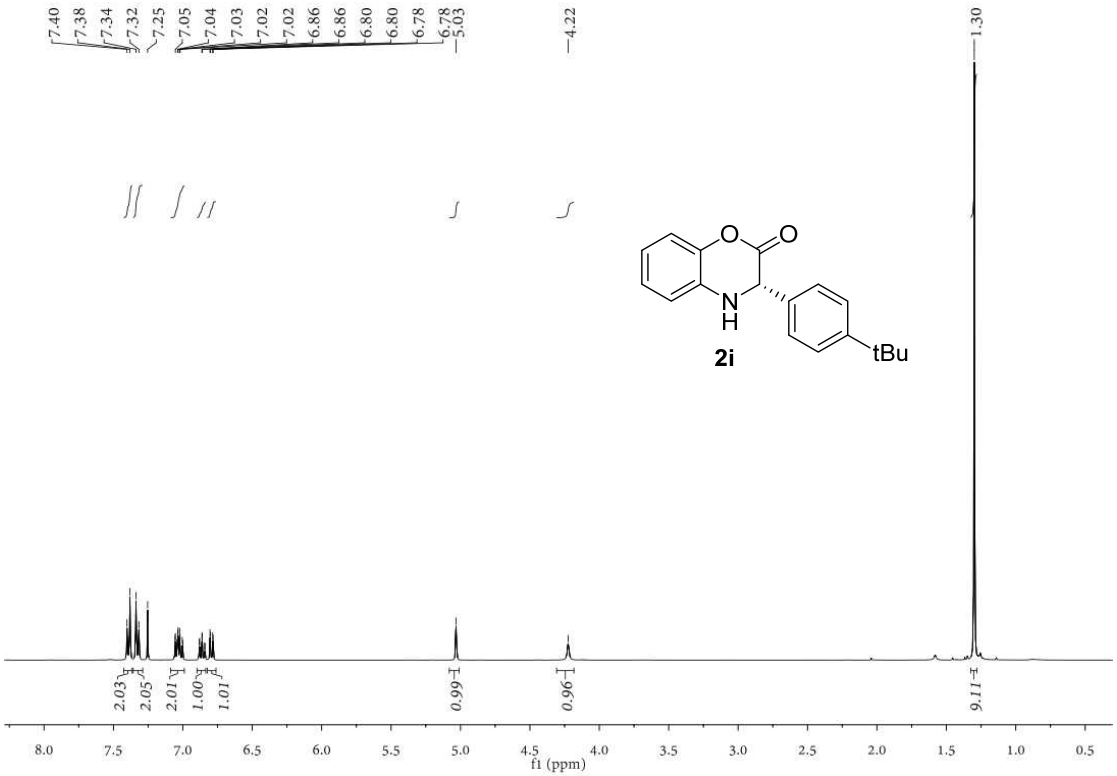

165.56
159.98
140.91
132.53
128.76
128.29
125.09
120.32
116.92
114.81
114.31
77.32
77.00
76.68
58.75
55.29
f1 (ppm)
2f
OMe

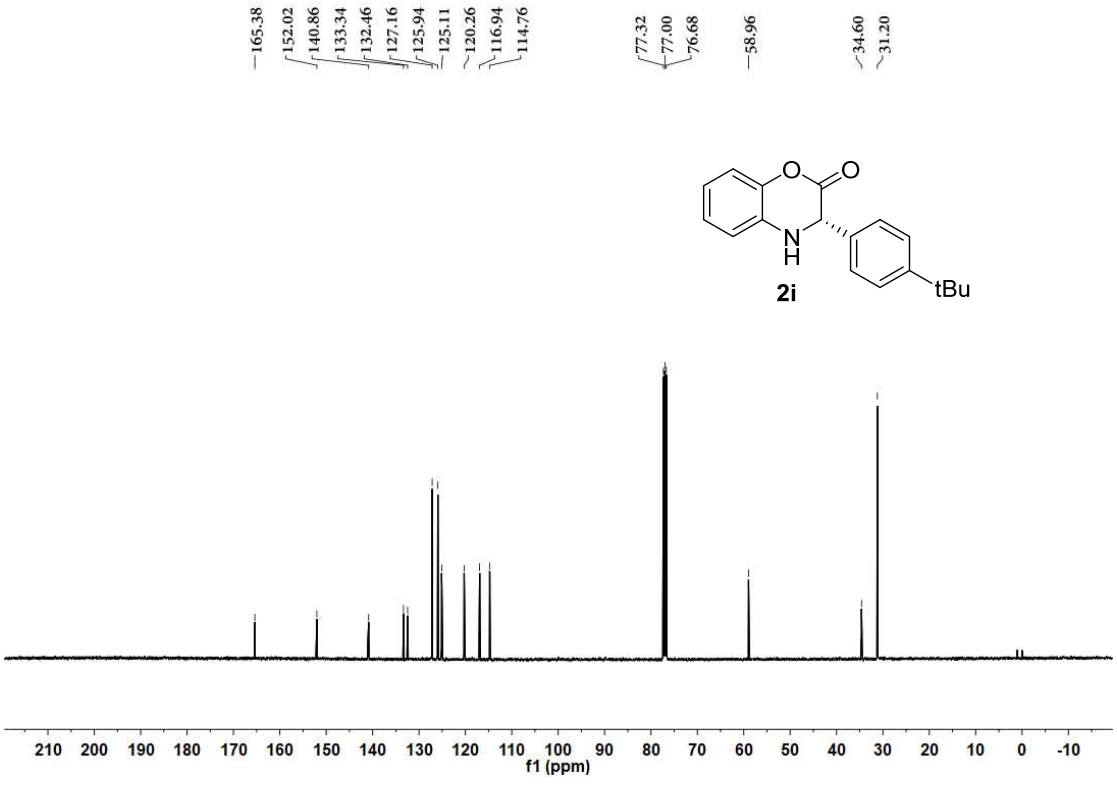

7.42
7.41
7.40
7.40
7.38
7.26
7.09
7.07
7.06
7.05
7.04
7.04
6.89
6.89
6.84
6.83
6.82
5.04
4.23
1.60
2.00
4.02
1.03
1.01
0.99
0.99
f1 (ppm)
O
O
N
H
F
2g

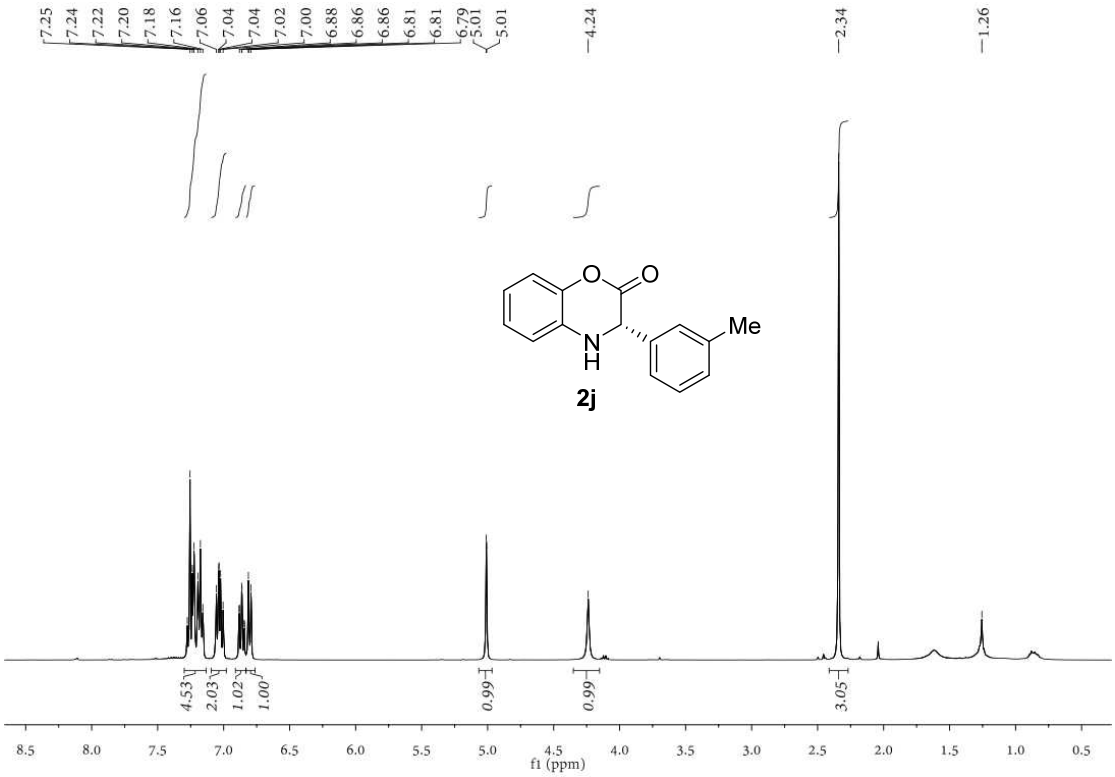

165.13
164.20
161.73
140.87
132.23
132.02
131.99
129.45
129.36
125.23
120.62
117.02
116.07
115.86
114.90
77.32
77.00
76.68
58.66
f1 (ppm)
O
O
N
H
F
2g

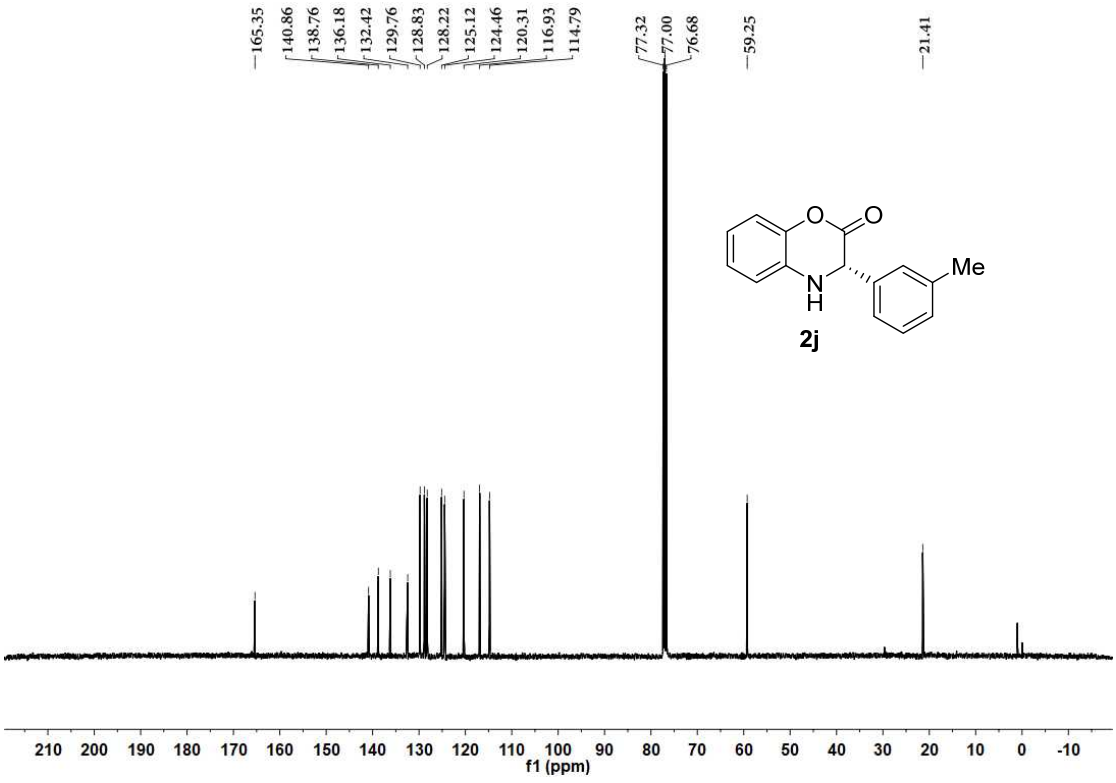

7.30
7.28
7.26
7.19
7.17
7.05
7.05
7.03
7.03
7.02
7.02
6.88
6.86
6.86
6.81
6.79
5.01
4.23
2.34
1.60
1.26
O
O
N
H
Me
2h
2.03
2.02
2.03
1.02
1.00
1.00
0.97
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

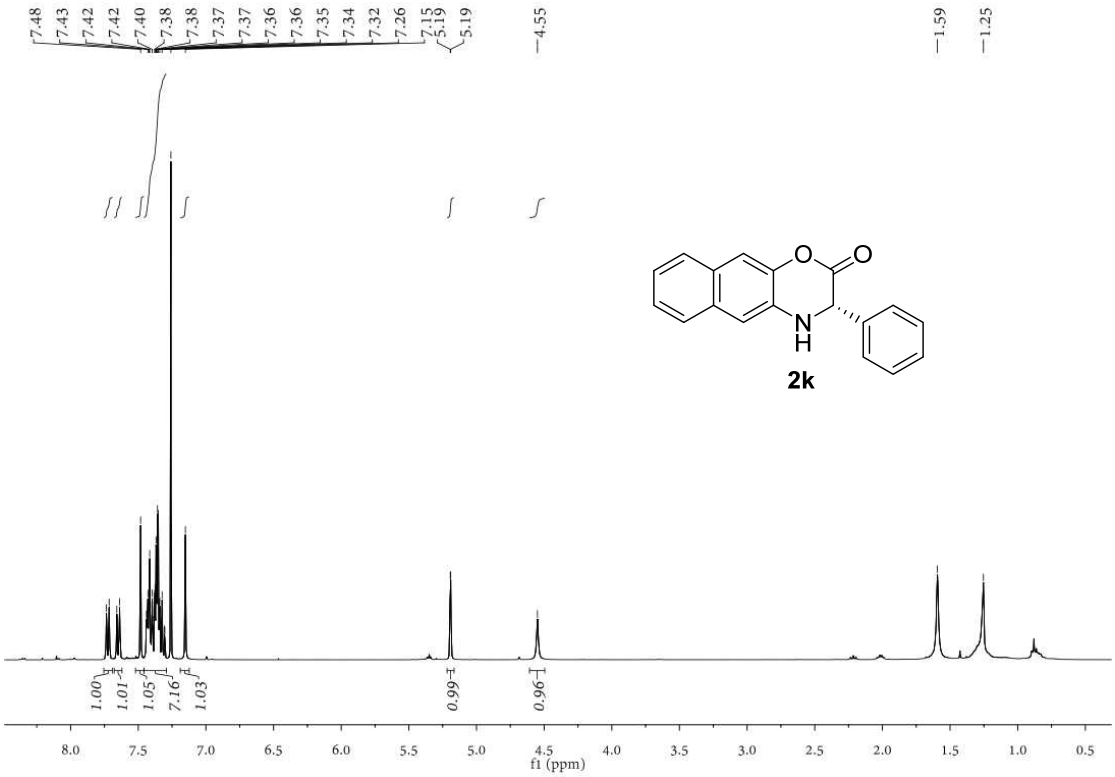

165.42
140.89
138.90
133.28
132.45
129.64
127.34
125.10
120.30
116.92
114.81
77.32
77.00
76.68
59.01
21.15
O
O
N
H
Me
2h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

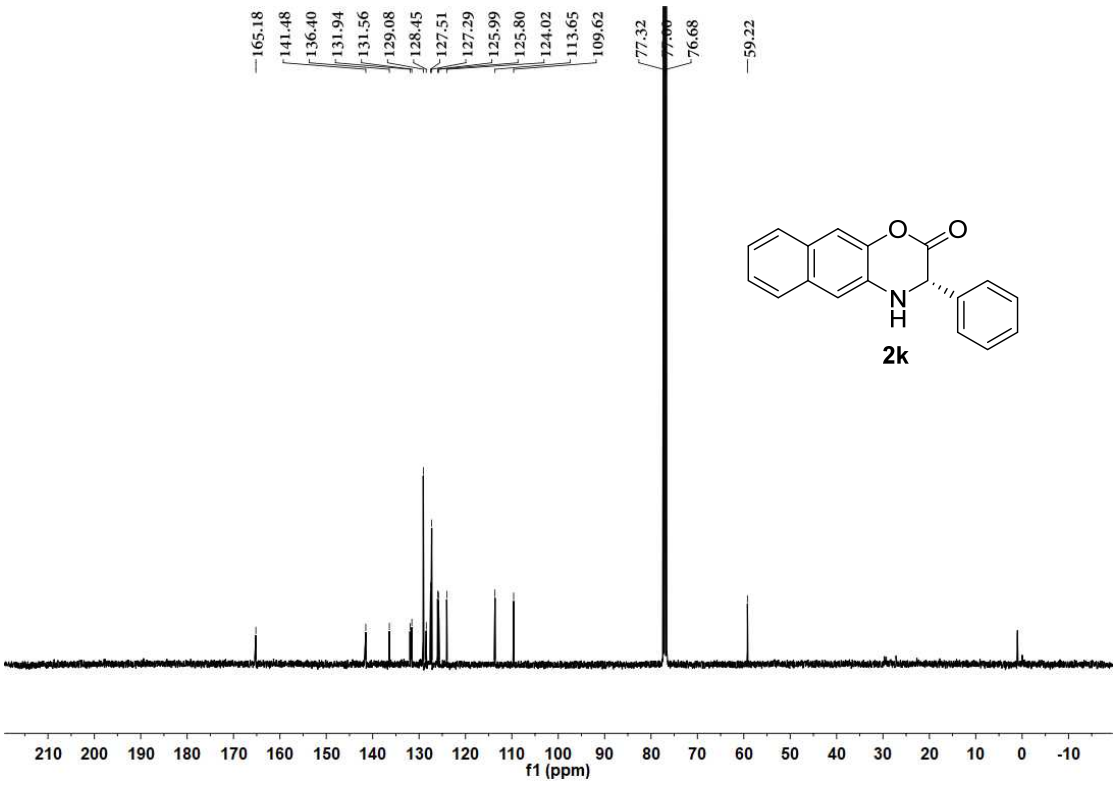

7.40
7.38
7.34
7.32
7.25
7.05
7.04
7.03
7.02
7.02
6.86
6.86
6.80
6.80
6.78
6.78
5.03
4.22
1.30
O
O
N
H
tBu
2i
2.03
2.05
2.01
1.00
1.01
0.99
0.96
9.11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

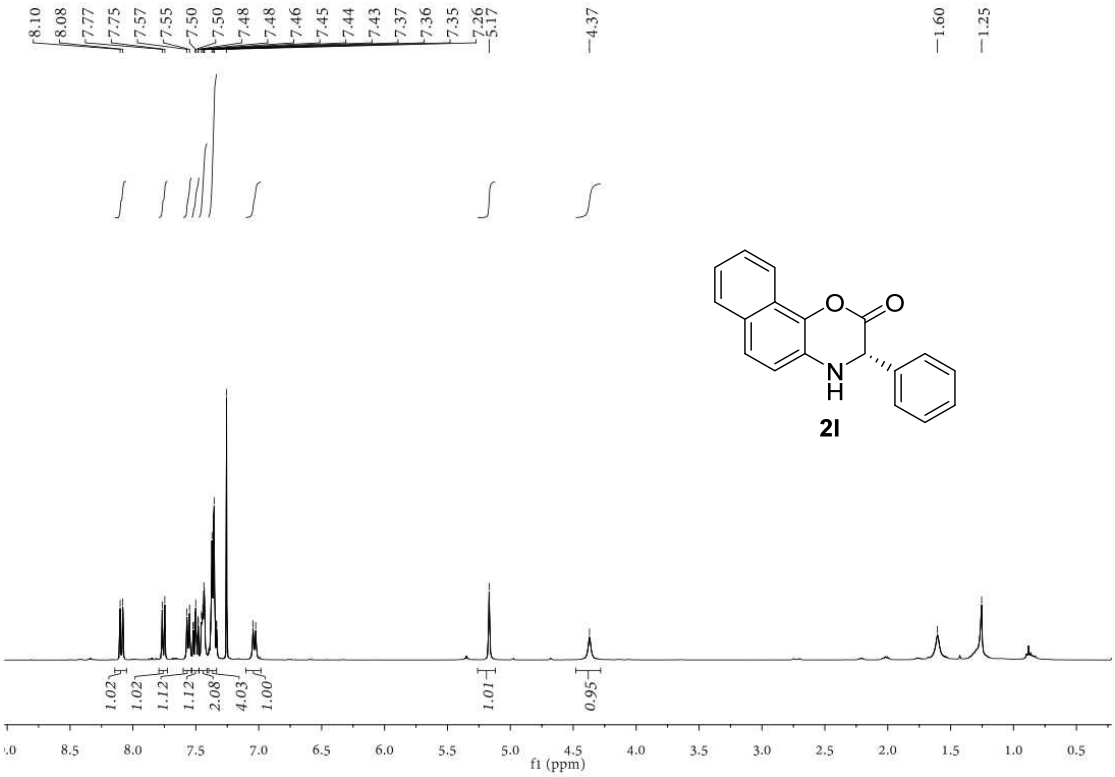

165.38
152.02
140.86
133.34
132.46
127.16
125.94
125.11
120.26
116.94
114.76
77.32
77.00
76.68
58.96
34.60
31.20
O
O
N
H
tBu
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.25
7.24
7.22
7.20
7.18
7.16
7.06
7.04
7.04
7.02
7.00
6.88
6.86
6.86
6.81
6.81
6.79
5.01
5.01
4.24
2.34
1.26
O
O
N
H
Me
2j
4.53
2.03
1.02
1.00
0.99
0.99
3.05
f1 (ppm)

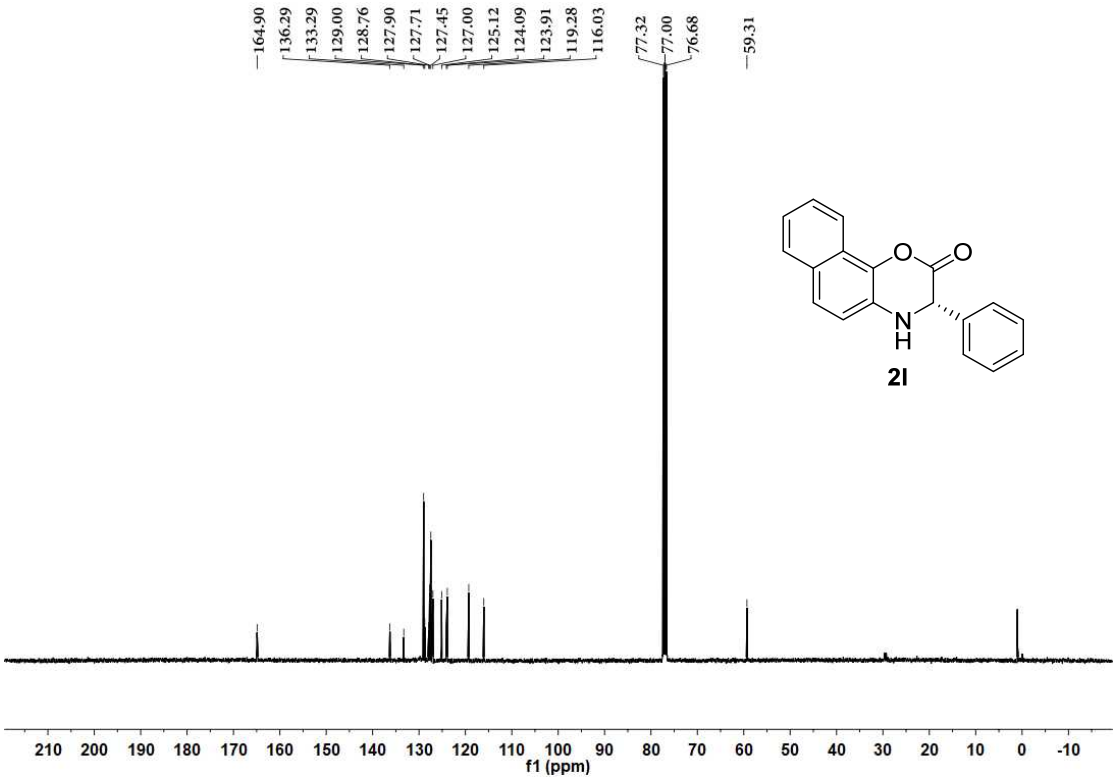

165.35
140.86
138.76
136.18
132.42
129.76
128.83
128.22
125.12
124.46
120.31
116.93
114.79
77.32
77.00
76.68
59.25
21.41
O
O
N
H
Me
2j
f1 (ppm)


7.48
7.43
7.42
7.42
7.40
7.38
7.38
7.37
7.37
7.36
7.36
7.35
7.34
7.32
7.26
7.15
5.19
5.19
4.55
1.59
1.25
1.00
1.01
1.05
7.16
1.03
0.99
0.96
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
O
O
N
H
2k

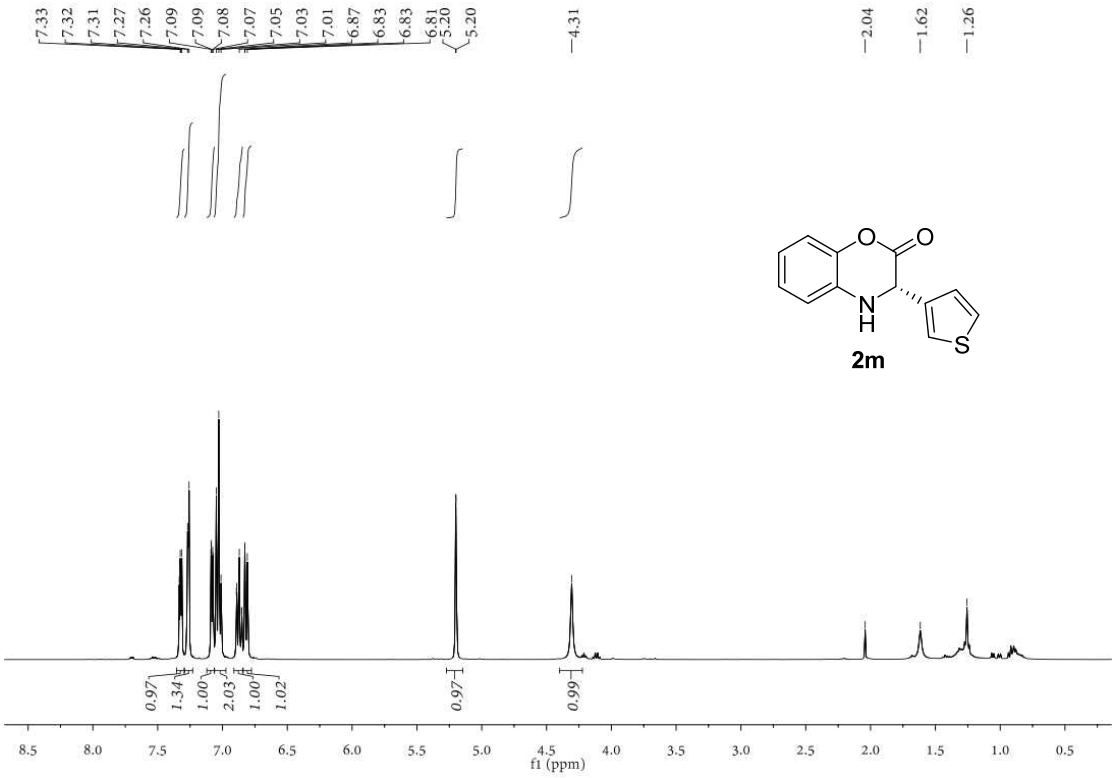

165.18
141.48
136.40
131.94
131.56
129.08
128.45
127.51
127.29
125.99
125.80
124.02
113.65
109.62
77.32
76.68
59.22
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
O
O
N
H
2k

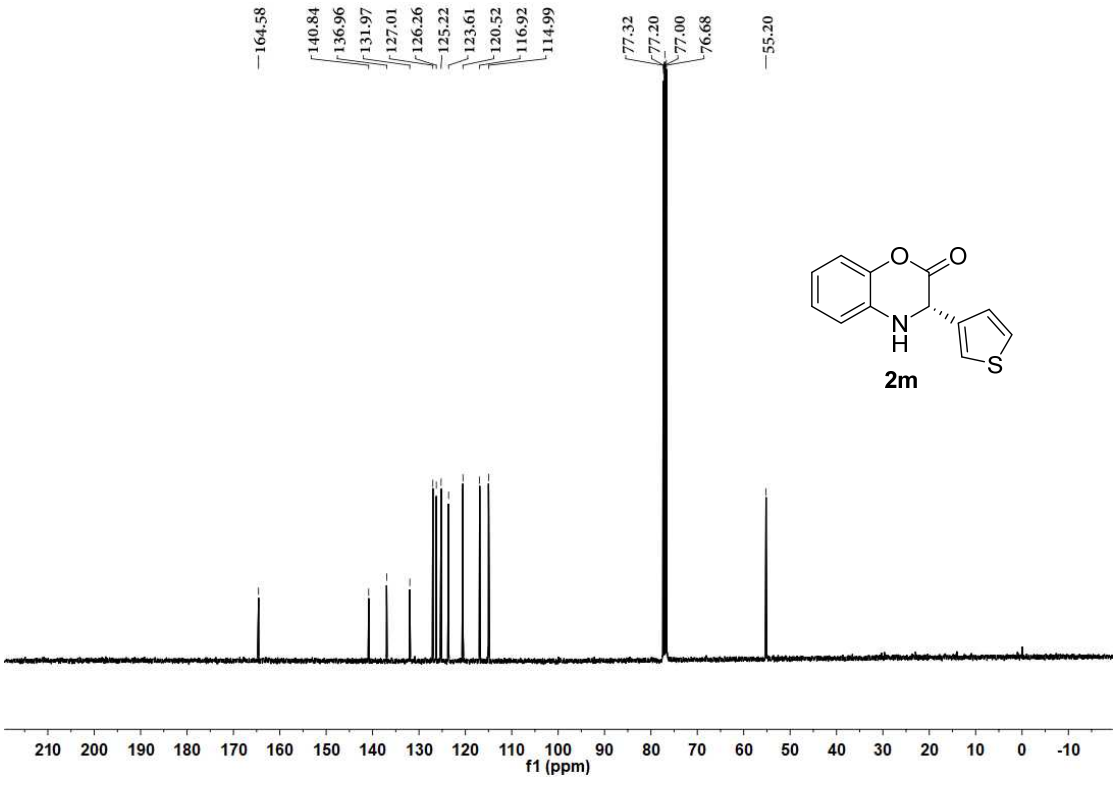

8.10
8.08
7.77
7.75
7.57
7.55
7.50
7.50
7.48
7.48
7.46
7.45
7.44
7.43
7.37
7.36
7.35
7.26
5.17
4.37
1.60
1.25
1.02
1.02
1.12
1.12
2.08
4.03
1.00
1.01
0.95
f1 (ppm)

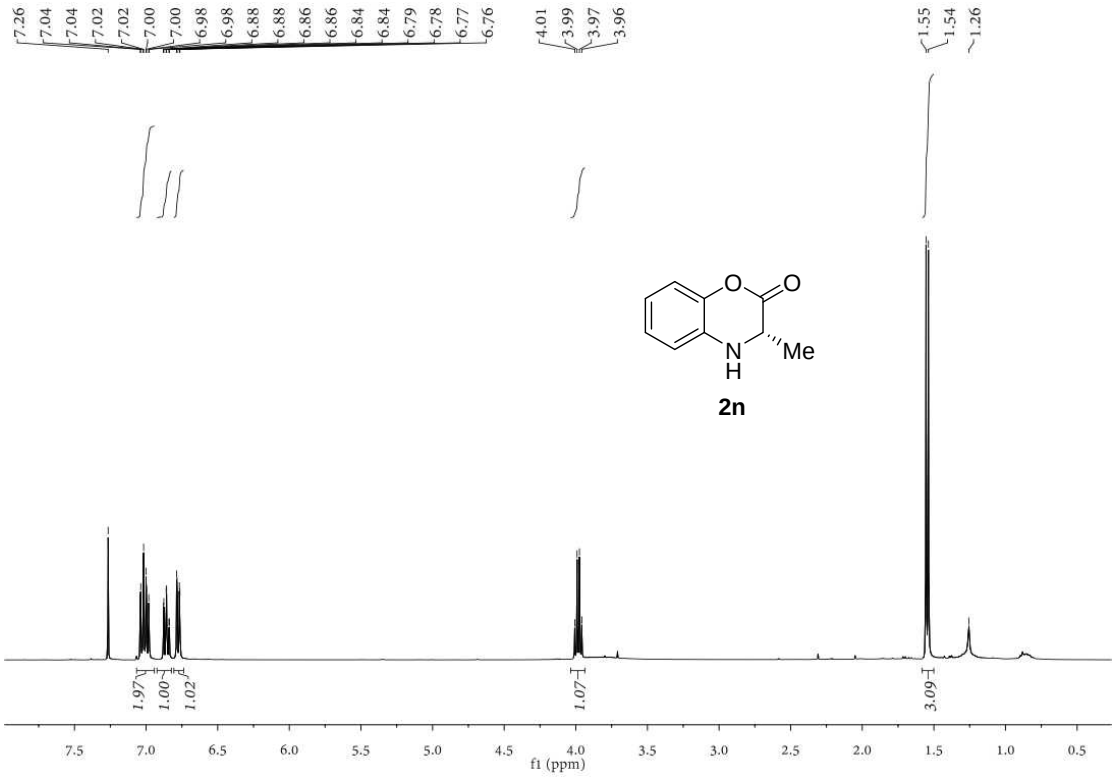
2l


164.90
136.29
133.29
129.00
128.76
127.90
127.71
127.45
127.00
125.12
124.09
123.91
119.28
116.03
77.32
77.00
76.68
59.31
f1 (ppm)

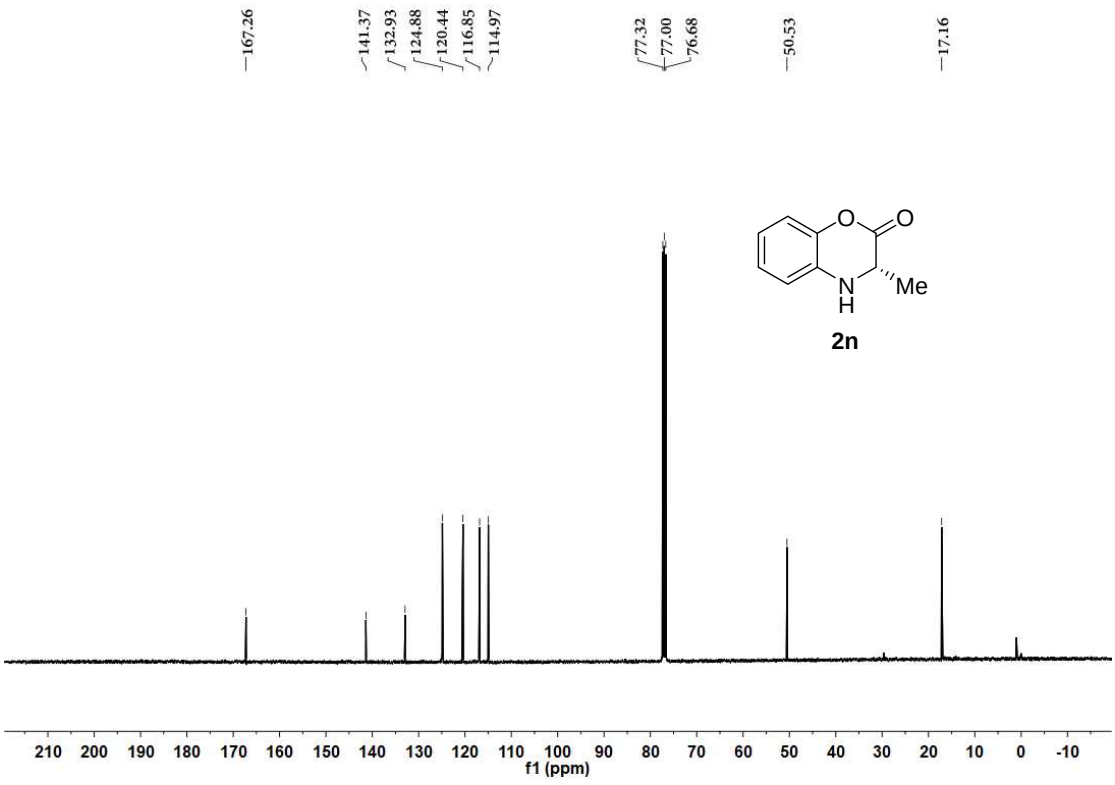
2l


7.33
7.32
7.31
7.27
7.26
7.09
7.09
7.08
7.07
7.05
7.03
7.01
6.87
6.83
6.83
6.81
5.20
5.20
4.31
2.04
1.62
1.26
O
O
N
H
S
2m
0.97
1.34
1.00
2.03
1.00
1.02
0.97
0.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

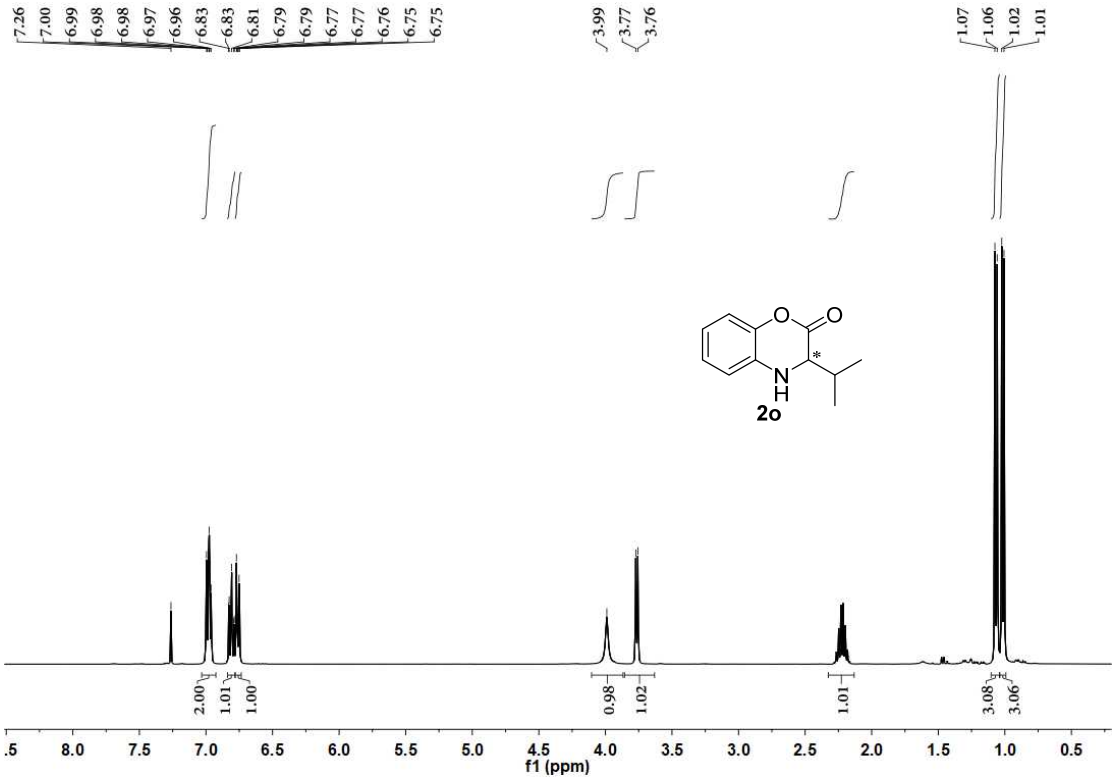

164.58
140.84
136.96
131.97
127.01
126.26
125.22
123.61
120.52
116.92
114.99
77.32
77.20
77.00
76.68
55.20
O
O
N
H
S
2m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

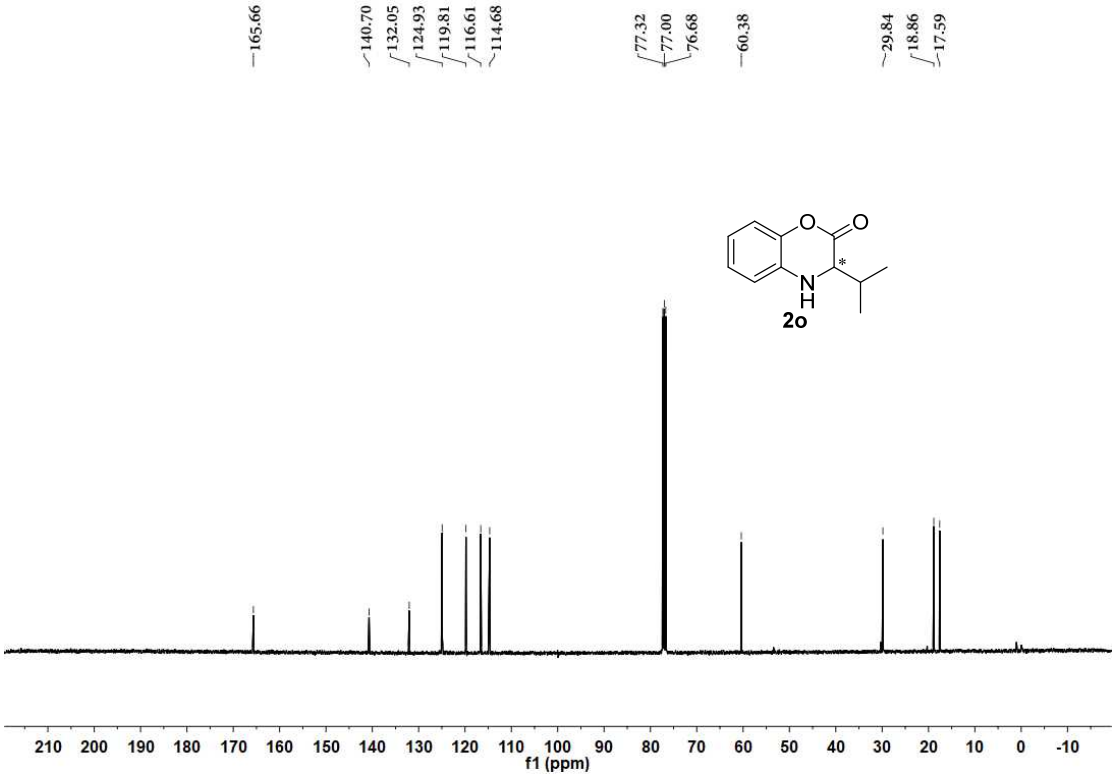

7.26
7.04
7.04
7.02
7.02
7.00
7.00
6.98
6.98
6.88
6.88
6.86
6.86
6.84
6.84
6.79
6.78
6.77
6.76
4.01
3.99
3.97
3.96
1.55
1.54
1.26
O
O
N
H
Me
1.97
1.00
1.02
1.07
3.09
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

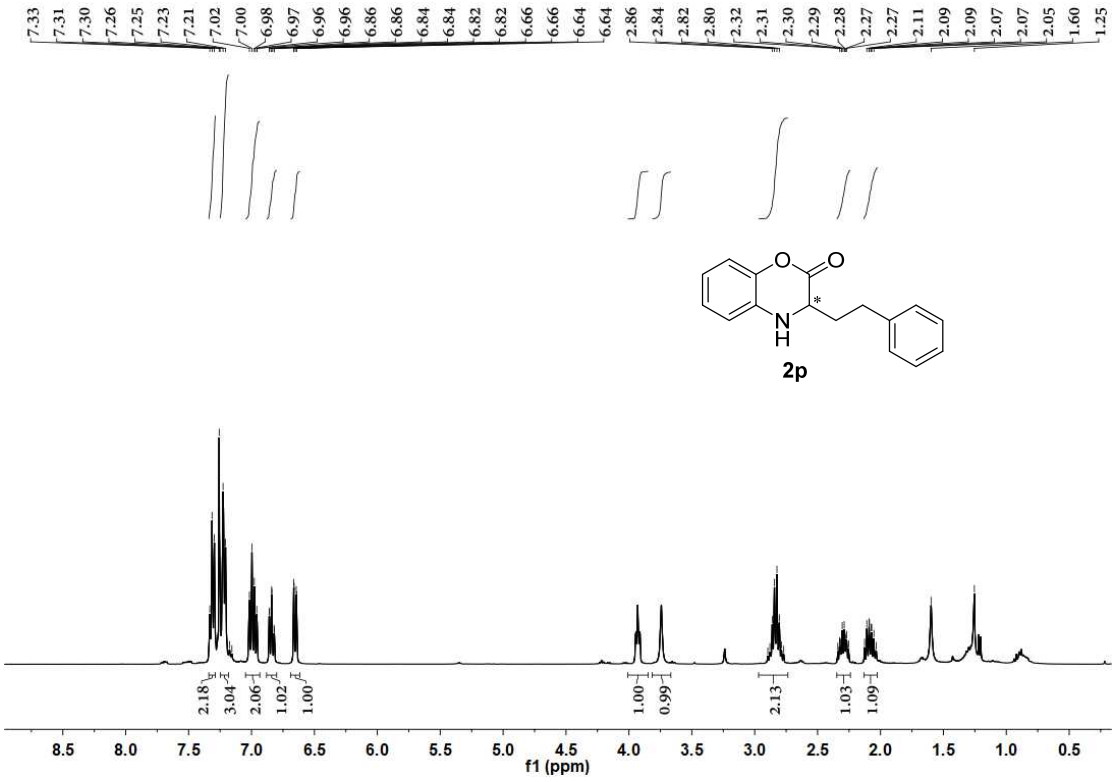
2n

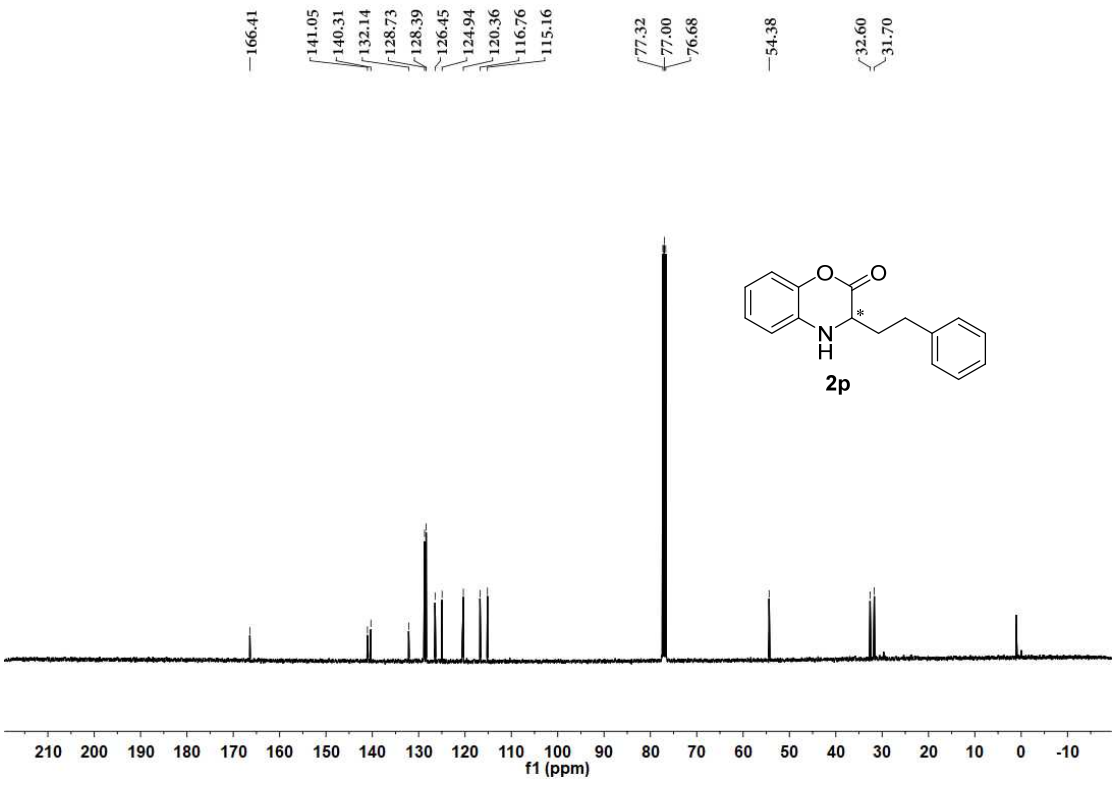

167.26
141.37
132.93
124.88
120.44
116.85
114.97
77.32
77.00
76.68
50.53
17.16
O
O
N
H
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

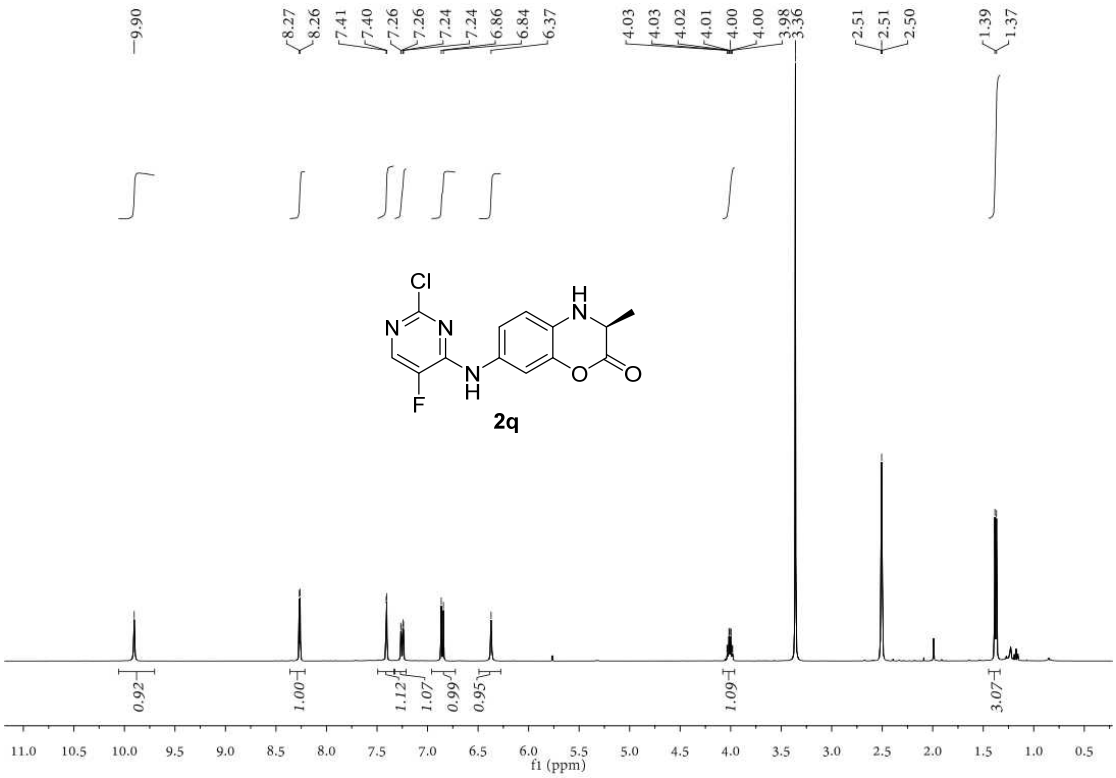
2n

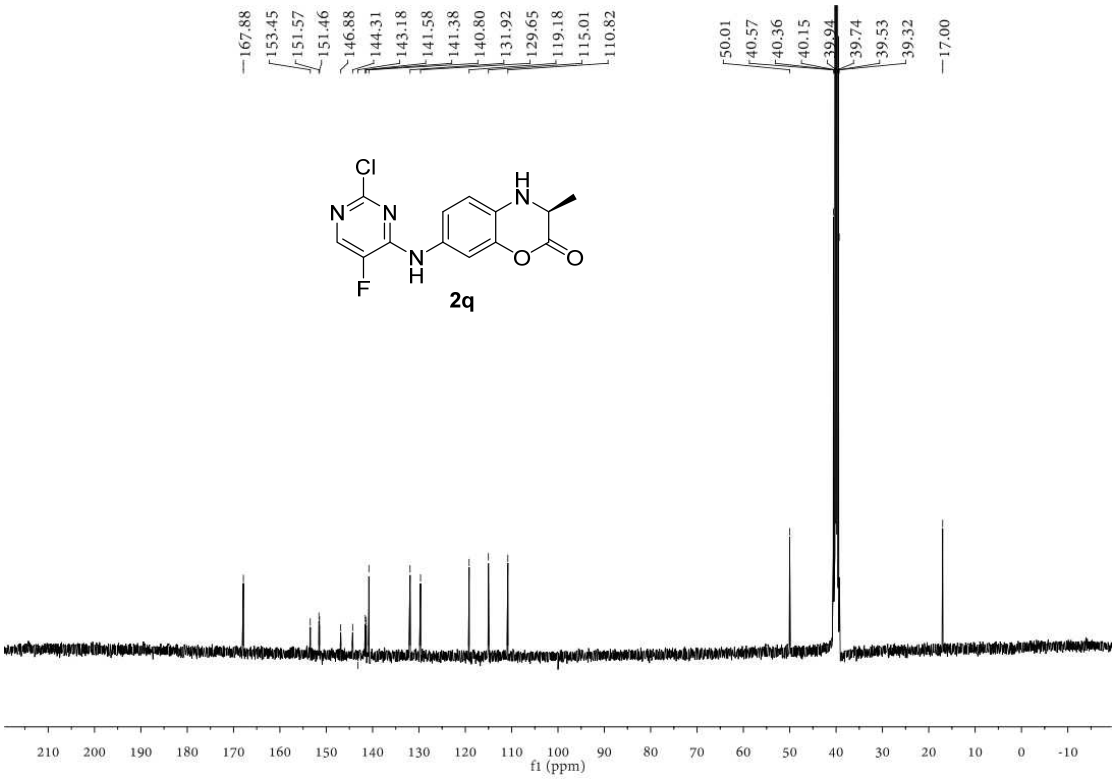

7.26
7.00
6.99
6.98
6.98
6.97
6.96
6.83
6.83
6.81
6.79
6.79
6.77
6.77
6.76
6.75
6.75
3.99
3.77
3.76
1.07
1.06
1.02
1.01
2o
2.00
1.01
1.00
0.98
1.02
1.01
3.08
3.06
f1 (ppm)

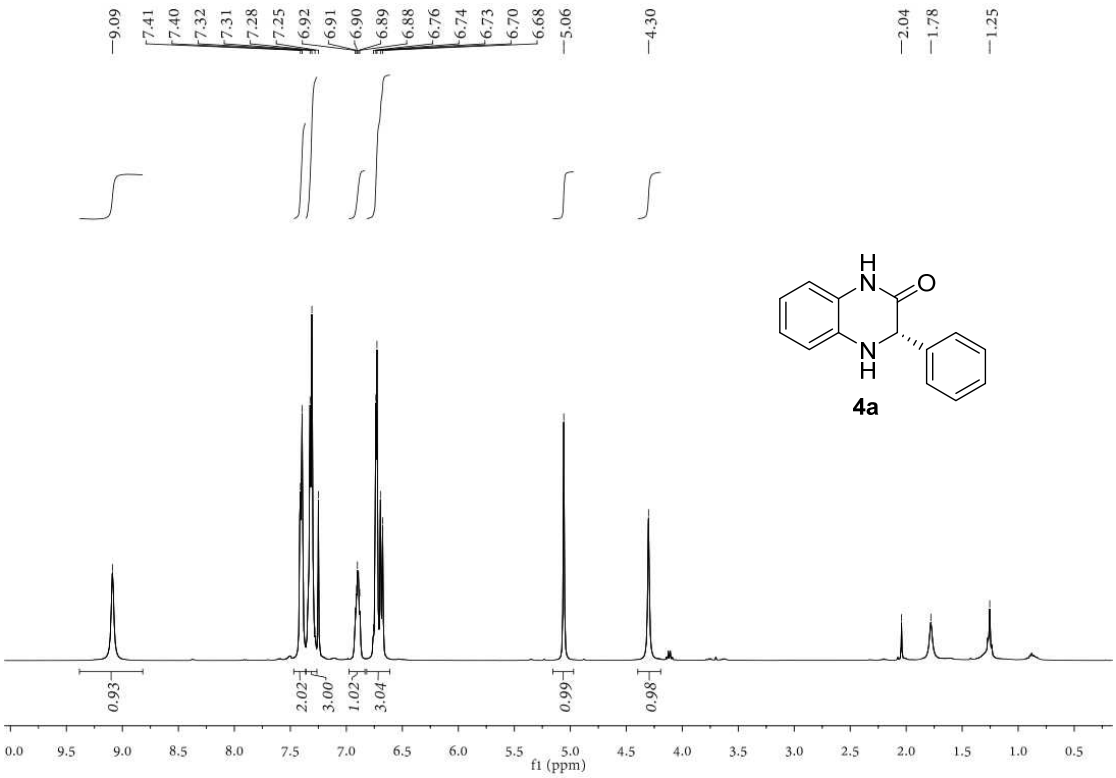

165.66
140.70
132.05
124.93
119.81
116.61
114.68
77.32
77.00
76.68
60.38
29.84
18.86
17.59
2o
f1 (ppm)


7.33 7.31 7.30 7.26 7.25 7.23 7.21 7.02 7.00 6.98 6.97 6.96 6.96 6.96 6.86 6.86 6.84 6.84 6.82 6.82 6.66 6.66 6.64 6.64
2.86 2.84 2.82 2.80 2.32 2.31 2.30 2.29 2.28 2.27 2.27 2.11 2.09 2.09 2.07 2.07 2.05 1.60 1.25
O
O
N
H
*
2p
2.18 3.04 2.06 1.02 1.00 1.00 0.99 2.13 1.03 1.09
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

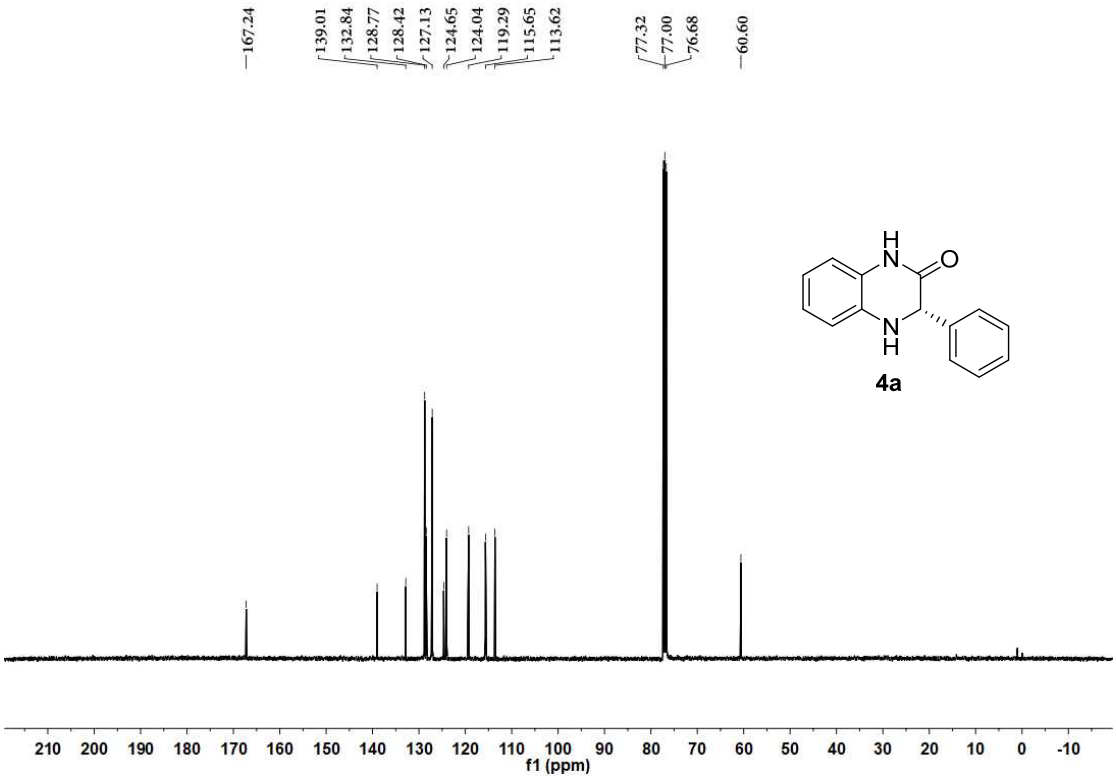

166.41
141.05 140.31 132.14 128.73 128.39 126.45 124.94 120.36 116.76 115.16
77.32 77.00 76.68
54.38
32.60 31.70
O
O
N
H
*
2p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


9.90
8.27
8.26
7.41
7.40
7.26
7.26
7.24
7.24
6.86
6.84
6.37
4.03
4.03
4.02
4.01
4.00
4.00
3.98
3.36
2.51
2.51
2.50
1.39
1.37
Cl
N
N
H
N
N
H
O
O
F
2q
0.92
1.00
1.12
1.07
0.99
0.95
1.09
3.07
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

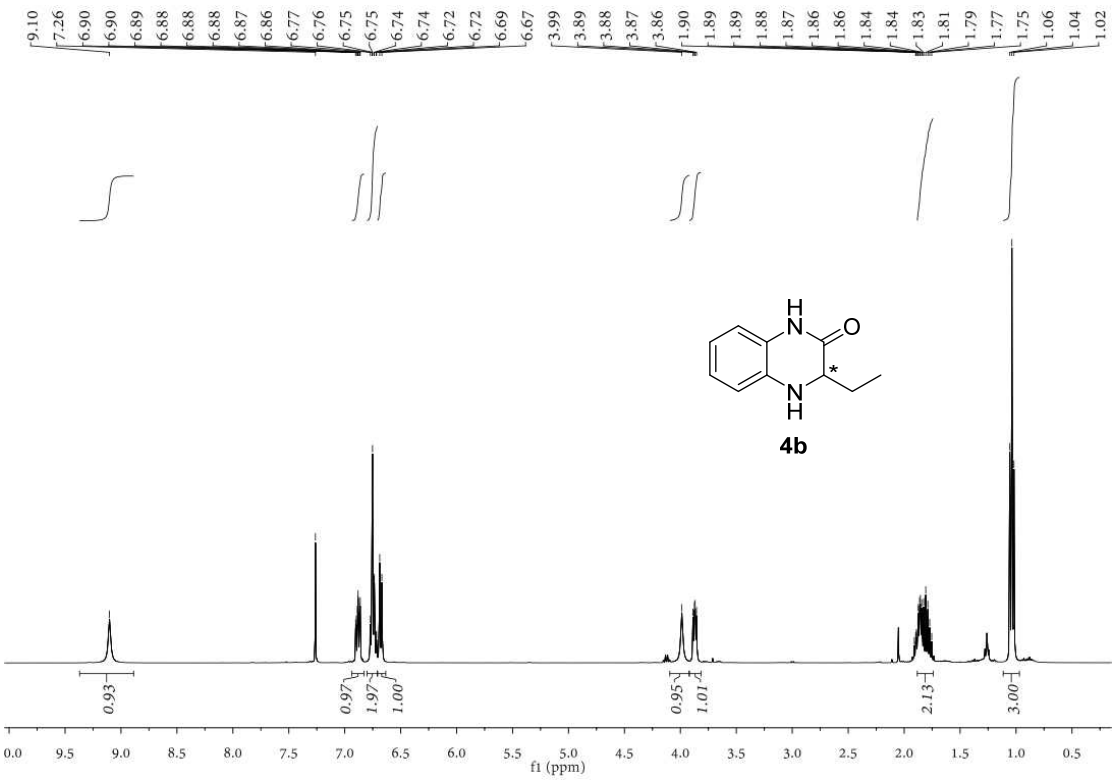

167.88
153.45
151.57
151.46
146.88
144.31
143.18
141.58
141.38
140.80
131.92
129.65
119.18
115.01
110.82
50.01
40.57
40.36
40.15
39.94
39.74
39.53
39.32
17.00
Cl
N
N
H
N
N
H
O
O
F
2q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

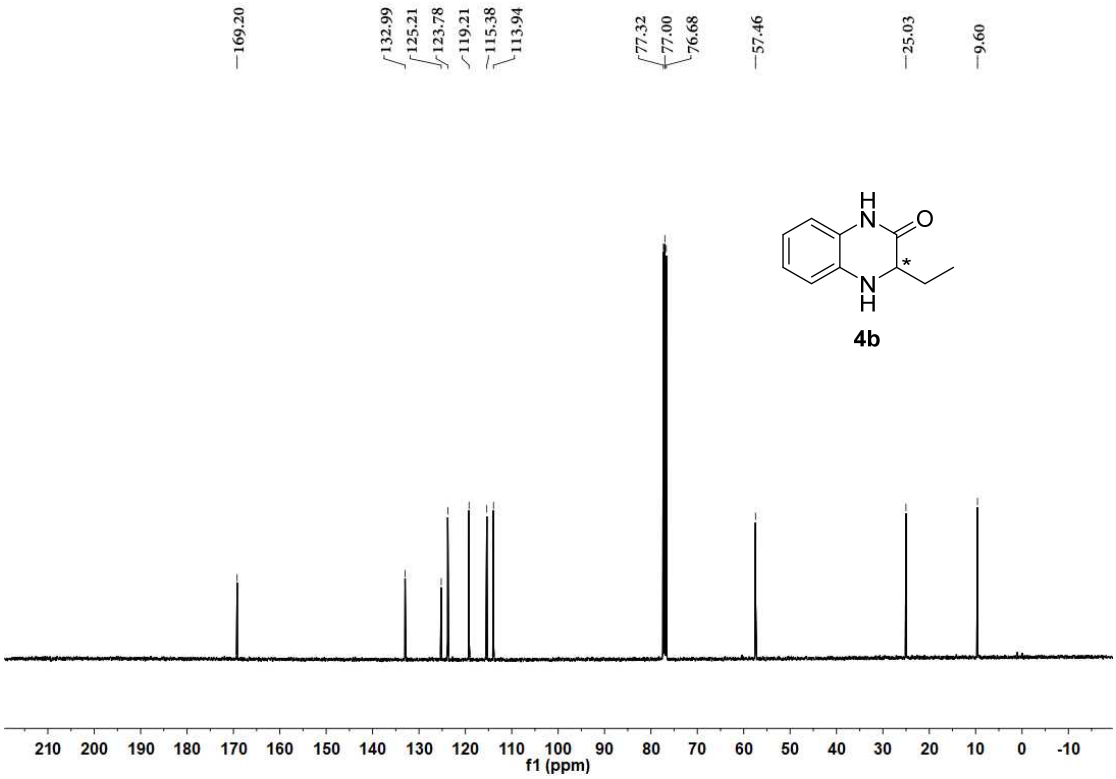

9.09
7.41
7.40
7.32
7.31
7.28
7.25
6.92
6.91
6.90
6.89
6.88
6.76
6.74
6.73
6.70
6.68
5.06
4.30
2.04
1.78
1.25
0.93
2.02
3.00
1.02
3.04
0.99
0.98
f1 (ppm)

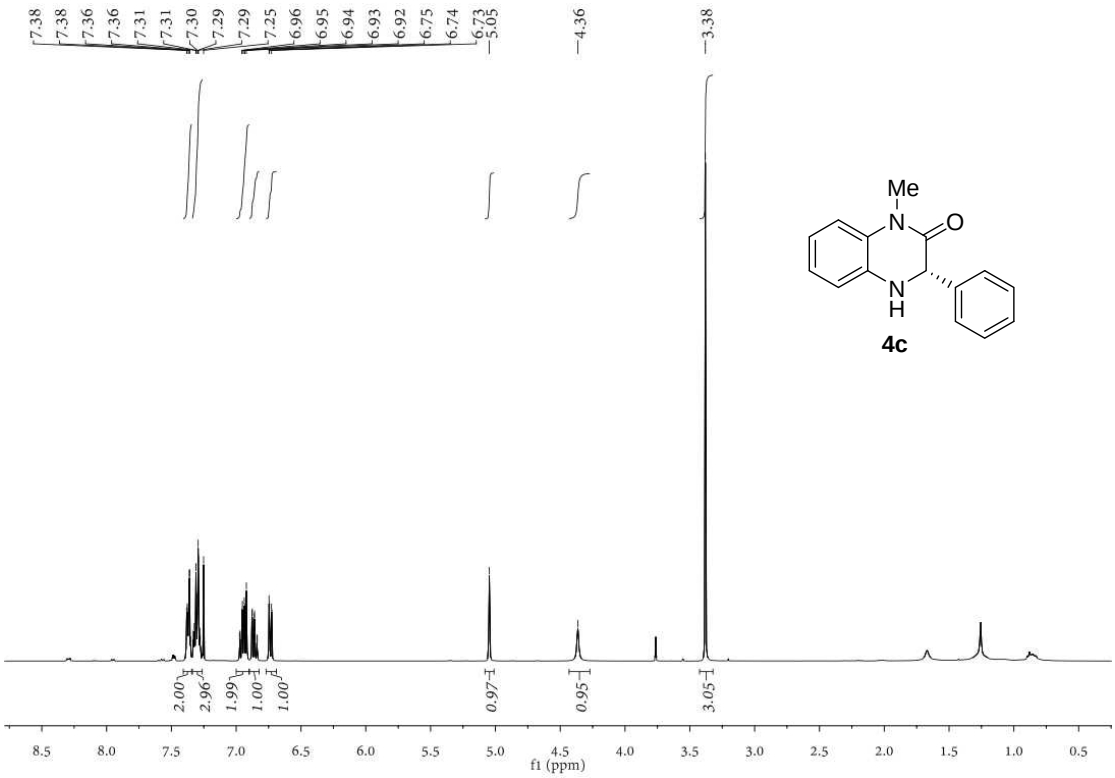
4a


167.24
139.01
132.84
128.77
128.42
127.13
124.65
124.04
119.29
115.65
113.62
77.32
77.00
76.68
60.60
f1 (ppm)

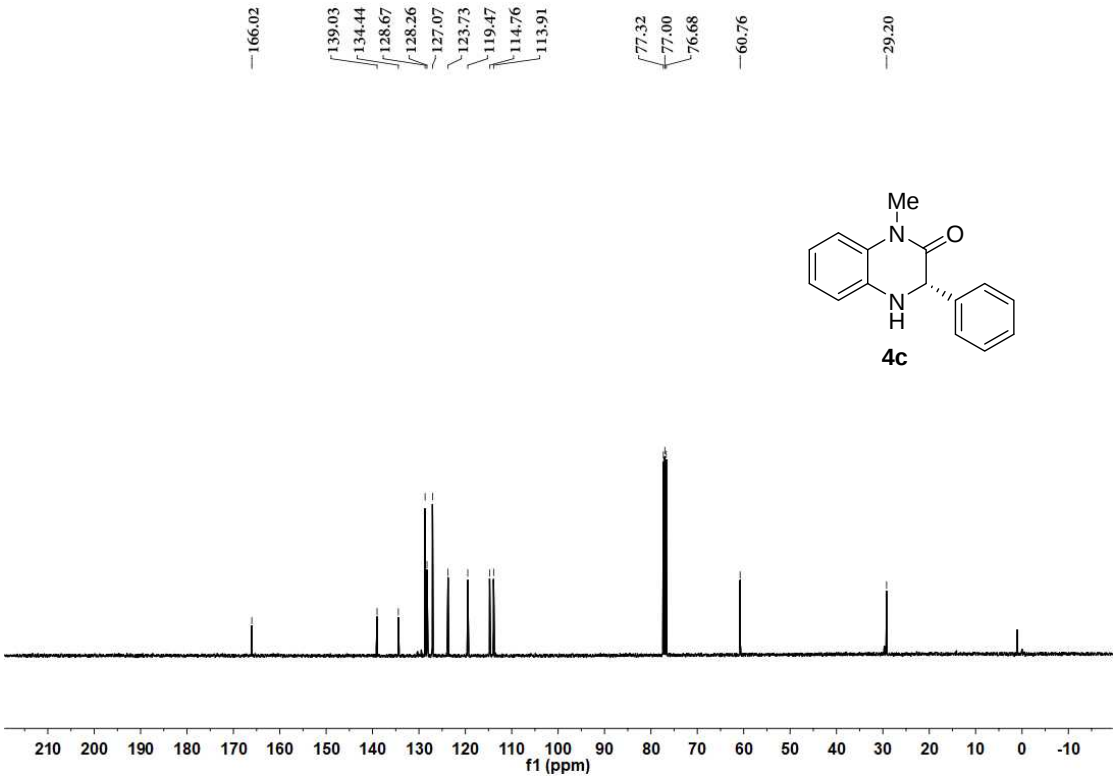
4a


9.10 7.26 6.90 6.90 6.89 6.88 6.88 6.88 6.87 6.86 6.77 6.76 6.75 6.75 6.74 6.74 6.72 6.72 6.69 6.67
3.99 3.89 3.88 3.87 3.86 1.90 1.89 1.89 1.88 1.87 1.86 1.86 1.84 1.84 1.83 1.81 1.79 1.77 1.75 1.06 1.04 1.02
H N O * N H
4b
0.93 0.97 1.97 1.00 0.95 1.01 2.13 3.00
f1 (ppm)

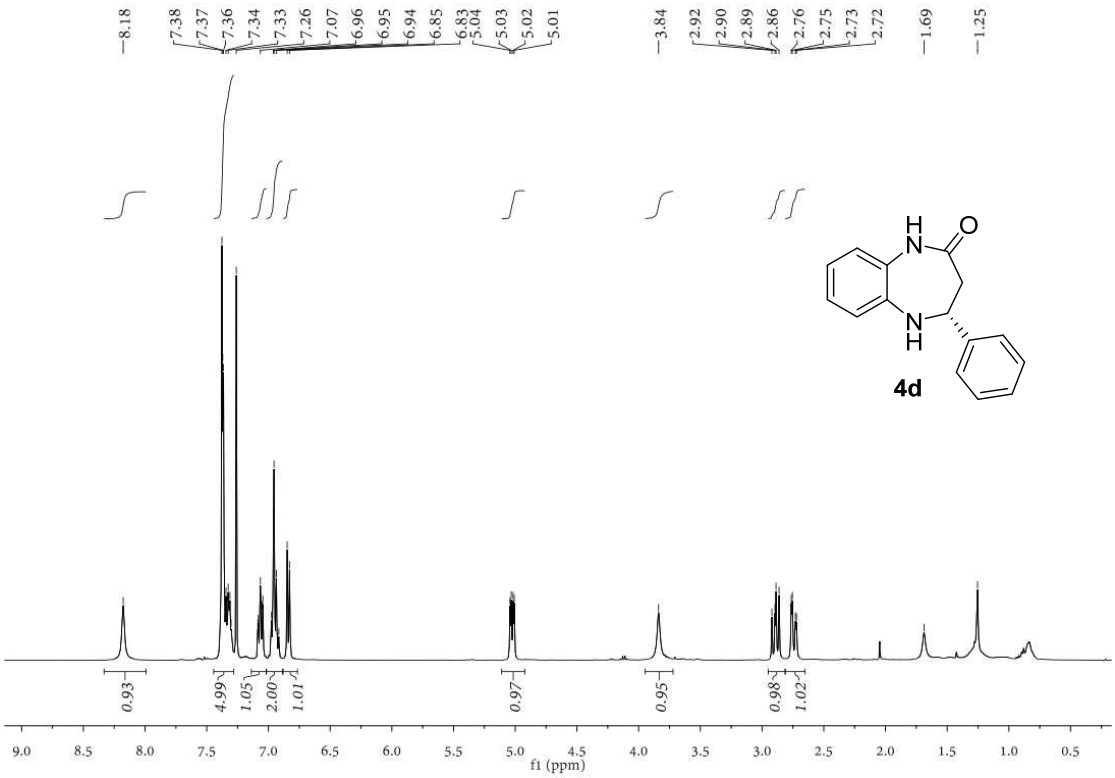

169.20
132.99 125.21 123.78 119.21 115.38 113.94
77.32 77.00 76.68
57.46
25.03
9.60
H N O * N H
4b
f1 (ppm)

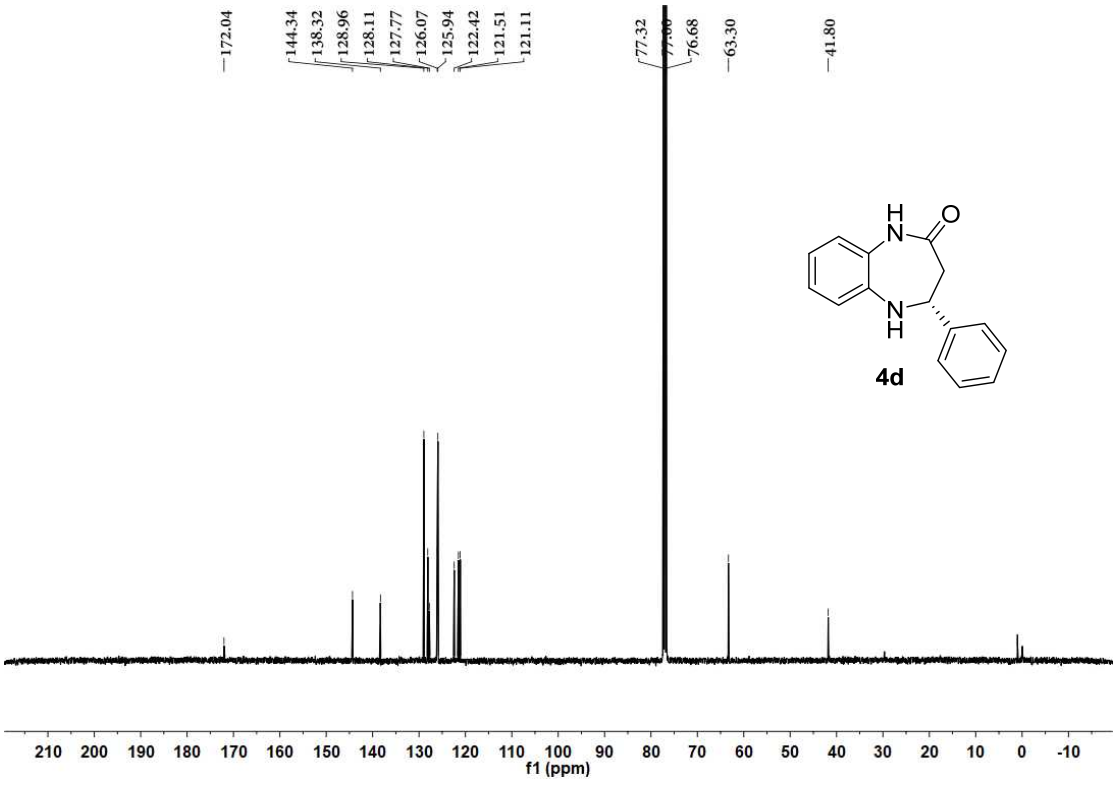

7.38
7.38
7.36
7.36
7.31
7.31
7.30
7.29
7.29
7.25
6.96
6.95
6.94
6.93
6.92
6.75
6.74
6.73
5.05
4.36
3.38
Me
N
O
N
H
2.00
2.96
1.99
1.00
1.00
0.97
0.95
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

4c


166.02
139.03
134.44
128.67
128.26
127.07
123.73
119.47
114.76
113.91
77.32
77.00
76.68
60.76
29.20
Me
N
O
N
H
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

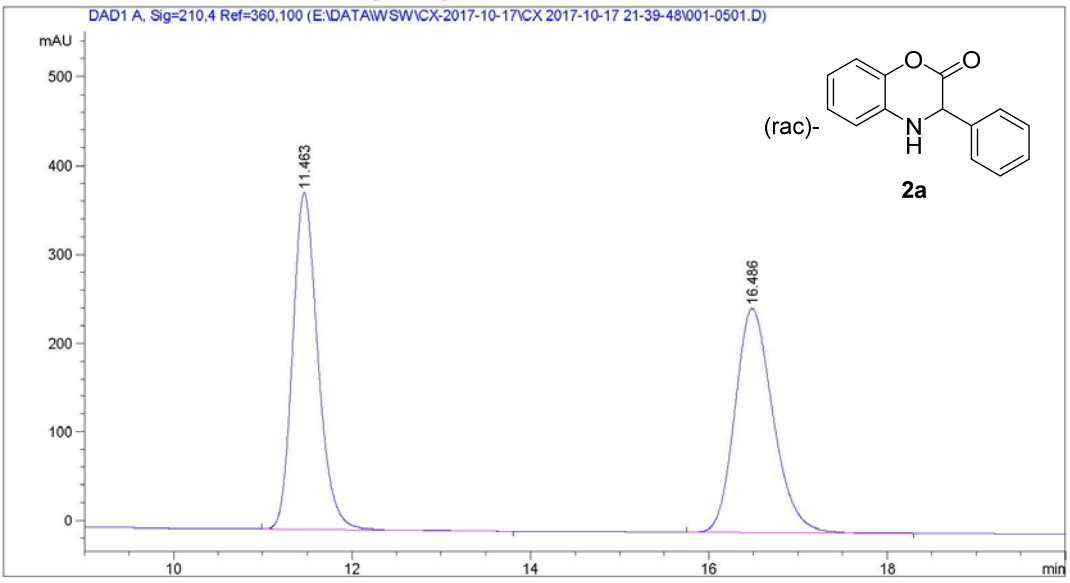
4c


8.18
7.38
7.37
7.36
7.34
7.33
7.26
7.07
6.96
6.95
6.94
6.85
6.83
5.04
5.03
5.02
5.01
3.84
2.92
2.90
2.89
2.86
2.76
2.75
2.73
2.72
1.69
1.25
0.93
4.99
1.05
2.00
1.01
0.97
0.95
0.98
1.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
4d


172.04
144.34
138.32
128.96
128.11
127.77
126.07
125.94
122.42
121.51
121.11
77.32
76.68
63.30
41.80
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
4d

VII. HPLC Spectra

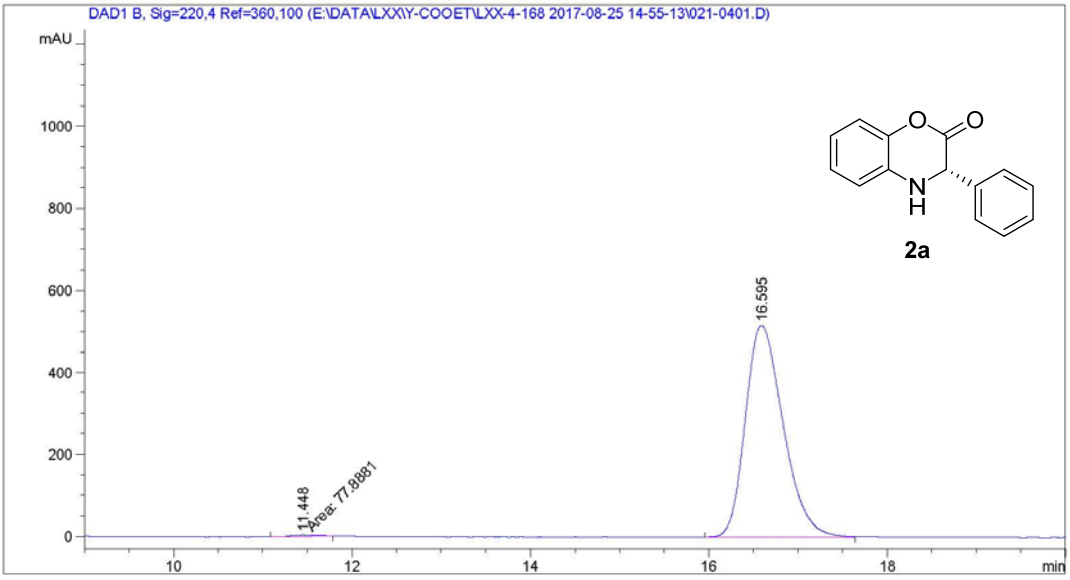
```
Data File E:\DATA\WSW\CX-2017-10-17\CX 2017-10-17 21-39-48\001-0501.D
Sample Name: HZY-STAND-RAC-PDC

    ========================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 1
    Injection Date  : 10/18/2017 12:04:44 AM             Inj :   1
                                                   Inj Volume : 1.000 µl
    Acq. Method     : E:\DATA\WSW\CX-2017-10-17\CX 2017-10-17 21-39-48\DAD-OD(1-2)-80-20-1ML-1UL-
                      25MIN.M
    Last changed    : 10/17/2017 9:41:26 PM by SYSTEM
    Analysis Method : E:\DATA\WSW\CX-2017-10-17\CX 2017-10-17 21-39-48\DAD-OD(1-2)-80-20-1ML-1UL-
                      25MIN.M (Sequence Method)
    Last changed    : 10/18/2017 2:29:25 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  11.463 BB    0.3012 7456.20361  379.57709  49.9735
       2  16.486 BB    0.4543 7464.11328  252.91653  50.0265

    Totals :                  1.49203e4   632.49362

1260HPLC-DAD 10/18/2017 2:29:32 PM SYSTEM                    Page   1 of 2
```

Data File E:\DATA\LXX\Y-COOET\LXX-4-168 2017-08-25 14-55-13\021-0401.D
Sample Name: HZY-N-ME-L

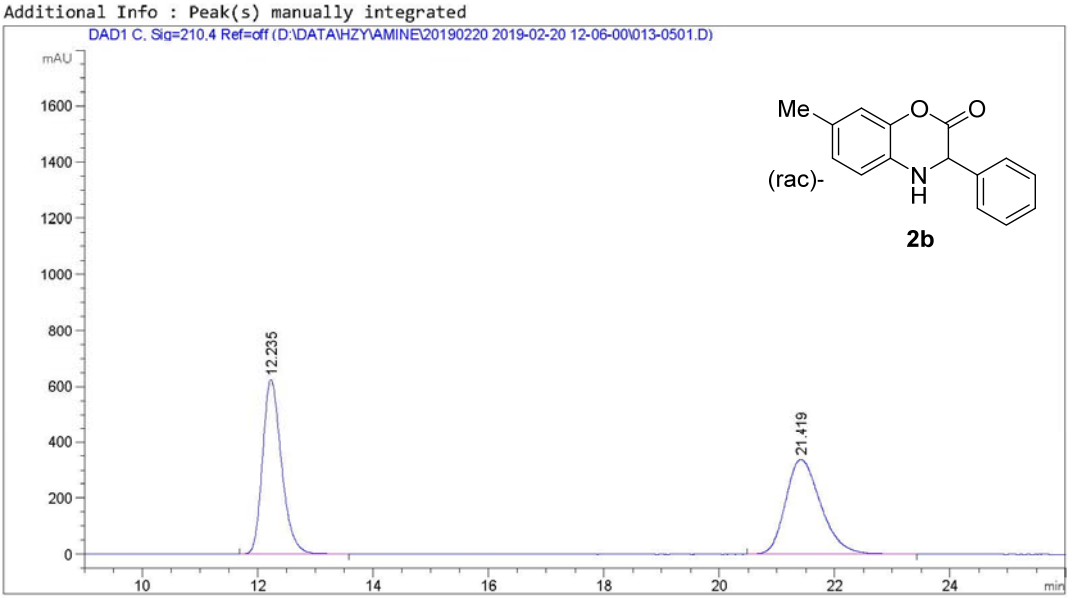
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 21
Injection Date  : 8/25/2017 4:19:18 PM               Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\LXX\Y-COOET\LXX-4-168 2017-08-25 14-55-13\DAD-OD(1-2)-80-20-1ML-1UL
                  -25MIN.M
Last changed    : 8/25/2017 3:16:42 PM by SYSTEM
Analysis Method : E:\DATA\LXX\Y-COOET\LXX-4-168 2017-08-25 14-55-13\DAD-OD(1-2)-80-20-1ML-1UL
                  -25MIN.M (Sequence Method)
Last changed    : 8/25/2017 6:31:11 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.448 MM    0.3217   77.88808    4.03542   0.5097
   2  16.595 BV    0.4370 1.52034e4   517.14484  99.4903

Totals :                  1.52813e4   521.18026
```

Data File D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\013-0501.D
Sample Name: Me-PdC

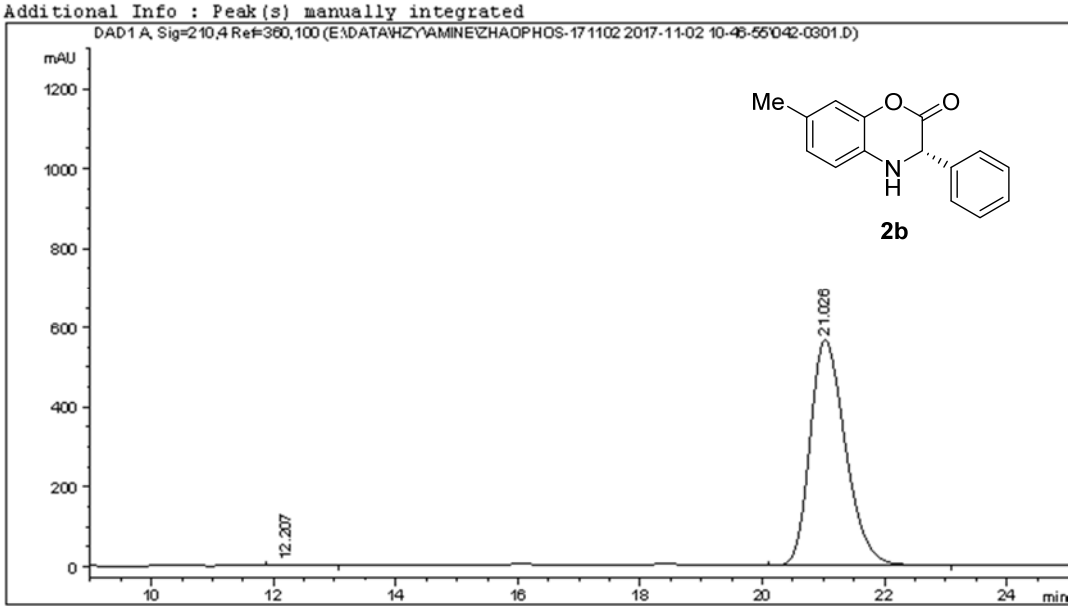
```
==========================================================================
Acq. Operator   :                              Seq. Line :   5
Acq. Instrument : Instrument 2                  Location : Vial 13
Injection Date  : 2/20/2019 1:31:27 PM               Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\DAD-OD(1-2)-80-20-1ML-1UL-
                  ALL-30MIN.M
Last changed    : 12/15/2018 5:07:00 PM
Analysis Method : D:\METHOD\HZY\DAD-OJ(1-6)-80-20-1ML-1UL-ALL-50MIN.M
Last changed    : 2/20/2019 3:31:40 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.235 BB    0.3519 1.42514e4   625.18842  49.8896
   2  21.419 BB    0.6407 1.43144e4   337.58340  50.1104

Totals :                  2.85658e4   962.77182
```

Data File E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\042-0301.D
Sample Name: H-2

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 42
    Injection Date  : 11/2/2017 11:24:42 AM             Inj :   1
                                                   Inj Volume : 1.000 µl
    Acq. Method     : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-25MIN.M
    Last changed    : 11/2/2017 10:46:57 AM by SYSTEM
    Analysis Method : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-25MIN.M (Sequence Method)
    Last changed    : 11/2/2017 2:58:04 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

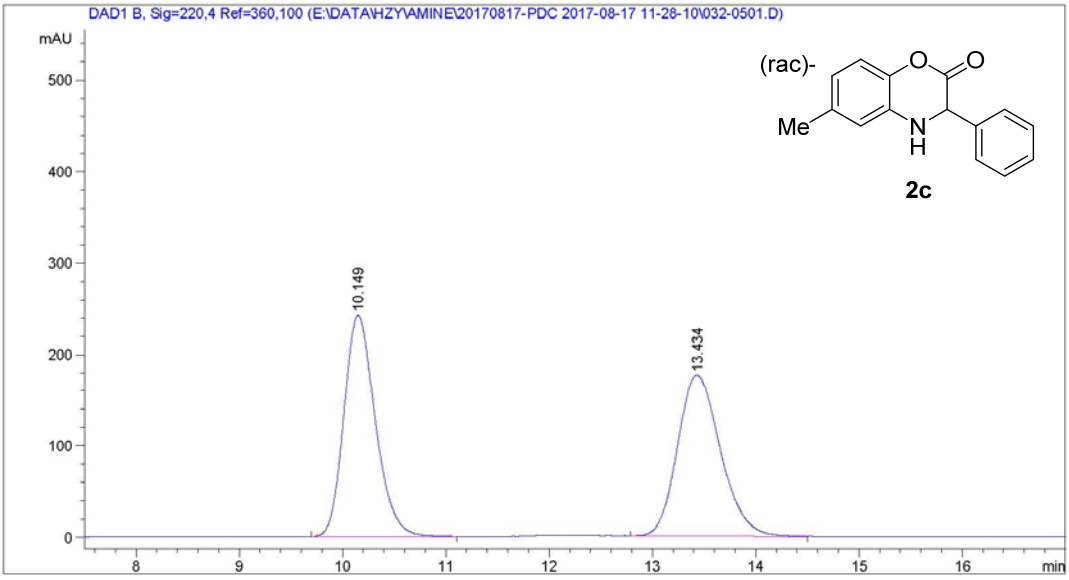


2b

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  12.207 BB    0.2839   63.06870    2.72591   0.2773
       2  21.026 BB    0.6202 2.26773e4   565.12823  99.7227

    Totals :                  2.27403e4   567.85415


    ====================================================================
                            *** End of Report ***
```

Data File E:\DATA\HZY\AMINE\20170817-PDC 2017-08-17 11-28-10\032-0501.D
Sample Name: pdc-2

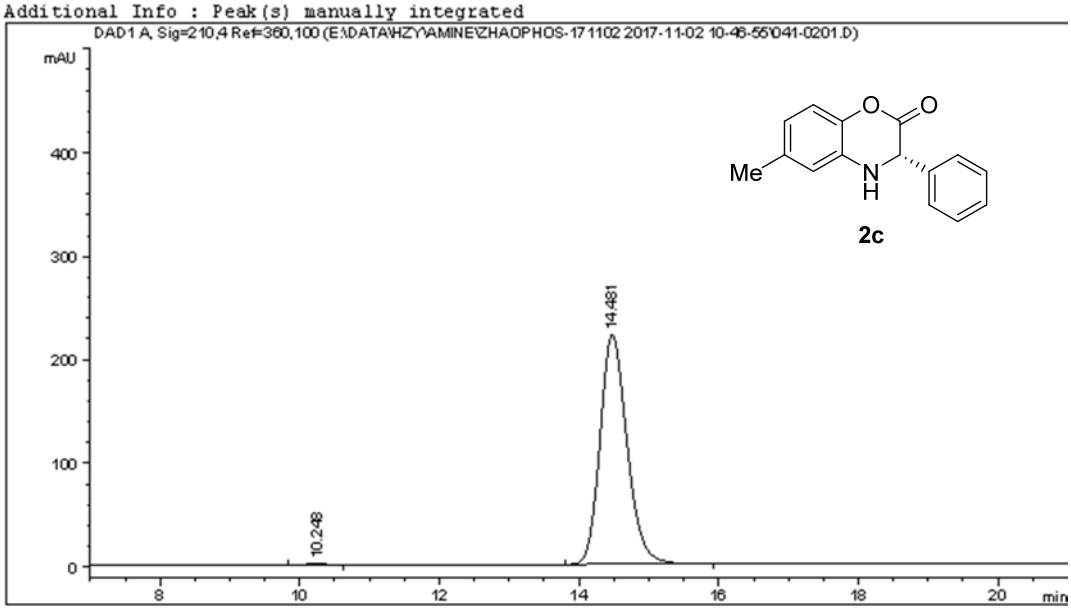
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 32
Injection Date  : 8/17/2017 12:58:00 PM              Inj :   1
                                                 Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\20170817-PDC 2017-08-17 11-28-10\DAD-OD(1-2)-80-20-1ML-
                  1UL-25MIN.M
Last changed    : 8/17/2017 11:28:10 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\20170817-PDC 2017-08-17 11-28-10\DAD-OD(1-2)-80-20-1ML-
                  1UL-25MIN.M (Sequence Method)
Last changed    : 8/17/2017 4:57:01 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.149 VB    0.3154 5104.18604  242.61790  50.0894
   2  13.434 BB    0.4222 5085.97412  176.48285  49.9106

Totals :                  1.01902e4   419.10075
```

Data File E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\041-0201.D
Sample Name: H-1

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 41
    Injection Date  : 11/2/2017 10:58:48 AM               Inj :   1
                                                   Inj Volume : 1.000 µl
    Acq. Method     : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-25MIN.M
    Last changed    : 11/2/2017 10:46:57 AM by SYSTEM
    Analysis Method : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-25MIN.M (Sequence Method)
    Last changed    : 11/2/2017 2:54:49 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

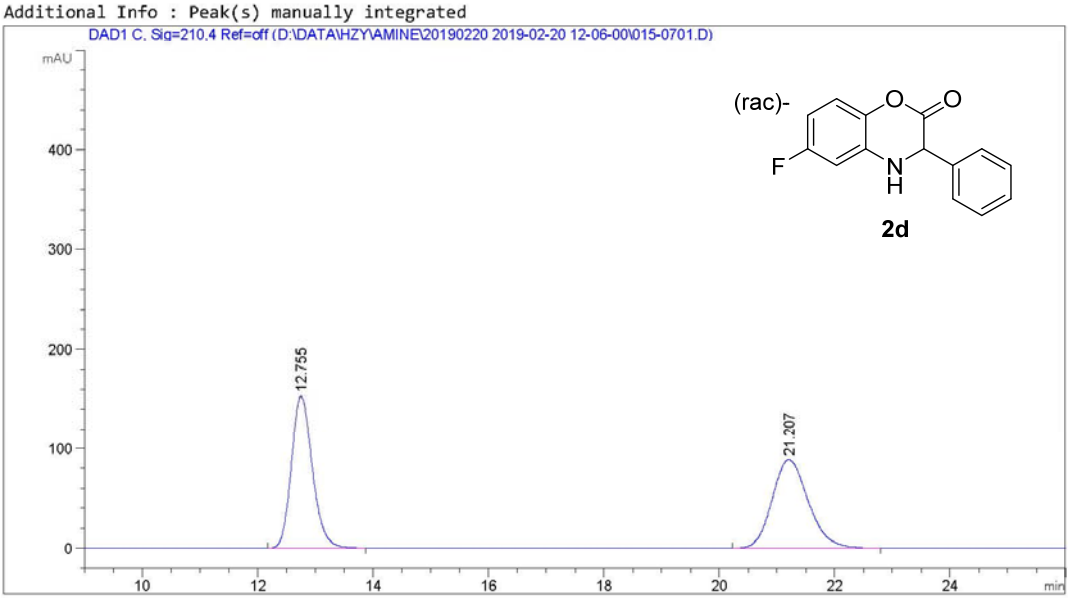


2c

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  10.248 BB    0.2408   24.53086    1.25108   0.4111
       2  14.481 BB    0.4152 5942.91553  221.38681  99.5889

    Totals :                  5967.44639  222.63789


    ====================================================================
                            *** End of Report ***
```

Data File D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\015-0701.D
Sample Name: F-PdC

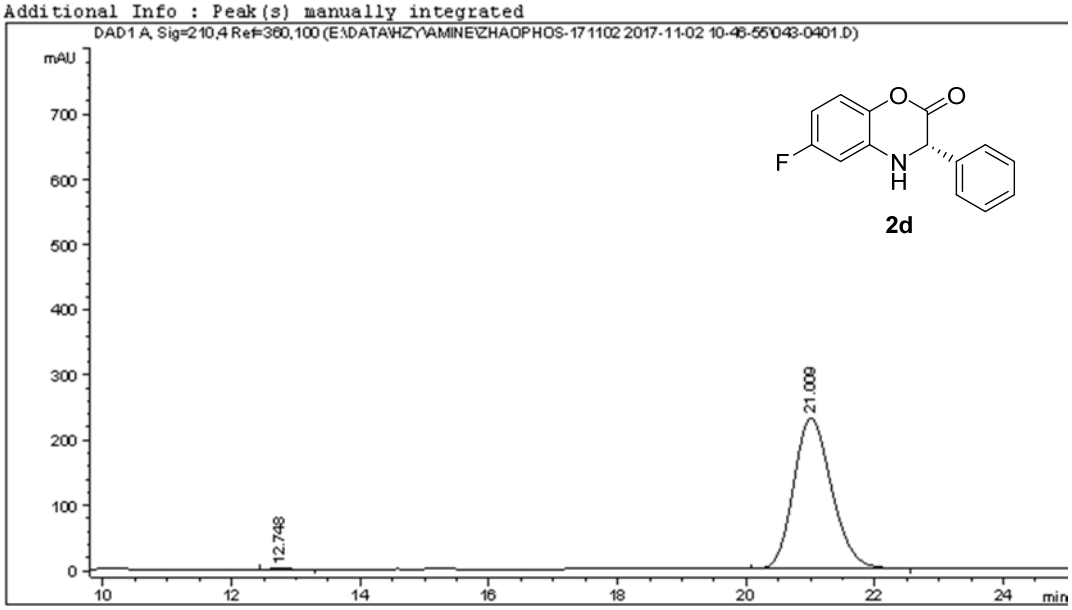
```
==========================================================================
Acq. Operator   :                              Seq. Line :   7
Acq. Instrument : Instrument 2                  Location : Vial 15
Injection Date  : 2/20/2019 2:33:18 PM               Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\DAD-OD(1-2)-80-20-1ML-1UL-
                  ALL-30MIN.M
Last changed    : 12/15/2018 5:07:00 PM
Analysis Method : D:\METHOD\HZY\DAD-OJ(1-6)-80-20-1ML-1UL-ALL-50MIN.M
Last changed    : 2/20/2019 3:33:28 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.755 BB    0.3867 3840.82959  153.07249  49.9734
   2  21.207 BB    0.6508 3844.91992   88.51109  50.0266

Totals :                  7685.74951  241.58359
```

Data File E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\043-0401.D
Sample Name: H-4

```
    ====================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   4
    Acq. Instrument : 1260HPLC-DAD                     Location : Vial 43
    Injection Date  : 11/2/2017 11:50:35 AM                 Inj :   1
                                                      Inj Volume : 1.000 µl
    Acq. Method     : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-25MIN.M
    Last changed    : 11/2/2017 10:46:57 AM by SYSTEM
    Analysis Method : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-25MIN.M (Sequence Method)
    Last changed    : 11/2/2017 3:00:28 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

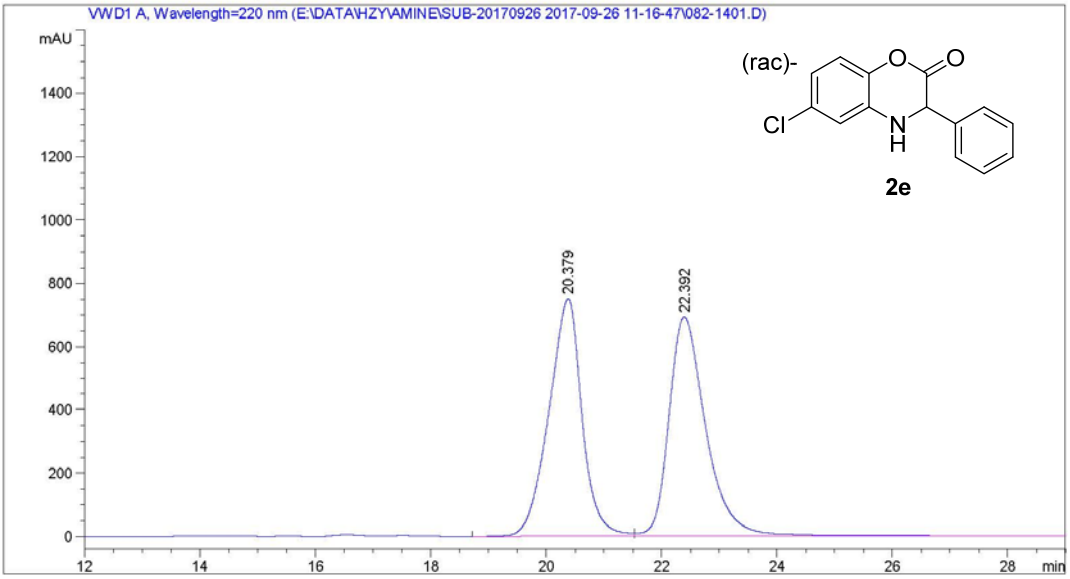


2d

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  12.748 BB    0.2726   30.38393    1.38122   0.3200
       2  21.009 BB    0.6325 9465.70020  230.79195  99.6800

    Totals :                  9496.08413  232.17317


    ====================================================================
                              *** End of Report ***
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\082-1401.D
Sample Name: H-2-RAC

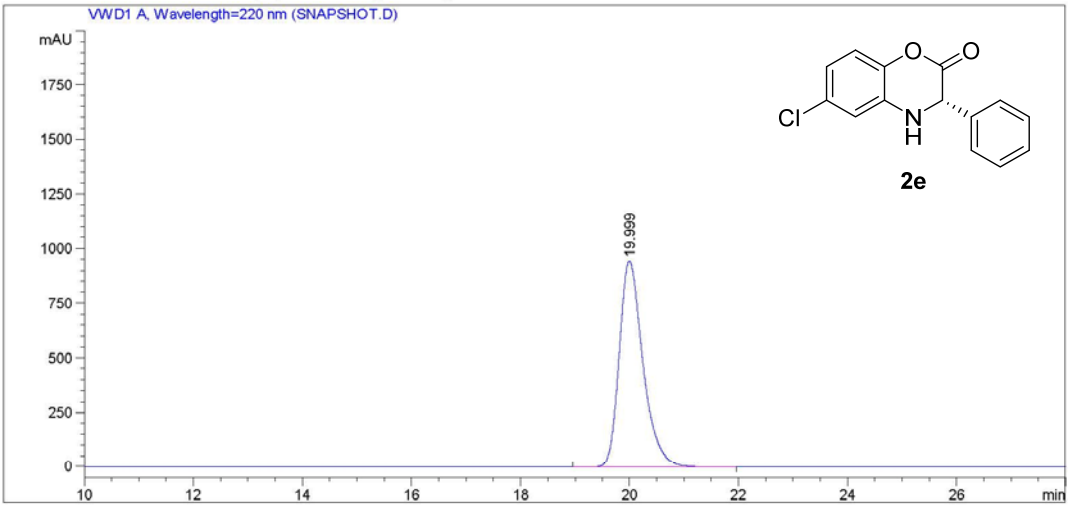
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :  14
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 82
Injection Date  : 9/26/2017 6:16:00 PM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-AD(1-6)-90-10-1ML-
                  5UL-220NM-45MIN.M
Last changed    : 9/26/2017 6:14:38 PM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-AD(1-6)-90-10-1ML-
                  5UL-220NM-45MIN.M (Sequence Method)
Last changed    : 9/26/2017 7:38:49 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.379 BV    0.5892 2.98932e4   751.27502  49.1149
   2  22.392 VB    0.6747 3.09706e4   693.93481  50.8851

Totals :                  6.08639e4  1445.20984
```

Data File C:\CHEM32\1\DATA\SNAPSHOT.D
Sample Name:

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  13
                                                 Location : Vial 72
Injection Date  : 9/26/2017 5:40:14 PM               Inj :   1
Acq. Method     : VWD-AD(1-6)-90-10-1ML-5UL-220NM-45MIN.M
Analysis Method : E:\DATA\HZY\AMINE\CL-PH-RAC170924 2017-09-24 18-48-03\VWD-AD(1-6)-90-10-1ML
                  -2UL-210NM-60MIN.M (Sequence Method)
Last changed    : 9/26/2017 6:12:38 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

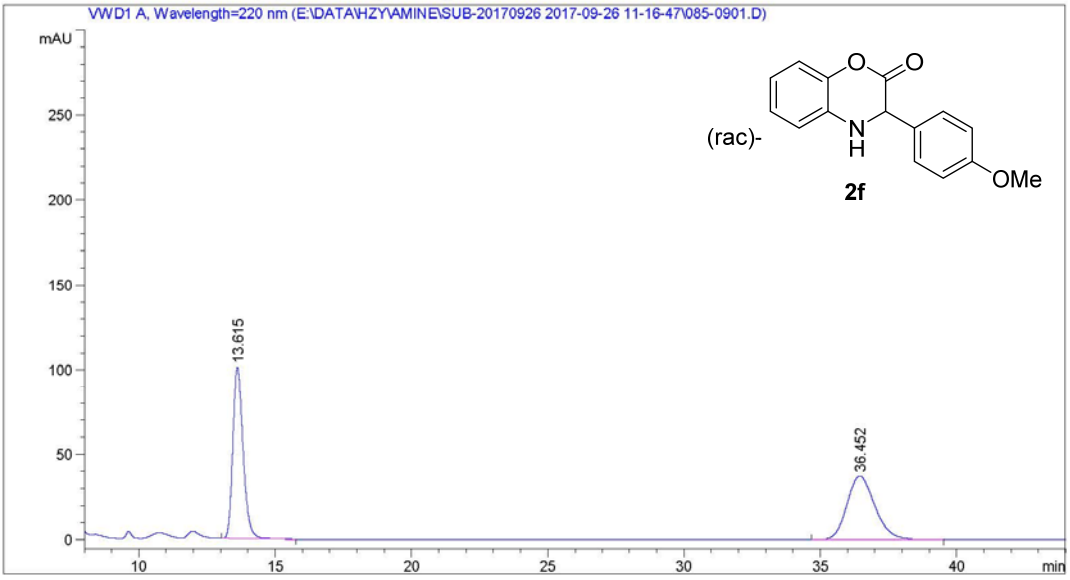


2e

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  19.999 BB    0.4579 2.84939e4   944.78058 100.0000

Totals :                  2.84939e4   944.78058

==================================================================
                        *** End of Report ***
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\085-0901.D
Sample Name: H-5-RAC

```
==================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   9
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 85
Injection Date  : 9/26/2017 3:12:00 PM               Inj :   1
                                               Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-45MIN.M
Last changed    : 9/26/2017 11:46:03 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-45MIN.M (Sequence Method)
Last changed    : 9/26/2017 4:08:27 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.615 BB    0.3988 2638.70752  101.32419  49.8710
   2  36.452 BB    1.0877 2652.35864   37.29427  50.1290

Totals :                  5291.06616  138.61846
```

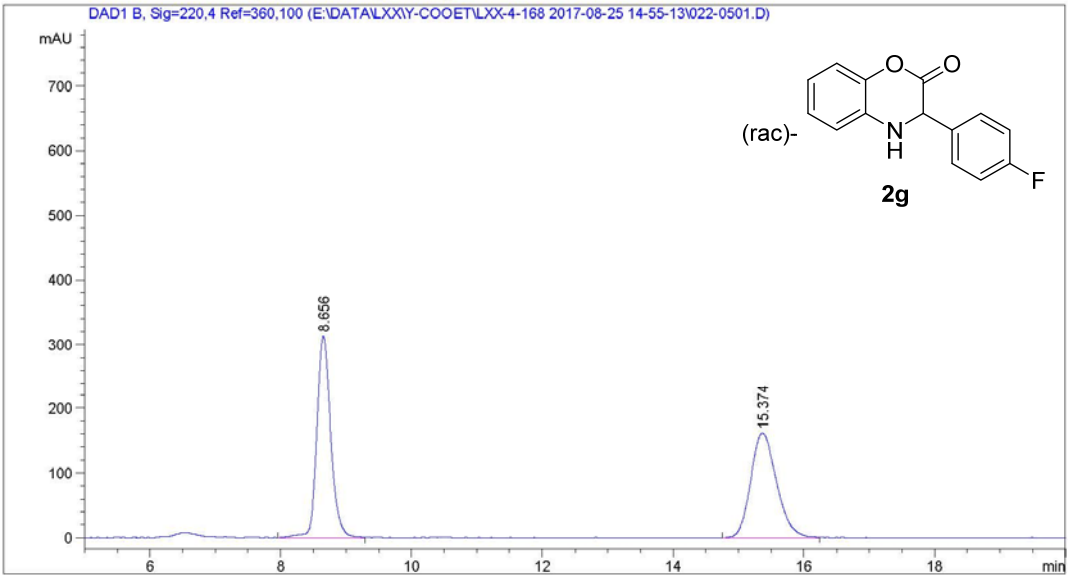
Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\075-0801.D
Sample Name: H-5-EE

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   8
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 75
Injection Date  : 9/26/2017 2:26:13 PM               Inj :   1
                                               Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-45MIN.M
Last changed    : 9/26/2017 11:46:03 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-45MIN.M (Sequence Method)
Last changed    : 9/26/2017 4:06:05 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```


```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.695 MM    0.4144   34.58571    1.39101   0.3469
   2  36.571 BB    1.1121 9935.48242  137.32356  99.6531

Totals :                  9970.06813  138.71457
```

Data File E:\DATA\LXX\Y-COOET\LXX-4-168 2017-08-25 14-55-13\022-0501.D
Sample Name: HZY-PH-4F-RAC

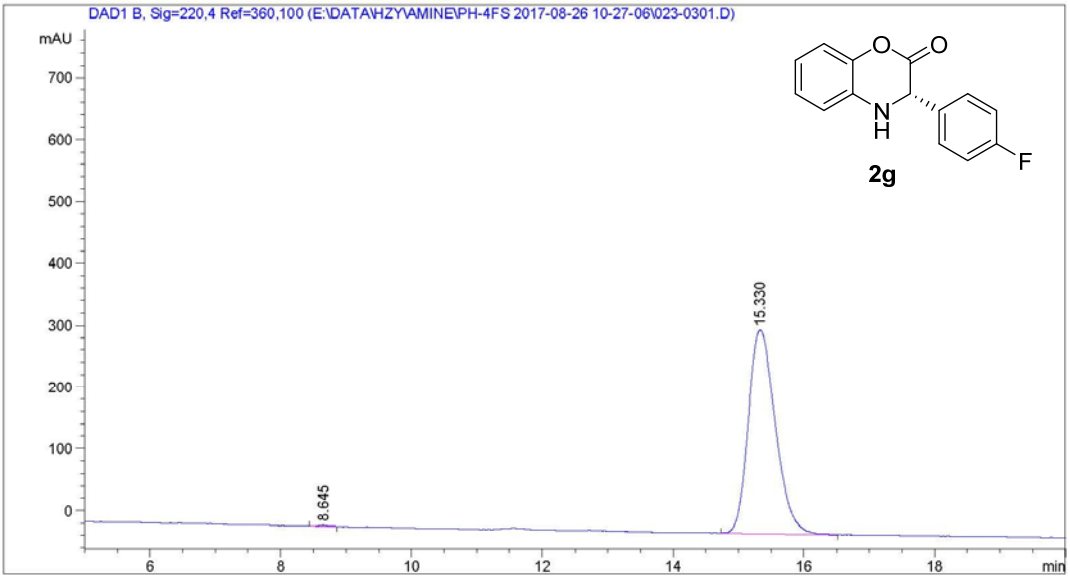
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   5
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 22
Injection Date  : 8/25/2017 4:45:20 PM               Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\LXX\Y-COOET\LXX-4-168 2017-08-25 14-55-13\DAD-OD(1-2)-80-20-1ML-1UL
                  -25MIN.M
Last changed    : 8/25/2017 3:16:42 PM by SYSTEM
Analysis Method : E:\DATA\LXX\Y-COOET\LXX-4-168 2017-08-25 14-55-13\DAD-OD(1-2)-80-20-1ML-1UL
                  -25MIN.M (Sequence Method)
Last changed    : 8/25/2017 6:32:43 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.656 BV    0.2250 4591.43213  313.25784  50.8430
   2  15.374 BV    0.4086 4439.17236  161.55267  49.1570

Totals :                  9030.60449  474.81052
```

Data File E:\DATA\HZY\AMINE\PH-4FS 2017-08-26 10-27-06\023-0301.D
Sample Name: 4F-EE-REPEAT

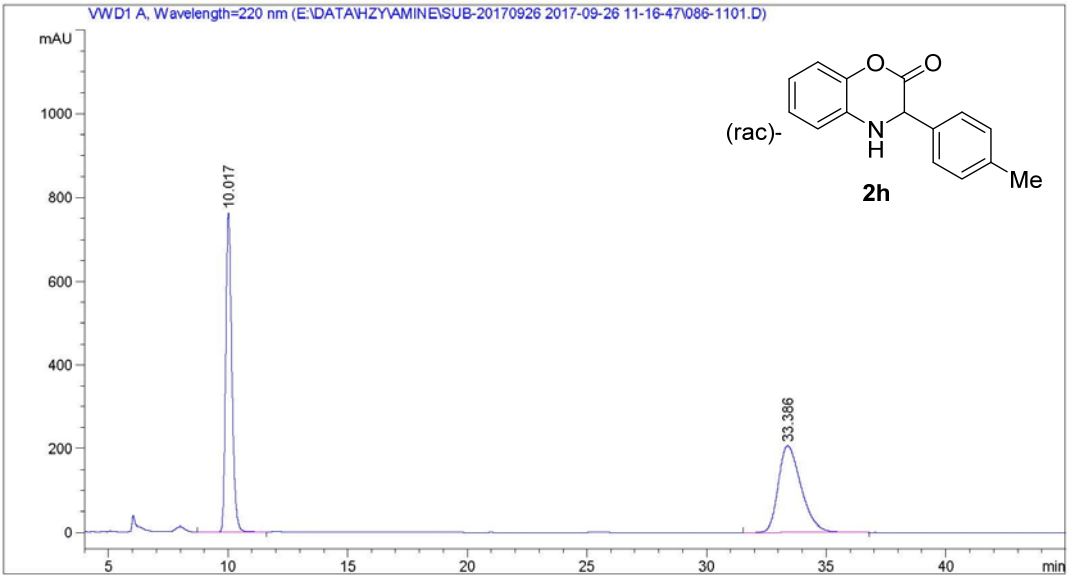
```
==================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 23
Injection Date  : 8/26/2017 11:05:11 AM             Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\PH-4FS 2017-08-26 10-27-06\DAD-OD(1-2)-80-20-1ML-1UL-
                  25MIN.M
Last changed    : 8/26/2017 11:04:04 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\PH-4FS 2017-08-26 10-27-06\DAD-OD(1-2)-80-20-1ML-1UL-
                  25MIN.M (Sequence Method)
Last changed    : 8/26/2017 11:32:59 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.645 BV    0.1455   23.99336    2.09691   0.2529
   2  15.330 VV    0.4379 9462.84766  330.66409  99.7471

Totals :                  9486.84101  332.76100
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\086-1101.D
Sample Name: H-6-RAC

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  11
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 86
Injection Date  : 9/26/2017 4:43:36 PM               Inj :   1
                                                Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-45MIN.M
Last changed    : 9/26/2017 11:46:03 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-45MIN.M (Sequence Method)
Last changed    : 9/26/2017 6:09:23 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

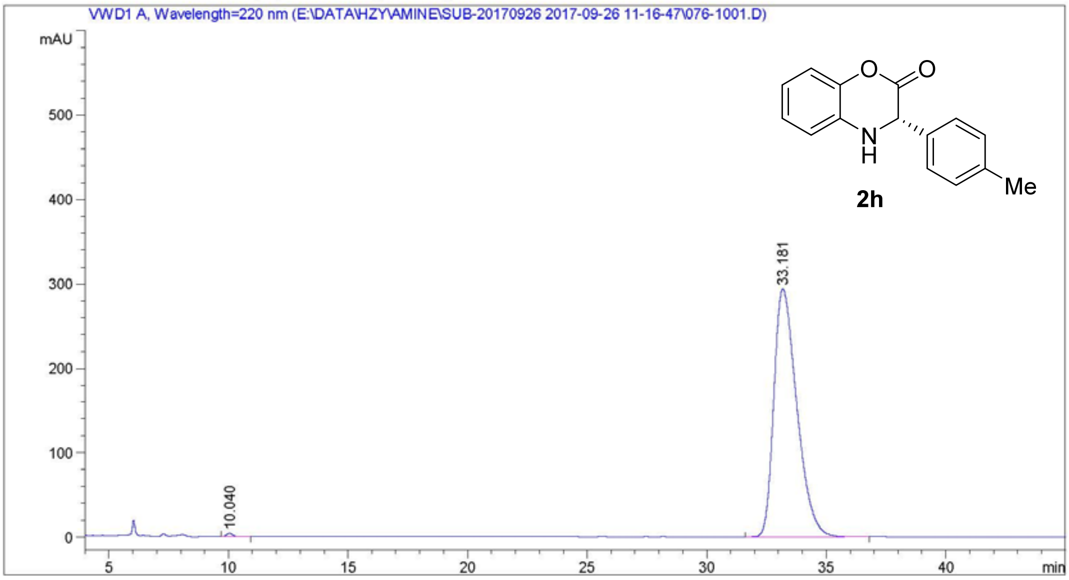



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.017 BB    0.2737 1.35925e4   763.52167  49.8999
   2  33.386 BB    1.0173 1.36470e4   205.88000  50.1001

Totals :                 2.72395e4   969.40167
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\076-1001.D
Sample Name: H-6-EE

===

Acq. Operator : SYSTEM   Seq. Line : 10
Acq. Instrument : 1260HPLC-VWD   Location : Vial 76
Injection Date : 9/26/2017 3:57:50 PM   Inj : 1
                                               Inj Volume : 3.000 µl
Acq. Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-3UL-220NM-45MIN.M
Last changed : 9/26/2017 11:46:03 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-3UL-220NM-45MIN.M (Sequence Method)
Last changed : 9/26/2017 5:16:58 PM by SYSTEM
(modified after loading)
Additional Info : Peak(s) manually integrated



===
Area Percent Report
===

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

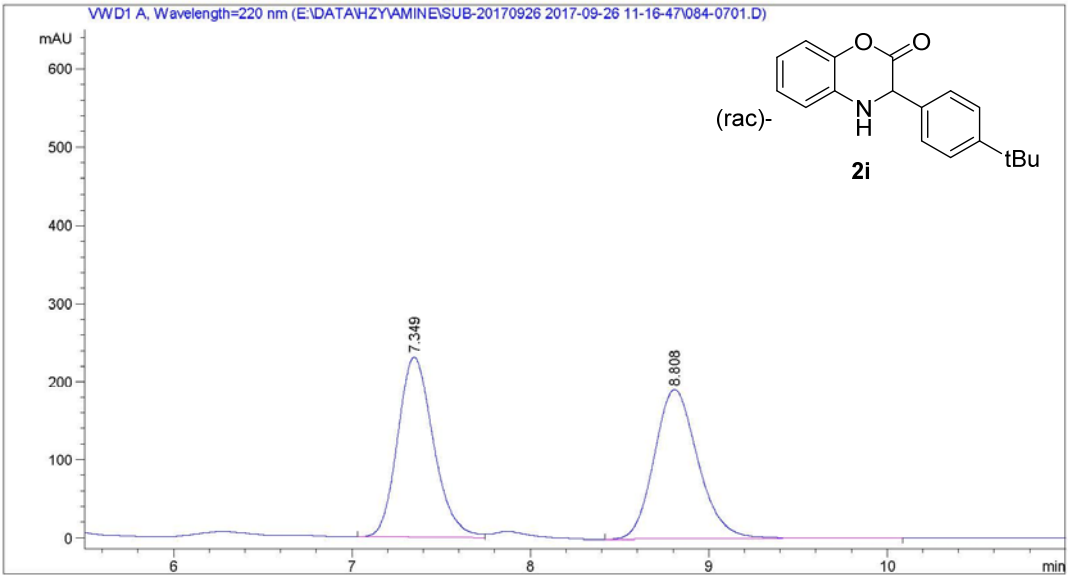
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.040	BB	0.2714	71.31303	4.03077	0.3616
2	33.181	BB	1.0232	1.96487e4	293.80399	99.6384

Totals : 1.97200e4 297.83475

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\084-0701.D
Sample Name: H-4-RAC

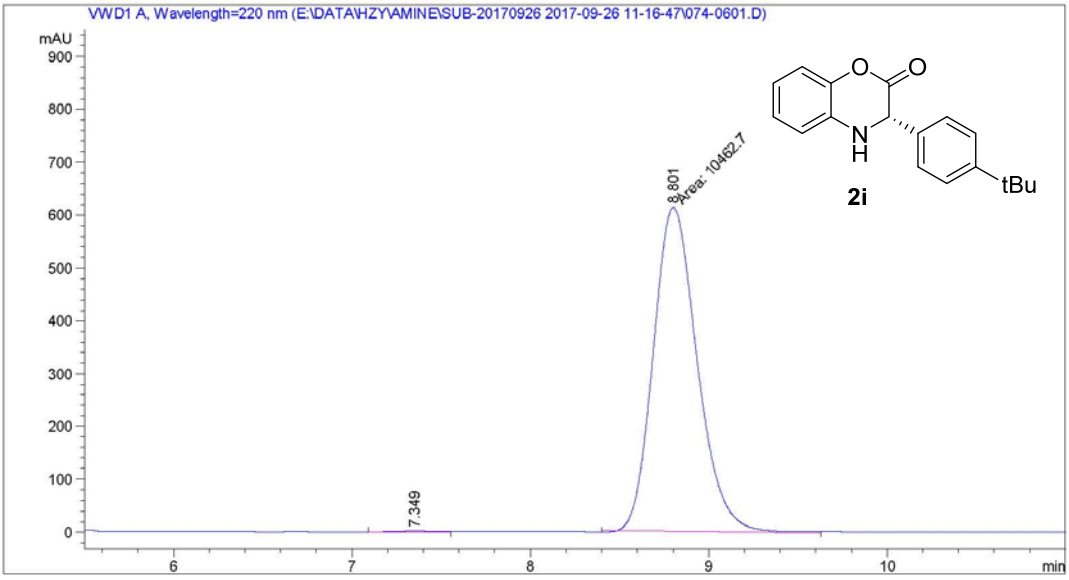
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   7
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 84
Injection Date  : 9/26/2017 2:00:20 PM               Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  2UL-220NM-25MIN.M
Last changed    : 9/26/2017 11:44:47 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  2UL-220NM-25MIN.M (Sequence Method)
Last changed    : 9/26/2017 2:48:41 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.349 BV    0.2148 3224.69531  231.08284  49.8026
   2   8.808 BB    0.2624 3250.25928  190.18265  50.1974

Totals :                  6474.95459  421.26549
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\074-0601.D
Sample Name: H-4-EE

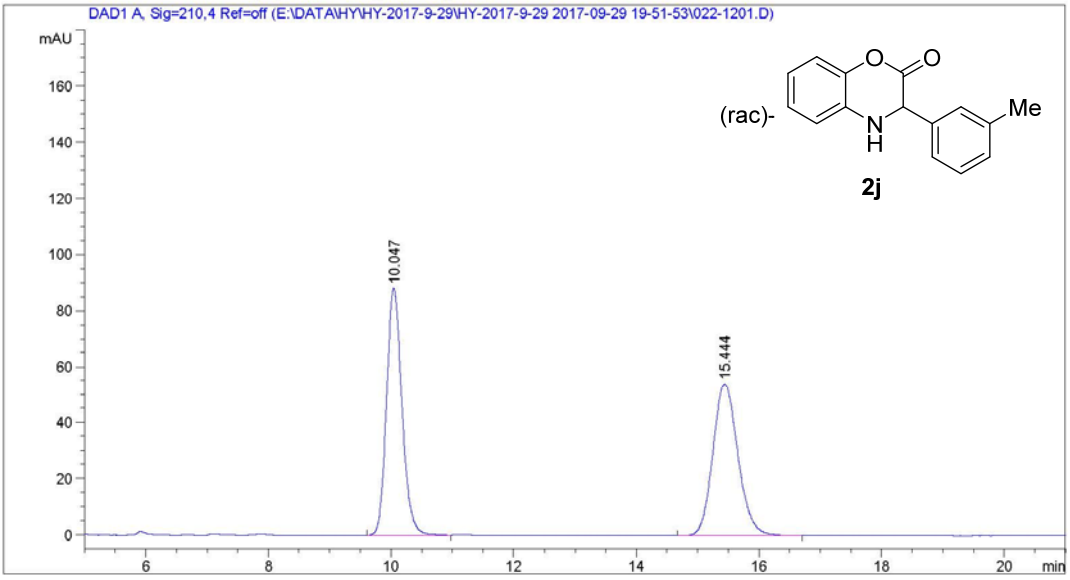
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   6
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 74
Injection Date  : 9/26/2017 1:34:34 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  2UL-220NM-25MIN.M
Last changed    : 9/26/2017 11:44:47 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  2UL-220NM-25MIN.M (Sequence Method)
Last changed    : 9/26/2017 2:49:32 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.349 BV    0.2105   32.48330    2.34623   0.3095
   2   8.801 MM    0.2845 1.04627e4   612.96301  99.6905

Totals :                  1.04952e4   615.30924
```

Data File E:\DATA\HY\HY-2017-9-29\HY-2017-9-29 2017-09-29 19-51-53\022-1201.D
Sample Name: PH-3ME-RAC

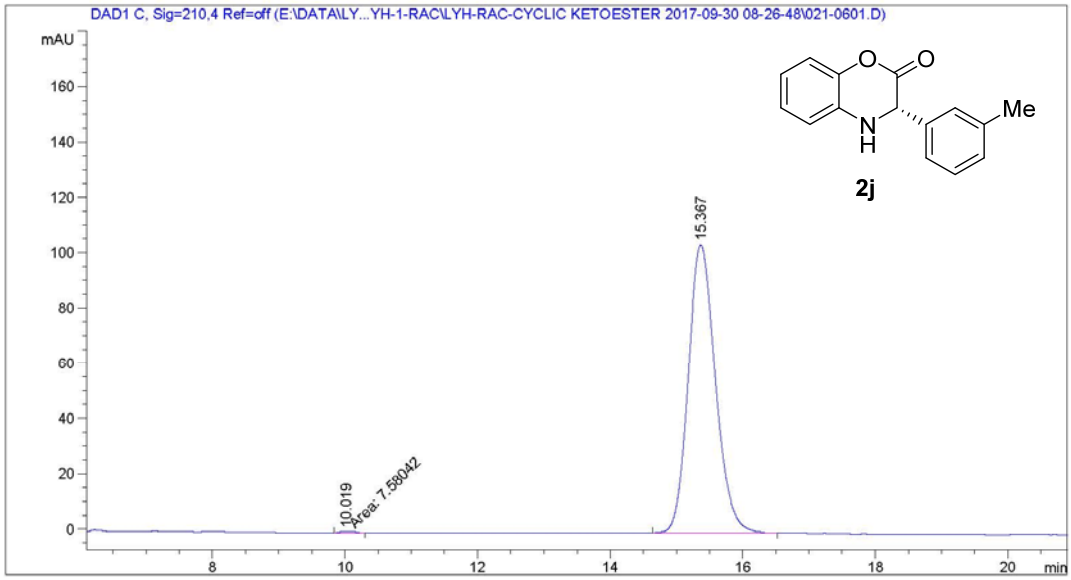
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :  12
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 22
Injection Date  : 9/29/2017 11:37:58 PM              Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HY\HY-2017-9-29\HY-2017-9-29 2017-09-29 19-51-53\DAD-OD(1-2)-80-20-
                  1ML-1UL- ALL-50MIN.M
Last changed    : 9/29/2017 8:27:55 PM by SYSTEM
Analysis Method : E:\DATA\HY\HY-2017-9-29\HY-2017-9-29 2017-09-29 19-51-53\DAD-OD(1-2)-80-20-
                  1ML-1UL- ALL-50MIN.M (Sequence Method)
Last changed    : 9/30/2017 9:12:45 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.047 BB    0.2665 1532.39600   88.28191  50.0109
   2  15.444 BB    0.4317 1531.72644   54.19786  49.9891

Totals :                  3064.12244  142.47977
```

Data File E:\DATA\LYH\LYH-1-RAC\LYH-RAC-CYCLIC KETOESTER 2017-09-30 08-26-48\021-0601.D
Sample Name: 3ME

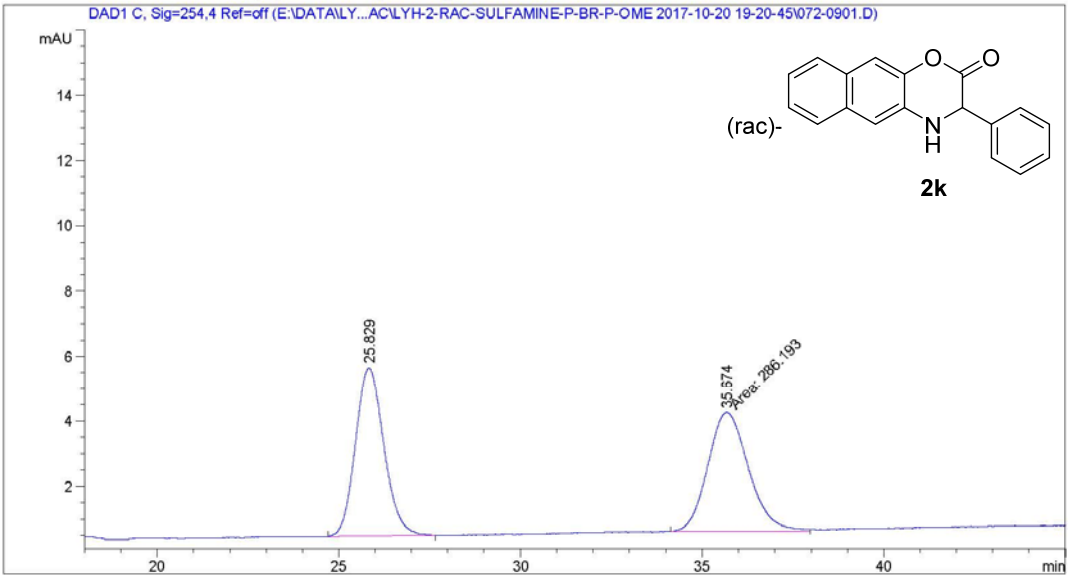
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   6
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 21
Injection Date  : 9/30/2017 10:32:26 AM             Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\LYH\LYH-1-RAC\LYH-RAC-CYCLIC KETOESTER 2017-09-30 08-26-48\DAD-OD(1
                  -2)-80-20-1.0ML-5-ALL-25MIN.M
Last changed    : 9/30/2017 10:51:11 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\LYH\LYH-1-RAC\LYH-RAC-CYCLIC KETOESTER 2017-09-30 08-26-48\DAD-OD(1
                  -2)-80-20-1.0ML-5-ALL-25MIN.M (Sequence Method)
Last changed    : 9/30/2017 10:56:16 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.019 MM    0.2668    7.58042 4.73497e-1   0.2560
   2  15.367 BB    0.4370 2953.37378  104.11096  99.7440

Totals :                  2960.95420  104.58446
```

Data File E:\DATA\LY...2-RAC\LYH-2-RAC-SULFAMINE-P-BR-P-OME 2017-10-20 19-20-45\072-0901.D
Sample Name: NAI1-PDC

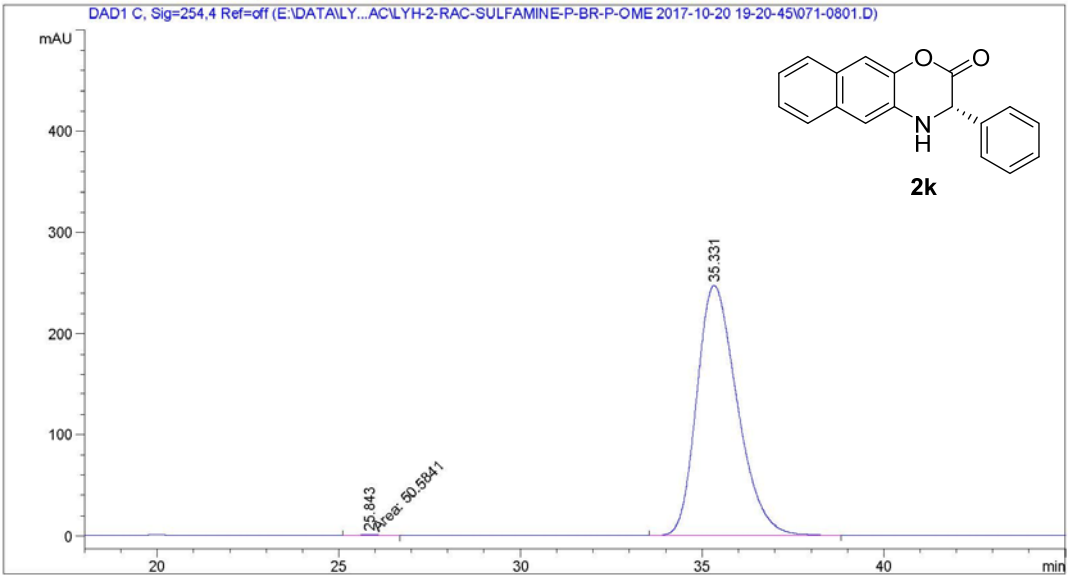
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   9
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 72
Injection Date  : 10/20/2017 11:44:08 PM             Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\LYH\LYH-2-RAC\LYH-2-RAC-SULFAMINE-P-BR-P-OME 2017-10-20 19-20-45
                  \DAD-OD(1-2)-80-20-1ML-1UL- ALL-50MIN.M
Last changed    : 10/20/2017 10:02:57 PM by SYSTEM
Analysis Method : E:\DATA\LYH\LYH-2-RAC\LYH-2-RAC-SULFAMINE-P-BR-P-OME 2017-10-20 19-20-45
                  \DAD-OD(1-2)-80-20-1ML-1UL- ALL-50MIN.M (Sequence Method)
Last changed    : 10/21/2017 9:29:28 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.829 BB    0.7353  280.31314    5.16417  49.4811
   2  35.674 MM    1.3062  286.19260    3.65182  50.5189

Totals :                   566.50574    8.81599
```

Data File E:\DATA\LY...2-RAC\LYH-2-RAC-SULFAMINE-P-BR-P-OME 2017-10-20 19-20-45\071-0801.D
Sample Name: NAI1-EE

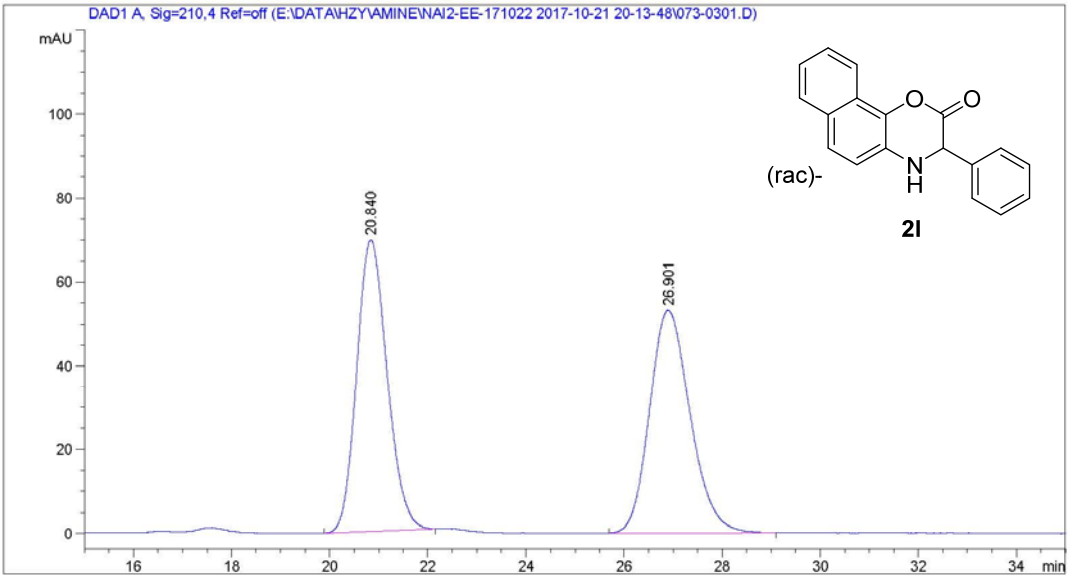
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   8
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 71
Injection Date  : 10/20/2017 10:53:14 PM             Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\LYH\LYH-2-RAC\LYH-2-RAC-SULFAMINE-P-BR-P-OME 2017-10-20 19-20-45
                  \DAD-OD(1-2)-80-20-1ML-1UL- ALL-50MIN.M
Last changed    : 10/20/2017 10:02:57 PM by SYSTEM
Analysis Method : E:\DATA\LYH\LYH-2-RAC\LYH-2-RAC-SULFAMINE-P-BR-P-OME 2017-10-20 19-20-45
                  \DAD-OD(1-2)-80-20-1ML-1UL- ALL-50MIN.M (Sequence Method)
Last changed    : 10/21/2017 9:32:18 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.843 MM    0.8746   50.58405 9.63933e-1   0.2643
   2  35.331 BB    1.1891 1.90906e4   247.16516  99.7357

Totals :                  1.91412e4   248.12909
```

Data File E:\DATA\HZY\AMINE\NAI2-EE-171022 2017-10-21 20-13-48\073-0301.D
Sample Name: NAI2-RAC

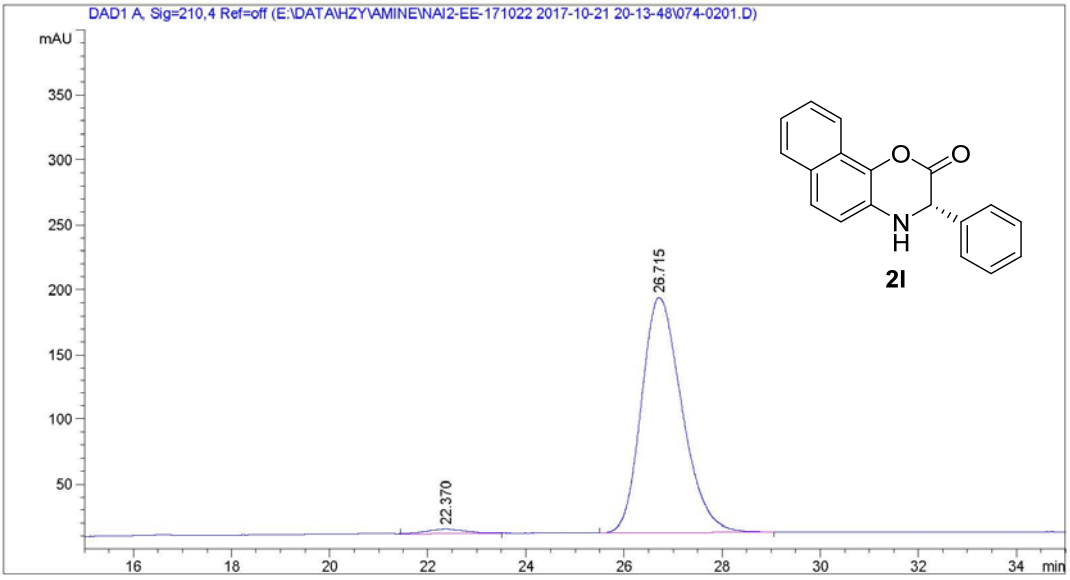
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 73
Injection Date  : 10/21/2017 9:19:14 PM              Inj :   1
                                                Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\NAI2-EE-171022 2017-10-21 20-13-48\DAD-OD(1-2)-80-20-1ML-
                  1UL- ALL-50MIN.M
Last changed    : 10/21/2017 8:13:49 PM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\NAI2-EE-171022 2017-10-21 20-13-48\DAD-OD(1-2)-80-20-1ML-
                  1UL- ALL-50MIN.M (Sequence Method)
Last changed    : 10/23/2017 9:47:27 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.840 BB    0.6517 2993.80566   69.61648  49.5497
   2  26.901 BB    0.8577 3048.21777   53.26270  50.4503

Totals :                  6042.02344  122.87918
```

Data File E:\DATA\HZY\AMINE\NAI2-EE-171022 2017-10-21 20-13-48\074-0201.D
Sample Name: NAI2-EE

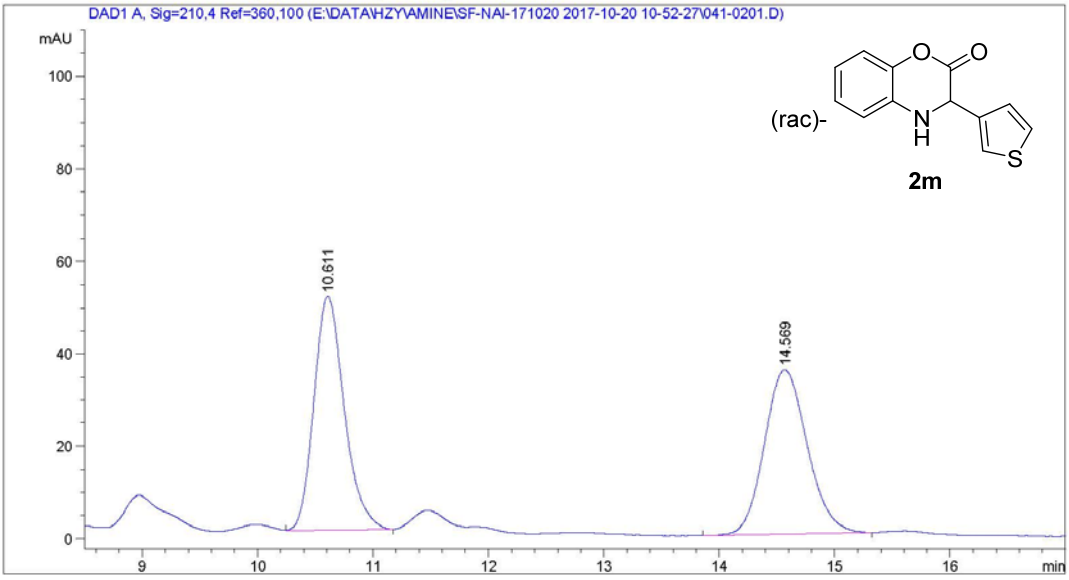
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 74
Injection Date  : 10/21/2017 8:28:18 PM             Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\NAI2-EE-171022 2017-10-21 20-13-48\DAD-OD(1-2)-80-20-1ML-
                  1UL- ALL-50MIN.M
Last changed    : 10/21/2017 8:13:49 PM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\NAI2-EE-171022 2017-10-21 20-13-48\DAD-OD(1-2)-80-20-1ML-
                  1UL- ALL-50MIN.M (Sequence Method)
Last changed    : 10/23/2017 9:48:35 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  22.370 BB    0.6377  180.75034    3.32791   1.7128
   2  26.715 BB    0.8809 1.03722e4   181.43884  98.2872

Totals :                  1.05529e4   184.76675
```

Data File E:\DATA\HZY\AMINE\SF-NAI-171020 2017-10-20 10-52-27\041-0201.D
Sample Name: SF-PDC

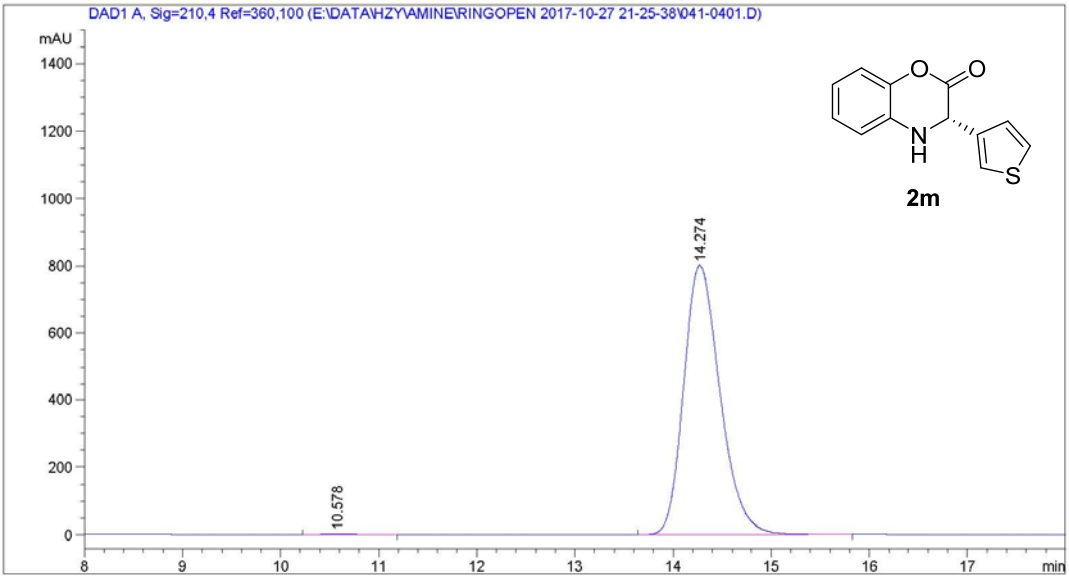
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 41
Injection Date  : 10/20/2017 11:04:22 AM             Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SF-NAI-171020 2017-10-20 10-52-27\DAD-OD(1-2)-80-20-1ML-
                  1UL-30MIN.M
Last changed    : 10/20/2017 10:52:28 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SF-NAI-171020 2017-10-20 10-52-27\DAD-OD(1-2)-80-20-1ML-
                  1UL-30MIN.M (Sequence Method)
Last changed    : 10/25/2017 10:38:23 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.611 VB    0.2784  921.38617   50.62161  50.3326
   2  14.569 BB    0.3980  909.20764   35.59722  49.6674

Totals :                  1830.59381   86.21883
```

Data File E:\DATA\HZY\AMINE\RINGOPEN 2017-10-27 21-25-38\041-0401.D
Sample Name: SF-20171027

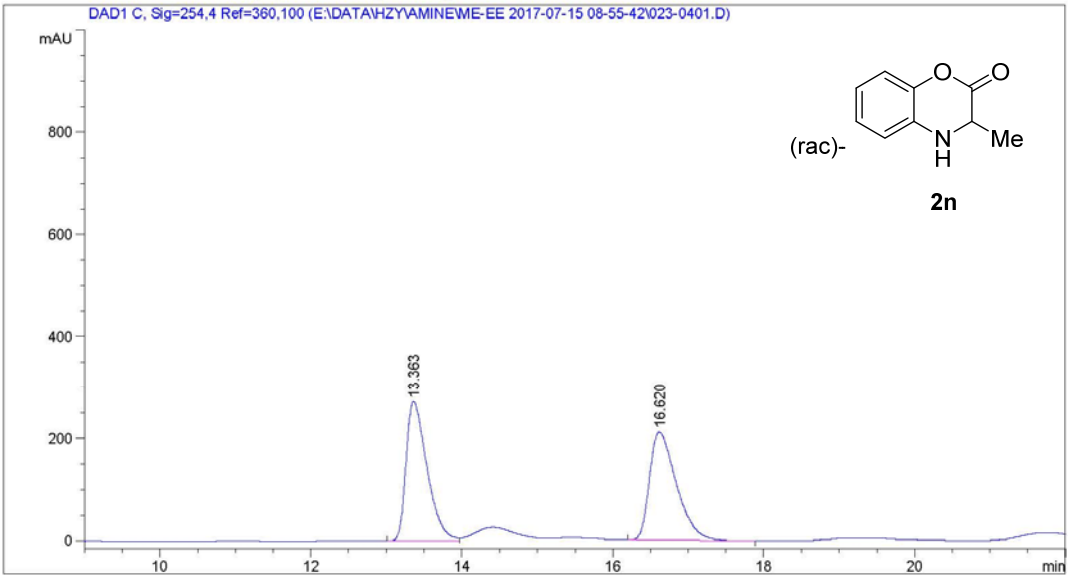
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 41
Injection Date  : 10/27/2017 10:27:04 PM             Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\RINGOPEN 2017-10-27 21-25-38\DAD-OD(1-2)-80-20-1ML-1UL-
                  30MIN.M
Last changed    : 10/27/2017 9:25:38 PM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\RINGOPEN 2017-10-27 21-25-38\DAD-OD(1-2)-80-20-1ML-1UL-
                  30MIN.M (Sequence Method)
Last changed    : 10/30/2017 9:05:45 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.578 BB    0.2685   53.75104    2.97956   0.2604
   2  14.274 BB    0.3959 2.05885e4   801.00885  99.7396

Totals :                  2.06422e4   803.98841
```

Data File E:\DATA\HZY\AMINE\ME-EE 2017-07-15 08-55-42\023-0401.D
Sample Name: ME-PDC

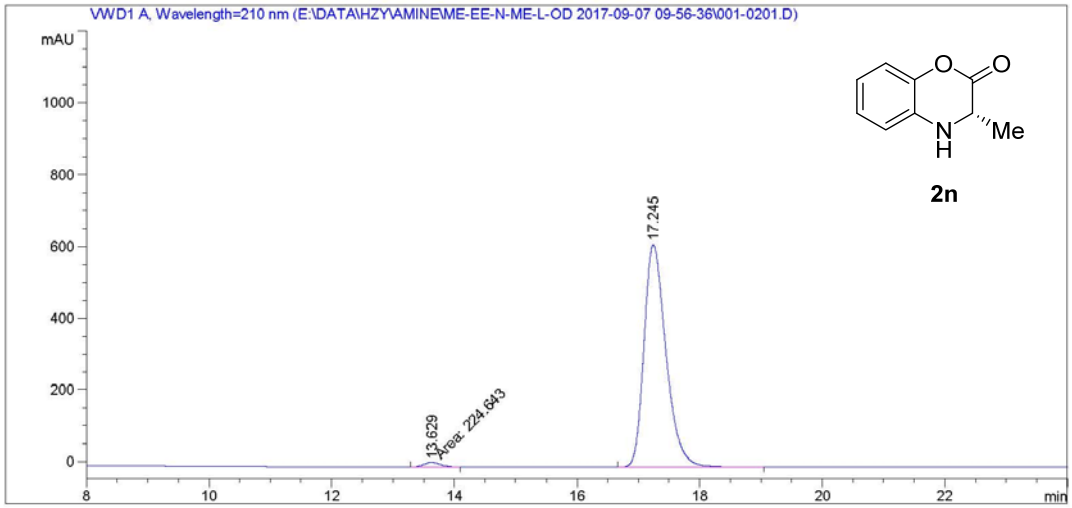
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 23
Injection Date  : 7/15/2017 9:40:12 AM               Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\ME-EE 2017-07-15 08-55-42\DAD-OD(1-2)-90-10-1ML-2UL-25MIN
                  .M
Last changed    : 7/15/2017 8:55:42 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\ME-EE 2017-07-15 08-55-42\DAD-OD(1-2)-90-10-1ML-2UL-25MIN
                  .M (Sequence Method)
Last changed    : 7/17/2017 10:18:56 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.363 BV    0.2925 5232.71631  271.98199  50.0964
   2  16.620 BB    0.3722 5212.57422  210.97859  49.9036

Totals :                 1.04453e4   482.96059
```

Data File E:\DATA\HZY\AMINE\ME-EE-N-ME-L-OD 2017-09-07 09-56-36\001-0201.D
Sample Name: Hex

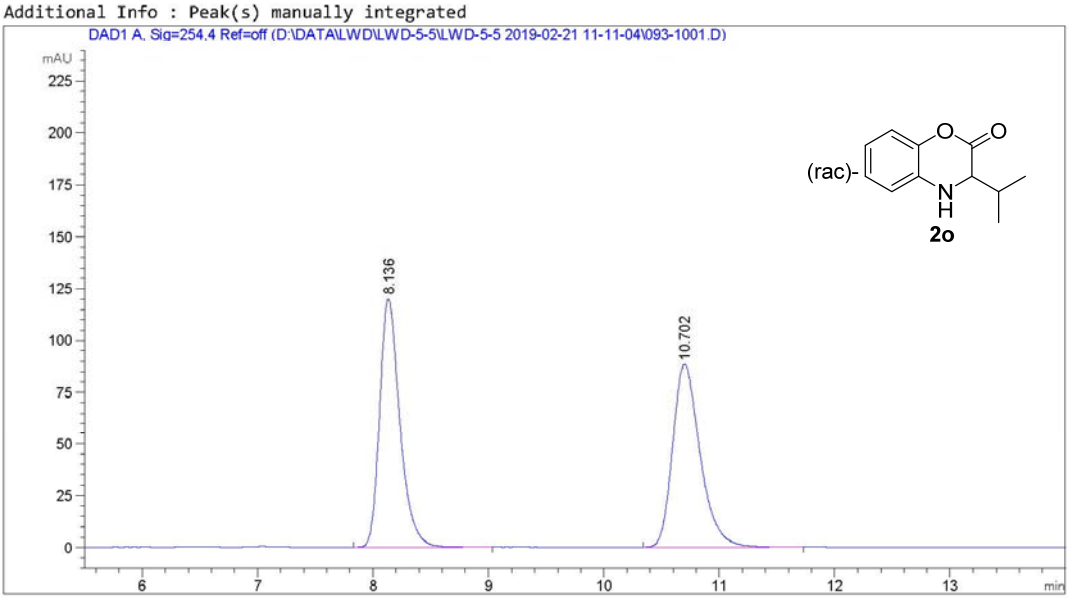
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 1
Injection Date  : 9/7/2017 10:10:03 AM               Inj :   1
                                                Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\ME-EE-N-ME-L-OD 2017-09-07 09-56-36\VWD-AD(1-6)-90-10-1ML
                  -1UL-10MIN.M
Last changed    : 9/7/2017 10:34:42 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\AMINE\ME-EE-N-ME-L-OD 2017-09-07 09-56-36\VWD-AD(1-6)-90-10-1ML
                  -1UL-10MIN.M (Sequence Method)
Last changed    : 9/7/2017 10:35:48 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.629 MM    0.2945  224.64258   12.71405   1.4388
   2  17.245 BB    0.3804 1.53891e4   620.48462  98.5612

Totals :                  1.56137e4   633.19866

==========================================================================
```

Data File D:\DATA\LWD\LWD-5-5\LWD-5-5 2019-02-21 11-11-04\093-1001.D
Sample Name: IPR-RAC

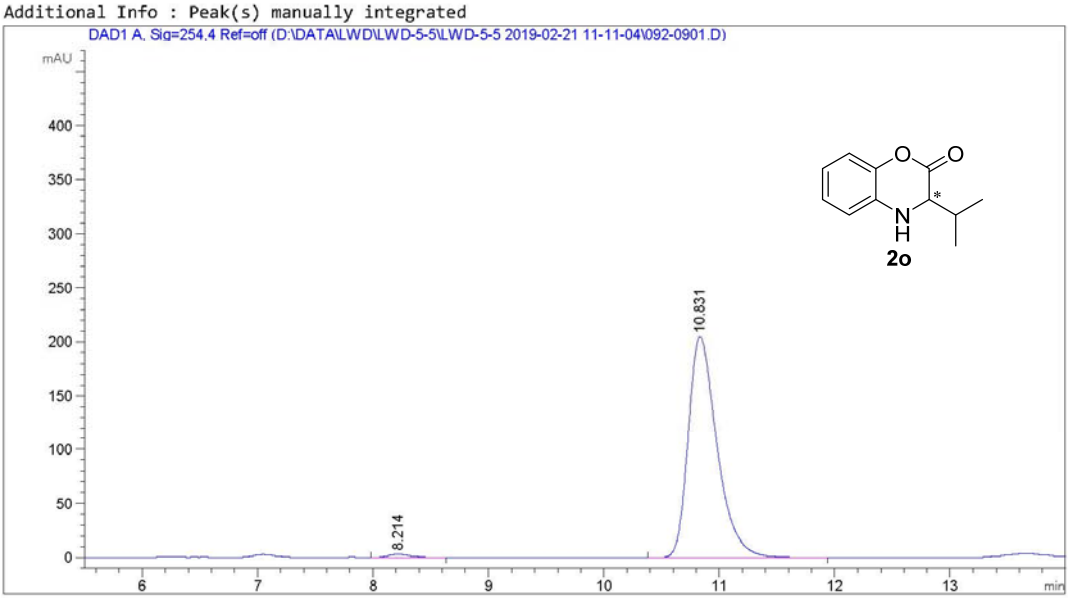
```
==========================================================================
Acq. Operator   :                              Seq. Line :  10
Acq. Instrument : Instrument 2                  Location : Vial 93
Injection Date  : 2/21/2019 2:51:38 PM               Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : D:\DATA\LWD\LWD-5-5\LWD-5-5 2019-02-21 11-11-04\DAD-OD(1-2)-90-10-1ML-2UL-
                  ALL-40MIN.M
Last changed    : 9/22/2018 11:08:15 AM
Analysis Method : D:\METHOD\HZY\DAD-OJ(1-6)-80-20-1ML-1UL-ALL-50MIN.M
Last changed    : 2/21/2019 4:18:37 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.136 BB    0.1837 1457.57532  120.01909  49.9095
   2  10.702 BB    0.2508 1462.86389   88.50265  50.0905

Totals :                  2920.43921  208.52174
```

Data File D:\DATA\LWD\LWD-5-5\LWD-5-5 2019-02-21 11-11-04\092-0901.D
Sample Name: IPR-EE

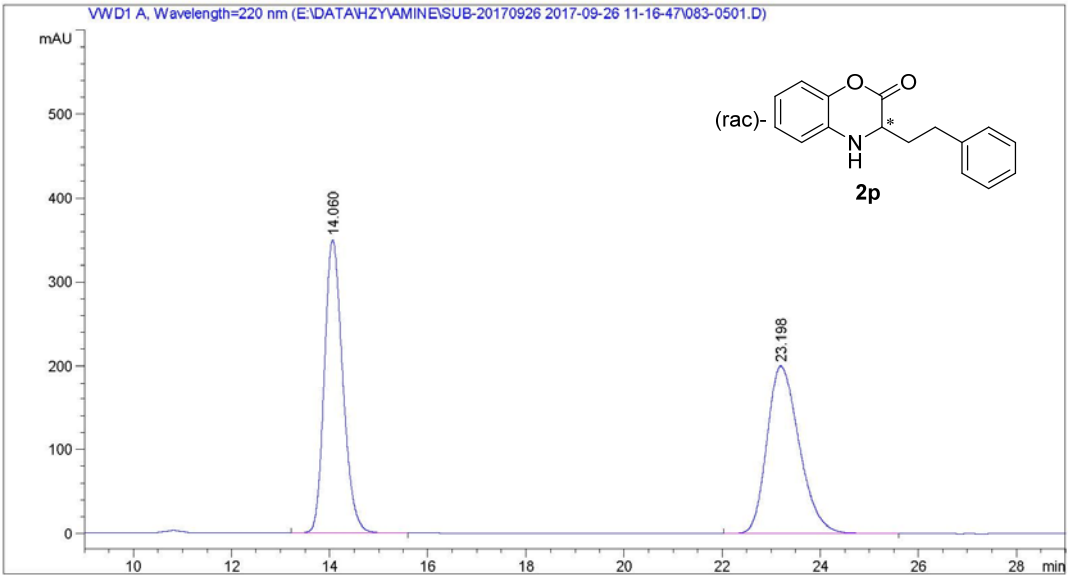
```
==========================================================================
Acq. Operator   :                              Seq. Line :   9
Acq. Instrument : Instrument 2                  Location : Vial 92
Injection Date  : 2/21/2019 2:10:40 PM               Inj :   1
                                              Inj Volume : 2.000 µl
Acq. Method     : D:\DATA\LWD\LWD-5-5\LWD-5-5 2019-02-21 11-11-04\DAD-OD(1-2)-90-10-1ML-2UL-
                  ALL-40MIN.M
Last changed    : 9/22/2018 11:08:15 AM
Analysis Method : D:\METHOD\HZY\DAD-OJ(1-6)-80-20-1ML-1UL-ALL-50MIN.M
Last changed    : 2/21/2019 4:20:02 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.214 BB    0.1930   39.29892    3.07772   1.1013
   2  10.831 BB    0.2611 3529.16602  204.70740  98.8987

Totals :                  3568.46493  207.78512
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\083-0501.D
Sample Name: H-3-RAC

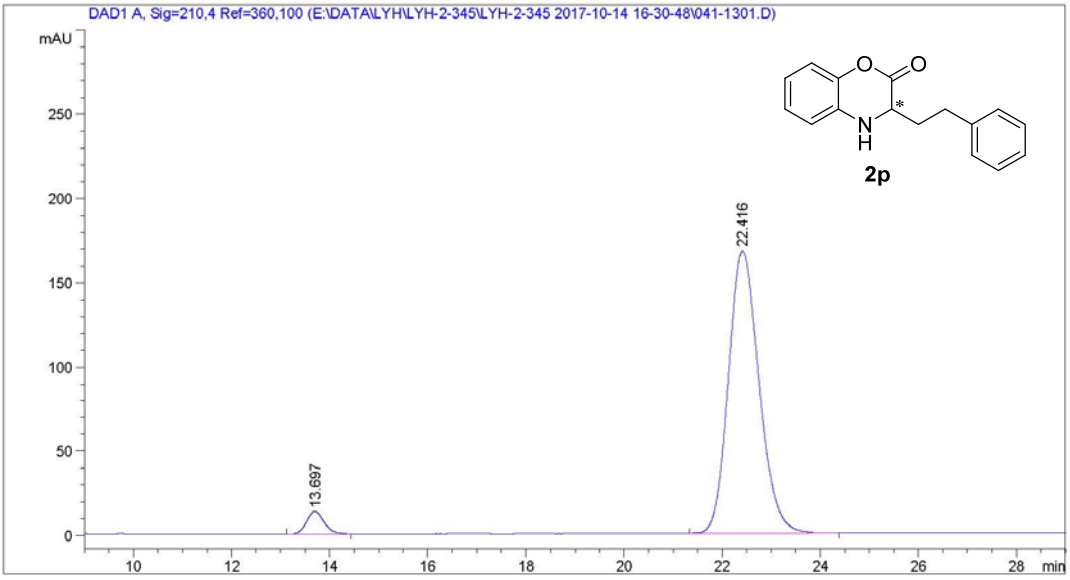
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : 1260HPLC-VWD                     Location : Vial 83
Injection Date  : 9/26/2017 12:58:42 PM                Inj :   1
                                                  Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-35MIN.M
Last changed    : 9/26/2017 11:47:09 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  3UL-220NM-35MIN.M (Sequence Method)
Last changed    : 10/16/2017 10:21:43 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.060 BB    0.4013 9087.33691  349.60501  49.9043
   2  23.198 BB    0.7052 9122.19727  199.98453  50.0957

Totals :                 1.82095e4   549.58954
```

Data File E:\DATA\LYH\LYH-2-345\LYH-2-345 2017-10-14 16-30-48\041-1301.D
Sample Name: HZY-MEMEPH

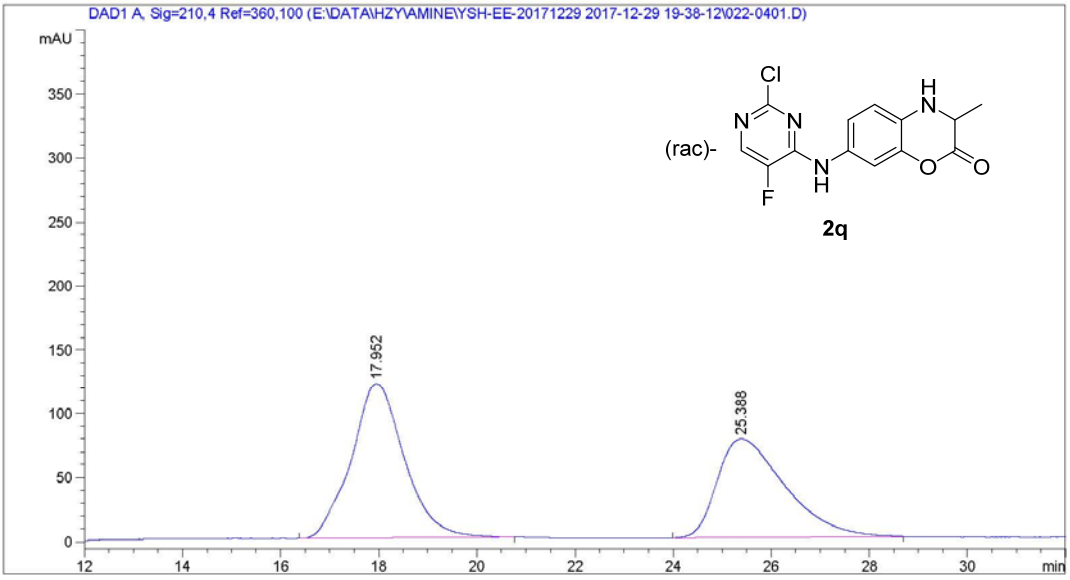
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  13
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 41
Injection Date  : 10/14/2017 9:53:16 PM               Inj :   1
                                                Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\LYH\LYH-2-345\LYH-2-345 2017-10-14 16-30-48\DAD-OD(1-2)-80-20-1ML-
                  1UL-30MIN.M
Last changed    : 10/14/2017 8:22:51 PM by SYSTEM
Analysis Method : E:\DATA\LYH\LYH-2-345\LYH-2-345 2017-10-14 16-30-48\DAD-OD(1-2)-80-20-1ML-
                  1UL-30MIN.M (Sequence Method)
Last changed    : 10/16/2017 10:20:06 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.697 BB    0.3763  324.98087   13.24320   4.2965
   2  22.416 BB    0.6643 7238.87744  167.45334  95.7035

Totals :                  7563.85831  180.69654
```

Data File E:\DATA\HZY\AMINE\YSH-EE-20171229 2017-12-29 19-38-12\022-0401.D
Sample Name: YSH-BINAP

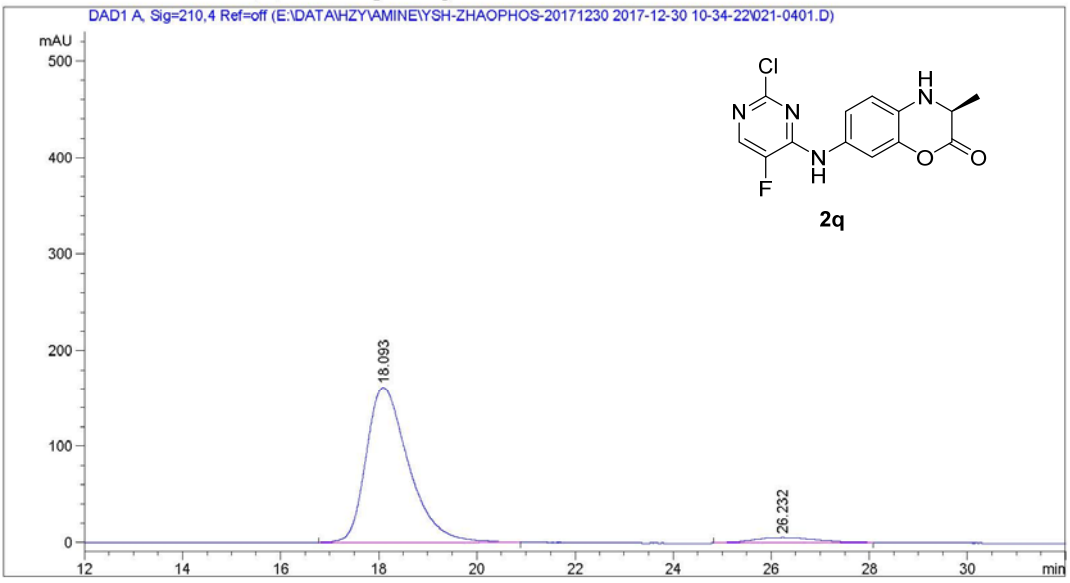
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 22
Injection Date  : 12/29/2017 9:22:22 PM             Inj :   1
                                               Inj Volume : 25.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\YSH-EE-20171229 2017-12-29 19-38-12\DAD-OD(1-2)-80-20-1ML
                  -25UL-70MIN.M
Last changed    : 12/29/2017 7:38:12 PM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\YSH-EE-20171229 2017-12-29 19-38-12\DAD-OD(1-2)-80-20-1ML
                  -25UL-70MIN.M (Sequence Method)
Last changed    : 1/3/2018 3:38:45 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.952 BB    1.0556 9064.62305  120.77467  55.2085
   2  25.388 BB    1.2547 7354.26172   76.69269  44.7915

Totals :                  1.64189e4   197.46736
```

Data File E:\DATA\HZY\AMINE\YSH-ZHAOPHOS-20171230 2017-12-30 10-34-22\021-0401.D
Sample Name: ysh-ee

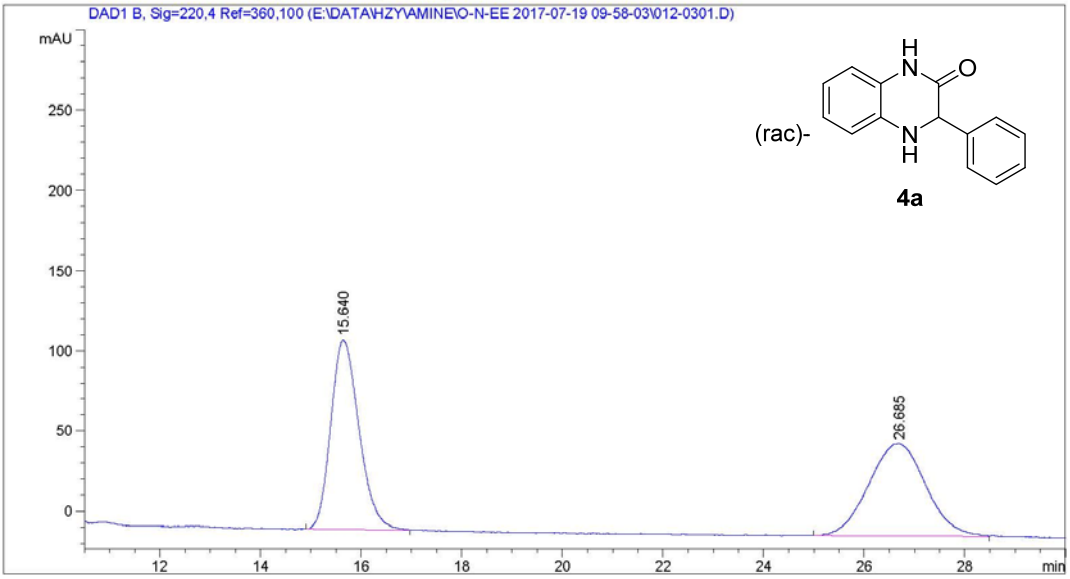
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 21
Injection Date  : 12/30/2017 12:01:28 PM             Inj :   1
                                                 Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\YSH-ZHAOPHOS-20171230 2017-12-30 10-34-22\DAD-OD(1-2)-80-
                  20-1ML-1UL- ALL-50MIN.M
Last changed    : 12/30/2017 10:34:26 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\YSH-ZHAOPHOS-20171230 2017-12-30 10-34-22\DAD-OD(1-2)-80-
                  20-1ML-1UL- ALL-50MIN.M (Sequence Method)
Last changed    : 1/3/2018 3:36:55 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.093 BB    0.8958 9664.89551  161.57863  95.3760
   2  26.232 BB    1.0222  468.56891    5.44397   4.6240

Totals :                  1.01335e4   167.02260
```

Data File E:\DATA\HZY\AMINE\O-N-EE 2017-07-19 09-58-03\012-0301.D
Sample Name: amide-RAC

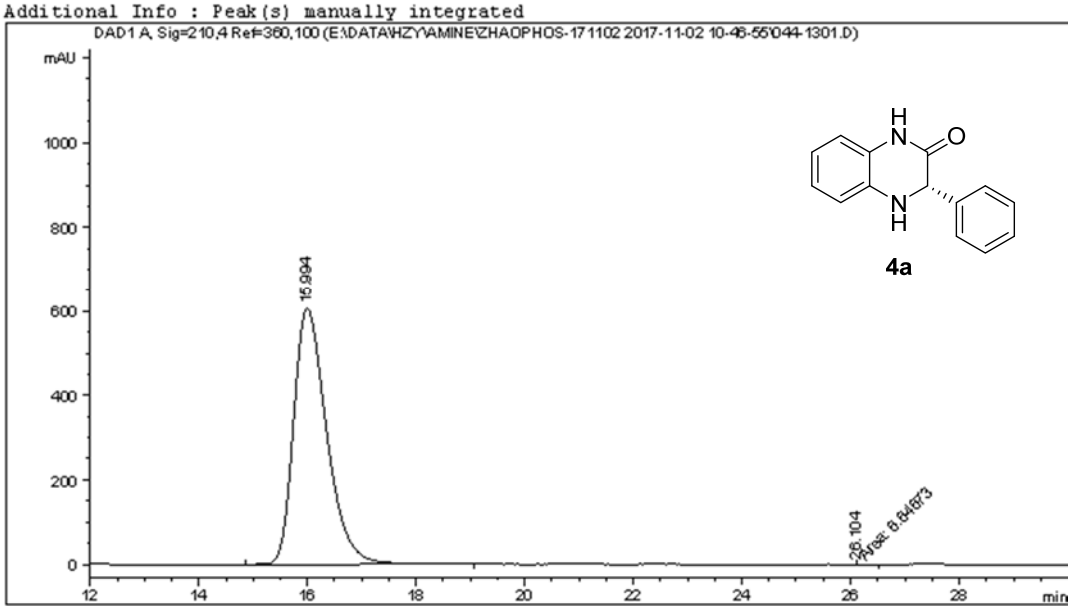
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 12
Injection Date  : 7/19/2017 10:44:30 AM               Inj :   1
                                                Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\O-N-EE 2017-07-19 09-58-03\DAD-OD(1-2)-80-20-1ML-1UL-
                  30MIN.M
Last changed    : 7/19/2017 9:59:10 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\O-N-EE 2017-07-19 09-58-03\DAD-OD(1-2)-80-20-1ML-1UL-
                  30MIN.M (Sequence Method)
Last changed    : 7/19/2017 4:30:58 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.640 BV    0.5234 4606.42627  118.53674  50.3981
   2  26.685 BV    0.9323 4533.64551   57.59998  49.6019

Totals :                  9140.07178  176.13672
```

Data File E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\044-1301.D
Sample Name: H-12-30MIN

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  13
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 44
    Injection Date  : 11/2/2017 4:29:24 PM               Inj :  1
                                                   Inj Volume : 1.000 µl
    Acq. Method     : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-30MIN.M
    Last changed    : 11/2/2017 3:04:51 PM by SYSTEM
    Analysis Method : E:\DATA\HZY\AMINE\ZHAOPHOS-171102 2017-11-02 10-46-55\DAD-OD(1-2)-80-20-
                      1ML-1UL-30MIN.M (Sequence Method)
    Last changed    : 11/2/2017 5:01:14 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

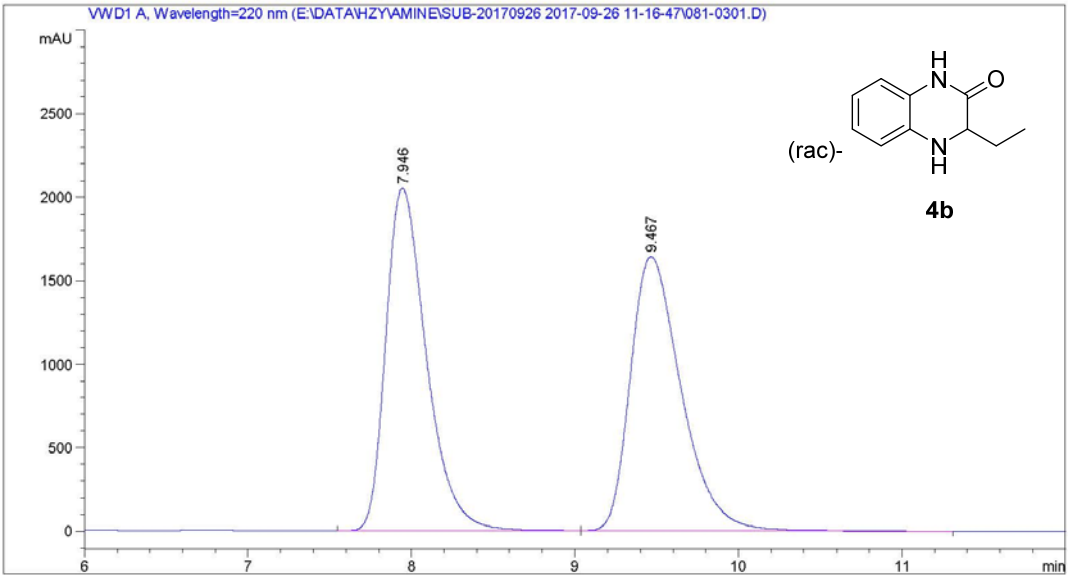


4a

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  15.994 BB    0.6469 2.56347e4   606.70441  99.9741
       2  26.104 MM    0.2573    6.64673 3.05338e-1   0.0259

    Totals :                  2.56414e4   607.00975


    ====================================================================
                              *** End of Report ***
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\081-0301.D
Sample Name: H-1-RAC

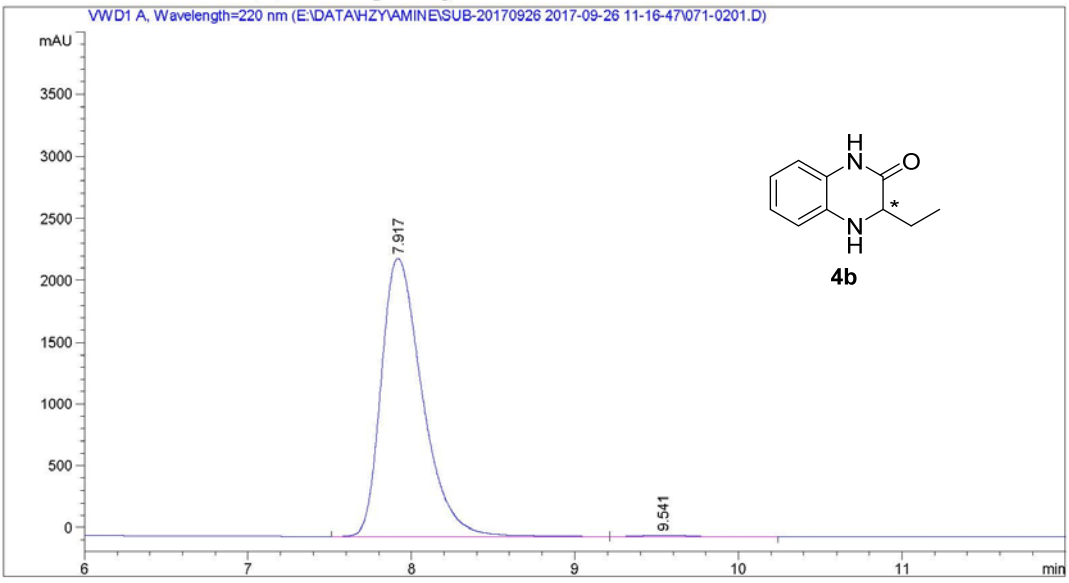
```
==================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 81
Injection Date  : 9/26/2017 11:57:10 AM              Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  2UL-220NM-25MIN.M
Last changed    : 9/26/2017 11:44:47 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  2UL-220NM-25MIN.M (Sequence Method)
Last changed    : 9/26/2017 2:39:29 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.946 VB    0.2637 3.52535e4  2049.50537  49.9302
   2   9.467 BB    0.3313 3.53521e4  1641.07715  50.0698

Totals :                  7.06057e4  3690.58252
```

Data File E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\071-0201.D
Sample Name: H-1-EE

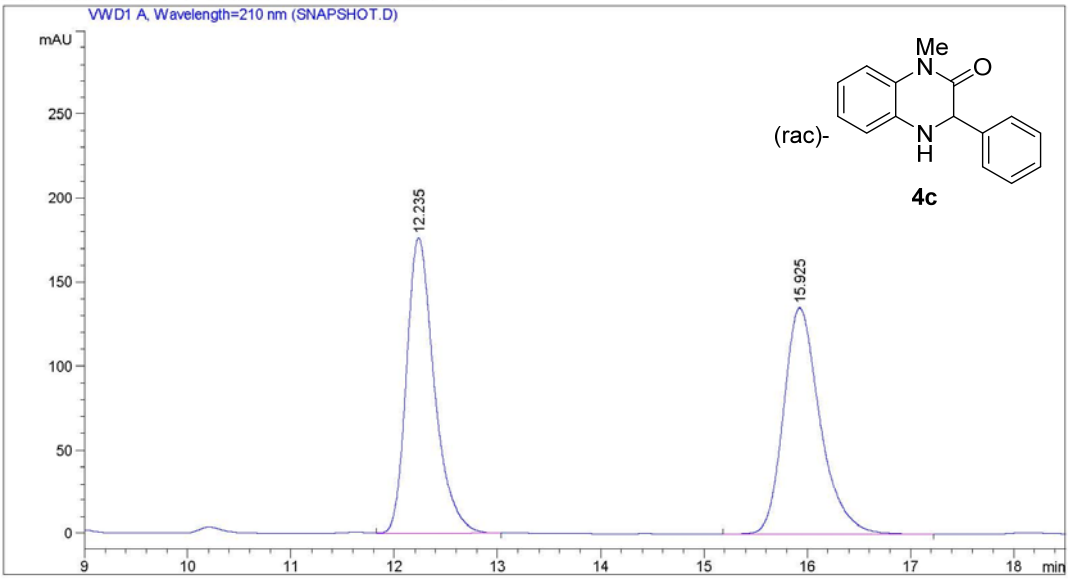
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 71
Injection Date  : 9/26/2017 11:31:23 AM            Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  5UL-220NM-25MIN.M
Last changed    : 9/26/2017 11:16:47 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\SUB-20170926 2017-09-26 11-16-47\VWD-OD(1-2)-80-20-1ML-
                  5UL-220NM-25MIN.M (Sequence Method)
Last changed    : 9/26/2017 2:40:58 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.917 BV    0.2672 3.91830e4  2249.79248  99.3748
   2   9.541 VB    0.3517  246.52602   10.62627   0.6252

Totals :                  3.94295e4  2260.41875
```

Data File C:\CHEM32\1\DATA\SNAPSHOT.D
Sample Name:

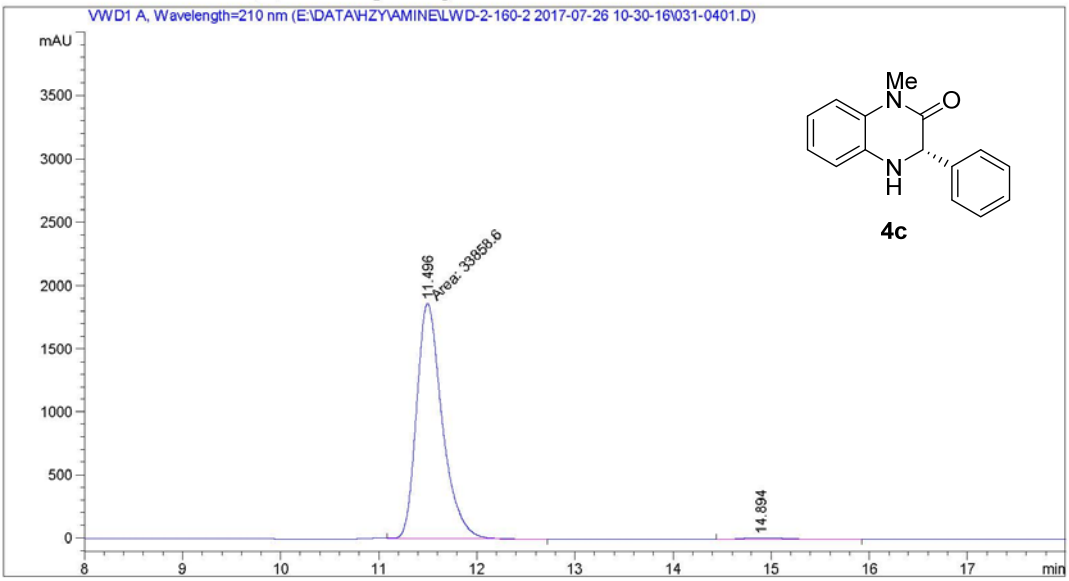
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   5
                                                 Location : Vial 32
Injection Date  : 7/26/2017 11:17:33 AM             Inj :   1
Acq. Method     : VWD-AD(1-2)-80-20-1.0ML-1UL-210NM-25MIN.M
Analysis Method : E:\METHODS\HZY\VWD-AD(1-2)-80-20-1.0ML-1UL-210NM-25MIN.M
Last changed    : 7/26/2017 11:40:17 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.235 VB    0.2831 3296.98560  176.39929  49.7965
   2  15.925 BB    0.3729 3323.93262  135.14771  50.2035

Totals :                  6620.91821  311.54700


==================================================================
                      *** End of Report ***
```

Data File E:\DATA\HZY\AMINE\LWD-2-160-2 2017-07-26 10-30-16\031-0401.D
Sample Name: N-ME

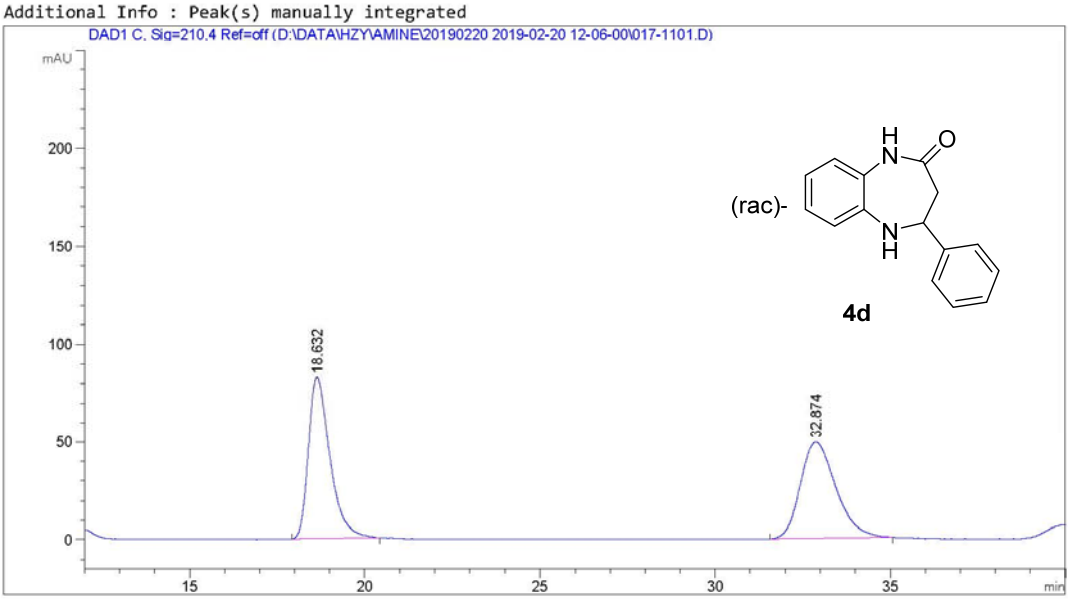
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 31
Injection Date  : 7/26/2017 10:51:50 AM            Inj :   1
                                                Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\AMINE\LWD-2-160-2 2017-07-26 10-30-16\VWD-AD(1-2)-80-20-1.0ML-
                  1UL-210NM-25MIN.M
Last changed    : 7/26/2017 10:31:56 AM by SYSTEM
Analysis Method : E:\DATA\HZY\AMINE\LWD-2-160-2 2017-07-26 10-30-16\VWD-AD(1-2)-80-20-1.0ML-
                  1UL-210NM-25MIN.M (Sequence Method)
Last changed    : 7/26/2017 11:37:48 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.496 MM    0.3023 3.38586e4  1866.80188  99.1713
   2  14.894 BB    0.3561  282.94363   11.95377   0.8287

Totals :                  3.41415e4  1878.75565
```

Data File D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\017-1101.D
Sample Name: S-PdC

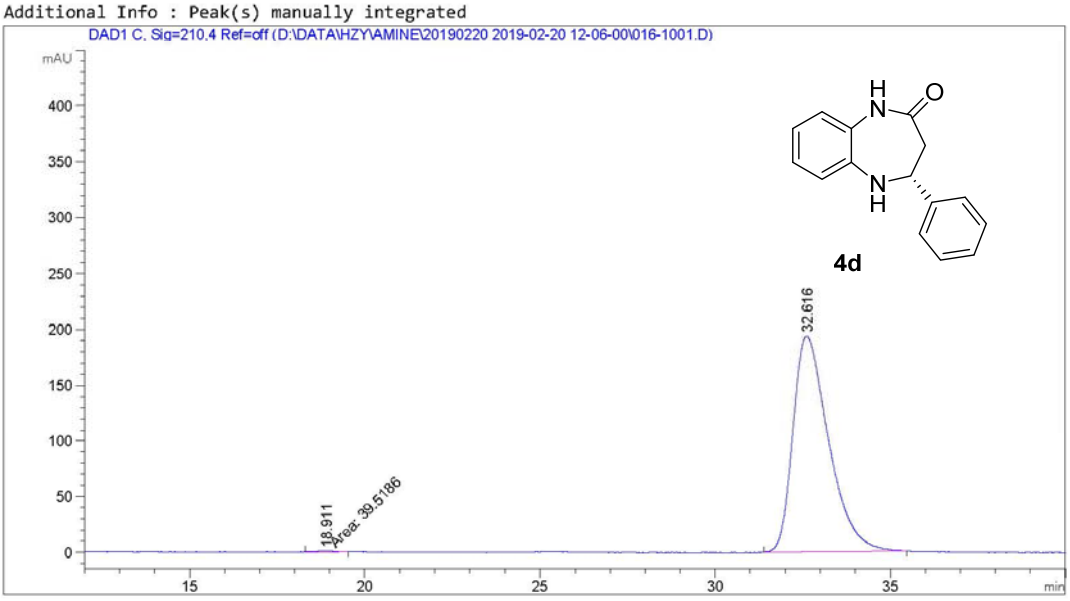
```
==================================================================
Acq. Operator   :                          Seq. Line :  11
Acq. Instrument : Instrument 2              Location : Vial 17
Injection Date  : 2/20/2019 4:17:10 PM           Inj :  1
                                          Inj Volume : 1.000 µl
Acq. Method     : D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\DAD-OJ(1-6)-80-20-1ML-1UL-
                  ALL-50MIN.M
Last changed    : 2/20/2019 12:04:34 PM
Analysis Method : D:\METHOD\HZY\DAD-OJ(1-6)-80-20-1ML-1UL-ALL-50MIN.M
Last changed    : 2/20/2019 5:36:09 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.632 BB    0.6099 3546.97827   83.10571  50.2678
   2  32.874 BB    0.8443 3509.19238   49.66652  49.7322

Totals :                  7056.17065  132.77223
```

Data File D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\016-1001.D
Sample Name: S-EE

```
======================================================================
Acq. Operator   :                              Seq. Line :  10
Acq. Instrument : Instrument 2                  Location : Vial 16
Injection Date  : 2/20/2019 3:26:17 PM               Inj :   1
                                              Inj Volume : 1.000 µl
Acq. Method     : D:\DATA\HZY\AMINE\20190220 2019-02-20 12-06-00\DAD-OJ(1-6)-80-20-1ML-1UL-
                  ALL-50MIN.M
Last changed    : 2/20/2019 12:04:34 PM
Analysis Method : D:\METHOD\HZY\DAD-OJ(1-6)-80-20-1ML-1UL-ALL-50MIN.M
Last changed    : 2/20/2019 5:37:55 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.911 MM    0.6028   39.51863    1.09261   0.2881
   2  32.616 BB    1.0034 1.36798e4   193.83629  99.7119

Totals :                  1.37193e4   194.92890
```