Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Synthesis of 1a,7a-dihydro-7a-phenyl-benzopyrano[2,3-*b*] azirin-7-ones *via* photoisomerization reaction

Qiuya Wang, [a,b] Zunting Zhang,*[a] Xi Zhang,[a] Jin Zhang,[a] Yang Kang [a] and Jufang Peng [a]

[a] *Key Laboratory of the Ministry of Education for Medicinal Resources and Natural Pharmaceutical Chemistry, National Engineering Laboratory for Resource Development of Endangered Crude Drugs in Northwest of China, and School of Chemistry and Materials Science, Shaanxi Normal University, Xi'an 710062, P.R. China.*

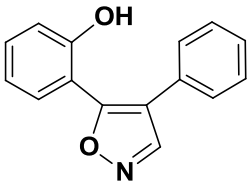
[b] *College of Chemistry and Life Science, WeiNan Normal University, WeiNan 714000, PRC*

E-mail: zhangzunting@sina.com and wqiuya1978@163.com

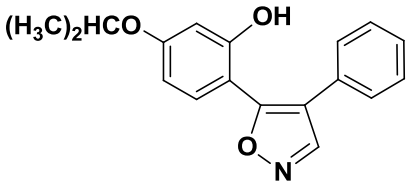
TABLE OF CONTENTS

Part Experimental Details and Data

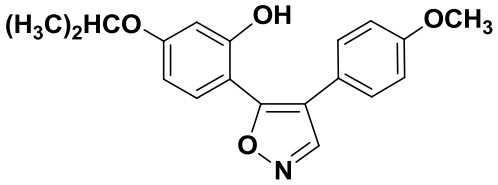
General Procedure for the Preparation of 4-phenyl-5-(2-hydroxyphenyl)isoxazoles (2a-w) The starting materials, 4-phenyl-5-(2-hydroxyphenyl)isoxazoles **2a-w** were synthesized according to the literature.[1] The corresponding isoflavone (1 mmol), hydroxylamine hydrochloride (0.28 g, 4 mmol), and Et_3N (0.56 ml, 4 mmol) were refluxed in EtOH (10 mL) for 4-10 h. The progress of the reactions was monitored by TLC until the disappearance of isoflavone. The reaction mixture was poured into water (50 mL) and acidified with a solution of 10% HCl to the neutrality. The precipitate was filtered off and was purified on silica gel column using petroleum ether-ethyl acetate to give 4-phenyl-5-(2-hydroxyphenyl)isoxazoles **2a-w**.



4-phenyl-5-(2-hydroxyphenyl)isoxazoles(2a): Yield: 81%; White solid; m.p. 170.5~171.7 ℃. ^{1}H NMR (400 MHz, DMSO-d_6), δ(ppm) 6.93~7.01(m, 2H), 7.26~7.40(m, 7H), 9.05(s, 1H), 10,04(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 114.9, 116.3, 117.0, 119.2, 126.7, 127.3, 128.6, 129.8, 130.7, 131.9, 150.6, 155.6, 162.6.

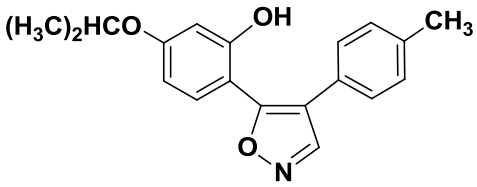


4-phenyl-5-(2-hydroxy1-4-isopropoxyphenyl)isoxazoles(2b): Yield: 88%; White solid; m.p. 157.0~157.8 ℃. ^{1}H NMR(400 MHz, DMSO- d_6), δ(ppm) 1.34(d, 6H, J = 6.0 Hz), 4.63(m, 1H), 6.54 (m, 2H), 7.22~7.44(m, 6H), 9.03(s, 1H), 10.02(s, 1H); ^{13}CNMR(100 MHz, DMSO-d_6), δ(ppm) 21.8, 69.4, 102.9, 106.6, 107.1, 116.4, 126.7, 127.1, 128.5, 130.1, 131.5, 150.6, 156.9,160.4, 162.8.

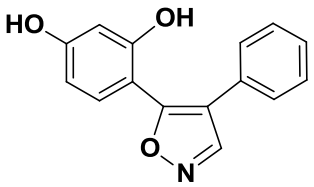


4-(4-methoxyphenyl)-5-(2-hydroxy1-4-isopropoxyphenyl)isoxazoles(2c): Yield: 86%; White solid; m.p. 158.7~159.3 ℃. ^{1}H NMR(400 MHz, DMSO- d_6), δ(ppm) 1.29 (d, 6H, J = 5.6 Hz), 3.73(s, 3H), 4.57(m,

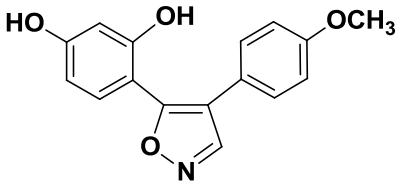
1H), 6.50(s, 2H), 6.90(d, 2H, *J* =8.4 Hz), 7.17(m, 1H), 7.30(d, 2H, *J* =8.4 Hz), 8.92(s, 1H), 9.99 (s, 1H); ^{13}CNMR(100 MHz, DMSO-d_6), δ(ppm) 23.2, 56.4, 70.8, 104.3, 107.8, 108.7, 115.4, 117.6, 123.7, 129.3, 132.9, 151.9, 158.3, 159.8, 161.6, 163.3.



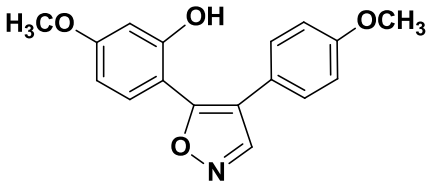
4-(4-methy1phenyl)-5-(2-hydroxy1-4-isopropoxyphenyl)isoxazoles(2d): Yield: 85%; White solid; m.p. 165.2~165.6 ℃. ^{1}H NMR(400 M Hz, DMSO-d_6), δ(ppm) 1.27 (m, 6H), 2.27(s, 3H), 4.58(m, 1H), 6.48(s, 2H), 7.12~7.27(m, 5H), 8.94(s, 1H), 9.98 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6), δ(ppm) 20.7, 21.8, 69.3, 102.9, 106.6, 107.2, 116.3, 126.6, 127.2, 129.1, 131.5, 136.4, 150.6, 156.9, 160.3, 162.4.



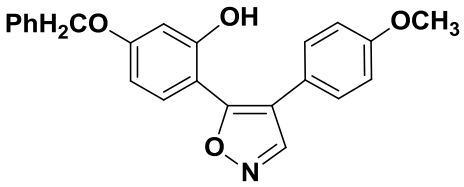
4-phenyl-5-(2,4-dihydroxyphenyl)isoxazoles(2e): Yield: 72%; White solid. m.p. 166.9~167.5 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 6.35(d, 1H, *J* = 8.4 Hz), 6.44(s, 1H), 7.09(d, 1H, *J* =8.4 Hz), 7.23~7.40(m, 5H), 8.97(s,1H), 9.84(s, 2H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 102.9, 105.8, 107.2, 116.1, 126.6, 127.0, 128.5, 130.2, 131.4, 150.5, 156.9, 160.5, 163.2.



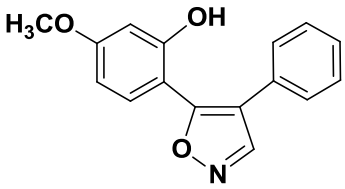
4-(4-methoxyphenyl)-5-(2,4-dihydroxyphenyl)isoxazoles(2f): Yield: 78%; White solid. m.p. 187.5~188.8 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.73(s, 3H), 6.34(d, 1H, *J* = 8.4 Hz), 6.43(s, 1H), 6.89 (d, 2H, *J* = 8.4 Hz), 7.06(d, 1H, *J* = 8.4 Hz),7.30(d, 2H, *J* = 8.4 Hz), 8.89(s, 1H), 9.84(s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6), δ(ppm) 56.4, 104.3, 107.4, 108.5, 115.4, 117.3, 123.9, 129.3, 132.9, 151.8, 158.3, 159.7, 161.8, 163.8.



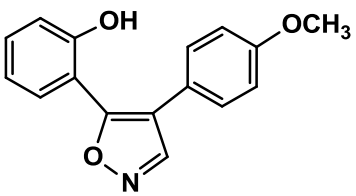
4-(4-methoxyphenyl)-5-(2-hydroxy1-4-methoxyphenyl)isoxazoles(2g): Yield: 90%; White solid; m.p. 190.9~191.5 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.74(s, 3H), 3.77(s, 3H), 6.53(m, 2H), 6.90(d, 2H, J = 8.4 Hz), 7.20(d, 1H, J = 8.4 Hz),7.30(d, 2H, J = 8.4 Hz), 8.92(s, 1H), 10.08(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.0, 55.1, 101.6, 105.4, 107.6, 114.0, 116.2, 122.3, 127.9, 131.6, 150.5, 156.9, 158.4, 161.9, 162.0.



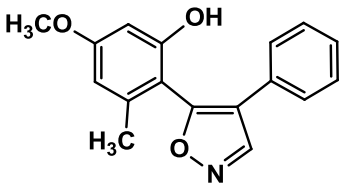
4-(4-methoxyphenyl)-5-(2-hydroxy1-4-benzyloxyphenyl)isoxazoles(2h): Yield: 89%; White solid. m.p. 161.5~ 162.5 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.74(s,3H), 5.11(s, 2H), 6.61(d, 2H, J = 8.8 Hz), 6.90(d, 2H, J = 8.8 Hz), 7.19~7.48(m, 8H), 8.93(s, 1H), 10.05(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.1, 69.3, 102.5, 106.1, 107.9, 114.0, 116.3, 122.3,127.8,127.9, 128.5, 131.6, 136.7, 150.5, 156.9, 158.4, 161.1, 161.8.



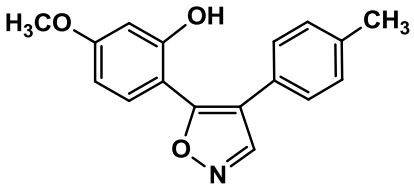
4-phenyl-5-(2-hydroxy1-4-methoxyphenyl)isoxazoles(2i): Yield: 87%; White solid. m.p. 173.9~ 174.8 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.77(s, 3H), 6.55(s, 2H), 7.22~ 7.39(m, 6H),9.00(s, 1H), 10.08(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.1, 101.6, 105.5, 107.5, 116.5, 126.7, 127.1, 128.5, 130.1, 131.6, 150.6, 156.9, 162.1, 162.7.



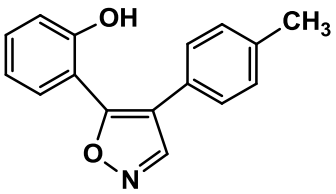
4-(4-methoxyphenyl)-5-(2-hydroxyphenyl)isoxazoles(2j): Yield: 85%; White solid; m.p. 141.3~142.1 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.74(s, 3H), 6.89~7.01(m, 4H), 7.28~7.40(m, 4H), 8.98(s, 1H), 10.06(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.1, 114.1, 115.0, 116.3, 116.7, 119.3, 122.1, 128.0, 130.8, 131.9, 155.7, 158.5, 161.8.



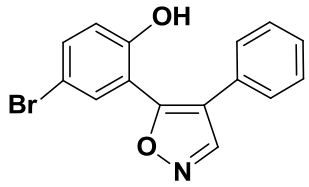
4-phenyl-5-(2-hydroxy1-4-methoxy-6-methylphenyl)isoxazoles(2k): Yield: 87%; White solid; m.p. 182.7~183.5 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 1.90(s, 3H), 3.76(s, 3H), 6.40~6.44 (m, 2H), 7.24~7.38(m, 5H), 9.11(s, 1H), 9.91(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 19.3, 55.0, 99.0, 106.8, 107.3, 117.4, 125.9, 127.2, 128.8, 129.8, 139.9, 150.1, 157.5, 161.6, 162.4.



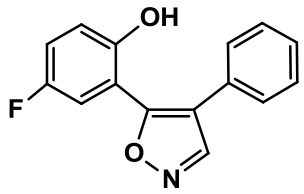
4-(4-methylphenyl)-5-(2-hydroxy1-4-methoxyphenyl)isoxazoles(2l): Yield: 84%; White solid; m.p. 161.0~161.5 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 2.27 (s, 3H), 3.78 (s, 3H), 6.52 (m, 2H), 7.12~7.27 (m, 5H), 8.95 (s, 1H), 10.05 (s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 20.7, 55.1, 101.5, 105.4, 107.6, 116.4, 126.6, 127.2, 129.1, 131.5, 136.4, 150.6, 156.9, 162.0, 162.4.



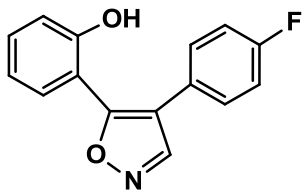
4-(4-methylphenyl)-5-(2-hydroxyphenyl)isoxazoles(2m): Yield: 82%; White solid; m.p. 113.2~114.3 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 2.27(s, 3H), 6.90~7.39(m, 8H), 8.99(s, 1H), 10.01(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 20.7, 115.0, 116.3, 116.9, 119.2, 126.6, 126.9, 129.1, 130.7, 131.8, 136.6, 150.6, 155.6, 162.2.



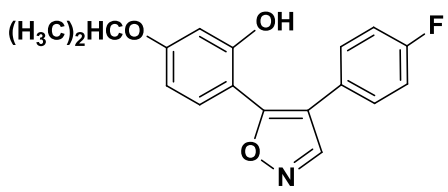
4-phenyl-5-(2-hydroxyl-5-bromophenyl)isoxazoles(2n): Yield:76%; White solid; m.p. 181.9~182.7 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 6.95(d, 1H, J = 8.8 Hz), 7.29~ 7.36(m, 5H), 7.49~ 7.55(m, 2H), 9.05(s, 1H), 10.42(s, 1H). ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 111.3, 118.4, 119.0, 119.9, 128.2, 128.9, 130.0, 130.9, 134.1, 135.9, 152.1, 156.5, 162.2.



4-phenyl-5-(2-hydroxyl-5-fluorophenyl)isoxazoles(2o): Yield: 73%; White solid; m.p. 133.9~134.5 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 6.98(m, 1H), 7.20~7.38(m, 7H), 9.07(s, 1H), 10.04 (s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 115.4(d, ^{3}J = 8.0 Hz), 116.6 (d, ^{2}J = 24.0 Hz), 117.4(d, ^{3}J = 8.0 Hz), 117.5, 118.5(d, ^{2}J = 23.6 Hz), 126.8, 127.4, 128.6, 129.6, 150.7, 152.1, 155.5(d, ^{1}J = 237.0 Hz), 161.1.

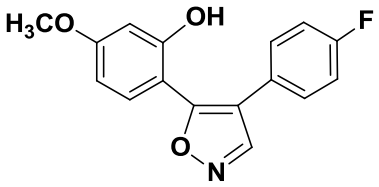


4-(4-fluorophenyl)-5-(2-hydroxyphenyl)isoxazoles(2p): Yield: 78%; White solid; m.p. 130.7~131.9 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 6.94~6.99(m, 2H), 7.01~7.21(m, 2H), 7.32~7.42(m, 4H), 9.03(s, 1H), 10.11(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 114.6, 115.5(d, ^{2}J = 21.8 Hz), 116.1, 116.3, 119.3, 126.3, 128.7(d, ^{3}J = 8.0 Hz), 130.7, 132.0, 150.6, 155.5, 161.3(d, ^{1}J = 243.0 Hz), 162.5.

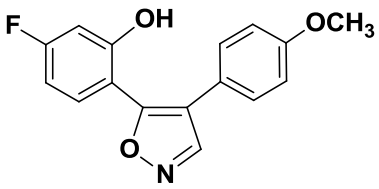


4-(4-fluorophenyl)-5-(2-hydroxyl-4-isopropoxyphenyl)isoxazoles(2q): Yield:83%; White solid; m.p. 151.2~151.6 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 1.29(m, 6H), 4.58(s,1H), 6.50(d, 2H, J = 8.8 Hz),

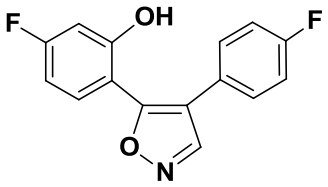
7.17~7.22(m, 3H), 7.39~7.43(m, 2H), 8.98(s, 1H), 10.02(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 21.8, 69.4, 102.9, 106.7, 106.9, 115.4(d, ^{2}J = 21.0 Hz), 126.6, 126.7, 128.7(d, ^{3}J = 8.0 Hz), 131.5, 150.6, 156.8, 160.4,161.2(d, ^{1}J = 243.0 Hz), 162.6.



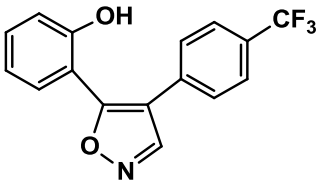
4-(4-fluorophenyl)-5-(2-hydroxyl-4-methoxyphenyl)isoxazoles(2r): Yield: 84%; White solid; m.p. 153.8~154.9 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.78(s, 3H), 6.53(d, 2H, J =8.4 Hz), 7.17~7.26(m, 3H), 7.39~7.42(m, 2H), 8.99(s, 1H), 10.09(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.1, 101.6, 105.6, 107.3, 115.4(d, ^{2}J = 22.4 Hz), 115.6, 126.6, 128.6(d, ^{3}J = 8.0 Hz), 131.5, 150.6, 156.8, 161.2(d, ^{1}J = 243.0 Hz), 162.1, 162.6.



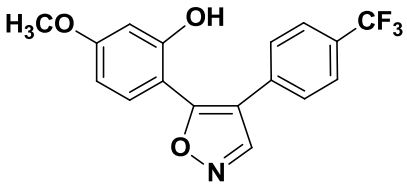
4-(4-methoxyphenyl)-5-(2-hydroxyl-5-fluorophenyl)isoxazoles(2s): Yield: 81%; White solid. m.p. 147.5~148.7 ℃. ^{1}H NMR(400 MHz, DMSO-d_6), δ(ppm) 3.74(s, 3H), 6.91~6.99(m, 3H), 7.17~7.31(m, 4H), 9.00(s, 1H), 10.00(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.1, 114.1, 115.6(d, ^{3}J = 8.0 Hz), 116.6(d, ^{2}J = 24.2 Hz), 117.2, 117.4(d, ^{3}J = 8.0 Hz), 118.4(d, ^{2}J = 23.4 Hz), 121.8, 128.1, 150.7, 152.1, 154.8(d, ^{1}J = 234.0 Hz), 158.6, 160.2.



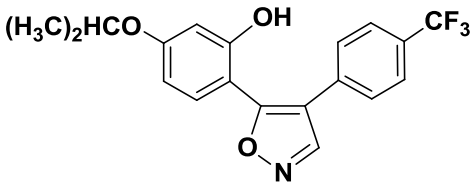
4-(4-fluorophenyl)-5-(2-hydroxyl-5-fluorophenyl)isoxazoles(2t): Yield: 74%; White solid. m.p. 126.6~127.9 ℃. ^{1}H NMR(600 MHz, DMSO- d_6), δ(ppm) 6.94~6.96(m, 1H), 7.17~ 7.24(m, 4H), 7.37~7.40(m, 2H), 9.04(s, 1H), 10.05(s, 1H); ^{13}C NMR(150 MHz, DMSO-d_6), δ(ppm) 115.7(d, ^{3}J = 8.0 Hz), 116.0(d, ^{2}J = 23.6 Hz), 116.1, 117.0, 117.1(d, ^{3}J = 5.4 Hz), 118.0(d, ^{2}J = 23.2 Hz) , 119.1(d, ^{2}J = 23.4 Hz), 126.6, 129.3(d, ^{3}J = 8.0 Hz), 151.9(d, ^{1}J = 180.0 Hz), 155.5(d, ^{1}J = 234.0 Hz), 161.1, 161.5, 162.8.



4-(4-trifluoromethylphenyl)-5-(2-hydroxyphenyl) isoxazoles(2u): Yield: 72%; White solid. m.p. 152.1~ 152.9 ℃. ^{1}H NMR(400 MHz, DM SO- d_6), δ(ppm) 6.94~7.00(m, 2H), 7.38~ 7.43(m, 2H), 7.57(d, 2H, J = 8.0 Hz), 7.70(d, 2H, J = 8.0 Hz), 9.13(s, 1H), 10.14(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 114.3, 115.9, 116.4, 119.4, 124.1(q, ^{1}J=270.4 Hz), 125.4(q, ^{3}J = 3.6 Hz), 127.2(q, ^{2}J = 32.0 Hz), 130.6, 132.2, 134.3, 150.7, 155.3, 163.2.



4-(4-trifluoromethylphenyl)-5-(2-hydroxy-4-methoxyphenyl) isoxazoles(2v): Yield:80%; White solid. m.p. 143.2~ 143.7 ℃. ^{1}H NMR (400 MHz, DMSO-d_6), δ(ppm) 3.81(s, 3H), 6.56~6.62(m, 2H), 7.35(d, 1H, J = 8.8 Hz), 7.61(d, 2H, J = 8.0 Hz), 7.74(d, 2H, J = 8.0 Hz), 9.11(s, 1H), 10.23(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 55.1, 101.6, 105.7, 107.0, 115.2, 124.2(q, ^{1}J = 270.2 Hz), 125.4(q, ^{3}J =3.6 Hz), 127.4(q, ^{2}J = 26.2 Hz), 131.5, 134.6, 150.7, 156.6, 162.3, 163.6.

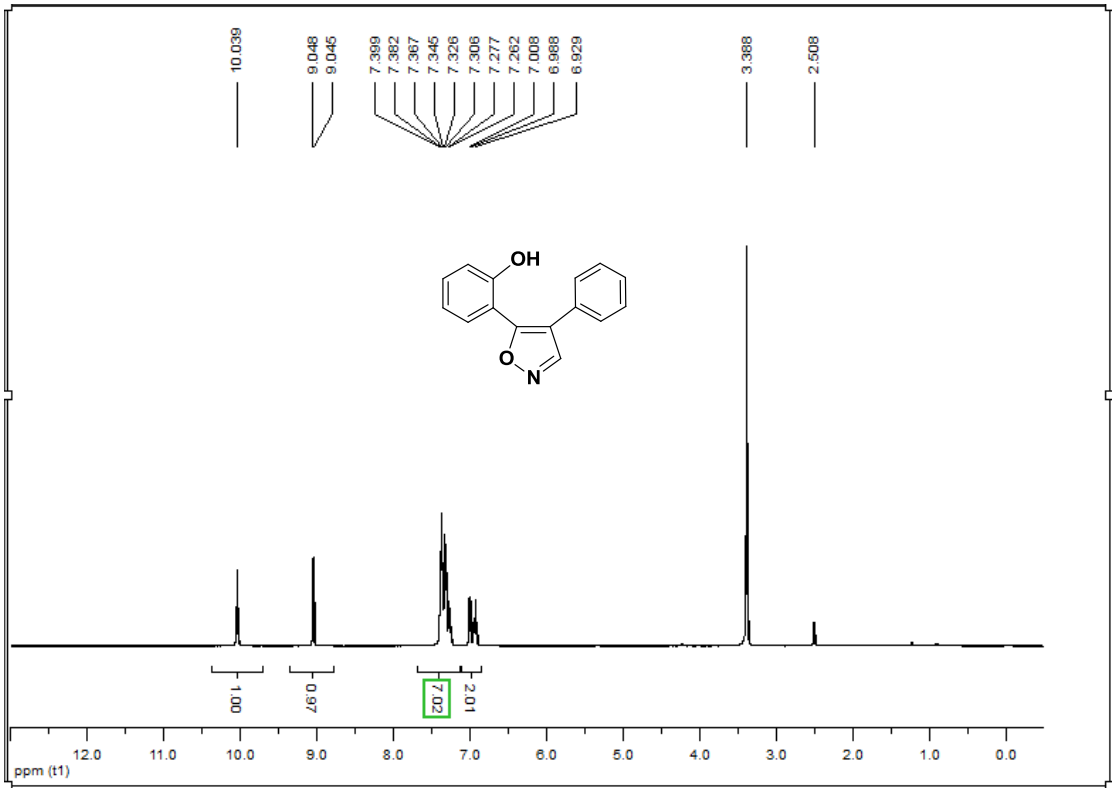


4-(4-trifluoromethylphenyl)-5-(2-hydroxy-4-isopropoxyphenyl) isoxazoles(2w): Yield: 78%; White solid. m.p. 181.3~ 182.5 ℃. ^{1}H NMR (400 MHz, DMSO-d_6), δ(ppm) 1.29(d, 6H, J = 5.6 Hz), 4.59(m, 1H), 6.49~6.55(m, 2H), 7.28 (d, 1H, J = 8.4 Hz), 7.58(d, 2H, J = 8.0 Hz), 7.70(d, 2H, J = 8.0 Hz), 9.07(s, 1H), 10.07(s, 1H); ^{13}C NMR(100 MHz, DMSO-d_6), δ(ppm) 23.1, 70.8, 104.3, 108.0, 108.2, 116.6, 125.6(q,^{1}J = 270.4 Hz), 126.7(q, ^{3}J = 3.7 Hz), 128.7(q, ^{2}J = 32.6 Hz), 132.8, 136.1, 152.1, 158.1, 162.0, 165.1.

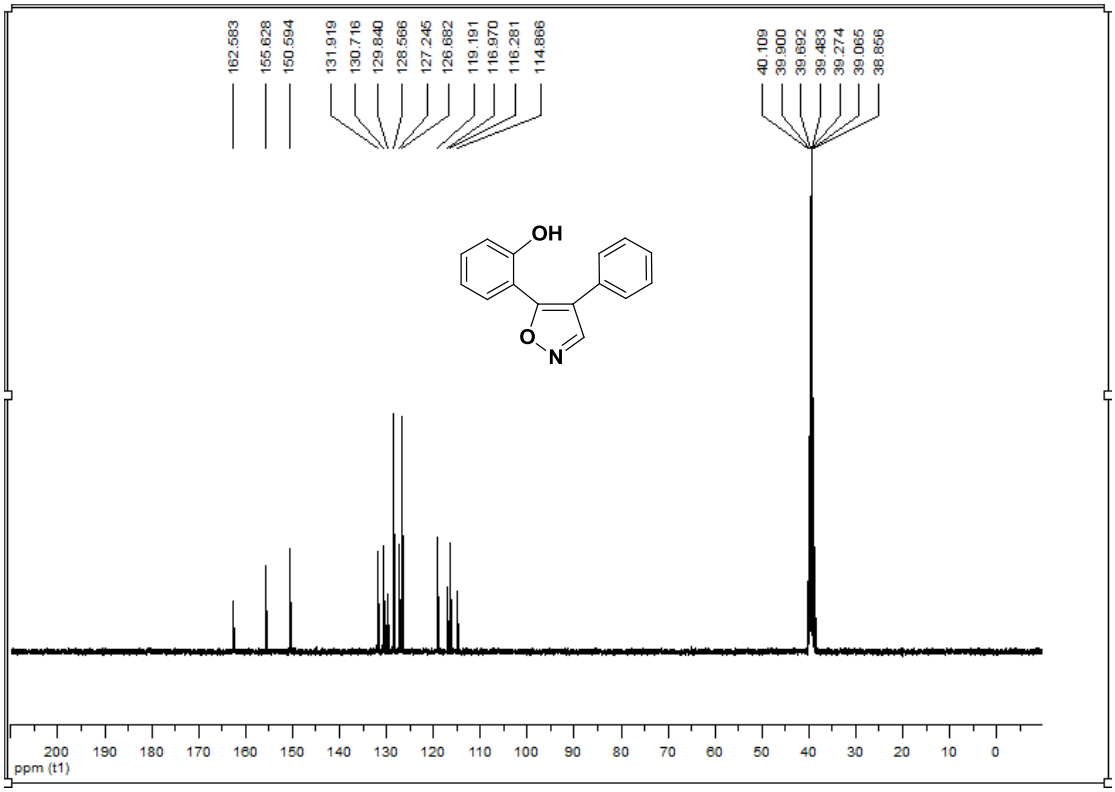
Reference

[1] Y. M. Wu, K. Foleky, C. Borella, *Patent*, WO2008033449.

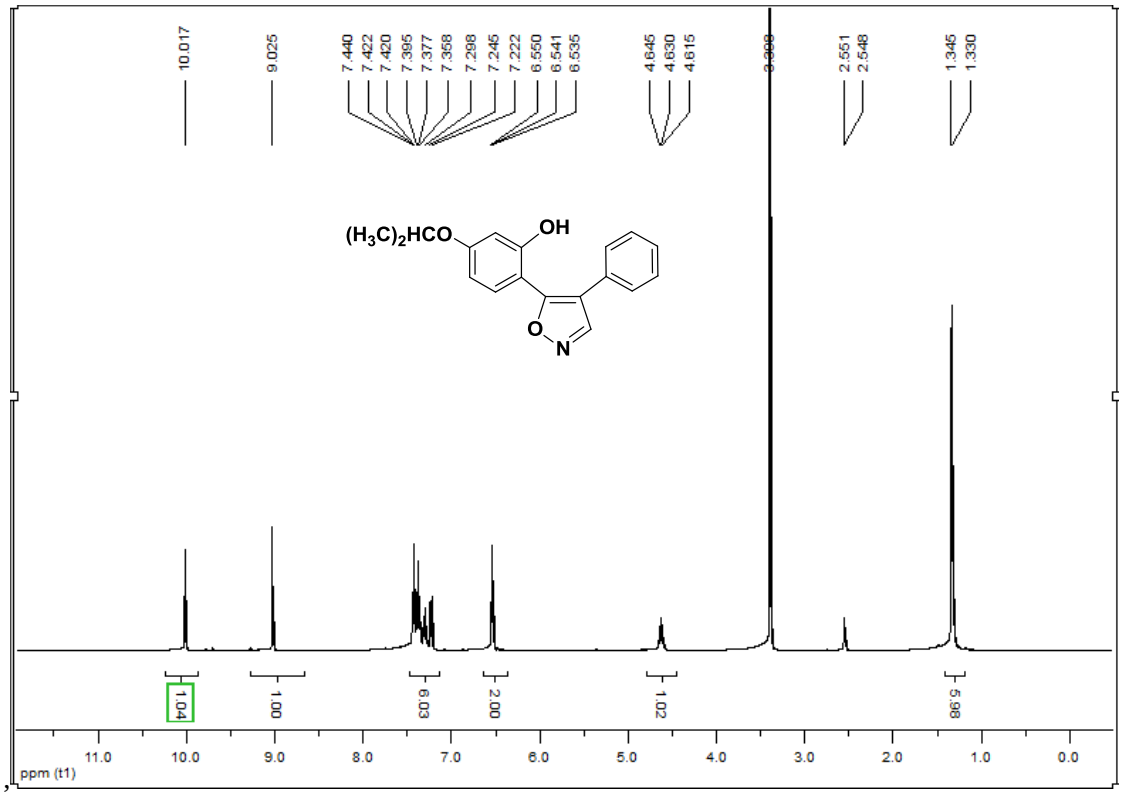
^{1}H NMR and ^{13}C NMR spectra for 2a-w.



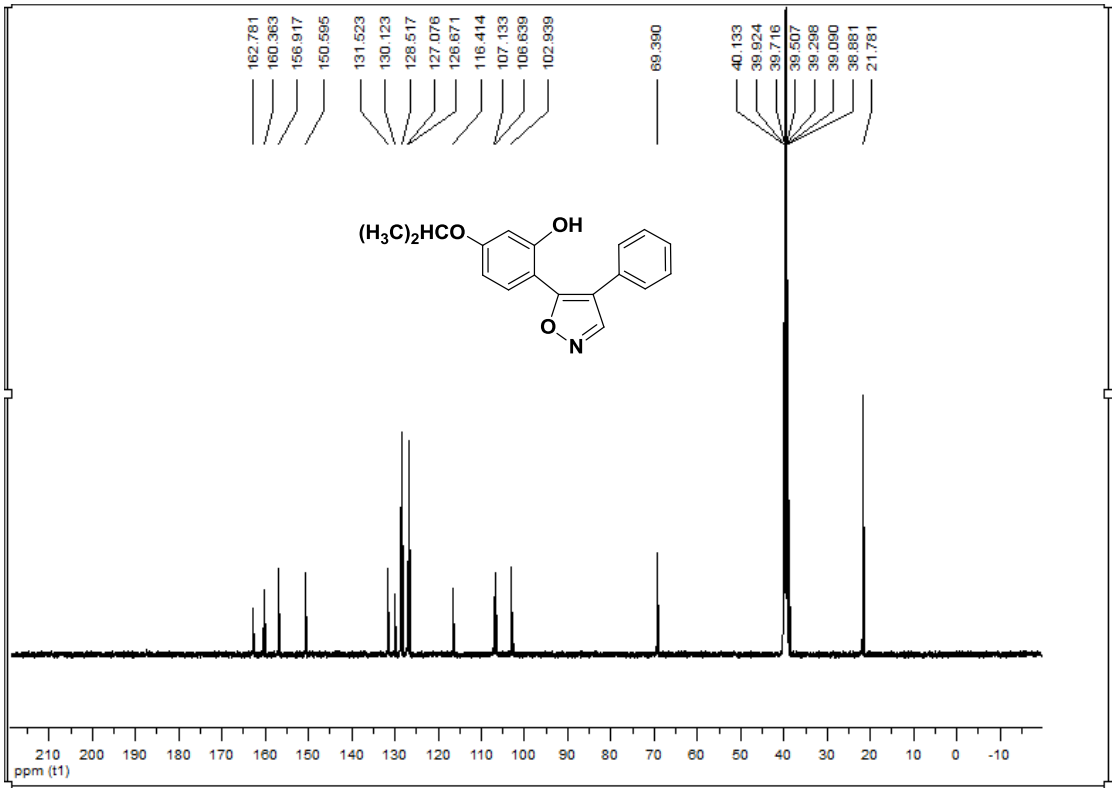
Compound 2a ^{1}H NMR(DMSO-d_6)



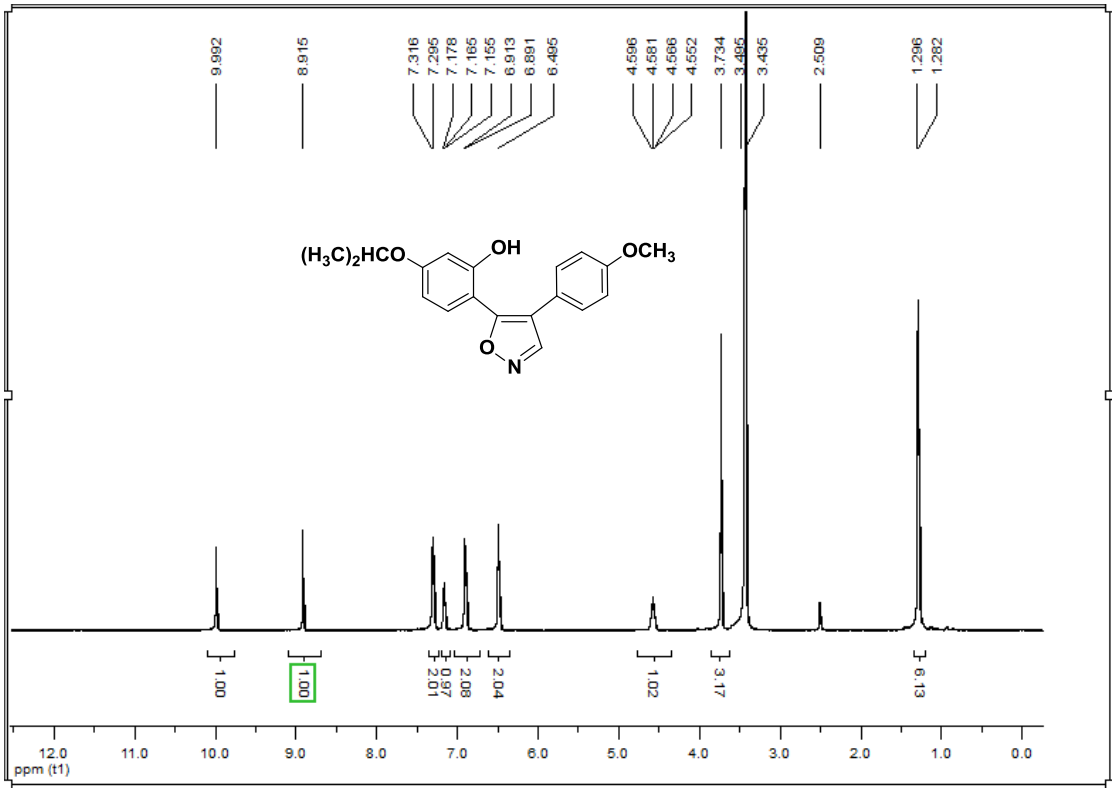
Compound 2a ^{13}C NMR(DMSO-d_6)



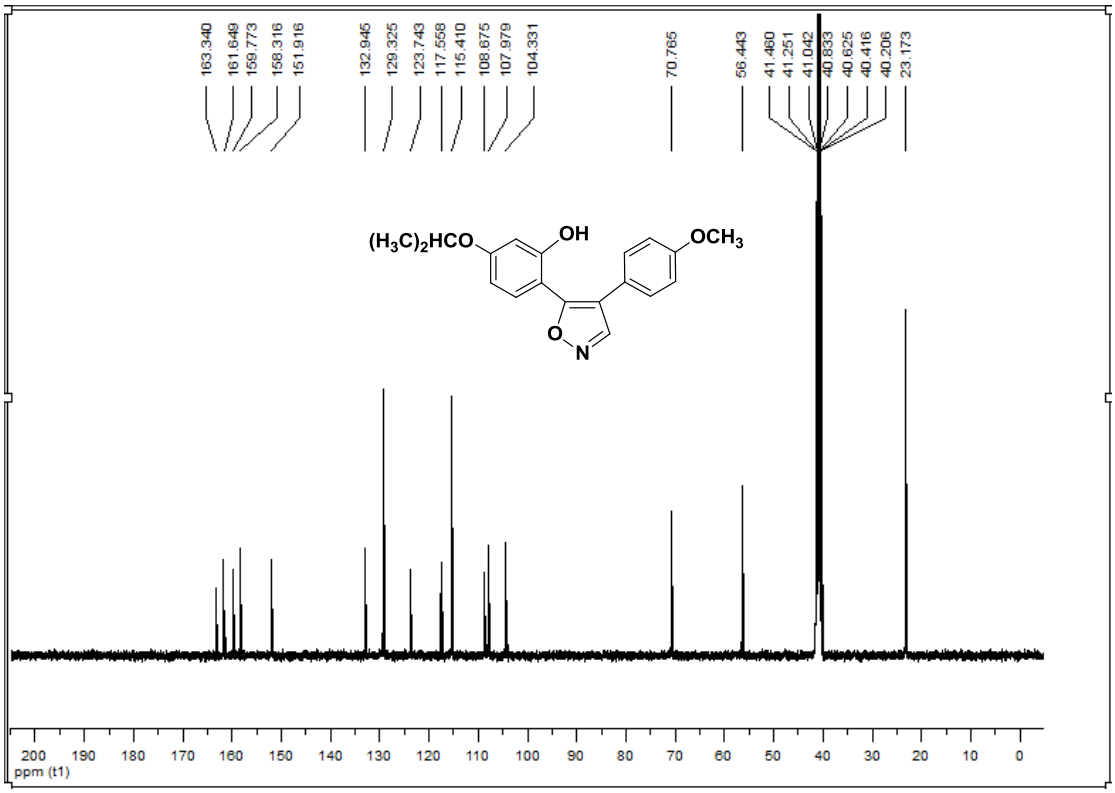
Compound 2b ^{1}H NMR(DMSO-d_6)



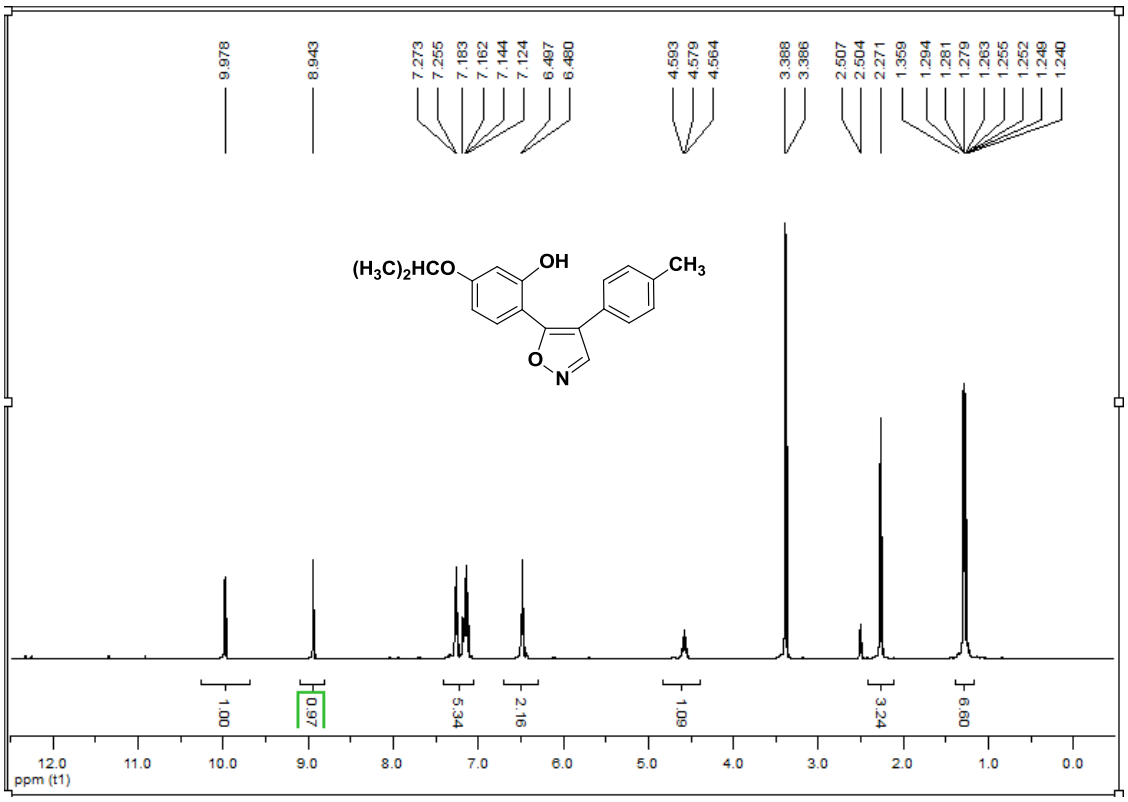
Compound 2b ^{13}C NMR(DMSO-d_6)



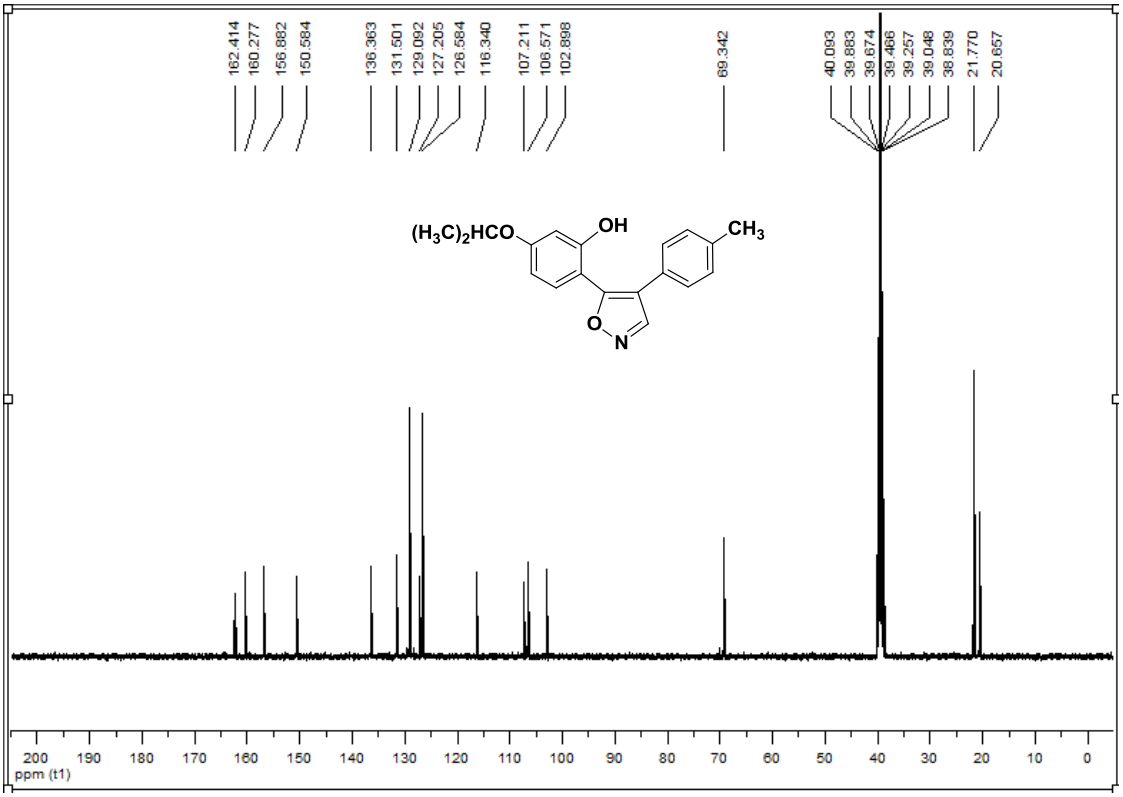
Compound 2c ^{1}H NMR(DMSO-d_6)



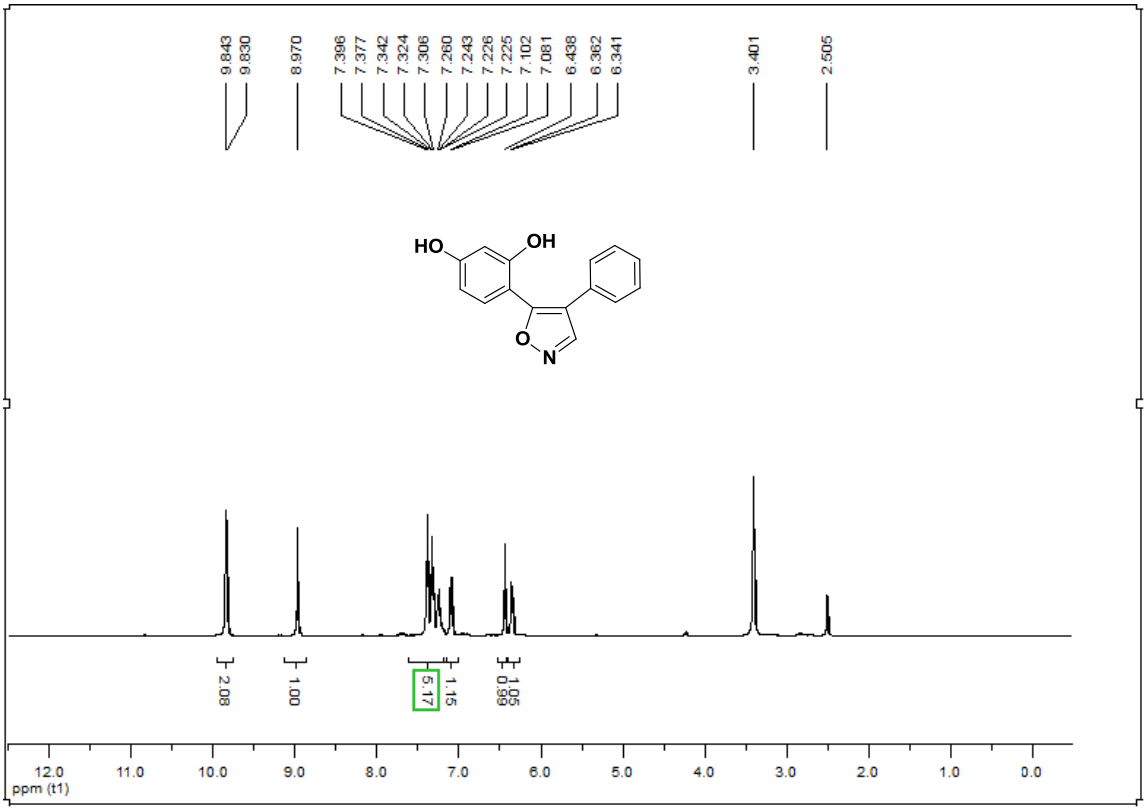
Compound 2c ^{13}C NMR(DMSO-d_6)



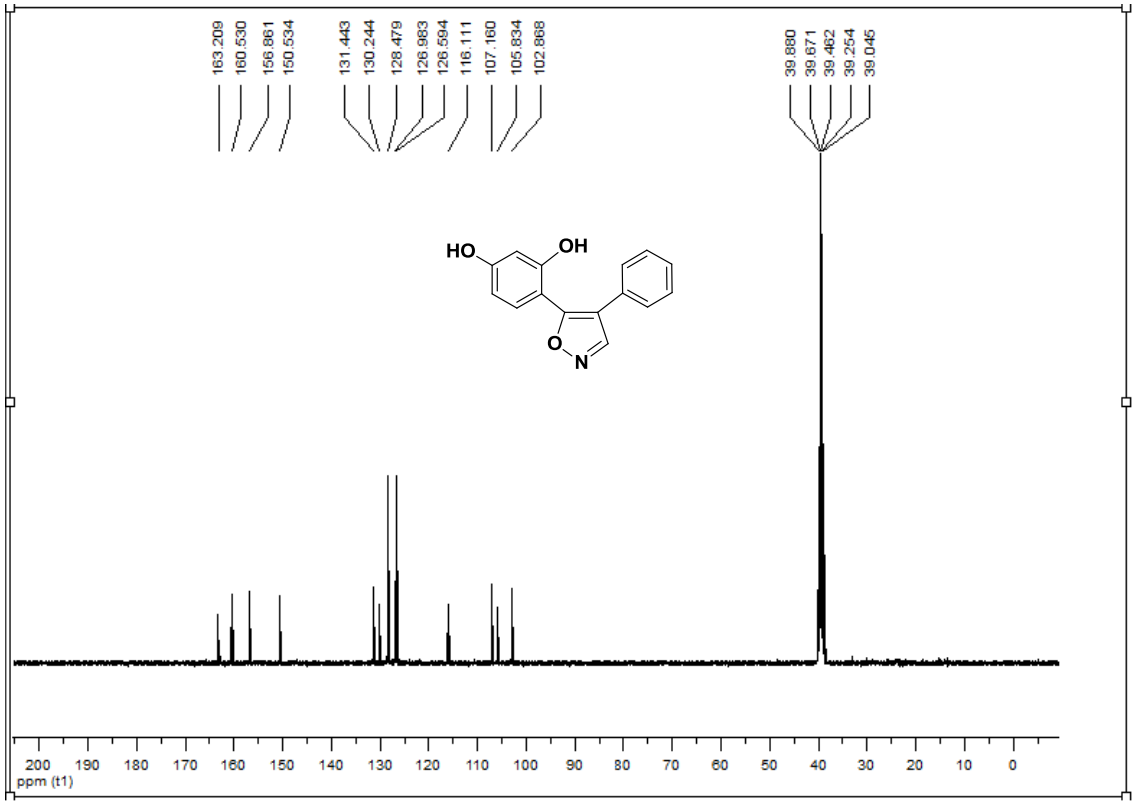
Compound 2d ^{1}H NMR(DMSO-d_6)



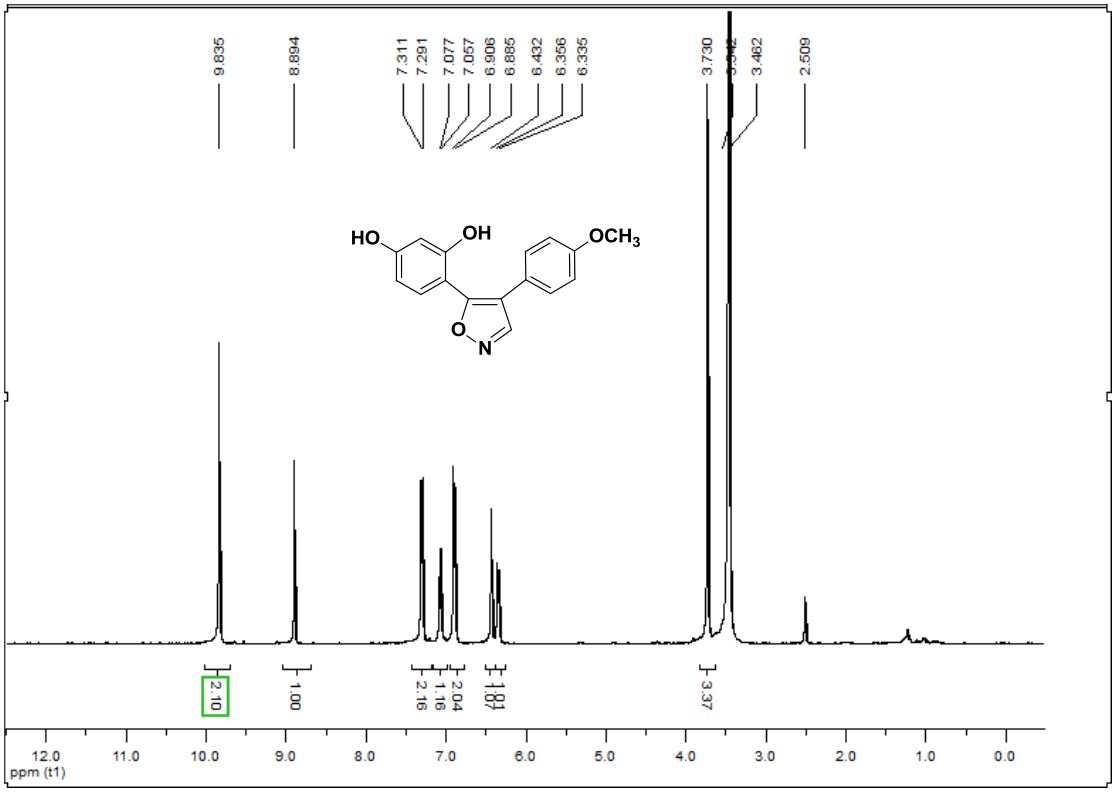
Compound 2d ^{13}C NMR(DMSO-d_6)



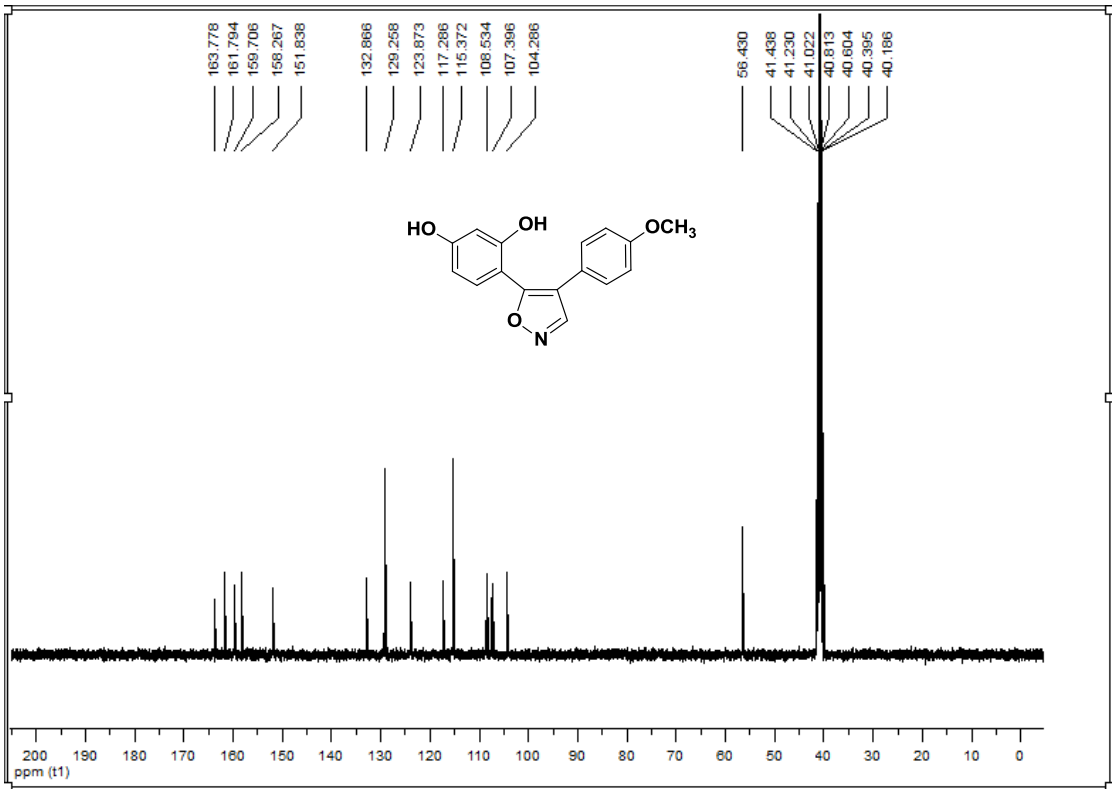
Compound 2e ^{1}H NMR(DMSO-d_6)



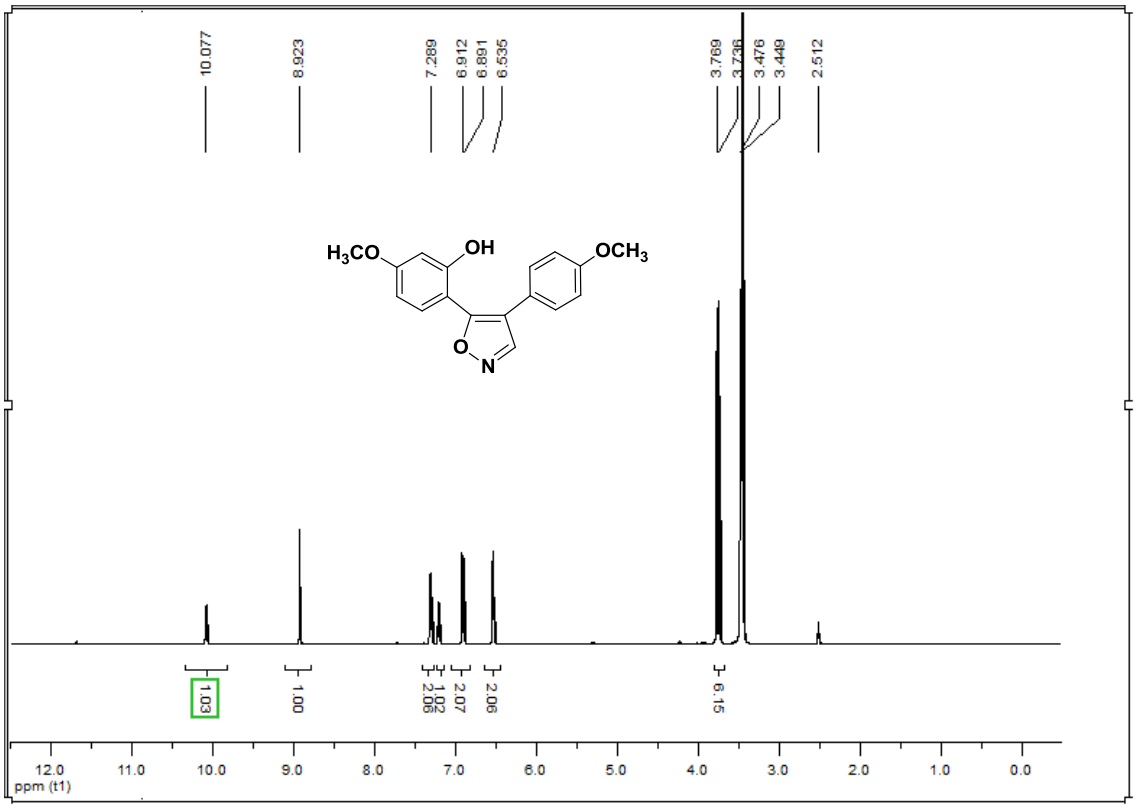
Compound 2e ^{13}C NMR(DMSO-d_6)



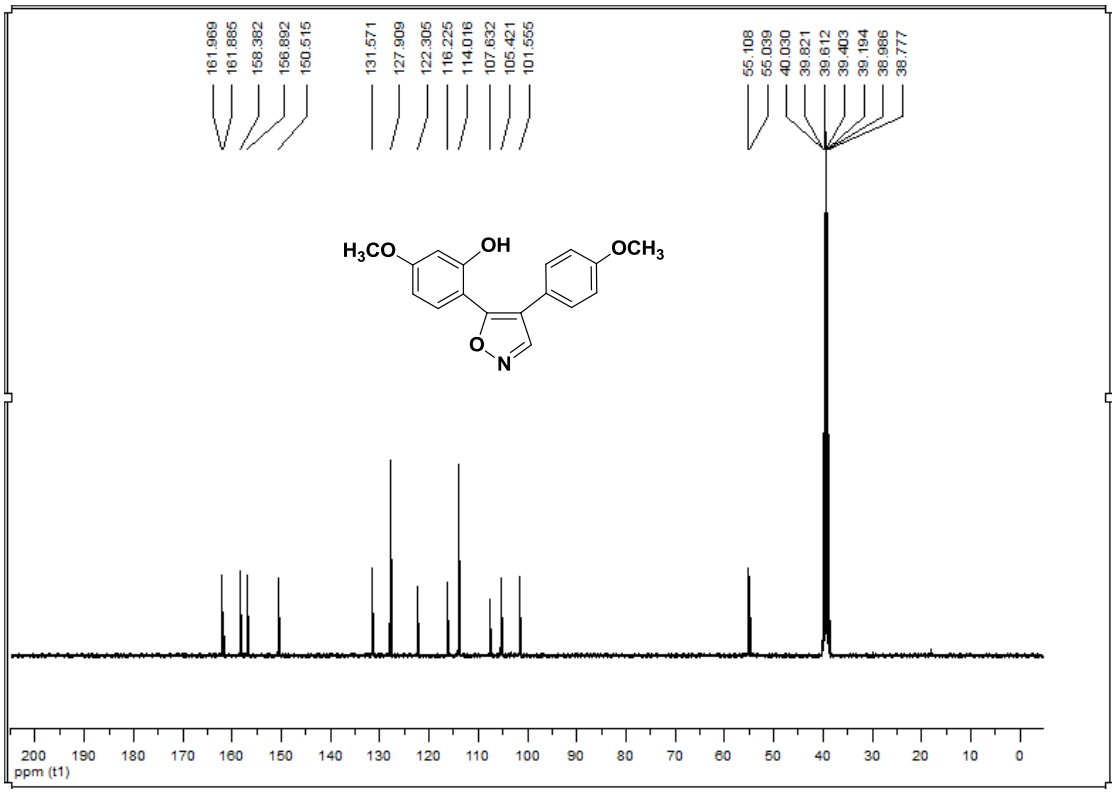
Compound 2f ^{1}H NMR(DMSO-d_6)



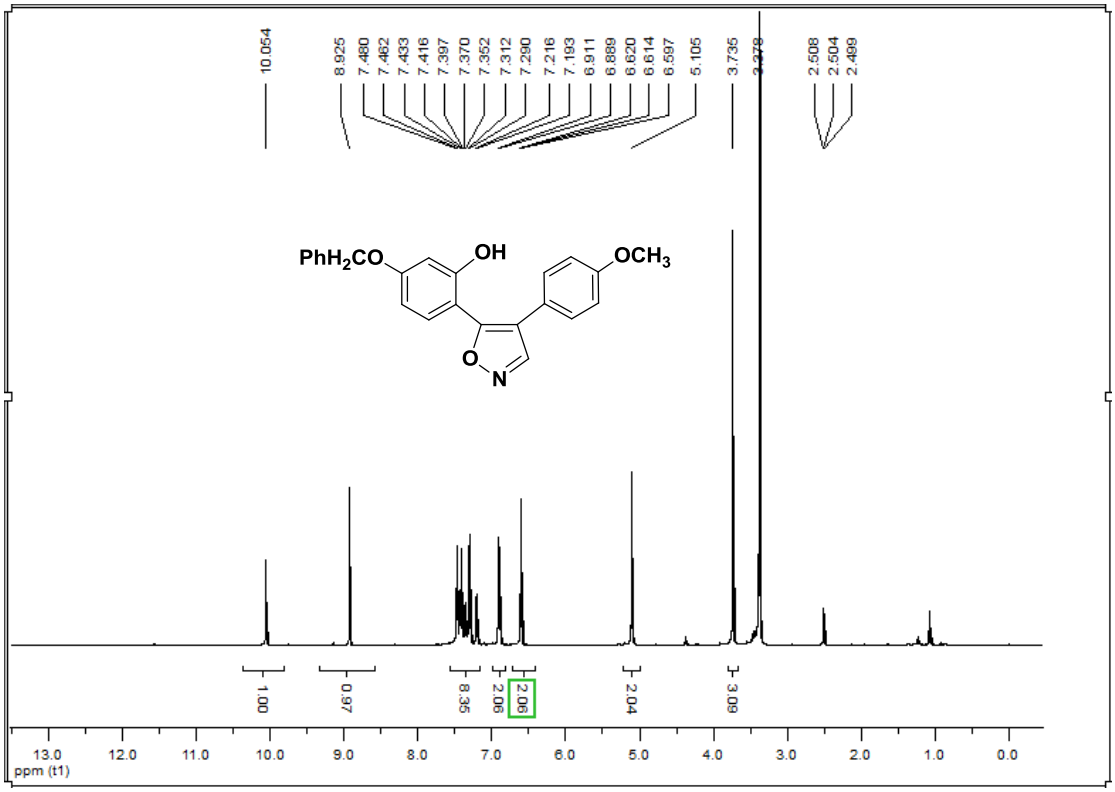
Compound 2f ^{13}C NMR(DMSO-d_6)



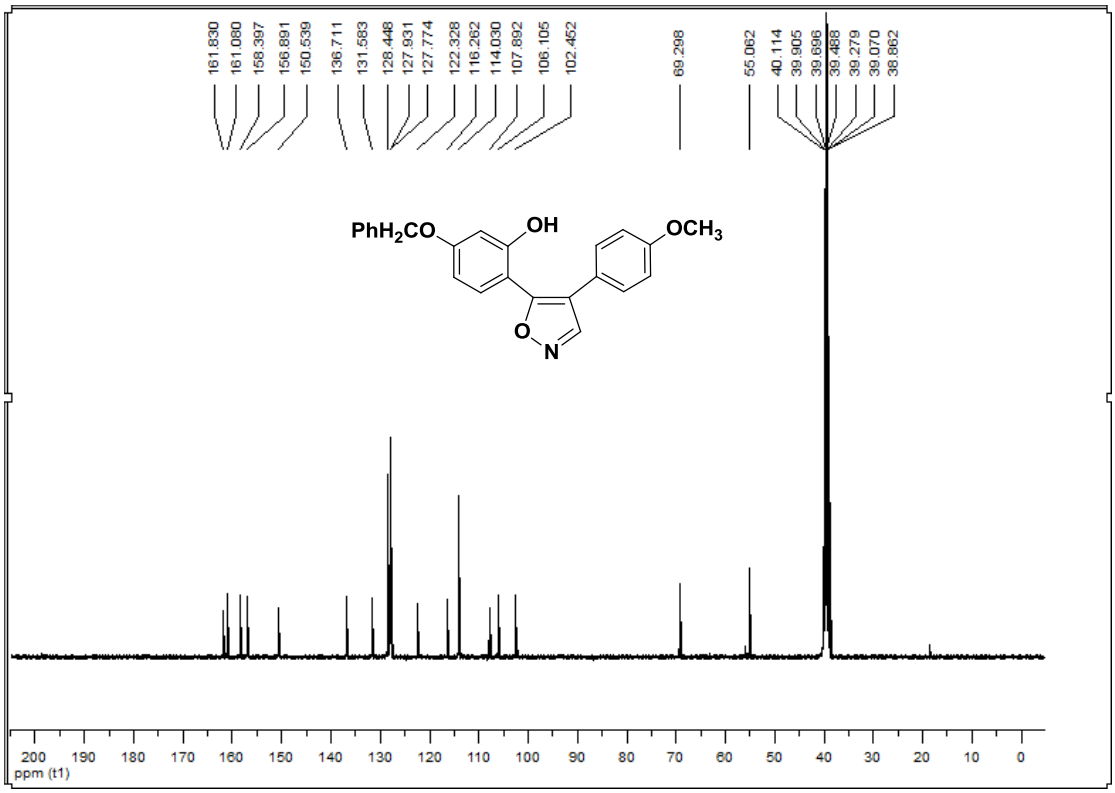
Compound 2g ^{1}H NMR(DMSO-d_6)



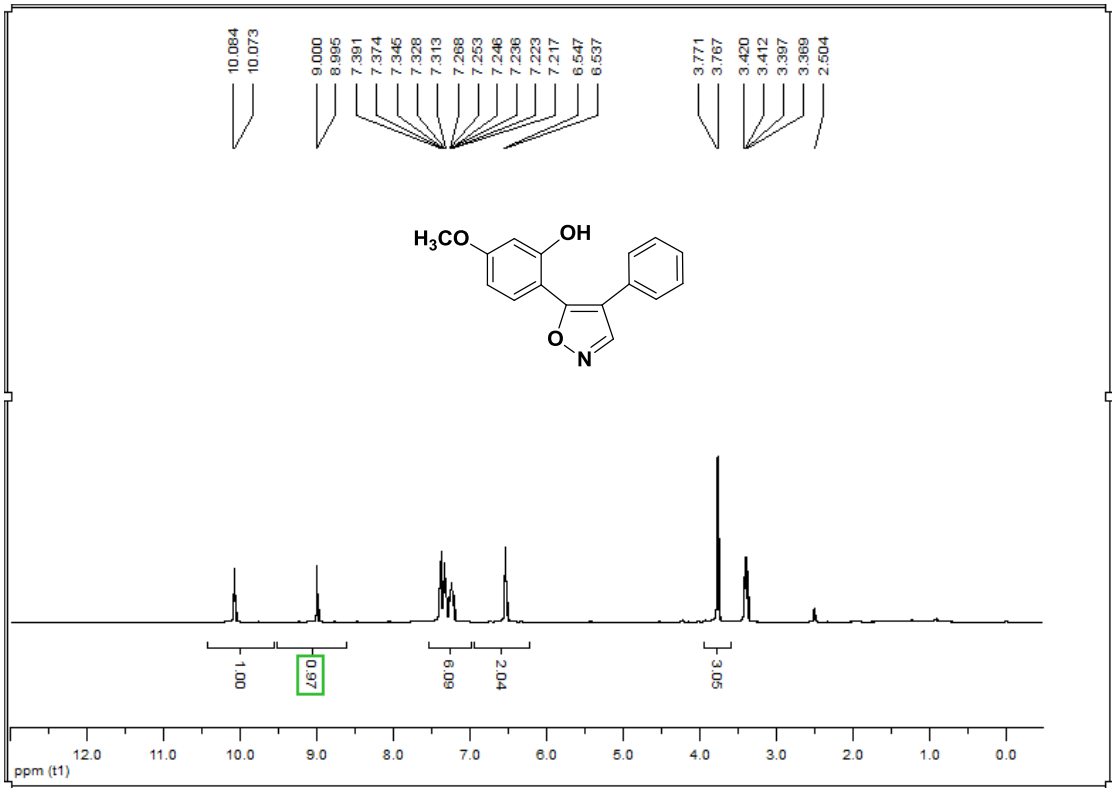
Compound 2g ^{13}C NMR(DMSO-d_6)



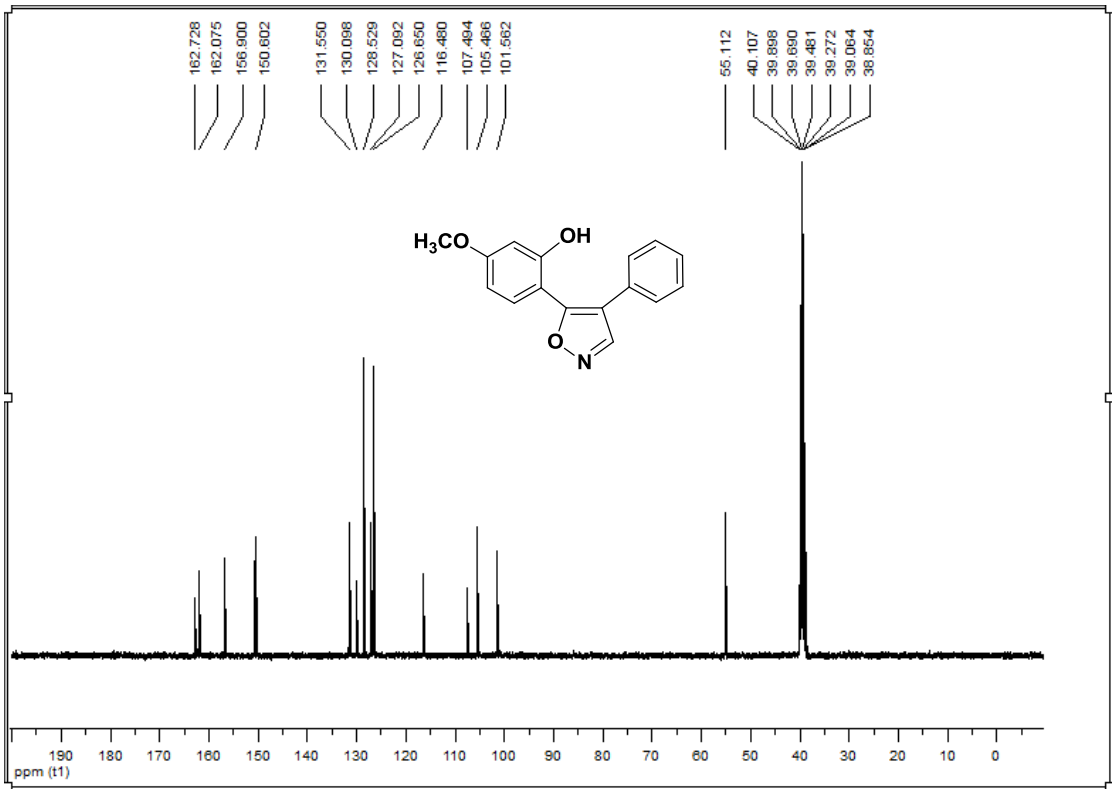
Compound 2h ^{1}H NMR(DMSO-d_6)



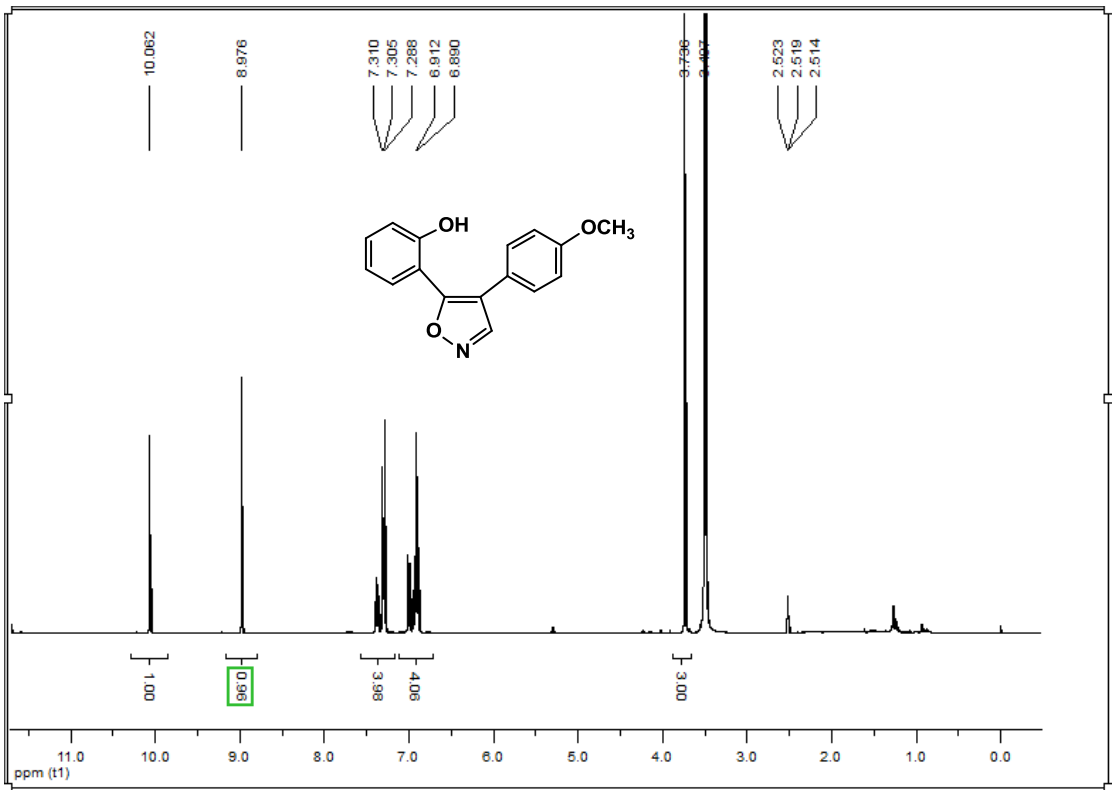
Compound 2h ^{13}C NMR(DMSO-d_6)



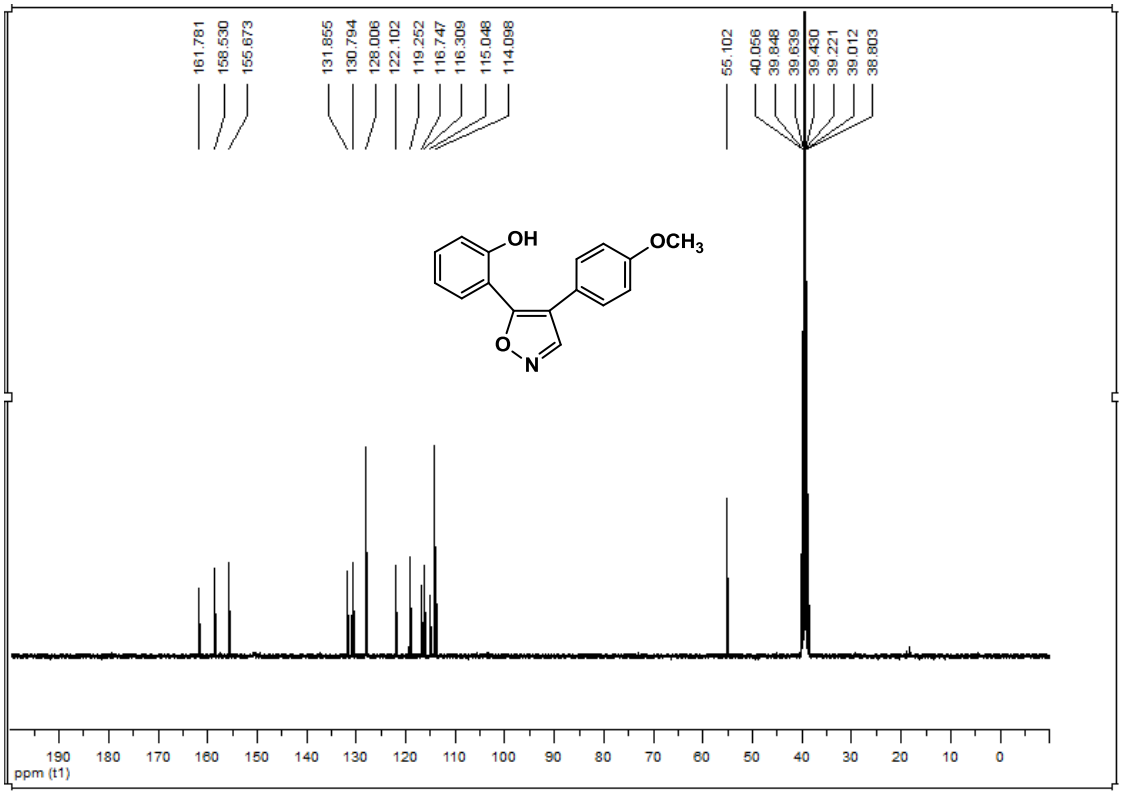
Compound 2i ^{1}H NMR(DMSO-d_6)



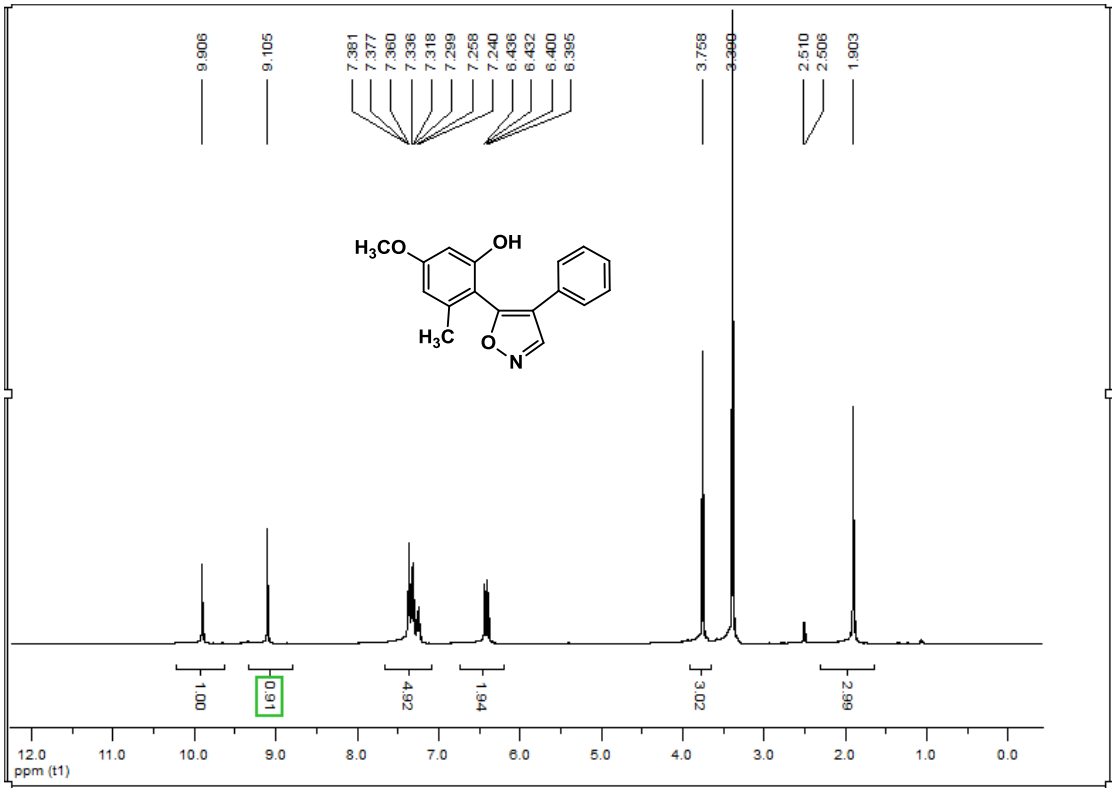
Compound 2i ^{13}C NMR(DMSO-d_6)



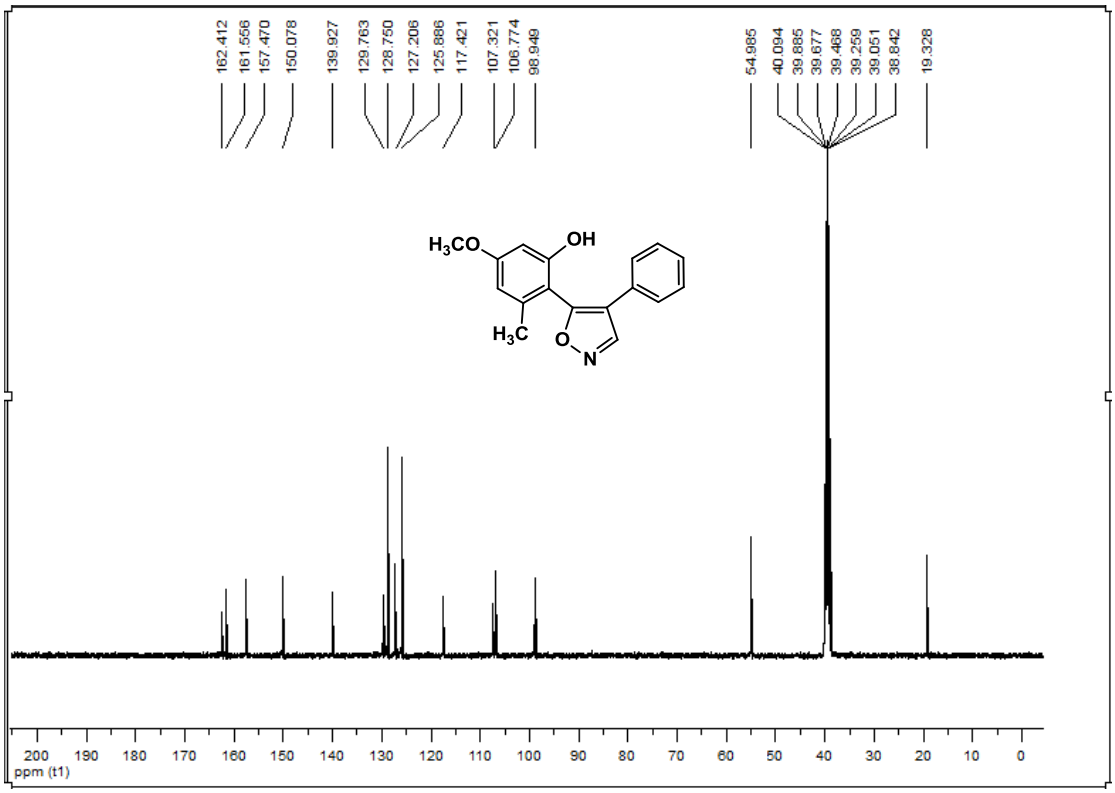
Compound 2j ^{1}H NMR(DMSO-d_6)



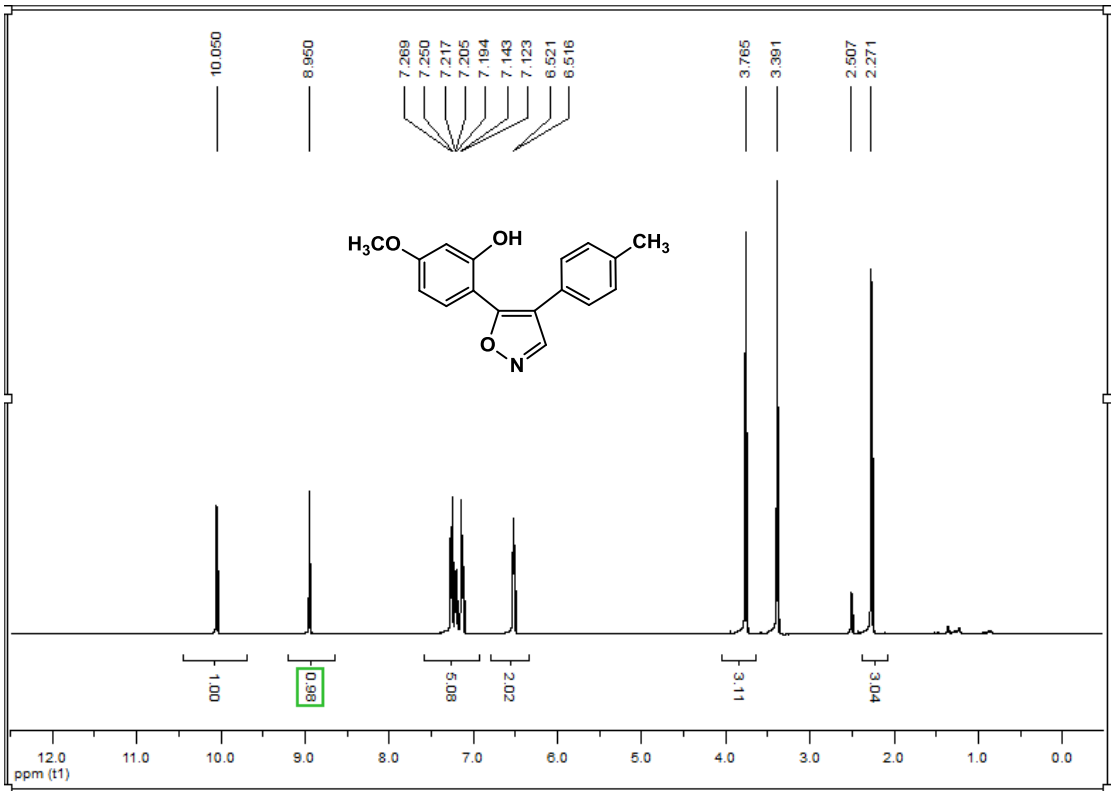
Compound 2j ^{13}C NMR(DMSO-d_6)



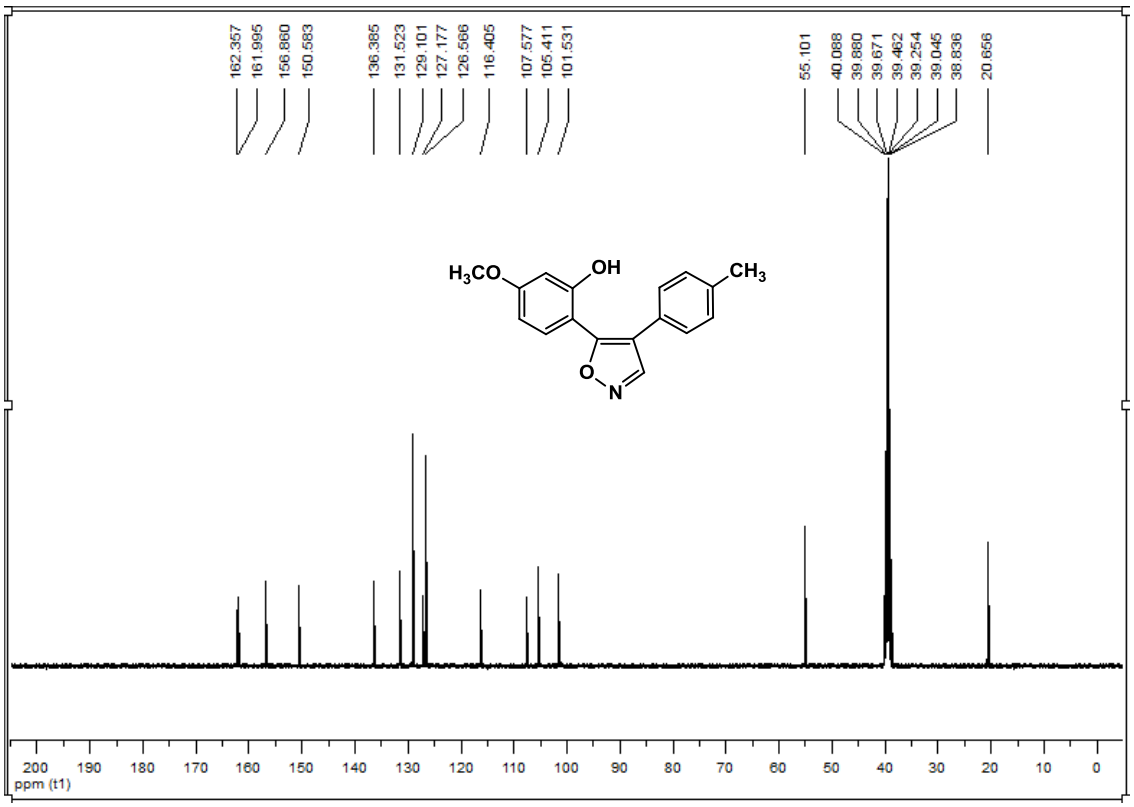
Compound 2k ^{1}H NMR(DMSO-d_6)



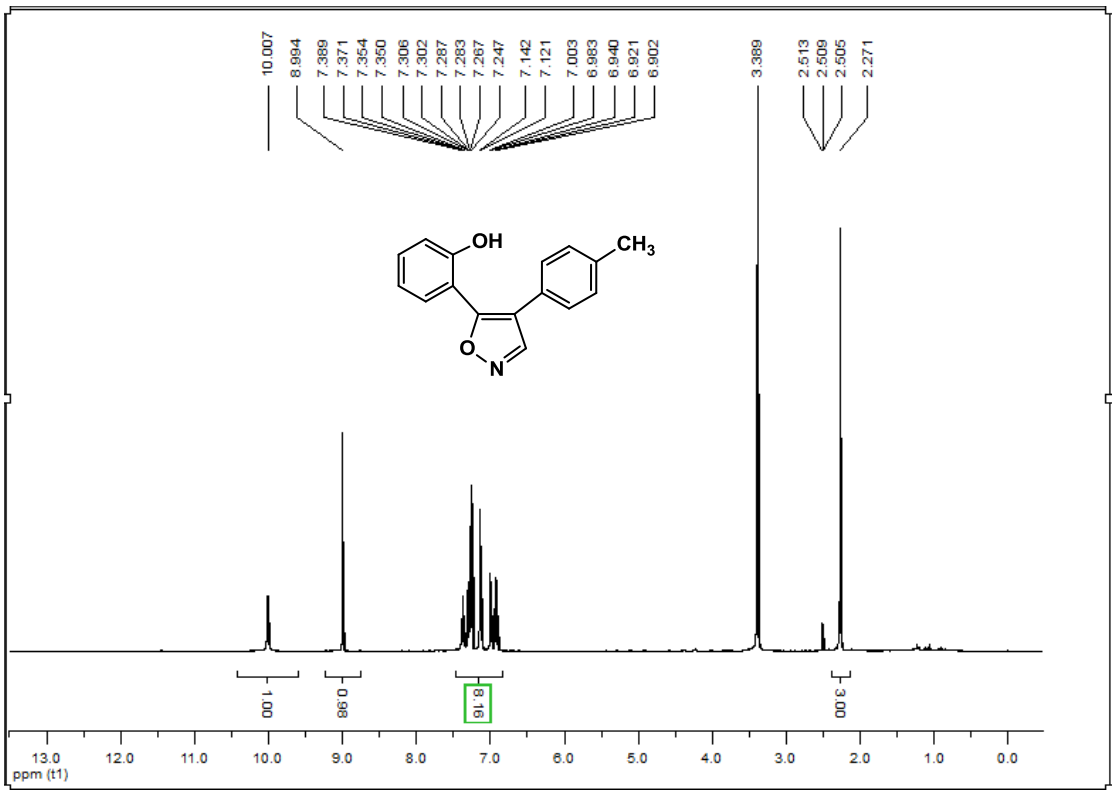
Compound 2k ^{13}C NMR(DMSO-d_6)



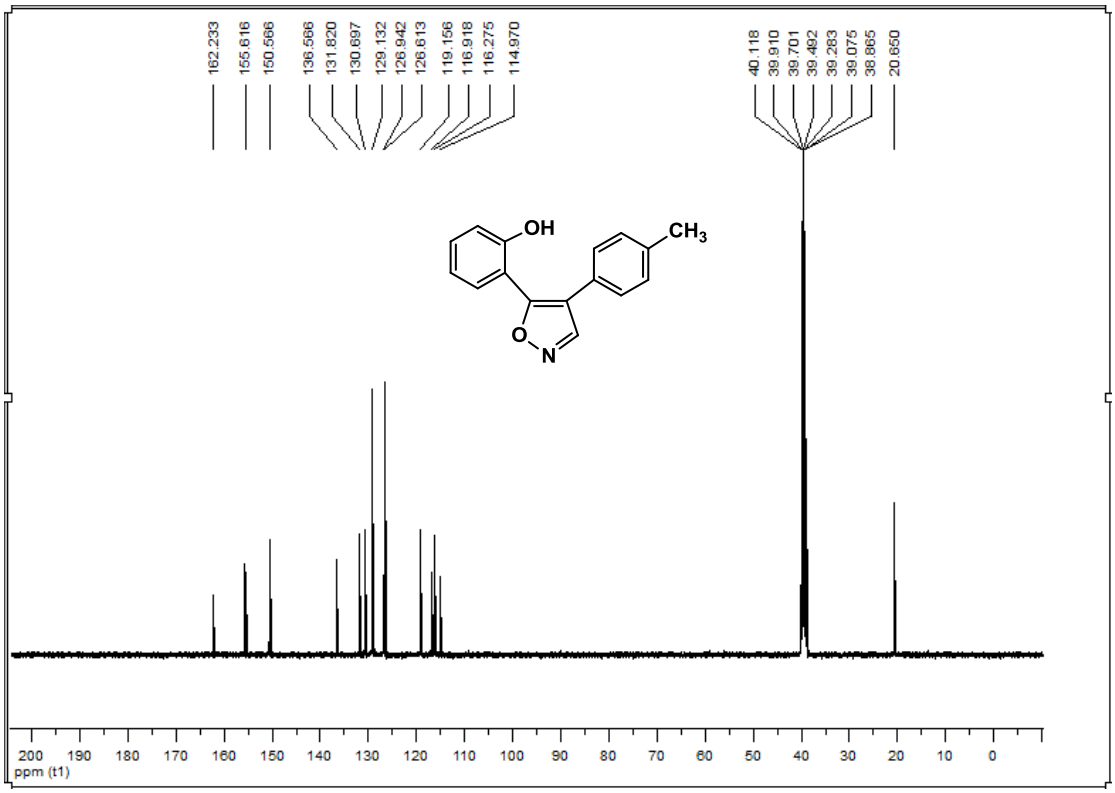
Compound 2l ^{1}H NMR(DMSO-d_6)



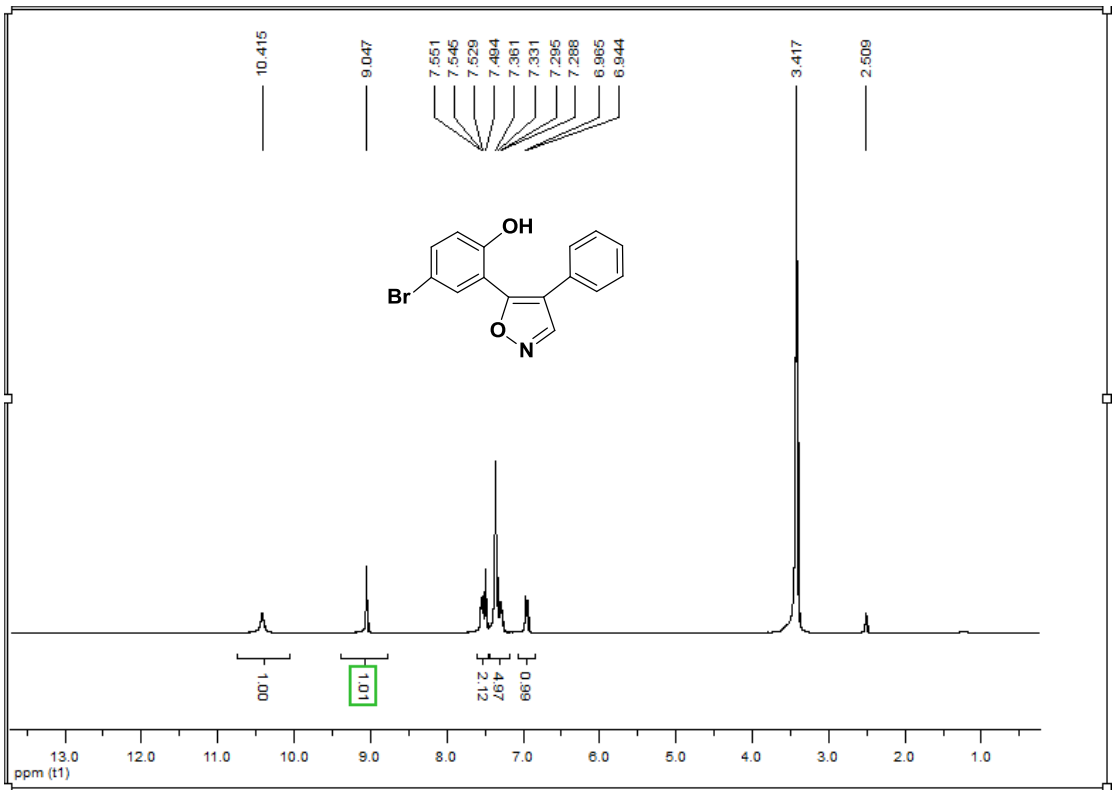
Compound 2l ^{13}C NMR(DMSO-d_6)



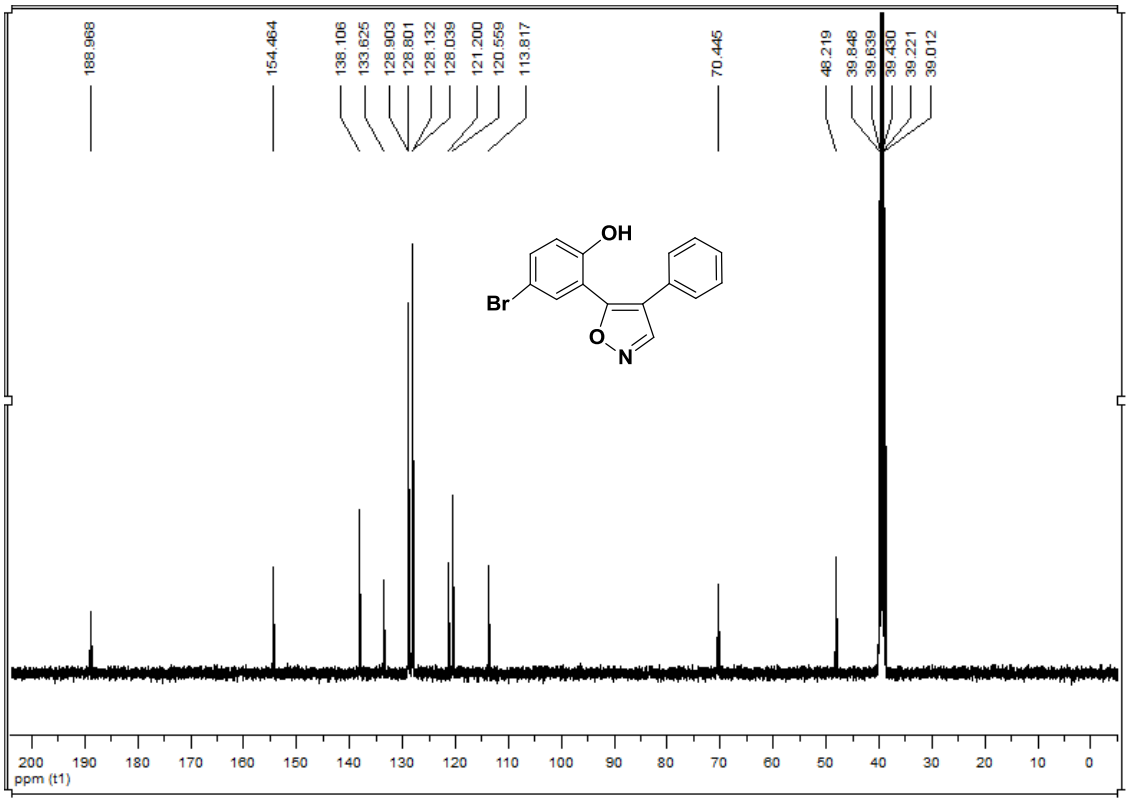
Compound 2m ^{1}H NMR(DMSO-d_6)



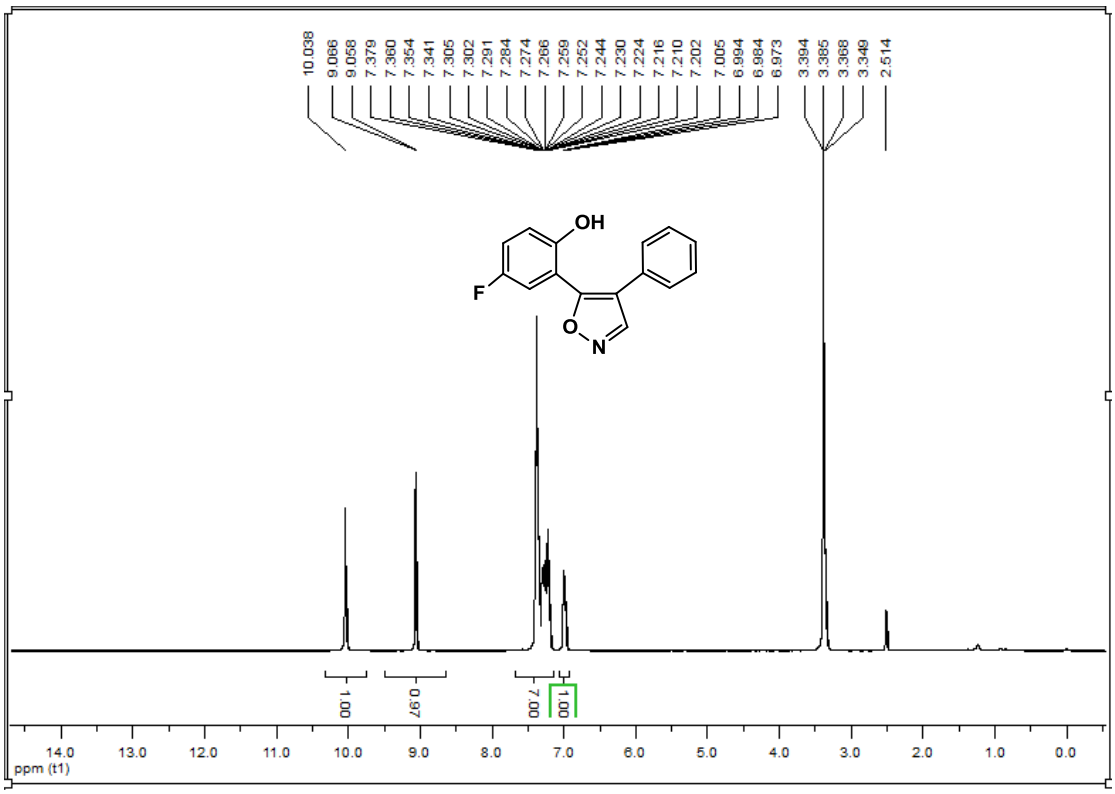
Compound 2m ^{13}C NMR(DMSO-d_6)



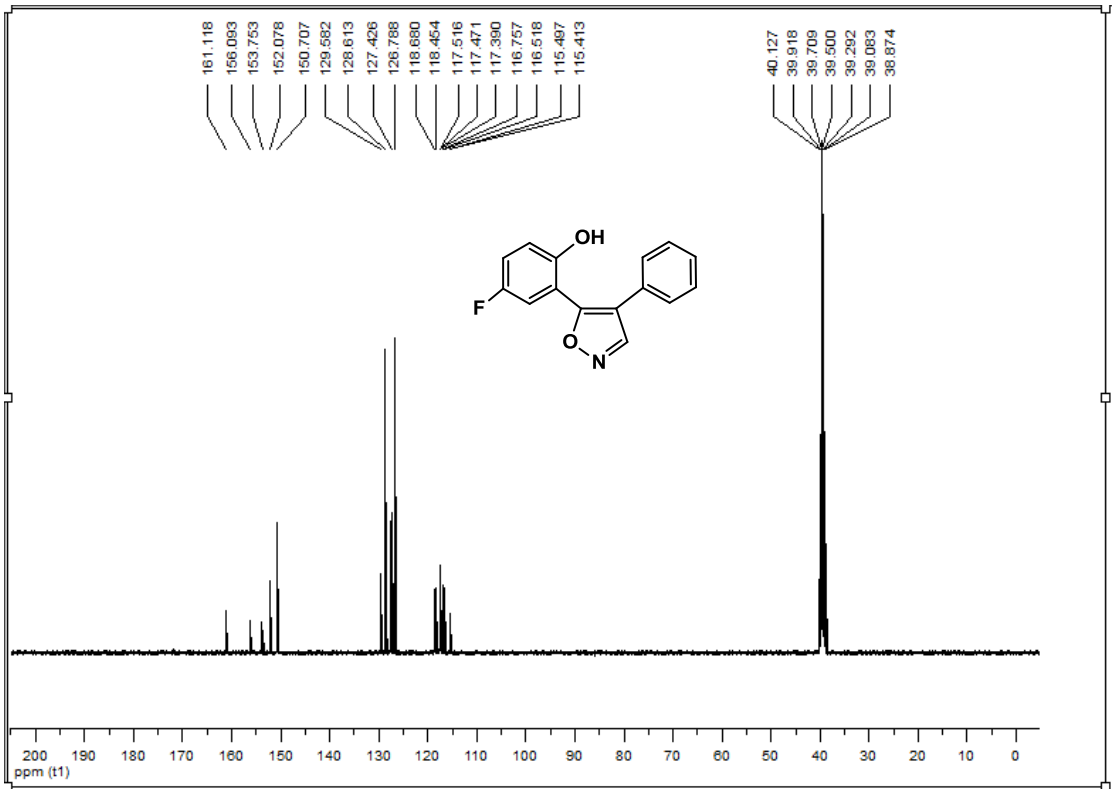
Compound 2n ^{1}H NMR(DMSO-d_6)



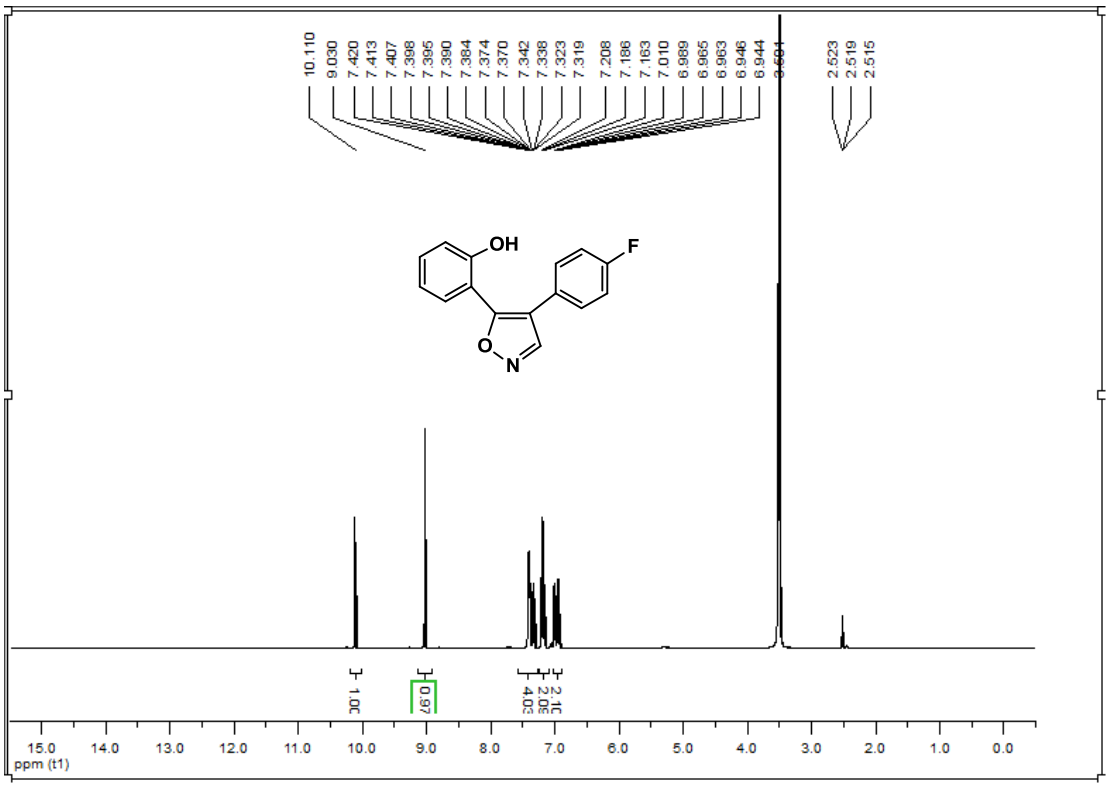
Compound 2n ^{13}C NMR(DMSO-d_6)



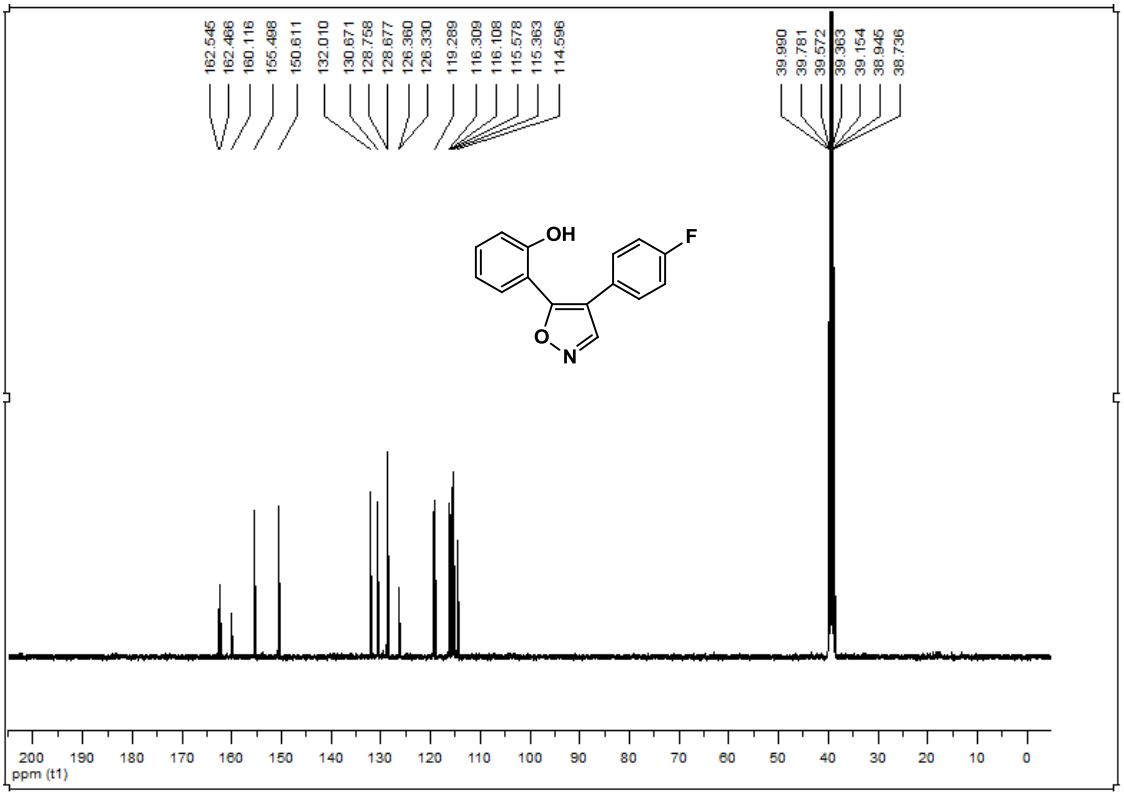
Compound 2o ^{1}H NMR(DMSO-d_6)



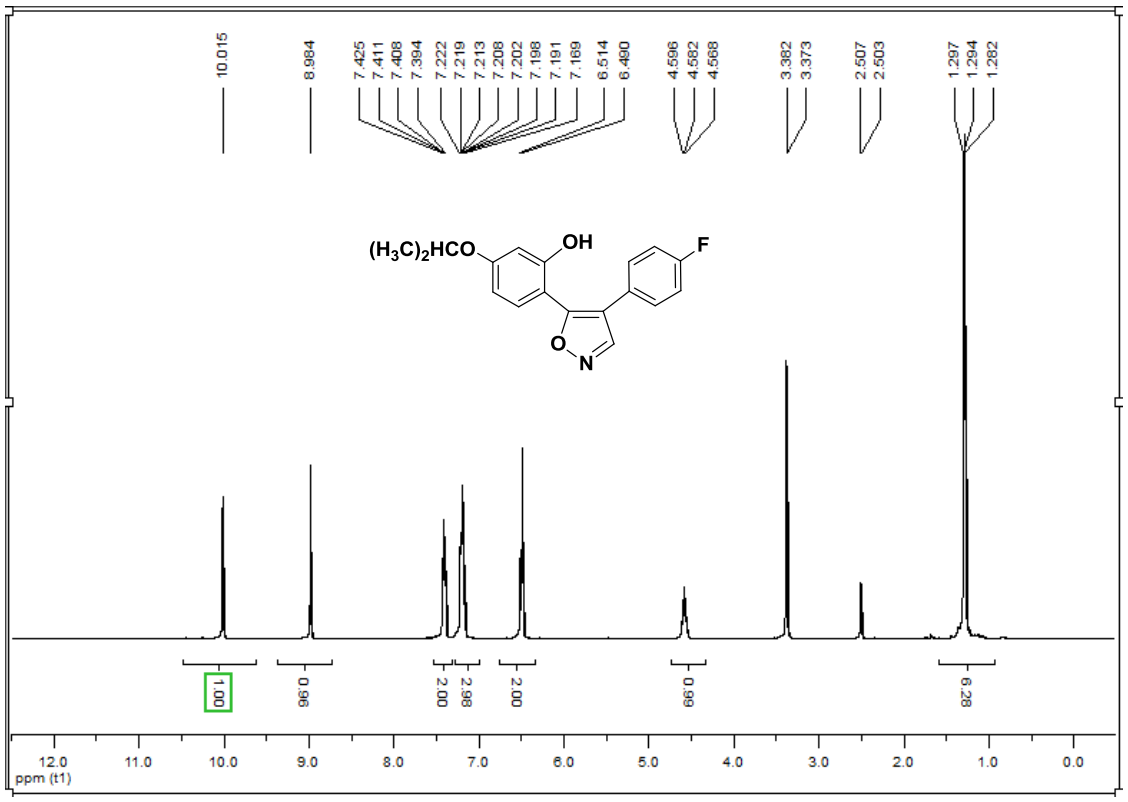
Compound 2o ^{13}C NMR(DMSO-d_6)



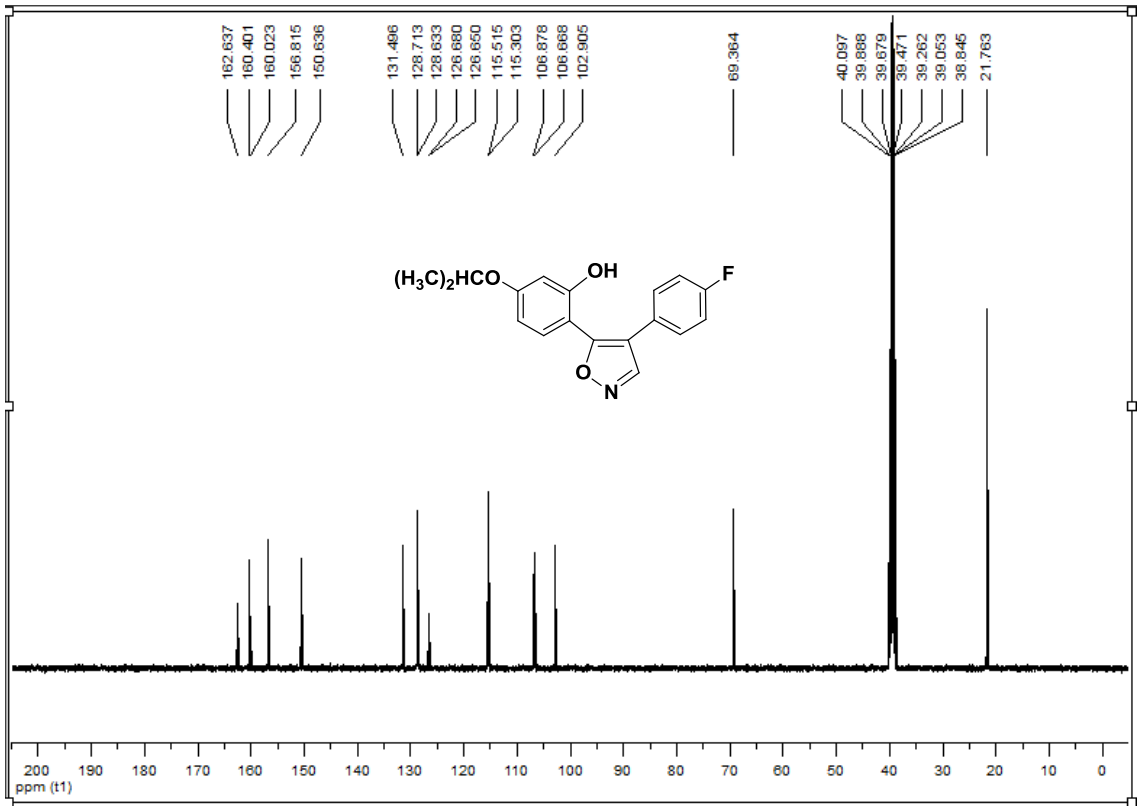
Compound 2p ^{1}H NMR(DMSO-d_6)



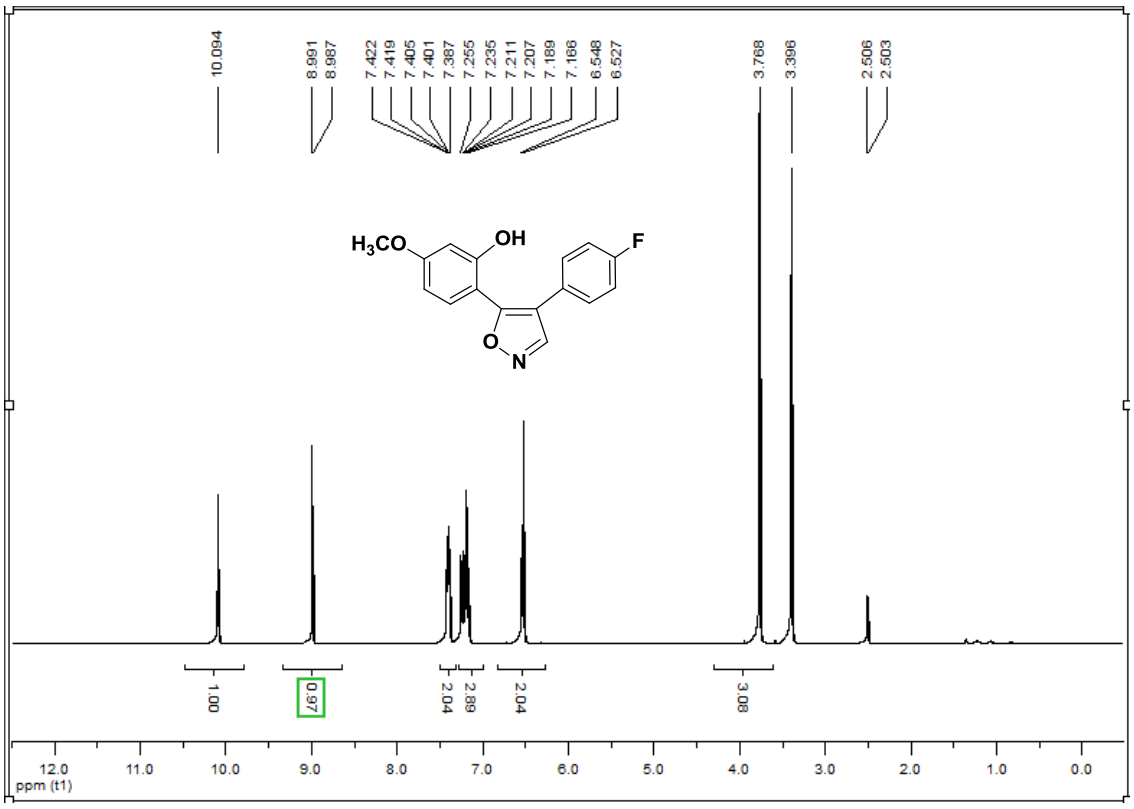
Compound 2p ^{13}C NMR(DMSO-d_6)



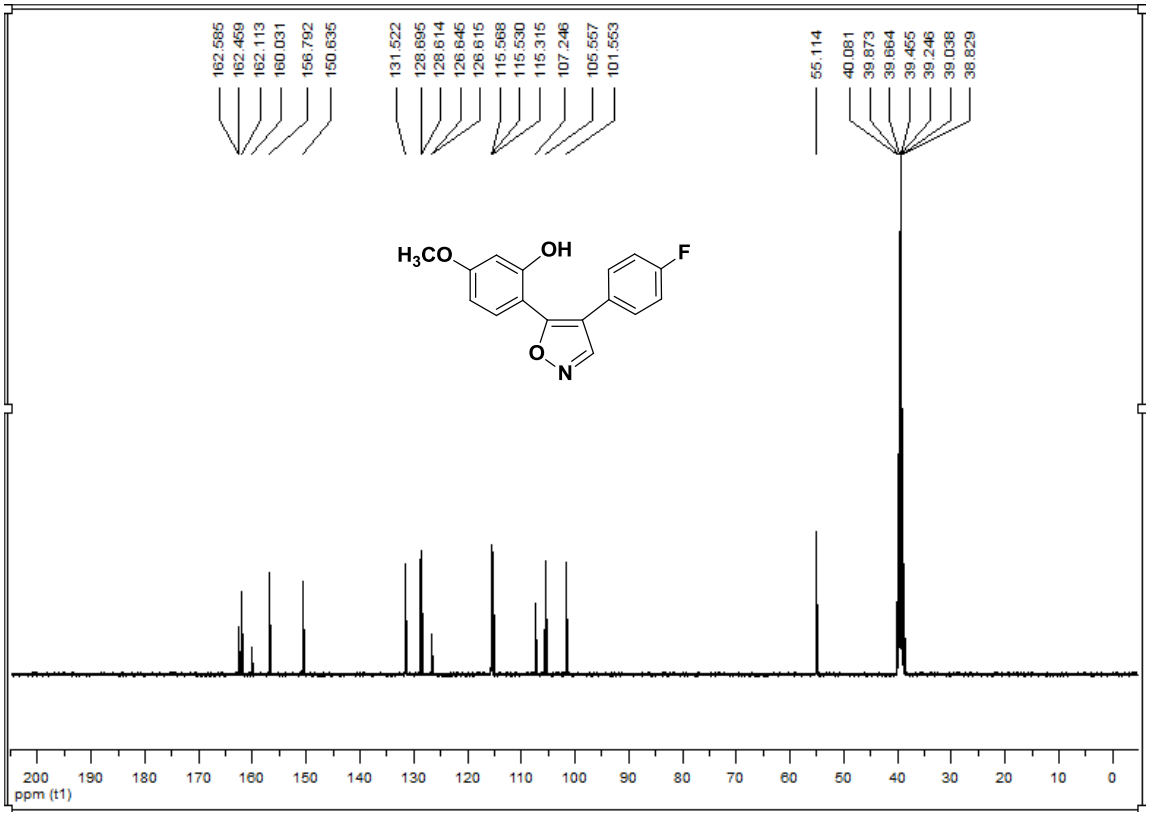
Compound 2q ^{1}H NMR(DMSO-d_6)



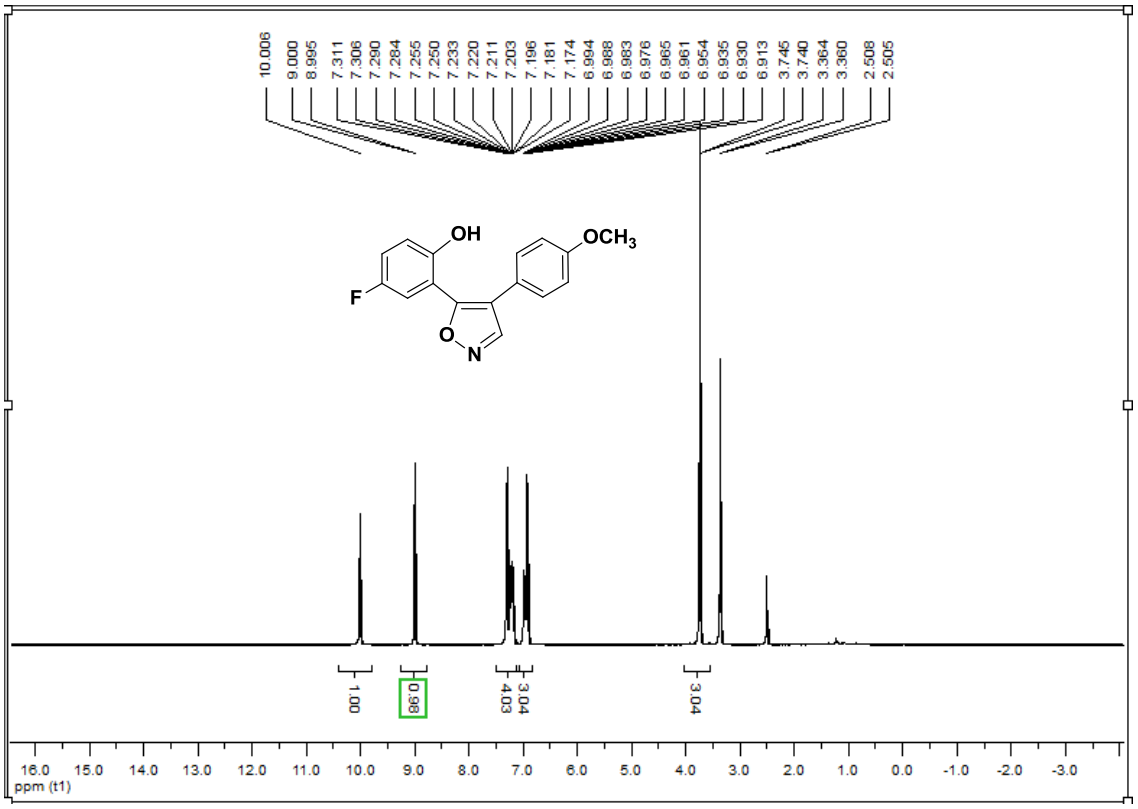
Compound 2q ^{13}C NMR(DMSO-d_6)



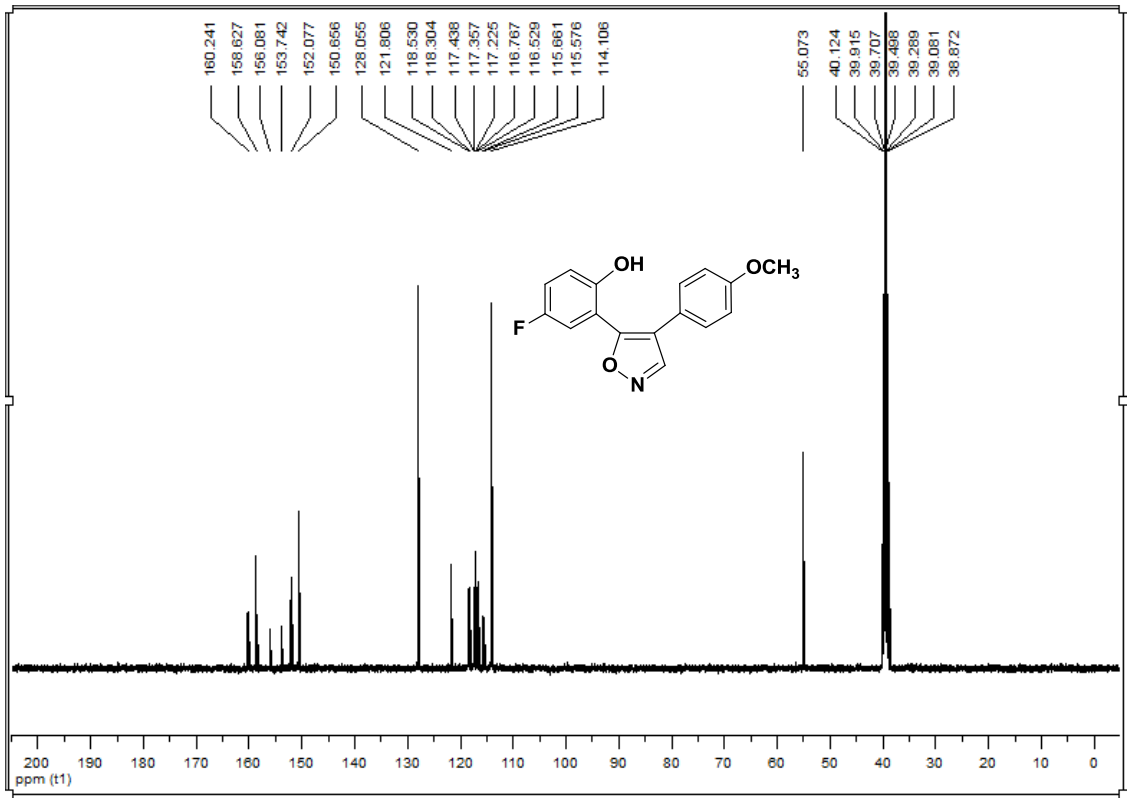
Compound 2r ^{1}H NMR(DMSO-d_6)



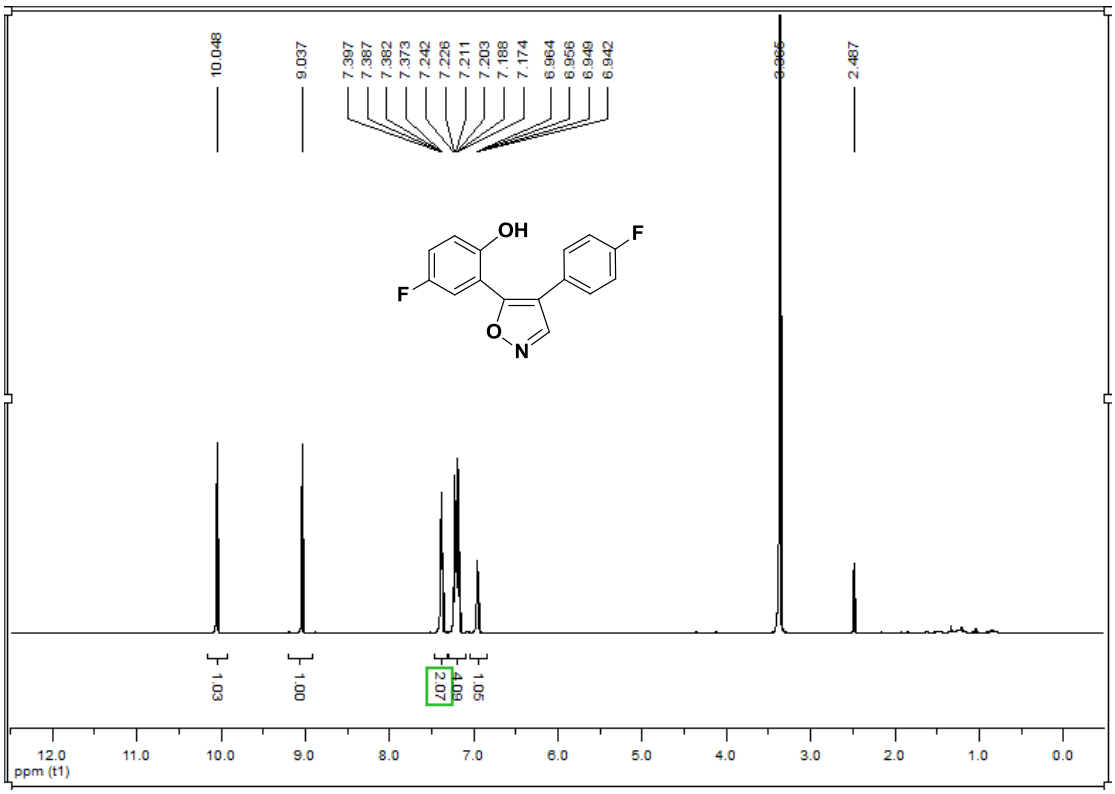
Compound 2r ^{13}C NMR(DMSO-d_6)



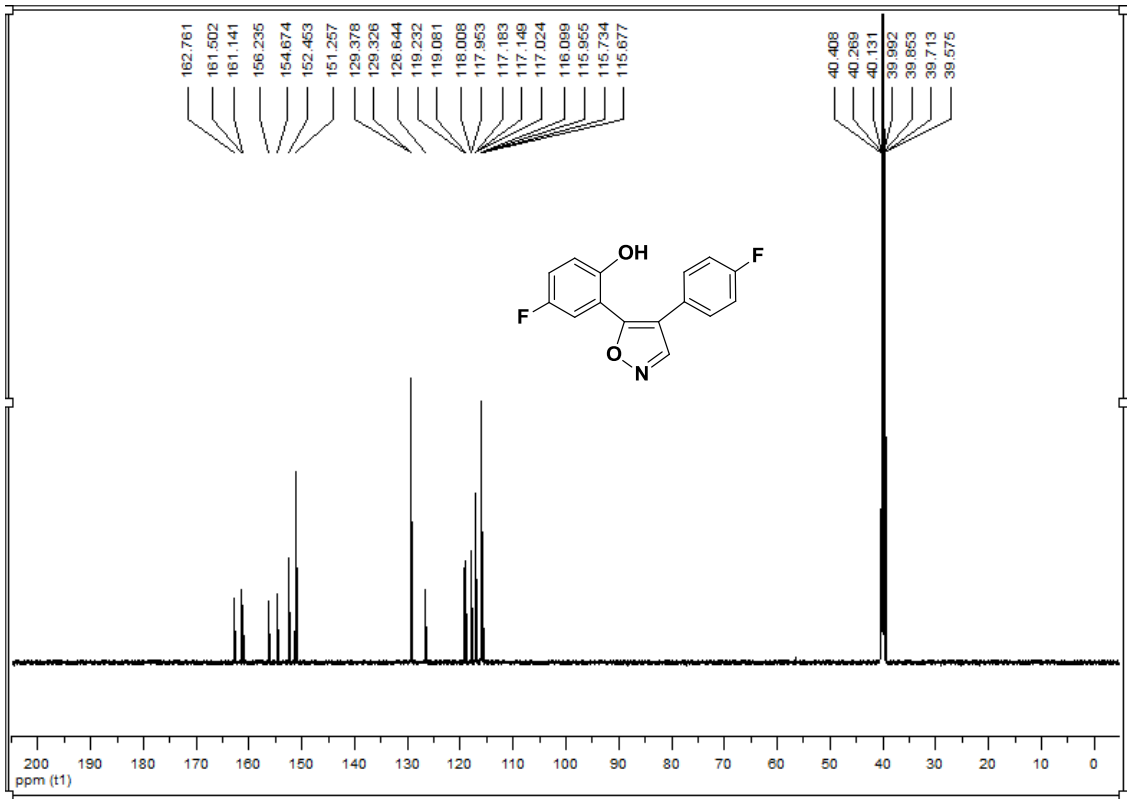
Compound 2s ^{1}H NMR(DMSO-d_6)



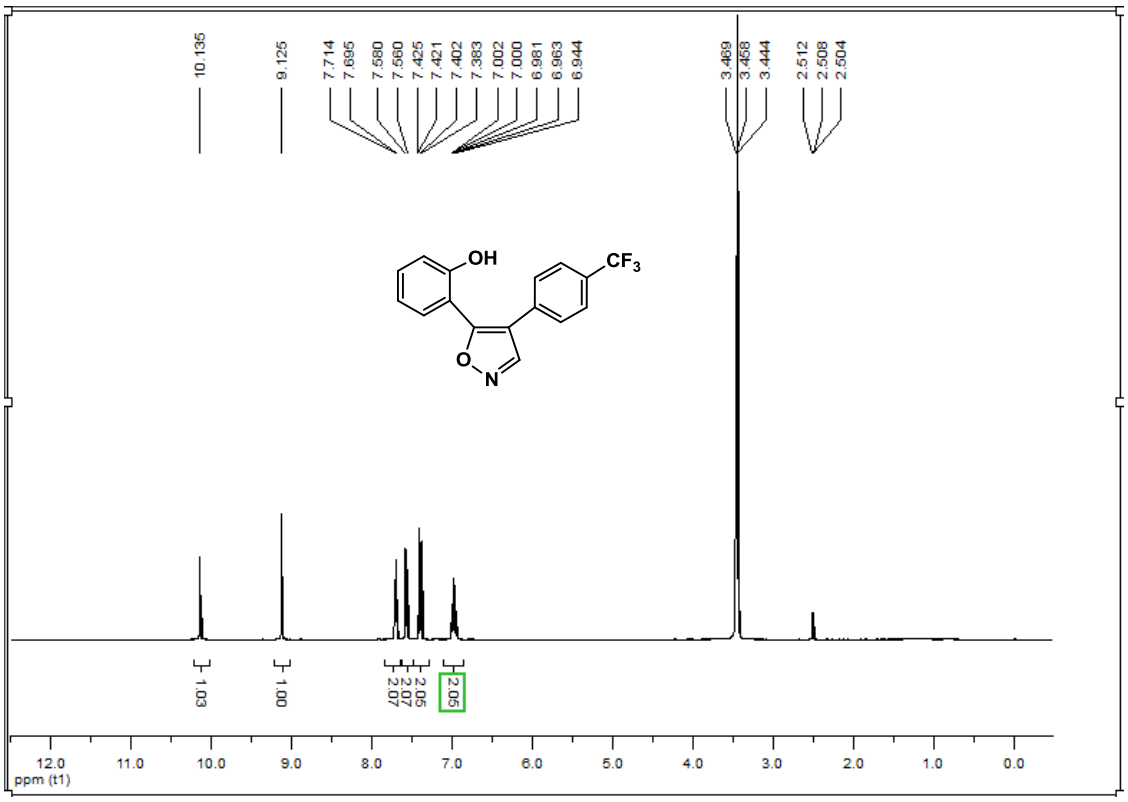
Compound 2s ^{13}C NMR(DMSO-d_6)



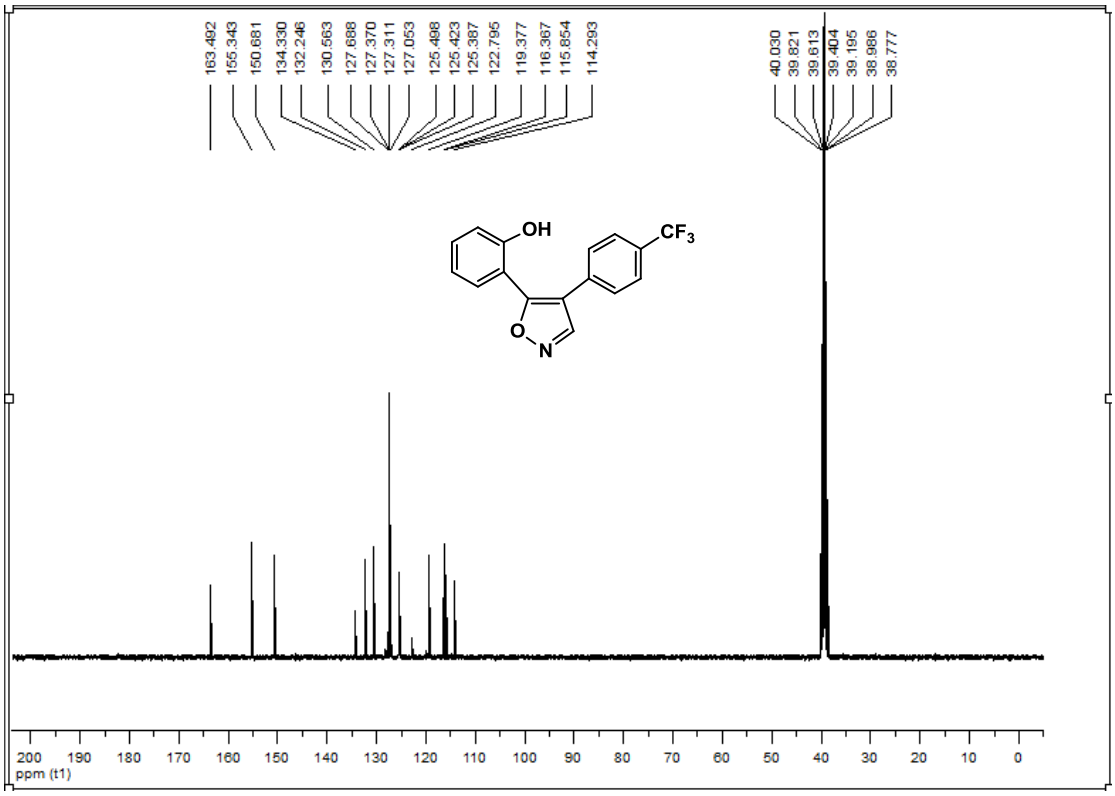
Compound 2t ^{1}H NMR(DMSO-d_6)



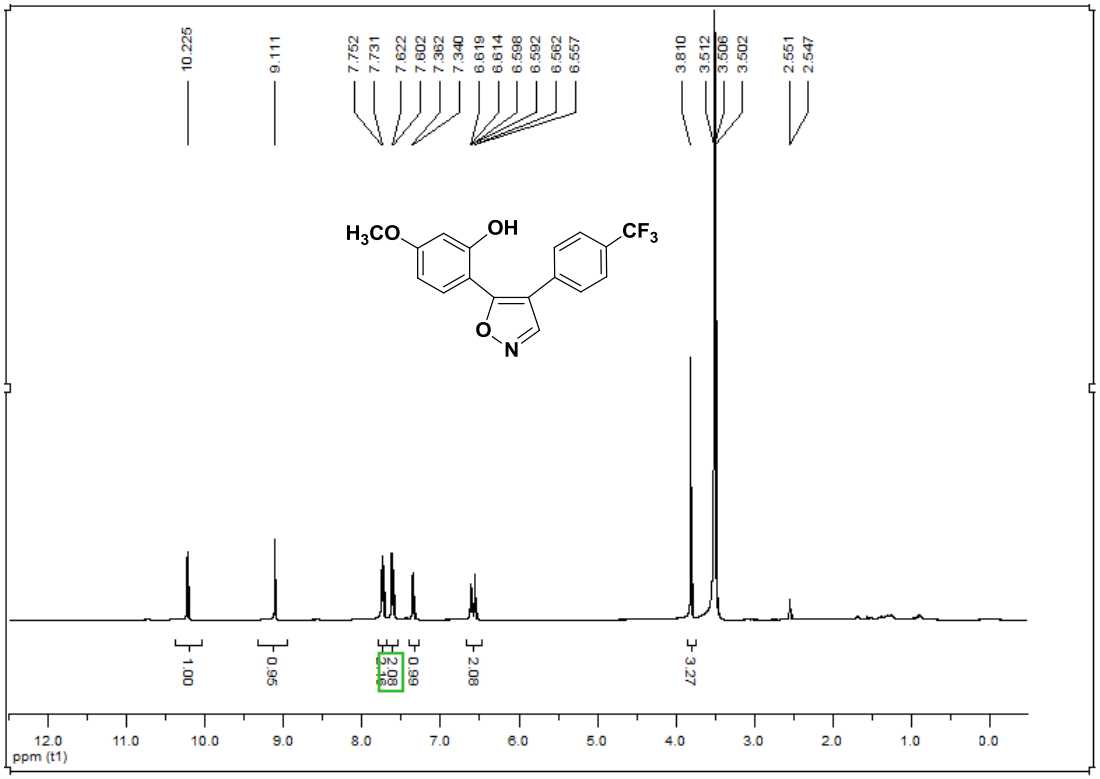
Compound 2t ^{13}C NMR(DMSO-d_6)



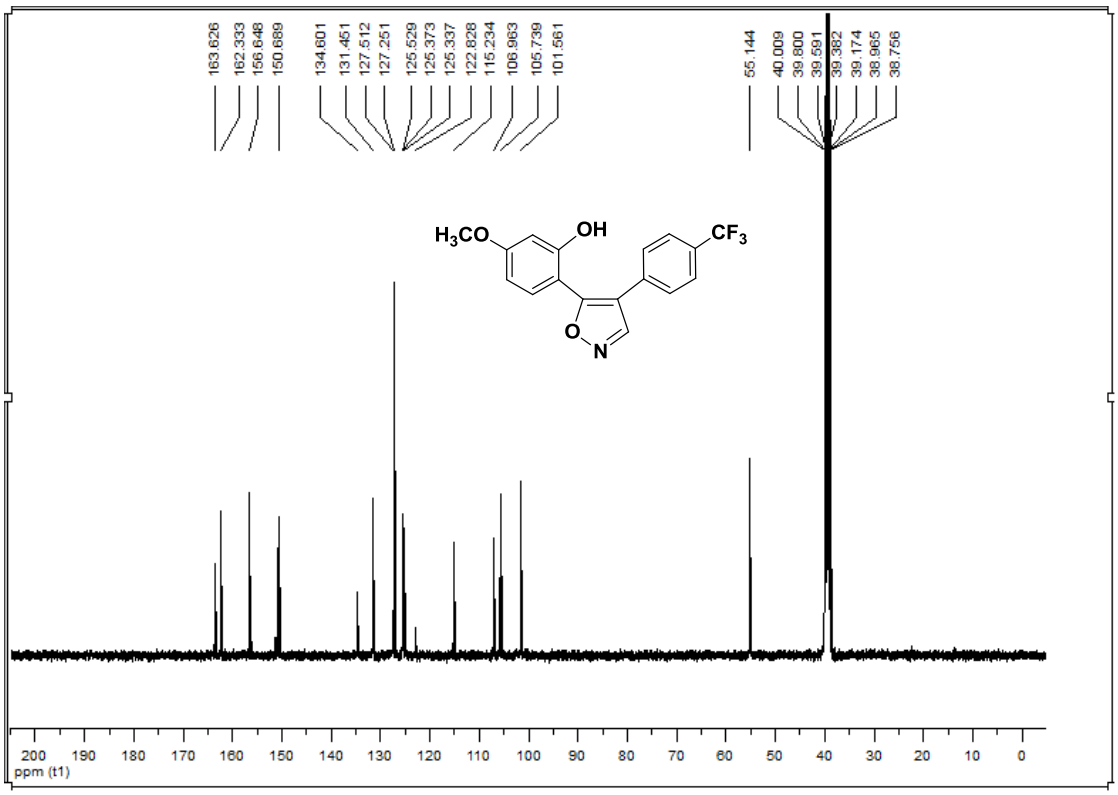
Compound 2u ^{1}H NMR(DMSO-d_6)



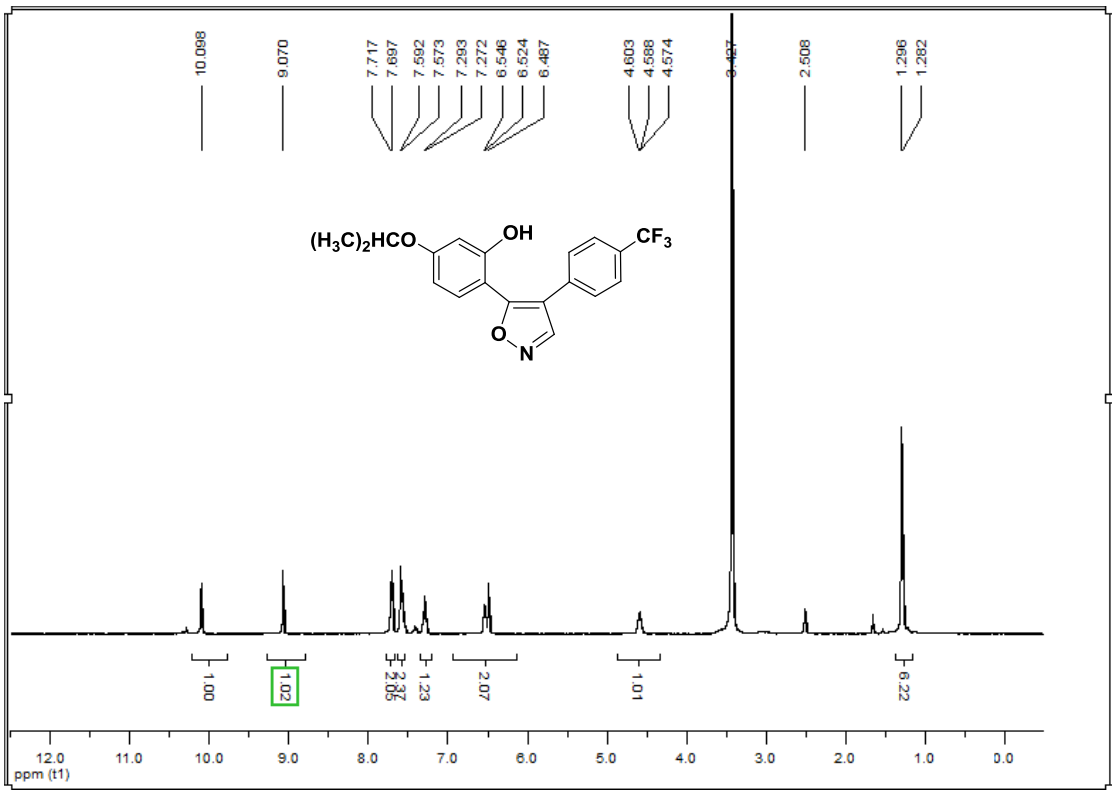
Compound 2u ^{13}C NMR(DMSO-d_6)



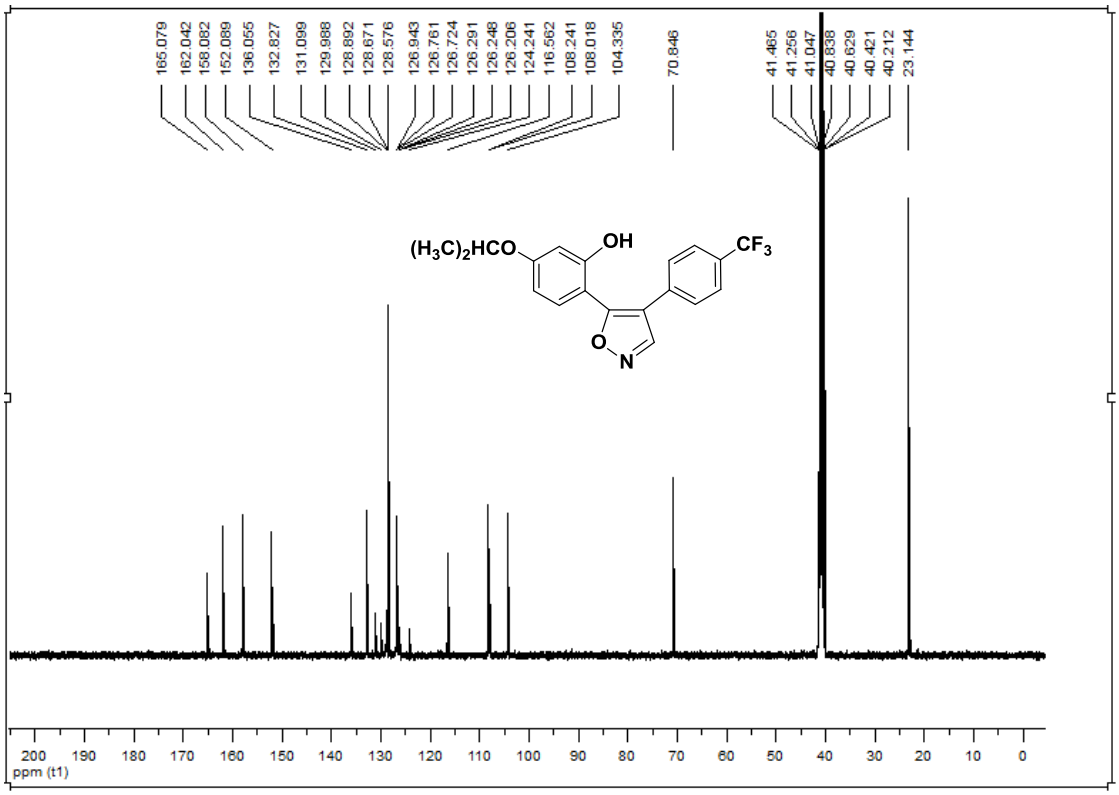
Compound 2v ^{1}H NMR(DMSO-d_6)



Compound 2v ^{13}C NMR(DMSO-d_6)

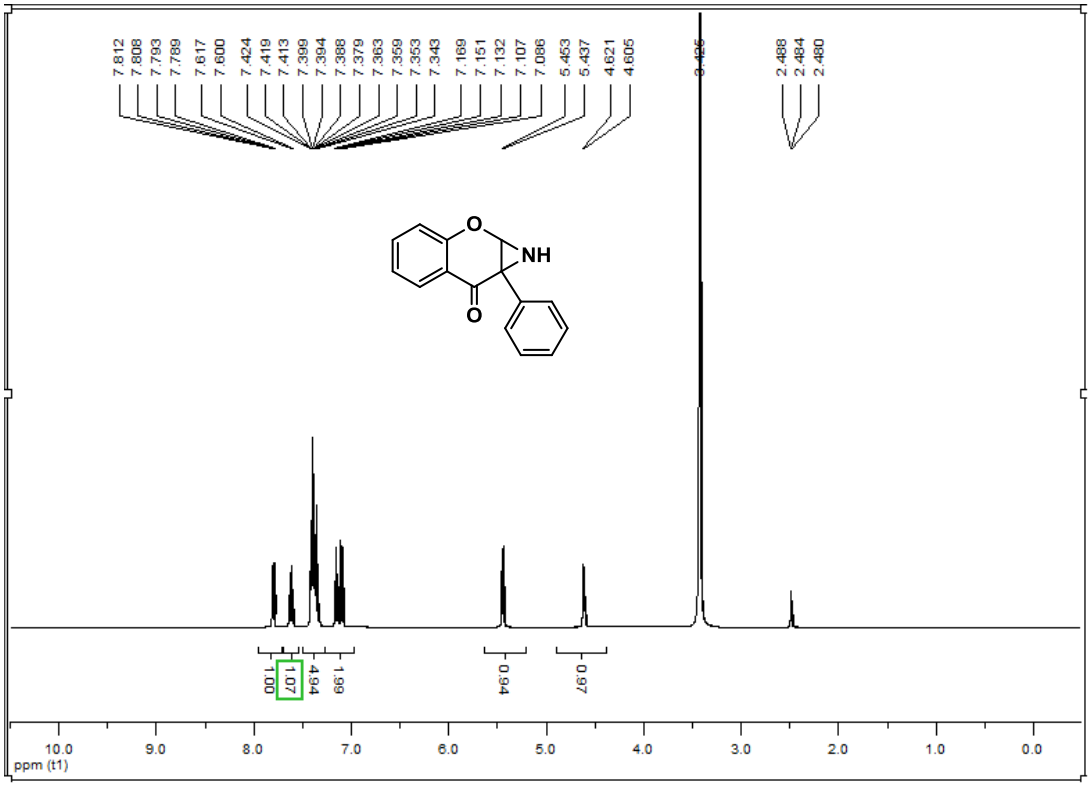


Compound 2w ^{1}H NMR(DMSO-d_6)

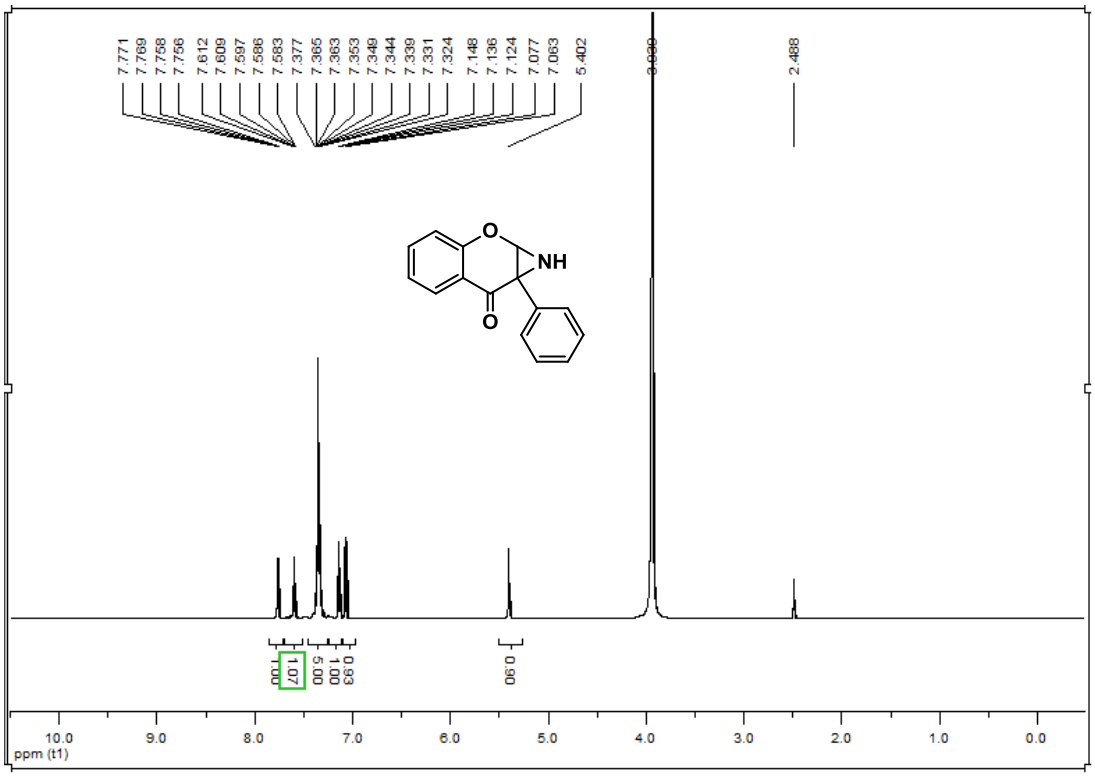


Compound 2w ^{13}C NMR(DMSO-d_6)

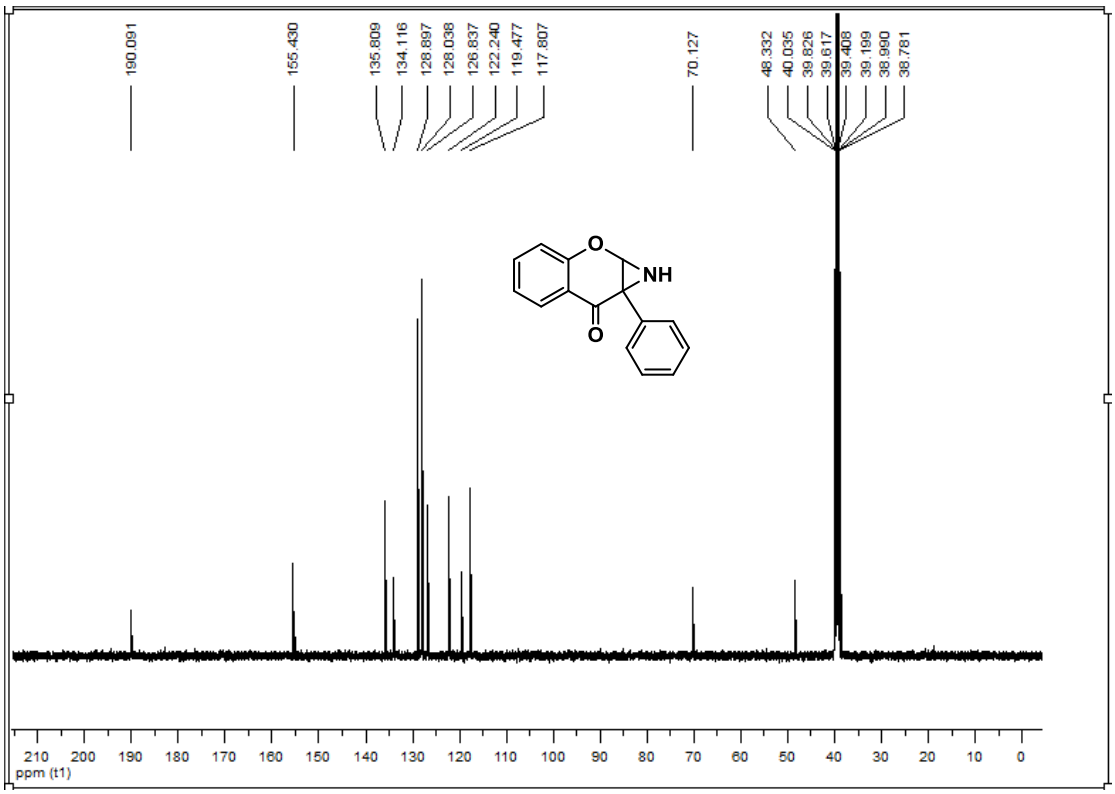
^{1}H NMR, ^{13}C NMR and HR MS spectra for products 1a-w.



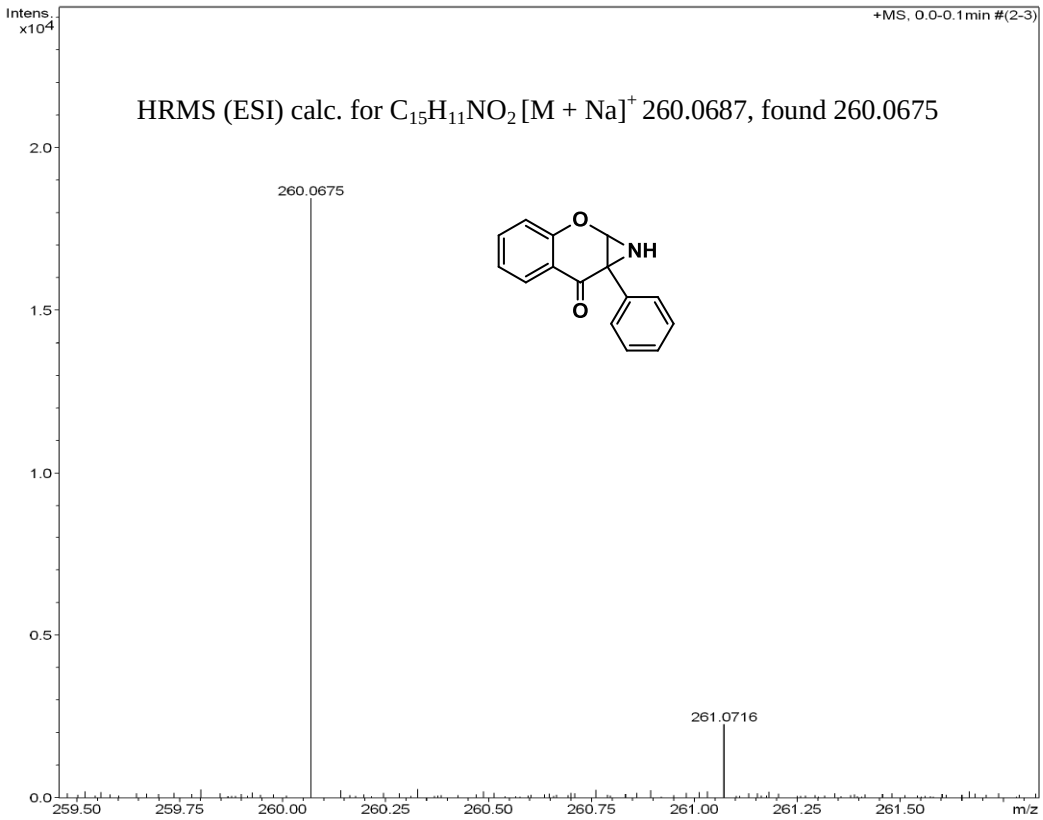
Compound 1a ^{1}H NMR(DMSO-d_6)



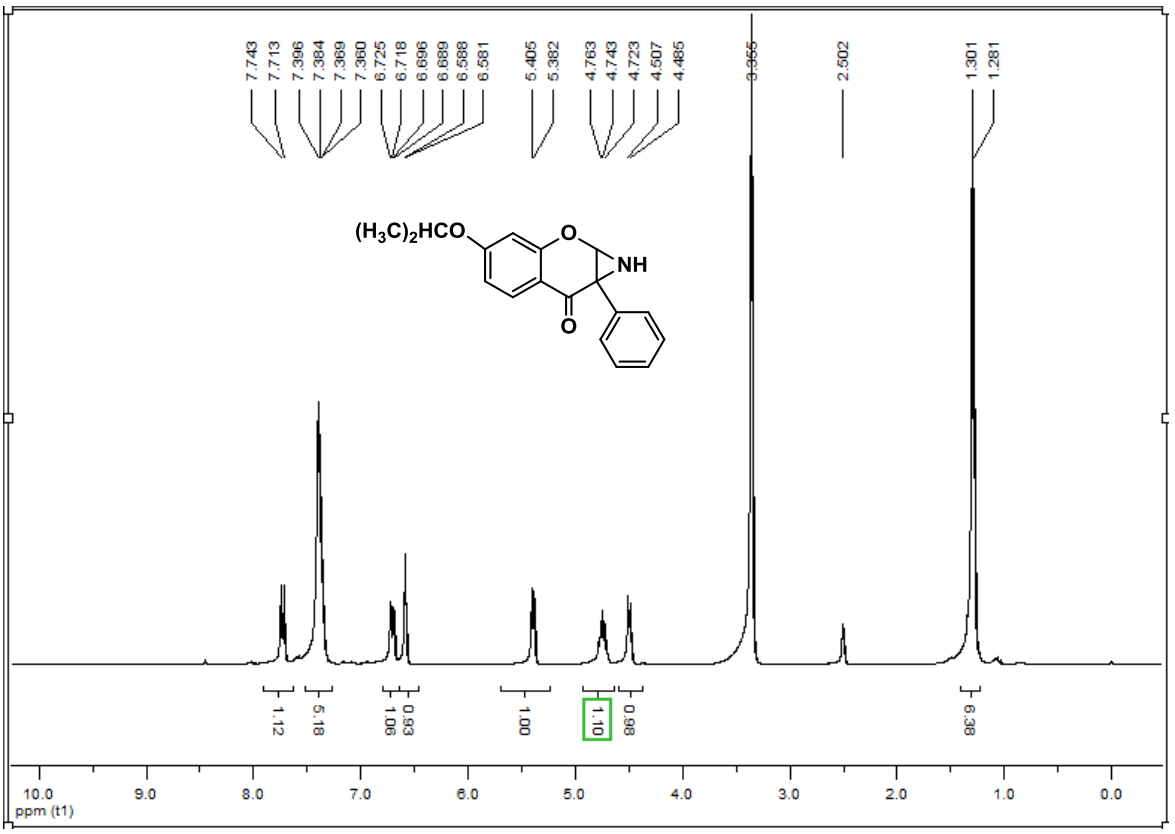
Compound 1a ^{1}H NMR(DMSO-d_6+D_2O)



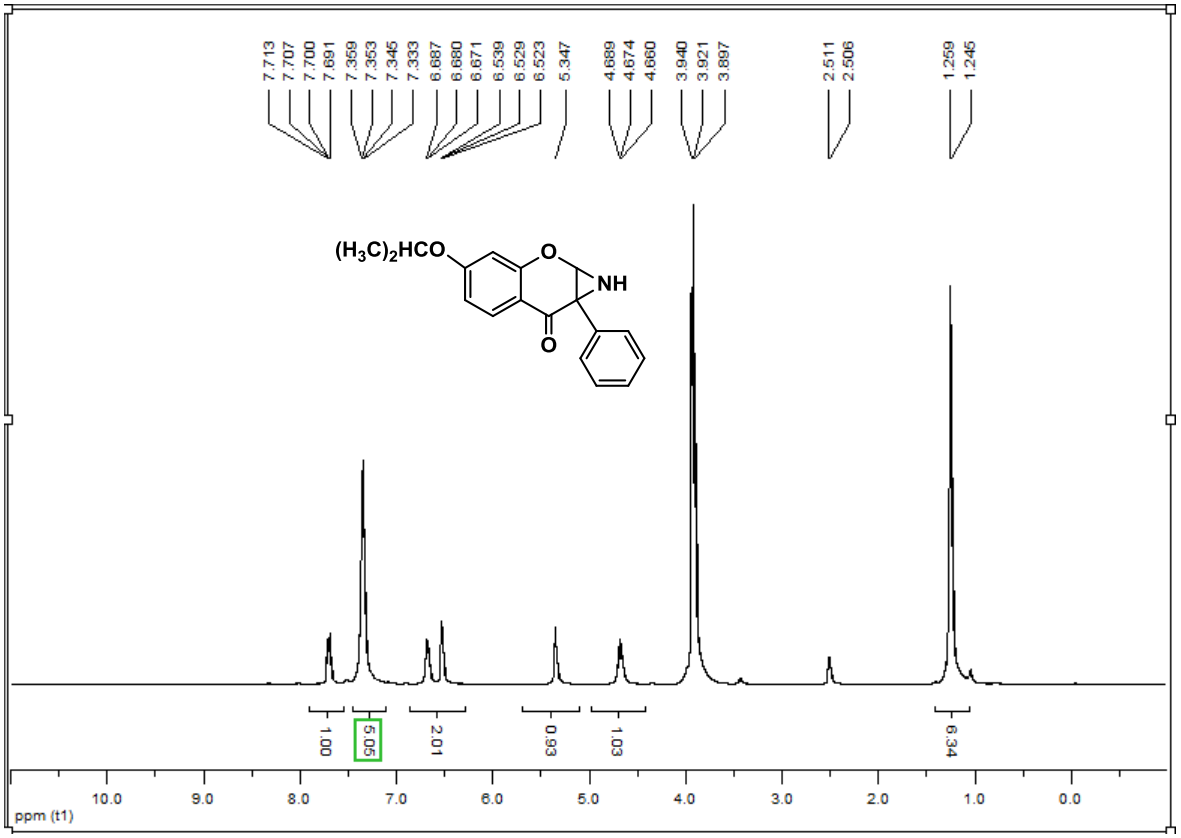
Compound 1a ^{13}C NMR(DMSO-d_6)



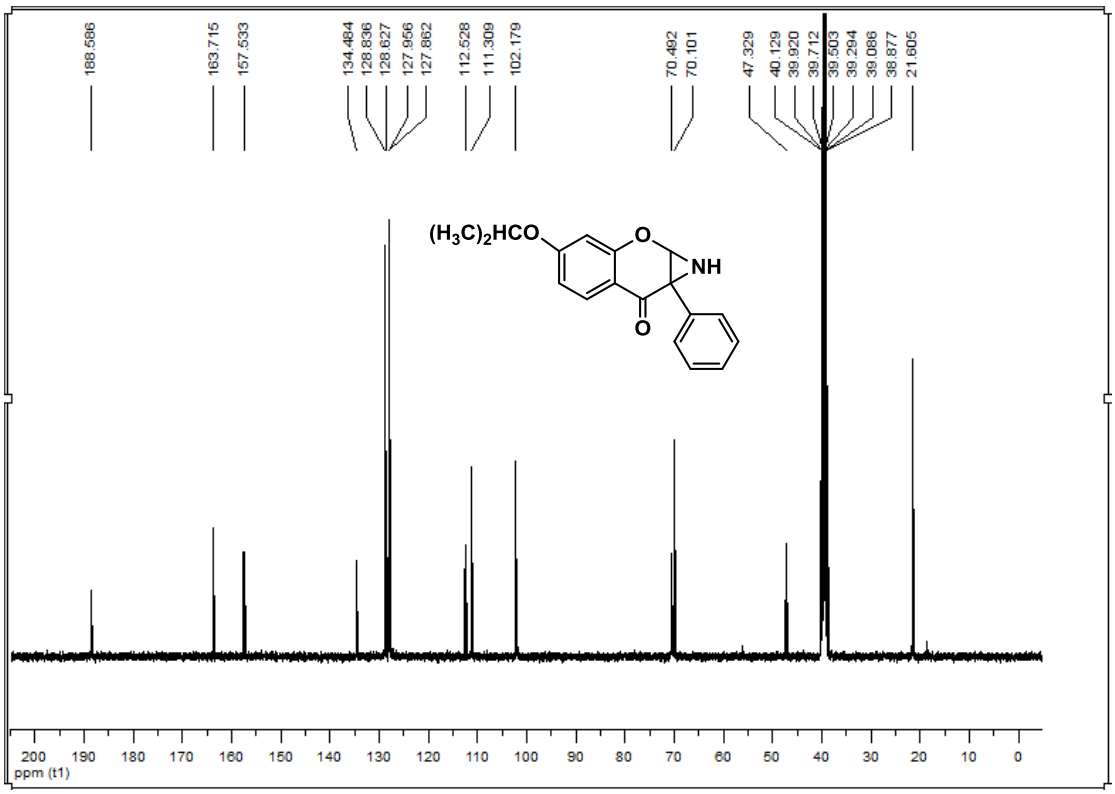
Compound 1a HR MS(CH_3OH)



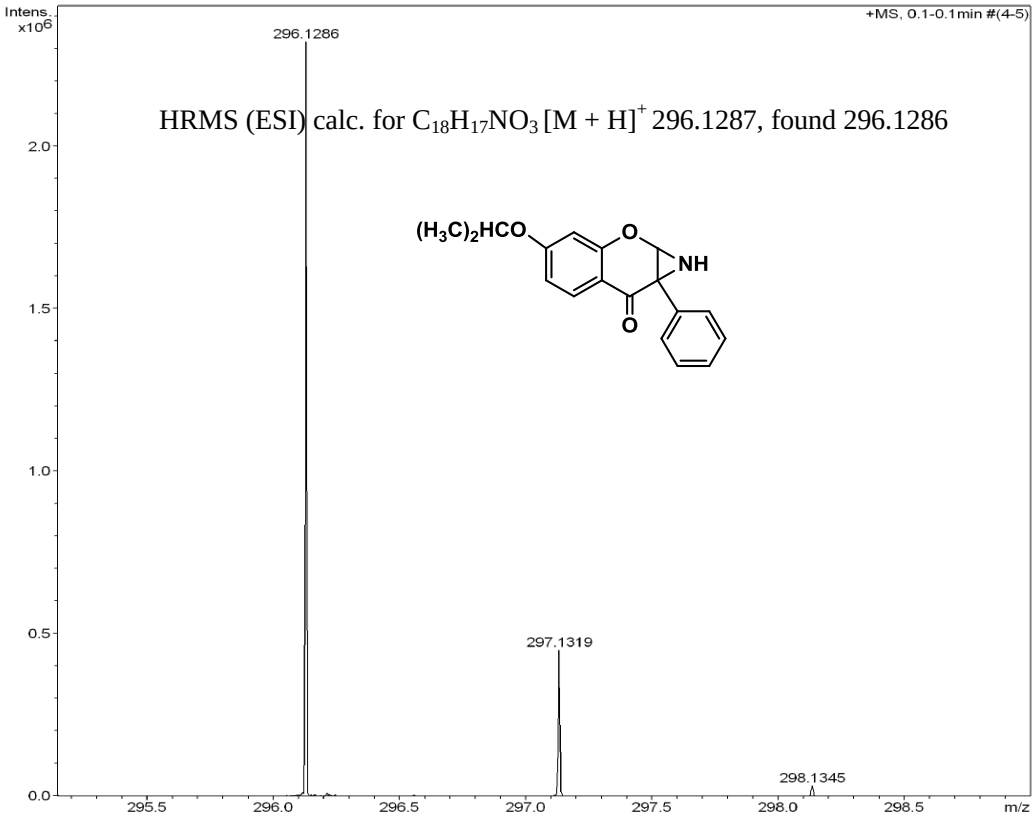
Compound 1b ^{1}H NMR(DMSO-d_6)



Compound 1b ^{1}H NMR(DMSO-d_6+D_2O)

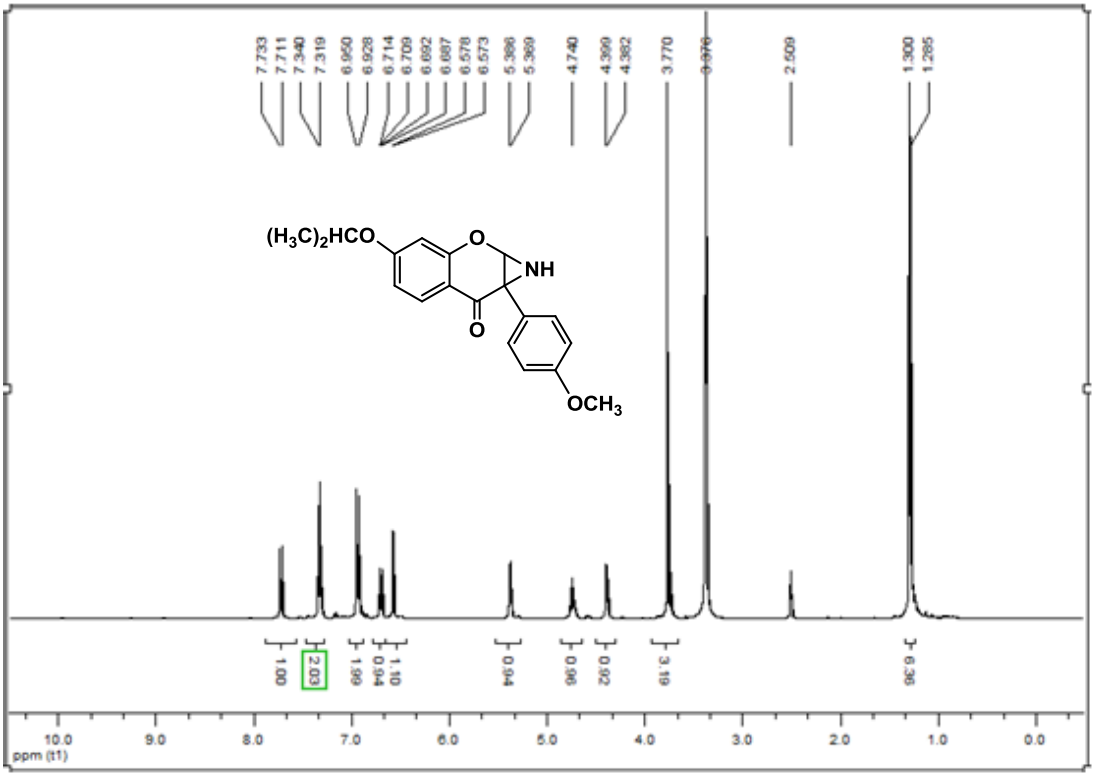


Compound 1b ^{13}C NMR(DMSO-d_6)

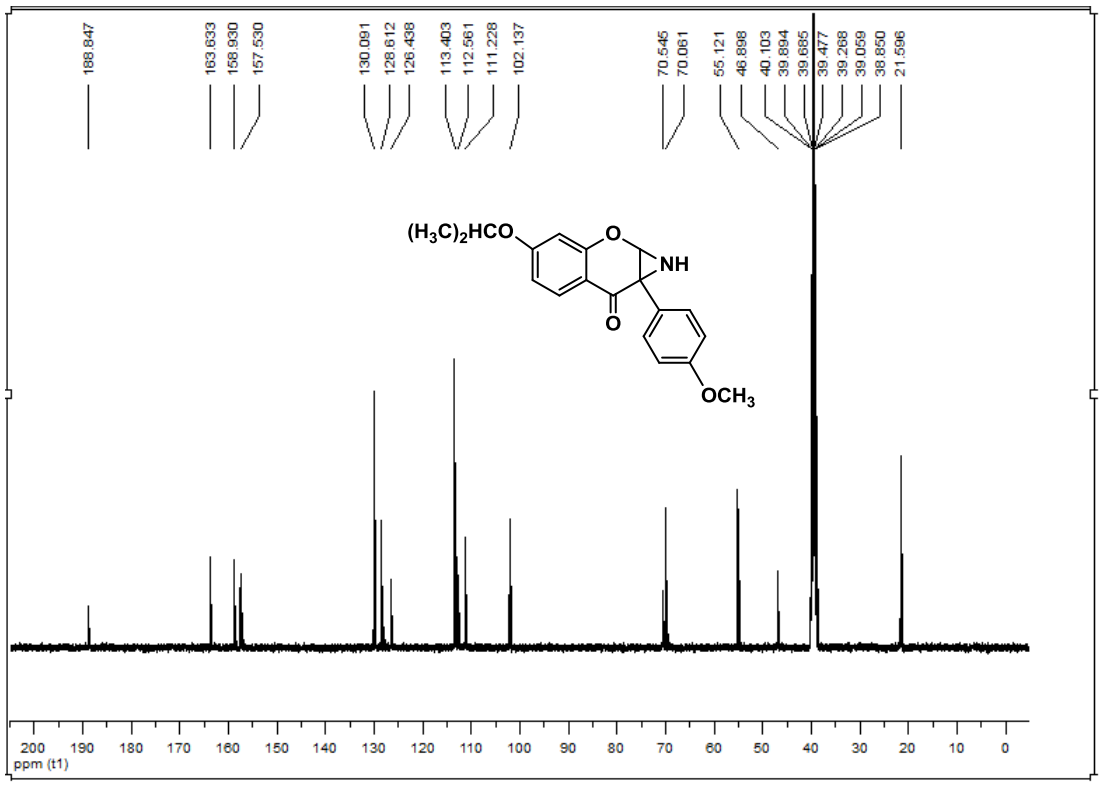


HRMS (ESI) calc. for $C_{18}H_{17}NO_3$ $[M + H]^+$ 296.1287, found 296.1286

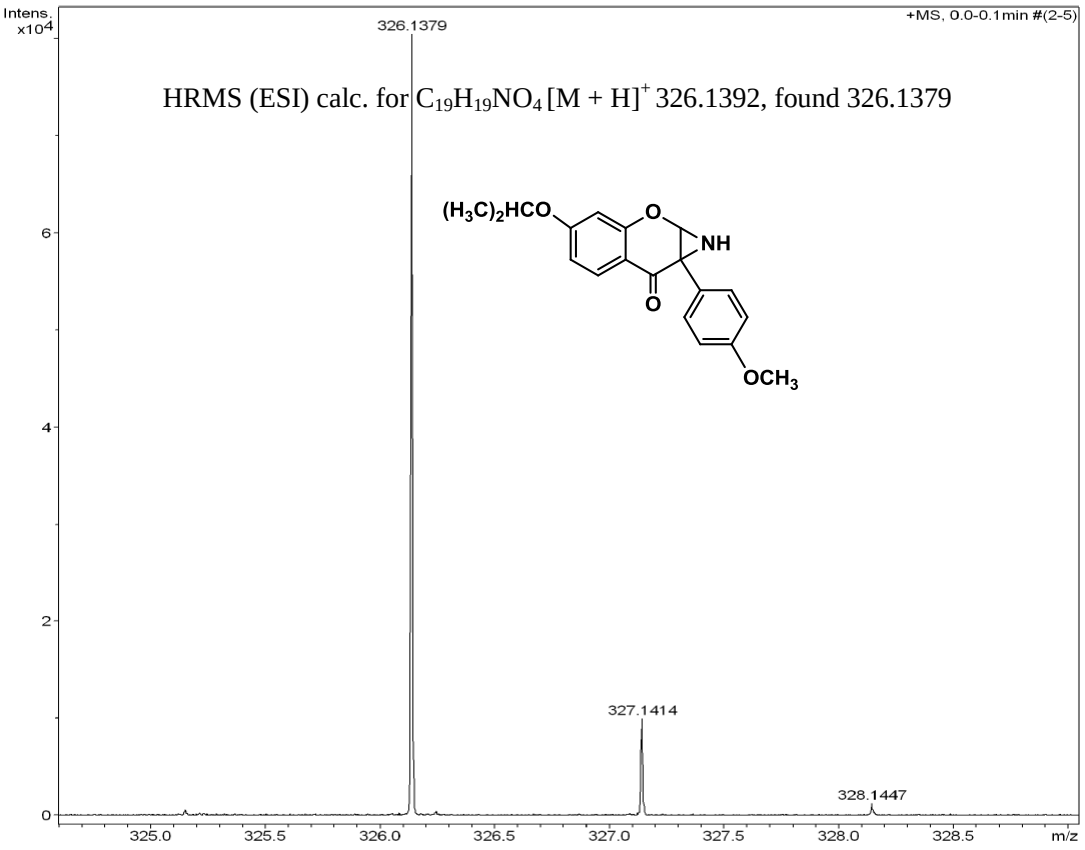
Compound 1b HR MS(CH_3OH)



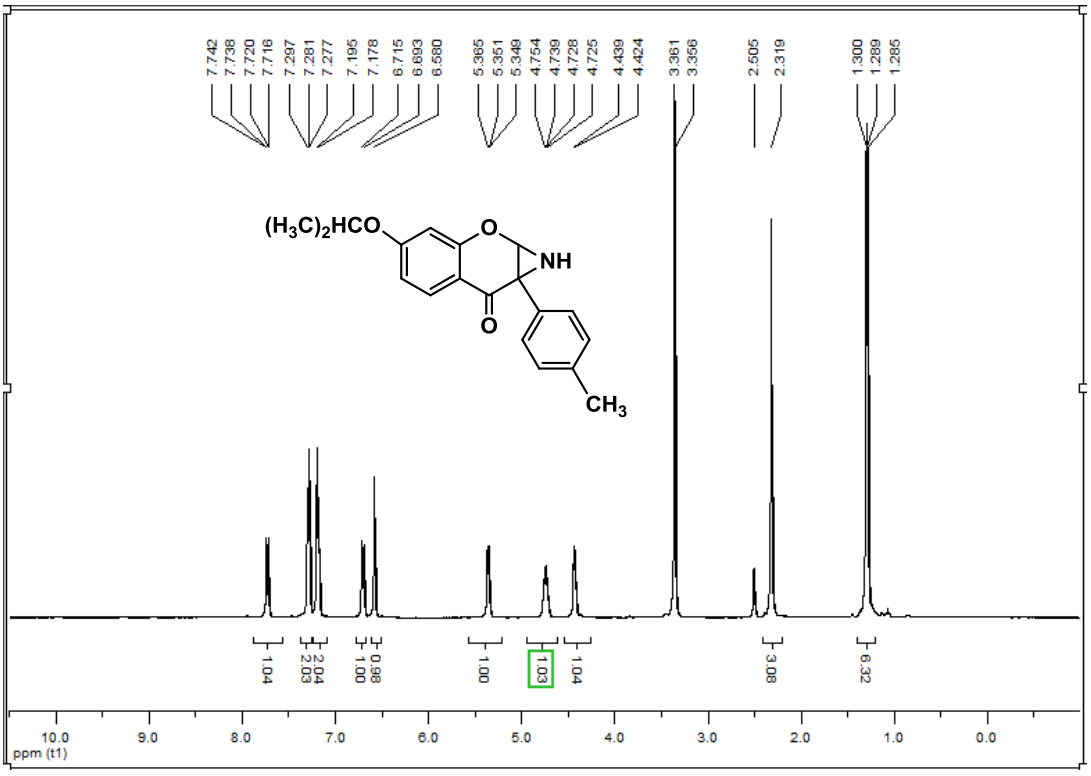
Compound 1c ^{1}H NMR(DMSO-d_6)



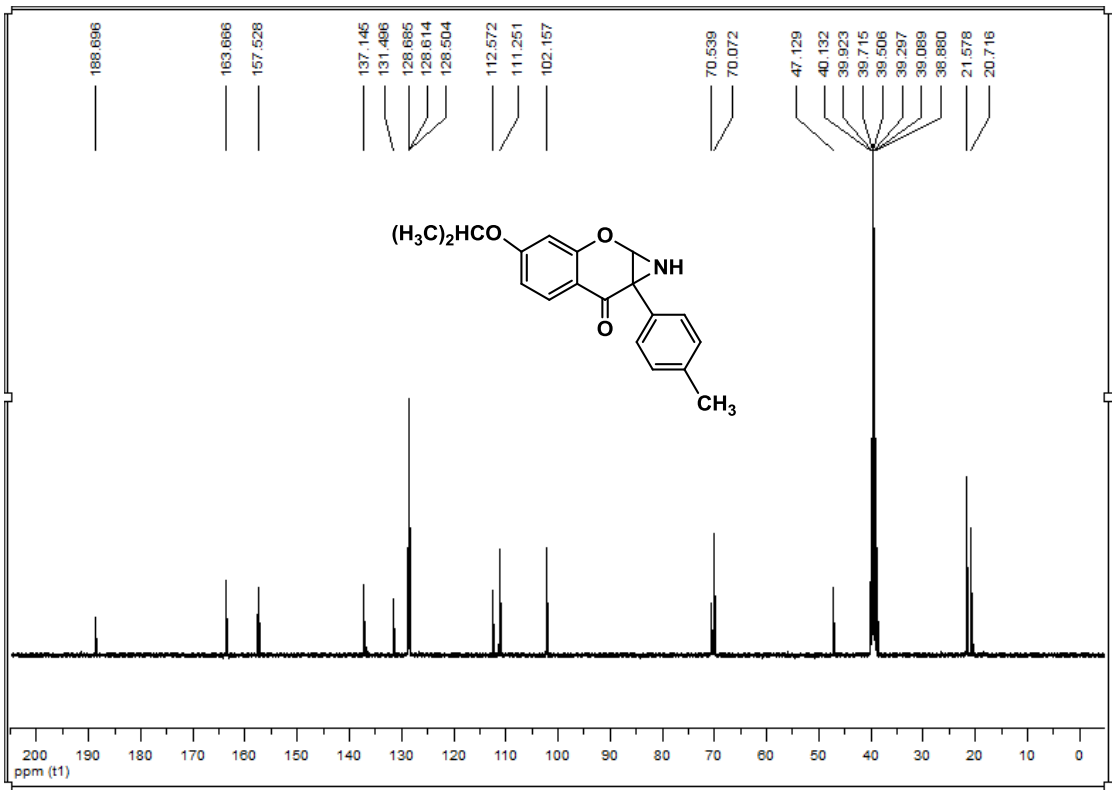
Compound 1c ^{13}C NMR(DMSO-d_6)



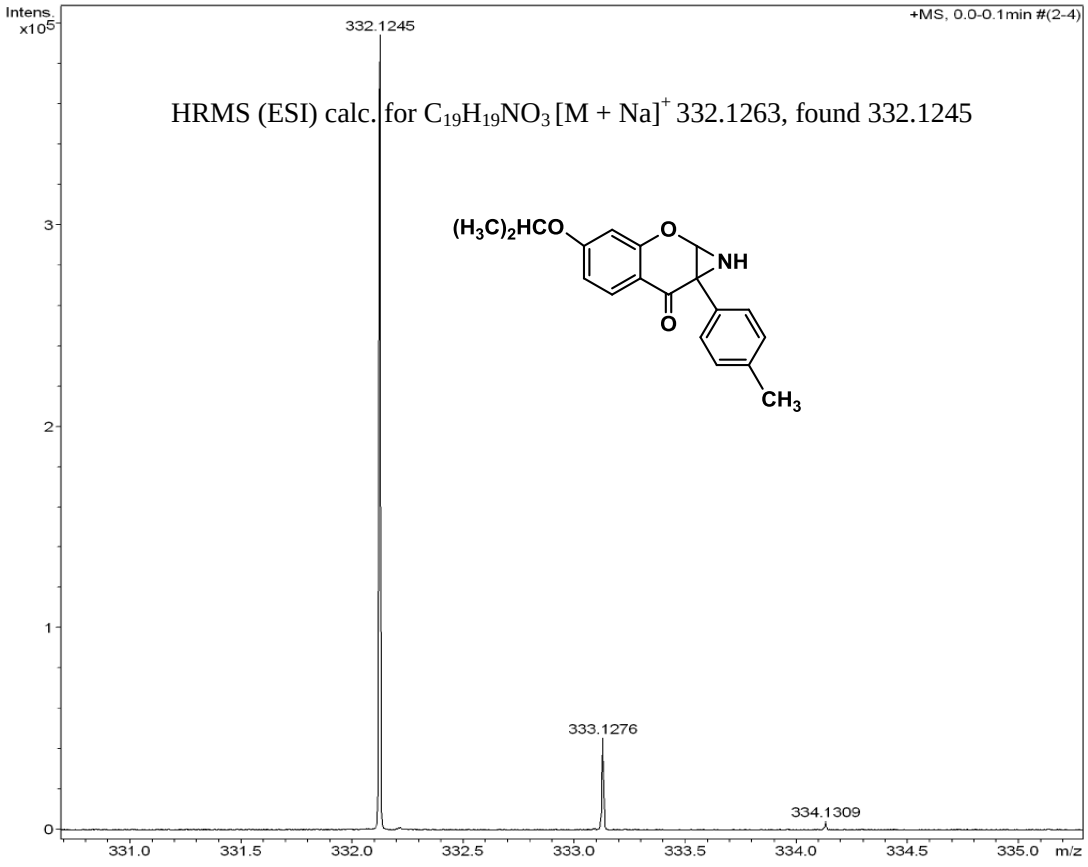
Compound 1c HR MS(CH_3OH)



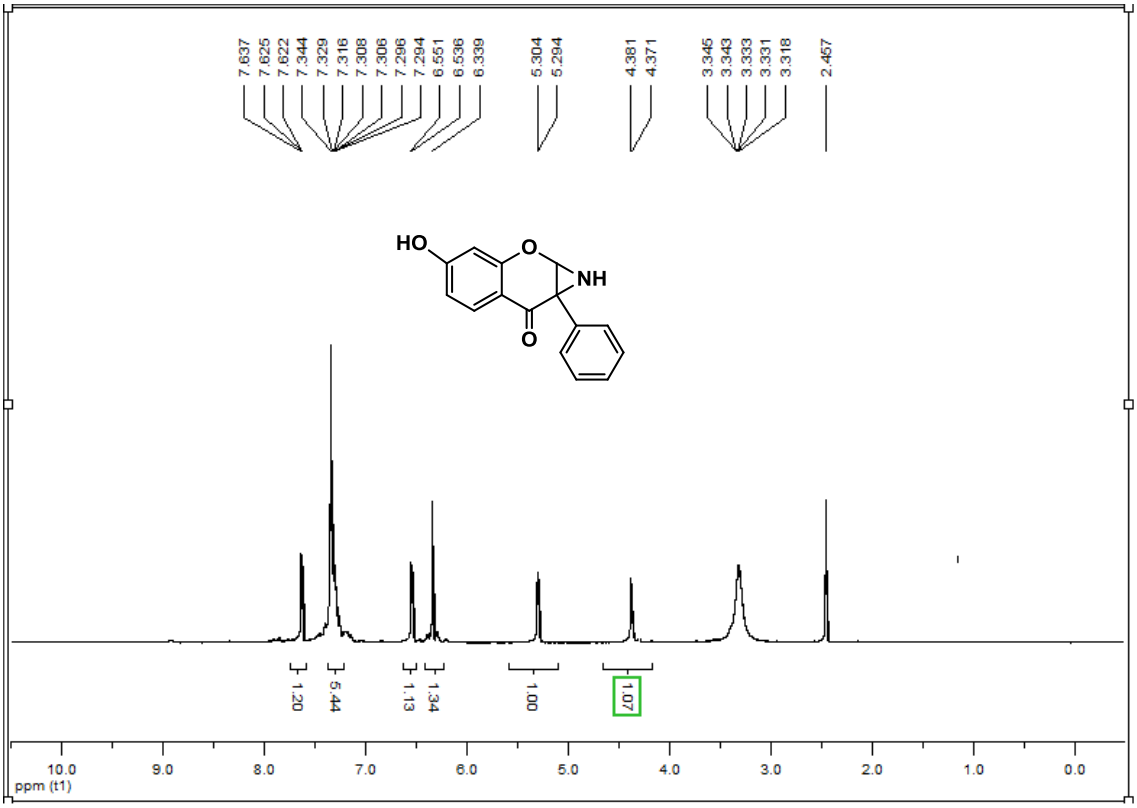
Compound 1d ^{1}H NMR(DMSO-d_6)



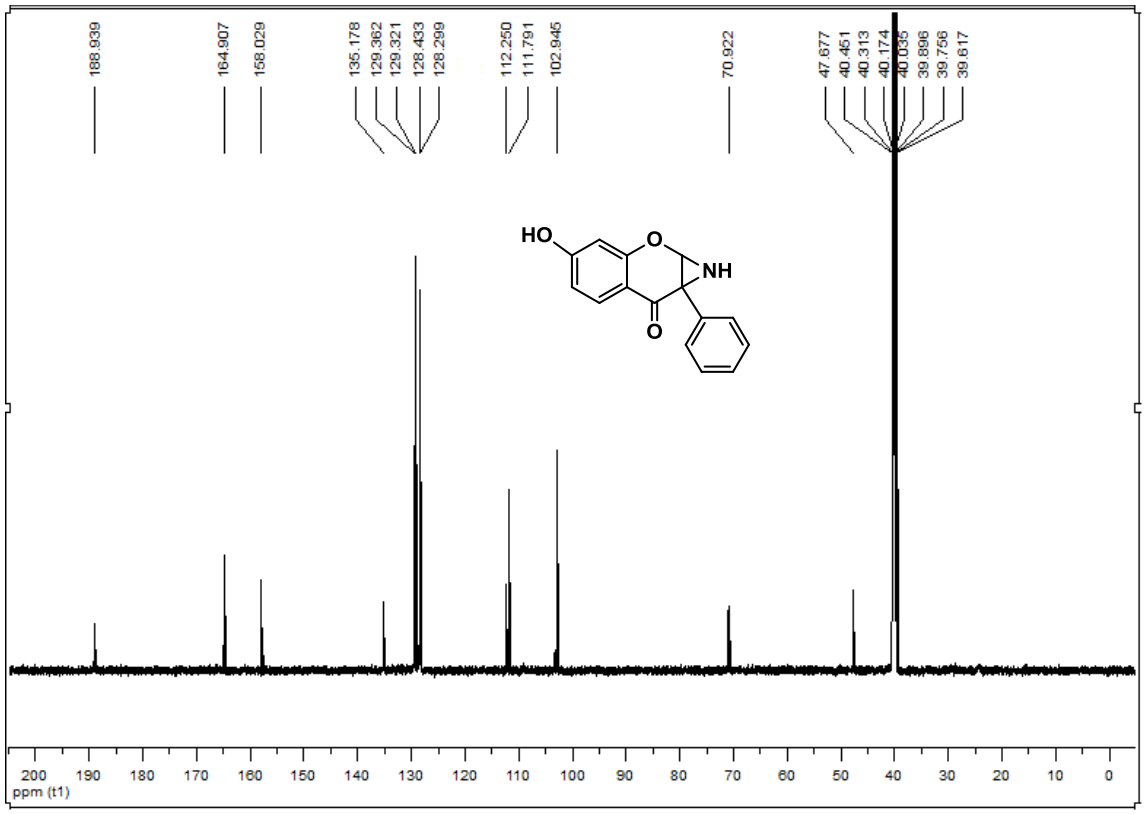
Compound 1d ^{13}C NMR(DMSO-d_6)



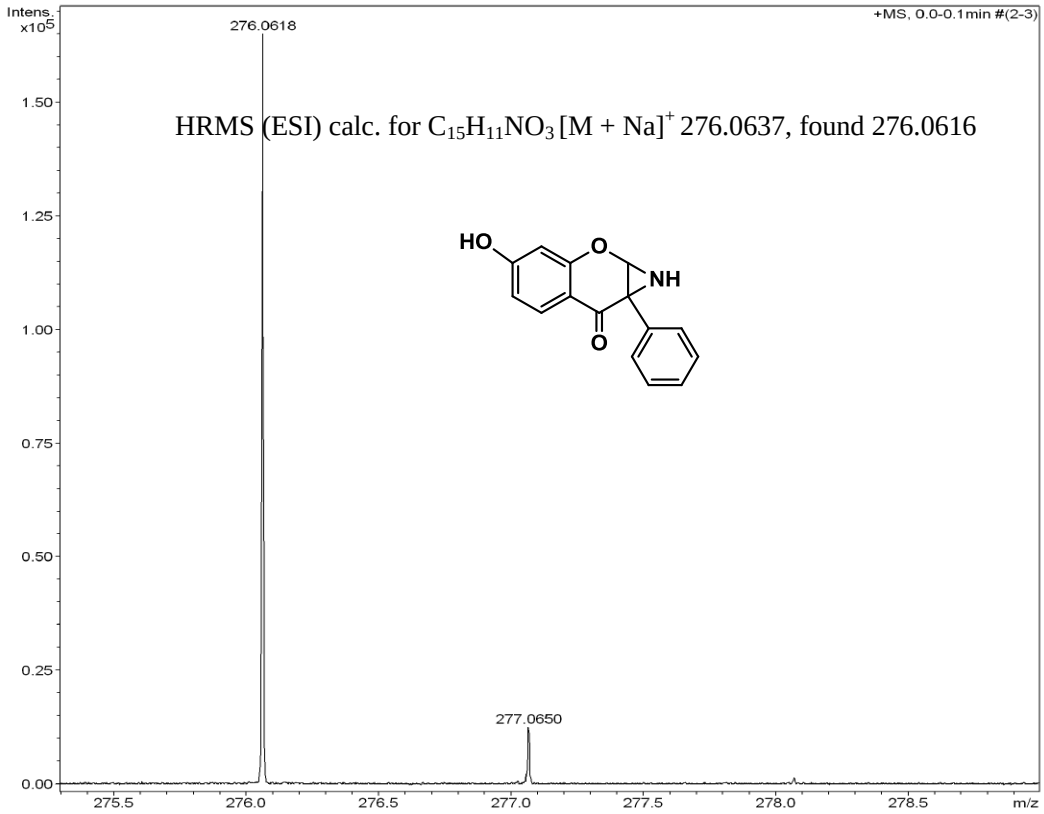
Compound 1d HR MS(CH_3OH)



Compound 1e ^{1}H NMR(DMSO-d_6)

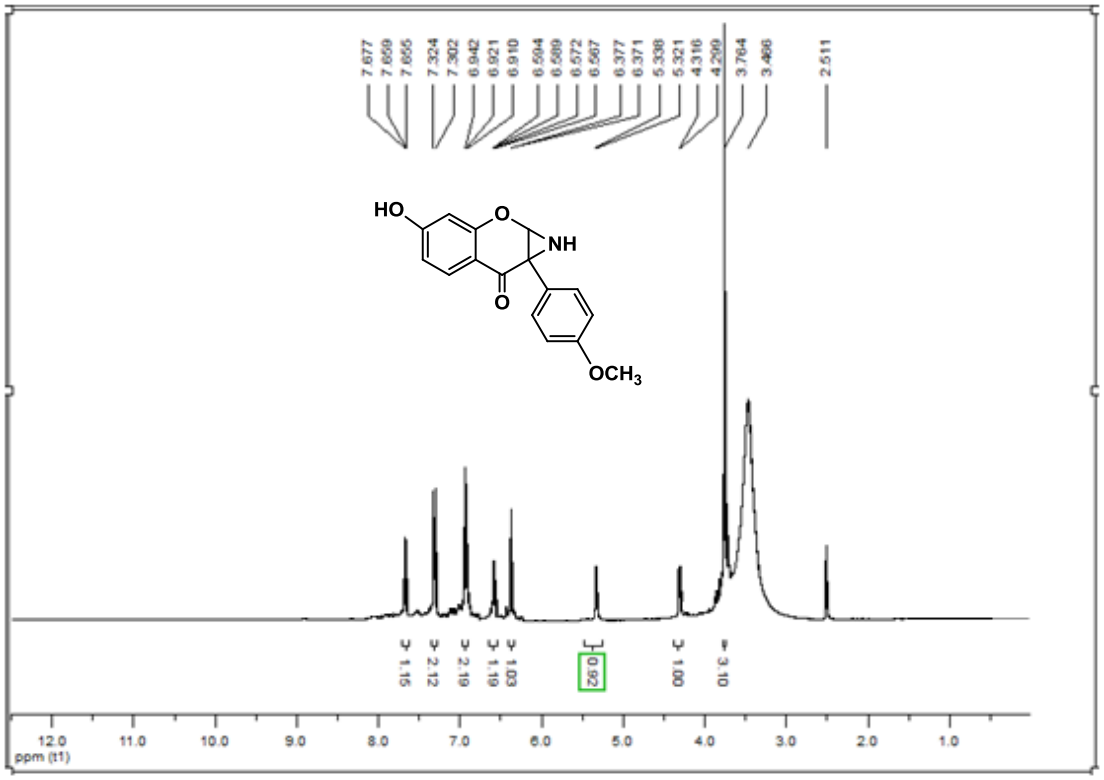


Compound 1e ^{13}C NMR(DMSO-d_6)

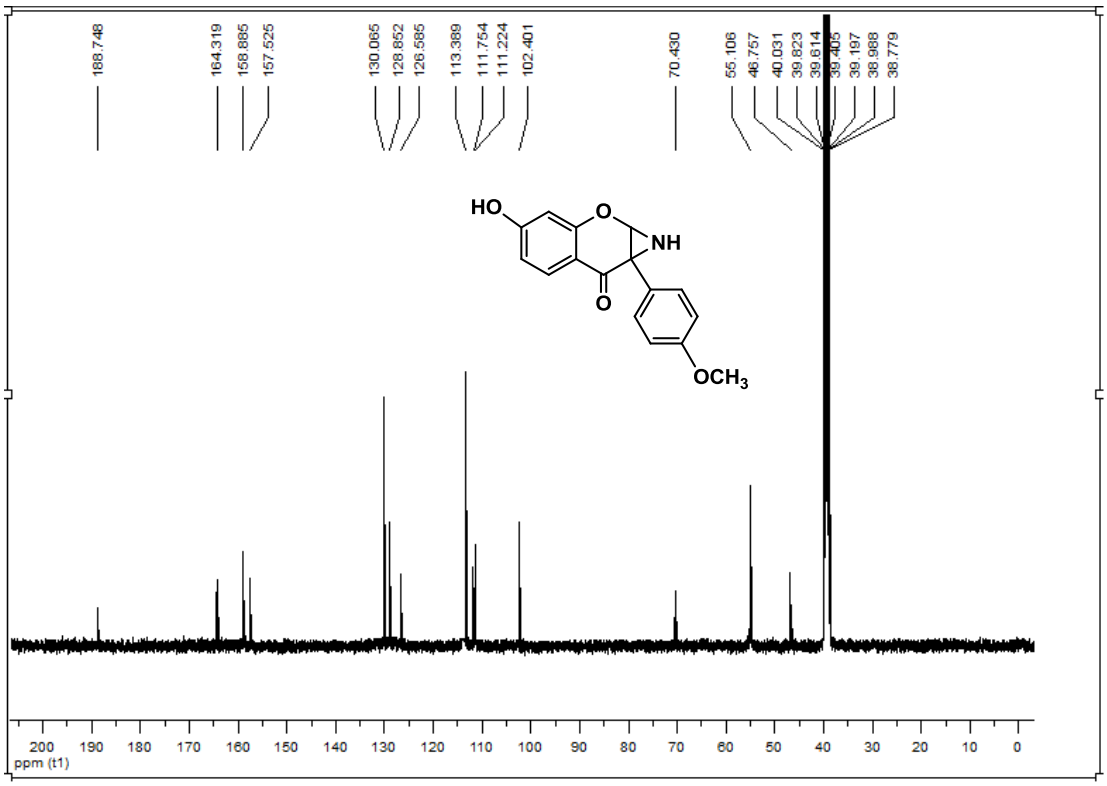


HRMS (ESI) calc. for $C_{15}H_{11}NO_3$ $[M + Na]^+$ 276.0637, found 276.0616

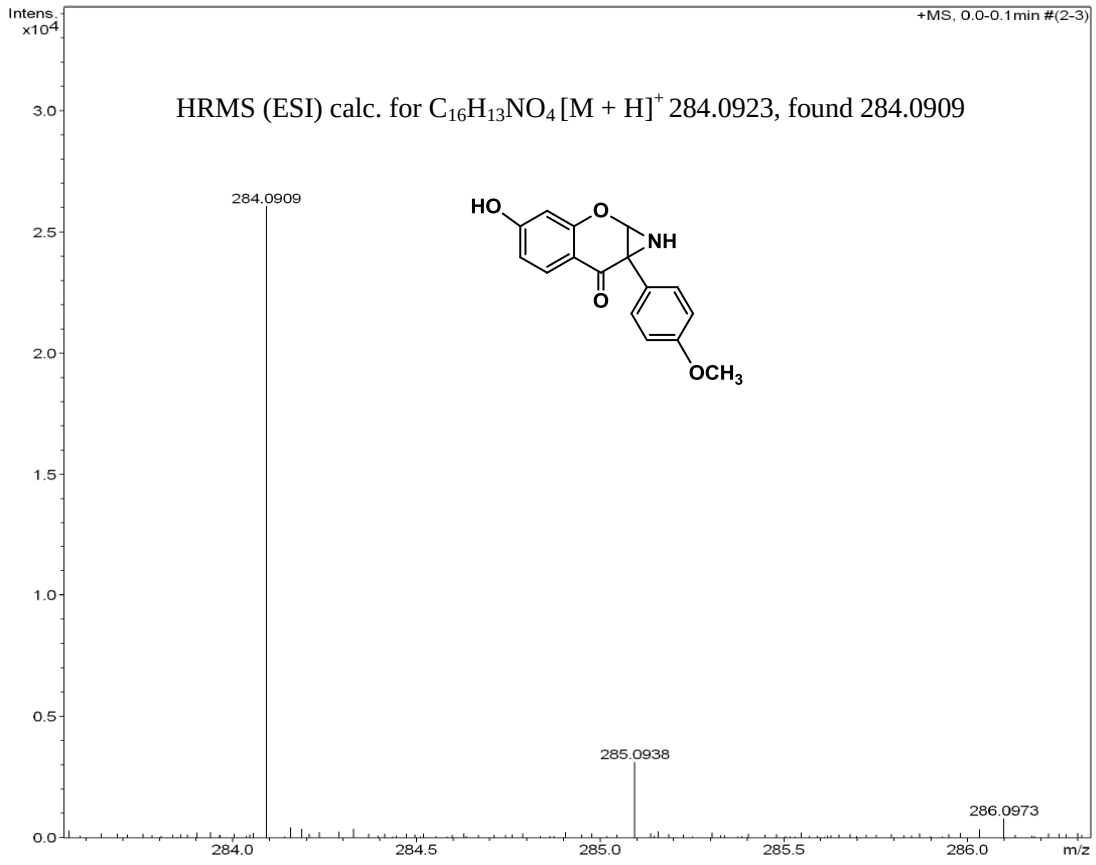
Compound 1e HR MS(CH_3OH)



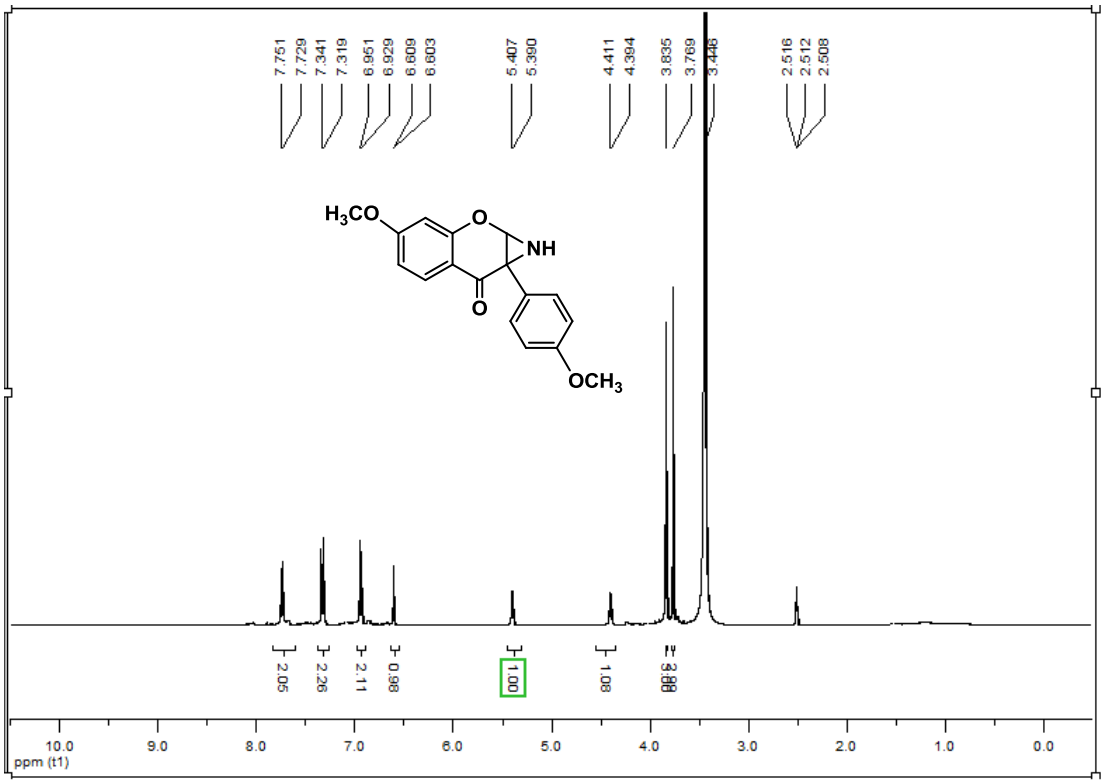
Compound 1f ^{1}H NMR(DMSO-d_6)



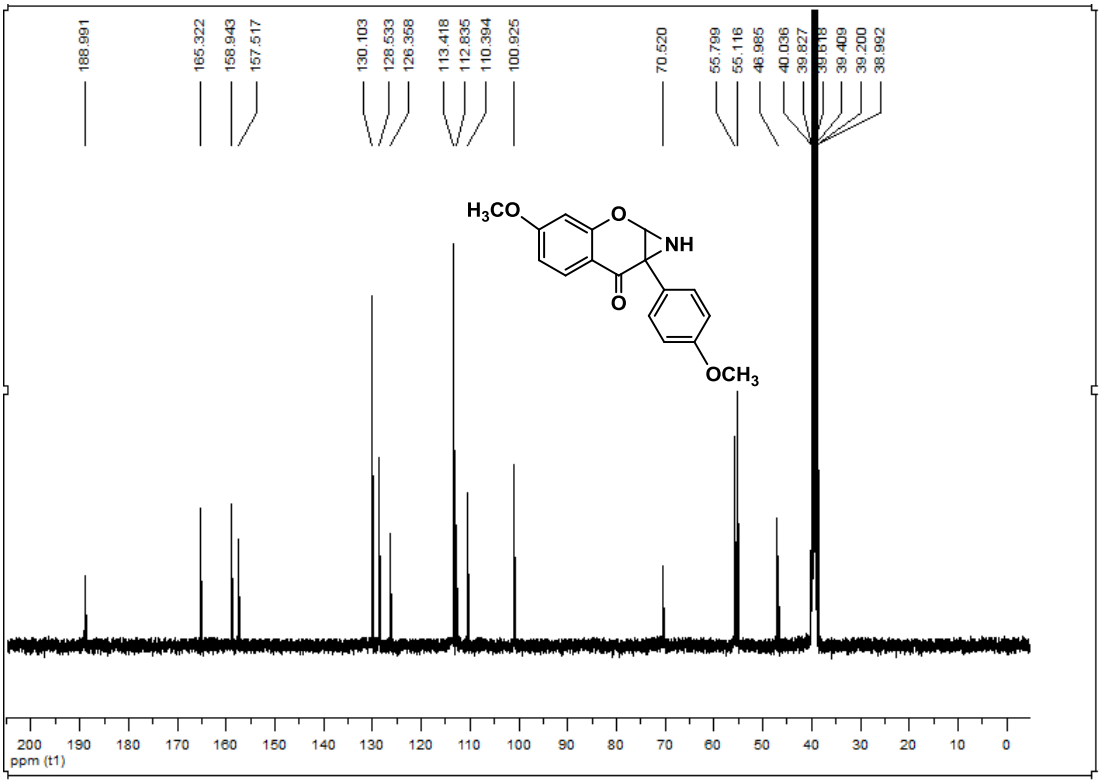
Compound 1f ^{13}C NMR(DMSO-d_6)



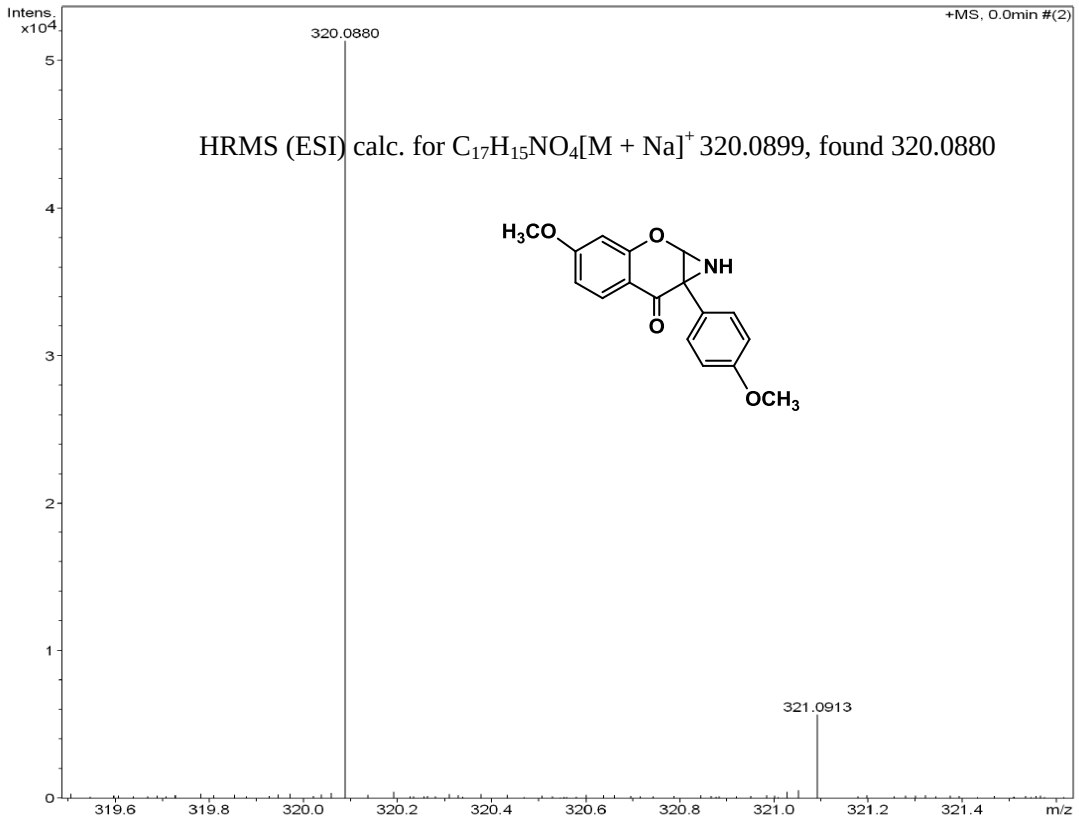
Compound 1f HR MS(CH_3OH)



Compound 1g ^{1}H NMR(DMSO-d_6)

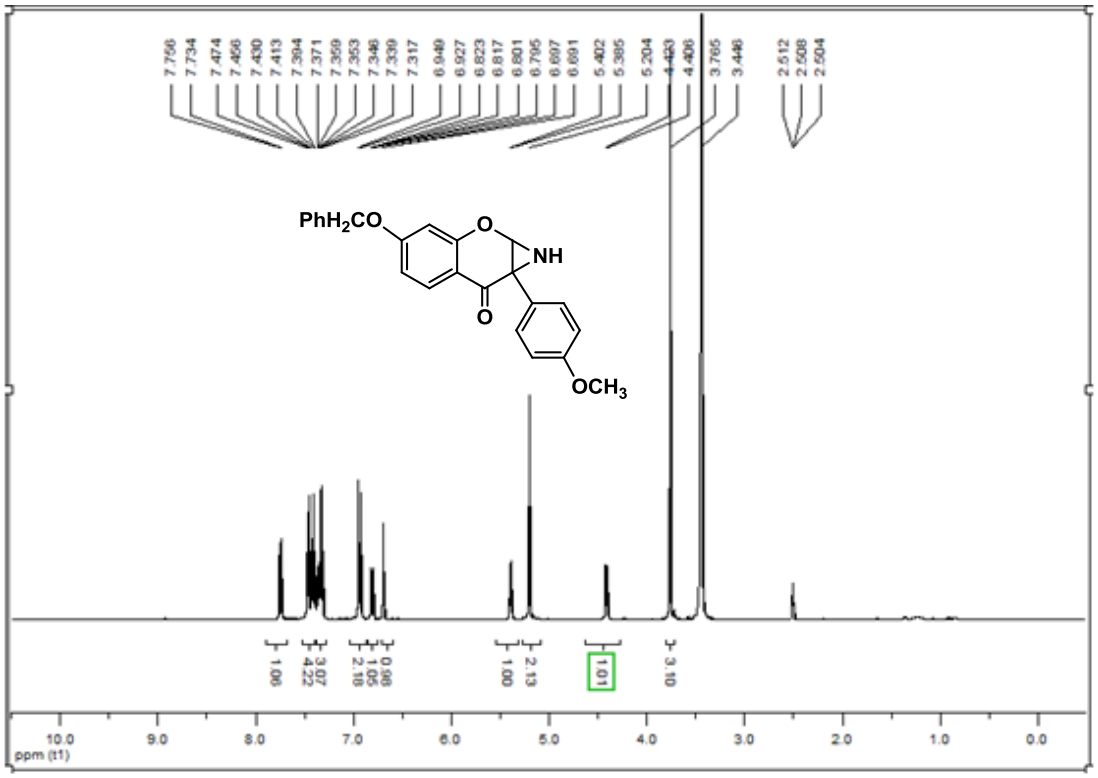


Compound 1g ^{13}C NMR(DMSO-d_6)

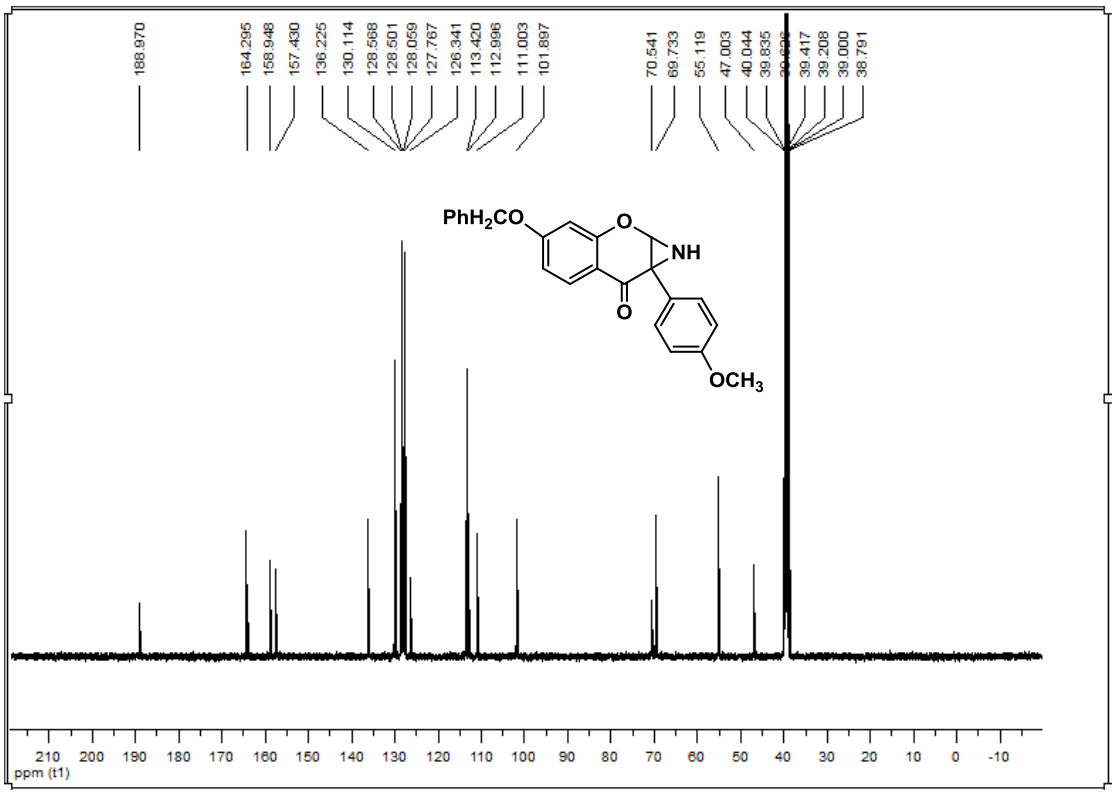


HRMS (ESI) calc. for $C_{17}H_{15}NO_4[M + Na]^+$ 320.0899, found 320.0880

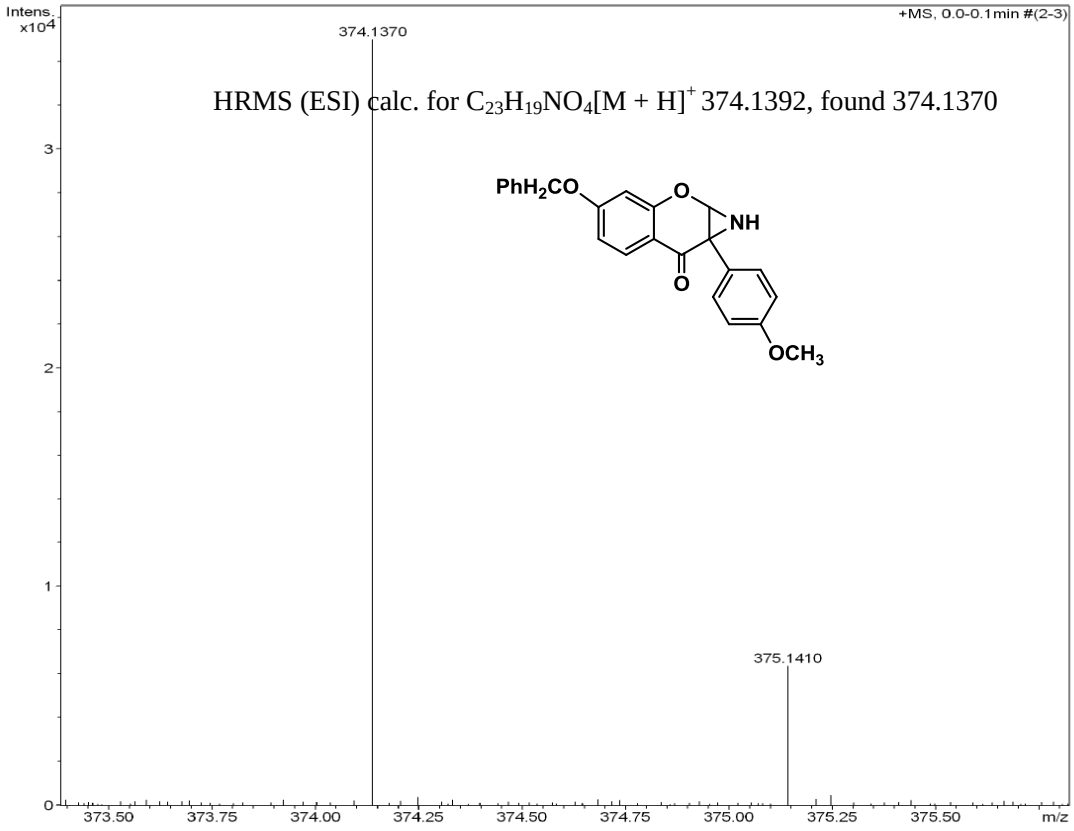
Compound 1g HR MS(CH_3OH)



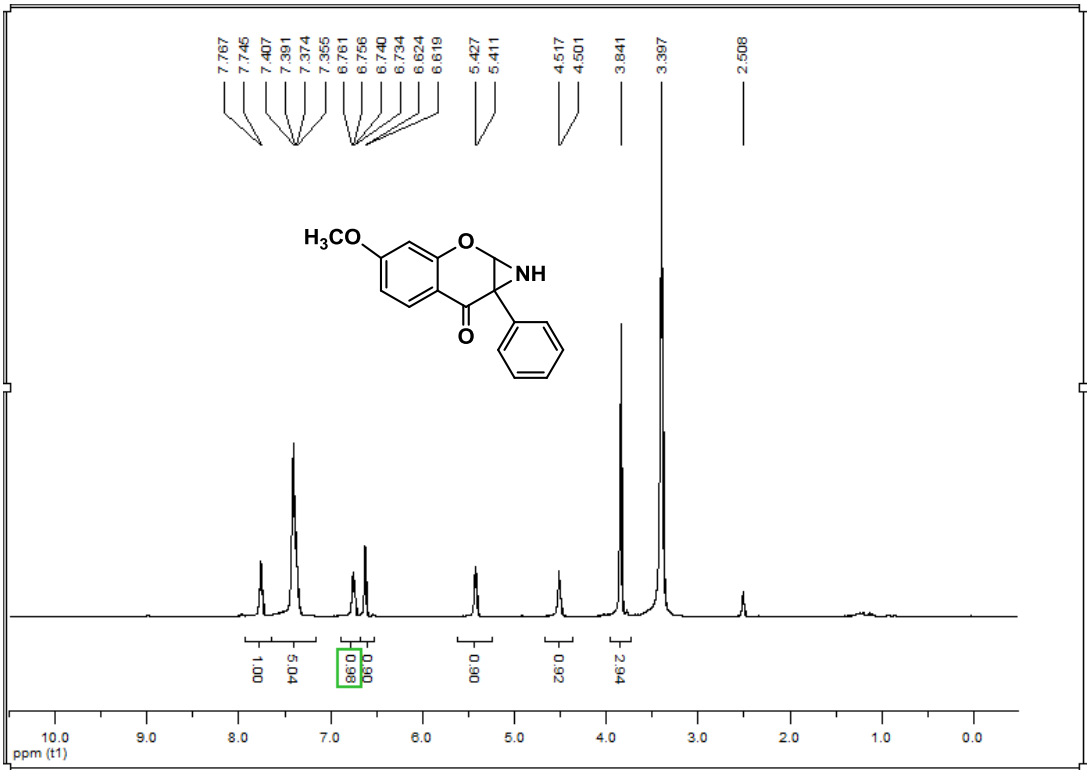
Compound 1h ^{1}H NMR(DMSO-d_6)



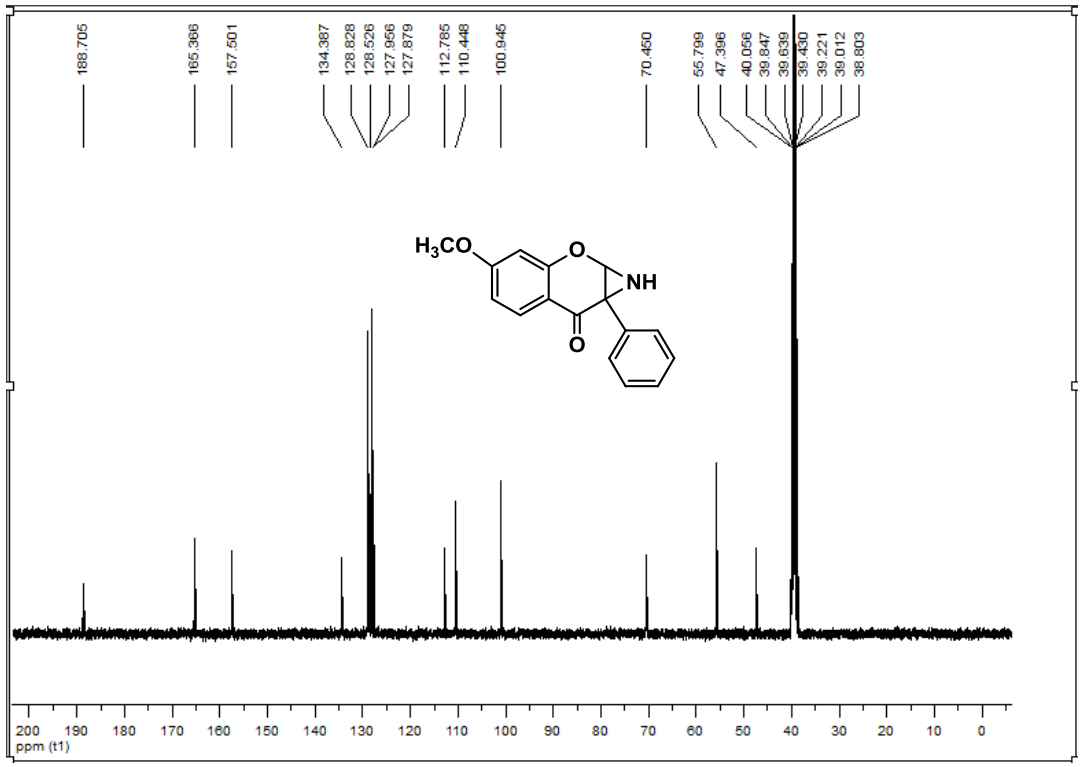
Compound 1h ^{13}C NMR(DMSO-d_6)



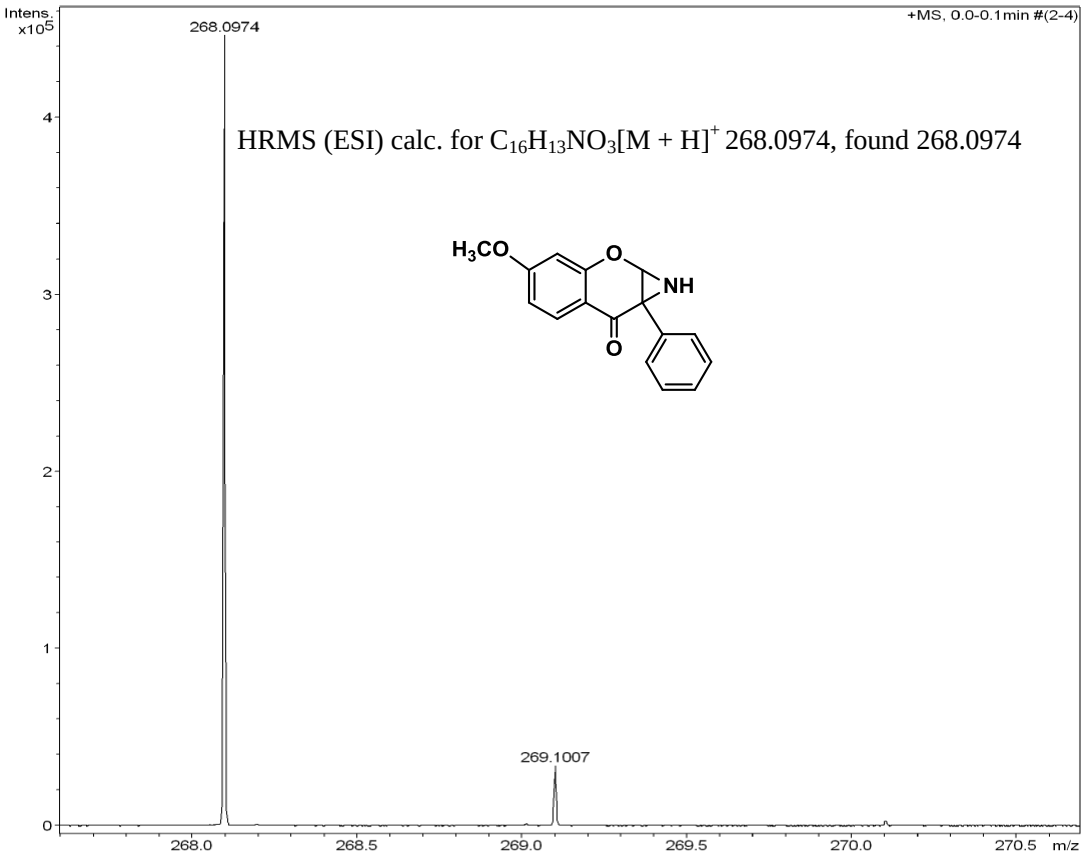
Compound 1h HR MS(CH_3OH)



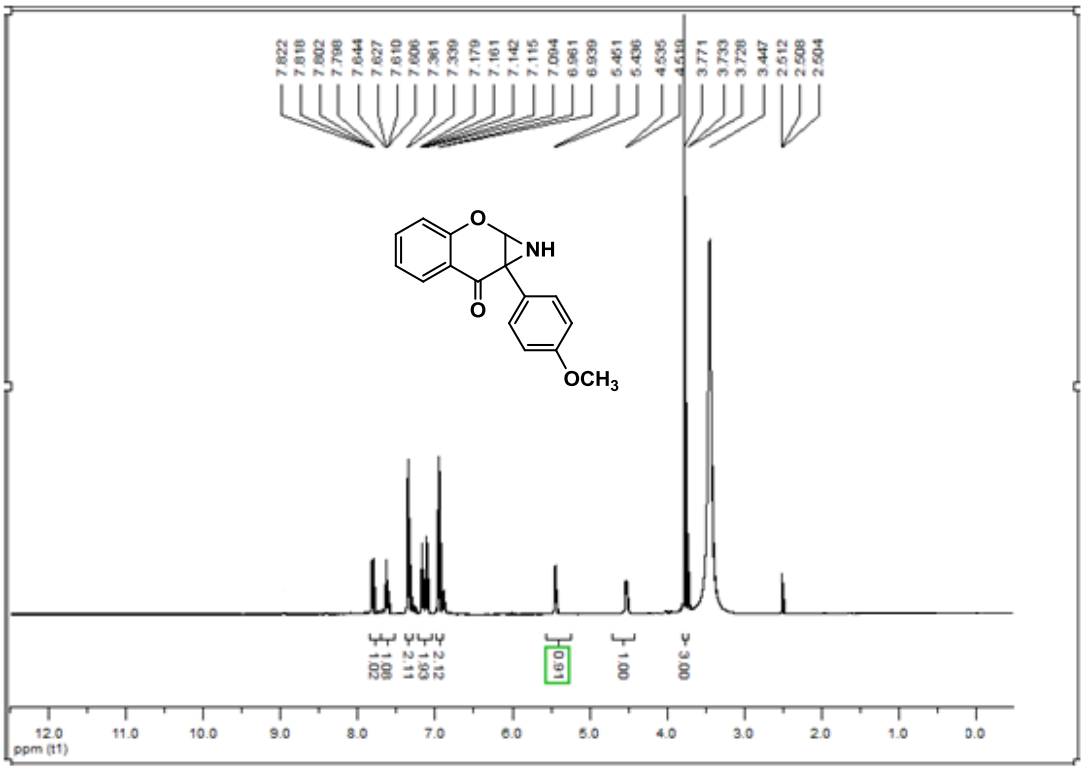
Compound 1i ^{1}H NMR(DMSO-d_6)



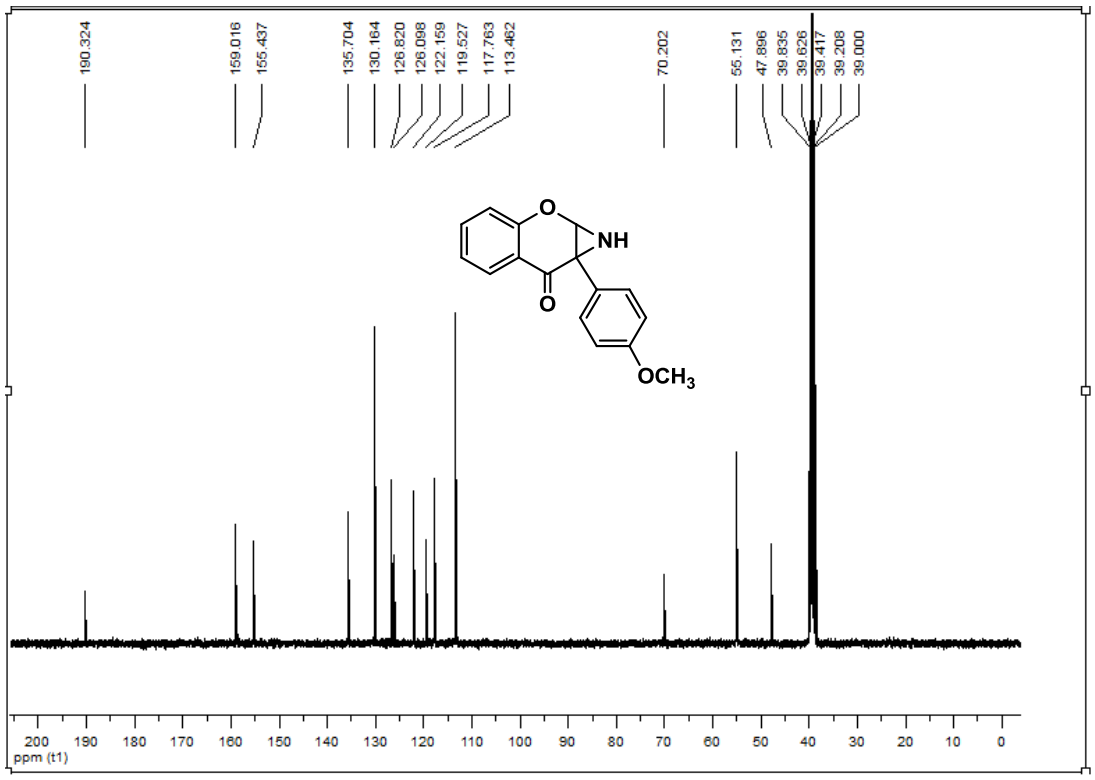
Compound 1i ^{13}C NMR(DMSO-d_6)



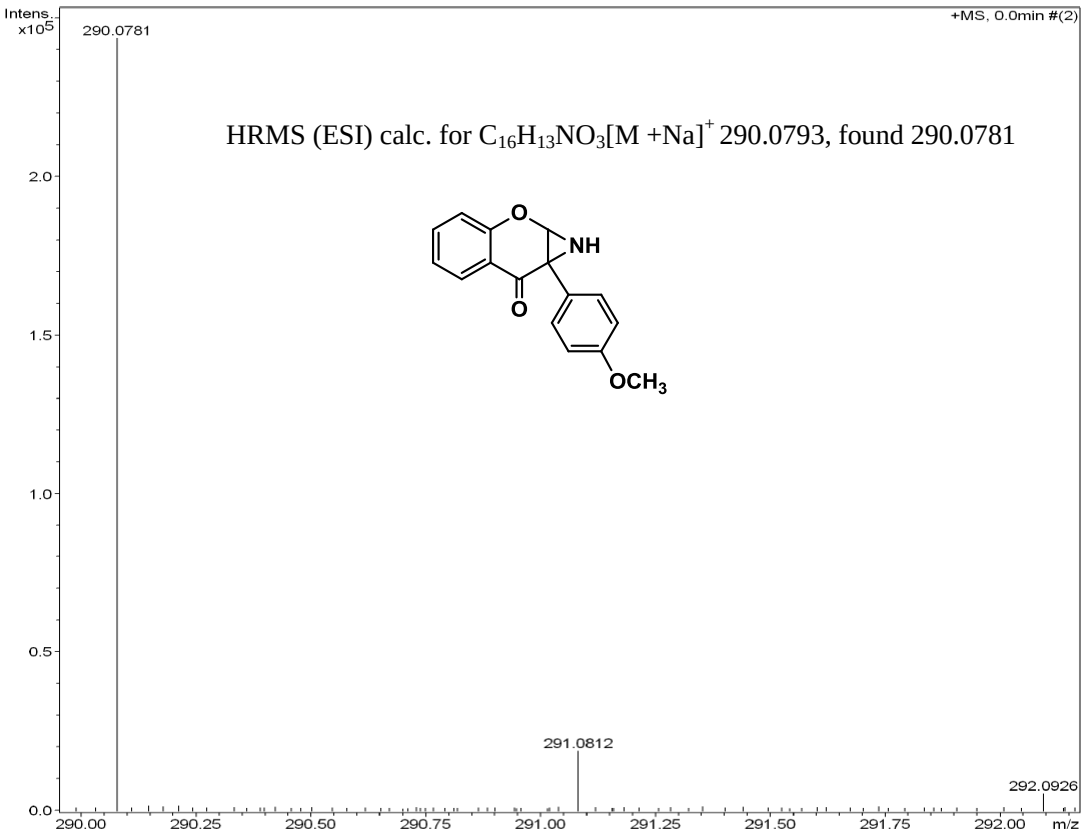
Compound 1i HR MS(CH_3OH)



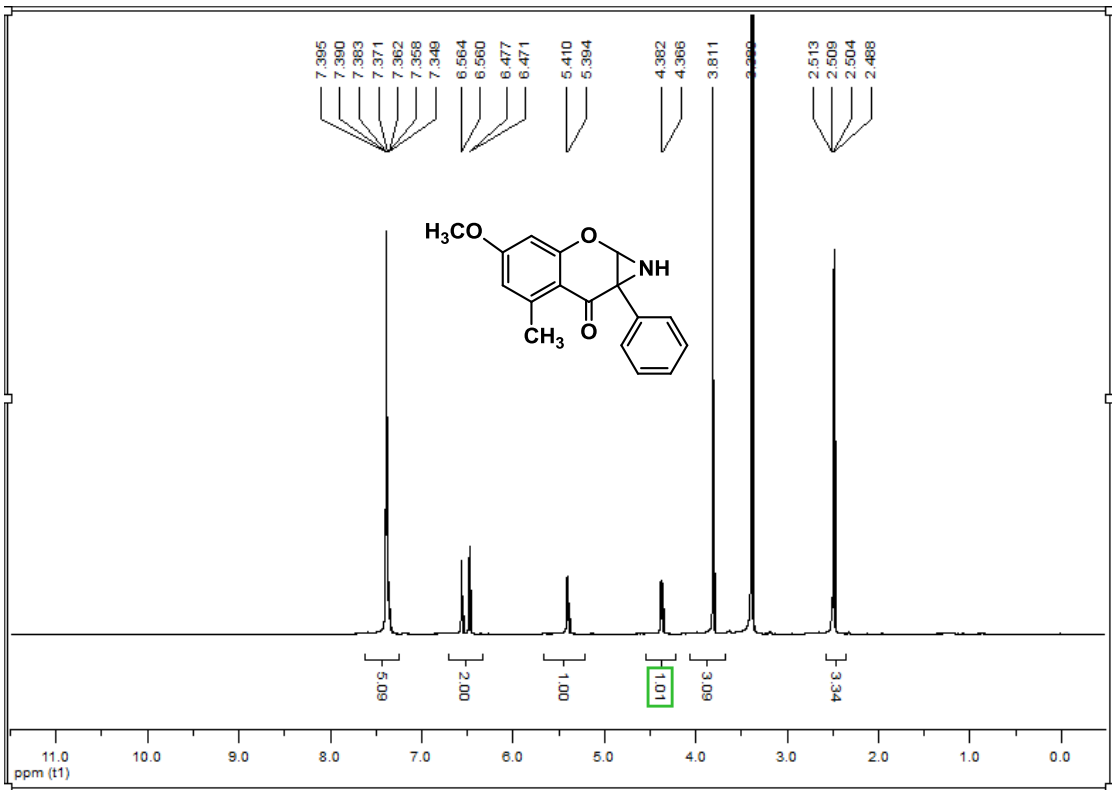
Compound 1j ^{1}H NMR(DMSO-d_6)



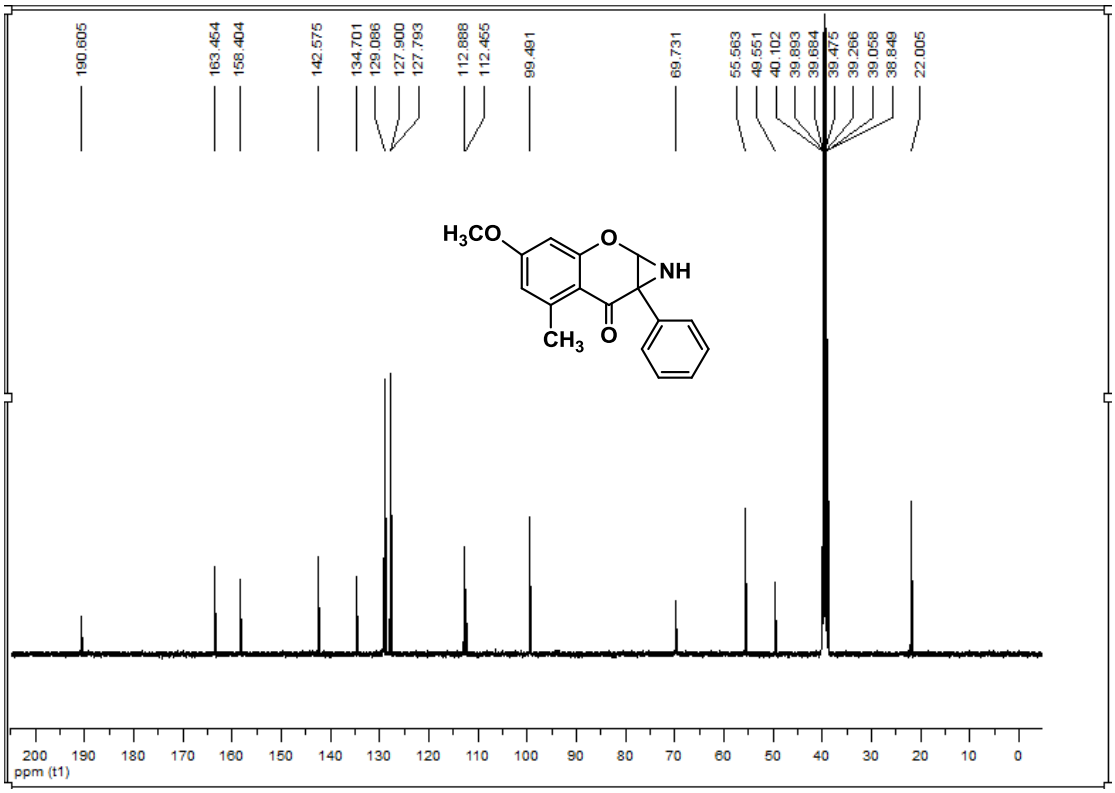
Compound 1j ^{13}C NMR(DMSO-d_6)



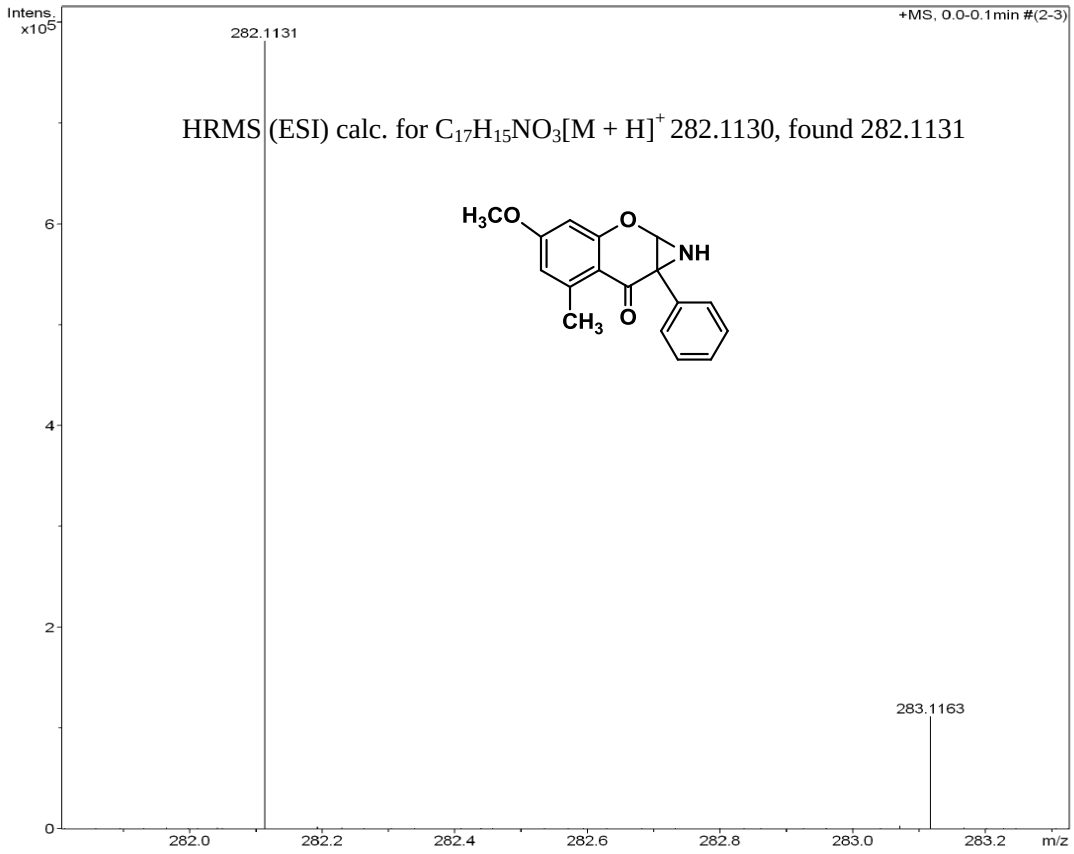
Compound 1j HR MS(CH_3OH)



Compound 1k ^{1}H NMR(DMSO-d_6)

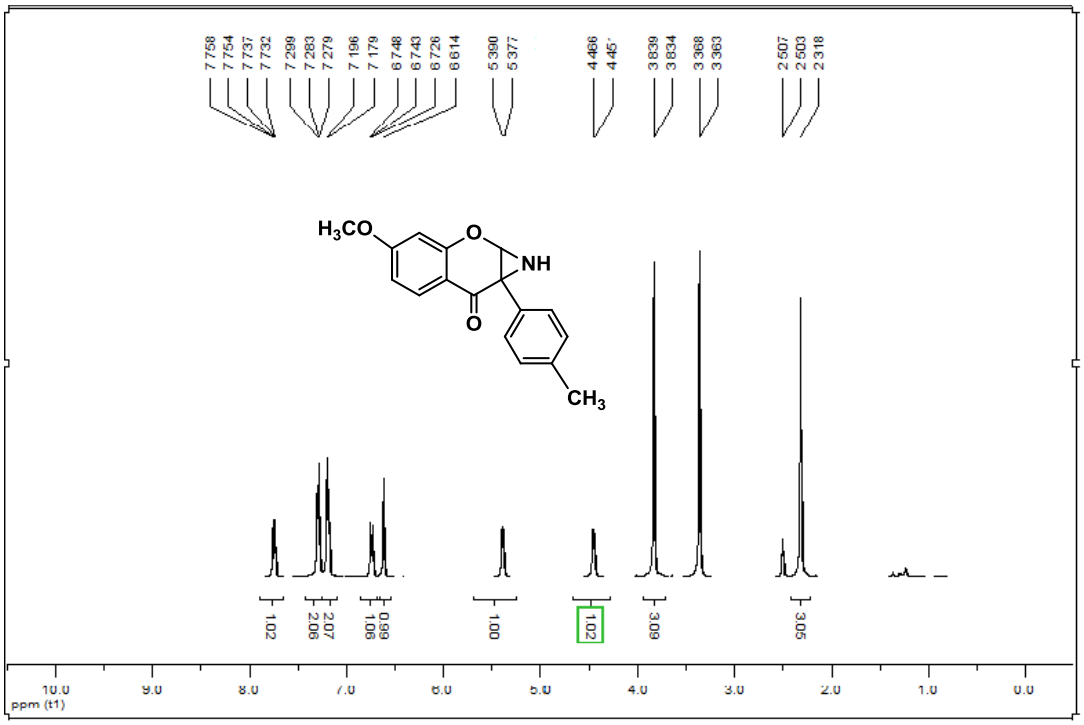


Compound 1k ^{13}C NMR(DMSO-d_6)

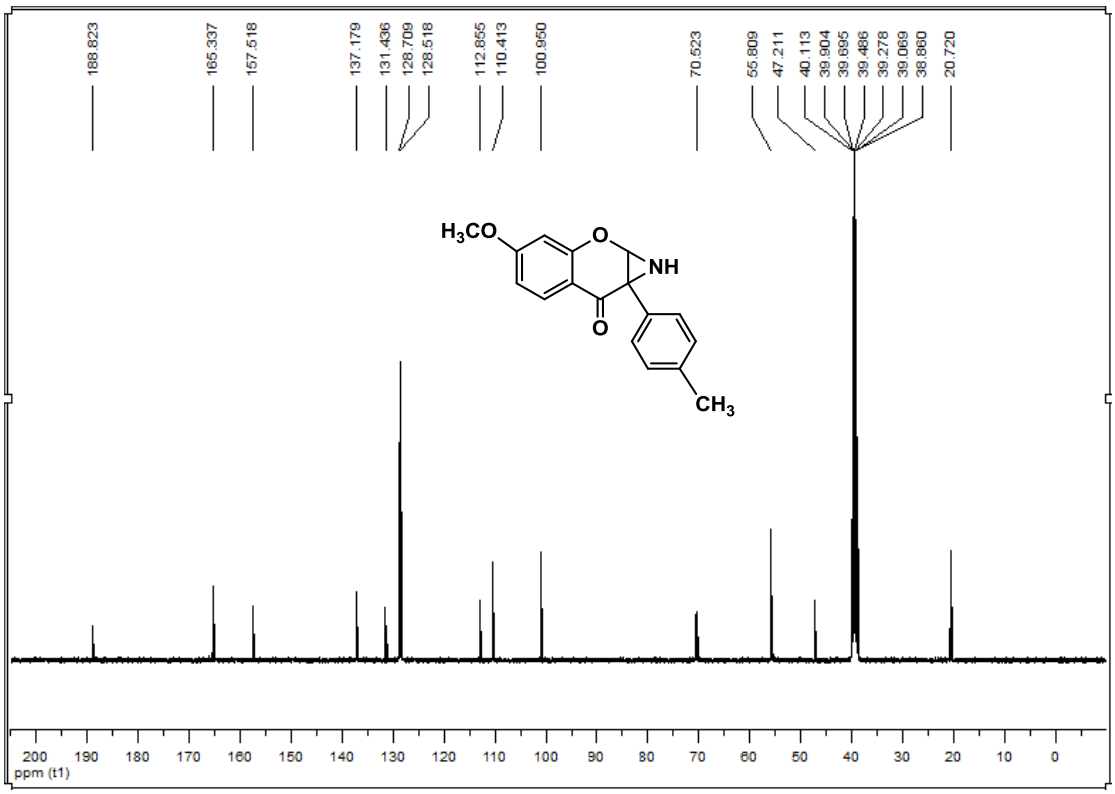


HRMS (ESI) calc. for $C_{17}H_{15}NO_3[M + H]^+$ 282.1130, found 282.1131

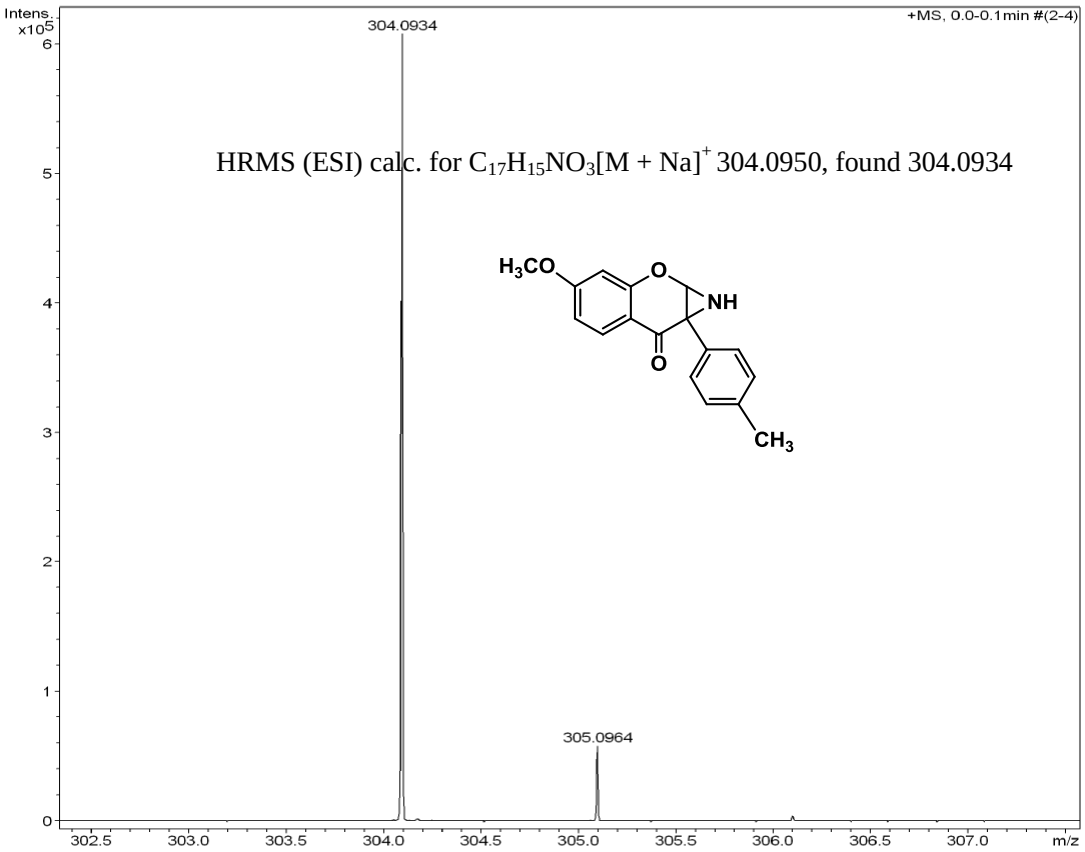
Compound 1k HR MS(CH_3OH)



Compound 1l ^{1}H NMR(DMSO-d_6)

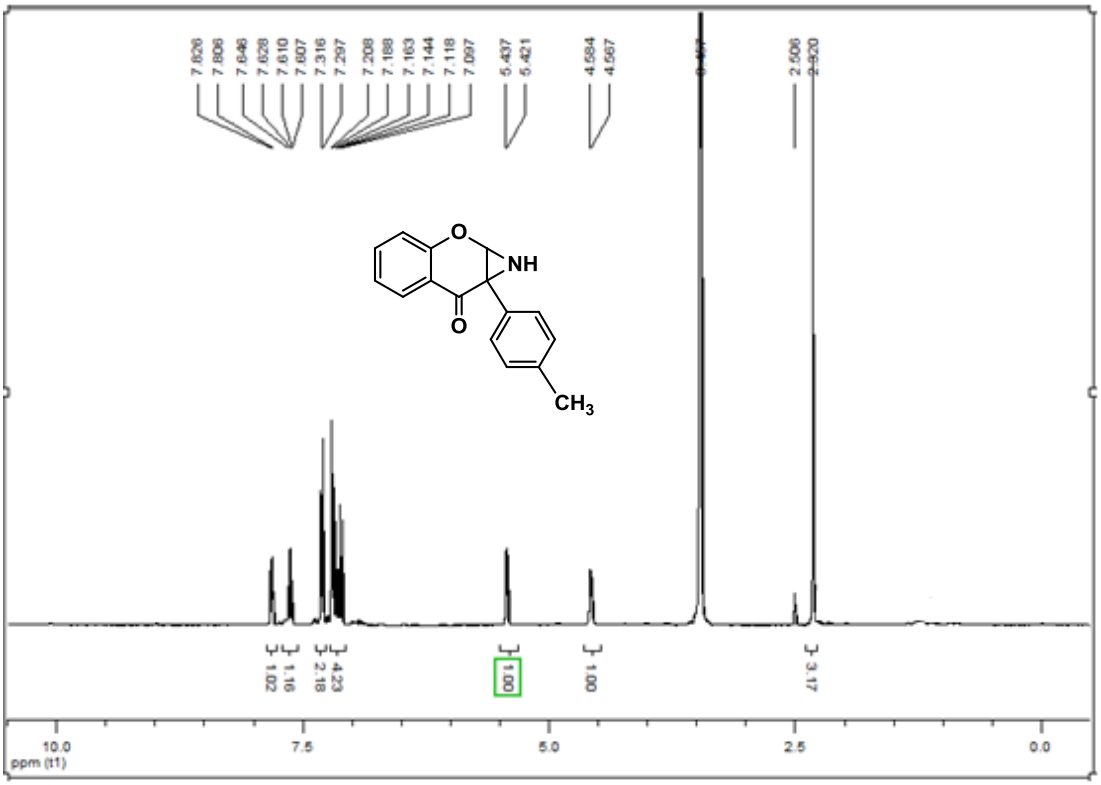


Compound 1l ^{13}C NMR(DMSO-d_6)

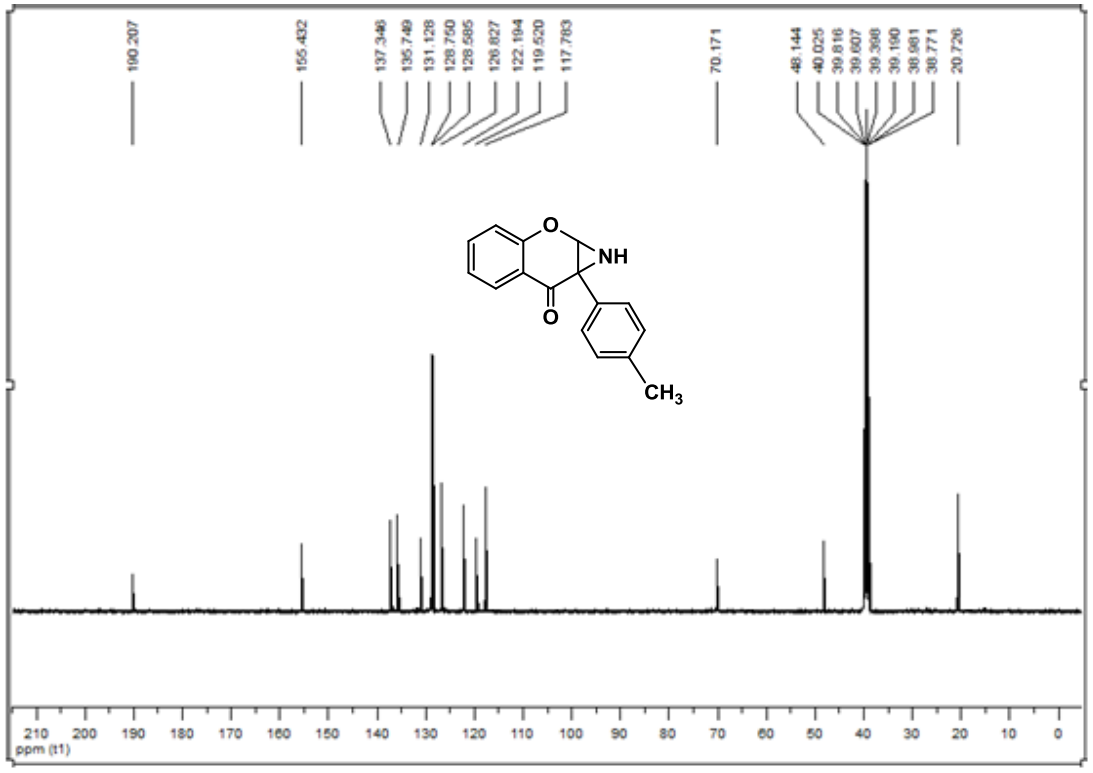


HRMS (ESI) calc. for $C_{17}H_{15}NO_3[M + Na]^+$ 304.0950, found 304.0934

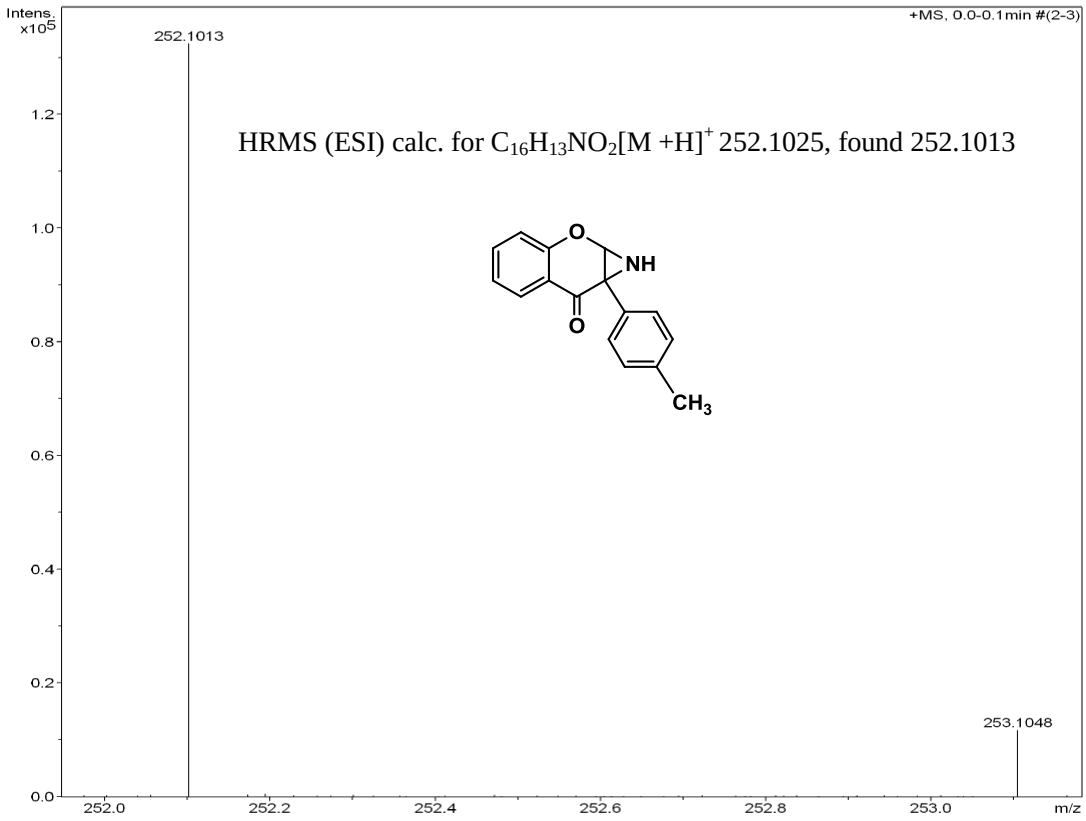
Compound 1l HR MS(CH_3OH)



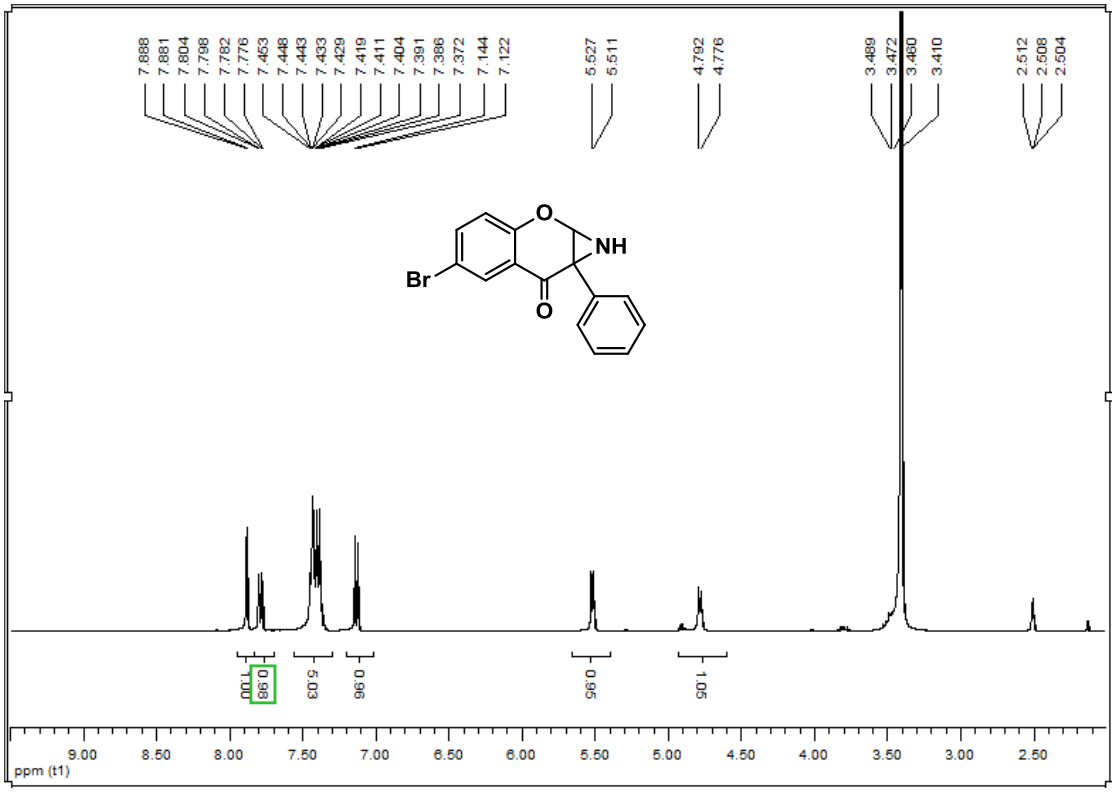
Compound 1m ^{1}H NMR(DMSO-d_6)



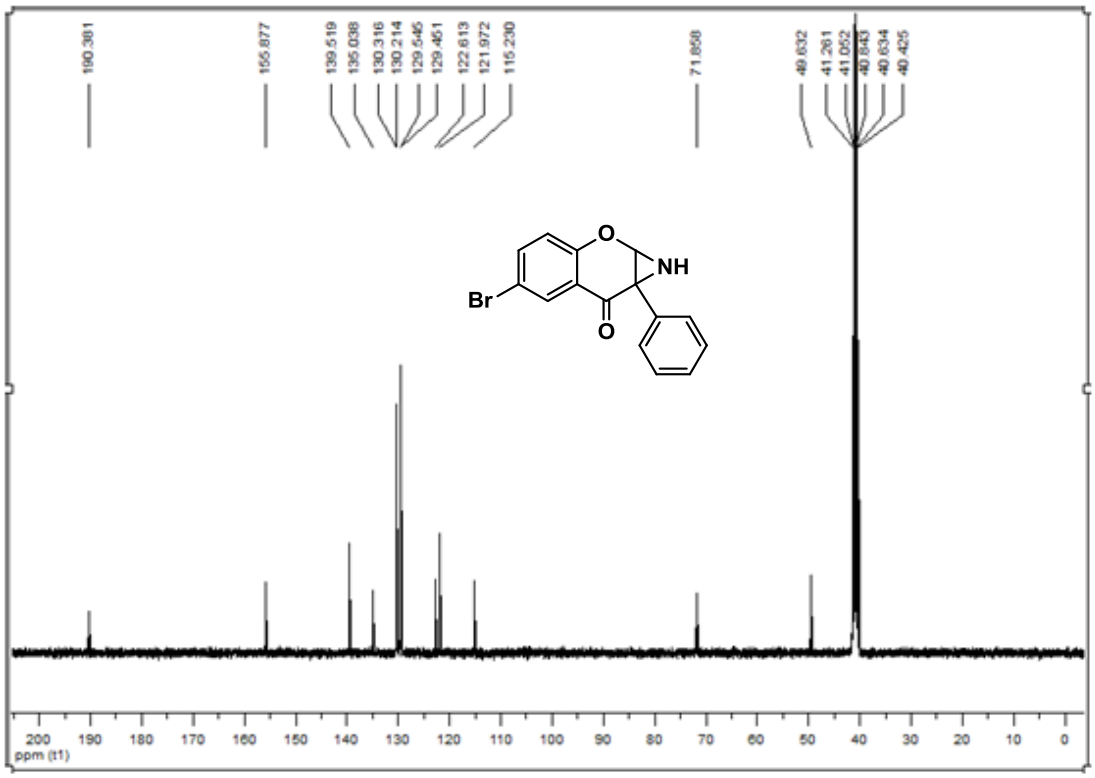
Compound 1m ^{13}C NMR(DMSO-d_6)



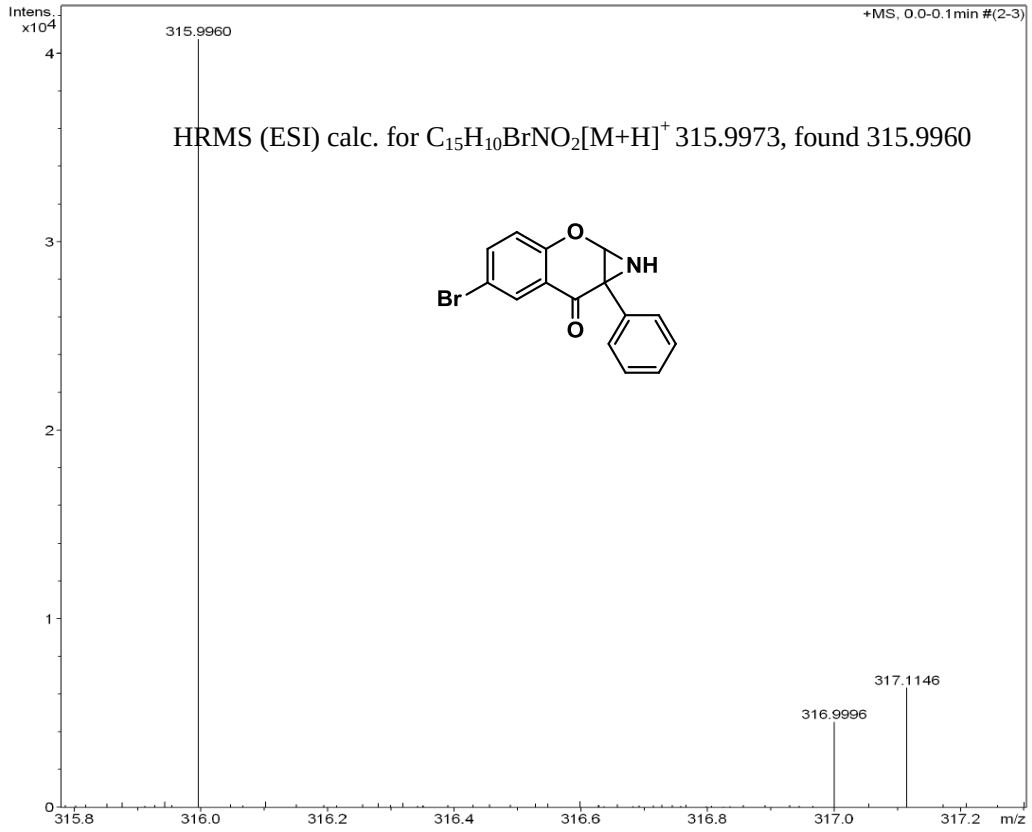
Compound 1m HR MS(CH_3OH)



Compound 1n ^{1}H NMR(DMSO-d_6)

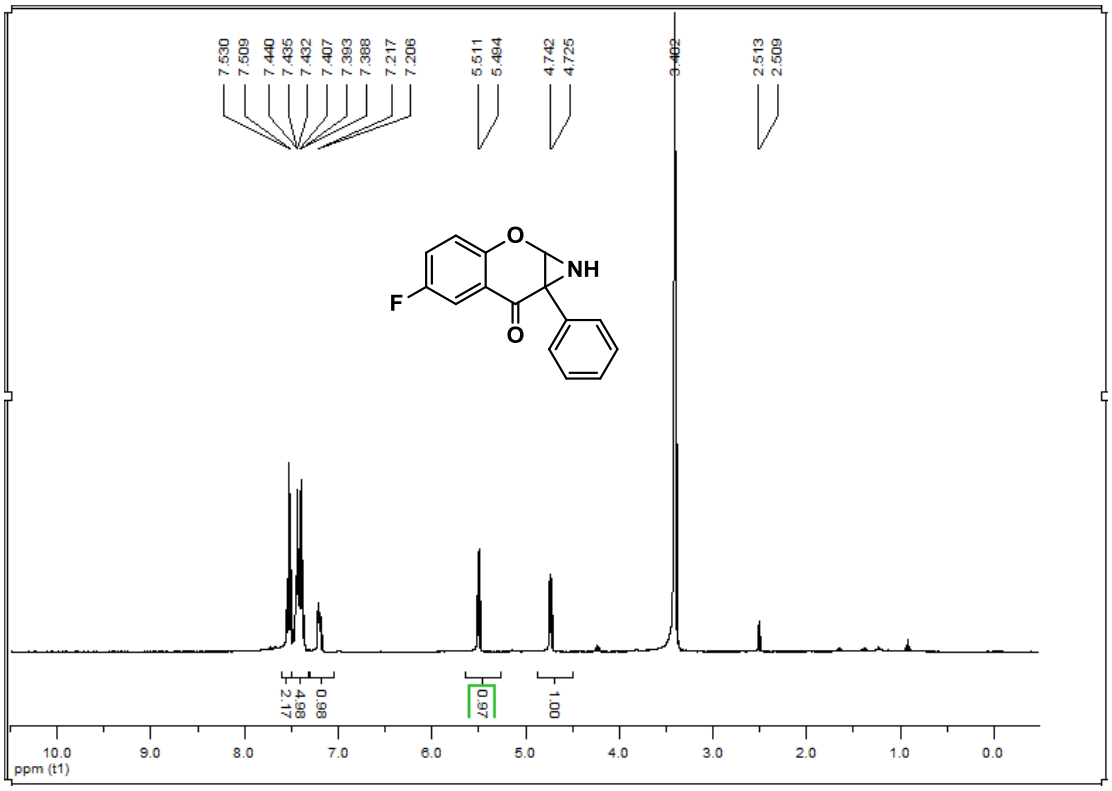


Compound 1n ^{13}C NMR(DMSO-d_6)

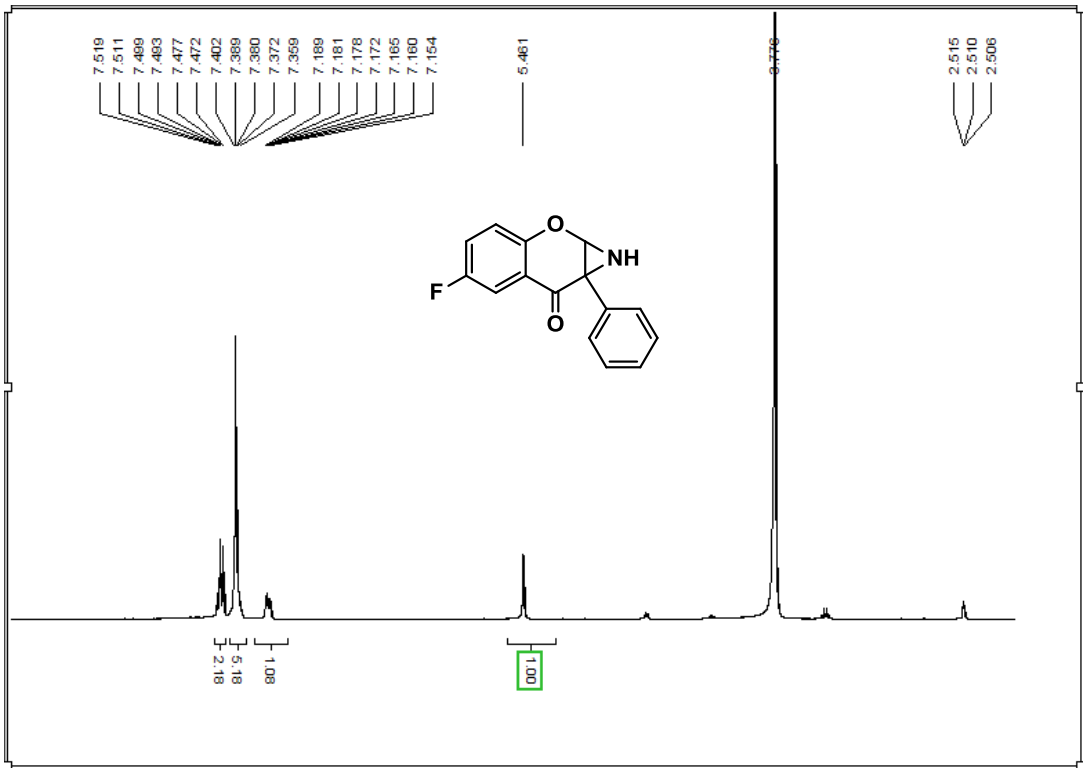


HRMS (ESI) calc. for $C_{15}H_{10}BrNO_2[M+H]^+$ 315.9973, found 315.9960

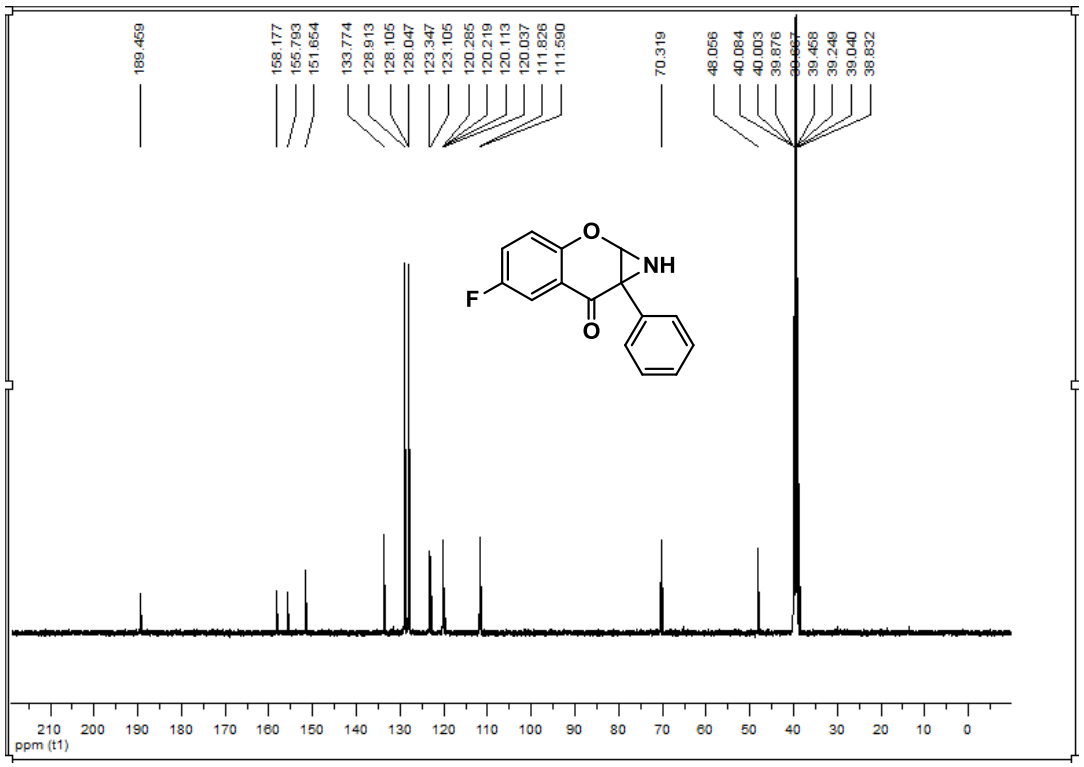
Compound 1n HR MS(CH_3OH)



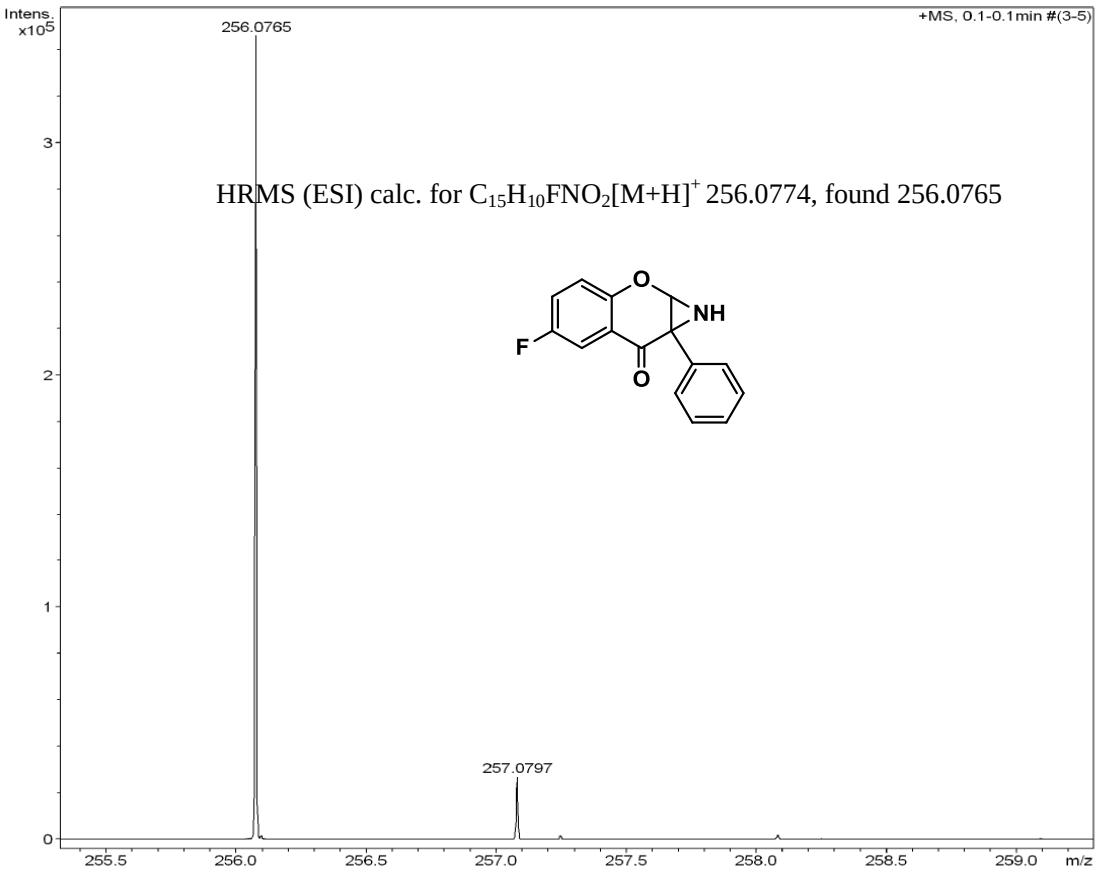
Compound 1o ^{1}H NMR(DMSO-d_6)



Compound 1o ^{1}H NMR(DMSO-d_6+D_2O)

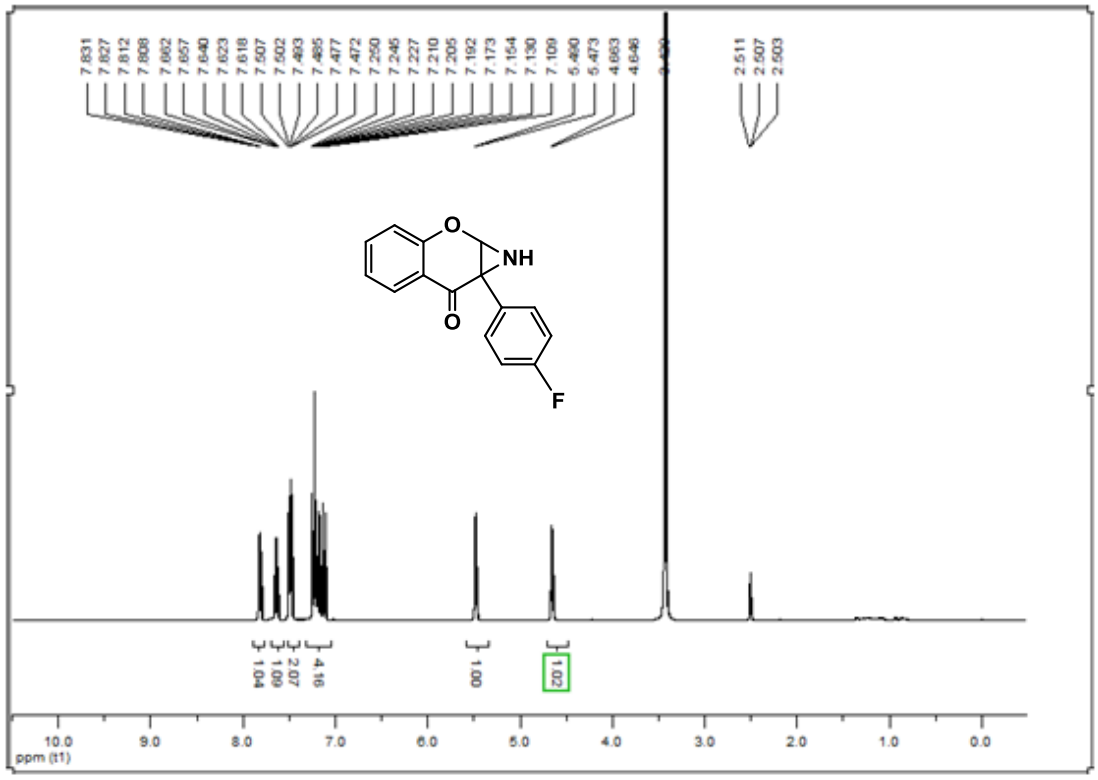


Compound 1o ^{13}C NMR(DMSO-d_6)

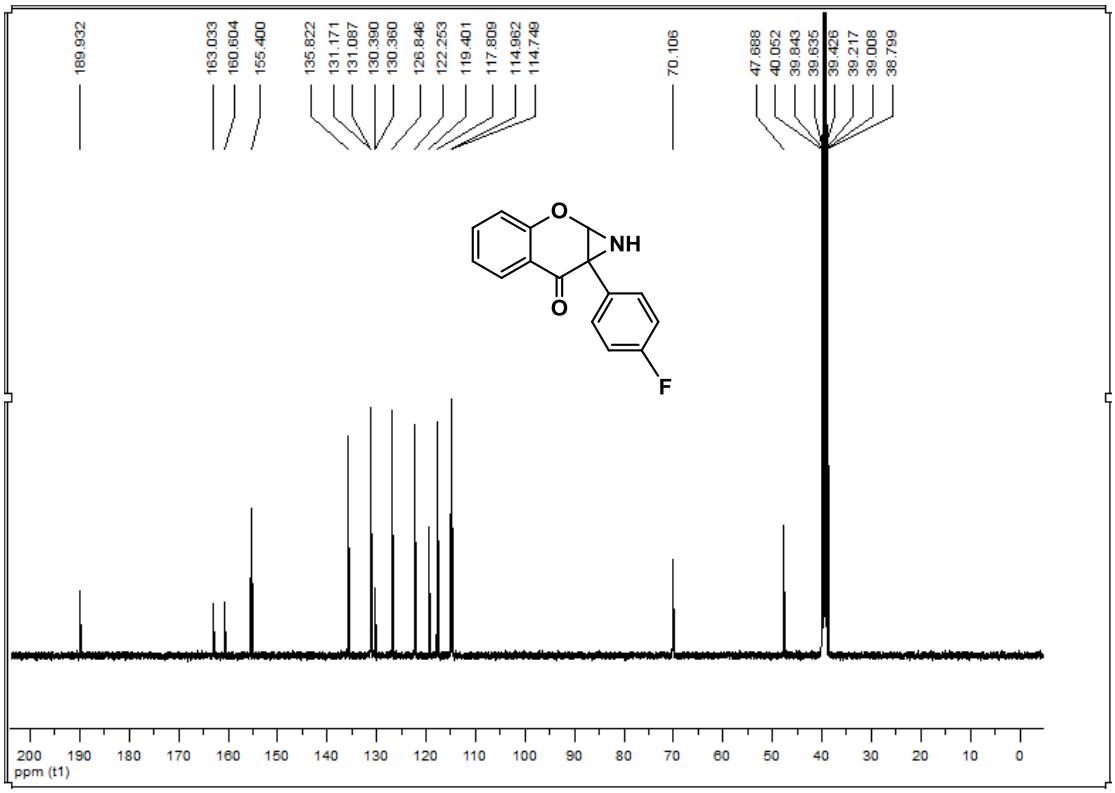


HRMS (ESI) calc. for $C_{15}H_{10}FNO_2[M+H]^+$ 256.0774, found 256.0765

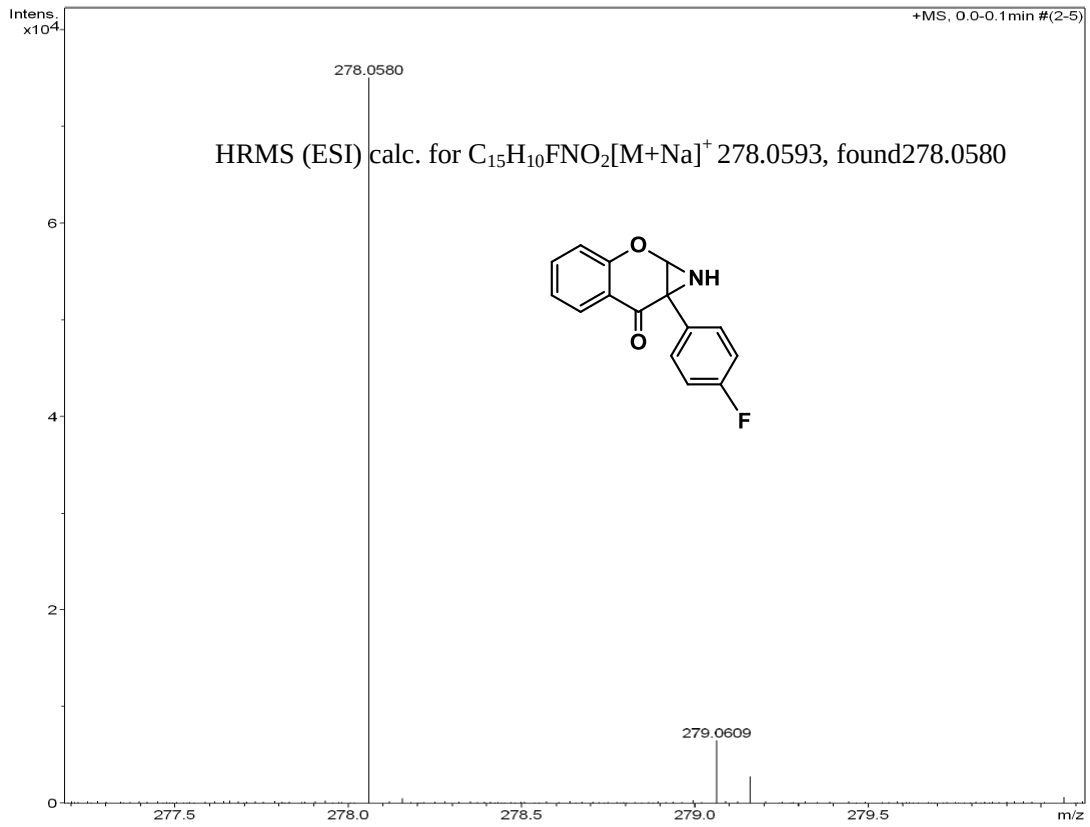
Compound 1o HR MS(CH_3OH)



Compound 1p ^{1}H NMR(DMSO-d_6)

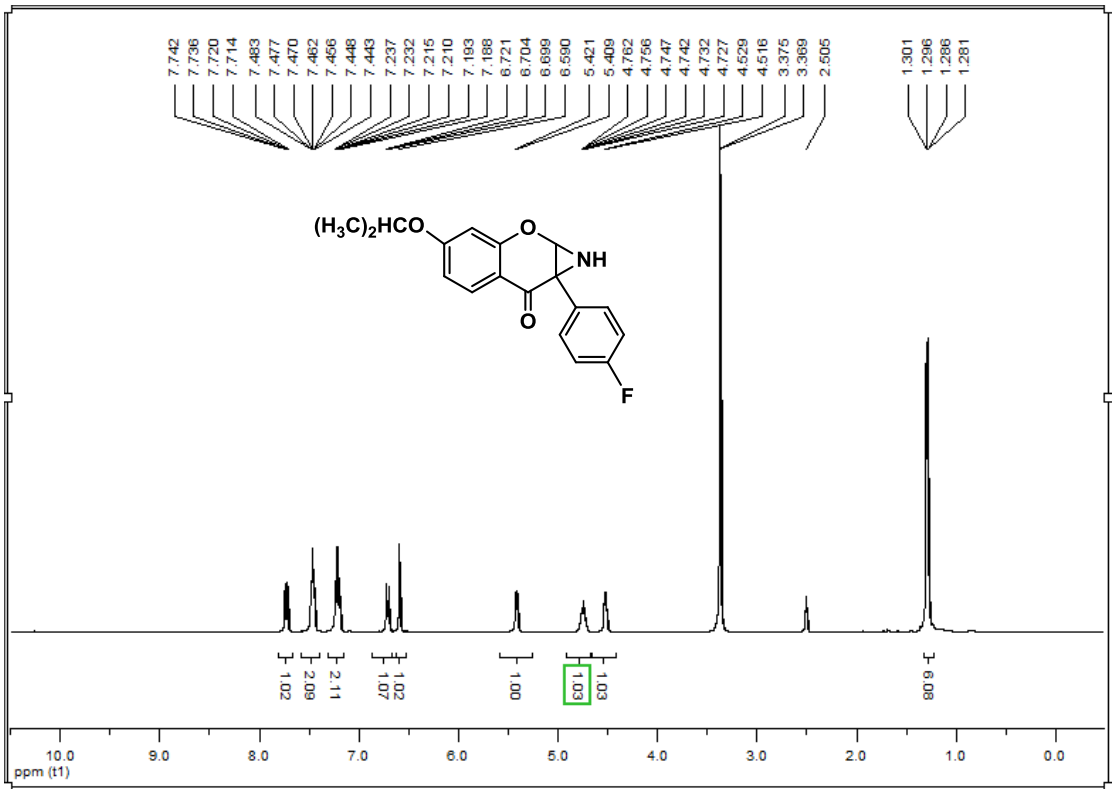


Compound 1p ^{13}C NMR(DMSO-d_6)

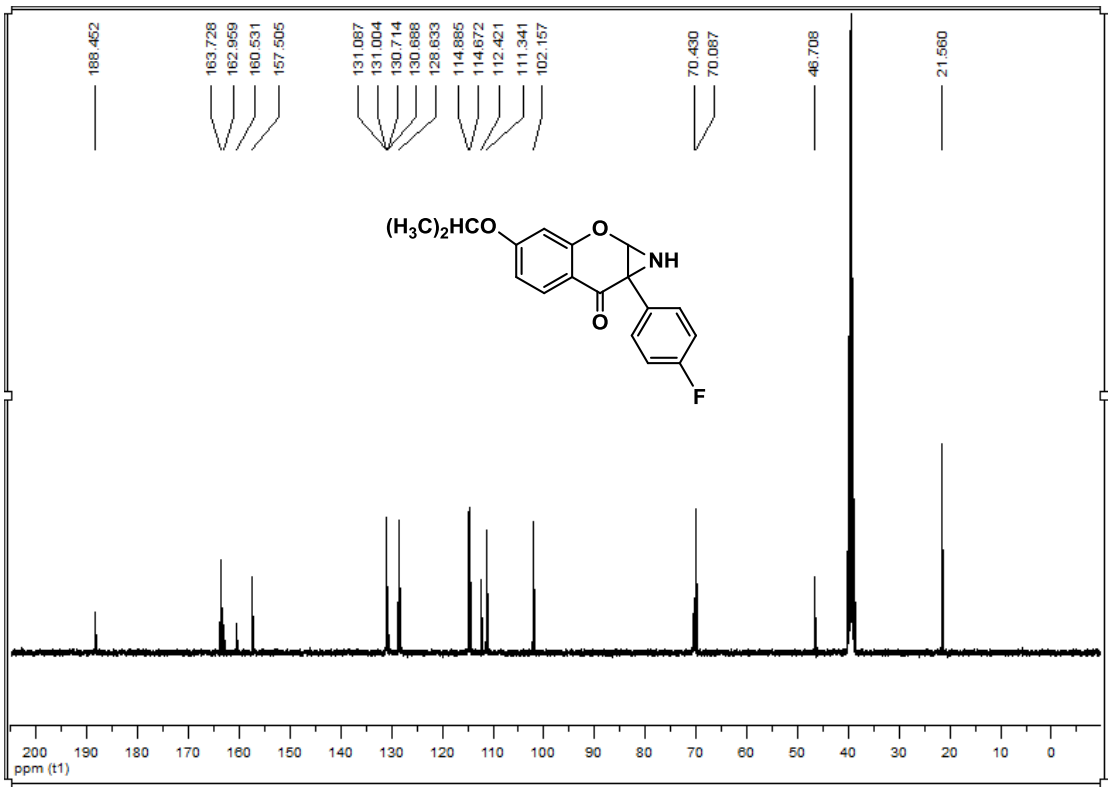


HRMS (ESI) calc. for $C_{15}H_{10}FNO_2[M+Na]^+$ 278.0593, found278.0580

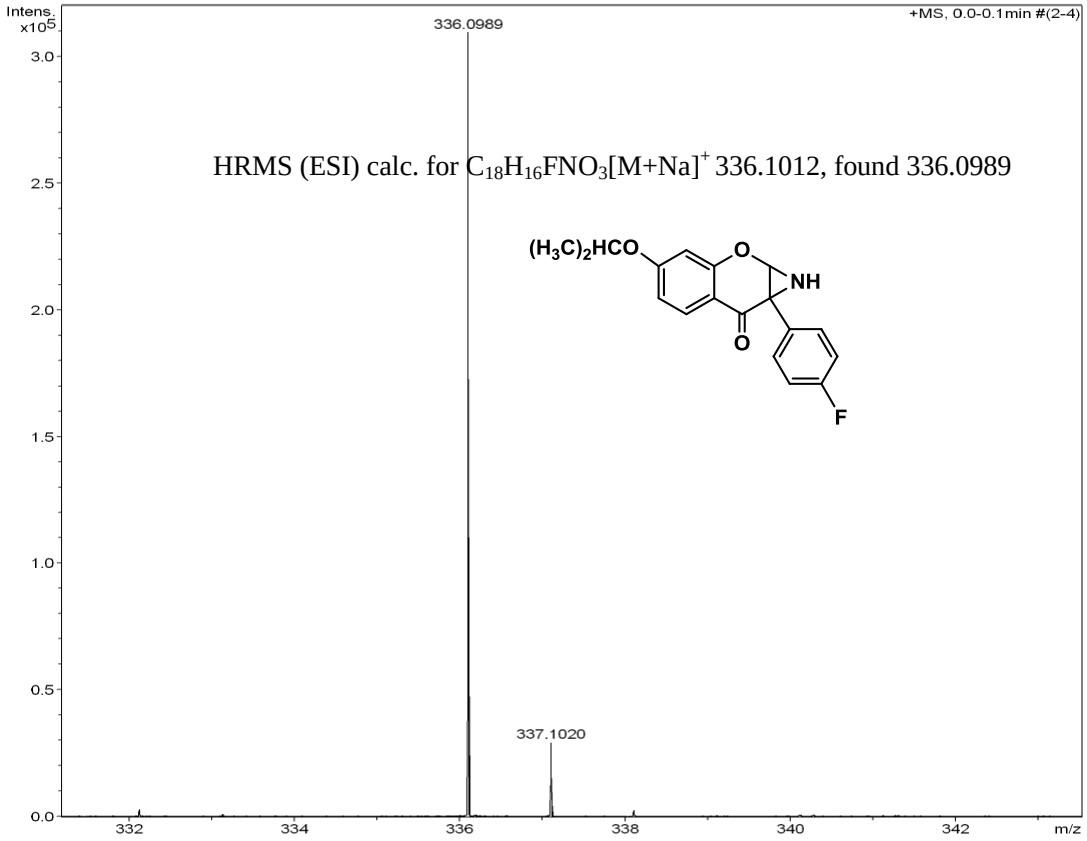
Compound 1p HR MS(CH_3OH)



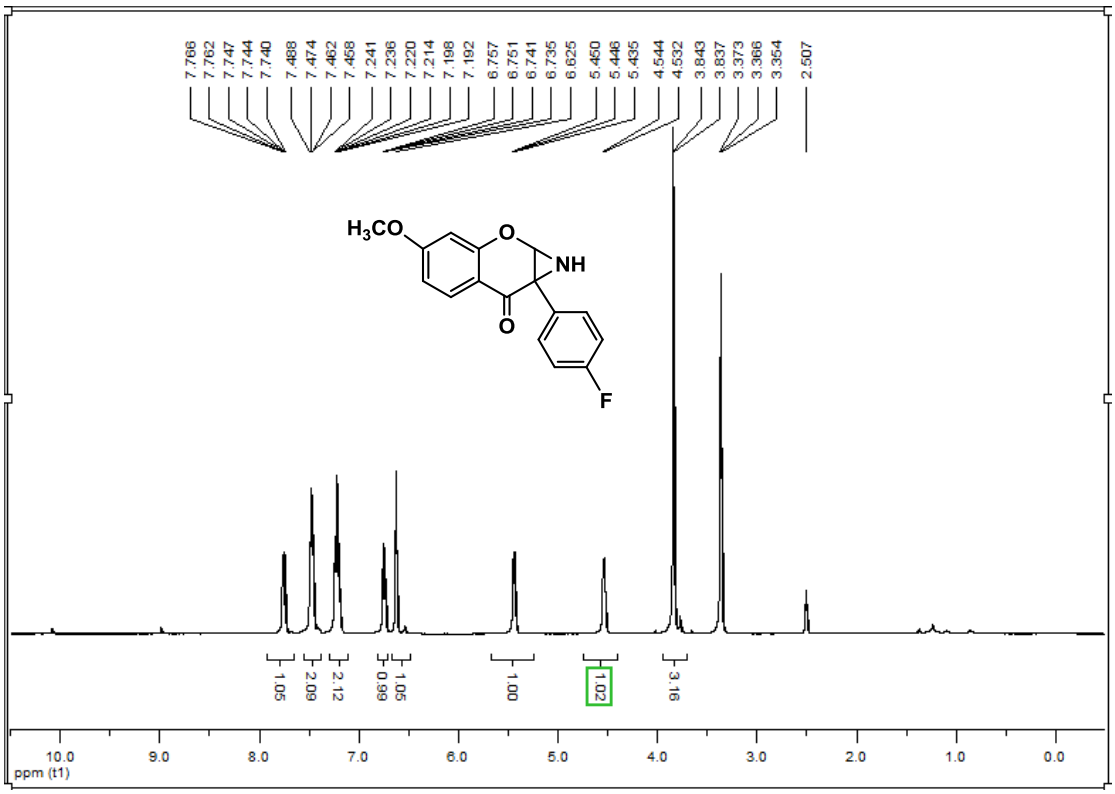
Compound 1q ^{1}H NMR(DMSO-d_6)



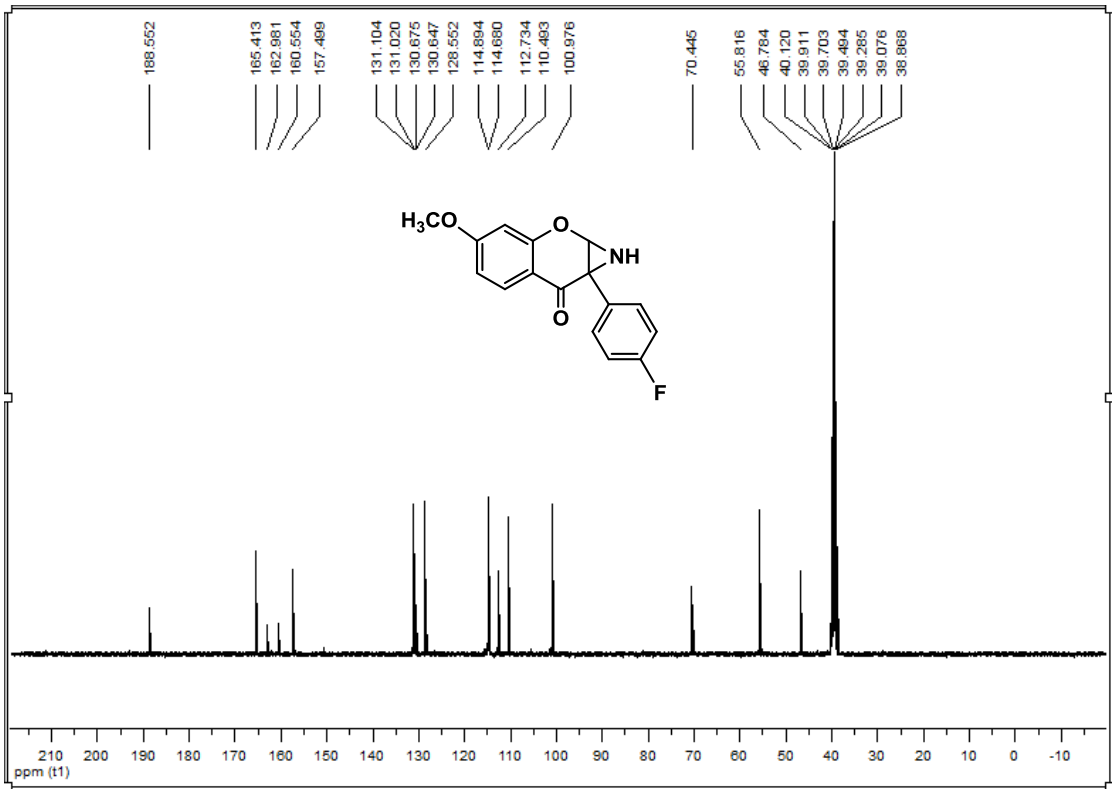
Compound 1q ^{13}C NMR(DMSO-d_6)



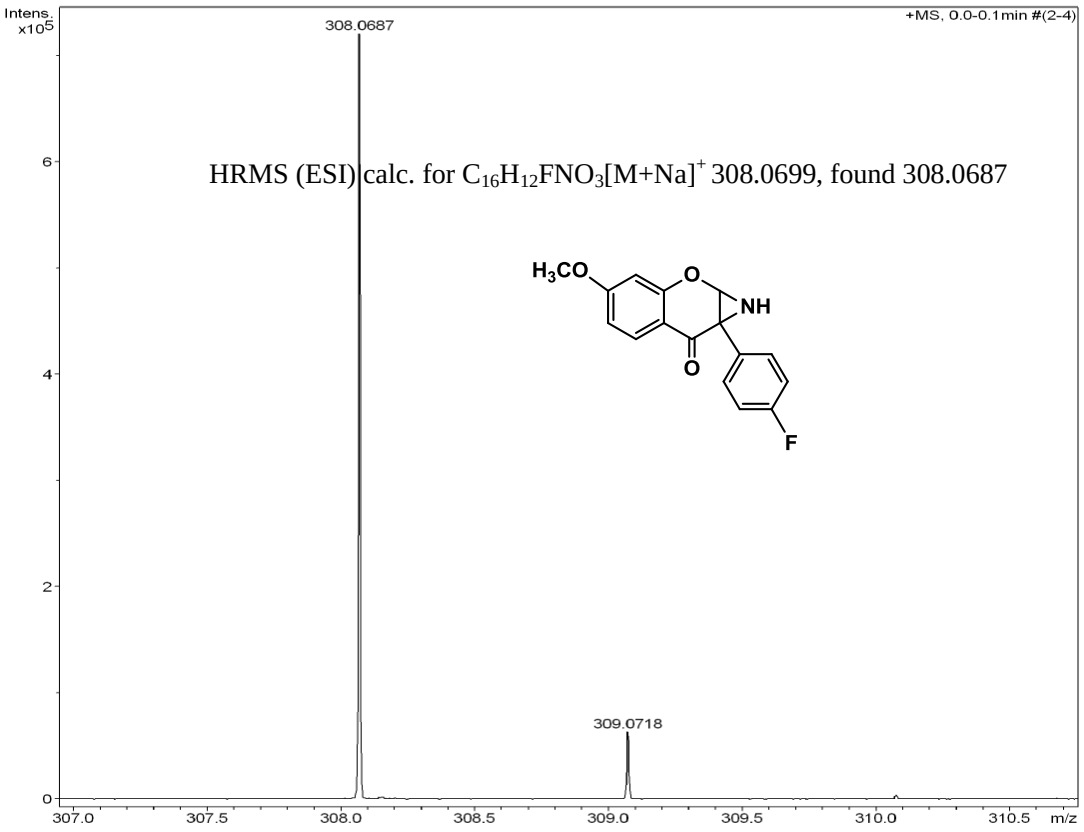
Compound 1q HR MS(CH_3OH)



Compound 1r ^{1}H NMR(DMSO-d_6)

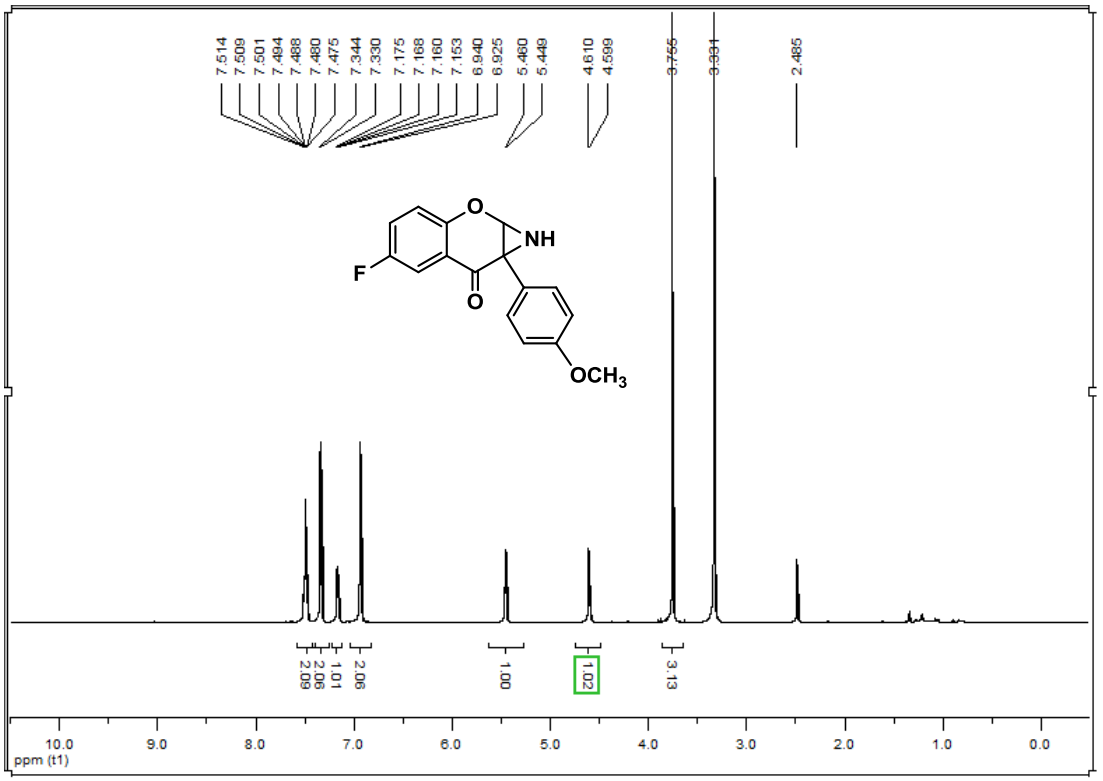


Compound 1r ^{13}C NMR(DMSO-d_6)

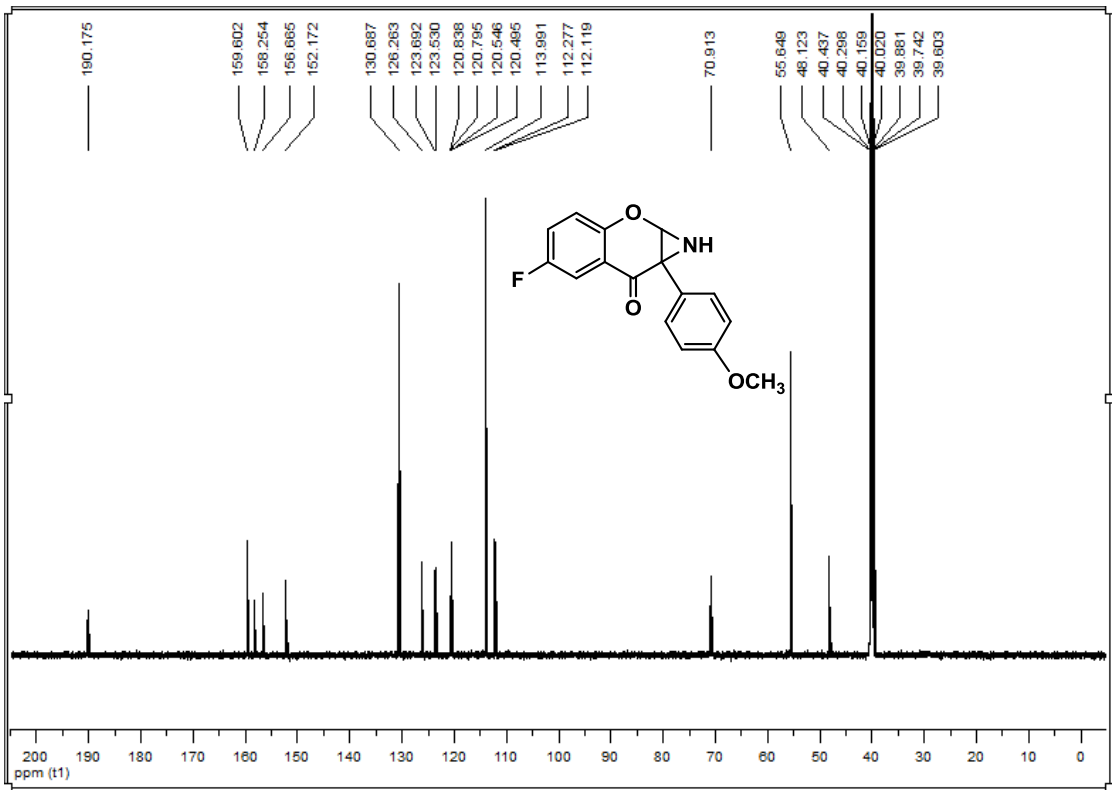


HRMS (ESI) calc. for $C_{16}H_{12}FNO_3[M+Na]^+$ 308.0699, found 308.0687

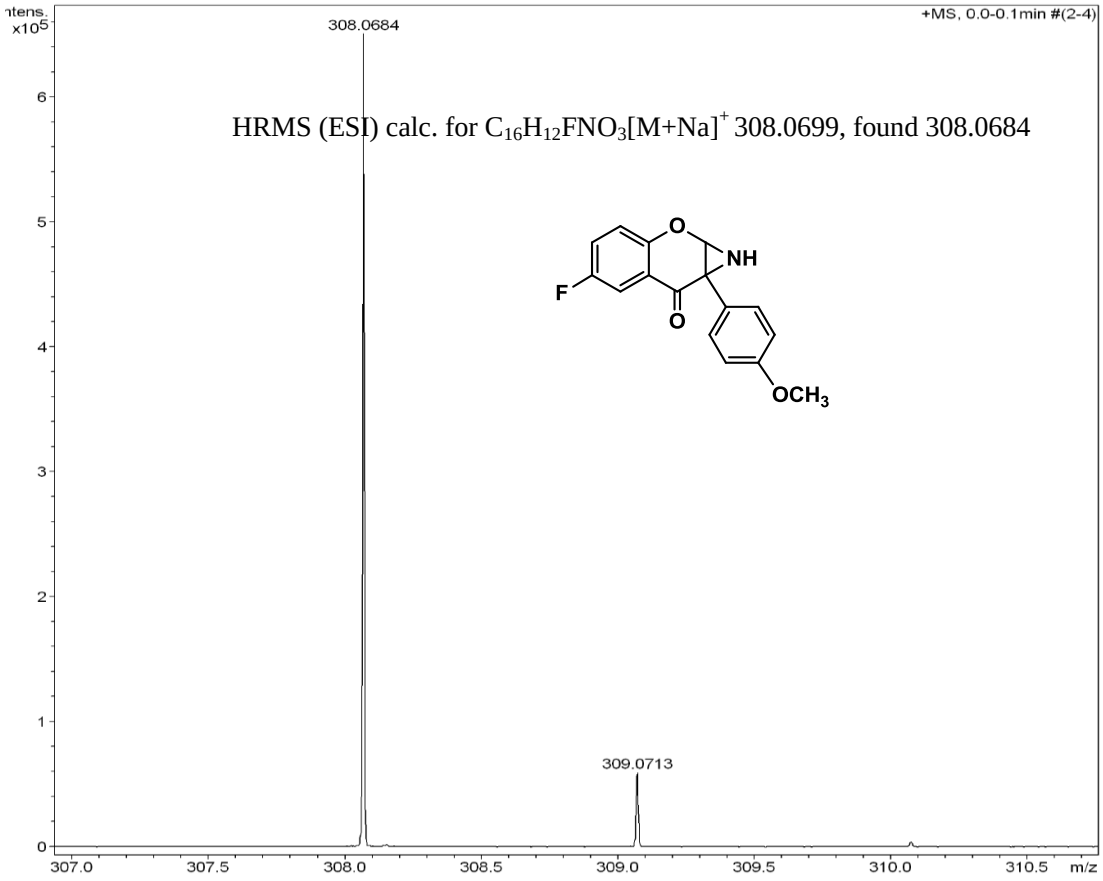
Compound 1r HR MS(CH_3OH)



Compound 1s ^{1}H NMR(DMSO-d_6)

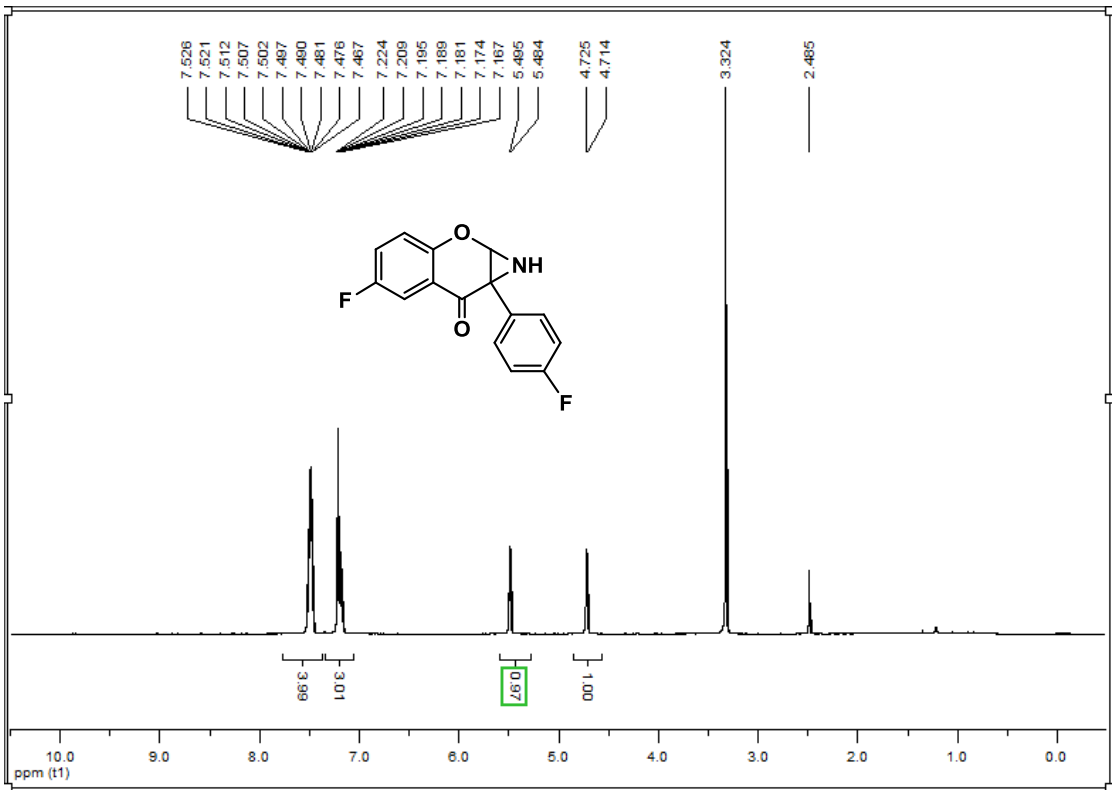


Compound 1s ^{13}C NMR(DMSO-d_6)

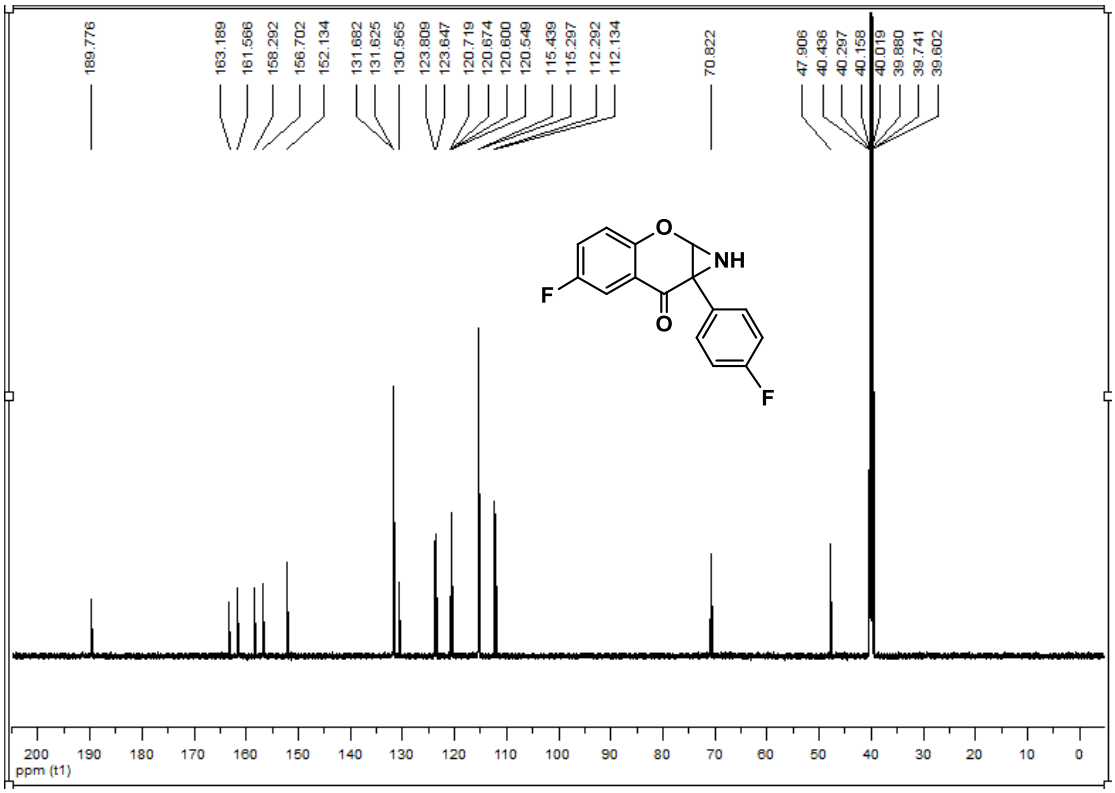


HRMS (ESI) calc. for $C_{16}H_{12}FNO_3[M+Na]^+$ 308.0699, found 308.0684

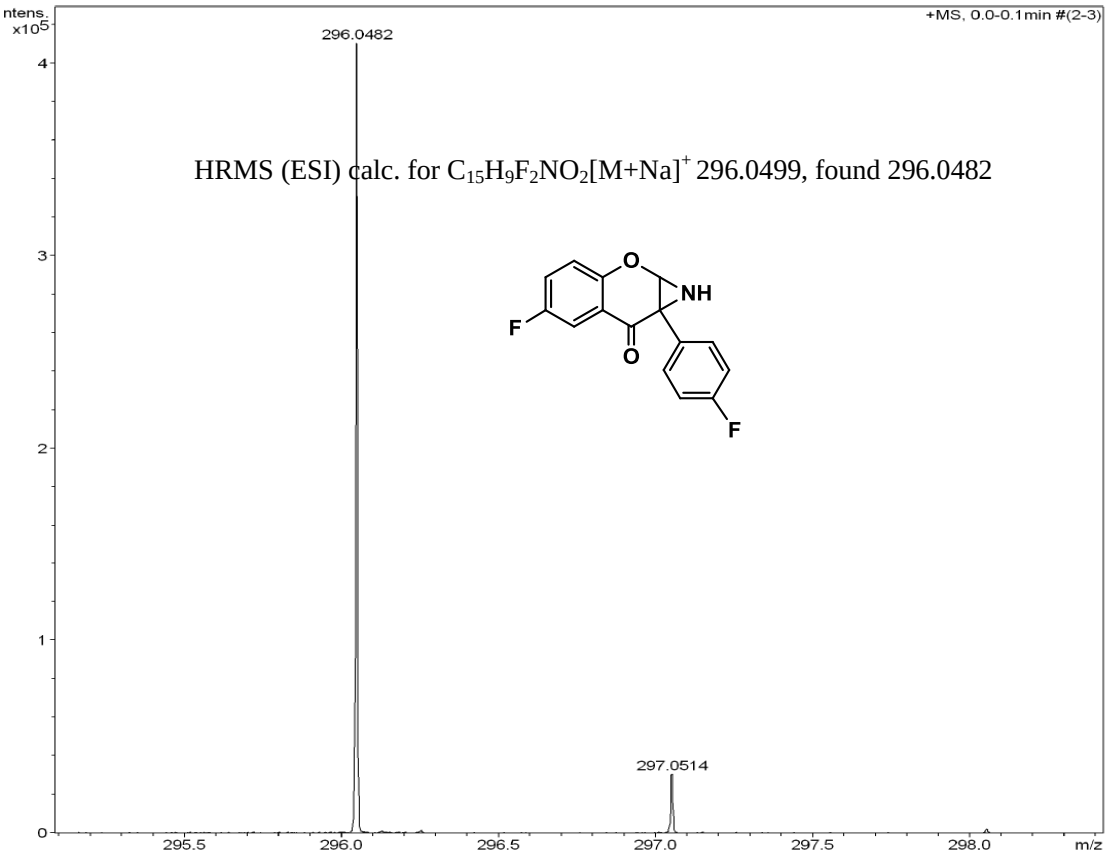
Compound 1s HR MS(CH_3OH)



Compound 1t ^{1}H NMR(DMSO-d_6)

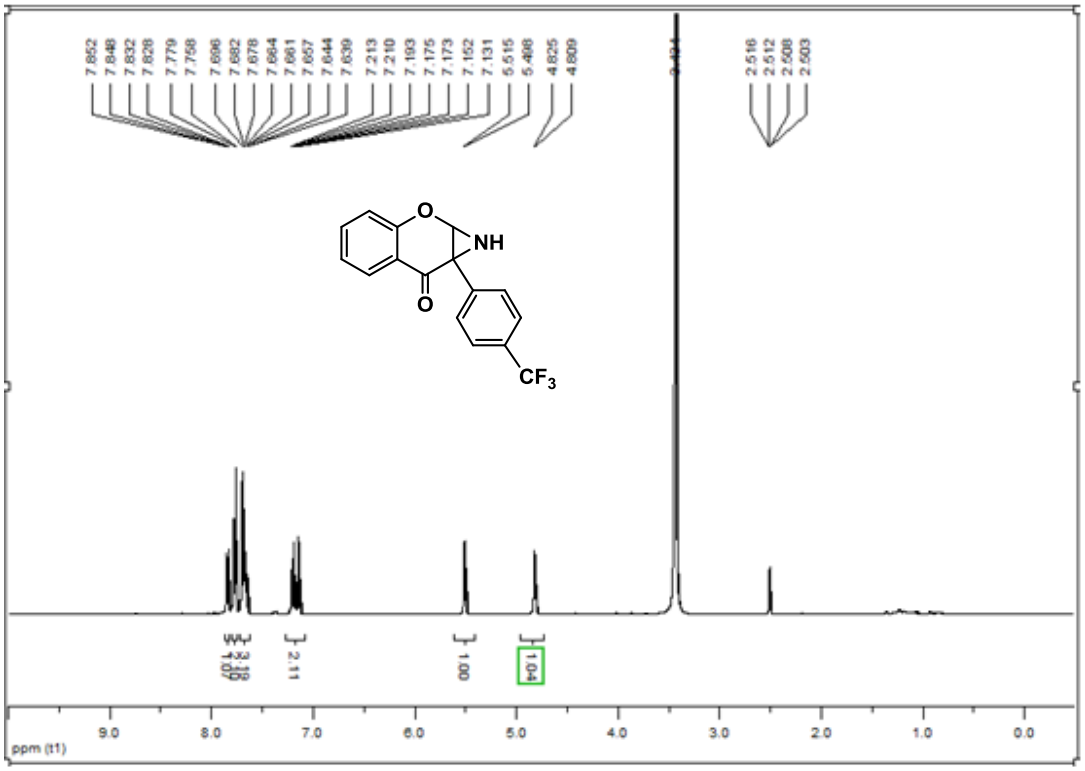


Compound 1t ^{13}C NMR(DMSO-d_6)

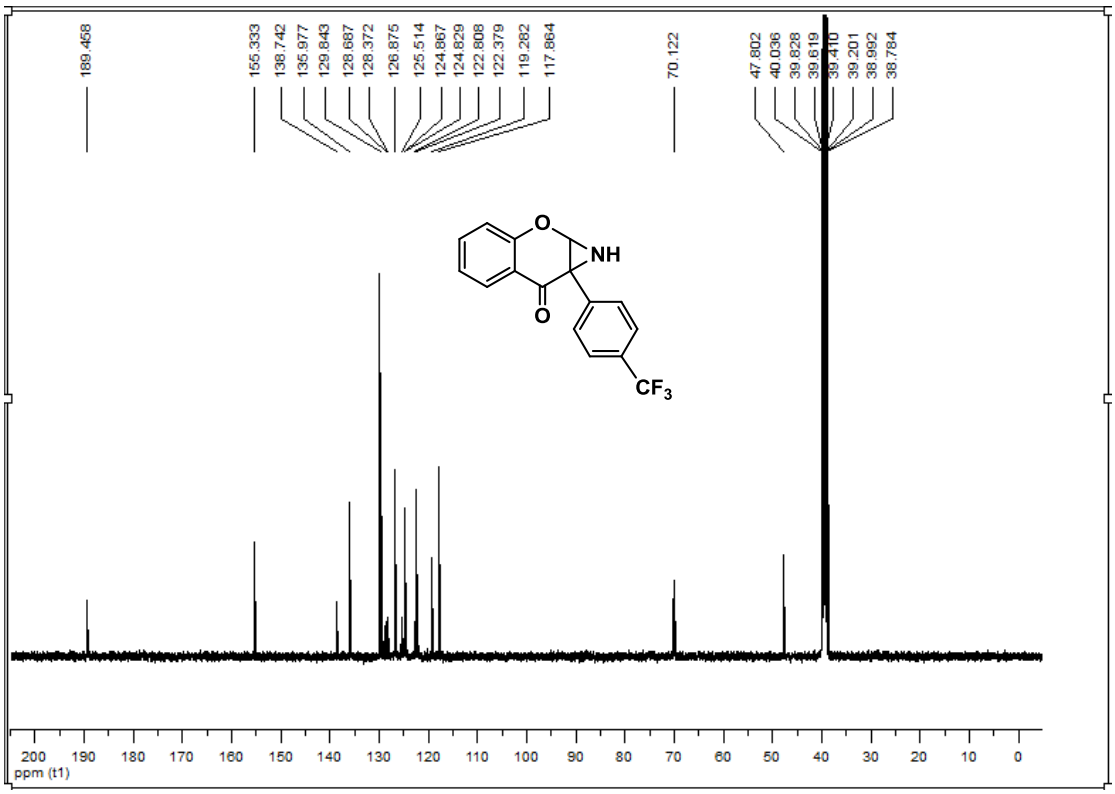


HRMS (ESI) calc. for $C_{15}H_9F_2NO_2[M+Na]^+$ 296.0499, found 296.0482

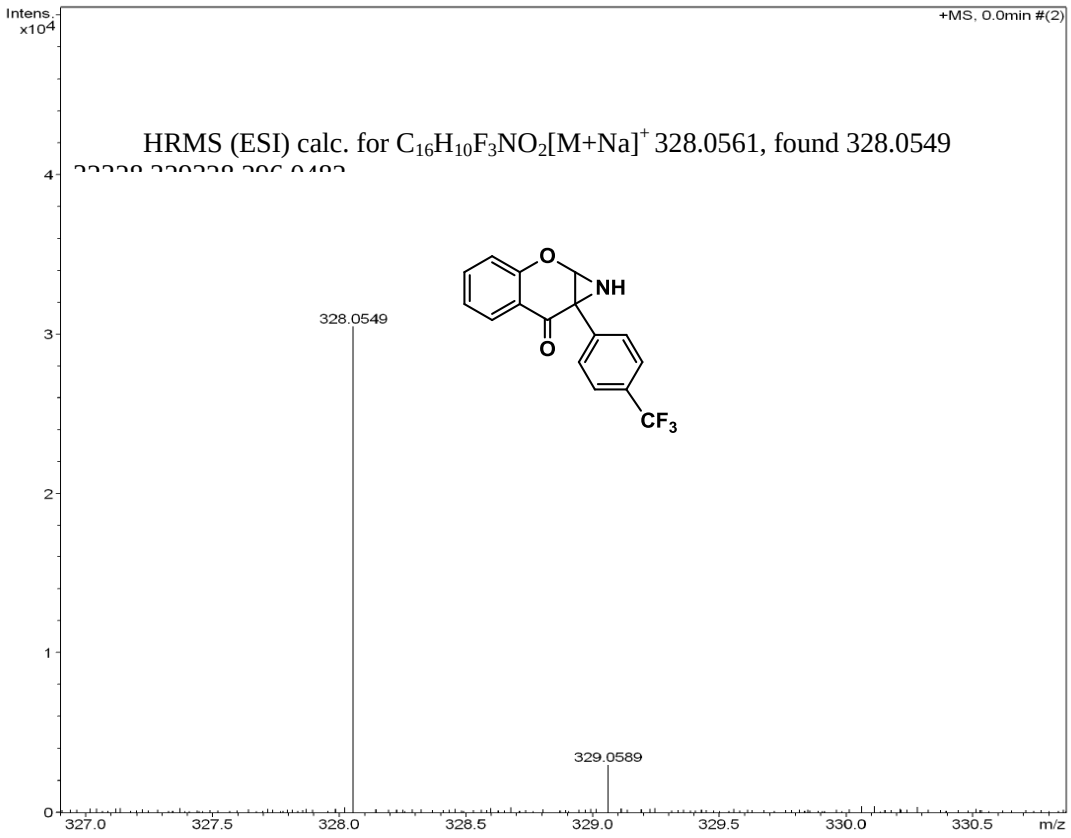
Compound 1t HR MS(CH_3OH)



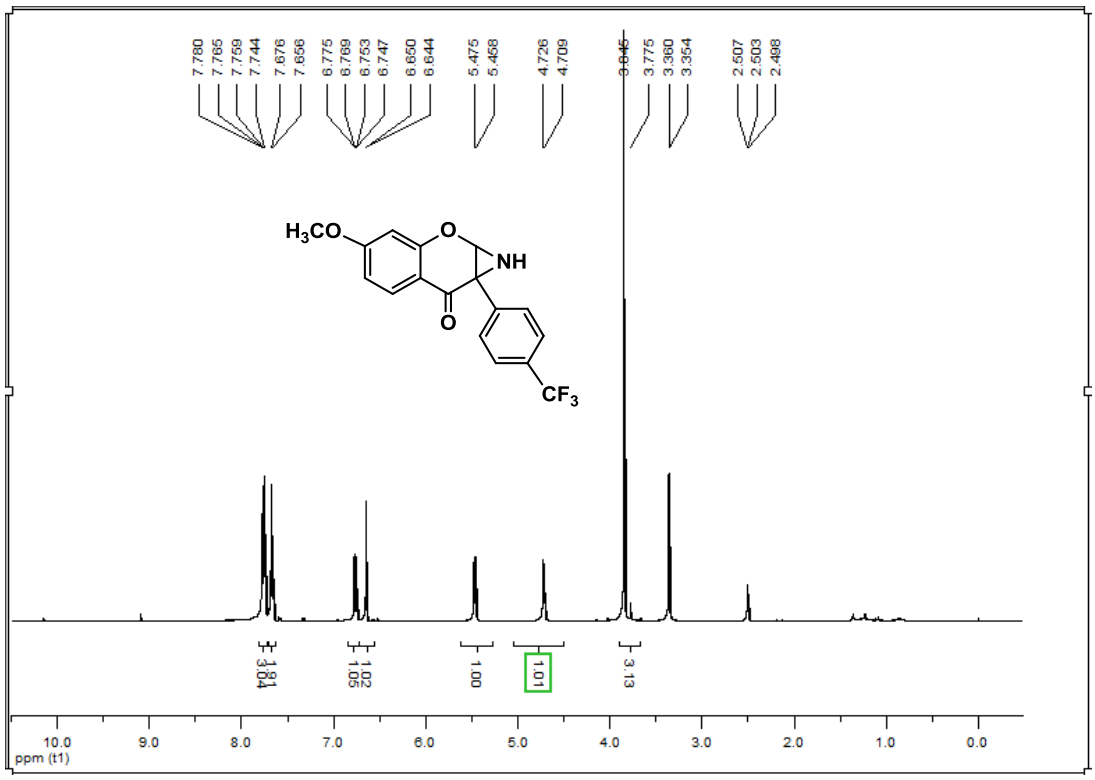
Compound 1u ^{1}H NMR(DMSO-d_6)



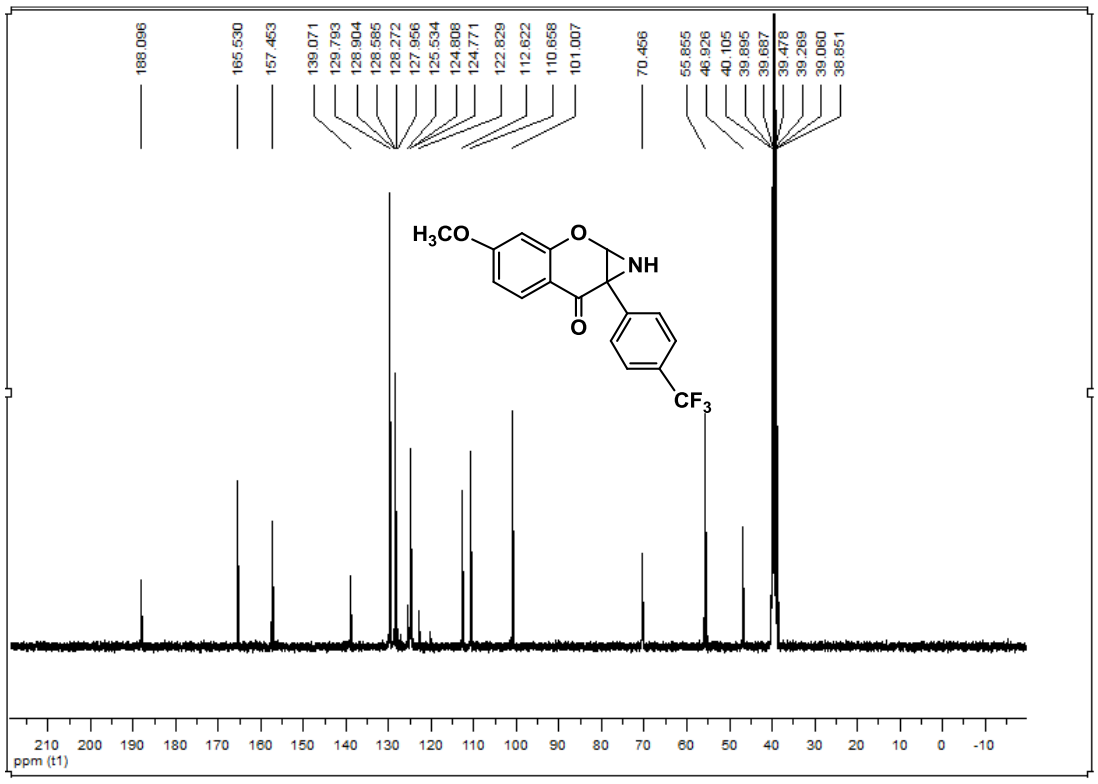
Compound 1u ^{13}C NMR(DMSO-d_6)



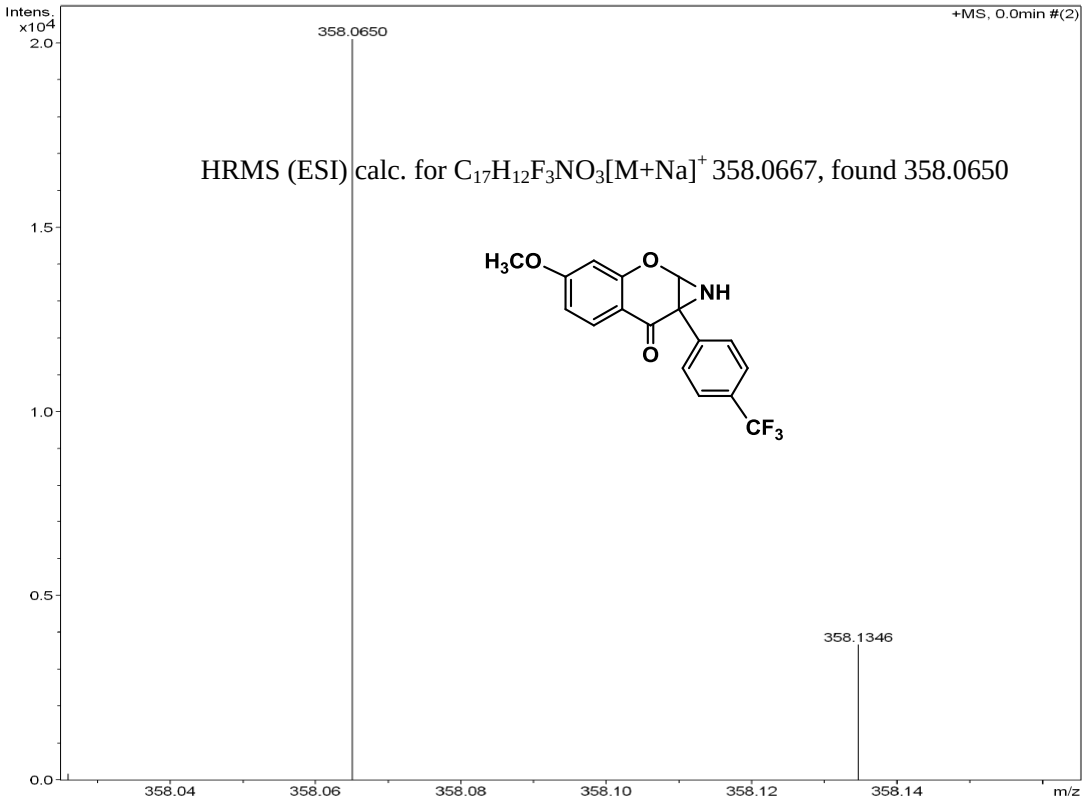
Compound 1u HR MS(CH_3OH)



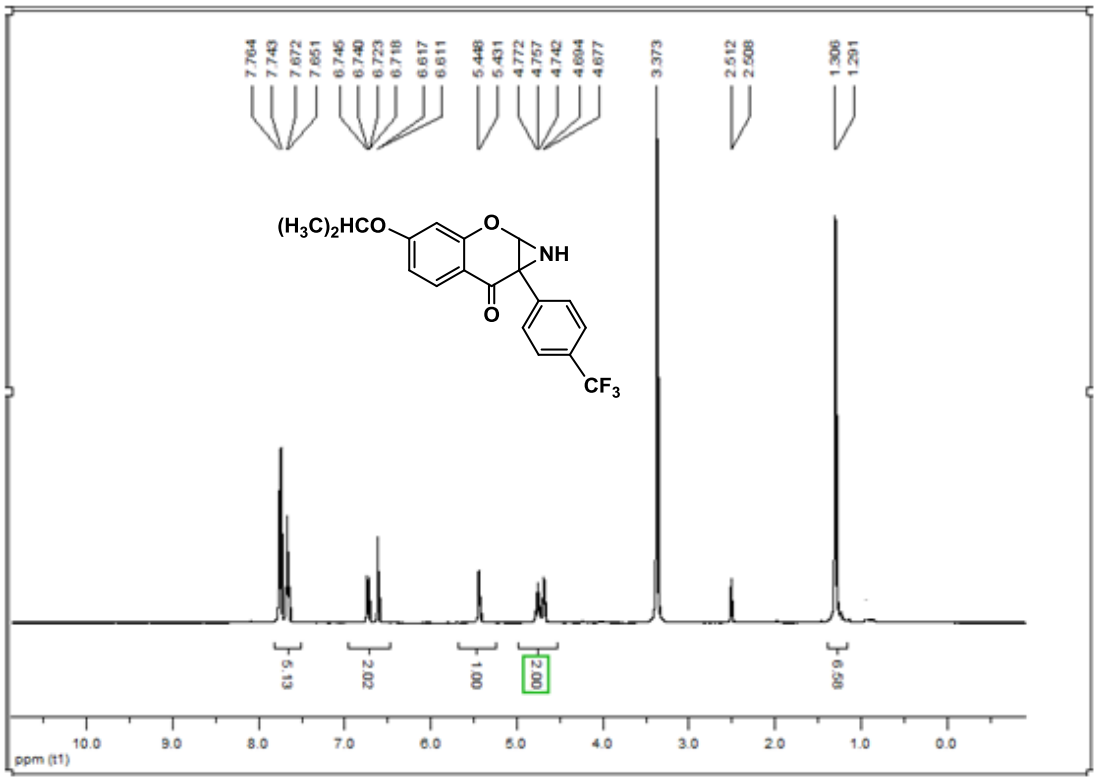
Compound 1v ^{1}H NMR(DMSO-d_6)



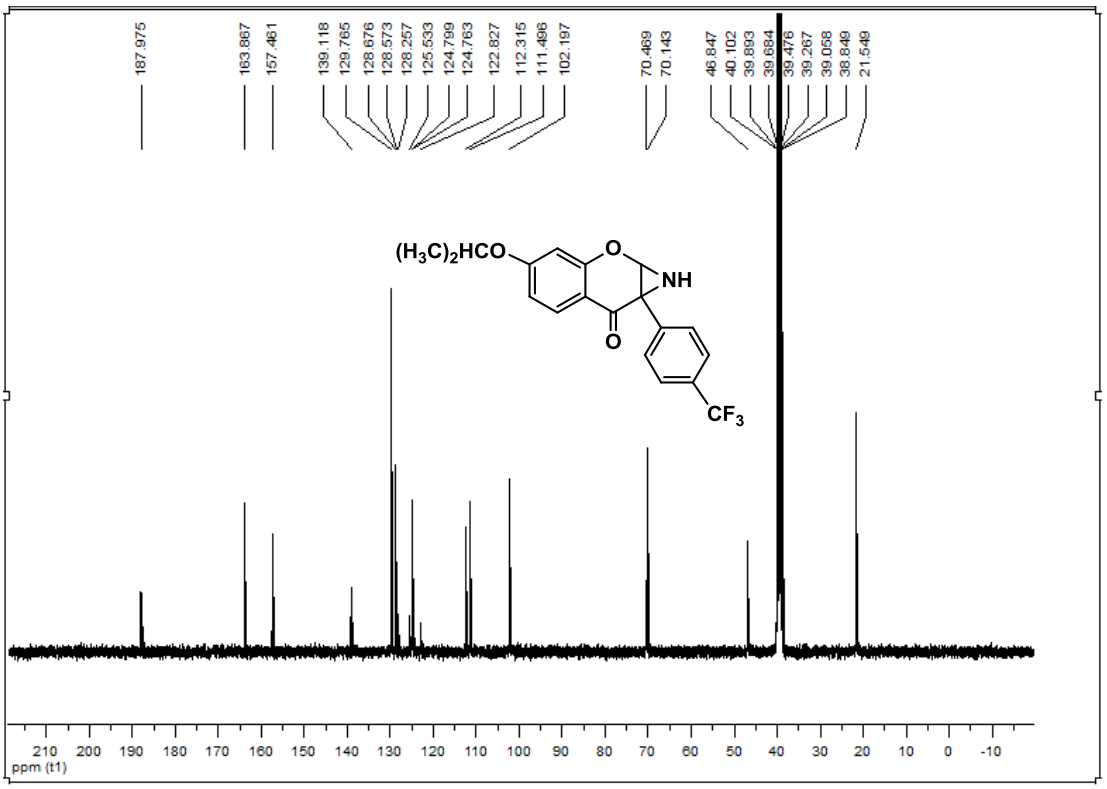
Compound 1v ^{13}C NMR(DMSO-d_6)



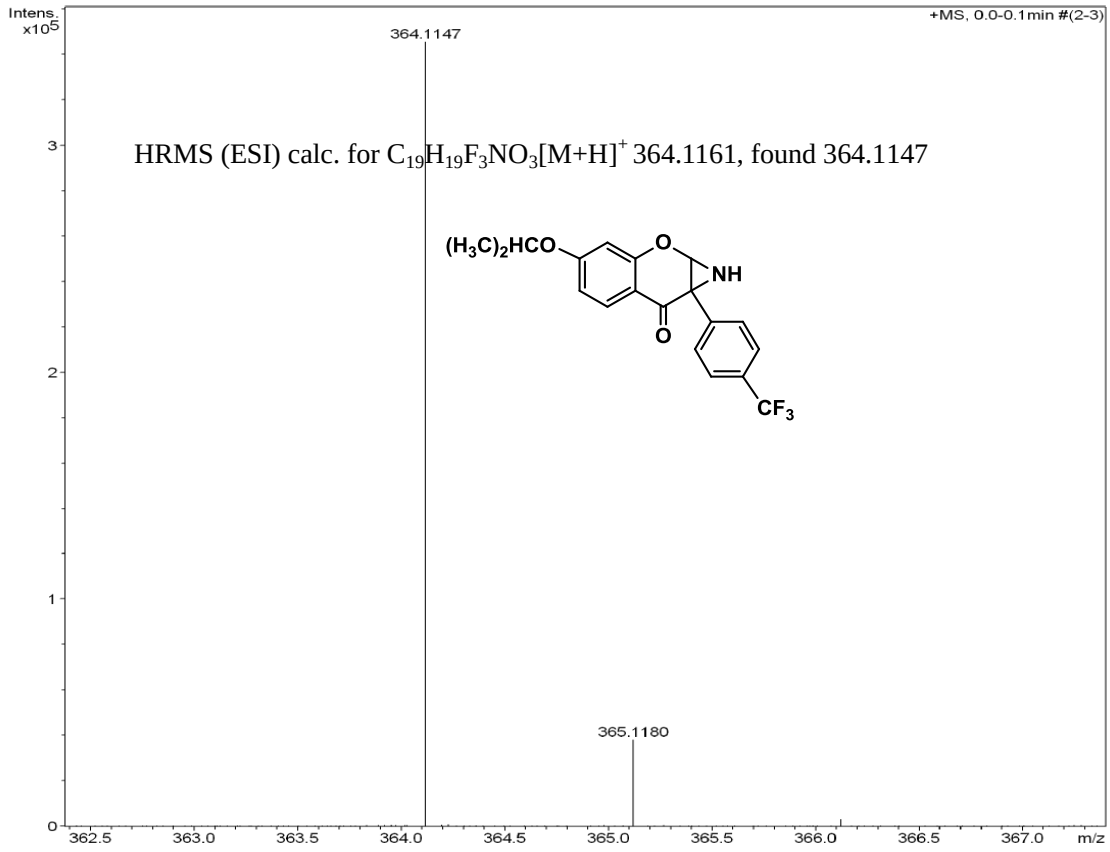
Compound 1v HR MS(CH_3OH)



Compound 1w ^{1}H NMR(DMSO-d_6)



Compound 1w ^{13}C NMR(DMSO-d_6)



HRMS (ESI) calc. for $C_{19}H_{19}F_3NO_3[M+H]^+$ 364.1161, found 364.1147

Compound 1w HR MS(CH_3OH)