Electronic Supplementary Material (ESI) for Green Chemistry.

Niobium-promoted reaction of α-phenylglyoxylic acid with *ortho*-functionalized anilines: synthesis of 2-arylbenzothiazoles and 3-aryl-*2H*-benzo[*b*][1,4]benzoxazin-2-ones

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General Information: The reactions were monitored by TLC carried out on Merk silica gel (60 F_{254}) by using UV light as visualization agent and the mixture between 5% of vanillin in 10% of H_2SO_4 under heating conditions as developing agents. Merck silica gel (particle size 0.040-0.063 mm) was used to flash chromatography. Hydrogen nuclear magnetic resonance spectra (^{1}H NMR) were obtained at 400 MHz on Bruker Nuclear Ascend 400 spectrometers. The spectra were recorded in $CDCl_3$ solutions. The chemical shifts are reported in ppm, referenced to tetramethysilane (TMS) as the external reference. Hydrogen coupling patterns are described as singlet (s), doublet (d), triplet (t) and multiplet (m). Coupling constants (*J*) are reported in Hertz. Carbon-13 nuclear magnetic resonance spectra (^{13}C NMR) were obtained at 100 MHz on Bruker Nuclear Ascend 400 spectrometers. The chemical shifts are reported in ppm, referenced to the solvent peak of $CDCl_3$. High-resolution mass spectra were obtained with a LTQ Orbitrap Discovery massa spectrometer (Thermo Scientific) and these experiments were realized by direct infusion (5 μL/min) in electrospray ionization source using the positive mode. The isotopes simulation were calculated using the Quad Browser v.2.0.7 (Thermo Fisher Scientific Inc.) to compare the elementary composition.

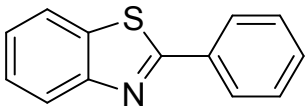
General Procedure for the Synthesis of the *2-(aryl)-benzothiazoles*

In a round-bottom flask were added α-phenylglyoxylic acids 1**a-d** (0.6 mmol), 2-aminothiophenoles **2a-b** (0.5 mmol), ANO (10 mol%, 0.019 g) and the PEG-400 (1 mL), respectively. The resulting solution was stirred for the time indicated on the Table 2 under conventional heating at 100 ºC and under ultrasound irradiation (20 KHz, 60% of ultrasonic amplitude). After that, the reaction mixture was received in water (20 mL), extracted with ethyl acetate (3 x 10 mL), dried over $MgSO_4$ and concentrated under vacuum. Thus, the residue was purified through chromatography column on silica gel utilizing hexane/ethyl acetate as the eluent.

General Procedure for the Synthesis of the *3-(aryl)-benzoxazin-2-onas*

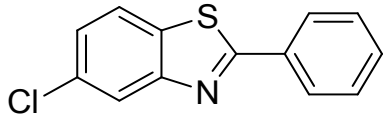
In a round-bottom flask were added α-phenylglyoxylic acids **1a-c** (0.6 mmol), 2-aminophenoles **4a-c** (0.5 mmol), ANO (10 mol%, 0.019 g) and the PEG-400 (1 mL), respectively. The resulting mixture was stirred for the time indicated on the Table 3 under conventional heating at 100 ºC and under ultrasound irradiation (20 KHz, 60% of ultrasonic amplitude). After that, the reaction mixture was received in water (20 mL), extracted with ethyl acetate (3X 10 mL), dried over $MgSO_4$ and concentrated under vacuum. Thus, the residue was purified through chromatography column on silica gel utilizing hexane/ethyl acetate as the eluent.

2-phenylbenzo[d]thiazole (3aa)



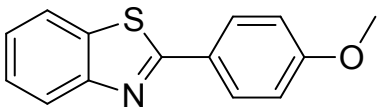
Yield: 0.090 g (85%); Light yellow solid; mp 107-110 ºC (Lit.[1]: 109-111 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.11 – 8.07 (m, 3H), 7.91 – 7.89 (m, 1H), 7.51 – 7.47 (m, 4H), 7.39 (ddd, *J* = 8.3, 7.3, 1.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 168.05, 154.13, 135.05, 133.61, 130.94, 129.00, 127.55, 126.29, 125.17, 123.22, 121.60.

5-chloro-2-phenylbenzo[d]thiazole (3ab)



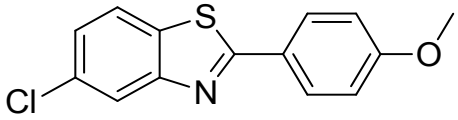
Yield: 0.096 g (77%); Yellow solid; mp 132-134 ºC (Lit.[2]: 139 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.05 – 8.03 (m, 3H), 7.75 (d, *J* = 8.5 Hz, 1H), 7.48 – 7.46 (m, 3H), 7.32 (dd, *J* = 8.5, 2.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.83, 154.91, 133.24, 133.17, 132.24, 131.26, 129.01, 127.53, 125.56, 122.96, 122.22.

2-(4-methoxyphenyl)benzo[d]thiazole (3ba)



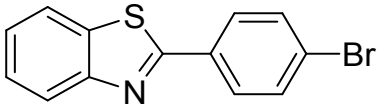
Yield: 0.080 g (66%); White needled solid; mp 114-116 ºC (Lit.[3]: 115-116 ºC) ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.03 – 8.00 (m, 3H), 7.86 – 7.84 (m, 1H), 7.45 (ddd, *J* = 8.2, 7.3, 1.2 Hz, 1H), 7.33 (ddd, *J* = 8.2, 7.3, 1.2 Hz, 1H), 6.99 – 6.97 (m, 2H), 3.85 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 167.78, 161.85, 154.17, 134.80, 129.04, 126.37, 126.13, 124.72, 122.76, 121.44, 114.30, 55.37.

5-chloro-2-(4-methoxyphenyl)benzo[d]thiazole (3bb)



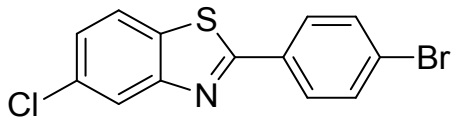
Yield: 0.045 g (30%); White solid: mp 138-140 ºC (Lit.[4]: not observed) ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.01 – 7.98 (m, 3H), 7.75 (d, *J* = 8.5 Hz, 1H), 7.31 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.00 – 6.97 (m, 2H), 3.87 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.65, 162.18, 155.07, 133.08, 132.13, 129.17, 126.02, 125.14, 122.59, 122.12, 114.40, 55.45.

2-(4-bromophenyl)benzo[d]thiazole (3ca)



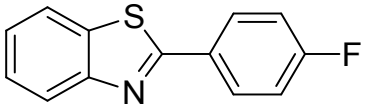
Yield: 0.09 g (63%); White solid: mp 124-126 ºC (Lit.[5]: 127-129 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.05 (d, *J* = 8.1 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 2H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.48 (t, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 7.7 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.62, 154.02, 134.99, 132.49, 132.17, 128.85, 126.45, 125.40, 125.37, 123.28, 121.61.

2-(4-bromophenyl)-5-chlorobenzo[d]thiazole (3cb)



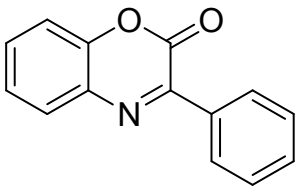
Yield: 0.096 g (60%); Light green solid: mp 140-143 ºC (Lit.[4]: not observed). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.05 (d, *J* = 2.0 Hz, 1H), 7.96 – 7.92 (m, 2H), 7.81 (d, *J* = 8.5 Hz, 1H), 7.65 – 7.62 (m, 2H), 7.38 (dd, *J* = 8.5, 2.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 168.53, 154.91, 133.27, 132.53, 132.32, 132.18, 128.93, 125.92, 125.86, 123.12, 122.34.

2-(4-fluorophenyl)benzo[d]thiazole (3da)



Yield: 0.076 g (67%); White solid: mp 98-100 ºC (Lit.[6]: 98-100 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.09 – 8.04 (m, 3H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.48 (ddd, *J* = 8.3, 7.2, 1.2 Hz, 1H), 7.37 (td, *J* = 8.3, 7.2, 1.2 Hz, 1H), 7.21 – 7.13 (m, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.67, 164.41 (d, *J* = 251.9 Hz), 154.05, 135.01, 129.92 (d, *J* = 3.3 Hz), 129.47 (d, *J* = 8.5 Hz), 126.36 , 125.19 , 123.15 , 121.56 , 116.10 (d, *J* = 22.2 Hz).

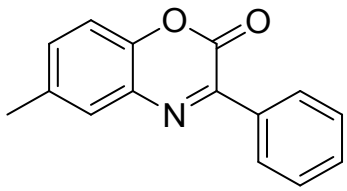
3-phenyl-2H-benzo[b][1,4]oxazin-2-one (5aa)



Yield: 0.095 g (80%); Beige solid: mp: 118-119 ºC (Lit.[7]: 121-122 ºC) ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.33 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.84 (d, *J* = 7.9 Hz, 1H), 7.53 – 7.49 (m, 4H), 7.38 (t, *J* = 8.1 Hz, 1H), 7.32 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 152.14,

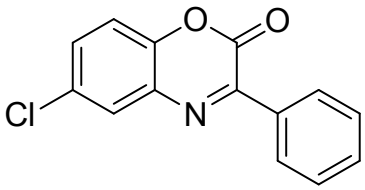
150.69, 146.37, 134.04, 131.55, 131.33, 131.02, 129.37, 129.36, 128.29, 125.46, 116.07.

6-methyl-3-phenyl-2H-benzo[b][1,4]oxazin-2-one (5ab)



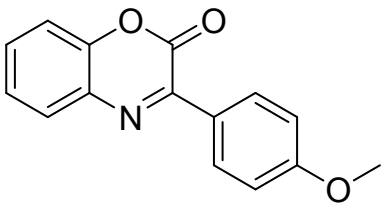
Yield: 0.1035 g (87%); Yellow needled solid: mp 126-128 ºC (Lit.[8]: 130-132 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.32-3.29 (m, 2H), 7.59 (s, 1H), 7.52 – 7.44 (m, 3H), 7.27 (d, *J* = 8.4 Hz, 1H), 7.16 (dd, *J* = 8.4, 2.1 Hz, 1H), 2.41 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 152.32, 150.44, 144.28, 135.35, 134.14, 131.97, 131.24, 131.18, 129.32, 129.15, 128.22, 115.59, 20.72.

6-chloro-3-phenyl-2H-benzo[b][1,4]oxazin-2-one (5ac)



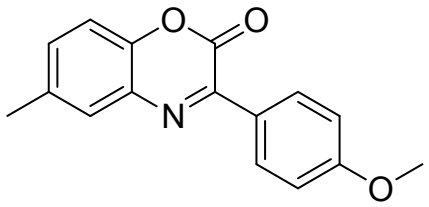
Yield: 0.0895 g (70%); Orange needled solid: mp 139-141 ºC (Lit.[8]: 138-139 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.36 – 8.33 (m, 2H), 7.85 (d, *J* = 2.5 Hz, 1H), 7.57 – 7.46 (m, 4H), 7.26 (d, *J* = 3.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.77, 151.67, 145.02, 133.70, 132.14, 131.87, 130.96, 130.67, 129.58, 128.83, 128.45, 117.27.

3-(4-methoxyphenyl)-2H-benzo[b][1,4]oxazin-2-one (5ba)



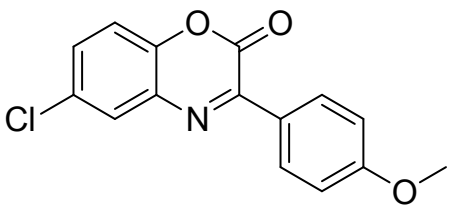
Yield: 0.082 g (75%); Light yellow needled solid: mp 132-134 ºC (Lit.[8]: 131-132 ºC). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.42 – 8.39 (m, 2H), 7.82 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.48 (ddd, *J* = 8.2, 7.4, 1.6 Hz, 1H), 7.37 (td, *J* = 7.9, 1.4 Hz, 1H), 7.32 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.03 – 6.99 (m, 2H), 3.89 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.38, 152.47, 149.89, 146.29, 131.78, 131.38, 130.43, 129.08, 126.82, 125.46, 116.06, 113.82, 55.44.

3-(4-methoxyphenyl)-6-methyl-2H-benzo[b][1,4]oxazin-2-one (5bb)



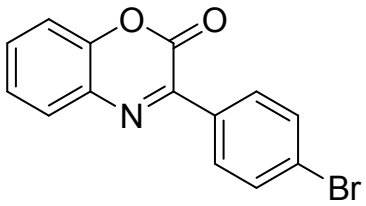
Yield: 0.115 g (86%); Dark yellow solid: mp 135-138 ºC. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.41 – 8.37 (m, 1H), 7.61 (m, 1H), 7.28 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.02 – 6.98 (m, 2H), 3.89 (s, 3H), 2.45 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.27, 152.70, 149.75, 144.24, 135.34, 131.50, 131.41, 131.32, 128.94, 126.95, 115.61, 113.78, 55.42, 20.85.

6-chloro-3-(4-methoxyphenyl)-2H-benzo[b][1,4]oxazin-2-one (5bc)



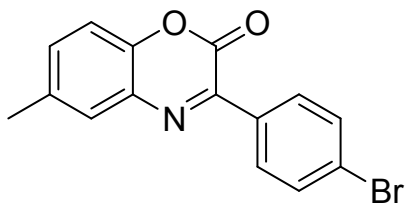
Yield: 0.100 g (70%); Yellow needled solid; mp 160-162 ºC. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.43 – 8.39 (m, 2H), 7.80 (d, *J* = 2.4 Hz, 1H), 7.42 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.24 (d, *J* = 8.8 Hz, 1H), 7.01 – 6.98 (m, 2H), 3.89 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.75, 151.85, 150.64, 144.80, 132.28, 131.60, 130.52, 130.19, 128.43, 126.36, 117.10, 113.89, 55.45. HRMS calcd. for $C_{15}H_{10}ClNO_3$ $[M+H]^+$ 287.0349. Found: 287.0338.

3-(4-bromophenyl)-2H-benzo[b][1,4]oxazin-2-one (5ca)



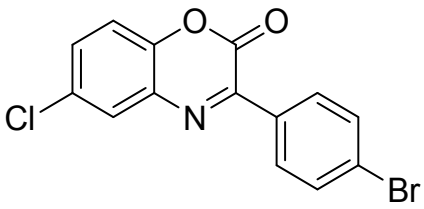
Yield: 0.1132 g (75%); Light yellow solid; mp 133-135 ºC (Lit.[8]: 134-135). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.29 – 8.25 (m, 2H), 7.84 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.65 – 7.60 (m, 2H), 7.53 (ddd, *J* = 8.2, 1.5 Hz, 1H), 7.40 (ddd, *J* = 7.9, 1.2 Hz, 1H), 7.34 (dd, *J* = 8.2, 1.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 152.08, 149.57, 146.45, 132.90, 131.63, 131.53, 131.42, 130.98, 129.48, 126.46, 125.69, 116.21.

3-(4-bromophenyl)-6-methyl-2H-benzo[b][1,4]oxazin-2-one (5cb)



Yield: 0.150 g (93%); Yellow solid: mp 129-131 ºC. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.27 – 8.23 (m, 2H), 7.62 – 7.59 (m, 3H), 7.32 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 2.45 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 152.29, 149.35, 144.38, 135.63, 133.02, 132.40, 131.56, 131.24, 130.92, 129.27, 126.29, 115.74, 20.82. HRMS calcd. for $C_{15}H_{10}BrNO_2$ $[M+H]^+$ 314.9895. Found: 314.9891.

3-(4-bromophenyl)-6-chloro-2H-benzo[b][1,4]oxazin-2-one (5cc)



Yield: 0.111 g (66%); Orange needled solid: 153-155 ºC. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.27 – 8.25 (m, 2H), 7.84 (d, *J* = 2.4 Hz, 1H), 7.64 – 7.62 (m, 2H), 7.48 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.28 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.48, 150.52, 144.98, 132.46, 131.99, 131.73, 131.25, 131.08, 130.84, 128.84, 127.04, 117.31. HRMS calcd. for $C_{14}H_7BrClNO_2$ $[M+H]^+$ 334.9349. Found: 334.9339

References

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[2] S. Kamila, H. Zhang and E. R. Biehl, *Heterocycles* 2005, **65**, 2119.

[3] (a) A. M. Youssed, H. M. Mohamed, C. Czezowski, A. Ata and A.S. Abd-El-Aziz, *Heterocycles* 2006, **68**, 347; (b) H. Hachiya, K. Hirano, T. Satoh and M. Miura, *Org. Lett.*, 2009, **11**, 1737.

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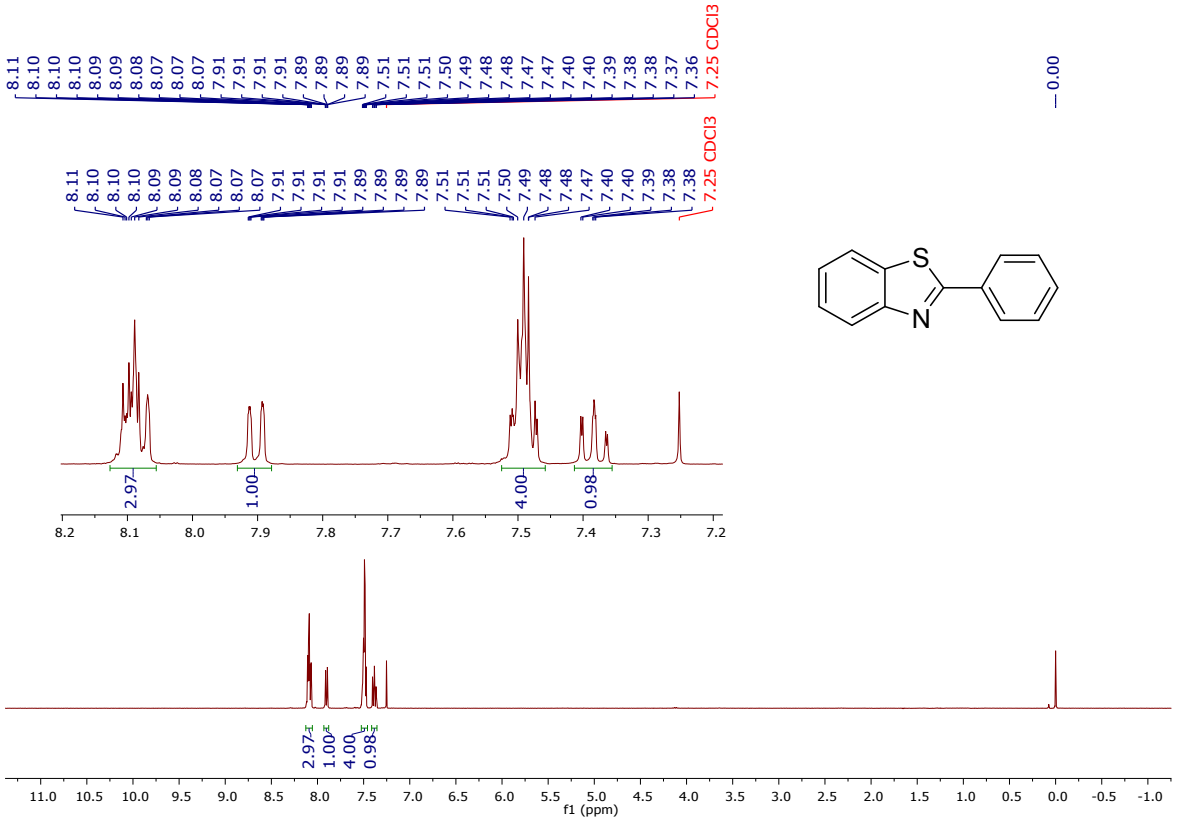
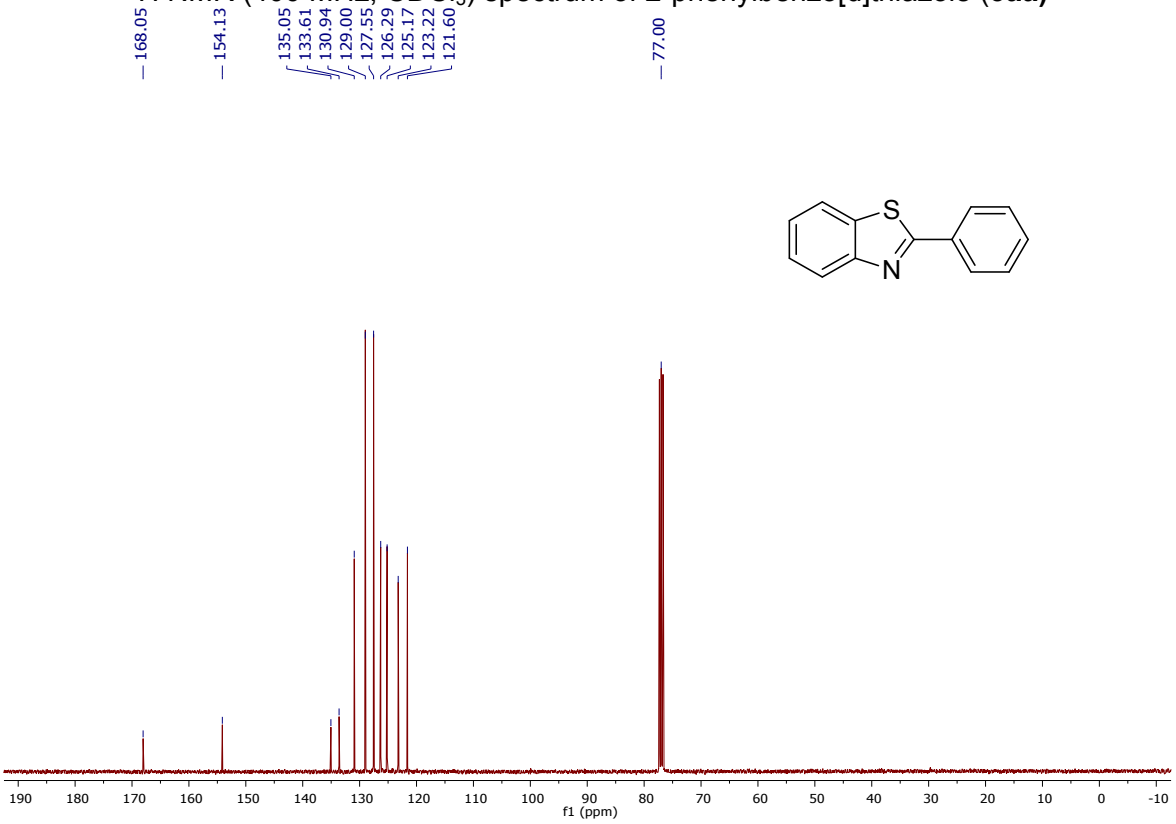
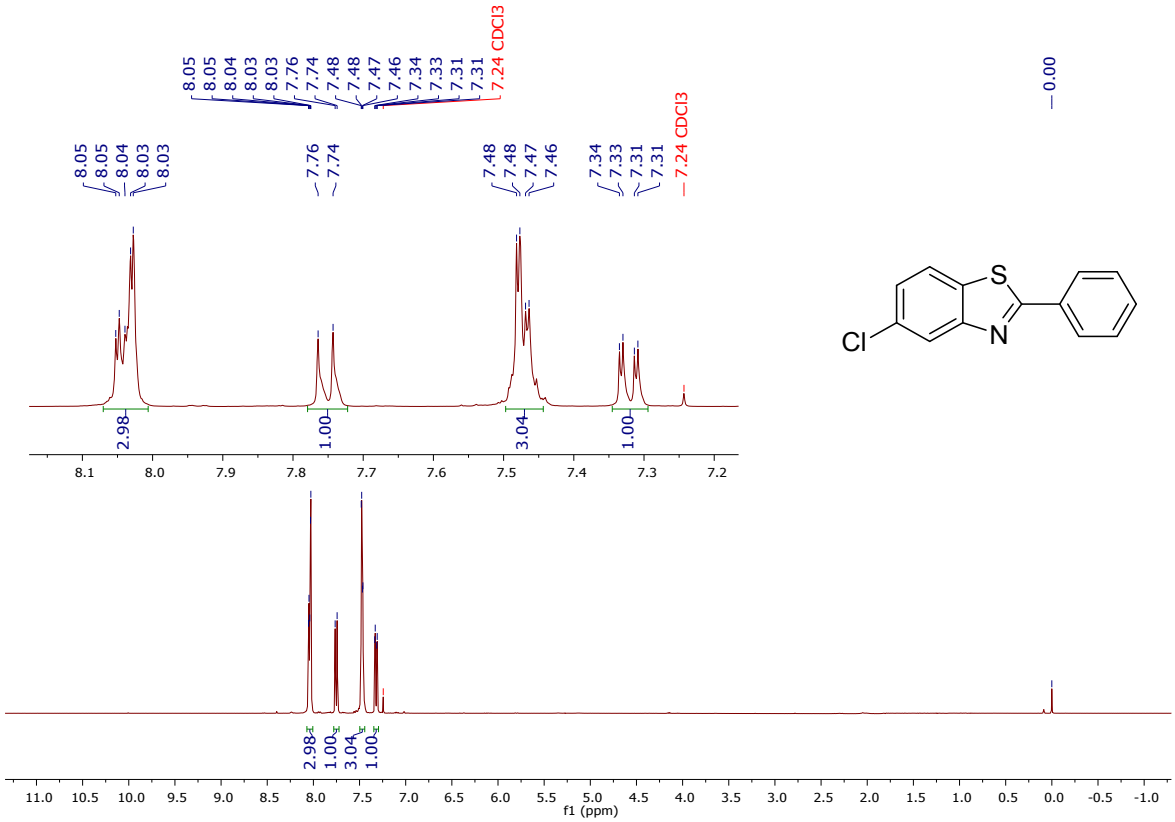
SELECTED SPECTRA



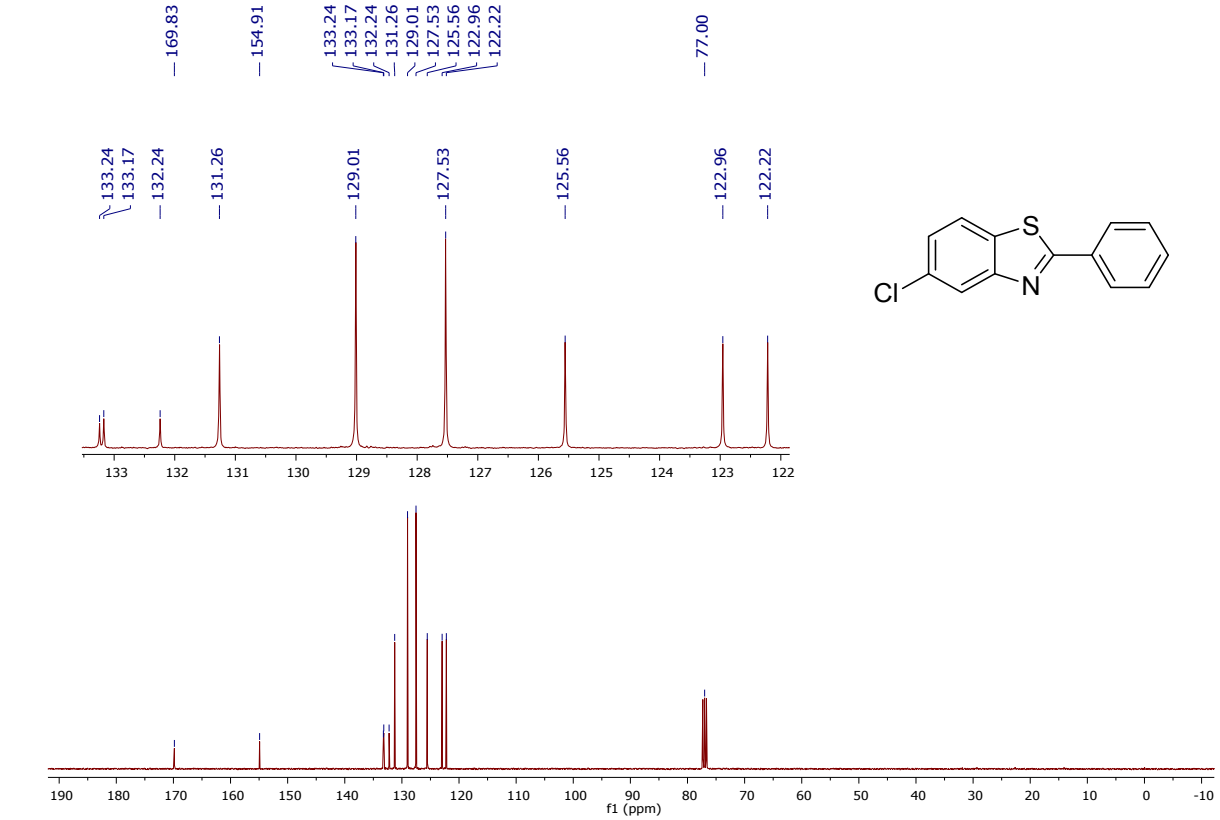
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2-phenylbenzo[d]thiazole (**3aa)**



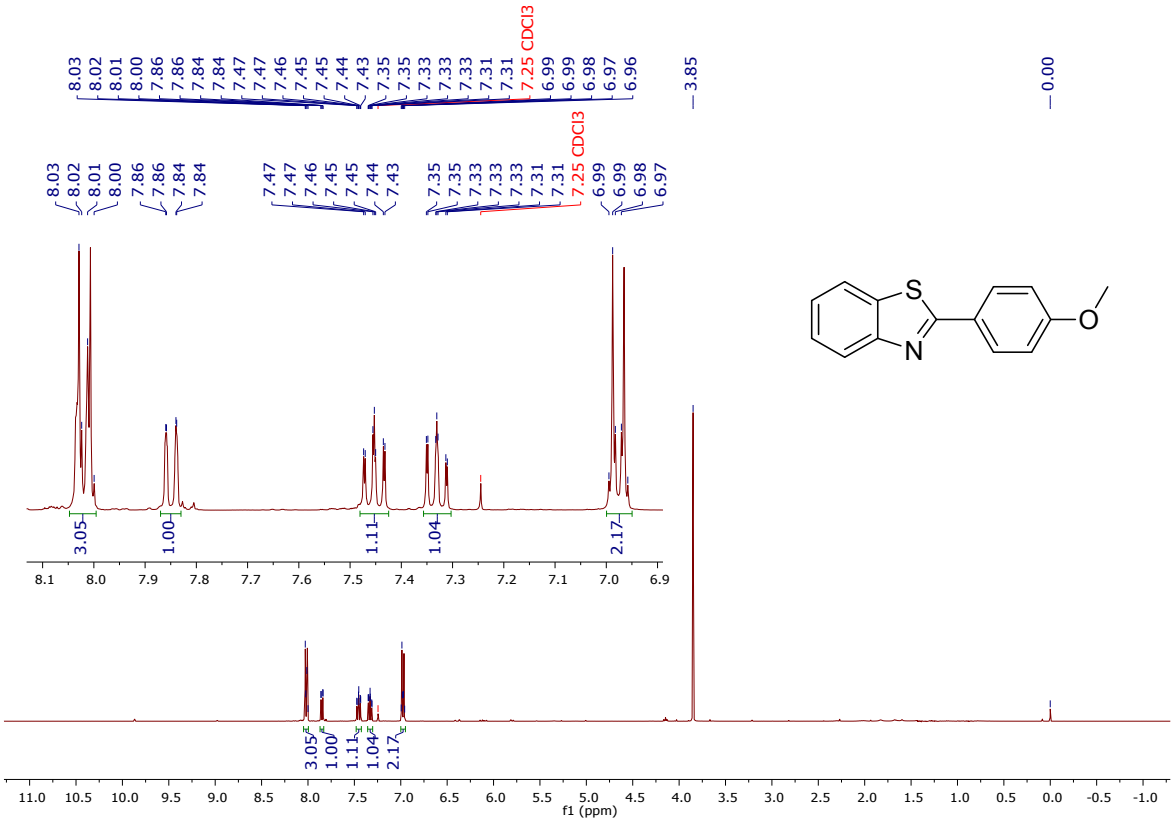
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2-phenylbenzo[d]thiazole (**3aa)**



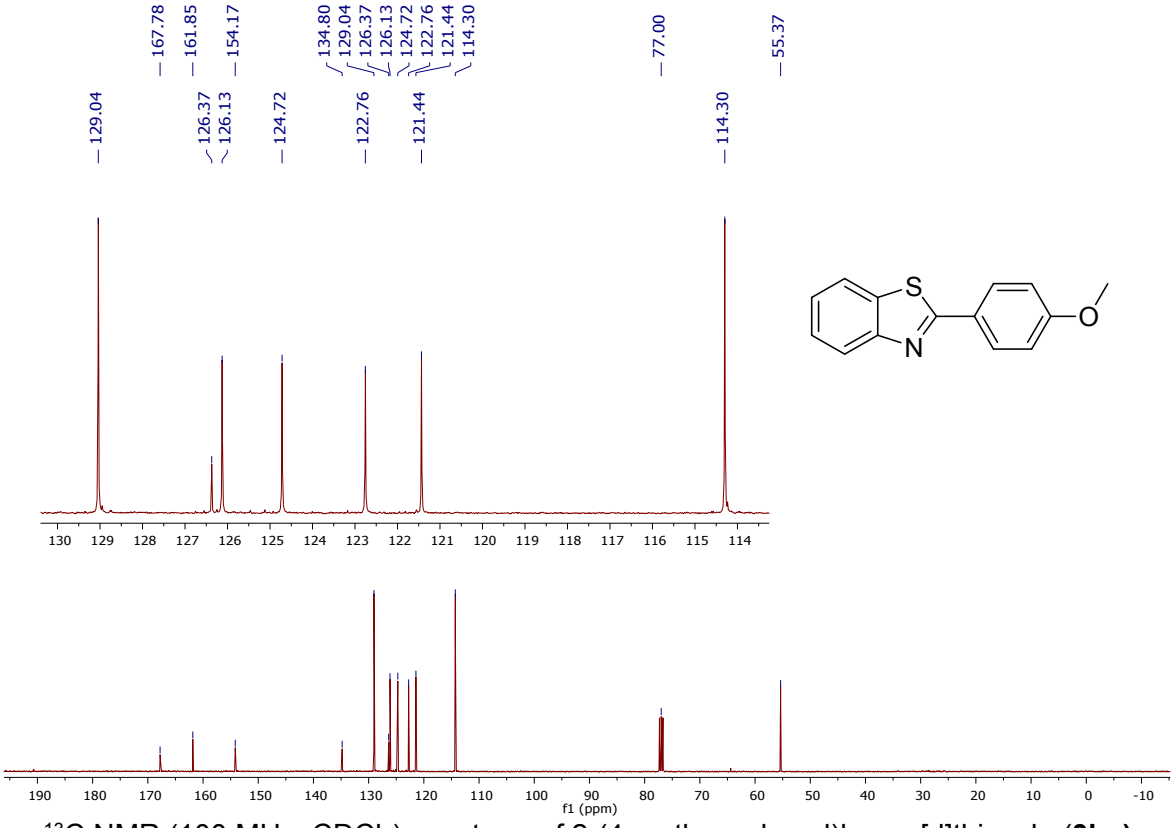
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-chloro-2-phenylbenzo[d]thiazole (**3ab)**



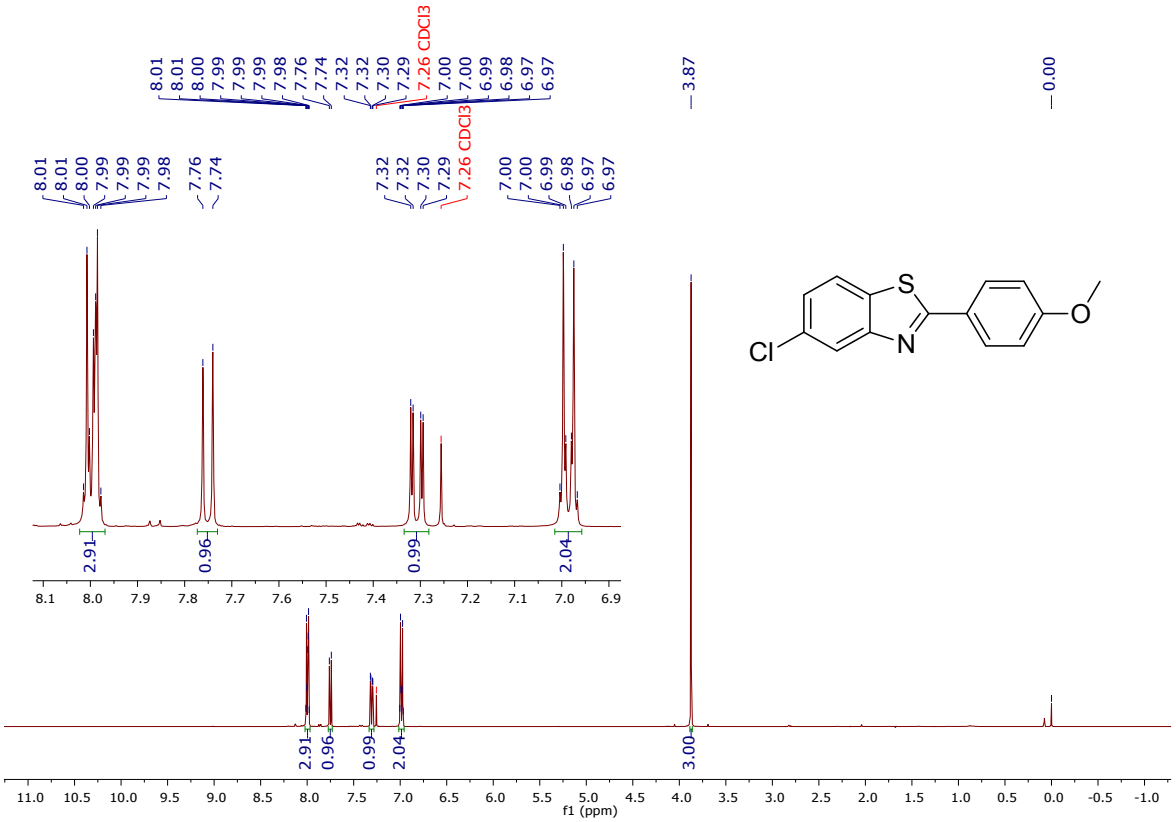
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 5-chloro-2-phenylbenzo[d]thiazole (**3ab)**



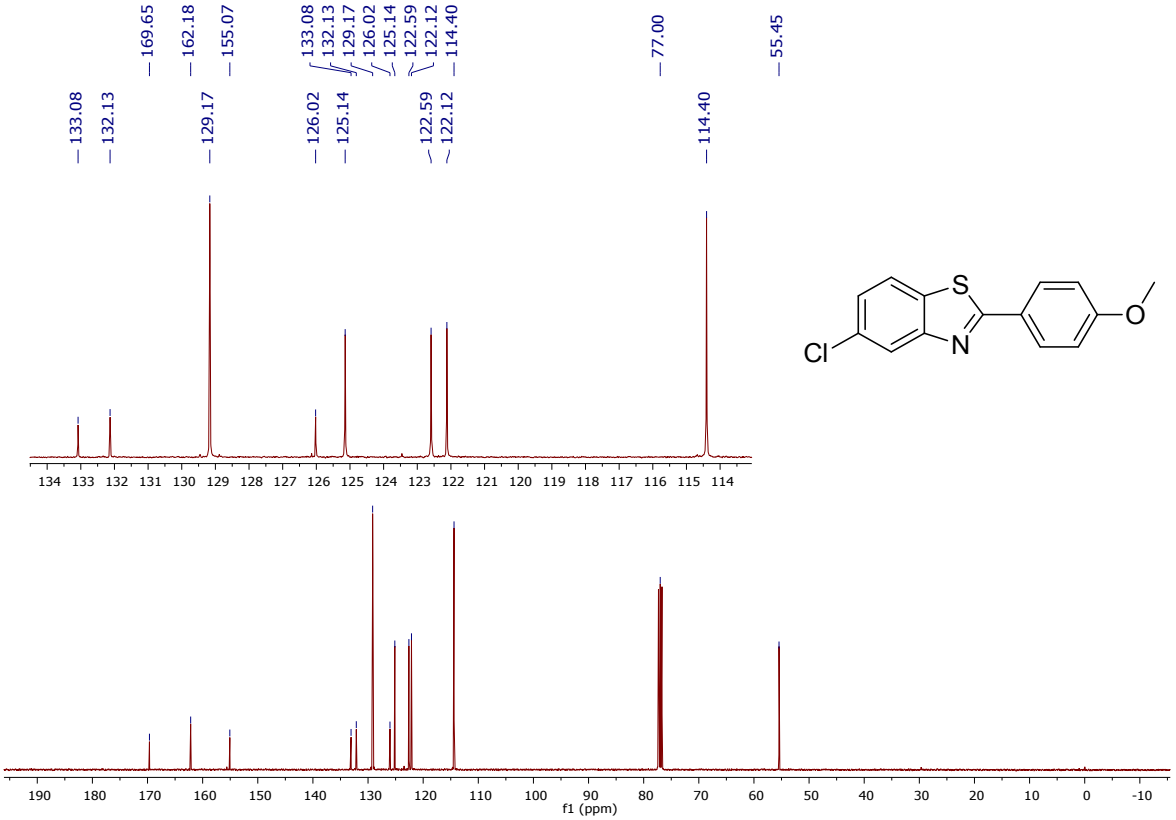
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2-(4-methoxyphenyl)benzo[d]thiazole (**3ba)**



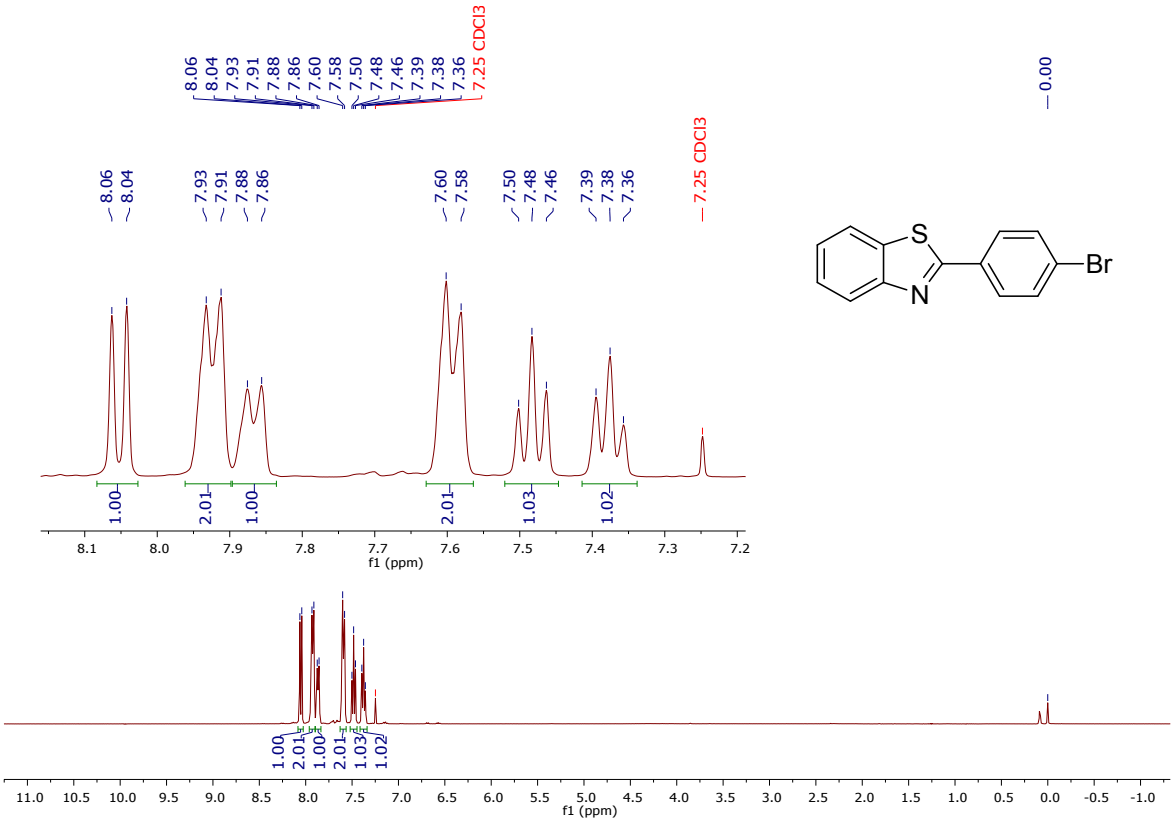
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2-(4-methoxyphenyl)benzo[d]thiazole (**3ba)**



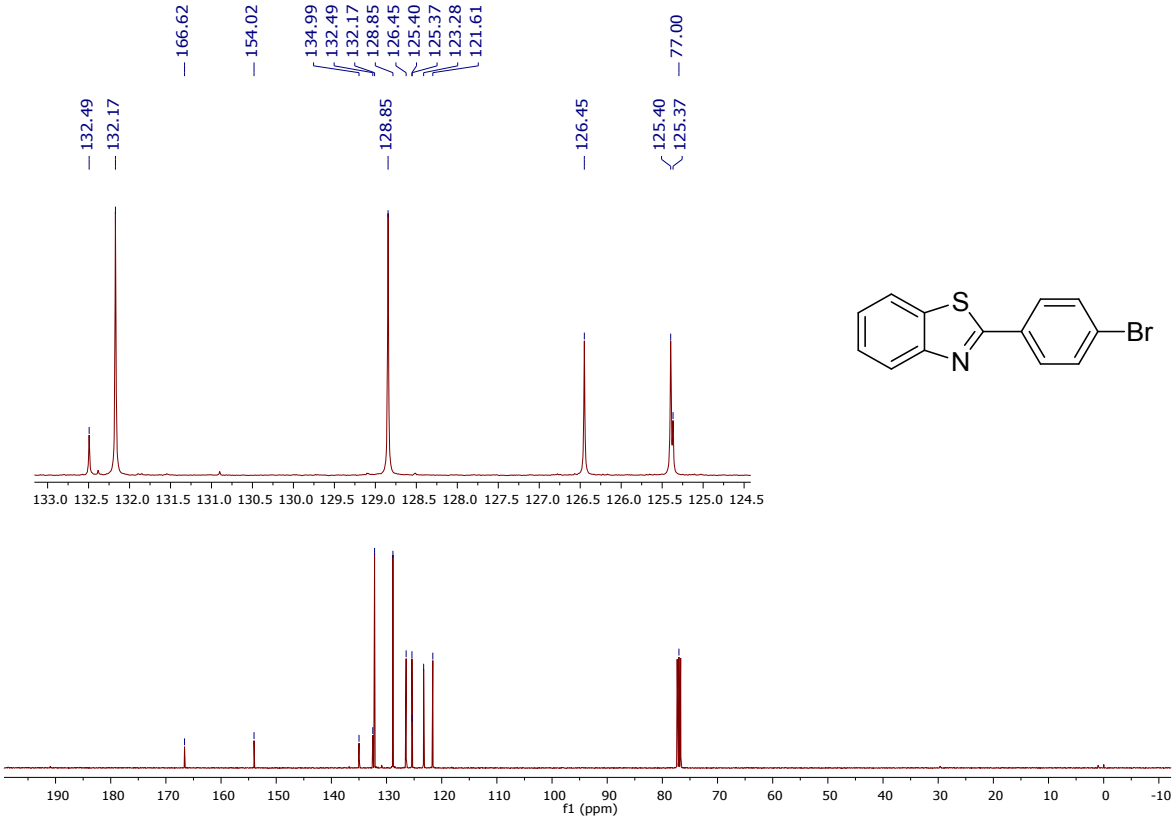
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-chloro-2-(4-methoxyphenyl)benzo[d]thiazole (**3bb)**



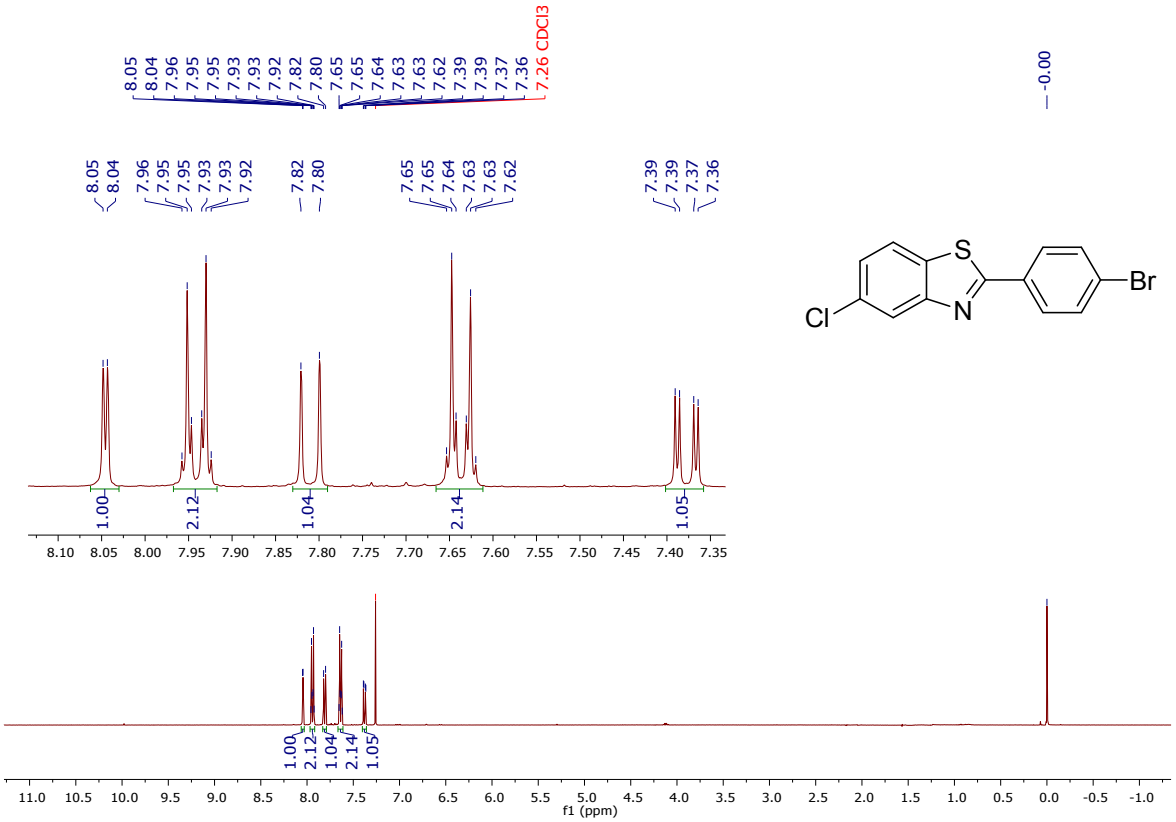
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 5-chloro-2-(4-methoxyphenyl)benzo[d]thiazole (**3bb)**



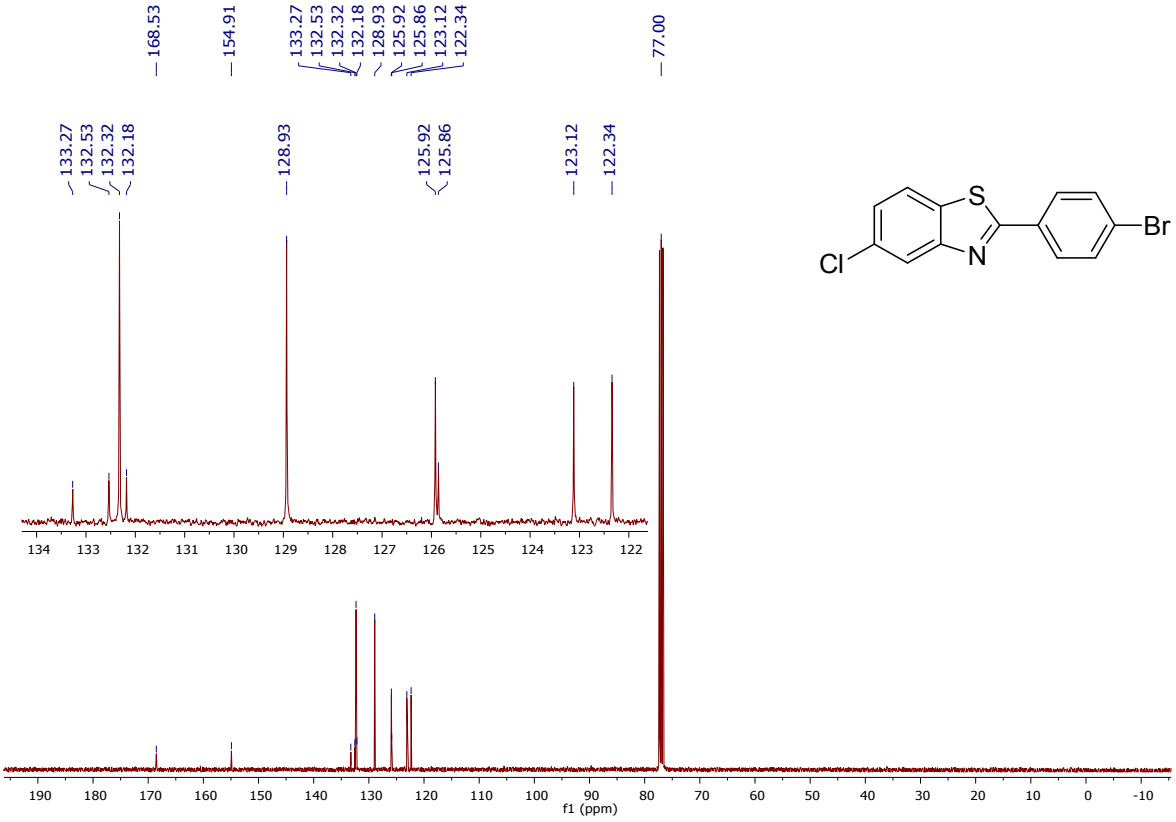
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2-(4-bromophenyl)benzo[d]thiazole (**3ca**)



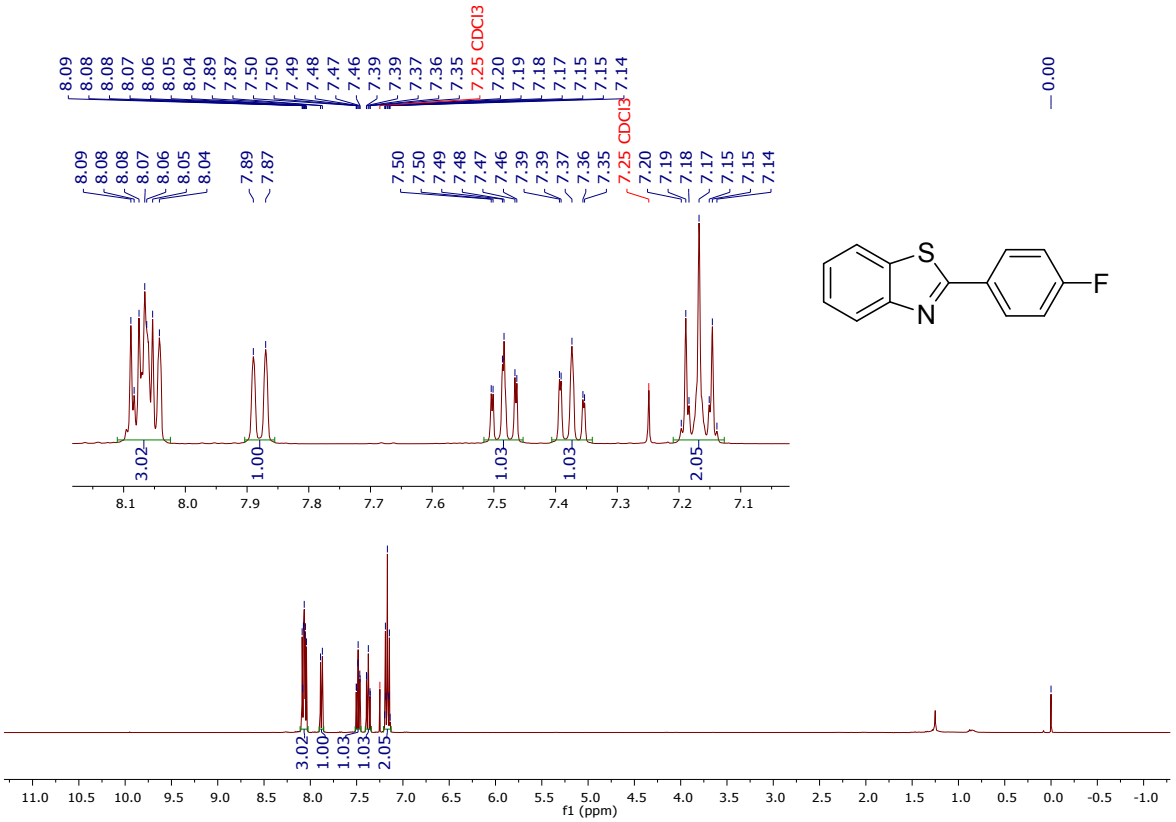
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2-(4-bromophenyl)benzo[d]thiazole (**3ca**)



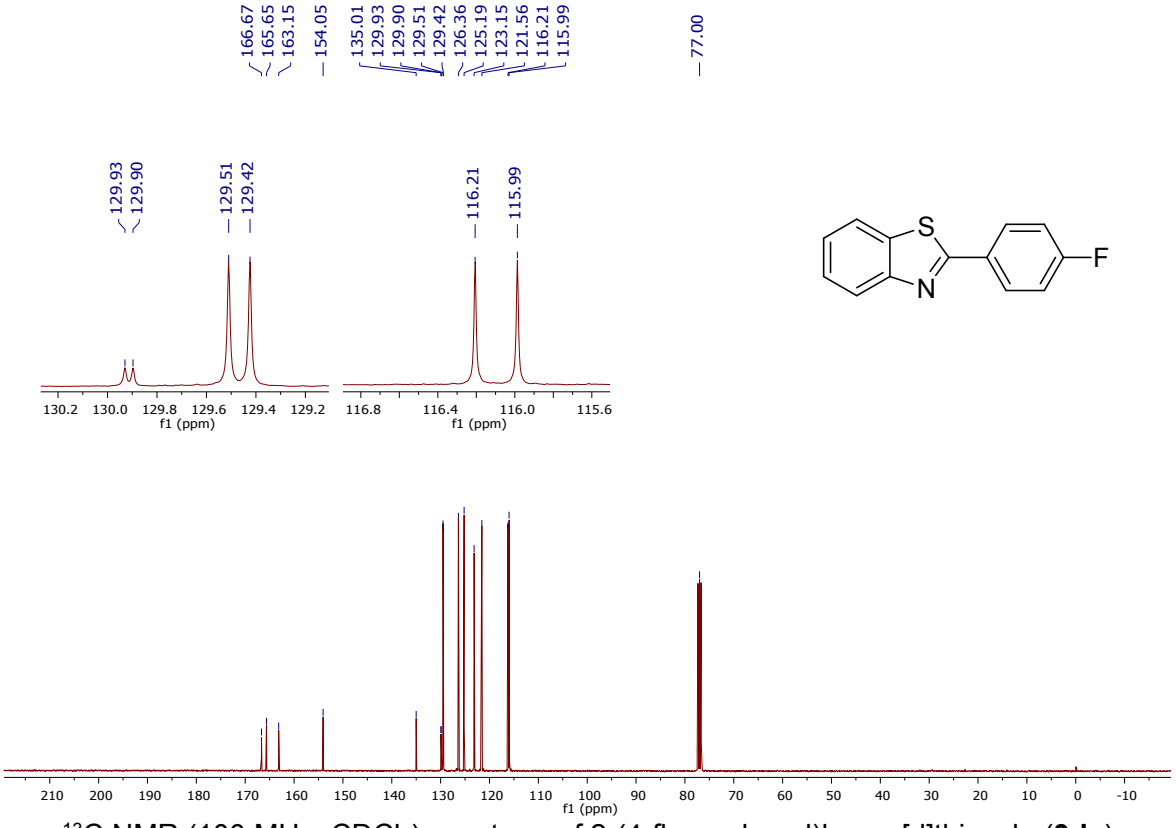
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2-(4-bromophenyl)-5-chlorobenzo[d]thiazole (**3cb**)



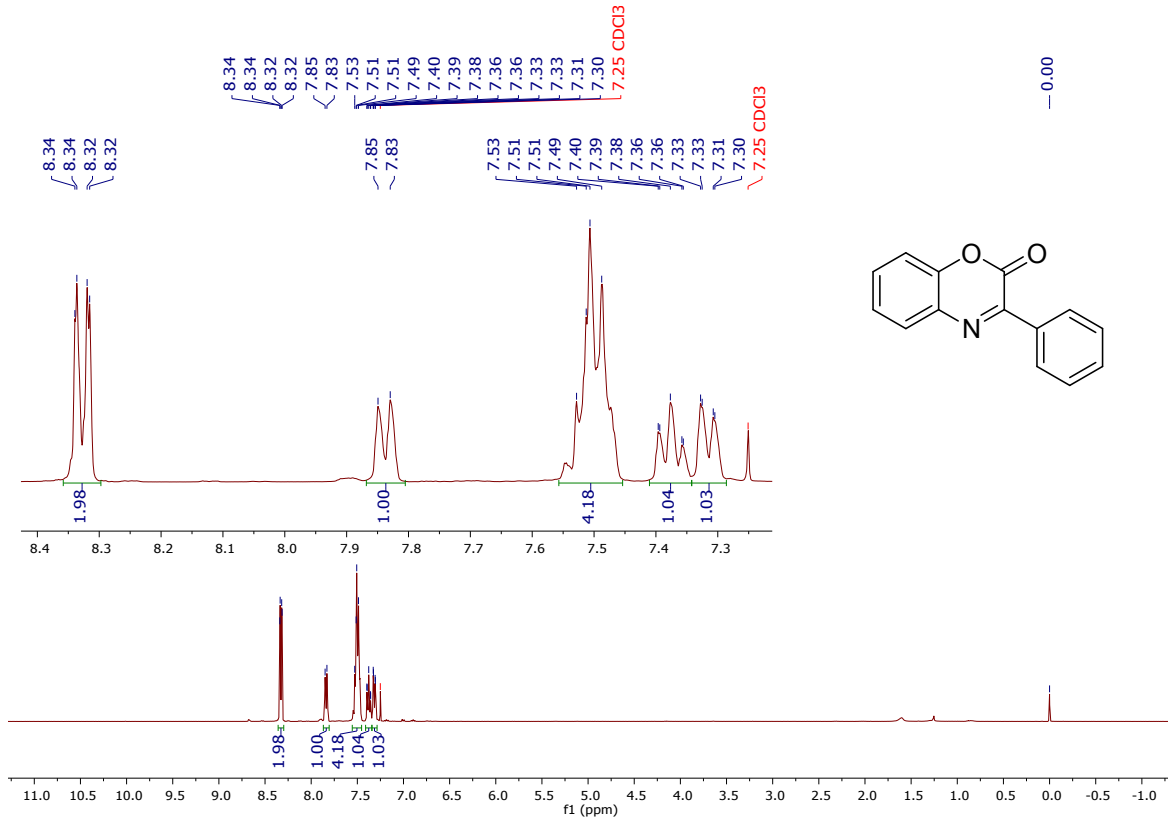
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2-(4-bromophenyl)-5-chlorobenzo[d]thiazole (**3cb**)



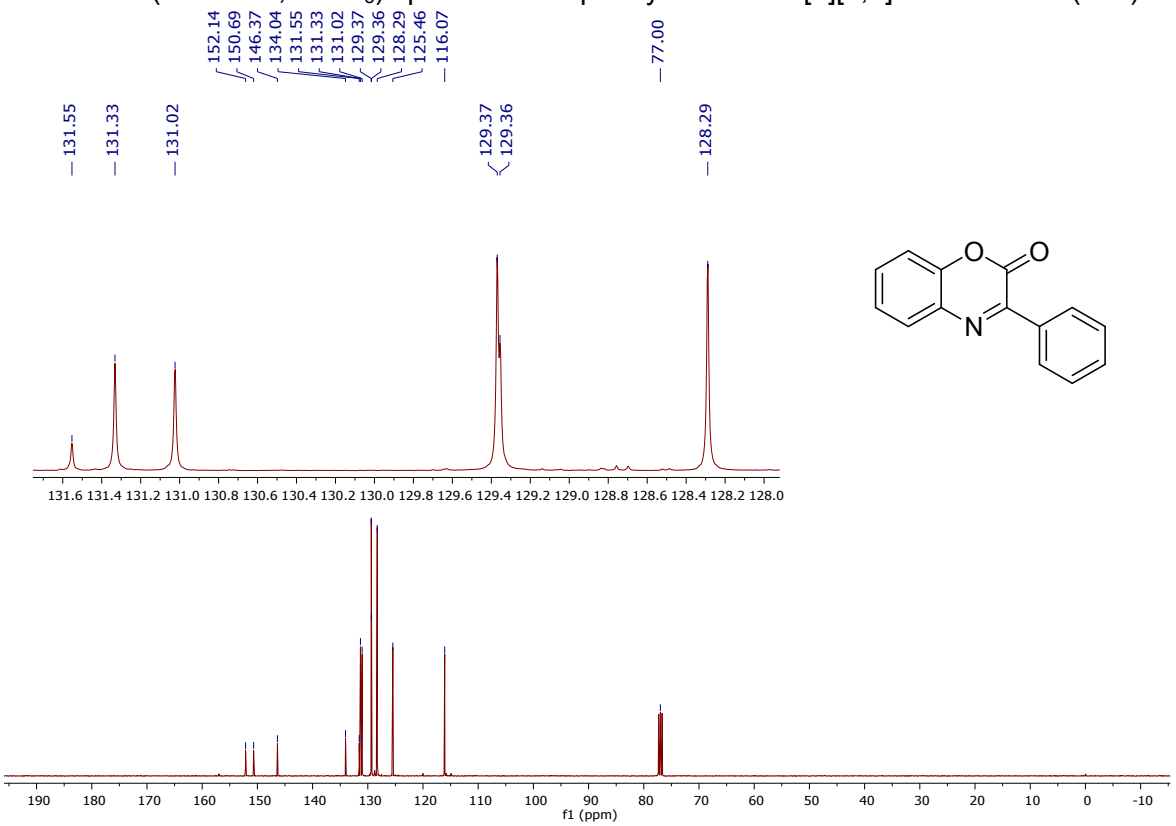
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2-(4-fluorophenyl)benzo[d]thiazole (**3da**)



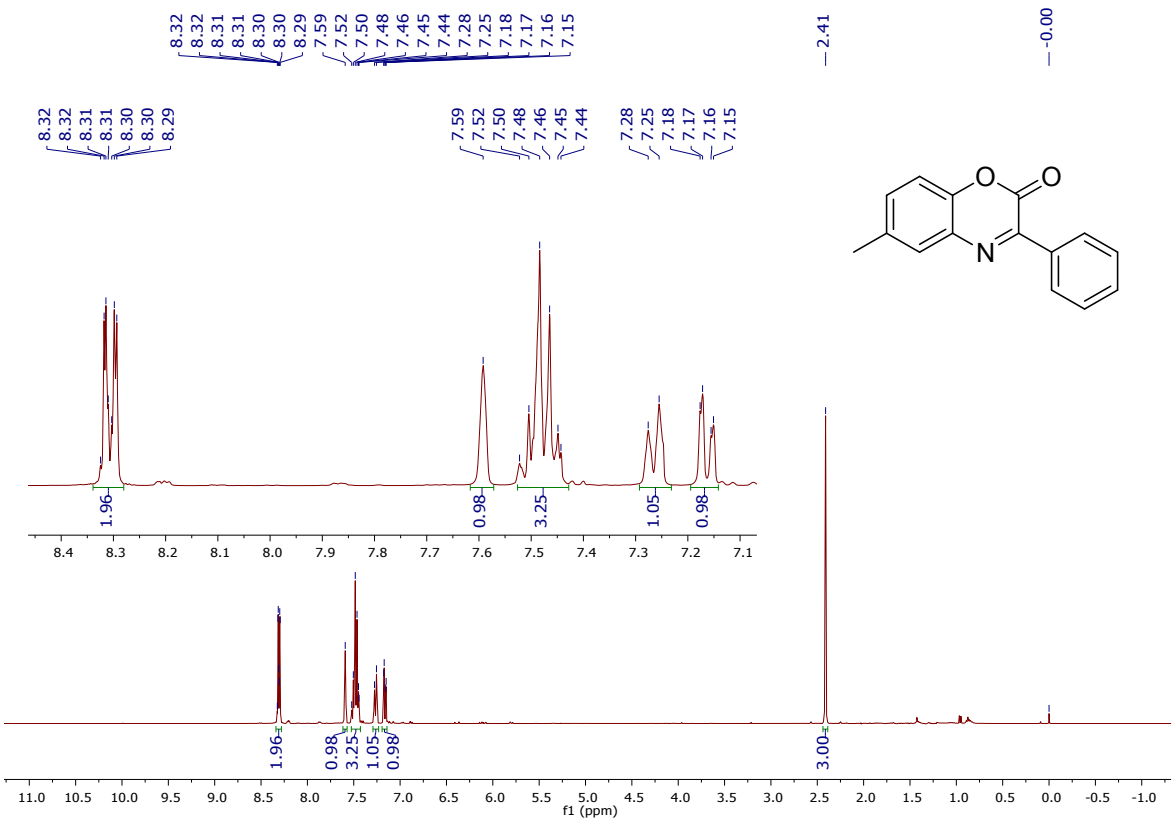
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2-(4-fluorophenyl)benzo[d]thiazole (**3da**)



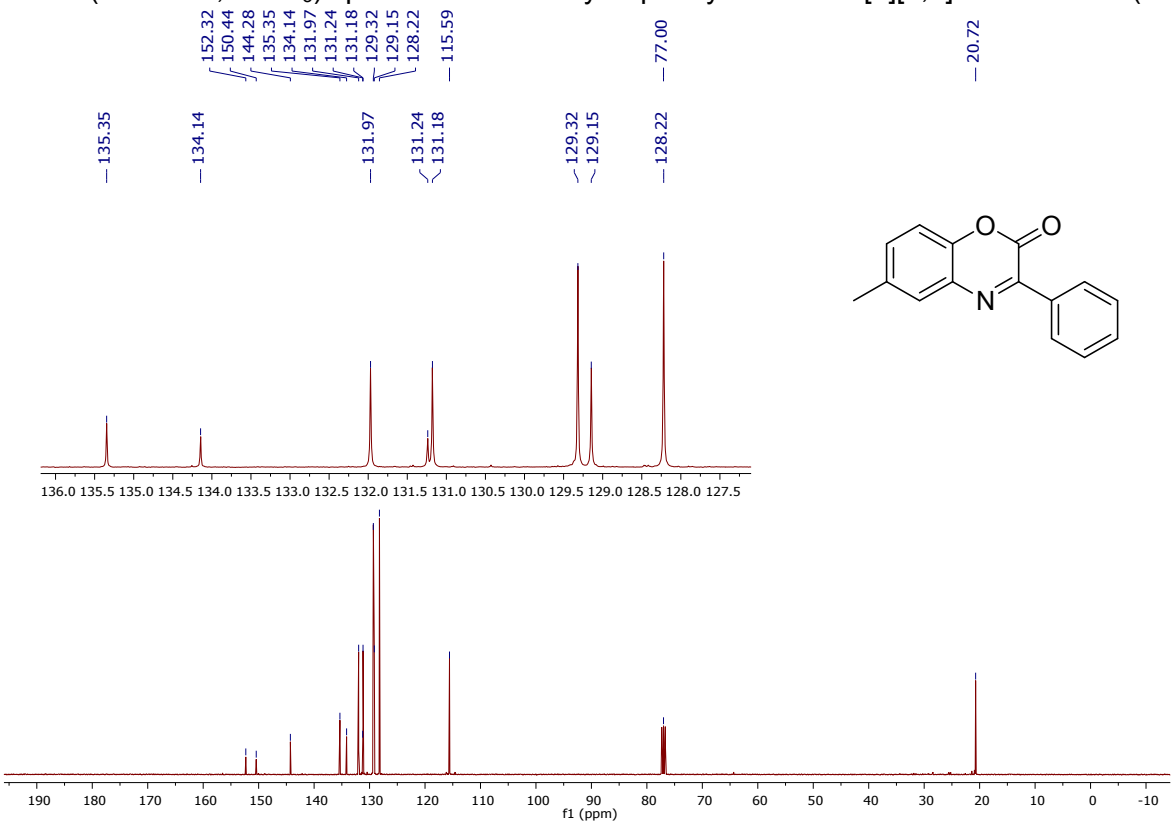
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5aa**)



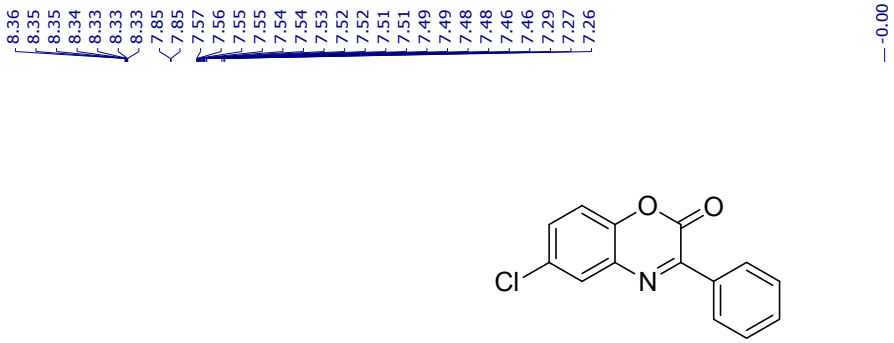
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5aa**)



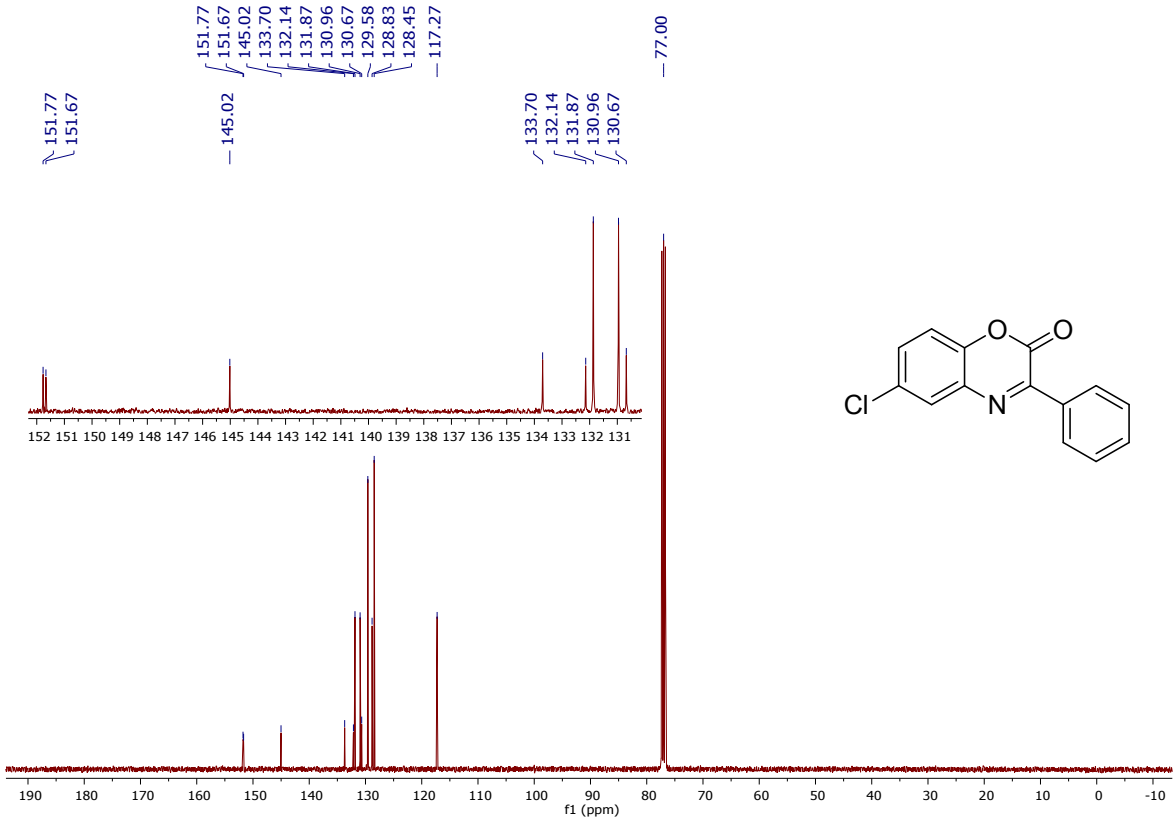
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 6-methyl-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ab**)



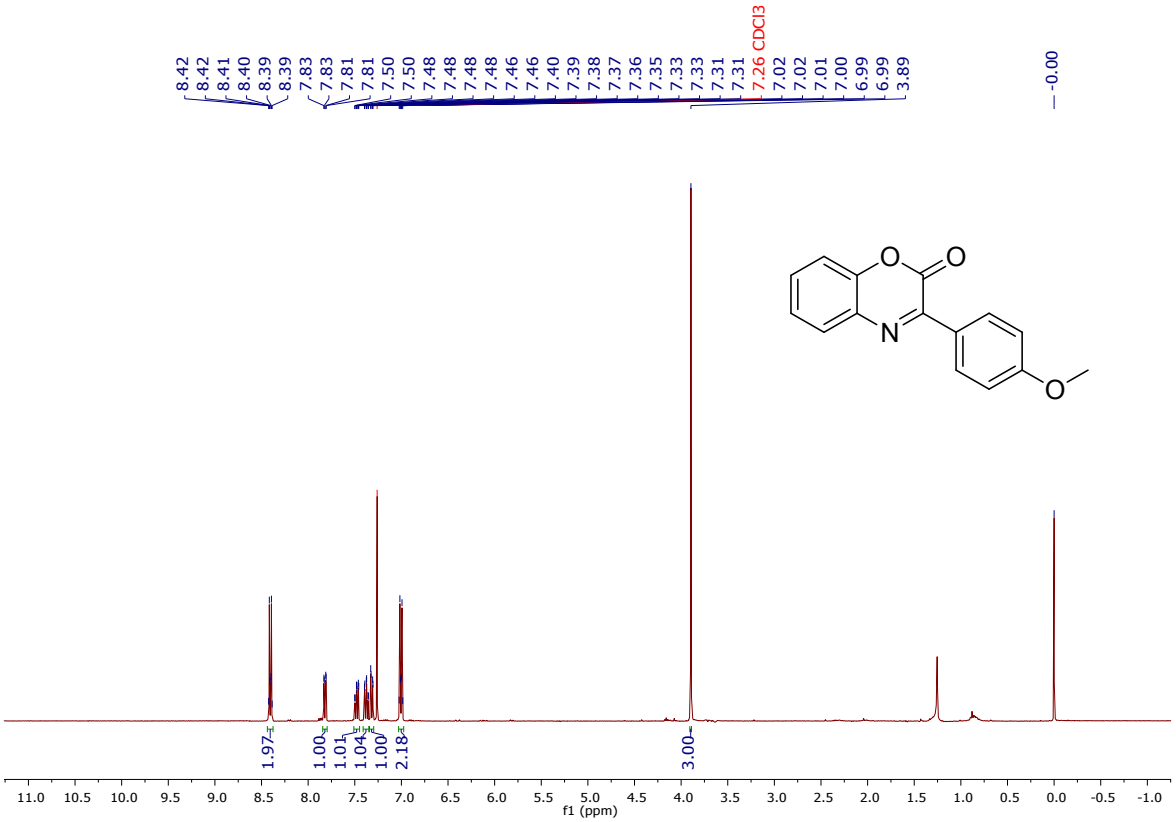
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 6-methyl-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ab**)



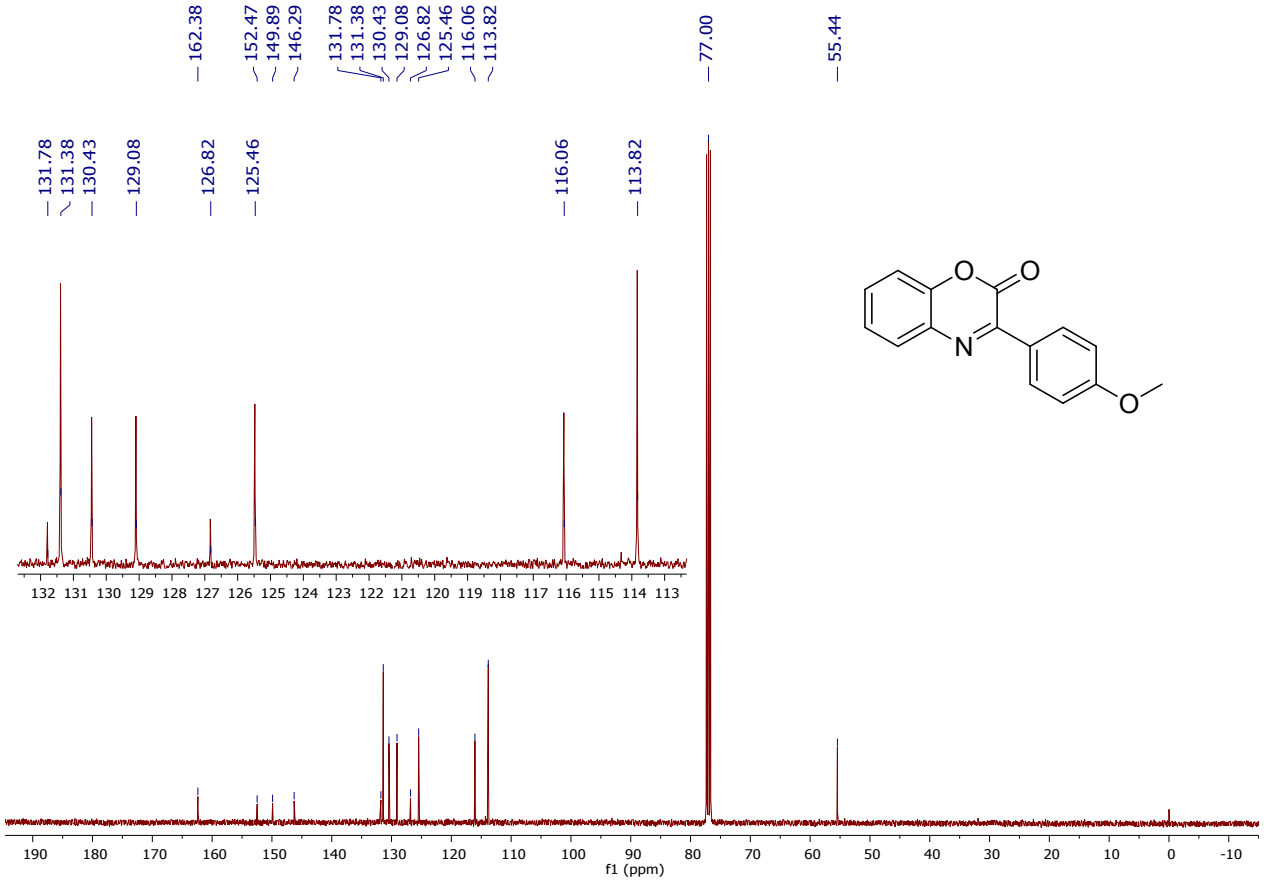
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 6-chloro-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ac**)



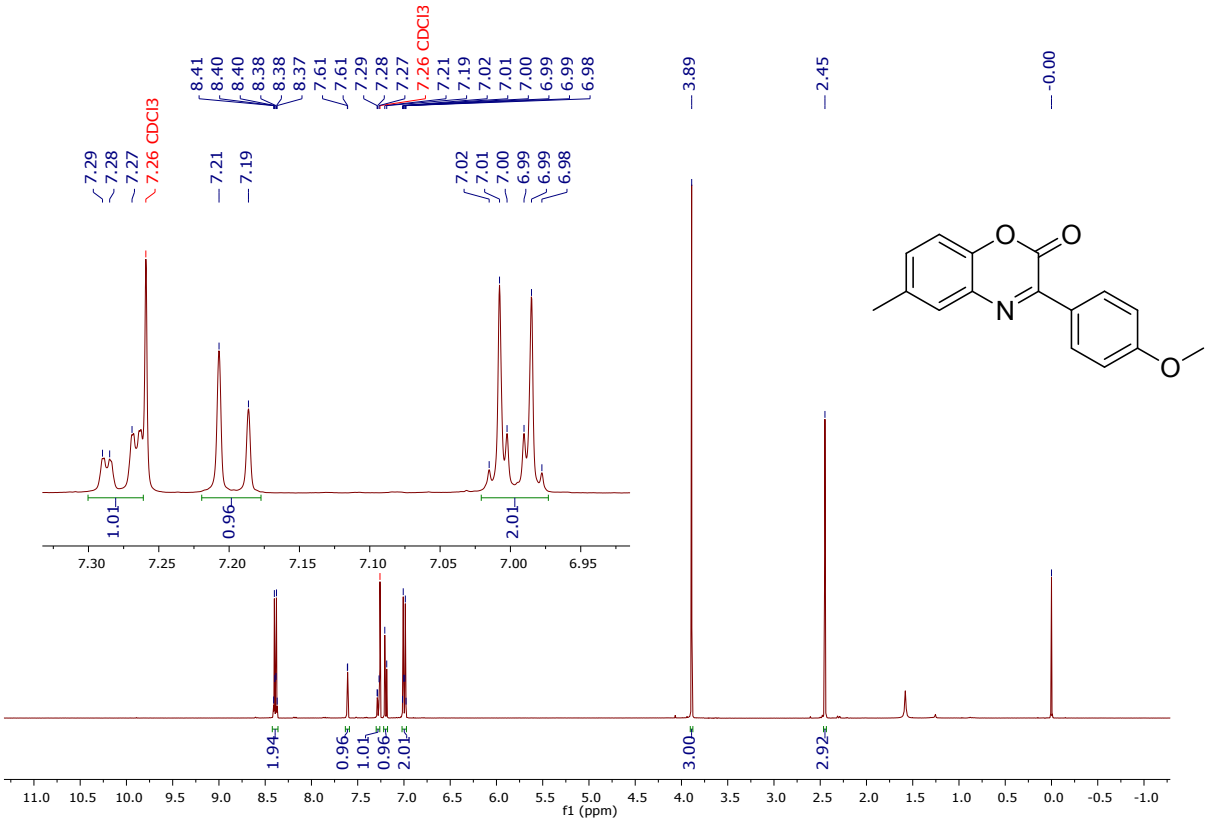
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 6-chloro-3-phenyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ac**)



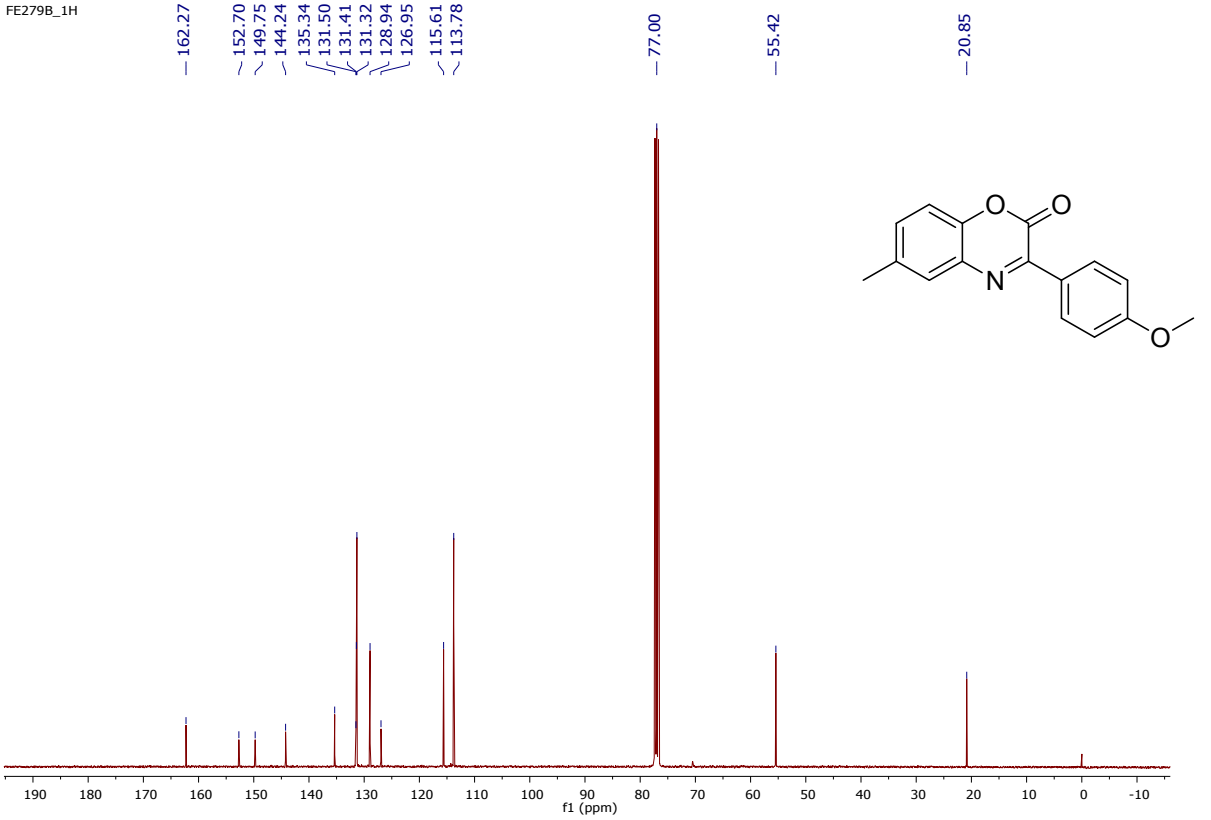
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(4-methoxyphenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ba**)



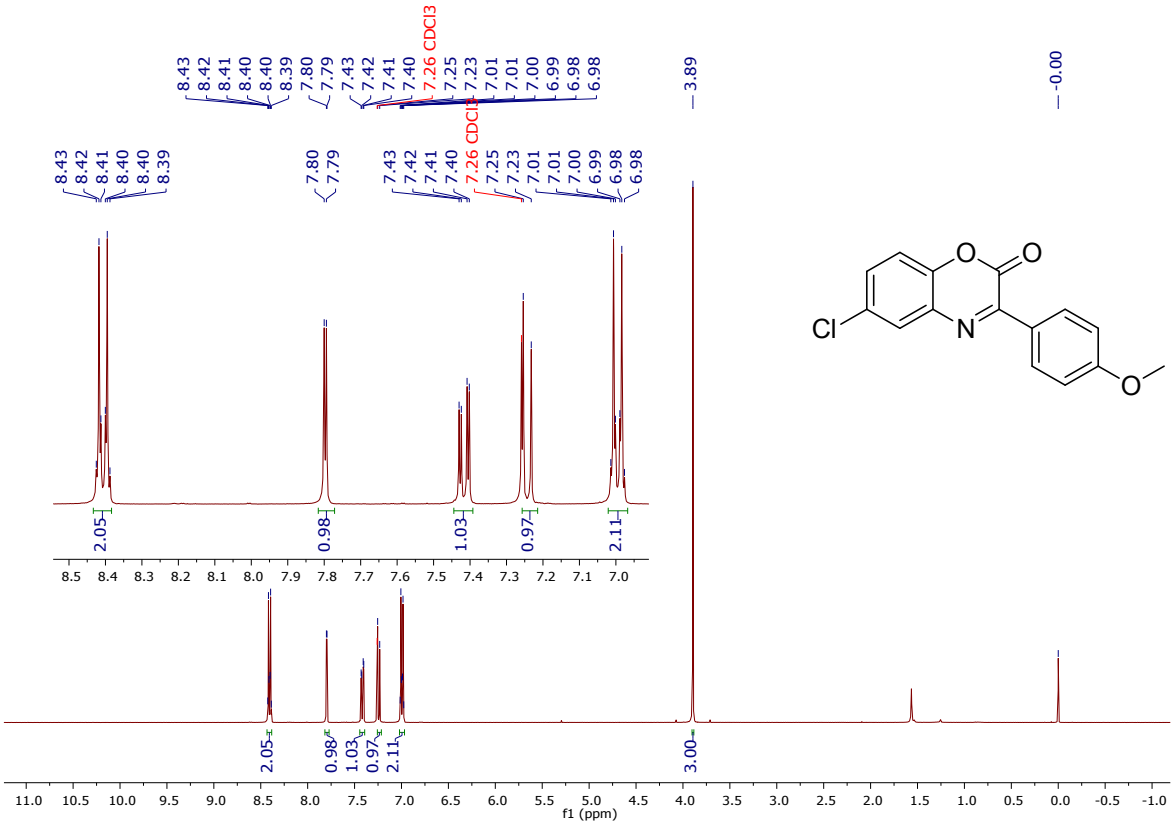
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3-(4-methoxyphenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ba**)



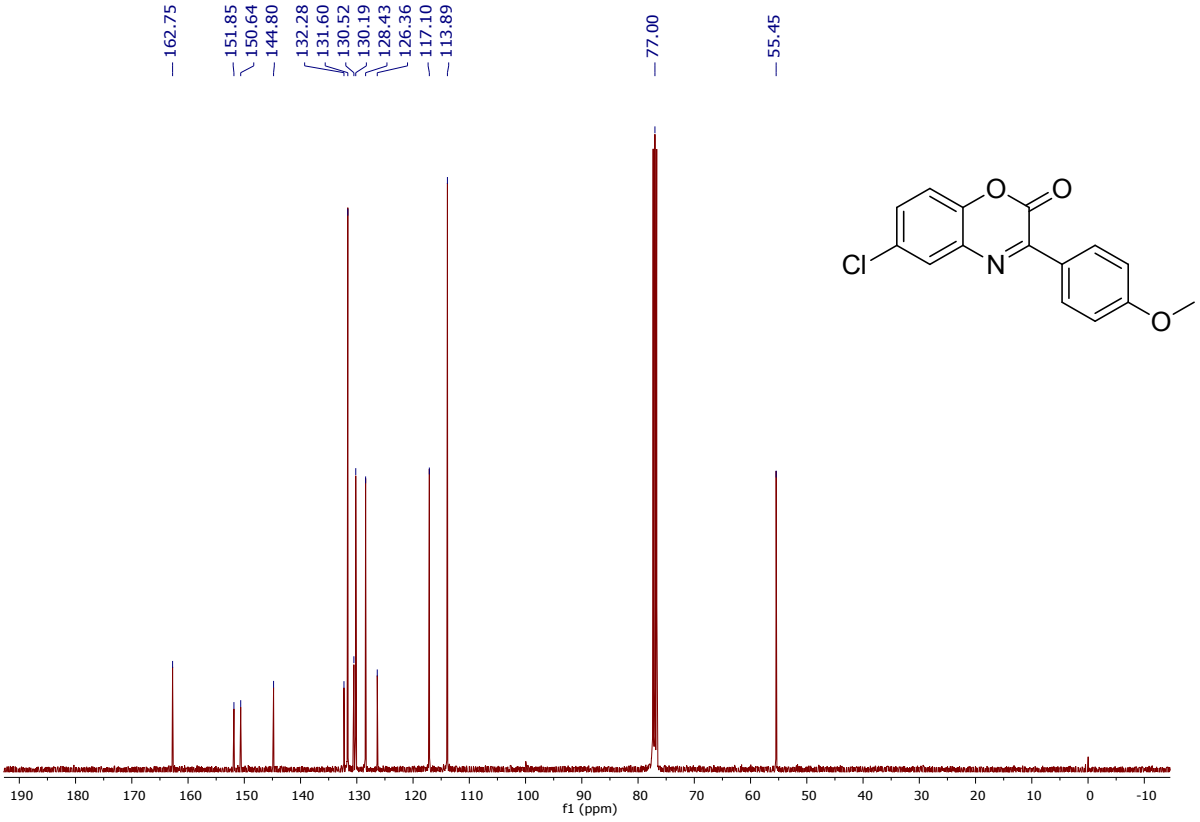
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(4-methoxyphenyl)-6-methyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5bb**)



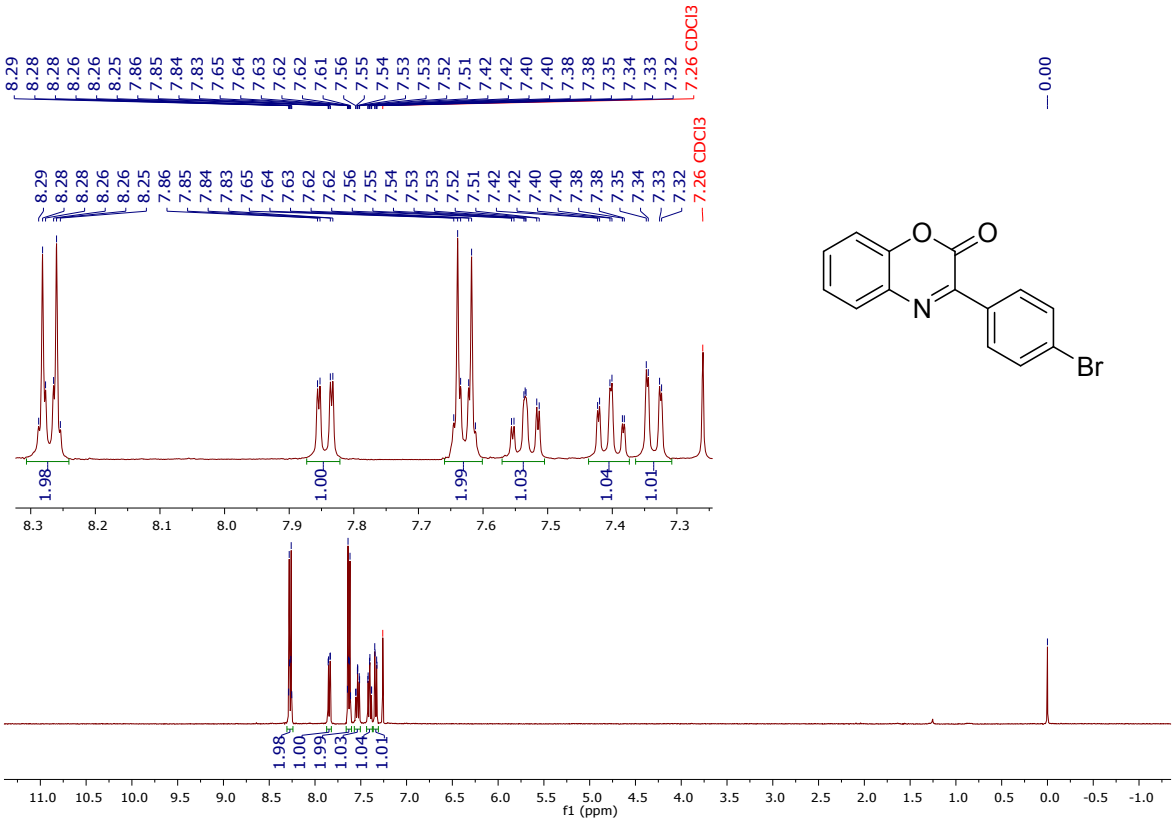
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3-(4-methoxyphenyl)-6-methyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5bb**)



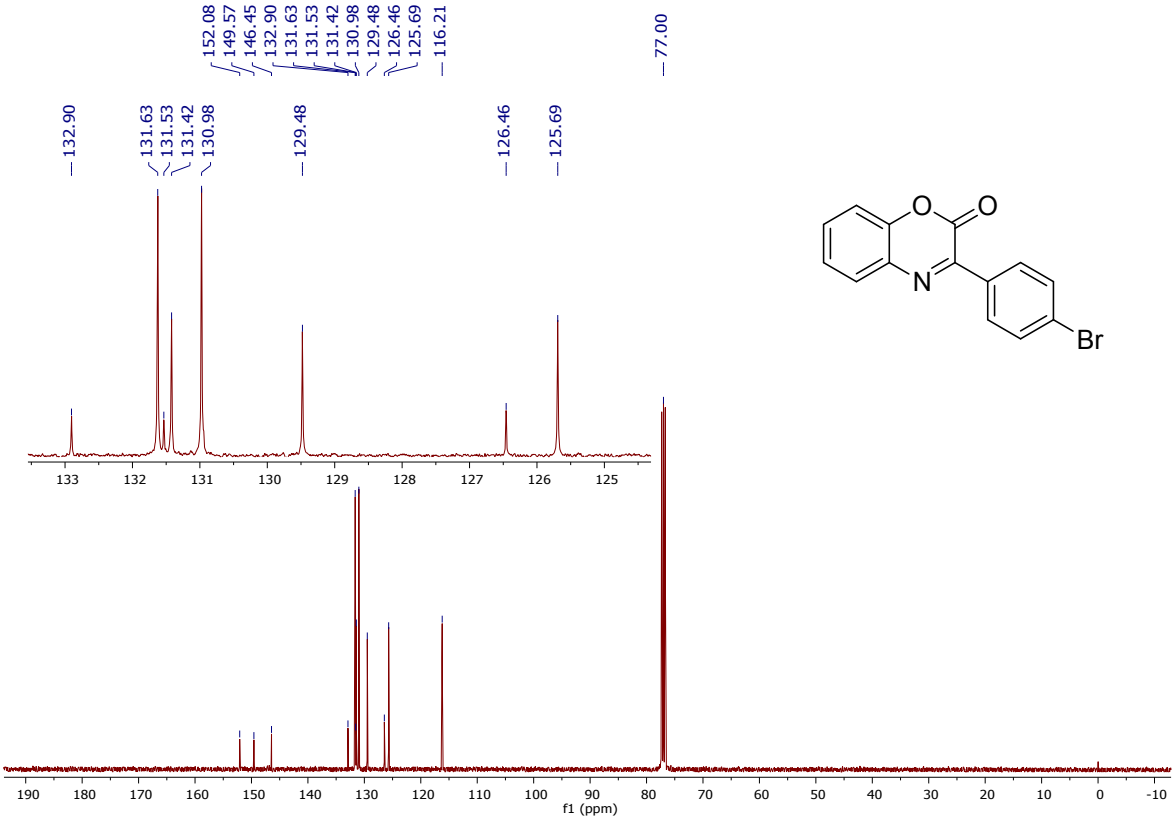
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 6-chloro-3-(4-methoxyphenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (**5bc**)



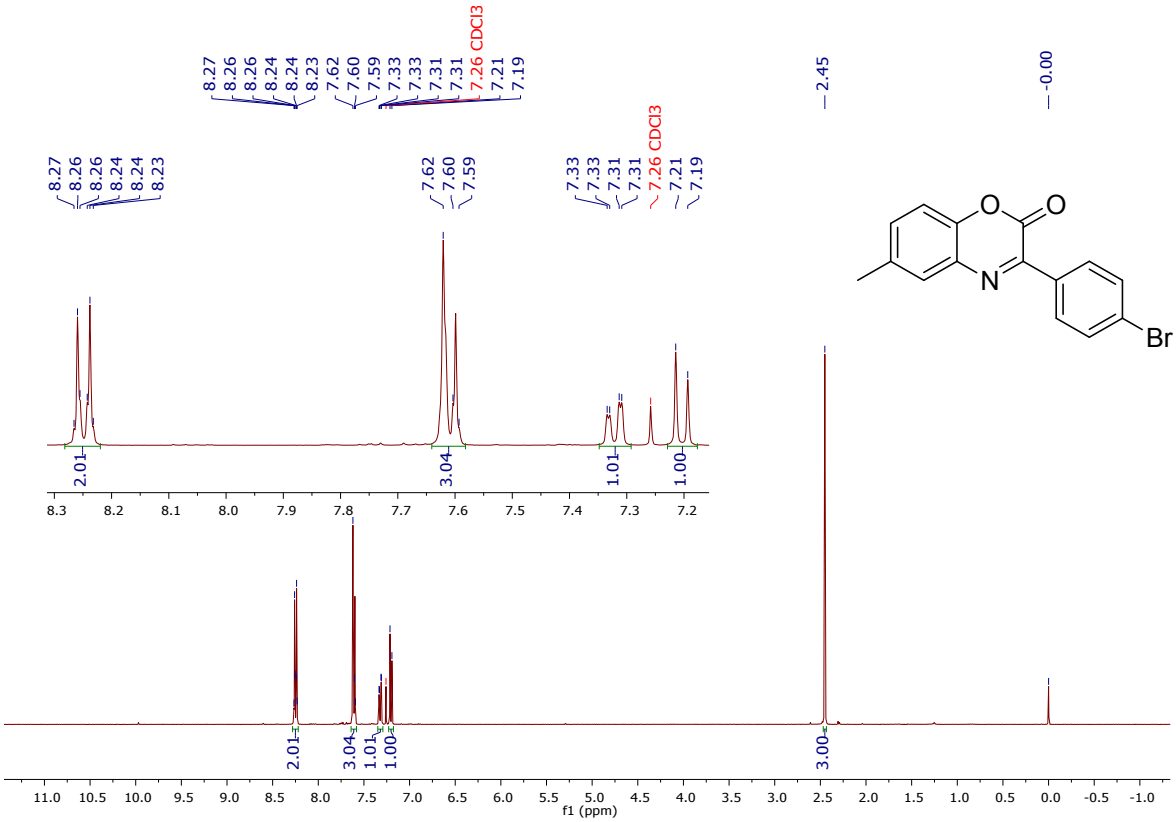
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 6-chloro-3-(4-methoxyphenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (**5bc**)



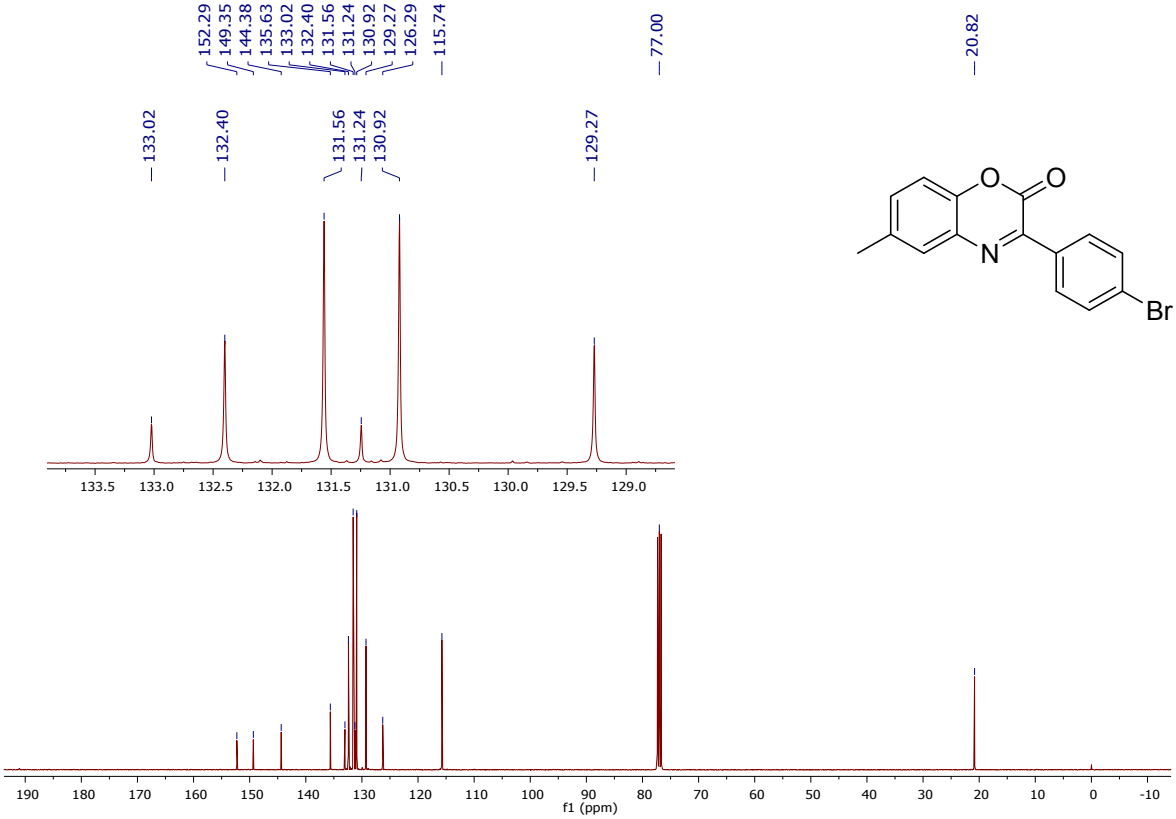
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(4-bromophenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ca**)



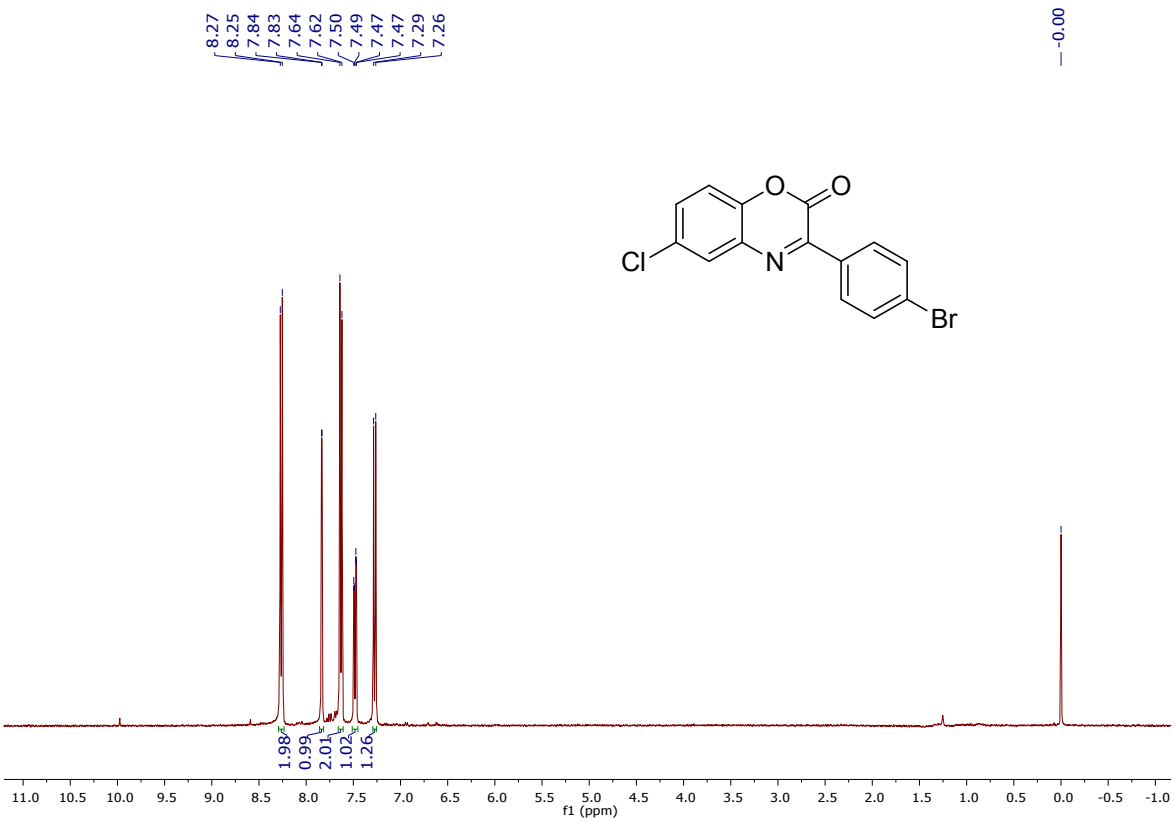
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3-(4-bromophenyl)-2*H*-benzo[*b*][1,4]oxazin-2-one (**5ca**)



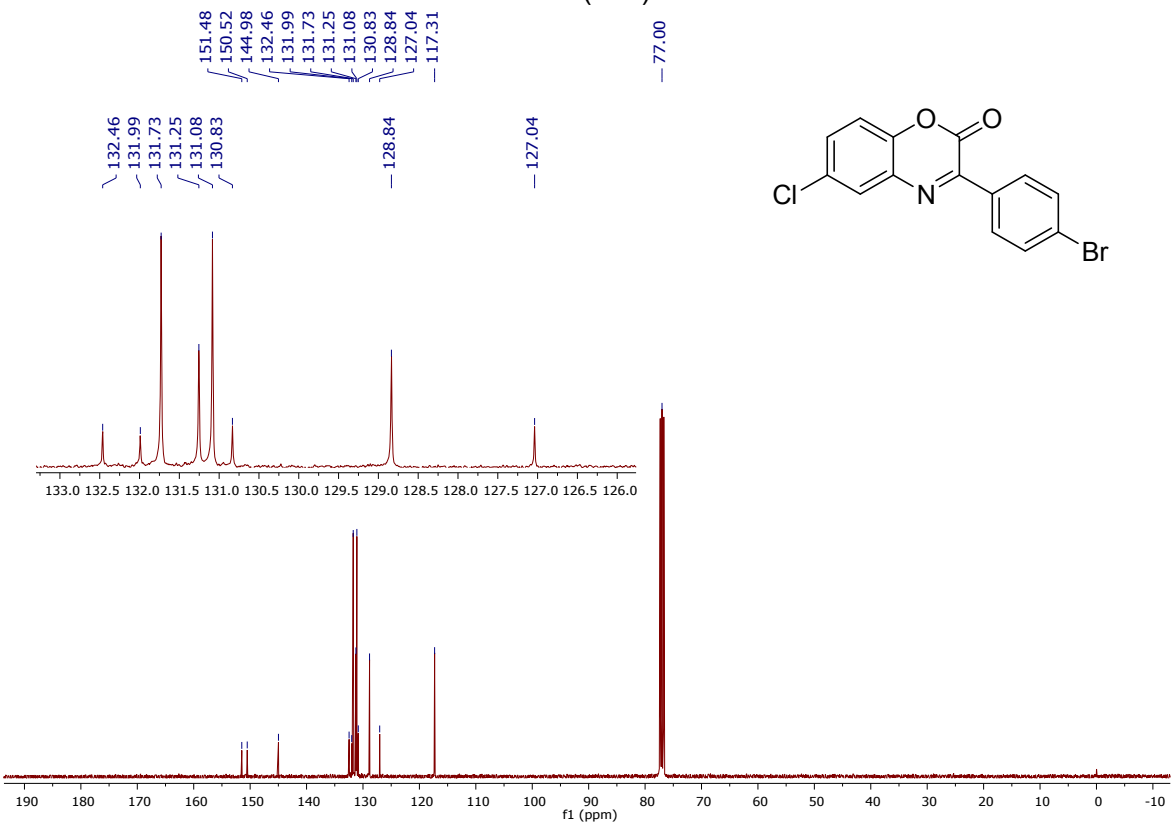
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(4-bromophenyl)-6-methyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5cb**)



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3-(4-bromophenyl)-6-methyl-2*H*-benzo[*b*][1,4]oxazin-2-one (**5cb**)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(4-bromophenyl)-6-chloro-2H-benzo[b][1,4]oxazin-2-one (**5cc**)



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3-(4-bromophenyl)-6-chloro-2H-benzo[b][1,4]oxazin-2-one (**5cc)**