Supporting Information

Photochemical Direct Alkylation of Heteroarenes with Alkanes, Alcohols, Amides, and Ethers

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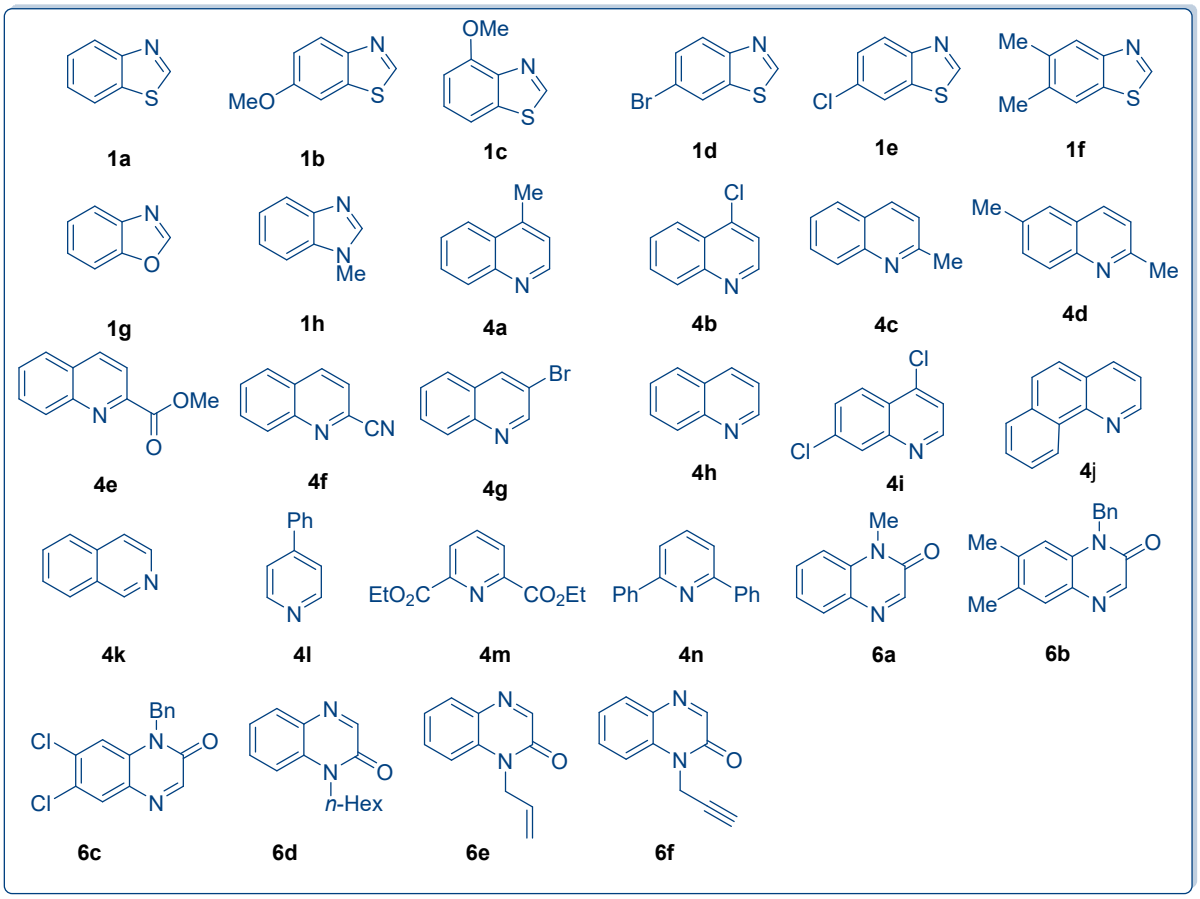
1. General Information

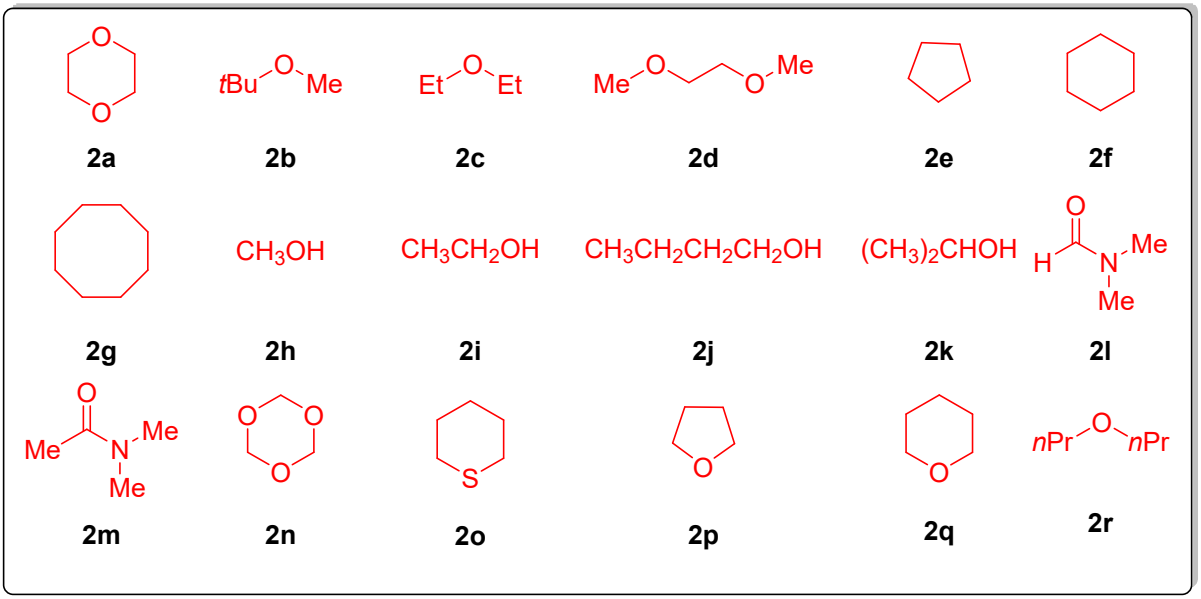
Unless otherwise mentioned, all catalysts, starting materials, and solvents were purchased from commercial sources (Sigma, TCI, Avra, SRL, Spectrochem, BLD Pharm) and used as received. All the reactions were carried out in a glass vial (10 mL) with magnetic stirring under air atmosphere in flame-dried glassware. In case air- or moisture-sensitive reagents were used, reactions were performed under N_2 atmosphere using standard Schlenk techniques. Yields refer to isolated compounds estimated to be > 95% pure, as determined by ^{1}H-NMR. Thin layer chromatogram (TLC) was performed on Merck TLC Silica gel 60 F254, TLC plates; detection under UV light at 254 nm. The column chromatographic purifications were performed using Silica gel (100−200 mesh ASTM) from Merck, if not mentioned otherwise. Melting points were determined in capillary tubes using Stuart melting point apparatus SMP10, the reported values are not corrected. Nuclear Magnetic Resonance (NMR) spectra ^{1}H NMR (400 MHz), ^{13}C NMR (101 MHz), ^{19}F NMR (471 MHz) with the Bruker AVANCE NEO 400 MHz spectrometer using TMS as an internal standard and $CDCl_3$Chemical shifts (δ) for ^{1}H and ^{13}C NMR spectra are given in ppm relative to tetramethylsilane (TMS) or the NMR solvents [δ 7.26 for ^{1}H (chloroform-d), δ 77.0 for ^{13}C (chloroform-d), ^{19}F-NMR spectra are not externally calibrated and chemical shifts is given relative to CCl_3F as received from the automatic data processing. High-resolution mass spectra (HRMS) were obtained from Orbitrap Elite Hybrid Ion Trap-Orbitrap (Thermo Fischer Scientific, Newington, NH, USA) Mass Spectrometer in electrospray ionization mode (ESI+). All IR spectra were recorded on the PerkinElmer Spectrum Two™ FT-IR-ATR device.

2. Starting materials used in this work

2.1 Heteroarenes



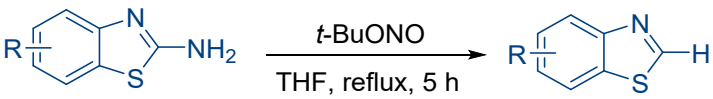
2.2. Alkane and Ethers



3. Experimental section: Starting material synthesis

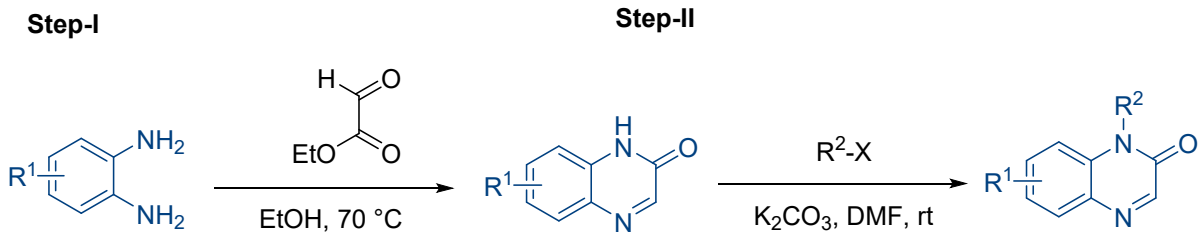
Heteroarenes **1a, 1g, 4a, 4b, 4c, 4d, 4e, 4f, 4g, 4h, 41, 4j, 4k, 4I, 4m, 4n** are commercially available and used as such without further purification (> 98%). The synthesized compounds were **1b, 1c, 1d, 1e, 1f, 6a, 6b, 6c, 6d, 6e, 6f**.

3.1. General procedure 1 (GP1): Synthesis of substituted benzothiazoles.



An oven-dried two-neck 100 mL round bottom flask initially fitted with a magnetic stir bar and condenser was added substituted 2-aminobenzothiazoles (16.0 mmol, 1.0 equiv) and 20 mL of tetrahydrofuran (THF) at room temperature. Then, *t*-BuONO (35.2 mmol, 2.2 equiv) was added dropwise over 10 mins at room temperature. After completion of the addition, the reaction mixture was refluxed for 5 h at 70 ˚C. Upon completion of the reaction time, the reaction mixture was cooled to room temperature and concentrated under reduced pressure using rotary evaporator. Then, the crude reaction mixture was purified by flash column chromatography on silica gel using EtOAc/*n*-hexane (5:95) to give the substituted benzothiazoles.[1]

3.2. General procedure 2 (GP2): Synthesis of substituted quinoxaline-2(1*H*)-one

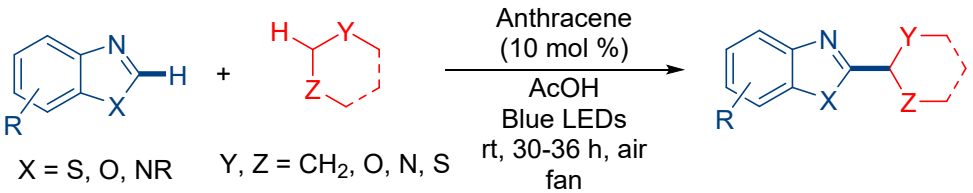


Step-I: A two-neck round bottom flask fitted with a magnetic stir bar and a condenser was added various substituted 1,2-phenylenediamine (20 mmol, 1.0 equiv) and ethanol (40 ml). To the resultant solution, ethyl glyoxylate in toluene 40% (24 mmol, 1.2 equiv) was added dropwise, and the reaction mixture was stirred at 70 ˚C for 1 h and then allowed to stir at room temperature for 12 h. After completion of the reaction time, the reaction mixture was filtered, and the solid filtrate was subsequently washed with ethanol (20 ml). Without further purification, the obtained solid quinoxaline-2(1*H*)-one was dried and used for the next step.[1]

Step-II: An oven-dried 100 mL round bottom flask fitted with a magnetic stir bar was charged with quinoxaline-2(*1H*)-one (10 mmol, 1.0 equiv), K_2CO_3 (1.65 g, 1.2 equiv) and DMF (16 mL). Alkyl halide (1.6 equiv) was added to this solution. Then, the reaction mixture was allowed to stir at room temperature for 12 h. The resulting reaction mixture was quenched with saturated ammonium chloride solution (5 mL) and extracted with EtOAc (3 x 20 mL). The combined organic solution was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The pure product N-alkylated quinoxaline-2(*1H*)-one was obtained by column chromatography on silica gel using EtOAc/*n*-hexane (2:1) as the eluent.[1]

4. Experimental section: Photochemical alkylation of heteroarenes

4.1. General procedure 3 (GP3): Visible light-induced alkylation of azoles with unactivated alkanes, alcohols, alkylamides, and ethers



X = S, O, NR

1a-1h

Y, Z = CH_2, O, N, S

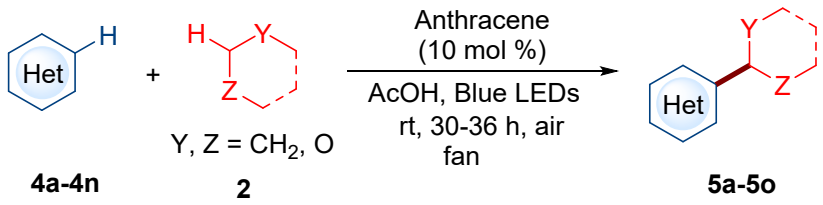
2

3a-3v

To a 15 mL Schlenk flask initially fitted with a magnetic stir bar and septa were added heteroarene **1a-1h** (0.4 mmol) and anthracene (10 mol %), followed by acetic acid (15 equiv). Then, alkane/ether **2** (1.0 mL) solvent was used. The resulting reaction mixture was irradiated using PR-160-427 nm Kessil LEDs and stirred at room temperature for 30-36 h under air cooling (fan). After the reaction time, the mixture was quenched with saturated aqueous NaHCO3 (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic solution was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc as the eluent to give alkylated heteroarenes **3a-3v** as pure product.

Figure S1. Photochemical reaction setup using Kessil PR160L-427 nm LED lights.

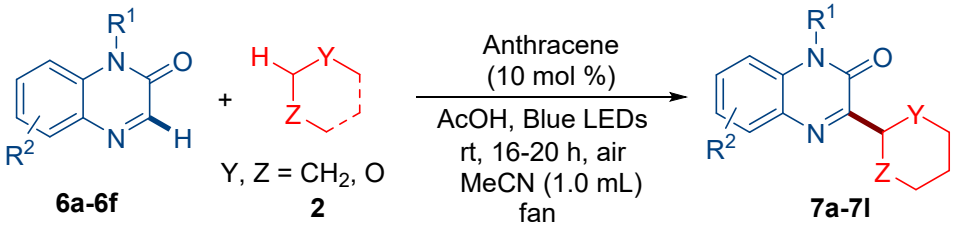
4.2. General procedure 4 (GP4): Synthesis of alkylated quinolines, isoquinolines, and pyridines



To a 15 mL Schenk flask initially fitted with a magnetic stir bar and septa were added heteroarene **4a-4n** (0.4 mmol) and anthracene (10 mol%), followed by trifluoroacetic acid (5.0 equiv). Then, alkane/ether **2** (1.0 mL) was added. The resulting reaction mixture was irradiated using PR160L-427 nm Kessil LEDs with stirring at room temperature for 30-36 h under air

cooling (fan). After the reaction time, the mixture was quenched with saturated aqueous NaHCO3 (10 mL). The resulting aqueous solution was extracted with EtOAc (10 x 3 mL), and the combined organic solution was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc (7:3) as the eluent to give alkylated heteroarenes **5a-5o** as pure product.

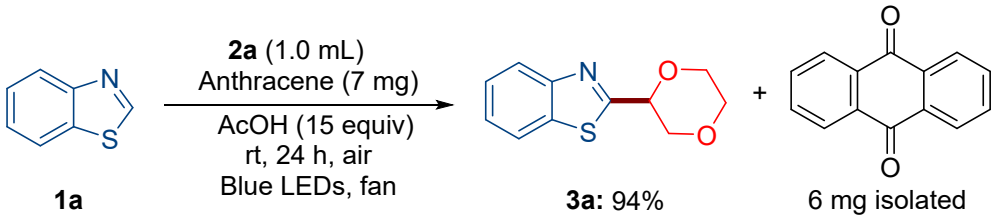
4.3. General procedure 5 (GP5): Synthesis of alkylated quinoxaline-2(1*H*)-ones



To a 15 mL Schlenk flask initially fitted with a magnetic stir bar and septa were added quinoxaline-2(*1H*)-ones **6a-6f** (0.3 mmol) and anthracene (10 mol %). Then acetic acid (10 equiv), alkane/ether (1.0 mL), and MeCN (1.0 mL) were added to the reaction mixture via a syringe. The resulting reaction mixture was irradiated using PR160L-427 nm Kessil LEDs and stirred at room temperature for 16 to 20 hours. After completion of the reaction time, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc (7:3) as the eluent to give alkylated heteroarenes **7a-7l** as pure product.

5. Mechanistic studies

5.1. Isolation of anthraquinone from the reaction mixture



To a 15 mL Schenk flask initially fitted with a magnetic stir bar and septa were added benzothiazole **1a** (0.4 mmol) and anthracene (7.0 mg, 10 mol %), followed by acetic acid (15 equiv). Then ether **2a** (1.0 mL) and AcCN (1.0 mL) as solvent was used. The resulting reaction mixture was irradiated using PR160L-427 nm Kessil LEDs with stirring at room temperature for 30 h. Upon completion of the reaction time, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc (9:1) as the eluent to give alkylated heteroarenes **3a** 94% along with anthraquinone **PC4** (76%, 6.0 mg) as pure product.

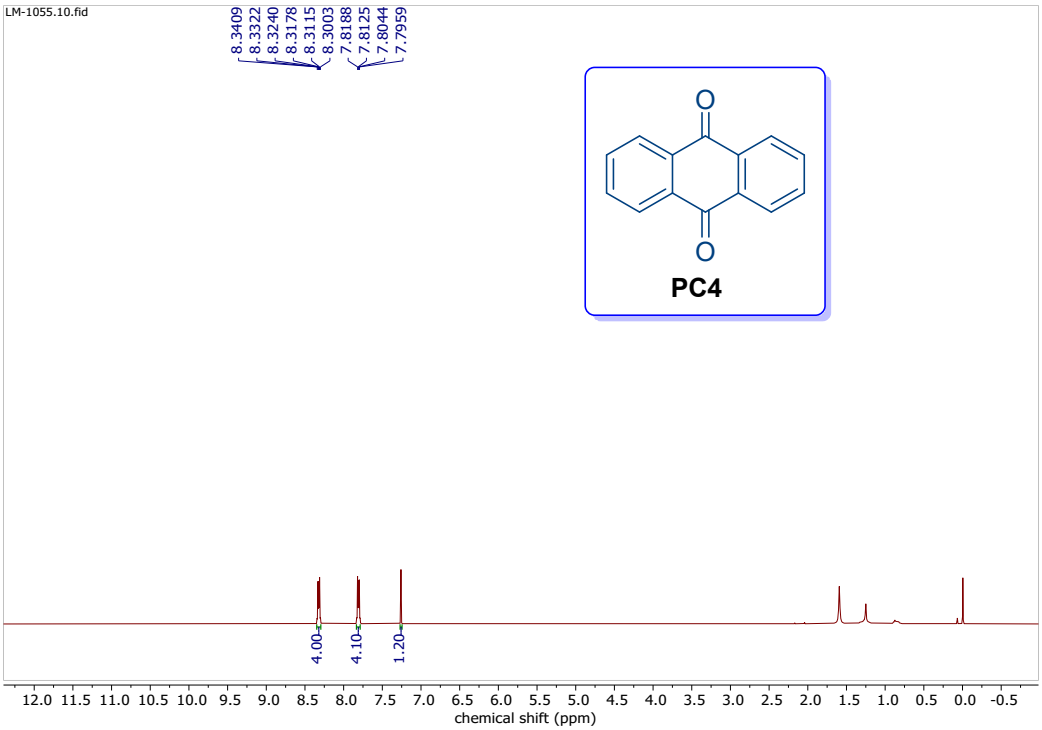


Figure S2. ^{1}H NMR spectrum of anthraquinone **(PC4)**.

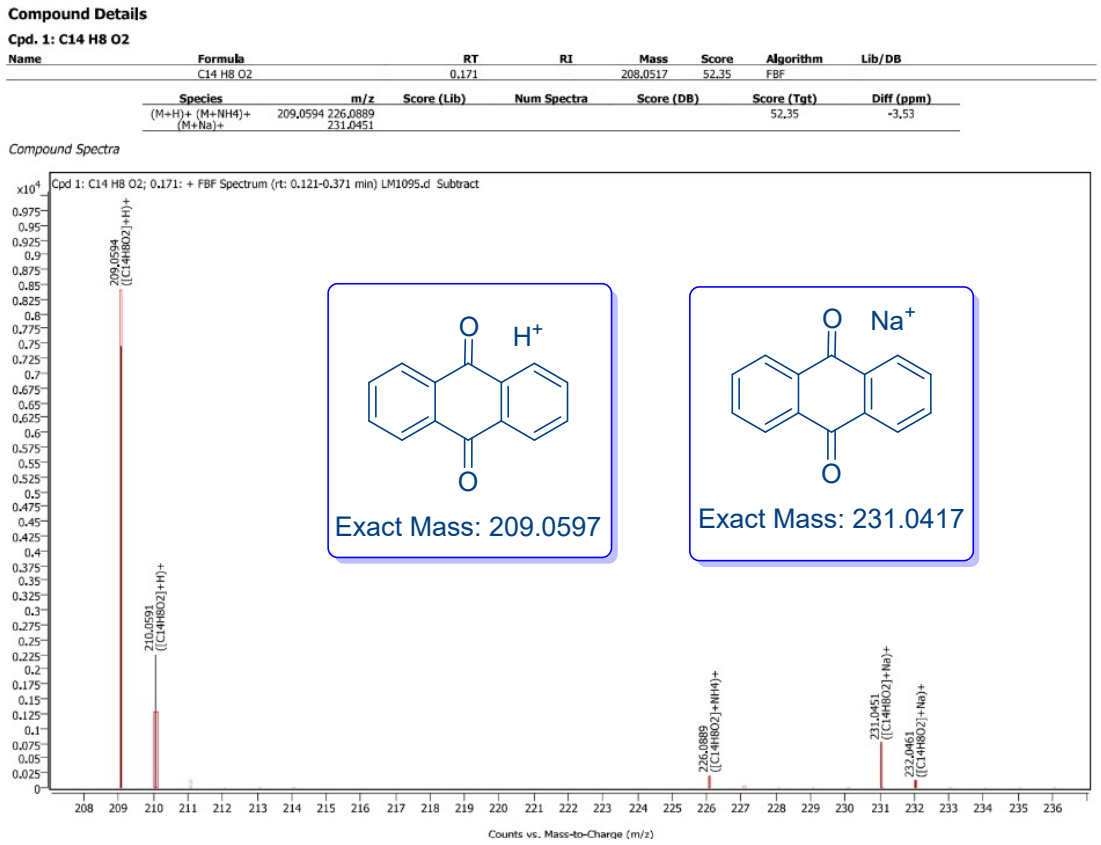
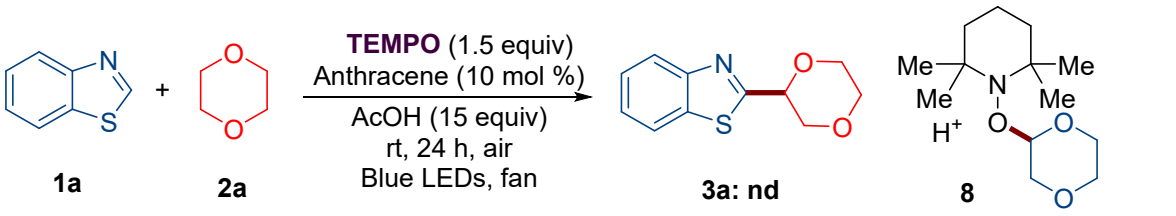


Figure S3. HRMS (ESI) spectrum of anthraquinone **(PC4)**.

5.2. Reaction in the presence of TEMPO



1a **2a** **3a: nd** **8**

Exact Mass: 244.1907
Detected Mass:244. 1923

A 15 mL Schlenk glass tube initially fitted with septa was charged with benzothiazole **1a** (0.3 mmol) and anthracene (10 mol %). Then, acetic acid (15.0 equiv), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (70 mg, 1.5 equiv), and 1,4-dioxane (1.0 mL) were added to the reaction mixture via a syringe. The resulting reaction mixture was irradiated using PR160-427 nm Kessil LEDs with stirring at room temperature for 24 h under an air cooling (fan). After completion of the reaction time, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under

reduced pressure. The crude reaction mixture was analyzed by HRMS to detect the radical TEMPO adduct.

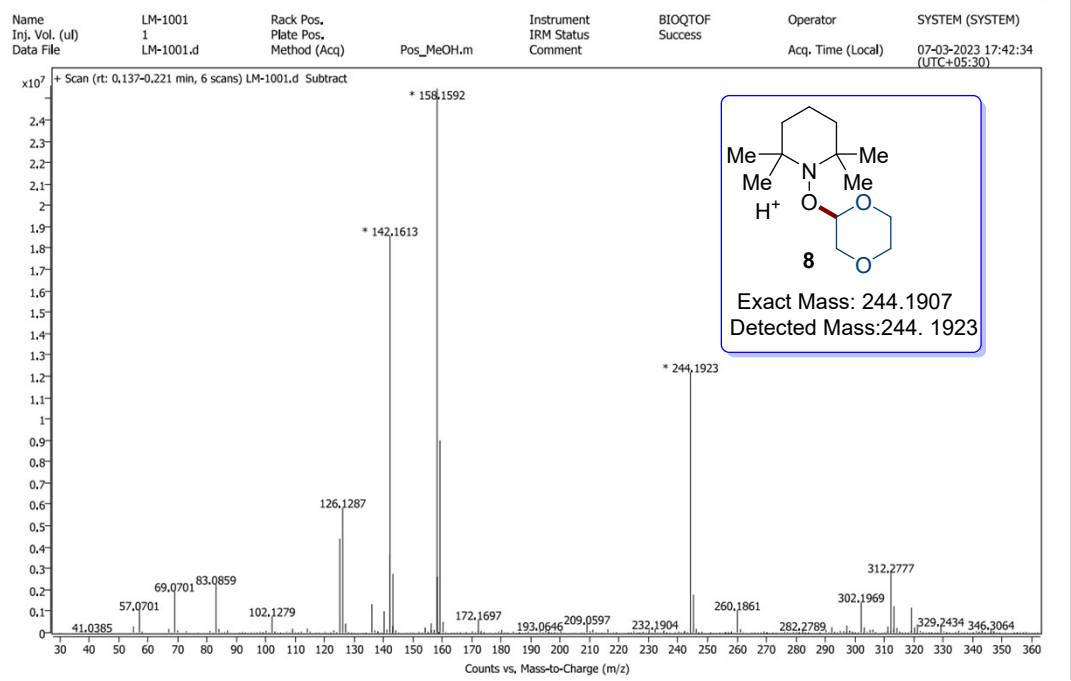
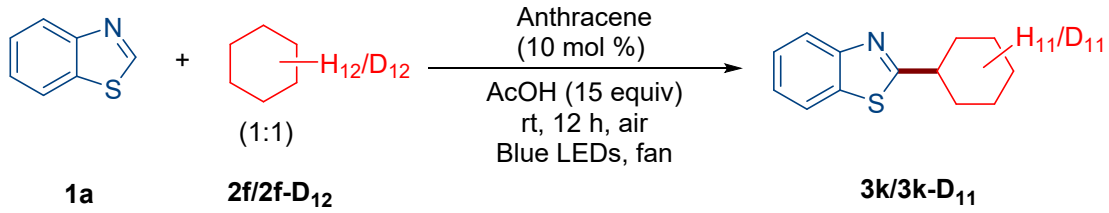


Figure S4. HRMS (ESI) spectra of TEMPO-dioxane adduct.

5.3. Kinetic isotopic experiments (KIE)



A 15 mL Schlenk glass tube initially fitted with septa was charged with benzothiazole **1a** (0.3 mmol) and anthracene (10 mol %). Then, acetic acid (15.0 equiv), **2f/2f-D$_{12}$** (1:1, 1.0 mL) were added to the reaction mixture via a syringe. The resulting reaction mixture was irradiated 427 nm Kessil LEDs with stirring at room temperature for 12 h under air cooling (fan). After completion of the reaction time, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The product **3k** + **3k-D$_{11}$** was isolated 77% (77 mg).

Kinetic isotopic effect (KIE) = k_H/k_D = 4.0

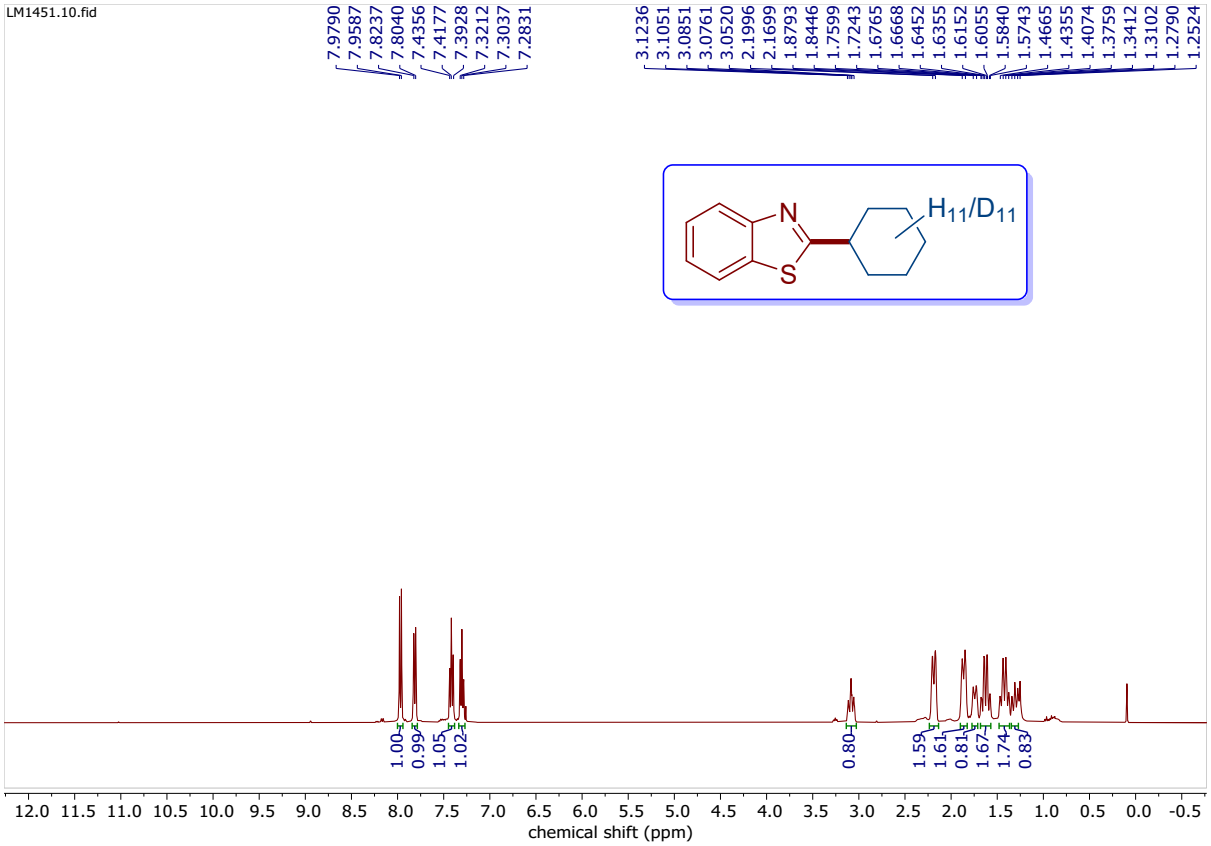
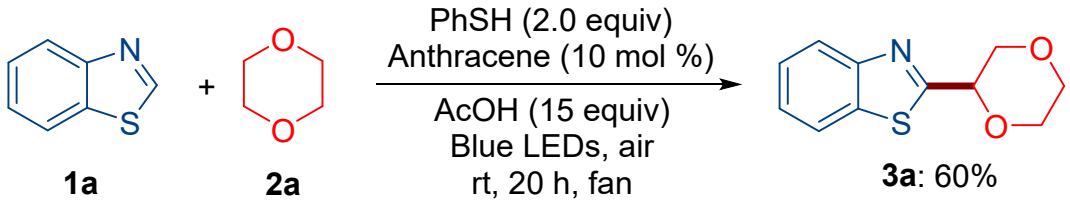


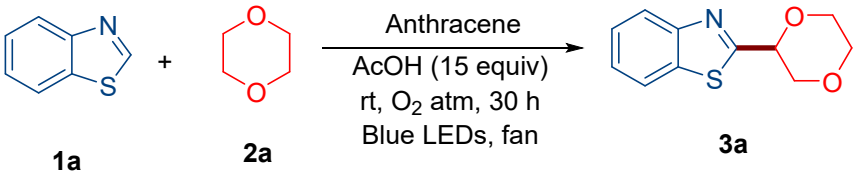
Figure S5. ^{1}H NMR of one pot KIE experiment.

5.4. Reaction in the presence of PhSH



A 15 mL Schlenk glass tube initially fitted with septa was charged with benzothiazole **1a** (0.3 mmol) and anthracene (10 mol %). Then, acetic acid (15.0 equiv), PhSH (66.10 mg, 2.0 equiv), and 1,4-dioxane (1.0 mL) were added to the reaction mixture via a syringe. The resulting reaction mixture was irradiated using PR160L-427 nm Kessil LEDs with stirring at room temperature for 30 h under an air cooling (fan). After completion of the reaction time, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc (9:1) as the eluent to give alkylated heteroarenes **3a** 60%.

5.5. Scalable rection



1a **2a** **3a**

To a 100 mL round bottom flask initially fitted with a magnetic stir bar and septa were added benzothiazole **1a** (0.5 g, 4.0 mmol) and anthracene (10 mol %), followed by acetic acid (15.0 equiv). Then 1.4-dioxane **2a** (8.0 mL) was added. The resulting reaction mixture was purged with O_2. Then, the reaction mixture was irradiated using PR160L-427 nm Kessil LEDs with stirring at room temperature for 30 hours under an oxygen atmosphere. After reaction time, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 20 mL) and combined organic layers dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc (3:1) as the eluent to give alkylated benzothiazole **3a,** 58% (0.513 g) as pure product.

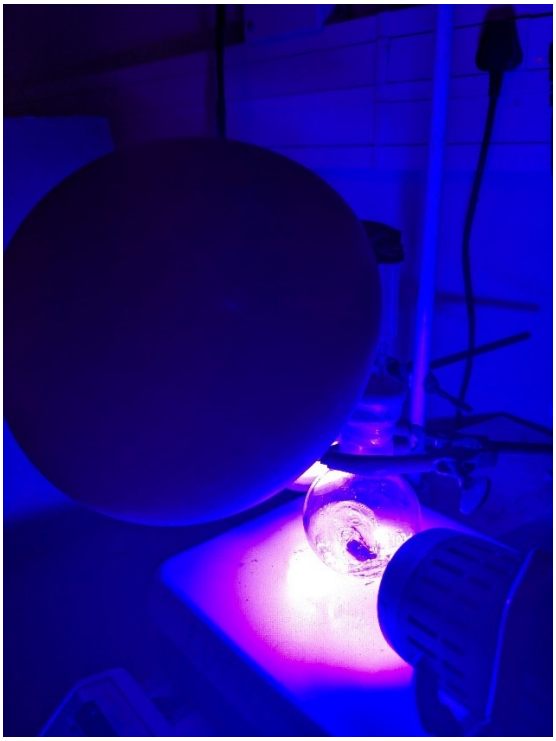
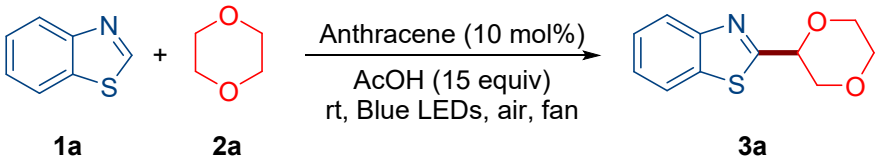


Figure S6. Photoreaction setup for large-scale synthesis. The reaction was performed using Kessil PR160-427 nm lights with 100% light intensity; the distance from the light source to the irradiation vessel is 4 cm.

5.6. Light on/off experiments



1a **2a** **3a**

Six 15 mL Schlenk glass tubes initially fitted with septa were charged with benzothiazole **1a** (0.3 mmol) and anthracene (10 mol %). Then acetic acid (15.0 equiv) and 1,4-dioxane **2a** (1.0 mL) were added to the reaction mixture. The reaction vials were irradiated under 420 nm Kessil LEDs with stirring at room temperature under air cooling (fan). After every 5 h, we stopped light irradiation and supplied the light source consecutively. After each light on and off reaction time was completed, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resulting aqueous solution was extracted with EtOAc (3 x 10 mL), and the combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography using *n*-hexane/EtOAc (3:1) as the eluent to give alkylated benzothiazole **3a** pure product.

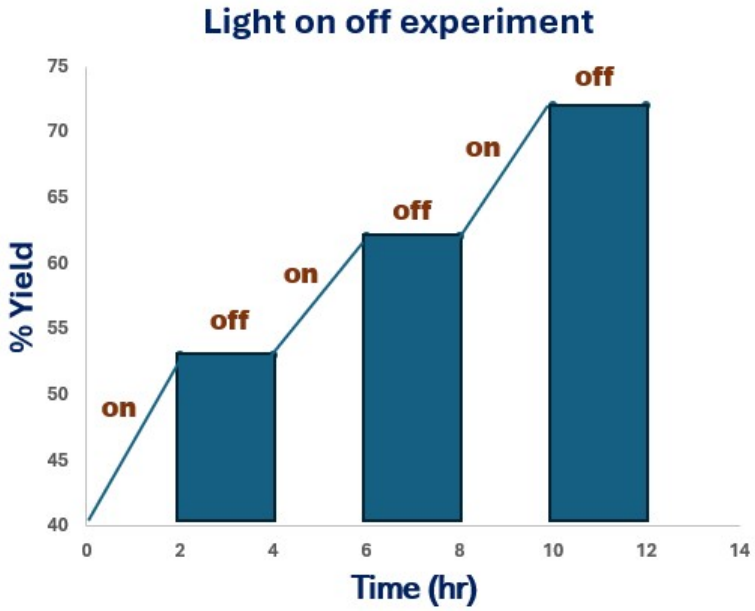


Figure S7. Light on/off study.

5.7. UV-Vis Absorption studies

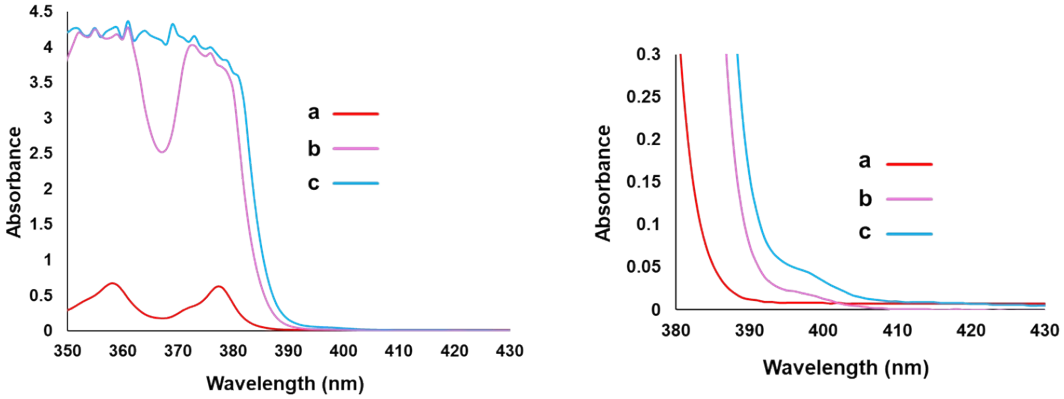


Figure S8. UV-Vis absorption study of anthracene. **a)** 35 µM anthracene in dioxane (3.0 mL). **b)** 35 µM anthracene in acetic acid (3.0 mL). **Stock solution:** anthracene (10 mg), glacial acetic acid (257 µL) in 1.0 mL dioxane with Kessil PR160-427 nm lights irradiation for 15 mins. Then, the reaction mixture was diluted with 3.0 mL 1,4-dioxane as stock solution. **c)** 100 µL above reaction stock solution in dioxane

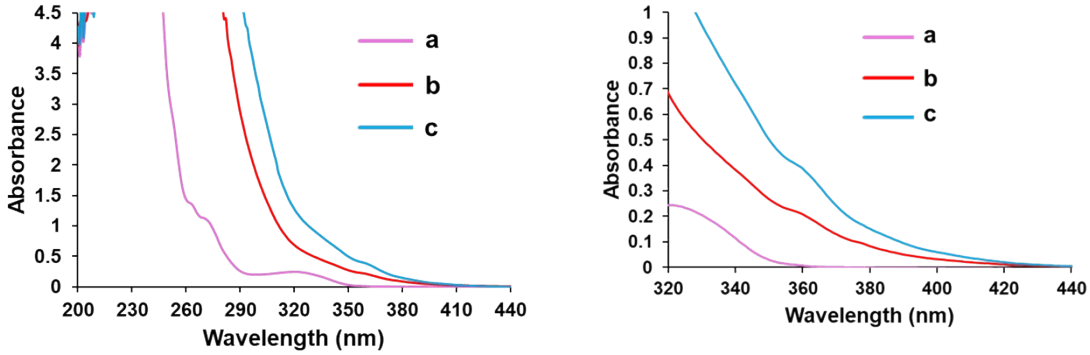


Figure S9. UV-Vis absorption study of anthraquinone. **a**) 75 µM anthraquinone in 1,4-dioxane (3.0 mL). **b**) 75 µM anthraquinone in acetic acid (3.0 mL). **Stock solution:** anthraquinone (10 mg), glacial acetic acid (257 µL) in 1.0 mL dioxane with Kessil PR160-427 nm lights irradiation for 15 mins. Then, the above reaction mixture was diluted with 3.0 mL dioxane. **c**) Stock solution 100 µL in dioxane (2.9 mL).

Conclusion: *The absorption studies using anthracene and anthraquinone photocatalysts showed that both photocatalysts* ***PC1*** *and* ***PC4*** *absorbed in the range of emission radiation of used PR-160-427 nm LED lights.*

5.8. Fluorescence quenching experiments

Preparation of the stock solution: A 0.5 mM solution of the anthracene was prepared in a standard flask by dissolving 8.91 mg of the anthracene in 100 mL of CH_3CN. The resulting solution was used for all the quenching studies. The freshly prepared solution was taken with the required amount using a micropipette from the stock solution and diluted further to 2.0 mL by adding CH_3CN in the cuvette. Similarly, 10 mL of 0.1 mM solution of benzothiazole **1a**, 1,4-dioxane **2a,** and acetic acid were prepared by dissolving the requisite amount of each substrate in CH_3CN.

Quenching studies: Fluorescence emission spectra of the photocatalyst in the presence of different reagents (**1a, 2a,** and acetic acid) were studied and analyzed in detail to figure the light emission properties of the pure catalyst anthracene and other reaction components. Emission intensities of anthracene were recorded with PerkinElmer FL6500 spectrometer using a 10.0 mm quartz cuvette. The catalyst exhibits an absorption maximum between 275-325 nm, confirmed by the literature.[2] The sample solution of anthracene with a proper concentration of 0.5 mM was excited with the wavelength of 365 nm; the emission maxima were observed at 397 nm. The reaction mixture has a quenching effect on the photocatalyst, and the emission intensity of the PC decreases gradually upon increasing the concentration of the reaction mixture (15, 30, 45, 60, and 75 µL) (0.1 mM).

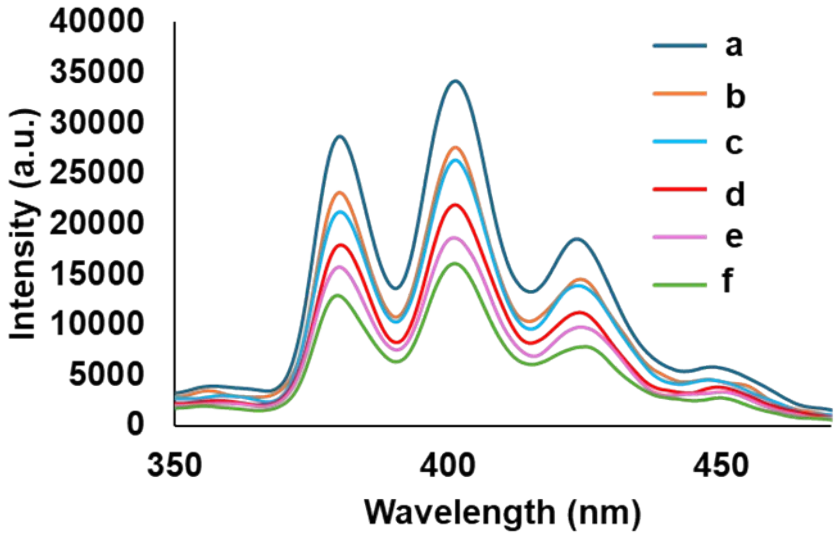


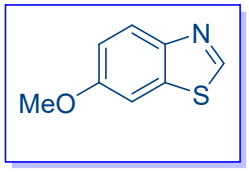
Figure S10. Fluorescence quenching studies of anthracene photocatalyst. **a**) 0.5 mM anthracene in MeCN. **b)** Addition of 15 µL of benzothiazole (0.1 mM), acetic acid (0.1 mM),

1,4-dioxane (0.1 mM) to 0.5 mM Anthracene in MeCN. **c)** Addition of 30 µL of benzothiazole (0.1 mM), acetic acid (0.1 mM), 1,4-dioxane (0.1 mM) to 0.5 mM Anthracene in MeCN. **d)** Addition of 45 µL of benzothiazole (0.1 mM), acetic acid (0.1 mM), 1,4-dioxane (0.1 mM) to 0.5 mM Anthracene in MeCN. **e**) Addition of 60 µL of benzothiazole (0.1 mM), acetic acid (0.1 mM), 1,4-dioxane (0.1 mM) to 0.5 mM Anthracene in MeCN. **f)** Addition of 75 µL of benzothiazole (0.1 mM), acetic acid (0.1 mM), 1,4-dioxane (0.1 mM) to 0.5 mM Anthracene in MeCN.

***Conclusion*:** *The fluorescence quenching studies of anthracene photocatalysts suggested that the reaction mixture quenches the emission of excited photocatalysts.*

6. Spectral data of starting materials

6-Methoxybenzo[*d*]thiazole (1b)



Compound **1b** was prepared according to the **GP-1** using 6-methoxybenzo[*d*]thiazol-2-amine (8.0 mmol).

Appearance: Yellow solid

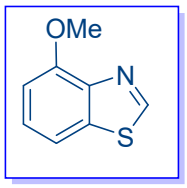
Yield: 82%

M.p.: 121 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.91 (s, 1H), 7.53 (d, *J* = 6.3 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 7.9 Hz, 1H), 4.07 (s, 3H).

IR (ATR): 3063, 1588, 1429, 1280, 1095, 879 cm^{-1}.

4-Methoxybenzo[*d*]thiazole (1c)



Compound **1c** was prepared according to the **GP-1** using 4-methoxybenzo[*d*]thiazole (8.0 mmol).

Appearance: Yellow solid

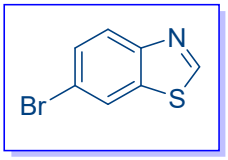
Yield: 76%

M.p.: 122 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.91 (s, 1H), 7.53 (d, *J* = 6.3 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 7.9 Hz, 1H), 4.07 (s, 3H).

IR (ATR): 3042, 1699, 1544, 1463, 1306, 1130, 1005 cm^{-1}.

6-Bromobenzo[*d*]thiazole (1d)



Compound **1d** was prepared according to the **GP-1** using 6-bromobenzo[*d*]thiazol-2-amine (16 mmol).

Appearance: Yellow solid.

Yield: 74%

M.p.: 188 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.96 (s, 1H), 8.09 (d, *J* = 1.9 Hz, 1H), 7.98 (d, *J* = 8.8 Hz, 1H), 7.61 (dd, *J* = 8.7, 1.9 Hz, 1H).

IR (ATR): 2919, 1393, 1280, 1130, 1005, 622, 425 cm^{-1}.

6-Chlorobenzo[*d*]thiazole (1e)

Compound **1e** was prepared according to the **GP-1** using 6-chlorobenzo[*d*]thiazol-2-amine (16 mmol).

Appearance: Yellow solid.

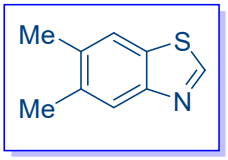
Yield: 76% (2.05 g)

M.p.: 155 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.97 (s, 1H), 8.03 (d, *J* = 8.8 Hz, 1H), 7.92 (d, *J* = 2.1 Hz, 1H), 7.47 (dd, *J* = 8.8, 2.1 Hz, 1H).

IR (ATR): 1656, 1464, 1302, 1095, 874, 757 cm^{-1} .

5,6-dimethylbenzo[*d*]thiazole (1f)



Compound **1f** was prepared according to the **GP-1** using 5,6-dimethylbenzo[*d*]thiazol-2-amine (16 mmol).

Appearance: Yellow solid

Yield: 84%

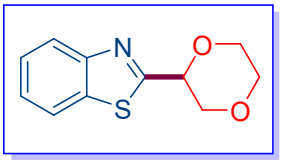
M.p.: 165 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.87 (s, 1H), 7.90 (s, 1H), 7.70 (s, 1H), 2.41 (s, 3H), 2.40 (s, 3H).

IR (ATR): 3063, 2919, 2728, 1538, 1430, 1279 cm^{-1}.

7. Spectral data of products

2-(1,4-Dioxan-2-yl) benzo[*d*]thiazole (3a)



Compound **3a** was prepared according to the **GP-3** using benzothiazole (**1a**) (40 mg, 0.3 mmol) and 1,4-dioxane (**2a**) (1.0 mL).

Appearance: White solid

Yield: 94% (63 mg)

M.p.: 135 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, *J* = 6.1 Hz, 1H), 7.90 (d, *J* = 6.9 Hz, 1H), 7.47 (t, *J* = 8.4, 7.3, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 5.05 (dd, *J* = 9.7, 3.1 Hz, 1H), 4.30 (dd, *J* = 11.6, 3.1 Hz, 1H), 4.04 – 3.93 (m, 2H), 3.86 – 3.66 (m, 3H).

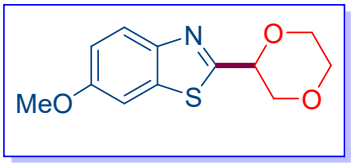
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 169.0 (C_q), 153.0 (C_q), 134.6 (C_q), 126.1 (*C*H), 125.2 (*C*H), 123.1 (*C*H), 121.8 (*C*H), 75.4 (*C*H), 70.5 (CH_2), 67.0 (CH_2), 66.4 (CH_2).

HRMS (ESI): m/z cald for $C_{11}H_{13}NO_2S$ $[M + H]^+$ 222.0583, found 222.0585.

IR(ATR): 2960, 2921, 2860, 1523, 1454, 1110, 909, 758, 731, 432 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

2-(1,4-Dioxan-2-yl)-6-methoxybenzo[*d*]thiazole (3b)



Compound **3b** was prepared according to the **GP-3** using 6-methoxybenzo[*d*]thiazole **(1b)** (66 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 76% (76 mg)

M.p.: 168 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.9 Hz, 1H), 7.34 (d, *J* = 2.5 Hz, 1H), 7.07 (dd, *J* = 8.9, 2.6 Hz, 1H), 5.00 (dd, *J* = 9.7, 3.1 Hz, 1H), 4.26 (dd, *J* = 11.6, 3.1 Hz, 1H), 4.03 – 3.92 (m, 2H), 3.87 (s, 3H), 3.85 – 3.74 (m, 2H), 3.70 (dd, *J* = 11.6, 9.8 Hz, 1H).

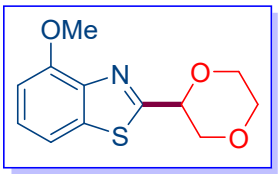
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 166.2 (C_q), 157.7 (C_q), 147.5 (C_q), 135.9 (C_q), 123.6 (*C*H), 115.6 (*C*H), 104.1 (*C*H), 75.4 (*C*H), 70.5 (CH_2), 66.9 (CH_2), 66.4 (CH_2), 55.8 (CH_3).

HRMS (ESI): m/z cald for $C_{12}H_{13}NO_3S$ $[M + Na]^+$ 274.0508, found 274.0520.

IR(ATR): 2921, 1735, 1576, 1486, 1442, 1362, 1098, 705 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[3]

2-(1,4-Dioxan-2-yl)-4-methoxybenzo[*d*]thiazole (3c)



Compound **3c** was prepared according to the **GP-3** using 4-methoxybenzo[d]thiazole **(1c)** (66 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 67% (67 mg)

M.p.: 166 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (d, *J* = 8.0 Hz, 1H), 7.34 (t, *J* = 8.1 Hz, 1H), 6.91 (d, *J* = 9.0 Hz, 1H), 5.10 (dd, *J* = 9.9, 3.1 Hz, 1H), 4.35 (dd, *J* = 11.6, 3.1 Hz, 1H), 4.04 (s, 3H), 4.01 – 3.93 (m, 2H), 3.86 – 3.71 (m, 2H), 3.60-3.66 (m, 1H).

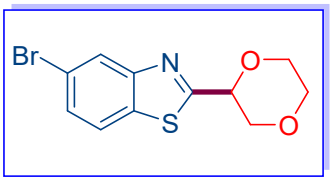
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 167.8 (C_q), 153.4 (C_q), 143.1 (C_q), 136.3 (C_q), 126.1 (*C*H), 113.7 (*C*H), 106.4 (*C*H), 75.7 (*C*H), 70.8 (CH_2), 67.0 (CH_2), 66.3 (CH_2), 55.9 (CH_3).

HRMS (ESI): m/z cald for $C_{12}H_{13}NO_3S$ $[M + H]^+$ 252.0689, found 252.0694.

IR(ATR):2923, 2853, 1734, 1601, 1465, 1245, 1116, 1054, 906, 828 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[3]

5-Bromo-2-(1,4-dioxan-2-yl) benzo[*d*]thiazole (3d)



Compound **3d** was prepared according to the **GP-3** using 5-bromobenzo[*d*]thiazole **(1d)** (97.66 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 65% (78 mg)

M.p.: 201 °C

^1H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 2.0 Hz, 1H), 7.77 (d, *J* = 8.6 Hz, 1H), 7.51 (dd, *J* = 8.7, 1.9 Hz, 1H), 4.95 (dd, *J* = 9.7, 3.1 Hz, 1H), 4.22 (dd, *J* = 11.6, 3.1 Hz, 1H), 3.98 – 3.86 (m, 2H), 3.80 – 3.65 (m, 2H), 3.61 (dd, *J* = 11.6, 9.6 Hz, 1H).

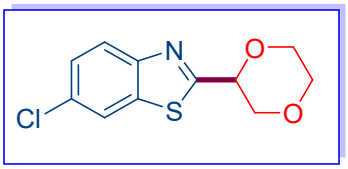
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 169.7 (C_q), 151.9 (C_q), 136.3 (C_q), 129.7 (*C*H), 124.4 (*C*H), 124.2 (*C*H), 118.8 (C_q), 75.2 (*C*H), 70.3 (CH_2), 67.0 (CH_2), 66.4 (CH_2).

HRMS (ESI): m/z cald for $C_{11}H_{10}BrNO_2S$ $[M + H]^+$ 299.9688, found 299.9683.

IR(ATR): 3074, 3053, 2962, 2919, 1587, 1523, 1441, 1419, 1112, 688 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[3]

6-Chloro-2-(1,4-dioxan-2-yl) benzo[*d*]thiazole (3e)



Compound **3e** was prepared according to the **GP-3** using 6-chlorobenzo[*d*]thiazole **(1e)** (92 mg, 0.3 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 56% (57 mg)

M.P: 171-173 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 – 7.80 (m, 2H), 7.37 (dd, *J* = 8.6, 2.3 Hz, 1H), 4.96 (dd, *J* = 9.6, 3.1 Hz, 1H), 4.22 (dd, *J* = 11.6, 3.1 Hz, 1H), 3.98 – 3.86 (m, 2H), 3.80 – 3.58 (m, 3H).

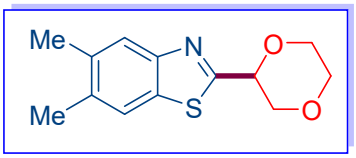
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 169.7 (C_q), 151.6 (C_q), 135.8 (C_q), 131.2 (C_q), 127.0 (*C*H), 123.9 (*C*H), 121.4 (*C*H), 75.2 (*C*H), 70.3 (CH_2), 67.0 (CH_2), 66.4 (CH_2).

HRMS (ESI): m/z cald for $C_{11}H_{10}ClNO_2S$ $[M + H]^+$ 256.0194, found 256.0196.

IR(ATR): 3079, 3056, 2966, 2921, 2855, 1591, 1524, 1112, 637, 654 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

2-(1,4-Dioxan-2-yl)-5,6-dimethylbenzo[*d*]thiazole (3f)



Compound **3f** was prepared according to the **GP-3** using 5,6-dimethylbenzo[*d*]thiazole **(1f)** (65.29 mg, 0.3 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 78% (77 mg)

M.p.: 114 °C

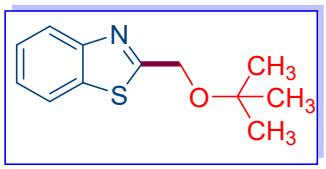
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (s, 1H), 7.65 (s, 1H), 5.03 (dd, *J* = 9.8, 3.1 Hz, 1H), 4.27 (dd, *J* = 11.6, 3.1 Hz, 1H), 4.05 – 3.93 (m, 2H), 3.86 – 3.64 (m, 3H), 2.39 (s, 3H), 2.38 (s, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 167.7 (C_q), 152.8 (C_q), 135.4 (C_q), 135.0 (C_q), 134.7 (C_q), 123.24 (*C*H), 121.66 (*C*H), 75.5 (*C*H) 70.58 (CH_2), 67.02 (CH_2), 66.41 (CH_2), 20.23 (2 CH_3).

HRMS (ESI): m/z cald for $C_{13}H_{15}NO_2S$ $[M + H]^+$ 250.0896, found 250.0903.

IR(ATR): 2921, 2852, 1741, 1525, 1455, 1332, 1126, 1048, 905 cm^{-1}.

2-(Tert-butoxymethyl) benzo[*d*]thiazole (3g)



Compound **3g** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and 2-methoxy-2-methylpropane **(2b)** (1.0 mL).

Appearance: Colourless liquid

Yield: 88% (78 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 7.6 Hz, 1H), 7.87 (d, *J* = 9.1 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 4.85 (s, 2H), 1.32 (s, 9H).

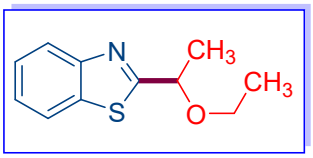
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 173.0 (C_q), 153.3 (C_q), 134.9 (C_q), 125.8 (*C*H), 124.7 (*C*H), 122.7 (*C*H), 121.7 (*C*H), 75.1 (*C*H), 62.6 (CH_2), 27.5 (3 CH_3).

HRMS (ESI): m/z cald for $C_{12}H_{15}NOS$ $[M + H]^+$ 222.0947, found 222.0951.

IR(ATR): 3379, 2929, 2831, 1651, 1602, 1522, 1470, 1088, 1025, 756, 459 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[4]

2-(1-Ethoxyethyl)benzo[*d*]thiazole (3h)



Compound **3h** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and diethyl ether **(2c)** (1.5 mL).

Appearance: Colourless liquid

Yield: 75% (62 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J* = 6.5 Hz, 1H), 7.85 (d, *J* = 7.9 Hz, 1H), 7.46 – 7.40 (m, 1H), 7.33 (t, *J* = 6.9 Hz, 1H), 4.83 (q, *J* = 6.6 Hz, 1H), 3.63 – 3.55 (m, 2H), 1.62 (d, *J* = 6.5 Hz, 3H), 1.24 (t, *J* = 7.0 Hz, 3H).

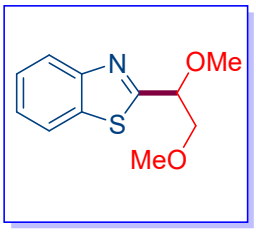
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 176.8 (C_q), 153.1 (C_q), 134.9 (C_q), 125.9 (*C*H), 125.0 (*C*H), 122.9 (*C*H), 121.9 (*C*H), 76.1 (*C*H), 65.5 (CH_2), 22.7 (CH_3), 15.3 (CH_3).

HRMS (ESI): m/z cald for $C_{11}H_{13}NOS$ $[M + H]^+$ 208.0791, found 208.0795.

IR(ATR): 2925, 2853, 1736, 1519, 1438,1313, 1190, 1106 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[4]

2-(1,2-Dimethoxyethyl) benzo[*d*]thiazole (3i)



Compound **3i** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and 1,2-dimethoxyethane **(2d)** (1.0 mL).

Appearance: Colourless liquid.

Yield: 41% (36 mg)

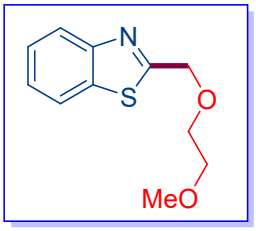
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 8.1 Hz, 1H), 7.89 (d, *J* = 7.1 Hz, 1H), 7.49 – 7.44 (m, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 4.83 (dd, *J* = 6.4, 3.8 Hz, 1H), 3.85 – 3.75 (m, 2H), 3.52 (s, 3H), 3.41 (s, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 171.6 (C_q), 152.9 (C_q), 135.0 (C_q), 126.1 (*C*H), 125.3 (*C*H), 123.1 (*C*H), 121.9 (*C*H), 81.1 (*C*H), 75.2 (CH_2), 59.5 (CH_3), 58.6 (CH_3).

HRMS (ESI): m/z cald for $C_{11}H_{13}NO_2S$ $[M + H]^+$ 224.0740, found 224.0738.

IR(ATR): 3372, 2851, 1625, 1147, 1055, 776 cm^{-1}.

(2-Methoxyethoxy) methyl) benzo[*d*]thiazole (3i′)



Compound **3i'** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and 1,2-dimethoxyethane **(2d)** (1.0 mL).

Appearance: Colourless liquid.

Yield: 30% (26 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.3 Hz, 1H), 7.83 (d, *J* = 7.5 Hz, 1H), 7.41 (m, 1H), 7.32 (m, 1H), 4.93 (s, 2H), 3.76 – 3.71 (m, 2H), 3.58 – 3.55 (m, 2H), 3.35 (s, 3H).

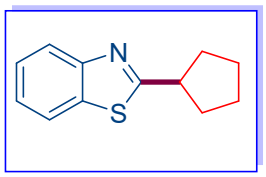
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 170.5 (C_q), 152.8 (C_q), 135.0 (C_q), 126.1 (*C*H), 125.2 (*C*H)), 122.9 (*C*H), 121.8 (*C*H), 71.8 (CH_2), 70.7 (CH_2), 70.7 (CH_2), 59.2 (CH_3).

HRMS (ESI): m/z cald for $C_{11}H_{13}NO_2S$ $[M + H]^+$ 224.0740, found 224.0735.

IR(ATR): 3336, 1621, 1144, 562, 441, 326 Cm^{-1}.

The analytical data are in accordance with those reported in the literature.[5]

2-Cyclopentylbenzo[*d*]thiazole (3j)



Compound **3j** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and cyclopentane **(2e)** (1.5 mL).

Appearance: Oily liquid

Yield: 38% (31 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, *J* = 8.1 Hz, 1H), 7.76 (d, *J* = 9.3 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 1H), 7.26 (t, *J* = 8.2 Hz, 1H), 3.48 (p, *J* = 8.1 Hz, 1H), 2.24 – 2.14 (m, 2H), 1.93 – 1.77 (m, 4H), 1.73 – 1.63 (m, 2H).

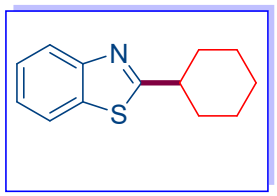
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 177.2 (C_q), 153.2 (C_q), 134.8 (C_q), 125.8 (*C*H), 124.5 (*C*H), 122.5 (*C*H), 121.5 (*C*H), 44.8 (*C*H), 34.1 (2 *C*H_2), 25.6 (2 *C*H_2).

HRMS (ESI): m/z cald for $C_{12}H_{13}NS$ $[M + H]^+$ 204.0841, found 204.0842.

IR(ATR): 3337, 1620, 1149, 532, 441, 326 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[5]

2-Cyclohexylbenzo[*d*]thiazole (3k)



Compound **3k** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and cyclohexane **(2f)** (1.0 mL).

Appearance: Oily liquid

Yield: 91% (79 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 7.9 Hz, 1H), 7.29 (t, *J* = 8.4 Hz, 1H), 7.18 (t, *J* = 8.3 Hz, 1H), 2.96 (tt, *J* = 11.6, 3.6 Hz, 1H), 2.10 – 2.02 (m, 2H), 1.74 (dt, *J* = 13.0, 3.4 Hz, 2H), 1.61 (dd, *J* = 15.2, 4.2 Hz, 1H), 1.50-1.54 (m, 2H), 1.36 – 1.10 (m, 3H).

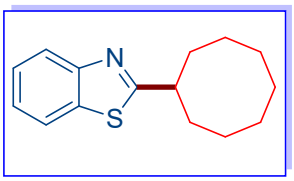
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 177.7 (C_q), 153.1 (C_q), 134.5 (C_q), 125.8 (*C*H), 124.5 (*C*H), 122.6 (*C*H), 121.6 (*C*H), 43.5 (*C*H), 33.4 (2 *C*H_2), 26.1 (2 *C*H_2), 25.8 (*C*H_2).

HRMS (ESI): m/z cald for $C_{13}H_{15}NS$ $[M + H]^+$ 218.0998, found 218.1000.

IR(ATR): 2926, 2852, 1449, 758, 729 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

2-Cyclooctylbenzo[*d*]thiazole (3l)



Compound **3i** was prepared according to the **GP-3** using benzothiazole **(2a)** (54 mg, 0.4 mmol) and cyclooctane **(2g)** (1.0 mL).

Appearance: Colourless liquid

Yield: 77% (76 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 8.1 Hz, 1H), 7.66 (d, *J* = 9.5 Hz, 1H), 7.26 (t, *J* = 8.3, 7.1 Hz, 1H), 7.15 (t, *J* = 7.7, 1.4 Hz, 1H), 3.25 – 3.17 (m, 1H), 2.04 – 1.93 (m, 2H), 1.86 – 1.74 (m, 2H), 1.68 (dt, *J* = 14.8, 11.4 Hz, 3H), 1.50 – 1.44 (m, 7H).

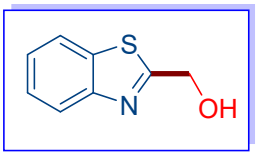
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 179.1 (C_q), 152.9 (C_q), 134.7 (C_q), 125.8 (*C*H), 124.5 (*C*H), 122.5 (*C*H), 121.5 (*C*H), 43.7 (*C*H), 32.9 (2 *C*H_2), 26.9 (2 *C*H_2), 26.1 (*C*H_2), 25.4 (2 *C*H_2).

HRMS (ESI): m/z cald for $C_{15}H_{19}NS$ $[M + H]^+$ 246.1311, found 246.1314.

IR(ATR): 3385, 2920, 2852, 1690, 1509, 1437, 1314, 1241, 757, 729 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[6]

Benzo[*d*]thiazol-2-ylmethanol (3m)



Compound **3m** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and methanol **(2h)** (1.0 mL).

Appearance: Colourless liquid.

Yield: 21% (14 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 7.5 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.48 (t, *J* = 7.0 Hz, 1H), 7.39 (t, *J* = 8.3 Hz, 1H), 5.08 (s, 2H).

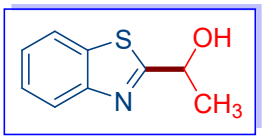
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 172.4 (C_q), 152.8 (C_q), 134.7 (C_q), 126.2 (*C*H), 125.1 (*C*H), 122.8 (*C*H), 121.9 (*C*H), 62.6 (CH_2).

HRMS (ESI): m/z cald for C_8H_7NOS $[M + H]^+$ 166.0321, found 166.0323.

IR(ATR): 3234, 2923, 2860, 1522, 1401, 1240, 1013, 1044, 825, 611.96 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[6]

1-(Benzo[*d*]thiazol-2-yl)ethan-1-ol (3n)



Compound **3n** was prepared according to the **GP-3** using benzothiazole **(2a)** (54 mg, 0.4 mmol) and ethanol **(2i)** (1.0 mL).

Appearance: Oily liquid

Yield: 46% (33 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 8.1 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 7.1 Hz, 1H), 7.37 (t, *J* = 8.2 Hz, 1H), 5.25 (q, *J* = 6.6 Hz, 1H), 3.70 (s, 1H), 1.70 (d, *J* = 6.6 Hz, 3H).

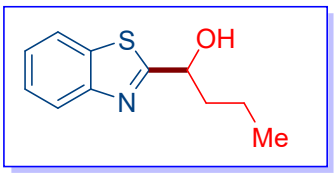
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 176.8 (C_q), 152.9 (C_q), 134.9 (C_q), 126.1 (*C*H), 125.0 (*C*H), 122.9 (*C*H), 121.9 (*C*H), 68.6 (*C*H), 29.7 (CH_3).

HRMS (ESI): m/z cald for Chemical Formula: C_9H_9NOS $[M + H]^+$ 180.0478, found 180.0482.

IR(ATR): 3233, 2923, 2877, 1522, 1402, 1066, 624 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[6]

1-(Benzo[*d*]thiazol-2-yl)butan-1-ol (3o)



Compound **3o** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and nbutanol **(2j)** (1.0 mL).

Appearance: Colourless liquid

Yield: 42% (35 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 8.1 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 7.1 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 5.12 – 5.07 (m, 1H), 3.57 (s, 1H), 2.05 – 1.82 (m, 2H), 1.62 – 1.46 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).

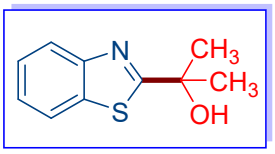
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 176.6 (C_q), 152.8 (C_q), 134.8 (C_q), 126.1 (*C*H), 125.0 (*C*H), 122.8 (*C*H), 121.8 (*C*H), 72.1 (*C*H), 40.2 (CH_2), 18.5 (CH_2), 13.8 (CH_3).

HRMS (ESI): m/z cald for $C_{11}H_{13}NOS$ $[M + NH_4]^+$ 225.1056, found 225.1069.

IR(ATR): 3199, 2964, 2937, 2872, 2815, 1512, 1292, 1156, 551 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[6]

2-(Benzo[*d*]thiazol-2-yl)propan-2-ol (3p)



Compound **3p** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and isopropanol **(2k)** (1.0 mL).

Appearance: Oily liquid.

Yield: 67% (52 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 9.5 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.46 (m, 1H), 7.39 – 7.34 (m, 1H), 3.37 (s, 1H), 1.75 (s, 6H).

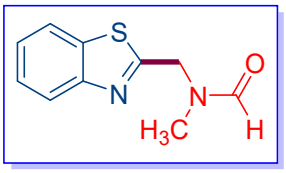
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 179.9 (C_q), 153.1 (C_q), 135.4 (C_q), 126.0 (*C*H), 124.9 (*C*H), 122.9 (*C*H), 121.8 (*C*H), 73.6 (C_q), 30.8 (2 *C*H_3).

HRMS (ESI): m/z cald for $C_{10}H_{11}NOS$ $[M + H]^+$ 194.0634, found 194.0634.

IR(ATR): 3303, 3117, 2975, 1788, 1646, 1510, 1585, 1435, 1174, 1243, 1044, 937 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[6]

***N*-(Benzo[*d*]thiazol-2-ylmethyl)-N-methylformamide (3q)**



Compound **3q** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and *N, N*-dimethylformamide **(2l)** (1.0 mL).

Appearance: Pale yellow liquid

Yield: 53% (44 mg) Both the *Z* and *E* isomers were obtained.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (s, 1H), 8.11 (s, 1H), 7.97 – 7.91 (m, 2H), 7.84 – 7.77 (m, 2H), 7.47 – 7.38 (m, 2H), 7.38 – 7.30 (m, 2H), 4.86 (s, 2H), 4.75 (s, 1H), 3.00 (s, 3H), 2.90 (s, 2H).

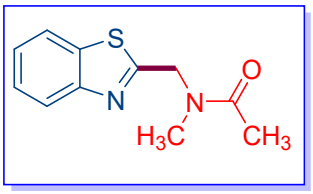
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 167.2 (C_q), 166.3 (C_q), 162.8 (C_q), 162.6 (C_q), 153.1 (C_q), 152.7 (C_q), 135.6 (C_q), 135.1 (C_q), 126.5 (*C*H), 126.2 (*C*H), 125.7 (*C*H), 125.4 (*C*H), 123.3 (*C*H), 123.1 (*C*H), 121.9 (*C*H), 121.8 (*C*H), 51.7 (CH_2), 46.2 (CH_2), 34.7 (CH_3), 30.40 (CH_3).

HRMS (ESI): m/z cald for $C_{10}H_{10}N_2OS$ $[M + H]^+$ 207.0587, found 207.0567.

IR(ATR): 3473, 2781, 1664, 1457, 1058, 639, 419 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[5]

N-(Benzo[d]thiazol-2-ylmethyl)-N-methylacetamide (3r)



Compound **3r** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and *N, N*-dimethylacetamide **(2m)** (1.0 mL).

Appearance: Colourless liquid

Yield: 48% (43 mg). Both the Z and E isomers were obtained.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 7.4 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 8.8 Hz, 1H), 7.40 (d, *J* = 8.8 Hz, 1H), 4.93 (d, *J* = 33.5 Hz, 2H), 3.12 (d, *J* = 13.0 Hz, 3H), 2.22 (d, *J* = 18.8 Hz, 3H).

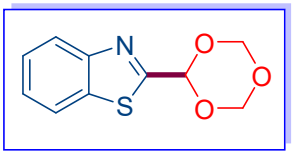
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 171.0 (C_q), 170.9 (C_q), 168.1 (C_q), 167.7 (C_q), 153.3 (C_q), 152.7 (C_q), 135.7 (C_q), 134.8 (C_q), 126.5 (*C*H), 126.1 (*C*H), 125.5 (*C*H), 125.3 (*C*H), 123.2 (*C*H), 122.9 (*C*H), 121.9 (*C*H), 121.8 (*C*H), 53.1 (CH_2), 49.4 (CH_2), 36.4 (CH_3), 34.5 (CH_3), 21.6 (CH_3), 21.5 (CH_3).

HRMS (ESI): m/z cald for $C_{11}H_{12}N_2OS$ $[M + Na]^+$ 243.0563, found 243.0551.

IR(ATR): 2927, 1801, 1650, 1288, 1172, 1038, 941 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[5]

2-(1,3,5-Trioxan-2-yl) benzo[*d*]thiazole (3s)



Compound **3s** was prepared according to the **GP-3** using benzothiazole **(1a)** (54 mg, 0.4 mmol) and 1,3,5-trioxane **(2n)** (10.0 equiv). and $CDCl_3$ (0.5 mL)

Appearance: White solid

Yield: 45 % (40 mg)

M.p.: 146 °C

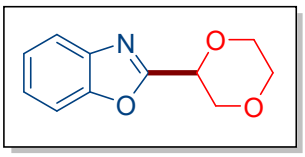
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, *J* = 8.3 Hz, 1H), 7.95 (d, *J* = 7.1 Hz, 1H), 7.55 – 7.49 (m, 1H), 7.47 – 7.41 (m, 1H), 6.27 (s, 1H), 5.42 (d, *J* = 6.8 Hz, 2H), 5.36 (d, *J* = 5.8 Hz, 2H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 165.6 (C_q), 152.7 (C_q), 134.9 (C_q), 126.5 (*C*H), 126.1 (*C*H), 124.0 (*C*H), 122.1 (*C*H), 98.1 (CH), 93.4 (2 CH_2).

HRMS (ESI): m/z cald for $C_{10}H_9NO_3S$ $[M + H]^+$ 224.0376, found 224.0367.

IR(ATR): 2919, 2851, 1744, 1527, 1411, 1187, 1164, 866, 796, 439 cm^{-1}.

2-(1,4-Dioxan-2-yl) benzo[*d*]oxazole (3t)



Compound **3t** was prepared according to the **GP-3** using benzoxazole **(1g)** (47.08 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 38% (31 mg)

M.p.: 141 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 – 7.72 (m, 1H), 7.58 – 7.53 (m, 1H), 7.40 – 7.32 (m, 2H), 4.98 (dd, *J* = 9.1, 3.0 Hz, 1H), 4.21 (dd, *J* = 11.8, 3.1 Hz, 1H), 4.03 (dd, *J* = 14.1, 4.9 Hz, 1H), 4.00 – 3.91 (m, 2H), 3.82 (dd, *J* = 9.4, 2.9 Hz, 2H).

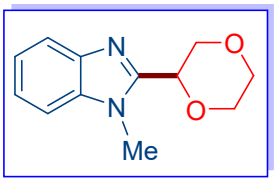
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 161.7 (C_q), 150.6 (C_q), 140.6 (C_q), 125.6 (*C*H), 124.7 (*C*H), 120.4 (*C*H), 110.9 (*C*H), 71.1 (*C*H), 68.5 (CH_2), 66.6 (CH_2), 66.4 (CH_2).

HRMS (ESI): m/z cald for $C_{11}H_{11}NO_3$ $[M + H]^+$ 206.0812, found 206.0823.

IR(ATR): 3391, 3104, 1657, 1578, 1454, 1405, 1040, 828, 472 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

2-(1,4-Dioxan-2-yl)-1-methyl-1*H*-benzo[*d*]imidazole (3u)



Compound **3u** was prepared according to the GP-3 using 1-methyl-1*H*-benzo[*d*]imidazole **(1h)** (52 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 39% (34 mg)

M.p.: 189 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.1 Hz, 1H), 7.38 – 7.32 (m, 1H), 7.32 – 7.24 (m, 2H), 4.93 (dd, *J* = 8.6, 3.9 Hz, 1H), 4.25 – 4.20 (m, 2H), 3.97 – 3.93 (m, 2H), 3.87 (s, 3H), 3.85 – 3.80 (m, 2H).

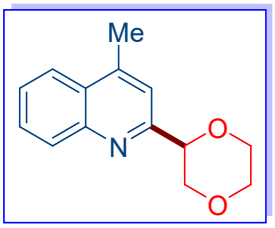
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 149.7 (C_q), 142.1 (C_q), 135.8 (C_q), 123.1 (*C*H), 122.3 (*C*H), 120.1 (*C*H), 109.3 (*C*H), 70.6 (*C*H), 68.6 (CH_2), 66.9 (CH_2), 66.5 (CH_2), 30.3 (CH_3).

HRMS (ESI): m/z cald for $C_{12}H_{14}N_2O_2$ $[M + H]^+$ 219.1128, found 219.1131.

IR(ATR): 3005, 2948, 2857, 2921, 1661, 1528, 1236, 1106, 1005, 731 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

2-(1,4-Dioxan-2-yl)-4-methylquinoline (5a)



Compound **5a** was prepared according to the **GP-4** using 4-methylquinoline **(4a)** (57.27 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: Oily liquid.

Yield: 61% (56 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 6.9 Hz, 1H), 7.98 (d, *J* = 8.3 Hz, 1H), 7.69 (m, 1H), 7.54 (m, 1H), 7.46 (s, 1H), 4.89 (dd, *J* = 10.1, 2.9 Hz, 1H), 4.23 (dd, *J* = 11.6, 2.9 Hz, 1H), 4.06 – 3.95 (m, 2H), 3.88 – 3.75 (m, 2H), 3.63 (dd, *J* = 11.6, 10.1 Hz, 1H), 2.72 (m, 3H).

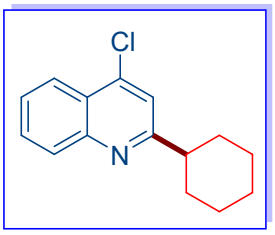
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 157.8 (C_q), 147.3 (C_q), 145.2 (C_q), 129.8 (*C*H), 129.3 (*C*H), 127.6 (*C*H), 126.2 (C_q), 123.7 (*C*H), 119.1 (*C*H), 78.8 (*C*H), 71.1 (CH_2), 67.1 (CH_2), 66.4 (CH_2), 18.9 (CH_3).

HRMS (ESI): m/z cald for $C_{14}H_{15}NO_2$ $[M + H]^+$ 230.1176, found 230.1172.

IR(ATR): 3365, 2971, 1718, 1604, 1101, 643 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

4-Chloro-2-cyclohexylquinoline (5b)



Compound **5b** was prepared according to the **GP-4** using 4-chloroquinoline **(4b)** (65.44 mg, 0.4 mmol) and cyclohexane **(2f)** (1.0 mL).

Appearance: Colourless liquid

Yield: 51% (50 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (dd, *J* = 8.3, 1.3 Hz, 1H), 8.05 (d, *J* = 8.5 Hz, 1H), 7.71 (td, *J* = 7.5, 6.9, 1.4 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.41 (s, 1H), 2.88 (tt, *J* = 12.0, 3.5 Hz, 1H), 2.07 – 1.97 (m, 2H), 1.84 (ddt, *J* = 43.4, 15.6, 3.5 Hz, 3H), 1.60 (qd, *J* = 12.4, 3.3 Hz, 2H), 1.45 (qt, *J* = 12.5, 3.3 Hz, 2H), 1.33 (tt, *J* = 12.6, 3.4 Hz, 1H).

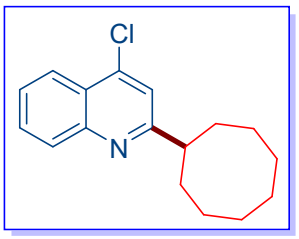
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 166.8 (C_q), 148.7 (C_q), 142.6 (C_q), 130.2 (*C*H), 129.3 (*C*H), 126.6 (*C*H), 125.1 (C_q), 123.9 (*C*H), 119.8 (*C*H), 47.4 (*C*H), 32.7 (2 *C*H_2), 26.4 (2 *C*H_2), 26.0 (*C*H_2)

HRMS (ESI): m/z cald for $C_{15}H_{16}ClN$ $[M + H]^+$ 246.1044, found 246.1049.

IR(ATR): 3173, 2925, 2850, 1493, 1551, 1408, 1011 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[7]

4-Chloro-2-cyclooctylquinoline (5c)



Compound **5c** was prepared according to the **GP-4** using 4-chloroquinoline **(4b)** (65.44 mg, 0.4 mmol) and cycloctane **(2g)** (1.0 mL).

Appearance: colorless liquid

Yield: 51% (56 mg)

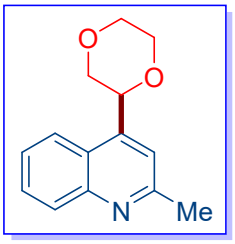
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.5 Hz, 1H), 7.65 (t, *J* = 7.7 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.31 (s, 1H), 3.11 – 3.00 (m, 1H), 1.97 – 1.87 (m, 2H), 1.80 (dt, *J* = 17.8, 8.1 Hz, 4H), 1.69 – 1.48 (m, 8H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 169.0 (C_q), 148.3 (C_q), 142.7 (C_q), 130.2 (*C*H), 129.2 (*C*H), 126.6 (*C*H), 125.0 (C_q), 123.9 (*C*H), 120.2 (*C*H), 47.4 (*C*H), 33.3 (2 *C*H_2), 26.6 (2 *C*H_2), 26.3 (CH_2), 26.0 (2 *C*H_2).

HRMS (ESI): m/z cald for $C_{17}H_{20}ClN$ $[M + H]^+$ 274.1357, found 274.1350.

IR(ATR): 2936, 2875, 1649, 1581, 1366, 1259, 1042, 598, 526 cm^{-1}.

(1,4-Dioxan-2-yl)-2-methylquinoline (5d)



Compound **5d** was prepared according to the **GP-4** using 2-methylquinoline **(4c)** (57.27 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: Sticky solid.

Yield: 61% (55 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (d, *J* = 8.5 Hz, 1H), 7.94 (d, *J* = 6.8 Hz, 1H), 7.70 – 7.64 (m, 1H), 7.54 – 7.46 (m, 2H), 5.34 (dd, *J* = 10.3, 3.0 Hz, 1H), 4.12 (dd, *J* = 11.8, 2.7 Hz, 1H), 4.08 – 4.03 (m, 2H), 3.91 – 3.86 (m, 1H), 3.80 (m, 1H), 3.46 (dd, *J* = 11.9, 9.9 Hz, 1H), 2.75 (s, 3H).

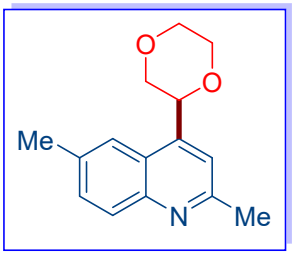
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 159.1 (C_q), 147.8 (C_q), 143.4 (C_q), 129.6 (*C*H), 129.2 (*C*H), 125.9 (*C*H), 123.5 (C_q), 122.3 (*C*H), 119.1 (*C*H), 74.2 (*C*H), 72.0 (CH_2), 67.3 (CH_2), 66.6 (CH_2), 25.5 (CH_3).

HRMS (ESI): m/z cald for $C_{14}H_{15}NO_2$ $[M + H]^+$ 230.1176, found 230.1178.

IR(ATR): 882, 909, 1111, 632, 755 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

(1,4-Dioxan-2-yl)-2,6-dimethylquinoline (5e)



Compound **5e** was prepared according to the **GP-4** using 2,6-dimethylquinoline **(4d)** (62.88 mg, 0.3 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: Sticky solid

Yield: 52% (52 mg)

^1H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.6 Hz, 1H), 7.68 (s, 1H), 7.50 (d, *J* = 10.6 Hz, 1H), 7.46 (s, 1H), 5.34 (dd, *J* = 9.9, 2.8 Hz, 1H), 4.13 (dd, *J* = 11.9, 2.8 Hz, 1H), 4.09 – 4.03 (m, 2H), 3.89 (d, *J* = 9.4 Hz, 1H), 3.85 – 3.76 (m, 1H), 3.44 (dd, *J* = 11.9, 9.9 Hz, 1H), 2.72 (s, 3H), 2.53 (s, 3H).

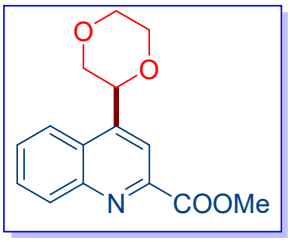
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 158.0 (C_q), 146.4 (C_q), 142.7 (C_q), 135.7 (C_q), 131.4 (*C*H), 129.3 (*C*H), 123.4 (C_q), 121.2 (*C*H), 118.9 (*C*H), 74.1 (*C*H), 72.0 (CH_2), 67.3 (CH_2), 66.6 (CH_2), 25.3 (CH_3), 21.9 (CH_3).

HRMS (ESI): m/z calcd for $C_{15}H_{17}NNaO_2$ $[M+Na]^+$ 266.1151, found 266.1151.

IR(ATR): 2993, 2913, 2859, 1672, 1603, 1277, 1172, 825 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

Methyl -4-(1,4-dioxan-2-yl) quinoline-2-carboxylate (5f)



Compound **5f** was prepared according to the **GP-4** using methyl quinoline-2-carboxylate **(4e)** (74.87 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid.

Yield: 52% (57 mg)

M.p.: 202 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40 (s, 1H), 8.34 (d, *J* = 8.6 Hz, 1H), 8.07 (d, *J* = 7.0 Hz, 1H), 7.79 (t, *J* = 8.4 Hz, 1H), 7.73 – 7.65 (m, 1H), 5.41 (dd, *J* = 9.8, 3.2 Hz, 1H), 4.13 (dd, *J* = 11.8, 2.6 Hz, 1H), 4.09-4.08 (s, 4H), 4.07 – 4.02 (m, 1H), 3.93 – 3.79 (m, 2H), 3.51 (dd, *J* = 11.9, 9.9 Hz, 1H).

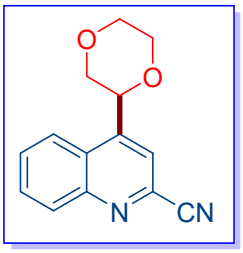
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 165.9 (C_q), 147.1 (C_q), 147.6 (C_q), 145.3 (C_q), 131.7 (*C*H), 130.0 (*C*H), 128.9 (*C*H), 126.4 (C_q), 122.5 (*C*H), 118.4 (*C*H), 74.1 (*C*H), 71.7 (CH_2), 67.3 (CH_2), 66.6 (CH_2), 53.2 (CH_3).

HRMS (ESI): m/z cald for $C_{15}H_{15}NO_4$ $[M + H]^+$ 274.1074, found 274.1062.

IR(ATR): 3303, 3117, 2975, 2924, 1744, 1698, 1435, 1492, 1527, 1456, 1243, 898 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

(1,4-Dioxan-2-yl)quinoline-2-carbonitrile (5g)



Compound **5g** was prepared according to the **GP-4** using quinoline-2-carbonitrile **(4f)** (61 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 31% (30 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (d, *J* = 8.5 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 7.87 (s, 1H), 7.77 (t, *J* = 6.9 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 5.32 (dd, *J* = 9.9, 2.9 Hz, 1H), 4.10 – 3.94 (m, 3H), 3.84 (dd, *J* = 11.8, 2.9 Hz, 1H), 3.78 – 3.70 (m, 1H), 3.35 (dd, *J* = 11.9, 9.9 Hz, 1H).

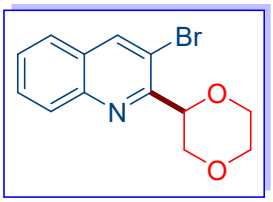
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 148.0 (C_q), 145.9 (C_q), 133.9 (C_q), 131.1 (*C*H), 130.8 (*C*H), 129.7 (*C*H), 125.7 (C_q), 122.6 (*C*H), 120.7 (*C*H), 117.5 (C_q),73.6 (*C*H), 71.8 (CH_2), 67.4 (CH_2), 66.6 (CH_2).

HRMS (ESI): m/z cald for $C_{14}H_{12}N_2O_2$ $[M + Na]^+$ 263.0791, found 263.0763.

IR(ATR): 2851, 2921, 2229, 1667, 1508, 1250, 1112, 1101, 912, 881, 765, 678 cm^{-1}**.**

The analytical data are in accordance with those reported in the literature.[1]

3-Bromo-2-(1,4-dioxan-2-yl)quinoline (5h)



Compound **5h** was prepared according to the **GP-4** using 3-bromoquinoline **(4g)** (83.22 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 55% (65 mg)

M.p.: 265 °C

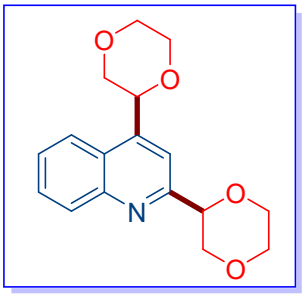
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (s, 1H), 8.19 (dq, *J* = 8.3, 1.1 Hz, 1H), 7.72 (td, *J* = 8.1, 1.3 Hz, 2H), 7.55 (m, 1H), 5.33 (dd, *J* = 9.8, 2.6 Hz, 1H), 4.20 – 3.81 (m, 6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 154.1 (C_q), 146.4 (C_q), 139.5 (*C*H), 130.0 (*C*H), 129.8 (*C*H), 128.6 (C_q), 127.8 (*C*H), 126.5 (*C*H), 116.9 (C_q), 77.11 (*C*H),69.6 (CH_2), 67.5 (CH_2), 66.3 (CH_2).

HRMS (ESI): m/z cald for $C_{13}H_{12}BrNO_2$ $[M + H]^+$ 294.0124, found 294.0132.

IR(ATR): 3339, 1637, 1452, 1262, 1115, 1015, 978, 431 cm^{-1}.

1,4-Dioxan-2-yl)-4-(1,4-dioxan-2-yl)quinoline (5i)



Compound **5i** was prepared according to the **GP-4** using quinoline **(4h)** (51.66 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid.

Yield: 62% (74 mg)

M.p.: 220 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (d, *J* = 4.3 Hz, 1H), 8.00 (dd, *J* = 8.9, 5.6 Hz, 1H), 7.82 (d, *J* = 10.4 Hz, 1H), 7.75 – 7.67 (m, 1H), 7.56 (t, *J* = 7.9 Hz, 1H), 5.42 – 5.35 (m, 1H), 4.93 (m, 1H), 4.26 (m,1H), 4.19 – 3.96 (m, 5H), 3.89 – 3.81 (m, 4H), 3.72 – 3.59 (m, 1H), 3.50 (1H).

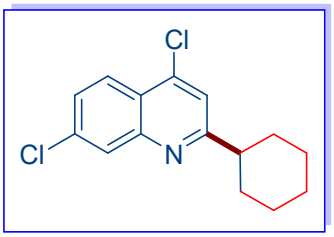
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 158.30 (*J* = 9.4 Hz) (C_q), 147.40 (*J* = 4.4 Hz) (C_q), 144.44 (*J* = 7.3 Hz) (C_q), 130.27 (*J* = 5.1 Hz) (*C*H), 129.37 (*J* = 3.6 Hz) (*C*H), 126.76 (*C*H), 124.60 (C_q), 122.46 (*J* = 5.1 Hz) (*C*H), 115.60 (*J* = 32.7 Hz) (*C*H), 78.83 (*C*H), 74.37 (*J* = 8.7 Hz) (*C*H), 71.89 (CH_2), 70.99 (*J* = 11.6 Hz) (CH_2), 67.33 (CH_2), 67.08 (*J* = 8.7 Hz) (CH_2), 66.57 (CH_2), 66.38 (*J* = 3.6 Hz) (CH_2).

HRMS (ESI): m/z cald for $C_{17}H_{19}NO_4$ $[M + H]^+$ 302.1387, found 302.1380.

IR(ATR): 2960, 2852, 1598, 1125, 1109, 1098, 892, 600 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[8]

4,7-Dichloro-2-cyclohexylquinoline (5j)



Compound **5j** was prepared according to the **GP-4** using 4,7-dichloroquinoline **(4i)** (79 mg, 0.4 mmol) and cyclohexane **(2f)** (1.0 mL).

Appearance: Colourless liquid.

Yield: 34% (38 mg)

^1H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.9 Hz, 1H), 7.99 (d, *J* = 2.1 Hz, 1H), 7.44 (dd, *J* = 8.9, 2.2 Hz, 1H), 7.33 (s, 1H), 2.79 (tt, *J* = 12.0, 3.4 Hz, 1H), 1.99 – 1.78 (m, 4H), 1.72 (d, *J* = 17.5 Hz, 1H), 1.51 (td, *J* = 12.3, 3.2 Hz, 2H), 1.38 (qt, *J* = 12.6, 3.2 Hz, 2H), 1.26 (ddd, *J* = 16.4, 12.4, 3.4 Hz, 1H).

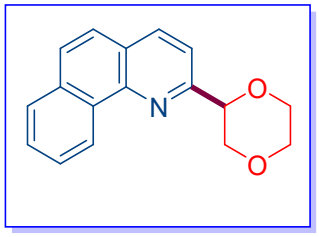
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 167.13 (C_q), 148.04 (C_q), 141.52 (C_q), 135.18 (C_q), 127.31 (*C*H), 126.52 (*C*H), 124.29 (*C*H), 122.60 (C_q), 119.11 (*C*H), 46.29 (*C*H), 31.54 (2 *C*H_2), 25.35 (2 *C*H_2), 24.93 (*C*H_2).

HRMS (ESI): m/z cald for $C_{15}H_{15}Cl_2N$ $[M + H]^+$, 280.0654 found 280.0649.

IR(ATR): 2923, 2851, 1607, 1546, 1448, 1404, 1262, 844, 879, 861, 634, 442 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[9]

(1,4-Dioxan-2-yl) benzo[*h*]quinoline (5k)



Compound **5k** was prepared according to the **GP-4** using benzo[*h*]quinoline **(4j)** (71.68 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 39% (41 mg)

M.p.: 265 °C

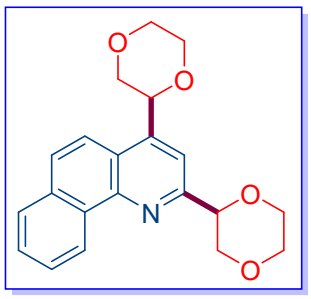
^{1}H NMR (400 MHz, $CDCl_3$): δ 9.28 (d, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 7.5 Hz, 1H), 7.80 – 7.71 (m, 3H), 7.71 – 7.64 (m, 2H), 5.03 (dd, *J* = 10.0, 3.0 Hz, 1H), 4.47 (dd, *J* = 11.6, 3.0 Hz, 1H), 4.06 – 4.01 (m, 2H), 3.90 – 3.69 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 157.0 (C_q), 145.5 (C_q), 136.5 (*C*H), 133.7 (C_q), 131.4 (C_q), 128.2 (*C*H), 127.7 (*C*H), 127.6 (*C*H), 127.0 (*C*H), 125.5 (C_q), 125.2 (*C*H), 124.5 (*C*H), 118.8 (*C*H), 78.5 (*C*H), 71.3 (CH_2), 67.1 (CH_2), 66.6 (CH_2).

HRMS (ESI): m/z cald for $C_{17}H_{15}NO_2$ $[M + H]^+$ 266.1176, found 266.1174.

IR(ATR): 2924, 2851, 1667, 1591, 1069, 1216, 1177, 972 cm^{-1}.

1,4-Dioxan-2-yl)-1,4-dioxan-2-yl) benzo[*h*]quinoline (5k′)



Compound **5k**' was prepared according to the **GP-4** using benzo[*h*]quinoline **(4j)** (71.68 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 28% (39 mg)

M.p.: 271 °C

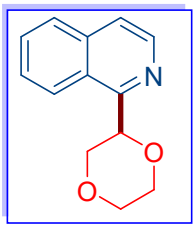
^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.30 (dd, *J* = 7.8, 3.0 Hz, 1H), 7.95 (d, *J* = 5.8 Hz, 1H), 7.90 – 7.84 (m, 2H), 7.82 (d, *J* = 9.1 Hz, 1H), 7.75 – 7.65 (m, 2H), 5.42 (dt, *J* = 10.0, 3.3 Hz, 1H), 5.02 (ddd, *J* = 13.1, 10.0, 2.9 Hz, 1H), 4.47 (ddd, *J* = 35.8, 11.6, 3.0 Hz, 1H), 4.16 – 3.99 (m, 5H), 3.93 – 3.77 (m, 4H), 3.72 (dd, *J* = 21.6, 11.6 Hz, 1H), 3.57 – 3.48 (m, 1H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 157.01 (*J* = 8.5 Hz) (C_q), 145.47 (C_q), 144.15 (*J* = 3.6 Hz) (C_q), 133.12 (C_q), 131.72 (C_q), 128.33 (*C*H), 127.94 (*C*H), 127.63 (*C*H), 127.21 (*C*H), 124.97 (*C*H), 122.22 (C_q) , 119.84 (*J* = 4.0 Hz) (*C*H), 116.02 (*C*H), 115.70 (*C*H), 78.64 ((*J* = 35.3 Hz) (*C*H), 74.58 (*J* = 6.6 Hz) (CH_2), 71.80 (CH_2), 71.33 (CH_2), 71.13 (CH_2), 67.28 (*J* = 18.2 Hz) (CH_2), 66.54 (*J* = 7.3 Hz) (CH_2).

HRMS (ESI): m/z cald for $C_{21}H_{21}NO_4$ $[M + H]^+$ 352.1543, found 352.1535.

IR(ATR): 2955, 2920, 2851, 1623, 1502, 1450, 1396, 1261, 906, 800 cm^{-1}.

(1,4-Dioxan-2-yl)isoquinoline (5l)



Compound **5l** was prepared according to the **GP-4** using isoquinoline **(4k)** (51.66 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 91% (78 mg)

M.p.: 127 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, *J* = 5.8 Hz, 1H), 8.28 (d, *J* = 8.3 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.68 – 7.54 (m, 3H), 5.43 (dd, *J* = 9.6, 3.1 Hz, 1H), 4.18 – 4.00 (m, 4H), 3.91 – 3.82 (m, 2H).

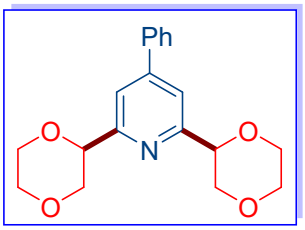
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 156.4 (C_q), 143.1 (*C*H), 135.6 (C_q), 133.9 (*C*H), 127.8 (*C*H), 127.7 (*C*H), 124.6 (CH), 122.4 (C_q), 120.0 (*C*H), 75.7 (*C*H), 70.2 (CH_2), 67.6 (CH_2), 66.5 (CH_2).

HRMS (ESI): m/z cald for $C_{13}H_{13}NO_2$ $[M + H]^+$ 216.1019, found 216.1016.

IR(ATR): 2921, 2821, 1456, 117, 754, 661, 540, 458 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

1,4-Dioxan-2-yl)-6-((*S*)-1,4-dioxan-2-yl)-4-phenylpyridine (5m)



Compound **5m** was prepared according to the **GP-4** using 4-phenylpyridine **(4i)** (62 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 48% (62 mg)

M.p.: 165 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (dd, *J* = 8.2, 1.6 Hz, 2H), 7.56 (s, 2H), 7.43 – 7.32 (m, 3H), 4.70 (dd, *J* = 10.0, 2.9 Hz, 2H), 4.12 (dt, *J* = 11.6, 3.5 Hz, 2H), 3.95 – 3.84 (m, 4H), 3.78 – 3.62 (m, 4H), 3.44 (m, *J* = 11.6, 10.1, 8.3 Hz, 2H).

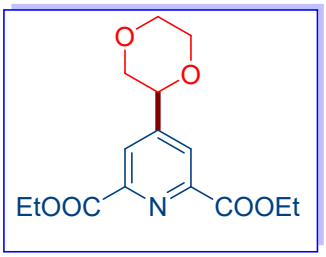
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 157.9 (C_q), 157.8 (C_q), 150.0 (C_q), 138.3 (C_q), 129.1 (*C*H), 129.0 (2 *C*H), 127.2 (2 *C*H), 117.7 (*C*H), 117.4 (*C*H), 78.1 (*C*H), 78.0 (*C*H), 71.4 (CH_2), 71.3 (CH_2), 67.0 (2 CH_2), 66.4 (2 CH_2).

HRMS (ESI): m/z cald for $C_{19}H_{21}NO_4$ $[M + Na]^+$ 350.1363, found 3250.1369.

IR(ATR): 2865, 1689, 1592, 1558, 1435, 1225, 1046, 1063, 691 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

(1,4-Dioxan-2-yl)pyridine-2,6-dicarboxylate (5n)



Compound **5n** was prepared according to the **GP-4** using diethyl pyridine-2,6-dicarboxylate **(4m)** (89 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid.

Yield: 41% (50 mg)

M.p.: 172 °C

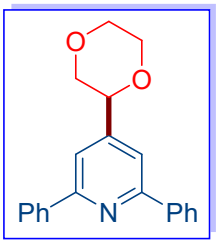
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (s, 2H), 4.78 (dd, *J* = 10.1, 2.9 Hz, 1H), 4.50 (q, *J* = 7.1 Hz, 4H), 4.05 – 3.90 (m, 3H), 3.80 (m, 2H), 3.45 – 3.36 (m, 1H), 1.46 (t, *J* = 7.1 Hz, 6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 164.7 (2 C_q), 150.2 (C_q), 149.0 (2 C_q), 125.1 (2 *C*H), 75.7 (*C*H), 71.5 (CH_2), 66.9 (CH_2), 66.3 (CH_2), 62.5 (2 CH_2), 14.3 (2 CH_3).

HRMS (ESI): m/z cald for $C_{15}H_{19}NO_6$ $[M + H]^+$310.1285, found 310.1290.

IR(ATR): 2916, 2843, 1717, 1817, 1603, 1241, 1201, 1021, 907, 881 cm^{-1}.

(1,4-Dioxan-2-yl)-2,6-diphenylpyridine (5o)



Compound **5o** was prepared according to the **GP-4** using 2,6-diphenylpyridine **(4n)** (92.51 mg, 0.4 mmol) and 1,4-dioxane (**2a**) (1.0 mL).

Appearance: White solid

Yield: 60% (76 mg)

M.p.: 225 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 7.4 Hz, 4H), 7.57 (s, 2H), 7.40 (t, *J* = 7.4 Hz, 4H), 7.33 (t, *J* = 7.3 Hz, 2H), 4.65 (dd, *J* = 10.1, 2.9 Hz, 1H), 3.94 – 3.80 (m, 3H), 3.69 (dtd, *J* = 22.6, 11.6, 3.1 Hz, 2H), 3.39 (dd, *J* = 11.7, 10.1 Hz, 1H).

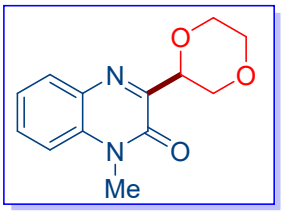
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 157.3 (2 C_q), 148.6 (C_q), 129.2 (2 C_q), 128.8 (2 *C*H), 139.4 (4 *C*H), 127.2 (4 *C*H), 116.0 (2 *C*H), 77.0 (*C*H), 72.2 (CH_2), 67.1 (CH_2), 66.5 (CH_2).

HRMS (ESI): m/z cald for $C_{21}H_{19}NO_2$ $[M + H]^+$ 318.1489, found 318.1489.

IR(ATR): 2946, 2868, 2647, 1585, 1557, 1131, 762 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

(1,4-Dioxan-2-yl)-1-methylquinoxalin-2(1*H*)-one (7a)



Compound **7a** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 52% (51 mg)

M.P: 103 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 6.5 Hz, 1H), 7.56 (dd, *J* = 9.9, 7.3 Hz, 1H), 7.38 – 7.28 (m, 2H), 5.28 (dd, *J* = 9.6, 2.7 Hz, 1H), 4.26 (dd, *J* = 11.2, 2.7 Hz, 1H), 4.09 (d, *J* = 11.8 Hz, 1H), 4.00 – 3.93 (m, 1H), 3.83 – 3.79 (m, 2H), 3.68 (s, 3H), 3.62 (dd, *J* = 11.2, 9.6 Hz, 1H).

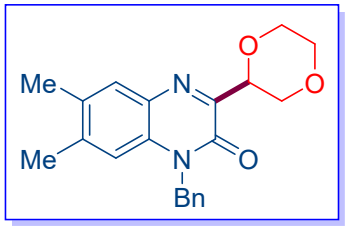
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 155.1 (C_q), 153.7 (C_q), 133.1 (C_q), 132.6 (C_q), 130.9 (*C*H), 130.7 (*C*H), 123.9 (*C*H), 113.7 (*C*H), 69.4 (*C*H),74.6 (CH_2), 67.5 (CH_2), 66.3 (CH_2), 29.0 (CH_3).

HRMS (ESI): m/z cald for $C_{13}H_{16}N_2O_2$ $[M + K]^+$ 285.0636, found 285.0630.

IR(ATR): 2932, 3036, 2855, 1572, 1296, 1210, 822, 696 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

(1,4-Dioxan-2-yl)-6,7-dimethylquinoxalin-2(1*H*)-one (7b)



Compound **7b** was prepared according to the **GP-5** using 1-benzyl-6,7-dimethylquinoxalin-2(1*H*)-one **(6b)** (105 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 69% (96 mg)

M.p.: 121 °C

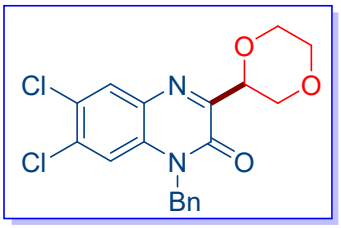
^1H NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.50 (s, 1H), 7.43 – 7.37 (m, 2H), 7.33 – 7.20 (m, 3H), 5.44 (q, *J* = 12.4 Hz, 2H), 5.14 (dd, *J* = 9.8, 2.6 Hz, 1H), 4.07 – 3.97 (m, 2H), 3.94 – 3.83 (m, 1H), 3.81 – 3.57 (m, 3H), 2.33 (s, 3H), 2.31 (s, 3H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 154.3 (C_q), 143.8 (C_q), 140.5 (C_q), 138.7 (C_q), 137.4 (C_q), 136.7 (C_q), 136.5 (C_q), 128.6 (2 *C*H), 128.6 (*C*H), 128.2 (3 *C*H), 126.3 (*C*H), 74.1 (*C*H), 69.6 (CH_2), 68.1 (CH_2), 67.6 (CH_2), 66.3 (CH_2), 20.3 (CH_3), 20.0 (CH_3).

HRMS (ESI): m/z cald for $C_{21}H_{22}N_2O_3$ $[M + H]^+$ 351.1703, found 351.1722.

IR(ATR): 3196, 2923, 2872, 1741, 1404, 1320, 1107 cm^{-1}.

1-Benzyl-6,7-dichloro-3-(1,4-dioxan-2-yl) quinoxalin-2(1*H*)-one (7c)



Compound **7c** was prepared according to the **GP-5** using 1-benzyl-6,7-dichloroquinoxalin-2(1*H*)-one **(6c)** (122 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 38% (59 mg)

M.p.: 169 °C

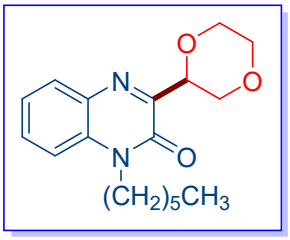
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (s, 1H), 7.96 (s, 1H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.44 – 7.33 (m, 3H), 5.61 – 5.50 (m, 2H), 5.23 (dd, *J* = 9.8, 2.6 Hz, 1H), 4.17 – 4.08 (m, 2H), 4.02 – 3.91 (m, 1H), 3.90 – 3.80 (m, 2H), 3.78-3.58 (m, 1H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 154.0 (C_q), 145.6 (C_q), 138.0 (C_q), 136.3 (C_q), 134.6 (C_q), 133.5 (C_q), 130.0 (C_q), 128.8 (*C*H), 127.7 (2 *C*H), 127.4 (*C*H), 127.3 (2 *C*H), 126.6 (*C*H), 73.0 (*C*H), 68.4 (CH_2), 67.8 (CH_2), 66.5 (CH_2), 65.3 (CH_2).

HRMS (ESI): m/z cald for $C_{19}H_{16}Cl_2N_2O_3$ $[M + H]^+$ 391.0611, found 391.0614.

IR(ATR): 2921, 2852, 1735, 1461, 1376, 1083, 927, 884, 706 cm^{-1}.

(1,4-Dioxan-2-yl)-1-hexylquinoxalin-2(1*H*)-one (7d)



Compound **7d** was prepared according to the **GP-5** using 1-hexylquinoxalin-2(1*H*)-one **(6d)** (92 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: sticky liquid

Yield: 46% (58 mg)

M.p.: 132 °C

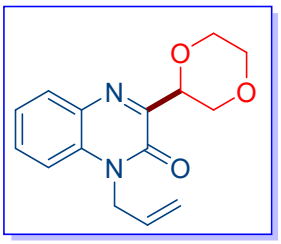
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 8.3 Hz, 1H), 7.80 (d, *J* = 9.9 Hz, 1H), 7.68 – 7.62 (m, 1H), 7.58 – 7.51 (m, 1H), 5.24 (dd, *J* = 9.8, 2.6 Hz, 1H), 4.51 (q, *J* = 6.6 Hz, 2H), 4.16 (td, *J* = 10.9, 2.6 Hz, 2H), 4.02 (td, *J* = 11.6, 11.1, 3.5 Hz, 1H), 3.97 – 3.82 (m, 2H), 3.71 (dd, *J* = 11.4, 9.7 Hz, 1H), 1.92 – 1.82 (m, 2H), 1.50 (p, *J* = 7.3 Hz, 2H), 1.37 (q, *J* = 3.2 Hz, 4H), 0.96 – 0.89 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 154.9 (C_q), 145.2 (C_q), 140.3 (C_q), 138.4 (C_q),130.0 (*C*H), 129.2 (*C*H), 126.8 (*C*H), 126.6 (*C*H), 74.3 (*C*H), 69.6 (*C*H_2), 67.6 (*C*H_2), 66.9 (*C*H_2), 66.4 (*C*H_2), 31.5 (*C*H_2), 28.7 (*C*H_2), 25.8 (*C*H_2), 22.6 (*C*H_2), 14.0 (*C*H_3).

HRMS (ESI): m/z cald for $C_{18}H_{24}N_2O_3$ $[M + Na]^+$, 339.1679 found 339.1667.

IR(ATR): 2924, 2871, 1743, 1403, 1466, 1239, 1219, 1149, 1065, 660 cm^{-1}.

1-Allyl-3-(1,4-dioxan-2-yl) quinoxalin-2(1*H*)-one (7e)



Compound **7e** was prepared according to the **GP-5** using 1-allylquinoxalin-2(1*H*)-one **(6e)** (74 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: White solid

Yield: 66% (71 mg)

M.p.: 132 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.42 (t, *J* = 8.8, 7.3 Hz, 1H), 7.26 – 7.15 (m, 2H), 5.86 – 5.74 (m, 1H), 5.22 – 5.12 (m, 2H), 5.03 (d, *J* = 17.3 Hz, 1H), 4.86

– 4.70 (m, 2H), 4.15 (dd, *J* = 11.2, 2.7 Hz, 1H), 3.99 (d, *J* = 11.6 Hz, 1H), 3.94 – 3.81 (m, 1H), 3.76 – 3.68 (m, 2H), 3.55 (dd, *J* = 11.3, 9.5 Hz, 1H).

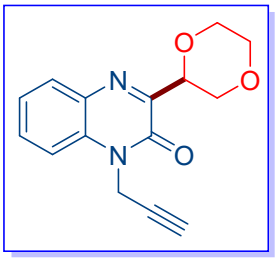
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 155.1 (C_q), 153.3 (C_q), 132.8 (C_q), 132.3 (C_q), 130.8 (2 *C*H), 130.3 (*C*H), 123.9 (*C*H), 118.3 (*C*H), 114.2 (*C*H), 74.6 (*C*H), 69.5 (CH_2), 67.5 (CH_2), 66.3 (CH_2), 44.5 (CH_2).

HRMS (ESI): m/z cald for $C_{15}H_{16}N_2O_3$ $[M + H]^+$ 273.1234, found 273.1233.

IR(ATR): 2927, 2853, 1661, 751, 1301, 1079, 1184 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[8]

(1,4-Dioxan-2-yl)-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one (7f)



Compound **7f** was prepared according to the **GP-5** using 1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one **(6f)** (73 mg, 0.4 mmol) and 1,4-dioxane **(2a)** (1.0 mL).

Appearance: Oily pale-yellow liquid

Yield: 38% (41 mg)

^1H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, *J* = 8.3 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 1H), 7.60 (t, *J* = 8.4 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 5.20 (dd, *J* = 9.8, 2.6 Hz, 1H), 5.15 (dd, *J* = 15.5, 2.5 Hz, 1H), 5.05 (dd, *J* = 15.4, 2.4 Hz, 1H), 4.15 – 4.04 (m, 2H), 3.95 (td, *J* = 11.8, 11.0, 3.7 Hz, 1H), 3.86 – 3.75 (m, 2H), 3.65 (dd, *J* = 11.4, 9.7 Hz, 1H), 2.45 (t, *J* = 2.4 Hz, 1H).

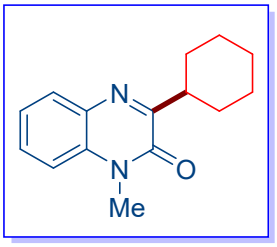
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$): δ 152.3 (C_q), 143.8 (C_q), 138.7 (C_q), 137.8 (C_q), 129.2 (*C*H), 128.2 (*C*H), 126.2 (*C*H), 125.9 (*C*H), 77.1 (C_q), 74.2 (*C*H), 73.0 (CH_2), 68.5 (CH_2), 66.6 (CH_2), 65.3 (CH_2), 53.0 (*C*H).

HRMS (ESI): m/z cald for $C_{15}H_{14}N_2O_3$ $[M + Na]^+$, 293.0897 found 293.0894.

IR(ATR): 2875, 2049, 1687, 1580, 1534, 1428, 1366, 1259, 869 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[8]

3-Cyclohexyl-1-methylquinoxalin-2(1*H*)-one (7g)



Compound **7g** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.4 mmol) and cyclohexane **(2f)** (1.0 mL).

Appearance: Oily liquid

Yield: 46% (45 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.43 (t, *J* = 6.9 Hz, 1H), 7.28 – 7.20 (m, 2H), 3.62 (s, 3H), 3.26 (tt, *J* = 11.5, 3.3 Hz, 1H), 1.88 (d, *J* = 12.1 Hz, 2H), 1.79 (dt, *J* = 12.9, 3.3 Hz, 2H), 1.71 (s, 1H), 1.56 – 1.33 (m, 4H), 1.28 – 1.20 (m, 1H).

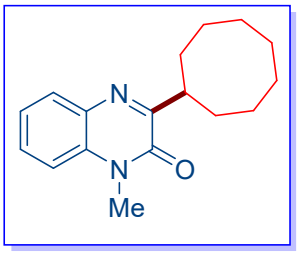
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 164.3 (C_q), 154.5 (C_q), 132.9 (C_q), 132.8 (C_q), 129.8 (*C*H), 129.4 (*C*H), 123.4 (*C*H), 113.5 (*C*H), 40.8 (*C*H), 30.5 (2 *C*H_2), 29.1 (*C*H_2), 26.3 (2 *C*H_2), 26.2 (*C*H_3).

HRMS (ESI): m/z cald for $C_{15}H_{18}N_2O$ $[M + H]^+$ 243.1492, found 243.1492.

IR(ATR): 3076, 2924, 2850, 1735,1643, 1599, 1590, 1470, 1447, 725, 457 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

3-Cyclooctyl-1-methylquinoxalin-2(1*H*)-one (7h)



Compound **7h** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.4 mmol) and cycloctane **(2g)** (1.0 mL).

Appearance: White liquid

Yield: 42% (45 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.53 – 7.47 (m, 1H), 7.33 (d, *J* = 7.5 Hz, 1H), 7.31 – 7.26 (m, 1H), 3.70 (s, 3H), 3.56 (p, *J* = 6.4 Hz, 1H), 1.92 – 1.76 (m, 6H), 1.74 – 1.58 (m, 8H).

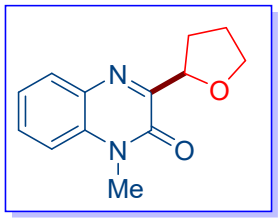
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 165.8 (C_q), 154.51 (C_q), 132.9 (C_q), 132.7 (C_q), 129.7 (*C*H), 129.3 (*C*H), 123.4 (*C*H), 113.5 (*C*H), 40.4 (*C*H), 30.6 (2 *C*H_2), 29.1 (*C*H_2), 26.7 (2 *C*H_2), 26.6 (CH_3), 25.9 (2 *C*H_2).

HRMS (ESI): m/z cald for $C_{17}H_{22}N_2O$ $[M + H]^+$ 271.1805, found 271.1807.

IR(ATR): 2919, 2850, 2248, 1650, 1600, 1471, 1446, 1310, 750, 729, 457 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

1-Methyl-3-(tetrahydrofuran-2-yl)quinoxalin-2(1*H*)-one (7i)



Compound **7i** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.4 mmol) and tetrahydrofuran **(2p)** (1.0 mL).

Appearance: White solid.

Yield: 58 % (53 mg)

M.p.: 123 °C

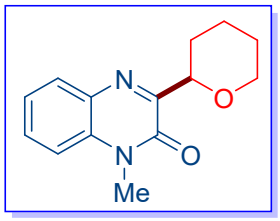
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.47 (m, 1H), 7.29 – 7.20 (m, 2H), 5.31 (dd, *J* = 7.7, 5.9 Hz, 1H), 4.19 – 4.12 (m, 1H), 3.97 – 3.90 (m, 1H), 3.62 (s, 3H), 2.48 – 2.36 (m, 1H), 2.02 – 1.90 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 159.4 (C_q), 154.0 (C_q), 133.1 (C_q), 132.5 (C_q), 130.4 (*C*H), 130.2 (*C*H), 123.7 (*C*H), 113.6 (*C*H), 77.6 (*C*H), 69.2 (*C*H_2), 30.5 (*C*H_2), 28.8 (*C*H_2), 25.6 (*C*H_3).

HRMS (ESI): m/z cald for $C_{13}H_{14}N_2O_2$ $[M + Na]^+$ 253.0947, found 253.0950.

IR(ATR): 2919, 2850, 1650, 1600, 1471, 1446, 1310, 907, 729 cm^{-1}.

1-Methyl-3-(tetrahydro-2*H*-pyran-2-yl) quinoxalin-2(1*H*)-one (7j)



Compound **7j** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.4 mmol) and tetrahydropyran **(2q)** (1.0 mL).

Appearance: White solid

Yield: 61% (60 mg)

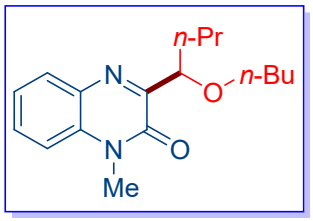
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 9.8 Hz, 1H), 7.48 (t, *J* = 8.7 Hz, 1H), 7.31 – 7.19 (m, 2H), 4.92 (dd, *J* = 10.9, 2.2 Hz, 1H), 4.26 – 4.18 (m, 1H), 3.63 (s, 3H), 2.08 (d, *J* = 12.6 Hz, 1H), 1.91 (d, *J* = 13.0 Hz, 1H), 1.73 (td, *J* = 12.4, 3.6 Hz, 3H), 1.59 – 1.47 (m, 2H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 158.8 (C_q), 153.7 (C_q), 133.0 (C_q), 132.7 (C_q), 130.6 (*C*H), 130.3 (*C*H), 123.7 (*C*H), 113.5 (*C*H),76.5 (*C*H), 69.5 (CH_2), 30.2 (CH_2), 29.0 (CH_2), 25.6 (CH_2), 23.6 (CH_3).

HRMS (ESI): m/z cald for $C_{14}H_{16}N_2O_2$ $[M + H]^+$ 245.1285, found 245.1293.

IR(ATR): 2920, 2851, 1637, 1593, 1458, 1224, 1025, 726 cm^{-1}.

3-(1-Butoxybutyl)-1-methylquinoxalin-2(1*H*)-one (7k)



Compound **7k** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.3 mmol) and dibutylether **(2r)** (1.0 mL).

Appearance: White solid

Yield: 57% (66 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 9.8 Hz, 1H), 7.49 (t, *J* = 6.8 Hz, 1H), 7.33 – 7.23 (m, 2H), 4.88 (dd, *J* = 8.5, 4.3 Hz, 1H), 3.64 (s, 3H), 3.52 (dt, *J* = 9.4, 6.3 Hz, 1H), 3.29 (dt, *J* = 9.4, 6.8 Hz, 1H), 1.82 – 1.64 (m, 3H), 1.61 – 1.45 (m, 3H), 1.39 – 1.28 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.83 (t, *J* = 7.4 Hz, 3H).

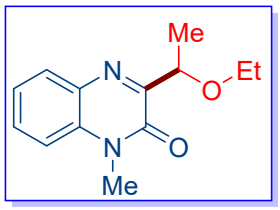
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 160.1 (C_q), 154.4 (C_q), 133.1 (C_q), 132.8 (C_q), 130.5 (*C*H), 130.2 (*C*H), 123.7 (*C*H), 113.4 (*C*H), 70.0 (*C*H), 36.2 (CH_2), 32.0 (CH_2), 28.9 (CH_2), 19.3 (2 CH_2), 13.9 (CH_3), 13.9 (CH_3).

HRMS (ESI): m/z cald for $C_{17}H_{24}N_2O_2$ $[M + H]^+$ 289.1911, found 289.1911.

IR(ATR): 2957, 2927, 2869, 1651, 1601, 1471, 1086, 751, 475 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

3-(1-Ethoxyethyl)-1-methylquinoxalin-2(1*H*)-one (7l)



Compound **7l** was prepared according to the **GP-5** using 1-methylquinoxalin-2(1*H*)-one **(6a)** (64 mg, 0.4 mmol) and diethylether **(2c)** (1.5 mL).

Appearance: White solid.

Yield: 39% (36 mg)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.0 Hz, 1H), 7.50 (t, *J* = 8.7 Hz, 1H), 7.33 – 7.23 (m, 2H), 5.05 (q, *J* = 6.6 Hz, 1H), 3.64 (s, 3H), 3.62 – 3.45 (m, 2H), 1.47 (d, *J* = 6.6 Hz, 3H), 1.20 (t, *J* = 7.0 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 160.1 (C_q), 154.3 (C_q), 133.1 (C_q), 132.7 (C_q), 130.5 (*C*H), 130.3 (*C*H), 123.7 (*C*H), 113.6 (*C*H), 73.2 (*C*H), 65.2 (CH_3), 29.0 (CH_2), 19.4 (CH_3), 15.5 (CH_3).

HRMS (ESI): m/z cald for $C_{13}H_{16}N_2O_2$ $[M + H]^+$ 233.1285, found 233.1281.

IR(ATR): 2924, 2850, 1643, 1599, 1590, 1470, 1447, 752 cm^{-1}.

The analytical data are in accordance with those reported in the literature.[1]

8. Reference

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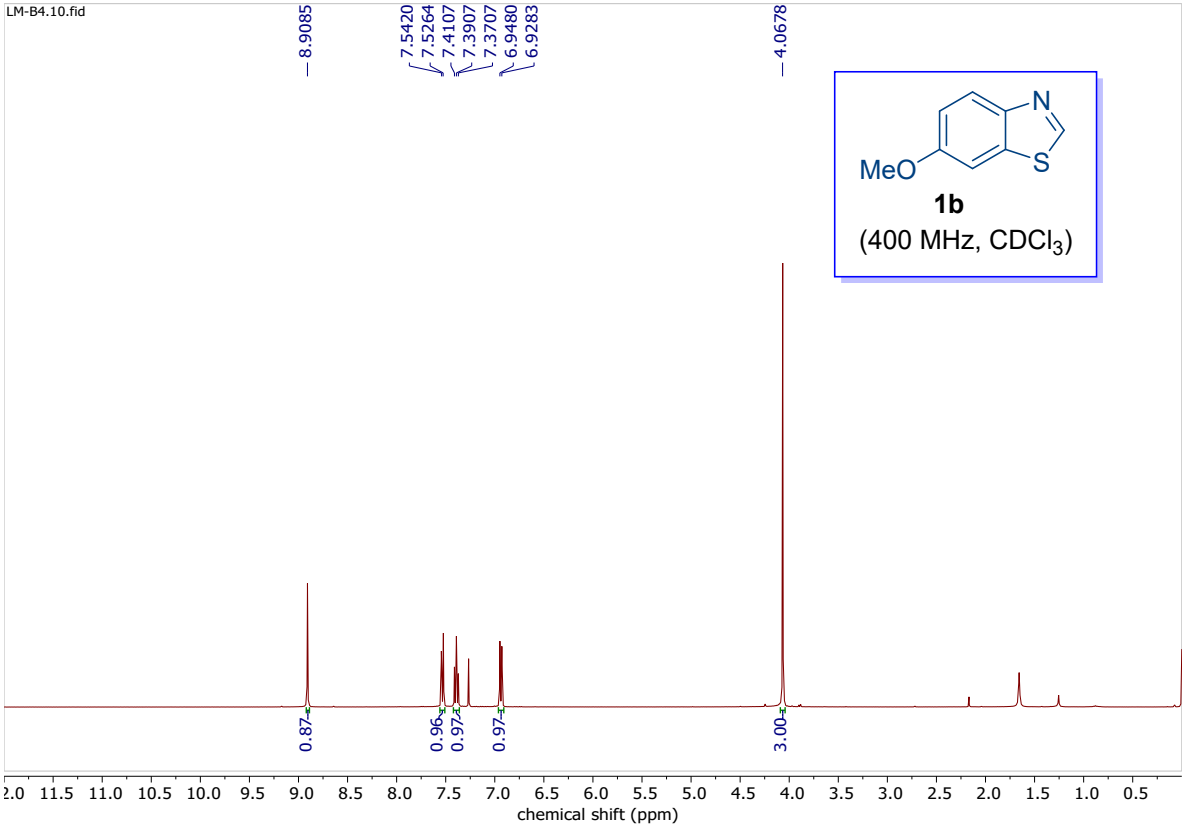
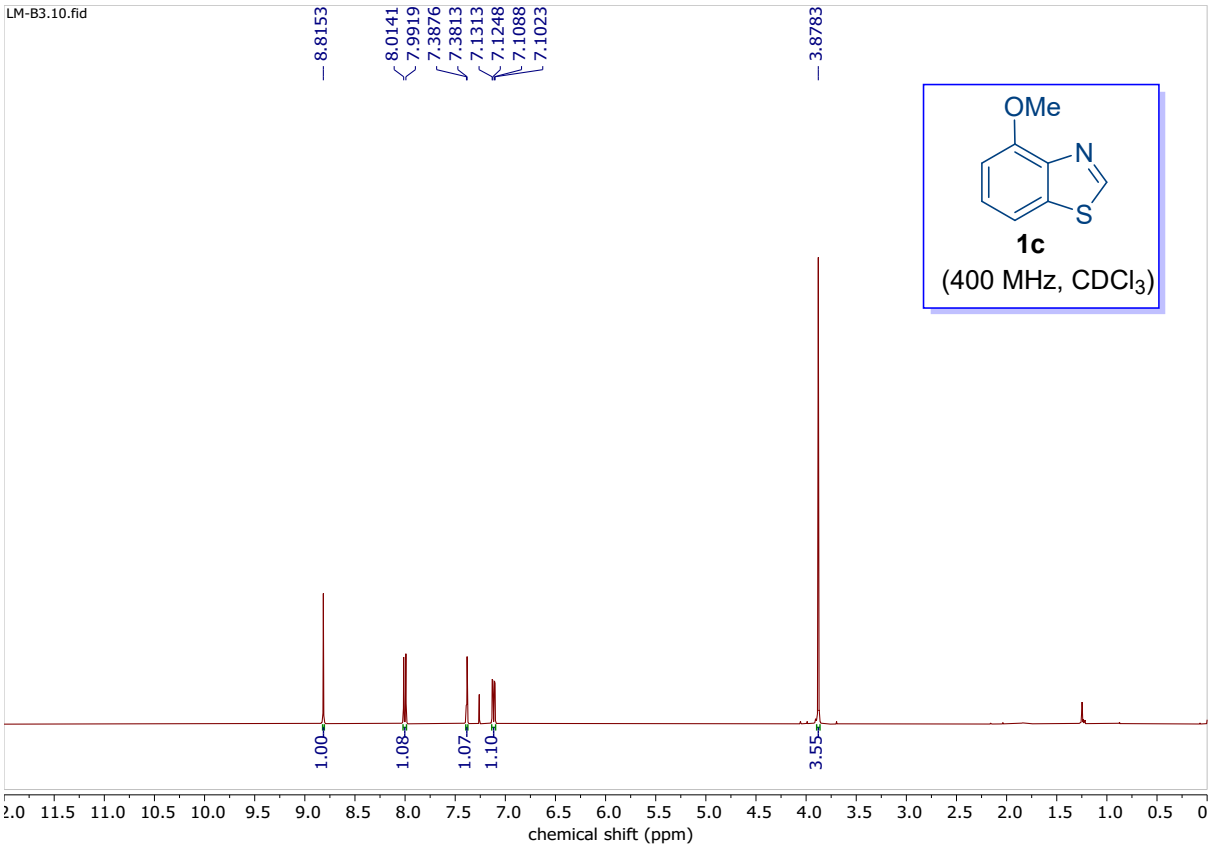
9. NMR Spectra of starting materials

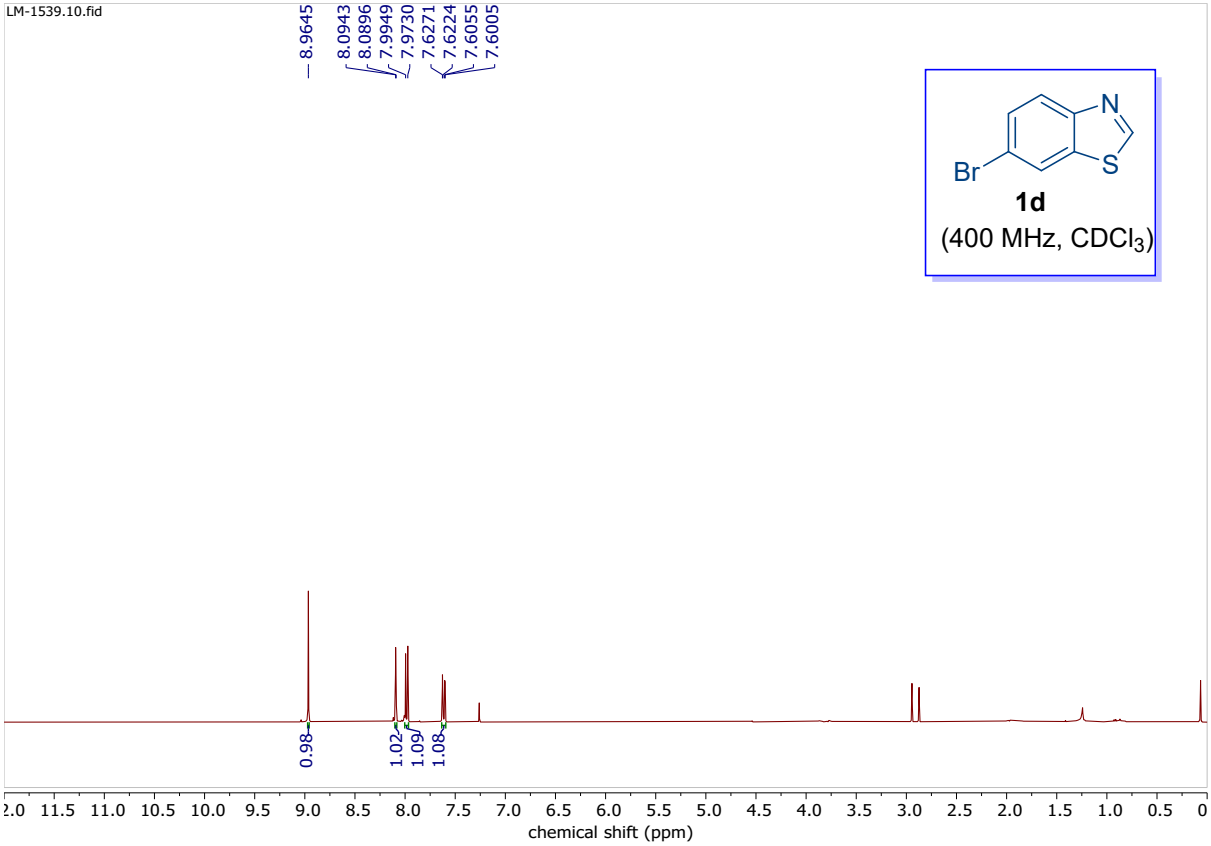
^{1}H NMR spectra of compound **1b**.



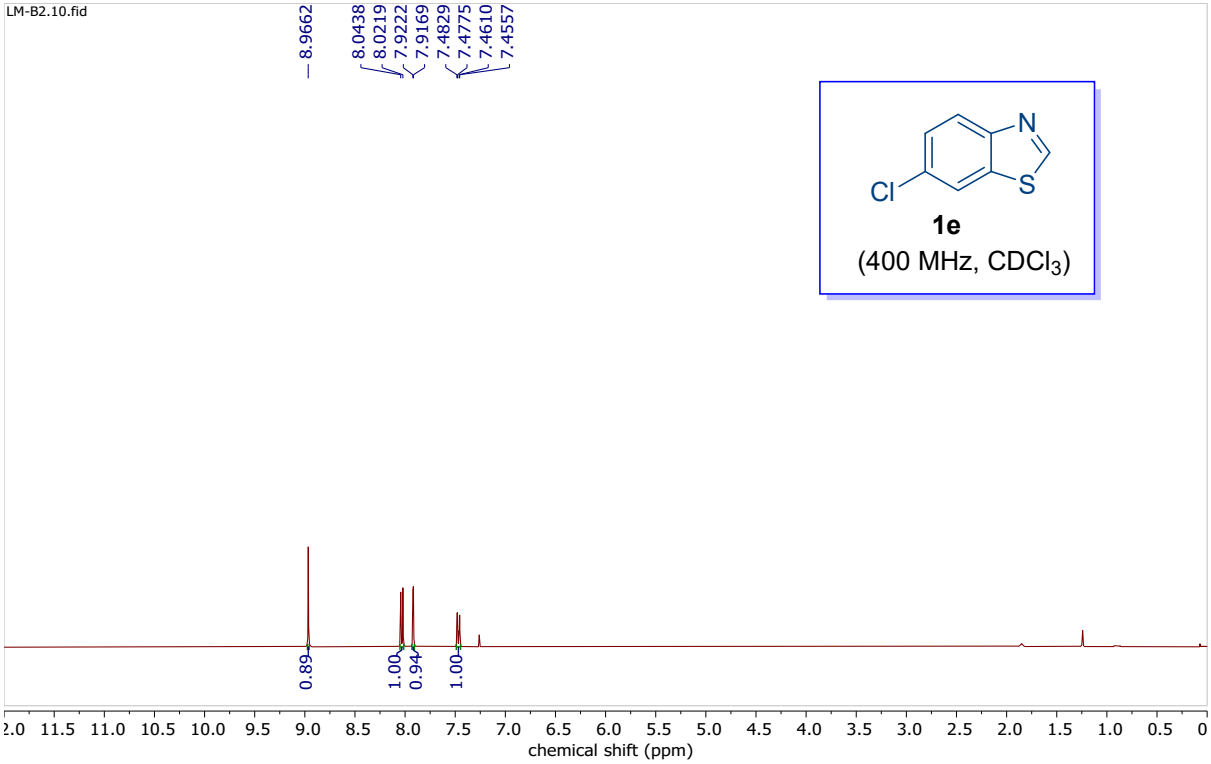
^{1}H NMR spectra of compound **1c**.



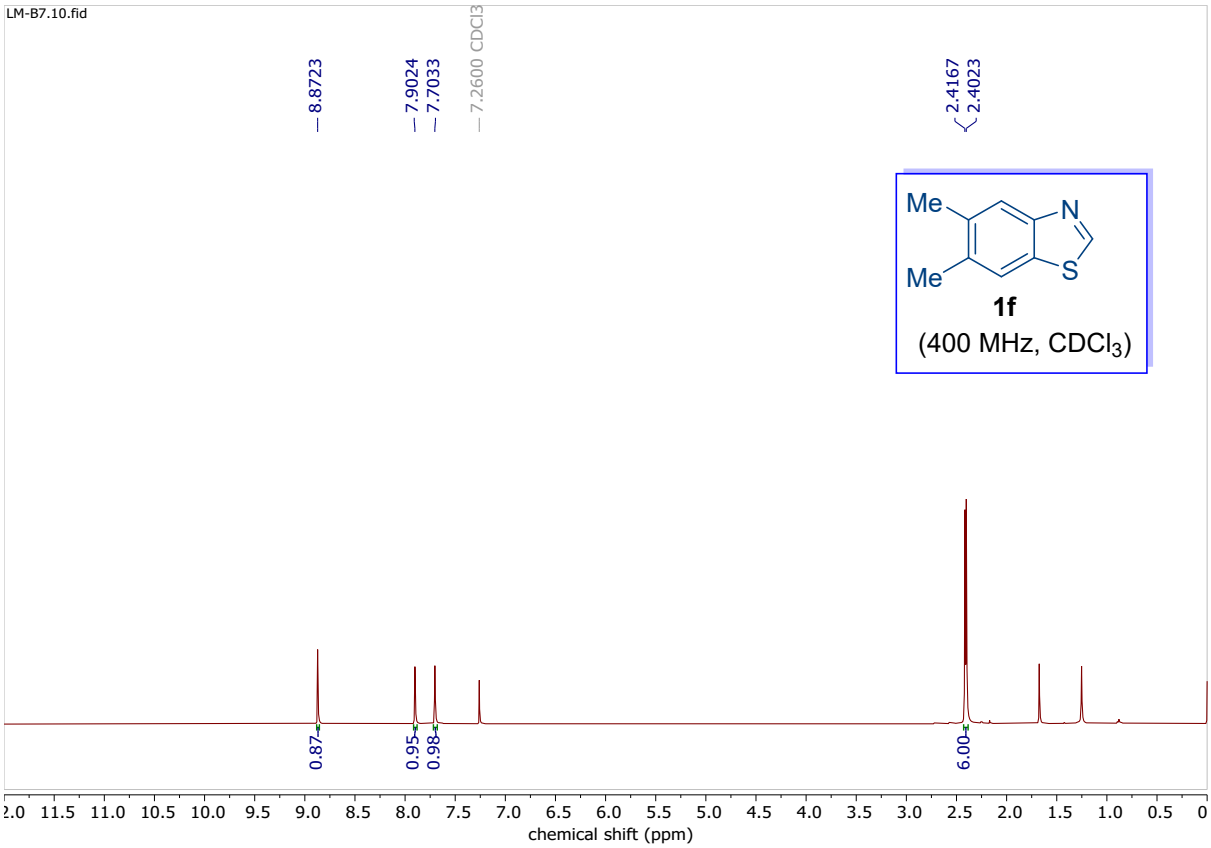
^{1}H NMR spectra of compound **1d**.



^{1}H NMR spectra of compound **1e**.

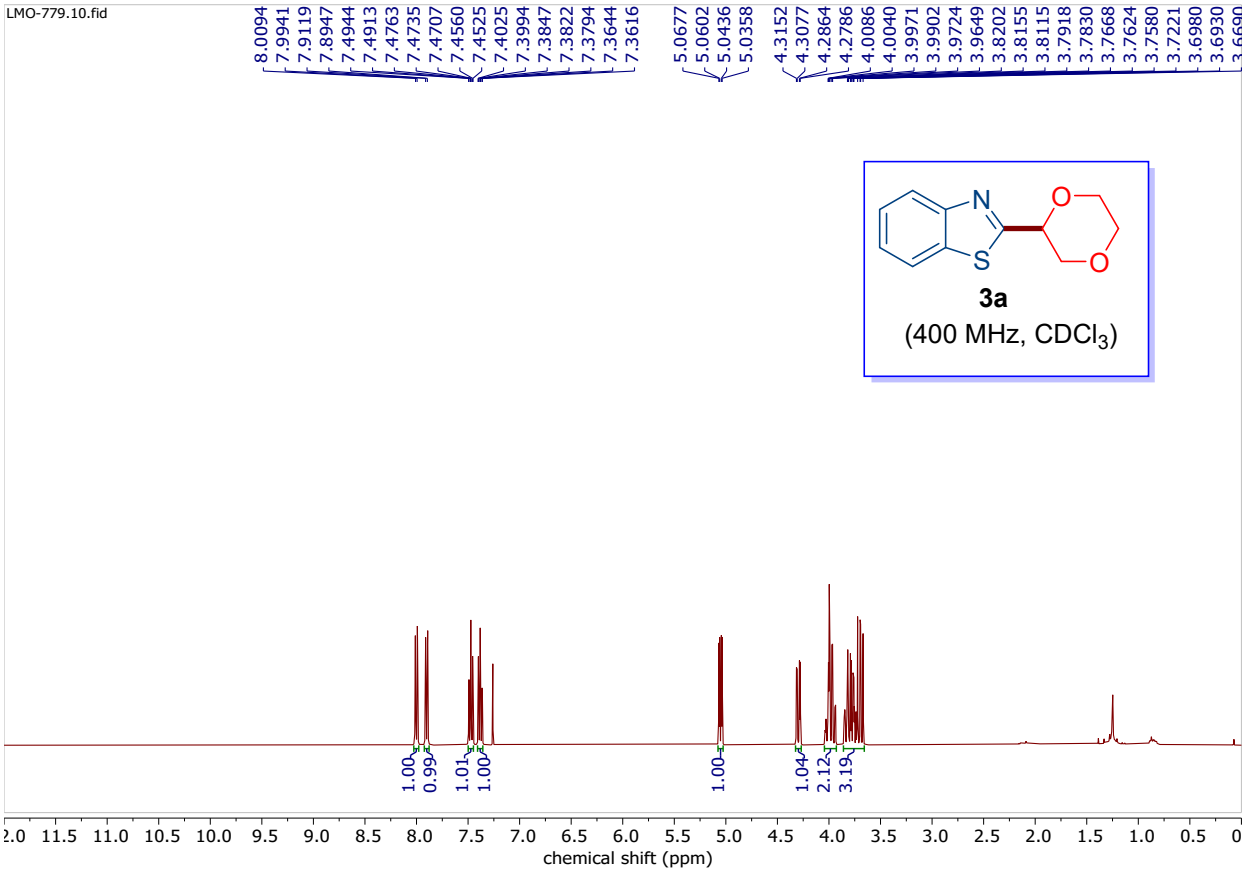


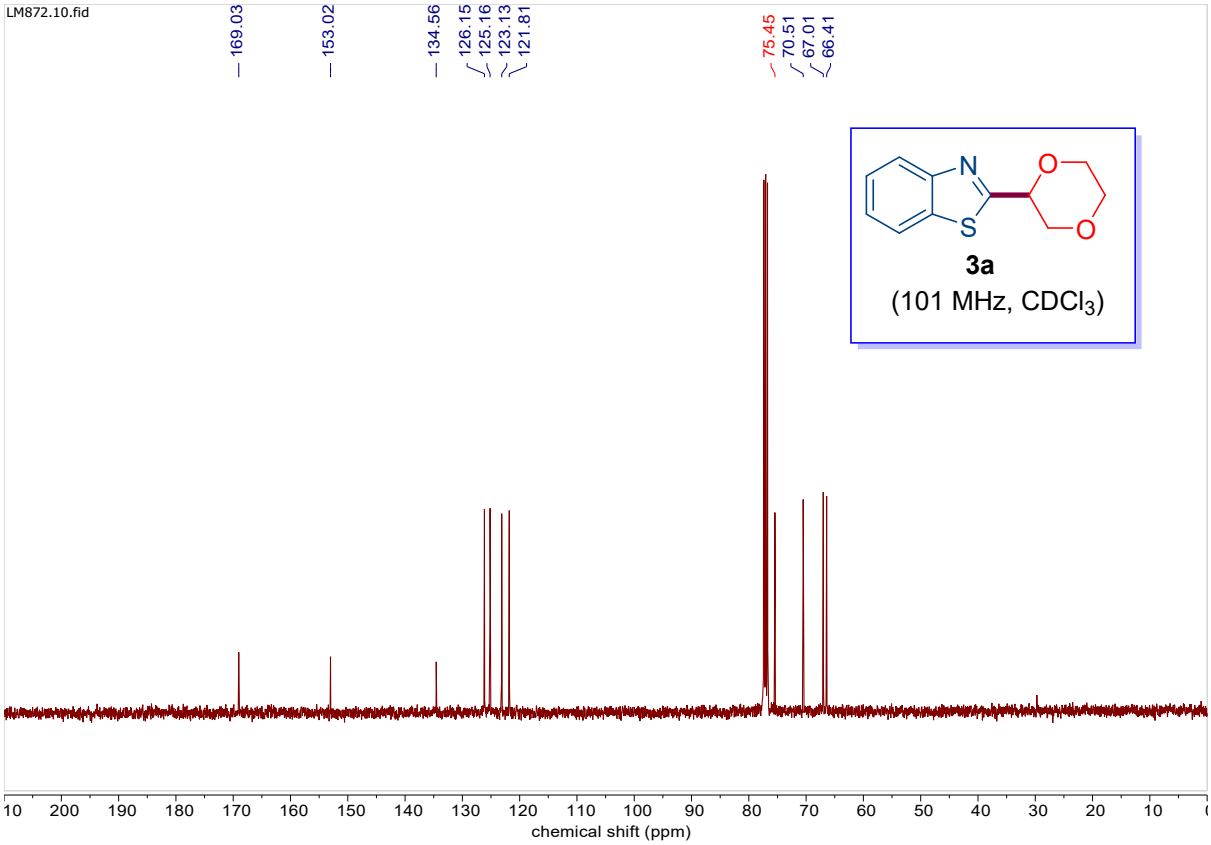
^{1}H NMR spectra of compound **1f**.



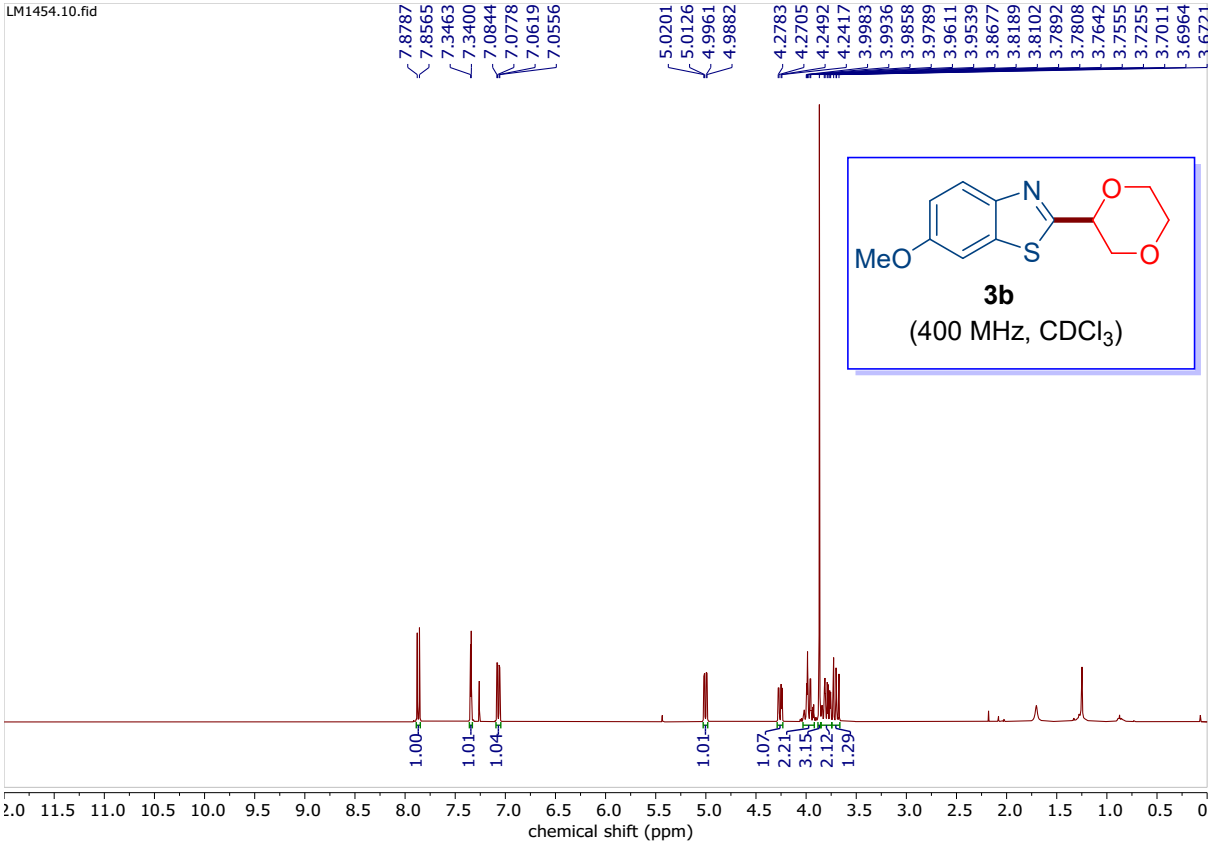
10. NMR spectra of products

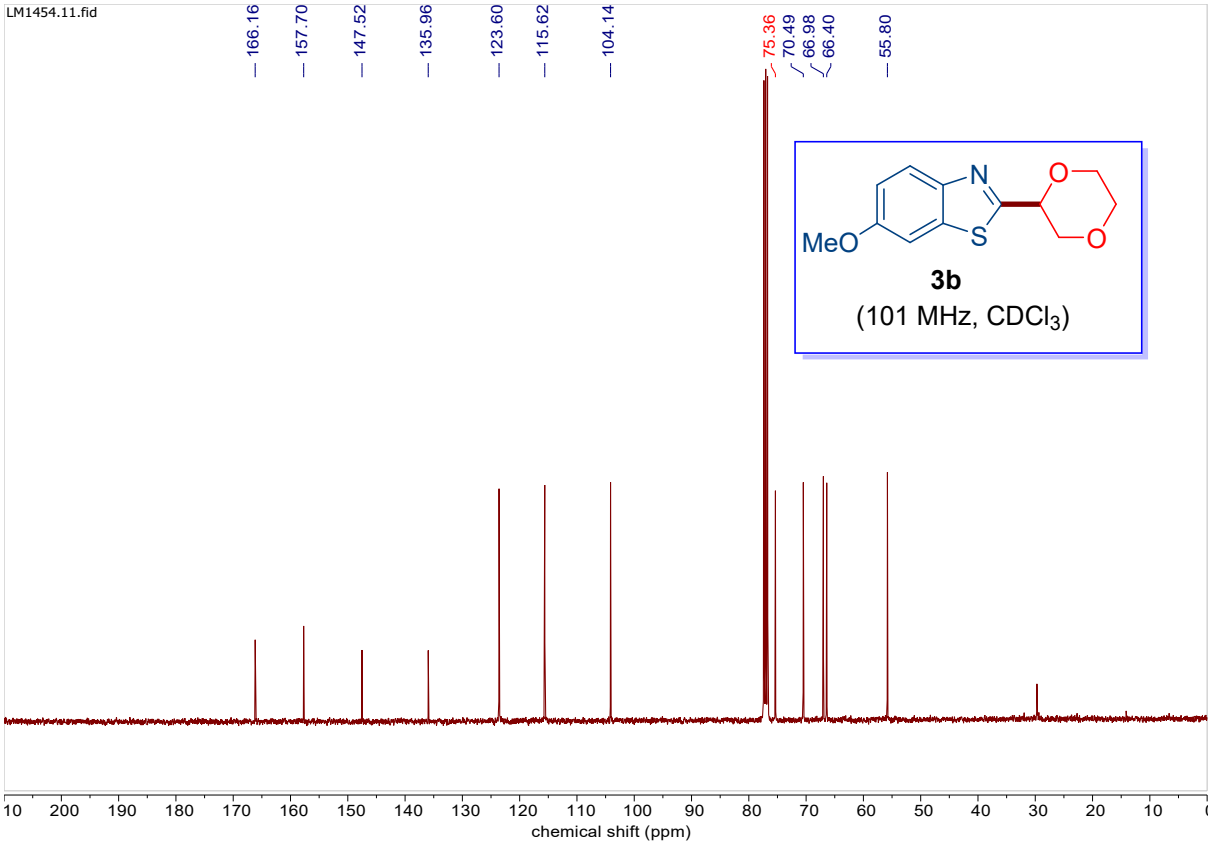
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3a**.



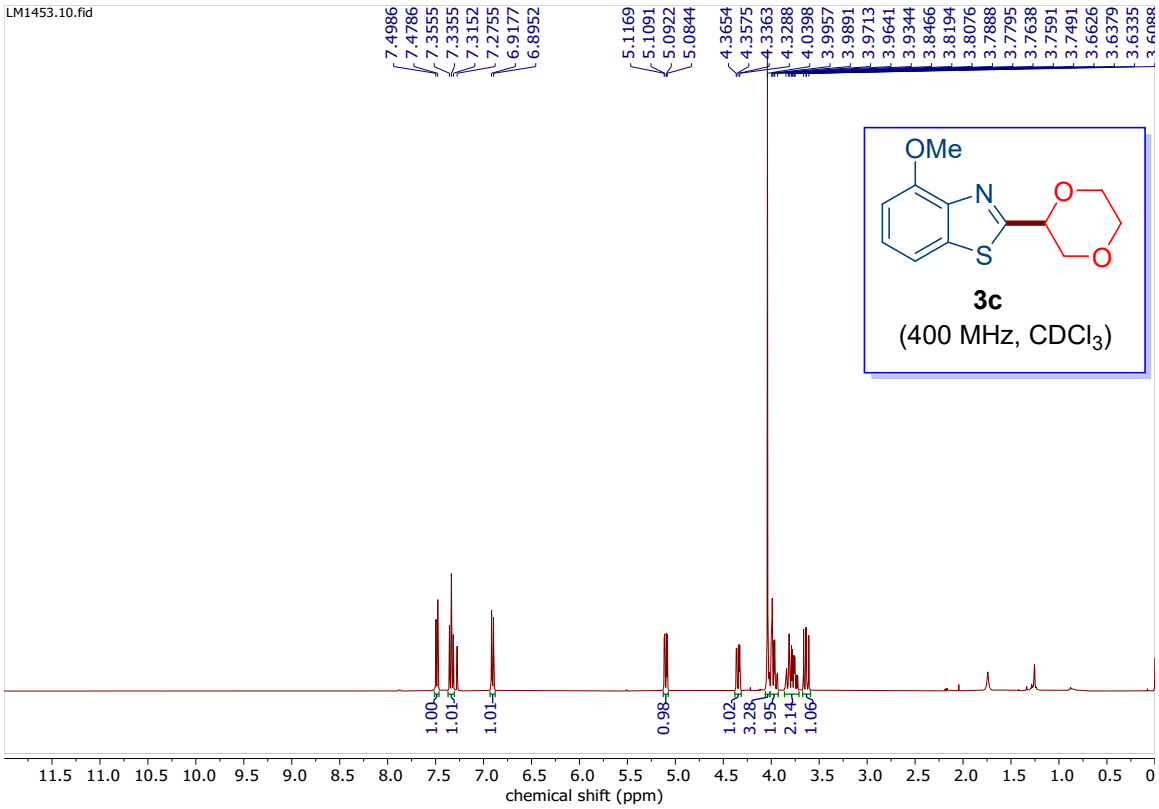


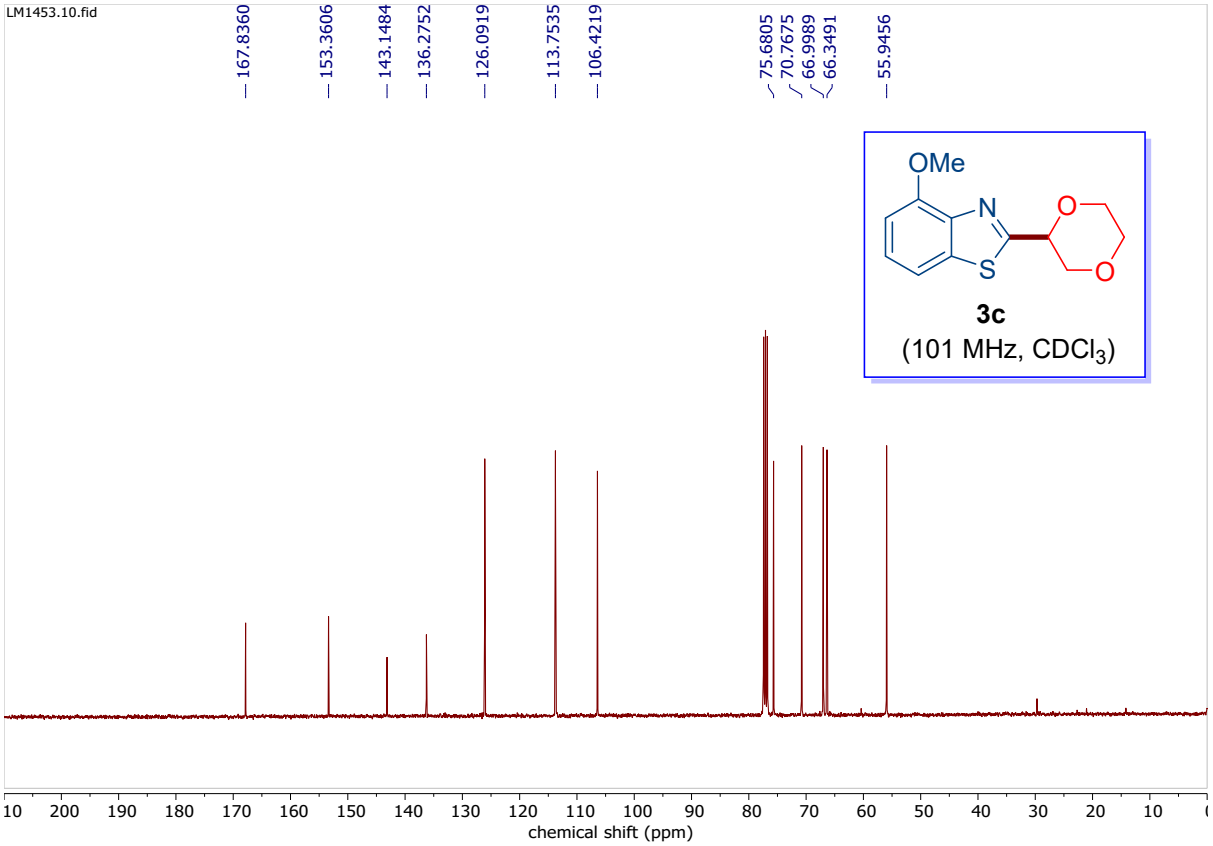
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3b**.



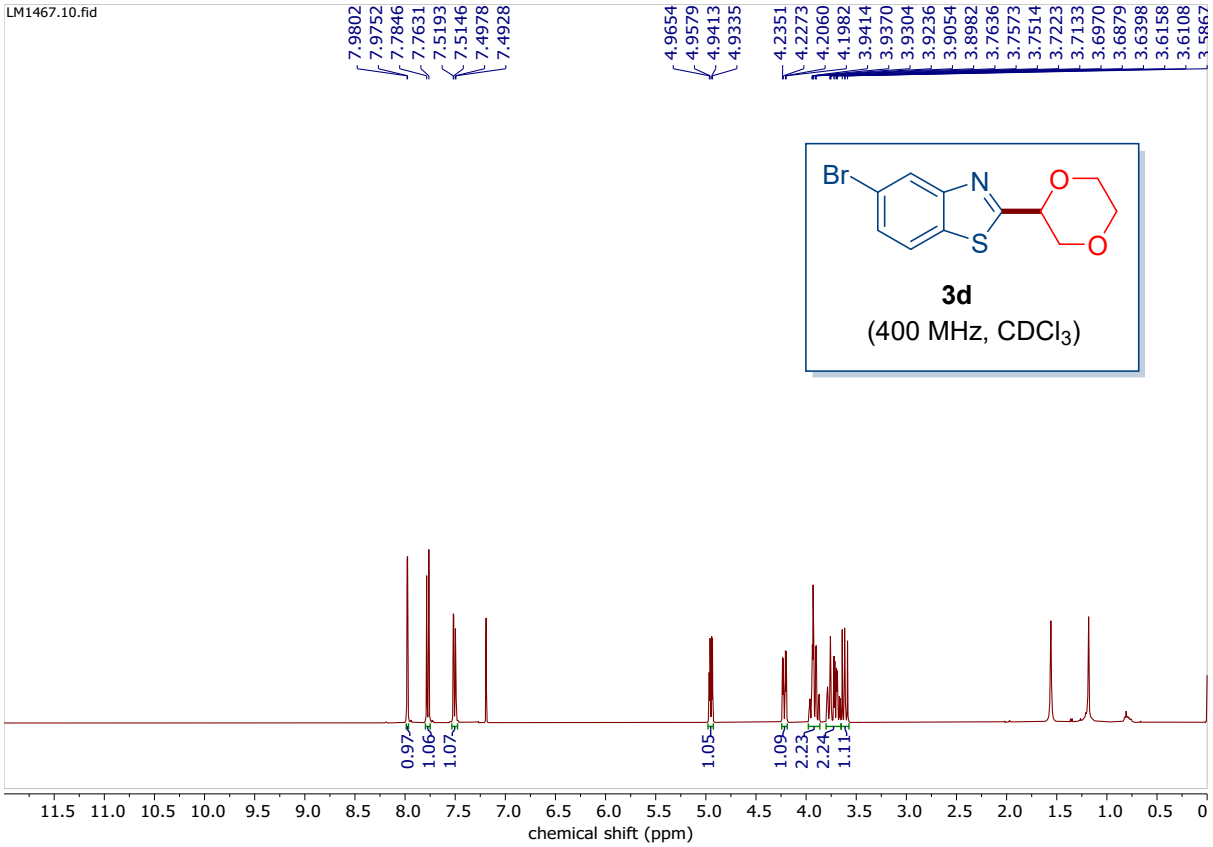


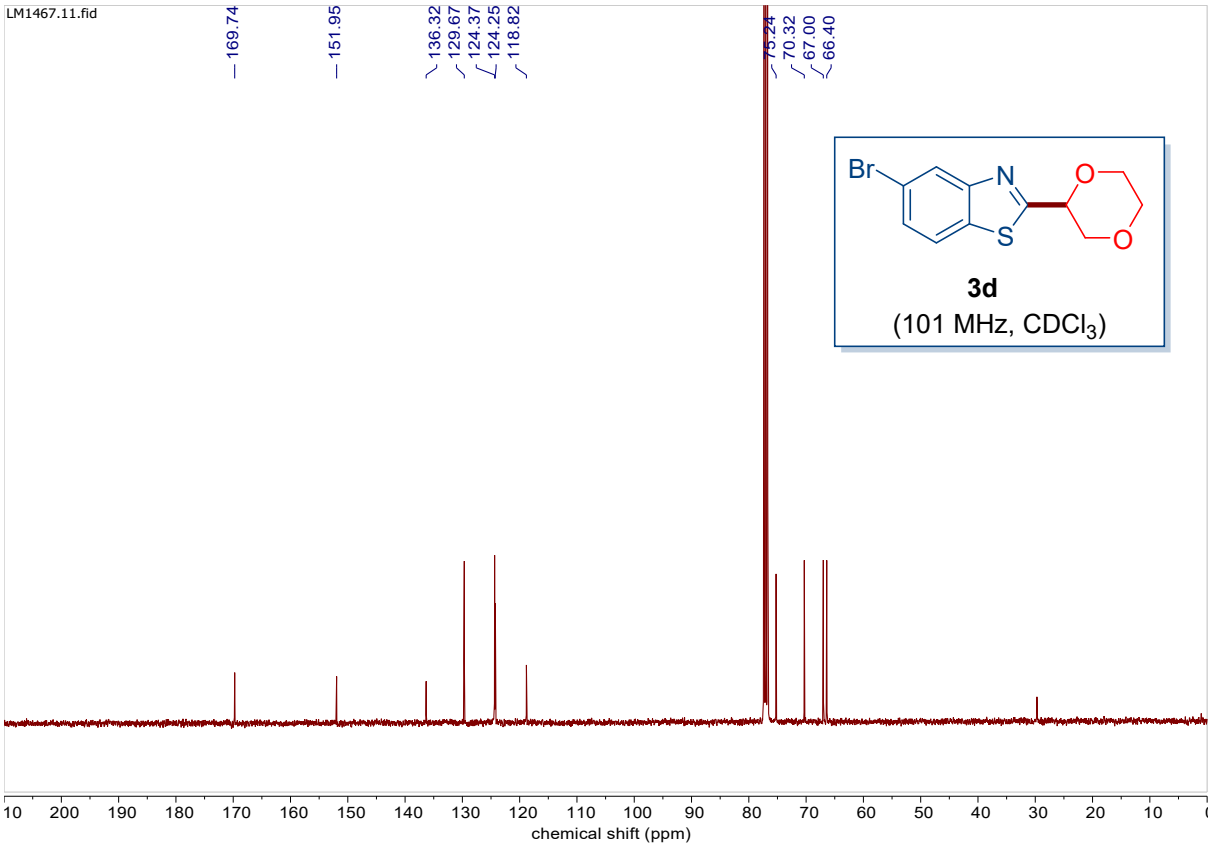
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3c**.



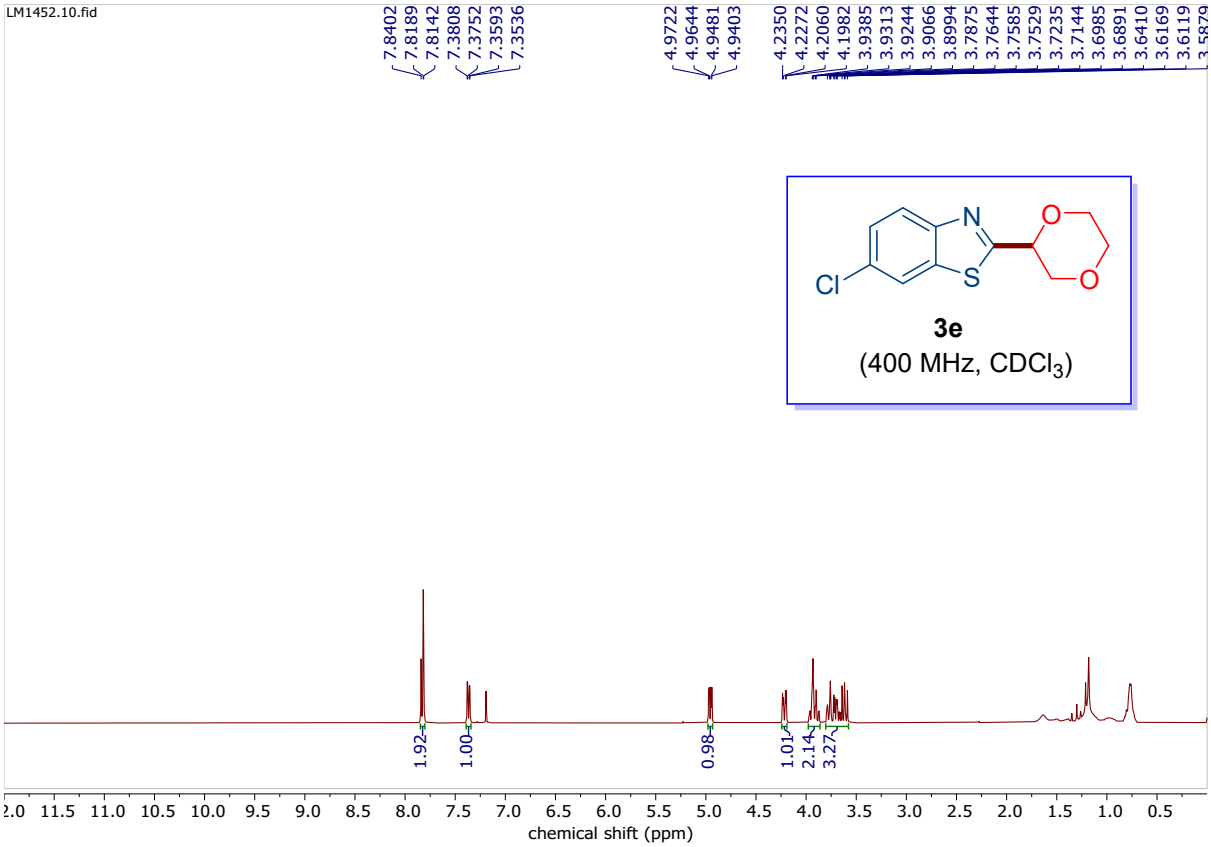


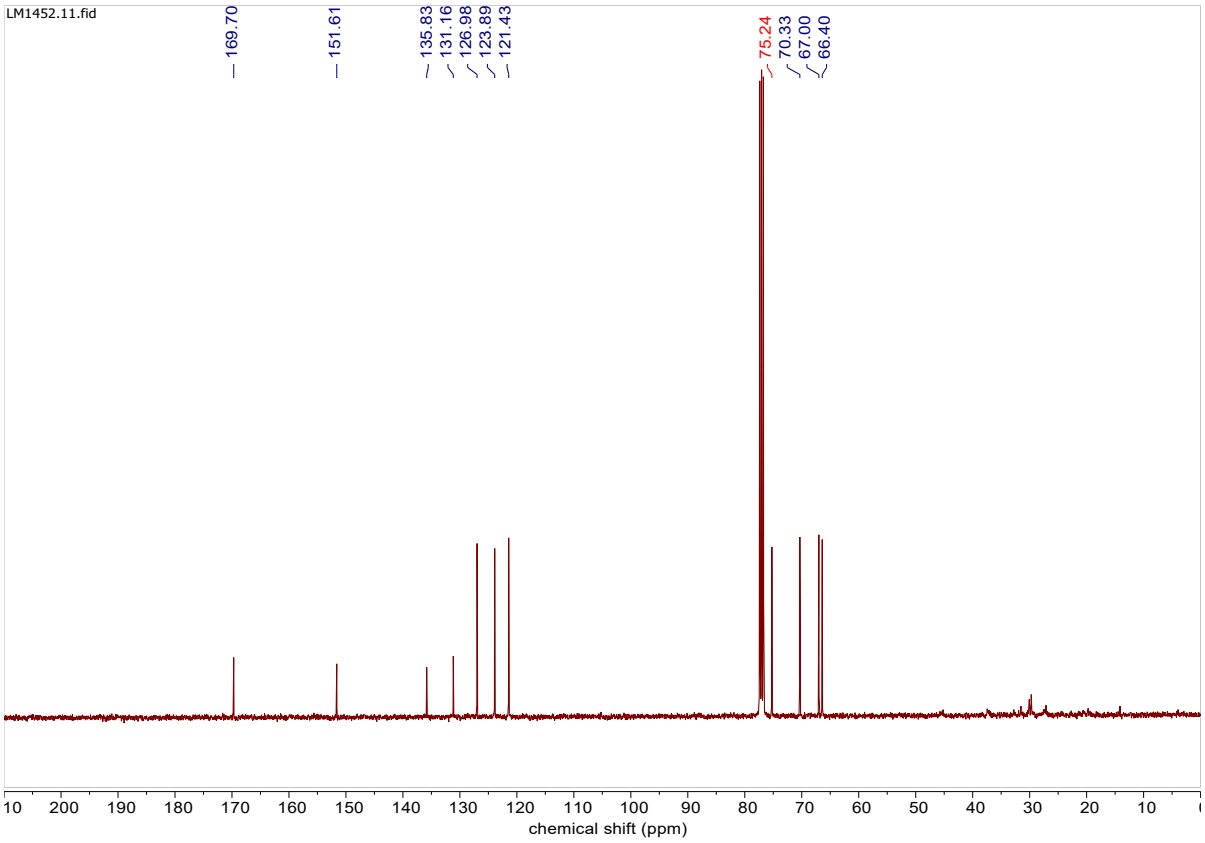
^{1}H and ^{13}C{1H} NMR spectra of compound **3d**.



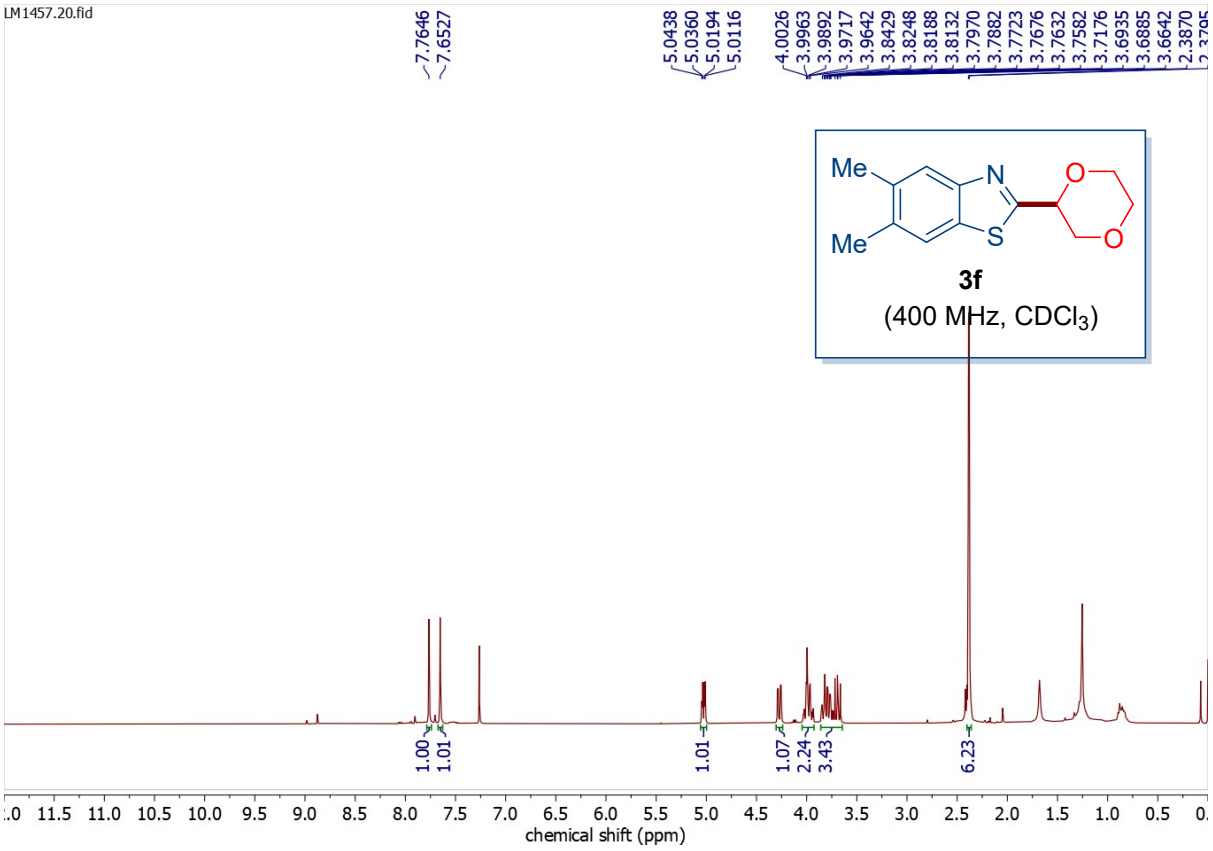


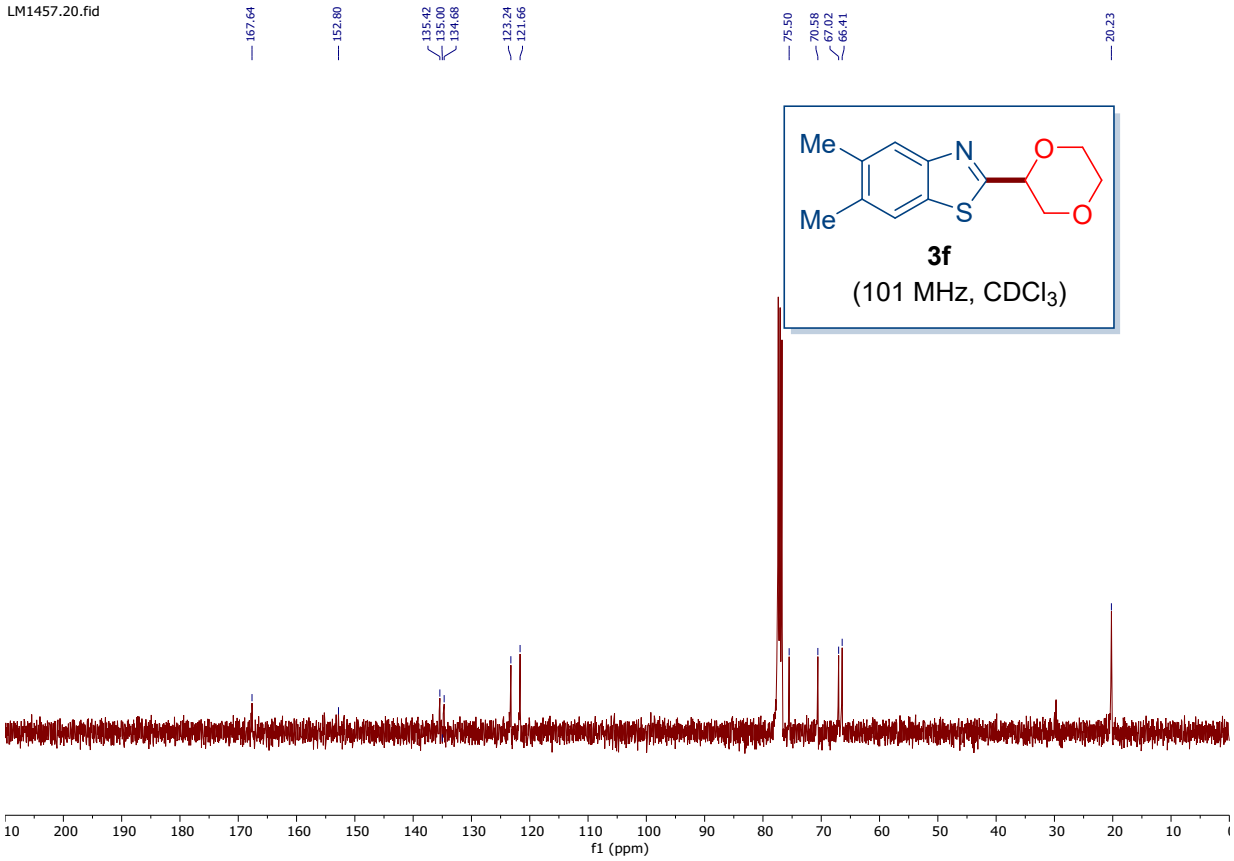
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3e**.



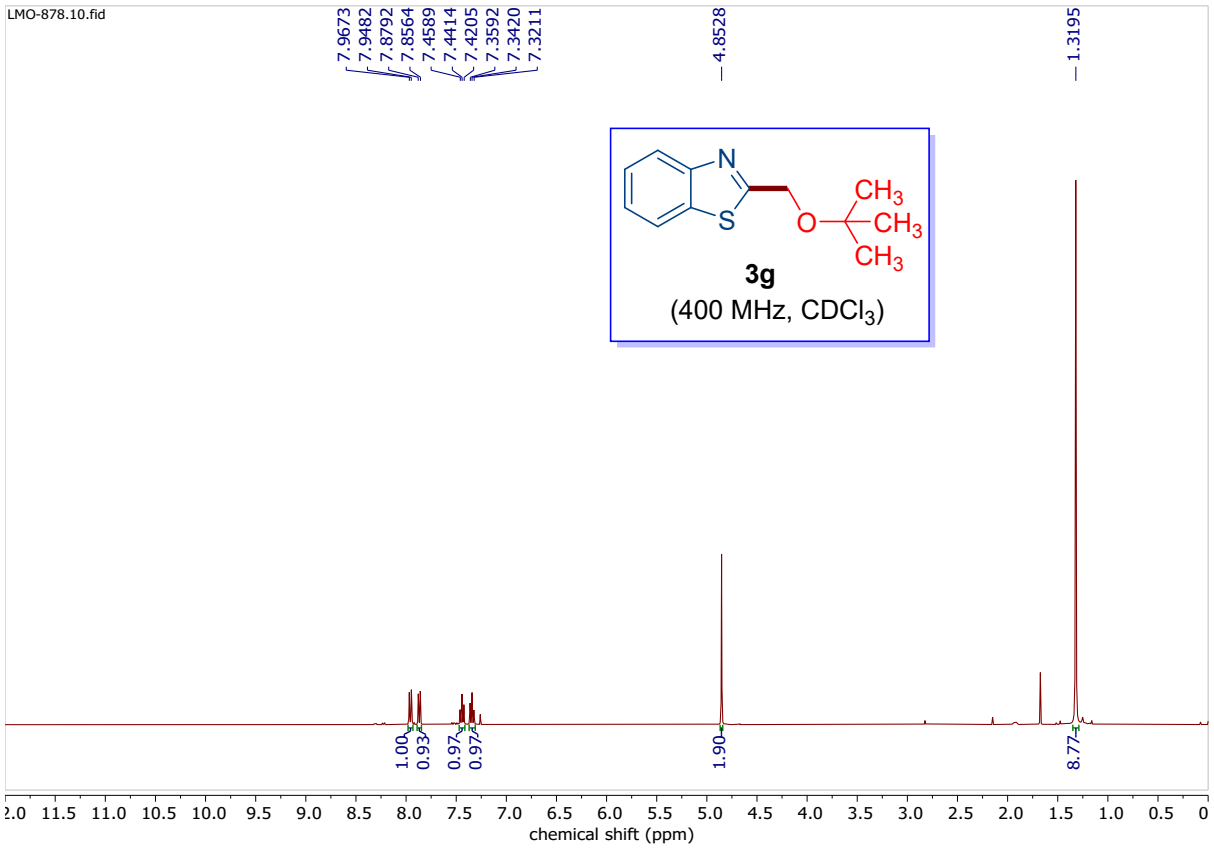


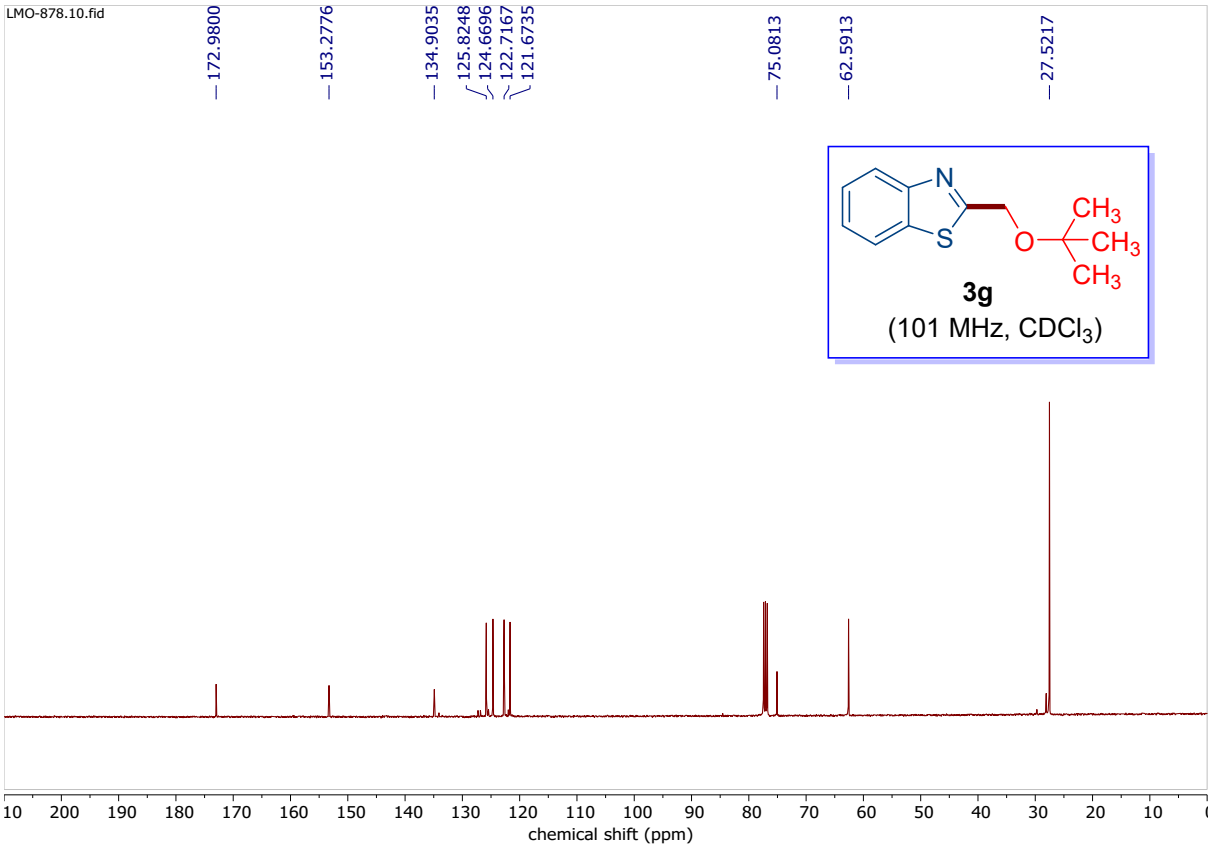
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3f**.



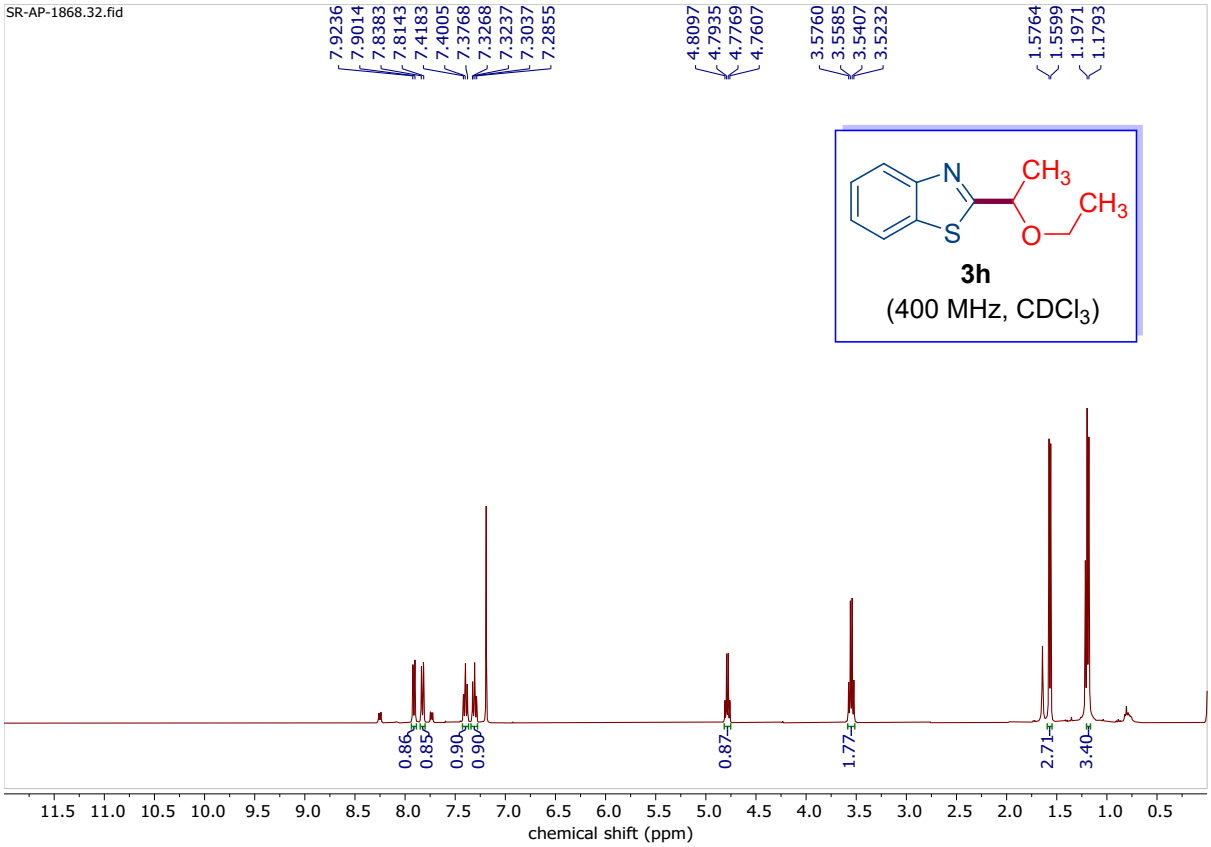


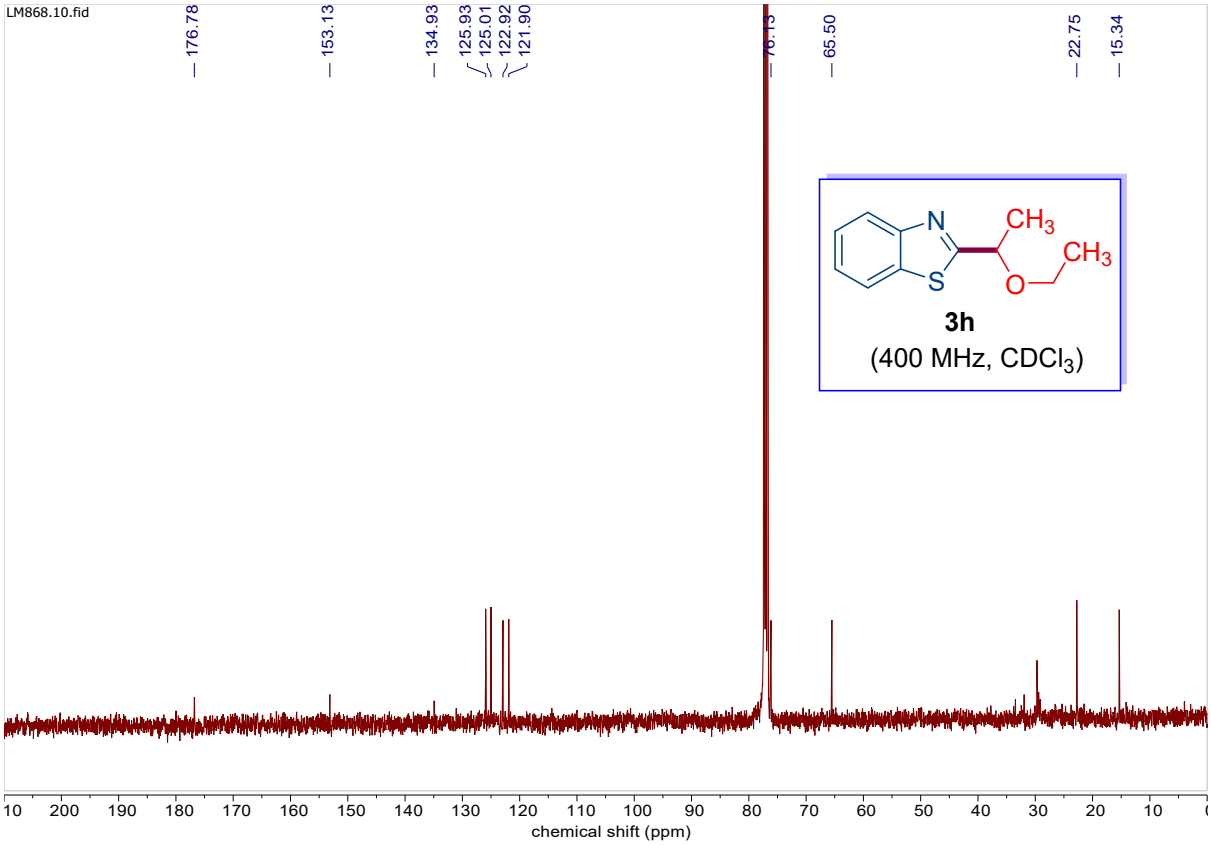
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3g**.



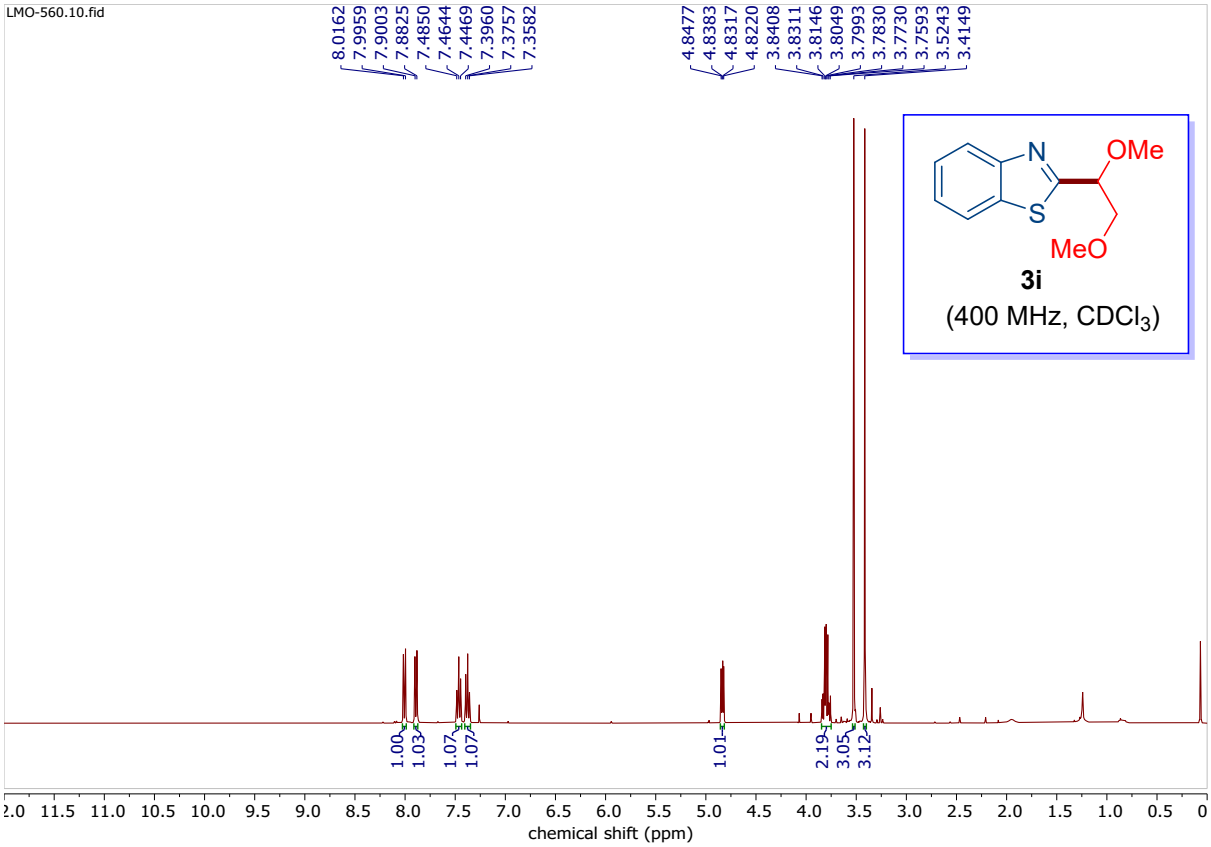


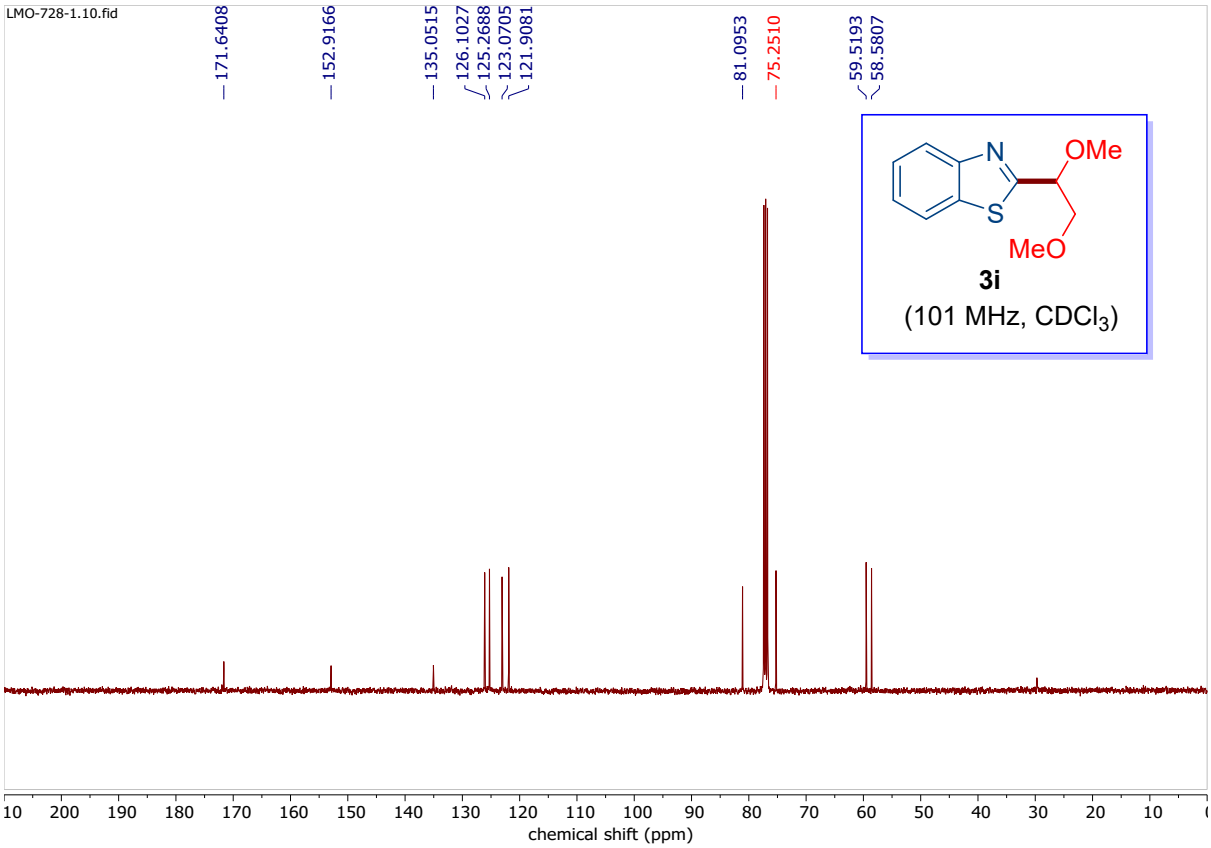
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3h**.



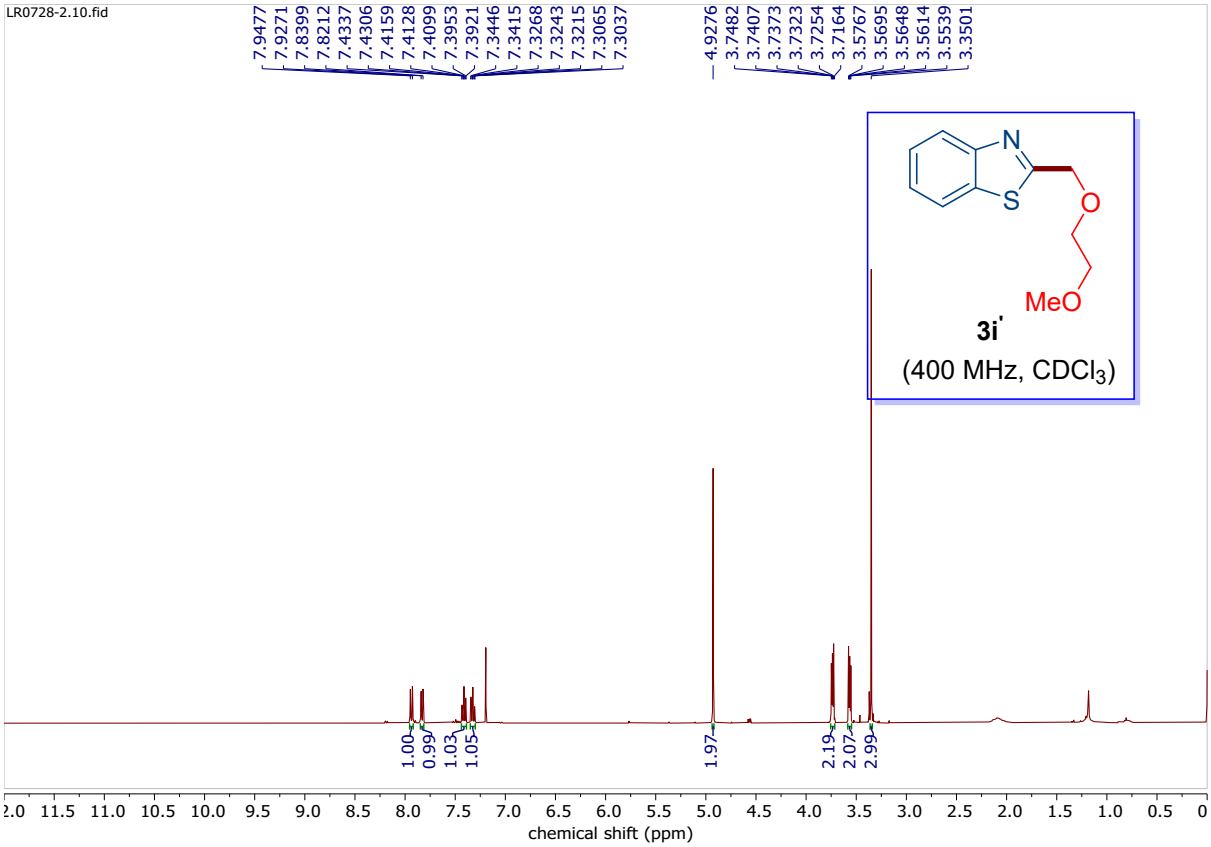


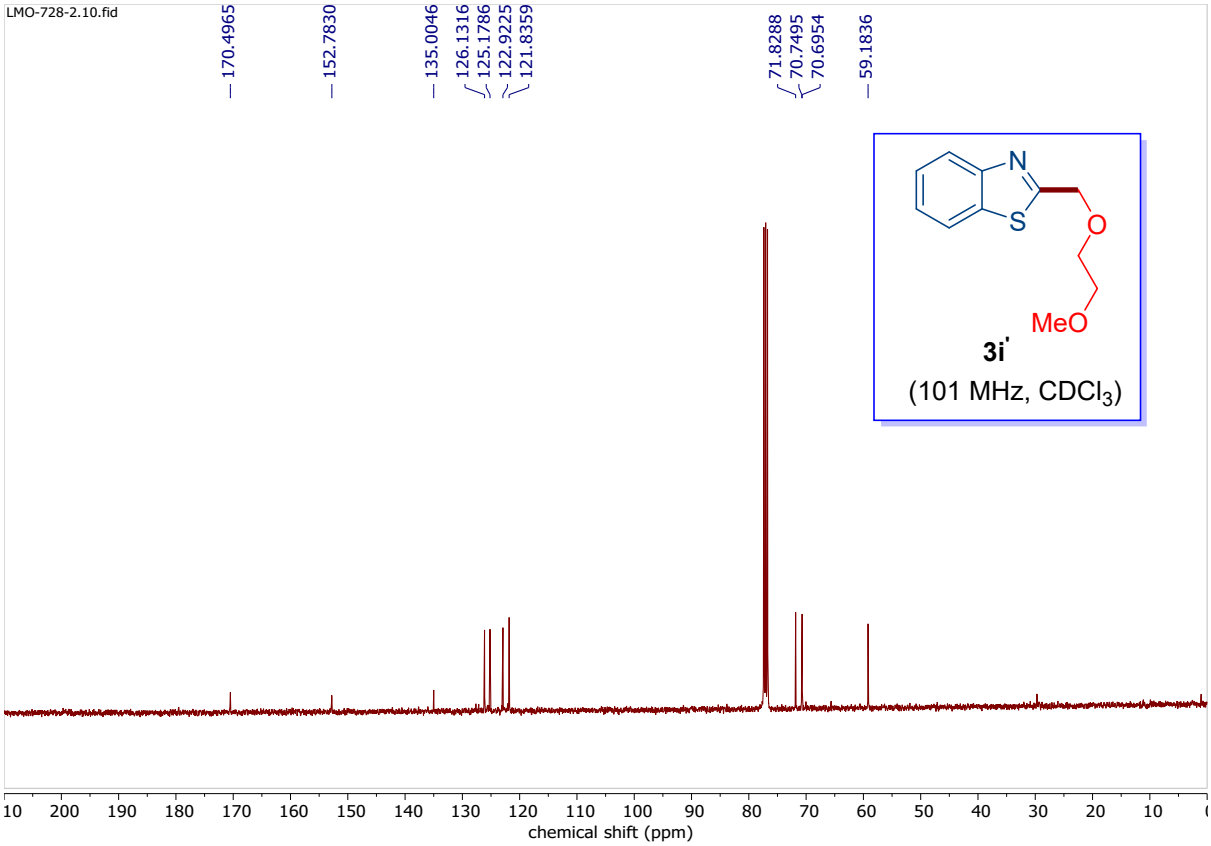
^{1}H and $^{13}C\{1H\}$ NMR spectra of compound **3i**.



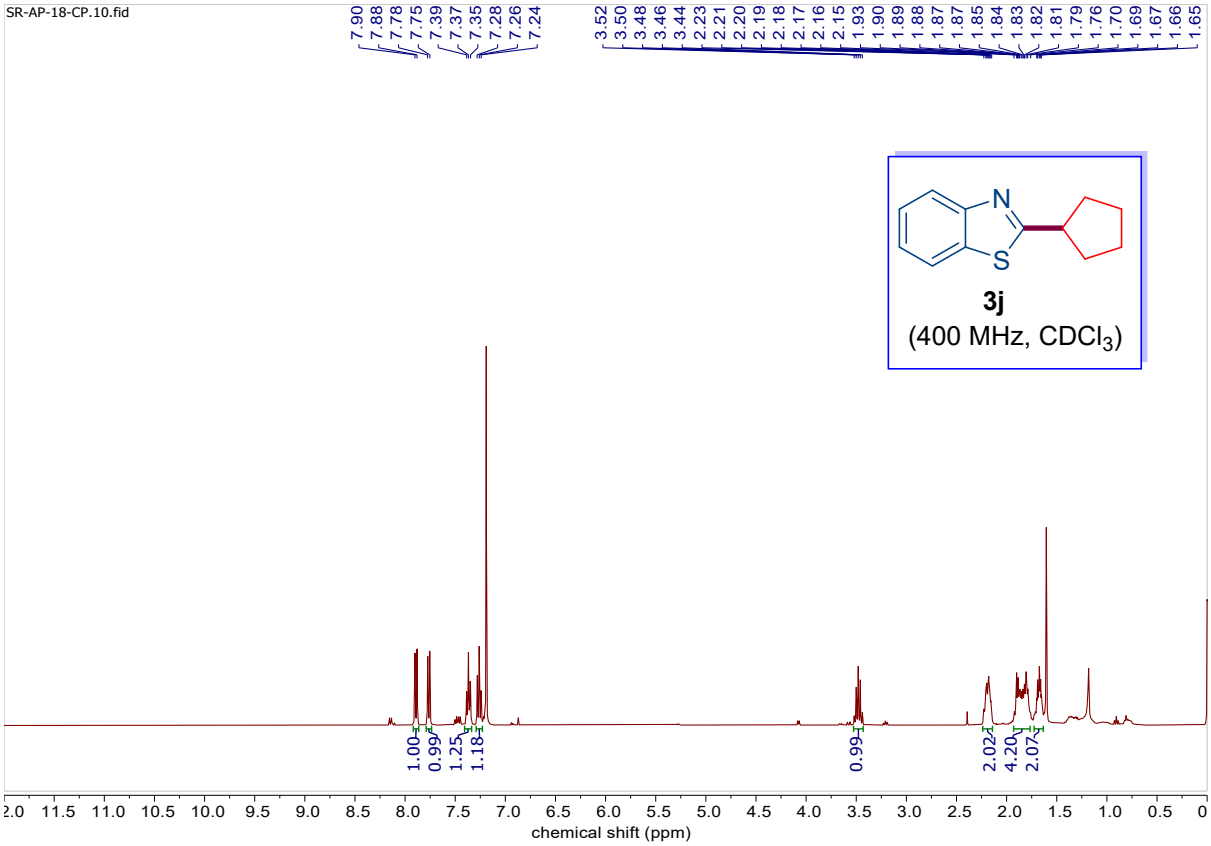


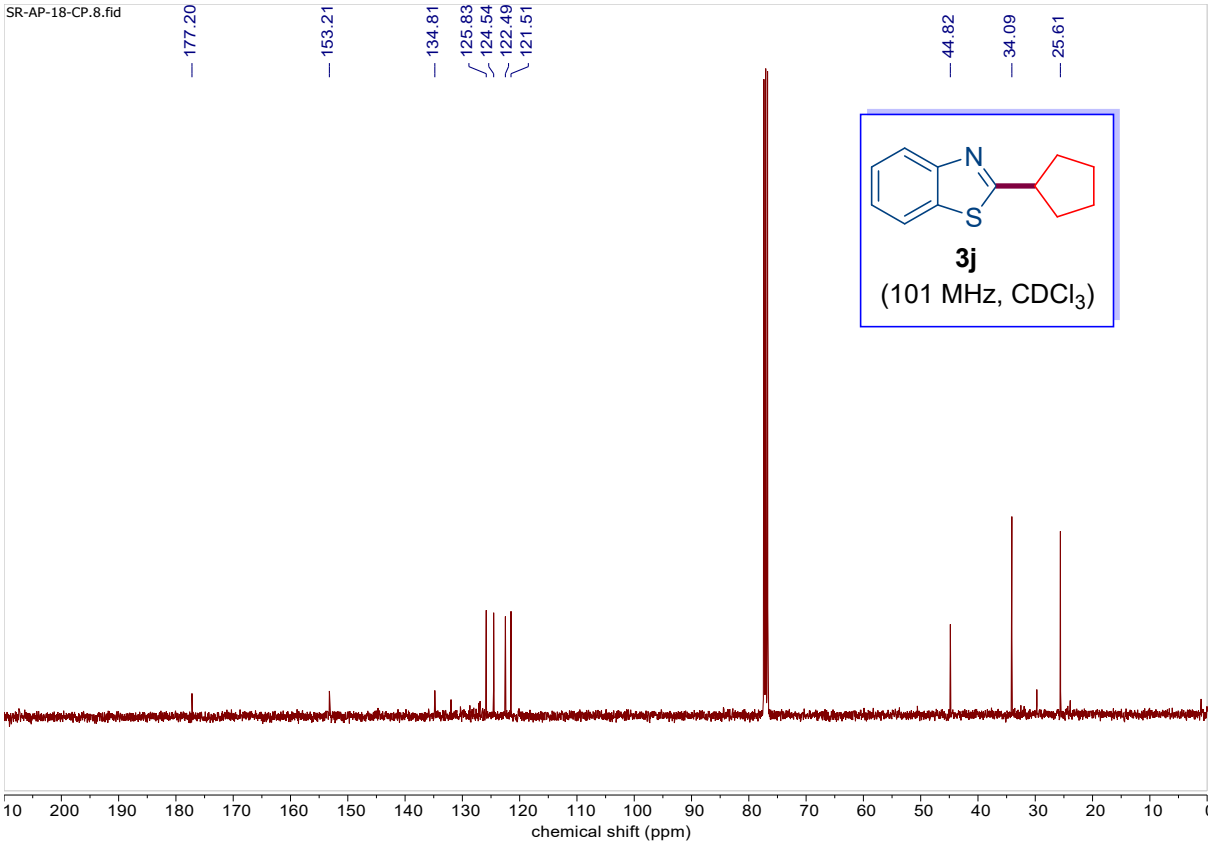
^{1}H and ^{13}C{1H} NMR spectra of compound **3i'**.



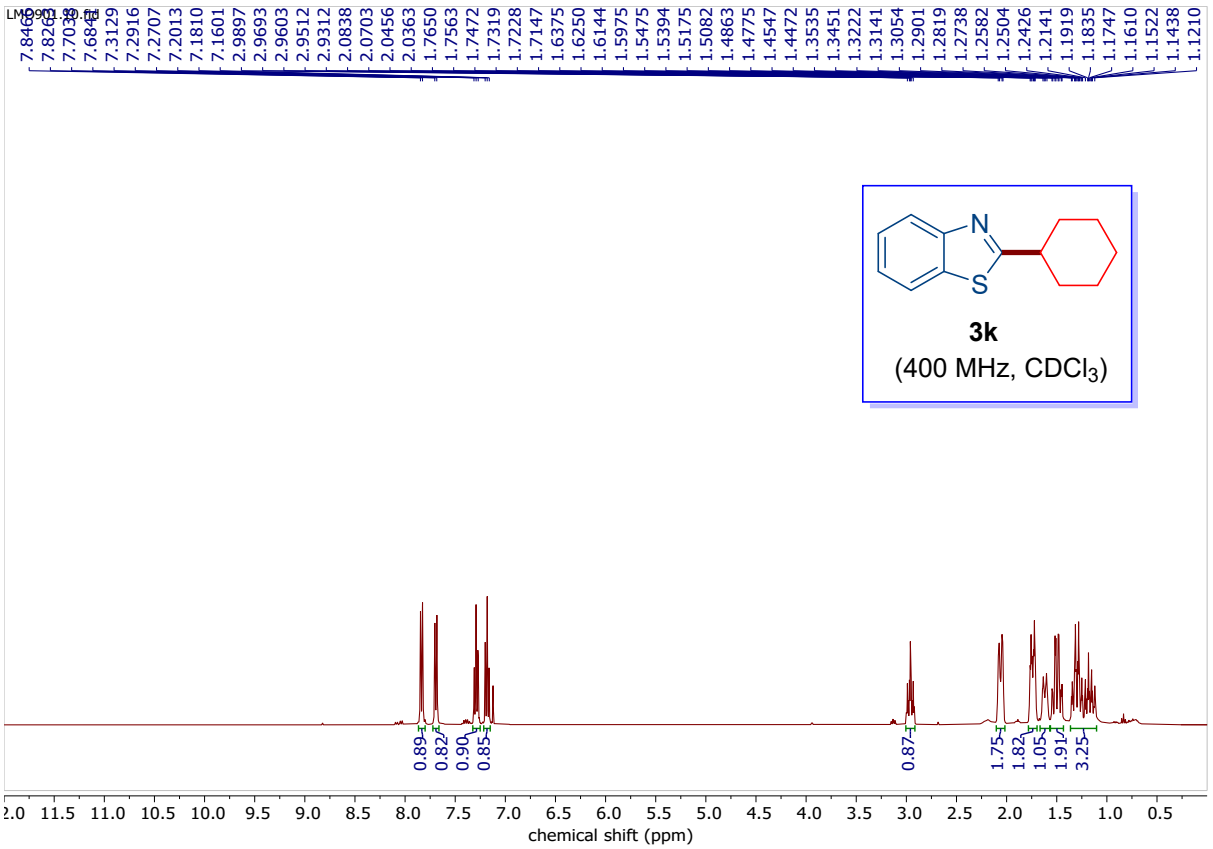


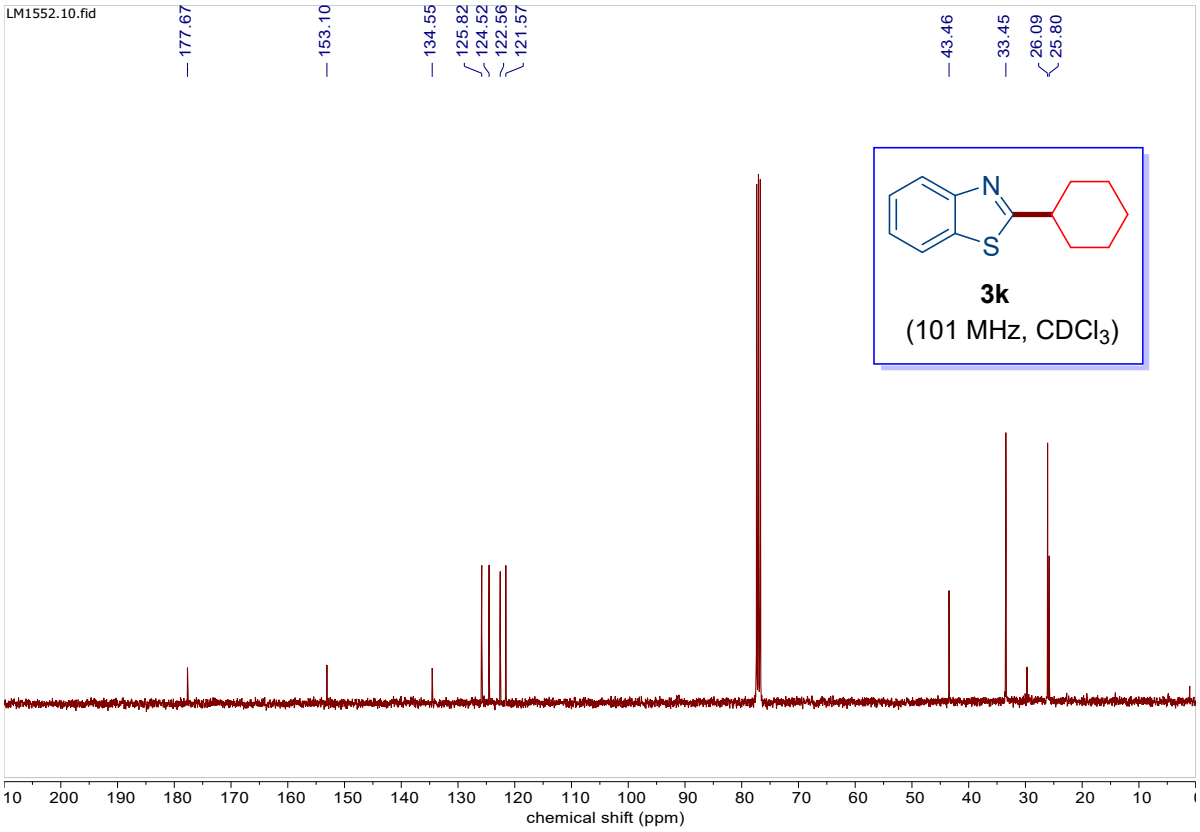
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3j**.



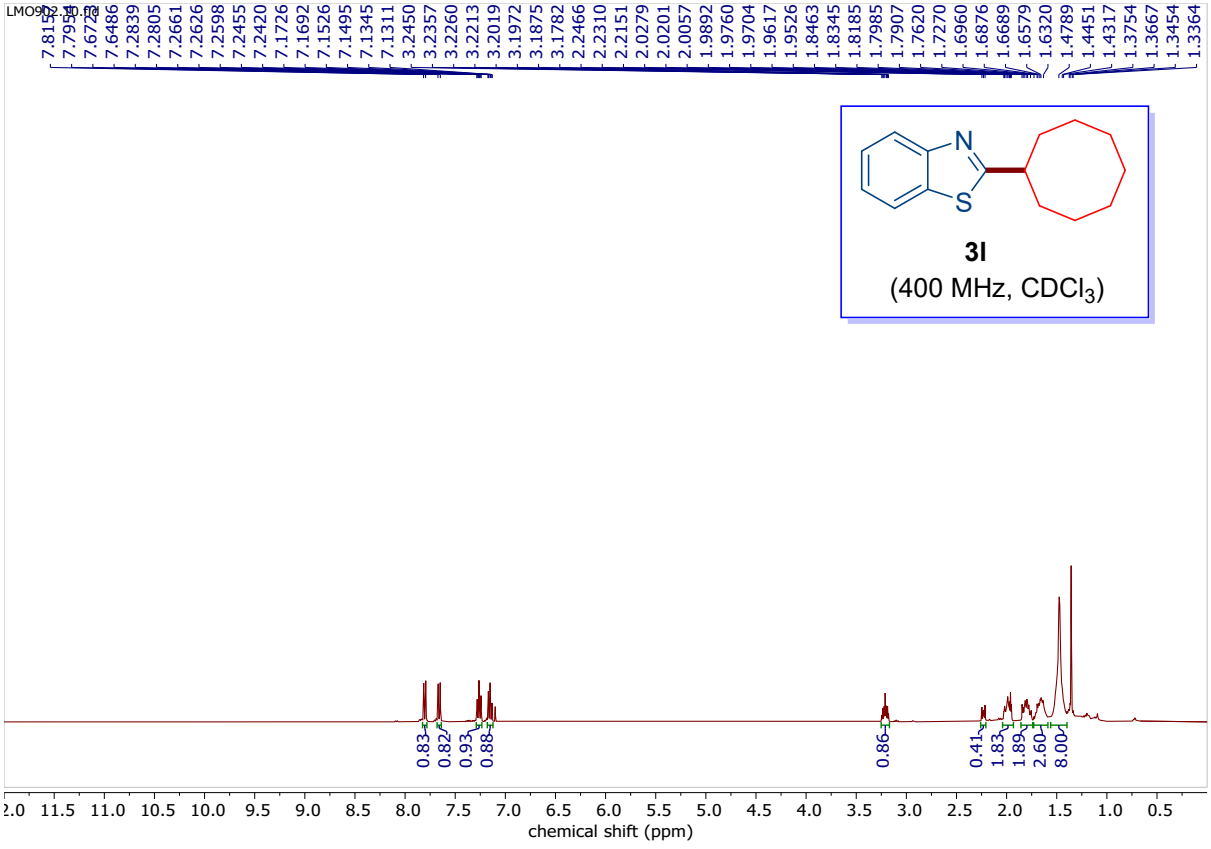


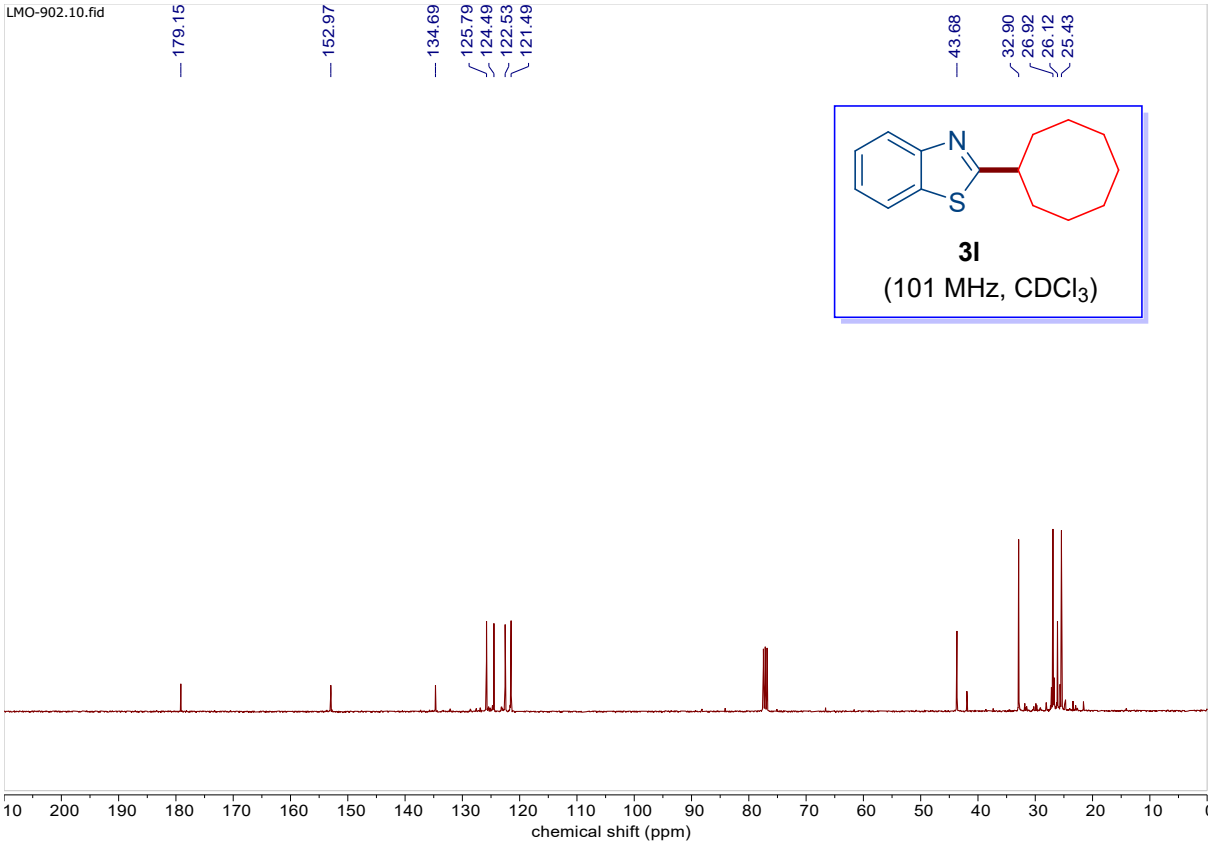
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3k**.



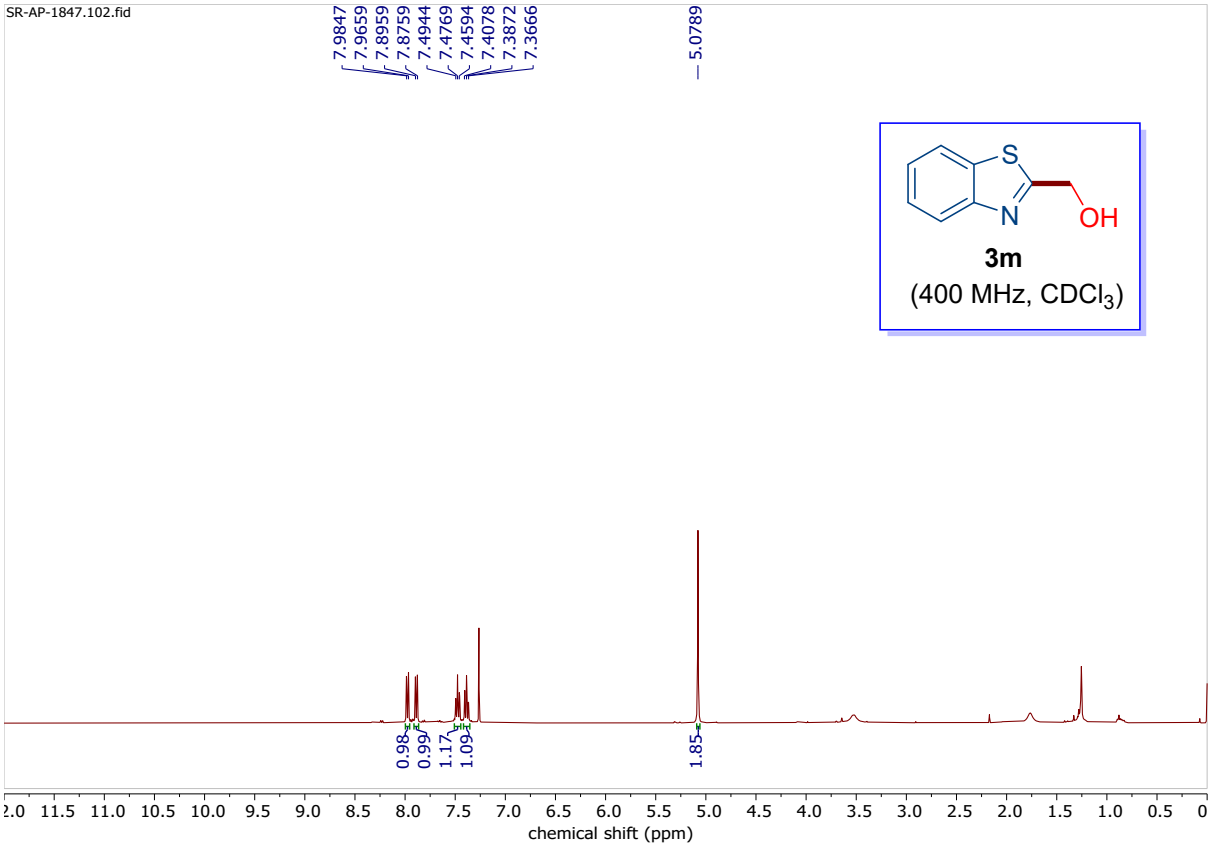


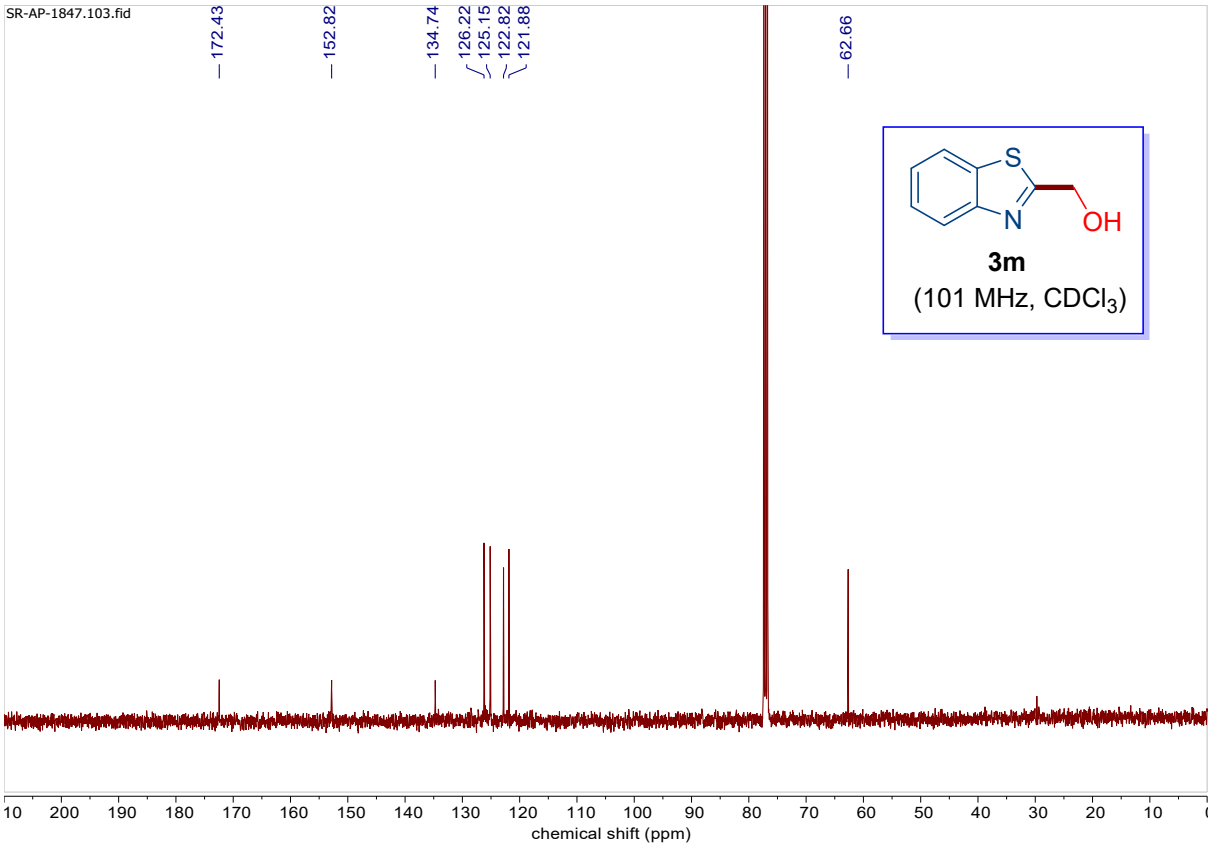
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3l.**



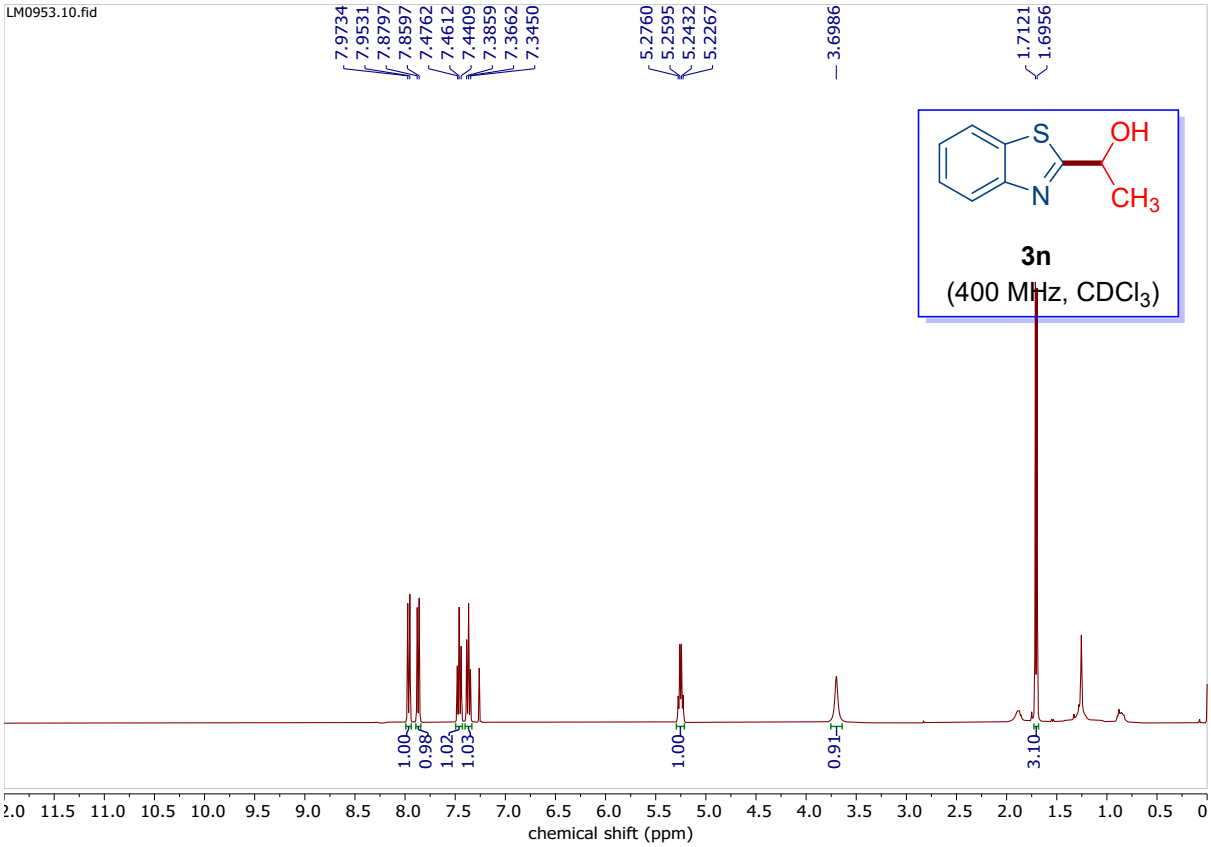


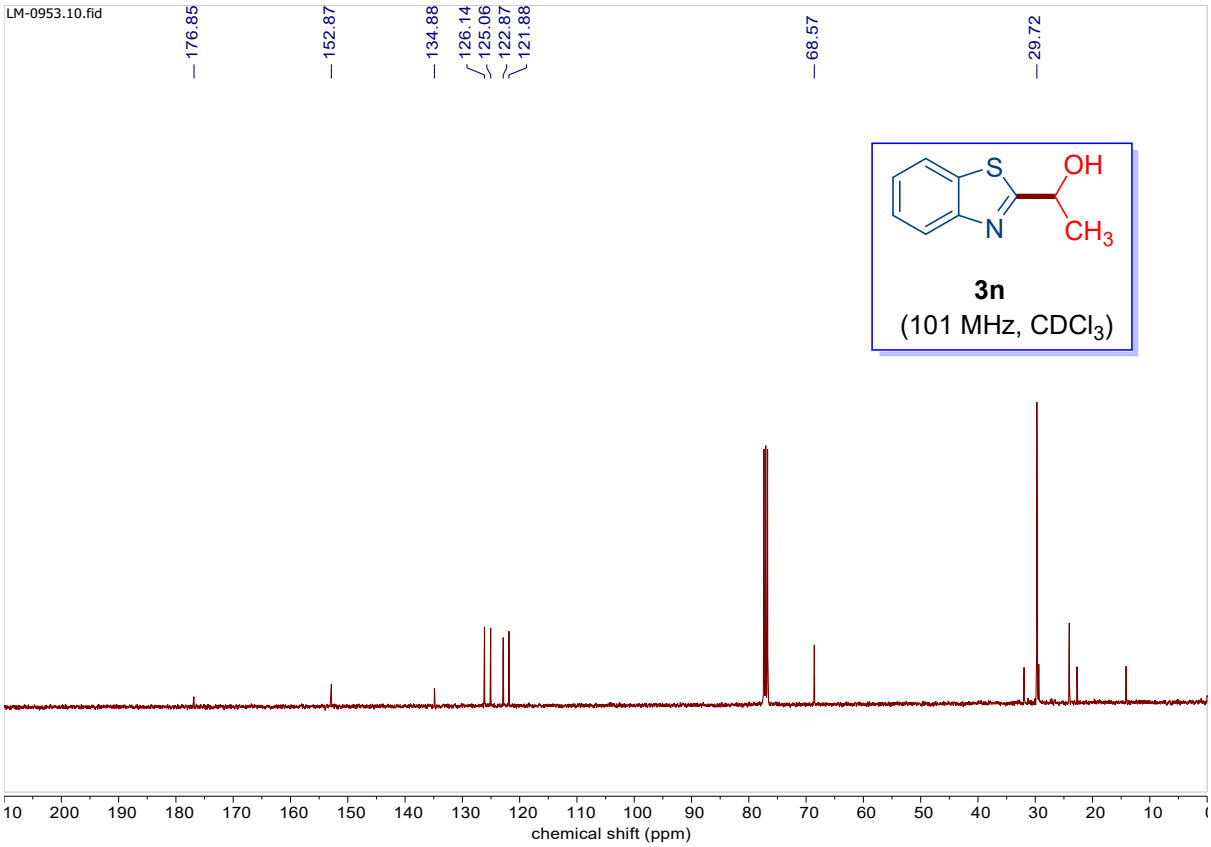
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3m.**



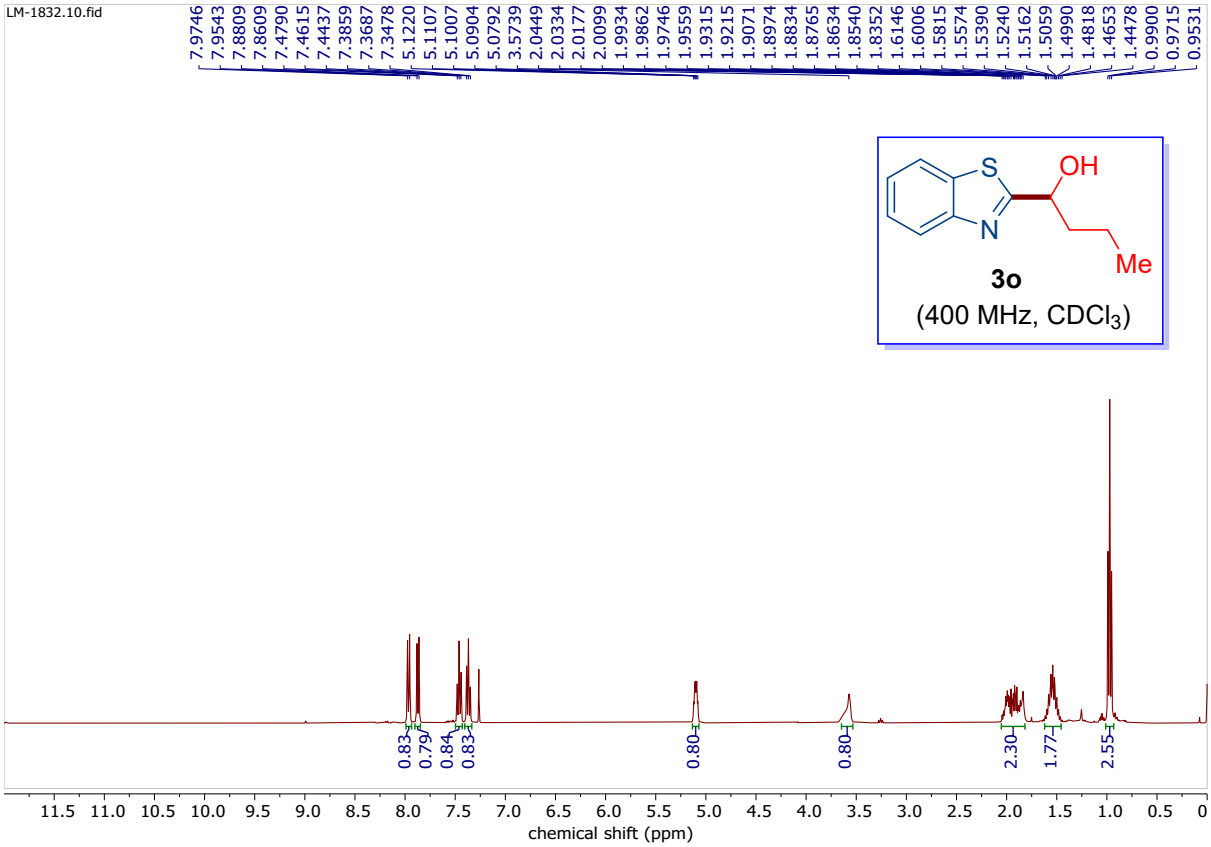


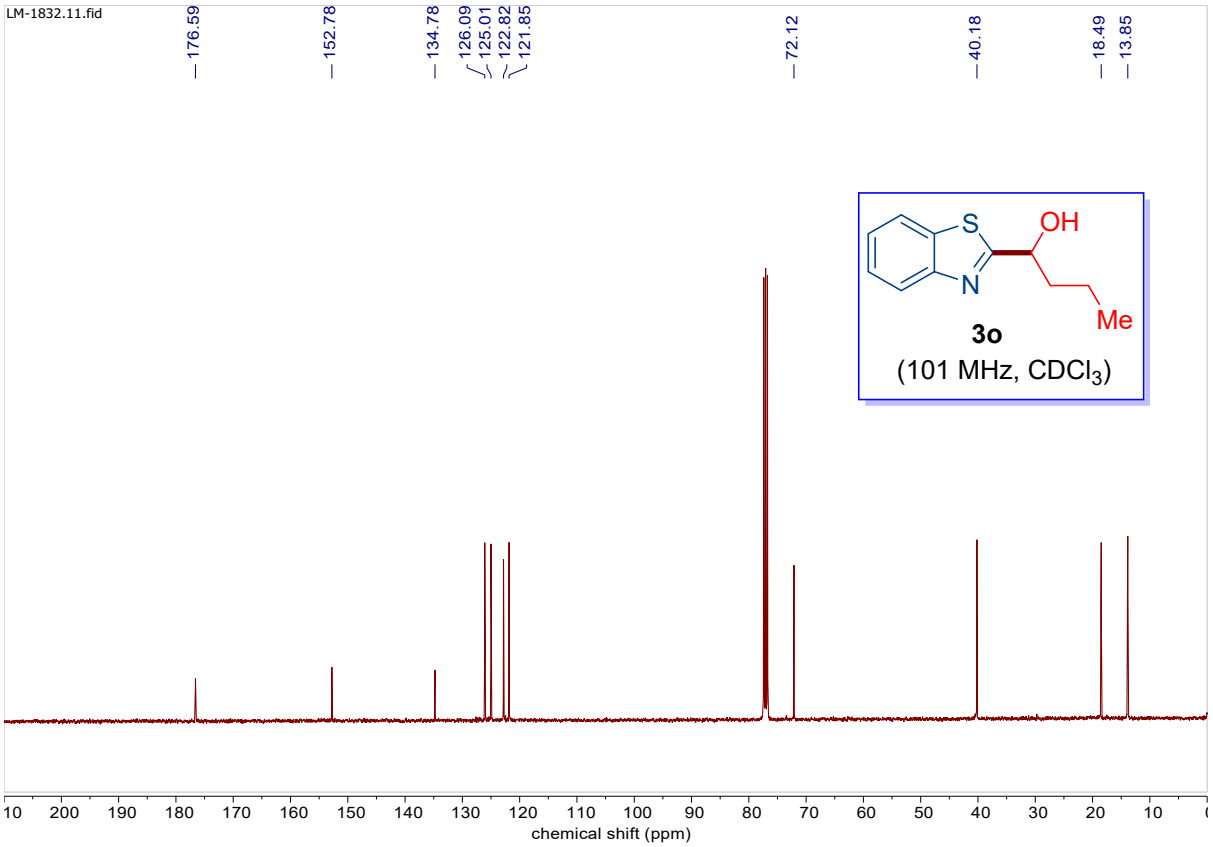
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3n.**



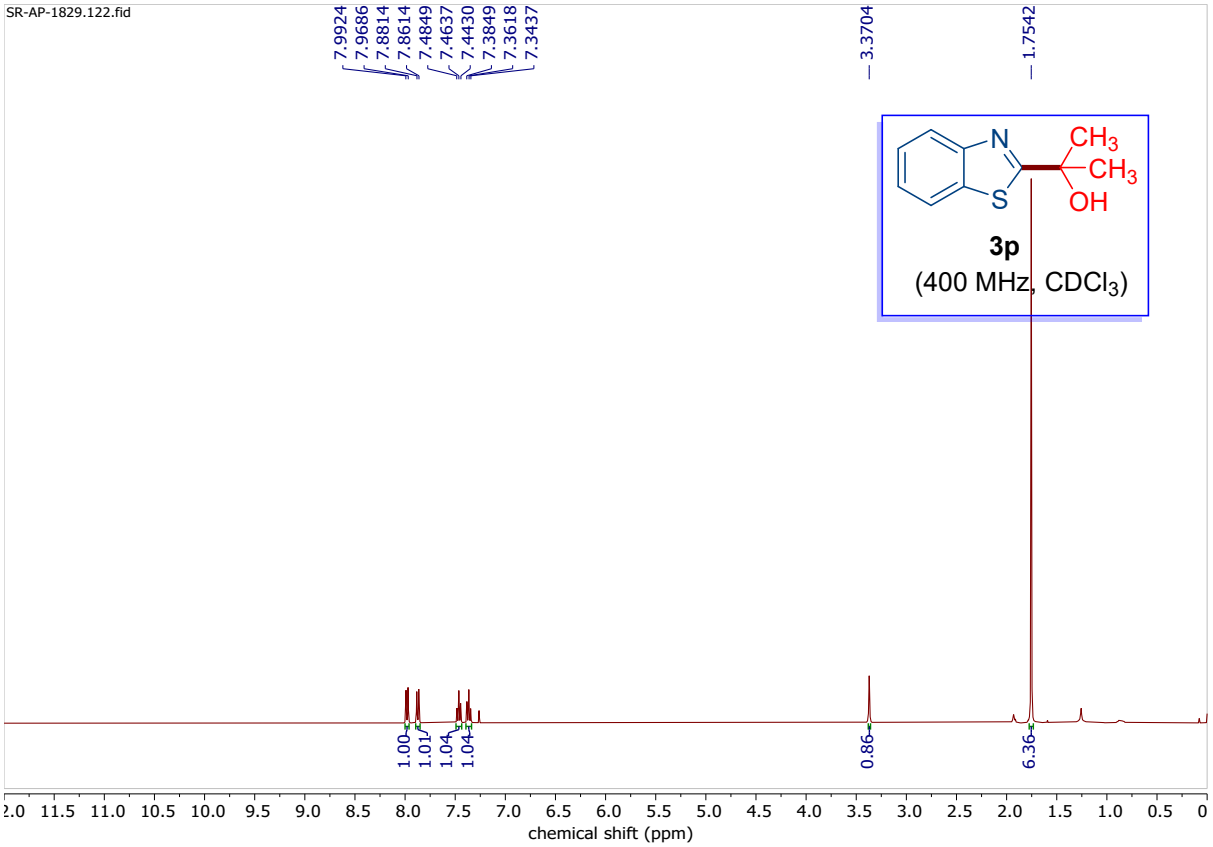


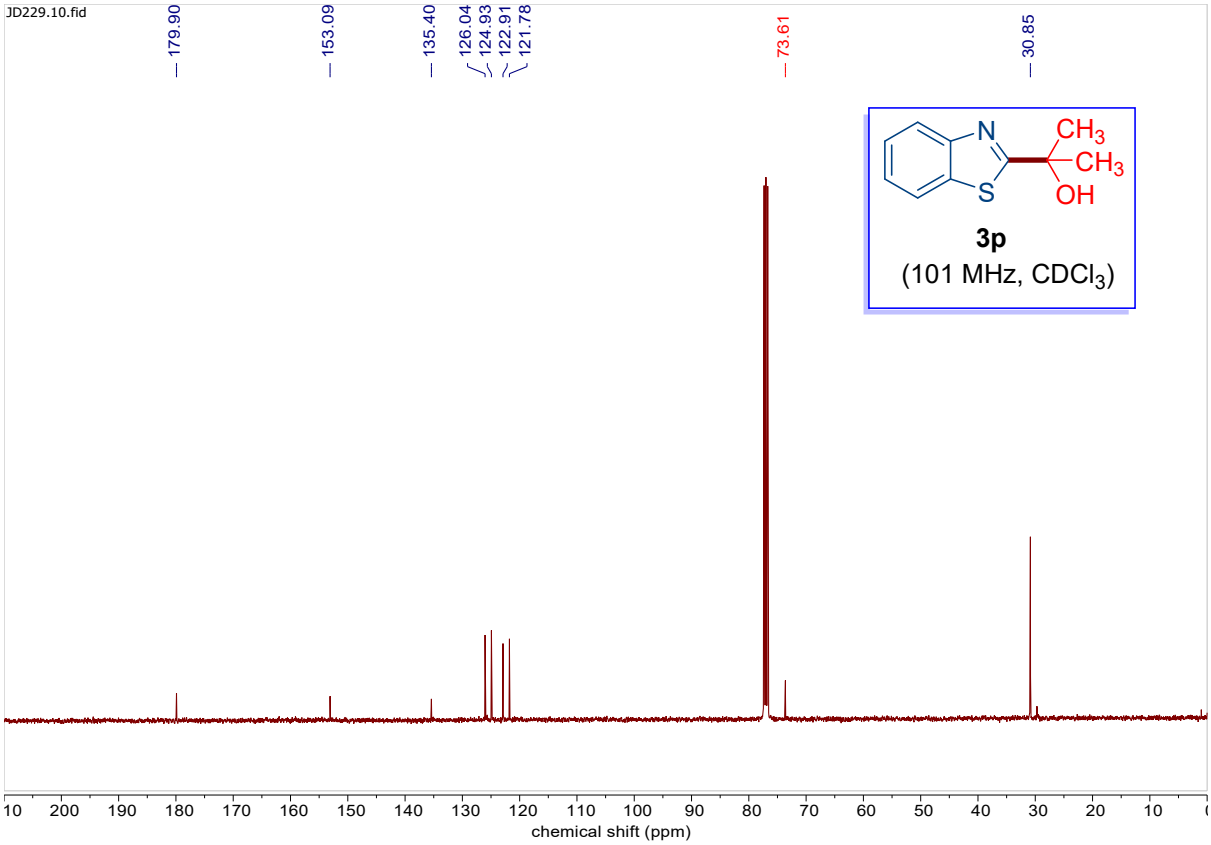
^{1}H and $^{13}C\{1H\}$ NMR spectra of compound **3o**.



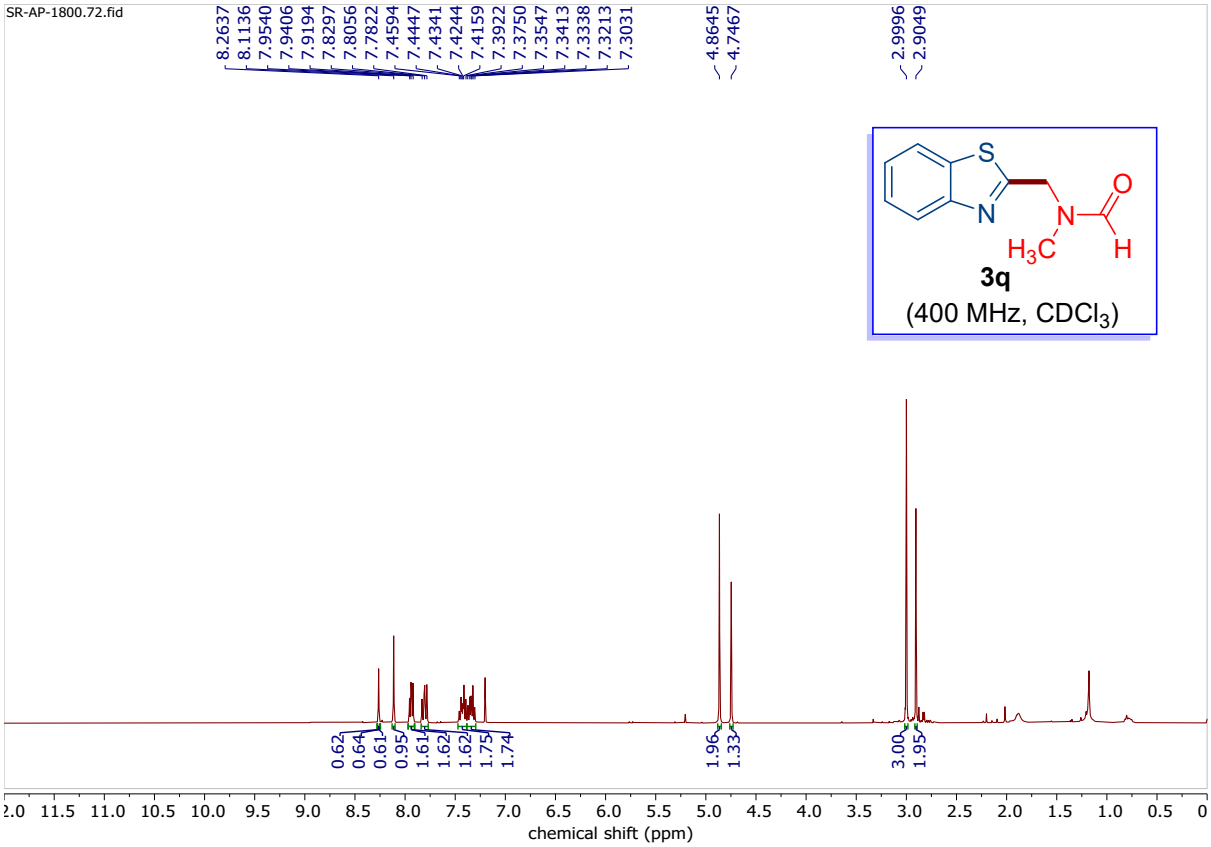


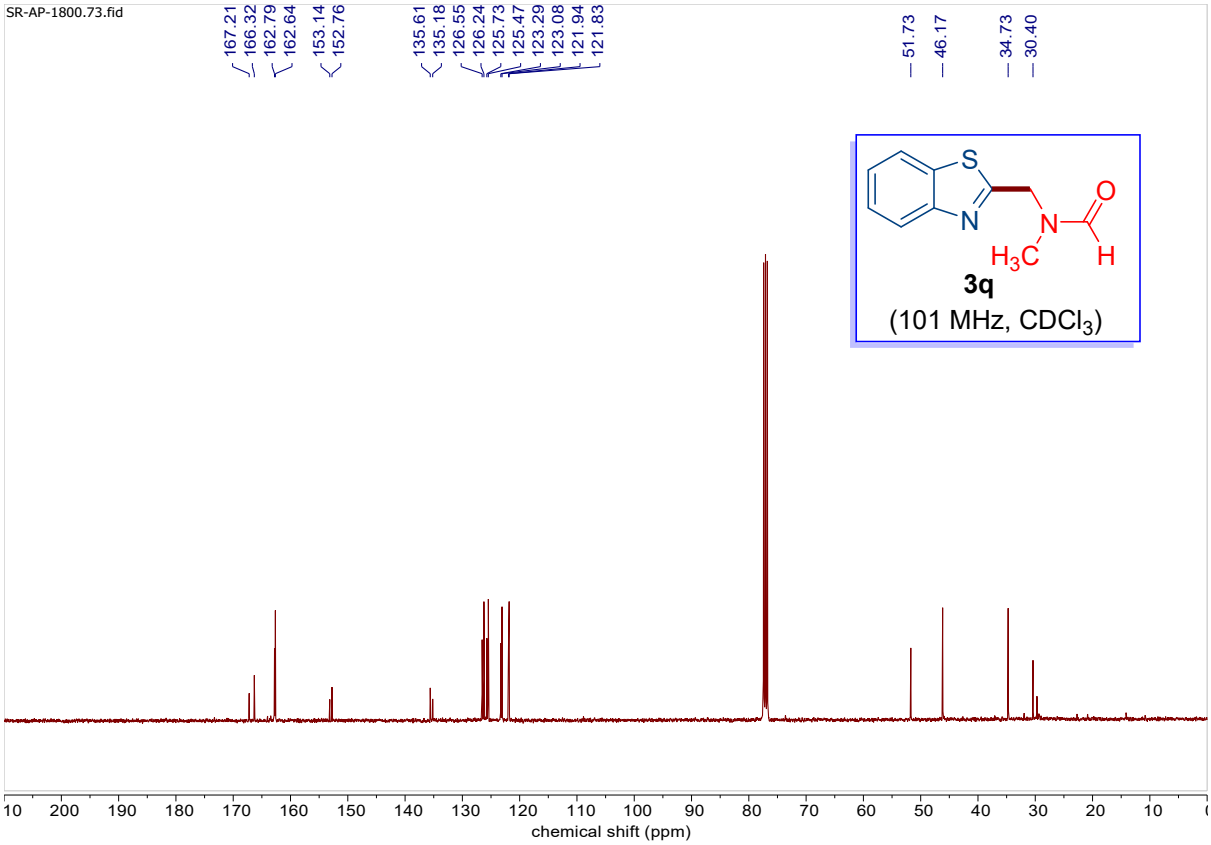
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3p.**



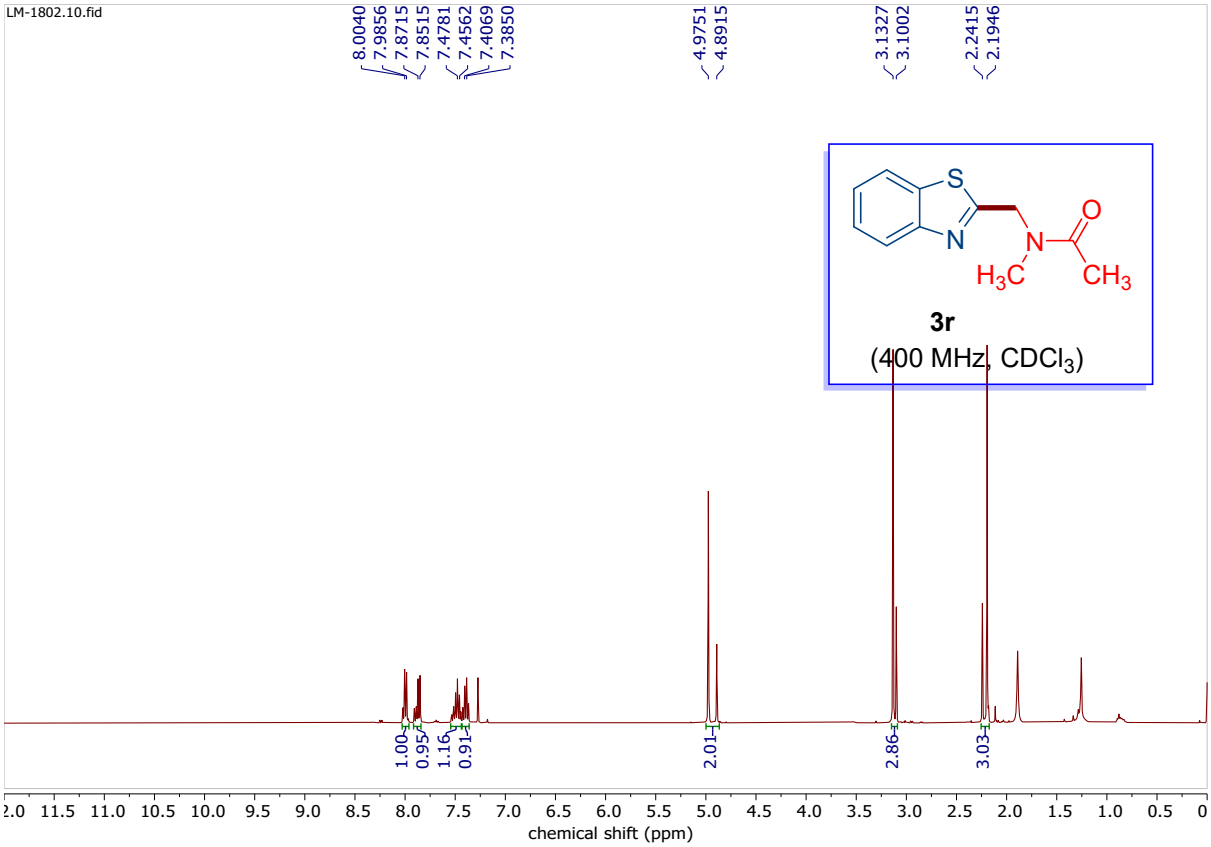


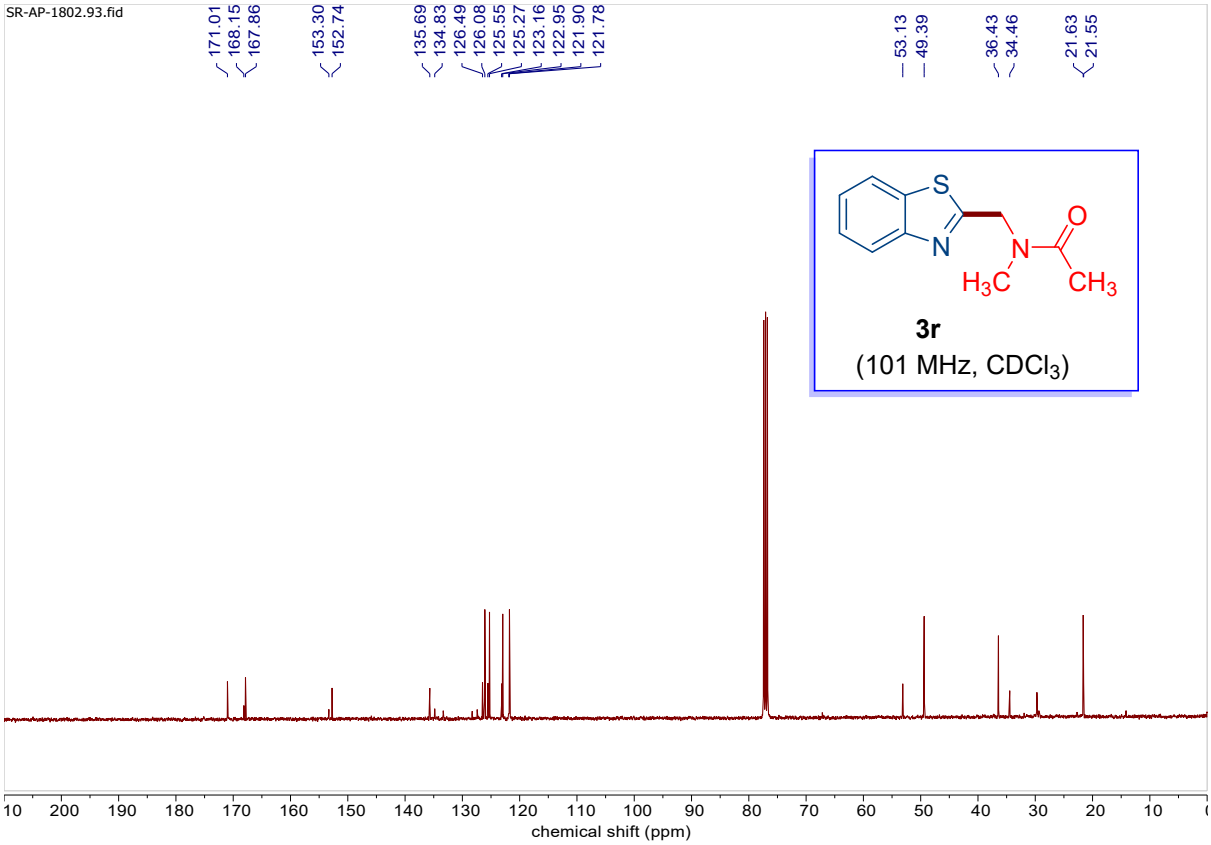
^{1}H and ^{13}C{^{1}H} NMR spectra of compound **3q**.



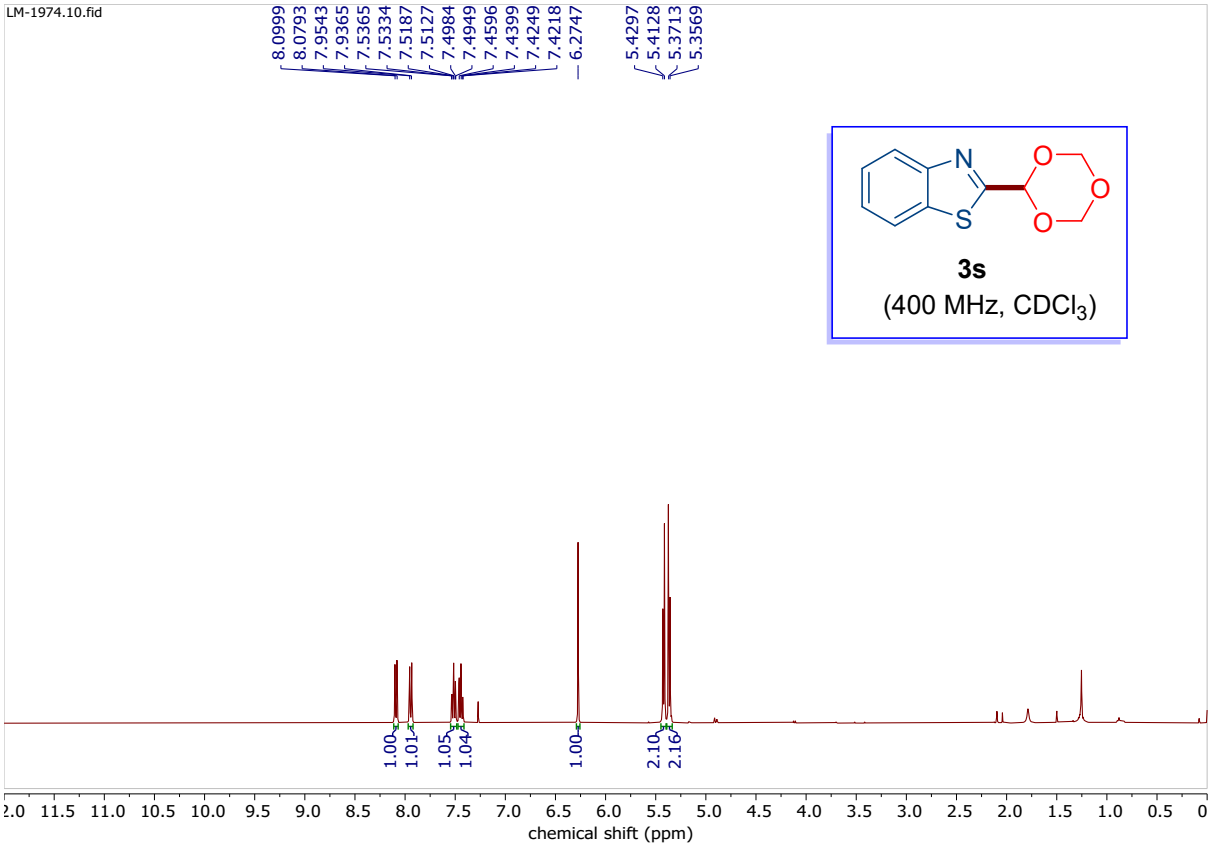


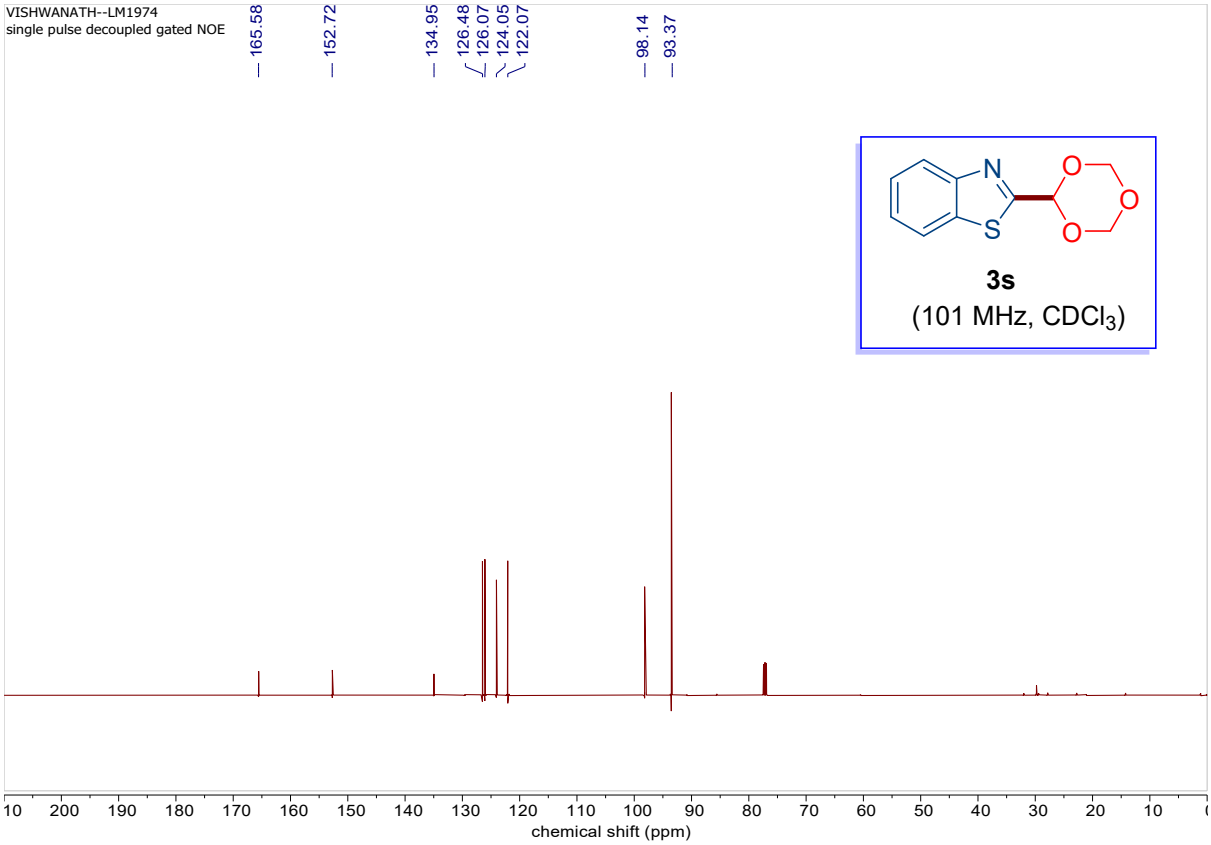
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3r.**



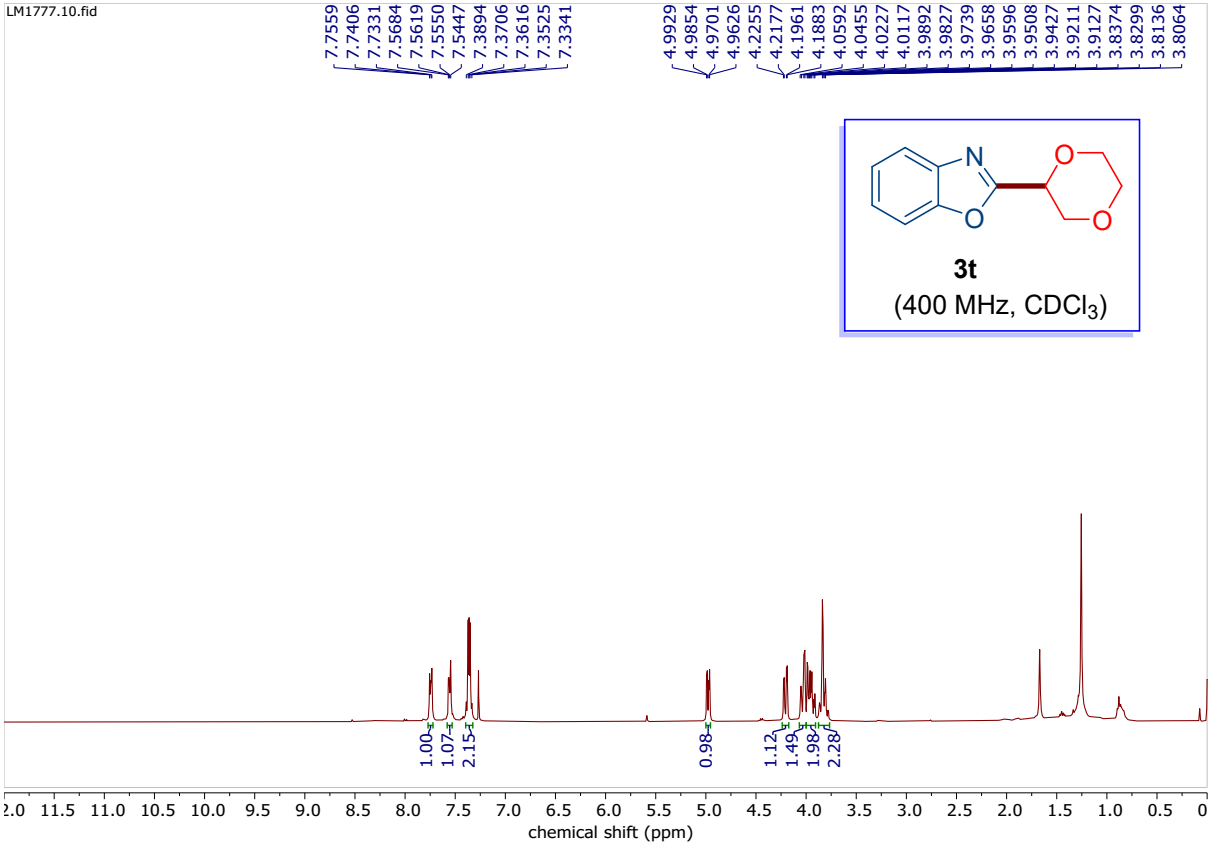


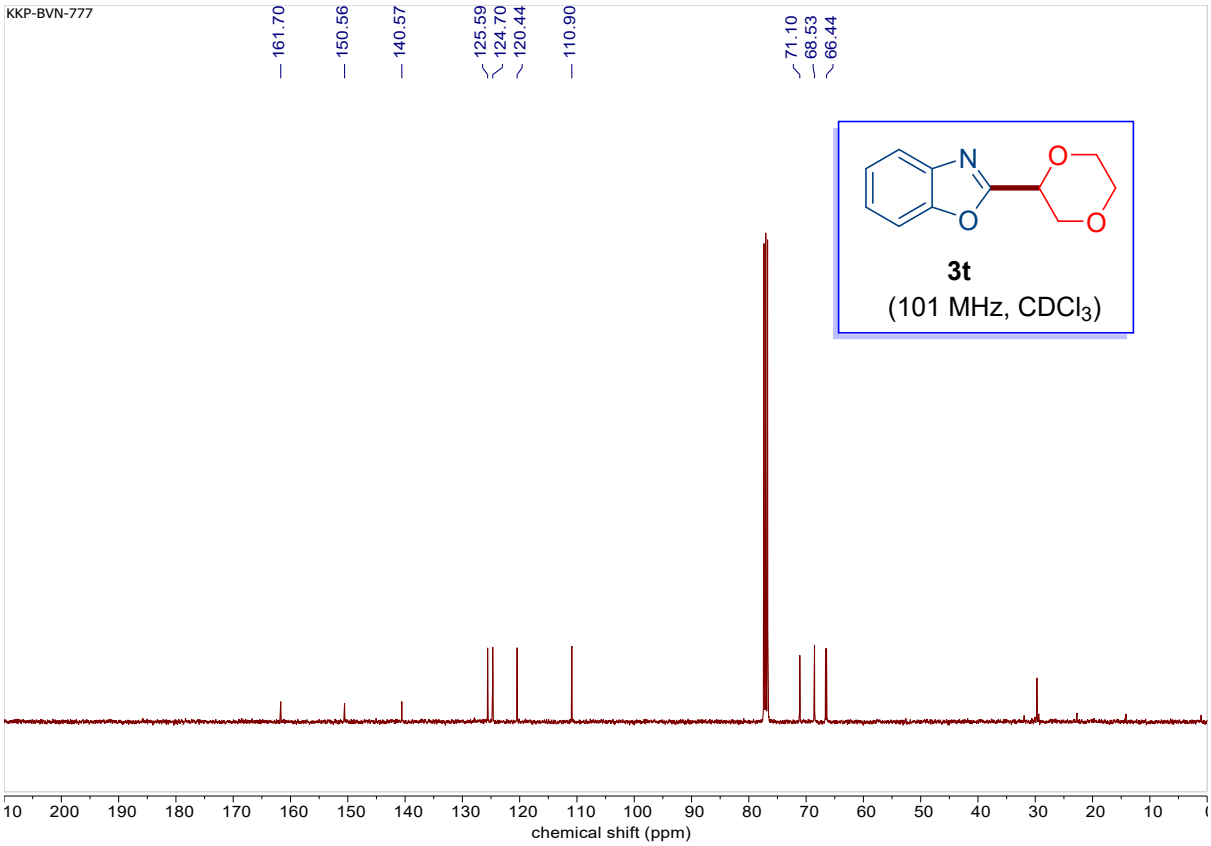
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3s**.



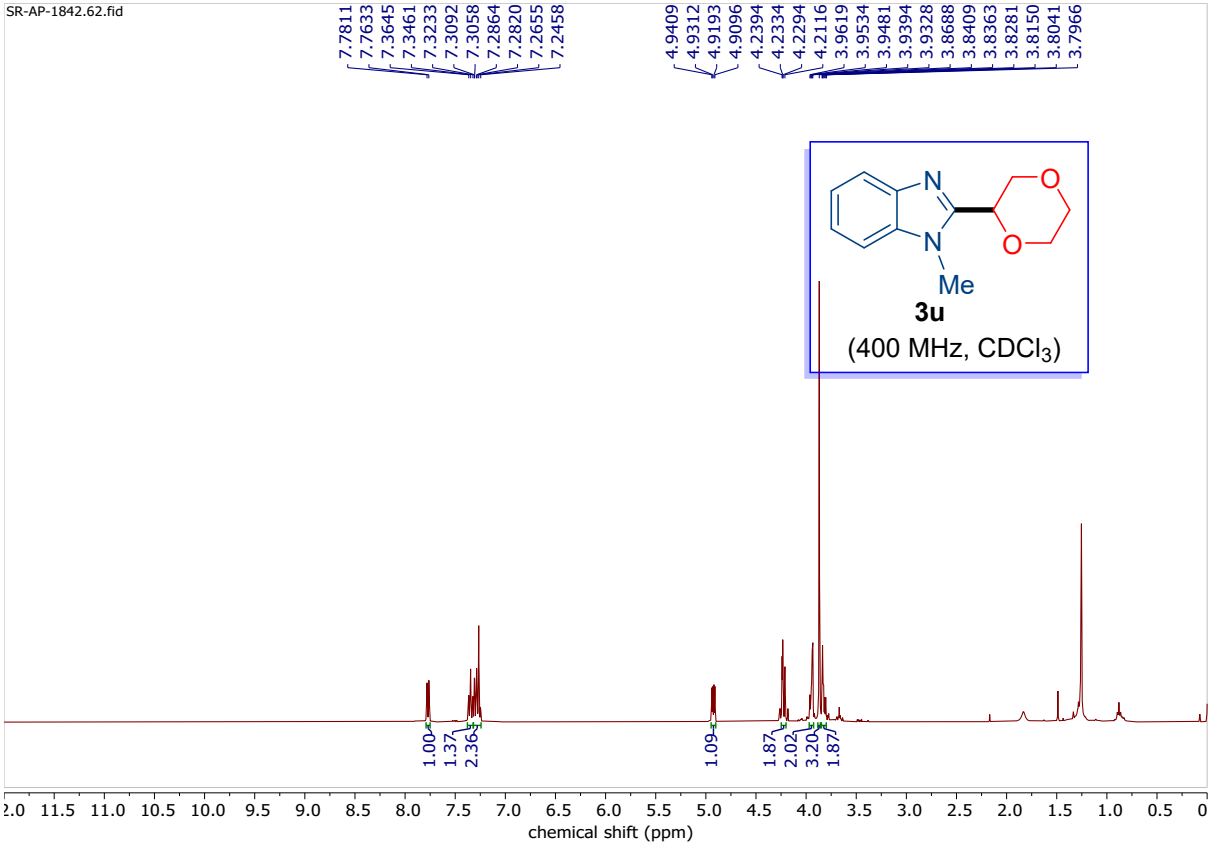

VISHWANATH--LM1974
single pulse decoupled gated NOE
165.58
152.72
134.95
126.48
126.07
124.05
122.07
98.14
93.37
N
S
O
O
O
3s
(101 MHz, CDCl3)
chemical shift (ppm)

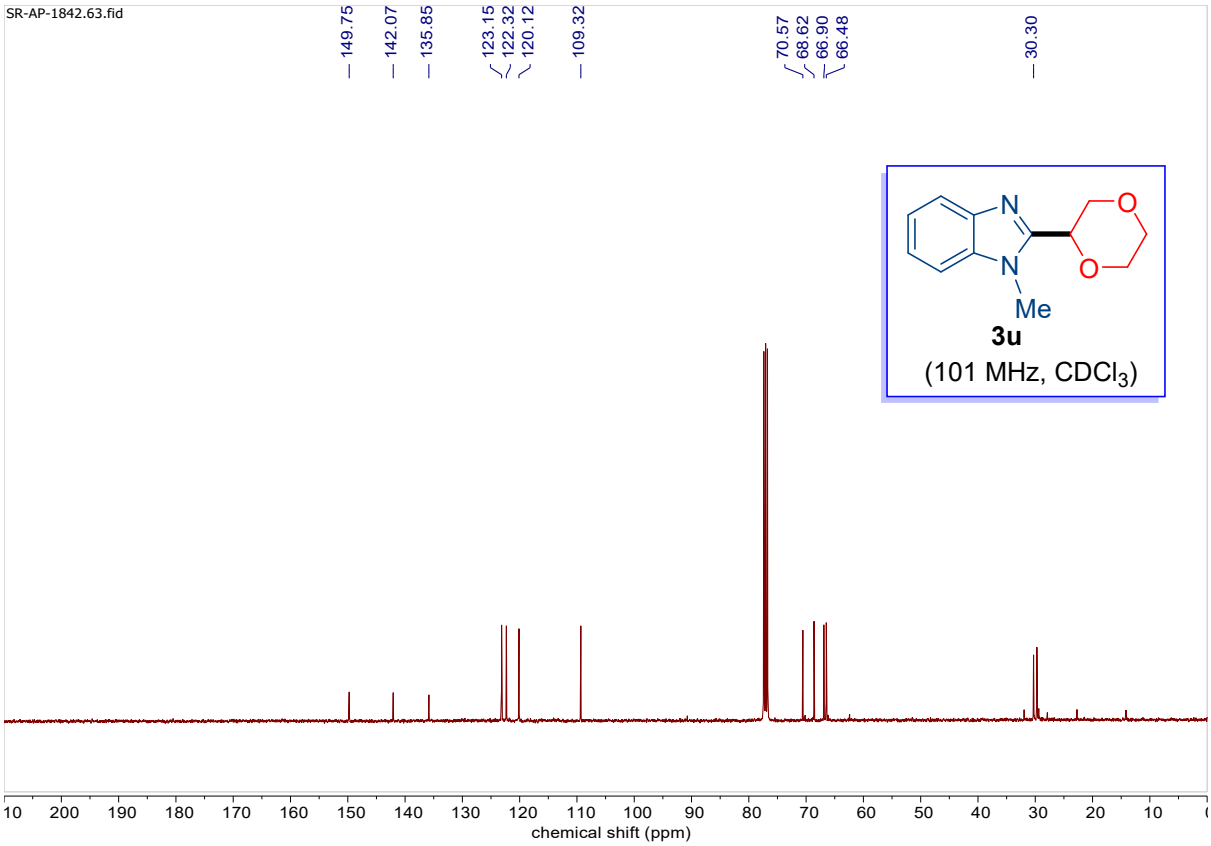
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3t.**



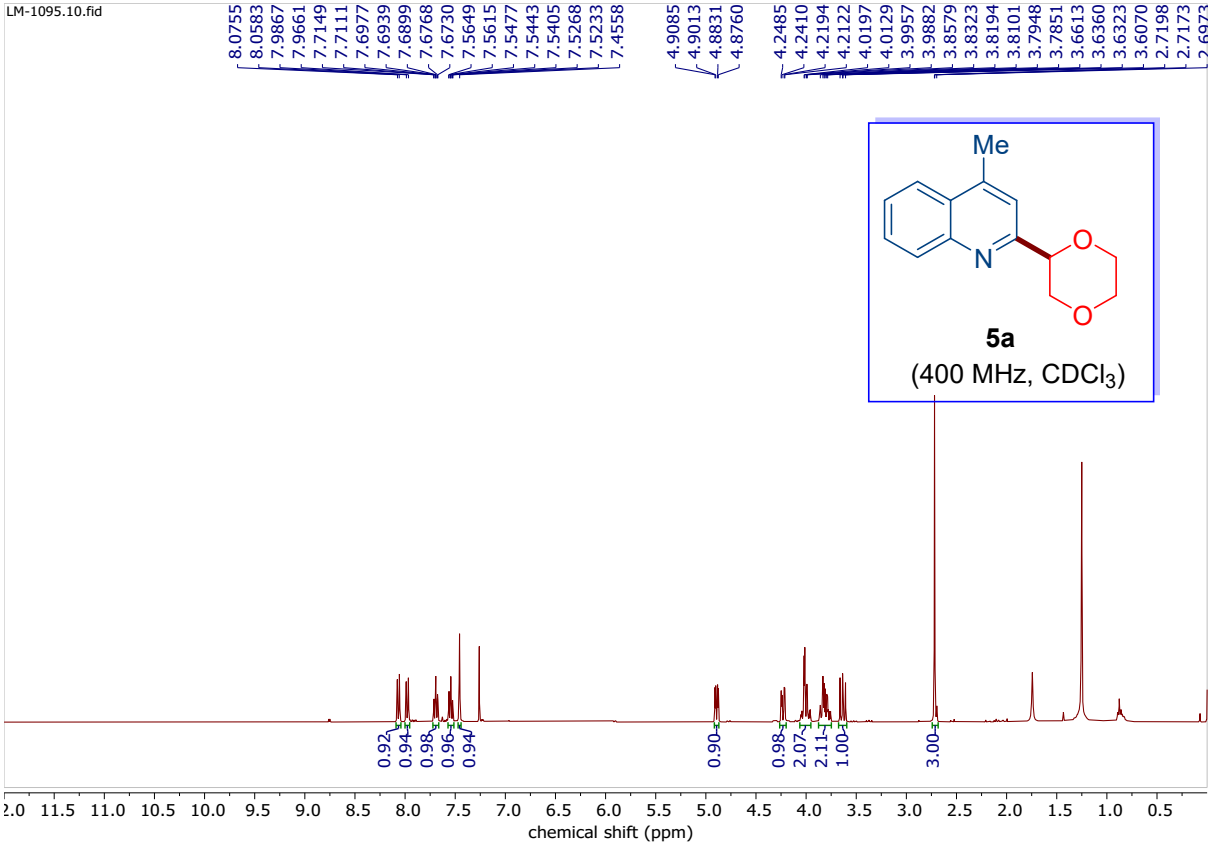


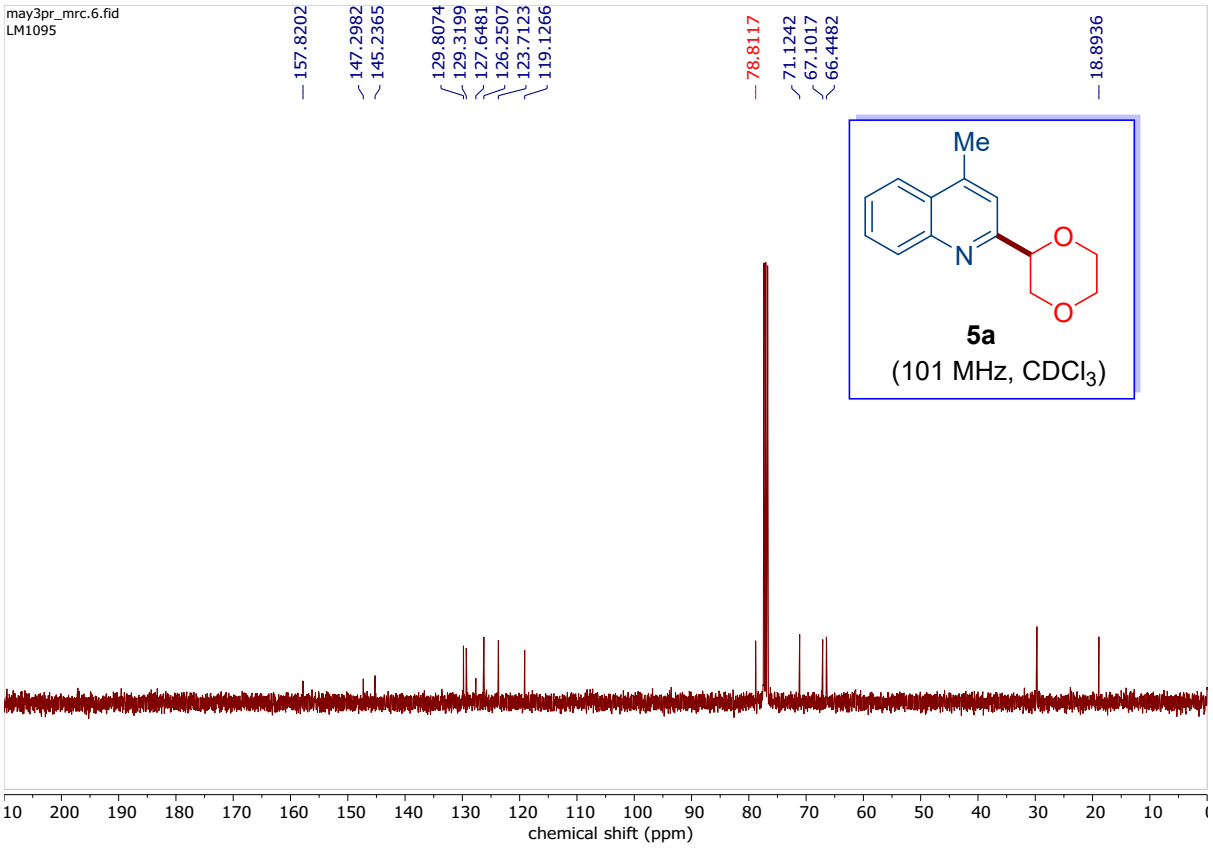
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **3u.**



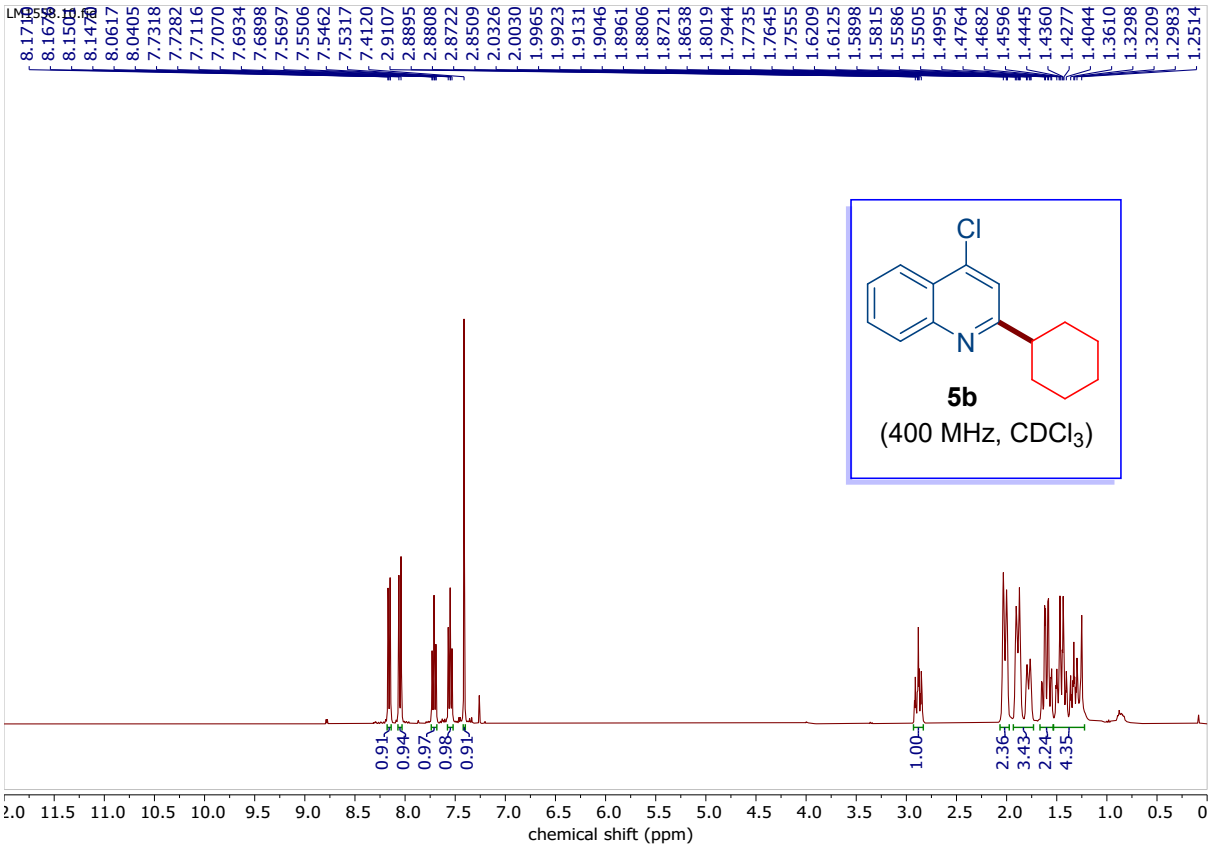


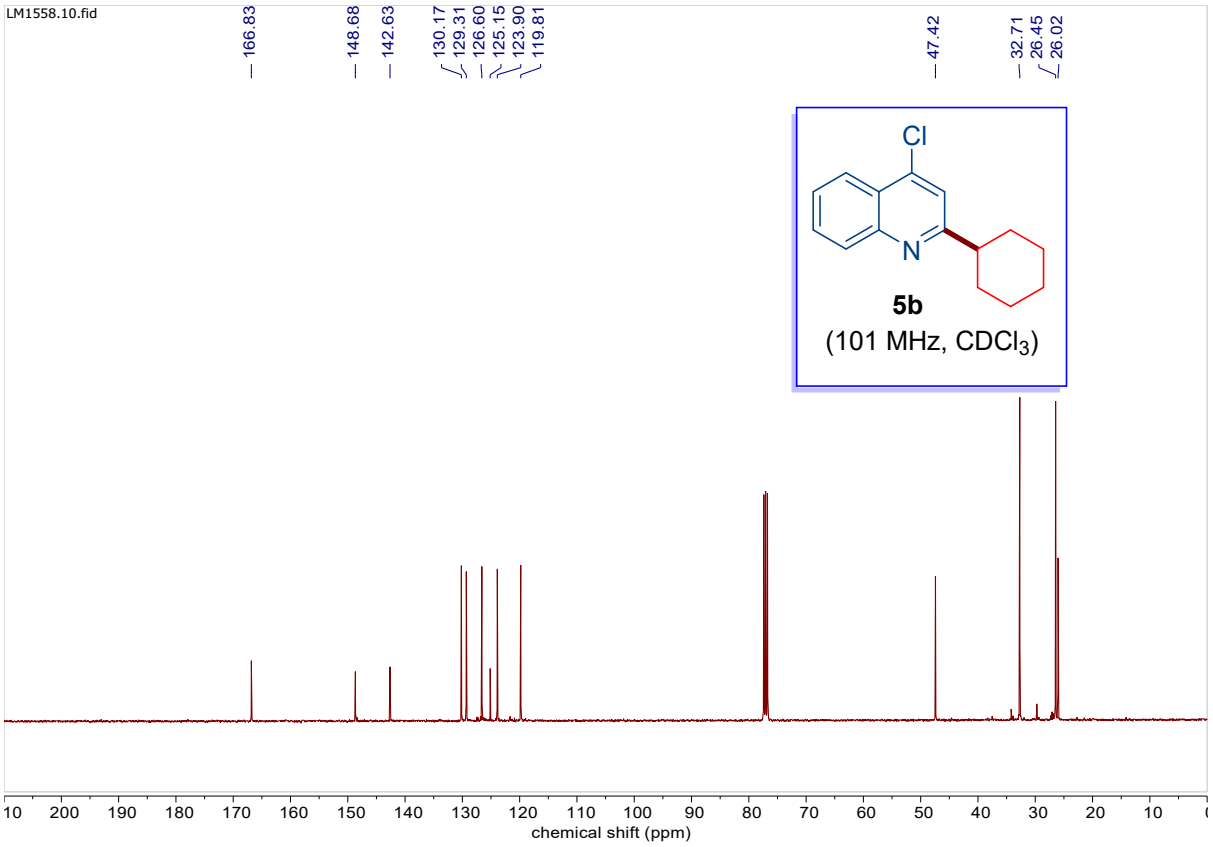
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5a.**



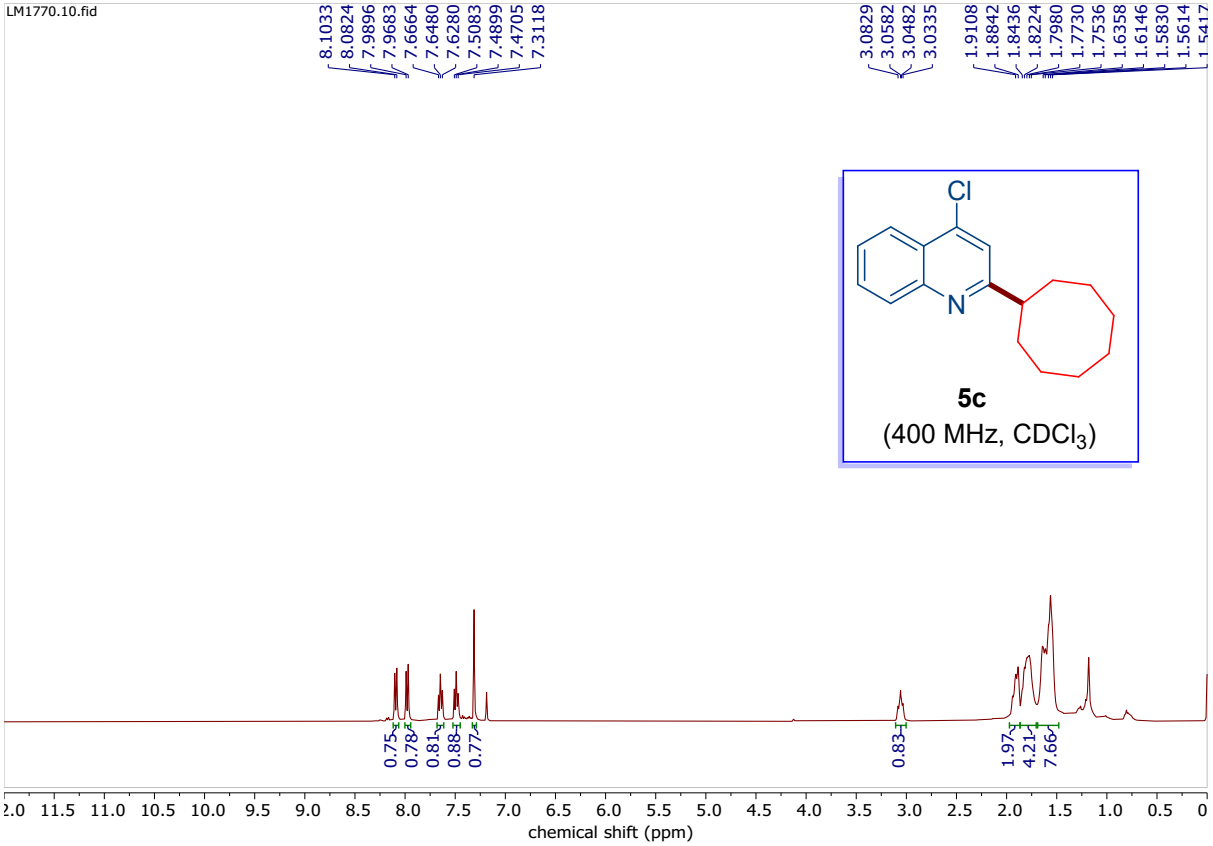


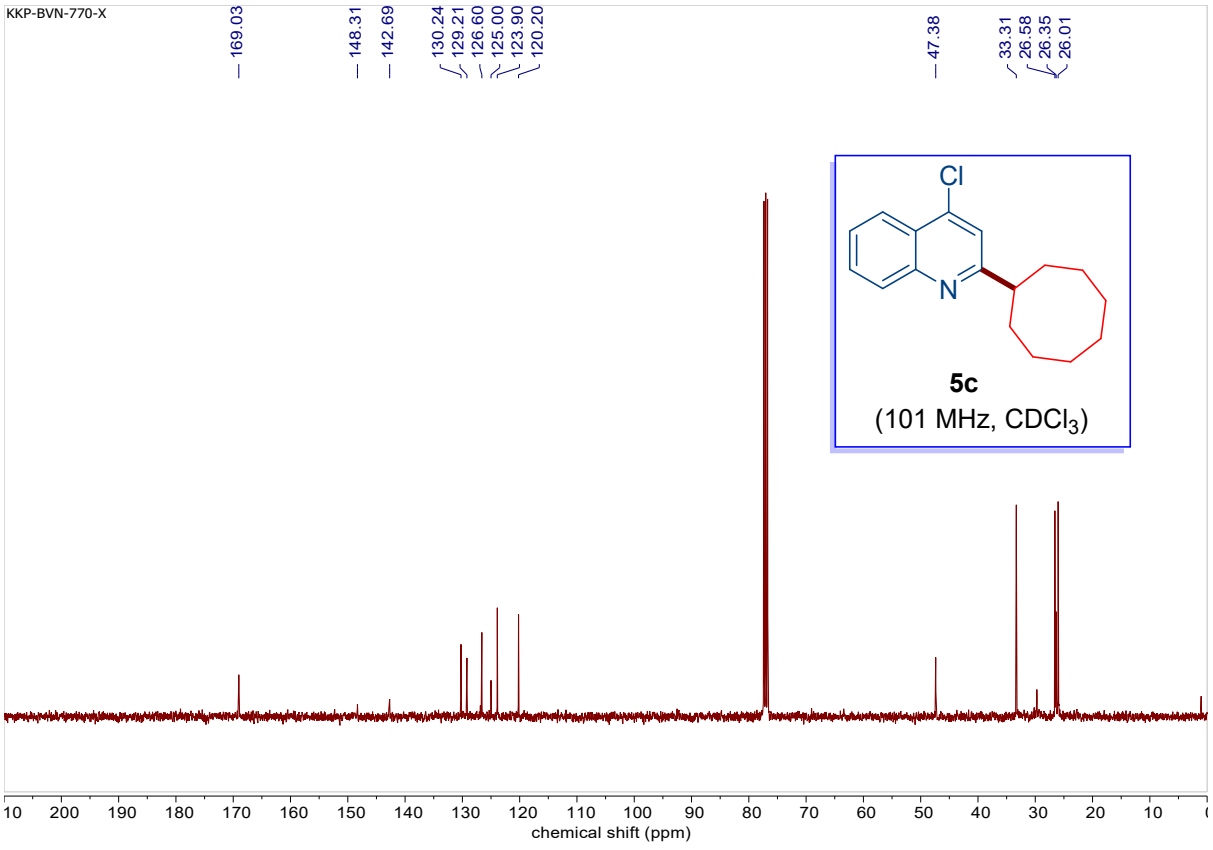
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5b.**



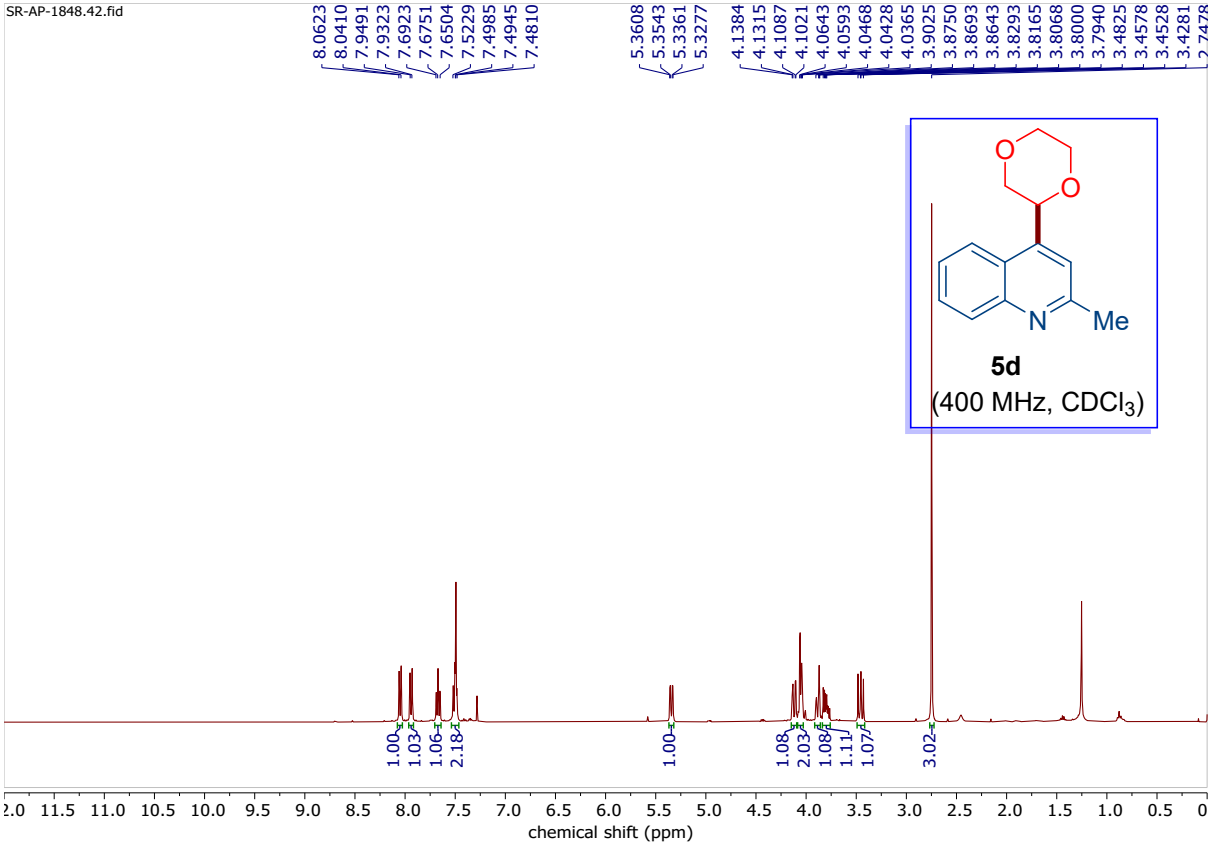


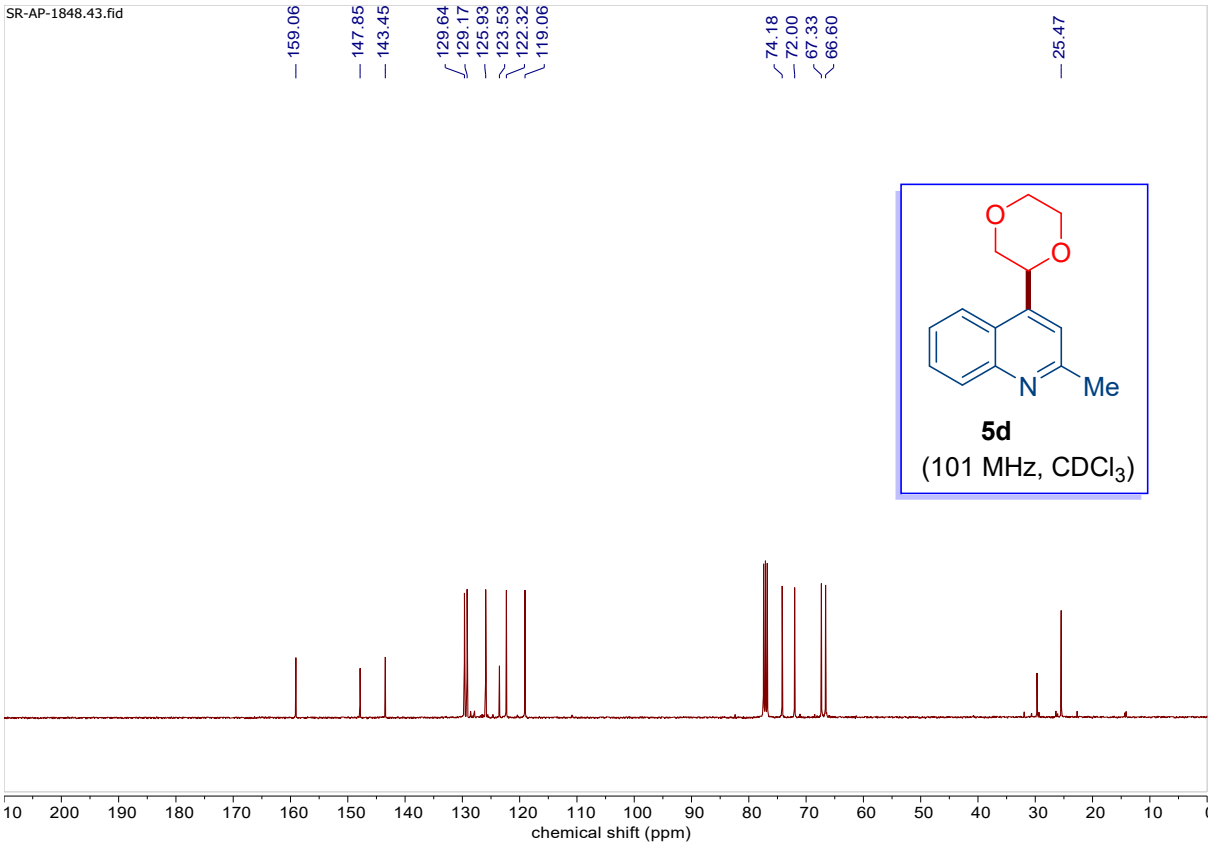
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5c.**



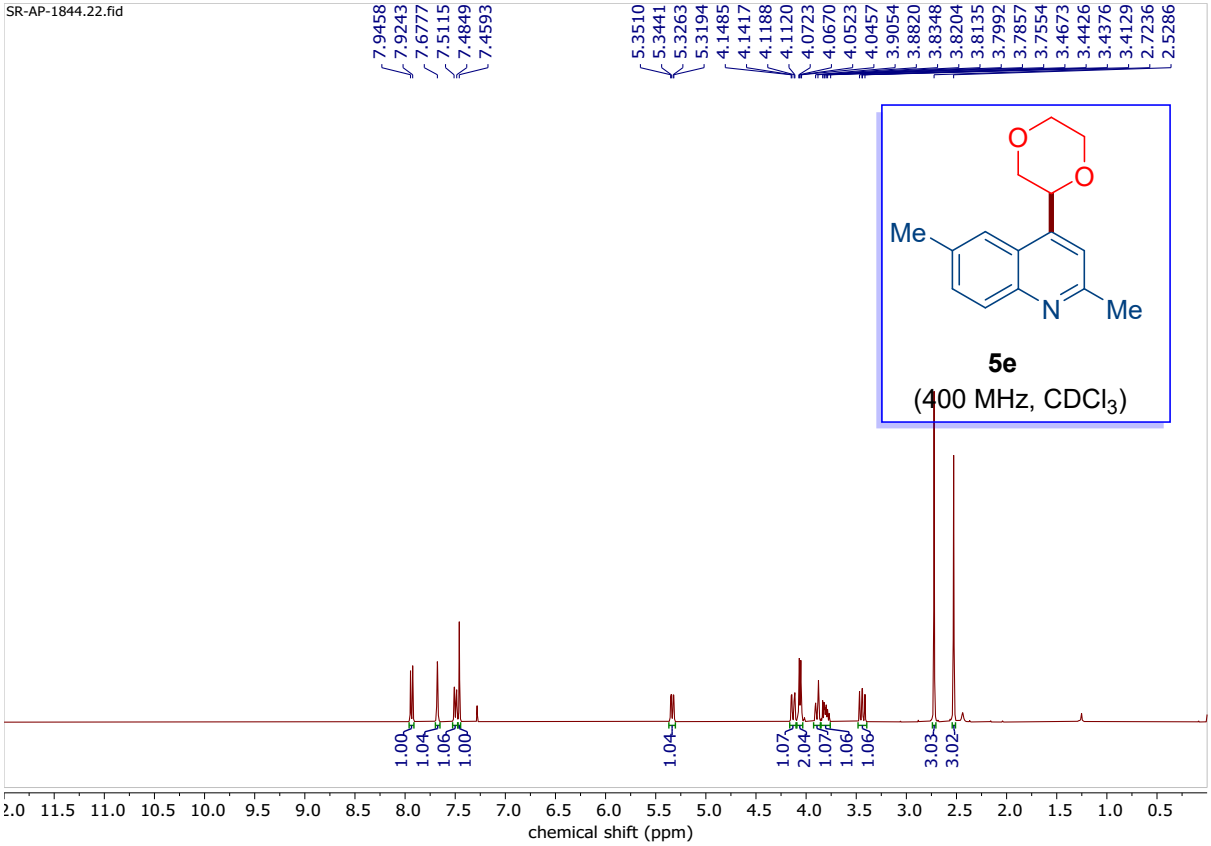


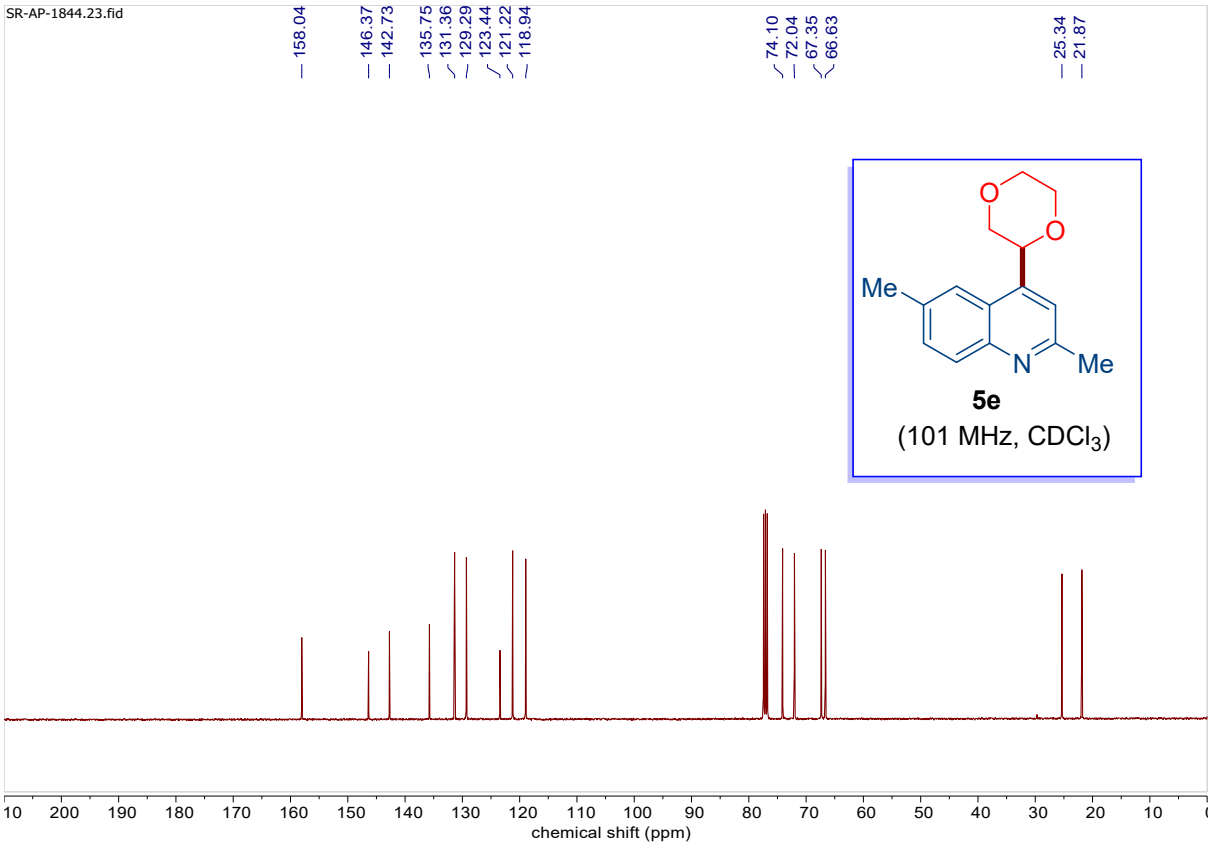
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5d**.



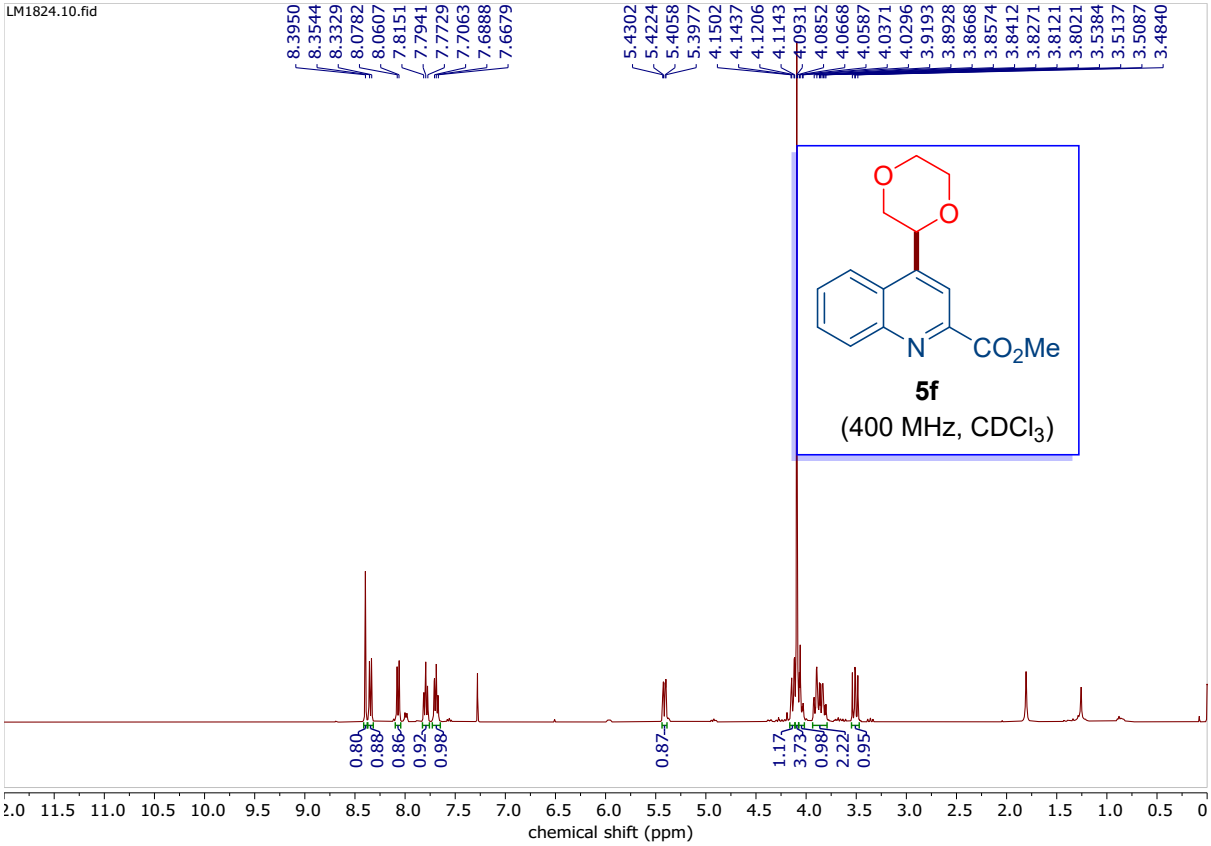


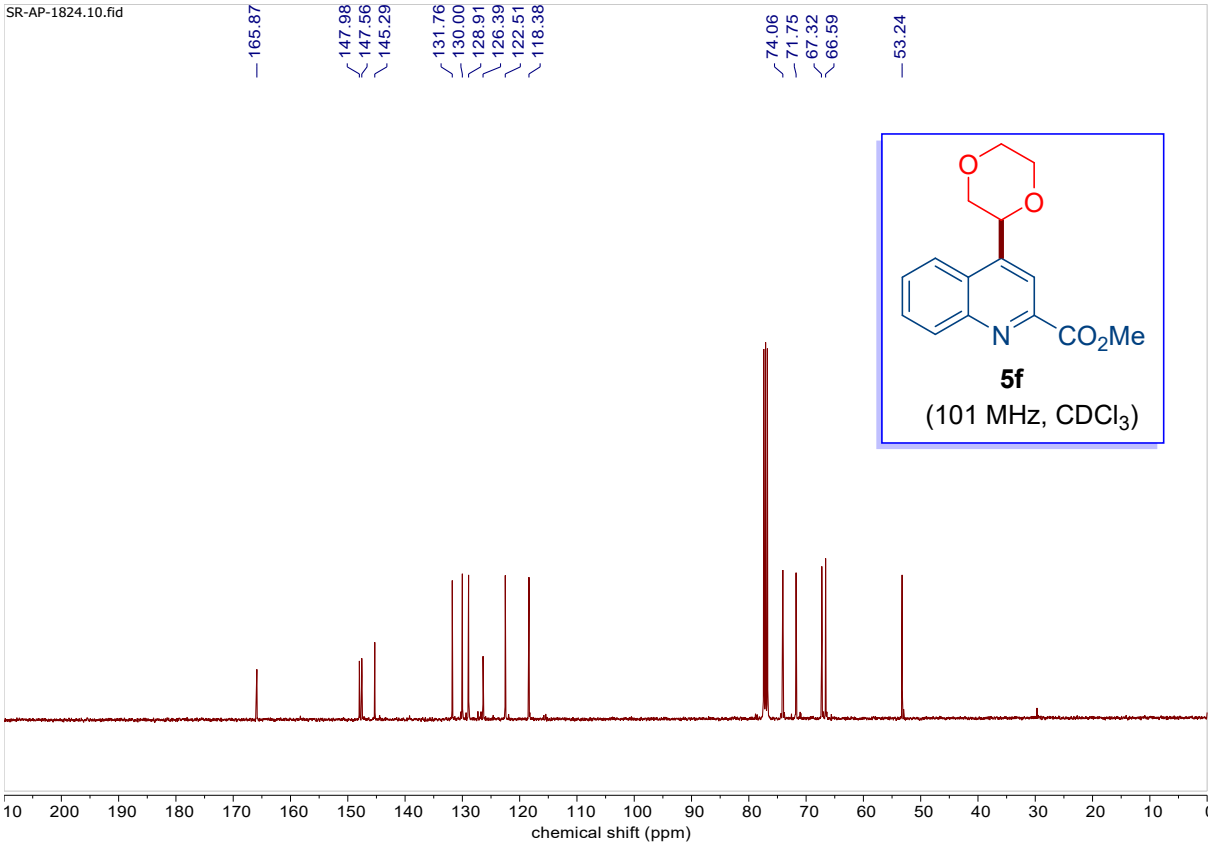
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5e.**



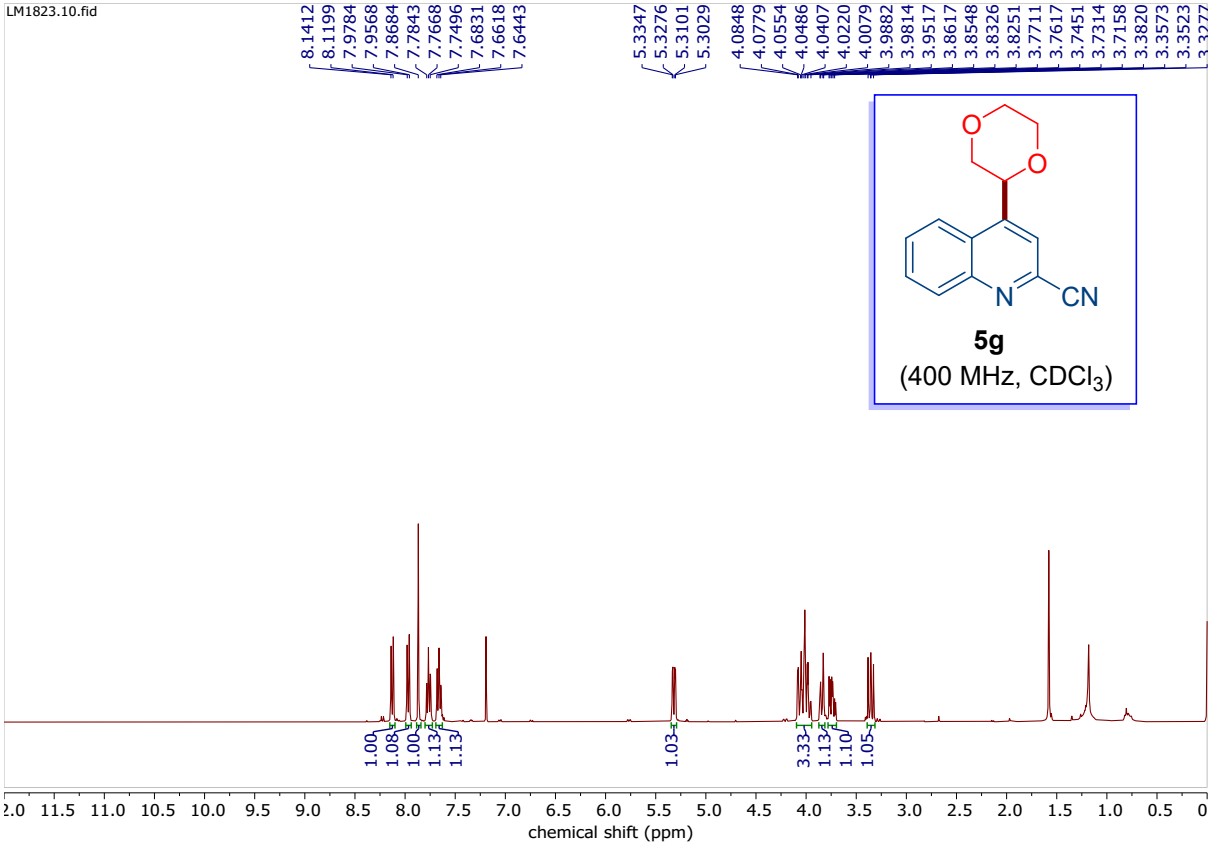


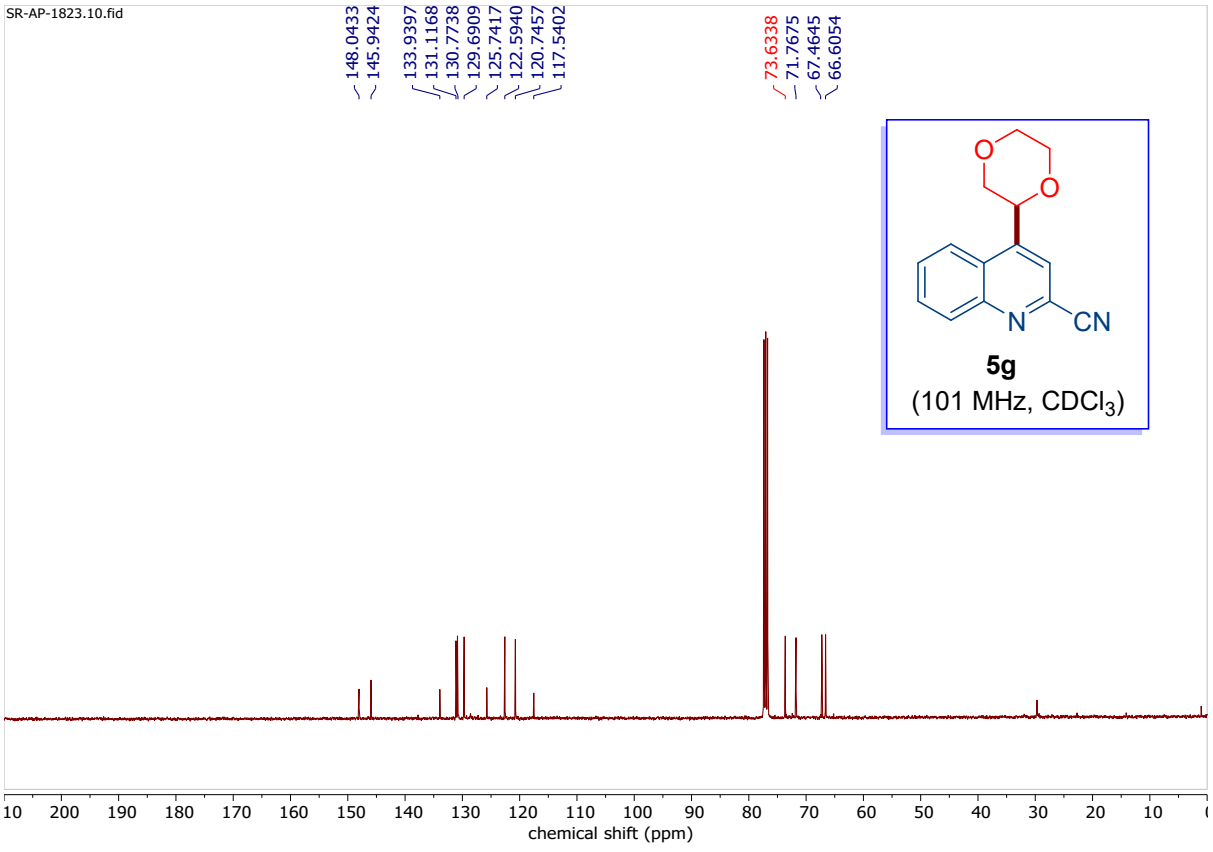
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5f.**



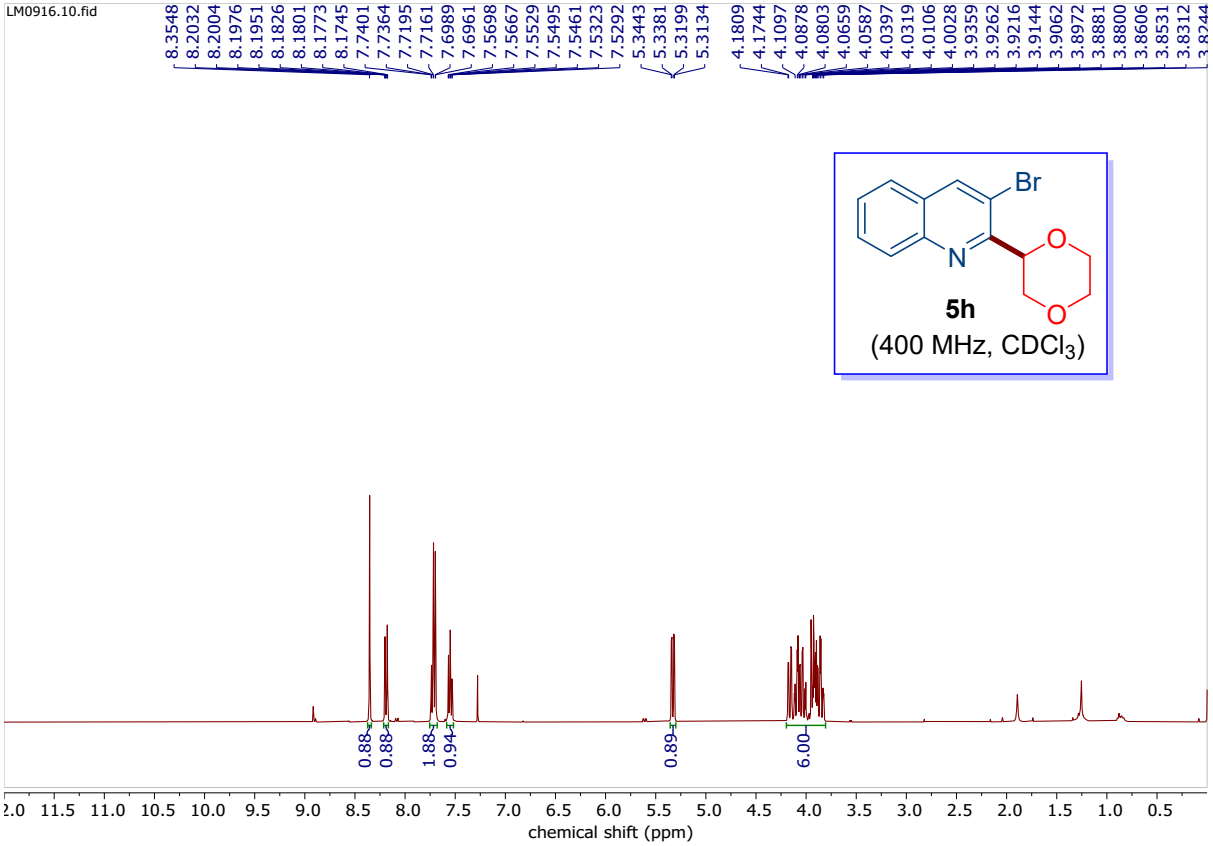


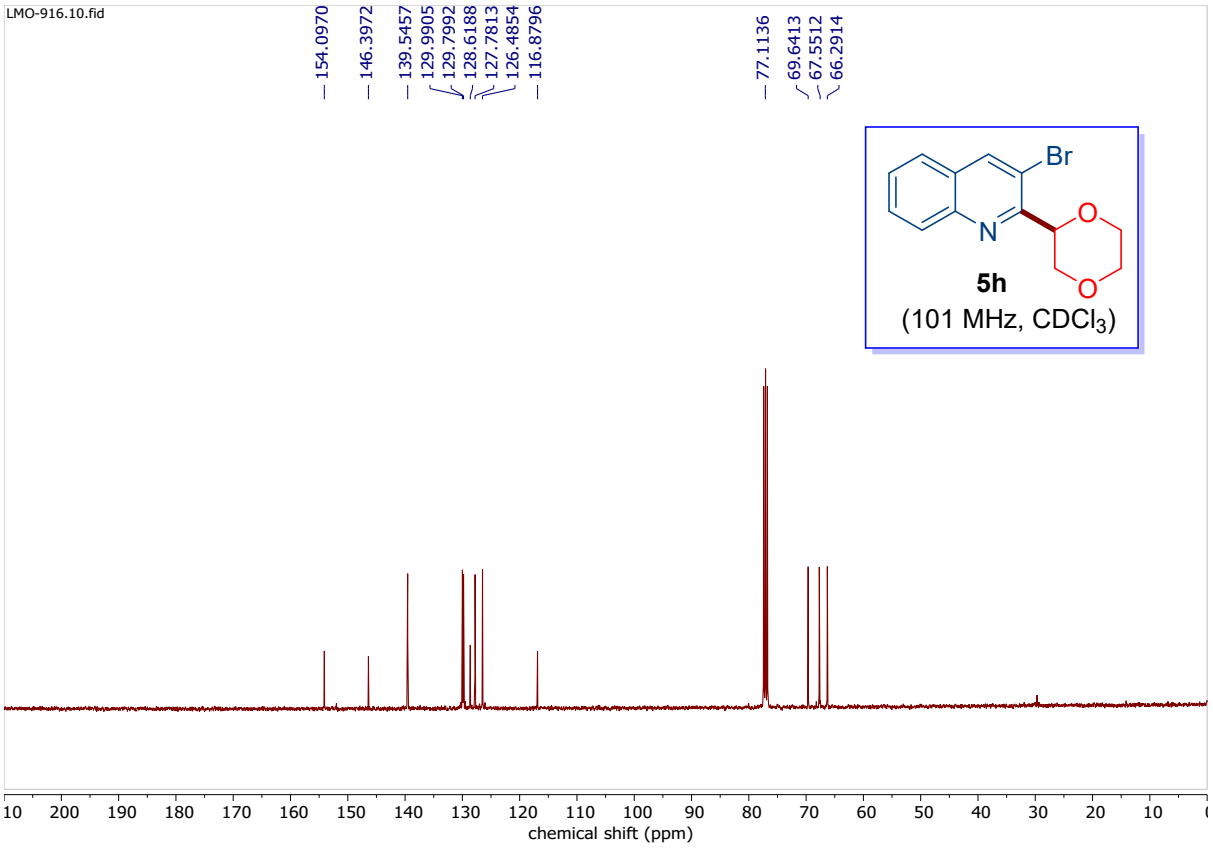
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5g**.



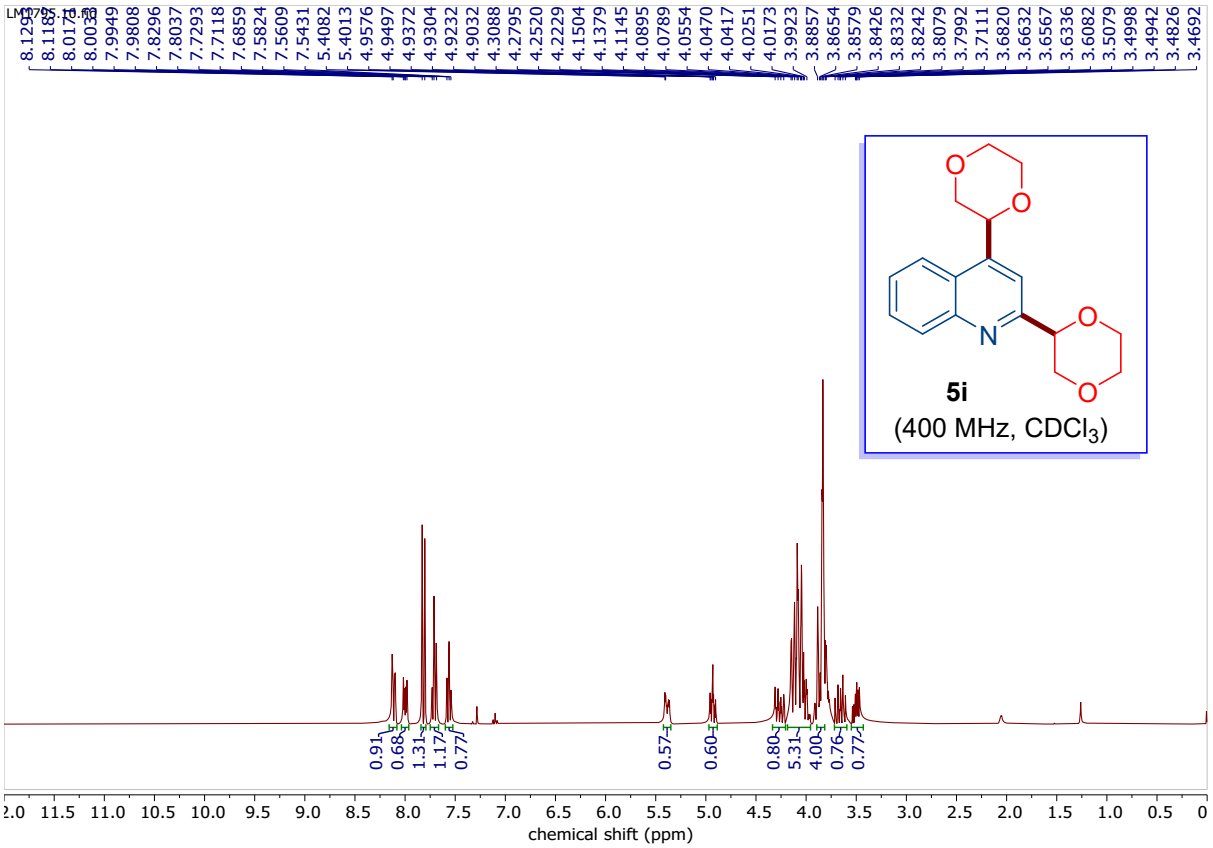


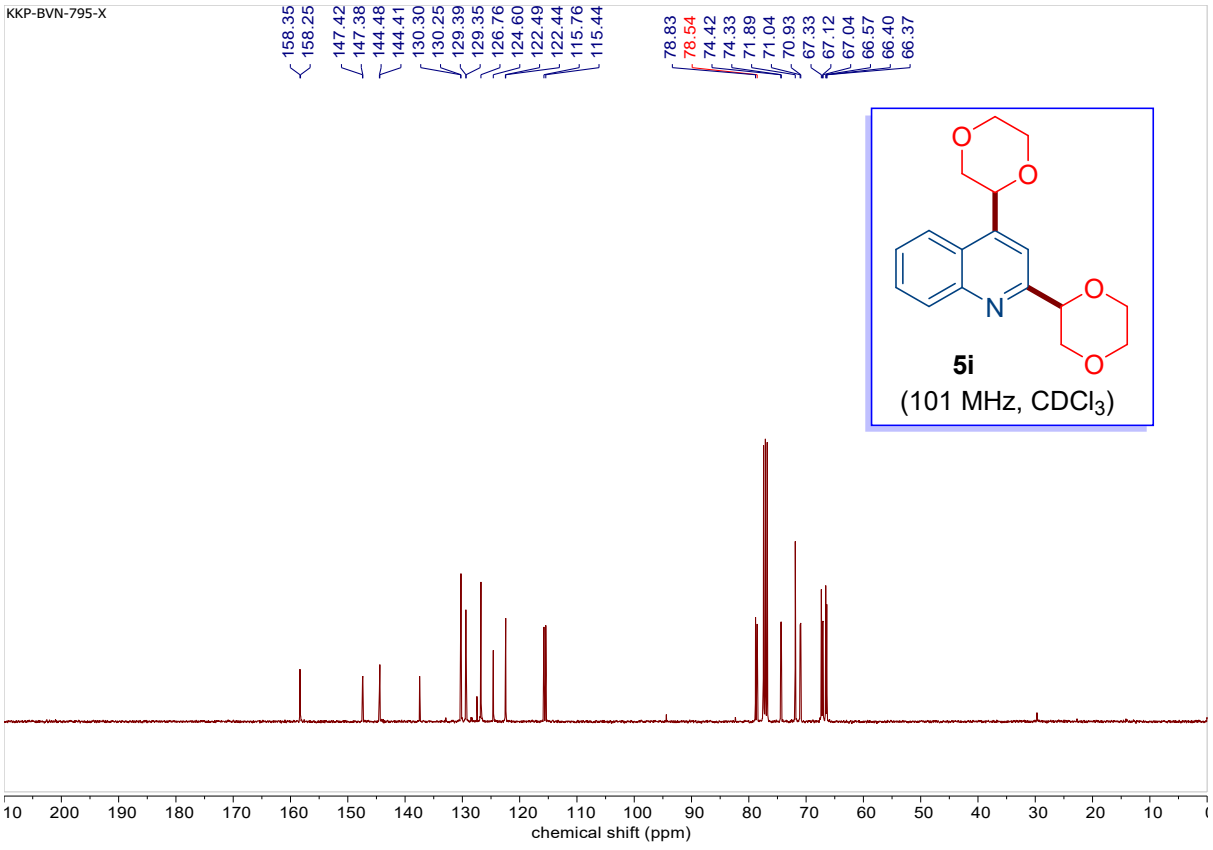
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5h**.



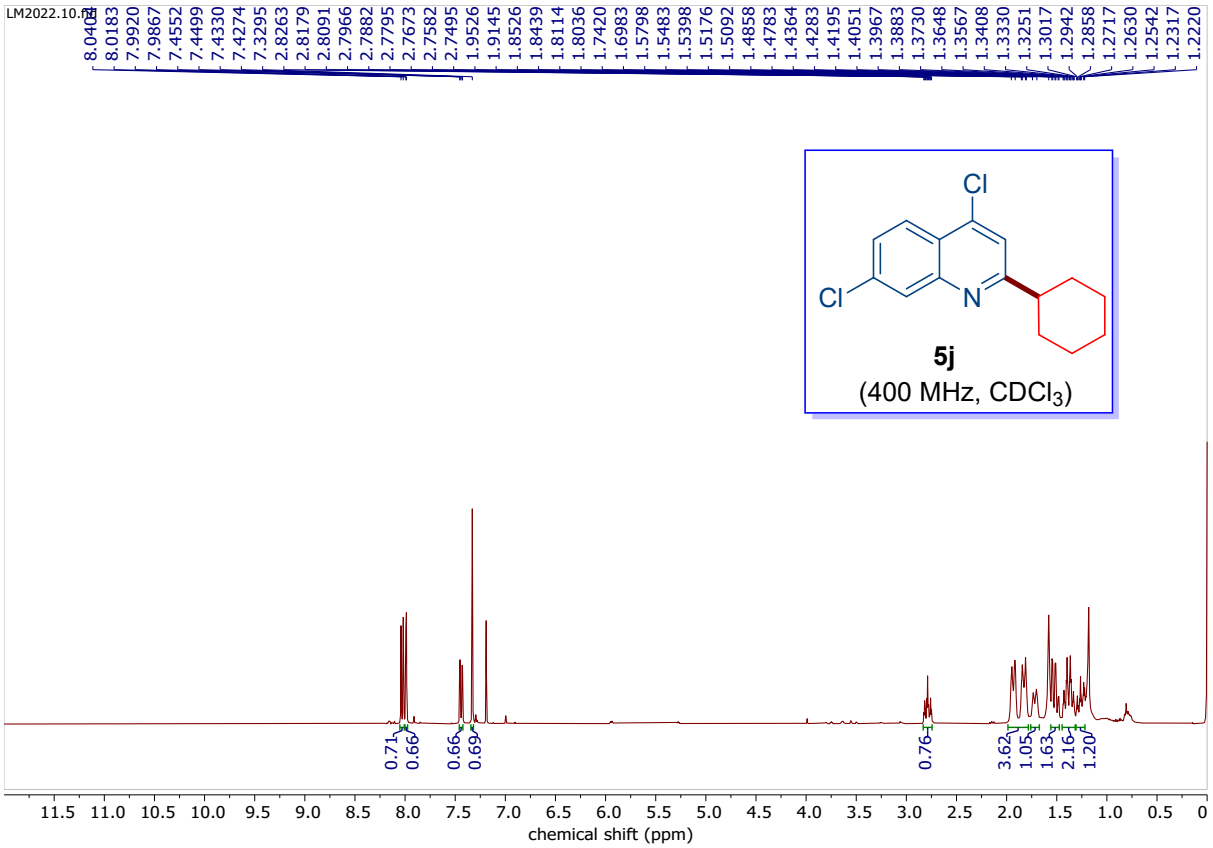


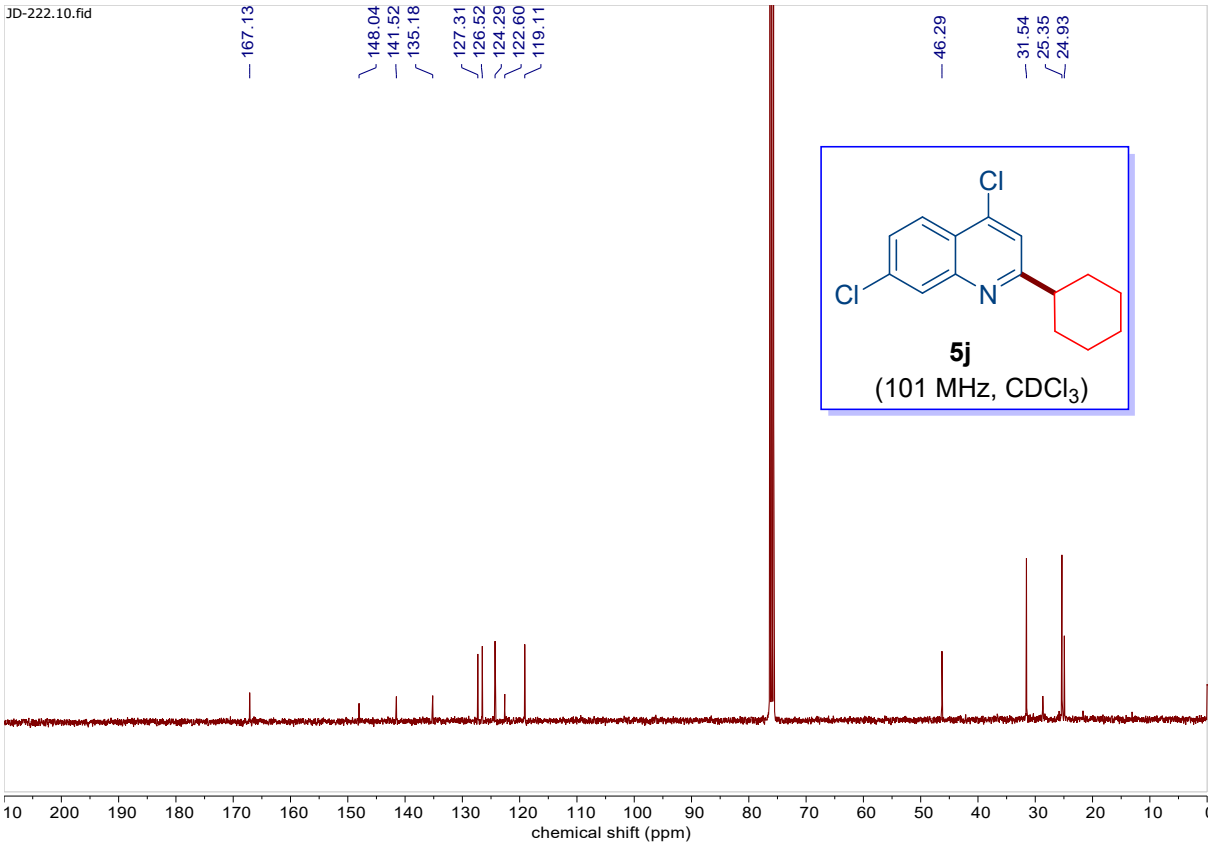
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5i.**



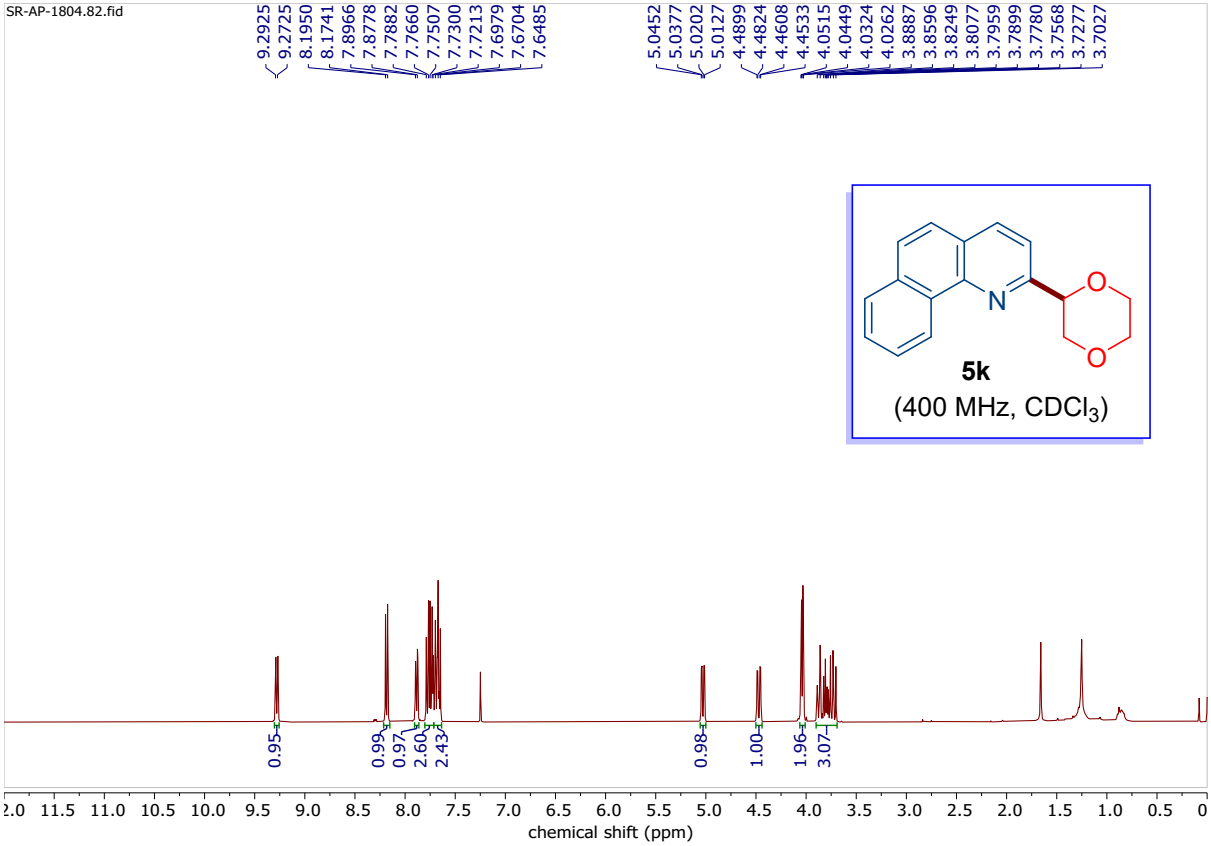


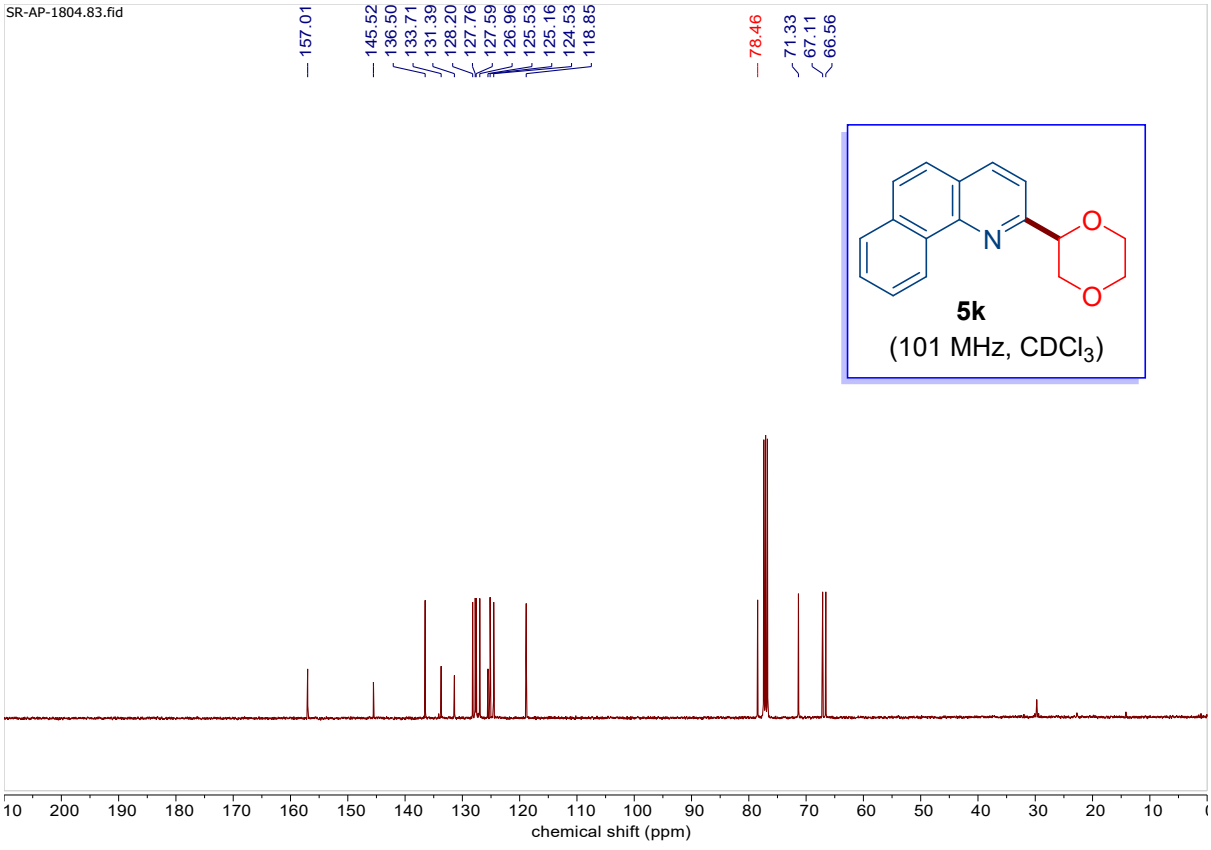
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5j.**



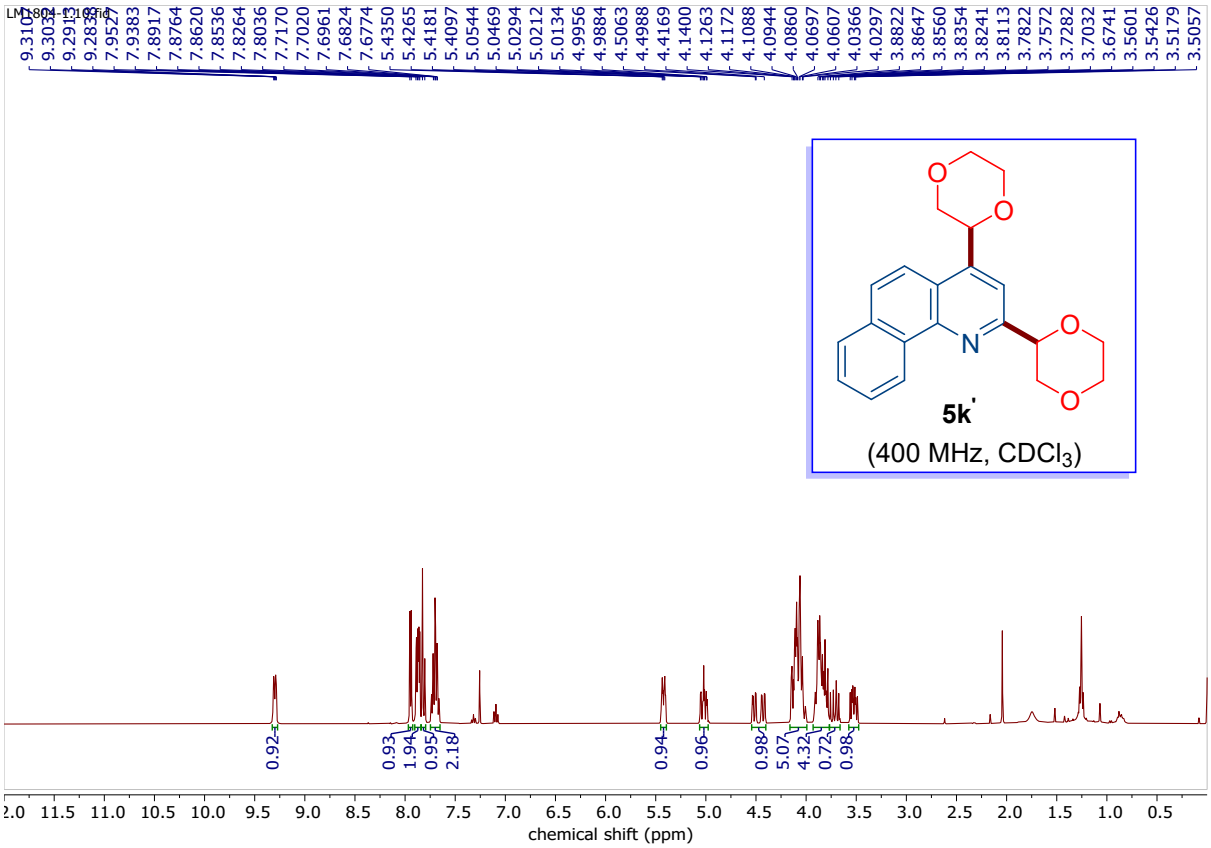


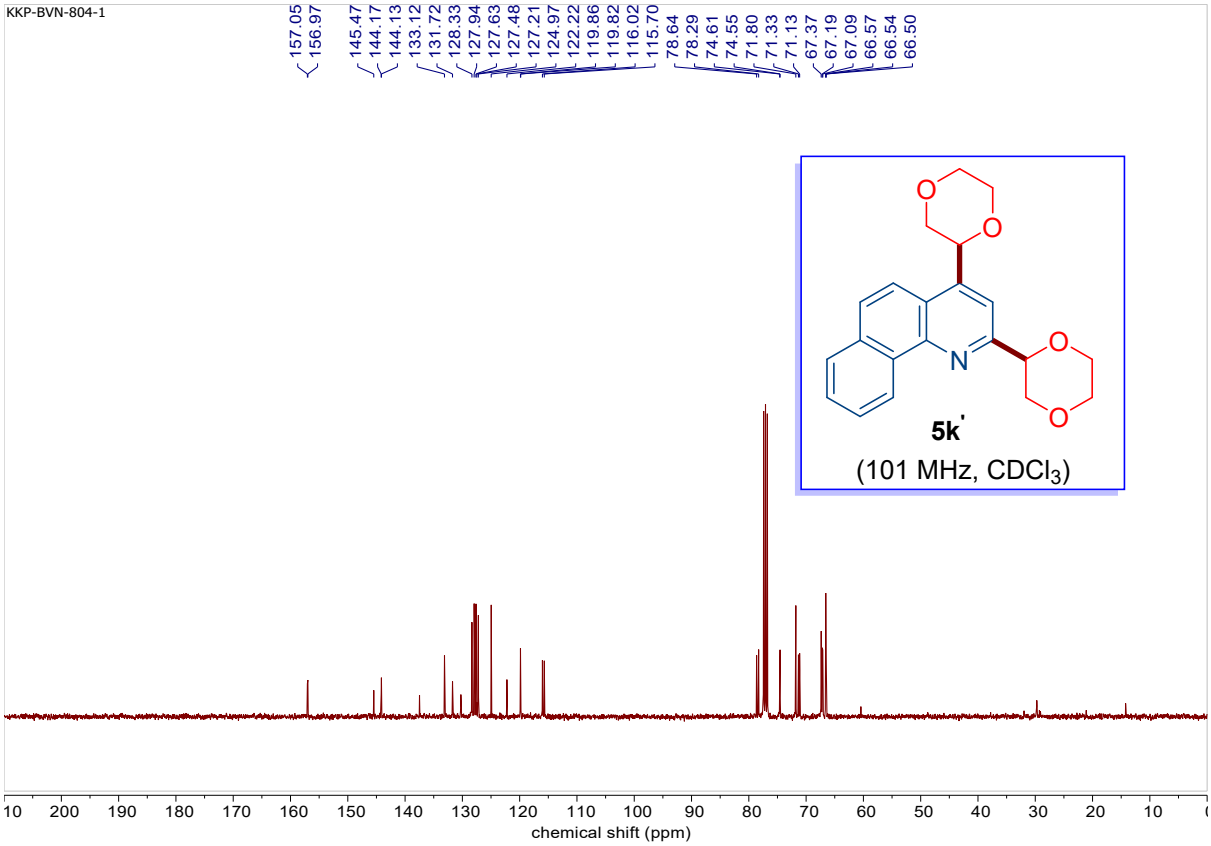
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5k.**



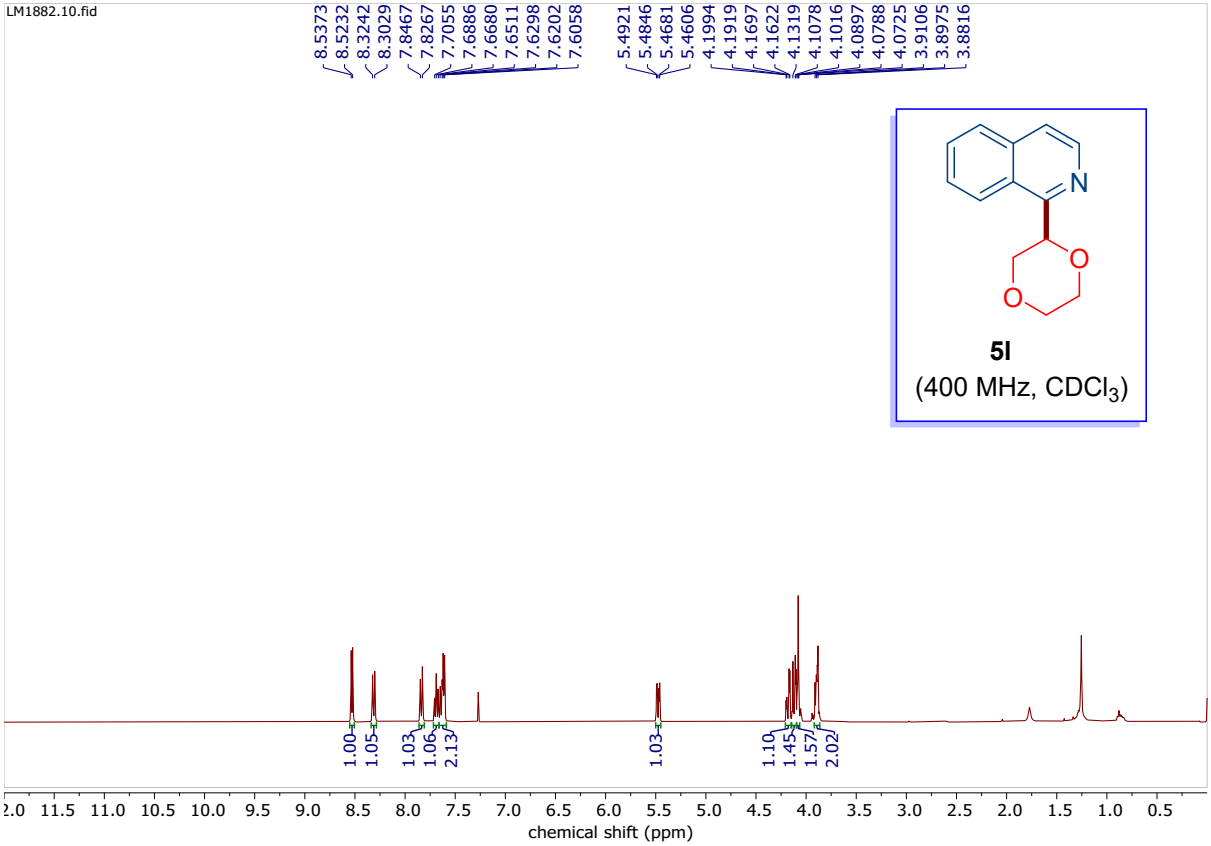


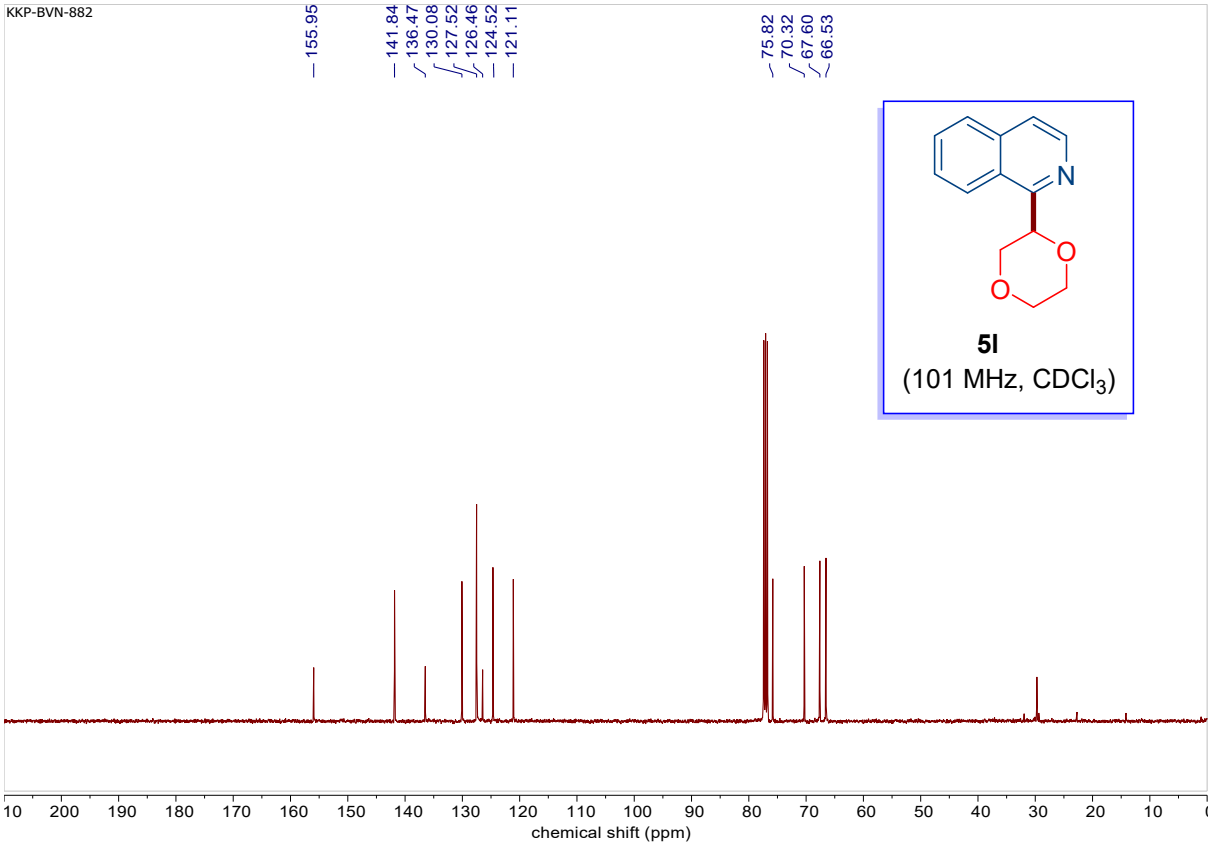
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5k'**.



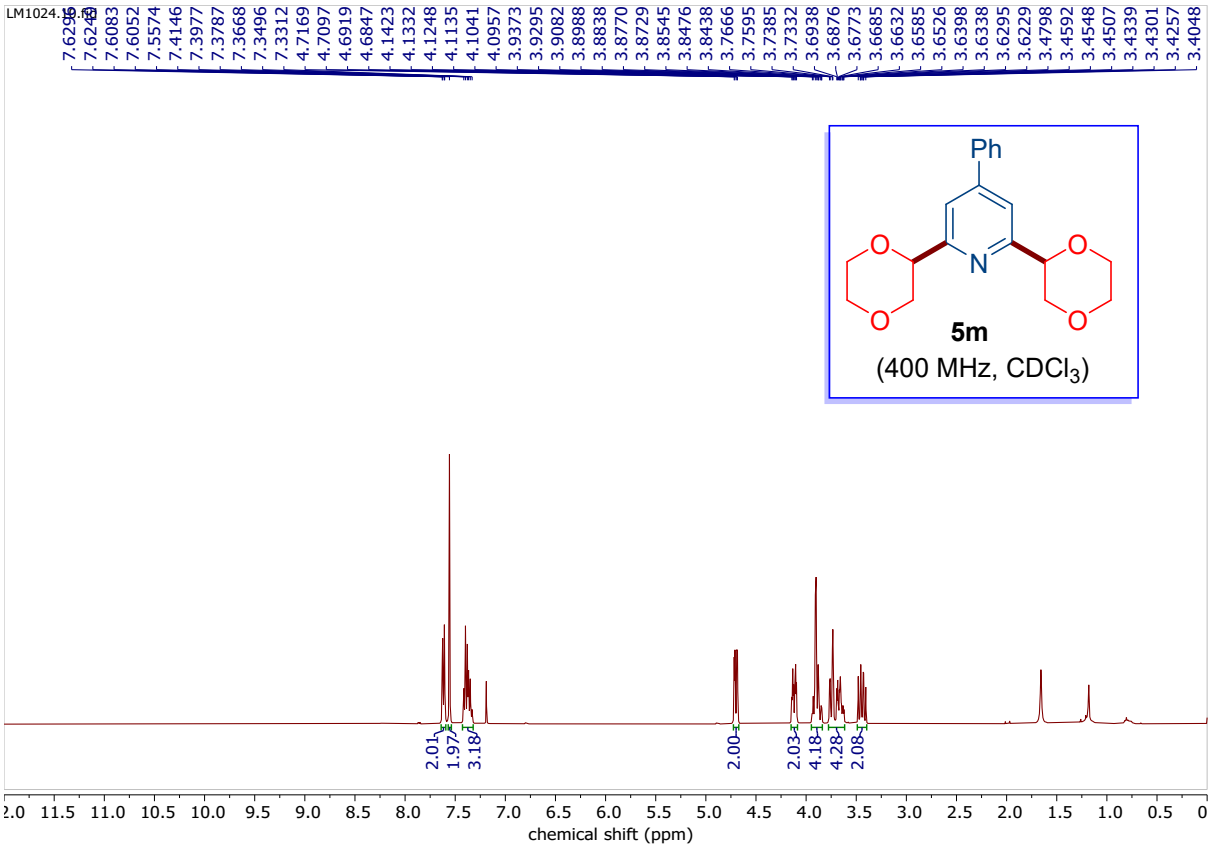


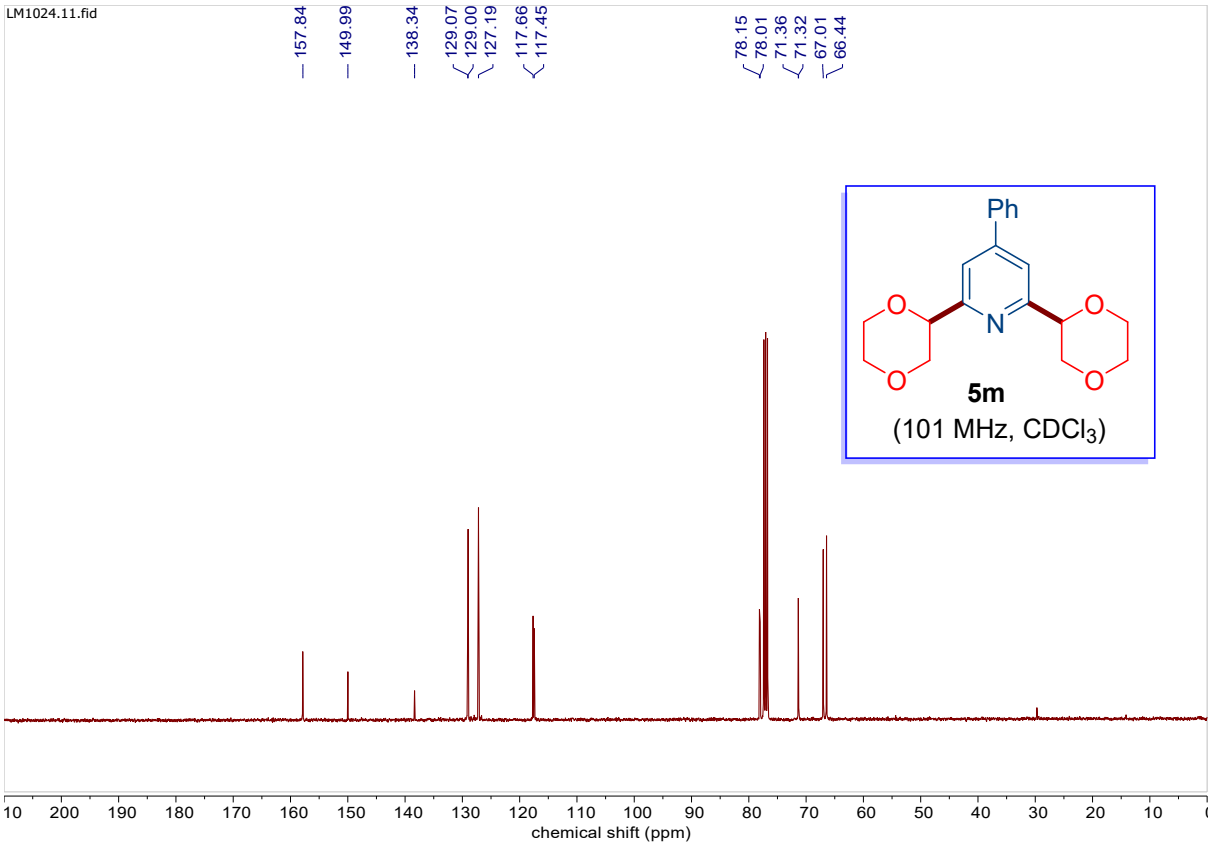
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5l**.



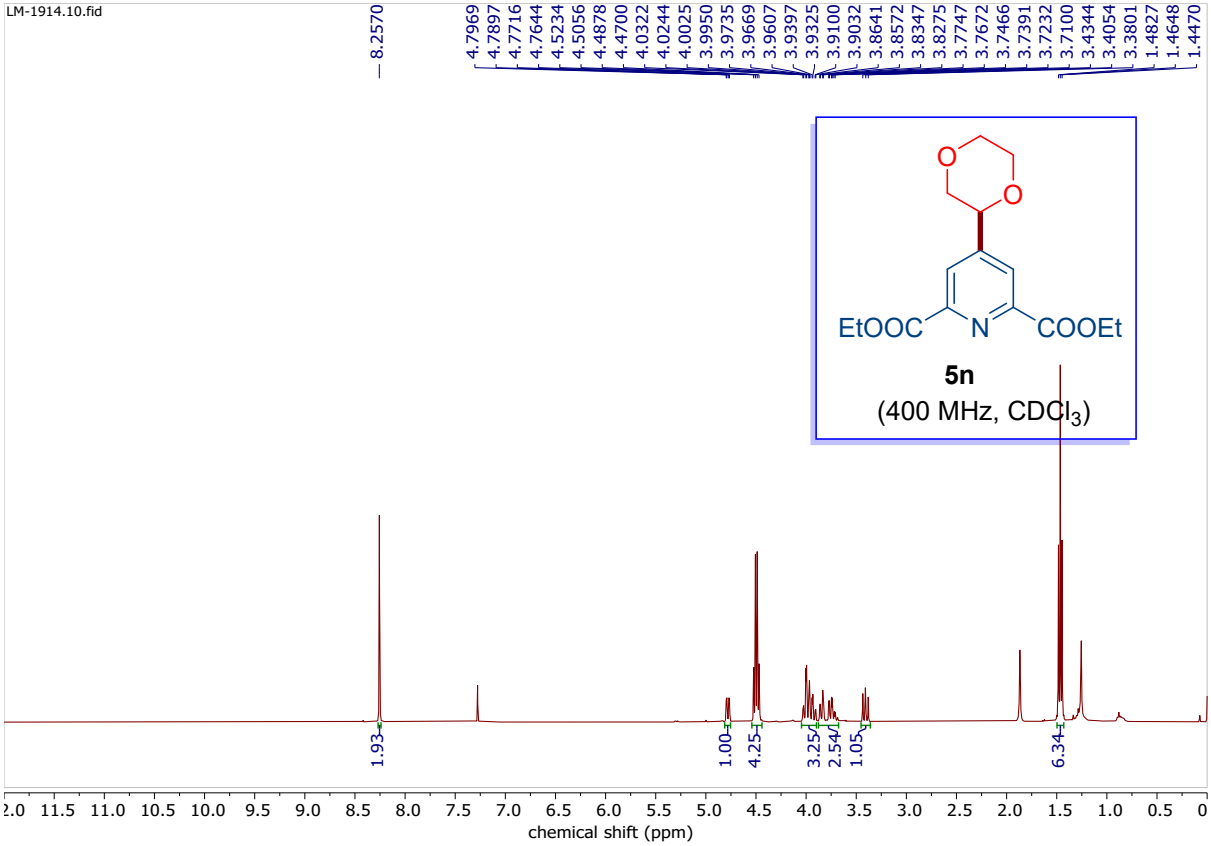


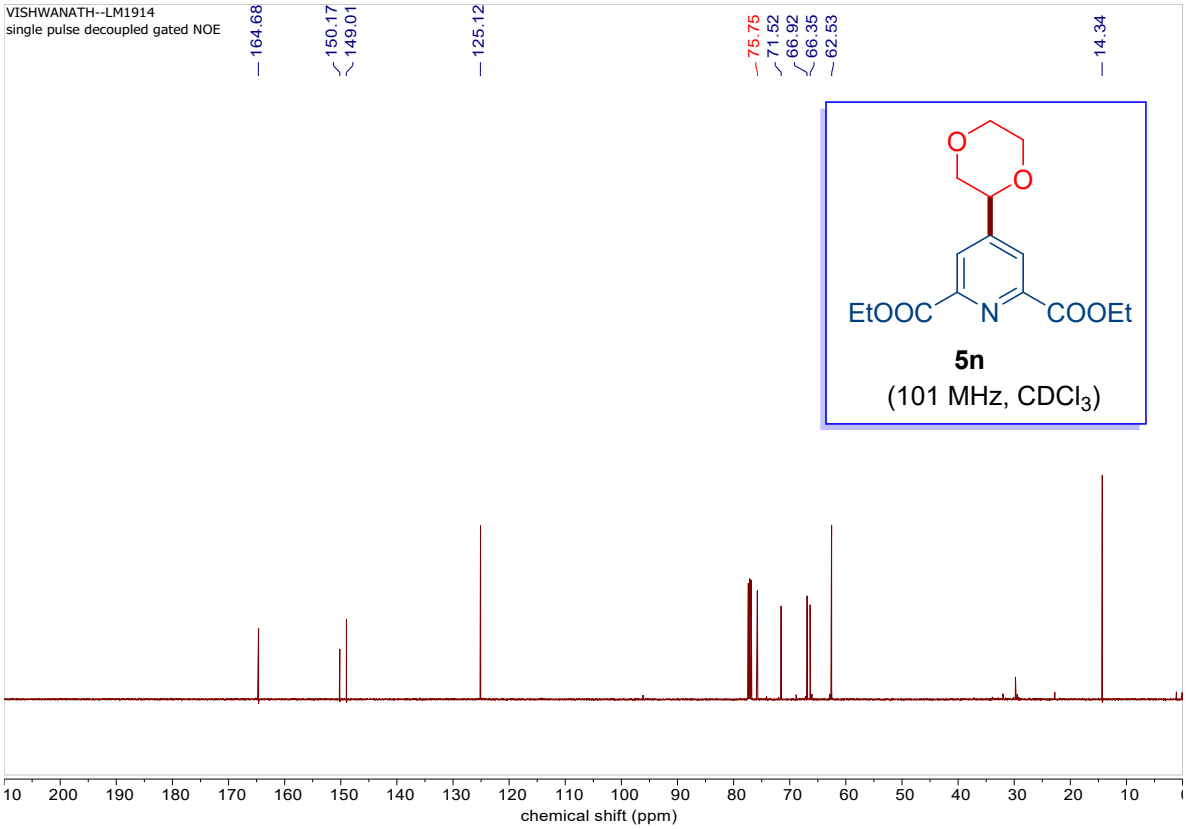
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5m**.



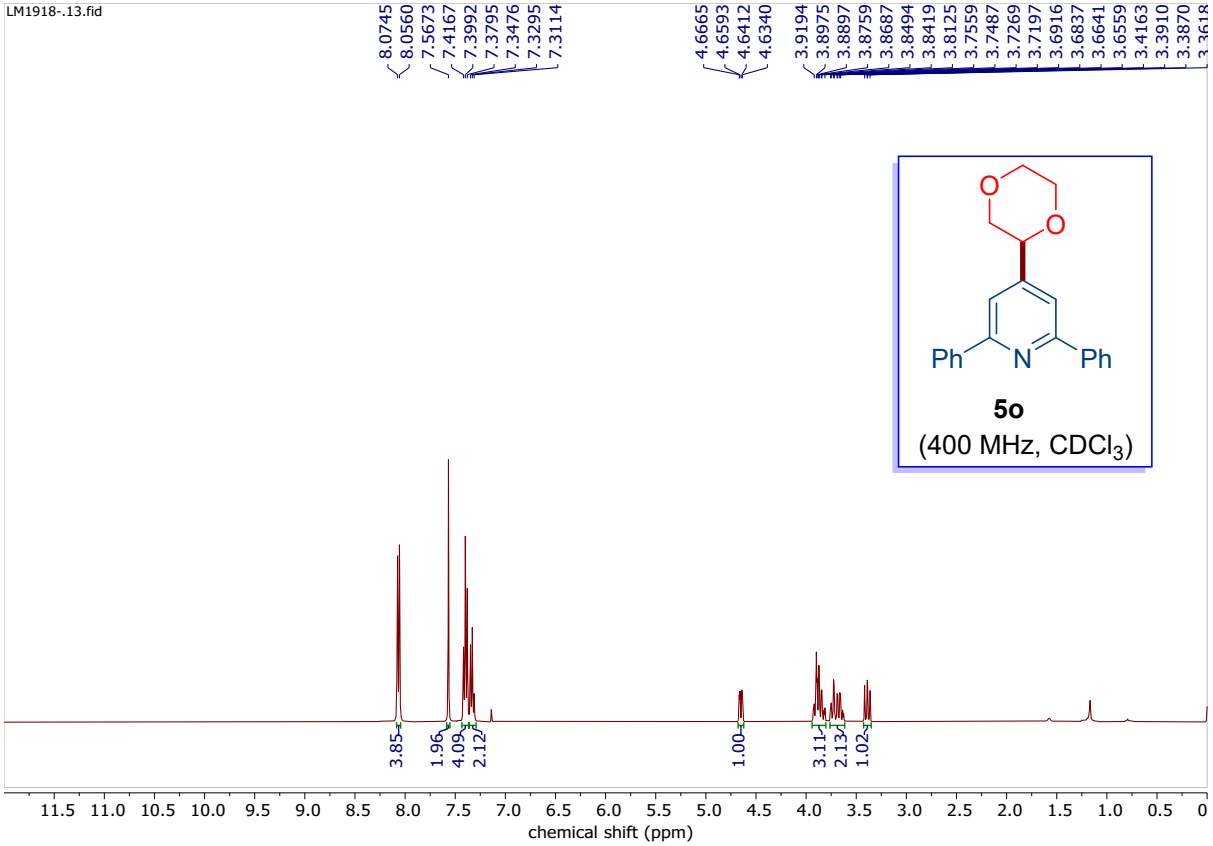


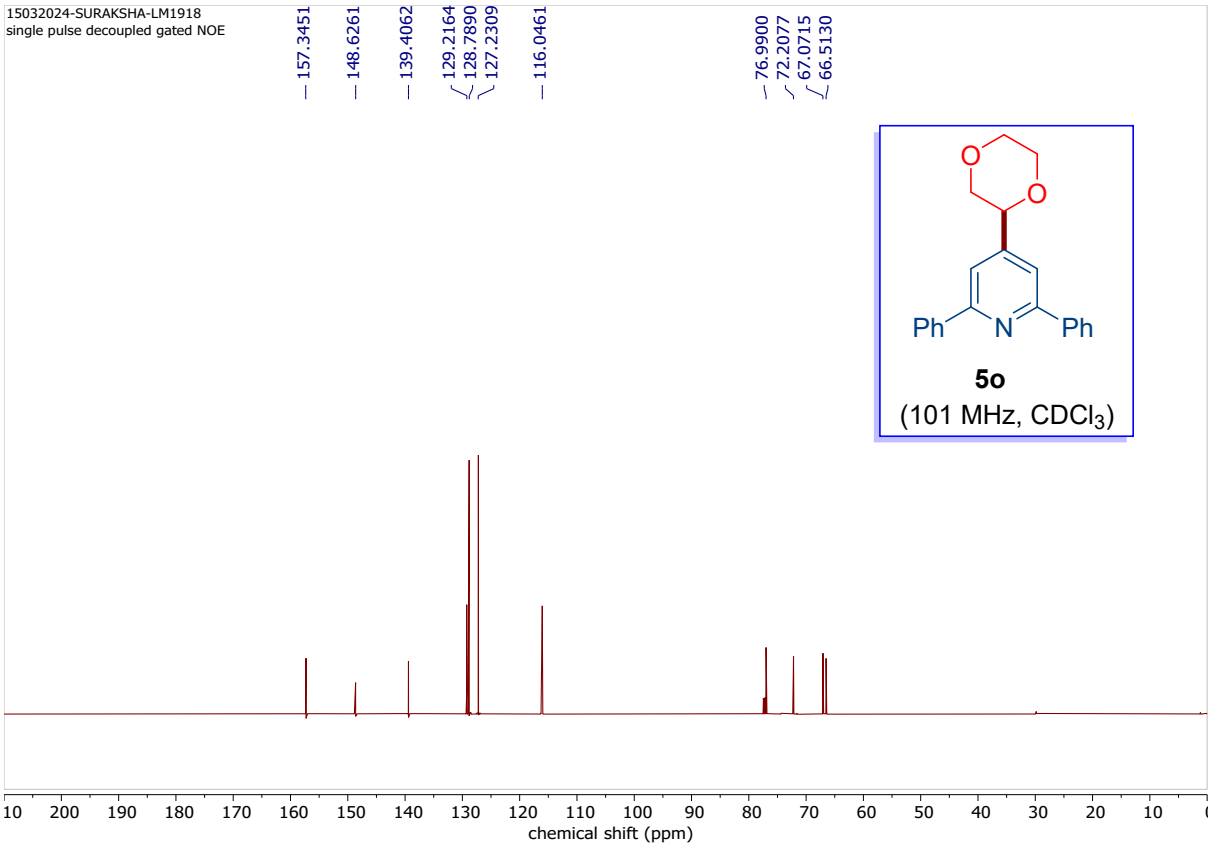
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5n**.



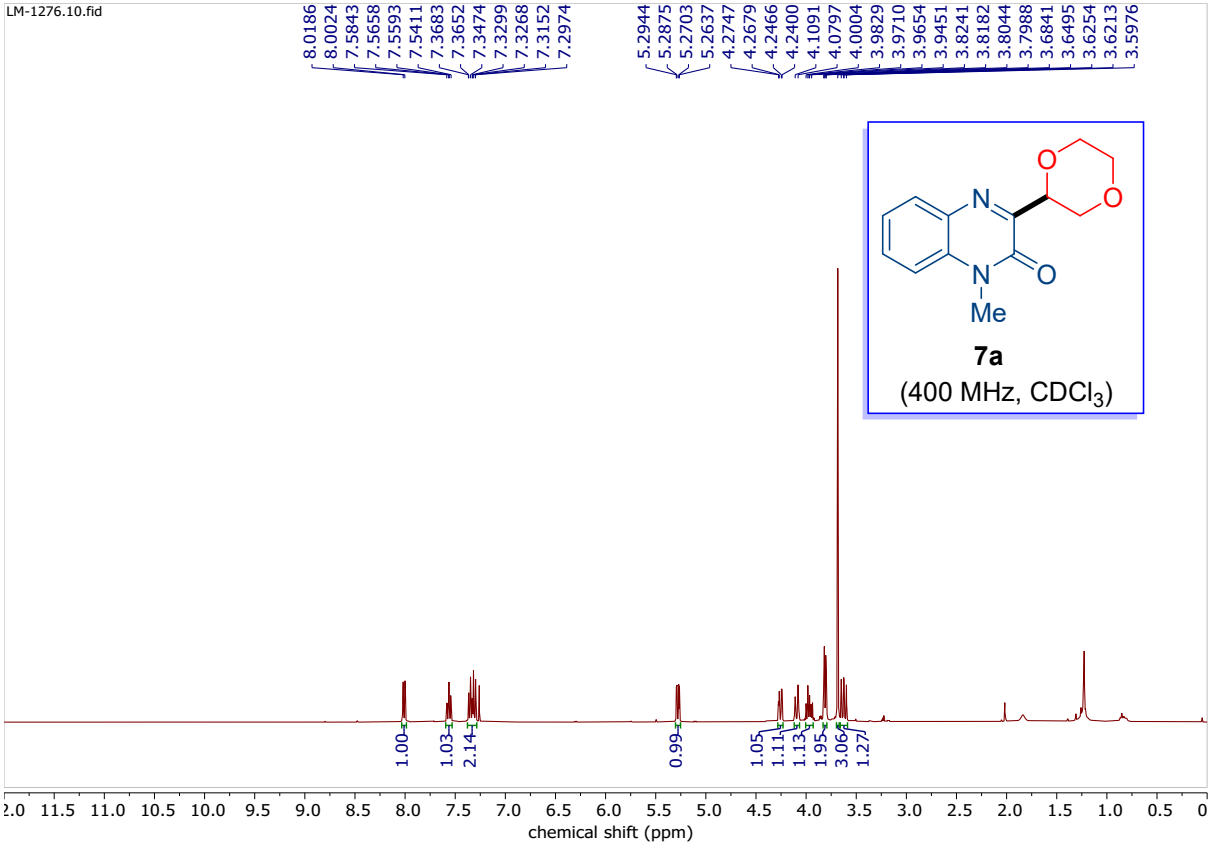


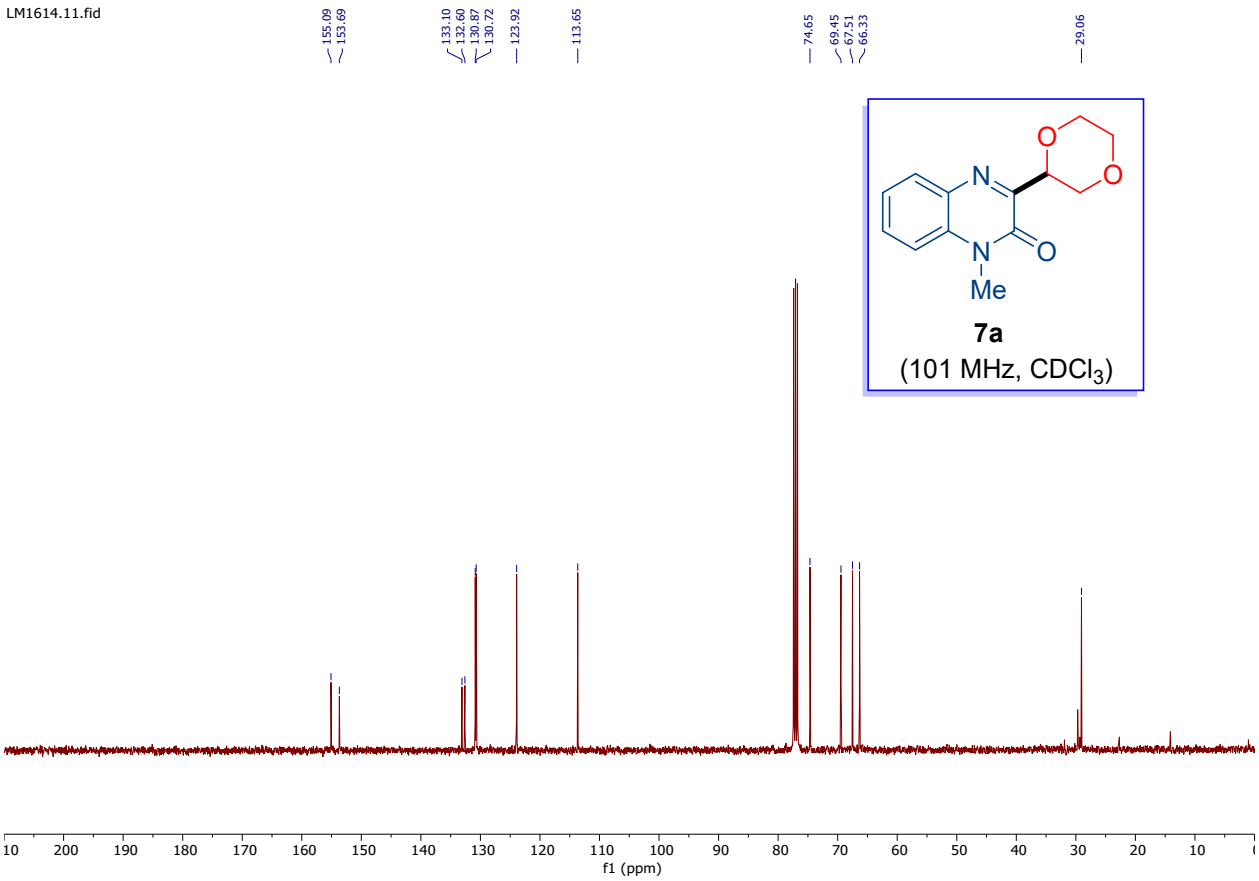
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **5o**.



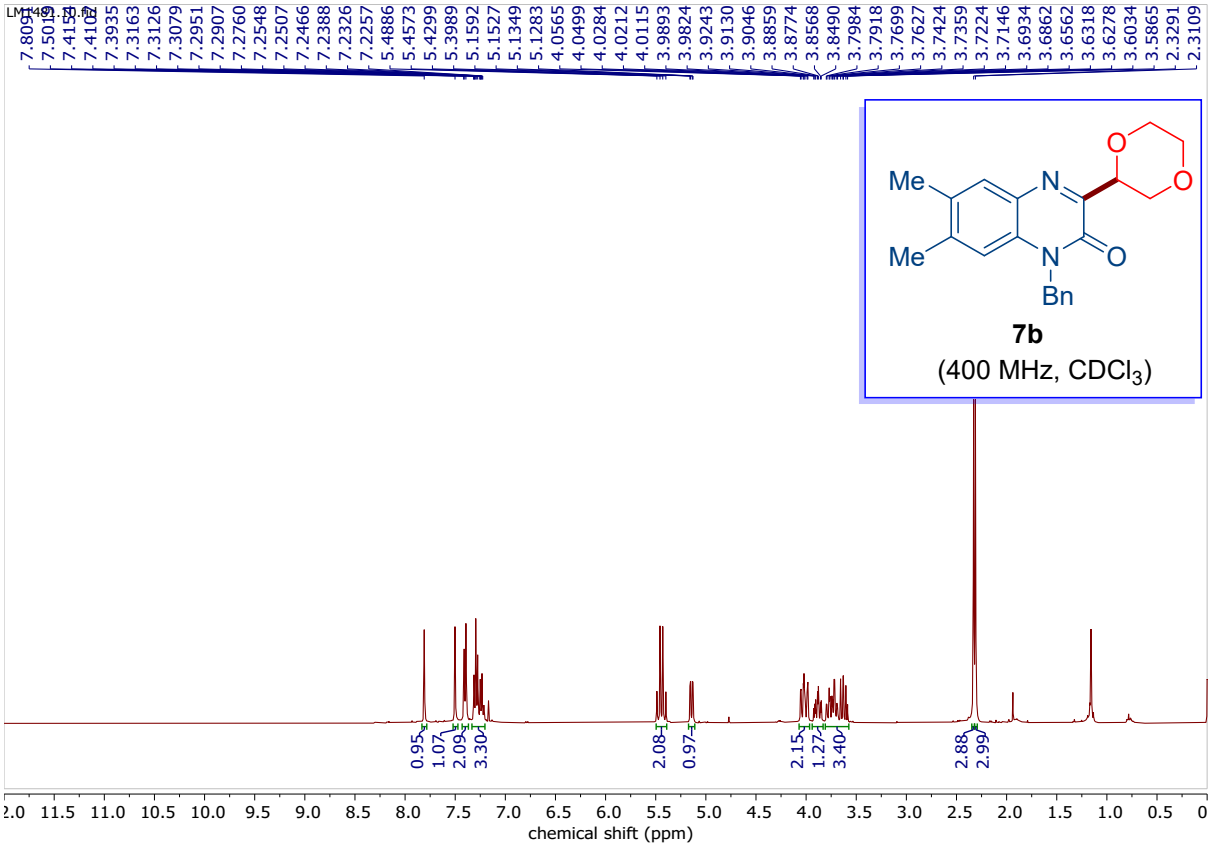


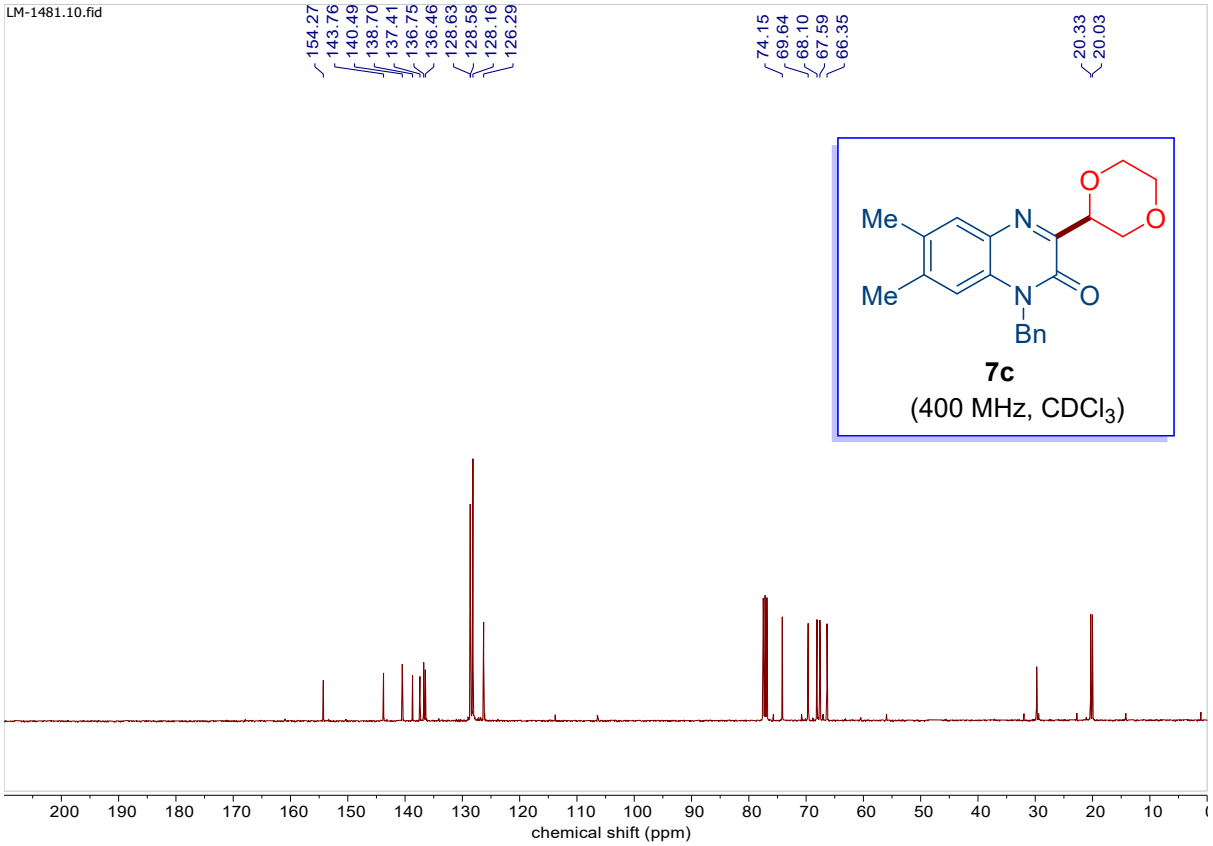
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7a.**



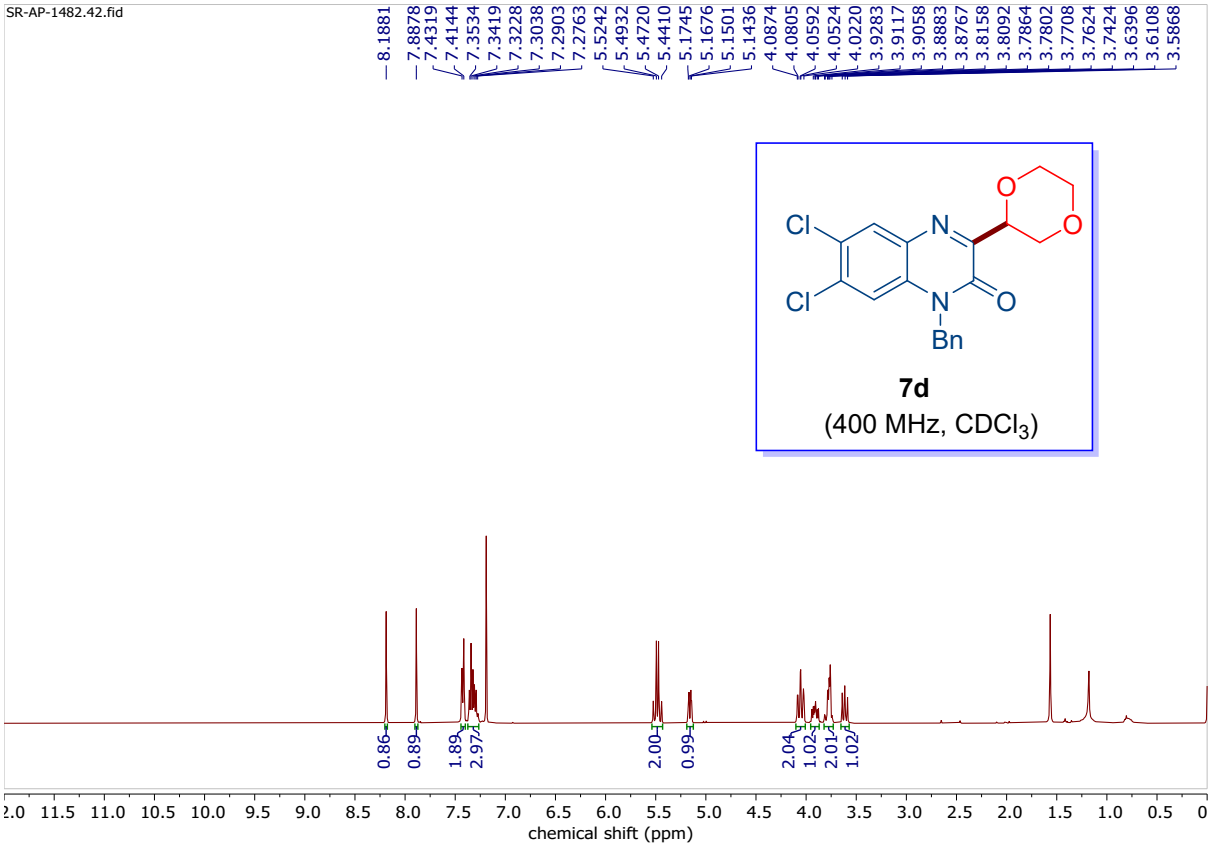


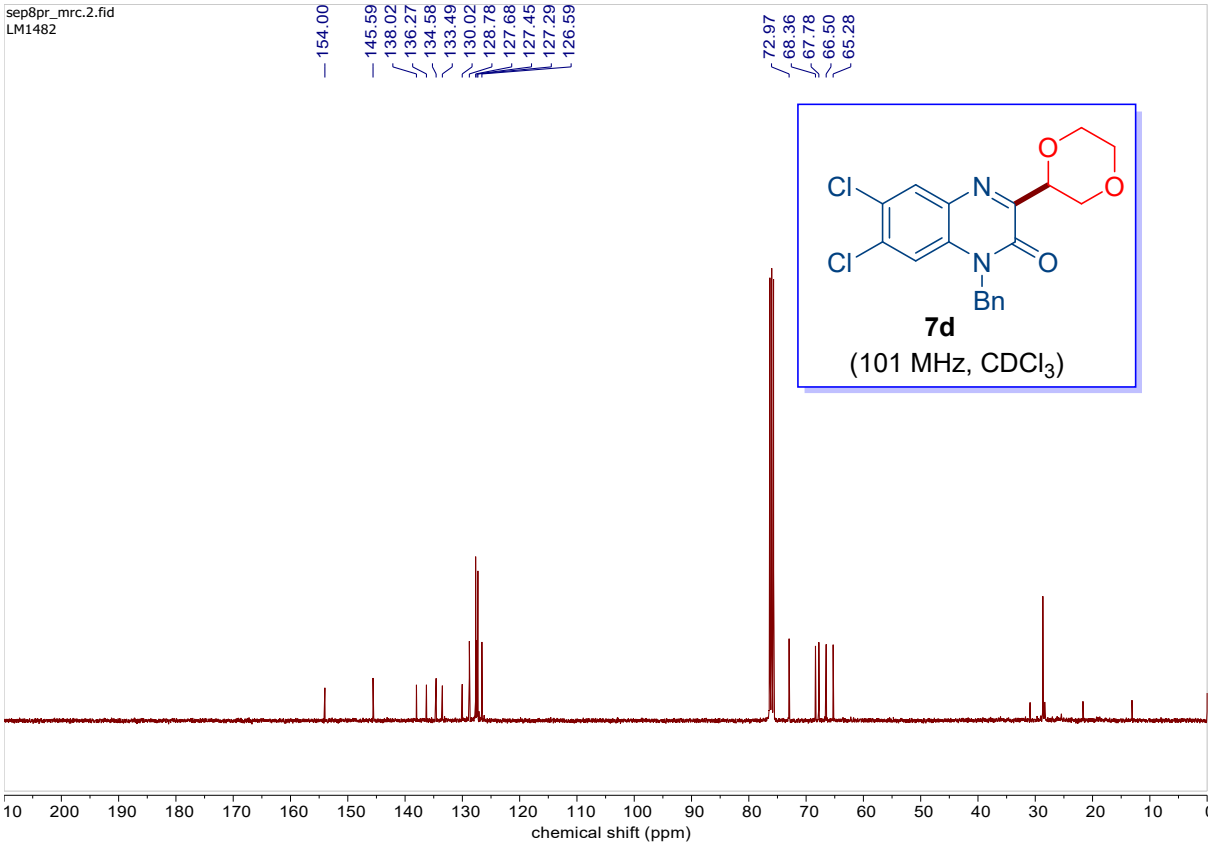
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7b.**



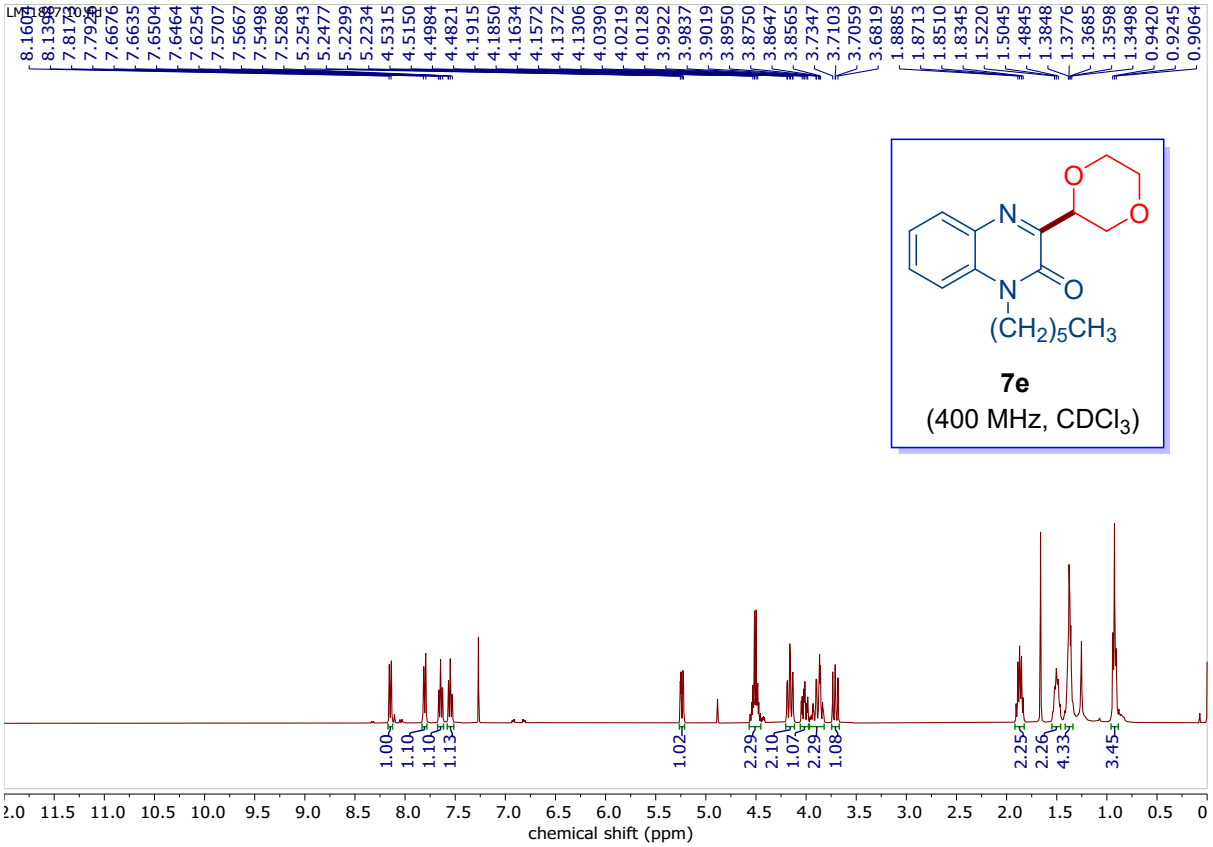


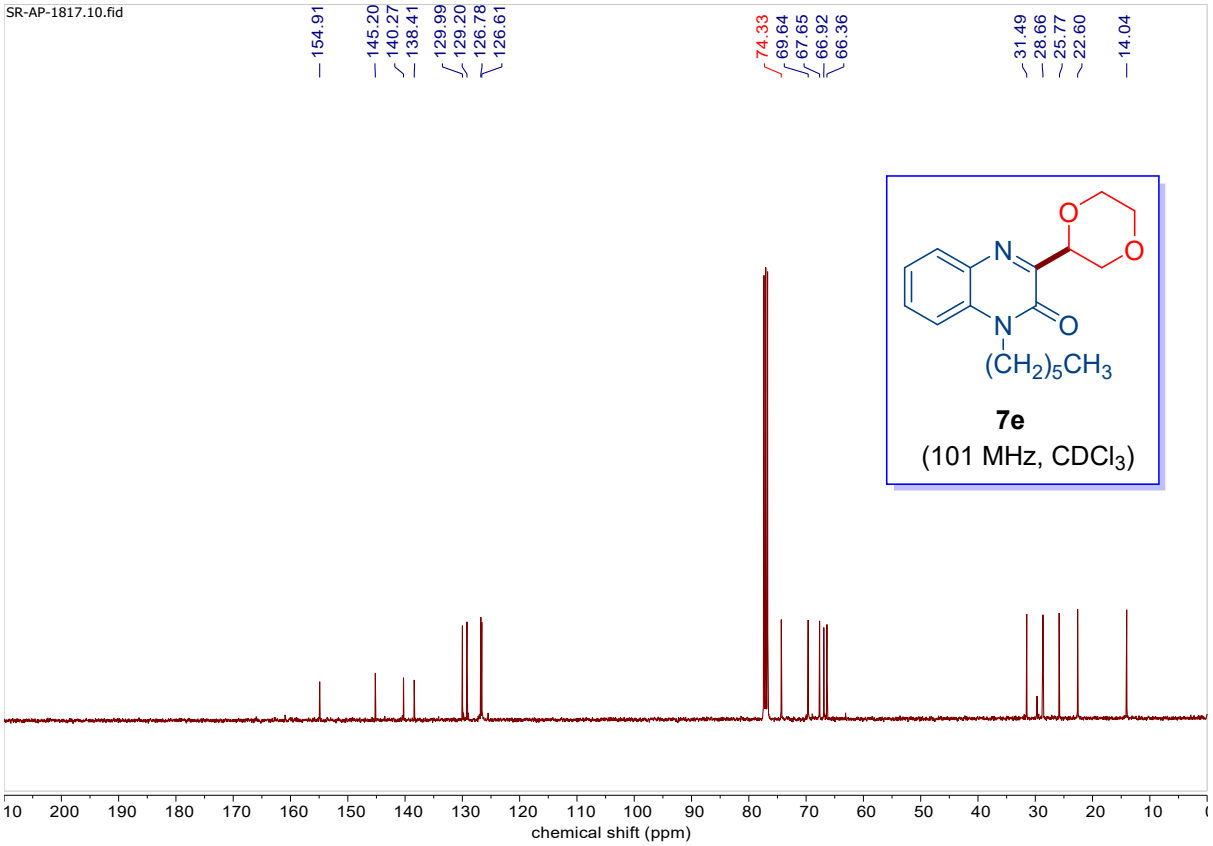
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7c**.



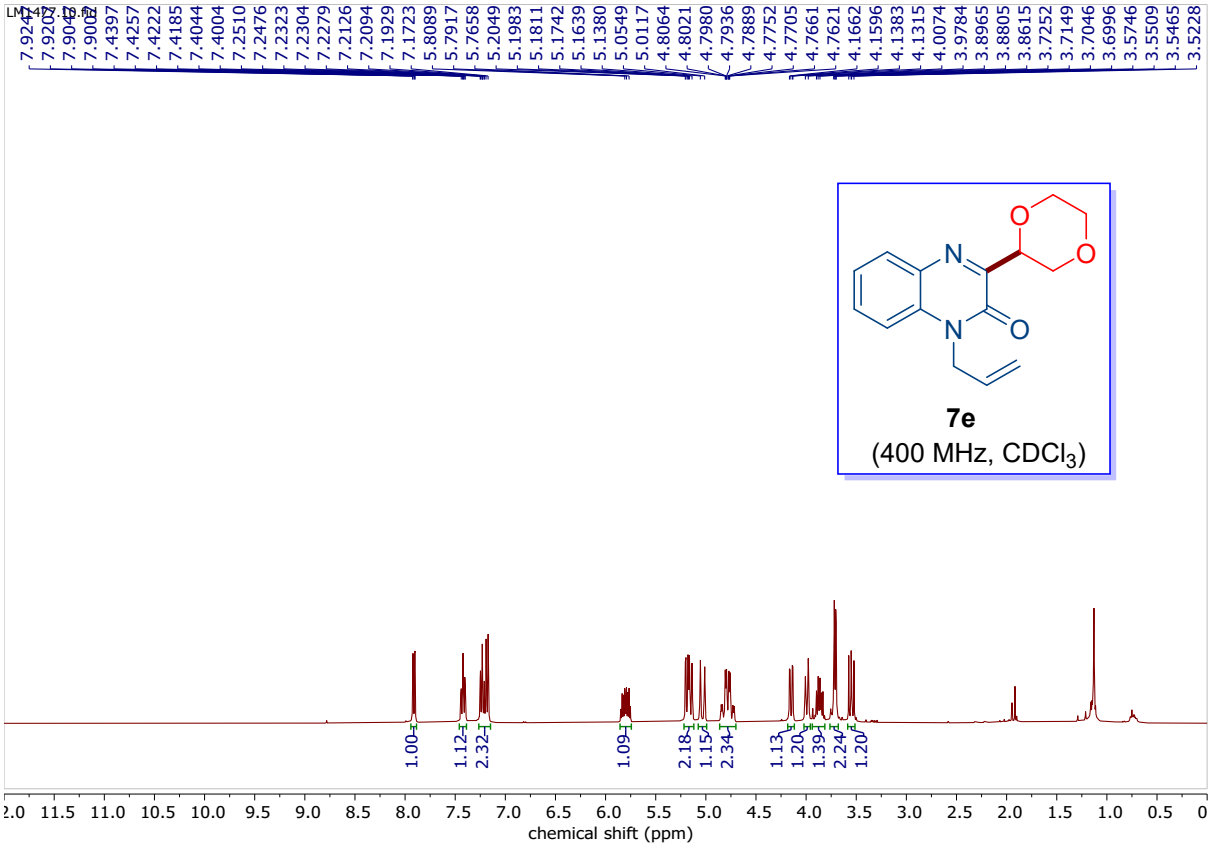


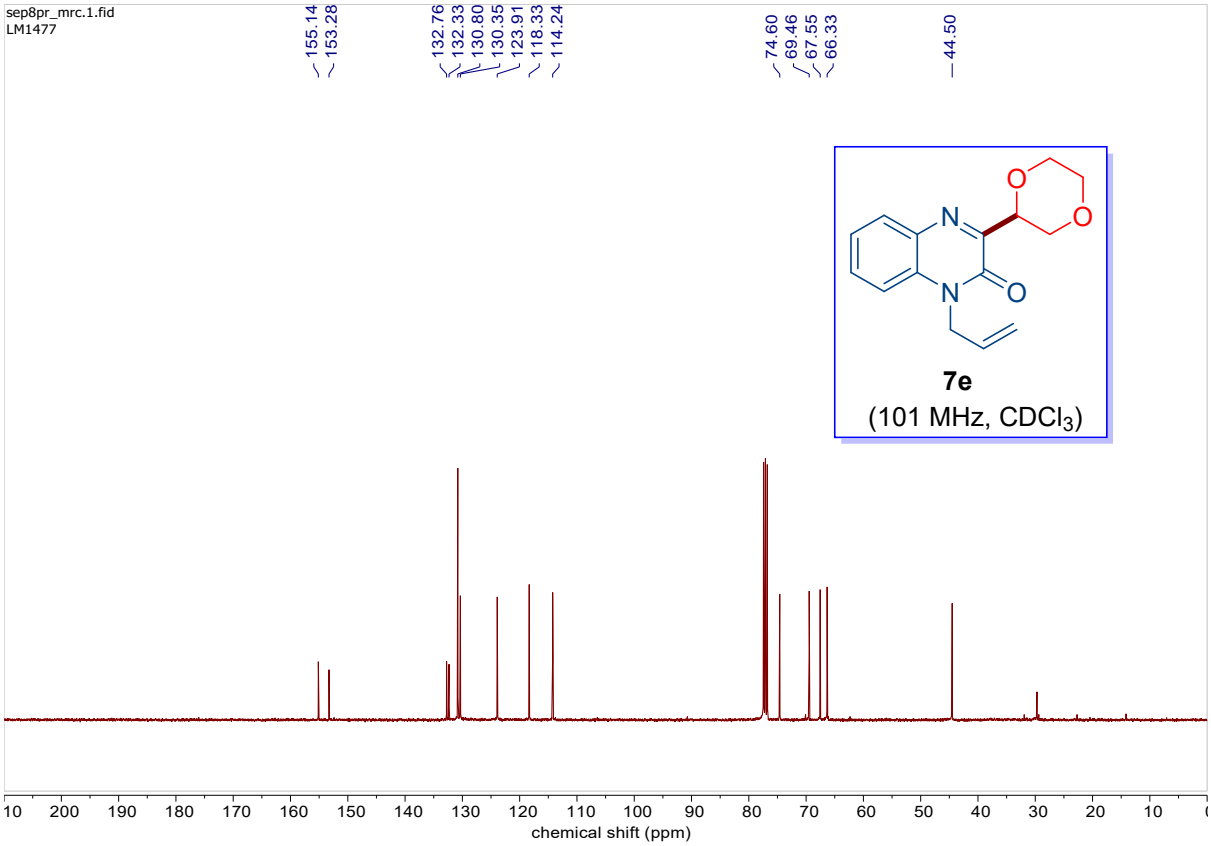
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7d**.



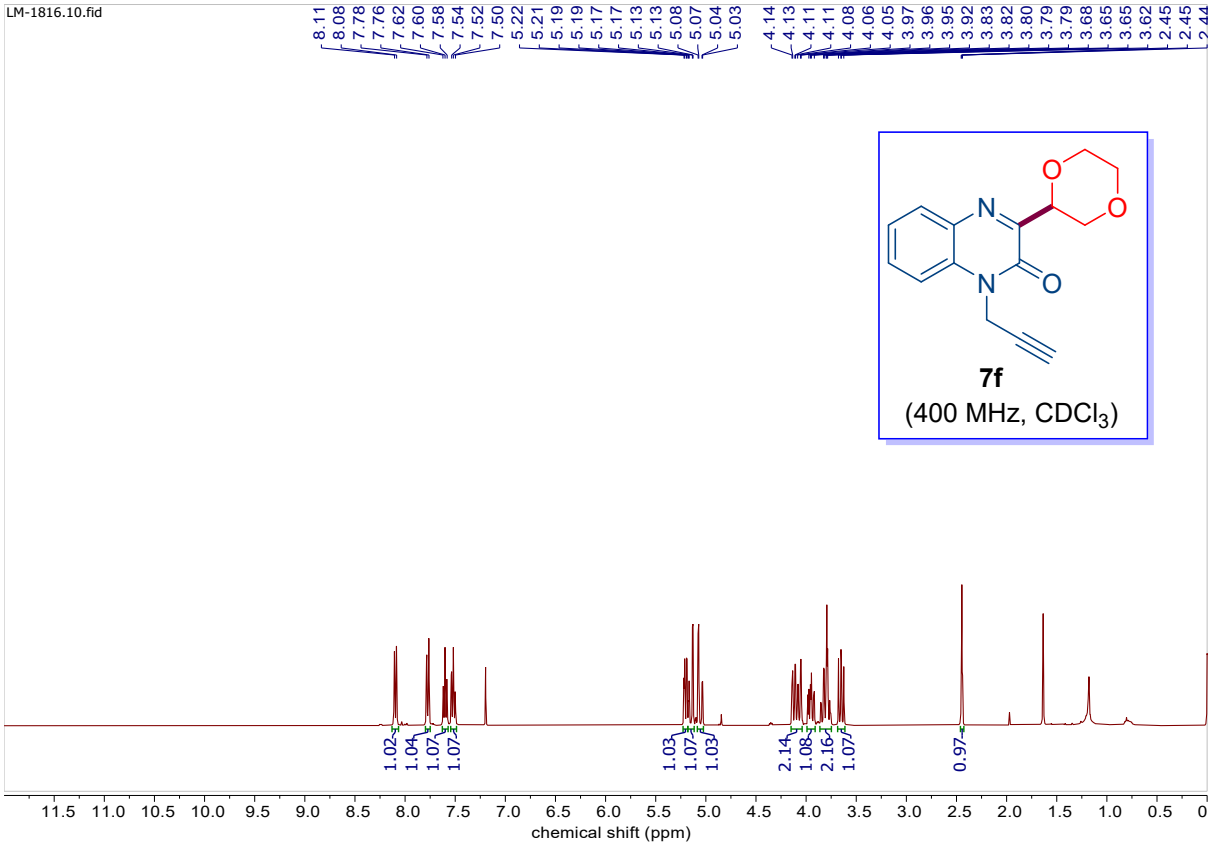


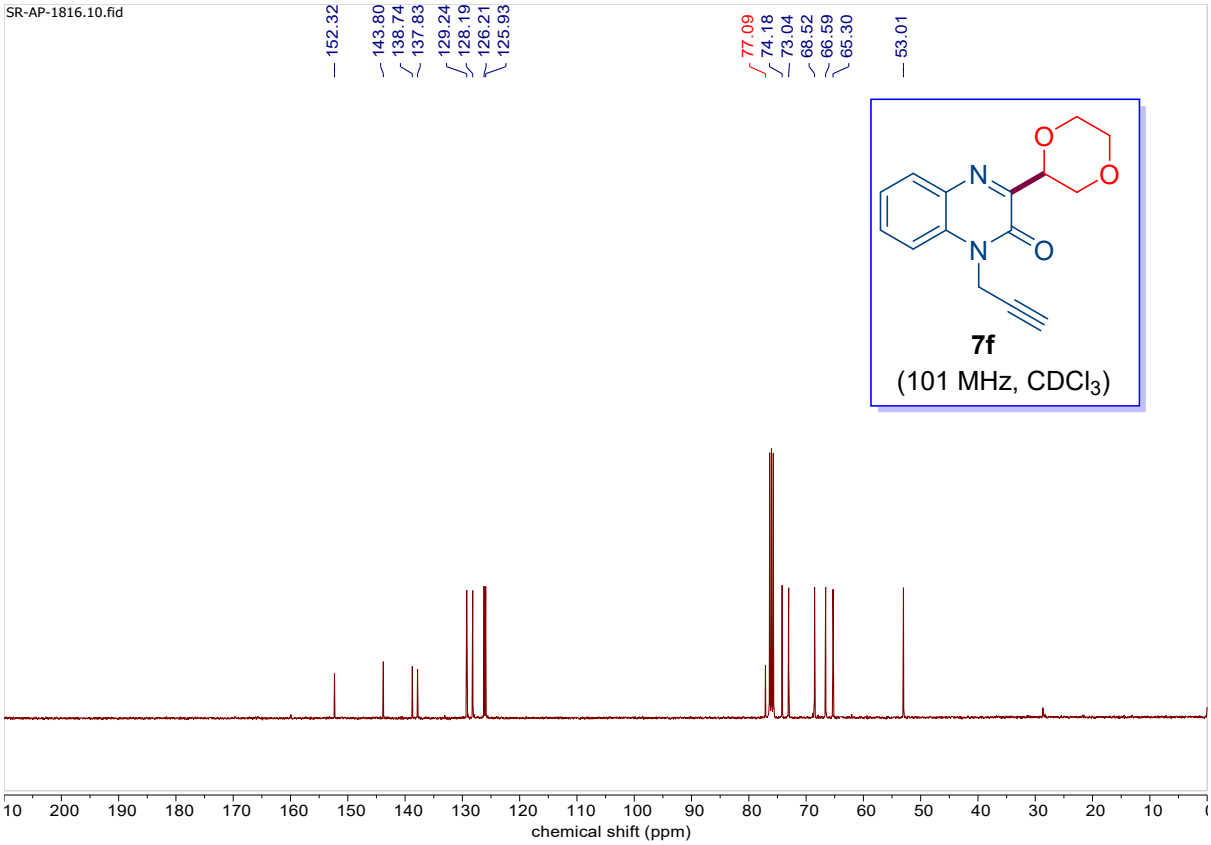
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7e.**



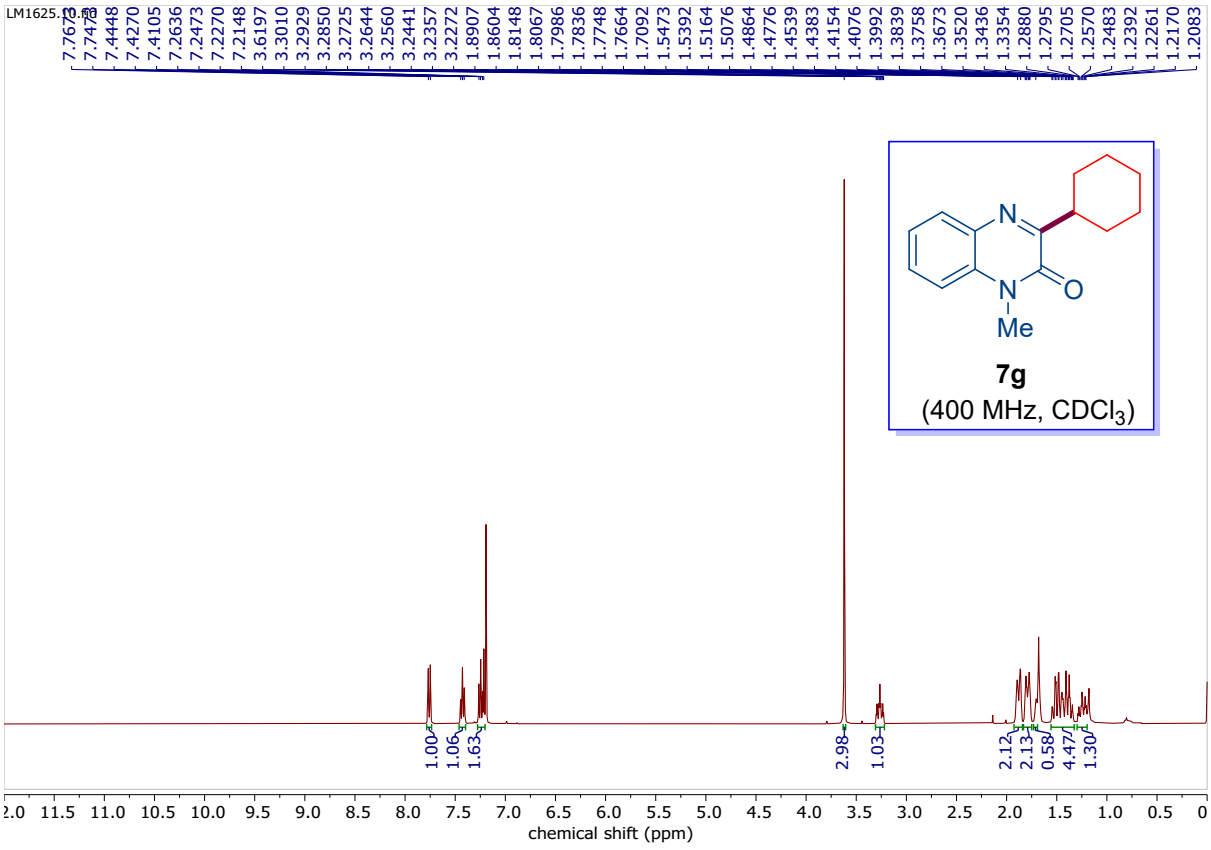


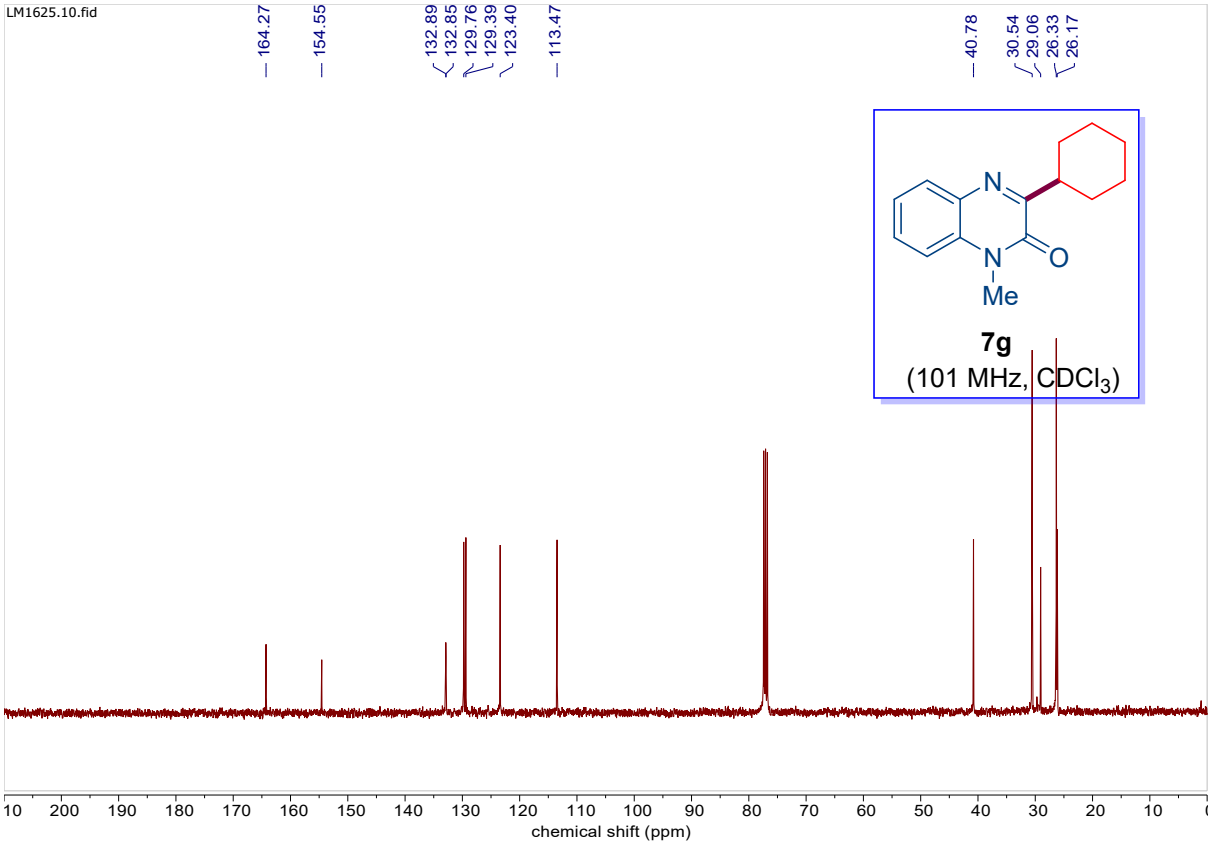
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7f.**



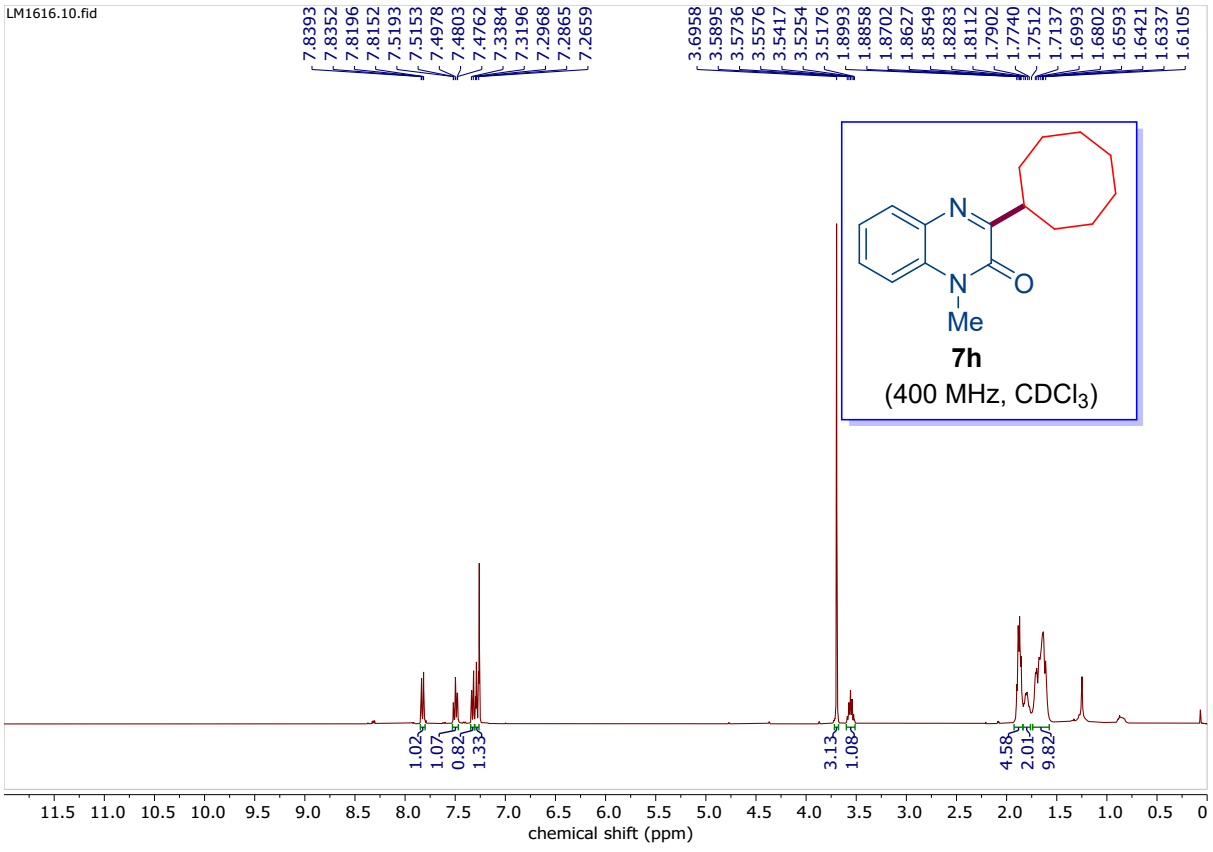


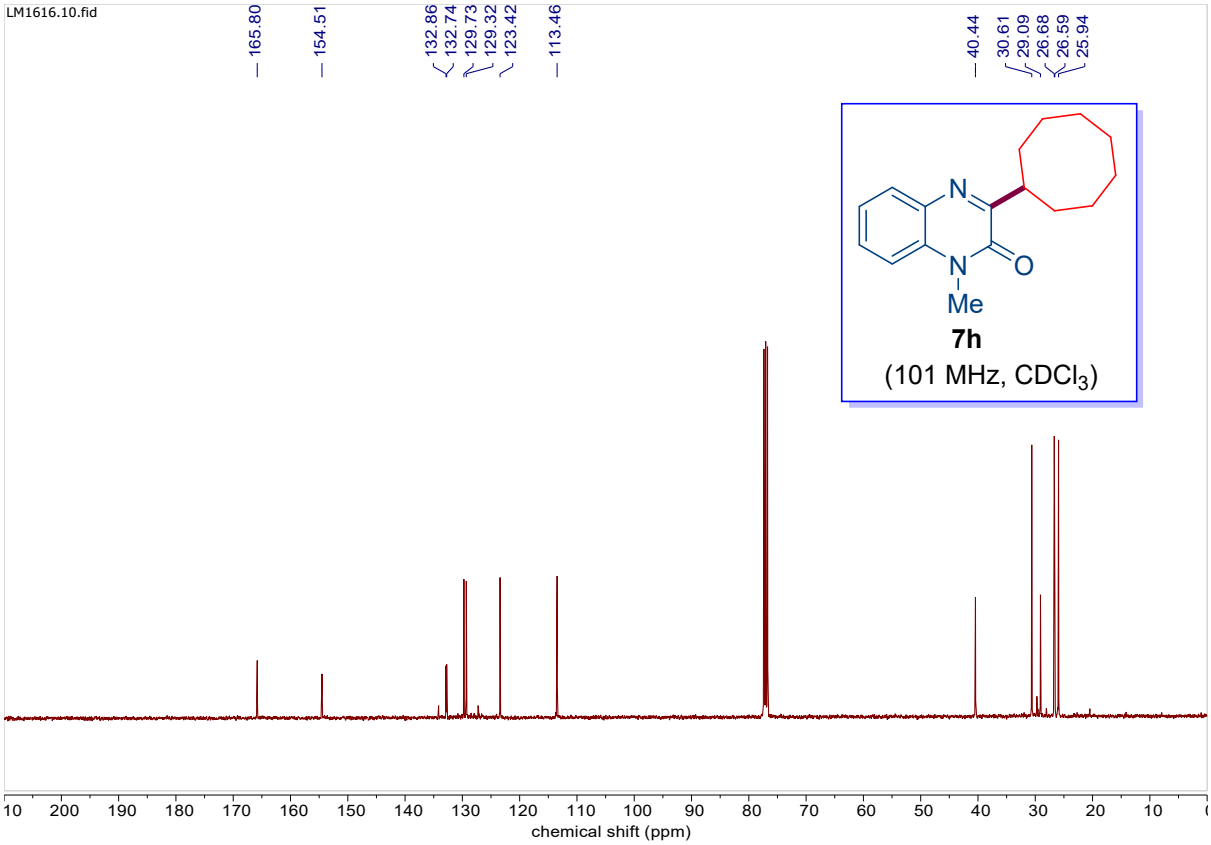
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7g.**



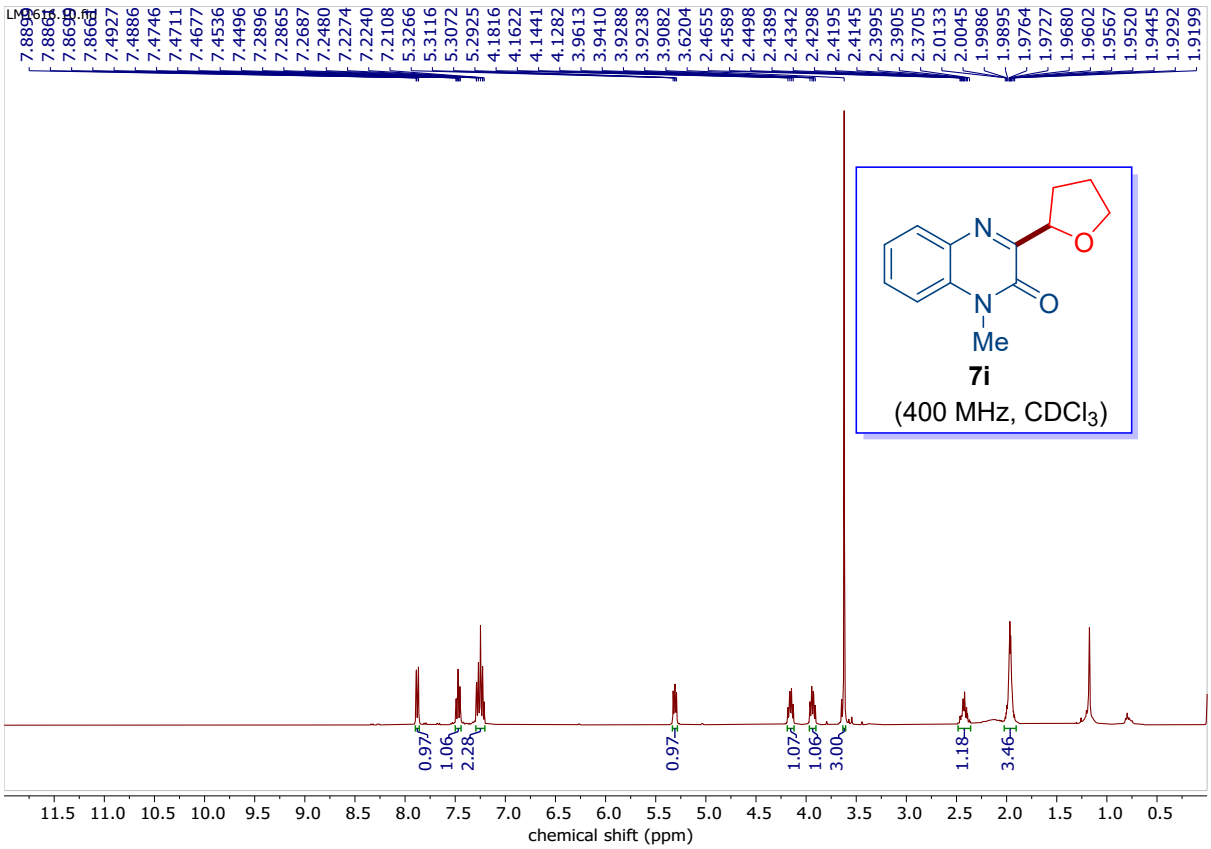


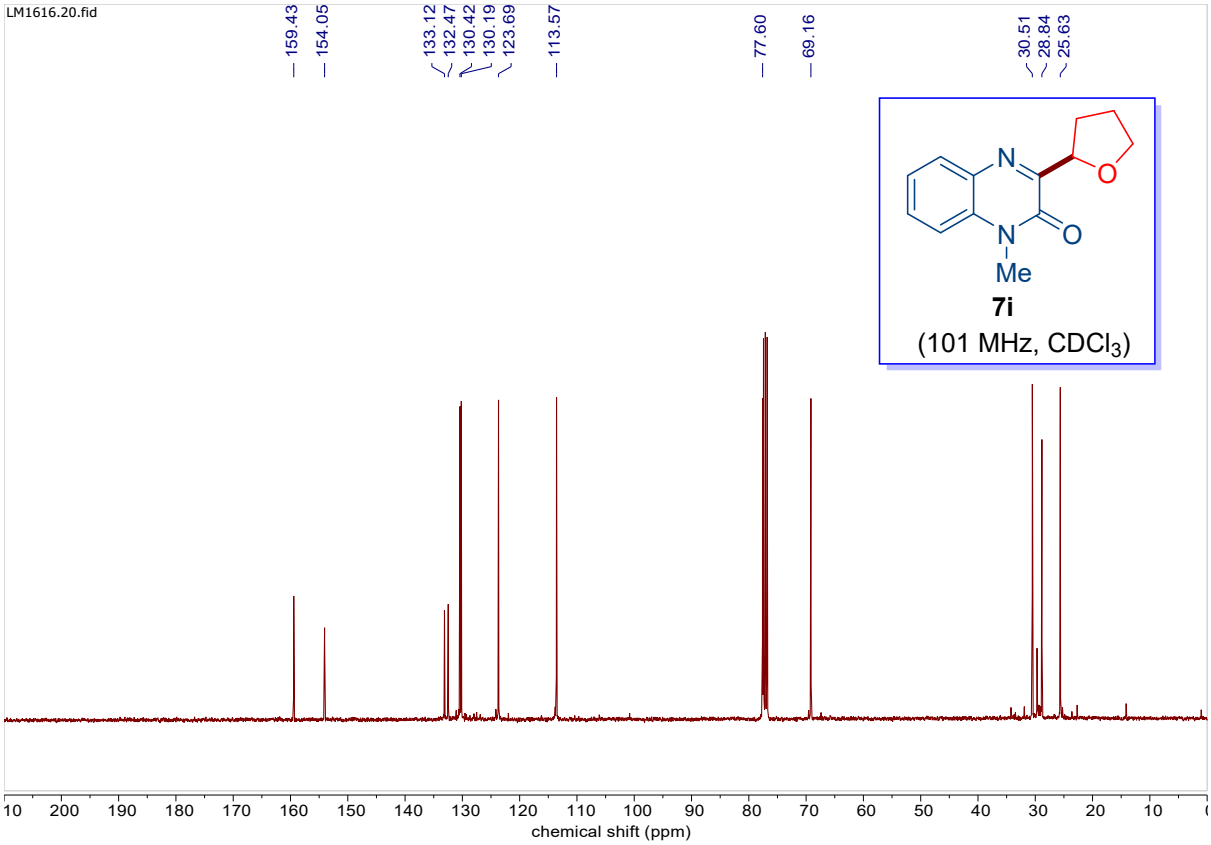
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7h.**



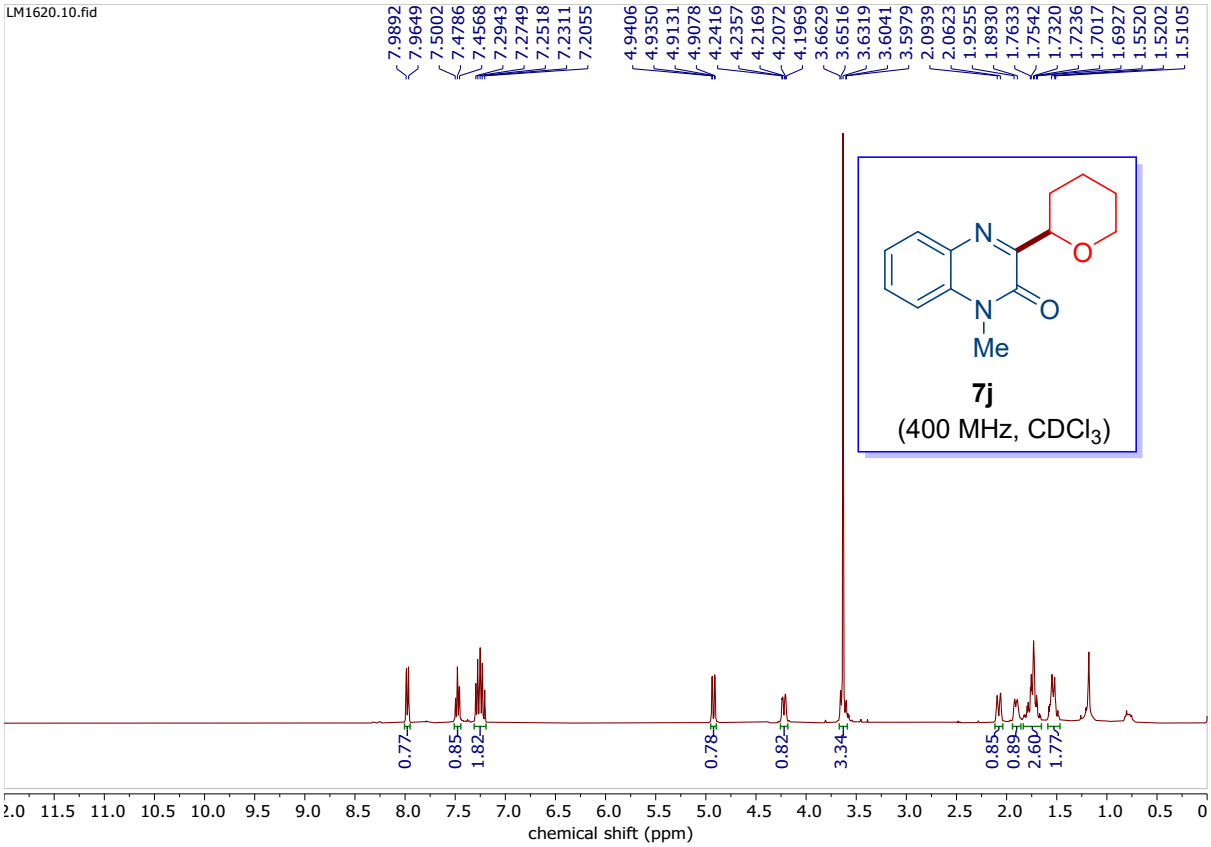


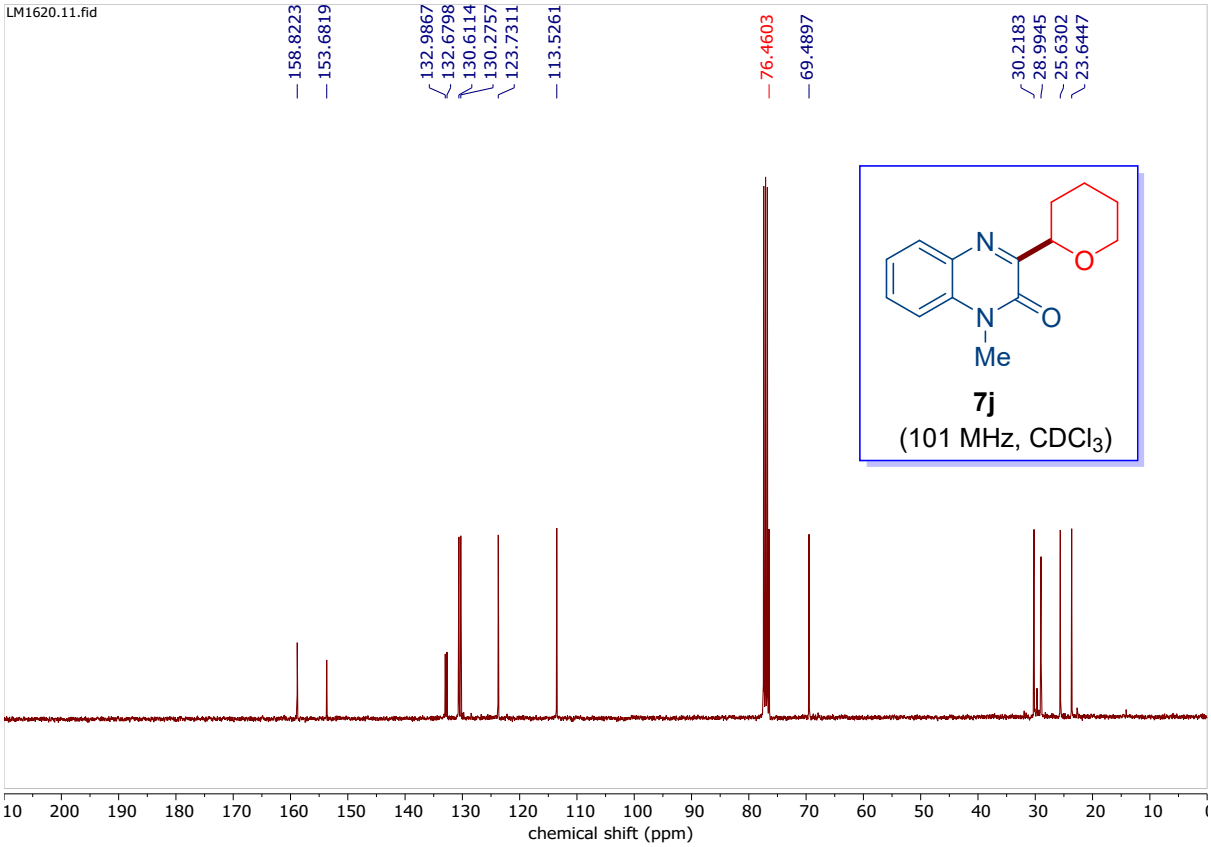
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7i.**



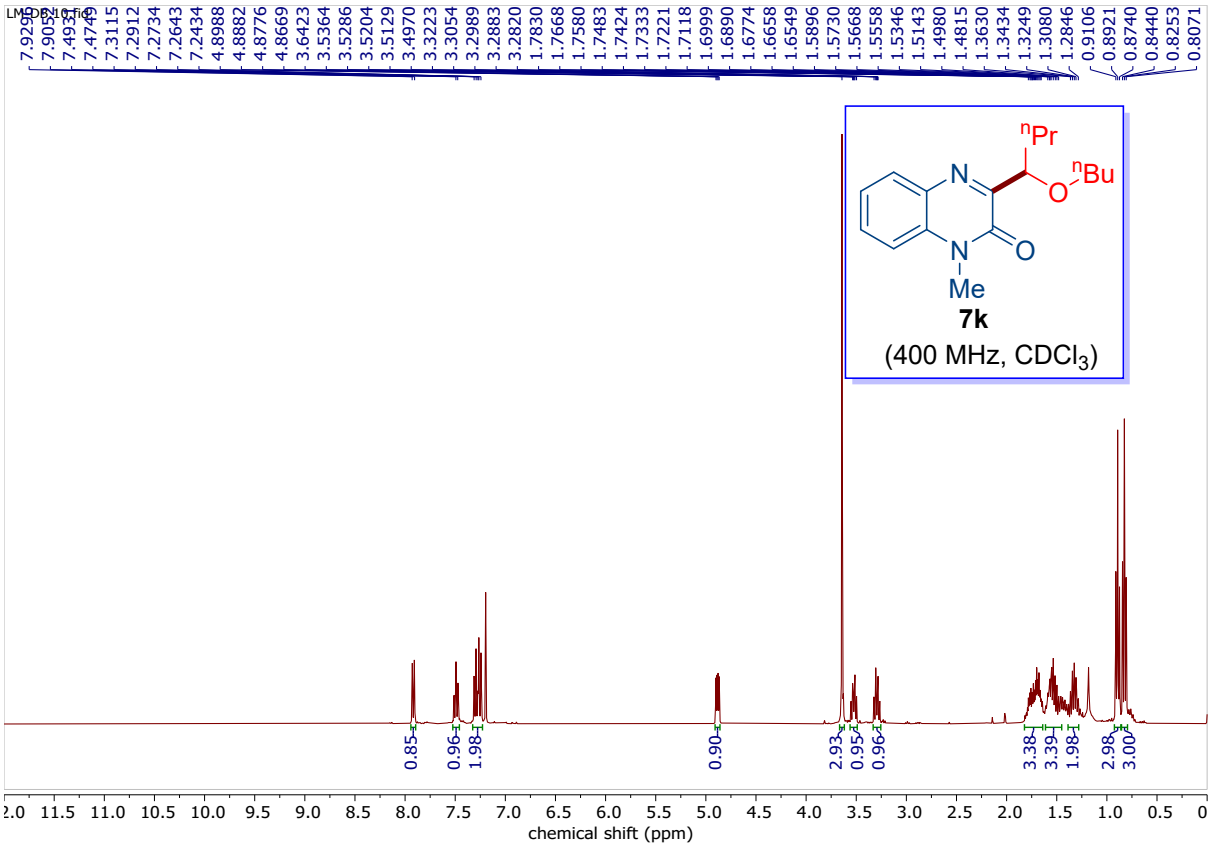


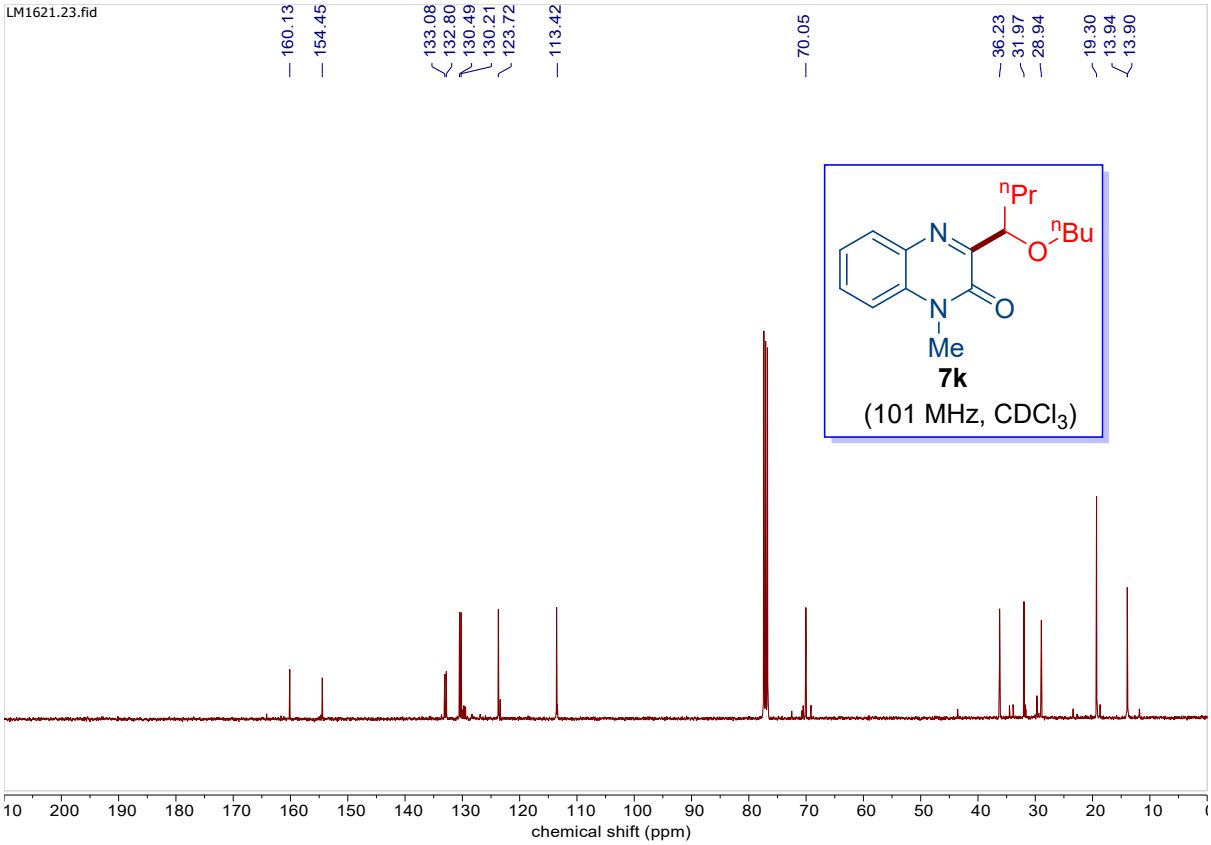
^{1}H and ^{13}C{^{1}H} NMR spectra of compound **7j.**



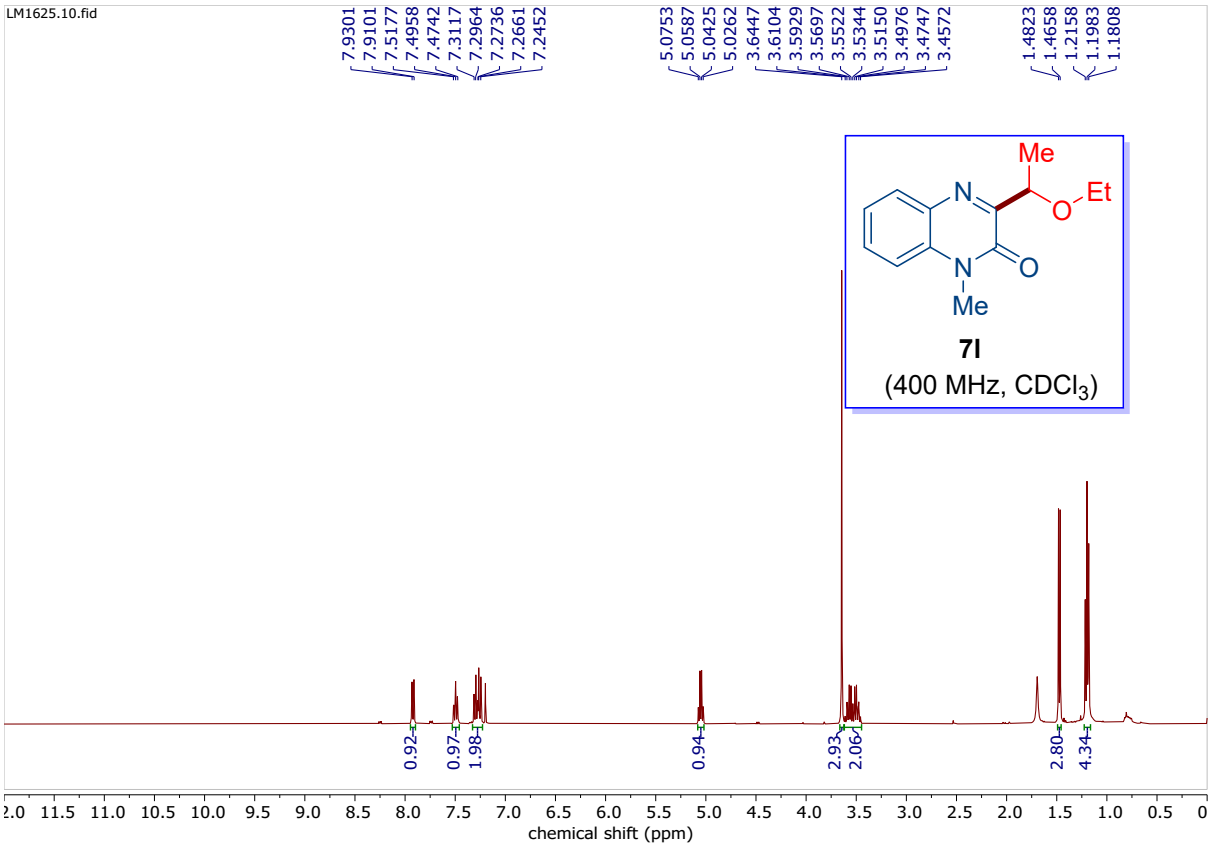


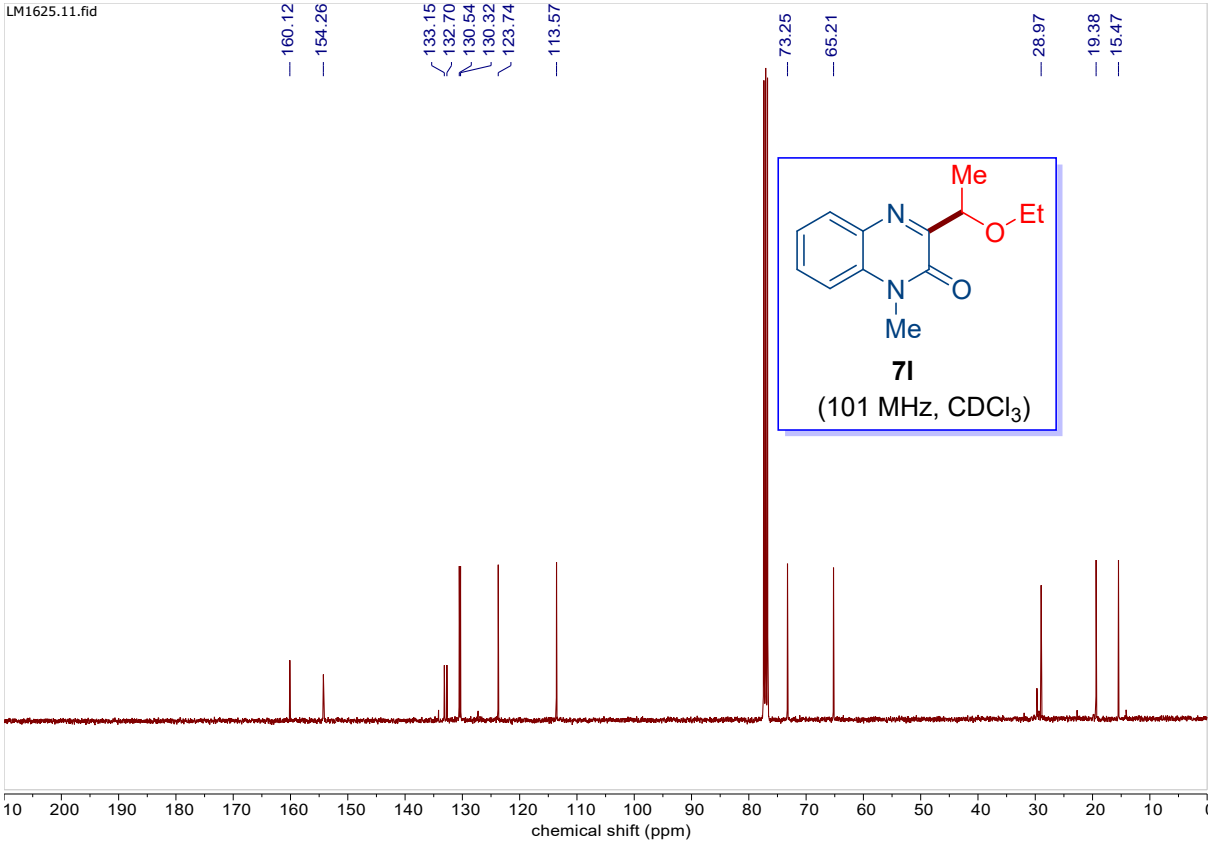
^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7k.**





^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of compound **7l**.

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7l
(101 MHz, CDCl3)
chemical shift (ppm)