Supplementary Information (SI) for Organic & Biomolecular Chemistry.

Supporting Information

for

N-Arylation of 1,2,4-oxadiazin-5(6*H*)-one derivatives by diaryliodonium salts

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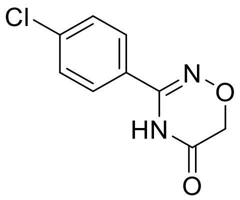
S1. General information

Starting materials were prepared according to the literature procedures: diaryliodonium salts [1-4] and oxadiazines (**1a-d, 1j-s**) [5,6]. All other reagents and solvents were purchased from Merck and used as is. Reactions were monitored by analytical thin layer chromatography (TLC) with Macherey–Nagel TLC plates Silufol UV–254 using UV light for detection. Column chromatography was carried out with silica gel grade 60 (0.040–0.063 mm) 230–400 mesh with a hexane/DCM mixture as eluent. NMR spectra were recorded with Bruker Avance DPX 400 (400 MHz, 101 MHz, and 376 MHz for ^{1}H, ^{13}C, and ^{19}F respectively) in DMSO–d_6 or in $CDCl_3$. Chemical shifts are reported as parts per million (δ, ppm); the solvent peaks were used as internal standards: 2.50 ppm for residual ^{1}H, 39.50 ppm for ^{13}C in DMSO–d_6; 7.26 ppm for residual ^{1}H, 77.16 ppm for ^{13}C in $CDCl_3$. Multiplicities are abbreviated as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet; coupling constants, *J*, are reported in Hertz (Hz). Melting points were determined in open capillary tubes with Electrothermal IA 9300 series Digital Melting Point Apparatus. High–resolution mass spectra (HRMS) were measured with Bruker Maxis–qTOF (ESI).

S2. Preparation of starting compounds 1 and 2

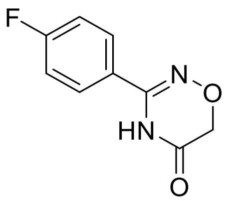
Synthesis of 1,2,4-oxadiazin-5(6*H*)-ones 1

General procedure[6]. To a solution of amidoxime **1** (2 mmol) in DMSO (3 mL) *t*-BuONa (384 mg, 4 mmol) was rapidly added at room temperature. The reaction mixture was stirred at room temperature for 10 min and ester **2** (2.4 mmol) was added. The reaction mixture was stirred at room temperature for another 18 h, then was diluted with HCl (10% solution in water) (30 mL). The resulted precipitate was filtered off, washed with cold water (5 mL), and dried in air at 50 °C.



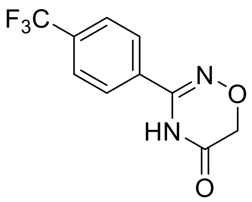
3-(4-Chlorophenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1e).

White powder; 55% yield; mp 147–149 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.75 (d, J = 8.6 Hz, 2H), 7.48 (d, J = 8.6 Hz, 2H), 4.47 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 150.0, 138.1, 129.5, 127.4, 127.0, 66.9. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_9H_8ClN_2O_2^+$ 211.0269; found 211.0270.



3-(4-Fluorophenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1f).

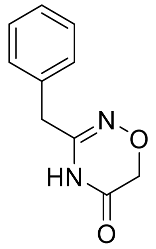
Pale pink powder; 13 % yield (after crystallization from toluene); mp 108–110 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.61 (s, 1H), 7.90–7.72 (m, 2H), 7.22–7.18 (m, 2H), 4.48 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 164.8 (d, J = 254.5 Hz), 150.2, 128.46 (d, J = 8.8 Hz), 124.65 (d, J = 3.3 Hz), 116.33 (d, J = 22.1 Hz) 66.74. ^{19}F NMR (376 MHz, $CDCl_3$) δ –107.16. HRMS (ESI), m/z: $[M–H]^–$ calcd for $C_9H_6FN_2O_2^–$ 193.0419; found 193.0420.



3-(4-(Trifluoromethyl)phenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1g).

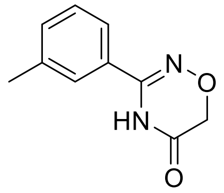
White powder; 10% yield; mp 110–112 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.96 (d, J = 8.2 Hz, 2H), 7.78 (d, J = 8.2 Hz, 2H), 4.52 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 149.6, 133.52 (q, J = 32.9 Hz), 131.89, 126.6, 126.2 (q, J = 3.7 Hz), 123.5 (q, J = 123.5 Hz), 66.8. ^{19}F

NMR (376 MHz, $CDCl_3$) δ –63.11. HRMS (ESI), m/z: $[M–H]^-$ calcd for $C_{10}H_6F_3N_2O_2^-$ 243.0387; found 243.0386.



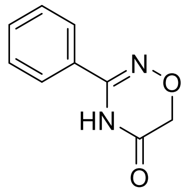
3-Benzyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1h).

White powder; 15% yield (after crystallization from toluene); mp 102–104 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.42–7.25 (m, 5H), 4.32 (s, 2H), 3.69 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 151.5, 133.3, 129.2, 128.9, 128.0, 66.6, 37.0. HRMS (ESI), m/z: $[M–H]^-$ calcd for $C_{10}H_9N_2O_2^-$ 189.0670; found 189.0664.



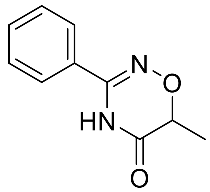
3-(3-Methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1i).

White powder; 45% yield; mp 77–79 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.53 (s, 1H), 7.62 (s, 1H), 7.56 (d, *J* = 6.8 Hz, 1H), 7.40–7.35 (m, 2H), 4.45 (s, 2H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 151.1, 139.1, 132.6, 129.0, 128.4, 126.8, 123.1, 66.9, 21.4. HRMS (ESI), m/z: $[M–H]^-$ calcd for $C_{10}H_9N_2O_2^-$ 189.0670; found 189.0670.



3-Phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1o).

White powder; 30% yield; mp 128–130 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.65 (s, 1H), 7.82–7.76 (m, 2H), 7.59–7.46 (m, 3H), 4.46 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 150.9, 131.8, 129.1, 128.5, 126.2, 66.9. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_9H_9N_2O_2^+$ 177.0659; found 177.0658.



6-Methyl-3-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (1p).

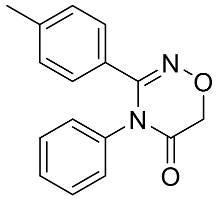
White crystals; 53% yield; mp 125–127 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.40 (s, 1H), 7.83–7.74 (m, 2H), 7.58–7.46 (m, 3H), 4.43–4.37 (m, 1H), 1.62–1.55 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 150.8, 131.6, 129.1, 128.7, 126.1, 72.6, 13.4. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{10}H_{11}N_2O_2^+$ 191.0815; found 191.0814.

Synthesis of diaryliodonium salts 2

Diaryliodonium triflates **2a–c** were synthesized from the arene and iodoarene according to the literature procedure employing Oxone as an oxidant.[35] Aryl(2,4,6-trimethoxyphenyl)iodonium trifluoroacetates **2d–i**,**k–m** were synthesized from the iodoarene according to the literature procedure employing Oxone as an oxidant.[35] (3-(Trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium trifluoroacetate **2j** was synthesized from the 3-iodobenzotriluoride according to the literature procedure employing *m*CPBA as an oxidant.[42] The analytical data are in accordance with previously reported.[35,42]

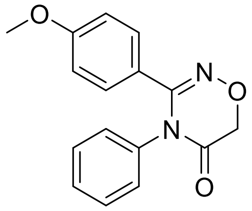
S3. Synthesis of *N*-aryl-1,2,4-oxadiazin-5(6*H*)-ones 3-4

General procedure. To a solution of 1,2,4-oxadiazin-5(6*H*)-one **1** (0.2 mmol) and DIPEA (0.3 mmol, 52 μL) in toluene (2.1 mL) diaryliodonium salt **2** (0.3 mmol) and CuI (10 mol%, 4 mg) were added under argon atmosphere. The resulted mixture was heated at 60 °C for 24 hours (compound **4i** were prepared at 100 °C). Then the solvent was removed under reduced pressure and the product was purified by silica gel column chromatography (gradient hexane:DCM = 2:1 → hexane:DCM = 1:12).



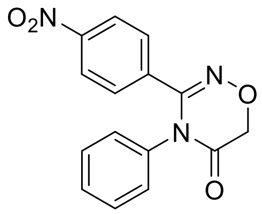
4-Phenyl-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3a).

White powder; 82% yield; mp 99–101 °C. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.30–7.17 (m, 5H), 7.12 (d, *J* = 8.1 Hz, 2H), 6.99 (d, *J* = 8.2 Hz, 2H), 4.62 (s, 2H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 164.5, 155.7, 140.9, 135.4, 129.3, 129.2, 129.0, 128.3, 128.2, 126.5, 68.7, 21.6. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{15}N_2O_2^+$ 267.1128; found 267.1125.



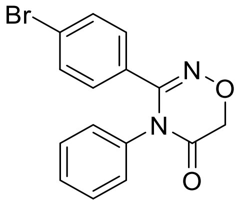
3-(4-Methoxyphenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3b).

White powder; 47% yield; mp 153–155 °C. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.34–7.28 (m, 4H), 7.27–7.21 (m, 1H), 7.18–7.12 (m, 2H), 6.76–6.71 (m, 2H), 4.65 (s, 2H), 3.76 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 164.4, 161.1, 155.3, 135.3, 130.4, 129.0, 128.0, 128.0, 121.3, 113.8, 68.5, 55.2. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{14}N_2O_3^+$ 283.1077; found 283.1077.



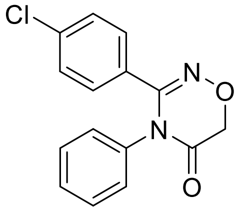
3-(4-Nitrophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3c).

Pale yellow powder; 51% yield; mp 201–203 °C. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.10 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.36–7.26 (m, 3H), 7.15 (d, *J* = 7.7 Hz, 2H), 4.71 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 163.3, 153.6, 148.6, 135.4, 134.4, 129.9, 129.5, 128.8, 127.8, 123.6, 68.6. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{15}H_{12}N_3O_4^+$ 298.0822; found 298.0822.



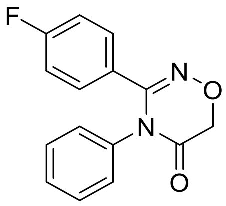
3-(4-Bromophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3d).

White powder; 72% yield; mp 148–150 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, J = 8.6 Hz, 2H), 7.35–7.23 (m, 5H), 7.14 (d, J = 7.4 Hz, 2H), 4.66 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.9, 154.6, 134.8, 131.7, 130.3, 129.2, 128.4, 128.2, 127.9, 125.0, 68.5. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{15}H_{12}BrN_2O_2^+$ 331.0077; found 331.0075.



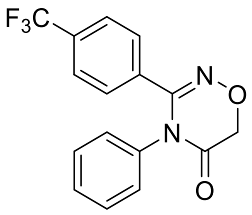
3-(4-Chlorophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3e).

White powder; 52% yield; mp 142–144 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, J = 7.0, 1.5 Hz, 2H), 7.33–7.21 (m, 5H), 7.18–7.10 (m, 2H), 4.68 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.9, 154.5, 136.6, 134.9, 130.1, 129.2, 128.7, 128.3, 127.9, 127.7, 68.5. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{15}H_{11}ClN_2O_2^+$ 287.0582; found 287.0581.



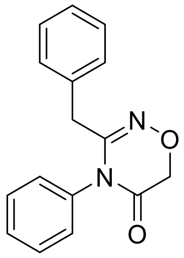
3-(4-Fluorophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3f).

White powder; 56% yield; mp 133–135 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.36 (m, 2H), 7.35–7.23 (m, 3H), 7.17–7.11 (m, 2H), 6.97–6.89 (m, 2H), 4.67 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.0, 163.6 (d, J = 253 Hz), 154.6, 135.0, 131.0 (d, J = 8.7 Hz), 129.1, 128.3, 128.0, 125.4 (d, J =3.0 Hz), 115.6 (d, J = 22.1 Hz), 68.4. ^{19}F NMR (376 MHz, $CDCl_3$) δ –108.72. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{15}H_{11}FN_2O_2^+$ 271.0877; found 271.0874.



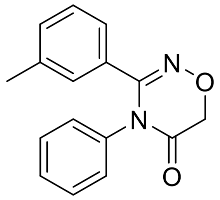
4-Phenyl-3-(4-(trifluoromethyl)phenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3g).

White powder; 48% yield; mp 78–80 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.46 (m, 4H), 7.37–7.22 (m, 3H), 7.15 (d, J = 7.3 Hz, 2H), 4.69 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.7, 134.6, 132.9, 132.2 (q, J = 33.1 Hz), 129.3 (d, J = 6.5 Hz), 128.5, 127.8, 127.5, 125.4, 125.3 (q, J = 3.7 Hz), 123.5 (q, J = 272.6 Hz), 68.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ –63.10. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{12}F_3N_2O_2^+$ 321.0845; found 321.0840.



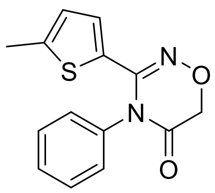
3-Benzyl-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3h).

White powder; 46% yield; mp 124–126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.33 (m, 3H), 7.26–7.16 (m, 3H), 6.93–6.88 (m, 2H), 6.87–6.81 (m, 2H), 4.55 (s, 2H), 3.57 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 154.2, 134.0, 133.7, 129.2, 128.9 (1CH+2CH), 128.6, 128.4, 127.4, 67.8, 36.5. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{15}N_2O_2^+$ 267.1128; found 267.1128.



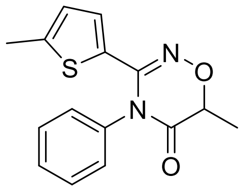
4-Phenyl-3-(3-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3i).

White powder; 55% yield; mp 112–114 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34–7.21 (m, 4H), 7.18–7.13 (m, 2H), 7.12–7.08 (m, 3H), 4.66 (s, 2H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 155.5, 138.2, 135.2, 131.2, 129.5, 129.1, 129.0, 128.1, 128.1, 128.0, 126.0, 68.4, 21.2. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{15}N_2O_2^+$ 267.1128; found 267.1122.



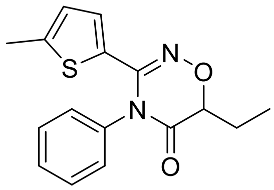
3-(5-Methylthiophen-2-yl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3j).

White powder; 77% yield; mp 138–140 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46–7.34 (m, 3H), 7.28–7.24 (m, 2H), 6.55–6.45 (m, 2H), 4.62 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 150.9, 144.6, 135.3, 131.3, 129.2, 128.7, 128.2, 127.8, 125.7, 68.5, 15.3. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{14}H_{13}N_2O_2S^+$ 273.0692; found 273.0692.



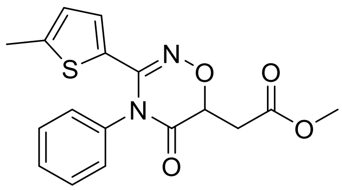
6-Methyl-3-(5-methylthiophen-2-yl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3k).

White powder; 76% yield; mp 172–173 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.34 (m, 3H), 7.25 (d, J = 7.0 Hz, 2H), 6.48 (q, J = 4.1 Hz, 2H), 4.52 (q, J = 6.7 Hz, 1H), 2.40 (d, J = 0.9 Hz, 3H), 1.64 (d, J = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 150.7, 144.3, 135.9, 131.0, 129.2, 128.6, 128.2, 128.2, 125.6, 73.9, 15.3, 13.6. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{15}H_{15}N_2O_2S^+$ 287.0849; found 287.0849.



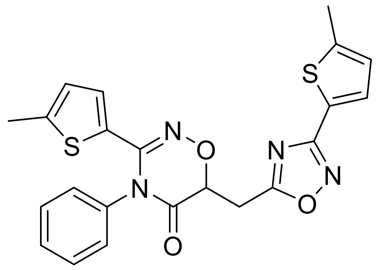
6-ethyl-3-(5-methylthiophen-2-yl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3l).

White powder; 92% yield; mp 125–127 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.33 (m, 3H), 7.25 (d, J = 7.0 Hz, 2H), 6.47 (s, 2H), 4.35 (dd, J = 8.2, 4.7 Hz, 1H), 2.40 (s, 3H), 2.20–2.10 (m, 1H), 2.02–1.92 (m, 1H), 1.19 (t, J = 7.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.0, 150.6, 144.3, 135.8, 130.9, 129.2, 128.6, 128.3 (C+2CH), 125.6, 78.6, 21.5, 15.3, 9.7. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{17}N_2O_2S^+$ 301.1005; found 301.1005.



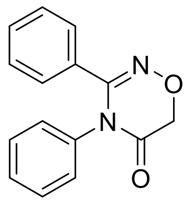
Methyl 2-(3-(5-methylthiophen-2-yl)-5-oxo-4-phenyl-5,6-dihydro-4*H*-1,2,4-oxadiazin-6-yl)acetate (3m).

White powder; 76% yield; mp 126–128 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (p, J = 6.5 Hz, 3H), 7.30–7.21 (m, 2H), 6.55–6.43 (m, 2H), 4.95–4.89 (m, 1H), 3.78 (s, 3H), 3.19 (dd, J = 16.7, 5.7 Hz, 1H), 2.92 (dd, J = 16.7, 7.1 Hz, 1H), 2.40 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 165.2, 151.0, 145.7, 135.6, 131.3, 129.2, 128.7, 128.1, 127.8, 125.7, 74.0, 52.3, 33.5, 15.3. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{17}H_{17}N_2O_4S^+$ 345.0904; found 345.0897.



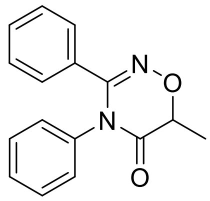
3-(5-Methylthiophen-2-yl)-6-((3-(5-methylthiophen-2-yl)-1,2,4-oxadiazol-5-yl)methyl)-4-phenyl-4H-1,2,4-oxadiazin-5(6H)-one (3n).

White powder; 67% yield; mp 174–176 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, J = 3.6 Hz, 1H), 7.47–7.35 (m, 3H), 7.28 (d, J = 7.2 Hz, 2H), 6.82 (d, J = 2.7 Hz, 1H), 6.53 (d, J = 3.7 Hz, 1H), 6.48 (d, J = 3.7 Hz, 1H), 5.10 (dd, J = 8.1, 4.9 Hz, 1H), 3.82 (dd, J = 16.4, 4.9 Hz, 1H), 3.50 (dd, J = 16.4, 8.1 Hz, 1H), 2.56 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.2, 164.6, 164.4, 151.2, 145.0, 144.6, 135.5, 131.5, 130.1, 129.3, 128.9, 128.1, 127.5, 126.3, 125.8, 125.6, 74.5, 26.5, 15.5, 15.3. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{22}H_{19}N_4O_3S_2^+$ 451.0893; found 451.0893.



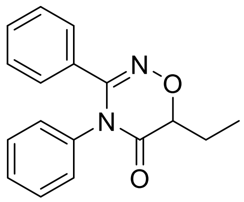
3,4-Diphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3o).

White powder; 97% yield; mp 129–131 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, J = 7.0, 1.5 Hz, 2H), 7.35–7.19 (m, 6H), 7.18–7.11 (m, 2H), 4.68 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 155.4, 135.1, 130.4, 129.2, 129.0, 128.9, 128.3, 128.1, 128.0, 68.5. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{15}H_{13}N_2O_2^+$ 253.0972; found 253.0973.



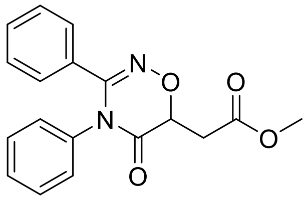
6-Methyl-3,4-diphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3p).

White powder; 53% yield; mp 128–130 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.36 (m, 2H), 7.33–7.18 (m, 6H), 7.14 (d, J = 7.4 Hz, 2H), 4.59 (q, J = 6.7 Hz, 1H), 1.68 (d, J = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 155.2, 135.6, 130.2, 129.6, 129.0, 128.8, 128.3, 128.0, 128.0, 73.8, 13.6. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{15}N_2O_2^+$ 267.1128; found 267.1128.



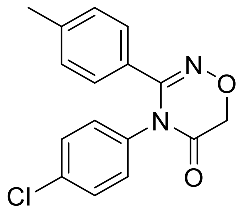
6-Ethyl-3,4-diphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (3q).

White powder; 68% yield; mp 79–80 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, J = 7.1 Hz, 2H), 7.33–7.18 (m, 6H), 7.14 (d, J = 7.4 Hz, 2H), 4.42 (dd, J = 8.1, 4.7 Hz, 1H), 2.25–2.10 (m, 1H), 1.96–2.07 (m, 1H), 1.22 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 155.0, 135.5, 130.2, 129.6, 128.9, 128.7, 128.3, 128.1, 128.0, 78.5, 21.6, 9.7. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{17}H_{17}N_2O_2^+$ 281.1285; found 281.1281.



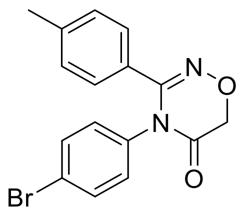
Methyl 2-(5-oxo-3,4-diphenyl-5,6-dihydro-4H-1,2,4-oxadiazin-6-yl)acetate (3r).

White powder; 75% yield; mp 91–93 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.36 (m, 2H), 7.33–7.20 (m, 6H), 7.14 (d, J = 7.4 Hz, 2H), 4.99 (dd, J = 6.9, 5.9 Hz, 1H), 3.80 (s, 3H), 3.23 (dd, J = 16.7, 5.8 Hz, 1H), 2.96 (dd, J = 16.7, 7.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 165.2, 155.5, 135.4, 130.4, 129.2, 129.0, 128.8, 128.4, 128.2, 128.0, 74.0, 52.3, 33.5. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{18}H_{17}N_2O_4^+$ 325.1183; found 325.1173.



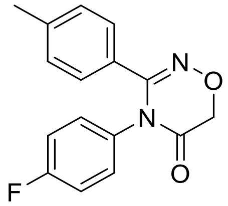
4-(4-Chlorophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4a).

White powder; 87% yield; mp 136–138 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.7 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 7.07 (d, J = 8.0 Hz, 2H), 7.05–7.01 (m, 2H), 4.64 (s, 2H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 155.1, 141.1, 134.2, 132.2, 129.4, 129.3, 128.7, 125.9, 121.9, 68.4, 21.4. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{14}ClN_2O_2^+$ 301.0738; found 301.0738.



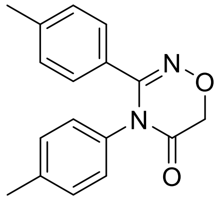
4-(4-Bromophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4b).

White powder; 85% yield; mp 138–140 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.23 (m, 4H), 7.14–7.02 (m, 4H), 4.65 (s, 2H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 155.1, 141.1, 134.2, 132.2, 129.5, 129.3, 128.7, 125.9, 121.9, 68.4, 21.4. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{14}BrN_2O_2^+$ 345.0233; found 345.0231.



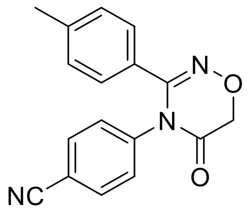
4-(4-Fluorophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4c).

Brown crystals; 76% yield; mp 50–51 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.2 Hz, 2H), 7.14 (dd, *J* = 4.8, 2.1 Hz, 2H), 7.06 (d, *J* = 8.1 Hz, 2H), 7.03–6.95 (m, 2H), 4.65 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.4, 161.7 (d, *J* = 248.9 Hz), 155.3, 140.9, 131.1 (d, *J* = 3.4 Hz), 129.7 (d, *J* = 8.8 Hz), 129.2, 128.8, 126.0, 116.0 (d, *J* = 23.0 H), 68.4, 21.4. ^{19}F NMR (376 MHz, $CDCl_3$) δ –112.55. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{14}FN_2O_2^+$ 357.1445; found 357.1438.



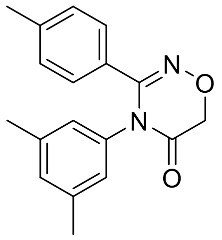
3,4-Di-4-methylphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4d).

White powder; 61% yield; mp 104–105 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.7 Hz, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.07–6.99 (m, 4H), 4.64 (s, 2H), 2.29 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.4, 155.6, 140.6, 138.1, 132.5, 129.7, 129.0, 128.8, 127.7, 126.4, 68.5, 21.4, 21.1. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{17}H_{17}N_2O_2^+$ 281.1285; found 281.1286.

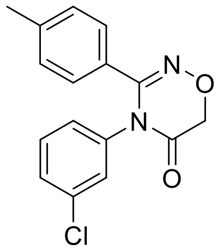


4-(5-Oxo-3-(4-methylphenyl)-5,6-dihydro-4*H*-1,2,4-oxadiazin-4-yl)benzonitrile (4e).

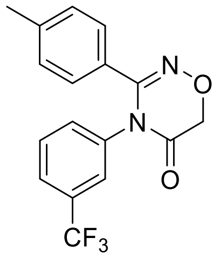
White powder; 60% yield; mp 124–126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.07 (d, *J* = 8.1 Hz, 2H), 4.66 (s, 2H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.0, 154.8, 141.5, 139.3, 132.8, 129.5, 128.6, 128.6, 125.6, 117.8, 111.8, 68.4, 21.4. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{17}H_{14}N_3O_2^+$ 292.1081; found 292.1080.



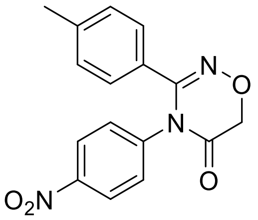
4-(3,5-Dimethylphenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4f).
White crystals; 54% yield; mp 158–159 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, J = 8.1 Hz, 2H), 7.04 (d, J = 8.0 Hz, 2H), 6.86 (s, 1H), 6.76 (s, 2H), 4.63 (s, 2H), 2.29 (s, 3H), 2.24 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.3, 155.6, 140.6, 138.7, 134.9, 129.9, 129.0, 128.7, 126.4, 125.7, 68.5, 21.4, 21.1. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{18}H_{19}N_2O_2^+$ 295.1439; found 295.1441.



4-(3-Chlorophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4g).
White powder; 25% yield; mp 144–145 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.20 (m, 5H), 7.07 (d, J = 8.0 Hz, 2H), 7.00–7.04 (m, 1H), 4.65 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.3, 155.3, 141.2, 136.4, 134.7, 130.0, 129.4, 128.8, 128.5, 128.4, 126.3, 126.0, 68.6, 21.5. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{14}ClN_2O_2^+$ 301.0738; found 301.0739.

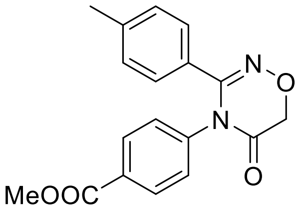


3-(4-Methylphenyl)-4-(3-(trifluoromethyl)phenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4h).
White powder; 24% yield; mp 106–108 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 7.9 Hz, 1H), 7.46–7.40 (m, 2H), 7.33 (d, J = 8.3 Hz, 1H), 7.24 (d, J = 8.2 Hz, 2H), 7.05 (d, J = 7.9 Hz, 2H), 4.67 (s, 2H), 2.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 155.1, 141.2, 135.8, 131.5 (q, J = 185.2 Hz), 131.2, 129.5, 129.3, 128.7, 125.7, 125.0 (q, J = 3.8 Hz), 124.8 (q, J = 3.6 Hz), 123.3 (q, J = 275 Hz), 68.4, 21.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ –62.89. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{17}H_{14}N_2O_2F_3^+$ 335.1002; found 335.1008.



4-(4-Nitrophenyl)-3-(4methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one (4i).

Pale yellow powder; 43% yield; mp 89–90 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 9.0 Hz, 2H), 7.35 (d, J = 9.0 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 7.07 (d, J = 8.0 Hz, 2H), 4.68 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 154.8, 146.6, 141.6, 140.8, 129.6, 128.6, 128.6, 125.6, 124.3, 68.4, 21.4. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{16}H_{14}N_3O_4^+$ 312.0977; found 312.0979.

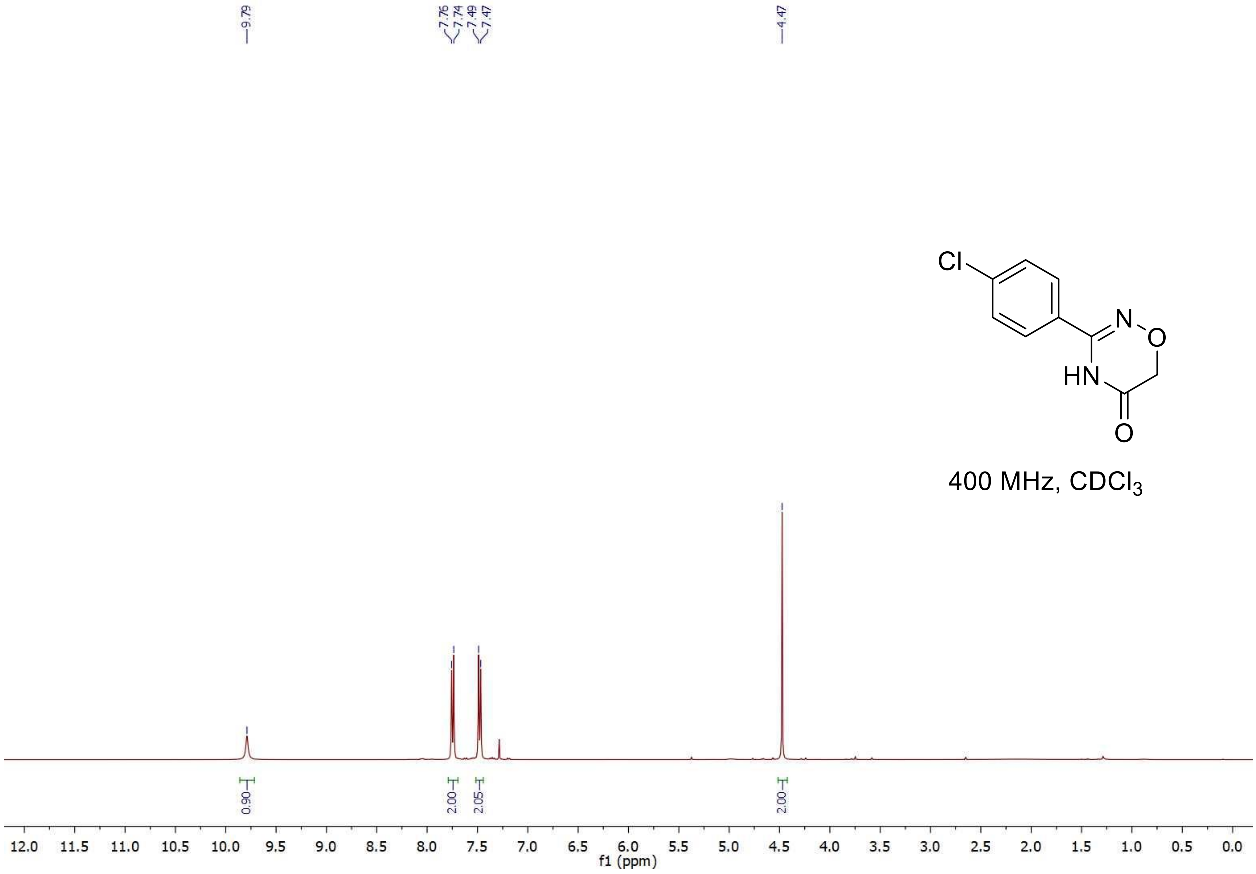


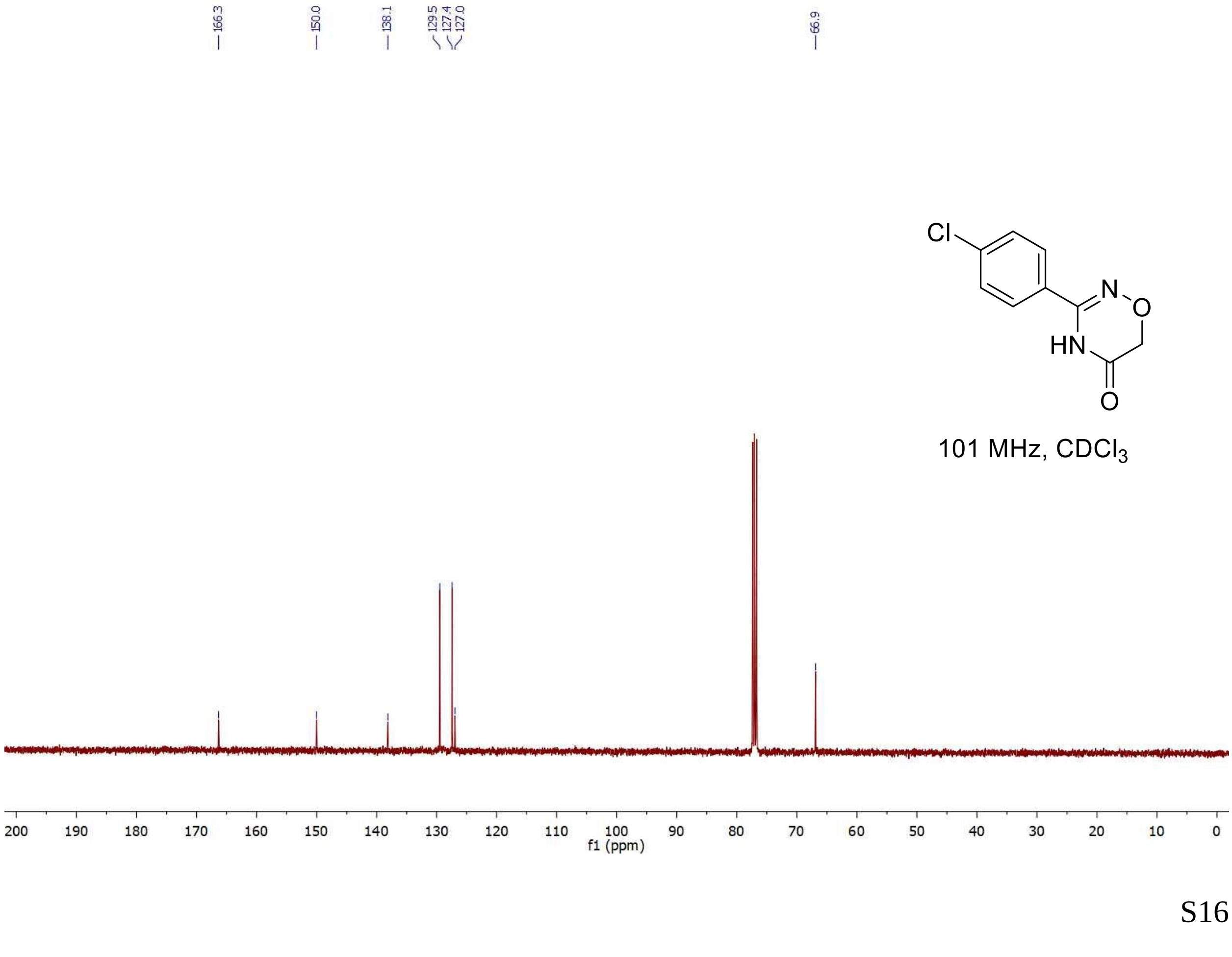
Methyl 4-(5-oxo-3-(4-methylphenyl)-5,6-dihydro-4*H*-1,2,4-oxadiazin-4-yl)benzoate (4j)

White powder; 31% yield; mp 113–115 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.6 Hz, 2H), 7.27–7.21 (m, 4H), 7.04 (d, J = 8.0 Hz, 2H), 4.66 (s, 2H), 3.90 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 164.2, 155.2, 141.1, 139.3, 130.3, 129.5, 129.3, 128.6, 127.8, 125.9, 68.4, 52.4, 21.4. HRMS (ESI), m/z: $[M+H]^+$ calcd for $C_{18}H_{17}N_2O_4{}^+$ 325.1179; found 325.1183.

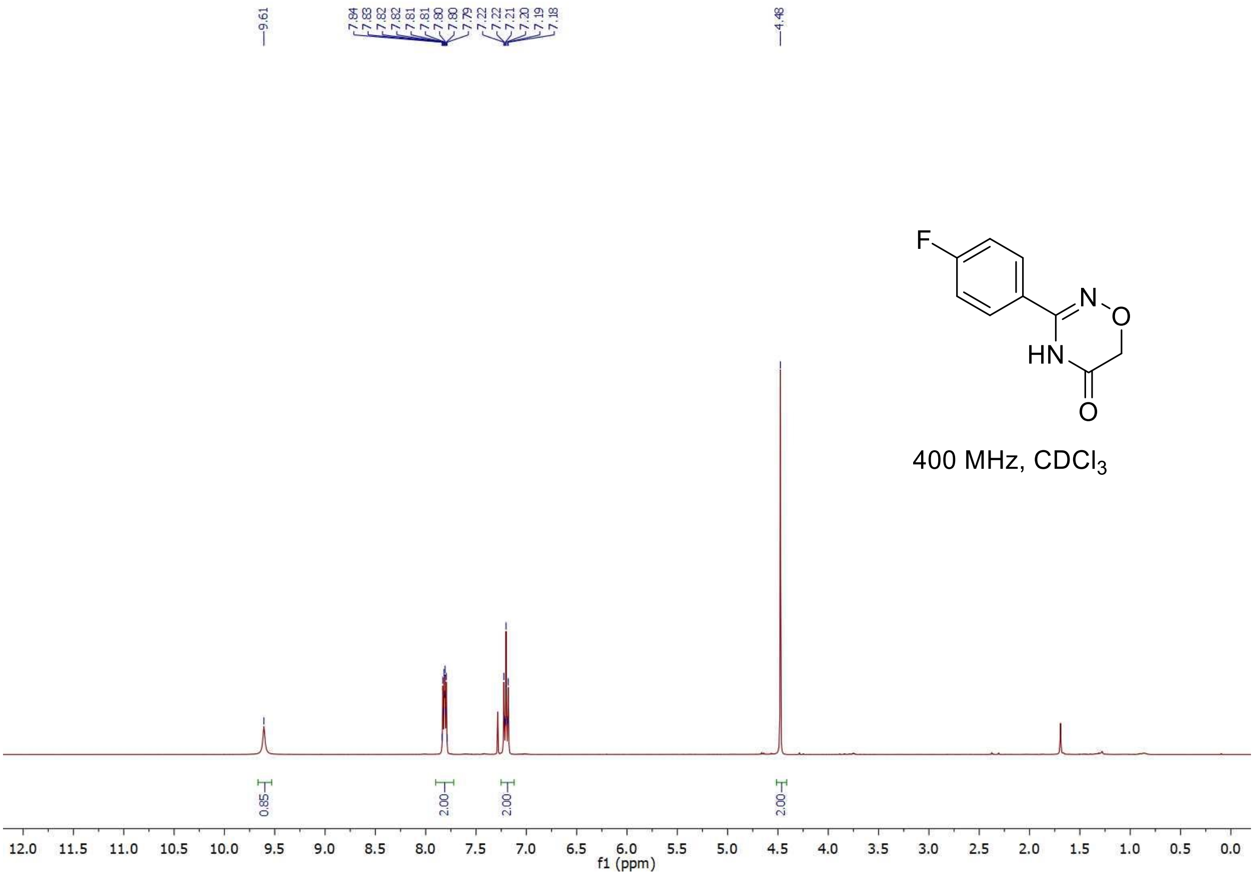
S4. ^{1}H, ^{13}C, and ^{19}F NMR spectra of compounds 1, 3, and 4

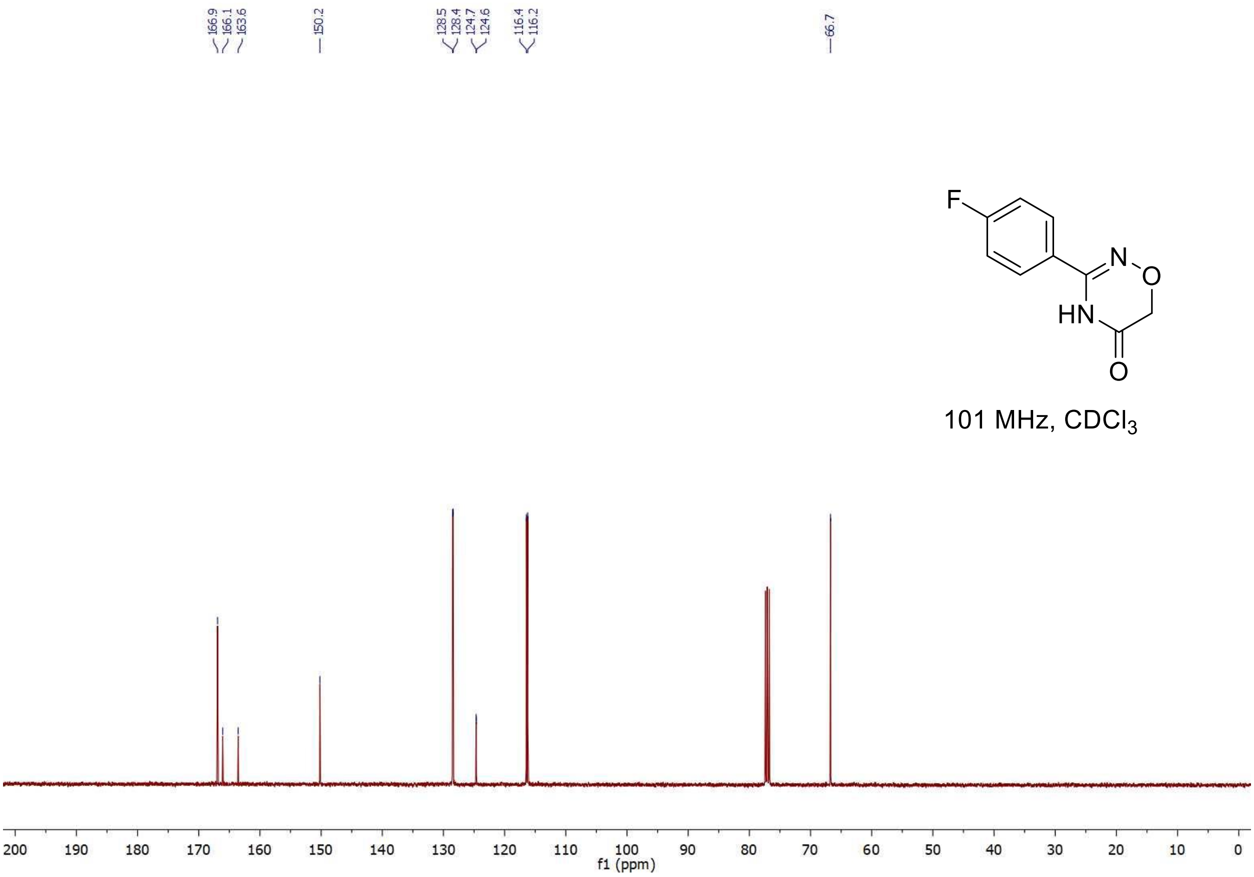
^{1}H and ^{13}C spectra of 3-(4-chlorophenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1e).**

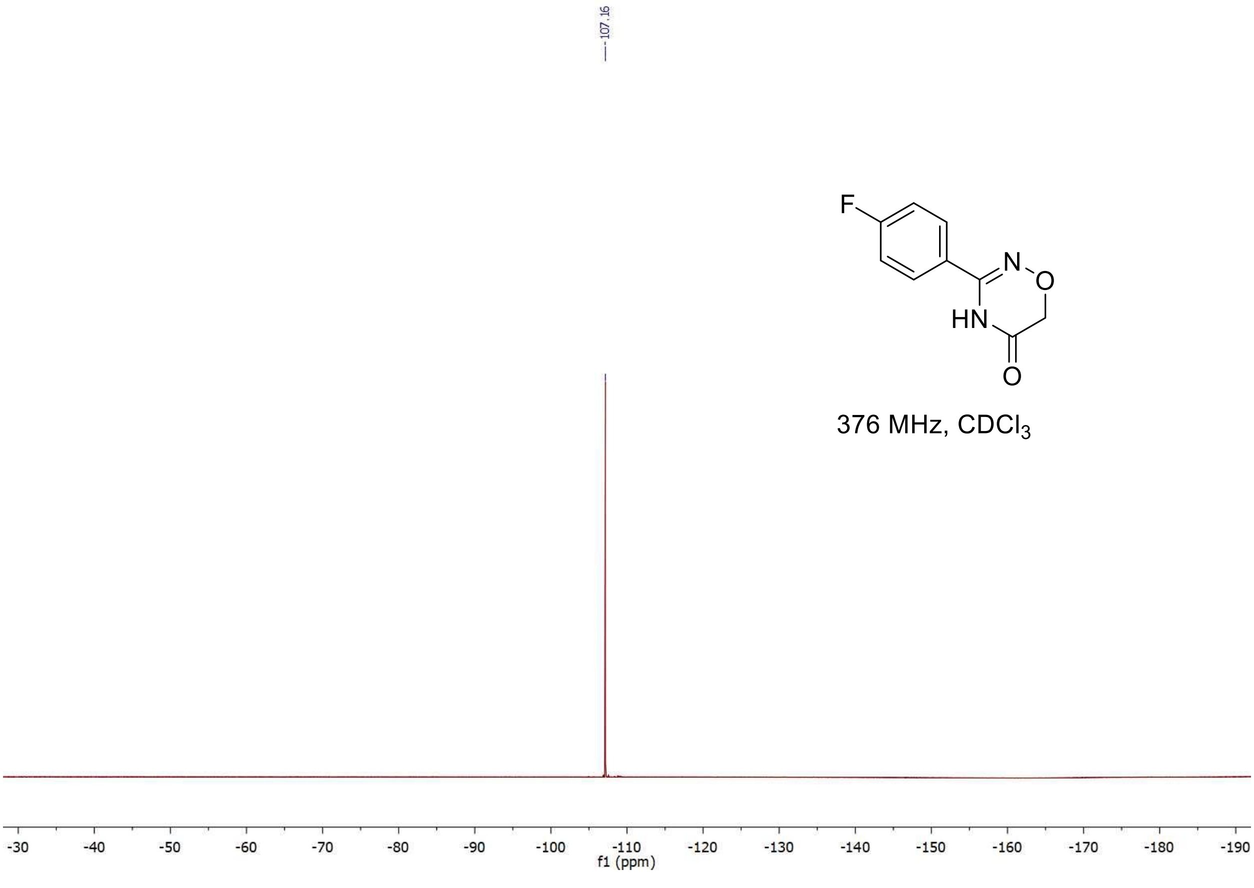




[1]H, [13]C and [19]F spectra of 3-(4-fluorophenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1f).**

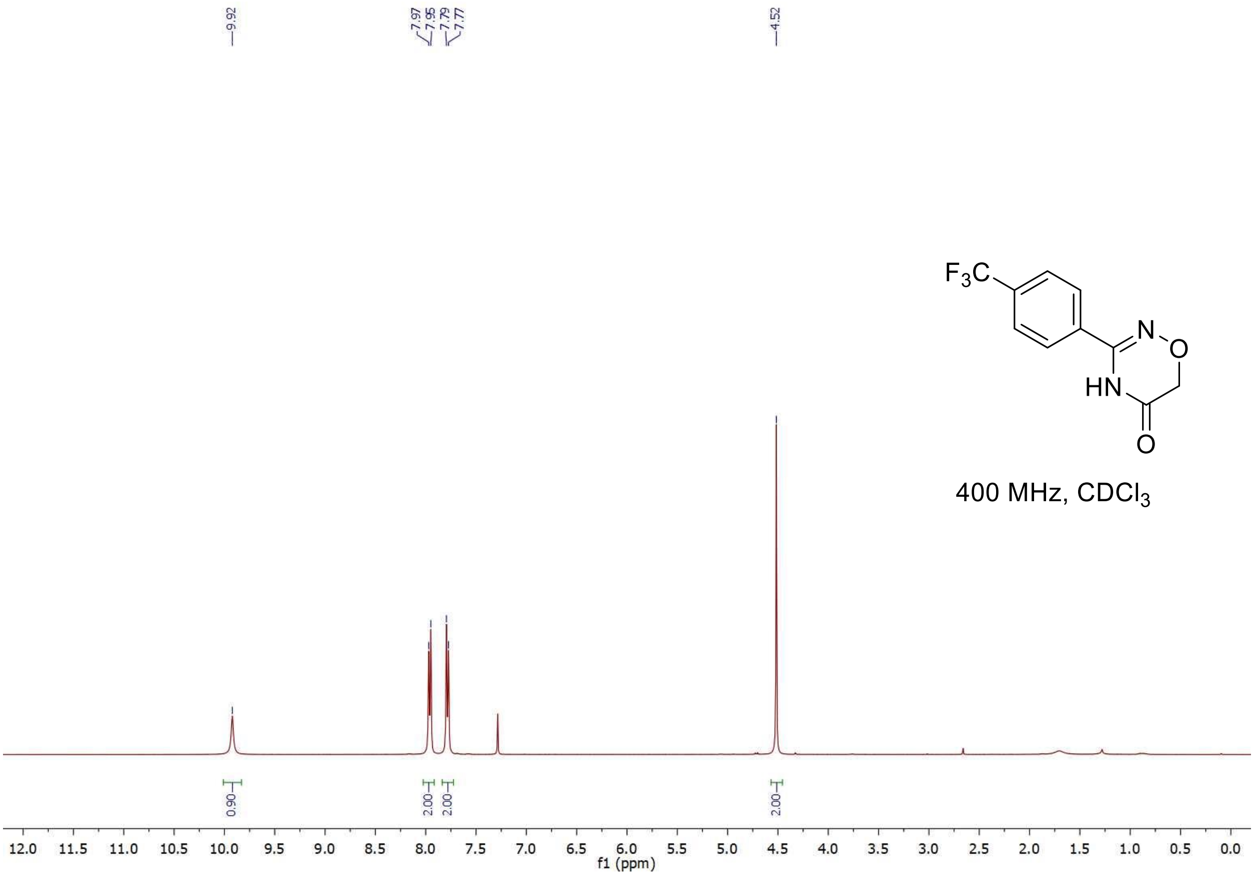


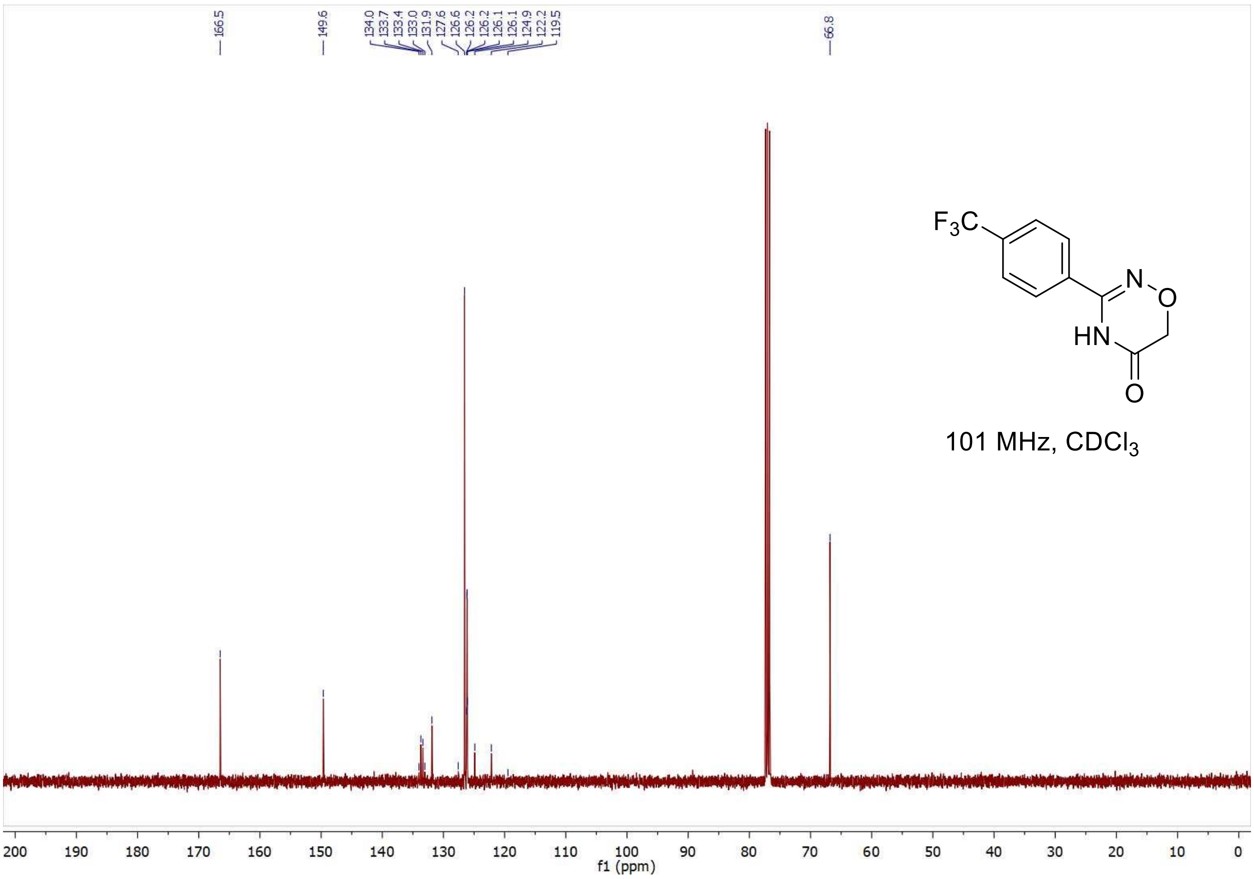


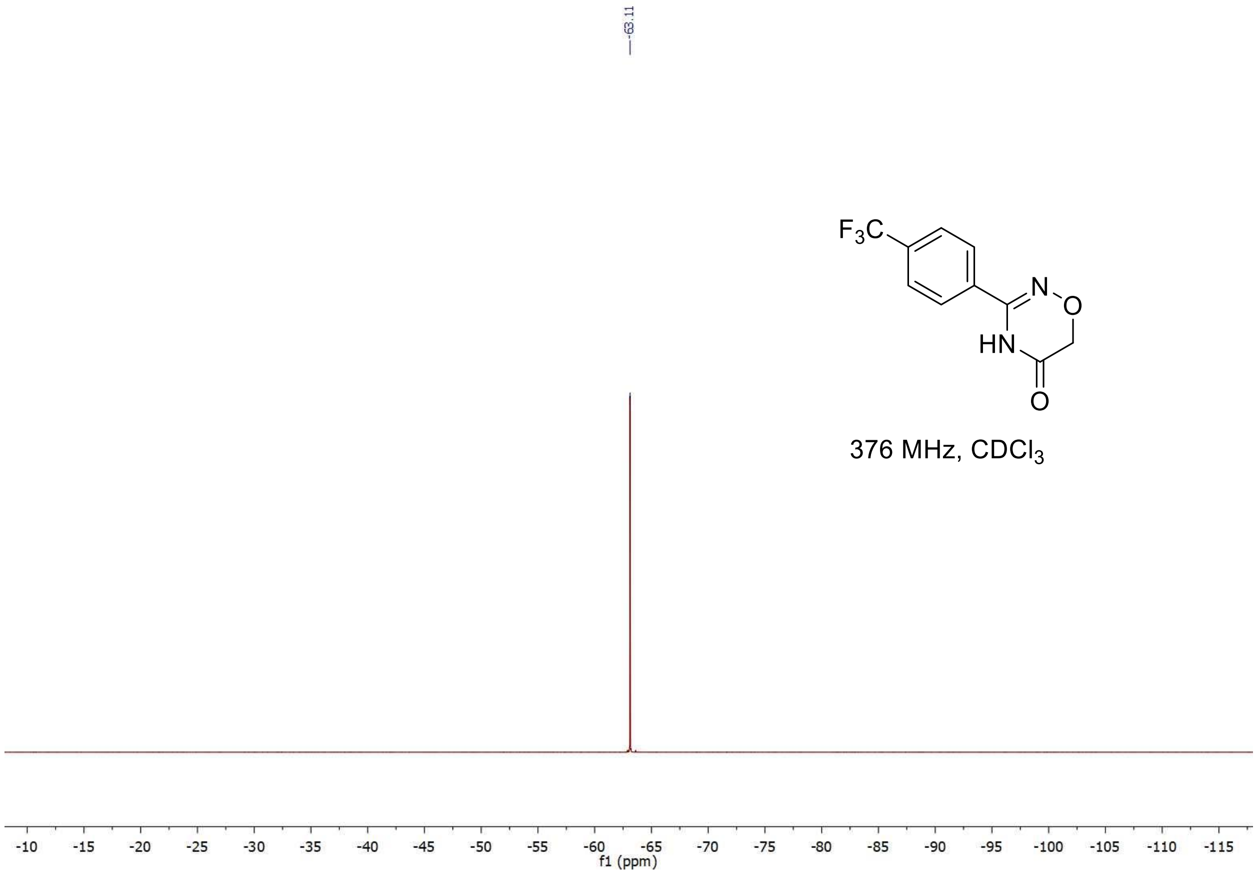


376 MHz, $CDCl_3$

^1H, ^{13}C and ^{19}F spectra of 3-(4-(trifluoromethyl)phenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1g).**



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F3C
N
O
HN
O
376 MHz, CDCl3
-10
-15
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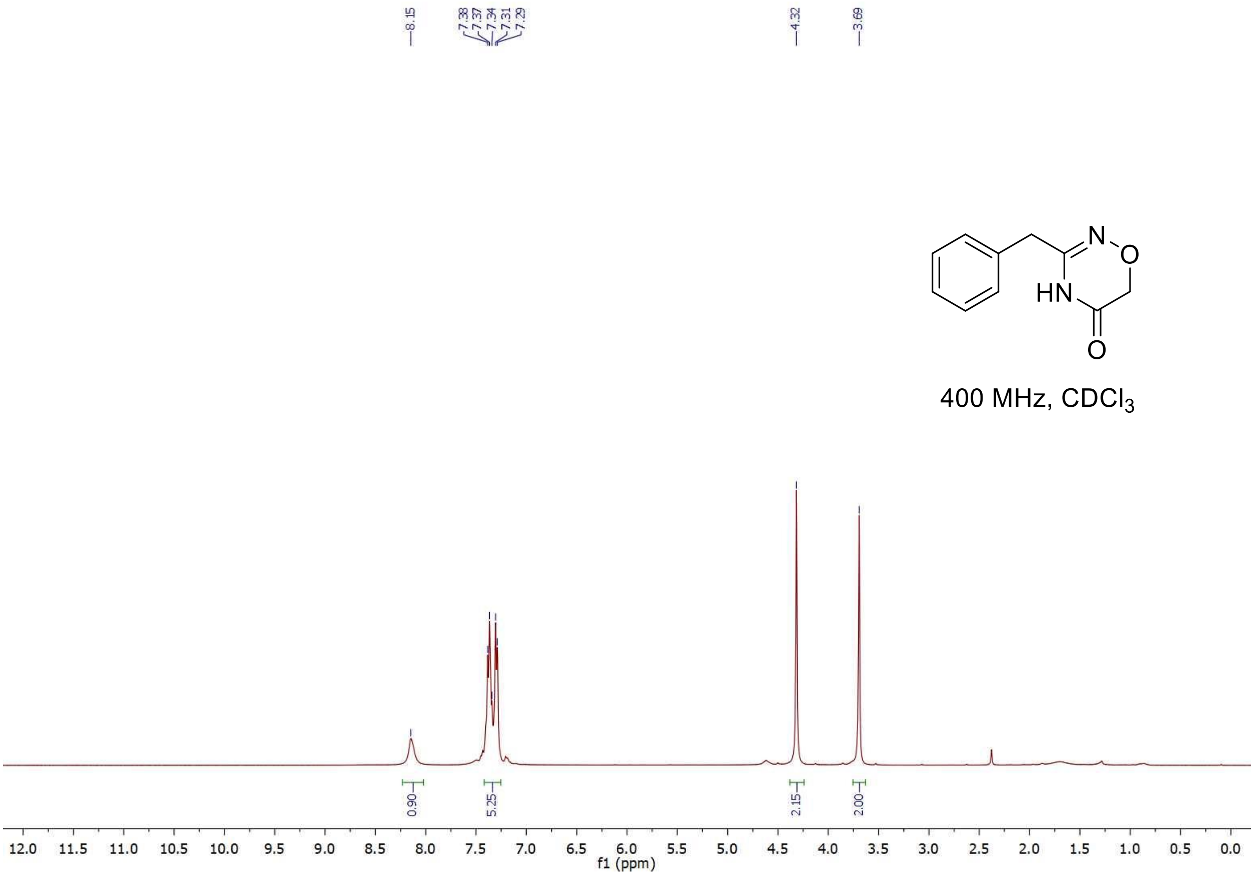
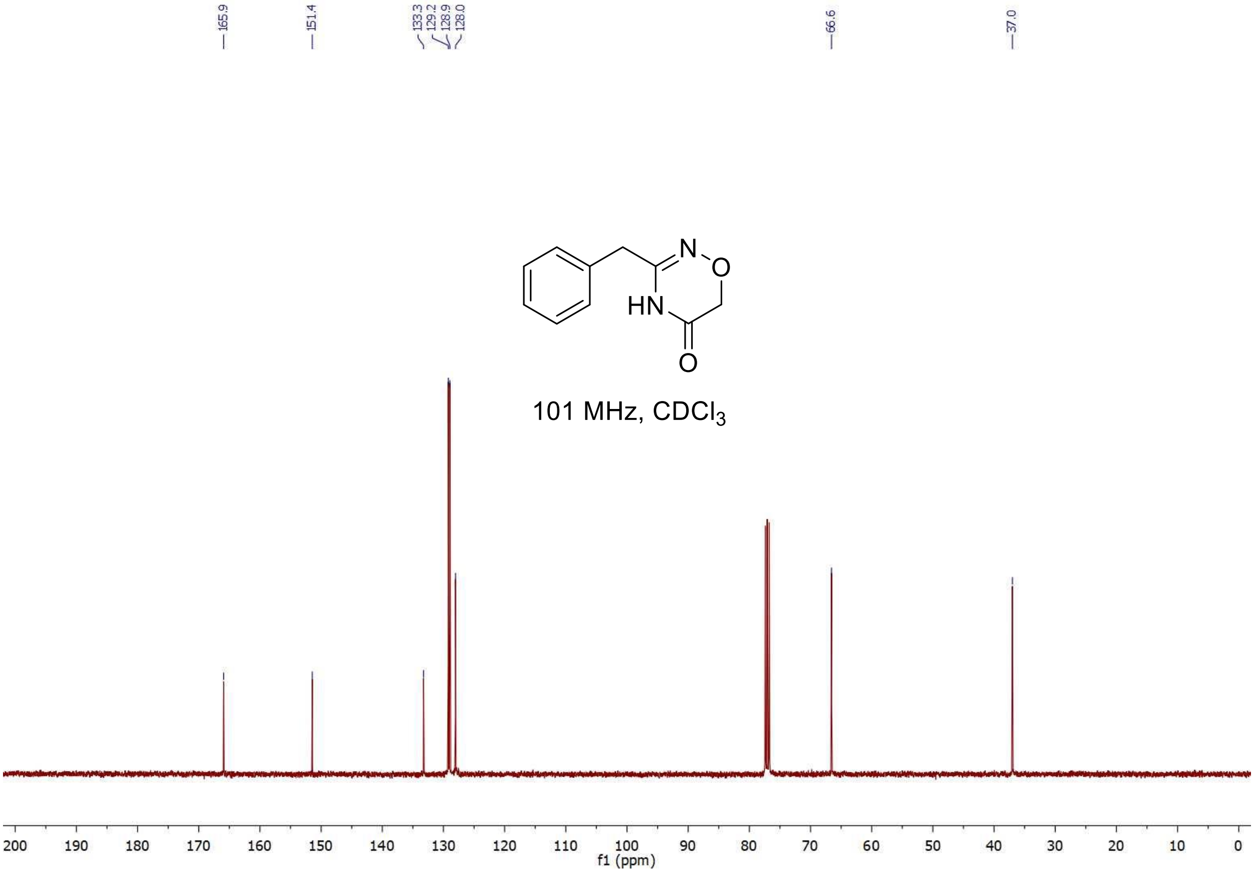
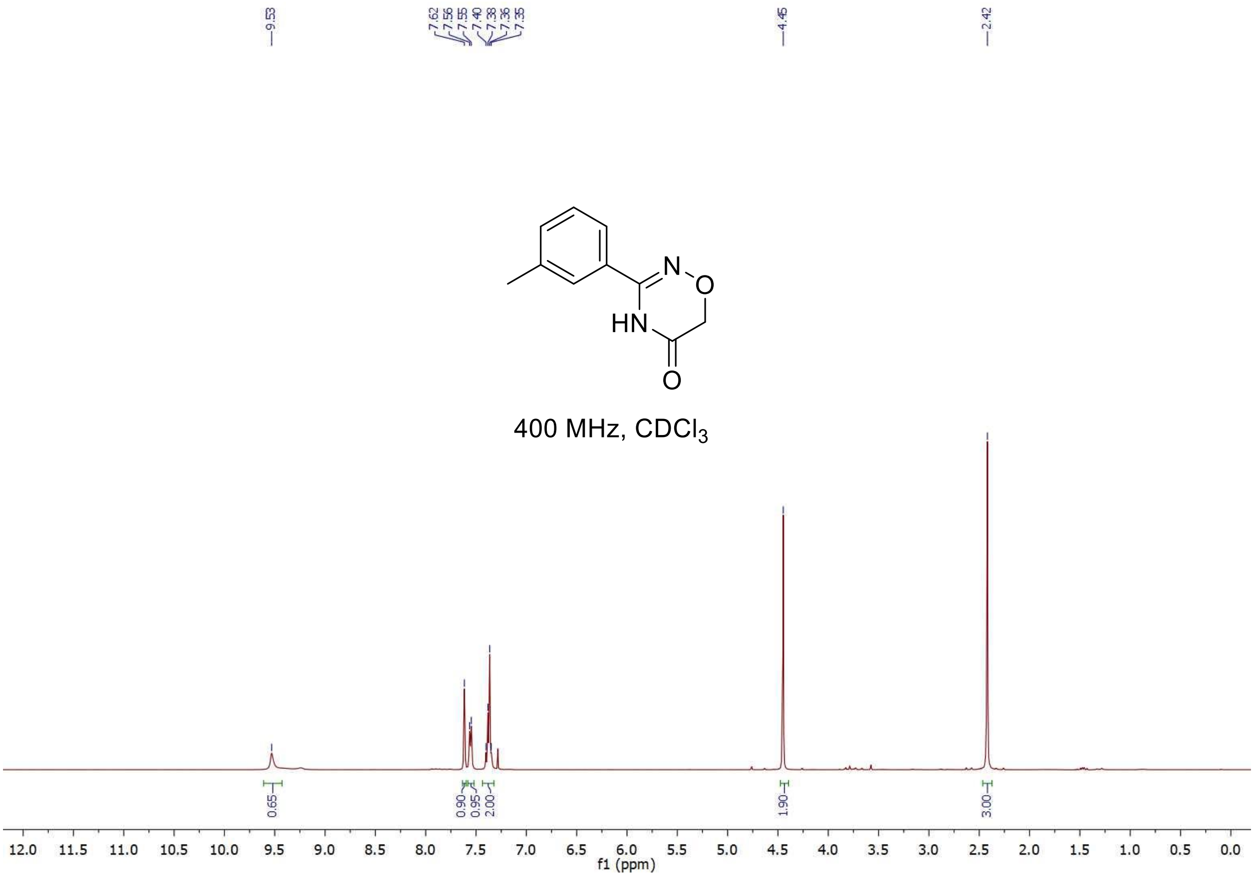
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
f1 (ppm)

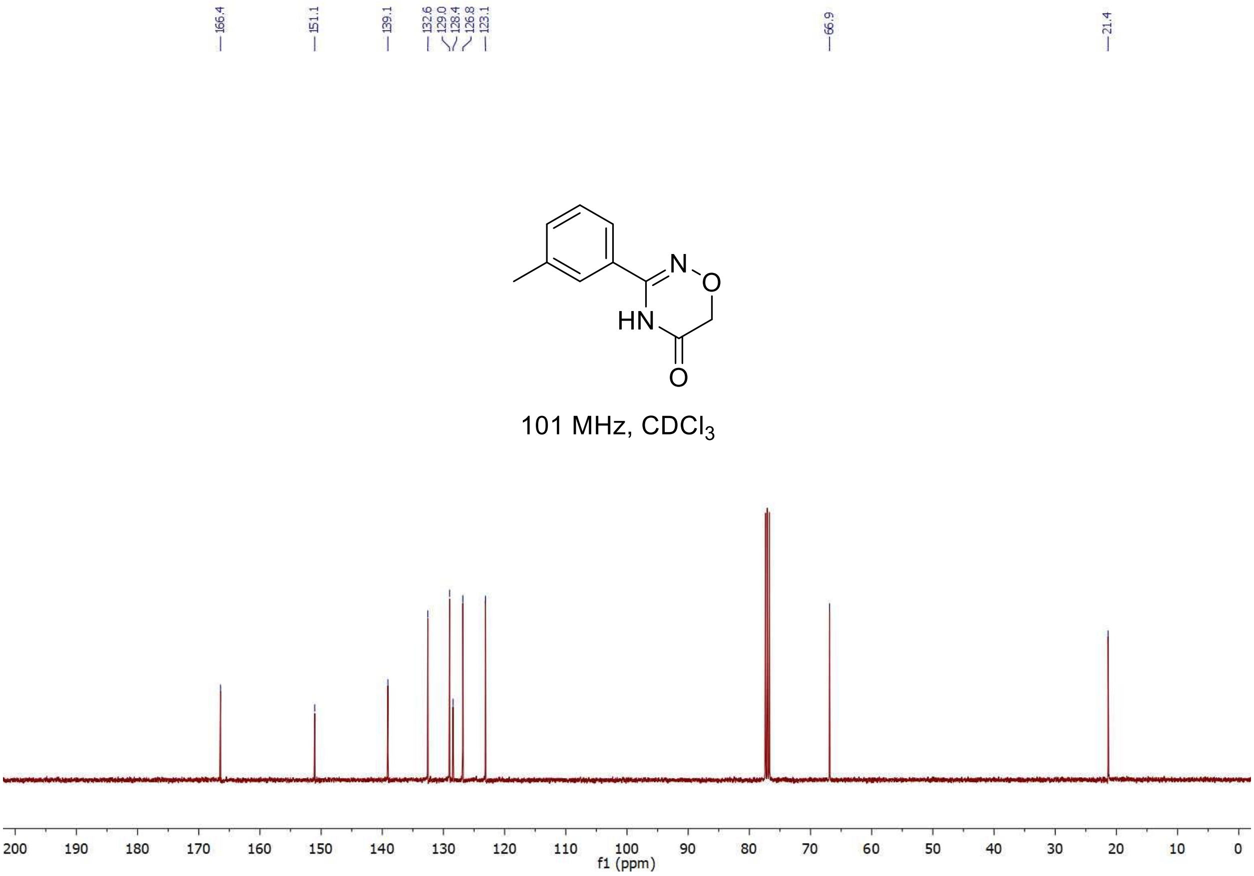
^{1}H and ^{13}C spectra of 3-benzyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1h).**



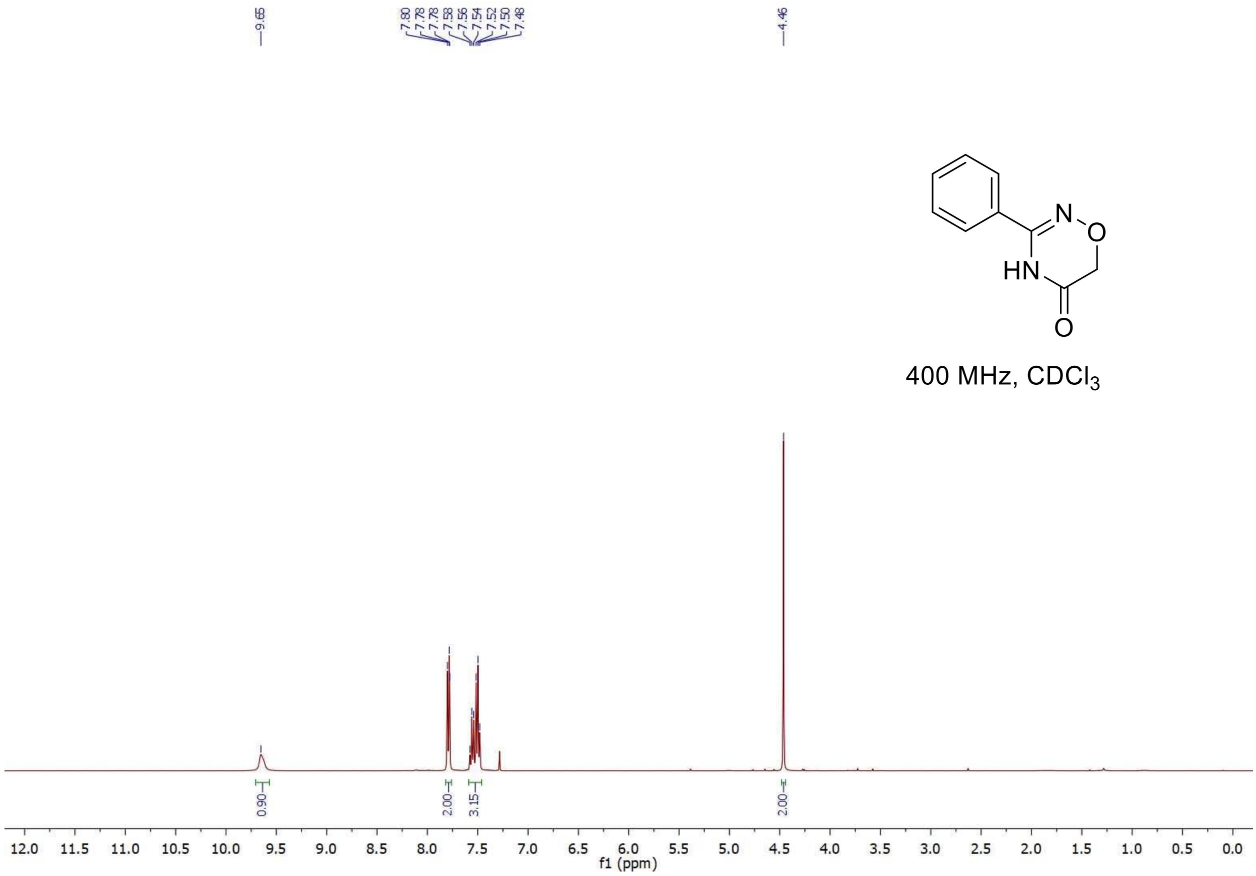


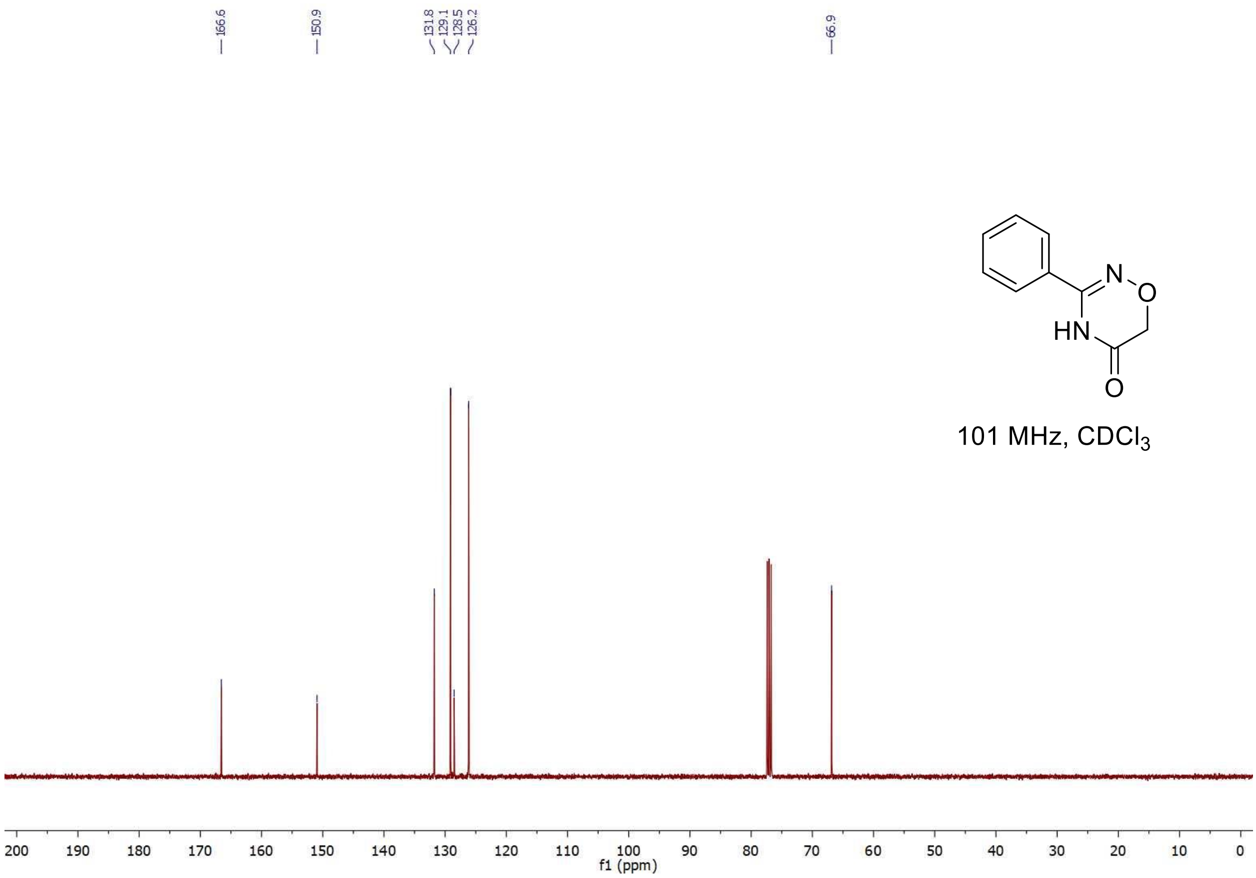
[1]H and [13]C spectra of 3-(3-Methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1i).**



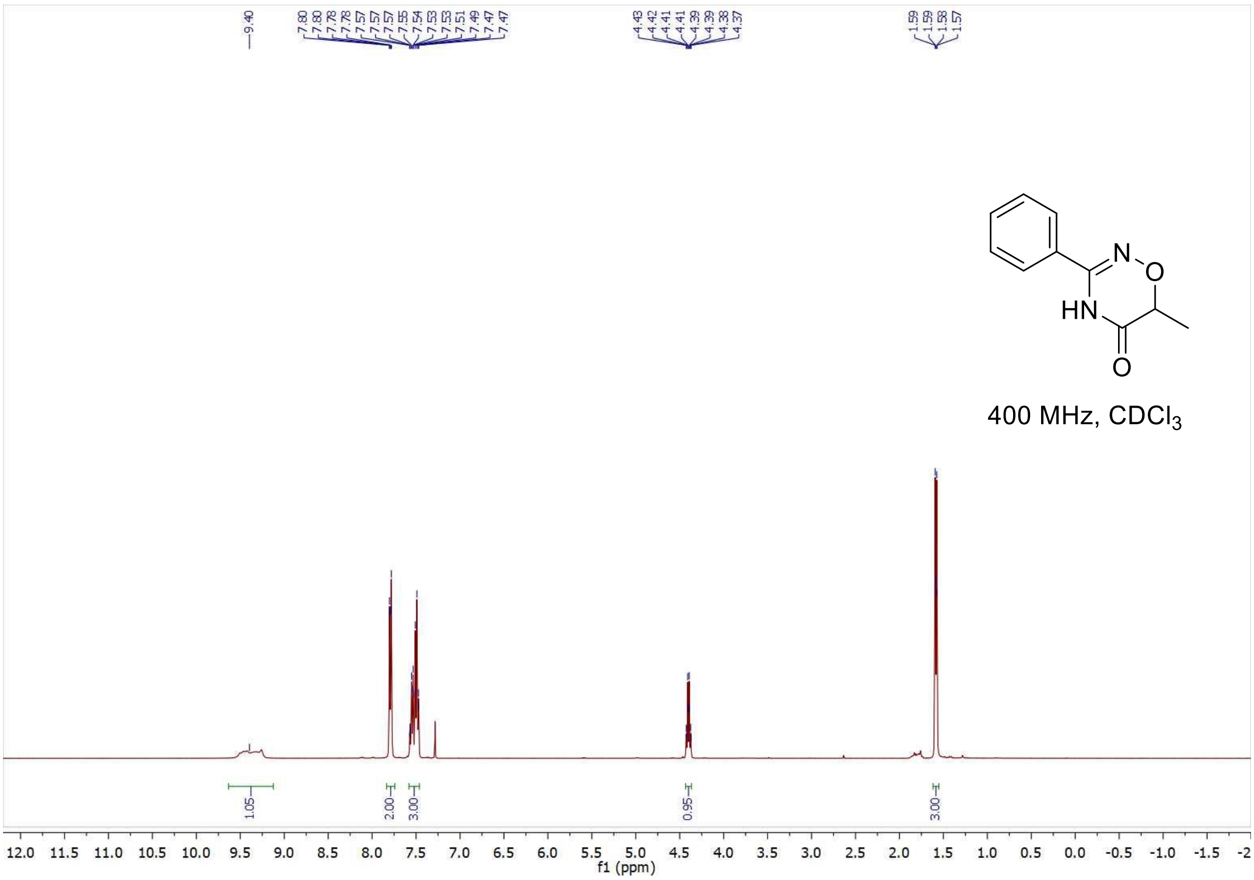


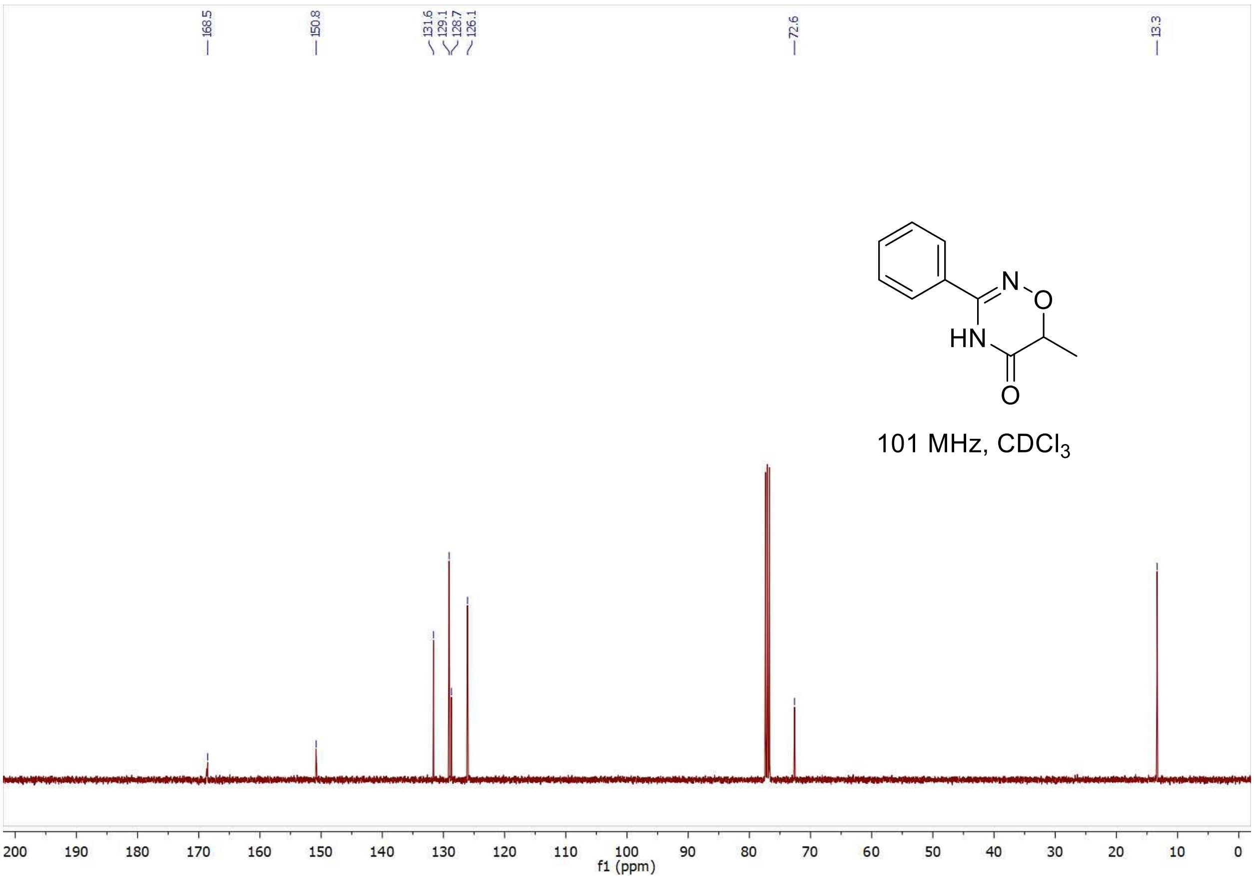
[1]H and [13]C spectra of 3-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1o).**



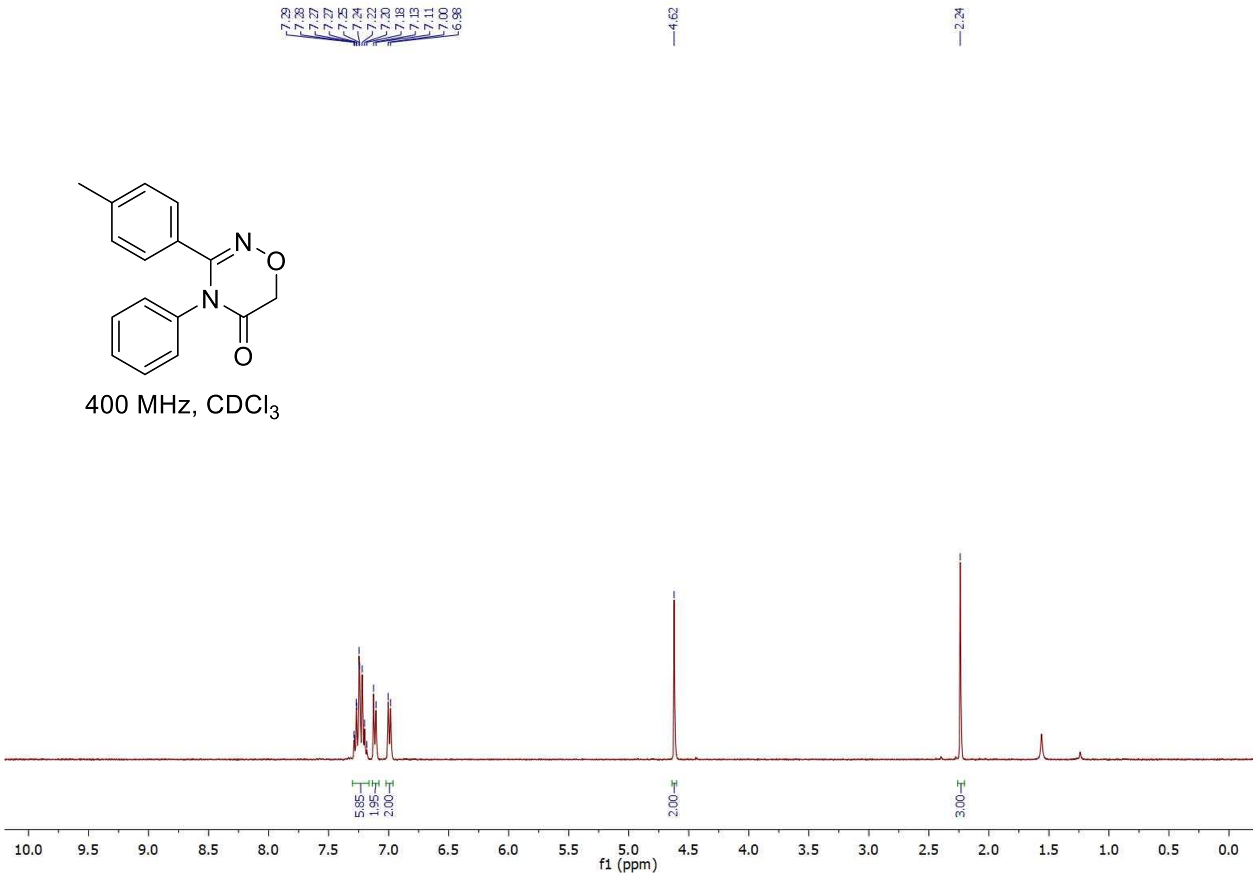


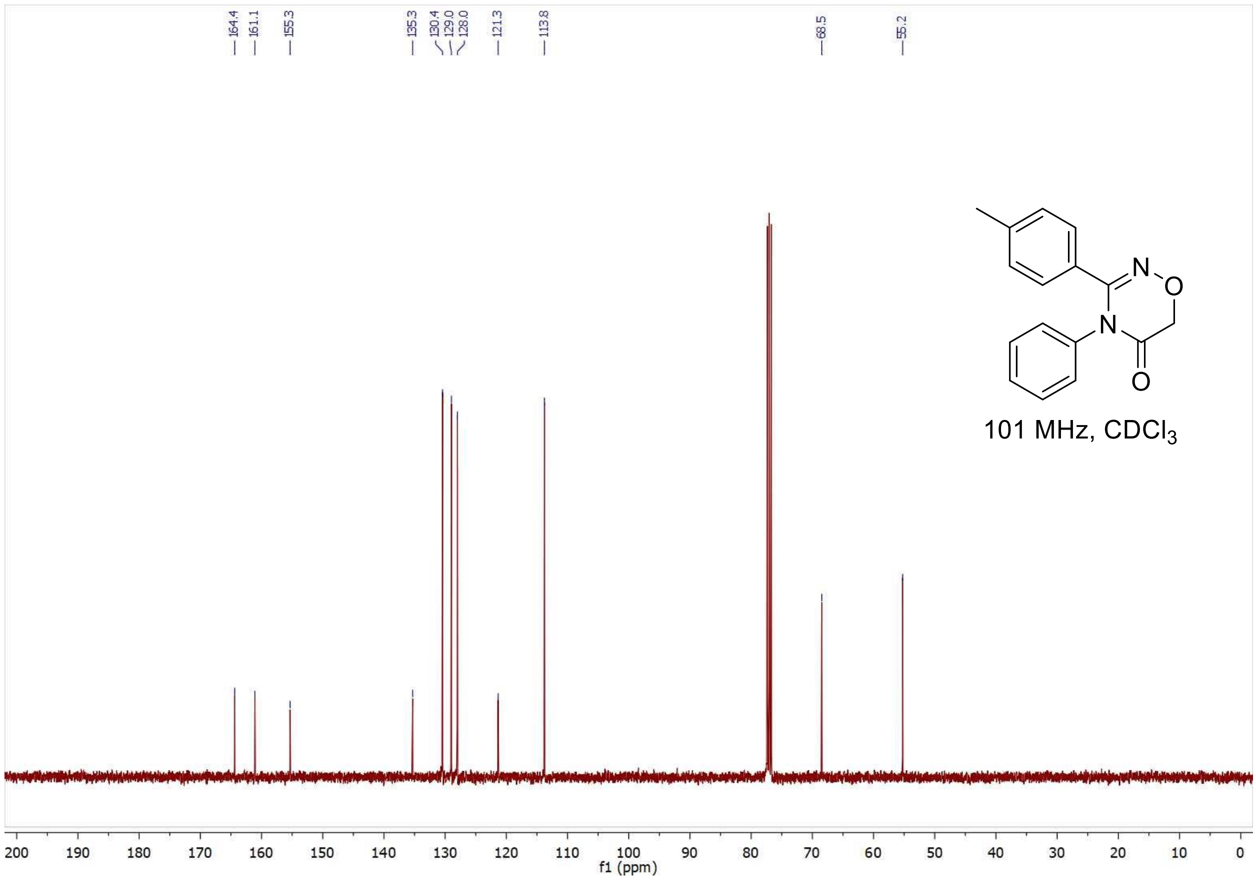
^{1}H and ^{13}C spectra of 6-methyl-3-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(1p).**



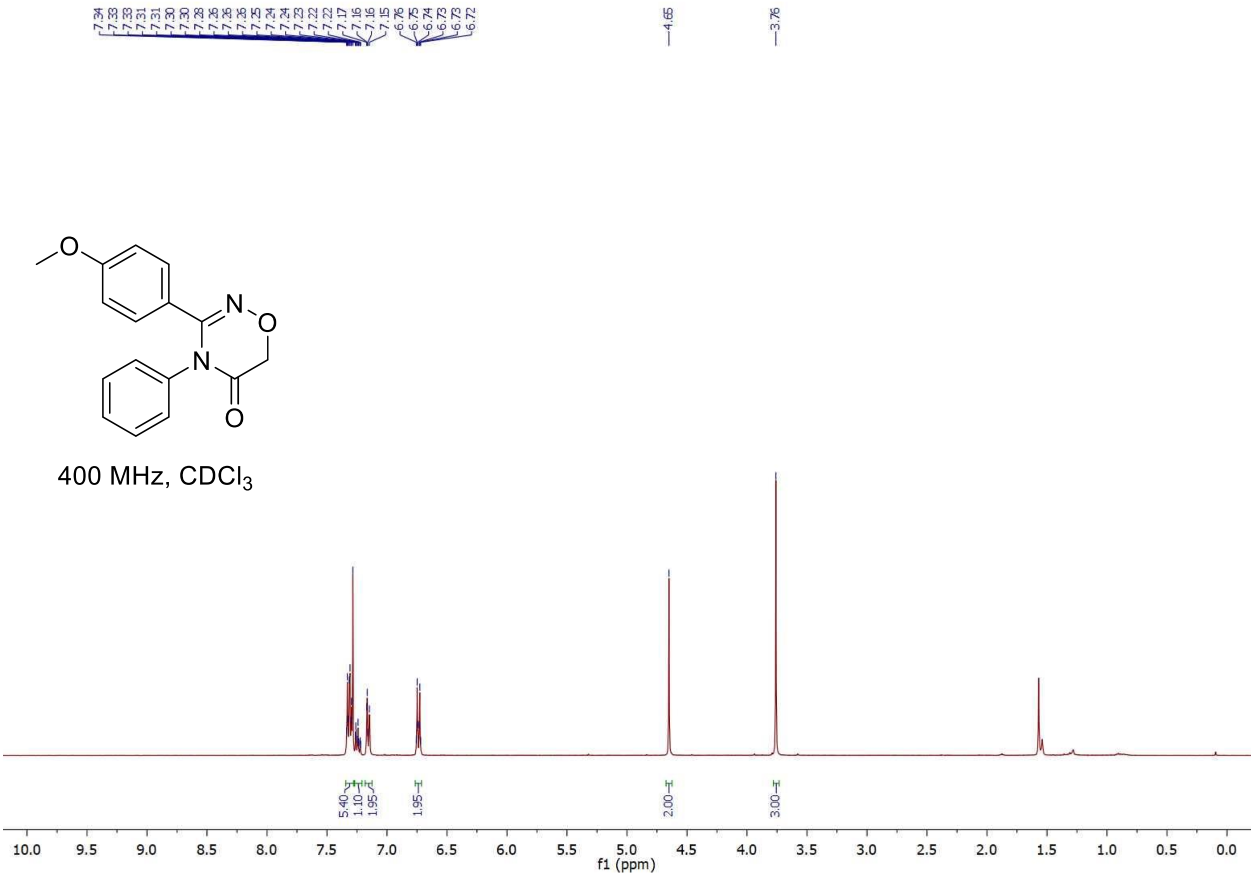


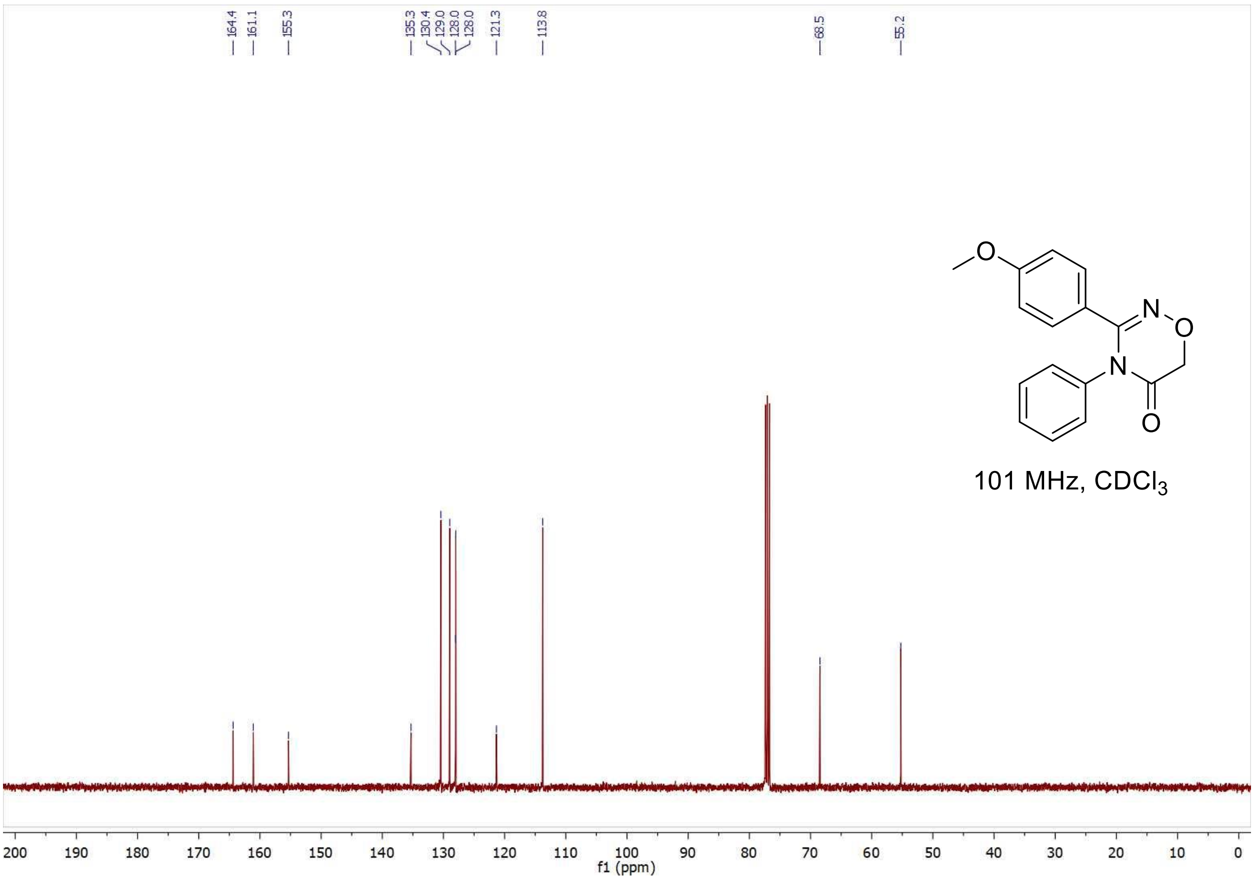
^{1}H and ^{13}C spectra of 4-phenyl-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3a).**



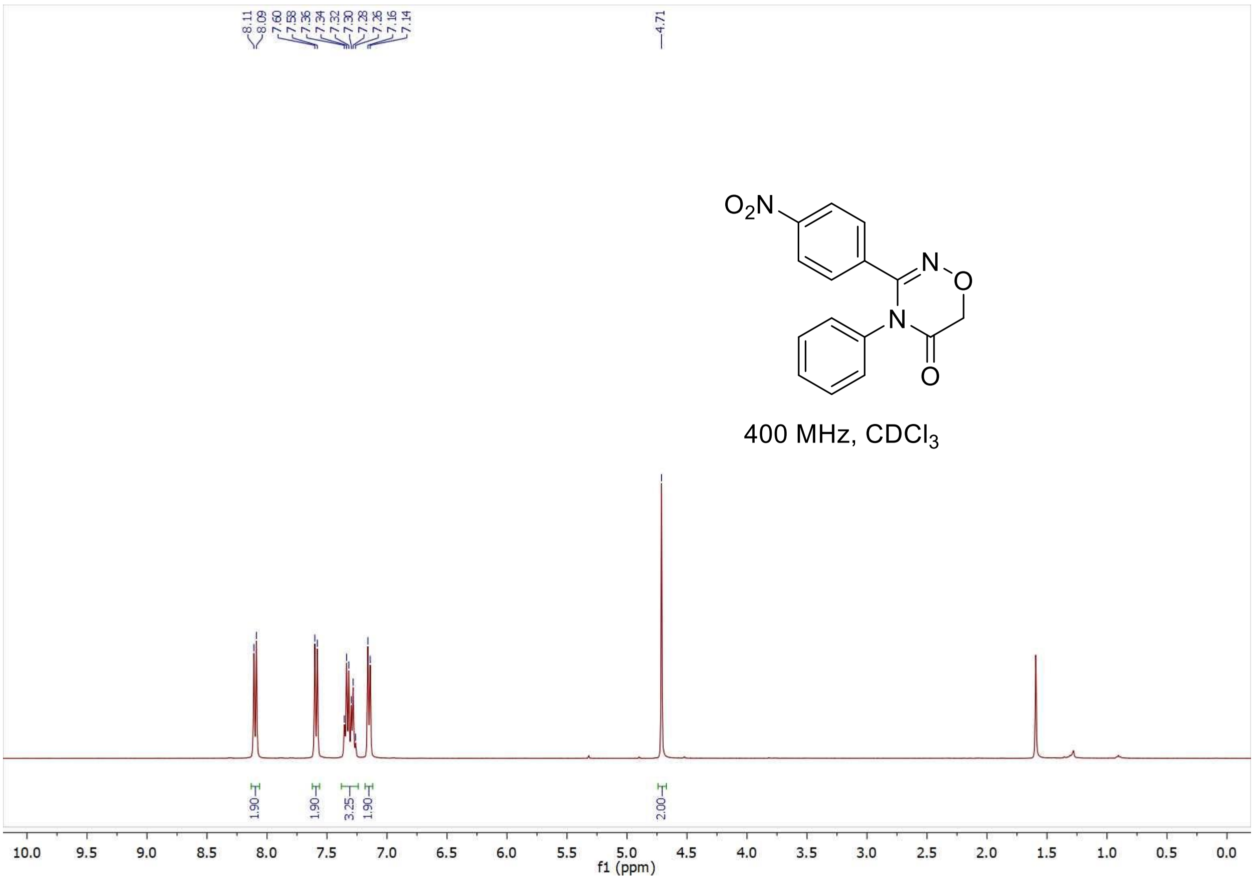


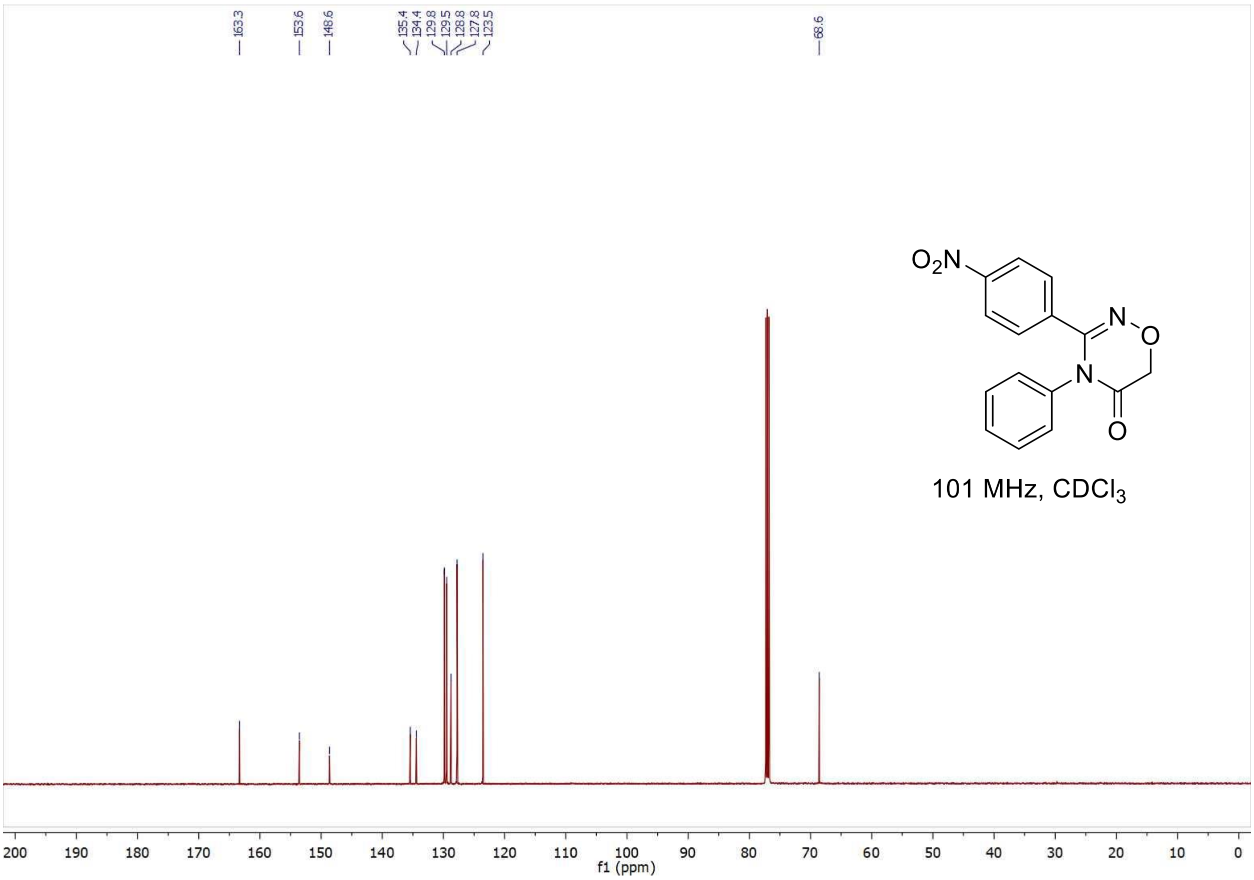
[1]H and [13]C spectra of 3-(4-methoxyphenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3b).**



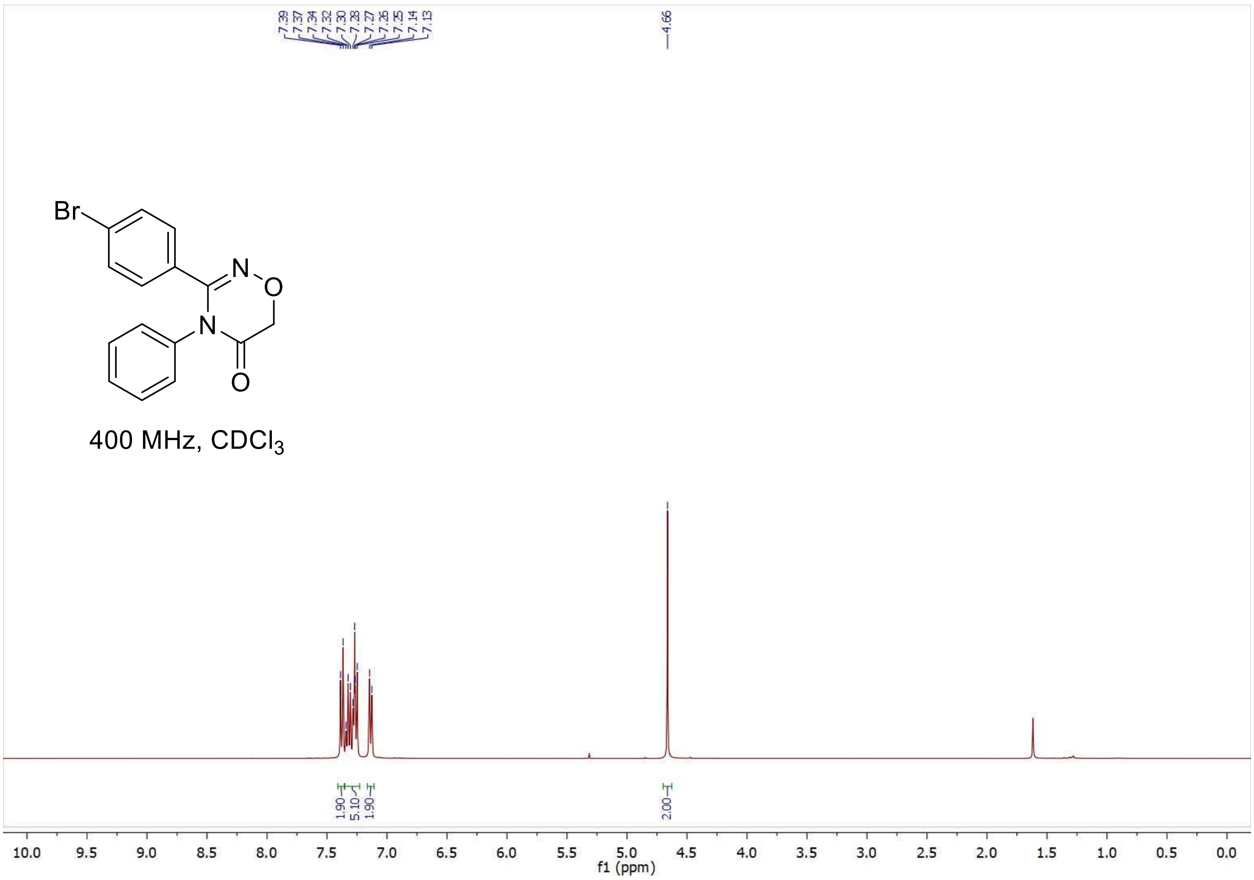


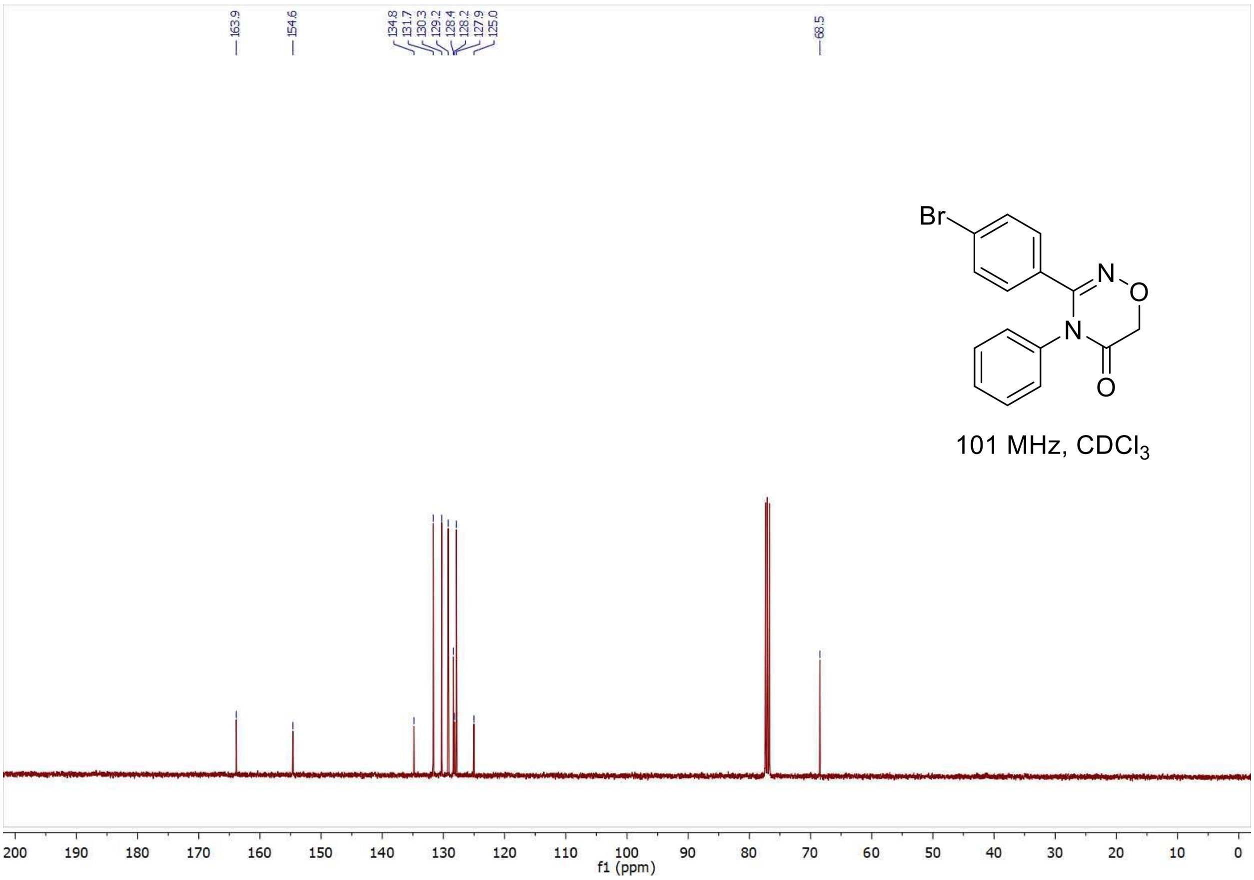
[1]H and [13]C spectra of 3-(4-nitrophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3c).**



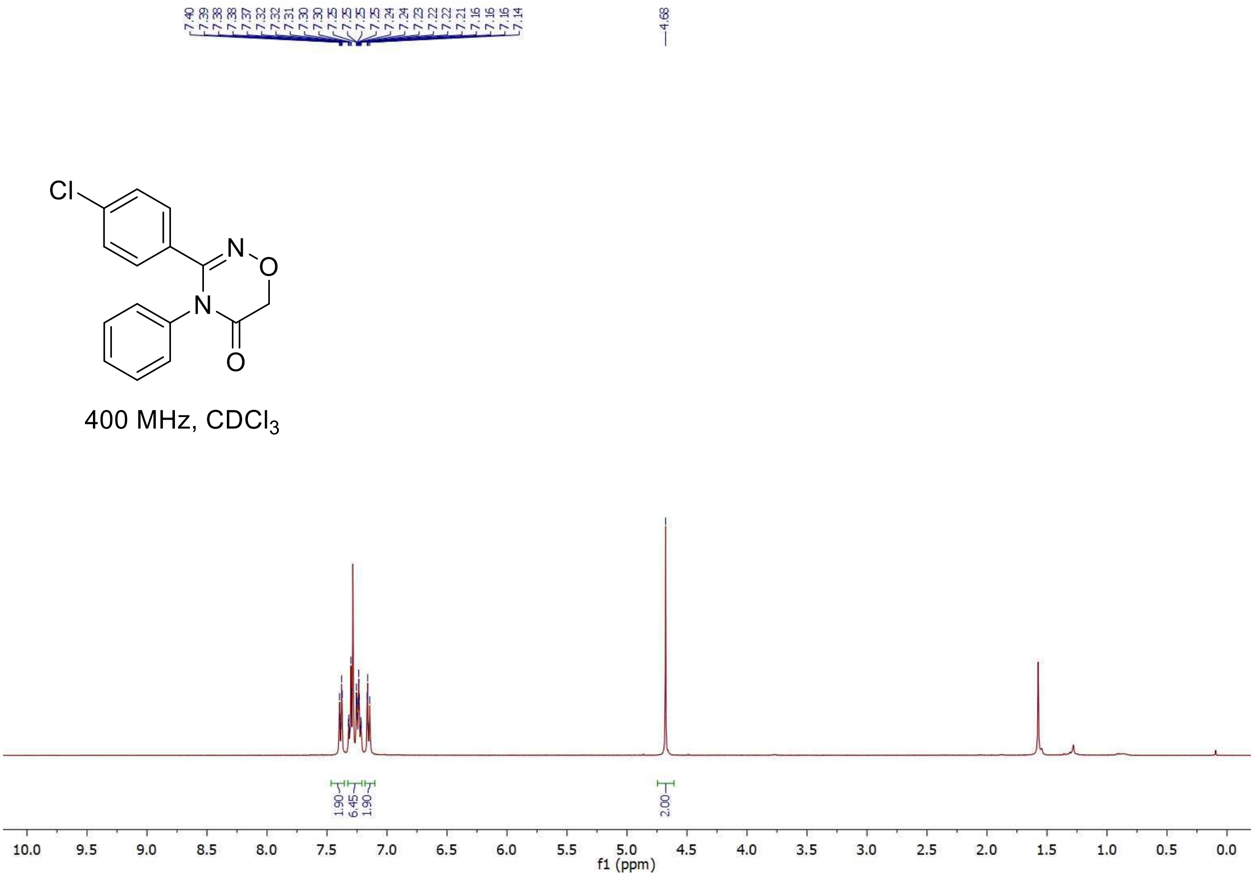


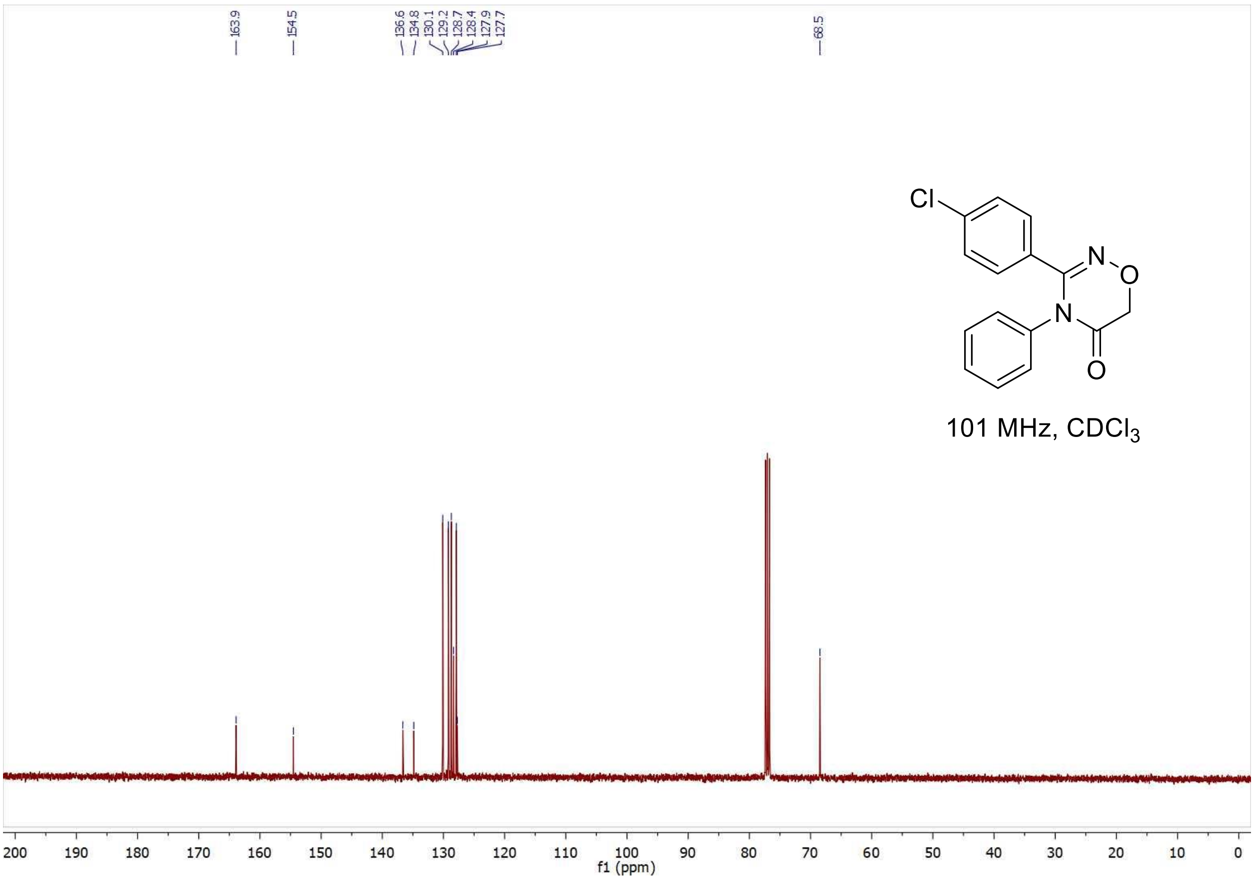
^{1}H and ^{13}C spectra of 3-(4-bromophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3d).**



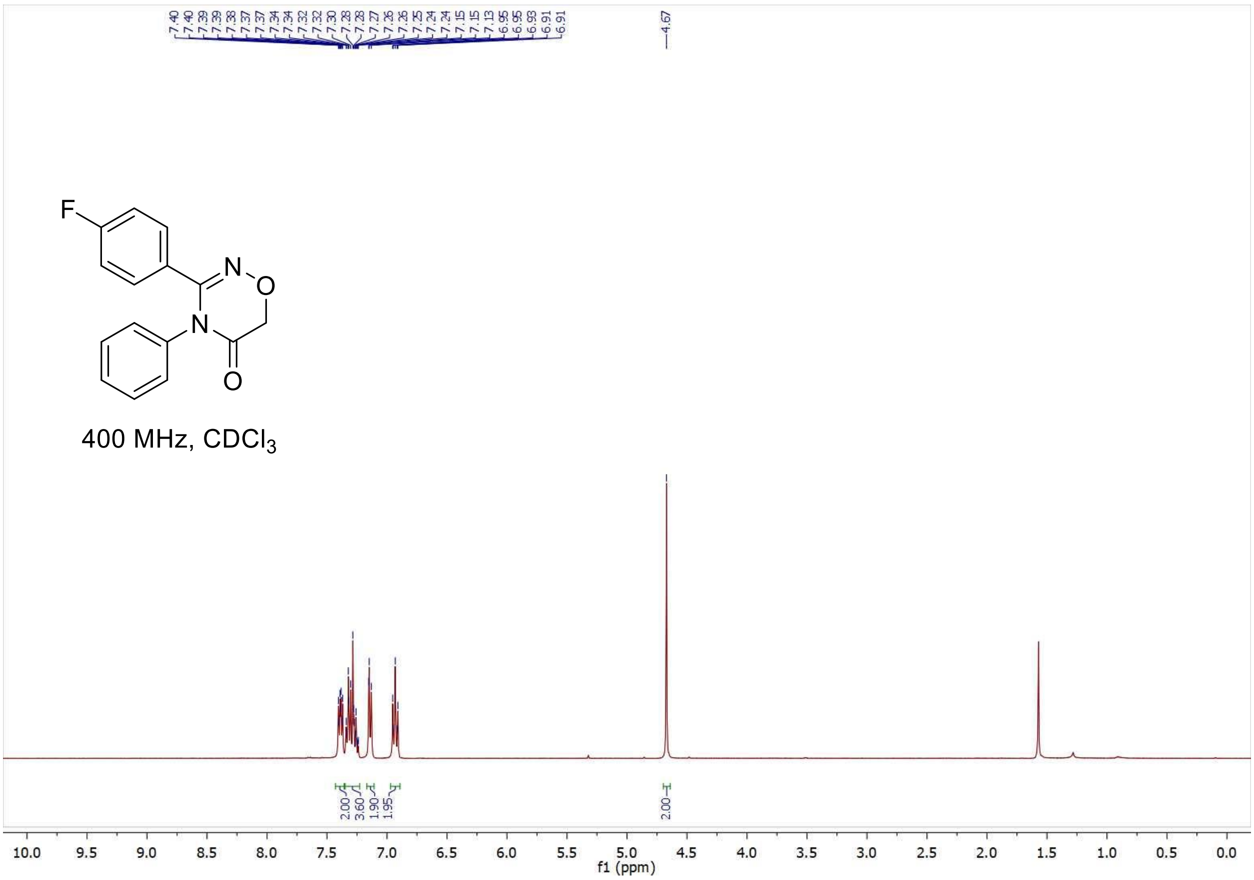


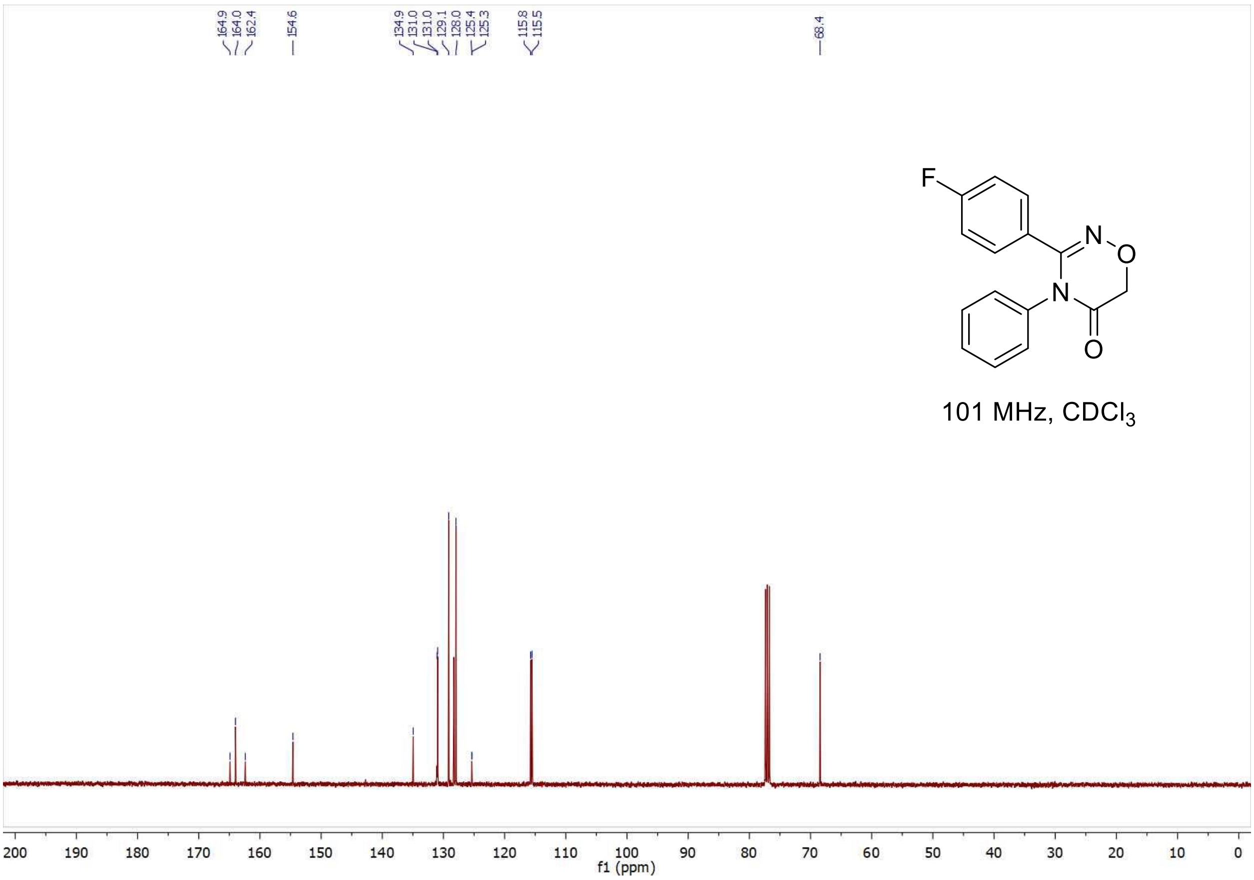
^{1}H and ^{13}C spectra of 3-(4-chlorophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3e).**

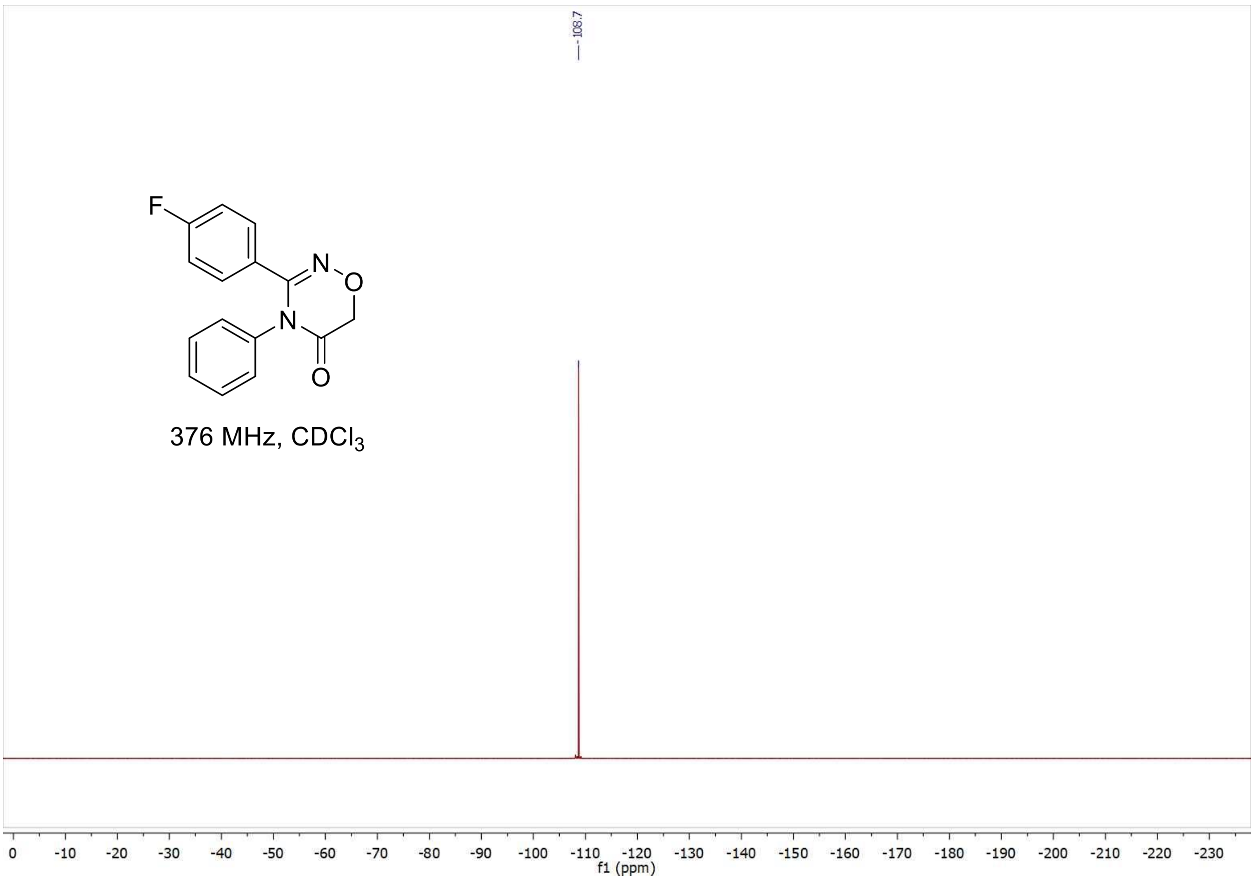




^{1}H, ^{13}C and ^{19}F spectra of 3-(4-fluorophenyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3f).**



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376 MHz, CDCl3
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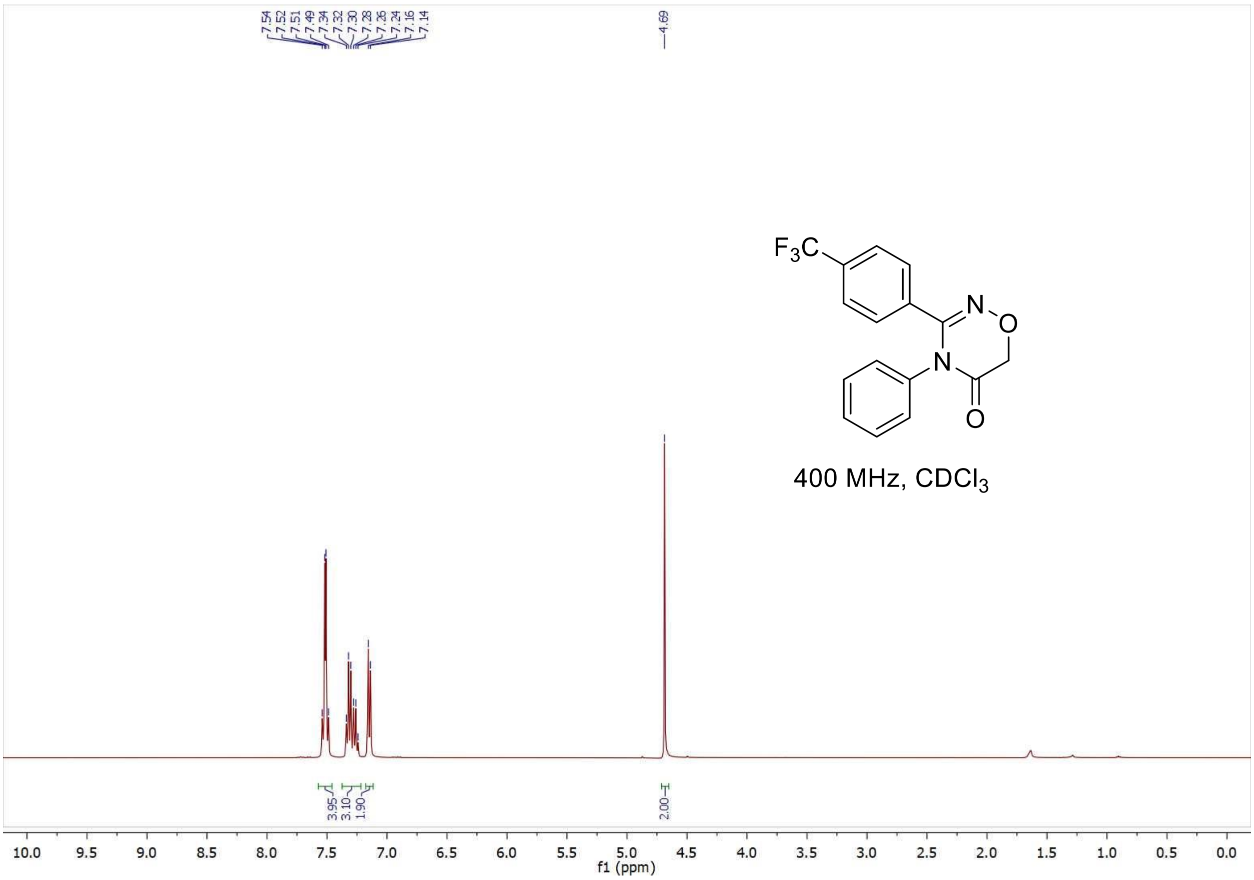
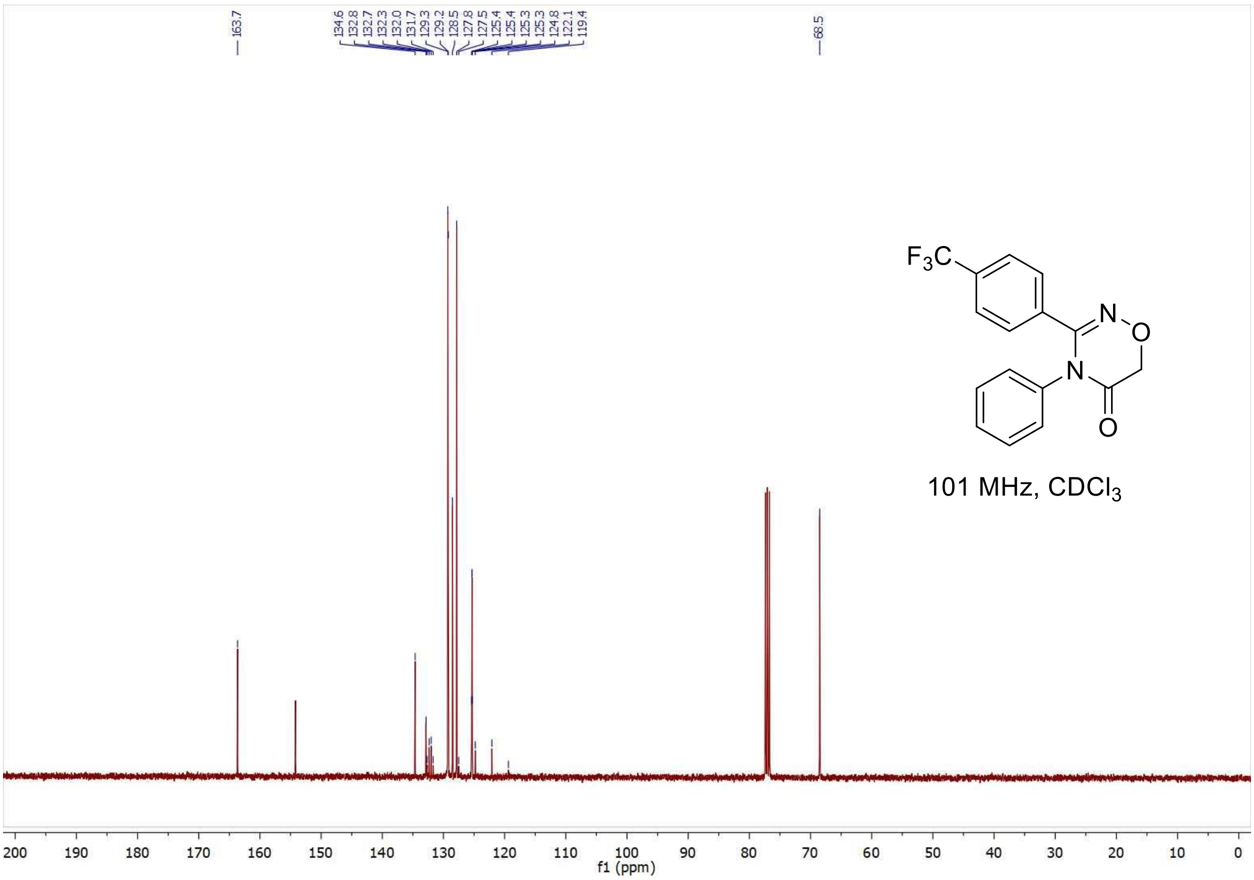
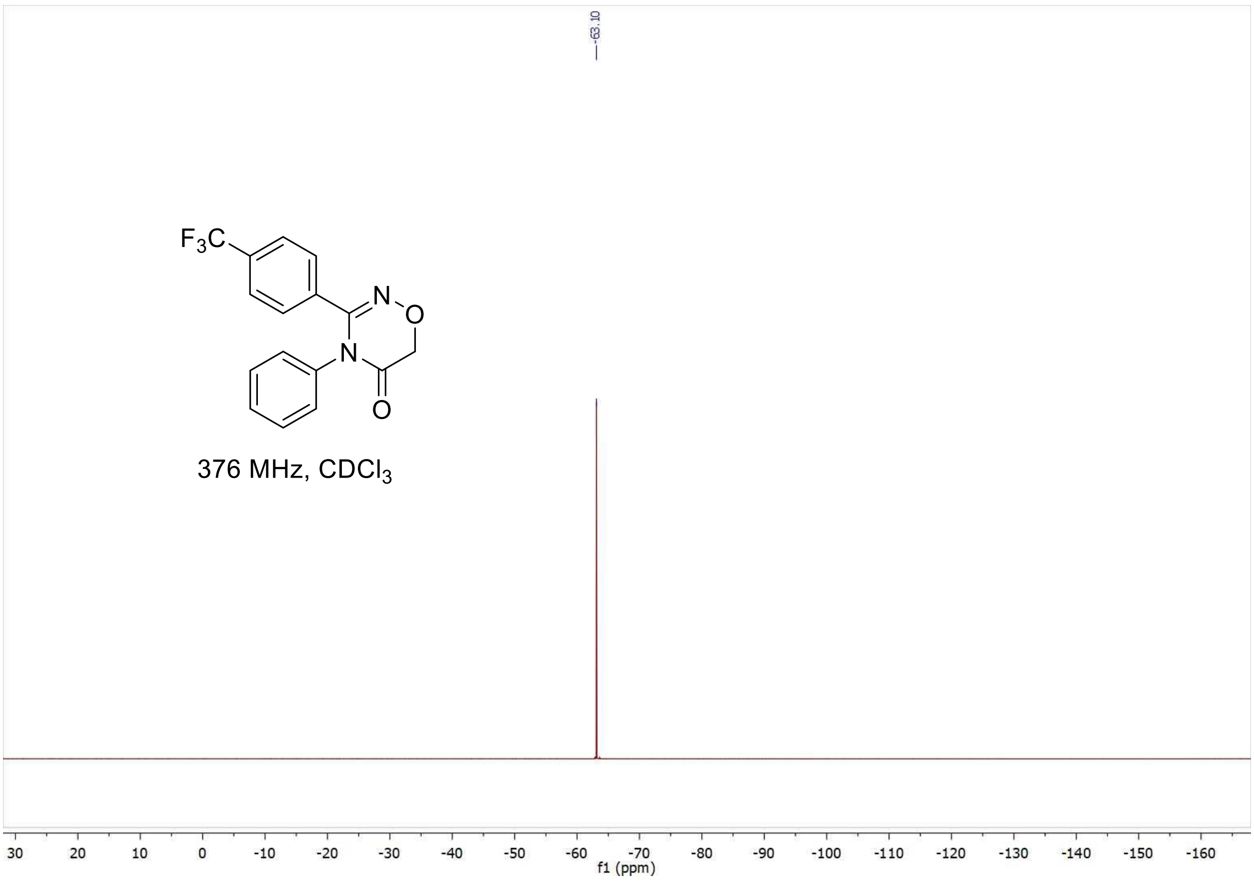
f1 (ppm)

^{1}H, ^{13}C and ^{19}F spectra of 4-phenyl-3-(4-(trifluoromethyl)phenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3g).**



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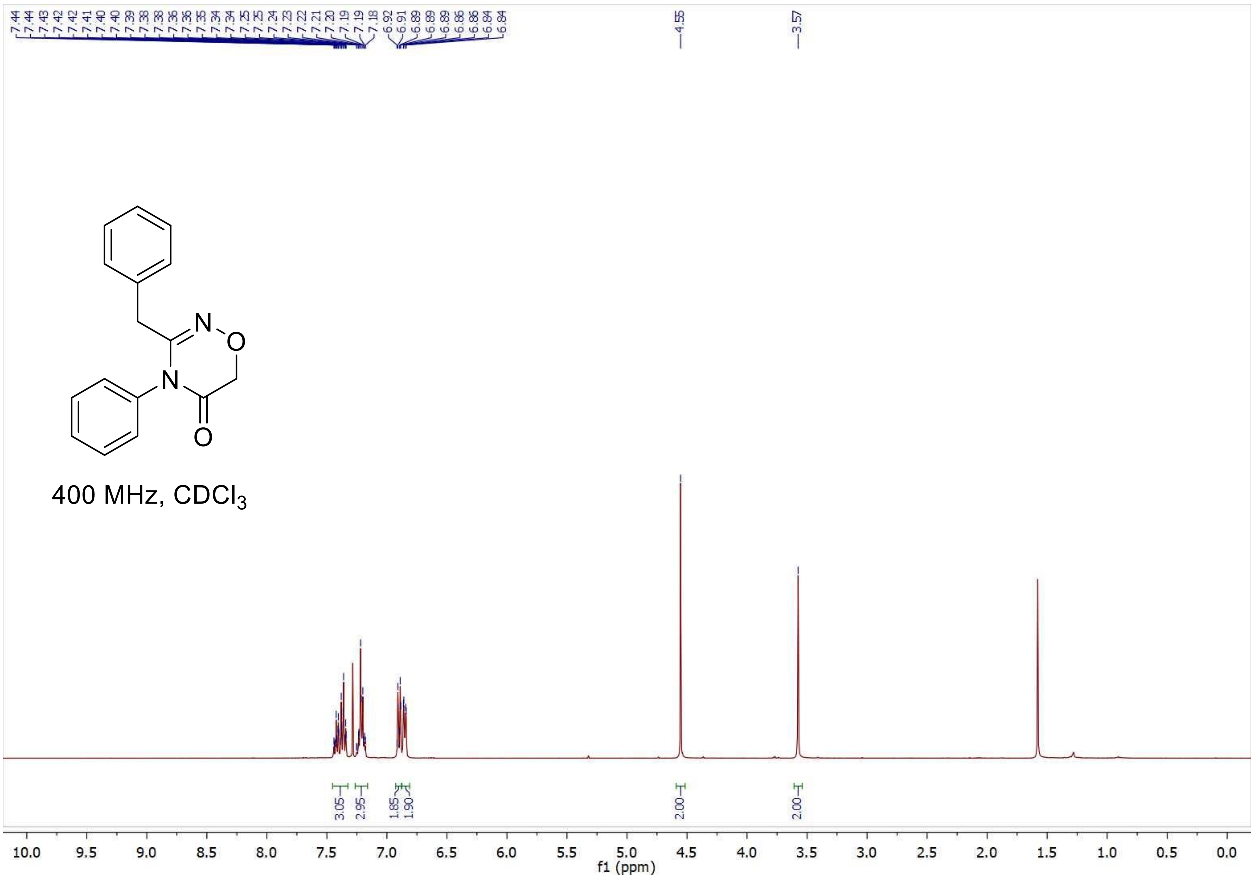
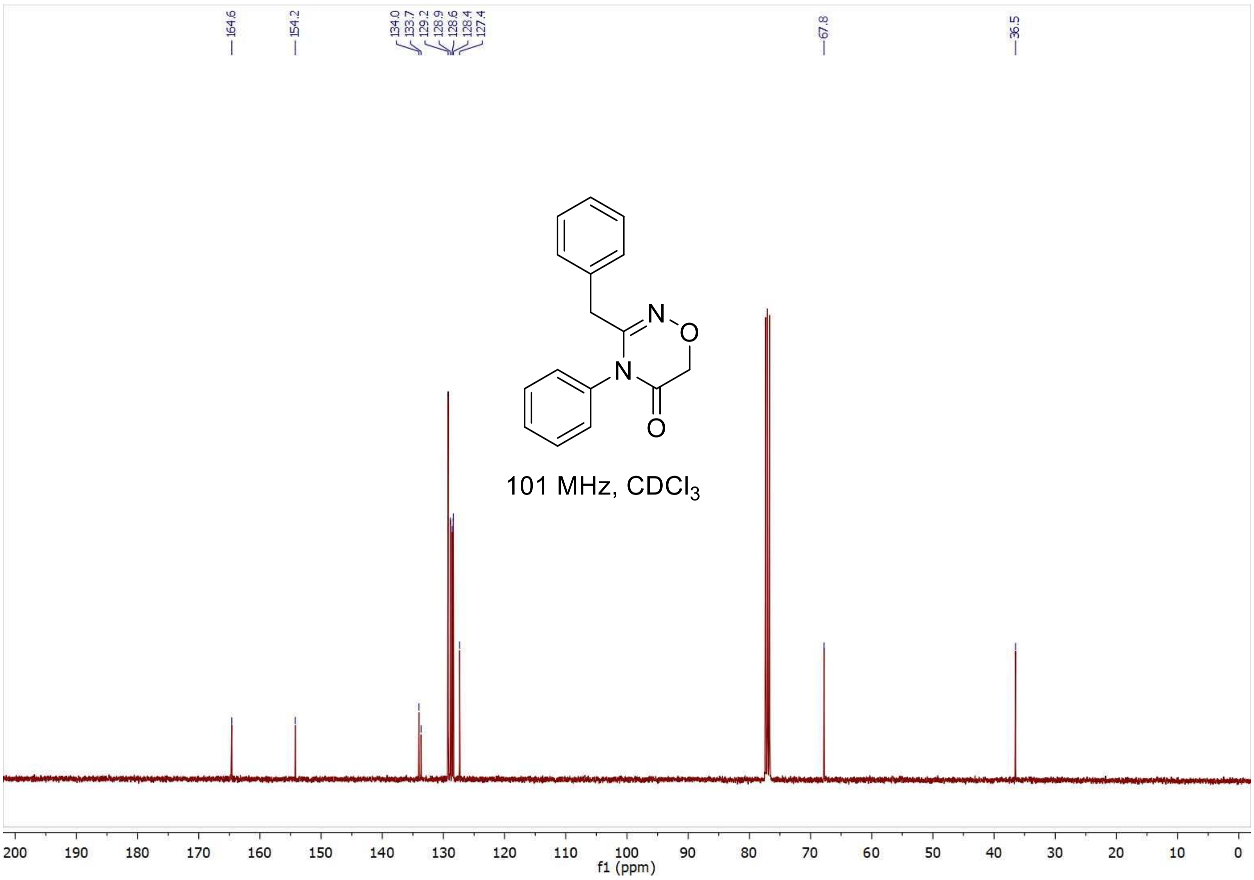
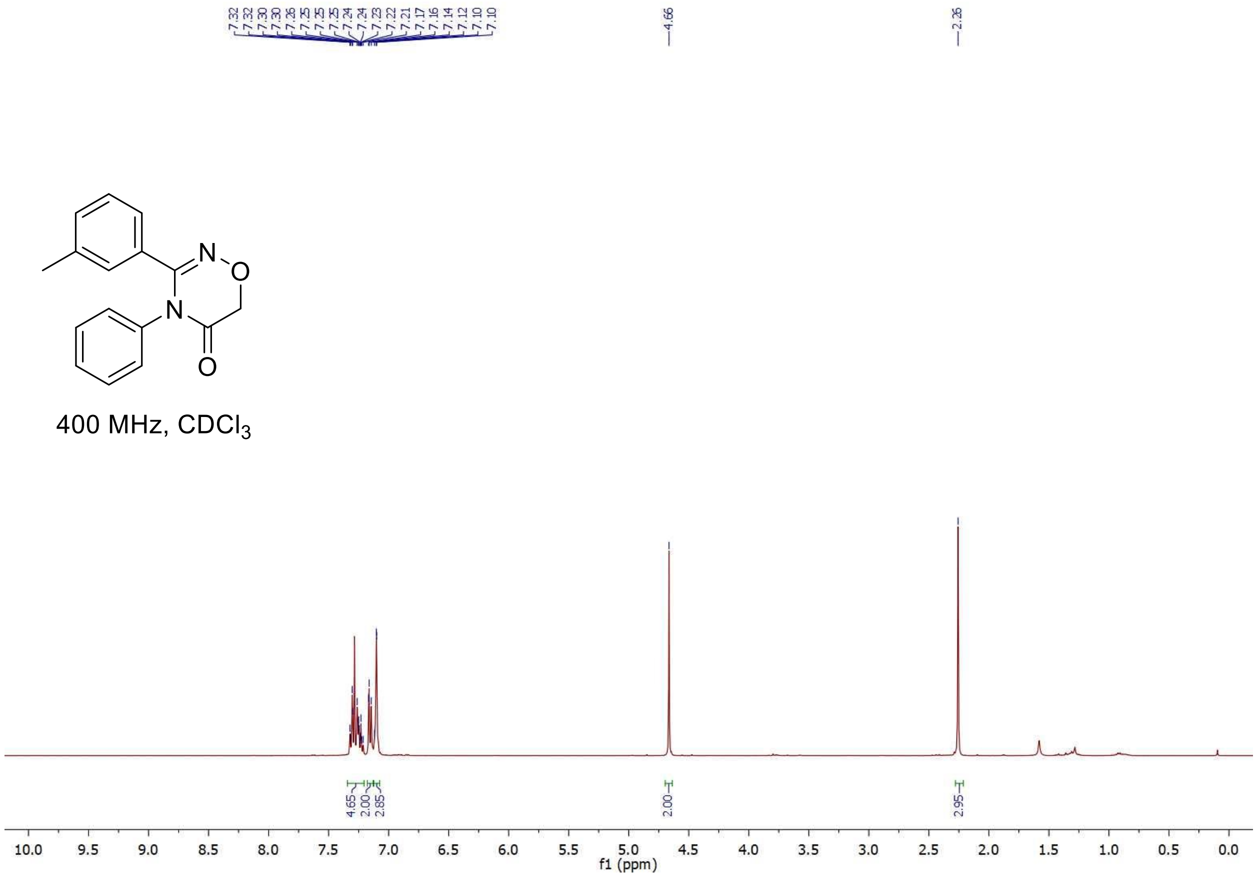
376 MHz, CDCl3
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

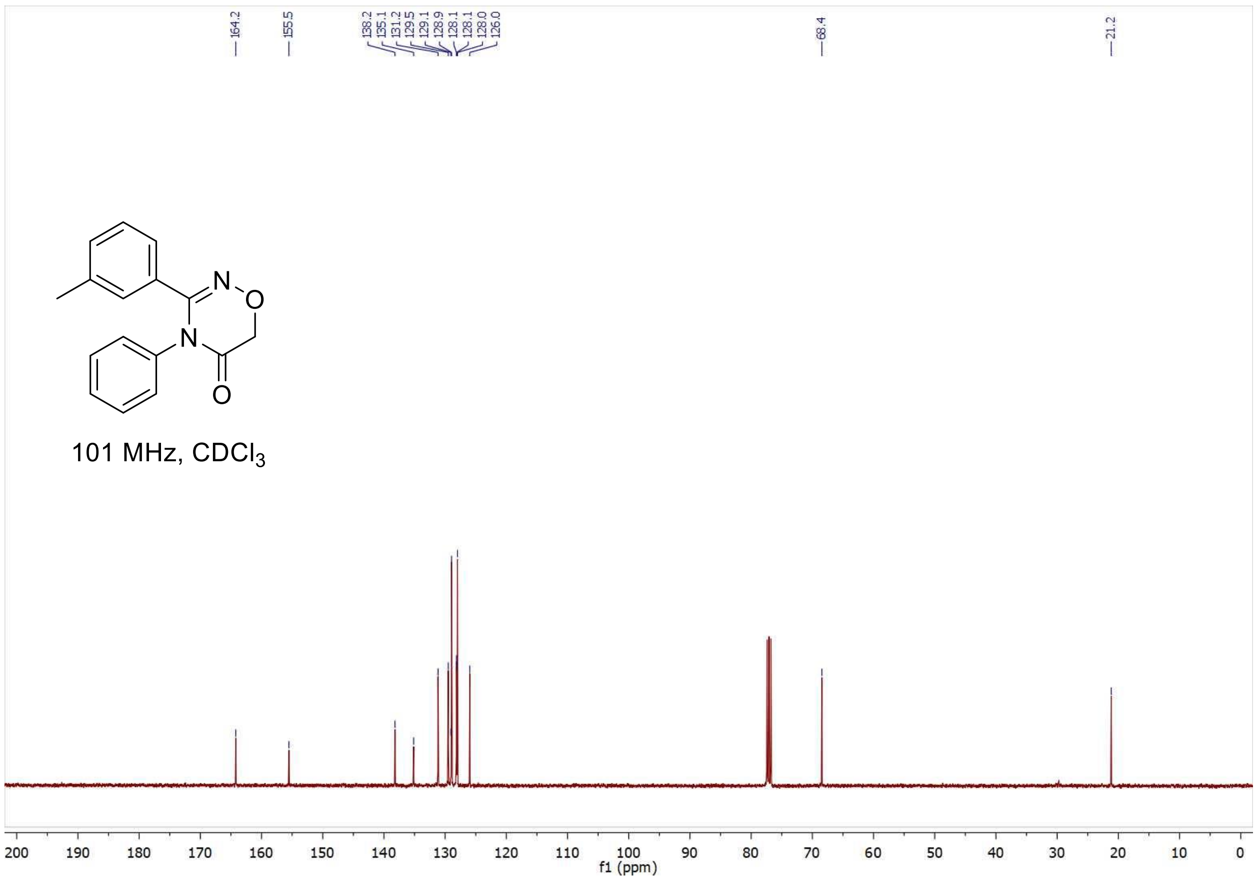
^{1}H and ^{13}C spectra of 3-benzyl-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3h).**



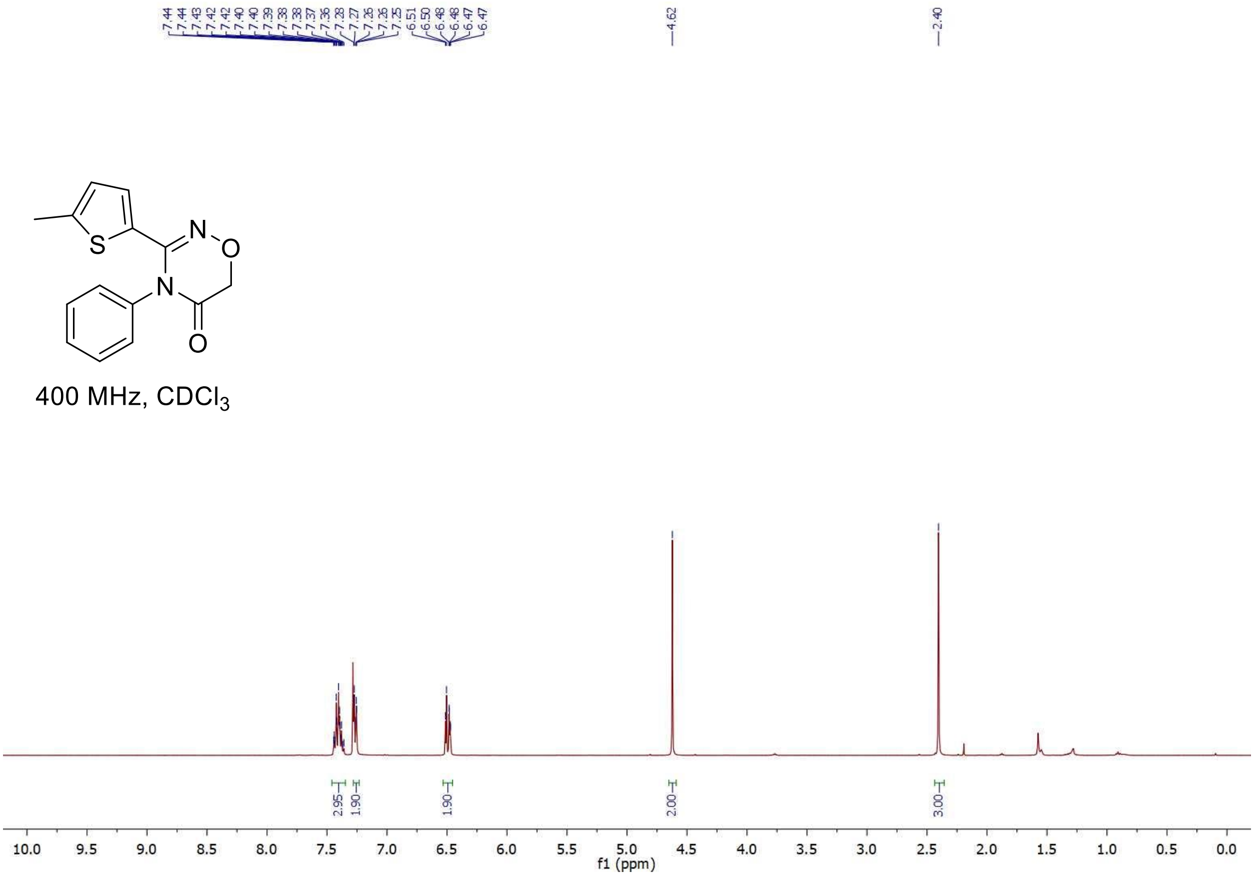


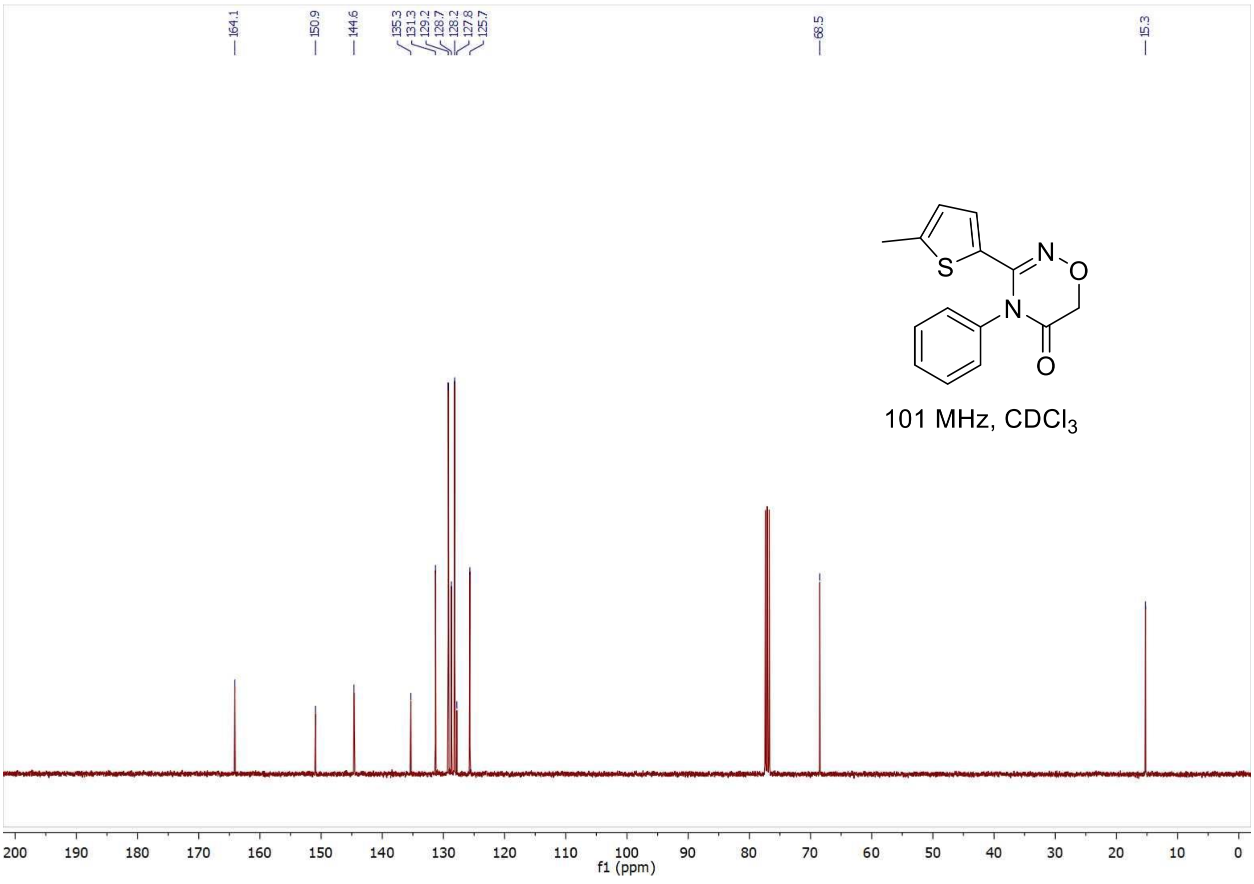
[1]H and [13]C spectra of 4-phenyl-3-(3-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3i).**



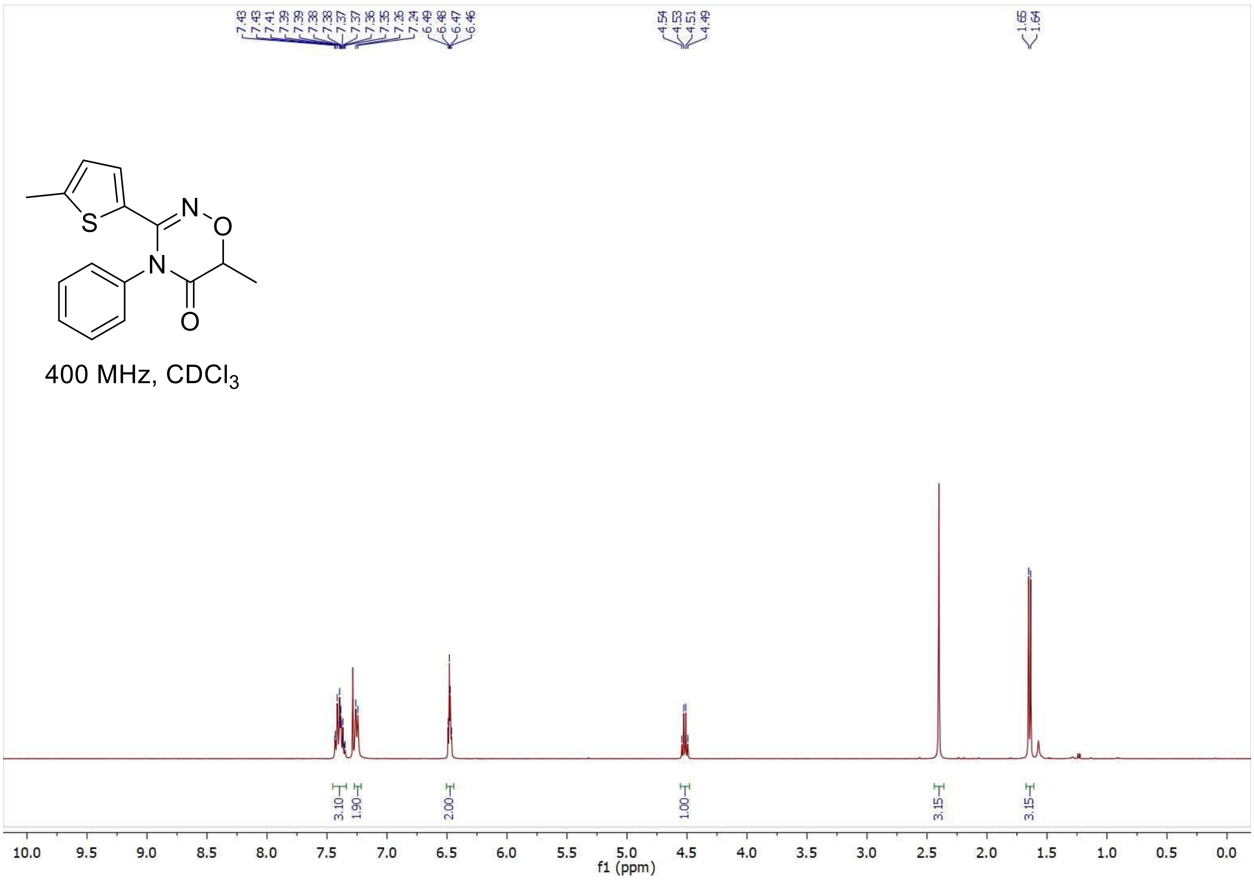


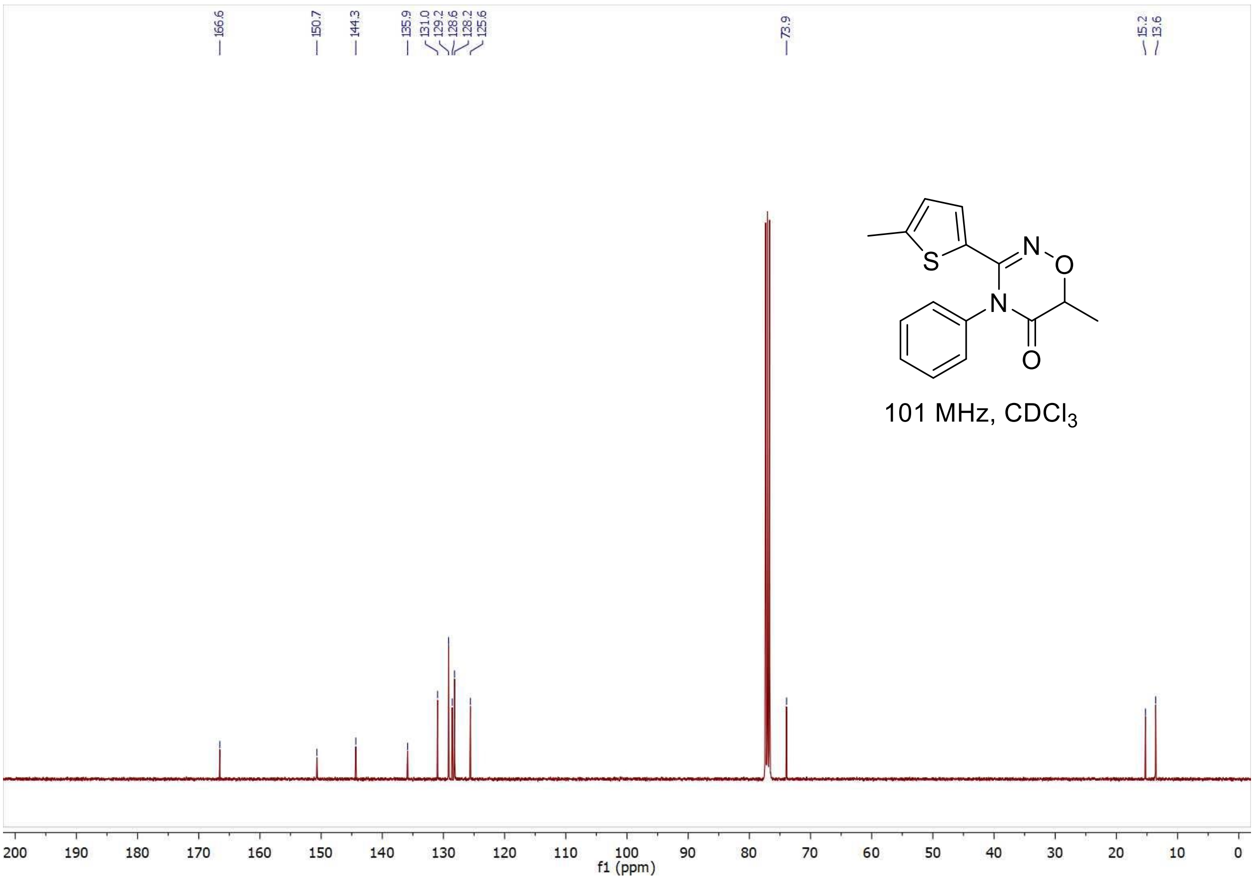
^{1}H and ^{13}C spectra of 3-(5-methylthiophen-2-yl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3j).**



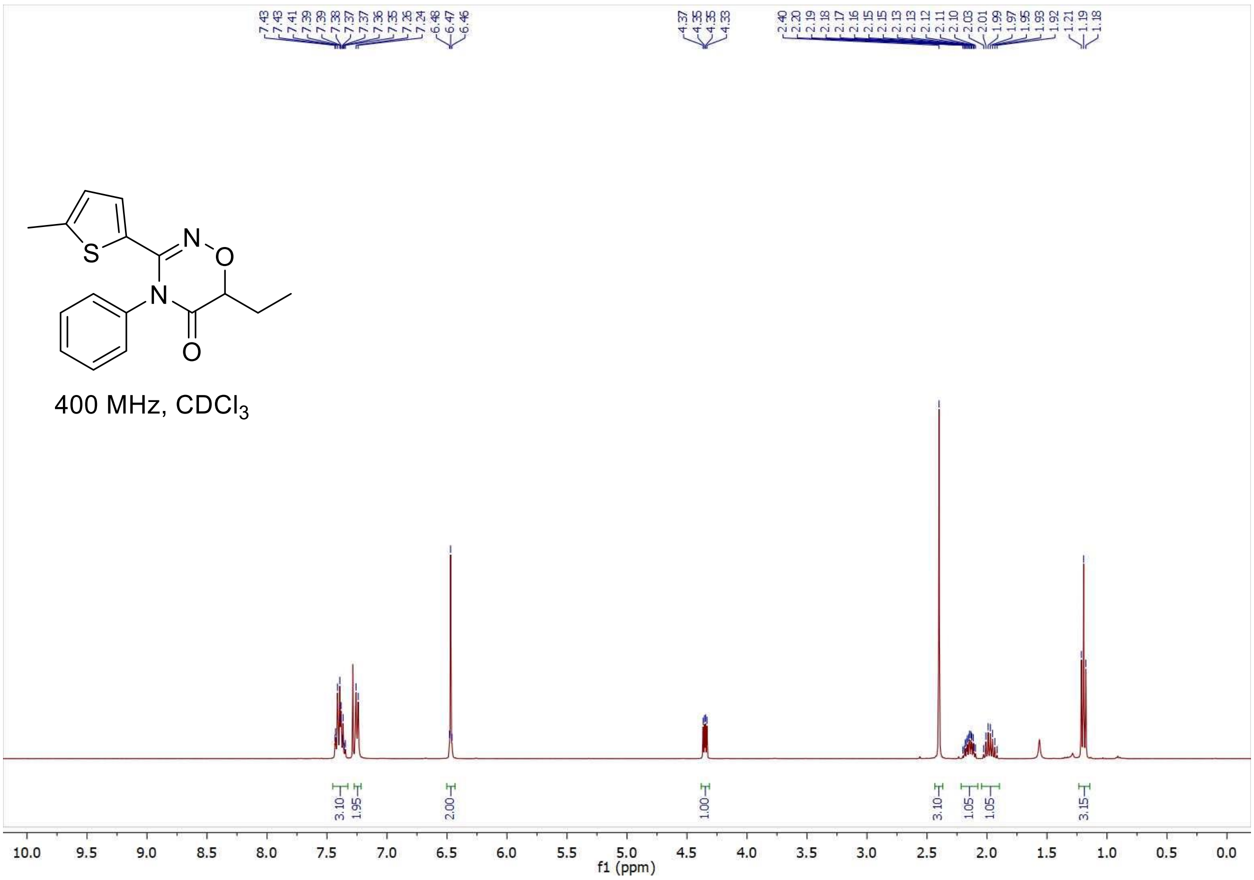


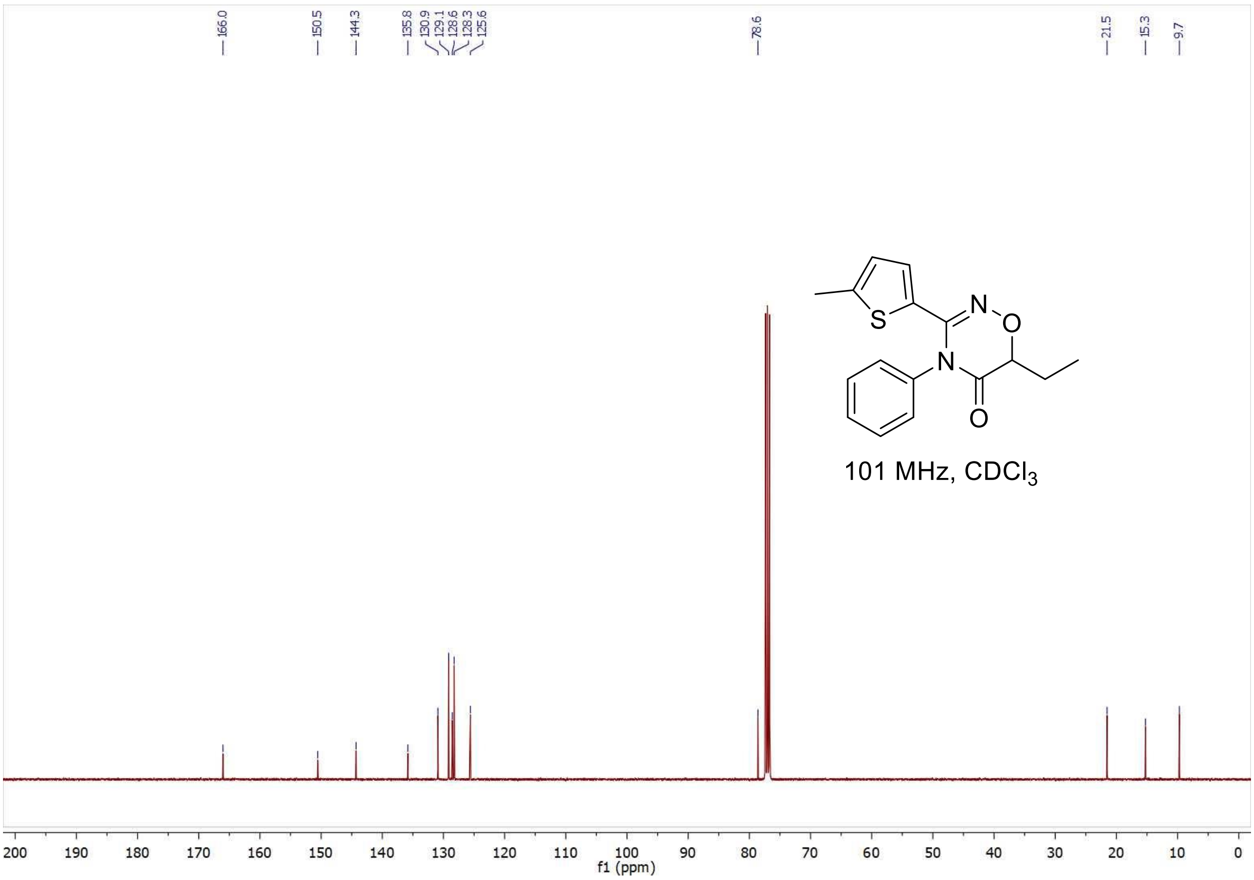
^{1}H and ^{13}C spectra of 6-methyl-3-(5-methylthiophen-2-yl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3k).**



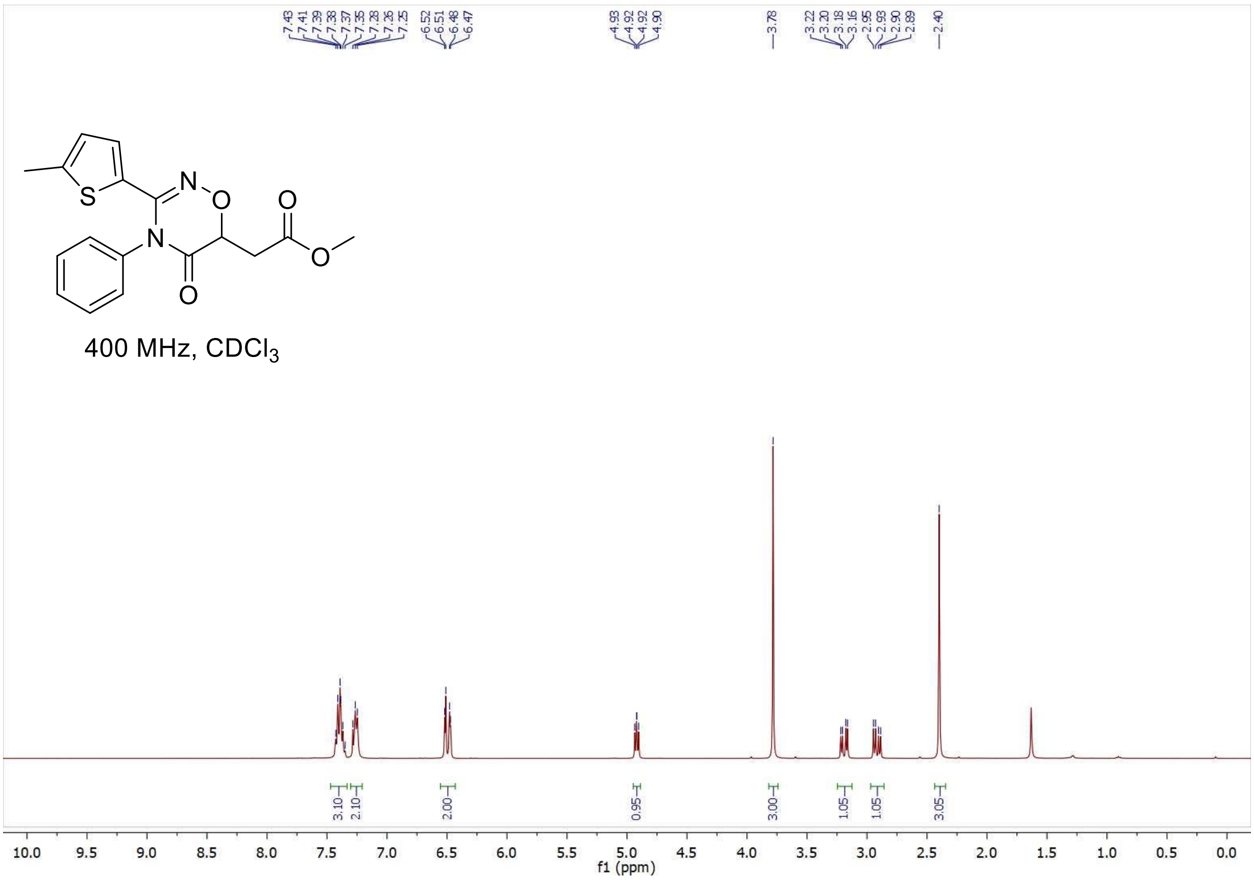


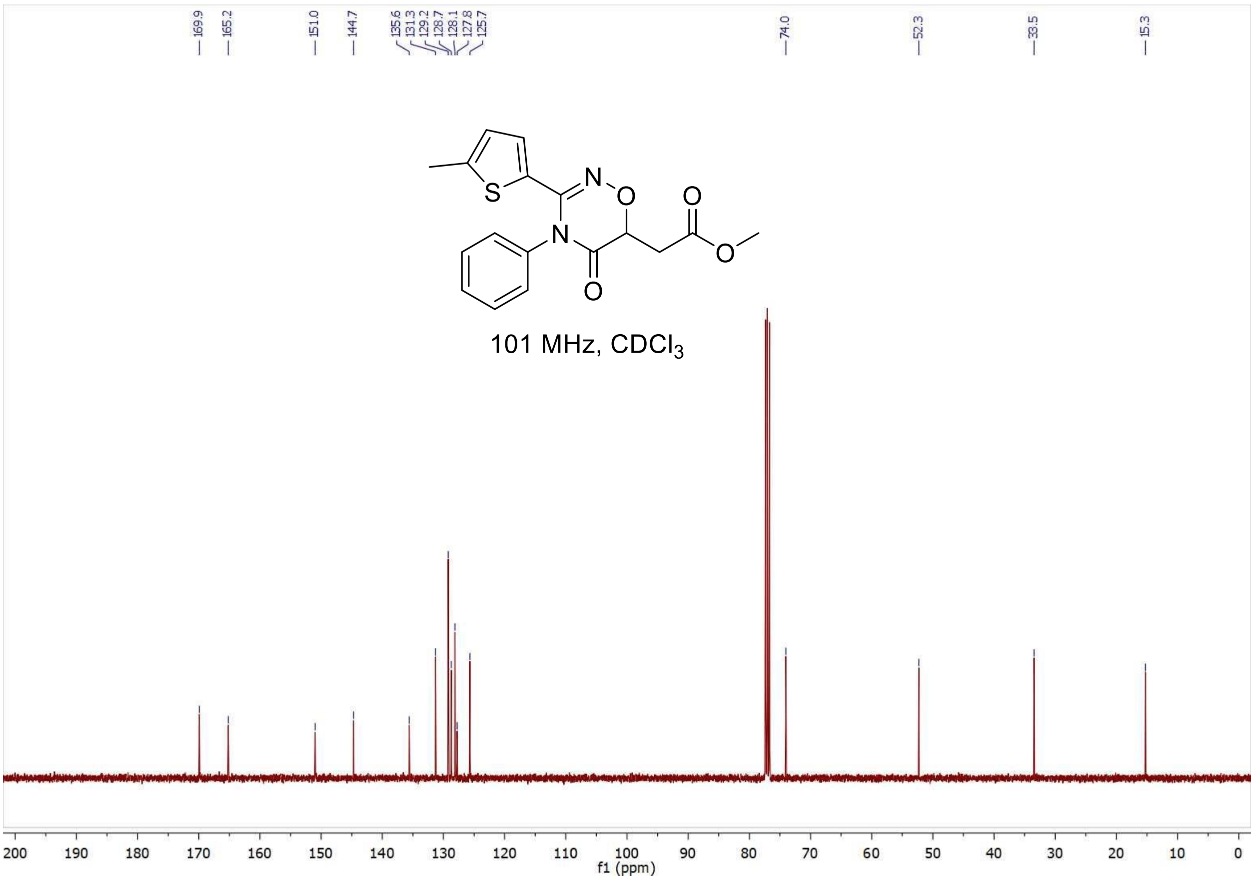
[1]H and [13]C spectra of 6-ethyl-3-(5-methylthiophen-2-yl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3l).**



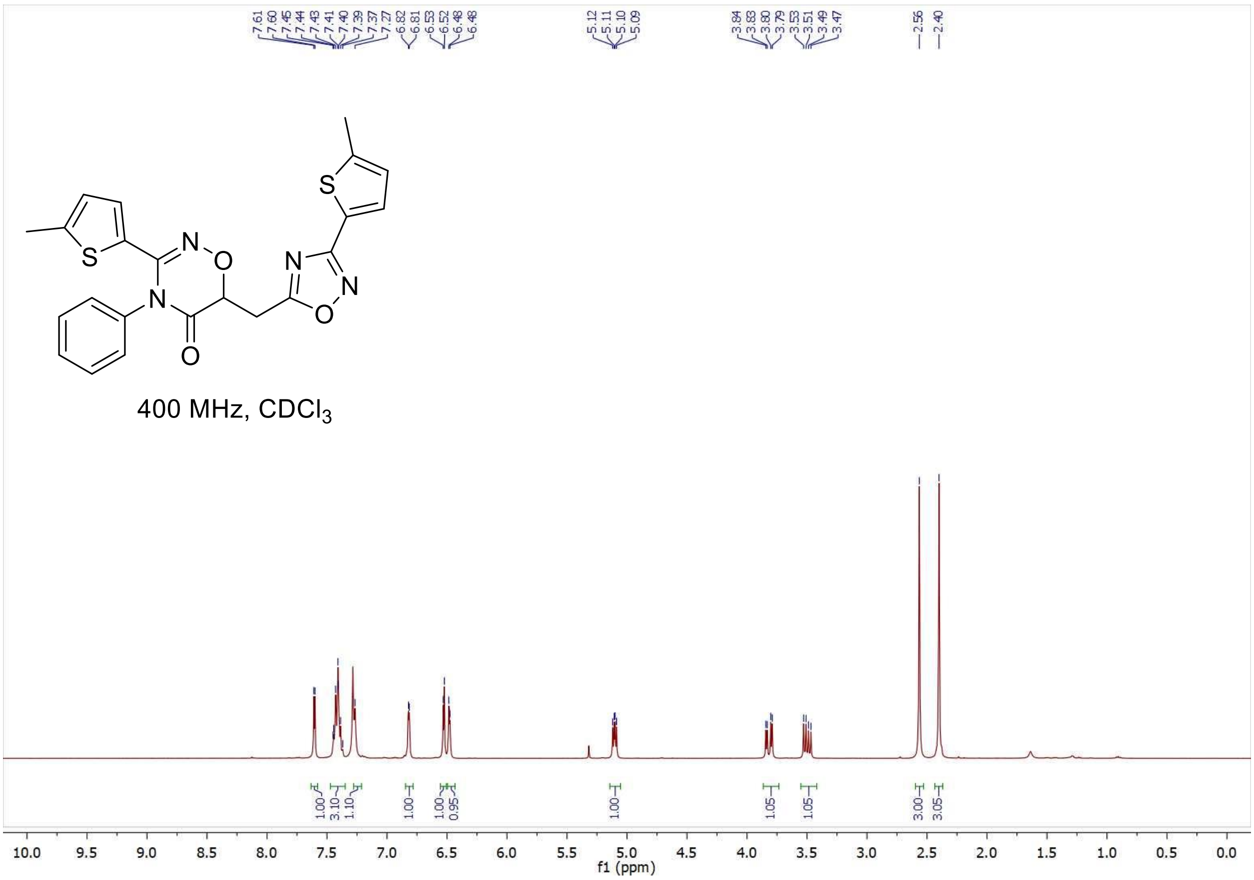


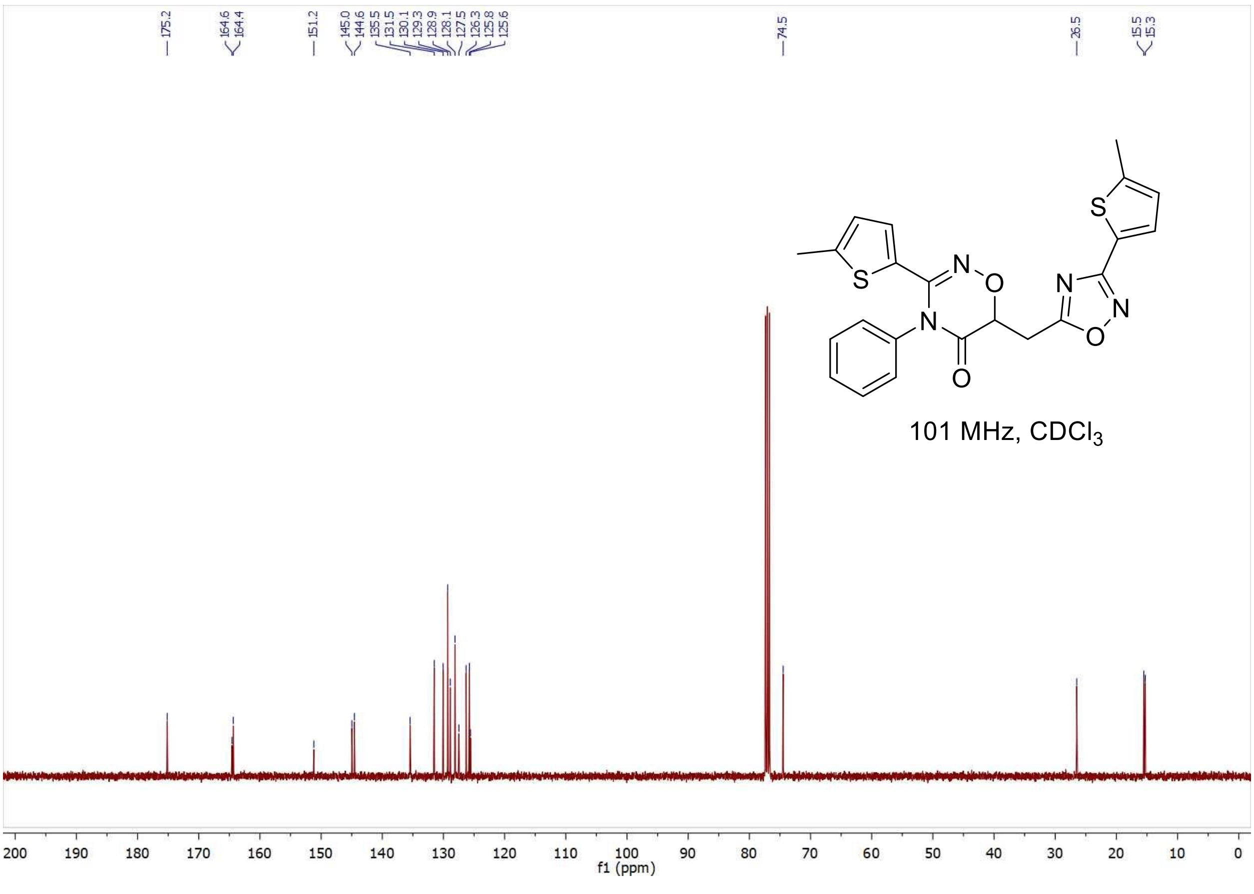
[1]H and [13]C spectra of methyl 2-(3-(5-methylthiophen-2-yl)-5-oxo-4-phenyl-5,6-dihydro-4*H*-1,2,4-oxadiazin-6-yl)acetate **(3m).**



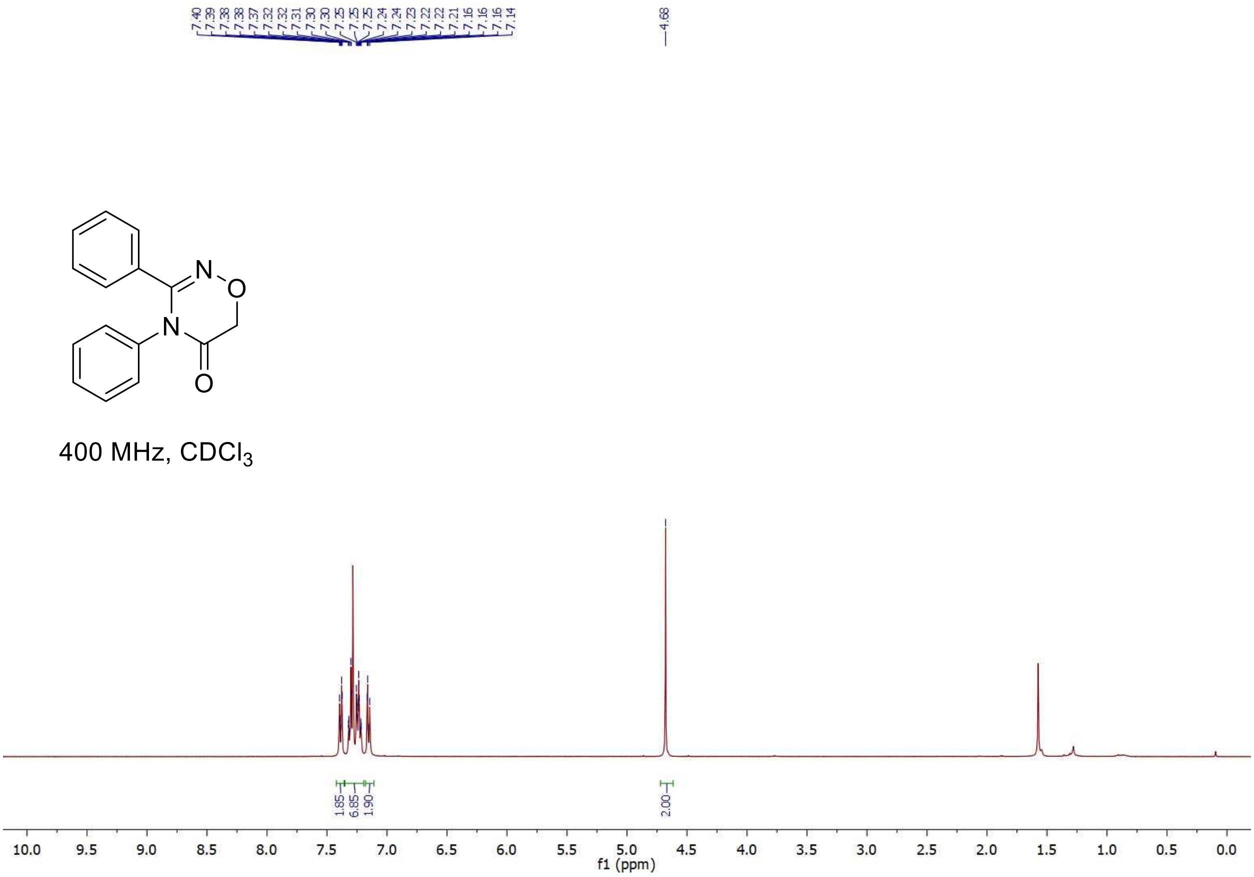


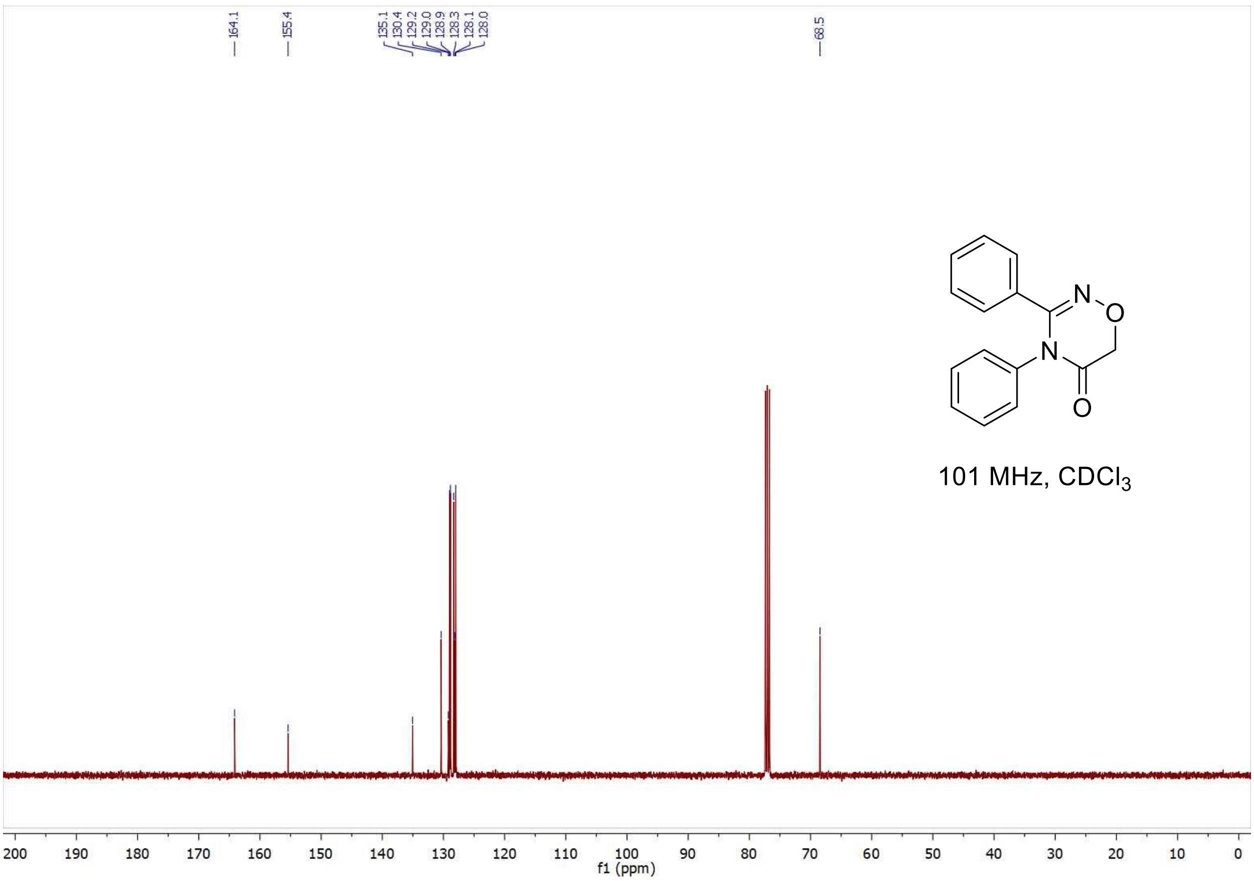
[1]H and [13]C spectra of 3-(5-methylthiophen-2-yl)-6-((3-(5-methylthiophen-2-yl)-1,2,4-oxadiazol-5-yl)methyl)-4-phenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3n).**



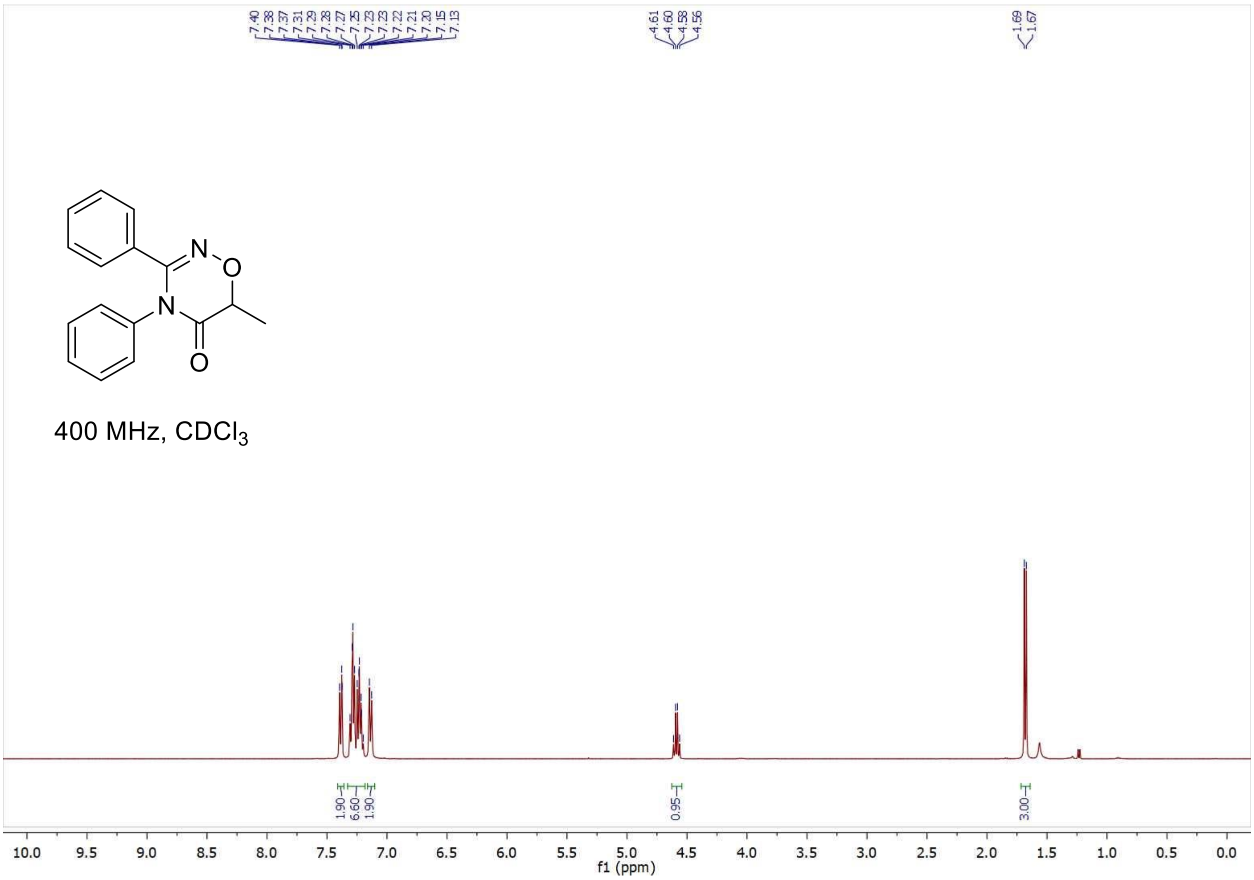


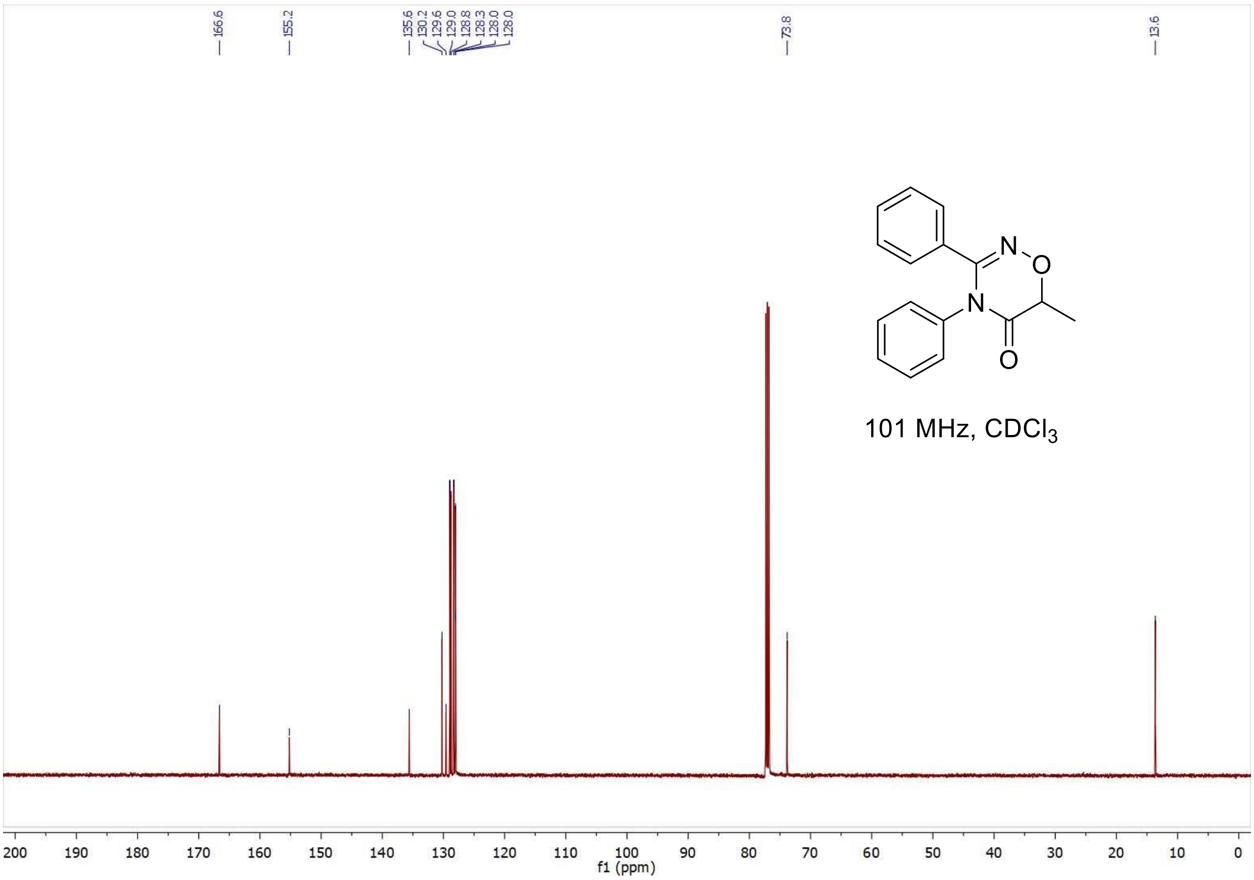
^{1}H and ^{13}C spectra of methyl 3,4-diphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3o).**



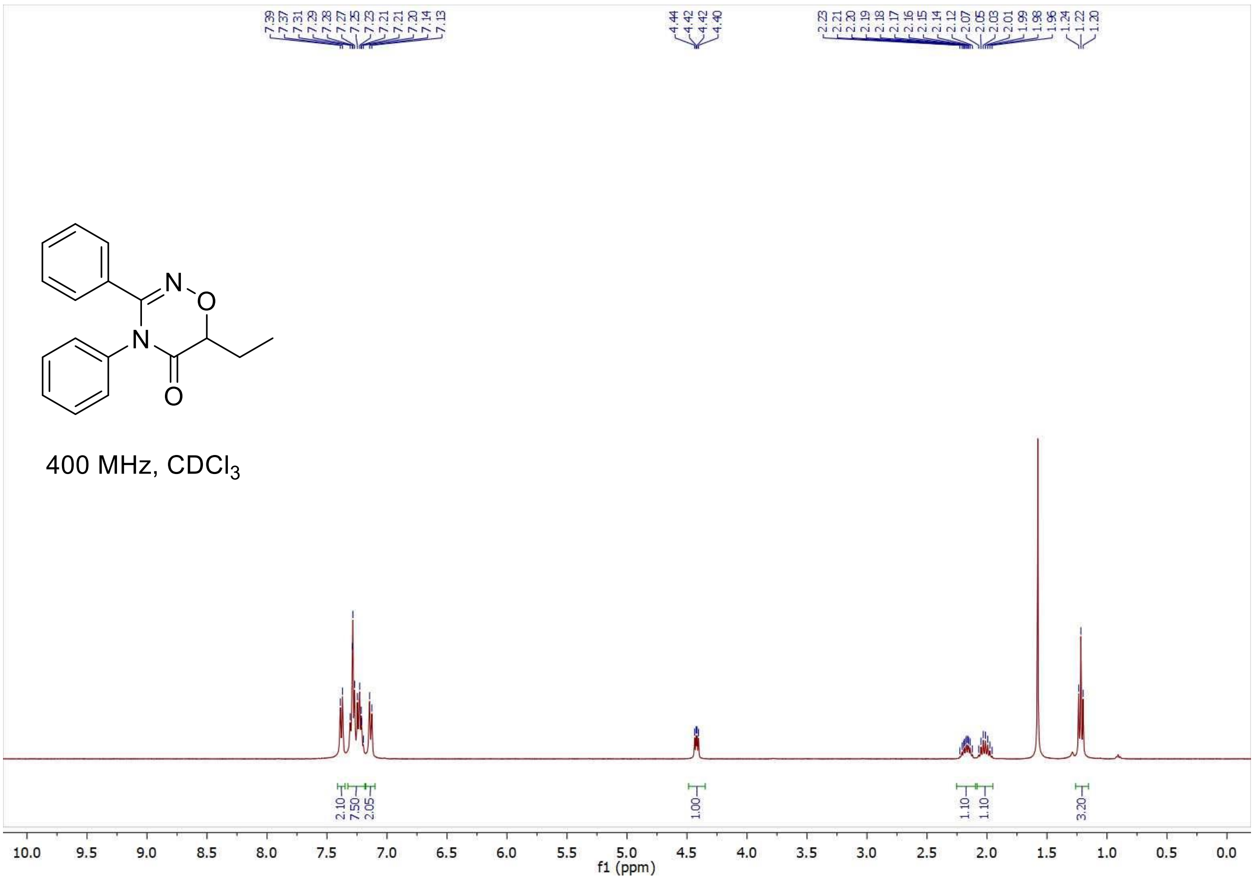


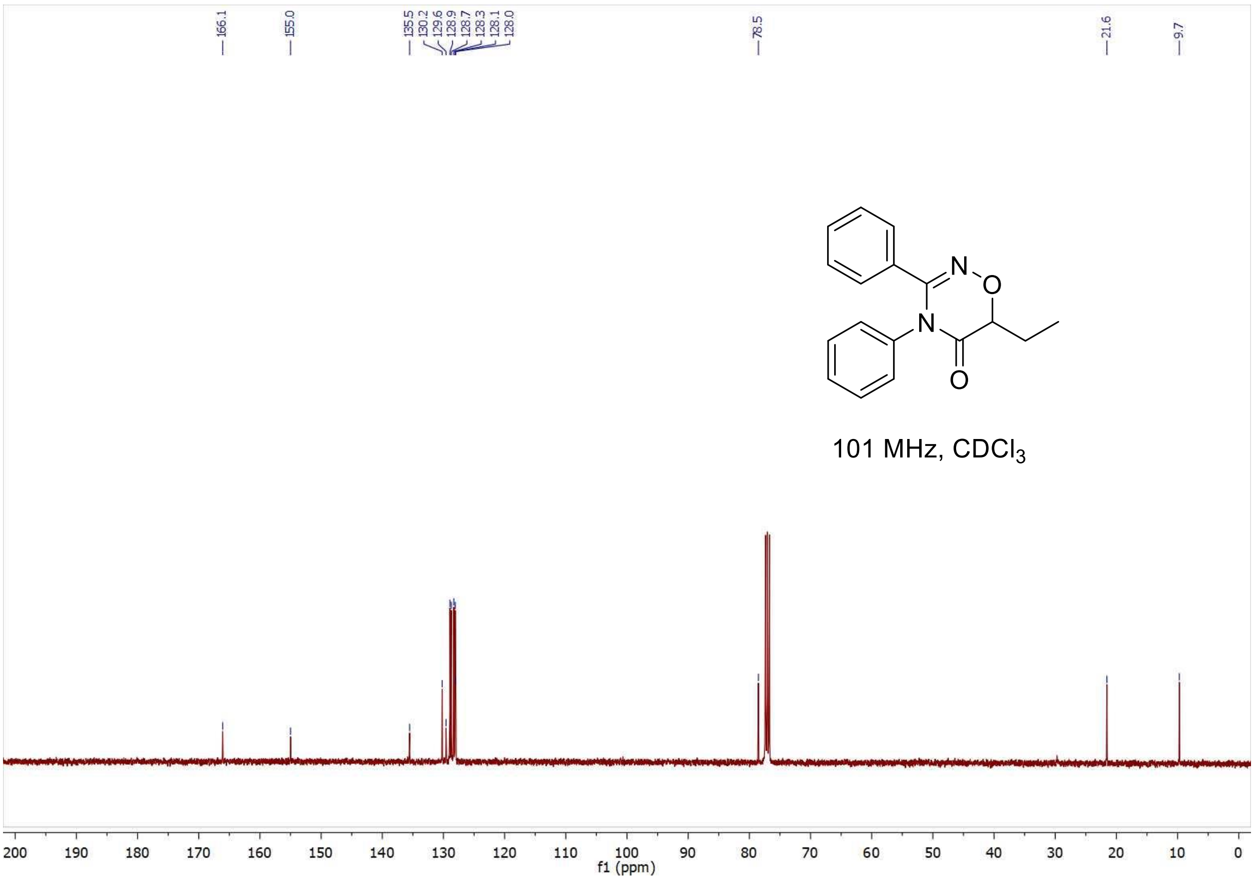
[1]H and [13]C spectra of methyl 6-methyl-3,4-diphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3p).**



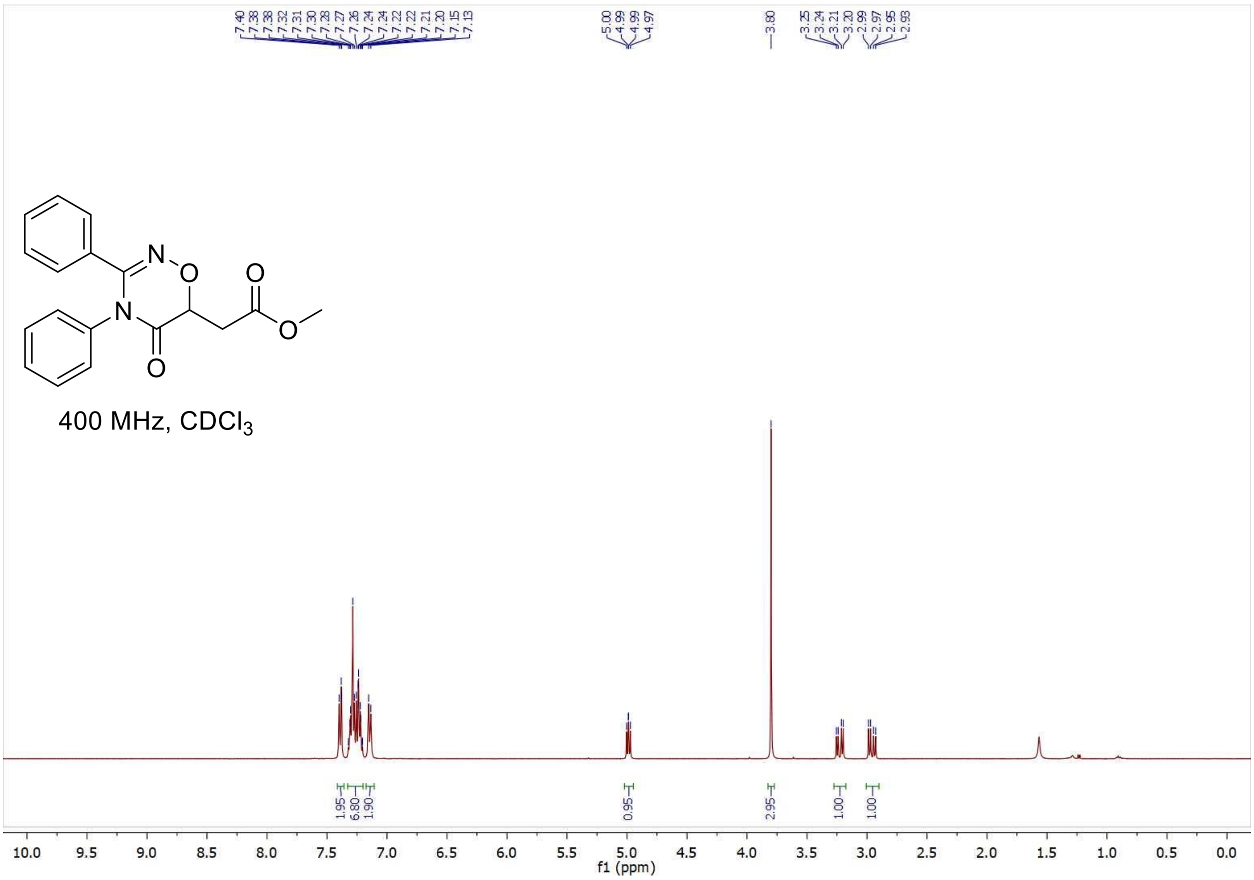


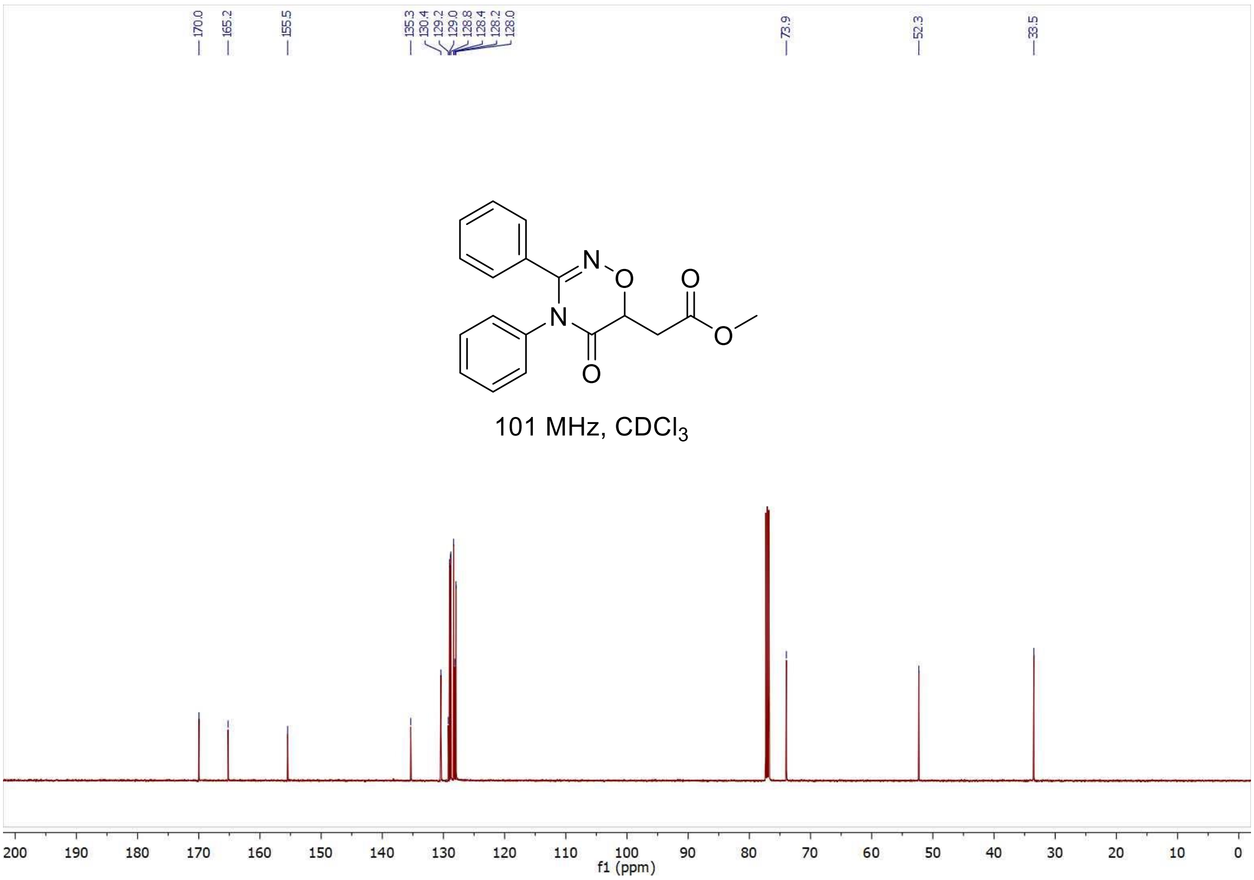
[1]H and [13]C spectra of methyl 6-ethyl-3,4-diphenyl-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(3q).**



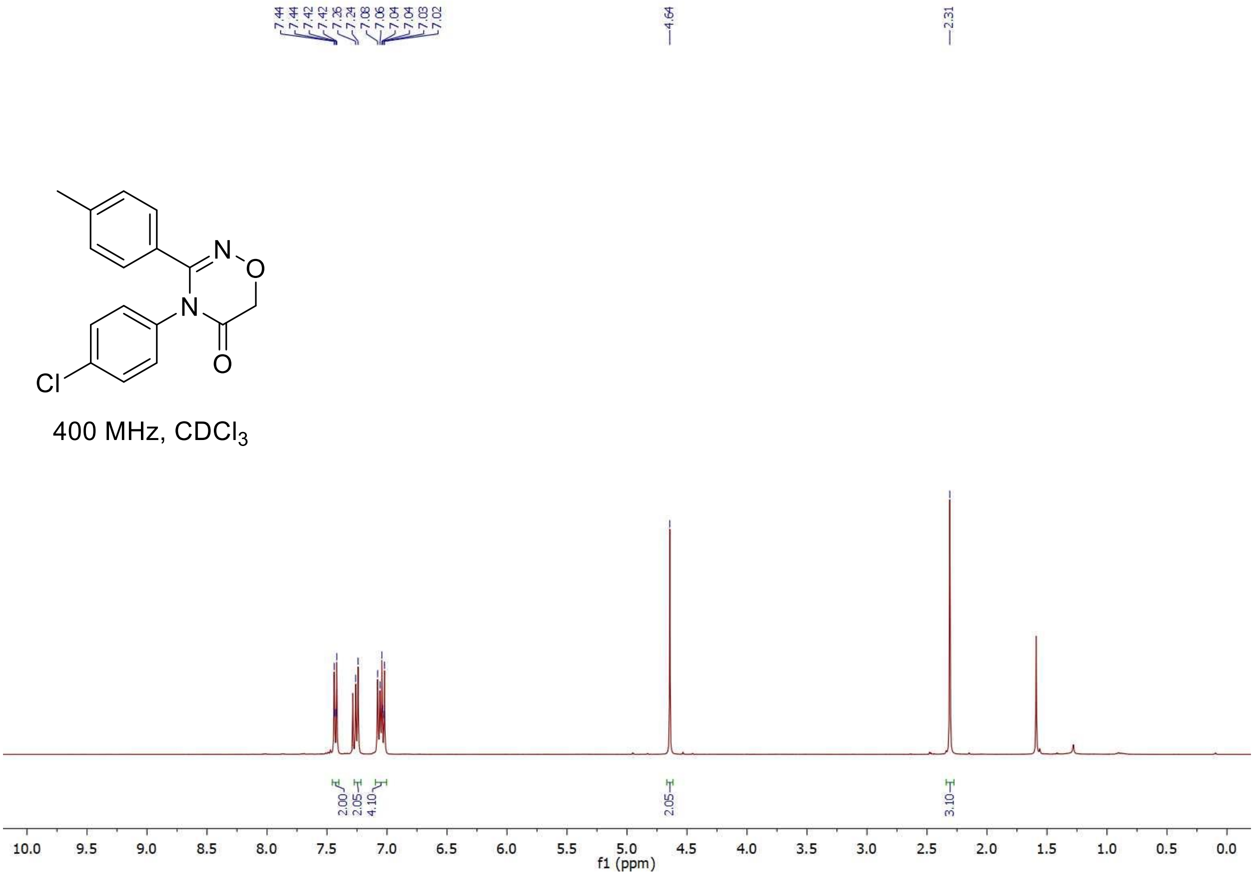


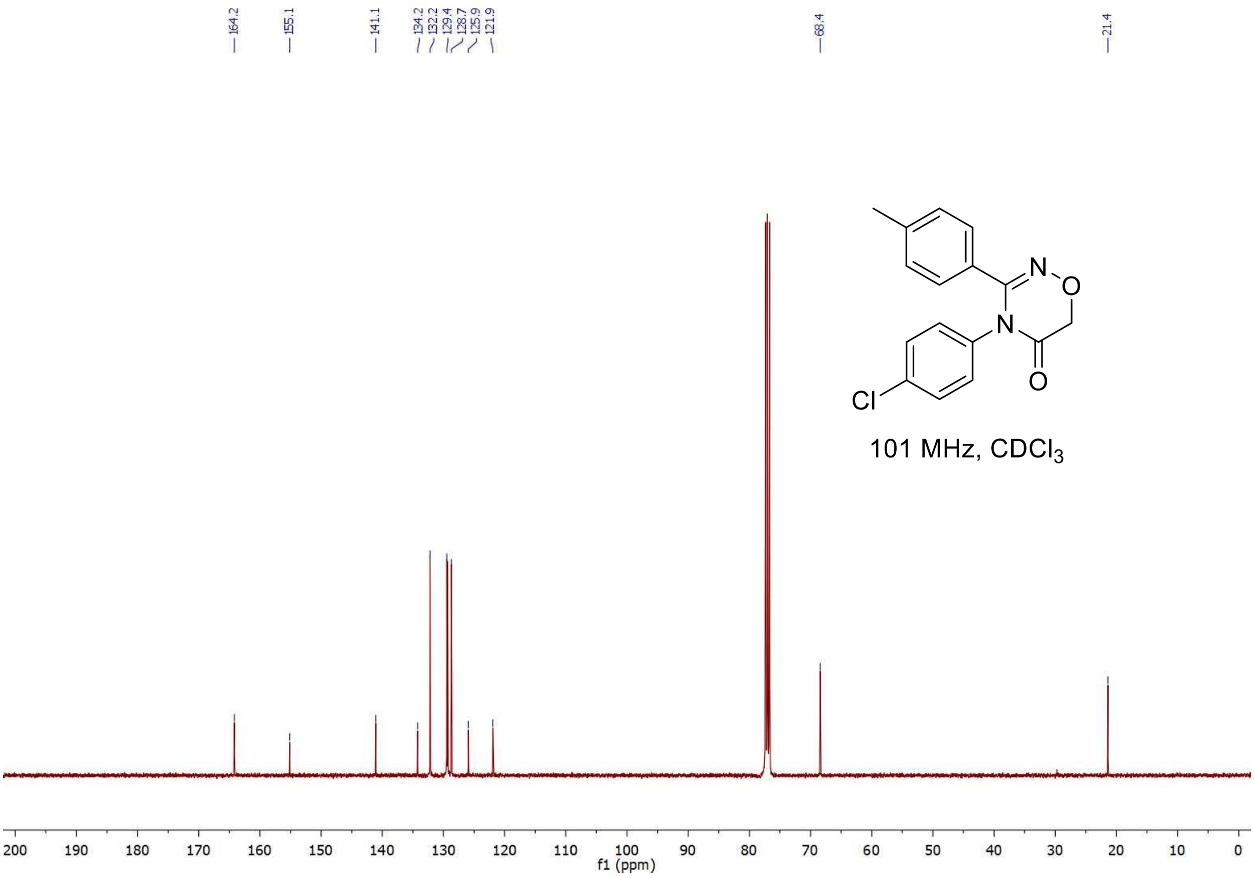
^{1}H and ^{13}C spectra of methyl 2-(5-oxo-3,4-diphenyl-5,6-dihydro-4*H*-1,2,4-oxadiazin-6-yl)acetate **(3r).**



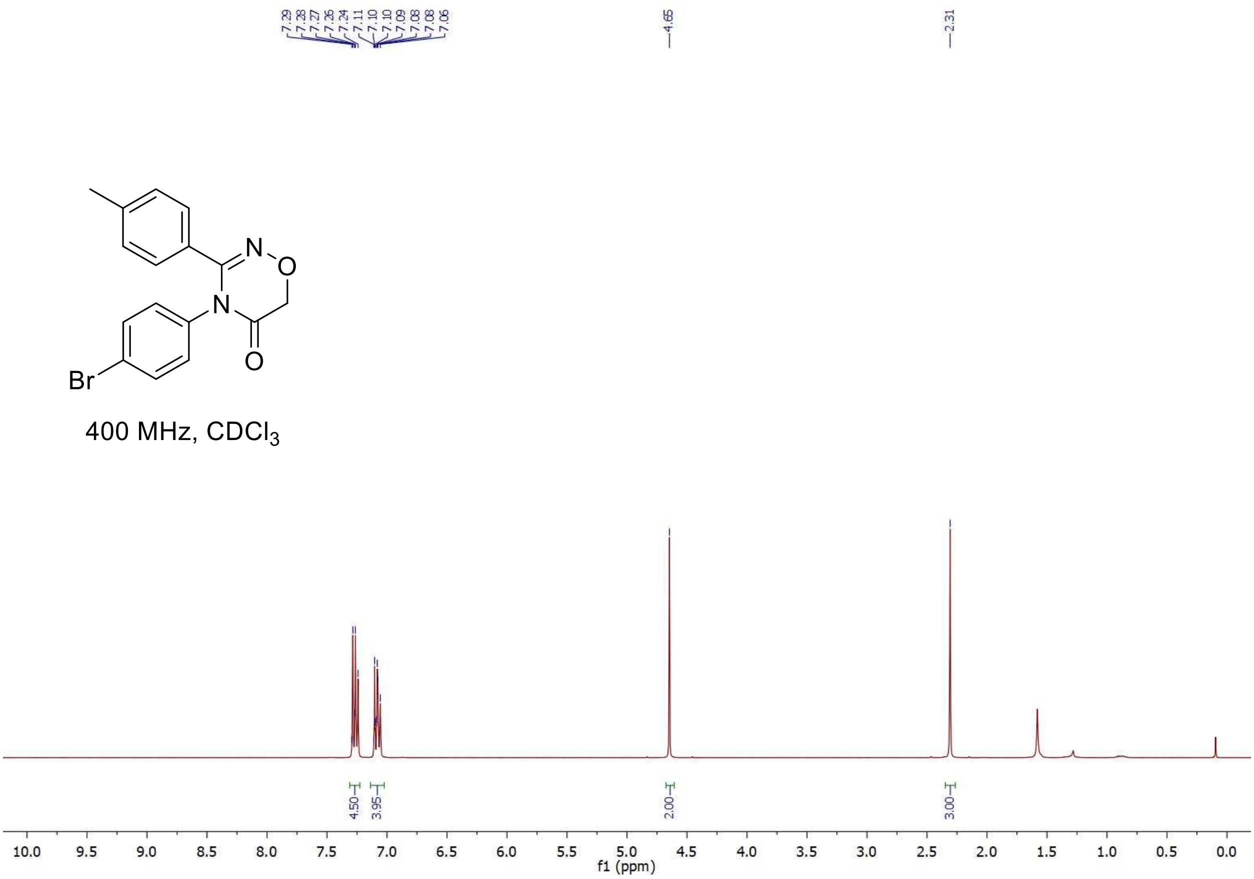


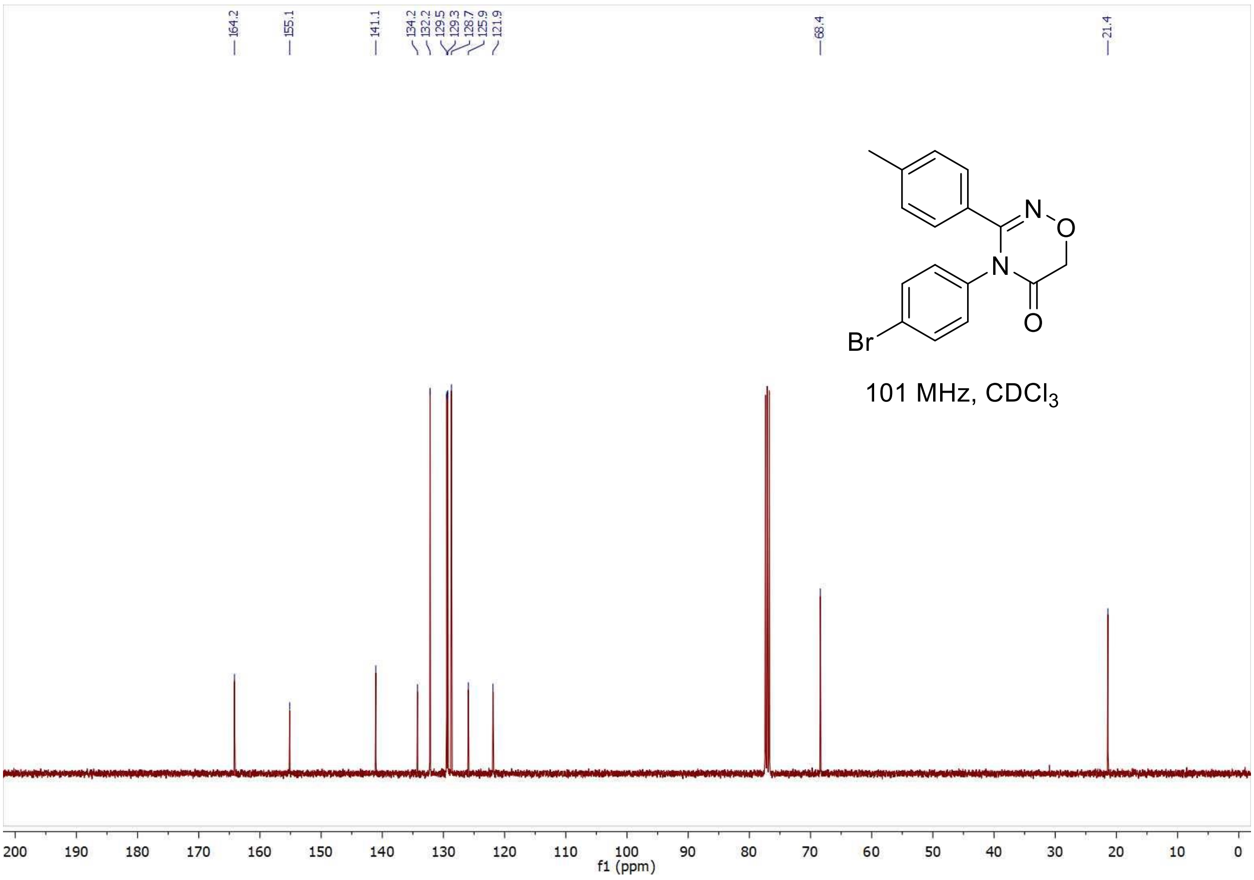
^{1}H and ^{13}C spectra of 4-(4-chlorophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4a).**



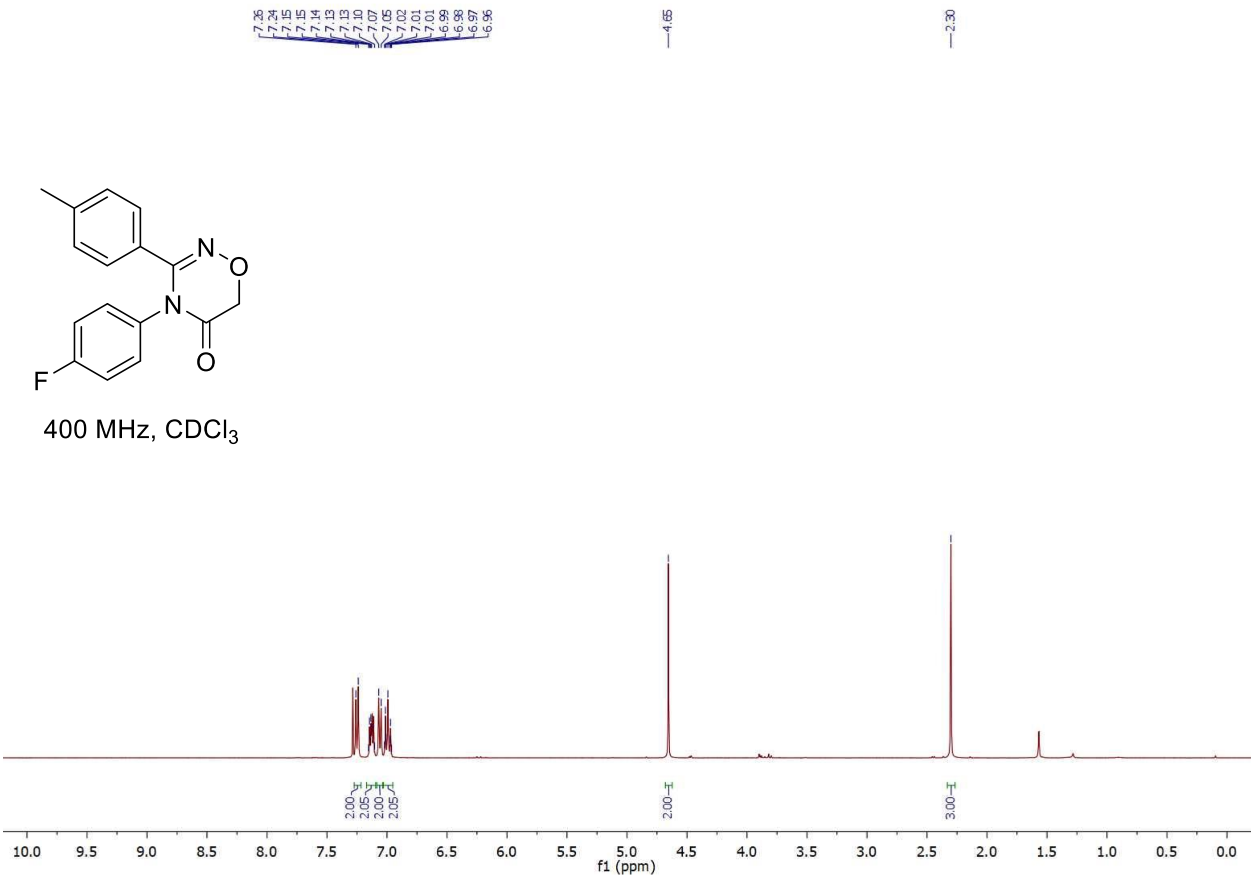


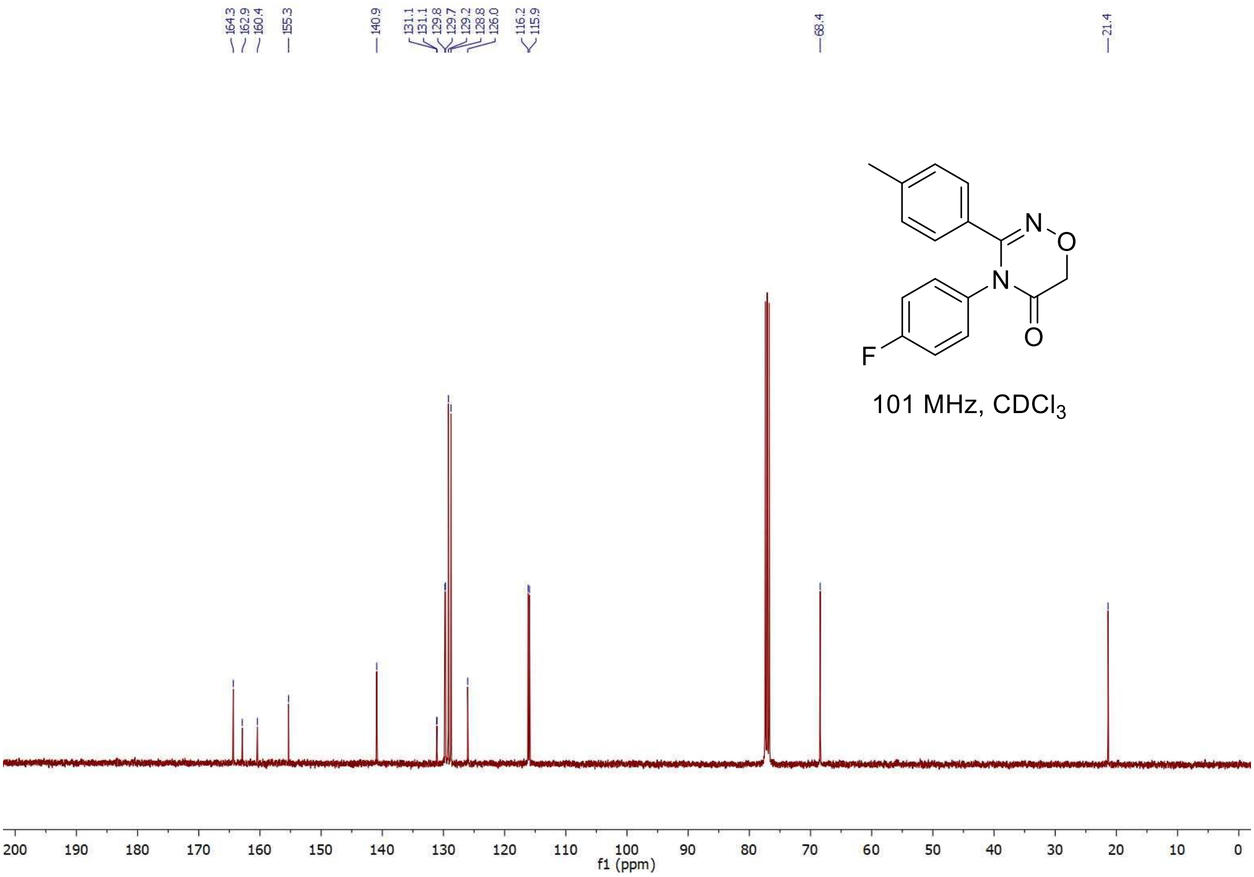
[1]H and [13]C spectra of 4-(4-bromophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4b).**

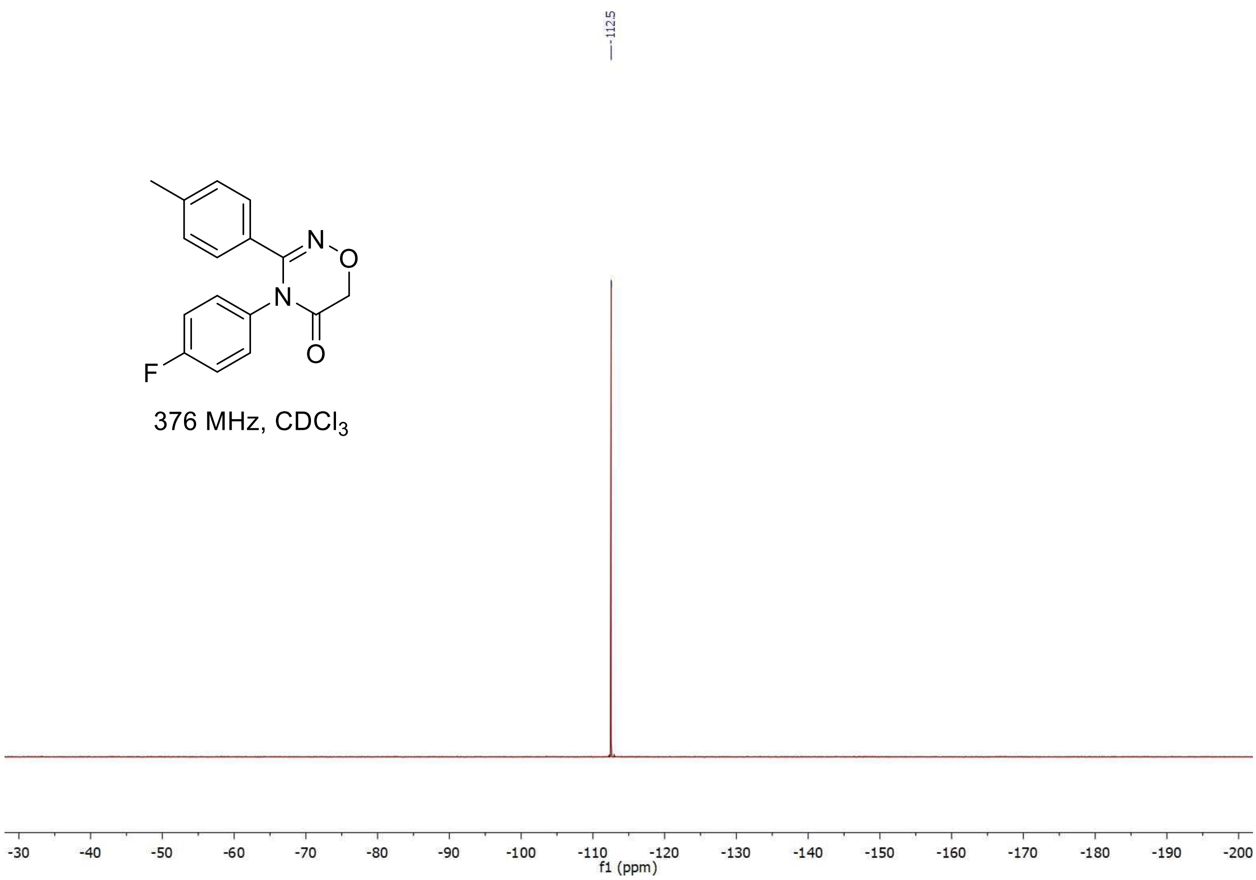




^{1}H, ^{13}C and ^{19}F spectra of 4-(4-fluorophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4c).**



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-190
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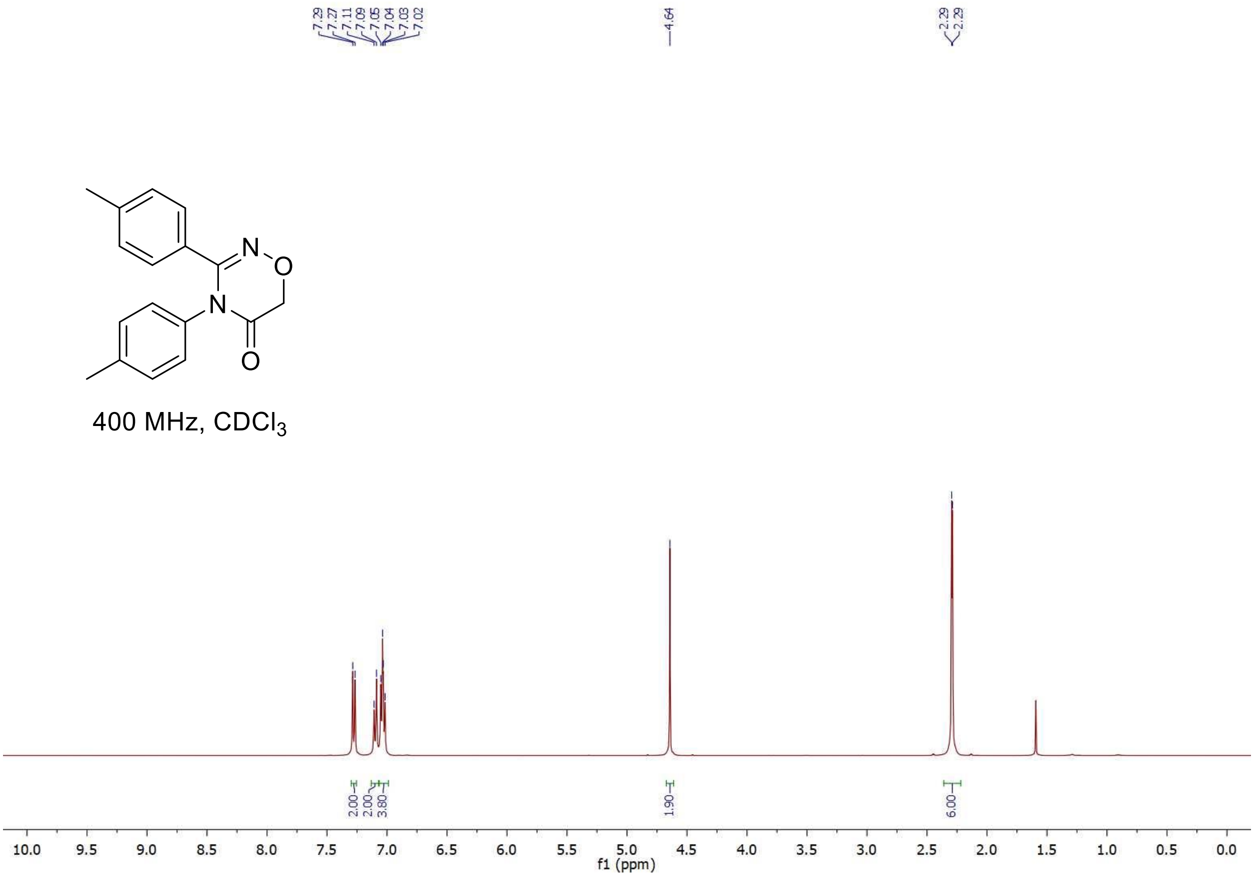
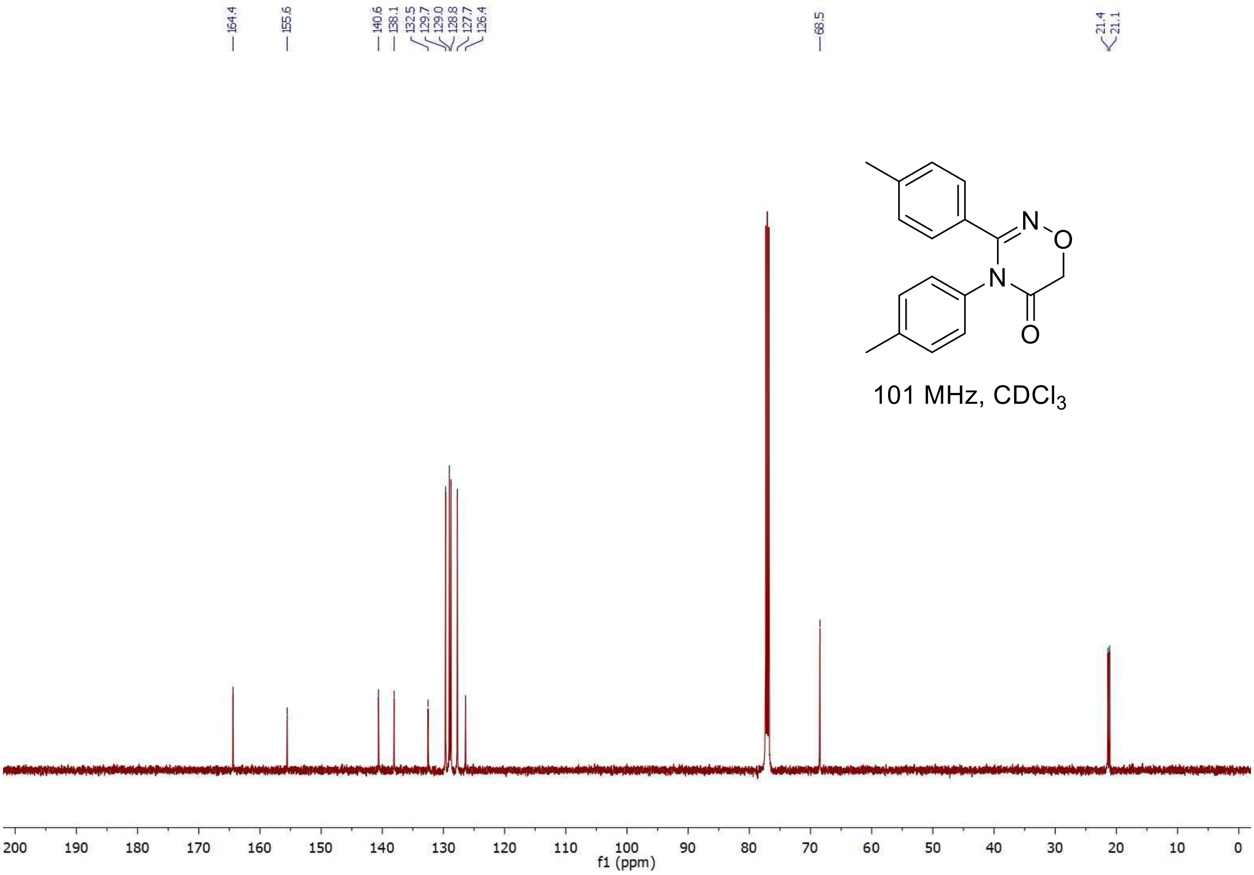
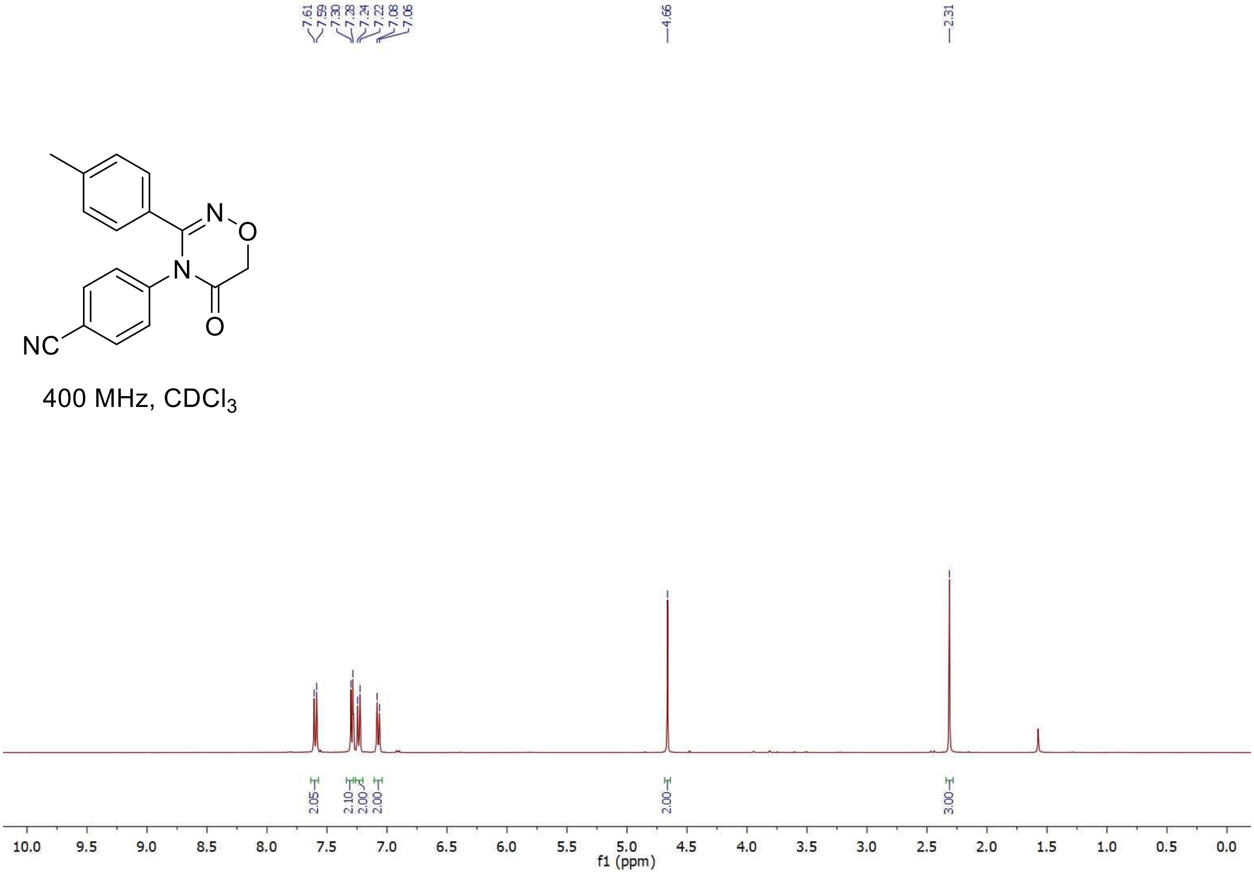
f1 (ppm)

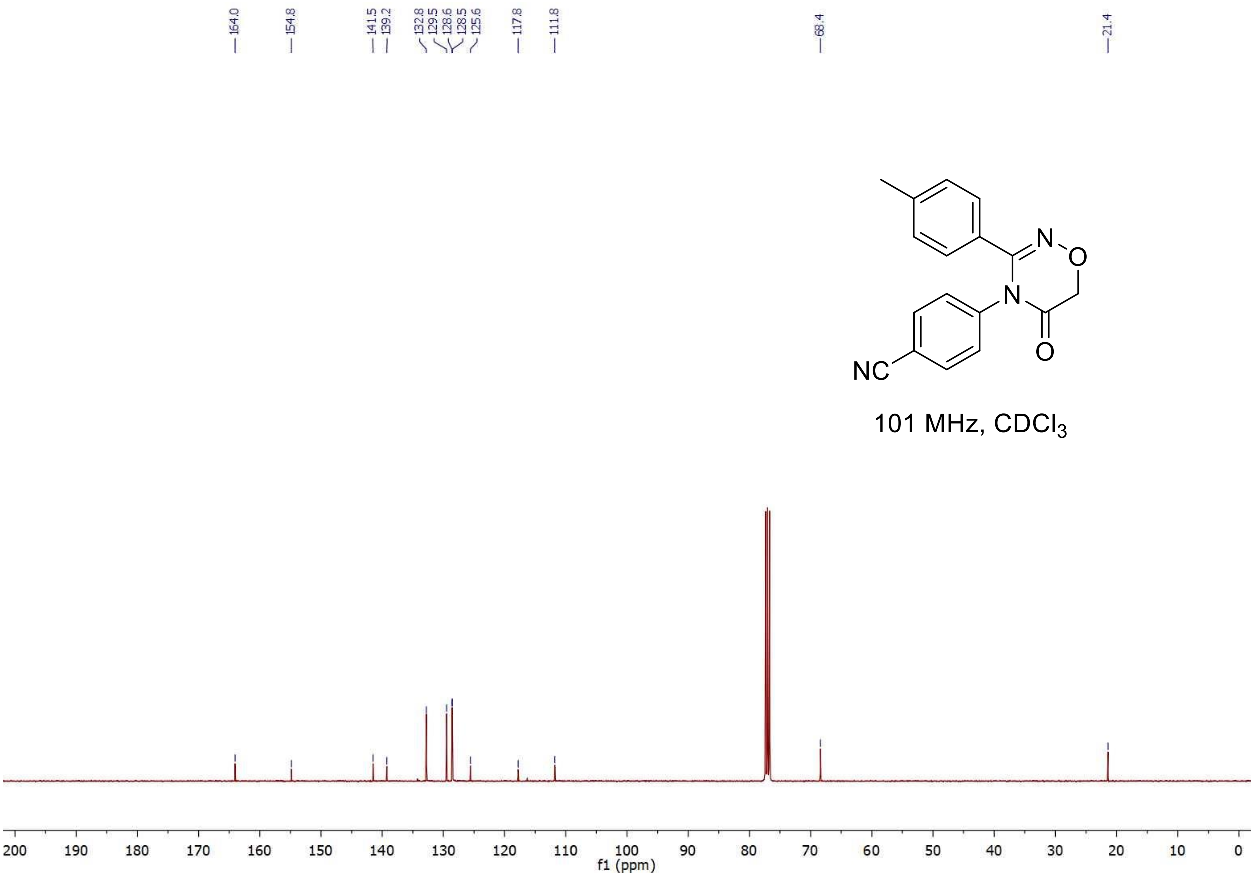
[1]H and [13]C spectra of 3,4-di-4-methylphenyl -4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4d).**



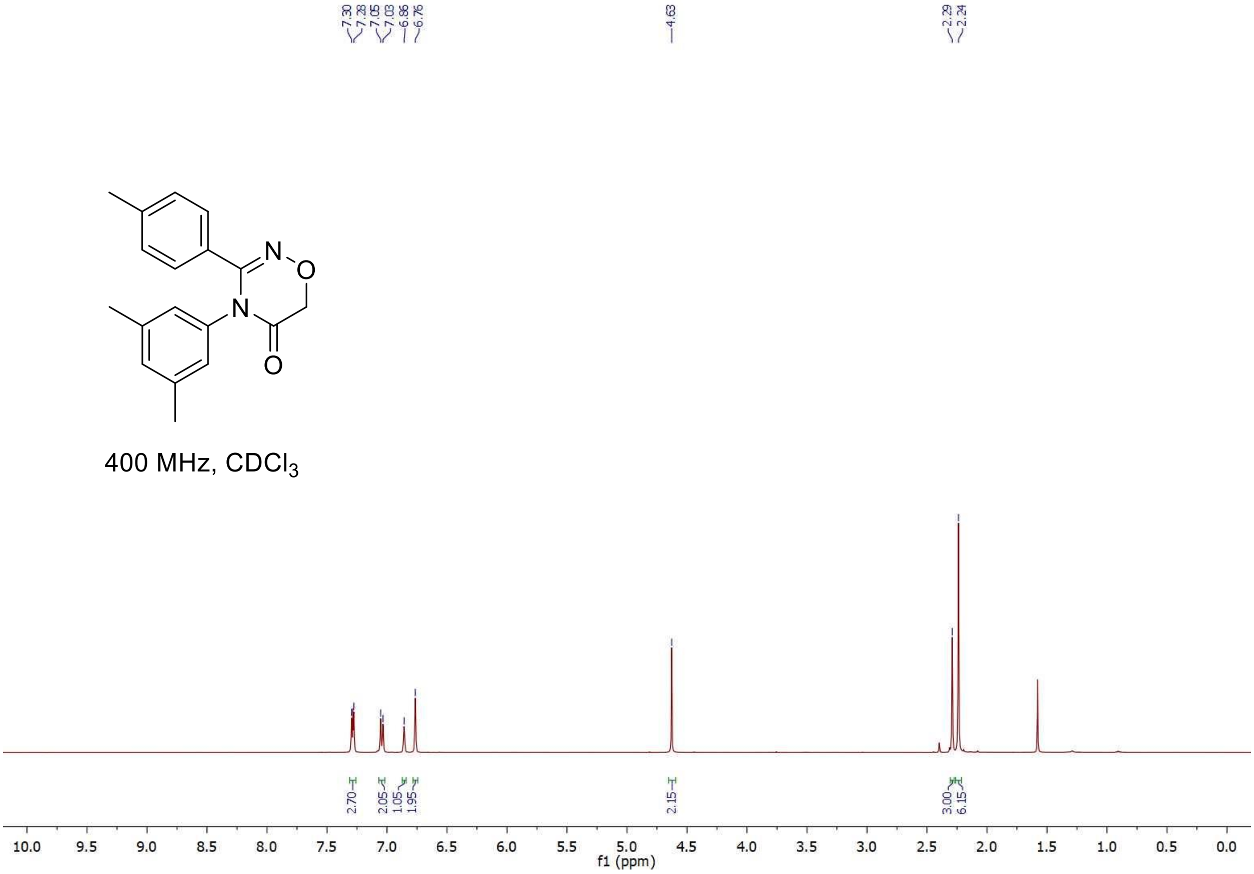


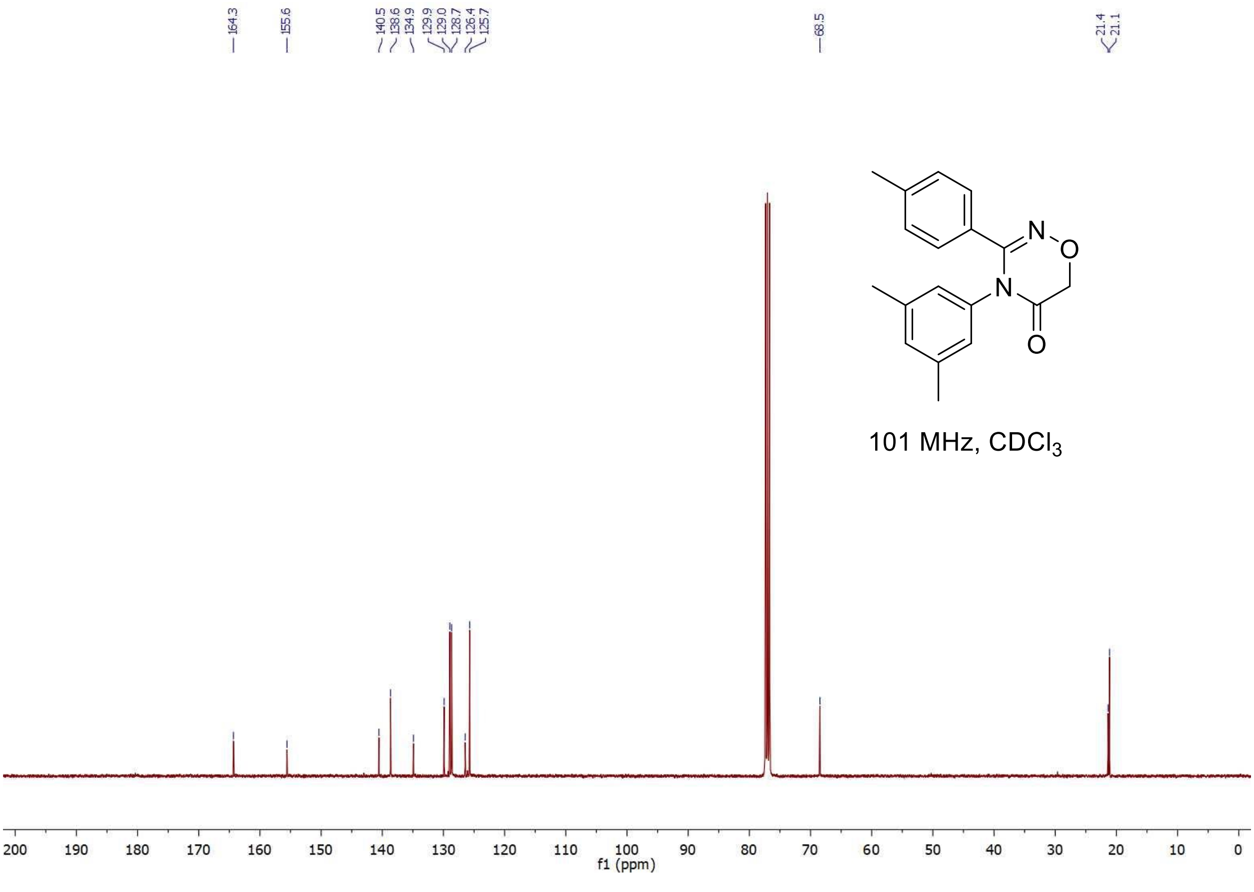
[1]H and [13]C spectra of 4-(5-oxo-3-(4-methylphenyl)-5,6-dihydro-4*H*-1,2,4-oxadiazin-4-yl)benzonitrile **(4e).**



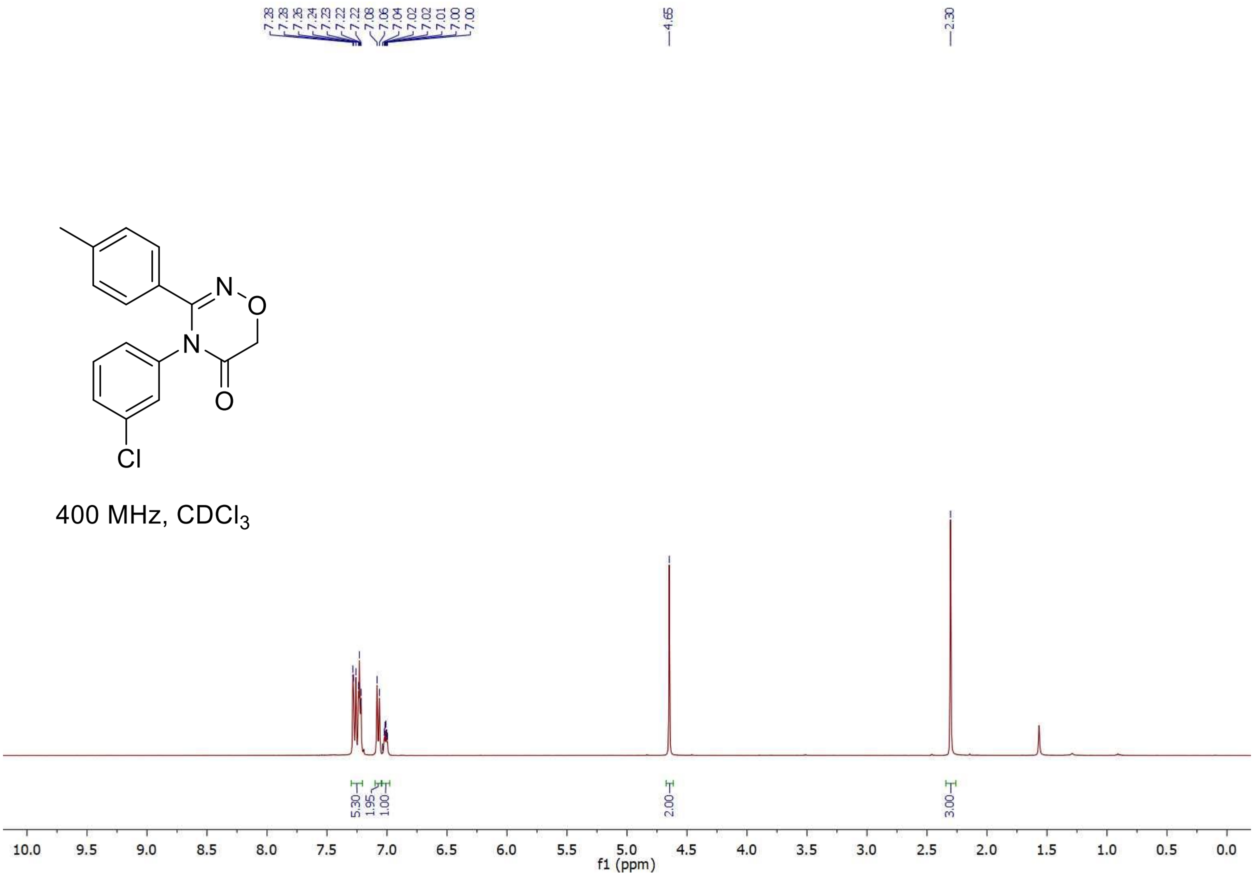


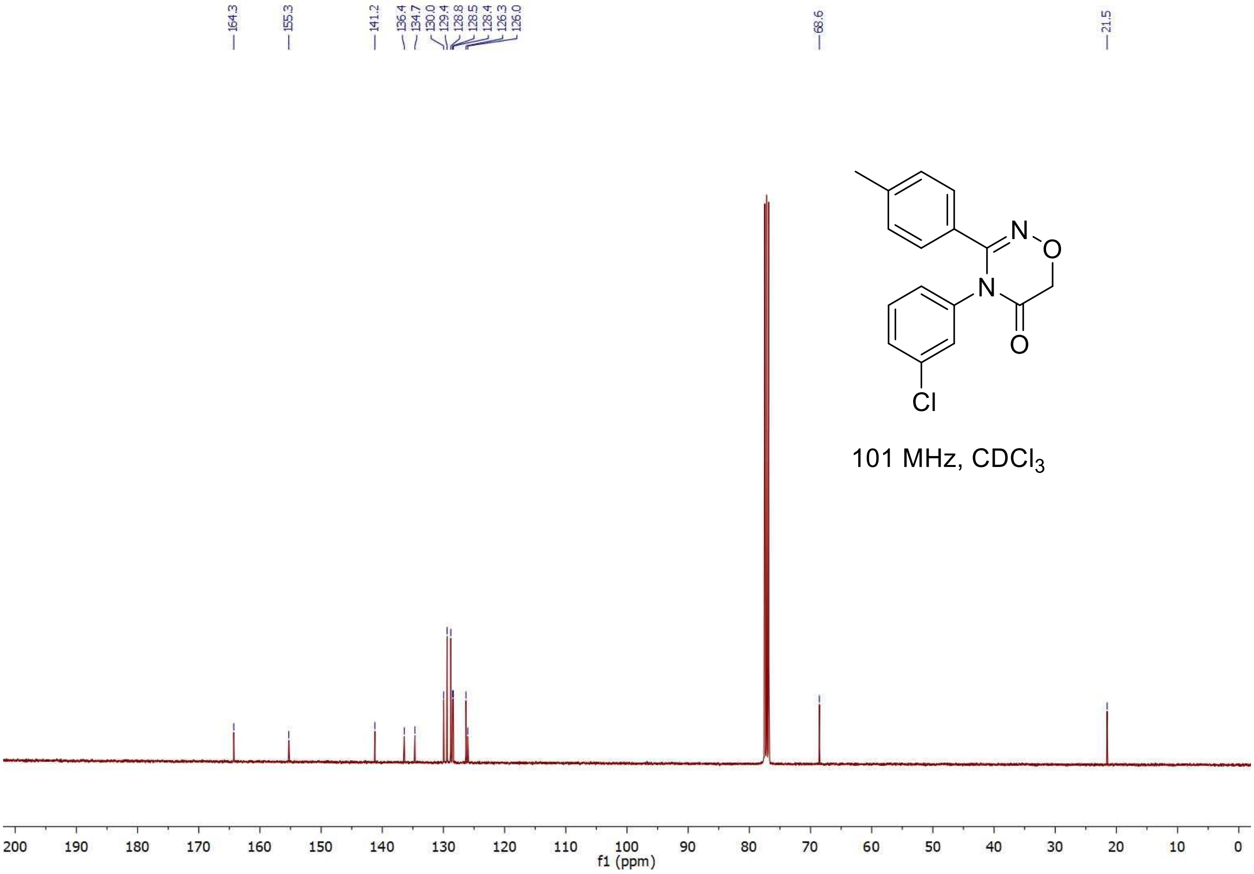
^{1}H and ^{13}C spectra of 4-(3,5-dimethylphenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4f).**



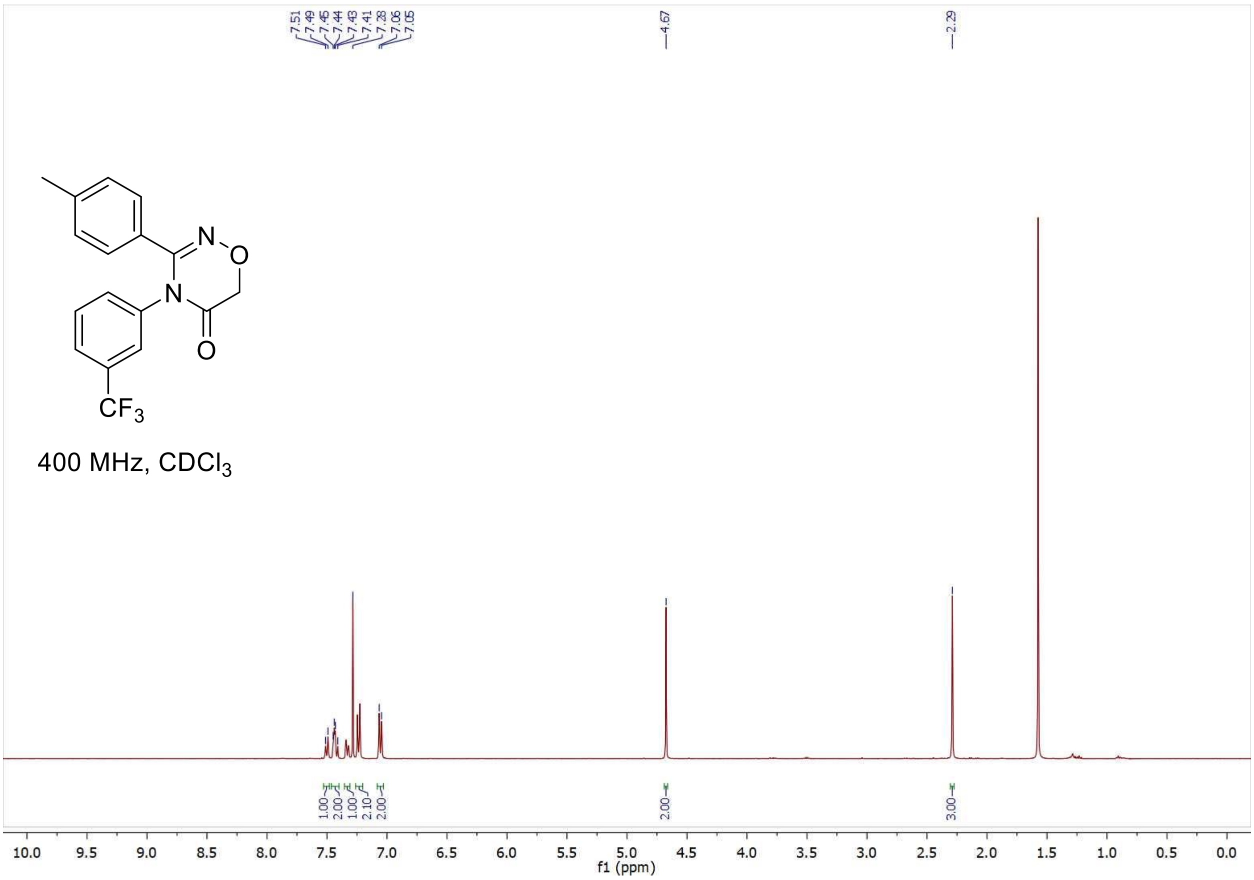


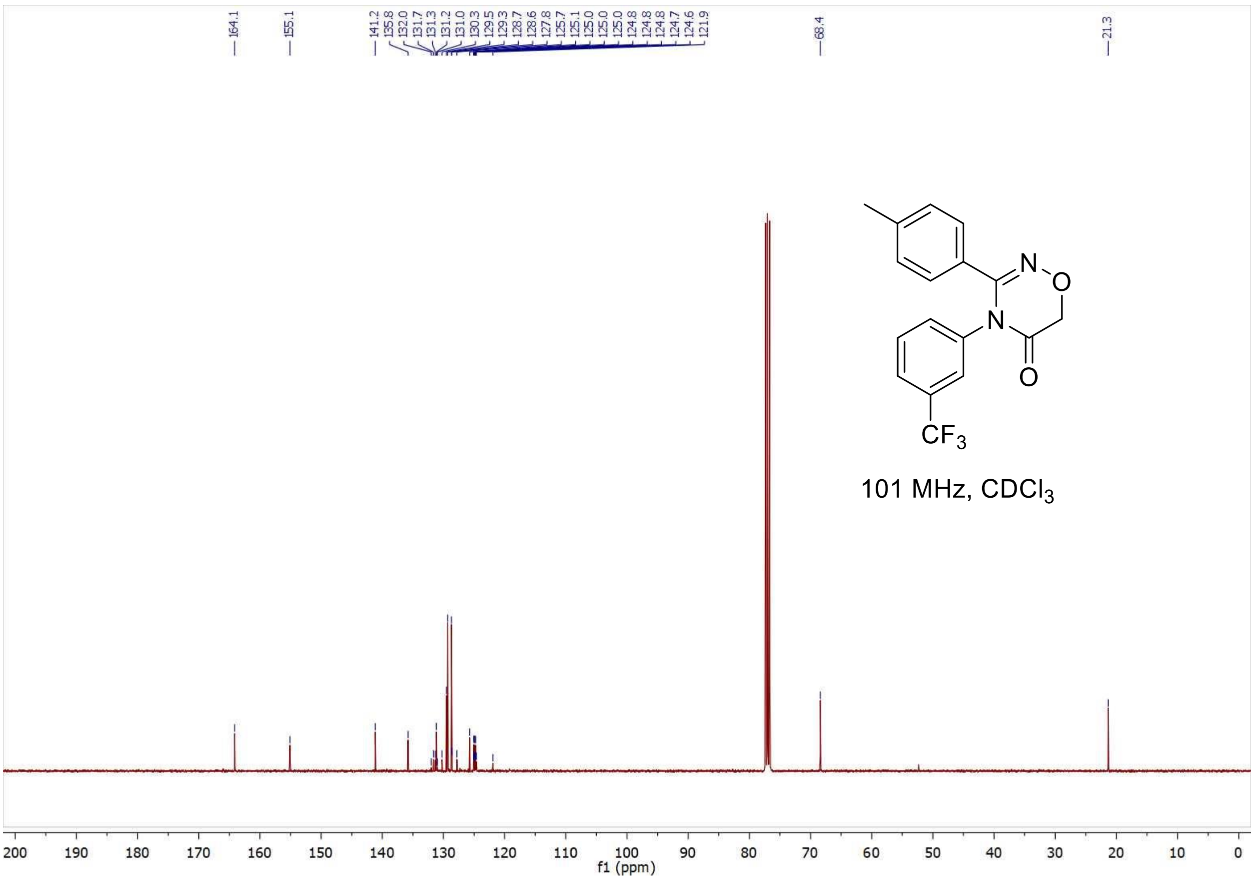
^{1}H and ^{13}C spectra of 4-(3-chlorophenyl)-3-(4-methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4g).**

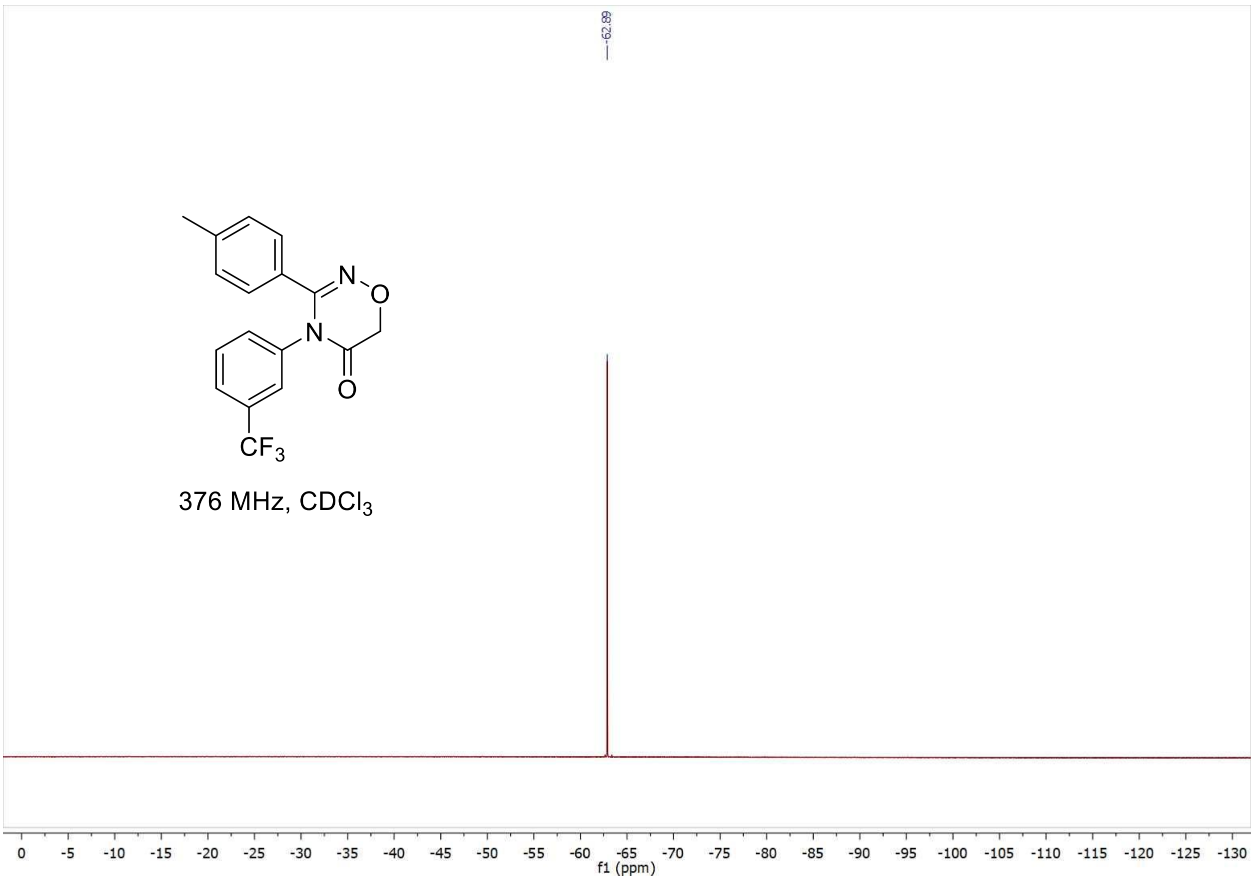




^{1}H, ^{13}C and ^{19}F spectra of 3-(4-methylphenyl)-4-(3-(trifluoromethyl)phenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4h).**







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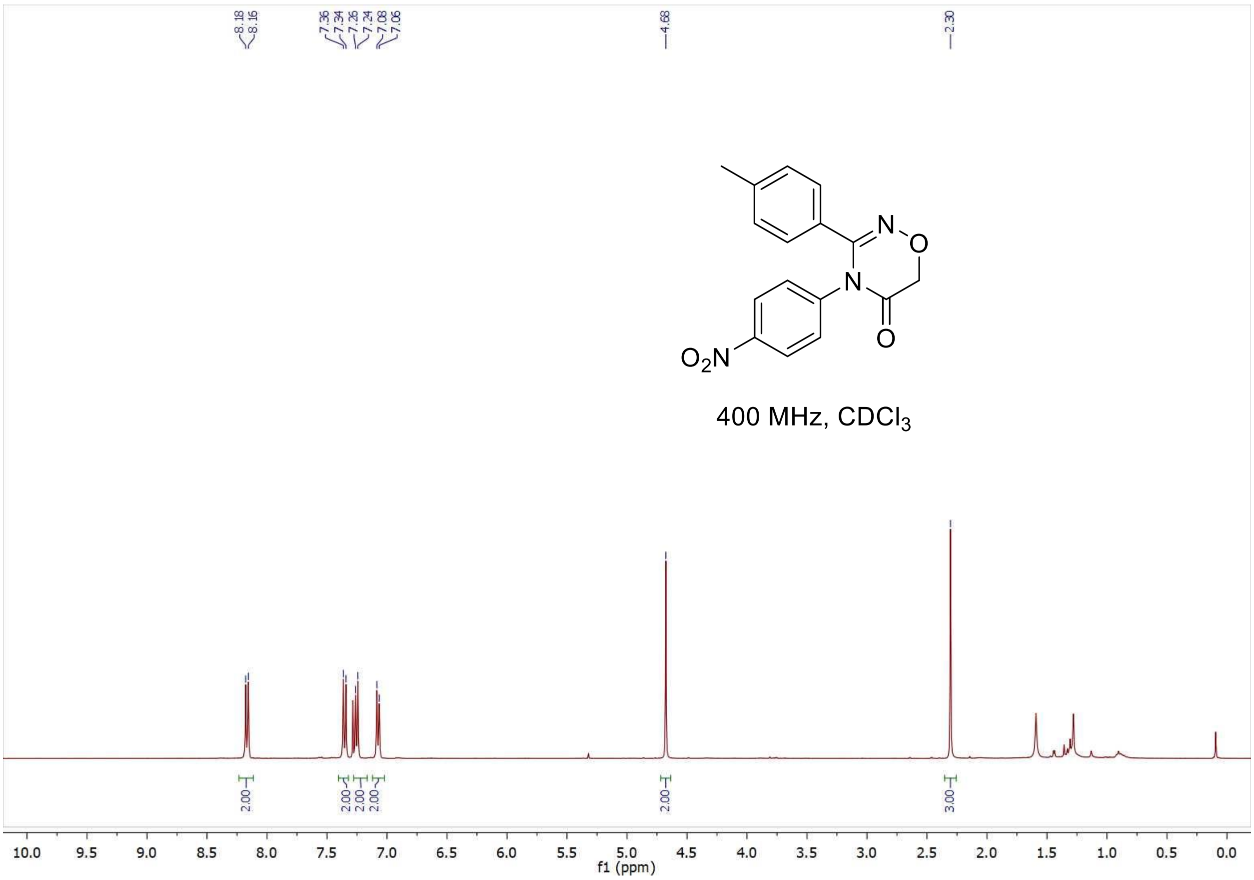
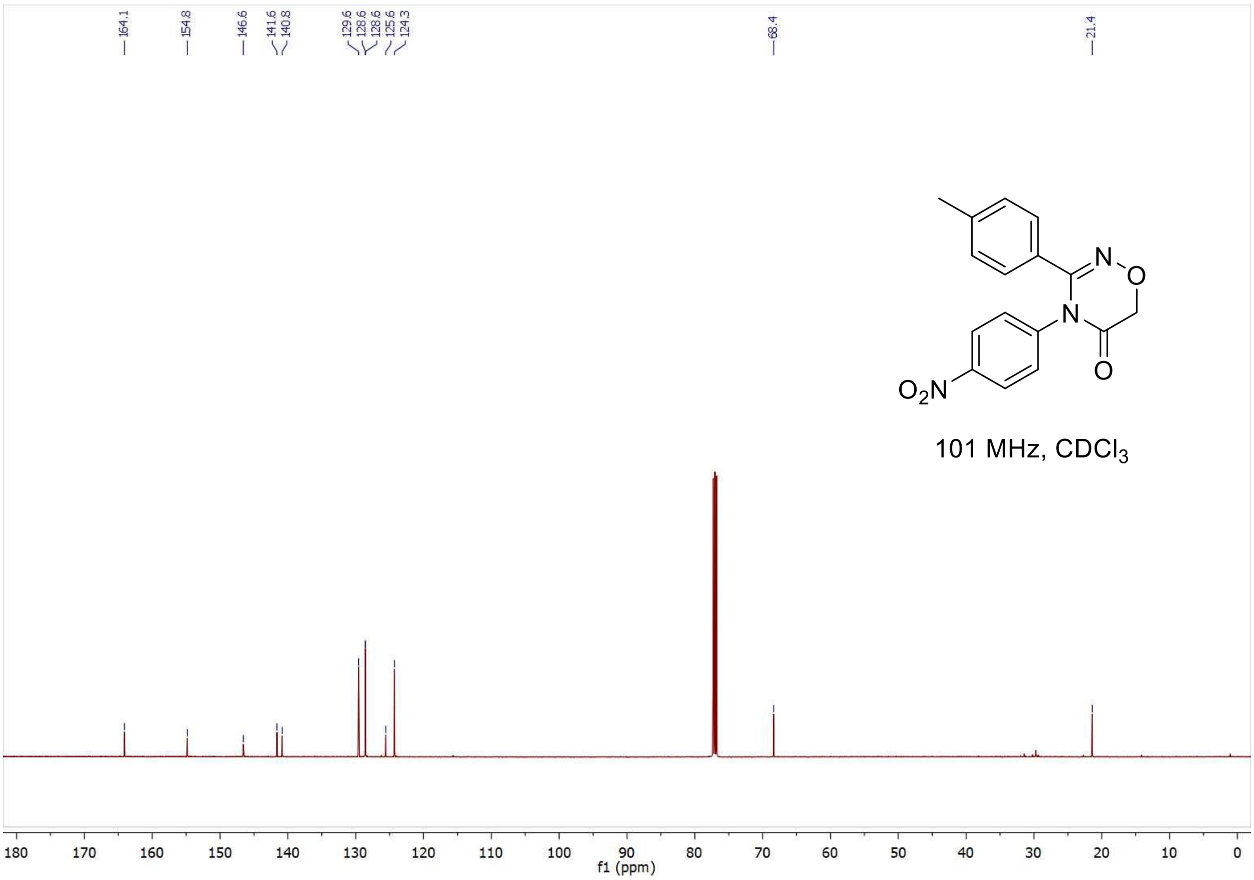
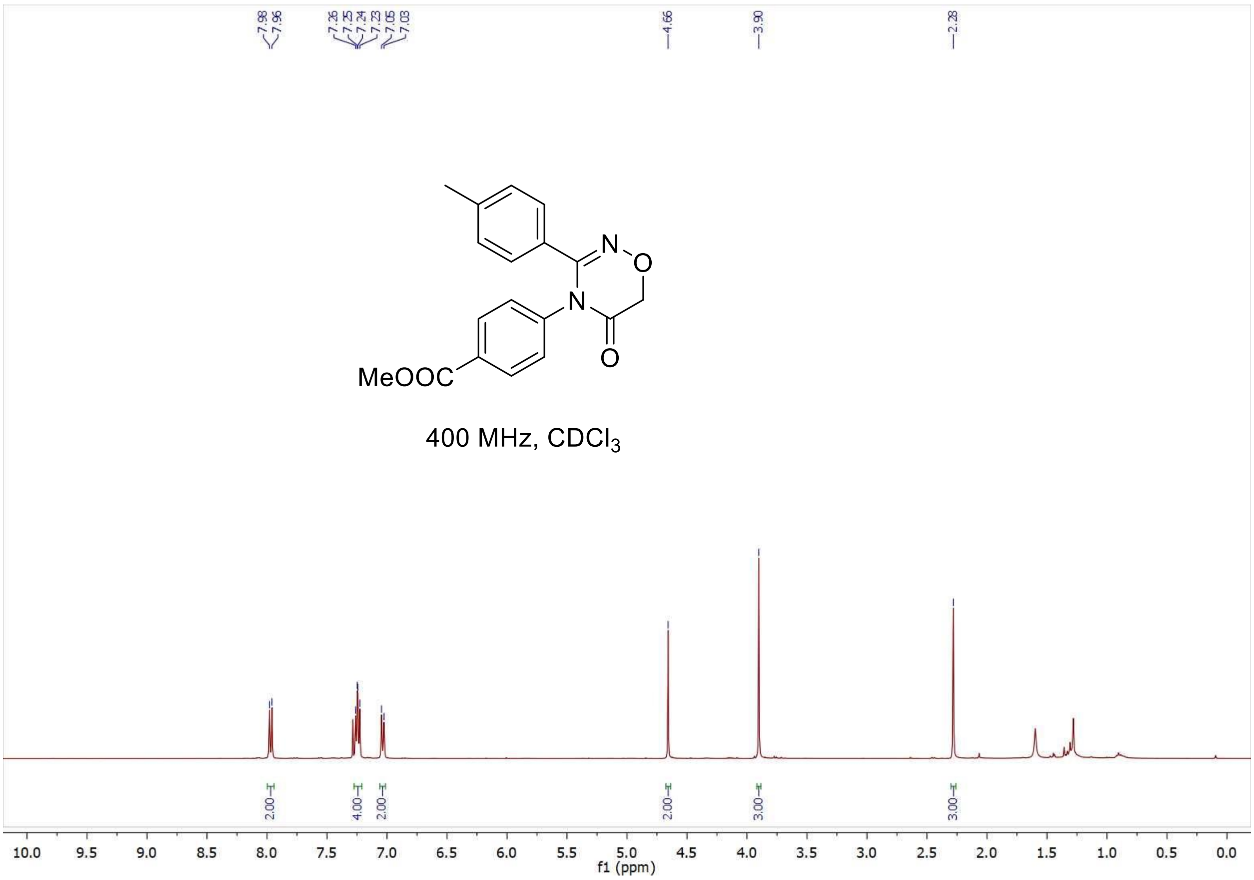
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130
f1 (ppm)

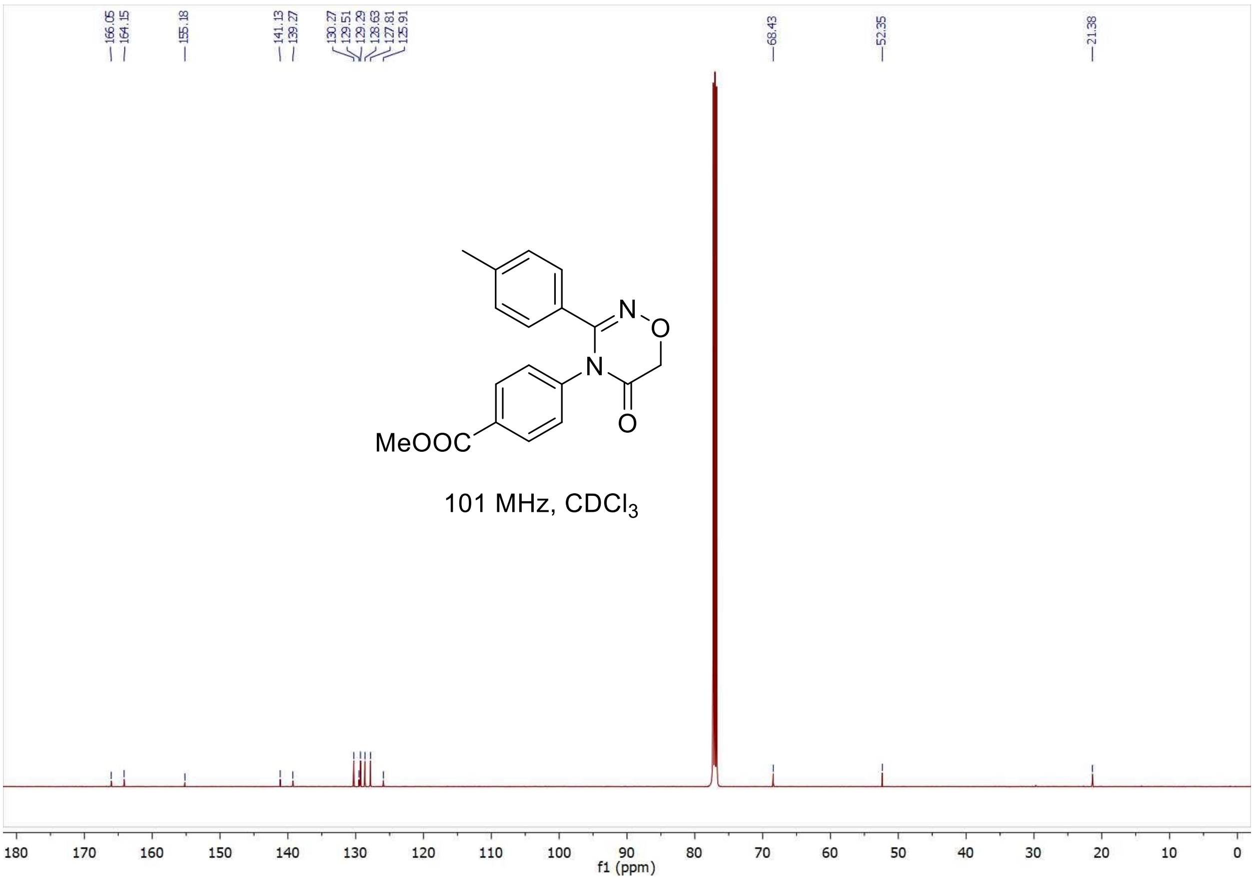
^1H and ^{13}C spectra of 4-(4-nitrophenyl)-3-(4methylphenyl)-4*H*-1,2,4-oxadiazin-5(6*H*)-one **(4i).**





^{1}H and ^{13}C spectra of methyl 4-(5-oxo-3-(4-methylphenyl)-5,6-dihydro-4*H*-1,2,4-oxadiazin-4-yl)benzoate **(4j).**





S5. X–ray diffraction data

Singe crystals for X-ray studying were obtained by slow evaporation of solutions of 1,2,4-oxadiazin-5(6*H*)-ones and *N*-aryl-1,2,4-oxadiazin-5(6*H*)-ones in $CDCl_3$ at RT in air. The crystals **1h, 1i, 3fa** and **3ha** were performed at 100(2) K on a Rigaku XtaLAB Synergy-S diffractometer (HyPix-6000HE type detector) using Cu Kα (λ = 1.54184 Å) radiation. The structures were solved with the ShelXT [7] structure solution program using Intrinsic Phasing and refined with the ShelXL [8] refinement package incorporated in the OLEX2 program package [9] using Least Squares minimization. Empirical absorption correction was applied in CrysAlisPro [10] program complex using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. The hydrogen atom positions were fixed geometrically at calculated distances and allowed to ride on the parent atoms. Supplementary crystallographic data for this paper have been deposited at Cambridge Crystallographic Data Centre and can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif. CCDC numbers 2404214 **(1e),** 2404213 **(1g),** 2401094 **(3e),** 2401093 **(3f).**

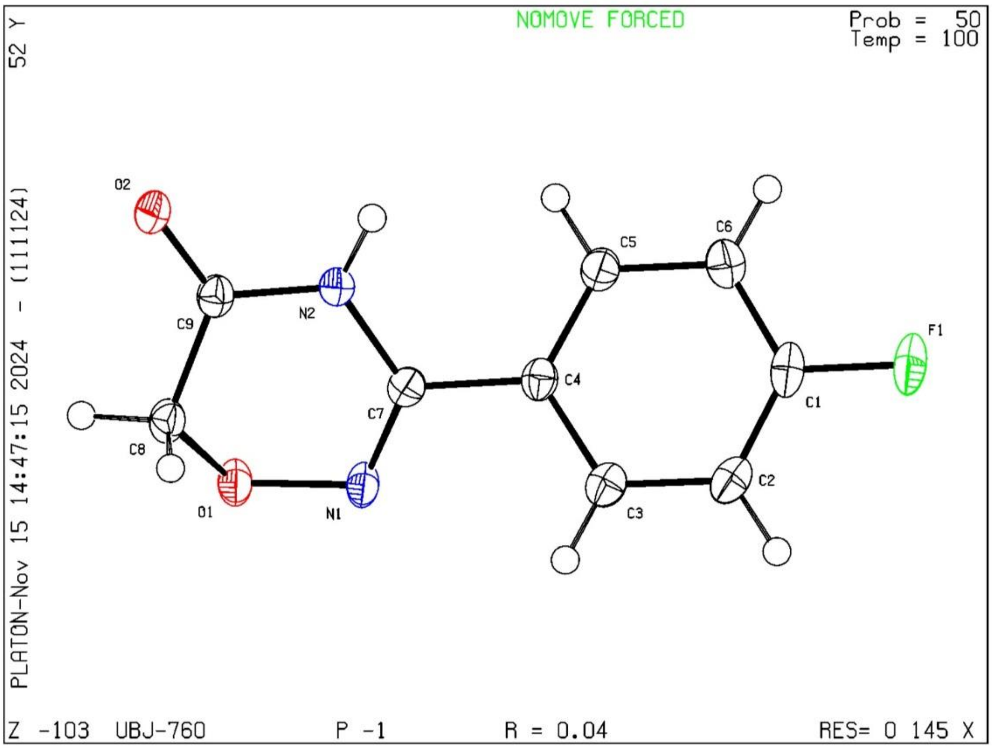


Figure S1. Views of the molecular structure of **1e**. Thermal ellipsoids are drawn at the 50% probability level.

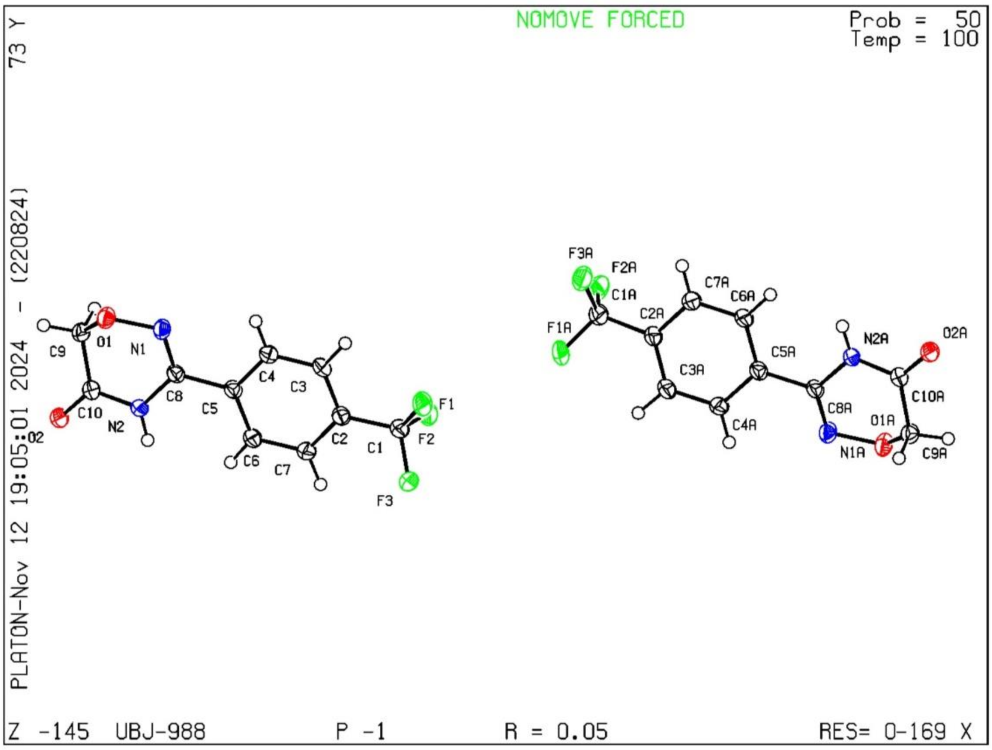


Figure S2. Views of the molecular structure of **1g**. Thermal ellipsoids are drawn at the 50% probability level.

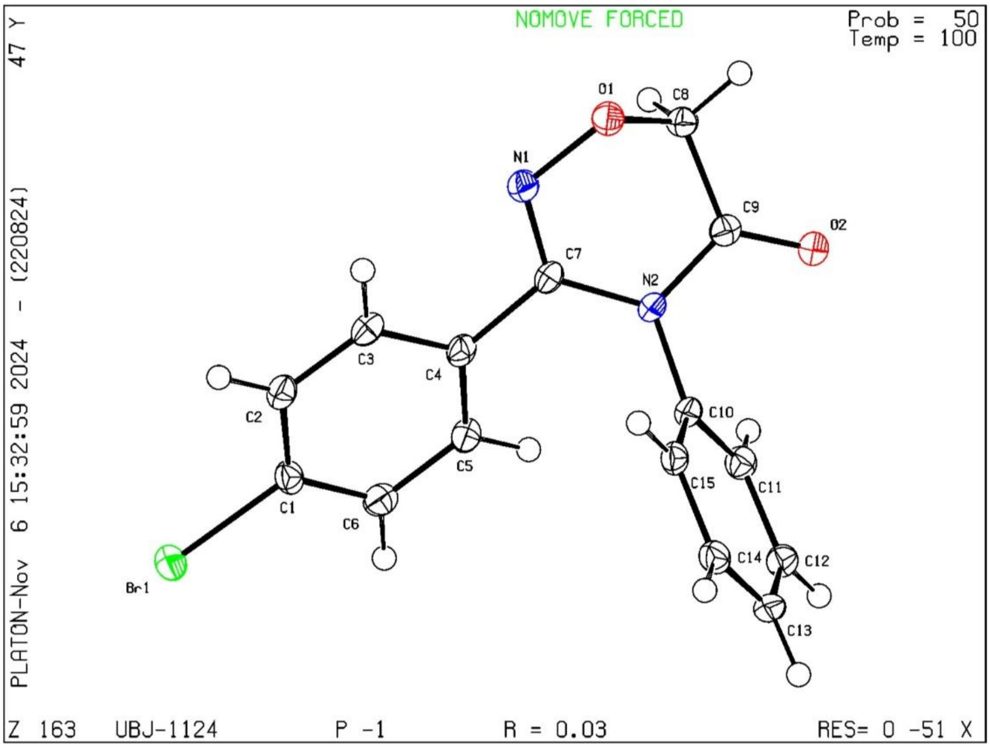


Figure S3. Views of the molecular structure of **3e**. Thermal ellipsoids are drawn at the 50% probability level.

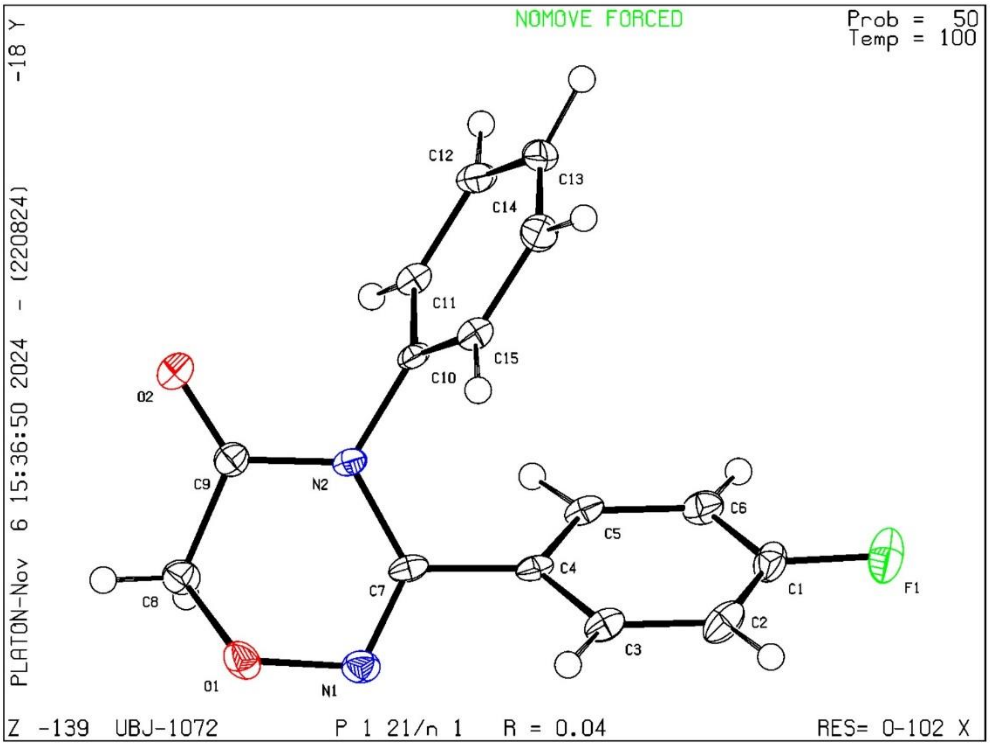


Figure S4. Views of the molecular structure of **3f**. Thermal ellipsoids are drawn at the 50% probability level.

Table S1. Crystal data and structure refinement parameters for **1h, 1i, 3e** and **3f**.

Compound	**1e**	**1g**	**3e**	**3f**
Identification code	UBJ-760	UBJ-988	UBJ-1124	UBJ-1072
CCDC number	2404214	2404213	2401094	2401093
Empirical formula	C9H7FN2O2	$C_{10}H_7F_3N_2O_2$	$C_{15}H_{11}N_2O_2Br$	$C_{15}H_{11}N_2O_2F$
Formula weight	194.167	244.174	331.170	270.265
Temperature, K	99.98(19)	99.99(10)	100.00(10)	99.99(10)
Crystal system	Triclinic	triclinic	triclinic	monoclinic
Space group	P-1	P-1	P-1	$P2_1/n$
a, Å	6.9399(3)	8.2385(3)	5.7761(3)	9.1769(3)
b, Å	7.2091(4)	11.3733(3)	9.1850(4)	7.4272(2)
c, Å	9.0838(4)	12.0528(4)	12.8929(4)	18.6039(6)
α, °	88.303(4)	116.505(3)	105.632(3)	90
β, °	68.988(4)	94.639(3)	92.203(3)	101.568(3)
γ, °	76.975(4)	100.632(3)	100.013(4)	90
Volume, Å^3	412.67(4)	976.03(6)	646.05(5)	1242.25(7)
Z	2	4	2	4
ρ_{calc}g, cm^3	1.563	1.662	1.702	1.445
μ, mm^{-1}	1.098	1.373	4.368	0.904
F(000)	200.8	498.4	331.6	562.1
Crystal size, mm^3	0.10 × 0.07 × 0.05	0.28 × 0.08 × 0.03	0.16 × 0.1 × 0.08	0.22 × 0.19 × 0.16
Radiation	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	CuKα (λ = 1.54184)	CuKα (λ = 1.54184)
2Θ range for data collection, °	10.44 to 138.24	8.34 to 160.02	7.14 to 139.98	9.7 to 152.76
Index ranges	$-8 \le h \le 8$, $-8 \le k \le 8$, $-11 \le l \le 10$	$-10 \le h \le 7$ $-14 \le k \le 14$ $-15 \le l \le 15$	$-7 \le h \le 7$ $-10 \le k \le 11$ $-16 \le l \le 14$	$-11 \le h \le 6$ $-9 \le k \le 8$ $-23 \le l \le 22$
Reflections collected	2997	12441	6851	5035
Independent reflections	1499 [R_{int} = 0.0224, R_{sigma} = 0.0349]	4010 [R_{int} = 0.0486, R_{sigma} = 0.0474]	2382 [R_{int} = 0.0427 R_{sigma} = 0.0367]	2514 [R_{int} = 0.0338 R_{sigma} = 0.0377]
Data/restraints/parameters	1499/0/131	4010/0/363	2382/0/181	2514/0/225
Goodness–of–fit on F^2	1.0873	1.047	1.042	1.055
Final R indexes [I>=2σ (I)]	R1 = 0.0381 wR2 = 0.1037	R_1 = 0.0505 wR_2 = 0.1420	R_1 = 0.0319 wR_2 = 0.0823	R_1 = 0.0425 wR_2 = 0.1137
Final R indexes [all data]	R1 = 0.0419 wR2 = 0.1079	R_1 = 0.0594 wR_2 = 0.1480	R_1 = 0.0325 wR_2 = 0.0830	R_1 = 0.0486 wR_2 = 0.1210
Largest diff. peak/hole/ eÅ^{-3}	0.2165	0.50/-0.54	1.00/-0.42	0.29/-0.31

S6. Referenses

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