

Supplementary Information

KO^tBu-promoted C3-homocoupling of quinoxalinones through single electron transfer from an sp² carbanion intermediate

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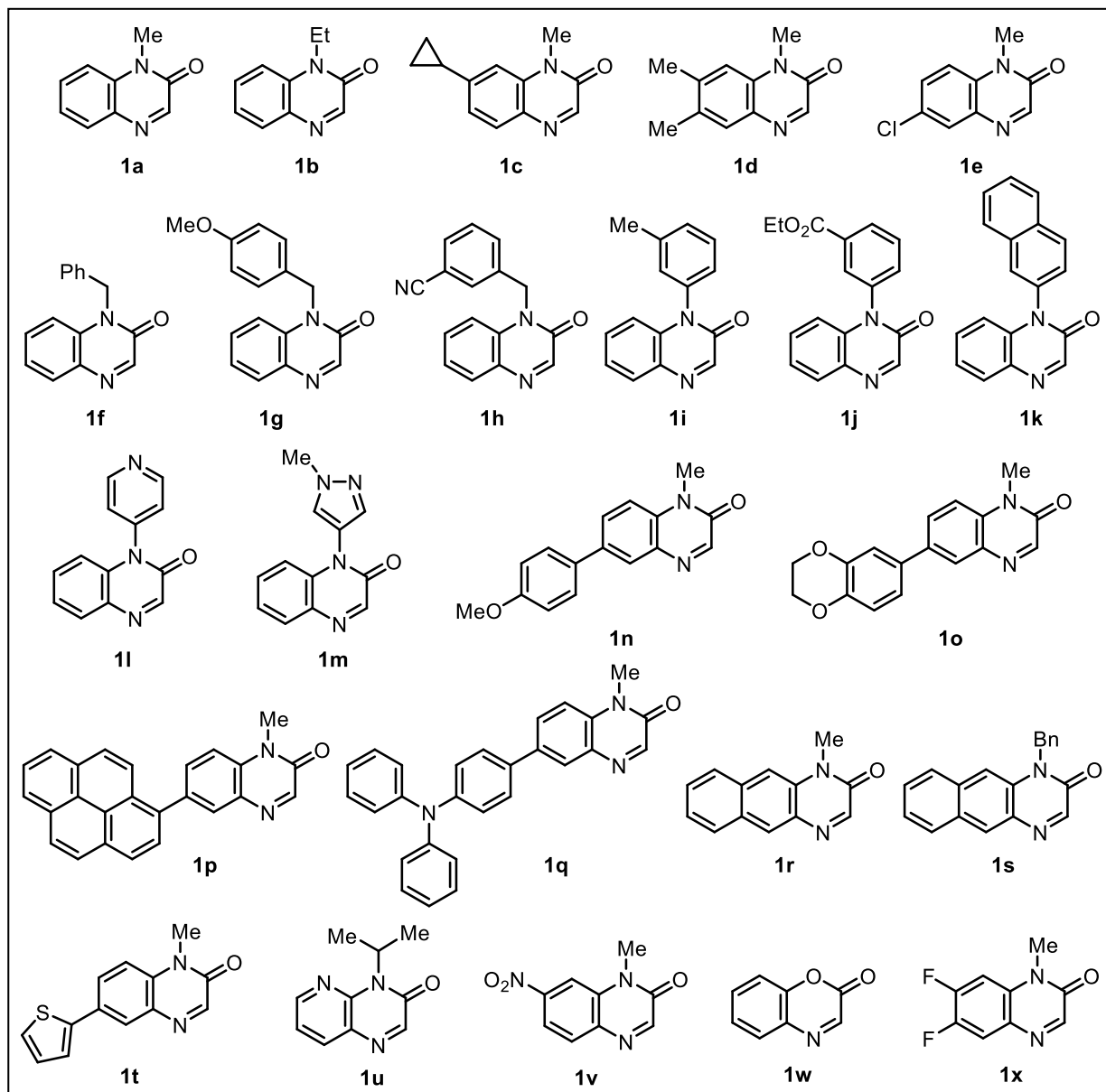
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General methods

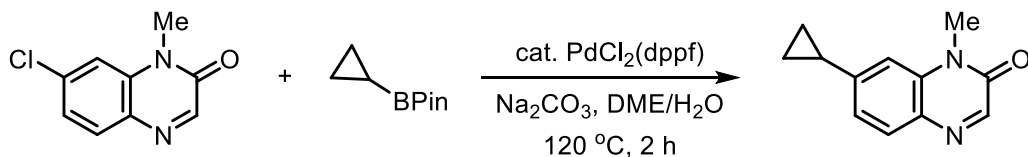
Commercially available reagents and solvents (purchased from Sigma-Aldrich, TCI, Alfa-Aesar, Acros, and Combi-block) were used without additional purification, unless otherwise stated. Sealed tubes ($13 \times 100 \text{ mm}^2$) were purchased from Fischer Scientific and dried in oven for overnight and cooled at room temperature prior to use. Thin layer chromatography was carried out using plates coated with Kieselgel 60 F₂₅₄ (Merck). For flash column chromatography, E. Merck Kieselgel 60 (230–400 mesh) was used. The physical texture of the reported compounds is recorded at room temperature. Melting points were obtained on a melting point apparatus and the data are uncorrected. Nuclear magnetic resonance spectra (¹H and ¹³C NMR) were recorded on a Bruker Unity 700, 500 and 400 spectrometers in CDCl₃ and DMSO-d₆ solution and chemical shifts are reported as parts per million (ppm). Resonance patterns are reported with the notations s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). Coupling constants (*J*) are reported in hertz (Hz). IR spectra were recorded on a JASCO FT/IR-4200 Infrared spectrophotometer and are reported as cm⁻¹. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-700 spectrometer. Electron paramagnetic resonance (EPR) studies were performed using a Bruker EMX/Plus spectrometer equipped with a dual mode cavity (ER 4116DM).

List of quinoxalinone substrates (1a–1x)



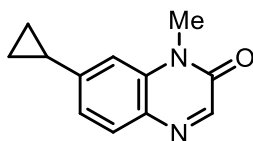
- *N*-Alkyl quinoxalinone **1a**, *N*-benzyl quinoxalinones (**1f** and **1h**), *N*-(hetero)aryl quinoxalinones **1i–1m** and pyrido-pyrazinone **1u** were synthesized according to a literature procedure.¹
- *N*-Alkyl quinoxalinones (**1b**, **1e**, **1v**, and **1x**) and *N*-benzyl quinoxalinone **1g** were synthesized according to a literature procedure.²
- *N*-Alkyl quinoxalinone **1d** and *N*-methyl benzo[*g*]quinoxalinone **1r** were synthesized according to a literature procedure.³
- *N*-Benzyl benzo[*g*]quinoxalinone **1s** was synthesized according to a literature procedure.⁴
- Benzoxazinone **1w** was synthesized according to a literature procedure.⁵

General procedure and characterization data for the synthesis of *N*-methyl-7-cyclopropyl quinoxalinone (**1c**)



To an oven-dried sealed tube charged with 7-chloro-1-methylquinoxalinone (1.0 mmol, 1.0 equiv.), cyclopropylboronic acid pinacol ester (1.5 mmol, 1.5 equiv.), and PdCl₂(dppf)·CH₂Cl₂ (0.2 mmol, 0.2 equiv.) was added DME (6 mL) and aqueous solution of 2 M Na₂CO₃ (2 mL) under argon atmosphere. The reaction mixture was allowed to stir at 120 °C for 2 h. The reaction mixture was cooled to room temperature and concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*-hexanes/acetone = 9:1) to afford the corresponding *N*-methyl-7-cyclopropyl quinoxalinone (**1c**).

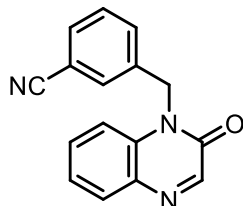
7-Cyclopropyl-1-methylquinoxalin-2(1*H*)-one (**1c**)



78.1 mg (39%); pale red solid; mp = 117.8–119.2 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.22 (s, 1H), 7.74 (d, *J* = 8.4 Hz, 1H), 7.04 (d, *J* = 2.0 Hz, 1H), 6.99 (dd, *J* = 8.4, 2.0 Hz, 1H), 3.69 (s, 3H), 2.08–2.02 (m, 1H), 1.16–1.07 (m, 2H), 0.85–0.81 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 148.8, 148.6, 132.9, 131.8, 130.4 (two carbons overlap), 121.0, 111.0, 28.8, 16.4, 10.6; IR (KBr) ν 3292, 3088, 3013, 2945, 1642, 1605, 1543, 1435, 1362, 1283, 1217, 1159, 1063, 1018, 965 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₂H₁₂N₂O [M]⁺ 200.0950, found 200.0950.

Characterization data for *N*-benzyl quinoxalinone 1h

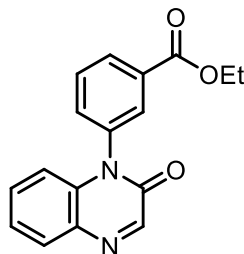
3-((2-Oxoquinoxalin-1(2*H*)-yl)methyl)benzonitrile (1h)



752.0 mg (42%); white solid; mp = 230.1–233.4 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.42 (s, 1H), 7.94 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.59 (dt, *J* = 7.2, 1.6 Hz, 1H), 7.55–7.49 (m, 3H), 7.46 (t, *J* = 8.0 Hz, 1H), 7.40–7.34 (m, 1H), 7.17 (dd, *J* = 8.0, 1.6 Hz, 1H), 5.50 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 155.1, 150.3, 136.8, 133.8, 132.3, 131.8, 131.5, 131.4, 131.2, 130.6, 130.1, 124.4, 118.4, 114.1, 113.4, 45.0; IR (KBr) ν 3048, 2958, 2926, 2229, 1654, 1603, 1589, 1555, 1464, 1371, 1314, 1273, 1139, 1062 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₆H₁₁N₃O [M]⁺ 261.0902, found 261.0902.

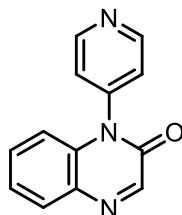
Characterization data for *N*-(hetero)aryl quinoxalinones (**1j**, **1l** and **1m**)

Ethyl 3-(2-oxoquinoxalin-1(2*H*)-yl)benzoate (**1j**)



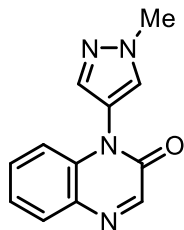
235.5 mg (80%); white solid; mp = 108.3–111.4 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.41 (s, 1H), 8.26 (dt, *J* = 8.0, 1.6 Hz, 1H), 8.00 (t, *J* = 2.0 Hz, 1H), 7.94 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.72 (t, *J* = 8.0 Hz, 1H), 7.51 (ddd, *J* = 7.6, 2.0, 1.2 Hz, 1H), 7.39 (td, *J* = 7.2, 1.6 Hz, 1H), 7.35 (td, *J* = 7.6, 1.6 Hz, 1H), 6.68 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.45–4.33 (m, 2H), 1.38 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.4, 154.7, 150.9, 135.5, 133.8, 133.3, 133.2, 132.8, 131.0, 130.9, 130.7, 130.4, 129.7, 124.3, 115.5, 61.6, 14.4; IR (KBr) ν 3053, 2982, 1719, 1672, 1603, 1555, 1484, 1461, 1442, 1270, 1214 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₇H₁₄N₂O₃ [M]⁺ 294.1004, found 294.1000.

1-(Pyridin-4-yl)quinoxalin-2(1*H*)-one (**1l**)



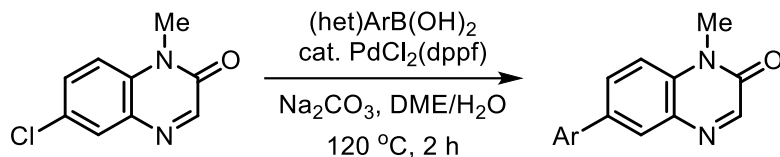
29.0 mg (14%); white solid; mp = 185.6–188.0 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.93 (d, *J* = 4.8 Hz, 2H), 8.39 (s, 1H), 7.95 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.43 (td, *J* = 7.6, 1.6 Hz, 1H), 7.38 (td, *J* = 7.6, 1.6 Hz, 1H), 7.33 (d, *J* = 1.6 Hz, 1H), 7.32 (d, *J* = 1.6 Hz, 1H), 6.70 (dd, *J* = 8.4, 1.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 153.9, 152.4, 150.7, 143.2, 133.2, 132.7, 131.2, 130.7, 124.7, 123.6, 115.1; IR (KBr) ν 3056, 3015, 1668, 1601, 1581, 1458, 1408, 1363, 1300, 1268, 1222, 962 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₃H₉N₃O [M]⁺ 223.0746, found 223.0743.

1-(1-Methyl-1*H*-pyrazol-4-yl)quinoxalin-2(1*H*)-one (1m)



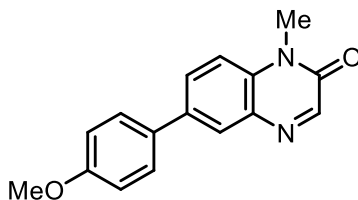
88.2 mg (39%); white solid; mp = 205.3–207.3 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.37 (s, 1H), 7.90 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.62 (s, 1H), 7.58 (s, 1H), 7.45 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.35 (ddd, *J* = 8.8, 8.0, 1.6 Hz, 1H), 7.12 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.03 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 155.0, 150.6, 137.2, 134.3, 133.4, 131.0, 130.3, 128.7, 124.3, 116.3, 115.7, 40.0; IR (KBr) ν 3119, 3055, 1656, 1602, 1587, 1550, 1464, 1365, 1291, 1268, 1223, 985 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₂H₁₀N₄O [M]⁺ 226.0855, found 226.0850.

General procedure and characterization data for the synthesis of *N*-methyl-6-(hetero)aryl quinoxalinones (**1n–1q** and **1t**)



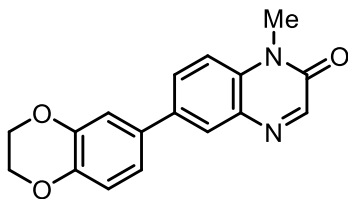
To an oven-dried sealed tube charged with 6-chloro-1-methylquinoxalinone (**1e**) (1.0 mmol, 1.0 equiv.), (hetero)arylboronic acid (1.5 mmol, 1.5 equiv.), and PdCl₂(dppf)·CH₂Cl₂ (0.2 mmol, 0.2 equiv.) was added DME (6 mL) and aqueous solution of 2 M Na₂CO₃ (2 mL) under argon atmosphere. The reaction mixture was allowed to stir at 120 °C for 2 h. The reaction mixture was cooled to room temperature, filtered through celite pad and the filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*-hexane/acetone) to afford the corresponding *N*-methyl-6-(hetero)aryl quinoxalinones (**1n–1q** and **1t**).

6-(4-Methoxyphenyl)-1-methylquinoxalin-2(1*H*)-one (**1n**)



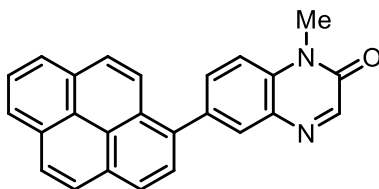
229.0 mg (86%); pale red solid; mp = 133.2–136.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.34 (s, 1H), 8.06 (d, *J* = 2.0 Hz, 1H), 7.80 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.62–7.55 (m, 2H), 7.39 (d, *J* = 8.5 Hz, 1H), 7.06–6.97 (m, 2H), 3.87 (s, 3H), 3.72 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 159.7, 155.1, 150.7, 136.8, 133.9, 132.1, 131.8, 129.7, 128.2, 128.0, 114.7, 114.3, 55.6, 29.0; IR (KBr) ν 3053, 2957, 2933, 2836, 1652, 1606, 1490, 1458, 1363, 1276, 1246, 1176, 1114, 1074, 1026 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₆H₁₄N₂O₂ [M]⁺ 266.1055, found 266.1050.

6-(2,3-Dihydrobenzo[*b*][1,4]dioxin-6-yl)-1-methylquinoxalin-2(1*H*)-one (**1o**)



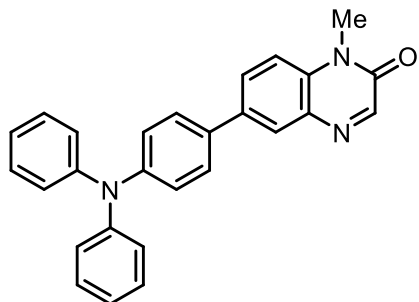
162.0 mg (55%); pale red solid; mp = 220.2–222.6 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.34 (s, 1H), 8.04 (d, J = 2.0 Hz, 1H), 7.78 (dd, J = 8.8, 2.4 Hz, 1H), 7.38 (d, J = 8.8 Hz, 1H), 7.17 (d, J = 2.4 Hz, 1H), 7.14 (dd, J = 8.4, 2.0 Hz, 1H), 6.97 (d, J = 8.4 Hz, 1H), 4.32 (s, 4H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.1, 150.7, 144.1, 143.7, 136.6, 133.8, 132.8, 132.2, 129.7, 128.1, 120.1, 118.0, 115.8, 114.3, 64.6, 64.5, 29.0; IR (KBr) ν 3054, 2987, 2931, 1655, 1587, 1490, 1362, 1309, 1284, 1267, 1249, 1223, 1066 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_3$ $[\text{M}]^+$ 294.1004, found 294.1003.

1-Methyl-6-(pyren-1-yl)quinoxalin-2(1H)-one (1p)



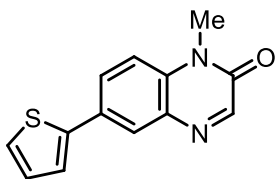
220.3 mg (61%); brown solid; mp = 248.2–250.5 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.41 (s, 1H), 8.27–8.13 (m, 7H), 8.07–8.00 (m, 3H), 7.88 (dd, J = 8.4, 2.0 Hz, 1H), 7.51 (d, J = 8.4 Hz, 1H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.2, 150.9, 137.1, 135.5, 133.6, 133.5, 132.6, 132.1, 131.6, 131.1, 131.0, 128.6, 128.1, 127.9, 127.8, 127.5, 126.3, 125.6, 125.2, 125.1, 125.0, 124.9, 124.7, 29.1; IR (KBr) ν 3052, 2925, 2854, 1662, 1578, 1552, 1456, 1416, 1362, 1268, 1223, 1064 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{25}\text{H}_{16}\text{N}_2\text{O}$ $[\text{M}]^+$ 360.1263, found 360.1259.

6-(4-(Diphenylamino)phenyl)-1-methylquinoxalin-2(1H)-one (1q)



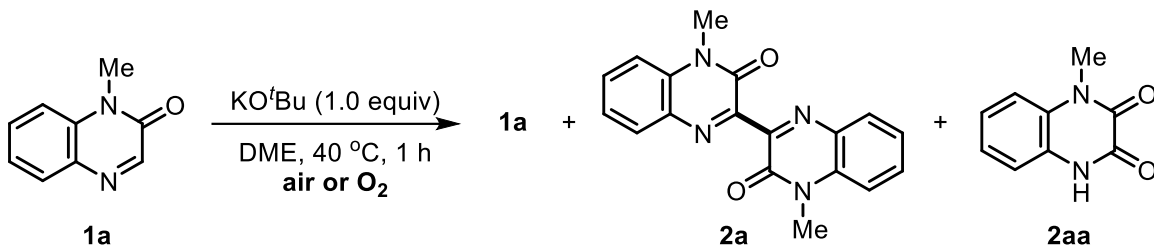
248.6 mg (89%); yellow solid; mp = 184.6–186.2 °C; ^1H NMR (700 MHz, CDCl_3) δ 8.34 (s, 1H), 8.08 (d, J = 2.1 Hz, 1H), 7.83 (dd, J = 9.1, 2.1 Hz, 1H), 7.56–7.51 (m, 2H), 7.39 (d, J = 9.1 Hz, 1H), 7.31–7.27 (m, 4H), 7.18–7.16 (m, 2H), 7.16–7.13 (m, 4H), 7.07–7.05 (m, 2H), 3.73 (s, 3H); ^{13}C NMR (175 MHz, CDCl_3) δ 155.1, 150.7, 147.9, 147.7, 136.7, 133.9, 132.8, 132.2, 129.6, 129.5, 127.9, 127.7, 124.8, 123.8, 123.4, 114.3, 29.0; IR (KBr) ν 3056, 2927, 1657, 1586, 1551, 1518, 1487, 1362, 1271, 1223, 1166, 1073 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{27}\text{H}_{21}\text{N}_3\text{O}$ $[\text{M}]^+$ 403.1685, found 403.1681.

1-Methyl-6-(thiophen-2-yl)quinoxalin-2(1H)-one (1t)



123.6 mg (51%); pale red solid; mp = 168.5–170.2 °C; ^1H NMR (500 MHz, CDCl_3) δ 8.33 (s, 1H), 8.10 (s, 1H), 7.83 (d, J = 11.0 Hz, 1H), 7.38–7.32 (m, 3H), 7.12 (t, J = 5.5 Hz, 1H), 3.70 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 155.0, 151.0, 142.5, 133.7, 132.5, 130.6, 128.9, 128.5, 127.2, 125.6, 123.8, 114.4, 29.0; IR (KBr) ν 3105, 3064, 1653, 1579, 1550, 1436, 1416, 1363, 1270, 1223, 1161, 1064 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{13}\text{H}_{10}\text{N}_2\text{OS}$ $[\text{M}]^+$ 242.0514, found 242.0515.

Experimental procedure and results of the control experiments for influence of molecular oxygen

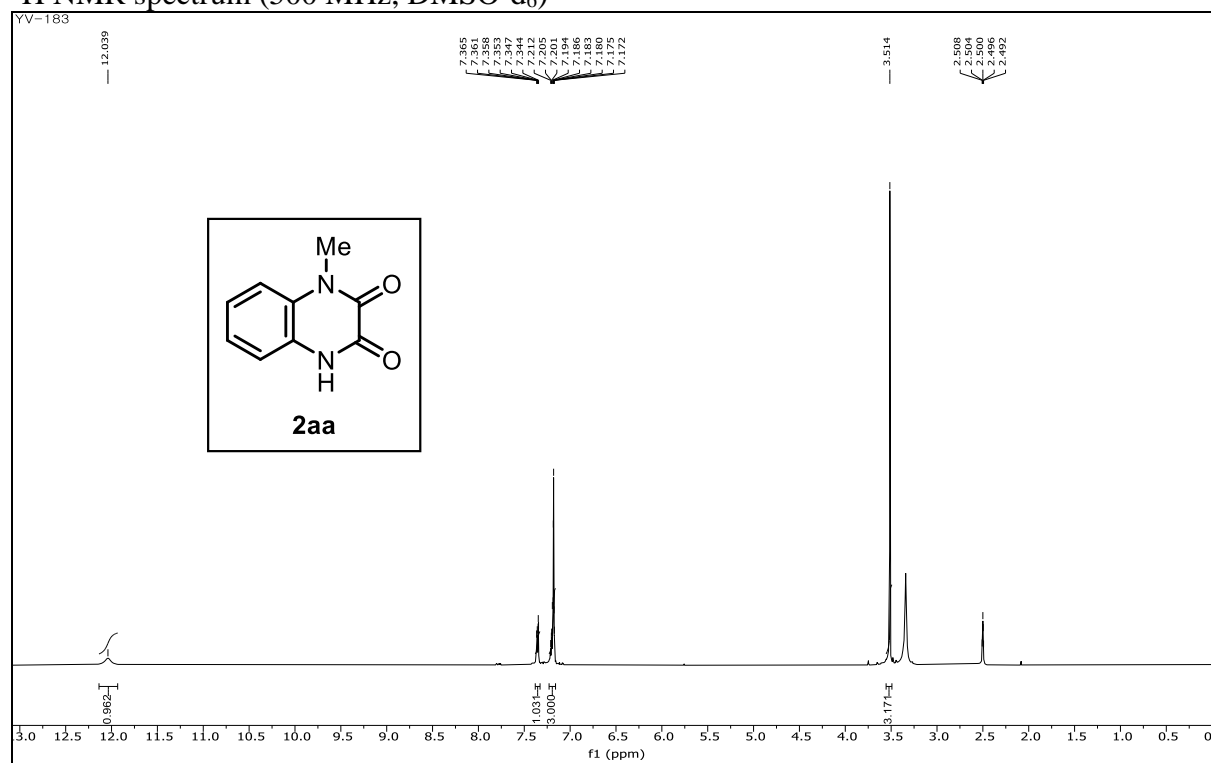


To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1H)-one (**1a**) (32 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.) was added dimethoxyethane (1 mL) at room temperature under air or O₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash chromatography (*n*-hexane/EtOAc to CH₂Cl₂/MeOH) to afford **2a**, **2aa**, and recovered **1a**.

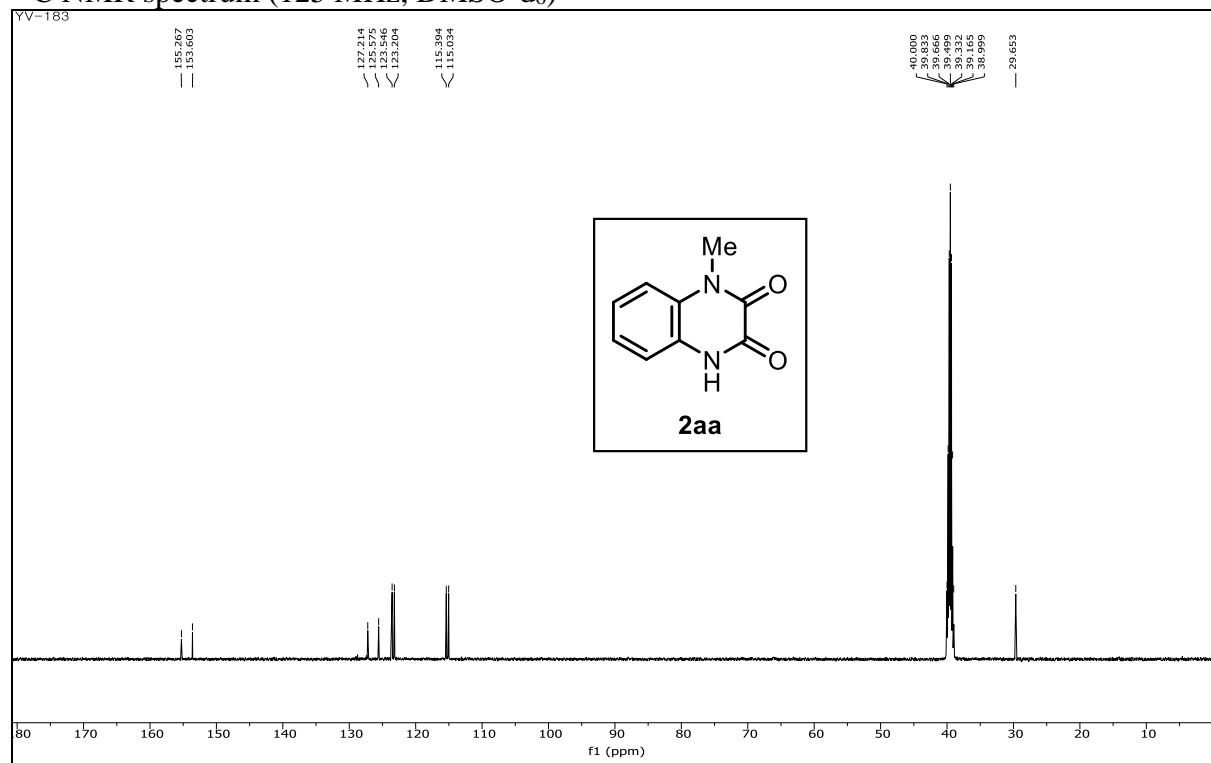
Table S1. Control experiment for the influence of molecular oxygen.

entry	atmosphere	Result (% yield)		
		1a	2a	2aa
1	air	17	61	10
2	O ₂	16	41	21

¹H NMR spectrum (500 MHz, DMSO-d₆)

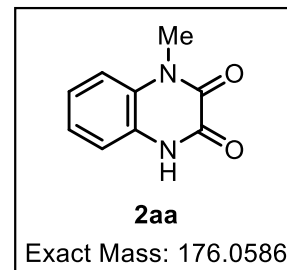
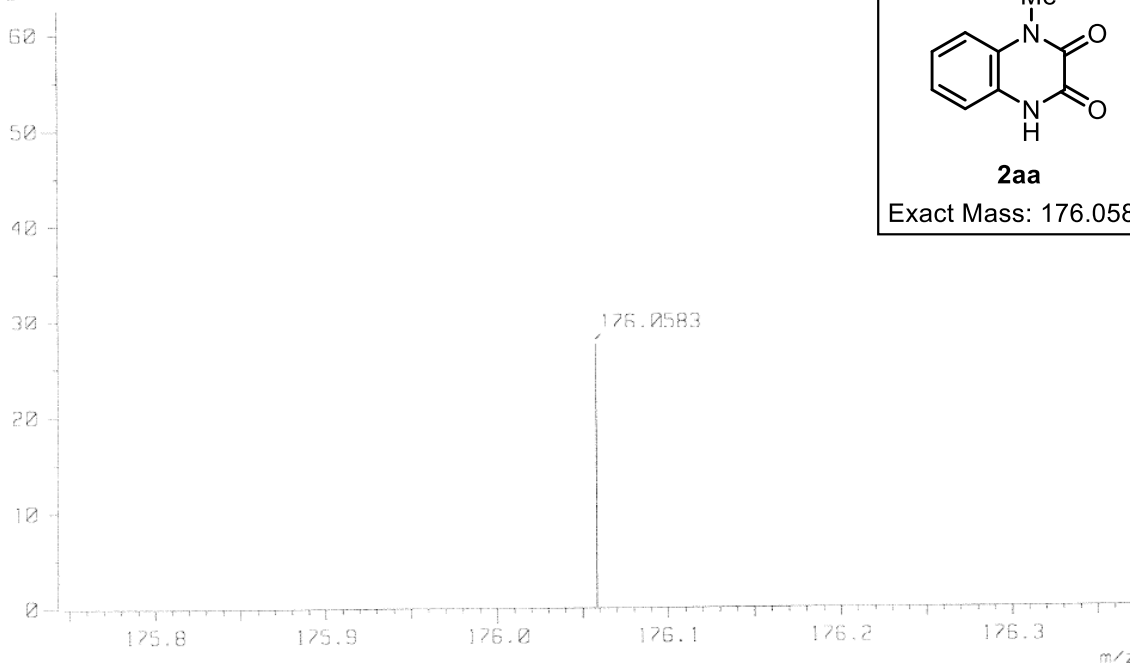


¹³C NMR spectrum (125 MHz, DMSO-d₆)



HRMS data of 2aa

[Mass Spectrum]
Data : HR-YV-183-005 Date : 10-Feb-2022 12:27
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
Spectrum Type : Normal Ion [MF-Linear]
RT : 0.54 min Scan# : 17+25+26
BP : m/z 207.1153 Int. : 37.63
Output m/z range : 175.7466 to 176.3722 Cut Level : 7.79 %
740612



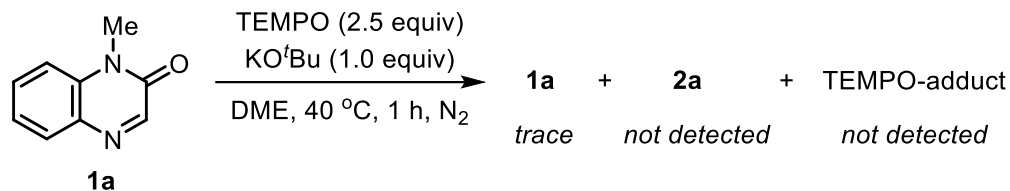
[Elemental Composition]
Data : HR-YV-183-005 Date : 10-Feb-2022 12:27
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 0.54 min Scan# : 17+25+26
Elements : C 10/0, H 10/0, O 2/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Page: 1

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
176.0583	27.4	-1.6 / -0.3	7.0	C 9 H 8 O 2 N 2

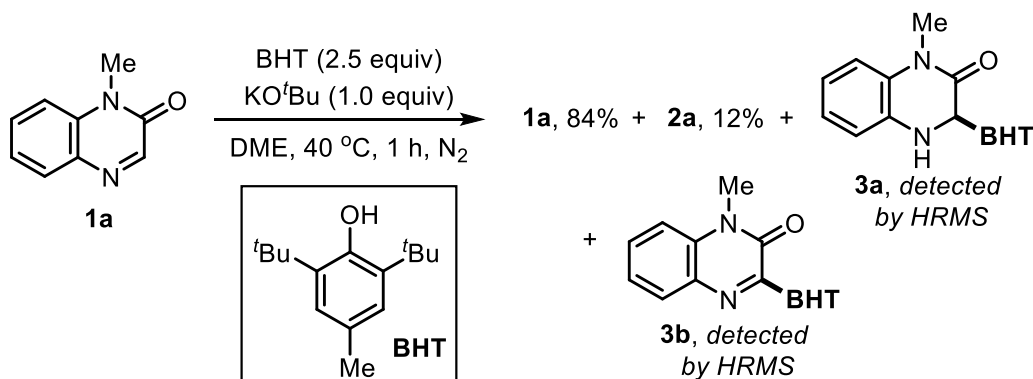
Experimental procedures and results for the radical scavenging reactions

Reaction of **1a** in the presence of TEMPO



To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1H)-one (**1a**) (32 mg, 0.2 mmol, 1.0 equiv.), KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.), and 2,2,6,6-tetramethyl-1-piperidinyloxy, free radical (TEMPO) (78.1 mg, 0.5 mmol, 2.5 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature and concentrated in vacuo. No adduct of TEMPO corresponding to **1a** was identified by the analysis of TLC. No adduct of TEMPO corresponding to **1a** was detected by either ¹H NMR (CDCl₃) or HRMS analysis of the residue.

Reaction of **1a** in the presence of BHT

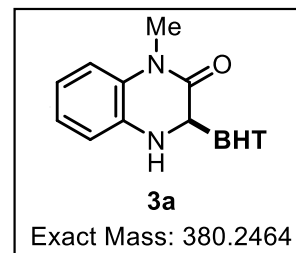
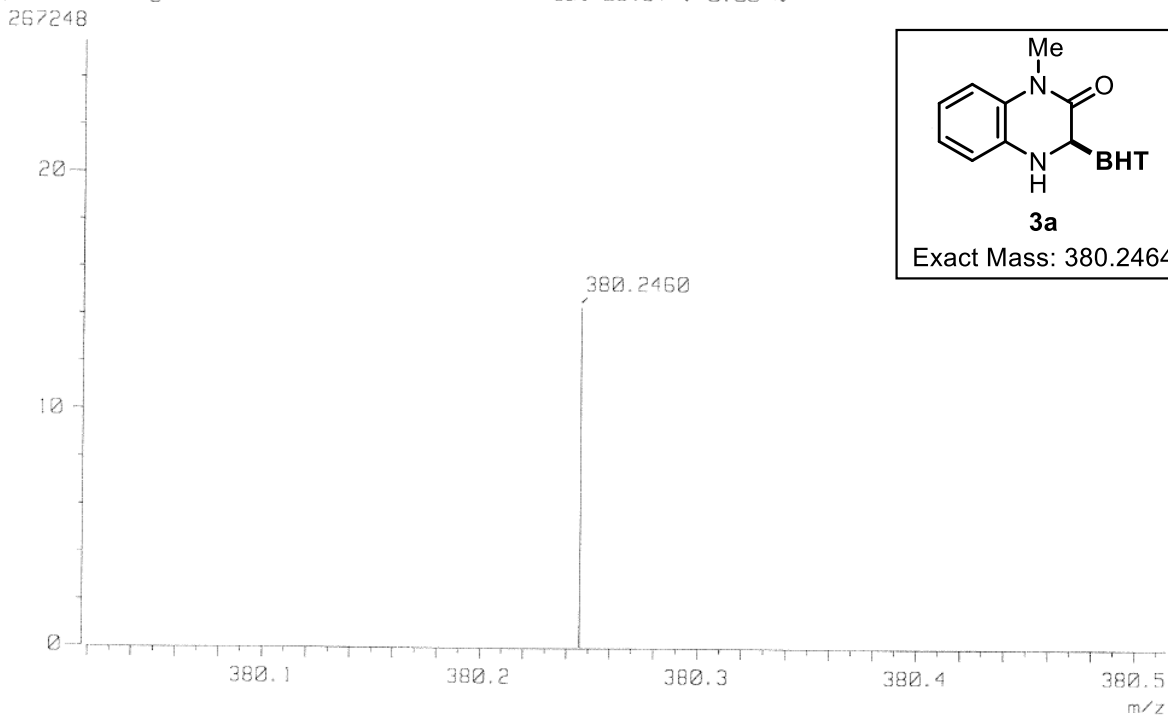


To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1H)-one (**1a**) (32 mg, 0.2 mmol, 1.0 equiv.), KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.), and butylated hydroxytoluene (BHT) (110.2 mg, 0.5 mmol, 2.5 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated in vacuo. The residue was purified by

flash column chromatography (*n*-hexane/EtOAc to CH₂Cl₂/MeOH) to afford **2a** (4.0 mg, 12%) and recovered **1a** (27.0 mg, 84%). BHT-adducts were detected by the HRMS analysis of the reaction mixture.

HRMS data of 3a

[Mass Spectrum]
Data : HRMS-YV568-001 Date : 03-Nov-2021 13:28
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
Spectrum Type : Normal Ion [MF-Linear]
RT : 1.20 min Scan# : 37
BP : m/z 368.2751 Int. : 99.94
Output m/z range : 380.0195 to 380.5147 Cut Level : 0.00 %

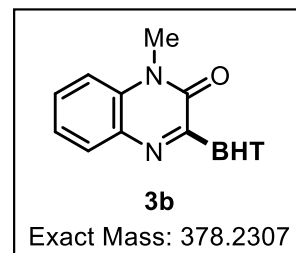
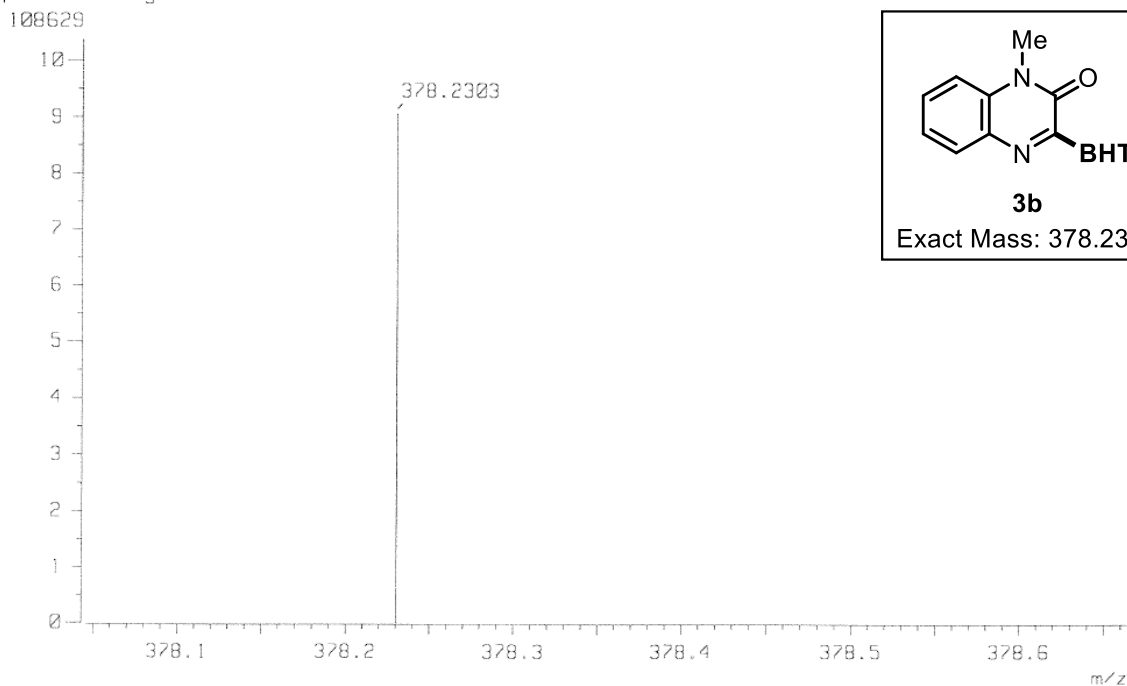


[Elemental Composition]
Data : HRMS-YV568-001 Date : 03-Nov-2021 13:28 Page: 1
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 1.20 min Scan# : 37
Elements : C 25/0, H 36/0, O 2/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
380.2460	14.3	-0.9 / -0.3	10.0 C 24 H 32 O 2 N 2

HRMS data of 3b

[Mass Spectrum]
 Data : HRMS-YV568-001 Date : 03-Nov-2021 13:28
 Sample: SKKU. Univ. Prof. Kim
 Note : SMLab Mass analysis
 Inlet : Direct Ion Mode : EI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.54 min Scan# : 47
 BP : m/z 368.2737 Int. : 99.94
 Output m/z range : 378.0460 to 378.6644 Cut Level : 0.00 %

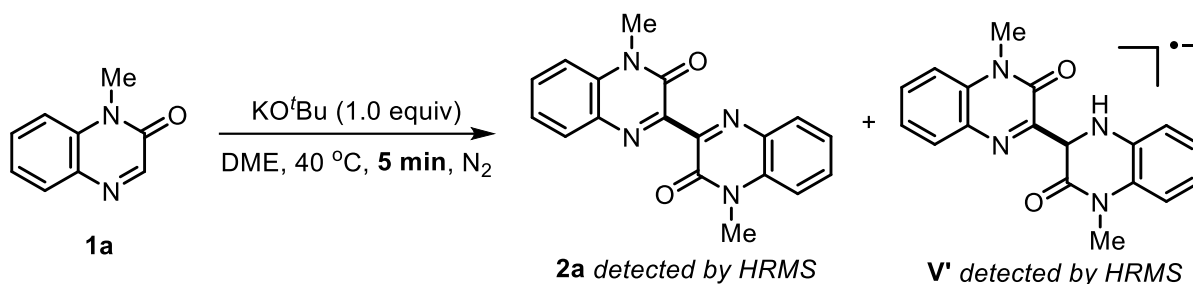


[Elemental Composition]
 Data : HRMS-YV568-001 Date : 03-Nov-2021 13:28
 Sample: SKKU. Univ. Prof. Kim
 Note : SMLab Mass analysis
 Inlet : Direct Ion Mode : EI+
 RT : 1.54 min Scan# : 47
 Elements : C 25/0, H 36/0, O 2/0, N 2/0
 Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
 Unsaturation (U.S.) : -0.5 - 70.0

Page: 1

Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
378.2303	9.1	-1.1 / -0.4	11.0 C 24 H 30 O 2 N 2

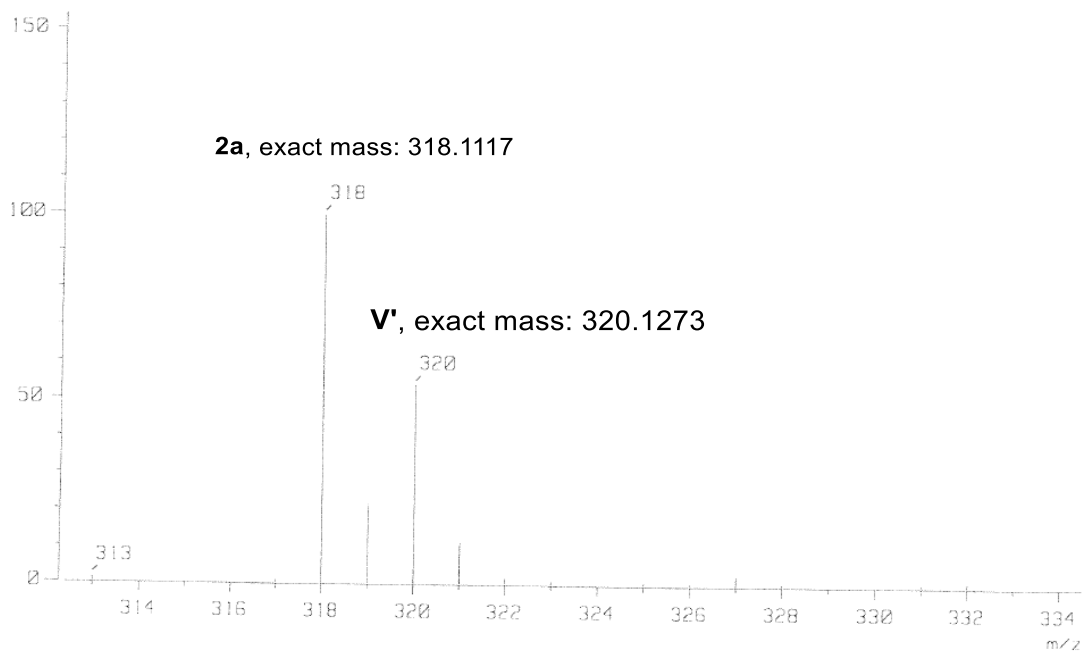
Experimental procedure for the characterization of intermediate V'



To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1*H*)-one (**1a**) (32 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 0.1 h. The reaction mixture was cooled to room temperature. Then the reaction mixture was directly analyzed by HRMS. Intermediate **V'** was detected by HRMS analysis of the reaction mixture.

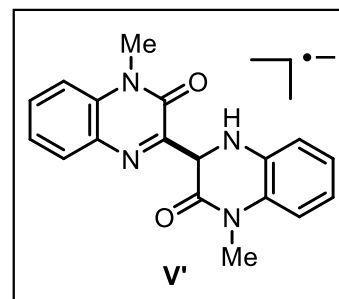
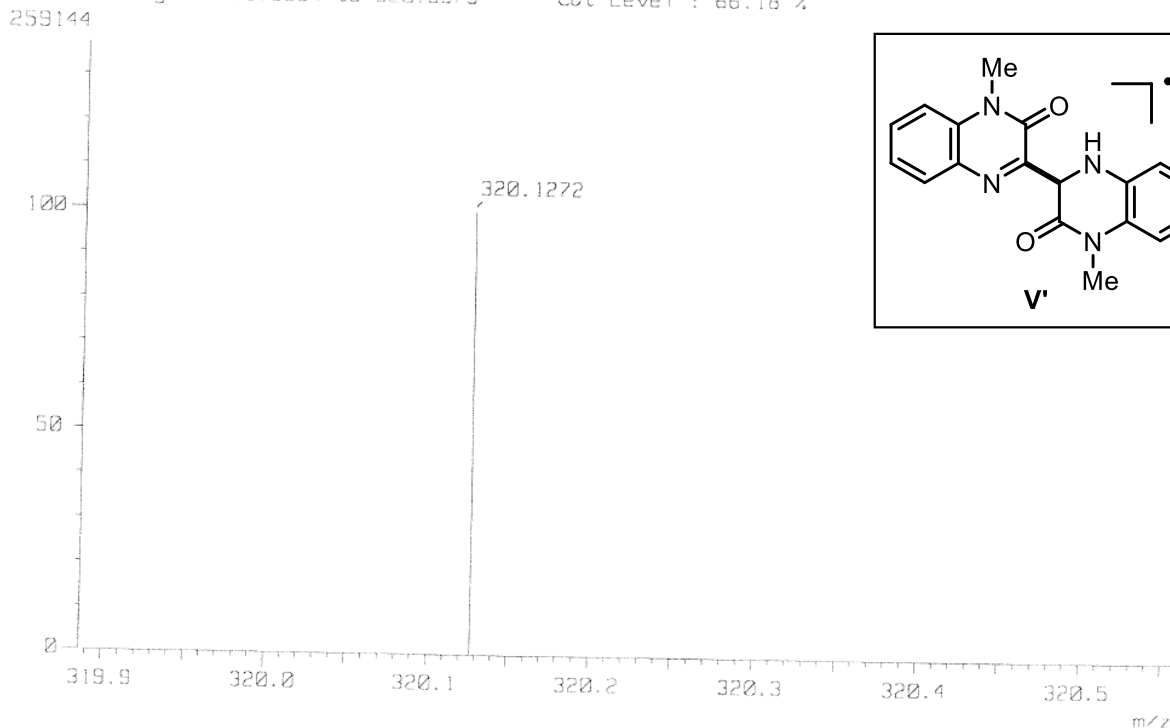
LRMS data of reaction mixture (**2a** and **V'**)

```
[ Mass Spectrum ]
Data : LR-YV456-005          Date : 10-Feb-2022 11:59
Sample:
Note : SM Lab Research Center
Inlet : Direct              Ion Mode : E1+
Spectrum Type : Normal Ion [MF-Linear]
RT : 2.60 min              Scan# : 79
BP : m/z 318.0000         Int. : 145.80
Output m/z range : 312.3876 to 334.5228   Cut Level : 0.10 %
2359183
```



HRMS data of V'

[Mass Spectrum]
 Data : HR-YV-456-001 Date : 10-Feb-2022 12:02
 Sample: SKKU Univ. Prof. Kim
 Note : SMLab Mass analysis
 Inlet : Direct Ion Mode : EI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 0.80 min Scan# : 25
 BP : m/z 320.1272 Int. : 18.03
 Output m/z range : 319.8894 to 320.5579 Cut Level : 66.18 %



[Elemental Composition]
 Data : HR-YV-456-001 Date : 10-Feb-2022 12:02
 Sample: SKKU Univ. Prof. Kim
 Note : SMLab Mass analysis
 Inlet : Direct Ion Mode : EI+
 RT : 0.80 min Scan#: 25
 Elements : C 20/0, H 26/0, O 2/0, N 5/0
 Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
 Unsaturation (U.S.) : -0.5 - 70.0

Page: 1

Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
320.1272	100.0	-0.3 / -0.1	13.0 C 18 H 16 O 2 N 4

Experimental procedure and result of EPR experiments

Experimental procedure with KO^tBu

To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1*H*)-one (**1a**) (32 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.) was added degassed dimethoxyethane (1 mL) under Ar at room temperature. The reaction mixture was degassed by freeze thawing (three cycles) with liquid nitrogen and backfilled with Ar gas. The reaction mixture was allowed to stir at 40 °C for 0.1 h under Ar gas. The reaction mixture was cooled to room temperature and 0.3 mL of it was transferred to a Low Pressure/Vacuum Suprasil EPR tube previously filled with Ar gas. The mixture in the EPR tube was degassed by freeze thawing (three cycles) with liquid nitrogen and subsequently analysed by EPR at 100 K.

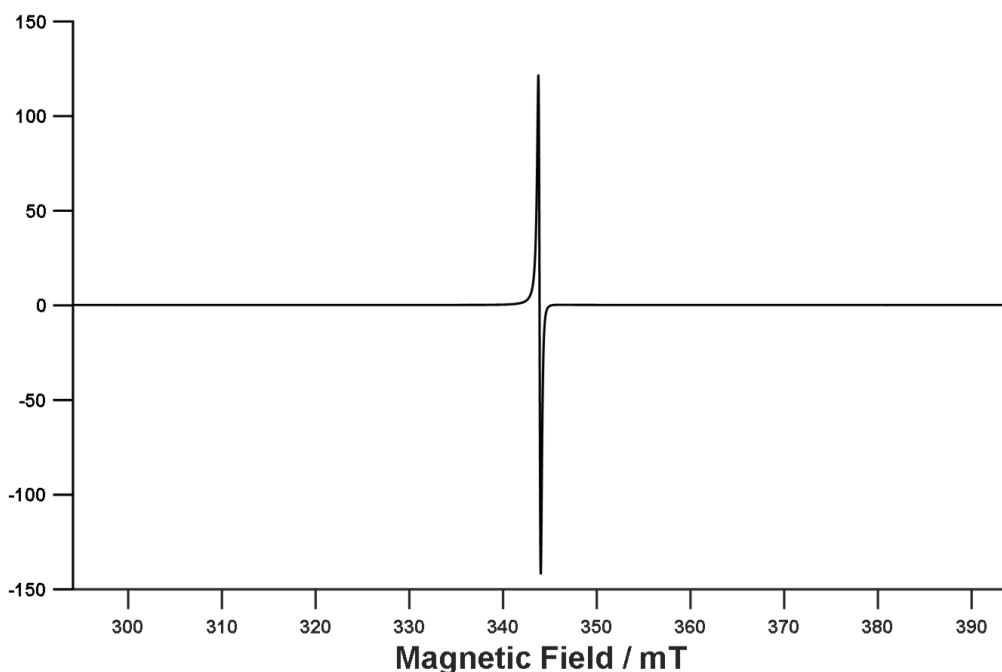


Figure S1: EPR spectra of the reaction mixture of **1a** and KO^tBu in dimethoxyethane recorded at 100 K. Electron paramagnetic resonance (EPR) studies were performed using a Bruker EMX/Plus spectrometer equipped with a dual mode cavity (ER 4116DM). Microwave frequencies: 9.64 GHz (perpendicular mode) and 9.4 GHz (parallel mode), modulation amplitude 1G, modulation frequency 100 kHz, and microwave power 0.001 mW.

Experimental procedure with NaHMDS

To an oven-dried schlenk tube charged with 1-methylquinoxalin-2(1*H*)-one (**1a**) (16 mg, 0.1 mmol, 1.0 equiv.) was added degassed THF (10 mL) under Ar at room temperature. NaHMDS (1.0 M in THF, 0.1 mL, 0.1 mmol, 1.0 equiv.) was added dropwise in the reaction mixture under Ar at 0 °C. The reaction mixture was degassed by freeze thawing (three cycles) with liquid nitrogen, backfilled with Ar gas and allowed to stir at room temperature for 0.1 h. Then 0.3 mL of the reaction mixture was transferred to a Low Pressure/Vacuum Suprasil EPR tube previously filled with Ar gas. The mixture in the EPR tube was degassed by freeze thawing (three cycles) with liquid nitrogen and subsequently analysed by EPR at 20 K.

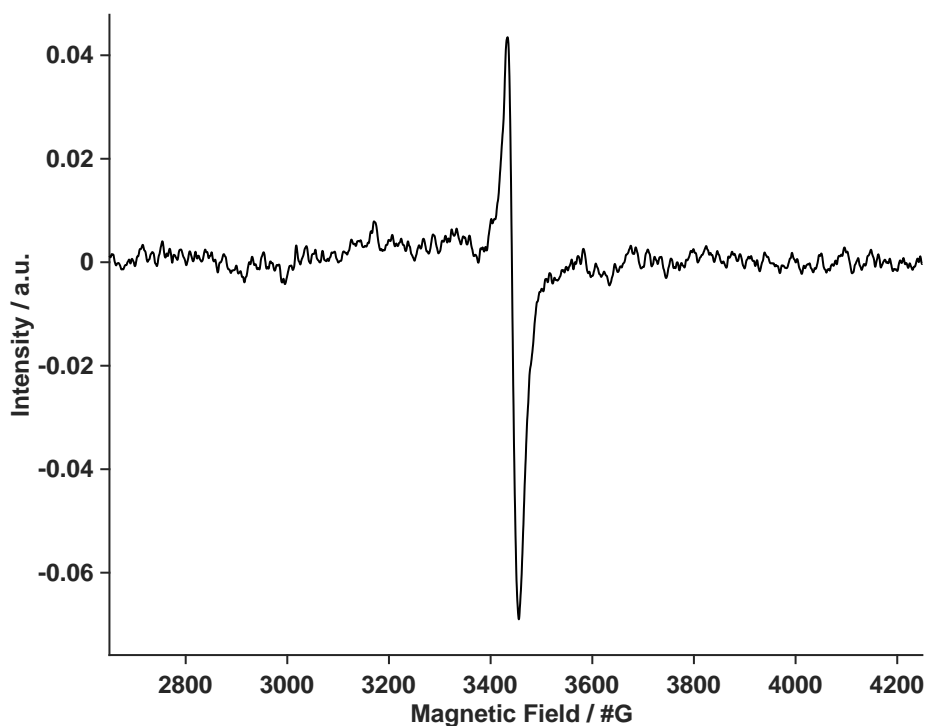


Figure S2: EPR spectra of the reaction mixture of **1a** and NaHMDS in THF recorded at 20 K. Electron paramagnetic resonance (EPR) studies were performed using a Bruker EMX/Plus spectrometer equipped with a dual mode cavity (ER 4116DM). Microwave frequencies: 9.65 GHz (perpendicular mode), modulation amplitude 5G, modulation frequency 100 kHz, and microwave power 0.93 mW.

Electrochemical measurements

Cyclic voltammograms

Electrochemical study was performed using a CH instrument (CHI600E model). The redox potentials of substrates (vs Ag/AgCl) were determined through cyclic voltammetry using a 5.0 mM solution of that material in 50 mM solution of Bu₄NPF₆ (purged DMF with N₂). Measurements employed a glassy carbon working electrode, platinum wire counter electrode, 3.0 M NaCl Ag/AgCl reference electrode, a scan rate 100 mV/s. The obtained value was referenced to Ag/AgCl and converted to SCE by subtracting 0.032 V.

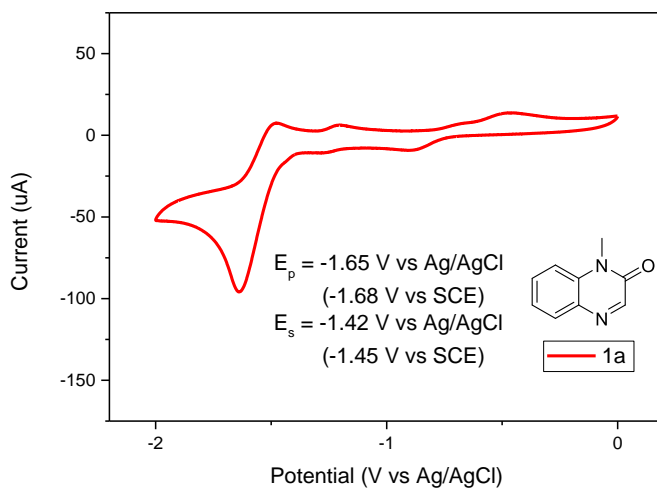


Figure S3. Cyclic voltammogram study for **1a**

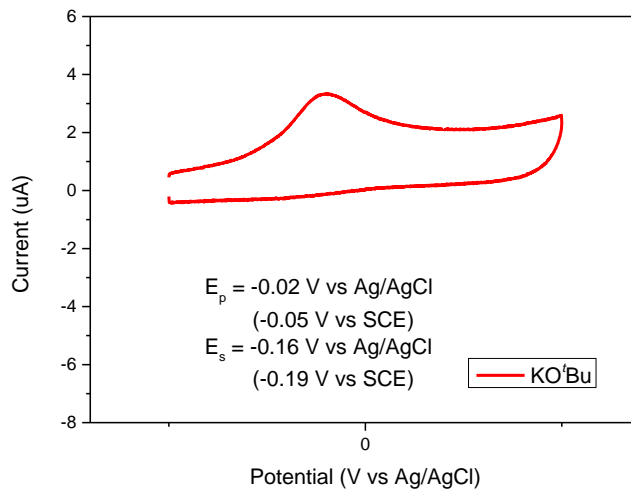


Figure S4. Cyclic voltammogram study for **KO'Bu**

General procedure for the C3-homocoupling of quinoxalinones (2a–2h, 2l–2r, 2t and 2u)

To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1*H*)-one (**1a**) (32 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (CH₂Cl₂/MeOH = 98:2) to afford 30.9 mg of **2a** in 98% yield.

Experimental procedure for the synthesis of dimerized quinoxalinones (2i, 2k and 2s)

To an oven-dried sealed tube charged with 1-(*m*-tolyl)quinoxalin-2(1*H*)-one (**1i**) (47.2 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.) was added THF (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (using only CH₂Cl₂) to afford 33.0 mg of **2i** in 70% yield.

Experimental procedure for the synthesis of dimerized quinoxalinone 2j

To an oven-dried sealed tube charged with ethyl 3-(2-oxoquinoxalin-1(2*H*)-yl)benzoate (**1j**) (58.8 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (17.9 mg, 0.16 mmol, 0.8 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (CH₂Cl₂/MeOH = 98:2) to afford 37.0 mg of **2j** in 63% yield.

Experimental procedure for the synthesis of dimerized quinoxalinone **2v**

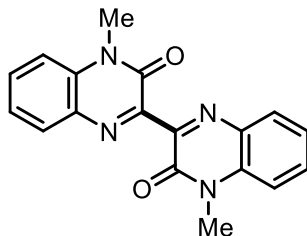
To an oven-dried sealed tube charged with 1-methyl-7-nitroquinoxalin-2(1*H*)-one (**1v**) (41.0 mg, 0.2 mmol, 1.0 equiv.) and KO^tBu (11.2 mg, 0.1 mmol, 0.5 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature and directly purified by flash column chromatography (only using CH₂Cl₂) to afford 8.2 mg of **2v** in 20% yield.

Experimental procedure for gram-scale reaction of **2a**

To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1*H*)-one (**1a**) (1 g, 6.24 mmol, 1.0 equiv.) and KO^tBu (0.7 g, 6.24 mmol, 1.0 equiv.) was added dimethoxyethane (32 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (300 mL, 9:1) and stirred under air for 2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (CH₂Cl₂/MeOH = 98:2) to afford 0.9 g of **2a** in 91% yield.

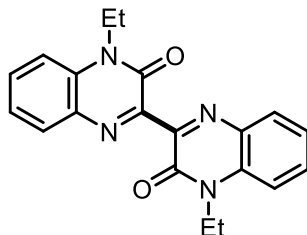
Characterization data for the products (2a–2v)

4,4'-Dimethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2a)



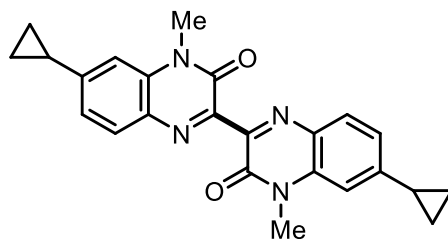
31.2 mg (98%); off-white solid; mp = 252.8–254.3 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.00 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.64 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 2H), 7.42–7.37 (m, 4H), 3.77 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 154.6, 154.2, 134.2, 133.1, 131.7, 131.2, 124.0, 113.9, 29.3; IR (KBr) ν 3055, 2853, 1647, 1602, 1557, 1472, 1417, 1363, 1268, 1224, 1211, 1126, 1039 cm⁻¹; HRMS (quadrupole, EI) calcd for C₁₈H₁₄N₄O₂ [M]⁺ 318.1117, found 318.1116.

4,4'-Diethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2b)



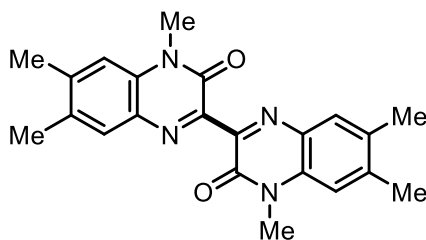
30.1 mg (87%); pale yellow solid; mp = 237.1–239.5 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.01 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.64 (ddd, *J* = 8.4, 7.2, 1.6 Hz, 2H), 7.42–7.36 (m, 4H), 4.39 (q, *J* = 7.2 Hz, 4H), 1.41 (t, *J* = 7.2 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 154.7, 153.7, 133.5, 133.1, 131.6, 131.5, 123.8, 113.8, 37.5, 12.5; IR (KBr) ν 3054, 2979, 2935, 1637, 1601, 1588, 1557, 1465, 1370, 1310, 1270, 1155, 1066, 984 cm⁻¹; HRMS (quadrupole, EI) calcd for C₂₀H₁₈N₄O₂ [M]⁺ 346.1430, found 346.1427.

6,6'-Dicyclopropyl-4,4'-dimethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2c)



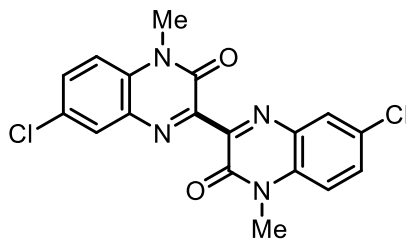
31.8 mg (80%); pale yellow solid; mp = 158.3–160.7 °C; ^1H NMR (700 MHz, CDCl_3) δ 7.85 (d, $J = 8.4$ Hz, 2H), 7.07 (d, $J = 1.4$ Hz, 2H), 7.00 (dd, $J = 8.4, 1.4$ Hz, 2H), 3.74 (s, 6H), 2.09–2.05 (m, 2H), 1.15–1.12 (m, 4H), 0.87–0.85 (m, 4H); ^{13}C NMR (175 MHz, CDCl_3) δ 154.5, 153.1, 149.0, 134.2, 131.5, 131.0, 121.0, 111.0, 29.1, 16.5, 10.7; IR (KBr) ν 3057, 3005, 1746, 1648, 1608, 1550, 1470, 1441, 1419, 1363, 1269, 1223 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{24}\text{H}_{22}\text{N}_4\text{O}_2$ $[\text{M}]^+$ 398.1743, found 398.1742.

4,4',6,6',7,7'-Hexamethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2d)



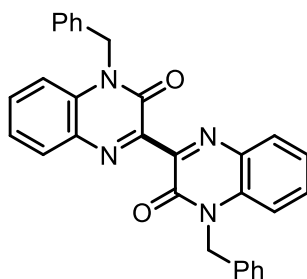
24.0 mg (64%); pale yellow solid; mp = 314.5–316.0 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.73 (s, 2H), 7.13 (s, 2H), 3.73 (s, 6H), 2.45 (s, 6H), 2.35 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.4, 153.6, 141.6, 132.9, 132.3, 131.7, 131.2, 114.4, 29.1, 20.9, 19.4; IR (KBr) ν 3057, 2923, 2855, 1642, 1616, 1547, 1469, 1359, 1268, 1223, 1139, 1047, 1007 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{22}\text{H}_{22}\text{N}_4\text{O}_2$ $[\text{M}]^+$ 374.1743, found 374.1741.

7,7'-Dichloro-4,4'-dimethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2e)



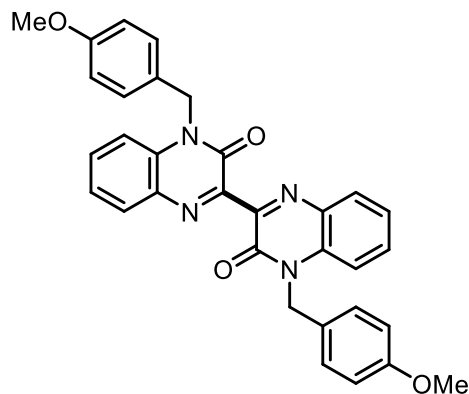
30.2 mg (78%); yellow solid; mp = 319.0–320.4 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.98 (d, J = 2.4 Hz, 2H), 7.61 (dd, J = 8.8, 2.4 Hz, 2H), 7.34 (d, J = 8.8 Hz, 2H), 3.75 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.5, 153.8, 133.5, 132.9, 131.9, 130.4, 129.5, 116.0, 29.5; IR (KBr) ν 3056, 2923, 2852, 1652, 1584, 1554, 1486, 1458, 1363, 1269, 1223, 1090, 1062 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{N}_4\text{O}_2$ $[\text{M}]^+$ 386.0337, found 386.0335.

4,4'-Dibenzyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2f)



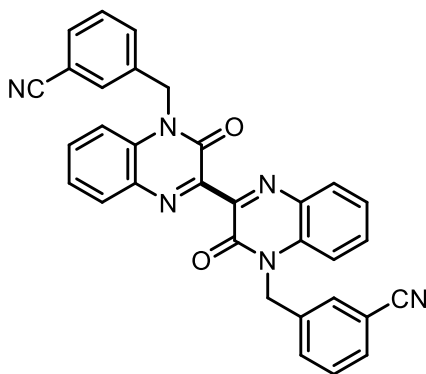
35.3 mg (75%); yellow solid; mp = 219.8–222.4 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.02 (dd, J = 8.0, 1.2 Hz, 2H), 7.51 (ddd, J = 8.4, 7.2, 1.6 Hz, 2H), 7.38–7.30 (m, 12H), 7.29–7.23 (m, 2H), 5.59 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.8, 154.4, 135.2, 133.5, 133.4, 131.7, 131.3, 129.1, 127.9, 127.2, 124.1, 114.8, 46.1; IR (KBr) ν 3057, 2924, 2854, 1646, 1600, 1558, 1453, 1362, 1314, 1267, 1223, 1160, 1007 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{30}\text{H}_{22}\text{N}_4\text{O}_2$ $[\text{M}]^+$ 470.1743, found 470.1738.

4,4'-Bis(4-methoxybenzyl)-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2g)



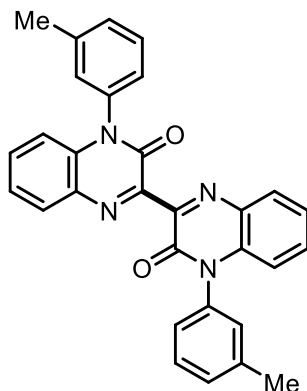
37.1 mg (70%); yellow solid; mp = 187.2–190.0 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.01 (dd, J = 8.0, 1.2 Hz, 2H), 7.52 (ddd, J = 8.4, 7.2, 1.6 Hz, 2H), 7.39–7.33 (m, 4H), 7.28–7.26 (m, 4H), 6.86–6.84 (m, 4H), 5.52 (s, 4H), 3.76 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.3, 154.9, 154.4, 133.5, 133.4, 131.6, 131.3, 128.7, 127.3, 124.0, 114.8, 114.5, 55.4, 46.4; IR (KBr) ν 3055, 3004, 2931, 2835, 1643, 1600, 1511, 1464, 1362, 1304, 1245, 1177, 1071, 1032 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{32}\text{H}_{26}\text{N}_4\text{O}_4$ $[\text{M}]^+$ 530.1954, found 530.1953.

3,3'-((3,3'-Dioxo-[2,2'-biquinoxaline]-4,4'(3*H*,3'*H*)-diyl)bis(methylene))dibenzonitrile (2h)



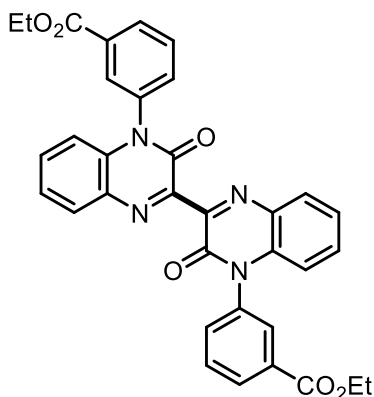
30.2 mg (58%); off-white solid; mp = 305.9–307.3 °C; ^1H NMR (700 MHz, CDCl_3) δ 8.07 (dd, J = 8.4, 1.4 Hz, 2H), 7.65–7.64 (m, 2H), 7.59–7.54 (m, 6H), 7.45 (t, J = 7.7 Hz, 2H), 7.42 (ddd, J = 8.4, 7.0, 1.4 Hz, 2H), 7.25 (dd, J = 8.4, 1.4 Hz, 2H), 5.59 (s, 4H); ^{13}C NMR (175 MHz, CDCl_3) δ 154.4, 154.2, 136.8, 133.5, 133.1, 132.1, 131.9, 131.8, 131.7, 130.9, 130.2, 124.6, 118.4, 114.2, 113.5, 45.3; IR (KBr) ν 3057, 2226, 1652, 1601, 1561, 1484, 1465, 1438, 1363, 1313, 1268, 1223, 1077 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{32}\text{H}_{20}\text{N}_6\text{O}_2$ $[\text{M}]^+$ 520.1648, found 520.1646.

4,4'-Di-*m*-tolyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2i)



33.0 mg (70%); off-white solid; mp = 285.9–287.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.04 (dd, $J = 7.6, 1.6$ Hz, 2H), 7.46 (t, $J = 7.6$ Hz, 2H), 7.44–7.39 (m, 2H), 7.38–7.30 (m, 4H), 7.15–7.11 (m, 4H), 6.77 (dd, $J = 8.4, 1.6$ Hz, 2H), 2.41 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.3, 153.8, 140.6, 140.5, 135.2, 135.1, 133.0, 131.2, 130.7, 130.4, 130.1, 128.9, 125.4, 124.0, 115.8, 21.5; IR (KBr) ν 3054, 2988, 1661, 1600, 1555, 1462, 1362, 1304, 1268, 1223, 1159, 1049 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{30}\text{H}_{22}\text{N}_4\text{O}_2$ $[\text{M}]^+$ 470.1743, found 470.1739.

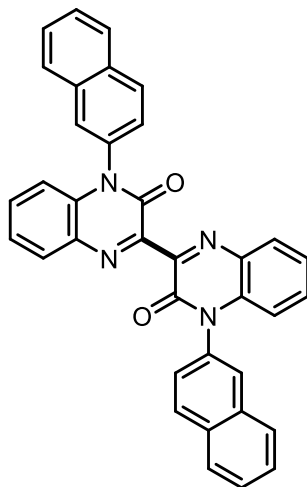
Diethyl 3,3'-(3,3'-dioxo-[2,2'-biquinoxaline]-4,4'(3*H*,3'*H*)-diyl)dibenzoate (2j)



35.2 mg (60%); off-white solid; mp = 202.1–204.9 °C; ^1H NMR (700 MHz, CDCl_3) δ 8.23 (dt, $J = 7.7, 1.4$ Hz, 2H), 8.06 (dd, $J = 8.4, 1.4$ Hz, 2H), 8.03 (q, $J = 1.4$ Hz, 2H), 7.68 (t, $J = 7.7$ Hz, 2H), 7.56–7.54 (m, 2H), 7.44 (ddd, $J = 9.1, 7.7, 1.4$ Hz, 2H), 7.39 (ddd, $J = 9.1, 7.7, 1.4$ Hz, 2H), 6.72 (dd, $J = 8.4, 1.4$ Hz, 2H), 4.42–4.33 (m, 4H), 1.37 (td, $J = 7.7, 1.4$ Hz, 6H); ^{13}C NMR (175 MHz, CDCl_3) δ 165.4, 155.0, 153.6, 135.5, 134.8, 133.1 (two carbons overlap), 133.0, 131.5, 131.0, 130.9, 130.5, 129.8, 124.4, 115.5, 61.6, 14.4; IR (KBr) ν 3057, 2987, 1716, 1658,

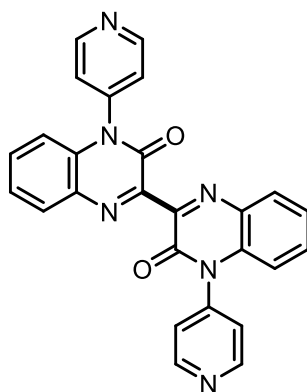
1602, 1556, 1463, 1442, 1365, 1268, 1223, 1100, 1080, 1051 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{34}\text{H}_{26}\text{N}_4\text{O}_6$ $[\text{M}]^+$ 586.1852, found 586.1852.

4,4'-Di(naphthalen-2-yl)-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2k)



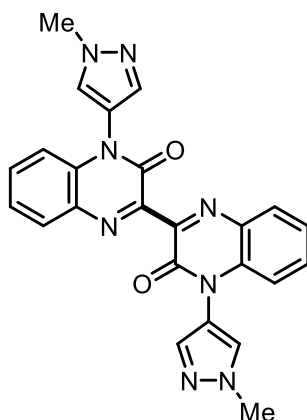
38.5 mg (71%); yellow solid; mp = 331.2–332.8 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3) δ 8.10–8.04 (m, 4H), 7.94 (d, J = 8.4 Hz, 2H), 7.88–7.85 (m, 4H), 7.61–7.53 (m, 4H), 7.42–7.33 (m, 6H), 6.81–6.78 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.3, 154.0, 135.2, 133.9, 133.5, 133.1, 132.7, 131.3, 130.8, 130.6, 128.4, 128.1, 127.9, 127.4, 127.0, 125.6, 124.2, 115.9; IR (KBr) ν 3056, 3007, 1658, 1600, 1555, 1463, 1362, 1315, 1306, 1268, 1223, 1049 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{36}\text{H}_{22}\text{N}_4\text{O}_2$ $[\text{M}]^+$ 542.1743, found 542.1738.

4,4'-Di(pyridin-4-yl)-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2l)



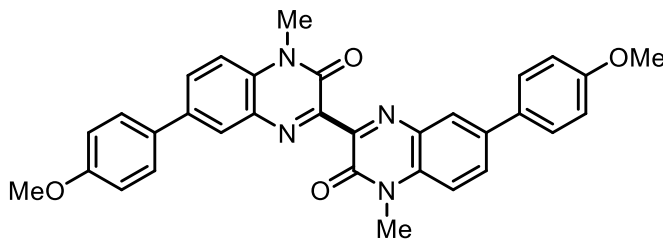
28.9 mg (65%); orange solid; mp = 341.7–343.9 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.90 (dd, *J* = 4.4, 1.6 Hz, 4H), 8.07 (dd, *J* = 7.6, 1.6 Hz, 2H), 7.48 (td, *J* = 7.6, 1.6 Hz, 2H), 7.42 (td, *J* = 8.0, 1.2 Hz, 2H), 7.37 (dd, *J* = 4.8, 2.0 Hz, 4H), 6.76 (dd, *J* = 8.0, 1.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 154.5, 152.9, 152.3, 143.1, 133.7, 132.9, 131.9, 131.2, 124.9, 123.7, 115.1; IR (KBr) ν 3055, 1668, 1602, 1580, 1546, 1463, 1415, 1363, 1320, 1269, 1223 cm⁻¹; HRMS (quadrupole, EI) calcd for C₂₆H₁₆N₆O₂ [M]⁺ 444.1335, found 444.1336.

4,4'-Bis(1-methyl-1*H*-pyrazol-4-yl)-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2m)



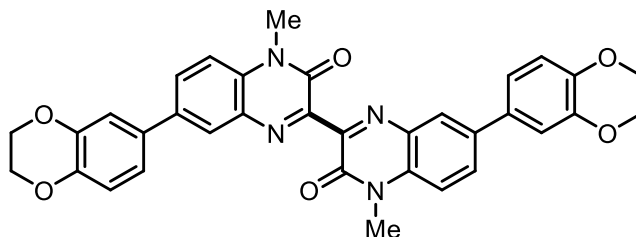
35.6 mg (79%); yellow solid; mp = > 350 °C; ¹H NMR (700 MHz, CDCl₃) δ 8.01 (dd, *J* = 7.7, 1.4 Hz, 2H), 7.65 (s, 2H), 7.61 (s, 2H), 7.49 (ddd, *J* = 8.4, 7.0, 1.4 Hz, 2H), 7.39–7.36 (m, 2H), 7.18 (dd, *J* = 8.4, 1.4 Hz, 2H), 4.00 (s, 6H); ¹³C NMR (175 MHz, CDCl₃) δ 154.7, 153.8, 137.3, 135.2, 133.1, 131.5, 130.9, 128.9, 124.4, 116.2, 115.6, 40.0; IR (KBr) ν 3117, 3055, 1663, 1601, 1555, 1461, 1363, 1303, 1269, 1223, 1053, 979 cm⁻¹; HRMS (quadrupole, EI) calcd for C₂₄H₁₈N₈O₂ [M]⁺ 450.1553, found 450.1550.

7,7'-Bis(4-methoxyphenyl)-4,4'-dimethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2n)



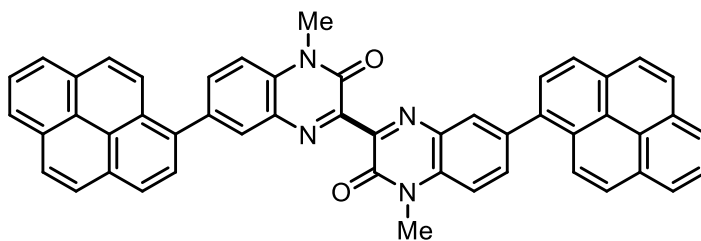
52.5 mg (99%); orange solid; mp = 279.4–281.7 °C; ¹H NMR (700 MHz, CDCl₃) δ 8.21 (d, *J* = 2.1 Hz, 2H), 7.86 (dd, *J* = 9.1, 2.1 Hz, 2H), 7.60 (d, *J* = 9.1 Hz, 4H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.02 (d, *J* = 9.1 Hz, 4H), 3.87 (s, 6H), 3.80 (s, 6H); ¹³C NMR (175 MHz, CDCl₃) δ 159.70, 155.0, 154.2, 136.8, 133.5, 132.9, 131.8, 130.2, 128.5, 128.1, 114.7, 114.3, 55.6, 29.4; IR (KBr) ν 3056, 3005, 2835, 1648, 1609, 1555, 1492, 1362, 1274, 1249, 1223, 1182, 1074, 1041 cm⁻¹; HRMS (quadrupole, EI) calcd for C₃₂H₂₆N₄O₄ [M]⁺ 530.1954, found 530.1957.

7,7'-Bis(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-4,4'-dimethyl-[2,2'-biquinoxaline]-3,3'-(4*H*,4'*H*)-dione (2o)



55.1 mg (94%); red solid; mp = 280.3–282.1 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.19 (d, *J* = 2.0 Hz, 2H), 7.82 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.42 (d, *J* = 8.8 Hz, 2H), 7.18–7.17 (m, 2H), 7.16–7.13 (m, 2H), 6.96 (d, *J* = 8.4 Hz, 2H), 4.30 (s, 8H), 3.79 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 155.0, 154.1, 144.1, 143.7, 136.6, 133.4, 133.0, 132.8, 130.2, 128.6, 120.1, 118.0, 115.9, 114.3, 64.6, 64.5, 29.4; IR (KBr) ν 3055, 2985, 2931, 2878, 1643, 1585, 1489, 1458, 1362, 1305, 1281, 1246, 1169, 1065 cm⁻¹; HRMS (quadrupole, FAB) calcd for C₃₄H₂₇N₄O₆ [M+H]⁺ 587.1931, found 587.1930.

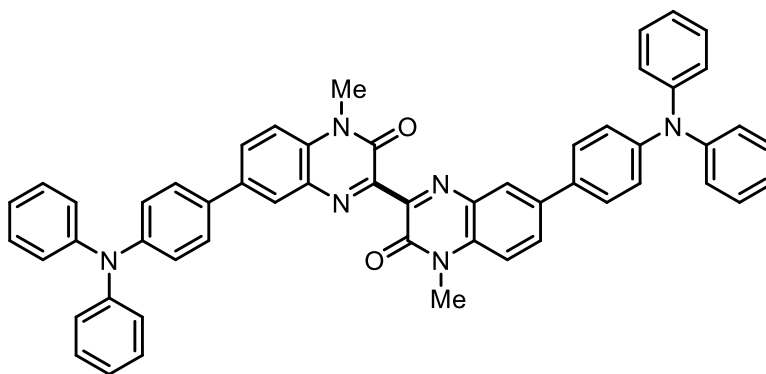
4,4'-Dimethyl-7,7'-di(pyren-1-yl)-[2,2'-biquinoxaline]-3,3'-(4*H*,4'*H*)-dione (2p)



27.0 mg (75%); yellow solid; mp = > 350 °C; ¹H NMR (700 MHz, CDCl₃) δ 8.34 (d, *J* = 2.1 Hz, 2H), 8.26 (d, *J* = 7.7 Hz, 2H), 8.22 (dd, *J* = 7.7, 1.4 Hz, 2H), 8.19–8.17 (m, 4H), 8.12 (s,

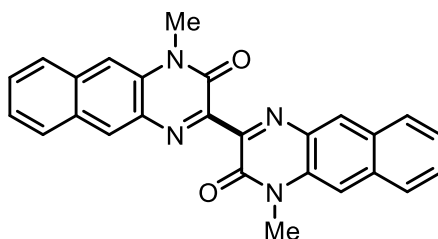
4H), 8.06–8.05 (m, 2H), 8.04–8.02 (m, 4H), 7.93 (dd, $J = 8.4, 2.1$ Hz, 2H), 7.59 (d, $J = 7.0$ Hz, 2H), 3.91 (s, 6H); ^{13}C NMR (175 MHz, CDCl_3) δ 155.3, 154.3, 137.3, 135.6, 134.1, 133.5, 133.2, 132.7, 131.6, 131.2, 131.1, 128.7, 128.1, 127.9, 127.8, 127.5, 126.3, 125.5, 125.2, 125.1, 125.0, 124.9, 124.8, 114.1, 29.5; IR (KBr) ν 3055, 1648, 1611, 1583, 1554, 1490, 1418, 1362, 1268, 1223 cm^{-1} ; HRMS (quadrupole, FAB) calcd for $\text{C}_{50}\text{H}_{31}\text{N}_4\text{O}_2$ $[\text{M}+\text{H}]^+$ 719.2447, found 719.2446.

7,7'-Bis(4-(diphenylamino)phenyl)-4,4'-dimethyl-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2q)



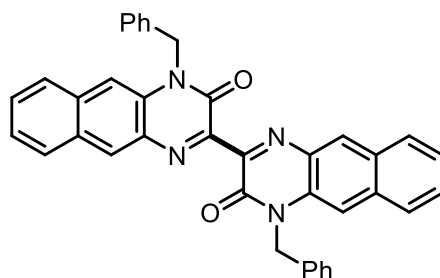
60.4 mg (75%); brown solid; mp = 293.5–295.5 $^{\circ}\text{C}$; ^1H NMR (700 MHz, CDCl_3) δ 8.23 (d, $J = 2.1$ Hz, 2H), 7.89 (dd, $J = 8.4, 2.1$ Hz, 2H), 7.55–7.53 (m, 4H), 7.44 (d, $J = 8.4$ Hz, 2H), 7.29–7.27 (m, 8H), 7.16–7.14 (m, 12H), 7.06–7.04 (m, 4H), 3.80 (s, 6H); ^{13}C NMR (175 MHz, CDCl_3) δ 155.0, 154.1, 147.9, 147.7, 136.6, 133.5, 133.0, 132.7, 130.0, 129.5, 128.4, 127.6, 124.8, 123.8, 123.4, 114.3, 29.4; IR (KBr) ν 3057, 1653, 1588, 1554, 1519, 1490, 1459, 1417, 1362, 1312, 1271, 1222, 1074 cm^{-1} ; HRMS (quadrupole, FAB) calcd for $\text{C}_{54}\text{H}_{41}\text{N}_6\text{O}_2$ $[\text{M}+\text{H}]^+$ 805.3291, found 805.2389.

4,4'-Dimethyl-[2,2'-bibenzo[*g*]quinoxaline]-3,3'(4*H*,4'*H*)-dione (2r)



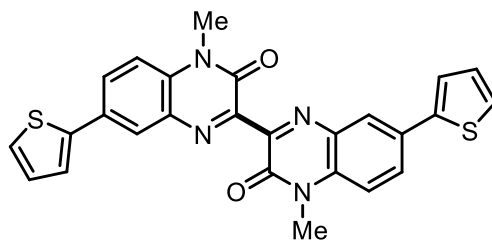
40.0 mg (95%); pale red solid; mp = 252.4–254.1 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.01 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.66 (s, 2H), 7.60 (s, 2H), 7.49 (ddd, *J* = 8.8, 7.6, 1.6 Hz, 2H), 7.37 (ddd, *J* = 8.4, 7.6, 1.6 Hz, 2H), 7.17 (dd, *J* = 8.4, 1.2 Hz, 2H), 4.00 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 154.6, 153.8, 137.4, 135.2, 133.1, 131.5, 130.9, 128.9, 124.4, 116.2, 115.7, 39.9; IR (KBr) ν 3118, 3056, 3006, 1664, 1600, 1552, 1460, 1417, 1363, 1271, 1223, 978 cm⁻¹; HRMS (quadrupole, EI) calcd for C₂₆H₁₈N₄O₂ [M]⁺ 418.1430, found 418.1426.

4,4'-Dibenzyl-[2,2'-bibenzo[*g*]quinoxaline]-3,3'(4*H*,4'*H*)-dione (2s)



37.1 mg (65%); yellow solid; mp = 267.0–270.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.56 (s, 2H), 7.98 (dd, *J* = 8.5, 1.0 Hz, 2H), 7.81 (dd, *J* = 8.5, 1.0 Hz, 2H), 7.65 (s, 2H), 7.56 (ddd, *J* = 8.0, 6.5, 1.5 Hz, 2H), 7.48 (ddd, *J* = 8.5, 7.0, 1.5 Hz, 2H), 7.42–7.38 (m, 4H), 7.34 (t, *J* = 7.5 Hz, 4H), 7.26 (t, *J* = 7.5 Hz, 2H), 5.66 (s, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 155.3, 154.4, 135.3, 134.4, 132.7, 131.3, 131.2, 129.9, 129.2, 128.9, 128.6, 127.9, 127.6, 127.2, 125.7, 111.5, 46.1; IR (KBr) ν 3058, 3031, 2925, 1654, 1627, 1596, 1454, 1363, 1269, 1223, 1092, 1071 cm⁻¹; HRMS (quadrupole, EI) calcd for C₃₈H₂₆N₄O₂ [M]⁺ 570.2056, found 570.2050.

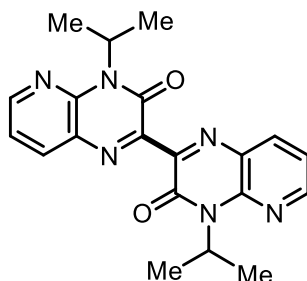
4,4'-Dimethyl-7,7'-di(thiophen-2-yl)-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2t)



35.2 mg (73%); red solid; mp = > 350 °C; ¹H NMR (700 MHz, CDCl₃) δ 8.27 (d, *J* = 2.1 Hz, 2H), 7.90 (dd, *J* = 9.1, 2.1 Hz, 2H), 7.41 (d, *J* = 9.1 Hz, 2H), 7.39 (dd, *J* = 3.5, 0.7 Hz, 2H),

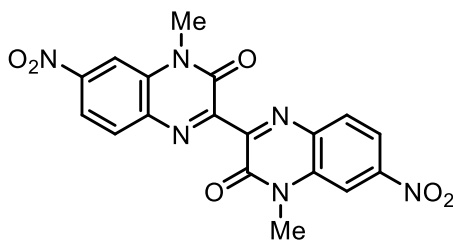
7.33 (dd, $J = 5.6, 1.4$ Hz, 2H), 7.12 (dd, $J = 4.9, 3.5$ Hz, 2H), 3.79 (s, 6H); ^{13}C NMR (175 MHz, CDCl_3) δ 155.2, 154.0, 142.6, 133.4, 133.3, 130.8, 129.4, 128.5, 127.7, 125.6, 123.8, 114.5, 29.4; IR (KBr) ν 3088, 3067, 1647, 1550, 1491, 1418, 1362, 1270, 1223, 1607 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{26}\text{H}_{18}\text{N}_4\text{O}_2\text{S}_2$ $[\text{M}]^+$ 482.0871, found 482.0871.

4,4'-Diisopropyl-[2,2'-bipyrido[2,3-*b*]pyrazine]-3,3'(4*H*,4'*H*)-dione (2u)



5.6 mg (15%); brown solid; mp = 221.1–223.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.62 (dd, $J = 4.2, 1.4$ Hz, 2H), 8.23 (dd, $J = 7.7, 2.1$ Hz, 2H), 7.33 (dd, $J = 7.7, 3.5$ Hz, 2H), 6.00–5.89 (m, 2H), 1.69 (s, 6H), 1.68 (s, 6H); ^{13}C NMR (175 MHz, CDCl_3) δ 156.7, 155.4, 150.2, 144.7, 138.5, 128.6, 119.7, 47.0, 19.4; IR (KBr) ν 3058, 3006, 2973, 1653, 1585, 1551, 1440, 1364, 1271, 1223, 1139, 1098, 1028 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{20}\text{H}_{20}\text{N}_6\text{O}_2$ $[\text{M}]^+$ 376.1648, found 376.1646.

4,4'-Dimethyl-6,6'-dinitro-[2,2'-biquinoxaline]-3,3'(4*H*,4'*H*)-dione (2v)



8.2 mg (20%); yellow solid; mp = > 350 °C; ^1H NMR (700 MHz, CDCl_3) δ 8.31 (d, $J = 2.8$ Hz, 2H), 8.25 (dd, $J = 8.4, 2.1$ Hz, 2H), 8.15 (d, $J = 9.1$ Hz, 2H), 3.85 (s, 6H); ^{13}C NMR (175 MHz, CDCl_3) δ 157.3, 153.5, 149.1, 136.1, 134.6, 132.5, 118.7, 110.0, 29.8; IR (KBr) ν 3087, 2920, 2850, 1655, 1593, 1527, 1463, 1345, 1318, 1241, 1105, 1061 cm^{-1} ; HRMS (quadrupole, FAB) calcd for $\text{C}_{18}\text{H}_{13}\text{N}_6\text{O}_6$ $[\text{M}+\text{H}]^+$ 409.0897, found 409.0897.

X-ray crystal structure determination

The X-ray diffraction data of **2e** were collected on a Bruker APEX-II diffractometer equipped with a monochromator in the Mo K α ($\lambda = 0.71073 \text{ \AA}$) incident beam. Each crystal was mounted on a glass fiber. The frames were integrated and scaled using the Bruker-S SAINT software package, and the structure was solved and refined using the Bruker SHELXTL software package. All hydrogen atoms were placed in the calculated positions. The crystallographic data for the three compounds are listed in Table S1. Structural information was deposited at the Cambridge Crystallographic Data Centre. The CCDC reference number is 2143525 for **2e**.

Table S2. Crystallographic data for compound **2e**.

Identification code	2143525	
Chemical formula	C ₁₈ H ₁₂ Cl ₂ N ₄ O ₂	
Formula weight	387.22 g/mol	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	0.040 x 0.070 x 0.080 mm	
Space group	P -1	
Unit cell dimensions	a = 7.4478(5) Å	α = 74.939(2)°
	b = 9.0425(6) Å	β = 86.769(2)°
	c = 12.7861(8) Å	γ = 84.018(2)°
Volume	826.61(9) Å ³	
Z	2	
Density (calculated)	1.556 g/cm ³	
Absorption coefficient	0.415 mm ⁻¹	
F(000)	396	
Crystal size	0.040 x 0.070 x 0.080 mm	
Theta range for data collection	1.65 to 28.28°	
Index ranges	-9 ≤ h ≤ 9, -12 ≤ k ≤ 12, -17 ≤ l ≤ 17	
Reflections collected	34242	
Independent reflections	4076 [R(int) = 0.0663]	
Coverage of independent reflections	99.8%	
Absorption correction	Multi-Scan	
Max. and min. transmission	0.9840 and 0.9680	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4076 / 0 / 238	
Goodness-of-fit on F ²	1.033	
Final R indices [I > 2σ(I)]	R ₁ = 0.0495, wR ₂ = 0.1238	
Final R indices (all data)	R ₁ = 0.1231, wR ₂ = 0.1801	
Largest diff. peak and hole	0.907 and -1.095 e.Å ⁻³	

X-ray crystallographic data of compound **2e** (CCDC 2143525)

Sample preparation (vapor diffusion): compound **2e** (10.1 mg) was dissolved with 1.5 mL of CHCl₃ and 0.5 mL of EtOAc in opened inner vessel, and *n*-hexane (6 mL) as an anti-solvent has been employed in closed outer vessel. After vapor diffusion for 14 days, the single crystals of compound **2e** were obtained.

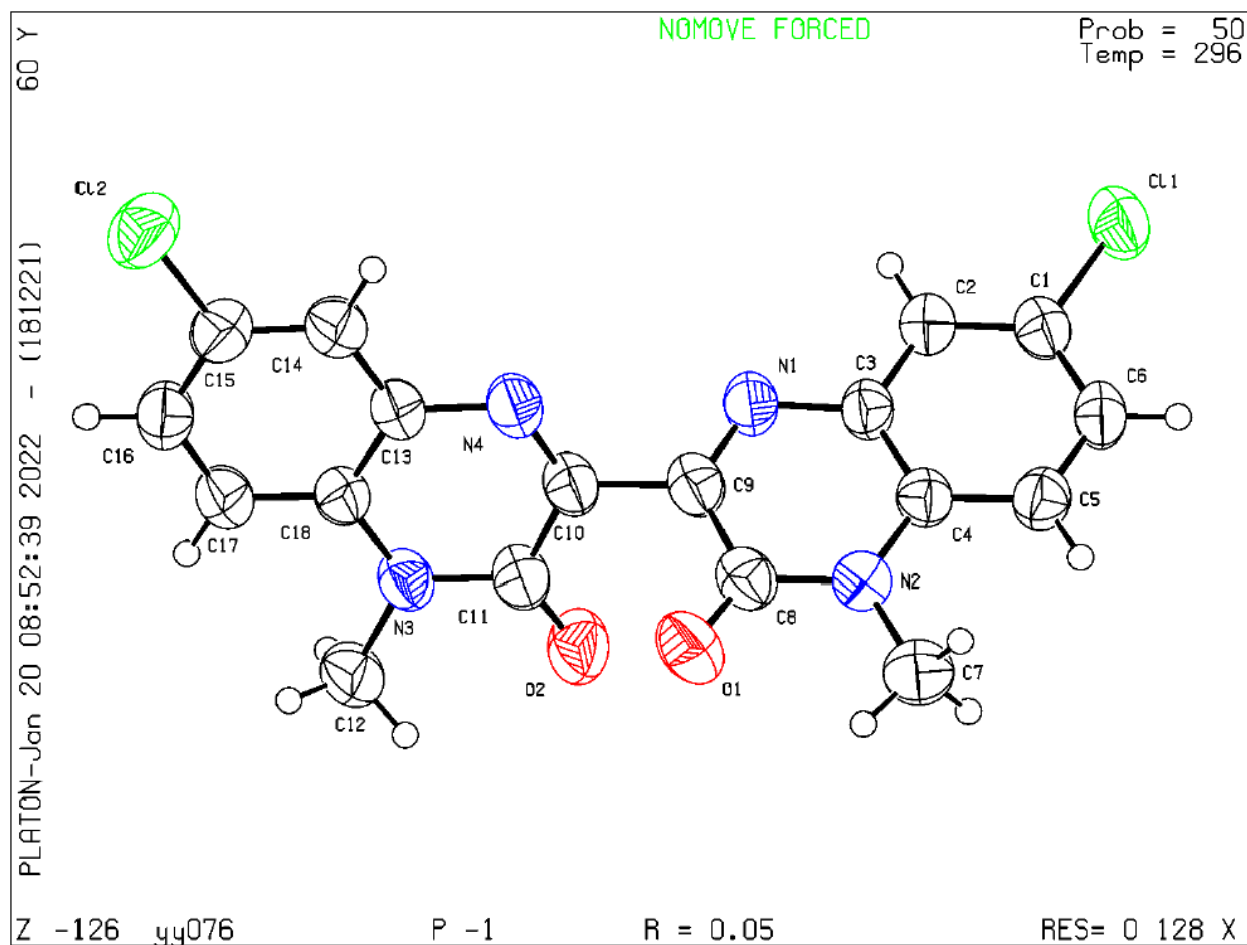
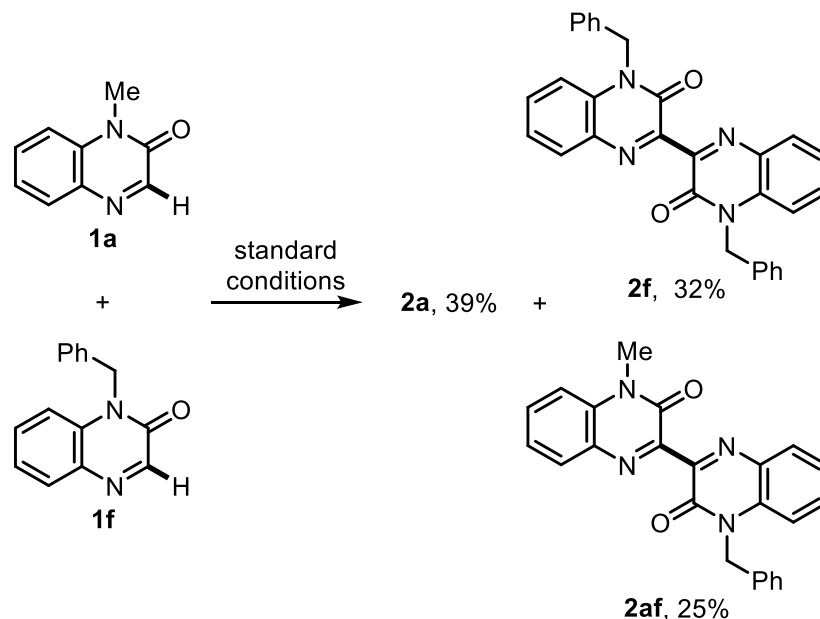


Figure S5. ORTEP diagram of compound **2e** (CCDC 2143525).

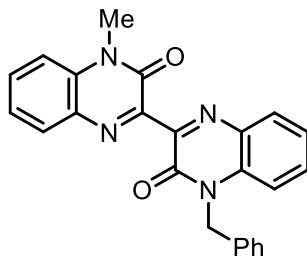
Experimental procedures for the competition experiments

Competition reaction between quinoxalinones **1a** and **1f**



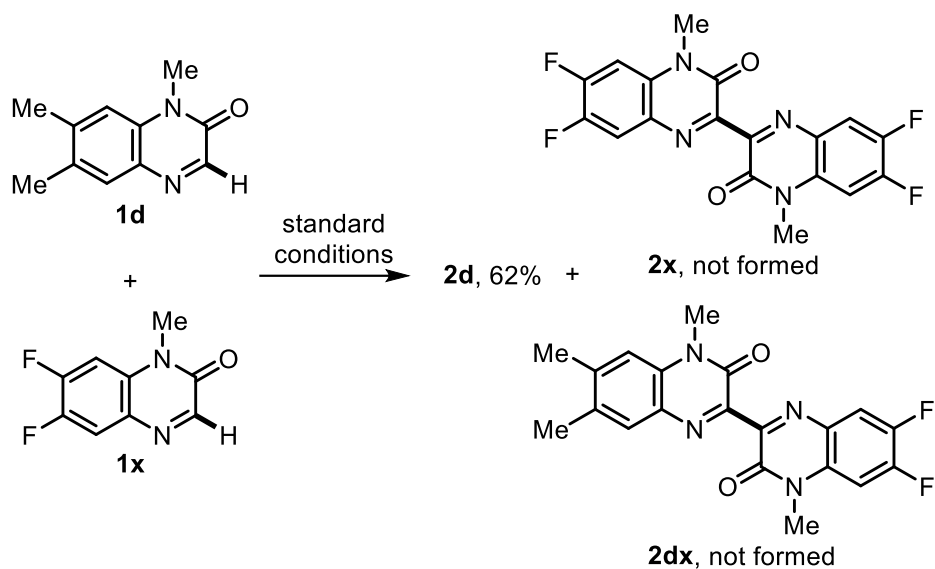
To an oven-dried sealed tube charged with 1-methylquinoxalin-2(1H)-one (**1a**) (16.1 mg, 0.1 mmol, 1.0 equiv.), 1-benzylquinoxalin-2(1H)-one (**1f**) (23.6 mg, 0.1 mmol, 1.0 equiv.), and KO^tBu (22.4 mg, 0.2 mmol, 2.0 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography to afford **2f** (9.7 mg, 32%, *n*-hexanes/EtOAc = 1:1) and cross-over product **2af** (9.9 mg, 25%, *n*-hexanes/EtOAc = 1:1), and **2a** (6.2 mg, 39%, CH₂Cl₂/MeOH = 98:2).

4-Benzyl-4'-methyl-[2,2'-biquinoxaline]-3,3'(4H,4'H)-dione (**2af**)



9.9 mg (25%); off-white solid; mp = 233.6–236.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.04–7.99 (m, 2H), 7.68–7.62 (m, 1H), 7.54–7.48 (m, 1H), 7.41 (t, $J = 7.2$ Hz, 2H), 7.37–7.27 (m, 7H), 5.57 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.8, 154.7, 154.4, 154.3, 135.2, 134.2, 133.4, 133.2, 131.7, 131.6, 131.3, 131.3, 129.1, 129.0, 127.9, 127.2, 124.1, 114.8, 113.9, 47.1, 29.3; IR (KBr) ν 3052, 2923, 2852, 1641, 1600, 1557, 1468, 1455, 1316, 1264, 1160, 1067 cm^{-1} ; HRMS (quadrupole, EI) calcd for $\text{C}_{24}\text{H}_{18}\text{N}_4\text{O}_2$ $[\text{M}]^+$ 394.1430, found 394.1433.

Competition reaction between quinoxalinones **1d** and **1x**



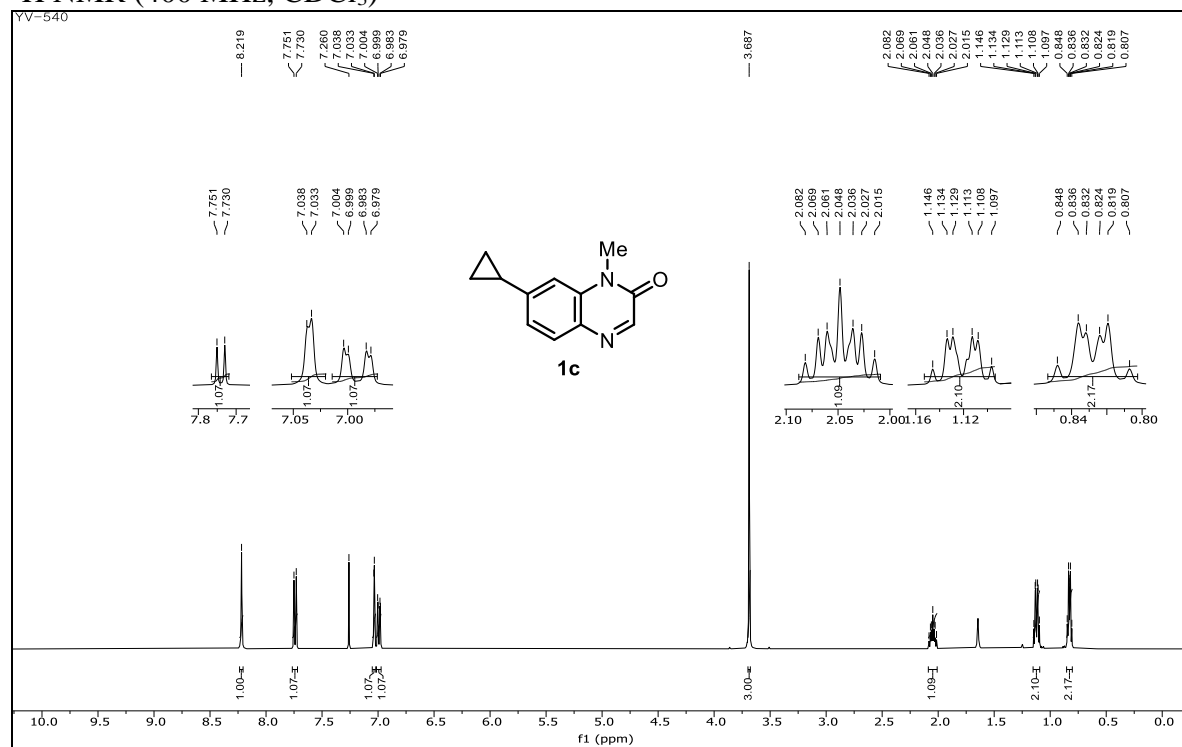
To an oven-dried sealed tube charged with 1,6,7-trimethylquinoxalin-2(1H)-one (**1d**) (18.8 mg, 0.1 mmol, 1.0 equiv.), 6,7-difluoro-1-methylquinoxalin-2(1H)-one (**1x**) (19.6 mg, 0.1 mmol, 1.0 equiv.), and KO^tBu (22.4 mg, 0.2 mmol, 1.0 equiv.) was added dimethoxyethane (1 mL) at room temperature under N₂. The reaction mixture was allowed to stir at 40 °C for 1 h. The reaction mixture was cooled to room temperature, diluted with CH₂Cl₂/MeOH (20 mL, 9:1) and stirred under air for 0.2 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (CH₂Cl₂/MeOH) to afford **2d** (11.6 mg, 62%) only. ¹H NMR spectra of the isolated product **2d** were found identical to those of the standard sample. No formation of dimerized product **2x** or cross-over product **2dx** was detected.

References

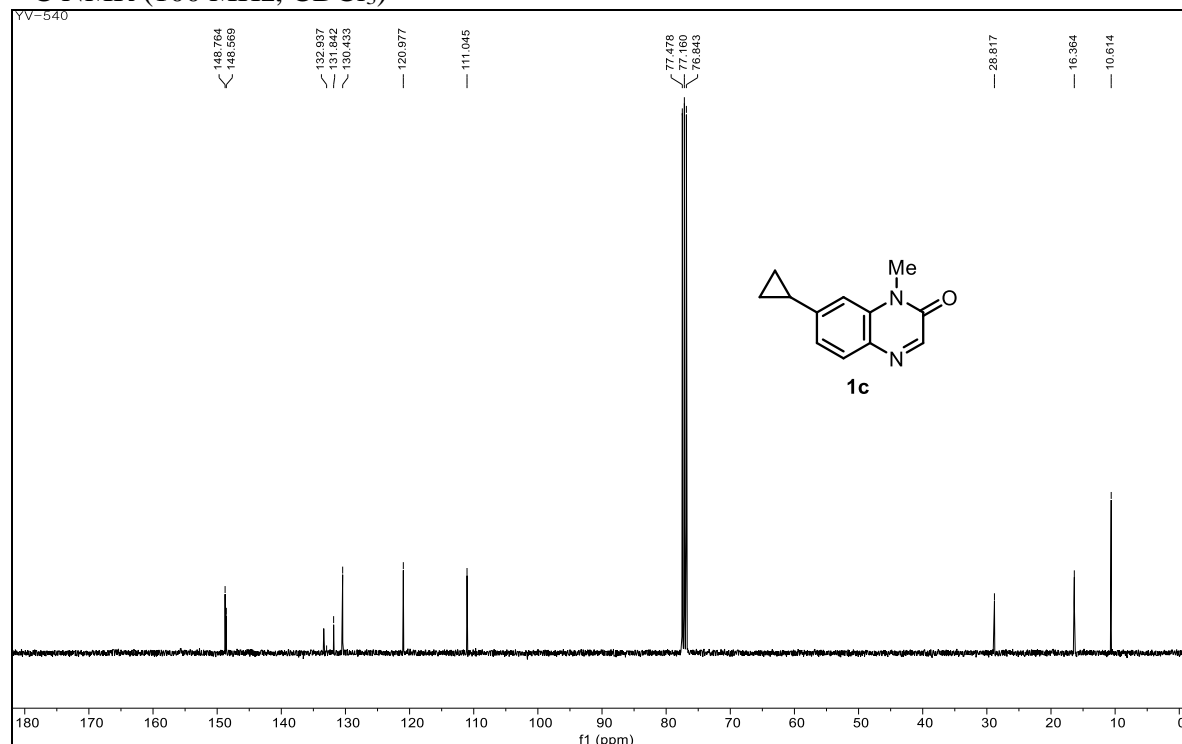
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2. Xie, L.-Y.; Bai, Y.-S.; Xu, X.-Q.; Peng, X.; Tang, H.-S.; Huang, Y.; Lin, Y.-W.; Cao, Z.; He, W.-M. Visible-light-induced decarboxylative acylation of quinoxalin-2(1*H*)-ones with α -oxo carboxylic acids under metal-, strong oxidant- and external photocatalyst-free conditions. *Green Chem.* **2020**, *22*, 1720–1725.
3. Guo, J.; Zhang, L.; Du, X.; Zhang, L.; Cai, Y.; Xia, Q. Metal-Free Direct Oxidative C–N Bond Coupling of Quinoxalin-2(1*H*)-ones with Azoles under Mild Conditions. *Eur. J. Org. Chem.* **2021**, *2021*, 2230–2238.
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¹H NMR and ¹³C NMR spectra of all compounds

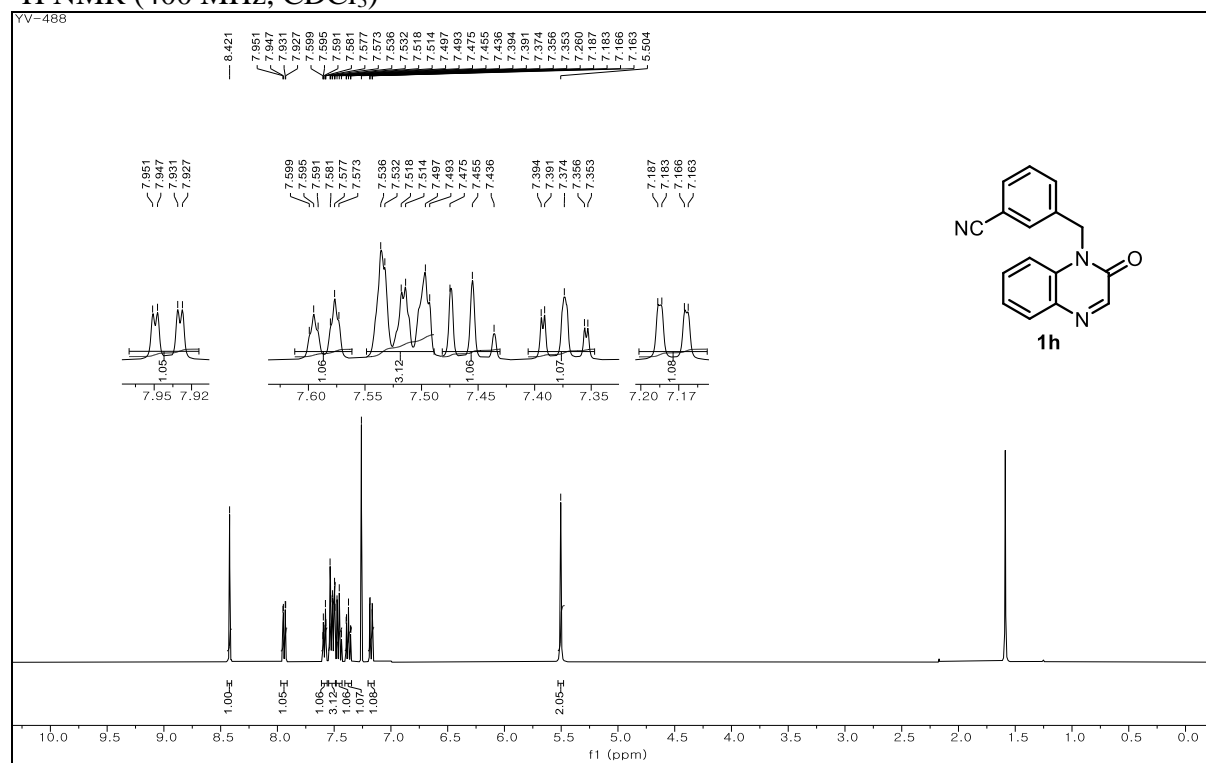
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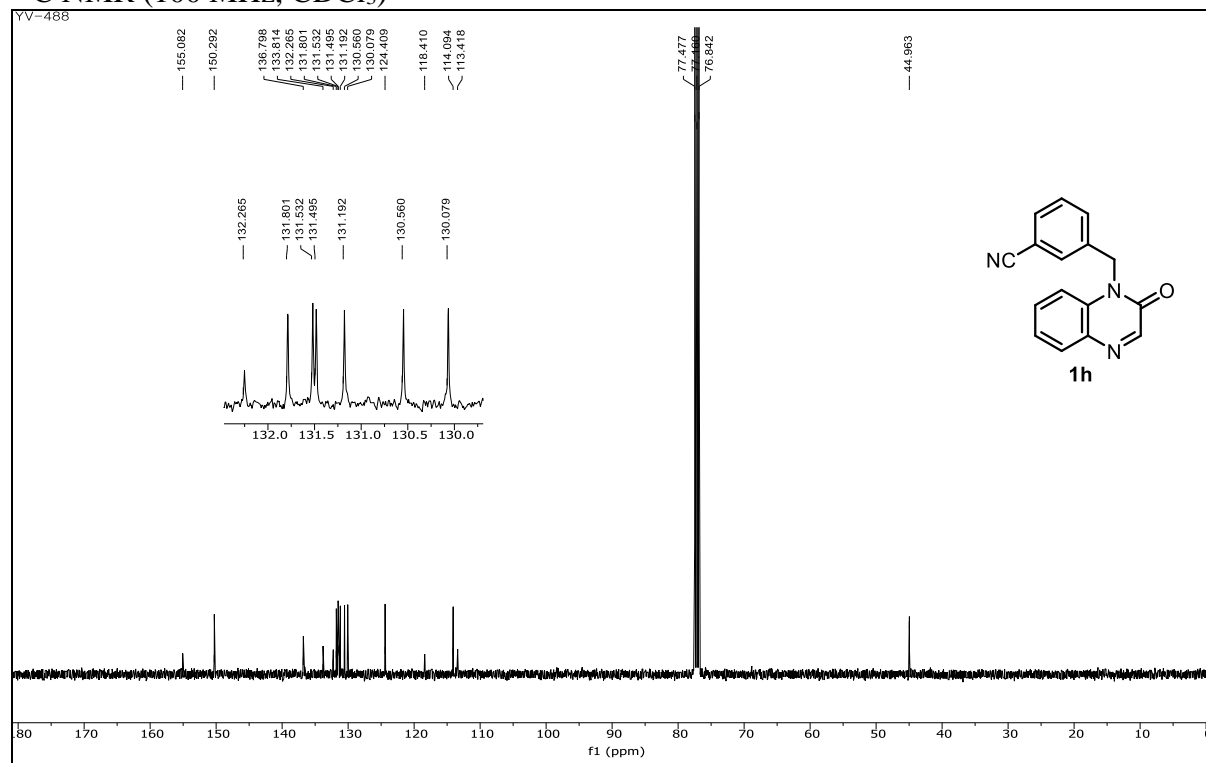
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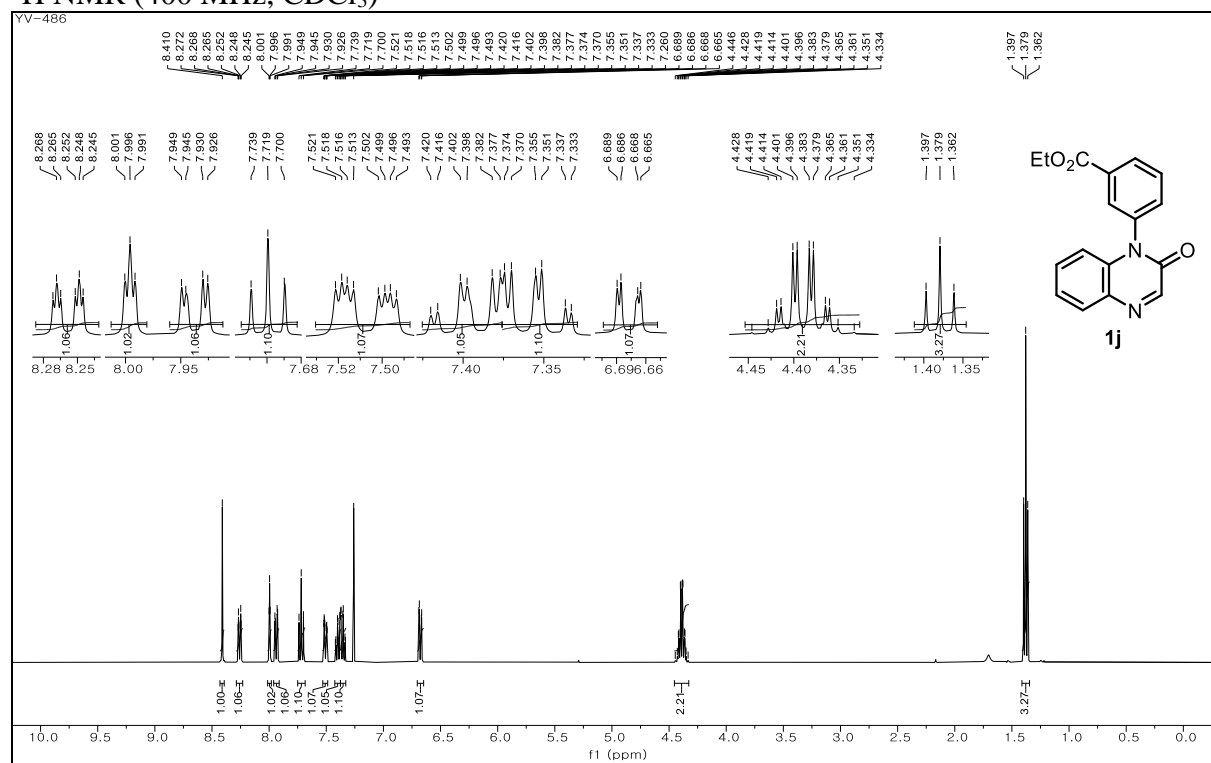
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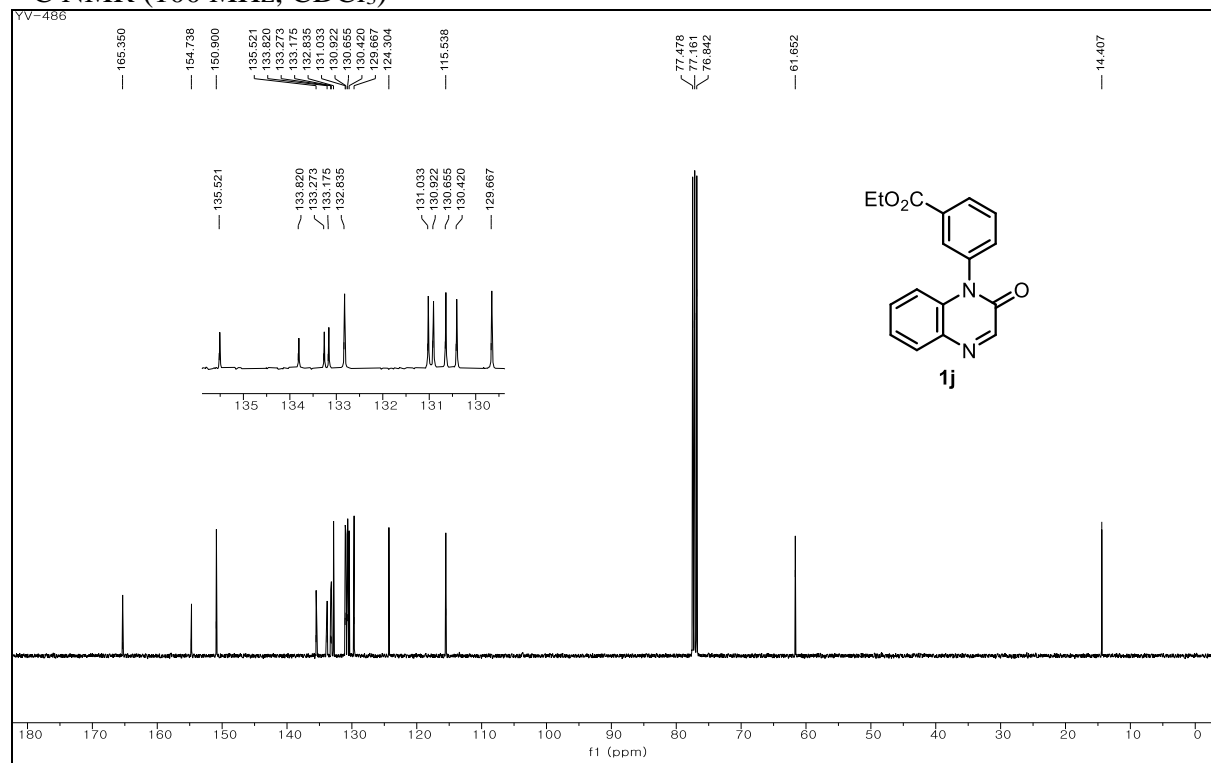
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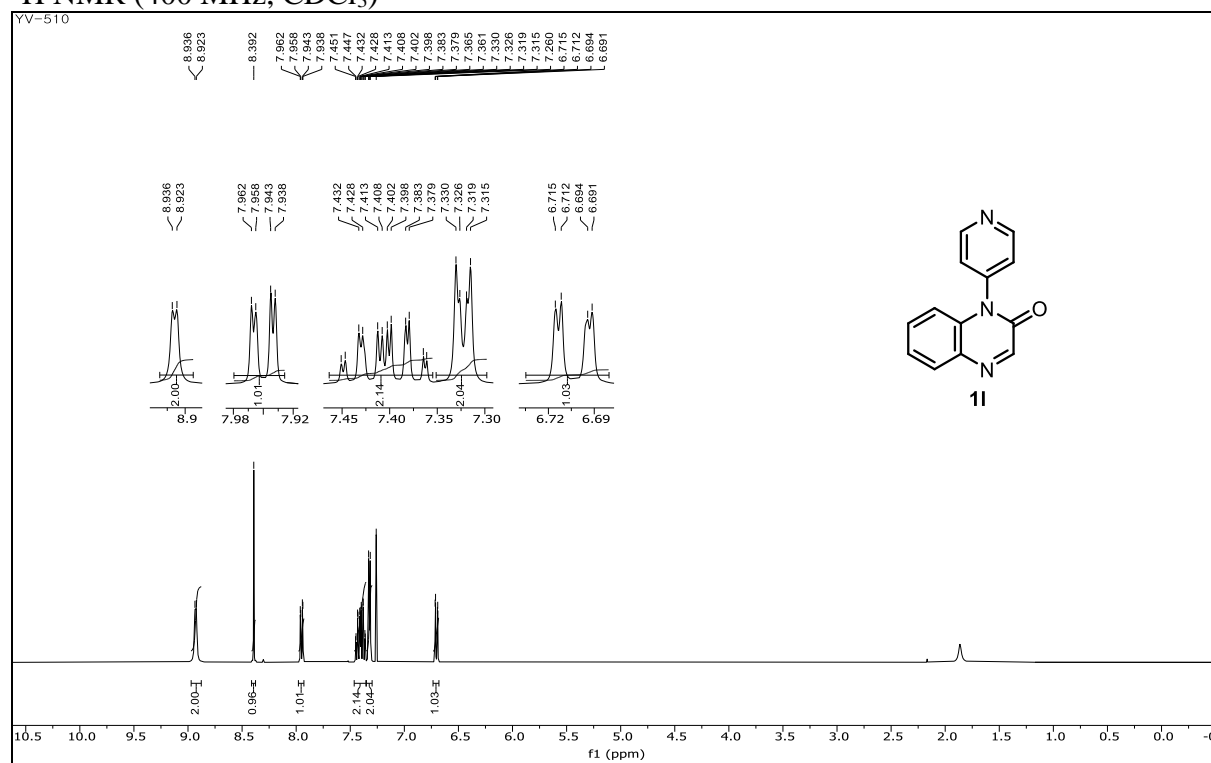
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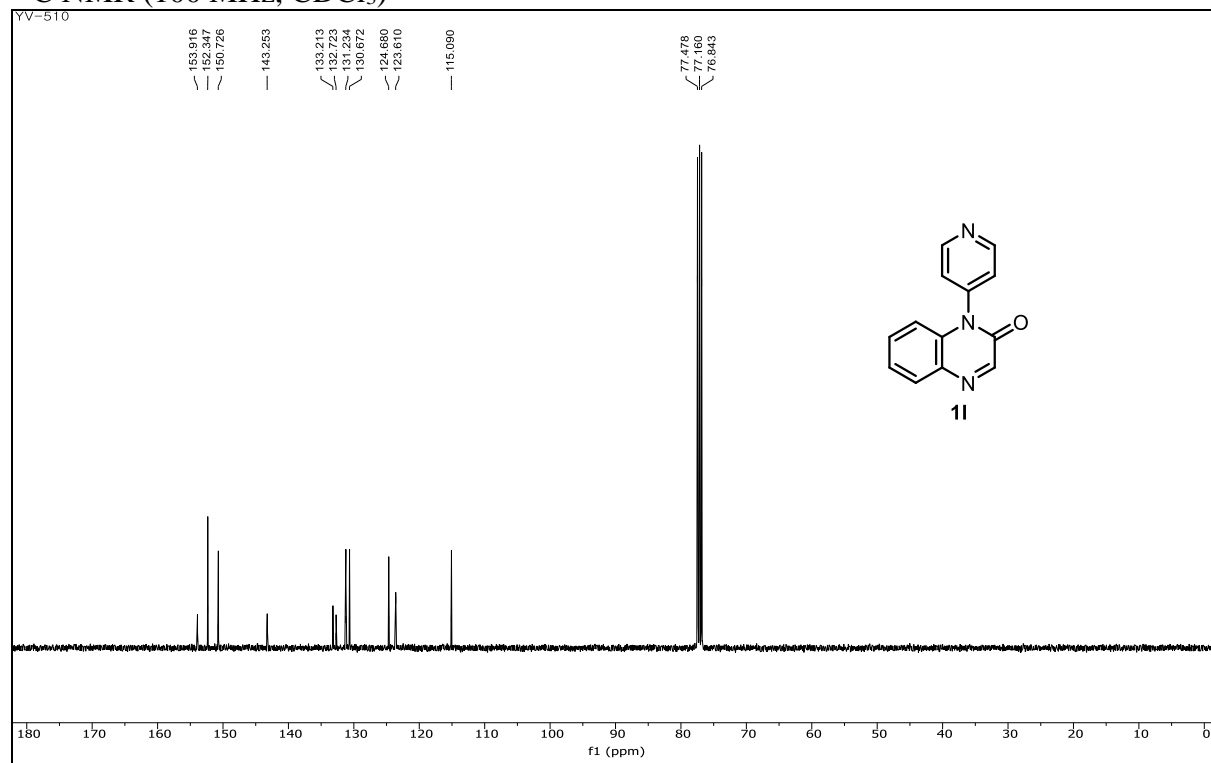
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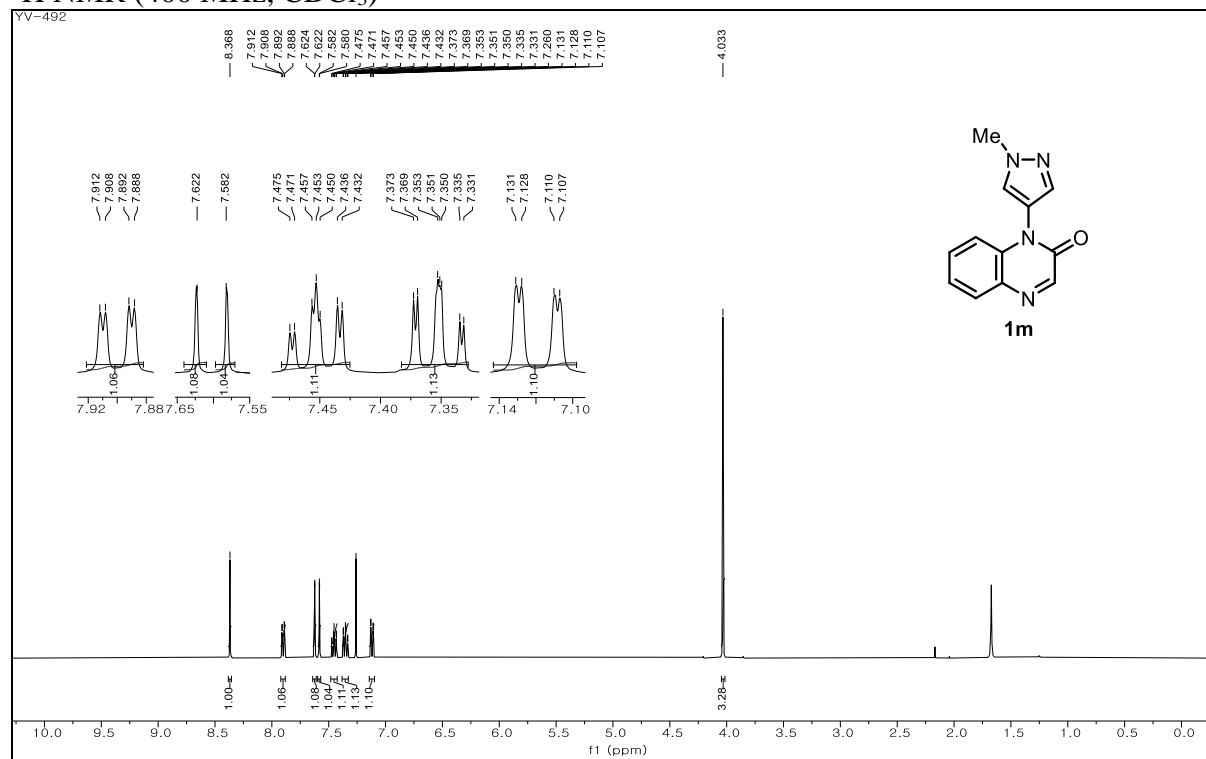
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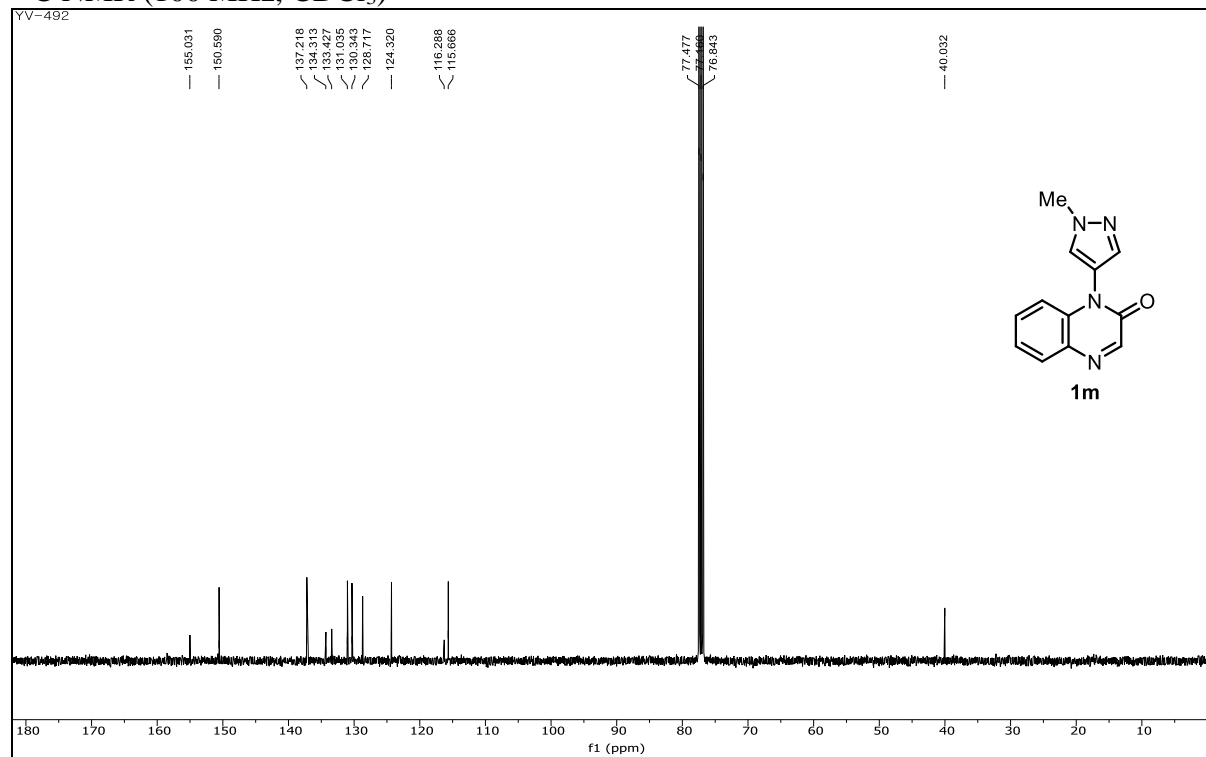
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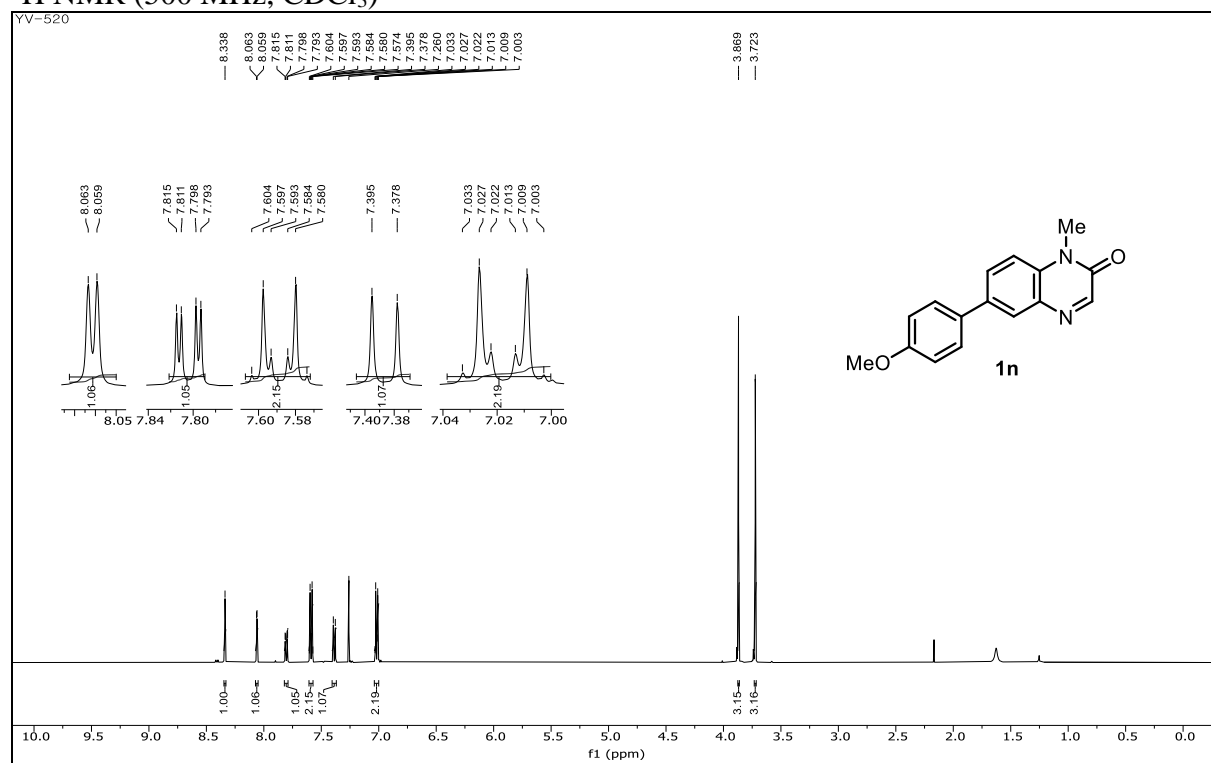
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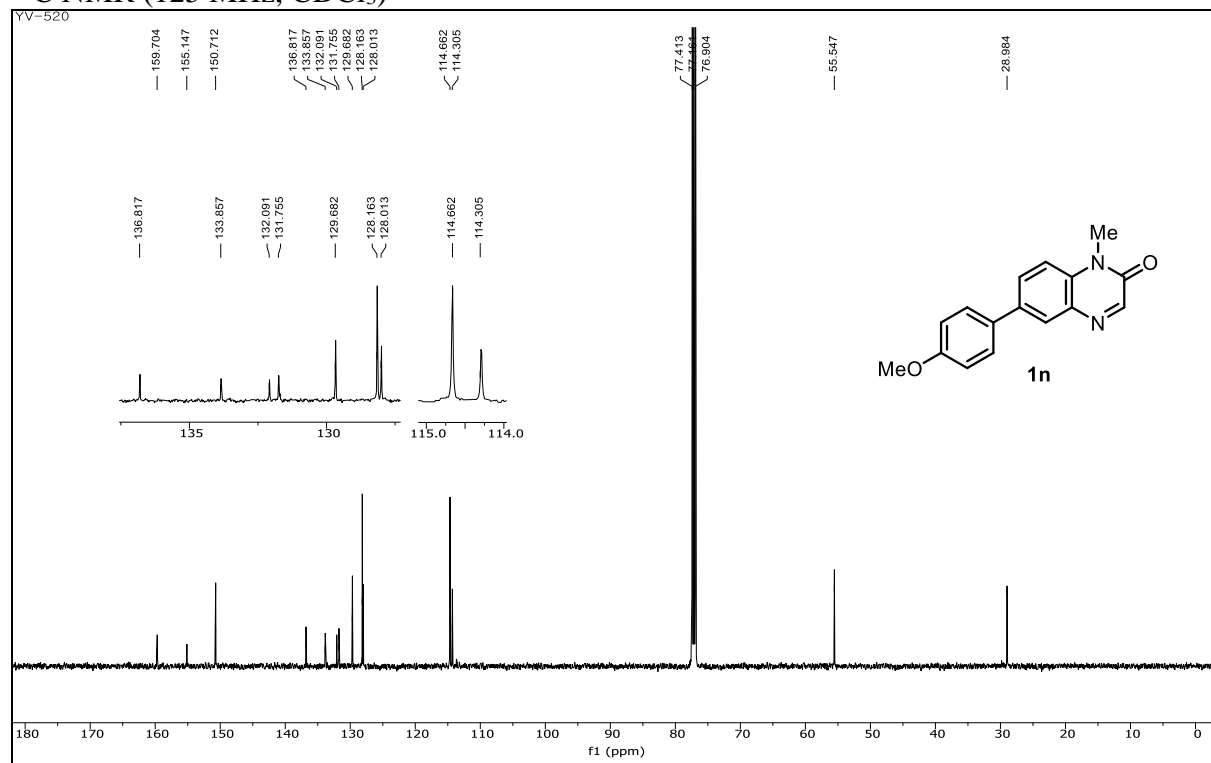
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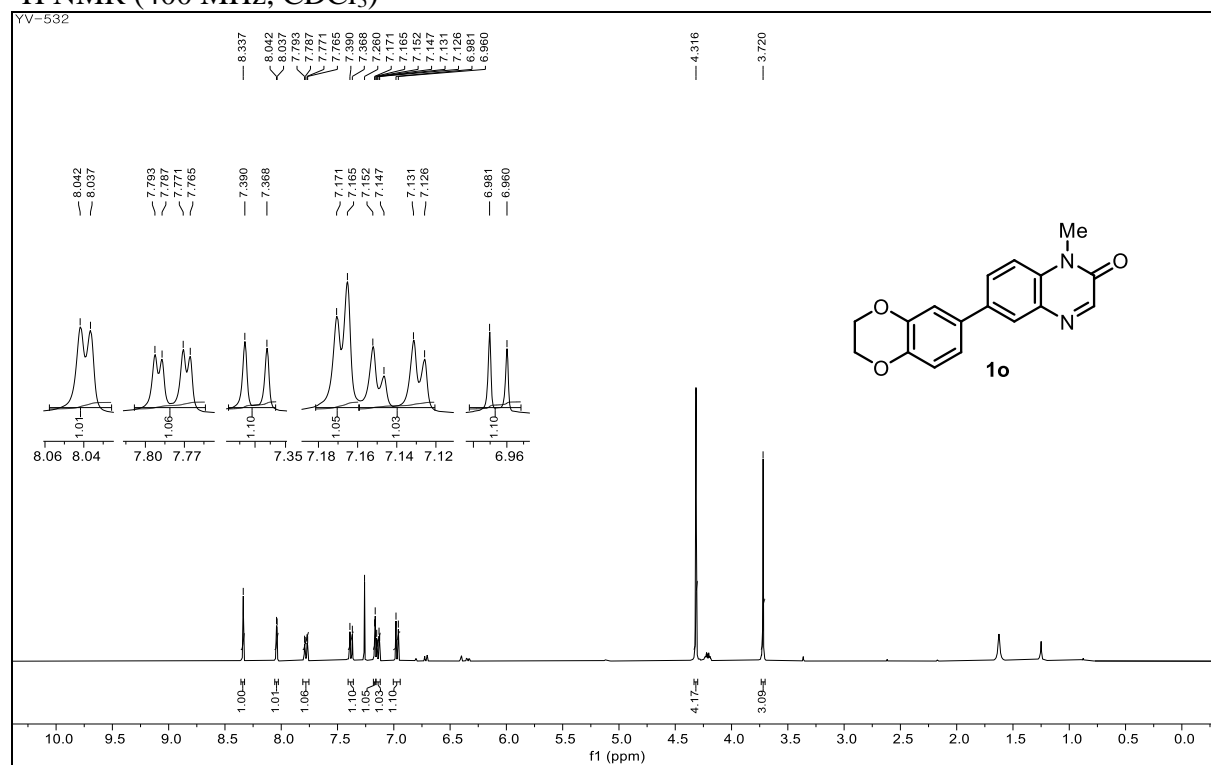
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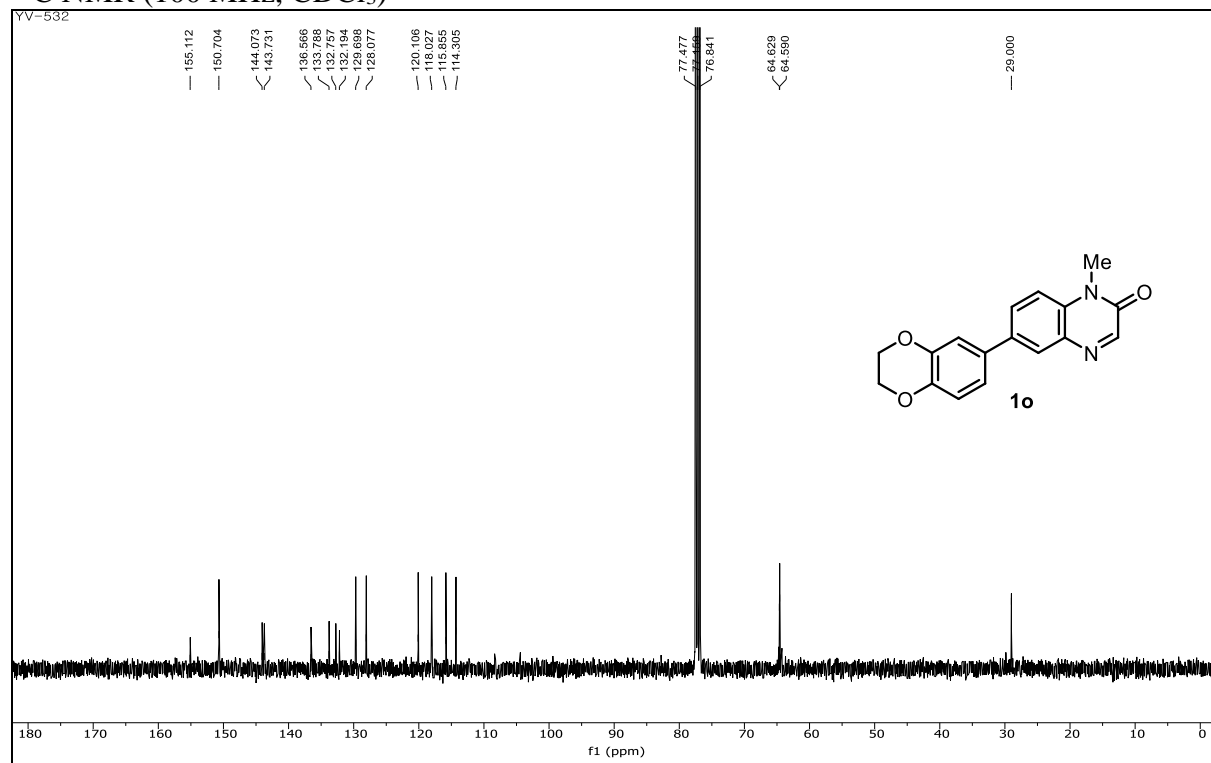
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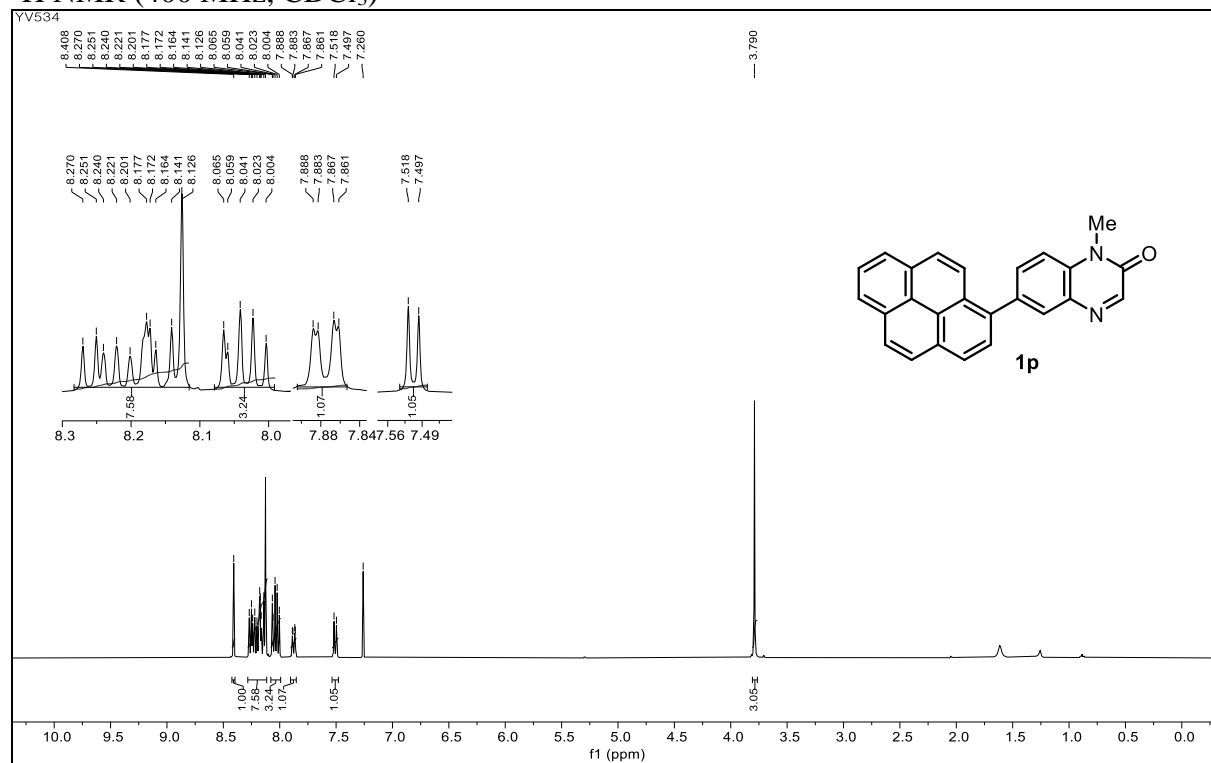
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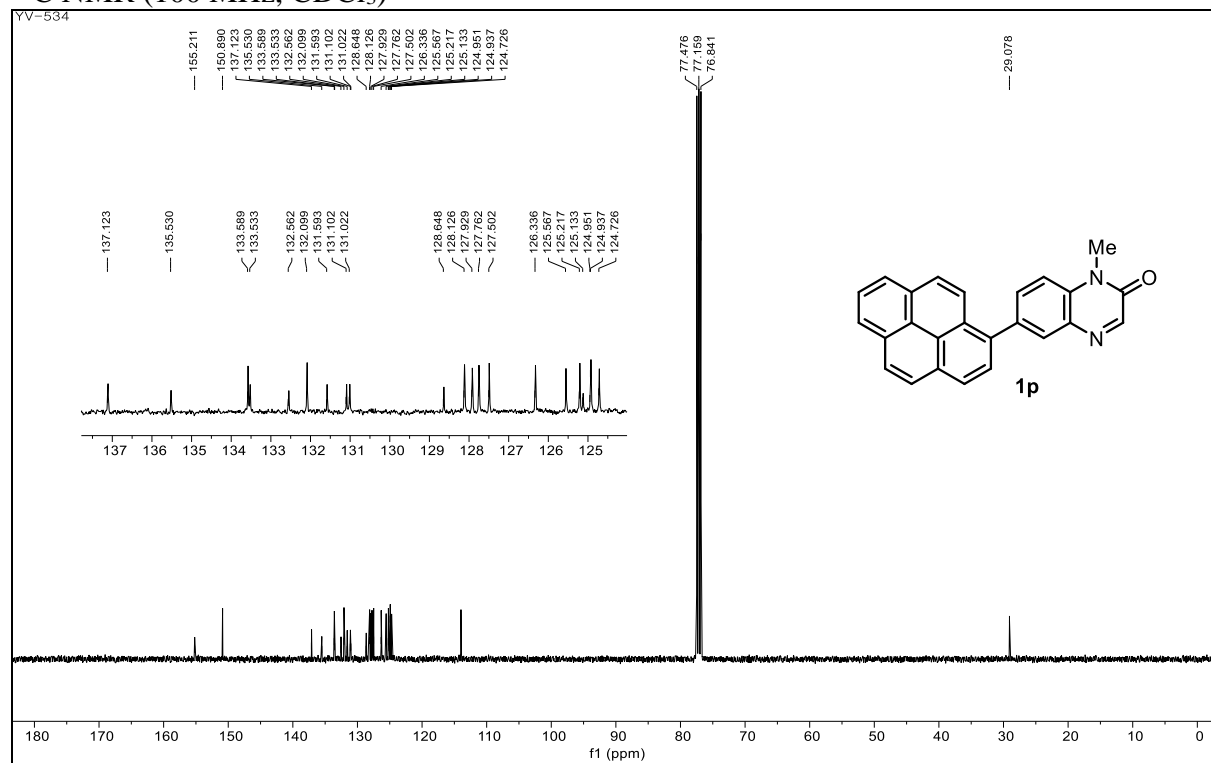
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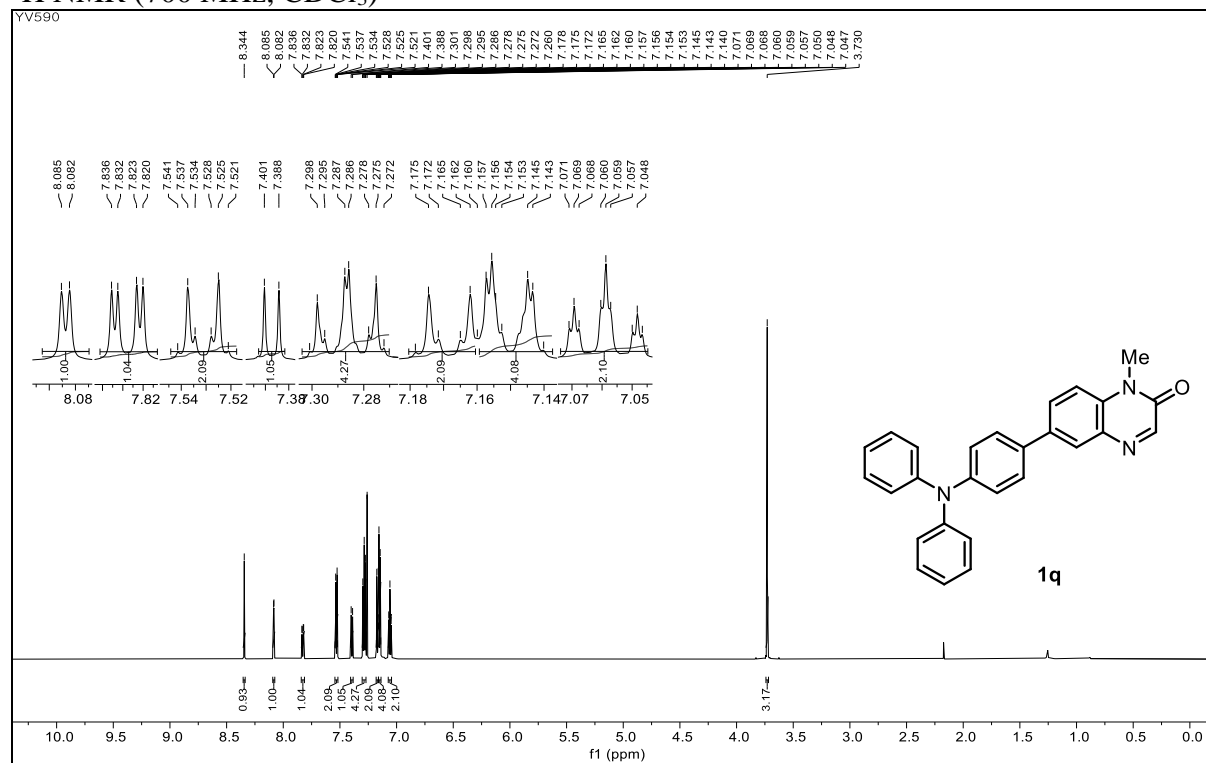
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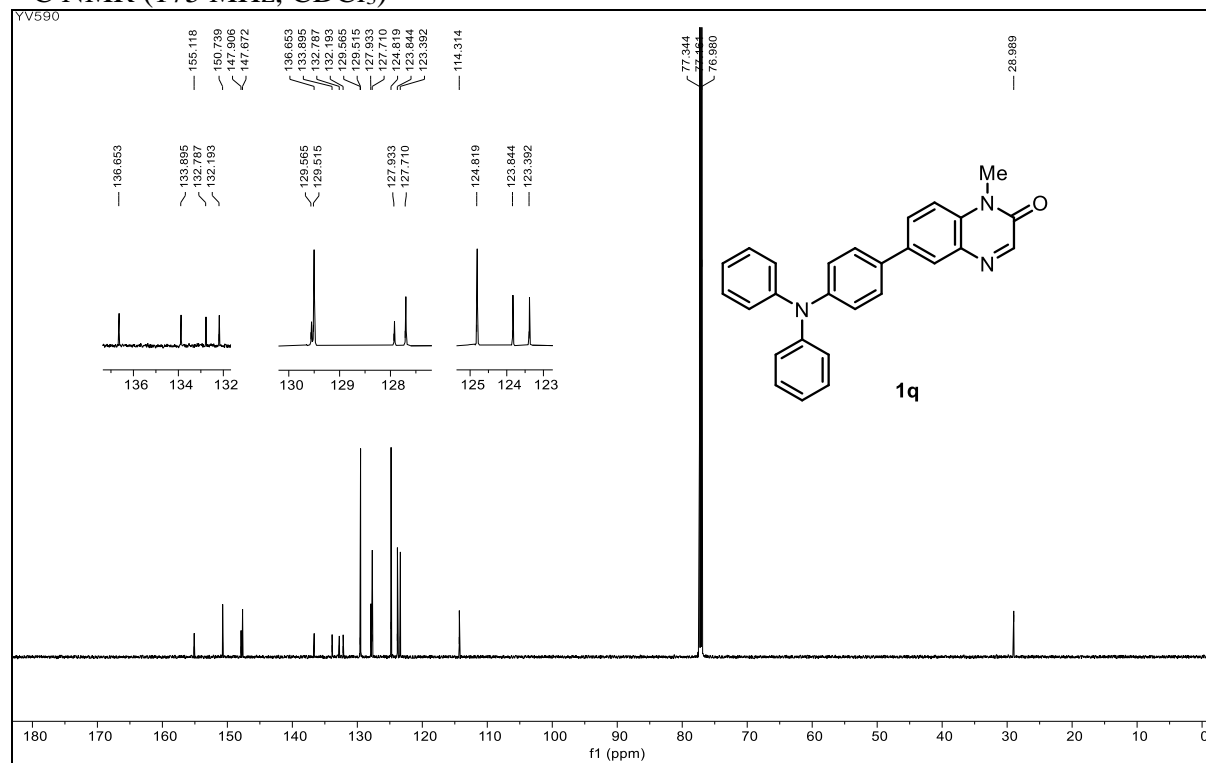
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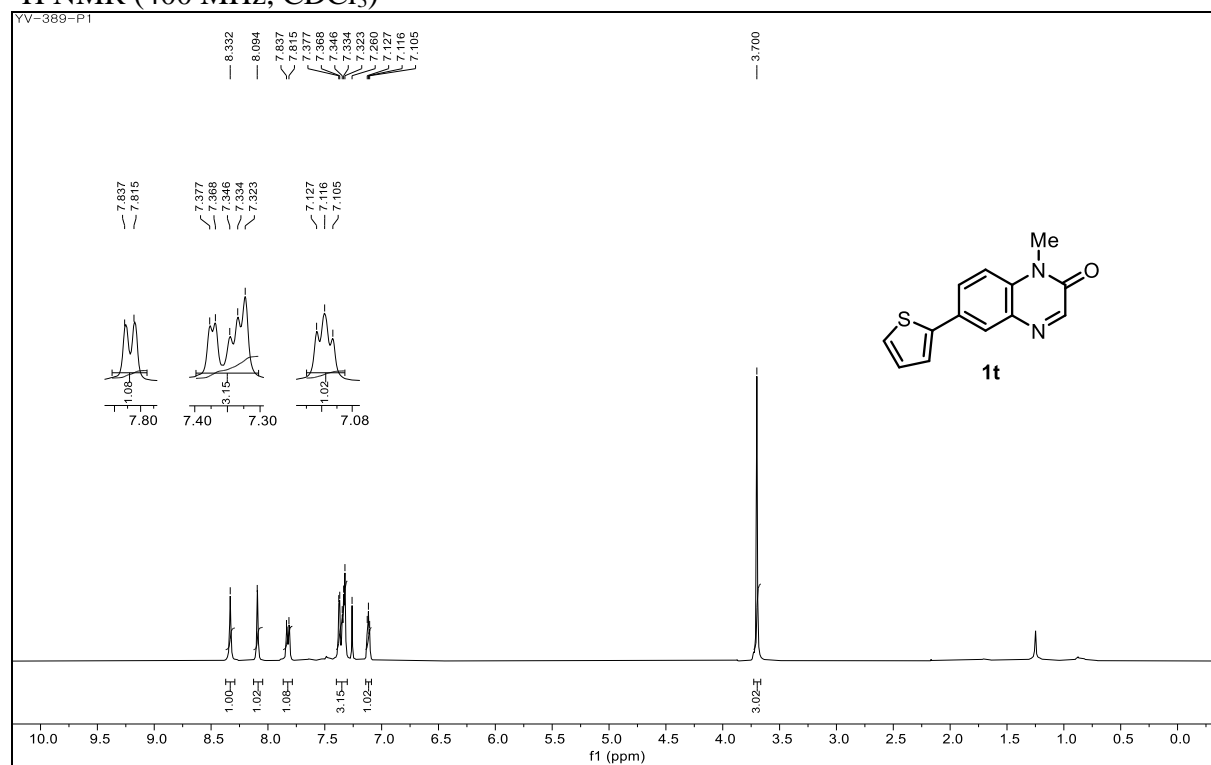
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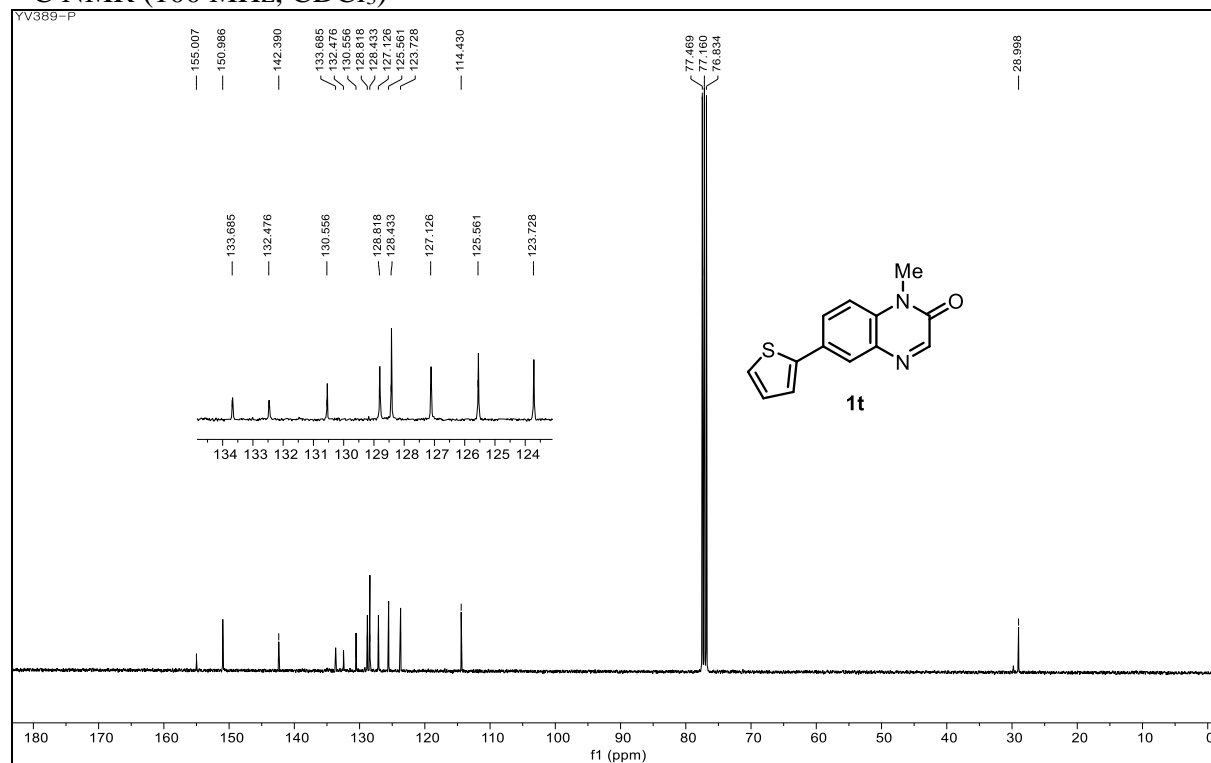
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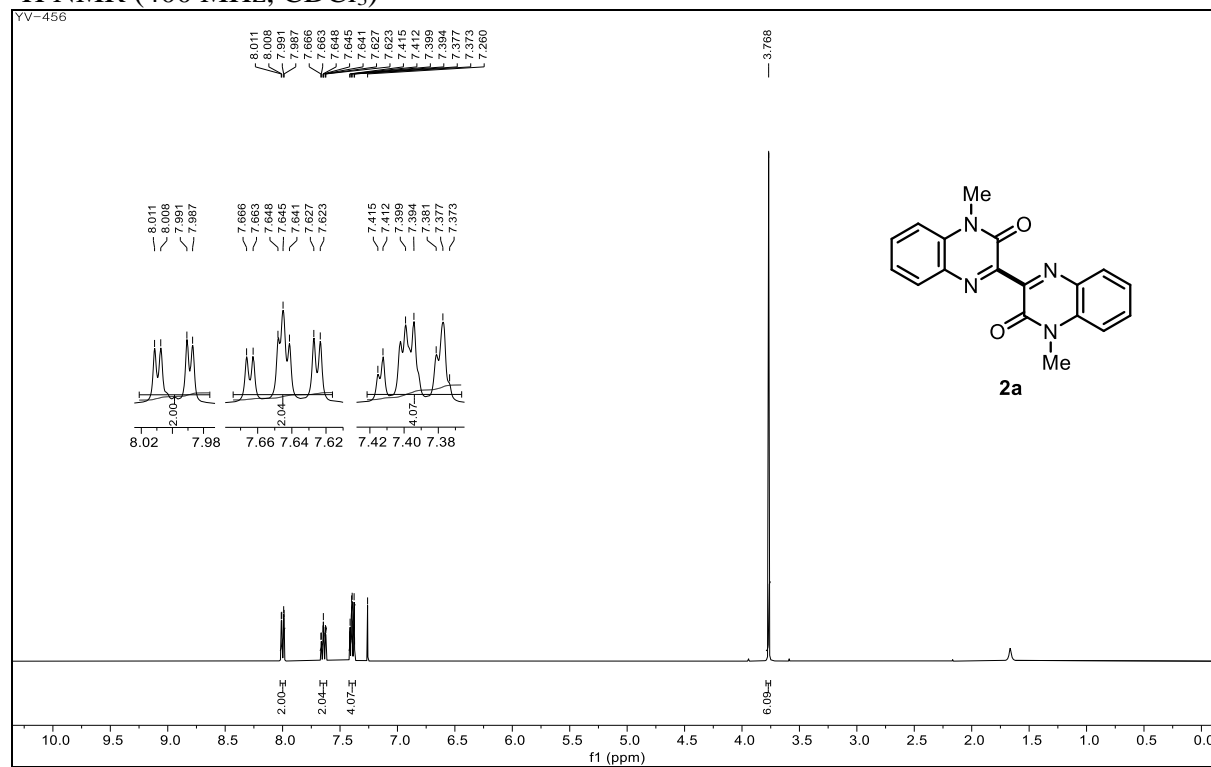
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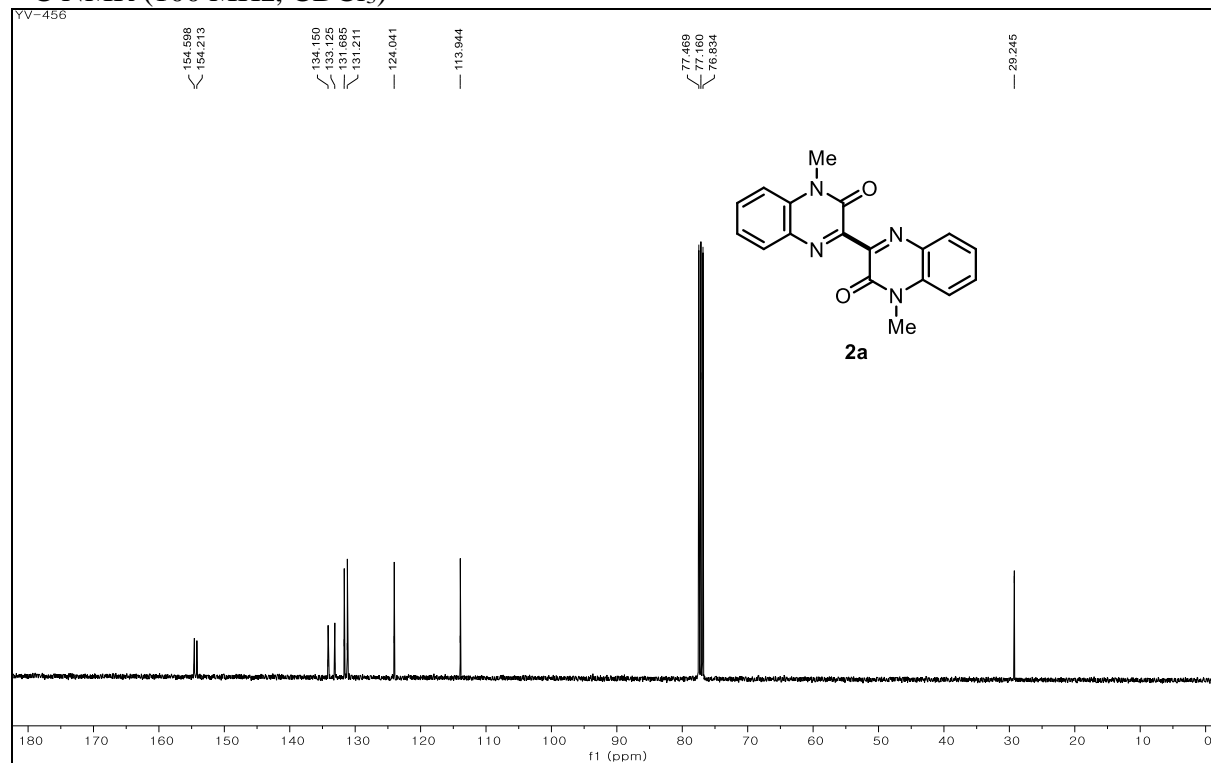
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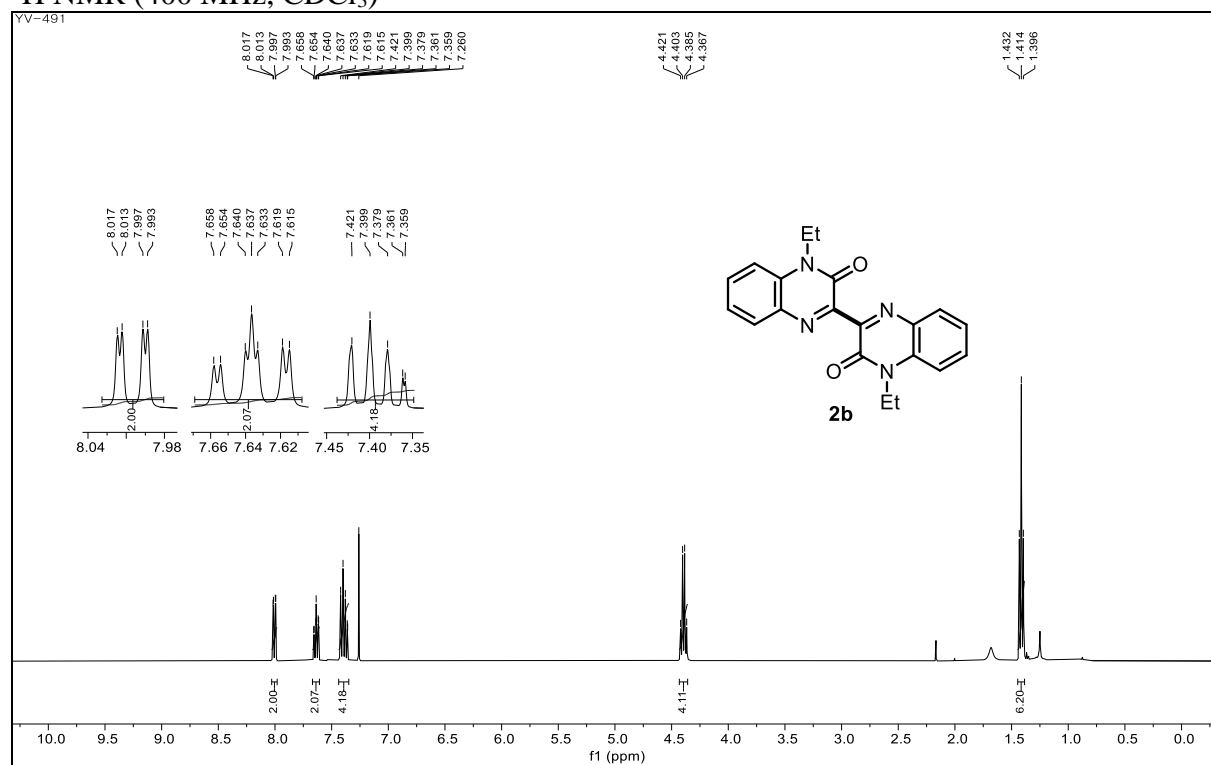
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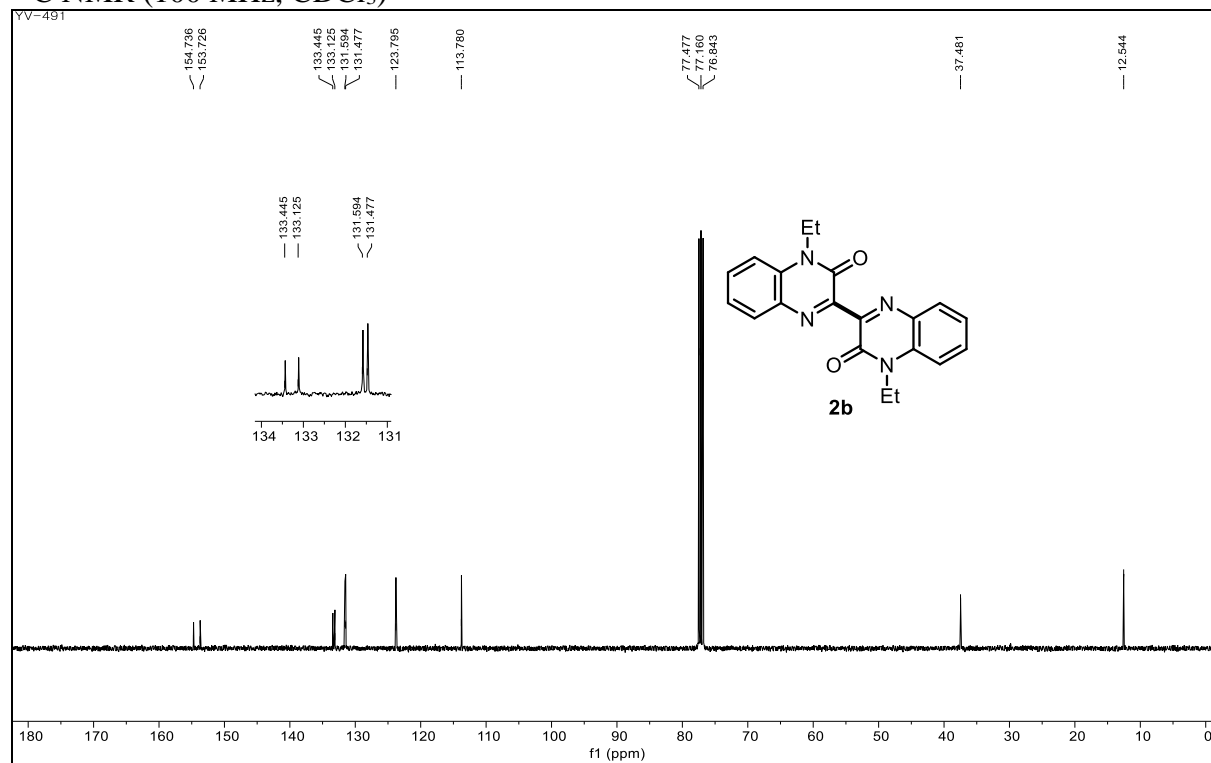
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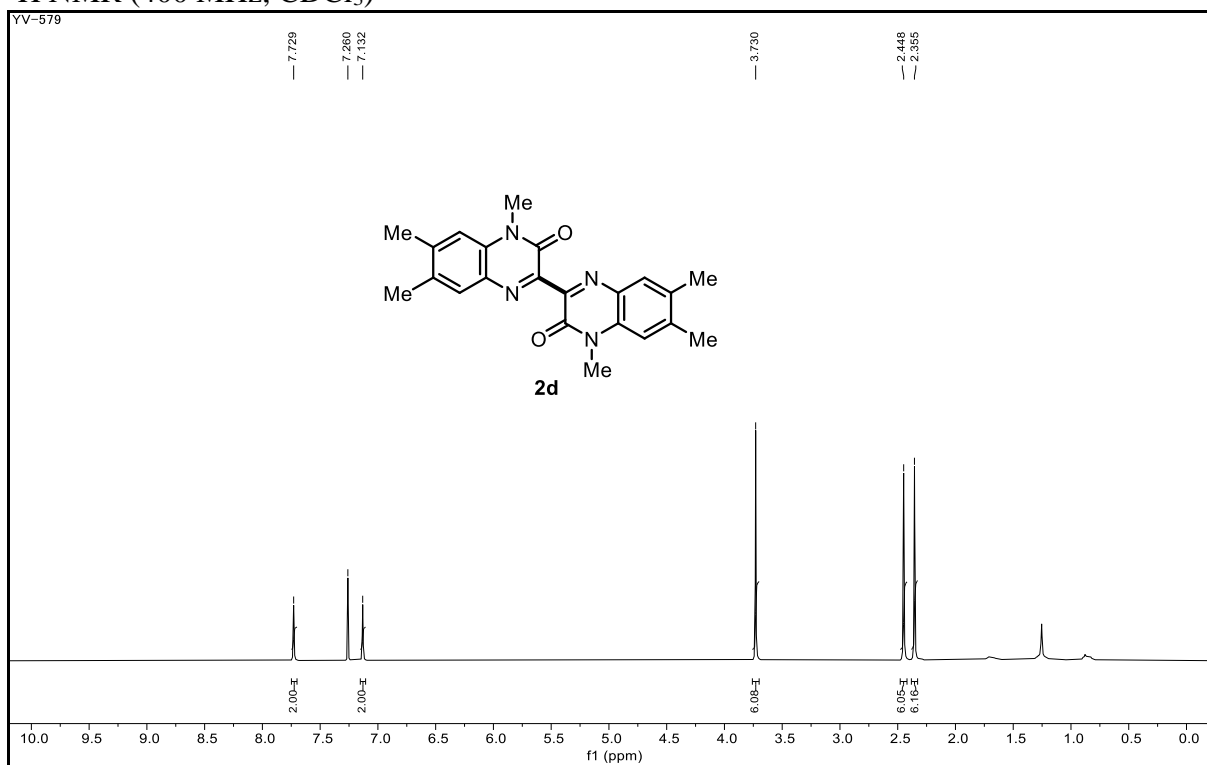
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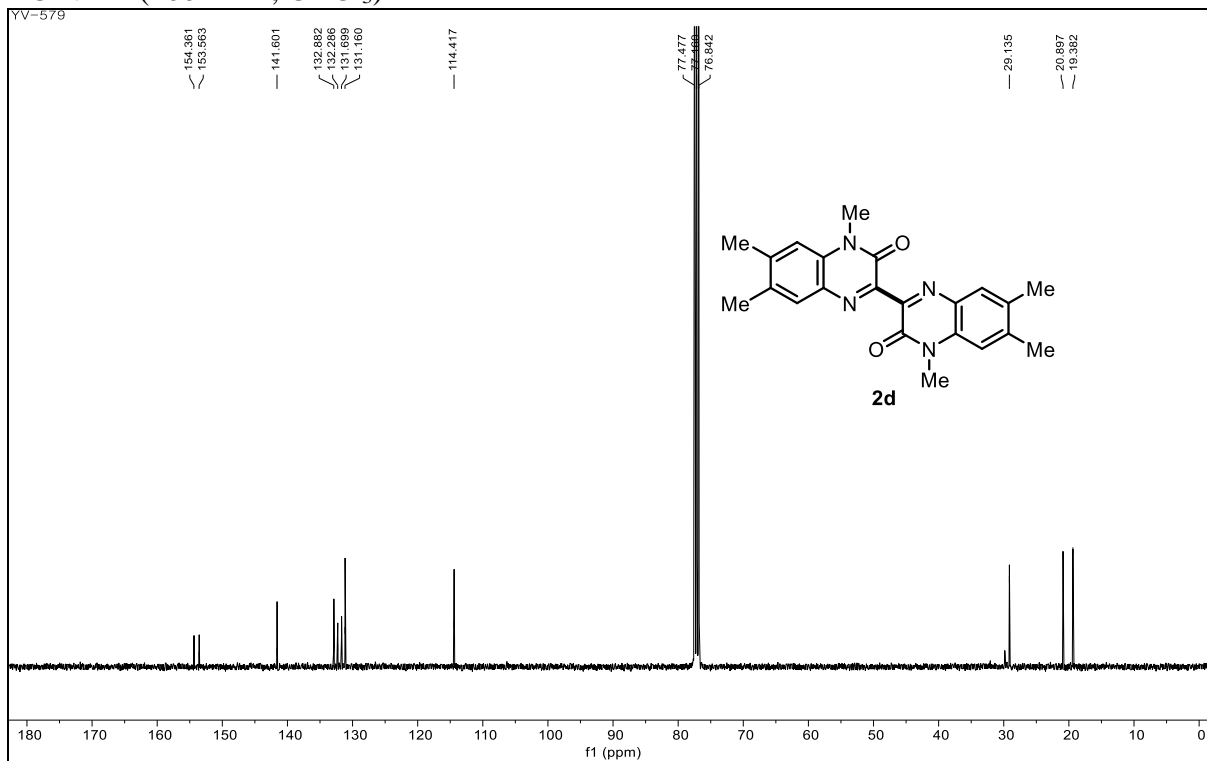
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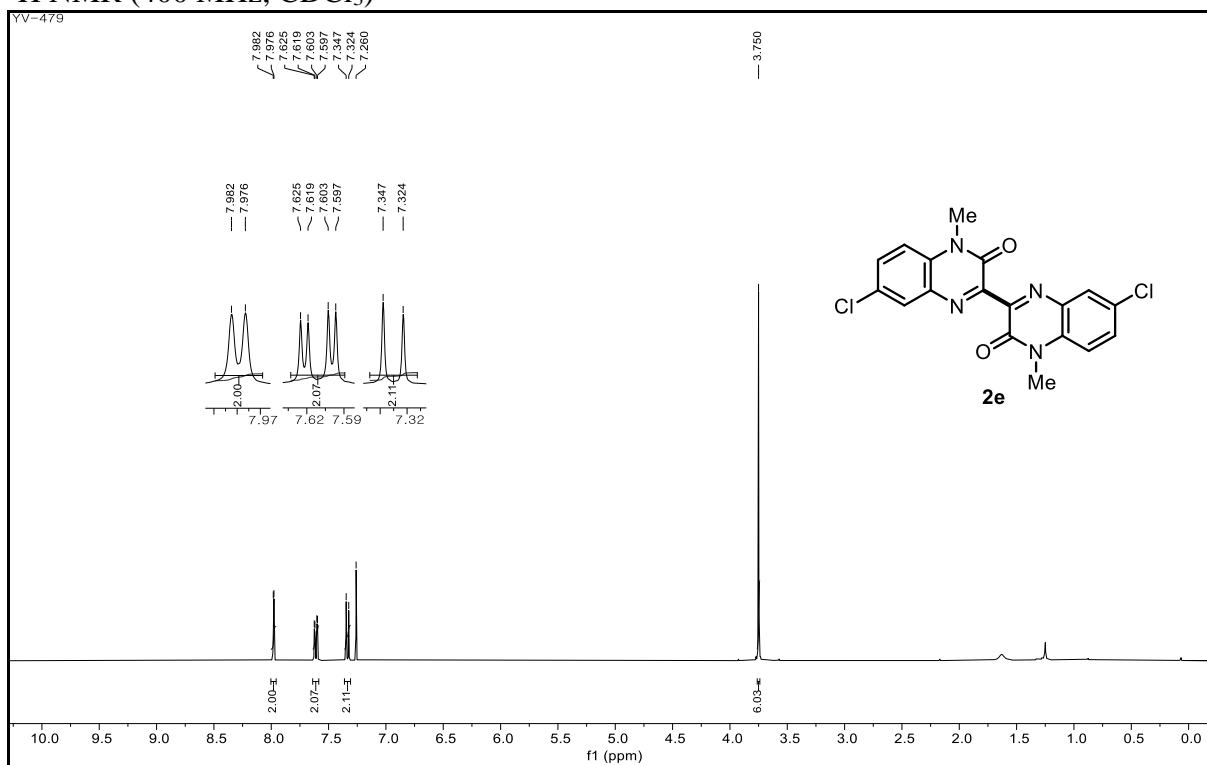
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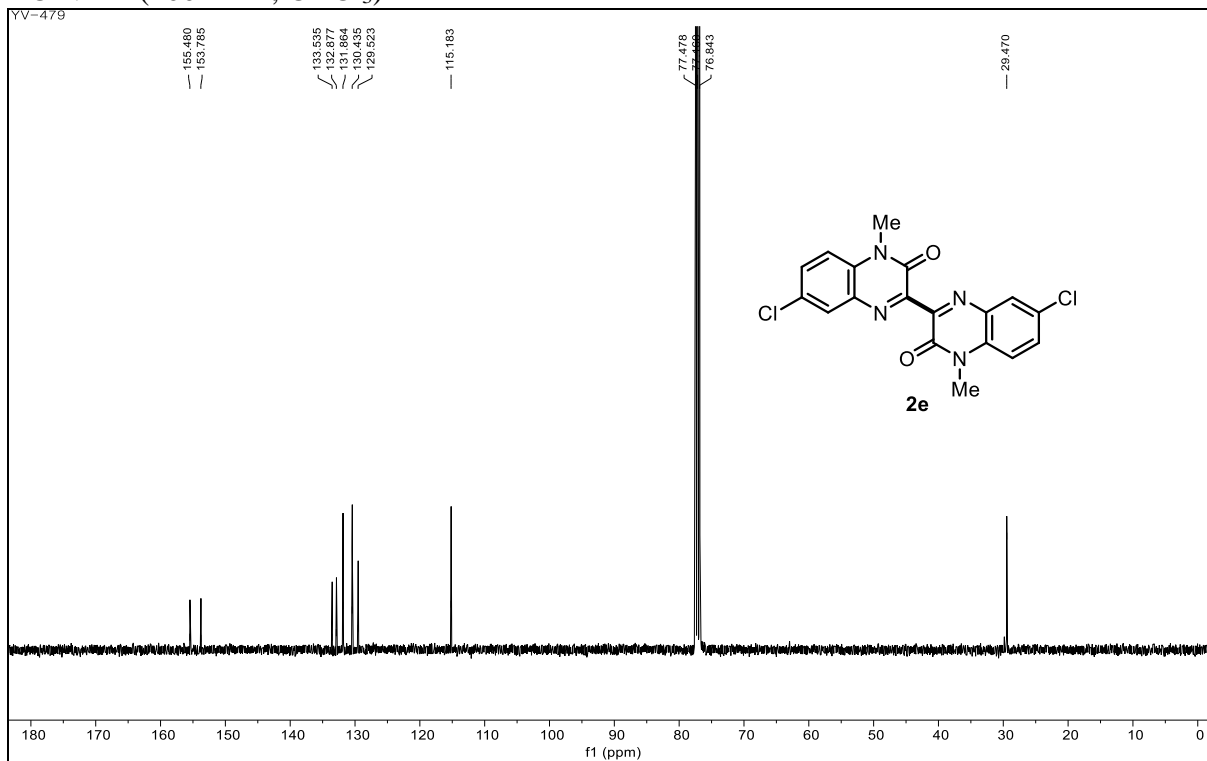
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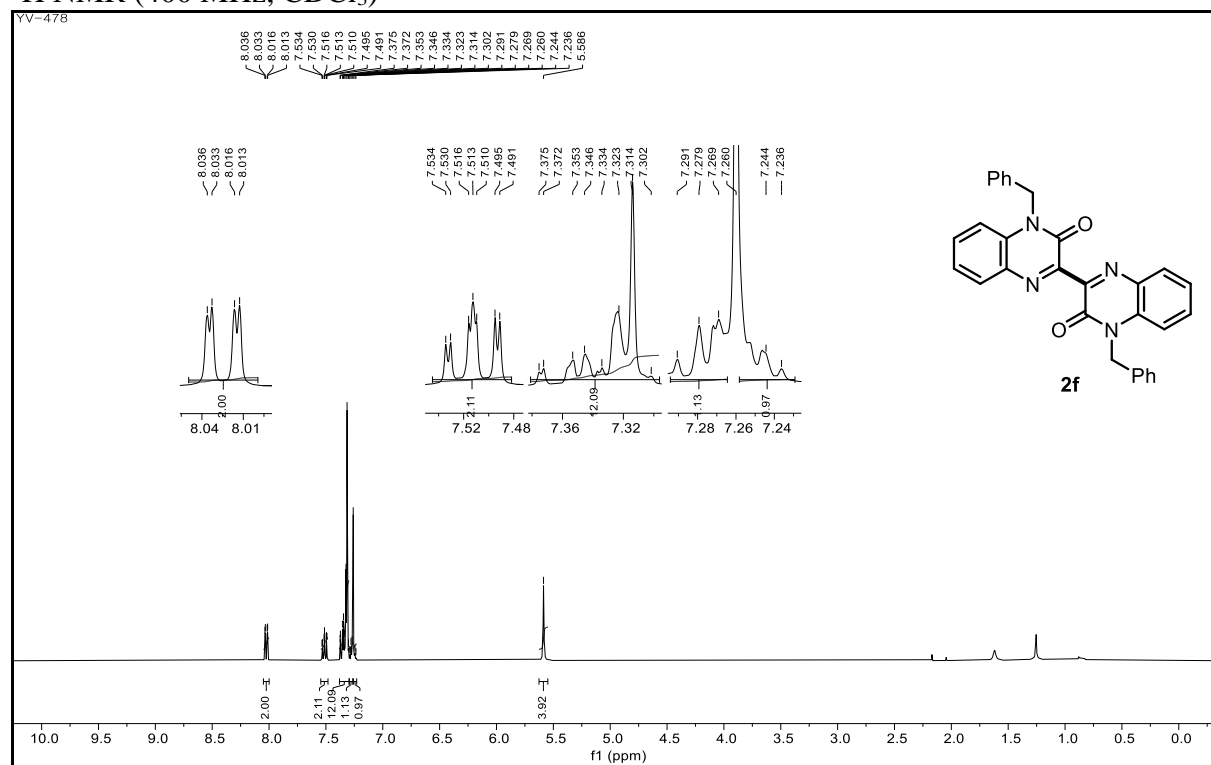
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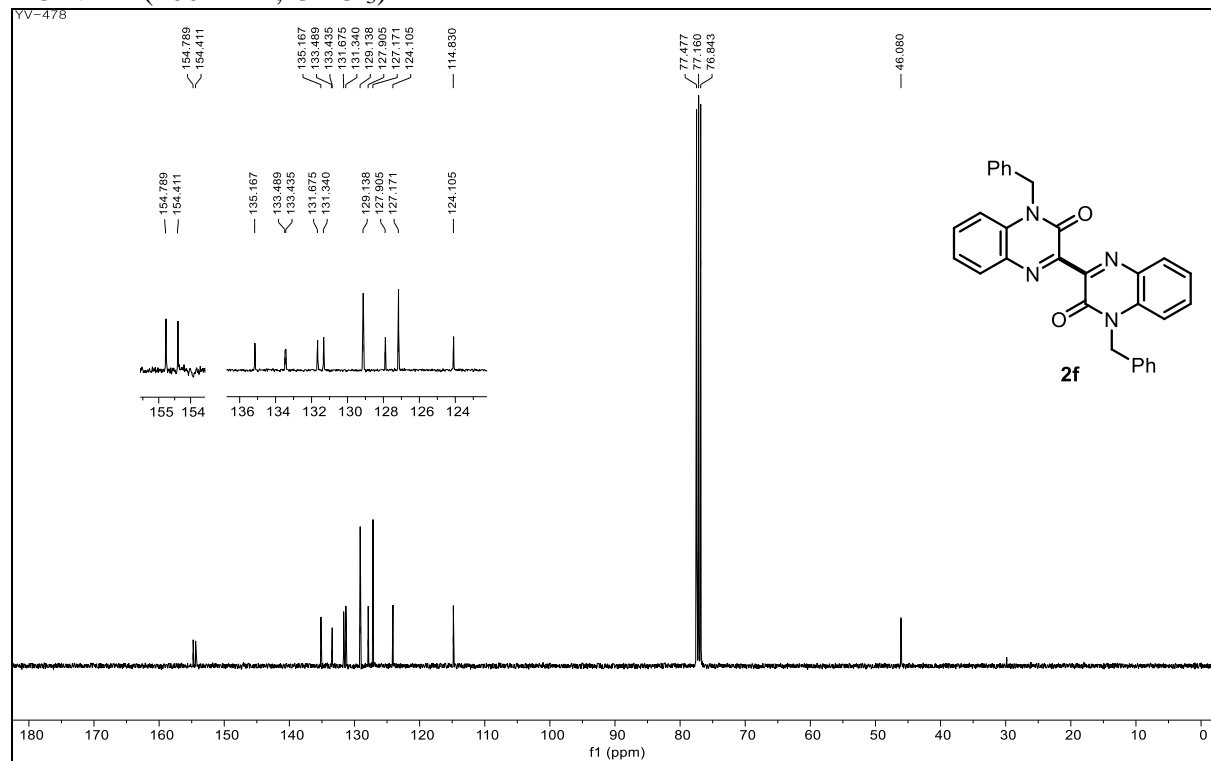
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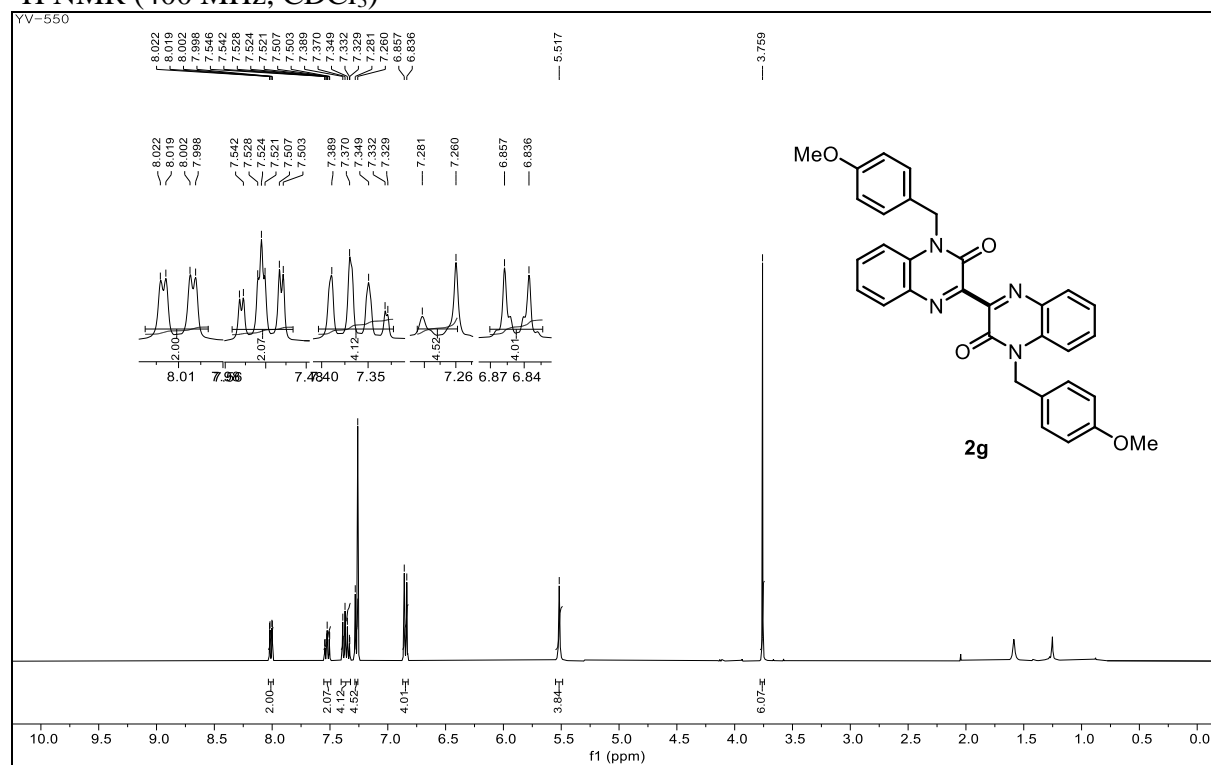
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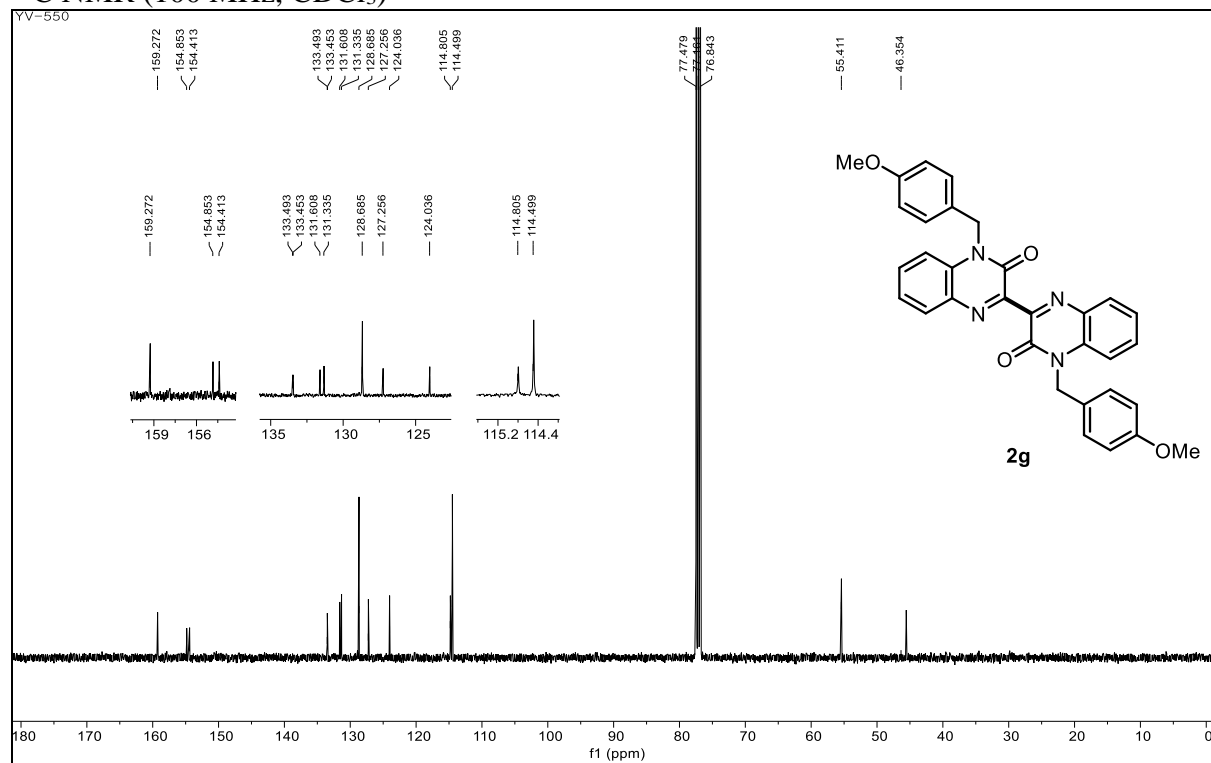
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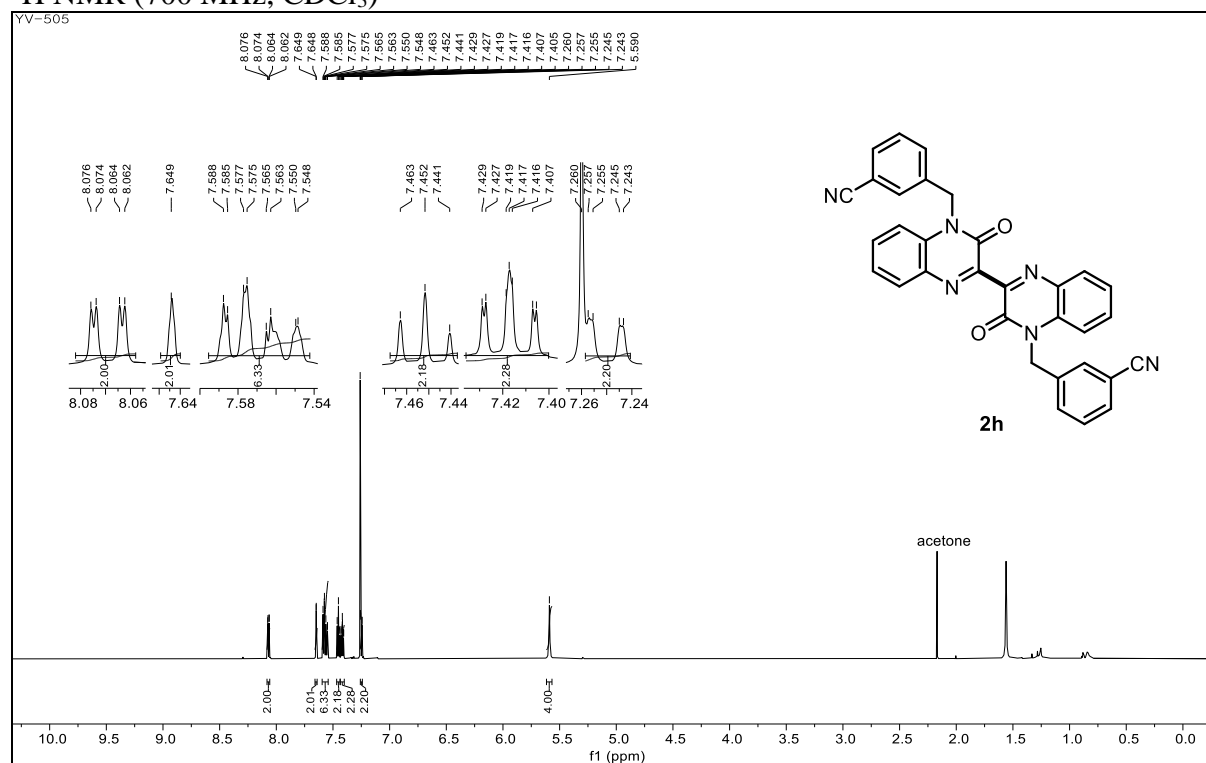
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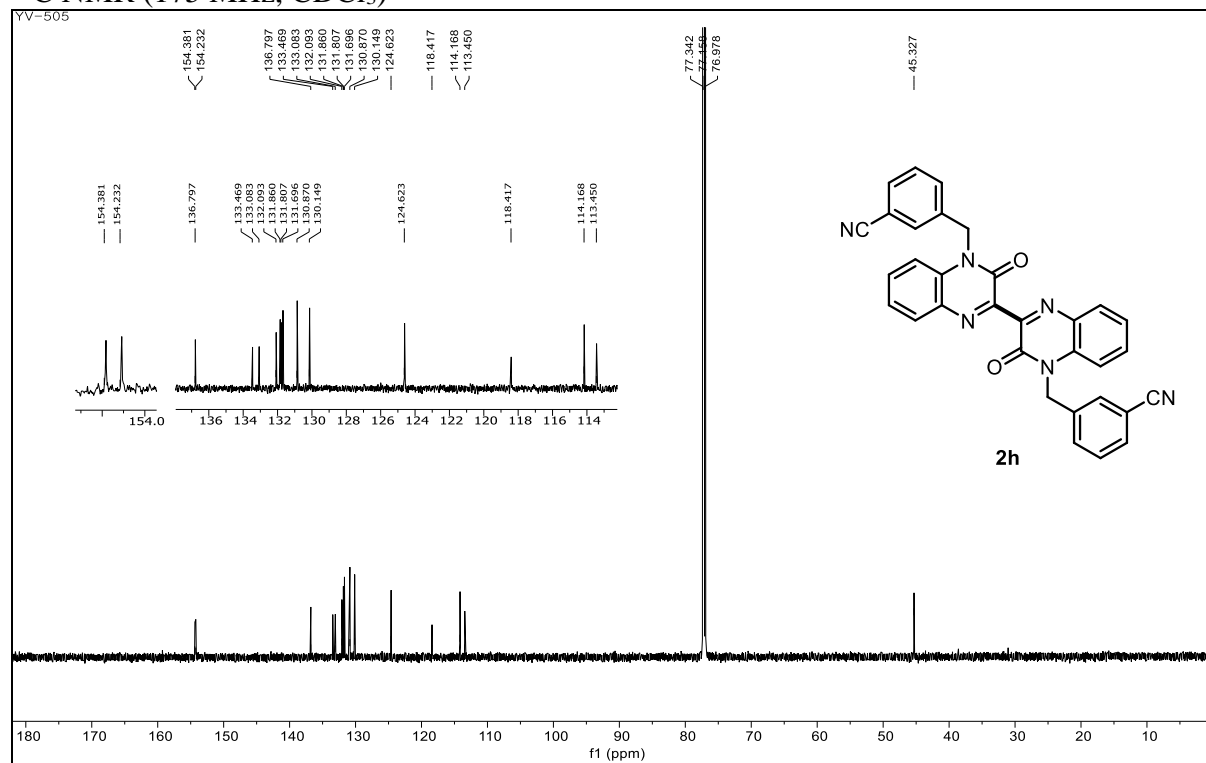
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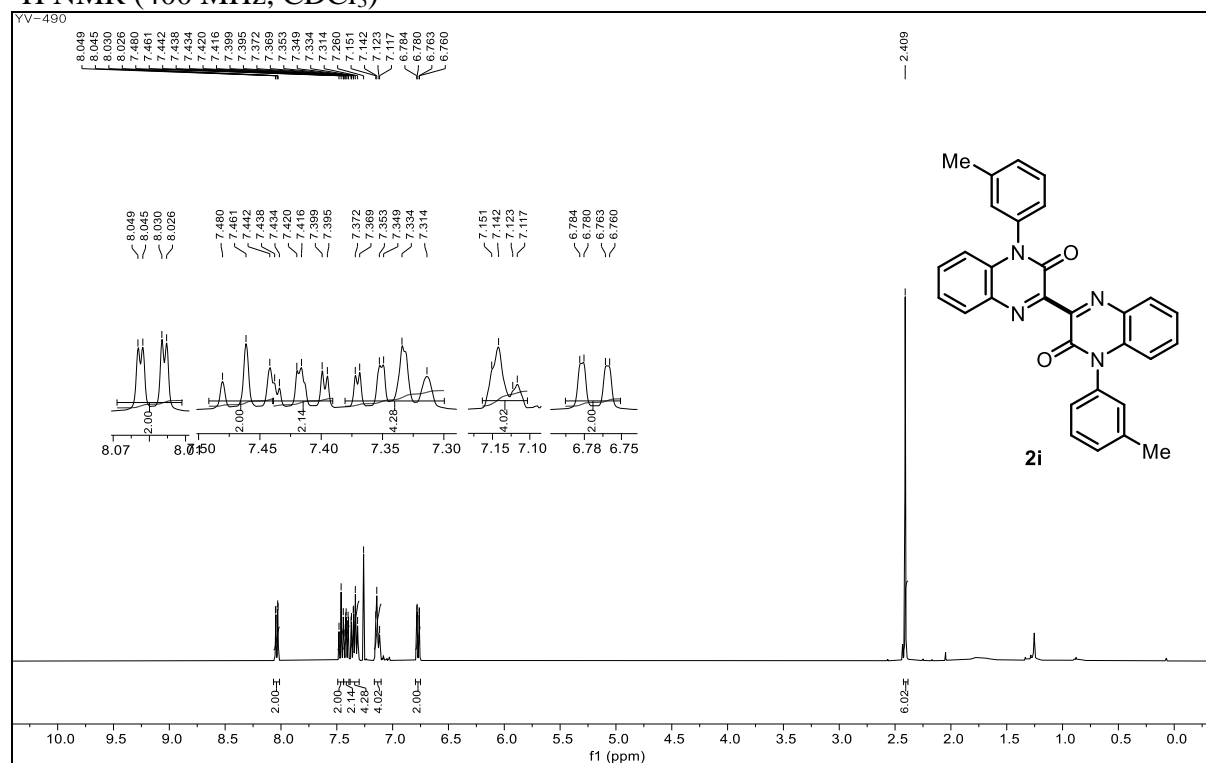
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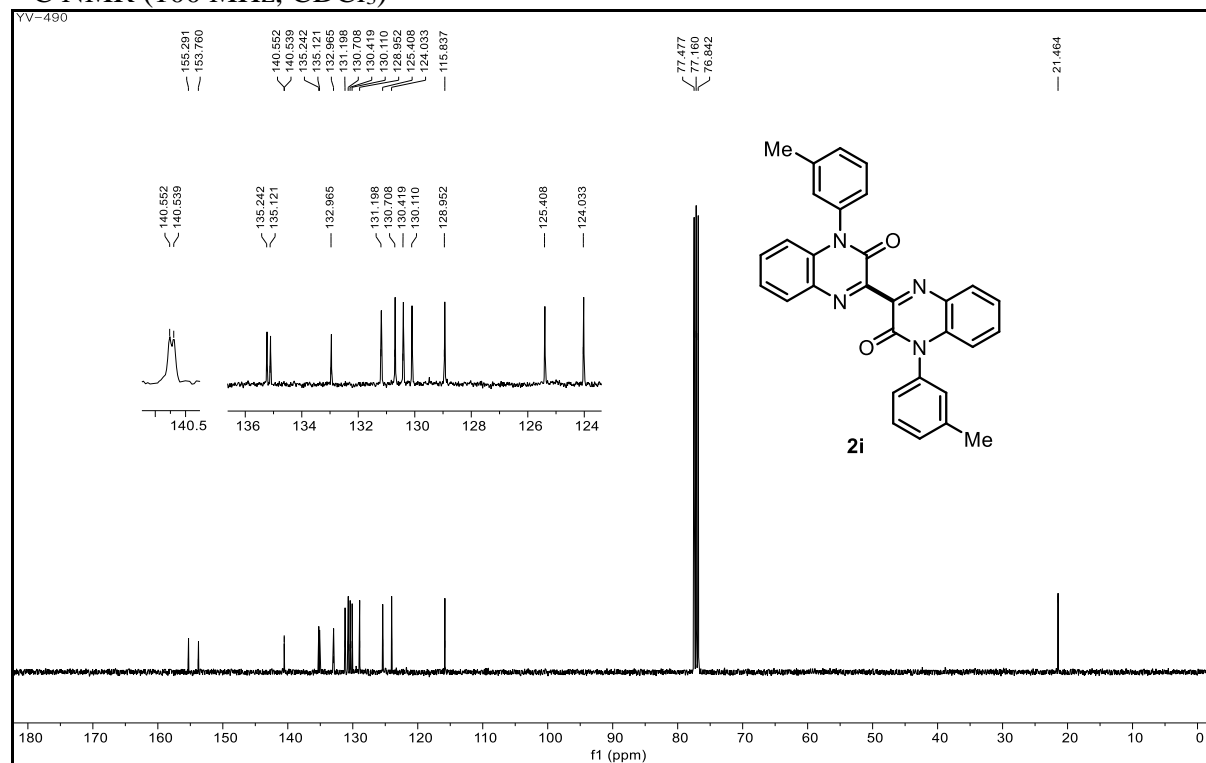
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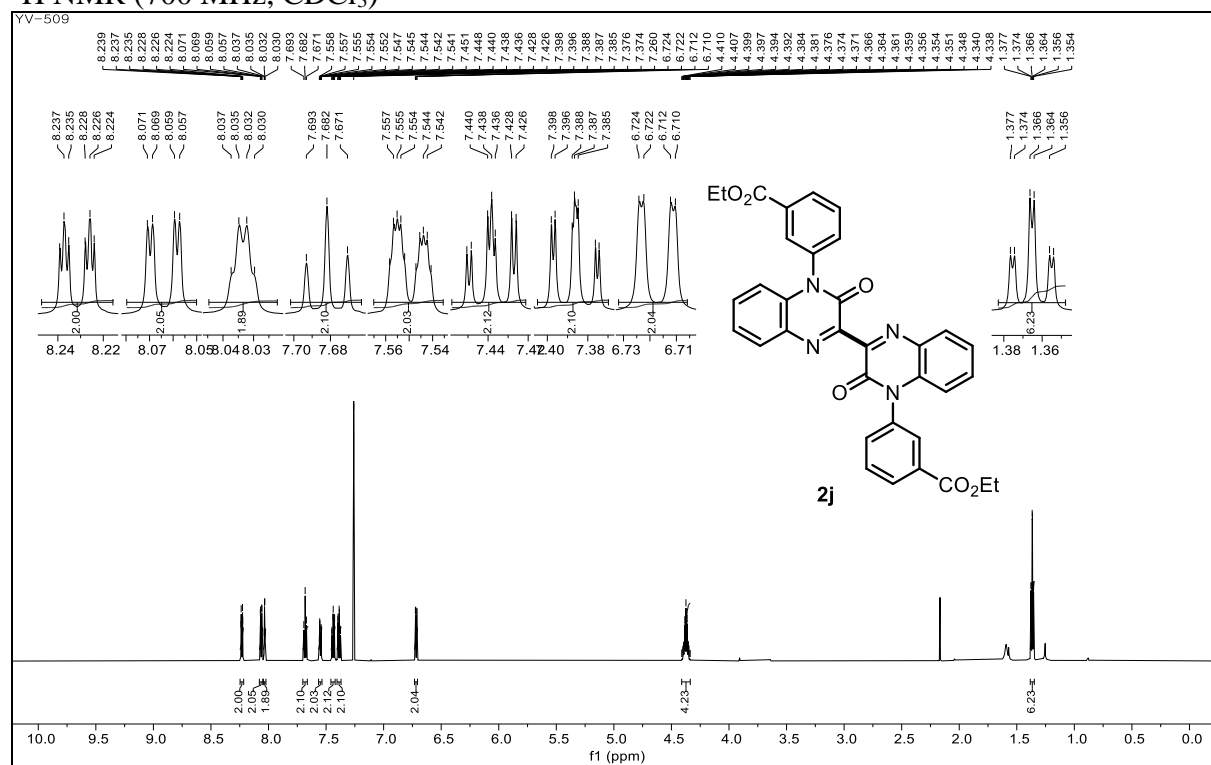
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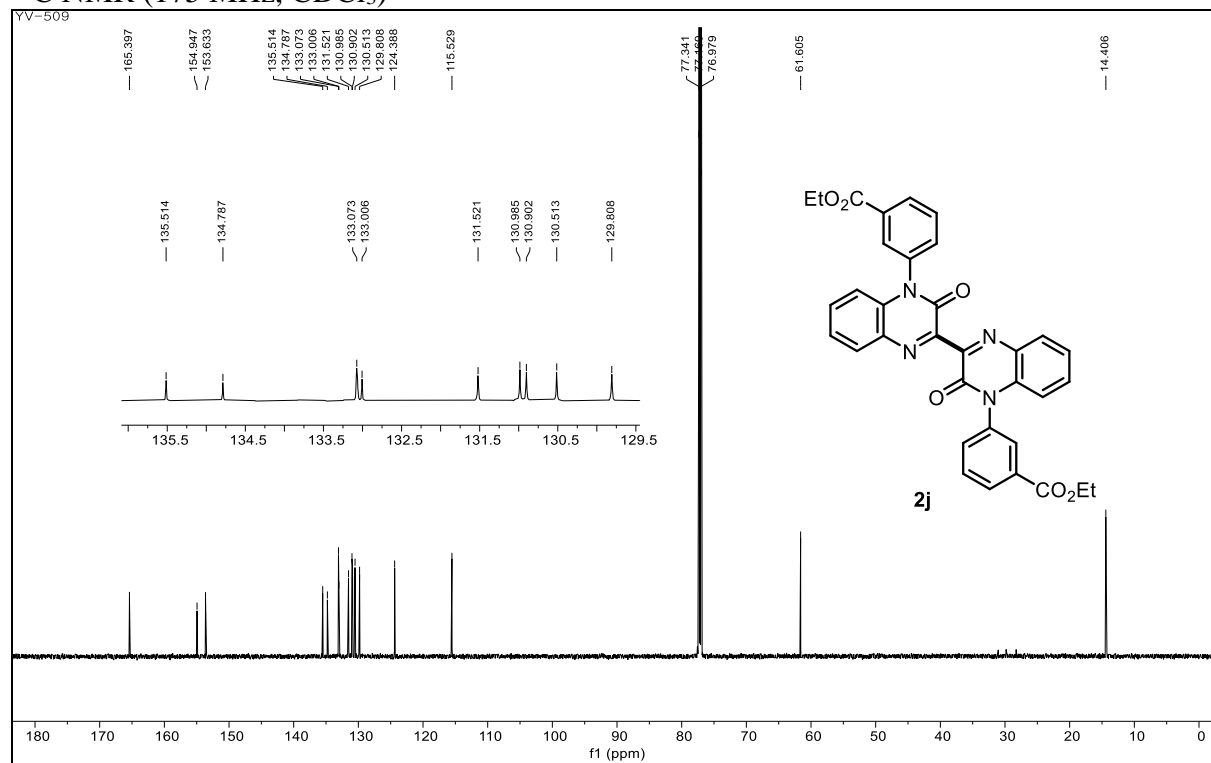
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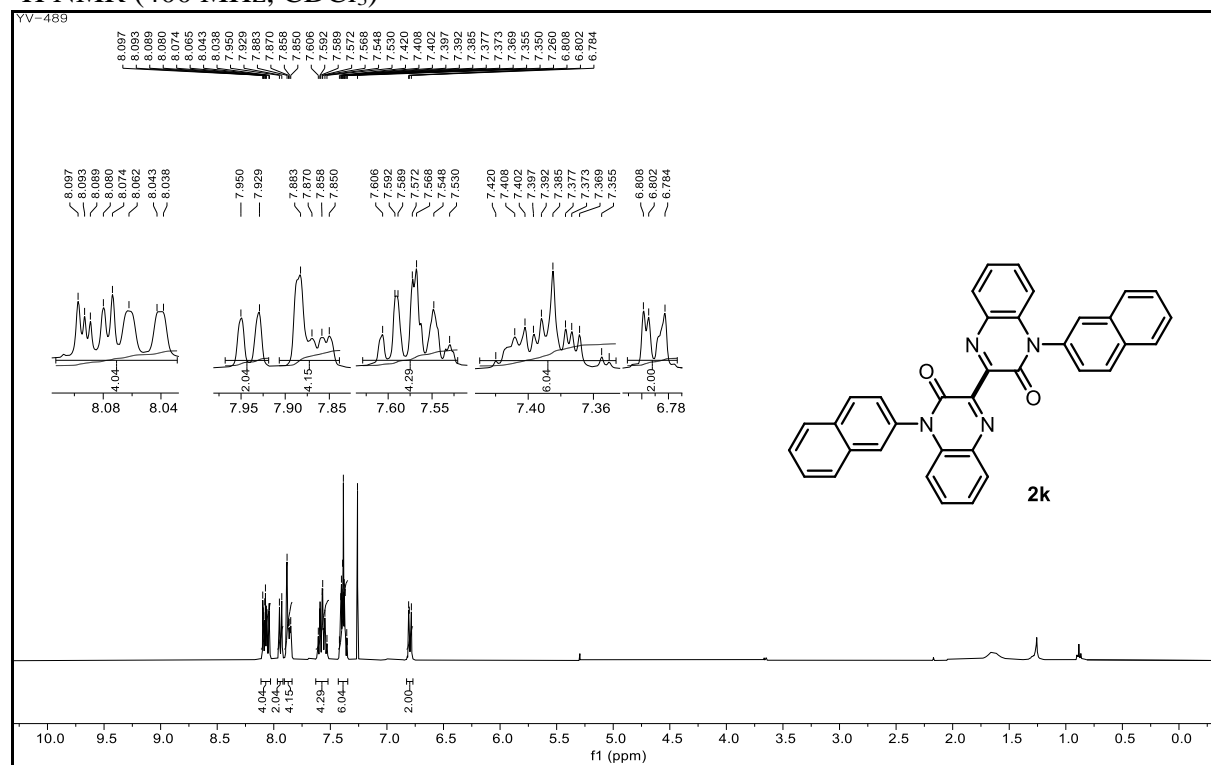
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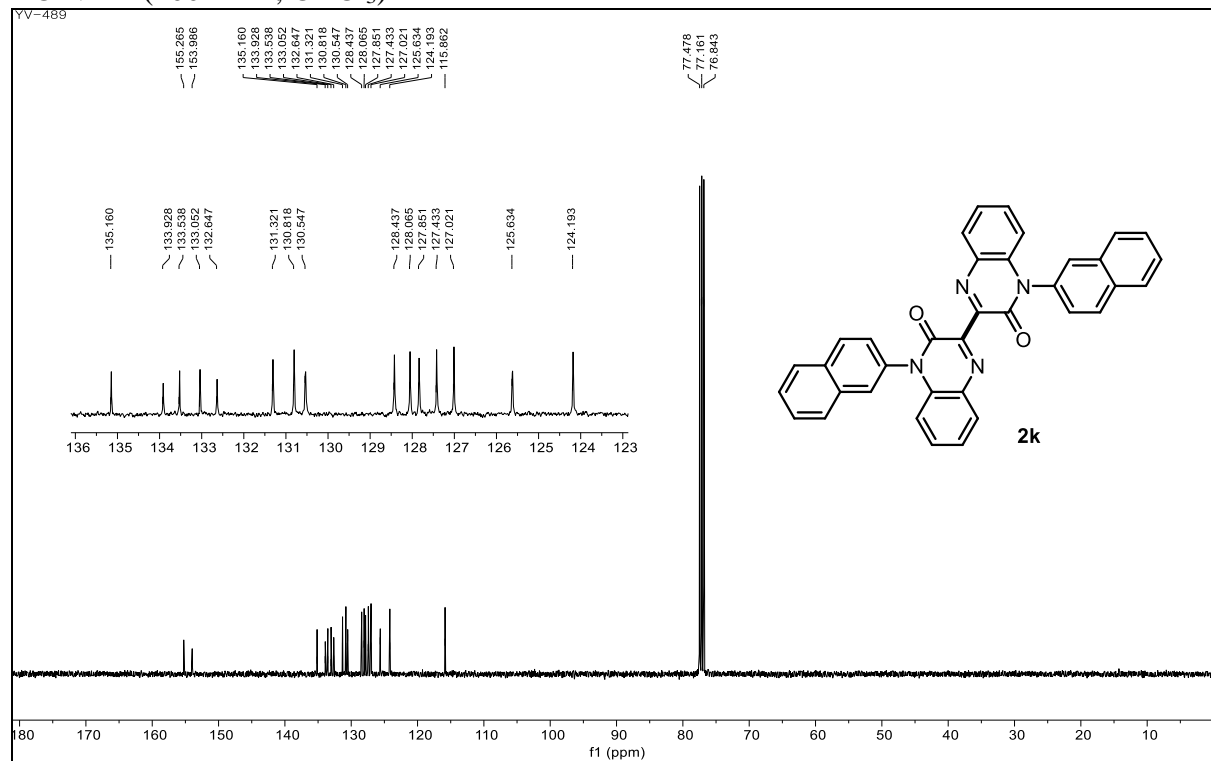
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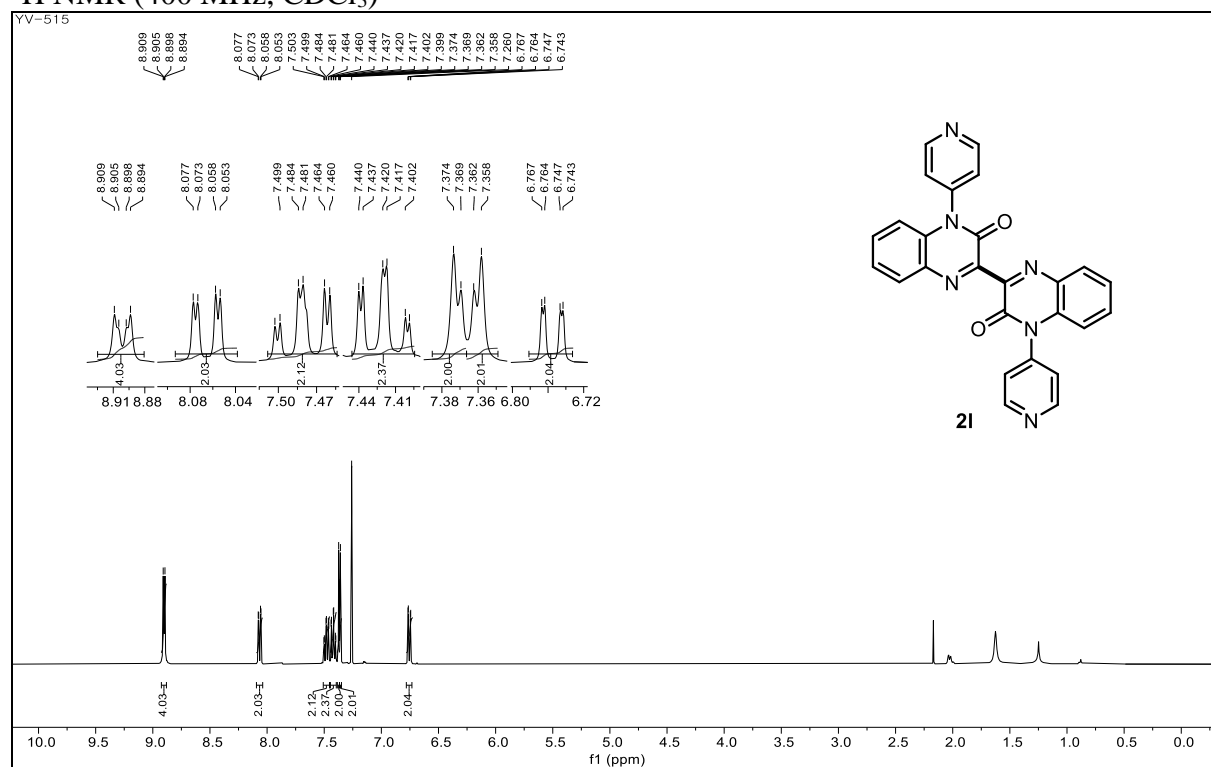
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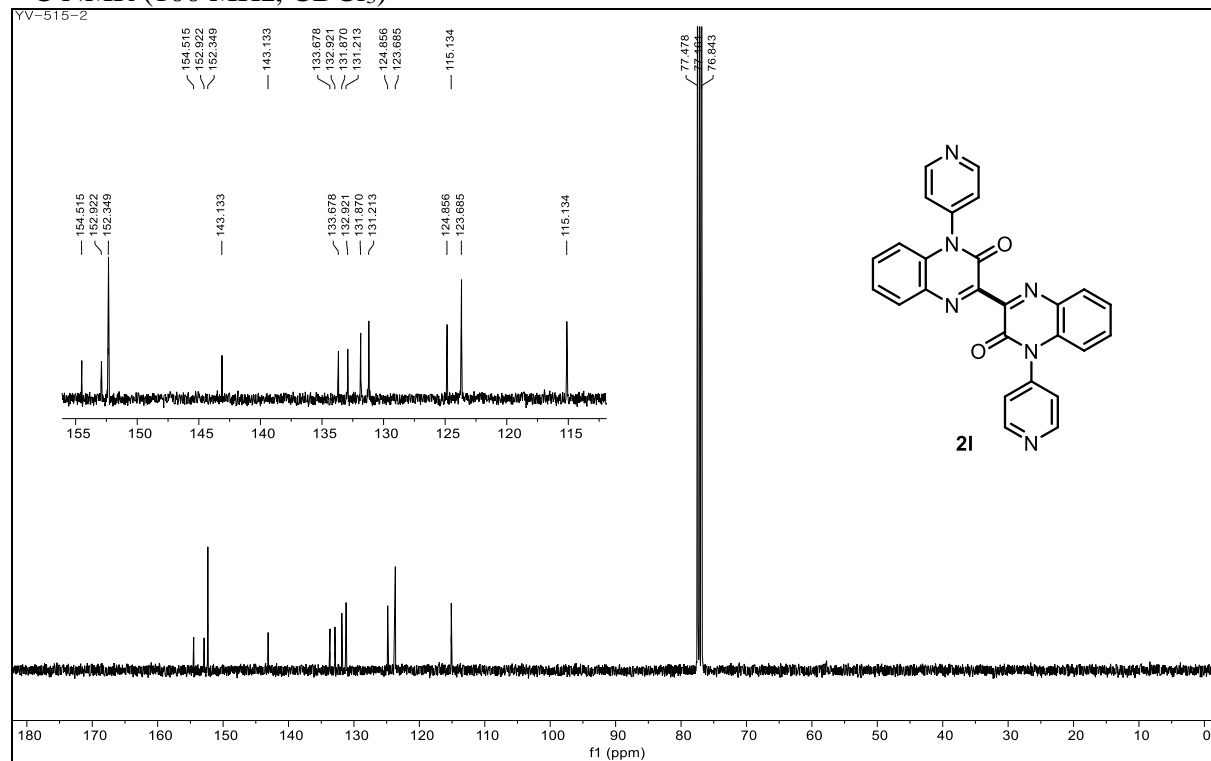
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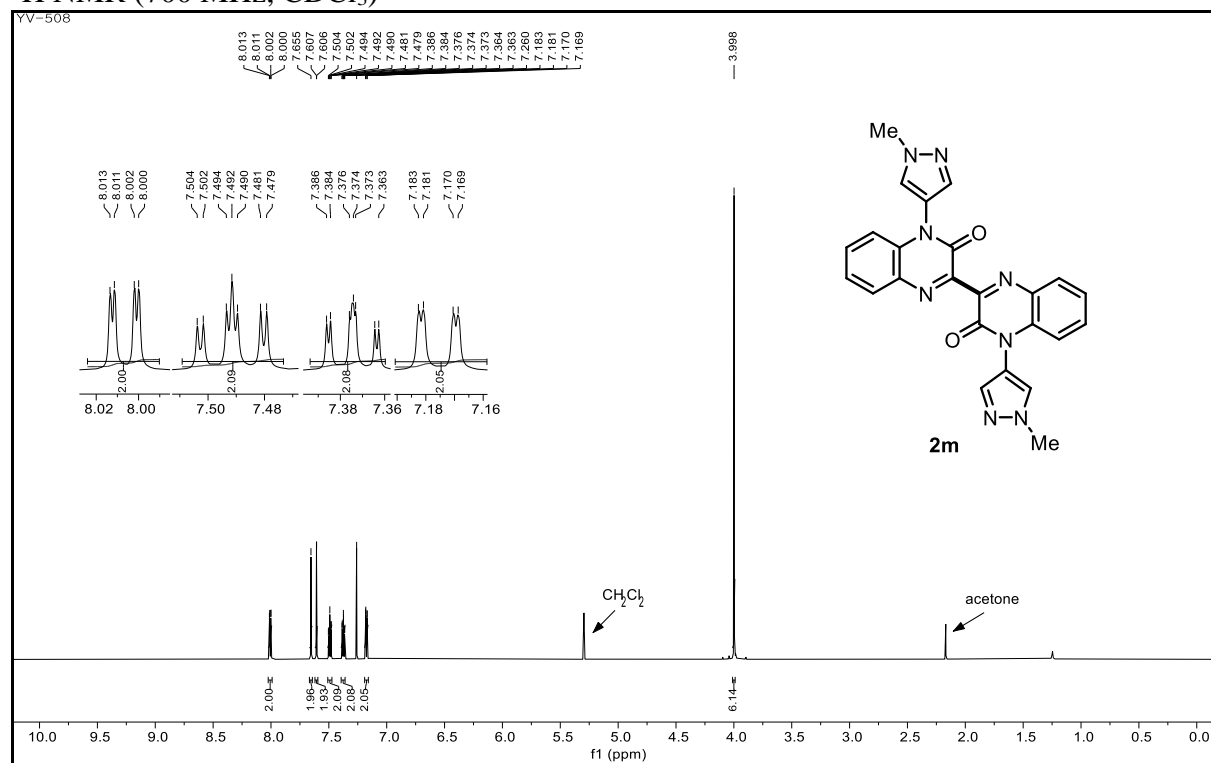
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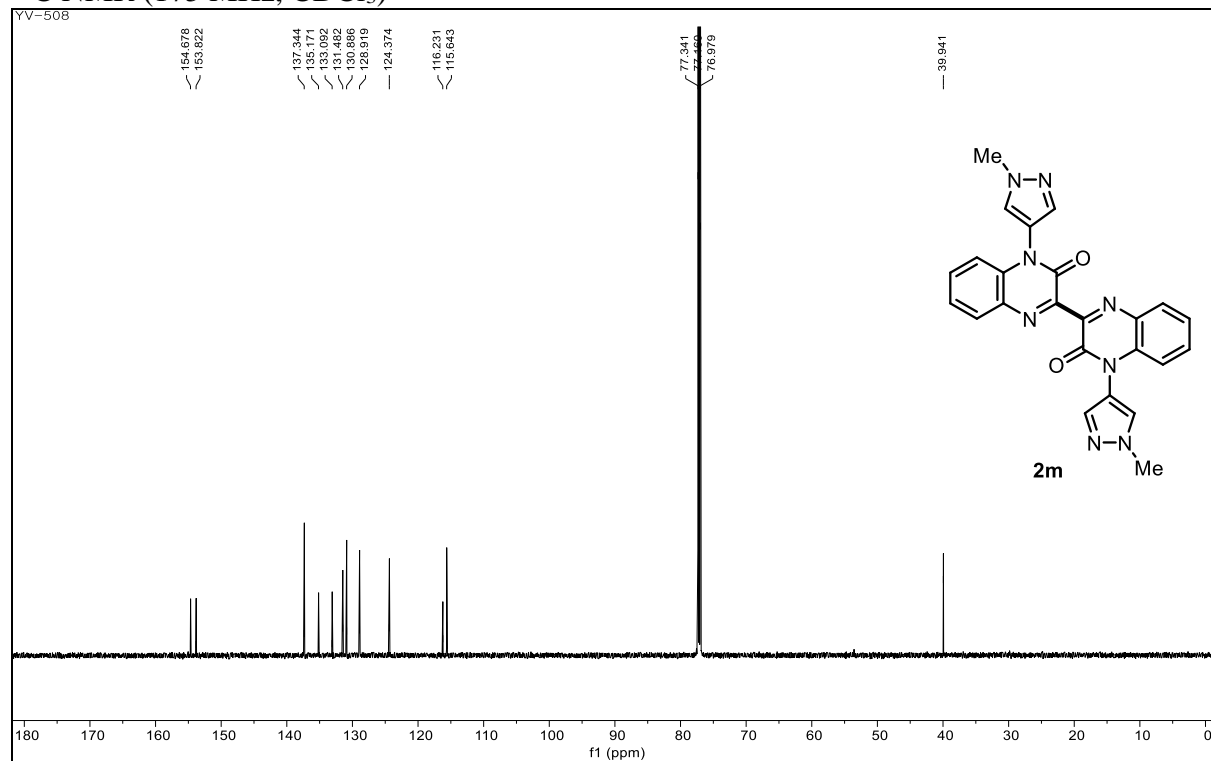
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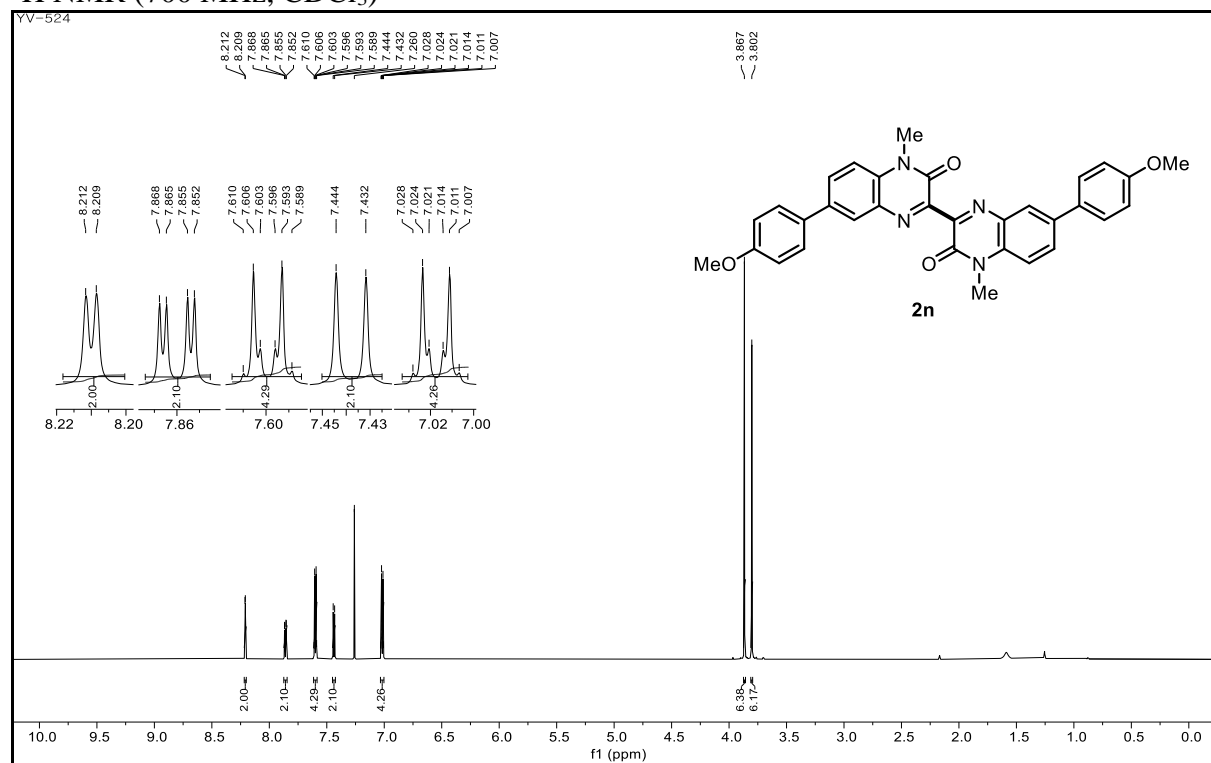
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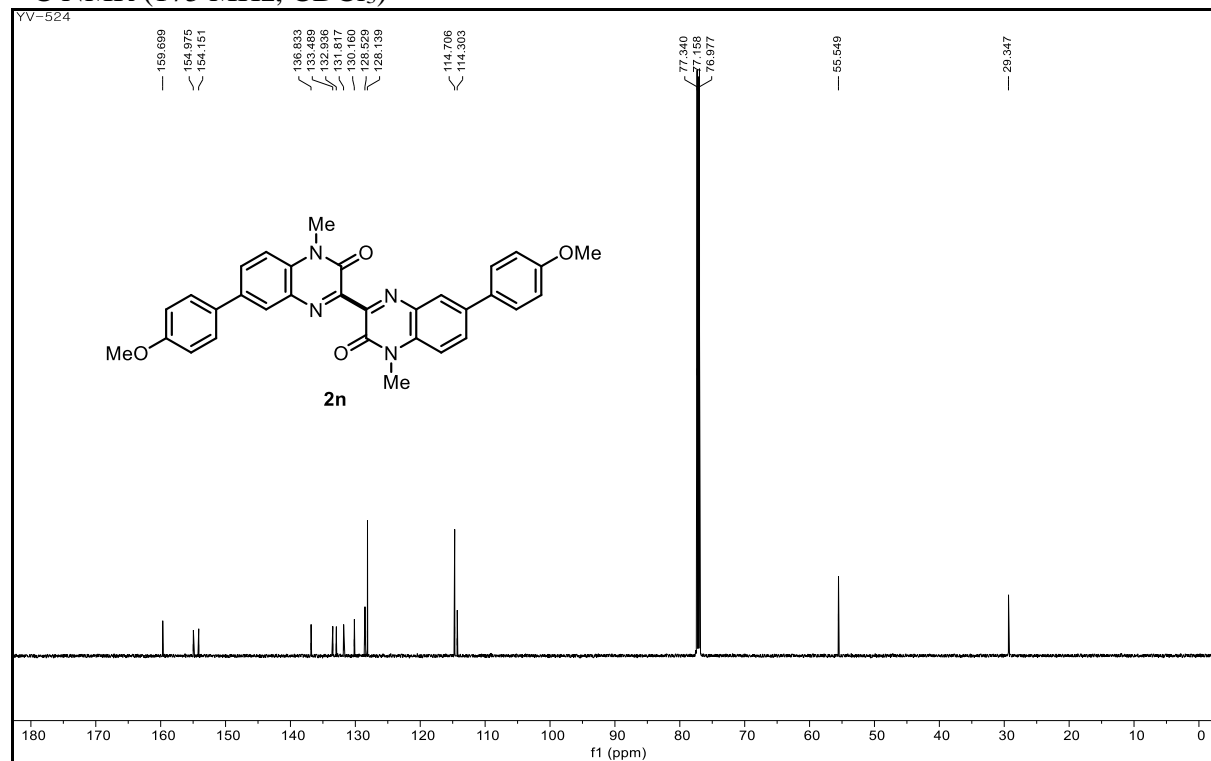
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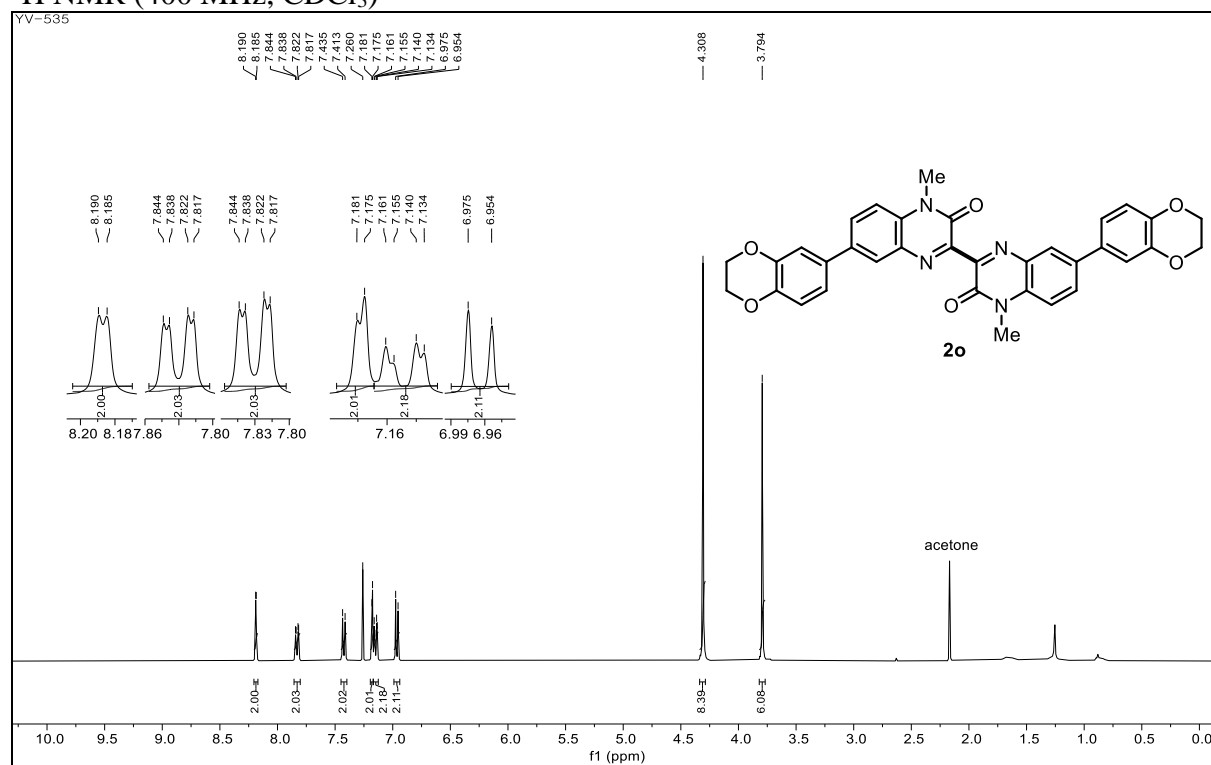
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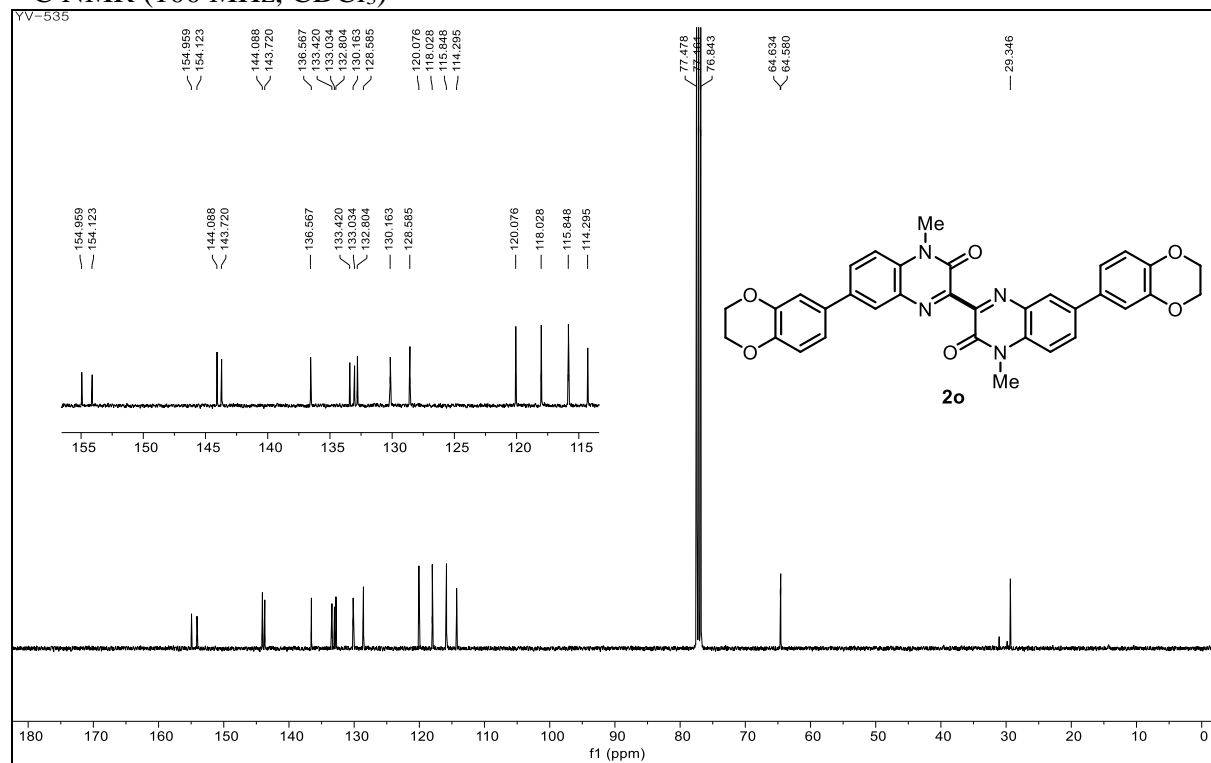
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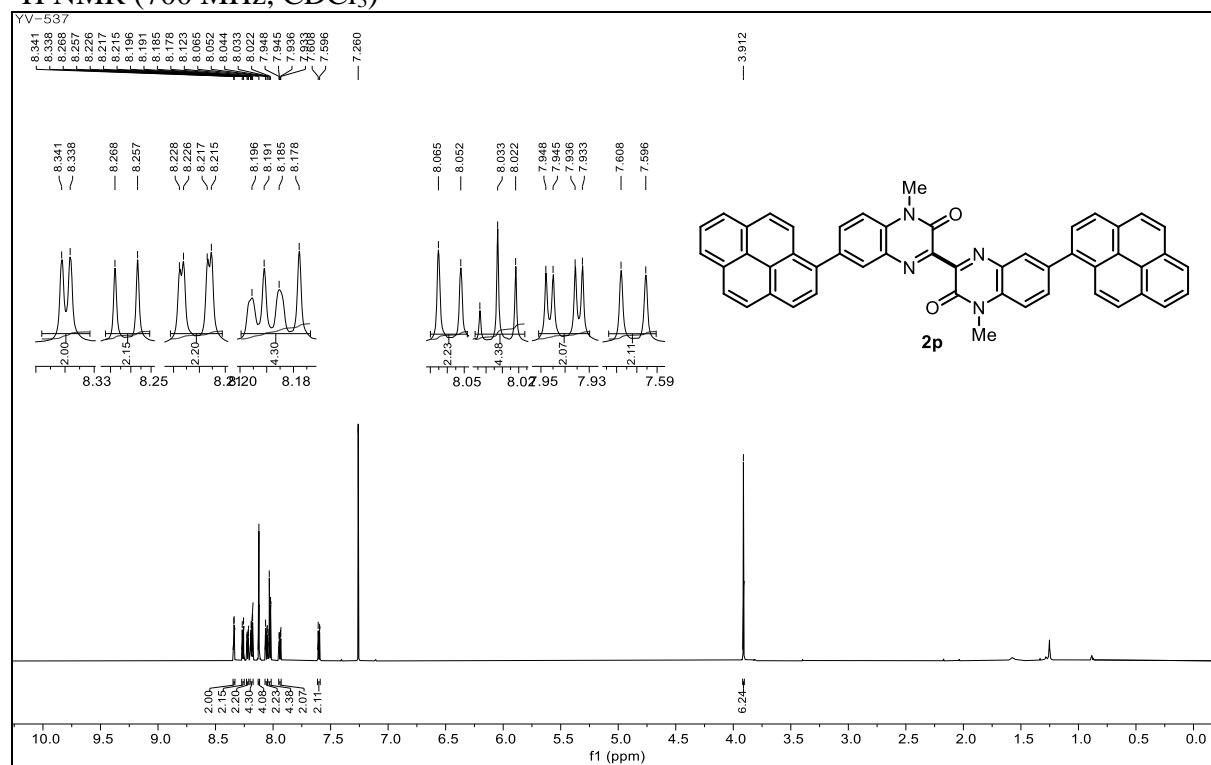
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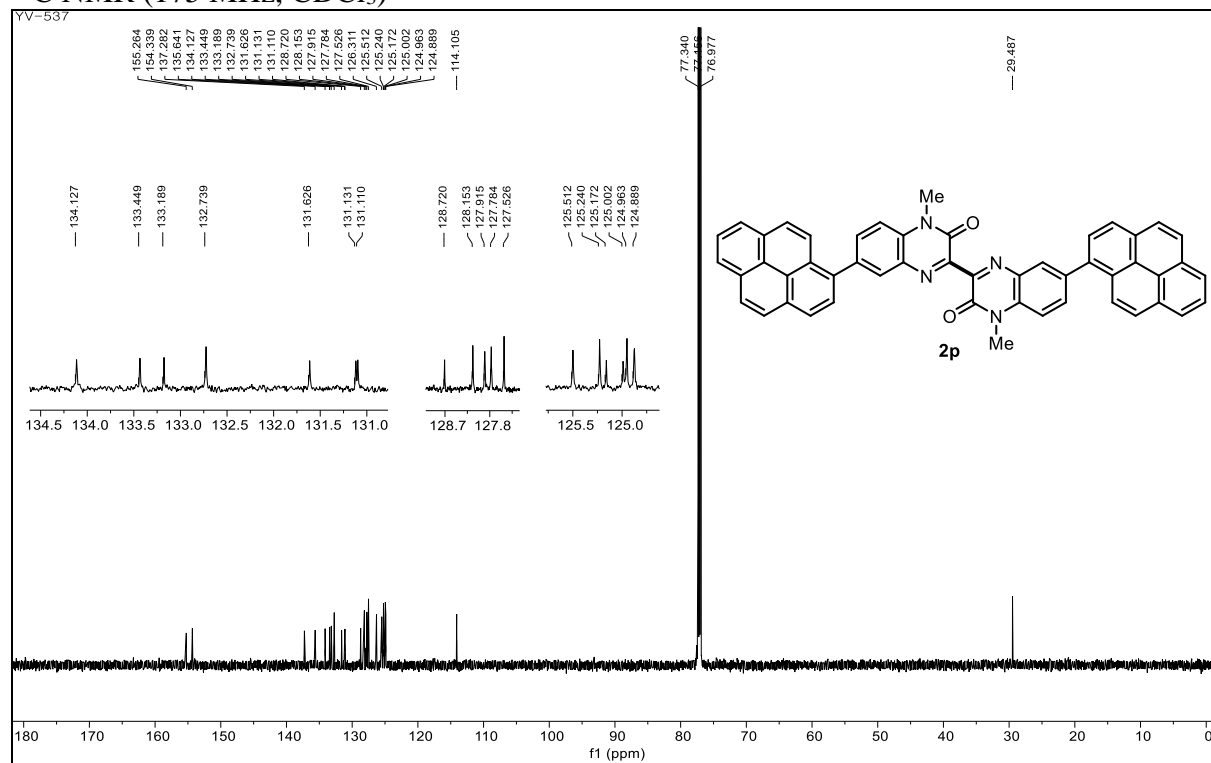
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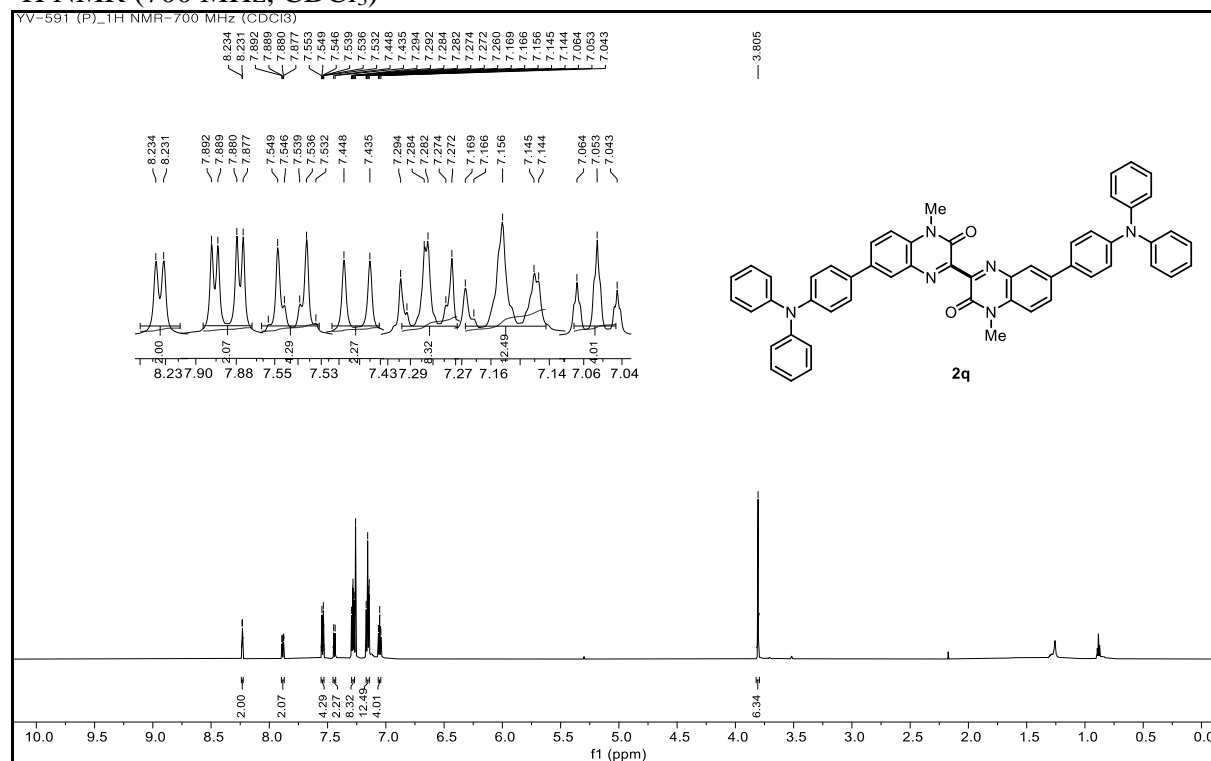
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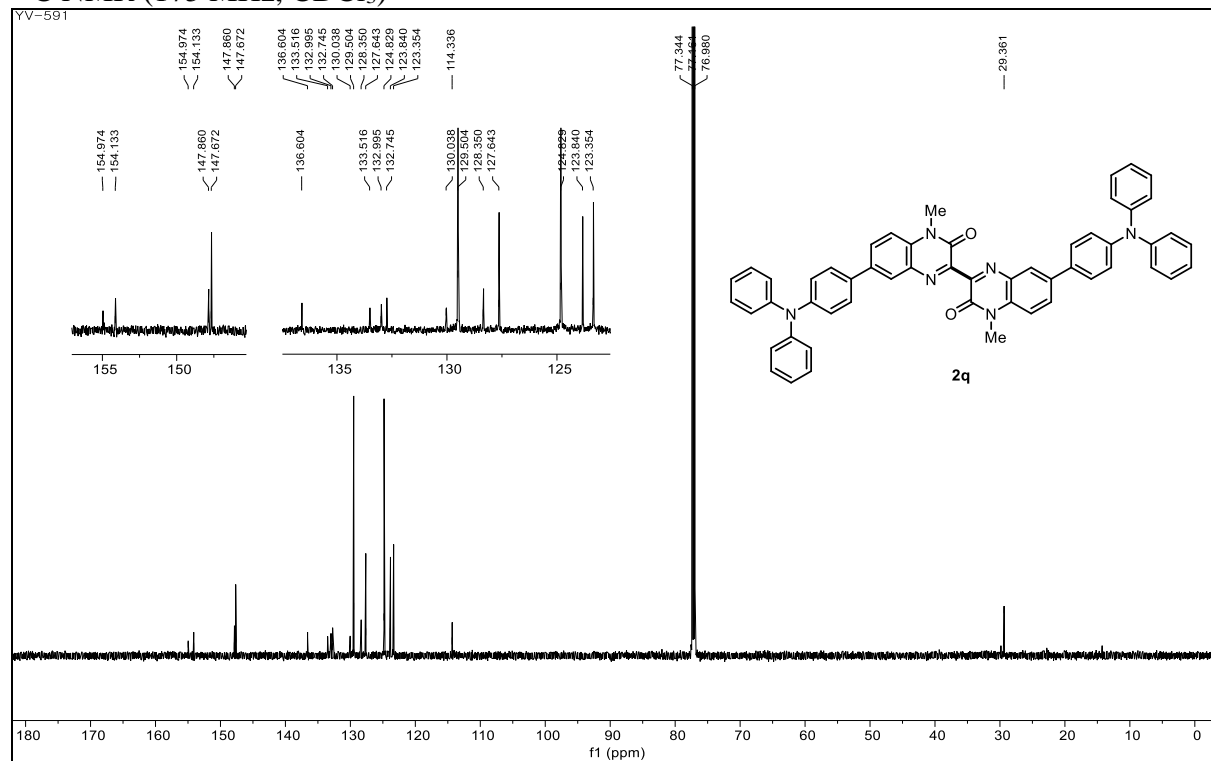
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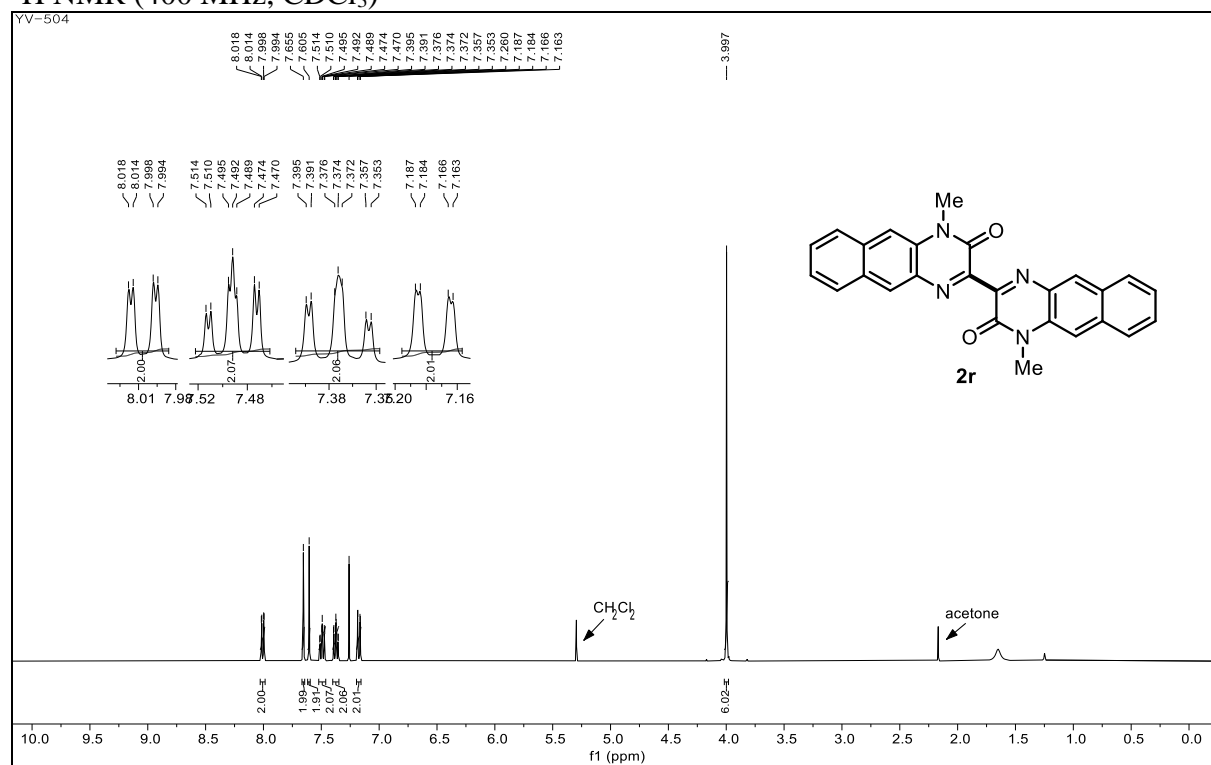
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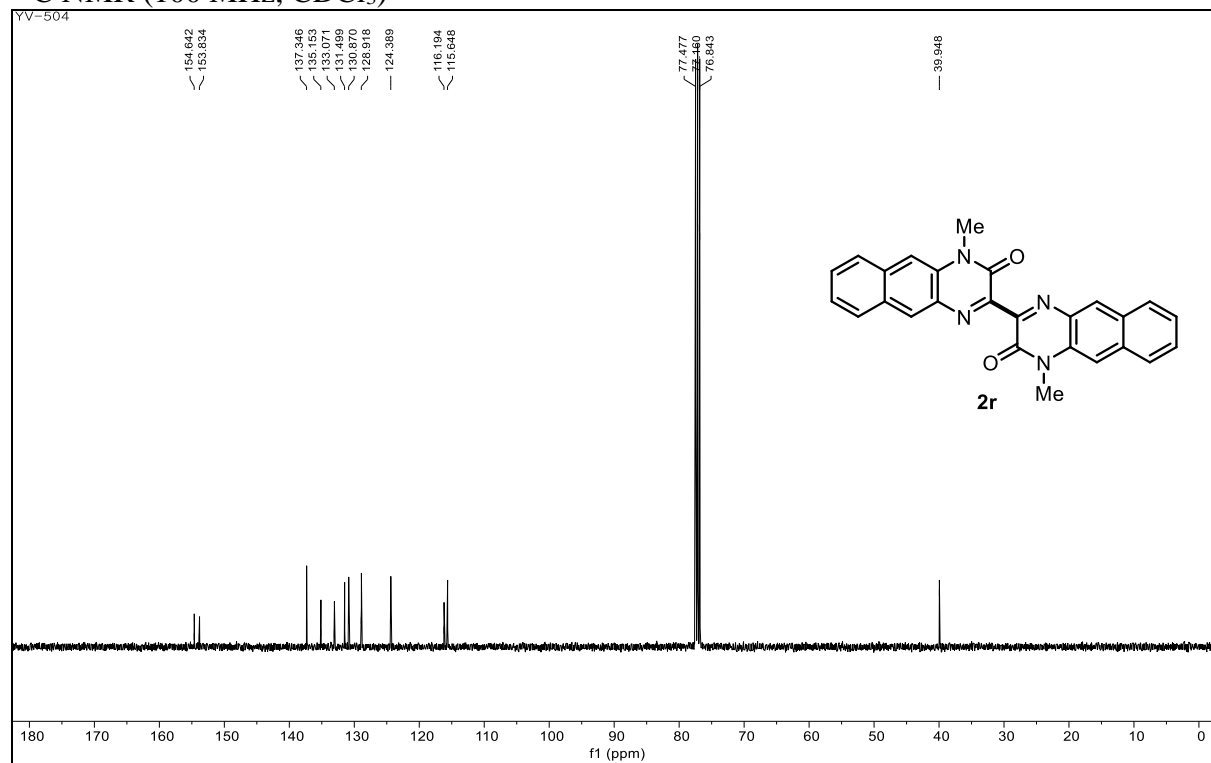
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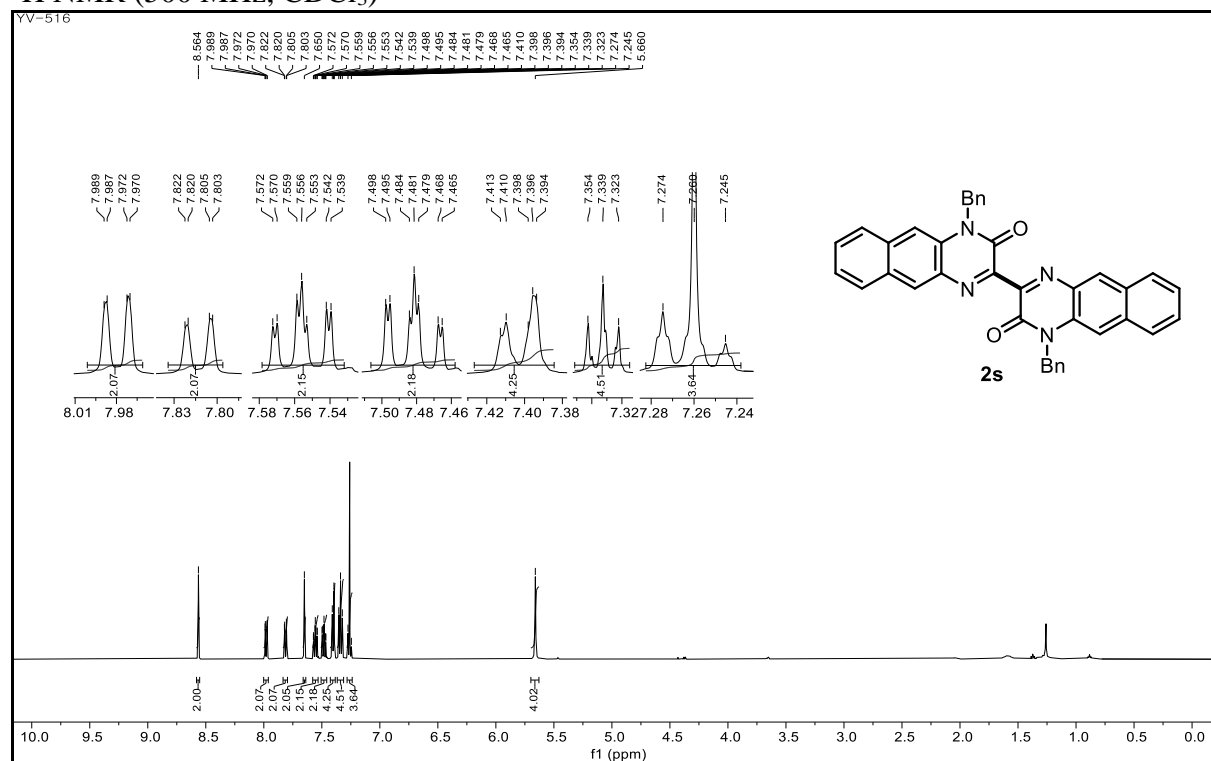
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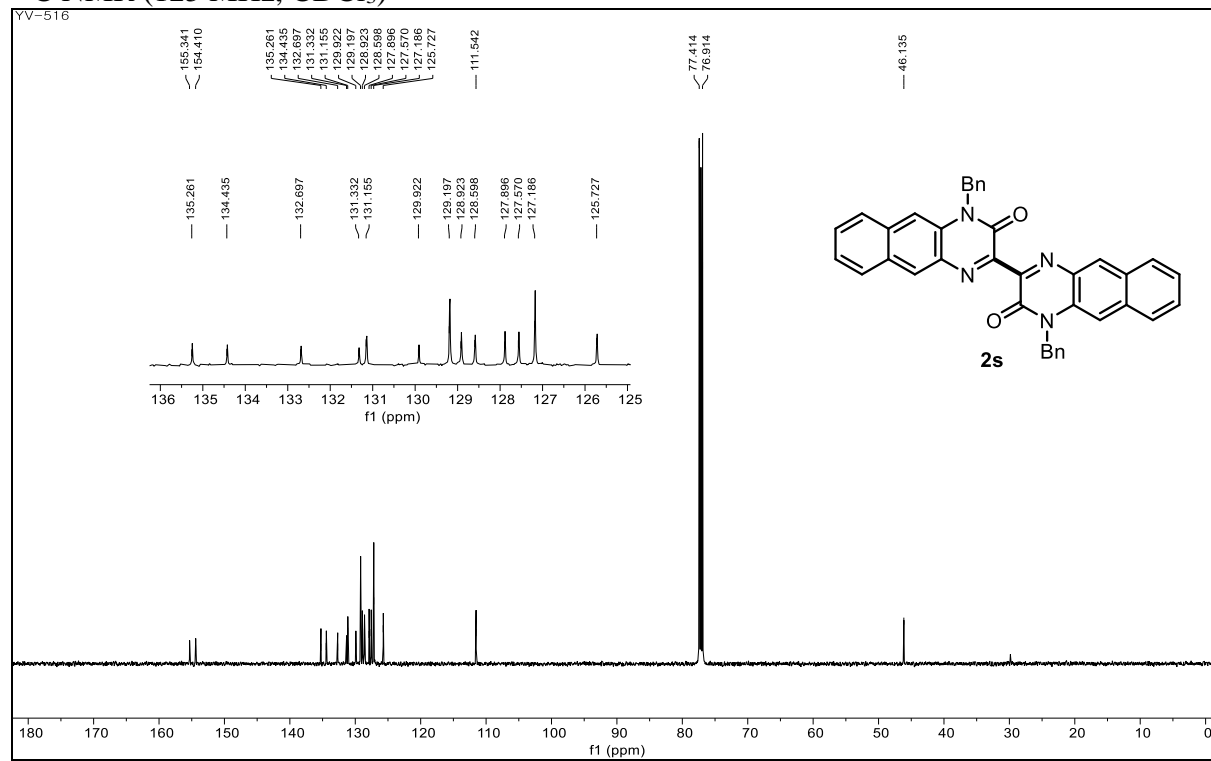
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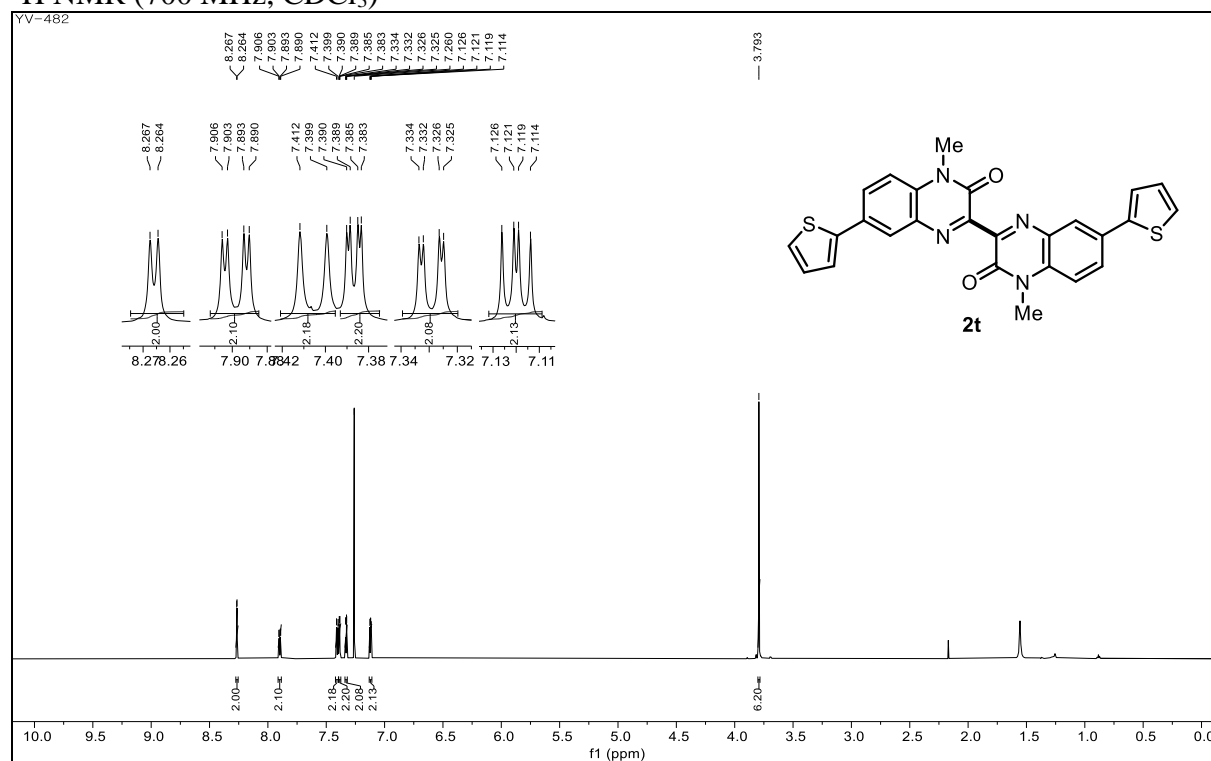
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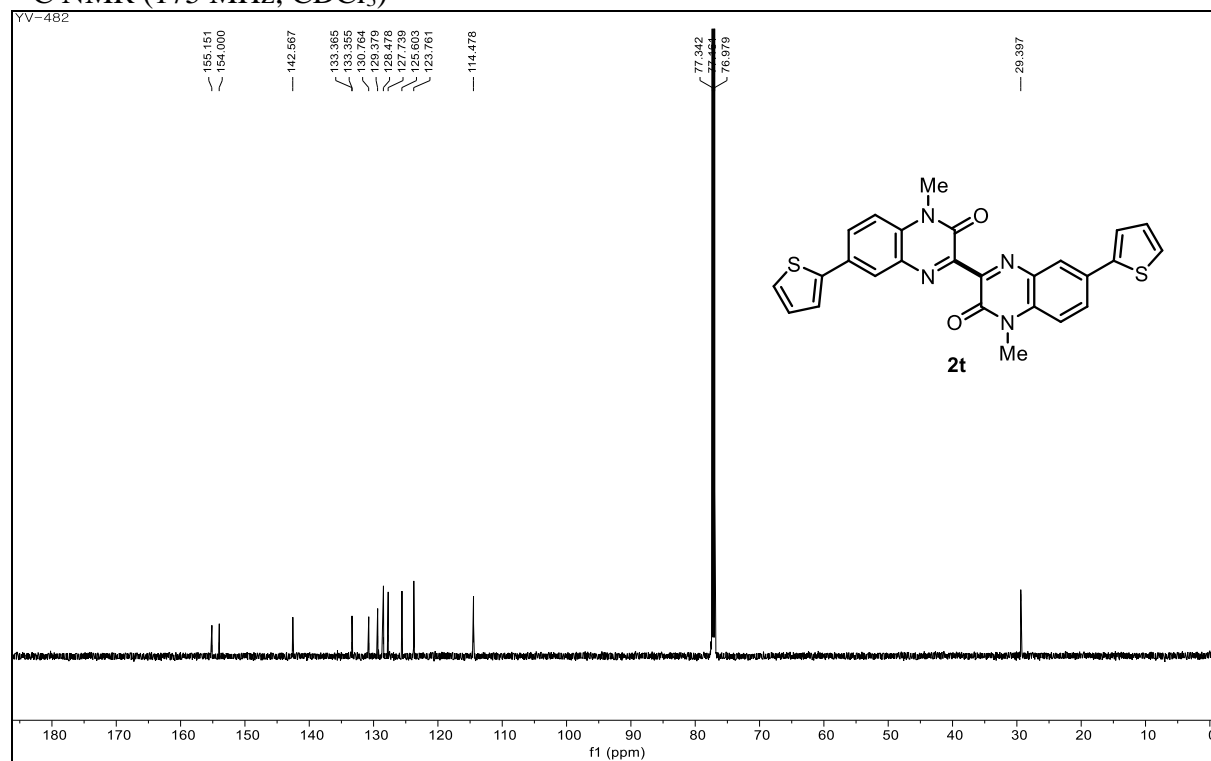
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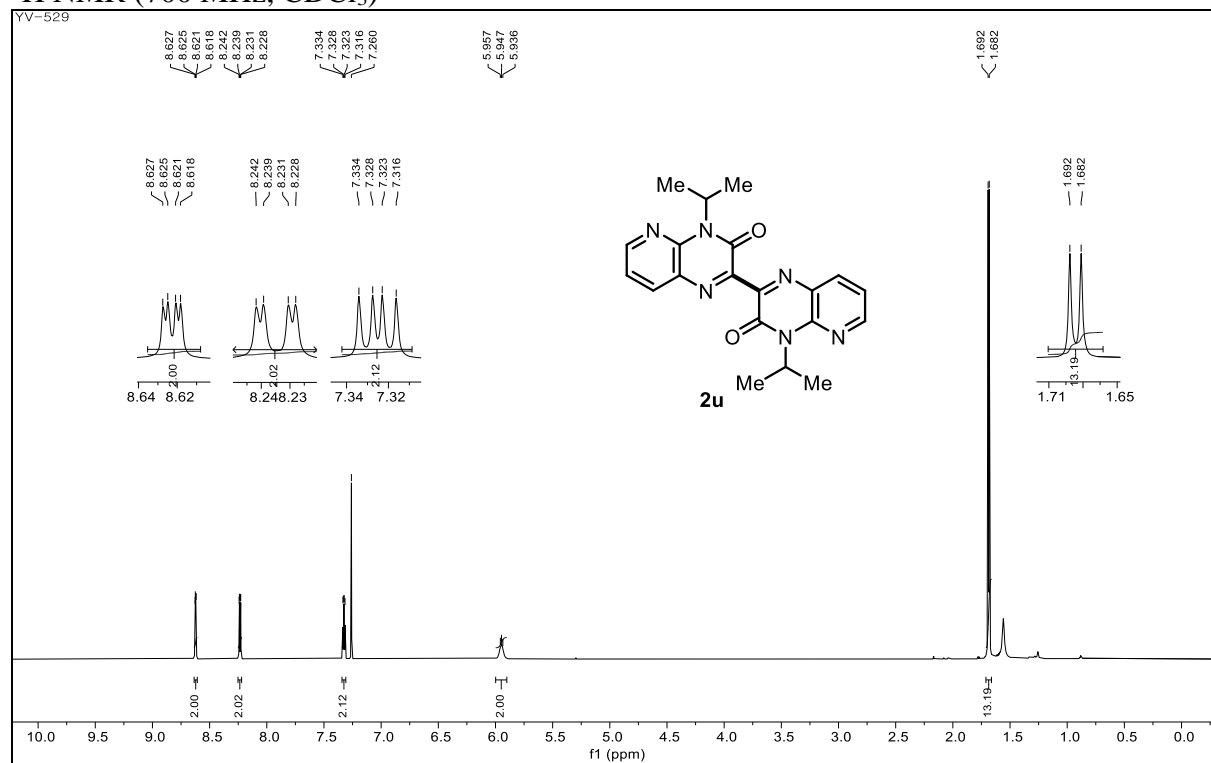
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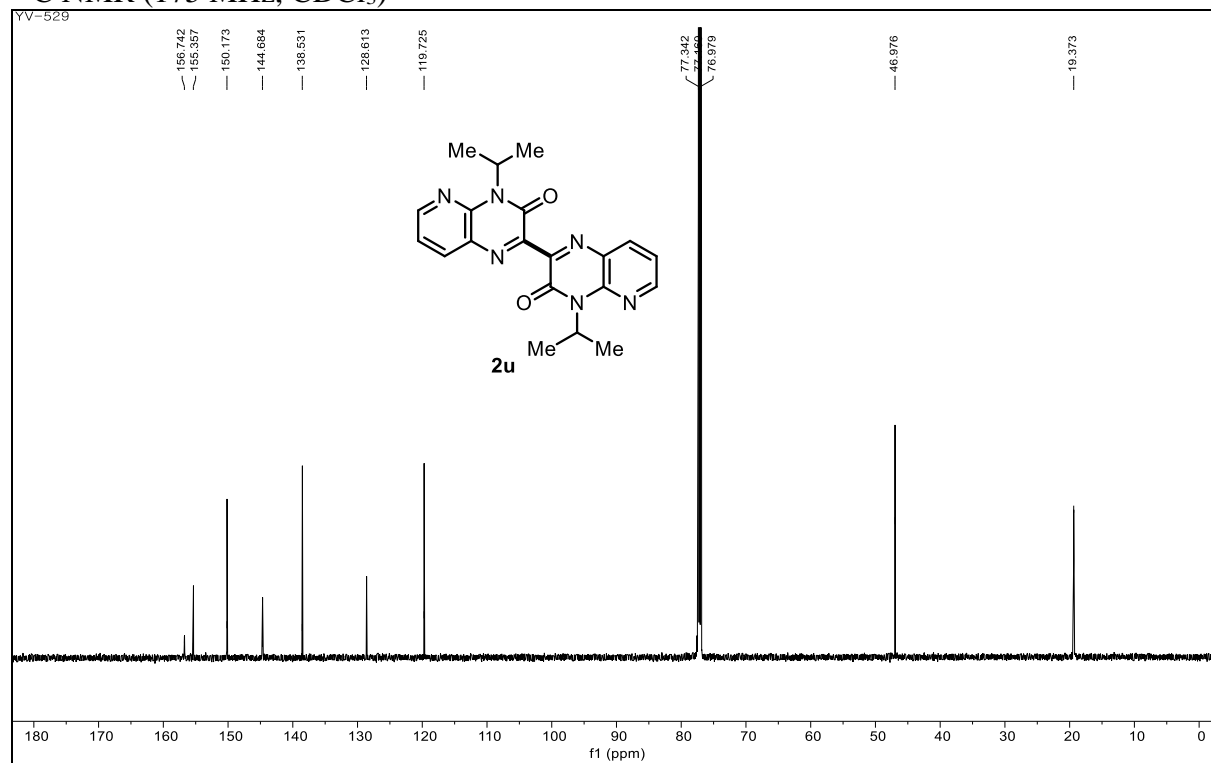
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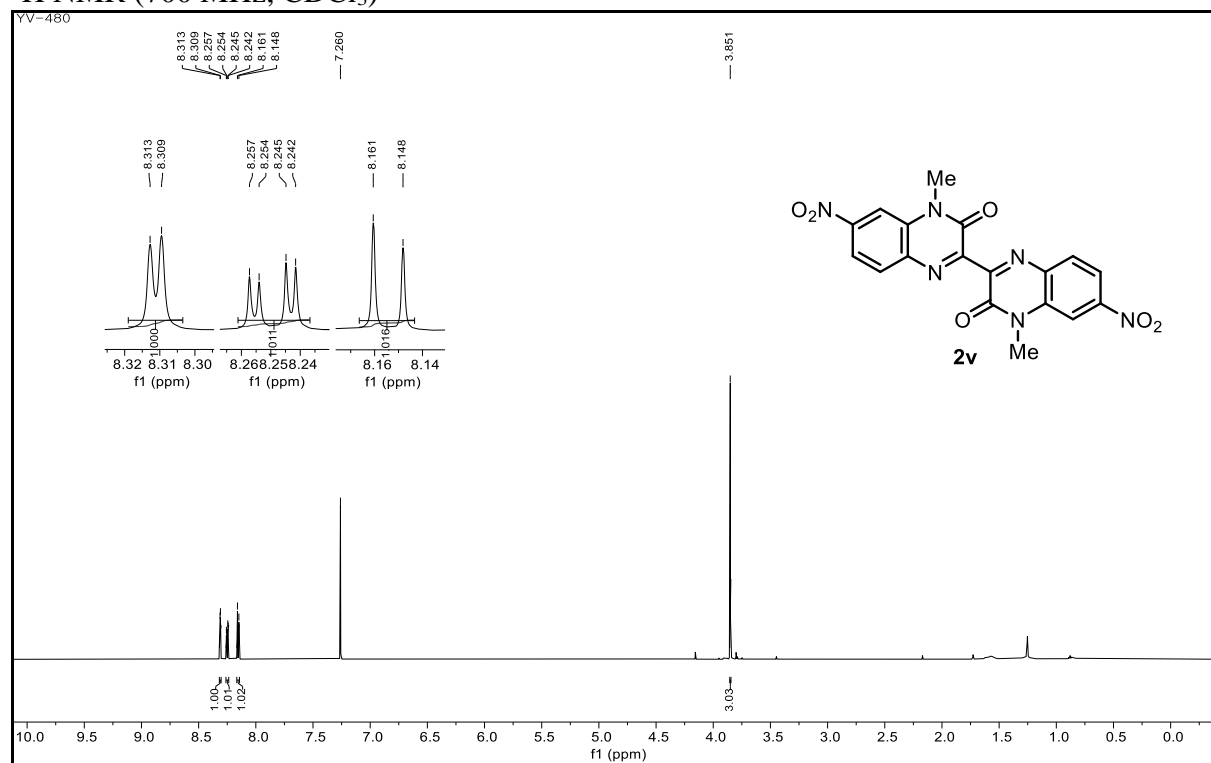
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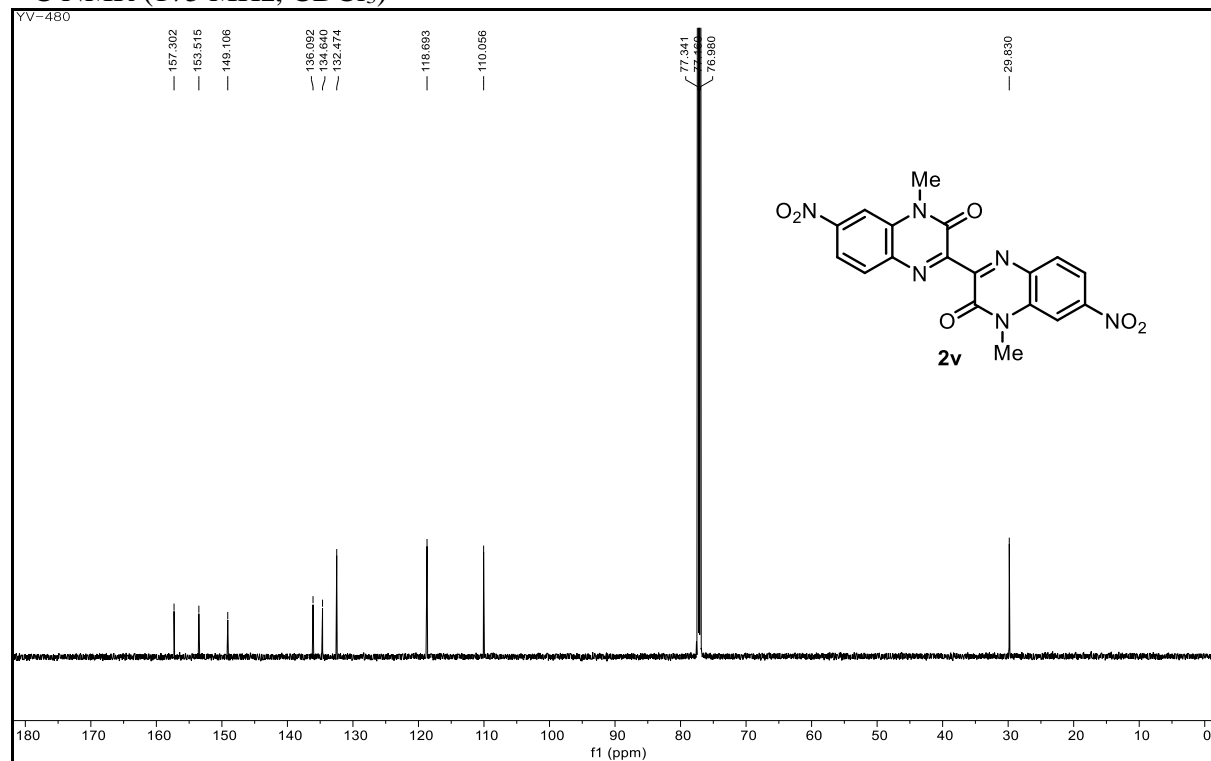
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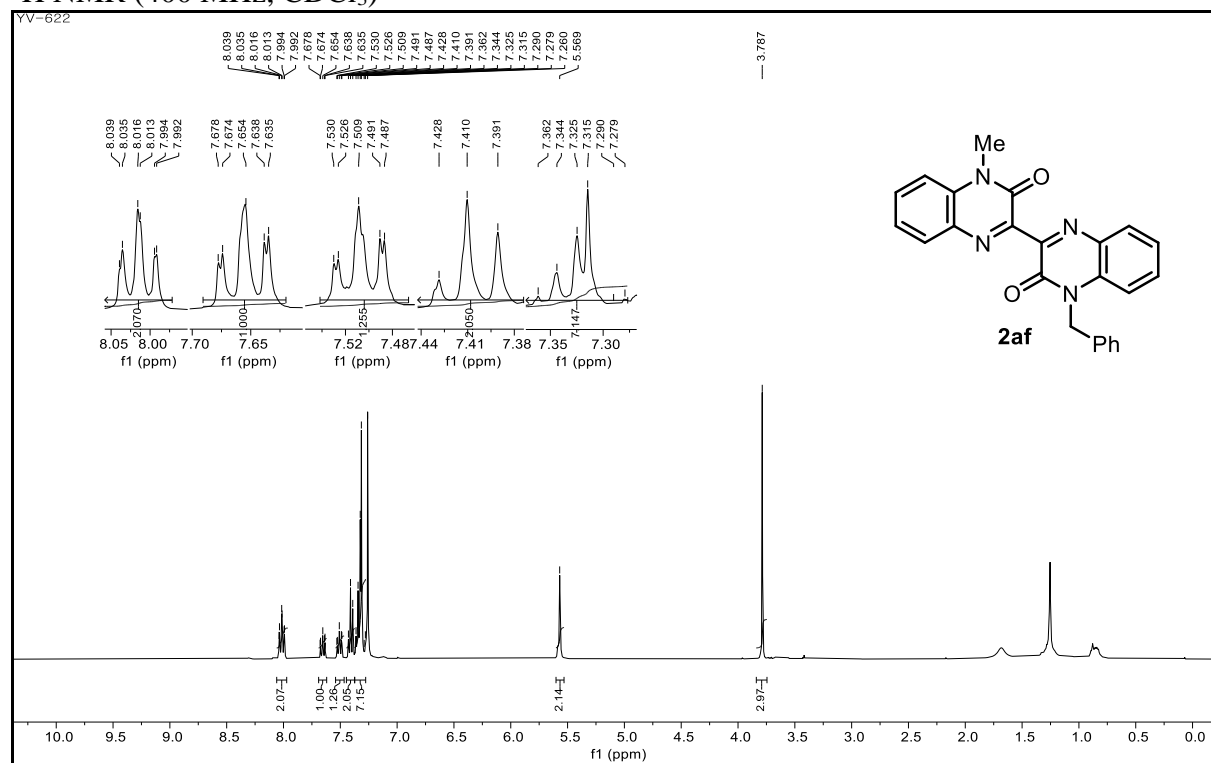
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¹³C NMR (175 MHz, CDCl₃)



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)

