Electronic Supplementary Material (ESI) for ChemComm.

Electronic Supporting Information

Ruthenium-catalyzed free amine directed (5+1) annulation of anilines with olefins: diverse synthesis of phenanthridine derivatives

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General Information

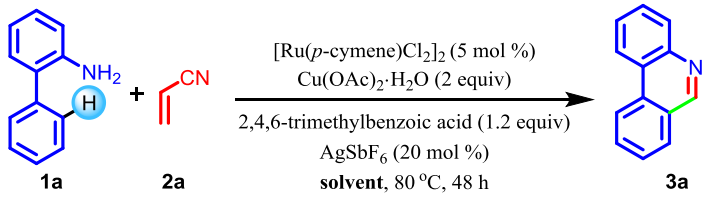
All reactions, unless mentioned otherwise were carried out under argon atmosphere in flame-dried glassware using anhydrous solvents and were stirred using a magnetic stir plate. DMSO, toluene, xylene, chlorobenzene, and DCE were dried using standard procedure. Dry DMF and TFE were purchased from Spectrochem. Tetrahydrofuran and 1,4-dioxane were freshly distilled over sodium ketyl before use. Catalyst $[Ru(p\text{-cymene})Cl_2]_2$ purchased from Alfa Aesar Company. 2-Aminobiaryls were prepared following literature procedures (*J. Org. Chem.* **2018**,*83*, 3840)[S1].

All reactions were monitored by thin layer chromatography (TLC) on Whatman Partisil® K6F TLC plates (silica gel 60 Å, 0.25 mm thickness) and visualized using a UV lamp (366 or 254 nm) or by use of one of the following visualization reagents: 0.75 g potassium permanganate, 5 g K_2CO_3 / 100mL water or iodine chamber. Products were isolated by column chromatography (Merck silica gel 100-200μm). Yields refer to chromatographically and spectroscopically homogenous materials unless noted otherwise. ^1H and ^{13}C NMR spectra were recorded on a Bruker 400 or Bruker 500 MHz spectrometers. Chemical shift values (δ) are reported in ppm and calibrated to the residual solvent peak $CDCl_3$ δ = 7.26 ppm for ^1H, δ = 77.16 ppm for ^{13}C, DMSO-d_6 δ = 2.50 ppm for ^1H, δ = 39.50 ppm for ^{13}C or calibrated to tetramethylsilane (δ = 0.00). All NMR spectra were recorded at ambient temperature (290 K) unless otherwise noted. ^1H NMR spectra are reported as follows: chemical shift (multiplicity, coupling constant, integration). The following abbreviations are used to indicate multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sext, sextet; sept, septet; m, multiplet; dd, doublet of doublet; dt, doublet of triplet; dq, doublet of quartet; td, triplet of doublet; tt, triplet of triplet; dq, doublet of quartet; br, broad; app, apparent.

Mass spectra were recorded by electron spry ionization (ESI) method on a Q-TOF Micro with lock spray source. Melting point of the synthesized compounds were measured by Guna 130 W melting point apparatus.

Screening of the Reaction Conditions:

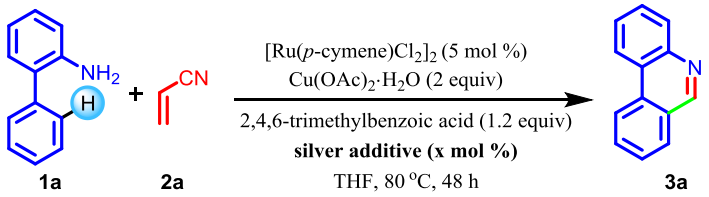
Table S1: Optimization of **Solvent**



entry	solvent	yield (%)[b]
1	toluene	NR
2	xylene	NR
3	chlorobenzene	NR
4	DMF	NR
5	DMSO	NR
6	methanol	NR
7	TFE	NR
8	DCE	32
9	^{t}amyl alcohol	NR
10	DME	38
11	dioxane	46
12	2-Me THF	37
13	**THF**	**72**
14	NMP	NR
15	acetic acid	NR

Reaction conditions: **1a** (0.3mmol), **2a** (0.45mmol), solvent (4.2 mL) for 48 h under argon atmosphere. [b]Isolated yields.

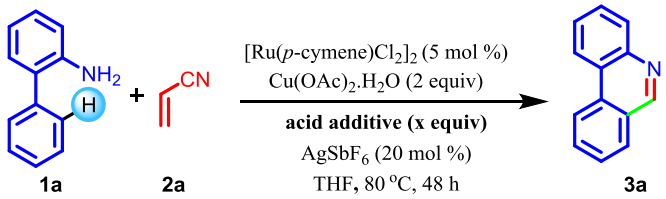
Table S2: Optimization of **Silver Additive**



entry	silver additive	yield (%)[b]
1	**$AgSbF_6$**(20 mol %)	**72**
2	$AgBF_4$(20 mol %)	16
3	AgOTf(20 mol %)	NR
4	AgOAc(20 mol %)	NR
5	KPF_6(20 mol %)	NR
6	$AgSbF_6$ (30 mol %)	52
7	$AgSbF_6$ (50 mol %)	46

Reaction conditions: **1a** (0.3mmol), **2a** (0.45mmol), solvent (4.2 mL) for 48 h under argon atmosphere. [b]Isolated yields.

Table S3: Optimization of **Acid Additive**

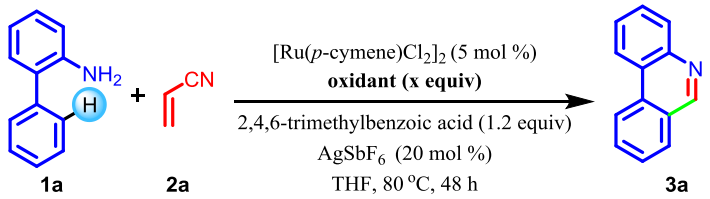


entry	acid additive	yield (%)[b]
1	acetic acid(1.2 equiv)	52
2	pivalic acid(1.2 equiv)	N.R.
3	1-AdCO_2H(1.2 equiv)	12
4	**2,4,6-trimethylbenzoicacid**(1.2equiv)	**72**
5	3,5-dimethylbenzoicacid(1.2 equiv)	16
6	4-nitrobenzoicacid(1.2 equiv)	N.R.

7	2,4,6-trimethylbenzoicacid(2equiv)	N.R.
8	2,4,6-trimethylbenzoicacid(0.5equiv)	35

Reaction conditions: **1a** (0.3mmol), **2a** (0.45mmol), solvent (4.2 mL) for 48 h under argon atmosphere. [b]Isolated yields.

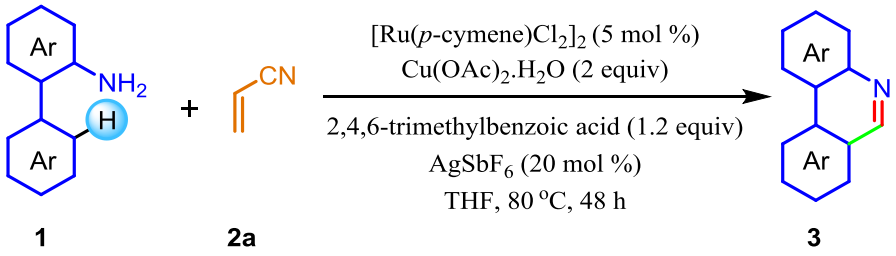
Table S4: Optimization of **Oxidant**



entry	oxidant	yield (%)[b]
1	**$Cu(OAc)_2·H_2O$ (2equiv)**	**72%**
2	CuO(2equiv)	11%
3	Ag_2CO_3(2equiv)	NR
4	$Mn(OAc)_3·4H_2O$(2equiv)	NR
5	$K_2S_2O_8$(2equiv)	NR
6	$(NH_4)_2S_2O_8$(2equiv)	NR
7	$Cu(OAc)_2·H_2O$ (1.2 equiv)	16
8	$Cu(OAc)_2·H_2O$ (0.5 equiv)	NR
9	$Cu(OAc)_2·H_2O$ (3 equiv)	65

Reaction conditions: **1a** (0.3mmol), **2a** (0.45mmol), solvent (4.2 mL) for 48 h under argon atmosphere. [b]Isolated yields.

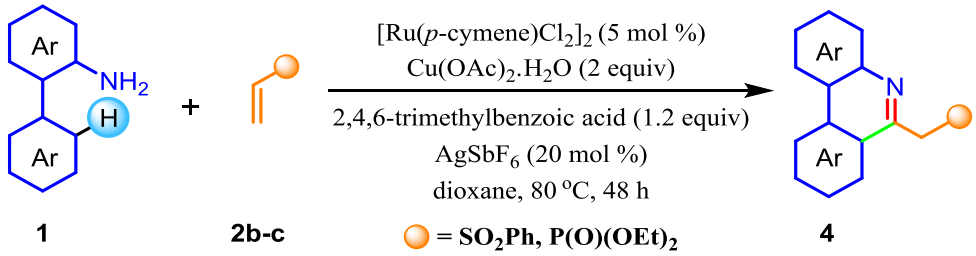
General Procedure for Ru(II)-Catalyzed (5+1) Annulation of 2-Aminobiaryls with Acrylonitrile:



Inside an argon filled glove-box, 2-aminobiaryls**1** (0.3 mmol), acrylonitrile **2a** (1.5equiv), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol %), 2,4,6-trimethylbenzoicacid (1.2 equiv), silver hexafluoroantimonate (20 mol %)

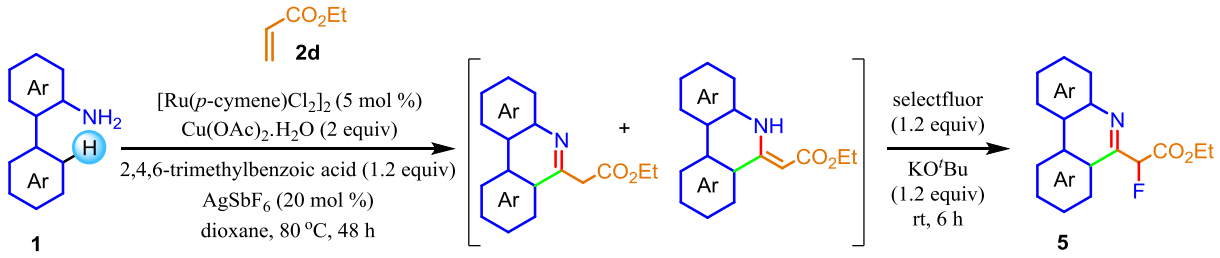
and $Cu(OAc)_2 \cdot H_2O$ (2 equiv) were weighed in an oven dried screw cap reaction tube with a magnetic stir bar. Then, freshly distilled dry THF (4.2 mL) was added with a syringe outside the glove-box under the flow of argon. The reaction tube was capped and the resulting mixture was heated at 80 ºC for 48 h. After completion of the reaction (monitored by TLC), it was allowed to cool to room temperature. The reaction mixture was diluted with ethyl acetate, washed with saturated sodium bicarbonate solution, and dried over anhydrous Na_2SO_4.After evaporation under reduced pressure, the resulting residue was purified by silica gel column chromatography (hexane : ethyl acetate = 85:15 to 70:30) to yield purephenanthridines**3**.

General Procedure for Ru(II)-Catalyzed (5+1) Annulation of 2-Aminobiaryls with Vinyl Sulfone/Vinyl Phosphonate:



Inside an argon filled glove-box, 2-aminobiaryls**1** (0.3mmol), phenyl vinyl sulfone**2b**(1.2 equiv) or diethylvinylphosphonate **2c** (1.2 equiv), $[Ru(p\text{-cymene})Cl_2]_2$ (5 mol %), 2,4,6-trimethylbenzoicacid (1.2 equiv), silver hexafluoroantimonate (20 mol %) and $Cu(OAc)_2 \cdot H_2O$ (2 equiv) were weighed in an oven dried screw cap reaction tube with a magnetic stir bar. Then, freshly distilled dry dioxane (4.2 mL) was added with a syringe outside glove-box under the flow of argon. The reaction tube was capped and the resulting mixture was heated at 80 ºC for 48 h. After completion of the reaction (monitored by TLC), it was allowed to cool to room temperature. The reaction mixture was extracted with ethyl acetate, the organic layers washed with saturated sodium bicarbonate solution and dried over anhydrous Na_2SO_4.After evaporation under reduced pressure, the resulting residue was purified by silica gel column chromatography to yield pure 6-substituted phenanthridines **4**.

General Procedure for Ru(II)-Catalyzed (5+1) Annulation of 2-Aminobiaryls with Ethyl Acrylate and Subsequent Fluorination:

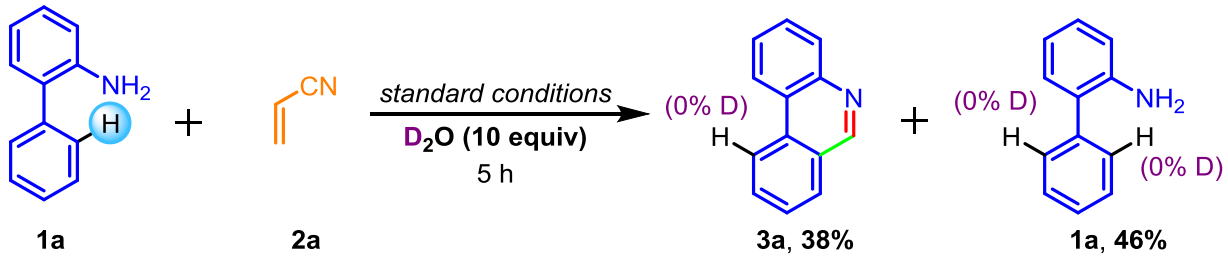


Inside an argon filled glove-box, 2-aminobiaryls **1** (0.3 mmol), ethyl acrylate **2d**(1.2 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol %), 2,4,6-trimethylbenzoicacid (1.2 equiv), silver hexafluoroantimonate (20 mol %) and $Cu(OAc)_2 \cdot H_2O$ (2equiv) were weighed in an oven dried screw cap reaction tube with a magnetic stir bar. Then, freshly distilled dry dioxane (4.2 mL) was added with a syringe outside glove-box under the flow of argon. The reaction tube was capped and the resulting mixture was heated at 80 ºC for 48 h. After completion of the reaction (monitored by TLC), it was allowed to cool to room temperature. The reaction mixture was extracted with ethyl acetate, the organic layers washed with saturated sodium bicarbonate solution and dried over anhydrous Na_2SO_4.After evaporation under reduced pressure, the resulting crude mixture was then exposed toselectfluor (1.2 equiv) and potassium *tert*-butoxide (1.2 equiv) in dry acetonitrile. The reaction mixture was stirred for 6 h at room temperature, the solvent was evaporated and the residue was subjected to column chromatography to yield ethyl 2-fluoro-2-(phenanthridin-6-yl)acetates **5**.

Mechanistic Studies:

- **H/D Exchange Study**

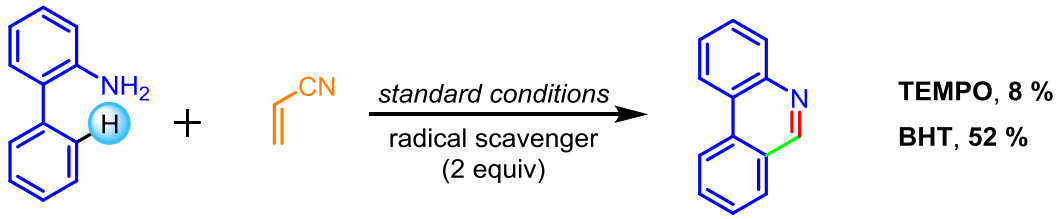
2-Aminobiphenyl **1a** was reacted with acrylonitrile **2a** in presence of D_2O under standard conditions. The reaction was monitored by TLC and stopped after 5 h. Extraction with ethyl acetate, evaporation under vacuum followed by column chromatography gave the phenanthridine product in 38% yield with 0% deuterium incorporation. The starting material was also isolated in 42% yield with 0% deuterium incorporation.



1a **2a** **3a, 38%** **1a, 46%**

- **Radical Trapping Experiments**

2-Aminobiphenyl **1a** was reacted with acrylonitrile **2a** in presence of radical scavengers like TEMPO and BHT (2equiv) under standard conditions. The reaction was monitored by TLC and stopped after 12 h. Extraction with ethyl acetate, evaporation under vacuum followed by column chromatography gave the phenanthridine product **3a** in 8% yield in case of TEMPO. However, in case of BHT the desired product was isolated in 52% yield.



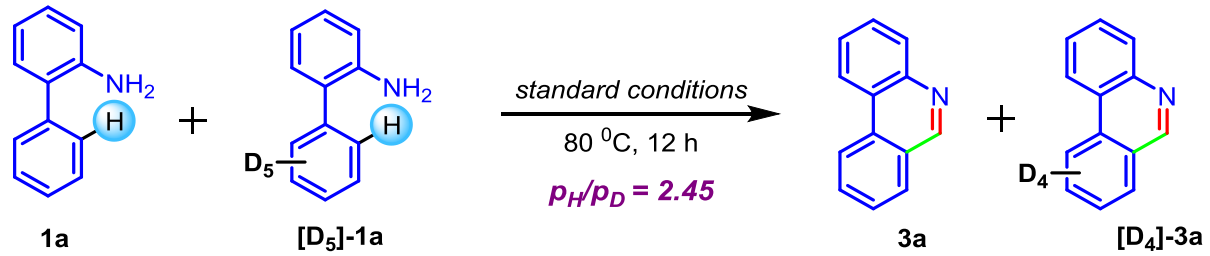
1a **2a** **3a**

TEMPO, 8 %

BHT, 52 %

➢ Kinetic Isotope Experiment Studies

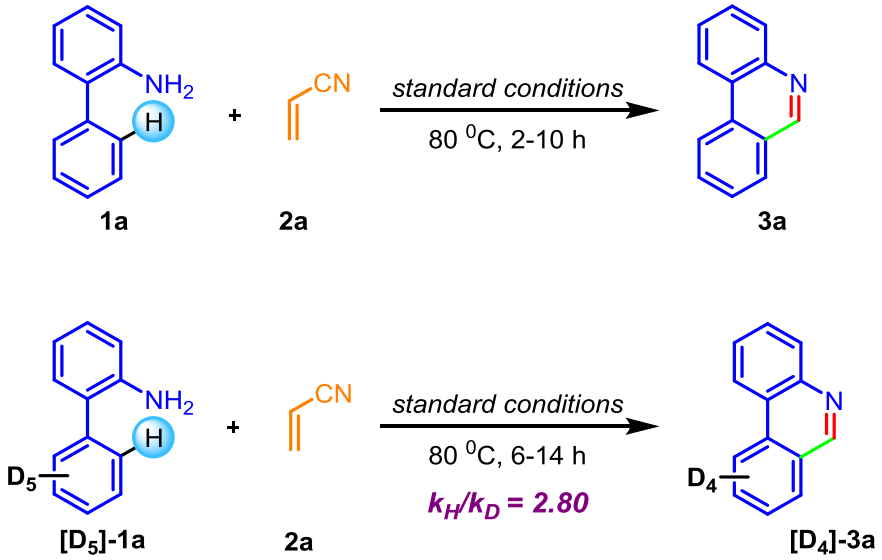
Through competitive experiment



A mixture of 2-aminobiphenyl**1a** and [D_5]-2-aminobiphenyl**[D_5]-1a** (0.15 mmol each) was treated with acrylonitrile**2a** under standard conditions. The crude reaction mixture was isolated via column chromatography to yield a mixture phenanthridine**3a** and D_4-phenanthridine**[D_4]-3a**. ^{1}H NMR analysis revealed a kinetic isotope effect value of 2.45 from the integration ratios.

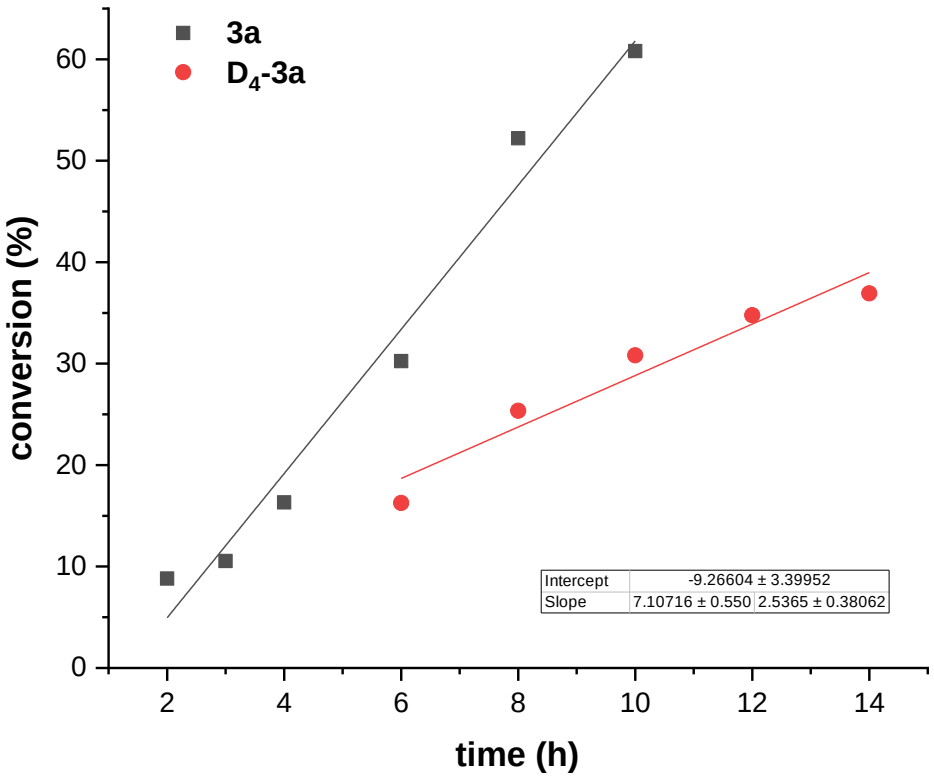
Through Parallel Experiment

2-Aminobiphenyl and [D_5]-2-aminobiphenyl (0.3 mmol each) were weighed in separate reaction tubes and reacted with acrylonitrile**2a** under standard conditions. 100 µl of aliquot was withdrawn from the two reaction mixtures at definite time intervals, filtered through neutral alumina, evaporated under vacuum and then subjected to GC-MS analysis. The data was plotted and the slope obtained revealed a kinetic isotope effect value of 2.80.



Based on GC-MS analysis the conversion was plotted against time for two cases, and slope was calculated for each instance. The ratio of two slopes gave the k_H/k_D value.

Phenanthridine, **3a** Retention time, 14.189		D_4-Phenanthridine, **D_4-3a** Retention time, 14.210	
Conversion (%)	Time (h)	Conversion (%)	Time (h)
8.82	2	16.26	6
10.54	3	25.36	8
16.32	4	30.82	10
30.24	6	34.77	12
52.21	8	36.92	14
60.81	10		



Slope for **3a** = 7.1071 ; Slope for **D_4-3a** = 2.5365. k_H/k_D = 7.1071/2.5365 = **2.801**.

Plausible Mechanism:

Based on the control experiments and literature precedents, we propose the following mechanism for our protocol which is schematically represented below. (Fig: S1)

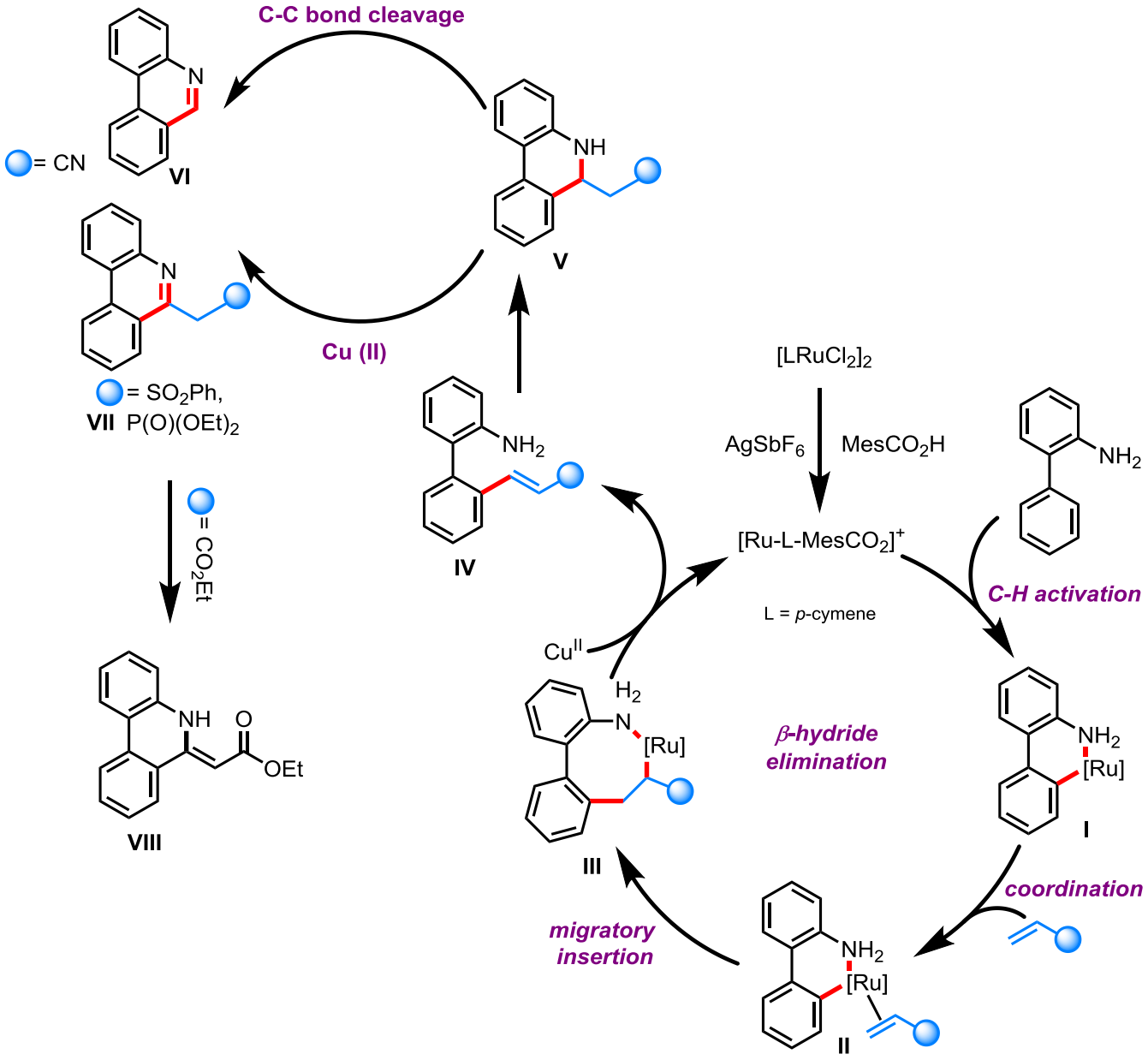


Figure: S1

Analytical Data:

Compounds (**3a**, **3j**, **3m**,**3o**,**3p**, **3q**, **7**, **8)**[S2]**, (3b**, **3e**, **3h**,**3r**, **3t, 3u)**[S3]**,(3d**, **3n)**[S4]**, (3g)**[S5]**, (3k)**[S6]**, (3s)**[S7] **and (4a)**[S8] are known in literature and thus only **^{1}H NMR** and **HRMS** data are provided for these compounds.

References:

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(S4) J. Tummatorn, S. Krajangsri, K. Norseeda, C. Thongsornkleeb and S. Ruchirawat, *Org. Biomol. Chem.*, 2014, **12**, 5077.

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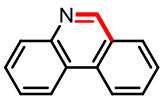
(S6) T. Gupta, J. B. Singh, K. Mishra, B. Maiti and R. M. Singh, *European J. Org. Chem.*, 2018, **2018**, 1130.

(S7) S. Y. Yang, W. Y. Han, D. L. Zhang, X. J. Zhou, M. Bai, B. D. Cui, N. W. Wan, W. C. Yuan and Y. Z. Chen, *European J. Org. Chem.*, 2017, **2017**, 996–1003.

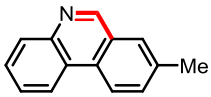
(S8) J. Tang, P. Sivaguru, Y. Ning, G. Zanoni and X. Bi, *Org. Lett.*, 2017, **19**, 4026.

Spectroscopic Data for:



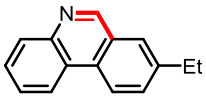
3a

Phenanthridine, **3a**: White solid, eluent (10% ethyl acetate in hexane). Yield: 72% (39 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.29 (s, 1H), 8.68 – 8.51 (m, 2H), 8.25 – 8.15 (m, 1H), 8.04 (d, *J* = 7.9 Hz, 1H), 7.85 (dd, *J* = 11.3, 4.1 Hz, 1H), 7.81 – 7.65 (m, 3H) ppm; **HRMS** (TOF MS ES+) calcd. for $C_{13}H_9NH^+$ [M + H^+] m/z 180.0813, found 180.0814.



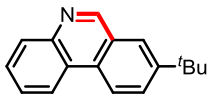
3b

8-Methylphenanthridine, **3b**: Yellow solid, eluent (10% ethyl acetate in hexane). Yield: 79% (46 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.27 (s, 1H), 8.67 – 8.42 (m, 2H), 8.26 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.88 (s, 1H), 7.81 – 7.65 (m, 3H), 2.62 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_{11}NH^+$ [M + H^+] m/z 194.0970, found 194.0976.



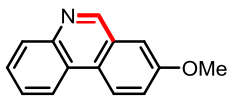
3c

8-Ethylphenanthridine, **3c**: Brown oil, eluent (12% ethyl acetate in hexane). Yield: 81% (50 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.26 (s, 1H), 8.52 (dd, *J* = 13.2, 8.3 Hz, 2H), 8.18 (d, *J* = 7.9 Hz, 1H), 7.80 (d, *J* = 21.3 Hz, 1H), 7.77 – 7.54 (m, 3H), 2.89 (q, *J* = 7.6 Hz, 2H), 1.37 (t, *J* = 7.6 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.6, 144.3, 144.0, 131.9, 130.8, 130.1, 128.3, 127.1, 127.0, 126.8, 124.4, 122.2, 122.0, 28.9, 15.5; **HRMS** (TOF MS ES+) calcd. for $C_{15}H_{13}NH^+$ [M + H^+] m/z 208.1126, found 208.1141.



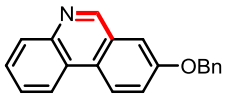
3d

8-Tert-butylphenanthridine, **3d**: Brown oil, eluent (7% ethyl acetate in hexane). Yield: 84% (59.8 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.28 (s, 1H), 8.55 (dd, *J* = 8.4, 3.8 Hz, 2H), 8.27 – 8.11 (m, 1H), 8.00 (d, *J* = 2.2 Hz, 1H), 7.97 – 7.92 (m, 1H), 7.76 – 7.63 (m, 2H), 1.48 (s, 9H); **HRMS** (TOF MS ES+) calcd. for $C_{17}H_{17}NH^+$ [M + H^+] m/z 236.1439, found 236.1433.



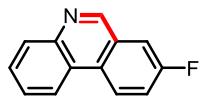
3e

8-Methoxyphenanthridine, **3e:** Yellow solid, eluent (16% ethyl acetate in hexane). Yield: 75% (47.1 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.24 (s, 1H), 8.51 (dd, *J* = 12.8, 5.2 Hz, 2H), 8.22 – 8.12 (m, 1H), 7.74 – 7.61 (m, 2H), 7.50 (dd, *J* = 9.0, 2.8 Hz, 1H), 7.40 (d, *J* = 2.7 Hz, 1H), 4.00 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_{11}NOH^+$ [M + H^+] m/z 210.0919, found 210.0919.



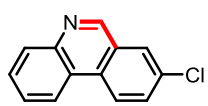
3f

8-(Benzyloxy)phenanthridine, **3f**: White solid, eluent (15% ethyl acetate in hexane). Yield: 80% (68.6 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.56 – 8.40 (m, 2H), 8.17 (d, *J* = 7.4 Hz, 1H), 7.74 – 7.59 (m, 2H), 7.58 – 7.49 (m, 3H), 7.47 – 7.41 (m, *J* = 9.2, 5.3 Hz, 3H), 7.40 – 7.34 (m, 1H), 5.23 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 152.9, 143.7, 136.4, 130.1, 128.8, 128.4, 127.9, 127.8, 127.7, 127.3, 127.2, 124.3, 123.8, 122.6, 121.8, 109.4, 77.5, 77.2, 76.8, 70.4; **HRMS** (TOF MS ES+) calcd. for $C_{20}H_{15}NOH^+$ [M + H^+] m/z 286.1232, found 286.1234.



3g

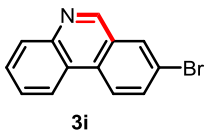
8-Fluorophenanthridine, **3g**: White solid, eluent (12% ethyl acetate in hexane). Yield: 73% (46.8 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.24 (s, 1H), 8.62 (dd, *J* = 8.9, 5.0 Hz, 1H), 8.53 (d, *J* = 8.0 Hz, 1H), 8.20 (d, *J* = 8.0 Hz, 1H), 7.73 (dt, *J* = 16.6, 7.8 Hz, 3H), 7.62 (t, *J* = 8.7 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for$C_{13}H_8FNH^+$ [M + H^+] m/z 198.0719, found 198.0719.



3h

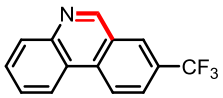
8-Chlorophenanthridine, **3h**: White solid, eluent (12% ethyl acetate in hexane). Yield: 78% (50 mg); **^{1}H NMR**(400 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.54 – 8.47 (m, 2H), 8.24 – 8.15 (m,

1H), 7.99 (t, *J* = 5.2 Hz, 1H), 7.78 (tdd, *J* = 12.2, 7.7, 4.8 Hz, 2H), 7.68 (dd, *J* = 15.8, 8.5 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{13}H_8ClNH^+$ [M + H^+] m/z 214.0424, found 214.0428.



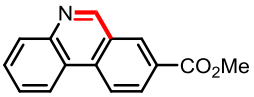
3i

8-Bromophenanthridine, **3i**: White solid, eluent (13% ethyl acetate in hexane), mp: 68-72 ^oC. Yield: 68% (43.6 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.51 (dd, *J* = 8.8, 7.7 Hz, 1H), 8.47 (d, *J* = 8.7 Hz, 1H), 8.25 – 8.13 (m, 2H), 7.93 (dd, *J* = 8.8, 2.2 Hz, 1H), 7.78 (ddd, *J* = 8.1, 7.1, 1.4 Hz, 1H), 7.70 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 152.3, 144.5, 134.3, 132.4, 131.4, 131.1, 130.5, 129.3, 127.7, 124.0, 123.7, 122.2, 121.4; **HRMS** (TOF MS ES+) calcd. for$C_{13}H_8BrNH^+$[M + H^+] m/z 257.9918, found 257.9923.



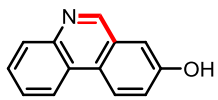
3j

8-(Trifluoromethyl)phenanthridine, **3j**:White solid, eluent (8% ethyl acetate in hexane). Yield: 63% (40 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.32 (s, 1H), 8.66 (dd, *J* = 7.7, 4.2 Hz, 1H), 8.55 (d, *J* = 4.5 Hz, 1H), 8.31 (s, 1H), 8.22 (d, *J* = 7.6 Hz, 1H), 8.02 (d, *J* = 8.5 Hz, 1H), 7.81 (t, *J* = 7.3 Hz, 1H), 7.72 (t, *J* = 7.2 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_8F_3NH^+$[M + H^+] m/z 248.0687, found 248.0687.



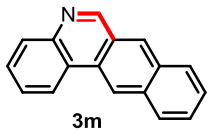
3k

Methyl phenanthridine-8-carboxylate, **3k:** Yellow solid, eluent (15% ethyl acetate in hexane). Yield: 58% (31.1 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.34 (s, 1H), 8.75 (s, 1H), 8.63 (d, *J* = 8.6 Hz, 1H), 8.58 (d, *J* = 8.2 Hz, 1H), 8.44 (dd, *J* = 8.6, 1.6 Hz, 1H), 8.21 (d, *J* = 8.2 Hz, 1H), 7.80 (t, *J* = 7.3 Hz, 1H), 7.71 (t, *J* = 7.6 Hz, 1H), 4.02 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{15}H_{11}NO_2H^+$ [M + H^+] m/z 238.0868, found 238.0876.



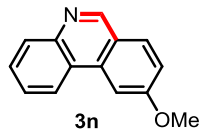
3l

8-Hydroxyphenanthridine, **3l**: White solid, eluent (30% ethyl acetate in hexane), mp: 114-118^{o}C. Yield: 70% (44.9 mg); **^{1}H NMR** (400 MHz, DMSO) δ 10.24 (s, 1H), 9.20 (s, 1H), 8.65 (t, *J* = 9.0 Hz, 2H), 8.02 (dd, *J* = 6.0, 3.4 Hz, 1H), 7.66 (dd, *J* = 6.0, 3.3 Hz, 2H), 7.53 – 7.35 (m, 2H); **^{13}C NMR** (101 MHz, DMSO) δ 157.1, 152.8, 142.9, 129.5, 127.8, 127.3, 127.1, 124.8, 124.1, 124.0, 122.1, 122.0, 111.1; **HRMS** (TOF MS ES+) calcd. for $C_{13}H_9NOH^+$ [M + H^+] m/z 196.0762 found 196.0763.



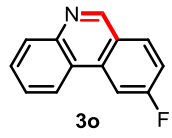
3m

Benzo[*j*]phenanthridine, **3m**: Yellow solid, eluent (14% ethyl acetate in hexane). Yield: 72% (49.84 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.42 (s, 1H), 9.05 (s, 1H), 8.71 (dd, *J* = 7.3, 1.9 Hz, 1H), 8.60 (s, 1H), 8.21 (d, *J* = 7.5 Hz, 1H), 8.14 (t, *J* = 7.5 Hz, 2H), 7.72 (ddd, *J* = 20.9, 13.9, 7.5 Hz, 3H), 7.63 (t, *J* = 7.5 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{17}H_{11}NH^+$ [M + H^+] m/z 230.0970 found 230.0978.



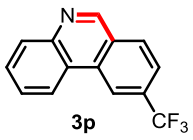
3n

9-Methoxyphenanthridine, **3n**: Brown solid, eluent (16% ethyl acetate in hexane). Yield: 68% (37.4 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.17 (s, 1H), 8.50 (d, *J* = 8.1 Hz, 1H), 8.17 (d, *J* = 8.1 Hz, 1H), 7.96 (d, *J* = 8.7 Hz, 1H), 7.91 (d, *J* = 0.9 Hz, 1H), 7.74 (t, *J* = 7.6 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.31 (dd, *J* = 8.7, 1.5 Hz, 1H), 4.04 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_{11}NOH^+$ [M + H^+] m/z 210.0919, found 210.0919.



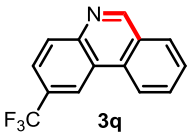
3o

9-Fluorophenanthridine, **3o**: Yellow solid, eluent (12% ethyl acetate in hexane). Yield: 65% (38.6 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.26 (s, 1H), 8.47 (dd, *J* = 8.2, 1.3 Hz, 1H), 8.25 – 8.17 (m, 2H), 8.08 (dd, *J* = 8.7, 5.7 Hz, 1H), 7.82 – 7.75 (m, 1H), 7.76 – 7.66 (m, 1H), 7.47 (dd, *J* = 8.2, 2.4 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{13}H_8FNH^+$[M + H^+] m/z 198.0719, found 198.0719.



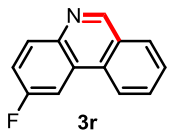
3p

9-(Trifluoromethyl)phenanthridine, **3p:** White solid, eluent (8% ethyl acetate in hexane). Yield: 71% (53 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.35 (s, 1H), 8.88 (s, 1H), 8.61 (dd, *J* = 8.2, 1.1 Hz, 1H), 8.24 (dt, *J* = 11.9, 5.9 Hz, 1H), 8.18 (d, *J* = 8.3 Hz, 1H), 7.92 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.86 – 7.79 (m, 1H), 7.79 – 7.72 (m, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_8F_3NH^+$[M + H^+] m/z 248.0687, found 248.0687.



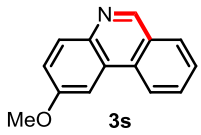
3q

2-(Trifluoromethyl)phenanthridine, **3q**: White solid, eluent (8% ethyl acetate in hexane). Yield: 75% (45.2 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.40 (s, 1H), 8.85 (s, 1H), 8.64 (dd, *J* = 8.2, 1.8 Hz, 1H), 8.34 (d, *J* = 8.6 Hz, 1H), 8.13 (d, *J* = 8.0 Hz, 1H), 8.01 – 7.91 (m, 2H), 7.81 (t, *J* = 7.5 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_8F_3NH^+$[M + H^+] m/z 248.0687, found 248.0687.



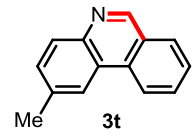
3r

2-Fluorophenanthridine, **3r**: Orange solid, eluent (10% ethyl acetate in hexane). Yield: 66% (39 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.24 (s, 1H), 8.47 (d, *J* = 8.3 Hz, 1H), 8.29 – 8.11 (m, 2H), 8.05 (d, *J* = 7.9 Hz, 1H), 7.86 (t, *J* = 7.6 Hz, 1H), 7.74 (t, *J* = 7.5 Hz, 1H), 7.47 (td, *J* = 8.5, 2.7 Hz, 1H); **HRMS** (TOF MS ES+) calcd. for $C_{13}H_8FNH^+$ [M + H^+] m/z 198.0719, found 198.0719.



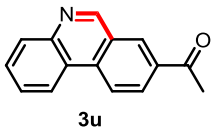
3s

2-Methoxyphenanthridine, **3s**: Grey solid, eluent (12% ethyl acetate in hexane). Yield: 70 % (54 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.17 (s, 1H), 8.53 (d, *J* = 8.3 Hz, 1H), 8.13 (t, *J* = 20.5 Hz, 1H), 8.05 (d, *J* = 7.9 Hz, 1H), 7.97 – 7.78 (m, 2H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.38 (dd, *J* = 8.9, 2.9 Hz, 1H), 4.02 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_{11}NOH^+$ [M + H^+] m/z 210.0919, found 210.0919.



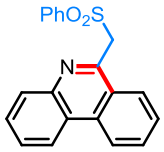
3t

2-Methyl phenanthridine, **3t:** Yellow solid, eluent (10% ethyl acetate in hexane). Yield: 62 % (36.1 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.22 (s, 1H), 8.59 (d, *J* = 8.3 Hz, 1H), 8.35 (s, 1H), 8.09 (d, *J* = 8.3 Hz, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.87 – 7.80 (m, 1H), 7.73 – 7.66 (m, 1H), 7.57 (dd, *J* = 8.3, 1.7 Hz, 1H), 2.63 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{14}H_{11}NH^+$ [M + H^+] m/z 194.0970, found 194.0976.



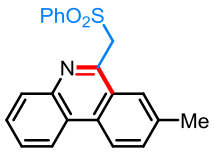
3u

1-(Phenanthridin-8-yl)ethanone, 3u: White solid, eluent (17% ethyl acetate in hexane). Yield 36 % (24 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.38 (s, 1H), 8.65 (m, 3H), 8.42 (d, *J* = 8.0 Hz, 1H), 8.23 (d, *J* = 7.8 Hz, 1H), 7.82 (d, *J* = 6.6 Hz, 1H), 7.75 (d, *J* = 7.1 Hz, 1H), 2.79 (s, 3H); **HRMS** (TOF MS ES+) calcd. for $C_{15}H_{11}NOH^+$ [M + H^+] m/z 221.0841, found 221.0839.



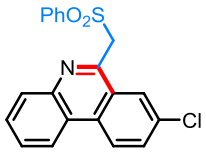
4a

6-((Phenylsulfonyl)methyl)phenanthridine, **4a**:White solid, eluent (15% ethyl acetate in hexane). Yield: 75% (75 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.3 Hz, 1H), 8.44 (dd, *J* = 5.6, 3.4 Hz, 1H), 8.26 (d, *J* = 8.2 Hz, 1H), 7.77 (t, *J* = 7.2 Hz, 2H), 7.60 (ddd, *J* = 10.6, 9.2, 5.5 Hz, 5H), 7.46 (t, *J* = 7.3 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 2H), 5.15 (s, 2H); **HRMS** (TOF MS ES+) calcd. for $C_{20}H_{15}NO_2SH^+$ [M + H^+] m/z 334.0902, found 334.0891.



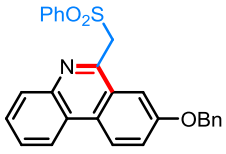
4b

8-Methyl-6-((phenylsulfonyl)methyl)phenanthridine, **4b**:White solid, eluent (17% ethyl acetate in hexane), mp: 168-172 °C.Yield: 84% (87.5 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.51 (t, *J* = 7.4 Hz, 2H), 8.05 (s, 1H), 7.80 – 7.74 (m, 1H), 7.71 – 7.60 (m, 5H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 5.16 (s, 2H), 2.60 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.4, 143.1, 138.5, 137.9, 133.8, 132.9, 131.2, 130.0, 128.9 (2C), 128.4, 127.8, 126.4, 125.8, 124.2, 122.4, 121.9, 62.6, 22.0; **HRMS** (TOF MS ES+) calcd. For $C_{21}H_{17}NO_2SH^+$[M + H^+] m/z 348.1058, found 348.1065.



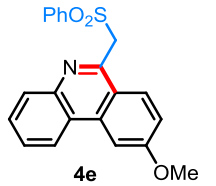
4c

8-Chloro-6-((phenylsulfonyl)methyl)phenanthridine,**4c**:White solid, eluent (20% ethyl acetate in hexane), mp: 165-168 °C.Yield: 80% (88.1 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.52 (t, *J* = 9.1 Hz, 1H), 8.50 – 8.44 (m, 1H), 8.22 (s, 1H), 7.82 (dd, *J* = 11.2, 6.3 Hz, 1H), 7.76 (dd, *J* = 10.7, 9.4 Hz, 1H), 7.68 (m, 4H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 5.10 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.7, 143.3, 138.4, 134.0, 133.9, 131.7, 130.2, 129.2, 129.1 (2C), 128.8 (2C), 128.3, 126.5, 126.3, 124.2, 123.5, 122.0, 62.5; **HRMS** (TOF MS ES+) calcd. for $C_{20}H_{14}ClNO_2SH^+$ [M + H^+] m/z 368.0512, found 368.0519.



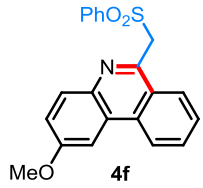
4d

8-(Benzyloxy)-6-((phenylsulfonyl)methyl)phenanthridine,**4d**: White solid, eluent (25% ethyl acetate in hexane, mp: 146-150 °C.Yield: 77% (101.4 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 9.0 Hz, 1H), 8.45 (d, *J* = 7.9 Hz, 1H), 7.81 (d, *J* = 2.4 Hz, 1H), 7.76 (d, *J* = 7.8 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 2H), 7.64 – 7.60 (m, 1H), 7.60 – 7.53 (m, 5H), 7.46 (t, *J* = 7.4 Hz, 2H), 7.39 (m, 3H), 5.29 (s, 2H), 5.12 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.2, 148.9, 142.7, 138.4, 136.4, 133.6, 129.9, 129.0, 128.9, 128.8, 128.4, 128.0, 127.9 (2C), 127.8, 127.0, 124.2, 124.1, 122.7, 121.6, 108.4, 70.7, 63.1; **HRMS**(TOF MS ES+) calcd. for$C_{27}H_{21}NO_3SH^+$ [M + H^+] m/z 440.1320, found440.1329.



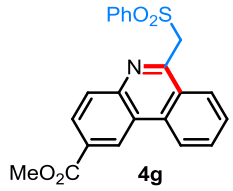
4e

9-Methoxy-6-((phenylsulfonyl)methyl)phenanthridine,**4e**:Brown solid, eluent (25% ethyl acetate in hexane), mp: 156-162 °C.Yield: 73% (79.5 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.45 (d, *J* = 8.6 Hz, 1H), 8.27 (d, *J* = 9.1 Hz, 1H), 7.92 (d, *J* = 1.3 Hz, 1H), 7.76 (d, *J* = 8.7 Hz, 1H), 7.63 (dt, *J* = 11.7, 7.7 Hz, 4H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.33 (d, *J* = 9.9 Hz, 1H), 5.11 (s, 2H), 4.04 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 161.7, 149.1, 143.8, 138.5, 135.6, 133.8, 130.0, 129.1, 129.0, 128.9, 128.8, 127.3, 124.0, 122.1, 120.8, 118.0, 103.2, 62.8, 55.8; **HRMS** (TOF MS ES+) calcd. for $C_{21}H_{17}NO_3SH^+$ [M + H^+] m/z 364.1007, found 364. 1017.



4f

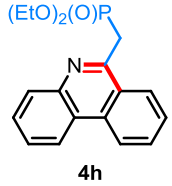
2-Methoxy-6-((phenylsulfonyl)methyl)phenanthridine,**4f**: White solid, eluent (25% ethyl acetate in hexane), mp: 128-132 °C.Yield: 68% (74.1 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.94 (d, *J* = 8.3 Hz, 1H), 8.71 (d, *J* = 8.1 Hz, 1H), 8.23 (dd, *J*= 8.0, 5.6 Hz, 2H), 8.16 – 8.07 (m, 2H), 8.06 (d, *J* = 7.5 Hz, 2H), 7.94 (t, *J* = 7.6 Hz, 1H), 7.77 (t, *J* = 7.6 Hz, 2H), 7.70 – 7.63 (m, 1H), 5.55 (s, 2H), 4.39 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.1, 146.9, 138.7, 138.5, 133.8, 132.8, 131.4, 130.7, 128.9, 128.8, 127.9, 127.1, 125.8, 125.4, 122.4, 119.1, 102.7, 77.5, 77.2, 76.8, 62.4, 55.8; **HRMS** (TOF MS ES+) calcd. for $C_{21}H_{17}NO_3SH^+$ [M + H^+] m/z 364.1007, found 364.1016.



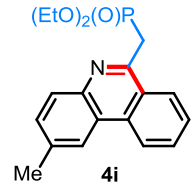
4g

Methyl 6-((phenylsulfonyl)methyl)phenanthridine-2-carboxylate, **4g**: Yellow solid, eluent (22% ethyl acetate in hexane), mp: 184-190 °C.Yield: 57% (67 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.27 (s, 1H), 8.73 (d, *J* = 8.0 Hz, 1H), 8.38 (d, *J* = 8.1 Hz, 1H), 8.25 (d, *J* = 8.4

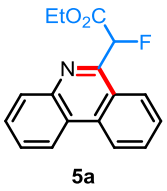
Hz, 1H), 7.99 – 7.89 (m, 1H), 7.86 – 7.74 (m, 2H), 7.72 – 7.63 (m, 2H), 7.61 – 7.51 (m, 1H), 7.46 – 7.31 (m, 2H), 5.18 (s, 2H), 4.02 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.9, 152.3, 145.6, 138.4, 133.9, 133.5, 131.8, 130.2, 129.0, 128.9 (2C), 128.8, 128.5, 127.4, 125.9, 124.9, 123.7, 122.7, 62.8, 52.6; **HRMS** (TOF MS ES+) calcd. for $C_{22}H_{17}NO_4SH^+$ [M + H^+] m/z 392.0957, found 392. 0960.



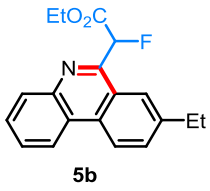
Diethyl ((phenanthridin-6-yl)methyl)phosphonate, **4h:** White solid, eluent (55% ethyl acetate in hexane), mp: 68-72 °C.Yield: 70% (69.1 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.62 (d, *J* = 12.0 Hz, 1H), 8.56 (d, *J* = 8.1 Hz, 1H), 8.37 (d, *J* = 8.2 Hz, 1H), 8.14 (t, *J* = 8.4 Hz, 1H), 7.85 (t, *J* = 7.6 Hz, 1H), 7.72 (dd, *J* = 14.6, 6.9 Hz, 2H), 7.65 (t, *J* = 7.5 Hz, 1H), 4.27 – 3.88 (m, 6H), 1.21 (t, *J* = 7.0 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.8 (d, *J* = 9.2 Hz), 143.5, 133.2, 131.0, 129.6, 128.9, 127.6, 127.5, 127.1, 125.6, 124.0, 122.4, 122.1, 62.6 (d, *J* = 6.5 Hz), 35.2 (d, *J* = 134.6 Hz), 16.4 (d, *J* = 6.2 Hz); **HRMS** (TOF MS ES+) calcd. for $C_{18}H_{20}NO_3PH^+$ [M + H^+] m/z 330.1259, found 330.1241.



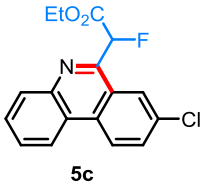
Diethyl((2-methyl-phenanthridin-6-yl)methyl)phosphonate, **4i:** Sticky black solid, eluent (50% ethyl acetate in hexane). Yield: 75% (77.2 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 8.3 Hz, 1H), 8.35 (d, *J* = 8.2 Hz, 2H), 8.02 (d, *J* = 8.4 Hz, 1H), 7.89 – 7.78 (m, 1H), 7.71 (dd, *J* = 9.2, 5.3 Hz, 1H), 7.62 – 7.48 (m, 1H), 4.13 – 4.05 (m, 4H), 4.06 – 3.98 (m, 2H), 2.62 (s, 3H), 1.20 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.7 (d, *J* = 9.1 Hz), 142., 137.0, 133.0, 130.7, 130.6, 129.5, 127.5, 127.4, 125.7, 123.9, 122.3, 121.8, 62.6 (d, *J* = 6.6 Hz), 35.2 (d, *J* = 134.5 Hz), 22.1, 16.4 (d, *J* = 6.3 Hz); **HRMS** (TOF MS ES+) calcd. for $C_{19}H_{22}NO_3PH^+$ [M + H^+] m/z 344.1416, found 344.1421.



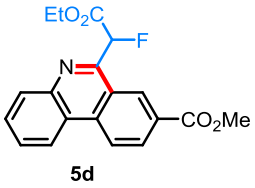
Ethyl 2-fluoro-2-(phenanthridin-6-yl)acetate, **5a:** White solid, eluent (5% ethyl acetate in hexane), mp: 97-101 °C. Yield: 77% (65.4 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.71 (d, *J* = 8.3 Hz, 1H), 8.62 (dd, *J* = 8.0, 1.4 Hz, 1H), 8.46 – 8.37 (m, 1H), 8.22 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.98 – 7.87 (m, 1H), 7.83 – 7.70 (m, 3H), 6.52 (d, *J* = 48.0 Hz, 1H), 4.43 – 4.18 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H) ; **^{13}C NMR** (126 MHz, $CDCl_3$) δ 168.2 (d, *J* = 22.7 Hz), 153.1 (d, *J* = 20.3 Hz), 143.1, 133.7, 131.2, 130.8, 129.1, 128.3, 128.0, 126.1 (d, *J* = 4.4 Hz), 124.7, 124.2, 122.7, 122.2, 91.5 (d, *J* = 187.4 Hz), 62.3, 14.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -180.7; **HRMS** (TOF MS ES+) calcd. for$C_{17}H_{14}FNO_2H^+$ [M + H^+] m/z 284.1087, found 284.1090.



Ethyl 2-fluoro-2-(8-ethyl-phenanthridin-6-yl)acetate, **5b:** White sticky solid, eluent (5% ethyl acetate in hexane). Yield: 86% (80.2 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.66 – 8.41 (m, 2H), 8.26 – 8.03 (m, 2H), 7.81 – 7.59 (m, 3H), 6.51 (d, *J* = 48.1 Hz, 1H), 4.34 (qd, *J* = 7.1, 1.1 Hz, 2H), 2.90 (q, *J* = 7.6 Hz, 2H), 1.37 (t, *J* = 7.6 Hz, 3H), 1.25 (t, *J* = 5.1 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.3 (d, *J* = 25.6 Hz),152.8 (d, *J* = 20.2 Hz), 144.3, 142.8, 131.9, 131.8, 130.7, 128.6, 128.2, 124.8, 124.5, 124.2 (d, *J* = 4.0 Hz), 122.7, 122.0, 91.4 (d, *J* = 187.5 Hz), 62.1, 29.2, 15.6, 14.2; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -181.1; **HRMS** (TOF MS ES+) calcd. for$C_{19}H_{18}FNO_2H^+$ [M + H^+] m/z 312.1400, found 312.1390.

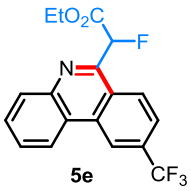


Ethyl 2-fluoro-2-(8-chloro-phenanthridin-6-yl)acetate, **5c:** White solid, eluent (10% ethyl acetate in hexane), mp: 112-116 °C. Yield: 81% (77 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.59 (t, *J* = 9.0 Hz, 1H), 8.54 – 8.45 (m, 1H), 8.42 – 8.30 (m, 1H), 8.22 – 8.10 (m, 1H), 7.82 (dt, *J* = 5.3, 2.7 Hz, 1H), 7.79 – 7.71 (m, 2H), 6.46 (d, *J* = 48.1 Hz, 1H), 4.46 – 4.25 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.8 (d, *J* = 25.3 Hz), 152.0 (d, *J* = 20.7 Hz), 143.0, 134.1, 132.1, 131.8, 131.0, 129.4, 128.8, 125.4 (d, *J* = 5.1 Hz), 125.1, 124.4, 124.1, 122.1, 91.2 (d, *J* = 188.1 Hz), 62.4, 14.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -181.1; **HRMS** (TOF MS ES+) calcd. for$C_{17}H_{13}ClFNO_2H^+$ [M + H^+] m/z 318.0697, found 318.0700.



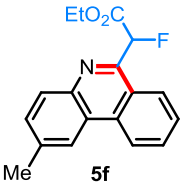
5d

Methyl 6-(2-ethoxy-1-fluoro-2-oxoethyl)phenanthridine-8-carboxylate, **5d:** White solid, eluent (12% ethyl acetate in hexane), mp: 101-104 ^{o}C.Yield: 80% (81.8 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.09 (s, 1H), 8.72 (d, *J* = 8.4 Hz, 1H), 8.60 (d, *J* = 8.1 Hz, 1H), 8.48 (d, *J* = 8.7 Hz, 1H), 8.21 (d, *J* = 8.1 Hz, 1H), 7.79 (dt, *J* = 23.1, 7.4 Hz, 2H), 6.59 (d, *J* = 48.1 Hz, 1H), 4.38 (dd, *J* = 13.4, 6.5 Hz, 2H), 4.04 (s, 3H), 1.30 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.8 (d, *J* = 25.1 Hz), 166.4, 153.5 (d, *J* = 20.1 Hz), 143.8, 136.6, 136.1, 131.0, 130.2, 128.8, 128.8, 128.3 (d, *J* = 4.3 Hz), 124.0, 123.7, 123.1, 122.7, 90.7 (d, *J* = 188.4 Hz), 62.3, 52.8, 14.2; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -180.1; **HRMS** (TOF MS ES+) calcd. for$C_{19}H_{16}FNO_4H^+$ [M + H^+] m/z 342.1142, found 342.1165.



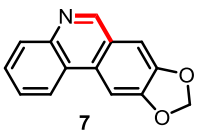
5e

Ethyl 2-fluoro-2-(9-(trifluoromethyl)phenanthridin-6-yl)acetate, **5e:** White solid, eluent (5% ethyl acetate in hexane), mp: 108-110 ^{o}C. Yield: 72% (75.8 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.95 (s, 1H), 8.65 – 8.61 (m, 1H), 8.54 (dt, *J* = 10.3, 4.7 Hz, 1H), 8.24 (dt, *J* = 6.5, 3.3 Hz, 1H), 7.93 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.88 – 7.76 (m, 1H), 6.55 (dd, *J* = 34.8, 25.6 Hz, 1H), 4.48 – 4.19 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.78 (d, *J* = 25.6 Hz), 152.55 (d, *J* = 20.6 Hz), 143.41, 133.56, 132.64 (q, *J* = 32.8 Hz), 131.10, 130.04, 129.09, 127.35 (d, *J* = 5.4 Hz), 125.28, 124.15, 123.93 (q, *J* = 11.2 Hz), 123.92 (q, J = 272.9 Hz), 122.56, 120.24 (q, *J* = 4.2 Hz), 91.50 (d, *J* = 187.9 Hz), 62.48, 14.25; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -62.7, -180.8; **HRMS** (TOF MS ES+) calcd. for$C_{18}H_{13}F_4NO_2H^+$ [M + H^+] m/z 352.0961, found 352.0957.



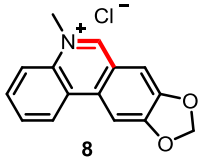
5f

Ethyl 2-fluoro-2-(2-methyl-phenanthridin-6-yl)acetate, **5f:** White solid, eluent (7% ethyl acetate in hexane), mp: 72-76 ^{o}C.Yield: 75% (65.4 mg); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.3 Hz, 1H), 8.37 (t, *J*= 5.2 Hz, 2H), 8.08 (d, *J* = 8.3 Hz, 1H), 7.90 – 7.83 (m, 1H), 7.75 – 7.69 (m, 1H), 7.59 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.49 (d, *J* = 48.0 Hz, 1H), 4.44 – 4.23 (m, 2H), 2.65 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 168.3 (d, *J* = 25.8 Hz), 152.1 (d, *J* = 20.4 Hz), 141.5, 138.5, 133.4, 130.9, 130.8, 130.6, 127.8, 126.1 (d, *J* = 4.2 Hz), 124.6, 124.3, 122.6, 121.8, 91.5 (d, *J* = 187.2 Hz), 62.2, 22.2, 14.2; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -180.5; **HRMS** (TOF MS ES+) calcd. for$C_{18}H_{16}FNO_2H^+$ [M + H^+] m/z 298.1243, found298.1238.



7

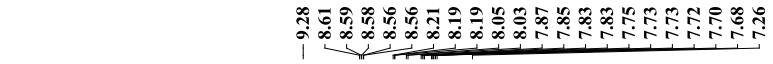
[1,3]Dioxolo[4,5-j]phenanthridine, **7**:White solid, eluent (8% ethyl acetate in hexane). Yield: 76% (84.7 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.23 (s, 1H), 8.32 (d, *J* = 7.9 Hz, 1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.6 Hz, 1H), 7.55 (dt, *J* = 15.1, 7.2 Hz, 2H), 7.30 (d, *J*= 8.6 Hz, 1H), 6.15 (s, 2H); **HRMS** (TOF MS ES+) calcd. for$C_{14}H_9NO_2H^+$ [M + H^+] m/z 224.0712, found 224.0711.



8

Bicolorine, **8**: Yellow solid; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.16 (s, 1H), 8.99 (d, *J* = 3.1 Hz, 1H), 8.57 (t, *J* = 20.0 Hz, 1H), 8.43 (s, 1H), 8.20 – 7.75 (m, 3H), 6.58 (s, 2H), 4.63 (s, 3H); **HRMS** (TOF MS ES+) calcd.for $C_{15}H_{12}NO_2^+$ [M] m/z 238.0863, found 238.0872.

^{1}H, ^{13}C NMR, and ^{19}FSpectra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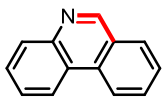
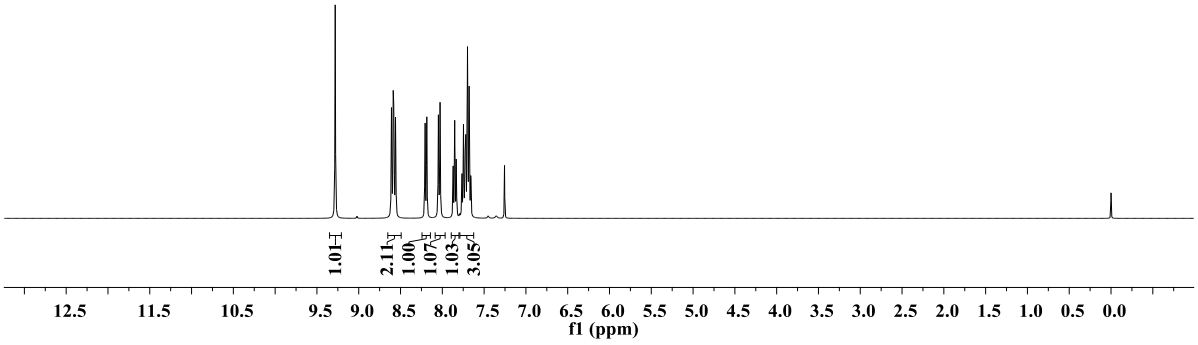
7.26


N

3a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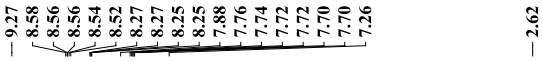
f1 (ppm)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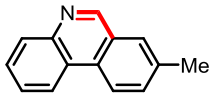
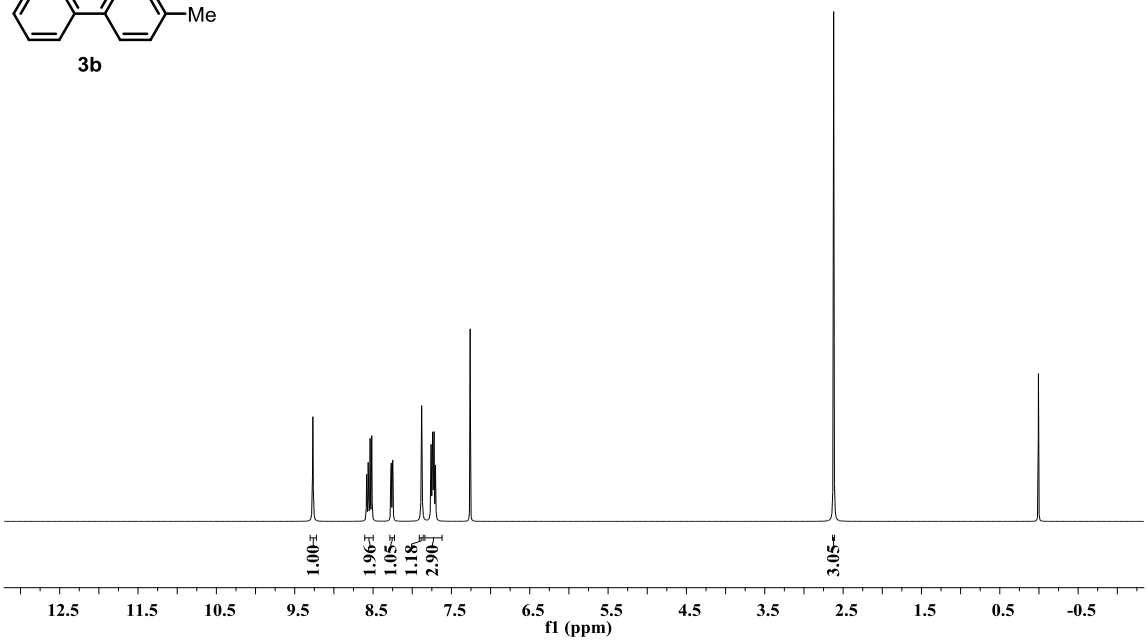
2.62


N
Me

3b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1.5
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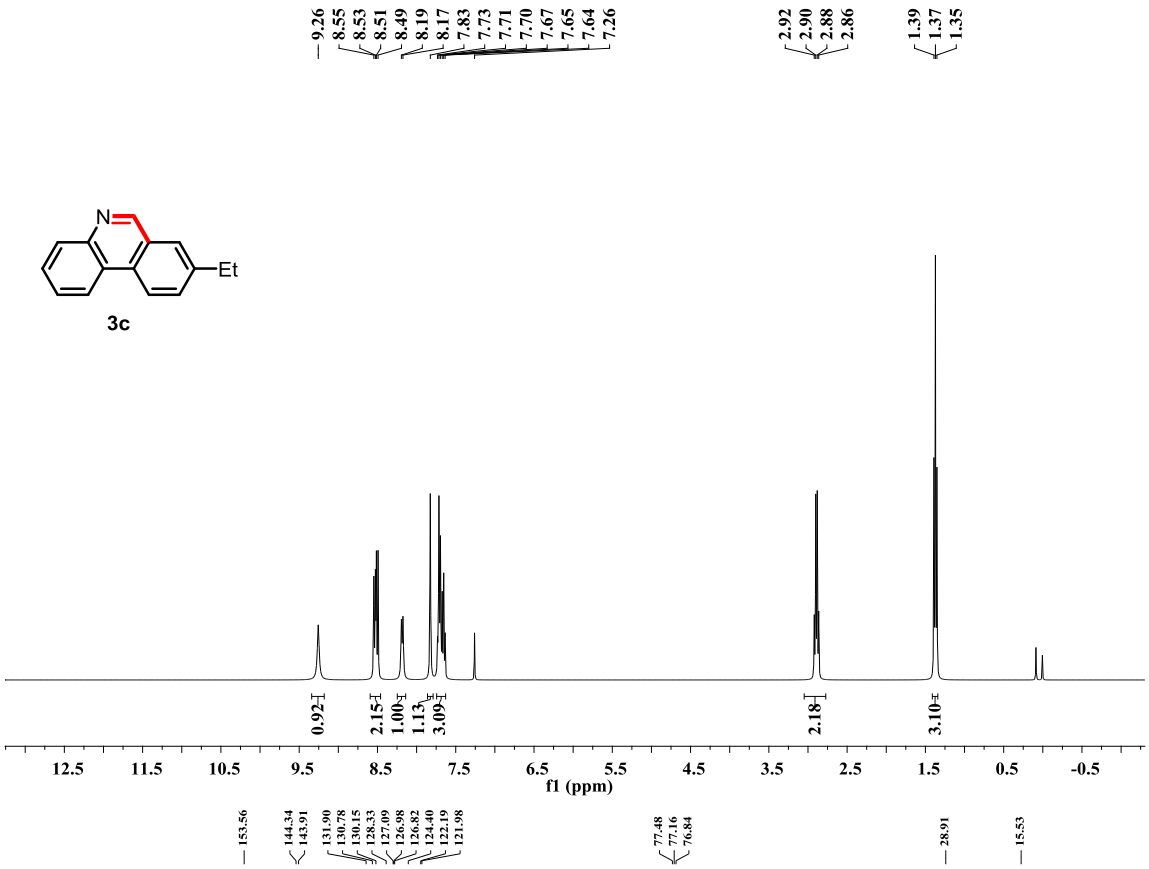
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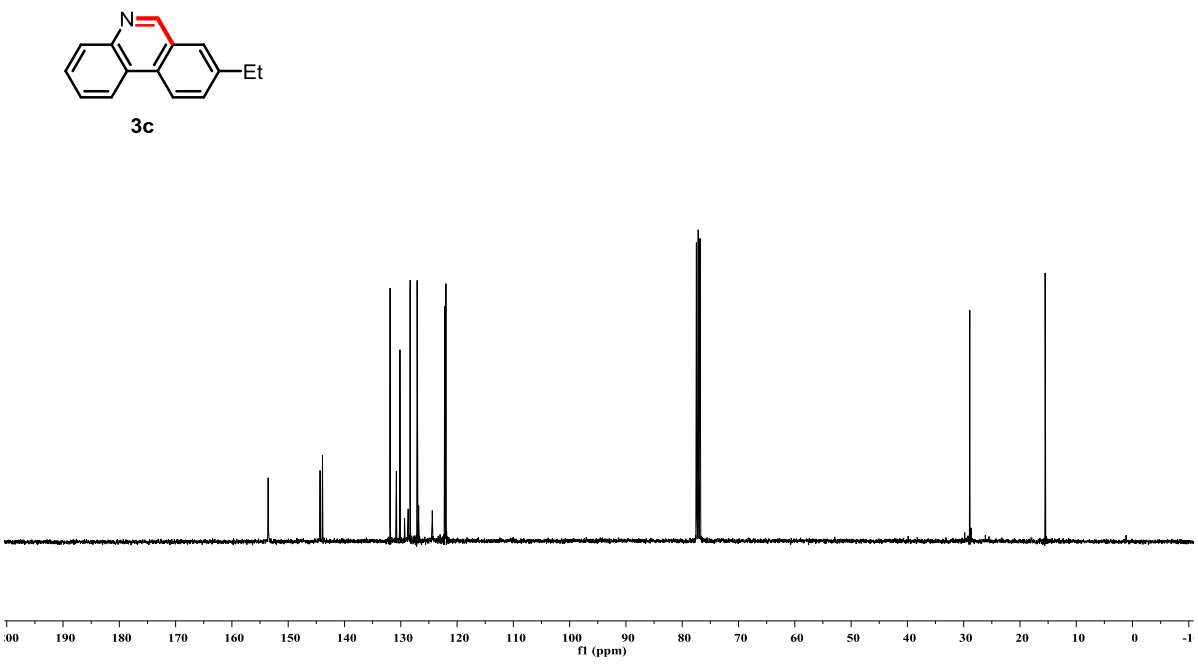
8.51
8.49
8.19
8.17
7.83
7.73
7.71
7.70
7.67
7.65
7.64
7.26
2.92
2.90
2.88
2.86
1.39
1.37
1.35
N
Et
3c
0.92
2.15
1.00
1.13
3.09
2.18
3.10
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)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130.15
128.33
127.09
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126.82
124.40
122.19
121.98
77.48
77.16
76.84
28.91
15.53
N
Et
3c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
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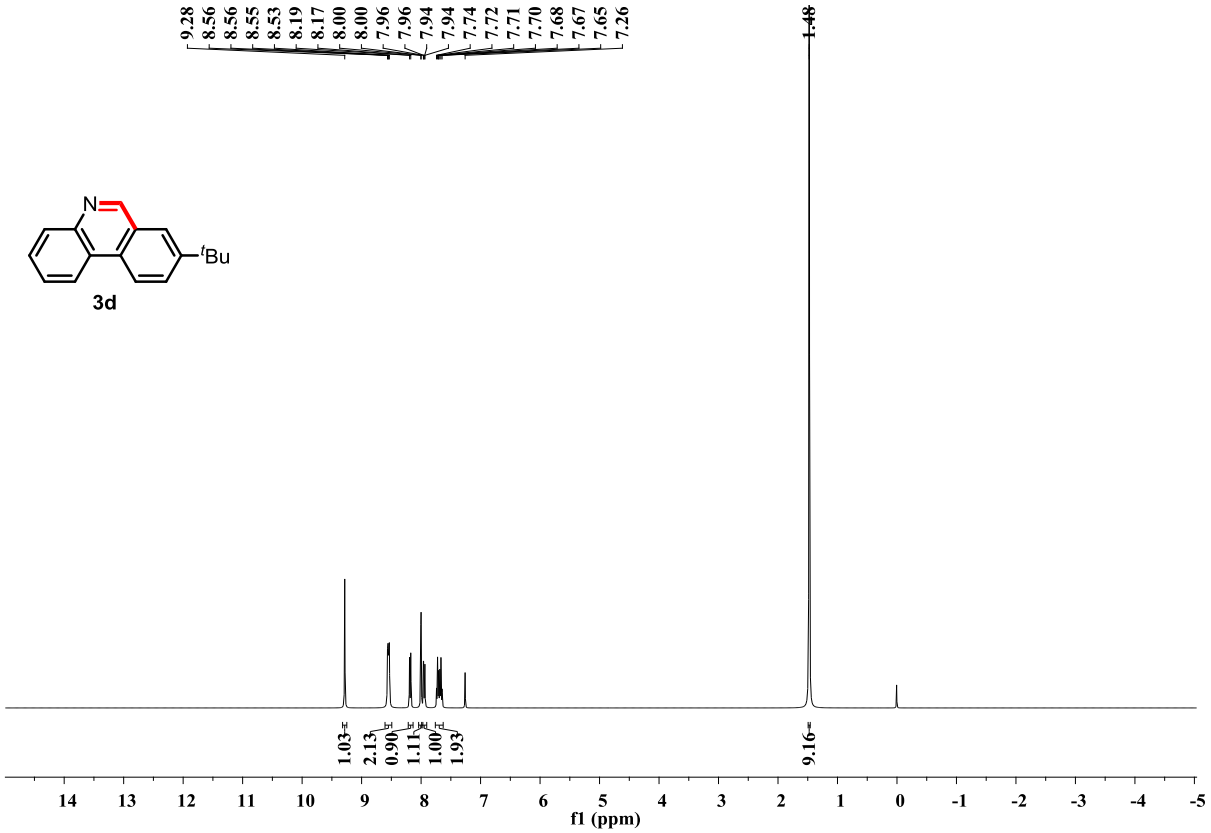
20
10
0
f1 (ppm)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8.56
8.56
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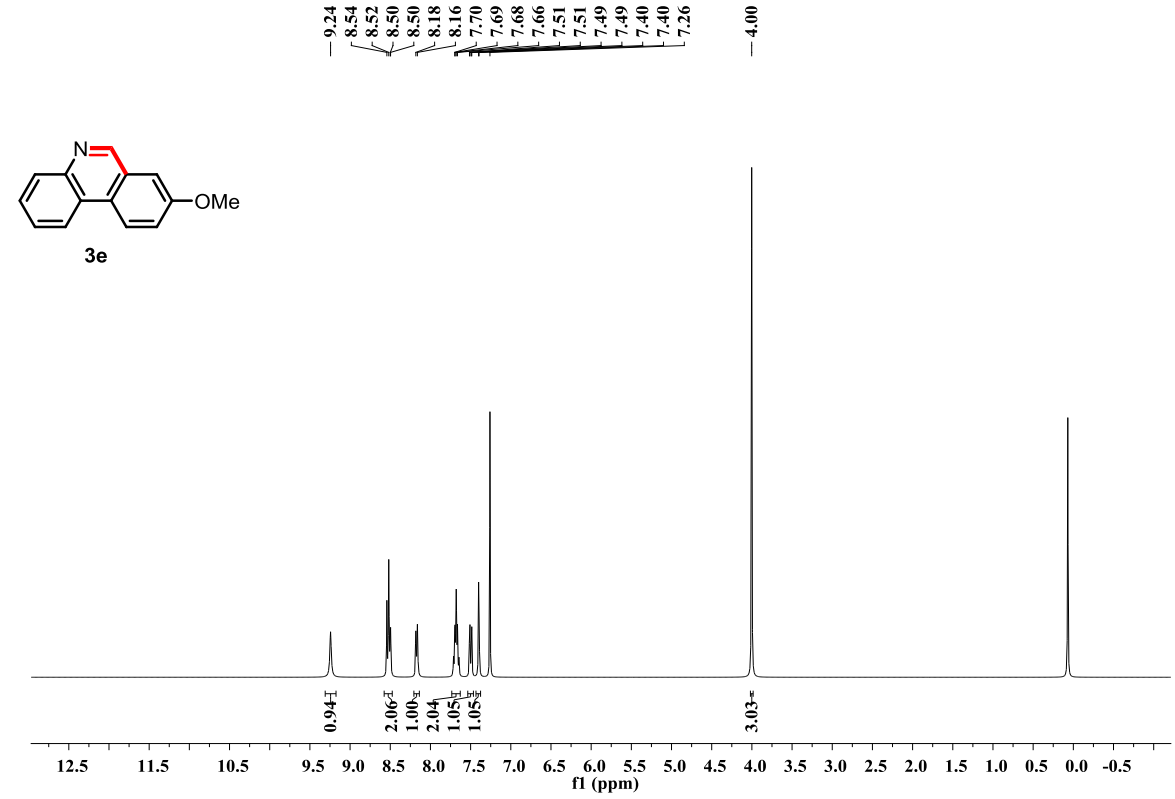
8.19
8.17
8.00
8.00
7.96
7.96
7.94
7.94
7.74
7.72
7.71
7.70
7.68
7.67
7.65
7.26
1.48
N
tBu
3d
1.03
2.13
0.90
1.11
1.00
1.93
9.16
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)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8.54
8.52
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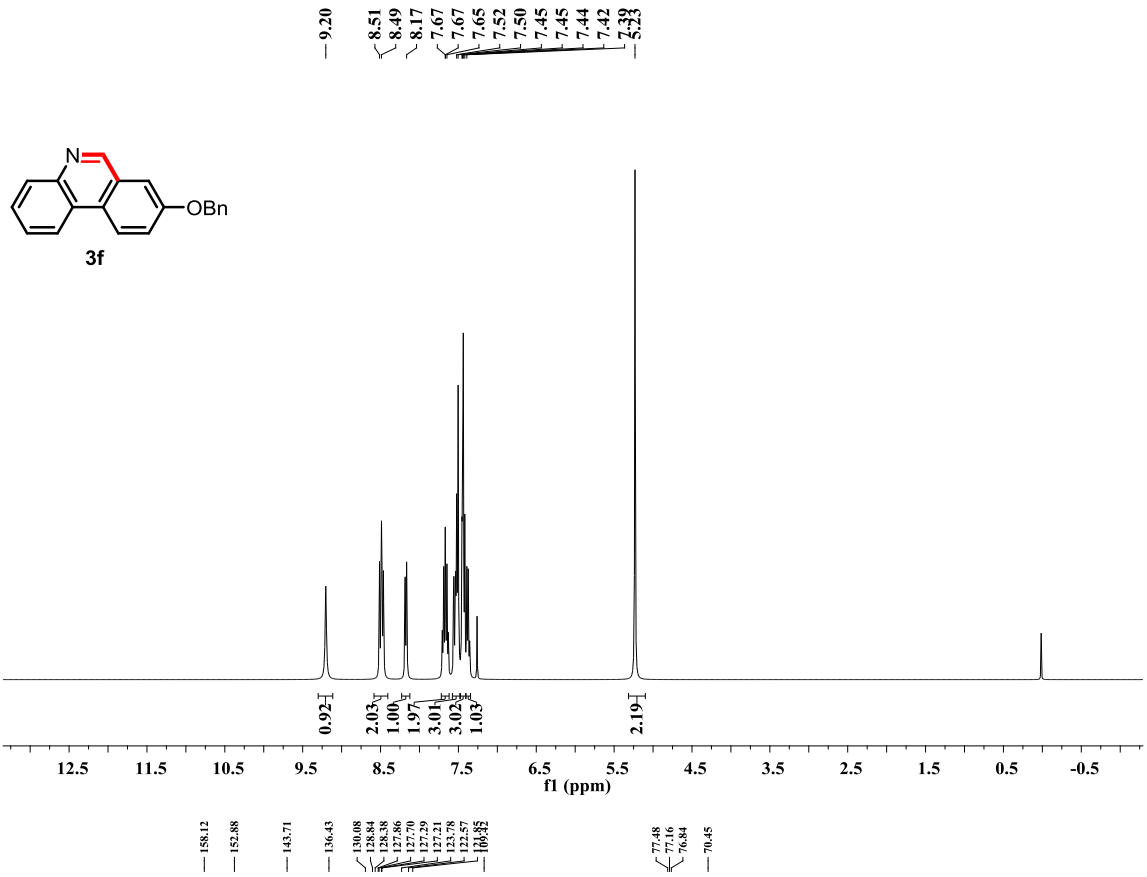
8.50
8.18
8.16
7.70
7.69
7.68
7.66
7.51
7.51
7.49
7.49
7.40
7.40
7.26
4.00
N
OMe
3e
0.94
2.06
1.00
2.04
1.05
1.05
3.03
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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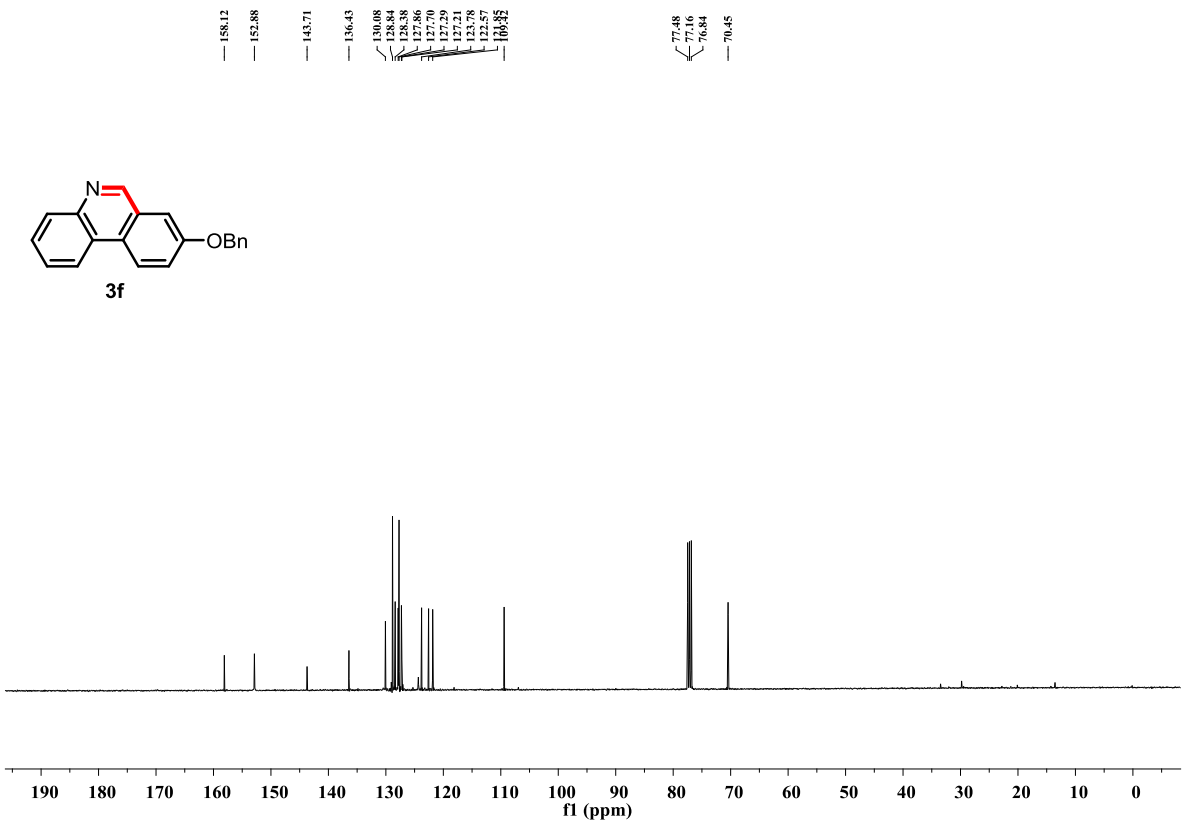
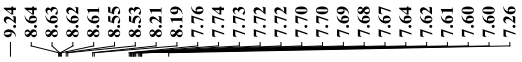
7.67
7.65
7.52
7.50
7.45
7.45
7.44
7.42
7.39
7.33
N
OBn
3f
0.92
2.03
1.00
1.97
3.01
3.02
1.03
2.19
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


158.12
152.88
143.71
136.43
130.08
128.84
128.38
127.86
127.70
127.29
127.21
123.78
122.57
110.85
110.42
77.48
77.16
76.84
70.45
N
OBn
3f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.55
8.53
8.21
8.19
7.76
7.74
7.73
7.72
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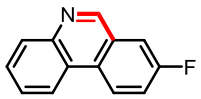
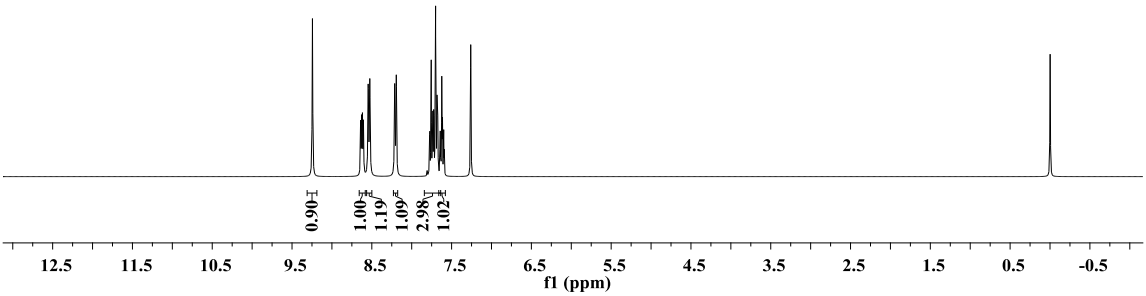
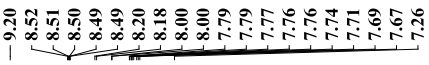
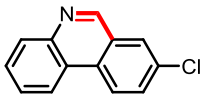
7.64
7.62
7.61
7.60
7.60
7.26


N
F

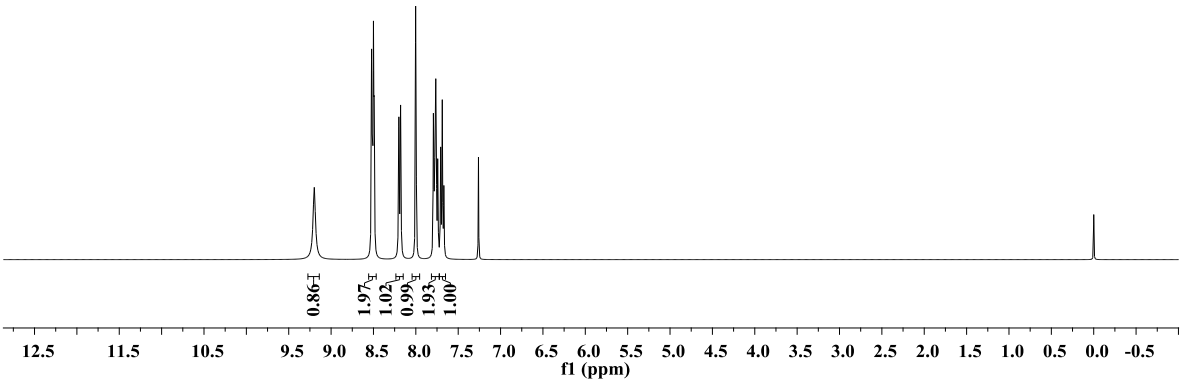
3g


0.90
1.00
1.19
1.09
2.98
1.02
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


9.20
8.52
8.51
8.50
8.49
8.49
8.20
8.18
8.00
8.00
7.79
7.79
7.77
7.76
7.76
7.74
7.71
7.69
7.67
7.26


N
Cl

3h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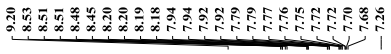
1.97
1.02
0.99
1.93
1.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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8.53
8.51
8.51
8.48
8.45
8.20
8.20
8.19
8.18
7.94
7.94
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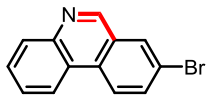
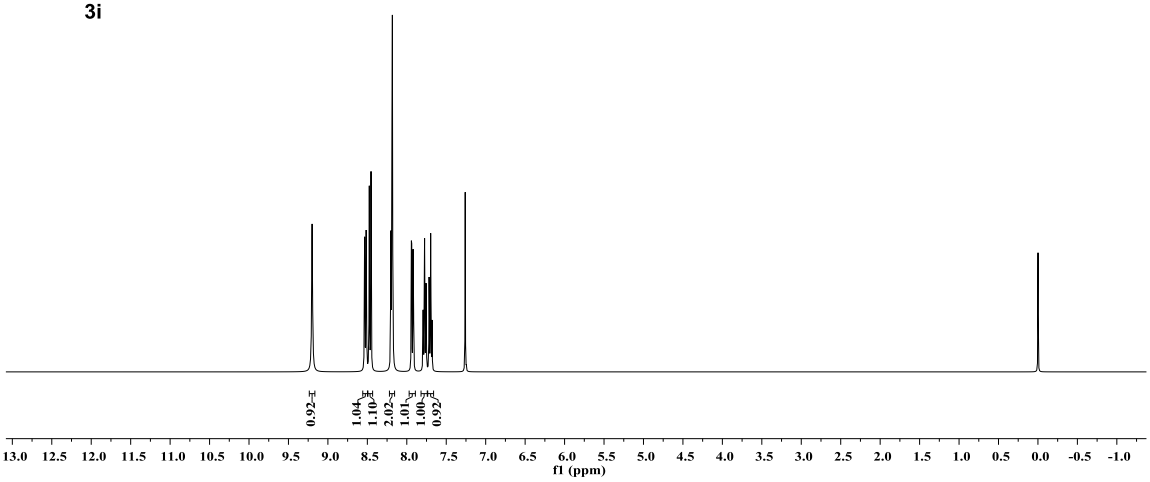
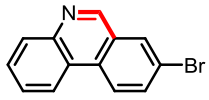
7.75
7.72
7.72
7.70
7.68
7.26


N
Br

3i

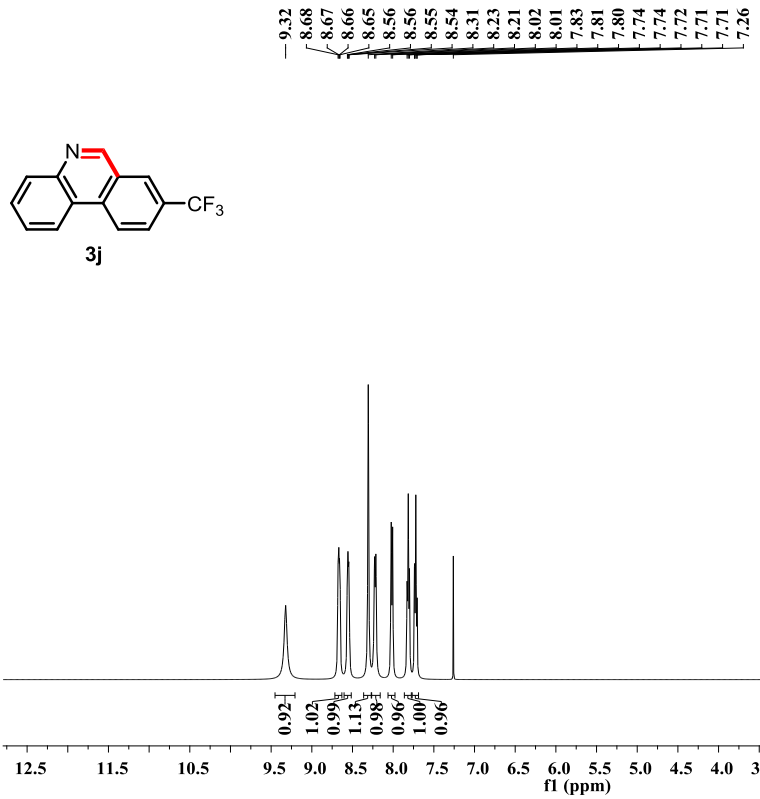

0.92
1.04
1.10
2.02
1.01
1.00
0.92
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

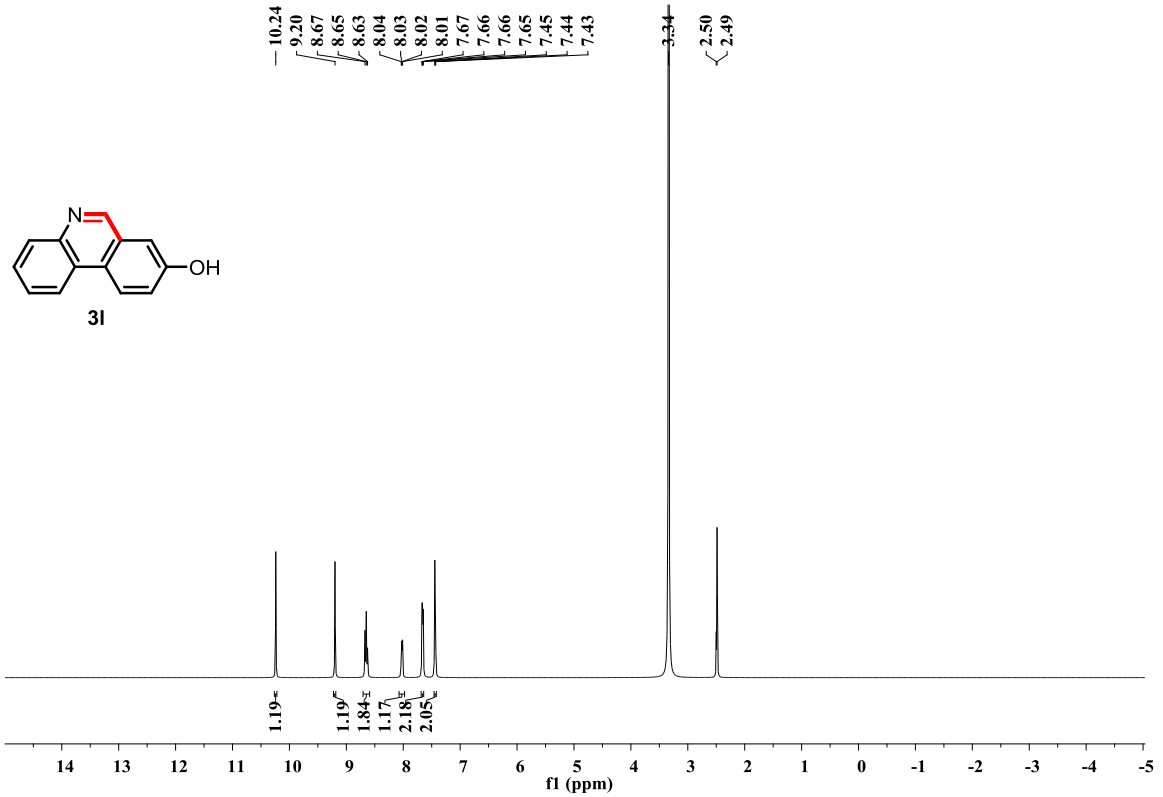

152.29
144.52
134.34
132.39
131.36
131.12
130.47
129.26
127.71
123.98
123.72
122.21
121.45
77.48
77.16
76.84

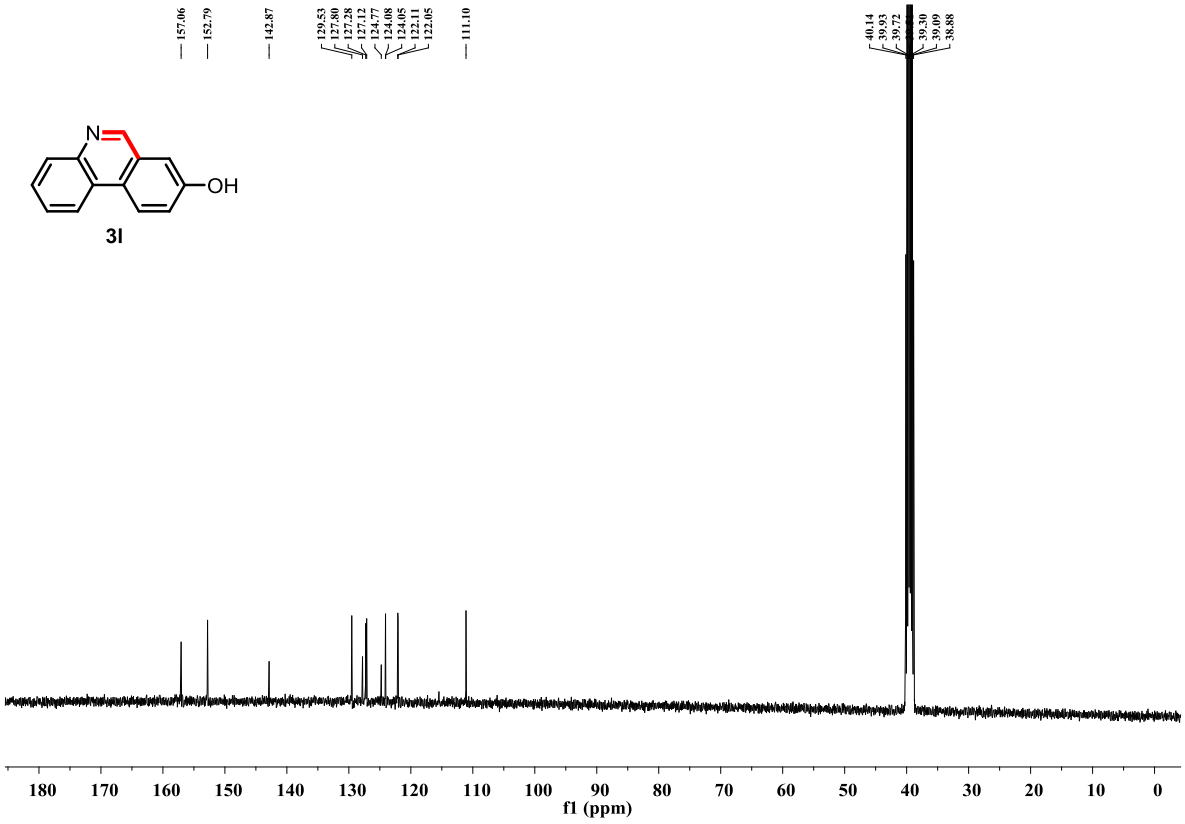

N
Br

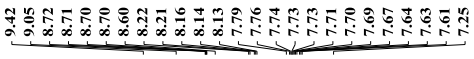
3i

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


9.32
8.68
8.67
8.66
8.65
8.56
8.56
8.55
8.54
8.31
8.23
8.21
8.02
8.01
7.83
7.81
7.80
7.74
7.74
7.72
7.71
7.71
7.26
N
CF3
3j
0.92
1.02
0.99
1.13
0.98
0.96
1.00
0.96
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
9.34
8.75
8.64
8.62
8.59
8.57
8.45
8.45
8.43
8.43
8.22
8.20
7.82
7.80
7.79
7.73
7.71
7.70
7.26
4.02
N
CO2Me
3k
0.95
0.84
1.17
1.09
0.94
1.04
0.99
1.00
3.09
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)


10.24
9.20
8.67
8.65
8.63
8.04
8.03
8.02
8.01
7.67
7.66
7.66
7.65
7.45
7.44
7.43
3.34
2.50
2.49
N
OH
3l
1.19
1.19
1.84
1.17
2.18
2.05
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)


157.06
152.79
142.87
129.53
127.80
127.28
127.12
124.77
124.08
124.05
122.11
122.05
111.10
40.14
39.93
39.72
39.30
39.09
38.88
N
OH
3l
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.42
9.05
8.72
8.71
8.70
8.70
8.60
8.22
8.21
8.16
8.14
8.13
7.79
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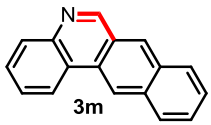
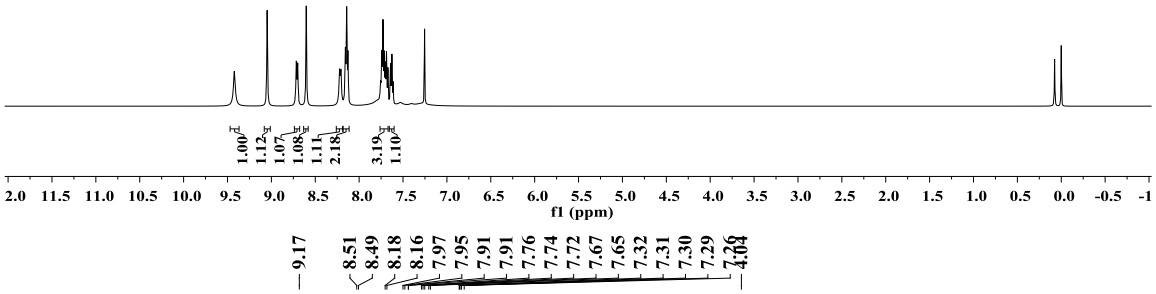
7.69
7.67
7.64
7.63
7.61
7.25


N
3m


1.00
1.12
1.07
1.08
1.11
2.18
3.19
1.10
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

9.17
8.51
8.49
8.18
8.16
7.97
7.95
7.91
7.91
7.76
7.74
7.72
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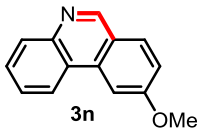
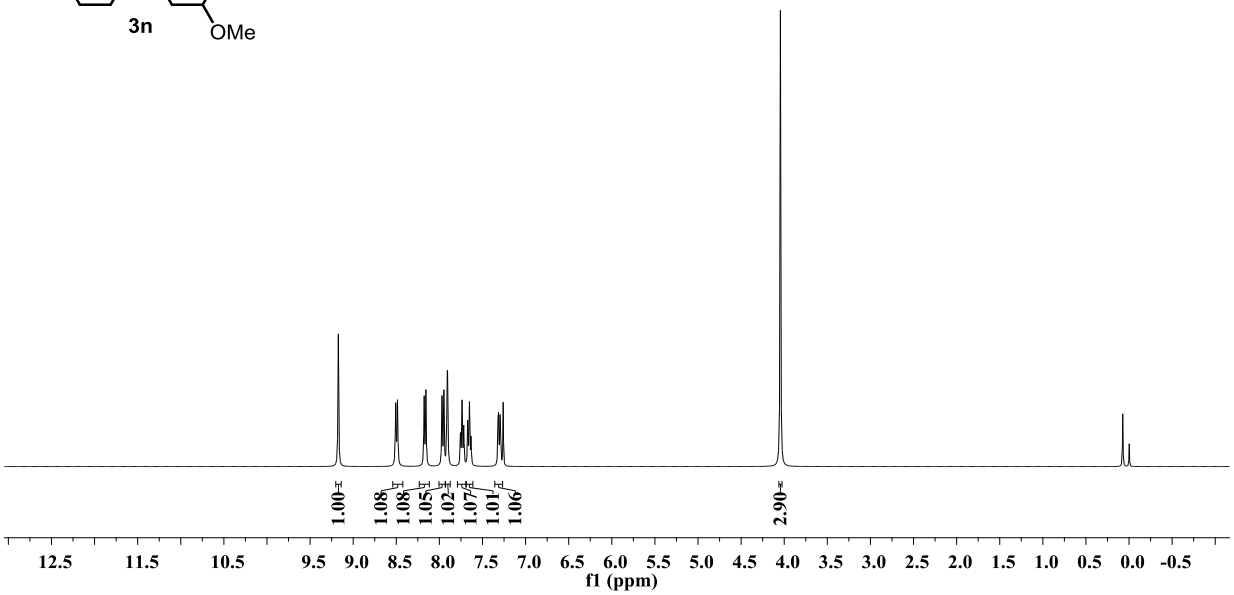
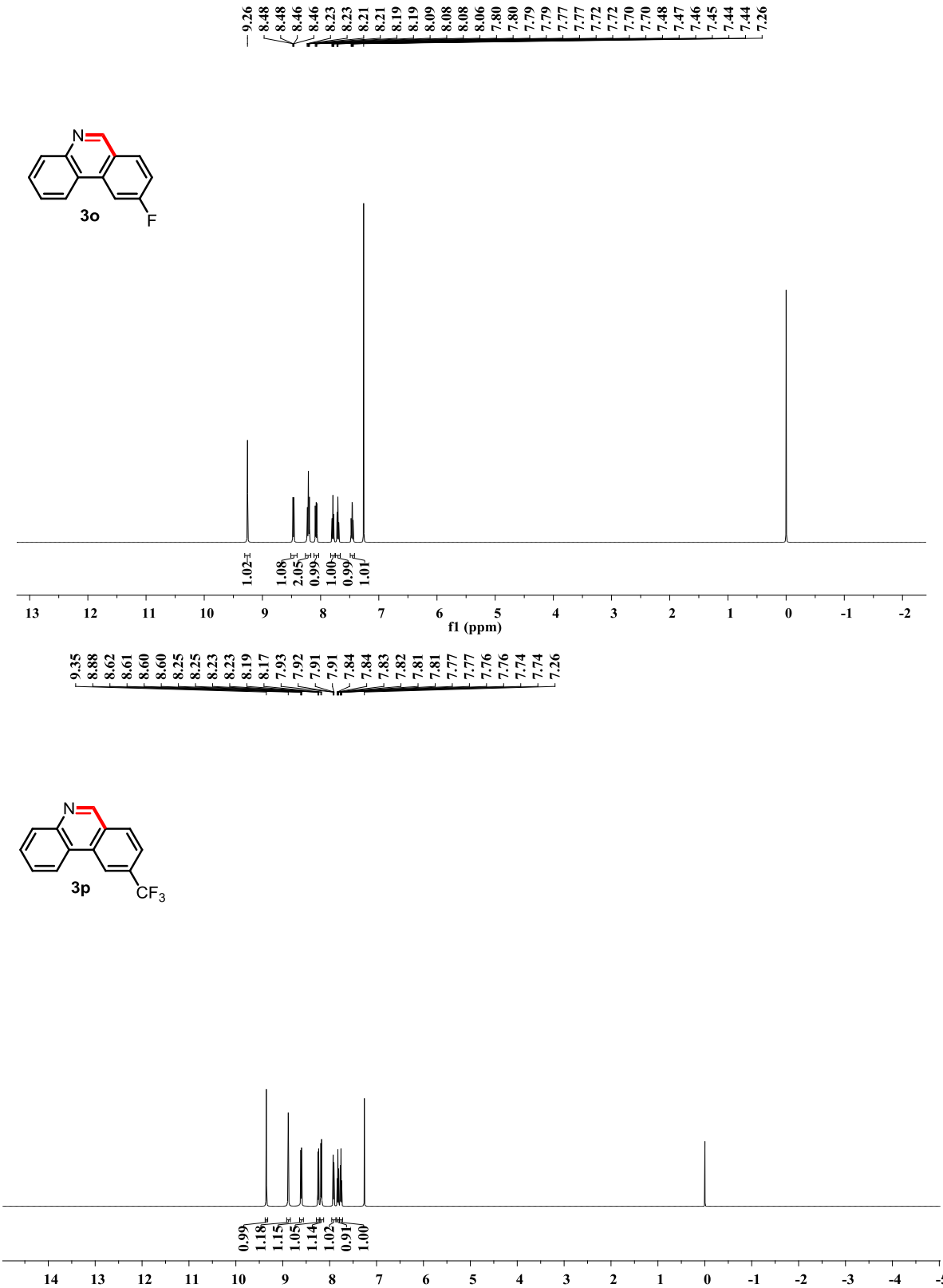
7.32
7.31
7.30
7.29
7.26
4.04


N
3n
OMe


1.00
1.08
1.08
1.05
1.02
1.07
1.01
1.06
2.90
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


9.26
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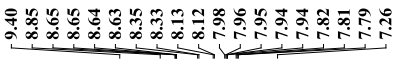
8.48
8.46
8.46
8.23
8.23
8.21
8.21
8.19
8.19
8.09
8.08
8.08
8.06
7.80
7.80
7.79
7.79
7.77
7.77
7.72
7.72
7.70
7.70
7.48
7.47
7.46
7.45
7.44
7.44
7.26
N
3o
F
1.02
1.08
2.05
0.99
1.00
0.99
1.01
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)
9.35
8.88
8.62
8.61
8.60
8.60
8.25
8.25
8.23
8.23
8.19
8.17
7.93
7.92
7.91
7.91
7.84
7.84
7.83
7.82
7.81
7.81
7.77
7.77
7.76
7.76
7.74
7.74
7.26
N
3p
CF3
0.99
1.18
1.15
1.05
1.14
1.02
0.91
1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)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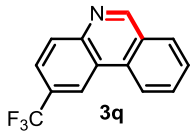
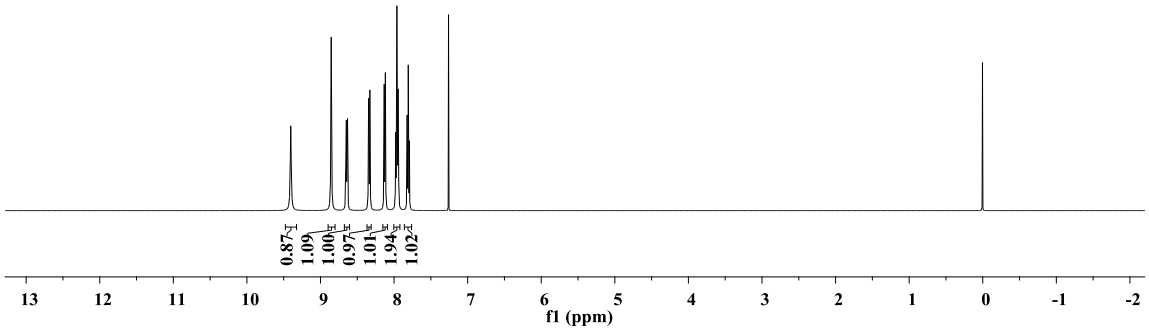
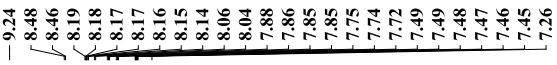
8.63
8.35
8.33
8.13
8.12
7.98
7.96
7.95
7.94
7.94
7.82
7.81
7.79
7.26


N
F3C
3q


0.87
1.09
1.00
0.97
1.01
1.94
1.02
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


9.24
8.48
8.46
8.19
8.18
8.17
8.17
8.16
8.15
8.14
8.06
8.04
7.88
7.86
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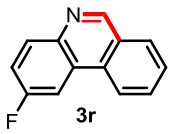
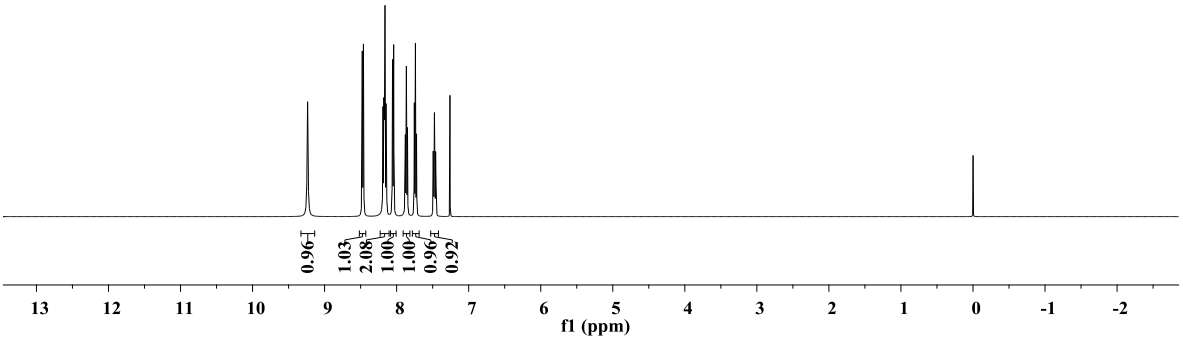
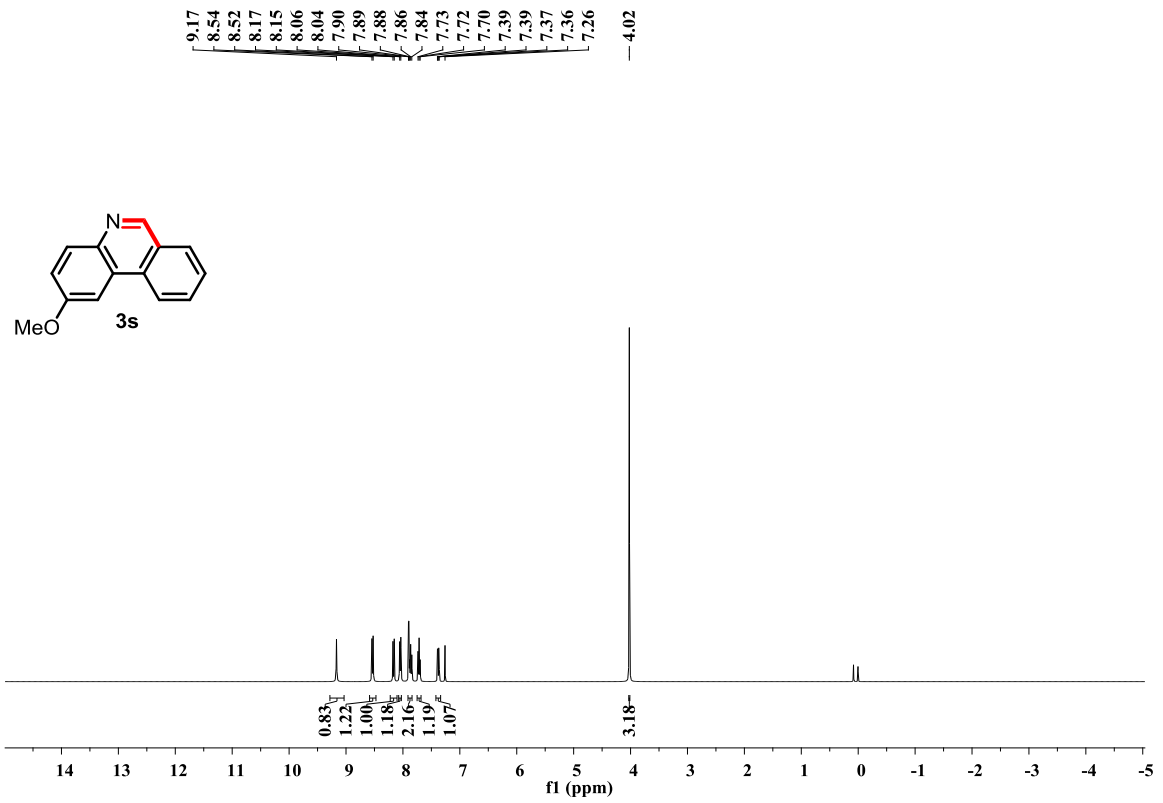
7.48
7.47
7.46
7.45
7.26


N
F
3r


0.96
1.03
2.08
1.00
1.00
0.96
0.92
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)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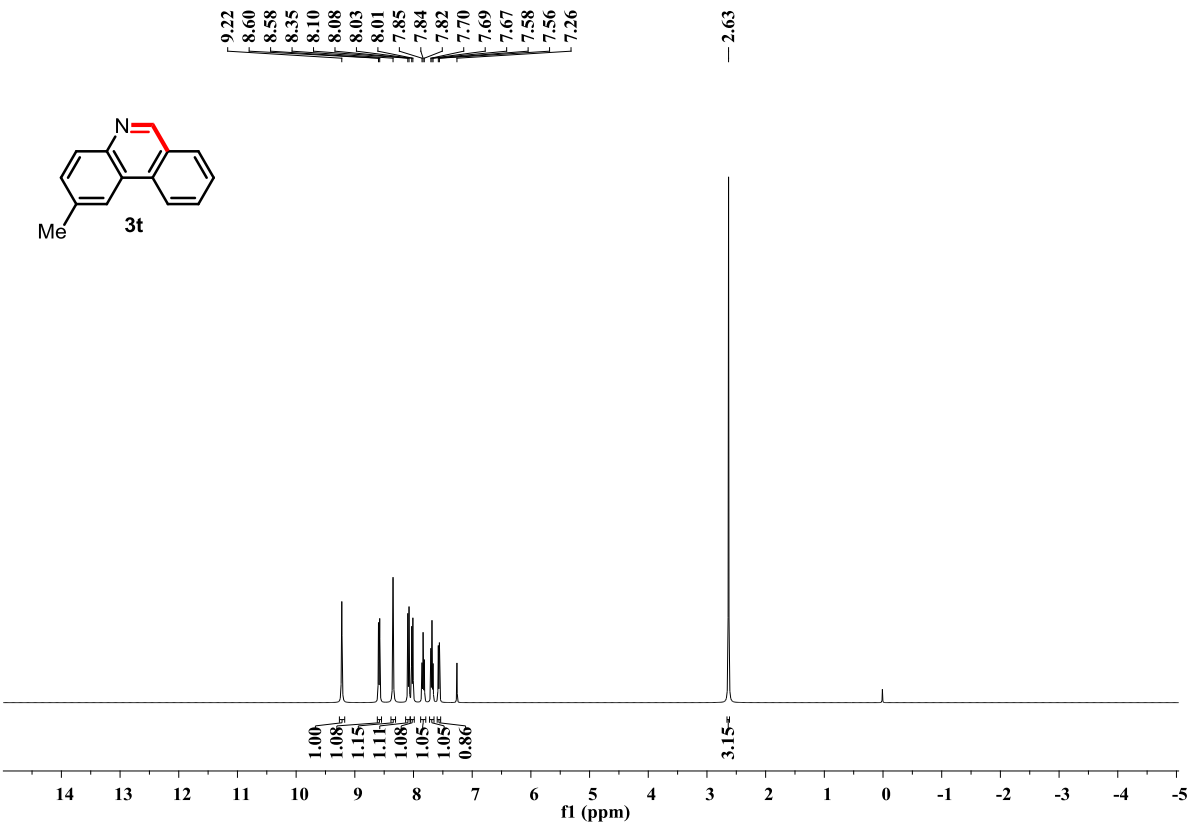
8.52
8.17
8.15
8.06
8.04
7.90
7.89
7.88
7.86
7.84
7.73
7.72
7.70
7.39
7.39
7.37
7.36
7.26
4.02
N
MeO
3s
0.83
1.22
1.00
1.18
2.16
1.19
1.07
3.18
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)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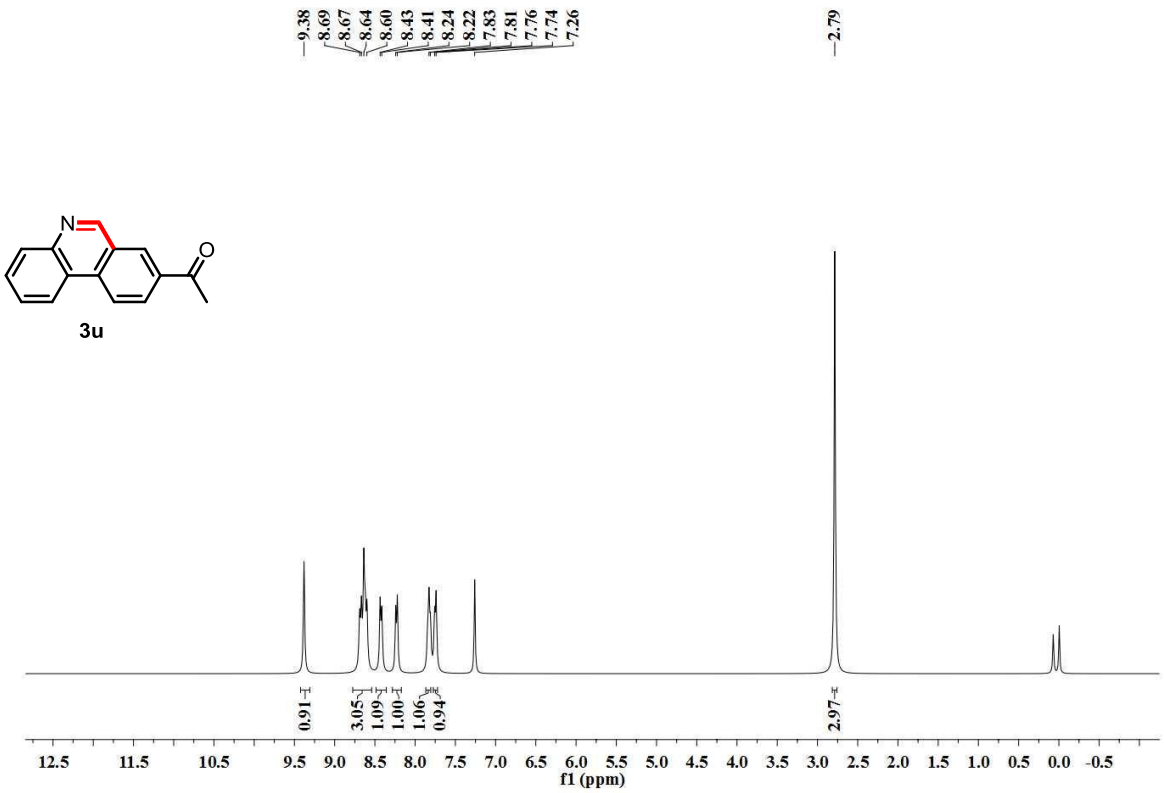
8.58
8.35
8.10
8.08
8.03
8.01
7.85
7.84
7.82
7.70
7.69
7.67
7.58
7.56
7.26
2.63
N
Me
3t
1.00
1.08
1.15
1.11
1.08
1.05
1.05
0.86
3.15
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)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8.67
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8.60
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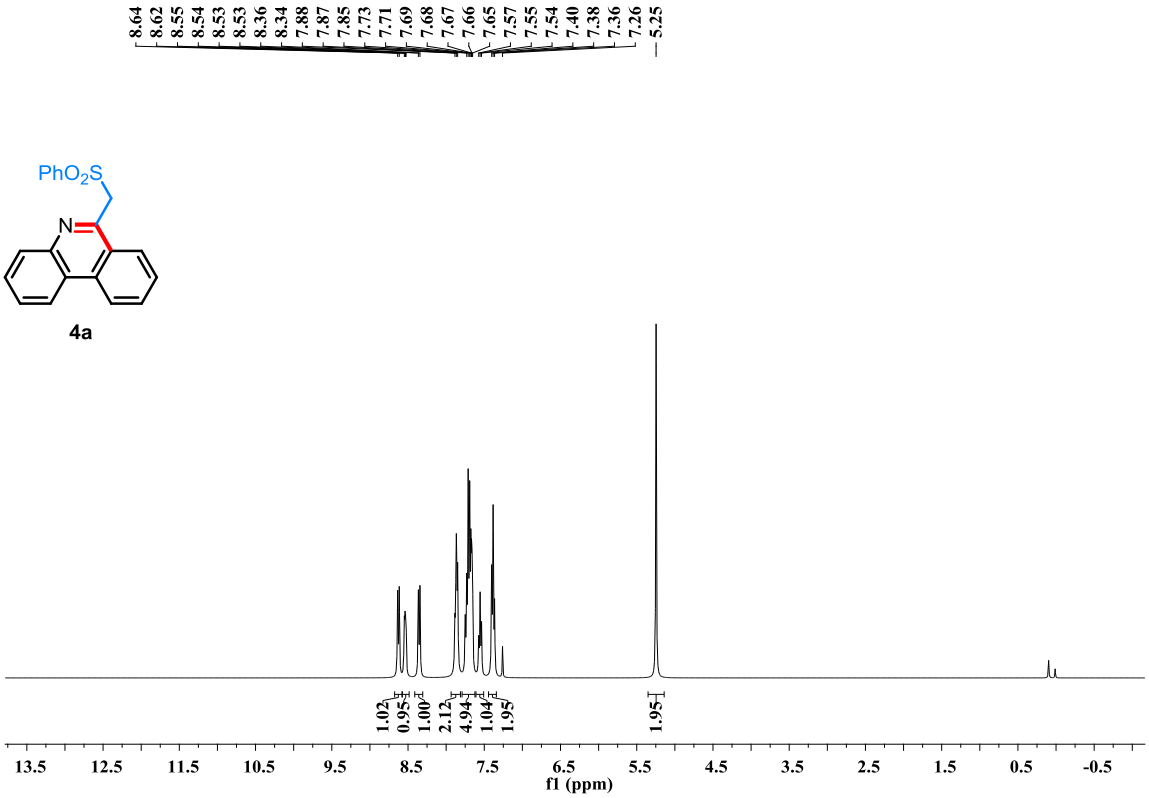
8.41
8.24
8.22
7.83
7.81
7.76
7.74
7.26
2.79
N
O
3u
0.91
3.05
1.09
1.00
1.06
0.94
2.97
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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8.54
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8.53
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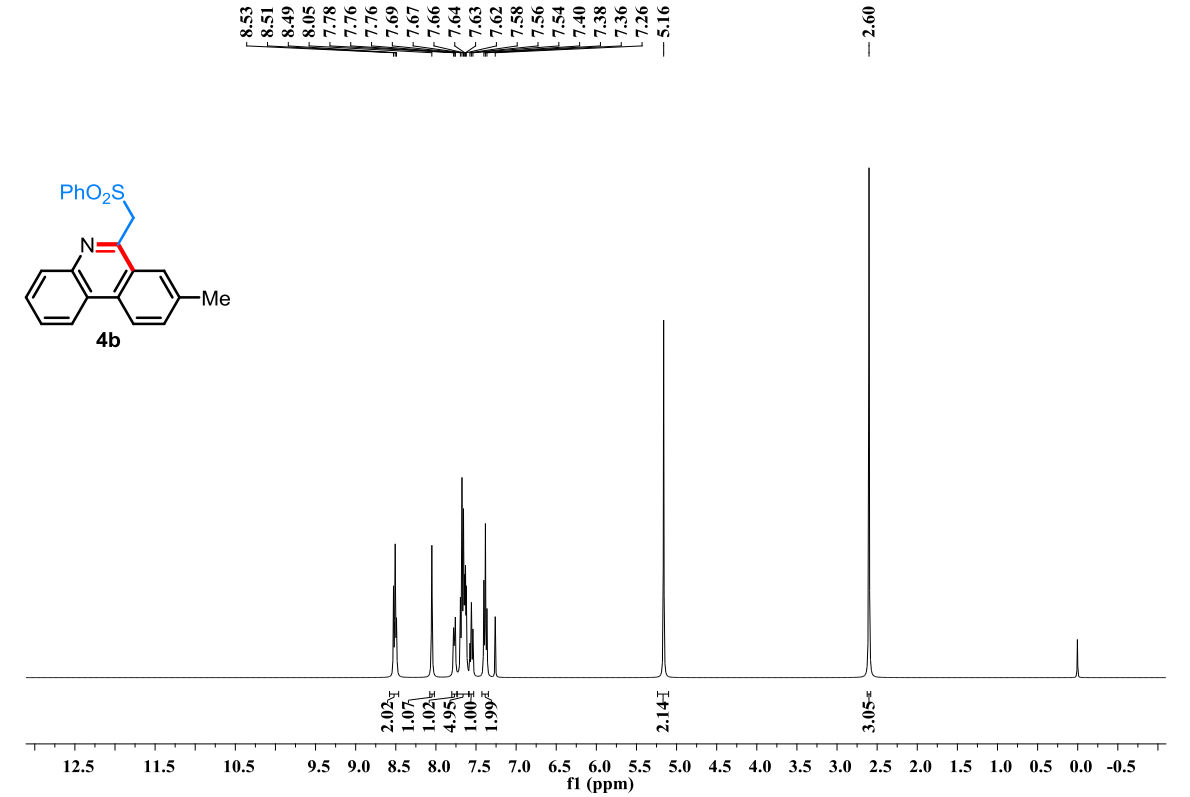
8.34
7.88
7.87
7.85
7.73
7.71
7.69
7.68
7.67
7.66
7.65
7.57
7.55
7.54
7.40
7.38
7.36
7.26
5.25
PhO2S
N
4a
1.02
0.95
1.00
2.12
4.94
1.04
1.95
1.95
13.5 12.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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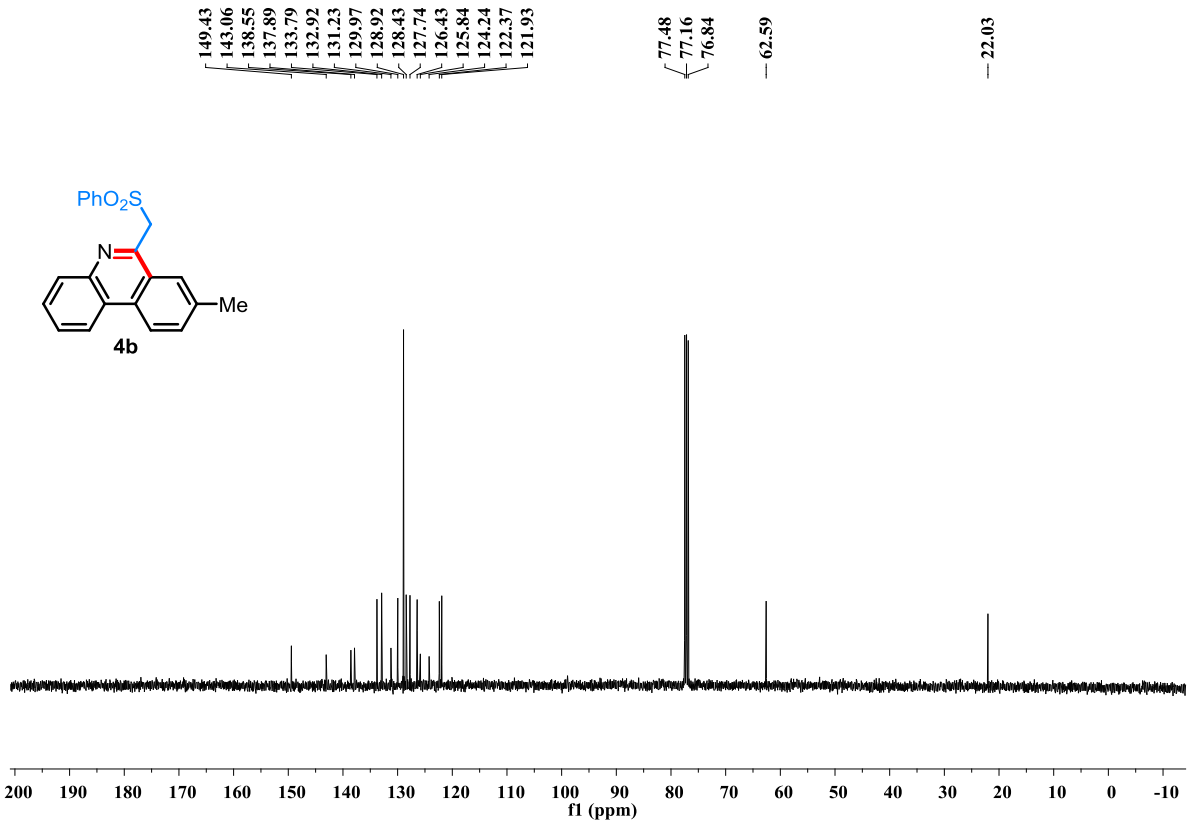
8.51
8.49
8.05
7.78
7.76
7.76
7.69
7.67
7.66
7.64
7.63
7.62
7.58
7.56
7.54
7.40
7.38
7.36
7.26
5.16
2.60
PhO2S
N
Me
4b
2.02
1.07
1.02
4.95
1.00
1.99
2.14
3.05
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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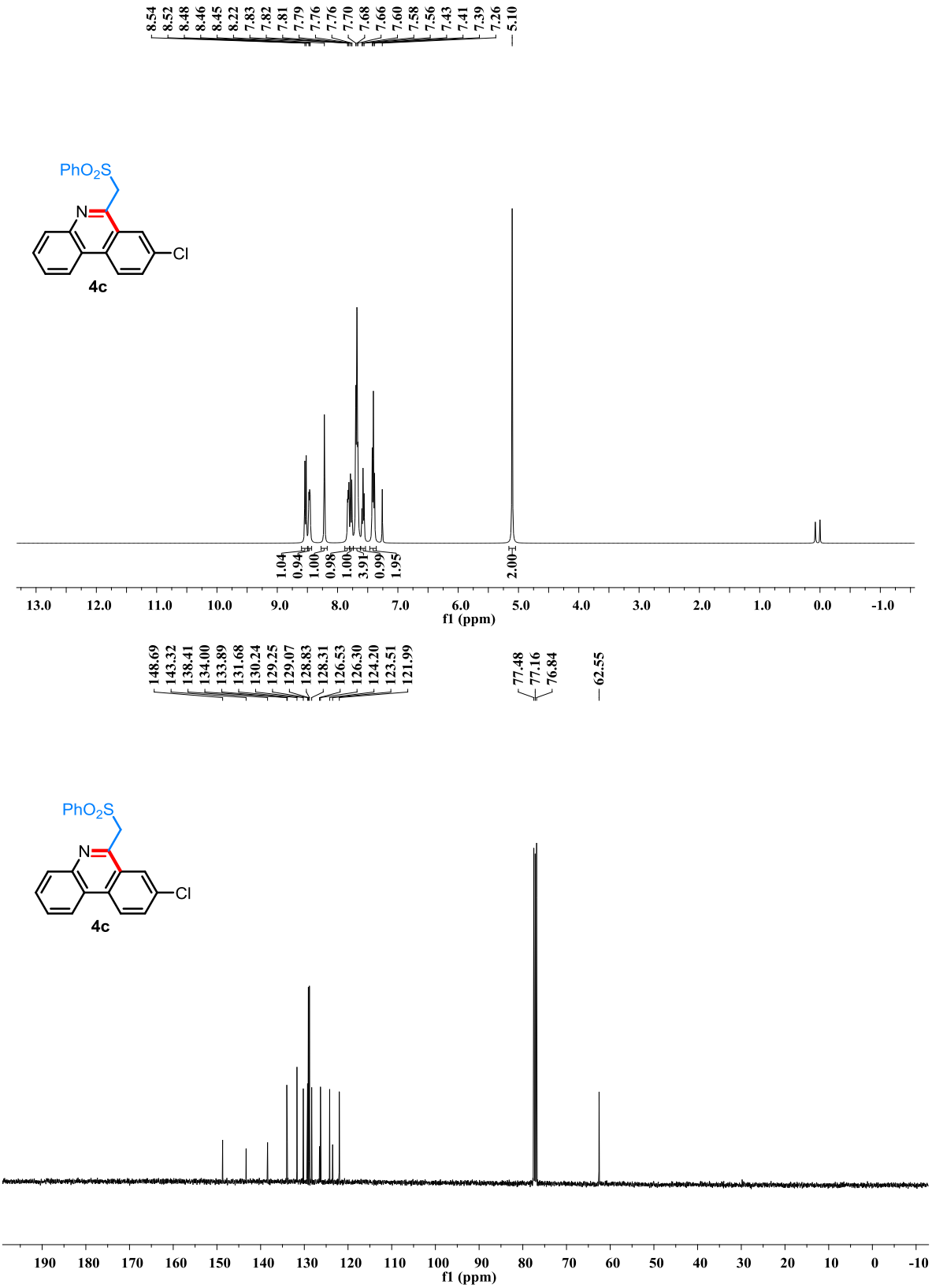
143.06
138.55
137.89
133.79
132.92
131.23
129.97
128.92
128.43
127.74
126.43
125.84
124.24
122.37
121.93
77.48
77.16
76.84
62.59
22.03
PhO2S
N
Me
4b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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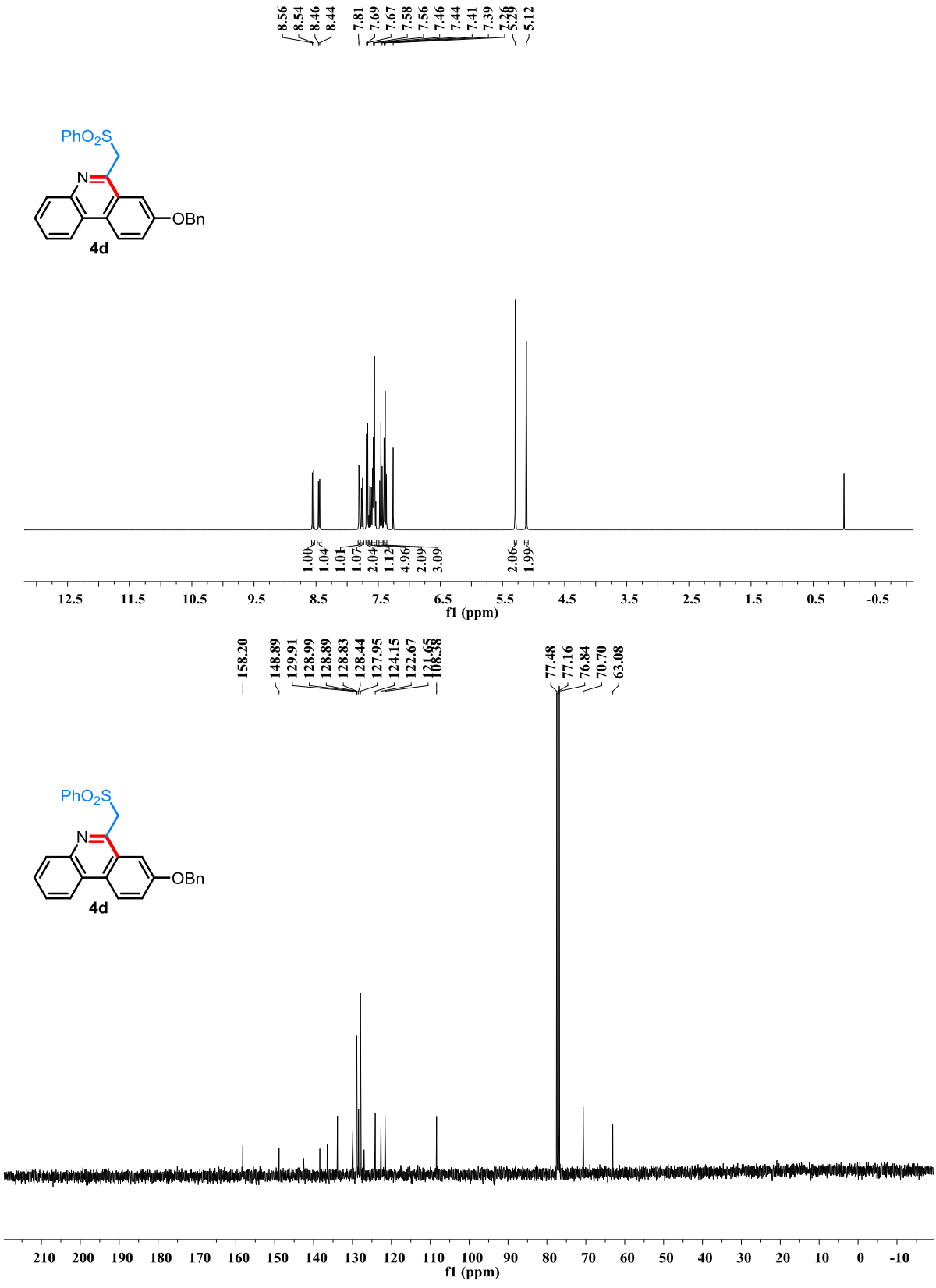
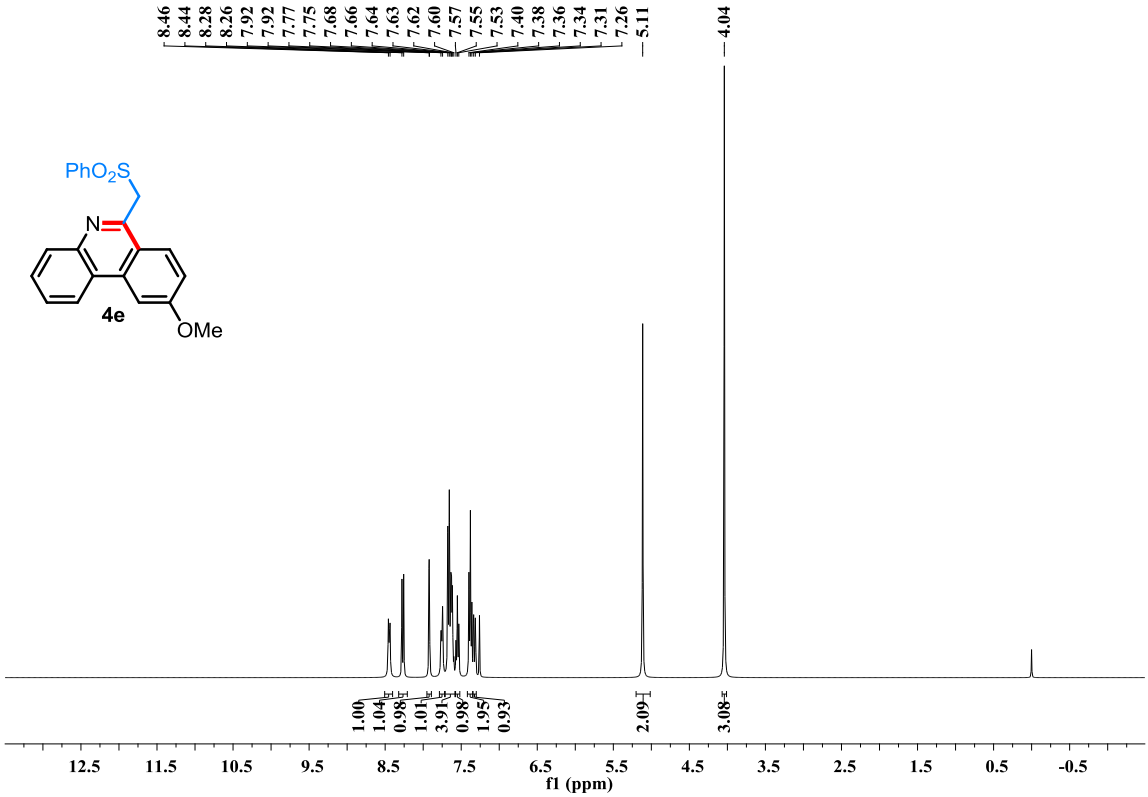
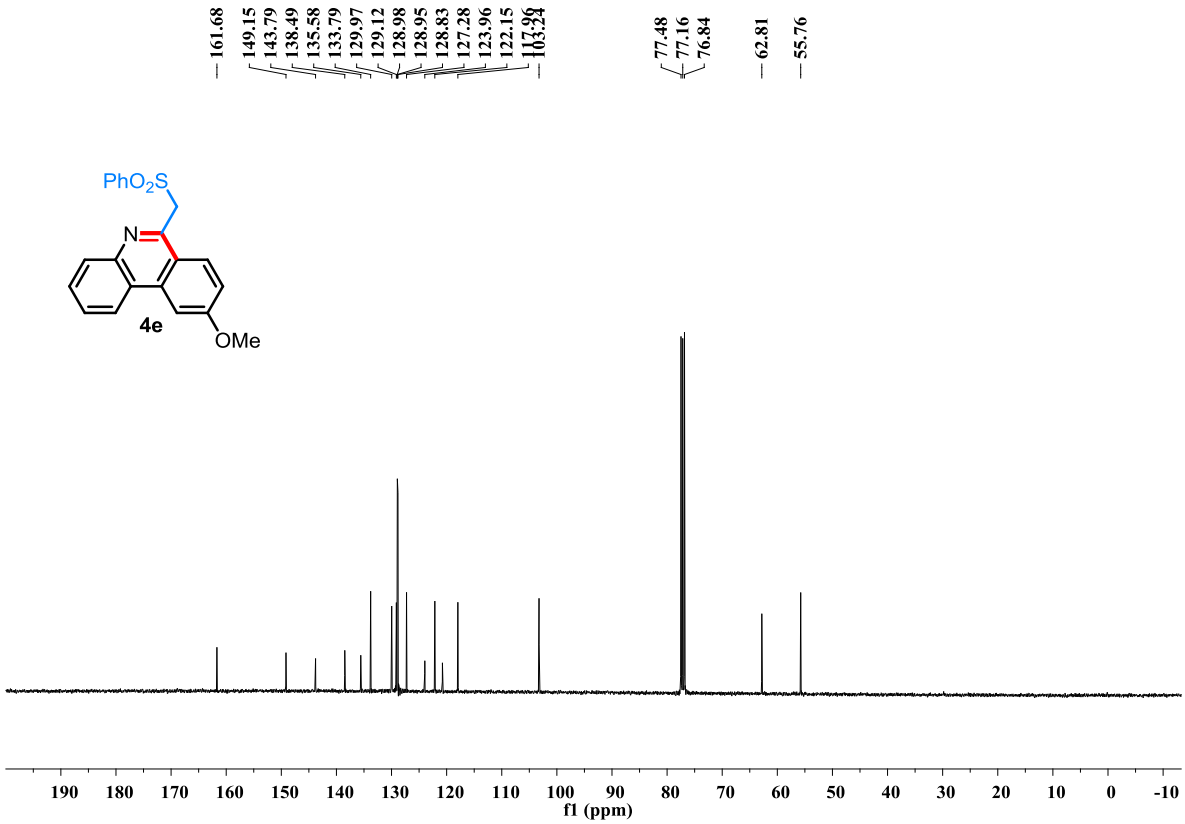
8.52
8.48
8.46
8.45
8.22
7.83
7.82
7.81
7.79
7.76
7.76
7.70
7.68
7.66
7.60
7.58
7.56
7.43
7.41
7.39
7.26
5.10
PhO2S
N
Cl
4c
1.04
0.94
1.00
0.98
1.00
3.91
0.99
1.95
2.00
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)
148.69
143.32
138.41
134.00
133.89
131.68
130.24
129.25
129.07
128.83
128.31
126.53
126.30
124.20
123.51
121.99
77.48
77.16
76.84
62.55
PhO2S
N
Cl
4c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.56
8.54
8.46
8.44
7.81
7.69
7.67
7.58
7.56
7.46
7.44
7.41
7.39
5.29
5.12
PhO2S
N
OBn
4d
1.00
1.04
1.01
1.07
2.04
1.12
4.96
2.09
3.09
2.06
1.99
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)
158.20
148.89
129.91
128.99
128.89
128.83
128.44
127.95
124.15
122.67
121.65
108.38
77.48
77.16
76.84
70.70
63.08
PhO2S
N
OBn
4d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.46
8.44
8.28
8.26
7.92
7.92
7.77
7.75
7.68
7.66
7.64
7.63
7.62
7.60
7.57
7.55
7.53
7.40
7.38
7.36
7.34
7.31
7.26
5.11
4.04
PhO2S
N
4e
OMe
1.00
1.04
0.98
1.01
3.91
0.98
1.95
0.93
2.09
3.08
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)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149.15
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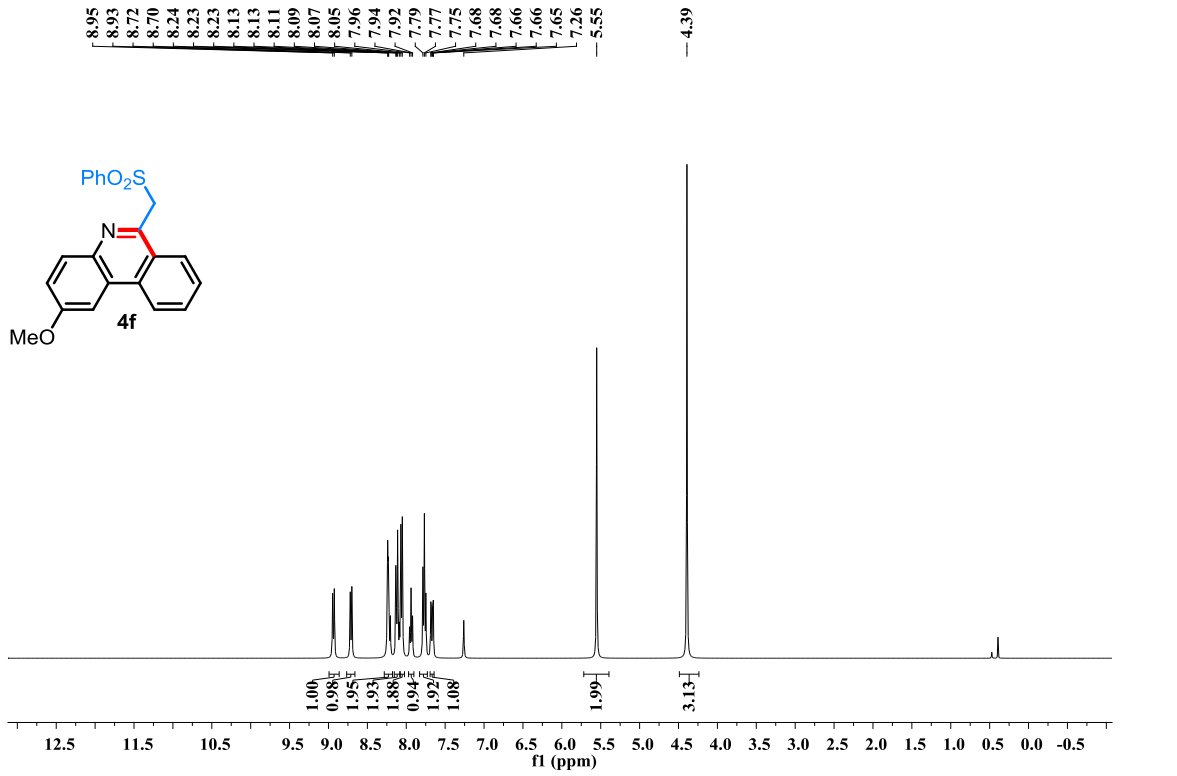
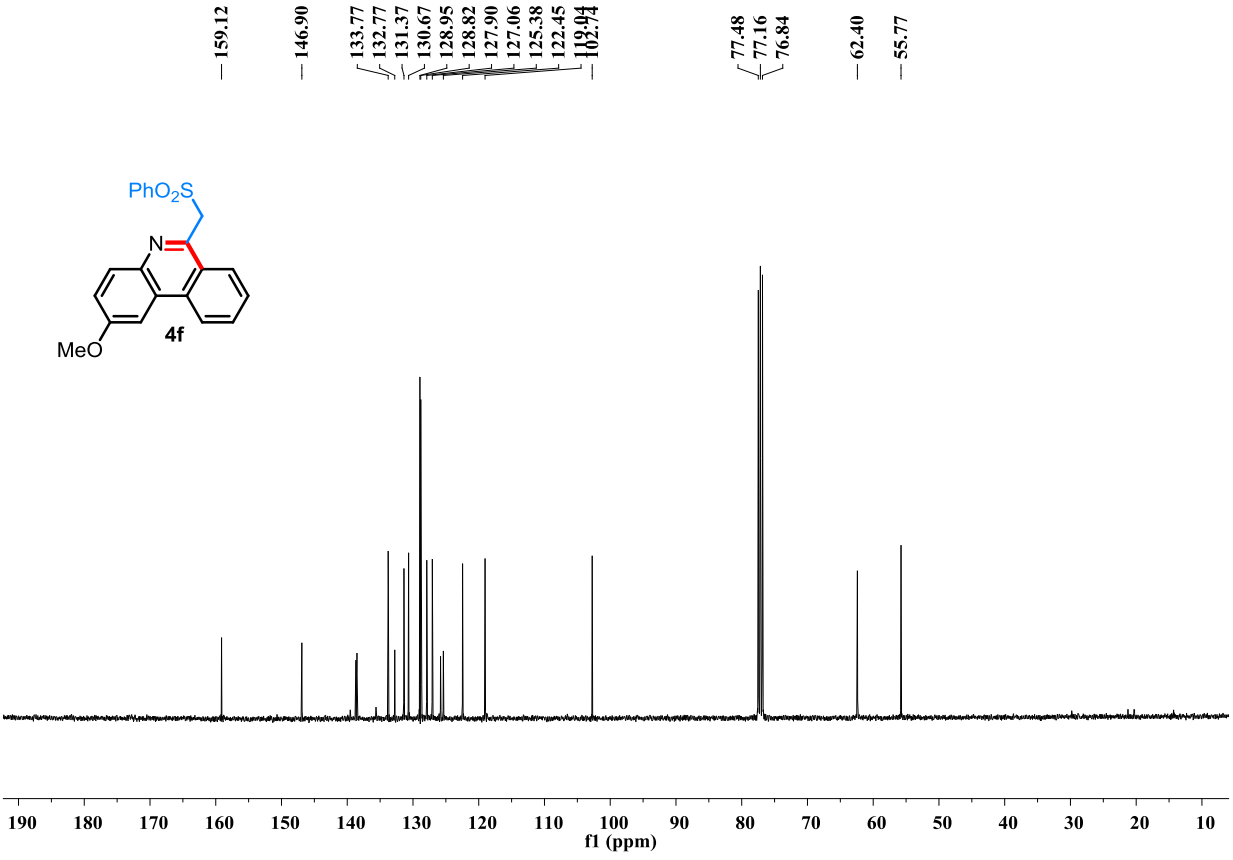
138.49
135.58
133.79
129.97
129.12
128.98
128.95
128.83
127.28
123.96
122.15
117.96
103.24
77.48
77.16
76.84
62.81
55.76
PhO2S
N
4e
OMe
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.95
8.93
8.72
8.70
8.24
8.23
8.23
8.13
8.13
8.11
8.09
8.07
8.05
7.96
7.94
7.92
7.79
7.77
7.75
7.68
7.68
7.66
7.66
7.65
7.26
5.55
4.39
PhO2S
N
MeO
4f
1.00
0.98
1.95
1.93
1.88
0.94
1.92
1.08
1.99
3.13
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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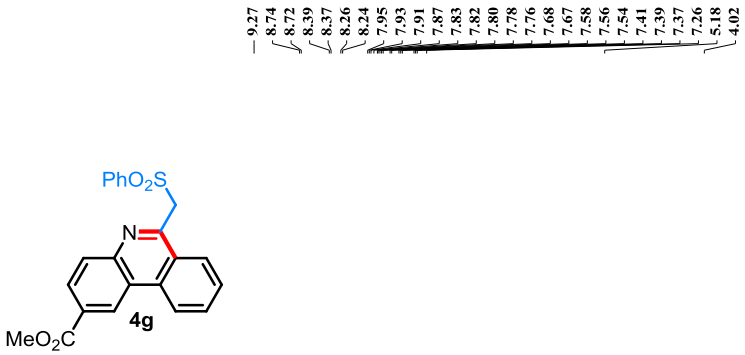
132.77
131.37
130.67
128.95
128.82
127.90
127.06
125.38
122.45
77.48
77.16
76.84
62.40
55.77
PhO2S
N
MeO
4f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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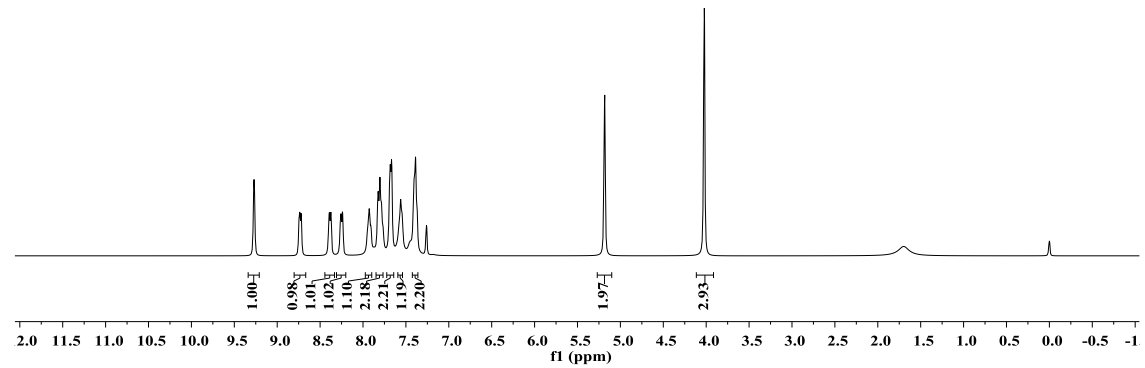
8.72
8.39
8.37
8.26
8.24
7.95
7.93
7.91
7.87
7.83
7.82
7.80
7.78
7.76
7.68
7.67
7.58
7.56
7.54
7.41
7.39
7.37
7.26
5.18
4.02
PhO2S
N
4g
MeO2C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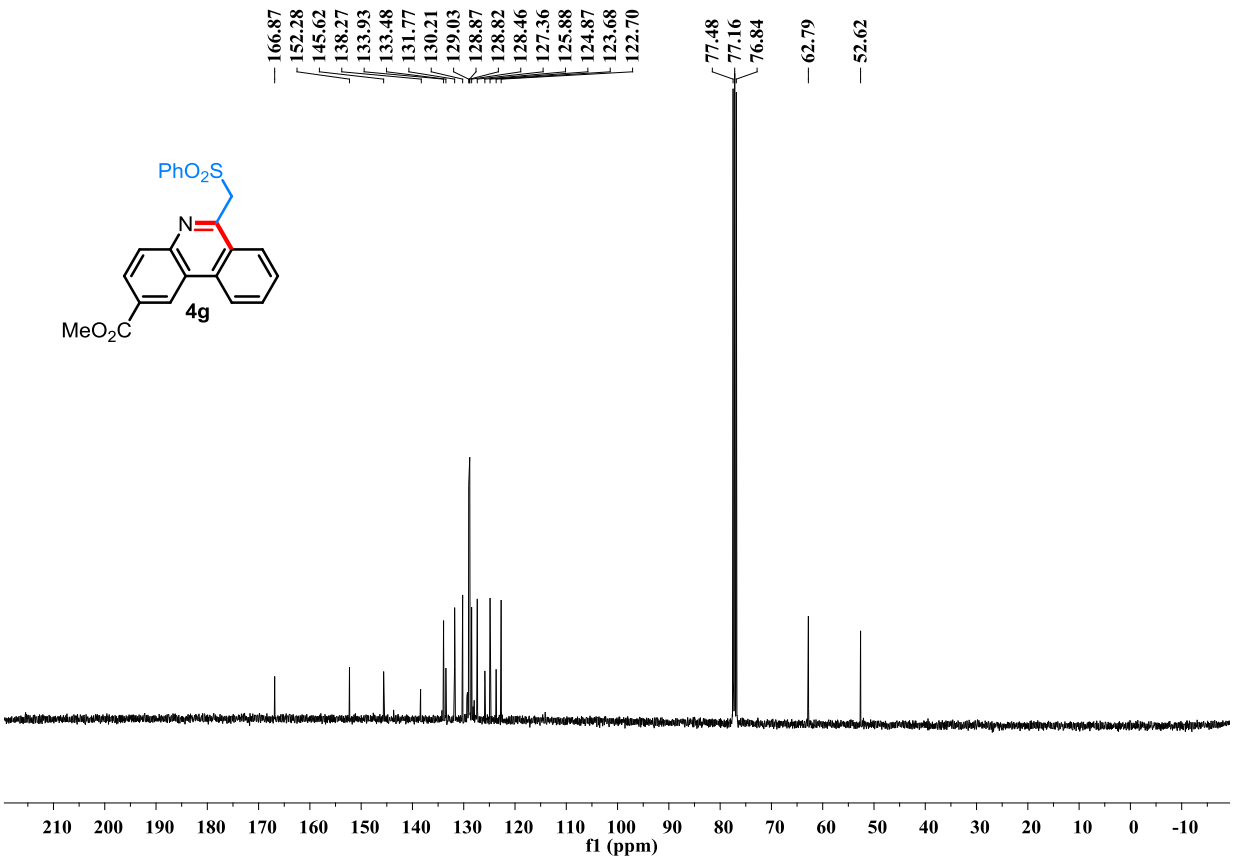
0.98
1.01
1.02
1.10
2.18
2.21
1.19
2.20
1.97
2.93
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


166.87
152.28
145.62
138.27
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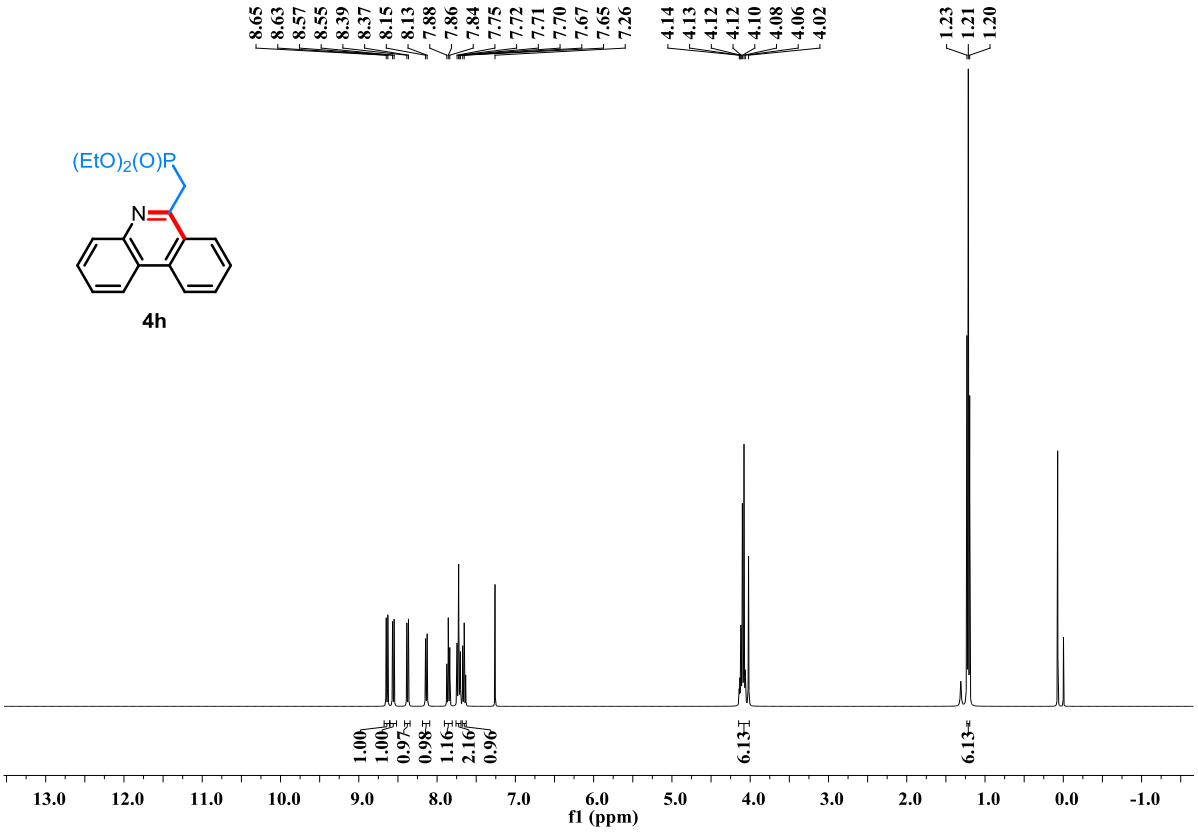
133.48
131.77
130.21
129.03
128.87
128.82
128.46
127.36
125.88
124.87
123.68
122.70
77.48
77.16
76.84
62.79
52.62
PhO2S
N
4g
MeO2C
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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8.63
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8.55
8.39
8.37
8.15
8.13
7.88
7.86
7.84
7.75
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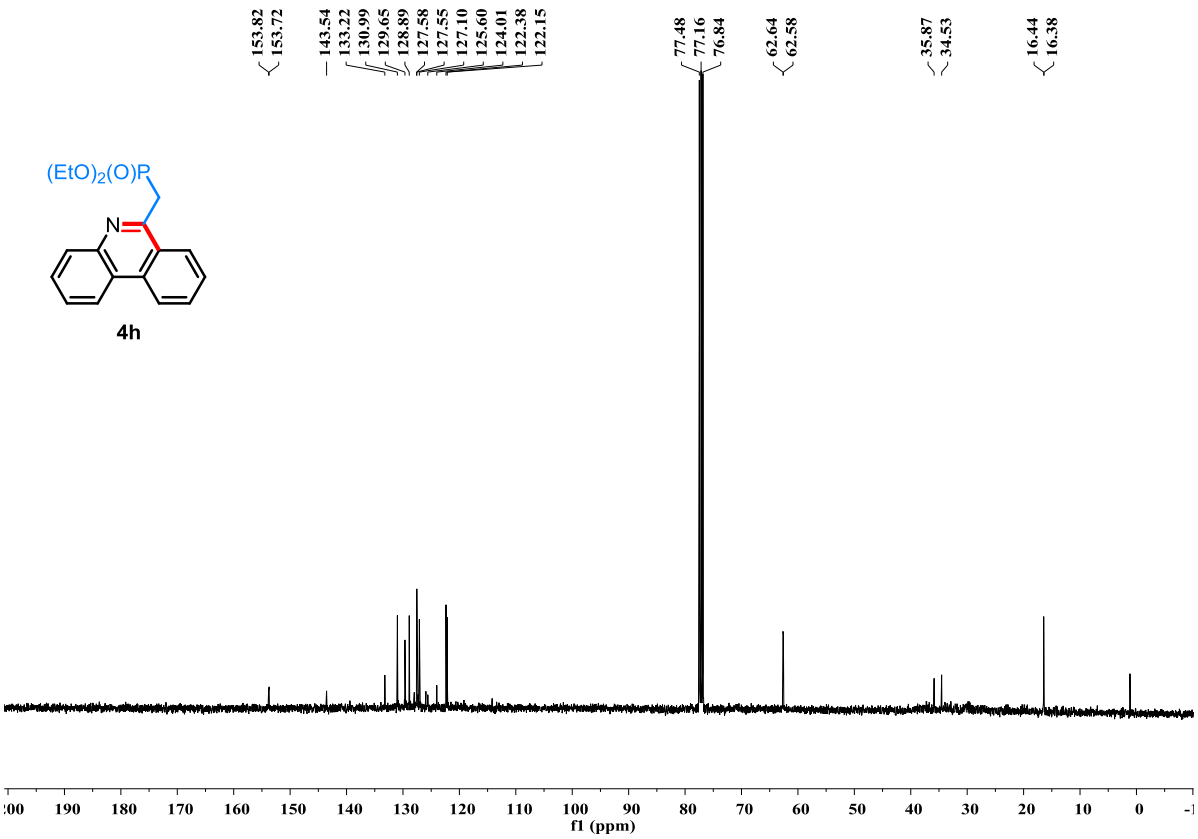
7.71
7.70
7.67
7.65
7.26
4.14
4.13
4.12
4.12
4.10
4.08
4.06
4.02
1.23
1.21
1.20
(EtO)2(O)P
N
4h
1.00
1.00
0.97
0.98
1.16
2.16
0.96
6.13
6.13
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)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143.54
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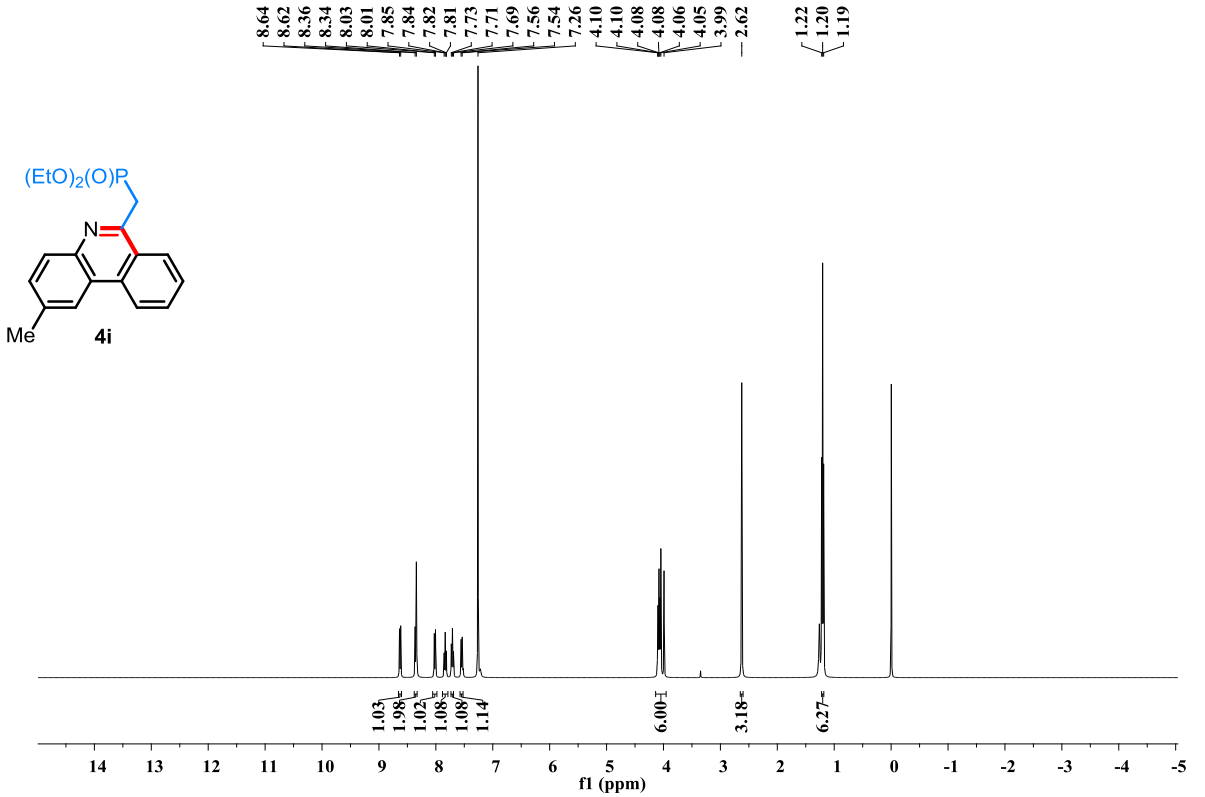
130.99
129.65
128.89
127.58
127.55
127.10
125.60
124.01
122.38
122.15
77.48
77.16
76.84
62.64
62.58
35.87
34.53
16.44
16.38
(EtO)2(O)P
N
4h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.36
8.34
8.03
8.01
7.85
7.84
7.82
7.81
7.73
7.71
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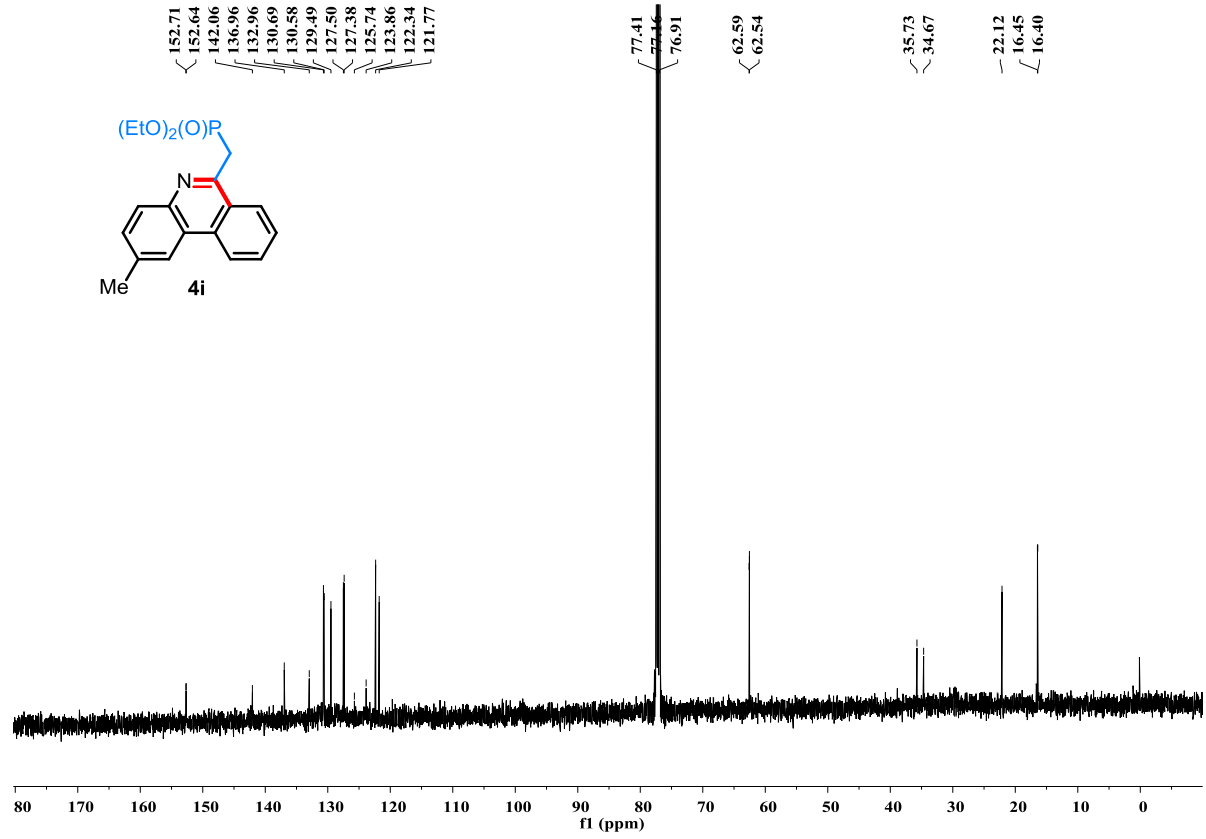
7.56
7.54
7.26
4.10
4.10
4.08
4.08
4.06
4.05
3.99
2.62
1.22
1.20
1.19
(EtO)2(O)P
N
Me
4i
1.03
1.98
1.02
1.08
1.08
1.14
6.00
3.18
6.27
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)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152.64
142.06
136.96
132.96
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130.58
129.49
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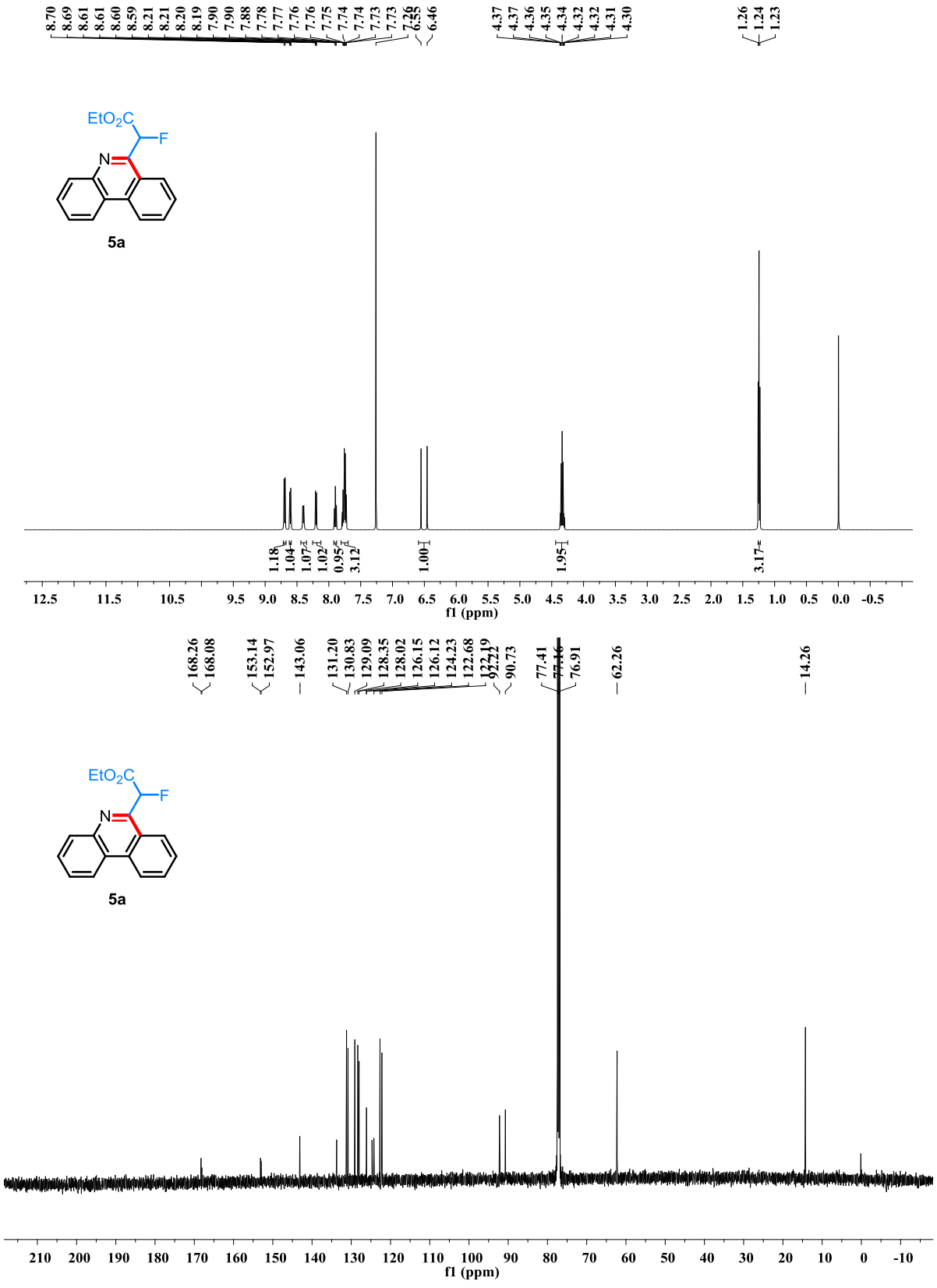
127.38
125.74
123.86
122.34
121.77
77.41
77.16
76.91
62.59
62.54
35.73
34.67
22.12
16.45
16.40
(EtO)2(O)P
N
Me
4i
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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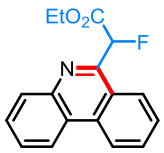
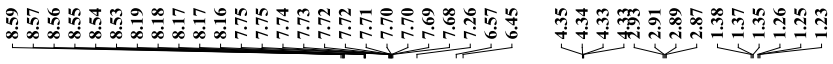
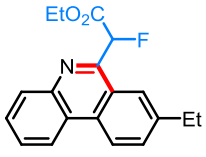
8.61
8.61
8.60
8.59
8.21
8.21
8.20
8.19
7.90
7.90
7.88
7.78
7.77
7.76
7.76
7.75
7.74
7.74
7.73
7.73
7.26
6.55
6.46
4.37
4.37
4.36
4.35
4.34
4.32
4.32
4.31
4.30
1.26
1.24
1.23
EtO2C
F
N
5a
1.18
1.04
1.07
1.02
0.95
3.12
1.00
1.95
3.17
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
168.26
168.08
153.14
152.97
143.06
131.20
130.83
129.09
128.35
128.02
126.15
126.12
124.23
122.68
122.19
90.73
77.41
77.16
76.91
62.26
14.26
EtO2C
F
N
5a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


EtO2C
F
N

5a

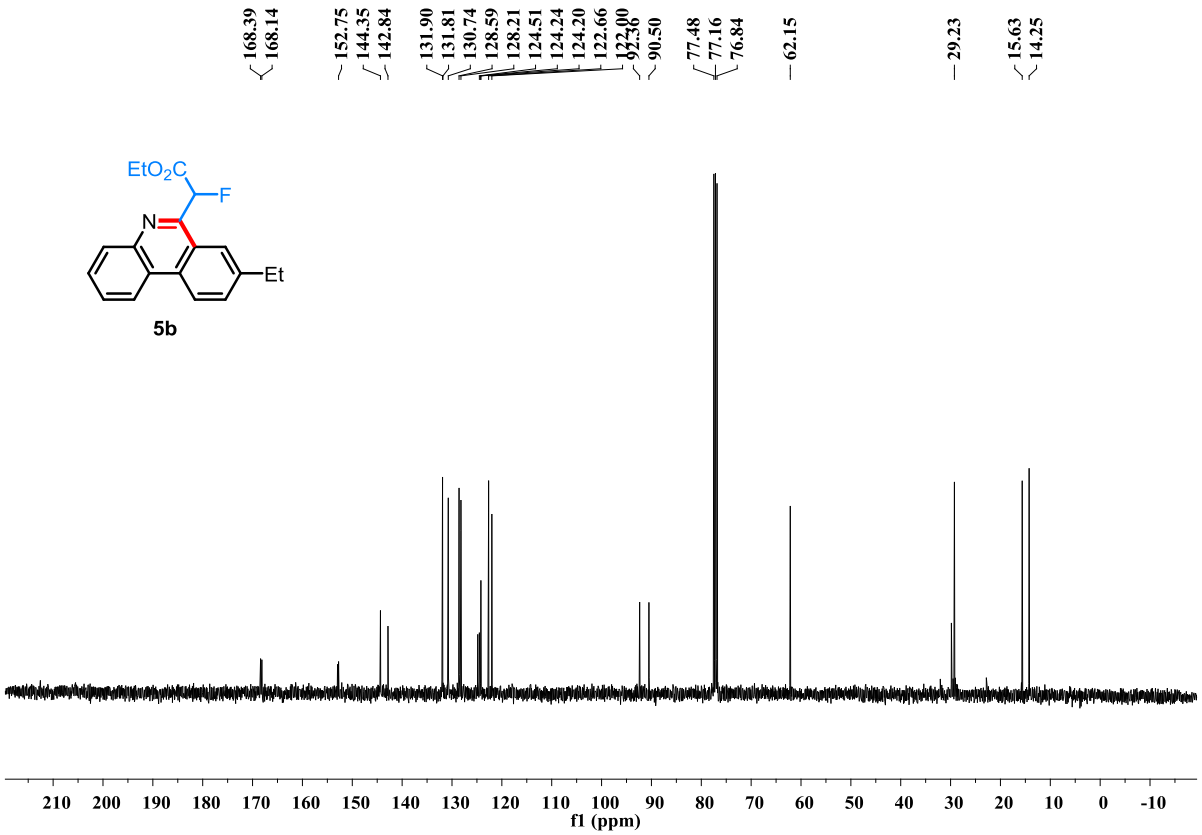
-180.71
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

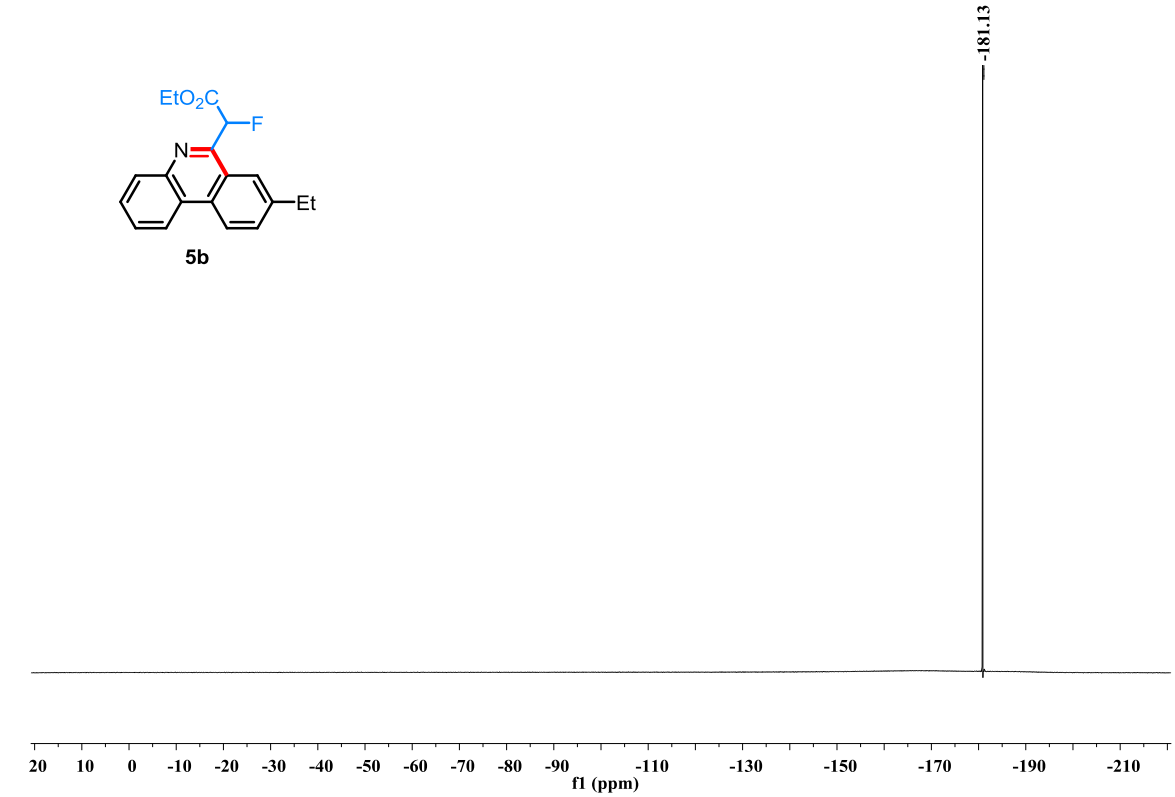

8.59 8.57 8.56 8.55 8.54 8.53 8.19 8.18 8.17 8.17 8.16 7.75 7.75 7.74 7.73 7.72 7.72 7.71 7.70 7.70 7.69 7.68 7.26 6.57 6.45
4.35 4.34 4.33 2.91 2.89 2.87 1.38 1.37 1.35 1.26 1.25 1.23


EtO2C
F
N
Et

5b

2.03 1.99 2.91 1.00 1.98 2.08 2.99 3.20
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)


168.39
168.14
152.75
144.35
142.84
131.90
131.81
130.74
128.59
128.21
124.51
124.24
124.20
122.66
122.00
90.50
77.48
77.16
76.84
62.15
29.23
15.63
14.25
EtO2C
F
N
Et
5b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-181.13
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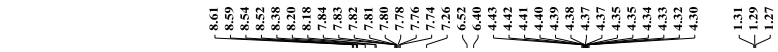
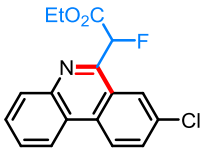
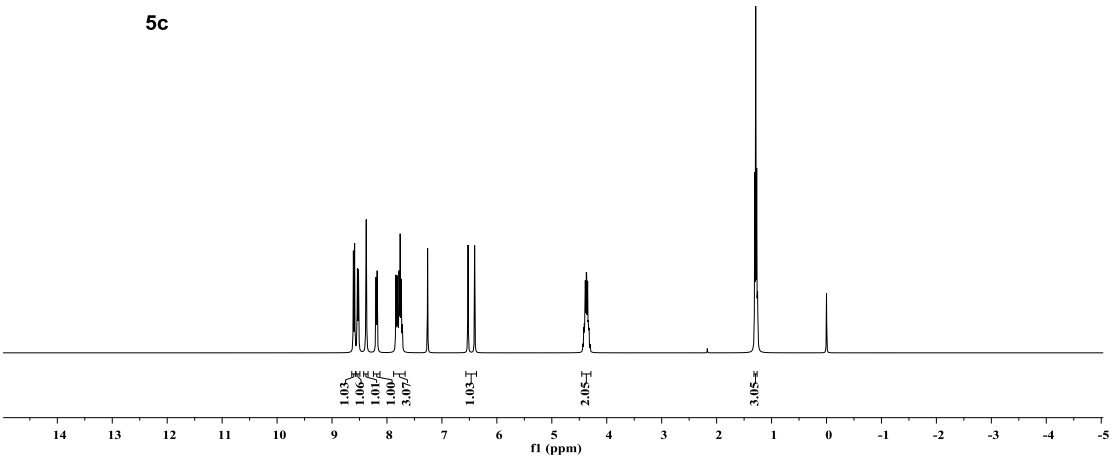
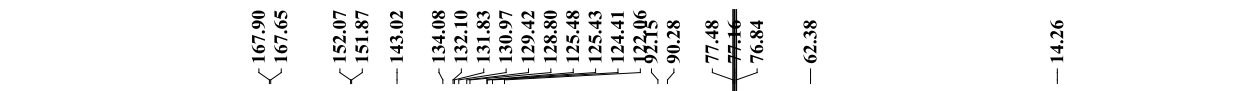
F
N
Et
5b
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)


8.61
8.59
8.54
8.52
8.38
8.20
8.18
7.84
7.83
7.82
7.81
7.80
7.78
7.76
7.74
7.26
6.52
6.40
4.43
4.42
4.41
4.40
4.39
4.38
4.37
4.37
4.35
4.35
4.34
4.33
4.32
4.30
1.31
1.29
1.27


EtO2C
F
N
Cl

5c


1.03
1.06
1.01
1.01
1.00
3.07
1.03
2.05
3.05
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
f1 (ppm)


167.90
167.65
152.07
151.87
143.02
134.08
132.10
131.83
130.97
129.42
128.80
125.48
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124.41
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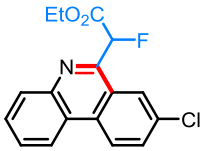
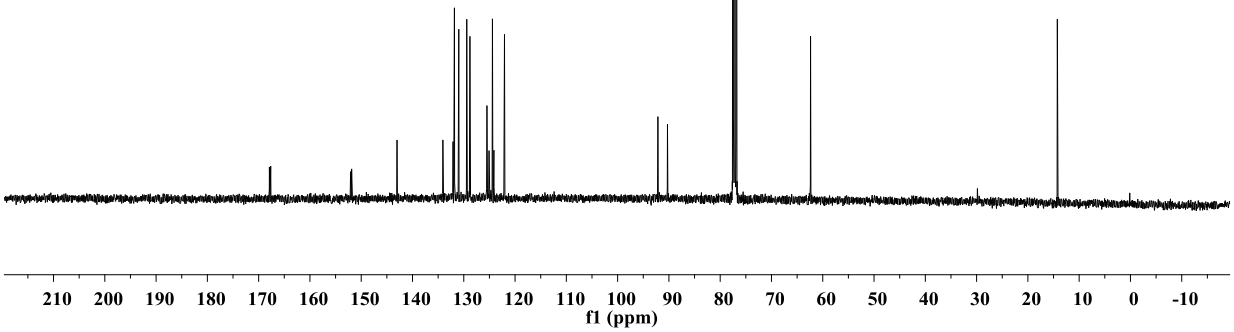
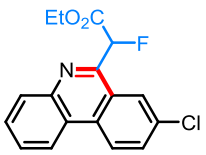
92.15
90.28
77.48
77.16
76.84
62.38
14.26


EtO2C
F
N
Cl

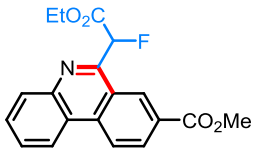
5c


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


EtO2C
F
N
Cl

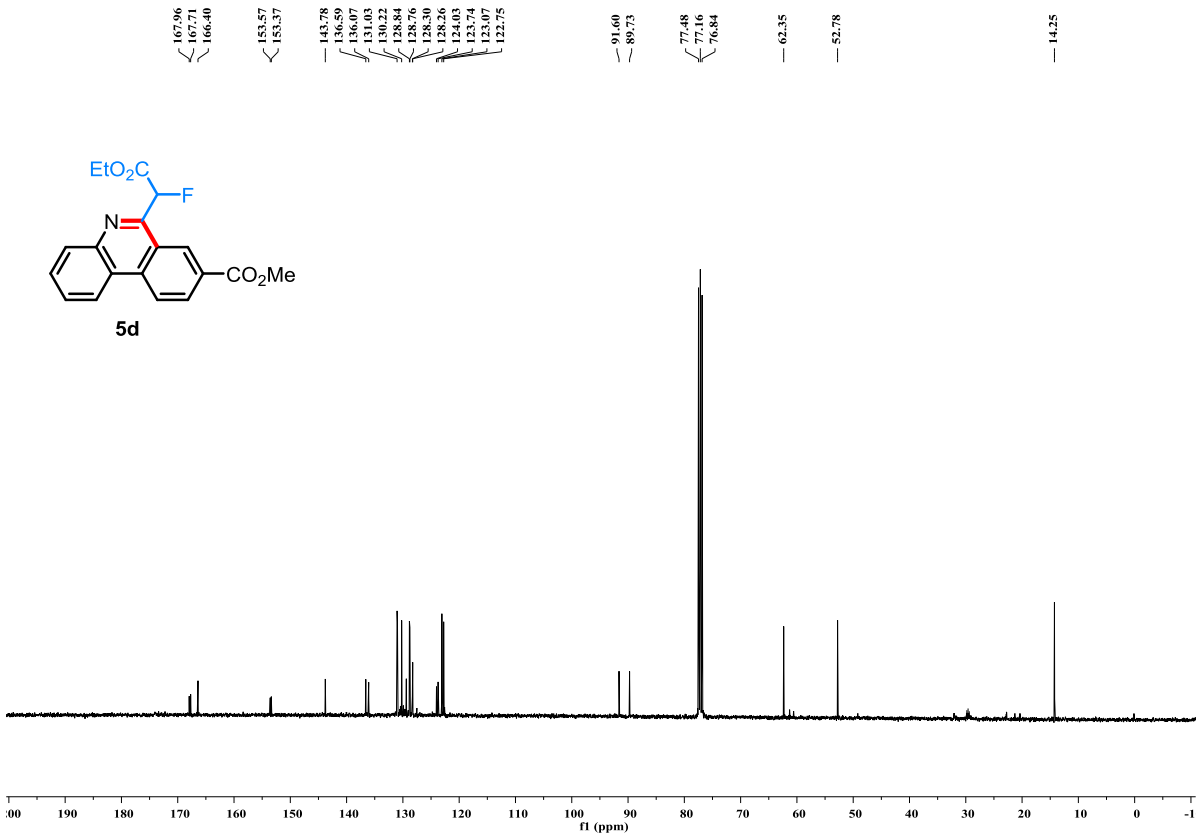
5c

-180.86
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)


EtO2C
F
N
CO2Me

5d

9.09 8.73 8.71 8.61 8.59 8.49 8.47 8.22 8.20 7.84 7.82 7.80 7.78 7.76 7.74 7.26 6.65 6.53
4.40 4.38 4.37 4.35 4.04
1.32 1.30 1.29
1.08 1.07 1.10 1.02 1.00 2.03 1.02 1.93 3.04 3.03
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)


167.96
167.71
166.40
153.57
153.37
143.78
136.59
136.07
131.03
130.22
128.84
128.76
128.30
128.26
124.03
123.74
123.07
122.75
91.60
89.73
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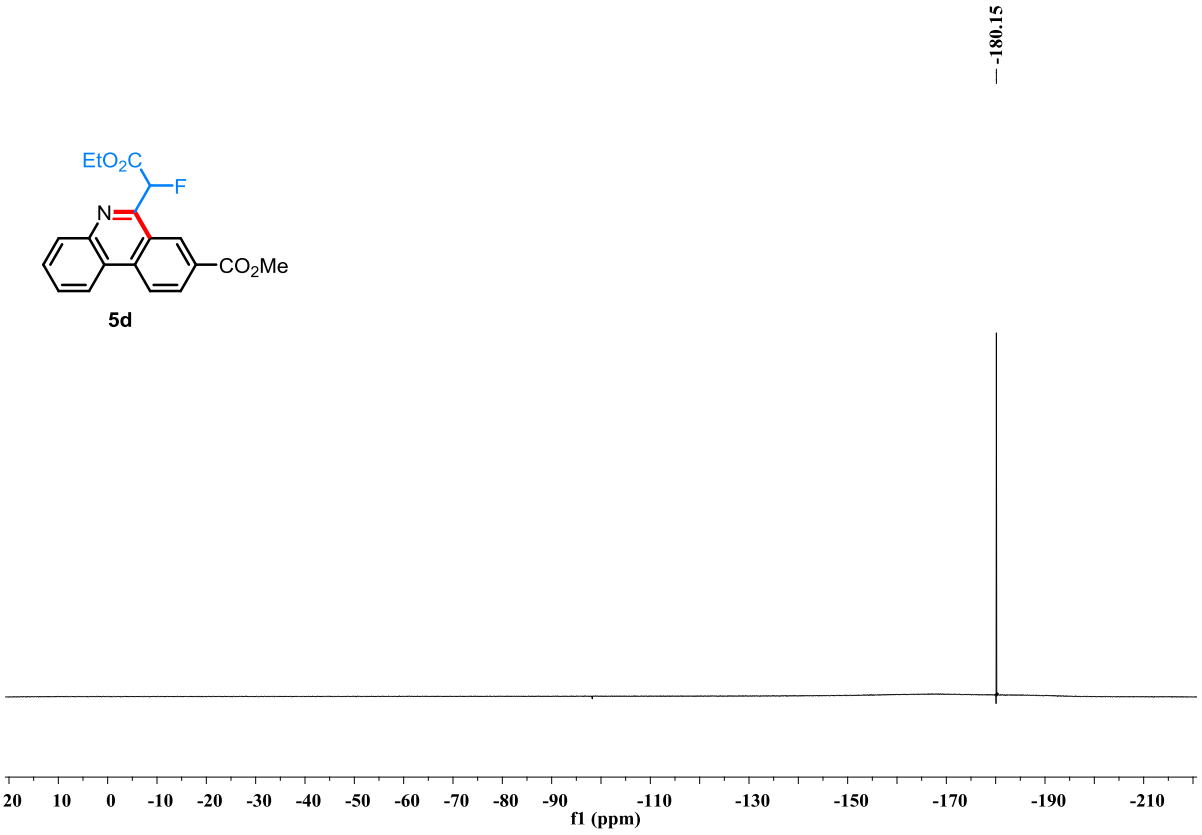
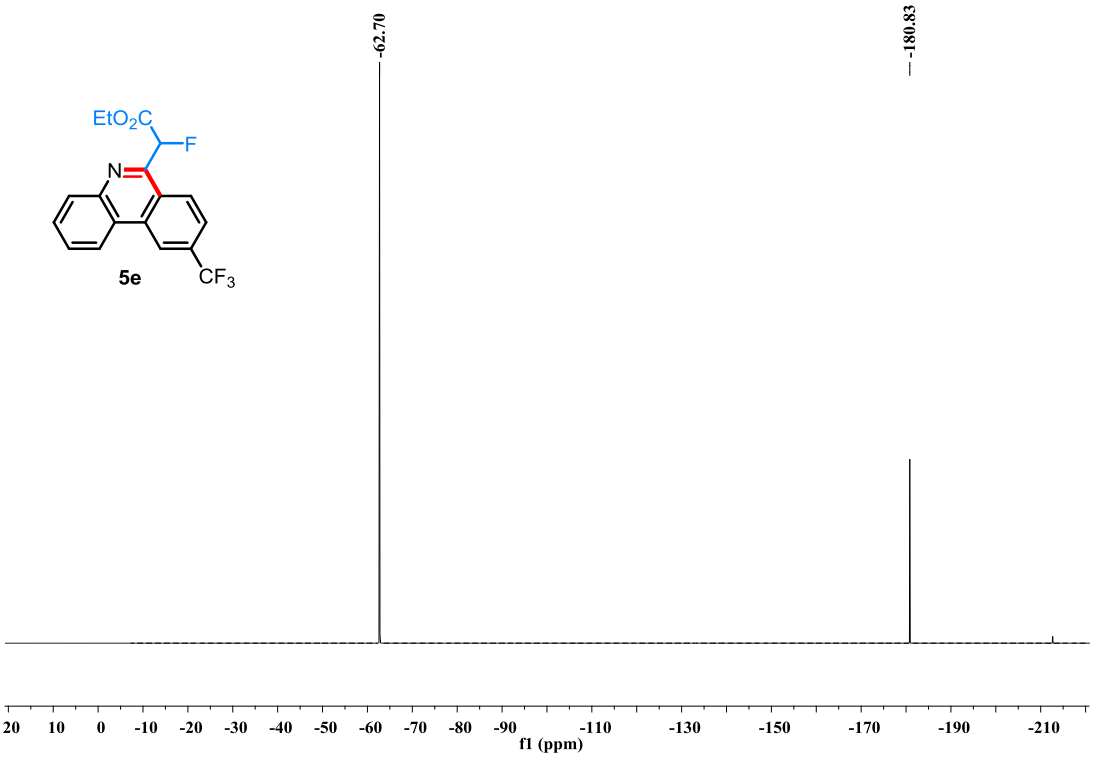
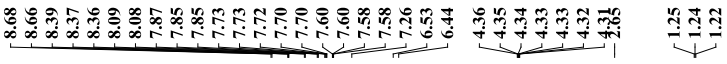
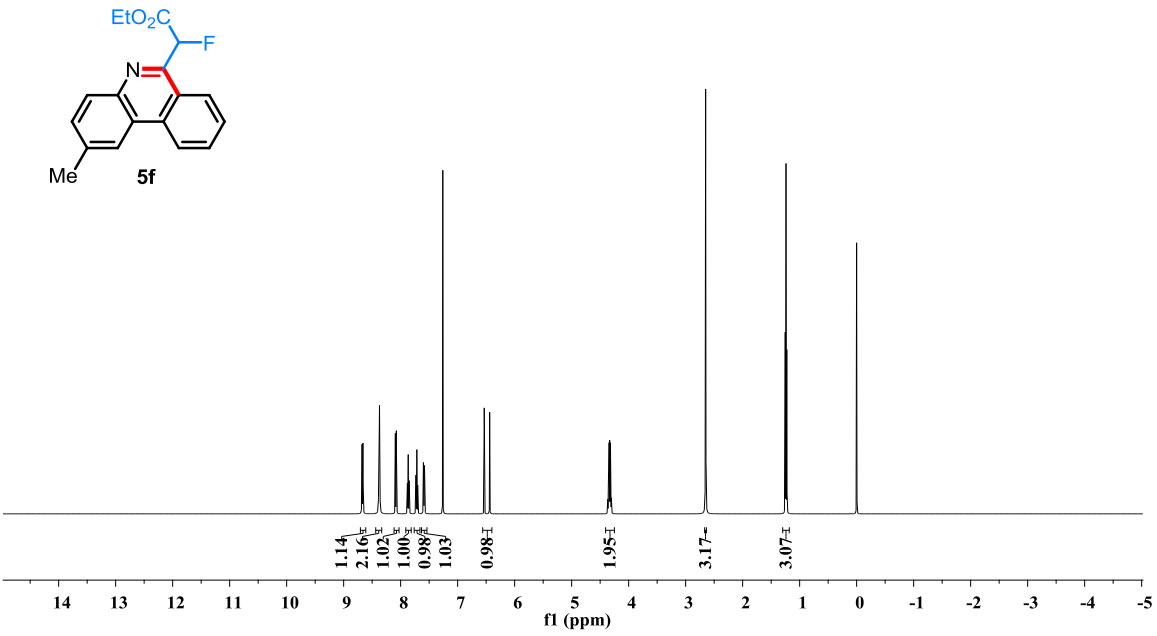
76.84
62.35
52.78
14.25
EtO2C
F
N
CO2Me
5d
f1 (ppm)

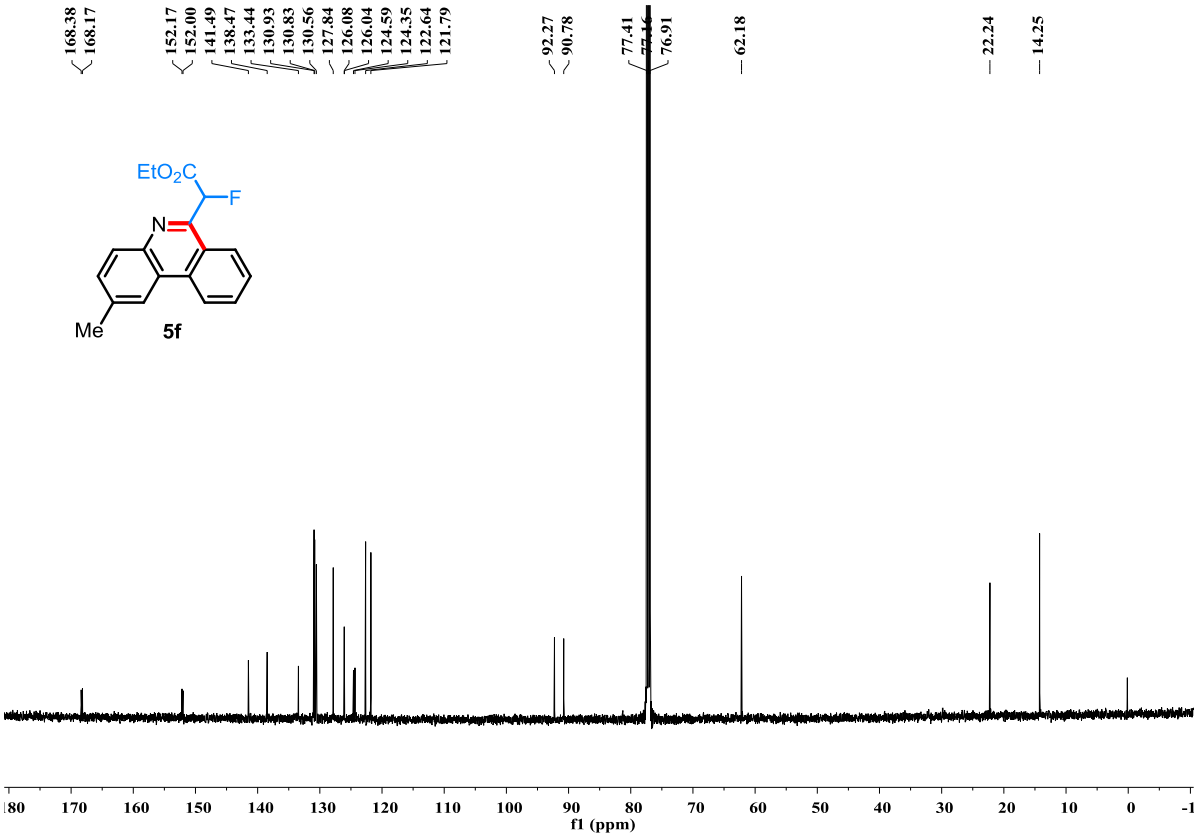

-180.15
EtO2C
F
N
CO2Me
5d
f1 (ppm)


8.95
8.63
8.63
8.62
8.61
8.56
8.55
8.54
8.54
8.25
8.25
8.24
8.23
7.94
7.92
7.92
7.86
7.84
7.84
7.83
7.83
7.82
7.82
7.80
7.80
7.26
6.55
6.45
4.40
4.39
4.37
4.36
4.33
4.33
4.32
4.31
4.31
4.30
4.28
1.27
1.26
1.25
EtO2C
F
N
5e
CF3
1.09
1.11
1.07
1.01
1.06
1.84
1.00
2.02
3.07
f1 (ppm)
167.91
167.66
152.65
152.45
143.41
133.56
131.10
130.04
129.09
127.38
127.33
123.95
122.23
120.26
120.22
92.43
90.56
77.48
77.16
76.84
62.48
14.25
EtO2C
F
N
5e
CF3
f1 (ppm)


EtO2C
F
N
CF3
5e
-62.70
-180.83
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)


8.68 8.66 8.39 8.37 8.36 8.09 8.08 7.87 7.85 7.85 7.73 7.73 7.72 7.70 7.70 7.60 7.60 7.58 7.58 7.26 6.53 6.44
4.36 4.35 4.34 4.33 4.32 4.31
2.85
1.25 1.24 1.22


EtO2C
F
N
Me
5f
1.14 2.16 1.02 1.00 0.98 1.03 0.98 1.95 3.17 3.07
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5
f1 (ppm)


168.38
168.17
152.17
152.00
141.49
138.47
133.44
130.93
130.83
130.56
127.84
126.08
126.04
124.59
124.35
122.64
121.79
92.27
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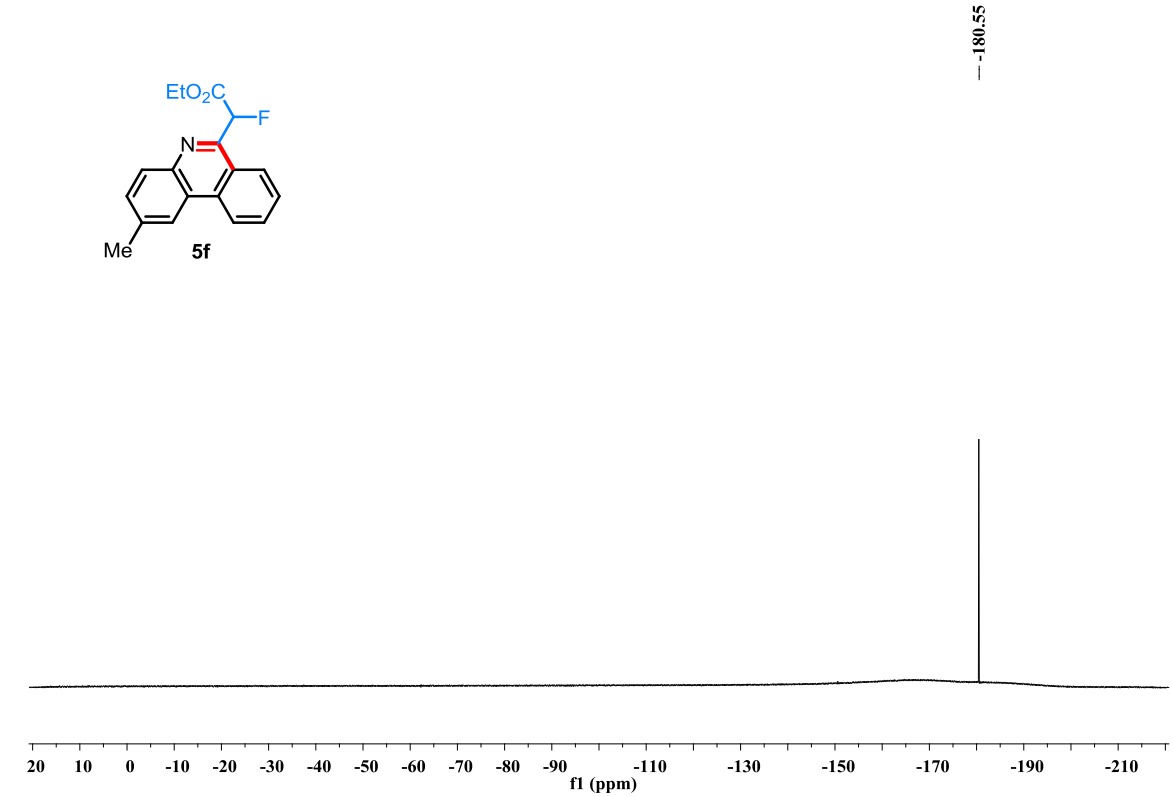
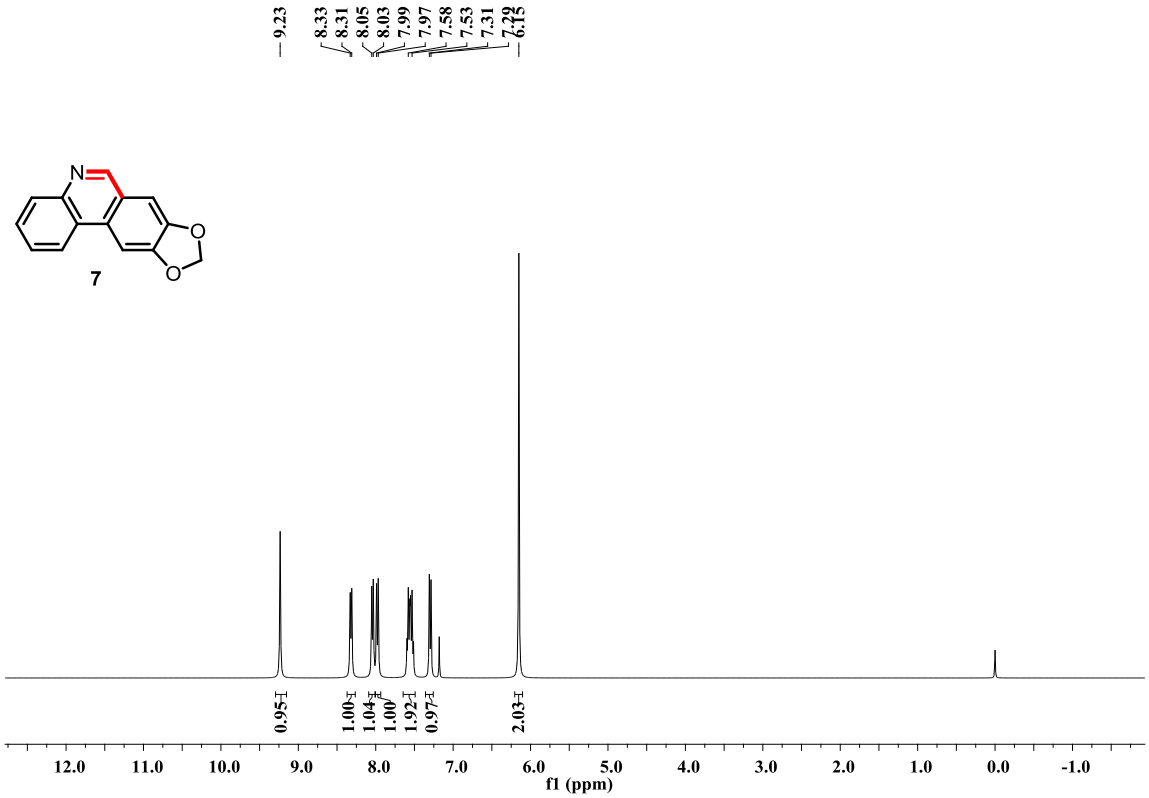
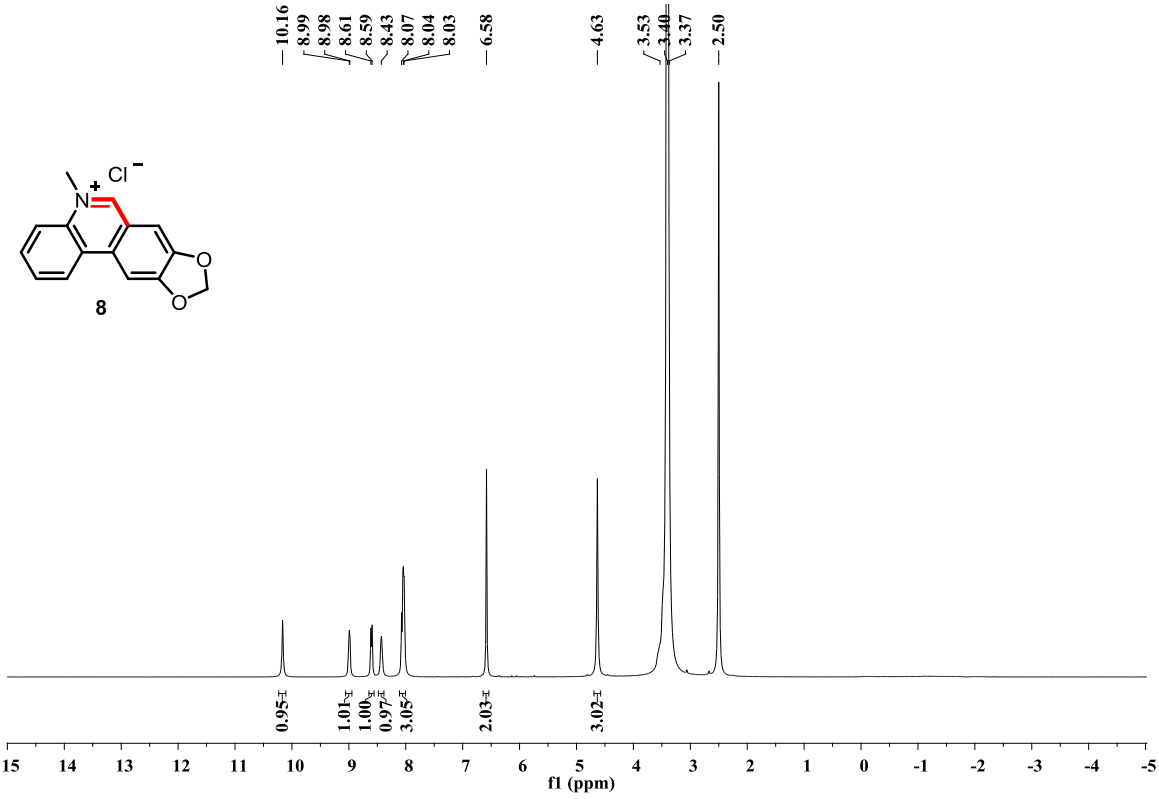
76.91
62.18
22.24
14.25
EtO2C
F
N
Me
5f
fl (ppm)


-180.55
EtO2C
F
N
Me
5f
fl (ppm)


9.23
8.33
8.31
8.05
8.03
7.99
7.97
7.58
7.53
7.31
7.29
6.15
N
O
O
7
0.95
1.00
1.04
1.00
1.92
0.97
2.03
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

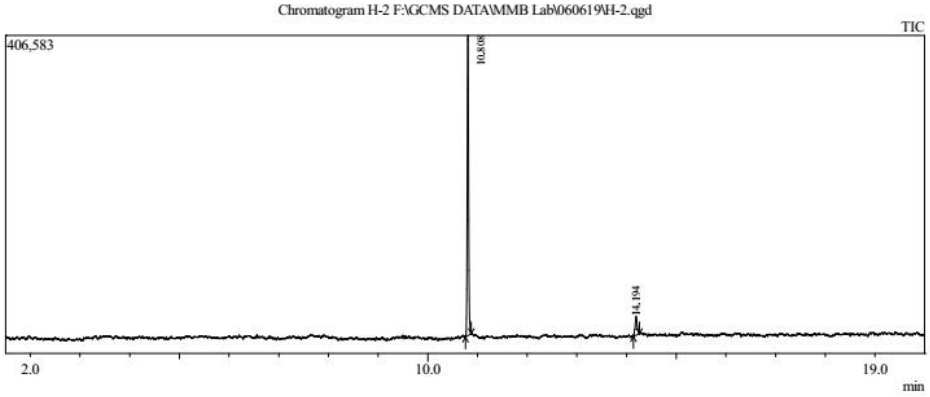

10.16
8.99
8.98
8.61
8.59
8.43
8.07
8.04
8.03
6.58
4.63
3.53
3.40
3.37
2.50
Cl
N
O
O
8
0.95
1.01
1.00
0.97
3.05
2.03
3.02
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
f1 (ppm)

GC-MS Traces

Sample Information

Analyzed by	: Admin
Analyzed	: 6/6/2019 6:58:52 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: H-2
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 2
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\060619\H-2.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\060619\H-2.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\040619.qgt
Modified by	: Admin
Modified	: 6/6/2019 8:26:51 PM

Chromatogram H-2 F:\GCMS DATA\MMB Lab\060619\H-2.qgd



Peak Report TIC

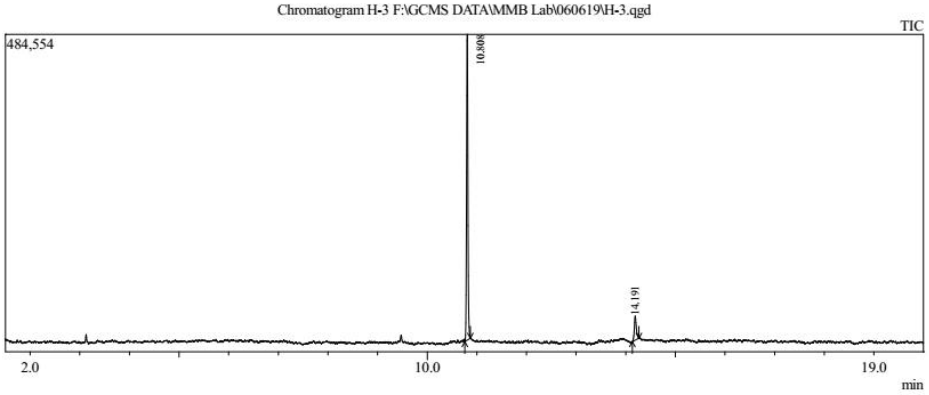
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.808	10.760	10.875	725808	91.18	383674	94.04	1.89		2-Aminobiphenyl
2	14.194	14.140	14.260	70178	8.82	24301	5.96	2.89		Phenanthridine
				795986	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/6/2019 7:25:25 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: H-3
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 3
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\060619\H-3.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\060619\H-3.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\040619.qgt
Modified by	: Admin
Modified	: 6/6/2019 8:26:28 PM

Chromatogram H-3 F:\GCMS DATA\MMB Lab\060619\H-3.qgd



Peak Report TIC

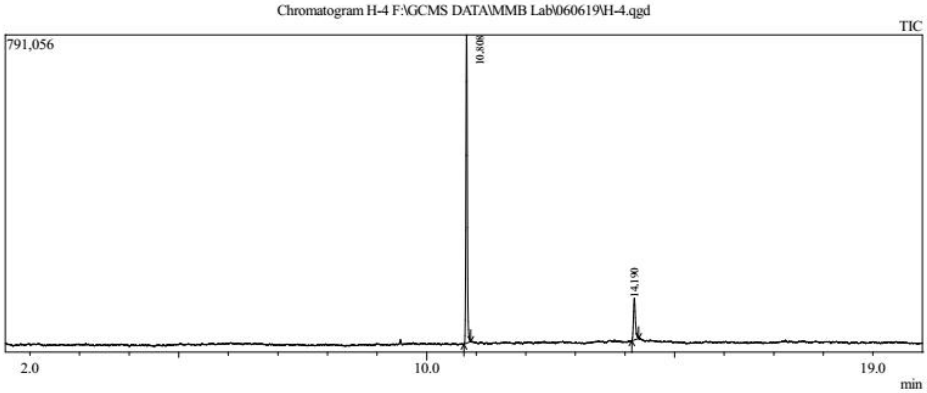
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.808	10.760	10.870	867970	89.46	466675	92.69	1.86		2-Aminobiphenyl
2	14.191	14.135	14.265	102289	10.54	36783	7.31	2.78		PHENANTHRIDINl
				970259	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/6/2019 9:11:42 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: H-4
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 7
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\060619\H-4.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\060619\H-4.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\040619.qgt
Modified by	: Admin
Modified	: 6/7/2019 9:30:53 AM

Chromatogram H-4 F:\GCMS DATA\MMB Lab\060619\H-4.qgd



Peak Report TIC

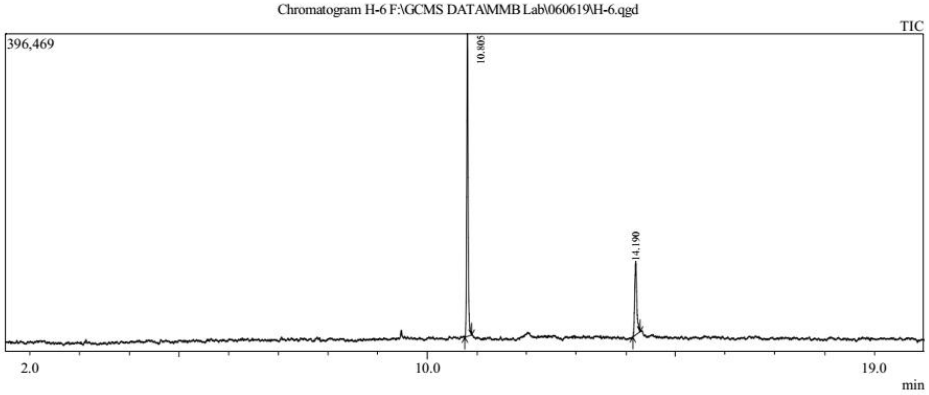
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.808	10.755	10.890	1493999	83.68	768683	88.05	1.94		2-Aminobiphenyl
2	14.190	14.140	14.275	291308	16.32	104312	11.95	2.79		PHENANTHRIDINI
				1785307	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/6/2019 10:25:47 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: H-6
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 9
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\060619\H-6.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\060619\H-6.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\040619.qgt
Modified by	: Admin
Modified	: 6/19/2019 1:42:10 PM

Chromatogram H-6 F:\GCMS DATA\MMB Lab\060619\H-6.qgd



Peak Report TIC

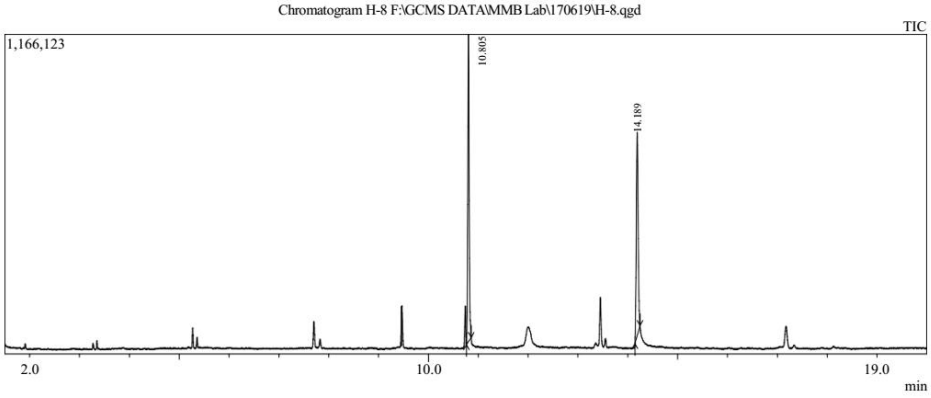
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.805	10.755	10.890	708034	69.76	357586	77.22	1.98		2-Aminobiphenyl
2	14.190	14.135	14.280	306922	30.24	105471	22.78	2.91		PHENANTHRIDIN
				1014956	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/17/2019 9:54:06 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: H-8
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 4
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\H-8.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\H-8.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:14:30 PM

Chromatogram H-8 F:\GCMS DATA\MMB Lab\170619\H-8.qgd



Peak Report TIC

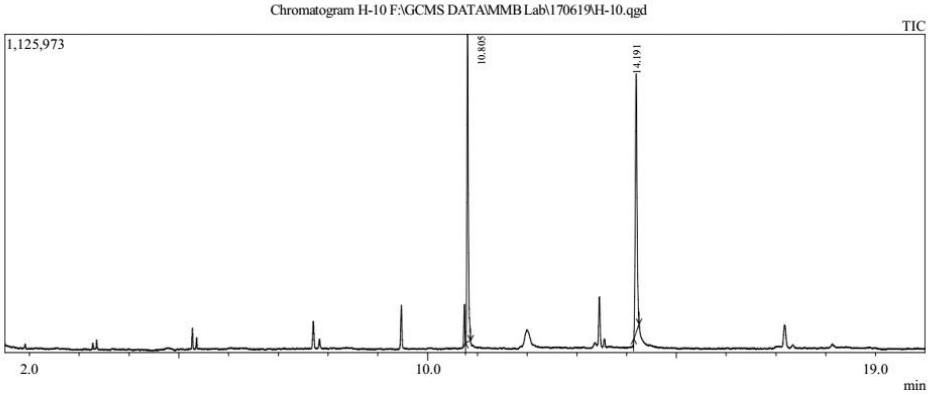
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.805	10.765	10.860	1872801	47.79	1012324	55.09	1.85		2-Aminobiphenyl
2	14.189	14.145	14.245	2046011	52.21	825002	44.91	2.48		Phenanthridine
				3918812	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/17/2019 10:20:37 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: H-10
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 5
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\H-10.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\H-10.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:14:56 PM

Chromatogram H-10 F:\GCMS DATA\MMB Lab\170619\H-10.qgd



Peak Report TIC

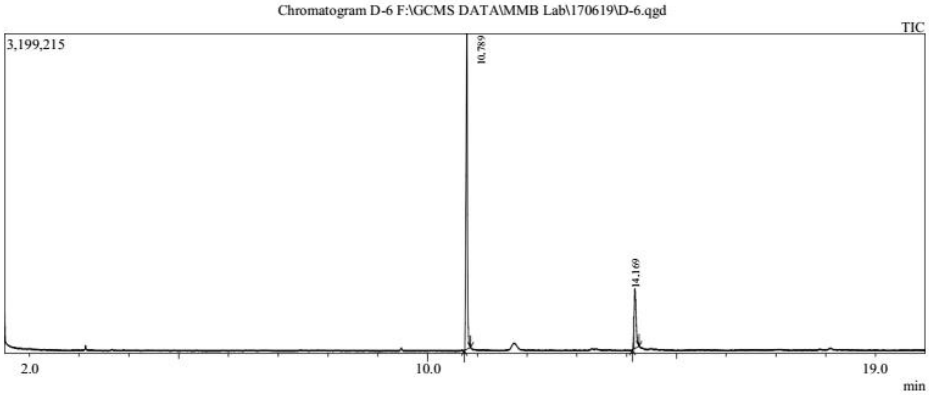
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.805	10.765	10.865	1707813	39.19	852863	51.86	1.98		2-Aminobiphenyl
2	14.191	14.145	14.250	2649962	60.81	791686	48.14	3.34		Phenanthridine
				4357775	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/18/2019 12:48:16 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: D-6
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 7
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\D-6.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\D-6.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:11:53 PM

Chromatogram D-6 F:\GCMS DATA\MMB Lab\170619\D-6.qgd



Peak Report TIC

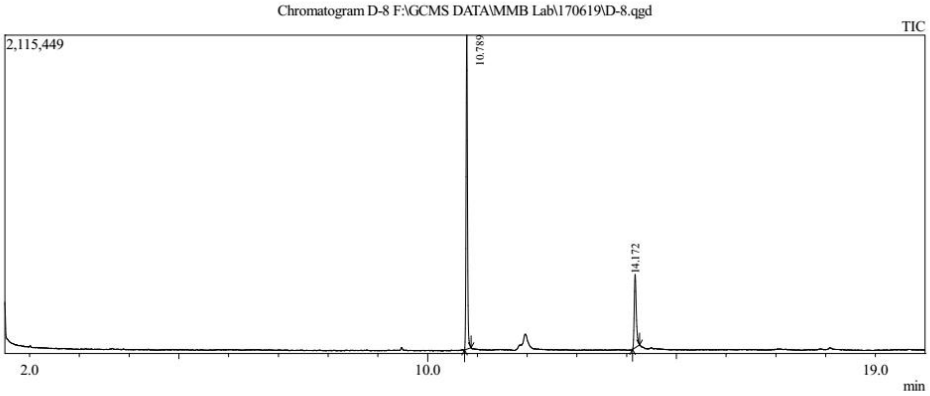
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.789	10.735	10.865	6516900	83.74	3158010	84.08	1.95		Julolidine
2	14.169	14.120	14.250	1265402	16.26	597867	15.92	2.73		9H-PYRIDO[2,3-B]
				7782302	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/18/2019 1:14:57 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: D-8
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 8
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\D-8.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\D-8.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:11:31 PM

Chromatogram D-8 F:\GCMS DATA\MMB Lab\170619\D-8.qgd



Peak Report TIC

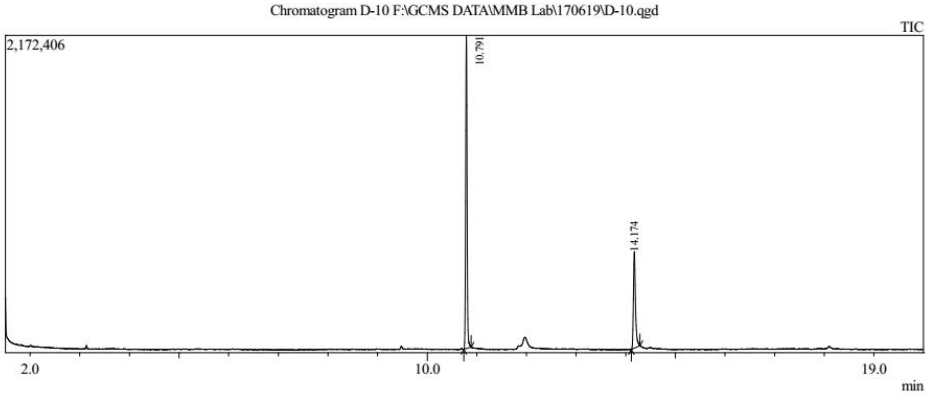
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.789	10.740	10.875	4056756	74.64	2085570	81.06	1.95		Julolidine
2	14.172	14.120	14.260	1378557	25.36	487186	18.94	2.83		9H-PYRIDO[2,3-B]I
				5435313	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/18/2019 1:41:30 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: D-10
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 9
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\D-10.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\D-10.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:12:07 PM

Chromatogram D-10 F:\GCMS DATA\MMB Lab\170619\D-10.qgd



Peak Report TIC

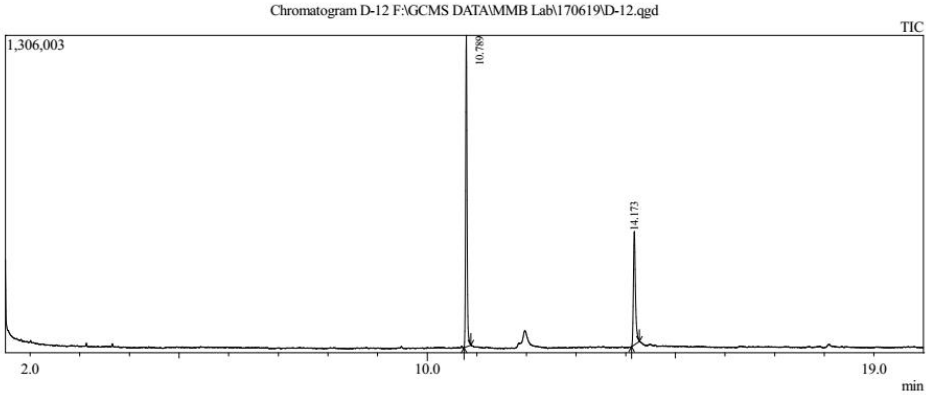
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.791	10.735	10.885	4218142	69.18	2143094	76.42	1.97		Julolidine
2	14.174	14.115	14.285	1879181	30.82	661159	23.58	2.84		9H-PYRIDO[2,3-B]I
				6097323	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/18/2019 2:08:10 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: D-12
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 10
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\D-12.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\D-12.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:12:21 PM

Chromatogram D-12 F:\GCMS DATA\MMB Lab\170619\D-12.qgd



Peak Report TIC

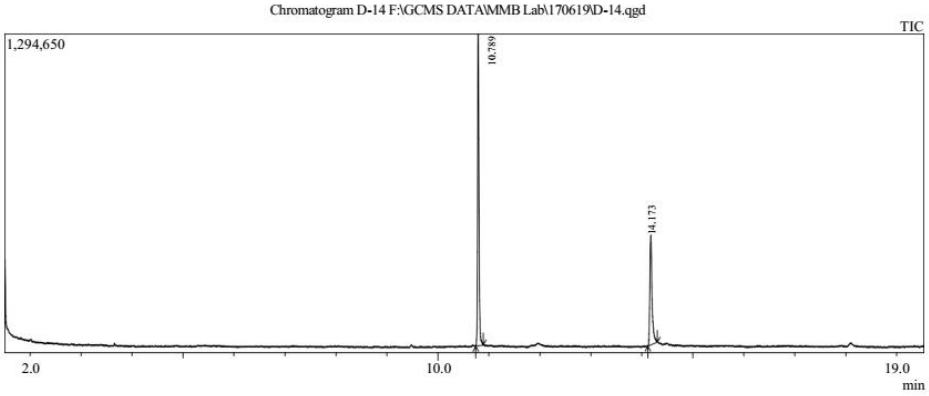
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.789	10.740	10.880	2506846	65.23	1281121	73.27	1.96		Julolidine
2	14.173	14.120	14.275	1335965	34.77	467397	26.73	2.86		9H-PYRIDO[2,3-B]I
				3842811	100.00					

Library

Sample Information

Analyzed by	: Admin
Analyzed	: 6/18/2019 2:34:48 PM
Sample Type	: Unknown
Level #	: 1
Sample Name	: D-14
Sample ID	:
IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 11
Injection Volume	: 1.00
Data File	: F:\GCMS DATA\MMB Lab\170619\D-13.qgd
Org Data File	: F:\GCMS DATA\MMB Lab\170619\D-13.qgd
Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Org Method File	: F:\GCMS Method\MMB-DEEPAN.qgm
Report File	: F:\Report\sample report.qgr
Tuning File	: F:\Tuning\2019\june2019\130619.qgt
Modified by	: Admin
Modified	: 6/18/2019 11:12:40 PM

Chromatogram D-14 F:\GCMS DATA\MMB Lab\170619\D-14.qgd



Peak Report TIC

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	10.789	10.740	10.890	2390722	63.08	1226011	72.30	1.95		Julolidine
2	14.173	14.115	14.305	1399261	36.92	469550	27.70	2.98		9H-PYRIDO[2,3-B]
				3789983	100.00					

Library