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Supporting Information

Direct Access to Acylated Quinoxalin-2(1*H*)-one *N*-oxides Enabled by Cu(I)/TBHP System

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Contents

1. General information

All reagents and starting materials were purchased from commercial sources and used without treatment unless otherwise indicated. All the solvents were dried and re-distilled upon use. NMR spectra were obtained on a Bruker AMX 400 system using chloroform-*d* ($CDCl_3$) as solvent. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded at 400 MHz and 100 MHz respectively. All chemical shifts were given in ppm with TMS at 0.0 ppm, and all coupling constants (*J* values) were reported in Hertz (Hz). Liquid Chromatography- High- Resolution Mass Spectrometry was recorded on the Bruker MicrOTOF QII instrument. Column chromatography was performed on silica gel (100-200 mesh or 200-300 mesh) with ethyl acetate and petroleum ether as eluents.

2. Preparation of starting materials

Step 1 Typical procedure: Potassium carbonate (6.0 mmol, 1.2 equiv.) and the corresponding halide (8.0 mmol, 1.6 equiv.) were added into a solution of quinoxalin-2(1*H*)-ones (5.0 mmol, 1.0 equiv.) in DMF (25 mL). The reaction mixture was stirred at room temperature overnight. Then water was added and the resulting mixture was extracted with ethyl acetate three times. The combined organic layers were washed with saturated solution of NH_4Cl and NaCl respectively, then dried over Na_2SO_4, filtered and evaporated under reduced pressure.[1] The residue was purified by column chromatography on silica gel to obtain the desired products **1a** to **1i**.

Step 2 Typical procedure: Ethyl 2-oxoacetate (5.5 mmol, 1.1 equiv.) was added into a suspension of substituted *ortho*-phenylenediamine (5.0 mmol, 1.0 equiv.) in ethanol (25 mL). The mixture was stirred at reflux for 4 h, then stirred at room temperature overnight.[2] The precipitated solid was filtered and washed with ethanol, then dried over Na_2SO_4 to give substituted quinoxalinone. Then, the obtained compound was subjected to the procedure as described in step 1) to yield the expected products **1j-1l**.

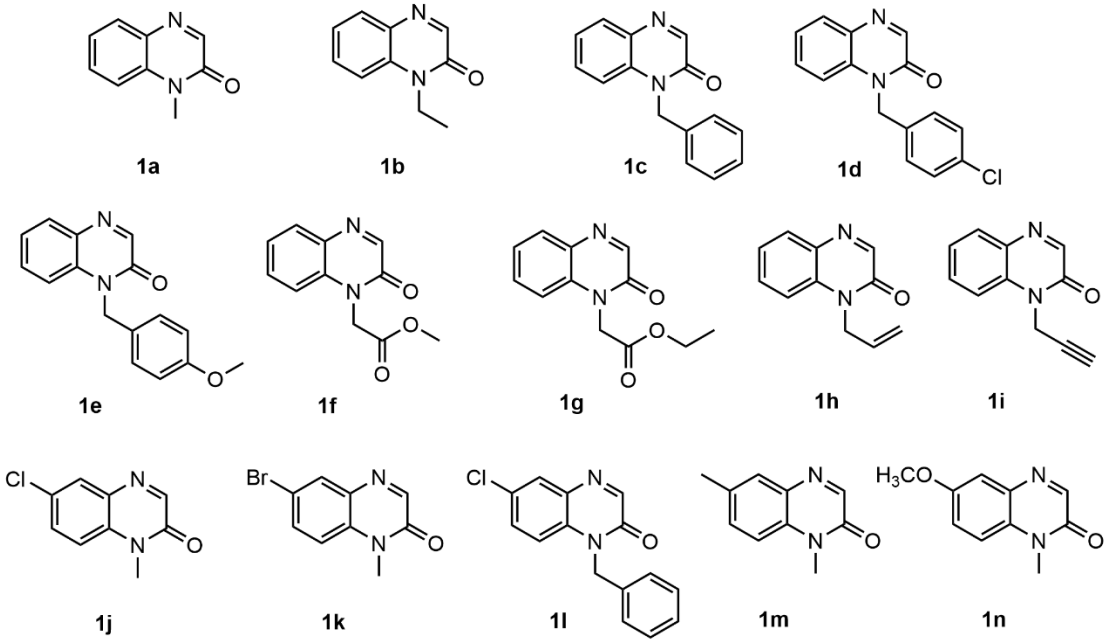


Figure S1 Reacted substrates.

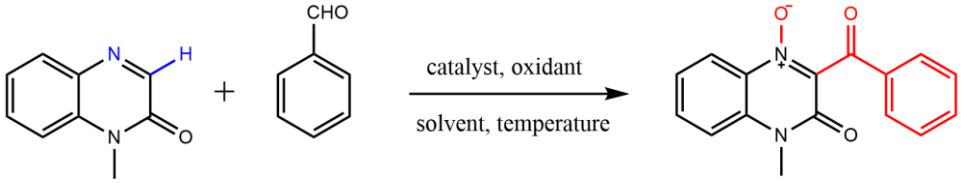
[1] K. Zhang, J. Xu, J. Xiao, R. Zhong, J. Li, *Eur. J. Org. Chem.*, 2023, ***26***, e202201432.

[2] J. W. Yuan, J. H. Fu, S. N. Liu, Y. M. Xiao, P. Mao, L. B. Qu, *Org. Biomol. Chem.*, 2018, ***16***, 3203-3212.

3. Experimental section

3.1 Screening the reaction conditions

Table S1 Screening the reaction conditions [a]



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

Entry	catalyst	solvent	T (℃)	t (h)	Yield [b] (%)
1	CuCl	EtOAc	70	12	24
2	$Cu(OAc)_2$	EtOAc	70	12	16
3	$CuCl_2{\cdot}2H_2O$	EtOAc	70	12	trace
4	CuBr	EtOAc	70	12	22
5	CuI	EtOAc	70	12	19
6	$NiCl_2$	EtOAc	70	12	8
7	$Ni(OTf)_2$	EtOAc	70	12	trace
8	$NiSO_4$	EtOAc	70	12	trace
9	$FeCl_3$	EtOAc	70	12	trace
10	$FeSO_4$	EtOAc	70	12	10
11[c]	CuCl	EtOAc	70	12	n.r
12[d]	CuCl	EtOAc	70	12	n.r
13[e]	CuCl	EtOAc	70	12	n.r
14[f]	CuCl	EtOAc	70	12	n.r
15[g]	CuCl	EtOAc	70	12	21
16[h]	CuCl	EtOAc	70	12	20
17	CuCl	DCM	70	12	17
18	CuCl	DCE	70	12	14
19	CuCl	$PhCF_3$	70	12	15
20	CuCl	HFIP	70	12	19
21	CuCl	DMF	70	12	n.r
22	CuCl	DMSO	70	12	trace
23	CuCl	CH_3NO_2	70	12	7
24	CuCl	THF	70	12	trace
25	CuCl	TFE	70	12	trace
26	CuCl	$PhCH_3$	70	12	11
27	CuCl	1,4-dioxane	70	12	trace
28	CuCl	$CHCl_3$	70	12	14
29	CuCl	EtOAc	70	12	5
30	CuCl	CH_3CN	70	12	9
31	CuCl	EtOAc	60	12	18
32	CuCl	EtOAc	80	12	29
33	CuCl	EtOAc	90	12	25
34	CuCl	EtOAc	100	12	14
35	CuCl	EtOAc	80	6	17
36	CuCl	EtOAc	80	24	42
37	**CuCl**	**EtOAc**	**80**	**36**	**55**
38	CuCl	EtOAc	80	48	50
39[i]	CuCl	EtOAc	80	36	52
40[j]	CuCl	EtOAc	80	36	53
41[k]	CuCl	EtOAc	80	12	6
42[l]	CuCl	EtOAc	80	12	15
43[m]	CuCl	EtOAc	80	12	22
44[n]	CuCl	EtOAc	80	12	26

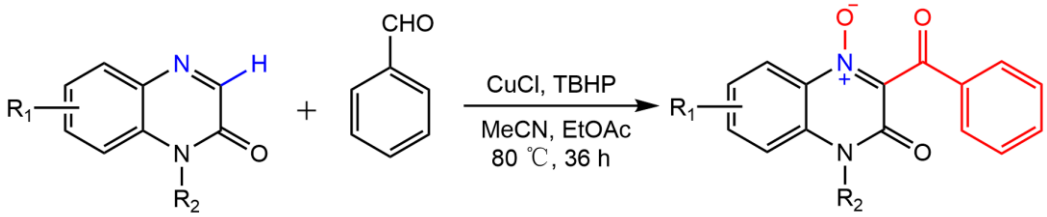
[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (1.0 mmol, 5.0 equiv.), TBHP (5.0-6.0 mol/L

in decane, 8.0 equiv.), catalyst (0.1 equiv.), MeCN (1.0 mL), solvent (1.0 mL) in sealed tube. [b] Isolated yield. [c] DTBP. [d] $K_2S_2O_8$. [e] $(NH_4)_2S_2O_8$. [f] no TBHP. [g] TBHP (5.0-6.0 mol/L in decane, 7.0 equiv.). [h] TBHP (5.0-6.0 mol/L in decane, 9.0 equiv.). [i] under N_2. [j] under O_2. [k] **2a** (0.4 mmol, 2.0 equiv.), [l] **2a** (0.6 mmol, 3.0 equiv.), [m] **2a** (0.8 mmol, 4.0 equiv.), [n] **2a** (1.2 mmol, 6.0 equiv.).

3.2 General procedure for the synthesis of target compounds

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

Quinoxalin-2(1*H*)-one **1a** (0.1 mmol, 1.0 equiv.), aromatic aldehyde **2** (0.5 mmol, 5.0 equiv.), CuCl (0.01 mmol, 0.1 equiv.), TBHP (0.8 mmol, 8.0 equiv.), were added into the solution of MeCN (1.0 mL) and EtOAc (1.0 mL) in a sealed tube under ambient condition. The reaction mixture was stirred and heated at 80 °C until the reaction completed (monitored by TLC). Then, the reaction mixture was cooled to room temperature and concentrated under reduced pressure. Subsequently, H_2O was added to the residue, and the aqueous layer was extracted twice with ethyl acetate. The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The resulting organic residue was purified by column chromatography (SiO_2, petroleum ether/ethyl acetate gradient) to provide the products **3aa-3al**.



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

Substituted quinoxalinone **1** (0.1 mmol, 1.0 equiv.), benzaldehyde **2a** (0.5 mmol, 5.0 equiv.), CuCl (0.01 mmol, 0.1 equiv.), TBHP (0.8 mmol, 8.0 equiv.), were added into the solution of MeCN (1.0 mL) and EtOAc (1.0 mL) in a sealed tube under ambient condition. The reaction mixture was stirred and heated at 80 °C until the

reaction completed (monitored by TLC). Then, the reaction mixture was cooled to room temperature and concentrated under reduced pressure. Subsequently, H_2O was added to the residue, and the aqueous layer was extracted twice with ethyl acetate. The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The resulting organic residue was purified by column chromatography (SiO_2, petroleum ether/ethyl acetate gradient) to provide the products **3ba-3la**.

3.3 Unreacted substrates

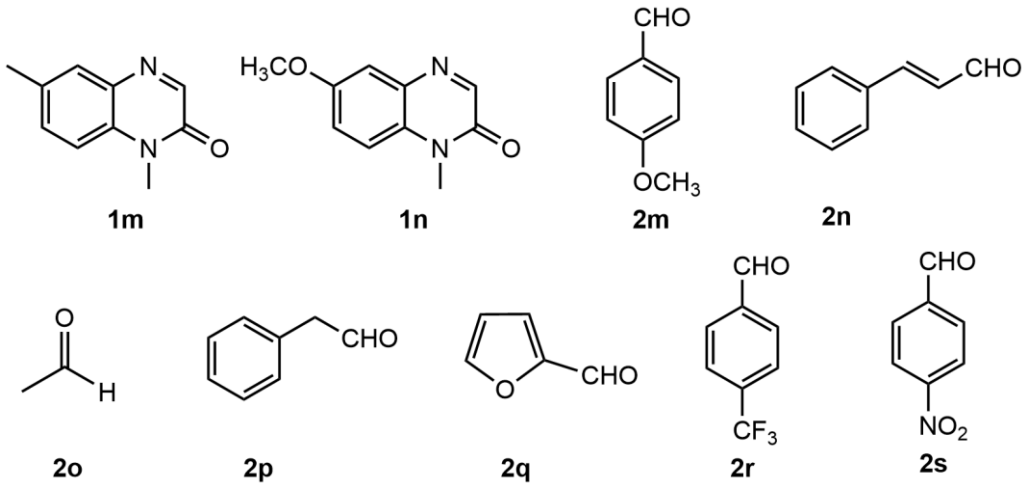


Figure S2 Unreacted substrates.

4. X-Ray structure and data of compound 3ja

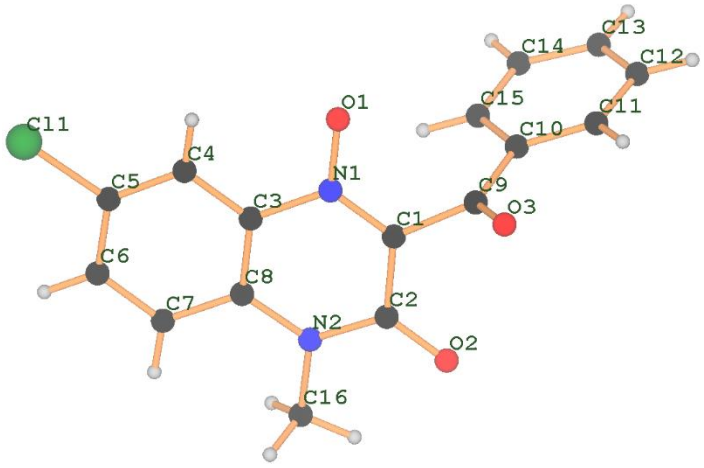
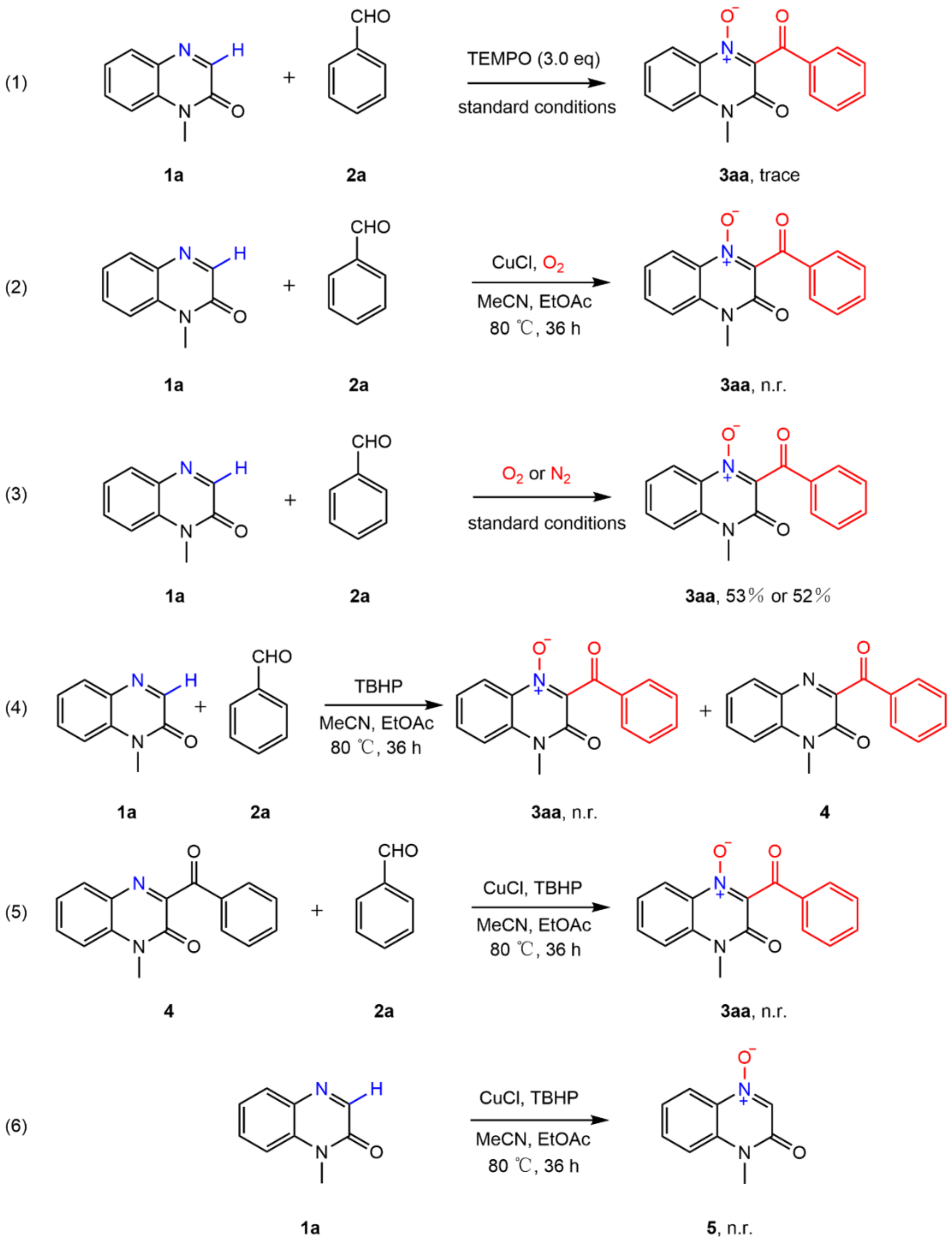


Figure S3 Crystal structure of **3ja**

Bond precision:	C-C = 0.0033 A	Wavelength=1.54178
Cell:	a=11.5686(2) b=9.5602(2) c=29.2255(5)	
	alpha=90 beta=96.480(1) gamma=90	
Temperature:	180 K	
	Calculated	Reported
Volume	3211.64(10)	3211.64(10)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C_{16} H_{11} Cl N_2 O_3 [+solvent]	C_{16} $H_{11}ClN_2O_3$, 0.5[CHCL3]
Sum formula	C_{16} H_{11} Cl N_2 O_3 [+ solvent]	$C_{16.50}$ $H_{11.50}$ $Cl_{2.50}$ N_2 O_3
MW	314.72	374.40
Dx (g cm^{-3})	1.302	1.549
Z	8	8
Mu (mm^{-1})	2.228	4.571
F000	1296.0	1528.0
F000’	1302.73	
h,k,l*max*	13,10,33	13,10,33
N*ref*	5099	5098
T*min*,T*max*	0.665,0.796	0.529,0.752
T*min*’	0.603	
Correction method=	# Reported T Limits: T*min*=0.529 T*max*=0.752	
	AbsCorr = MULTI-SCAN	
Data completeness= 1.000	Theta(*max*)= 62.371	
R(reflections)= 0.0549 (3819)		wR2(reflections)= 0.1556 (5098)
S = 1.041	N*par*= 352	

5. Mechanism investigation

Control experiments



Scheme S1 Control experiments.

The reaction was sufficiently suppressed when TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy) (3.0 equiv.) was used as a radical inhibitor (Scheme S1, Eq. 1). When quinoline *N*-oxide **1a** was treated with benzaldehyde **2a** under oxygen atmosphere in the absence of TBHP, no desired product was detected (Scheme S1, Eq. 2). Whereas, the above reaction proceeded smoothly under nitrogen atmosphere (or oxygen atmosphere) using 8.0 equiv. TBHP as oxidant and provided the desired product **3aa** in 52% (or 53%) yield (Scheme S1, Eq. 3), which was close to the result under the optimal reaction conditions (entry 37, Table S1). These above results indicated that the reaction was insensitive to external gas and TBHP played an indispensable role in the construction of *N*-oxides. It was noted that product **3aa** was not detected and the non-oxidized benzoylation product **4** was obtained in the absence of CuCl (Scheme S1, Eq. 4). The reaction did not proceed at all when starting material **1a** was replaced by the compound **4** (Scheme S1, Eq. 5). The results exclude the possibility that compound **4** was involved as reactive intermediate in this transformation. The corresponding *N*-oxide **5** was not obtained under standard conditions (Scheme S1, Eq. 6), indicating that the process did not proceed via oxidation as the first step.

6. Exploration of further application

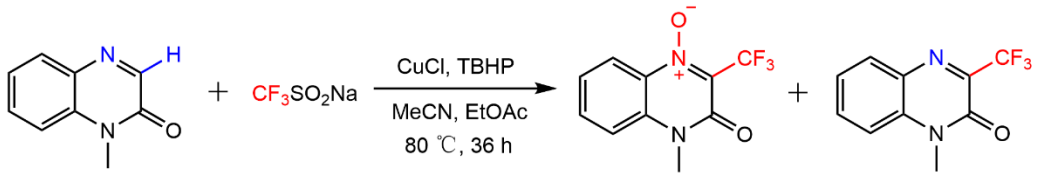
(1) **gram-scale reaction**

1a (0.80 g, 5.0 mmol) **2a** **3aa** (0.67 g, 48%)

(2) **styrene substrate scope experiment**

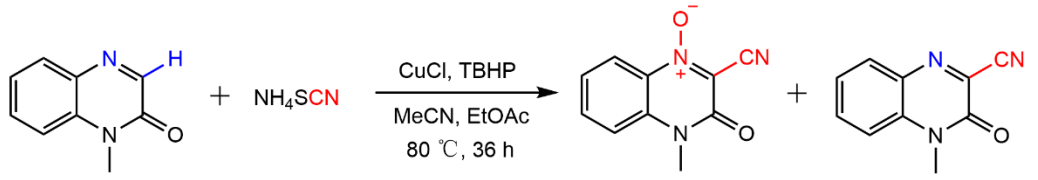
1a **9** **3aa**, 42%

(3) **sodium trifluoromethanesulfinate substrate scope experiment**



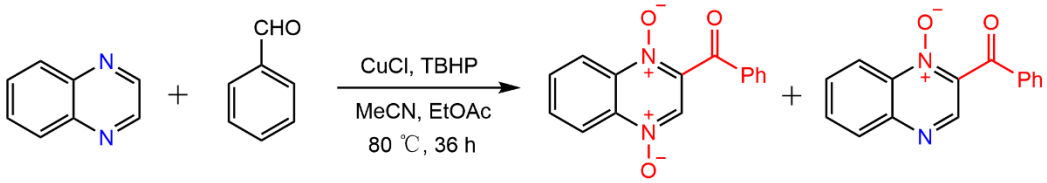
1a **10** **11,** n.r. **12,** 90%

(4) **ammonium thiocyanate substrate scope experiment**



1a **13** **14,** n.r. **15,** n.r.

(5) **quinoxaline substrate scope experiment**



16 **2a** **17,** n.r. **18,** n.r.

Scheme S2 Exploration of further application

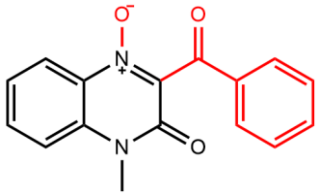
A gram-scale reaction was performed to demonstrate the potential application, using **1a** and **2a** as model reactants (Scheme S2, Eq. 1). The scaled-up reaction proceeded well to form the product **3aa** in 48% yield. Styrene **9** also confirmed to be a suitable substrate for this protocol, and product **3aa** was obtained with a yield of 42% (Scheme S2, Eq. 2). The reaction involved the oxidation of styrene to produce benzaldehyde as reactive species. This also proves that aromatic alkenes are applicable in our experimental protocol. The sodium trifluoromethanesulfinate and ammonium thiocyanate were also assessed under standard conditions (Scheme S2, Eq. 3&4). Unfortunately, the quinoxalin-2(1*H*)-one *N*-oxided trifluoromethylation product **11** and the *N*-oxided cyanation product **14** were not detected. Only the trifluoromethylation product **12** was detected,[3] and the cyanation product **15** was not produced at all.[4] For quinoxaline **16**, no reaction occurred under standard conditions, and only the starting material remained in the reaction mixture (Scheme S2, Eq. 5). We speculated that the electron-withdrawing carbonyl group played important role in stabilizing the reactive intermediate in the reaction process. All these results demonstrated the potential application and the scope of reactants for our synthetic method.

[3] N. B. Dutta, J. Bori, P. Gogoi, Baishya G. *Chemistry Select.*, 2021, ***6***, 1471-1477.

[4] J. Wang, B. Sun, L. Zhang, T. Xu, Y. Xie, C. Jin, *Org. Chem. Front*., 2020, ***7***, 113-118.

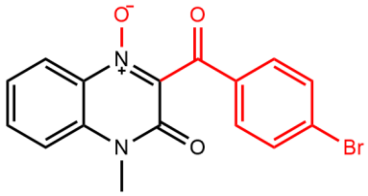
7. Characterization data of products

3aa



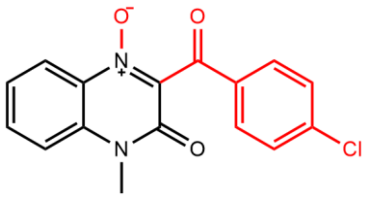
Yellow solid, isolated yield: 55 %; m.p: 115-116 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 7.2 Hz, 2H), 7.79 (t, *J* = 7.9 Hz, 1H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.51 – 7.43 (m, 4H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.72, 155.14, 135.34, 134.74, 134.72, 133.86, 133.63, 130.96, 129.16, 129.12, 124.23, 121.01, 114.87, 29.14. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{13}N_2O_3$: 281.0921, found: 281.0927.

3ab



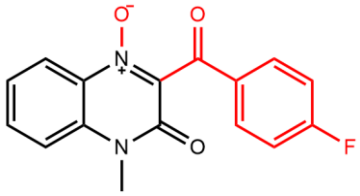
Yellow solid, isolated yield: 47 %; m.p: 123-124 ℃; R_f= 0.25 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.4 Hz, 1H), 7.78 (d, *J* = 8.8 Hz, 3H), 7.63 (d, *J* = 8.6 Hz, 2H), 7.52 – 7.43 (m, 2H), 3.74 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.79, 155.06, 134.78, 133.89, 133.80, 133.57, 132.49, 130.94, 130.52, 130.10, 124.34, 121.01, 114.92, 29.18. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}BrN_2O_3$: 359.0026, found: 359.0036.

3ac



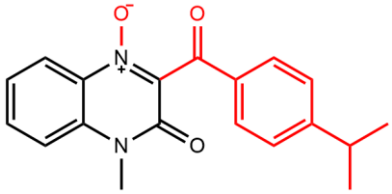
Yellow solid, isolated yield: 48 %; m.p: 128-129 ℃; R_f= 0.25 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 7.4 Hz, 1H), 7.87 (d, *J* = 8.5 Hz, 2H), 7.79 (t, *J* = 7.2 Hz, 1H), 7.52 – 7.44 (m, 4H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.55, 155.07, 141.23, 134.83, 133.90, 133.78, 133.18, 130.96, 130.50, 129.51, 124.33, 121.03, 114.91, 29.17. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}ClN_2O_3$: 315.0531, found: 315.0536.

3ad



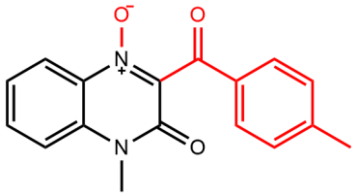
Yellow solid, isolated yield: 53 %; m.p: 117-118 ℃; R_f= 0.20 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 8.4 Hz, 1H), 7.98 – 7.94 (m, 2H), 7.79 (t, *J* = 7.9 Hz, 1H), 7.52 – 7.44 (m, 2H), 7.17 (t, *J* = 8.6 Hz, 2H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.11, 166.72 (d, J = 255.8 Hz), 155.09, 133.88, 133.72, 132.02, 131.93, 131.30 (d, J = 2.8 Hz), 130.97, 124.31, 121.05, 116.45 (d, J = 22.2 Hz), 114.88, 29.16. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}FN_2O_3$: 299.0826, found: 299.0831.

3ae



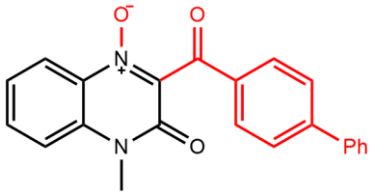
Yellow solid, isolated yield: 62 %; m.p: 124-125 ℃; R_f=0.30 (Ethyl acetate: Petroleum ether, 1:4 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 8.2 Hz, 2H), 7.78 (t, *J* = 8.4 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 2H), 3.73 (s, 3H), 2.99 – 2.92 (m, 1H). 1.25 (d, *J* = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.19, 156.56, 155.18, 135.61, 133.85, 133.49, 132.66, 131.01, 129.46, 127.31, 124.15, 121.06, 114.80, 34.51, 29.10, 23.56. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{19}H_{19}N_2O_3$: 323.1390, found: 323.1403.

3af



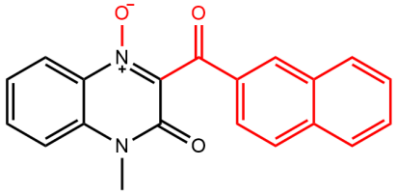
Yellow solid, isolated yield: 60 %; m.p: 120-121 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:4 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.3 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 2H), 7.77 (t, *J* = 7.3 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.74 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.24, 155.16, 145.93, 135.54, 133.84, 133.52, 132.40, 130.99, 129.83, 129.28, 124.14, 121.03, 114.82, 29.10, 21.93. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{17}H_{15}N_2O_3$: 295.1077, found: 295.1080.

3ag



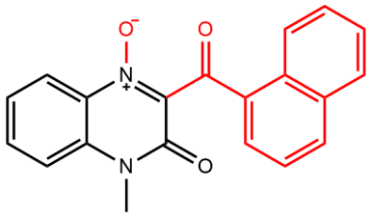
Yellow solid, isolated yield: 59 %; m.p: 129-130 ℃; R_f= 0.35 (Ethyl acetate: Petroleum ether, 1:4 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 8.4 Hz, 1H), 8.00 (d, *J* = 8.4 Hz, 2H), 7.78 (t, *J* = 7.9 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.52 – 7.39 (m, 5H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.28, 155.18, 147.48, 139.77, 135.37, 133.88, 133.65, 133.50, 130.97, 129.76, 128.99, 128.46, 127.85, 127.36, 124.24, 121.00, 114.91, 29.16. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{22}H_{17}N_2O_3$: 357.1234, found: 357.1245.

3ah



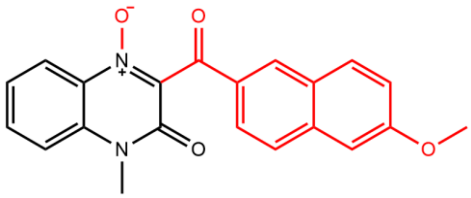
Yellow solid, isolated yield: 58 %; m.p: 121-122 ℃; R_f= 0.25 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.3 Hz, 1H), 8.35 (s, 1H), 8.06 (d, *J* = 8.6 Hz, 1H), 7.94 (d, *J* = 8.6 Hz, 1H), 7.91 – 7.87 (m, 2H), 7.80 (t, *J* = 7.8 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.53 (d, *J* = 8.9 Hz, 2H), 7.47 (t, *J* = 8.0 Hz, 1H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.75, 155.26, 136.51, 135.49, 133.89, 133.64, 132.62, 132.27, 131.91, 131.02, 129.79, 129.19, 127.92, 126.90, 124.24, 123.68, 121.04, 121.03, 114.93, 29.19. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{20}H_{15}N_2O_3$: 331.1077, found: 331.1079.

3ai



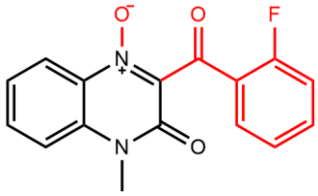
Yellow solid, isolated yield: 61 %; m.p: 122-123 ℃; R_f= 0.25 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.27 (d, *J* = 8.7 Hz, 1H), 8.46 (d, *J* = 8.4, 1H), 8.11 (d, *J* = 8.2 Hz, 1H), 7.93 (t, *J* = 6.8 Hz, 2H), 7.83 – 7.73 (m, 2H), 7.63 (t, *J* = 7.1 Hz, 1H), 7.54 – 7.45 (m, 3H), 3.79 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.13, 155.30, 136.50, 135.55, 134.05, 133.76, 133.49, 132.12, 131.18, 130.95, 130.79, 129.22, 128.58, 126.97, 125.93, 124.61, 124.21, 120.95, 114.84, 29.13. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{20}H_{15}N_2O_3$: 331.1077, found: 331.1086.

3aj



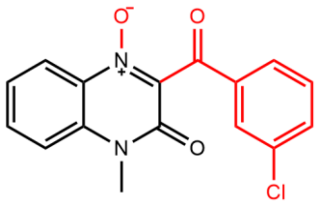
Yellow solid, isolated yield: 65 %; m.p: 126-127 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.4 Hz, 1H), 8.26 (s, 1H), 8.02 (d, *J* = 6.8 Hz, 1H), 7.82 – 7.75 (m, 3H), 7.52 (d, *J* = 8.2 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.17 – 7.14 (m, 2H), 3.94 (s, 3H), 3.76 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.37, 160.32, 155.26, 138.41, 135.67, 133.83, 133.55, 131.82, 131.39, 130.99, 130.31, 127.94, 124.18, 124.49, 124.18, 120.99, 119.81, 114.90, 105.96, 55.47, 29.15. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{21}H_{17}N_2O_4$: 361.1183, found: 361.1194.

3ak



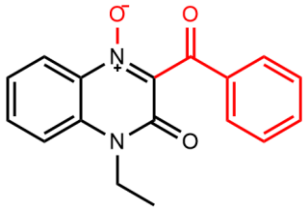
Yellow solid, isolated yield: 37 %; m.p: 113-114 ℃; R_f= 0.20 (Ethyl acetate: Petroleum ether, 1:4 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 – 8.15 (m, 1H), 7.75 – 7.69 (m, 1H), 7.37 – 7.26 (m, 6H), 3.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.31 (d, *J* = 261.2 Hz), 159.52 (d, *J* = 4.2 Hz), 154.96, 148.92, 136.93 (d, *J* = 9.3 Hz), 132.90, 126.42, 125.48, 125.17, 124.75 (d, *J* = 4.2 Hz), 124.70, 117.44 (d, *J* = 21.5 Hz), 114.98, 114.05 (d, *J* = 10.8 Hz), 112.20, 30.64. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}FN_2O_3$: 299.0826, found: 299.0811.

3al



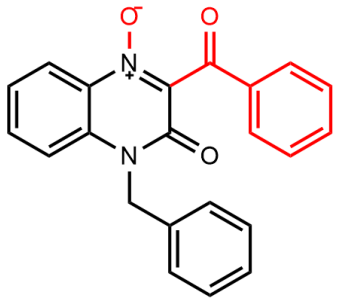
Yellow solid, isolated yield: 40 %; m.p: 125-126 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:4 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 8.4 Hz, 1H), 7.88 (s, 1H), 7.80 (t, *J* = 8.1 Hz, 2H), 7.60 (d, *J* = 9.0 Hz, 1H), 7.51 (d, *J* = 8.4 Hz, 1H), 7.48 – 7.42 (m, 2H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.59, 155.05, 136.33, 135.41, 134.66, 134.59, 133.94, 133.82, 130.98, 130.46, 128.98, 127.21, 124.35, 121.06, 114.92, 29.70. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}ClN_2O_3$: 315.0531, found: 315.0533.

3ba



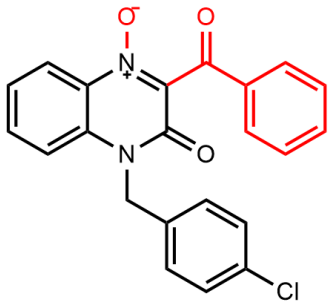
Yellow solid, isolated yield: 58 %; m.p: 122-123 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:4 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 8.0 Hz, 2H), 7.77 (t, *J* = 7.9 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.47 – 7.52 (m, 3H), 7.43 (t, *J* = 7.8 Hz, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.86, 154.73, 135.25, 134.79, 134.78, 134.70, 133.59, 132.98, 131.13, 129.12, 124.03, 121.21, 114.71, 37.52, 12.72. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{17}H_{14}N_2O_3$: 295.1077, found: 295.1082.

3ca



Yellow solid, isolated yield: 57 %; m.p: 108-109 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.4 Hz, 1H), 7.97 (d, *J* = 7.1 Hz, 2H), 7.67 – 7.62 (m, 2H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.45 (d, *J* = 8.1 Hz, 1H), 7.41 – 7.36 (m, 2H), 7.35 – 7.30 (m, 4H), 5.52 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.67, 155.42, 135.26, 134.79, 134.76, 133.49, 133.29, 131.22, 130.64, 129.16, 129.15, 128.09, 126.97, 126.82, 124.26, 121.07, 115.65, 45.83. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{22}H_{17}N_2O_3$: 357.1234, found: 357.1243.

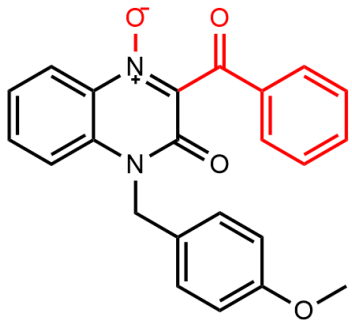
3da



Yellow solid, isolated yield: 54 %; m.p: 113-114 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 8.3 Hz, 1H), 7.95 (d, *J* = 7.2 Hz, 2H), 7.65 (t, *J* = 7.9 Hz, 2H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.43 – 7.38

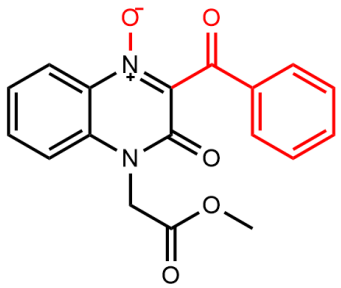
(m, 2H), 7.33 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 5.48 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.57, 155.35, 135.20, 134.81, 134.74, 134.04, 133.57, 133.29, 133.08, 131.25, 129.34, 129.18, 129.15, 128.47, 124.41, 121.21, 115.38, 45.22. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{22}H_{16}ClN_2O_3$: 391.0844, found: 391.0843.

3ea



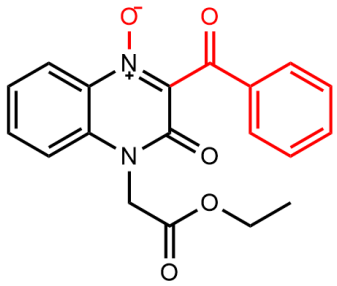
Yellow solid, isolated yield: 59 %; m.p: 112-113 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 2H), 7.67 – 7.62 (m, 2H), 7.53 – 7.48 (m, 3H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 5.45 (s, 2H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.76, 159.36, 155.41, 135.24, 134.80, 134.75, 133.47, 133.28, 131.17, 130.28, 129.16, 128.56, 126.81, 124.20, 120.99, 115.67, 114.49, 55.32, 45.32. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{23}H_{19}N_2O_4$: 387.1339, found: 387.1349.

3fa



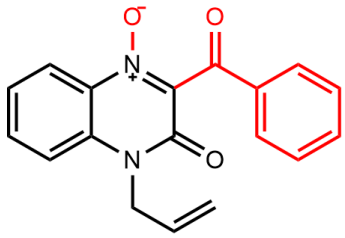
Yellow solid, isolated yield: 52 %; m.p: 109-110 ℃; R_f= 0.35 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 8.4 Hz, 1H), 7.96 (d, *J* = 7.7 Hz, 2H), 7.77 (t, *J* = 7.8 Hz, 1H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.54 – 7.46 (m, 4H), 5.11 (s, 2H), 3.84 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.39, 167.26, 154.92, 134.98, 134.83, 134.64, 133.78, 133.02, 131.15, 129.16, 124.57, 121.22, 114.44, 53.10, 43.01. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{18}H_{15}N_2O_5$: 339.0975, found: 339.0972.

3ga



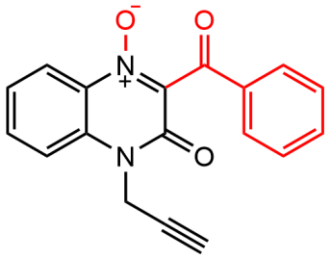
Yellow solid, isolated yield: 50 %; m.p: 115-116 ℃; R_f= 0.35 (Ethyl acetate: Petroleum ether, 1:5 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 7.8 Hz, 2H), 7.72 (t, *J* = 7.9 Hz, 1H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.50 – 7.40 (m, 4H), 5.05 (s, 2H), 4.26 (q, *J* = 7.1 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.40, 166.74, 154.92, 134.98, 134.81, 134.65, 133.73, 133.06, 131.11, 129.15, 124.51, 121.18, 114.47, 62.39, 43.14, 14.12. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{19}H_{17}N_2O_5$: 353.1132, found: 353.1140.

3ha



Yellow solid, isolated yield: 47 %; m.p: 103-104 ℃; R_f= 0.35 (Ethyl acetate: Petroleum ether, 1:6 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 7.4 Hz, 2H), 7.74 (t, *J* = 8.8 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 3H), 7.43 (t, *J* = 7.8 Hz, 1H), 5.99 – 5.91 (m, 1H), 5.36 – 5.28 (m, 2H), 4.94 (d, *J* = 5.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.71, 154.81, 135.20, 134.74, 133.50, 133.18, 131.05, 130.61, 130.37, 129.13, 124.22, 120.98, 118.81, 115.51, 44.47. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{18}H_{15}N_2O_3$: 307.1077, found: 307.1083.

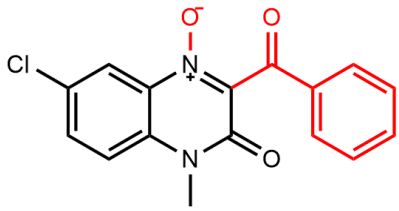
3ia



Yellow solid, isolated yield: 48 %; m.p: 107-108 ℃; R_f= 0.35 (Ethyl acetate: Petroleum ether, 1:6 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 7.9 Hz, 1H), 7.93 (d, *J* = 7.5 Hz, 2H), 7.81 (t, *J* = 7.3 Hz, 1H), 7.69 – 7.62 (m, 2H), 7.52 – 7.46 (t, m, 3H), 5.09 (d, *J* = 2.4 Hz, 2H), 2.36 (t, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.47, 154.28, 134.82, 134.65, 133.64, 132.37, 131.26, 130.07, 129.19,

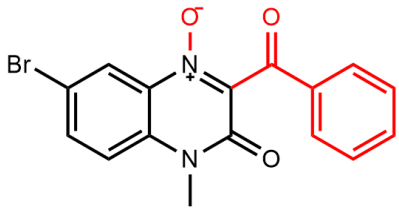
129.14, 124.60, 121.11, 115.46, 76.26, 74.05, 31.47. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{18}H_{13}N_2O_3$: 305.0921, found: 305.0907.

3ja



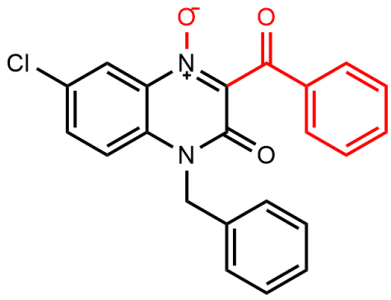
Yellow solid, isolated yield: 53 %; m.p: 124-125 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 2.2 Hz, 1H), 7.91 (d, *J* = 7.5 Hz, 2H), 7.72 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 2H), 7.44 (d, *J* = 9.0 Hz, 1H), 3.72 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.30, 154.81, 135.84, 134.89, 134.53, 133.76, 132.50, 131.35, 130.32, 129.16, 129.14, 120.71, 116.33, 29.28. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}ClN_2O_3$: 315.0531, found: 315.0543.

3ka



Yellow solid, isolated yield: 52 %; m.p: 126-127 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, *J* = 2.2 Hz, 1H), 7.91 (d, *J* = 7.3 Hz, 2H), 7.86 (d, *J* = 8.9 Hz, 1H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.37 (d, *J* = 8.9 Hz, 1H), 3.72 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.26, 154.81, 136.55, 135.79, 134.91, 134.50, 132.92, 131.53, 129.16, 123.77, 117.34, 116.45, 29.27. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{16}H_{12}BrN_2O_3$: 359.0026, found: 359.0034.

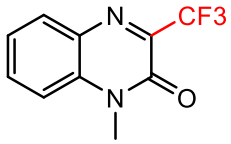
3la



Yellow solid, isolated yield: 55 %; m.p: 132-133 ℃; R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:3 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 2.4 Hz, 1H),

7.94 (d, *J* = 7.3 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.57 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.52 (t, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.9 Hz, 3H), 7.33 – 7.28 (m, 3H), 5.49 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.27, 155.12, 135.76, 134.96, 134.56, 134.36, 133.68, 131.87, 131.64, 130.44, 129.26, 129.22, 129.18, 128.28, 126.89, 120.84, 117.05, 45.98. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{22}H_{16}ClN_2O_3$: 391.0844, found: 391.0845.

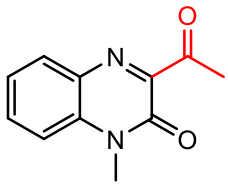
12



This product was obtained using CF_3SO_2Na as substrate under standard conditions.

Yellow solid, isolated yield: 90 %, R_f= 0.30 (Ethyl acetate: Petroleum ether, 1:6 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.1 Hz, 1H), 7.74 (t, *J* = 7.9 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.5 Hz, 1H), 3.76 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.61, 143.87 (q, *J* = 33.9 Hz), 134.59, 133.56, 131.75, 130.90, 124.52, 119.91 (q, *J* = 274.5 Hz), 114.03, 29.17. HRMS (ESI): m/z: calcd for $[M+H]^+$ $C_{10}H_8F_3N_2O$: 229.0583, Found: 229.0593.

19[1]



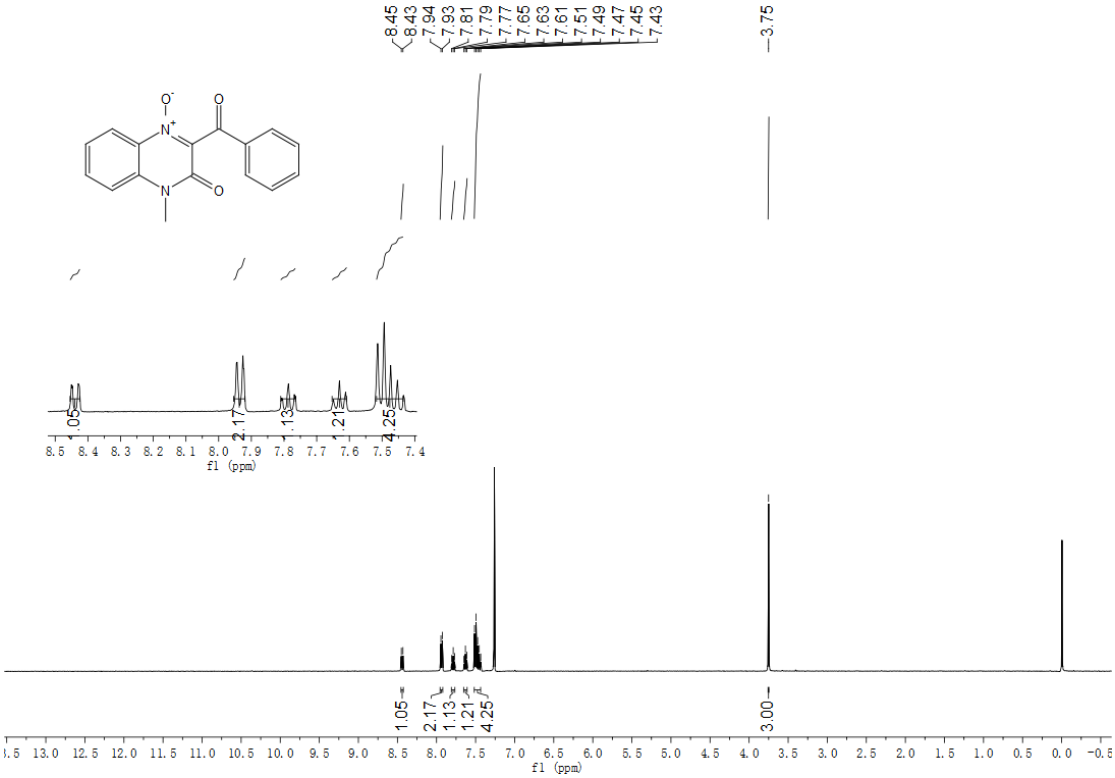
This product was obtained using acetaldehyde as substrate under standard conditions.

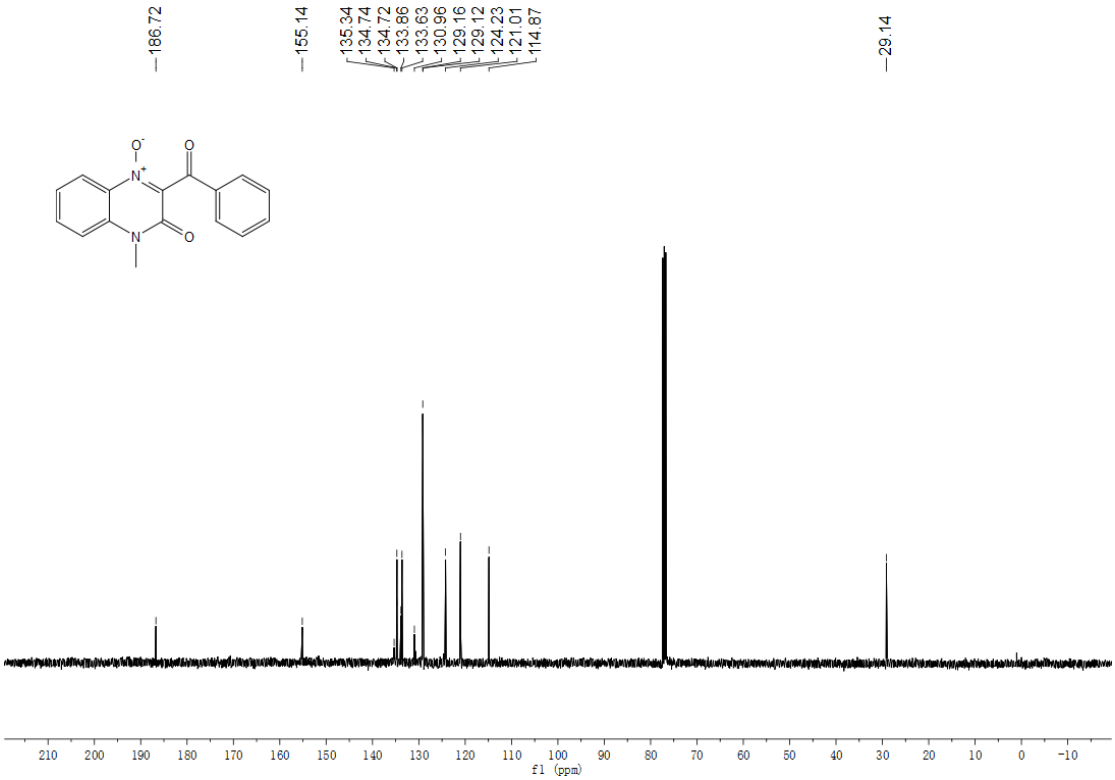
Yellow solid, isolated yield: 65 %, R_f= 0.20 (Ethyl acetate: Petroleum ether, 1:6 (v/v)). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 1H), 7.68 (t, *J* = 7.9 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 1H), 7.36 (d, *J* = 8.5 Hz, 1H), 3.73 (s, 3H), 2.72 (s, 3H).

[1] K. Zhang, J. Xu, J. Xiao, R. Zhong, J. Li, *Eur. J. Org. Chem.*, 2023, ***26***, e202201432

8. ^{1}H and ^{13}C NMR spectra

3aa





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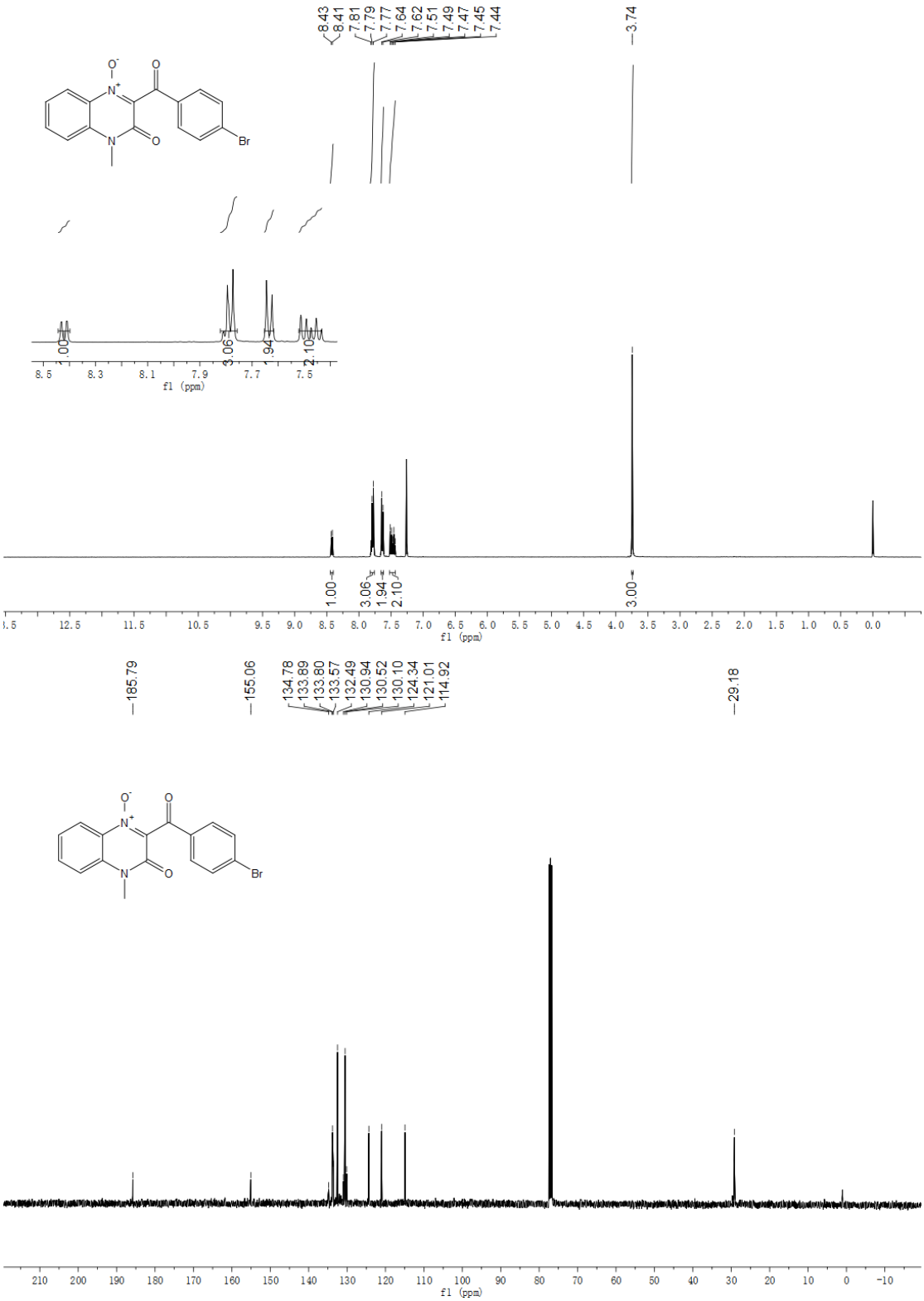
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7.51
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7.47
7.45
7.44
3.74
Br
1.00
3.06
1.94
2.10
3.00
f1 (ppm)
185.79
155.06
134.78
133.89
133.80
133.57
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130.94
130.52
130.10
124.34
121.01
114.92
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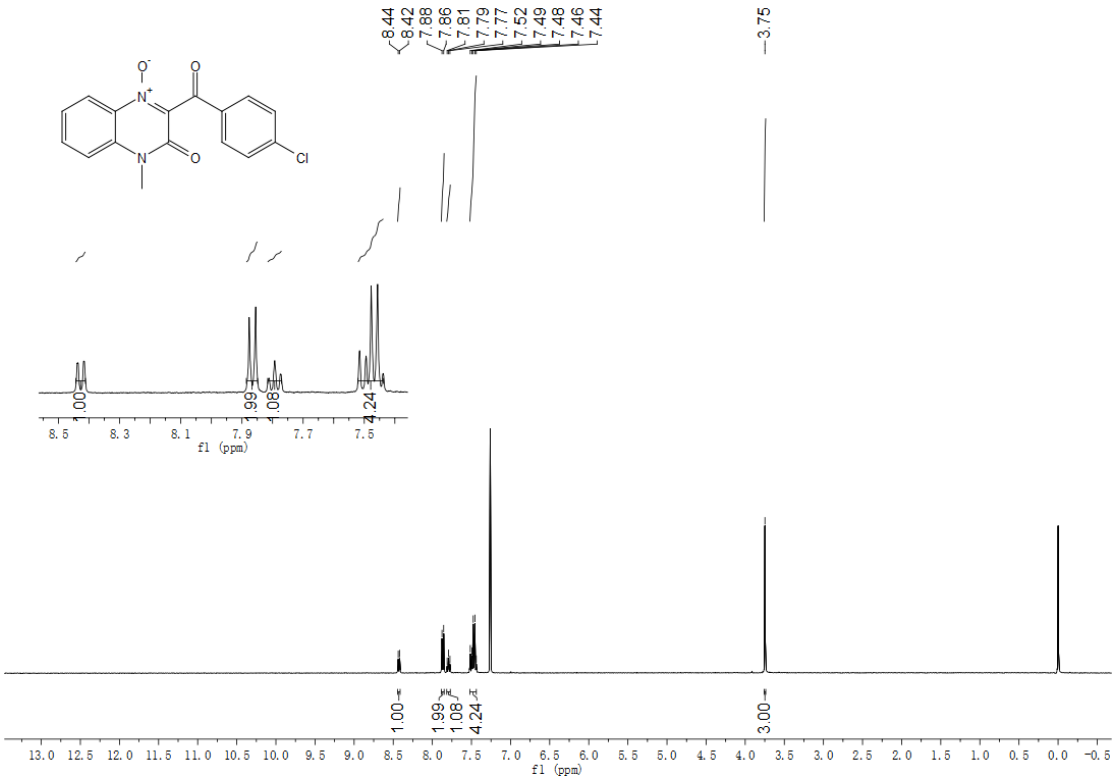
Br
f1 (ppm)

3ac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7.77
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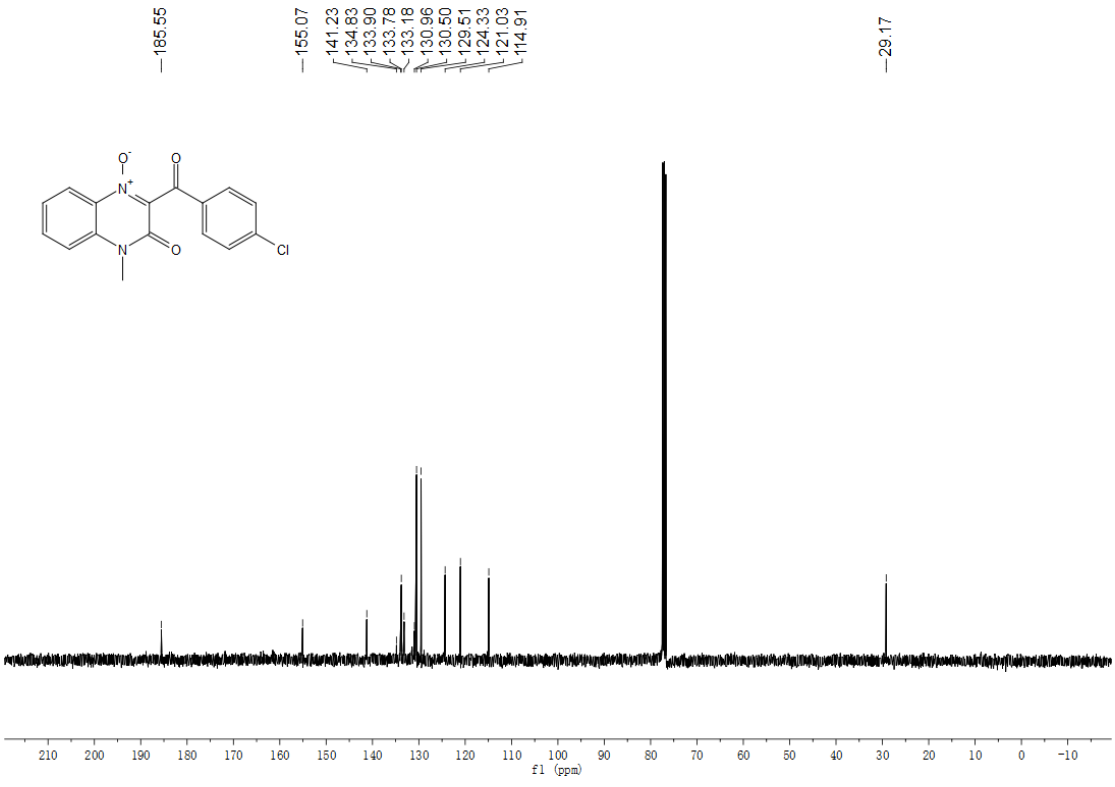
f1 (ppm)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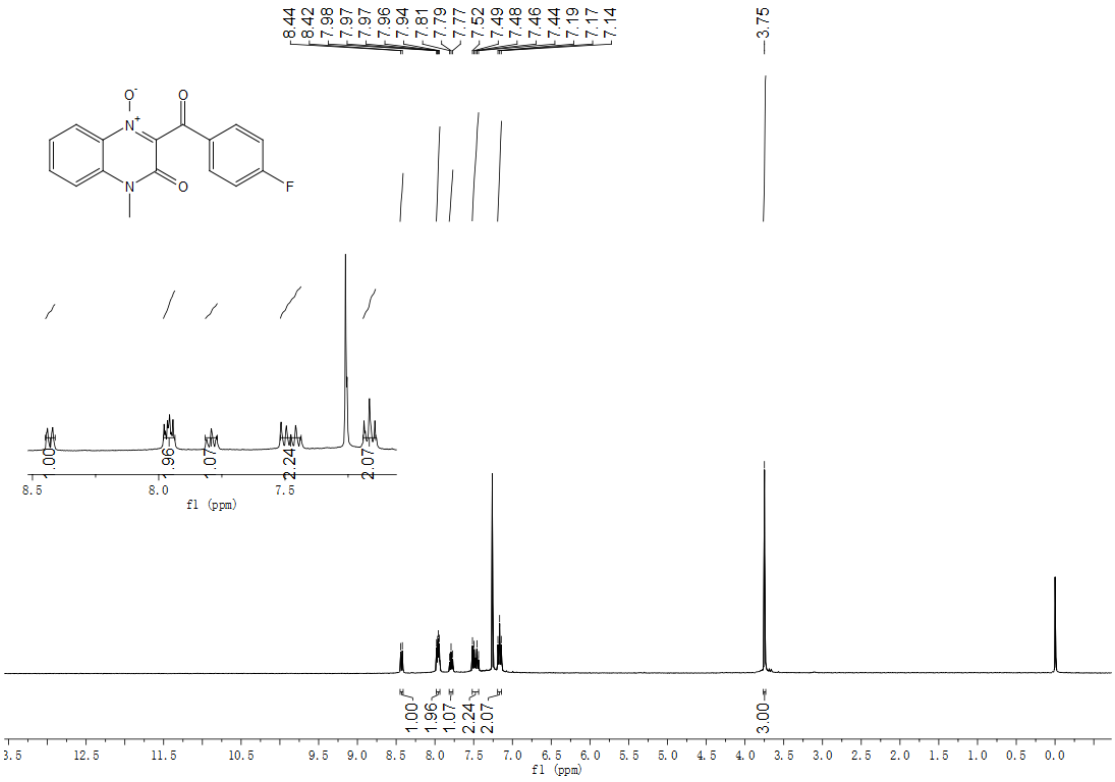
29.17
f1 (ppm)

3ad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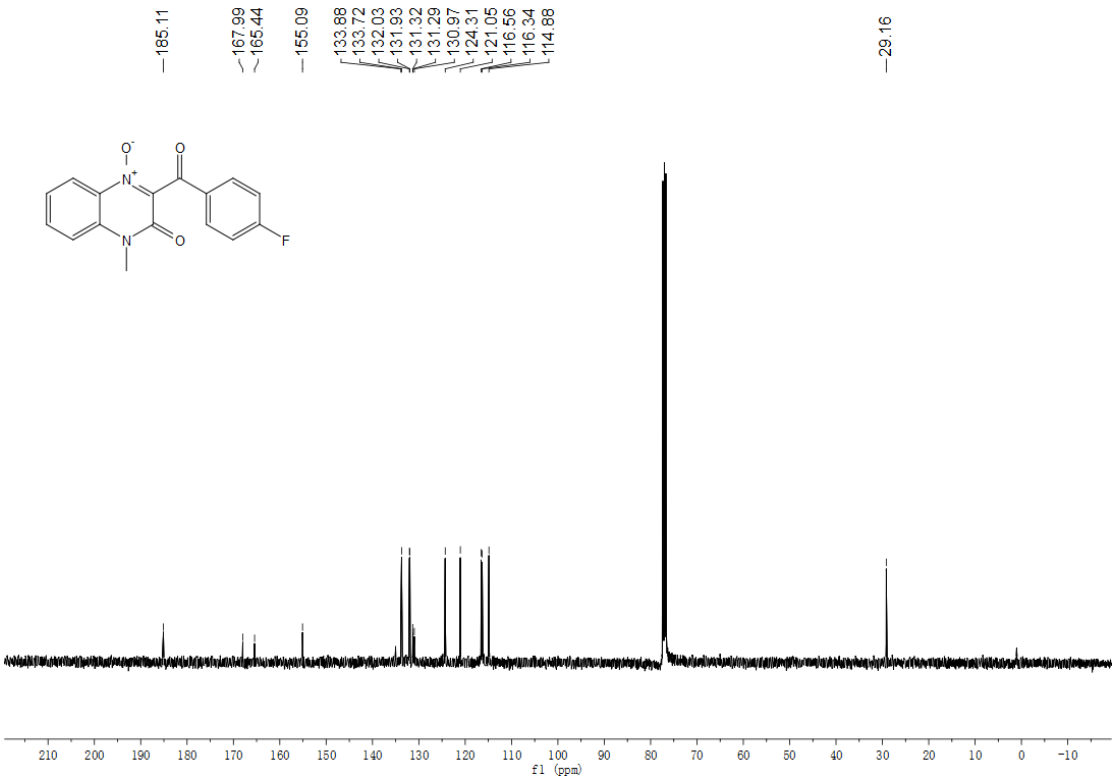
3.00
f1 (ppm)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121.05
116.56
116.34
114.88
29.16
f1 (ppm)

3ae


8.44
8.42
7.86
7.84
7.79
7.77
7.75
7.50
7.48
7.45
7.43
7.41
7.34
7.32
3.73
2.99
2.98
2.96
2.94
2.92
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1.24
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1.03
2.04
3.19
1.08
6.04
f1 (ppm)
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121.06
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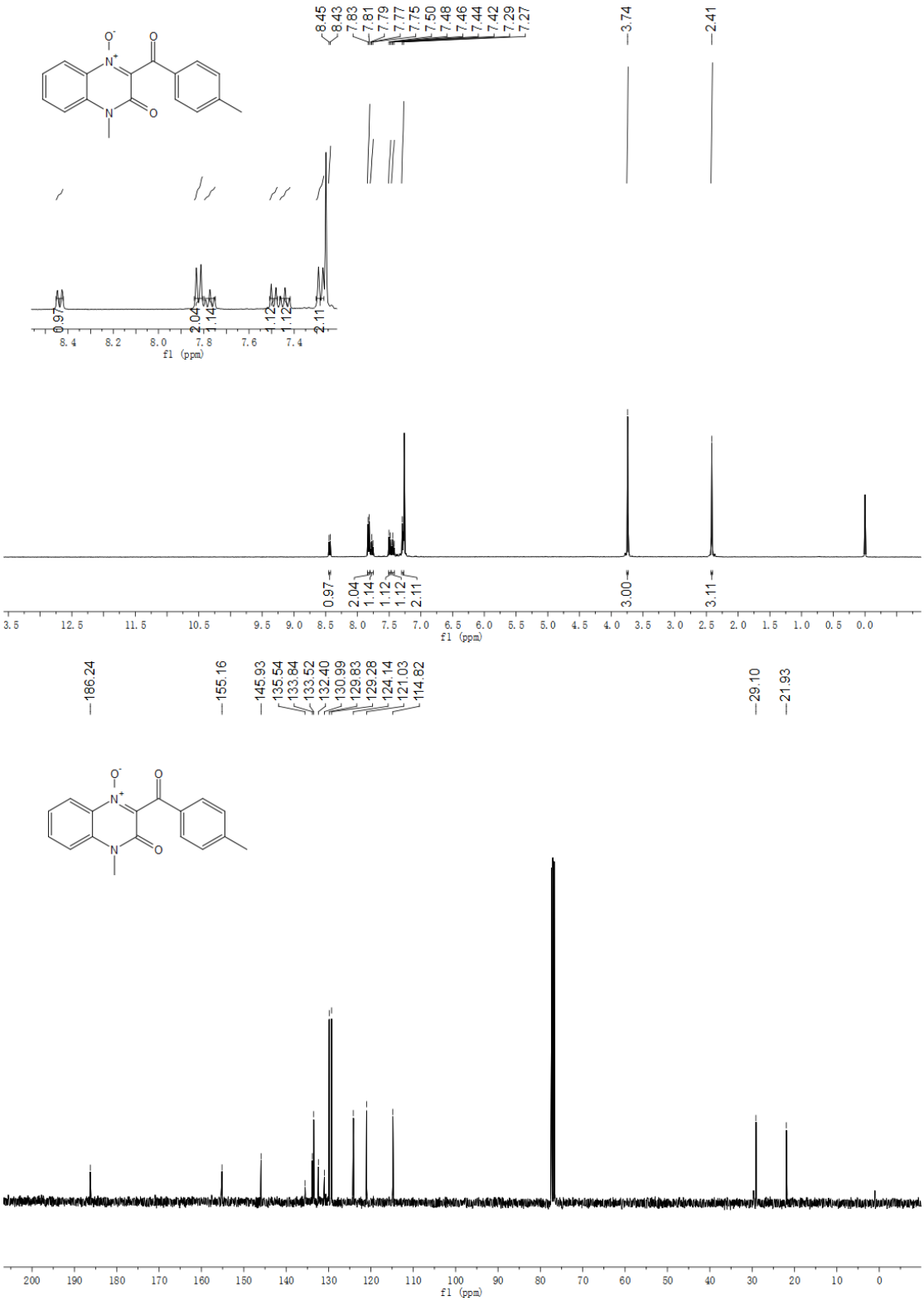
f1 (ppm)

3af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7.27
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1.14
1.12
1.12
2.11
3.00
3.11
f1 (ppm)
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155.16
145.93
135.54
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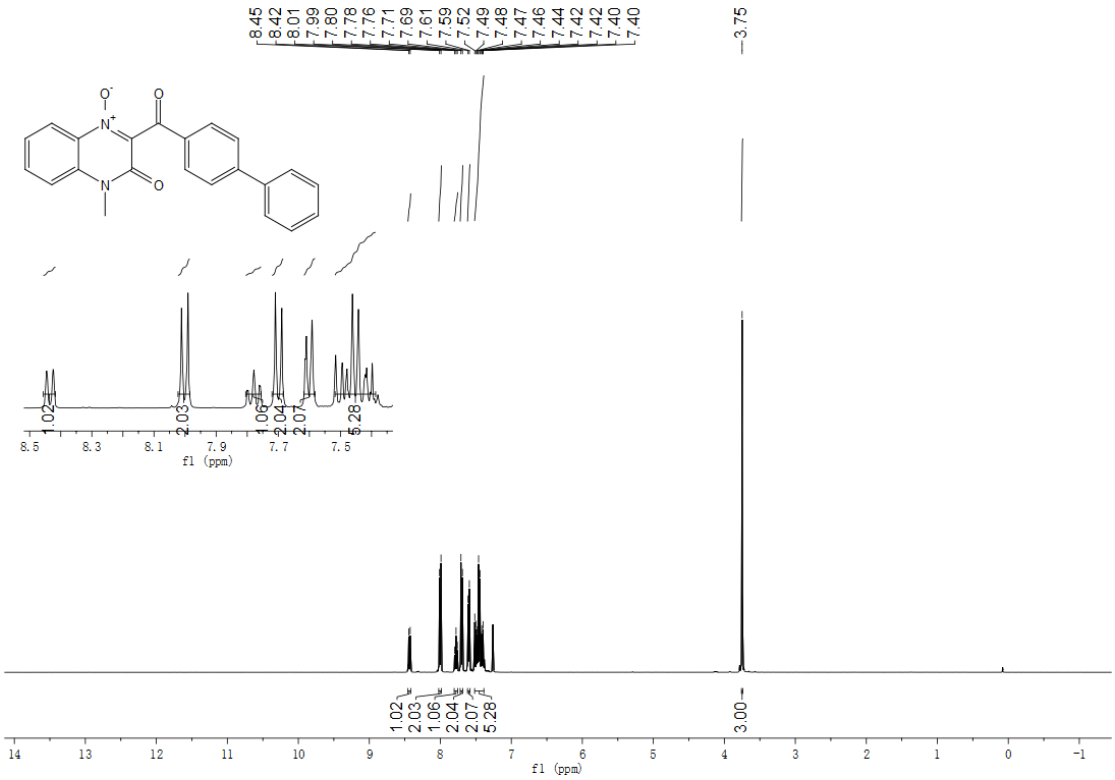
f1 (ppm)

3ag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7.48
7.47
7.46
7.44
7.42
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7.40
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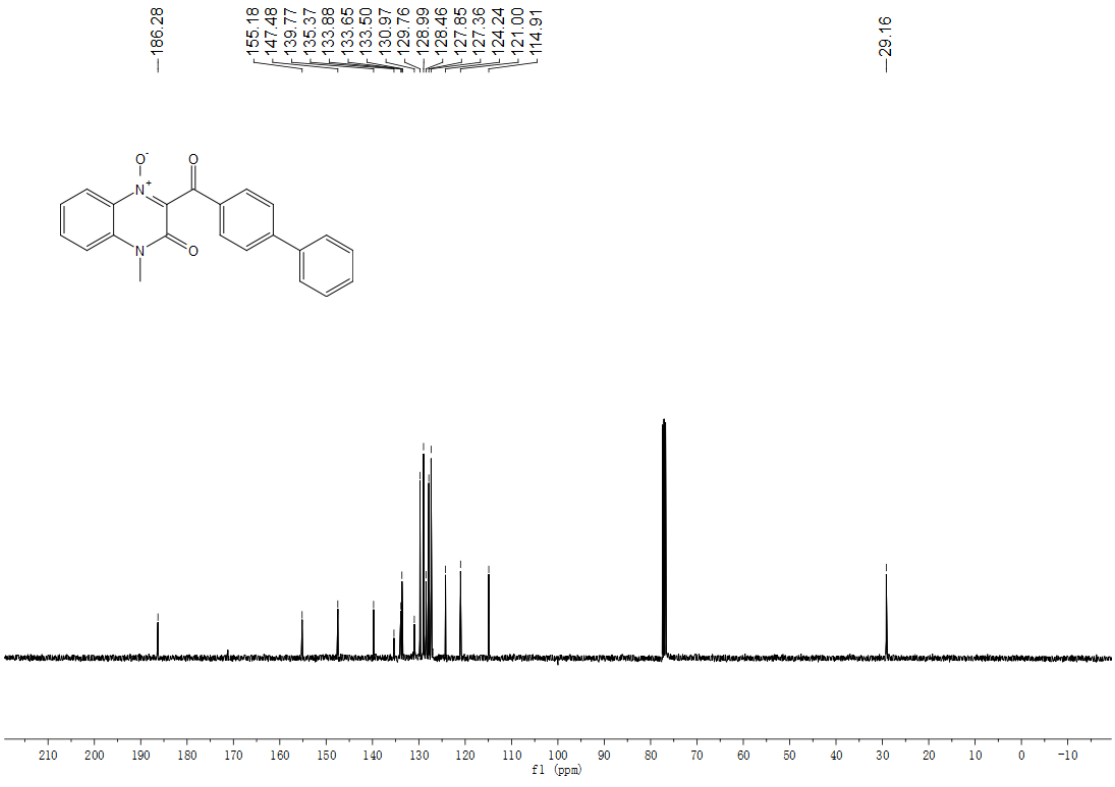
1.06
2.04
2.07
5.28
3.00
f1 (ppm)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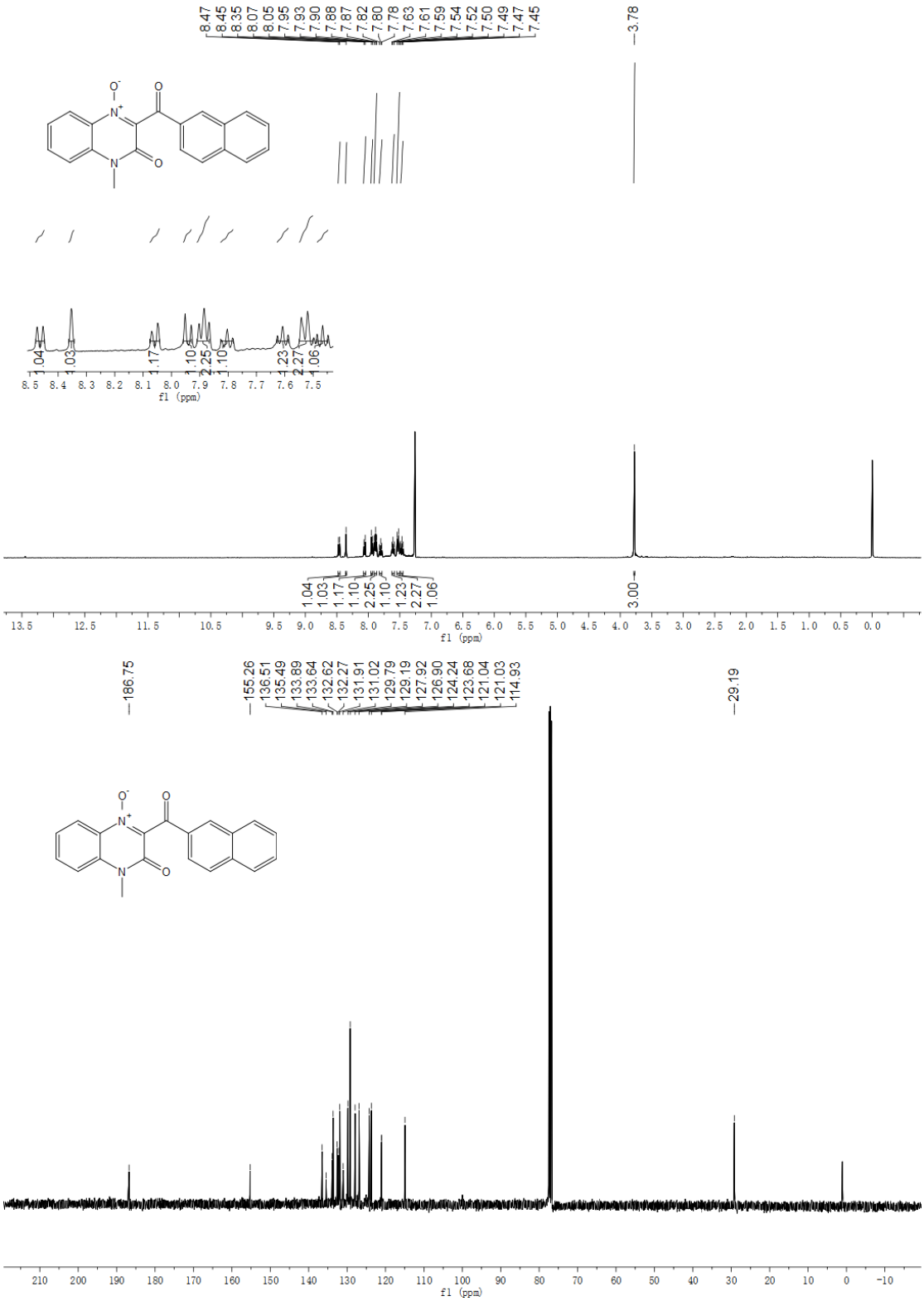
127.36
124.24
121.00
114.91
29.16
f1 (ppm)

3ah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7.78
7.63
7.61
7.59
7.54
7.52
7.50
7.49
7.47
7.45
3.78
1.04
1.03
1.17
1.10
2.25
1.10
1.23
2.27
1.06
3.00
f1 (ppm)
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155.26
136.51
135.49
133.89
133.64
132.62
132.27
131.91
131.02
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126.90
124.24
123.68
121.04
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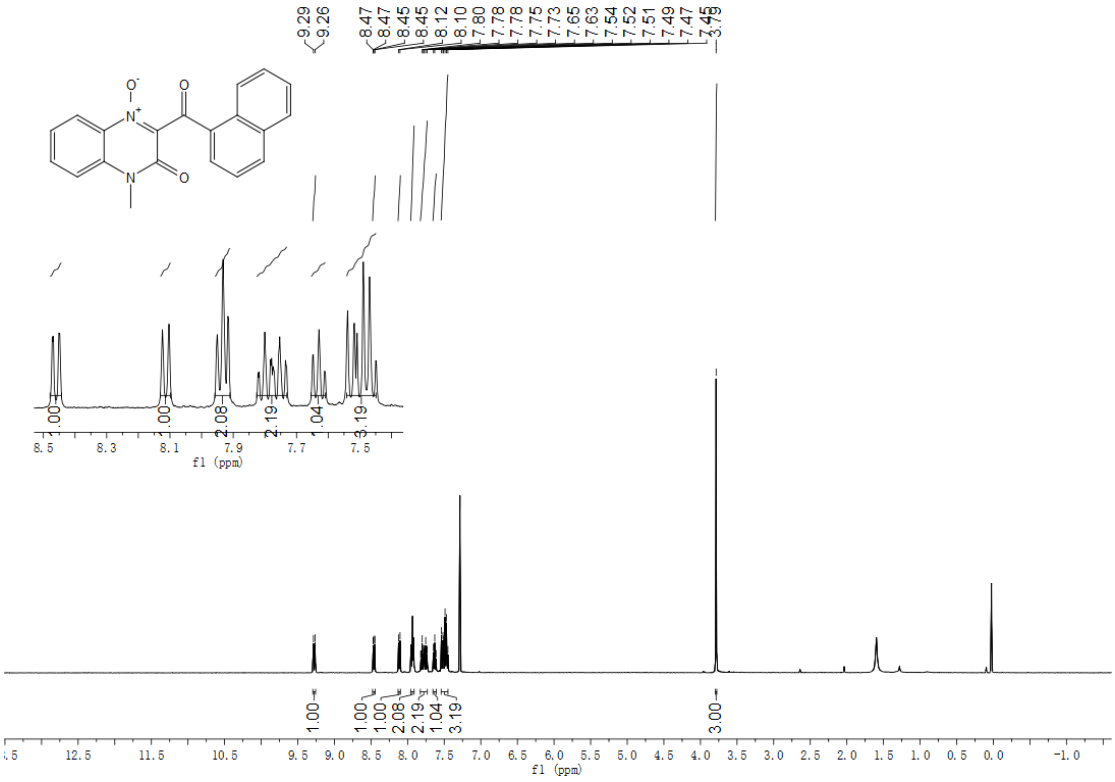
114.93
29.19
f1 (ppm)

3ai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7.65
7.63
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7.51
7.49
7.47
7.45
3.79
1.00
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2.19
1.04
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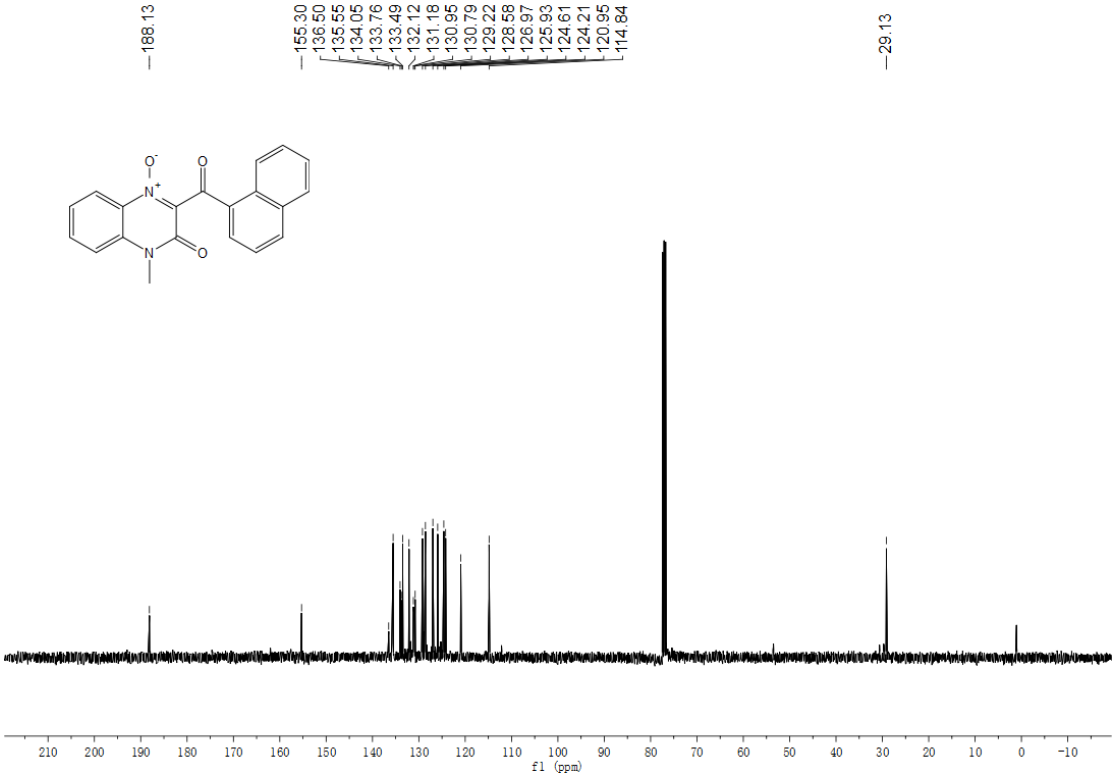
3.00
f1 (ppm)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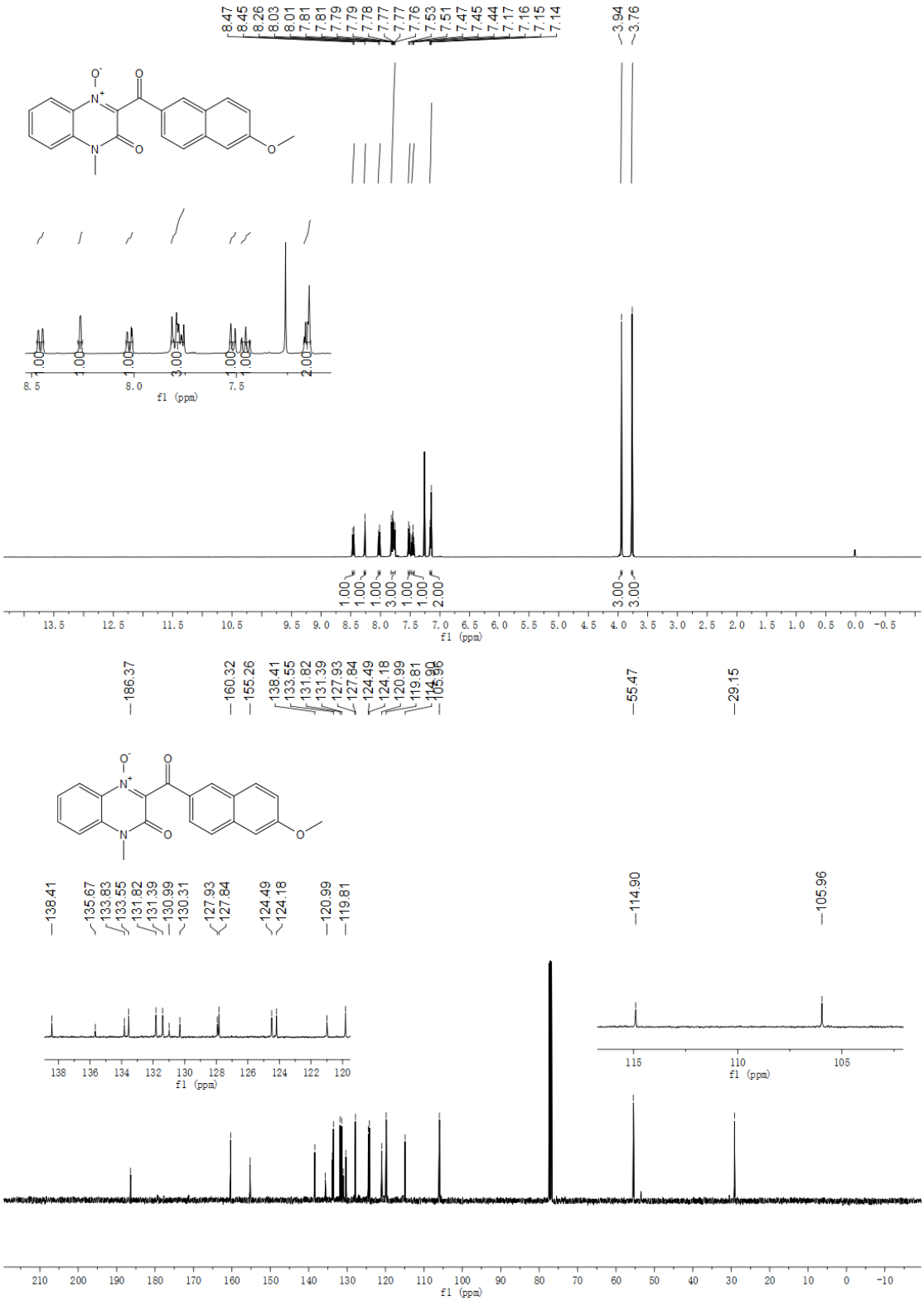
3aj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7.79
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7.77
7.76
7.53
7.51
7.47
7.45
7.44
7.17
7.16
7.15
7.14
3.94
3.76
f1 (ppm)
186.37
160.32
155.26
138.41
133.55
131.82
131.39
127.93
127.84
124.49
124.18
120.99
119.81
114.90
105.96
55.47
29.15
138.41
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133.83
133.55
131.82
131.39
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124.18
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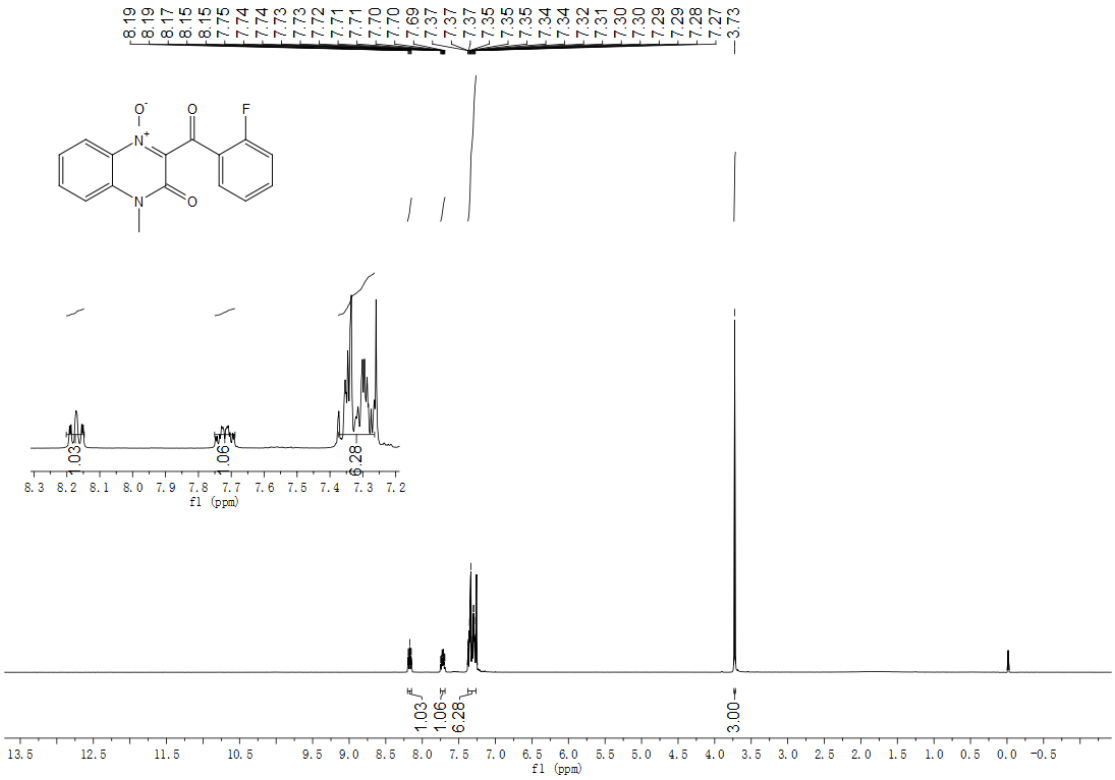
f1 (ppm)

3ak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7.37
7.35
7.35
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7.34
7.34
7.32
7.31
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7.27
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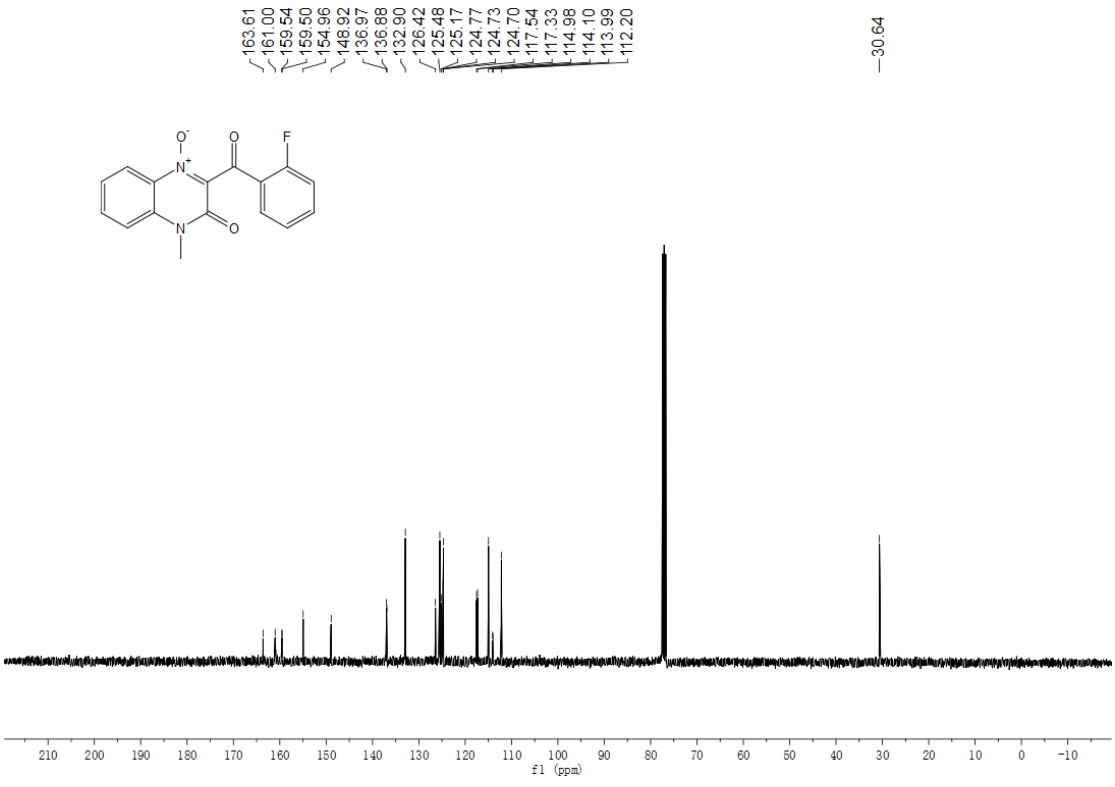
f1 (ppm)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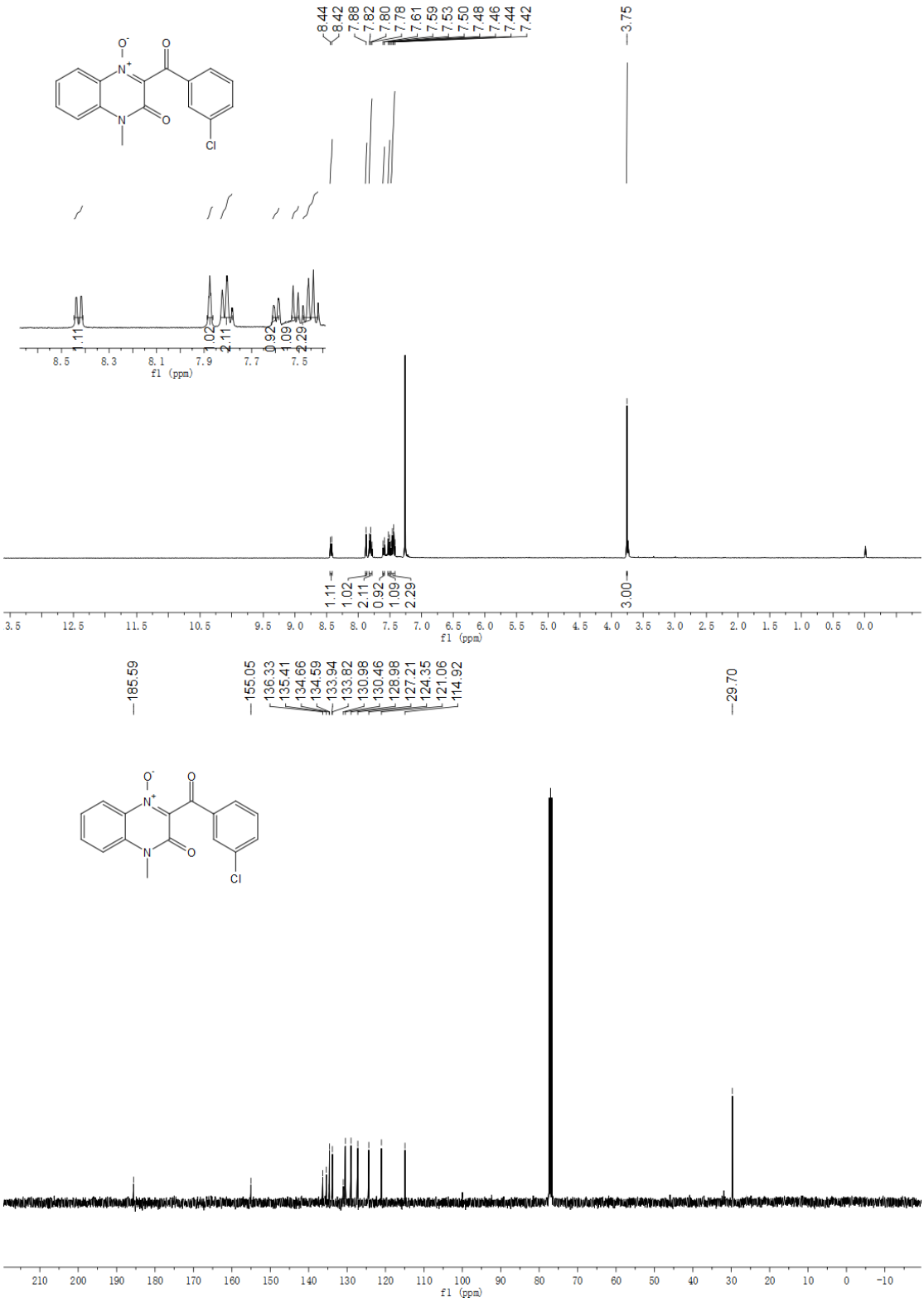
3al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7.50
7.48
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7.44
7.42
3.75
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1.02
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2.29
3.00
f1 (ppm)
185.59
155.05
136.33
135.41
134.66
134.59
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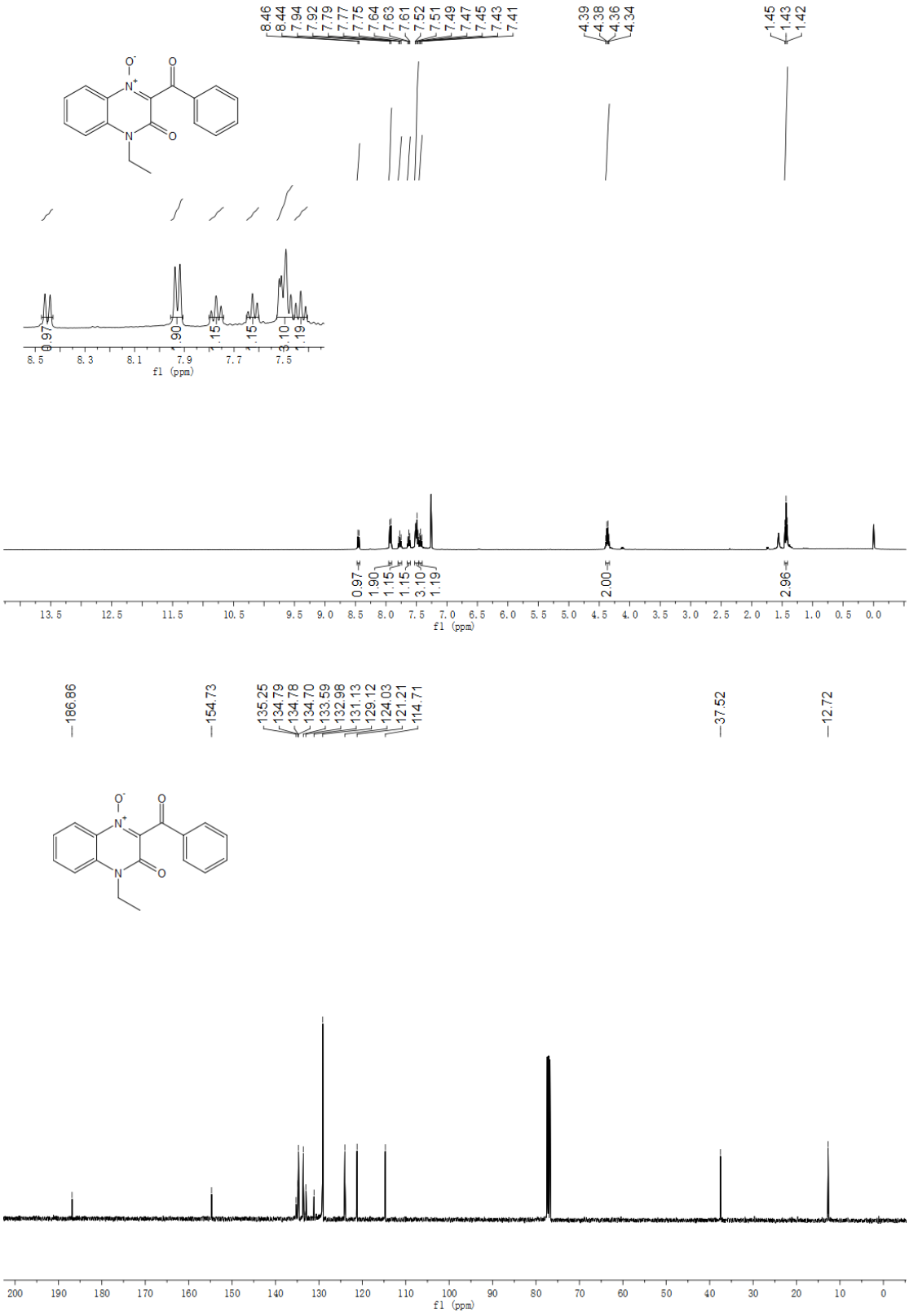
3ba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7.47
7.45
7.43
7.41
4.39
4.38
4.36
4.34
1.45
1.43
1.42
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1.90
1.15
1.15
3.10
1.19
2.00
2.96
f1 (ppm)
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134.79
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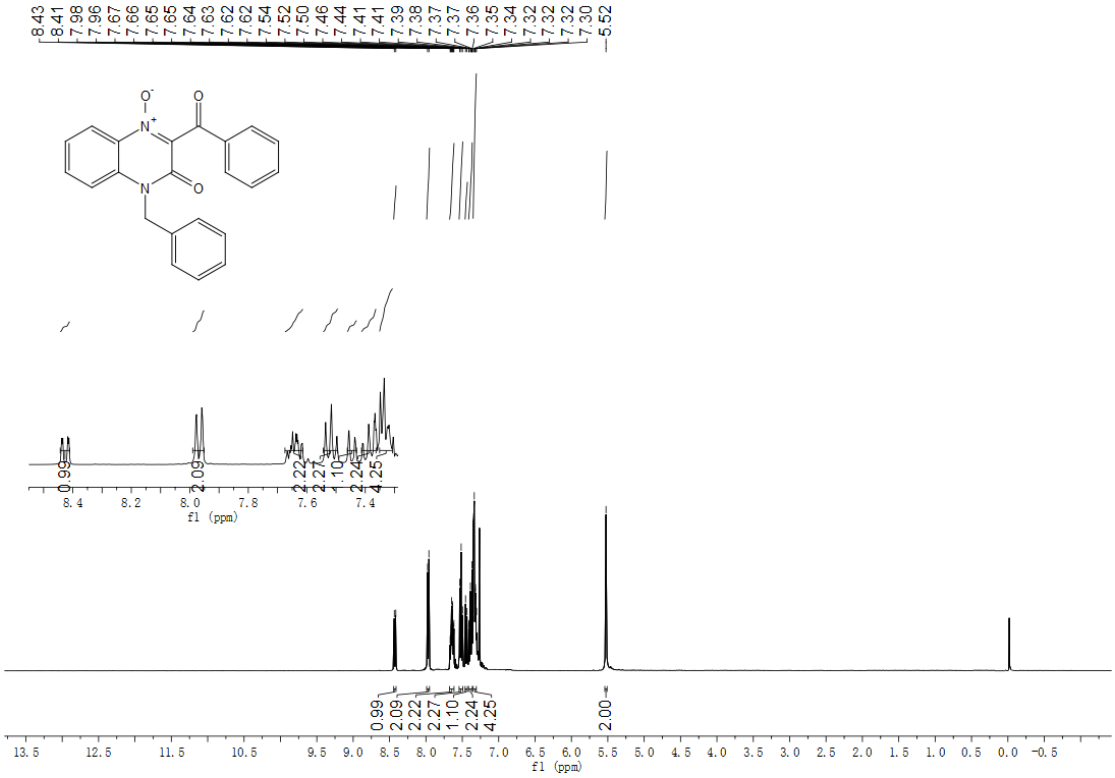
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7.41
7.39
7.38
7.37
7.37
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7.35
7.34
7.32
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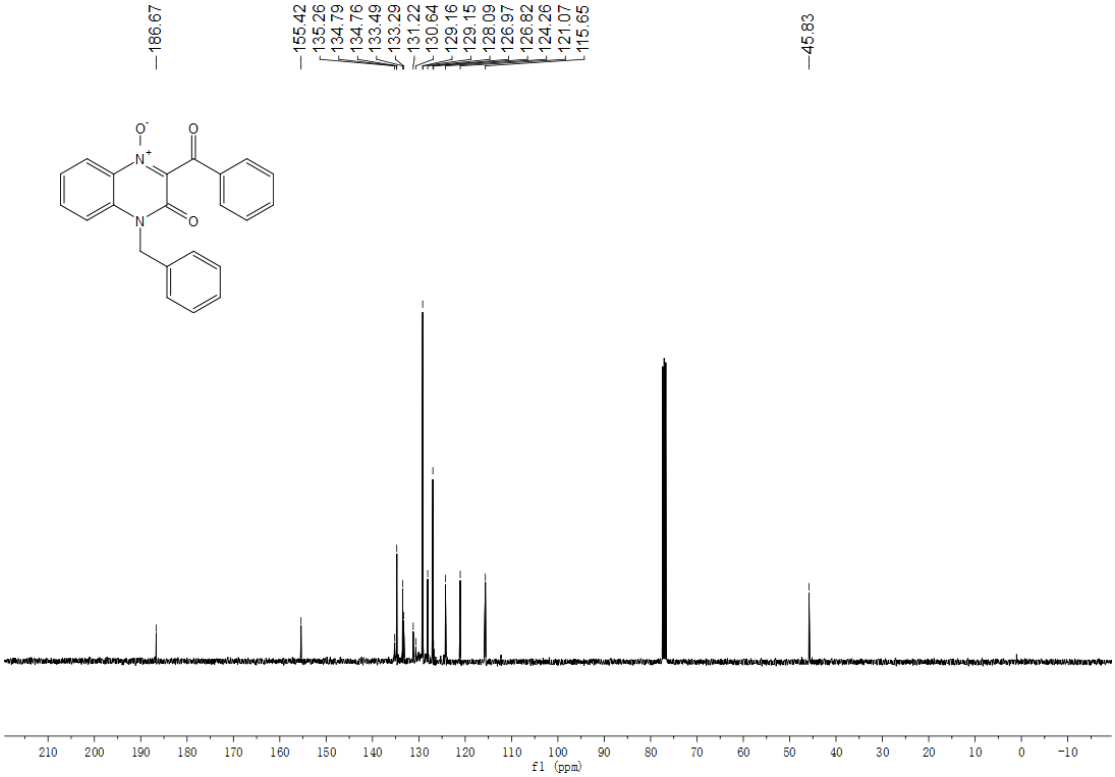
f1 (ppm)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124.26
121.07
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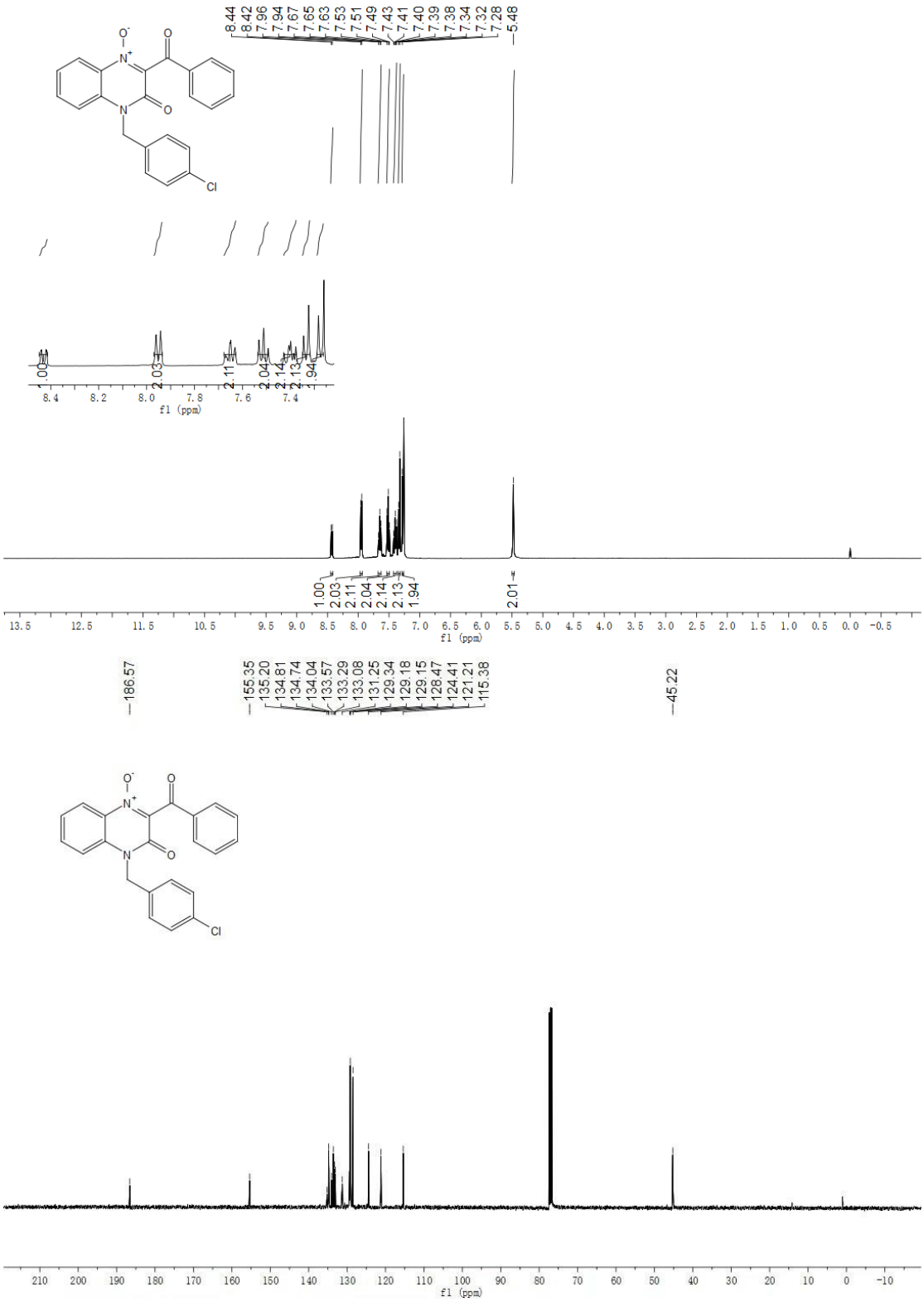
3da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7.67
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7.40
7.39
7.38
7.34
7.32
7.28
5.48
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2.03
2.11
2.04
2.14
2.13
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2.01
f1 (ppm)
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128.47
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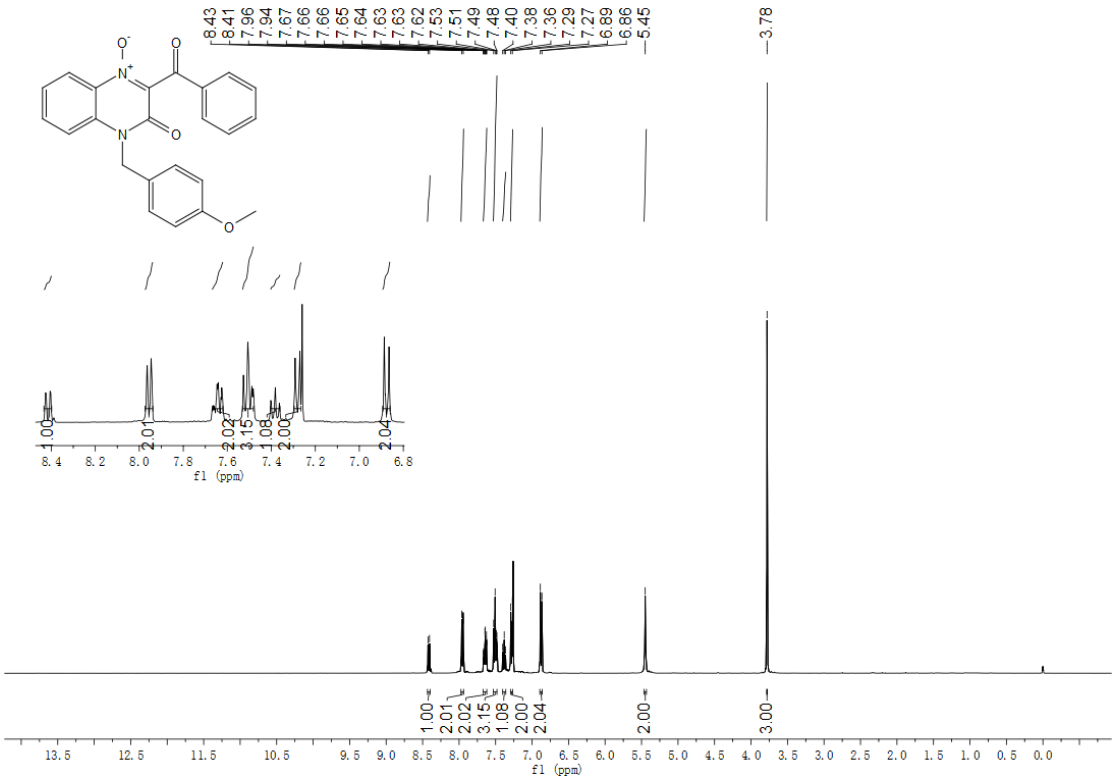
45.22
f1 (ppm)

3ea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7.29
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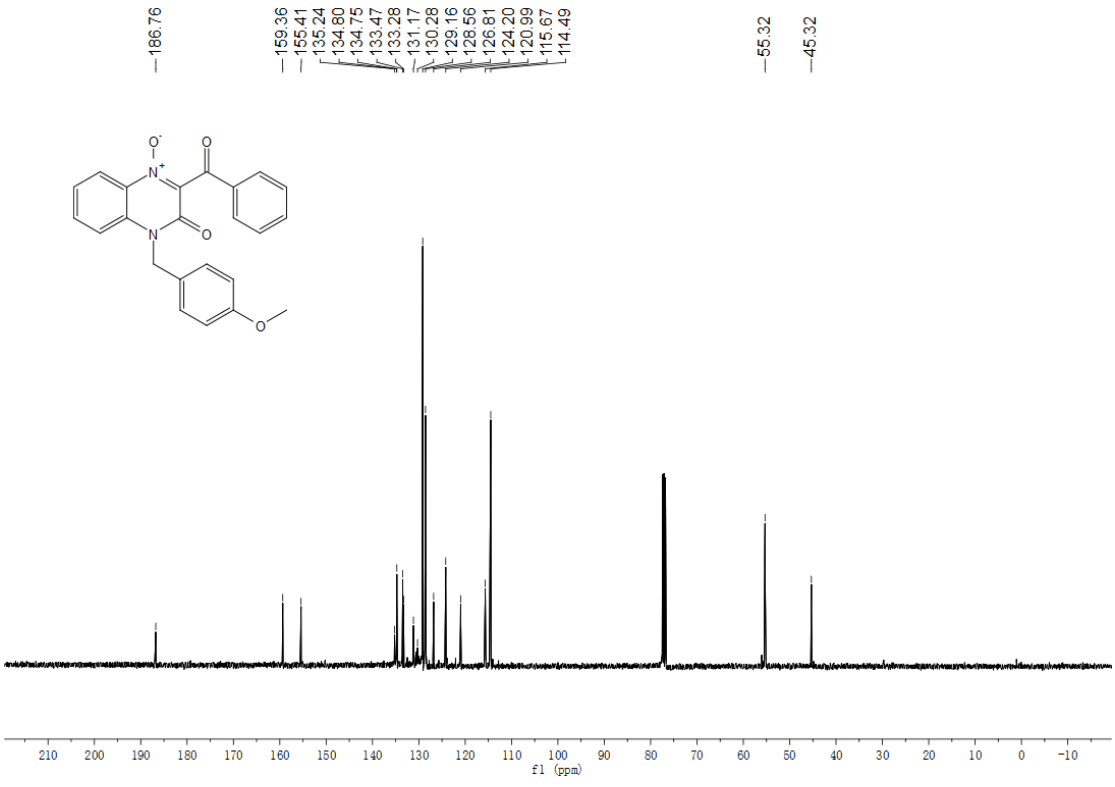
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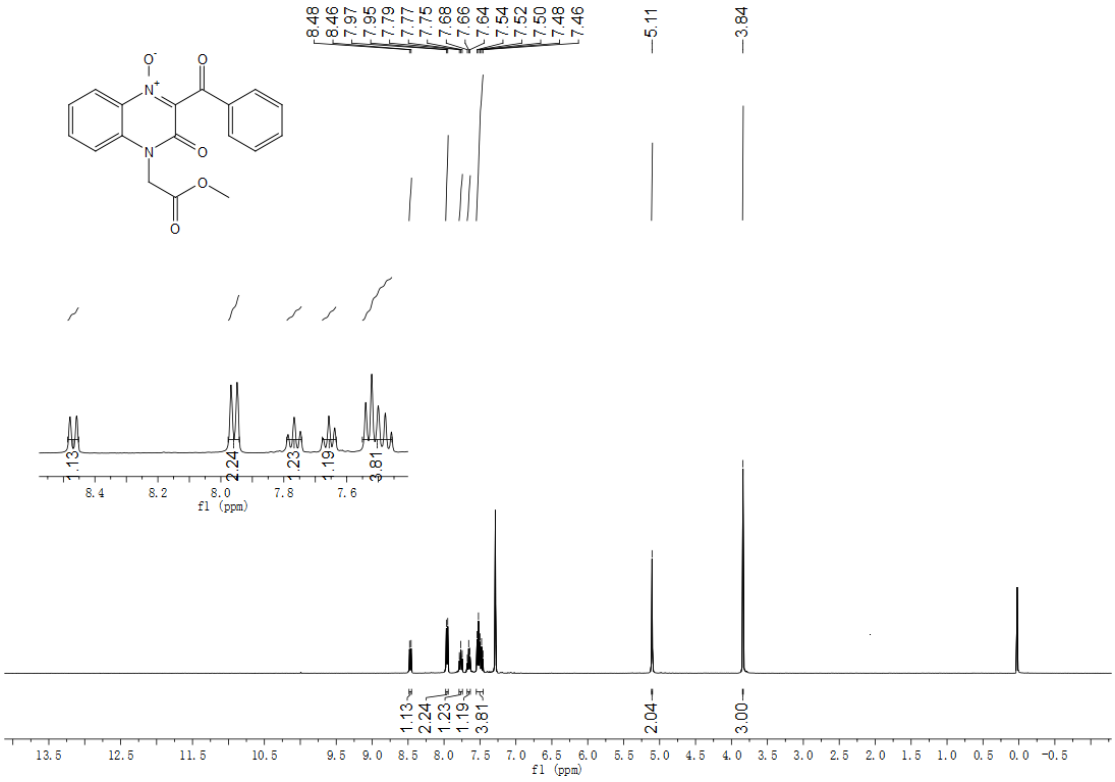
45.32
f1 (ppm)

3fa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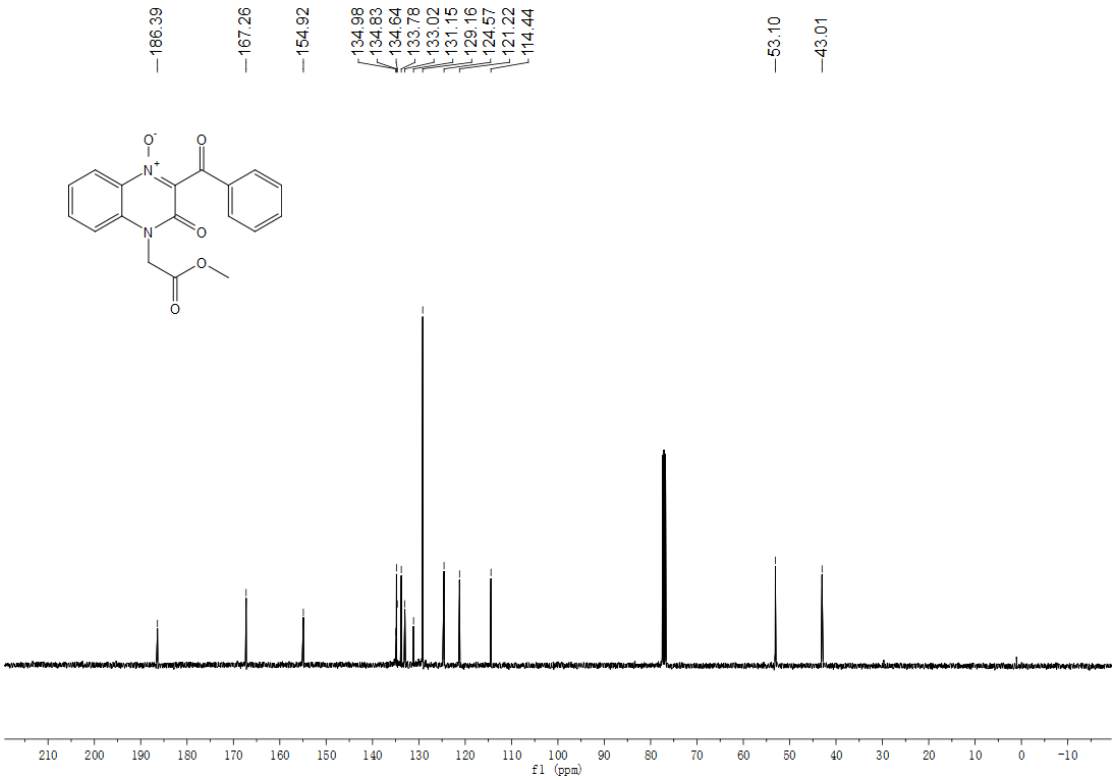
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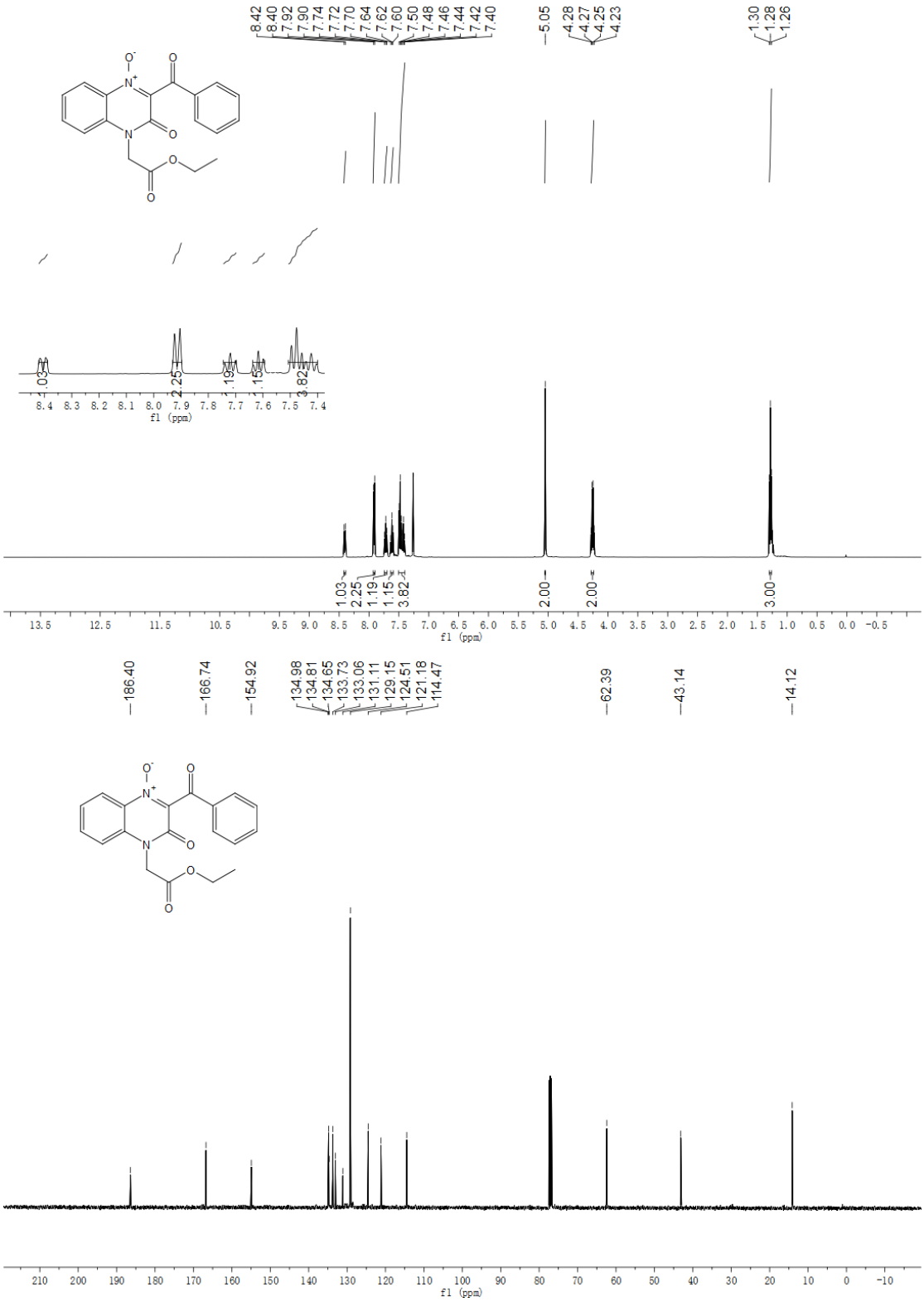
f1 (ppm)

3ga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7.46
7.44
7.42
7.40
5.05
4.28
4.27
4.25
4.23
1.30
1.28
1.26
1.03
2.25
1.19
1.15
3.82
2.00
2.00
3.00
f1 (ppm)
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166.74
154.92
134.98
134.81
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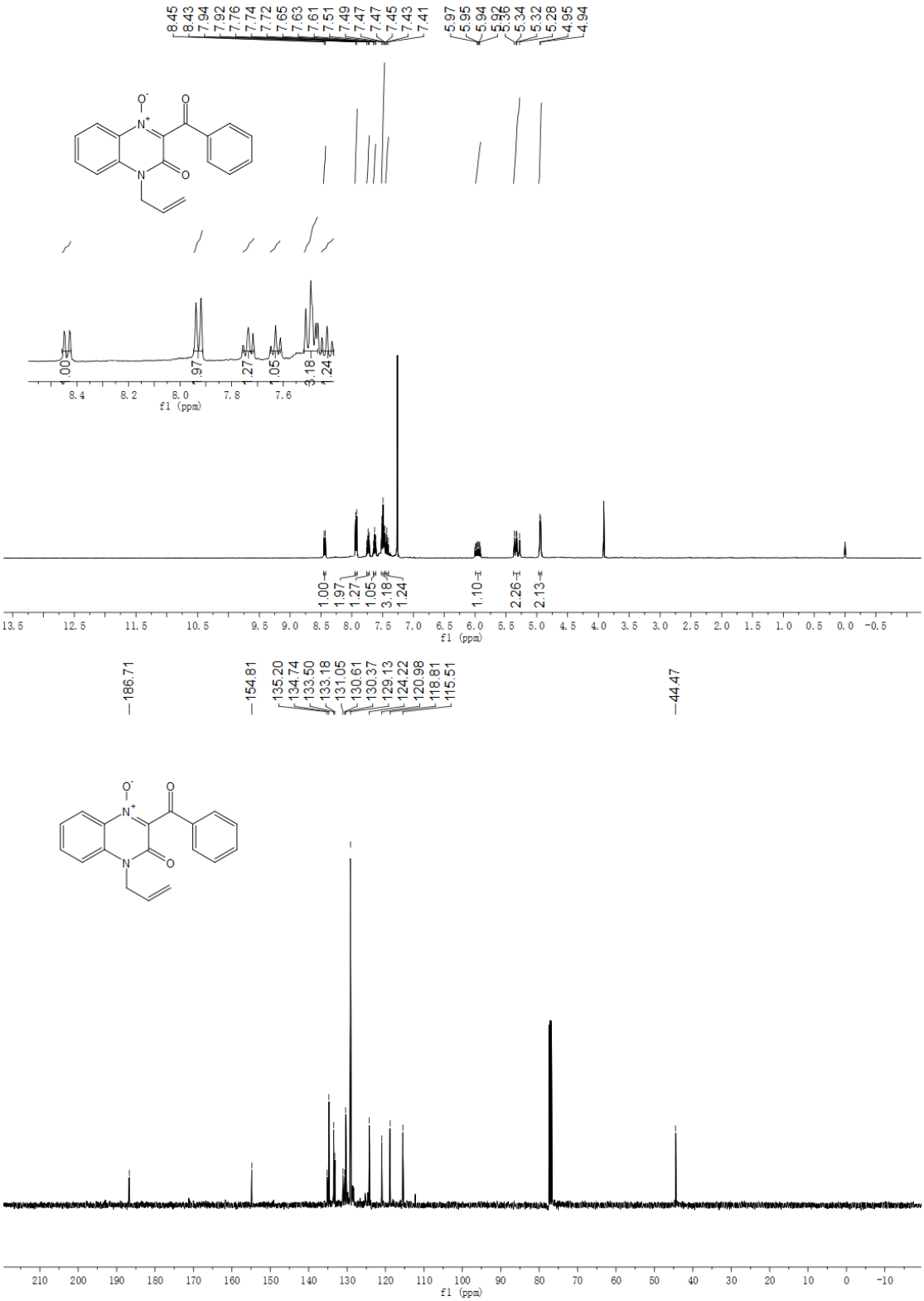
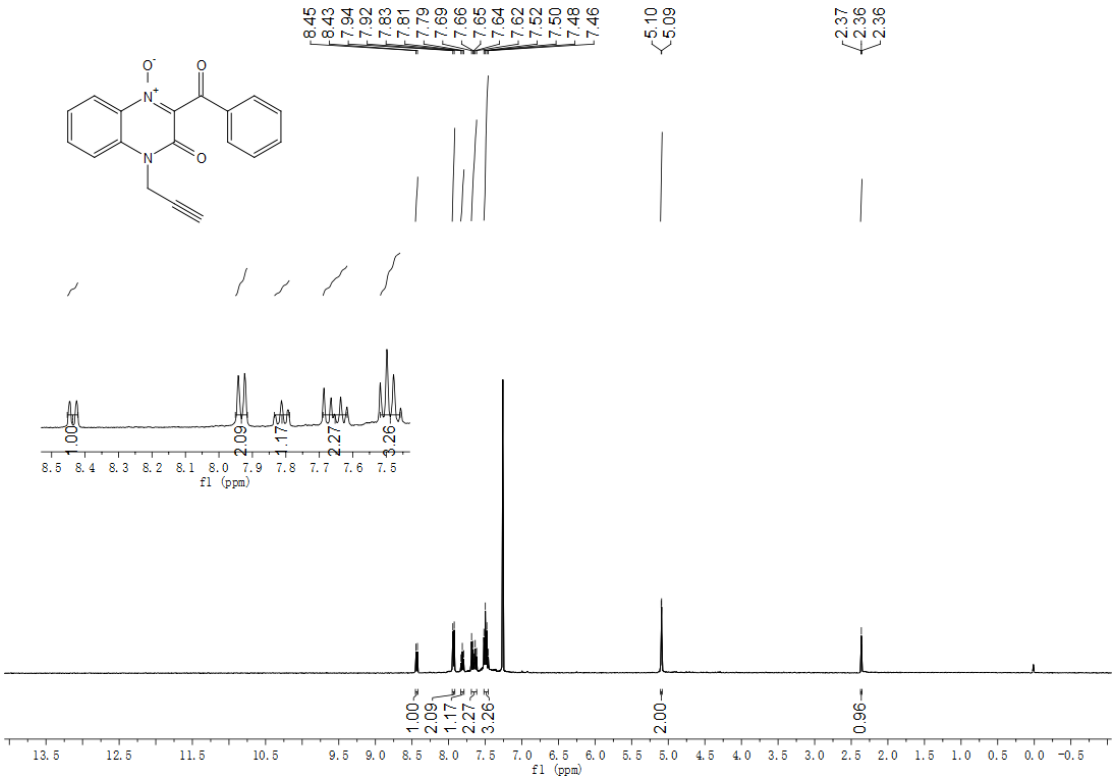
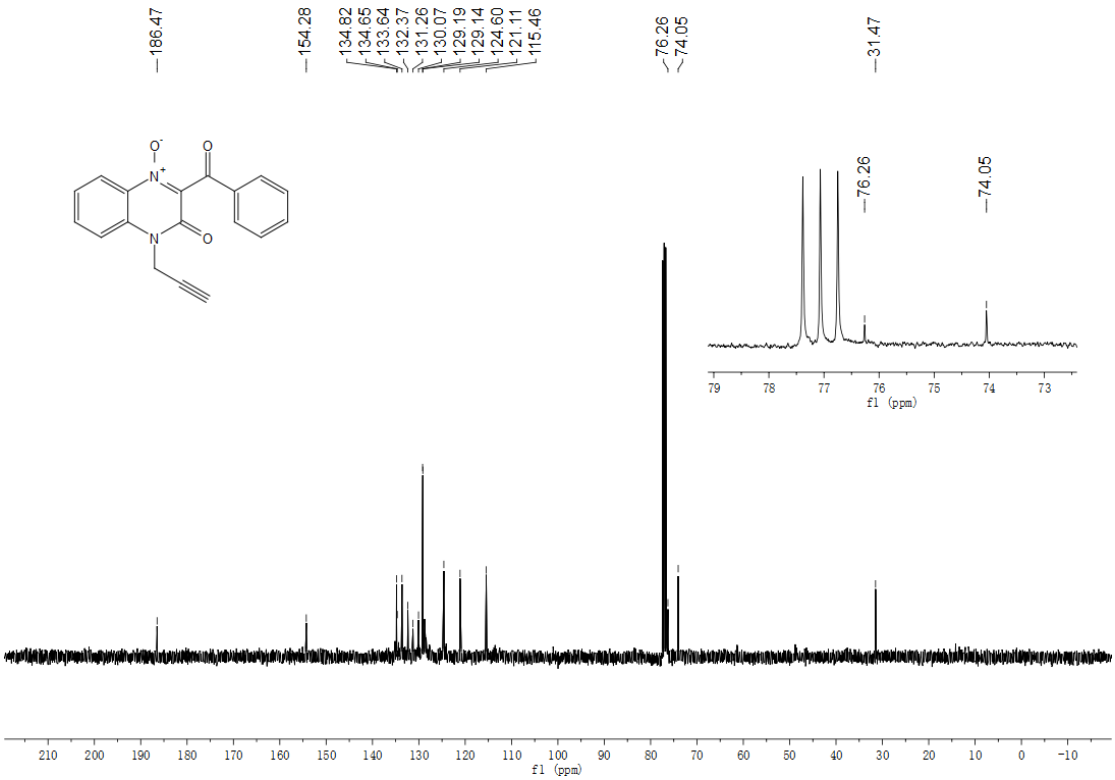
f1 (ppm)

3ha

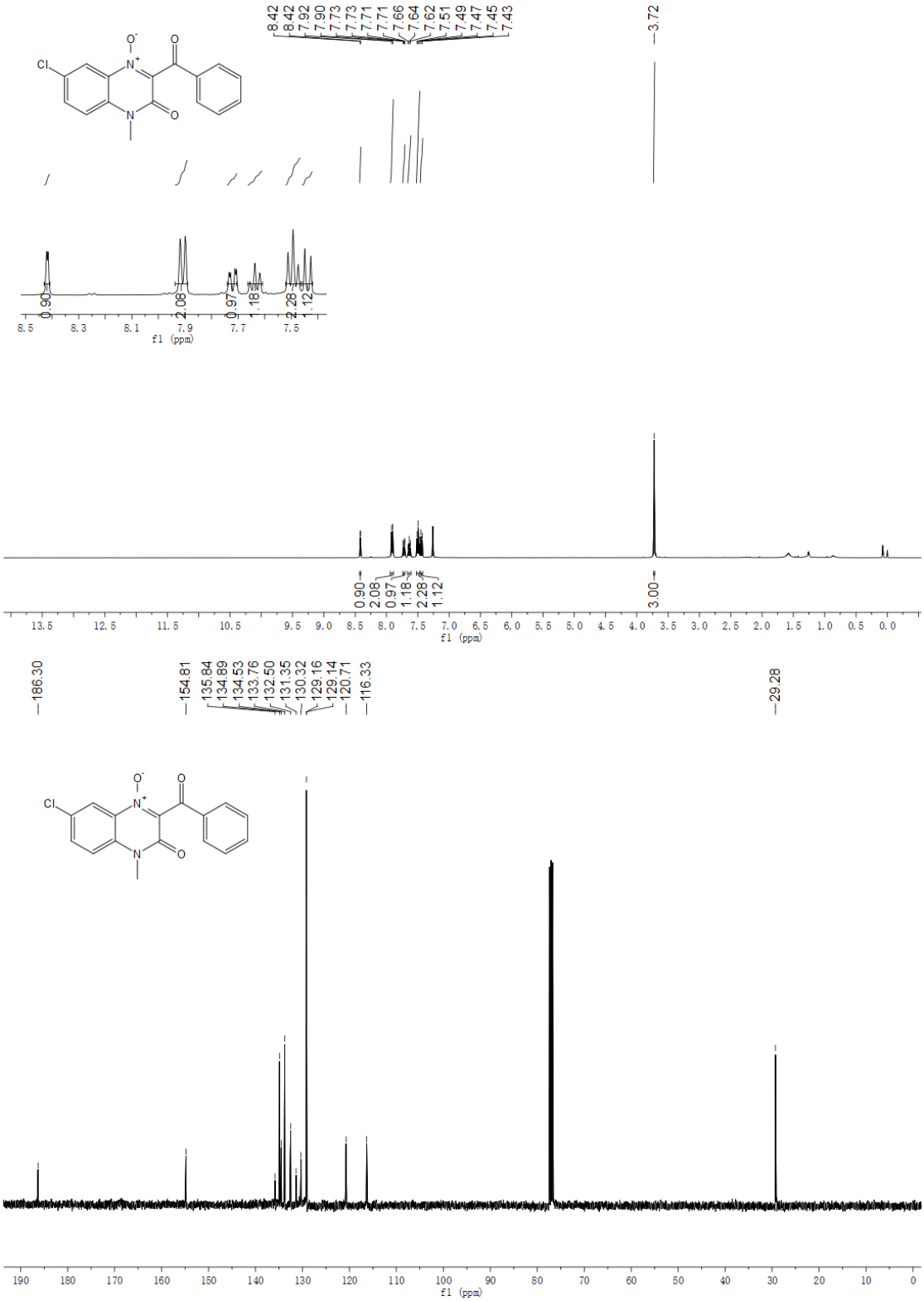

8.45
8.43
7.94
7.92
7.76
7.74
7.72
7.65
7.63
7.61
7.51
7.49
7.47
7.47
7.45
7.43
7.41
5.97
5.95
5.94
5.92
5.36
5.34
5.32
5.28
4.95
4.94
1.00
1.97
1.27
1.05
3.18
1.24
1.10
2.26
2.13
f1 (ppm)
186.71
154.81
135.20
134.74
133.50
133.18
131.05
130.61
130.37
129.13
124.22
120.98
118.81
115.51
44.47
f1 (ppm)

3ia

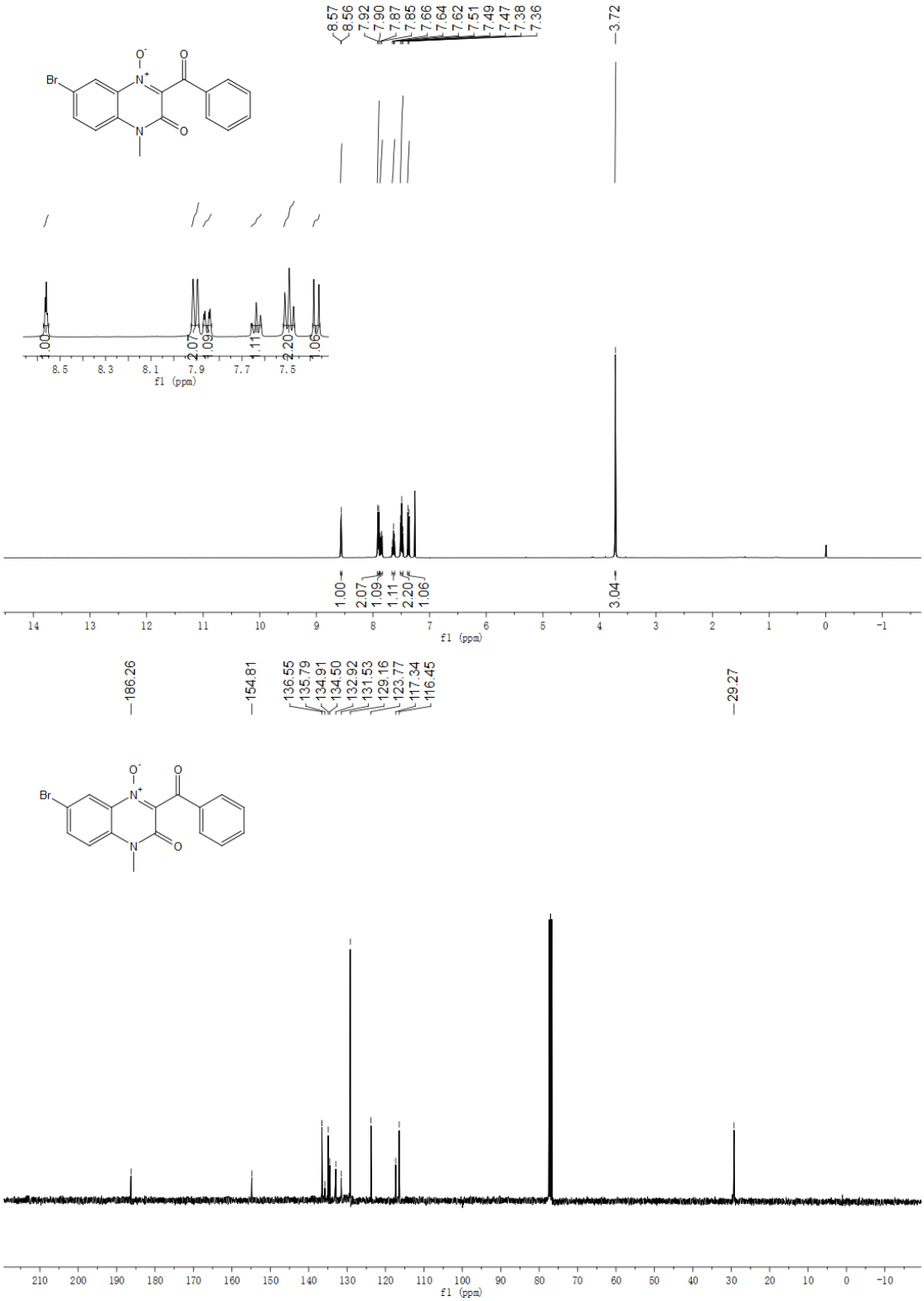

8.45
8.43
7.94
7.92
7.83
7.81
7.79
7.69
7.66
7.65
7.64
7.62
7.52
7.50
7.48
7.46
5.10
5.09
2.37
2.36
2.36
1.00
2.09
1.17
2.27
3.26
2.00
0.96
f1 (ppm)


186.47
154.28
134.82
134.65
133.64
132.37
131.26
130.07
129.19
129.14
124.60
121.11
115.46
76.26
74.05
31.47
f1 (ppm)

3ja


8.42
8.42
7.92
7.90
7.73
7.73
7.71
7.71
7.66
7.64
7.62
7.51
7.49
7.47
7.45
7.43
3.72
0.90
2.08
0.97
1.18
2.28
1.12
3.00
f1 (ppm)
186.30
154.81
135.84
134.89
134.53
133.76
132.50
131.35
130.32
129.16
129.14
120.71
116.33
29.28
Cl
O
N
O
O

3ka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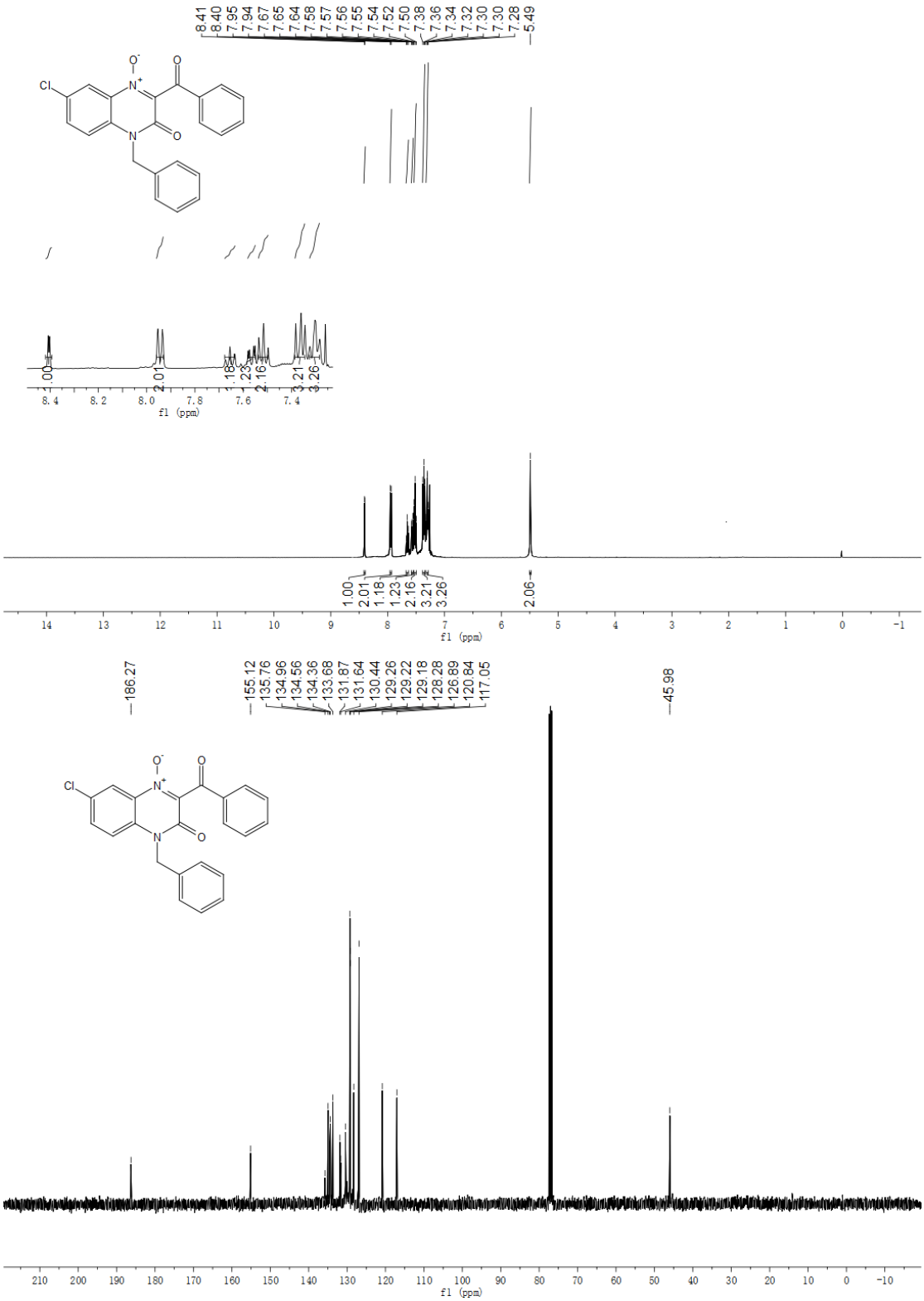
8.56
7.92
7.90
7.87
7.85
7.66
7.64
7.62
7.51
7.49
7.47
7.38
7.36
3.72
1.00
2.07
1.09
1.11
2.20
1.06
3.04
f1 (ppm)
186.26
154.81
136.55
135.79
134.91
134.50
132.92
131.53
129.16
123.77
117.34
116.45
29.27
f1 (ppm)

3la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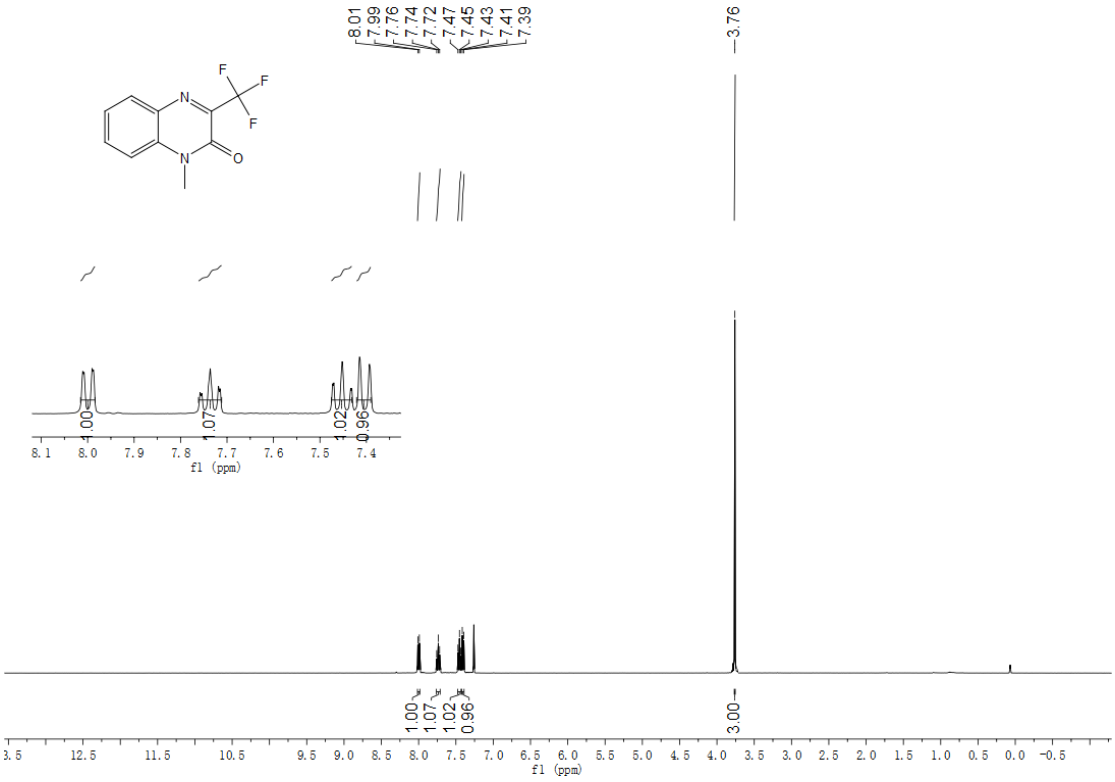
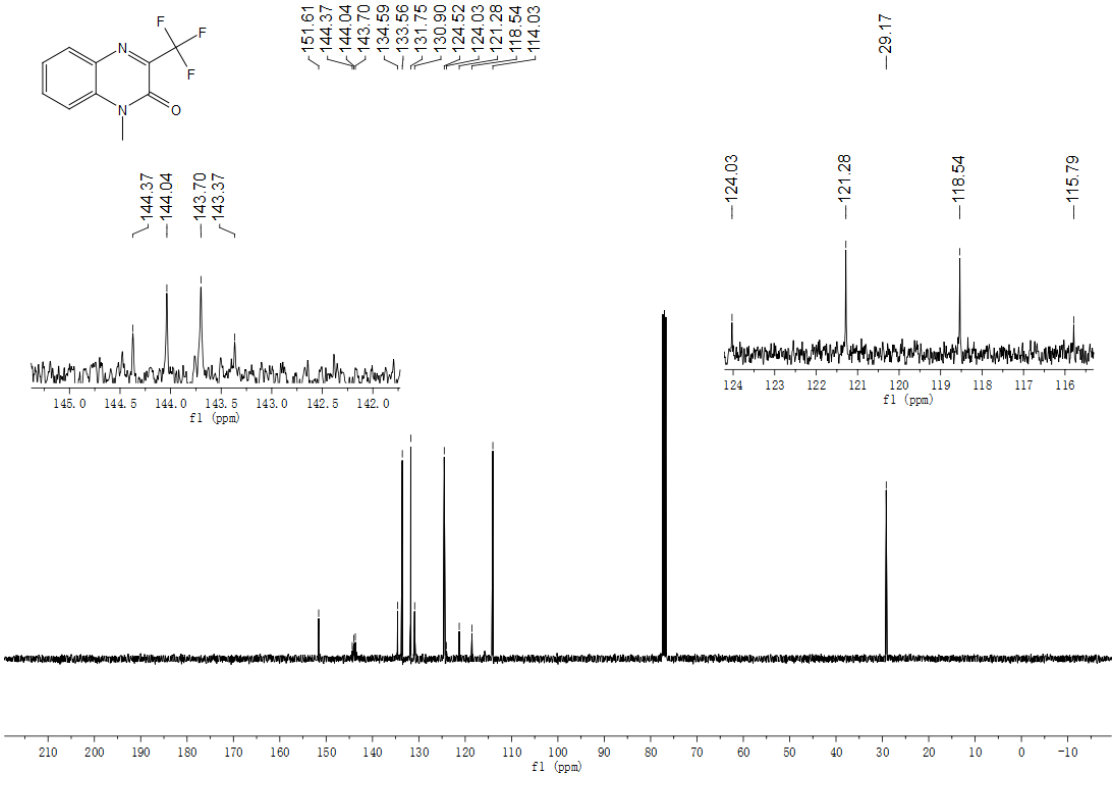
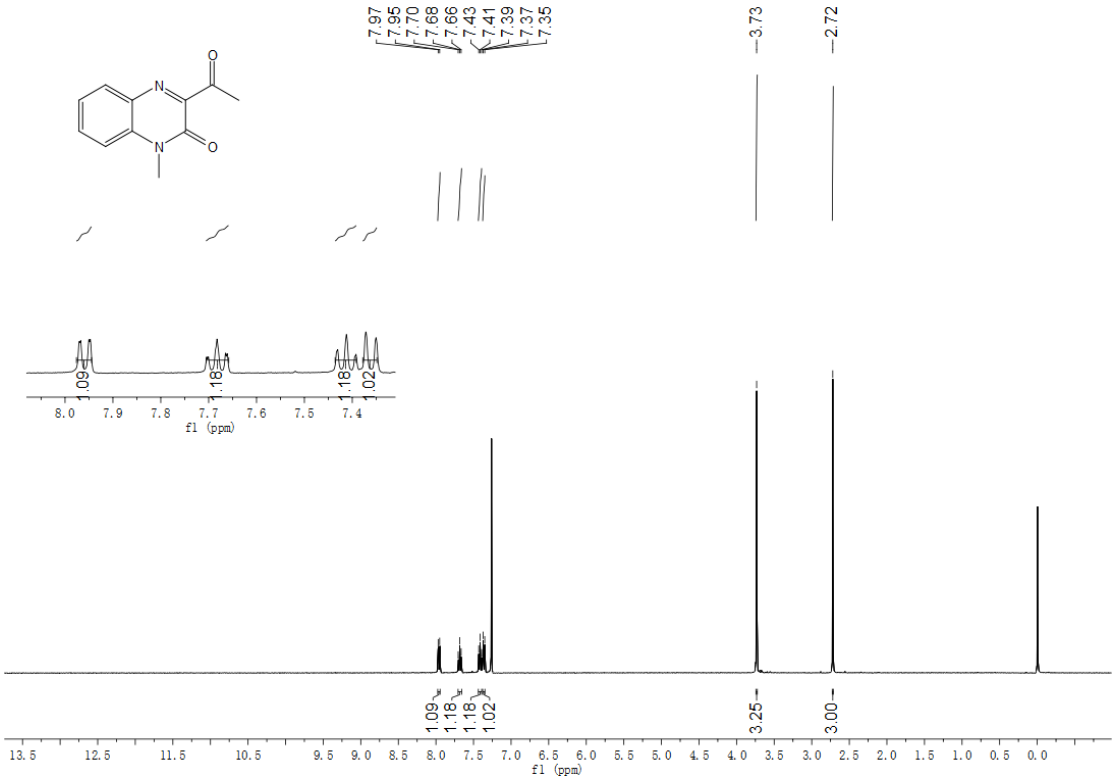
8.40
7.95
7.94
7.67
7.65
7.64
7.58
7.57
7.56
7.55
7.54
7.52
7.50
7.38
7.36
7.34
7.32
7.30
7.30
7.28
5.49
Cl
O
N
O
N
O
1.00
2.01
1.18
1.23
2.16
3.21
3.26
2.06
f1 (ppm)
186.27
155.12
135.76
134.96
134.56
134.36
133.68
131.87
131.64
130.44
129.26
129.22
129.18
128.28
126.89
120.84
117.05
45.98
f1 (ppm)

12


8.01
7.99
7.76
7.74
7.72
7.47
7.45
7.43
7.41
7.39
3.76
1.00
1.07
1.02
0.96
3.00
f1 (ppm)

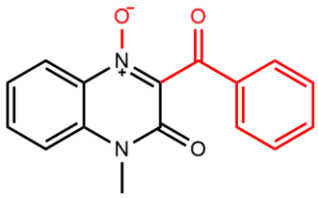

151.61
144.37
144.04
143.70
134.59
133.56
131.75
130.90
124.52
124.03
121.28
118.54
114.03
29.17
144.37
144.04
143.70
143.37
124.03
121.28
118.54
115.79
f1 (ppm)

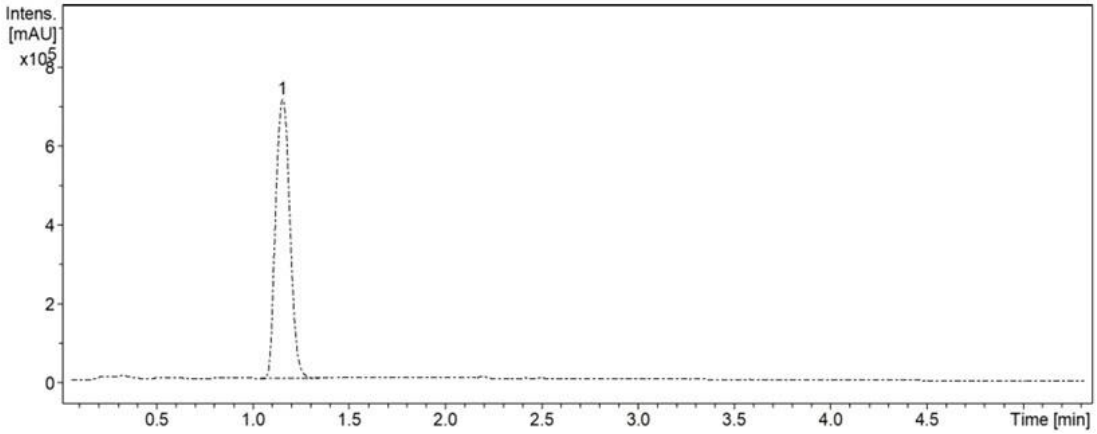
19


7.97
7.95
7.70
7.68
7.66
7.43
7.41
7.39
7.37
7.35
3.73
2.72
1.09
1.18
1.18
1.02
3.25
3.00
f1 (ppm)

9. LC-MS spectra

3aa





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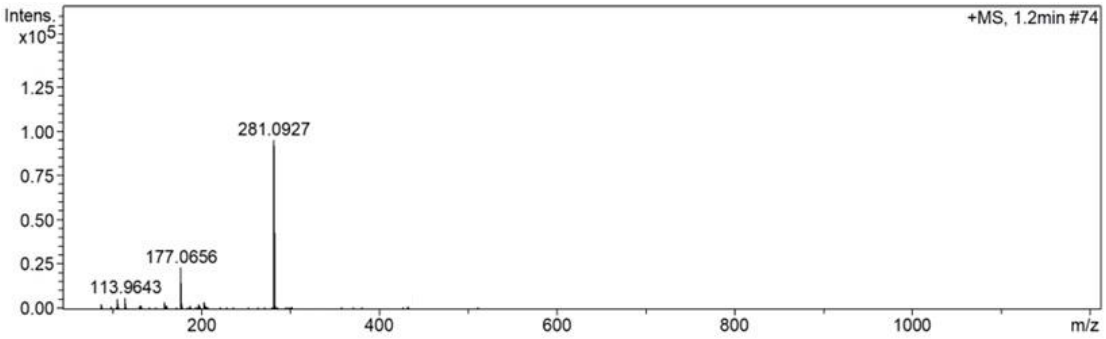


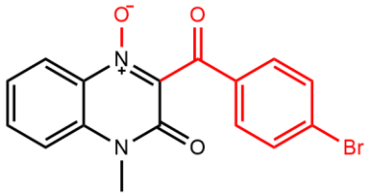
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
281.0927								
	1	C 16 H 13 N 2 O 3	281.0921	-2.1	11.5	ok	even	5.17
	2	C 12 H 9 N 8 O	281.0894	-11.7	12.5	ok	even	9.76
	3	C 21 H 13 O	281.0961	12.2	15.5	ok	even	32.05
	4	C 15 H 18 Cl O 3	281.0939	4.4	6.5	ok	even	142.71
	5	C 11 H 14 Cl N 6 O	281.0912	-5.2	7.5	ok	even	155.01
	6	C 10 H 18 Cl N 2 O 5	281.0899	-9.9	2.5	ok	even	159.90

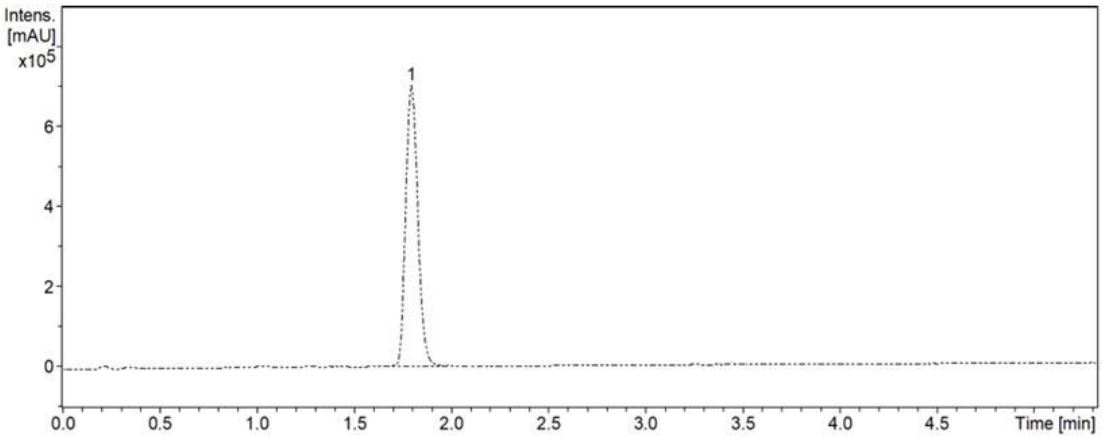
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ab





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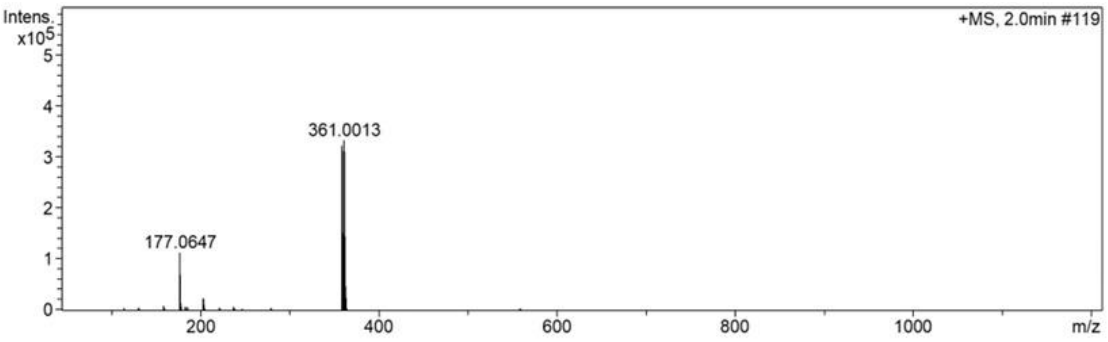


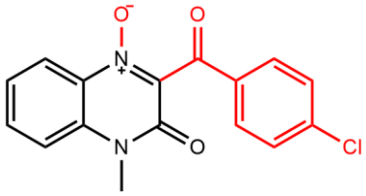
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
359.0036								
	1	C 12 H 8 Br N 8 O	358.9999	-10.3	12.5	ok	even	19.01
	2	C 16 H 12 Br N 2 O 3	359.0026	-2.8	11.5	ok	even	19.10
	3	C 21 H 12 Br O	359.0066	8.4	15.5	ok	even	41.45
	4	C 16 H 7 O 10	359.0034	-0.6	13.5	ok	even	488.29
	5	C 17 H 3 N 4 O 6	359.0047	3.1	18.5	ok	even	559.96
	6	C 12 H 3 N 6 O 8	359.0007	-8.1	14.5	ok	even	562.24
361.0013								
	1	C 13 H 5 N 4 O 9	361.0051	10.5	13.5	ok	even	4.22
	2	C 9 H N 10 O 7	361.0024	3.1	14.5	ok	even	9.91
	3	C 12 H 9 O 13	361.0038	6.8	8.5	ok	even	11.69
	4	C 8 H 5 N 6 O 11	361.0011	-0.6	9.5	ok	even	22.25
	5	C 19 H 5 O 8	360.9979	-9.4	17.5	ok	even	32.11
	6	C 20 H N 4 O 4	360.9992	-5.7	22.5	ok	even	46.38
	7	C 25 H N 2 O 2	361.0033	5.4	26.5	ok	even	73.68
	8	C 8 H 10 Br N 8 O 4	361.0003	-2.8	7.5	ok	even	432.47
	9	C 9 H 6 Br N 12	361.0016	0.9	12.5	ok	even	432.57
	10	C 13 H 10 Br N 6 O 2	361.0043	8.3	11.5	ok	even	437.50
	11	C 12 H 14 Br N 2 O 6	361.0030	4.6	6.5	ok	even	438.03

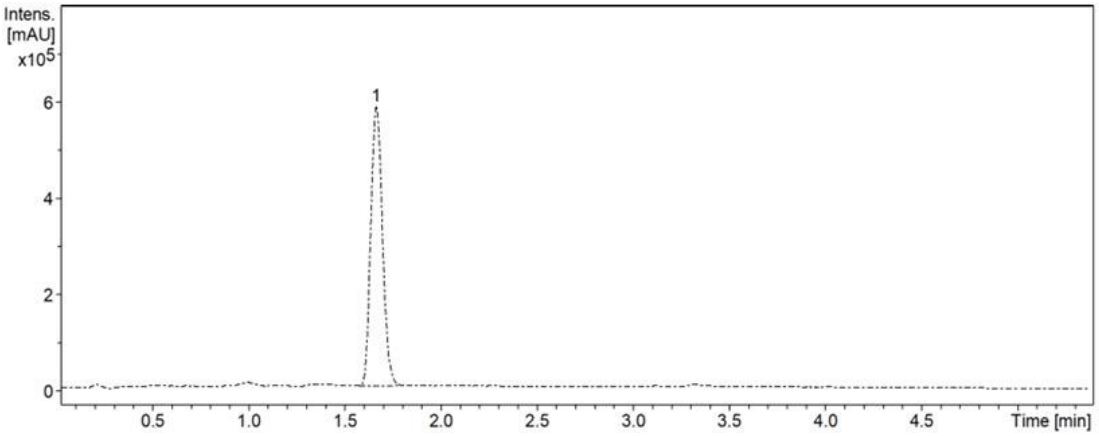
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ac





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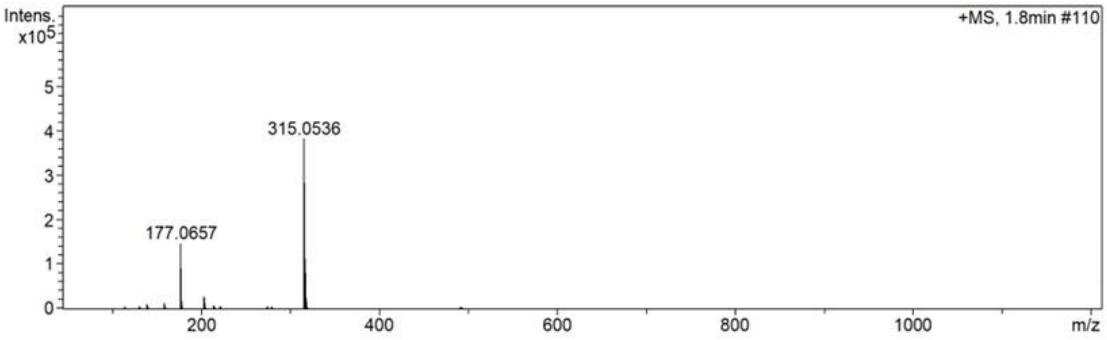


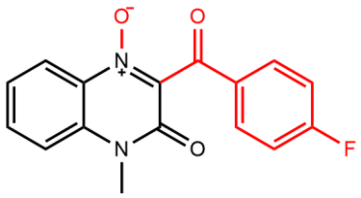
Meas. m/z 315.0536	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
	1	C 12 H 8 Cl N 8 O	315.0504	-10.2	12.5	ok	even	19.96
	2	C 16 H 12 Cl N 2 O 3	315.0531	-1.7	11.5	ok	even	23.16
	3	C 21 H 12 Cl O	315.0571	11.1	15.5	ok	even	37.24
	4	C 16 H 11 O 7	315.0499	-11.7	11.5	ok	even	152.23
	5	C 17 H 7 N 4 O 3	315.0513	-7.5	16.5	ok	even	155.51
	6	C 9 H 15 O 12	315.0558	6.9	2.5	ok	even	155.83
	7	C 10 H 11 N 4 O 8	315.0571	11.2	7.5	ok	even	156.92
	8	C 22 H 7 N 2 O	315.0553	5.3	20.5	ok	even	158.99

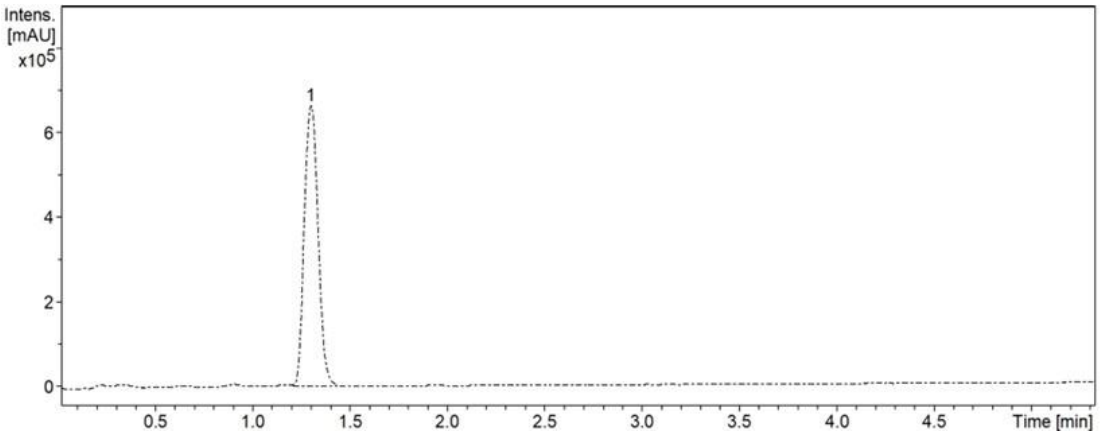
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ad





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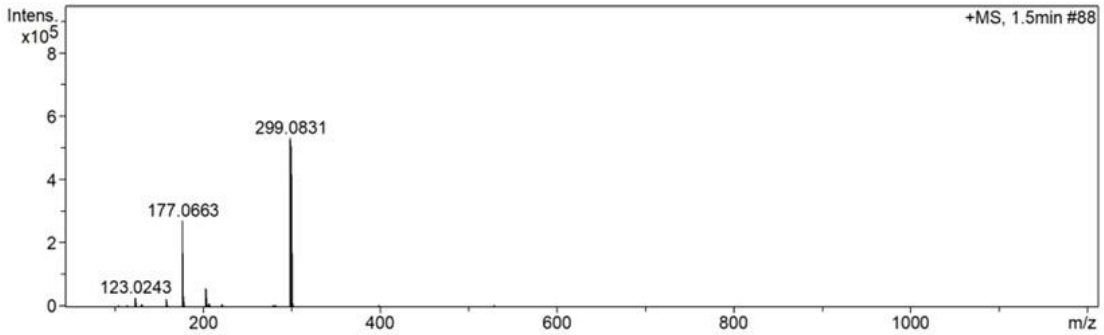


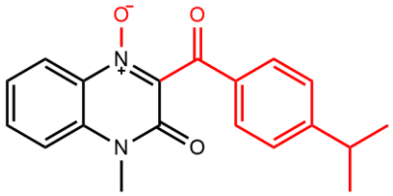
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
299.0831								
	1	C 19 H 11 N 2 O 2	299.0815	-5.4	15.5	ok	even	2.17
	2	C 21 H 12 F O	299.0867	11.9	15.5	ok	even	6.13
	3	C 16 H 12 F N 2 O 3	299.0826	-1.6	11.5	ok	even	21.06
	4	C 24 H 11	299.0855	8.0	19.5	ok	even	25.06

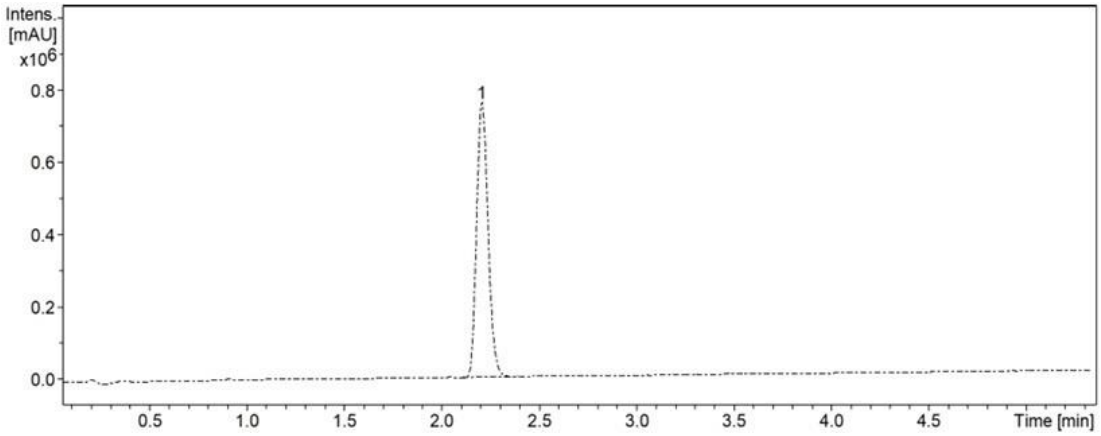
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ae





#	RT [min]	Area	Area Frac. %
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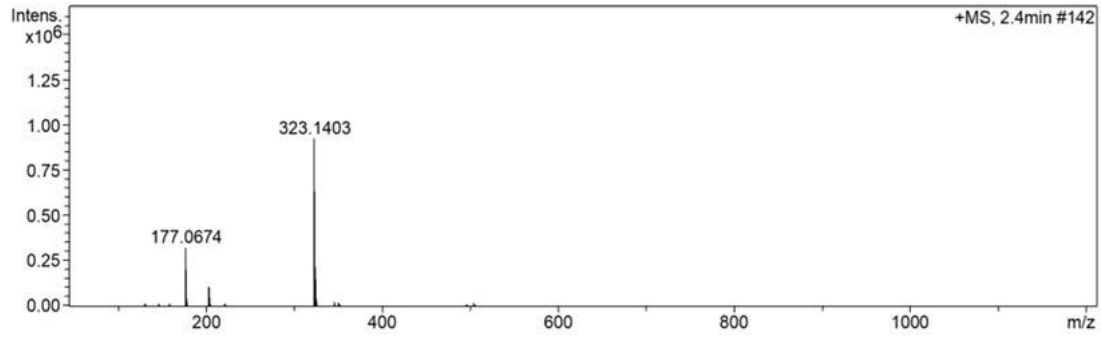


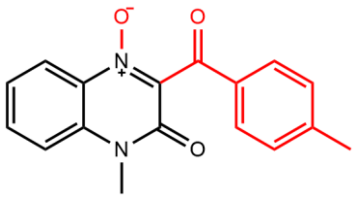
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
323.1403								
	1	C 19 H 19 N 2 O 3	323.1390	-4.0	11.5	ok	even	11.70
	2	C 24 H 19 O	323.1430	8.4	15.5	ok	even	15.47
	3	C 15 H 15 N 8 O	323.1363	-12.3	12.5	ok	even	24.68

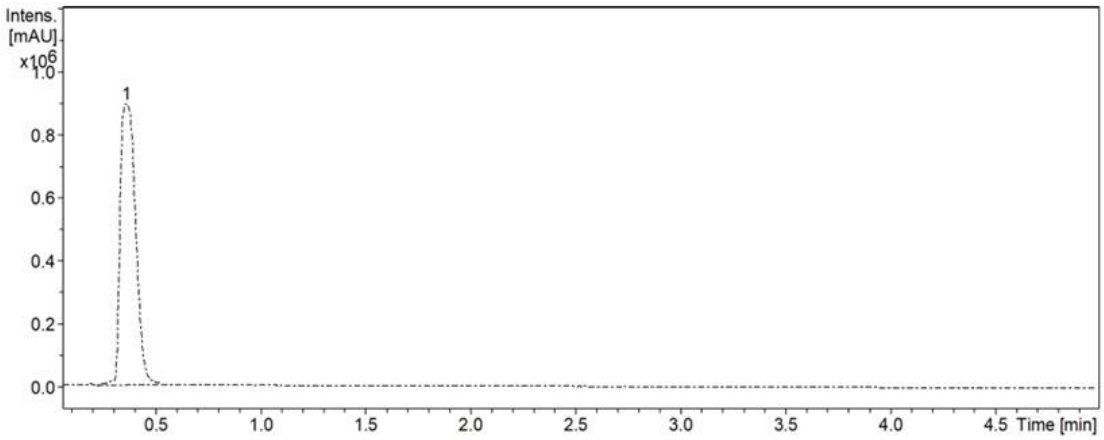
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3af





#	RT [min]	Area	Area Frac. %
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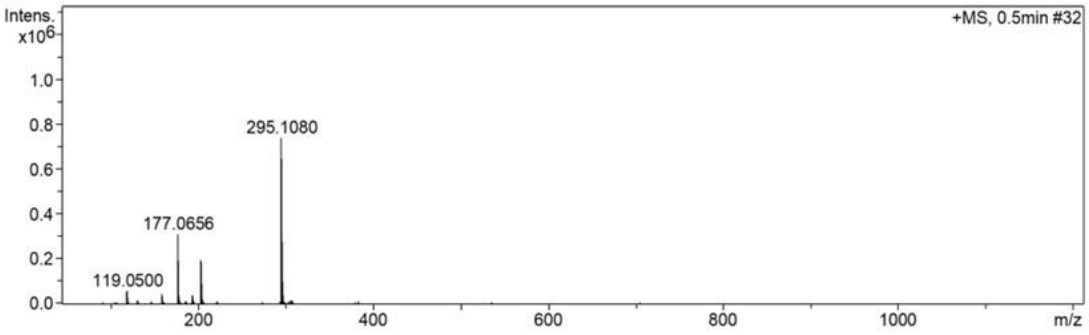


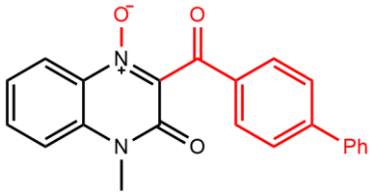
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
295.1080								
	1	C 13 H 11 N 8 O	295.1050	-10.2	12.5	ok	even	9.87
	2	C 17 H 15 N 2 O 3	295.1077	-1.1	11.5	ok	even	20.78
	3	C 22 H 15 O	295.1117	12.5	15.5	ok	even	47.81

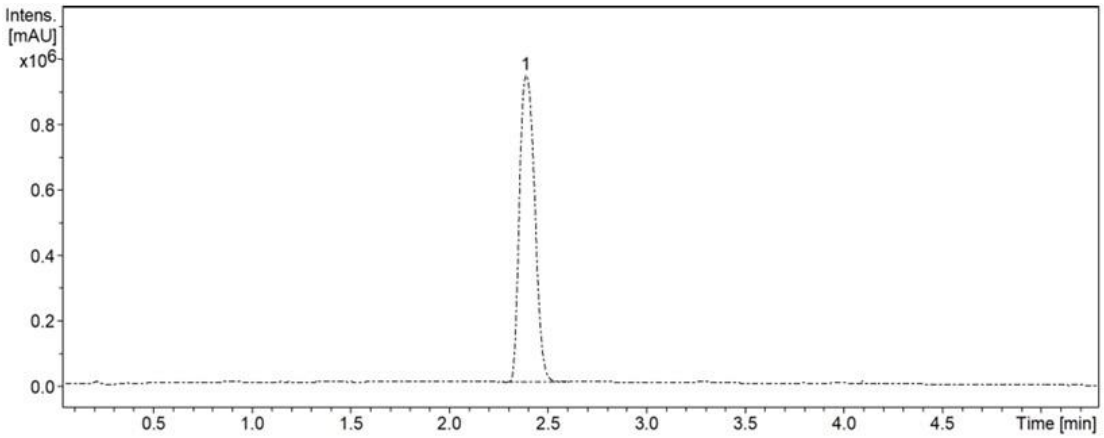
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ag





#	RT [min]	Area	Area Frac. %
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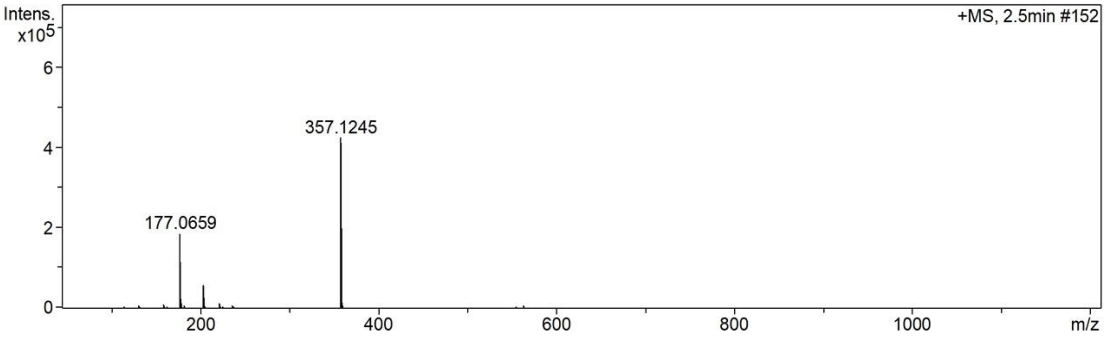


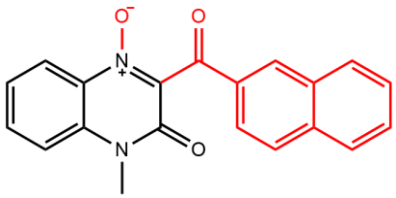
Meas. m/z 357.1245	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
	1	C 22 H 17 N 2 O 3	357.1234	-3.1	15.5	ok	even	5.48
	2	C 18 H 13 N 8 O	357.1207	-10.6	16.5	ok	even	18.74
	3	C 27 H 17 O	357.1274	8.1	19.5	ok	even	18.89

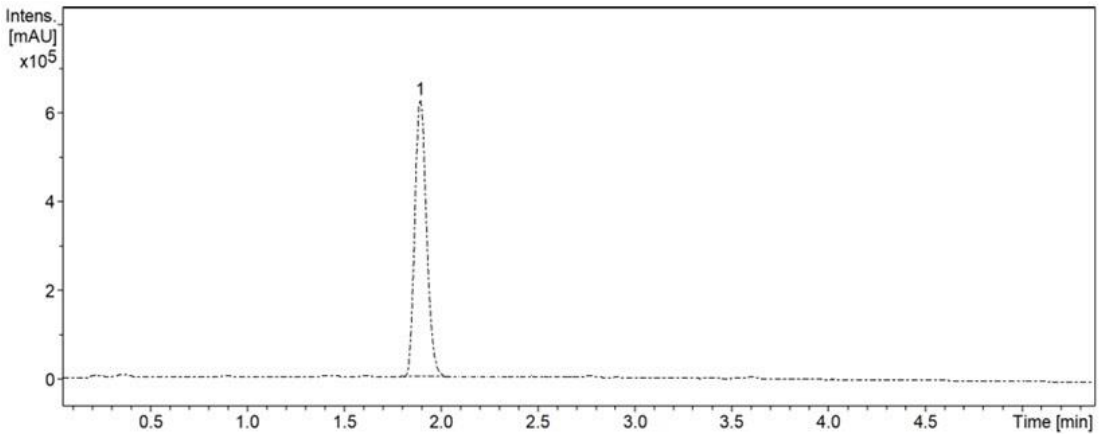
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ah





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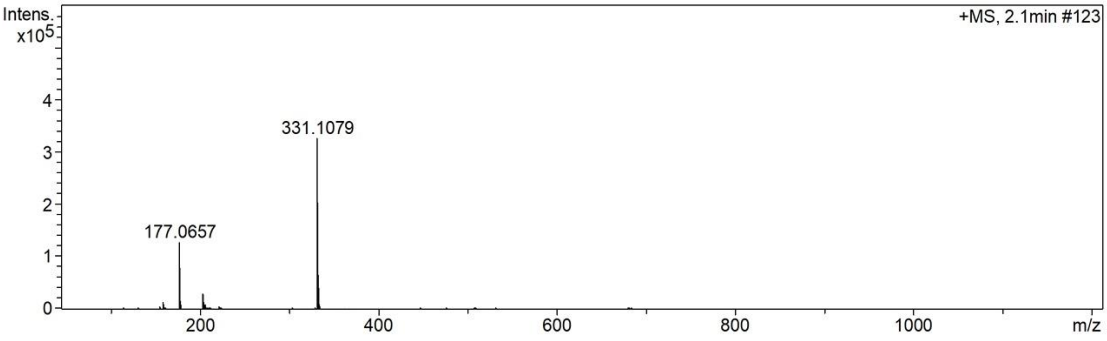


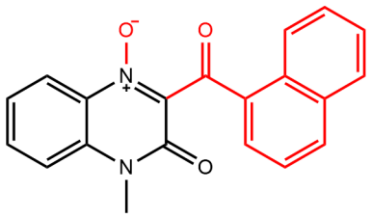
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
331.1079								
	1	C 16 H 11 N 8 O	331.1050	-8.7	15.5	ok	even	4.81
	2	C 20 H 15 N 2 O 3	331.1077	-0.6	14.5	ok	even	15.14
	3	C 25 H 15 O	331.1117	11.6	18.5	ok	even	42.24

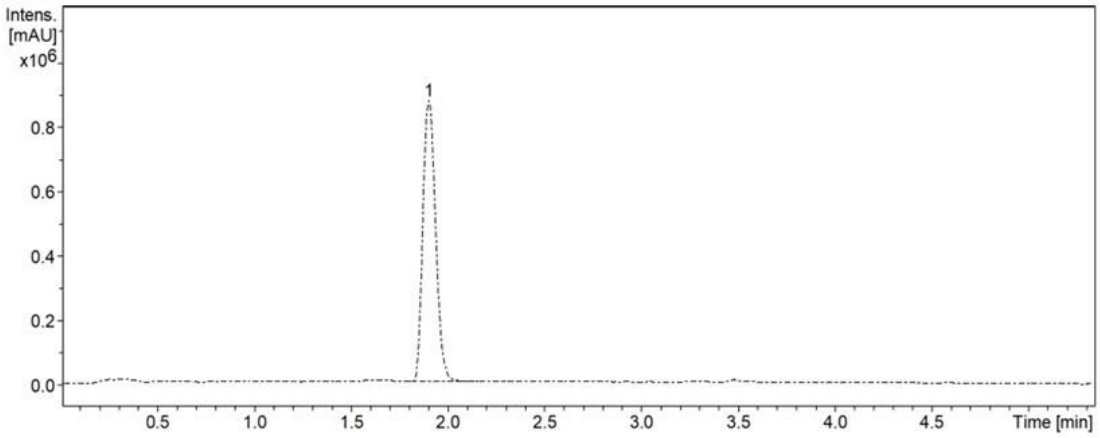
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ai





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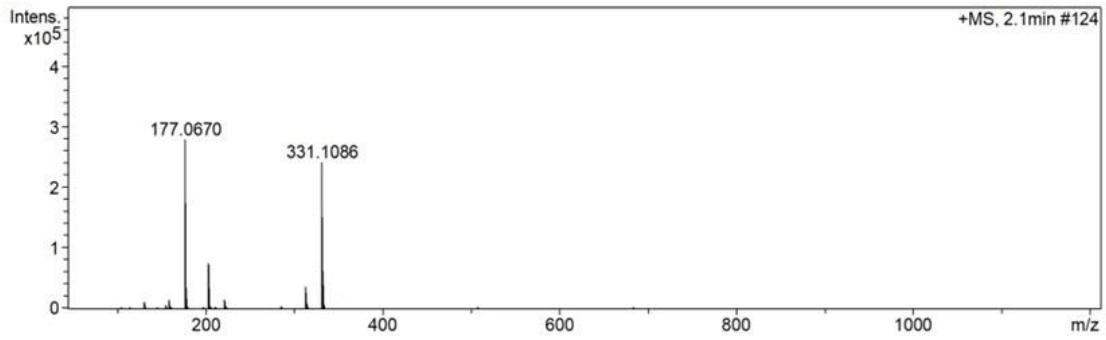


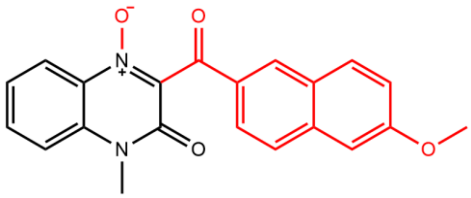
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
331.1086								
	1	C 25 H 15 O	331.1117	9.4	18.5	ok	even	7.74
	2	C 20 H 15 N 2 O 3	331.1077	-2.7	14.5	ok	even	19.38

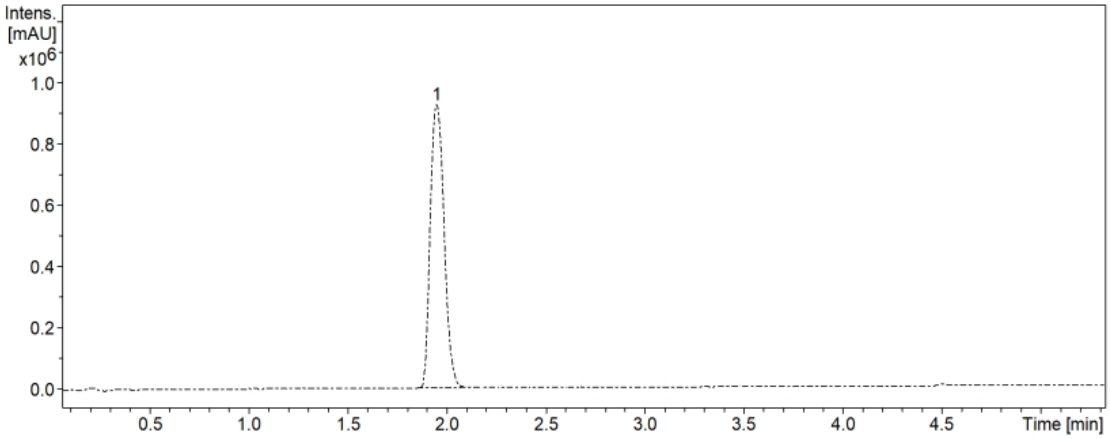
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3aj





#	RT [min]	Area	Area Frac. %
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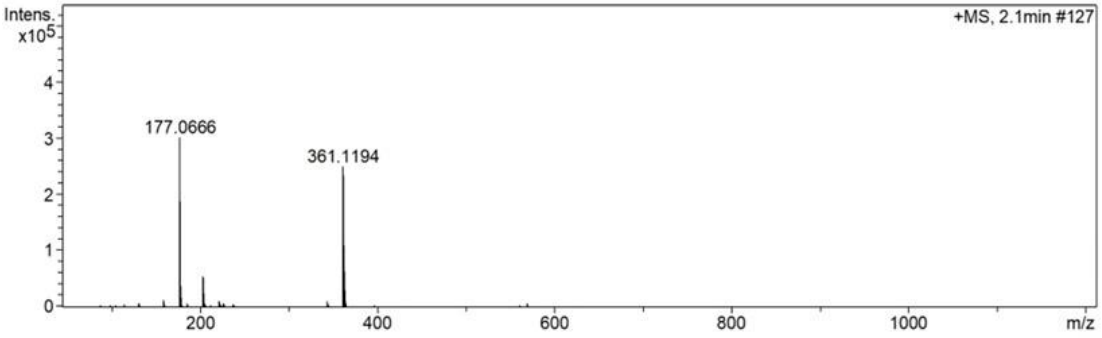


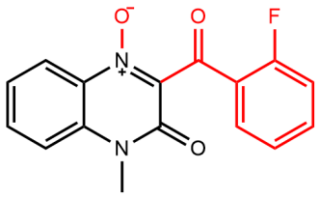
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
361.1194								
	1	C 26 H 17 O 2	361.1223	8.2	18.5	ok	even	7.14
	2	C 22 H 13 N 6	361.1196	0.7	19.5	ok	even	7.93
	3	C 21 H 17 N 2 O 4	361.1183	-3.0	14.5	ok	even	20.42

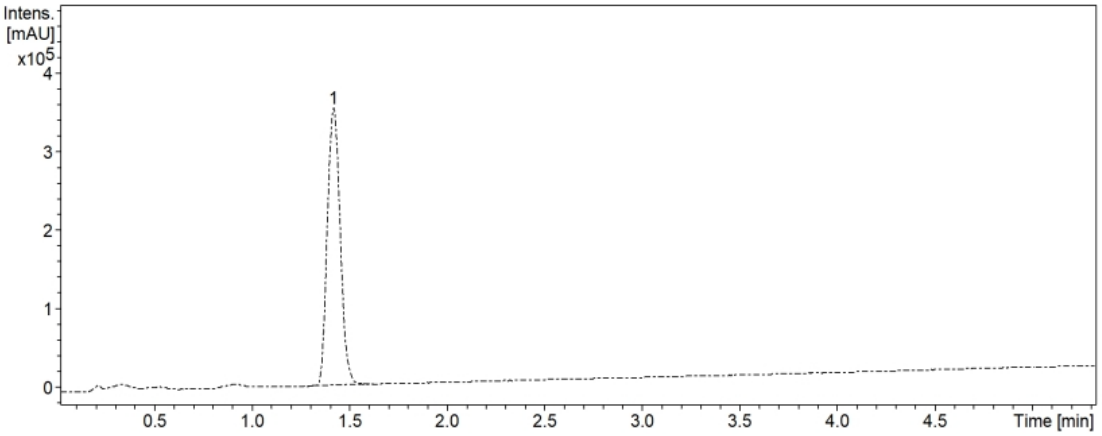
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ak





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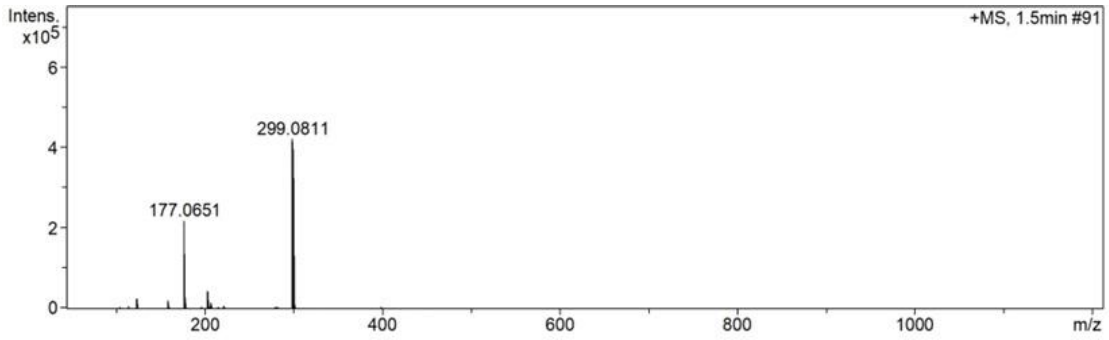


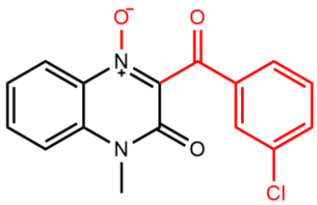
Meas. m/z 299.0811	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
	1	C 19 H 11 N 2 O 2	299.0815	1.4	15.5	ok	even	1.70
	2	C 15 H 7 N 8	299.0788	-7.6	16.5	ok	even	12.48
	3	C 16 H 12 F N 2 O 3	299.0826	5.2	11.5	ok	even	17.81
	4	C 14 H 11 N 4 O 4	299.0775	-12.1	11.5	ok	even	25.95

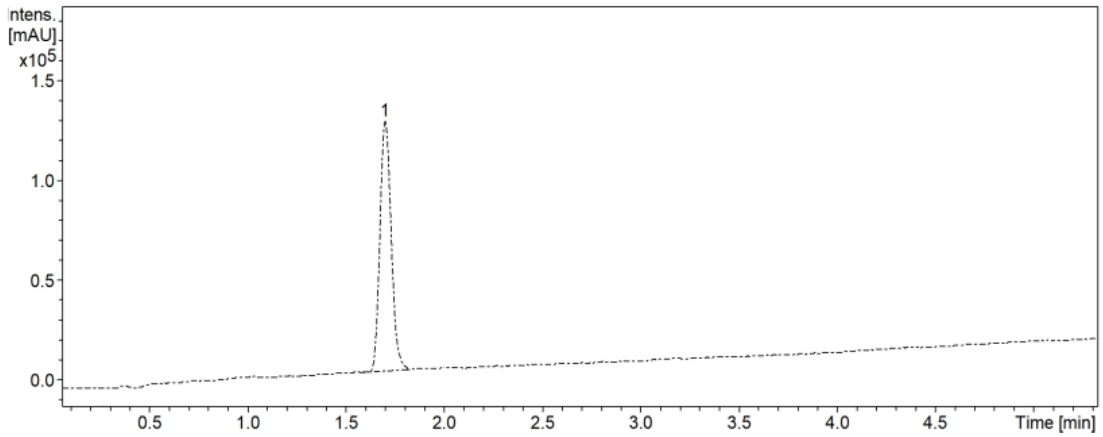
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3al





#	RT [min]	Area	Area Frac. %
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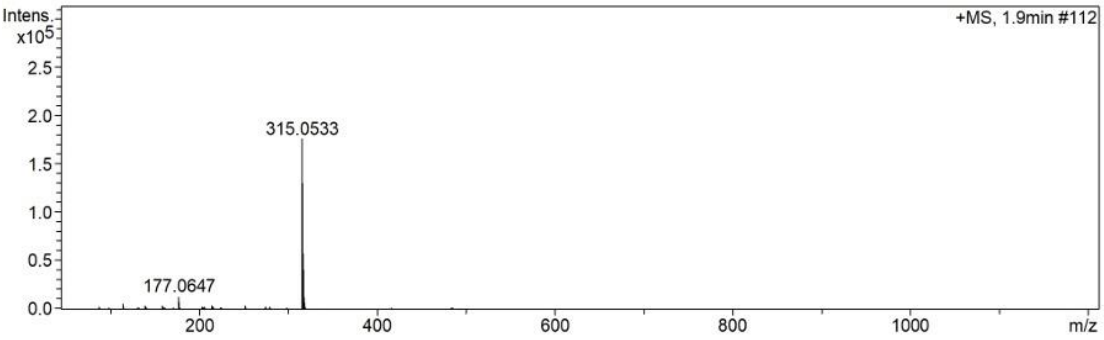


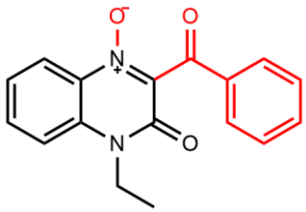
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
315.0533								
	1	C 12 H 8 Cl N 8 O	315.0504	-9.2	12.5	ok	even	5.25
	2	C 16 H 12 Cl N 2 O 3	315.0531	-0.7	11.5	ok	even	15.18
	3	C 21 H 12 Cl O	315.0571	12.1	15.5	ok	even	36.45
	4	C 16 H 11 O 7	315.0499	-10.7	11.5	ok	even	173.59
	5	C 9 H 15 O 12	315.0558	7.9	2.5	ok	even	174.71
	6	C 10 H 11 N 4 O 8	315.0571	12.2	7.5	ok	even	176.68
	7	C 17 H 7 N 4 O 3	315.0513	-6.5	16.5	ok	even	177.43
	8	C 22 H 7 N 2 O	315.0553	6.3	20.5	ok	even	181.48

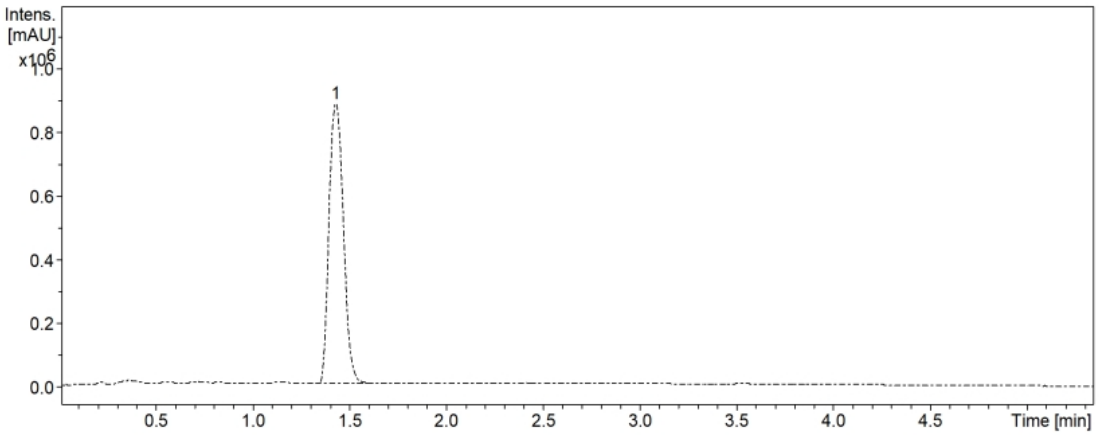
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ba





#	RT [min]	Area	Area Frac. %
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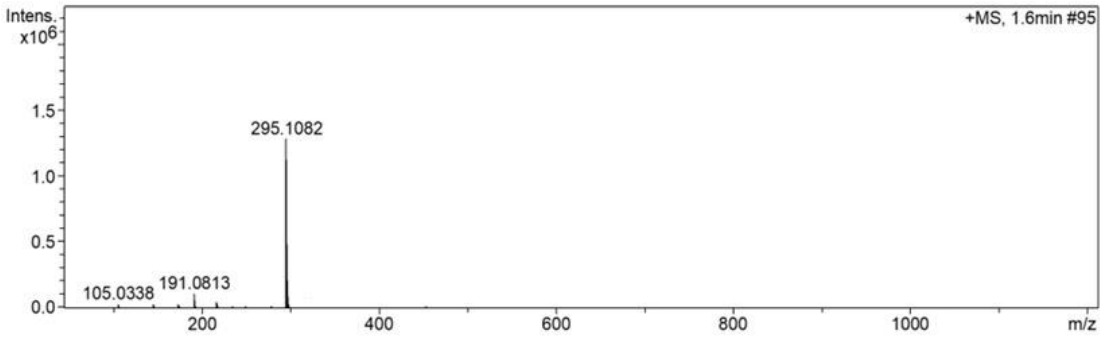


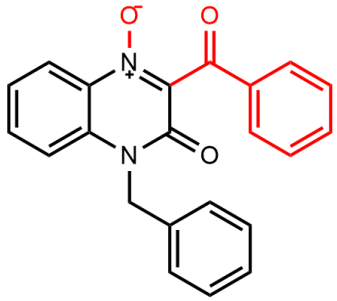
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
295.1082								
	1	C 17 H 15 N 2 O 3	295.1077	-1.5	11.5	ok	even	4.07
	2	C 13 H 11 N 8 O	295.1050	-10.6	12.5	ok	even	10.23
	3	C 22 H 15 O	295.1117	12.1	15.5	ok	even	31.06

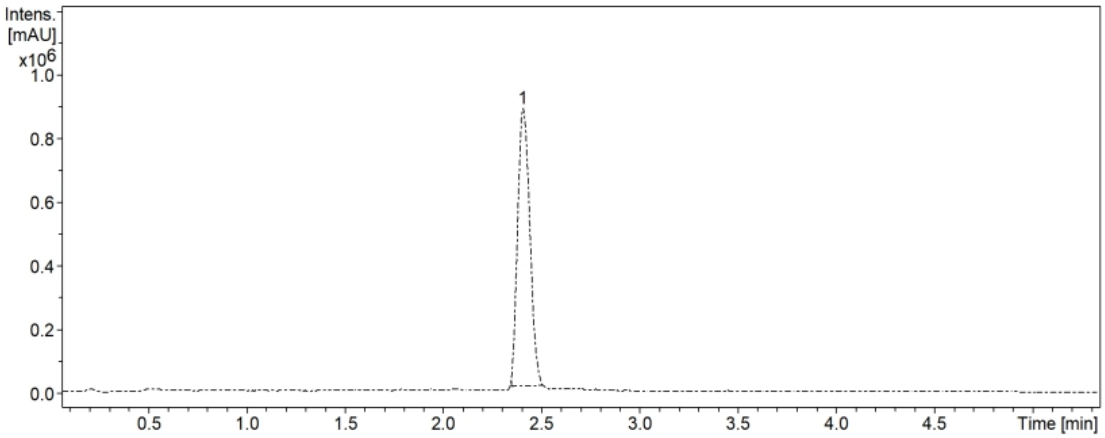
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ca





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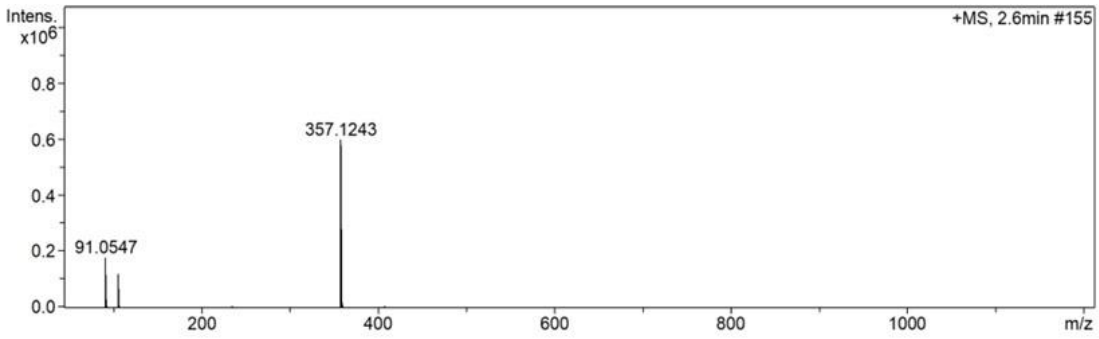


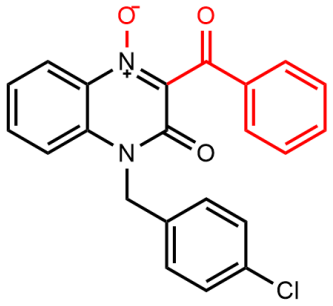
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
357.1243								
	1	C 18 H 13 N 8 O	357.1207	-10.1	16.5	ok	even	8.19
	2	C 22 H 17 N 2 O 3	357.1234	-2.6	15.5	ok	even	8.92
	3	C 27 H 17 O	357.1274	8.7	19.5	ok	even	31.10

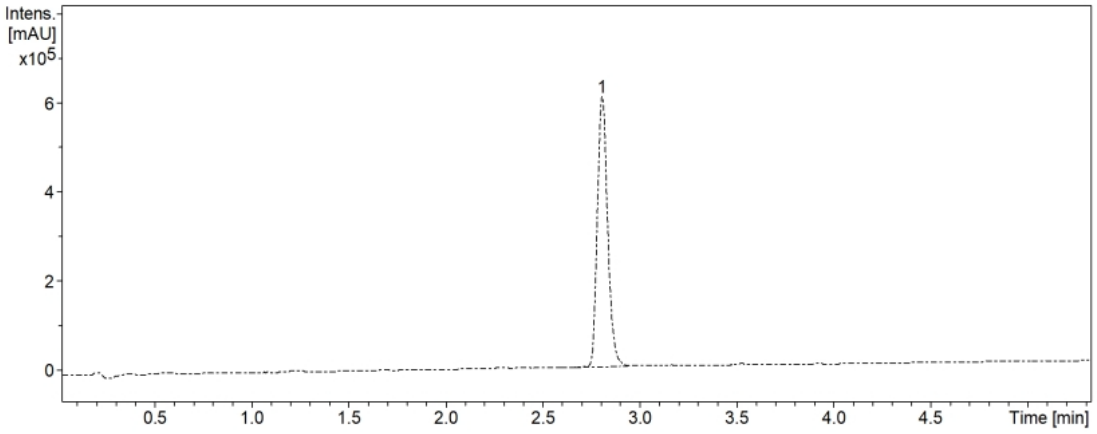
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3da





#	RT [min]	Area	Area Frac. %
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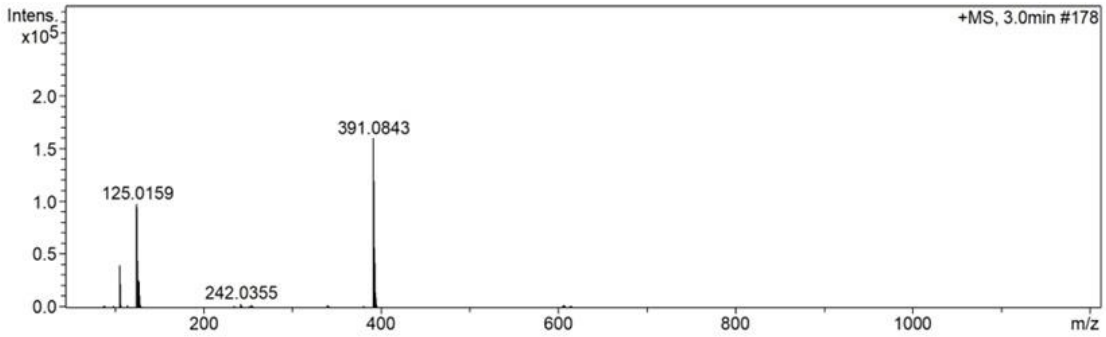


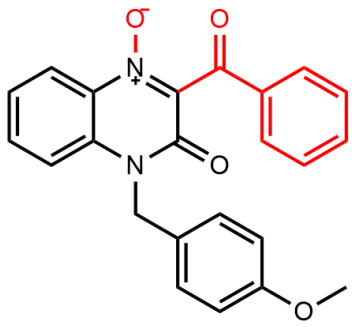
Meas. m/z 391.0843	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
	1	C 22 H 16 Cl N 2 O 3	391.0844	0.3	15.5	ok	even	4.30
	2	C 18 H 12 Cl N 8 O	391.0817	-6.6	16.5	ok	even	7.55
	3	C 17 H 16 Cl N 4 O 5	391.0804	-10.0	11.5	ok	even	17.75
	4	C 28 H 11 N 2 O	391.0866	5.9	24.5	ok	even	161.94
	5	C 15 H 19 O 12	391.0871	7.2	6.5	ok	even	165.71
	6	C 22 H 15 O 7	391.0812	-7.8	15.5	ok	even	180.18
	7	C 23 H 11 N 4 O 3	391.0826	-4.4	20.5	ok	even	181.99

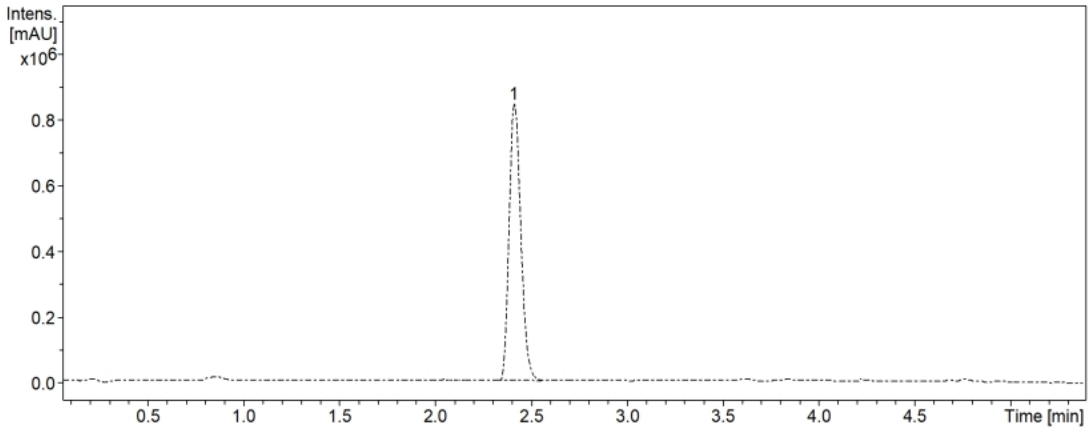
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ea





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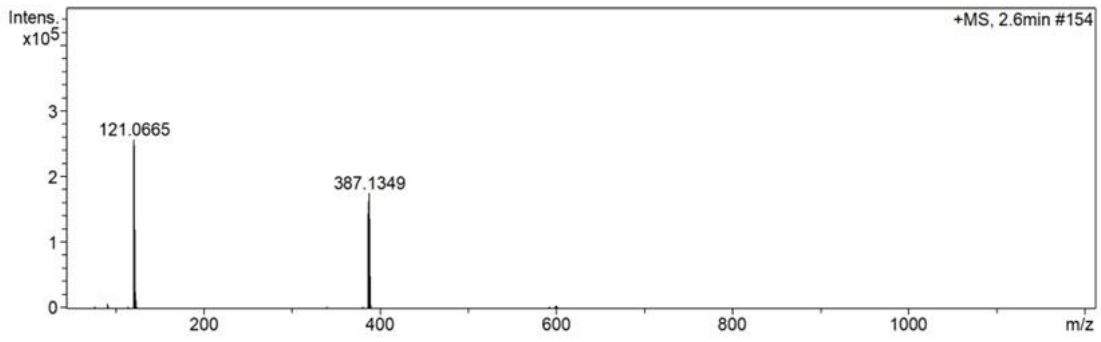


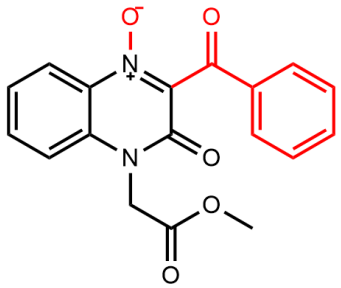
Meas. m/z 387.1349	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
	1	C 23 H 19 N 2 O 4	387.1339	-2.6	15.5	ok	even	6.56
	2	C 19 H 15 N 8 O 2	387.1312	-9.5	16.5	ok	even	8.06
	3	C 24 H 15 N 6	387.1353	0.9	20.5	ok	even	21.36
	4	C 28 H 19 O 2	387.1380	7.8	19.5	ok	even	29.31

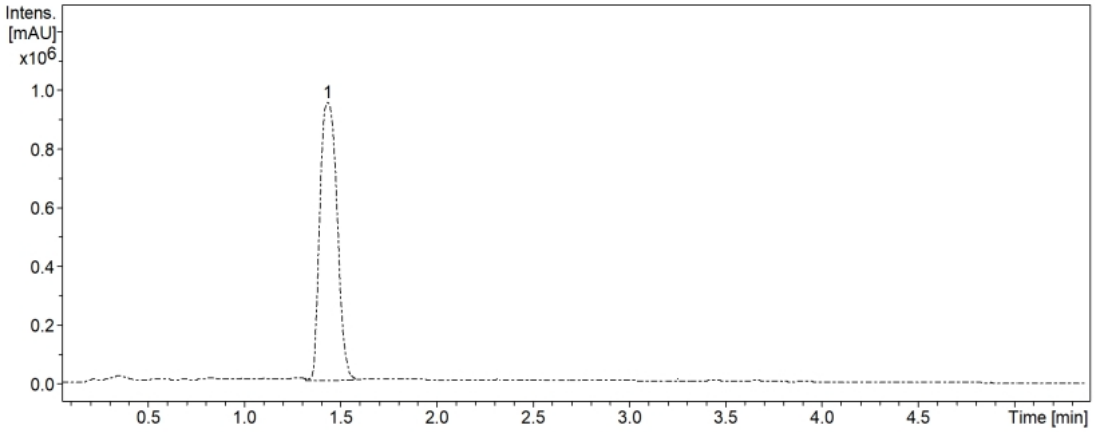
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3fa





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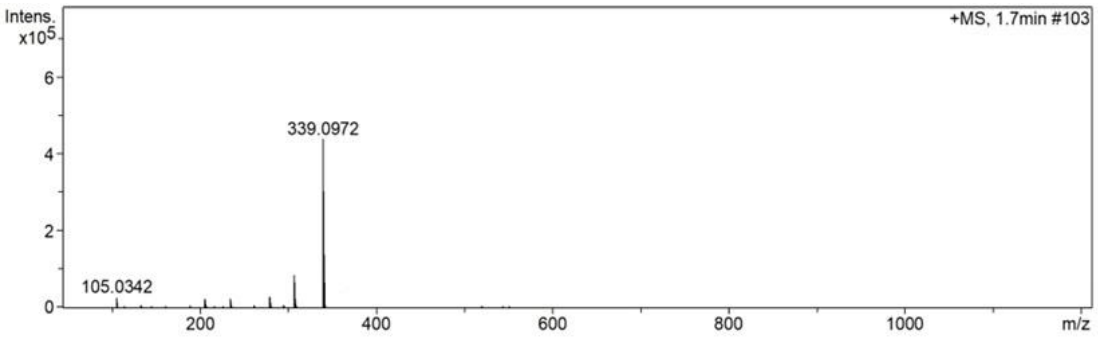


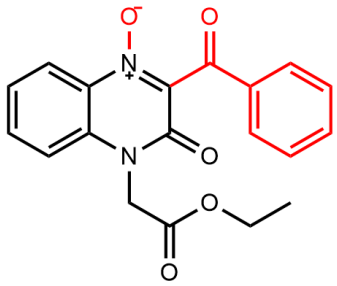
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
339.0972								
	1	C 19 H 11 N 6 O	339.0989	5.1	17.5	ok	even	24.60
	2	C 18 H 15 N 2 O 5	339.0975	1.1	12.5	ok	even	38.25

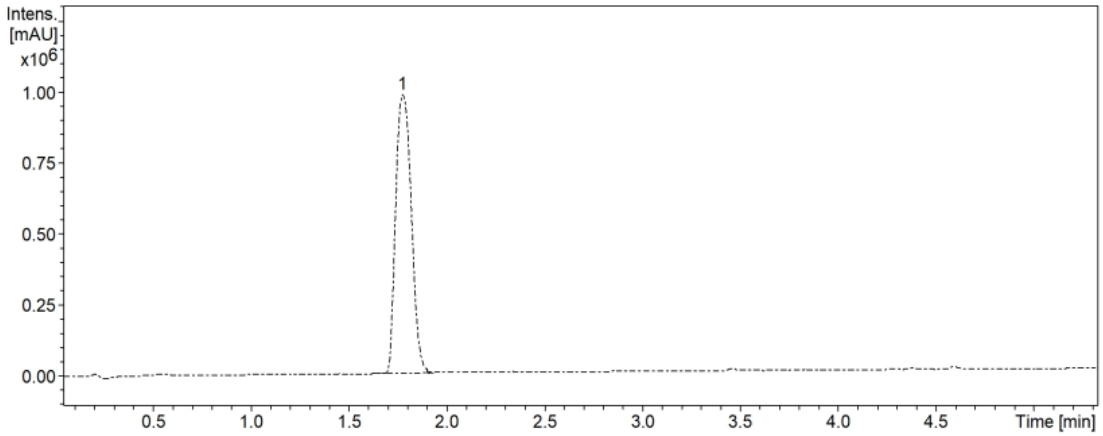
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ga





#	RT [min]	Area	Area Frac. %
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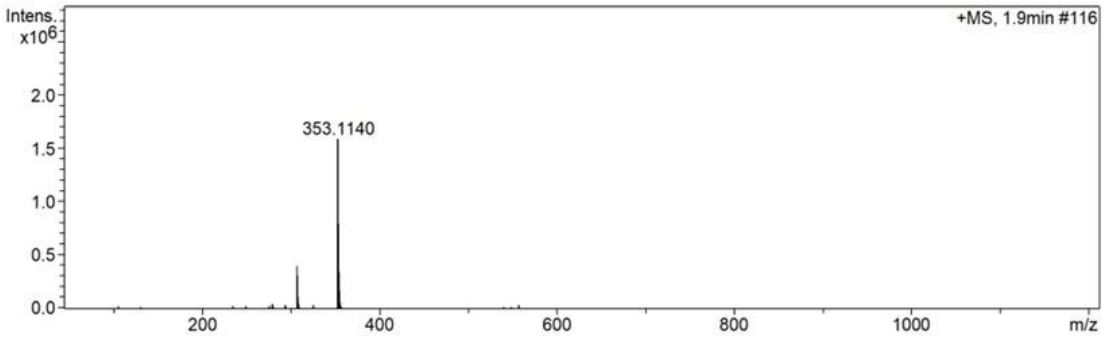


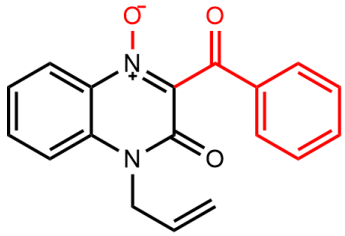
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
353.1140								
	1	C 19 H 17 N 2 O 5	353.1132	-2.1	12.5	ok	even	5.73
	2	C 20 H 13 N 6 O	353.1145	1.6	17.5	ok	even	13.13
	3	C 15 H 13 N 8 O 3	353.1105	-9.7	13.5	ok	even	18.43
	4	C 24 H 17 O 3	353.1172	9.3	16.5	ok	even	23.23

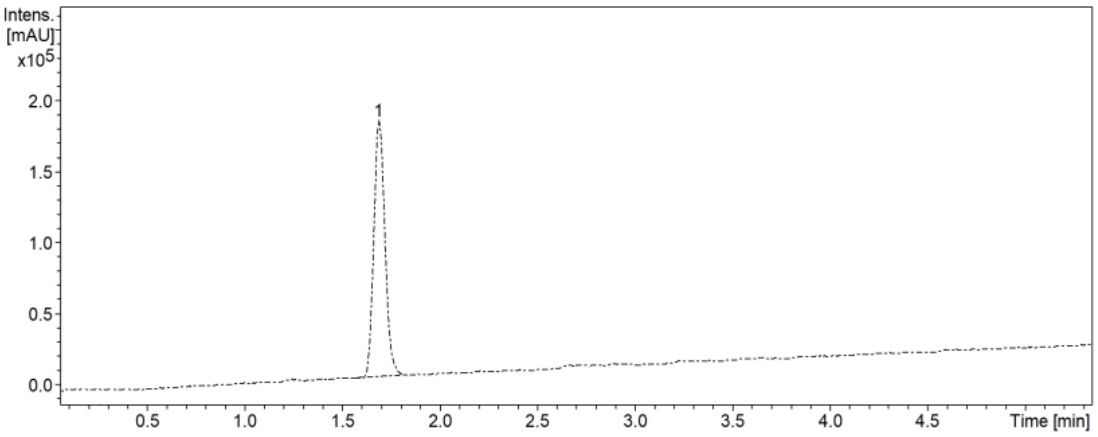
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ha





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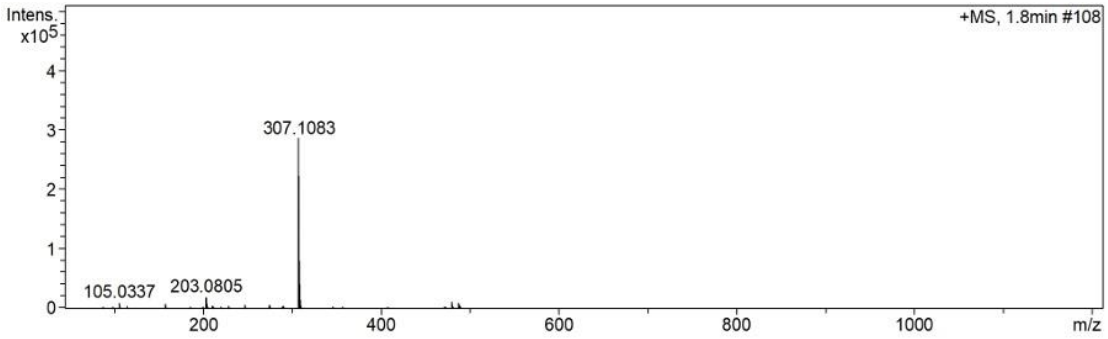


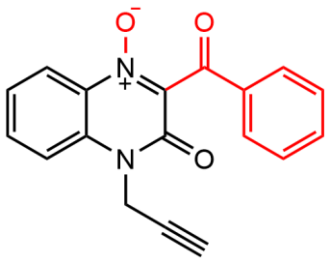
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
307.1083								
	1	C 14 H 11 N 8 O	307.1050	-10.7	13.5	ok	even	6.49
	2	C 18 H 15 N 2 O 3	307.1077	-1.9	12.5	ok	even	7.76
	3	C 23 H 15 O	307.1117	11.2	16.5	ok	even	34.80

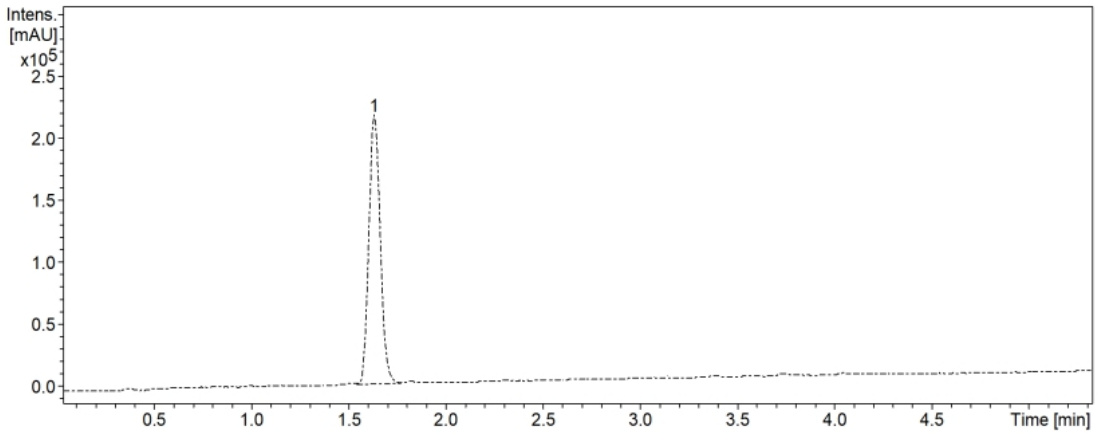
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm

3ia





#	RT [min]	Area	Area Frac. %
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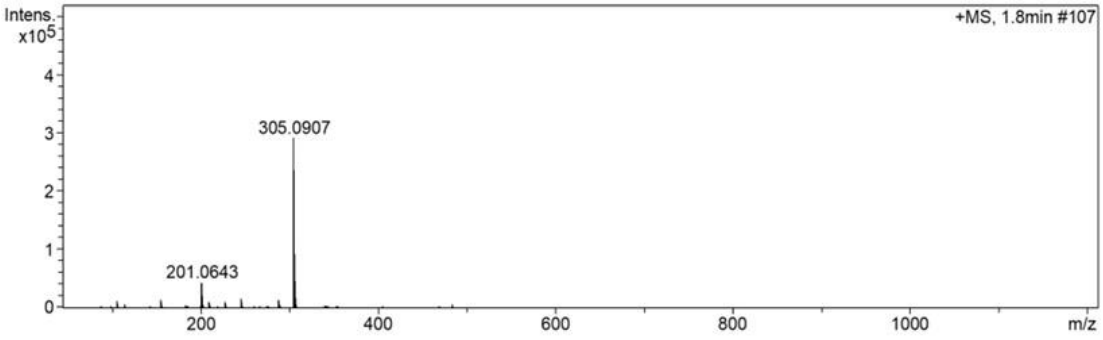


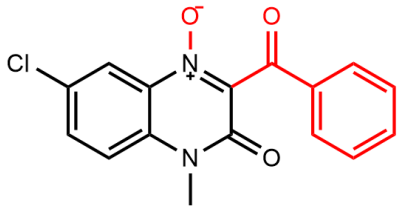
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
305.0907								
	1	C 18 H 13 N 2 O 3	305.0921	4.5	13.5	ok	even	4.01
	2	C 14 H 9 N 8 O	305.0894	-4.3	14.5	ok	even	9.81
	3	C 13 H 13 N 4 O 5	305.0880	-8.7	9.5	ok	even	23.06
	4	C 12 H 17 O 9	305.0867	-13.1	4.5	ok	even	37.79

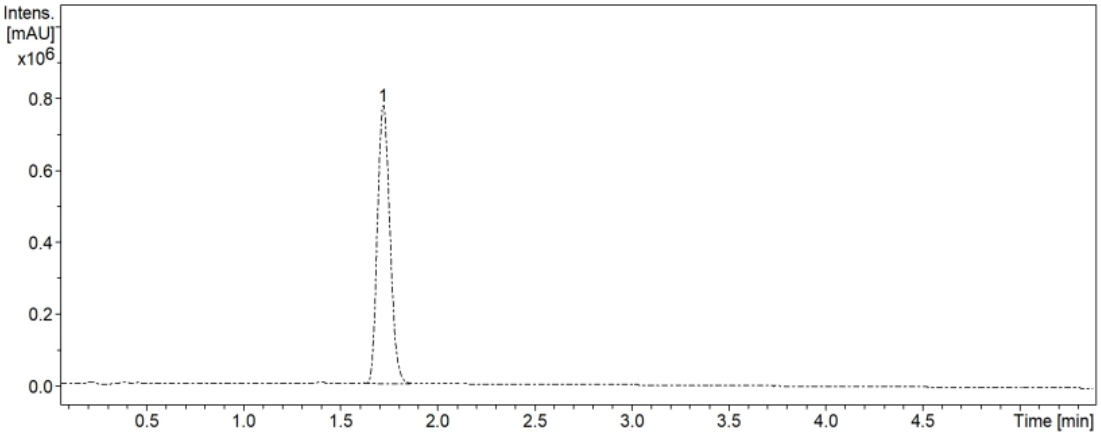
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

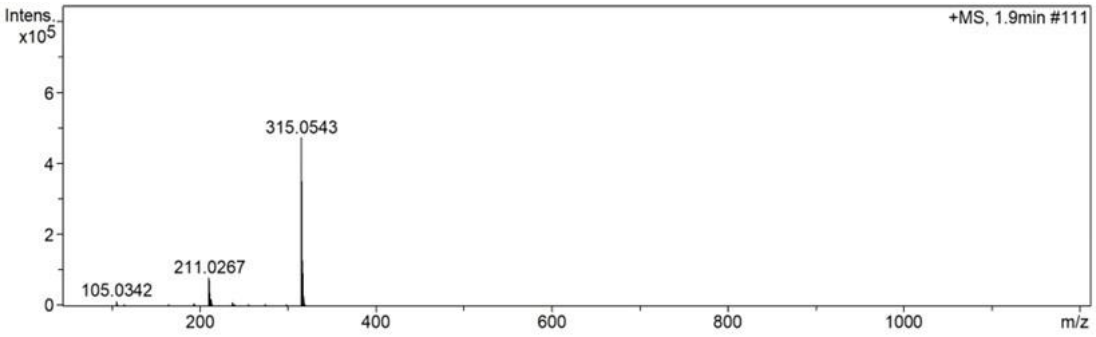
Flow rate: 0.7ml/min **Detection**: UV254nm

3ja





#	RT [min]	Area	Area Frac. %
1	1.7	3469028	100.0



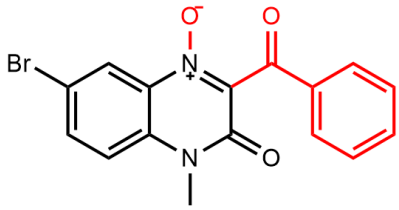
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
315.0543								
	1	C 12 H 8 Cl N 8 O	315.0504	-12.5	12.5	ok	even	30.70
	2	C 16 H 12 Cl N 2 O 3	315.0531	-3.9	11.5	ok	even	34.81
	3	C 21 H 12 Cl O	315.0571	8.8	15.5	ok	even	49.30
	4	C 9 H 15 O 12	315.0558	4.7	2.5	ok	even	141.96
	5	C 10 H 11 N 4 O 8	315.0571	8.9	7.5	ok	even	143.70
	6	C 17 H 7 N 4 O 3	315.0513	-9.7	16.5	ok	even	145.25
	7	C 22 H 7 N 2 O	315.0553	3.0	20.5	ok	even	150.93

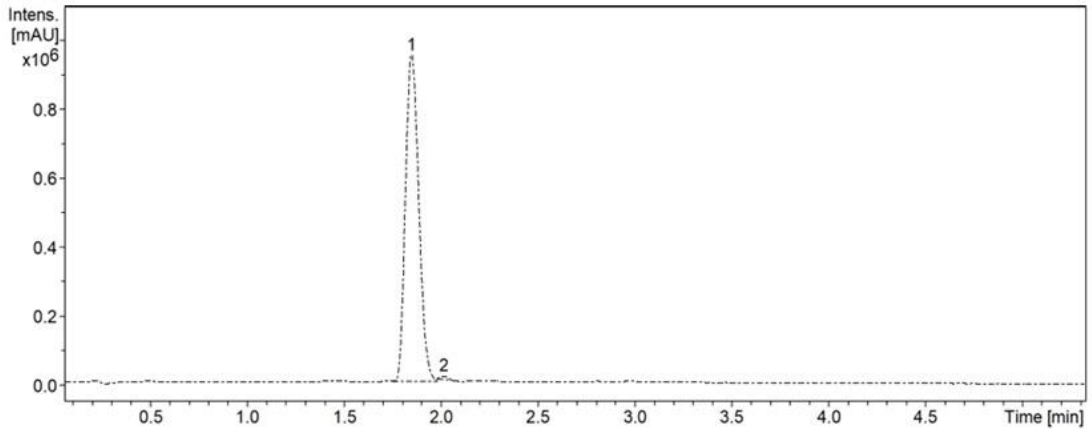
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

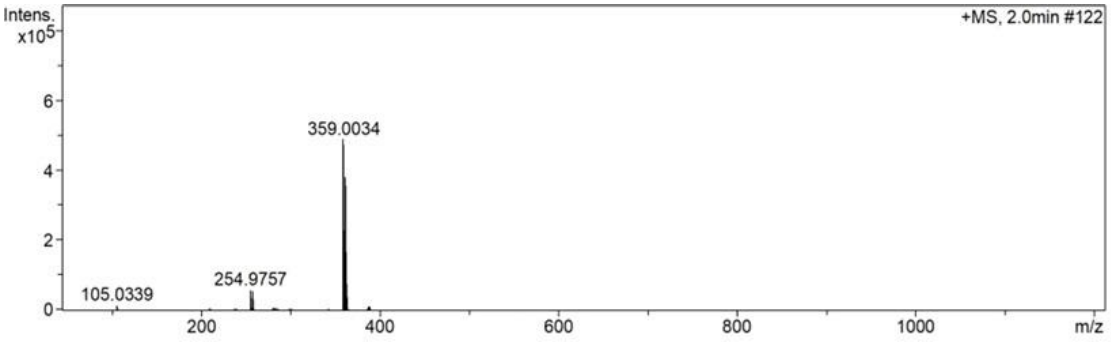
Flow rate: 0.7ml/min **Detection**: UV254nm

3ka





#	RT [min]	Area	Area Frac. %
1	1.8	4552110	99.4
2	2.0	26249	0.6



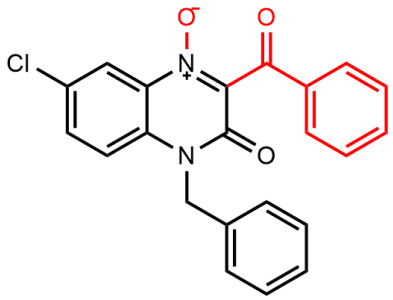
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
359.0034								
	1	C 12 H 8 Br N 8 O	358.9999	-9.8	12.5	ok	even	93.36
	2	C 16 H 12 Br N 2 O 3	359.0026	-2.3	11.5	ok	even	97.65
	3	C 21 H 12 Br O	359.0066	8.9	15.5	ok	even	104.95
	4	C 16 H 7 O 10	359.0034	-0.1	13.5	ok	even	379.37
	5	C 17 H 3 N 4 O 6	359.0047	3.6	18.5	ok	even	430.70
	6	C 12 H 3 N 6 O 8	359.0007	-7.6	14.5	ok	even	432.78

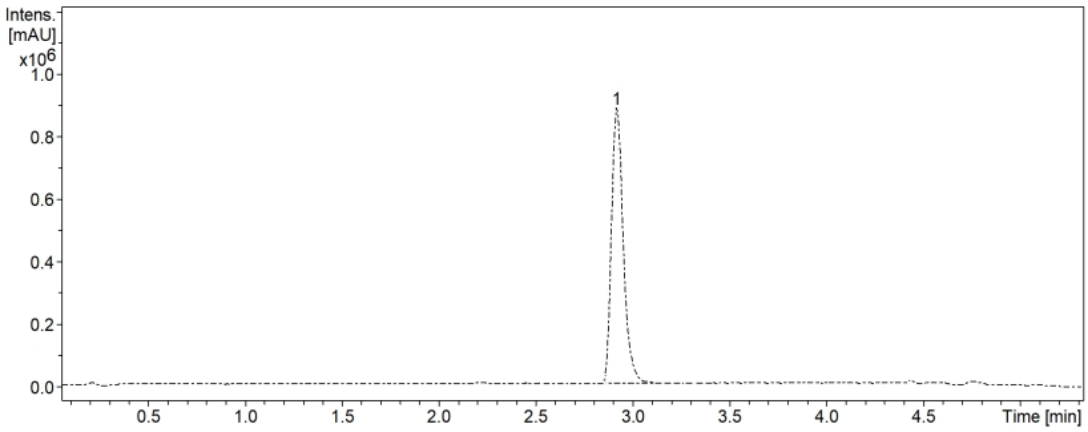
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

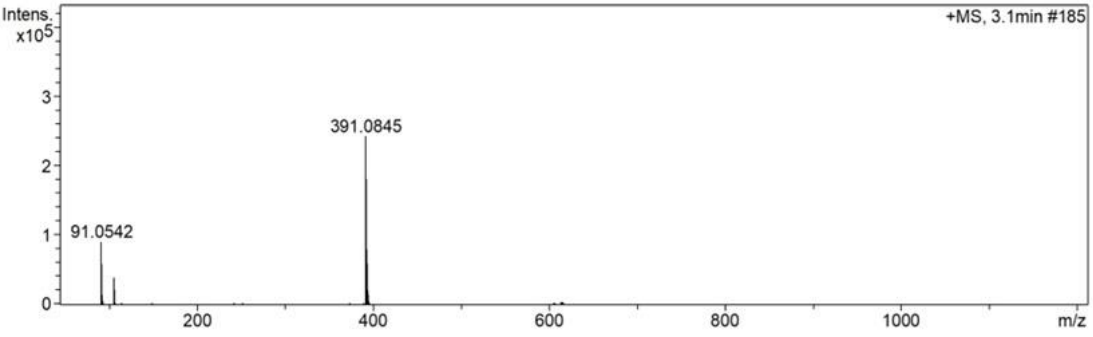
Flow rate: 0.7ml/min **Detection**: UV254nm

3la





#	RT [min]	Area	Area Frac. %
1	2.9	3607590	100.0



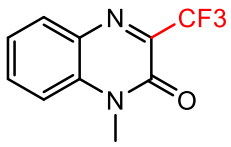
Meas. m/z	#	Formula	m/z	err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
391.0845								
	1	C 18 H 12 Cl N 8 O	391.0817	-7.2	16.5	ok	even	13.59
	2	C 22 H 16 Cl N 2 O 3	391.0844	-0.3	15.5	ok	even	14.08
	3	C 27 H 16 Cl O	391.0884	10.0	19.5	ok	even	28.06
	4	C 28 H 11 N 2 O	391.0866	5.3	24.5	ok	even	147.55
	5	C 22 H 15 O 7	391.0812	-8.4	15.5	ok	even	162.76
	6	C 23 H 11 N 4 O 3	391.0826	-5.0	20.5	ok	even	164.25
	7	C 16 H 15 N 4 O 8	391.0884	10.0	11.5	ok	even	170.65

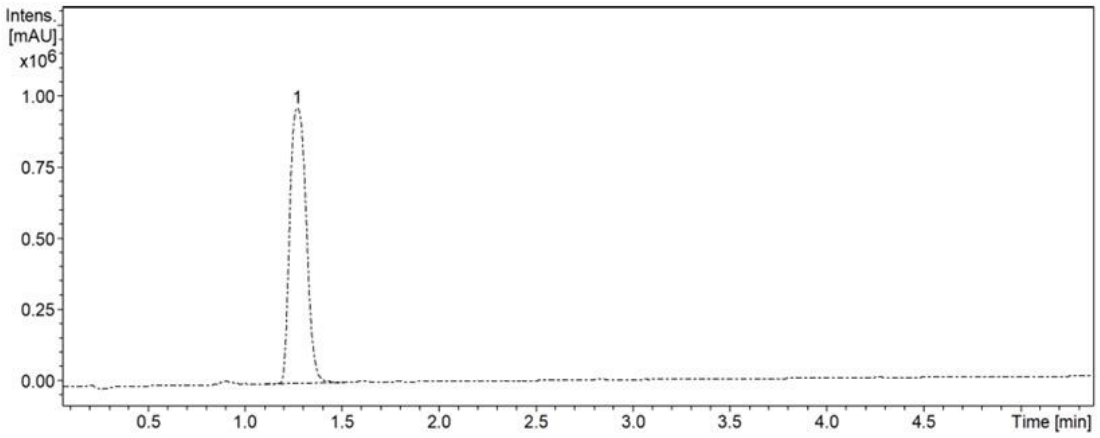
HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

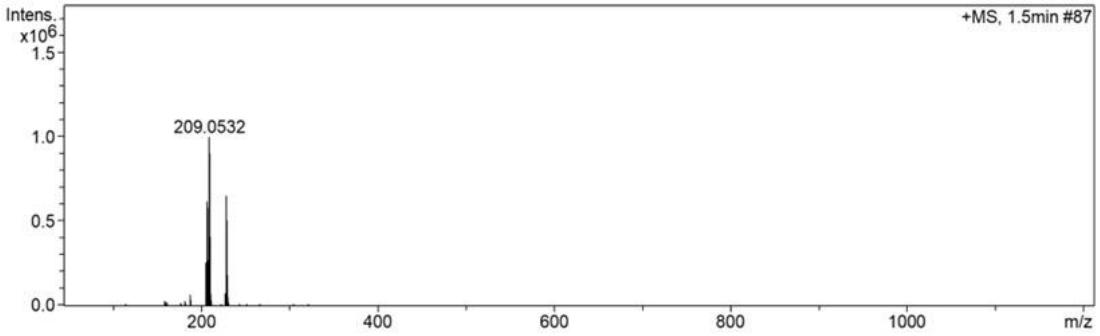
Flow rate: 0.7ml/min **Detection**: UV254nm

12





#	RT [min]	Area	Area Frac. %
1	1.3	5715645	100.0



Meas. m/z 229.0593

#	Formula	m/z	err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma
1	C 9 H 10 F N 2 O 4	229.0619	11.2	5.5	ok	even	3.24
2	C 7 H 9 N 4 O 5	229.0567	-11.3	5.5	ok	even	6.87
3	C 10 H 8 F 3 N 2 O	229.0583	-4.4	6.5	ok	even	7.94
4	C 8 H 5 N 8 O	229.0581	-5.5	10.5	ok	even	9.44
5	C 5 H 6 F N 8 O 2	229.0592	-0.5	6.5	ok	even	10.69
6	C 10 H 6 F N 6	229.0632	17.1	10.5	ok	even	16.37
7	C 6 H 11 F 2 N 2 O 5	229.0631	16.2	1.5	ok	even	17.20
8	C 12 H 9 N 2 O 3	229.0608	6.2	9.5	ok	even	20.68
9	C 6 H 13 O 9	229.0554	-17.2	0.5	ok	even	22.20
10	C 4 H 10 F N 4 O 6	229.0579	-6.3	1.5	ok	even	25.51
11	C 13 H 7 F 2 N 2	229.0572	-9.4	10.5	ok	even	26.60

HPLC Condition: **Column**: Agilent Zorbax SB C18, 2.1*50 mm, 1.8 um

Eluent: ACN/H_2O, A % 0-5-7 min, 30-70-70 %

Flow rate: 0.7ml/min **Detection**: UV254nm