

Supplementary Material

Electrochemical synthesis of 4-quinazolinone derivatives mediated by acetic acid

Tanay Ghoshal*

Shri MM Patel Institute of Sciences and Research, Department of Chemistry, Kadi Sarva Vishwavidyalaya, Gandhinagar-382024, Gujarat, India

Email: ghoshal.tanay@gmail.com

Table of Contents

Electrochemical reaction set up	S2
Analytical data of compounds 3a-3q,4,5	S3

1. Electrochemical Reactor set up

The electrochemical reactor was set up starting with a 5v mobile charger and a multiple USB port. A mobile phone charger was attached to a multiple USB port to divert the current to different reaction vessels. The wire from USB port was attached to electrodes through alligator clips, the electrodes were mainly carbon (0.5 mm diameter) and wire of aluminium (0.5 mm diameter) (Figure 1). Constant electric current of 0.35A cm⁻² was passed through the reaction mixture for required time at room temperature for complete conversion. This was helpful in carrying out multiple reactions at a time.



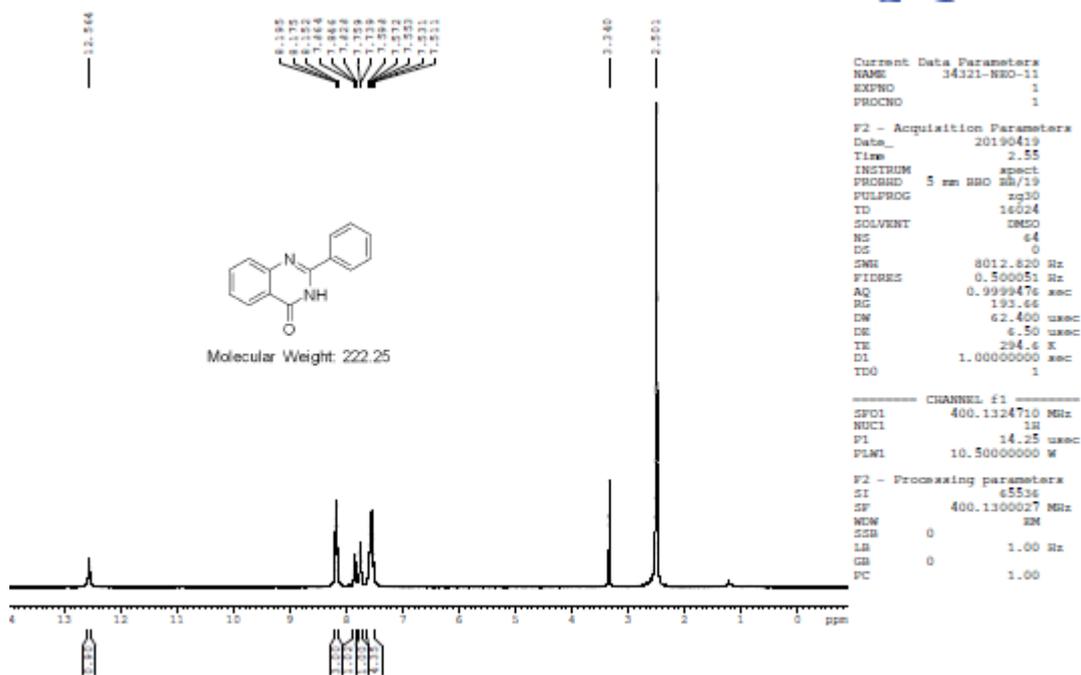
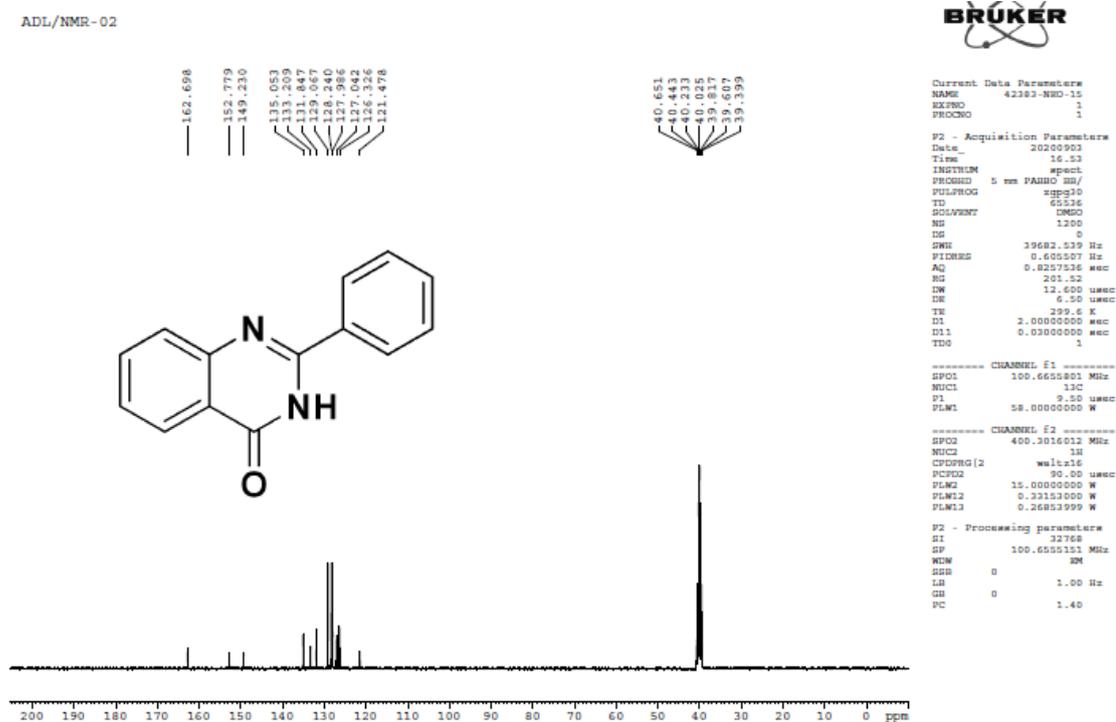
Figure 1. Multiple reaction assembly.

Scale up assembly to keep reaction in bigger scale. 5g batches were done. (Figure 2)

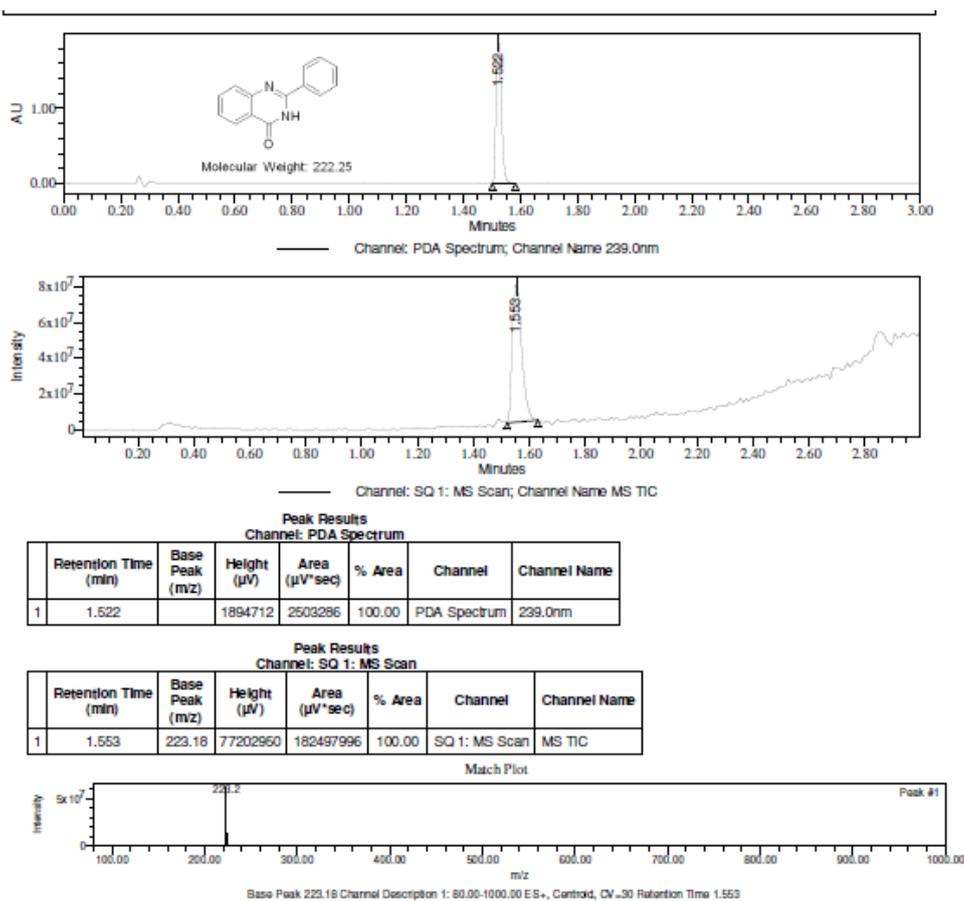
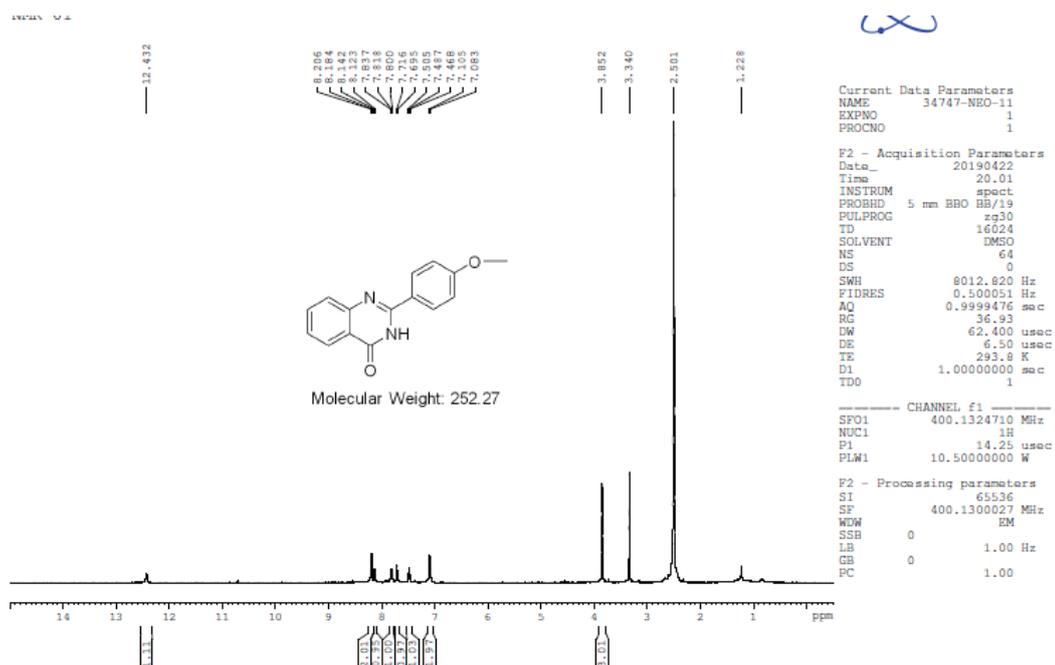


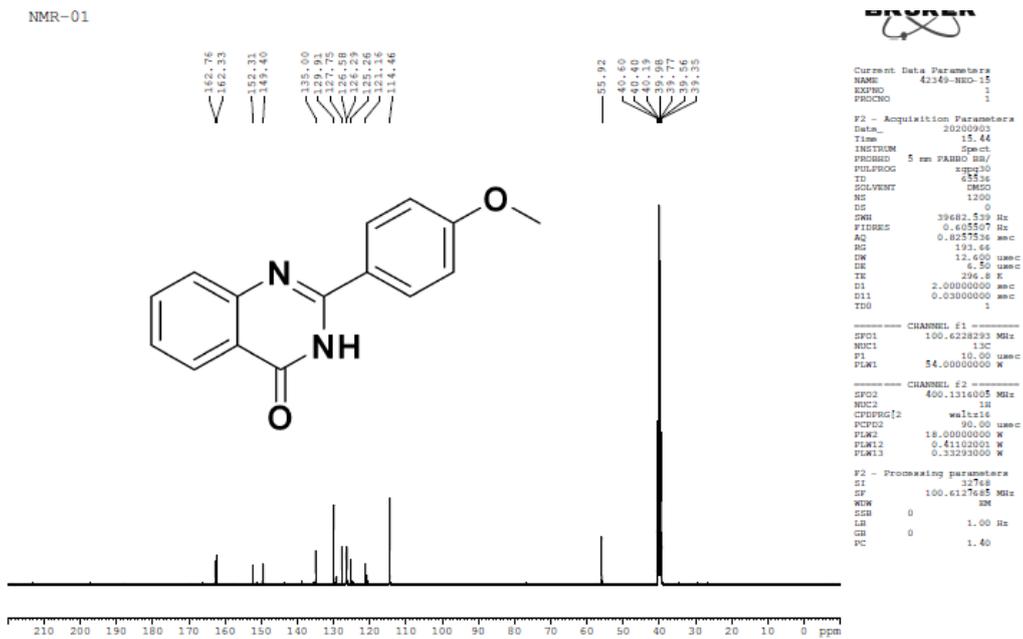
Figure 2. Electrochemical batch of a 5g reaction.

2. Analytical data of Compound 3a to 3q, 4 and 5.

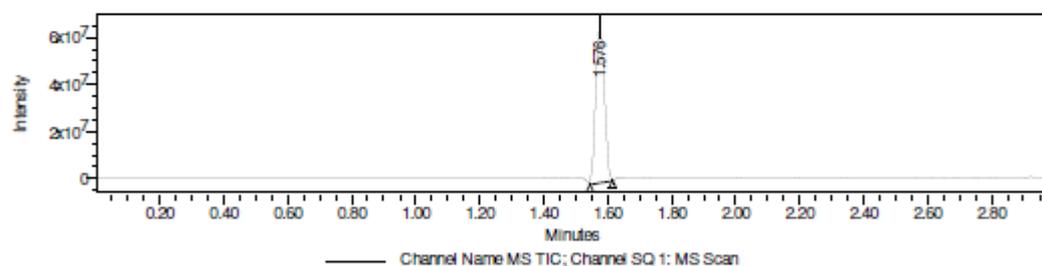
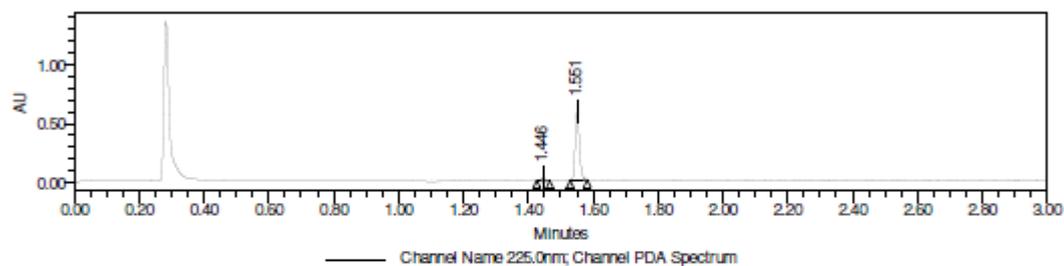
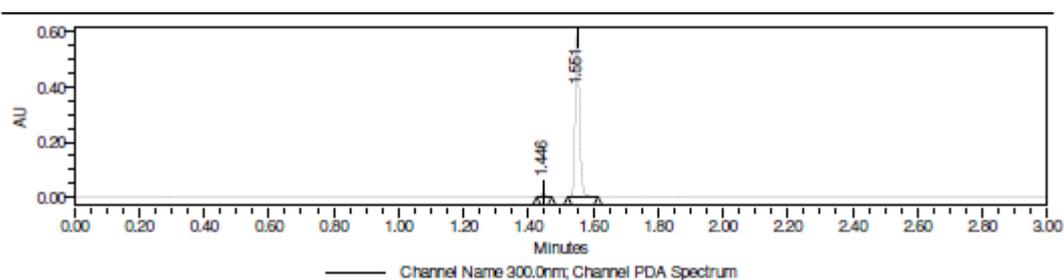
¹H NMR of Compound 3a¹³C NMR of Compound 3a

LCMS of Compound 3a

¹H NMR of Compound 3b

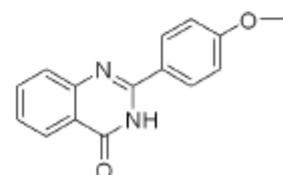
¹³C NMR of Compound 3b

LCMS of Compound 3b



Peak Results
Channel: PDA Spectrum

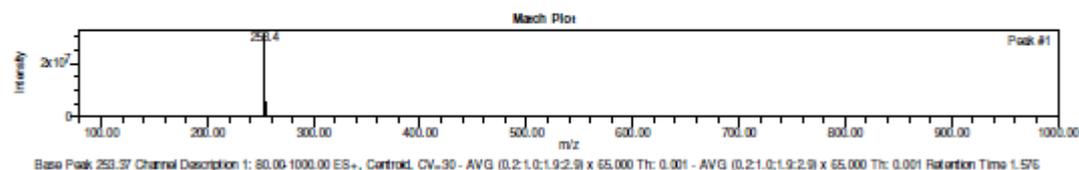
	Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	1.446	26090	26090	22652	4.18	PDA Spectrum	225.0nm
2	1.446	15356	15356	13000	2.34	PDA Spectrum	300.0nm
3	1.551	590012	590012	541821	97.66	PDA Spectrum	300.0nm
4	1.551	580879	580879	524360	95.82	PDA Spectrum	225.0nm



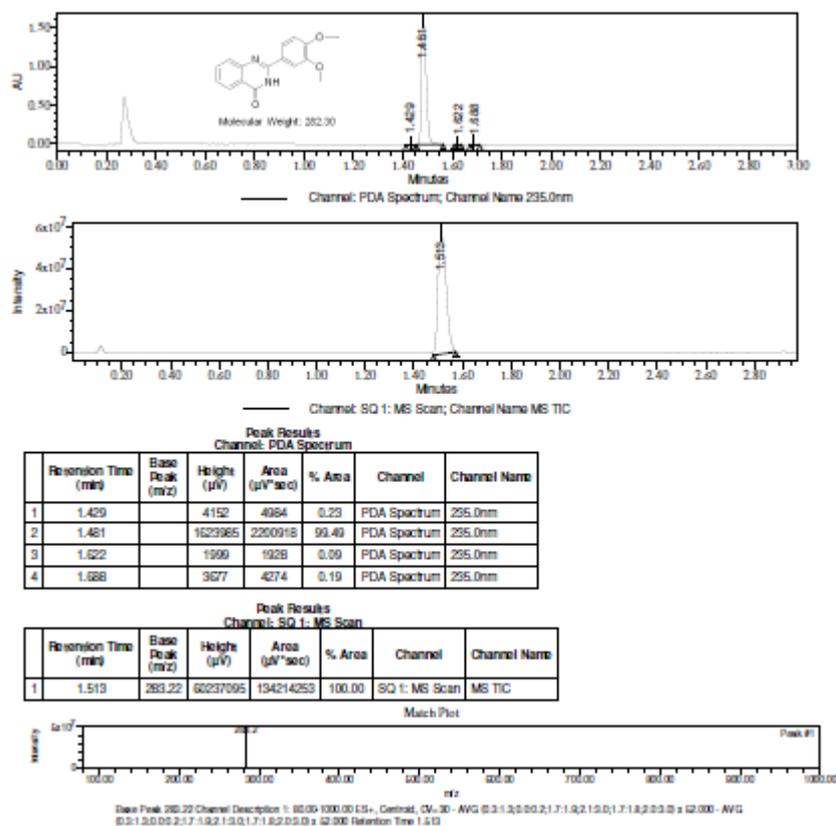
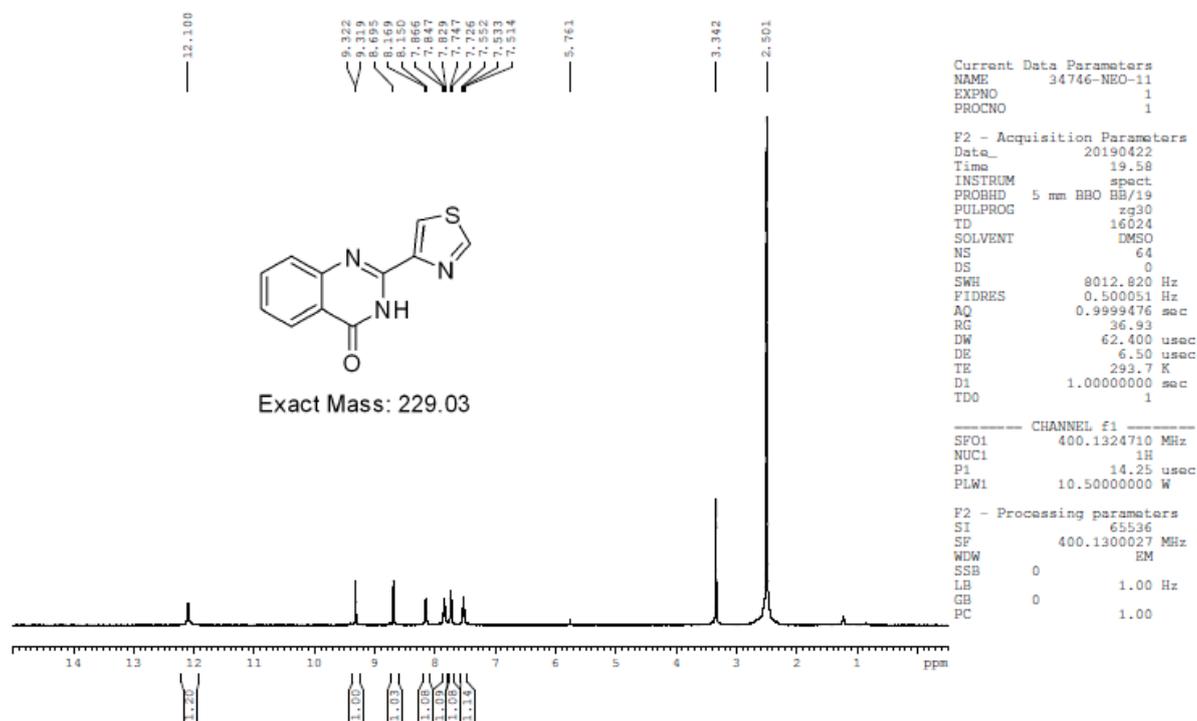
Molecular Weight: 252.27

Channel: SQ 1: MS scan

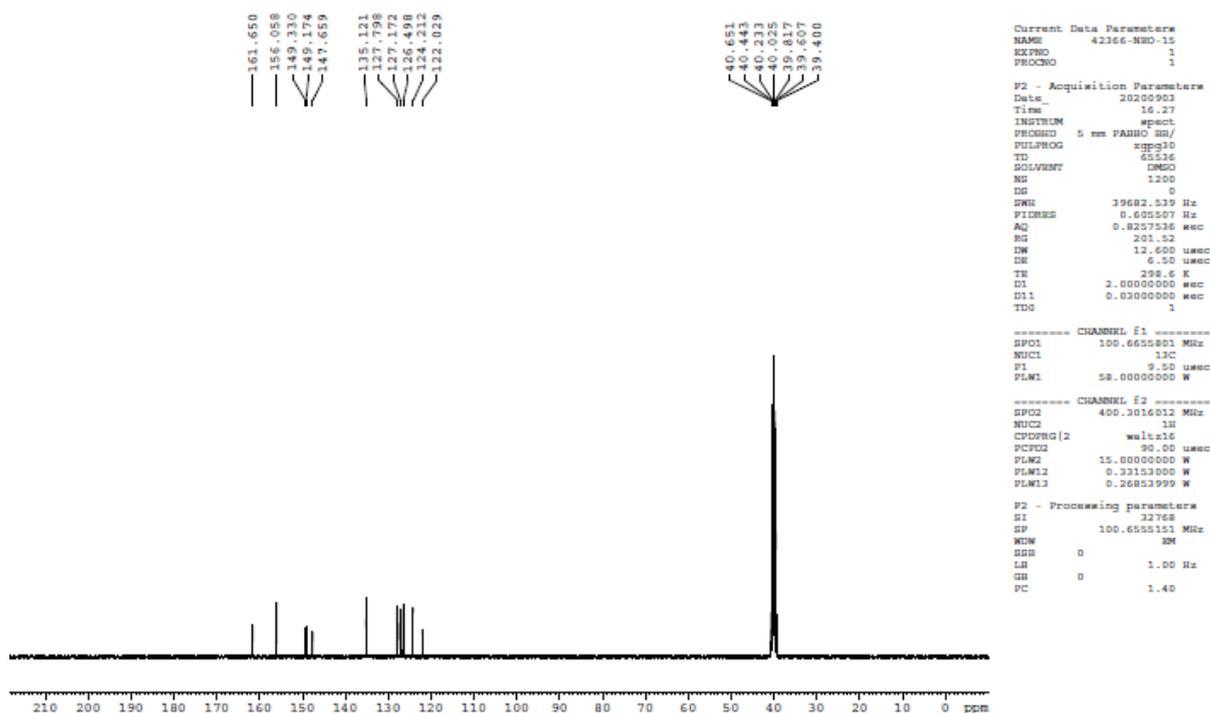
	Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	1.576	253.37	68456741	125318621	100.00	SQ 1: MS Scan	MS TIC



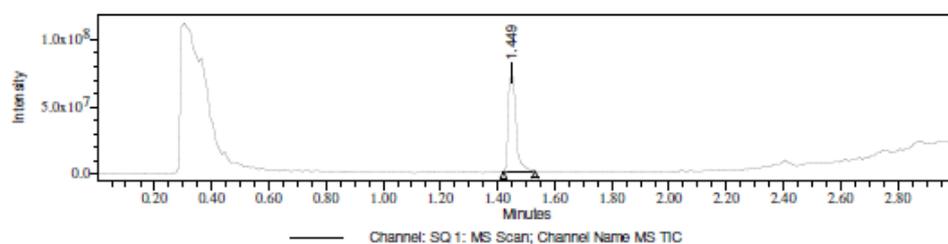
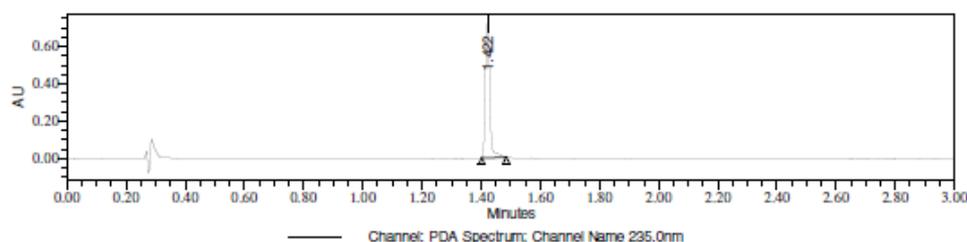
LCMS of Compound 3c

¹H NMR of Compound 3d

¹³C NMR of Compound 3d



LCMS of Compound 3d

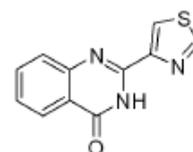


Peak Results
Channel: PDA Spectrum

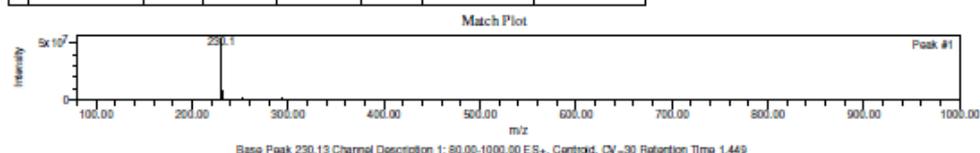
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	1.422	728003	769045	100.00	PDA Spectrum	235.0nm

Peak Results
Channel: SQ 1: MS Scan

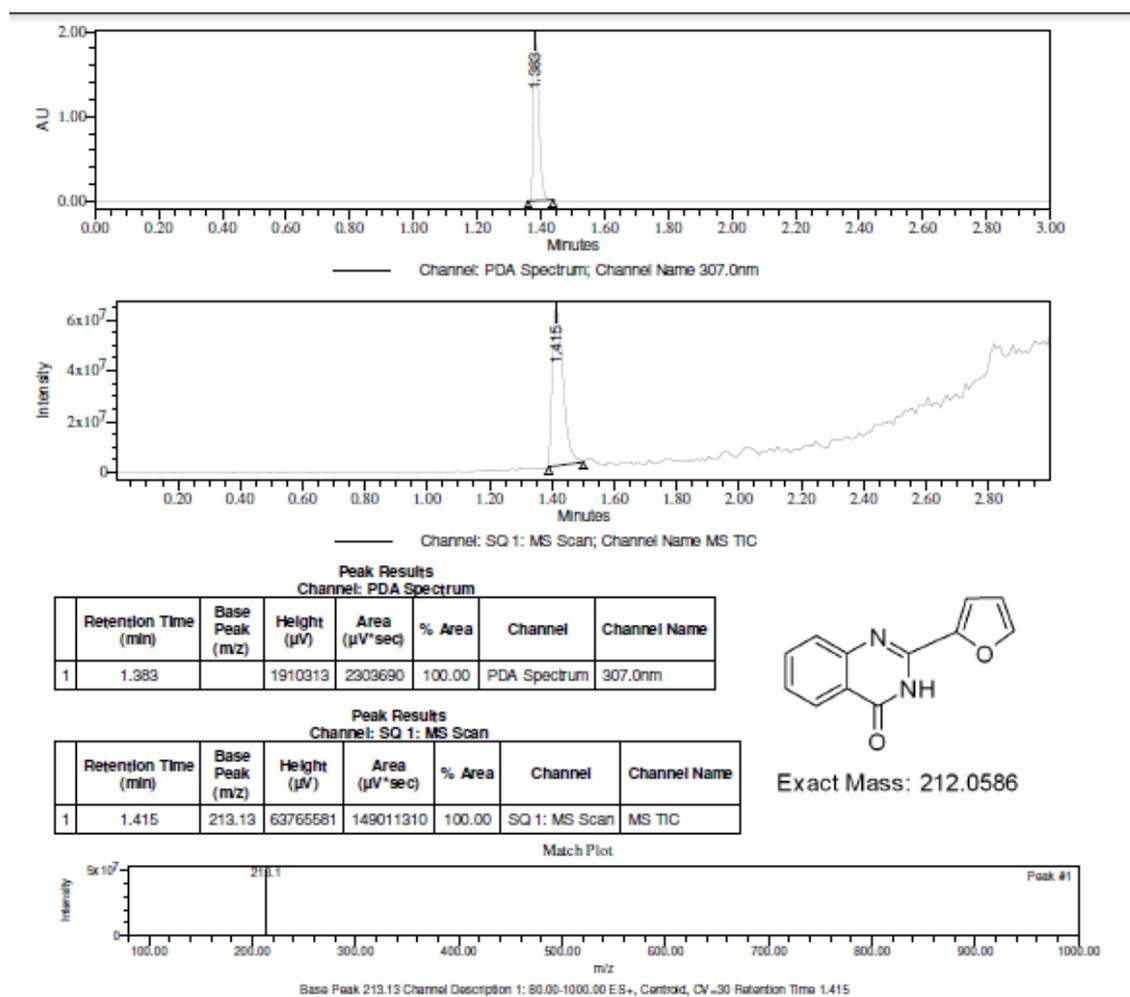
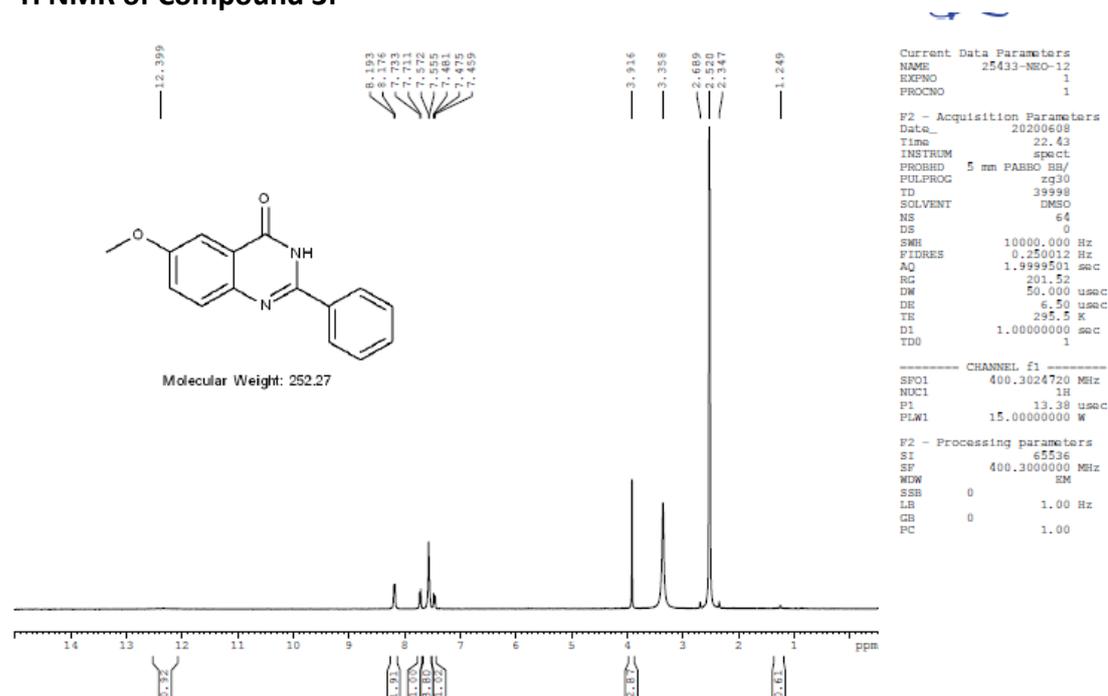
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	1.449	290.13	138396585	100.00	SQ 1: MS Scan	MS TIC



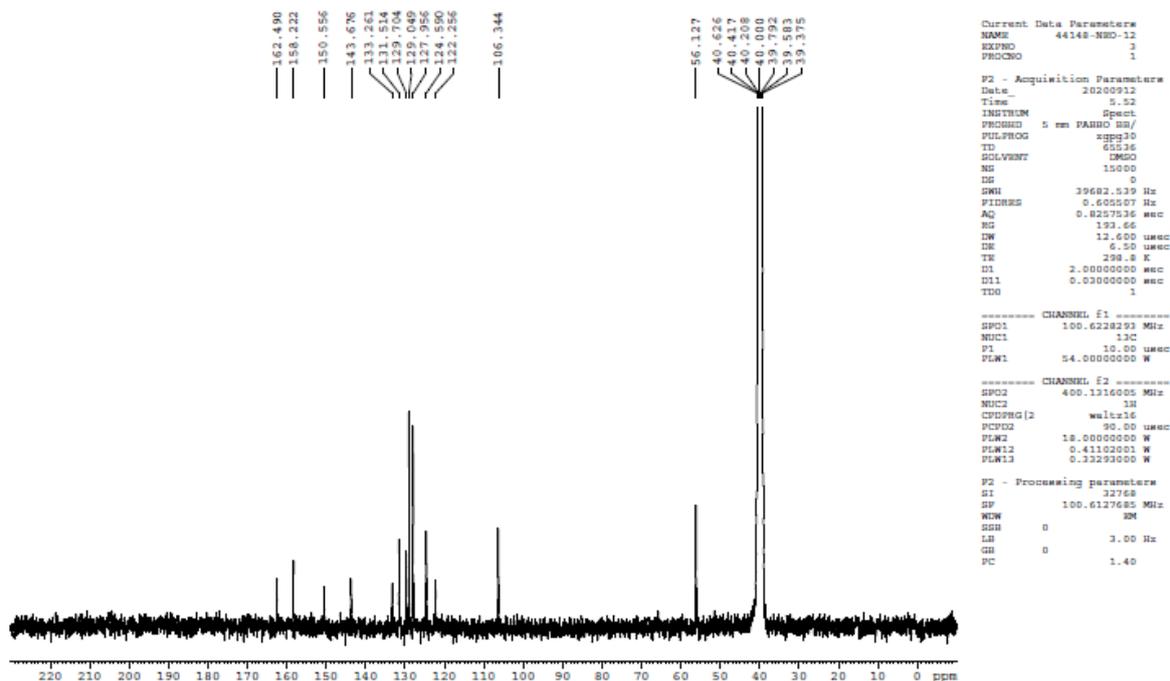
Exact Mass: 229.03



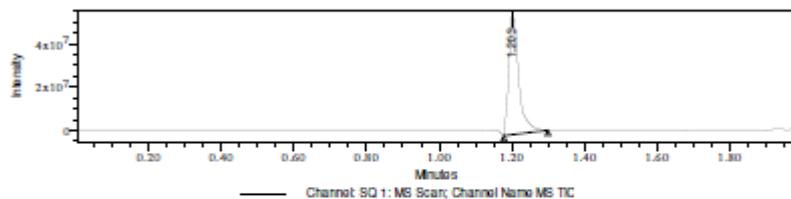
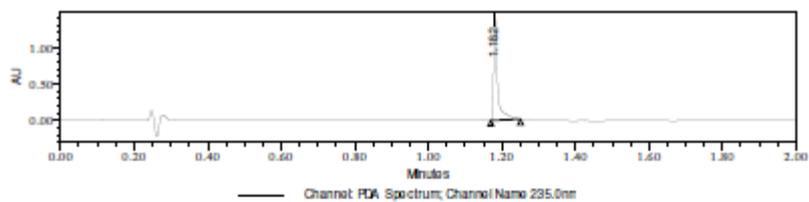
LCMS of Compound 3e

¹H NMR of Compound 3f

¹³C NMR of Compound 3f



LCMS of Compound 3f

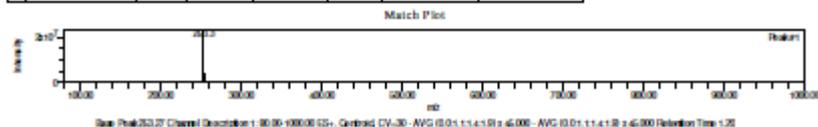
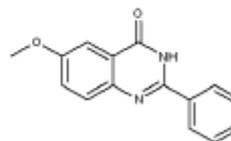


Peak Results
Channel: PDA Spectrum

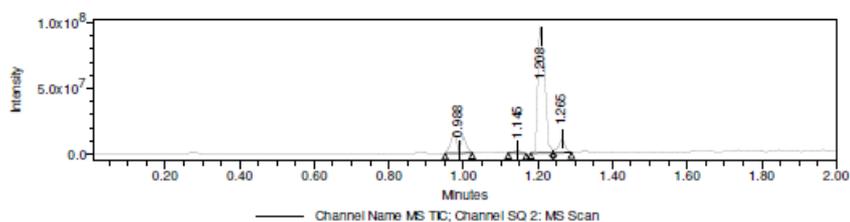
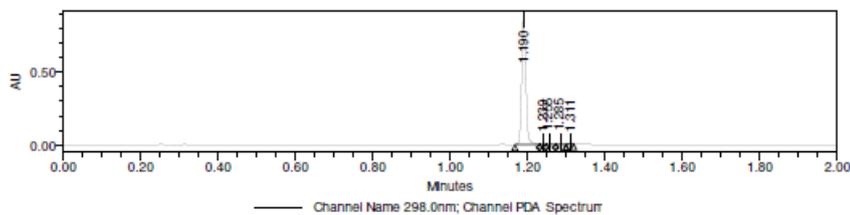
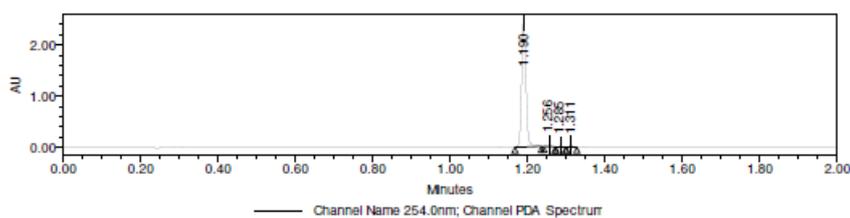
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1.182		1405045	1058090	100.00	PDA Spectrum	235.0nm

Peak Results
Channel: SQ 1: MS Scan

Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1.203	253.27	57960183	107342817	100.00	SQ 1: MS Scan	MS TIC

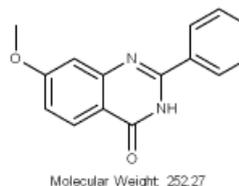


LCMS of Compound 3g



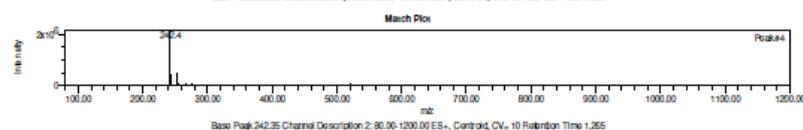
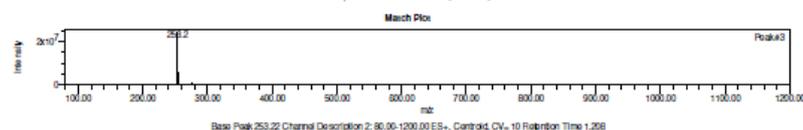
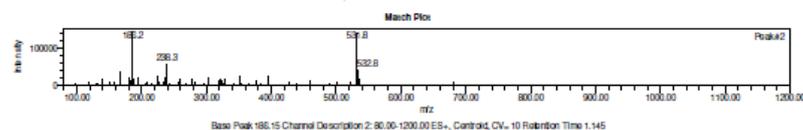
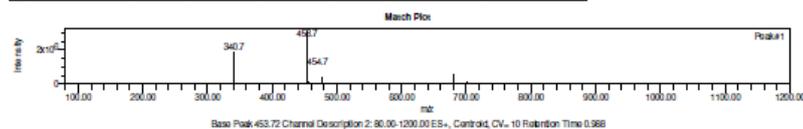
Peak Results
Channel: PDA Spectrum

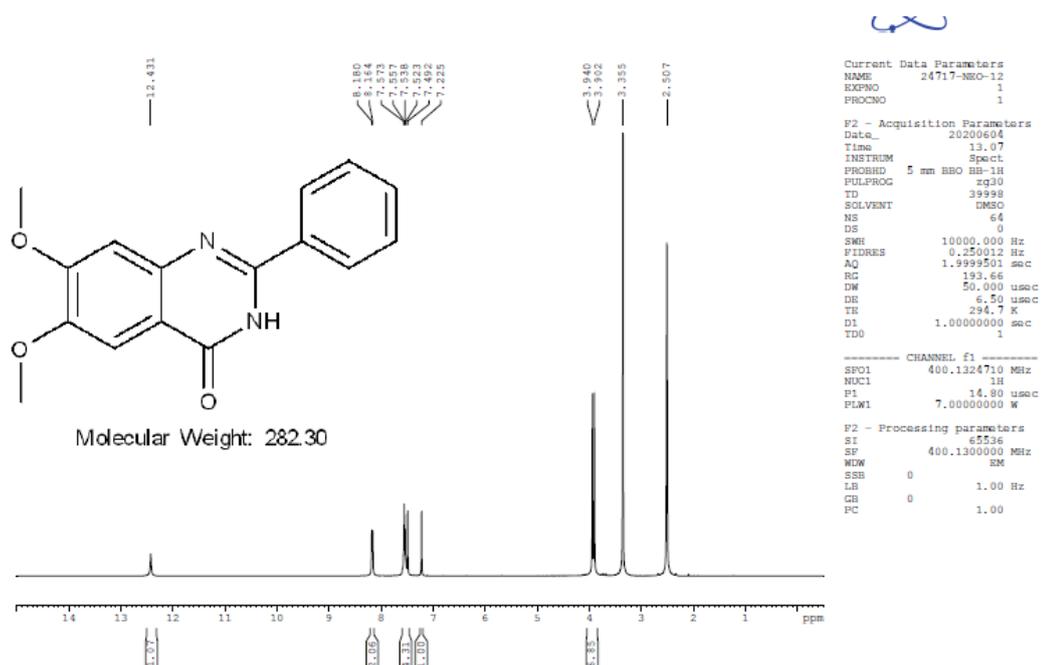
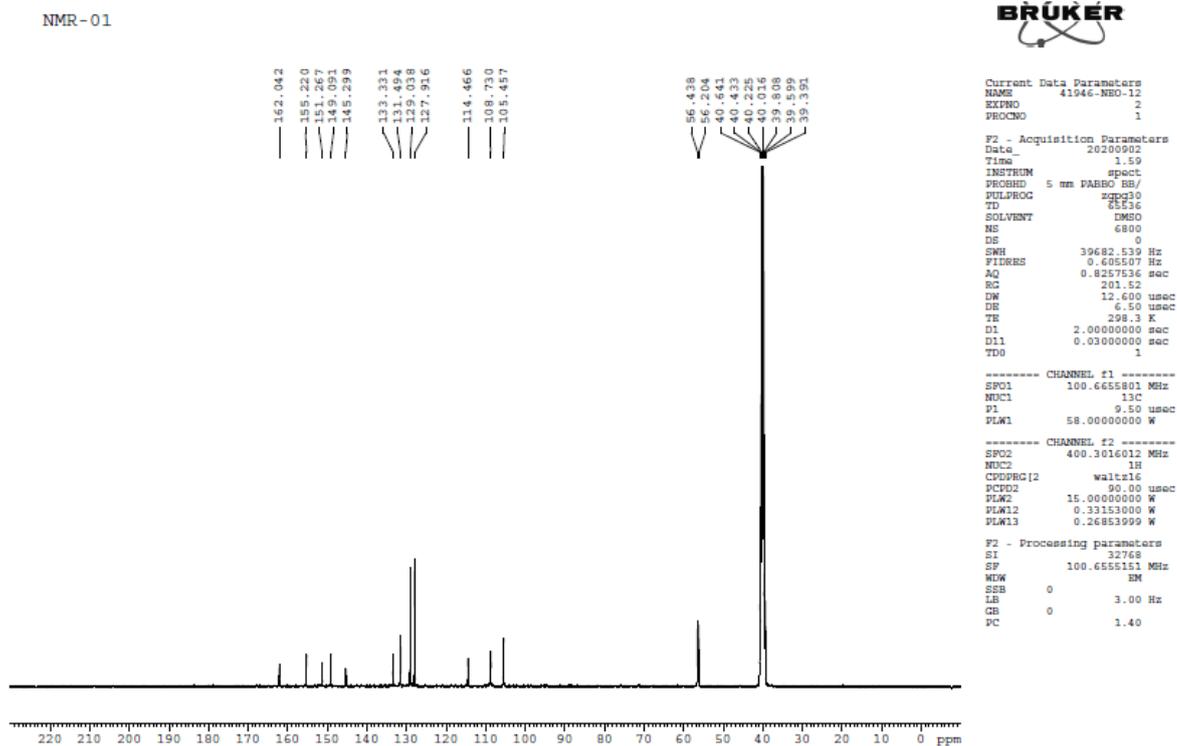
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	1.190	876820	663753	95.92	PDA Spectrum	298.0nm
2	1.190	2468664	1923434	97.17	PDA Spectrum	254.0nm
3	1.239	9118	7607	1.10	PDA Spectrum	298.0nm
4	1.255	11548	9802	1.42	PDA Spectrum	298.0nm



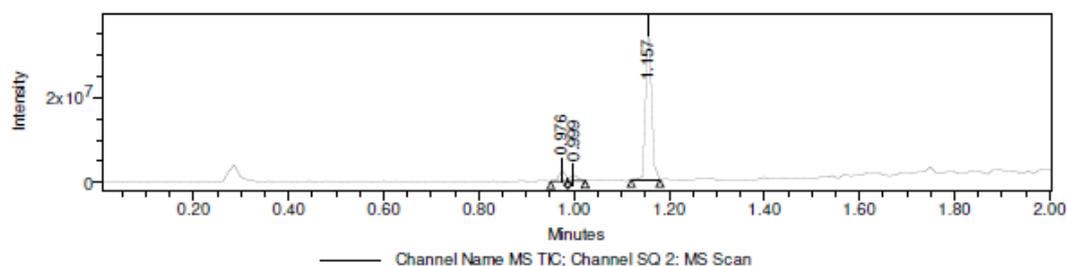
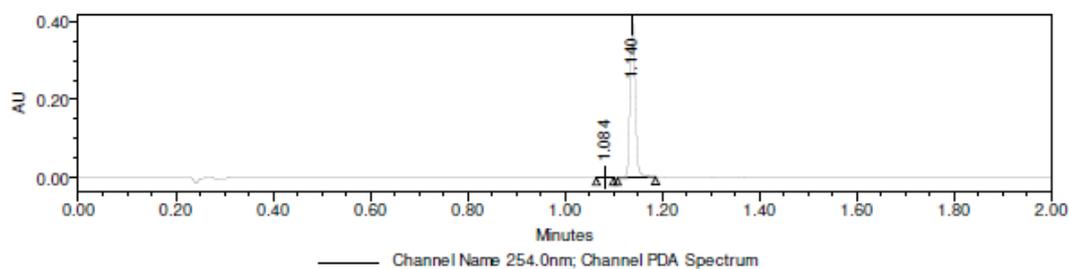
Peak Results
Channel: SQ 2: MS Scan

Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	0.988	453.72	10573559	29386772	SQ 2: MS Scan	MS TIC
2	1.145	186.15	2458930	2687435	SQ 2: MS Scan	MS TIC
3	1.208	253.22	100527137	122977346	SQ 2: MS Scan	MS TIC
4	1.265	242.35	11206300	12954456	SQ 2: MS Scan	MS TIC



¹H NMR of Compound 3h¹³C NMR of Compound 3h

LCMS of Compound 3h

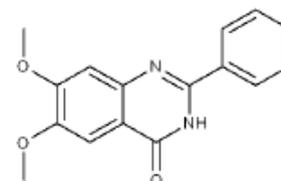


Peak Results
Channel: PDA Spectrum

	Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	1.084		1317	1337	0.45	PDA Spectrum	254.0nm
2	1.140		395224	293927	99.55	PDA Spectrum	254.0nm

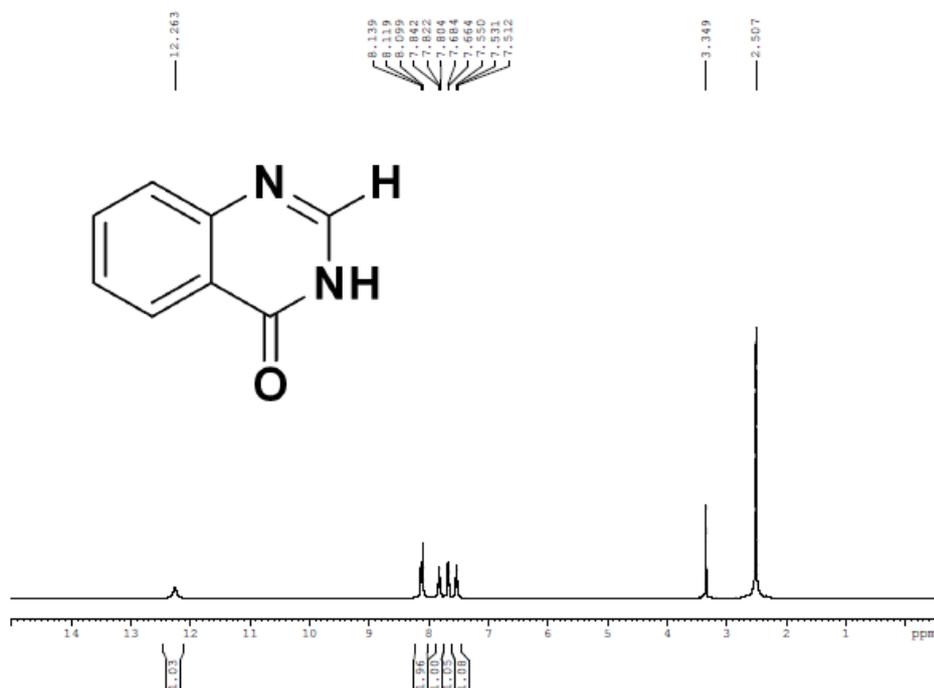
Peak Results
Channel: SQ 2: MS Scan

	Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	0.976	453.75	2653982	2390565	7.02	SQ 2: MS Scan	MS TIC
2	0.999	453.77	1359675	1484974	4.36	SQ 2: MS Scan	MS TIC
3	1.157	283.25	37418163	30196247	88.63	SQ 2: MS Scan	MS TIC



¹H NMR of Compound 3i

ADL/NMR-02



BRUKER

Current Data Parameters
 NAME 41570-NAPIER-SU-49
 EXPNO 1
 PROCNO 1

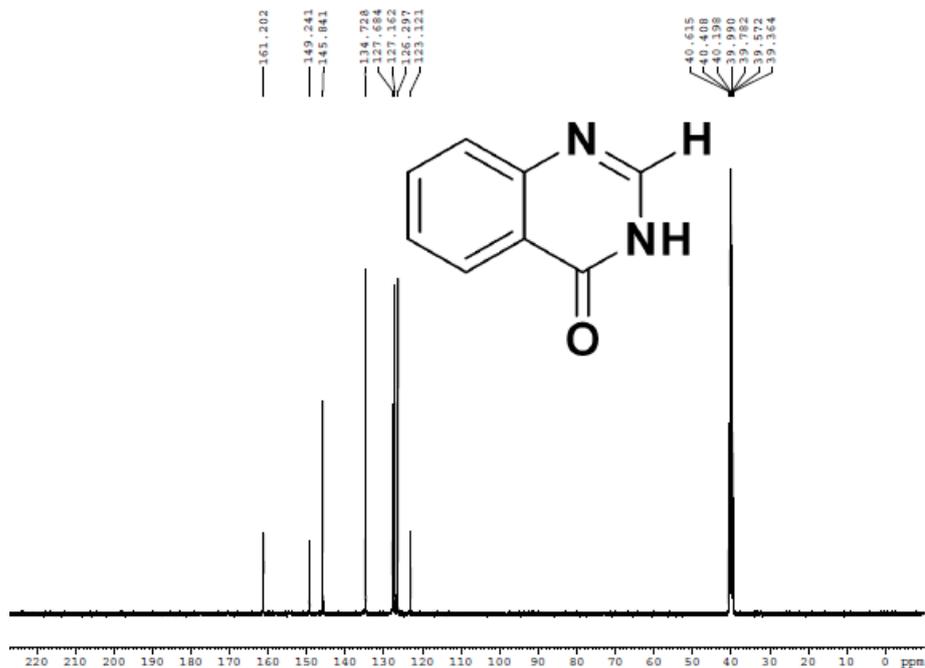
F2 - Acquisition Parameters
 Date_ 20200831
 Time 12.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 39998
 SOLVENT DMSO
 NS 64
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.250012 Hz
 AQ 1.9999501 sec
 RG 193.66
 DW 50.000 usec
 DE 6.50 usec
 TE 294.8 K
 D1 2.00000000 sec
 TDO 1

CHANNEL f1
 SFO1 400.1324710 MHz
 NUCL1 1H
 P1 13.60 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

¹³C NMR of Compound 3i

ADL/NMR-02



BRUKER

Current Data Parameters
 NAME 41677-NAPIER-SU-49
 EXPNO 1
 PROCNO 1

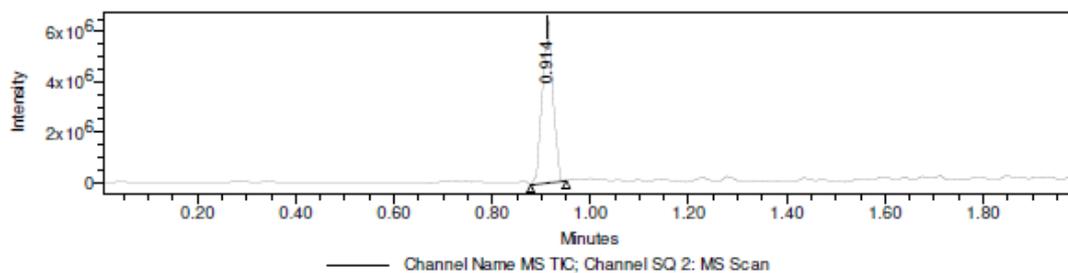
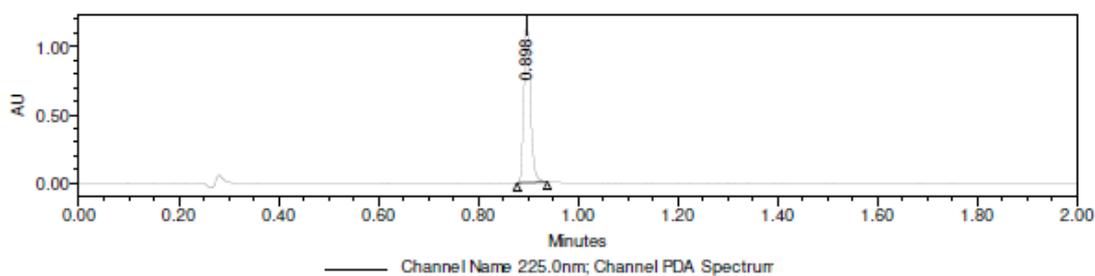
F2 - Acquisition Parameters
 Date_ 20200831
 Time 17.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1200
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8237536 sec
 RG 201.52
 DW 12.600 usec
 DE 6.50 usec
 TE 298.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

CHANNEL F1
 SFO1 100.6255801 MHz
 NUCL1 13C
 P1 9.50 usec
 PLW1 58.00000000 W

CHANNEL F2
 SFO2 400.3016012 MHz
 NUCL2 1H
 CPDPRG2 waltz16
 PCDPRG2 90.00 usec
 PLW2 15.00000000 W
 PLW12 0.32153000 W
 PLW13 0.268539999 W

F2 - Processing parameters
 SI 32768
 SF 100.6255151 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

LCMS of Compound 3i

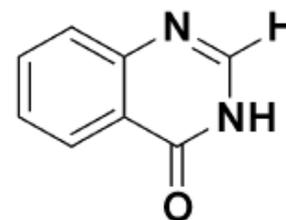


Peak Results
Channel: PDA Spectrum

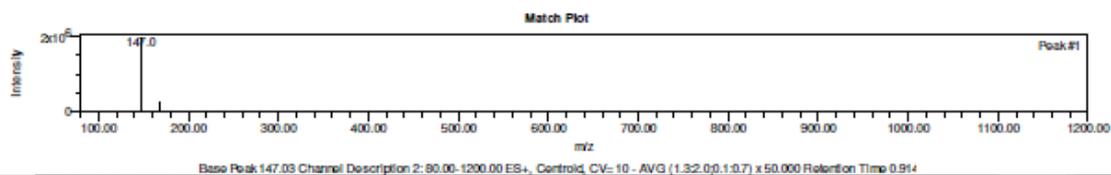
Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	0.898	1167548	1074415	100.00	PDA Spectrum	225.0nm

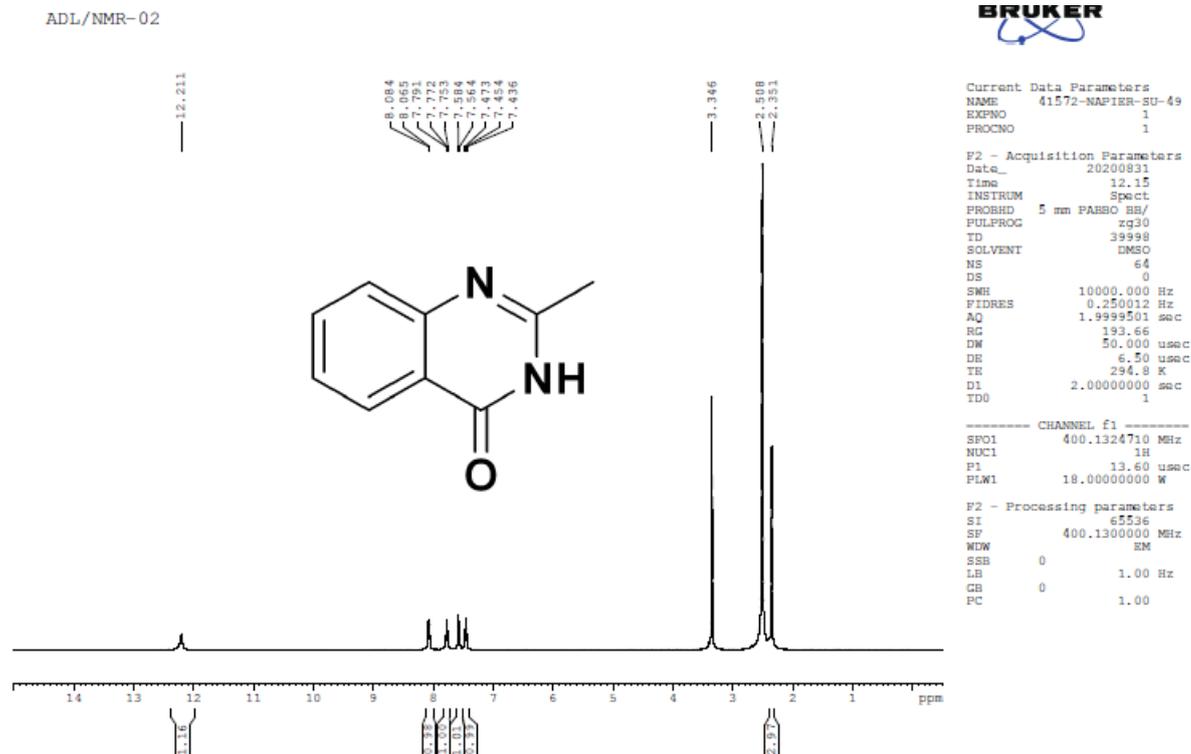
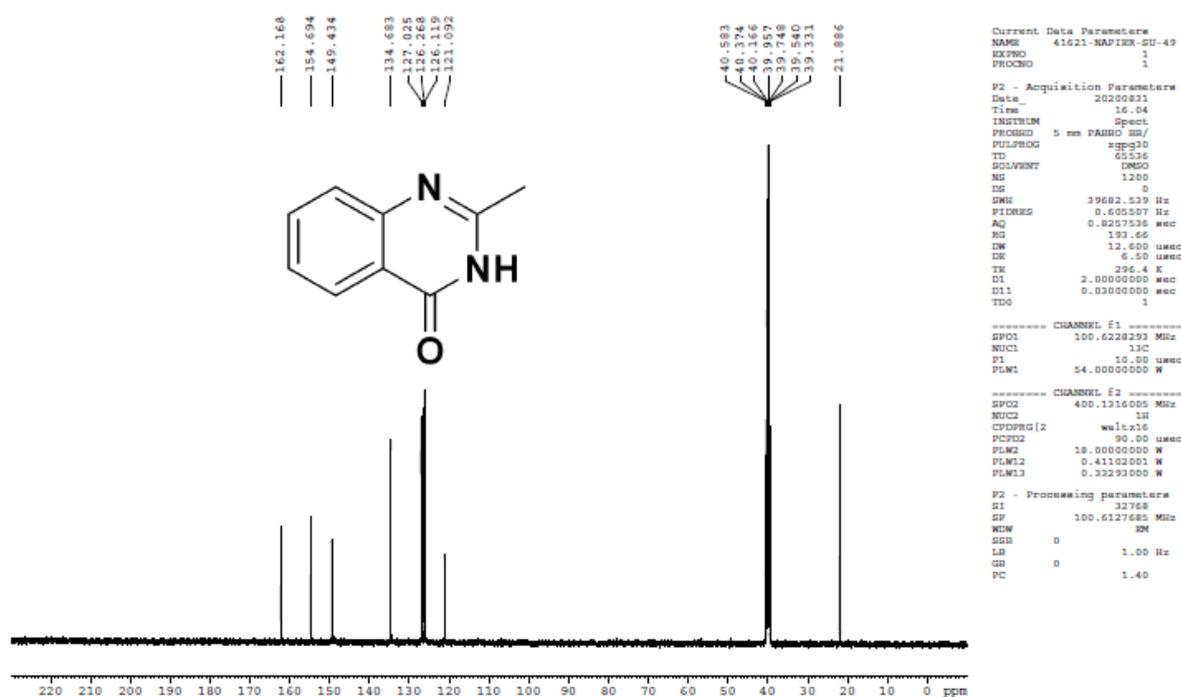
Peak Results
Channel: SQ 2: MS Scan

Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name	
1	0.914	147.03	6484181	9578072	100.00	SQ 2: MS Scan	MS TIC

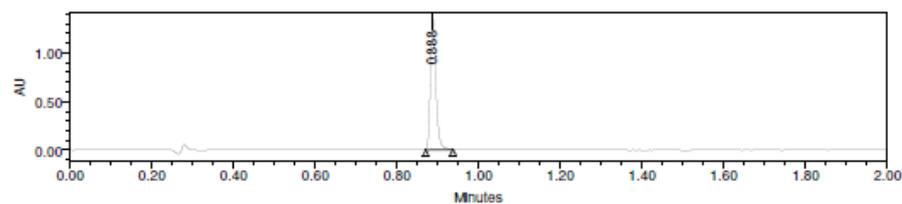


Exact Mass: 146.05

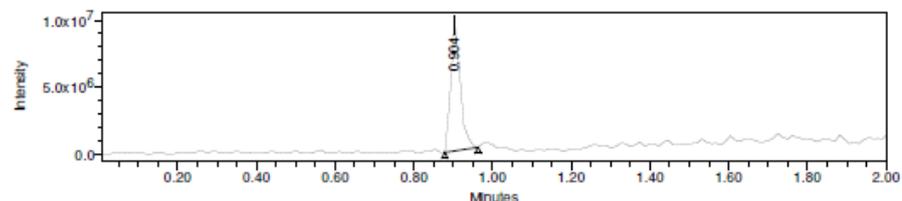


¹H NMR of Compound 3j¹³C NMR of Compound 3j

LCMS of Compound 3j



Channel Name 225.0nm; Channel PDA Spectrum



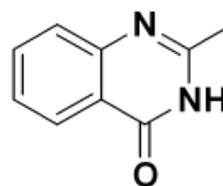
Channel Name MS TIC; Channel SQ 2: MS Scan

Peak Results
Channel: PDA Spectrum

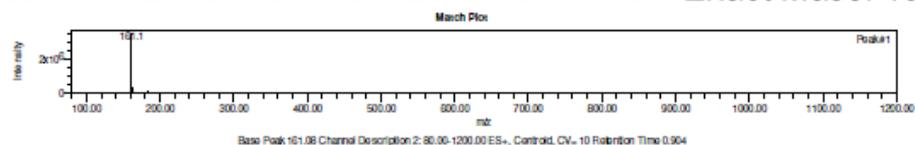
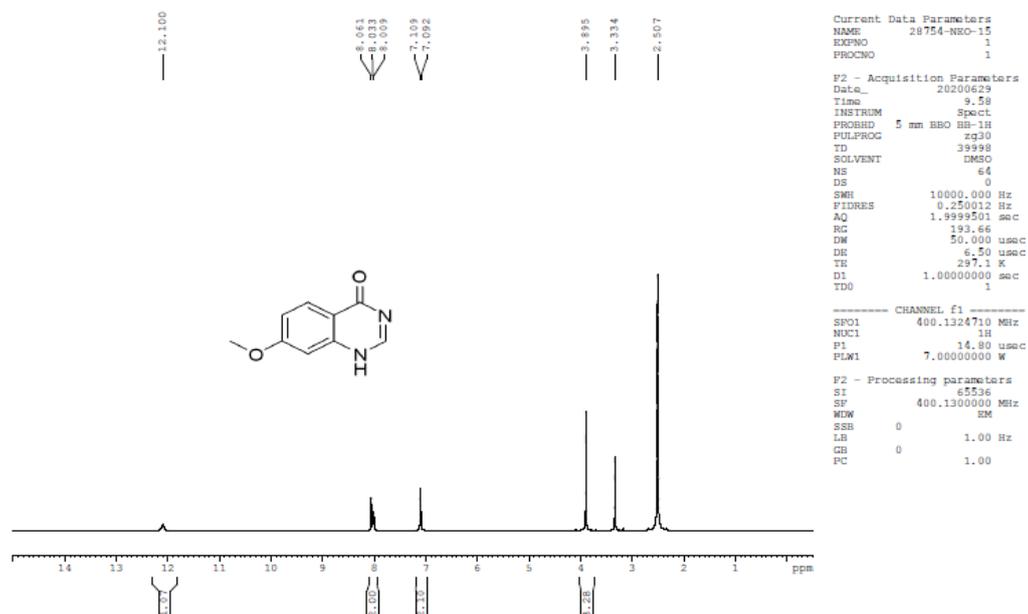
Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	0.888	1349228	1282966	100.00	PDA Spectrum	225.0nm

Peak Results
Channel: SQ 2: MS Scan

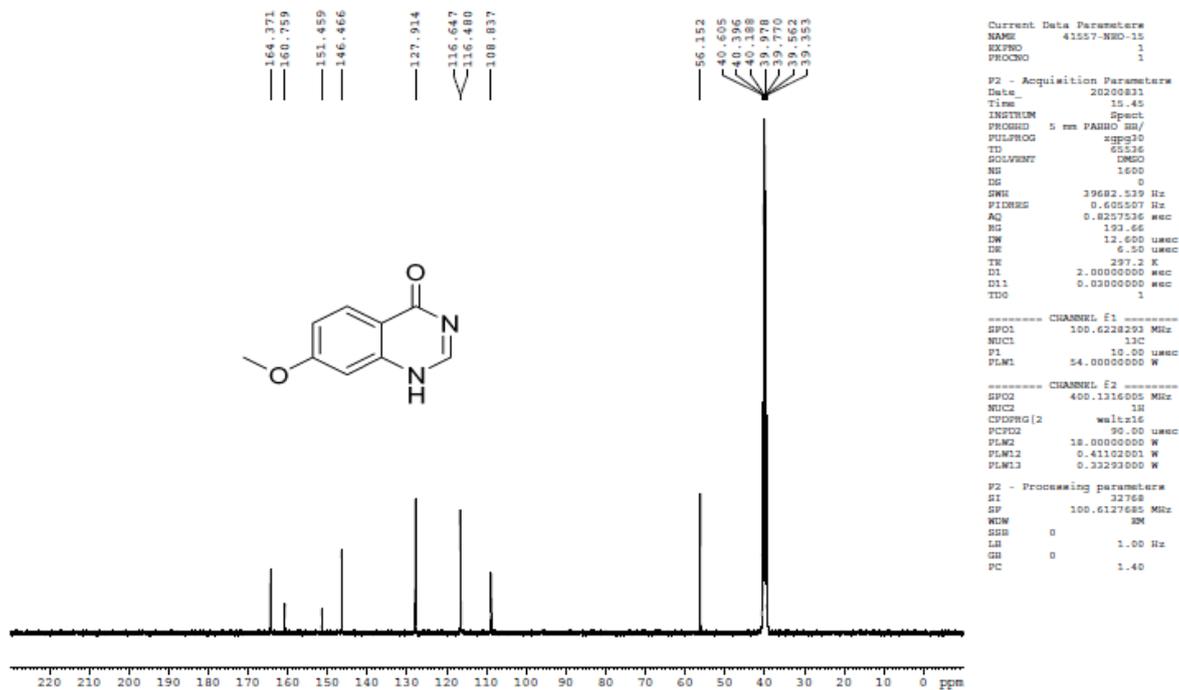
Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	0.904	161.08	16589953	100.00	SQ 2: MS Scan	MS TIC



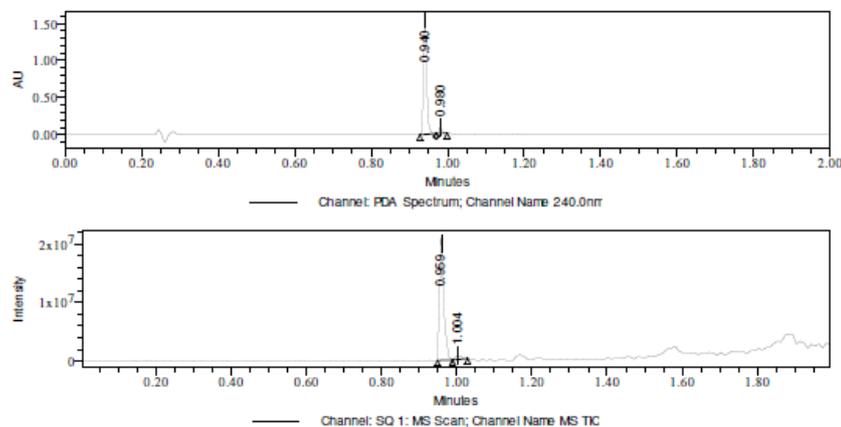
Exact Mass: 160.06

¹H NMR of Compound 3k

¹³C NMR of Compound 3k



LCMS of Compound 3k

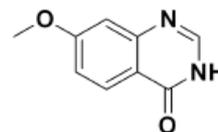


Peak Results
Channel: PDA Spectrum

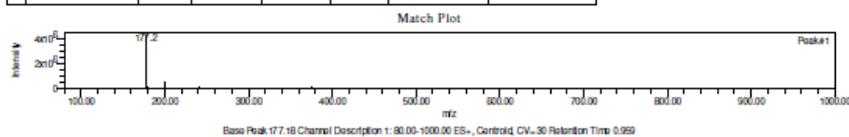
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
0.940		1573641	910073	94.70	PDA Spectrum	240.0nm
0.980		83887	50934	5.30	PDA Spectrum	240.0nm

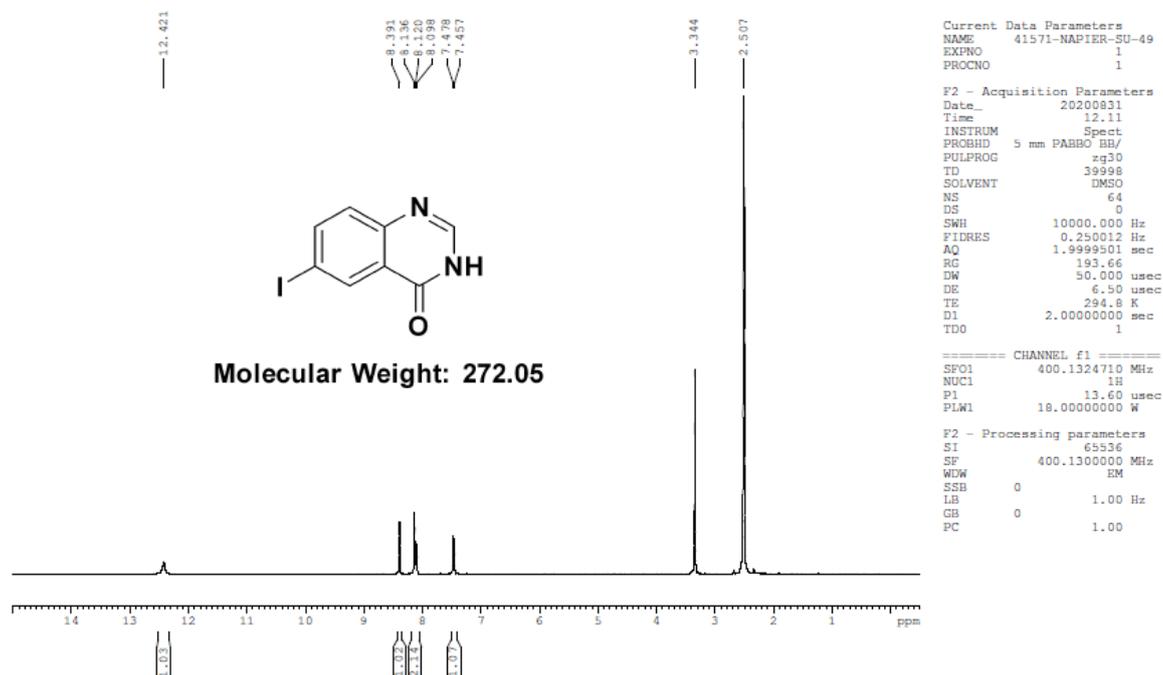
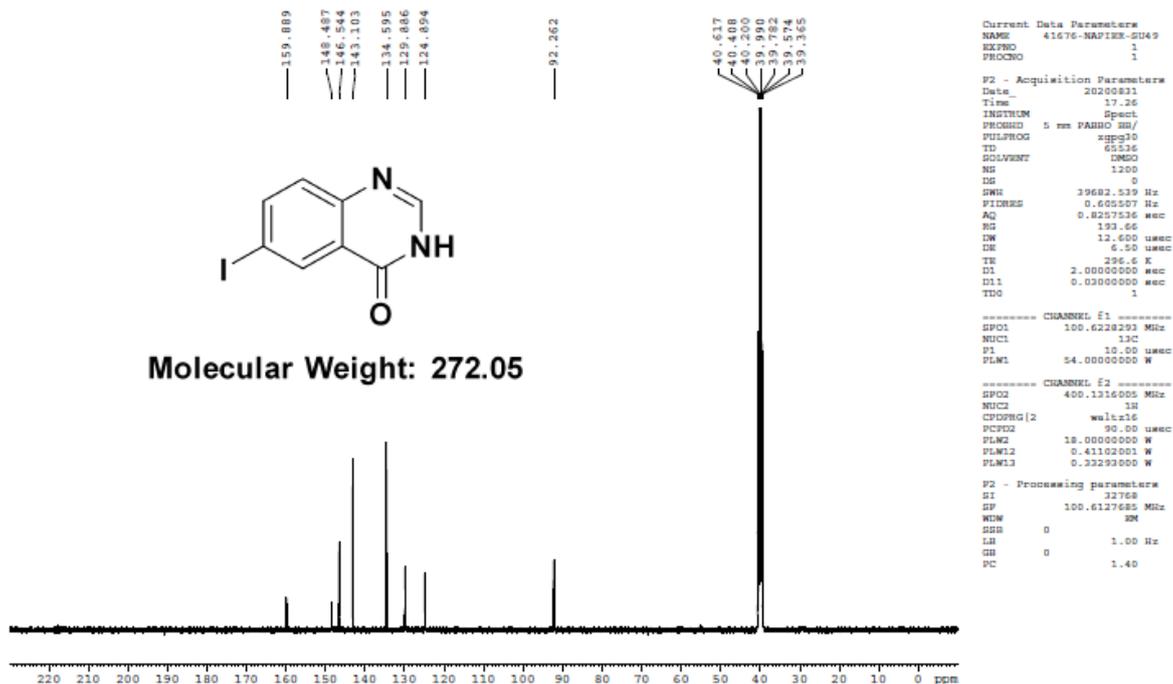
Peak Results
Channel: SQ 1: MS Scan

Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
0.959	177.18	21149185	16501433	95.84	SQ 1: MS Scan	MS TIC
1.004	211.20	668222	717121	4.16	SQ 1: MS Scan	MS TIC

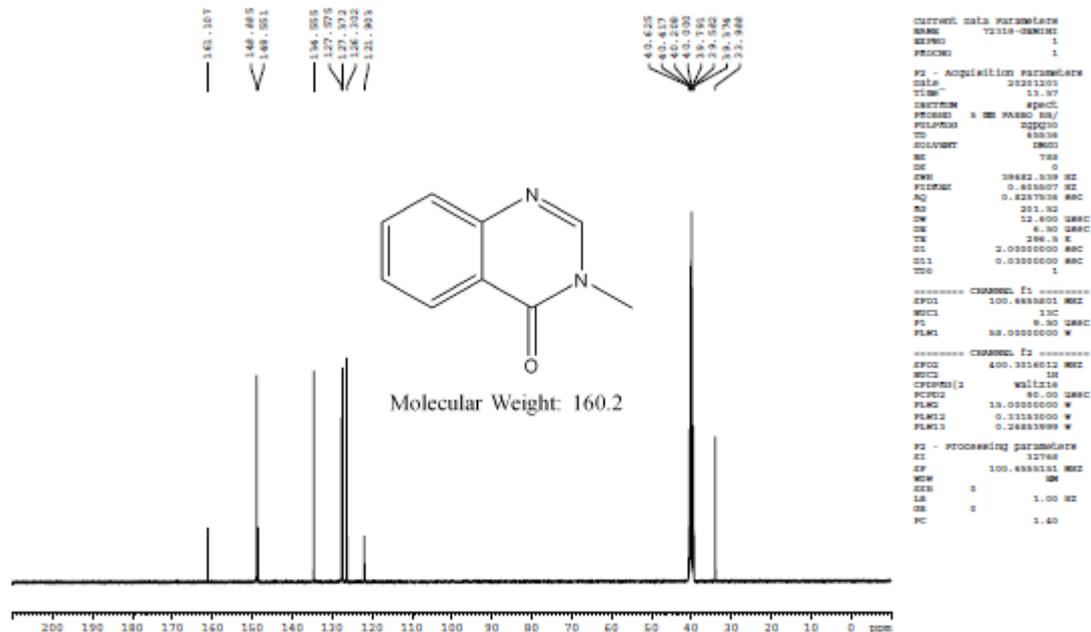


Molecular Weight: 176.18

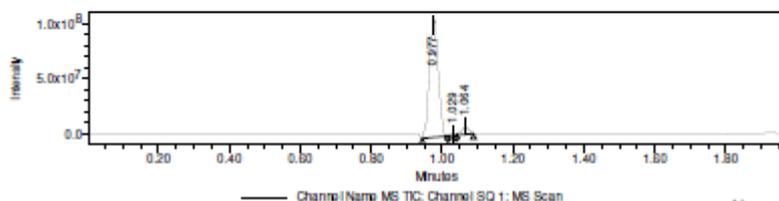
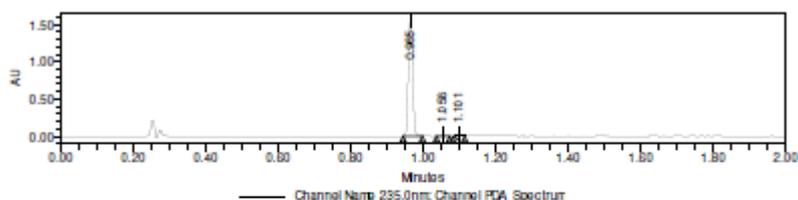


¹H NMR of Compound 3I¹³C NMR of Compound 3I

¹³C NMR of Compound 3m

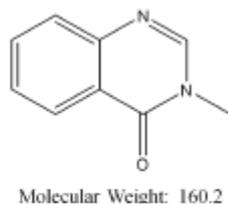


LCMS of Compound 3m



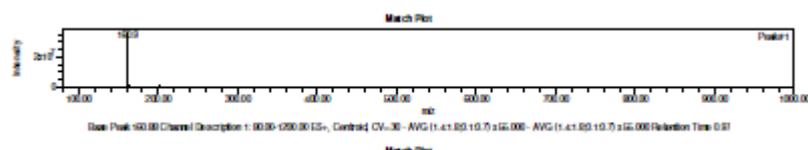
Peak Results
 Channel: PDA Spectrum

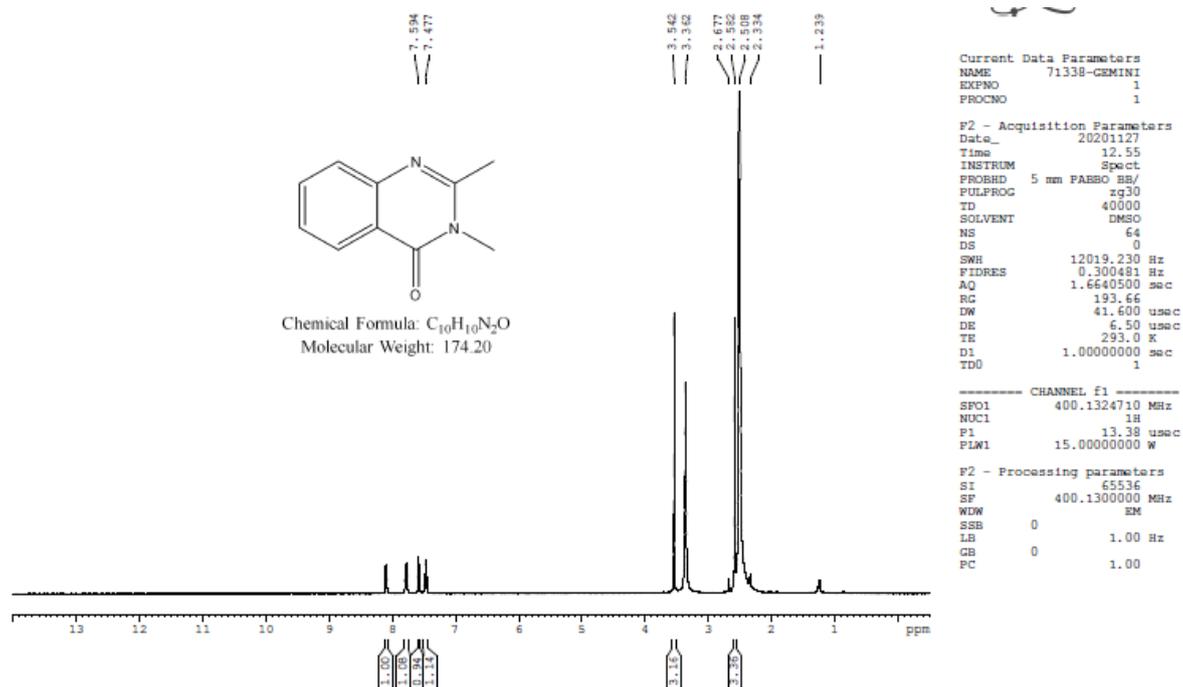
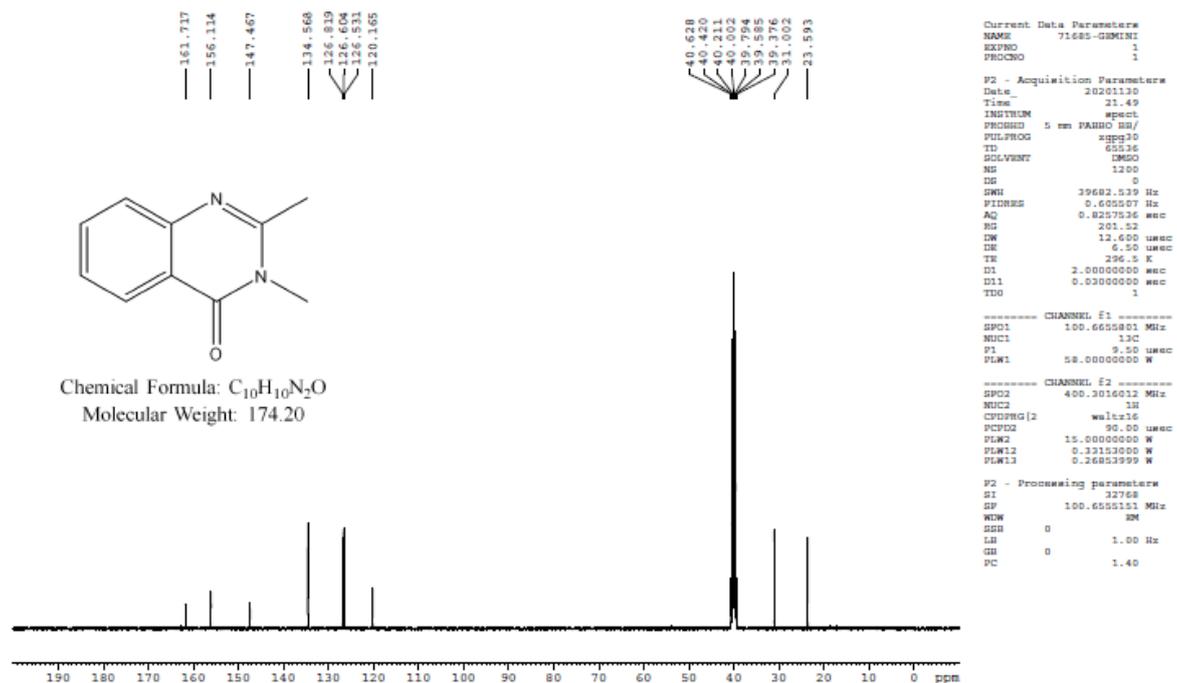
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	0.965	1582102	1295137	98.14	PDA Spectrum	235.0nm
2	1.058	21680	16190	1.23	PDA Spectrum	235.0nm
3	1.101	11195	8379	0.63	PDA Spectrum	235.0nm



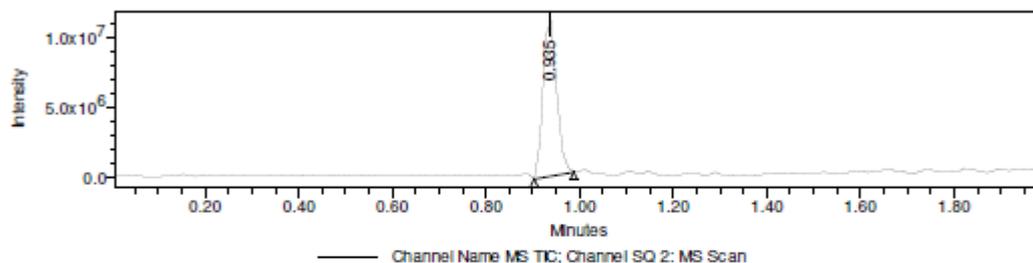
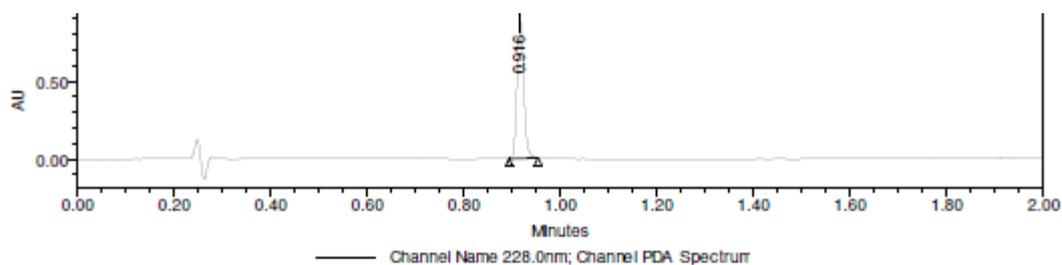
Peak Results
 Channel: SQ 1: MS Scan

Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name	
1	0.977	160.98	110044507	183552763	93.92	SQ 1: MS Scan	MS TIC
2	1.029	1105.02	1609890	1716392	0.88	SQ 1: MS Scan	MS TIC
3	1.064	216.03	5642525	10173522	5.21	SQ 1: MS Scan	MS TIC



¹H NMR of Compound 3n¹³C NMR of Compound 3n

LCMS of Compound 3n

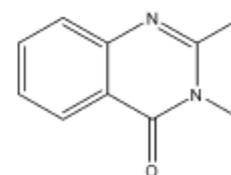


Peak Results
Channel: PDA Spectrum

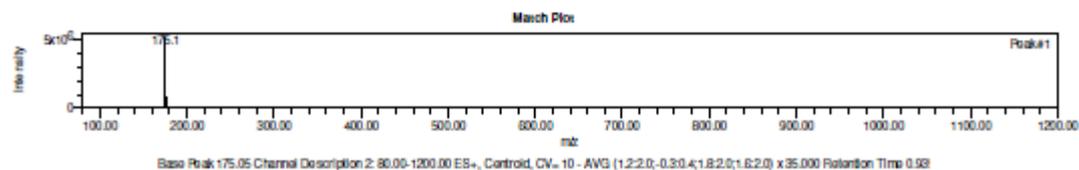
	Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	0.916		889491	870217	100.00	PDA Spectrum	228.0nm

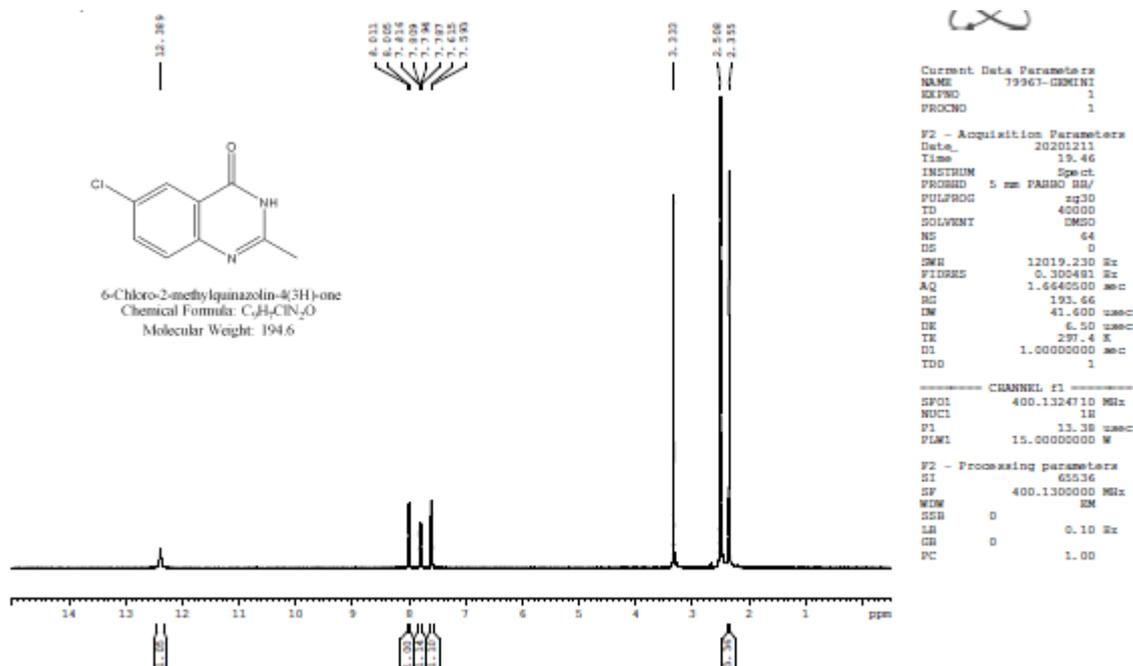
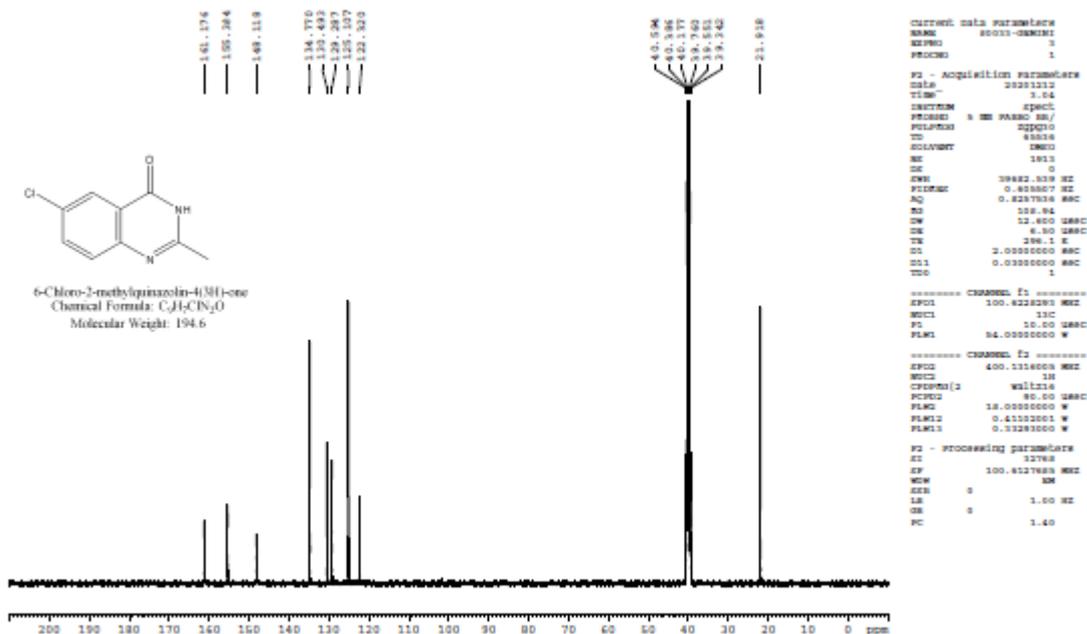
Peak Results
Channel: SQ 2: MS Scan

	Retention Time (min)	Base Peak (m/z)	Height (μV)	Area (μV*sec)	% Area	Channel	Channel Name
1	0.935	175.05	11581758	23012430	100.00	SQ 2: MS Scan	MS TIC

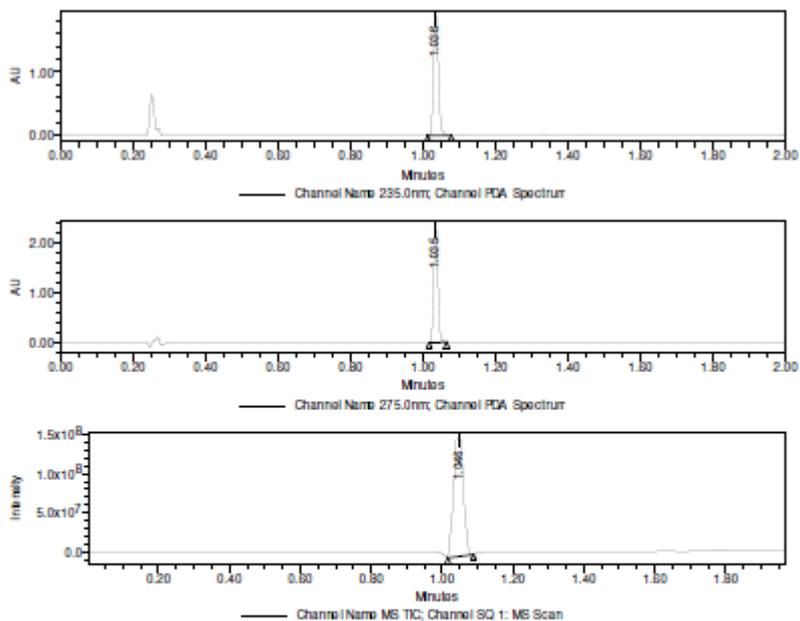


Chemical Formula: C₁₀H₁₀N₂O
Molecular Weight: 174.20



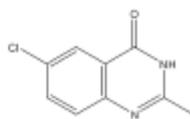
¹H NMR of Compound 3o¹³C NMR of Compound 3o

LCMS of Compound 3o



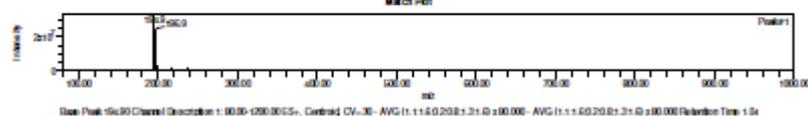
Peak Results
Channel: PDA Spectrum

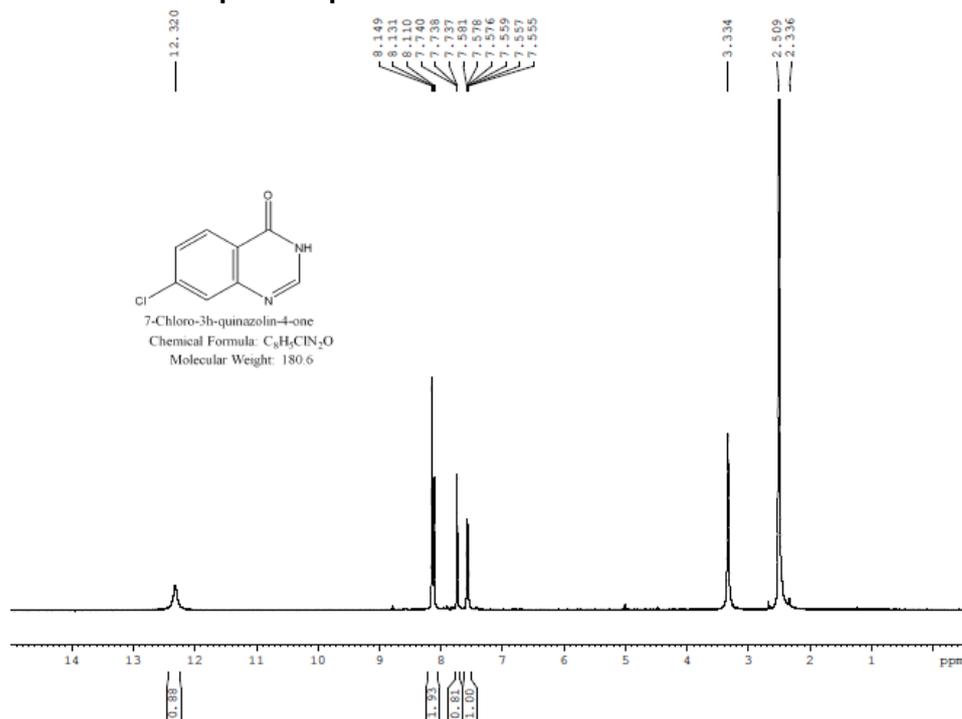
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	1.035	2283576	1903690	100.00	PDA Spectrum	275.0nm
2	1.036	1872125	1710508	100.00	PDA Spectrum	235.0nm



6-Chloro-2-methylquinazolin-4(3H)-one
Chemical Formula: C₉H₈ClN₂O
Molecular Weight: 194.6

Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name	
1	1.046	194.90	163240865	295294048	100.00	SQ 1: MS Scan	MS TIC



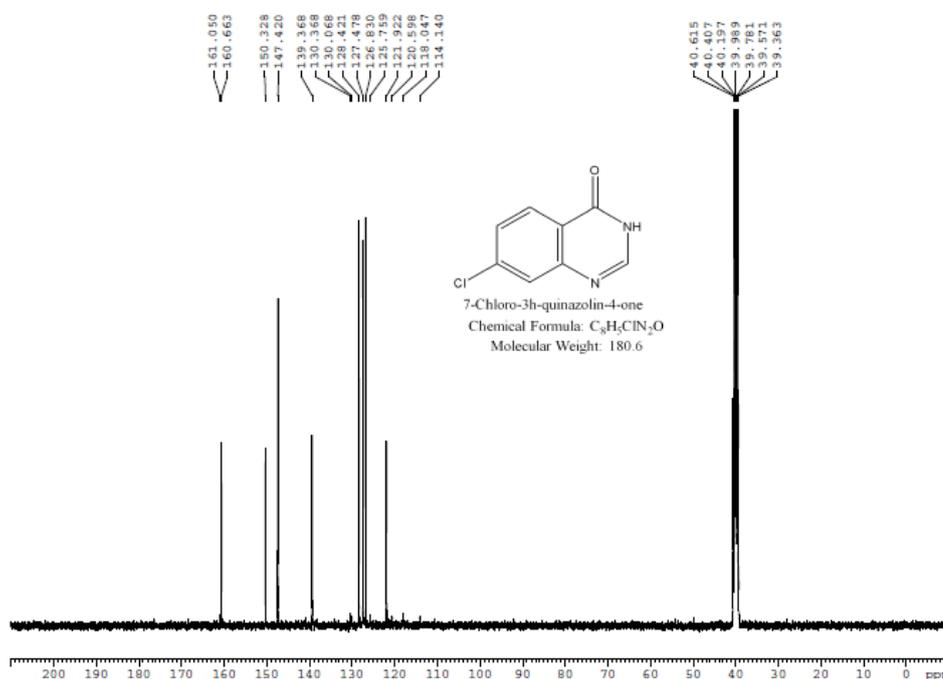
¹H NMR of Compound 3p

Current Data Parameters
NAME 79965-GEMINI
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201211
Time 19.37
INSTRUM Spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 40000
SOLVENT DMSO
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.300481 Hz
AQ 1.6640500 sec
RG 193.66
DW 41.600 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 400.1324710 MHz
NUC1 1H
P1 13.38 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹³C NMR of Compound 3p

Current Data Parameters
NAME 80065-GEMINI
EXPNO 1
PROCNO 1

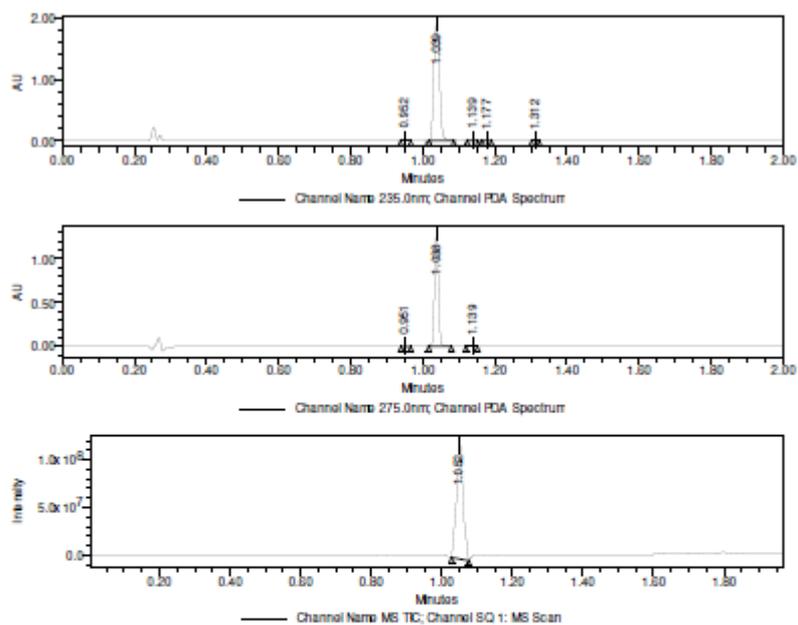
F2 - Acquisition Parameters
Date_ 20201212
Time 0.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2981
DS 0
SWH 39682.539 Hz
FIDRES 0.605507 Hz
AQ 0.8257536 sec
RG 201.52
DW 12.600 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 100.6258801 MHz
NUC1 13C
P1 9.50 usec
PLW1 58.00000000 W

----- CHANNEL f2 -----
SFO2 400.3016012 MHz
NUC2 1H
CDDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33153000 W
PLW13 0.26853999 W

F2 - Processing parameters
SI 32768
SF 100.6258801 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

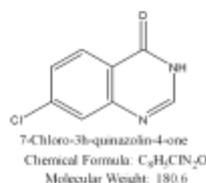
LCMS of Compound 3p



Peak Results

Channel: PDA Spectrum

Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	0.951	1748	1249	0.13	PDA Spectrum	275.0nm
2	0.952	10488	7484	0.35	PDA Spectrum	235.0nm
3	1.038	1296247	954597	99.85	PDA Spectrum	275.0nm
4	1.039	1927208	2100831	98.95	PDA Spectrum	235.0nm
5	1.139	2057	2129	0.22	PDA Spectrum	275.0nm
6	1.139	12982	8963	0.42	PDA Spectrum	235.0nm



Channel: PDA spectrum

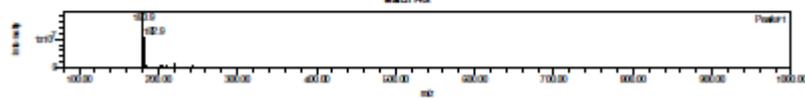
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
7	1.177	4356	2829	0.13	PDA Spectrum	235.0nm
8	1.312	4281	2916	0.14	PDA Spectrum	235.0nm

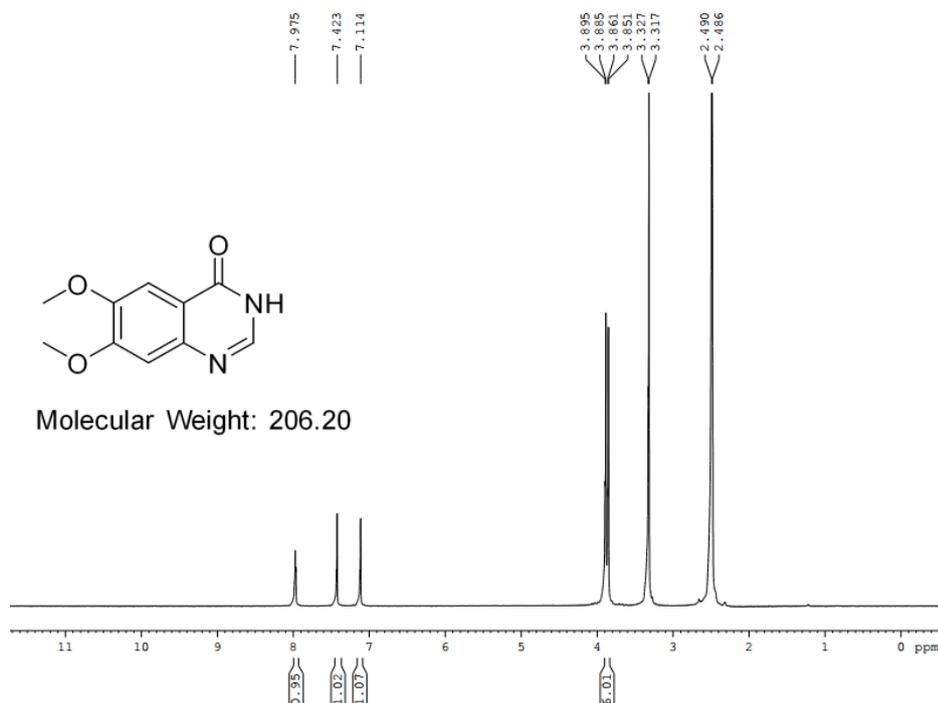
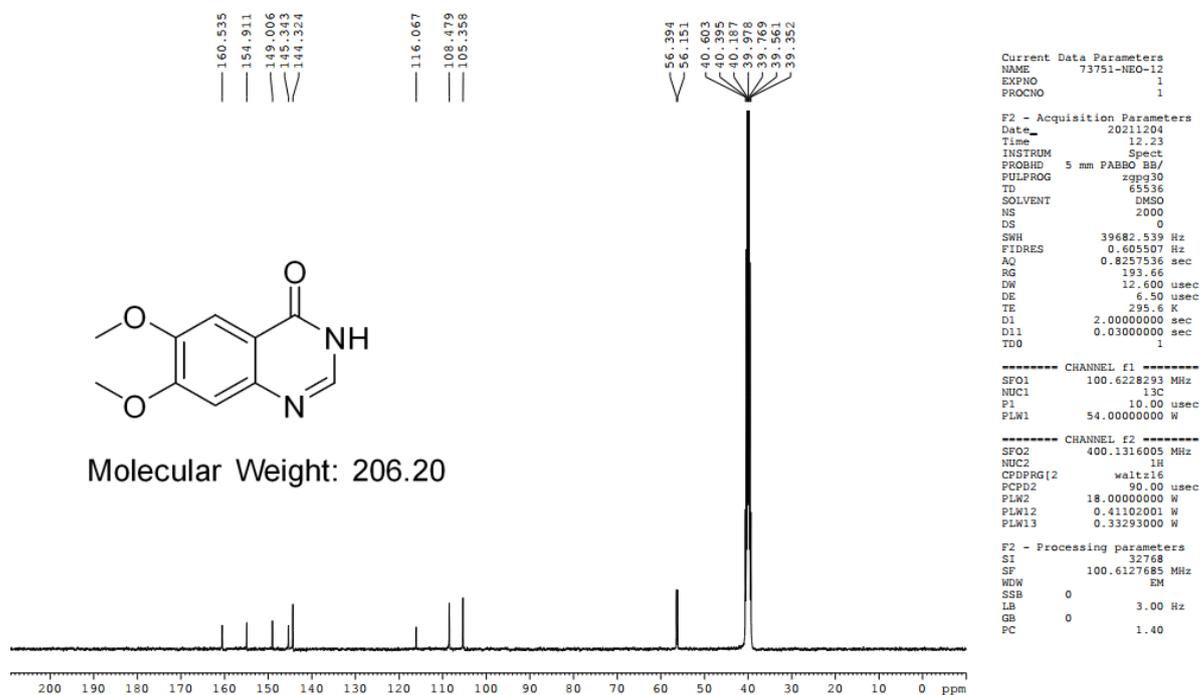
Peak Results

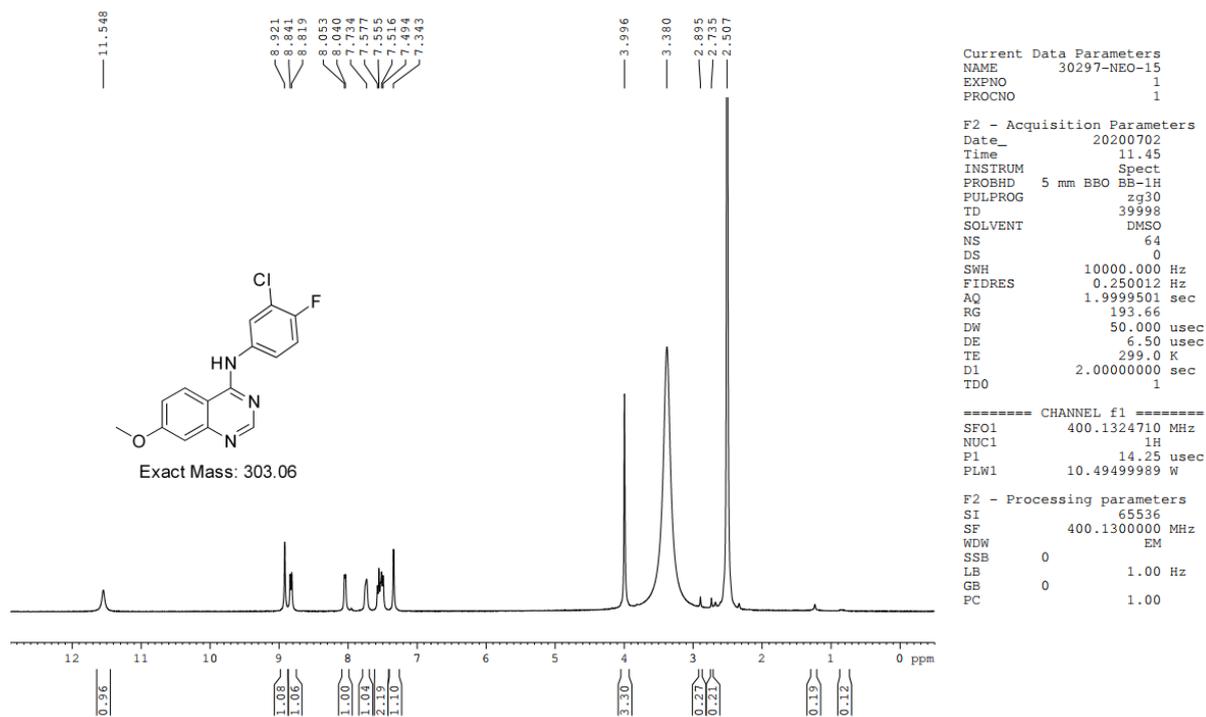
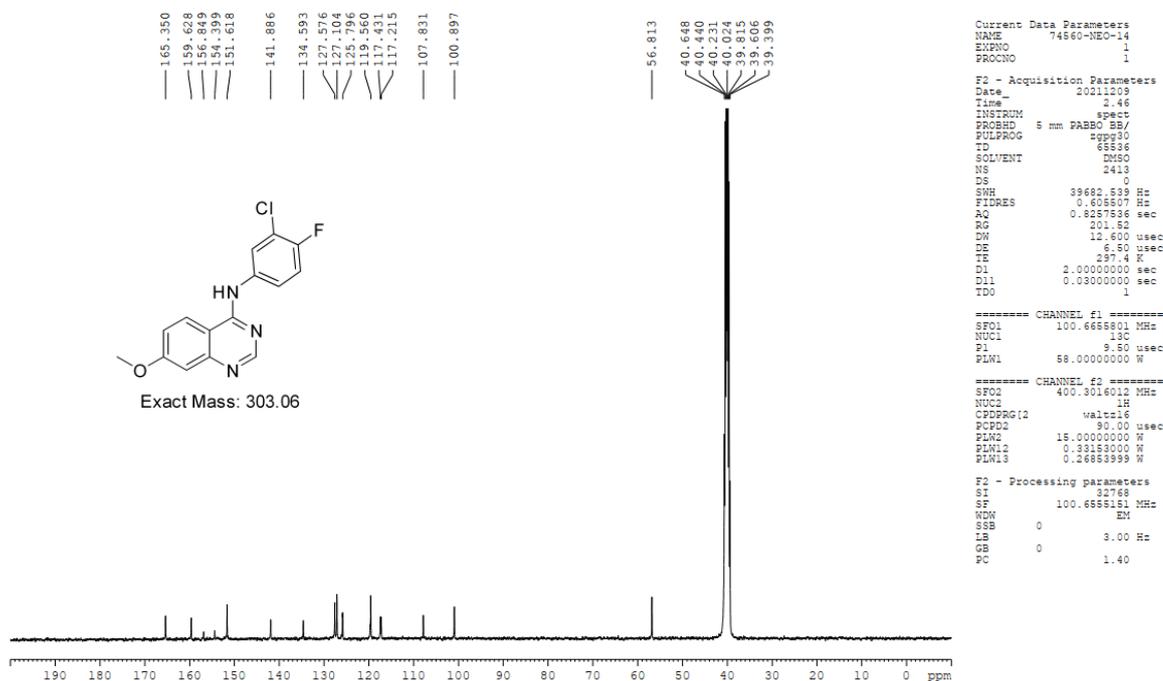
Channel: SQ 1: MS Scan

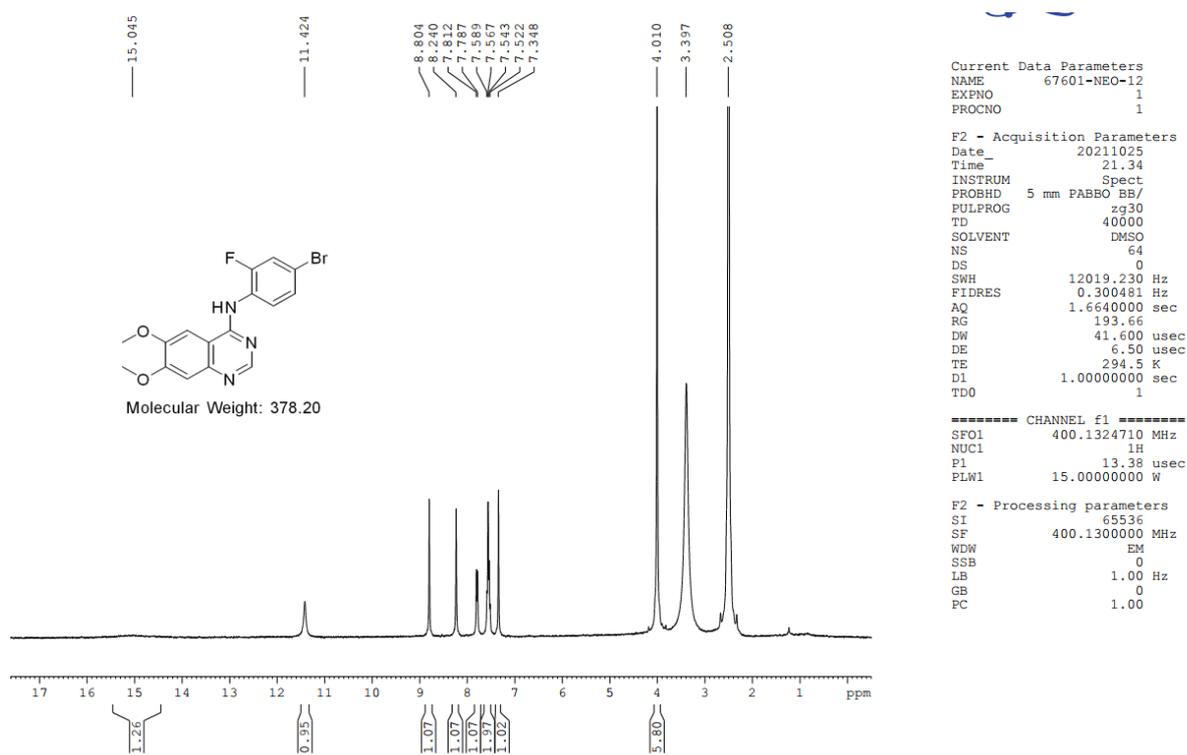
Retention Time (min)	Base Peak (m/z)	Height (µV)	Area (µV*sec)	% Area	Channel	Channel Name
1	1.052	180.89	122853485	100.00	SQ 1: MS Scan	MS TIC

Match Plot



¹H NMR of Compound 3q¹³C NMR of Compound 3q

¹H NMR of Compound 4¹³C NMR of Compound 4

¹H NMR of Compound 5¹³C NMR of Compound 5