Identification, characterization and quantification of process-related and degradation impurities in Lisdexamfetamine dimesylate: Two identified as new compounds

Shenghua Gao[1], Lili Meng[1], Chunjie Zhao[2], Tao Zhang[1], Pengcheng Qiu [1,*]and Fuli Zhang[1,*]

* Correspondence: zhangfuli1@sinopharm.com(F.Z.); pcqiu@aliyun.com (P.Q.);

Tel.:+86-21-2057-2000(ext.5036) (F.Z.); +86-21-2057-2000(ext.5077) (P.Q.)

Supporting Information

Table of contents

Molecules 2018, *23*, 3125; doi:10.3390/molecules23123125

Figure S5 The LC-MS spectrum of LDX and its impurities

(a) The LC chromatogram of LDX and its impurities;

(b) The MS spectrum of Imp-L;

(c) The MS spectrum of Imp-M;

(d) The MS spectrum of Imp-A;

(e) The MS spectrum of Imp-H;

(f) The MS spectrum of LDX;

(g) The MS spectrum of Imp-E;

(h) The MS spectrum of Imp-D;

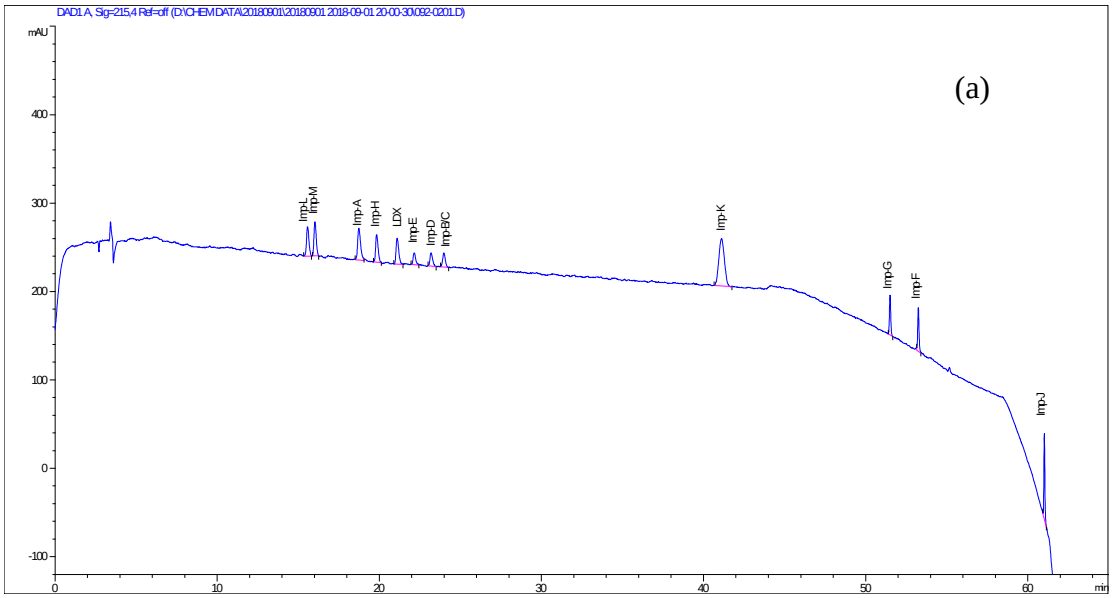
(i) The MS spectrum of Imp-B or C;

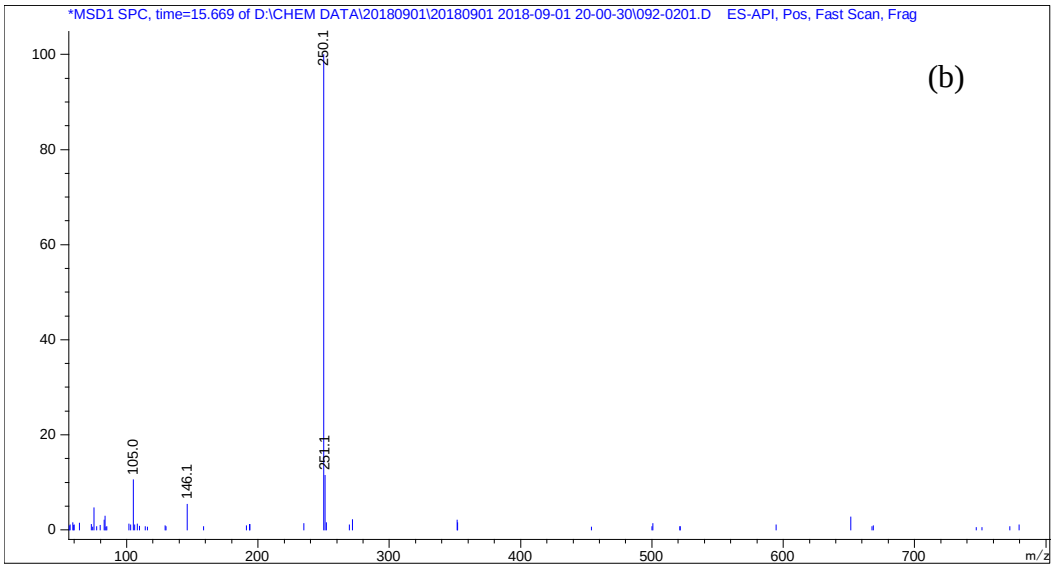
(j) The MS spectrum of Imp-K;

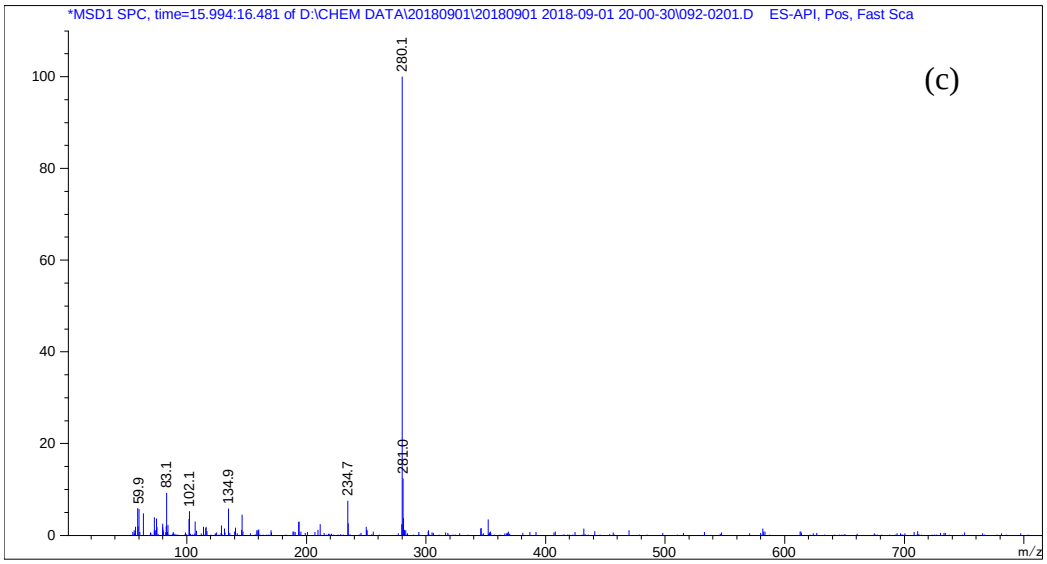
(k) The MS spectrum of Imp-G;

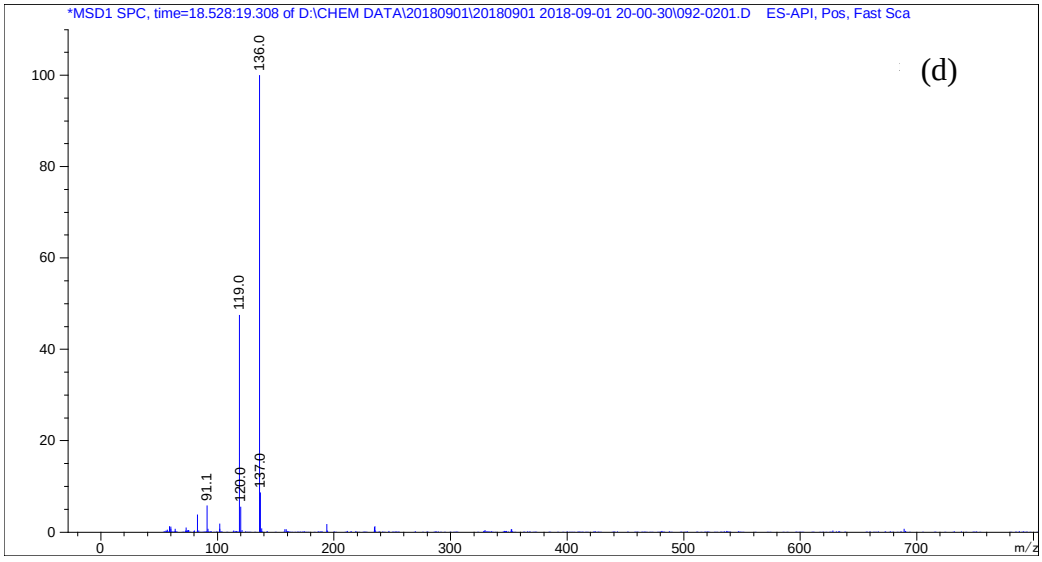
(l) The MS spectrum of Imp-F;

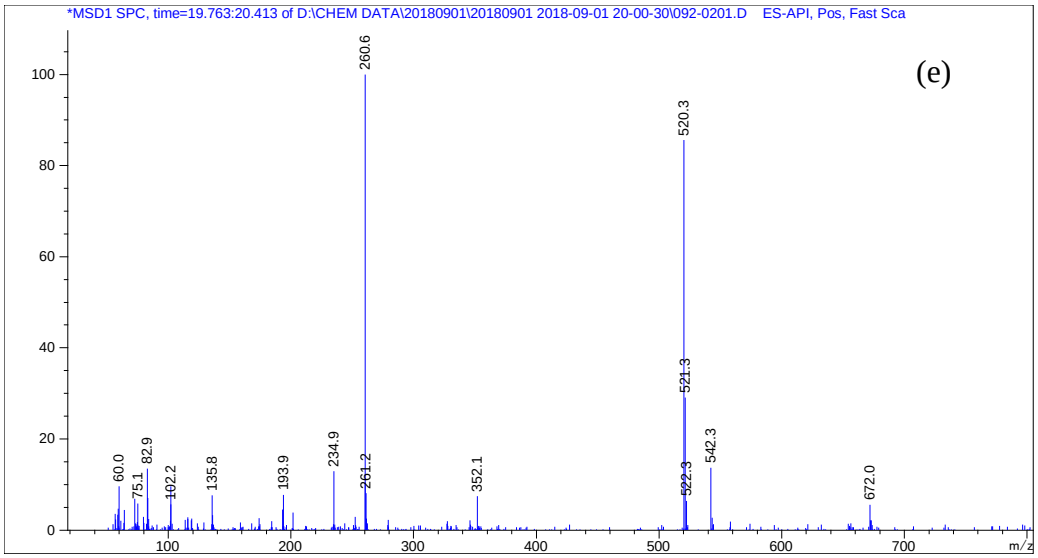
(m) The MS spectrum of Imp-J.

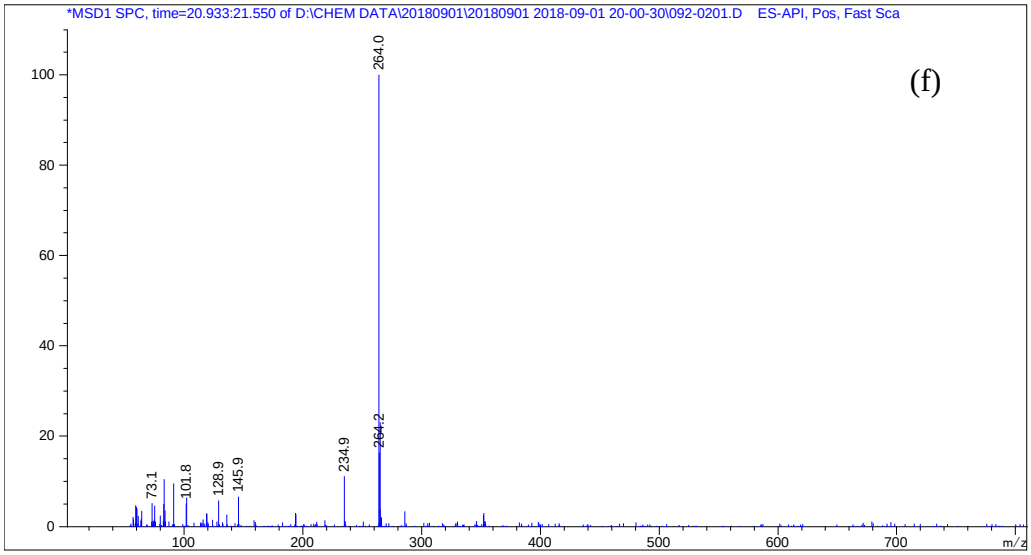


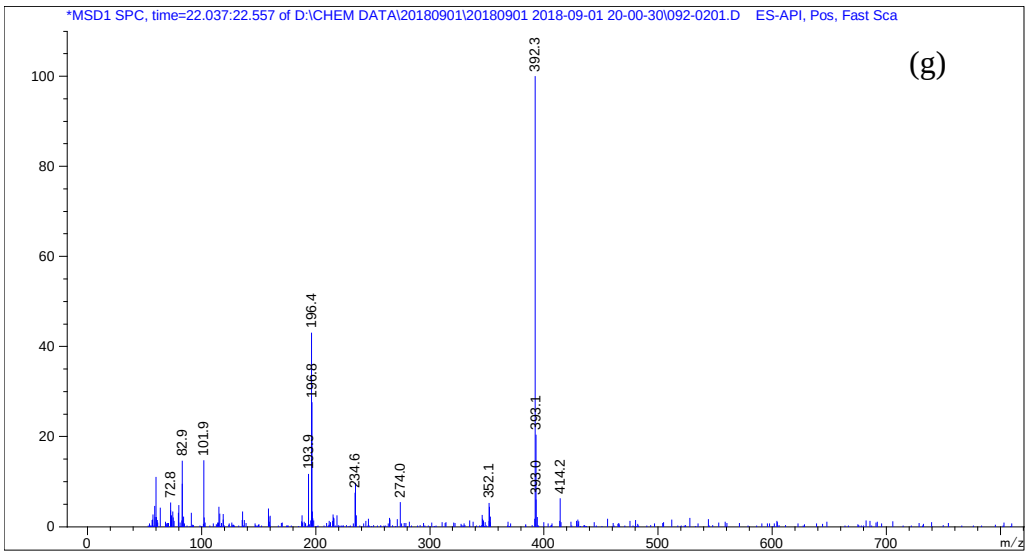

*MSD1 SPC, time=15.669 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Scan, Frag
(b)
250.1
105.0
146.1
251.1
m/z

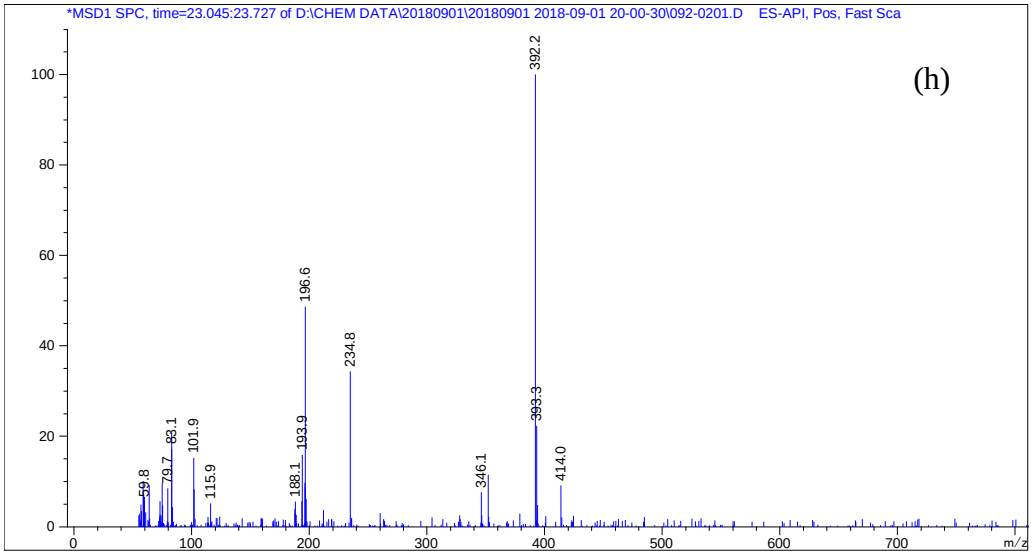

*MSD1 SPC, time=15.994:16.481 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(c)
280.1
59.9
83.1
102.1
134.9
234.7
281.0
m/z

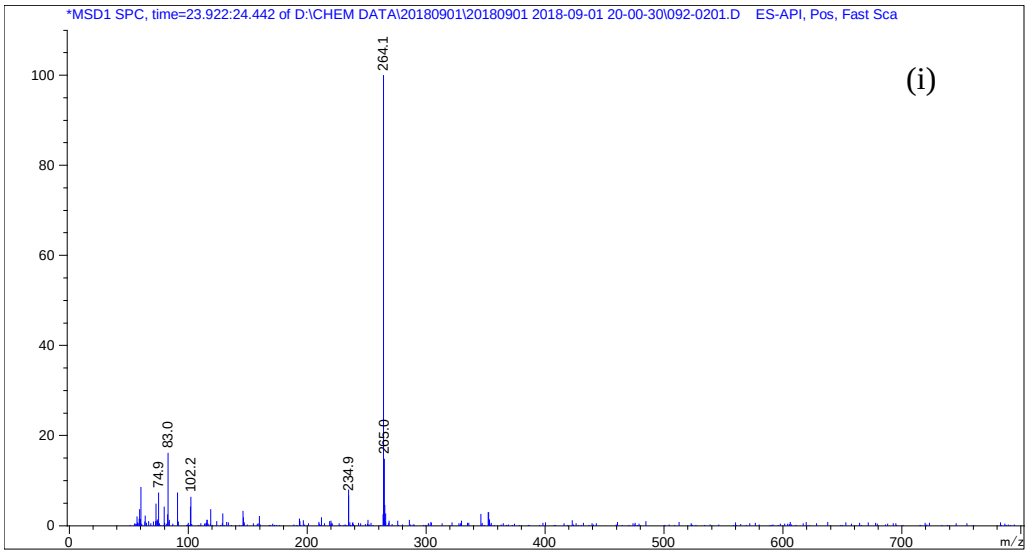

*MSD1 SPC, time=18.528:19.308 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(d)
136.0
119.0
91.1
120.0
137.0
m/z

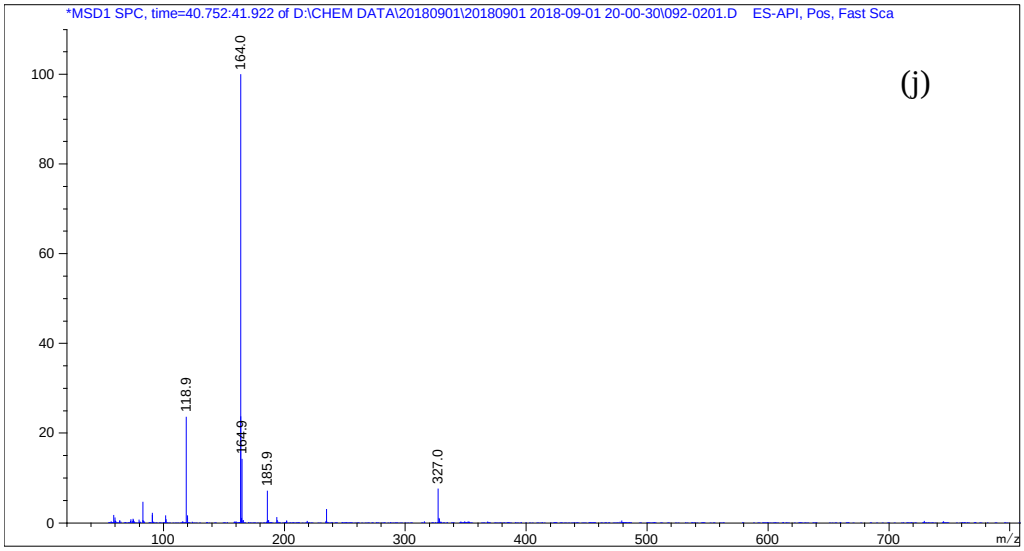

*MSD1 SPC, time=19.763:20.413 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(e)
260.6
520.3
521.3
60.0
82.9
75.1
102.2
135.8
193.9
234.9
261.2
352.1
522.3
542.3
672.0
m/z

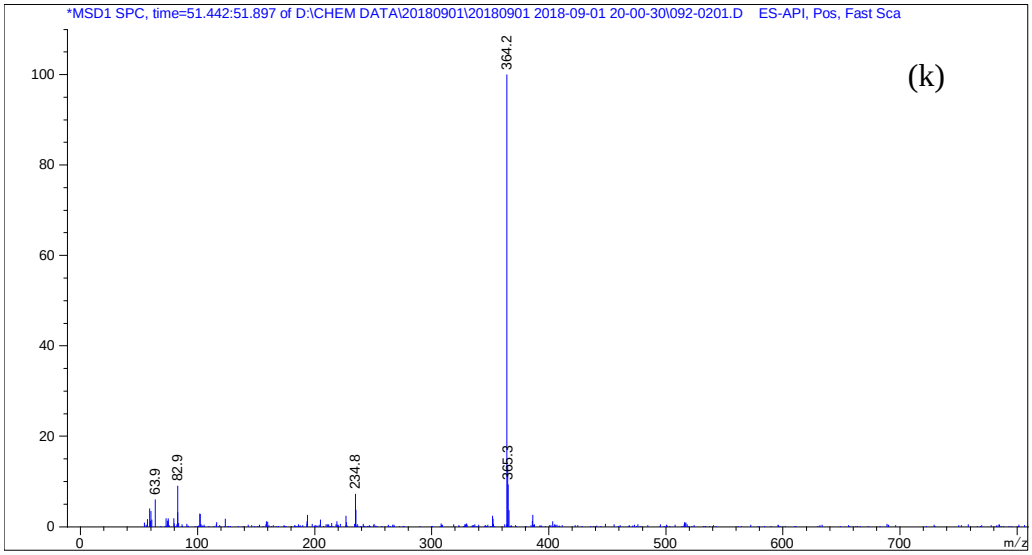

*MSD1 SPC, time=20.933:21.550 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(f)
264.0
264.2
234.9
73.1
101.8
128.9
145.9
m/z

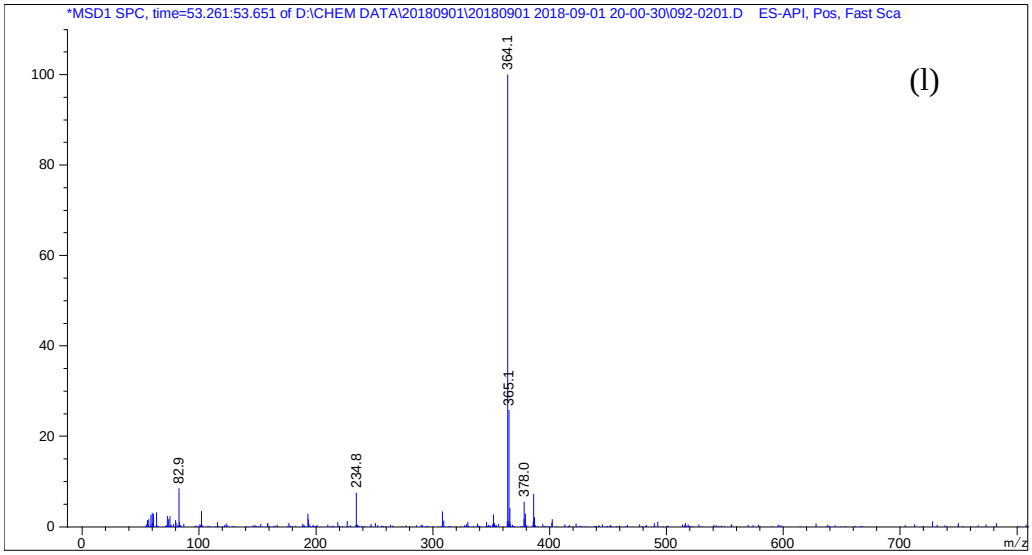

*MSD1 SPC, time=22.037:22.557 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(g)
392.3
196.4
196.8
393.1
82.9
101.9
72.8
193.9
234.6
274.0
352.1
393.0
414.2
m/z

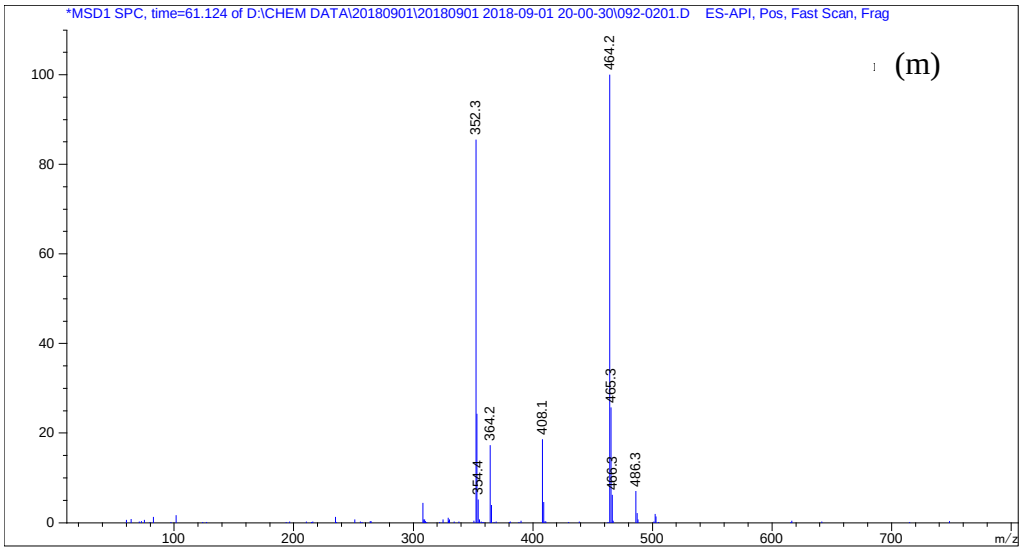

*MSD1 SPC, time=23.045:23.727 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(h)
392.2
196.6
234.8
393.3
83.1
101.9
193.9
59.8
79.7
115.9
188.1
346.1
414.0
m/z


*MSD1 SPC, time=23.922:24.442 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(i)
264.1
83.0
265.0
74.9
102.2
234.9
m/z


*MSD1 SPC, time=40.752:41.922 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(j)
164.0
118.9
164.9
185.9
327.0
m/z


*MSD1 SPC, time=51.442:51.897 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(k)
364.2
365.3
63.9
82.9
234.8
m/z


*MSD1 SPC, time=53.261:53.651 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Sca
(l)
364.1
365.1
82.9
234.8
378.0
m/z


*MSD1 SPC, time=61.124 of D:\CHEM DATA\20180901\20180901 2018-09-01 20-00-30\092-0201.D ES-API, Pos, Fast Scan, Frag
(m)
464.2
352.3
465.3
364.2
408.1
354.4
466.3
486.3
m/z

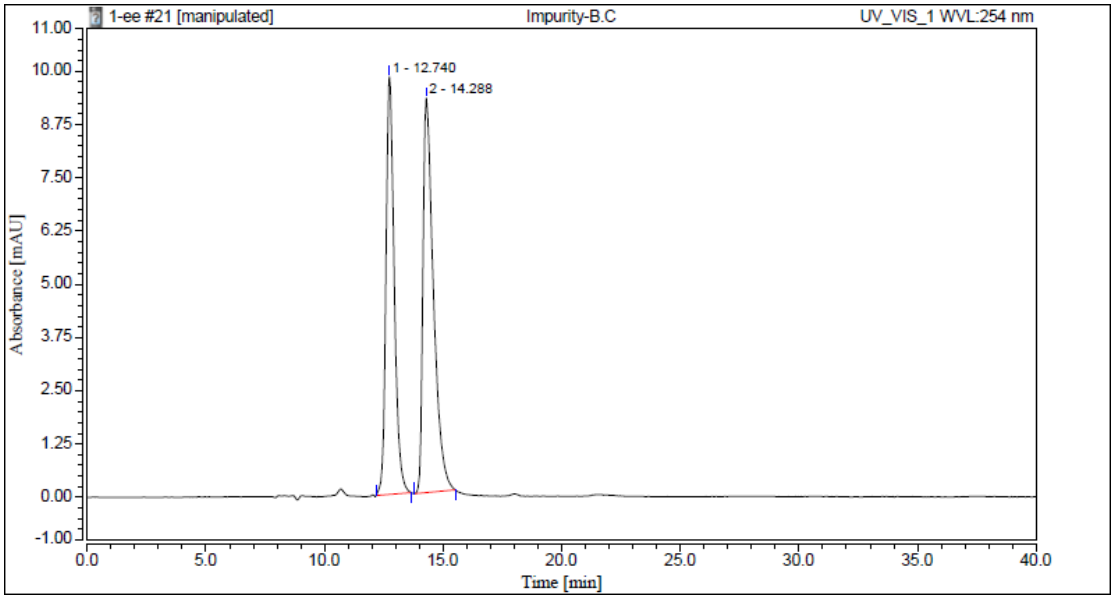


Figure S6 The spectrum of Imp-B and Imp-C in normal-phase chromatography

FigureS7 The spectrums of LDX and its impurities

(X-1) the ^{1}H NMR spectrum of LDX;

(X-2) the ^{13}C NMR spectrum of LDX;

(X-3) the DEPT spectrum of LDX;

(X-4) the IR spectrum of LDX;

(X-5) the HRMS spectrum of LDX;

(A-1) the ^{1}H NMR spectrum of Imp-A;

(A-2) the ^{13}C NMR spectrum of Imp-A;

(A-3) the DEPT spectrum of Imp-A;

(A-4) the IR spectrum of Imp-A;

(A-5) the HRMS spectrum of Imp-A;

(B-1) the ^{1}H NMR spectrum of Imp-B;

(C-1) the ^{1}H NMR spectrum of Imp-C;

(C-2) the ^{13}C NMR spectrum of Imp-C;

(C-3) the DEPT spectrum of Imp-C;

(C-4) the HRMS spectrum of Imp-C;

(D-1) the ^{1}H NMR spectrum of Imp-D;

(D-2) the ^{13}C NMR spectrum of Imp-D;

(D-3) the DEPT spectrum of Imp-D;

(D-4) the HSQC spectrum of Imp-D;

(D-5) the HMBC spectrum of Imp-D;

(D-6) the enlarged view of the HMBC spectrum of Imp-D;

(D-7) the IR spectrum of Imp-D;

(D-8) the HRMS spectrum of Imp-D;

(E-1) the ^{1}H NMR spectrum of Imp-E;

(E-2) the ^{13}C NMR spectrum of Imp-E;

(E-3) the DEPT spectrum of Imp-E;

(E-4) the HSQC spectrum of Imp-E;

(E-5) the HMBC spectrum of Imp-E;

(E-6) the enlarged view of the HMBC spectrum of Imp-E;

(E-7) the IR spectrum of Imp-E;

(E-8) the HRMS spectrum of Imp-E;

(F-1) the ^{1}H NMR spectrum of Imp-F;

(F-2) the ^{13}C NMR spectrum of Imp-F;

(F-3) the DEPT spectrum of Imp-F;

(F-4) the IR spectrum of Imp-F;

(F-5) the HRMS spectrum of Imp-F;

(G-1) the ^{1}H NMR spectrum of Imp-G;

(G-2) the ^{13}C NMR spectrum of Imp-G;

(G-3) the DEPT spectrum of Imp-G;

(G-4) the IR spectrum of Imp-G;

(G-5) the HRMS spectrum of Imp-G;

(H-1) the ^{1}H NMR spectrum of Imp-H;

(H-2) the ^{13}C NMR spectrum of Imp-H;

(H-3) the DEPT spectrum of Imp-H;

(H-4) the HMBC spectrum of Imp-H;

(H-5) the enlarged view of the HMBC spectrum of Imp-H;

(H-6) the HSQC spectrum of Imp-H;

(H-7) the COSY spectrum of Imp-H;

(H-8) the enlarged view of the COSY spectrum of Imp-H;

(H-9) the IR spectrum of Imp-H;

(H-10) the HRMS spectrum of Imp-H;

(J-1) the ^{1}H NMR spectrum of Imp-J;

(J-2) the ^{13}C NMR spectrum of Imp-J;

(J-3) the DEPT spectrum of Imp-J;

(J-4) the HRMS spectrum of Imp-J;

(K-1) the ^{1}H NMR spectrum of Imp-K;

(K-2) the ^{13}C NMR spectrum of Imp-K;

(K-3) the HRMS spectrum of Imp-K;

(L-1) the ^{1}H NMR spectrum of Imp-L;

(L-2) the ^{13}C NMR spectrum of Imp-L;

(L-3) the DEPT spectrum of Imp-L;

(L-4) the IR spectrum of Imp-L;

(L-5) the HRMS spectrum of Imp-L;

(M-1) the ^{1}H NMR spectrum of Imp-M;

(M-2) the ^{13}C NMR spectrum of Imp-M;

(M-3) the DEPT spectrum of Imp-M;

(M-4) the COSY spectrum of Imp-M;

(M-5) the HSQC spectrum of Imp-M;

(M-6) the HMBC spectrum of Imp-M;

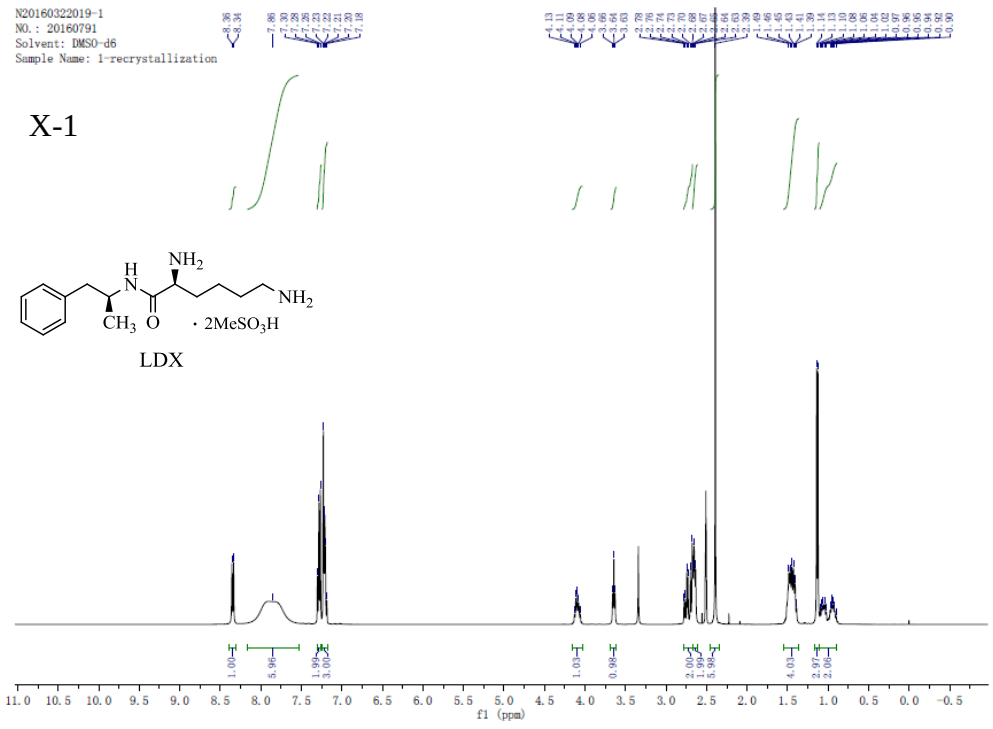
(M-7) the enlarged view of the HMBC spectrum of Imp-M;

(M-8) the enlarged view of the HMBC spectrum of Imp-M;

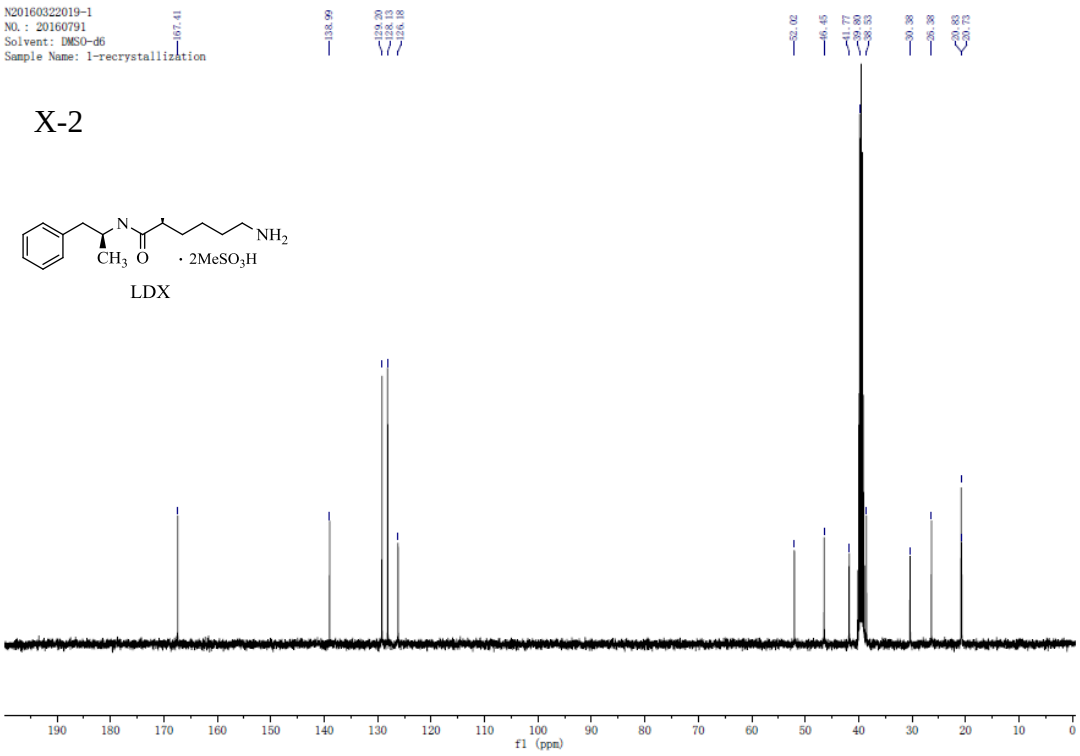
(M-9) the HRMS spectrum of Imp-M;

(N-1) the ^{1}H NMR spectrum of B-3;

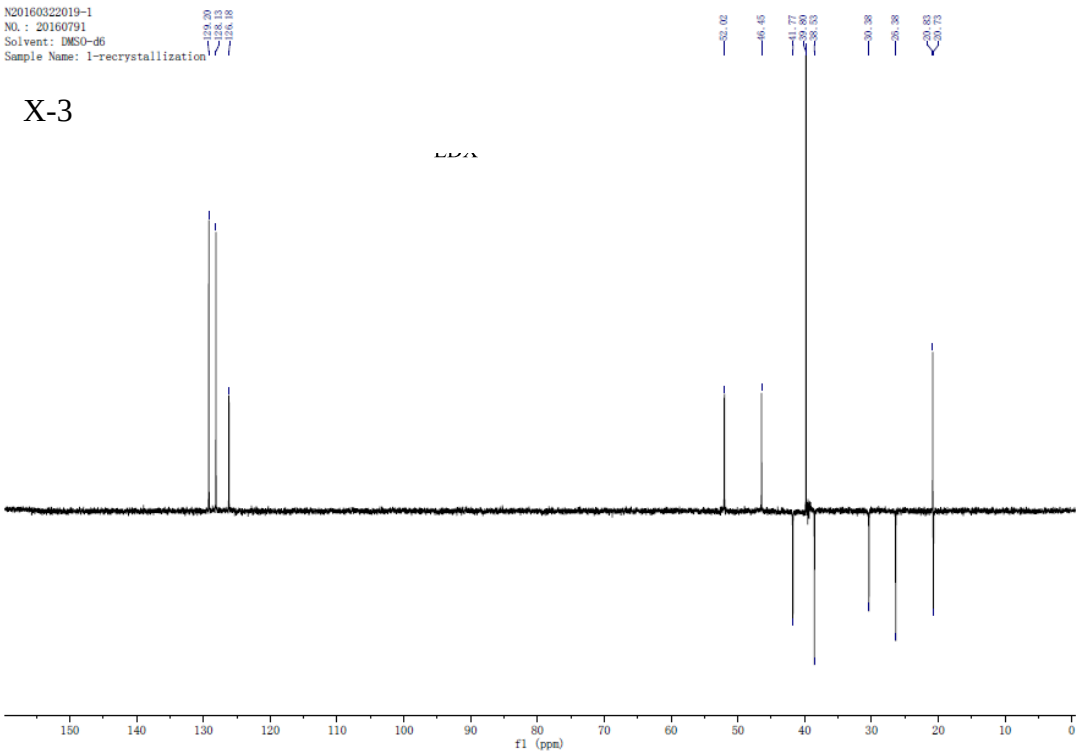
(N-2) the ^{1}H NMR spectrum of B-2;



LDX ^{1}H NMR DMSO 400 MHz

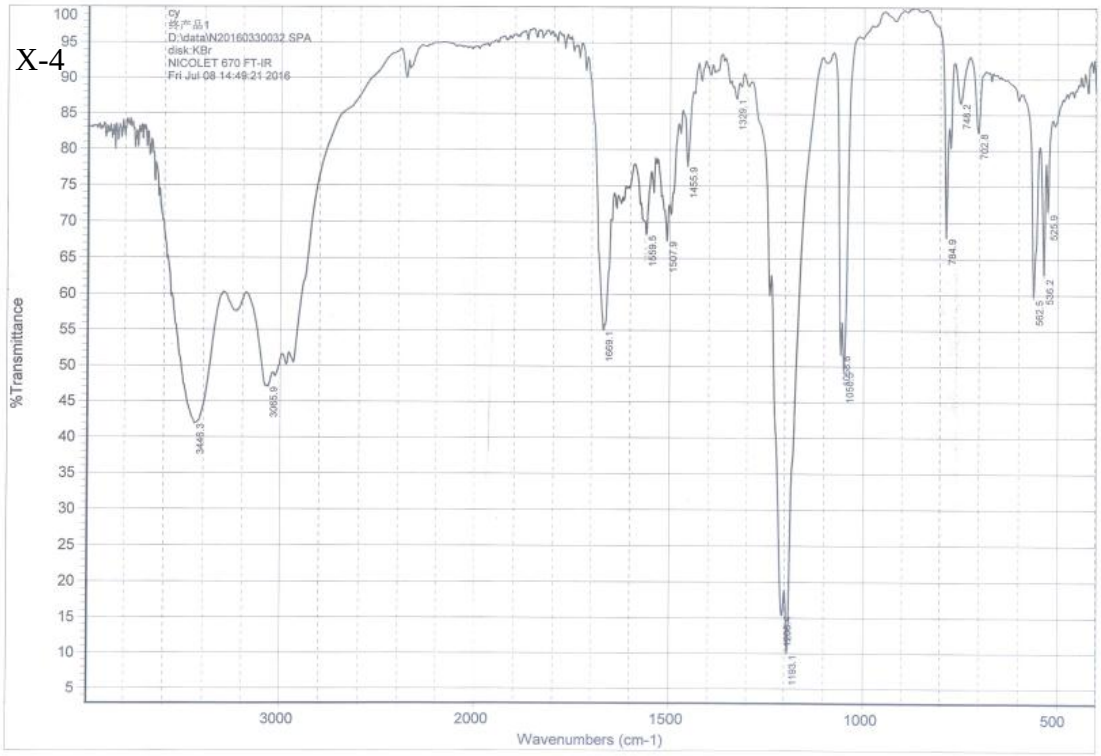


LDX ^{13}C NMR DMSO 101 MHz



LDX DEPT DMSO 101 MHz

X-4



LDX IR

X-5

Qualitative Compound Report

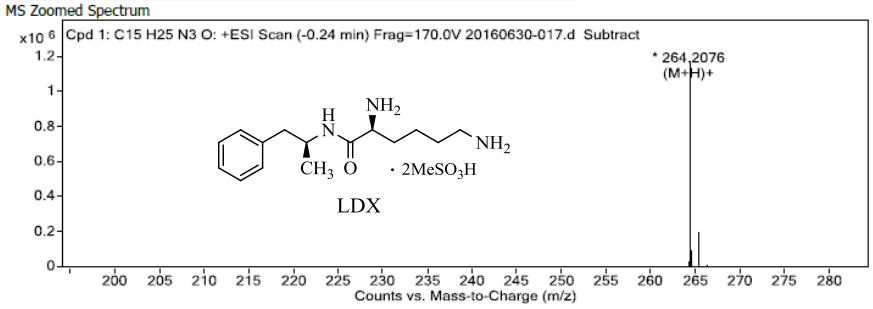
Data File	20160630-017.d	**Sample Name**	LYBBA-1
Sample Type	Sample	**Position**	Vial 50
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160530015

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C15 H25 N3 O	-0.24	263.2004	1178593	C15 H25 N3 O	263.1998	2.3

Compound Label	RT	Algorithm	Mass
Cpd 1: C15 H25 N3 O	-0.24	Find By Formula	263.2004

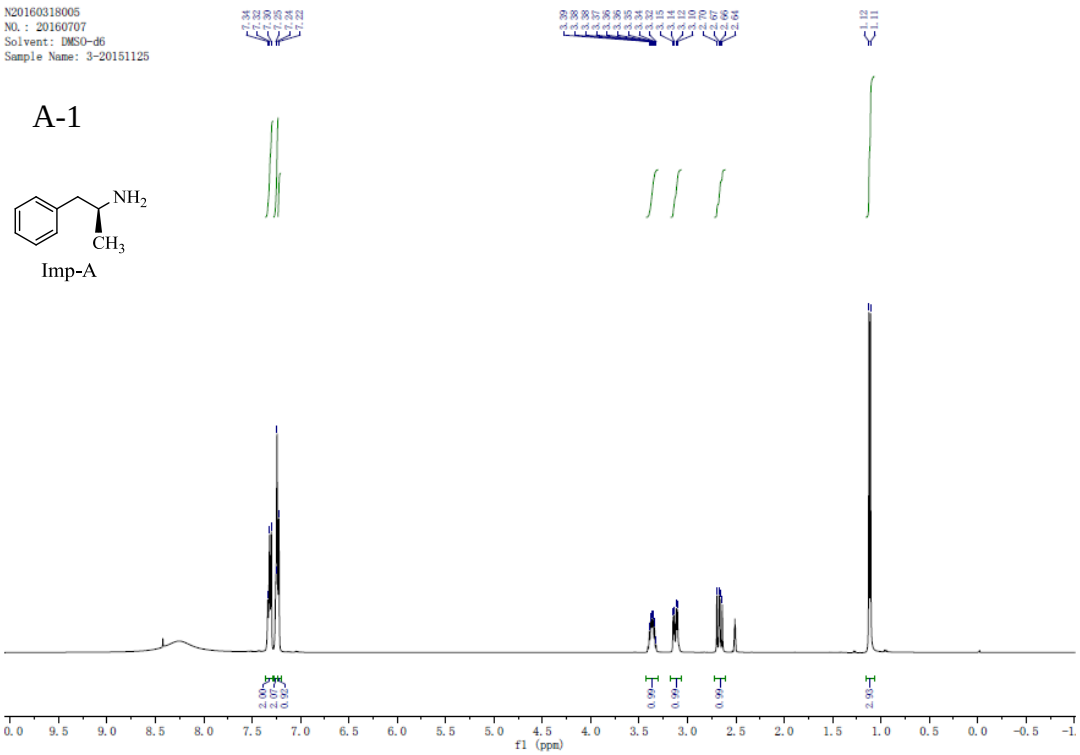
MS Zoomed Spectrum



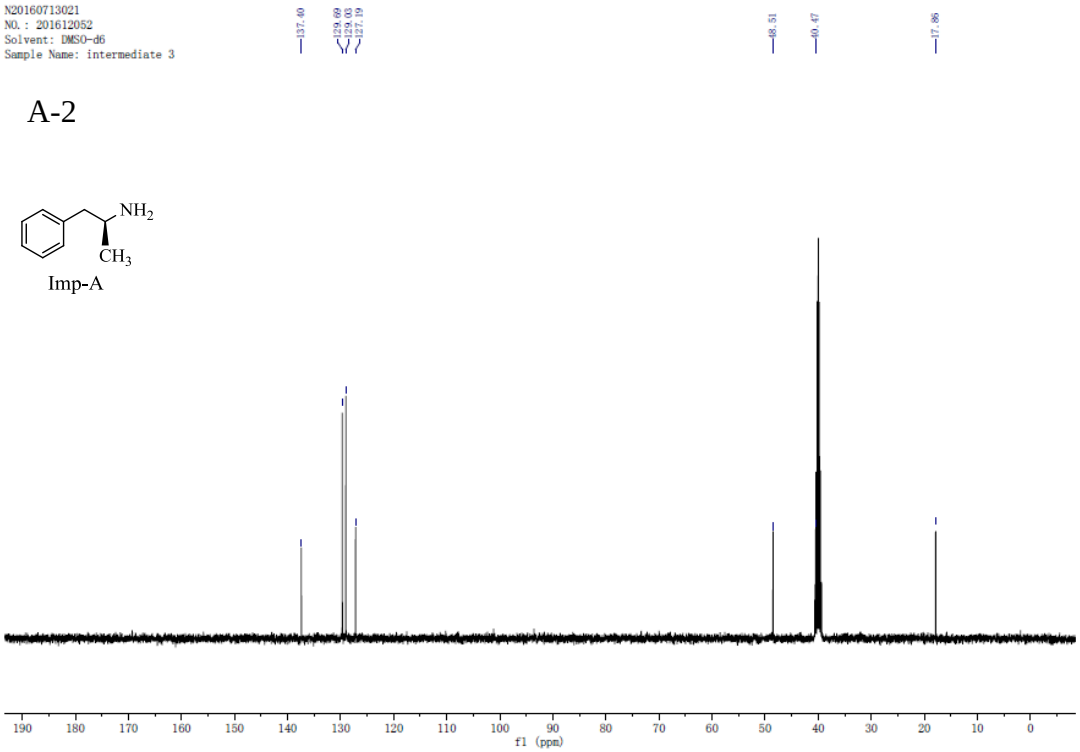
LDX

MS Spectrum Peak List

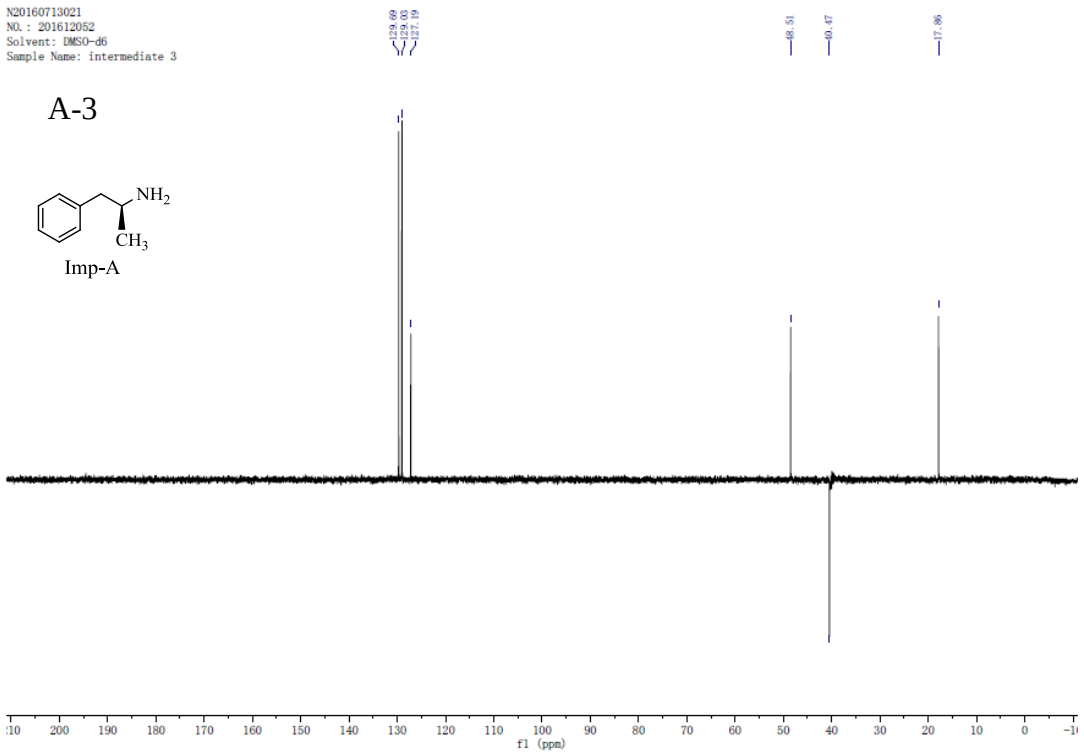
m/z	*Calc m/z*	Diff(ppm)	Abund	Formula	Ion
264.2076	264.207	2.3	1178593	C15 H26 N3 O	(M+H)+



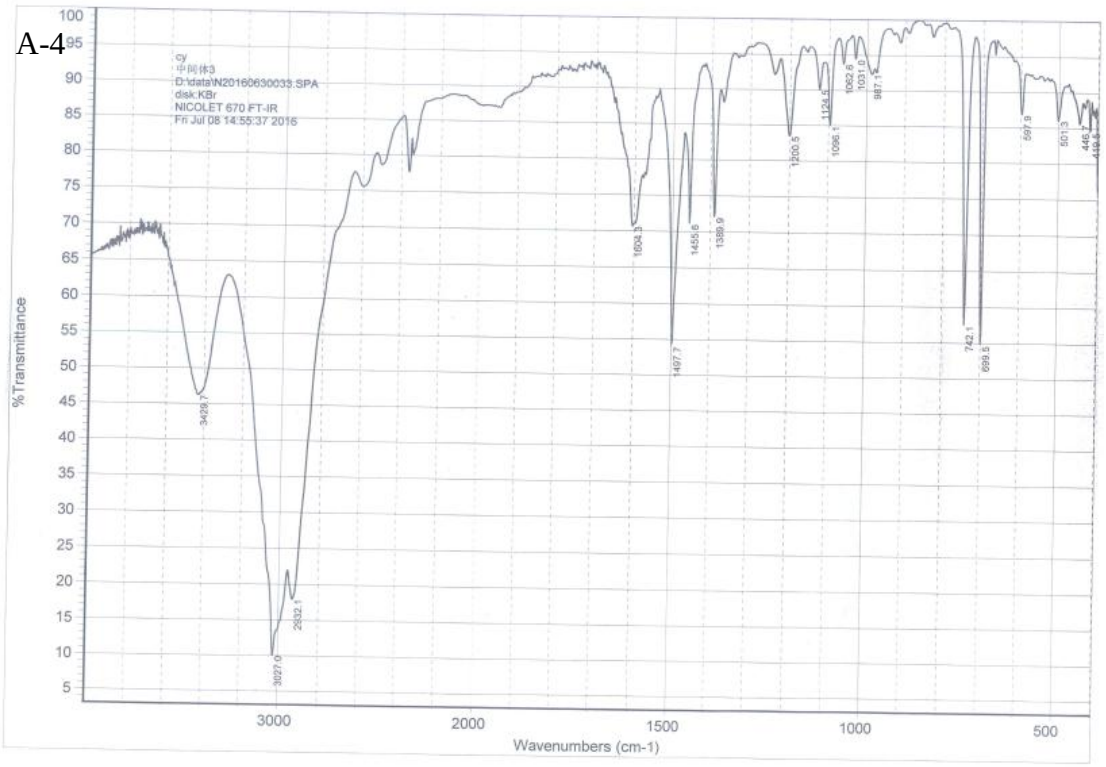
Imp-A ^{1}H NMR DMSO 400 MHz



Imp-A ^{13}C NMR DMSO 101 MHz



Imp-A DEPT DMSO 101 MHz



Imp-A IR

A-5

Qualitative Compound Report

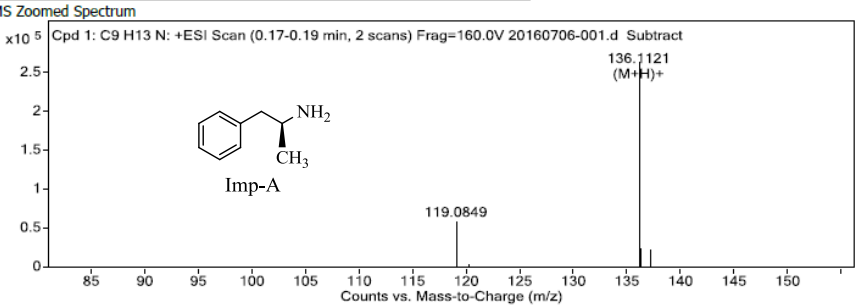
Data File	20160706-001.d	**Sample Name**	Int-3
Sample Type	Sample	**Position**	Vial 76
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160706003

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C9 H13 N	0.17	135.1048	264278	C9 H13 N	135.1048	-0.11

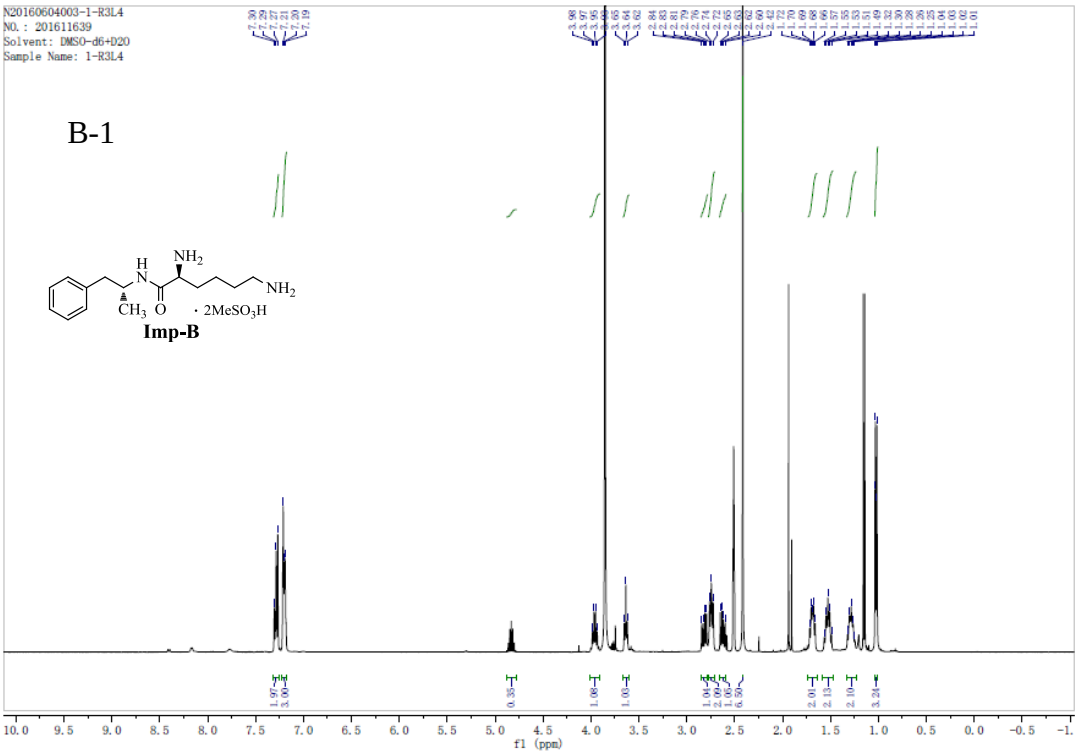
Compound Label	RT	Algorithm	Mass
Cpd 1: C9 H13 N	0.17	Find By Formula	135.1048

MS Zoomed Spectrum

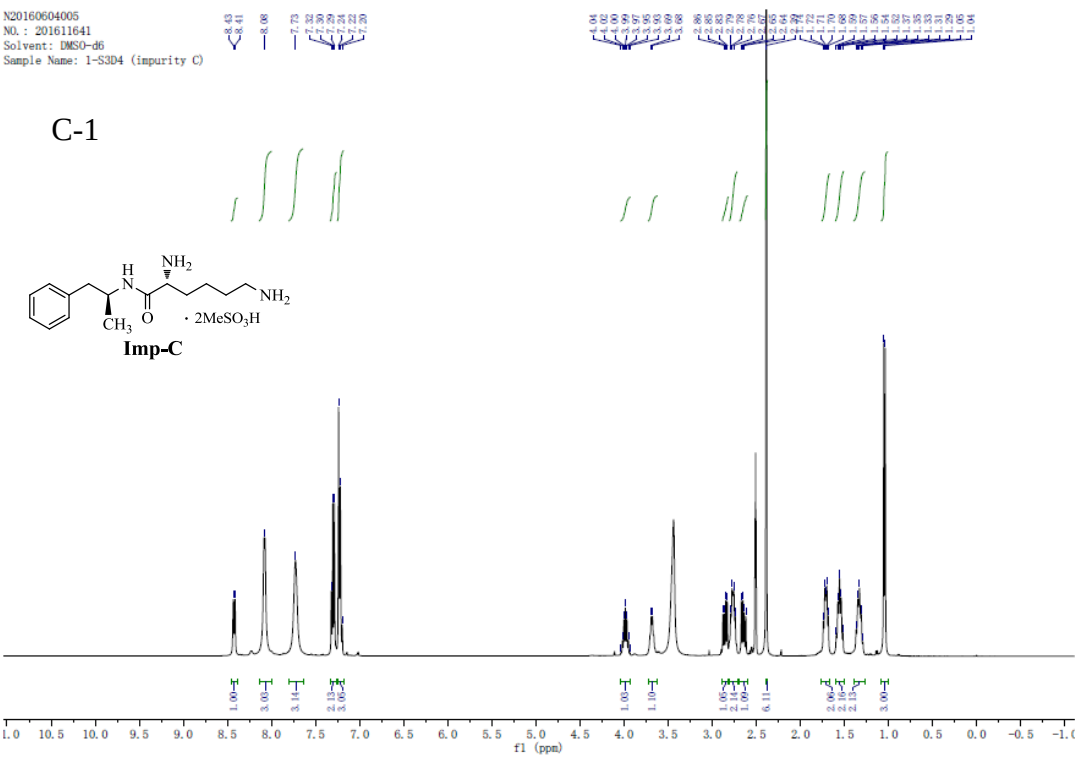


MS Spectrum Peak List

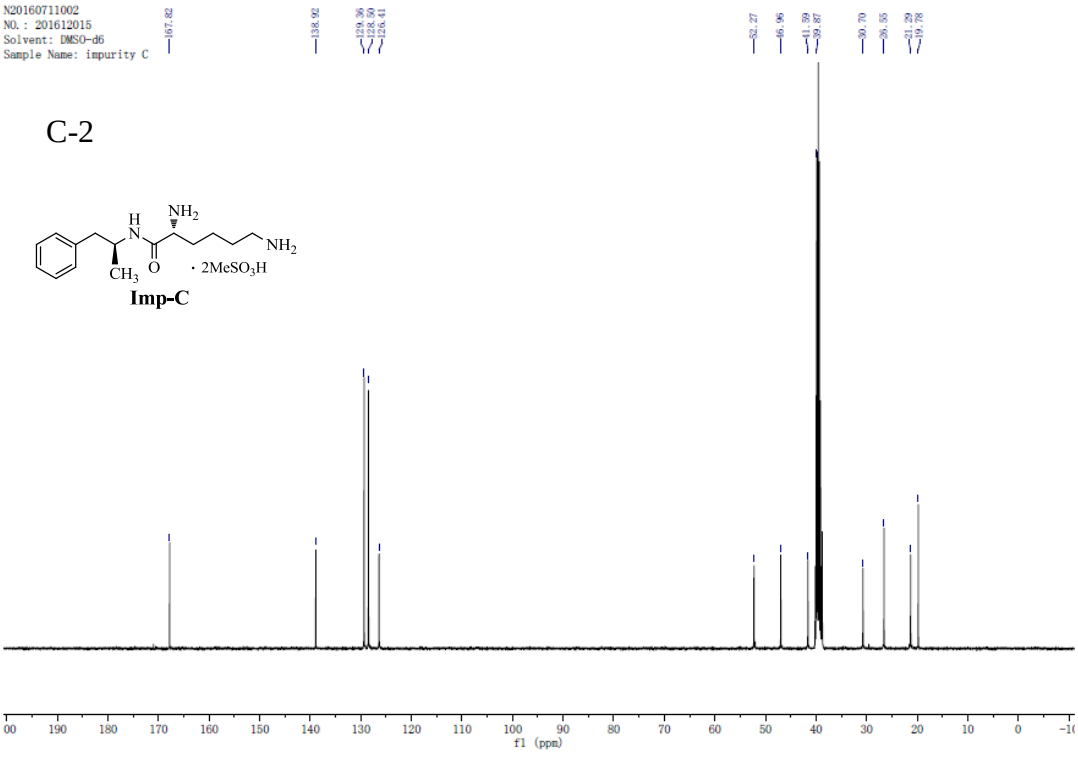
m/z	*Calc m/z*	Diff(ppm)	z	Abund	Formula	Ion
136.1121	136.1121	-0.11	1	264278	C9 H14 N	(M+H)+



Imp-B ^{1}H NMR DMSO 400 MHz

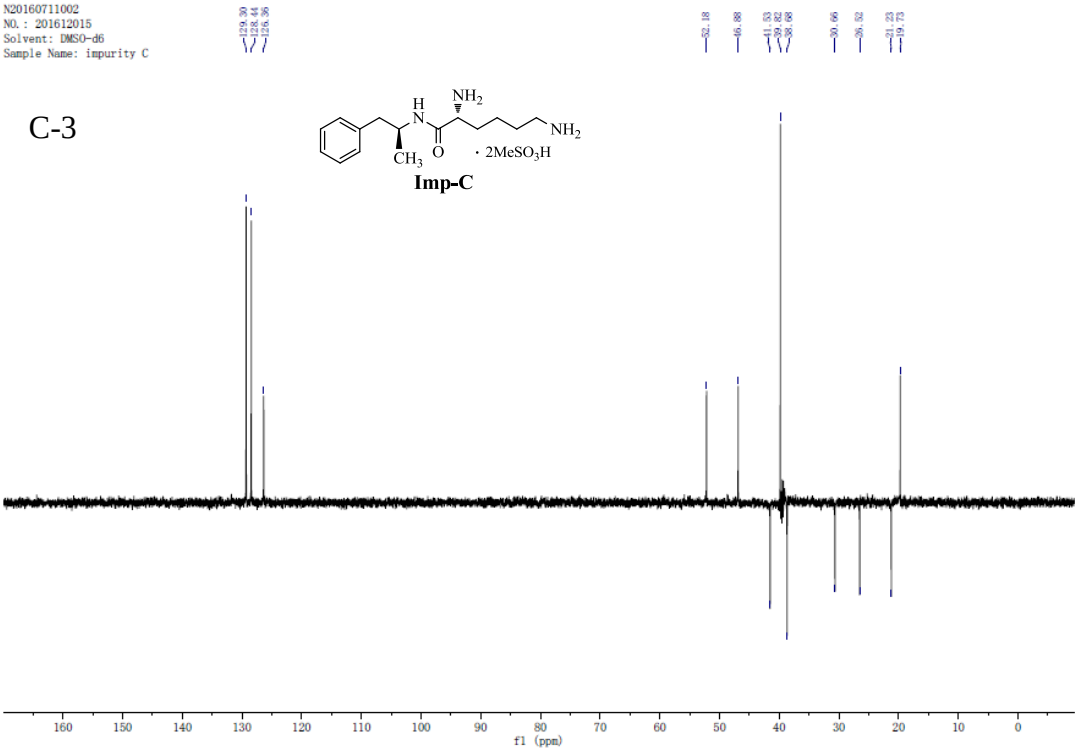


Imp-C ^{1}H NMR DMSO 400 MHz



Imp-C ^{13}C NMR DMSO 101 MHz

C-3



Imp-C DEPT DMSO 101 MHz

C-4

Qualitative Compound Report

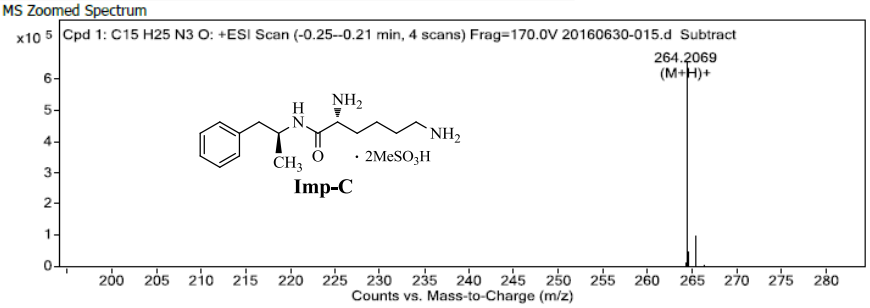
Data File	20160630-015.d	**Sample Name**	Impurity C
Sample Type	Sample	**Position**	Vial 49
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160630031

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C15 H25 N3 O	-0.25	263.1997	660907	C15 H25 N3 O	263.1998	-0.37

Compound Label	RT	Algorithm	Mass
Cpd 1: C15 H25 N3 O	-0.25	Find By Formula	263.1997

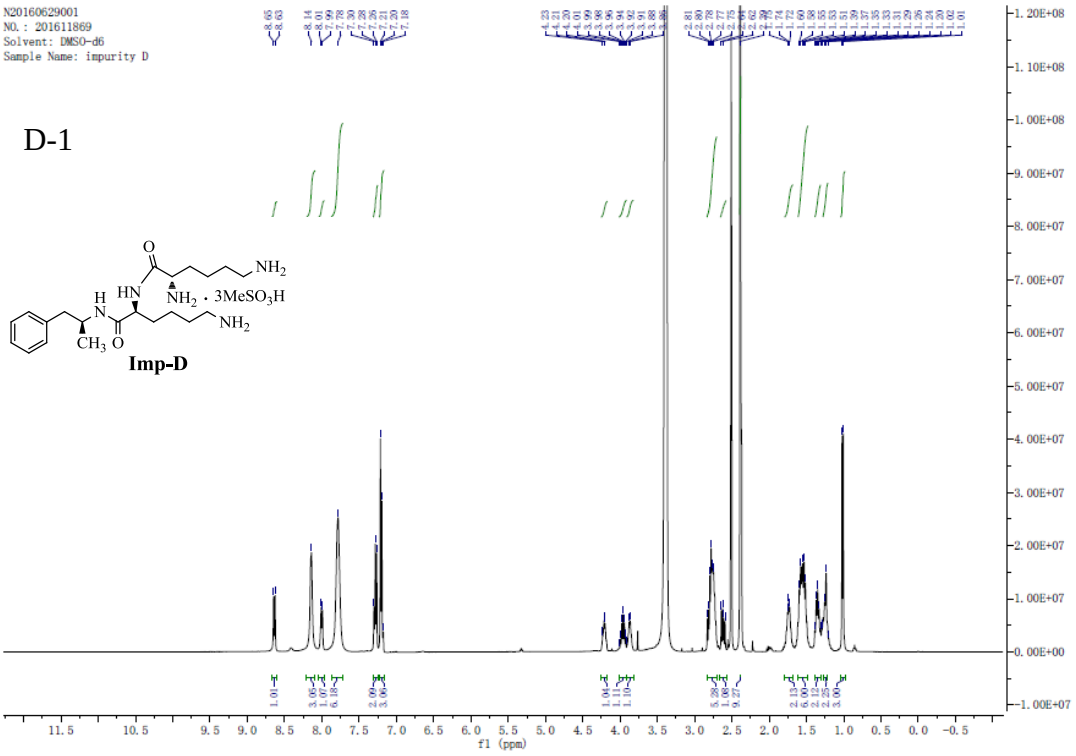
MS Zoomed Spectrum



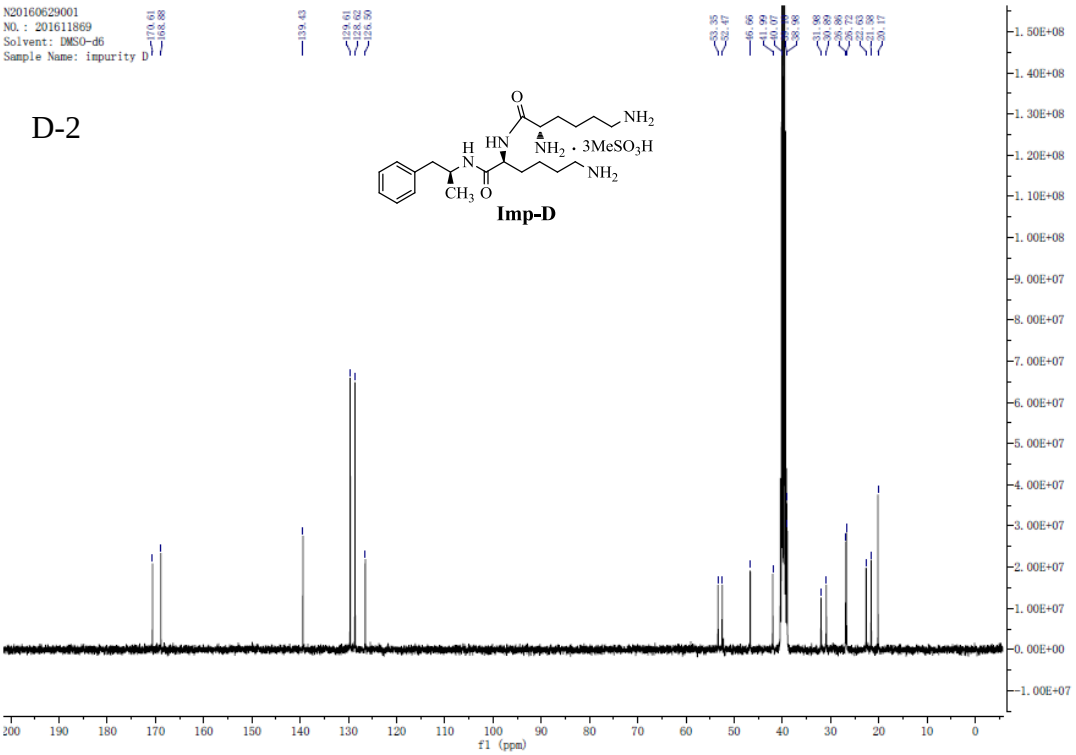
MS Spectrum Peak List

m/z	*Calc m/z*	Diff(ppm)	Abund	Formula	Ion
264.2069	264.207	-0.36	660907	C15 H26 N3 O	(M+H)+

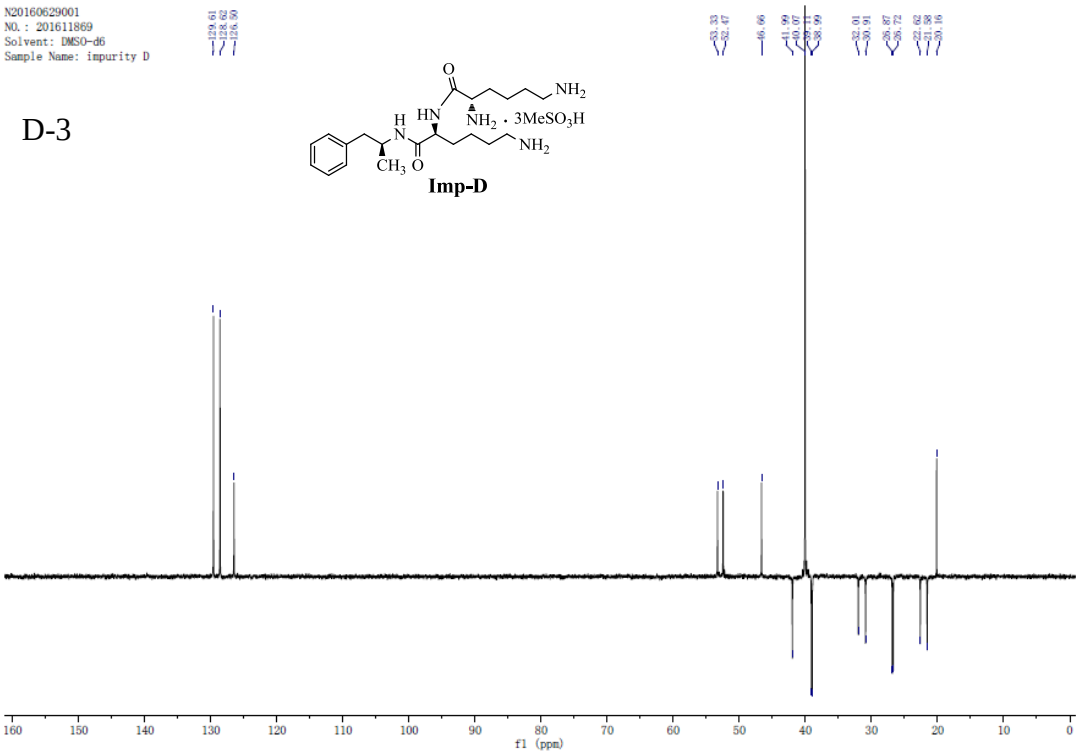
--- End Of Report ---



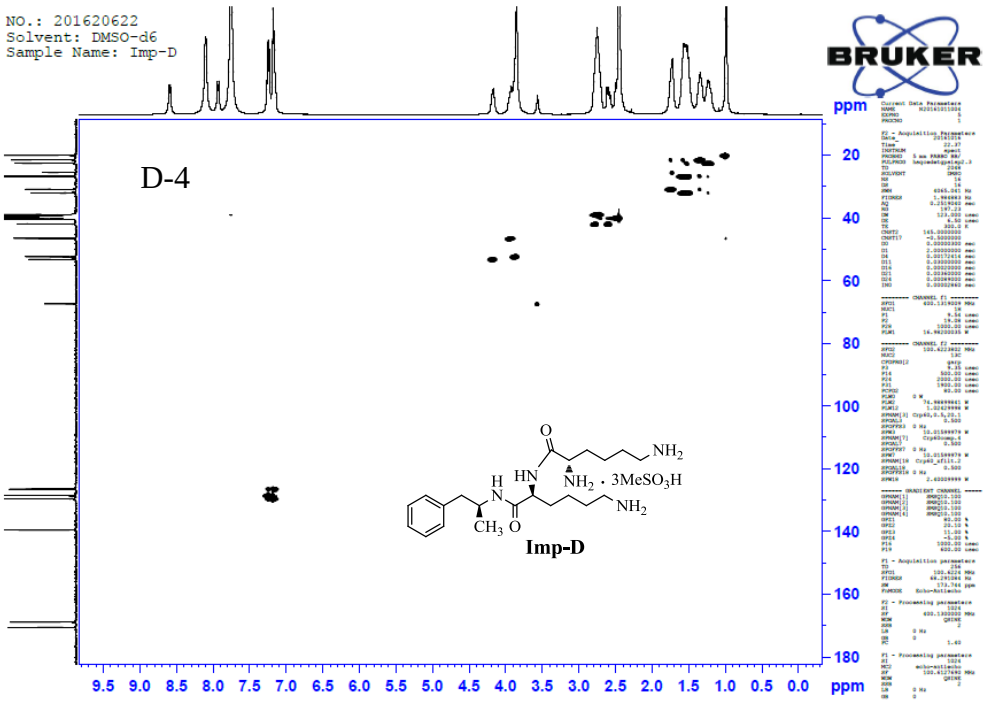
Imp-D ^{1}H NMR DMSO 400 MHz



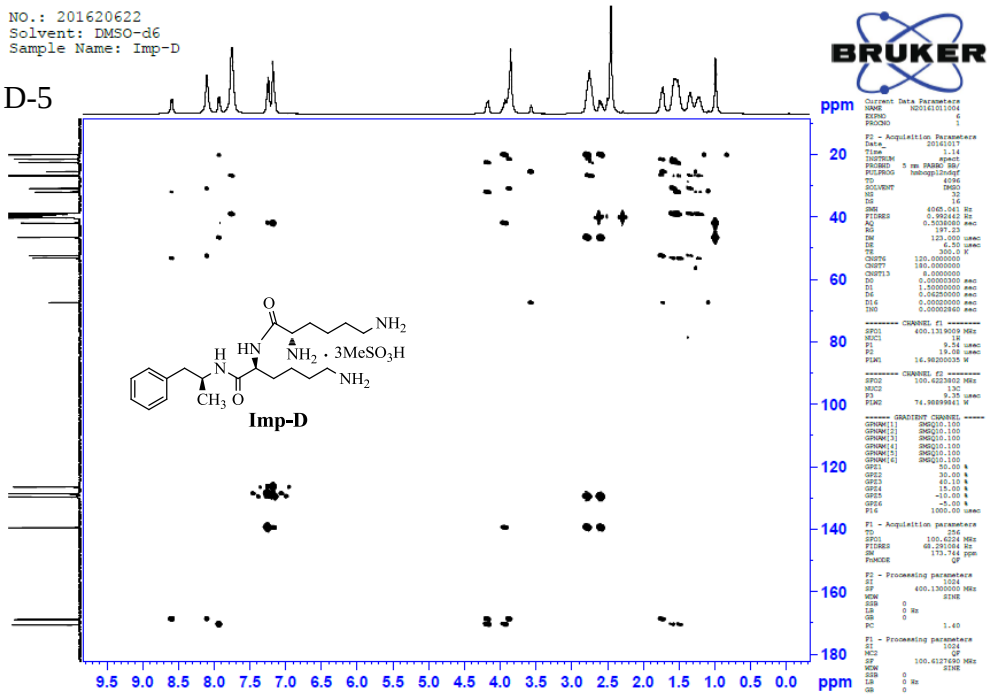
Imp-D ^{13}C NMR DMSO 101 MHz



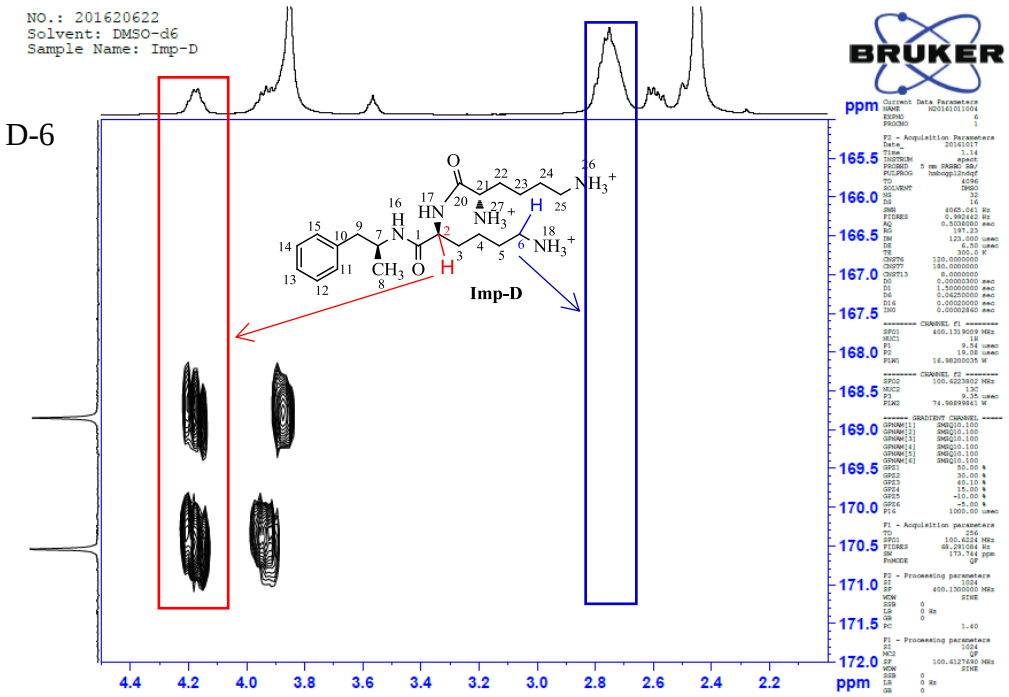
Imp-D DEPT DMSO 101 MHz



HSQC Imp-D DMSO

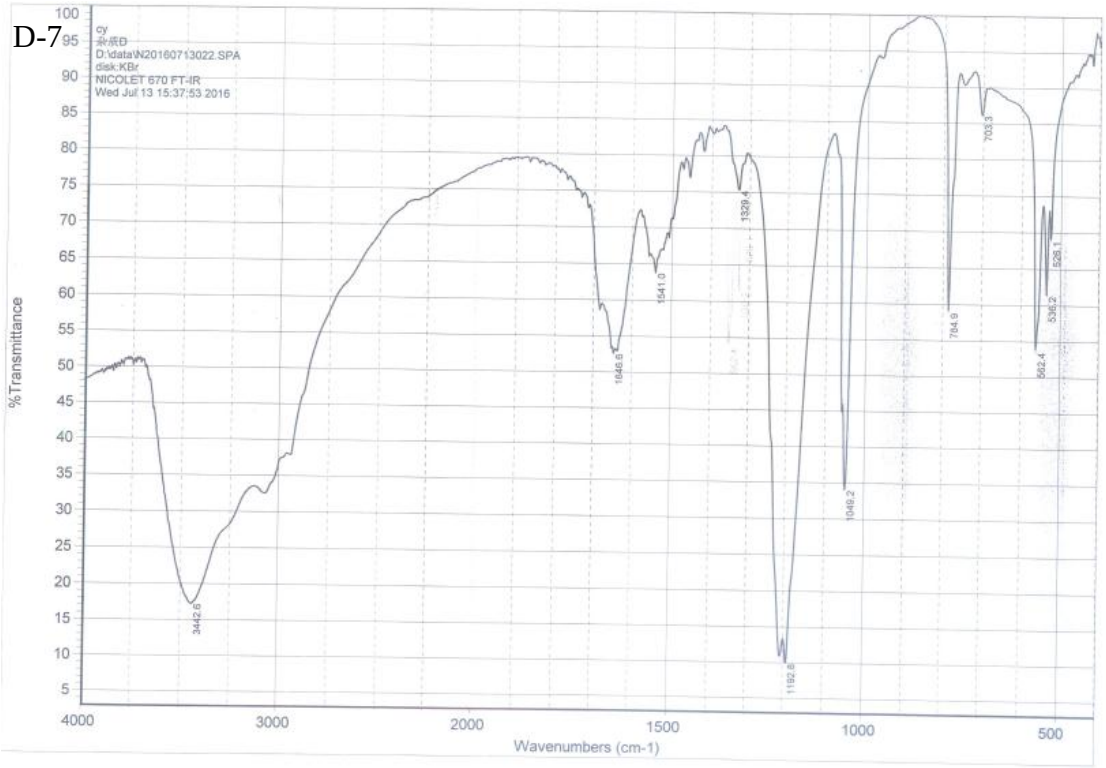


HMBC Imp-D DMSO



The the enlarged view of the HMBC spectrum of Imp-D

D-7



Imp-D IR

D-8

Qualitative Compound Report

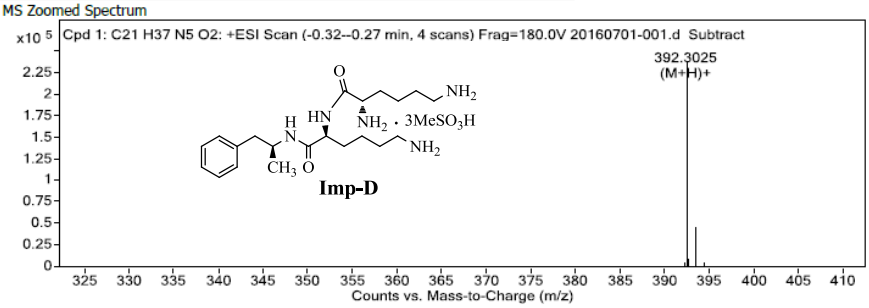
Data File	20160701-001.d	**Sample Name**	Impurity D
Sample Type	Sample	**Position**	Vial 52
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160627018

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C21 H37 N5 O2	-0.32	391.2952	239384	C21 H37 N5 O2	391.2947	1.31

Compound Label	RT	Algorithm	Mass
Cpd 1: C21 H37 N5 O2	-0.32	Find By Formula	391.2952

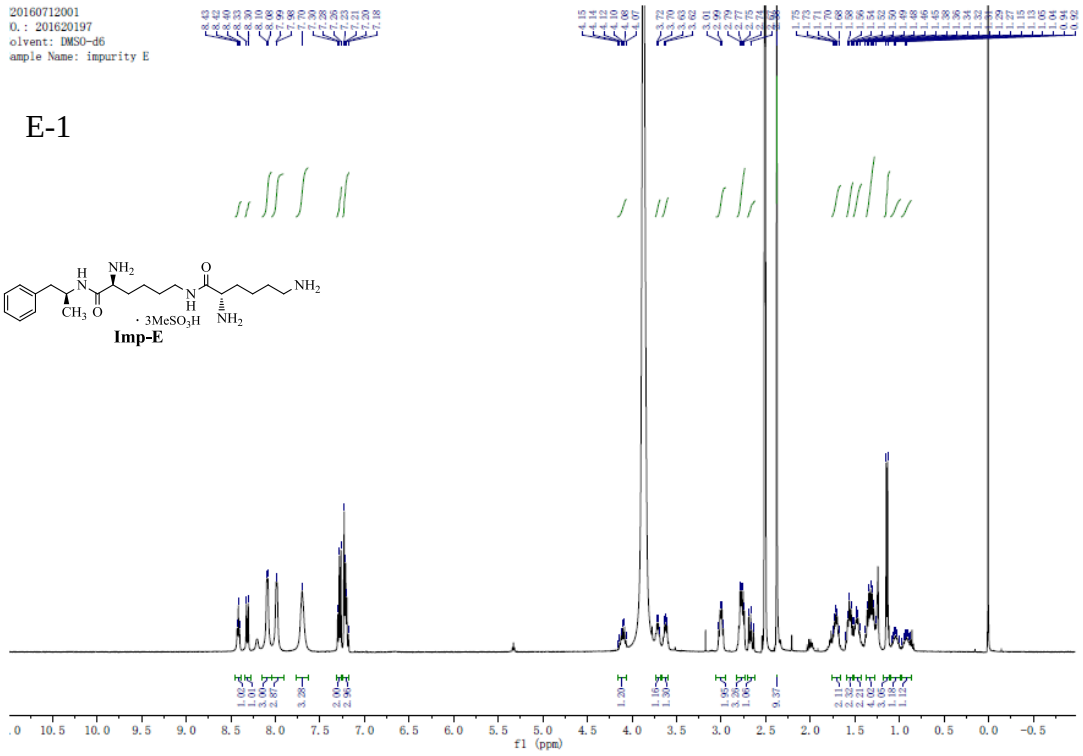
MS Zoomed Spectrum



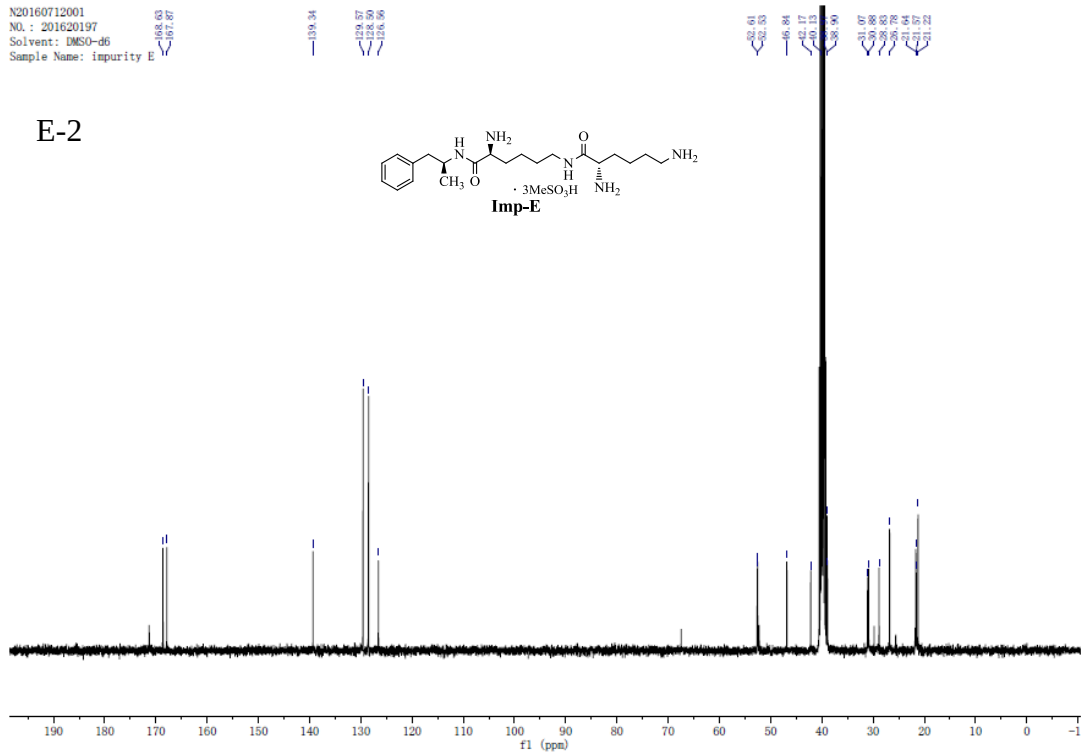
MS Spectrum Peak List

m/z	*Calc m/z*	Diff(ppm)	Abund	Formula	Ion
392.3025	392.302	1.32	239384	C21 H38 N5 O2	(M+H)+

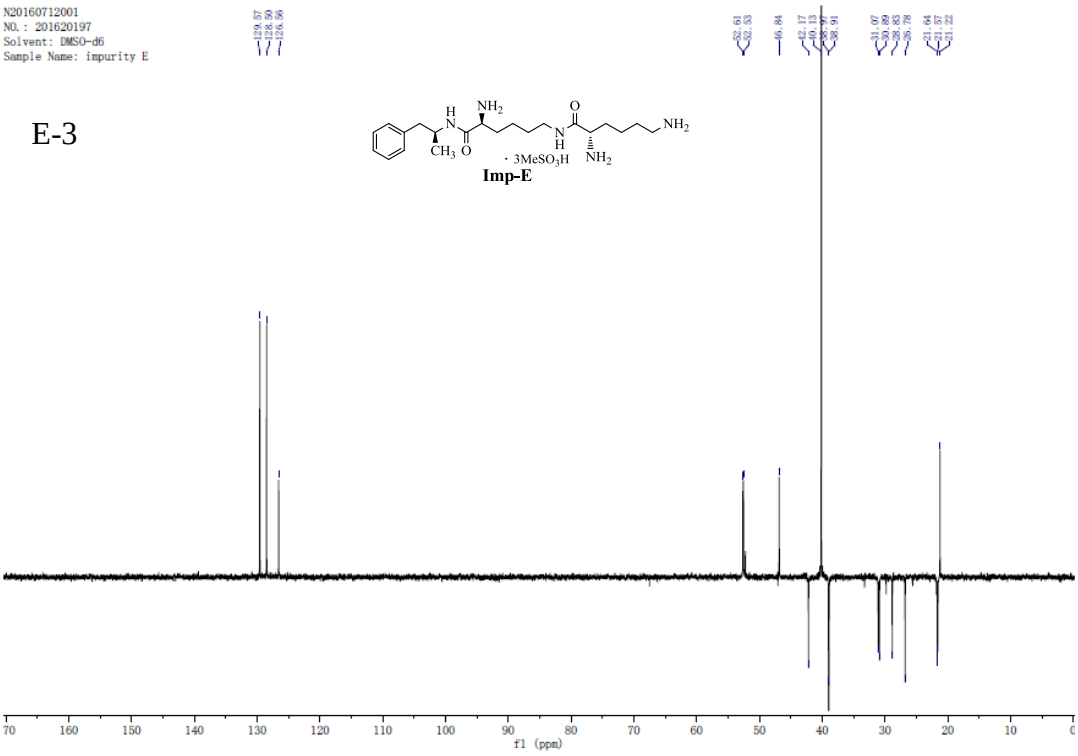
--- End Of Report ---



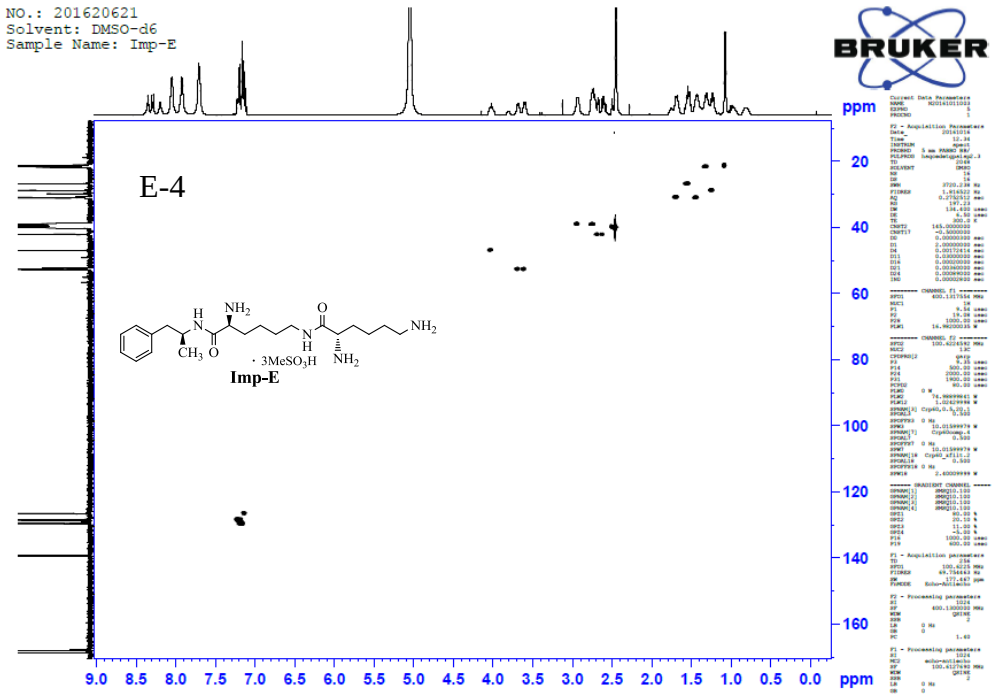
Imp-E ^{1}H NMR DMSO 400 MHz



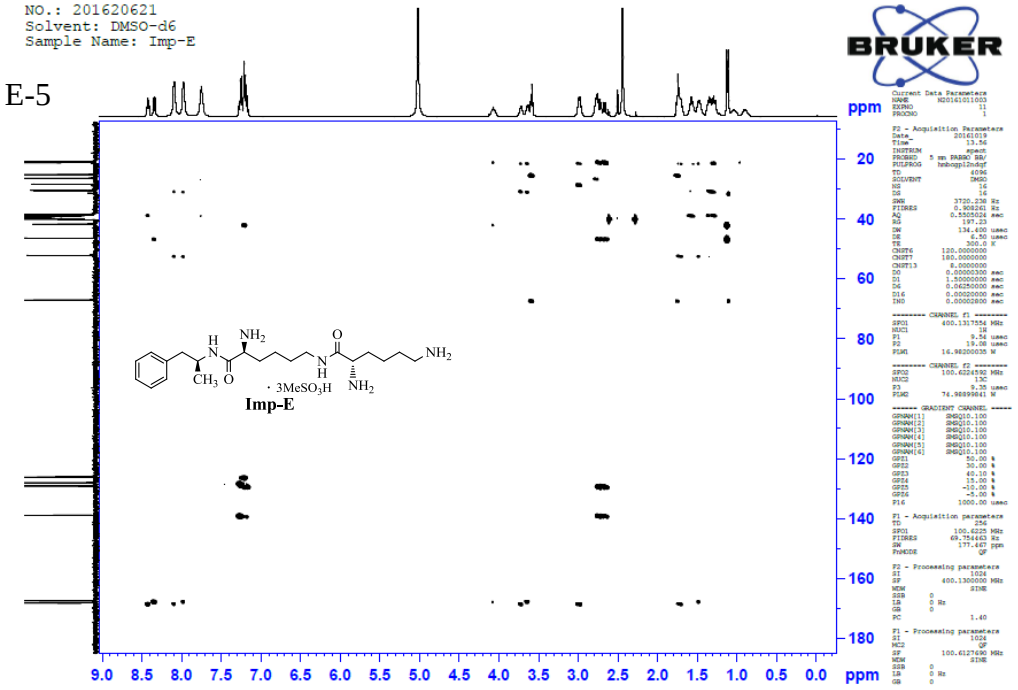
Imp-E ^{13}C NMR DMSO 101 MHz



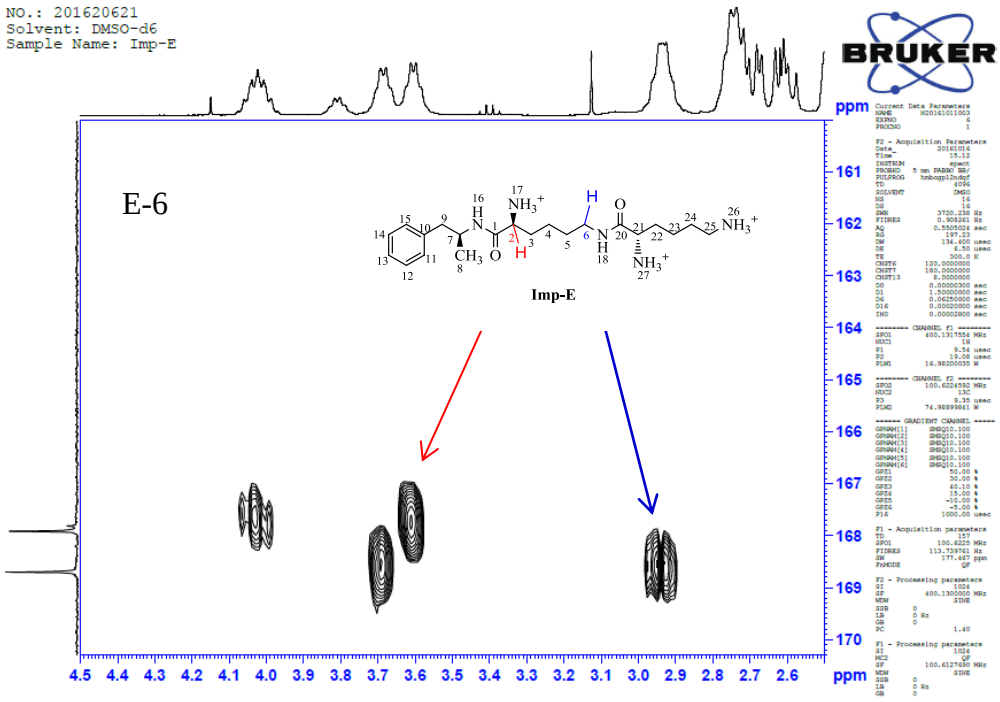
Imp-E DEPT DMSO 101 MHz



HSQC Imp-E DMSO

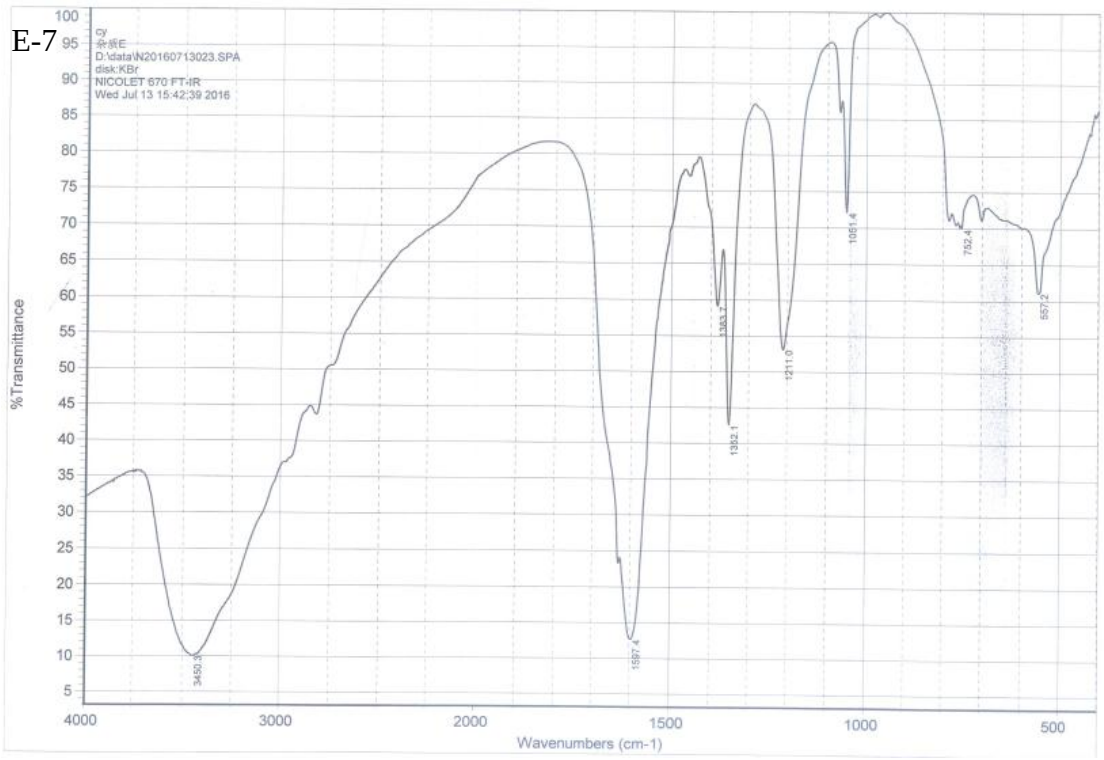


HMBC Imp-E DMSO



The the enlarged view of the HMBC spectrum of Imp-E

E-7



Imp-E IR

E-8

Qualitative Compound Report

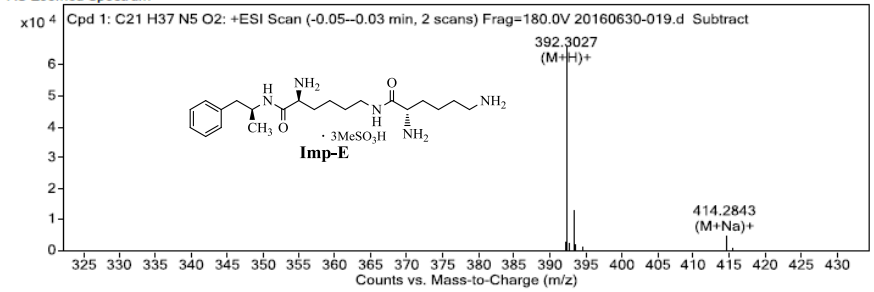
Data File	20160630-019.d	**Sample Name**	Impurity E
Sample Type	Sample	**Position**	Vial 51
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160627019

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C21 H37 N5 O2	-0.05	391.2954	66624	C21 H37 N5 O2	391.2947	1.66

Compound Label	RT	Algorithm	Mass
Cpd 1: C21 H37 N5 O2	-0.05	Find By Formula	391.2954

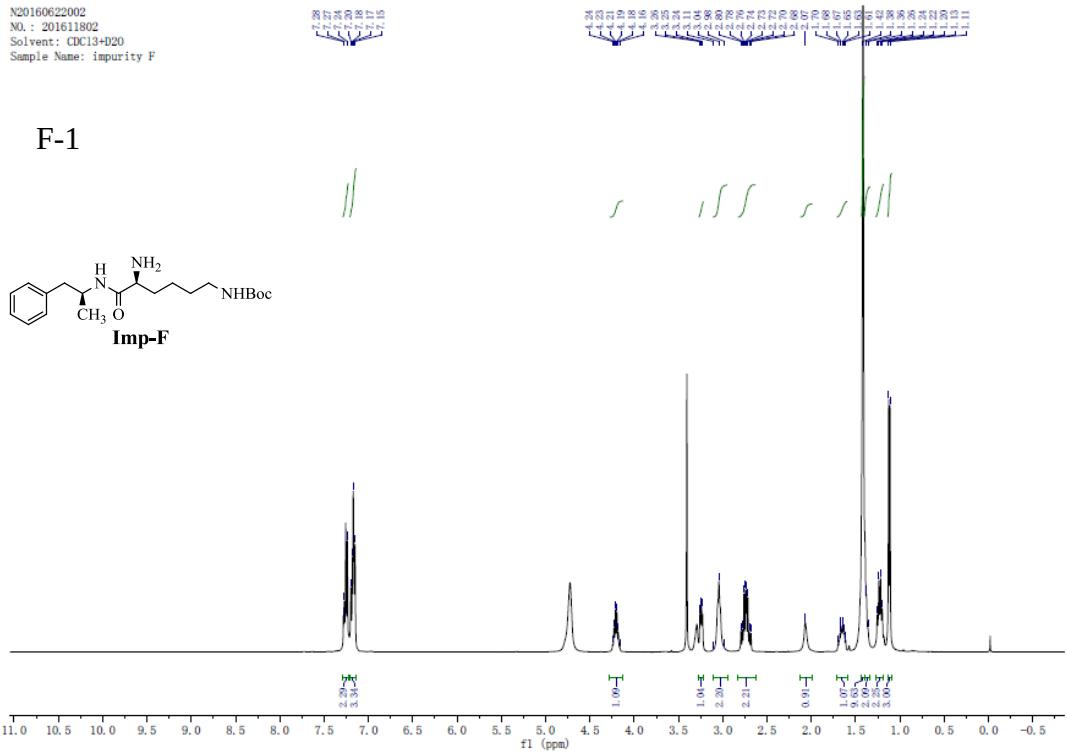
MS Zoomed Spectrum



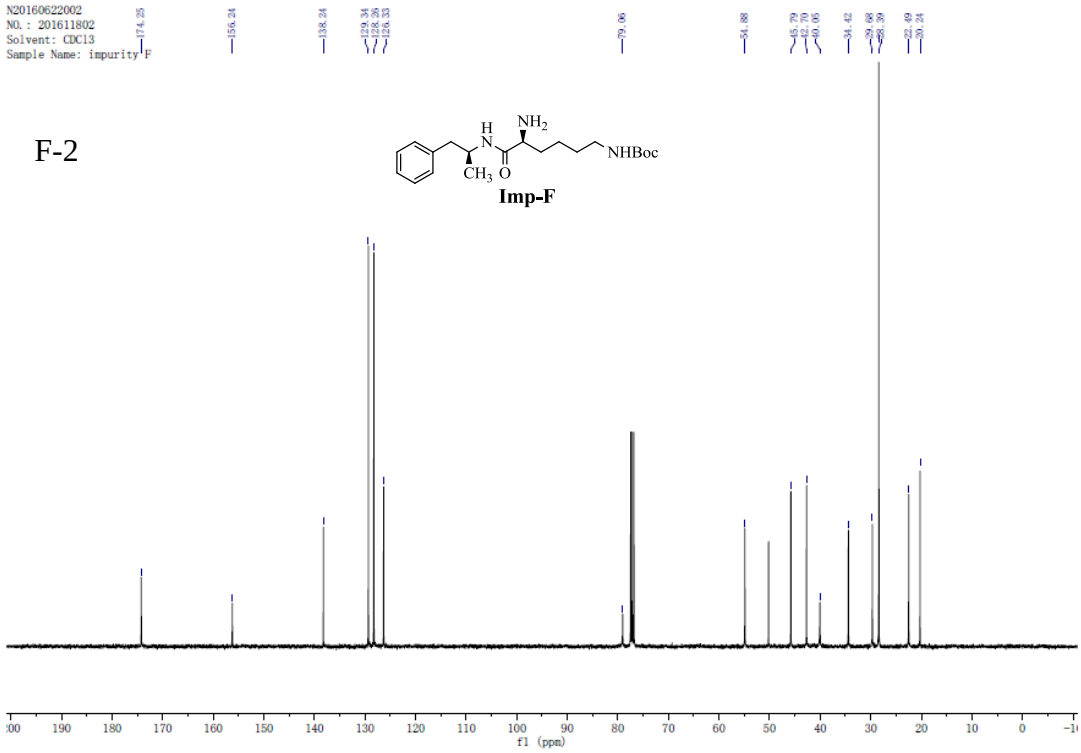
MS Spectrum Peak List

m/z	*Calc m/z*	Diff(ppm)	z	Abund	Formula	Ion
392.3027	392.302	1.66		66624	C21 H38 N5 O2	(M+H)+
414.2843	414.2839	0.82	1	4866	C21 H37 N5 Na O2	(M+Na)+

Imp-E HRMS

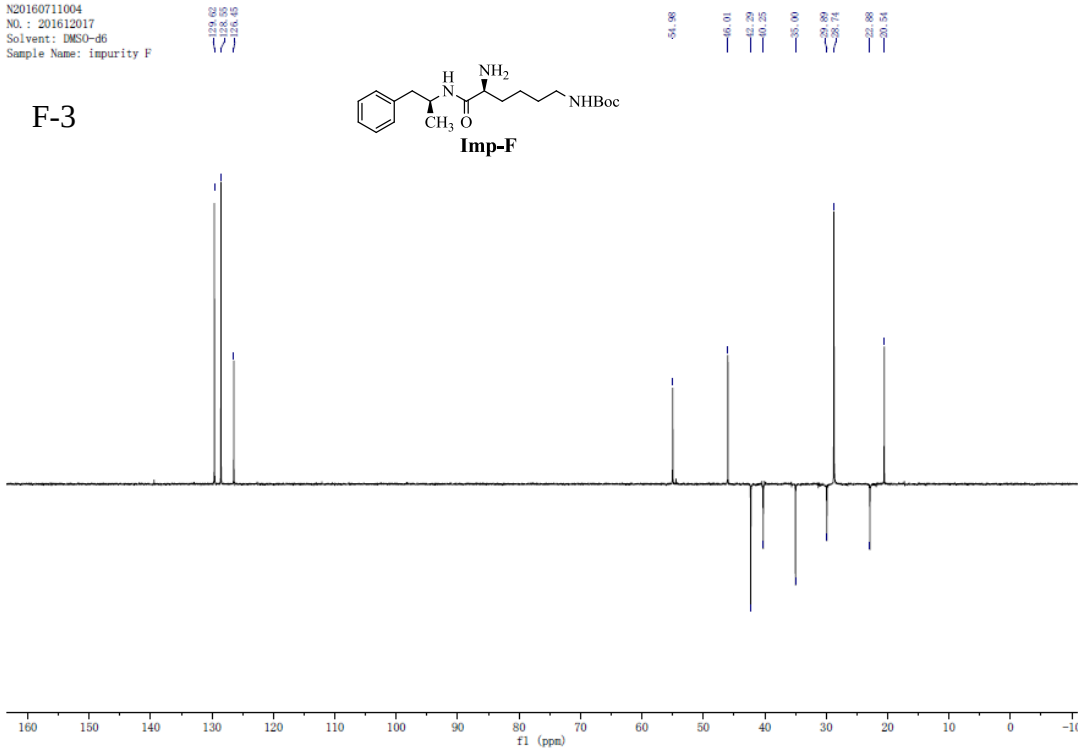


Imp-F ^{1}H NMR $CDCl_3$ 400 MHz



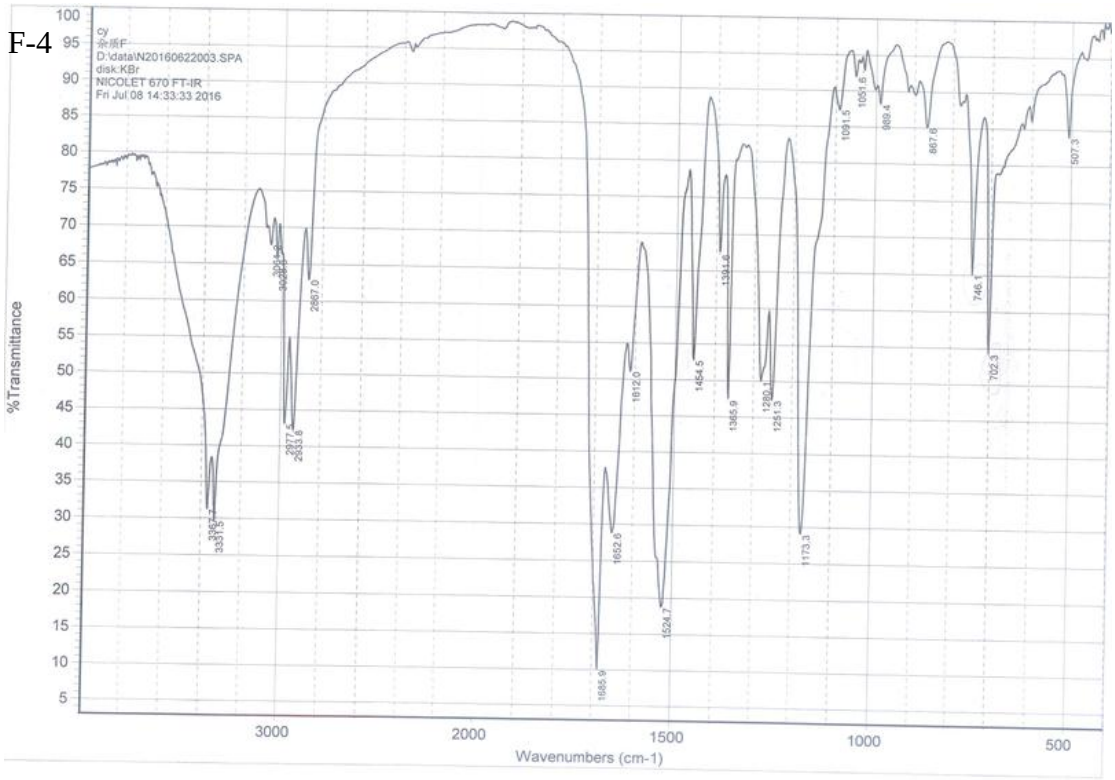
Imp-F ^{13}C NMR $CDCl_3$ 101 MHz

F-3



Imp-F DEPT $CDCl_3$ 101 MHz

F-4



Imp-F IR

F-5

Qualitative Compound Report

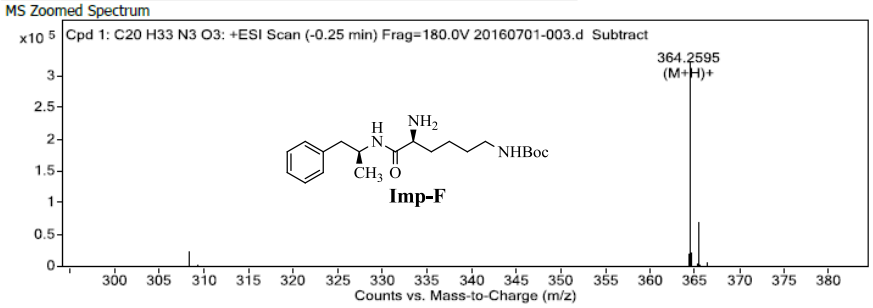
Data File	20160701-003.d	**Sample Name**	Impurity F
Sample Type	Sample	**Position**	Vial 53
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160622003

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C20 H33 N3 O3	-0.25	363.2522	324479	C20 H33 N3 O3	363.2522	-0.03

Compound Label	RT	Algorithm	Mass
Cpd 1: C20 H33 N3 O3	-0.25	Find By Formula	363.2522

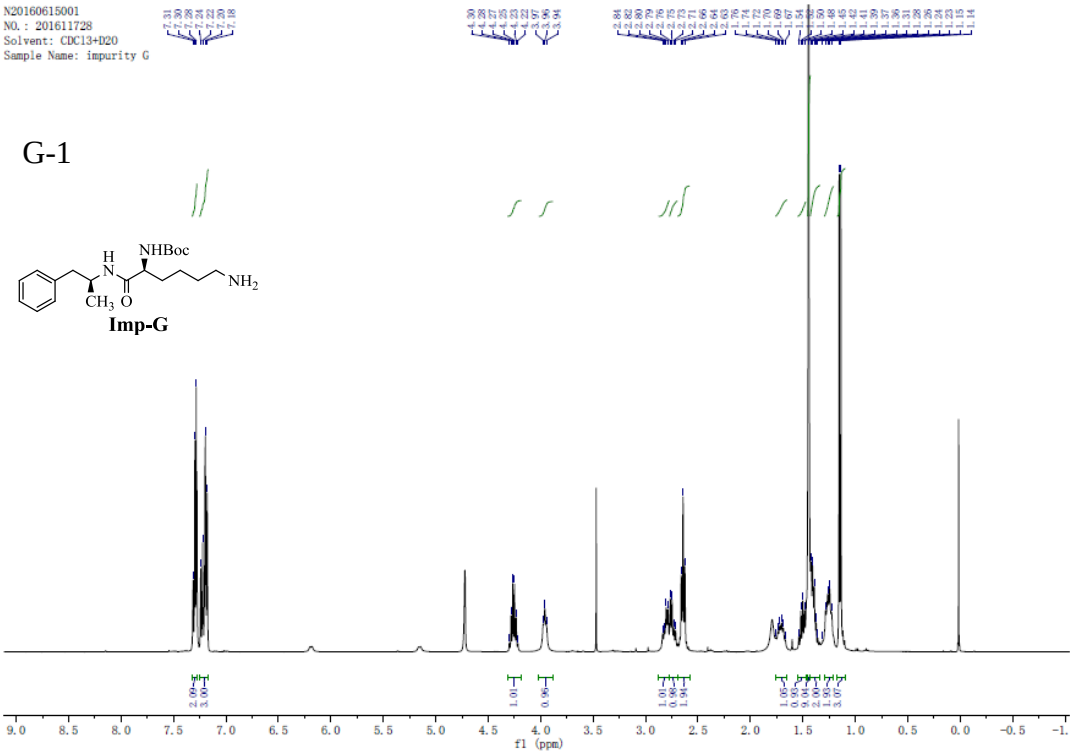
MS Zoomed Spectrum



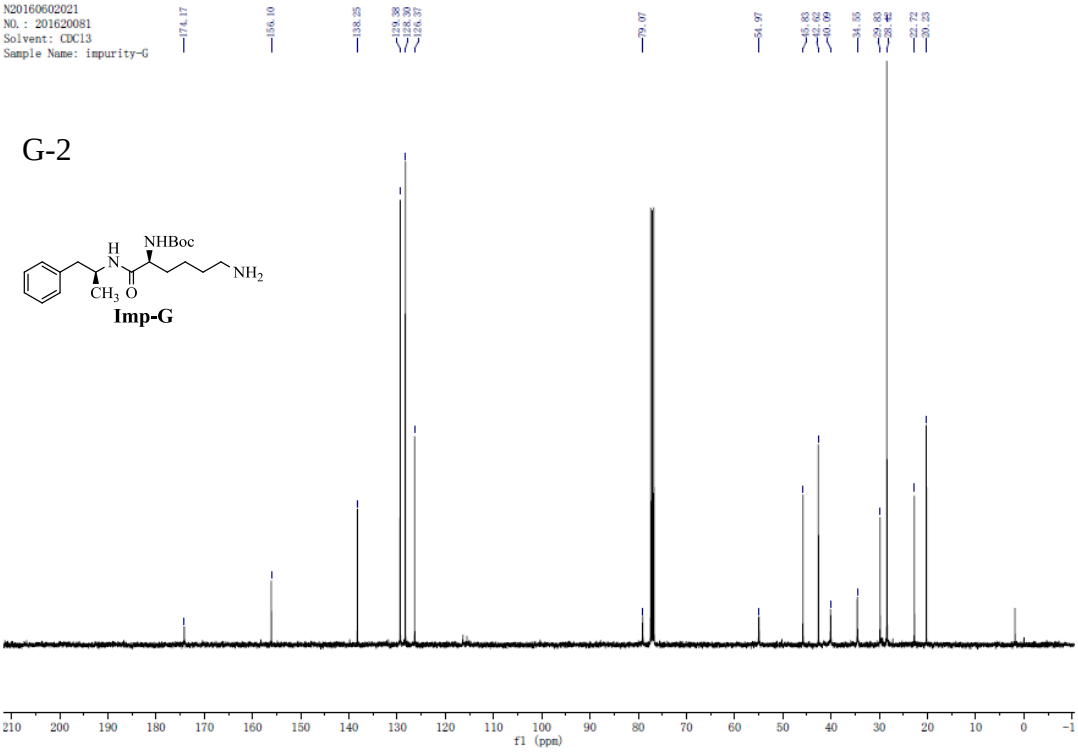
MS Spectrum Peak List

m/z	*Calc m/z*	Diff(ppm)	Abund	Formula	Ion
364.2595	364.2595	-0.01	324479	C20 H34 N3 O3	(M+H)+

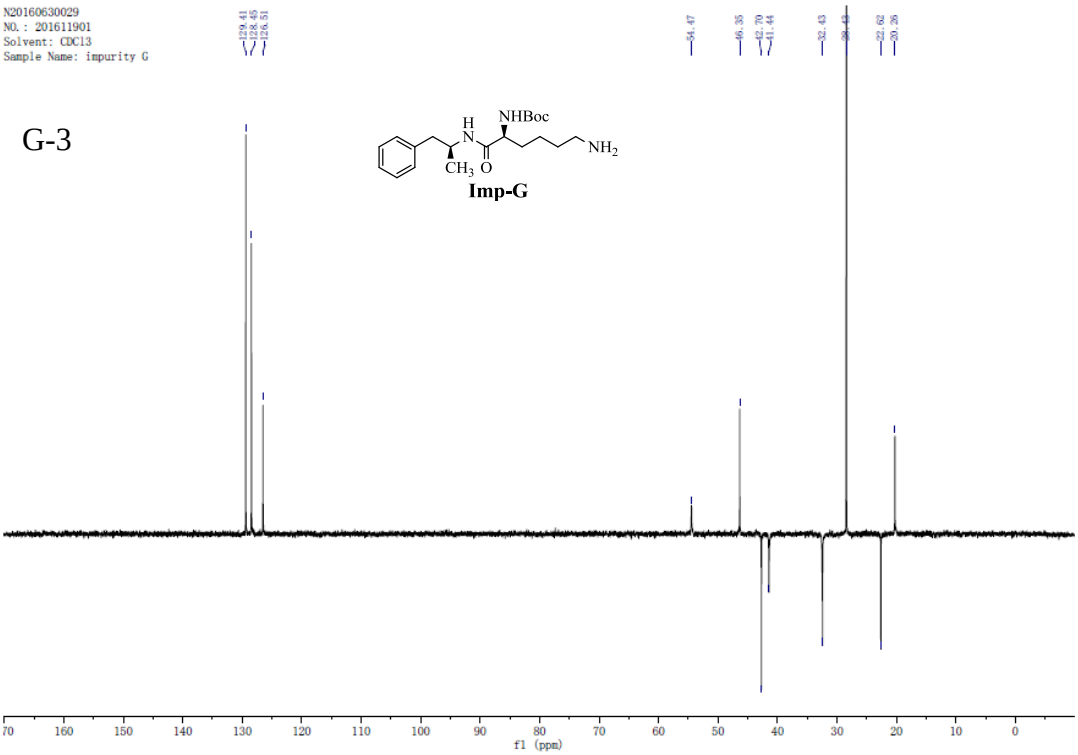
Imp-F HRMS



Imp-G ^{1}H NMR $CDCl_3$ 400 MHz

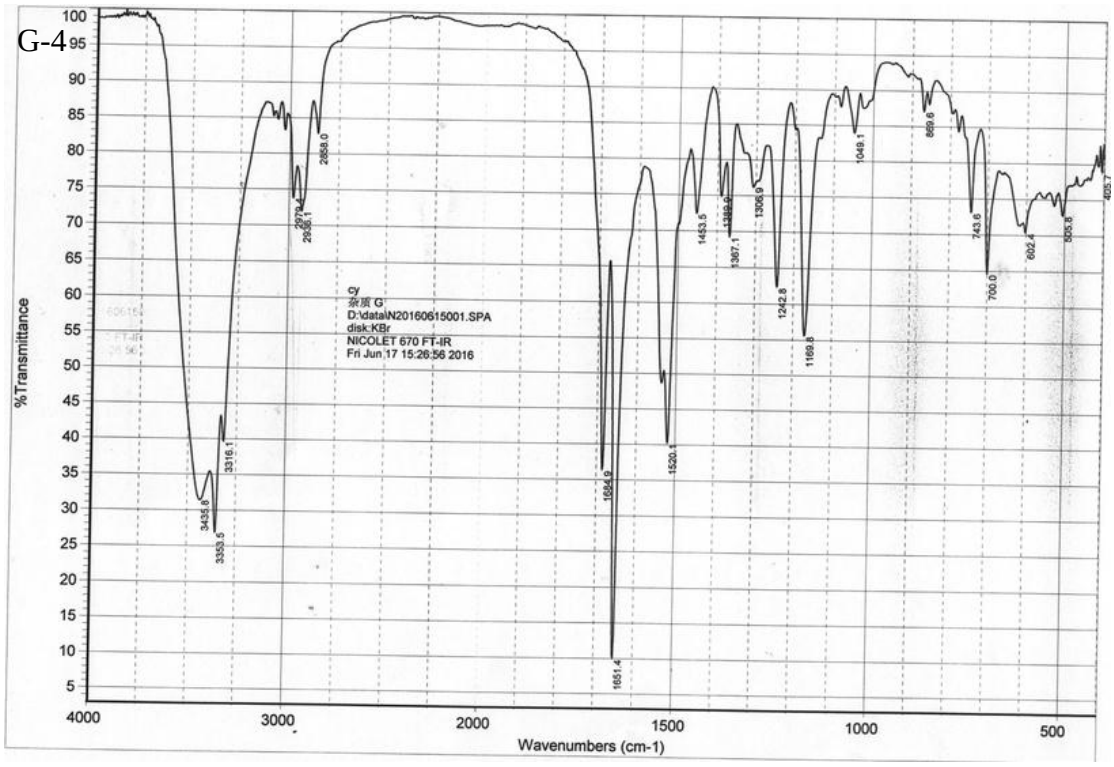


Imp-G ^{13}C NMR $CDCl_3$ 101 MHz



Imp-G DEPT $CDCl_3$ 101 MHz

G-4



Imp-G IR

G-5

Qualitative Compound Report

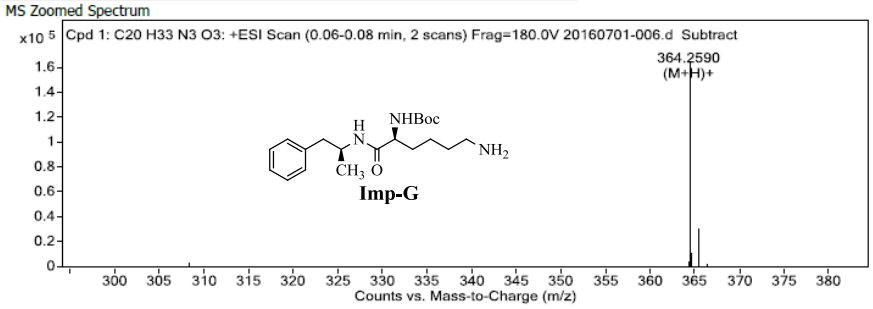
Data File	20160701-006.d	**Sample Name**	Impurity G
Sample Type	Sample	**Position**	Vial 55
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160615001

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C20 H33 N3 O3	0.06	363.2517	165528	C20 H33 N3 O3	363.2522	-1.27

Compound Label	RT	Algorithm	Mass
Cpd 1: C20 H33 N3 O3	0.06	Find By Formula	363.2517

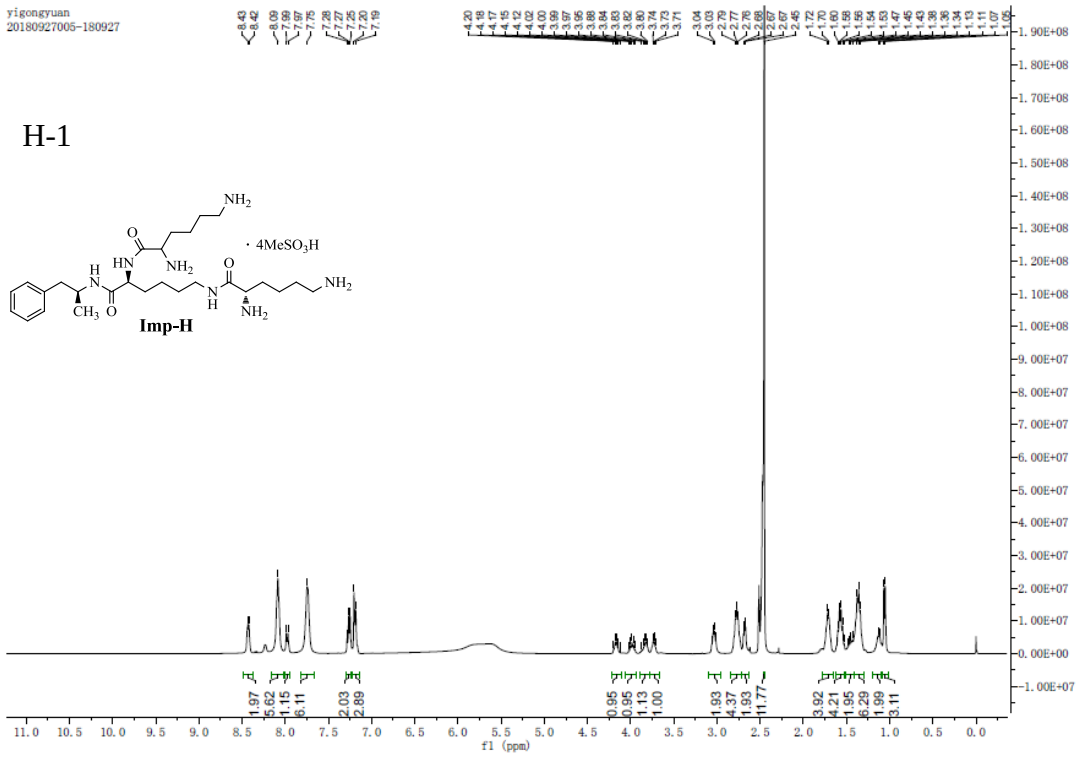
MS Zoomed Spectrum



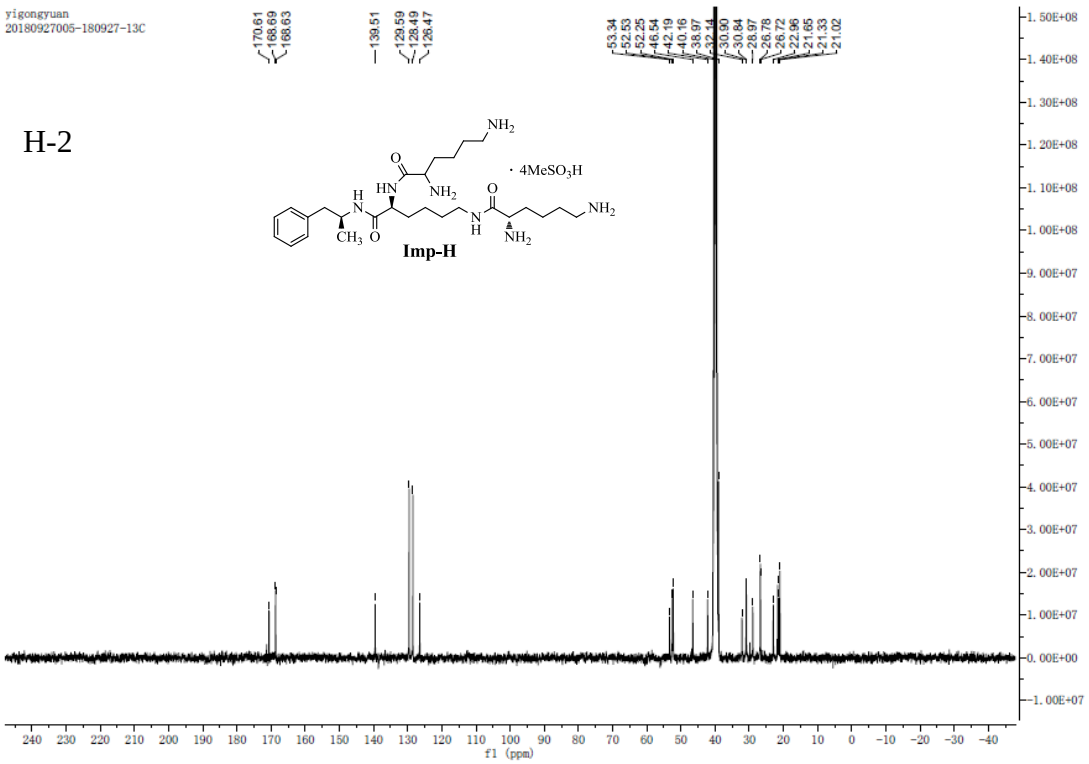
MS Spectrum Peak List

m/z	*Calc m/z*	Diff(ppm)	Abund	Formula	Ion
364.259	364.2595	-1.27	165528	C20 H34 N3 O3	(M+H)+

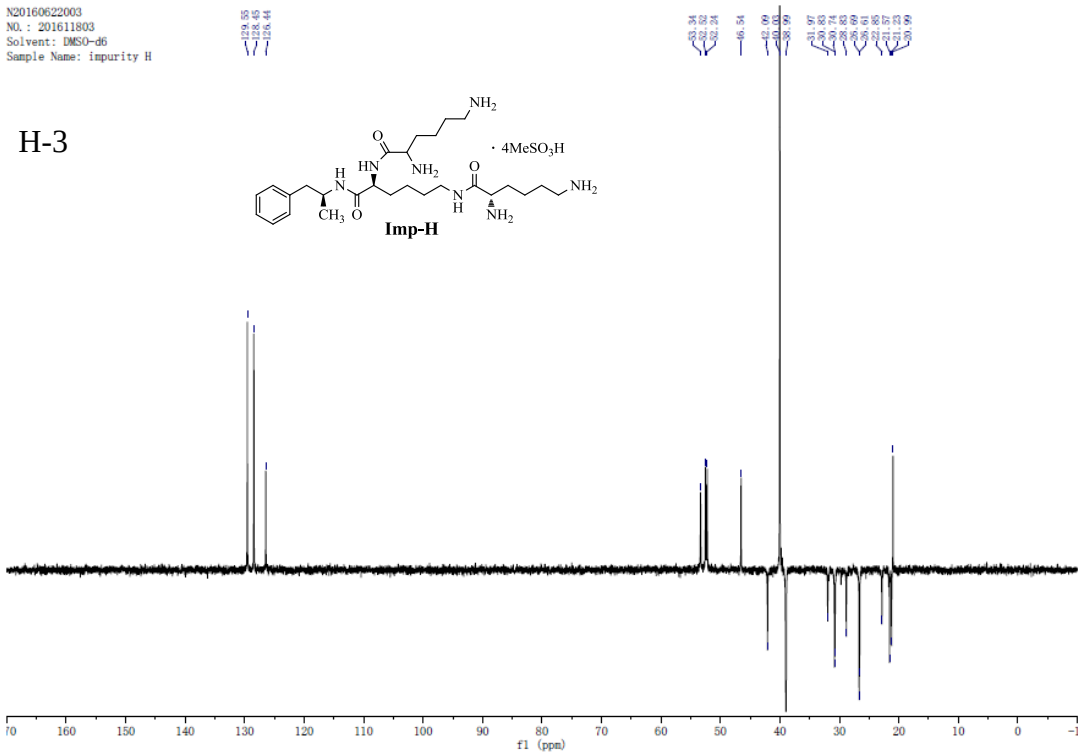
Imp-G HRMS



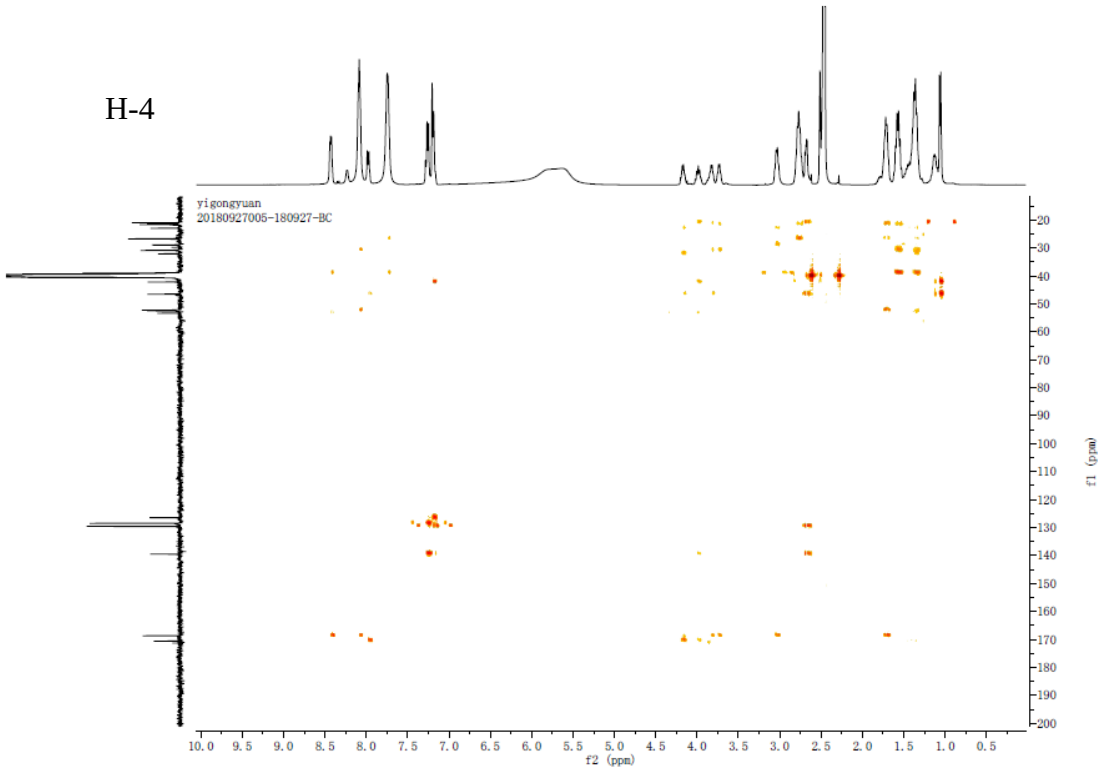
Imp-H ^{1}H NMR DMSO 400 MHz



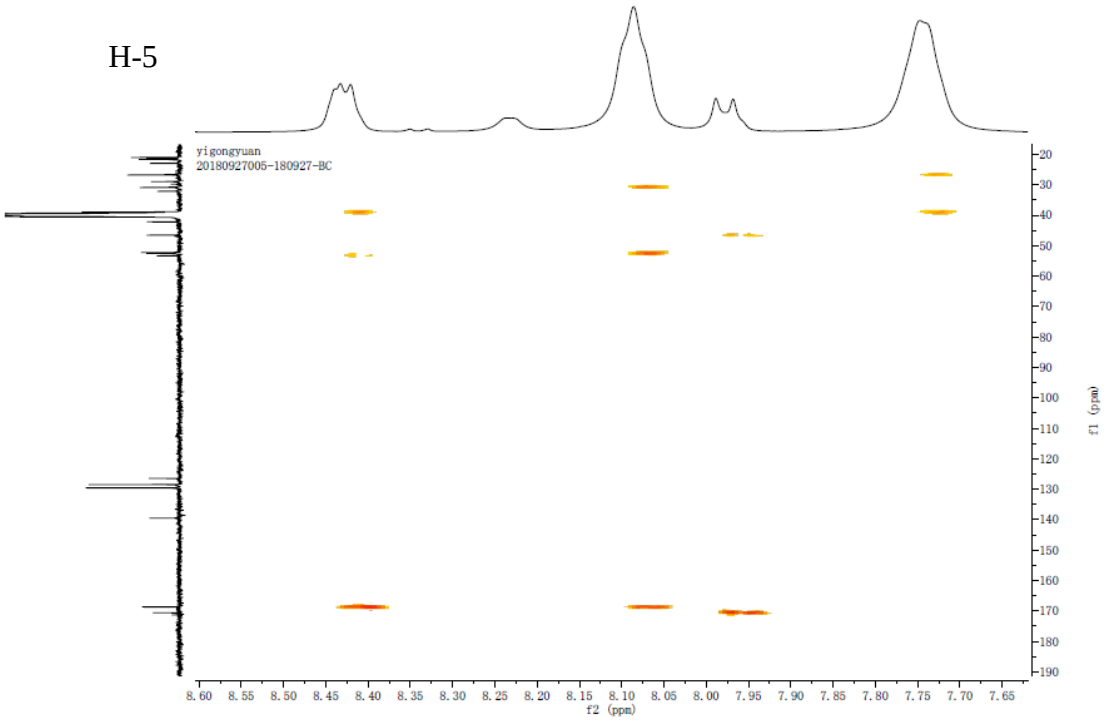
Imp-H ^{13}C NMR DMSO 101 MHz



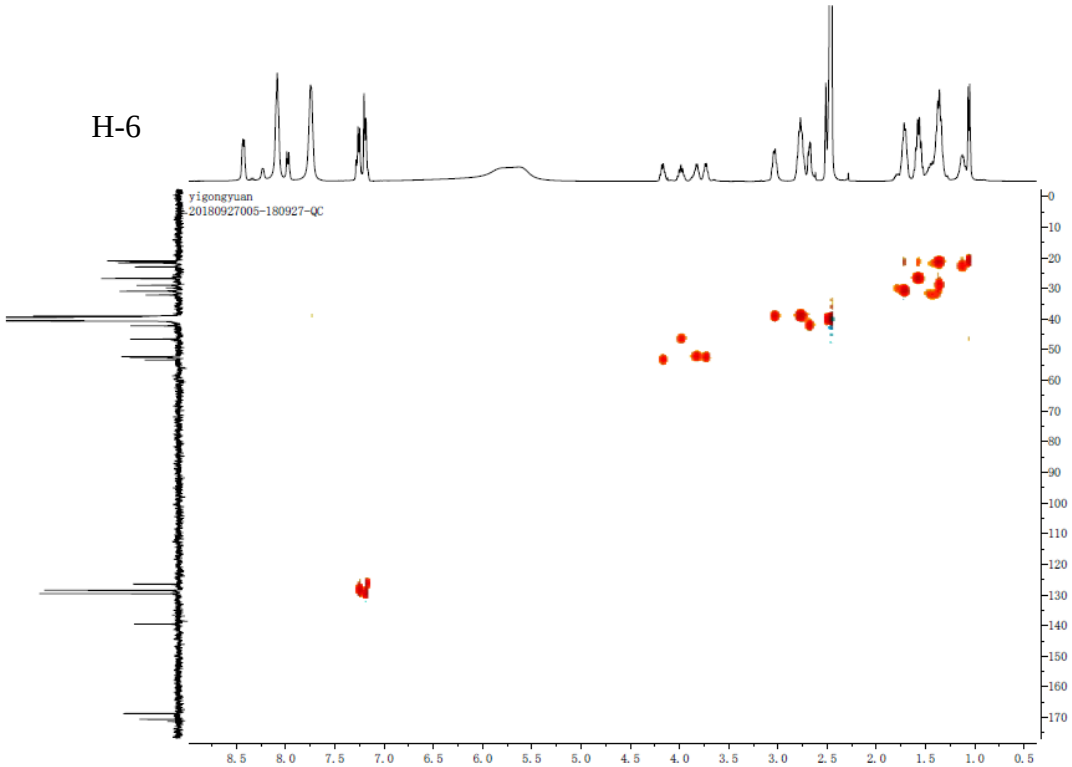
Imp-H DEPT DMSO 101 MHz



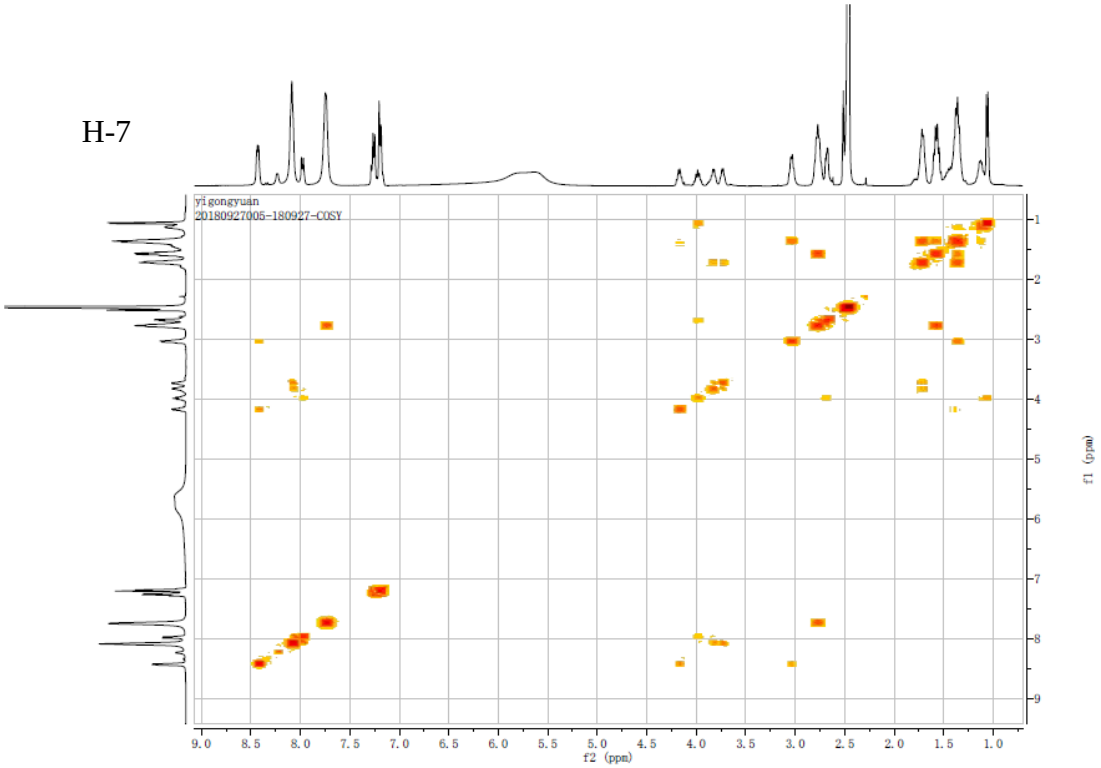
Imp-H HMBC DMSO



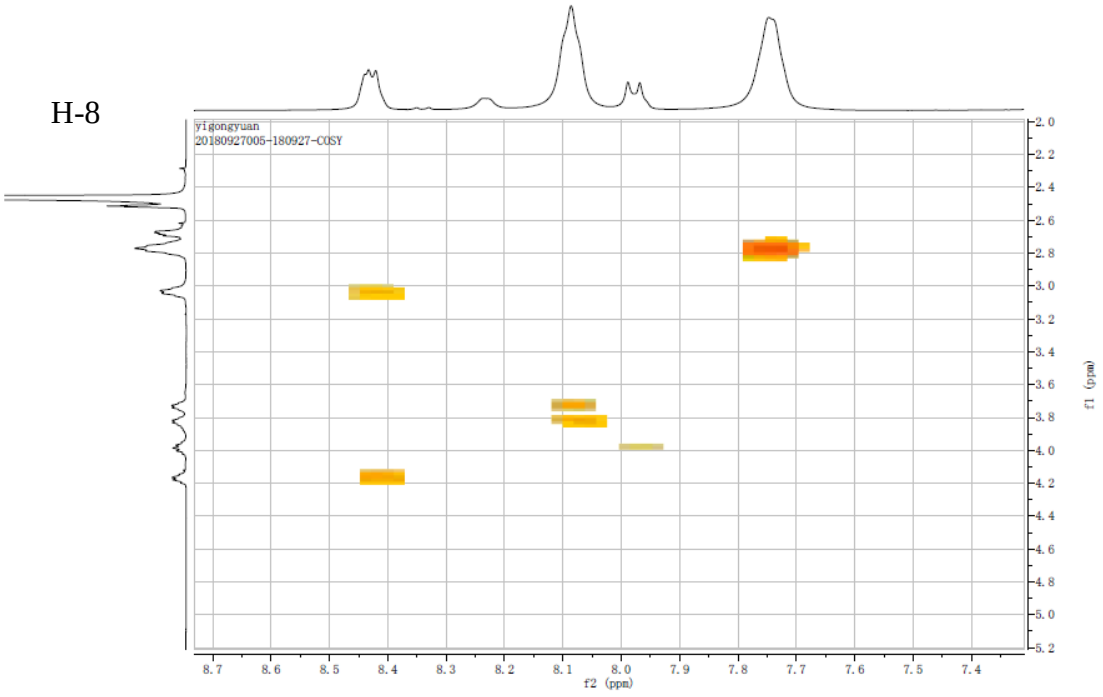
Imp-H the enlarged view of HMBC DMSO



Imp-H HSQC DMSO

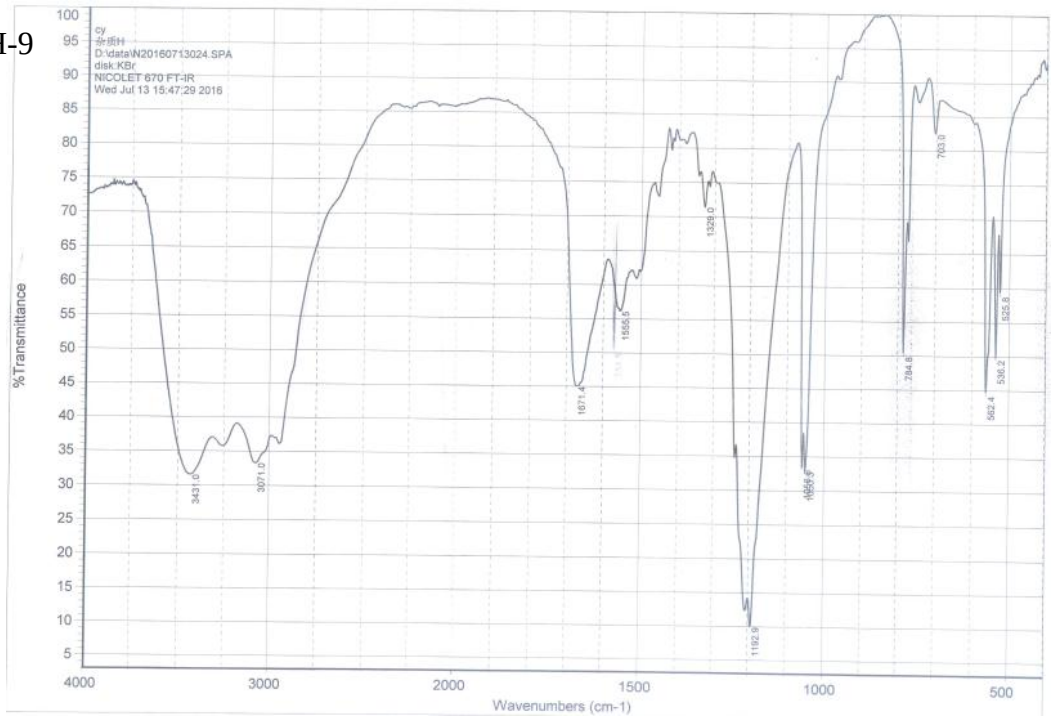


Imp-H COSY DMSO



Imp-H the enlarged view of COSY DMSO

H-9



Imp-H IR

H-10

Qualitative Compound Report

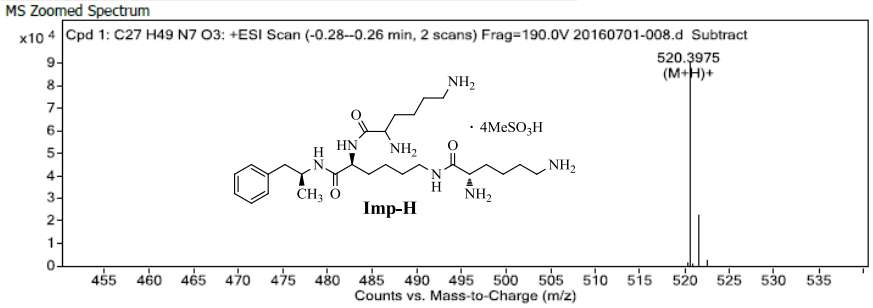
Data File	20160701-008.d	**Sample Name**	Impurity H
Sample Type	Sample	**Position**	Vial 57
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160622004

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C27 H49 N7 O3	-0.26	519.3903	91317	C27 H49 N7 O3	519.3897	1.13

Compound Label	RT	Algorithm	Mass
Cpd 1: C27 H49 N7 O3	-0.26	Find By Formula	519.3903

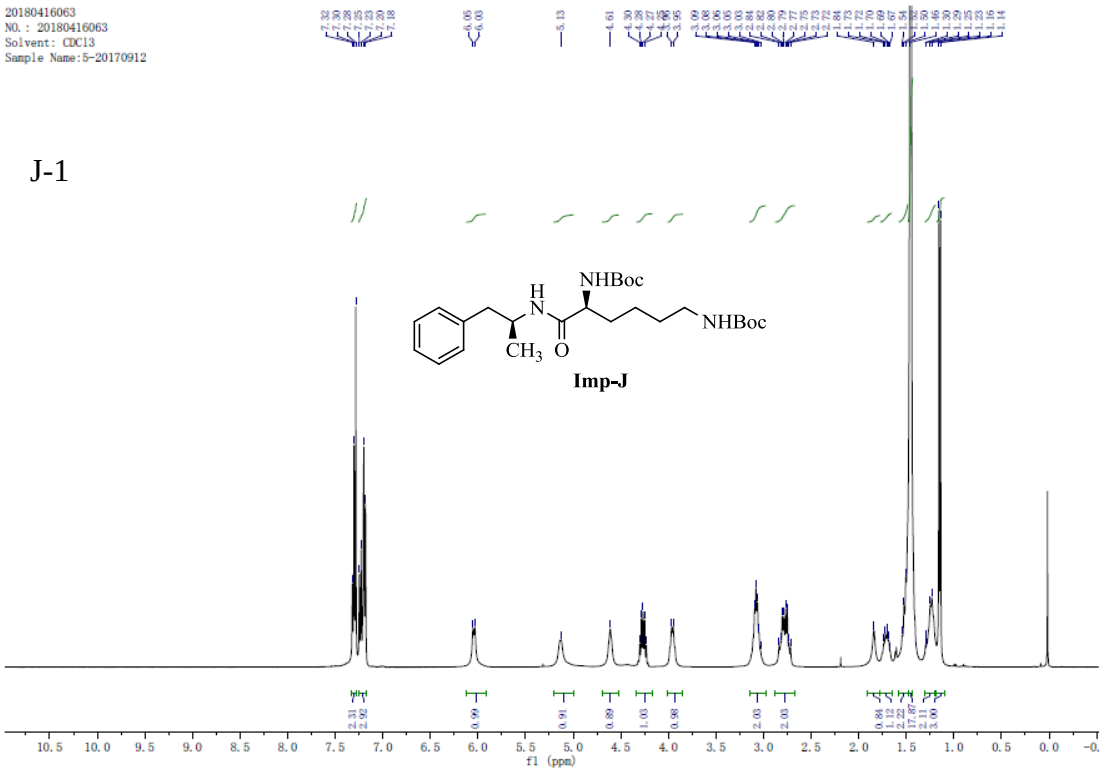
MS Zoomed Spectrum



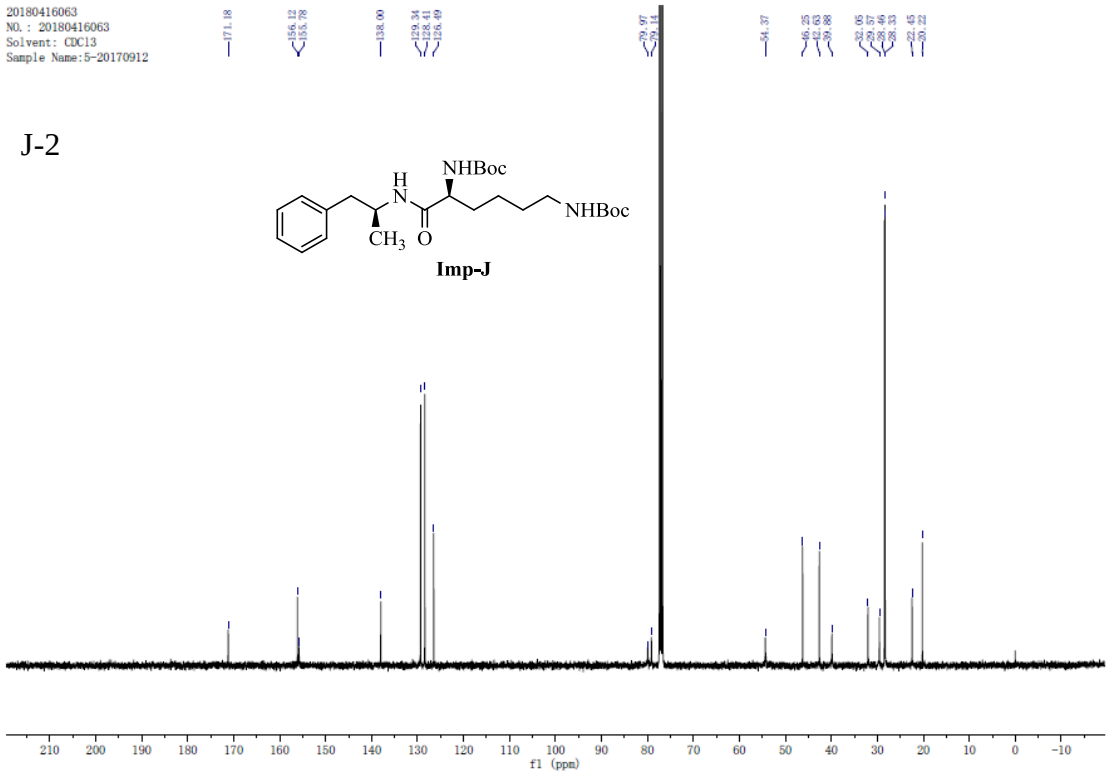
MS Spectrum Peak List

m/z	*Calc m/z*	Diff(ppm)	z	Abund	Formula	Ion
520.3975	520.397	1.12	1	91317	C27 H50 N7 O3	(M+H)+

Imp-H HRMS

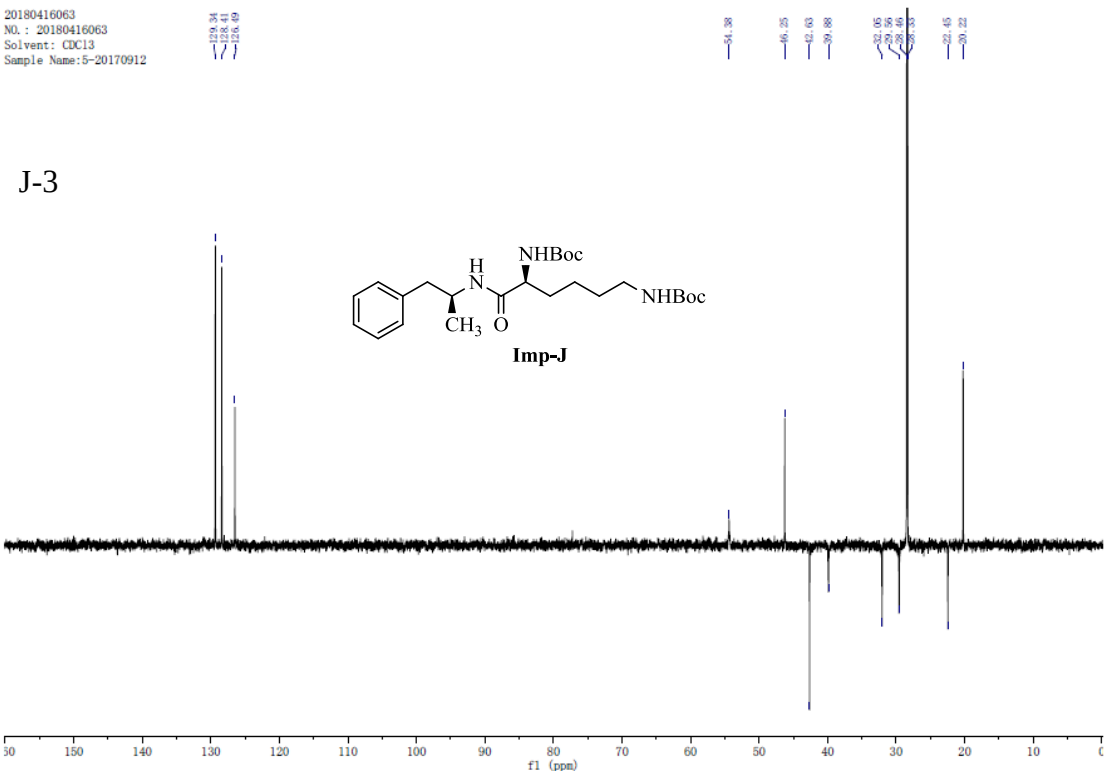


Imp-J ^{1}H NMR $CDCl_3$ 400 MHz



Imp-J ^{13}C NMR $CDCl_3$ 101 MHz

J-3



Imp-J DEPT $CDCl_3$ 101 MHz

J-4

Qualitative Compound Report

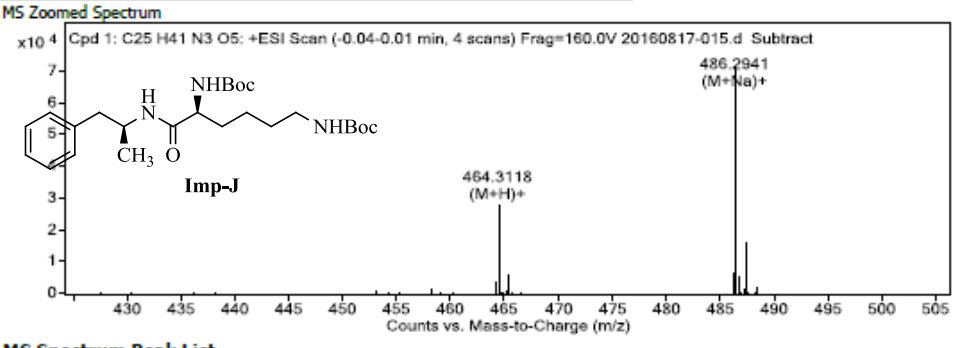
Data File	20160817-015.d	**Sample Name**	LYBBA-5
Sample Type	Sample	**Position**	Vial 14
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160816003

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C25 H41 N3 O5	-0.04	463.3048	71303	C25 H41 N3 O5	463.3046	0.44

Compound Label	RT	Algorithm	Mass
Cpd 1: C25 H41 N3 O5	-0.04	Find By Formula	463.3048

MS Zoomed Spectrum

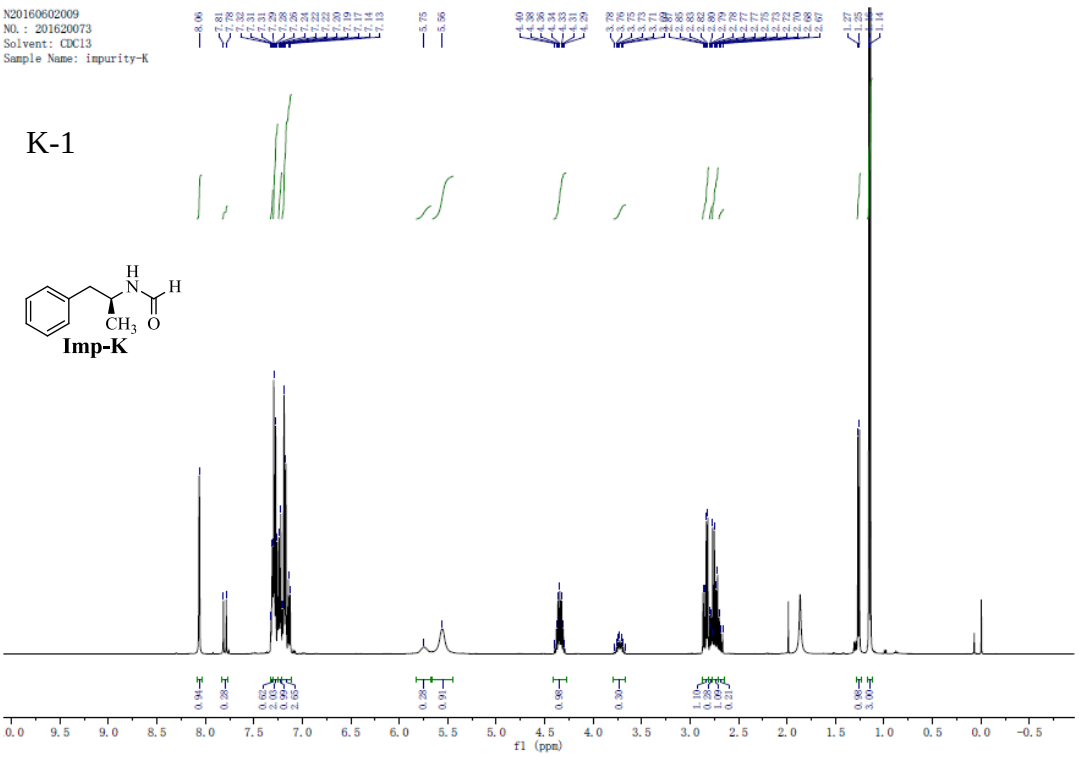


MS Spectrum Peak List

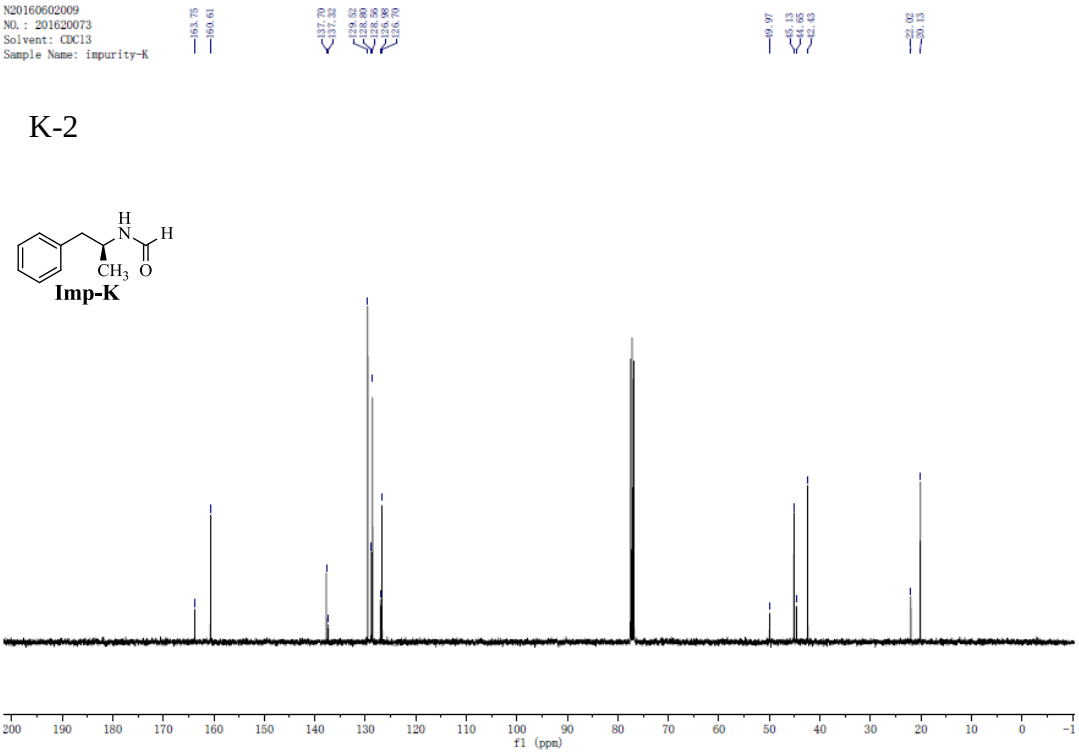
m/z	Calc m/z	Diff(ppm)	z	Abund	Formula	Ion
464.3118	464.3119	-0.3	1	28188	C25 H42 N3 O5	(M+H)+
486.2941	486.2938	0.48		71303	C25 H41 N3 Na O5	(M+Na)+

--- End Of Report ---

Imp-J HRMS



Imp-K ^{1}H NMR $CDCl_3$ 400 MHz



Imp-K ^{13}C NMR $CDCl_3$ 101 MHz

K-3

Qualitative Compound Report

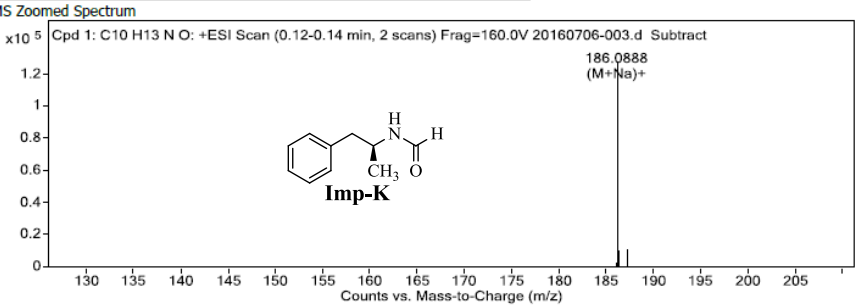
Data File	20160706-003.d	**Sample Name**	Impurity-K
Sample Type	Sample	**Position**	Vial 77
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160706004

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C10 H13 N O	0.14	163.0996	128172	C10 H13 N O	163.0997	-0.88

Compound Label	RT	Algorithm	Mass
Cpd 1: C10 H13 N O	0.14	Find By Formula	163.0996

MS Zoomed Spectrum

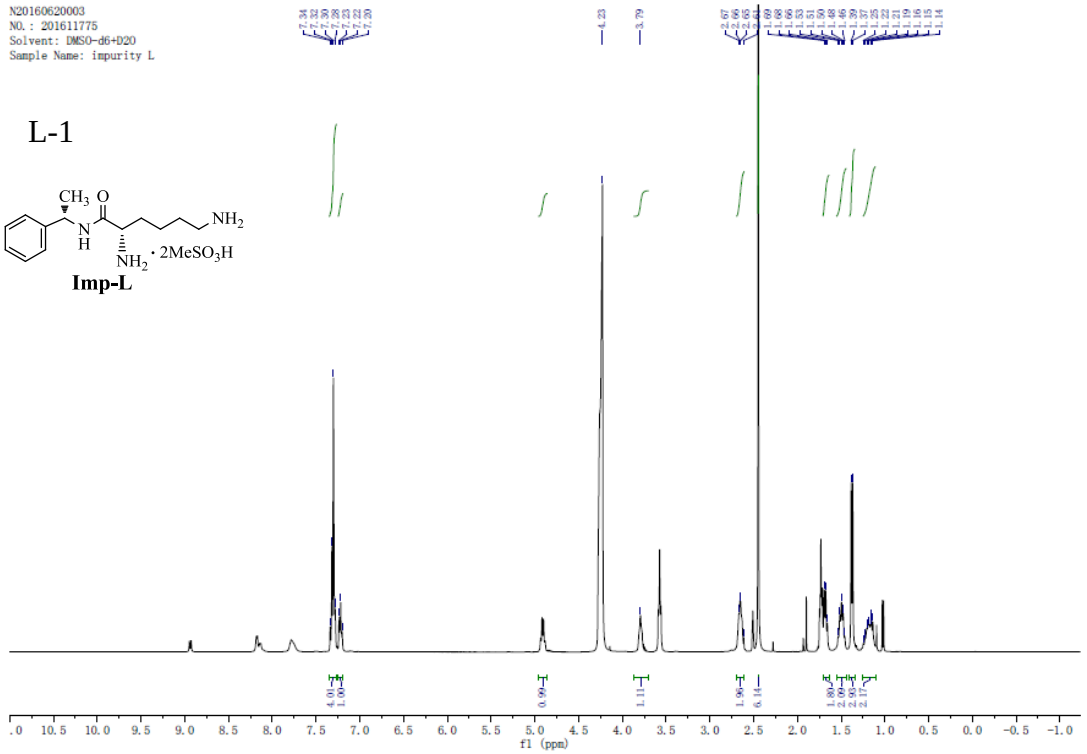


MS Spectrum Peak List

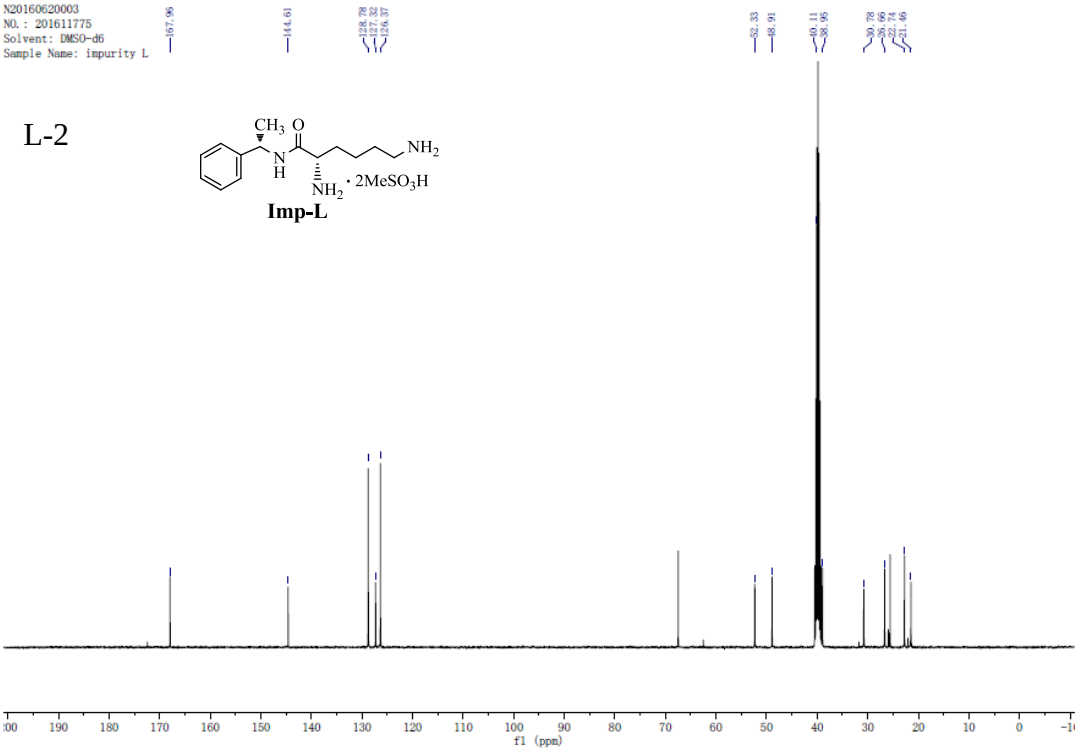
m/z	*Calc m/z*	Diff(ppm)	z	Abund	Formula	Ion
186.0888	186.0889	-0.77	1	128172	C10 H13 N Na O	(M+Na)+

--- End Of Report ---

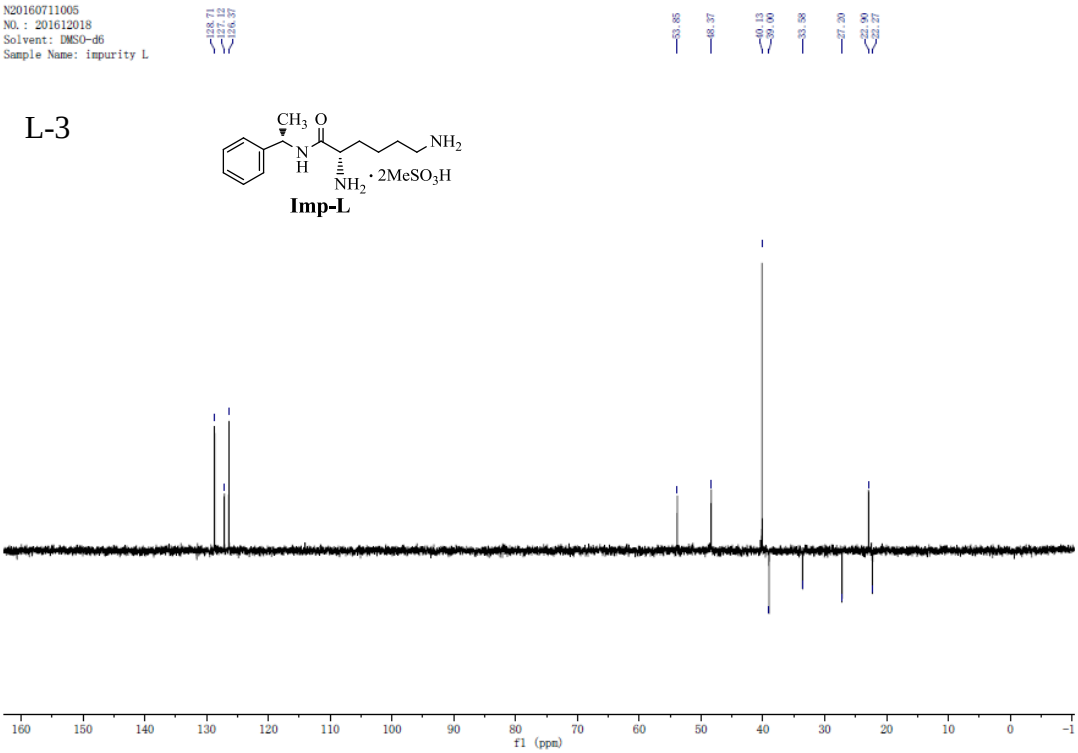
Imp-K HRMS



Imp-L ^{1}H NMR DMSO 400 MHz

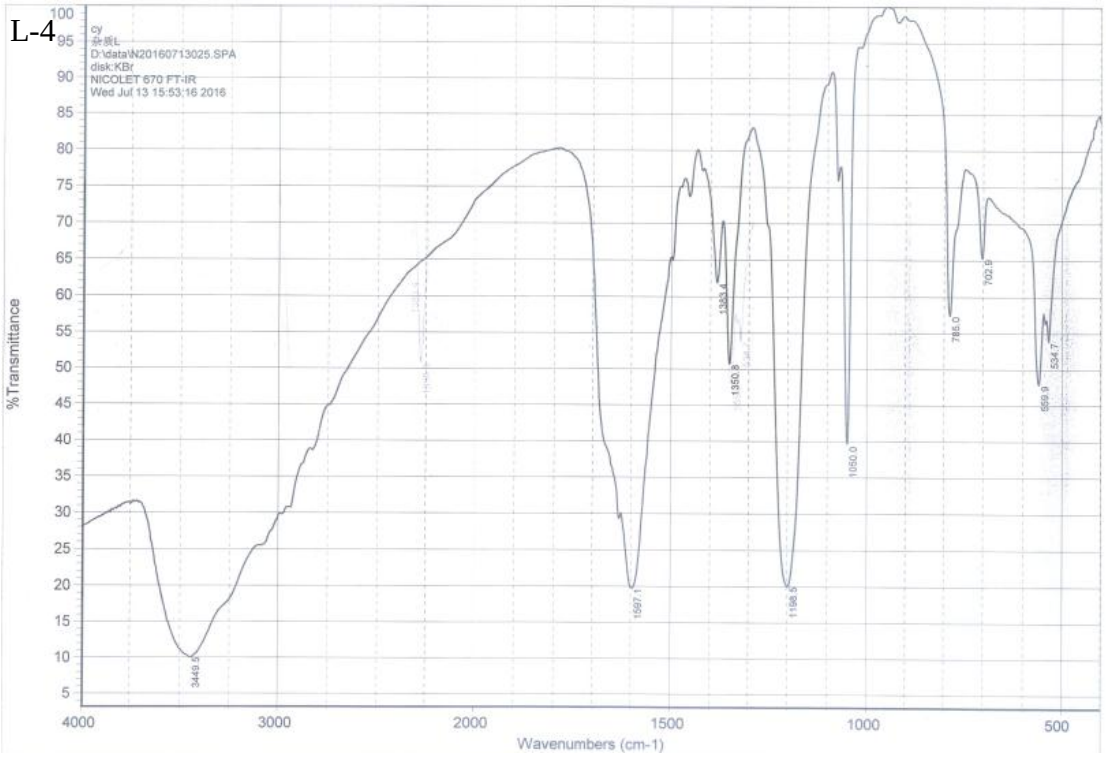


Imp-L ^{13}C NMR DMSO 101 MHz



Imp-L DEPT DMSO 101 MHz

L-4



Imp-L IR

L-5

Qualitative Compound Report

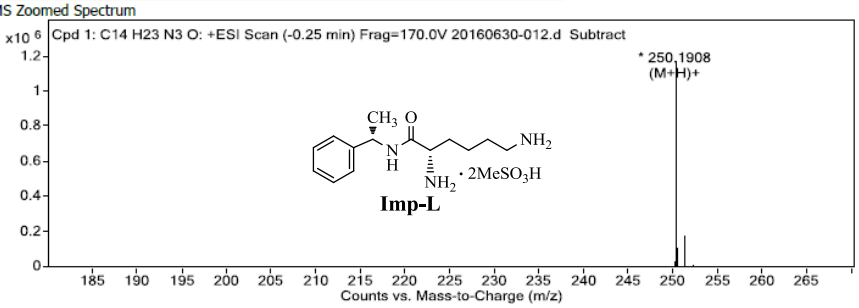
Data File	20160630-012.d	**Sample Name**	Impurity L
Sample Type	Sample	**Position**	Vial 48
Instrument Name	Instrument 1	**User Name**	
Acq Method		**IRM Calibration Status**	Success
DA Method	MS.m	**Comment**	N20160630030

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C14 H23 N3 O	-0.25	249.1835	1176045	C14 H23 N3 O	249.1841	-2.39

Compound Label	RT	Algorithm	Mass
Cpd 1: C14 H23 N3 O	-0.25	Find By Formula	249.1835

MS Zoomed Spectrum

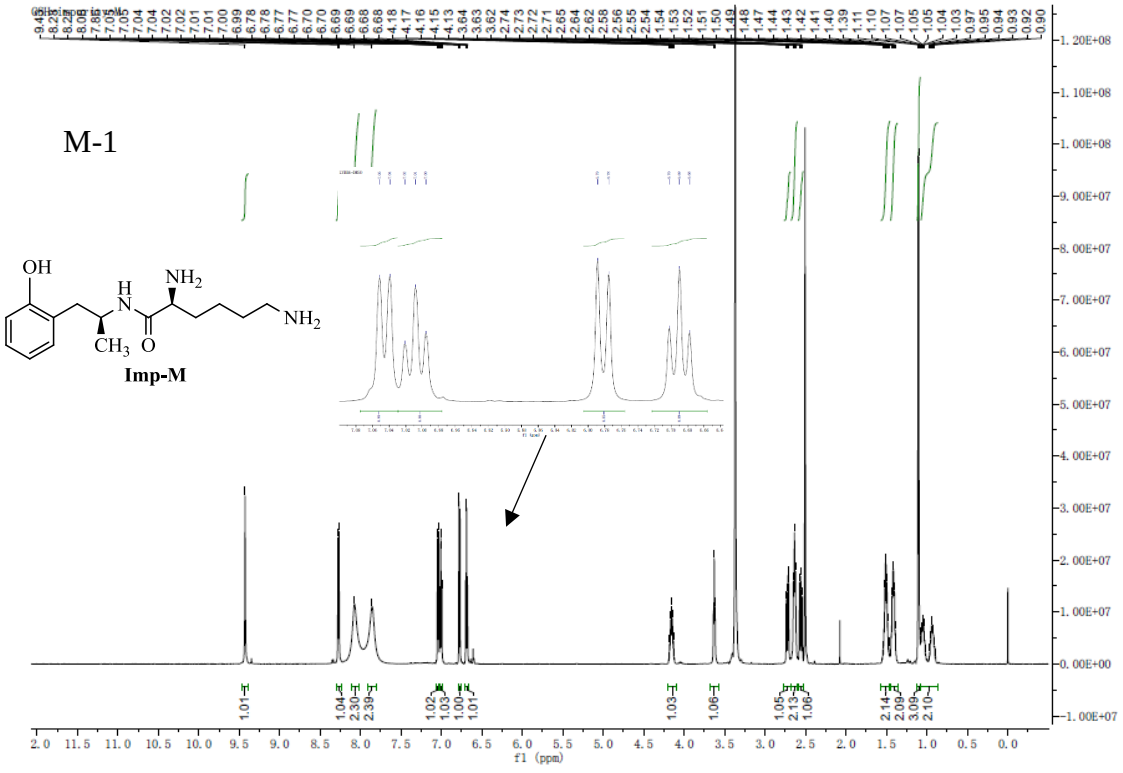


MS Spectrum Peak List

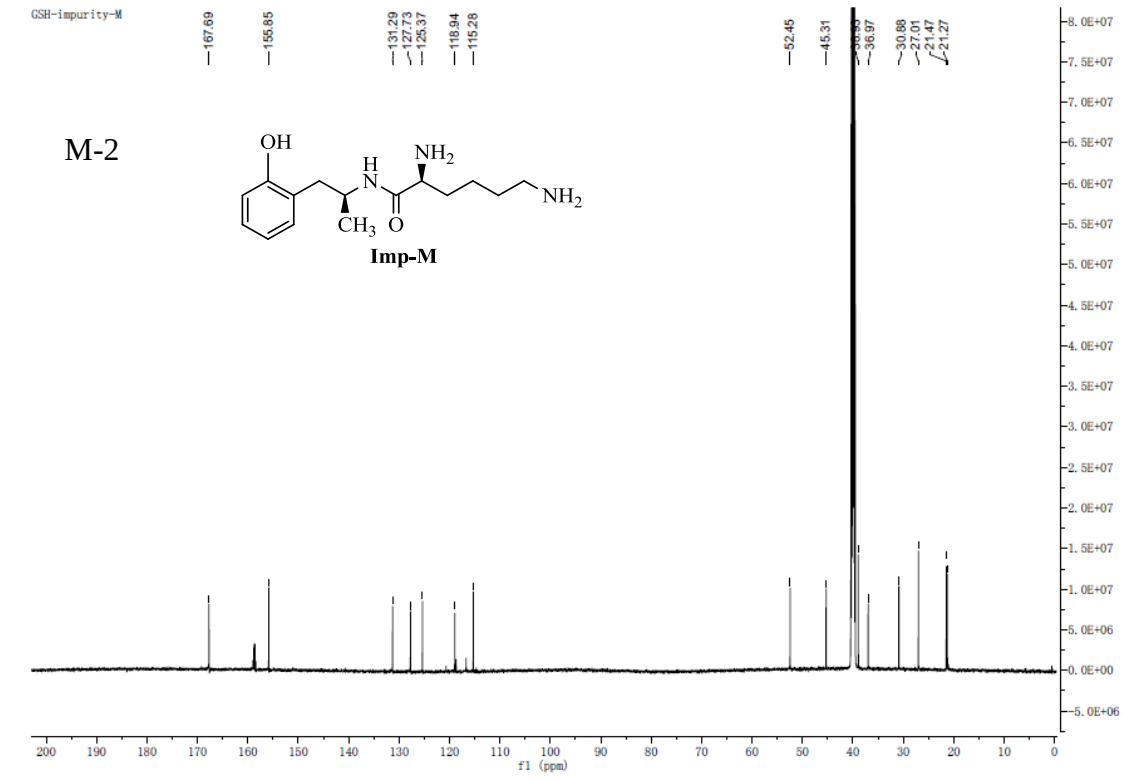
m/z	*Calc m/z*	Diff(ppm)	Abund	Formula	Ion
250.1908	250.1914	-2.37	1176045	C14 H24 N3 O	(M+H)+

--- End Of Report ---

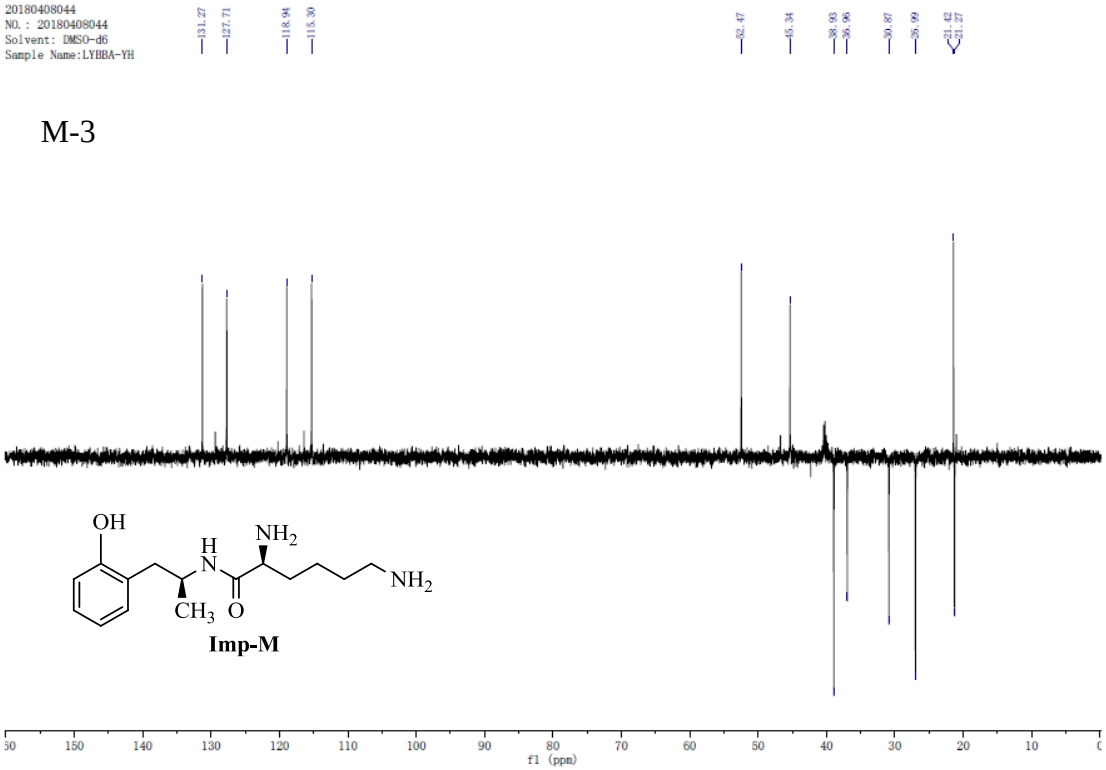
Imp-L HRMS



Imp-M ^{1}H NMR DMSO 400 MHz

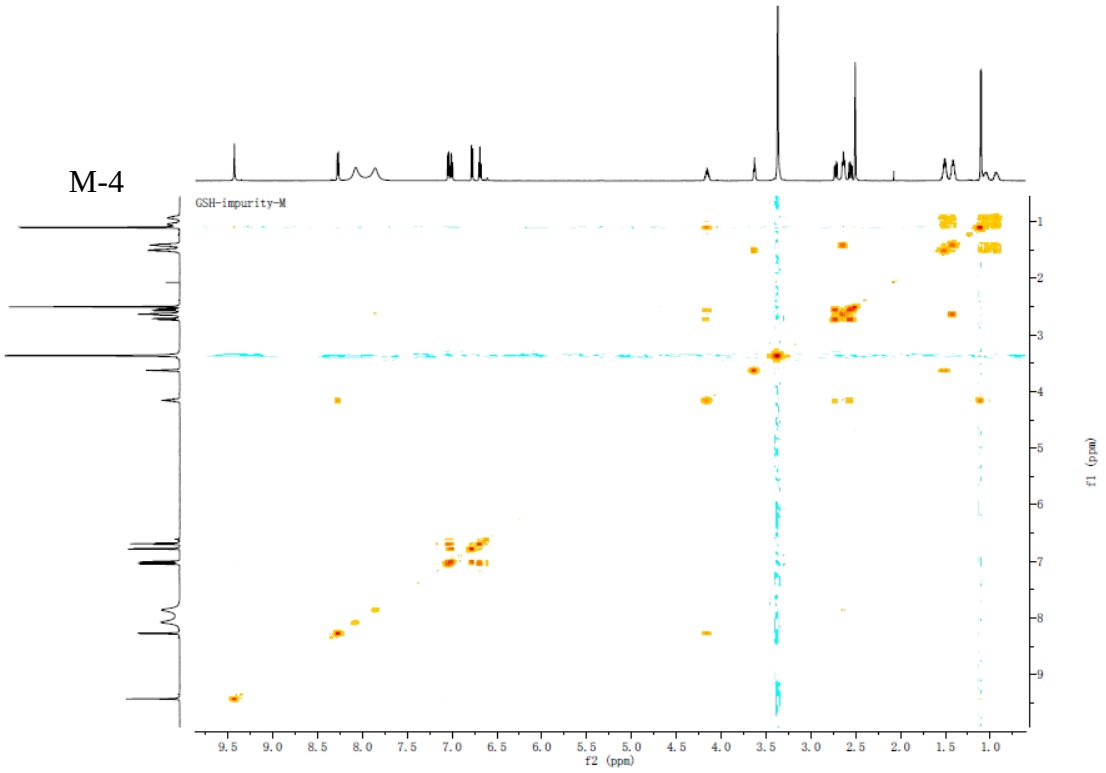


Imp-M ^{13}C NMR DMSO 101 MHz



M-3

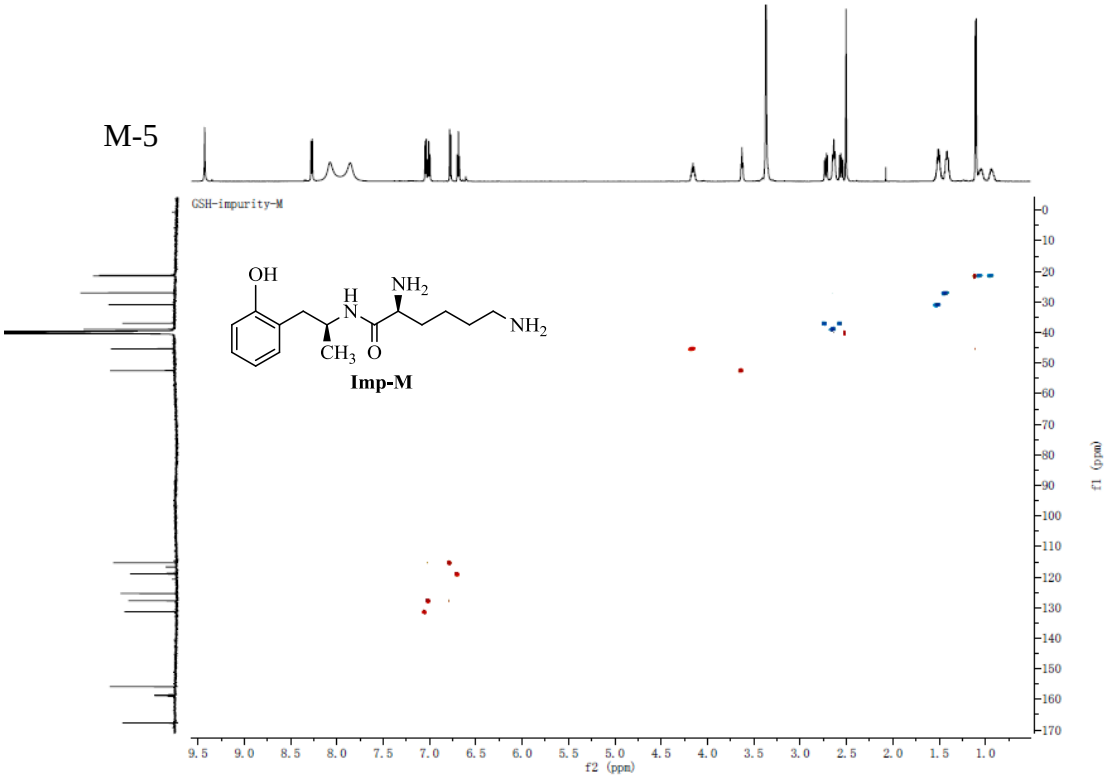
Imp-M DEPT DMSO 101 MHz



M-4

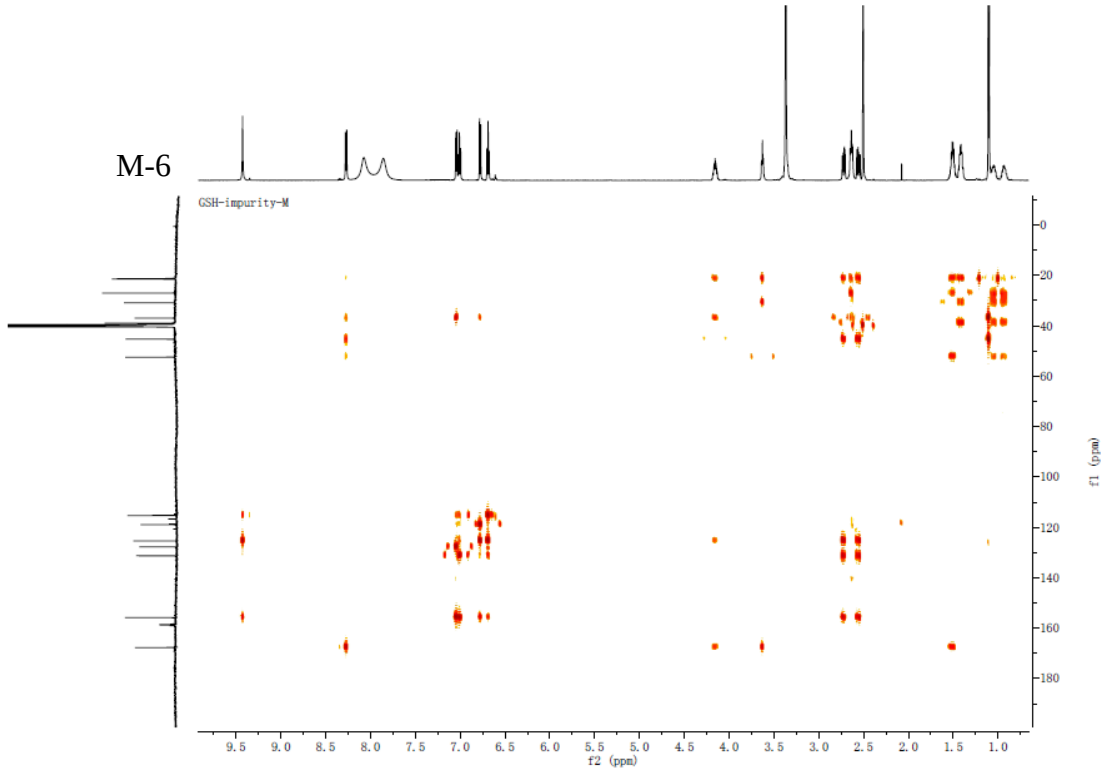
Imp-M COSY DMSO

M-5



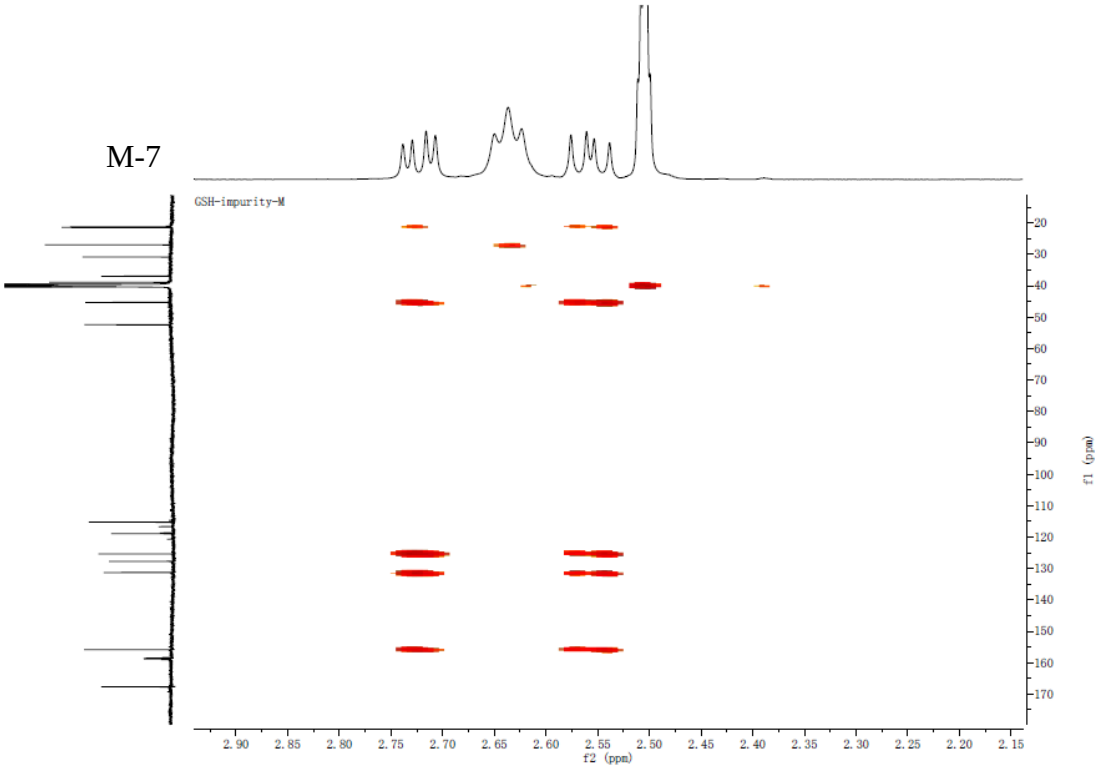
Imp-M HSQC DMSO

M-6



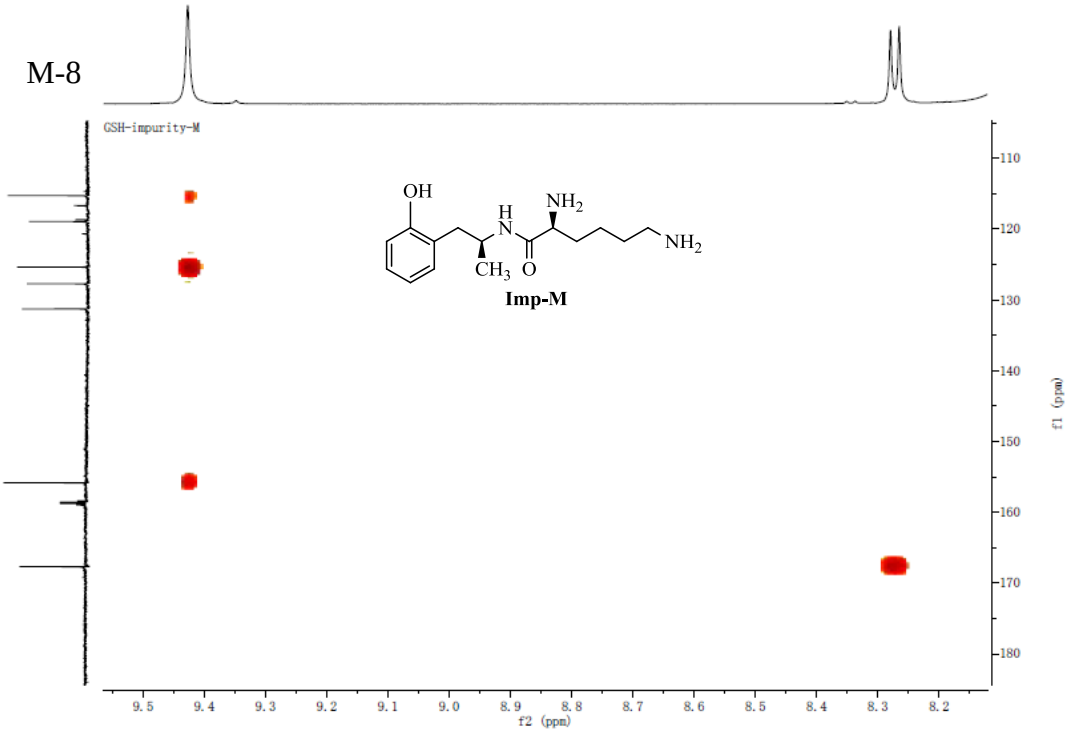
Imp-M HMBC DMSO

M-7



Imp-M　　the enlarged view of HMBC　　DMSO

M-8



Imp-M　　the enlarged view of HMBC　　DMSO

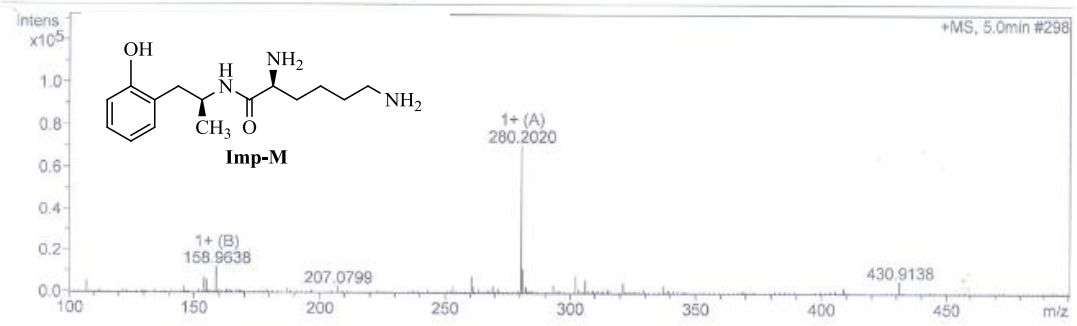
M-9 Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\SHUJVFENXI\MADAWEIGROUP\LYBBA-2_GA3_01_10594.d	Acquisition Date	1/31/2018 5:53:48 AM
Method	20150915.m	Operator	BDAL@DE
Sample Name	LYBBA-2	Instrument / Ser#	maXis 4G 21240
Comment			

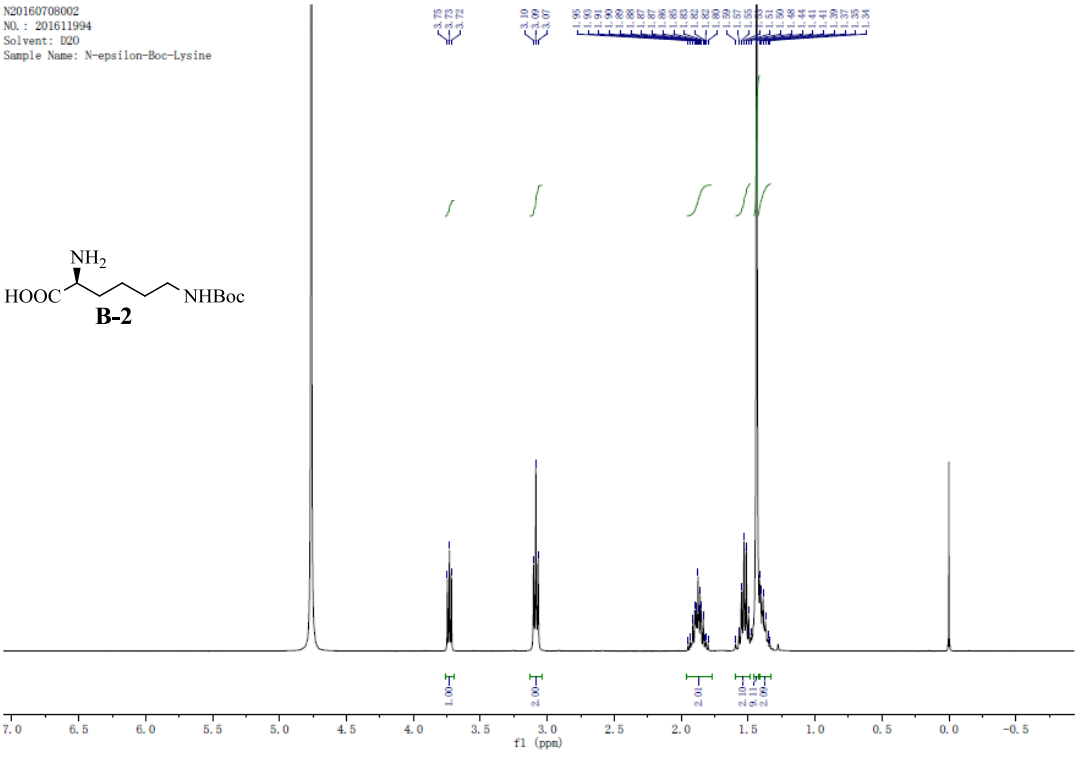
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	220 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



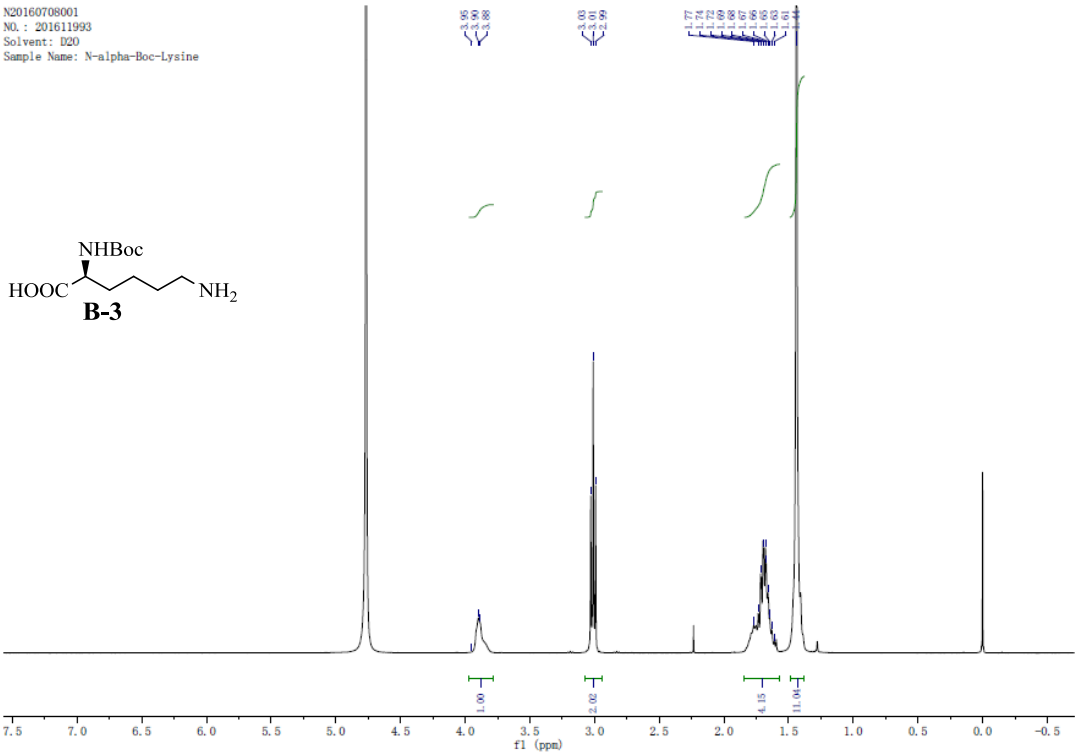
Imp-M

Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
280.2020	1	C 15 H 26 N 3 O 2	100.00	280.2020	-0.3	-0.0	8.2	4.5	even	ok
	2	C 18 H 27 N Na	34.06	280.2036	5.5	5.8	22.6	5.5	even	ok
	3	C 10 H 24 N 8 Na	0.00	279.2016	5.9	10.2	767.3	2.5	even	ok
	4	C 13 H 28 N O 5	0.00	278.1962	-1.5	-5.6	812.7	0.5	even	ok
	5	C 14 H 24 N 5 O	0.00	278.1975	2.7	8.1	816.3	5.5	even	ok
	6	C 12 H 25 N 5 Na O	0.00	278.1951	-6.5	-0.5	816.3	2.5	even	ok



B-2

B-2 ^{1}H NMR D_2O 400 MHz



B-3 ^{1}H NMR D_2O 400 MHz