Electronic Supplementary Material (ESI) for RSC Advances

Evans chiral auxiliary based highly stereoselective $TiCl_4$-Catalyzed Michael reaction with β-Nitrostyrene. Efficient Accesses to Optically Active Quinolizidinones, Piperidinones and Pyrrolidinones

Siva R. Kamma,[a][b], U. Viplava Prasad[b] and Subhabrata Sen*[a]

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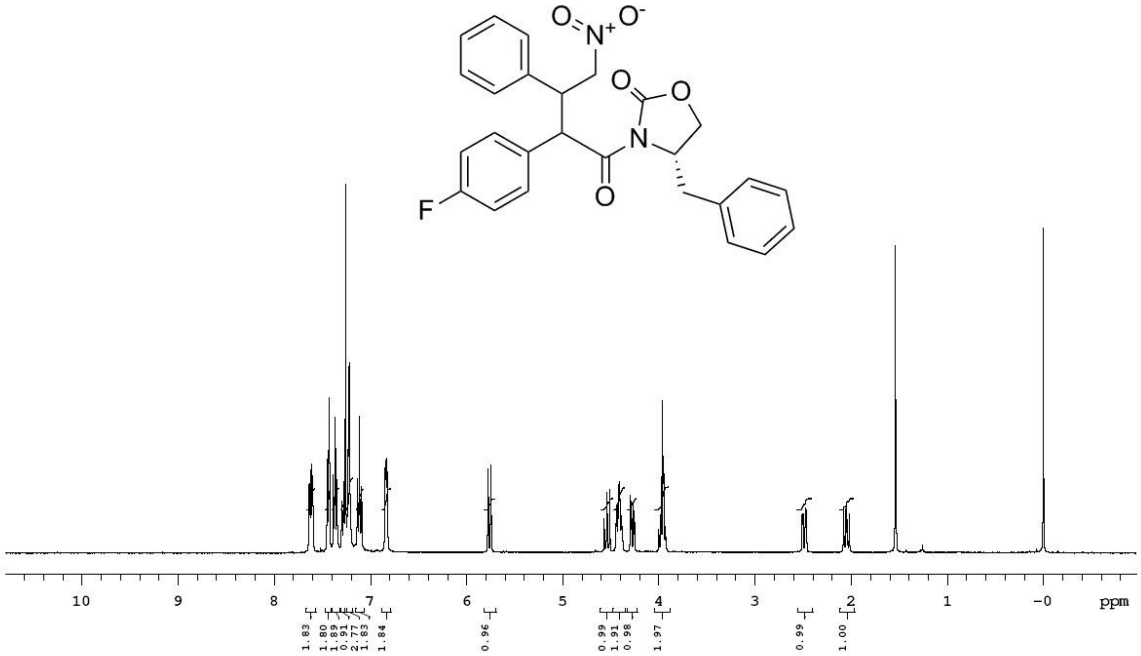
O
N+
O-
O
O
N
O
F
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9
8
7
6
5
4
3
2
1
-0
ppm
1.83
1.80
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1.83
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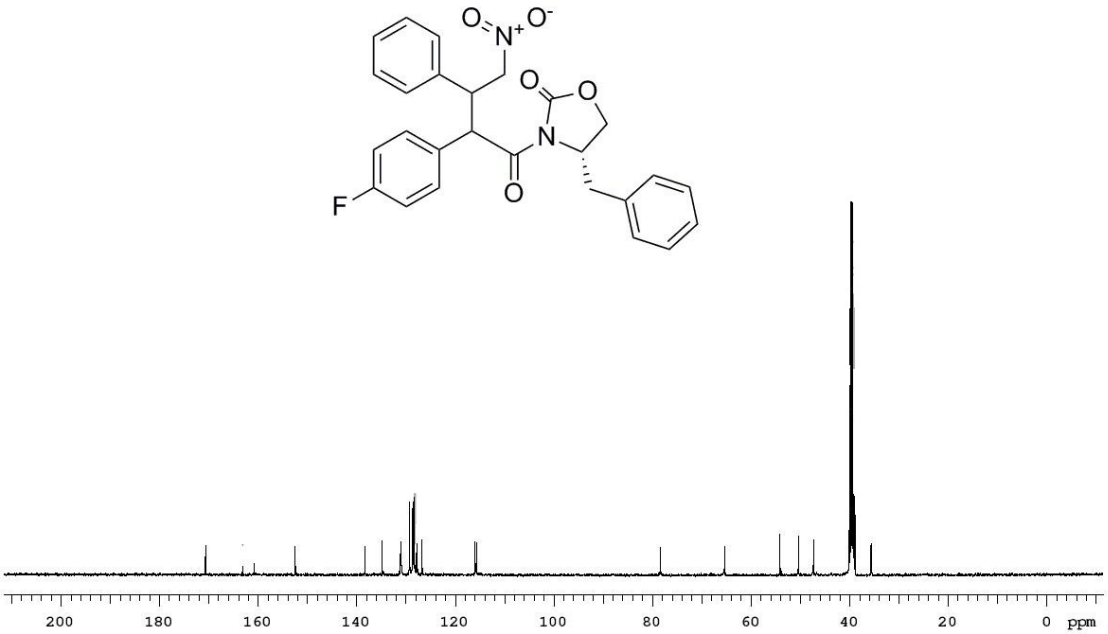
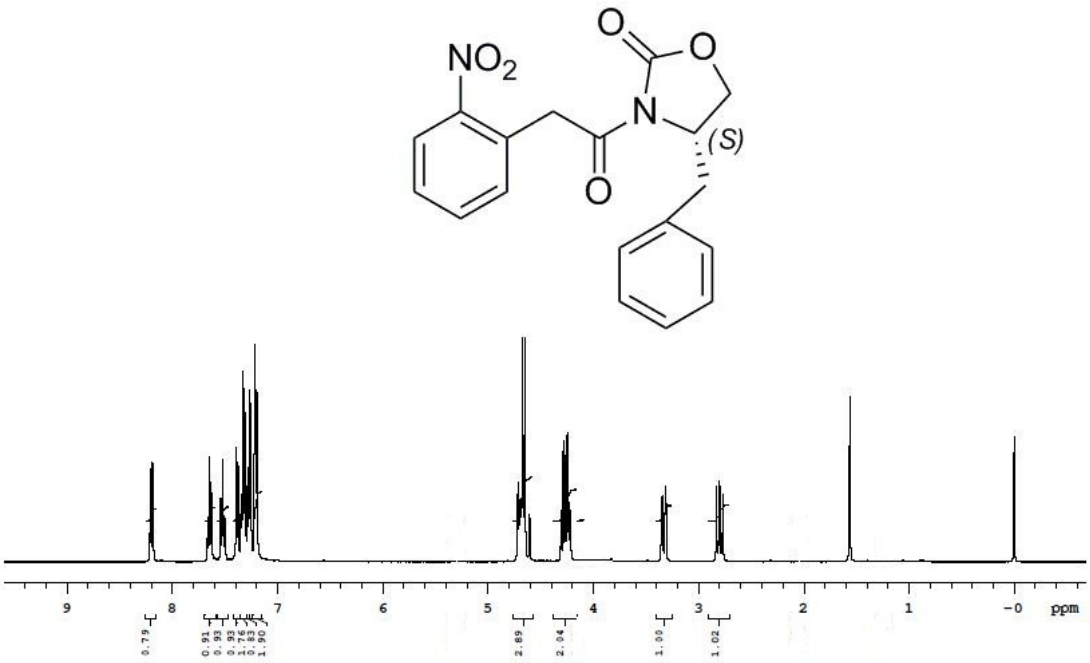
0.96
0.99
1.91
0.98
1.97
0.99
1.00


O
N+
O-
O
O
N
O
F
200
180
160
140
120
100
80
60
40
20
0
ppm

12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O
O
N
(S)
O
9
8
7
6
5
4
3
2
1
-0
ppm
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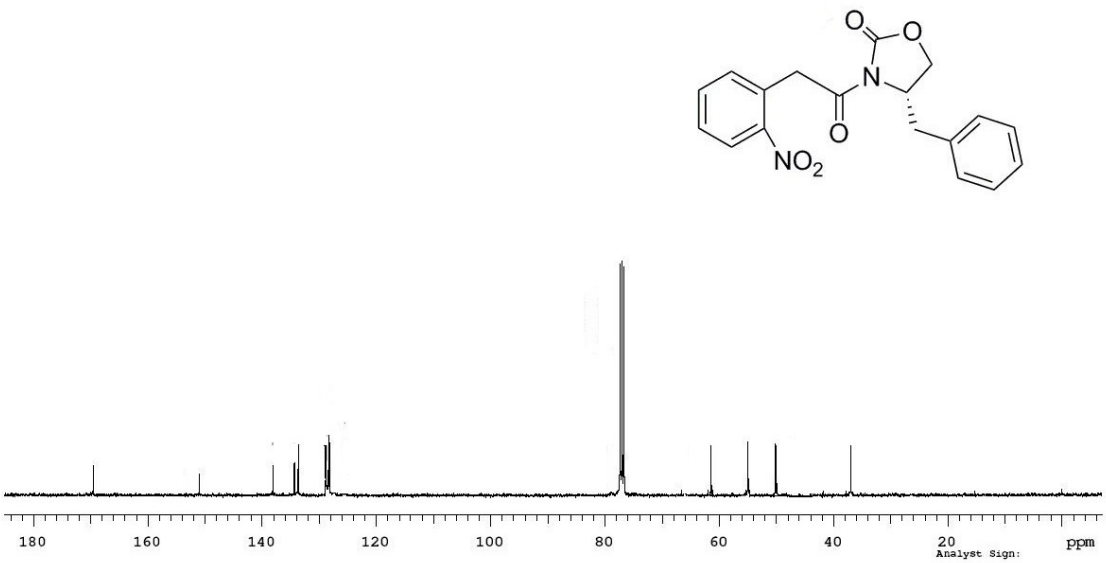
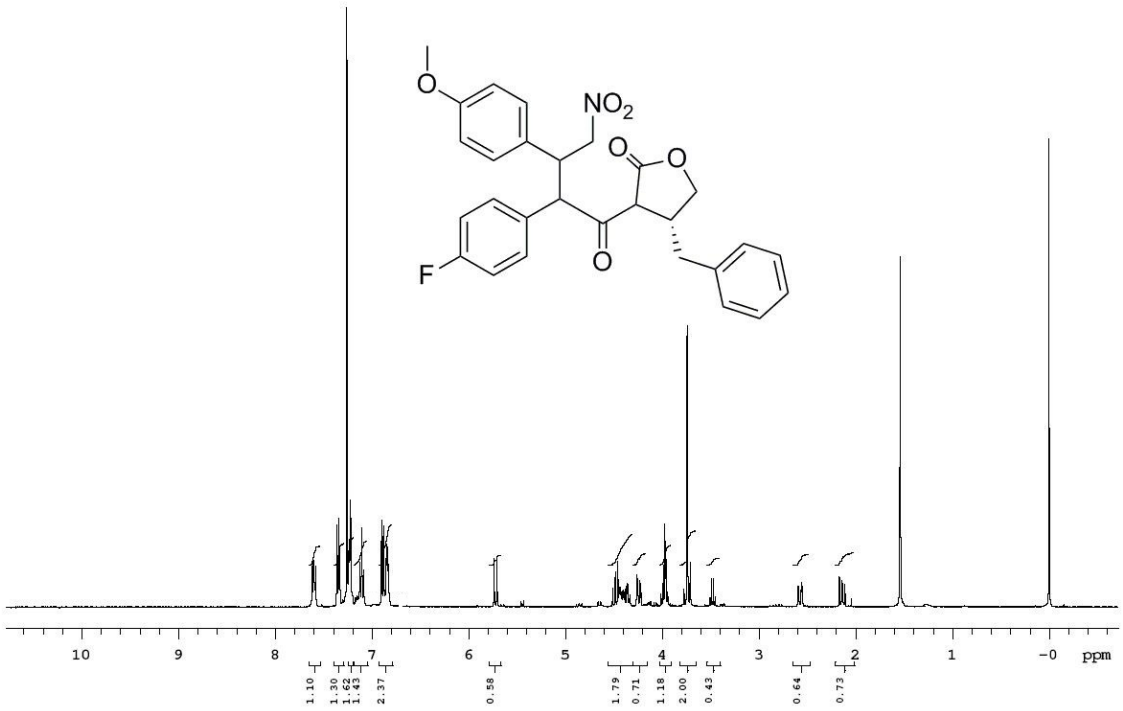
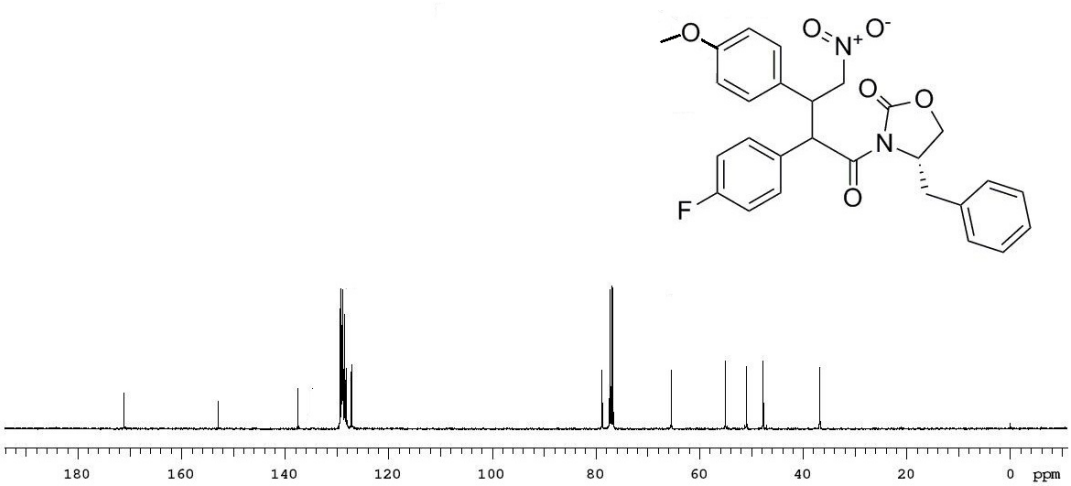
0.91
0.93
0.93
0.96
0.85
1.90
2.89
2.04
1.00
1.02

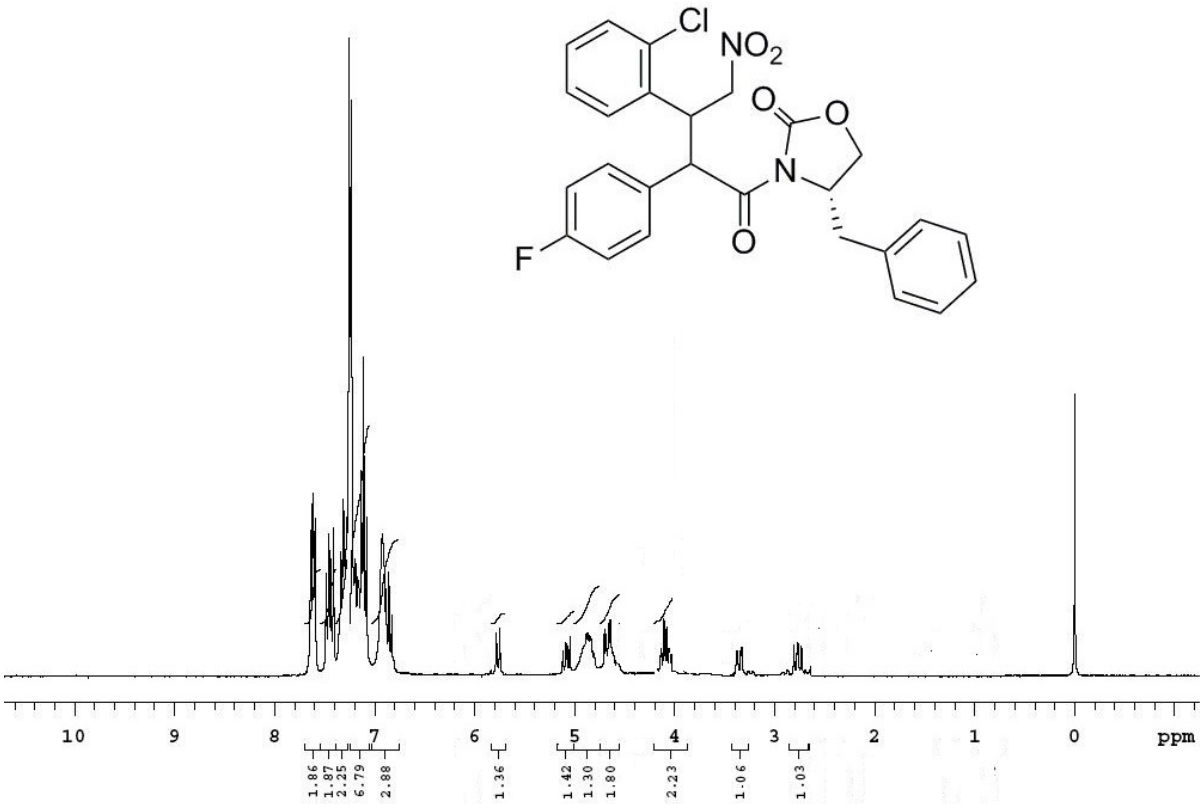

O
O
N
O
NO2
180
160
140
120
100
80
60
40
20
ppm
Analyst Sign:

15


O
NO2
O
O
F
O
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.10
1.30
1.62
1.48
2.37
0.58
1.79
0.71
1.18
2.00
0.43
0.64
0.79


O
O
N+
O-
O
O
N
F
O
180
160
140
120
100
80
60
40
20
0
ppm

16


Cl
NO2
O
O
N
O
F
10
9
8
7
6
5
4
3
2
1
0
ppm
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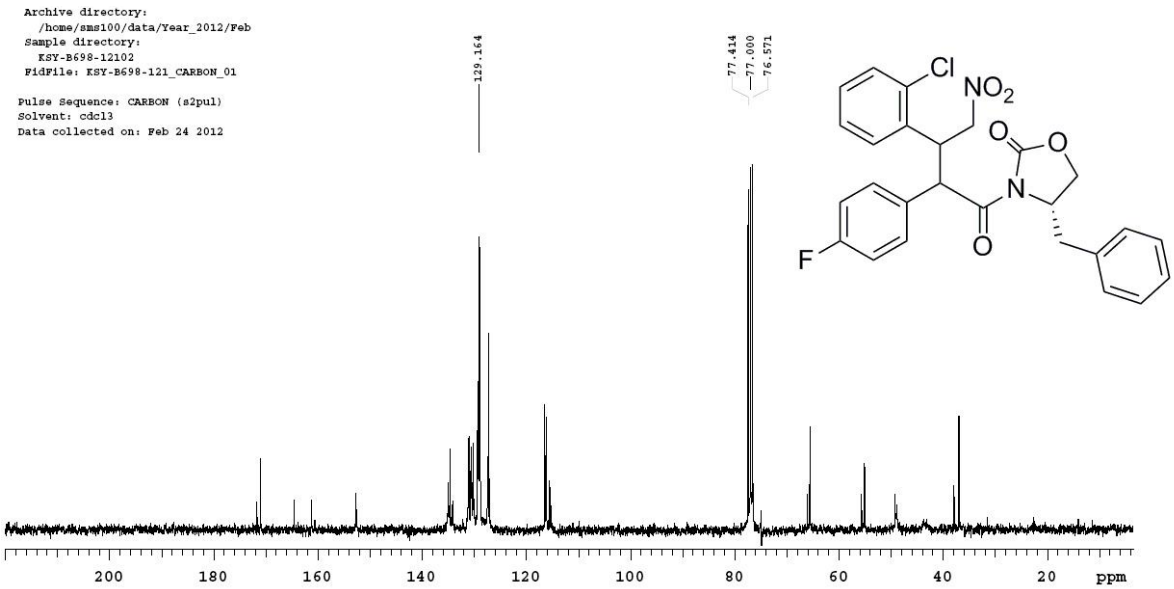
1.87
2.25
6.79
2.88
1.36
1.42
1.30
1.80
2.23
1.06
1.03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KSY-B698-12102
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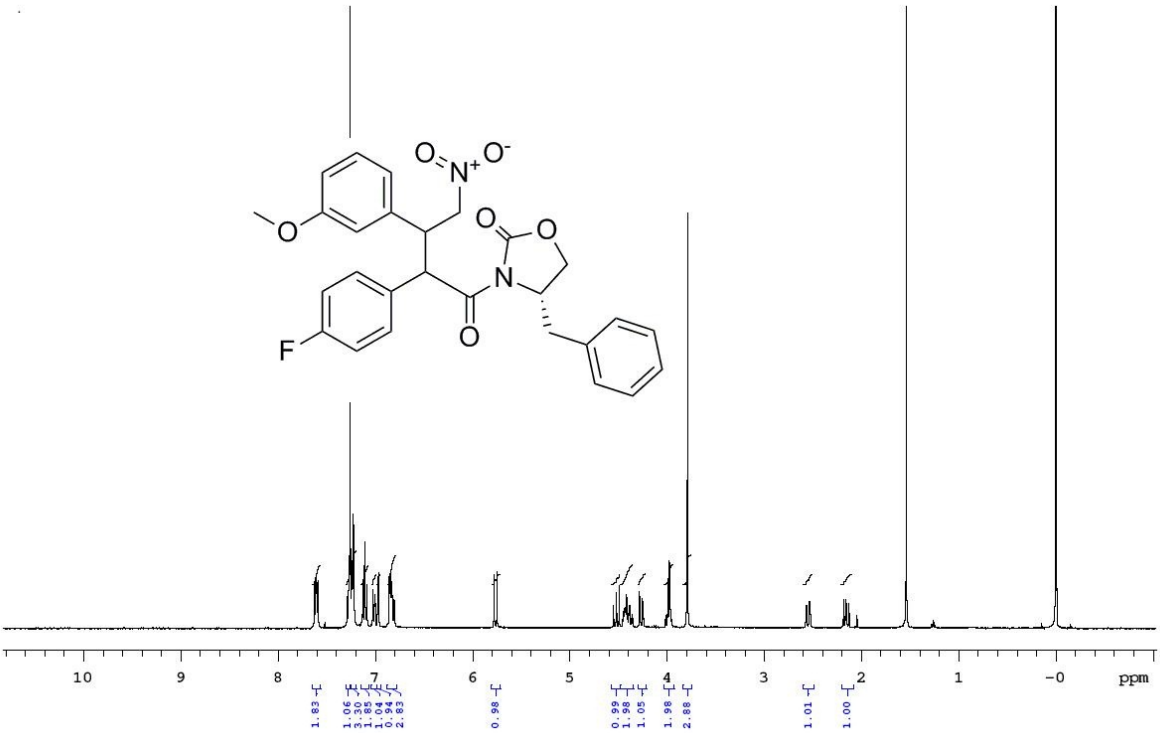
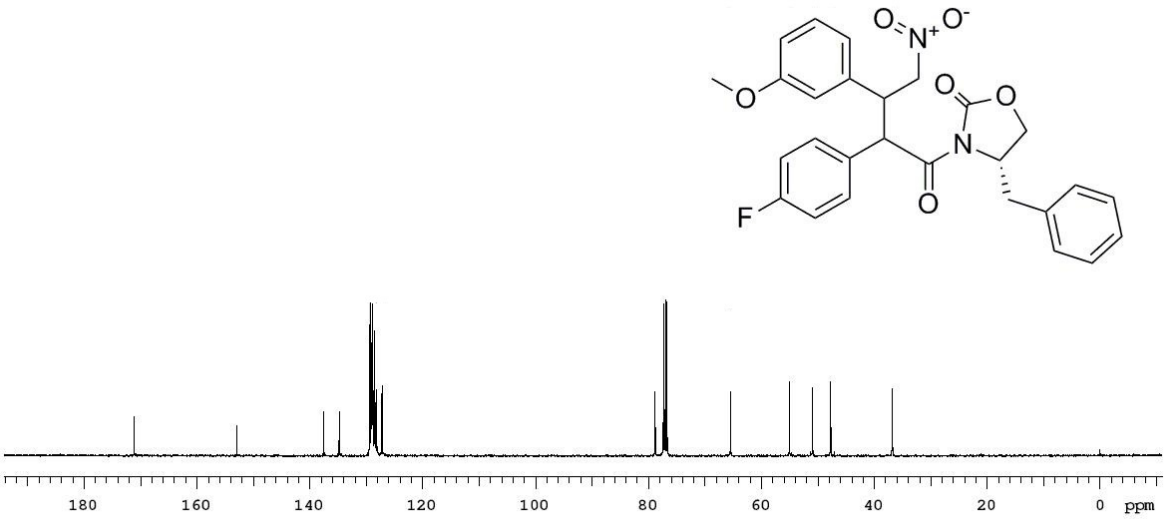
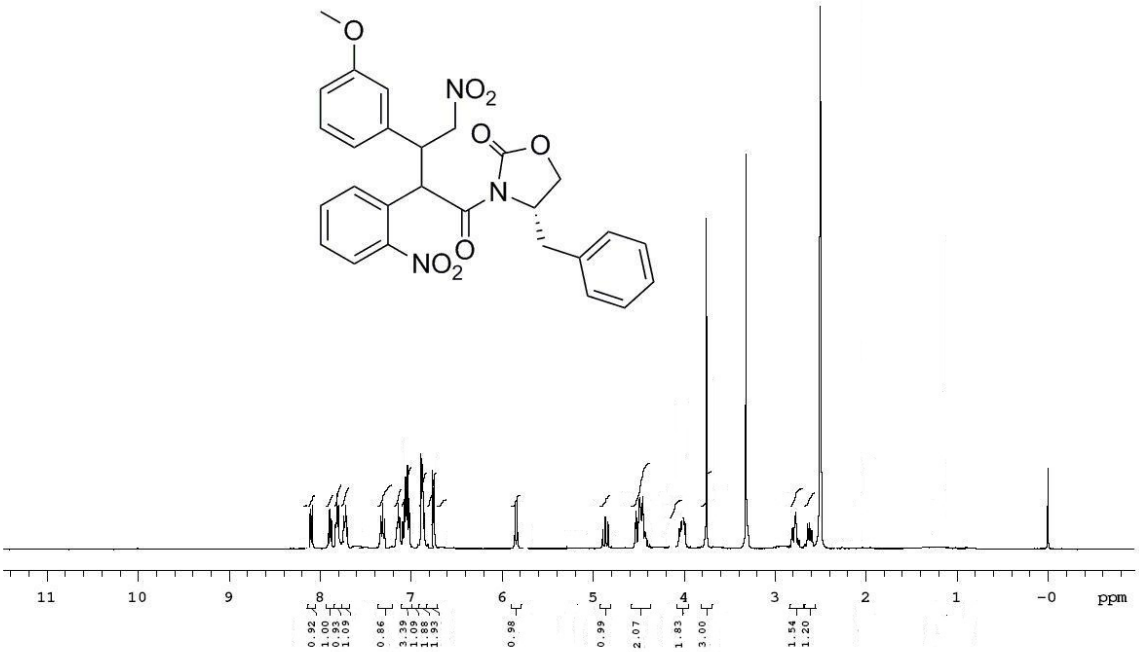
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 24 2012
129.164
77.414
77.000
76.571
Cl
NO2
O
O
N
O
F
200
180
160
140
120
100
80
60
40
20
ppm

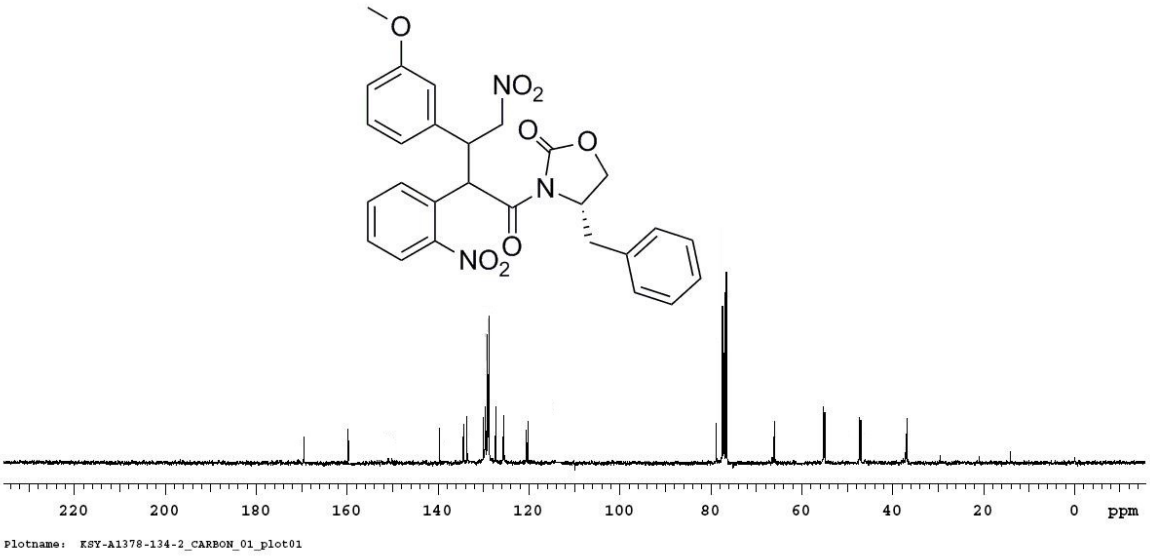
17


10
9
8
7
6
5
4
3
2
1
-0
ppm
1.83
1.06
3.30
1.85
1.04
0.94
2.83
0.98
0.99
1.98
1.05
1.98
2.88
1.01
1.00

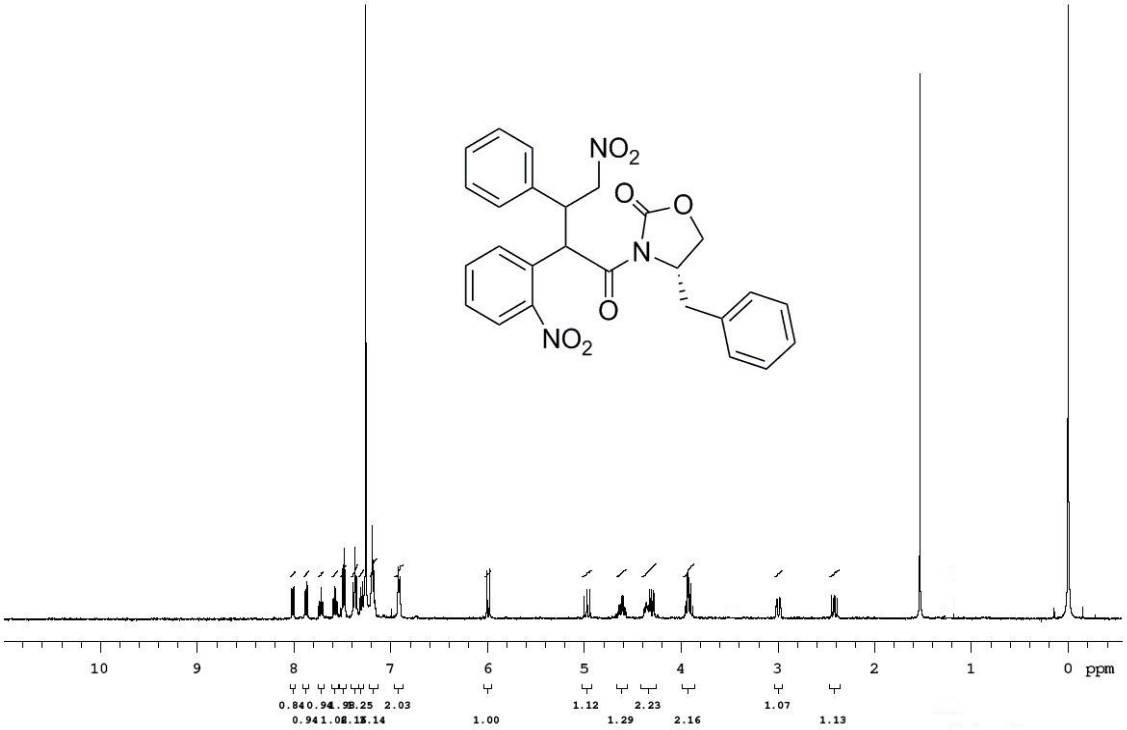

180
160
140
120
100
80
60
40
20
0
ppm

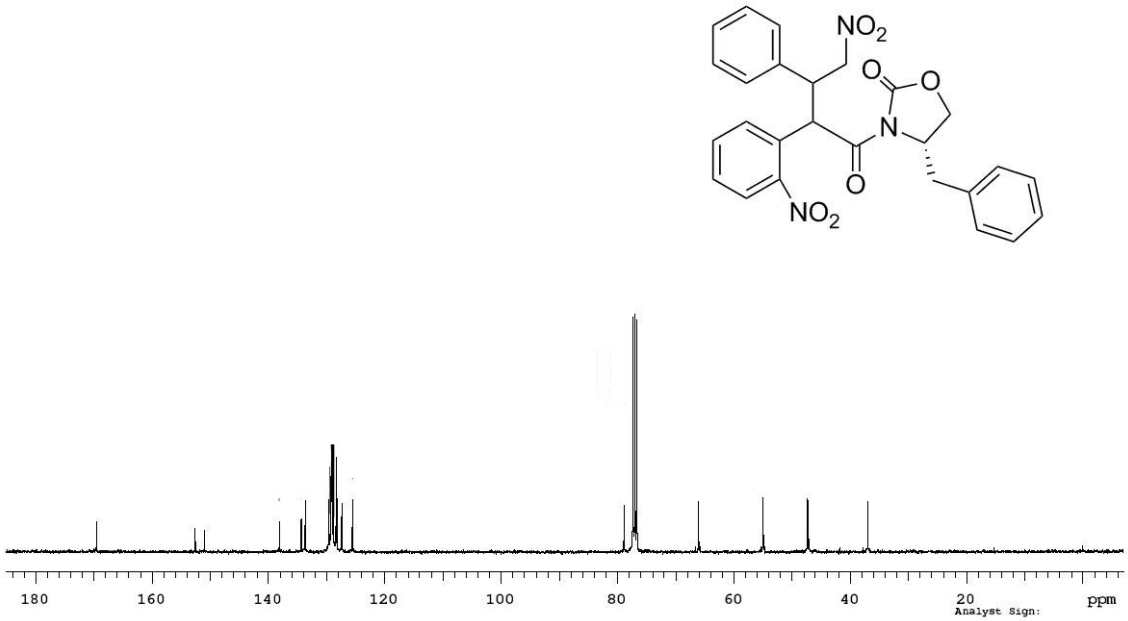
18


O
NO2
O
O
N
O
NO2
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.92
1.00
0.93
1.09
0.86
3.39
1.09
1.88
1.93
0.98
0.99
2.07
1.83
3.00
1.54
1.20

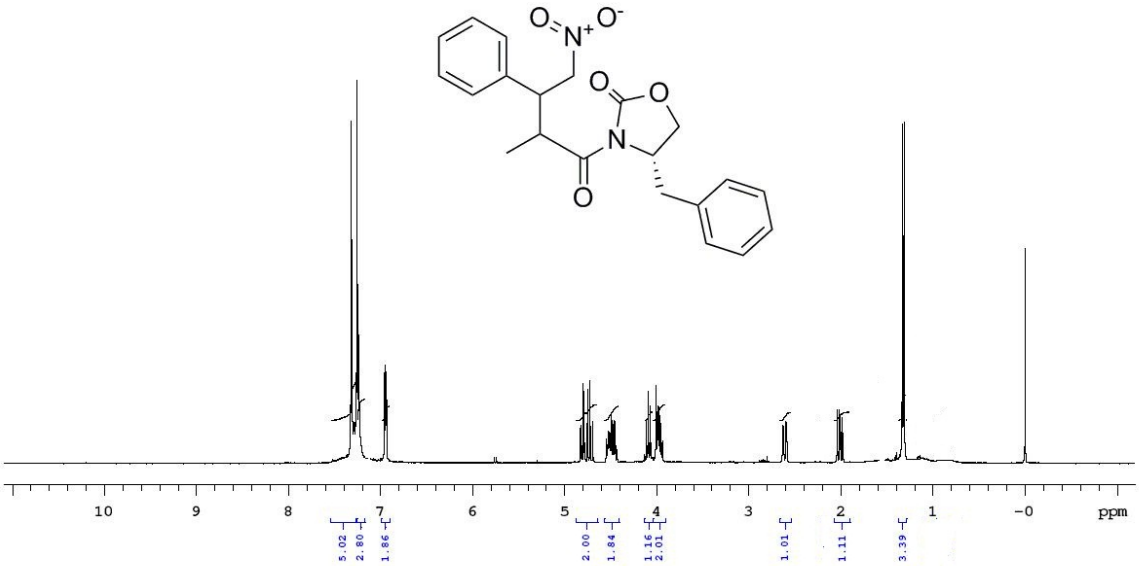

O
NO2
O
O
N
O
NO2
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Plotname: KSY-A1378-134-2_CARBON_01_plot01

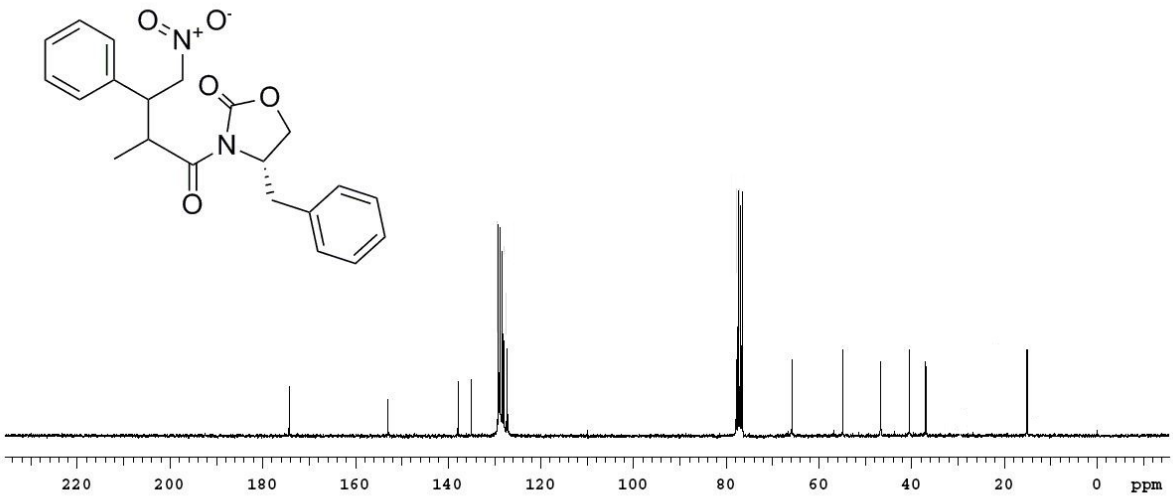
19


NO2
O
O
N
O
NO2
10
9
8
7
6
5
4
3
2
1
0 ppm
0.84 0.94 4.95 3.25 2.03
0.94 1.08 1.12 1.14
1.00
1.12
2.23
1.29
2.16
1.07
1.13

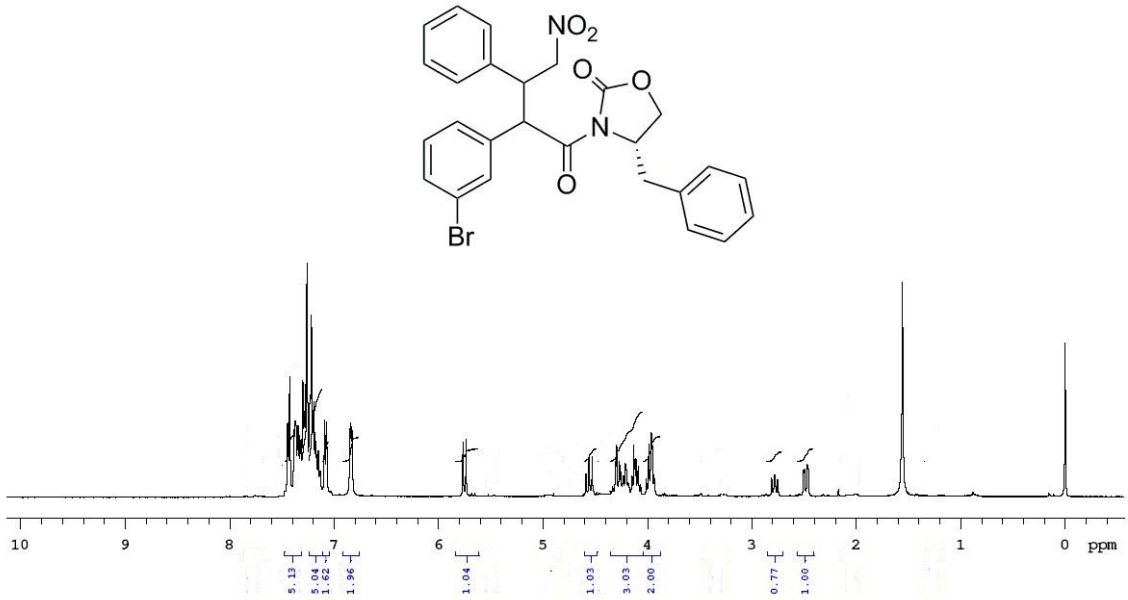

NO2
O
O
N
O
NO2
180
160
140
120
100
80
60
40
20
ppm
Analyst Sign:

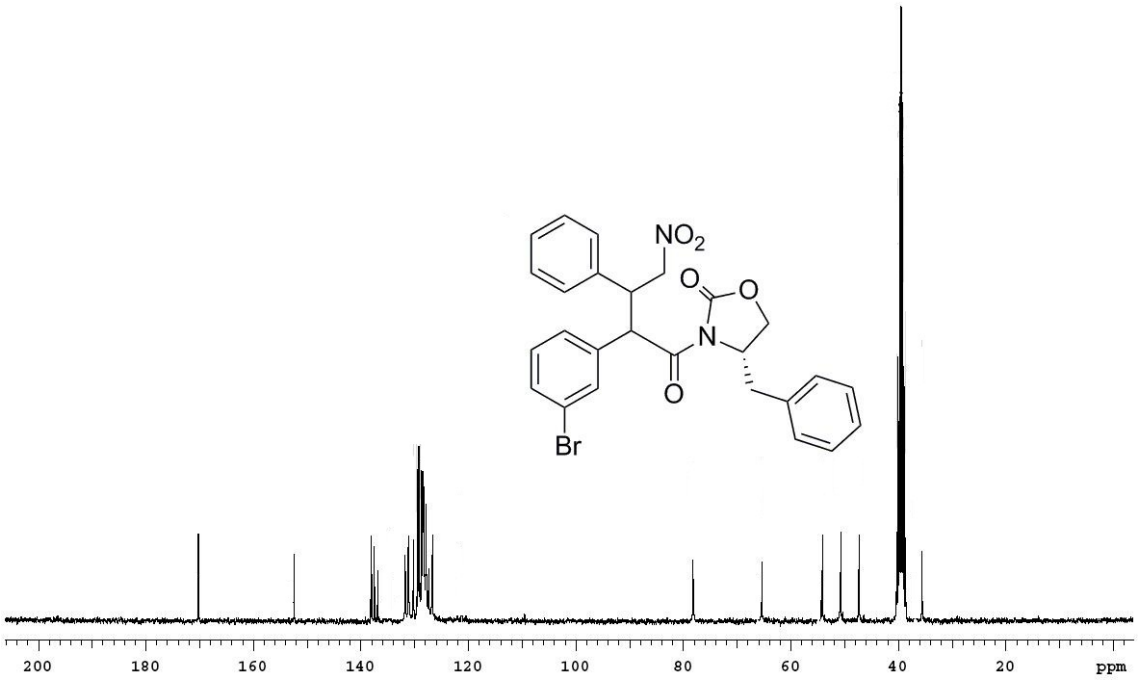
20


10
9
8
7
6
5
4
3
2
1
-0
ppm
5.02
2.80
1.86
2.00
1.84
1.16
2.01
1.01
1.11
3.39


220
200
180
160
140
120
100
80
60
40
20
0
ppm

21


NO2
O
O
N
O
Br
10
9
8
7
6
5
4
3
2
1
0
ppm
5.13
5.04
1.62
1.96
1.04
1.03
3.03
2.00
0.77
1.00


NO2
O
O
N
O
Br
200
180
160
140
120
100
80
60
40
20
ppm
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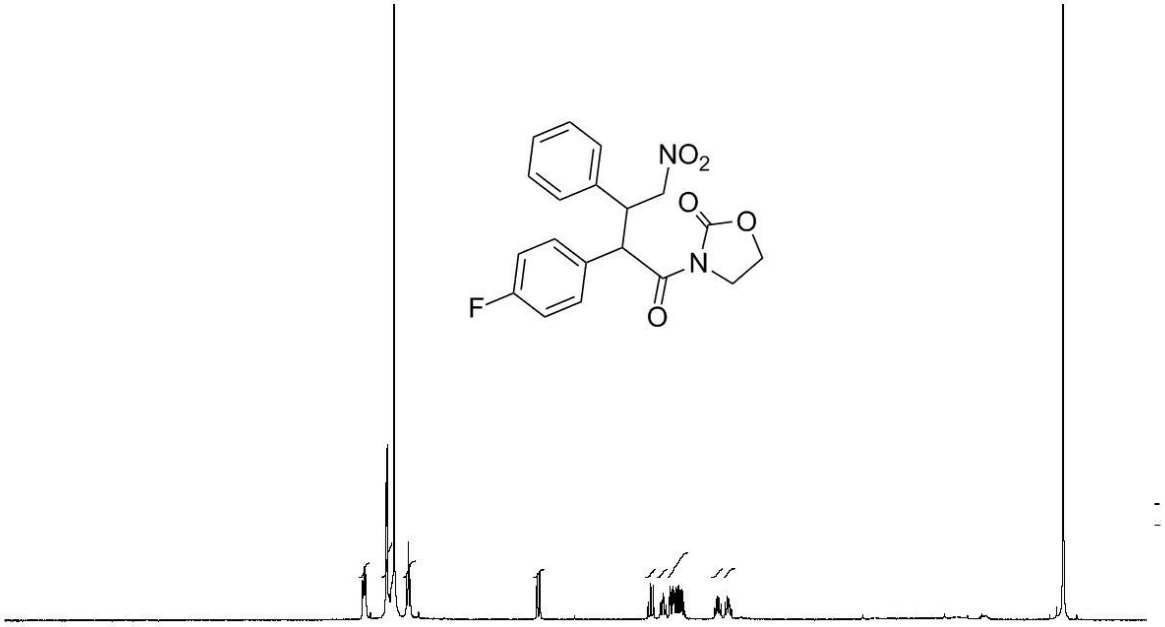
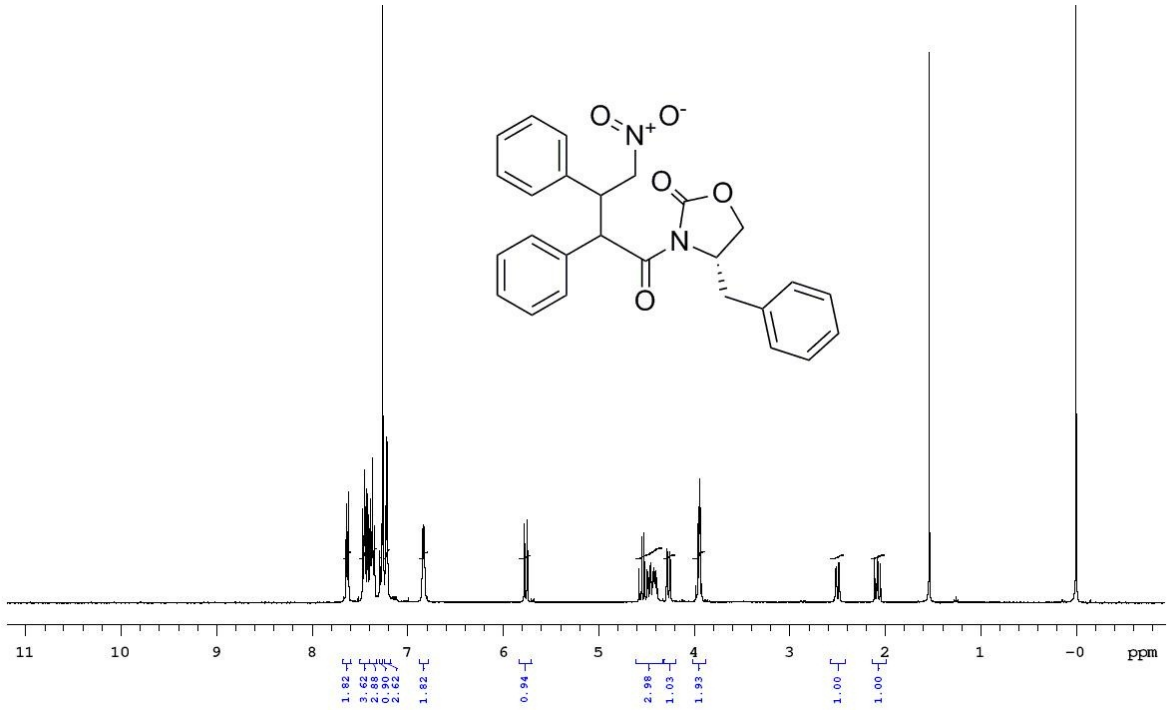
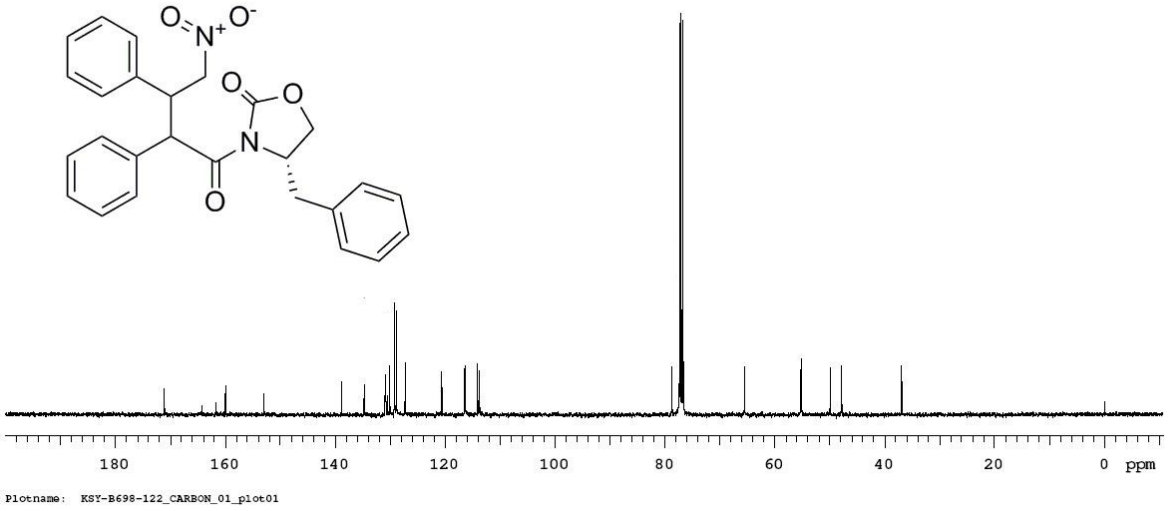
22



23


11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.82
3.62
2.88
0.90
2.62
1.82
0.94
2.98
1.03
1.93
1.00
1.00


180
160
140
120
100
80
60
40
20
0
ppm
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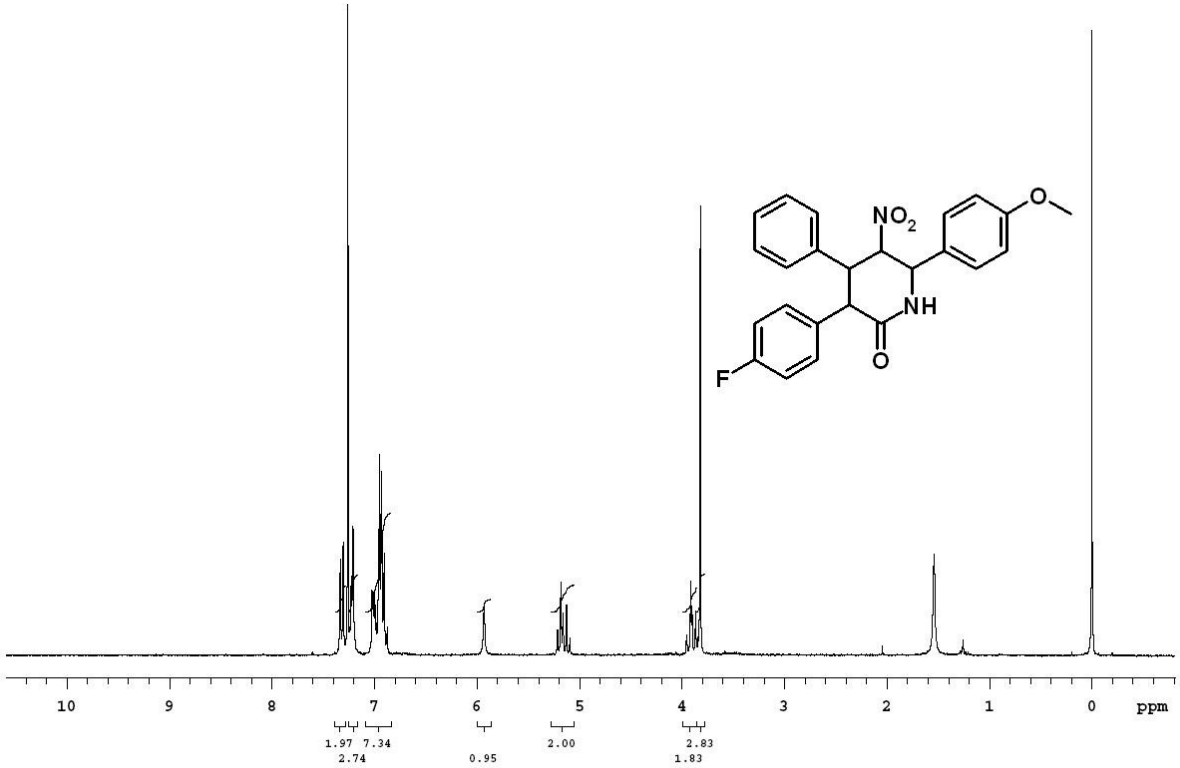
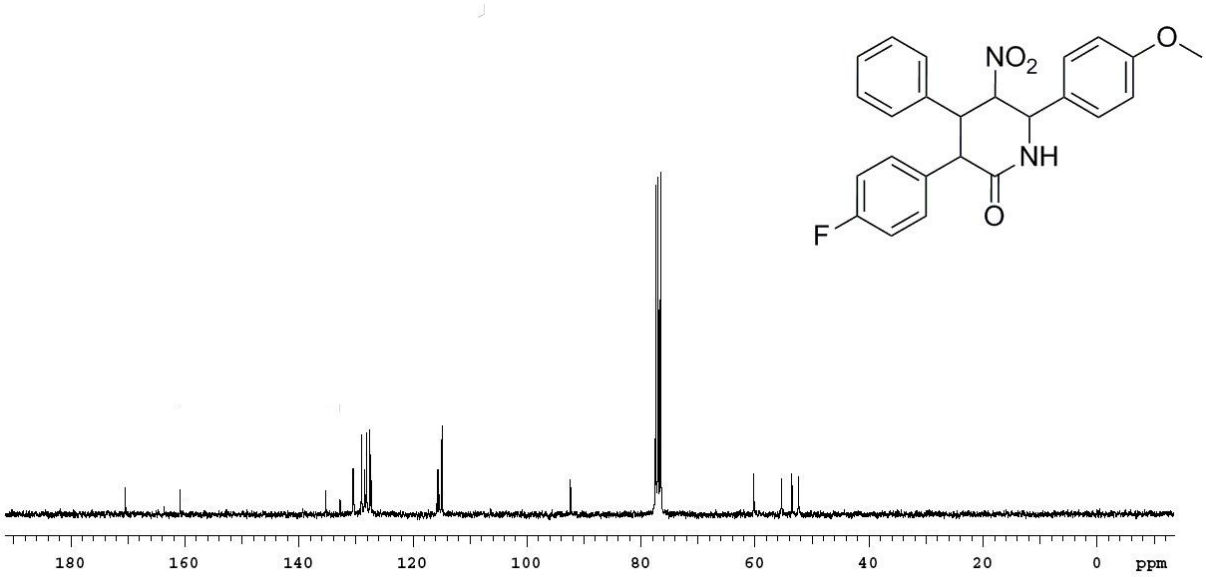
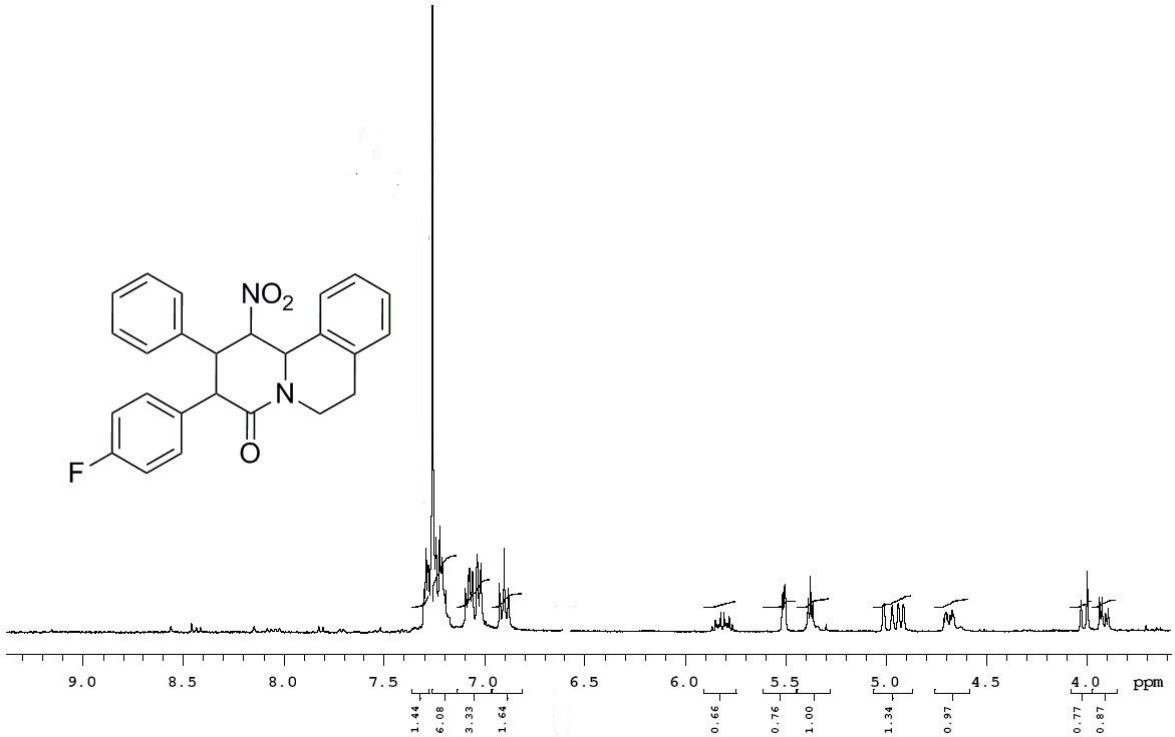
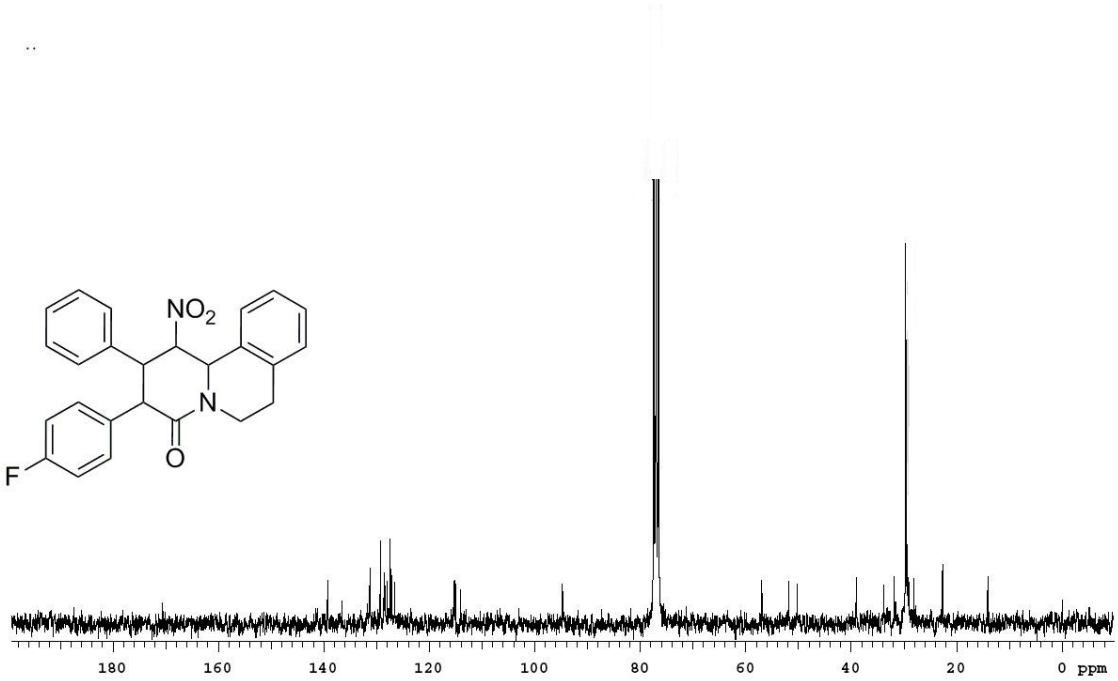
24


NO2
O
NH
O
F
10
9
8
7
6
5
4
3
2
1
0
ppm
1.97 7.34
2.74
0.95
2.00
2.83
1.83


NO2
O
NH
O
F
180
160
140
120
100
80
60
40
20
0
ppm

25





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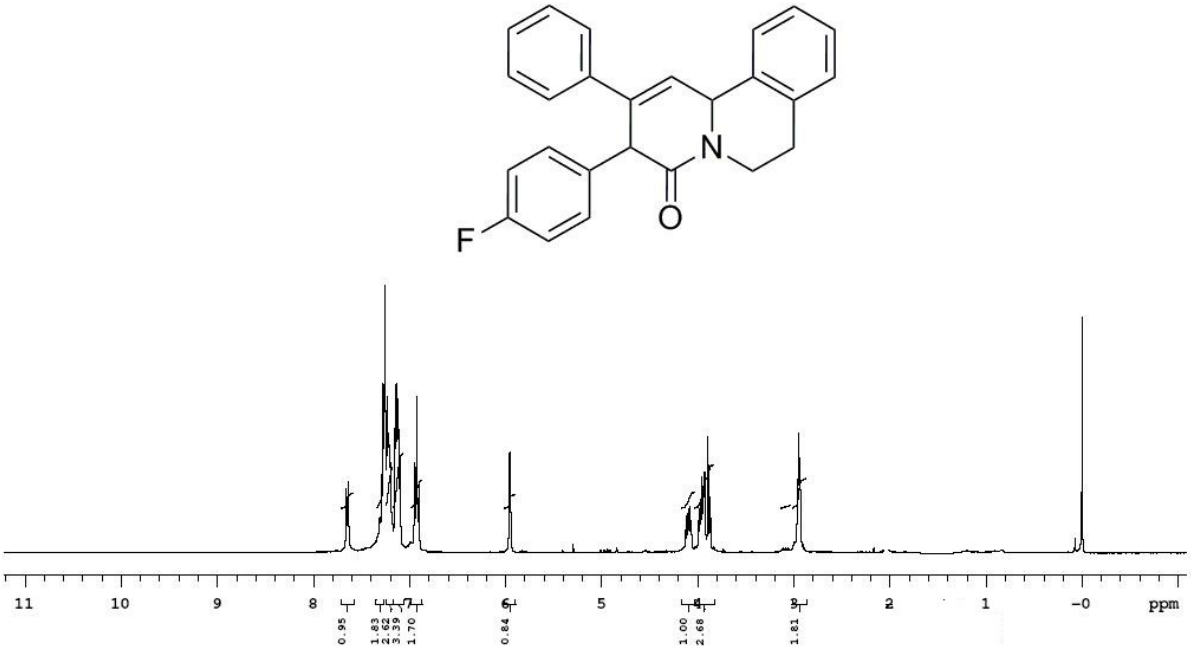
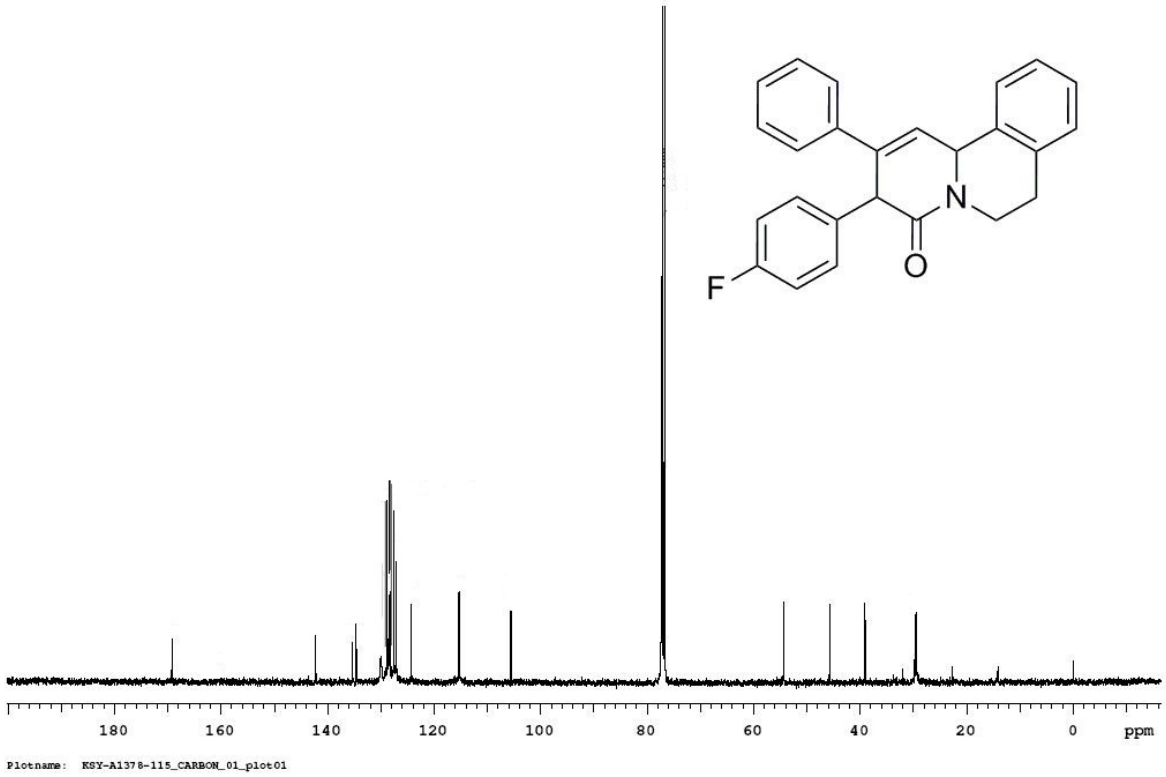
26


N
O
F
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.95
1.83
2.62
3.39
1.70
0.84
1.00
2.68
1.81


N
O
F
180
160
140
120
100
80
60
40
20
0
ppm
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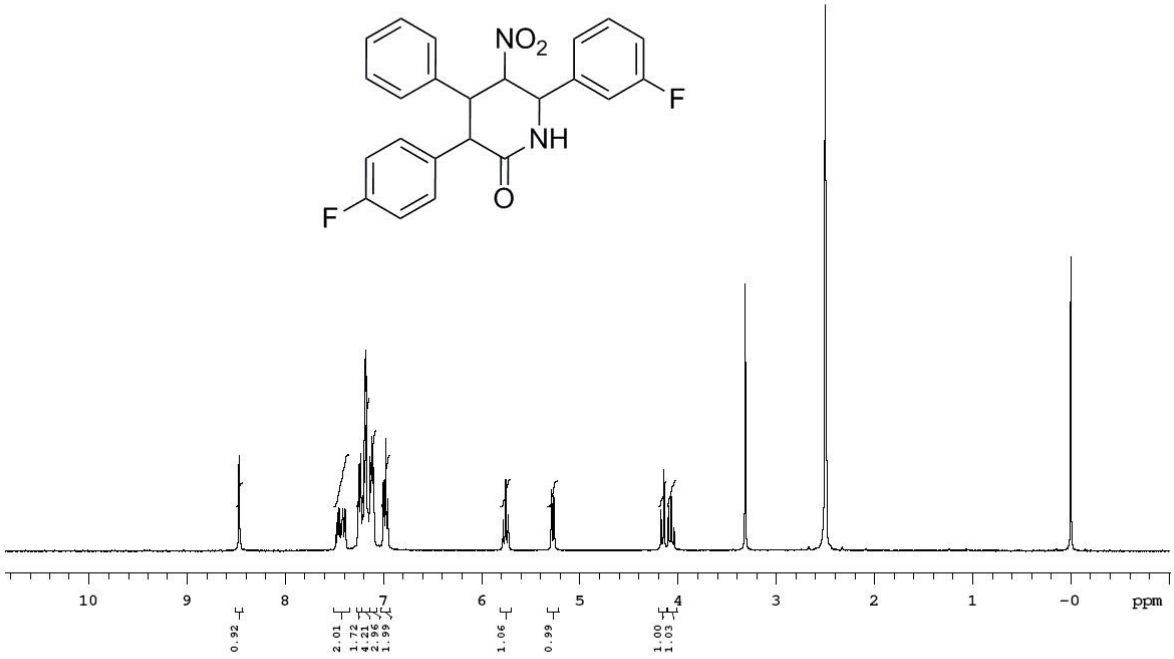
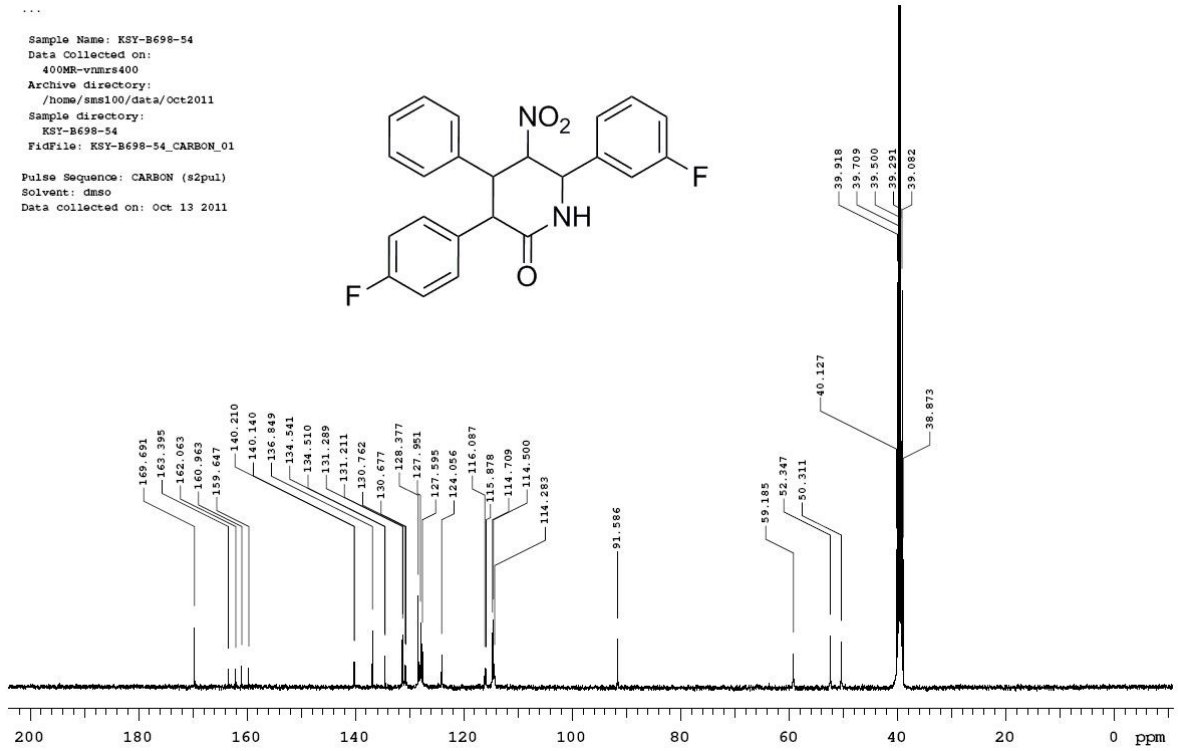
27


NO2
F
NH
O
F
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.92
2.01
1.72
4.21
2.96
1.99
1.06
0.99
1.00
1.03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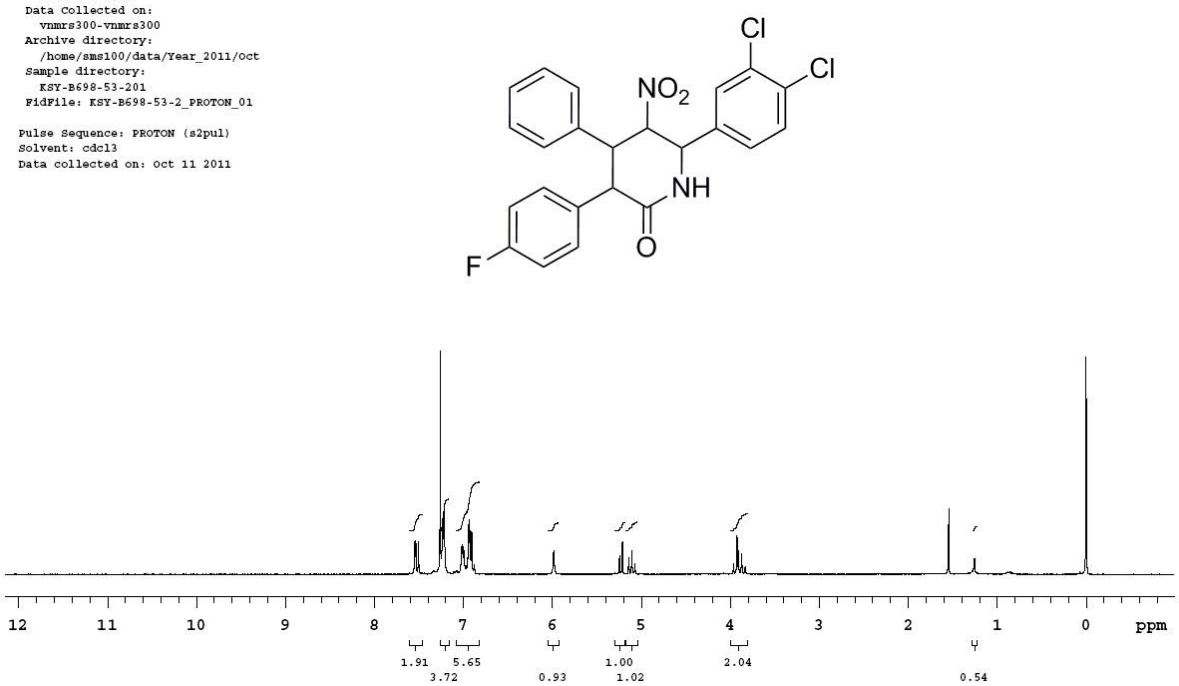
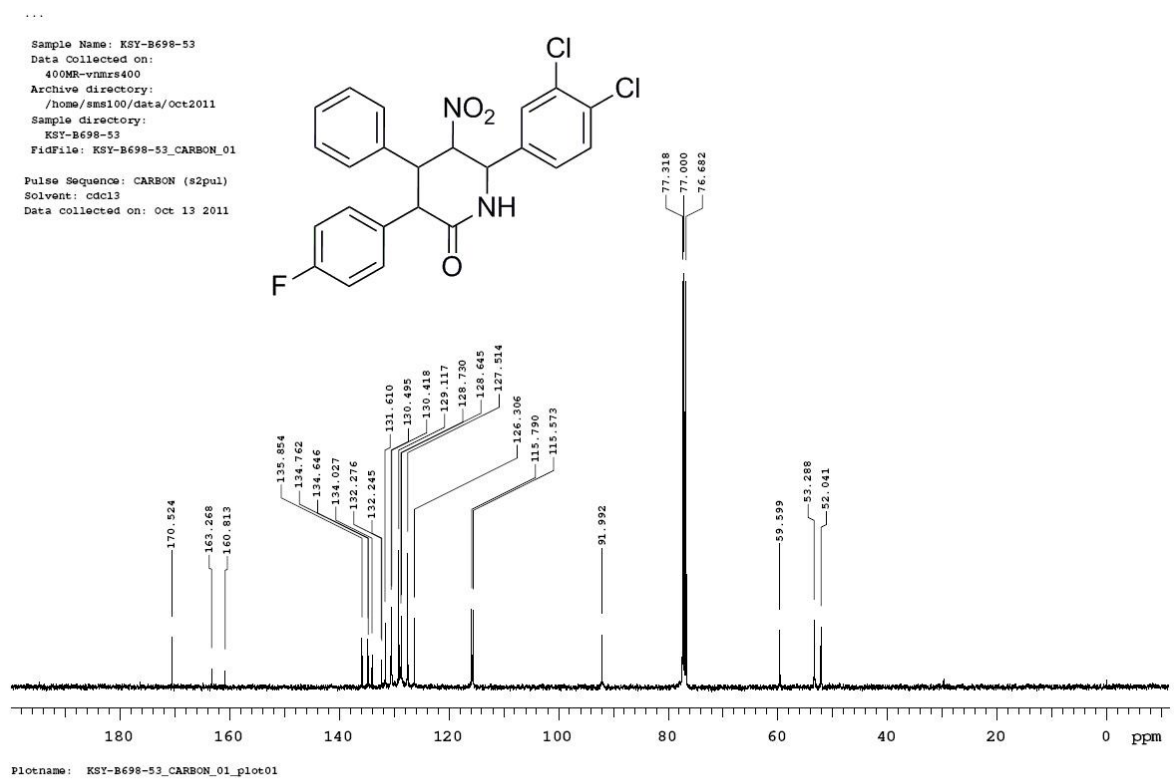
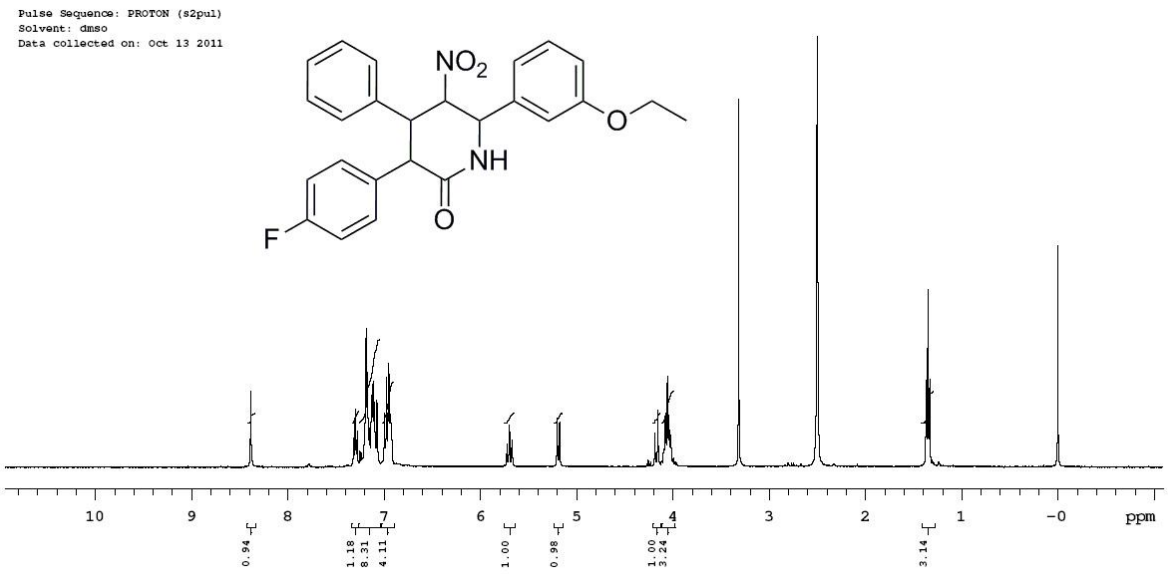
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Oct2011
Sample directory:
KSY-B698-54
FidFile: KSY-B698-54_CARBON_01
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Oct 13 2011
NO2
F
NH
O
F
39.918
39.709
39.500
39.291
39.082
40.127
38.873
169.691
163.395
162.063
160.963
159.647
140.210
140.140
136.849
134.541
134.510
131.289
131.211
130.762
130.677
128.377
127.951
127.595
124.056
116.087
115.878
114.709
114.500
114.283
91.586
59.185
52.347
50.311
200
180
160
140
120
100
80
60
40
20
0
ppm

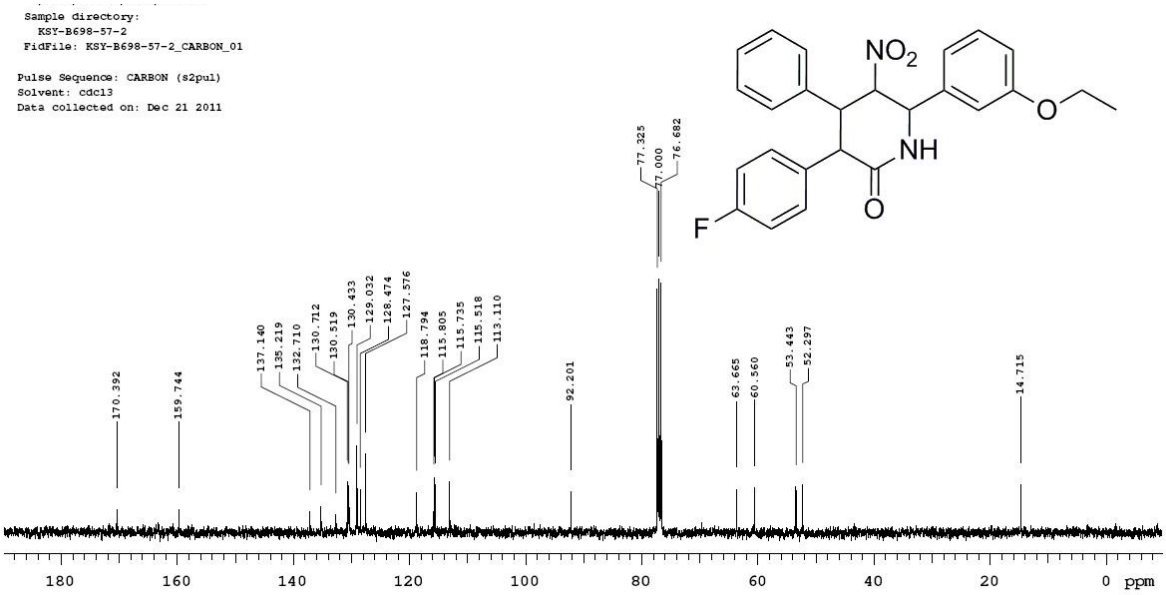
28


Data Collected on:
vnmrs300-vnmrs300
Archive directory:
/home/sms100/data/Year_2011/Oct
Sample directory:
KSY-B698-53-201
FidFile: KSY-B698-53-2_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 11 2011
Cl
Cl
NO2
NH
O
F
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.91 5.65 3.72 0.93 1.00 1.02 2.04 0.54


...
Sample Name: KSY-B698-53
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Oct2011
Sample directory:
KSY-B698-53
FidFile: KSY-B698-53_CARBON_01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 13 2011
Cl
Cl
NO2
NH
O
F
170.524
163.268
160.813
135.854
134.762
134.646
134.027
132.276
132.245
131.610
130.495
130.418
129.117
128.730
128.645
127.514
126.306
115.790
115.573
91.992
77.318
77.000
76.682
59.599
53.288
52.041
180 160 140 120 100 80 60 40 20 0 ppm
Plotname: KSY-B698-53_CARBON_01_plot01

29


Pulse Sequence: PROTON (s2pul)
Solvent: dmso
Data collected on: Oct 13 2011
NO2
O
NH
O
F
10 9 8 7 6 5 4 3 2 1 -0 ppm
0.94
1.18
8.31
4.11
1.00
0.98
1.00
3.24
3.14


Sample directory:
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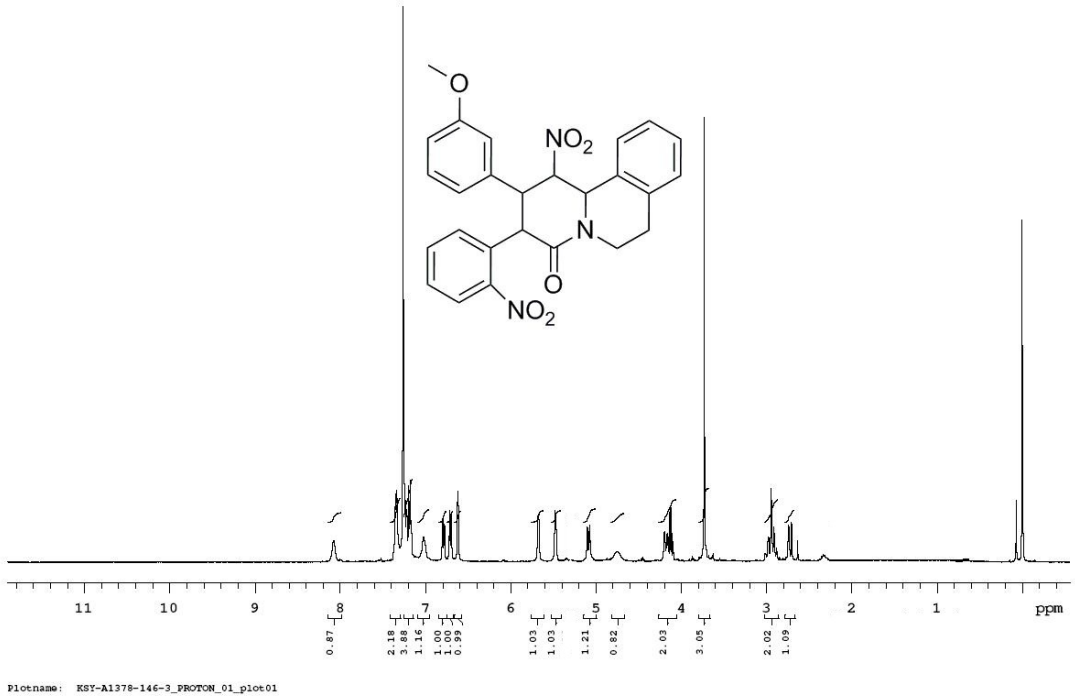
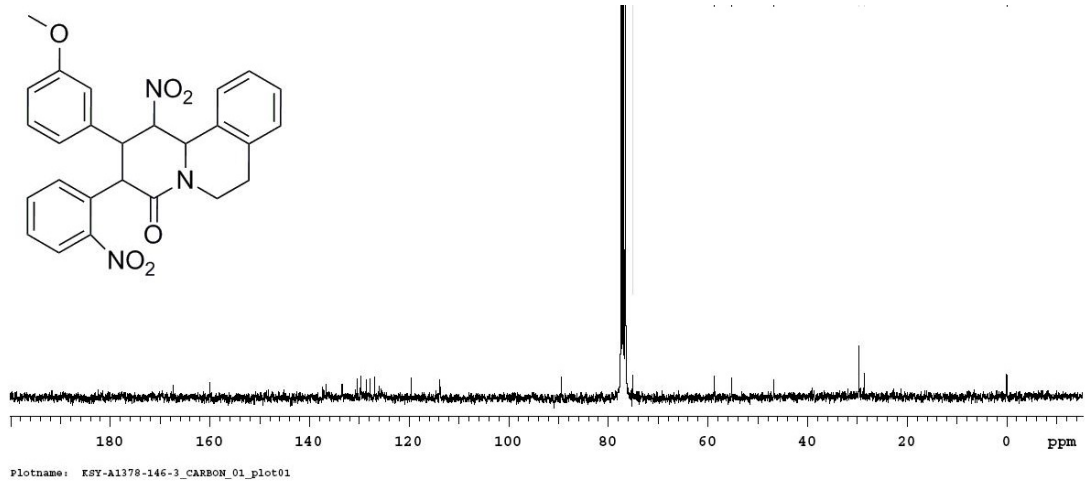
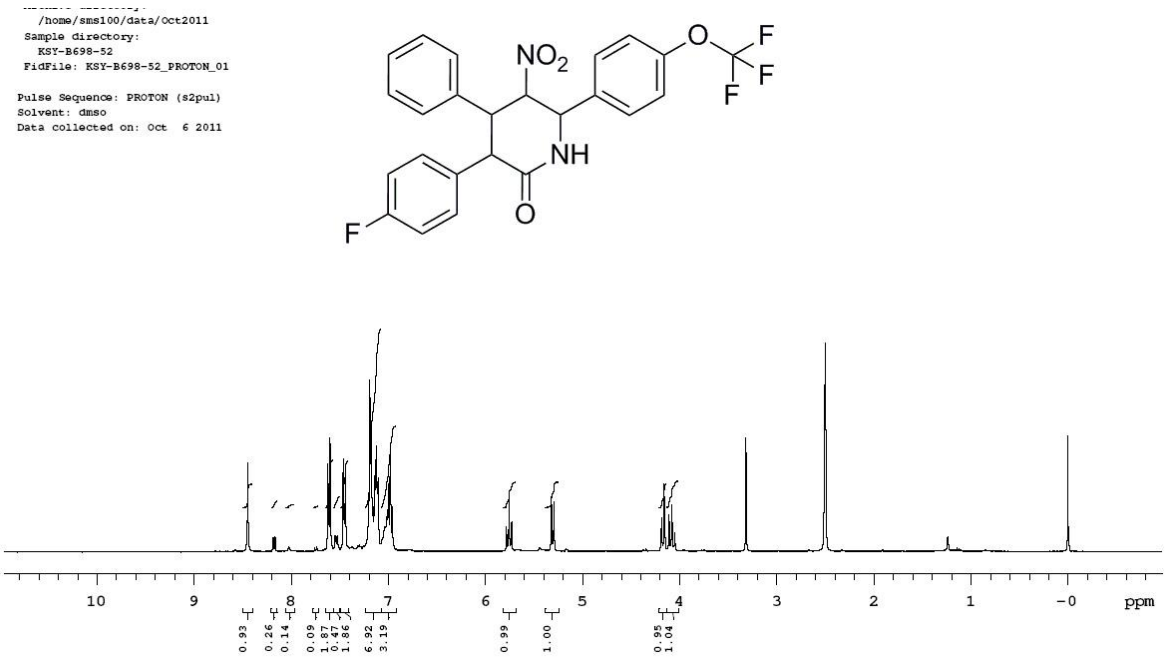
FidFile: KSY-B698-57-2_CARBON_01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 21 2011
NO2
O
NH
O
F
170.392
159.744
137.140
135.219
132.710
130.712
130.519
130.433
129.032
128.474
127.576
118.794
115.805
115.735
115.518
113.110
92.201
77.325
77.000
76.682
63.665
60.560
53.443
52.297
14.715
180 160 140 120 100 80 60 40 20 0 ppm

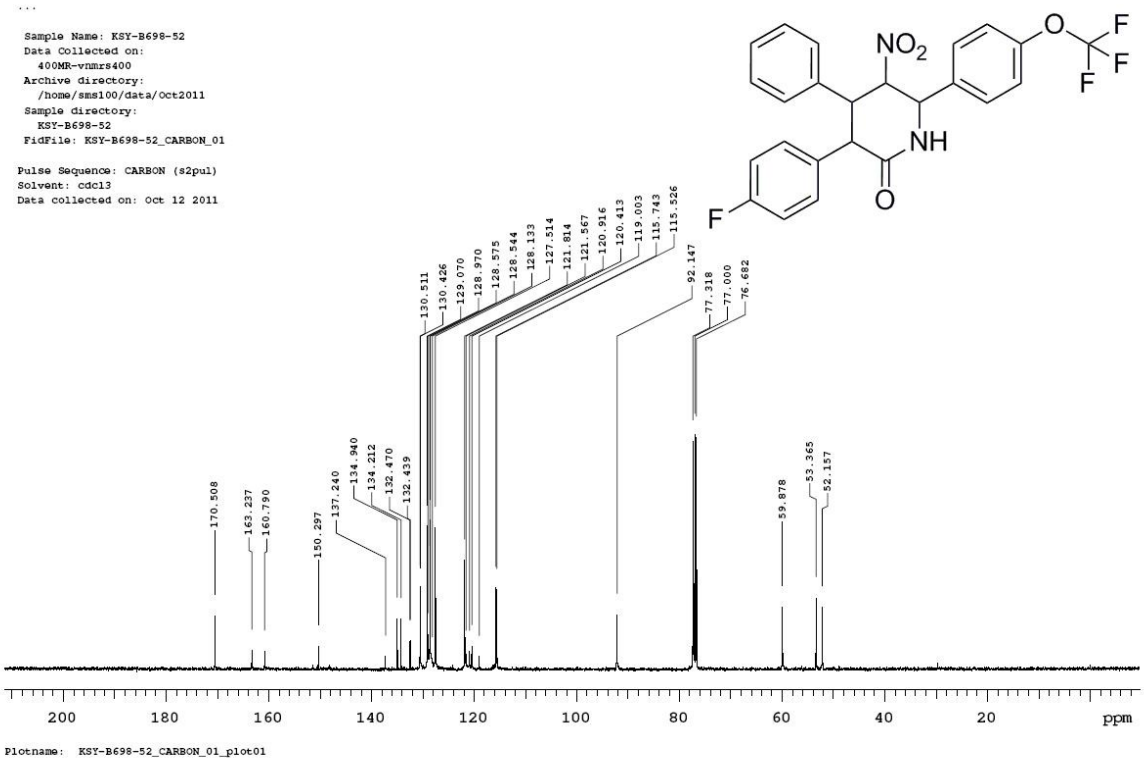
30


O
NO2
N
O
NO2
11
10
9
8
7
6
5
4
3
2
1
ppm
0.87
2.18
3.88
1.16
1.00
1.00
0.99
1.03
1.03
1.21
0.82
2.03
3.05
2.02
1.09
Plotname: KSY-A1378-146-3_PROTON_01_plot01

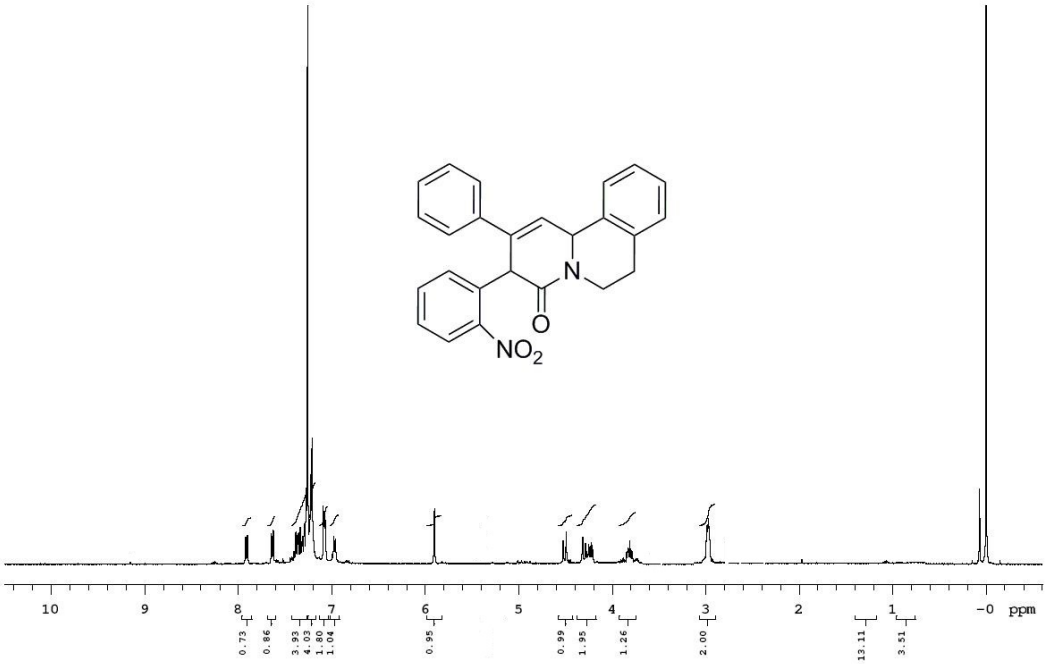

O
NO2
N
O
NO2
180
160
140
120
100
80
60
40
20
0
ppm
Plotname: KSY-A1378-146-3_CARBON_01_plot01

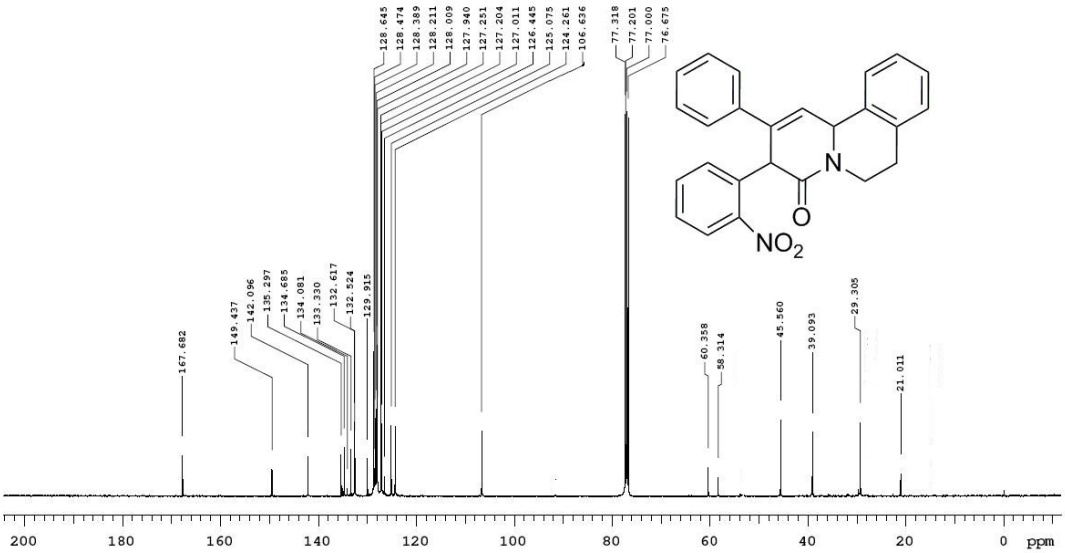
31


/home/sms100/data/Oct2011
Sample directory:
KSY-B698-52
FidFile: KSY-B698-52_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: dmso
Data collected on: Oct 6 2011
NO2
O
F
F
F
NH
O
F
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.93
0.26
0.14
0.09
1.87
0.47
1.86
6.92
3.19
0.99
1.00
0.95
1.04

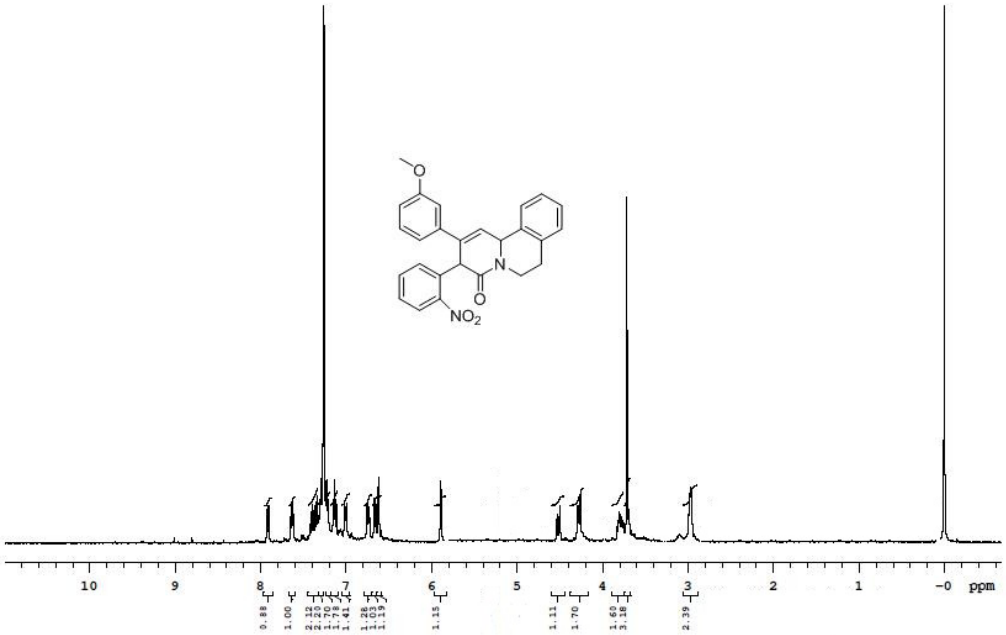

Sample Name: KSY-B698-52
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Oct2011
Sample directory:
KSY-B698-52
FidFile: KSY-B698-52_CARBON_01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 12 2011
NO2
O
F
F
F
NH
O
F
170.508
163.237
160.790
150.297
137.240
134.940
134.212
132.470
132.439
130.511
130.426
129.070
128.970
128.575
128.544
128.133
127.514
121.814
121.567
120.916
120.413
119.003
115.743
115.526
92.147
77.318
77.000
76.682
59.878
53.365
52.157
200
180
160
140
120
100
80
60
40
20
ppm
Plotname: KSY-B698-52_CARBON_01_plot01

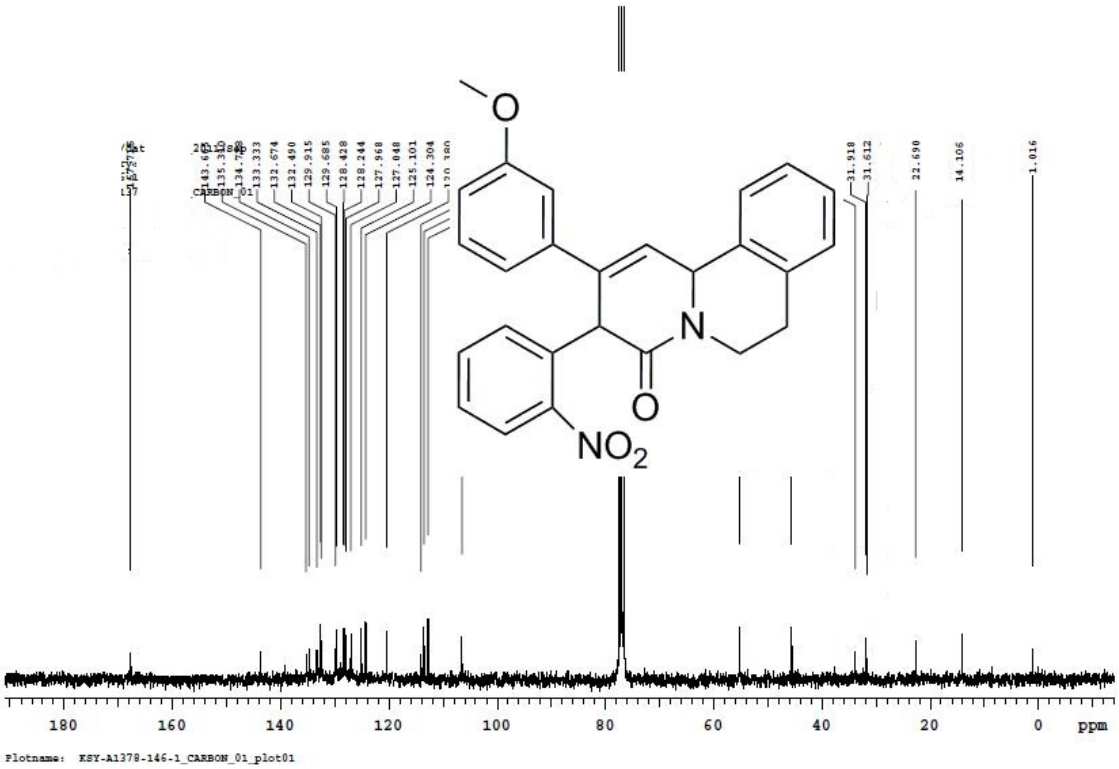
32


N
O
NO2
10
9
8
7
6
5
4
3
2
1
-0 ppm
0.73
0.86
3.93
4.03
1.80
1.04
0.95
0.99
1.95
1.26
2.00
13.11
3.51

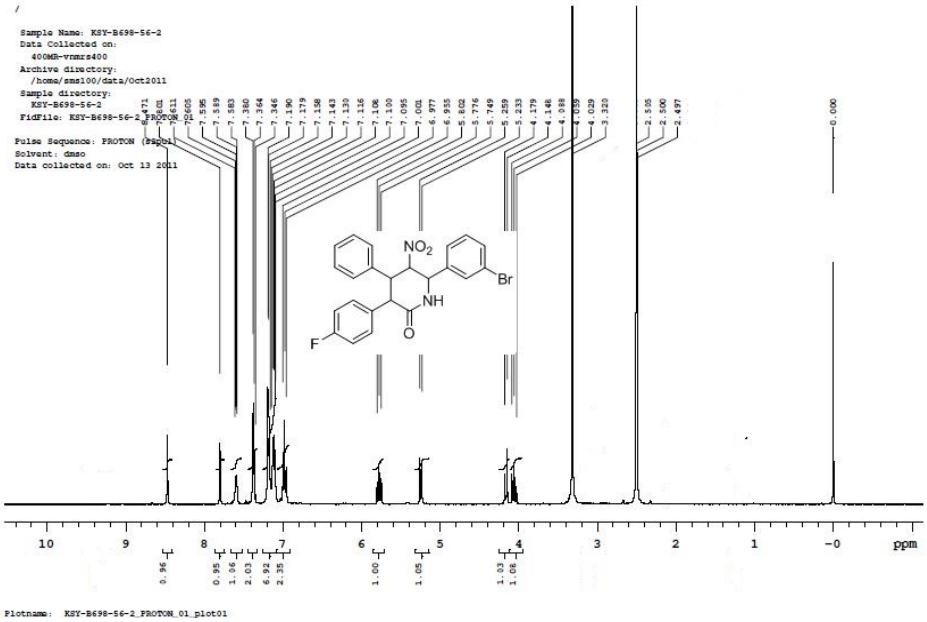

128.645
128.474
128.389
128.211
128.009
127.940
127.251
127.204
127.011
126.445
125.075
124.261
106.636
77.318
77.201
77.000
76.675
167.682
149.437
142.096
135.297
134.685
134.081
133.330
132.617
132.524
129.915
60.358
58.314
45.560
39.093
29.305
21.011
N
O
NO2
200
180
160
140
120
100
80
60
40
20
0 ppm

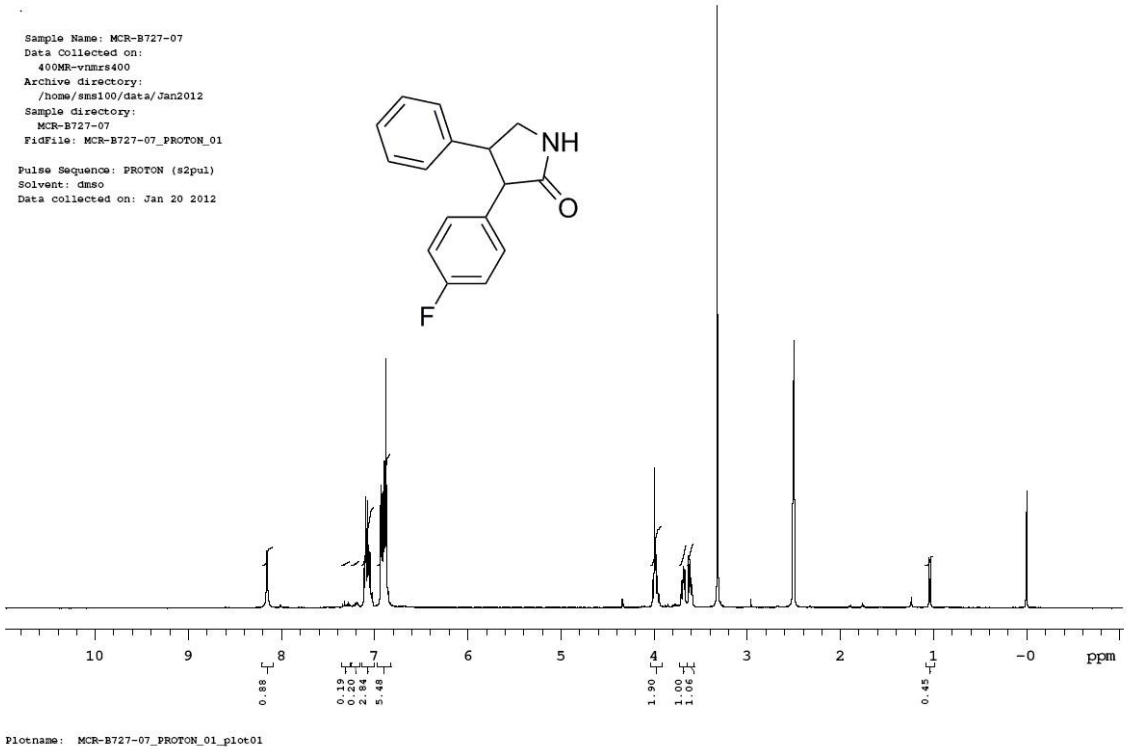
33

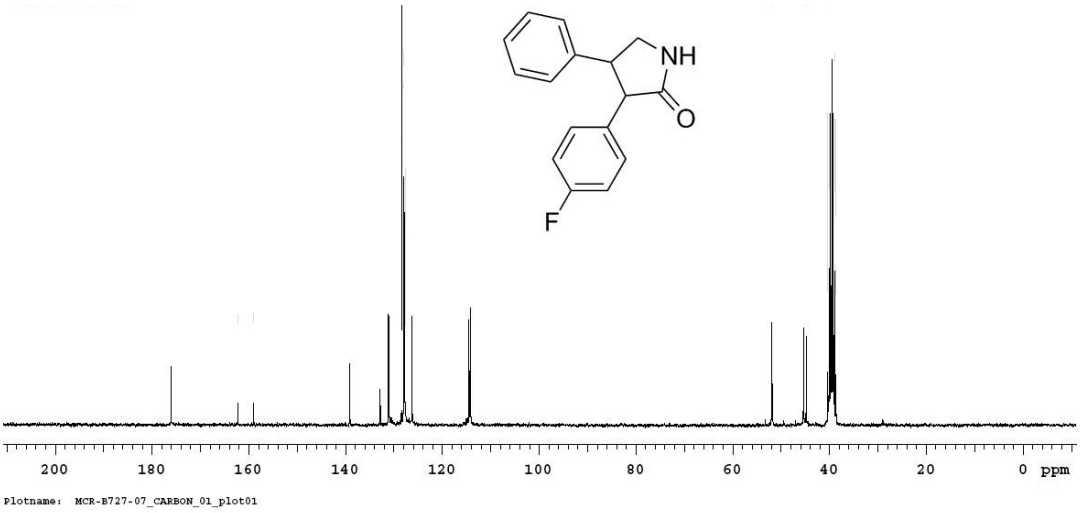

O
N
O
NO2
10
9
8
7
6
5
4
3
2
1
-0
ppm


O
N
O
NO2
CARBON_01
31.918
31.612
22.690
14.106
1.016
180
160
140
120
100
80
60
40
20
0
ppm
Plotname: KSY-A1378-146-1_CARBON_01_plot01

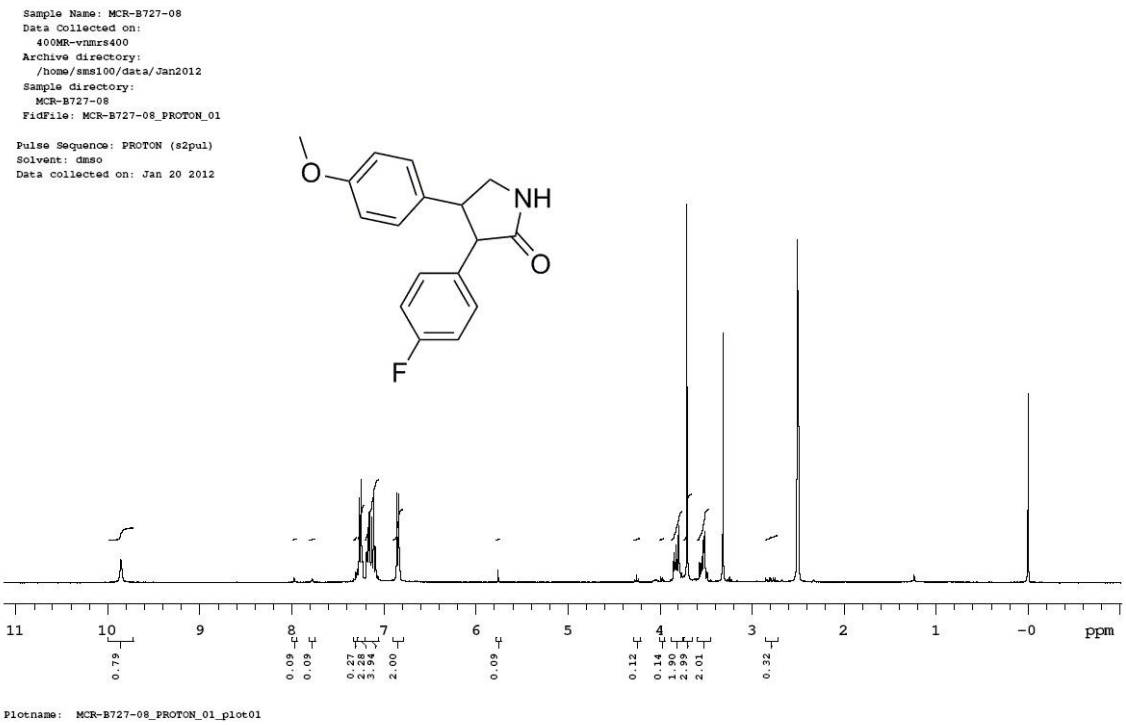
37

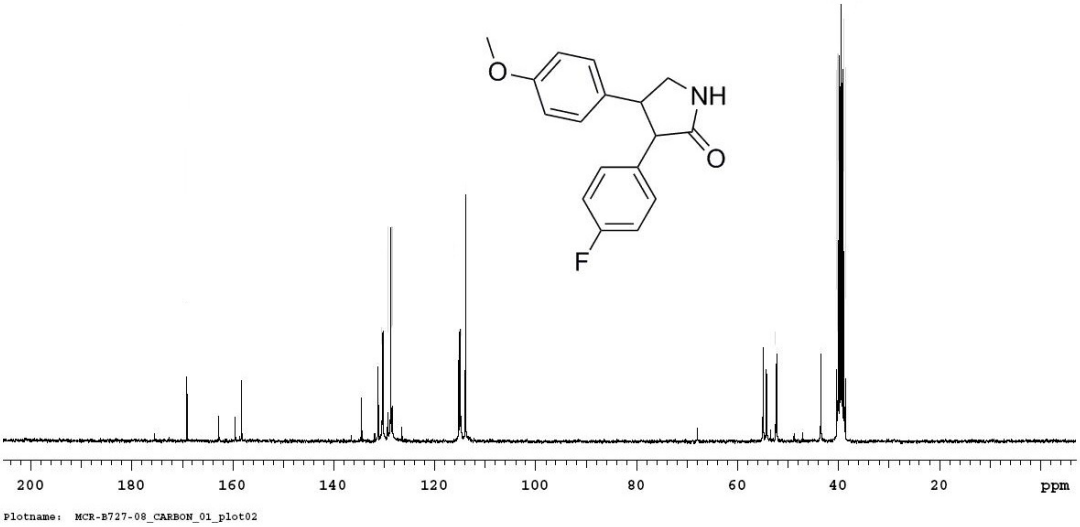

Sample Name: KSY-B698-56-2
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Oct2011
Sample directory:
KSY-B698-56-2
FidFile: KSY-B698-56-2_PROTON_01
Pulse Sequence: PROTON (zgpg)
Solvent: dmso
Data collected on: Oct 13 2011
NO2
Br
NH
O
F
ppm
Plotname: KSY-B698-56-2_PROTON_01_plot01


Sample Name: MCR-B727-07
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Jan2012
Sample directory:
MCR-B727-07
FidFile: MCR-B727-07_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: dmso
Data collected on: Jan 20 2012
NH
O
F
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.88
0.19
0.20
2.84
5.48
1.90
1.00
1.06
0.45
Plotname: MCR-B727-07_PROTON_01_plot01

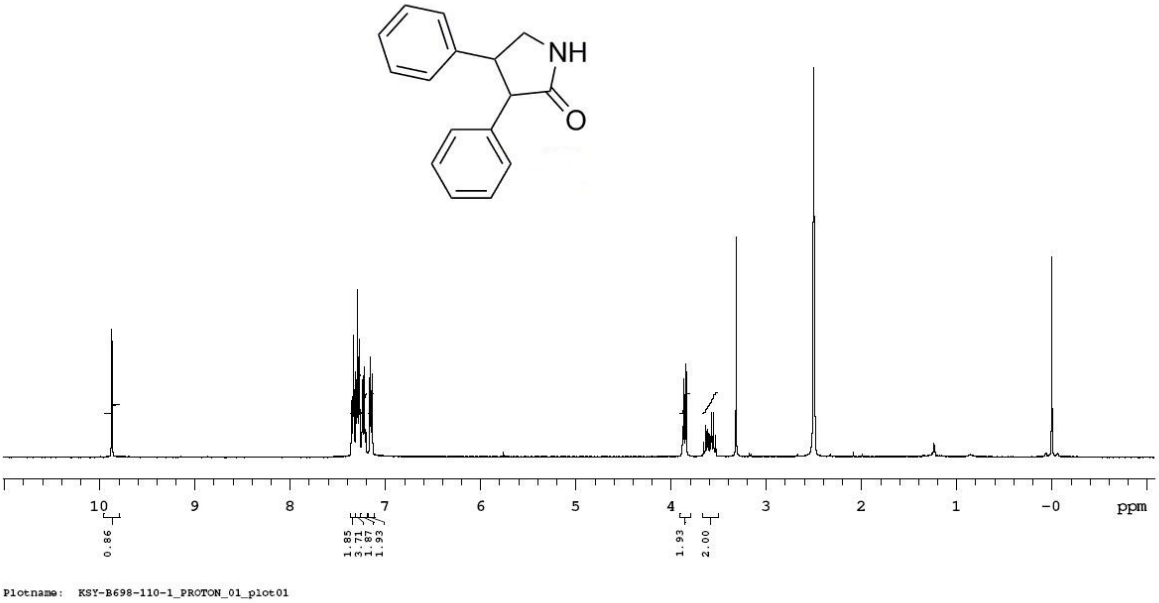

NH
O
F
200
180
160
140
120
100
80
60
40
20
0 ppm
Plotname: MCR-B727-07_CARBON_01_plot01

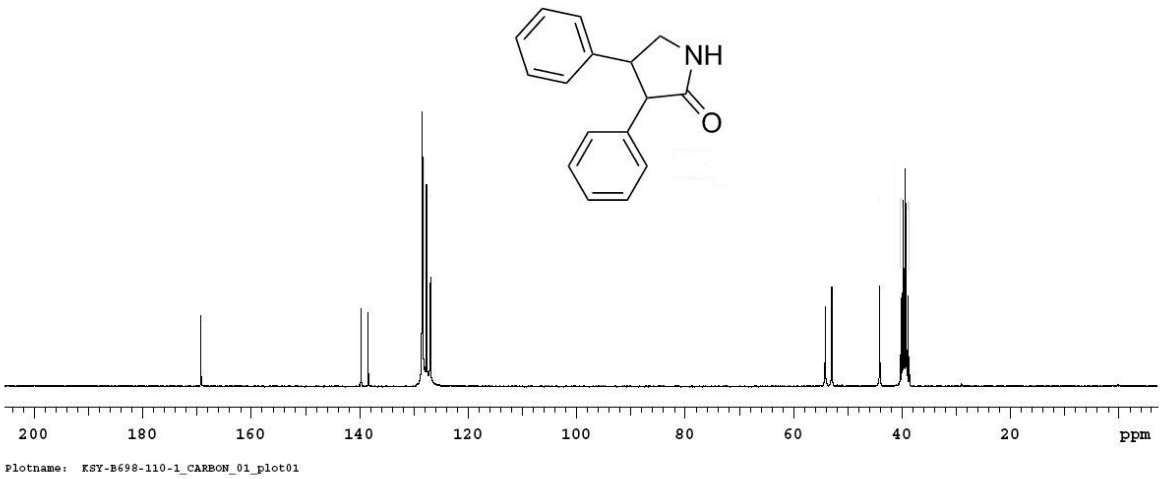
35


Sample Name: MCR-B727-08
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Jan2012
Sample directory:
MCR-B727-08
FidFile: MCR-B727-08_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: dmso
Data collected on: Jan 20 2012
O
NH
O
F
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.79
0.09
0.09
0.27
2.28
3.94
2.00
0.09
0.12
0.14
1.90
2.99
2.01
0.32
Plotname: MCR-B727-08_PROTON_01_plot01


O
NH
O
F
200
180
160
140
120
100
80
60
40
20
ppm
Plotname: MCR-B727-08_CARBON_01_plot02

36


NH
O
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.86
1.95
3.71
1.87
1.93
1.93
2.00
Plotname: KSY-B698-110-1_PROTON_01_plot01


NH
O
200
180
160
140
120
100
80
60
40
20
ppm
Plotname: KSY-B698-110-1_CARBON_01_plot01

22

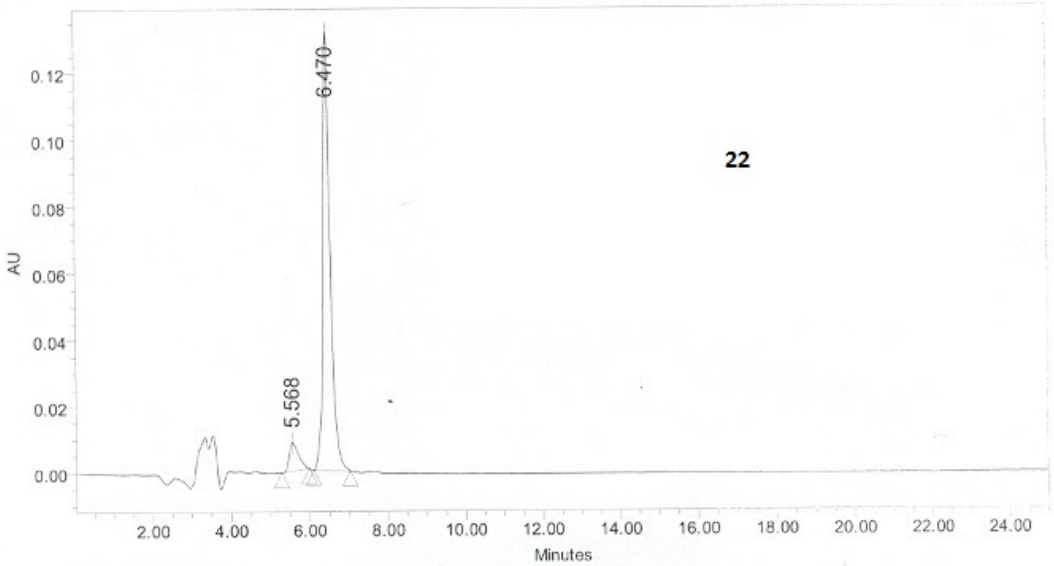
CHIRAL PAK IA_RPT

SAMPLE INFORMATION

Injection Volume :	15.00 ul	Proc. Chnl. Descr :	PDA 215.0 nm
Run Time :	25.0 Minutes	System Name :	ANL_MCL_HPLC_02

HPLC CONDITIONS

Column :	CHIRAL PAK IA 4.6 X 250mm , 5µm
Colunm ID :	ANL_CHIRAL _110
Mobile Phase :	A= n-HEXANE ,B=IPA
ISOCRATIC :	60:40
Flow Rate :	1.0 ml/min
Column Temp :	NA
Diluent :	Ethanol



Peak Results

	RT	Area	% Area
1	5.568	143303	7.87
2	6.470	1677926	92.13

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GVK BIOSCIENCES PVT LTD
ANALYTICAL REPORT - MCL-III

SAMPLE INFORMATION

Injection Volume: 2.00 ul
Vial: 13
Run Time: 20.0 Minutes

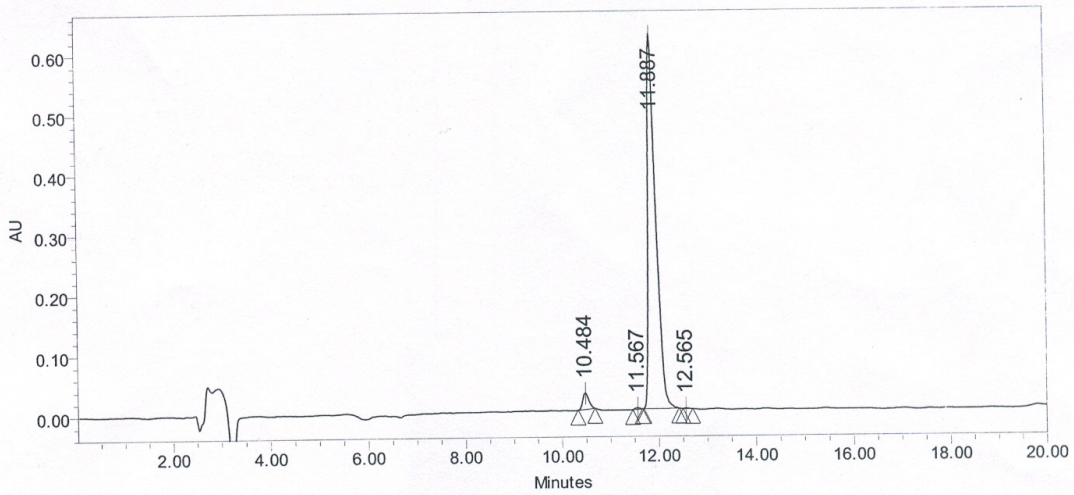
Acquired By : Analyst
Date Acquired : 8/2/2011 3:12:56 PM IST
Proc. Chnl. Descr : PDA 215.0 nm Blank Subtracted
System Name : ANL_HPLC_07

CONDITIONS

Column : SUNFIREC18 250x4.6 mm , 5 µm
Column ID : ANL_C158
Mobile Phase : A=ACN,B=0.1%TFA
T/%B : 0/80,2/80,6/20,18/20,19/80,20/80
Flow Rate : 1.0ml/min
Column Temp : NA
Diluent : MEOH

2

Purity Chromatogram



Peak Results

	RT	Area	% Area
1	10.484	226393	3.12
2	11.567	16260	0.22
3	11.887	7005281	96.59
4	12.565	4446	0.06

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GVK BIOSCIENCES PVT LTD
ANALYTICAL REPORT - MCL-III

SAMPLE INFORMATION

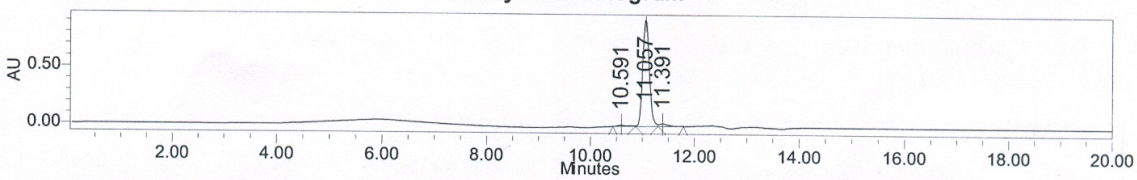
Sample Name:	KSY-B698-COMPOUND-15	Acquired By	Analyst
Injection Volume:	2.00 ul	Date Acquired	8/29/2012 4:00:48 PM IST
Vial:	3	Proc. Chnl. Descr	Channel Name 215.0nm
Run Time :	20.0 Minutes	System Name	:ANL_MCL_HPLC_2

HPLC CONDITIONS

Column :YMC TRIART C18 (4.6 X 150mm) 5µm
Mobile Phase :B:0.01M Ammonium acetate(Aq) : D:MeOH
Time of %D : 0/10,2/10,6/90,18/90,19/10,20/10
Flow Rate : 1.0 ml/min
Column Temp : Ambient
Diluent : ACN

15

Purity chromatogram



Sample Name KSY-B698-COMPOUND-15; Vial 3; Injection 1; Channel W2996 ; Date Acquired 8/29/2012 4:00:48 PM IST

Processed Channel: PDA 215.0 nm Blank Subtracted from BLANK, Vial 1 Inj. 1

	Retention Time (min)	Area	% Area
1	10.59	148745	1.74
2	11.06	8130103	95.02
3	11.39	277020	3.24

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ANALYTICAL REPORT
GVK BIOSCIENCES PVT LTD

SAMPLE INFORMATION

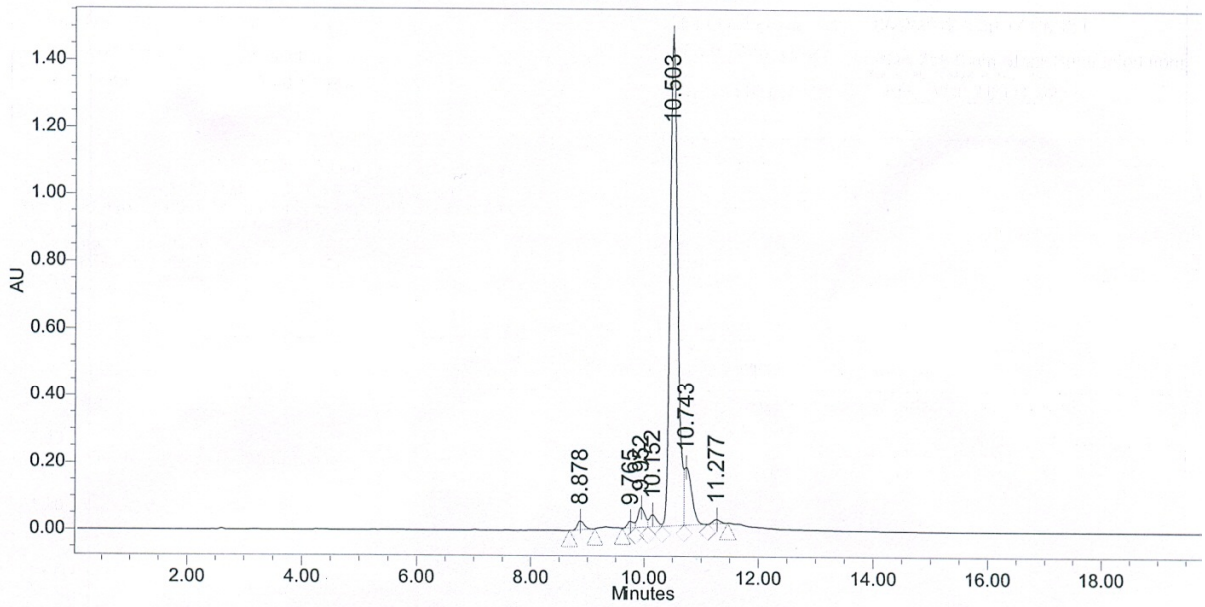
Sample Name :	KSY-B698-COMPOUND-16	Acquired By :	Analyst
Vial No :	5	Date Acquired :	8/29/2012 5:59:17 PM IST
Injection Volume :	2.00 ul	Proc. Chnl. Descr :	PDA 215.0 nm Blank Subtracted from
Run Time :	20.0 Minutes	System Name :	ANL_MCL_HPLC_02

HPLC CONDITIONS

Column :YMC TRAIT C18(4.6 X 250mm) 5µm
Mobile Phase :B:0.01M Ammonium acetate(Aq) : D:MeOH
Time of %D : 0/10,2/10,6/90,18/90,19/10,20/10
Flow Rate : 1.0 ml/min
Column Temp : Ambient
Diluent : ACN

16

HPLC purity chromatogram



Peak Results

	RT	Area	% Area
1	8.878	213924	1.30
2	9.765	154077	0.93
3	9.952	502215	3.04
4	10.152	326792	1.98
5	10.503	13607221	82.44
6	10.743	1590657	9.64
7	11.277	110240	0.67

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ANALYTICAL REPORT
GVK BIOSCIENCES PVT LTD

SAMPLE INFORMATION

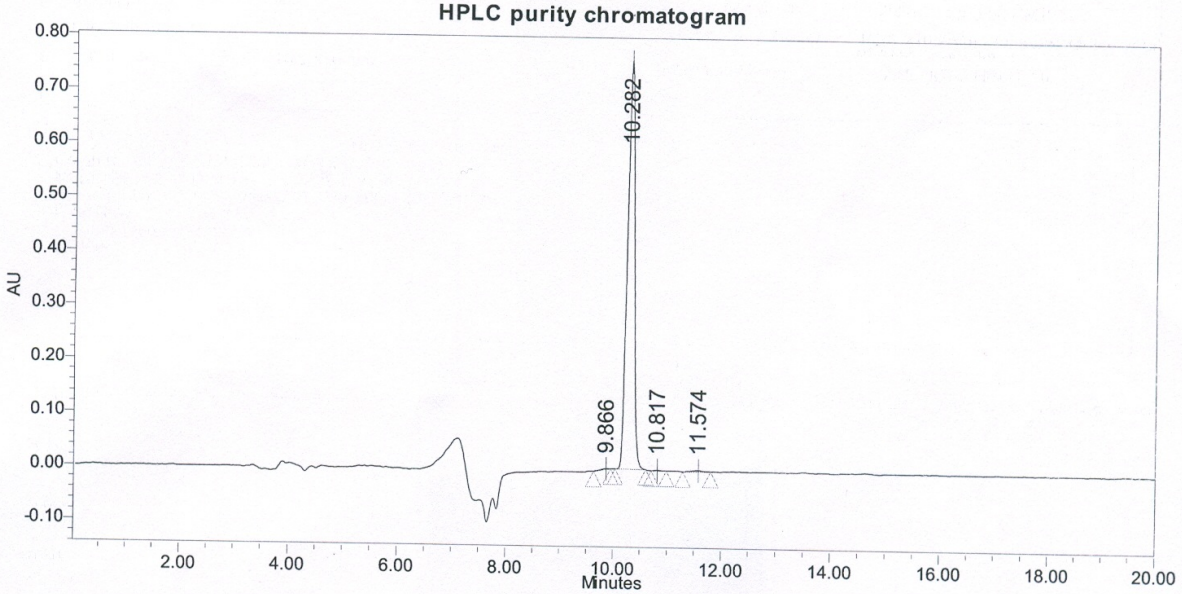
Sample Name :	KSY-B698-123	Acquired By :	Analyst
Vial No :	52	Date Acquired :	8/29/2012 2:55:50 PM IST
Injection Volume :	2.00 ul	Proc. Chnl. Descr :	PDA 210.0 nm Blank Subtracted from
Run Time :	20.0 Minutes	System Name :	ANL_MCL_HPLC_10

HPLC CONDITIONS

Column : INERTSIL C-8 (4.6 X 250mm) 5µm
Mobile Phase : D:MeOH; B:0.1% TFA (Aq)
Time(%T/D) : 0/10,2/10,5/70,8/90,18/90,19/10,20/10
Flow Rate : 1.0 ml/min
Column Temp : Ambient
Diluent : ACN

17

HPLC purity chromatogram



Peak Results

	RT	Area	% Area
1	9.866	14922	0.21
2	10.282	6950536	99.23
3	10.817	7743	0.11
4	11.574	31425	0.45

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GVK BIOSCIENCES (P) LTD

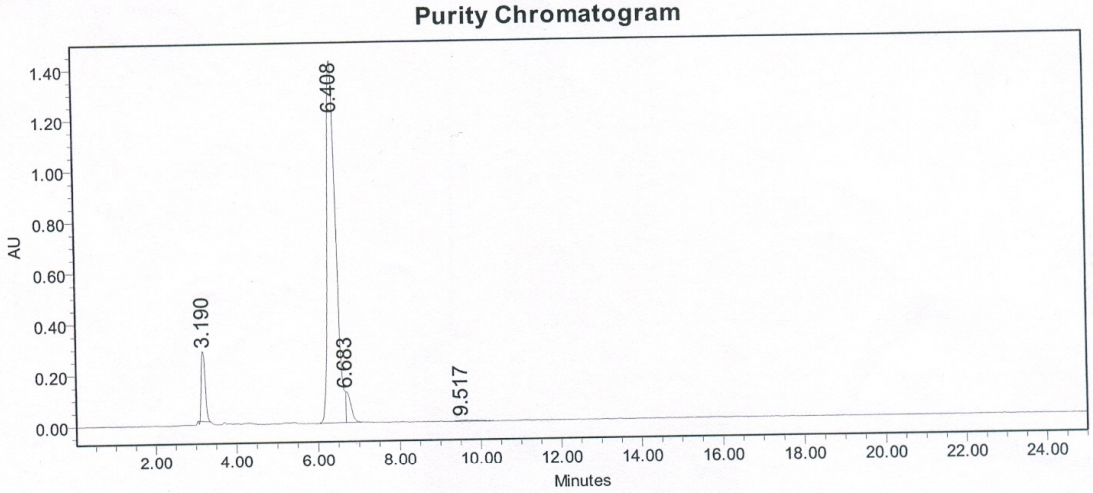
Sample Name:	KSY-B698-123	Acquired By:	nrm12_mcl
Vial:	29	Date Acquired:	3/4/2012 11:11:55 AM IST
Injection Volume:	3.00 ul	Proc. Chnl. Descr :	PDA 210.0 nm
Run Time:	25.0 Minutes	Sytem Name :	NRM2_PDA2

Chromatograhic Conditions

Column : SUNFIREC18 250x4.6 mm , 5 µm
Column ID : ANL_C158
Mobile Phase : A=ACN,B=0.1%TFA
T/%B : 0/80,2/80,6/20,18/20,19/80,20/80
Flow Rate : 1.0ml/min
Column Temp : NA
Diluent : MEOH

18

Purity Chromatogram



	RT	Area	% Area
1	3.19	1909965	8.00
2	6.41	20831617	87.22
3	6.68	1006617	4.21

	RT	Area	% Area
4	9.52	134527	0.56

Page: 1 of 2

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ANALYTICAL REPORT
GVK BIOSCIENCES PVT LTD

SAMPLE INFORMATION

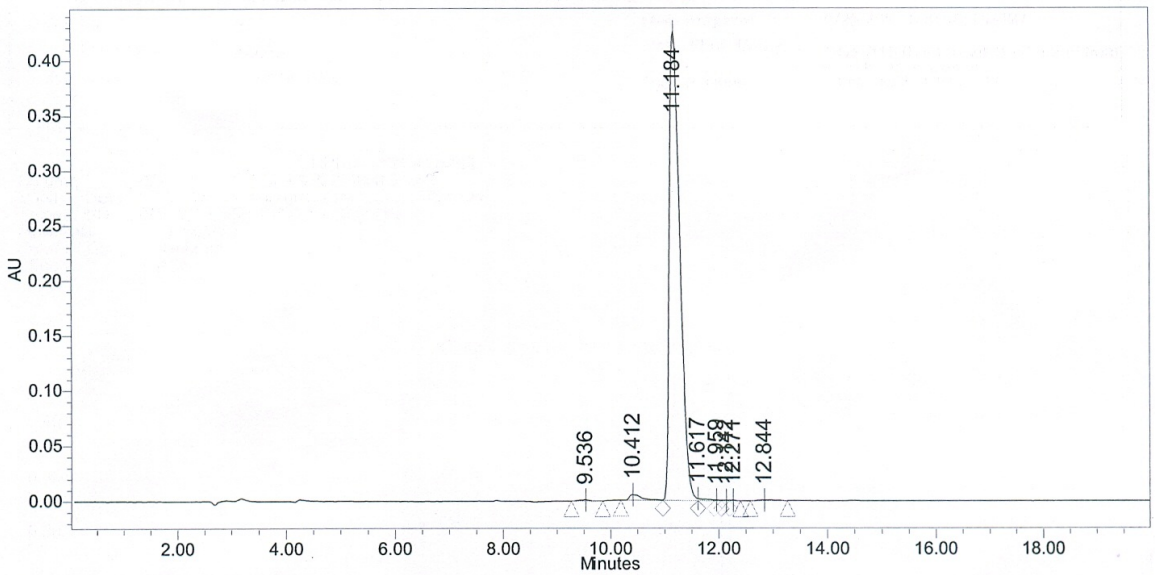
Sample Name :	KSY-B698-COMPOUND-19	Acquired By :	Analyst
Vial No :	4	Date Acquired :	8/29/2012 5:34:32 PM IST
Injection Volume :	2.00 ul	Proc. Chnl. Descr :	PDA 215.0 nm Blank Subtracted from
Run Time :	20.0 Minutes	System Name :	ANL_MCL_HPLC_02

HPLC CONDITIONS

Column :YMC TRAIT C18(4.6 X 250mm) 5µm
Mobile Phase :B:0.01M Ammonium acetate(Aq) : D:MeOH
Time of %D : 0/10,2/10,6/90,18/90,19/10,20/10
Flow Rate : 1.0 ml/min
Column Temp : Ambient
Diluent : ACN

19

HPLC purity chromatogram



Peak Results

	RT	Area	% Area
1	9.536	11643	0.20
2	10.412	87215	1.47
3	11.184	5776797	97.54
4	11.617	23501	0.40
5	11.959	4460	0.08
6	12.142	3562	0.06
7	12.271	4178	0.07
8	12.844	11405	0.19

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GVK BIOSCIENCES (P) LTD

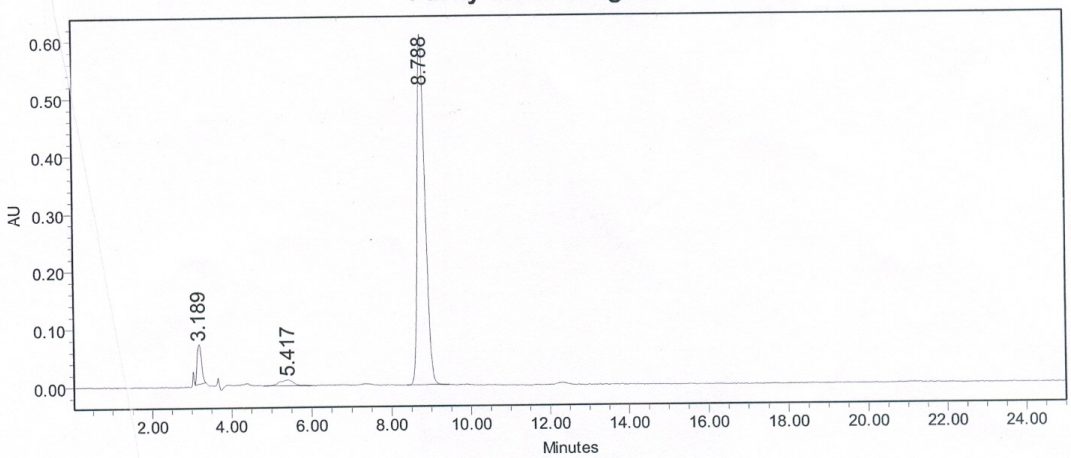
Sample Name:		Acquired By:	nrm12_mcl
Vial:	27	Date Acquired:	3/4/2012 10:20:20 AM IST
Injection Volume:	4.00 ul	Proc. Chnl. Descr :	PDA 210.0 nm
Run Time:	25.0 Minutes	Sytem Name :	NRM2_PDA2

Chromatograhic Conditions

Column : SUNFIREC18 250x4.6 mm , 5 µm
Column ID : ANL_C158
Mobile Phase : A=ACN,B=0.1%TFA
T/%B : 0/80,2/80,6/20,18/20,19/80,20/80
Flow Rate : 1.0ml/min
Column Temp : NA
Diluent : MEOH

20

Purity Chromatogram



	RT	Area	% Area
1	3.19	536037	5.95
2	5.42	244843	2.72
3	8.79	8233371	91.34

Empower2 software

ANALYTICAL REPORT
GVK BIOSCIENCES PVT LTD

SAMPLE INFORMATION

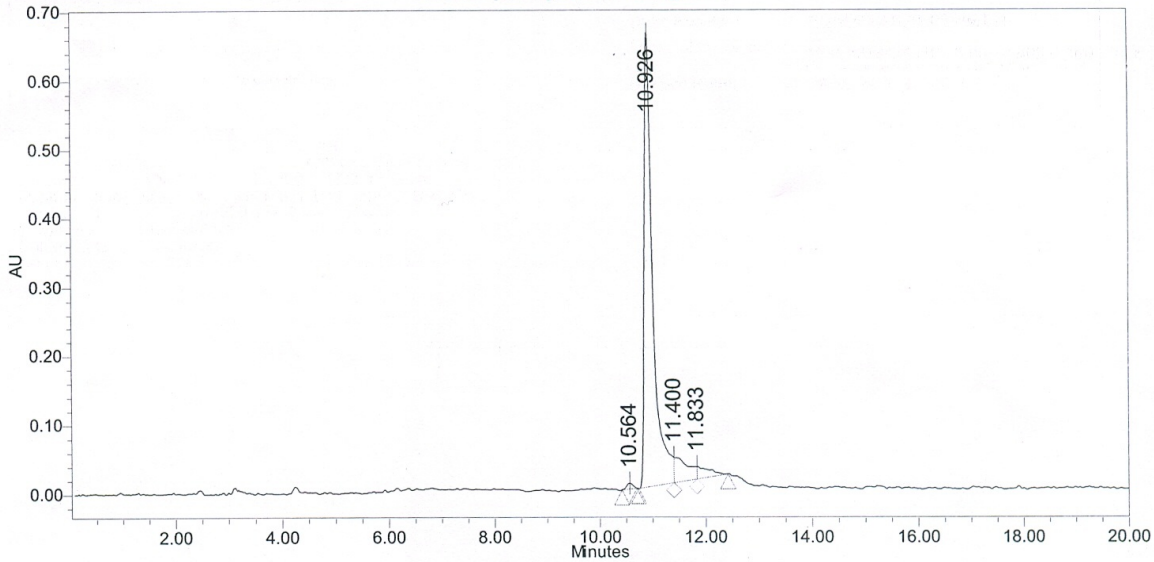
Sample Name :	KSY-B698-122	Acquired By :	Analyst
Vial No :	6	Date Acquired :	8/29/2012 6:24:03 PM IST
Injection Volume :	2.00 ul	Proc. Chnl. Descr :	PDA MaxPlot (190.0 nm to 800.0 nm) Blank
Run Time :	20.0 Minutes	System Name :	ANL_MCL_HPLC_02

HPLC CONDITIONS

Column :YMC TRAIT C18(4.6 X 250mm) 5µm
Mobile Phase :B:0.01M Ammonium acetate(Aq) : D:MeOH
Time of %D : 0/10,2/10,6/90,18/90,19/10,20/10
Flow Rate : 1.0 ml/min
Column Temp : Ambient
Diluent : ACN

21

HPLC purity chromatogram



Peak Results

	RT	Area	% Area
1	10.564	63992	0.77
2	10.926	7273320	87.88
3	11.400	661267	7.99
4	11.833	277934	3.36

Empower 2 software

GVK BIOSCIENCES (P) LTD

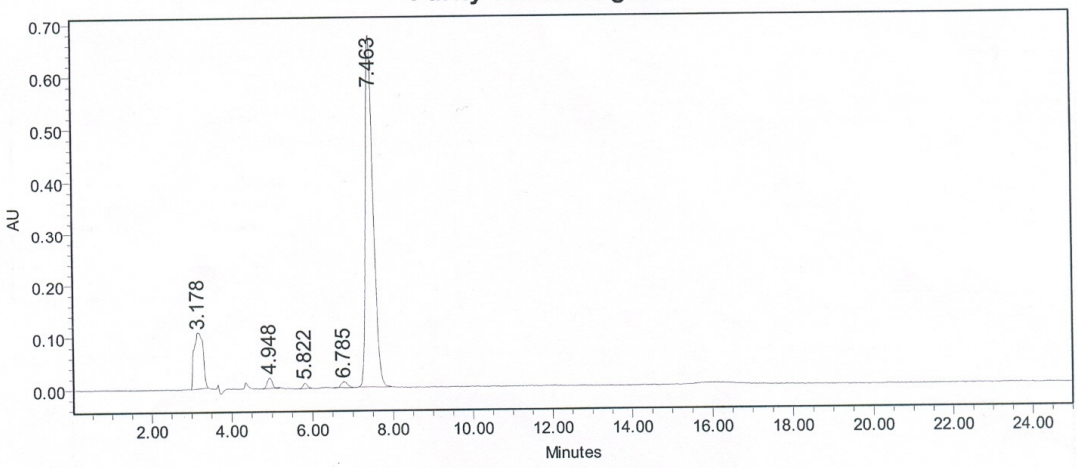
Sample Name:	KSY-B698-122	Acquired By:	nrm12_mcl
Vial:	28	Date Acquired:	3/4/2012 10:46:08 AM IST
Injection Volume:	4.00 ul	Proc. Chnl. Descr :	PDA 210.0 nm
Run Time:	25.0 Minutes	Sytem Name :	NRM2_PDA2

Chromatograhic Conditions

Column : SUNFIREC18 250x4.6 mm , 5 µm
Column ID : ANL_C158
Mobile Phase : A=ACN,B=0.1%TFA
T/%B : 0/80,2/80,6/20,18/20,19/80,20/80
Flow Rate : 1.0ml/min
Column Temp : NA
Diluent : MEOH

23

Purity Chromatogram



	RT	Area	% Area
1	3.18	1685443	15.41
2	4.95	172104	1.57
3	5.82	74040	0.68

	RT	Area	% Area
4	6.79	121570	1.11
5	7.46	8881680	81.22

Empower2 software

CHIRAL_IA_RPT

ANALYTICAL REPORT-MCL-III
GVK BIOSCIENCES PVT LTD

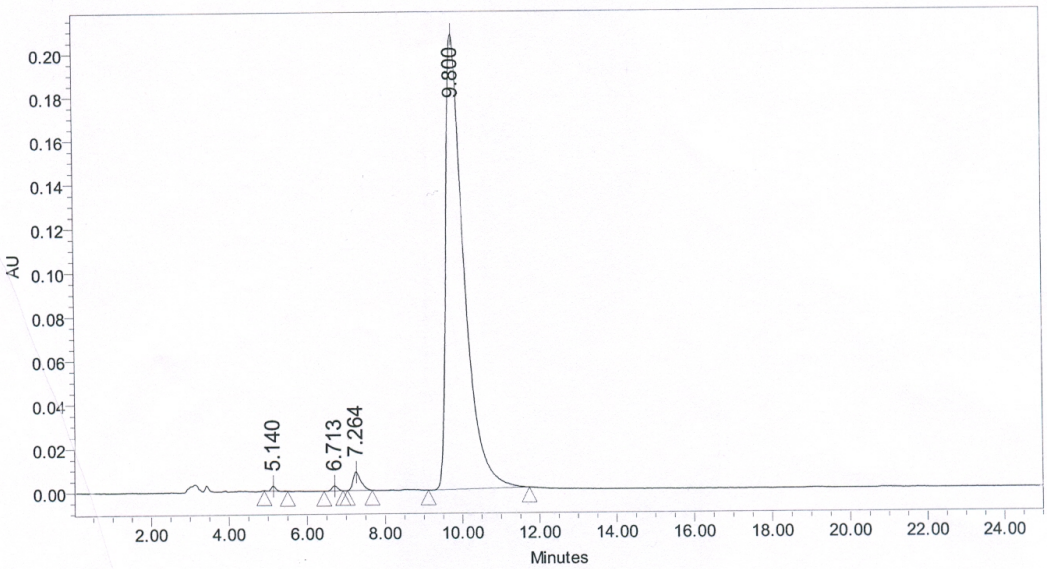
SAMPLE INFORMATION

Sample Name	KSY-B698-54-1	Acquired By	System
Vial No	16	Date Acquired	10/14/2011 11:29:08 PM IST
Injection Volume	6.00 ul	Proc. Chnl. Descr	PDA 215.0 nm
Run Time	25.0 Minutes		ANL_MCL_HPLC_02

HPLC CONDITIONS

Column	CHIRAL PAK IA 4.6 X 250mm , 5µm
Colunm ID	ANL_CHIRAL IA_110
Mobile Phase	A= n-Hexane : B=IPA
ISOCRATIC	70:30
Flow Rate	1.0 ml/min
Column Temp	-NA-
Diluent	Ethanol

27



Peak Results

	RT	Area	% Area
1	5.140	16918	0.25
2	6.713	22225	0.32
3	7.264	115479	1.68
4	9.800	6730055	97.75

Empower2 software

CHIRAL IA RPT

ANALYTICAL REPORT-MCL-III
GVK BIOSCIENCES PVT LTD

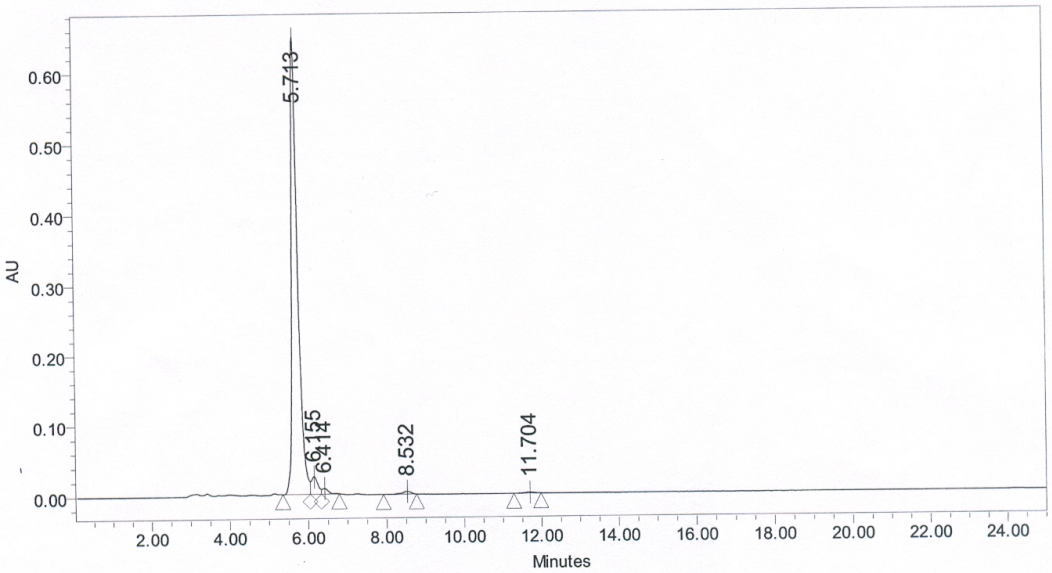
SAMPLE INFORMATION

Sample Name	: KSY-B698-52-1	Acquired By	: System
Vial No	: 17	Date Acquired	: 10/14/2011 11:54:55 PM IST
Injection Volume	: 6.00 ul	Proc. Chnl. Descr	: PDA 215.0 nm ANL_MCL_HPLC_02
Run Time	: 25.0 Minutes		

HPLC CONDITIONS

Column : CHIRAL PAK IA 4.6 X 250mm , 5µm
Colunm ID : ANL_CHIRAL IA_110
Mobile Phase : A= n-Hexane : B=IPA
ISOCRATIC : 70:30
Flow Rate : 1.0 ml/min
Column Temp : -NA-
Diluent : Ethanol

31



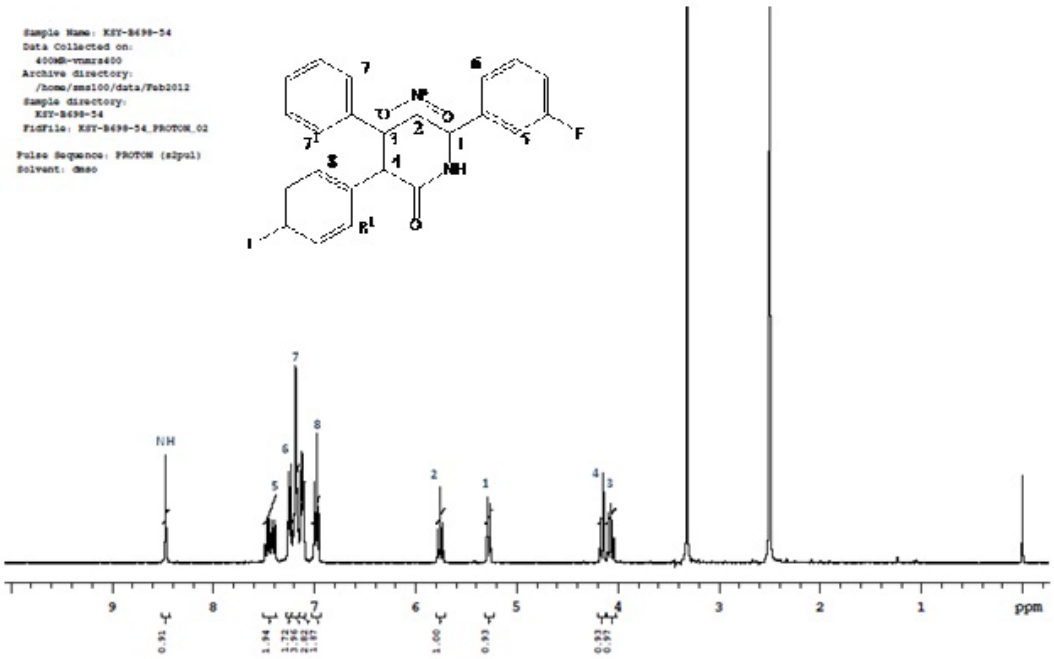
Peak Results

	RT	Area	% Area
1	5.713	7103075	93.97
2	6.155	284824	3.77
3	6.414	84840	1.12
4	8.532	63412	0.84
5	11.704	22693	0.30

Structure elucidation of **27**:

Experiments conducted for structural conformation: ^{1}H NMR and NOESY 1D

1) ^{1}H-NMR



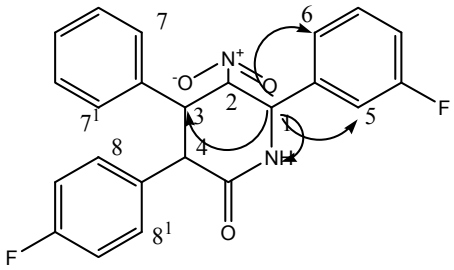
Observations:

S.No	^{1}H(label)	Chemical shift (ppm)	Coupling constant (Hz)	Integration	Multiplicity
1	1	5.28	10-11	1	Doublet
2	2	5.75	10-11	1	Triplet
3	3	4.09	12-13	1	Triplet
4	4	4.17	12-13	1	Doublet
5	5	7.41	10-11	1	Doublet
6	6	7.25	8-9	1	Doublet
7	7&7^{1}	7.21	-	2	Bunch of peaks
8	8&8^{1}	7.13	-	2	Bunch of peaks

9	NH	8.47	-	1	Singlet(broad)

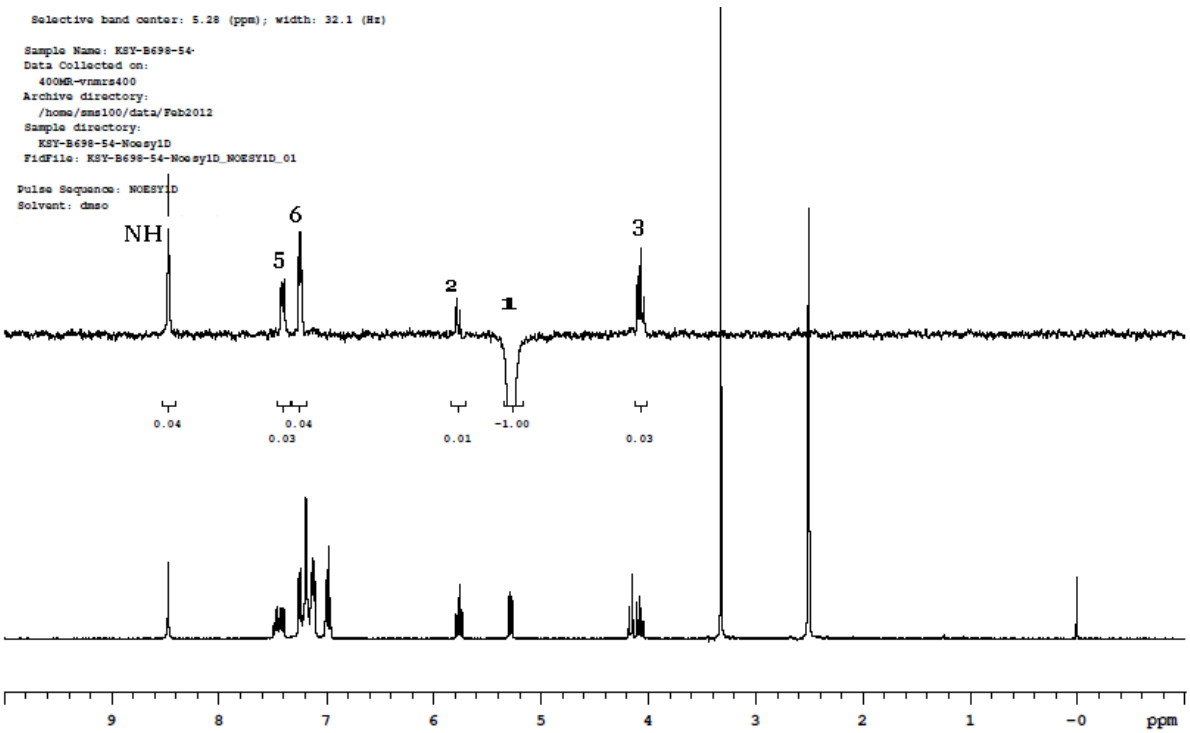
1) Noesy 1D

Observations:



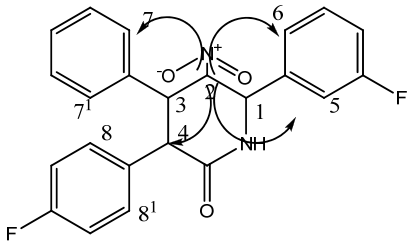
a) While irradiating the proton **"1"**, Protons **3**, **5**, **6 and NH** are getting more enhanced. It is supports that ^{1}H-**1** and ^{1}H-**3** are having more spatial contributions

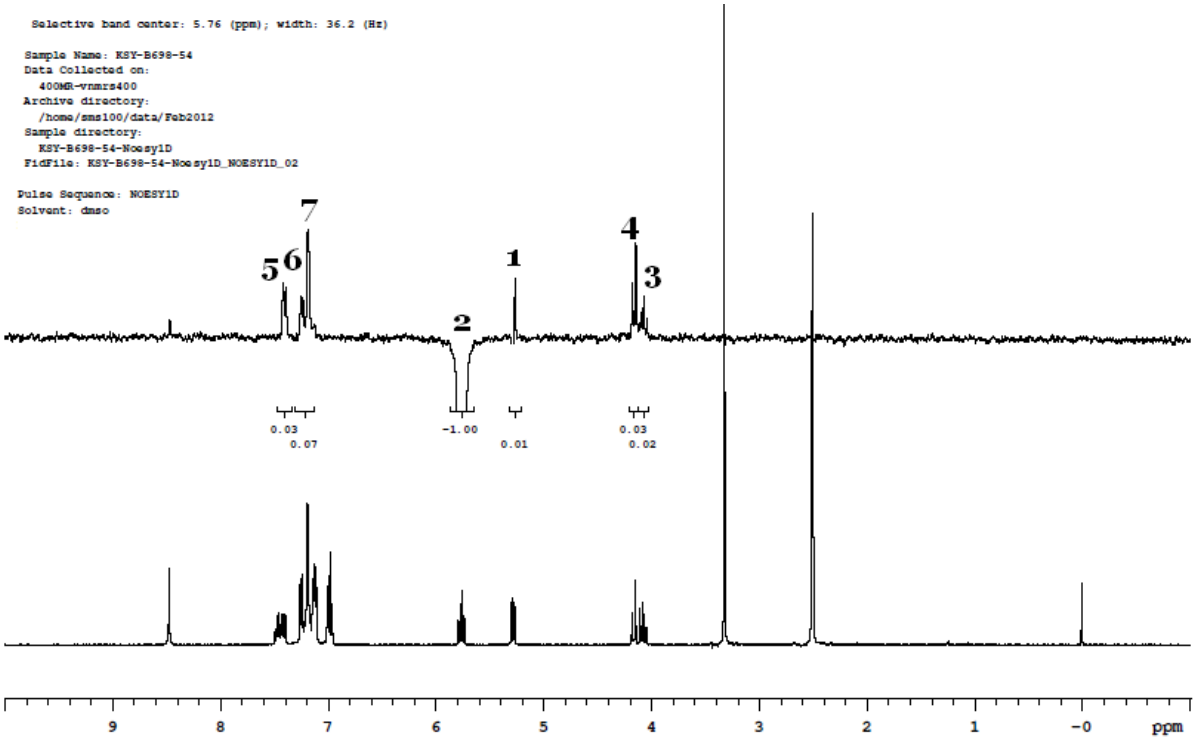
From this we can say that ^{1}H-**1** and ^{1}H-**3** are in the same plane



b) While irradiating the proton **"2"**, protons **4**, **5**, **6** and **7** are getting more enhanced. It supports that ^{1}H-**2** and **4** have more spatial contributions

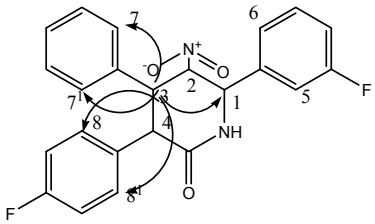
Hence we can conclude ^{1}H-**2** is in plane with ^{1}H-**4**

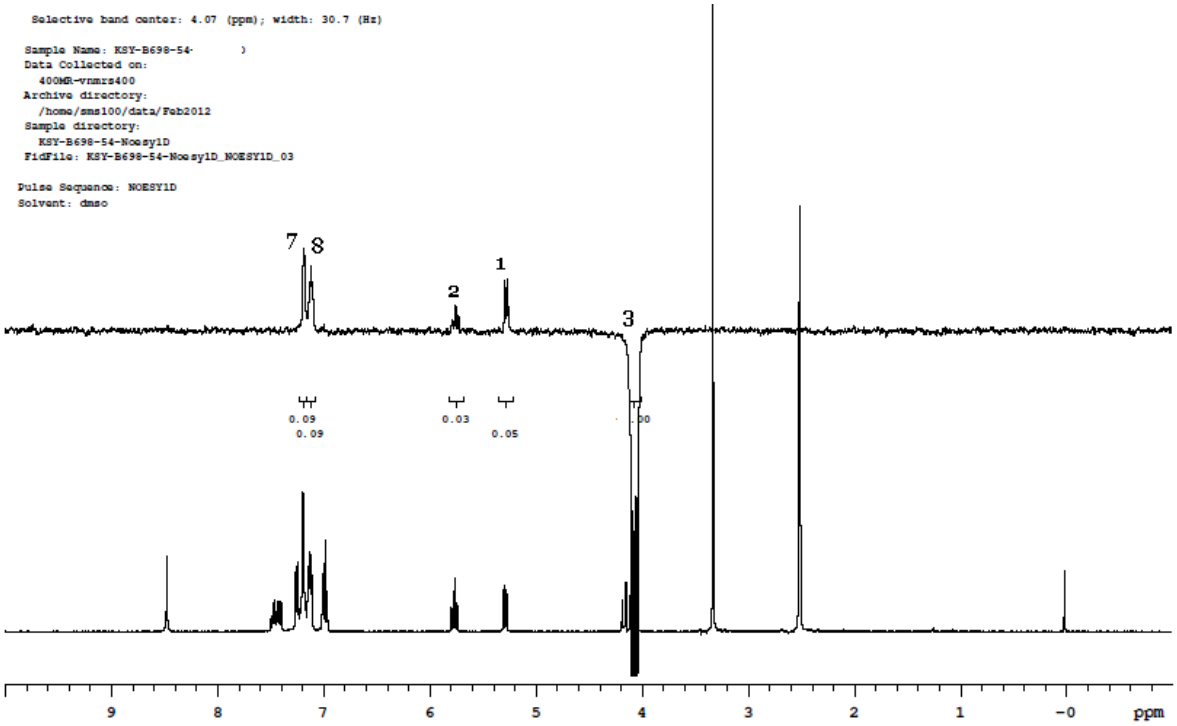




c) While irradiating the proton **"3"** , Protons **1, 4, 7 and 8** are getting more enhancement

This supports that ^{1}H-**3** and ^{1}H-**1** are having more spatial contributions. Hence we conclude that ^{1}H-**3** is is in plane with ^{1}H-**1 and H-2**.





Conclusion:

Hence the final structure is: