

**A Sequential Synthetic Strategy towards Unexplored Dibenzo[*b,f*][1,4]thiazepine
Carboxamides: Copper Catalysed C-S Cyclisation followed by Ugi type 3CC Cascade**

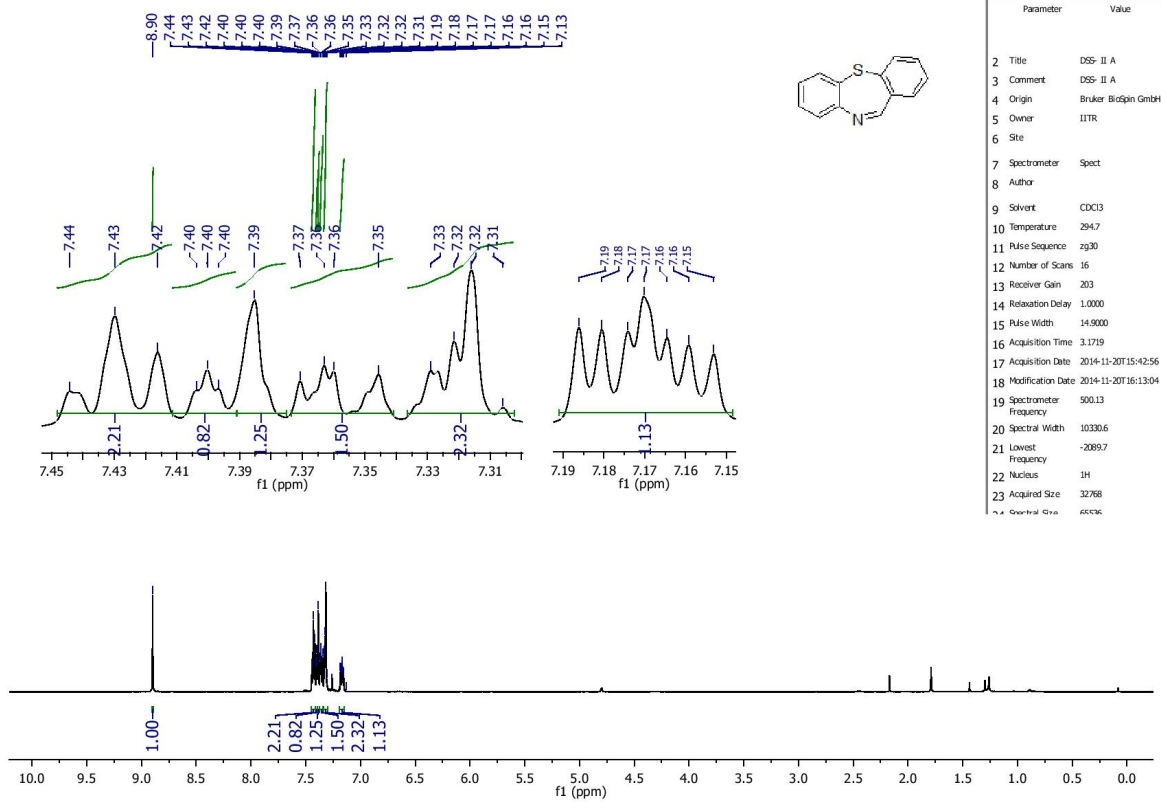
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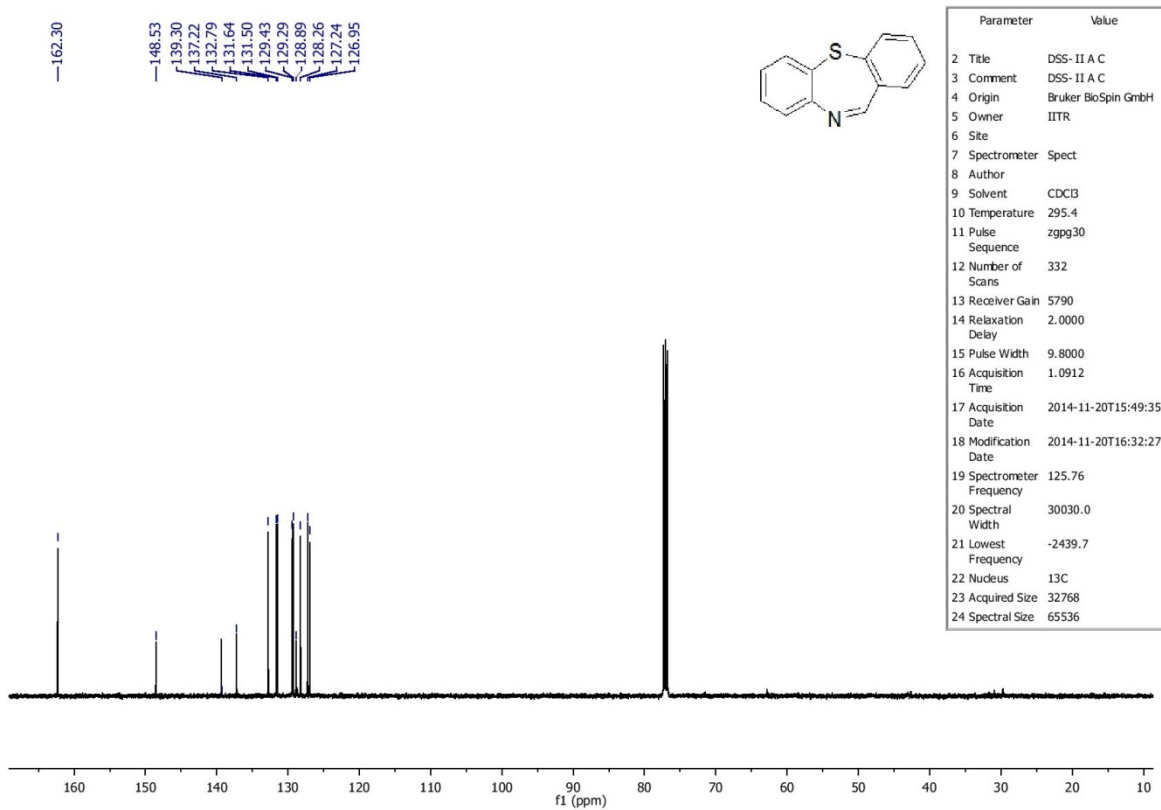
*e-mail: anujsfcy@iitr.ernet.in

1. Copies of ¹H and ¹³C NMR spectrum of **dibenzo[*b,f*][1,4]thiazepines(3a-k)** 2-19.
2. Copies of ¹H and ¹³C NMR spectrum of
10, 11-dihydrodibenzo[*b,f*][1,4]thiazepine-11-carboxamides (6a-m) 19-39.

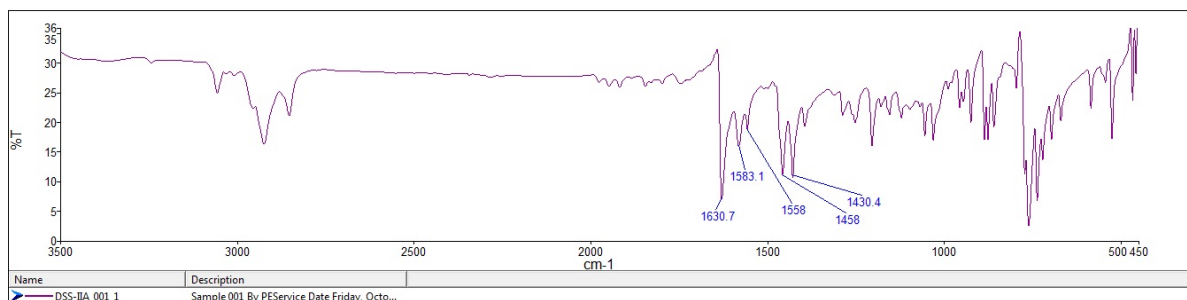
1) Dibenzo[b,f][1,4]thiazepine 3a



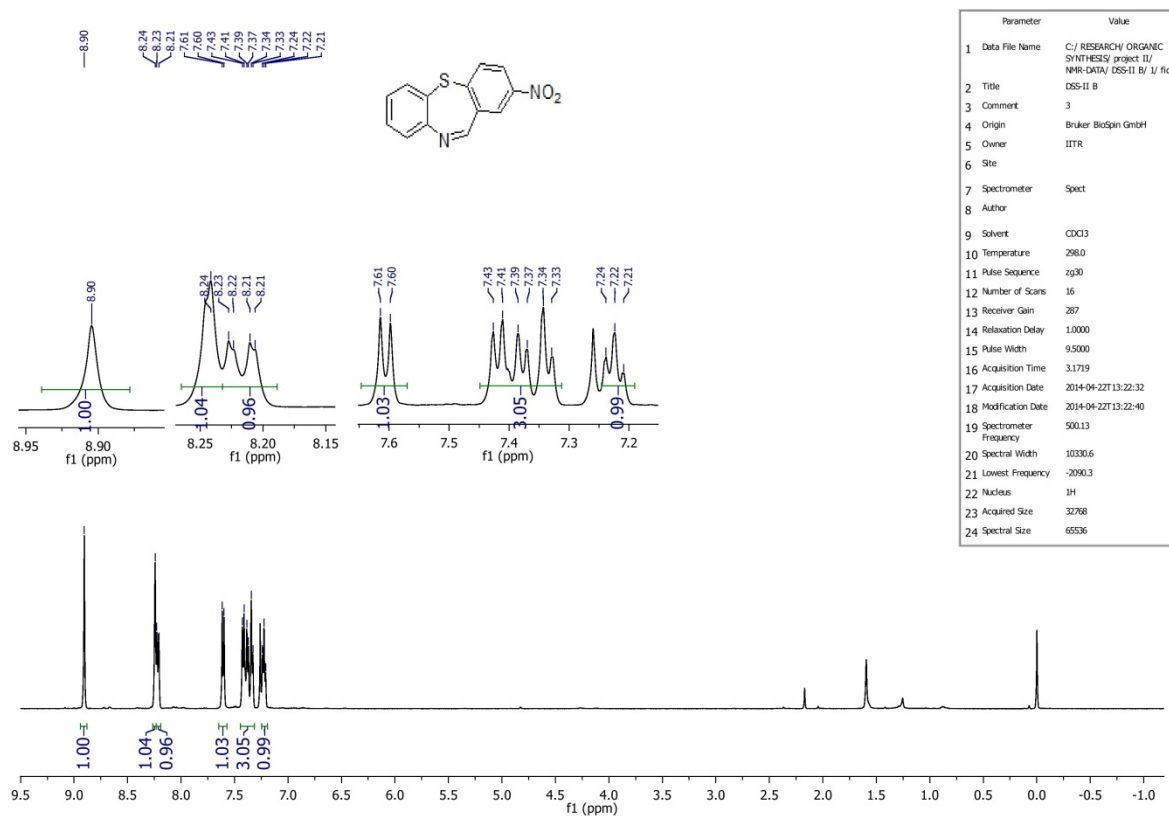
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5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
10 Temperature	294.7
11 Pulse Sequence	zg30
12 Number of Scans	16
13 Receiver Gain	203
14 Relaxation Delay	1.0000
15 Pulse Width	14.9000
16 Acquisition Time	3.1719
17 Acquisition Date	2014-11-20T15:42:56
18 Modification Date	2014-11-20T16:13:04
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20 Spectral Width	10300.6
21 Lowest Frequency	-2089.7
22 Nucleus	1H
23 Acquired Size	32768
24 Spectral Size	65536

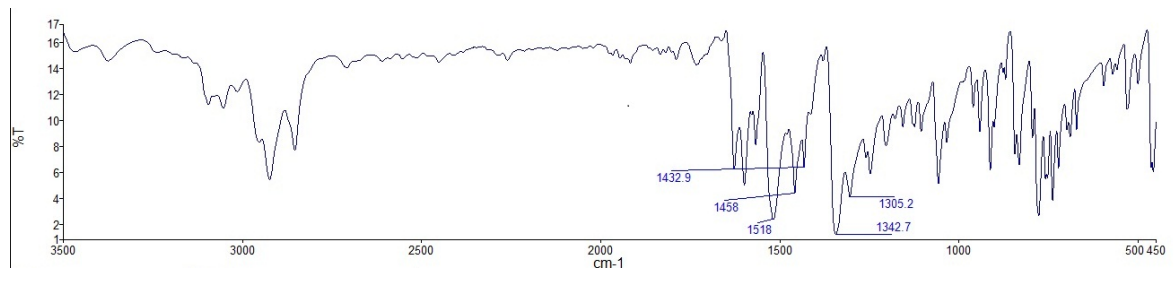
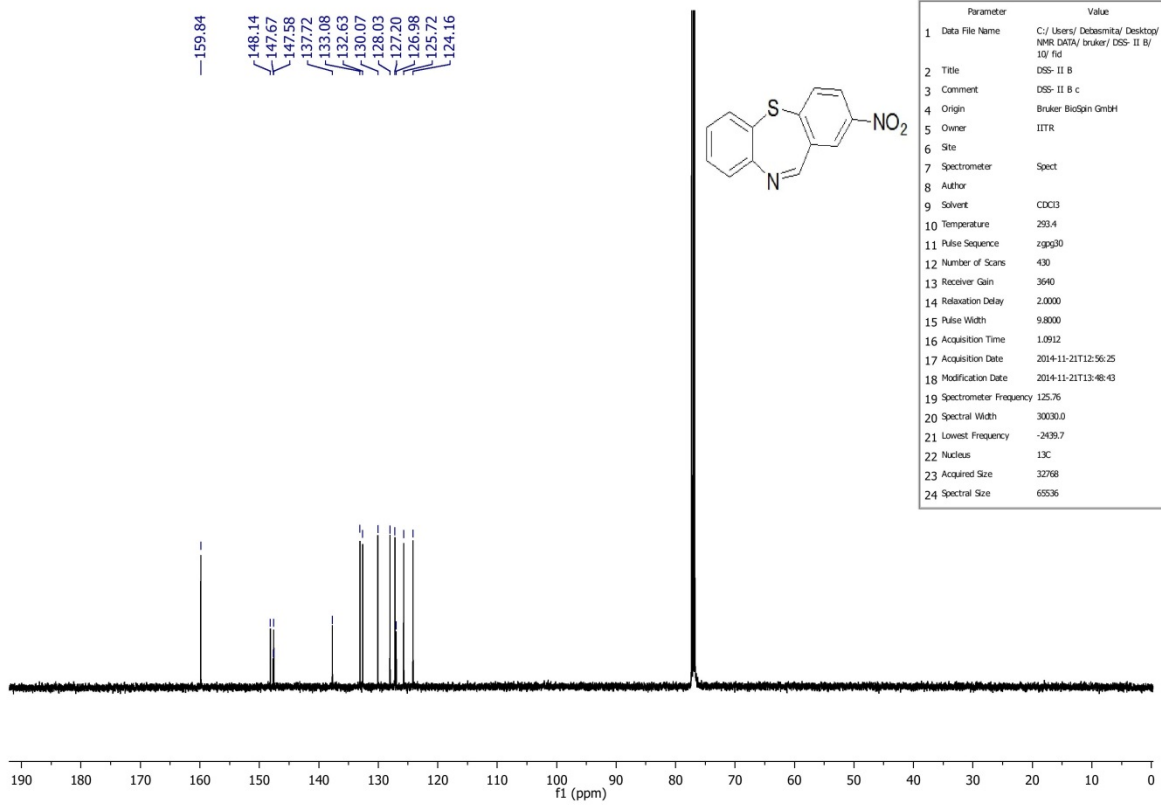


Parameter	Value
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5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
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11 Pulse Sequence	zgpg30
12 Number of Scans	
13 Receiver Gain	5790
14 Relaxation Delay	2.0000
15 Pulse Width	9.8000
16 Acquisition Time	1.0912
17 Acquisition Date	2014-11-20T15:49:35
18 Modification Date	2014-11-20T16:32:27
19 Spectrometer Frequency	125.76
20 Spectral Width	30030.0
21 Lowest Frequency	-2439.7
22 Nucleus	13C
23 Acquired Size	32768
24 Spectral Size	65536

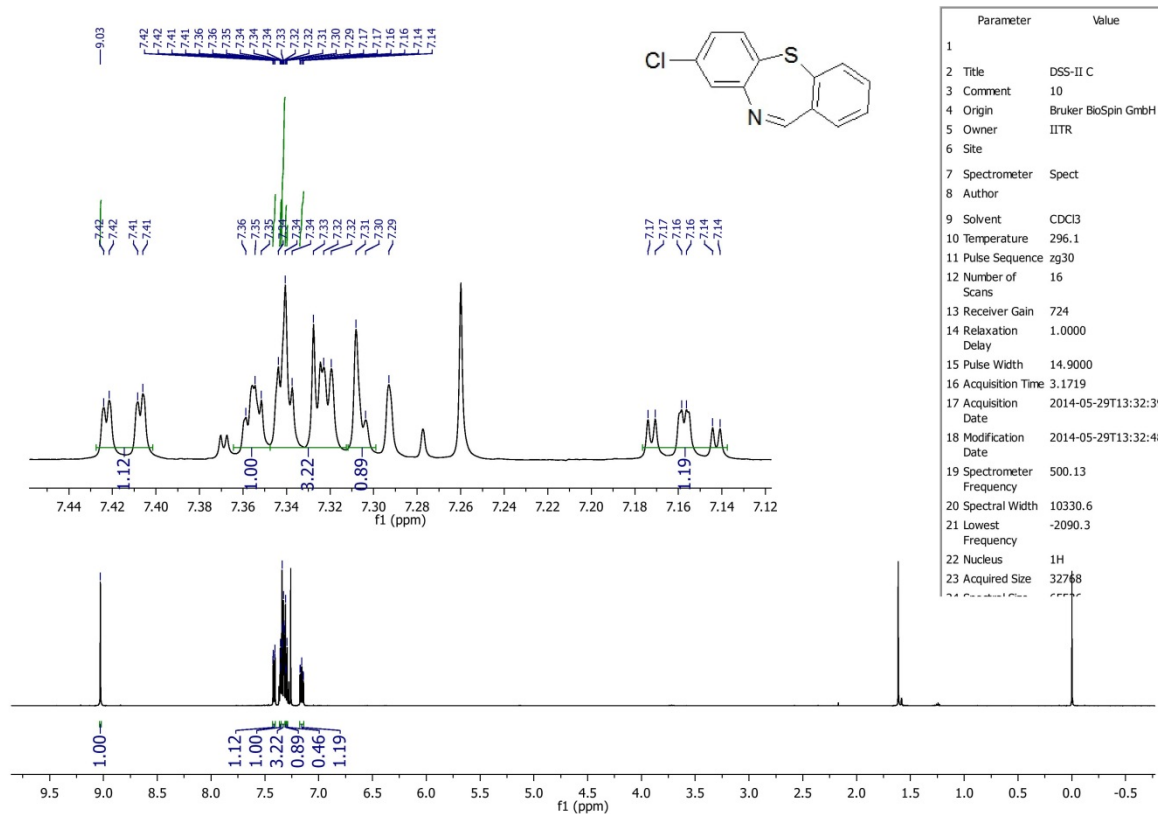


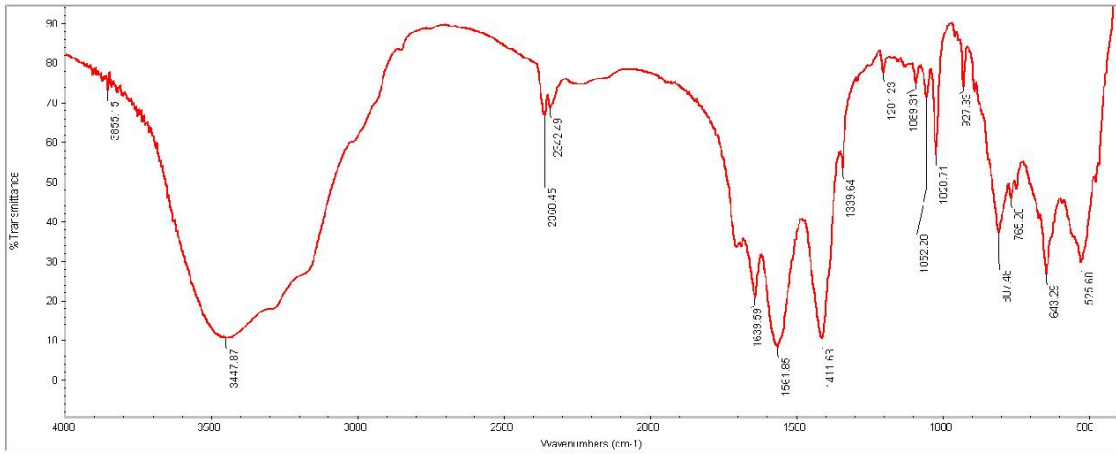
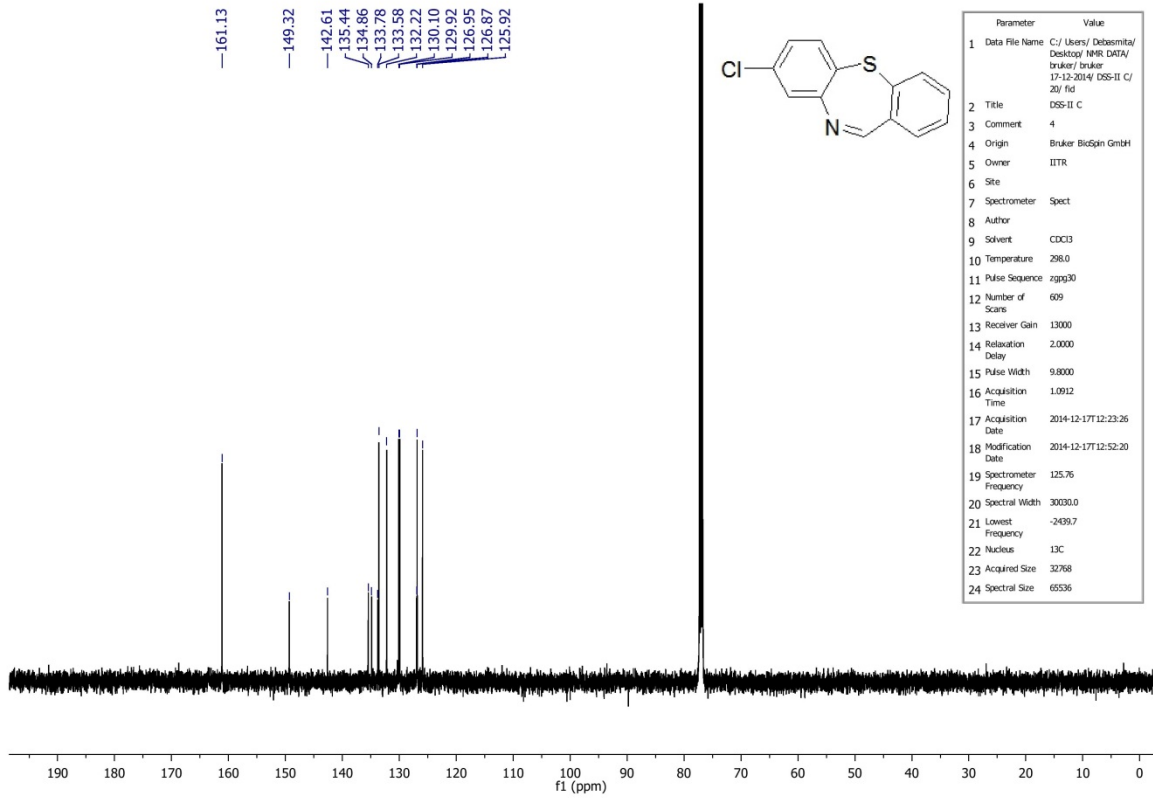
2) 2-nitrodibenzo[*b,f*][1,4]thiazepine 3b



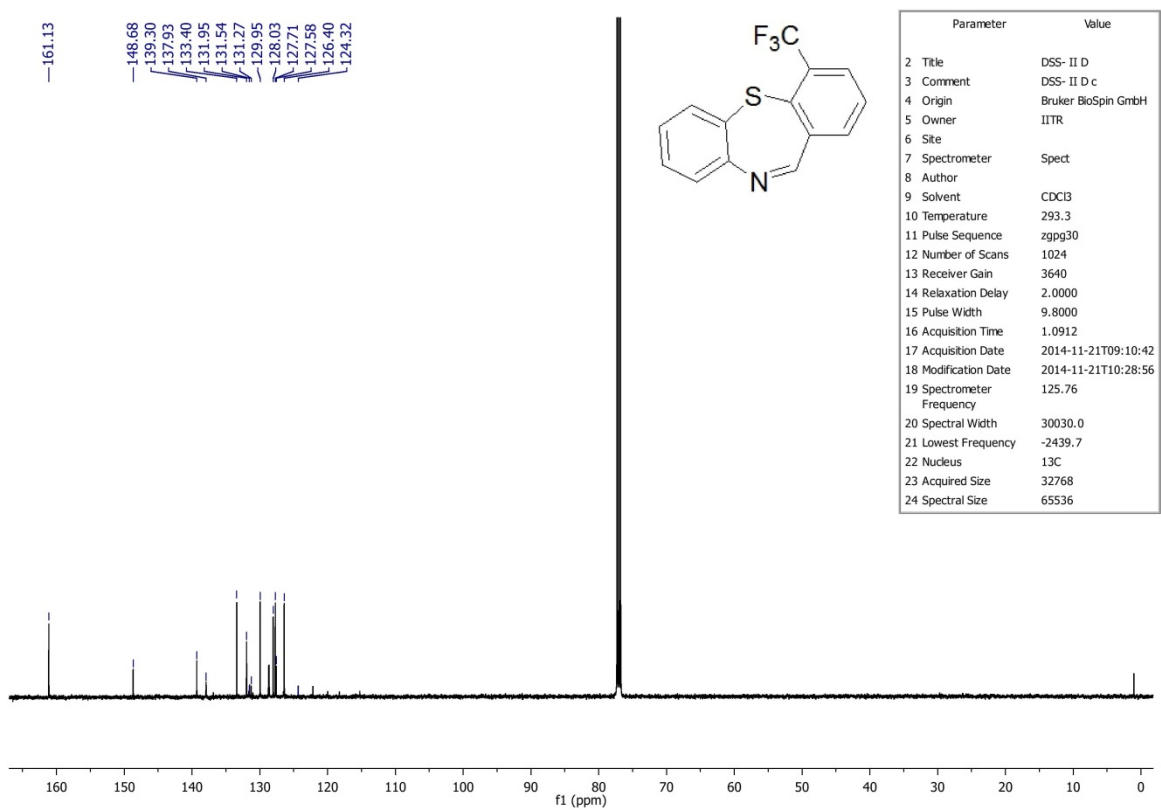
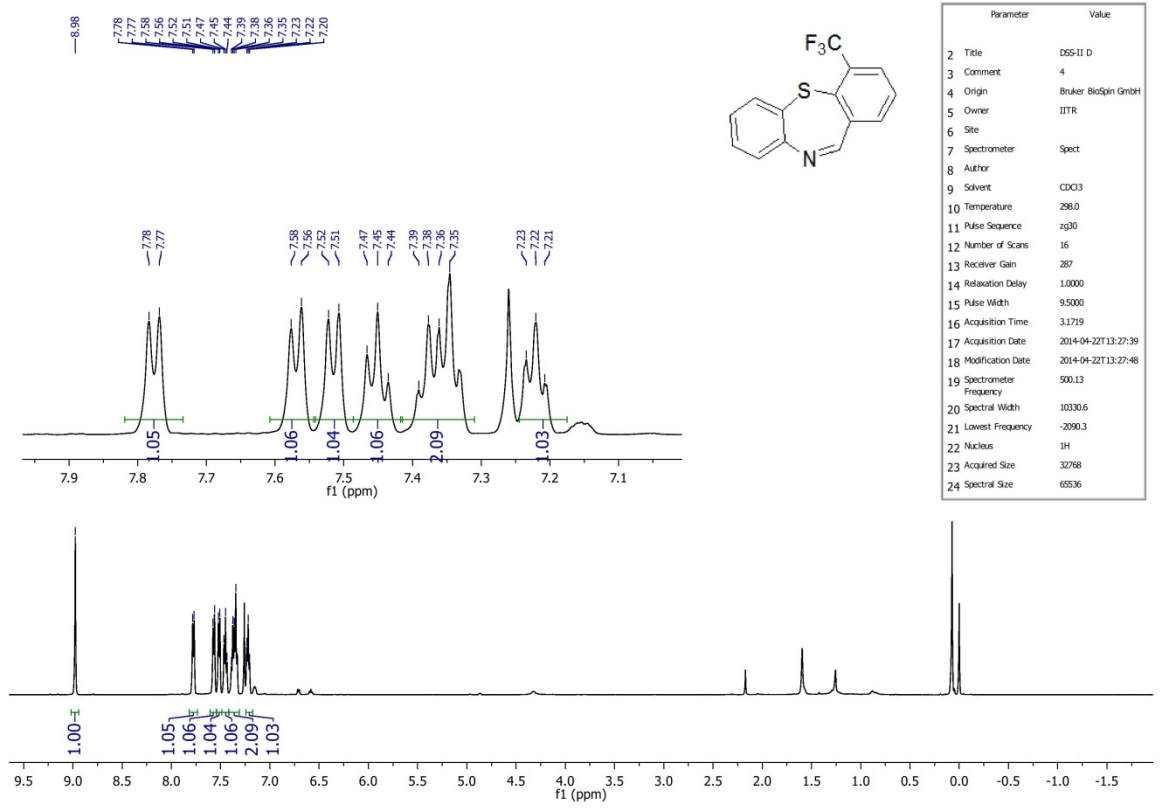


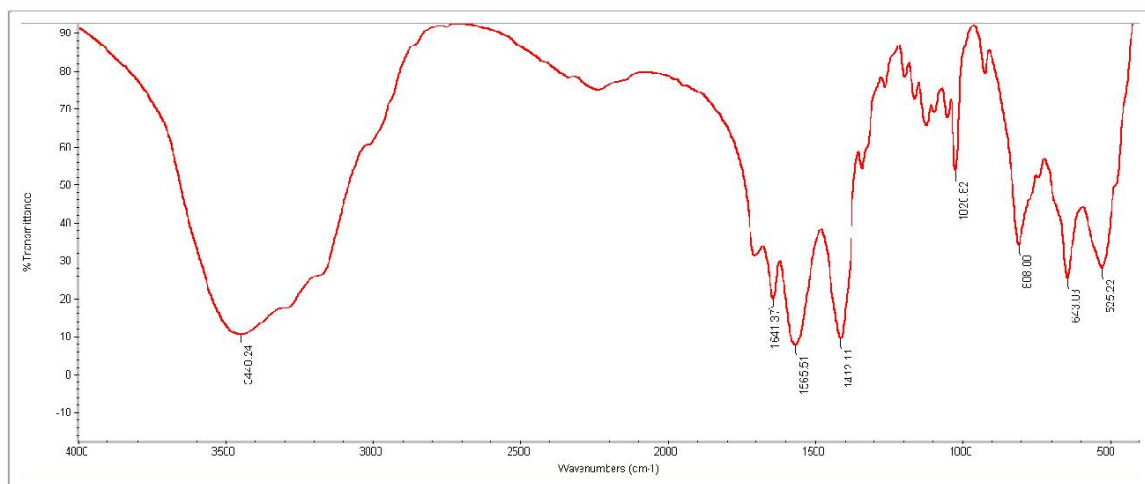
3) 8-chlorodibenzo[b,f][1,4]thiazepine 3c



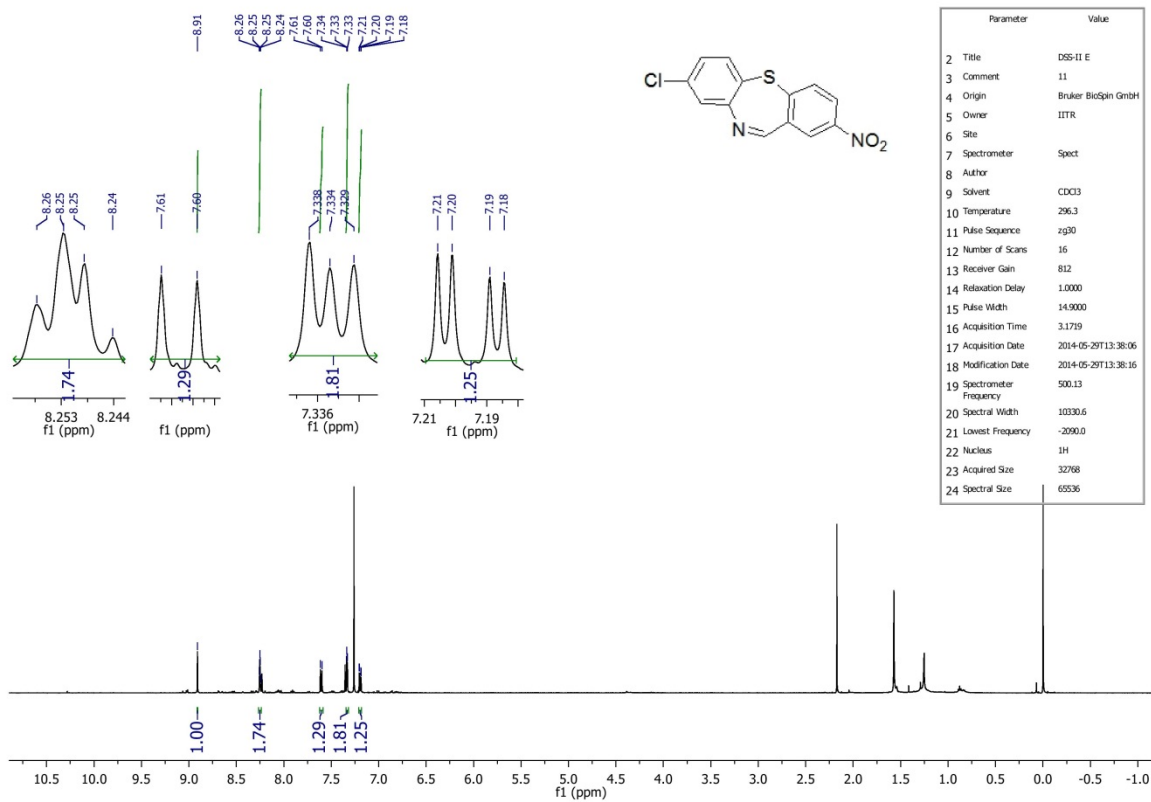


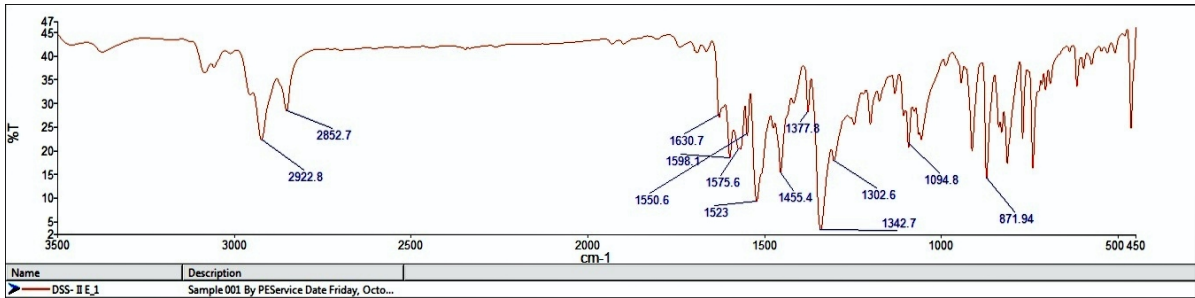
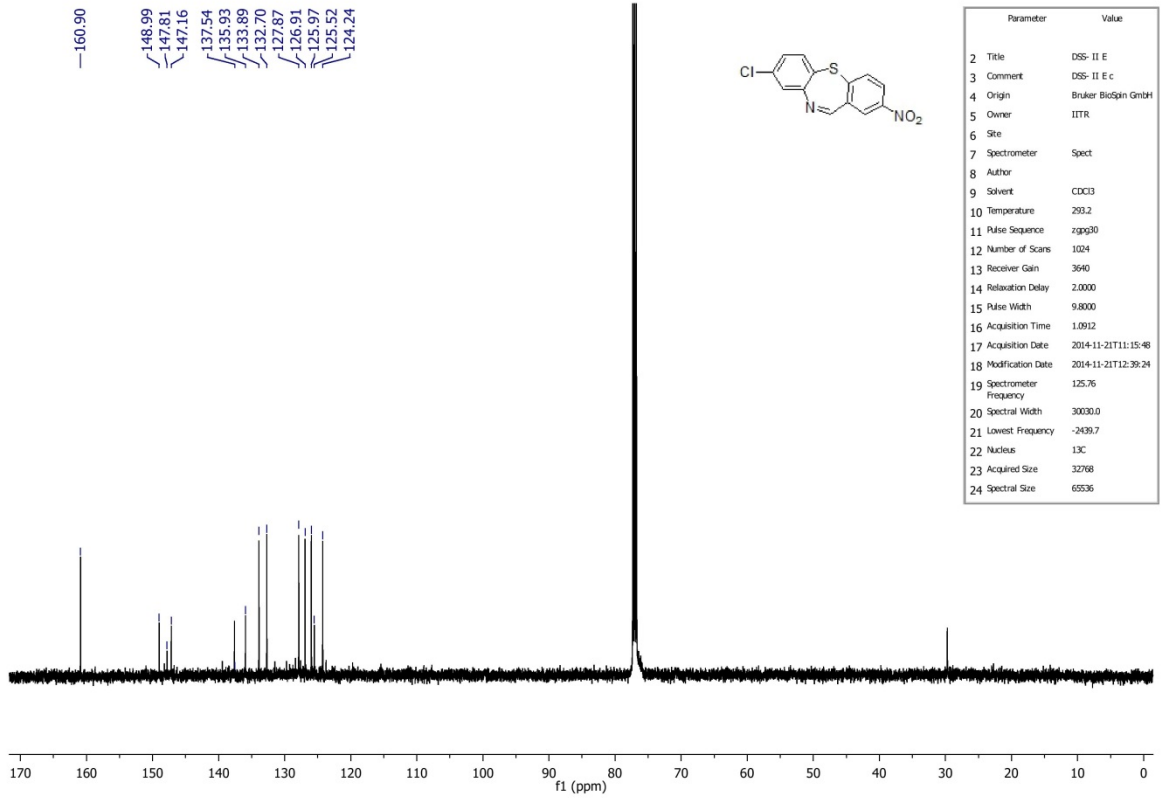
4) 4-(trifluoromethyl)dibenzo[b,f][1,4]thiazepine 3d



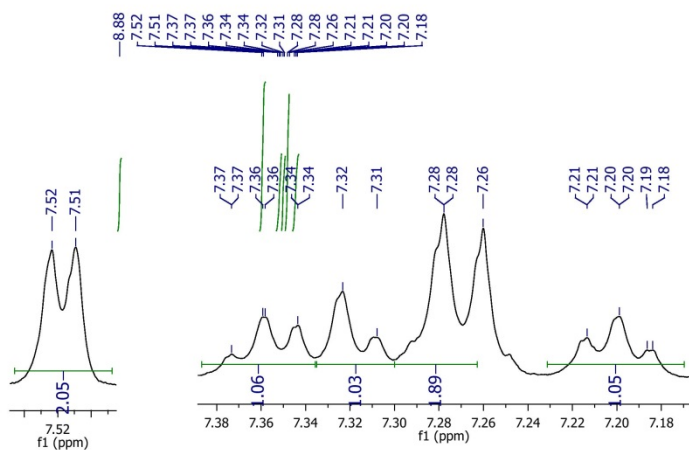


5) 8-chloro-2-nitrodibenzo[*b,f*][1,4]thiazepine 3e

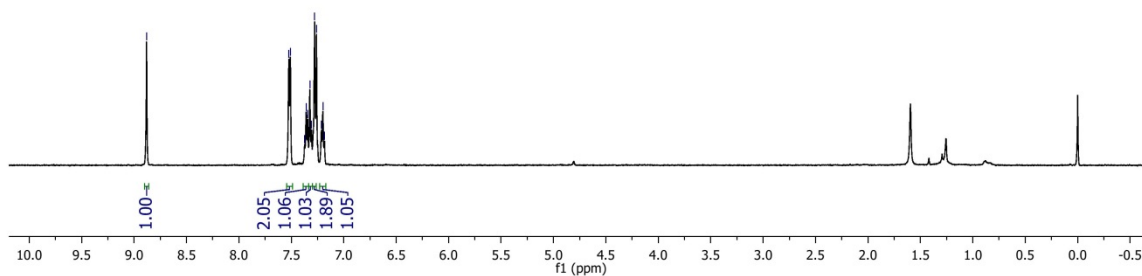




6)4-Chlorodibenzo[b,f][1,4]thiazepine 3f

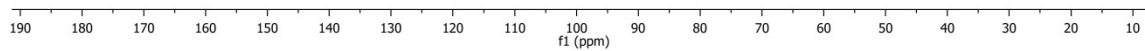


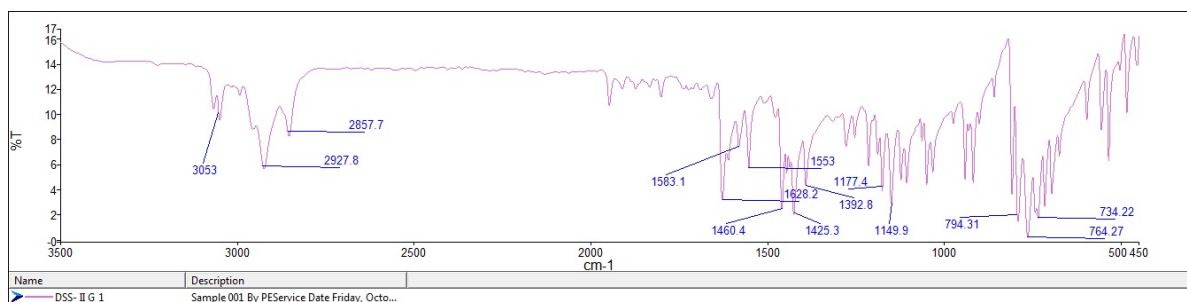
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4 Origin	Bruker BioSpin GmbH
5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
10 Temperature	298.0
11 Pulse Sequence	zg30
12 Number of Scans	16
13 Receiver Gain	2890
14 Relaxation Delay	1.0000
15 Pulse Width	9.5000
16 Acquisition Time	3.1719
17 Acquisition Date	2014-04-23T13:25:19
18 Modification Date	2014-04-23T13:25:28
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20 Frequency	
21 Spectral Width	10330.6
22 Lowest Frequency	-2000.3
23 Nucleus	1H
24 Acquired Size	32768



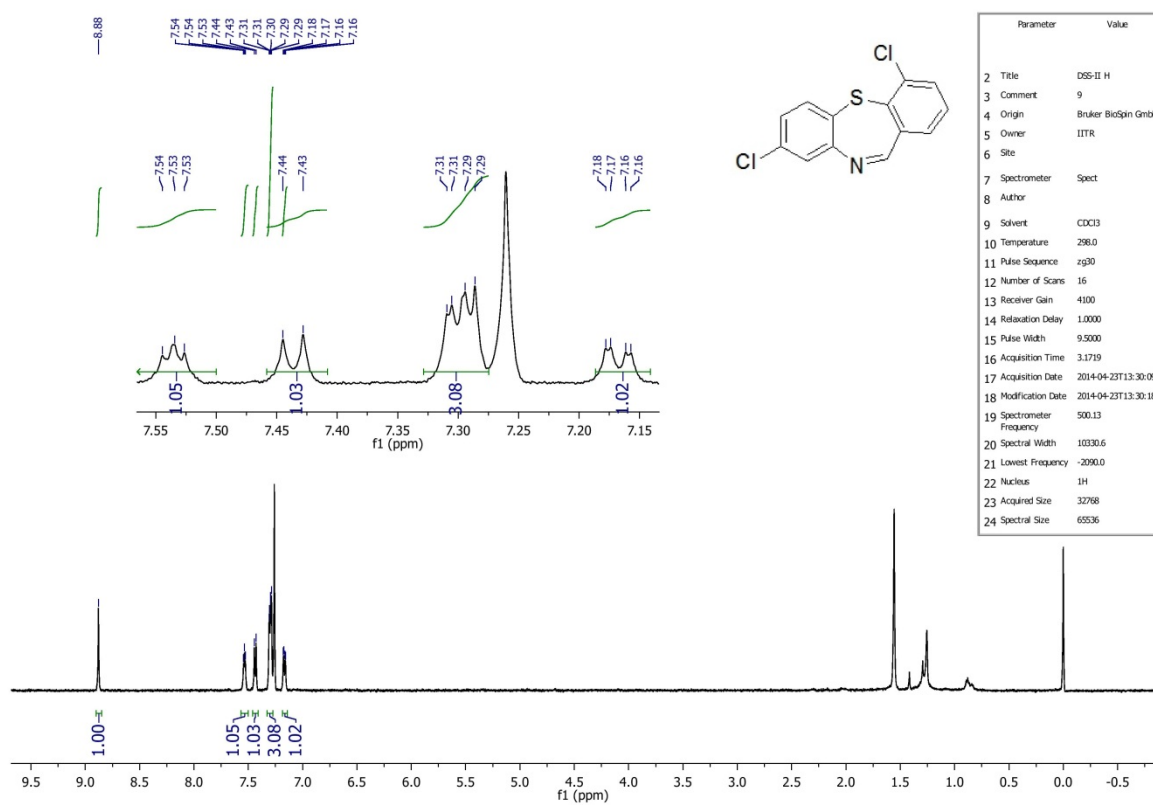
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148.57
138.73
137.80
135.82
133.66
131.74
129.77
128.87
127.57
127.54
127.47
126.64

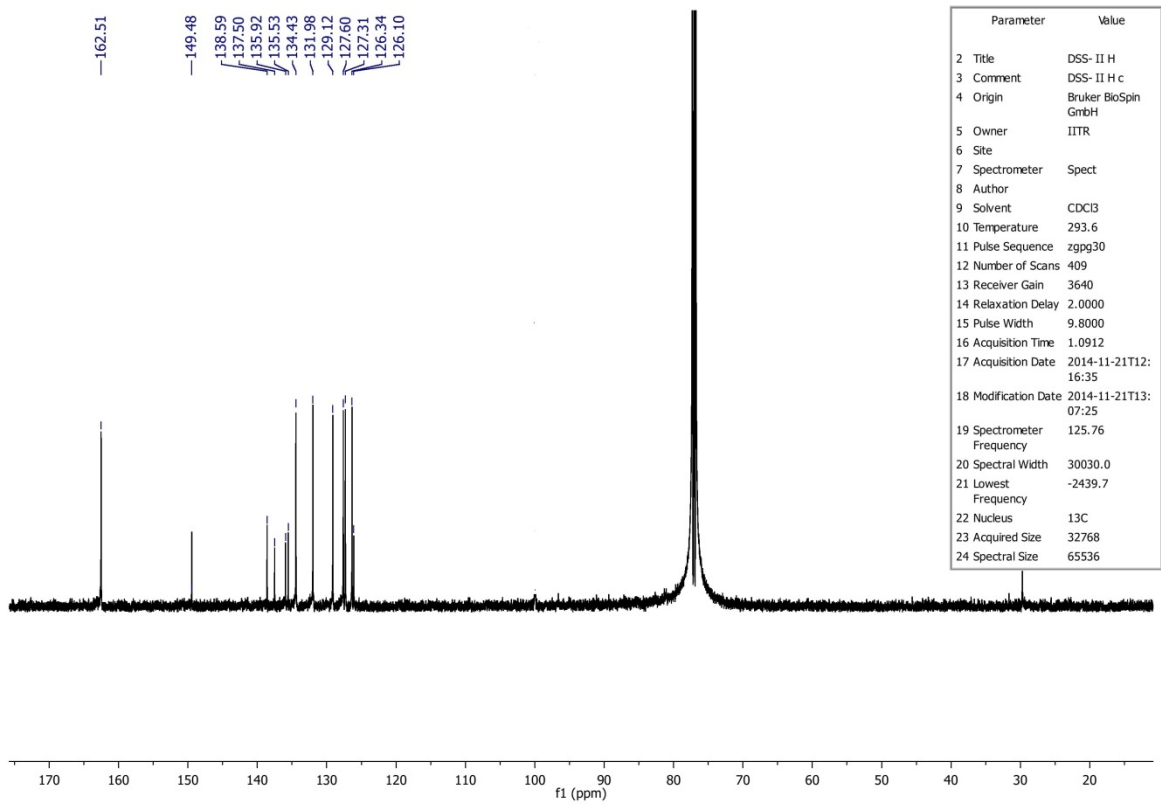
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5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
10 Temperature	293.5
11 Pulse Sequence	zgpg30
12 Number of Scans	129
13 Receiver Gain	3640
14 Relaxation Delay	2.0000
15 Pulse Width	9.8000
16 Acquisition Time	1.0912
17 Acquisition Date	2014-11-21T11:05:20
18 Modification Date	2014-11-21T11:39:49
19 Spectrometer	125.76
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23 Nucleus	13C
24 Acquired Size	32768
25 Spectral Size	65536



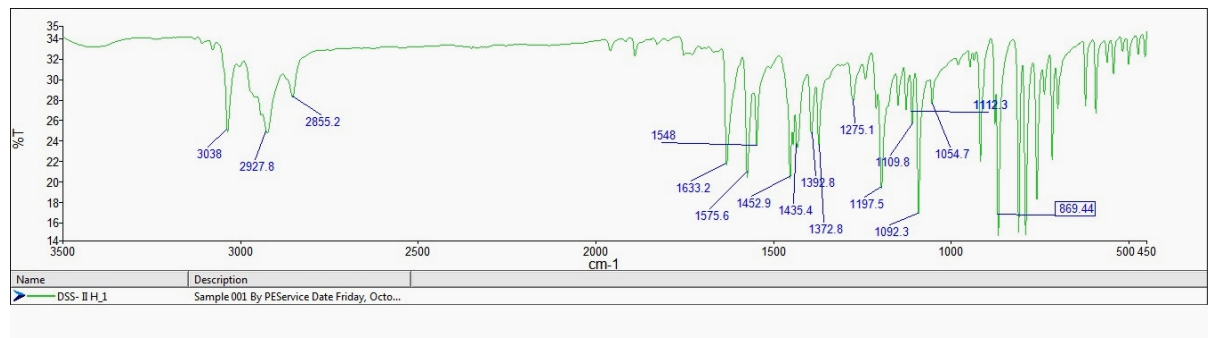


7) 4,8-dichlorodibenzo[b,f][1,4]thiazepine 3g



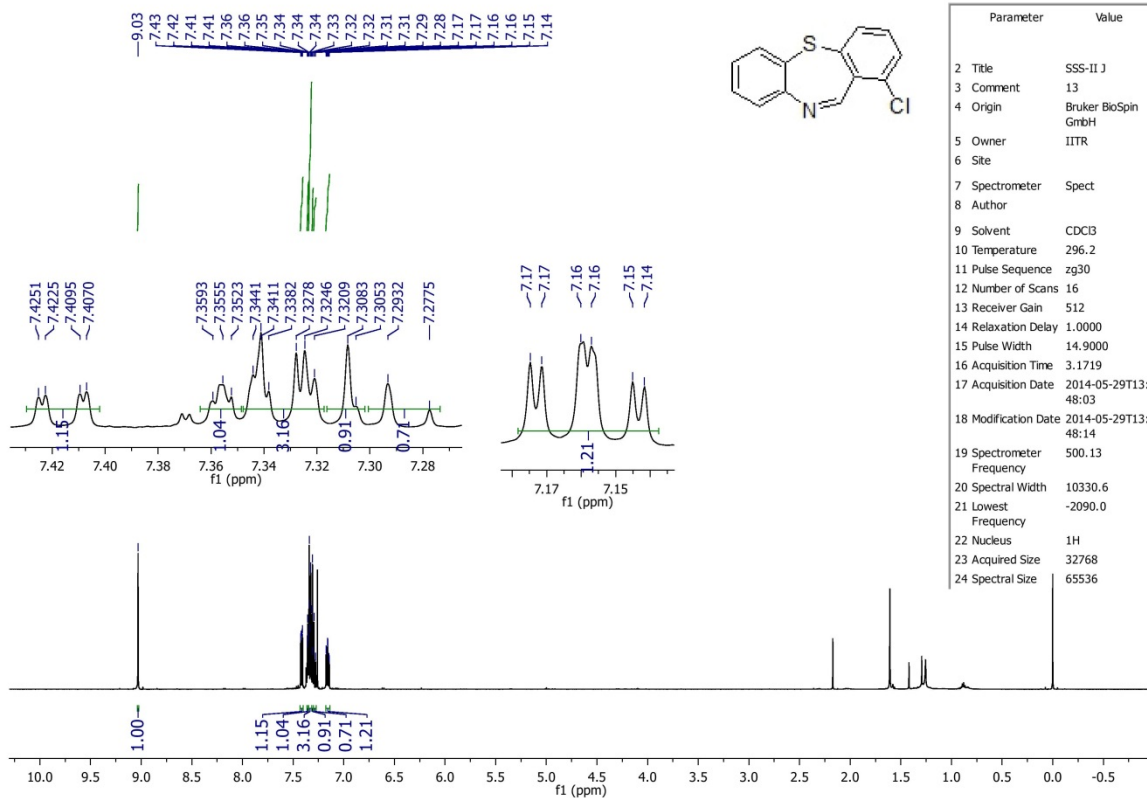


Parameter	Value
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6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
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11 Pulse Sequence	zgpg30
12 Number of Scans	409
13 Receiver Gain	3640
14 Relaxation Delay	2.0000
15 Pulse Width	9.8000
16 Acquisition Time	1.0912
17 Acquisition Date	2014-11-21T12:16:35
18 Modification Date	2014-11-21T13:07:25
19 Spectrometer Frequency	125.76
20 Spectral Width	30030.0
21 Lowest Frequency	-2439.7
22 Nucleus	13C
23 Acquired Size	32768
24 Spectral Size	65536



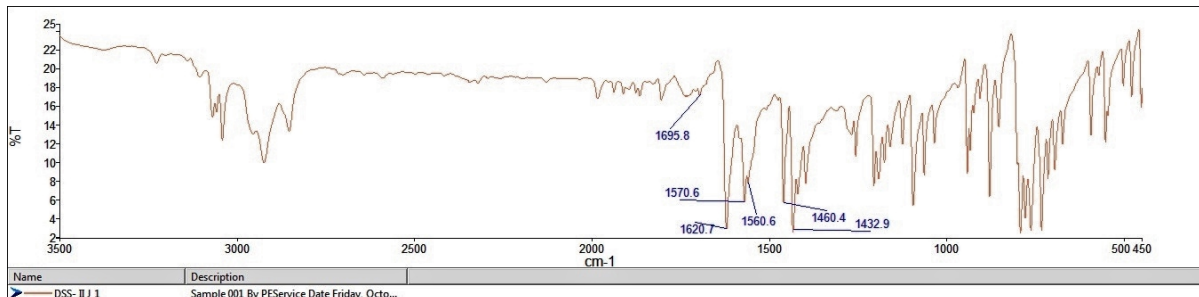
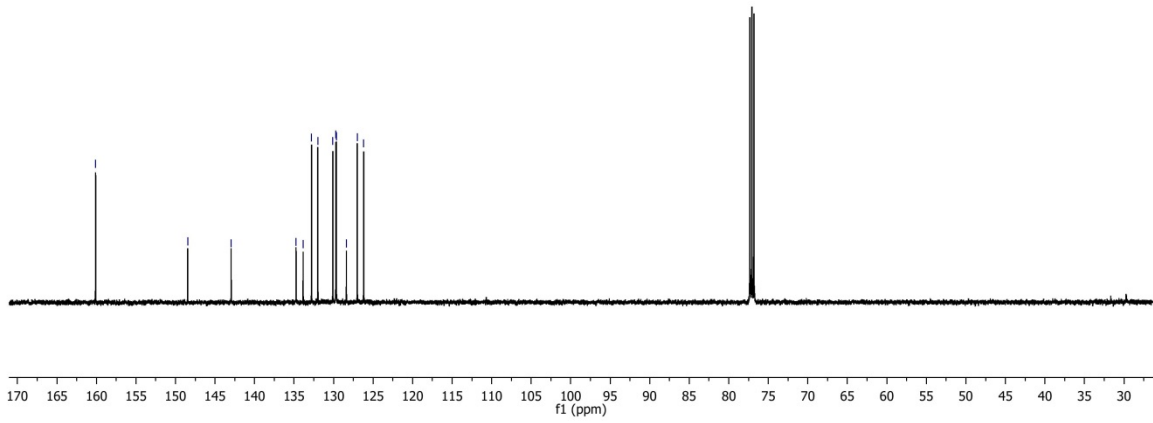
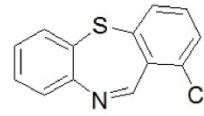
Name	Description
DSS- II H_1	Sample 001 By PEService Date Friday, Octo...

8)1-chlorodibenzo[b,f][1,4]thiazepine 3h

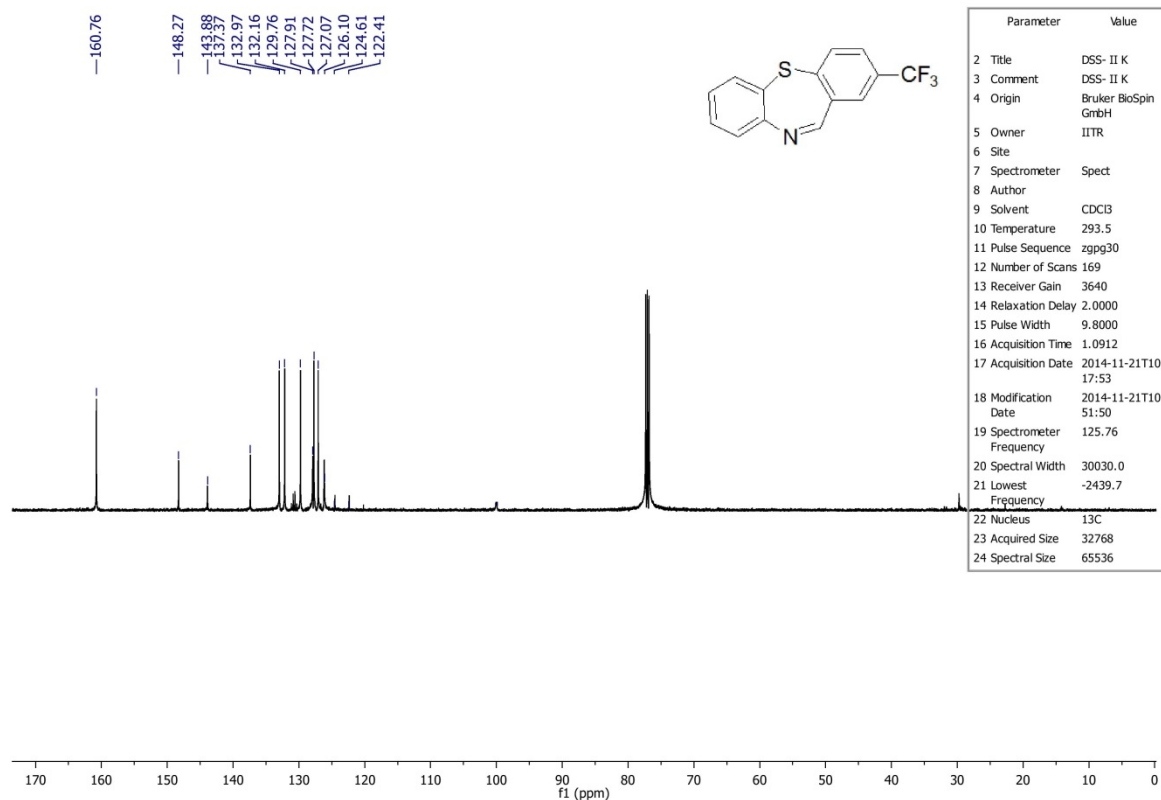
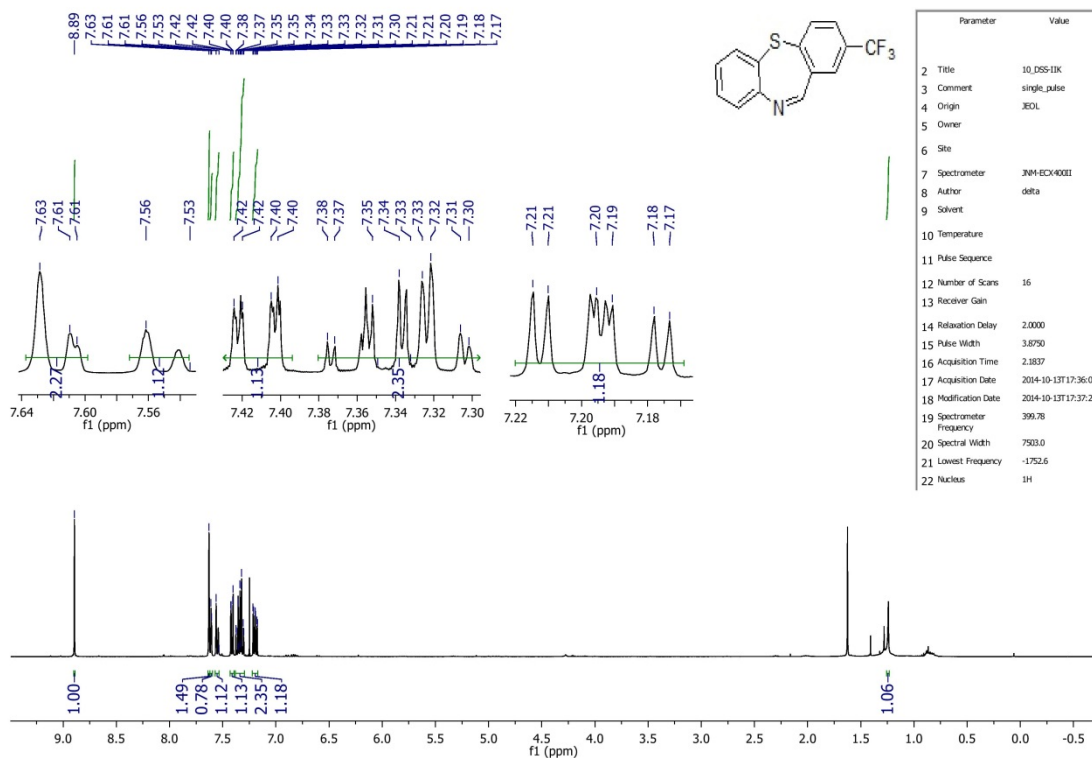


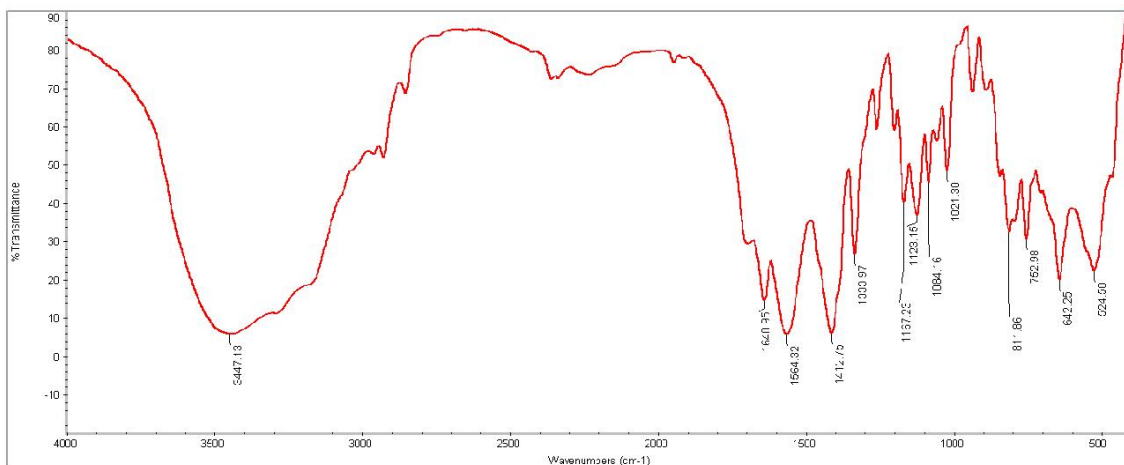
DSS- II J
DSS- II J c

- 160.11
- 148.45
- 142.96
- 134.75
- 133.85
- 132.79
- 131.99
- 130.10
- 129.71
- 129.65
- 128.38
- 127.00
- 126.19

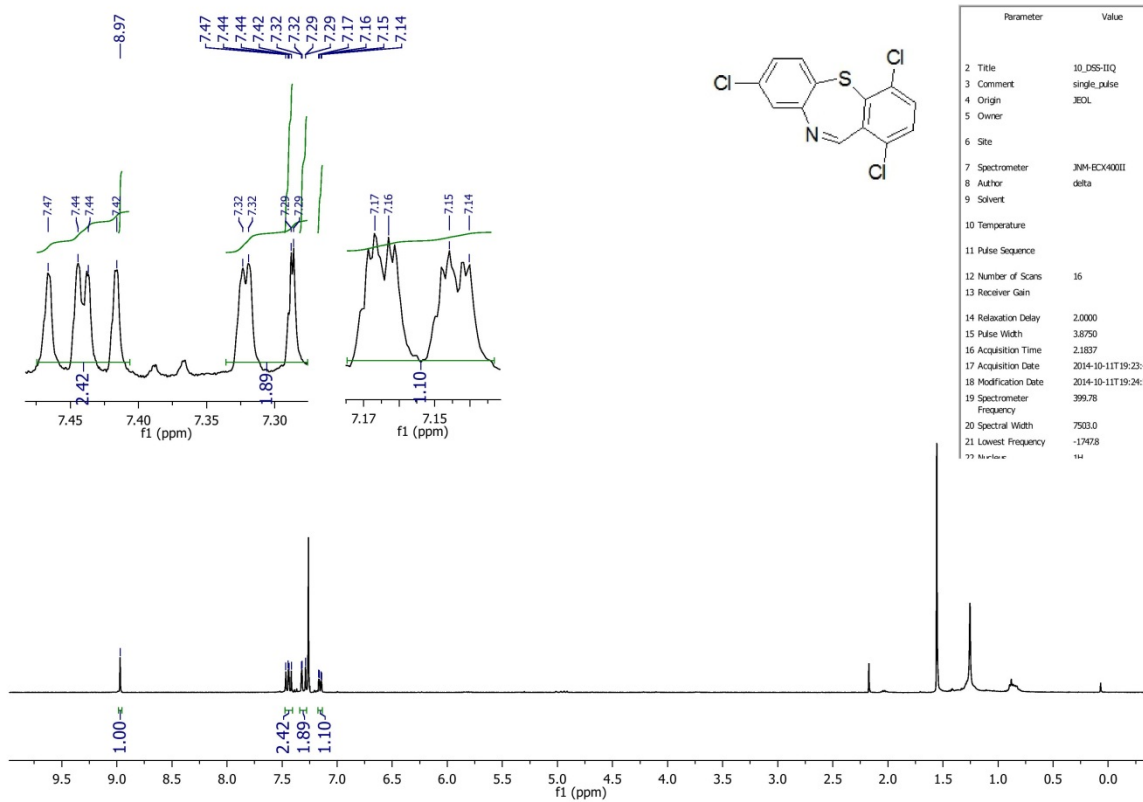


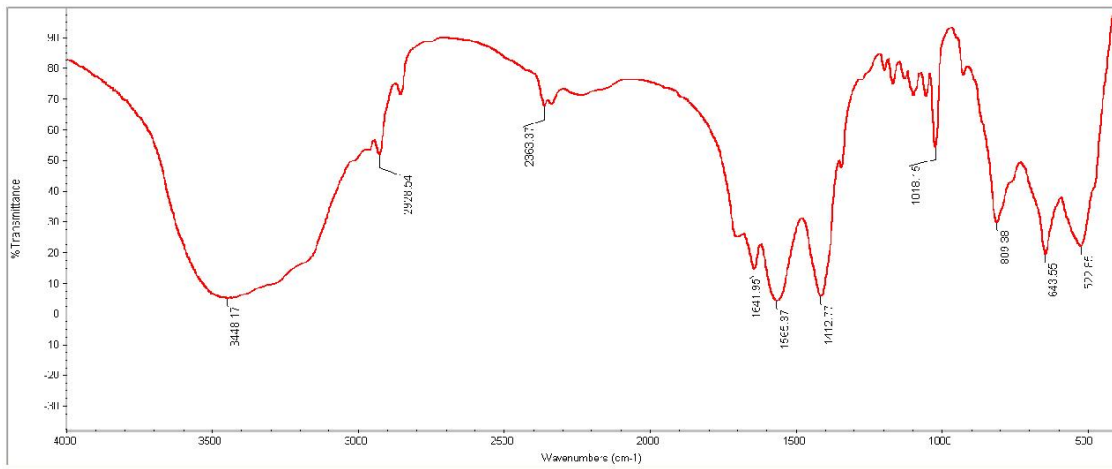
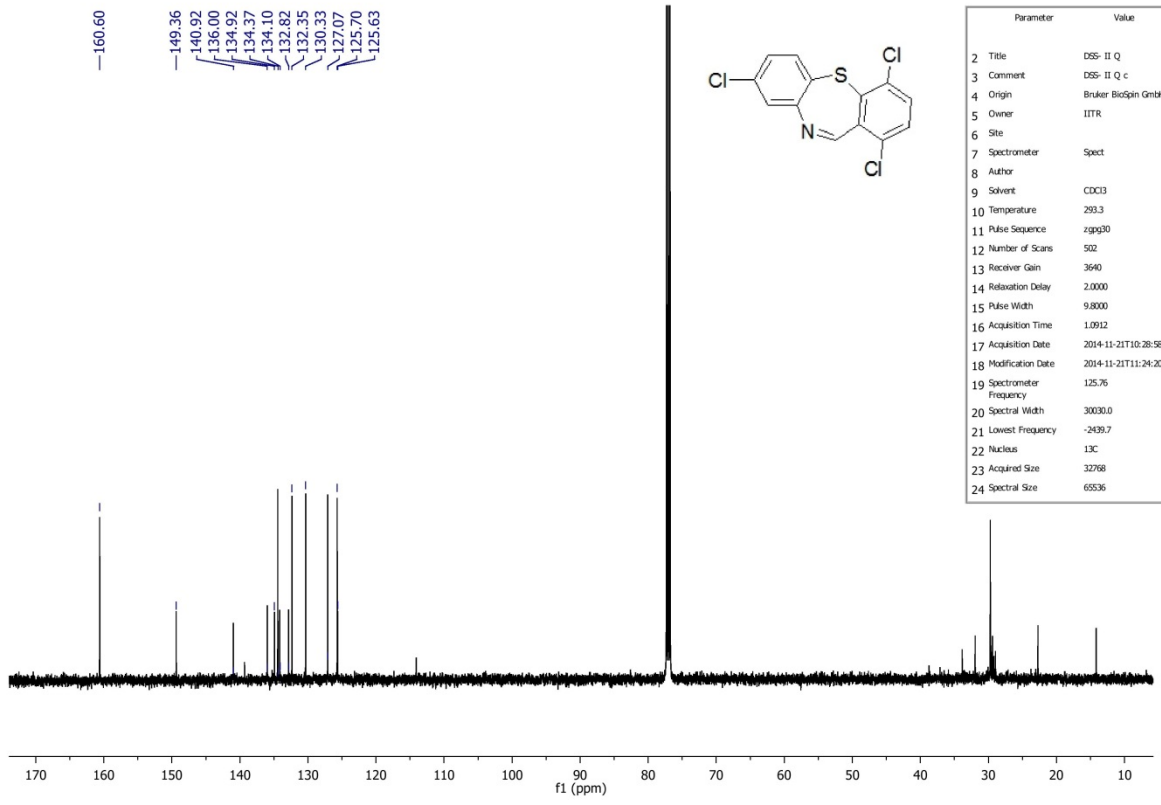
9) 4-chloro-8-(trifluoromethyl)dibenzo[b,f][1,4]thiazepine 3i



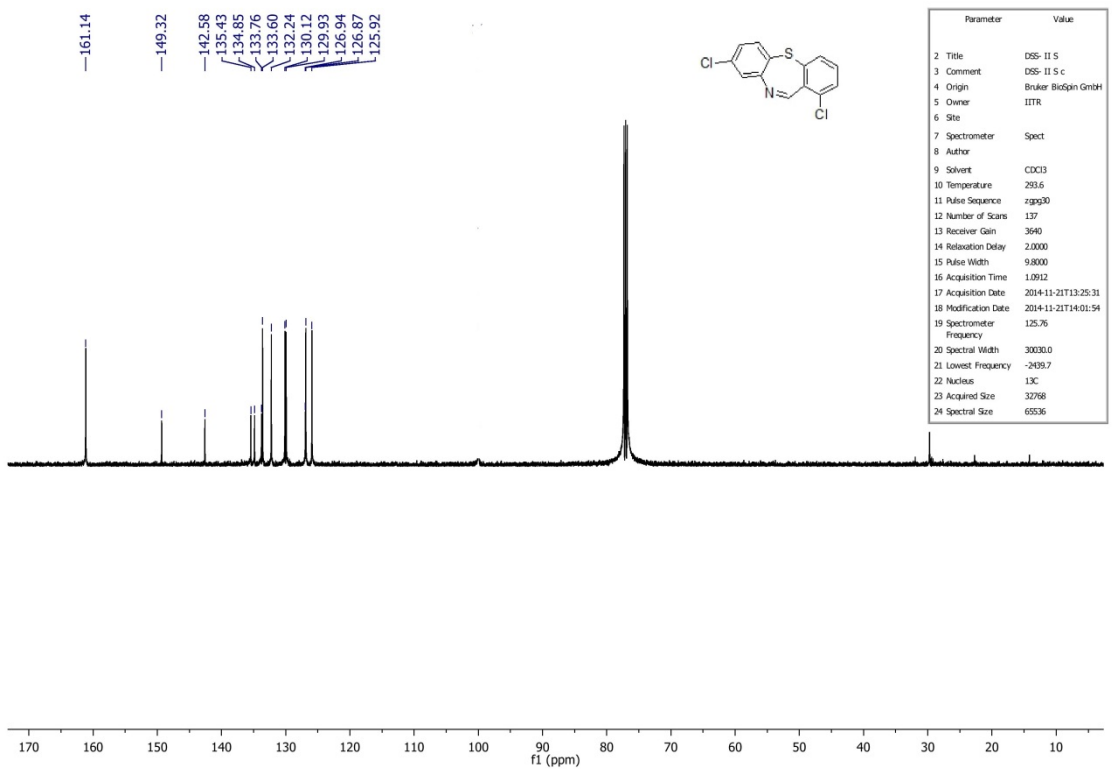
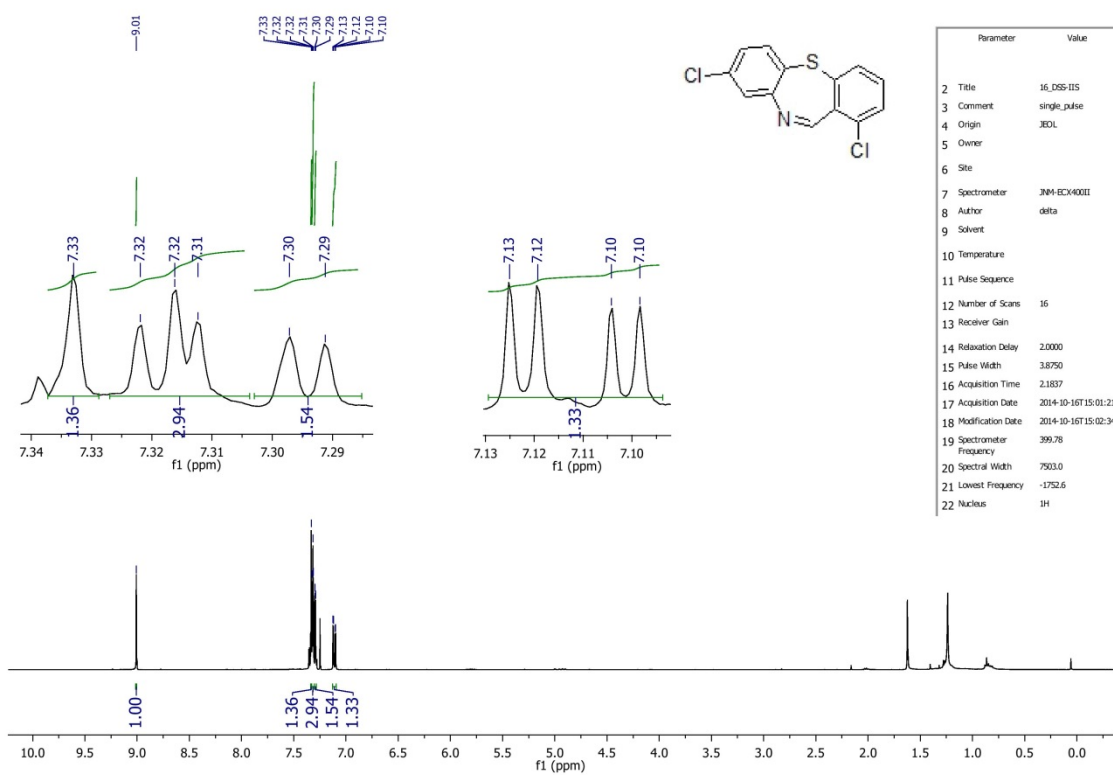


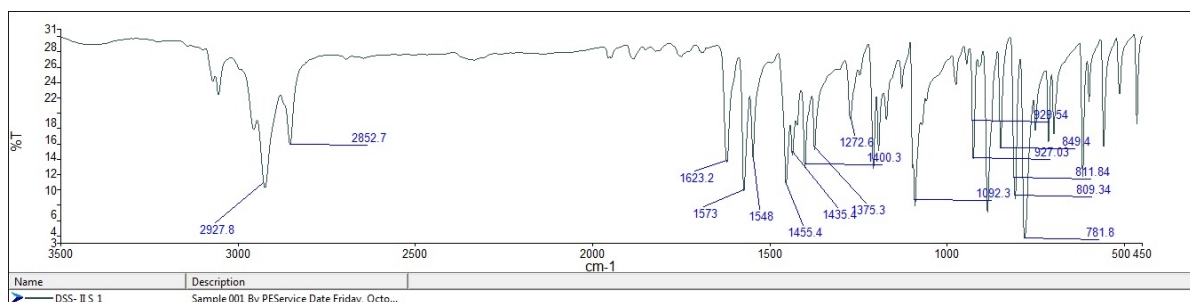
10) 1,4,8-trichlorodibenzo[*b,f*][1,4]thiazepine 3j



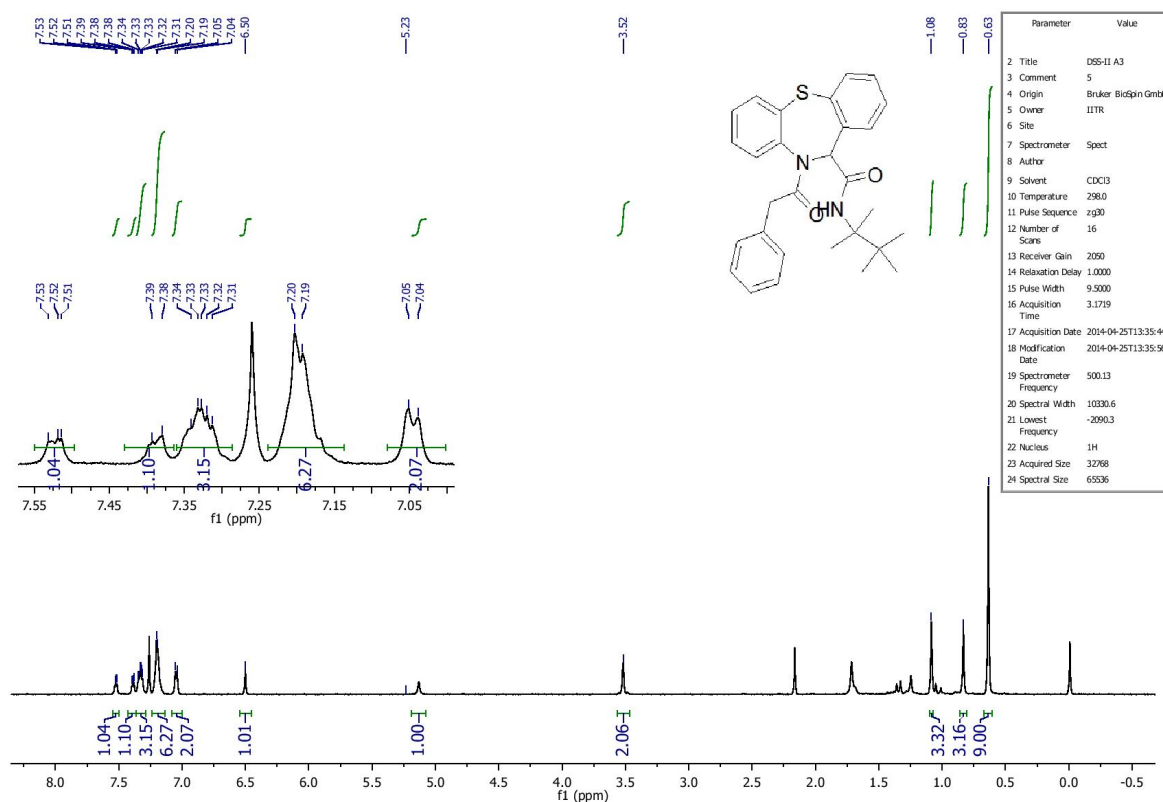


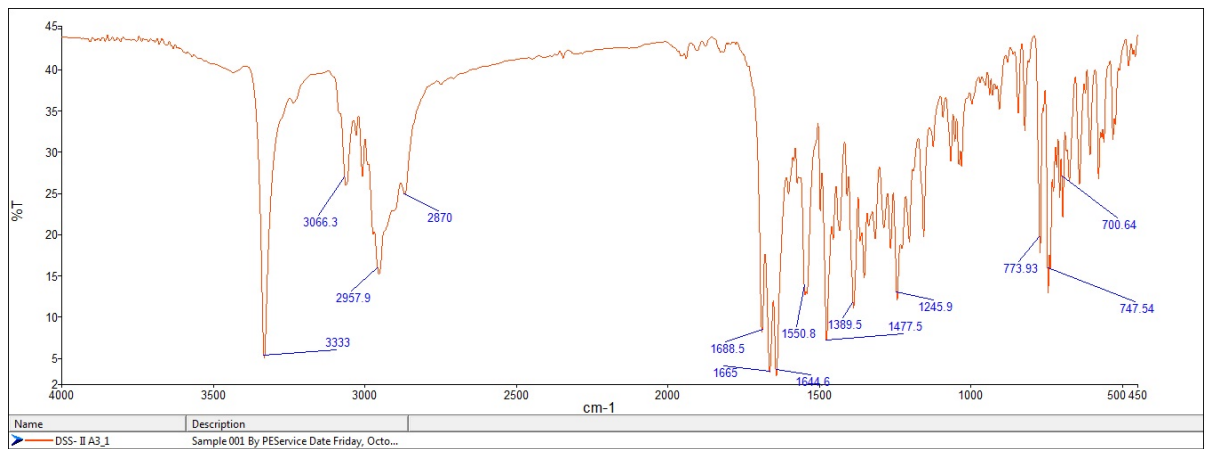
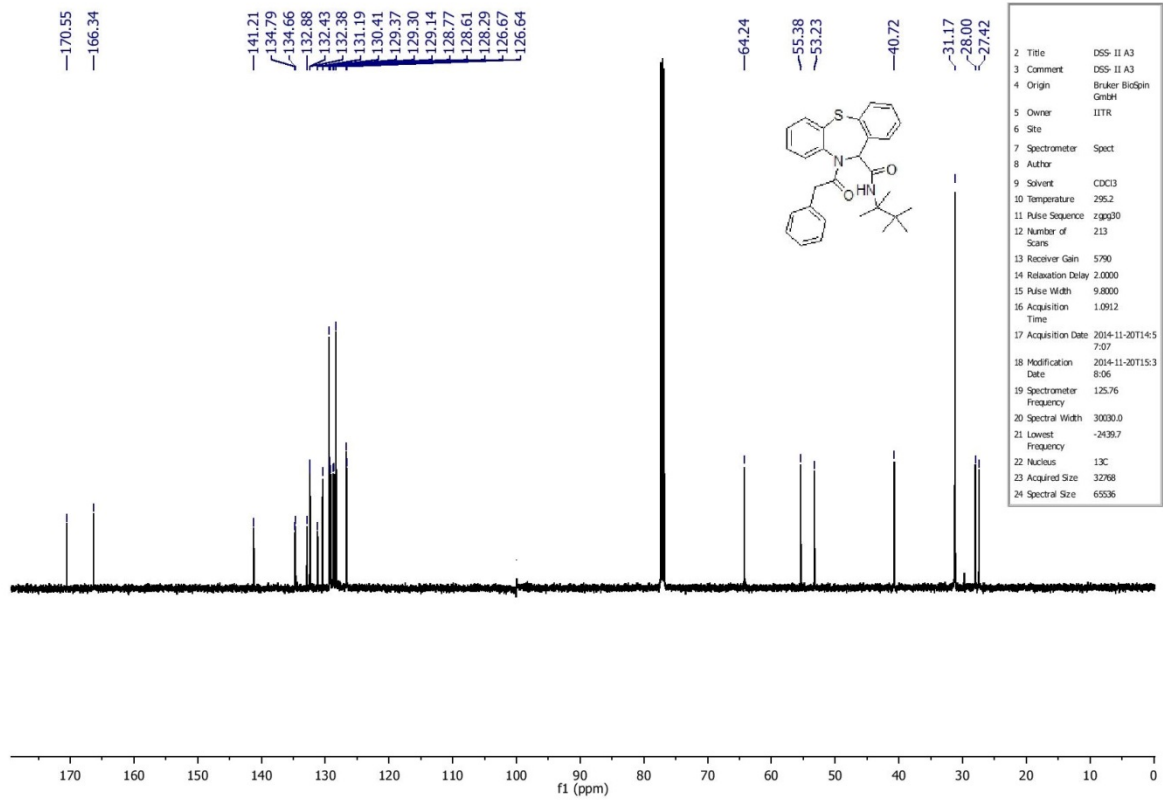
11)1,8-dichlorodibenzo[b,f][1,4]thiazepine 3k



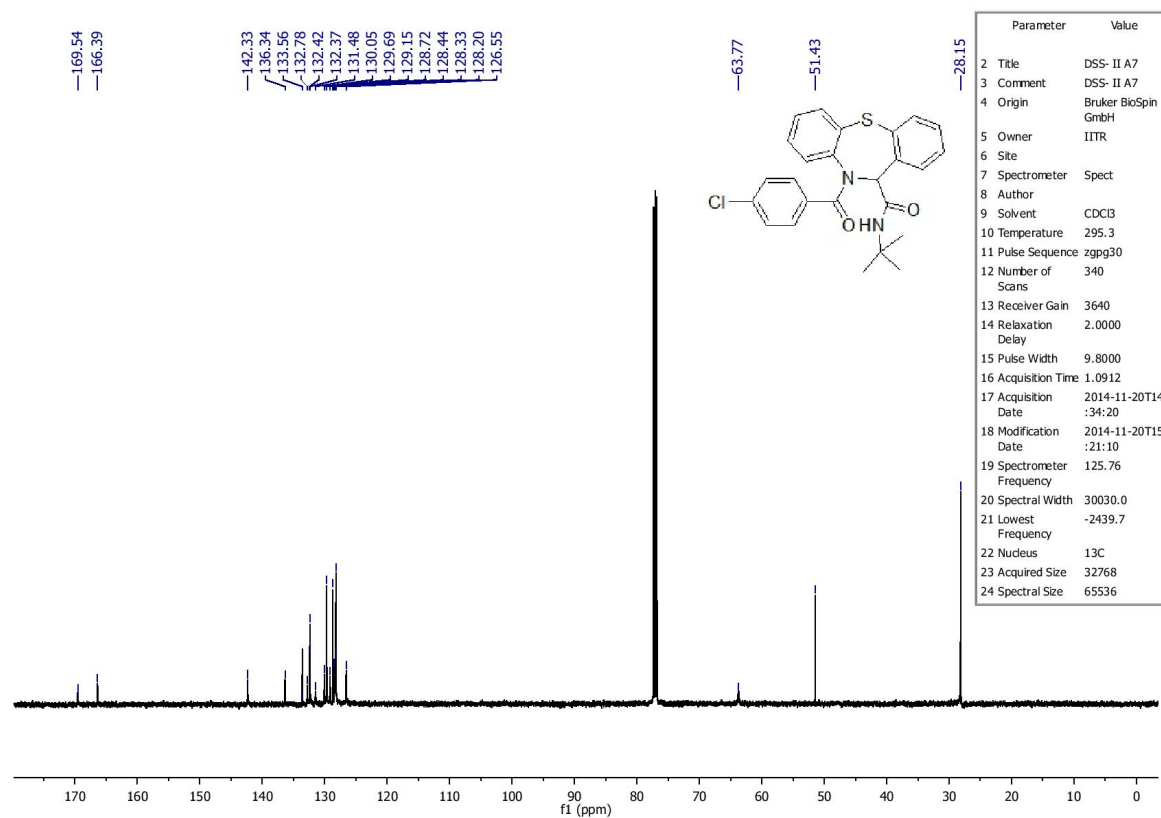
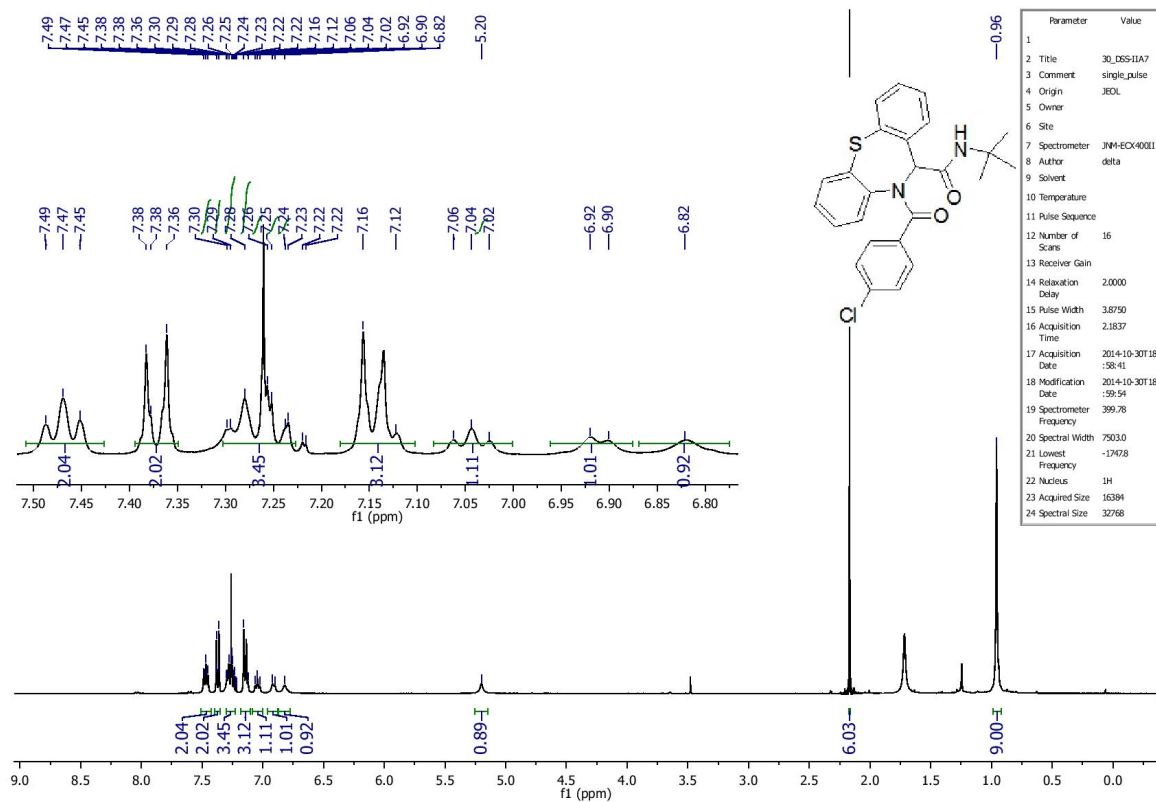


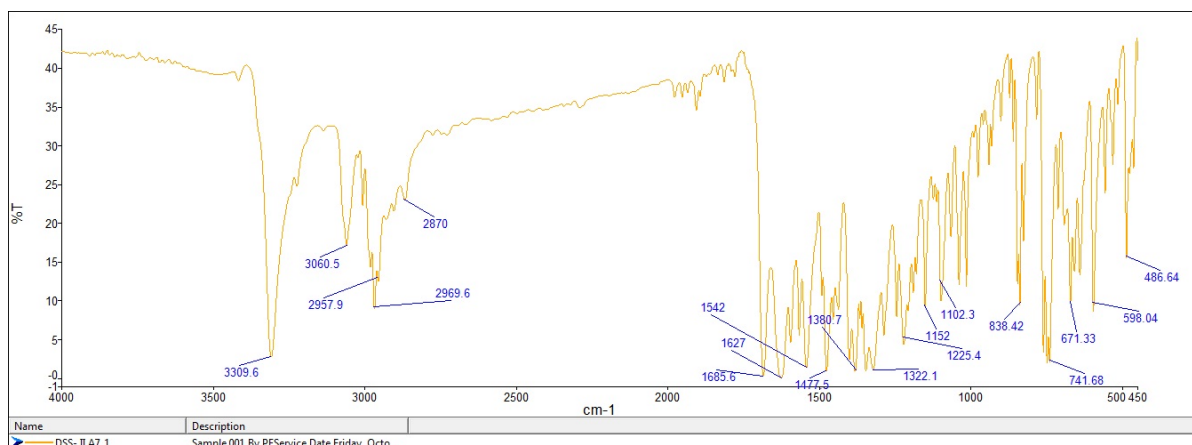
1) 10-(2-phenylacetyl)-N-(2,3,3-trimethylbutan-2-yl)-10,11 dihydrodibenzo[b,f][1,4]thiazepine-11-carboxamide 6a



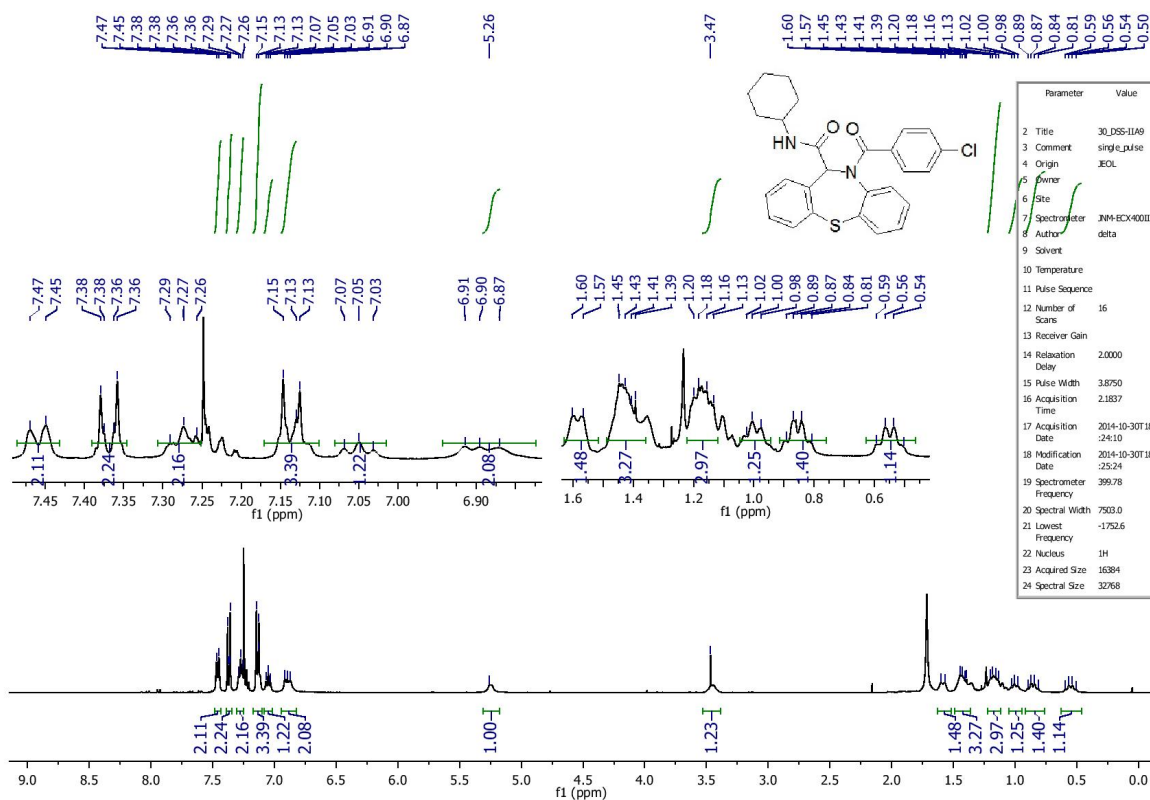


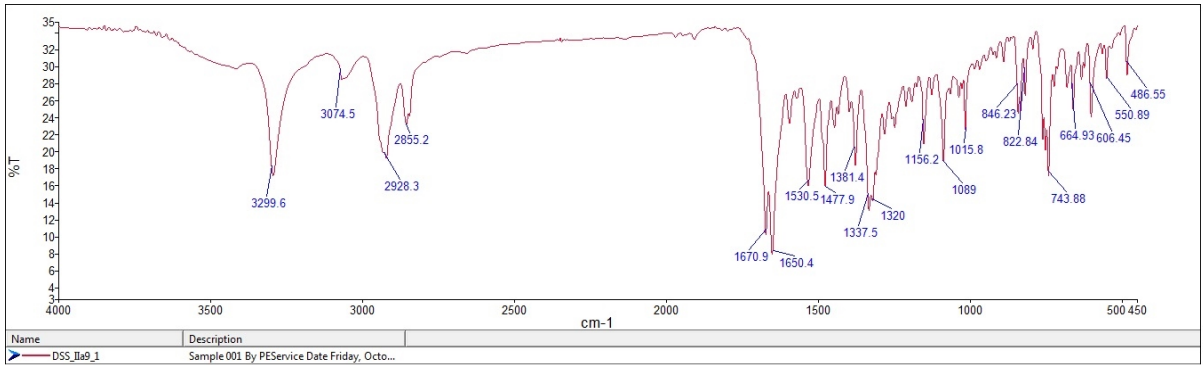
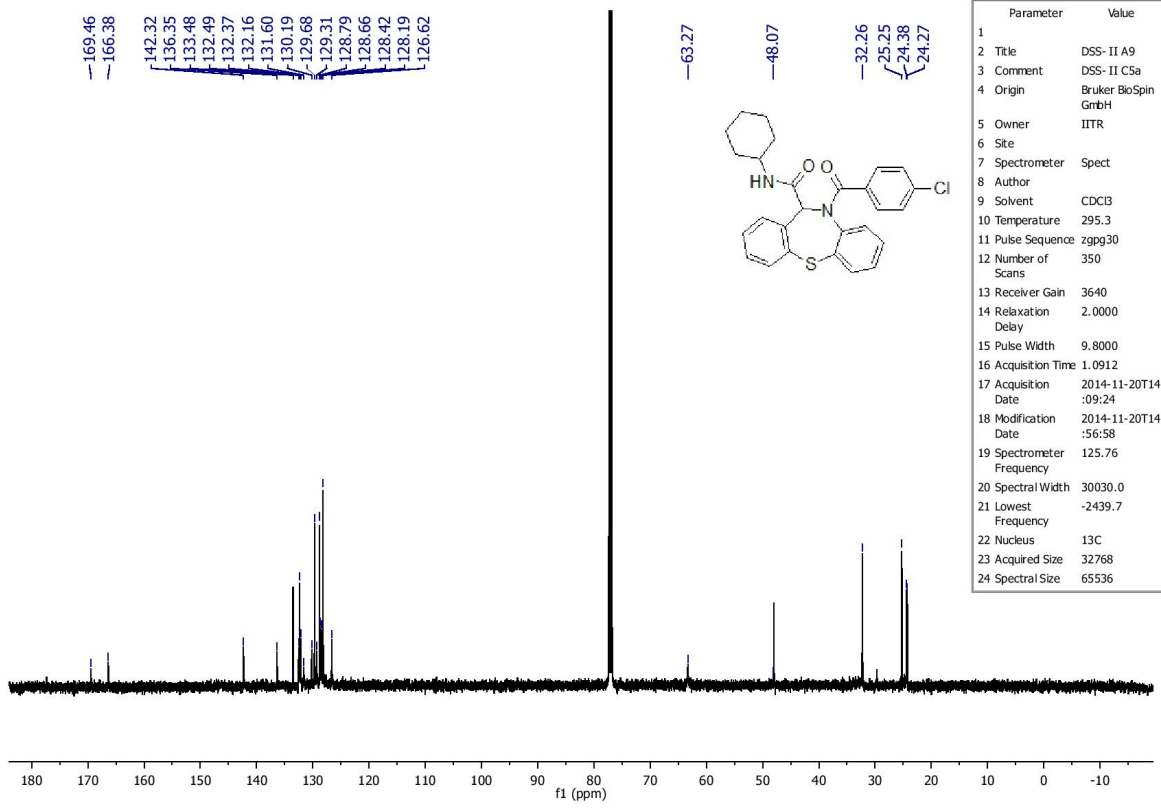
2) *N*-(*tert*-butyl)-10-(4-chlorobenzoyl)-10,11-dihydrodibenzo[*b,f*][1,4]thiazepine-11 carboxamide **6b**



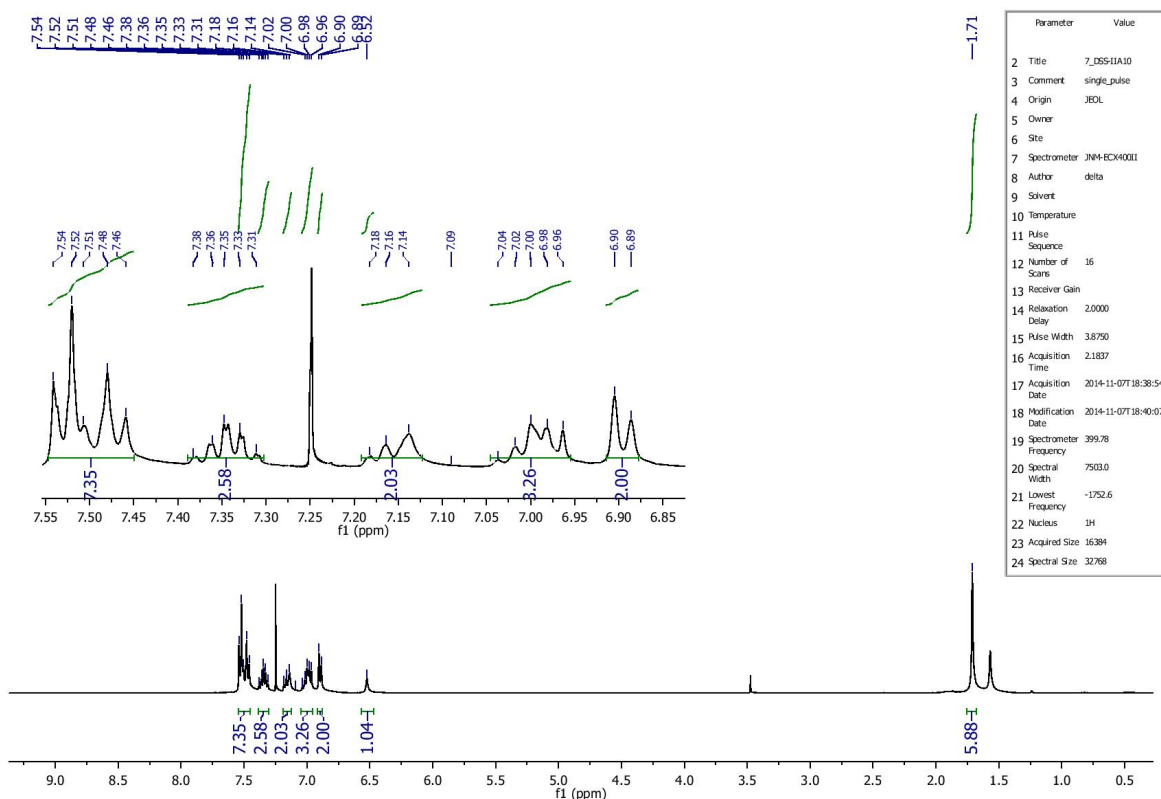


3) 10-(4-chlorobenzoyl)-*N*-cyclohexyl-10,11-dihydrobenzo[*b,f*][1,4]thiazepine-11-carboxamide 6c

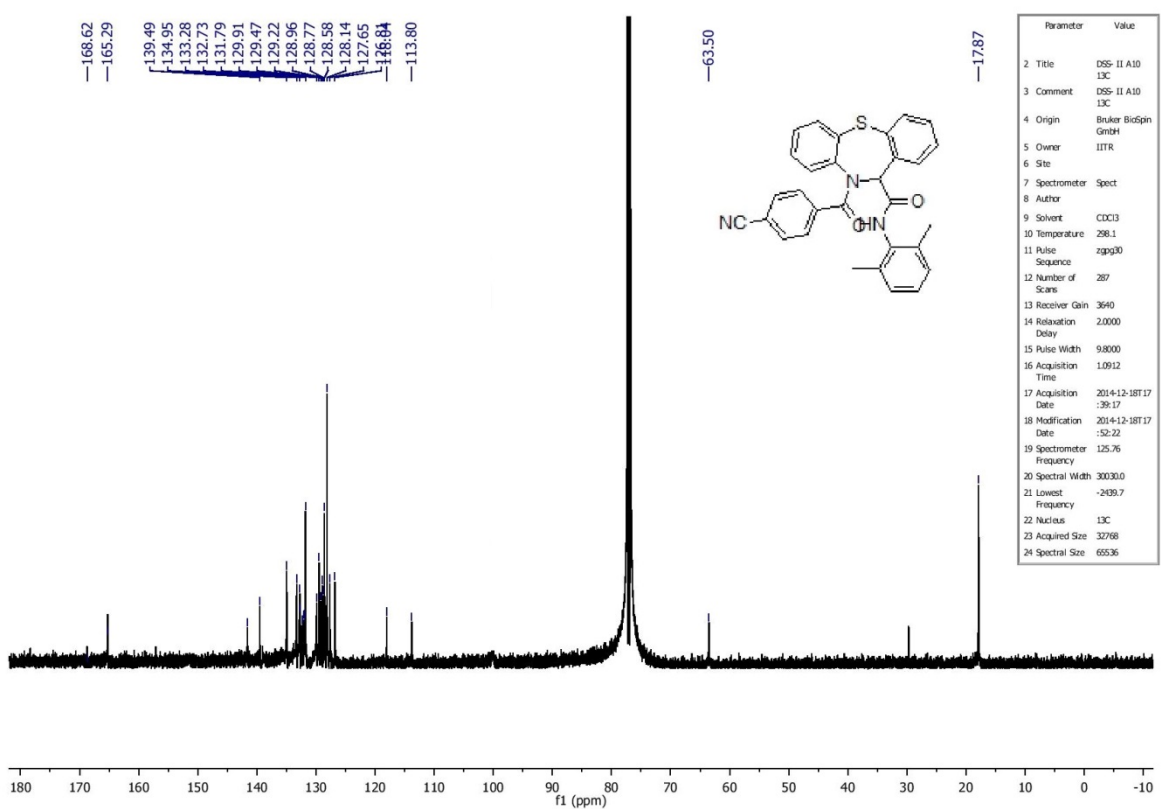




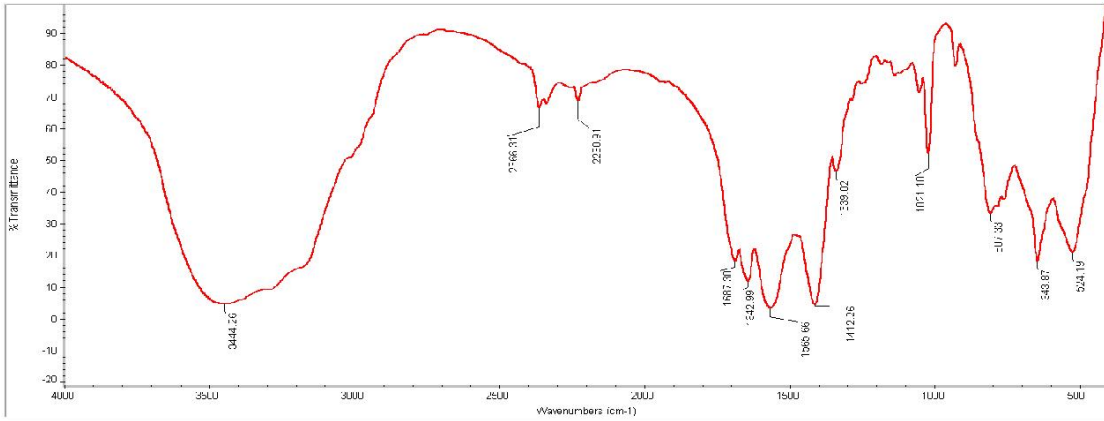
4) 10-(4-cyanobenzoyl)-*N*-(2,6-dimethylphenyl)-10,11-dihydrodibenzo[*b,f*][1,4]thiazepine-11-carboxamide **6d**



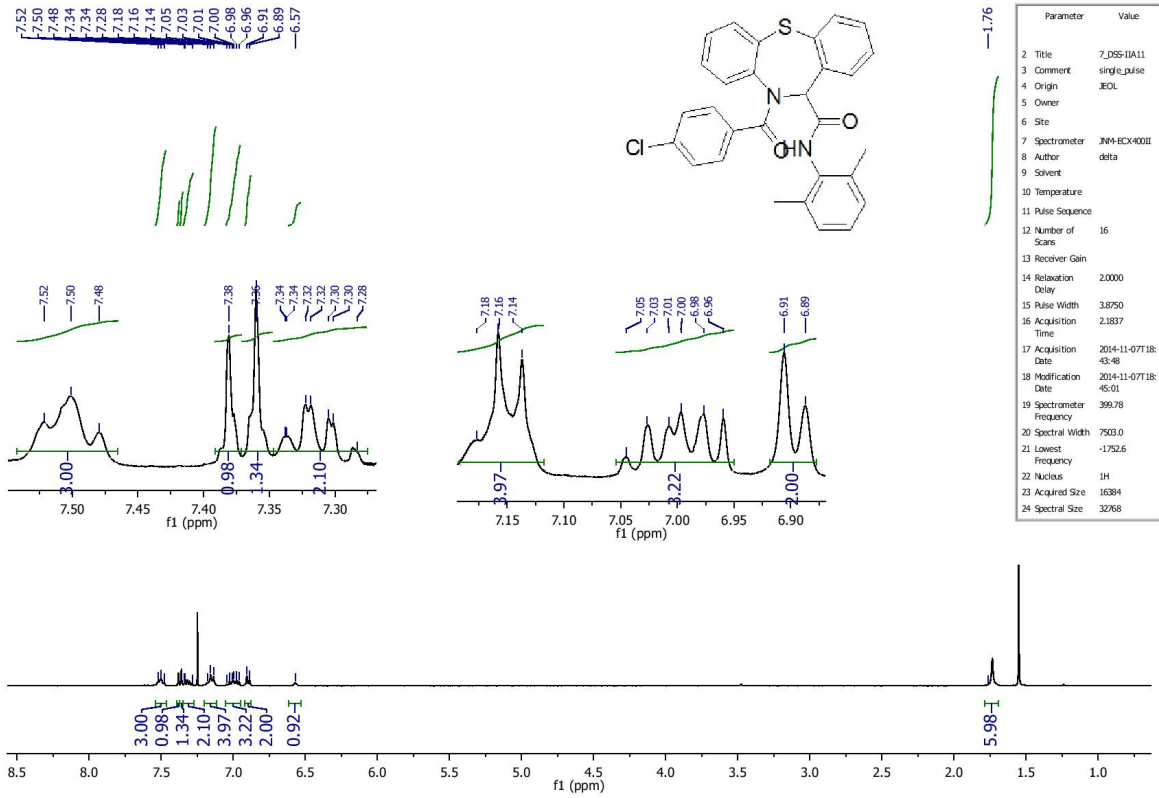
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8 Author	delta
9 Solvent	
10 Temperature	
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13 Receiver Gain	
14 Relaxation Delay	2.0000
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16 Acquisition Time	2.1837
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22 Nucleus	1H
23 Acquired Size	16394
24 Spectral Size	32768

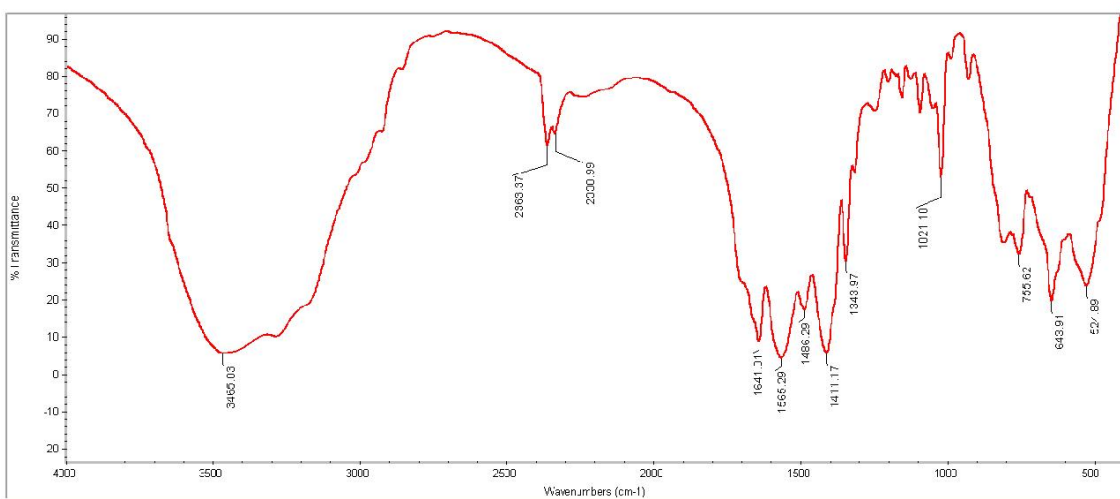
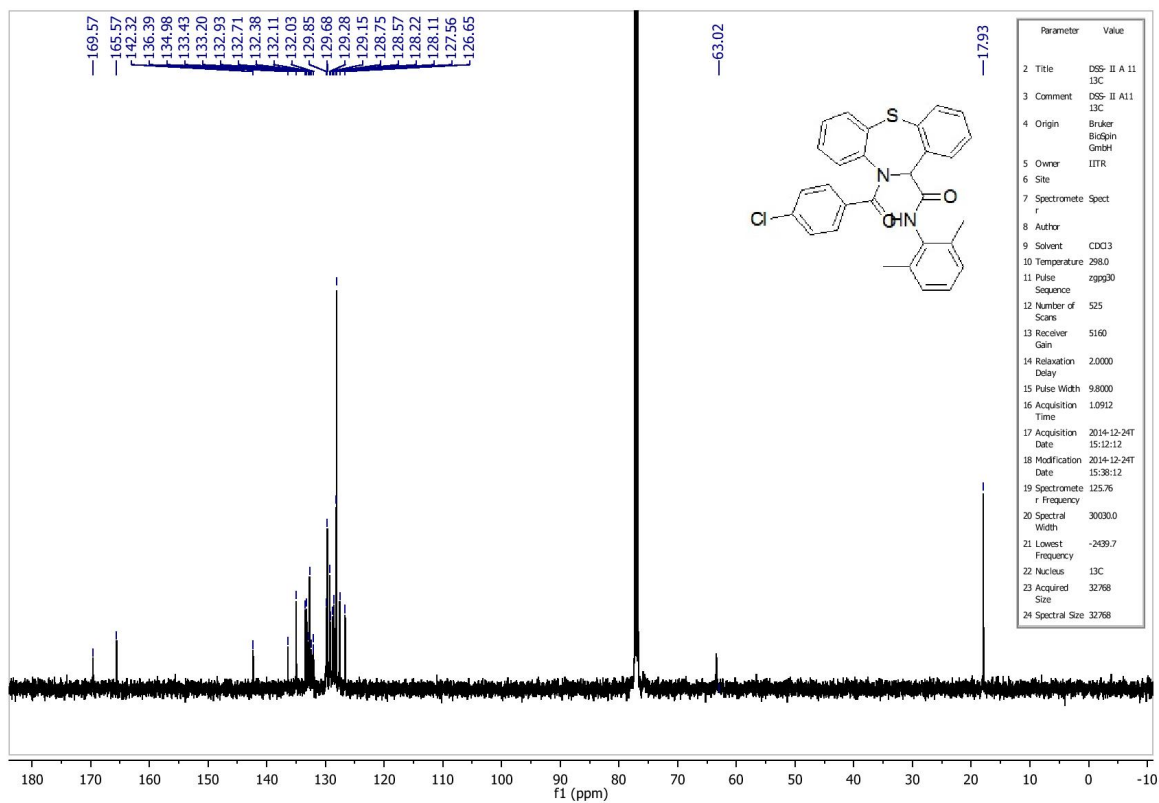


Parameter	Value
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3 Comment	13C
4 Origin	Bruker BioSpin GmbH
5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
10 Temperature	296.1
11 Pulse Sequence	zgpg30
12 Number of Scans	287
13 Receiver Gain	3640
14 Relaxation Delay	2.0000
15 Pulse Width	9.8000
16 Acquisition Time	1.0912
17 Acquisition Date	2014-12-18T17:39:17
18 Modification Date	2014-12-18T17:52:22
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21 Lowest Frequency	-2499.7
22 Nucleus	13C
23 Acquired Size	32768
24 Spectral Size	65536

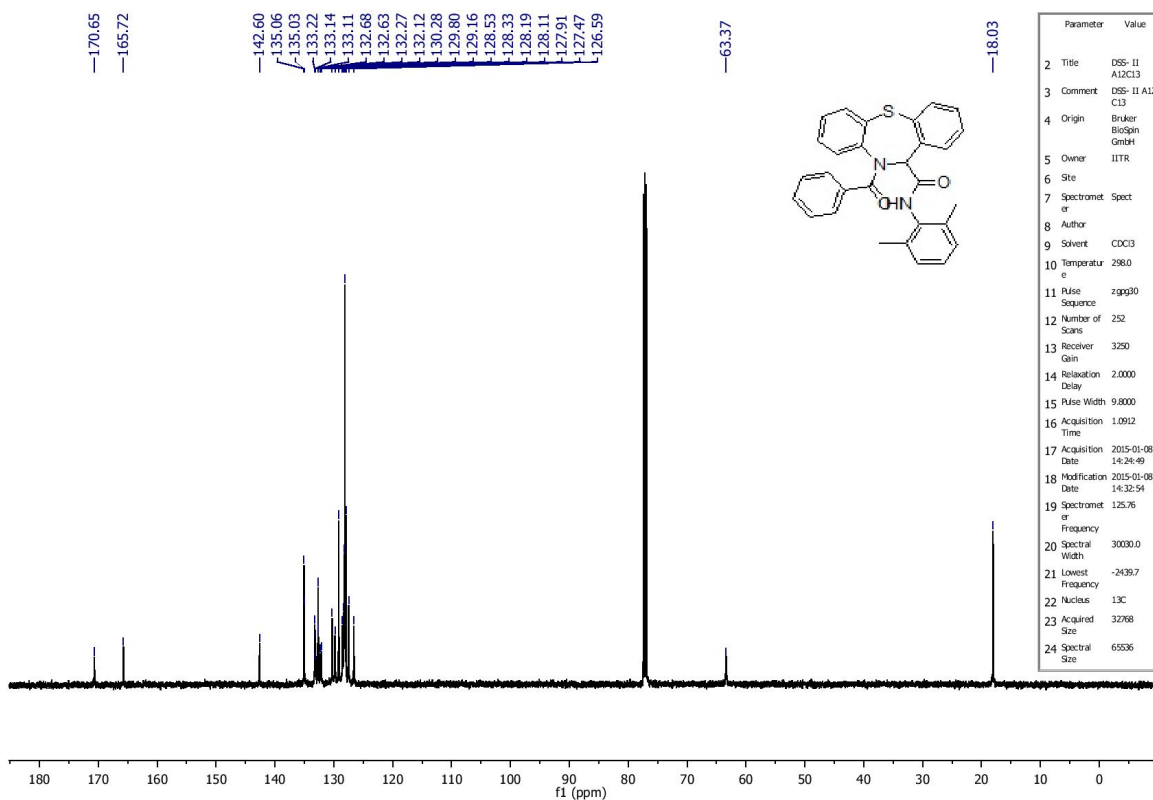
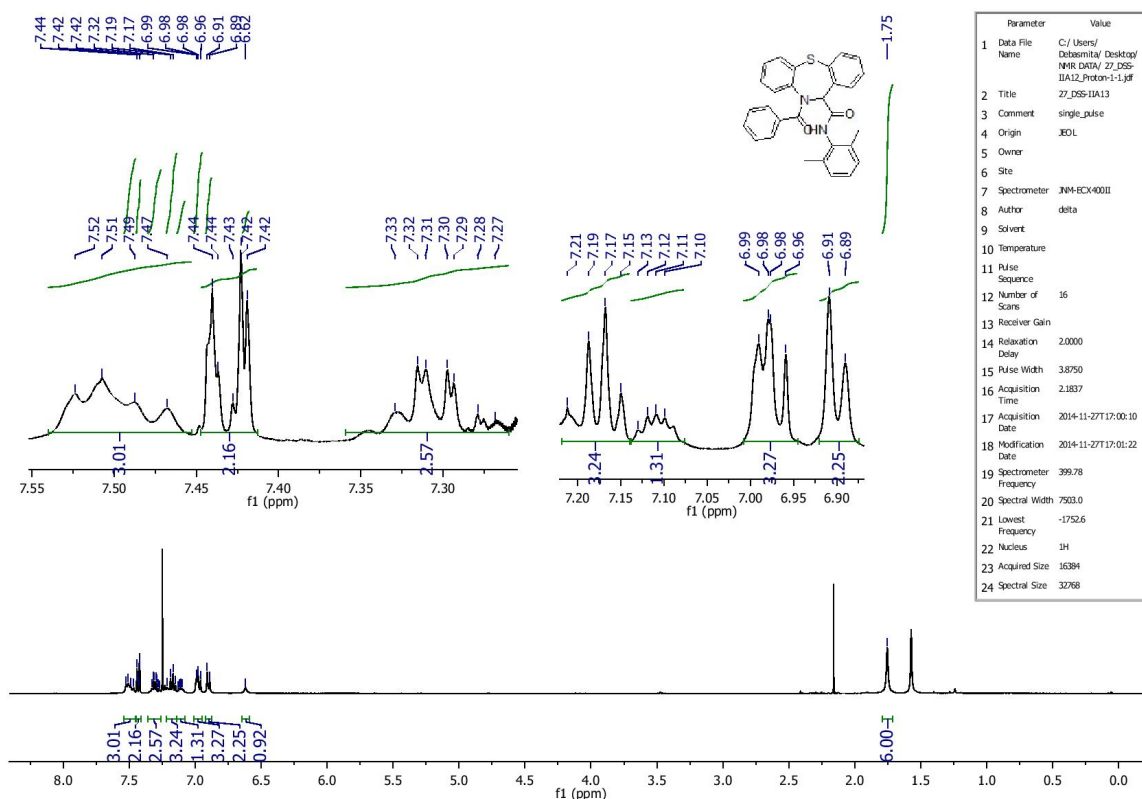


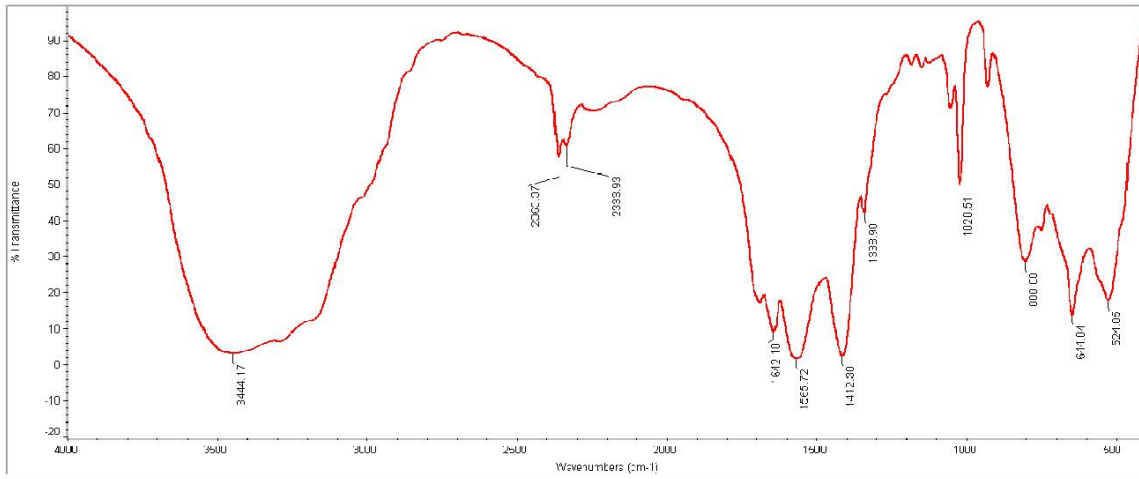
5) 10-(4-chlorobenzoyl)-N-(2,6-dimethylphenyl)-10,11-dihydrodibenzo[b,f][1,4]thiazepine-11-carboxamide 6c



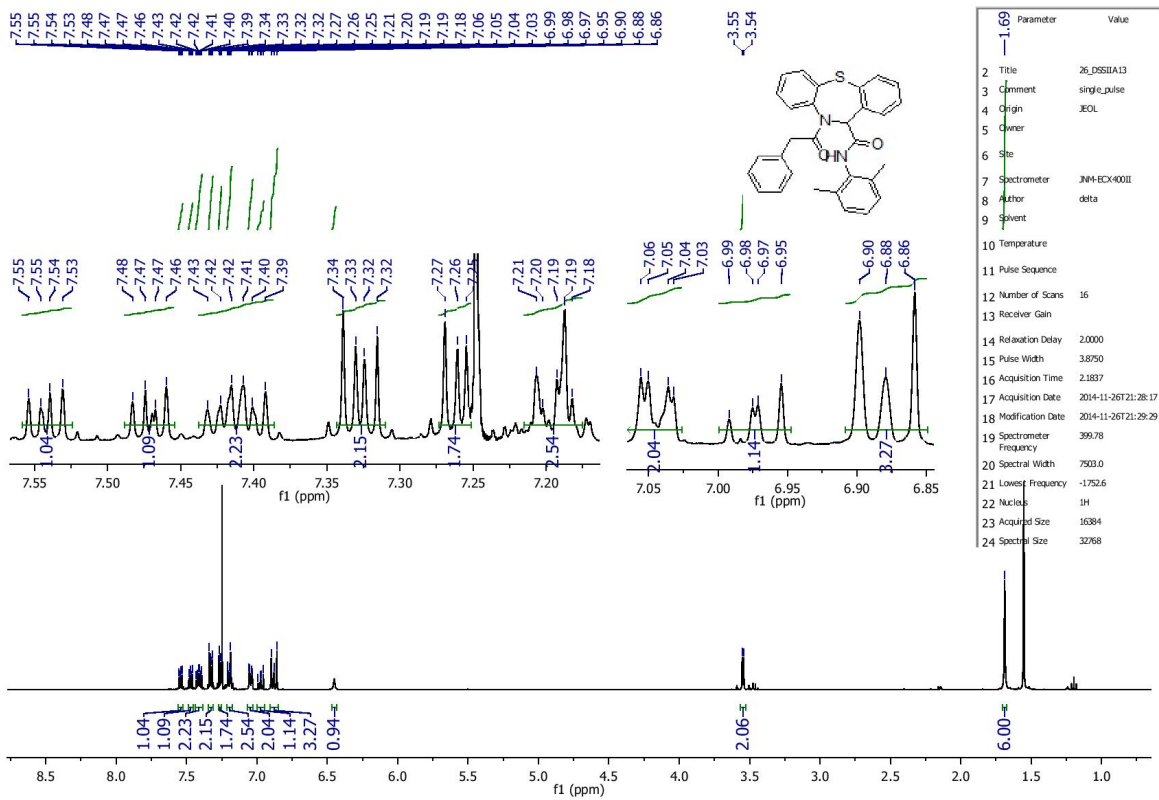


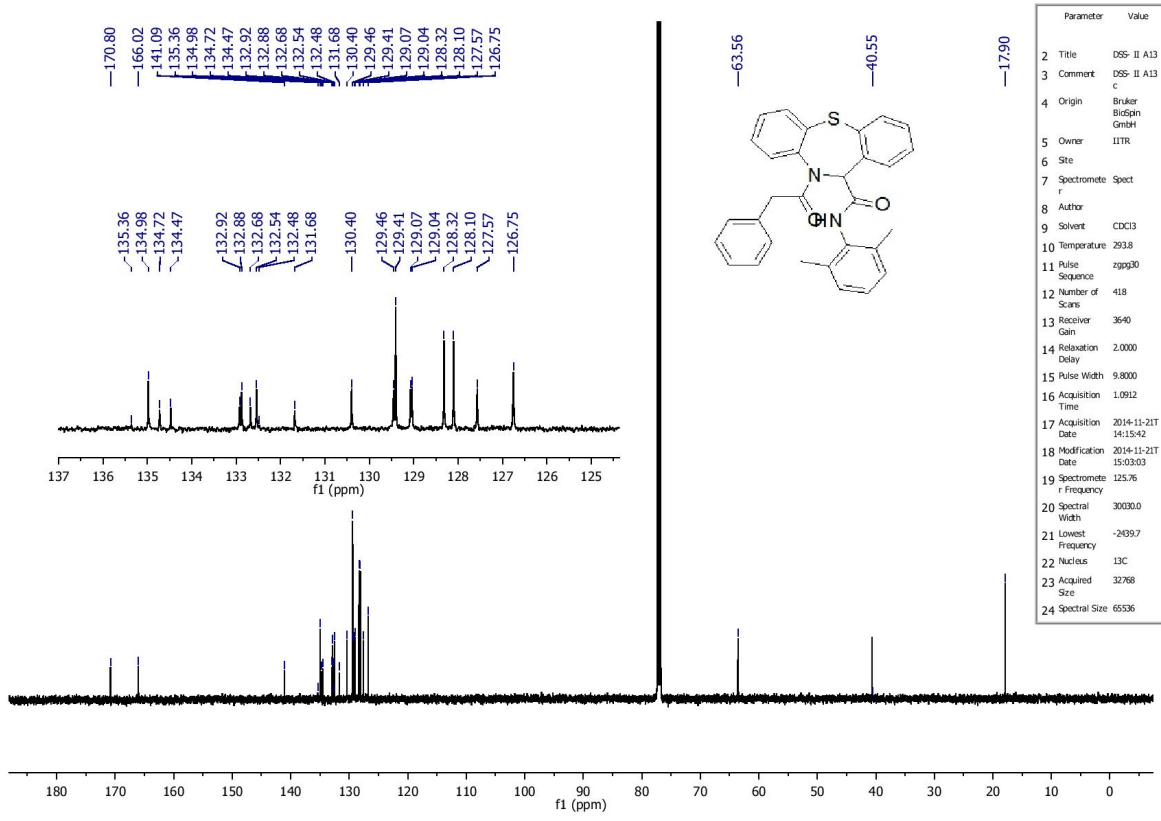
6) 10-benzoyl-N-(2,6-dimethylphenyl)-10,11-dihydrodibenzo[b,f][1,4]thiazepine-11-carboxamide 6f



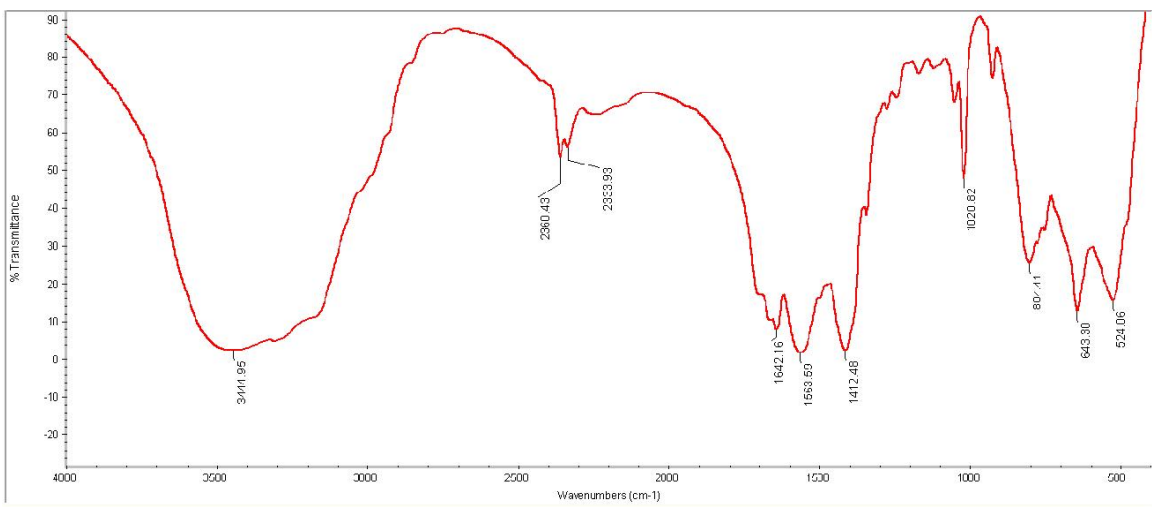


7) *N*-(2, 6-dimethylphenyl)-10-(2-phenylacetyl)-10, 11-dihydrodibenzo[*b,f*][1,4]thiazepine-11-carboxamide **6g**

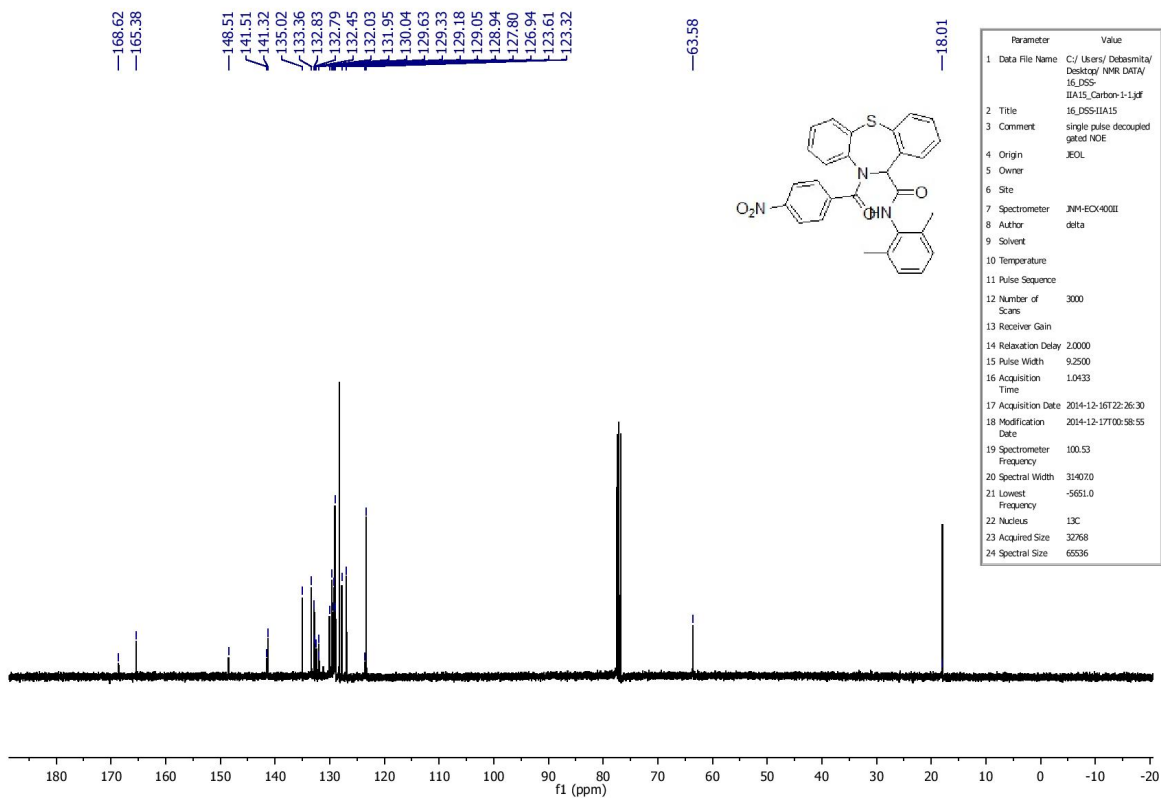
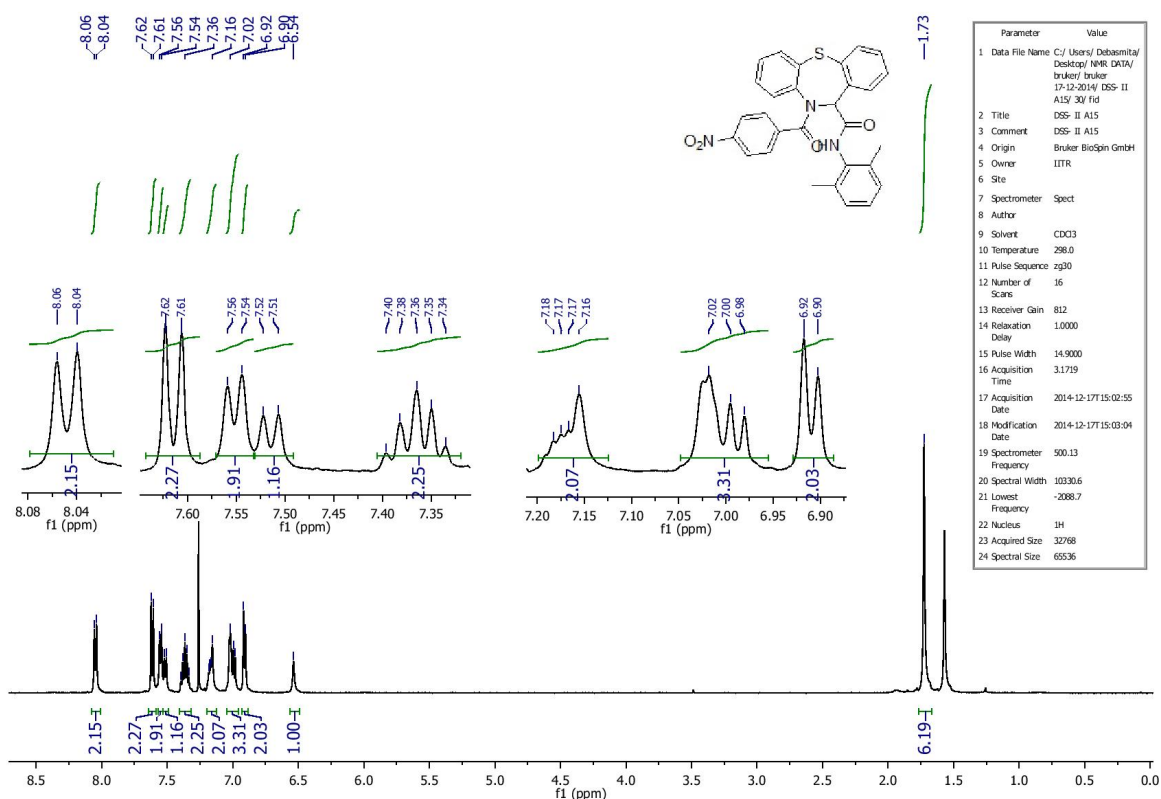


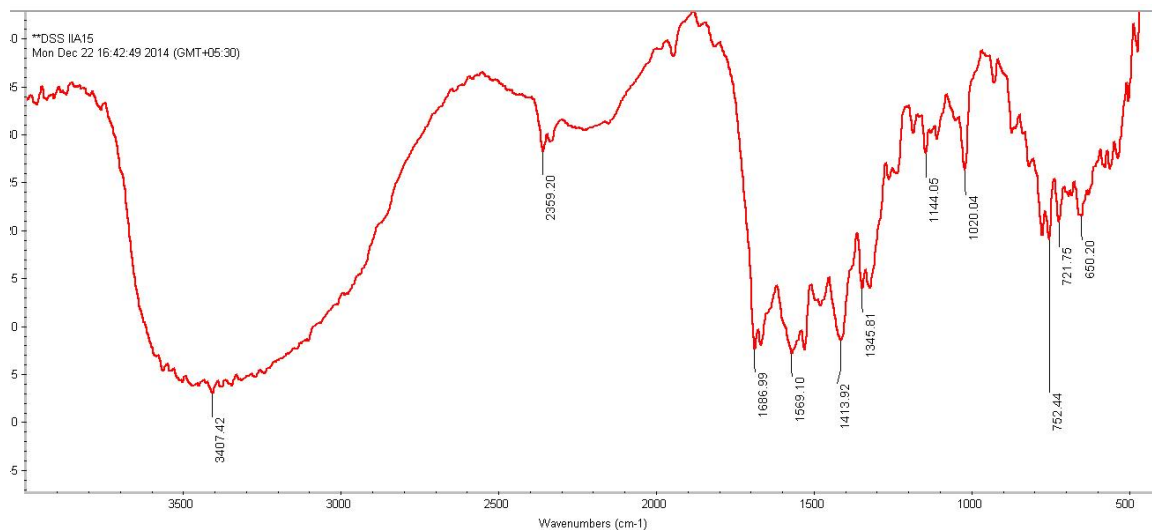


Parameter	Value
2 Title	DSS- II A13
3 Comment	DSS- II A13
4 Origin	Bruker BioSpin GmbH
5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
10 Temperature	293.8
11 Pulse Sequence	zgpg30
12 Number of Scans	418
13 Receiver Gain	3640
14 Relaxation Delay	2.0000
15 Pulse Width	9.8000
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20 Spectral Width	30000.0
21 Lowest Frequency	-2499.7
22 Nucleus	13C
23 Acquired Size	32768
24 Spectral Size	65536

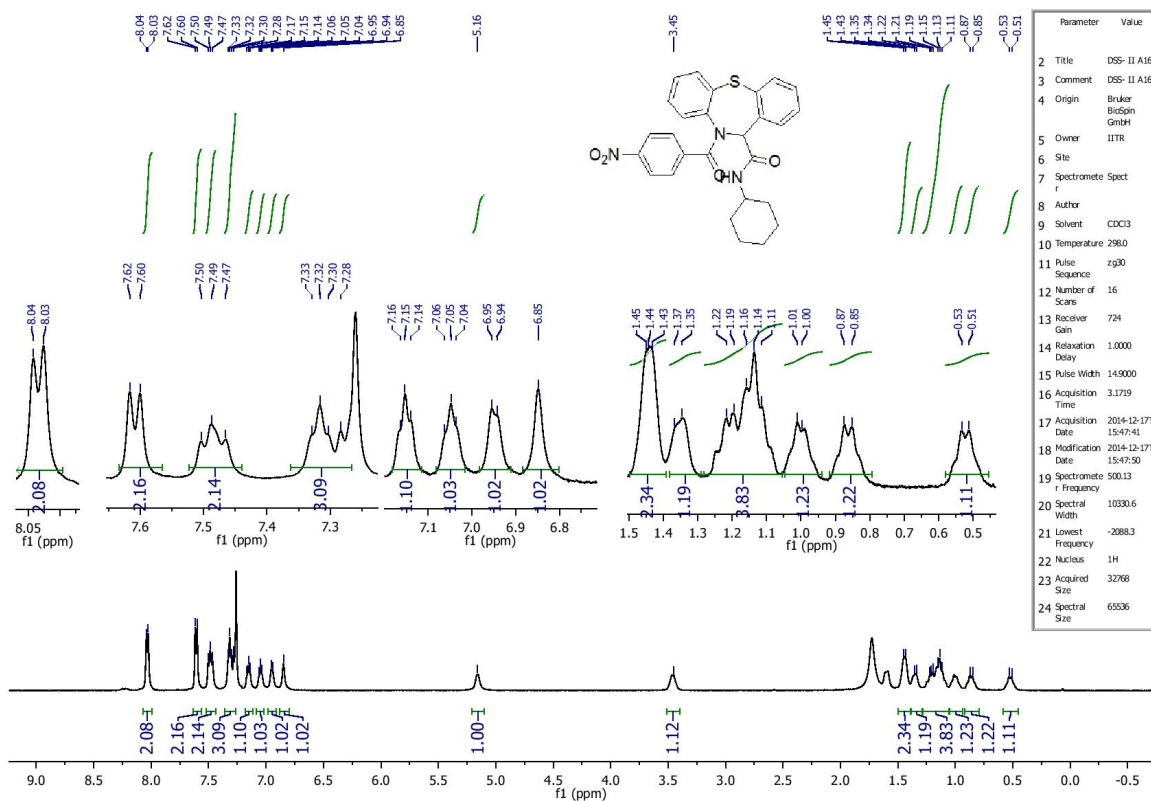


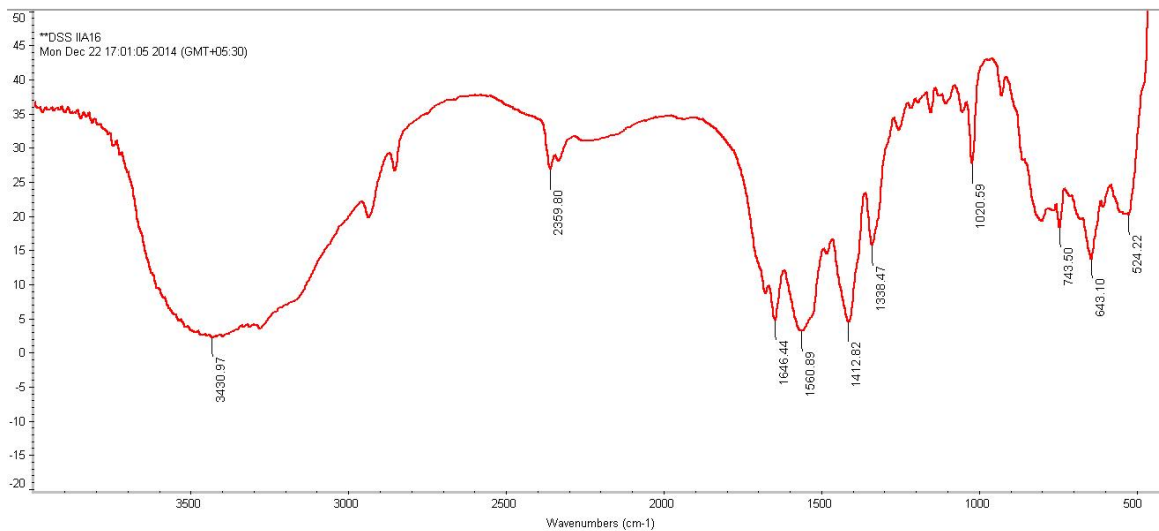
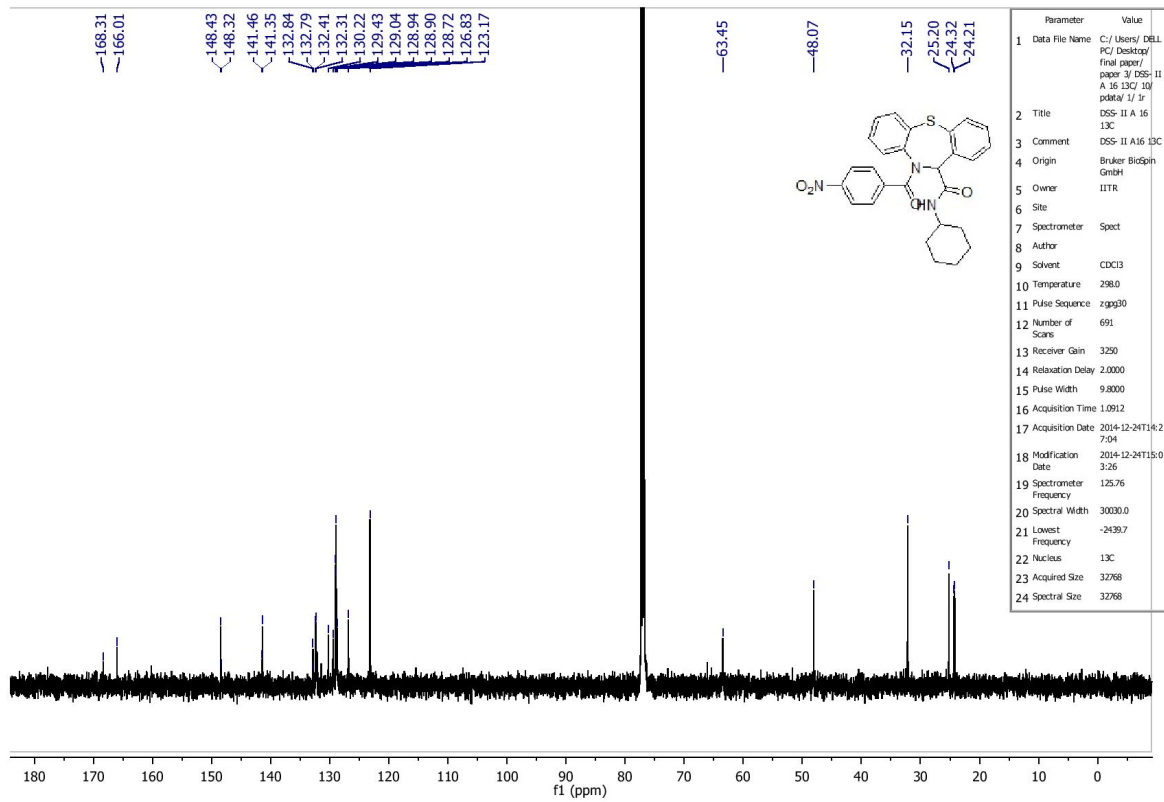
8) *N*-(2,6-dimethylphenyl)-10-(4-nitrobenzoyl)-10,11-dihydrodibenzo[*b,f*][1,4]thiazepine-11-carboxamide **6h**



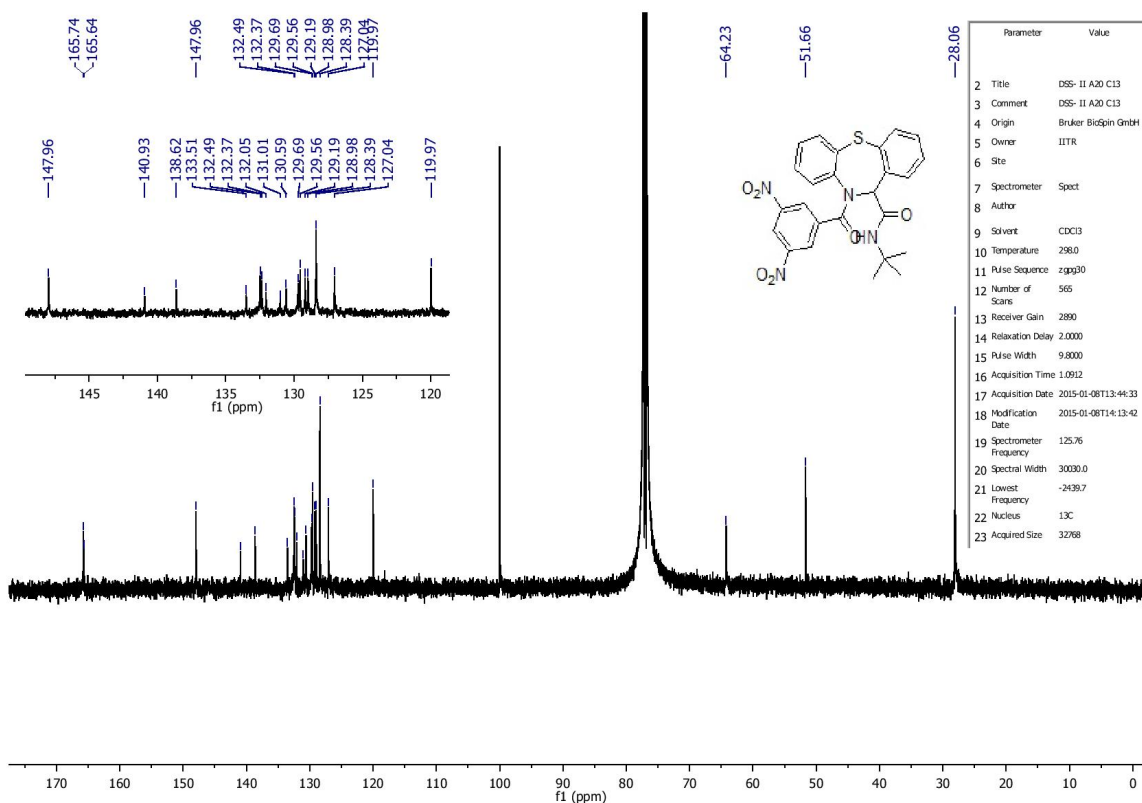
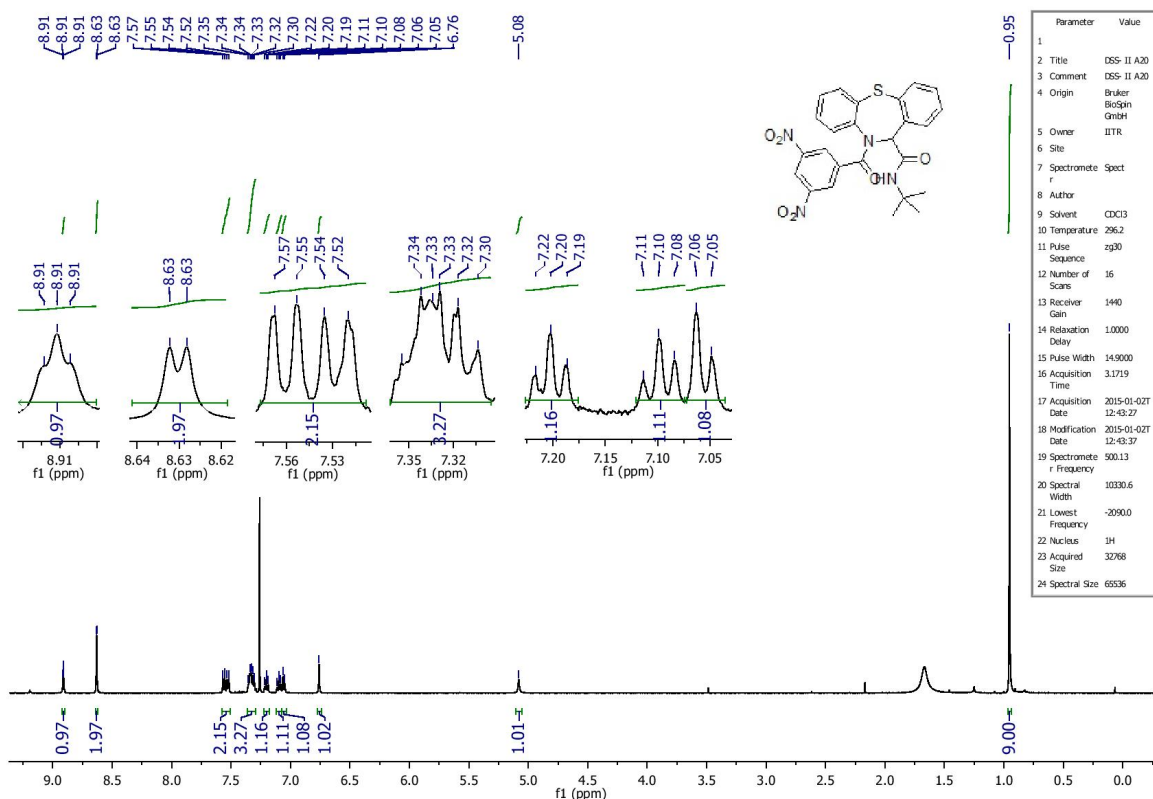


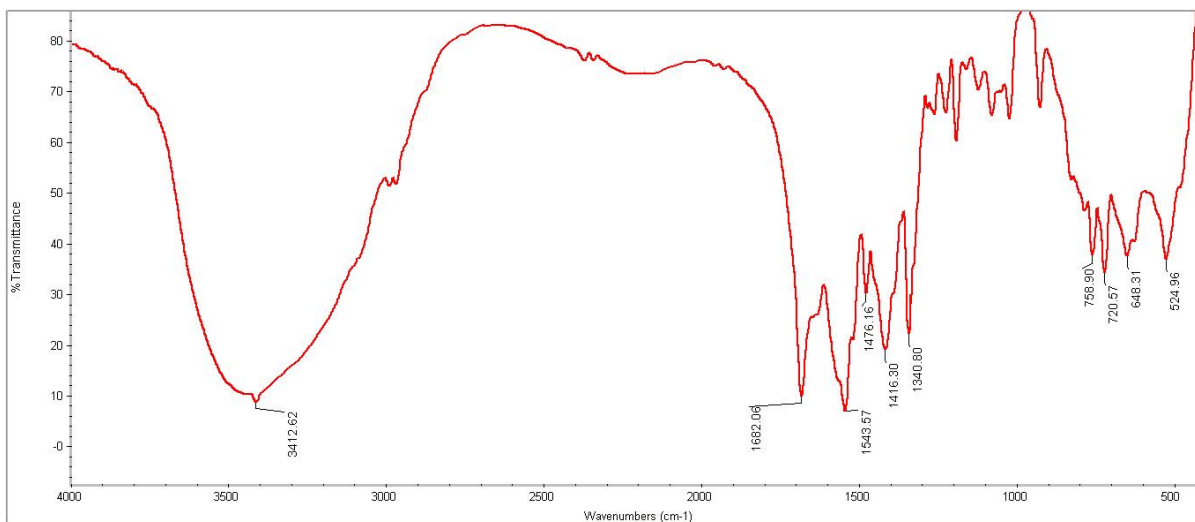
9) *N*-cyclohexyl-10-(4-nitrobenzoyl)-10,11-dihydrodibenzo[*b,f*][1,4]thiazepine-11-carboxamide **6i**



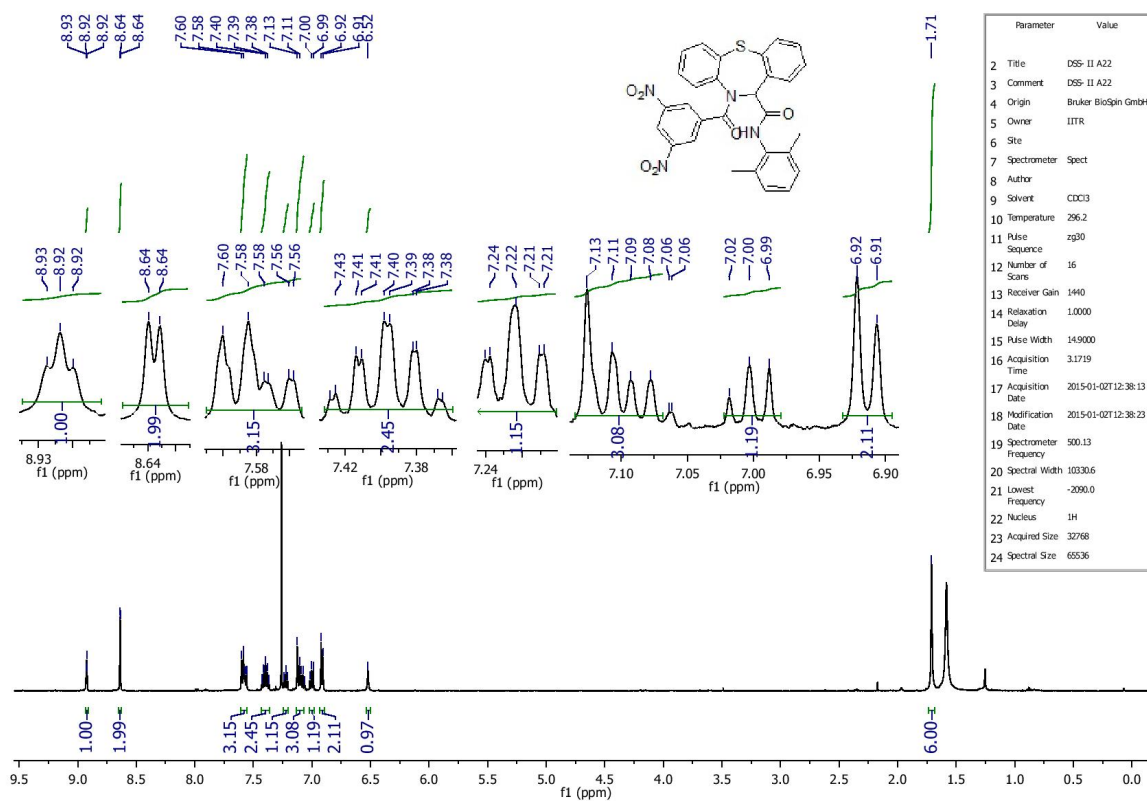


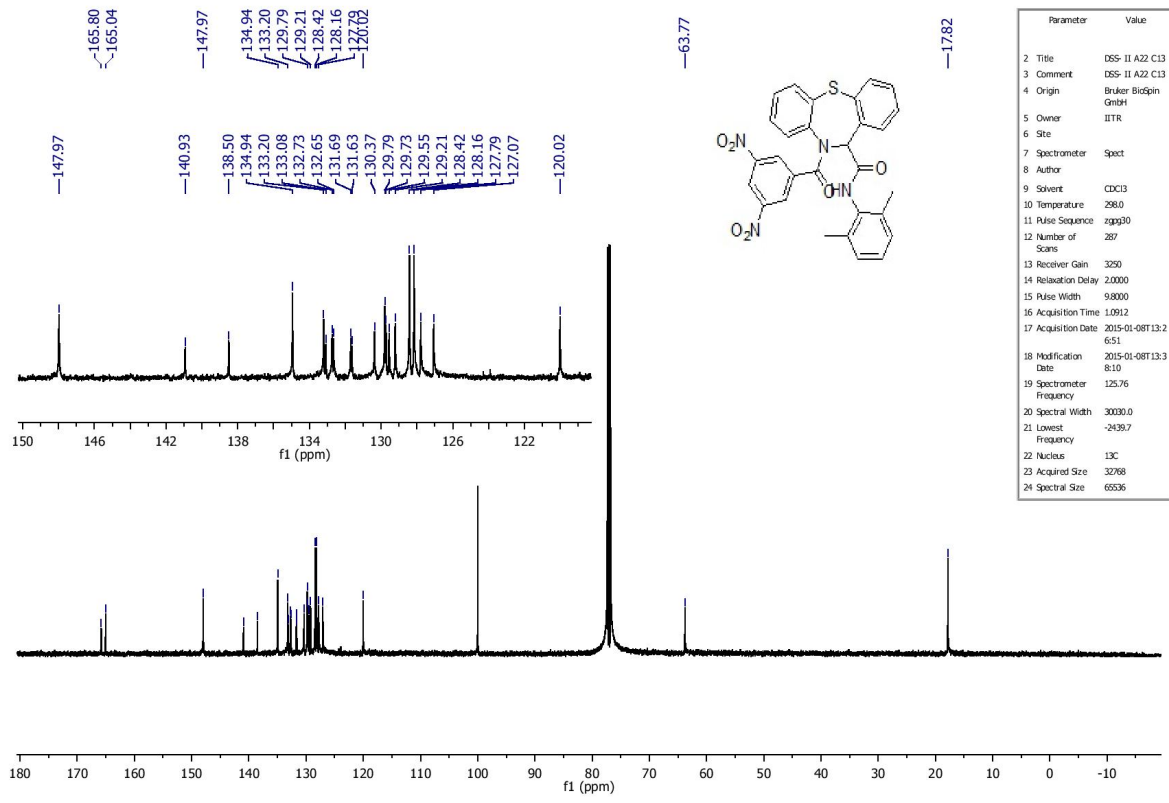
10) *N*-(*tert*-butyl)-10-(3,5-dinitrobenzoyl)-10,11-dihydrodibenzo[*b,f*][1,4]thiazepine-11-carboxamide **6j**



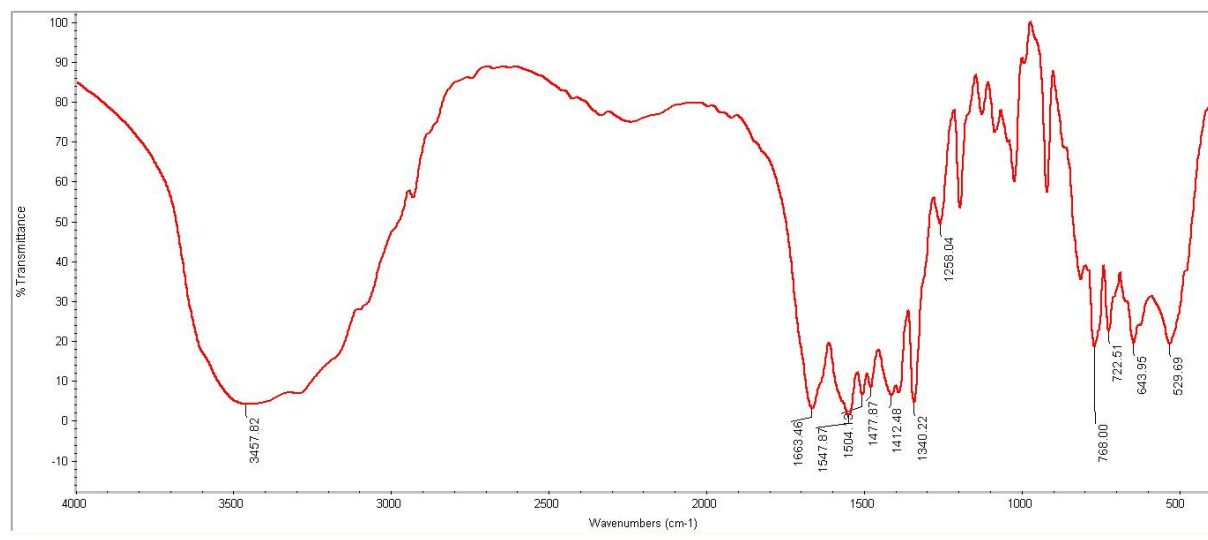


11) *N*-(2,6-dimethylphenyl)-10-(3,5-dinitrobenzoyl)-10,11 dihydrodibenzo[*b,f*] [1,4]thiazepine-11-carboxamide **6k**

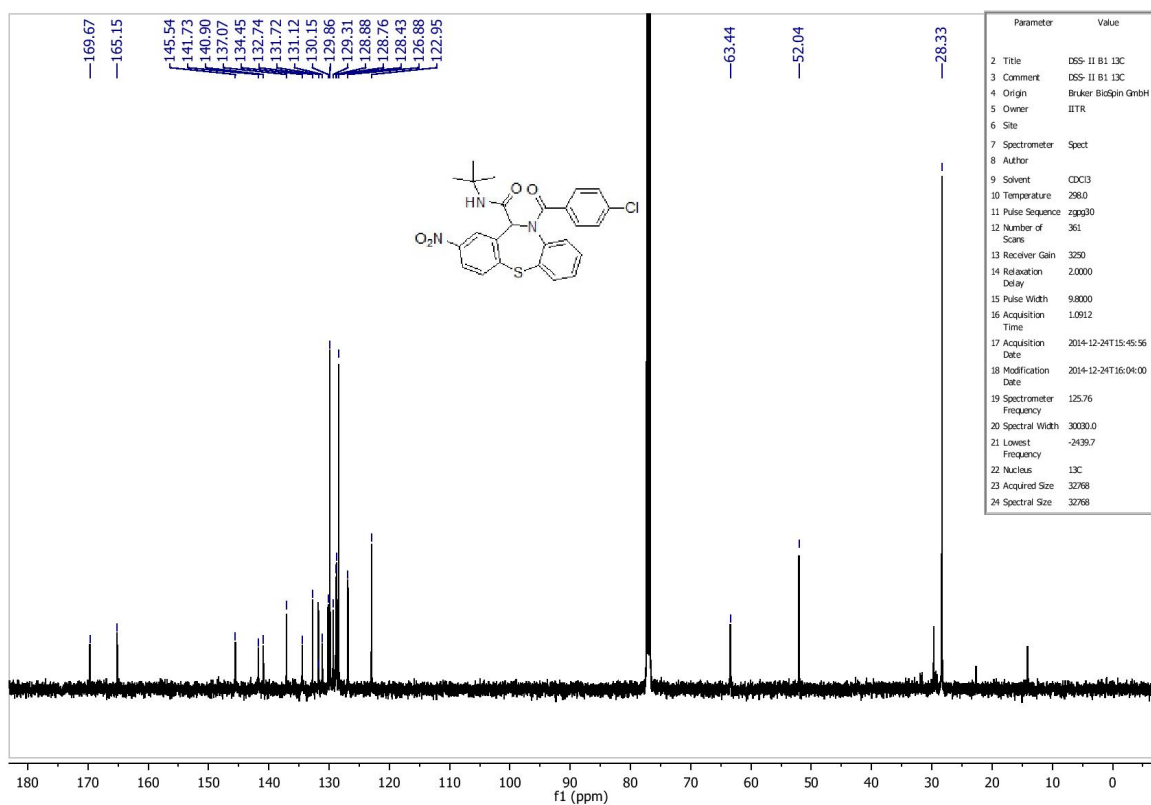
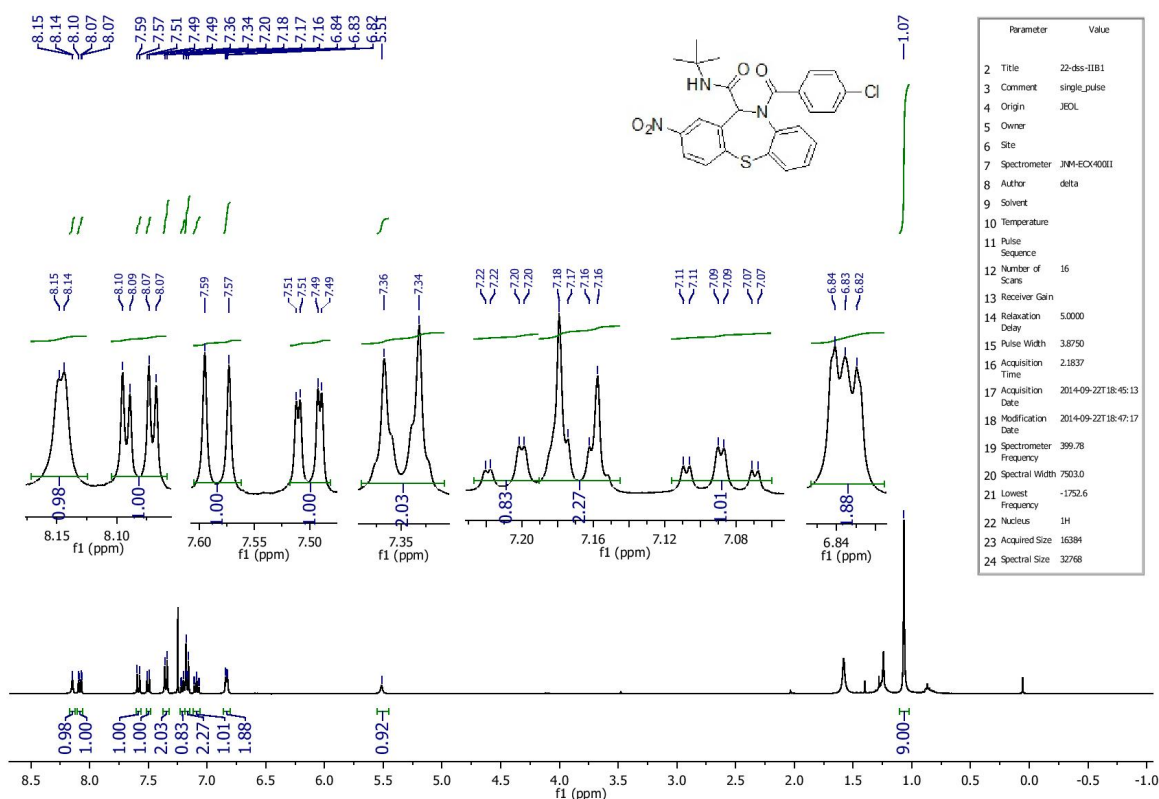


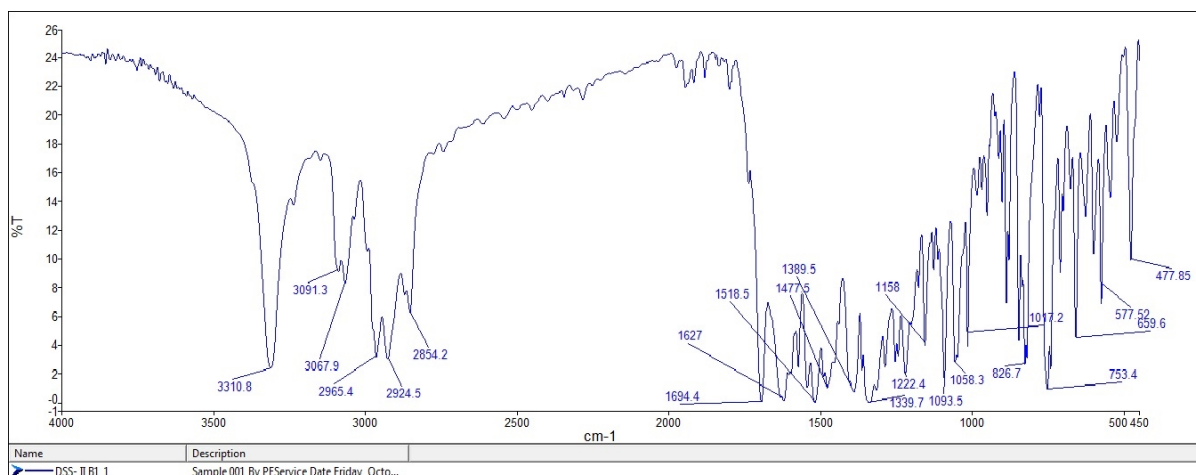


Parameter	Value
2 Title	DSS-11 A22 C13
3 Comment	DSS-11 A22 C13
4 Origin	Bruker BioSpin GmbH
5 Owner	IITR
6 Site	
7 Spectrometer	Spect
8 Author	
9 Solvent	CDCl3
10 Temperature	298.0
11 Pulse Sequence	zgpg30
12 Number of Scans	287
13 Receiver Gain	3250
14 Relaxation Delay	2.0000
15 Pulse Width	9.8000
16 Acquisition Time	1.0912
17 Acquisition Date	2015-01-08T13:26:51
18 Modification Date	2015-01-08T13:38:10
19 Spectrometer Frequency	125.76
20 Spectral Width	30030.0
21 Lowest Frequency	-2439.7
22 Nucleus	13C
23 Acquired Size	32768
24 Spectral Size	6536

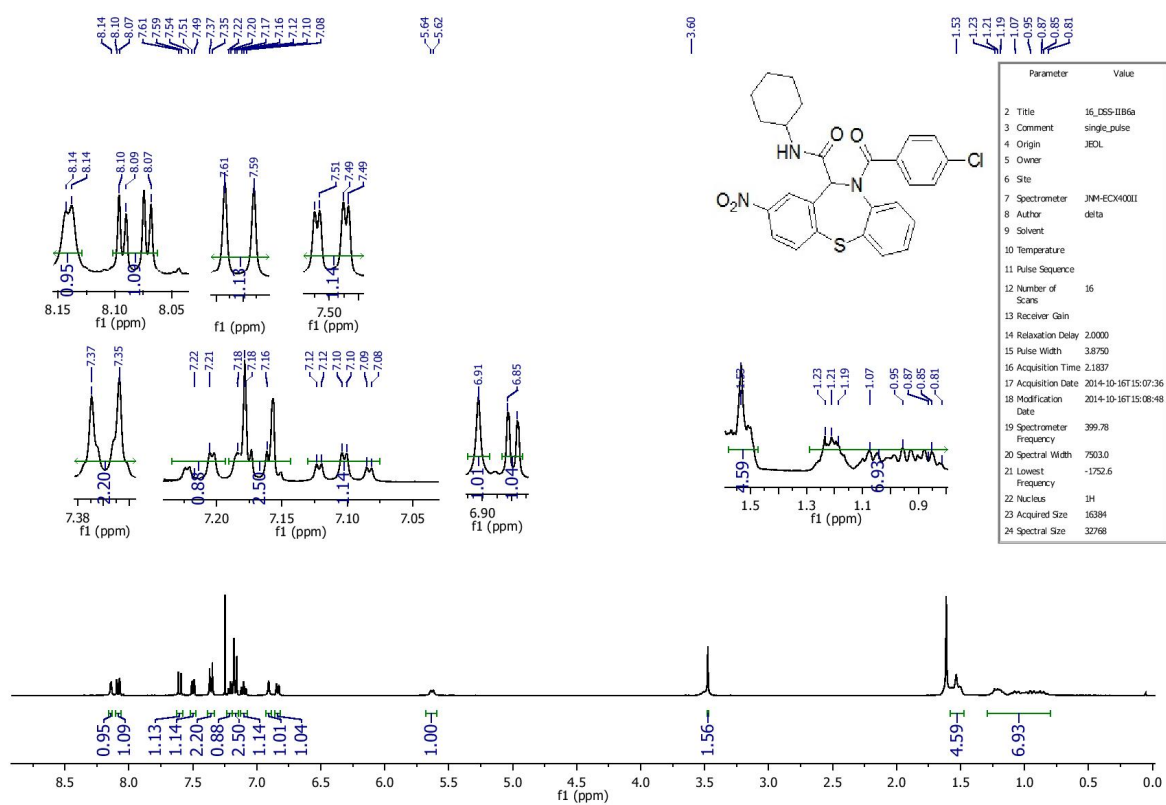


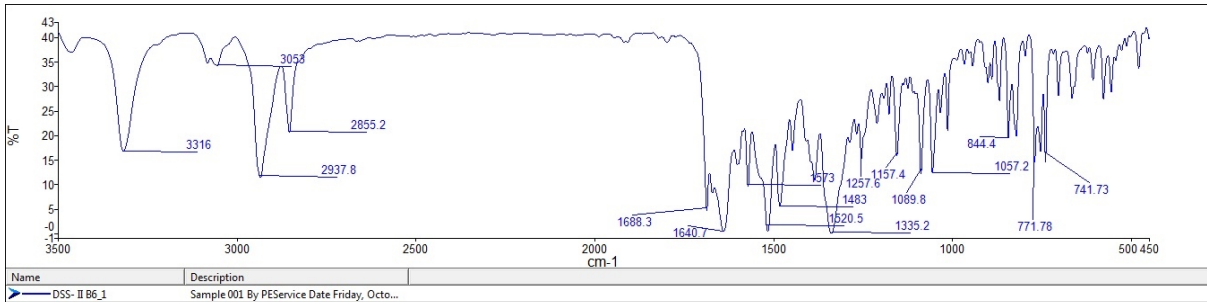
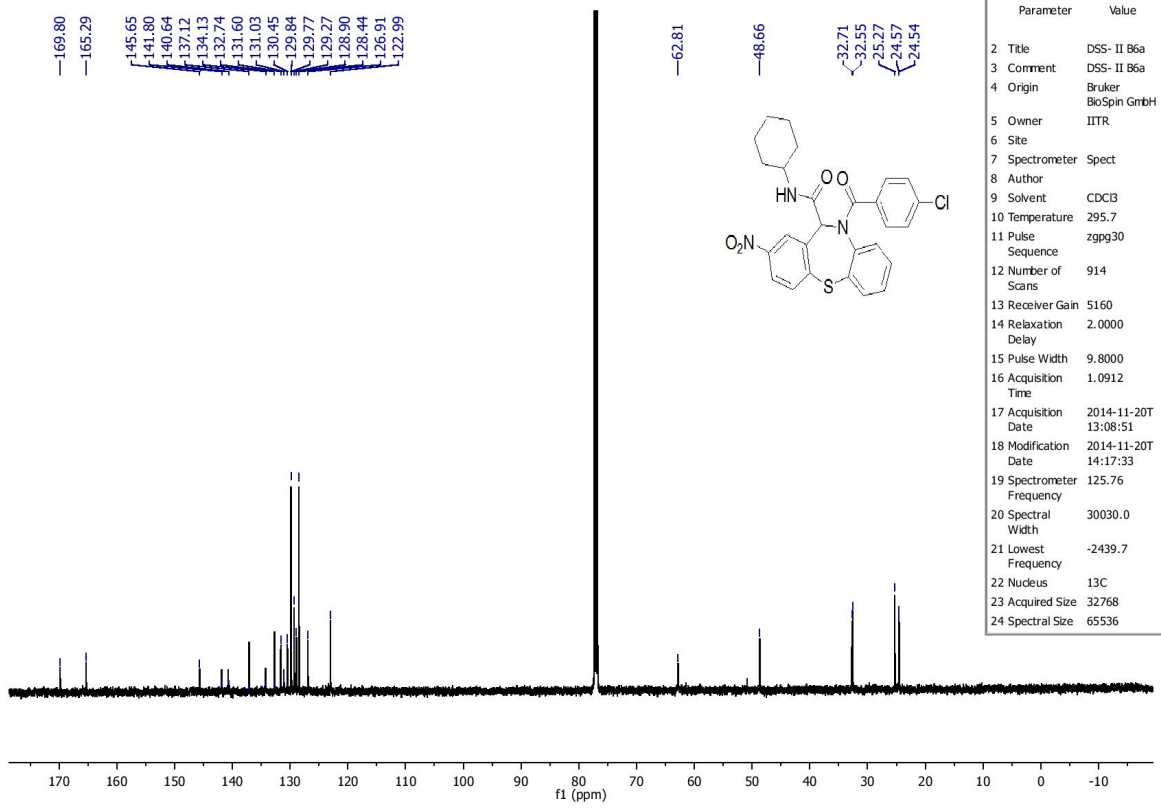
12) N-(tert-Butyl)-10-(4-chlorobenzoyl)-2-nitro-10,11-dihydrodibenzo[b,f][1,4]thiazepine-11-carboxamide 6l.



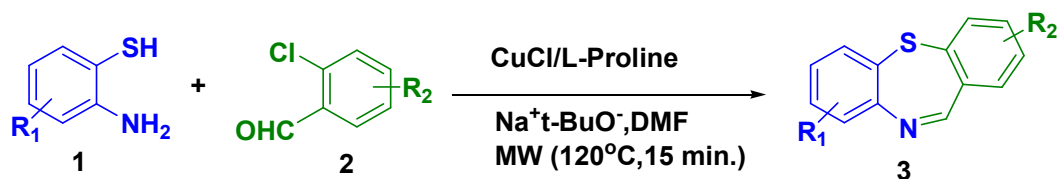


13) 10-(4-Chlorobenzoyl)-N-cyclohexyl-2-nitro-10,11-dihydrodibenzo[b,f][1,4]thiazepine-11-carboxamide 6m.





GC Analysis of the reaction between 1a and 2a



The above reaction was monitored by GC-MS. After 2 min the GC Chromatogram indicated the formation of the Schiff base **B** with mass 245 (retention time: 18.91min.) in maximum percentage.

