

Copies of  $^1\text{H}$  and  $^{13}\text{C}$  Spectra

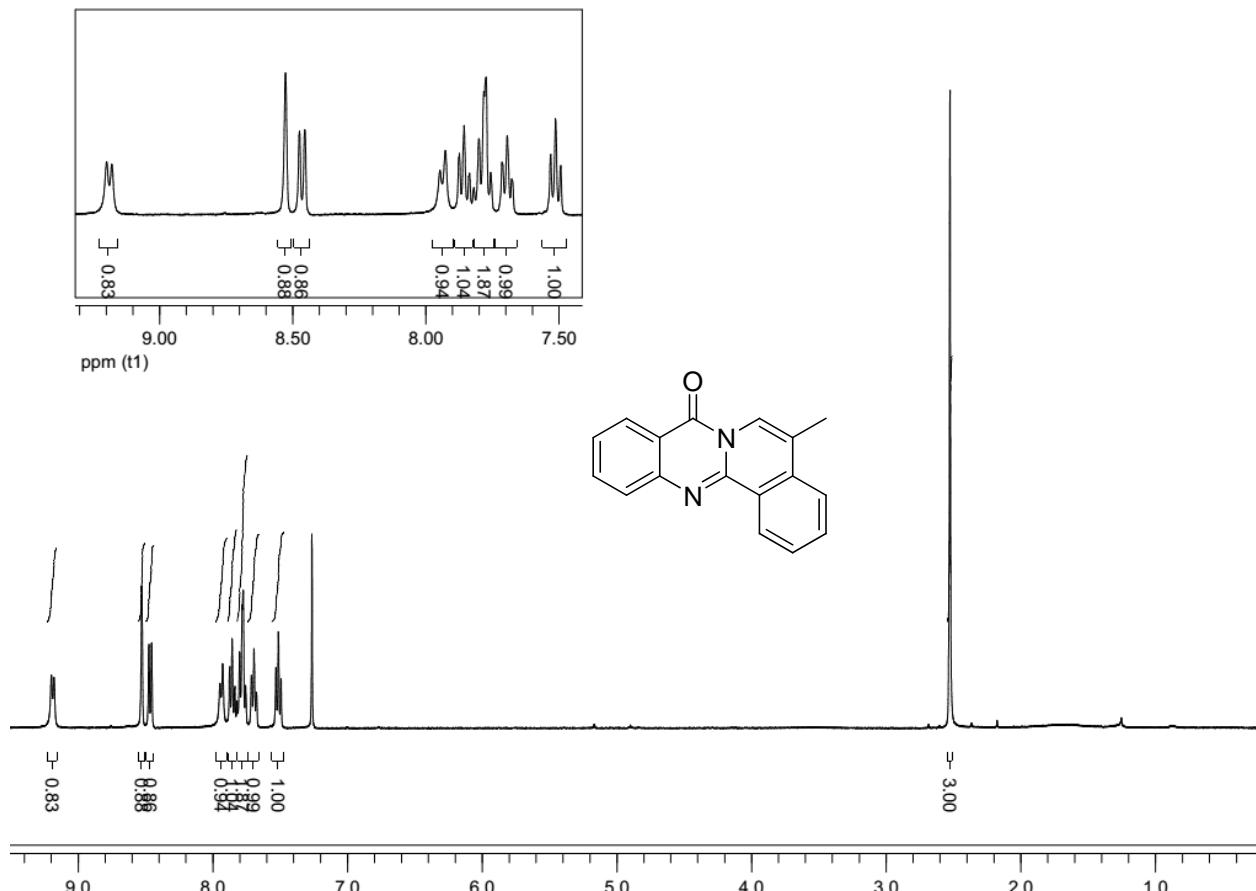


Fig. 1:  $^1\text{H}$  NMR spectra of compound **4a** ( $\text{CDCl}_3$ , 400 MHz)

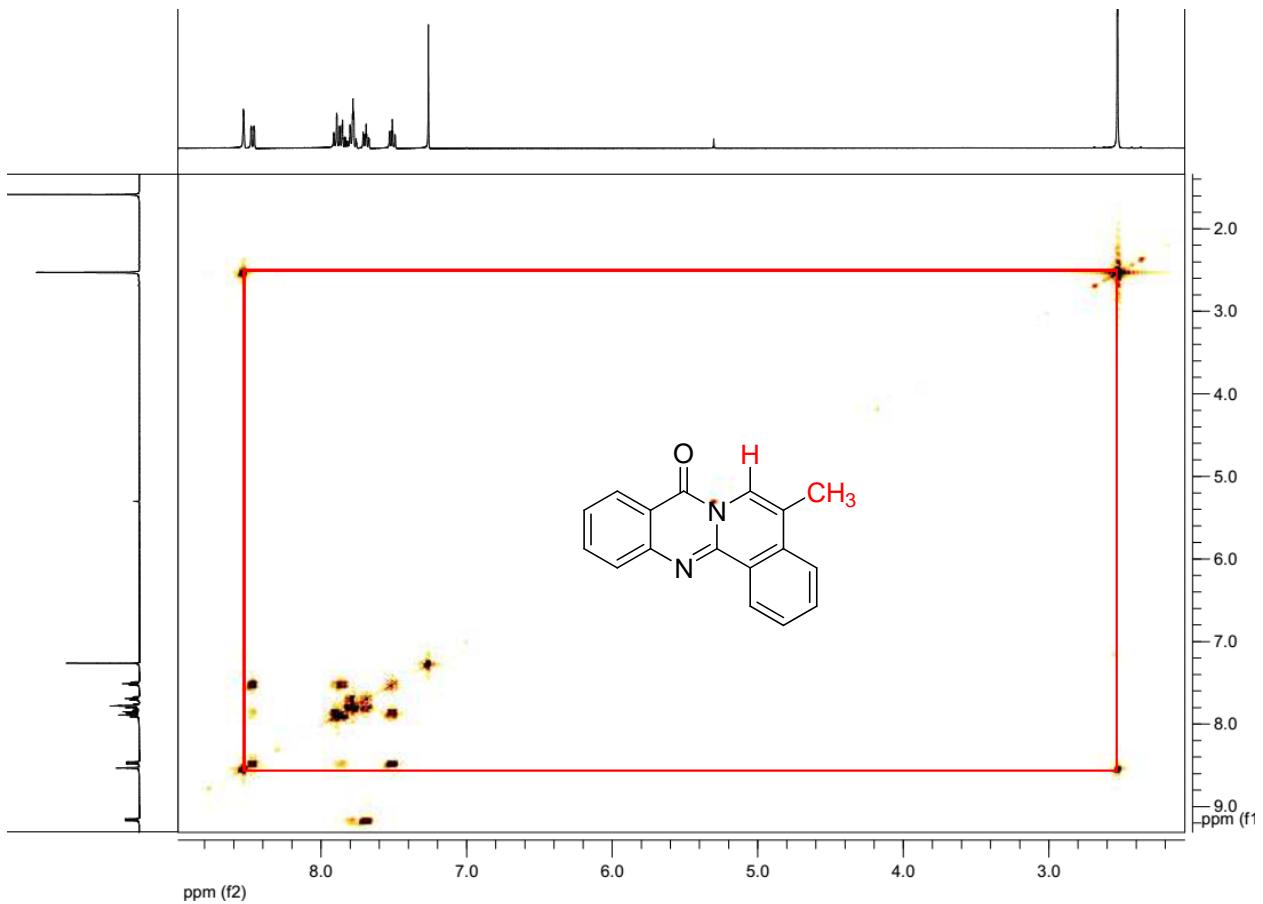


Fig. 2:  $^1\text{H}$ - $^1\text{H}$  COSY spectra of compound **4a** ( $\text{CDCl}_3$ , 400 MHz)

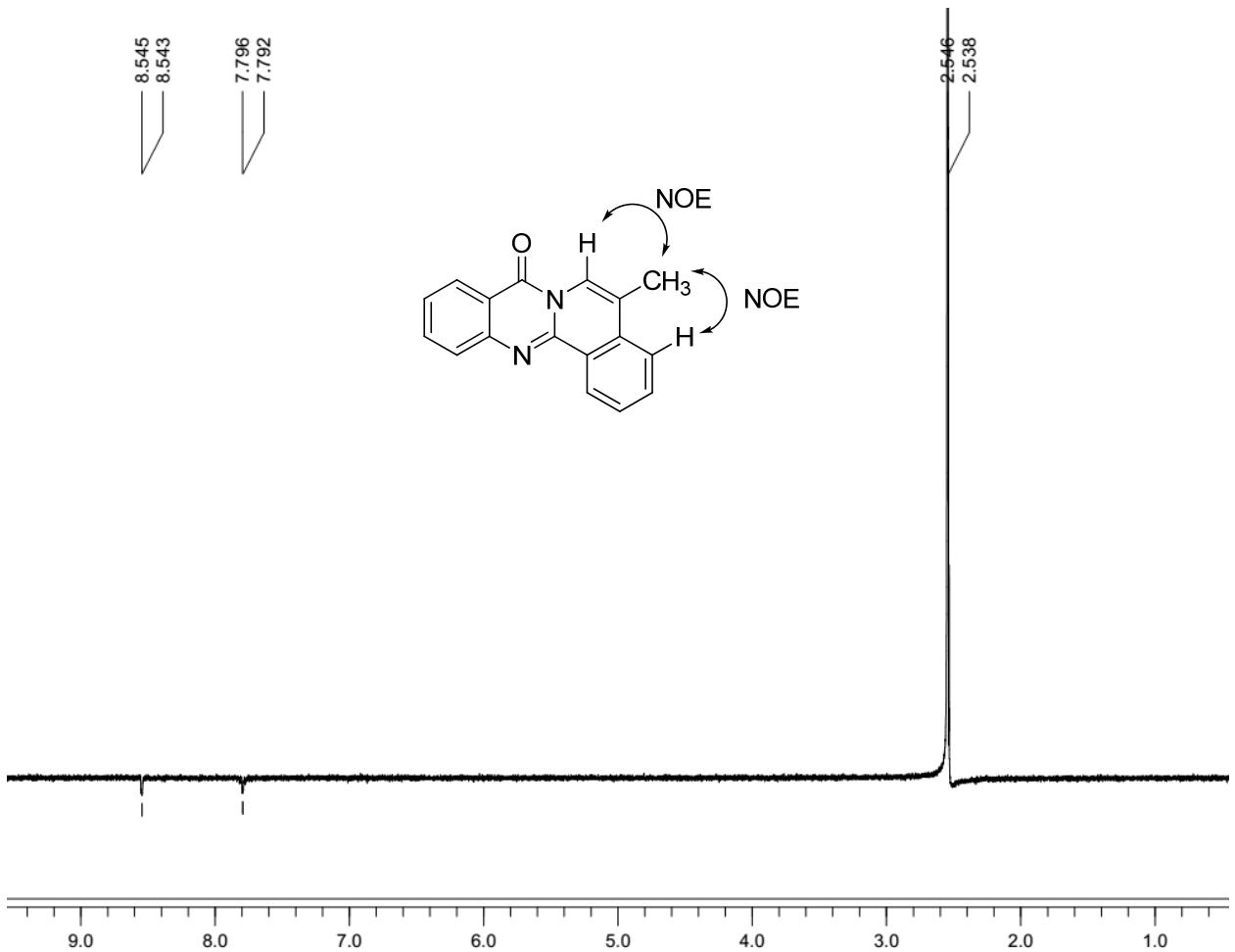


Fig. 3: 1D-NOE spectra of compound **4a** ( $\text{CDCl}_3$ , 400 MHz)

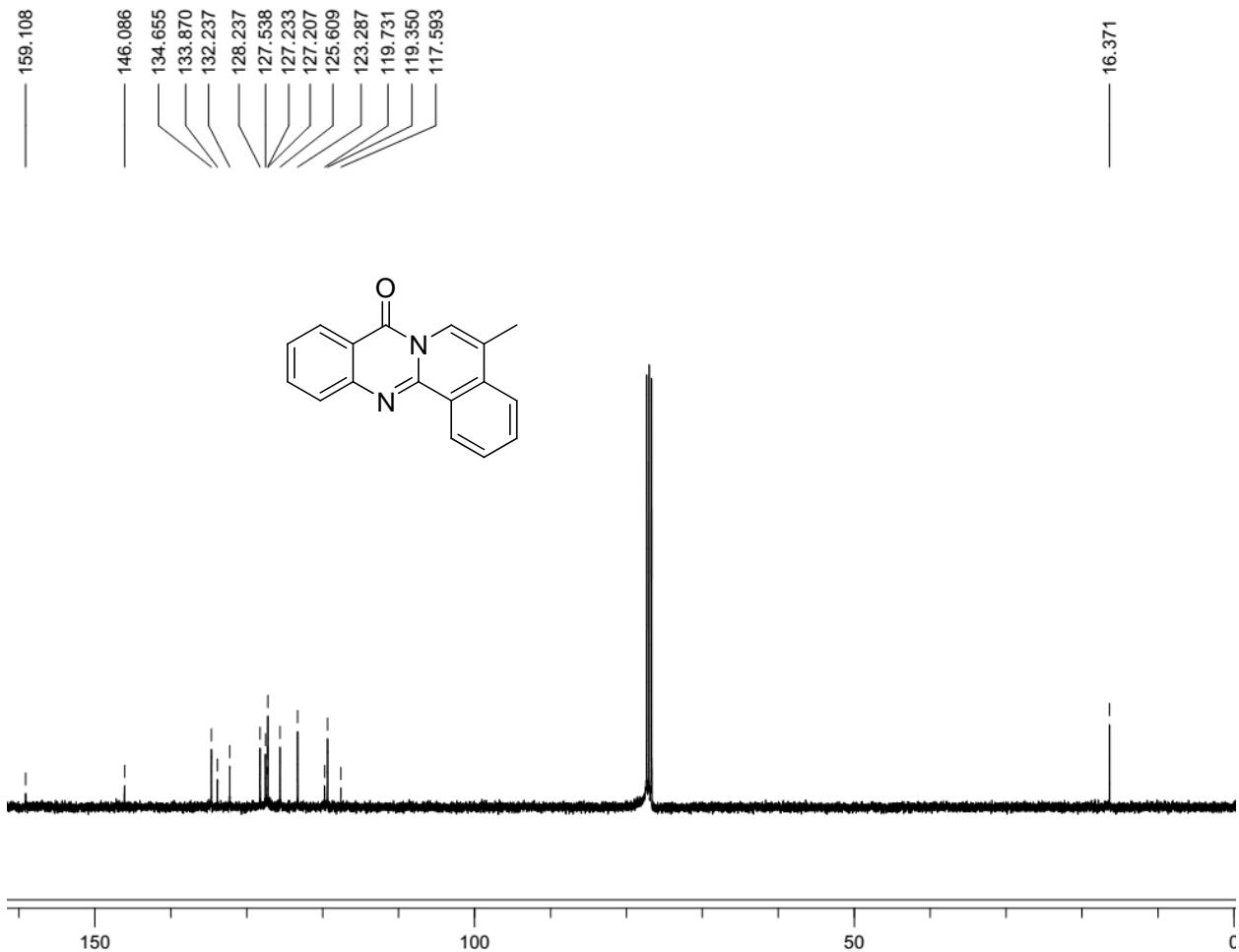
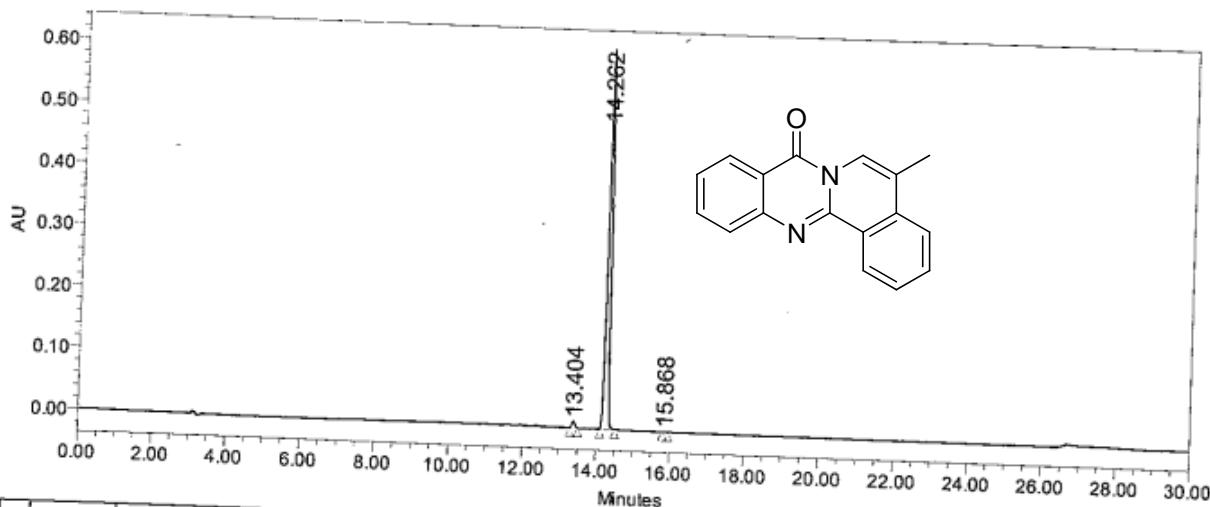


Fig. 4:  $^{13}\text{C}$  NMR spectra of compound **4a** ( $\text{CDCl}_3$ , 100 MHz)

### SAMPLE INFORMATION

Sample Name:	ILS/ARJ/5/1	Sample Set Name:	240214
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	27	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	280.0nm
Injection Volume:	10.00 ul	Proc. Chnl. Descr.:	PDA 280.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/24/2014 12:13:33 PM IST		
Date Processed:	2/24/2014 12:49:24 PM IST		

Column: X TERRA RP-18 250\*4.6mm 5μm  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	13.404	59643	1.64	10771
2	14.262	3579738	98.20	613639
3	15.868	6021	0.17	1270

Analysed By *NV* 24/02/14

Fig. 5: HPLC of compound 4a

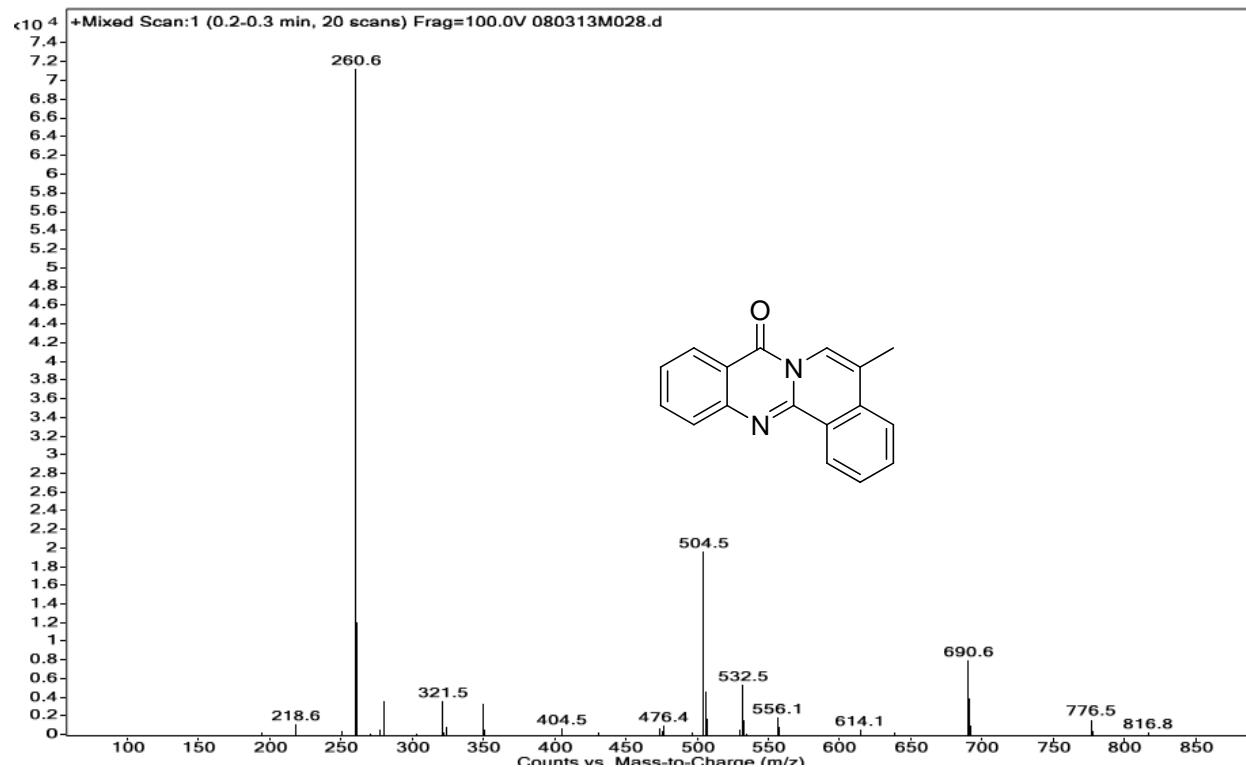


Fig. 6: Mass of compound **4a**

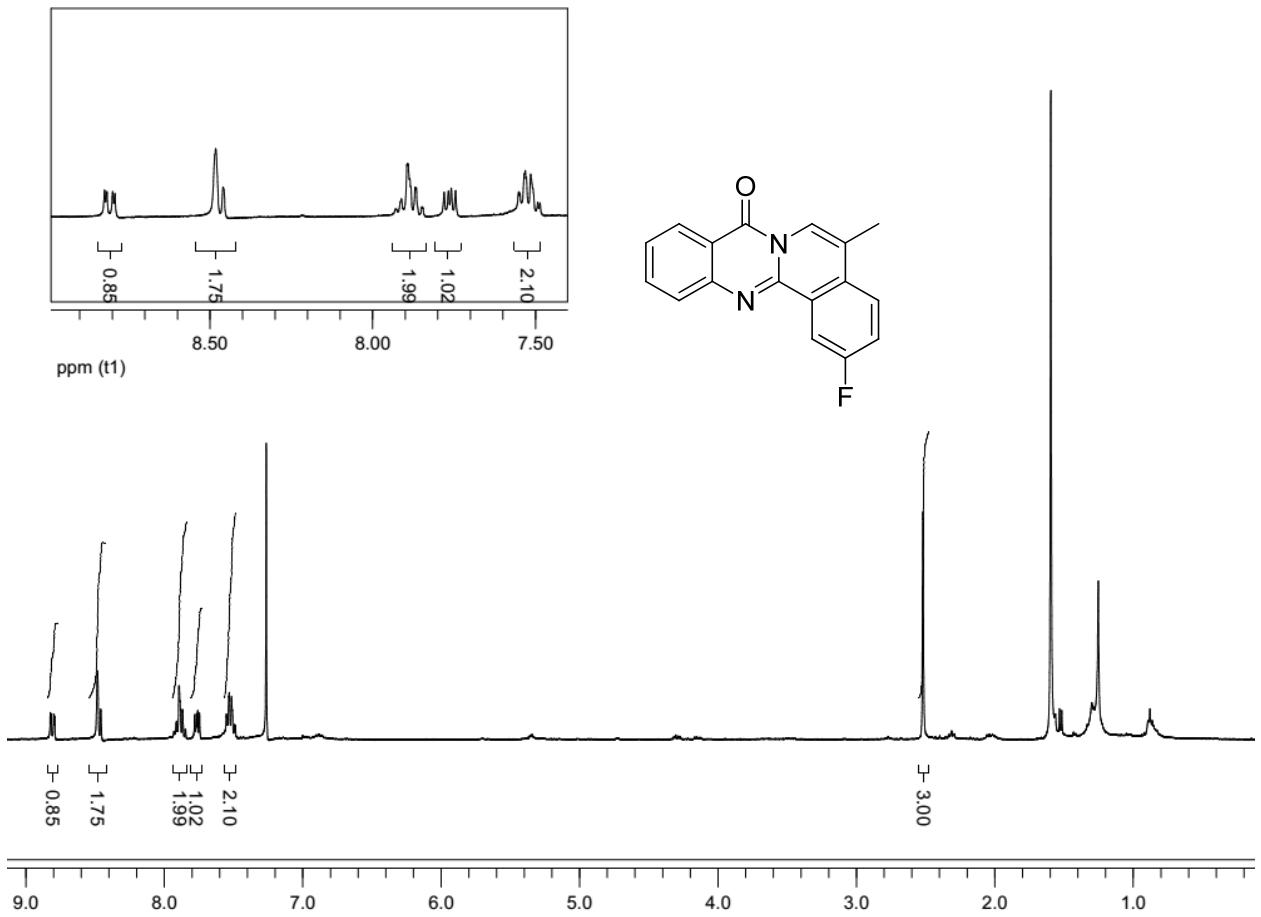


Fig. 7:  $^1\text{H}$  NMR spectra of compound **4b** ( $\text{CDCl}_3$ , 400 MHz)

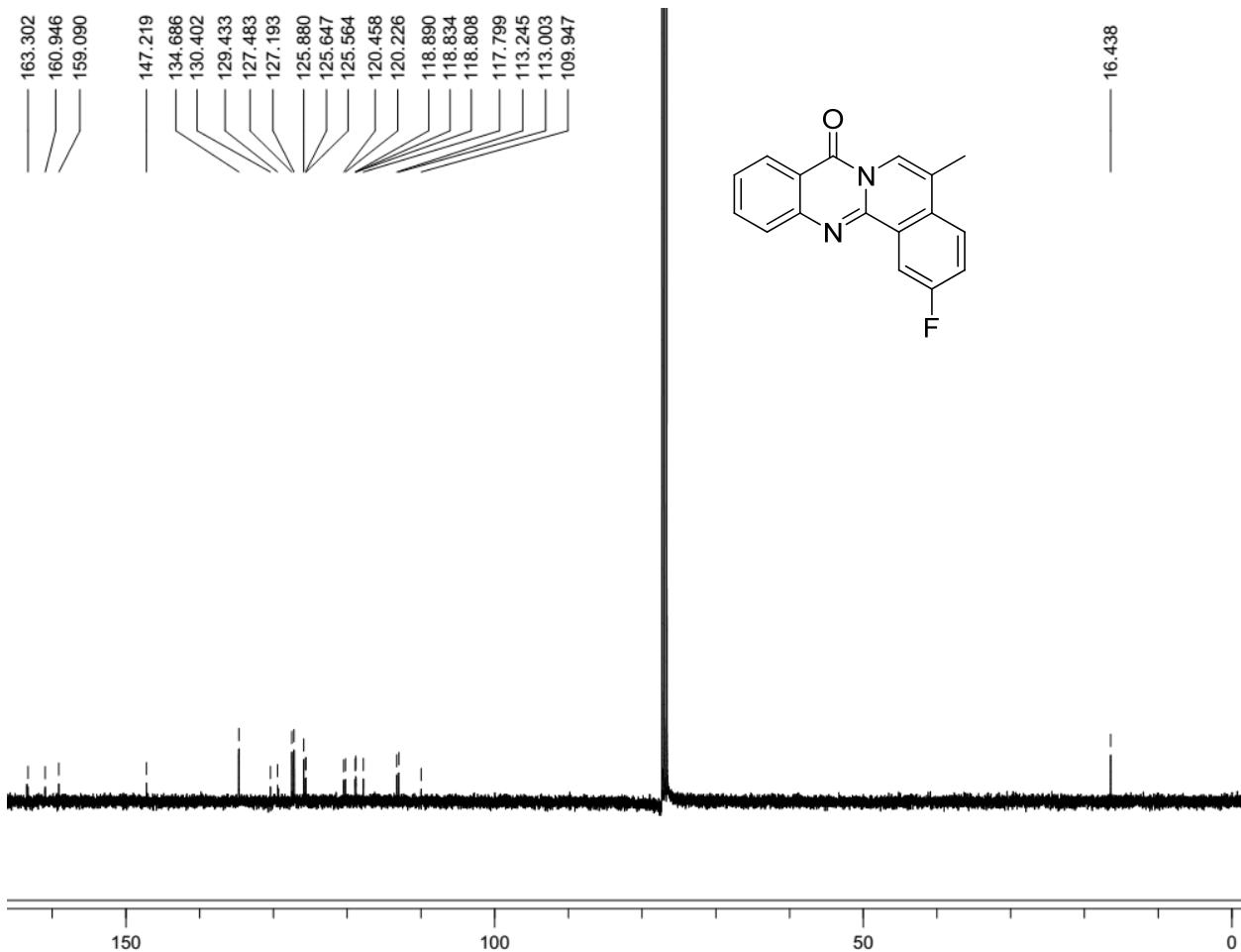
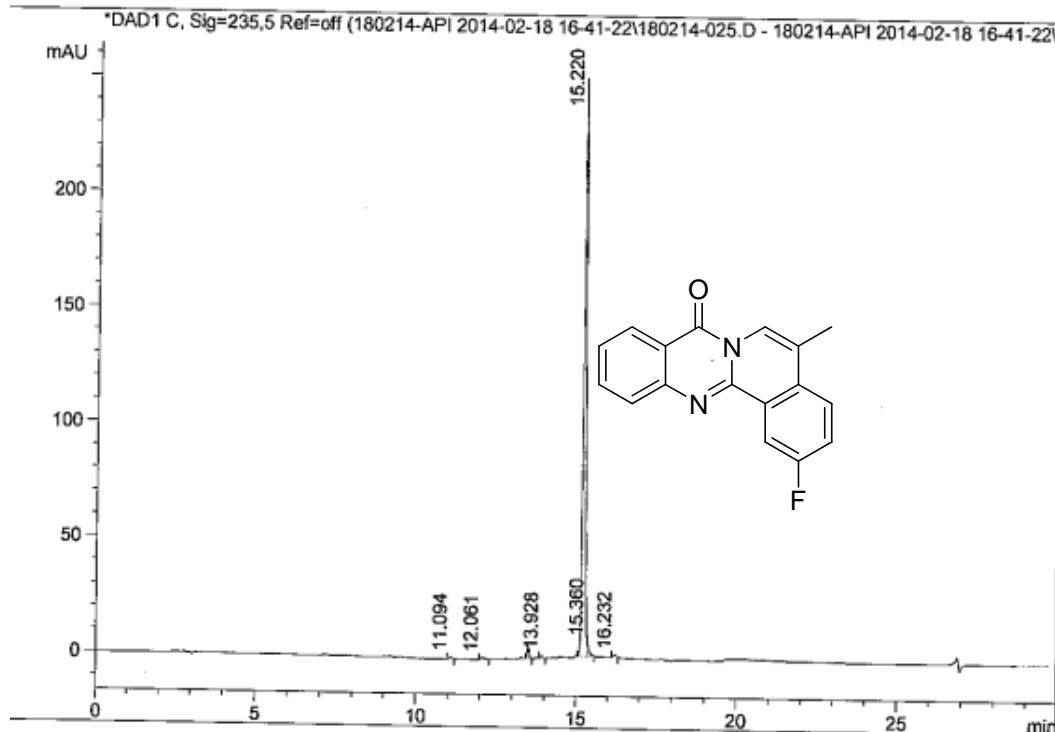


Fig. 8:  $^{13}\text{C}$  NMR spectra of compound **4b** ( $\text{CDCl}_3$ , 100 MHz)

Injection Date : Wed, 19. Feb. 2014  
 Sample Name : ILS-ARJ-5-2  
 Acq Operator : RADHA  
 Acq. Method : D:\chem32\1\DATA\180214-API 2014-02-18 16-41-22\API ->  
 Analysis Method : D:\CHEM32\_002\1\METHODS\API MKT.M  
 Method Info : Column : X-Terra RP18 250\*4.6mm, 5µm  
 Mobile phase: A)0.1%TFA in Water B) ACN (gradient )  
 T/B%:0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow:1.0 ml/min, Diluent: MEOH



Peak	RT [min]	Area	Area %
1	11.094	5.254	0.370
2	12.061	6.277	0.442
3	13.513	23.071	1.625
4	13.928	6.979	0.492
5	15.220	1350.562	95.117
6	15.360	21.910	1.543
7	16.232	5.846	0.412

M. S. Vir

Fig. 9: HPLC of compound 4b

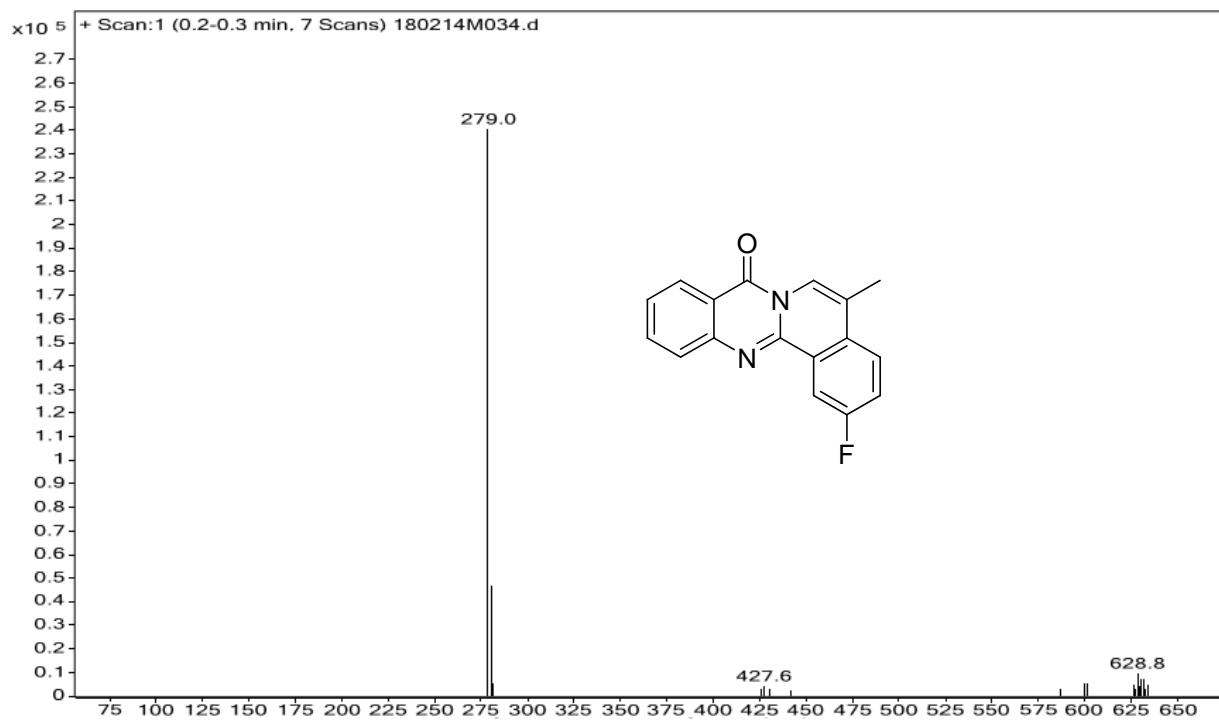


Fig. 10: Mass of compound **4b**

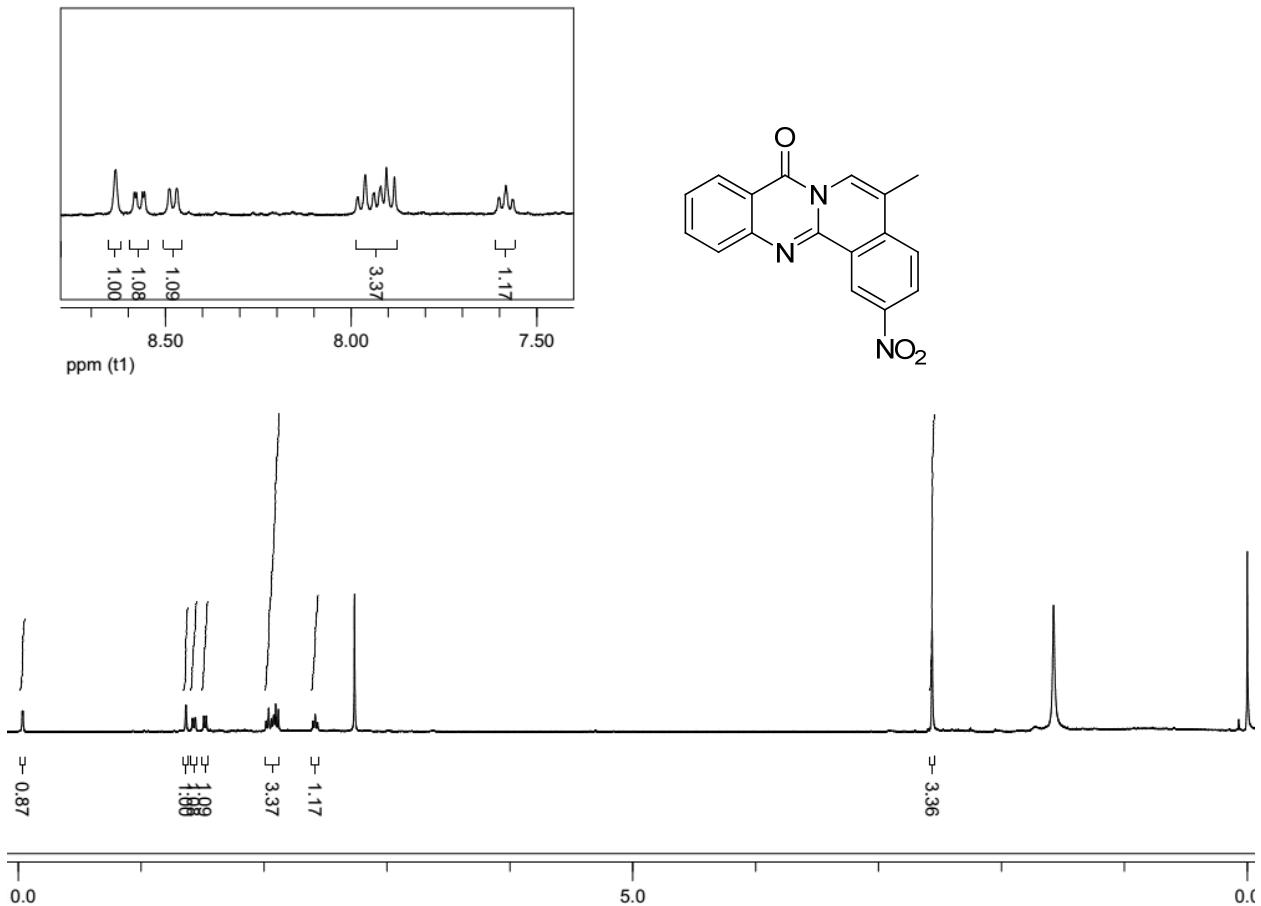


Fig. 11: <sup>1</sup>H NMR spectra of compound **4c** ( $\text{CDCl}_3$ , 400 MHz)

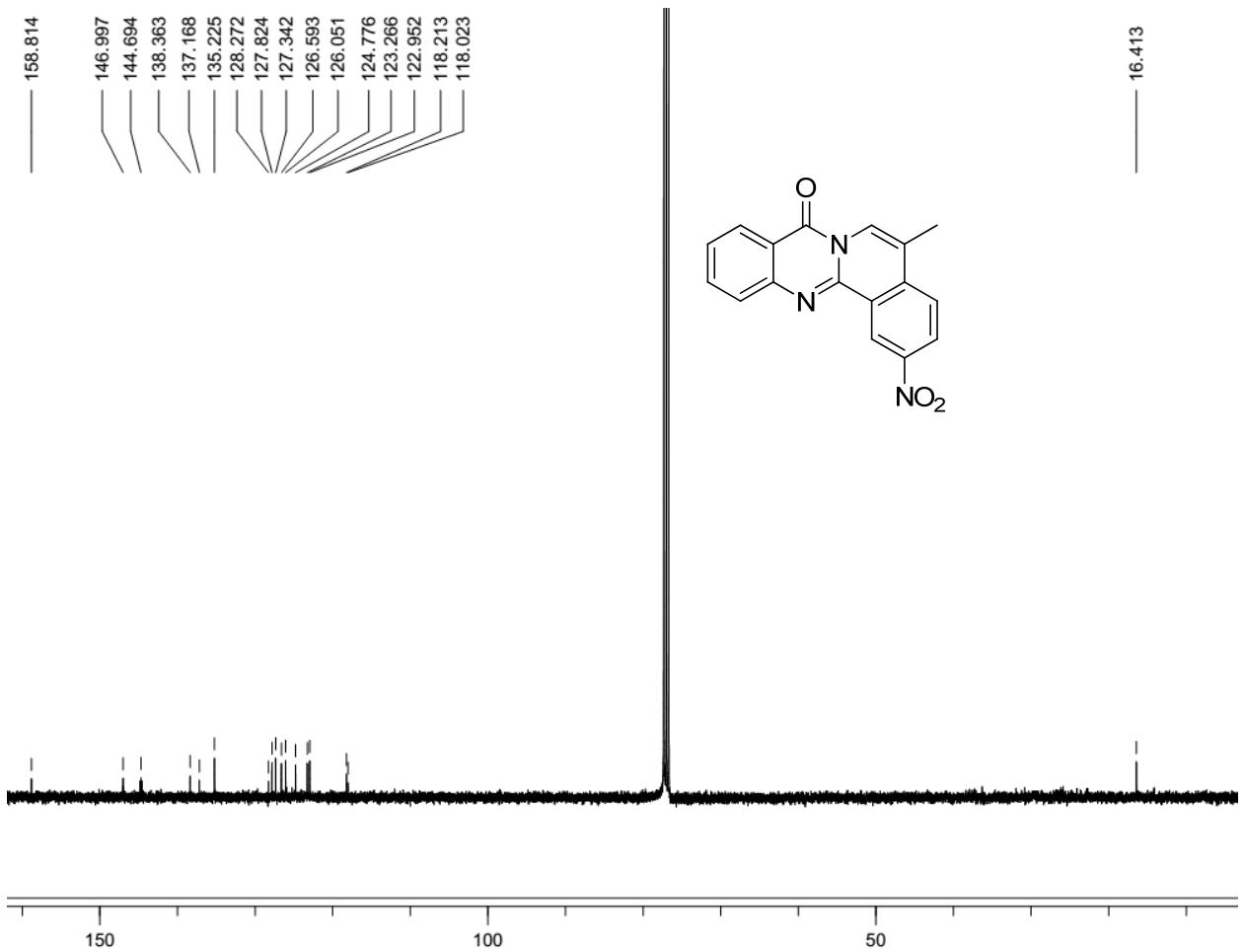
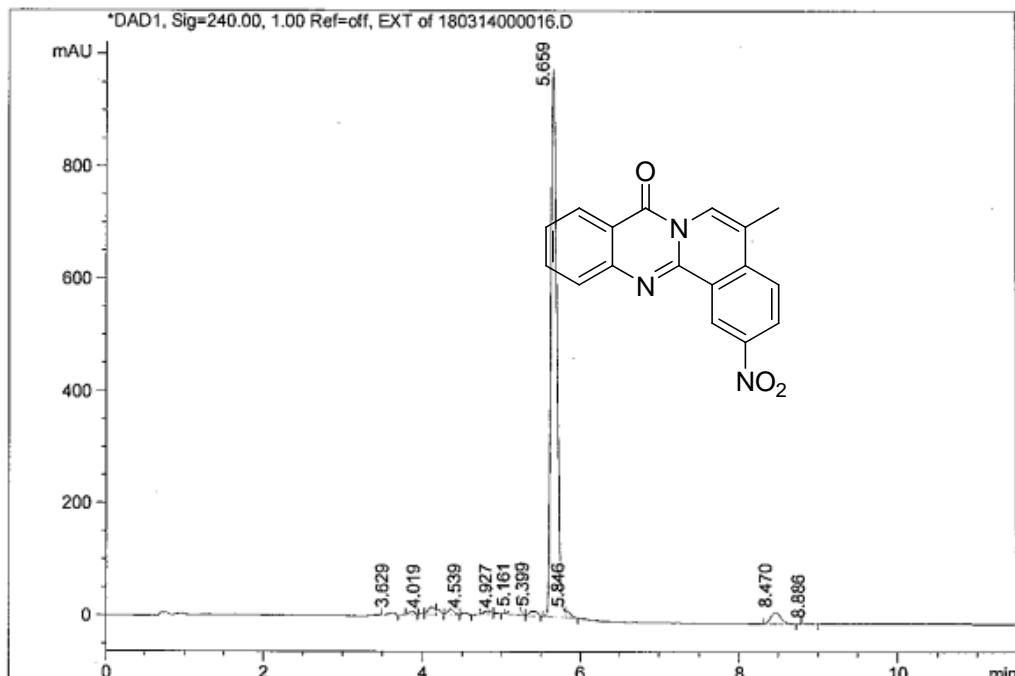


Fig. 12:  $^{13}\text{C}$  NMR spectra of compound **4c** ( $\text{CDCl}_3$ , 100 MHz)

Seq Line : 0  
 Injection Date : Wed, 19. Mar. 2014  
 Location : Vial 24  
 Sample Name : ILS/ARJ/5/15  
 Inj. No. : 0  
 Acq Operator : RADHA  
 Inj. Vol. : 0  $\mu$ l  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A20B80.M  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18-A70B30G.M  
 Method Info : Column :Symmetry C-18 75\*4.6mm, 3.5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water,B) ACN (gradient)  
 T/B%:0/20,1/20,4/98,10/98,10.5/20,12/20.  
 FLOW:1.0ml/min Dil: ACN:Water(80:20)



=====  
 Signal 1: DAD1, Sig=240.00, 1.00 Ref=off, EXT

Peak#	RT [min]	Area	Area %	Peak#	RT [min]	Area	Area %
1	3.629	25.202	0.416	12	5.659	5987.527	91.288
2	3.873	23.804	0.393	13	5.846	45.037	0.743
3	4.019	4.807	0.079	14	8.470	167.897	2.770
4	4.119	71.525	1.180	15	8.886	4.301	0.071
5	4.192	25.839	0.426				
6	4.361	41.430	0.684				
7	4.539	12.341	0.204				
8	4.825	29.262	0.483				
9	4.927	6.569	0.108				
10	5.161	24.979	0.354				
11	5.399	48.567	0.801				

Fig. 13: HPLC of compound 4c

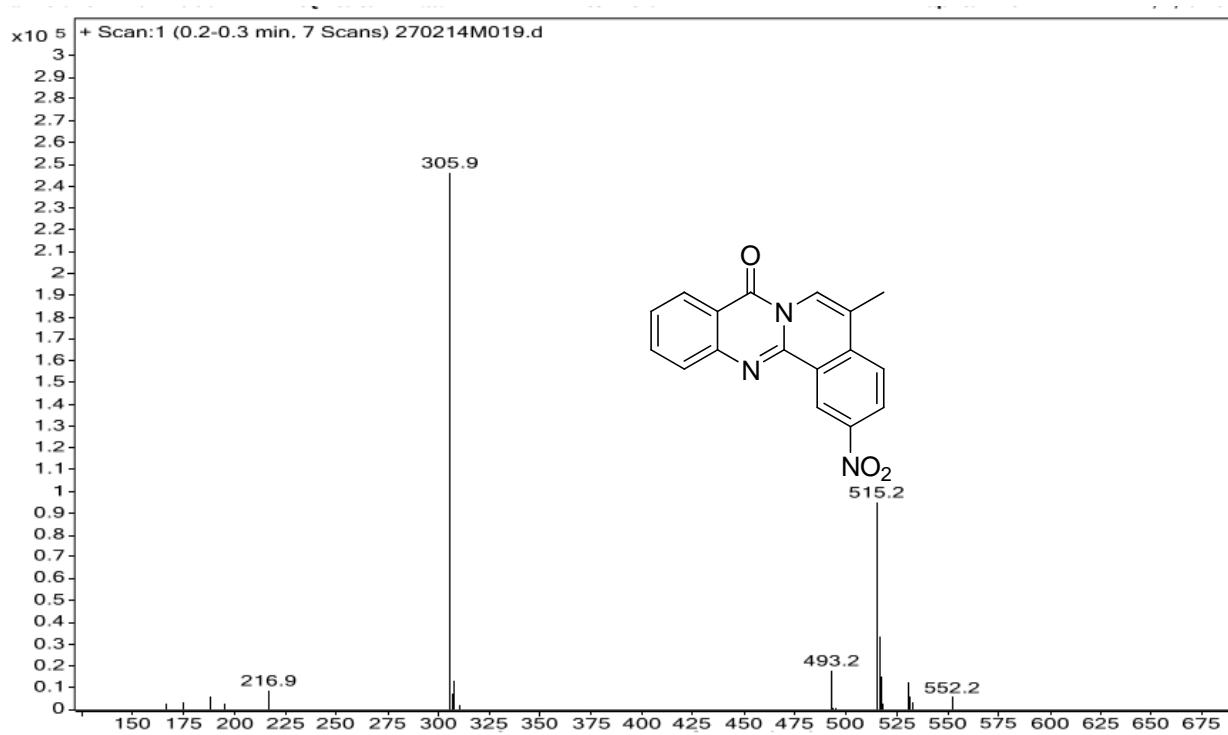


Fig. 14: Mass of compound 4c

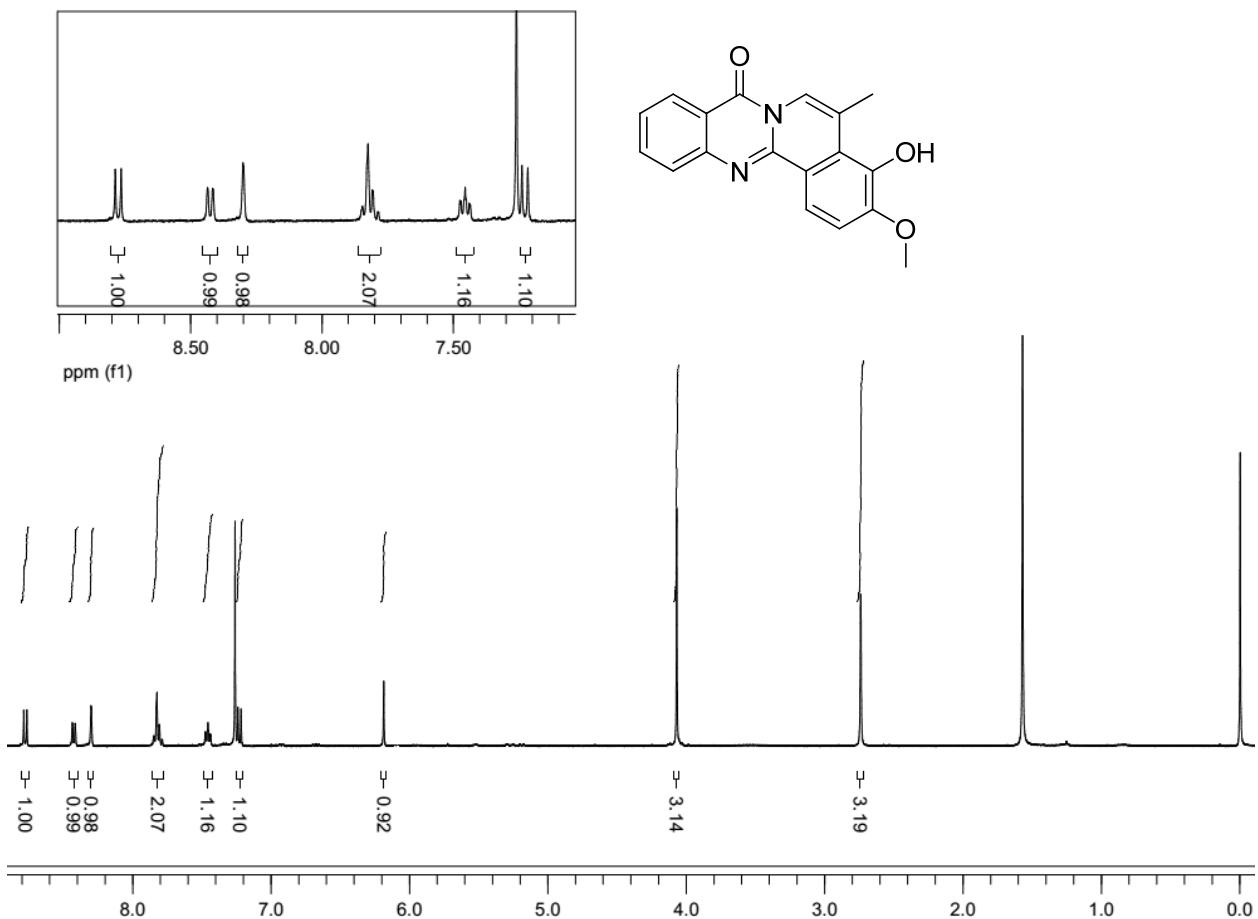


Fig. 15: <sup>1</sup>H NMR spectra of compound **4d** ( $\text{CDCl}_3$ , 400 MHz)

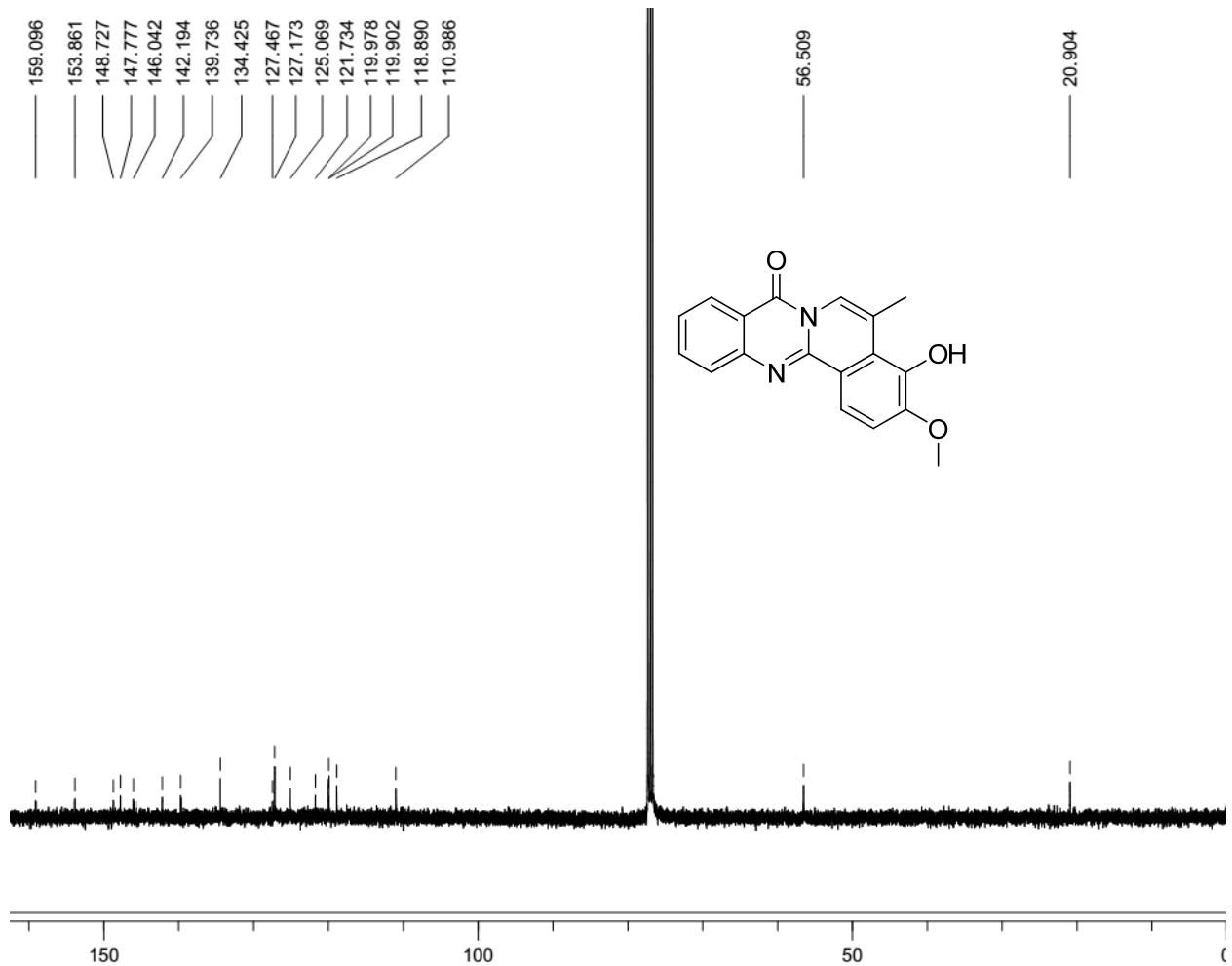
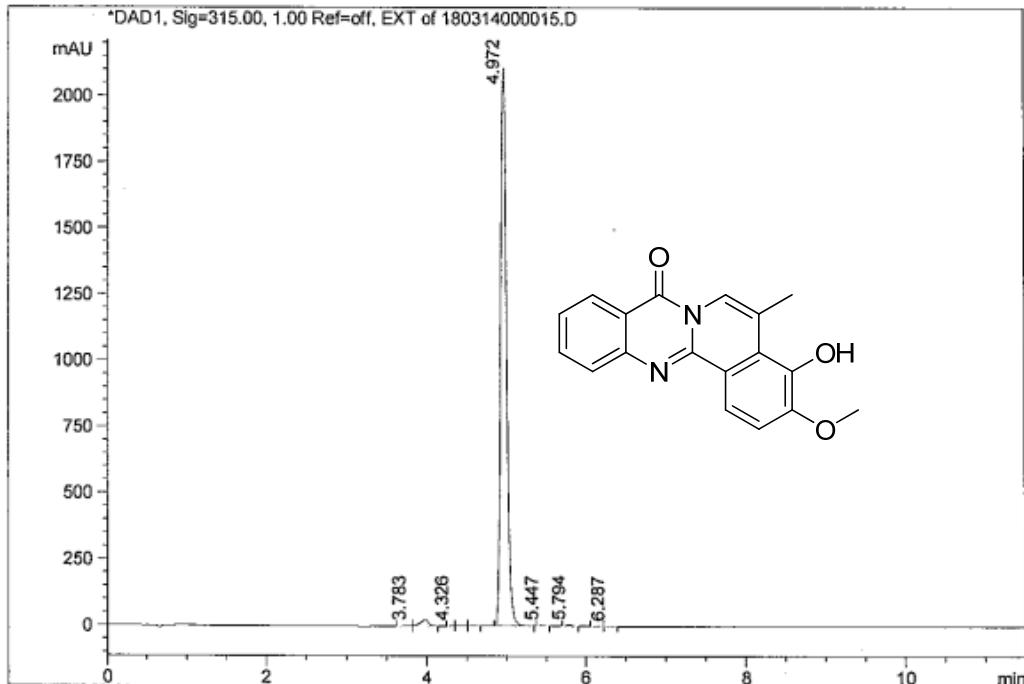


Fig. 16:  $^{13}\text{C}$  NMR spectra of compound **4d** ( $\text{CDCl}_3$ , 100 MHz)

Seq Line : 0  
 Injection Date : Wed, 19. Mar. 2014 Location : Vial 23  
 Sample Name : ILS/ARJ/5/14 Inj. No. : 0  
 Acq Operator : RADHA Inj. Vol. : 0  $\mu$ l  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A20B80.M  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18-A70B30G.M  
 Method Info : Column :Symmetry C-18 75\*4.6mm, 3.5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water,B) ACN (gradient)  
 T/B%:0/20,1/20,4/98,10/98,10.5/20,12/20.  
 FLOW:1.0ml/min Dil: ACN:Water(80:20)



Signal 1: DAD1, Sig=315.00, 1.00 Ref=off, EXT

Peak	RT	Area	Area %
#	[min]		
1	3.783	14.608	0.134
2	3.981	157.912	1.450
3	4.326	1.662	0.015
4	4.440	10.346	0.095
5	4.587	7.519	0.069
6	4.972	10664.799	97.961
7	5.447	4.873	0.045
8	5.794	20.334	0.187
9	6.140	2.189	0.020
10	6.287	2.564	0.024

✓ 19/03/14

Fig. 17: HPLC of compound 4d

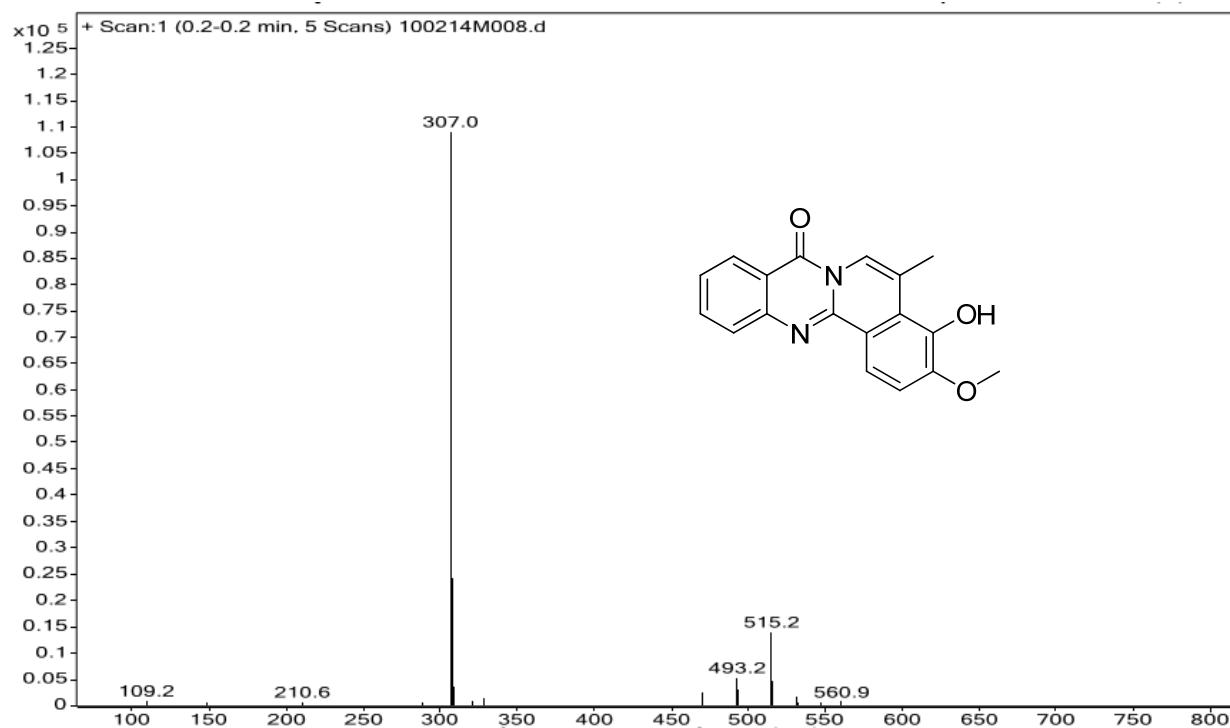


Fig. 18: Mass of compound 4d

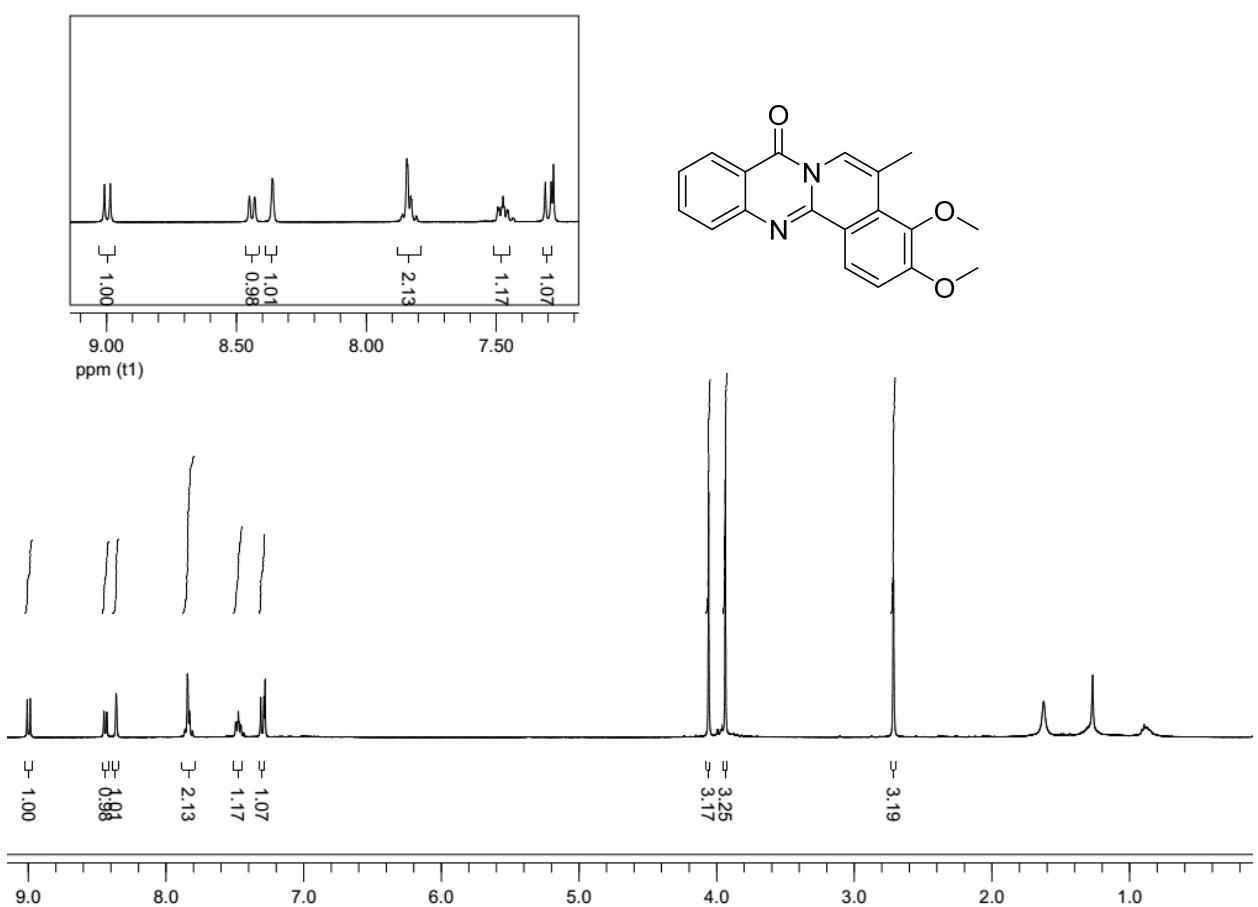


Fig. 19: <sup>1</sup>H NMR spectra of compound **4e** (CDCl<sub>3</sub>, 400 MHz)

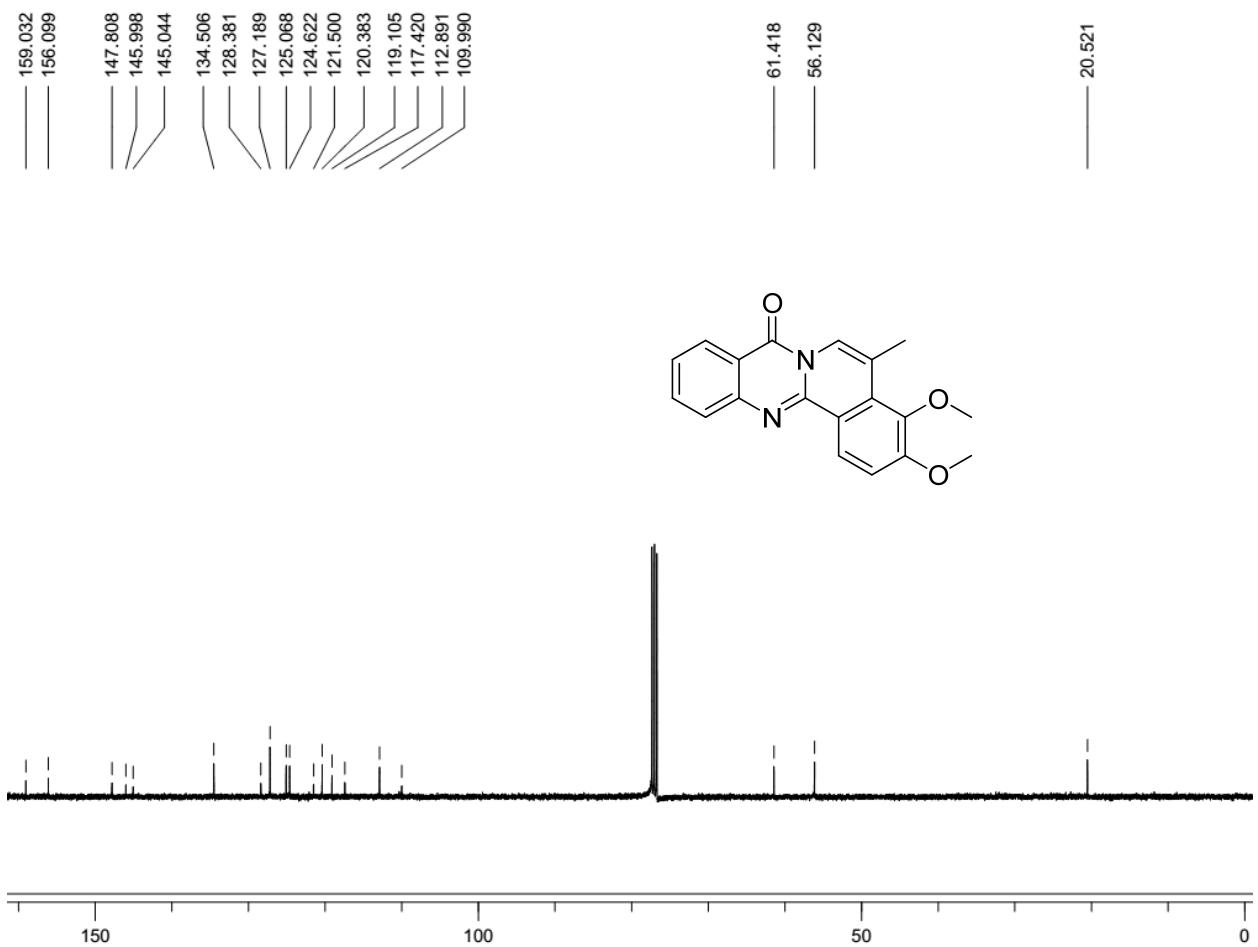
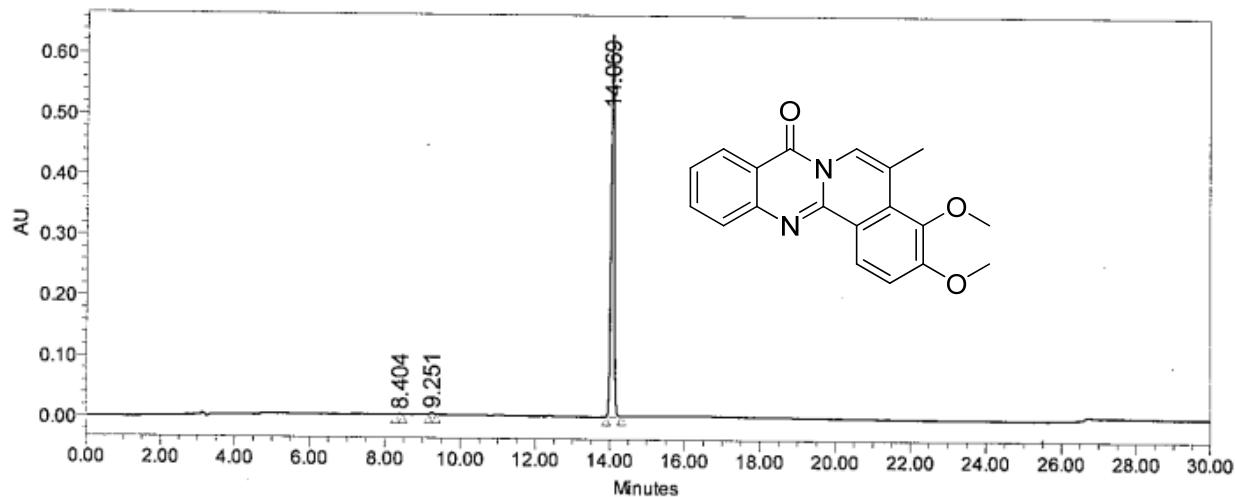


Fig. 20:  $^{13}\text{C}$  NMR spectra of compound **4e** ( $\text{CDCl}_3$ , 100 MHz)

## SAMPLE INFORMATION

Sample Name:	ILS/ARJ/5/11	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	36	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	290.0nm
Injection Volume:	5.00 ul	Proc. Chnl. Descr.:	PDA 290.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/22/2014 12:44:32 AM IST		
Date Processed:	2/24/2014 11:59:50 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5µm  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



*M. Sankar*  
Analysed By

Fig. 21: HPLC of compound 4e

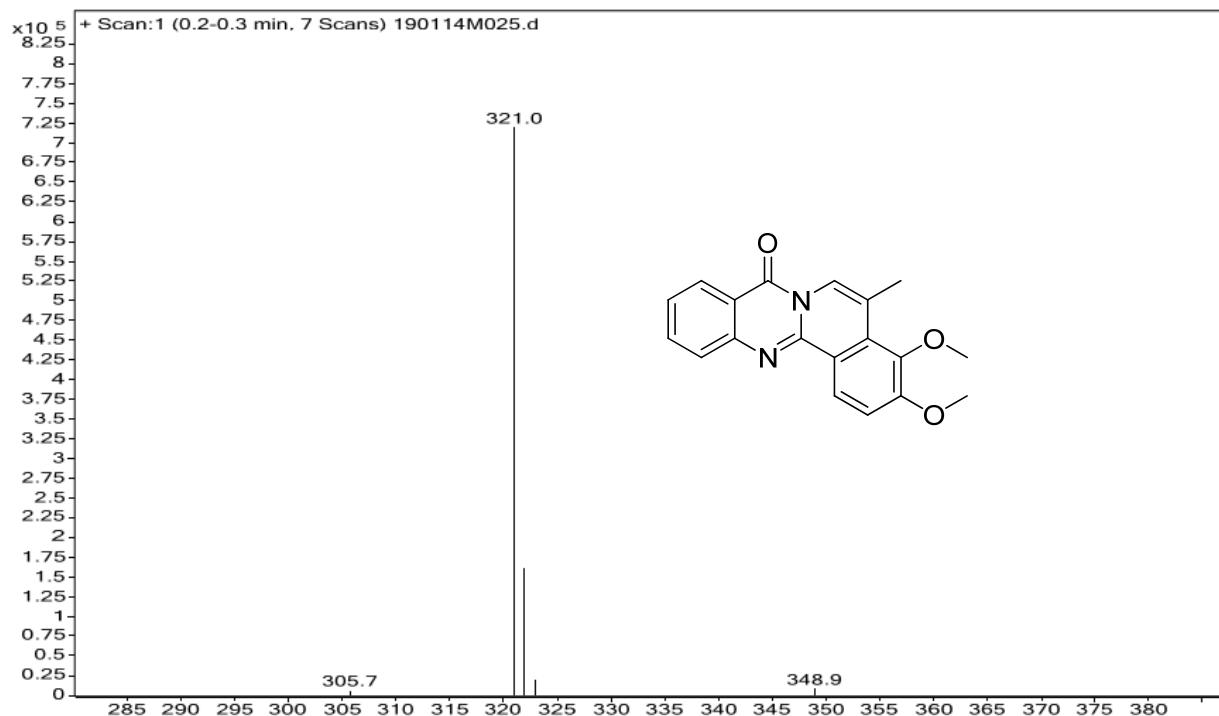


Fig. 22: Mass of compound **4e**

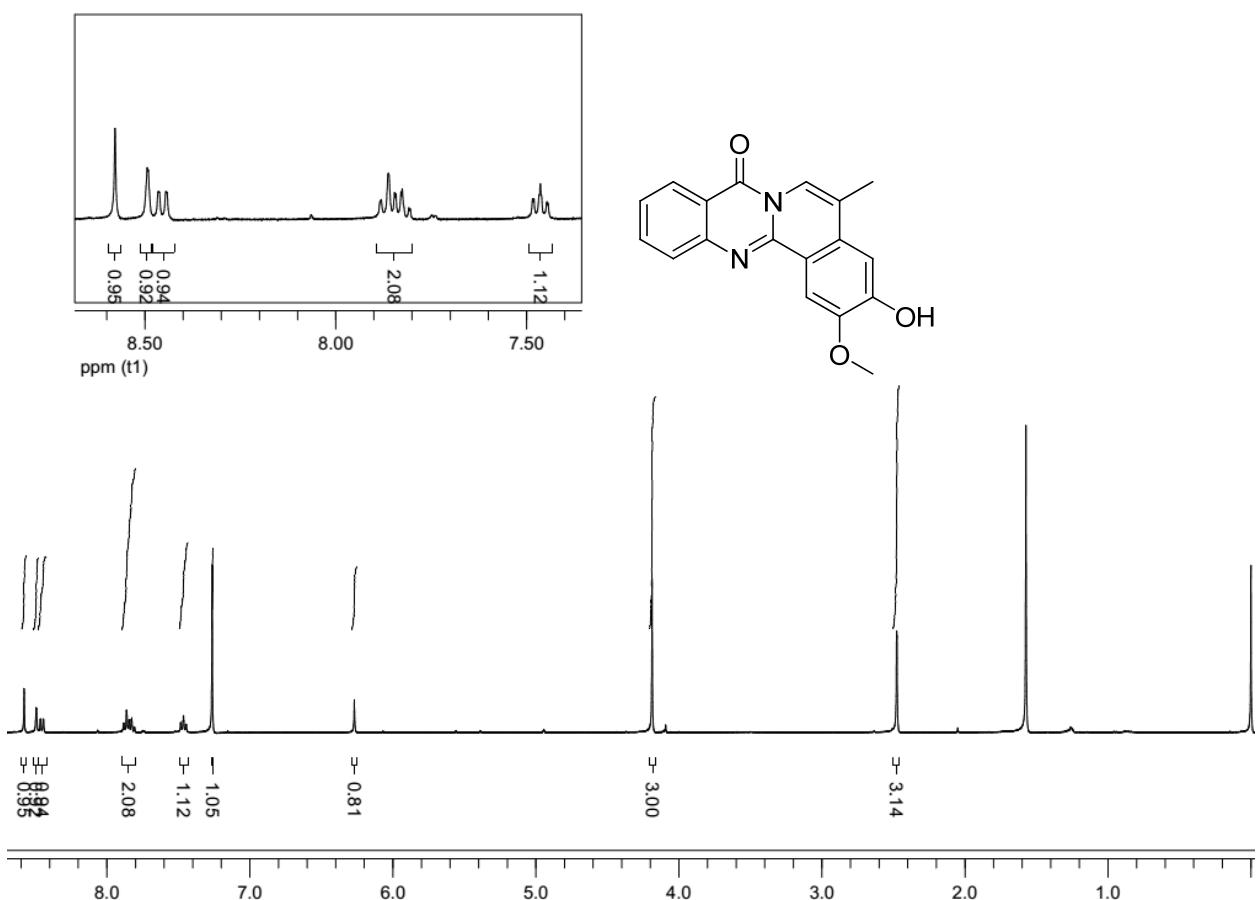


Fig. 23: <sup>1</sup>H NMR spectra of compound **4f** ( $\text{CDCl}_3$ , 400 MHz)

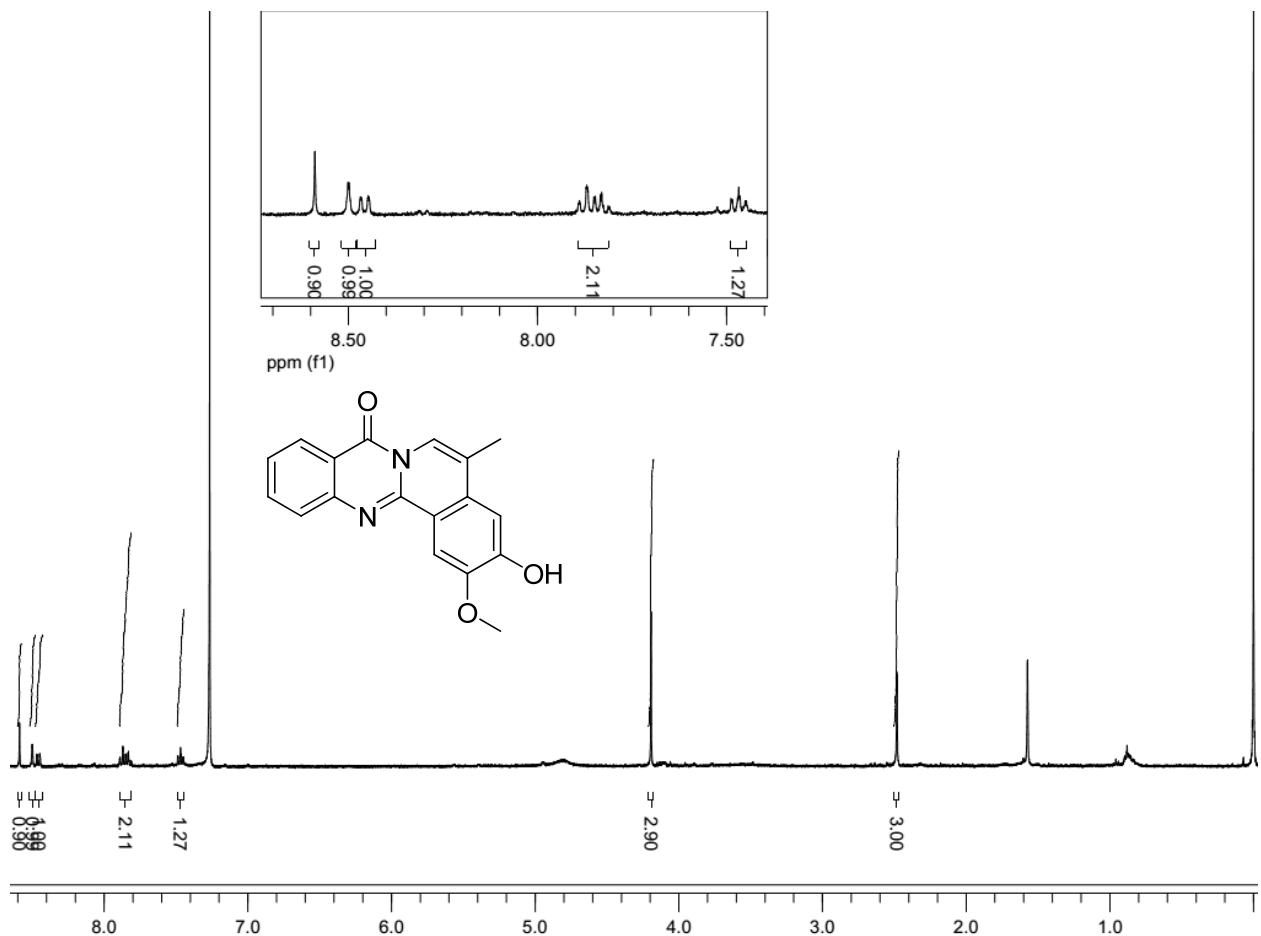


Fig. 24:  $\text{D}_2\text{O}$  exchange spectra of compound **4f** ( $\text{CDCl}_3$ , 400 MHz)

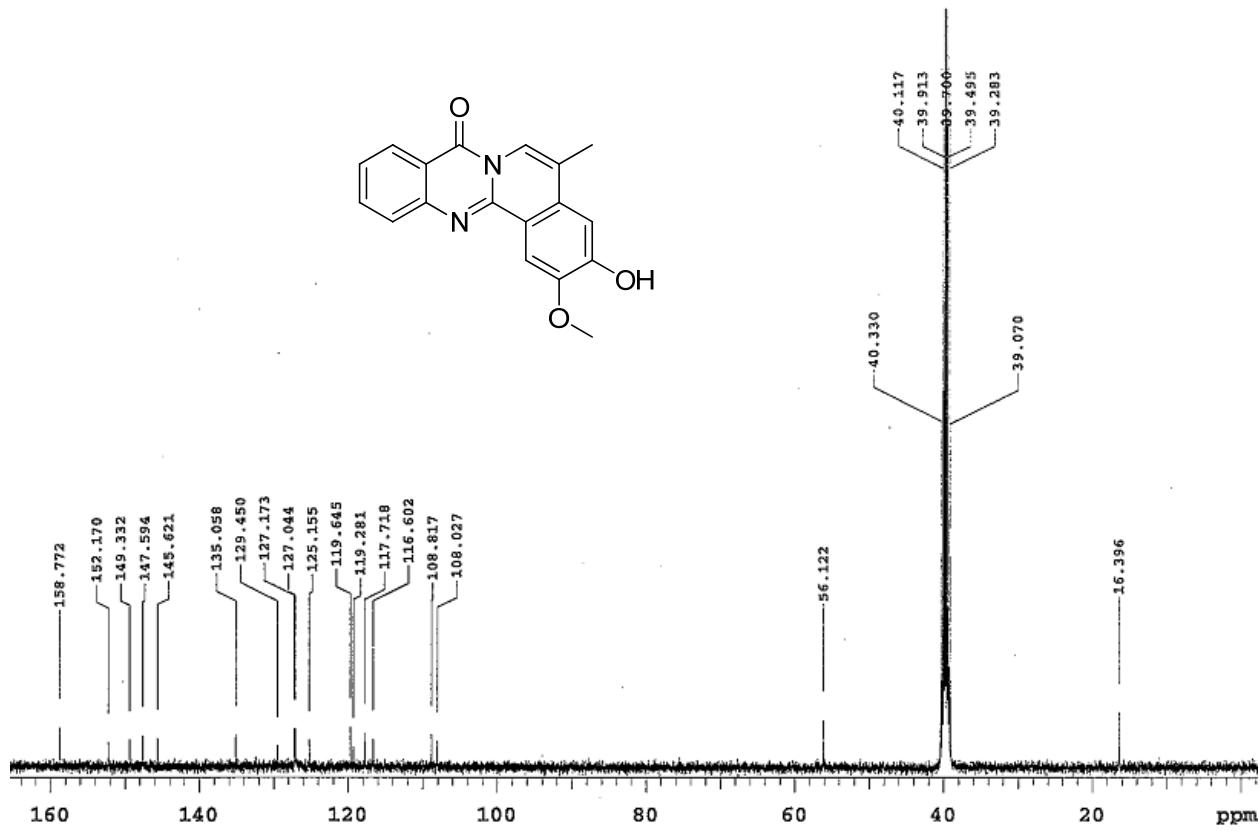
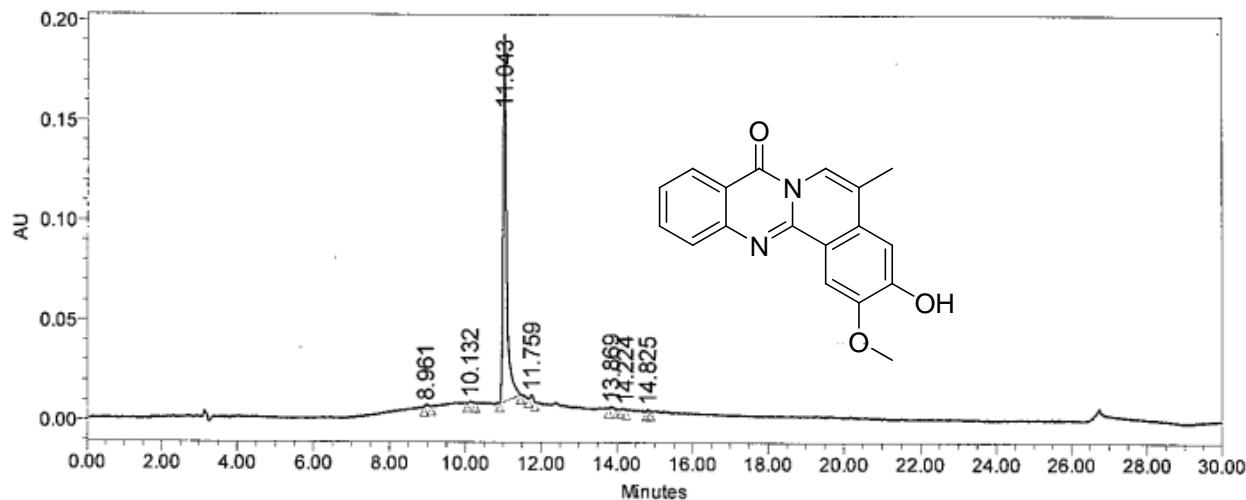


Fig. 25:  $^{13}\text{C}$  NMR spectra of compound **4f** (DMSO-*d*<sub>6</sub>, 100 MHz)

Sample Information	
Sample Name:	ILS/ARJ/5/10
Sample Type:	Unknown
Vial:	35
Injection #:	1
Injection Volume:	5.00 ul
Run Time:	30.0 Minutes
Sample Set Name:	240214
Acq. Method Set:	CFZ
Processing Method:	CFZ_PRO
Channel Name:	280.0nm
Proc. Chnl. Descr.:	PDA 280.0 nm
Date Acquired:	2/24/2014 11:37:36 AM IST
Date Processed:	2/24/2014 12:51:16 PM IST

Column: X TERRA RP-18 250\*4.6mm 5μm  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	8.961	7878	0.63	1231
2	10.132	6741	0.54	1037
3	11.043	1212492	96.38	183725
4	11.759	13554	1.08	2704
5	13.869	8926	0.71	1238
6	14.224	5084	0.40	843
7	14.825	3320	0.26	1082

M 26/02/14  
 Analysed By

Fig. 26: HPLC of compound 4f

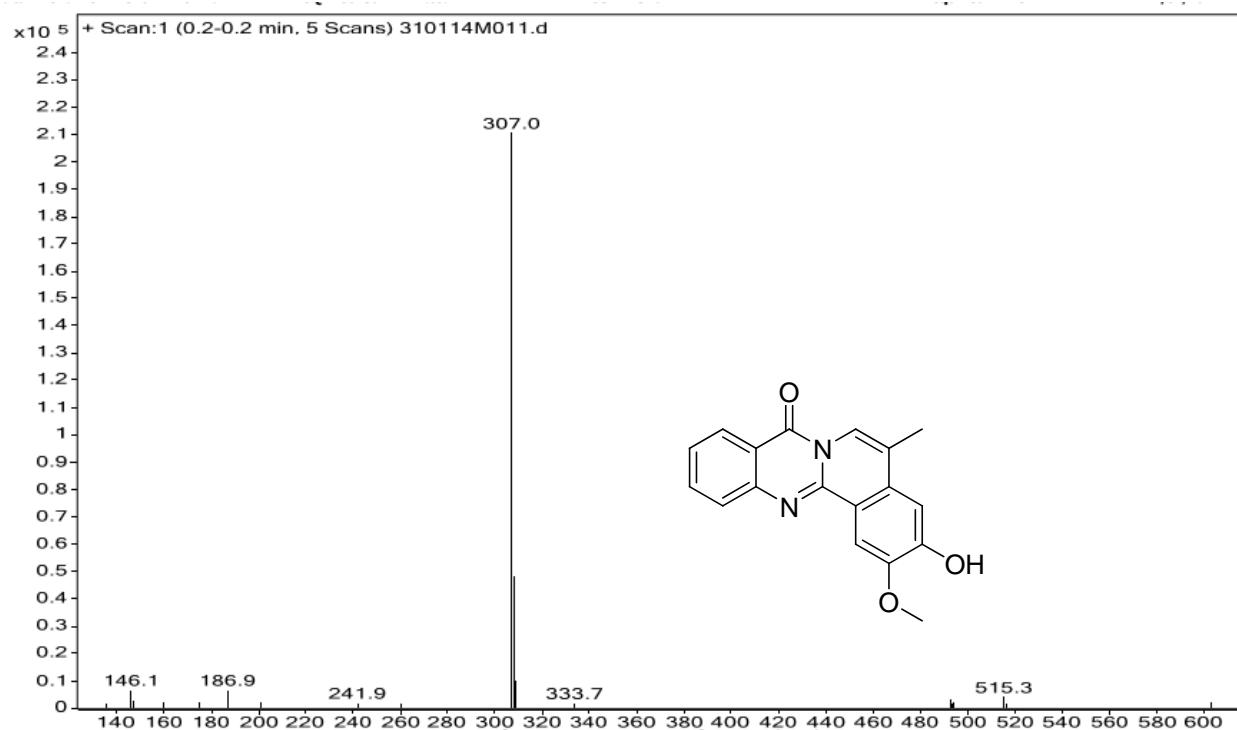


Fig. 27: Mass of compound **4f**

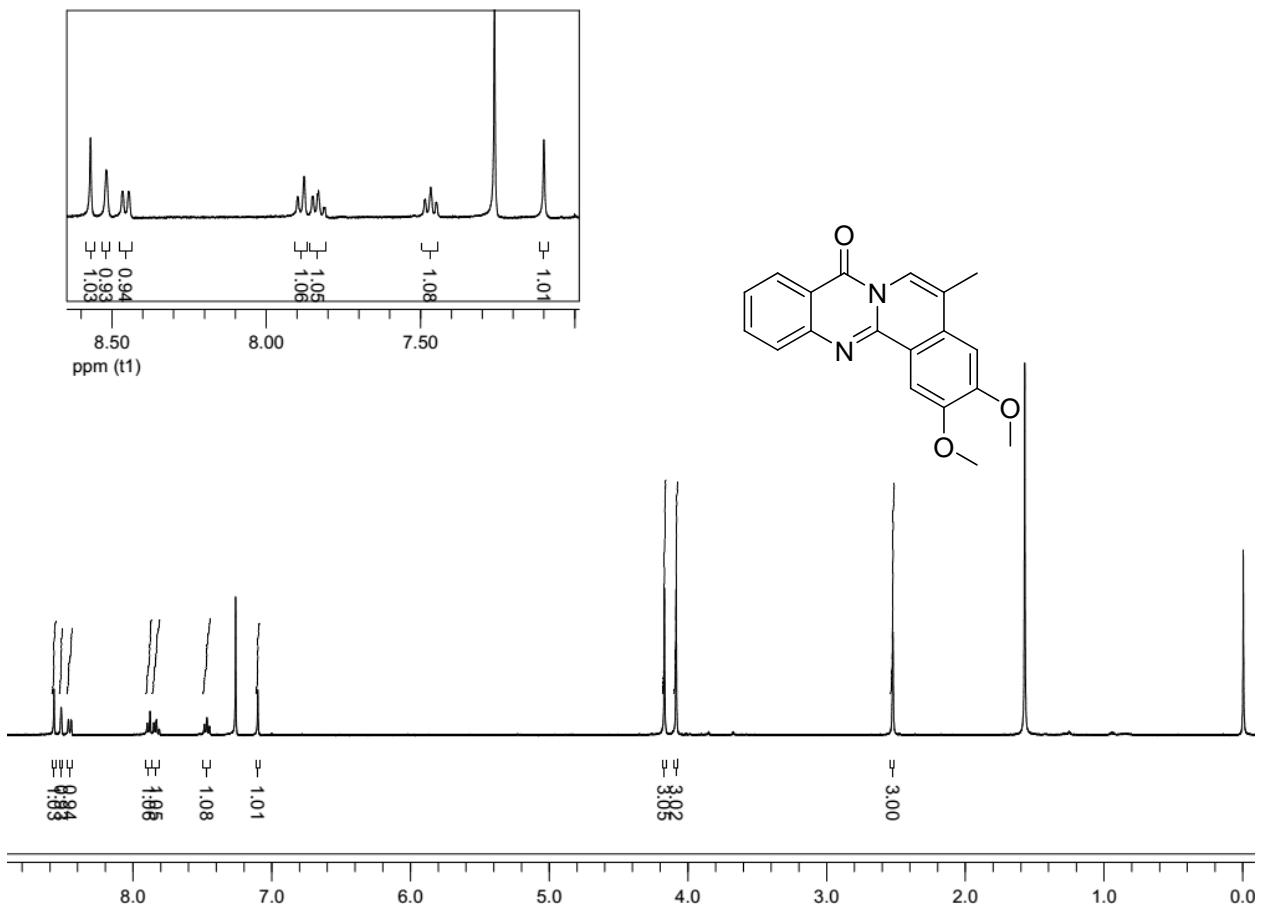


Fig. 28: <sup>1</sup>H NMR spectra of compound **4g** ( $\text{CDCl}_3$ , 400 MHz)

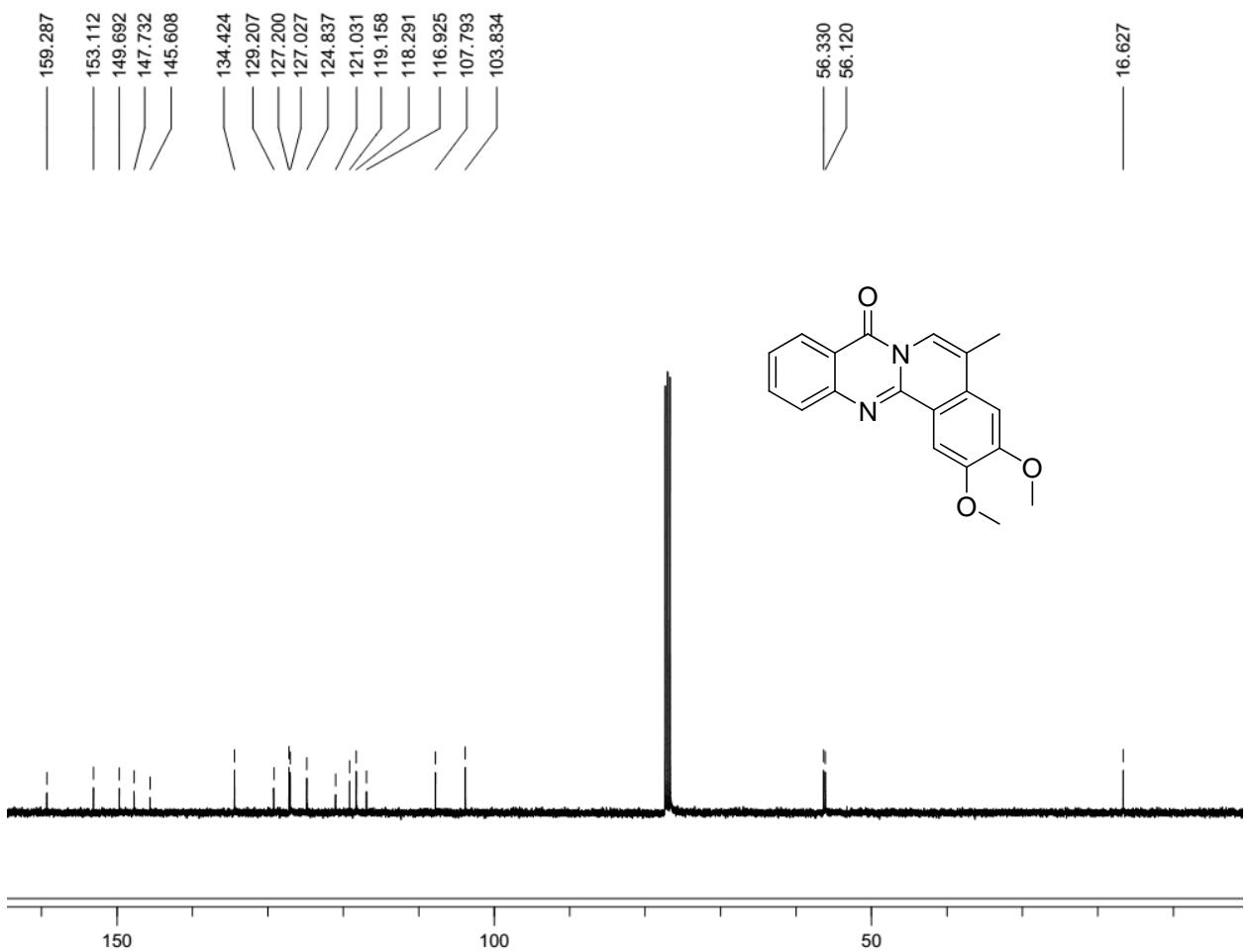
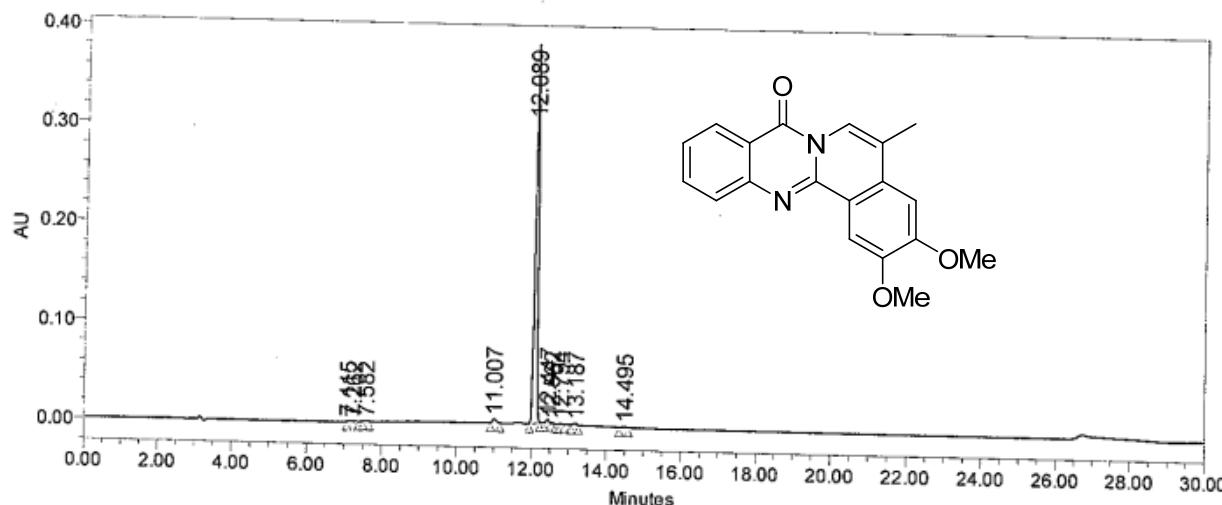


Fig. 29:  $^{13}\text{C}$  NMR spectra of compound **4g** ( $\text{CDCl}_3$ , 100 MHz)

## SAMPLE INFORMATION

Sample Name:	ILS/ARJ/5/3	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	28	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	275.0nm
Injection Volume:	5.00 $\mu$ l	Proc. Chnl. Descr.:	PDA 275.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 7:58:58 PM IST		
Date Processed:	2/24/2014 11:38:42 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T%:B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	7.115	6667	0.32	901
2	7.262	3501	0.17	851
3	7.582	5312	0.25	819
4	11.007	27257	1.29	4274
5	12.089	2011708	95.33	381742
6	12.447	21684	1.03	4054
7	12.562	5996	0.28	1158
8	12.794	6958	0.33	1533
9	13.187	11476	0.54	1760
10	14.495	9621	0.46	1371

*M. Alankar*  
Analysed By

Fig. 30: HPLC of compound 4g

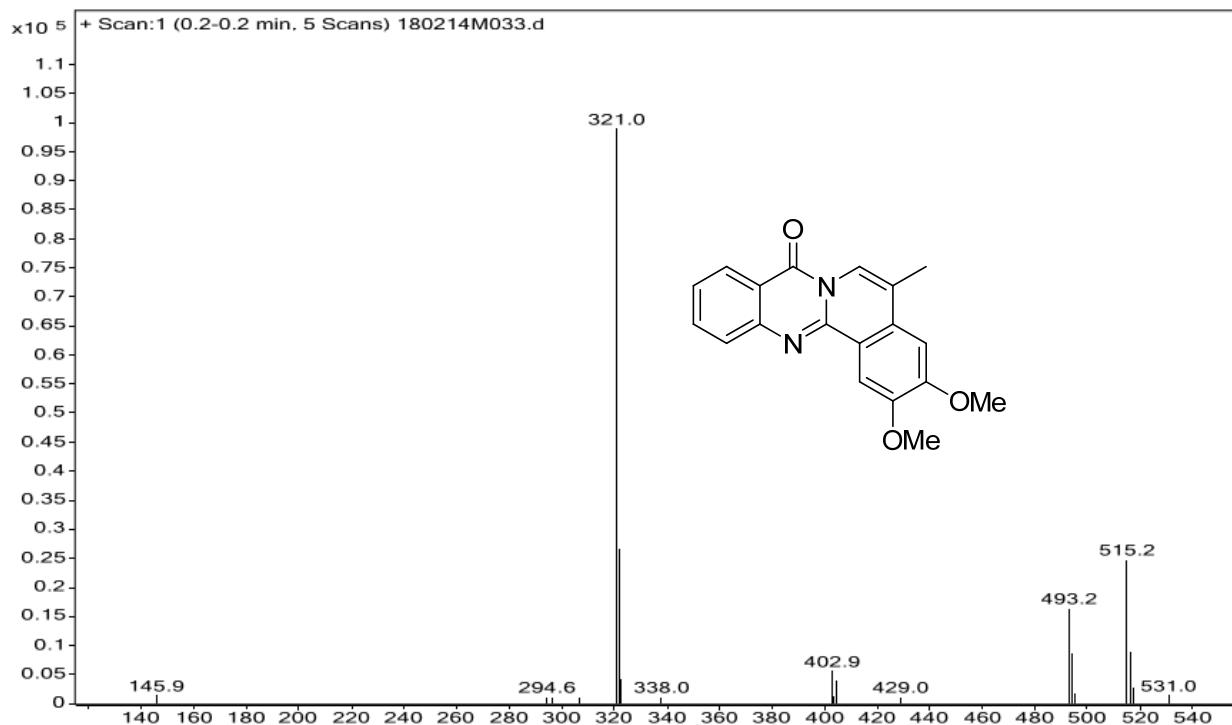


Fig. 31: Mass of compound 4g

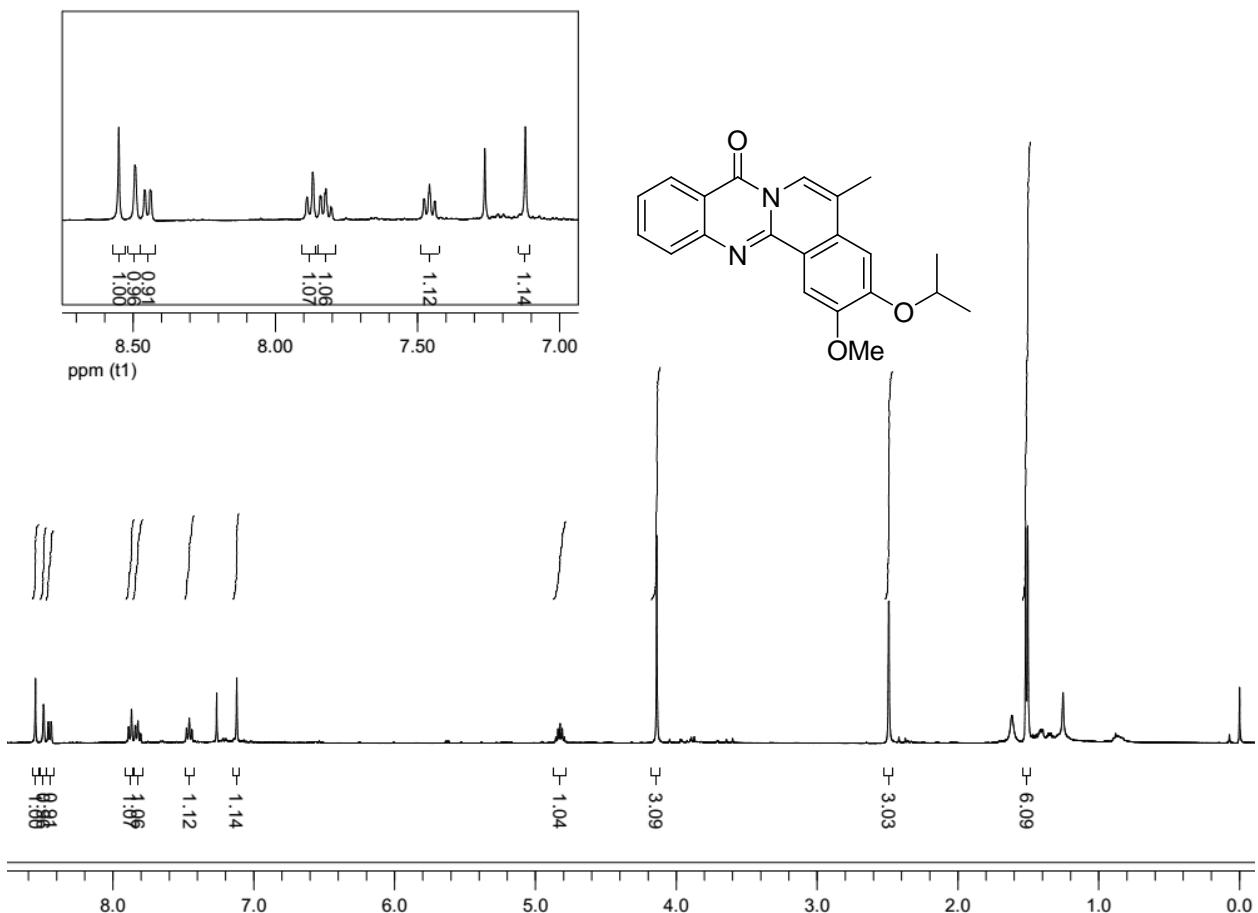


Fig. 32: <sup>1</sup>H NMR spectra of compound **4h** (CDCl<sub>3</sub>, 400 MHz)

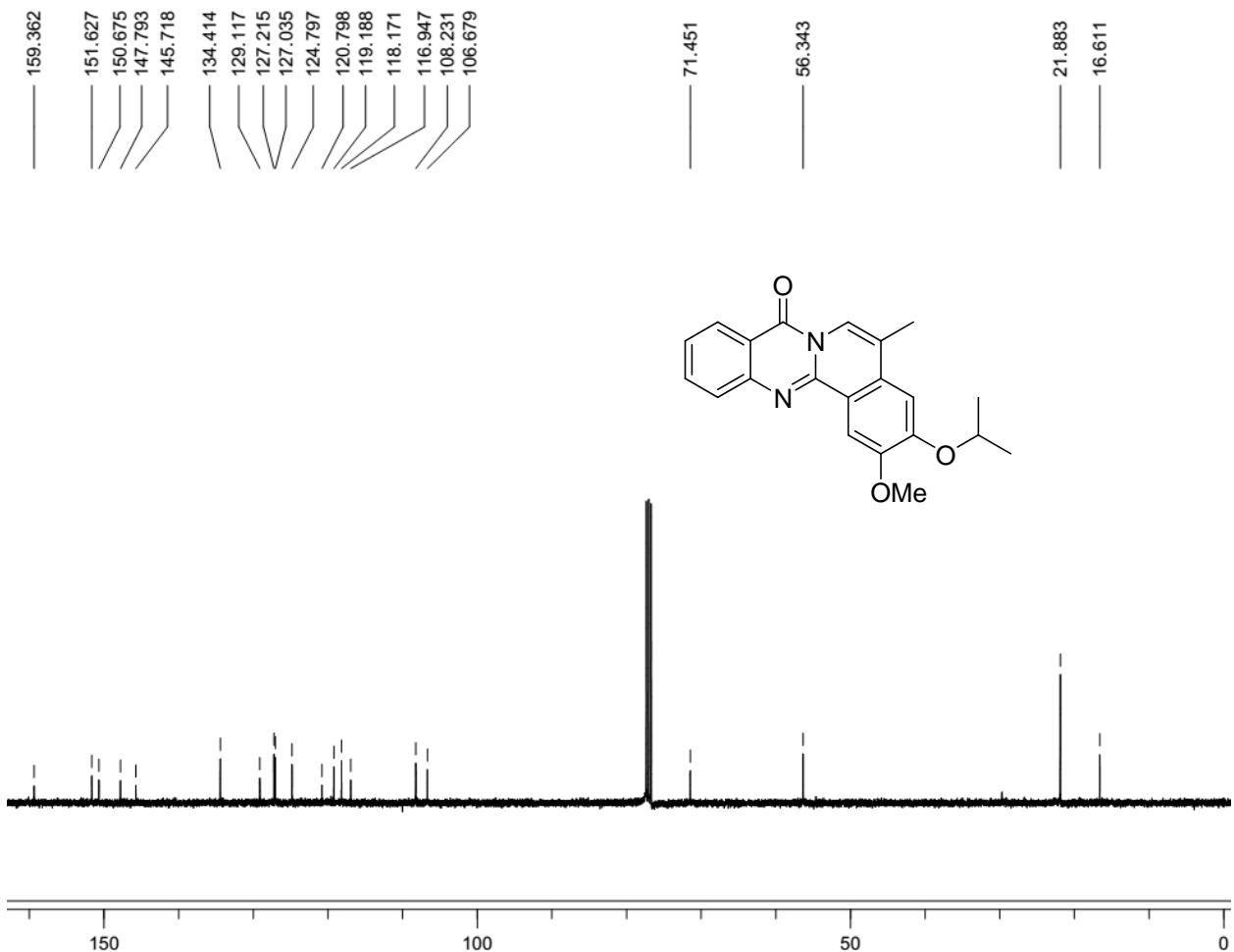
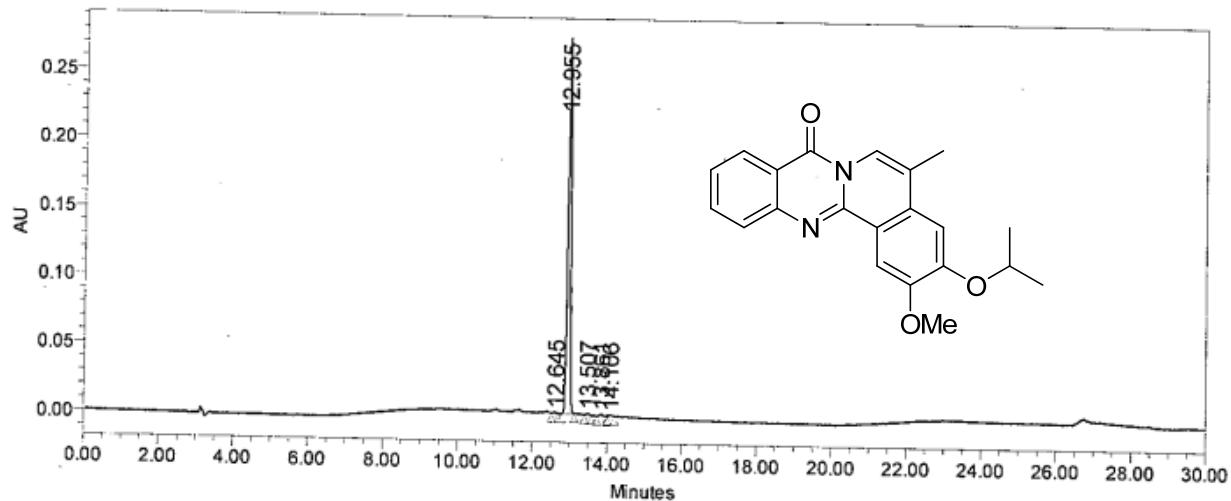


Fig. 33:  $^{13}\text{C}$  NMR spectra of compound **4h** ( $\text{CDCl}_3$ , 100 MHz)

### S A M P L E   I N F O R M A T I O N

Sample Name:	ILS/ARJ/5/9	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	34	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	280.0nm
Injection Volume:	5.00 ul	Proc. Chnl. Descr.:	PDA 280.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 11:33:12 PM IST		
Date Processed:	2/24/2014 11:52:31 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T%:B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	12.645	7085	0.47	1130
2	12.955	1469317	97.64	274357
3	13.507	7735	0.51	1525
4	13.861	13652	0.91	1656
5	14.106	6975	0.46	1150

Analysed by  
*M. S. Patel*

Fig. 34: HPLC of compound 4h

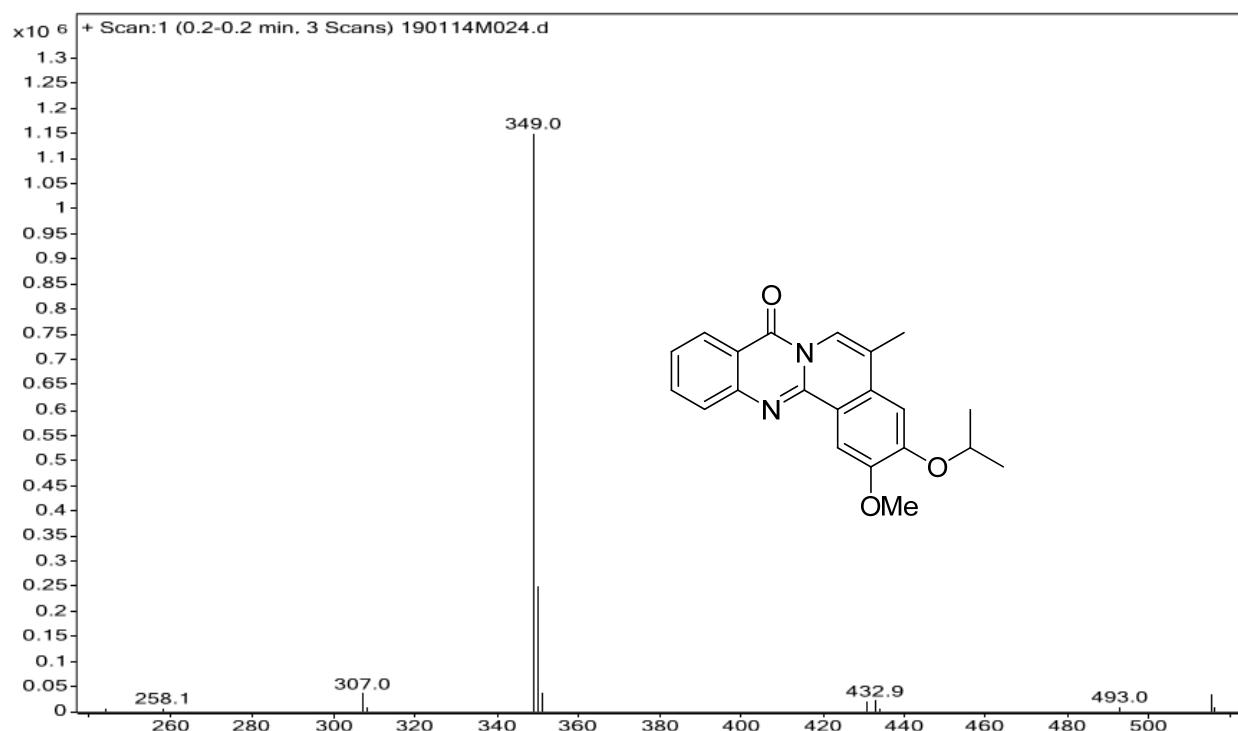


Fig. 35: Mass of compound 4h

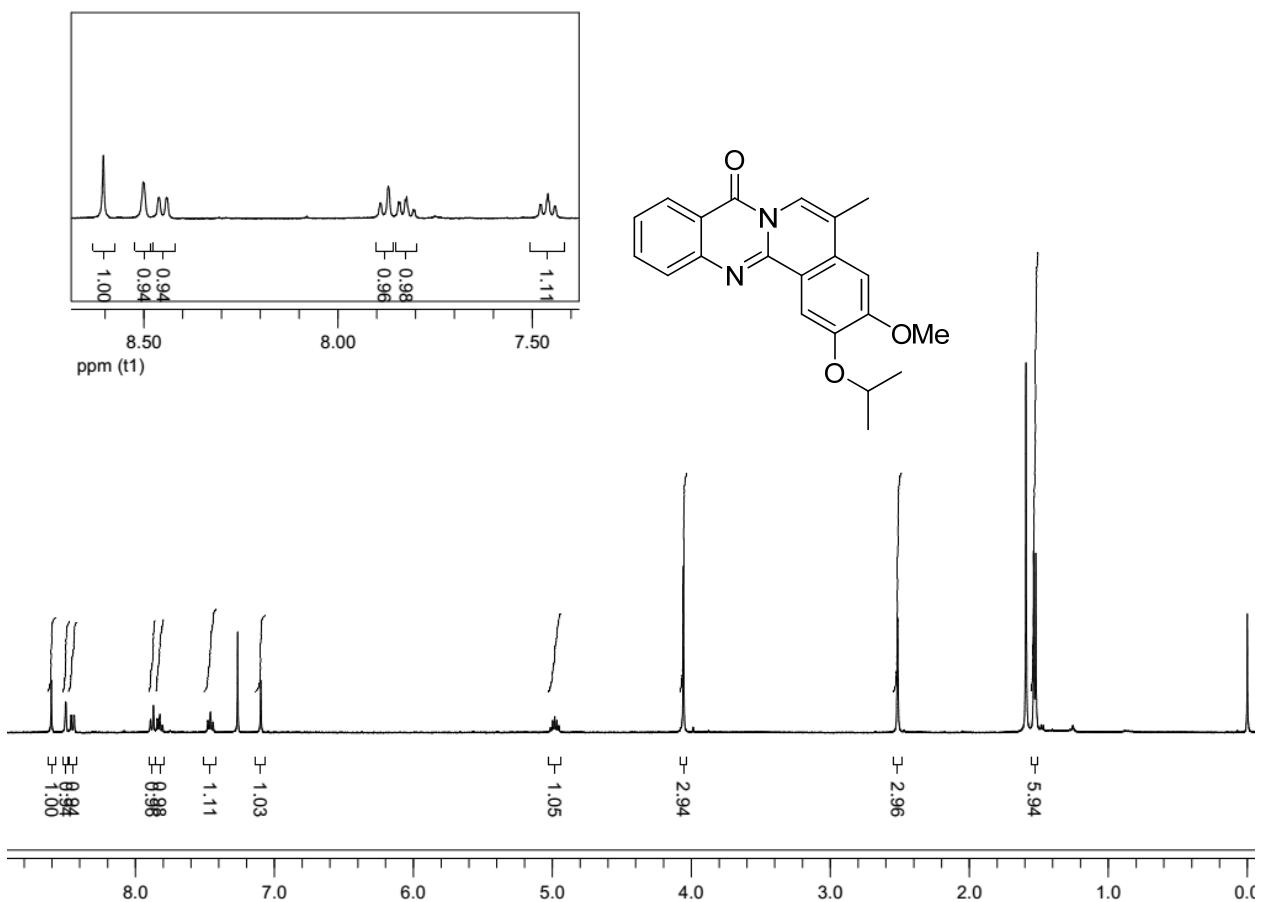


Fig. 36: <sup>1</sup>H NMR spectra of compound **4i** ( $\text{CDCl}_3$ , 400 MHz)

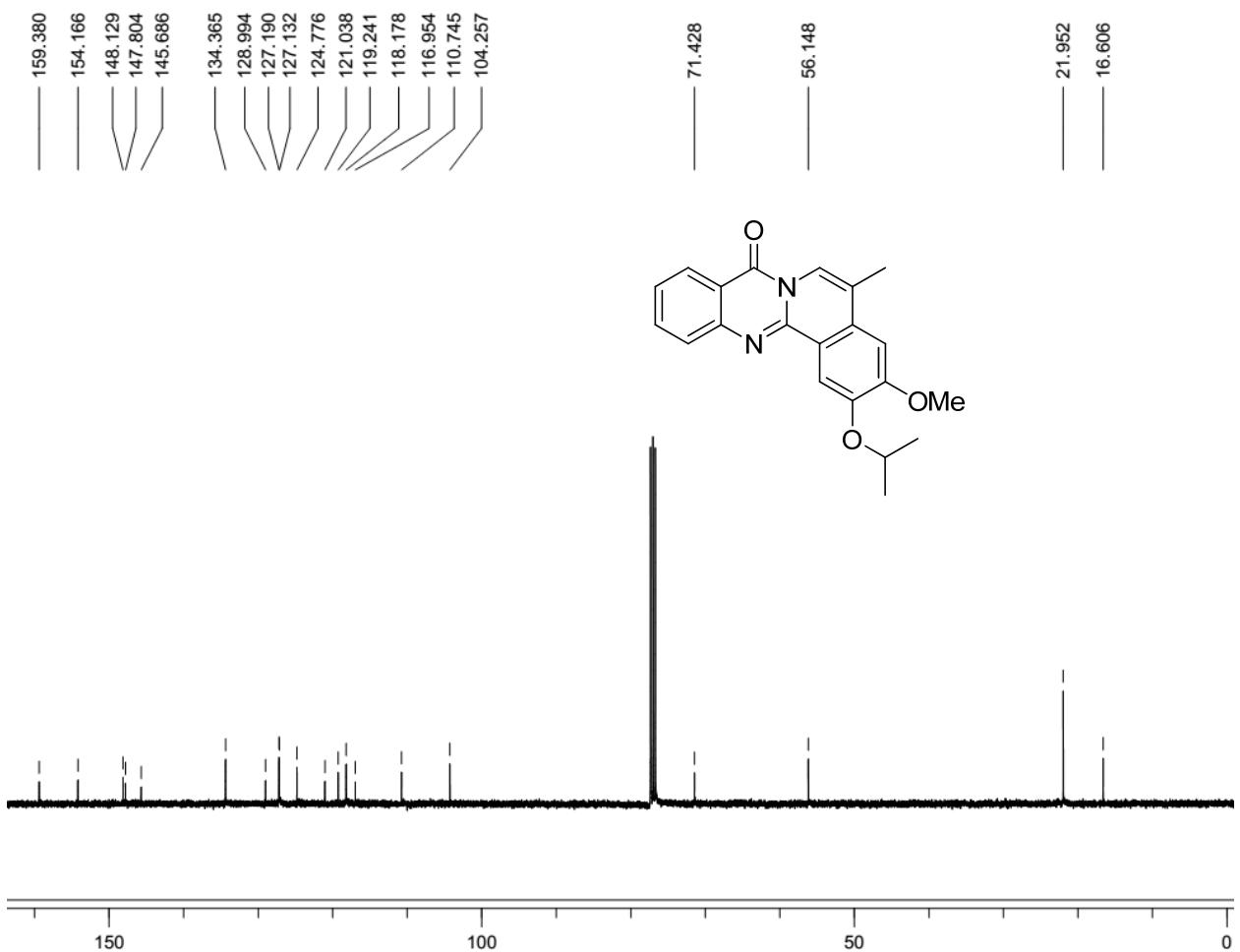
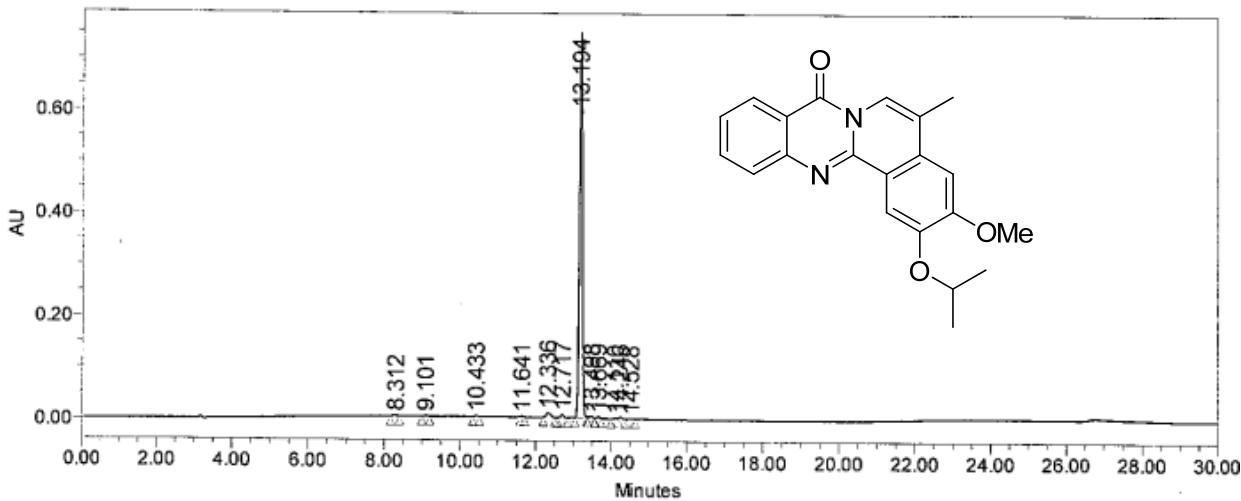


Fig. 37:  $^{13}\text{C}$  NMR spectra of compound **4i** ( $\text{CDCl}_3$ , 100 MHz)

Sample Name:	ILS/ARJ/5/7	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	32	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	280.0nm
Injection Volume:	5.00 ul	Proc. Chnl. Descr.:	PDA 280.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 10:21:46 PM IST		
Date Processed:	2/24/2014 11:48:10 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5μm  
Mobile phase: A) 0.1% TFA in water B) ACN  
T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	8.312	13314	0.32	2010
2	9.101	12744	0.31	2054
3	10.433	12741	0.31	2338
4	11.641	5357	0.13	1433
5	12.336	73027	1.76	8790
6	12.717	39457	0.95	7038
7	13.194	3905451	94.32	744484
8	13.498	15985	0.39	3020
9	13.669	17349	0.42	3161
10	14.110	13534	0.33	2321

	RT	Area	% Area	Height
11	14.246	25763	0.62	4145
12	14.528	6093	0.15	801

Analysed By

Fig. 38: HPLC of compound 4i

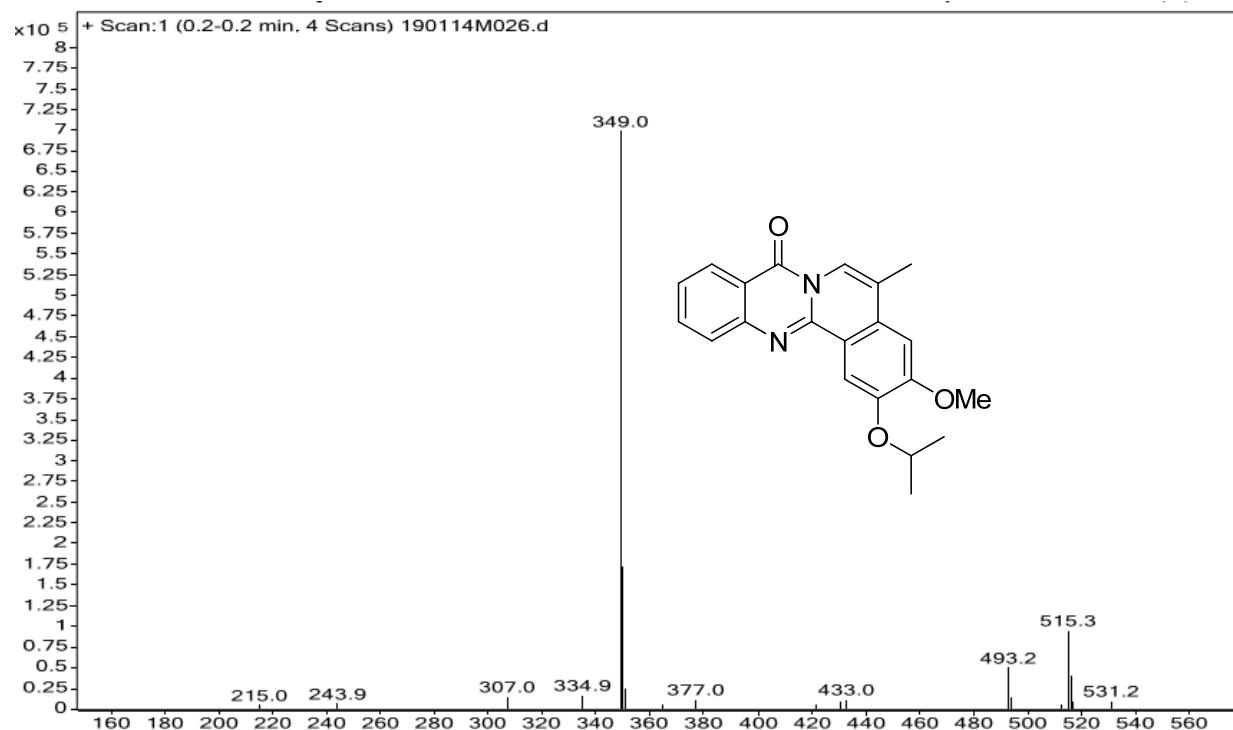


Fig. 39: Mass of compound 4i

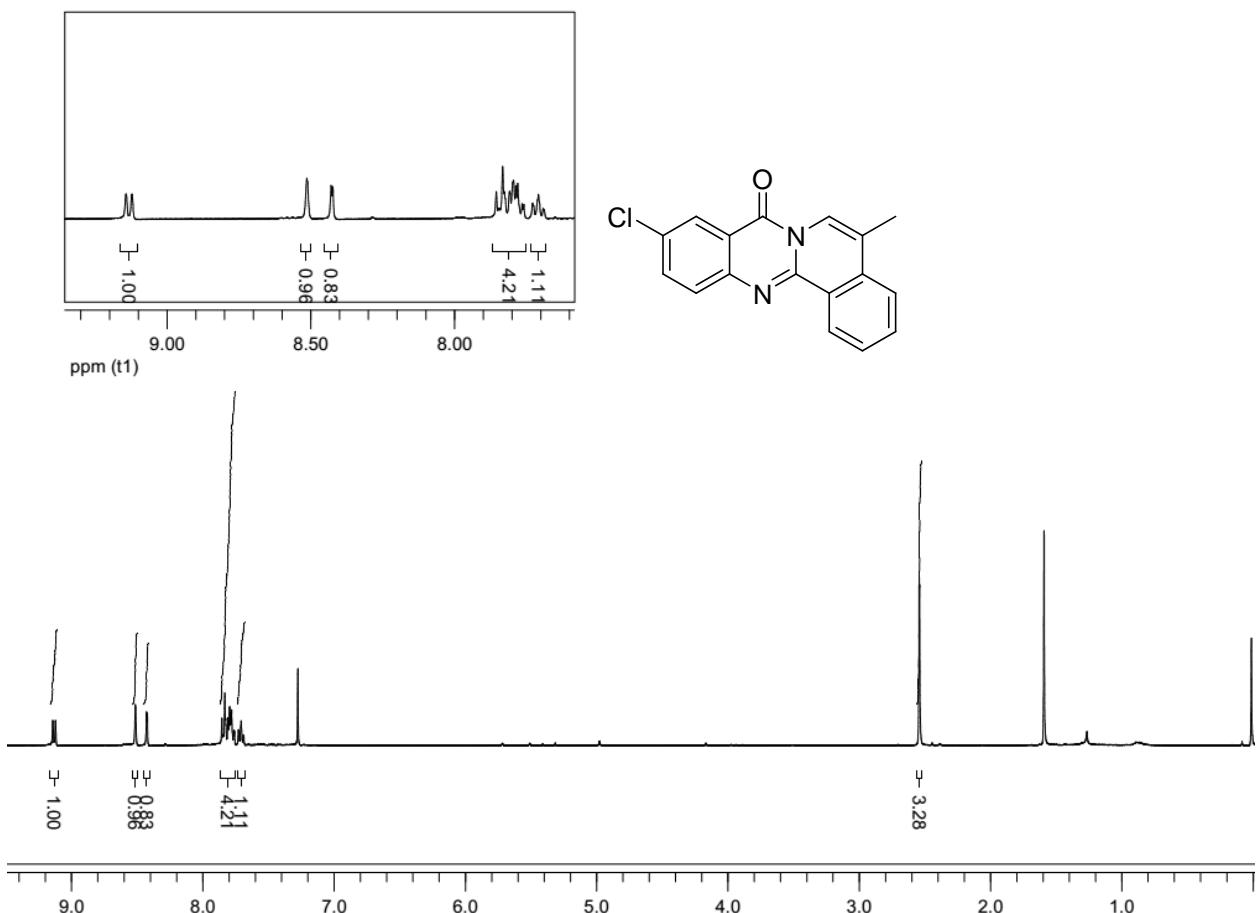


Fig. 40: <sup>1</sup>H NMR spectra of compound **4j** ( $\text{CDCl}_3$ , 400 MHz)

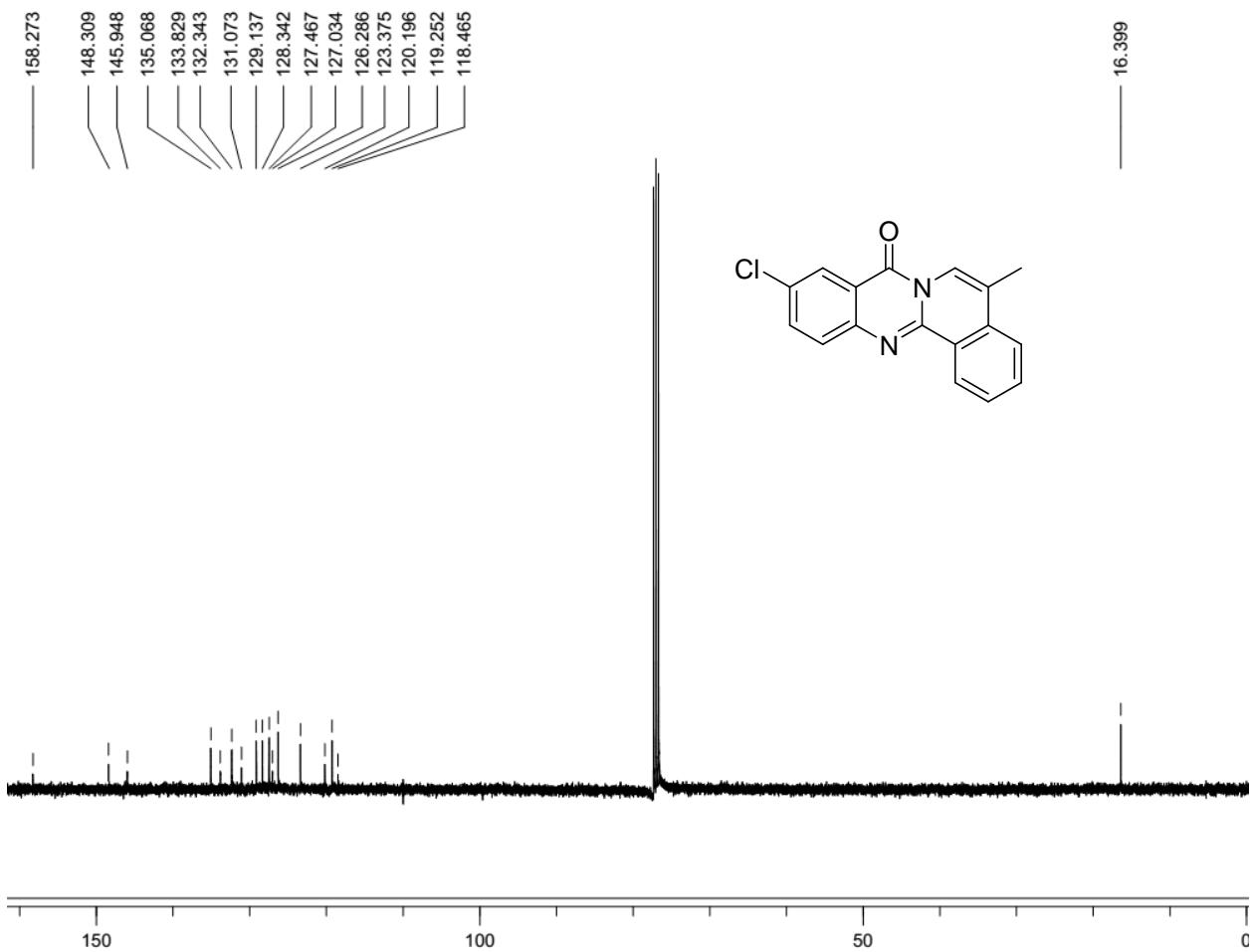
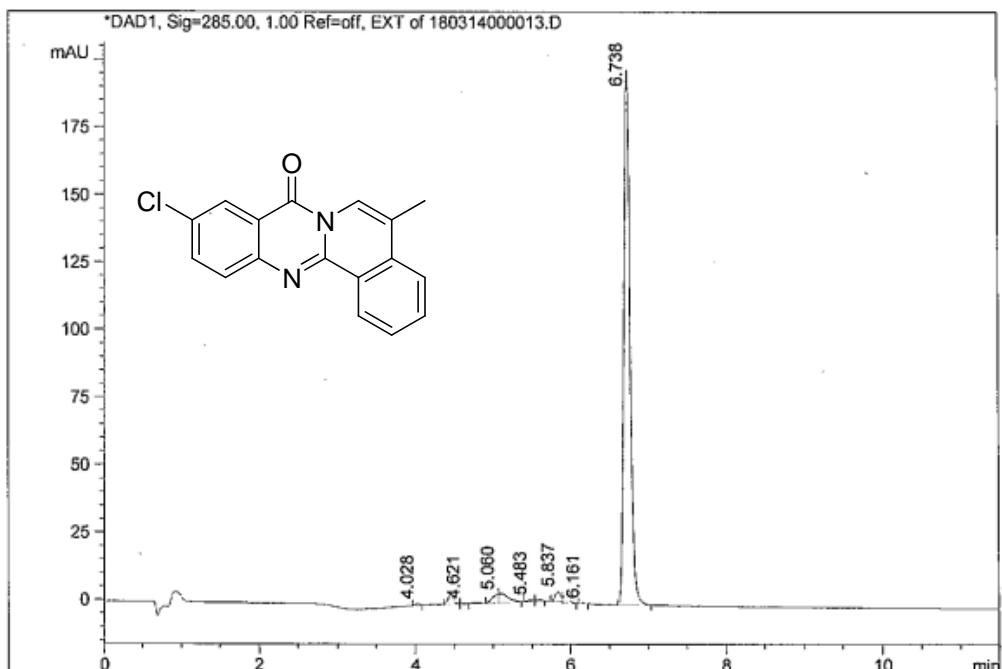


Fig. 41:  $^{13}\text{C}$  NMR spectra of compound **4j** ( $\text{CDCl}_3$ , 100 MHz)

Seq Line : 0  
 Injection Date : Wed, 19. Mar. 2014 Location : Vial 21  
 Sample Name : ILS/ARJ/5/12 Inj. No. : 0  
 Acq Operator : RADHA Inj. Vol. : 0 µl  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A20B80.M  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18-A70B30G.M  
 Method Info : Column :Symmetry C-18 75\*4.6mm, 3.5µm  
 Mobile phase: A) 0.1% TFA in water,B) ACN (gradient)  
 T/B%:0/20,1/20,4/98,10/98,10.5/20,12/20.  
 FLOW:1.0ml/min Dil: ACN:Water(80:20)



=====  
 Signal 1: DAD1, Sig=285.00, 1.00 Ref=off, EXT

Peak #	RT [min]	Area	Area %
1	4.028	2.023	0.166
2	4.451	17.173	1.408
3	4.621	0.928	0.076
4	5.060	15.705	1.288
5	5.109	27.077	2.220
6	5.483	1.724	0.141
7	5.590	2.553	0.209
8	5.837	17.519	1.436
9	5.947	20.322	1.666
10	6.161	0.857	0.070
11	6.738	1113.814	91.319

Fig. 42: HPLC of compound 4j

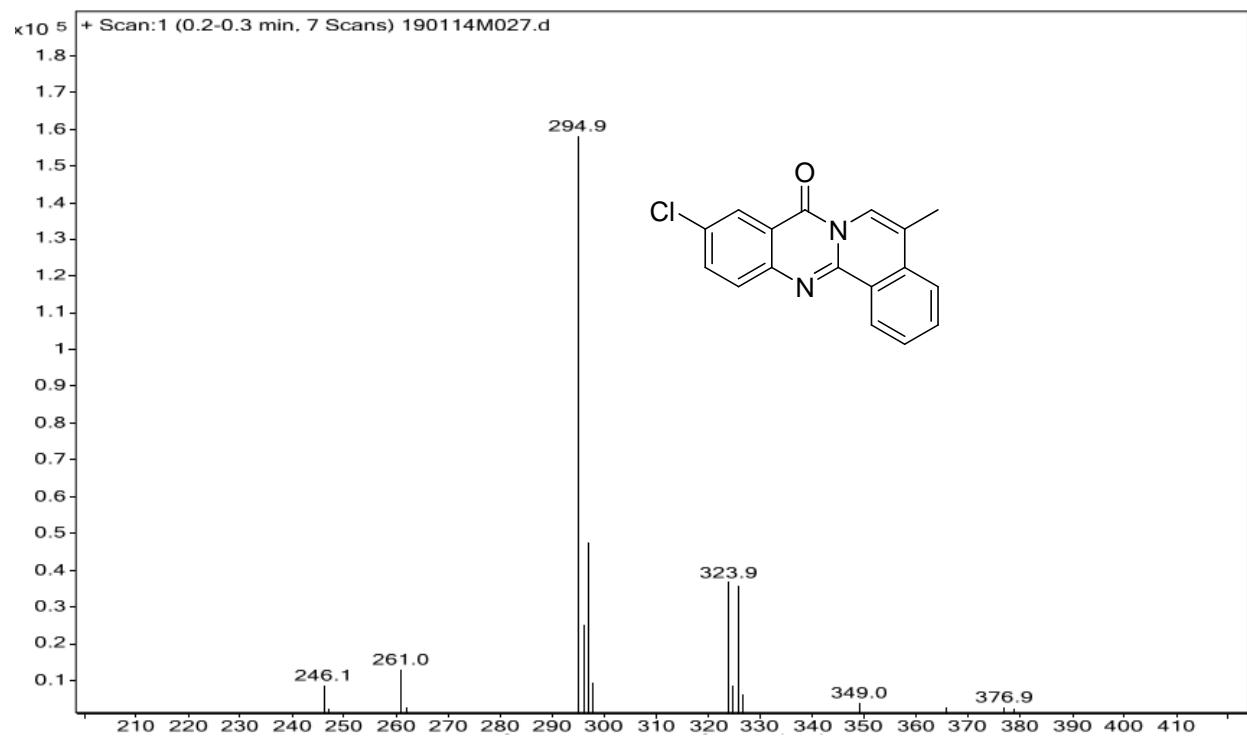


Fig. 43: Mass of compound **4j**

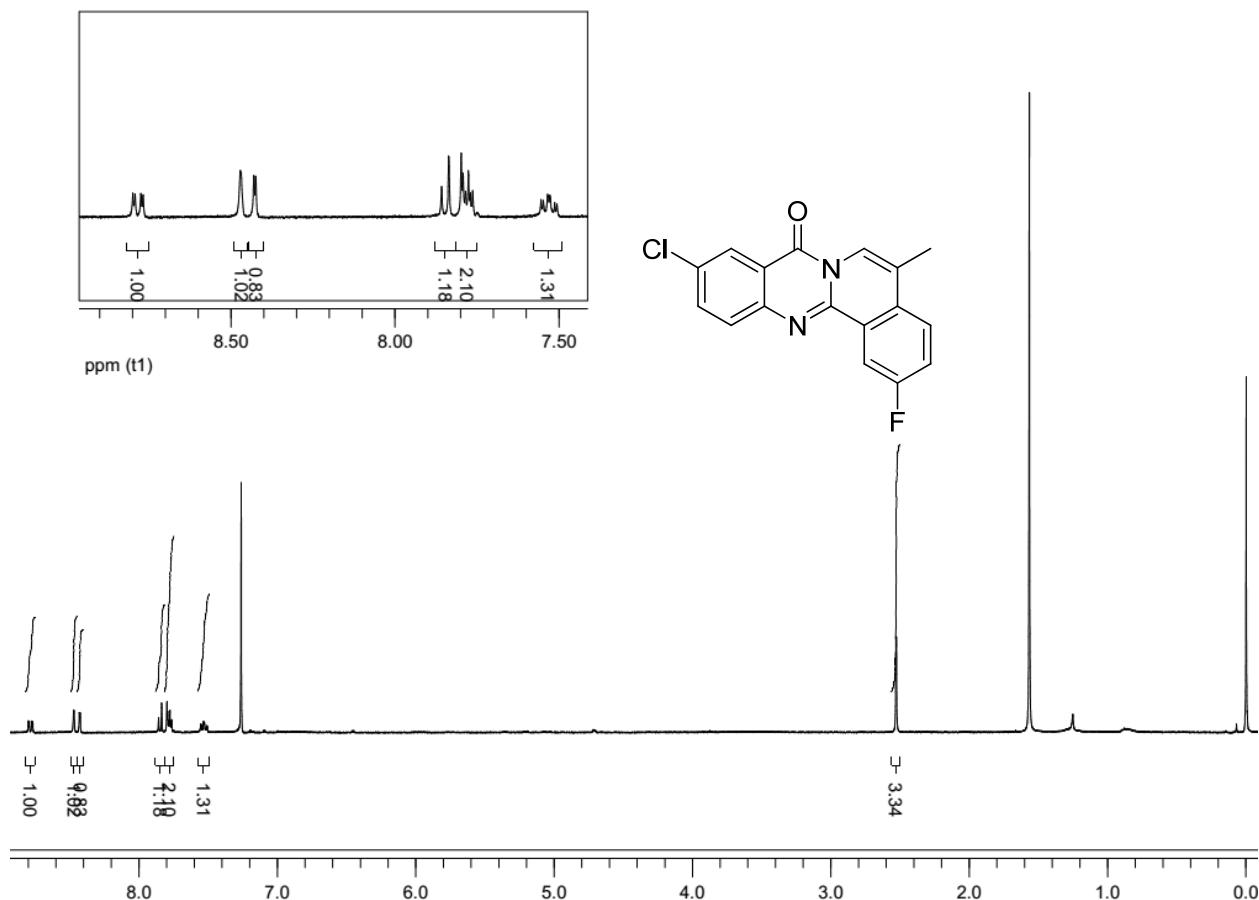


Fig. 44: <sup>1</sup>H NMR spectra of compound **4k** ( $\text{CDCl}_3$ , 400 MHz)

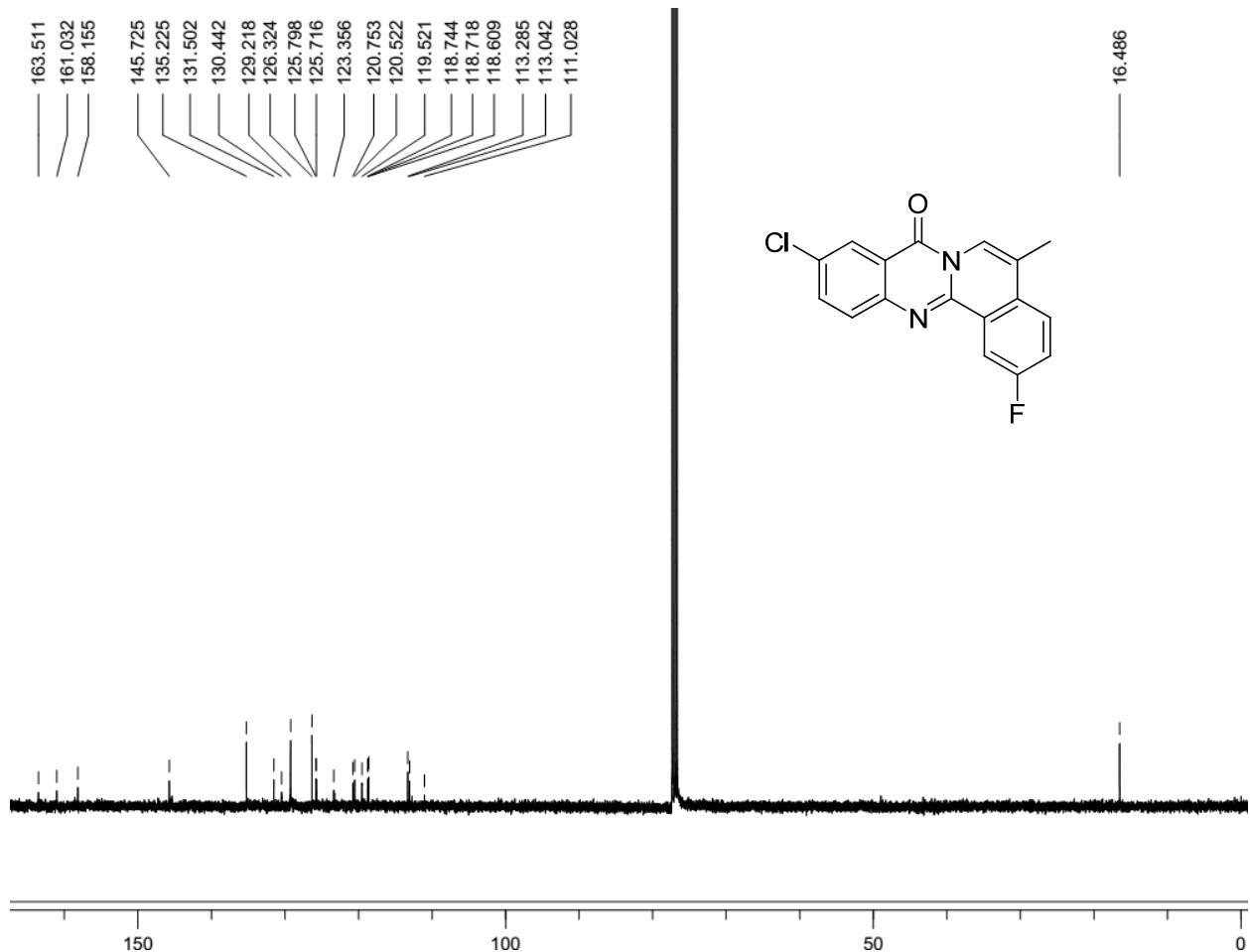
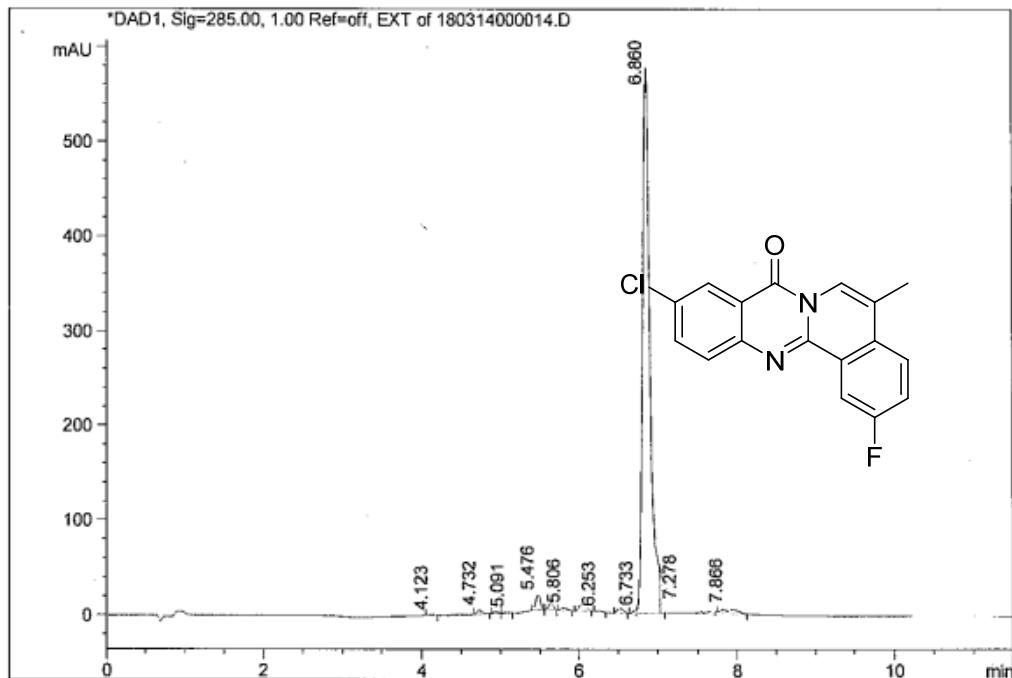


Fig. 45: <sup>13</sup>C NMR spectra of compound **4k** (CDCl<sub>3</sub>, 100 MHz)

Injection Date : Wed, 19. Mar. 2014 Location : Vial 22  
 Sample Name : ILS/ARJ/5/13 Inj. No. : 0  
 Acq Operator : RADHA Inj. Vol. : 0  $\mu$ l  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A20B80.M  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18-A70B30G.M  
 Method Info : Column :Symmetry C-18 75\*4.6mm, 3.5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water,B) ACN (gradient)  
 T/B%:0/20,1/20,4/98,10/98,10.5/20,12/20.  
 FLOW:1.0ml/min Dil: ACN:Water(80:20)



Signal 1: DAD1, Sig=285.00, 1.00 Ref=off, EXT

Peak#	RT [min]	Area	Area %	Peak#	RT [min]	Area	Area %
1	4.123	3.570	0.075	12	6.860	3595.715	92.094
2	4.732	19.446	0.406	13	7.278	1.796	0.035
3	4.932	7.481	0.156	14	7.866	63.376	1.324
4	5.091	5.208	0.109				
5	5.476	66.204	1.383				
6	5.643	32.734	0.684				
7	5.806	13.061	0.273				
8	6.041	46.150	0.964				
9	6.253	5.979	0.125				
10	6.524	25.533	0.533				
11	6.733	11.030	0.230				

Fig. 46: HPLC of compound 4k

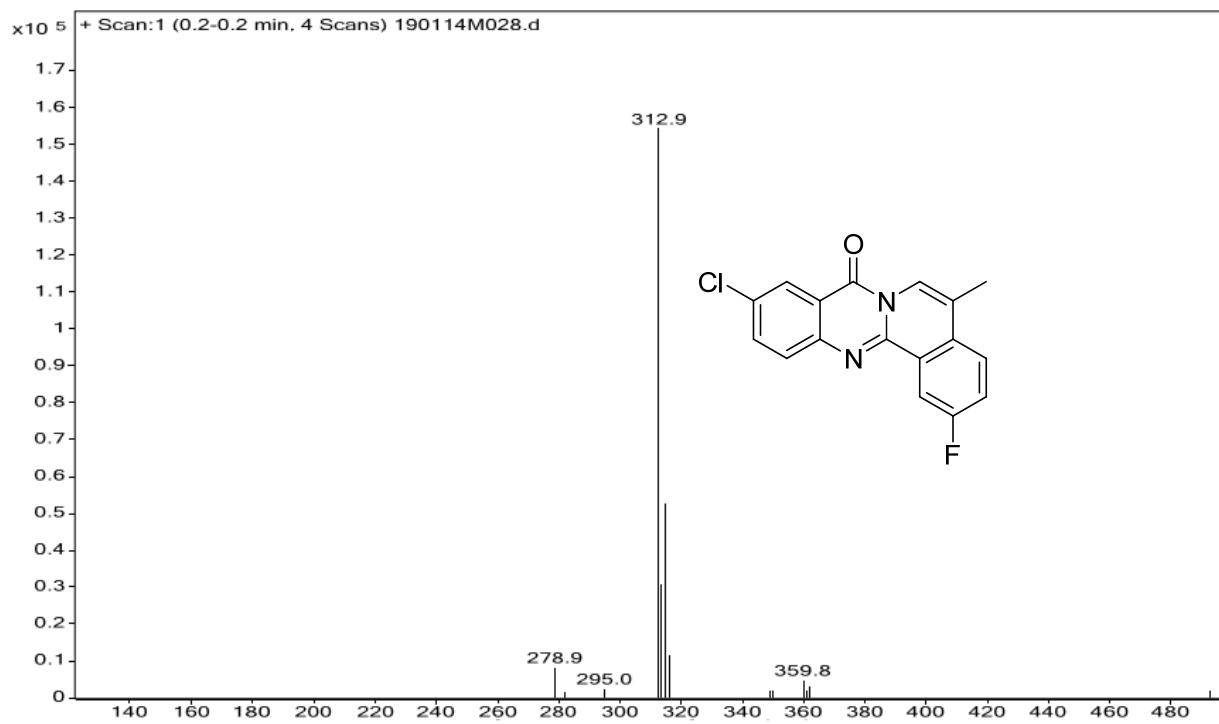


Fig. 47: Mass of compound 4k

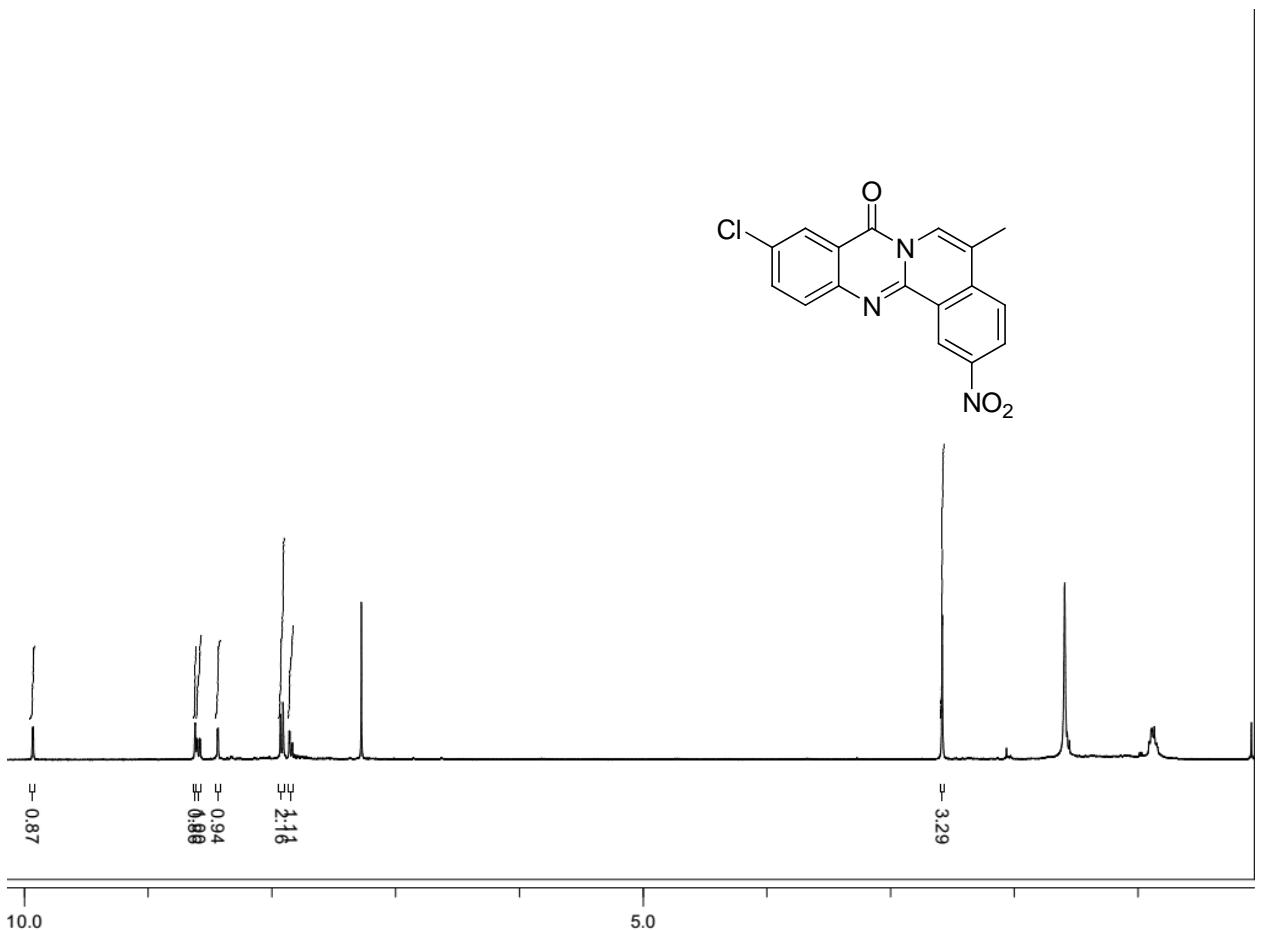


Fig. 48: <sup>1</sup>H NMR spectra of compound **4l** ( $\text{CDCl}_3$ , 400 MHz)

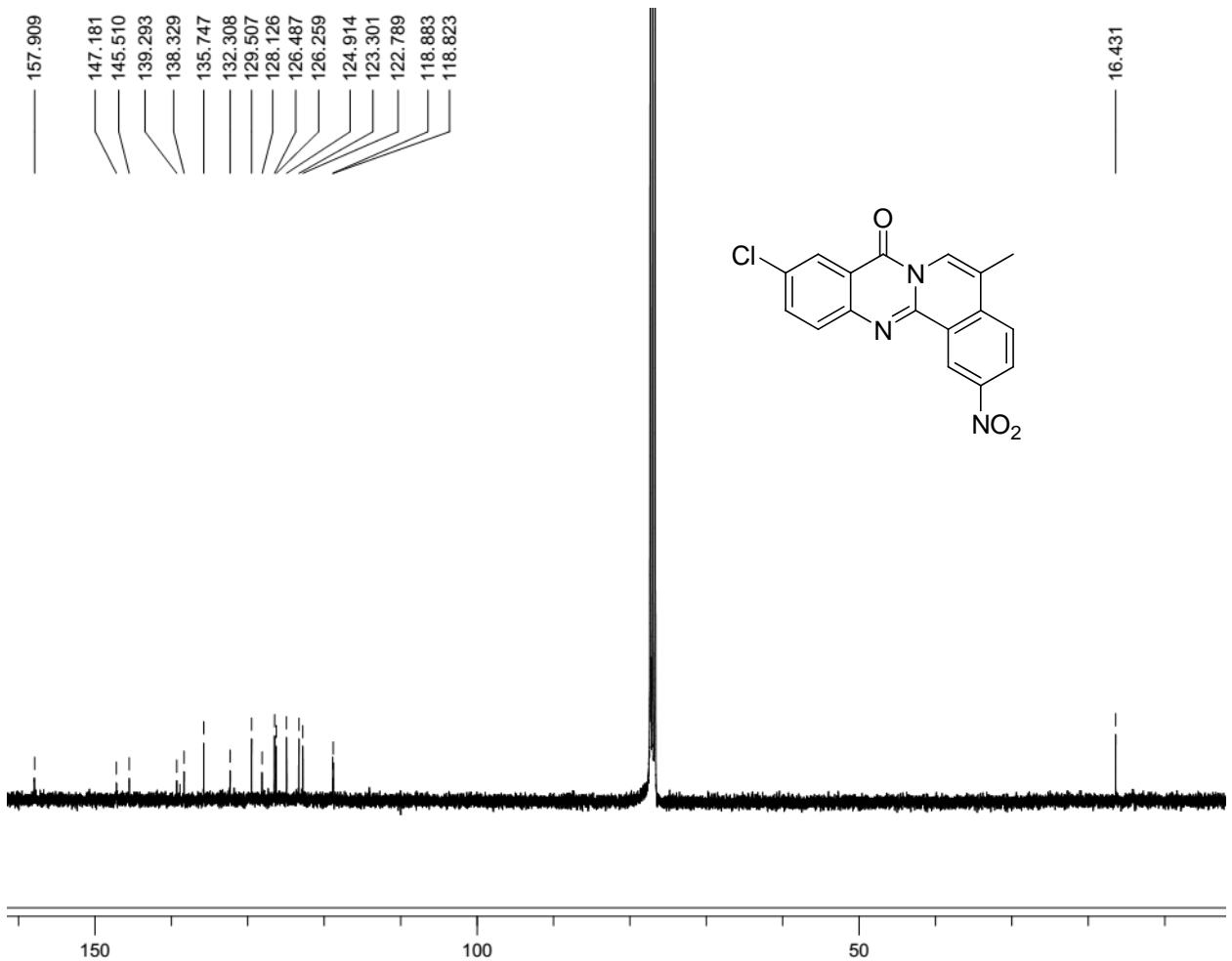
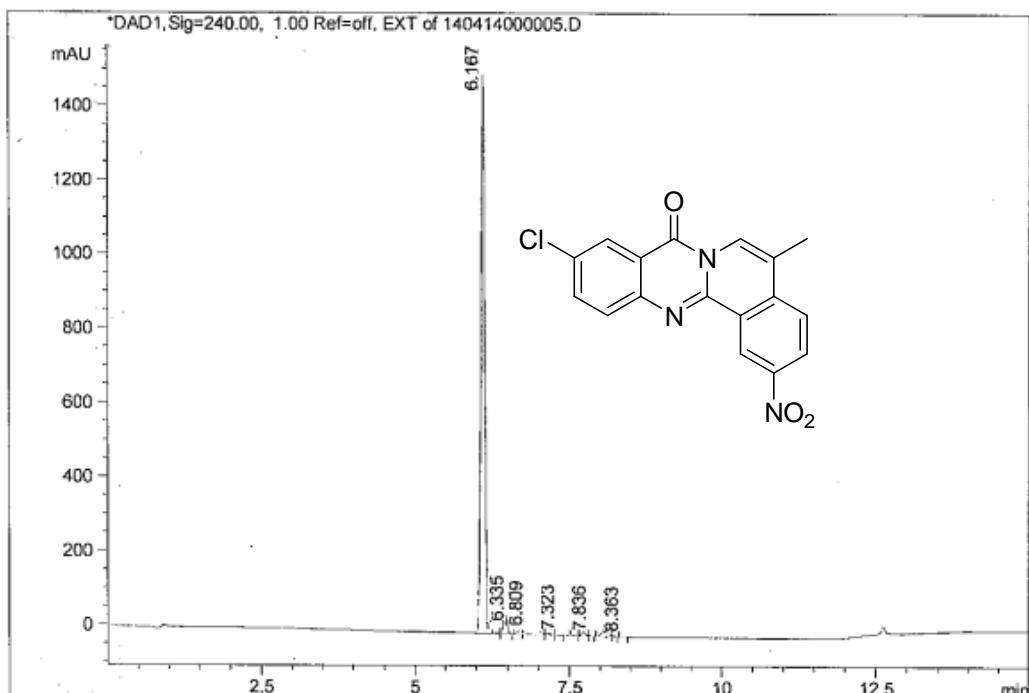


Fig. 49: <sup>13</sup>C NMR spectra of compound **4l** ( $\text{CDCl}_3$ , 100 MHz)

Inj Date : Mon, 14. Apr. 2014                          Acq Operator: SHASHIDHAR  
 Sample Name : ILS/ARJ/5/17                          Vial 3  
 A.R Number : CM14D008                          -> Inj. Vol. : 10 $\mu$ L  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A80B20.M  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18 A80B20.M  
 Method Info : Column : Symmetry C-18 75\*4.6mm3.5 $\mu$ m  
                   Mobile phase: A) 0.1% TFA in water , B) ACN  
                   T/B% : 0/20,1/20,4/98,10/98,10.5/20,12/20.  
                   Flow: 1.0 ml/min Diluent: ACN:Water(80:20)



Signal 1: DAD1, Sig=240.00, 1.00 Ref=off, EXT

Peak #	RT [min]	Area	Area %
1	6.167	6501.815	92.296
2	6.335	9.078	0.128
3	6.464	193.773	2.727
4	6.684	29.836	0.420
5	6.809	178.952	2.518
6	7.164	17.489	0.246
7	7.323	6.065	0.085
8	7.579	7.627	0.107
9	7.730	40.471	0.570
10	7.836	2.209	0.031
11	8.052	66.167	0.931

Fig. 50: HPLC of compound 4l

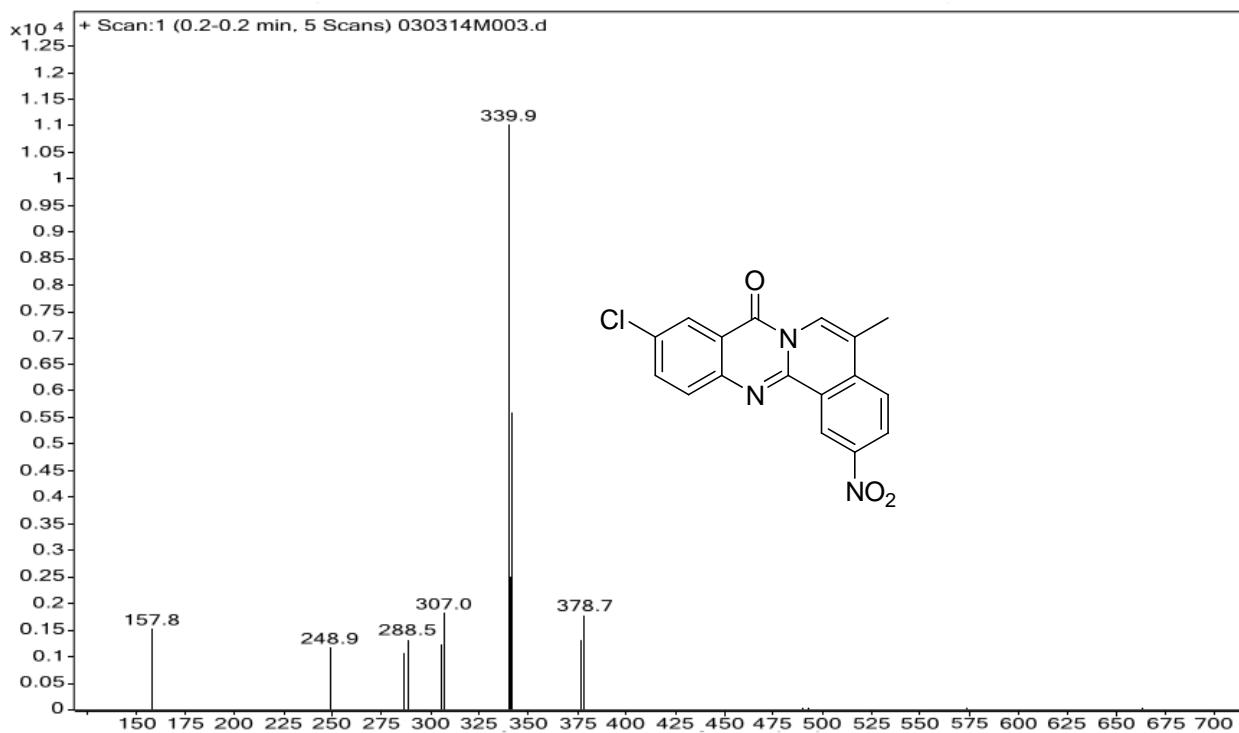


Fig. 51: Mass of compound 4l

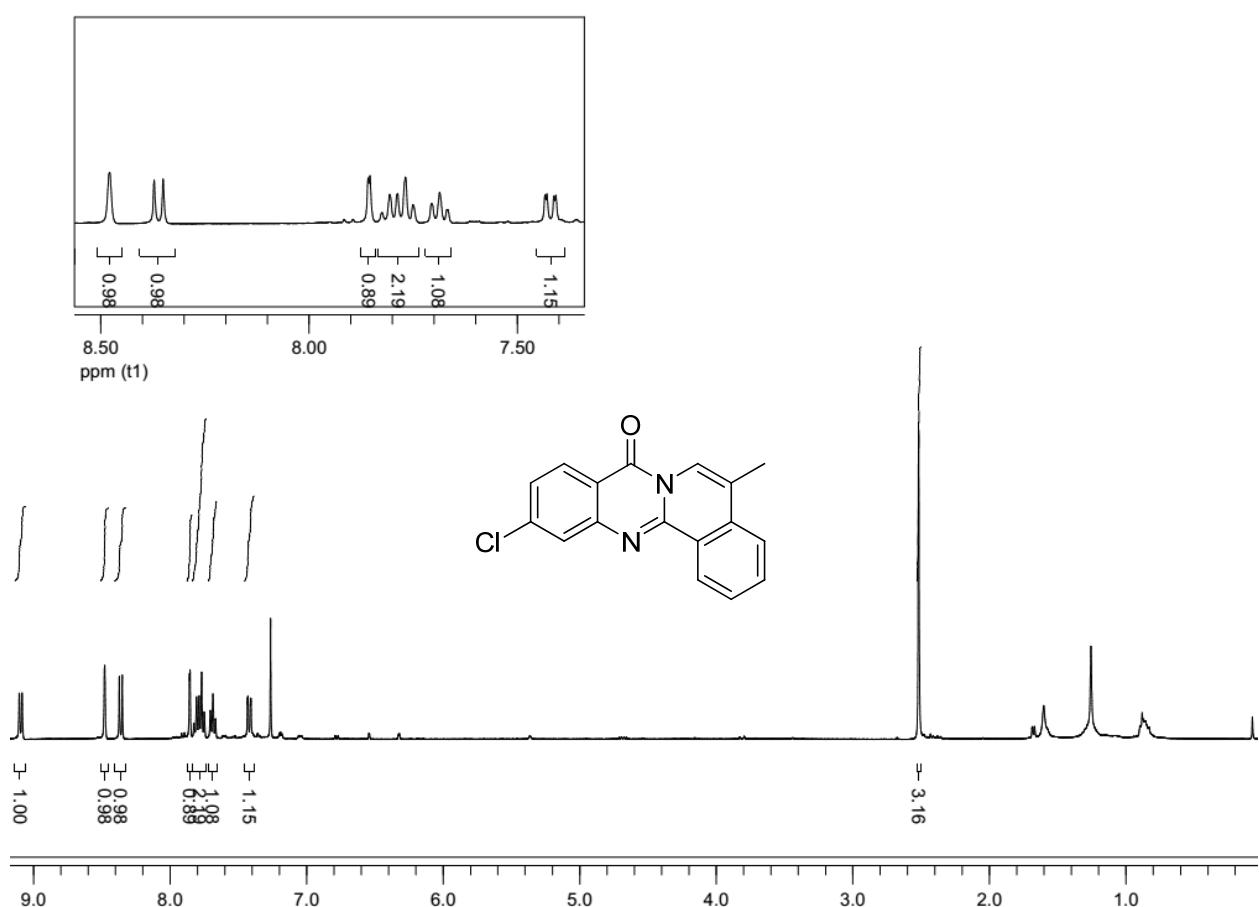


Fig. 52:  $^1\text{H}$  NMR spectra of compound **4m** ( $\text{CDCl}_3$ , 400 MHz)

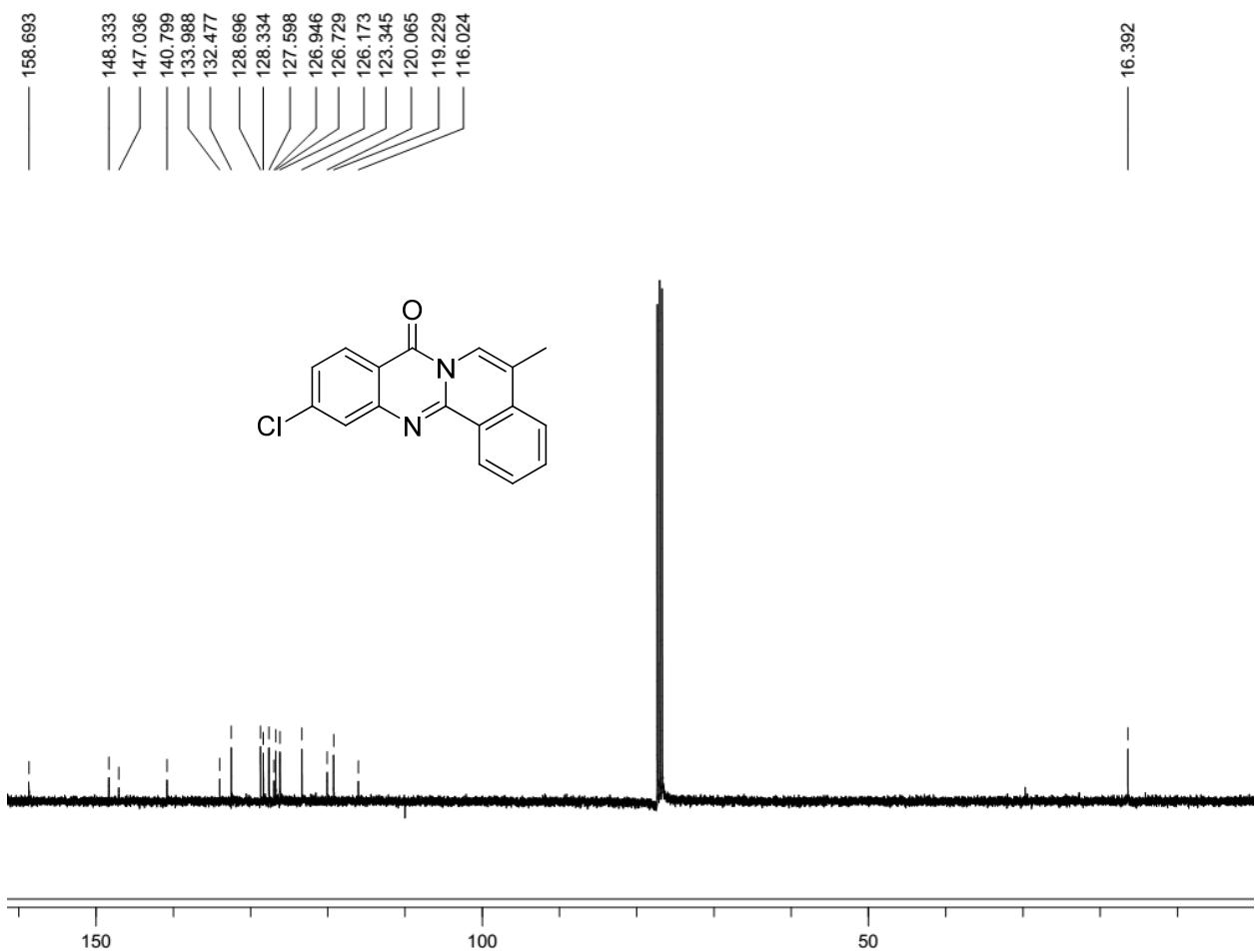
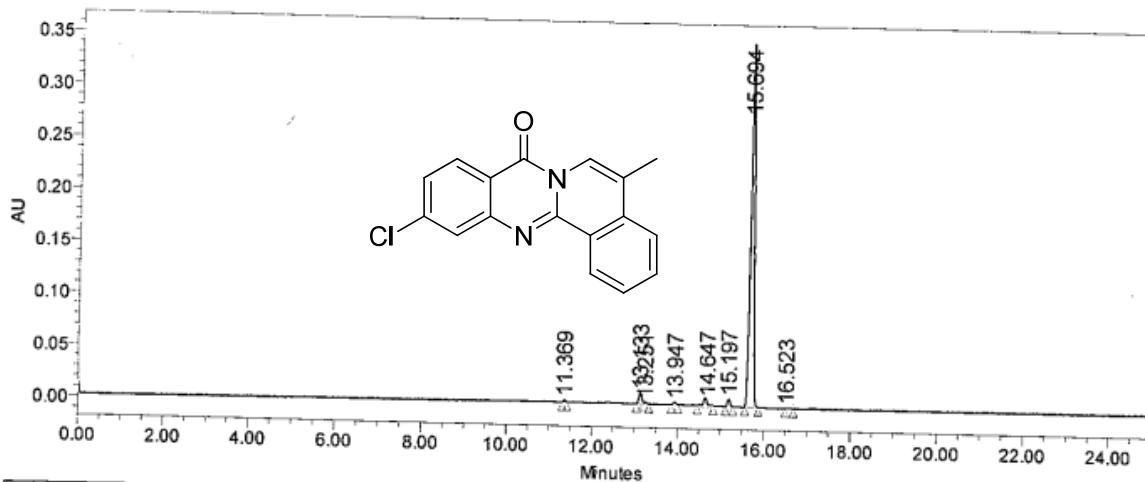


Fig. 53: <sup>13</sup>C NMR spectra of compound **4m** ( $\text{CDCl}_3$ , 100 MHz)

### SAMPLE INFORMATION

Sample Name:	ILS/ARJ/5/4	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	29	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	PDA Max Plot 190.0 - 800.0 @2
Injection Volume:	5.00 $\mu$ l	Proc. Chnl. Descr.:	PDA MaxPlot (190.0 nm to 800.0)
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 8:34:40 PM IST		
Date Processed:	2/24/2014 11:42:25 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	11.369	6594	0.26	2043
2	13.133	48203	1.88	10278
3	13.251	7473	0.29	1724
4	13.947	8836	0.35	2135
5	14.647	31224	1.14	7243
6	15.197	31644	1.24	7308
7	15.694	2434497	95.64	347062
8	16.523	5198	0.20	710

*M. 24/2/14*  
Analysed By

Fig. 54: HPLC of compound 4m

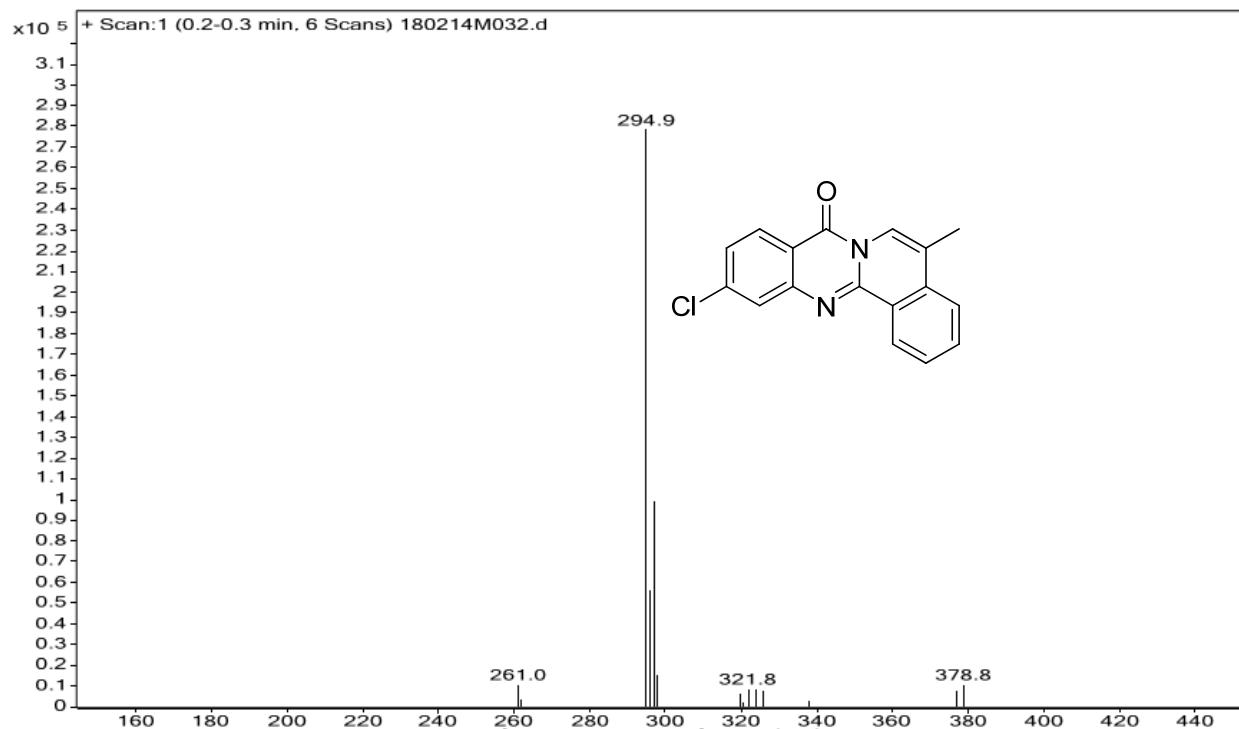


Fig. 55: Mass of compound **4m**

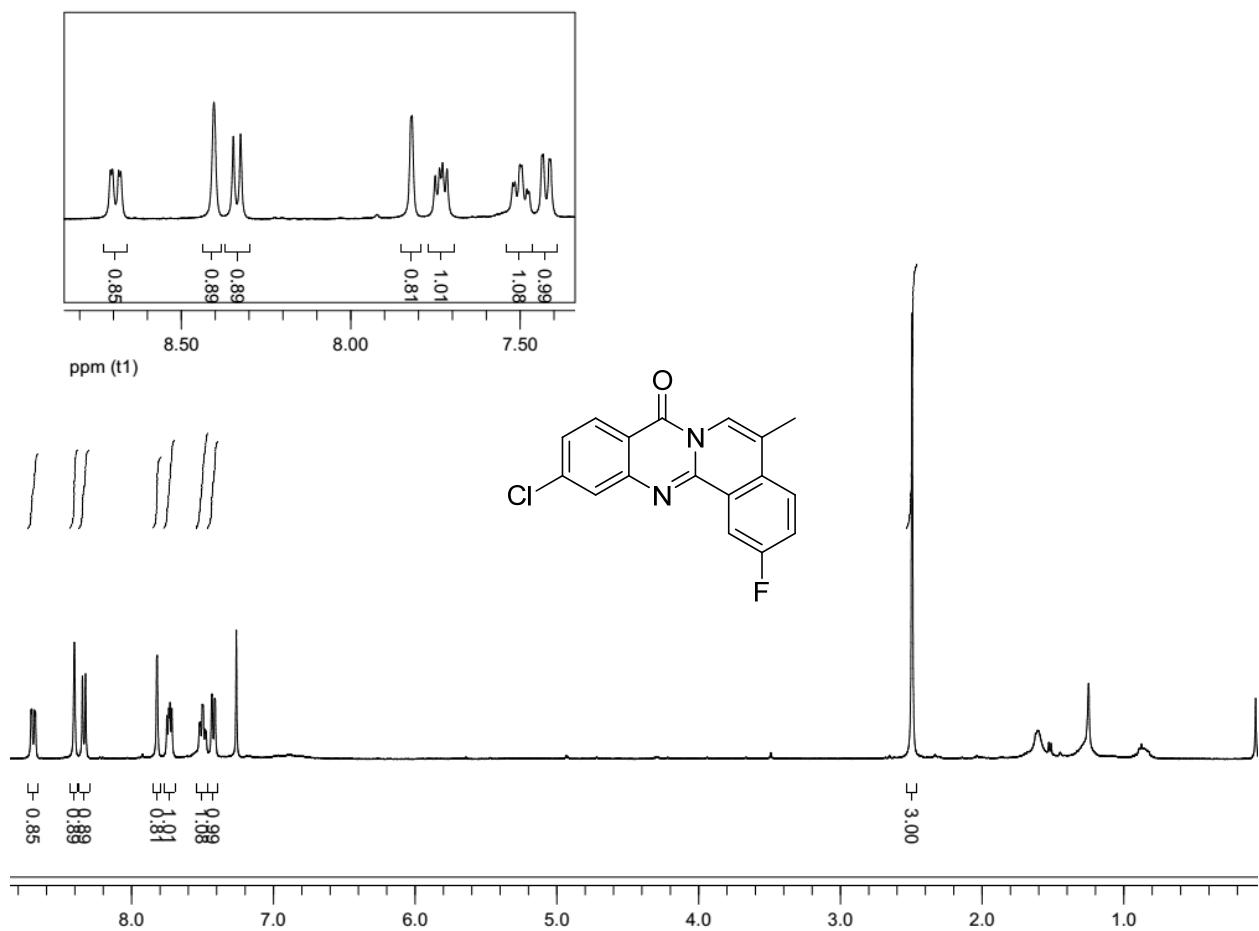


Fig. 56: <sup>1</sup>H NMR spectra of compound **4n** ( $\text{CDCl}_3$ , 400 MHz)

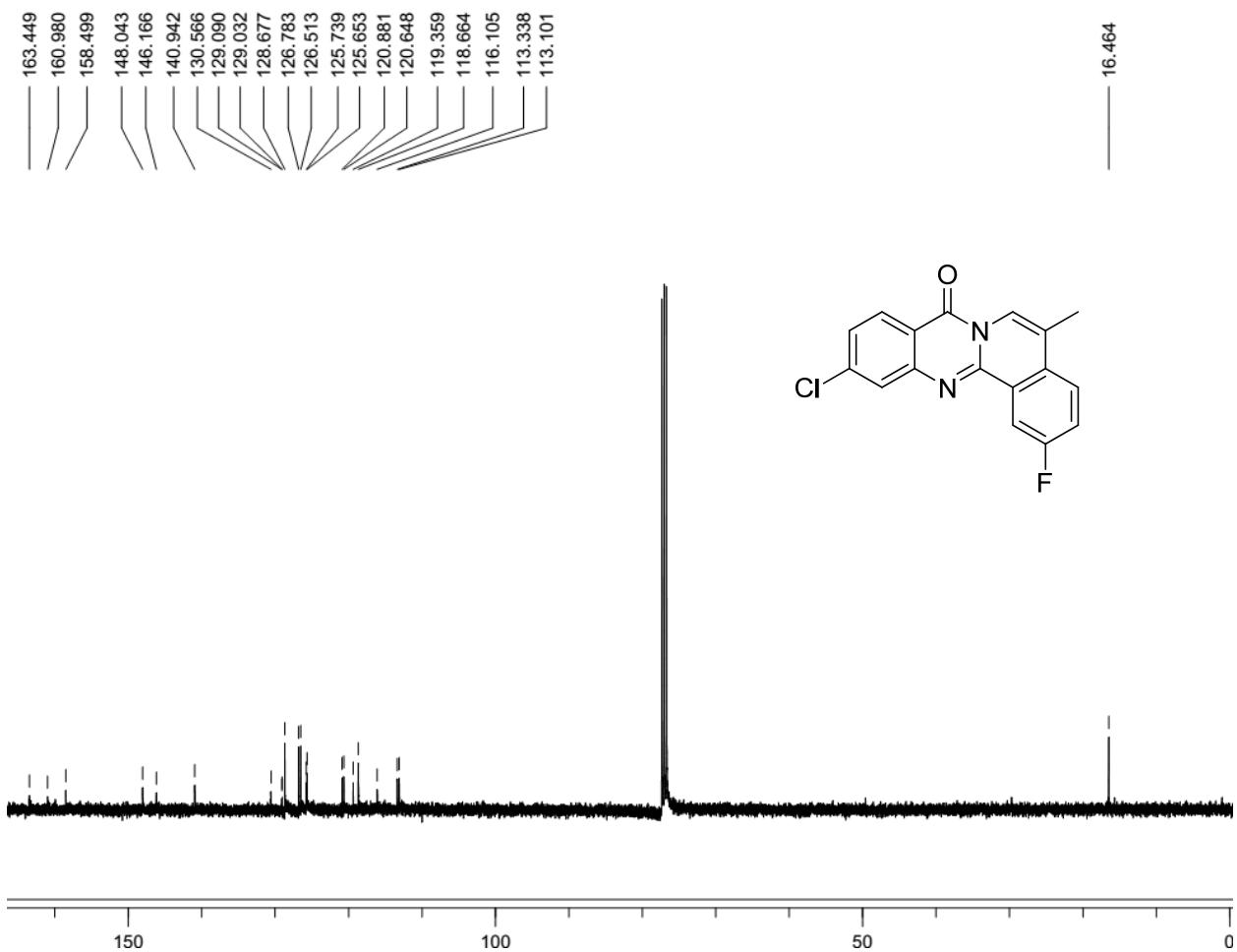
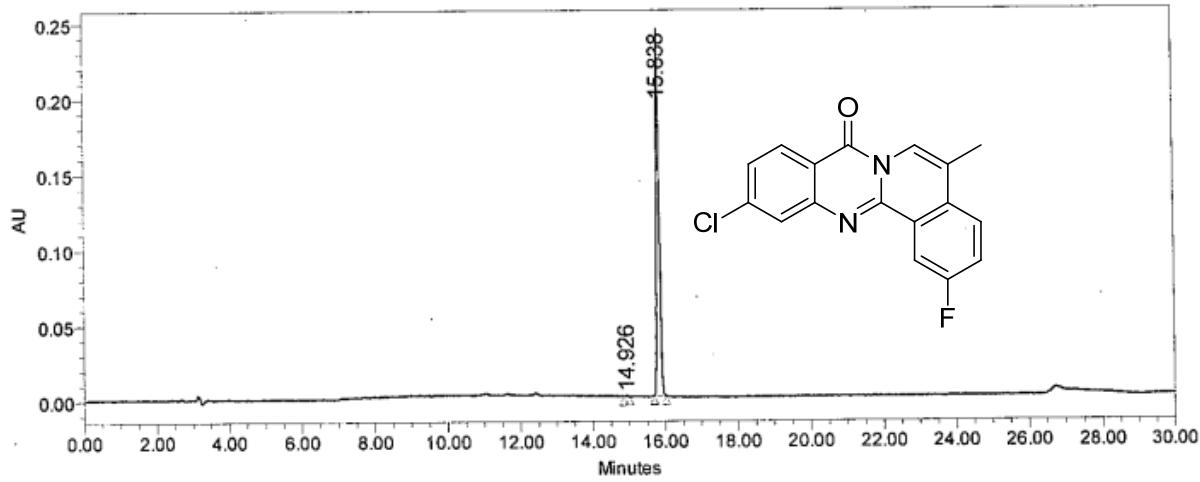


Fig. 57:  $^{13}\text{C}$  NMR spectra of compound **4n** ( $\text{CDCl}_3$ , 100 MHz)

Sample Name:	ILS/ARJ/5/5	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	30	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	285.0nm
Injection Volume:	5.00 ul	Proc. Chnl. Descr.:	PDA 285.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 9:10:24 PM IST		
Date Processed:	2/24/2014 11:43:53 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5μm  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	14.926	5800	0.42	1166
2	15.838	1371486	99.58	243204

M  
Analysed By  
24/02/14

Fig. 58: HPLC of compound 4n

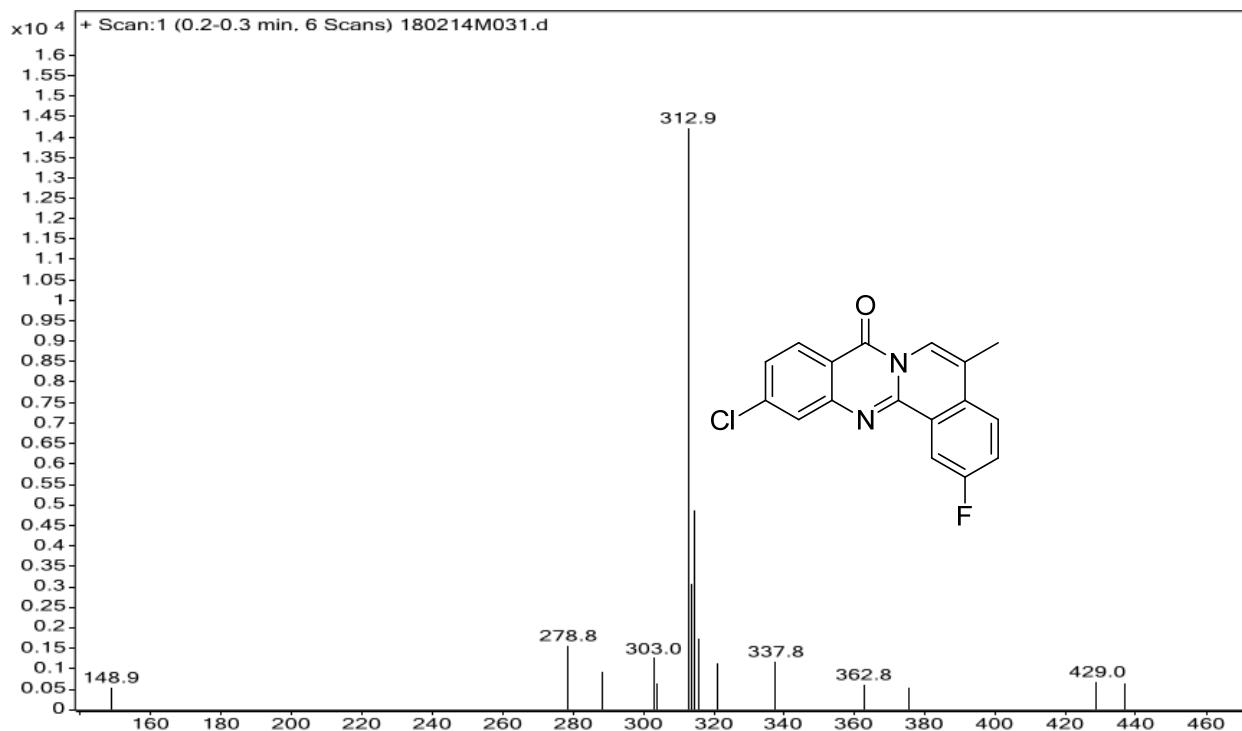


Fig. 59: Mass of compound 4n

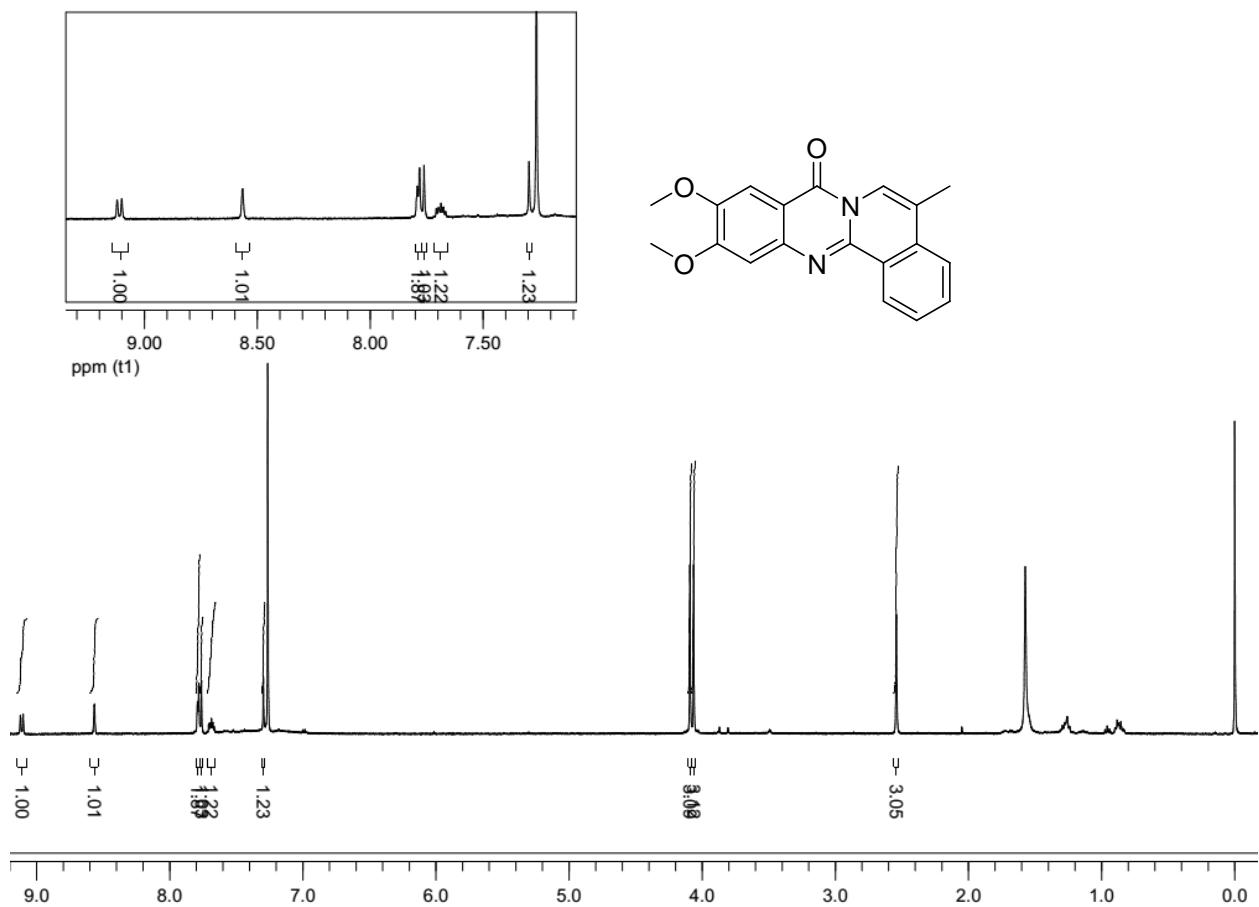


Fig. 60: <sup>1</sup>H NMR spectra of compound **4o** (CDCl<sub>3</sub>, 400 MHz)

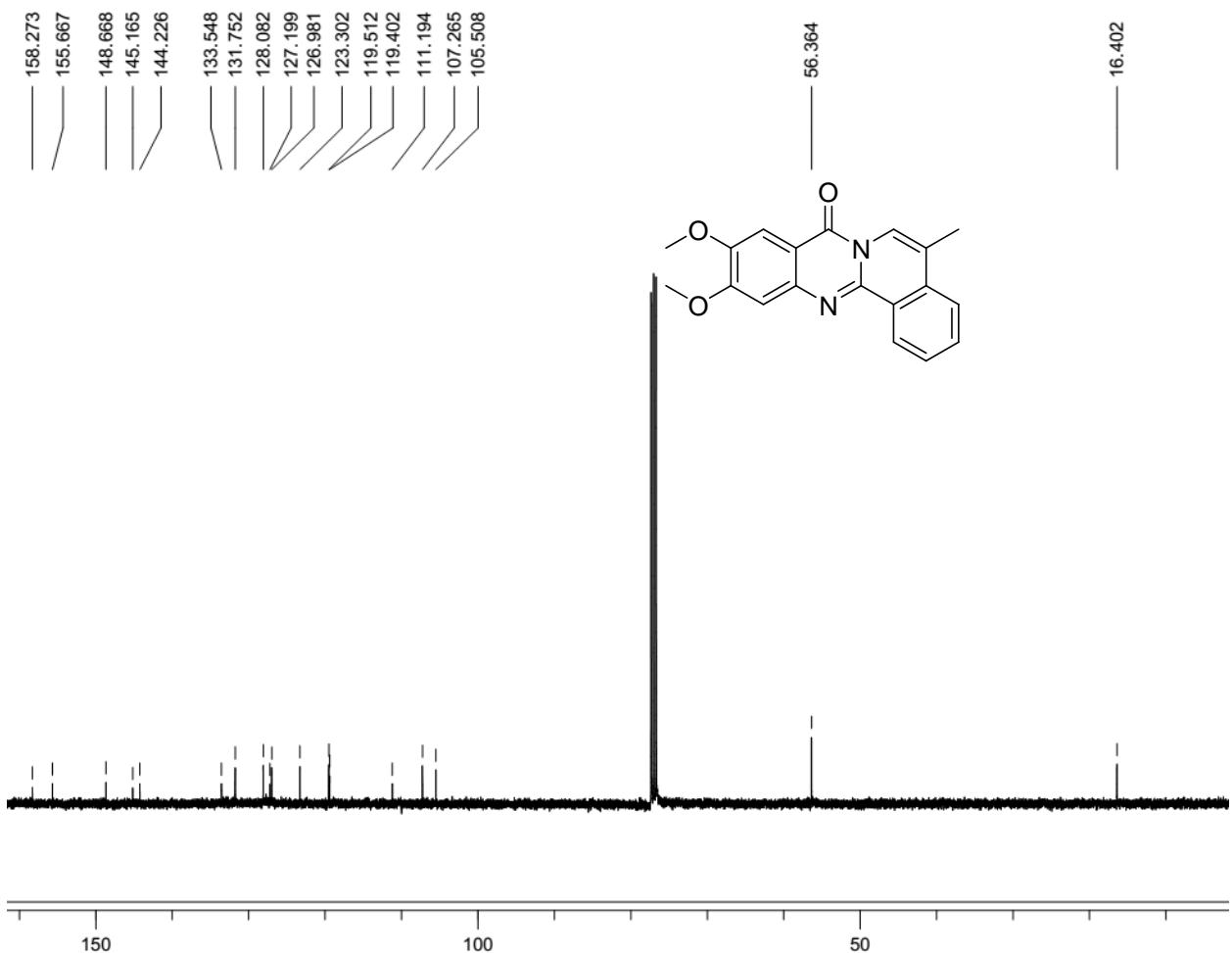
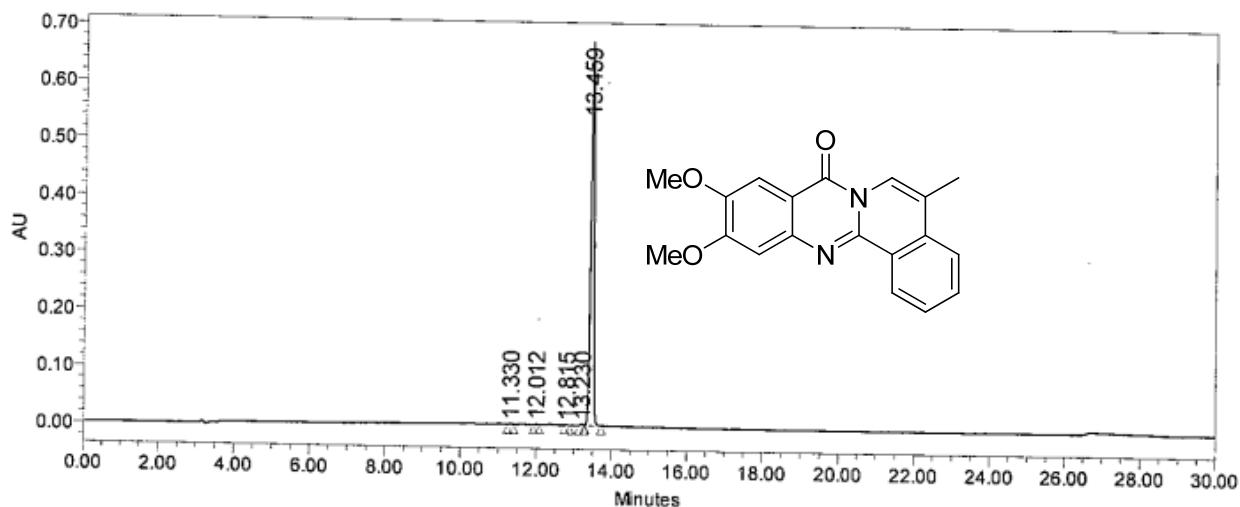


Fig. 61:  $^{13}\text{C}$  NMR spectra of compound **4o** ( $\text{CDCl}_3$ , 100 MHz)

## SAMPLE INFORMATION

Sample Name:	ILS/ARJ/5/6	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	31	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	285.0nm
Injection Volume:	5.00 ul	Proc. Chnl. Descr.:	PDA 285.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 9:46:05 PM IST		
Date Processed:	2/24/2014 11:45:55 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	11.330	9221	0.23	1936
2	12.012	8144	0.21	1652
3	12.815	5267	0.13	1257
4	13.230	12278	0.31	2533
5	13.459	3903991	99.11	670715

*M*  
Analysed by  
24/02/14

Fig. 62: HPLC of compound 4o

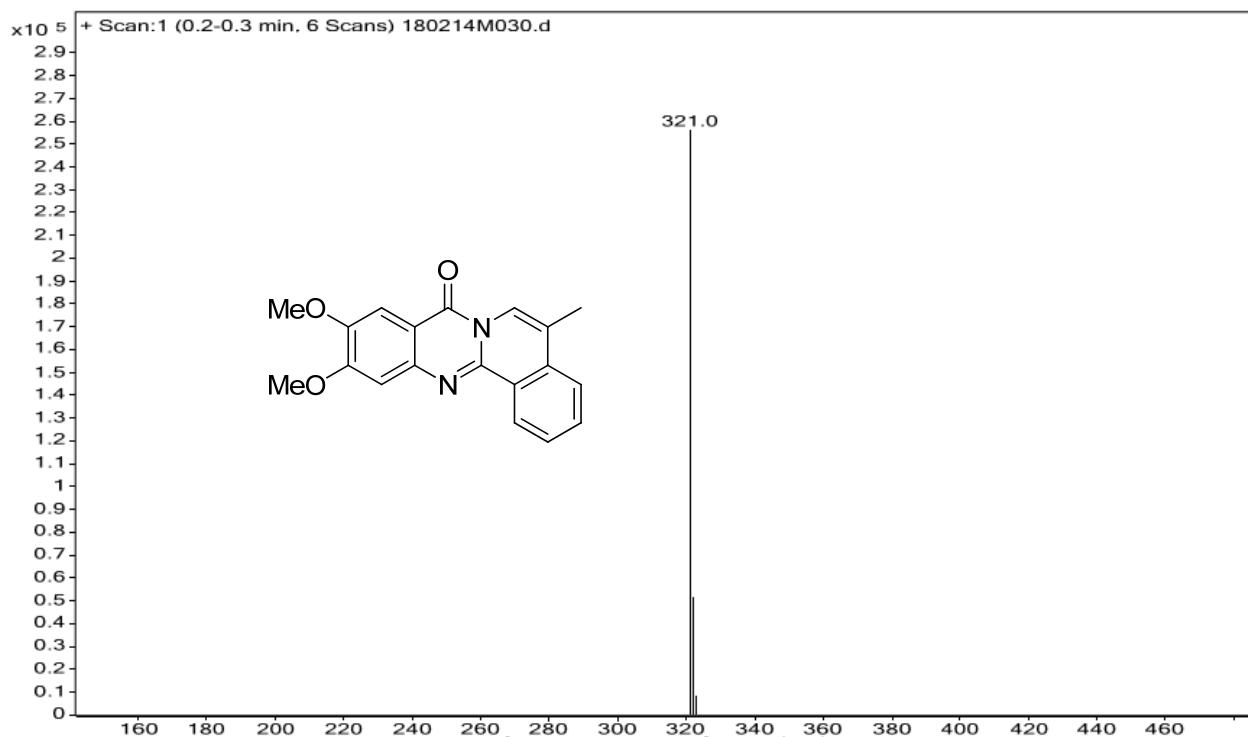


Fig. 63: Mass of compound **4o**

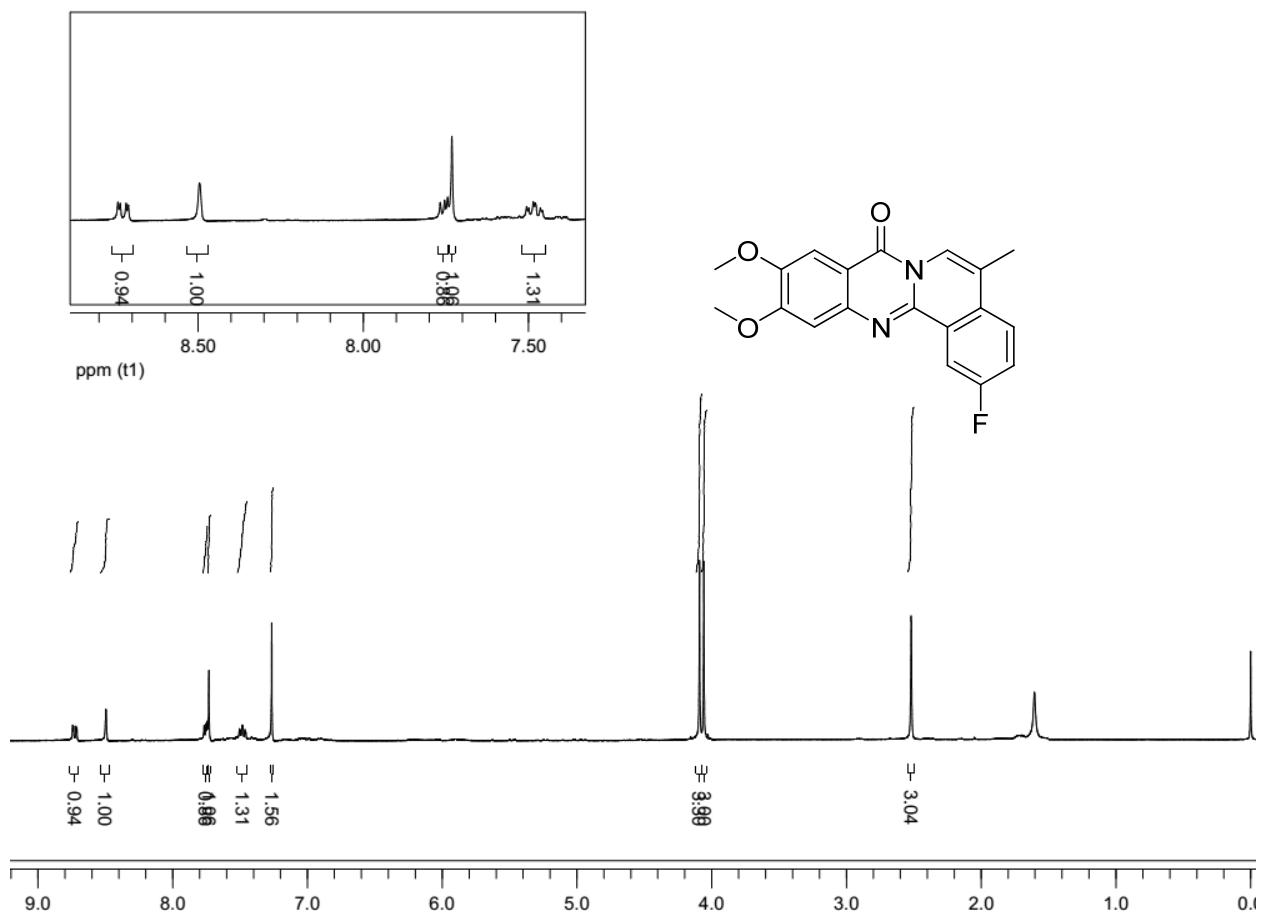


Fig. 64: <sup>1</sup>H NMR spectra of compound **4p** ( $\text{CDCl}_3$ , 400 MHz)

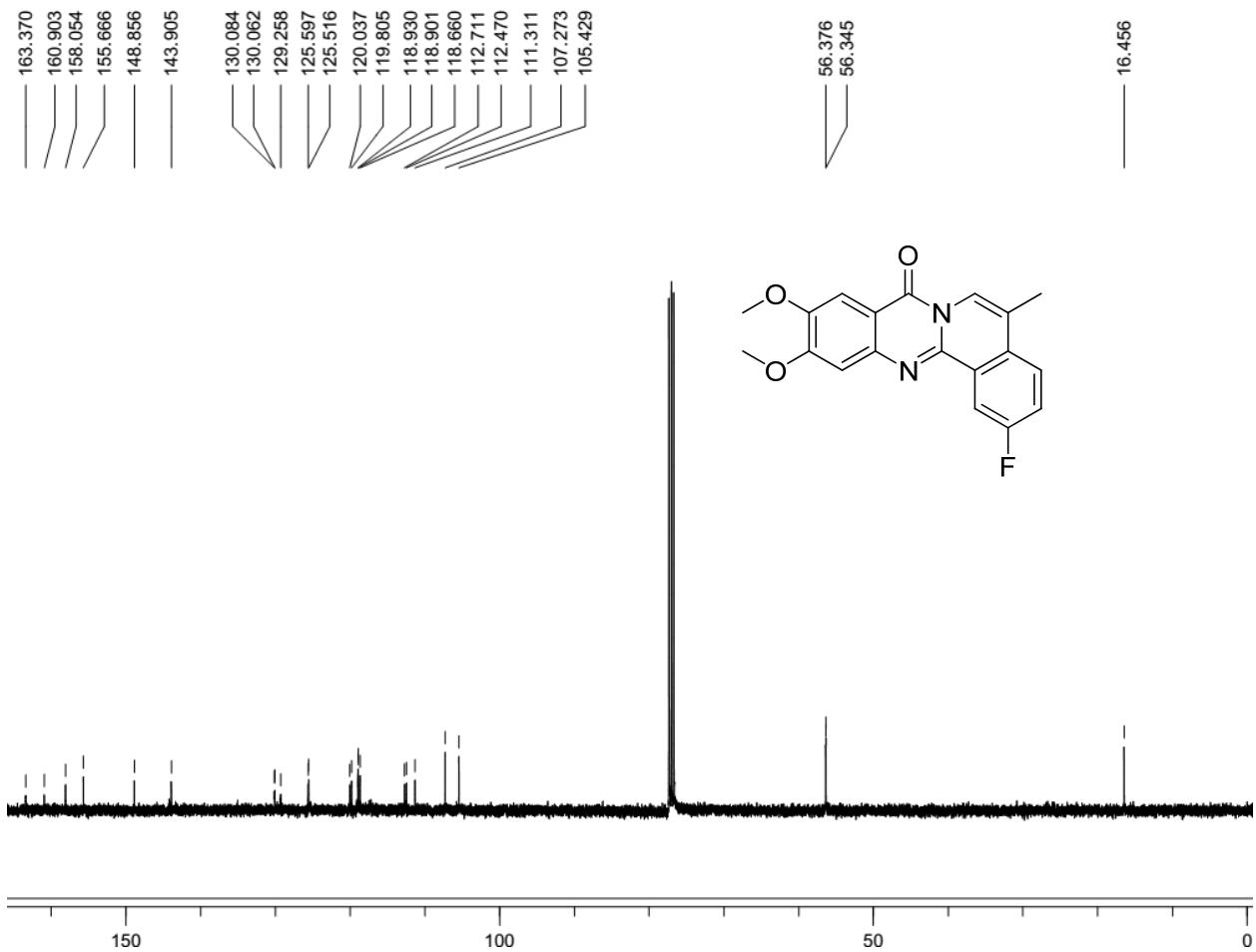
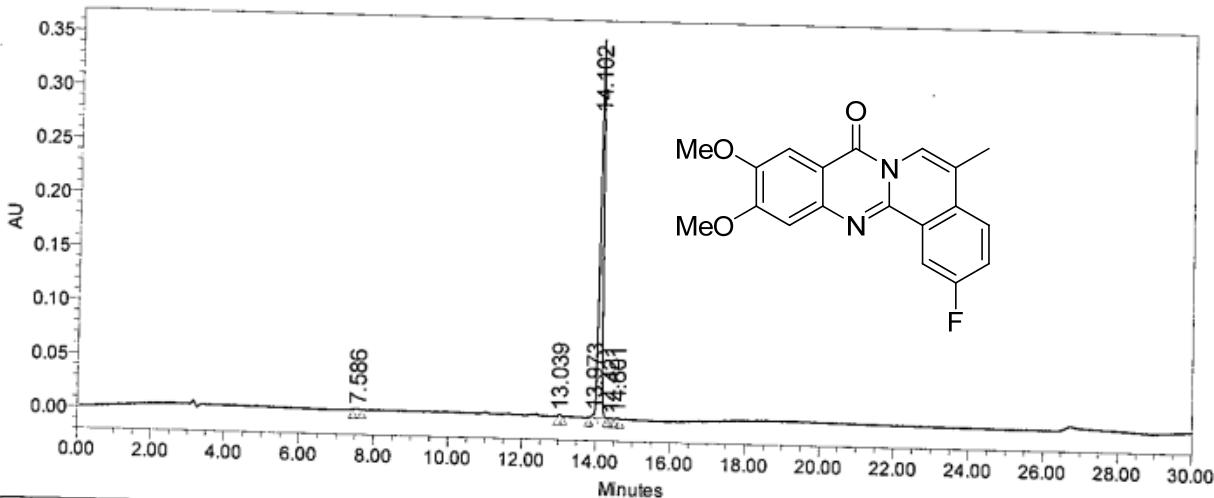


Fig. 65: <sup>13</sup>C NMR spectra of compound **4p** ( $\text{CDCl}_3$ , 100 MHz)

**SAMPLE INFORMATION**

Sample Name:	ILS/ARJ/5/8	Sample Set Name:	210214_1
Sample Type:	Unknown	Acq. Method Set:	CFZ
Vial:	33	Processing Method:	CFZ_PRO
Injection #:	1	Channel Name:	280.0nm
Injection Volume:	5.00 $\mu$ l	Proc. Chnl. Descr.:	PDA 280.0 nm
Run Time:	30.0 Minutes		
Date Acquired:	2/21/2014 10:57:31 PM IST		
Date Processed:	2/24/2014 11:49:42 AM IST		

Column: X TERRA RP-18 250\*4.6mm 5 $\mu$ m  
 Mobile phase: A) 0.1% TFA in water B) ACN  
 T/%B: 0/20, 3/20, 12/95, 23/95, 25/20, 30/20  
 Flow: 1.0 ml /min, Diluent: ACN: WATER (80:20)



	RT	Area	% Area	Height
1	7.586	9268	0.45	1179
2	13.039	10391	0.51	2108
3	13.973	14424	0.71	3361
4	14.102	1996801	97.91	350689
5	14.421	2820	0.14	779
6	14.601	5752	0.28	1289

*M. 20/02/14*  
Analysed By

Fig. 66: HPLC of compound 4p

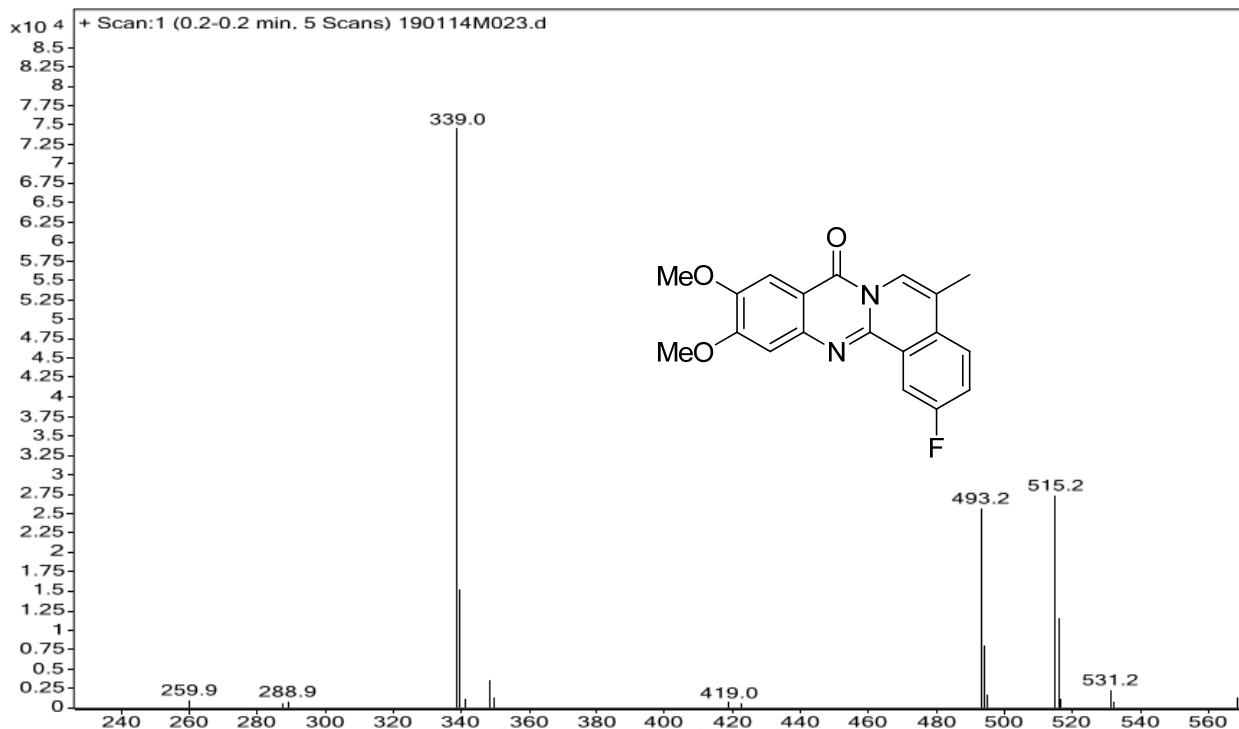


Fig. 67: Mass of compound 4p

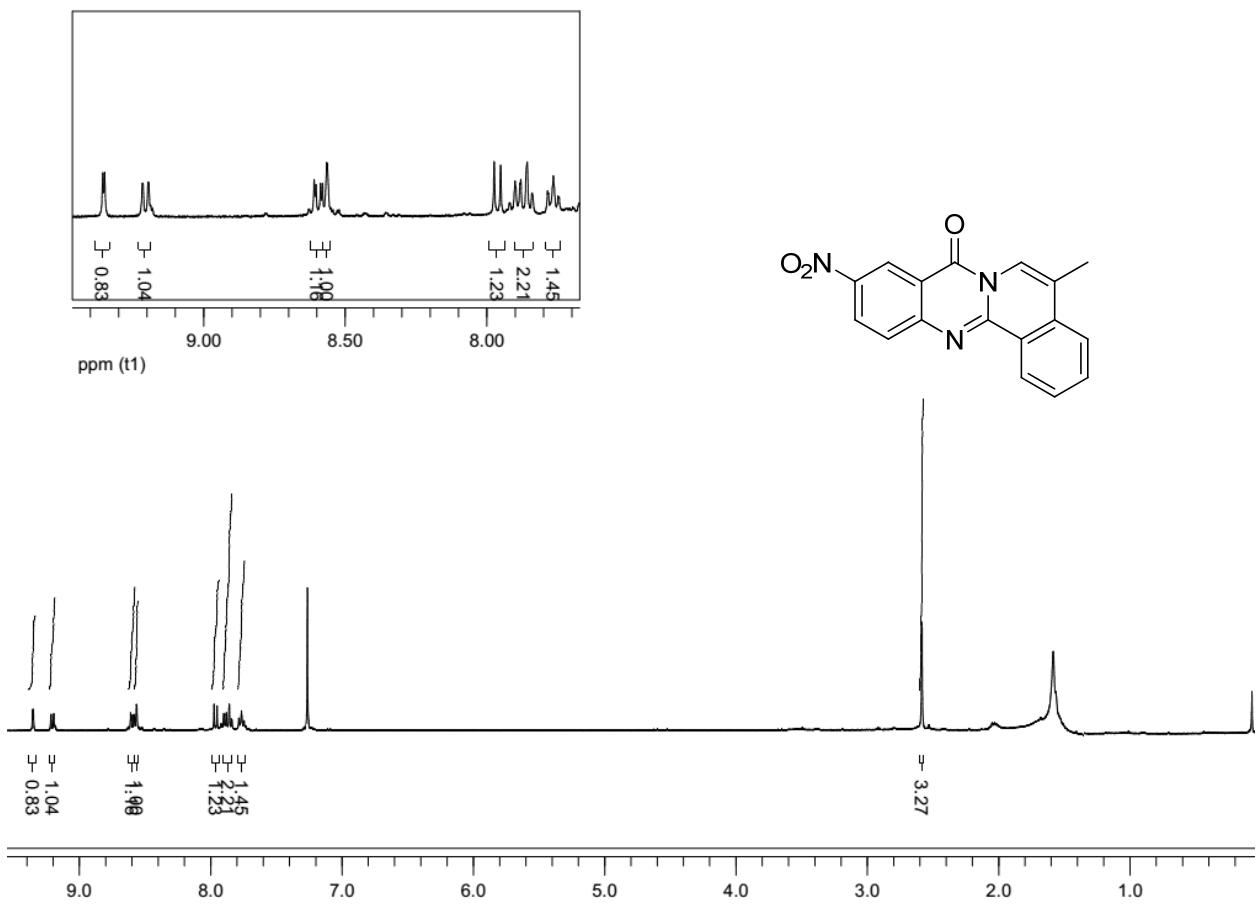


Fig. 68: <sup>1</sup>H NMR spectra of compound **4q** ( $\text{CDCl}_3$ , 400 MHz)

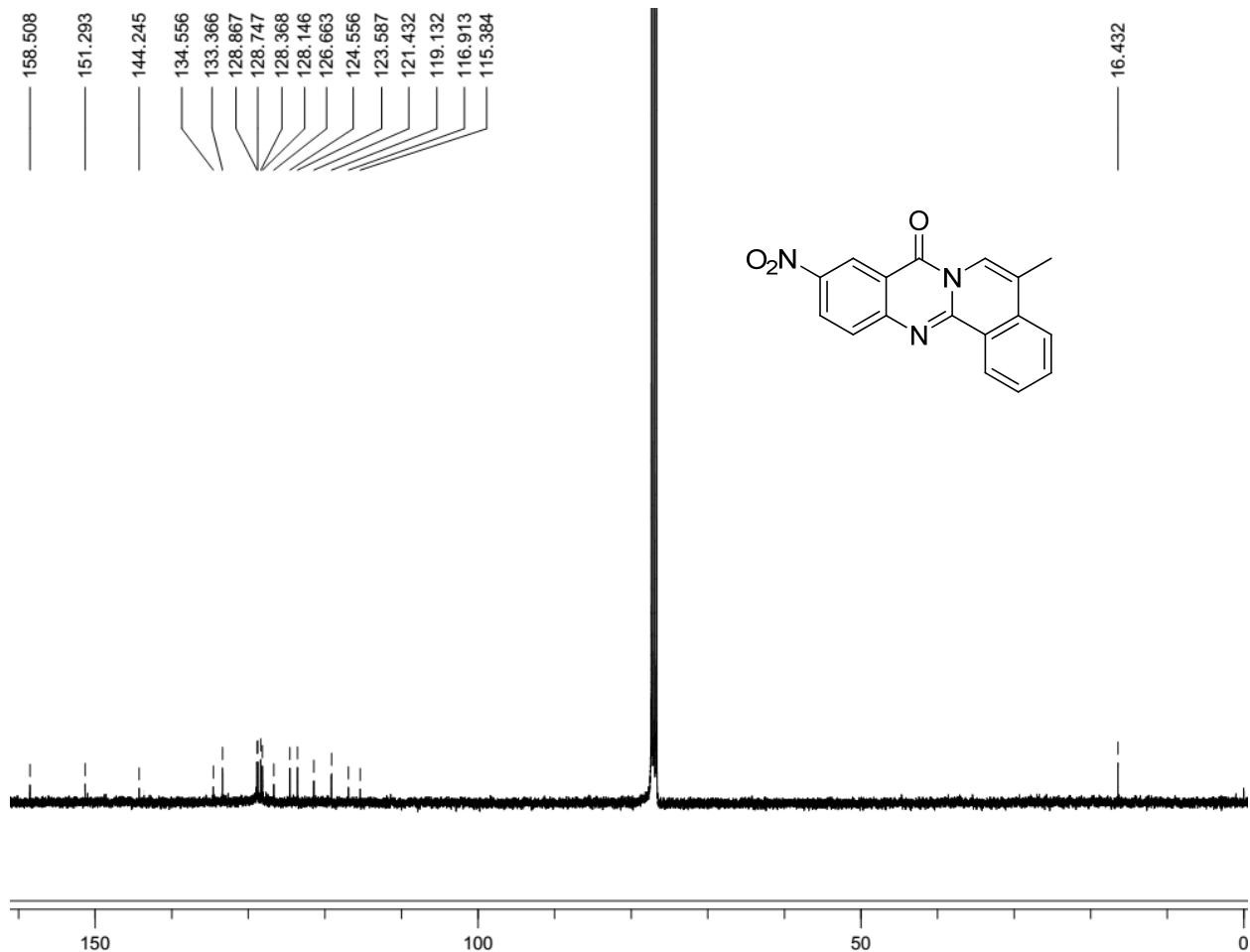
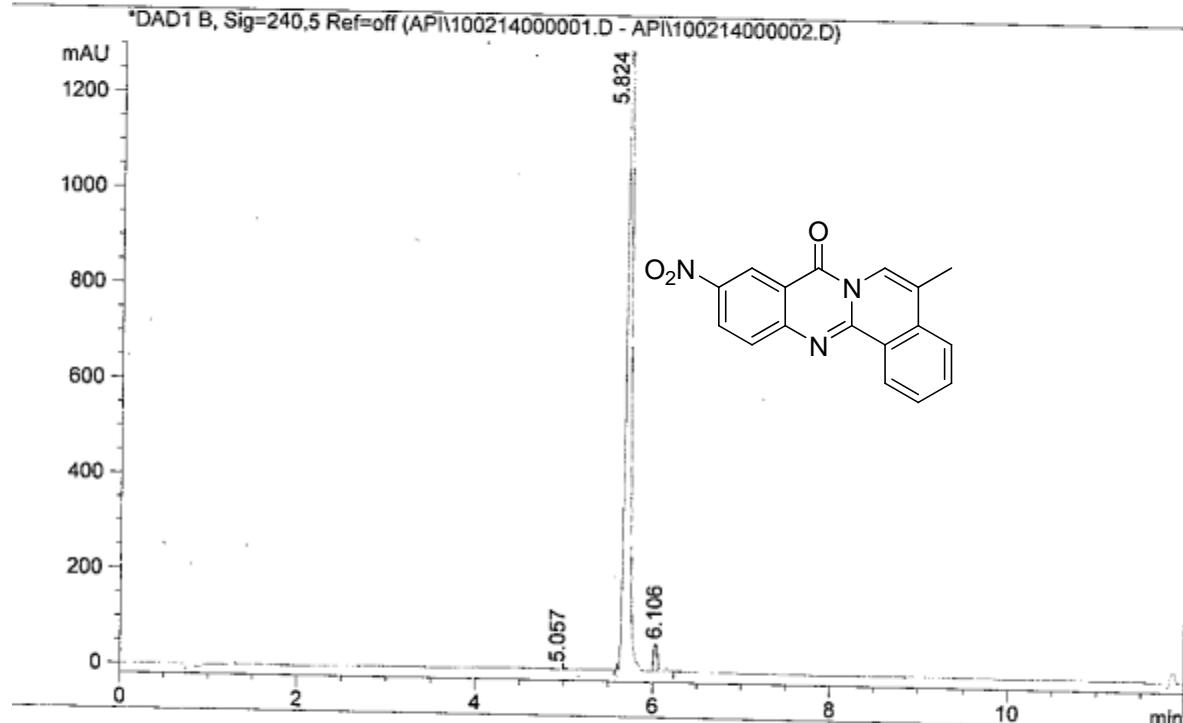


Fig. 69: <sup>13</sup>C NMR spectra of compound **4q** ( $\text{CDCl}_3$ , 100 MHz)

Injection Date : Mon, 10. Feb. 2014 Seq Line : 0  
 Sample Name : ILS-BPS-3-171 Location : Vial 1  
 Acq Operator : RADHA Inj. No. : 0  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A80B20GS.M Inj. Vol. : 15  $\mu$ l  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18 A80B20GS.M  
 Method Info : Column : Symmetry C-18 75\*4.6mm, 3.5 $\mu$ m  
 Mobile phase: A) 0.1% HCOOH in Water , B) ACN  
 T/B%:0/20, 0.5/20, 4/98, 10/98, 10.5/20, 12/20  
 Flow: 1.0 ml/min, Diluent: ACN:Water(80:20)



Signal 1: DAD1 B, Sig=240,5 Ref=off

Peak	RT [min]	Area	Area %
1	5.057	6.209	0.110
2	5.824	5350.146	94.165
3	6.106	280.288	5.724

\*\*\* End of Report \*\*\*

✓  
10/02/

Fig. 70: HPLC of compound 4q

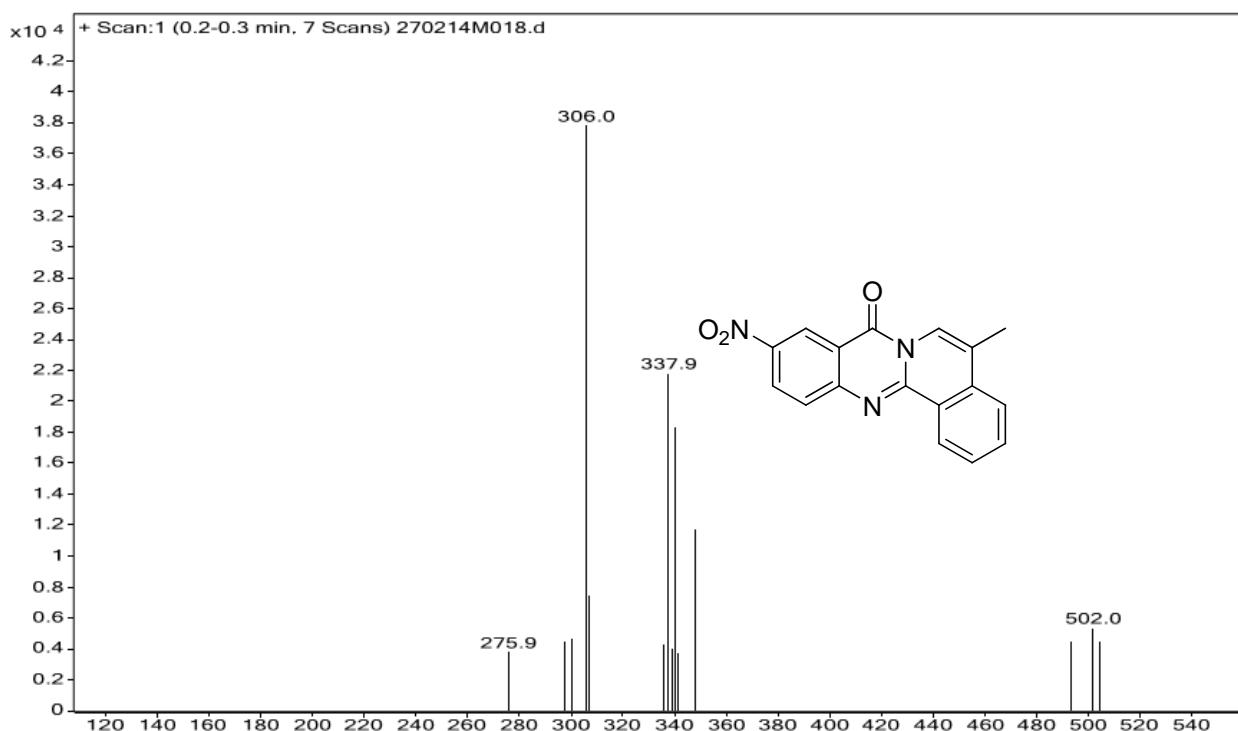


Fig. 71: Mass of compound 4q

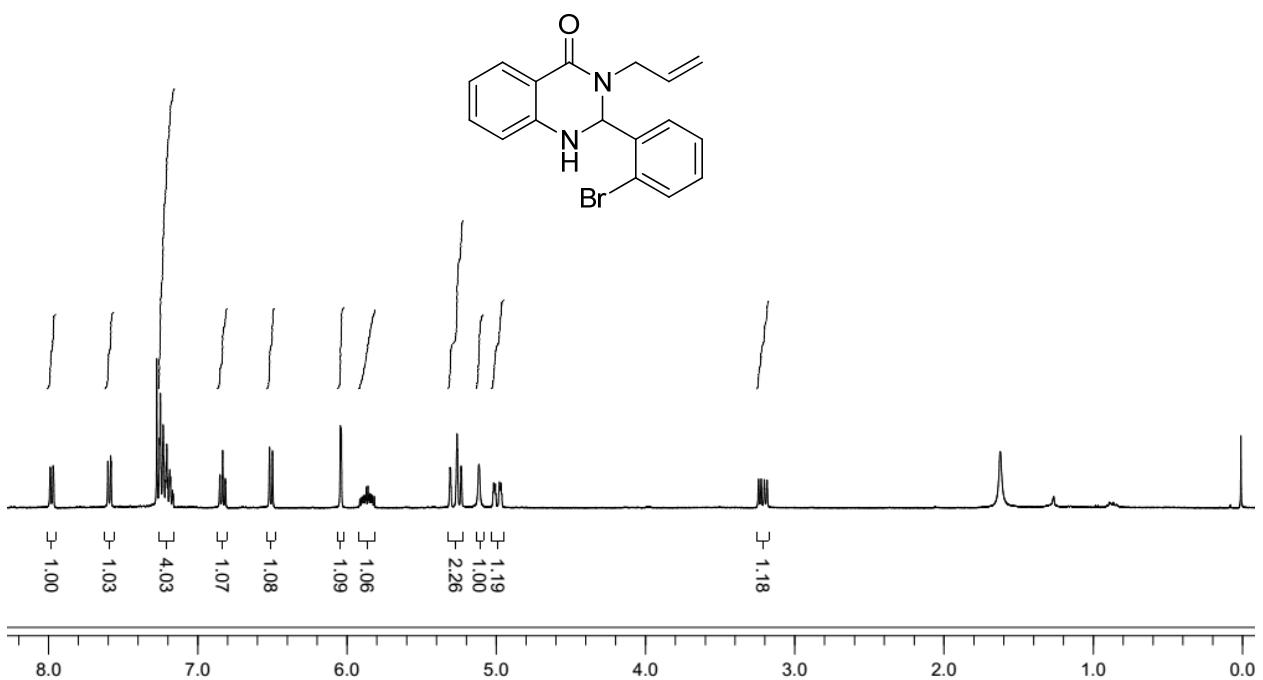


Fig. 72:  $^1\text{H}$  NMR spectra of compound 5 ( $\text{CDCl}_3$ , 400 MHz)

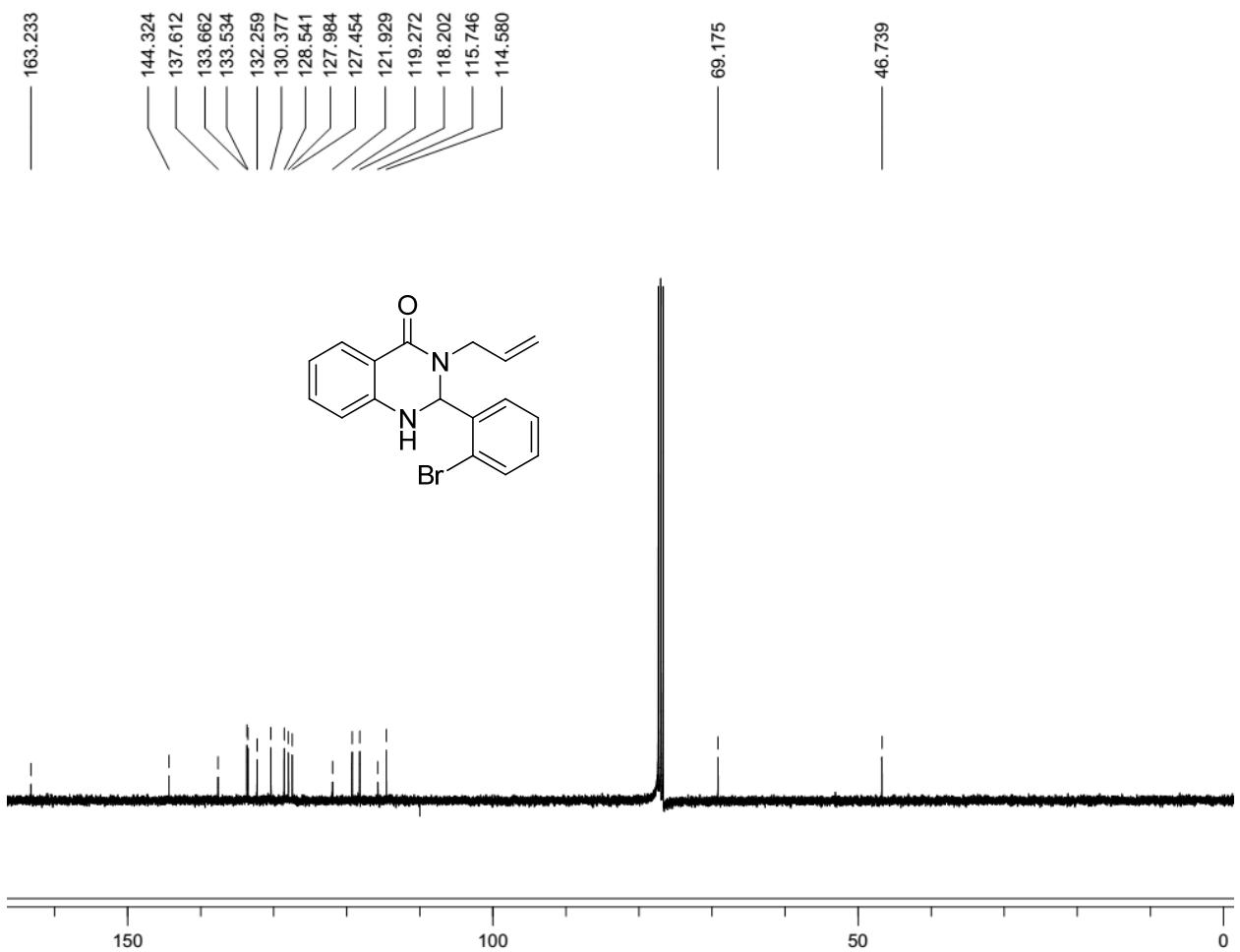


Fig. 73:  $^{13}\text{C}$  NMR spectra of compound **5** ( $\text{CDCl}_3$ , 100 MHz)

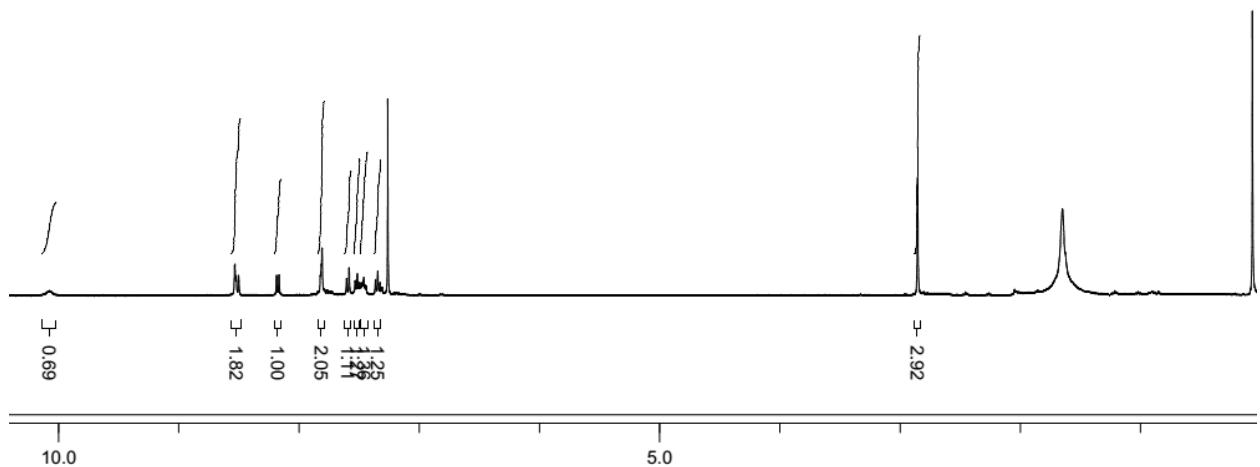
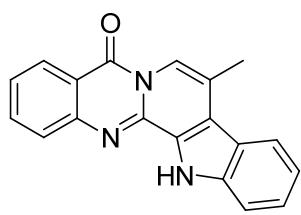


Fig. 74: <sup>1</sup>H NMR spectra of compound 6 (CDCl<sub>3</sub>, 400 MHz)

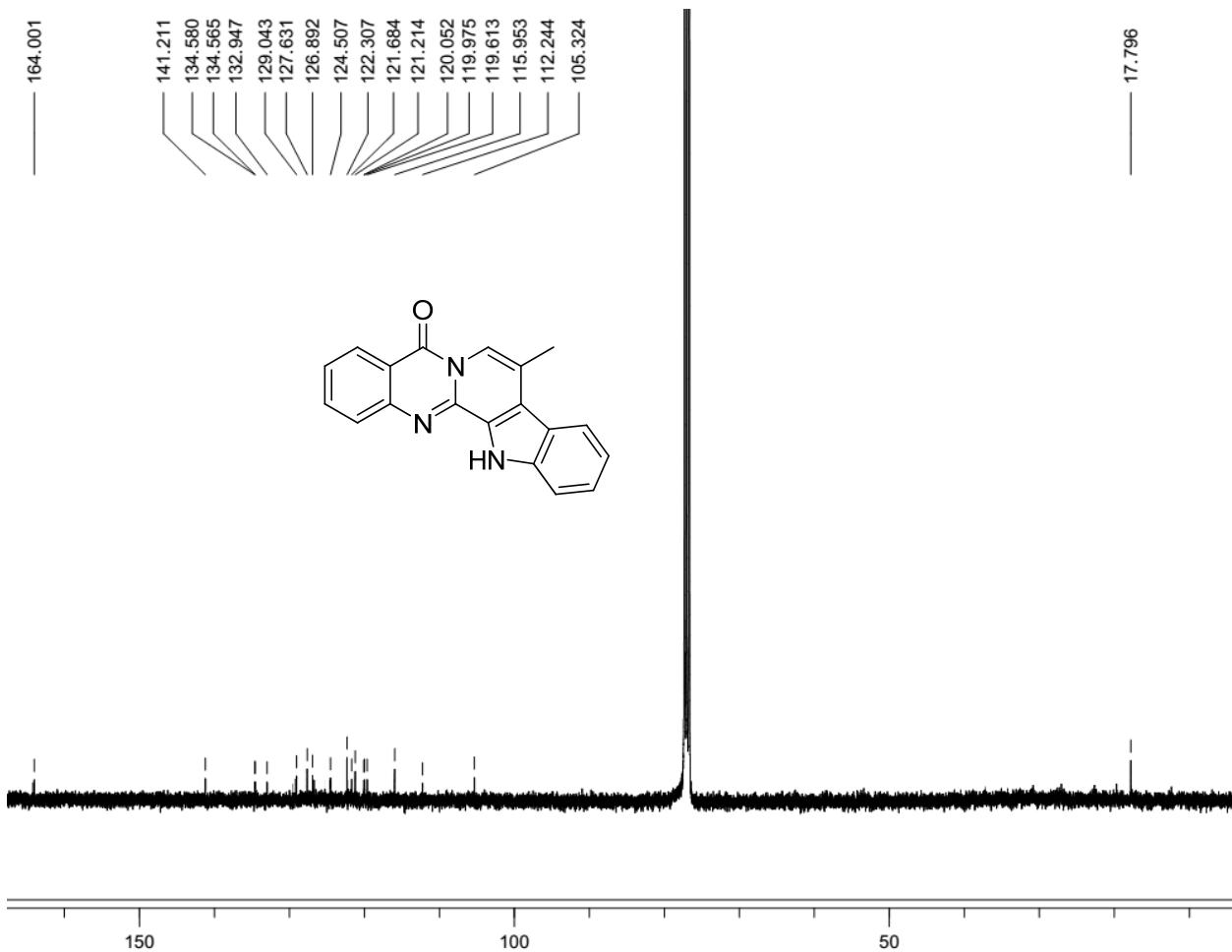
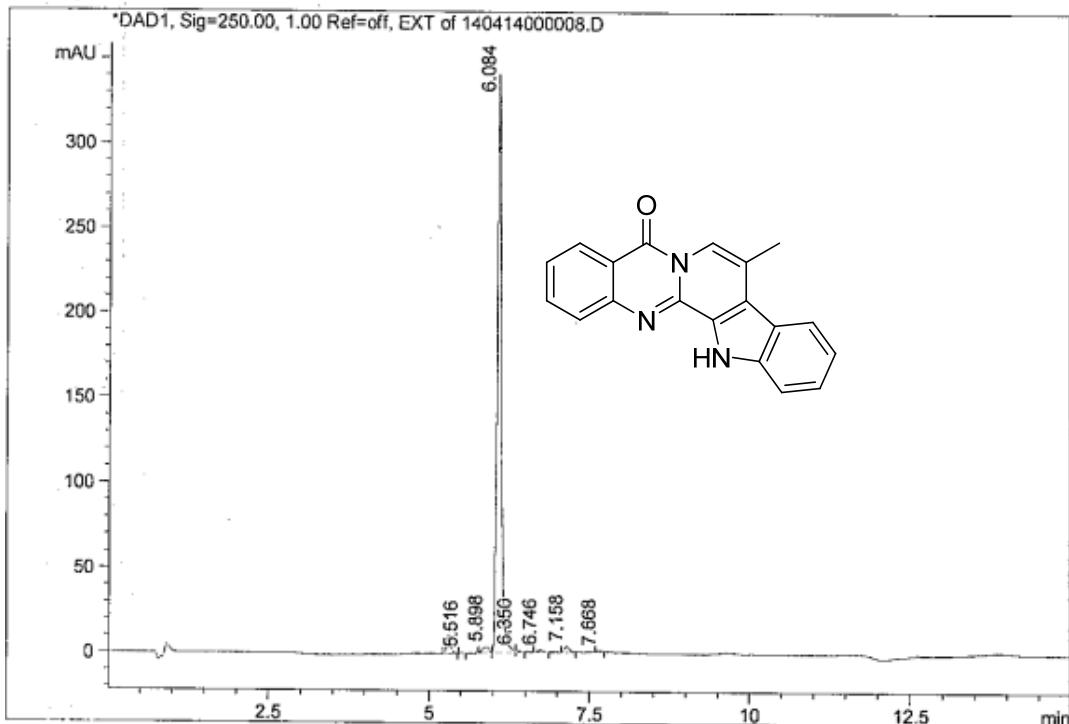


Fig. 75:  $^{13}\text{C}$  NMR spectra of compound **6** ( $\text{CDCl}_3$ , 100 MHz)

Inj Date : Mon, 14. Apr. 2014 Acq Operator: SHASHIDHAR  
 Sample Name : ILS/ARJ/5/26 Vial 5  
 A.R Number : CM14D010 -> Inj. Vol. : 10µL  
 Acq. Method : D:\CHEM32\_002\1\METHODS\C-18 A80B20.M  
 Analysis Method : D:\CHEM32\_002\1\METHODS\C-18 A80B20.M  
 Method Info : Column : Symmetry C-18 75\*4.6mm3.5µm  
                  Mobile phase: A) 0.1% TFA in water , B) ACN  
                  T/B% : 0/20,1/20,6/98,10/98,12/20,15/20.  
                  Flow: 1.0 ml/min Diluent: ACN:Water(80:20)



Signal 1: DAD1, Sig=250.00, 1.00 Ref=off, EXT

Peak	RT	Area	Area %
#	[min]		
1	5.308	47.263	2.778
2	5.516	1.210	0.071
3	5.898	21.233	1.248
4	6.084	1601.773	94.132
5	6.350	3.514	0.207
6	6.746	7.196	0.423
7	7.158	16.680	0.980
8	7.668	2.752	0.162

Fig. 76: HPLC of compound **6**

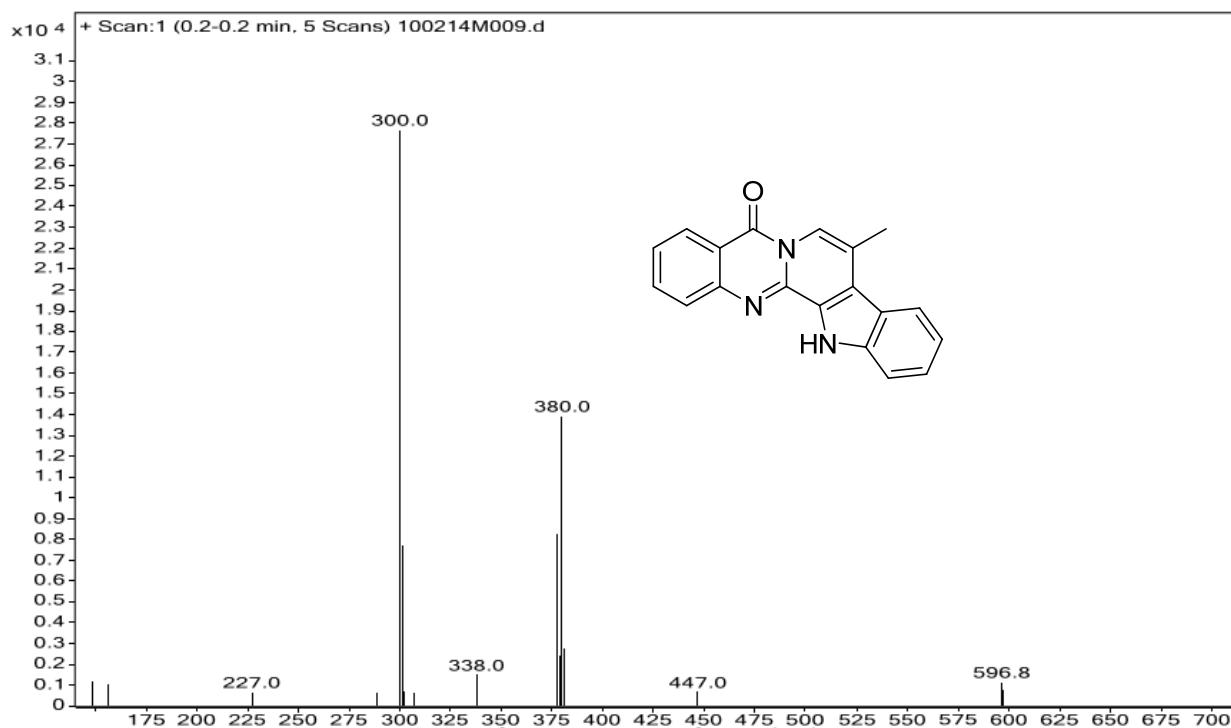


Fig. 77: Mass of compound 6