

Design and synthesis of coumarin-glyoxal hybrids for spermicidal and antimicrobial actions: A dual approach to contraception.

Swati Gupta,^aBhavana Kushwaha,^bAkansha Srivastava,^aJagdamba Prasad Maikhuri,^bSatya N. Sankhwar^c, Gopal Gupta,^b Anil Kumar Dwivedi^{a*}

^aPharmaceutics Division, CSIR-Central Drug Research Institute, Lucknow, 226031, India

^bEndocrinology Division, CSIR-Central Drug Research Institute, Lucknow, 226031, India

^cUrology Department, King George's Medical University, Lucknow-226003, India

Correspondence- Dr. A. K. Dwivedi, Pharmaceutics Division, CSIR- Central Drug Research Institute, Jankipuram Extension, Sitapur Road,Lucknow–226031,India.

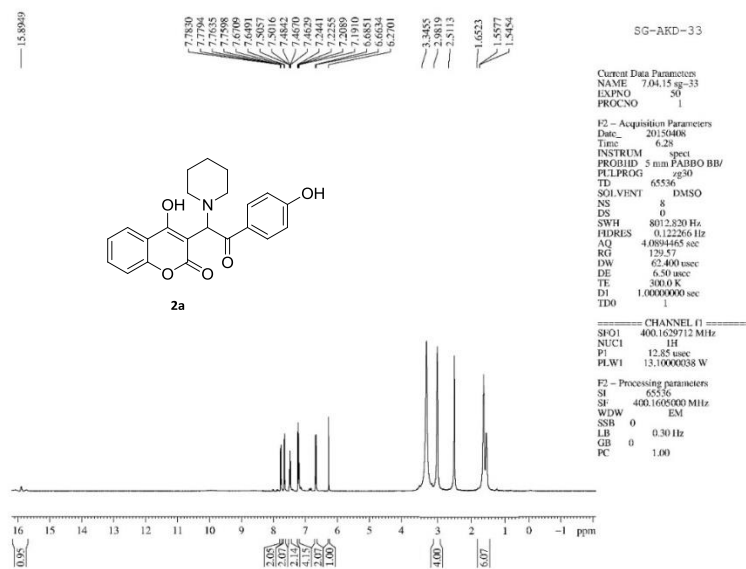
*Email- anilcdri@gmail.com. **Fax** -+91-522-91-522-2623938, 91-522-2623405

Supporting Information

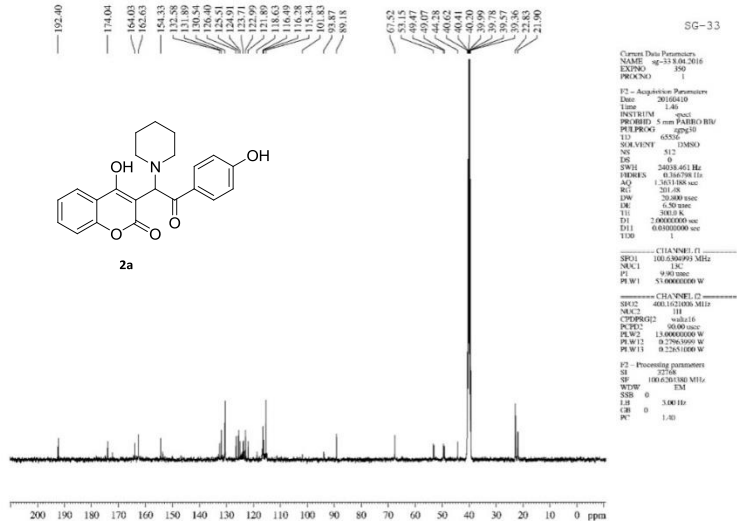
1. Scan data of compounds 2a-2h	S2-S13
2. Scan data of compounds 3a-3h	S14-25
3. Scan data of compounds 4a-4h	S25-S37
4. Scan data of compounds 5a-5f	S37-S46
5. HPLC data of all compounds	S47-S56
6. Mass and NMR spectra of dicoumarol	S56-S58

Copies of ^1H , ^{13}C NMR and HRMS spectra of compounds:

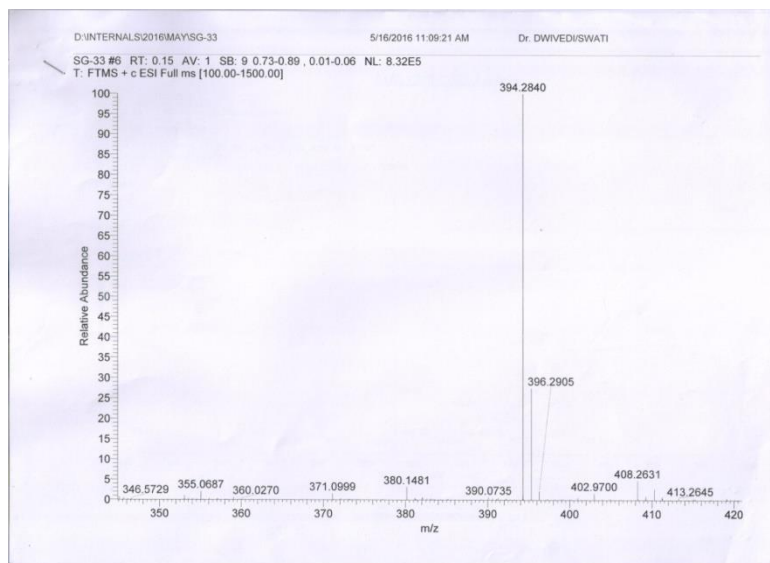
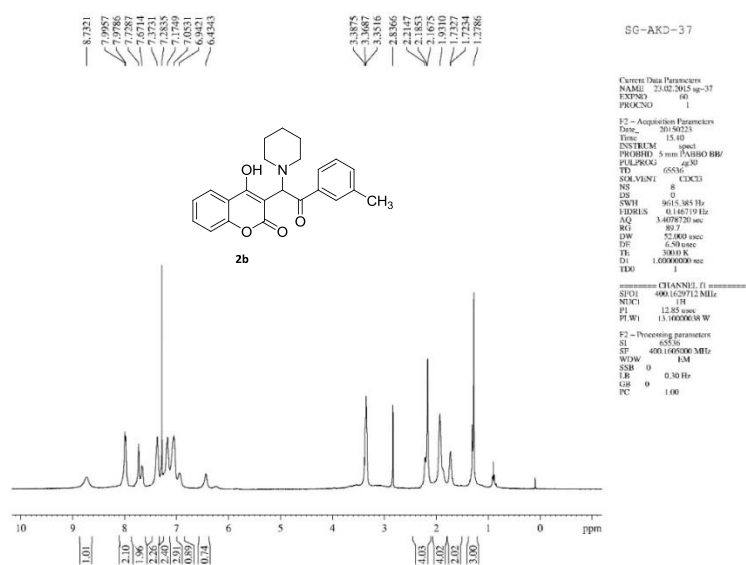
^1H NMR spectrum of compound 2a:



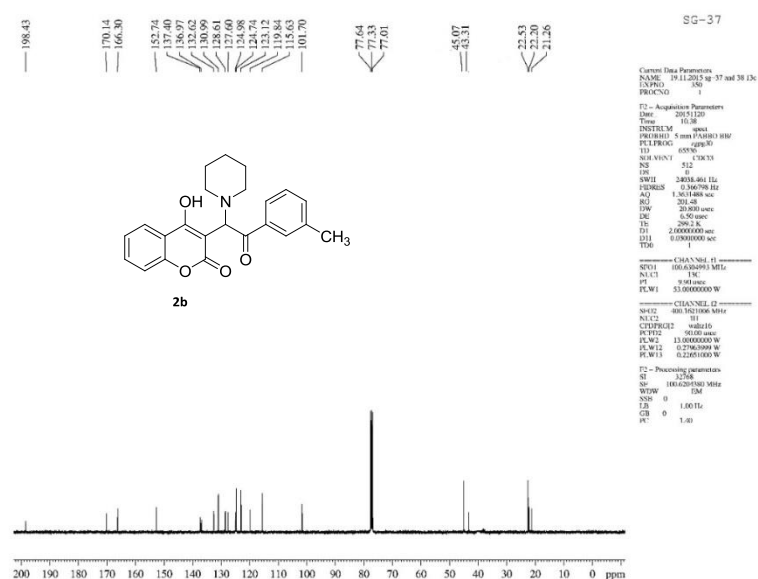
^{13}C NMR spectrum of compound 2a:



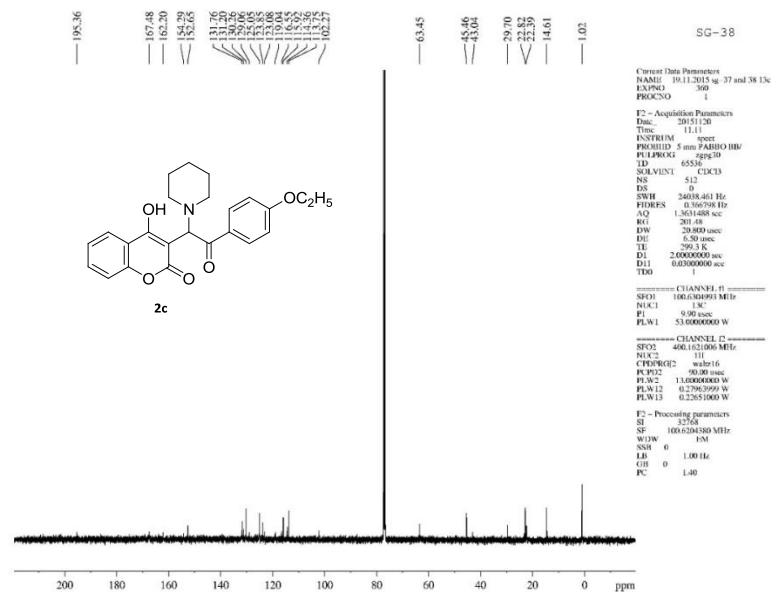
HRMS spectra of compound 2a:

 ^1H NMR spectra of compound 2b:

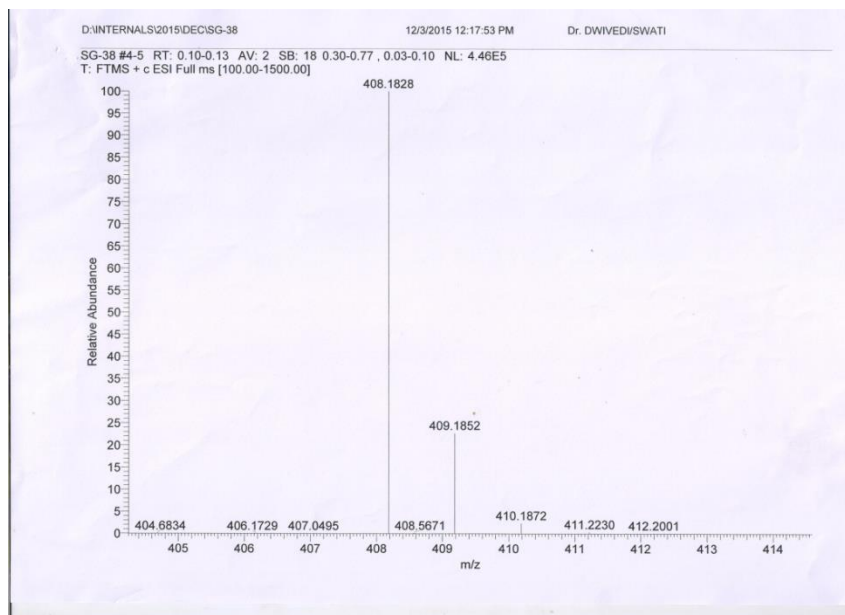
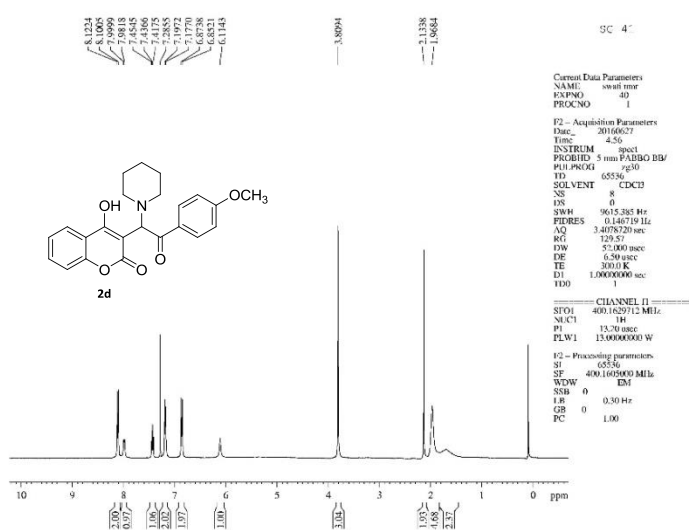
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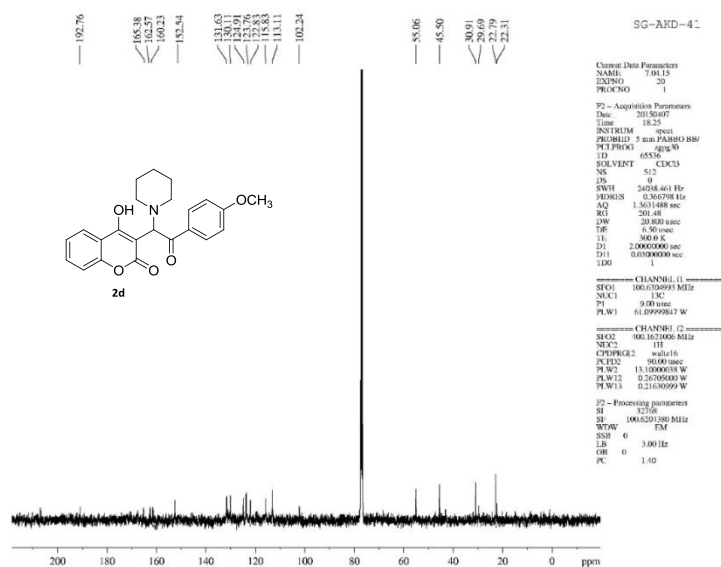
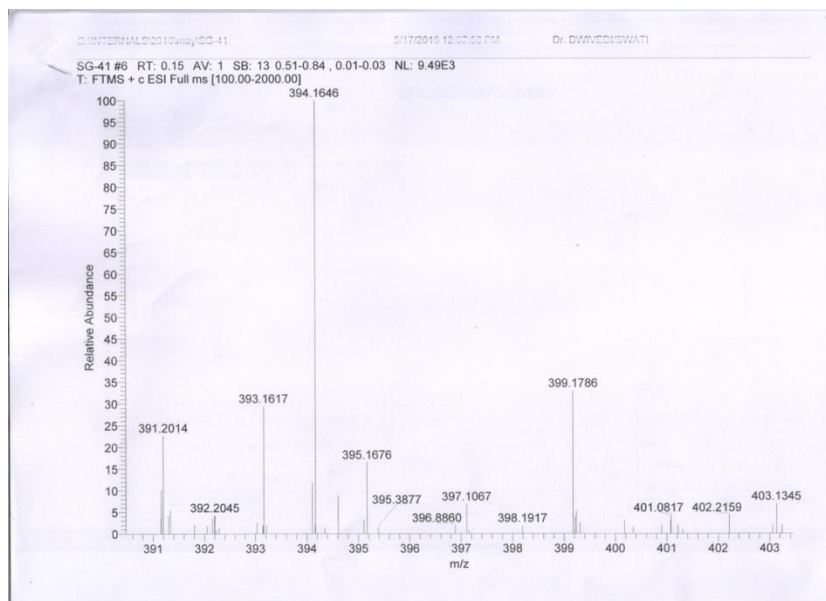


^{13}C NMR spectra of compound 2c:

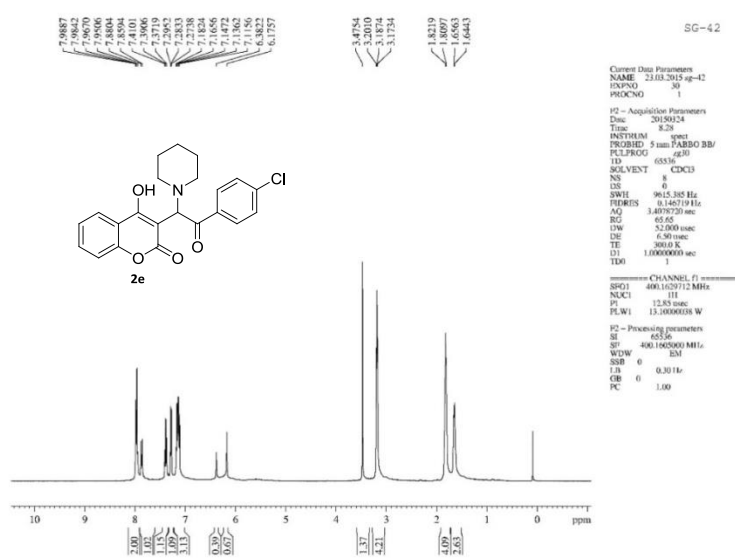


HRMS spectra of compound 2c:

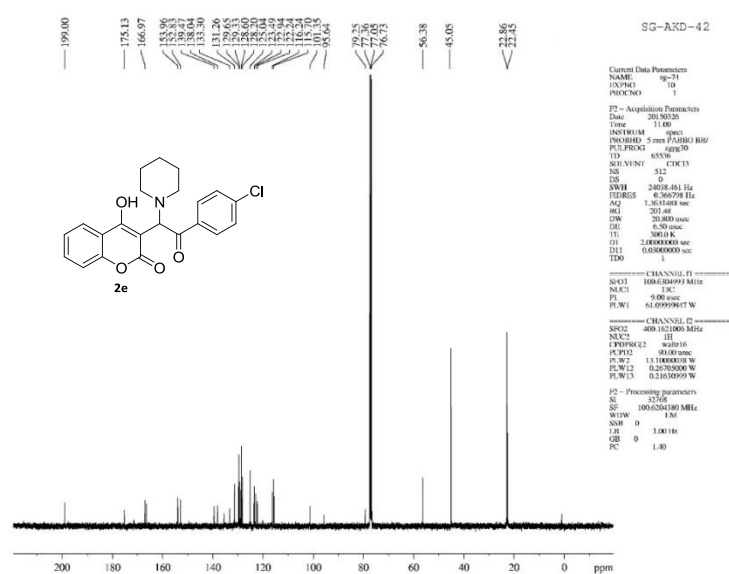
 ^1H NMR spectra of compound 2d:

¹³C NMR spectra of compound 2d:**HRMS spectra of compound 2d:**

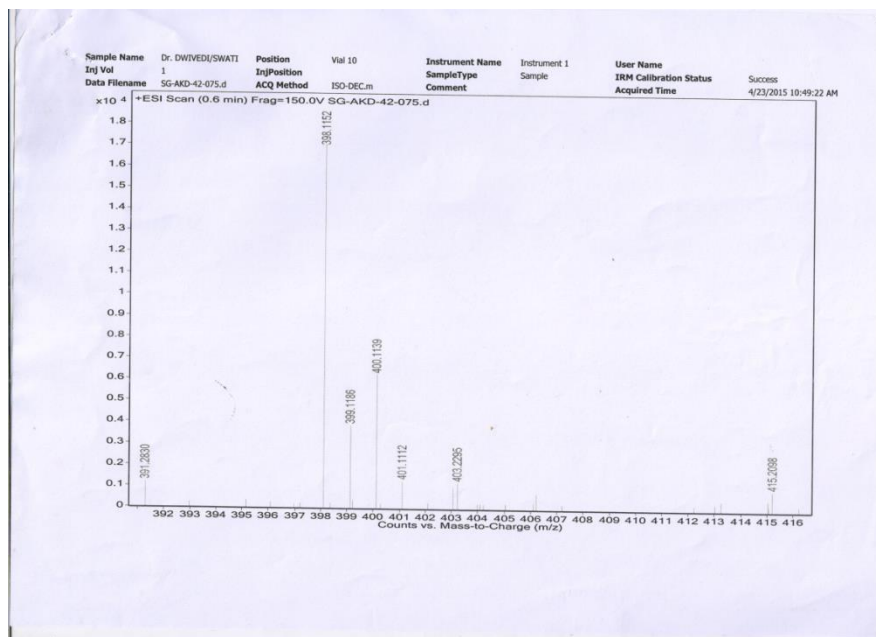
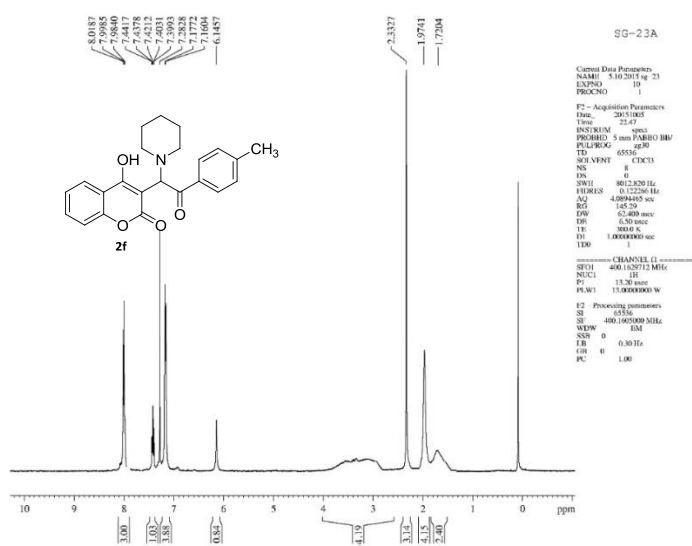
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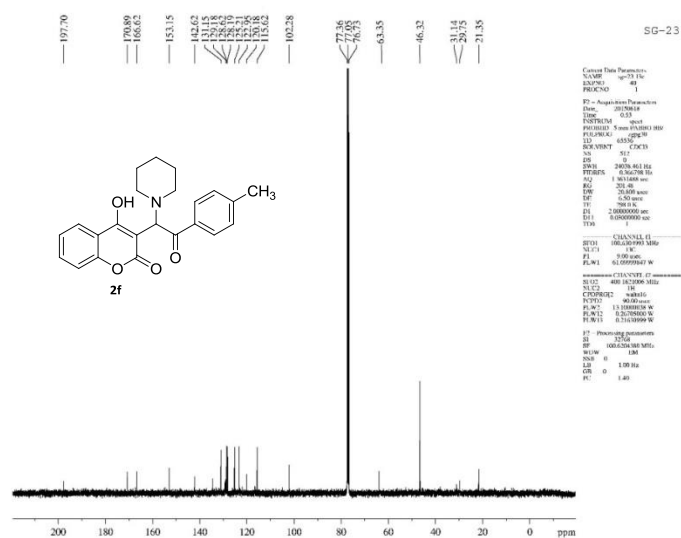
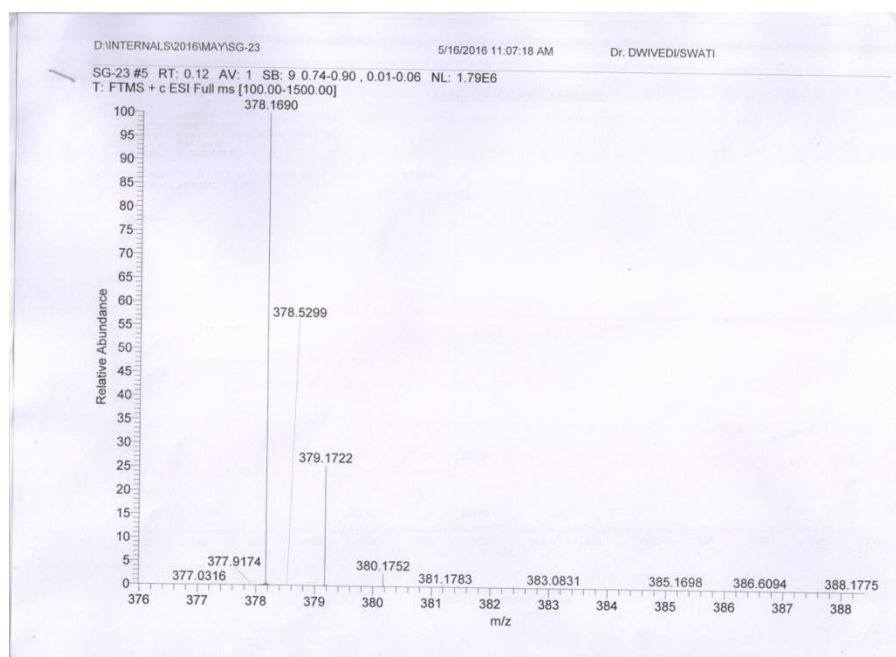


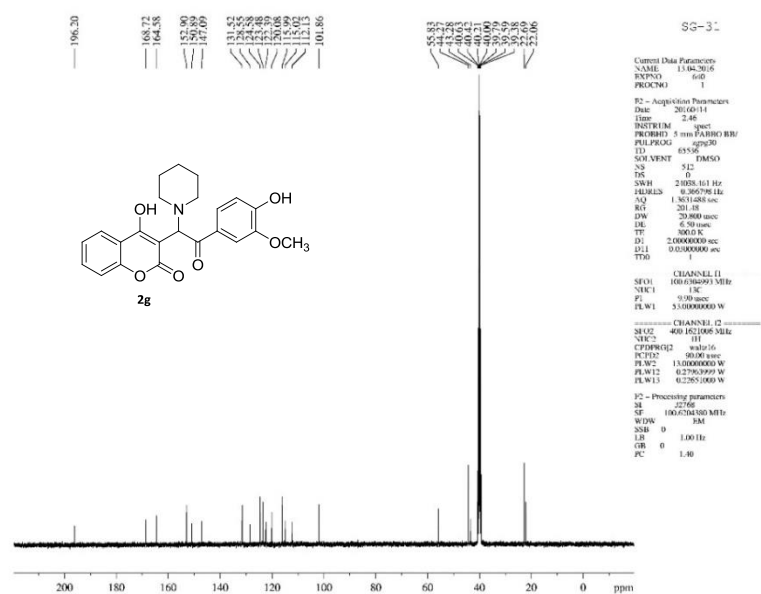
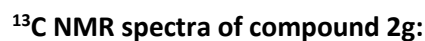
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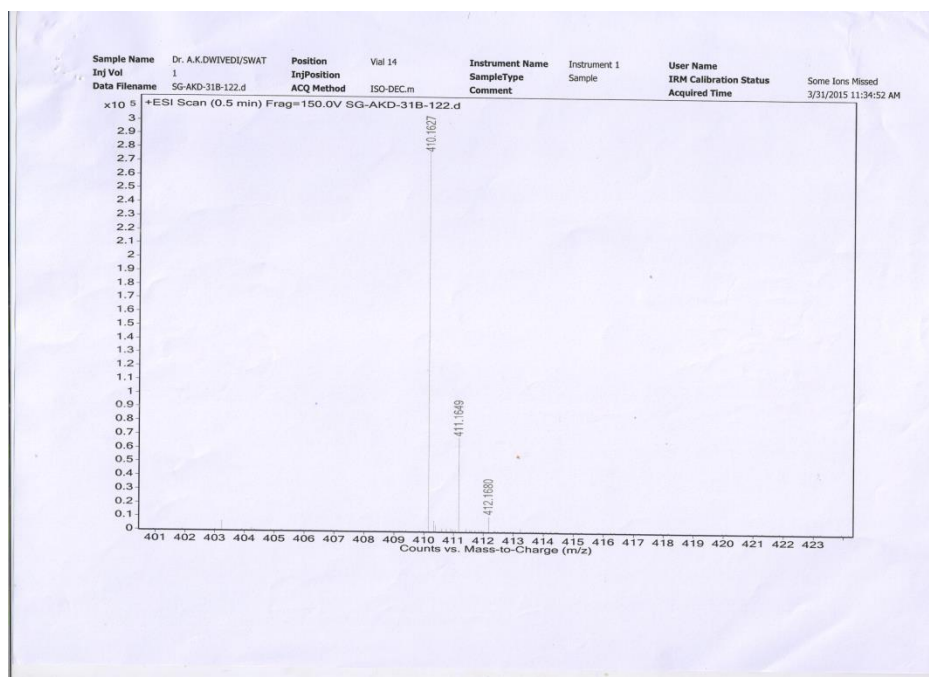
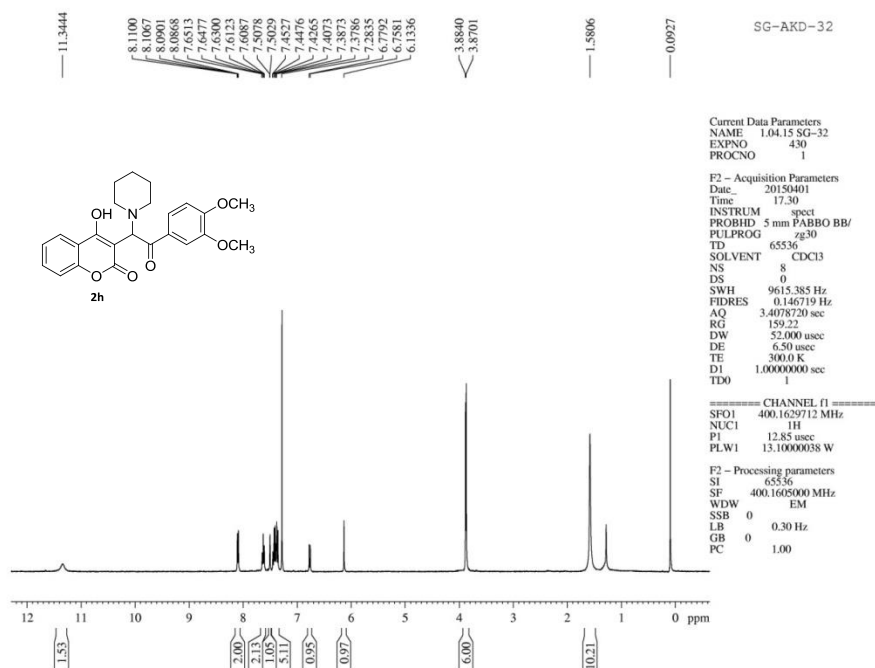
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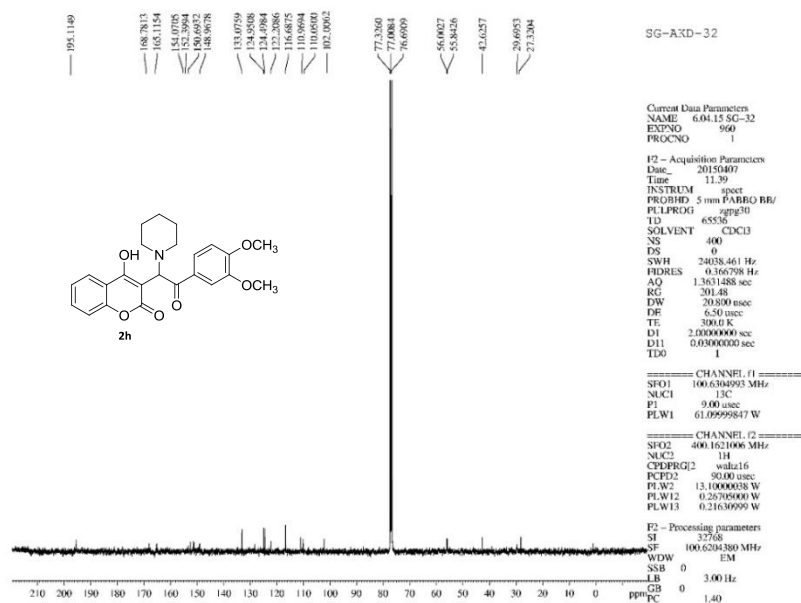
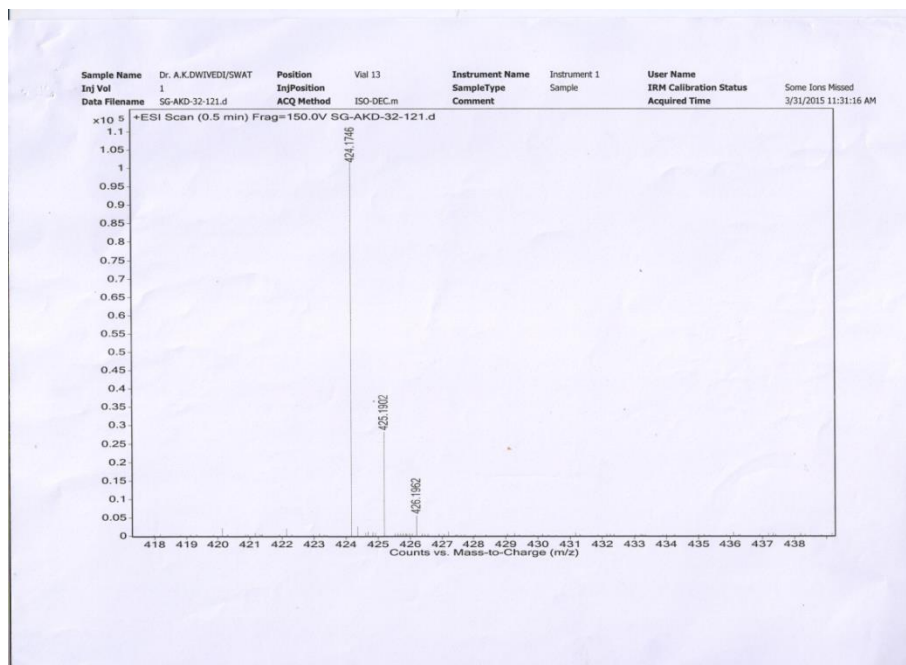
¹H NMR spectra of compound 2f:

¹³C NMR spectra of compound 2f:**HRMS spectra of compound 2f:**

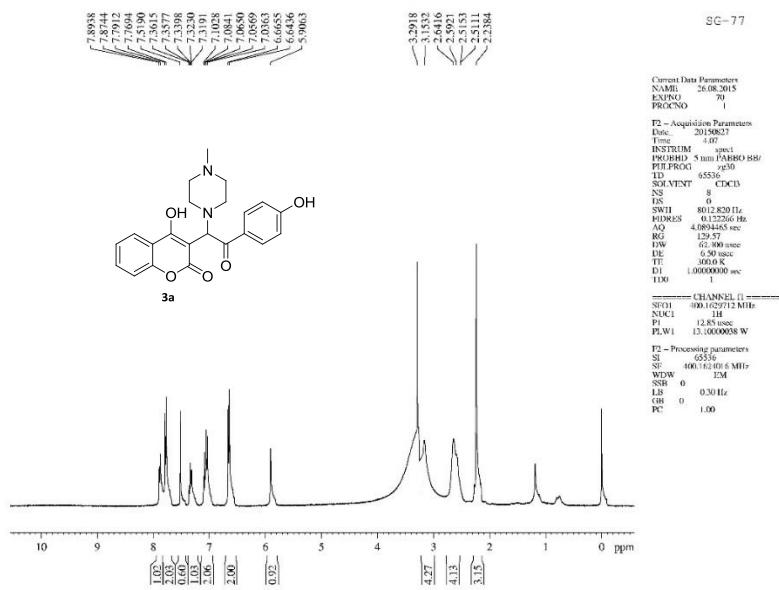


HRMS spectra of compound 2g:

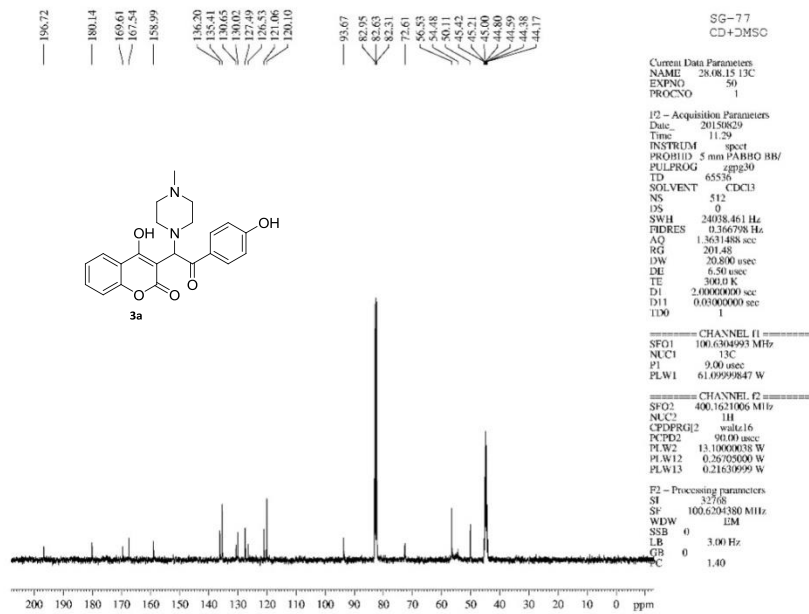
¹H NMR spectra of compound 2h:

¹³C NMR spectra of compound 2h:**HRMS spectra of compound 2h:**

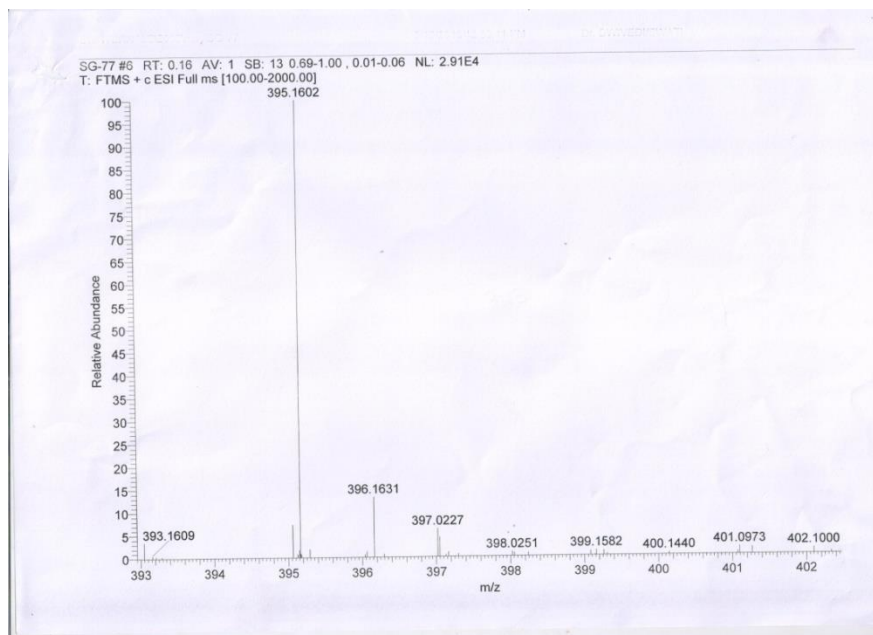
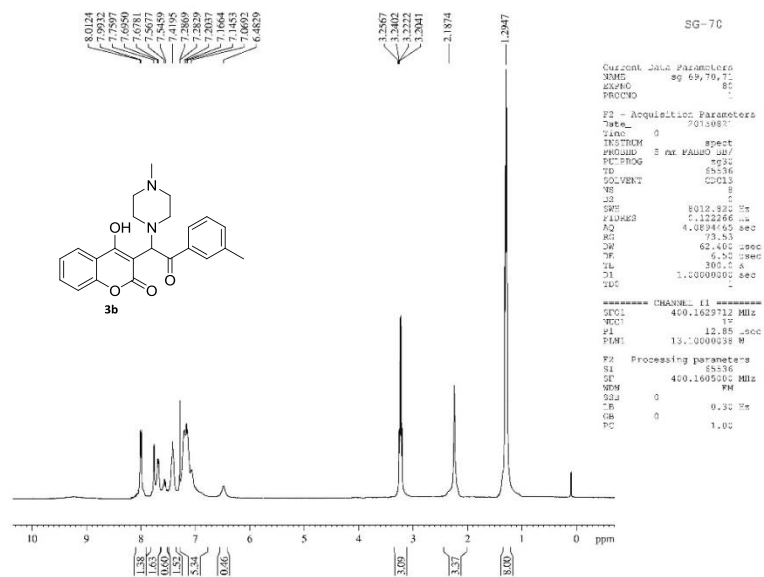
¹H NMR spectra of compound 3a:

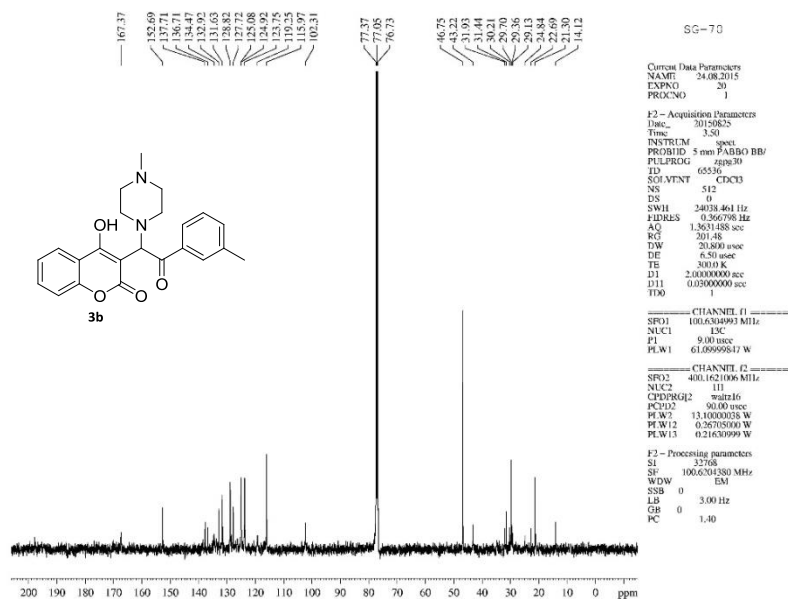
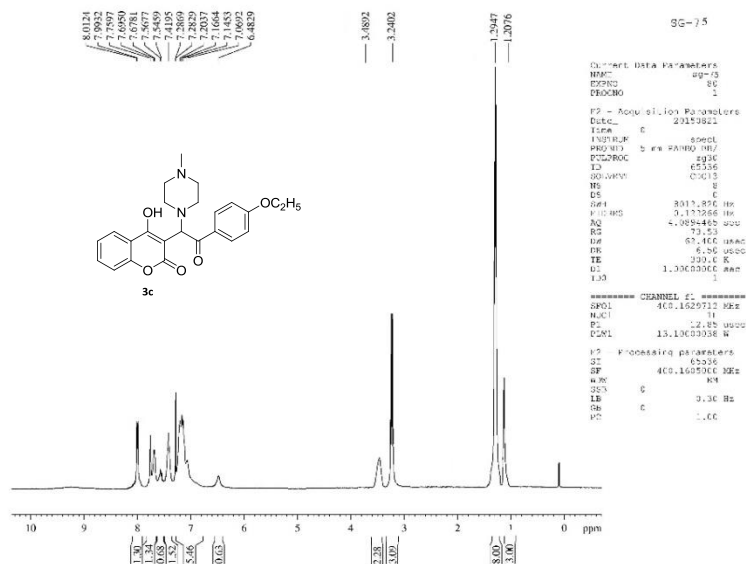


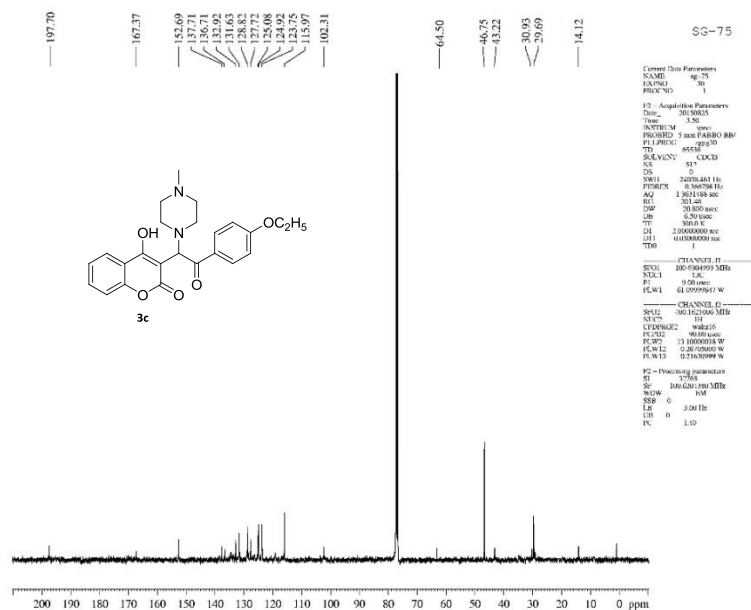
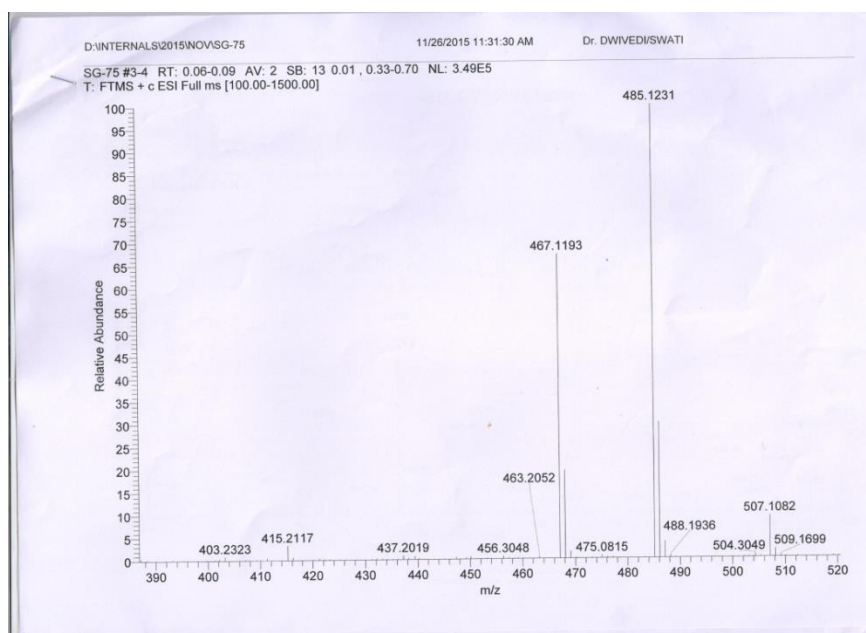
^{13}C NMR spectra of compound 3a:

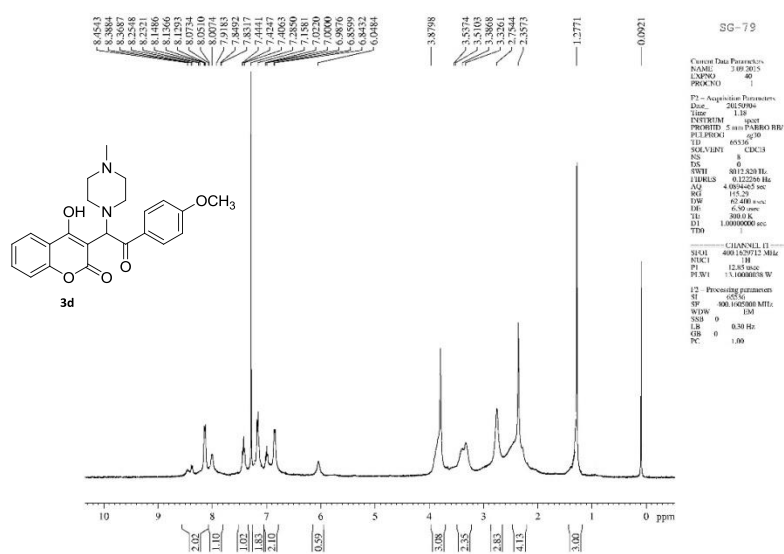
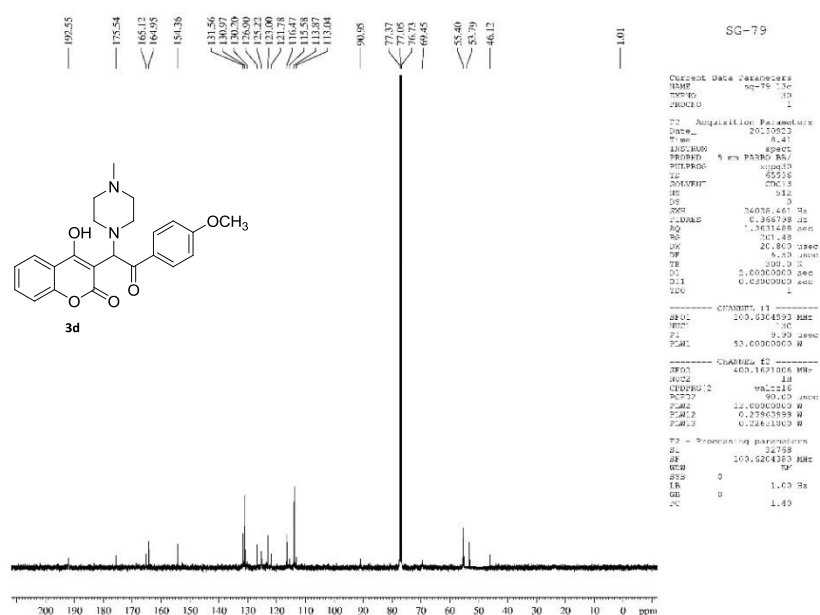


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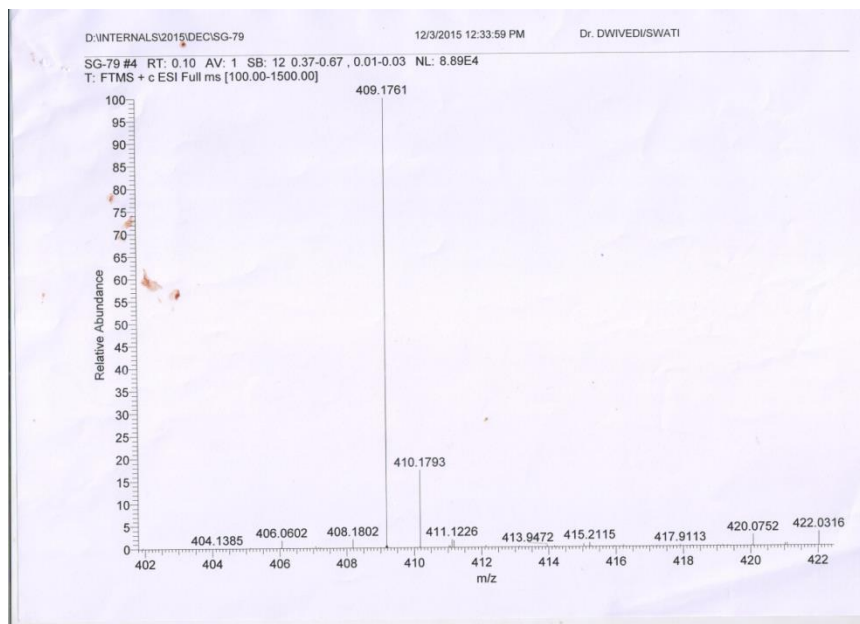
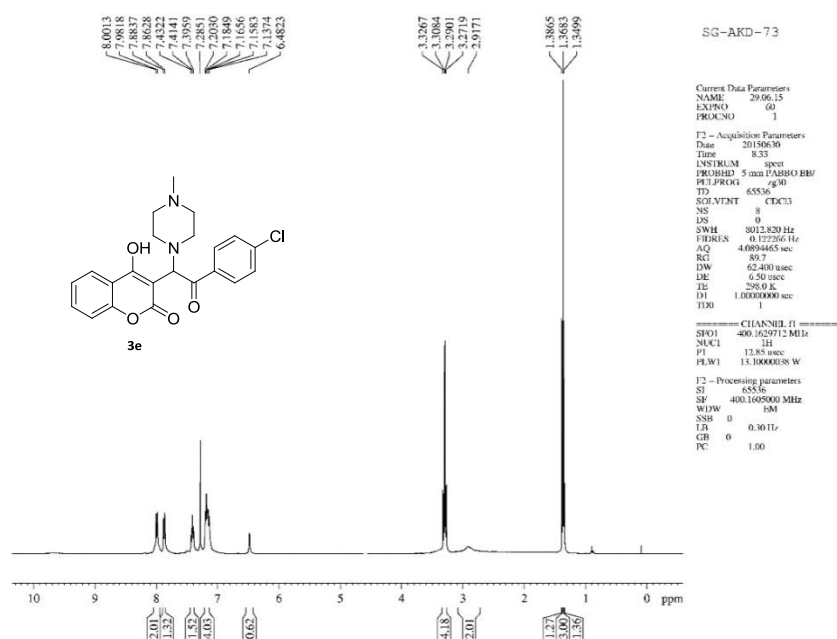
 ^1H NMR spectra of compound 3b:

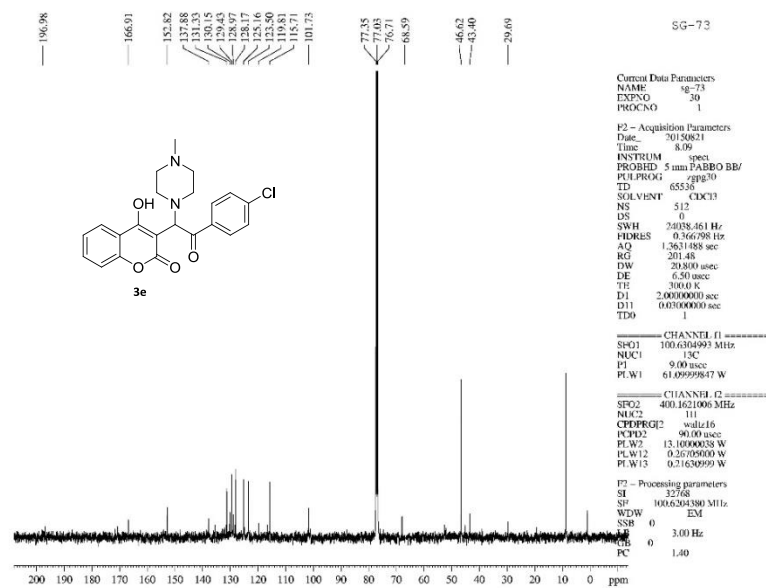
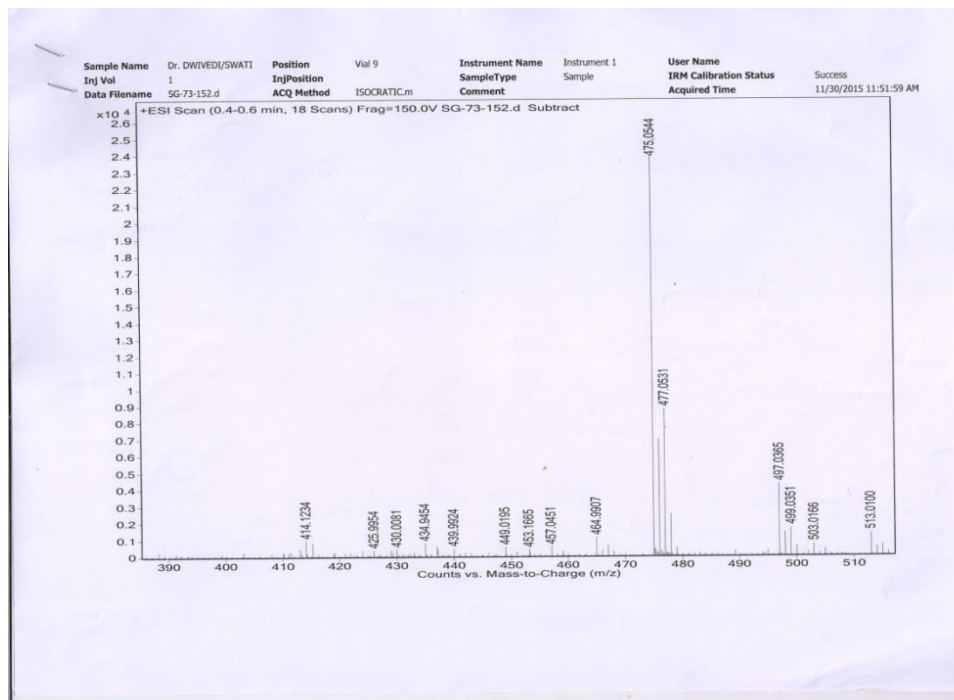
¹³C NMR spectra of compound 3b:**¹H NMR spectra of compound 3c:**

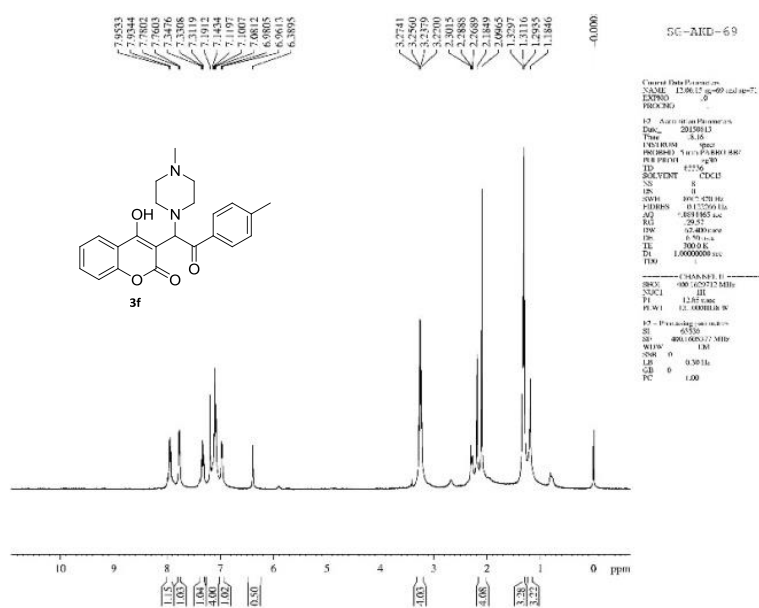
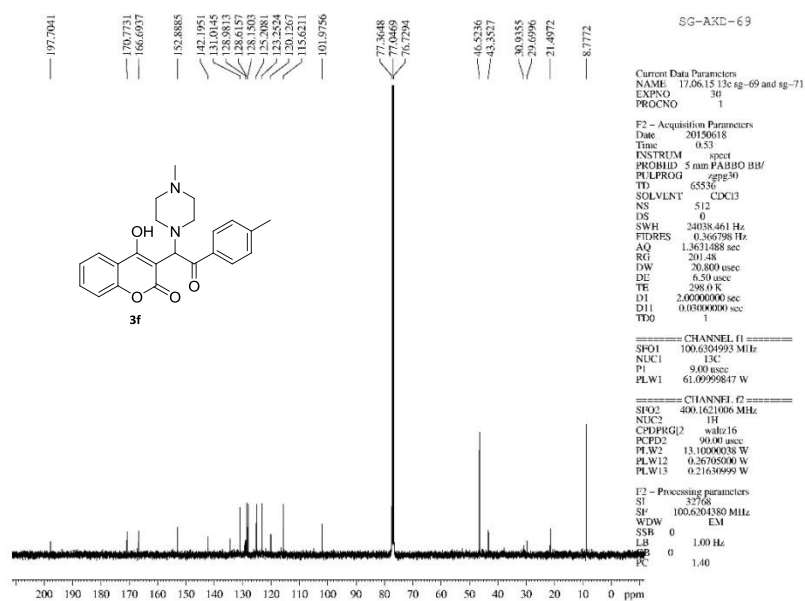
^{13}C NMR spectra of compound 3c:**HRMS spectra of compound 3c:**

¹H NMR spectra of compound 3d:**¹³C NMR spectra of compound 3d:**

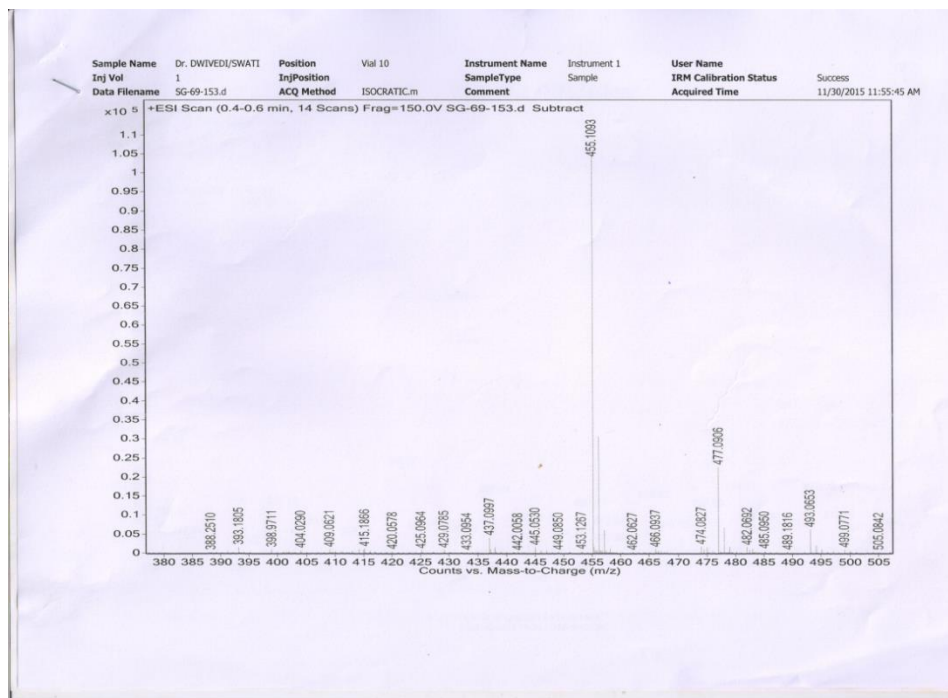
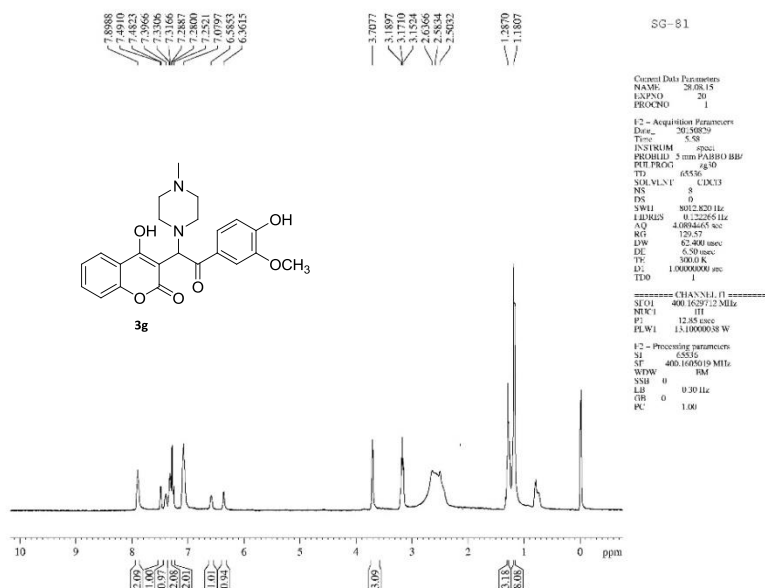
HRMS spectra of compound 3d:

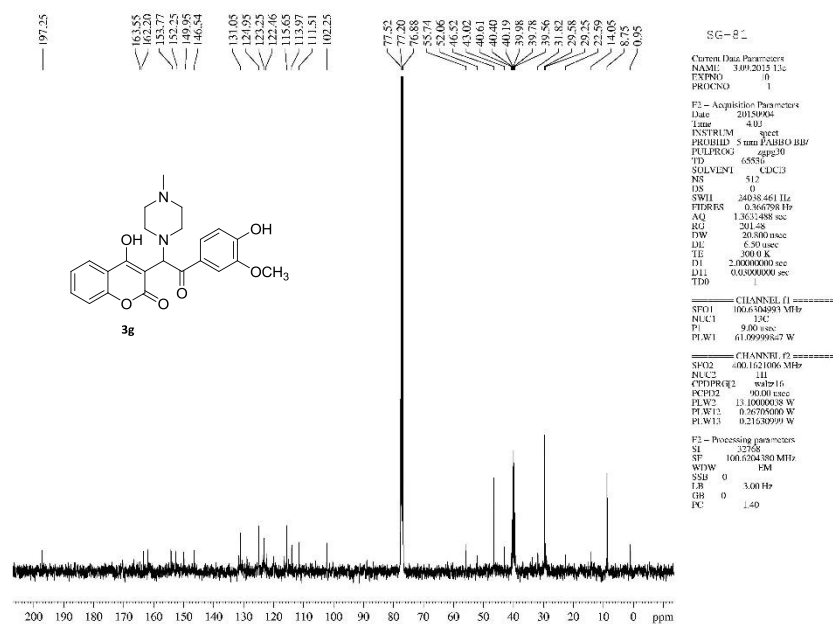
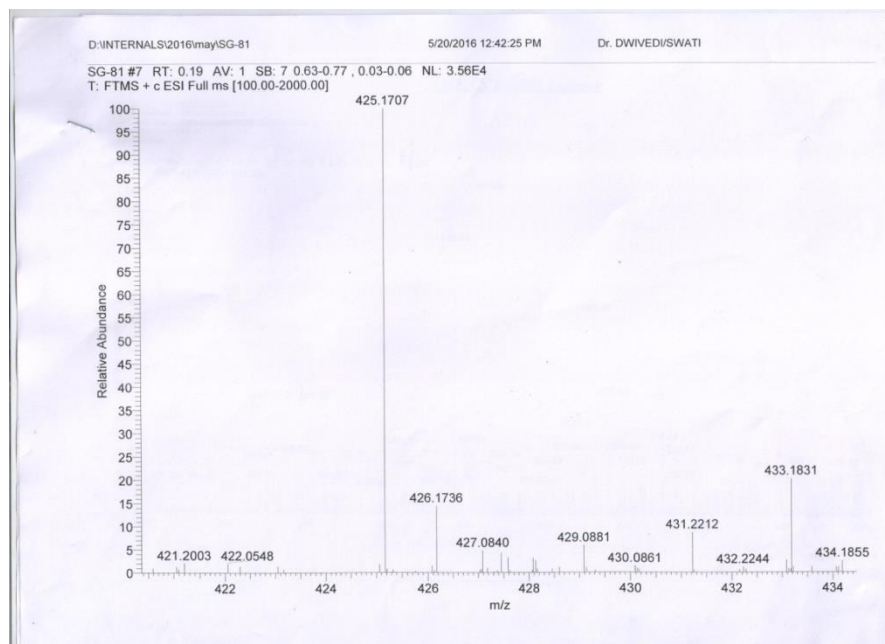
¹H NMR spectra of compound 3e:

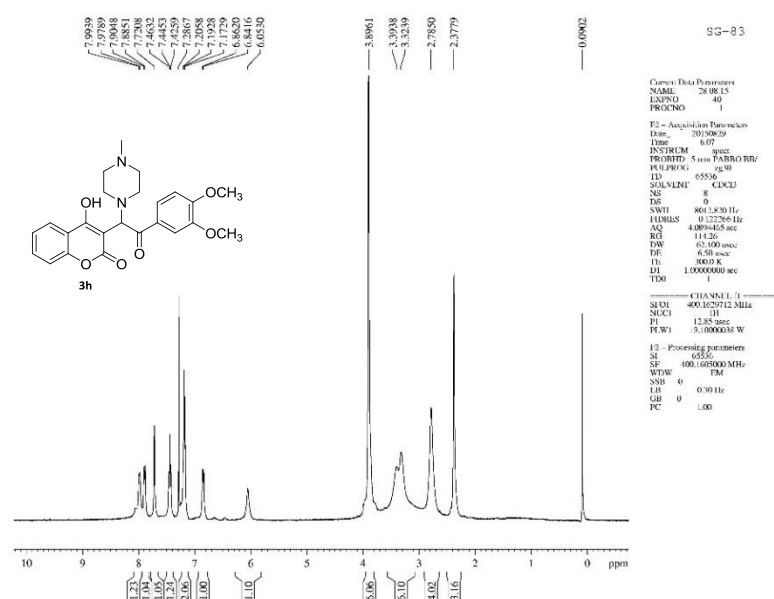
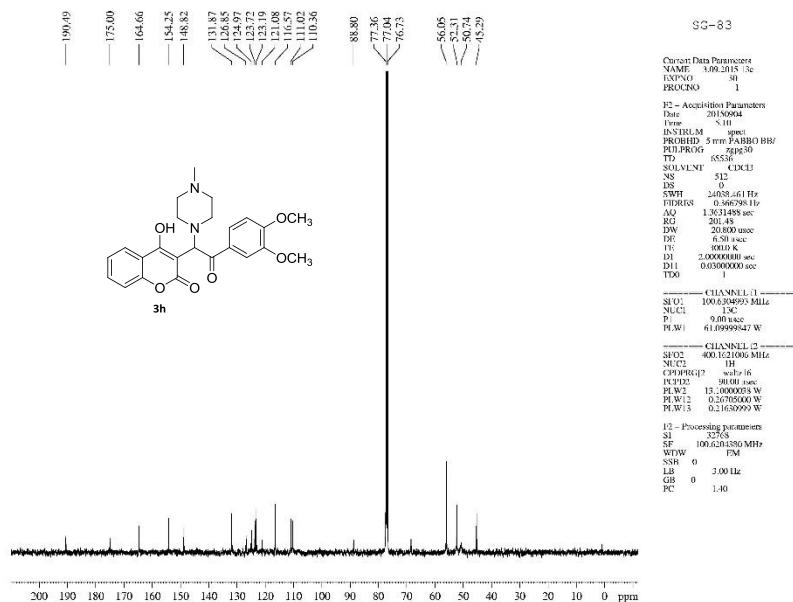
¹³C NMR spectra of compound 3e:**HRMS spectra of compound 3e:**

¹H NMR spectra of compound 3f:**¹³C NMR spectra of compound 3f:**

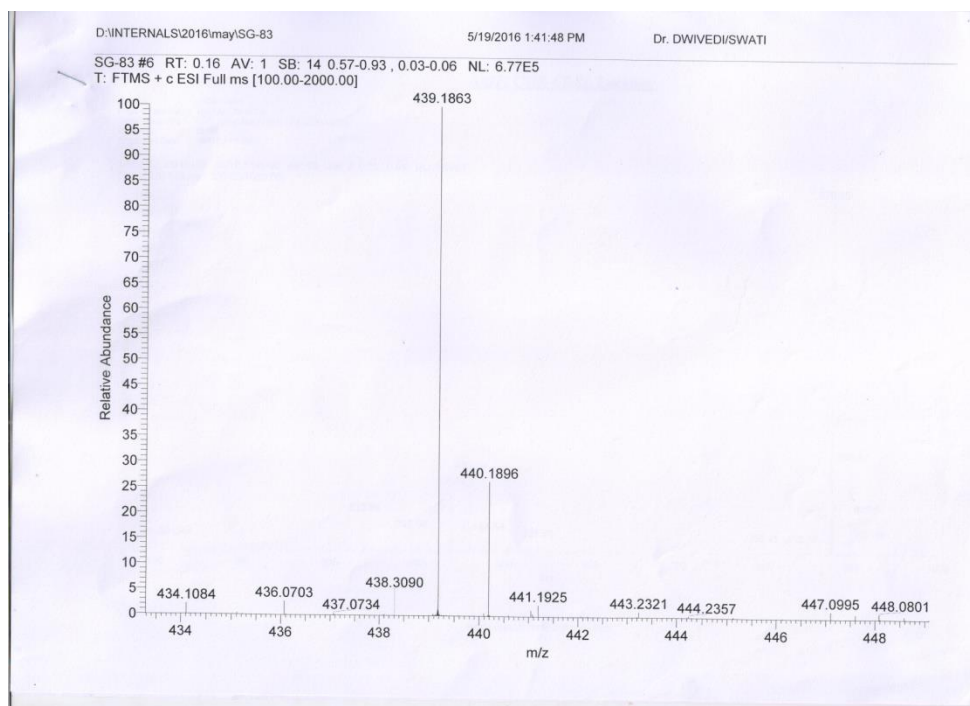
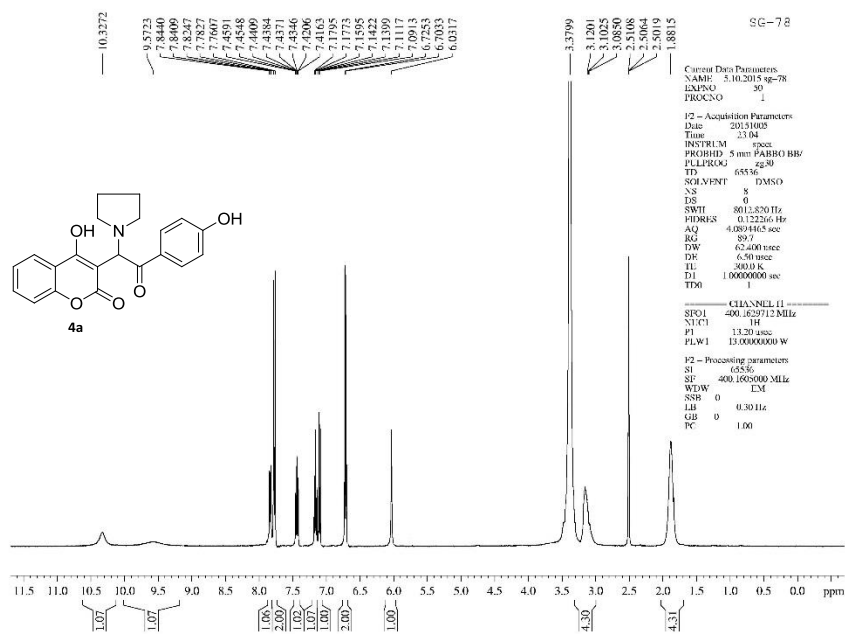
HRMS spectra of compound 3f:

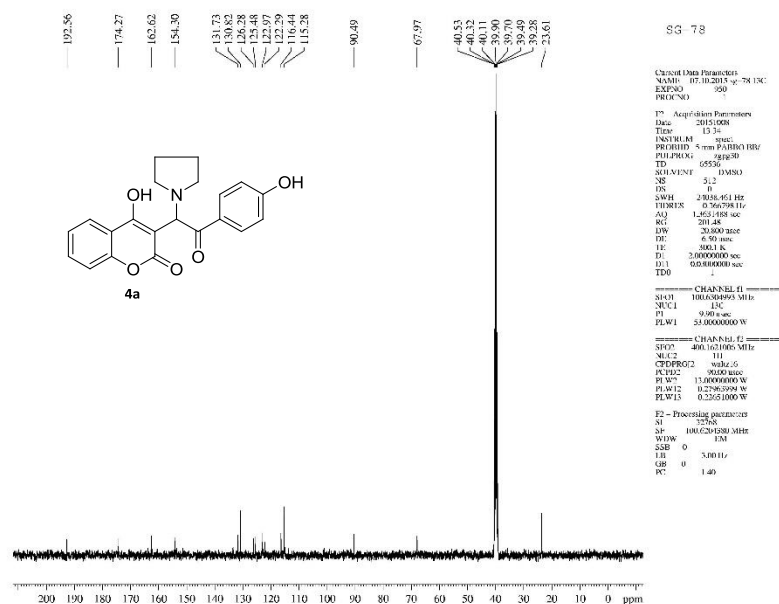
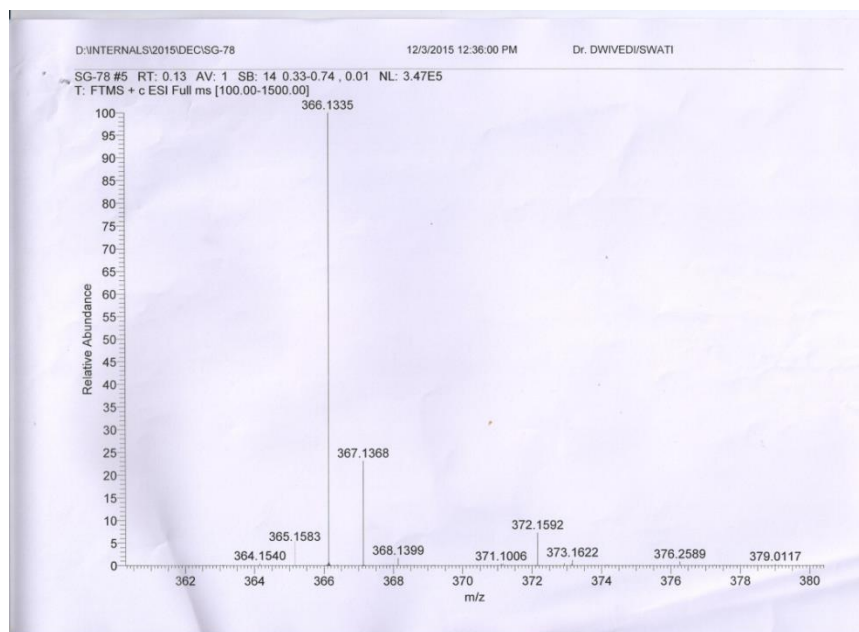
 ^1H NMR spectra of compound 3g:

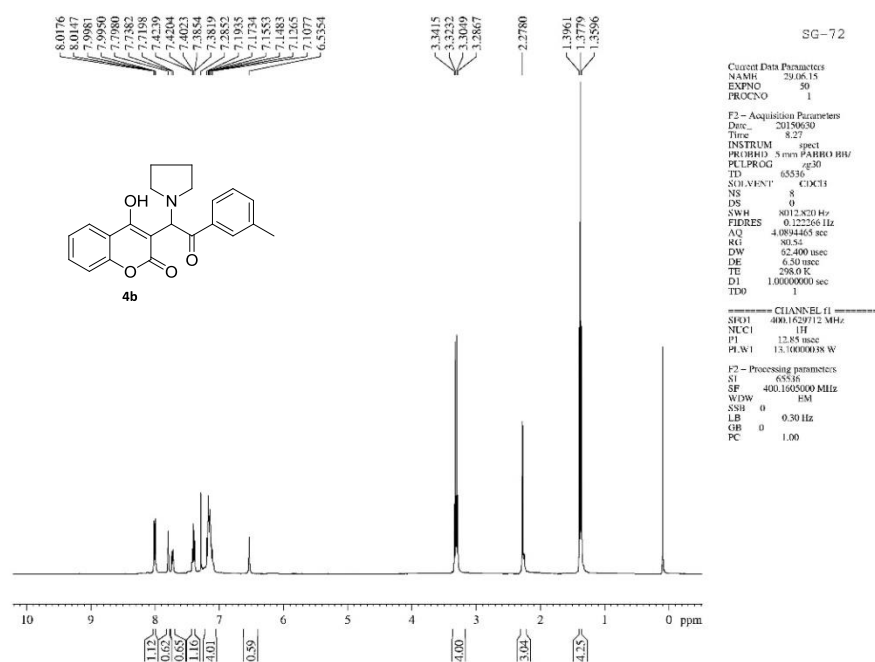
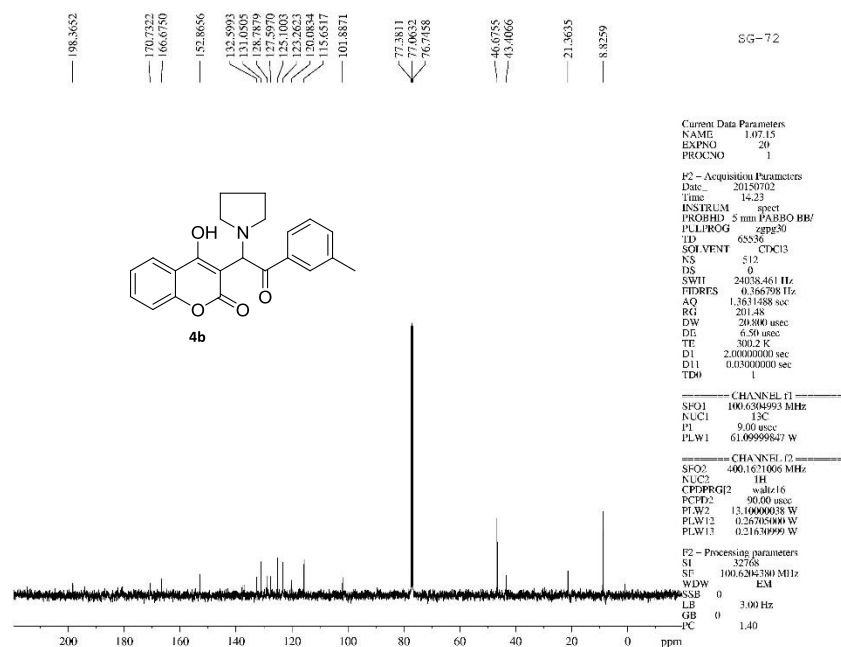
¹³C NMR spectra of compound 3g:**HRMS spectra of compound 3g:**

¹H NMR spectra of compound 3h:**¹³C NMR spectra of compound 3h:**

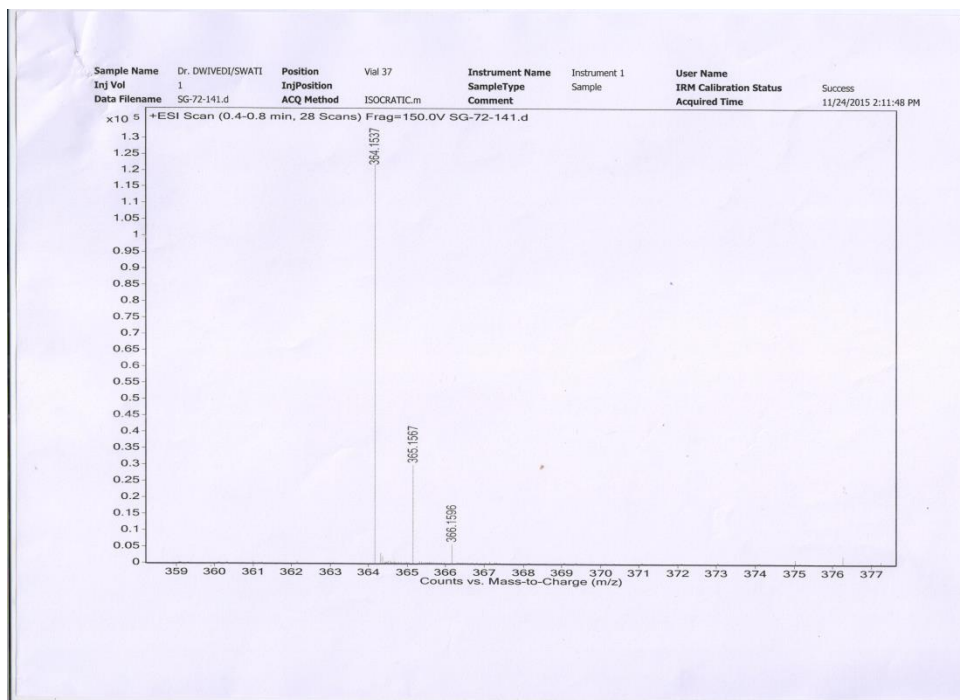
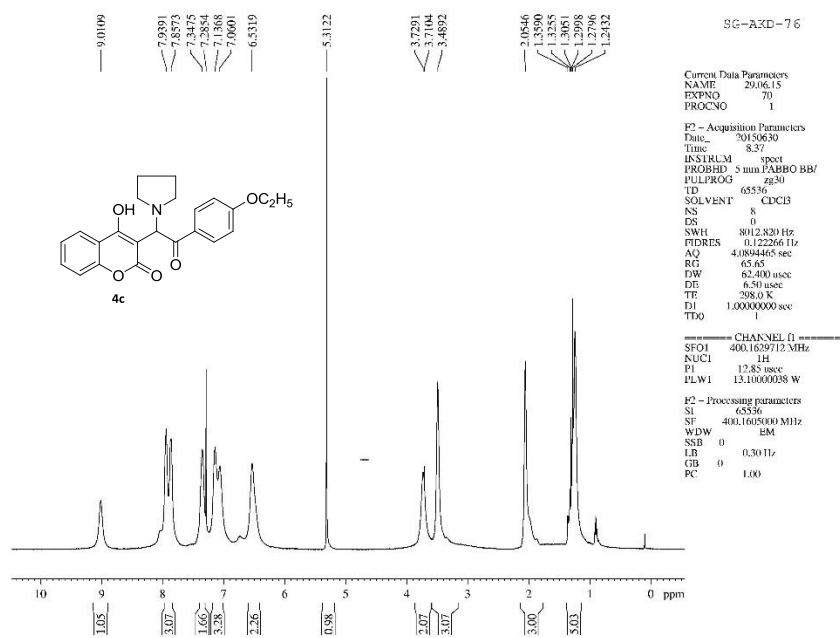
HRMS spectra of compound 3h:

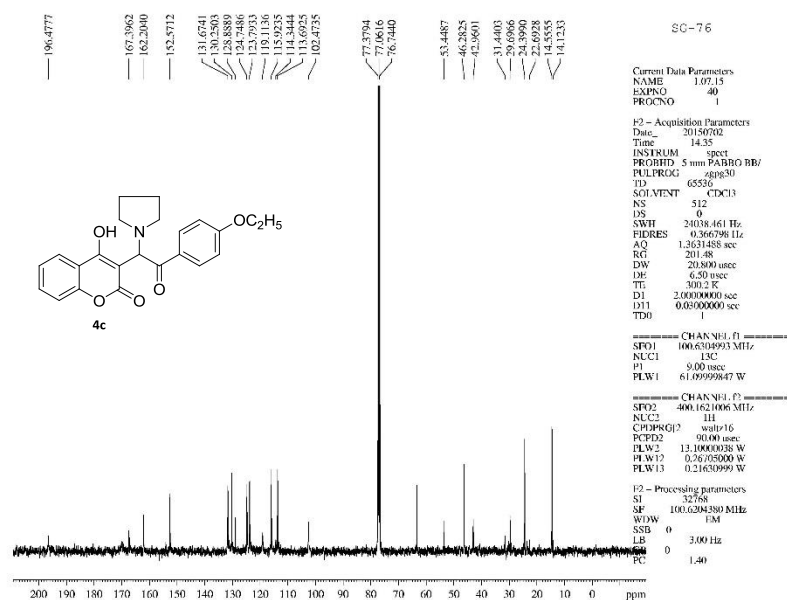
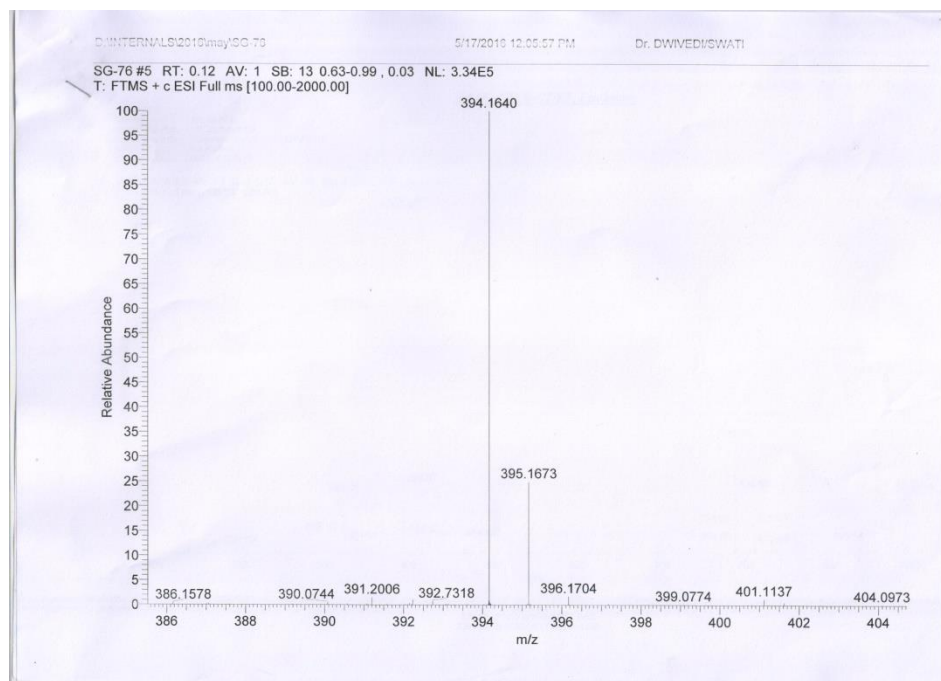
¹H NMR spectra of compound 4a:

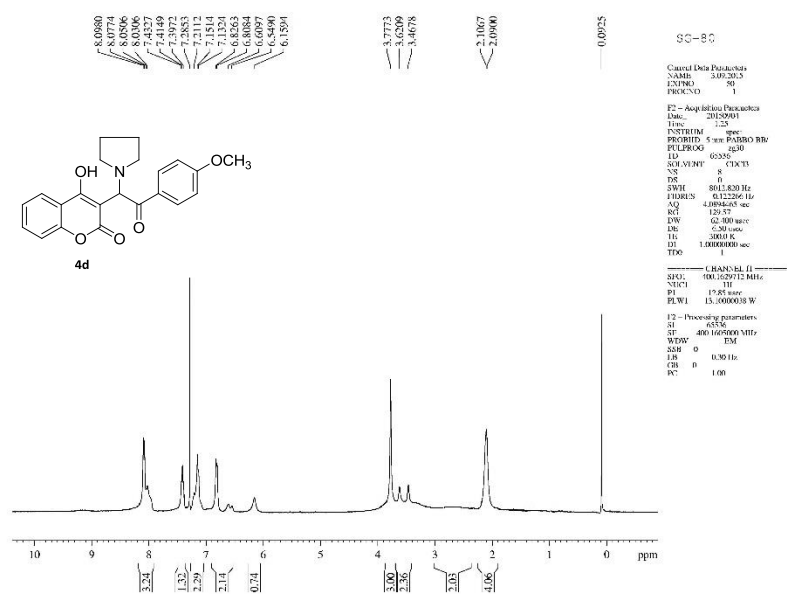
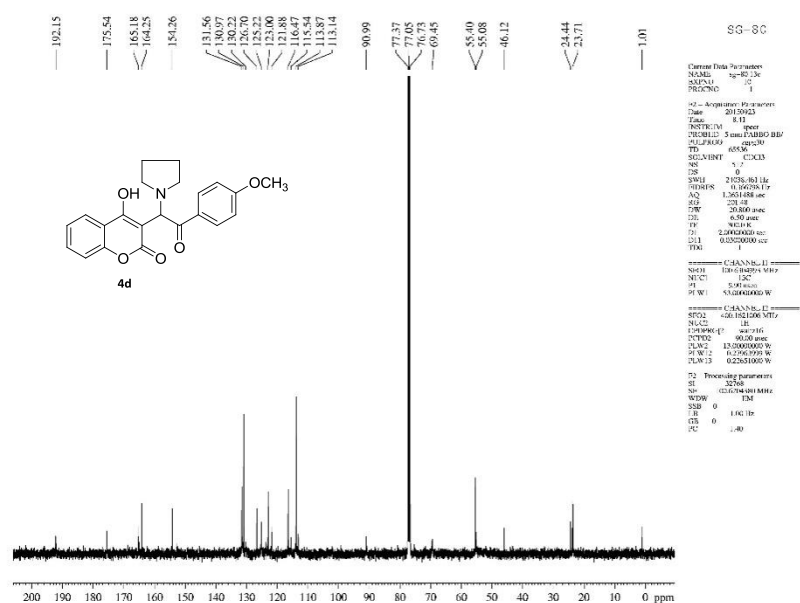
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¹H NMR spectra of compound 4b:¹³C NMR spectra of compound 4b:

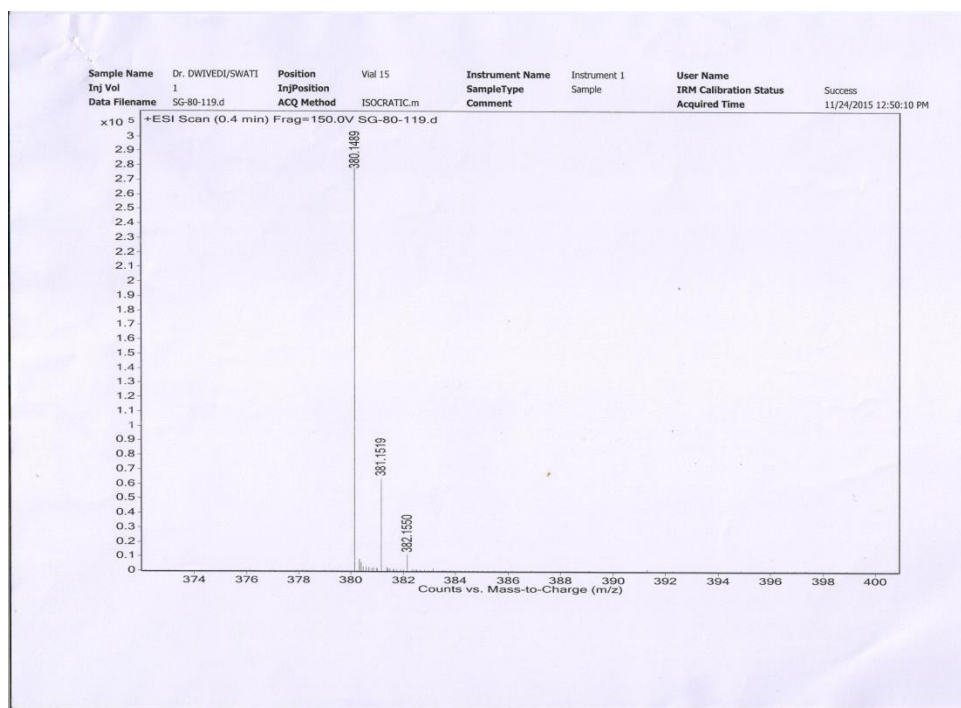
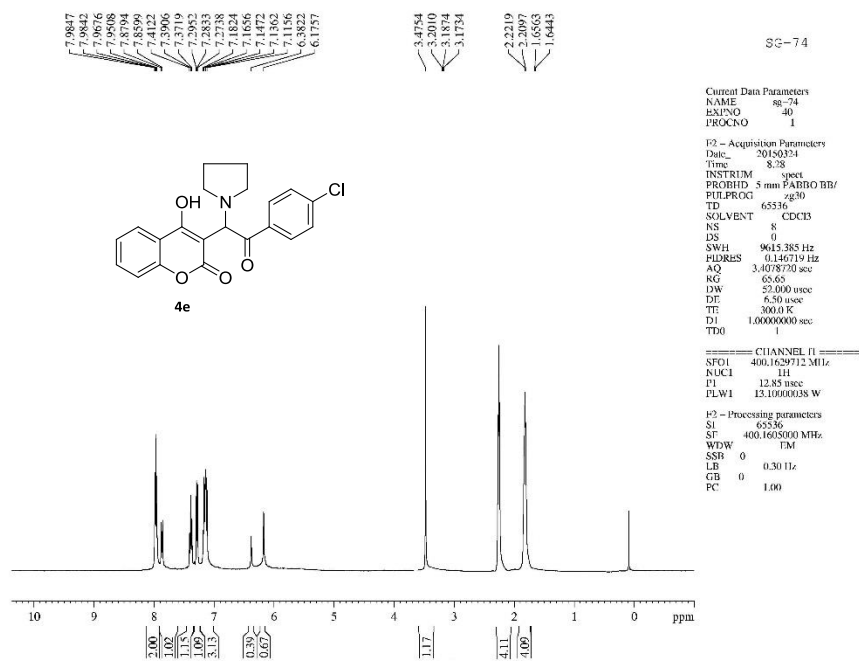
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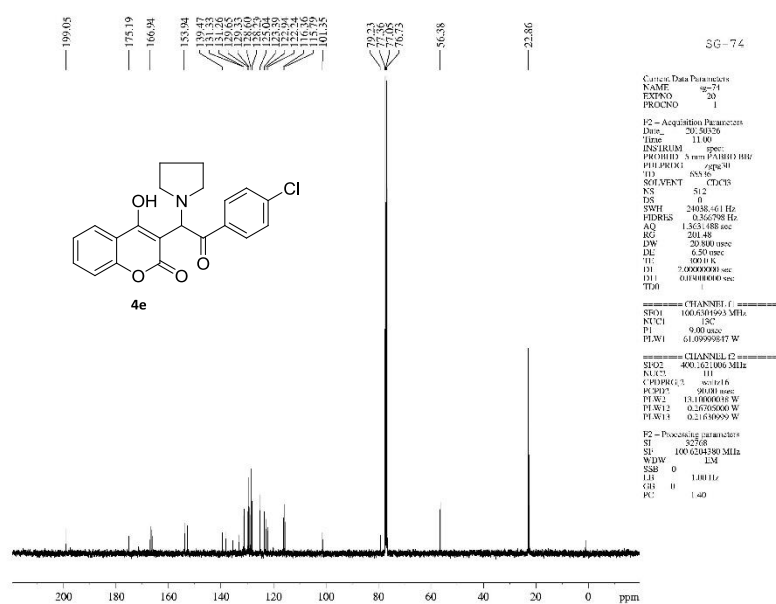
¹H NMR spectra of compound 4c:

¹³C NMR spectra of compound 4c:**HRMS spectra of compound 4c:**

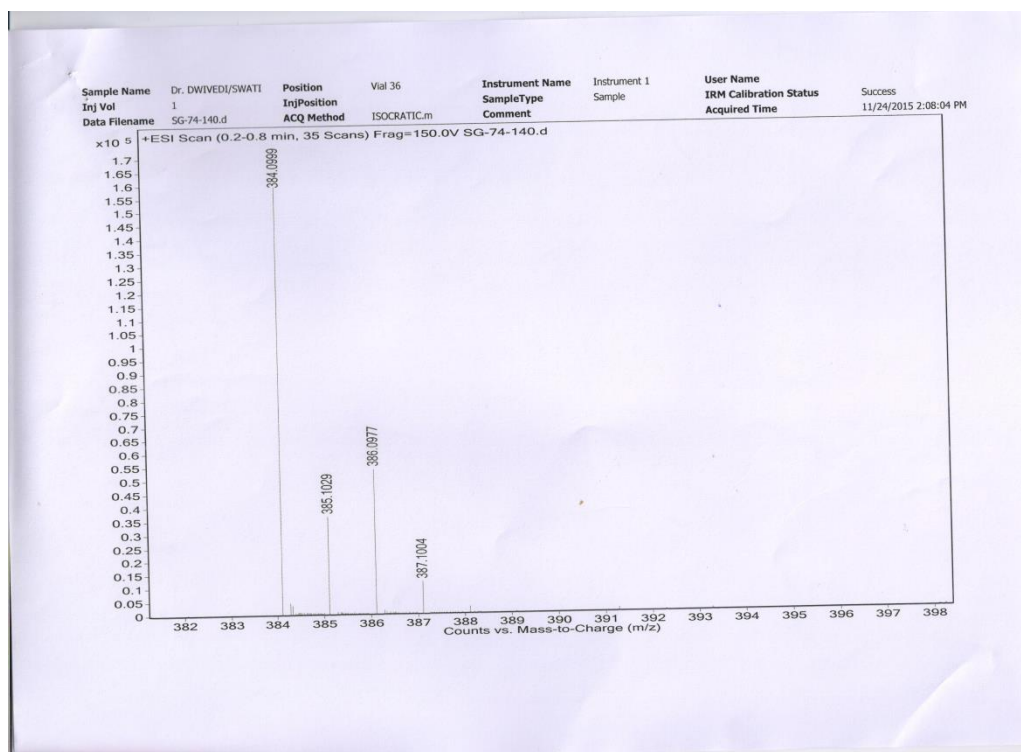
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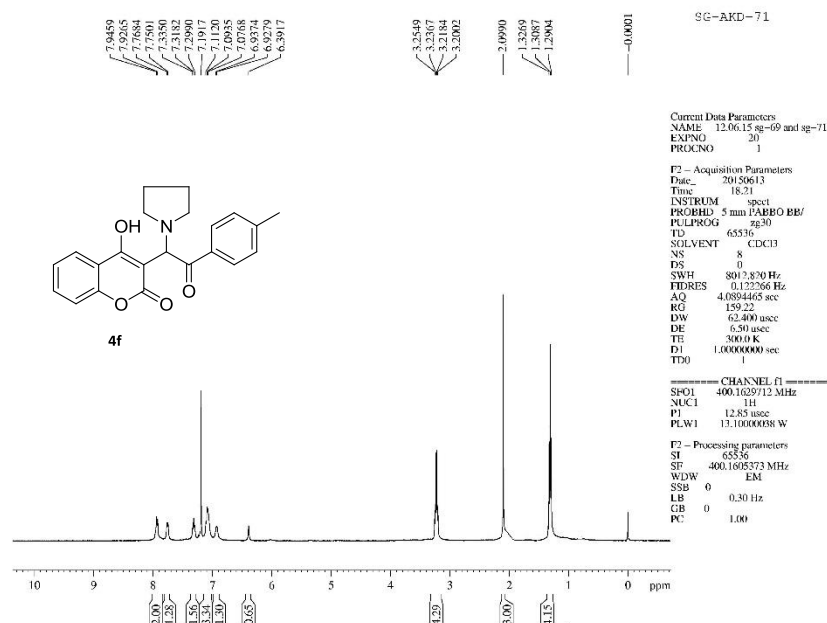
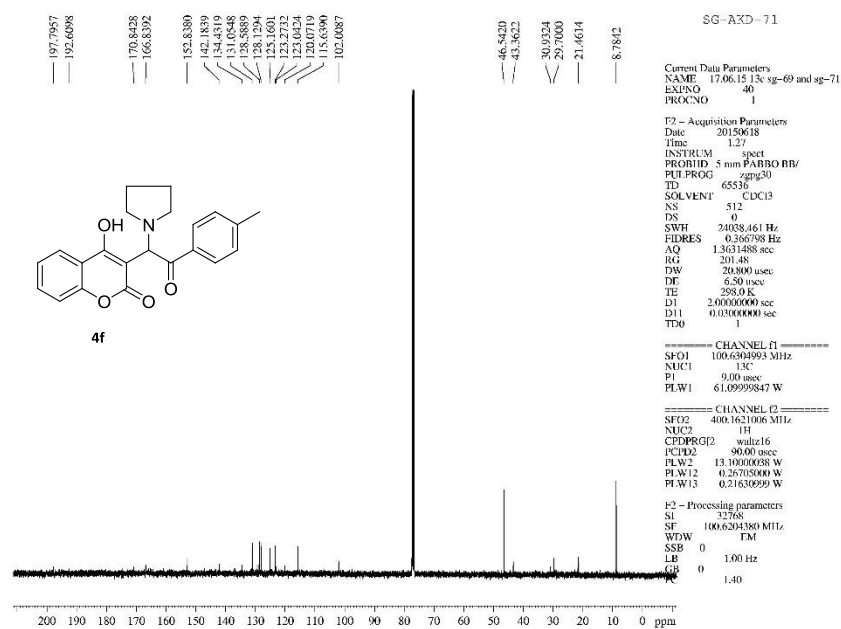
HRMS spectra of compound 4d:

 ^1H NMR spectra of compound 4e:

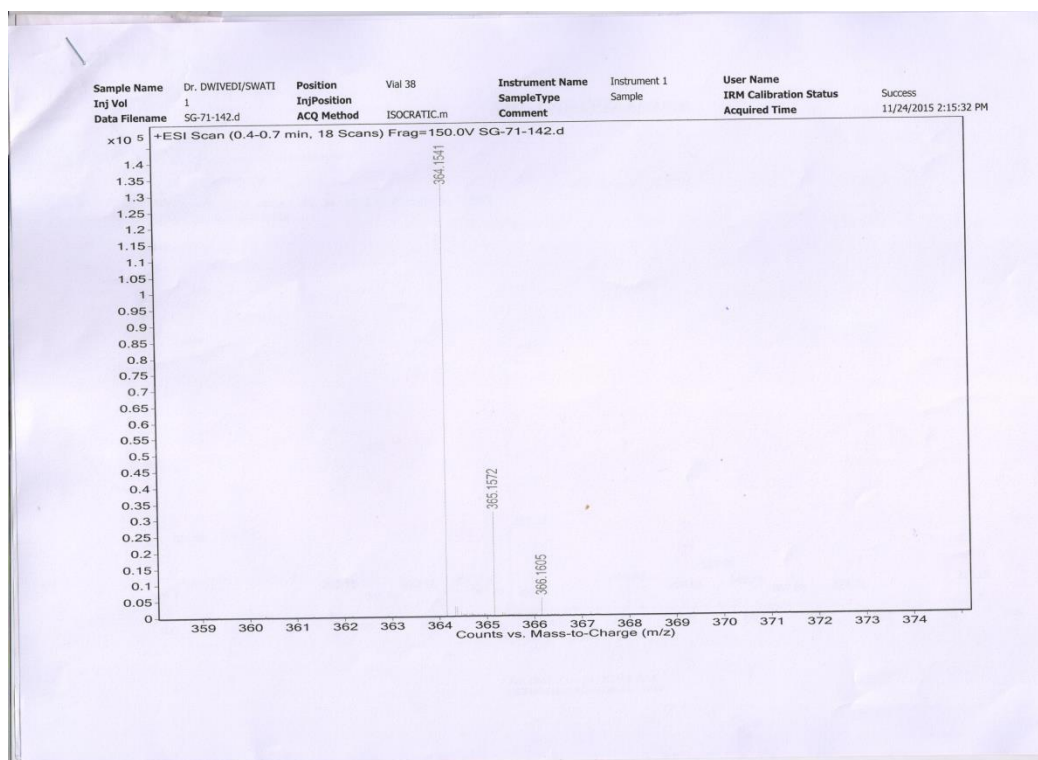
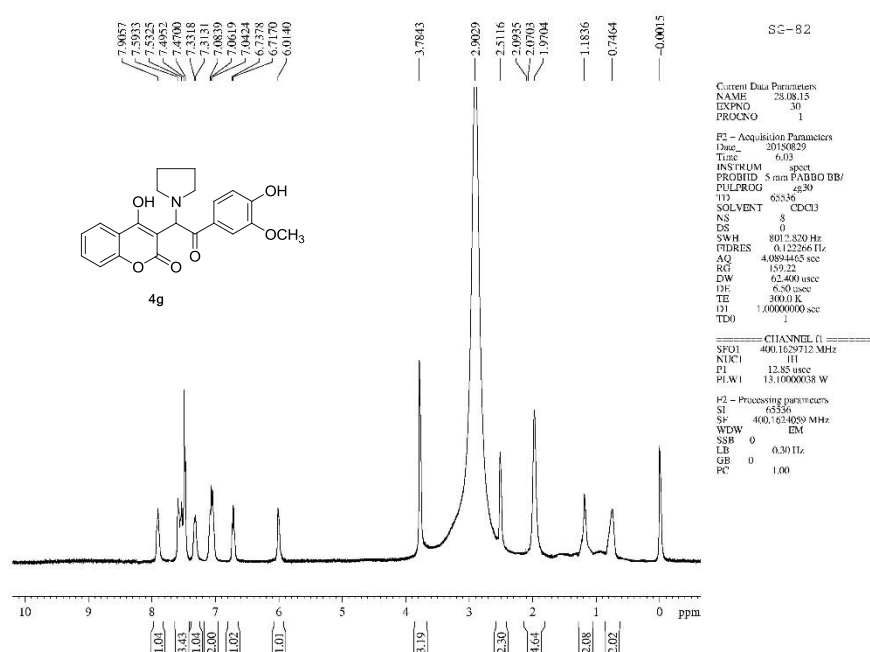
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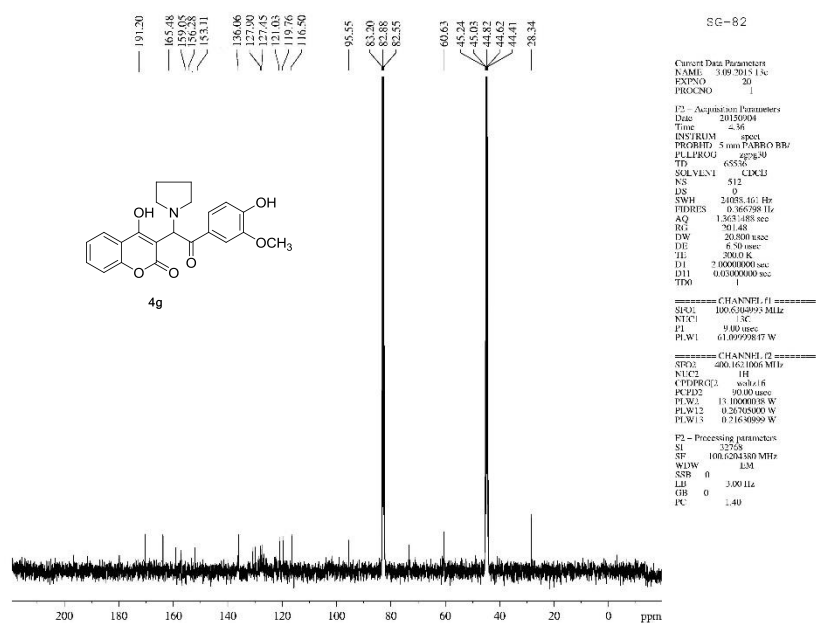
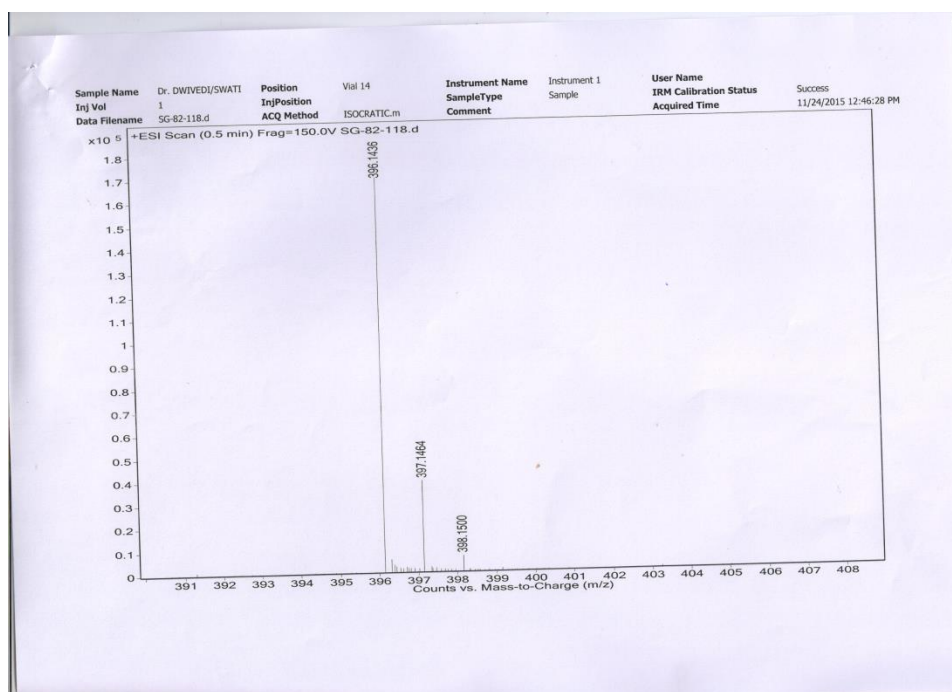
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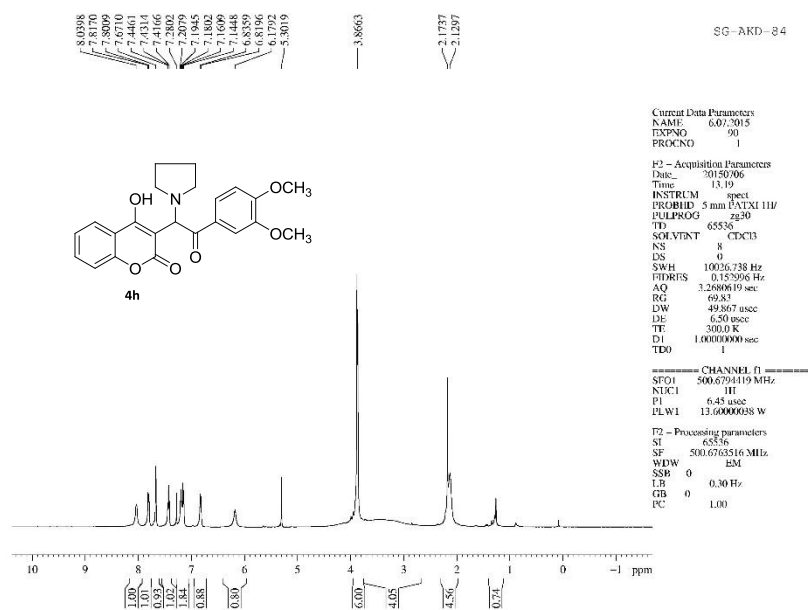
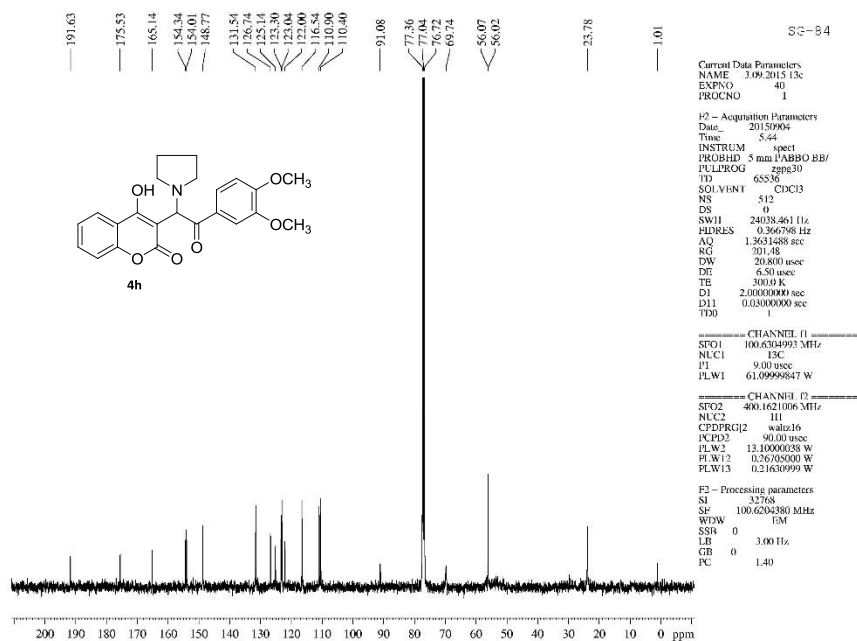


¹H NMR spectra of compound 4f:¹³C NMR spectra of compound 4f:

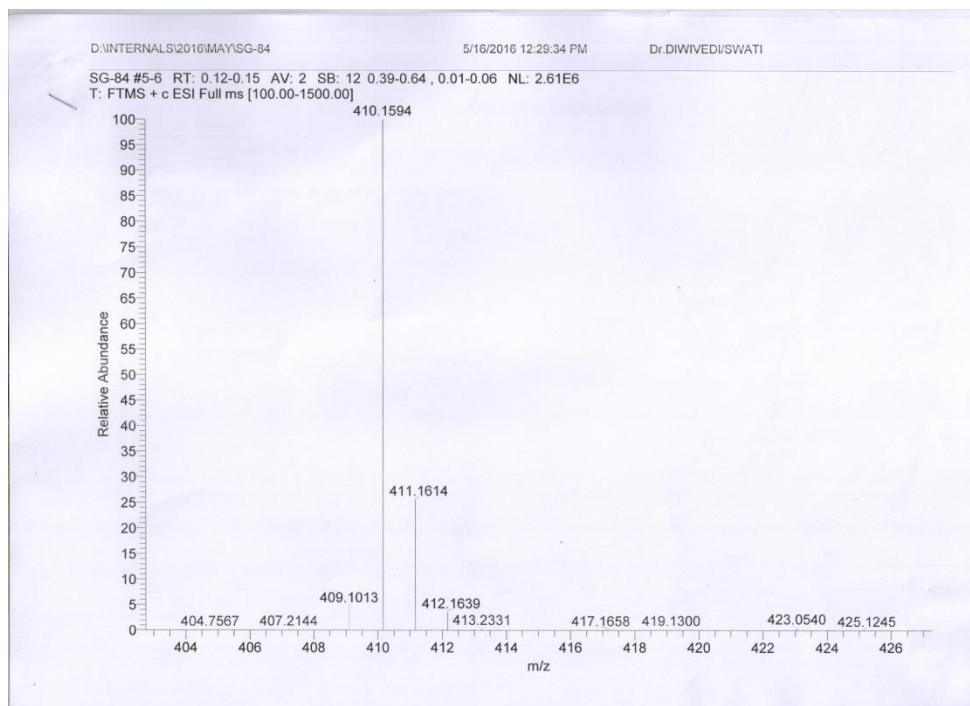
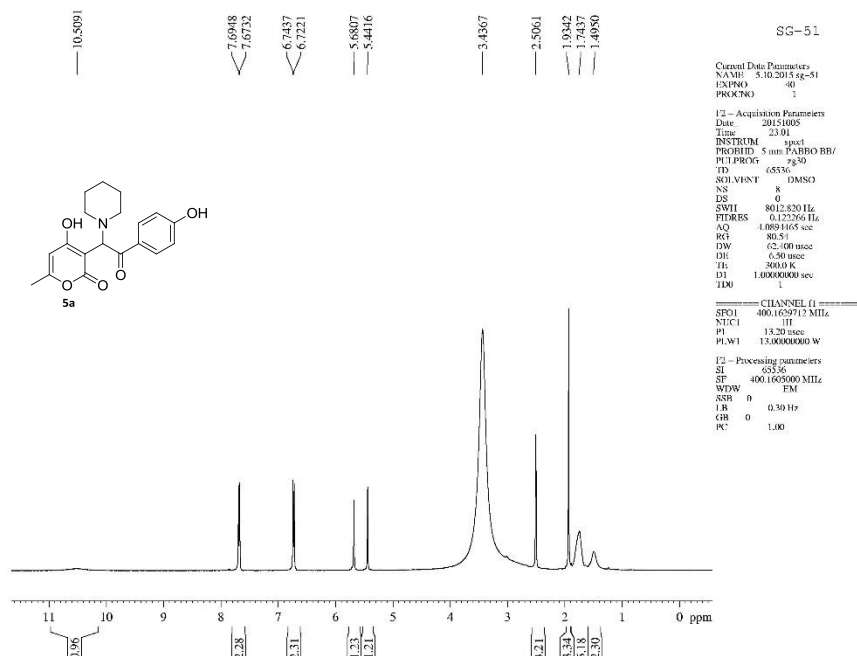
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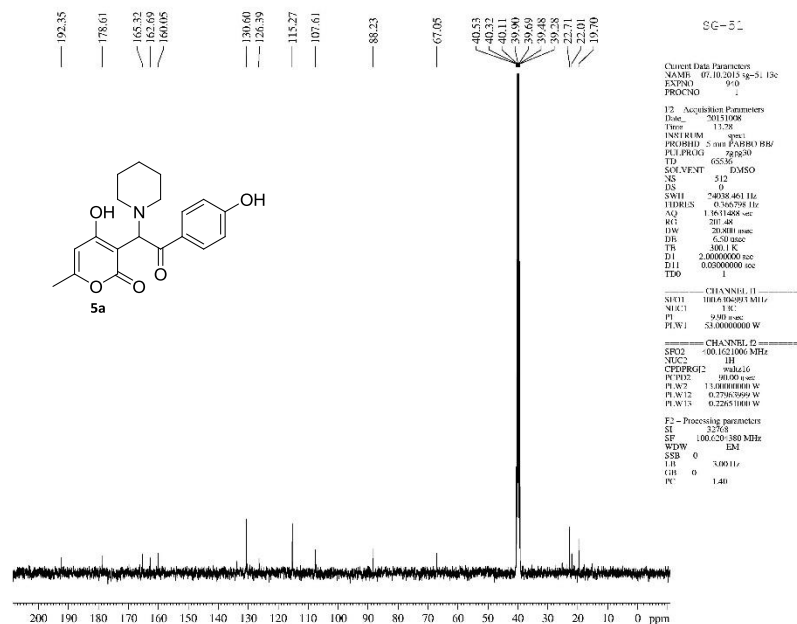
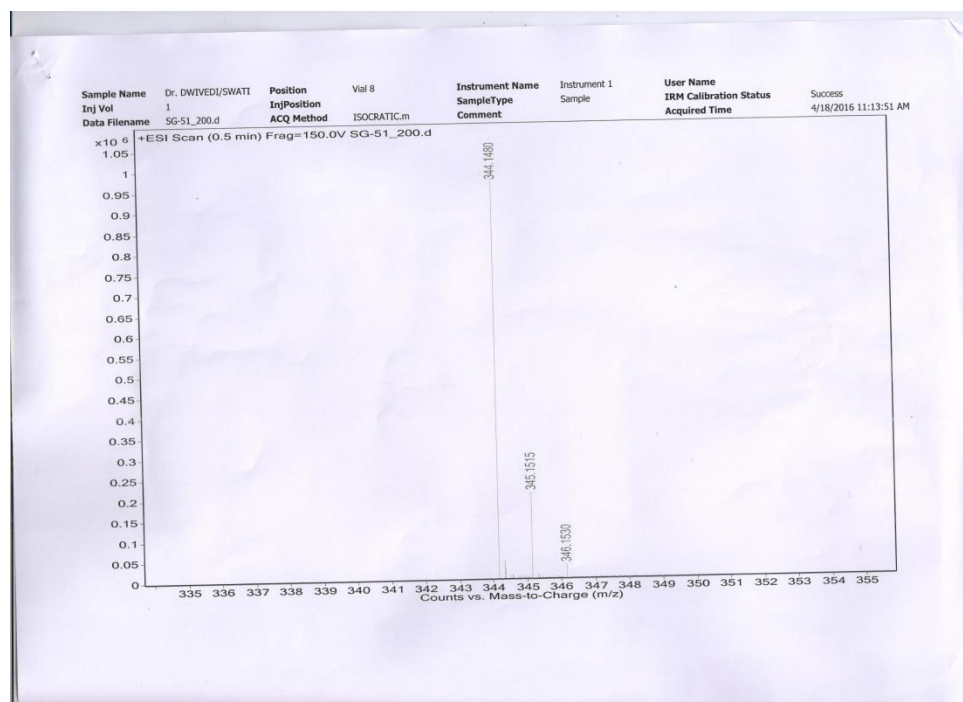
¹H NMR spectra of compound 4g:

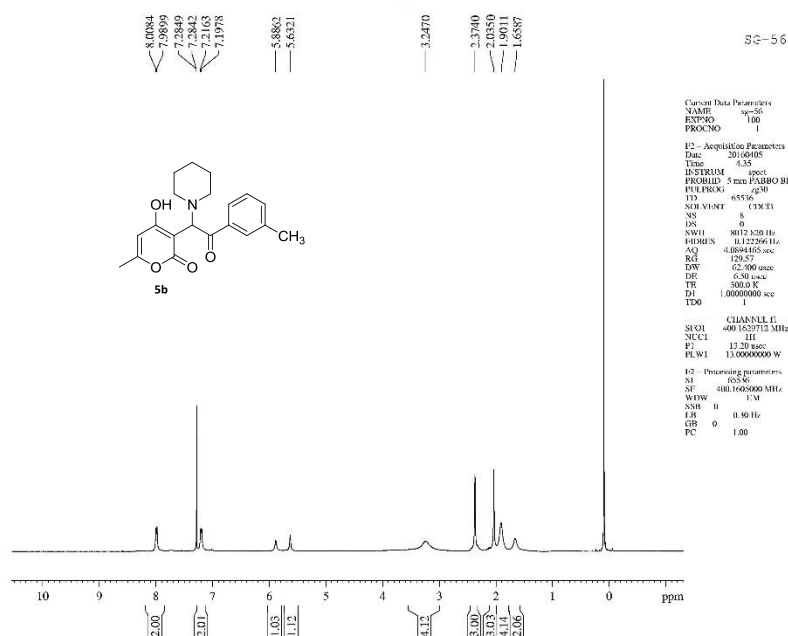
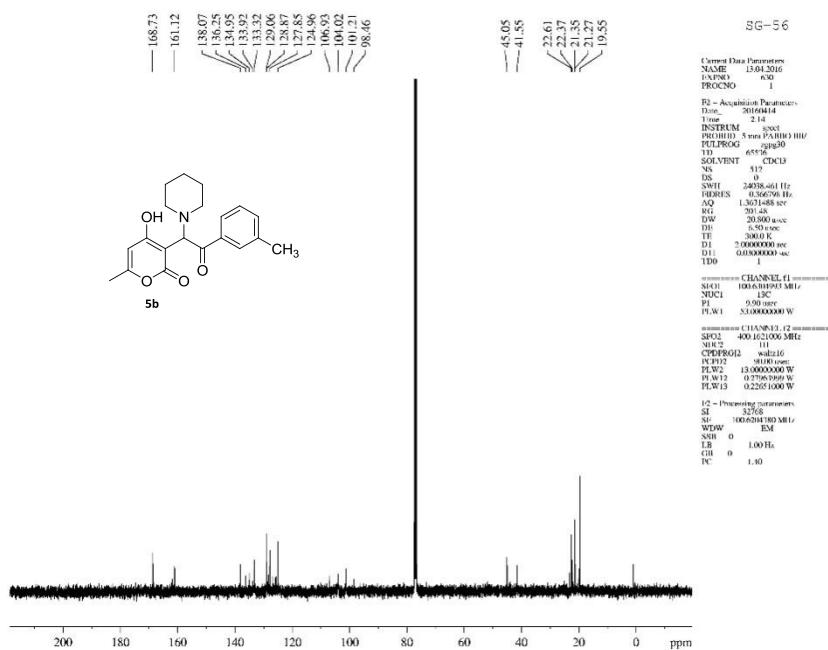
¹³C NMR spectra of compound 4g:**HRMS spectra of compound 4g:**

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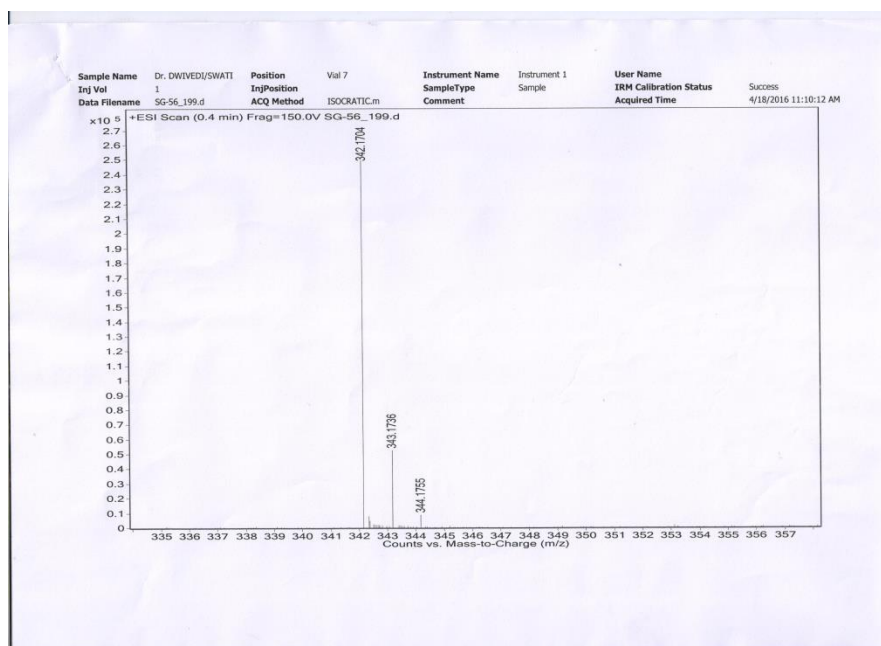
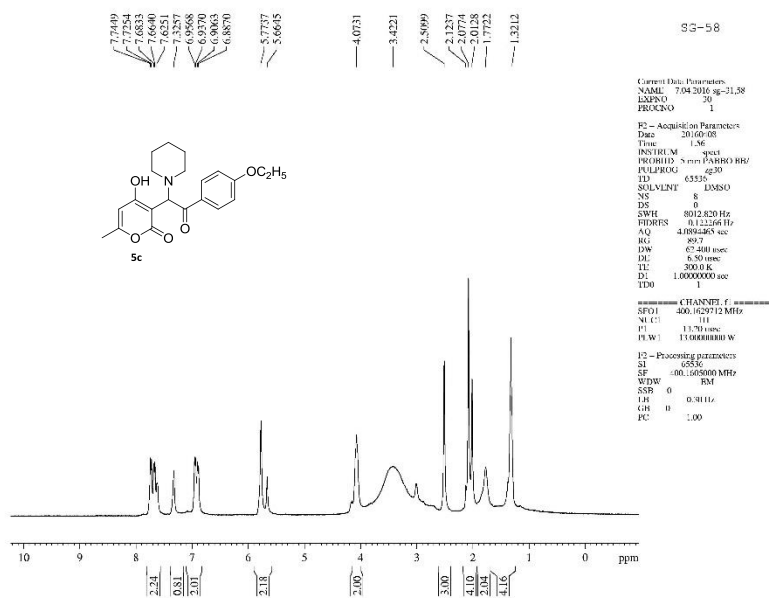
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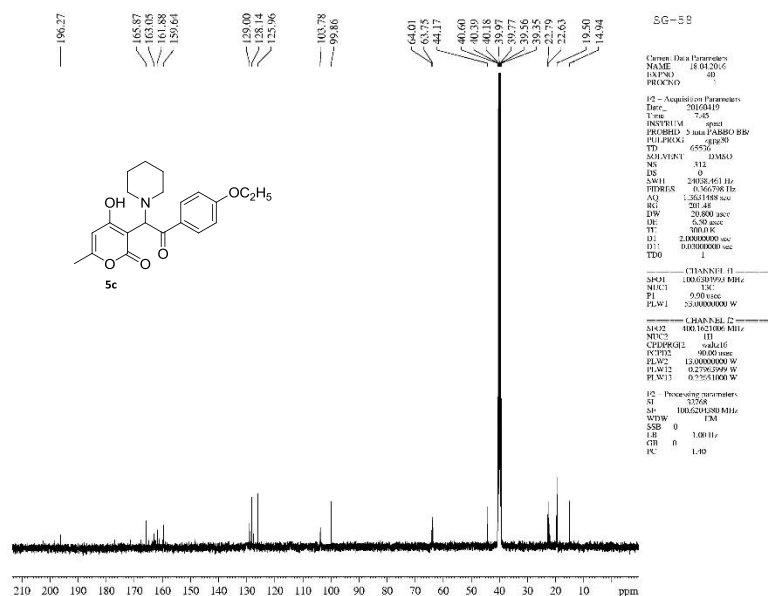
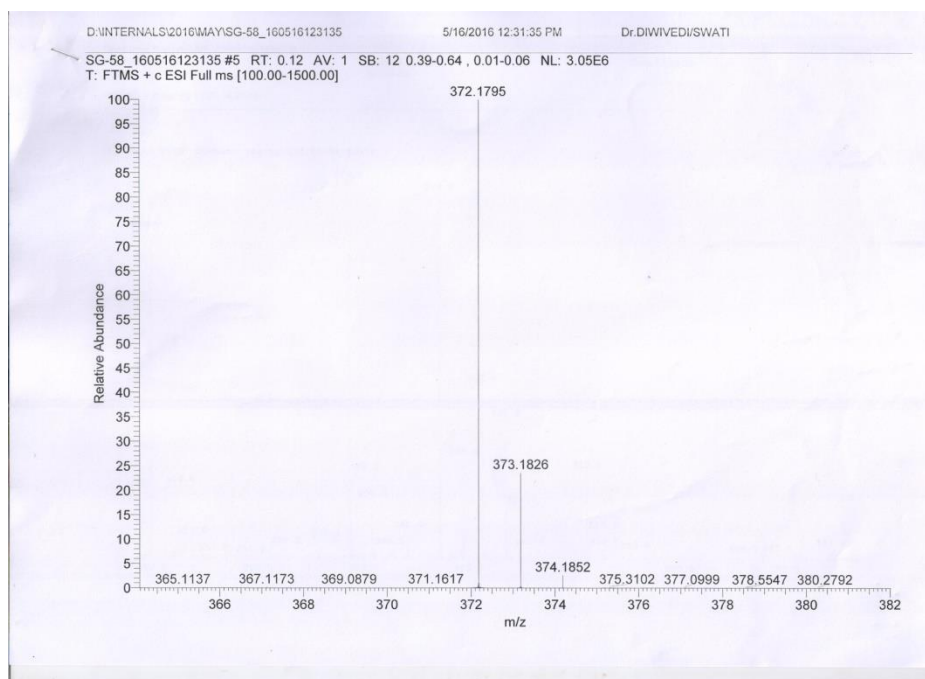
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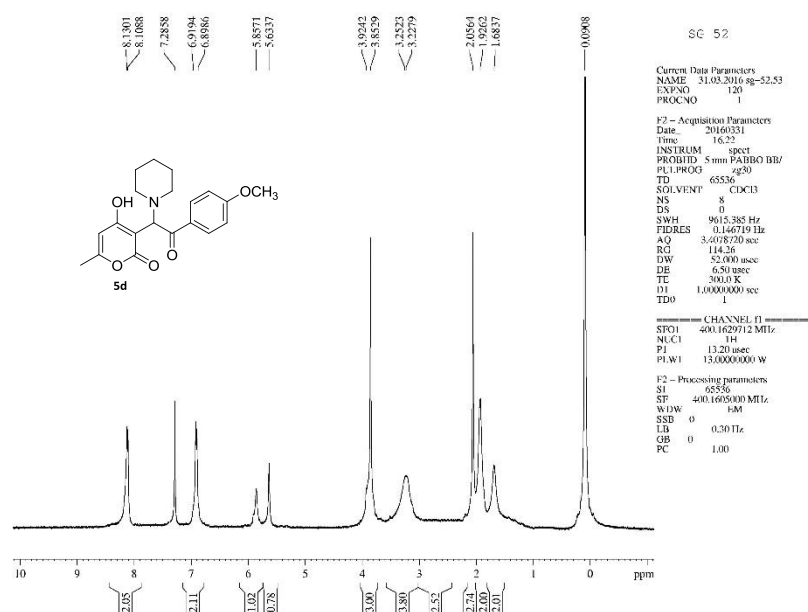
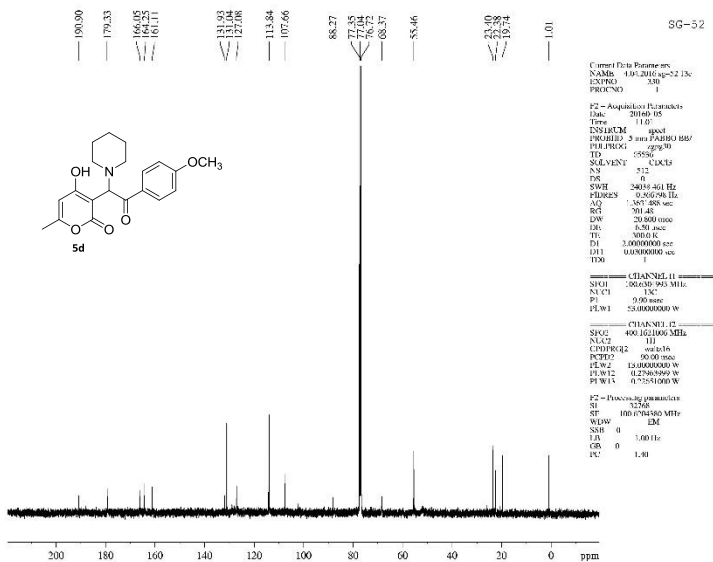
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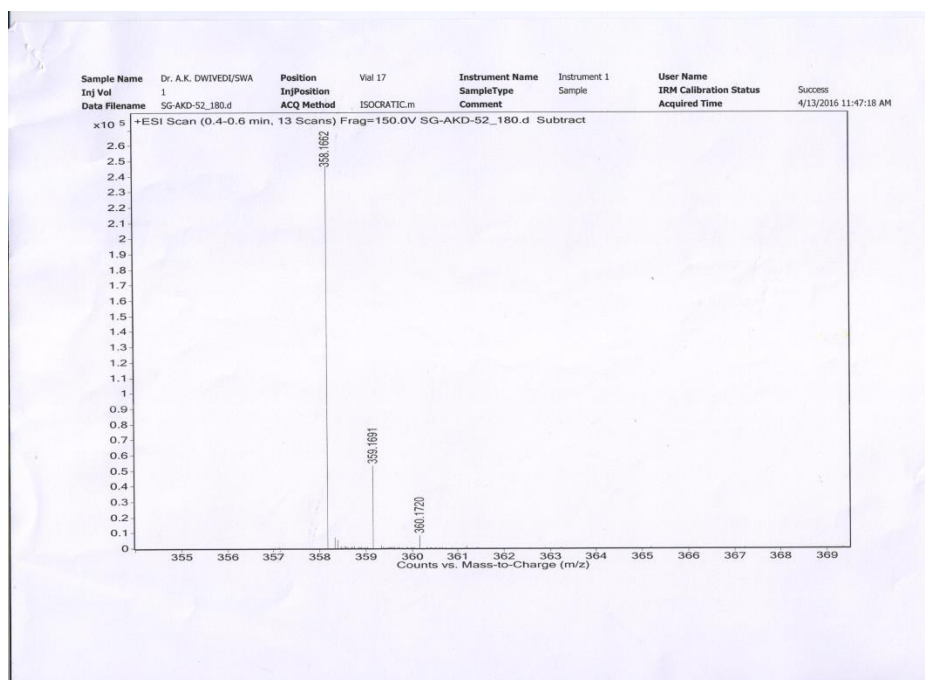
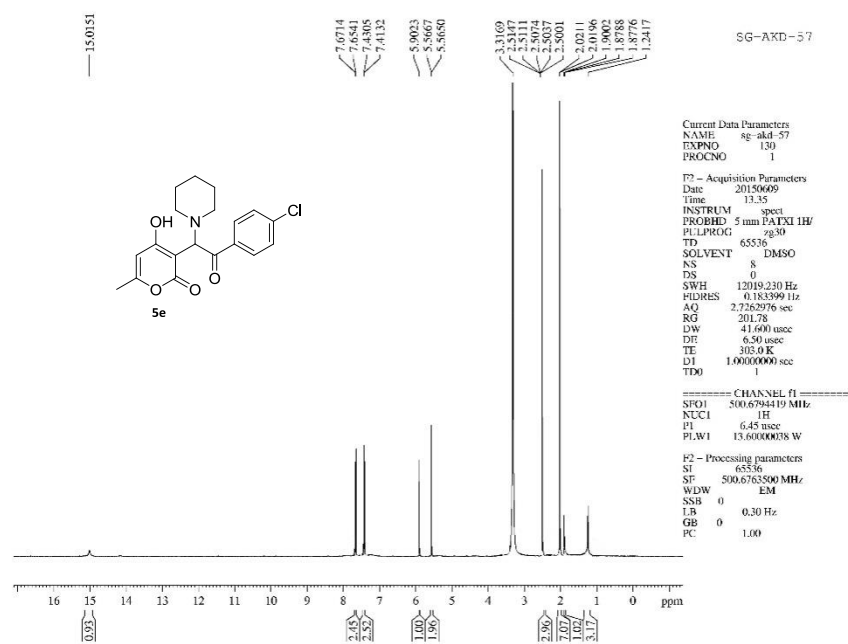
HRMS spectra of compound 5b:

 ^1H NMR spectra of compound 5c:

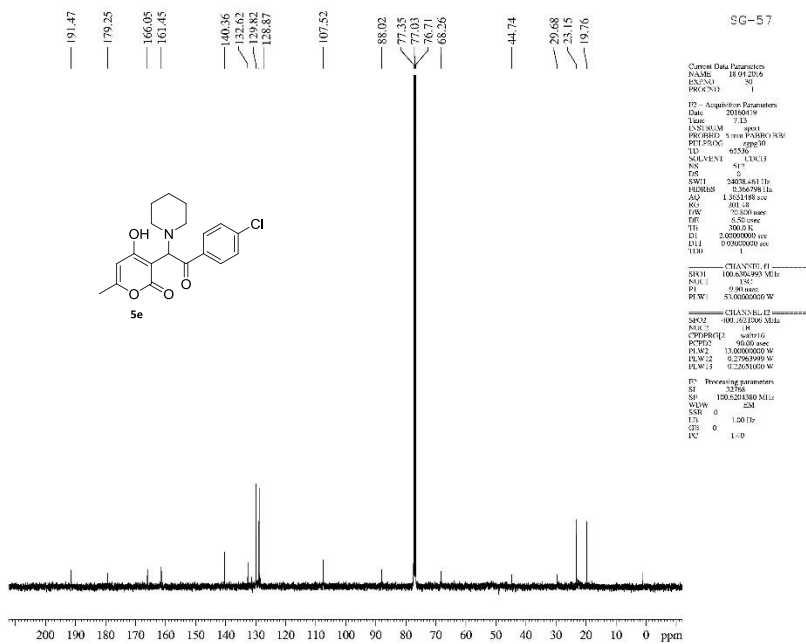
¹³C NMR spectra of compound 5c:**HRMS spectra of compound 5c:**

¹H NMR spectra of compound 5d:¹³C NMR spectra of compound 5d:

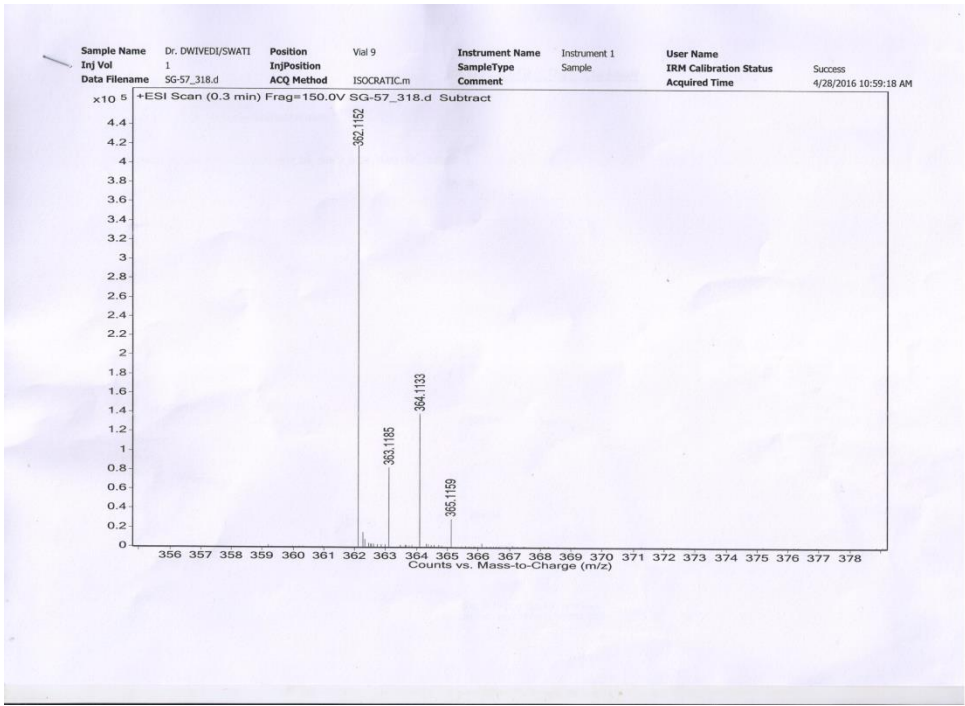
HRMS spectra of compound 5d:

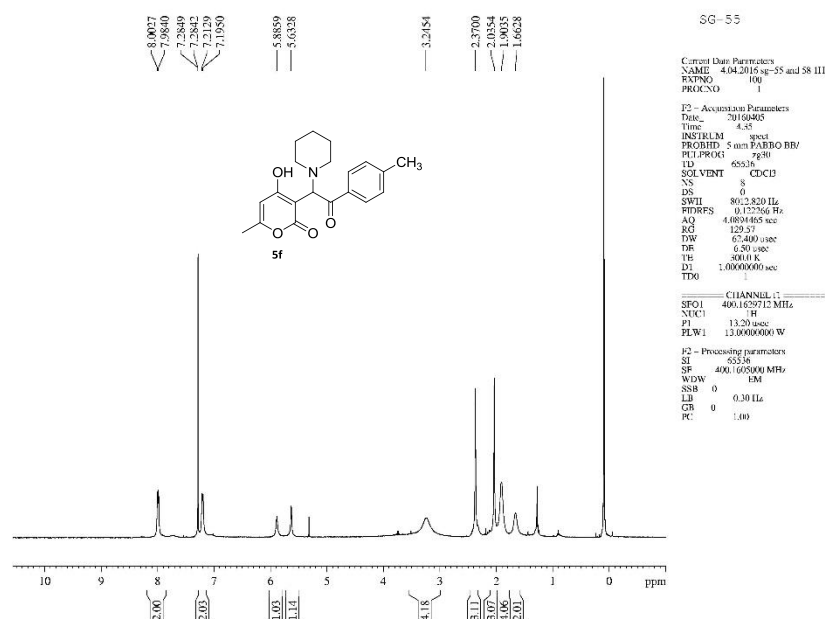
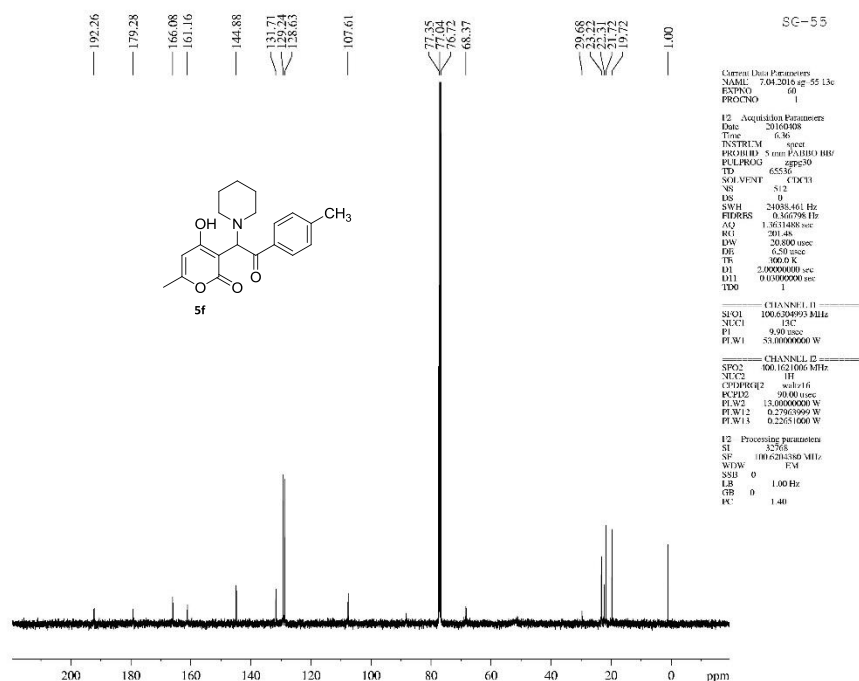
¹H NMR spectra of compound 5e:

¹³C NMR spectra of compound 5e:

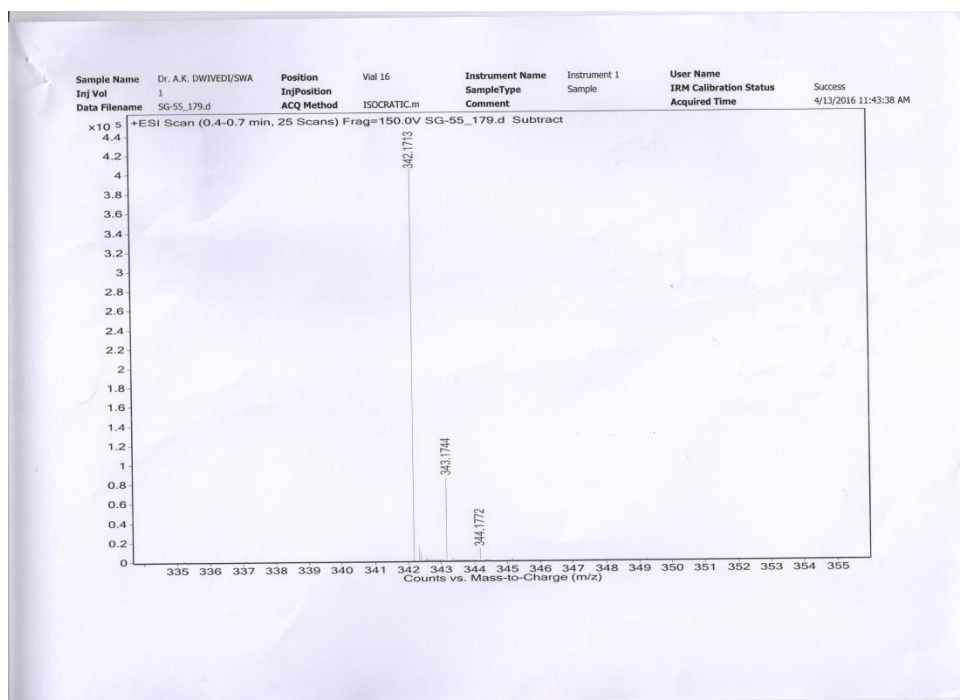


HRMS spectra of compound 5e:



¹H NMR spectra of compound 5f:¹³C NMR spectra of compound 5f:

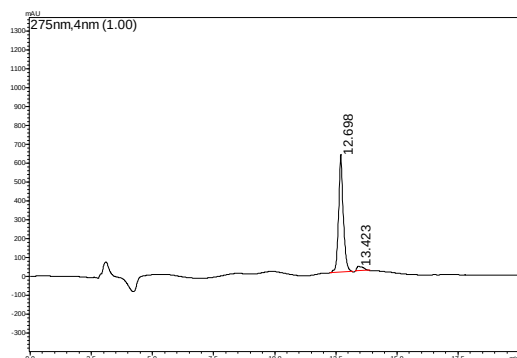
HRMS spectra of compound 5f:



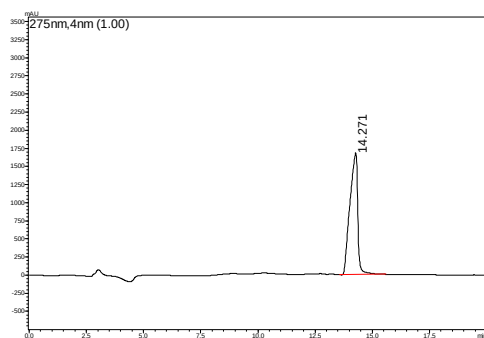
HPLC purity determination assay

Purity of compounds was determined by Reverse phase HPLC method using RP-18 Lichrocart[®], Merck column (5 μ m, 250 X 4.0 mm, id) under low pressure gradient condition using mobile phase Acetonitrile/water with 0.5% glacial acetic acid in the ratio between 10 :90 to 90:10, %v/vat a flow rate of 1.0 mL/min. The HPLC system (Shimadzu, Kyoto, Japan) equipped with Photo Diode Array detector (SPD-M20A), a pump (LC-20AD), controller (CBM-20A), Rheodyne (Cotati, CA, USA) model 7125 injector with a 20 μ l loop. Data was acquired in LC solution software (Shimadzu, Japan). compounds were monitored at 274 nm with a UV-Vis multiple wavelength detector. Mobile phase was filtered through 0.22- μ m Millipore filter (Billerica, USA) and degassed using ultrasonicator for 15 min before use.

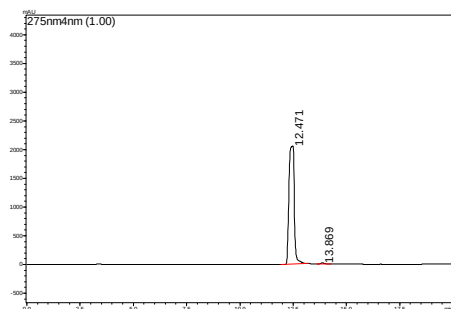
HPLC Chromatogram of compound 2a:



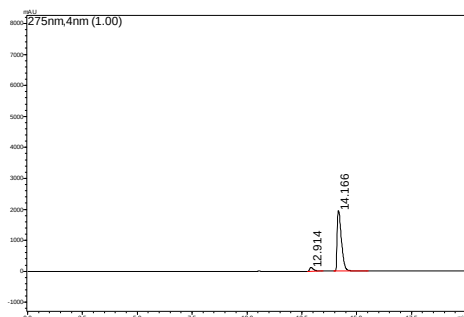
Peak no.	Retention time	Area%
1.	12.698	95.0123
2.	13.423	4.9877

HPLC Chromatogram of compound 2b:

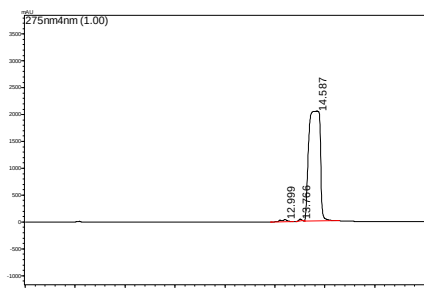
Peak no.	Retention time	Area%
1.	14.271	100

HPLC Chromatogram of compound 2c:

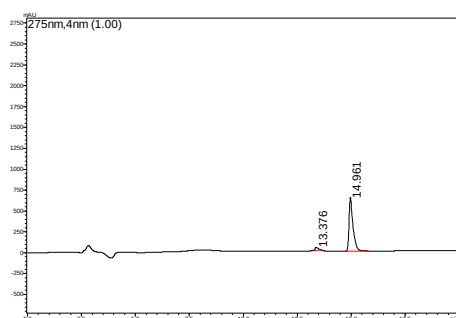
Peak no.	Retention time	Area%
1.	12.471	99.3582
2.	13.869	0.6418

HPLC Chromatogram of compound 2d:

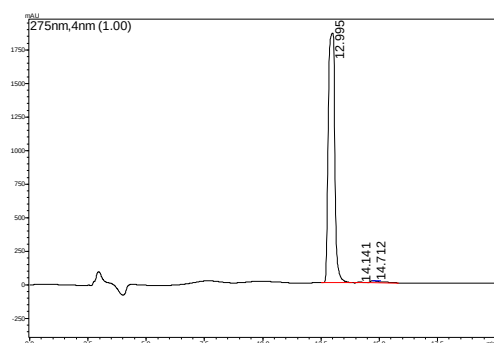
Peak no.	Retention time	Area%
1.	12.914	4.2882
2.	14.166	95.7118

HPLC Chromatogram of compound 2e:

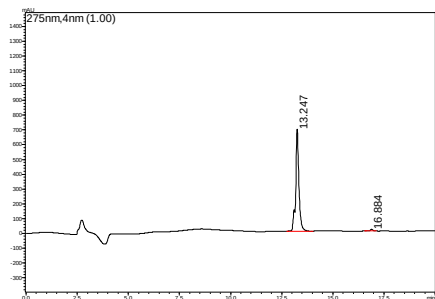
Peak no.	Retention time	Area%
1.	12.999	0.8738
2.	13.766	0.4368
3.	14.587	98.6893

HPLC Chromatogram of compound 2f:

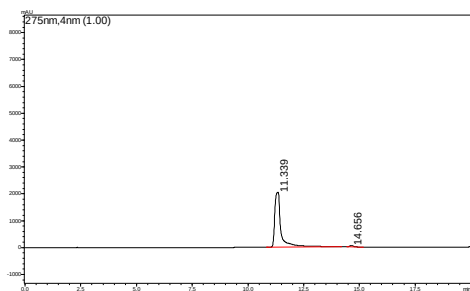
Peak no.	Retention time	Area%
1.	13.376	4.3516
2.	14.961	95.6484

HPLC Chromatogram of compound 2g:

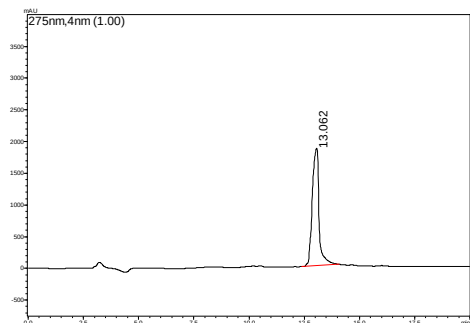
Peak no.	Retention time	Area%
1.	12.995	98.3201
2.	14.141	0.2342
3.	14.712	1.1983

HPLC Chromatogram of compound 2h:

Peak no.	Retention time	Area%
1.	12.383	1.0086
2.	13.23	97.4742
3.	14.562	1.5172

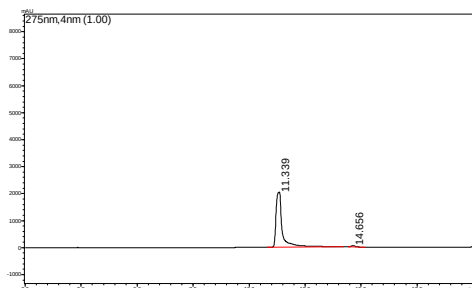
HPLC Chromatogram of compound 3a:

Peak no.	Retention time	Area%
1.	11.339	98.8874
2.	14.656	1.1126

HPLC Chromatogram of compound 3b:

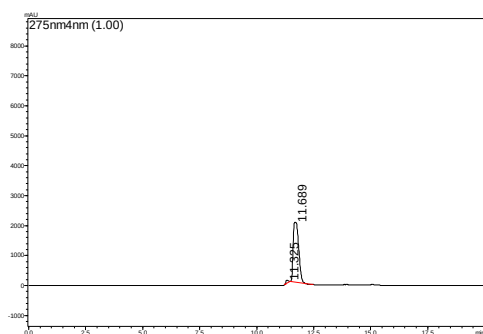
Peak no.	Retention time	Area%
1.	13.062	100

HPLC Chromatogram of compound 3c:



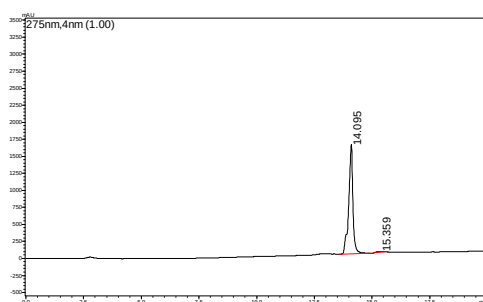
Peak no.	Retention time	Area%
1.	11.339	98.8874
2.	14.656	1.1126

HPLC Chromatogram of compound 3d:



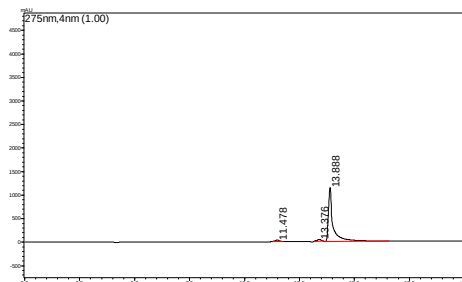
Peak no.	Retention time	Area%
1.	11.325	2.384
2.	11.689	97.616

HPLC Chromatogram of compound 3e:



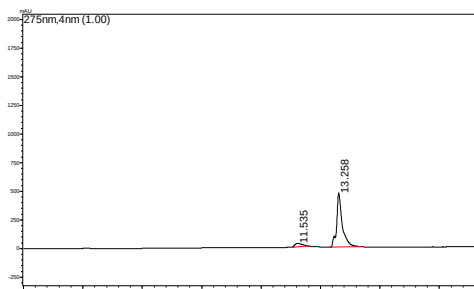
Peak no.	Retention time	Area%
1.	14.095	98.3623
2.	15.359	1.6377

HPLC Chromatogram of compound 3f:



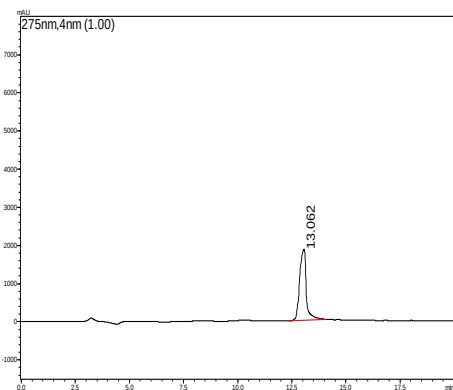
Peak no.	Retention time	Area%
1.	11.478	1.701
2.	13.376	3.1184
3.	13.888	95.1806

HPLC Chromatogram of compound 3g:



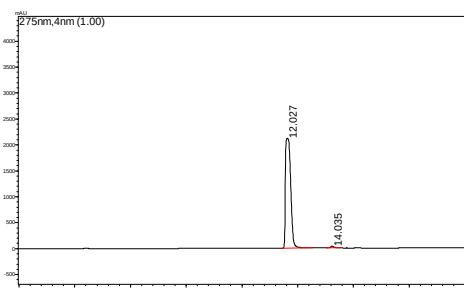
Peak no.	Retention time	Area%
1.	11.993	99.6571
2.	13.578	0.3429

HPLC Chromatogram of compound 3h:



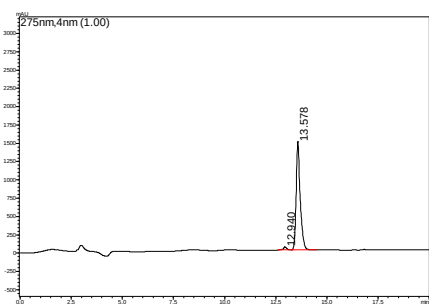
Peak no.	Retention time	Area%
1.	13.062	100

HPLC Chromatogram of compound 4a:



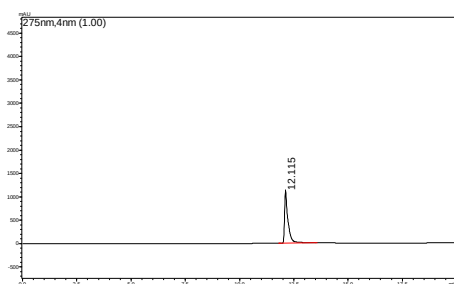
Peak no.	Retention time	Area%
1.	12.027	98.9693
2.	14.035	1.0307

HPLC Chromatogram of compound 4b:



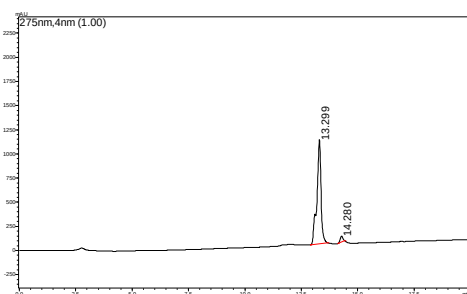
Peak no.	Retention time	Area%
1.	12.94	2.1801
2.	13.578	97.8199

HPLC Chromatogram of compound 4c:



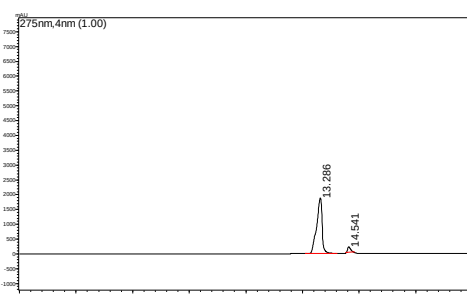
Peak no.	Retention time	Area%
1.	12.115	100

HPLC Chromatogram of compound 4d:



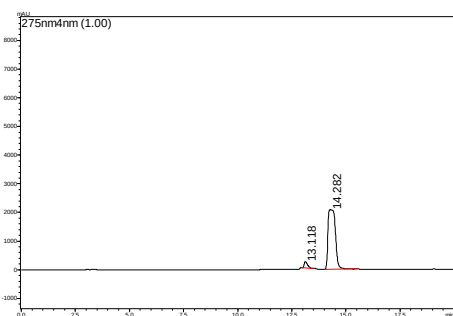
Peak no.	Retention time	Area%
1.	13.299	96.2584
2.	14.28	3.7416

HPLC Chromatogram of compound 4e:



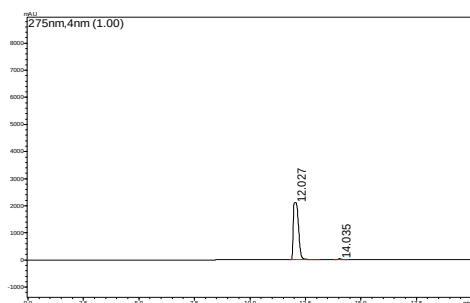
Peak no.	Retention time	Area%
1.	13.286	95.2063
2.	14.541	4.7937

HPLC Chromatogram of compound 4f:



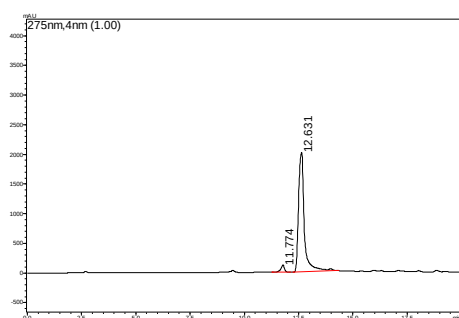
Peak no.	Retention time	Area%
1.	13.118	4.465
2.	14.282	95.535

HPLC Chromatogram of compound 4g:



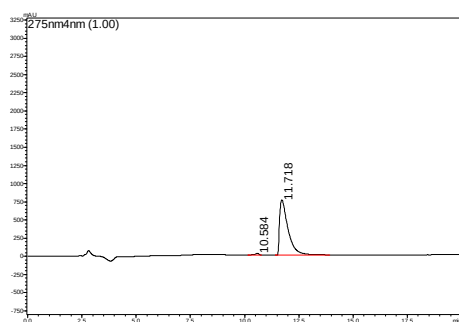
Peak no.	Retention time	Area%
1.	12.027	98.9693
2.	14.035	1.0307

HPLC Chromatogram of compound 4h:



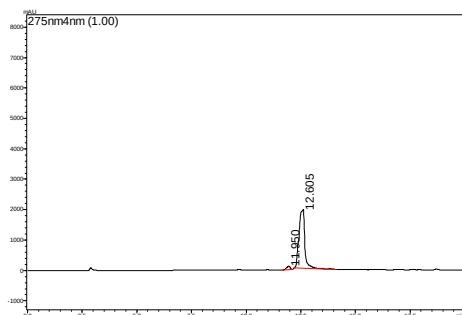
Peak no.	Retention time	Area%
1.	11.774	3.6956
2.	12.631	96.3044

HPLC Chromatogram of compound 5a:



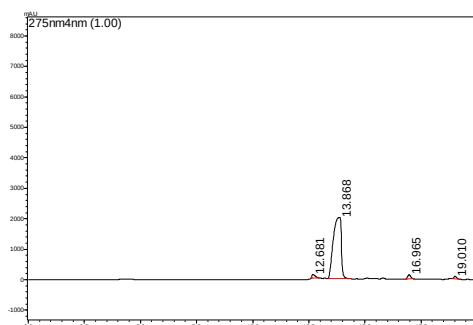
Peak no.	Retention time	Area%
1.	10.584	1.3536
2.	11.718	98.6464

HPLC Chromatogram of compound 5b:



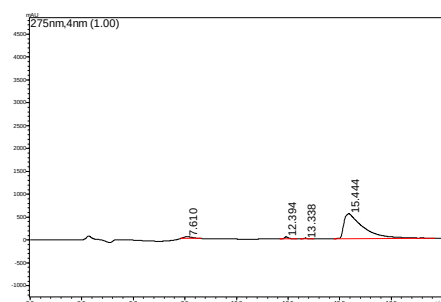
Peak no.	Retention time	Area%
1.	11.95	3.2661
2.	12.605	96.7339

HPLC Chromatogram of compound 5c:



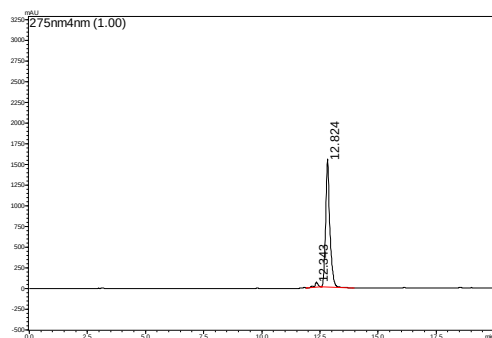
Peak no.	Retention time	Area%
1.	12.681	2.0039
2.	13.868	95.0504
3.	16.965	2.0446
4.	19.01	0.9011

HPLC Chromatogram of compound 5d:



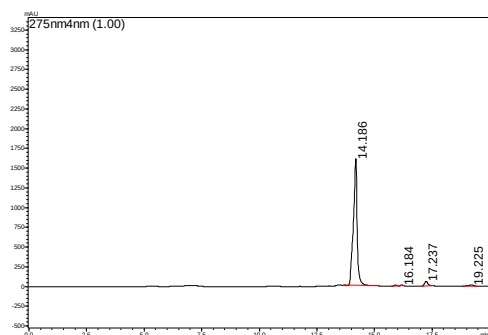
Peak no.	Retention time	Area%
1.	7.61	2.7642
2.	12.394	1.6246
3.	13.338	0.5484
4.	15.444	95.0628

HPLC Chromatogram of compound 5e:



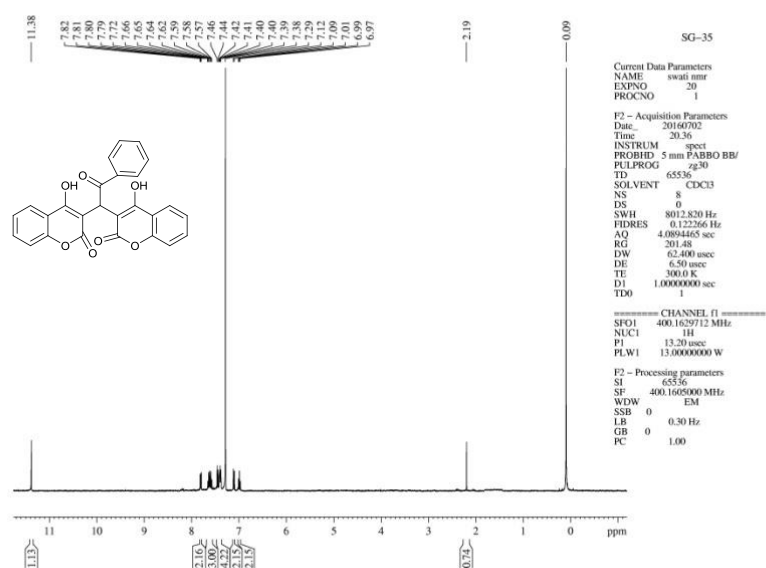
Peak no.	Retention time	Area%
1.	12.343	3.024
2.	12.824	96.976

HPLC Chromatogram of compound 5f:

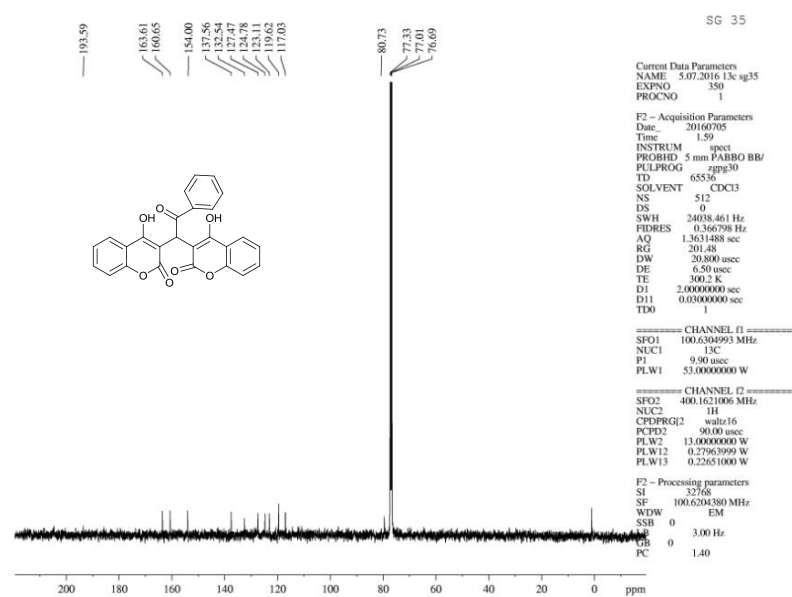


Peak no.	Retention time	Area%
1.	14.186	95.2955
2.	16.184	0.3994
3.	17.237	3.0847
4.	19.225	1.2205

¹H NMR spectra of dicoumarol:



¹³C NMR spectra of dicoumarol:



Mass spectra of dicoumarol:

