



AN OXAZINO QUINOLINE DERIVATIVES COMBAT AGAINST HIV VIRUS SYNTHESISED BY USING A MAGNETIC NANO COPPER FERRITE AS CATALYST UNDER ULTRASONICATION

Sruthi Vasamsetty¹, Swathi Bandaru², Paul Douglas Sanasi³

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Abstract:

These one-pot synthesised 4H-oxazino quinoline derivatives are prepared through cyclisation of aryl aldehyde, different substituted amides and 8-hydroxy quinoline under ultrasonication is described. The formed compounds are characterised by FT-IR, HRMS, ¹H NMR and ¹³C NMR, further concealed biological activity studies.

Keywords: 4H-oxazino quinoline derivatives, Heterogeneous nano copper ferrite catalyst, biological activity, Ultrasonication.

¹Department of BS&H, Vignan Institute of Information Technology, Visakhapatnam, Andhra Pradesh, INDIA-530049,

²Department of Chemistry, Rajiv Gandhi University of Knowledge technologies-Srikakulam, Andhra Pradesh, INDIA-532410,

³Department of Engineering Chemistr, AU College of Engineering (A), Andhra University, Visakhapatnam, Andhra Pradesh, INDIA-530003

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1. Introduction

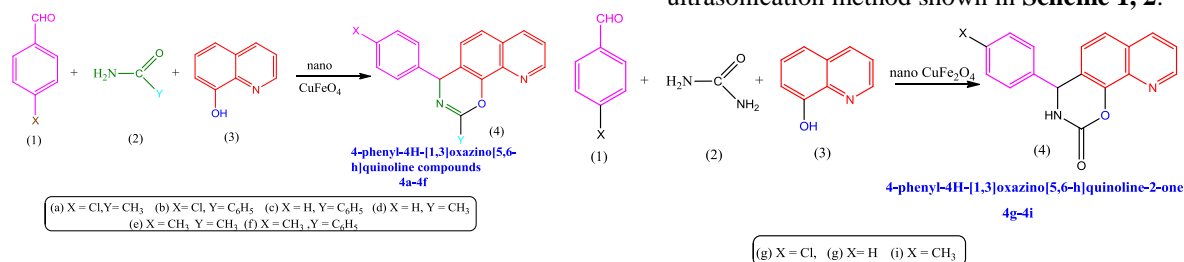
From the last decade, one pot synthesis of various reactions take part in organic synthesis. In which the reactants are subjected into a single container, to form corresponding products with high yields.[1-3]

Quinoline moieties have medicinal applications such as antihistamatic, antimalarial, and antiinflammatory.[4] Similarly Oxazinone derivatives have a property, to fight against HIV virus.[5] From the literature survey, different methods have been reported for the synthesis of oxazino quinoline derivatives by using various solvents like p-TSA [6], H₂O/TEBA [7], Toulene [8], N,N-dimethylformamide [9] Sulphonic acid-carbon [10], Phosphonitrilic chlorides [11], Citric acid [12], MgSO₄ [13], nano TiCl₄.SiO₂ [14], Magnesium perchlorate [15], L-pyrrolidine-2-carboxylic acid-4-hydrogen sulphate [16], N-bromophthalimide [17], SnCl₄ [18], Succinic acid [19], Bronsted acidic ionic liquid [20], P₂O₅ on solid support [21], H₃BO₃ [22], Caros acid/silica gel [23], nano silica supported boron trifluoride [24], poly ethylene

glycol based ionic liquid [25], triphenylphosphene Ruthenium chloride [26] and methane sulphonic acid[27]. The disadvantages of the above procedures like longer reaction times [26], using toxic reagents [15], [19], [20], [22]. In this method, organic scaffolds can be prepared by using milder reaction conditions to get higher yields.

Though these methods involve their own like long interval times [26], using toxic reagents and catalyst recovery [15], [17], [19], [20]. The main attention on our research is the use of cubic magnetic nano ferrite catalysts in organic synthesis. Very previously, different heterocyclic compounds are reported by usage of nano copper ferrite. [28], [29], [30], [31], [32].

Now we are reporting an efficient synthesis of 4-phenyl-4H-[1,3]oxazino[5,6-h]quinoline and 4-phenyl-4H-[1,3]oxazino[5,6-h]quinoline-2-one through cyclization of substituted aromatic aldehyde, various amides and 8-hydroxy quinoline in presence of a separable cubic nano copper ferrite catalyst under



Scheme 1: Synthesis of oxazino quinoline derivatives catalyzed by nano copper ferrite
Scheme 2: Synthesis of Oxazino quinoline 2-one derivatives catalyzed by nano copper ferrite

EXPERIMENTAL PROCEDURE

All chemicals used in this process are of pure and elegant. The synthesized oxazino quinolines were screened by FTIR, MASS, ¹H NMR and ¹³C NMR and their biological activities are characterised by using microbial assays.

GENERAL PROCEDURE

500 mg of catalyst was taken in a microwave consistar, irradiated at 500 °C for 2 minutes and cooled to room temperature before processing of experiment. The one-pot synthesis of oxazino quinoline derivatives was carried out in 100ml beaker taking of aromatic aldehydes (10 mmol), substituted amide (10 mmol), 8-hydroxyquinoline (10 mmol) and activated nano copper ferrite catalyst were mixed together and made to dissolve in 15 ml of ethanol and placed in sonicator bath for the reaction to proceed. The

advancement and completion of the reaction was monitored by TLC technique. The product was isolated from the catalyst magnetically from the mixture. So,formed product is characterized by various spectral techniques.

2. Results and Discussion

Here, 500 mg of nano CuFe₂O₄ catalyst was enough to obtain 94% yield of oxazino quinoline derivatives. Mostly aromatic aldehydes consisting of electron withdrawing groups have high reactivity than the electron donating groups. Whenever the substituent might be present on the ring, it does not have any impact on the yield of the product. It is also understood that benzamide shows faster rate of condensation with aldehyde than the acetamide shown in **Table 1**.

Table 1: Synthesis of oxazino quinoline and oxazino quinoline-2-one derivatives in presence and absence of nano CuFe₂O₄ catalyst :

S.no	X in aldehyde	Y in amide	Product	Tim (min) with catalyst (500mg)		Time(min) without catalyst		Yield
				sonication	reflux	sonication	reflux	
1	Cl	CH ₃	4a	9	30	18	45	94
2	Cl	C ₆ H ₅	4b	7	25	15	40	95
3	H	C ₆ H ₅	4c	10	35	15	45	93
4	H	CH ₃	4d	13	40	18	55	92
5	CH ₃	CH ₃	4e	15	45	18	60	85
6	CH ₃	C ₆ H ₅	4f	13	38	15	55	91
7	Cl	NH ₂	4g	10	35	12	50	89
8	H	NH ₂	4h	12	40	15	60	87
9	CH ₃	NH ₂	4i	15	45	18	60	84

Differentiation of nano copper ferrite catalyst with other Catalysts shown in Table 2

It is observed that under reflux conditions the reaction processed time is immensely high. Using nano CuFe₂O₄ catalyst in the synthesis of above

mentioned derivatives by ultrasonication method is too low, when compared to the reactions catalysed by p-toulenesulphonicacid[5], Triethylbenzylammoniumchloride[6].

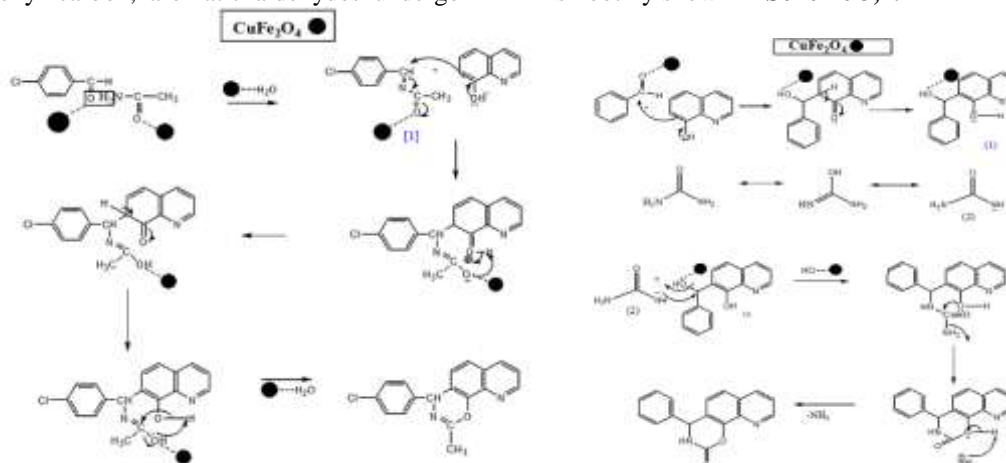
Table 2: Comparative study of nano CuFe₂O₄ catalyst with other catalysts:

S.No	various catalyst	solvent	time	Product yield	Ref No
1	Triethylbenzylammoniumchloride	water	4 hrs(reflux)	85	[6]
2	L-pyrrolidine-2-carboxylicacid-4-hydrogen sulfate	Solvent free	3 hrs(reflux)	94	[14]
3	Magnesiumperchlorate	Solvent free	40min(reflux)	92	[15]
4	N-bromophthalimide	Solvent free	160min(reflux)	75	[16]
5	nano silica supported boron trifluoride	ethyl acetate	120min(reflux)	98	[24]
6	Triphenylphosphene Ruthenium Chloride	toluene	12hrs(reflux)	85	[26]

Plausible mechanism

Here oxide (o⁻²) of the copper ferrite nanoparticles acts as a lewis base and Fe³⁺ as a lewis acid coordinates with the carbonyl oxygen atom of aldehydes, thus increasing the electrophilicity of the carbonyl carbon, aromatic aldehydes undergo

condensation reaction with substituted amides to form an intermediate iminium ion [1]. This intermediate undergoes nucleophilic addition with 8-hydroxy quinoline followed by condensation to form tetra hydro oxazino quinoline derivatives smoothly shown in **Scheme 3,4**.



Scheme 3,4 : Plausible mechanism of oxazino quinoline derivatives

Spectral data of synthesised compounds

4-(4-chlorophenyl)-2-methyl-4H-[1,3]oxazino[5,6-h]quinoline (4a)

White solid, IR (cm⁻¹): 3047(CH str), 1505(-C=C-str), 1167(-C-O-C str); ¹HNMR (δ/ppm): 8.8-8.7 (m, Ar-H), 8.2-8.1 (m, Ar-H), 7.48-7.46 (m, Ar-H), 7.44-7.42 (m, Ar-H), 7.41-7.40 (m, Ar-H), 7.34-7.32 (m, Ar-H), 7.27-7.26 (m, Ar-H), 7.20-7.19 (m, Ar-H), 4.1 (s, 1H), 2.1 (s, methyl proton); Mass m/z [M]=308, [M+1]=309, ¹³C NMR (ppm) 166.68, 152.44, 148.06, 140.33, 138.47, 136.33, 128.70, 128.40, 127.91, 121.97, 118.06, 110.33, 52.20, 21.1, 94% yield

4-(4-chlorophenyl)-2-phenyl-4H-[1,3]oxazino[5,6-h]quinoline (4b)

White solid, IR (cm⁻¹): 3057(CH str), 1578(-C=C-str), 1143 (-C-O-C str); ¹HNMR (δ/ppm): 8.737-8.730 (m, Ar-H), 8.17-8.15 (m, Ar-H), 8.06 (m, Ar-H), 7.88-7.86 (m, Ar-H), 7.82 (d, Ar-H), 7.54-7.50 (m, Ar-H), 7.48-7.45 (m, Ar-H), 7.44-7.43 (m, Ar-H), 7.38-7.36 (m, Ar-H), 7.34-7.30 (m, Ar-H), 7.26-7.24 (m, Ar-H), 6.71-6.69 (m, Ar-H), 5.7 (s, 1H); Mass: m/z [M]=370, [M+1]=371, ¹³CNMR (ppm), 166.68, 149.38, 148.54, 140.26, 138.52, 136.37, 134.46, 131.83, 128.79, 128.73, 128.51, 128.03, 27.29, 122.30, 122.08, 118.57, 55.4, 95% yield

2,4-diphenyl-4H-[1,3]oxazino[5,6-h]quinoline (4c)

White solid, IR (cm⁻¹): 3062 (CH str), 1578(-C=C-str), 1282(-C-O-C str), 1097(-C=N str); ¹HNMR (δ/ppm): 8.8-8.7 (m, Ar-H), 8.13-8.12 (m, Ar-H), 8.06(m, Ar-H), 7.89-7.87(m, Ar-H), 7.82- 7.80 (m, Ar-H), 7.5-7.4 (m, Ar-H), 7.44-7.43(m, Ar-H), 7.3-7.2(m, Ar-H), 7.389-7.36(m, Ar-H), 7.3-7.4(m, Ar-H), 7.2-7.3(m, Ar-H), 5.7(s, 1H); Mass: m/z [M]=336, [M+1]=337, ¹³CNMR(ppm), 166.68, 149.38, 148.54, 140.26, 138.52, 136.37, 134.46, 131.83, 128.79, 128.73, 128.51, 128.03, 127.29, 122.30, 122.08, 118.57, 55.24, 93% yield.

2-methyl-4phenyl 4H-[1,3]oxazino[5,6-h]quinoline (4d)

White solid, IR (cm⁻¹): 2927 (CH str), 1640 (C=C, str), 1281(-C-O-C str), 1097(-C=N str); ¹HNMR (δ/ppm): 8.79-8.71 (m, Ar-H), 8.785-8.781 (m, Ar-H), 8.169-8.165 (m, Ar-H), 8.148-8.145 (m, Ar-H), 7.47.41 (m, Ar-H), 7.36-7.32 (d, Ar-H), 7.325-7.20 (m, Ar-H), 7.20-7.18 (m, Ar-H), 5.1 (s, 1H), 2.1 (s, methyl proton); Mass: m/z [M]=274, [M+1]=275, ¹³CNMR (ppm), 166.63, 149.40, 142.40, 138.56, 136.31, 134.68, 131.69, 128.83, 128.74, 128.70, 127.94, 127.47, 127.30, 127.11, 122.60, 122.12, 118.36, 55.67, 22.60, 92% yield

2-methyl-4-p-tolyl-4H-[1,3]oxazino[5,6-h]quinoline (4e)

White solid, IR (cm⁻¹): 3048(CH str), 1578(-C=C-str), 1224(-C-O-C str), 1138(-C=N str); ¹HNMR (δ/ppm): 8.79 (m, Ar-H), 8.789 (m, Ar-H), 8.156 (m, Ar-H), 8.135 (m, Ar-H), 7.48-7.44 (m, Ar-H), 7.43-7.40 (d, Ar-H), 7.34-7.32 (m, Ar-H), 7.21-7.198 (m, Ar-H), 4.1 (s, 1H), 2.3 (s, methyl proton), 2.4 (s, methyl proton); Mass: m/z [M]=288, [M+1]=289, ¹³CNMR (ppm), 166.68, 152.42, 148.08, 138.50, 136.21, 128.70, 127.92, 122.00, 118.06, 110.20, 54.85, 21.21, 85% yield

2-phenyl-4-p-tolyl-4H-[1,3]Oxazino[5,6-h]quinoline (4f)

White solid, IR (cm⁻¹): 2777(CH str), 1576(-C=C-str), 1295(-C-O-C str), 1121(-C=N str); ¹HNMR (δ/ppm): 8.781 (m, Ar-H), 8.77 (m, Ar-H), 8.169 (m, Ar-H), 8.148 (m, Ar-H), 7.97 (m, Ar-H), 7.82-7.80 (d, Ar-H), 7.53 (m, Ar-H), 7.52-7.51 (m, Ar-H), 7.49 (m, Ar-H), 7.47 (m, Ar-H), 7.44 (m, Ar-H), 7.42-7.40 (m, Ar-H), 7.34-7.32 (m, Ar-H), 7.2-7.1 (m, Ar-H), 5.5 (s, 1H), 2.4(s, methyl proton); Mass [M]=350, [M+1]=351, ¹³CNMR (ppm), 166.68, 152.44, 148.06, 140.33, 138.47, 136.33, 128.70, 128.40, 127.91, 121.97, 118.06, 110.33, 52.20, 21.01, 91% yield.

4-(4-chlorophenyl)-3,4-dihydro-[1,3]oxazino[5,6-h]quinoline-2-one (4g)

White solid, IR (cm⁻¹): 3401(NH str), 1634(-C=O str), 1564(-C-C= str), 1276 (-C-O-C str) 1091(-C=N str); ¹HNMR(δ/ ppm): 8.75 (m, Ar-H), 8.70 (m, Ar-H), 8.672 (m, Ar-H), 8.163 (m, Ar-H), 7.84-7.82 (m, Ar-H), 7.53-7.51 (m, Ar-H), 7.45 (m, Ar-H), 7.43-7.39 (m, Ar-H), 7.32-7.30 (m, Ar-H), 7.21-7.19 (m, Ar-H), 6.37-6.30 (m, Ar-H), 5.2 (s, 1H), 9.9(s, NH), Mass [M]=310, [M+1]=311, ¹³CNMR (ppm), 166.68, 156.29, 152.44, 148.06, 138.46, 136.29, 128.70, 127.88, 121.93, 118.04, 110.33, 56.29, 89% yield.

4-phenyl-3,4-dihydro-[1,3]oxazino[5,6-h]quinoline-2-one (4h)

White solid, IR(cm⁻¹): 3213(NH str), 1673(-C=O str), 1506(-C-C= str), 1285(-C-O-C str), 1093(-C=N str), ¹HNMR(δ/ppm): 8.79-8.78 (m, Ar-H), 8.77-8.76 (m, Ar-H), 8.77-8.76 (m, Ar-H), 8.175-8.172 (d, Ar-H), 8.155-8.151 (m, Ar-H), 7.40-7.43 (m, Ar-H), 7.33-7.32 (m, Ar-H), 7.19-7.1 (m, Ar-H), 7.427 (s, NH), 5.96 (s, 1H). Mass [M]=276, [M+1]=277, ¹³CNMR (ppm), 166.68, 152.44, 148.06, 147.96, 136.40, 133.54, 132.13, 128.75, 127.93, 127.50, 121.98, 118.06, 110.27, 54.20, 87% yield

4-p-tolyl-3,4-dihydro-[1,3]oxazino[5,6-h]quinoline-2-one (4i)

White solid, IR (cm⁻¹): 3345(NH str), 1644(-C=O str), 1505(-C=C-str), 1280(-C-O-C str), 1095(-C=N str), ¹HNMR (δ/ppm): 8.769 (s, NH), 8.732 (d, Ar-H), 8.675 (m, Ar-H), 8.17 (m, Ar-H), 8.125 (m, Ar-H), 8.05 (m, Ar-H), 8.01 (m, Ar-H), 7.48-7.1 (m,

Ar-H), 7.0 (m, Ar-H), 6.9 (m, Ar-H), 4.5 (s, 1H), 2.2 (s, methyl proton); Mass [M]=290, [M+1]=291, ¹³CNMR (ppm), 166.68, 152.44, 148.06, 147.96, 136.40, 133.54, 132.13, 128.75, 127.93, 127.50, 121.98, 118.06, 110.27, 54.20, 27.20, 84% yield.

CONCLUSION

From this study, we report a green synthesis of oxazino quinoline derivatives using a heterogeneous nano copper ferrite catalyst.

3. References

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Electronic Supplementary Data of Oxazinoquinolines

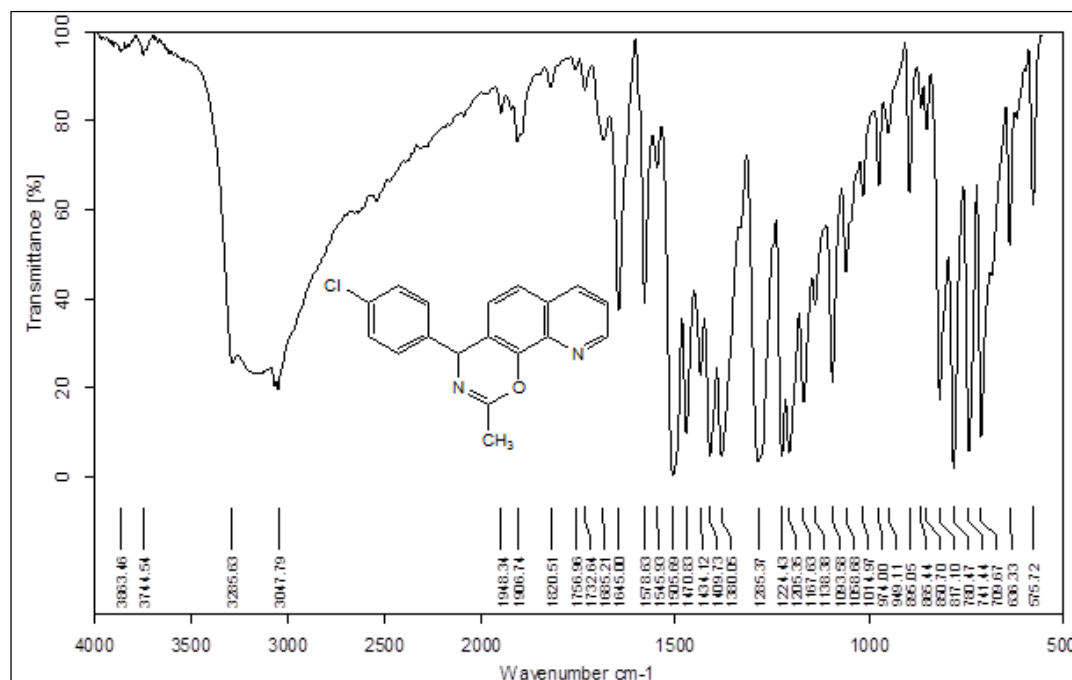


Figure 1.0 FT-IR spectrum of 4a

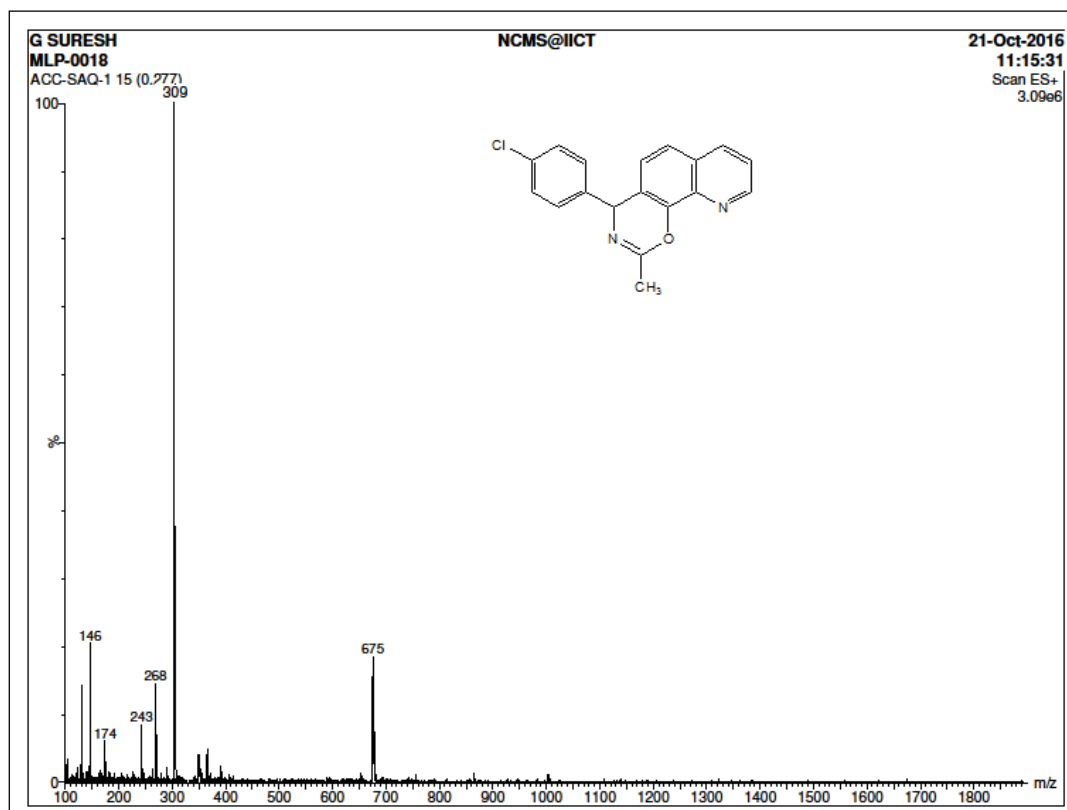


Figure 1.2 MASS spectrum of 4a

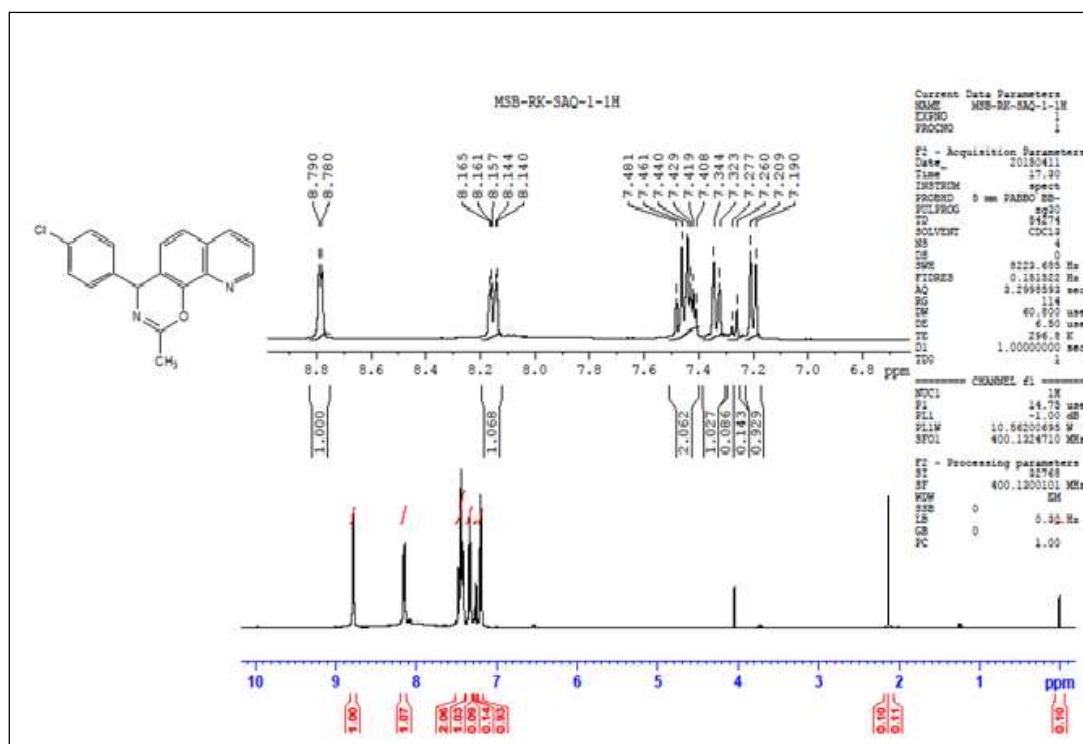


Figure 1.3 ¹H NMR spectrum of 4a

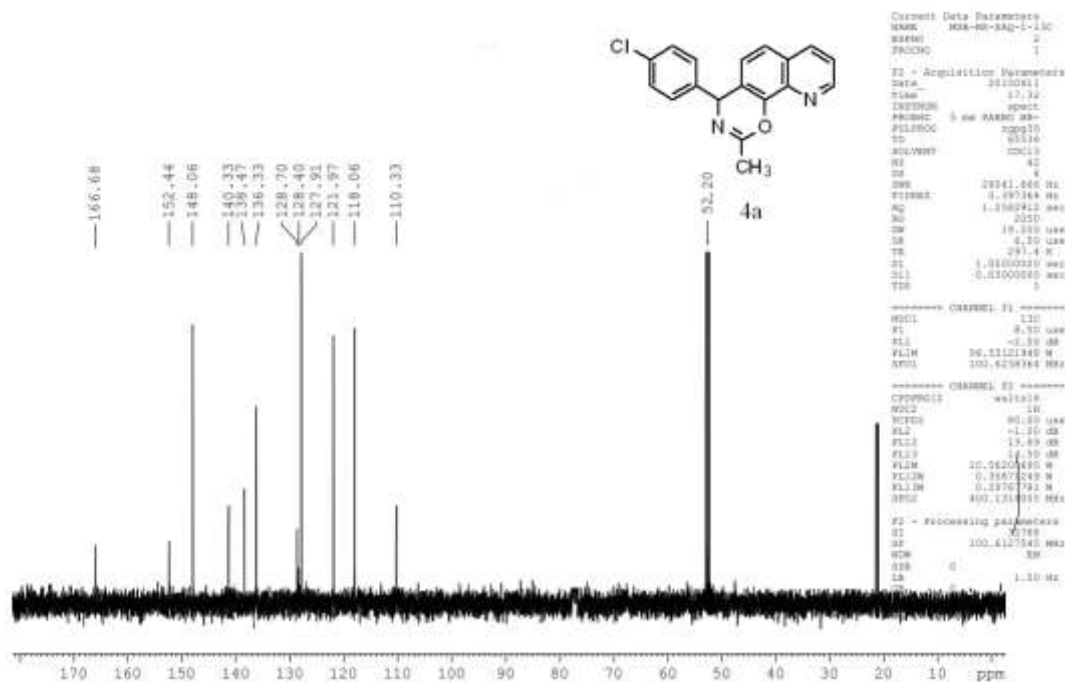


Figure: 1.4 ¹³C NMR of 4a

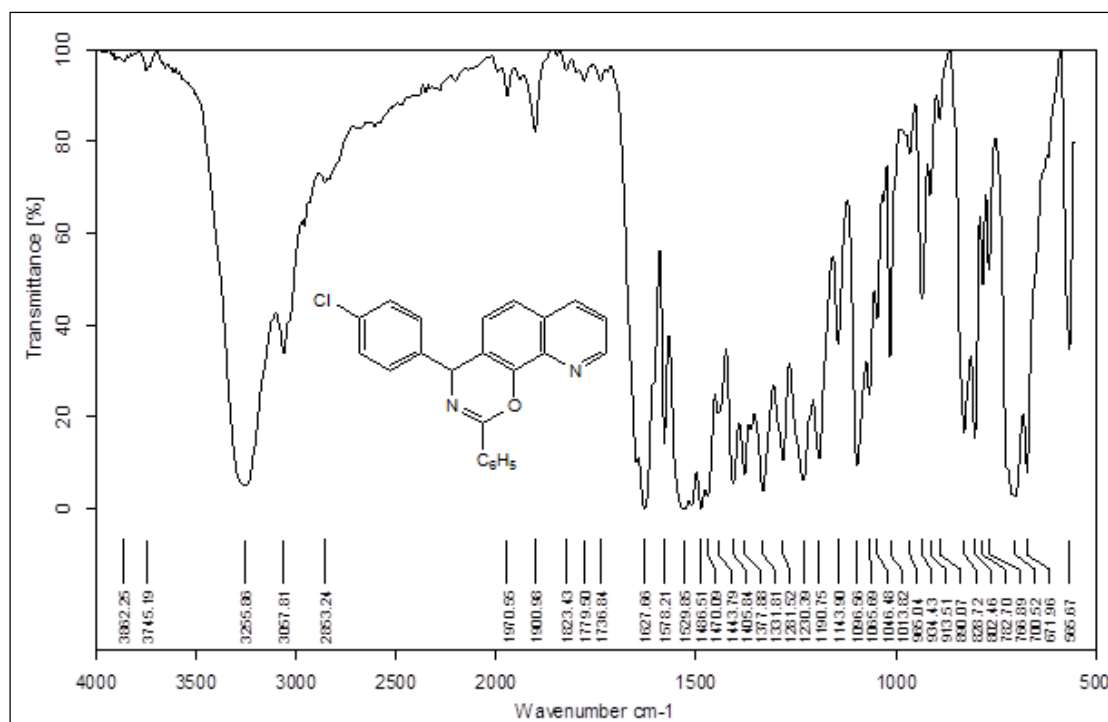


Figure 1.5: FT-IR spectrum of 4b

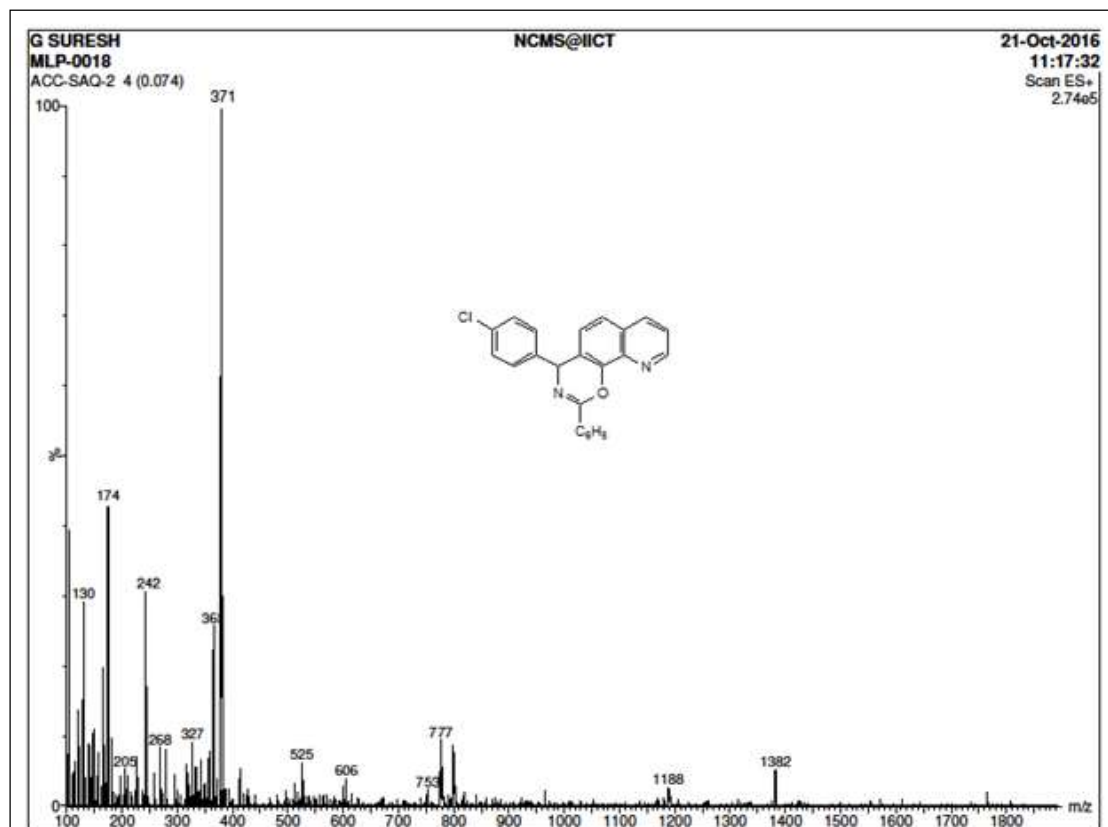


Figure 1.6: MASS spectrum of 4b

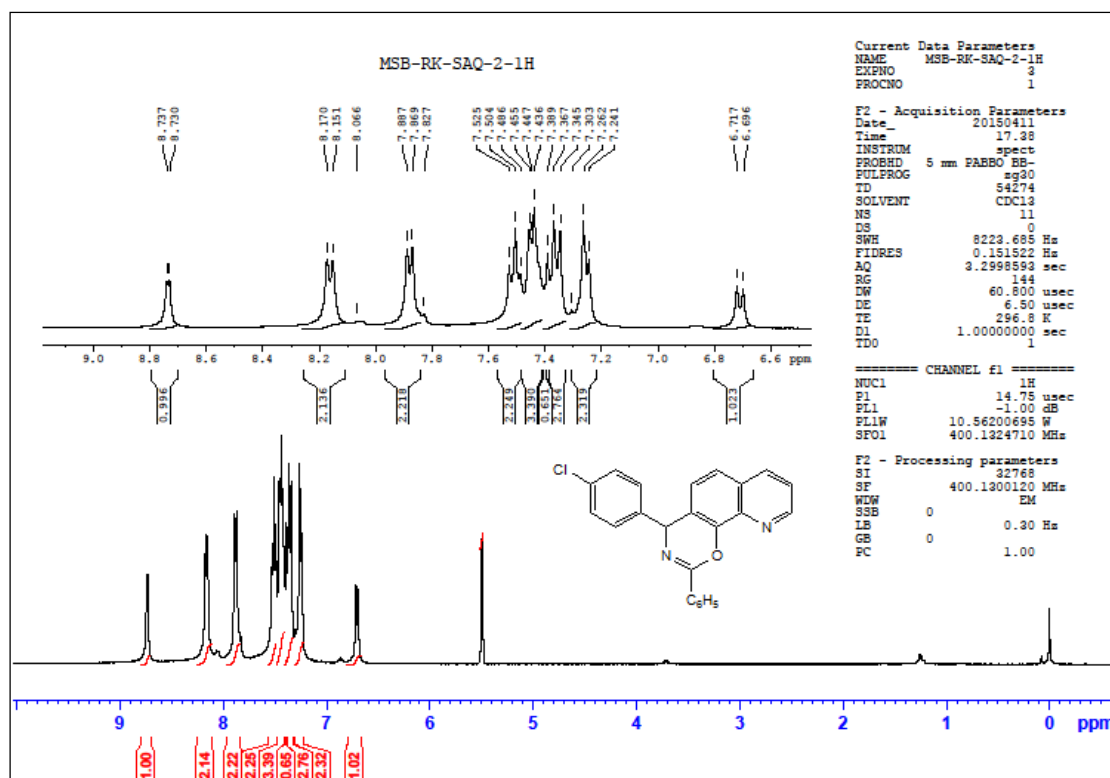


Figure 1.7: ¹H NMR spectrum of 4b

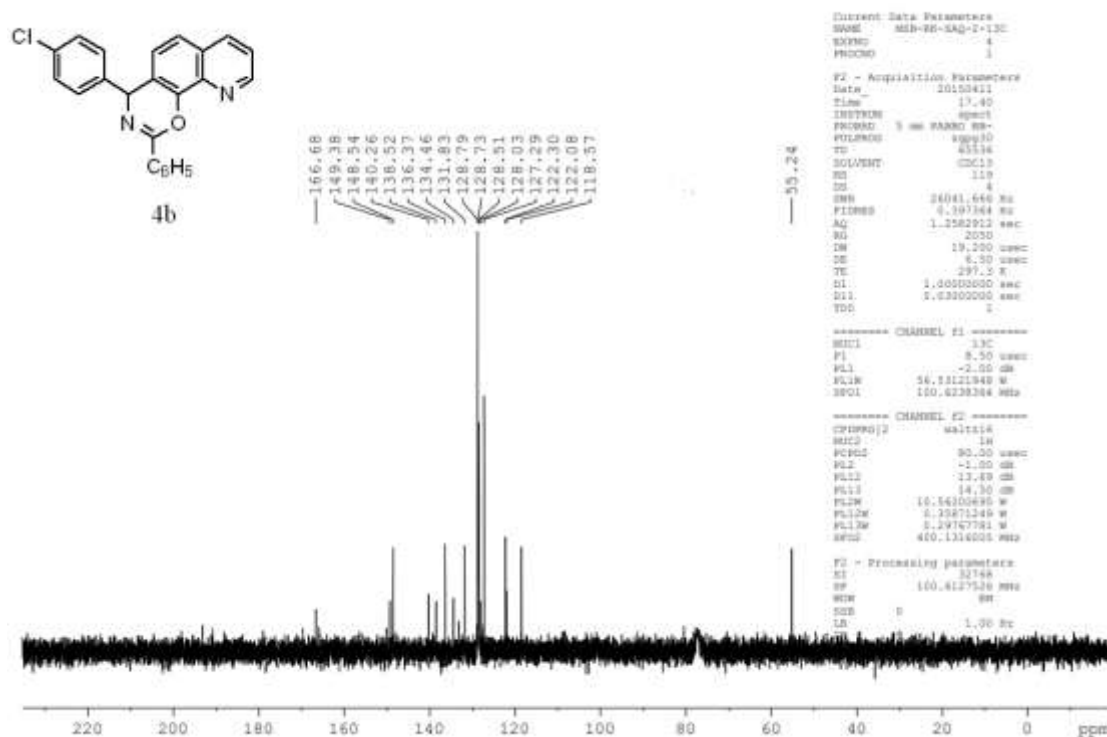


Figure 1.8: ¹³C NMR of 4b

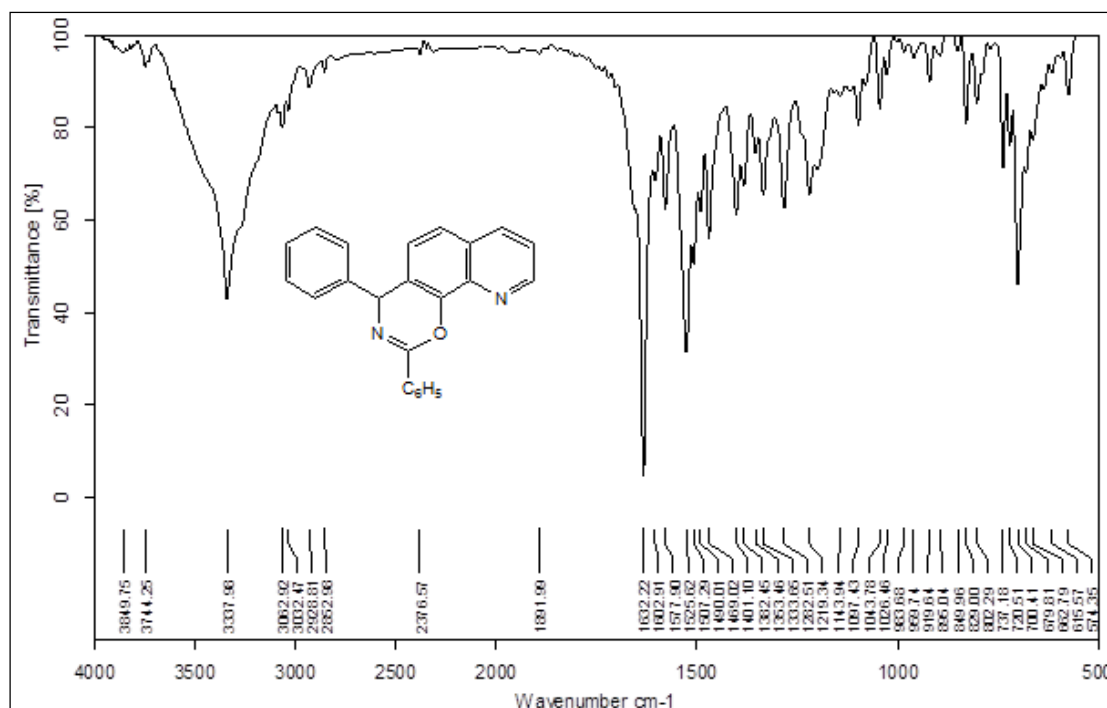


Figure 1.9: FT-IR spectrum of 4c

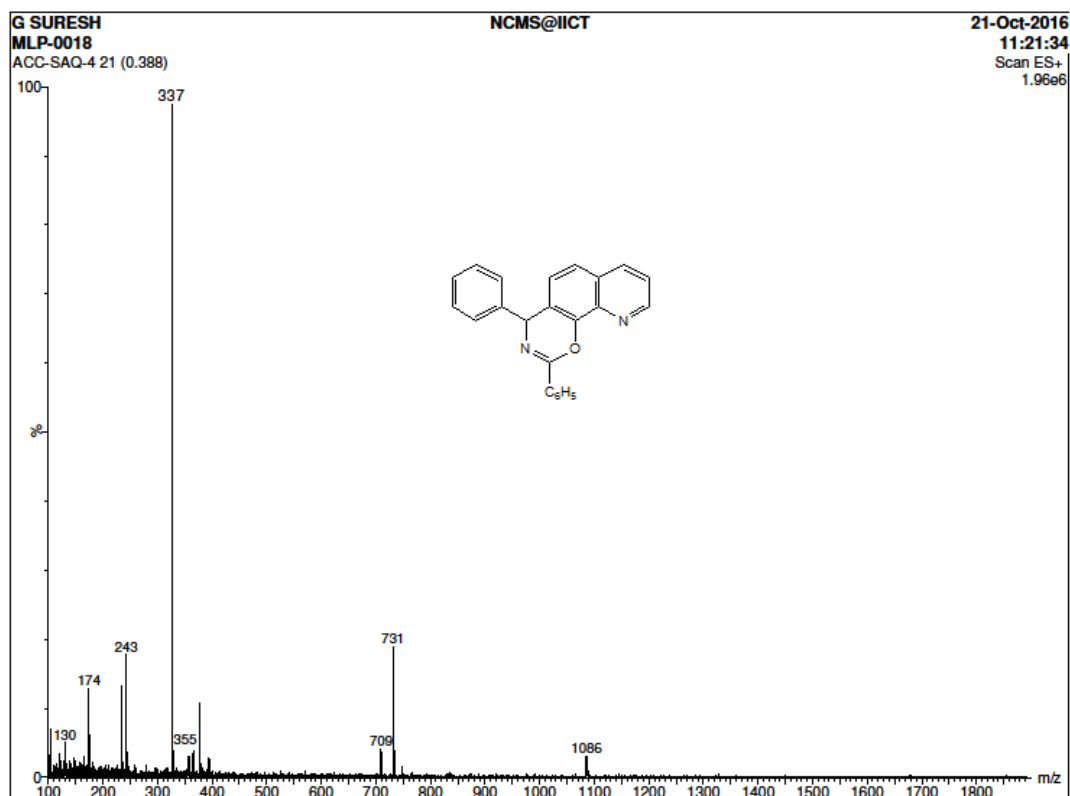


Figure 1.10.: MASS spectrum of 4c

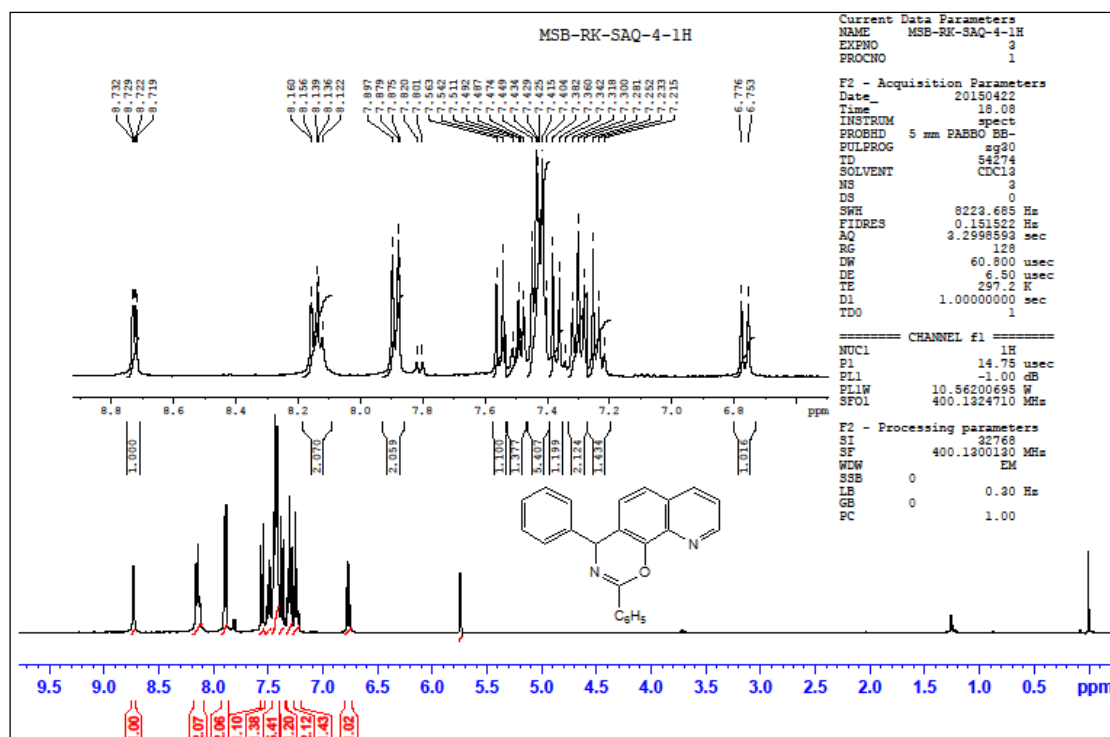


Figure 1.11: ¹H NMR spectrum of 4c

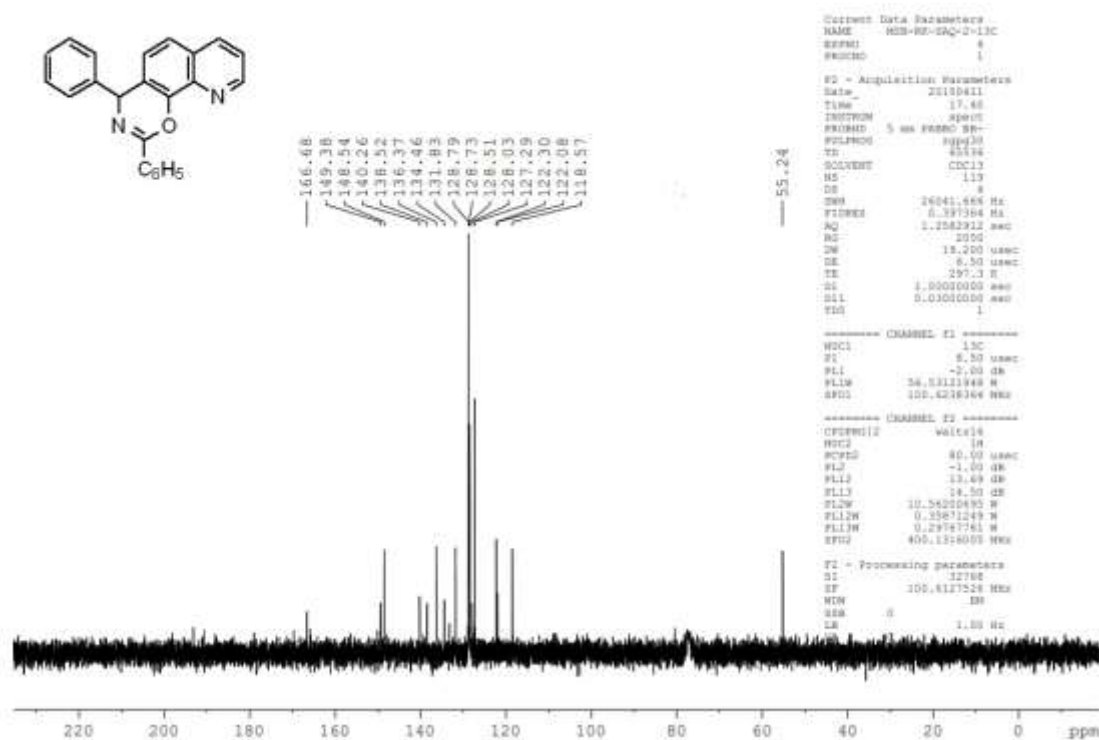


Figure 1.12: ¹³C NMR of 4c

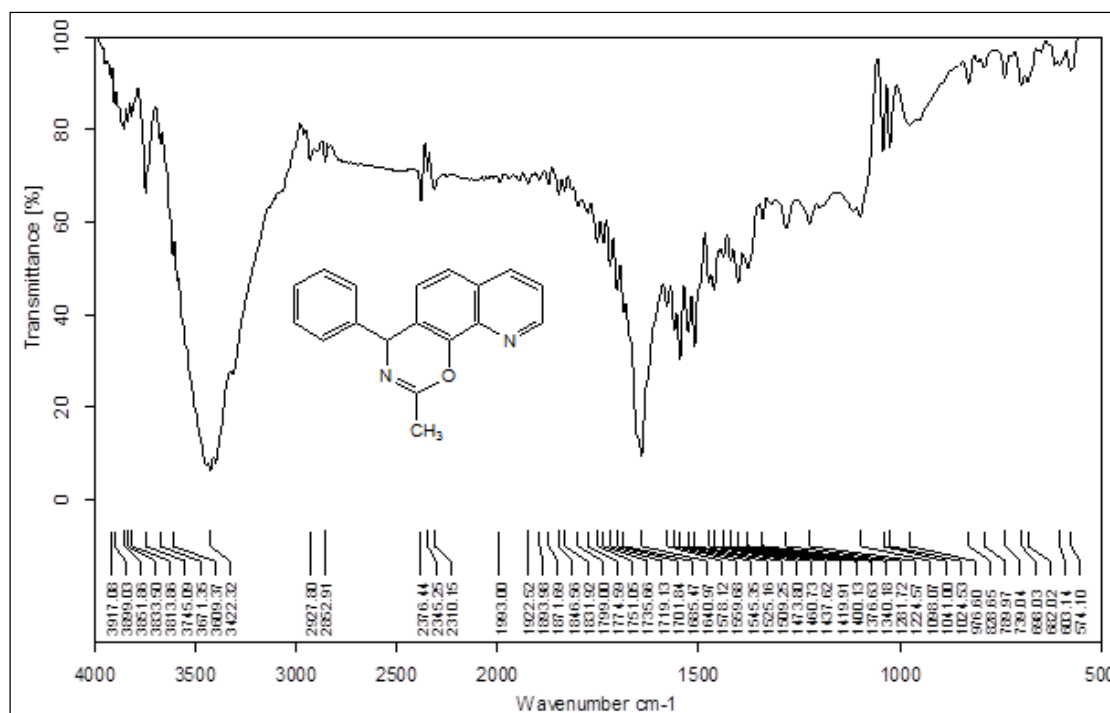


Figure 1.13: FT-IR spectrum of 4d

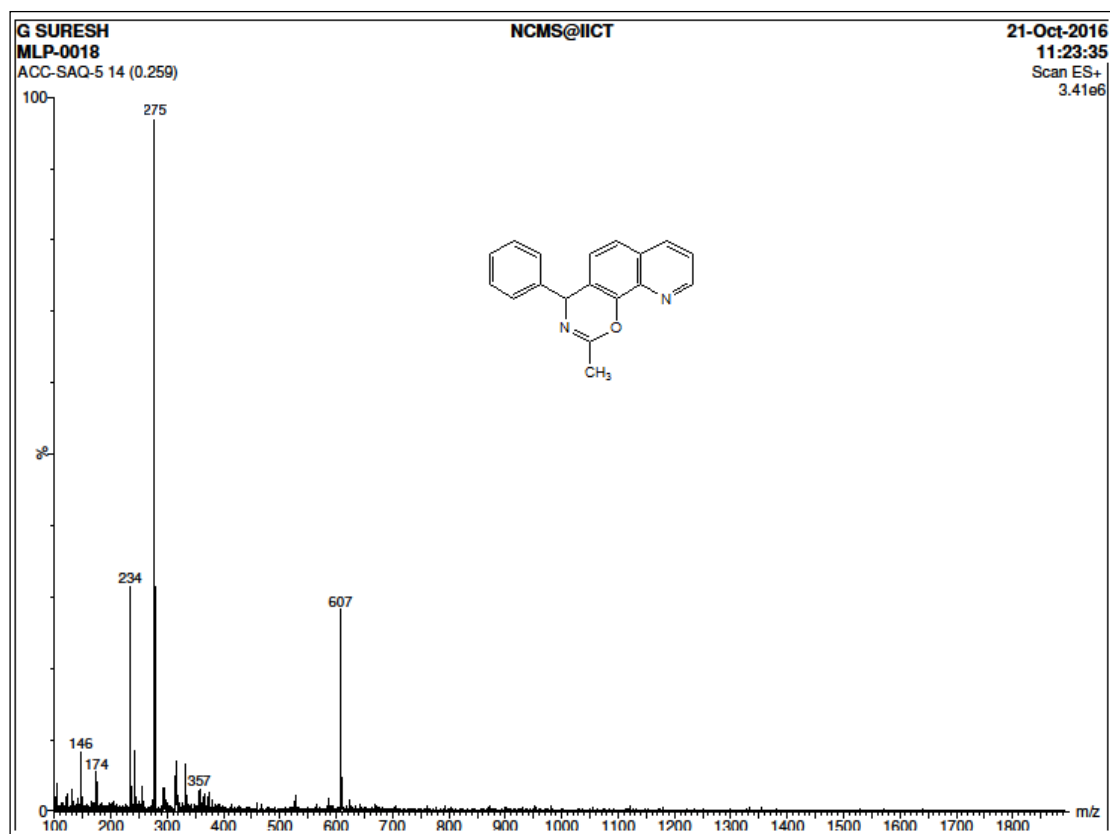


Figure 1.14: MASS spectrum of 4d

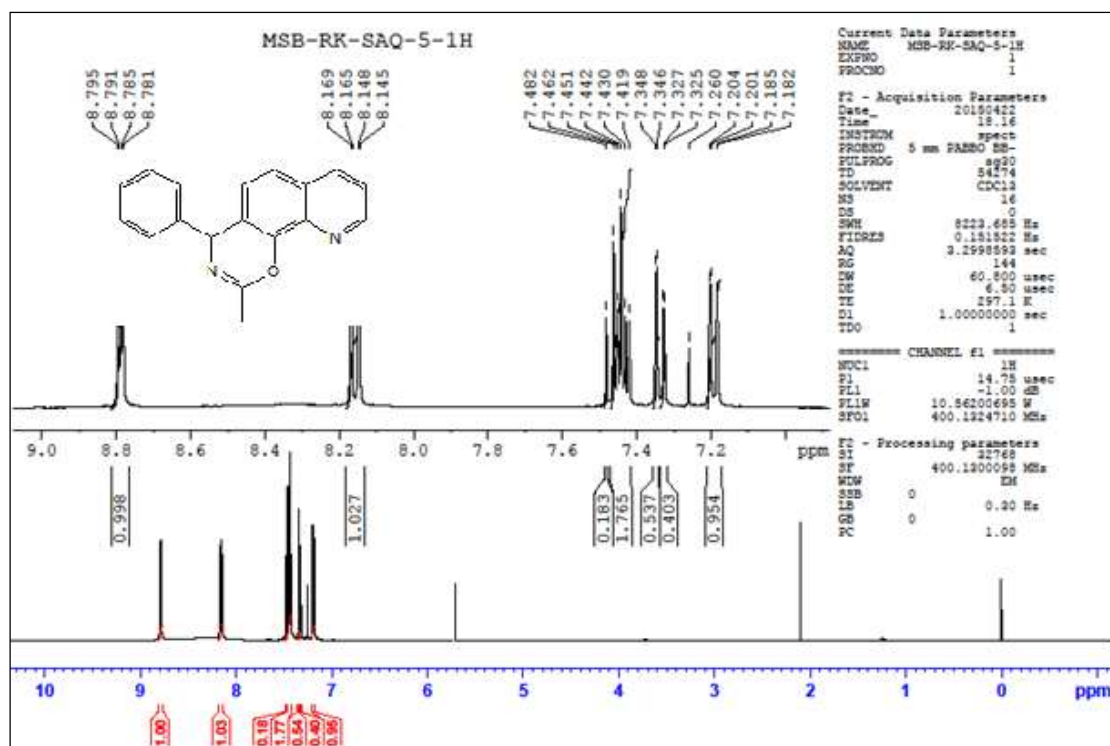


Figure 1.15: ¹H NMR spectrum of 4d

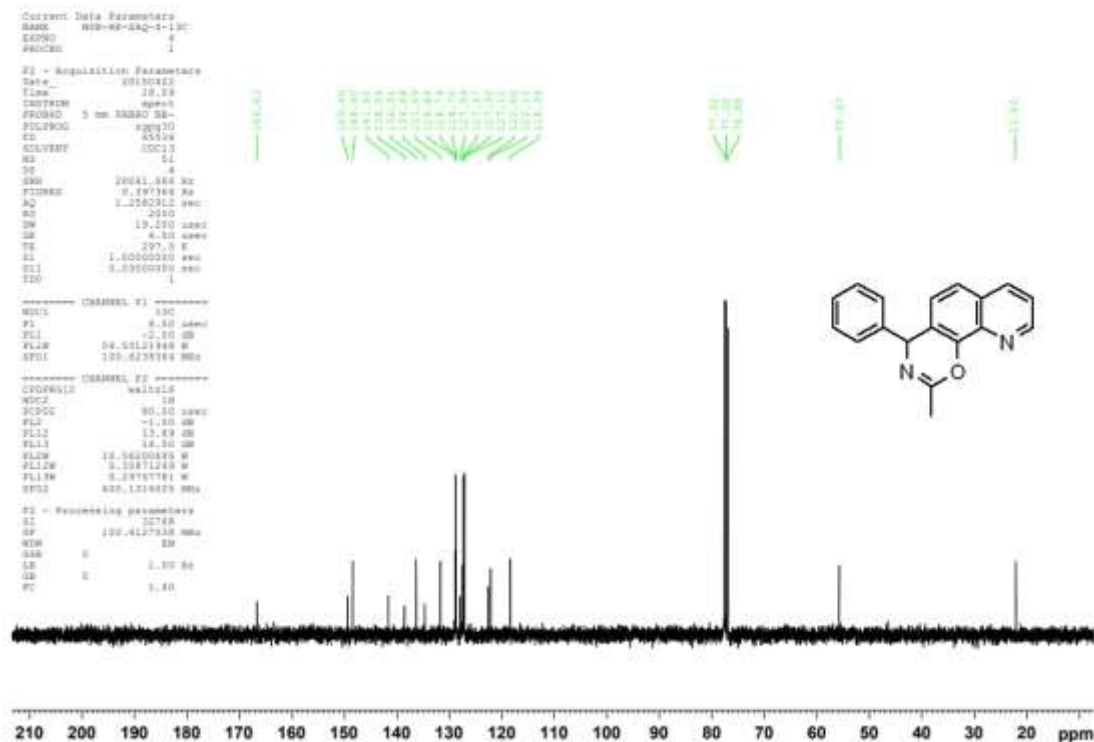


Figure 1.16: ¹³C NMR of 4d

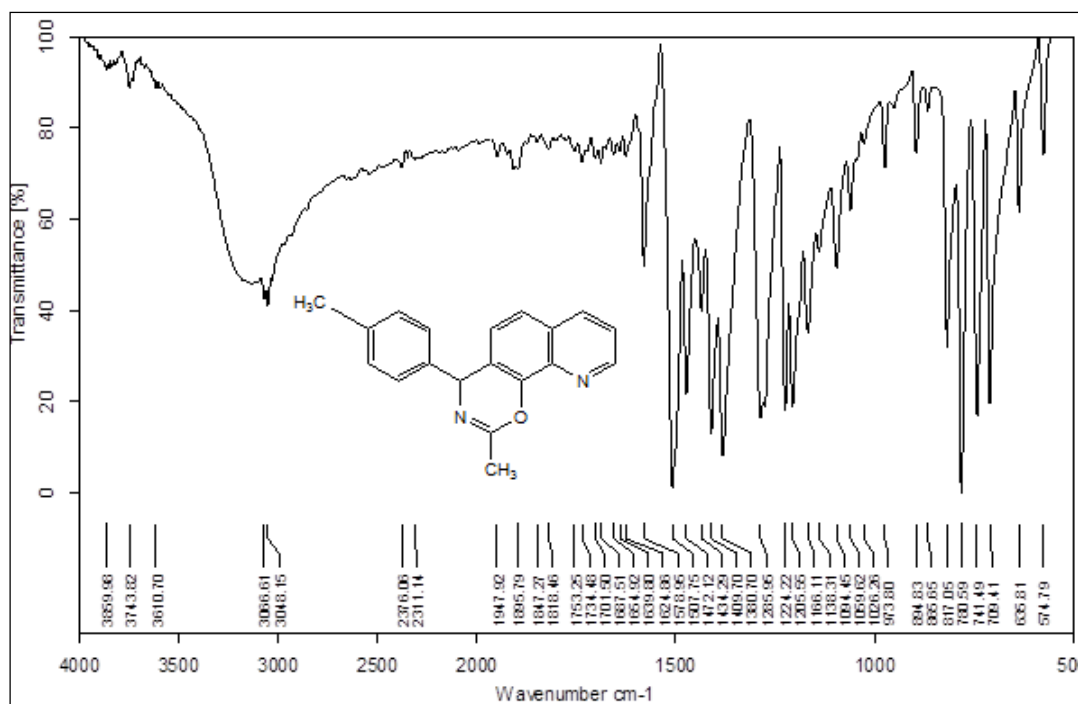


Figure 1.17: FT-IR spectrum of 4e

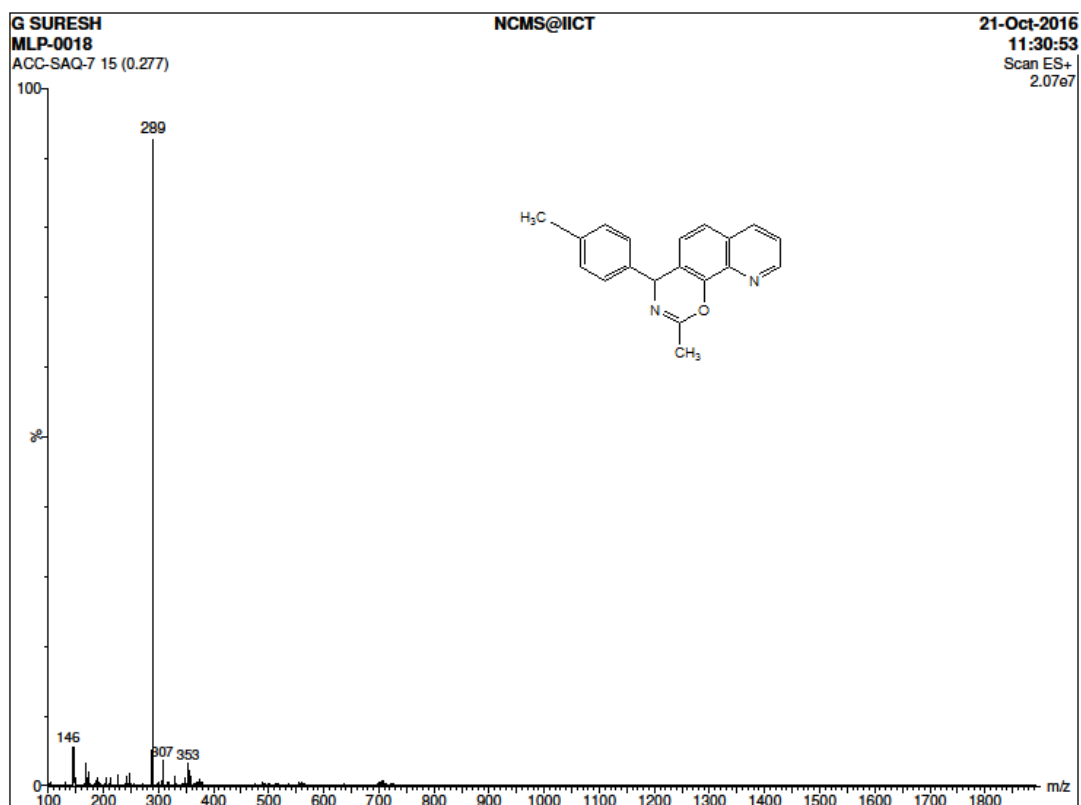


Figure 1.18: MASS spectrum of 4e

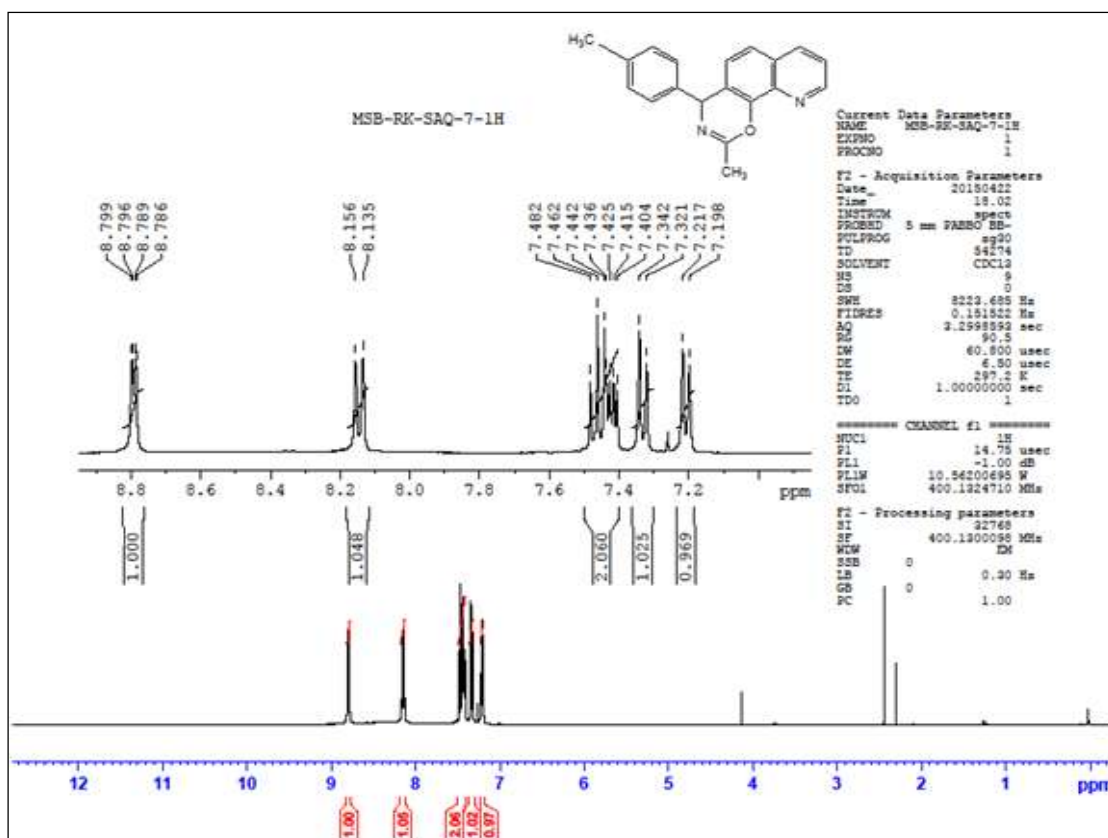


Figure1. 19: ¹H NMR spectrum of 4e

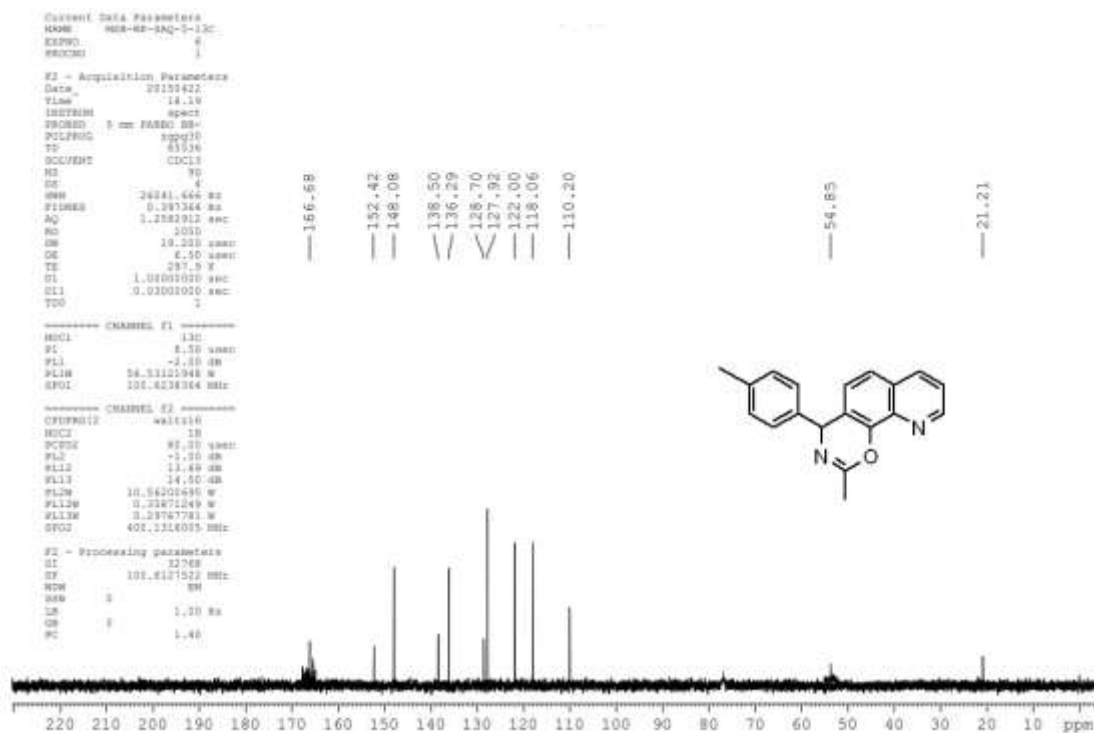


Figure 1.20: ¹³C NMR of 4e

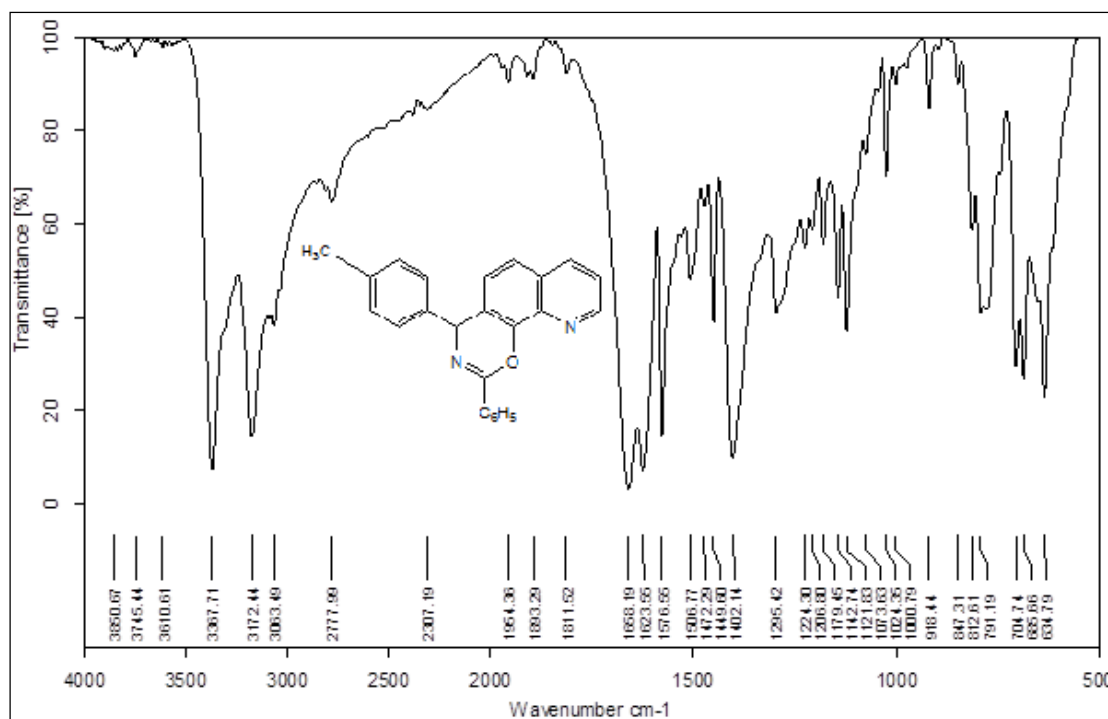


Figure 1.21: FT-IR spectrum of 4f

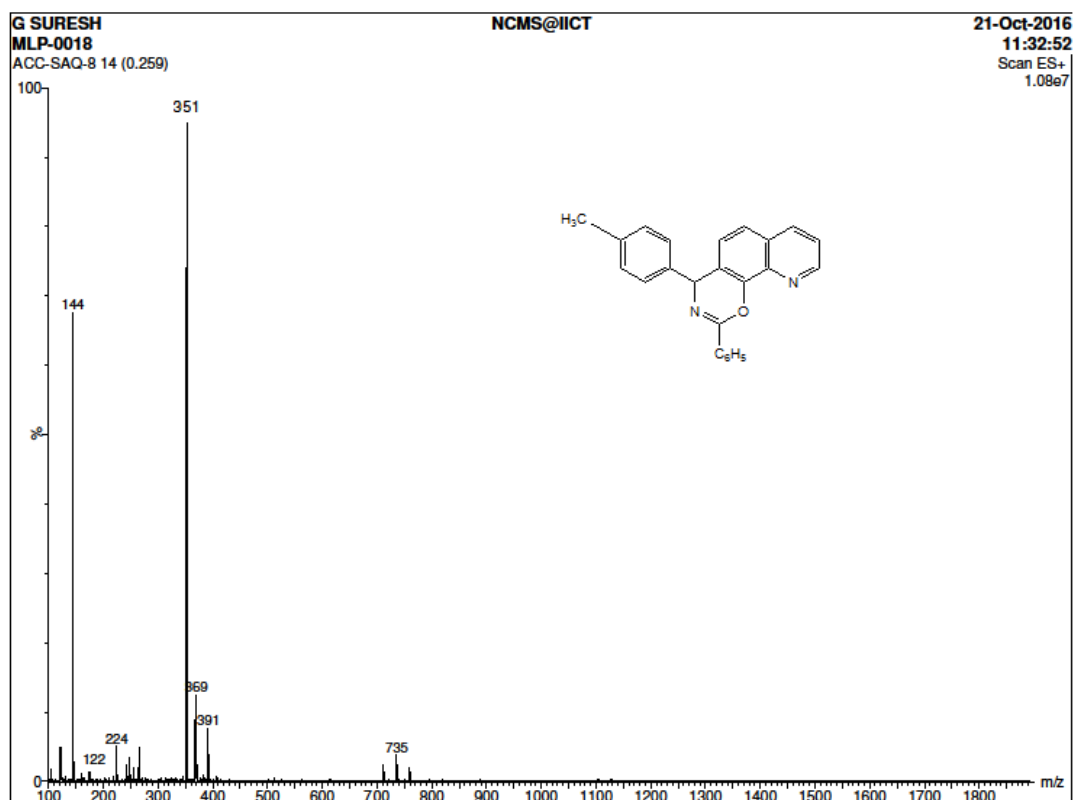
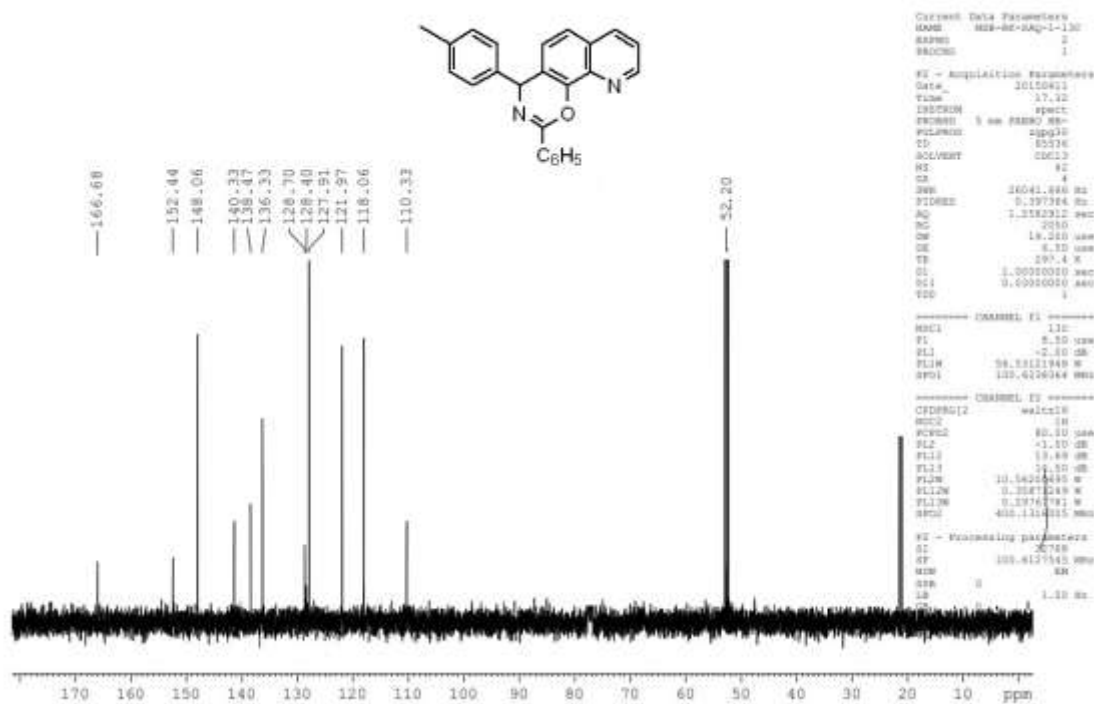
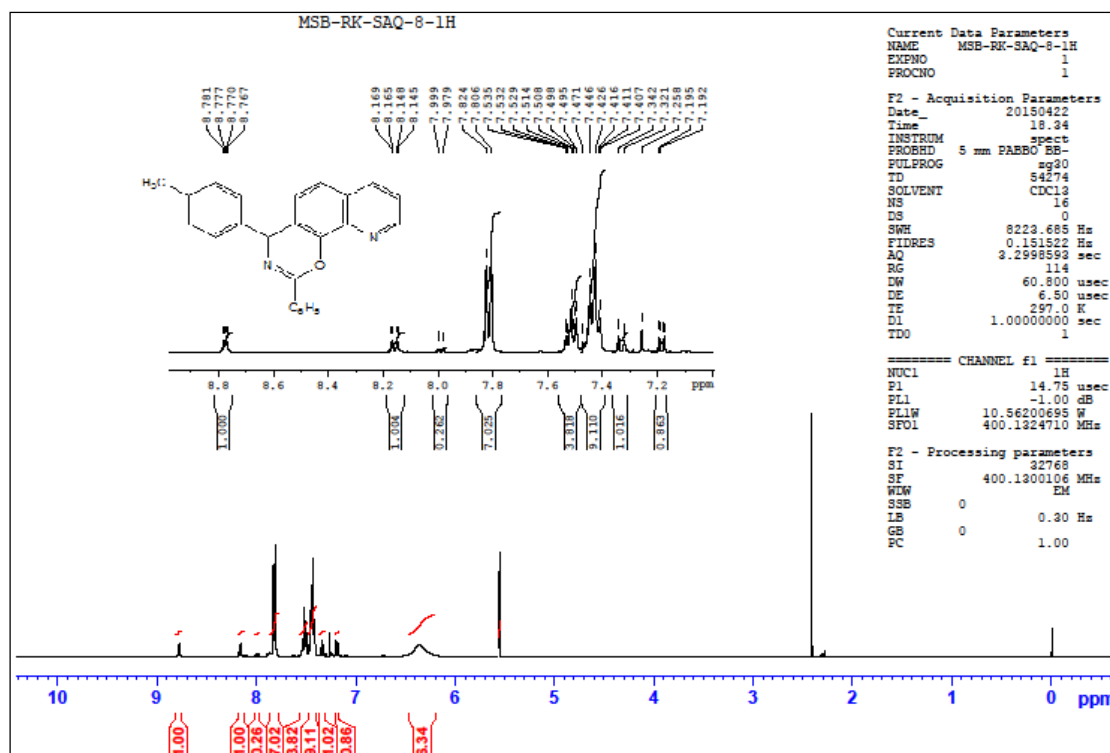
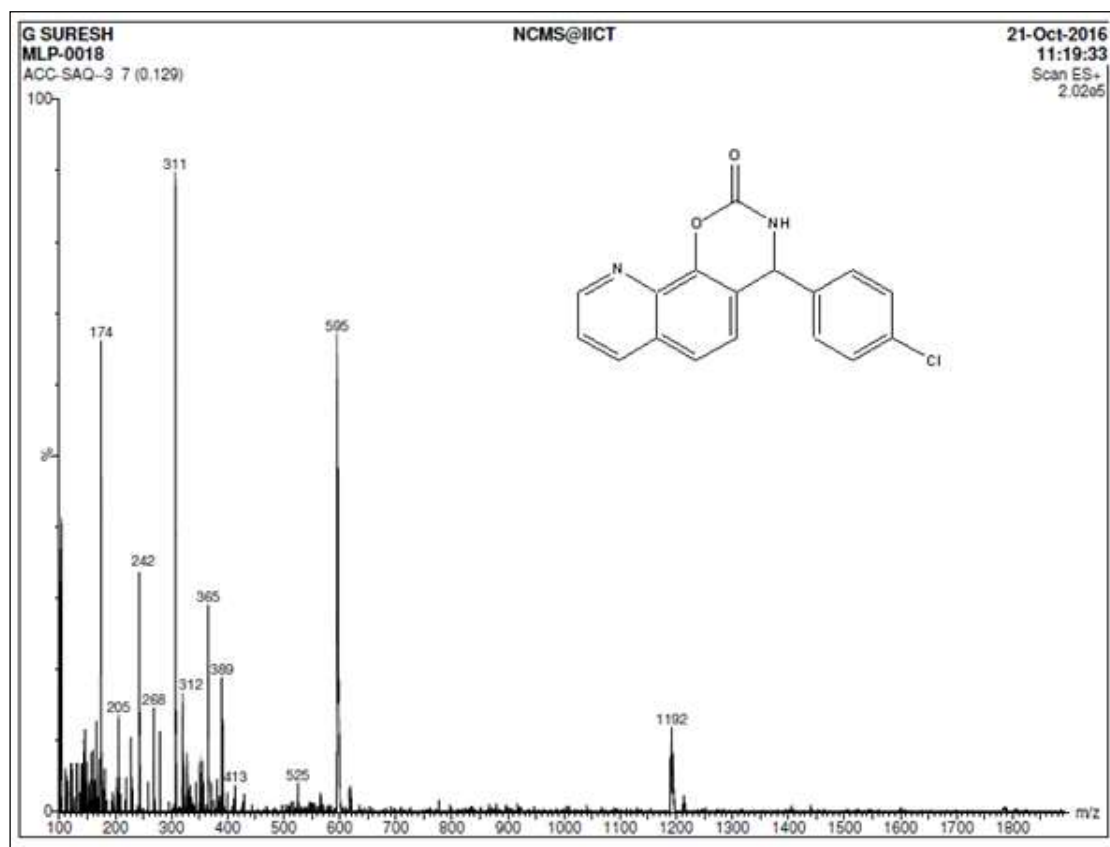
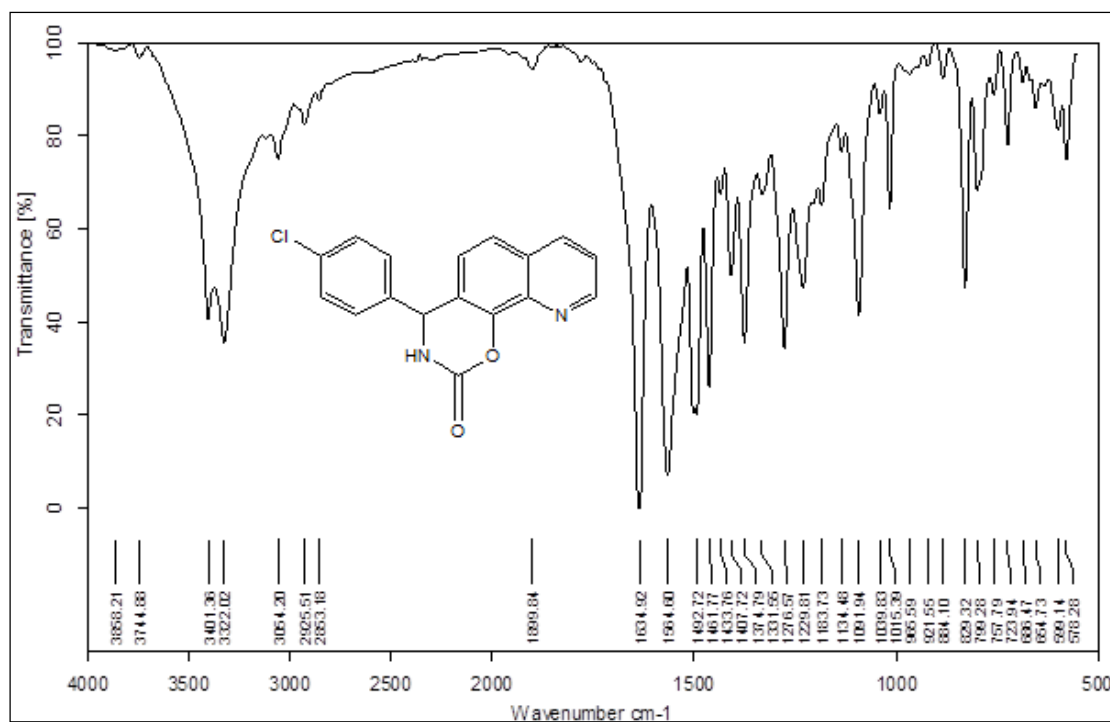


Figure 1.22: MASS spectrum of 4f





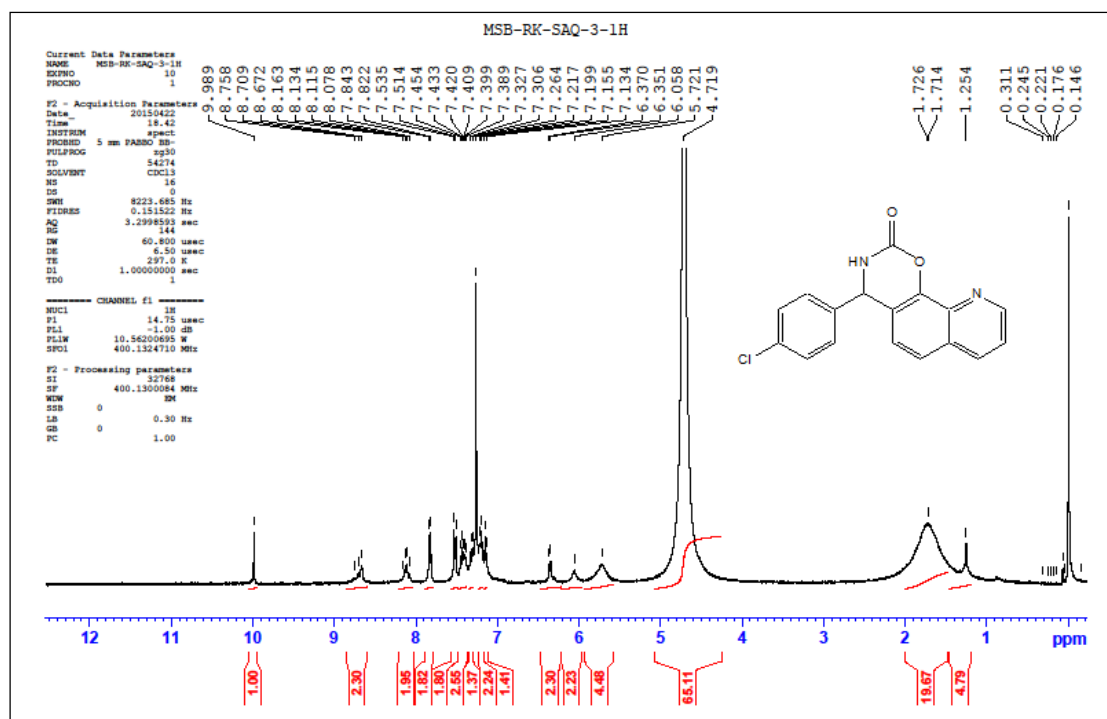


Figure 1.27: ¹H NMR spectrum of 4g

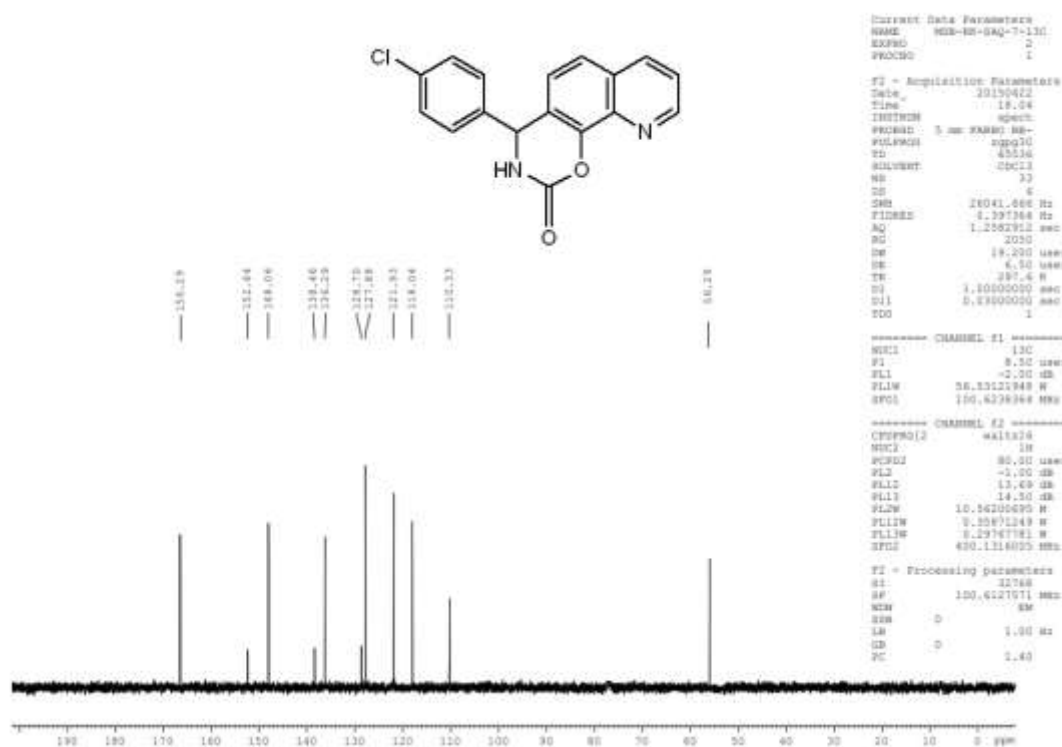


Figure 1.28: ¹³C NMR of 4g

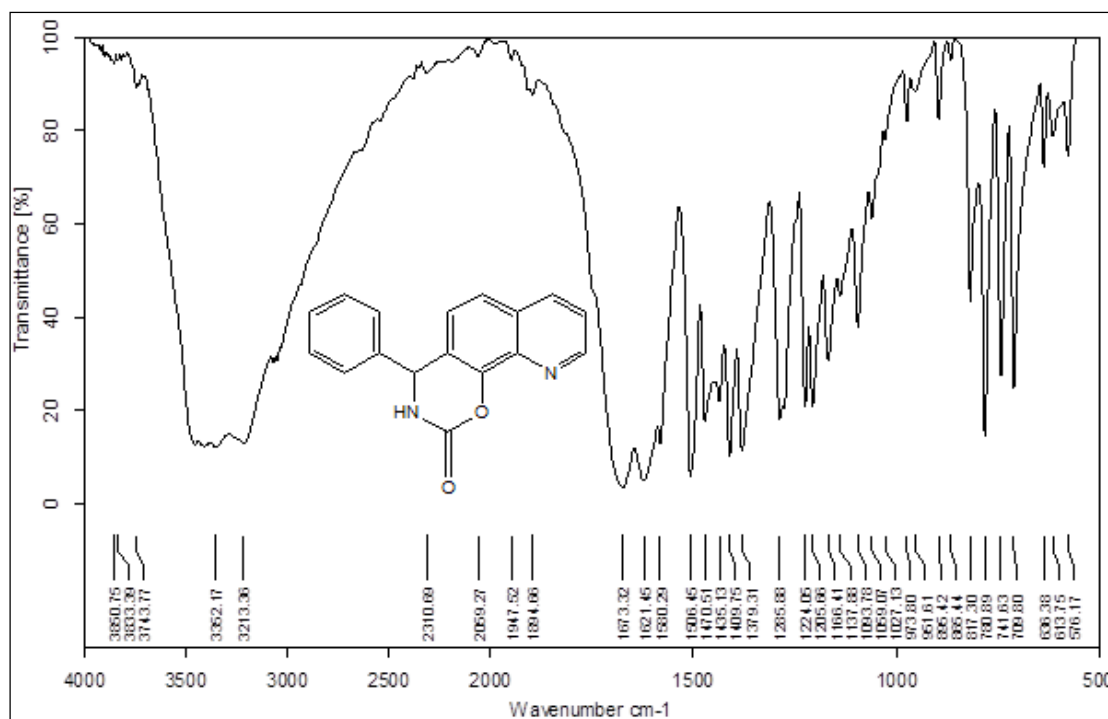


Figure 1.29: FT-IR spectrum of 4h

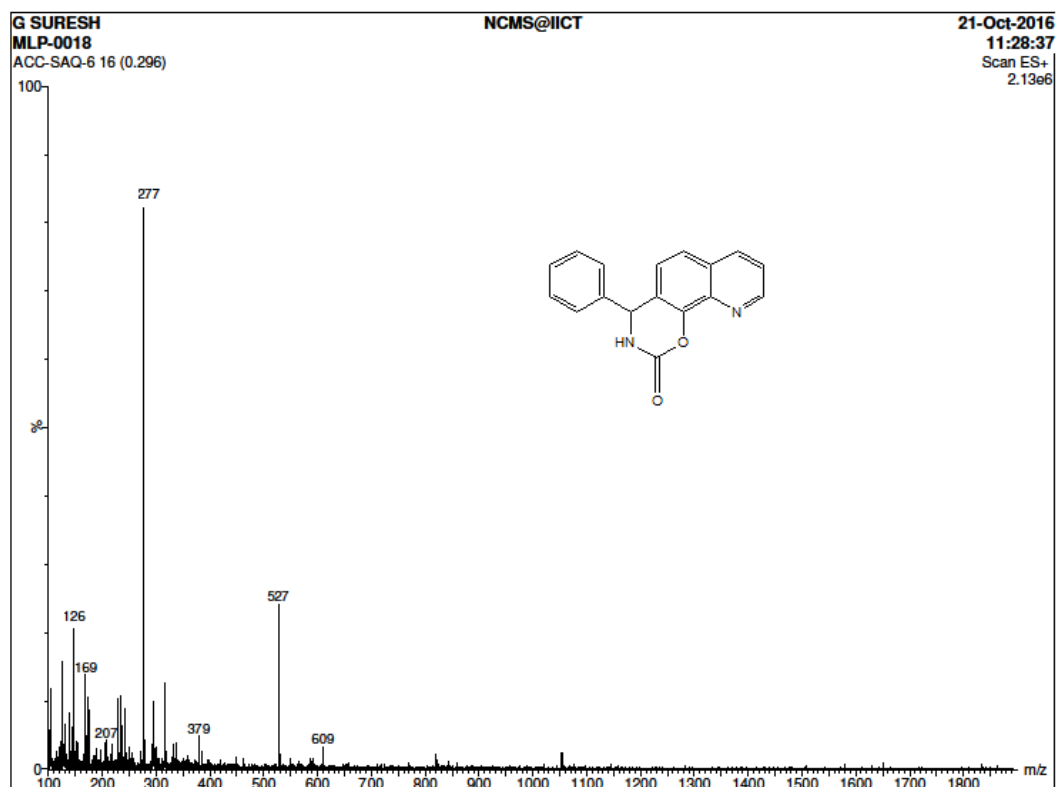


Figure 1.30: MASS spectrum of 4h

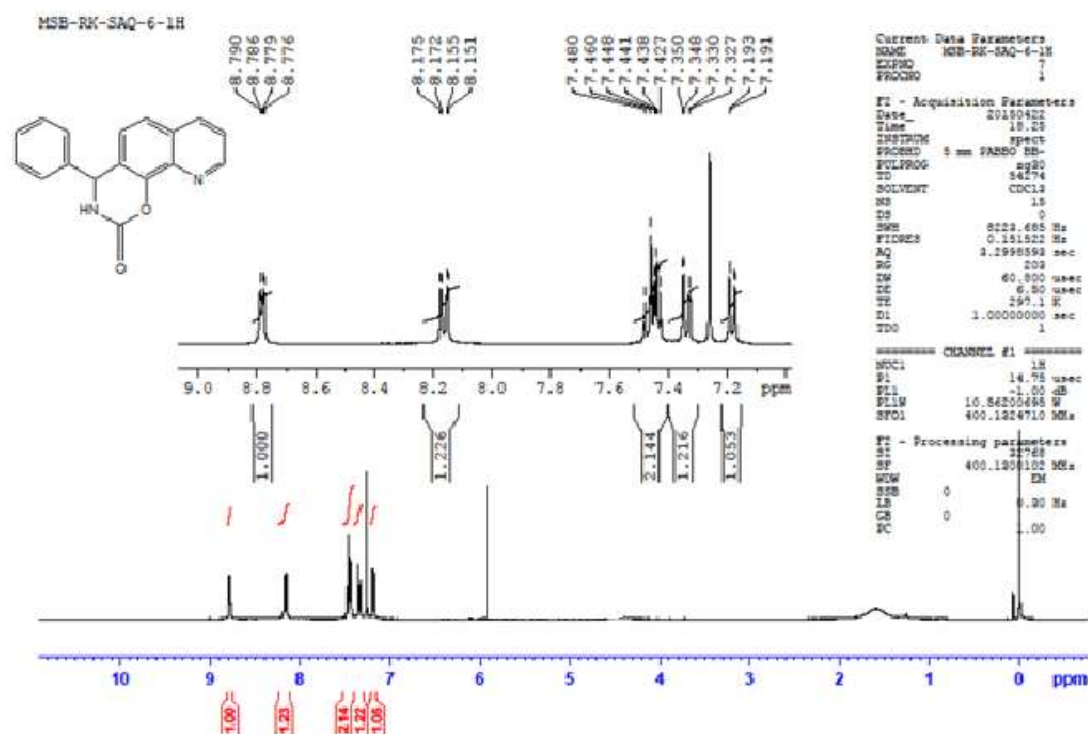


Figure 1.31: ¹H NMR spectrum of 4h

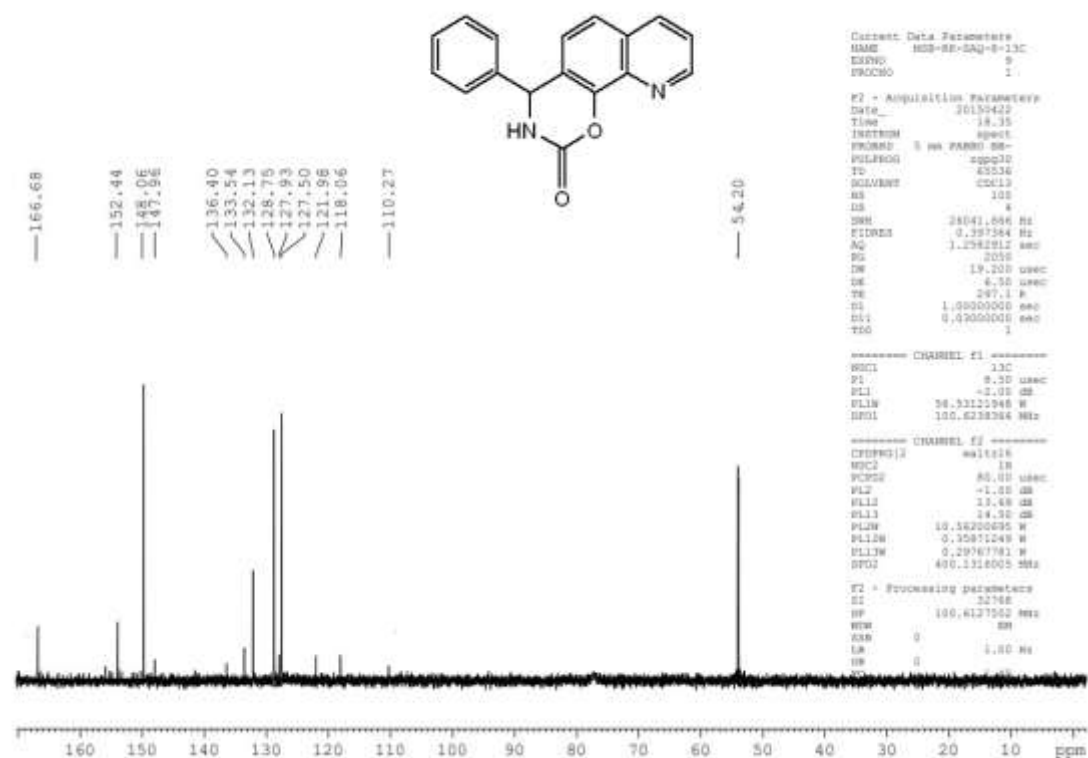


Figure 1.32: ¹³C NMR of 4h

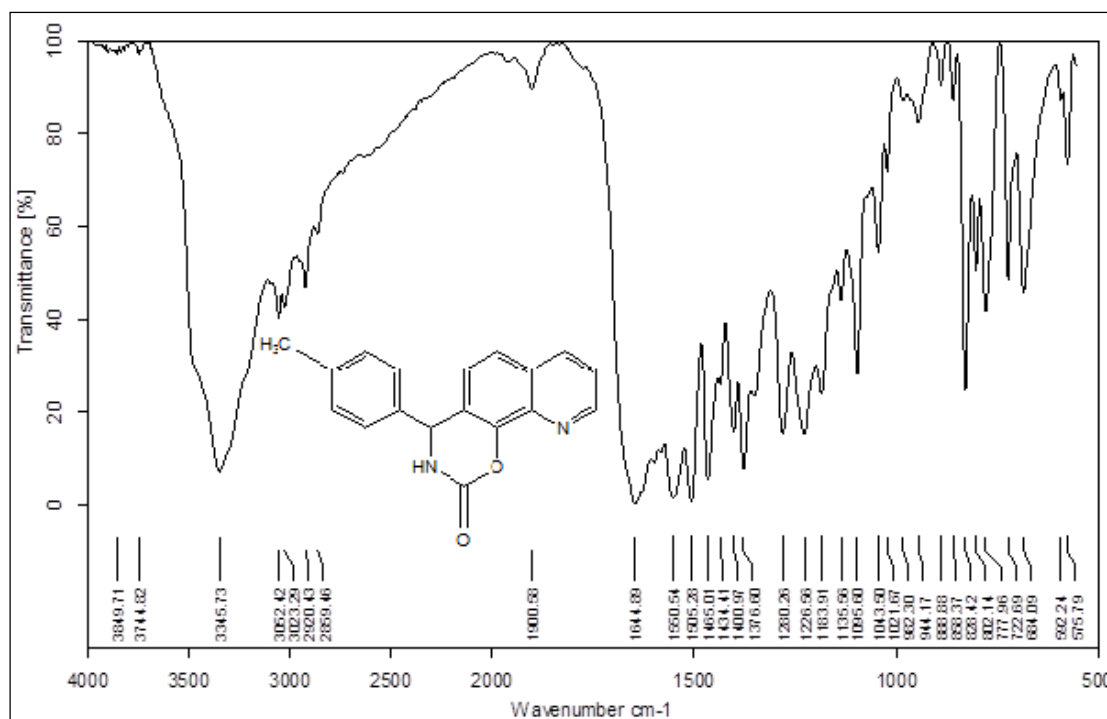


Figure 3. 29: FT-IR spectrum of 4i

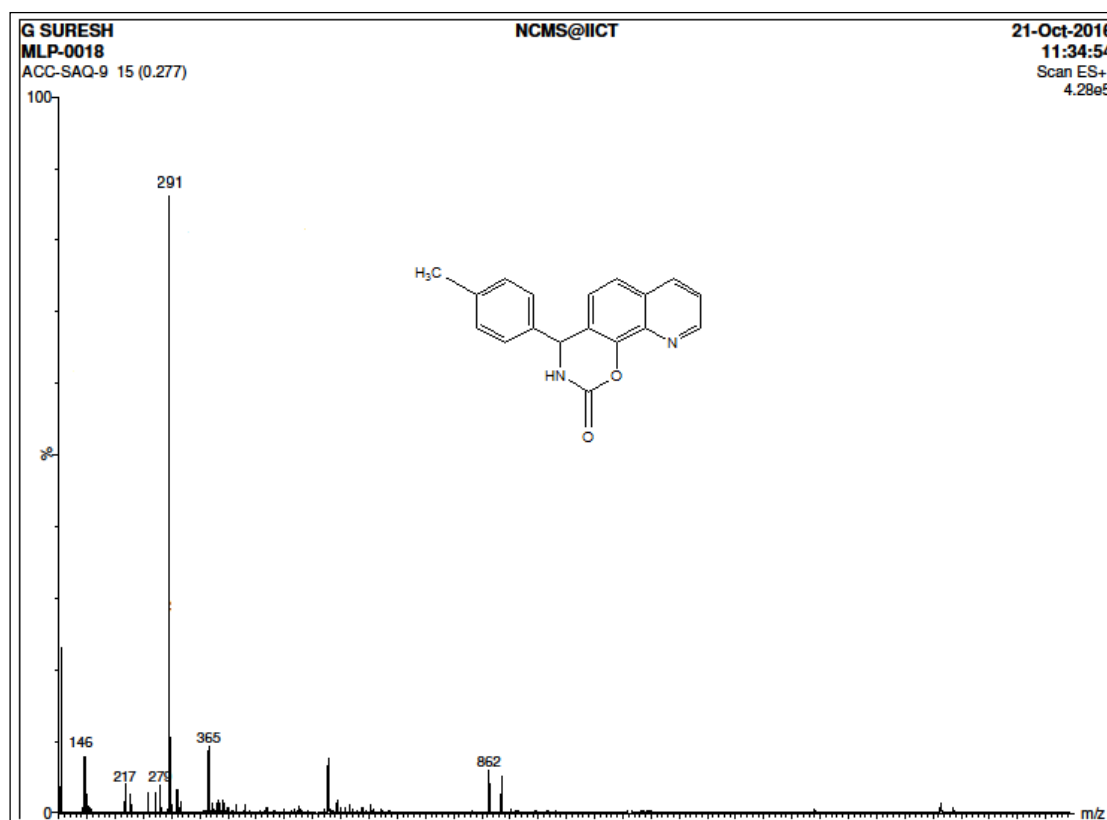


Figure 3. 30. MASS spectrum of 4i

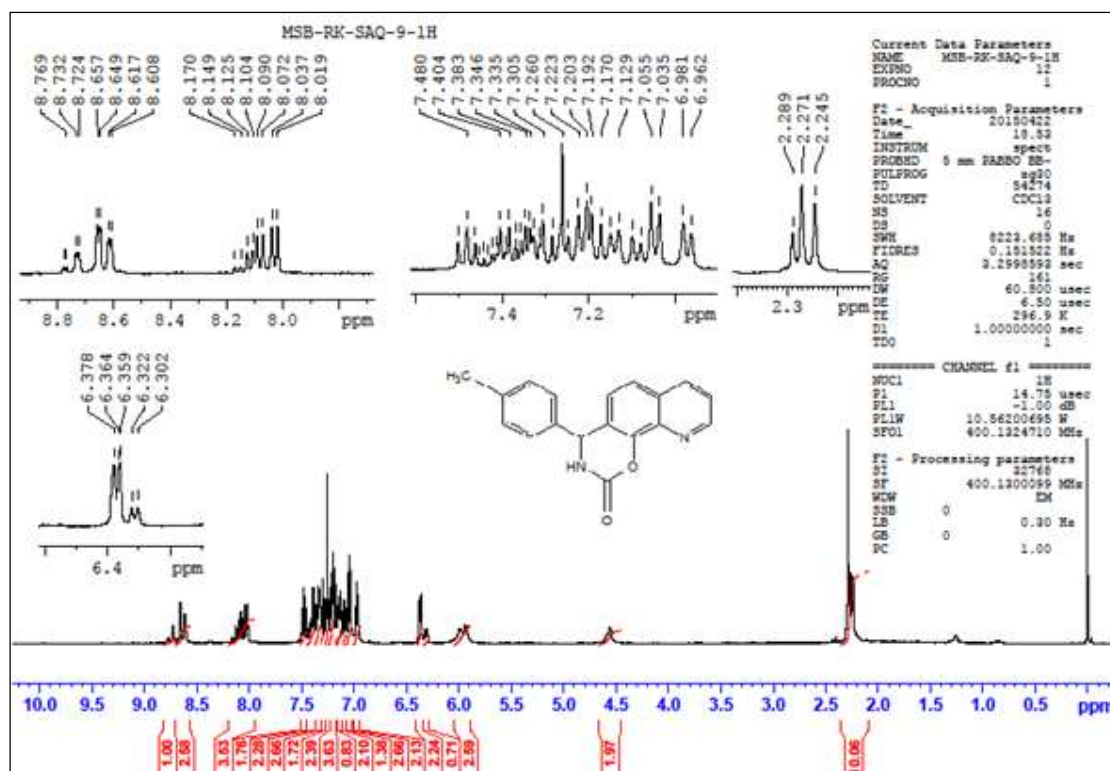
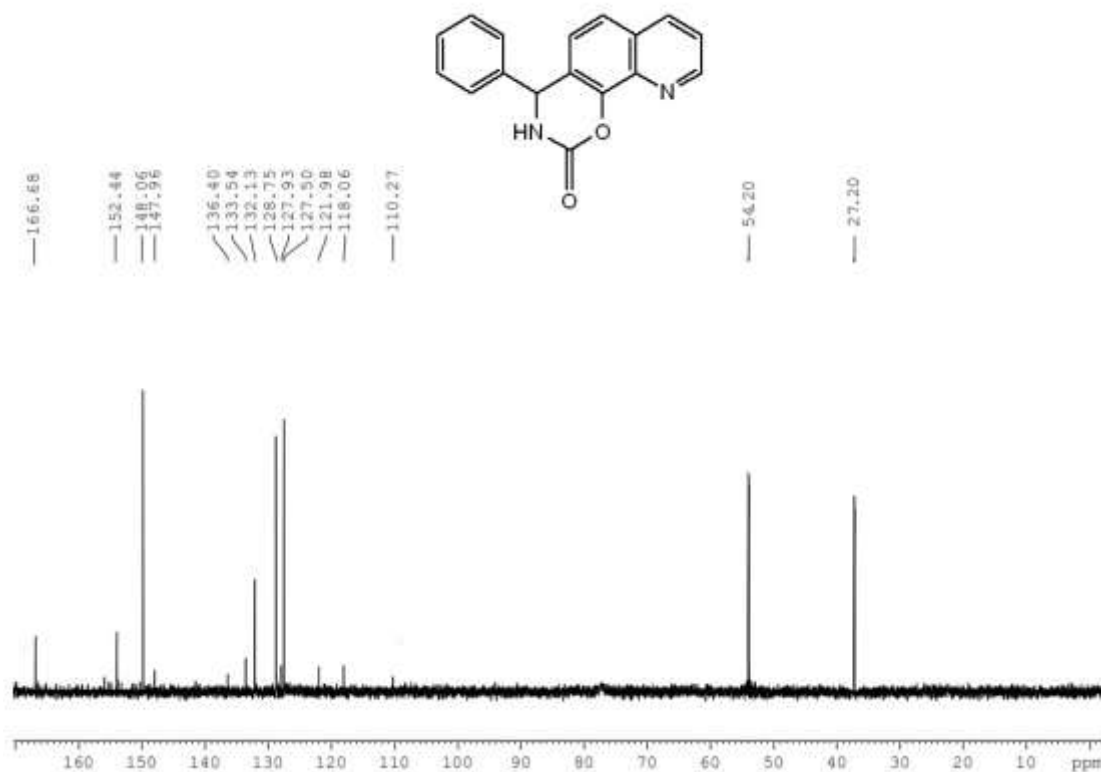


Figure 3. 31: ¹H NMR spectrum of 4i



¹³C NMR of 4i