

Chapter-7

References

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Chapter-8

Appendices

^1H , ^{13}C , FTIR, HRMS, and HPLC spectra of representative compounds

SERIES I

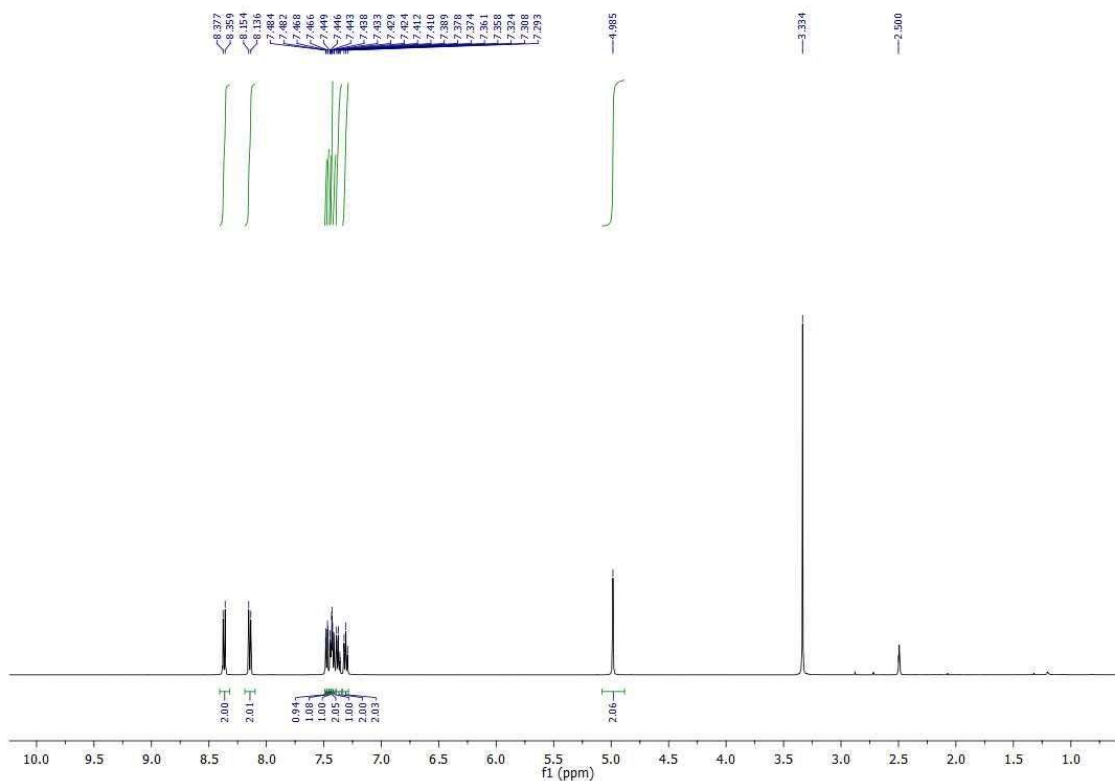


Figure 8.1. ^1H NMR spectra of compound 6a

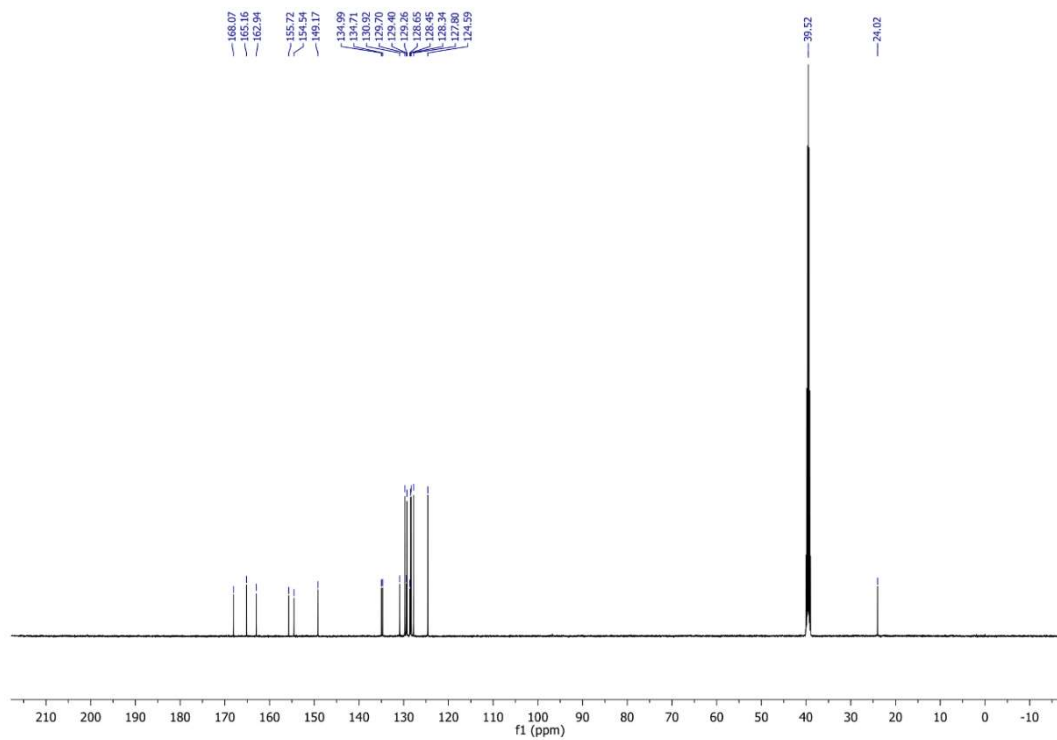


Figure 8.2. ^{13}C NMR spectra of compound 6a

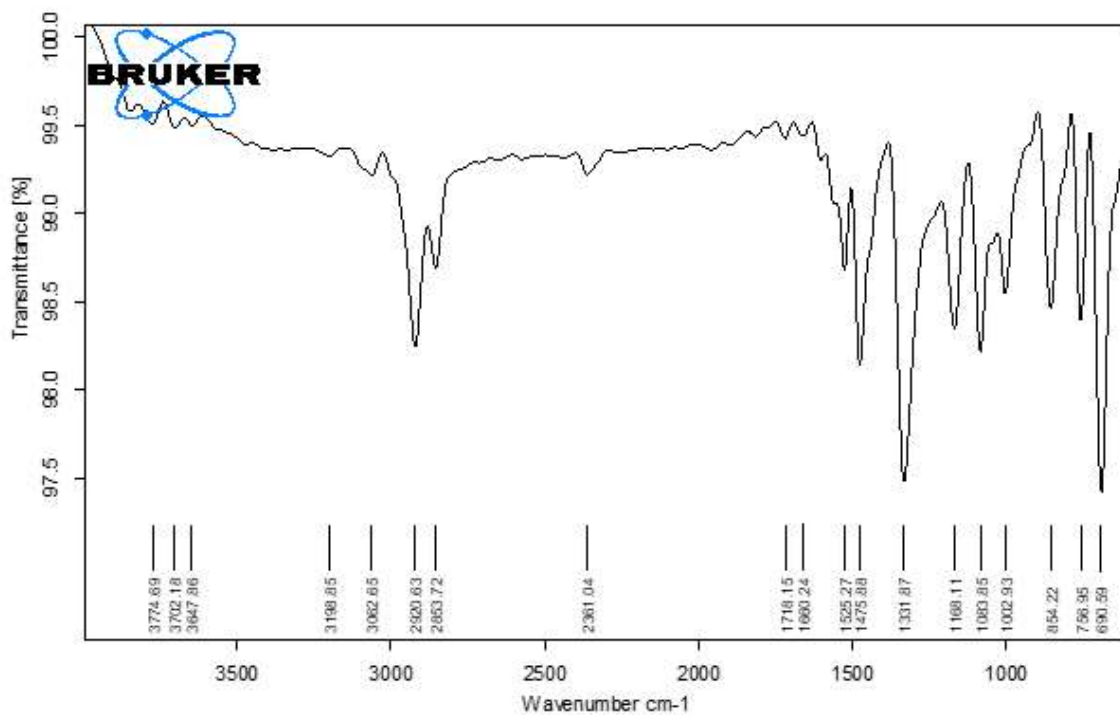


Figure 8. 3. FTIR spectra of compound 6a

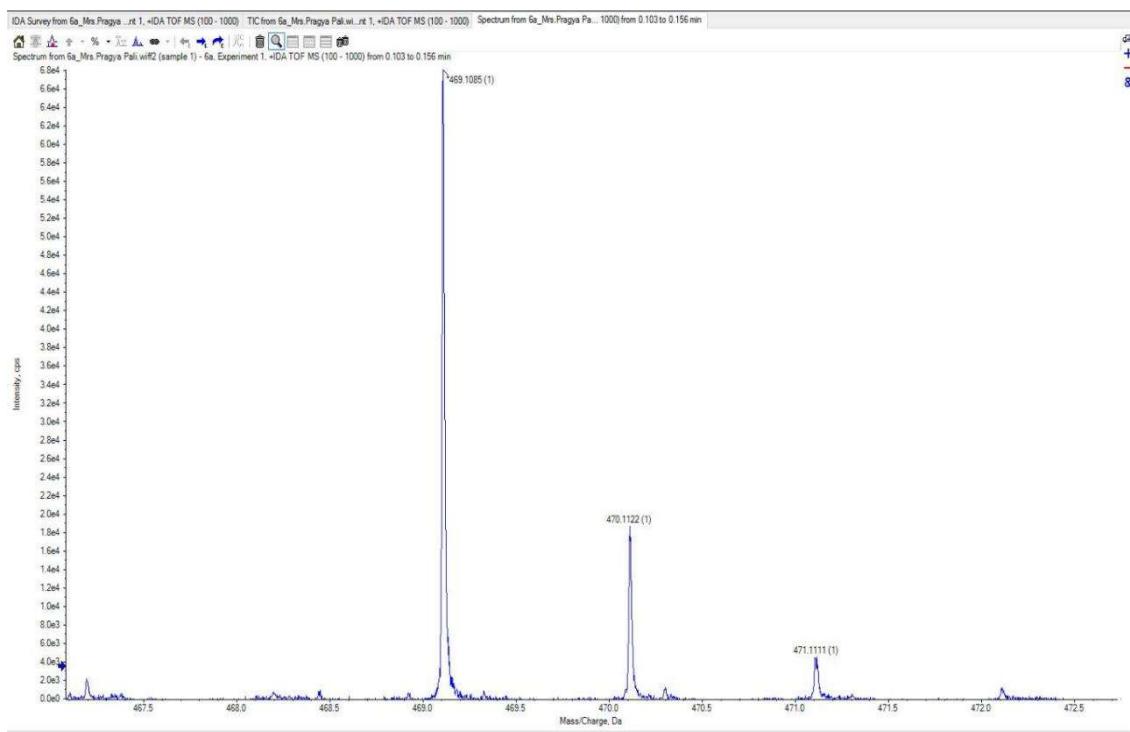
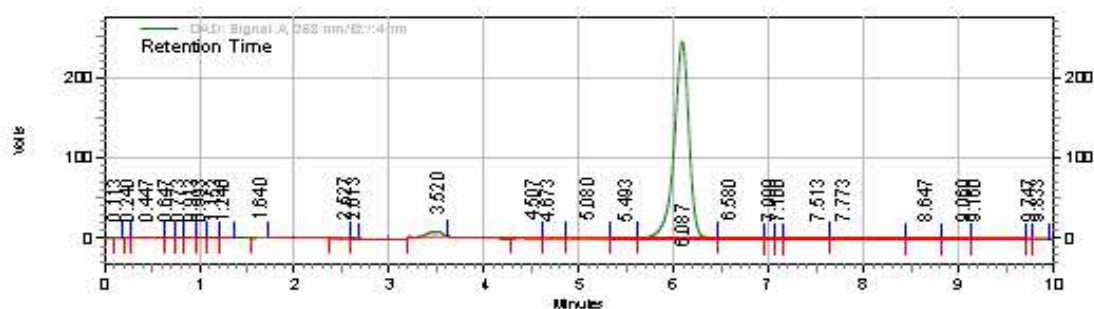


Figure 8. 4. Mass Spectra of compound 6a.



**DAD: Signal A,
258 nm/Bw:4 nm
Results**

Retention Time	Area	Area %	Height	Height %
2.527	41140	0.71	7365	1.35
2.613	18388	0.32	4633	0.85
3.520	134388	2.31	12415	2.27
4.507	10061	0.17	1255	0.23
4.673	3278	0.06	367	0.07
5.080	22330	0.38	2430	0.44
5.493	6605	0.11	722	0.13
6.087	5509097	94.54	510740	93.42
6.580	25492	0.44	1910	0.35
7.000	849	0.01	144	0.03
7.100	576	0.01	130	0.02
7.513	14149	0.24	913	0.17
7.773	21543	0.37	1149	0.21
8.647	2871	0.05	182	0.03
9.060	3665	0.06	260	0.05
9.160	4504	0.08	248	0.05
Totals	5827172	100.00	546706	100.00

Figure 8. 5. HPLC chromatogram and report of compound 6a.

Percentage purity of compound:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5µm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 µl
- ✓ Detection range: λ_{max} = 254

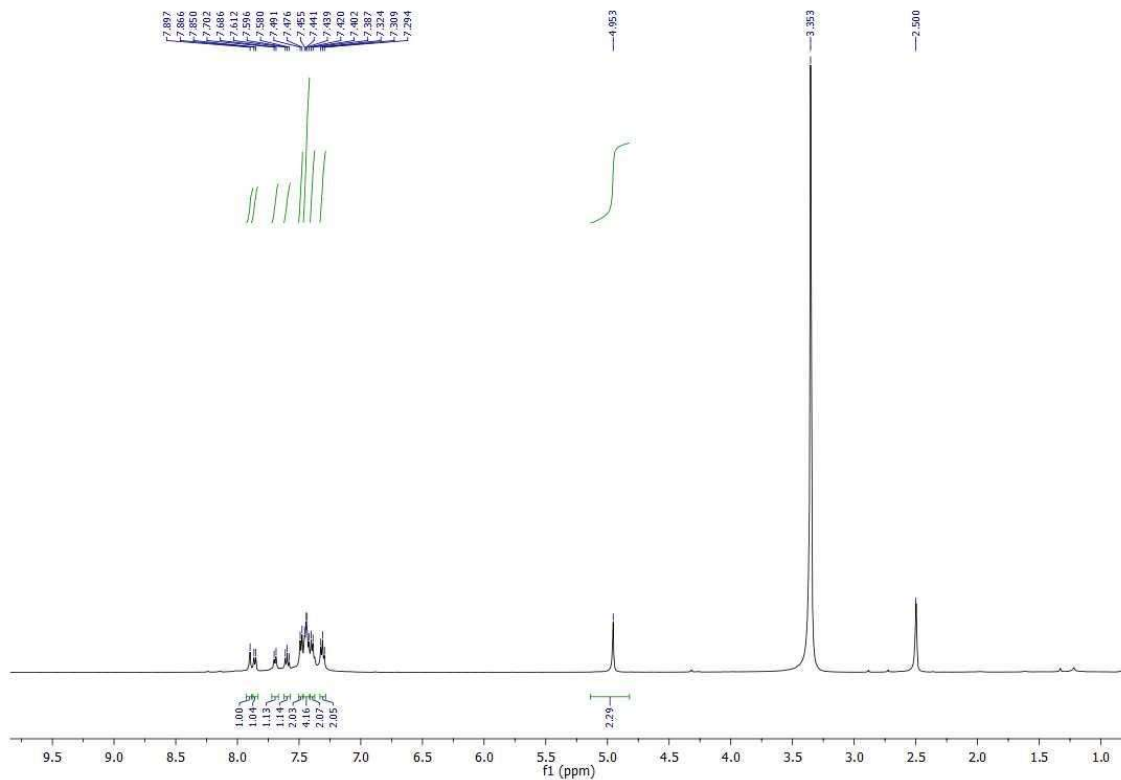


Figure 8. 6. ¹H NMR spectra of compound 6c

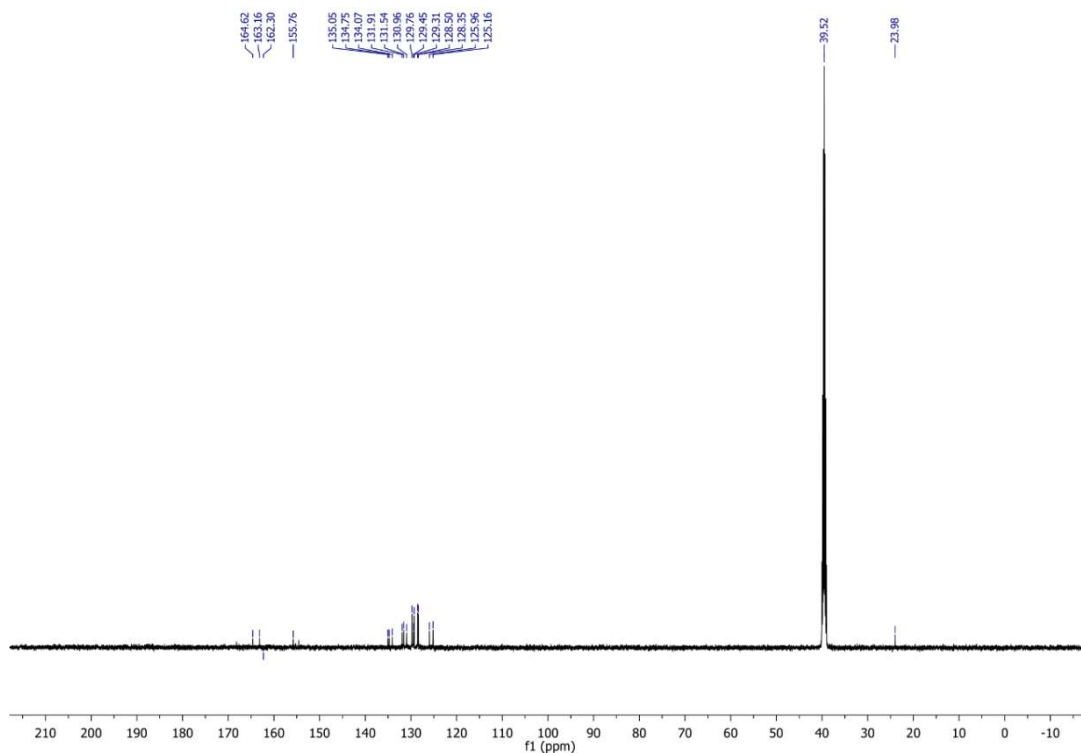


Figure 8. 7. ¹³C NMR spectra of compound 6c

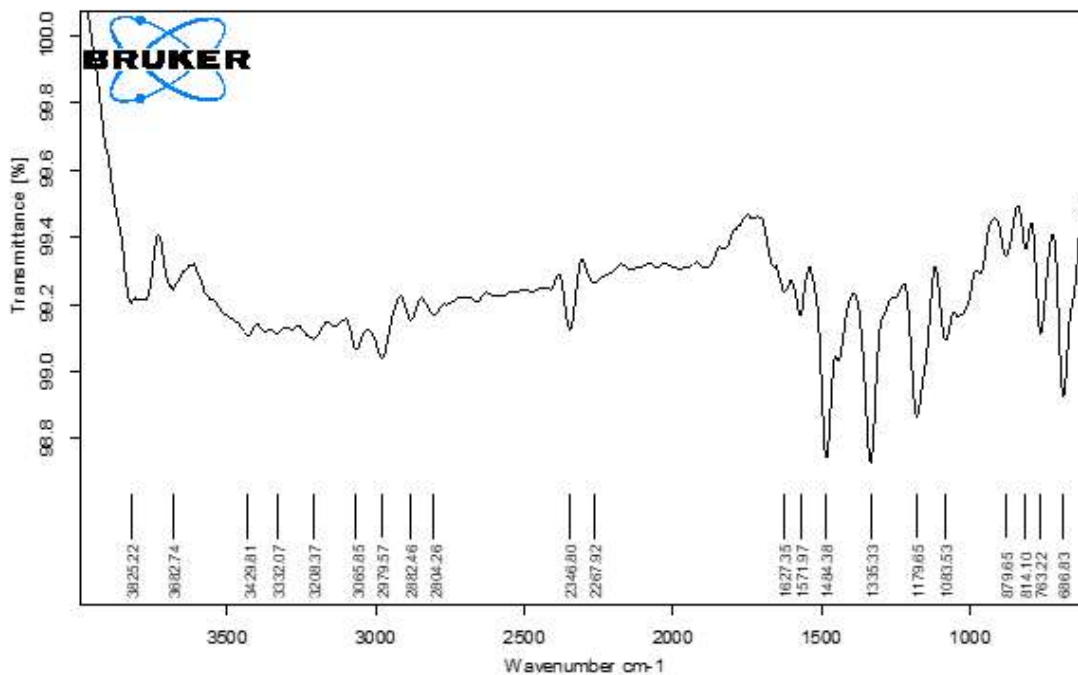


Figure 8. 8. FTIR spectra of compound 6c

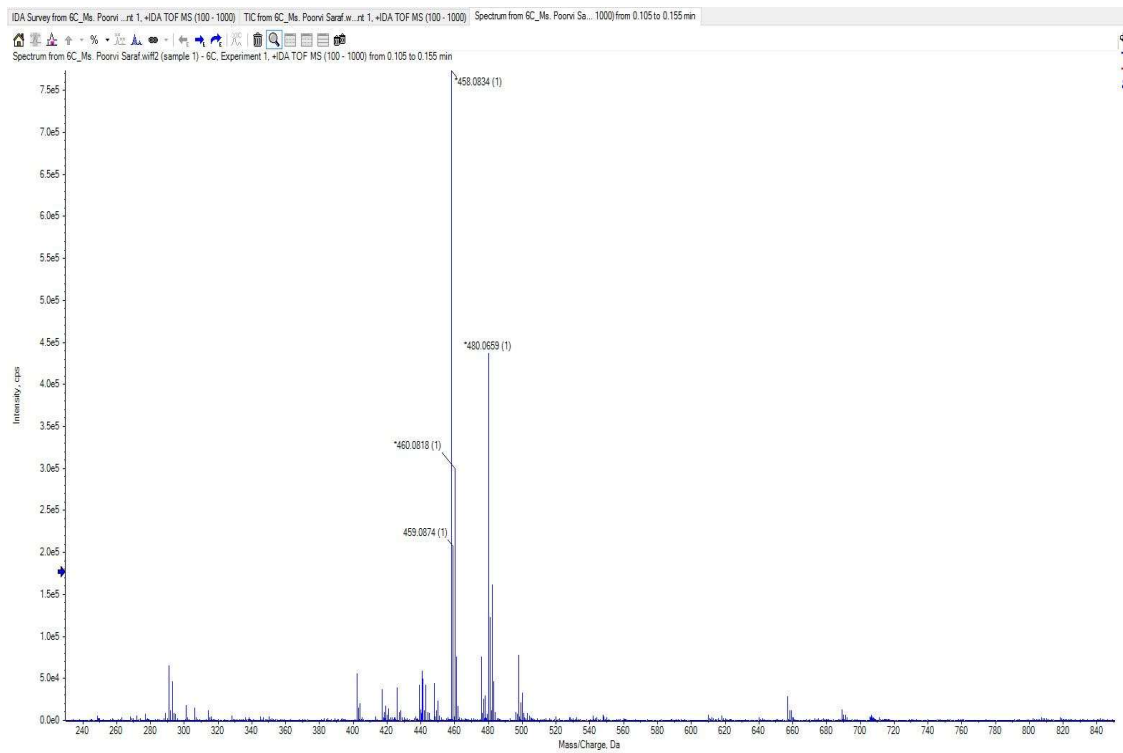
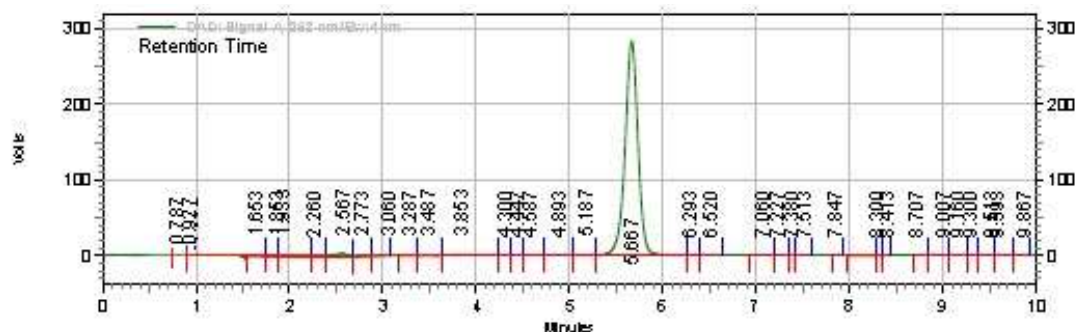


Figure 8. 9. Mass Spectra of compound 6c.



DAD: Signal A,
258 nm/Bw:4 nm

Results

Retention Time	Area	Area %	Height	Height %
1.653	6699	0.11	667	0.11
1.853	12707	0.21	2079	0.33
1.933	48459	0.81	2604	0.42
2.260	32023	0.53	2891	0.46
2.567	98708	1.64	9576	1.54
2.773	34955	0.58	4560	0.73
3.060	15019	0.25	628	0.10
3.287	2706	0.04	342	0.05
3.487	12615	0.21	1748	0.28
3.853	14632	0.24	1086	0.17
4.893	2543	0.04	324	0.05
5.187	9331	0.16	1516	0.24
5.667	5716873	95.04	592828	95.31
6.293	992	0.02	190	0.03
Totals	6015243	100.00	622027	100.00

Figure 8. 10. HPLC chromatogram and report of compound 6c.

Percentage purity of compound:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5µm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 µl
- ✓ Detection range: λ_{max} = 254

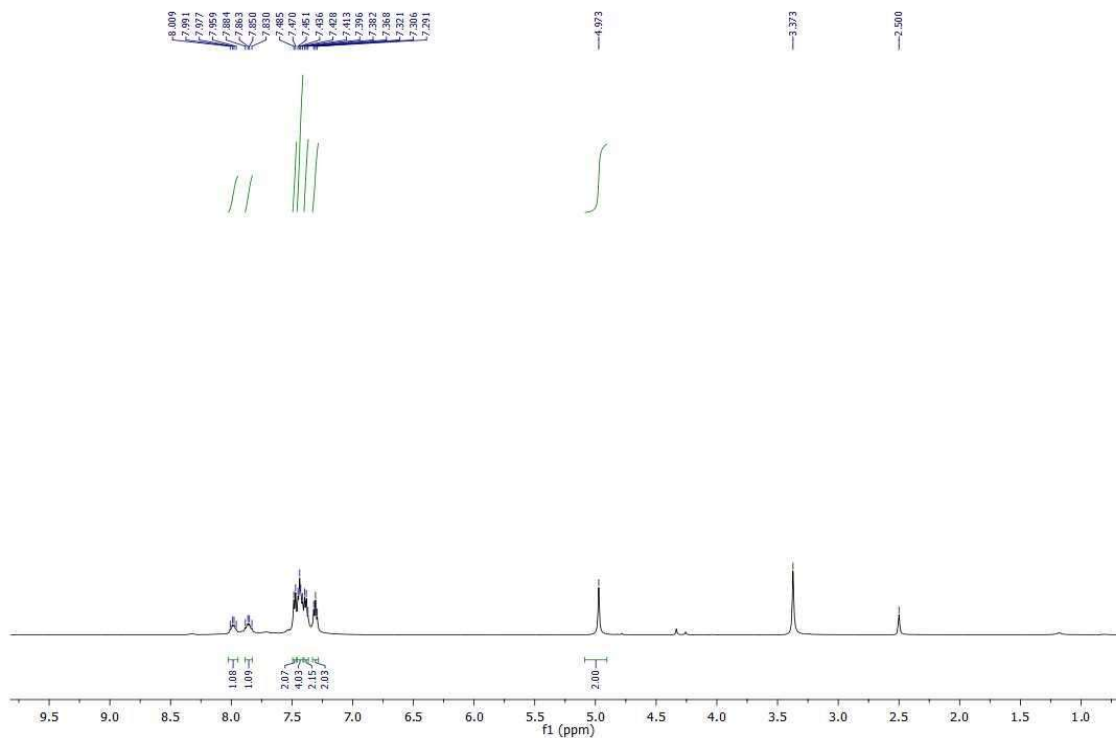


Figure 8. 11. ¹H NMR spectra of compound 6h

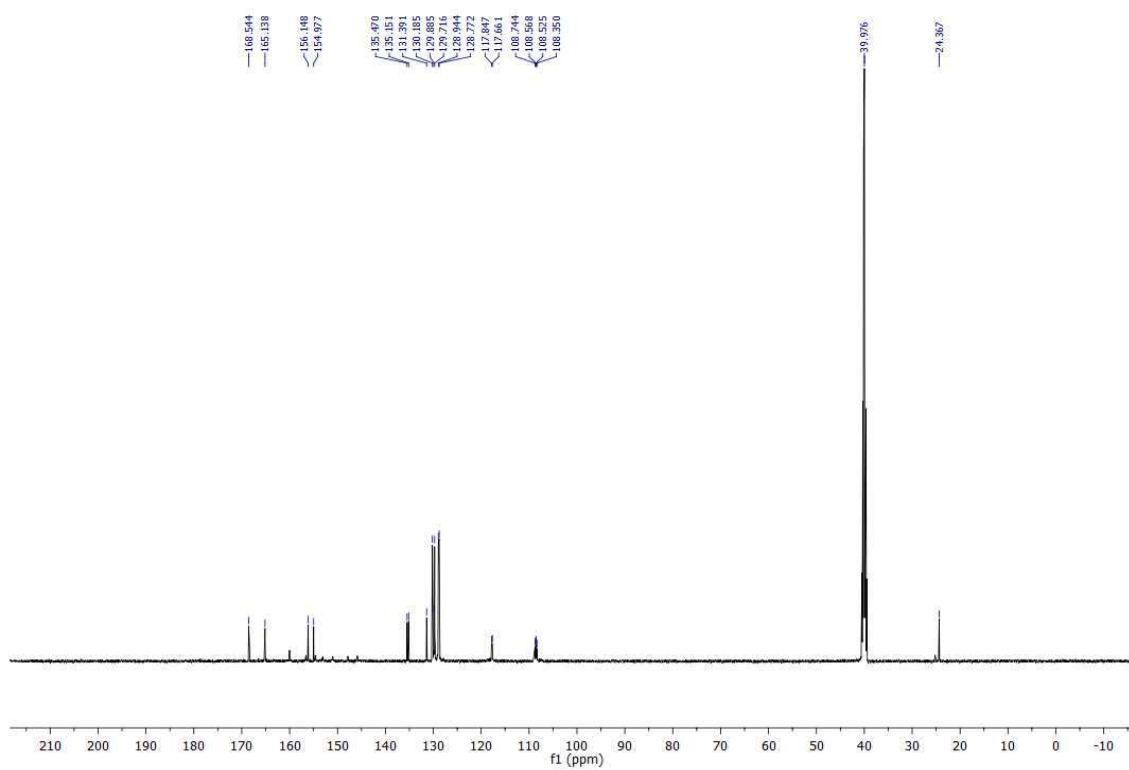


Figure 8. 12. ¹³C NMR spectra of compound 6h

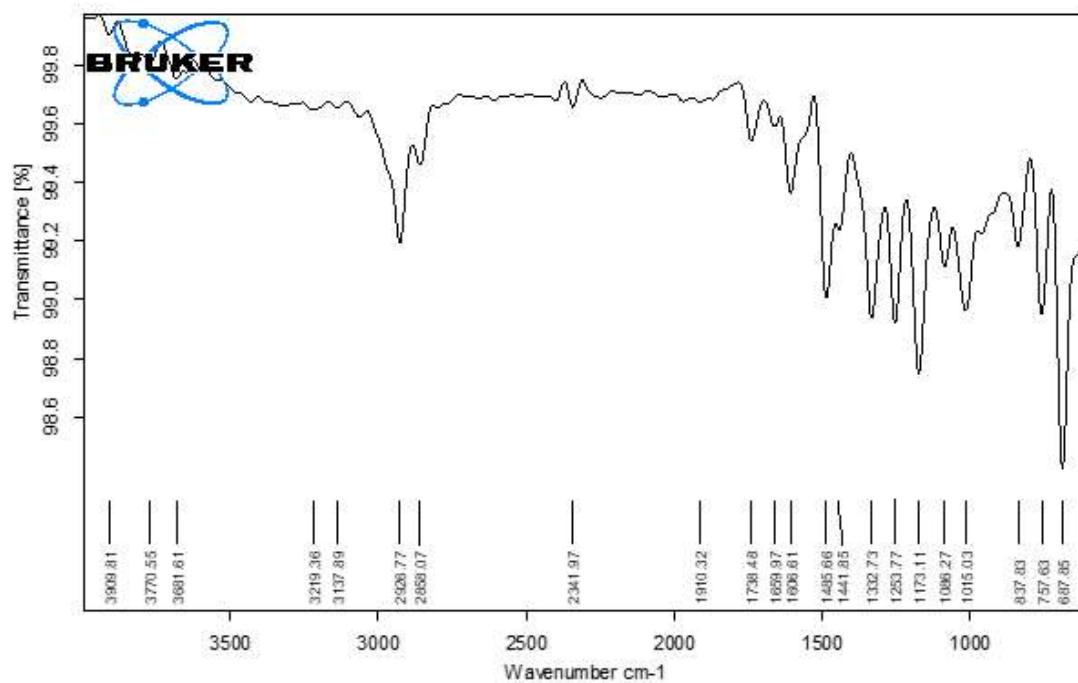


Figure 8. 13. FTIR spectra of compound 6h

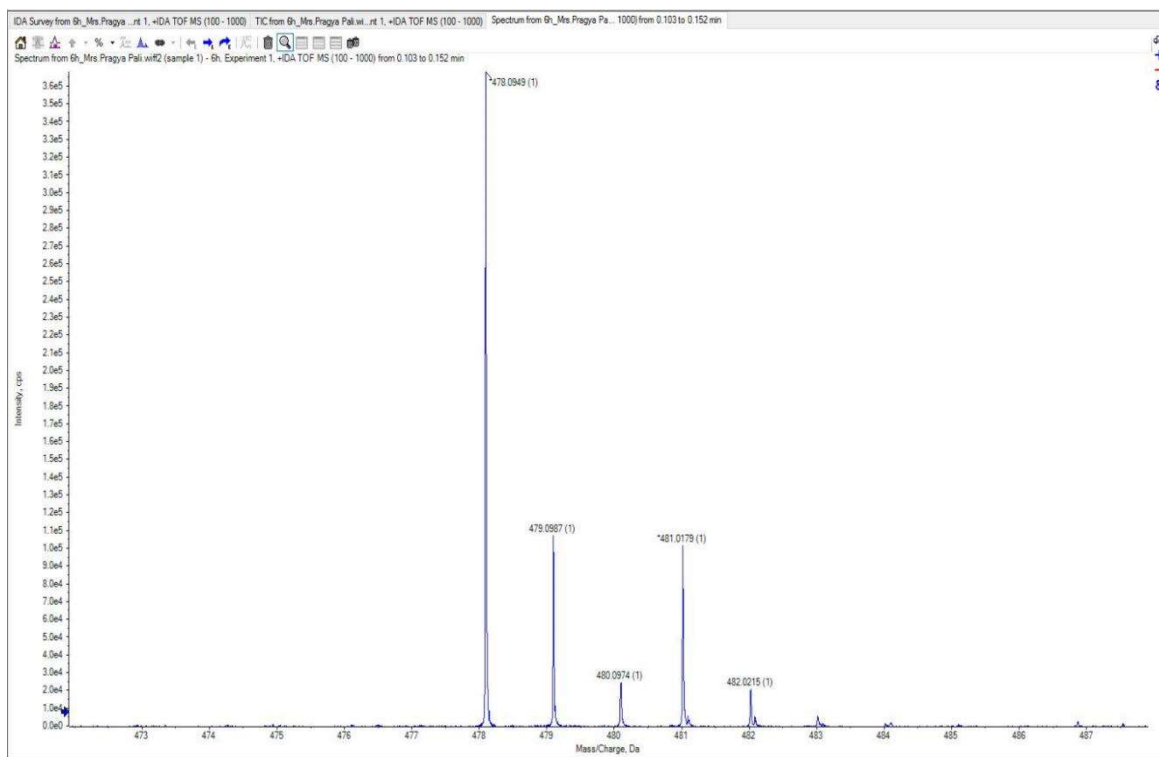
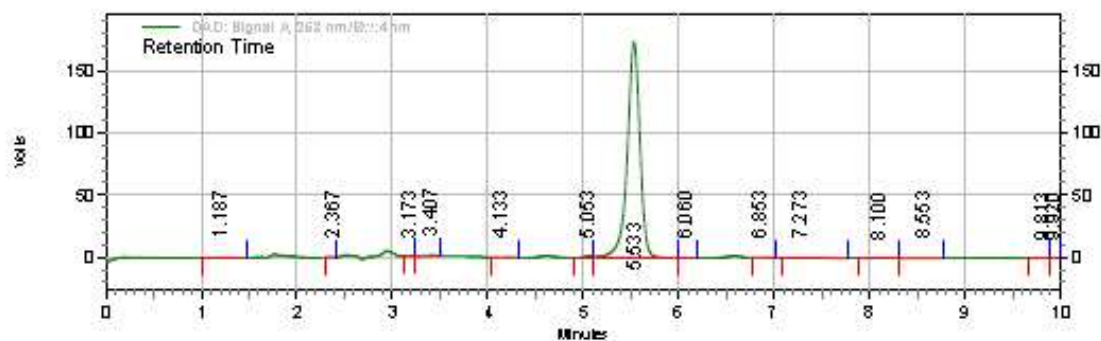


Figure 8. 14. Mass Spectra of compound 6h.



DAD: Signal A,
258 nm/Bw:4 nm
Results

Retention Time	Area	Area %	Height	Height %
1.187	3226	0.09	228	0.06
2.367	1229	0.04	317	0.09
3.173	622	0.02	203	0.05
3.407	11369	0.33	1697	0.46
4.133	4748	0.14	490	0.13
5.053	12268	0.36	1848	0.50
5.533	3368117	98.32	364087	98.16
6.060	2220	0.06	317	0.09
6.853	4378	0.13	477	0.13
7.273	11062	0.32	686	0.18
8.100	2763	0.08	228	0.06
8.553	3035	0.09	254	0.07
9.813	370	0.01	49	0.01
9.920	154	0.00	39	0.01
Totals	3425561	100.00	370920	100.00

Figure 8. 15. HPLC chromatogram and report of compound 6h.

Percentage purity of compound:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5µm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 µl
- ✓ Detection range: λ_{max} = 254

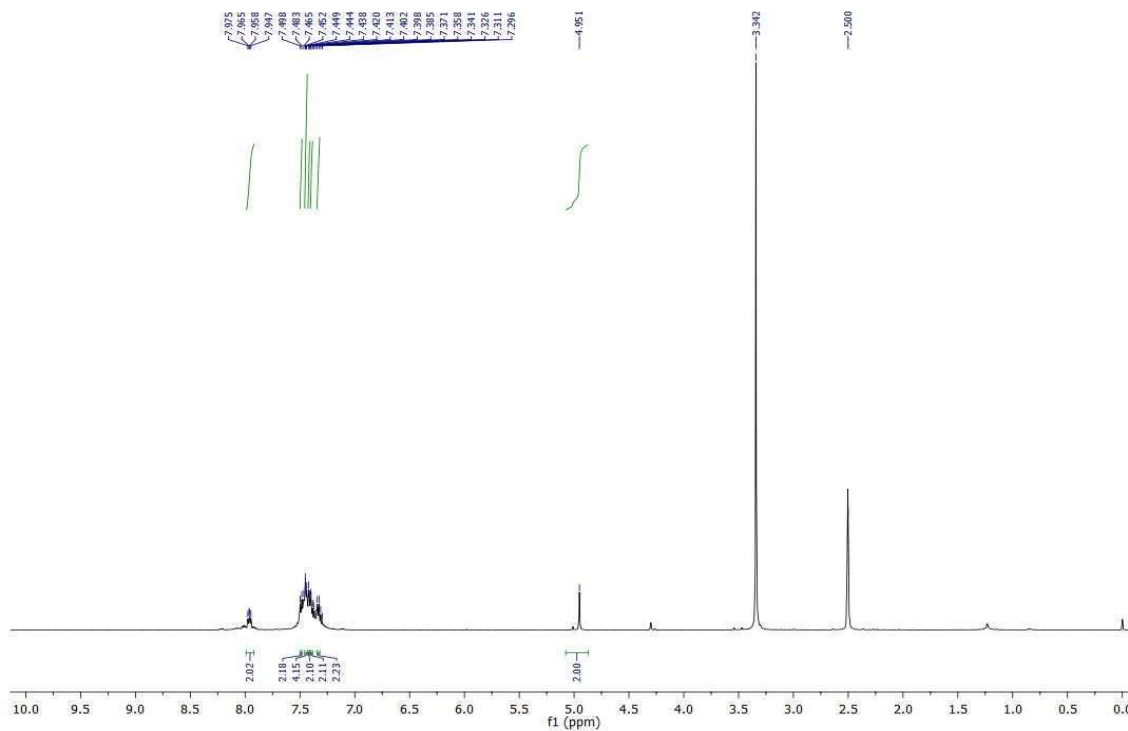


Figure 8. 16. ¹H NMR spectra of compound 6k

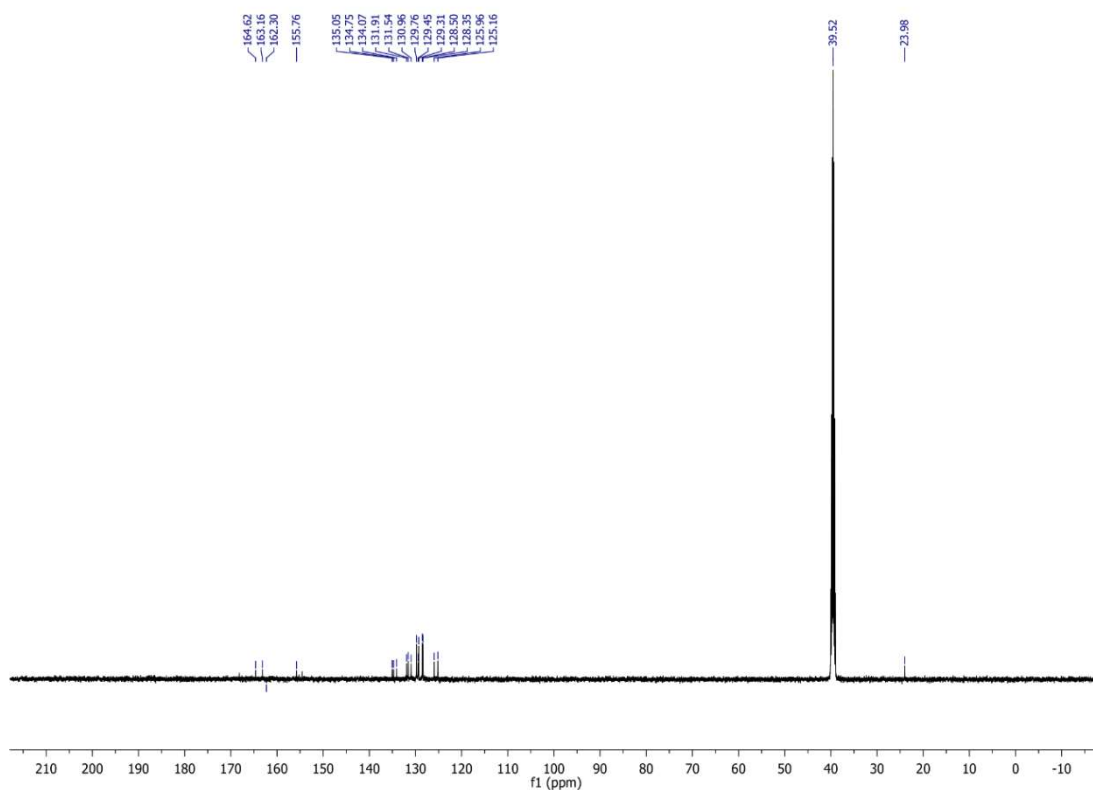


Figure 8. 17. ¹³C NMR spectra of compound 6k

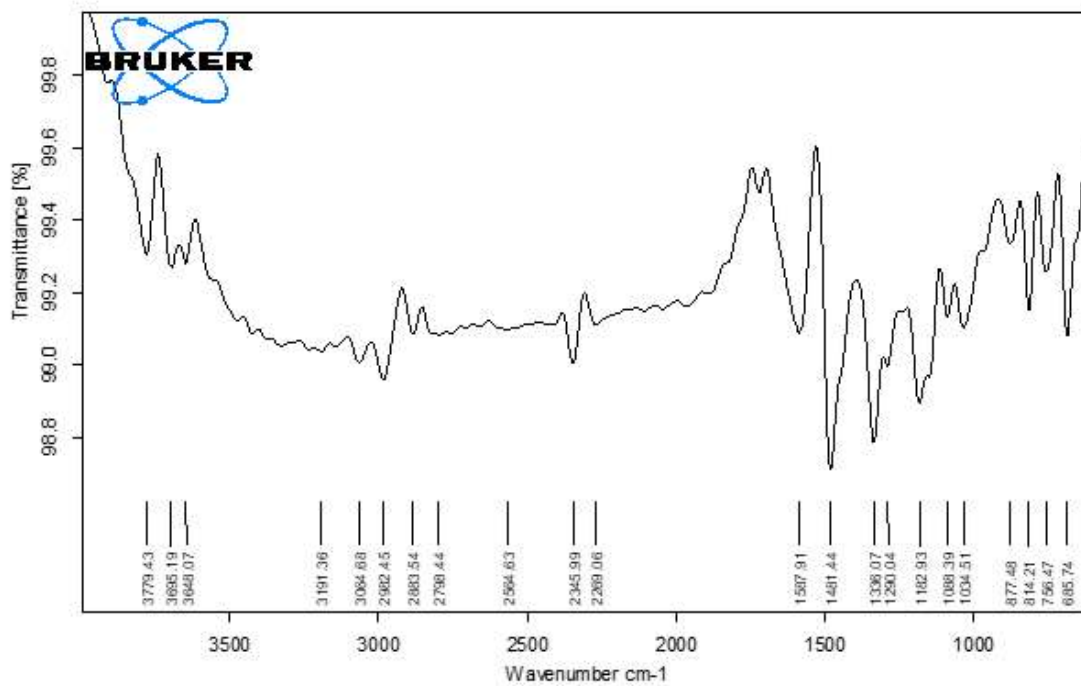


Figure 8. 18. FTIR spectra of compound 6k

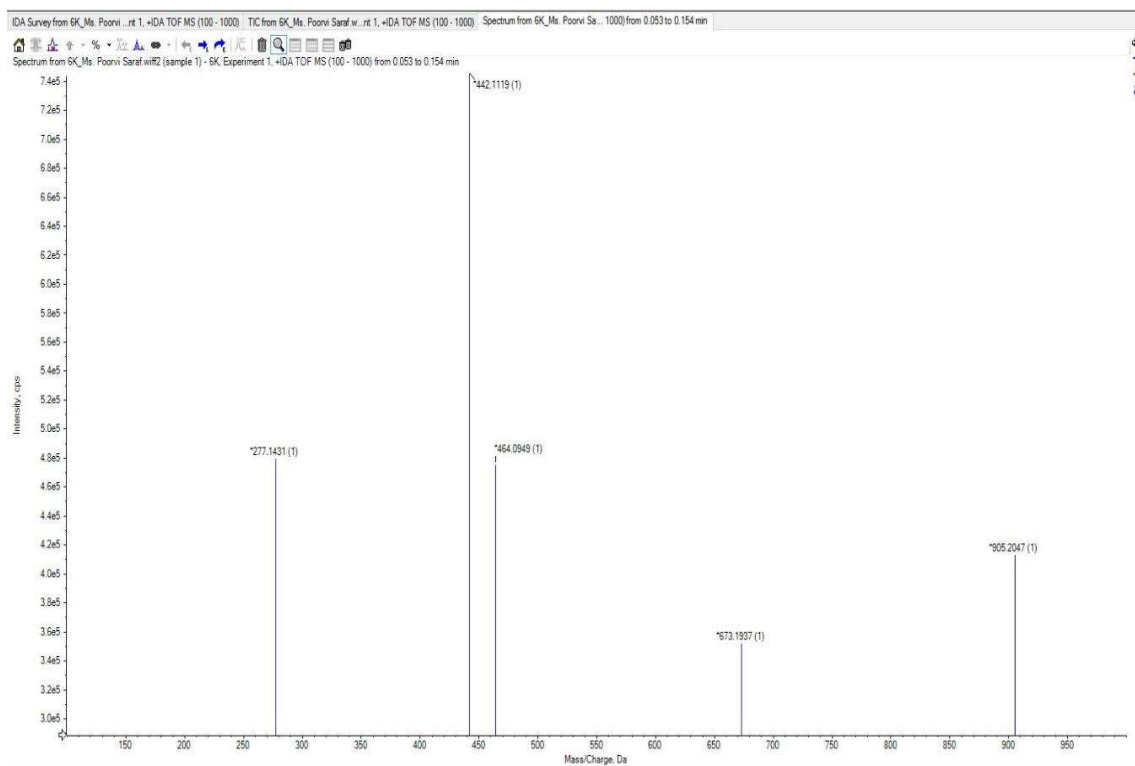
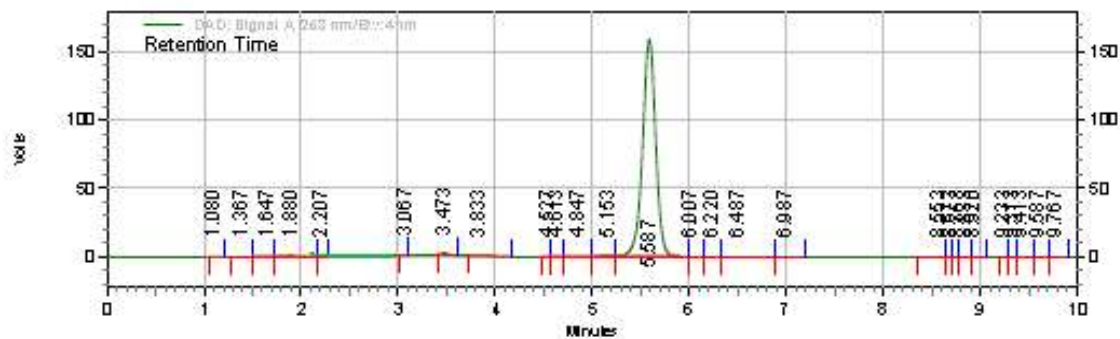


Figure 8. 19. Mass Spectra of compound 6k



**DAD: Signal A,
258 nm/Bw:4 nm
Results**

Retention Time	Area	Area %	Height	Height %
1.080	217	0.01	33	0.01
1.367	241	0.01	36	0.01
1.647	2263	0.07	319	0.09
1.880	11461	0.36	1026	0.30
2.207	364	0.01	84	0.02
3.067	1072	0.03	283	0.08
3.473	8774	0.27	1529	0.45
3.833	5965	0.19	619	0.18
4.847	1538	0.05	185	0.05
5.153	9094	0.28	1513	0.44
5.587	3142532	98.42	333355	98.04
6.007	1778	0.06	263	0.08
6.220	744	0.02	117	0.03
6.487	3384	0.11	153	0.04
6.987	1234	0.04	104	0.03
Totals	3193054	100.00	340020	100.00

Figure 8. 20. HPLC chromatogram and report of compound 6k.

Percentage purity of compound:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5µm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 µl
- ✓ Detection range: λ_{max} = 254

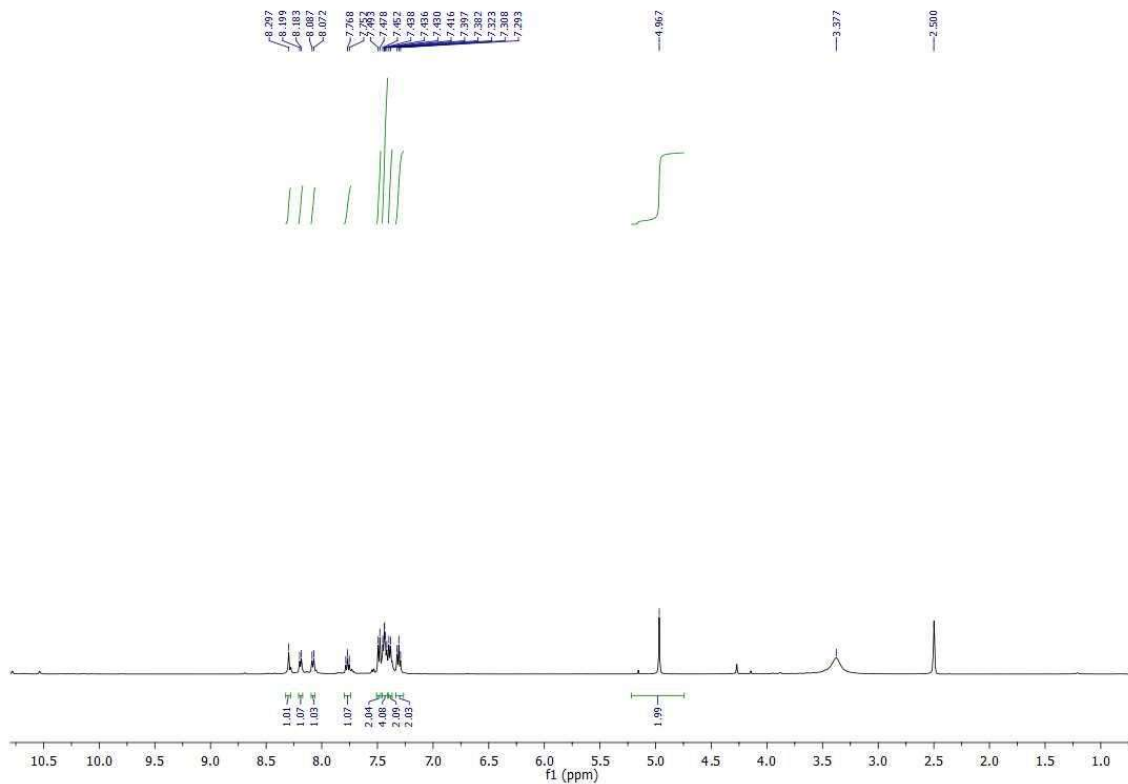


Figure 8. 21. ¹H NMR spectra of compound 6n

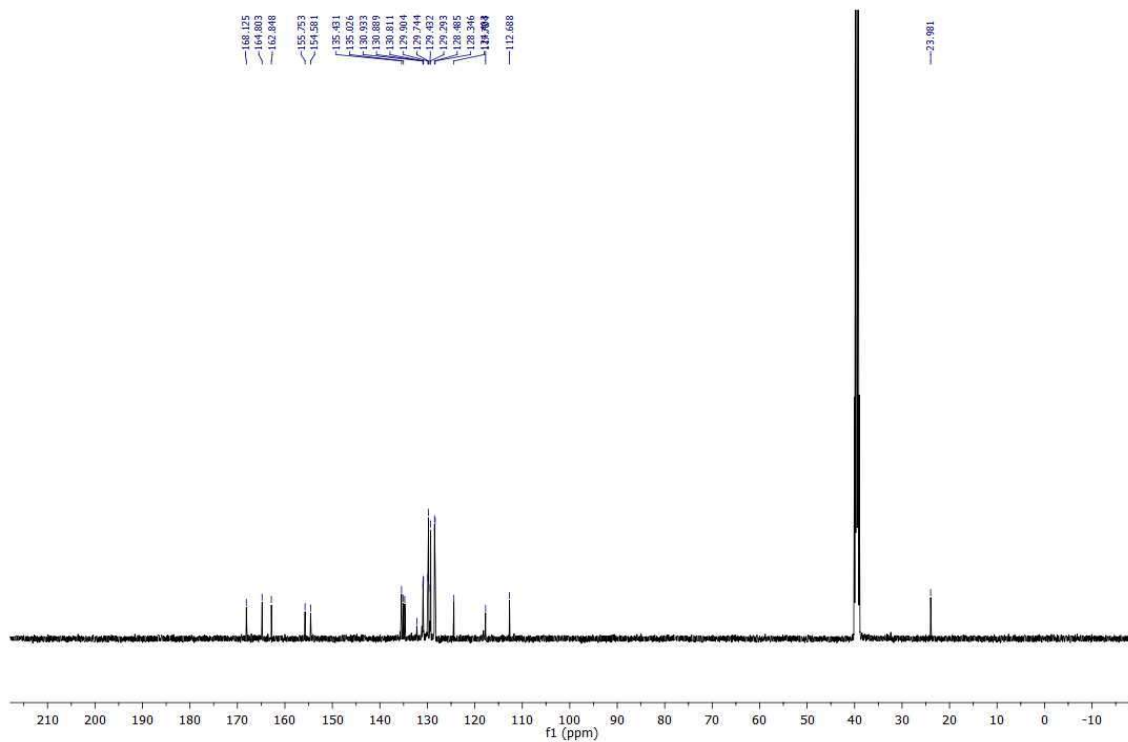


Figure 8. 22. ¹³C NMR spectra of compound 6n

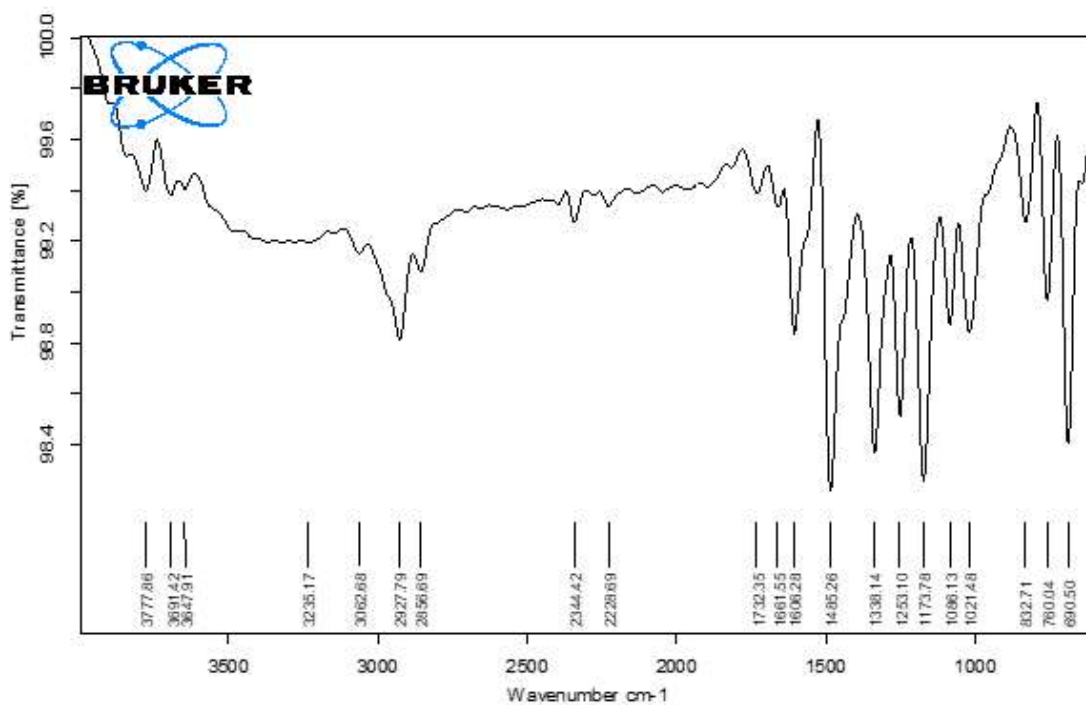


Figure 8. 23. FTIR spectra of compound 6n

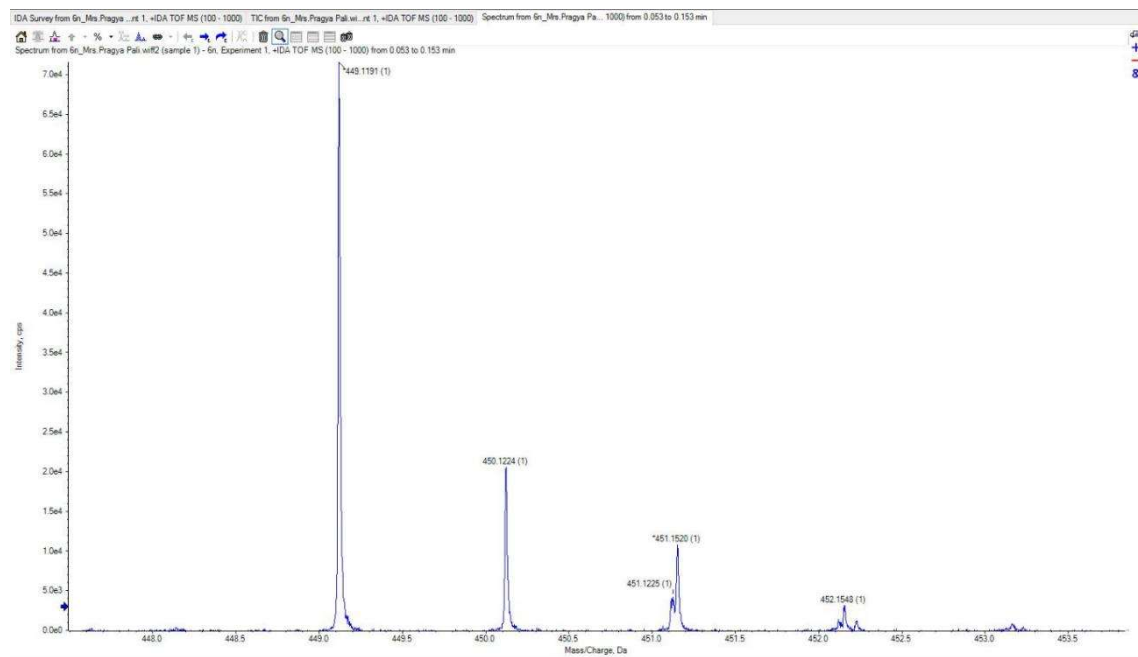
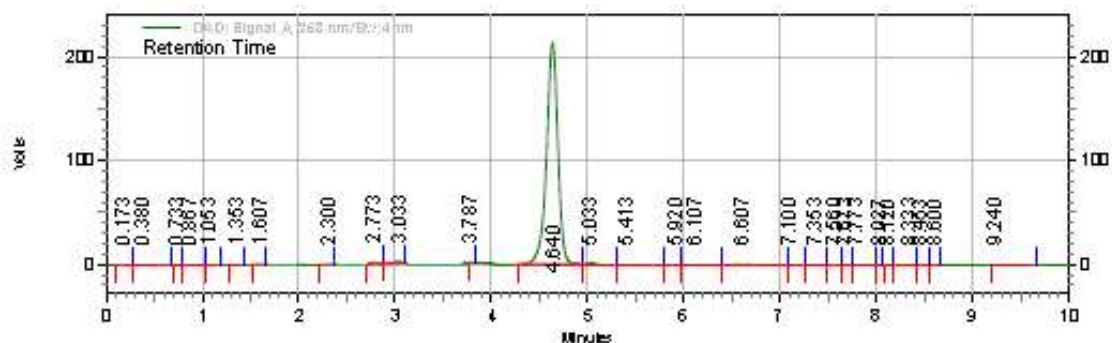


Figure 8. 24. Mass Spectra of compound 6n.



**DAD: Signal A,
258 nm/Bw:4 nm
Results**

Retention Time	Area	Area %	Height	Height %
0.173	358	0.01	43	0.01
0.380	1166	0.03	85	0.02
1.053	137	0.00	26	0.01
1.353	403	0.01	92	0.02
1.607	2244	0.06	531	0.12
2.300	910	0.02	223	0.05
2.773	21562	0.57	3672	0.80
3.033	17709	0.47	2990	0.65
3.787	455	0.01	176	0.04
4.640	3684579	97.16	447282	97.16
5.033	16101	0.42	1871	0.41
5.413	8575	0.23	376	0.08
5.920	1616	0.04	226	0.05
6.107	10443	0.28	584	0.13
6.607	20366	0.54	1558	0.34
7.100	845	0.02	81	0.02
Totals	3792379	100.00	460363	100.00

Figure 8. 25. HPLC chromatogram and report of compound 6n.

Percentage purity of compound:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5µm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 µl
- ✓ Detection range: λ_{max} = 254

¹H and ¹³C spectra of representative intermediate compounds
SERIES II

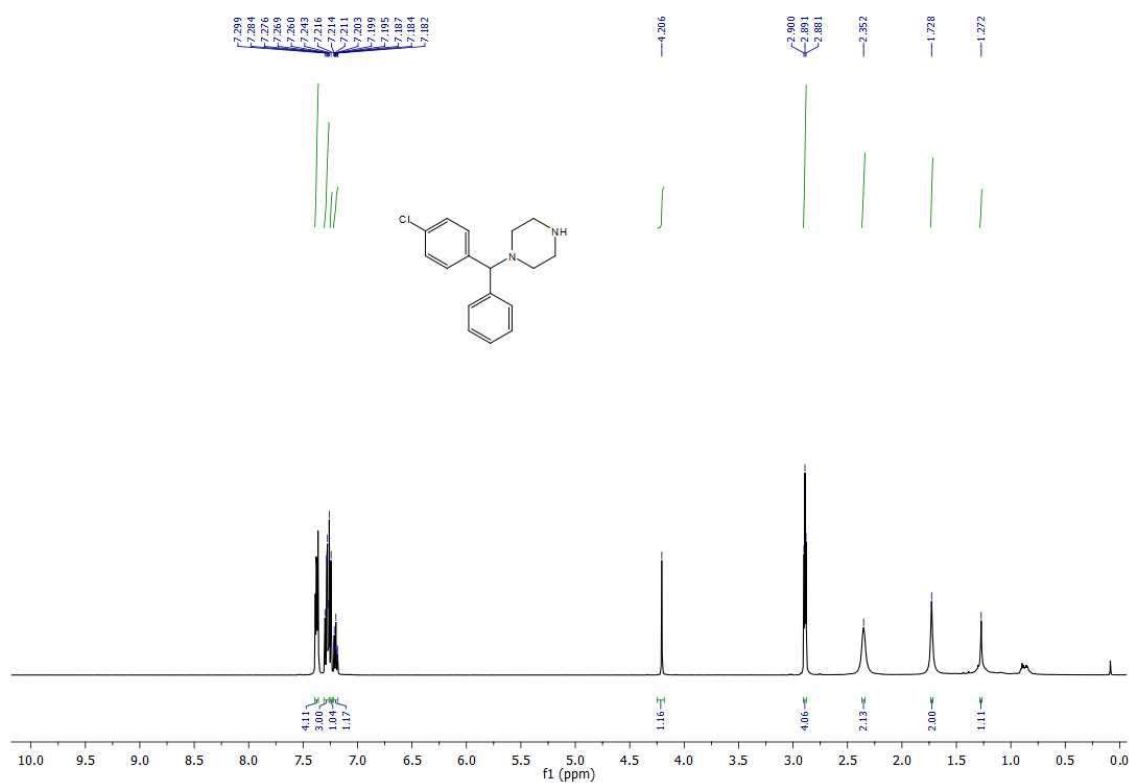


Figure 8.26. ¹H NMR spectra of target compound 5.

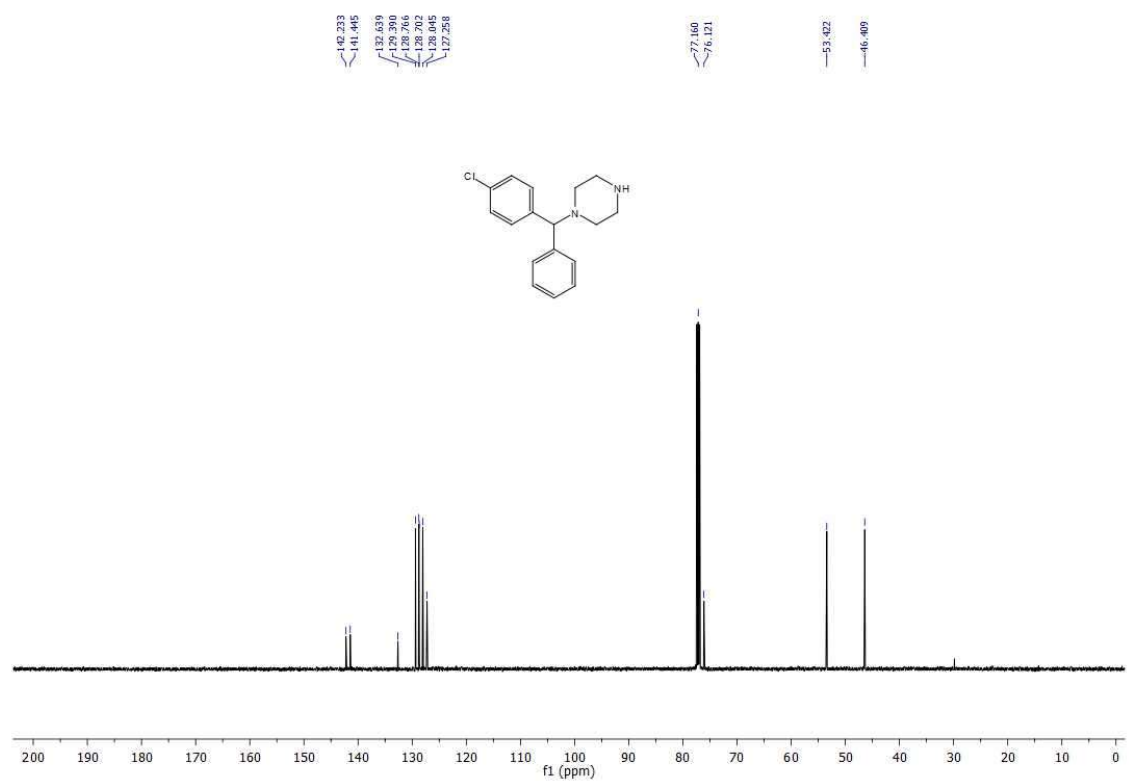


Figure 8.27. ¹³C NMR spectra of target compound 5.

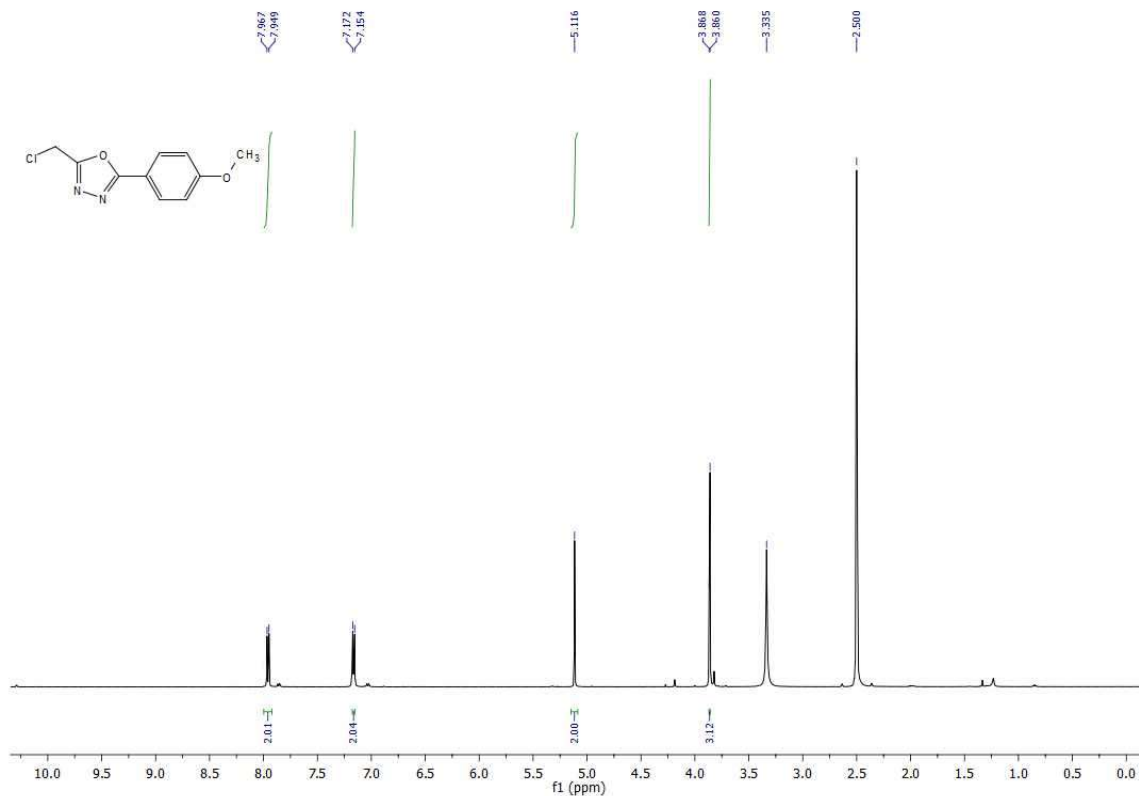


Figure 8. 28. ¹H NMR spectra of target compound 8b

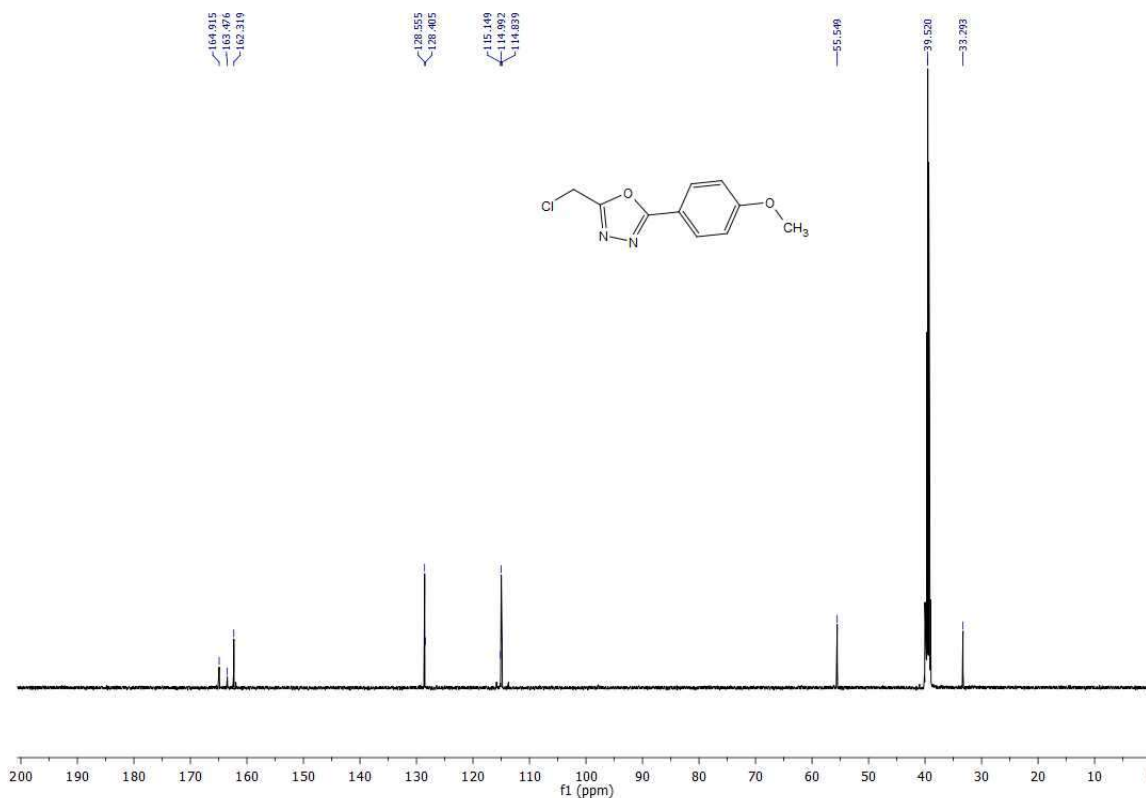


Figure 8. 29. ¹³C NMR spectra of target compound 8b

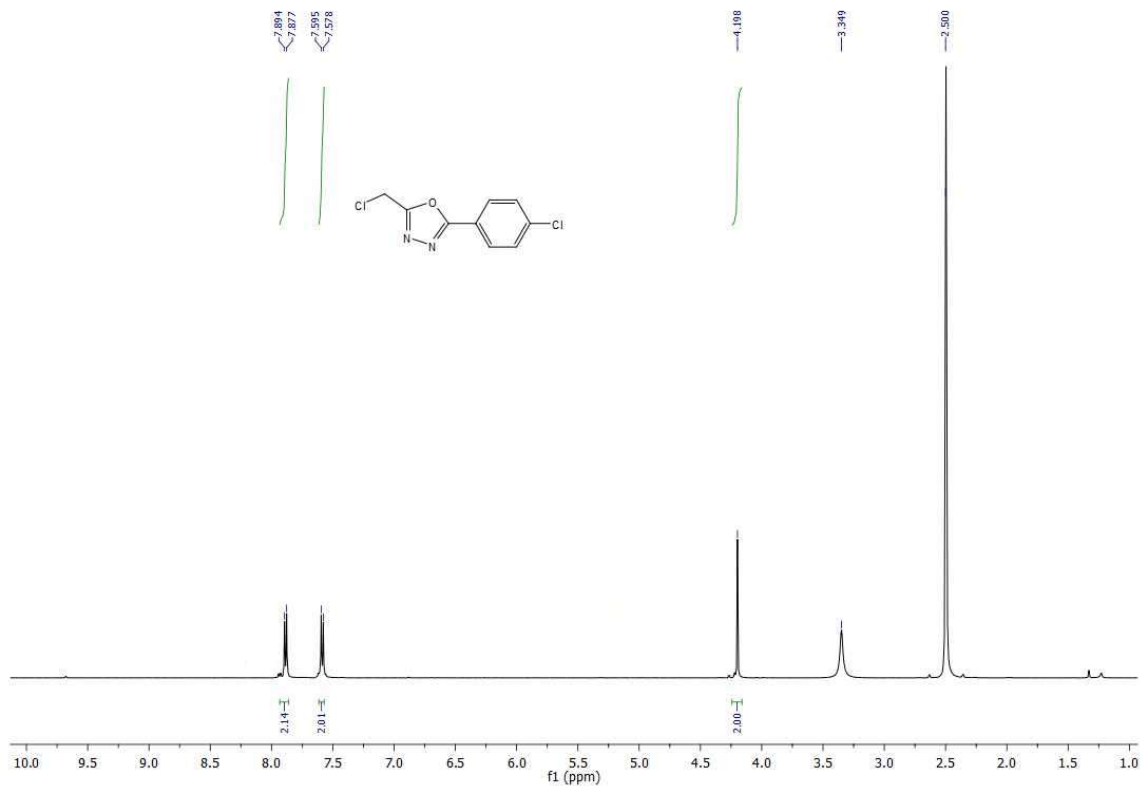


Figure 8. 30. ¹H NMR spectra of target compound 8d.

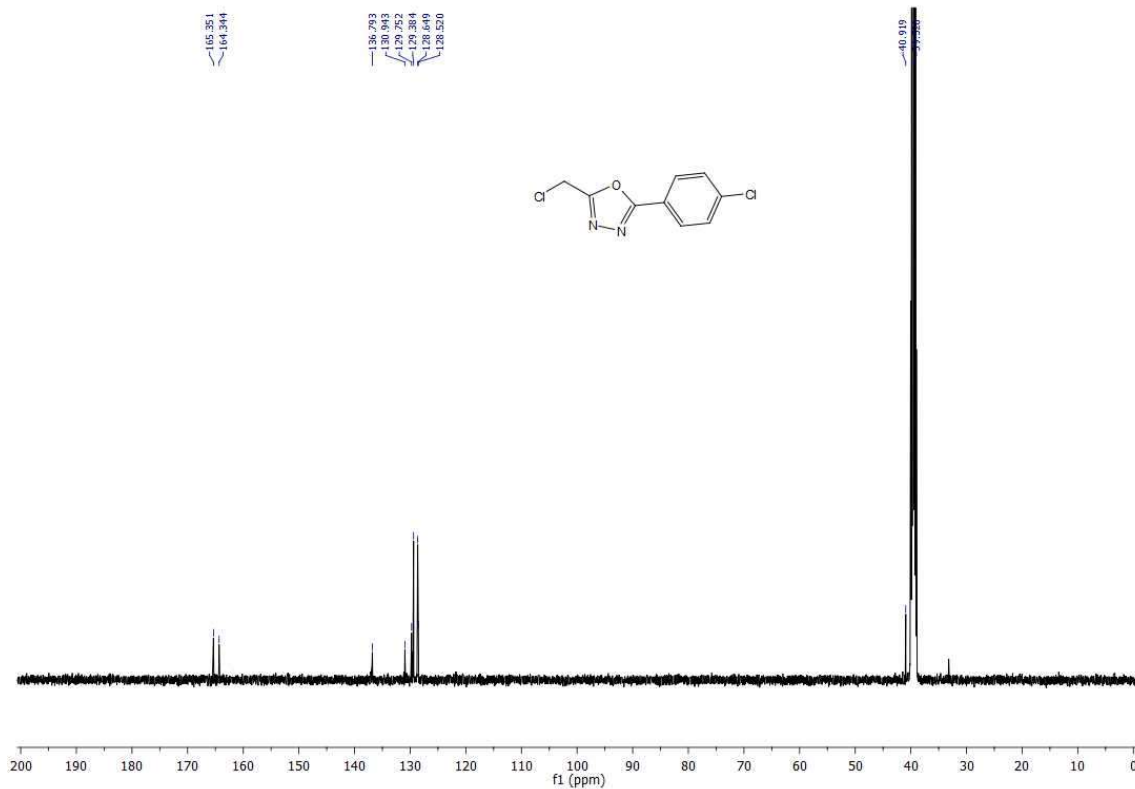


Figure 8. 31. ¹³C NMR spectra of target compound 8d.

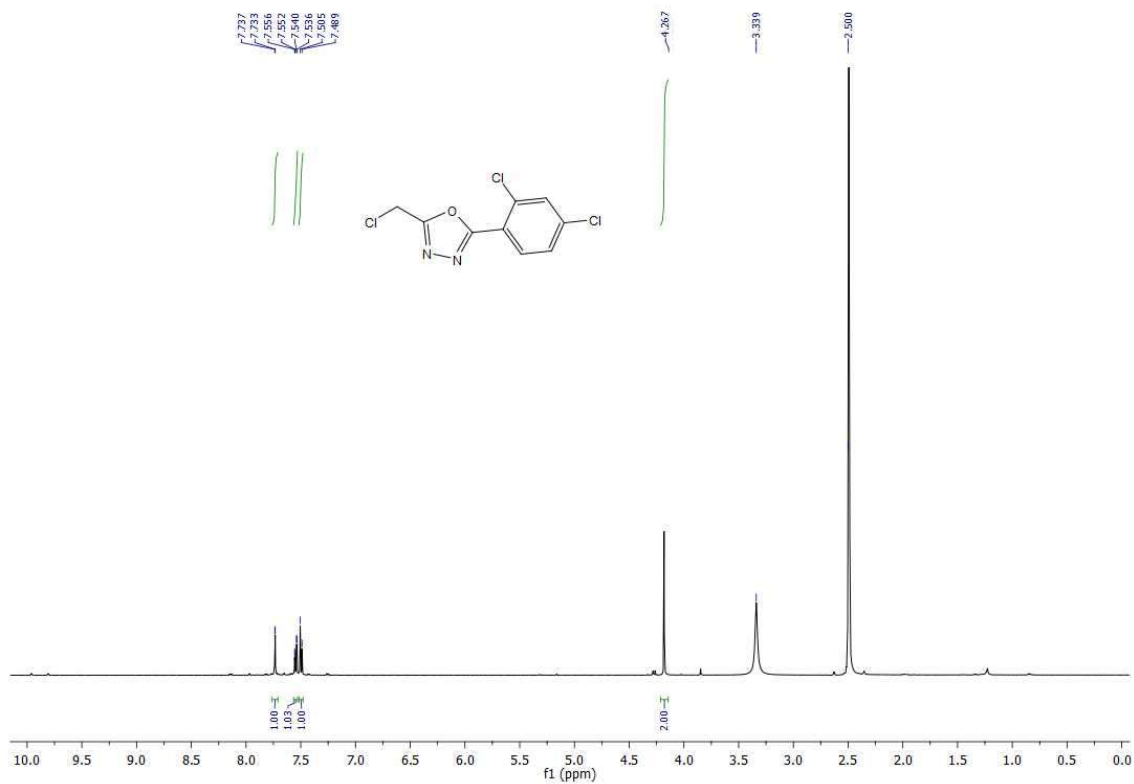


Figure 8. 32. ¹H NMR spectra of target compound 8e.

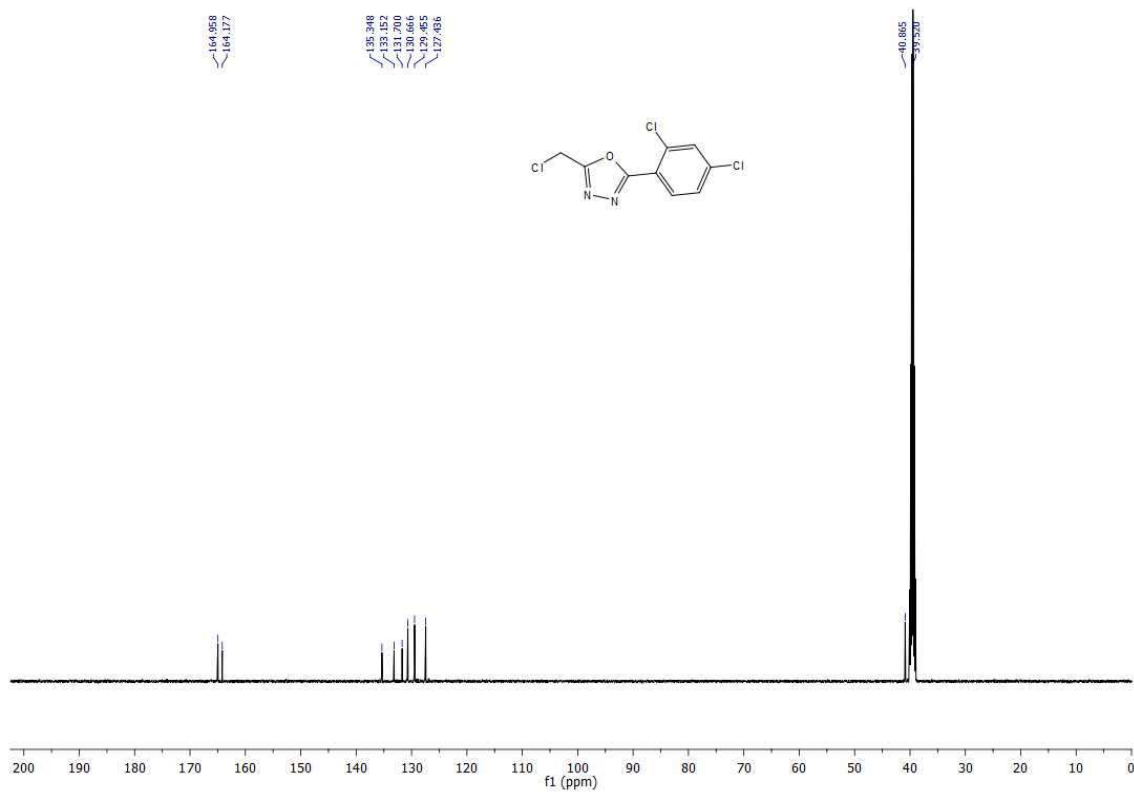


Figure 8. 33. ¹³C NMR spectra of target compound 8e.

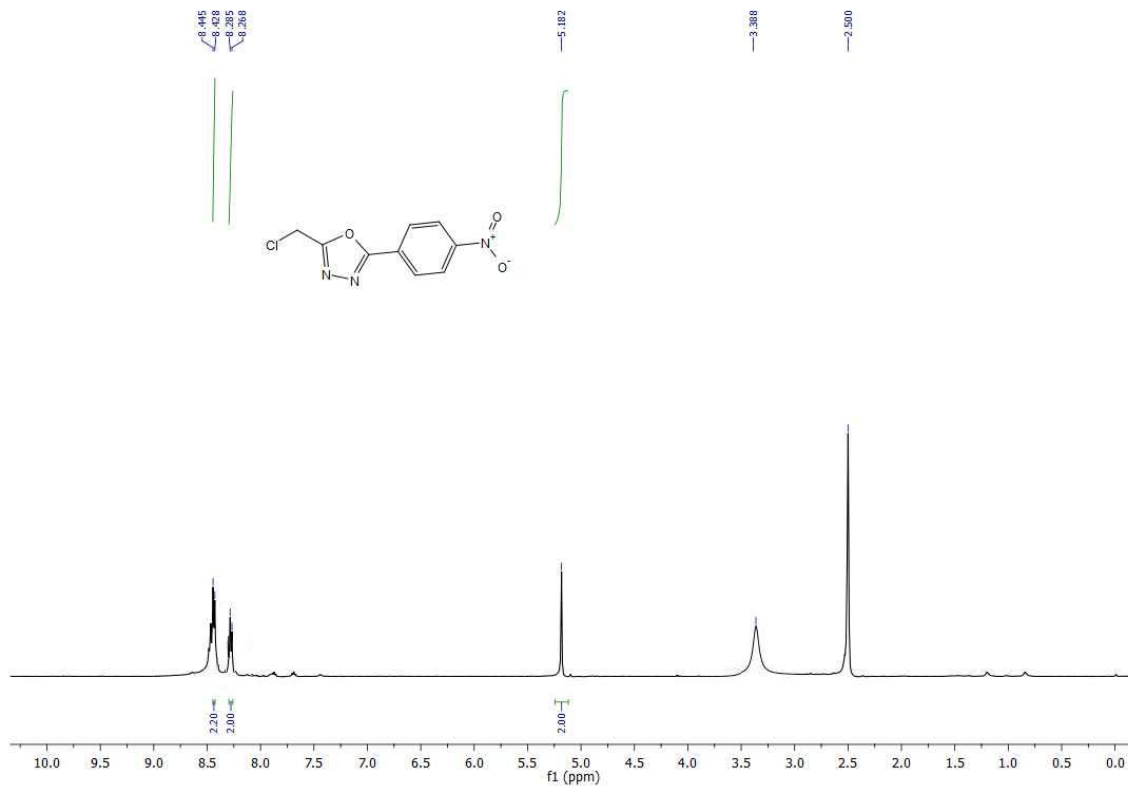


Figure 8. 34. ¹H NMR spectra of target compound 8g.

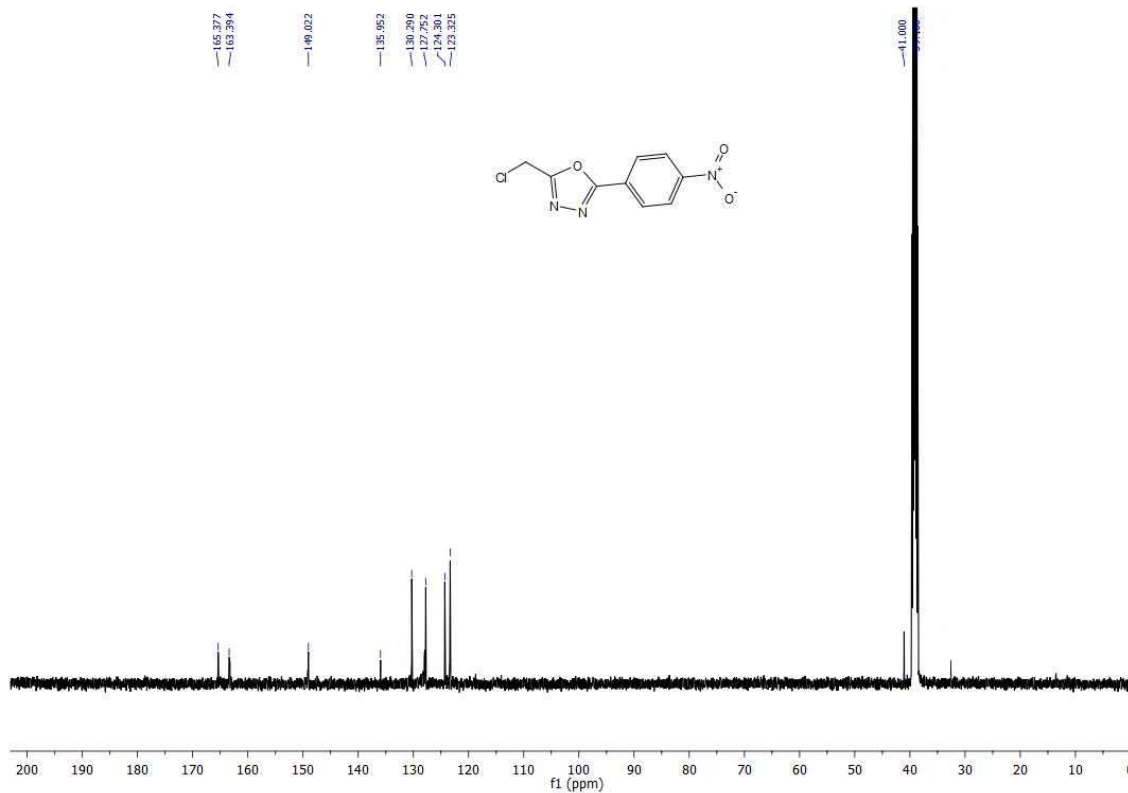


Figure 8. 35. ¹³C NMR spectra of target compound 8g.

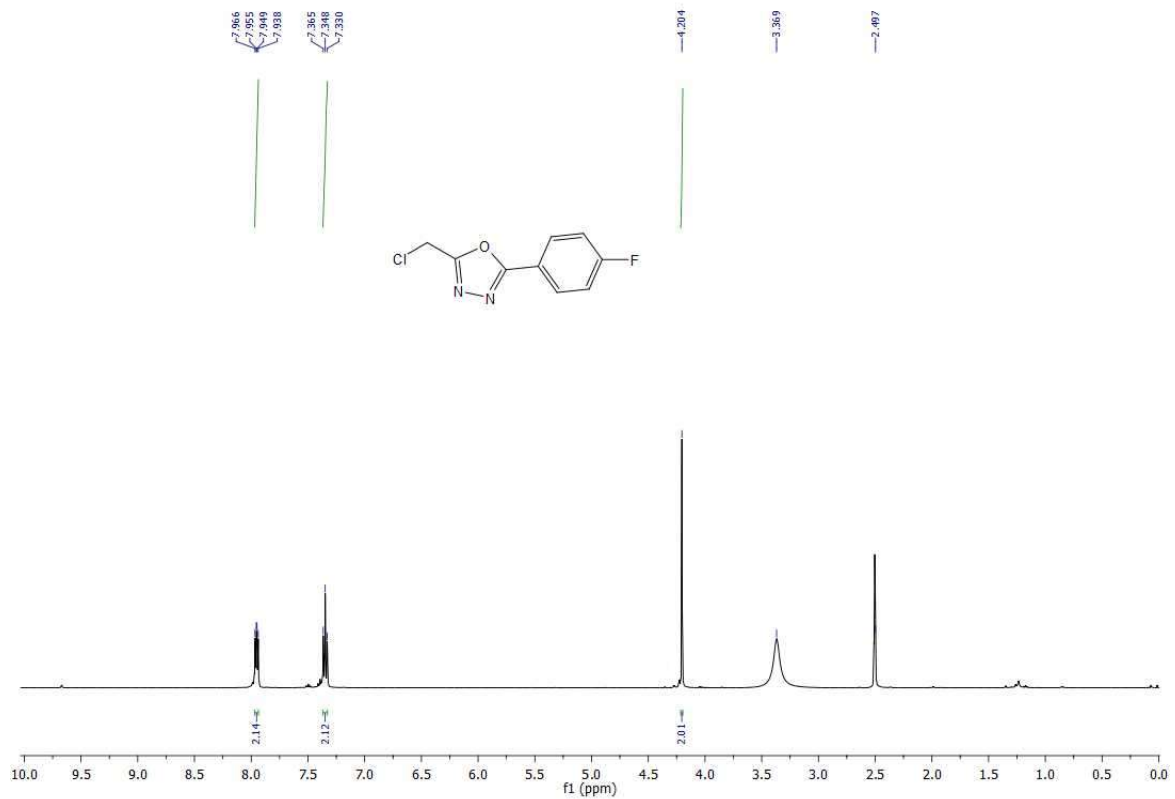


Figure 8. 36. ¹H NMR spectra of target compound 8l.

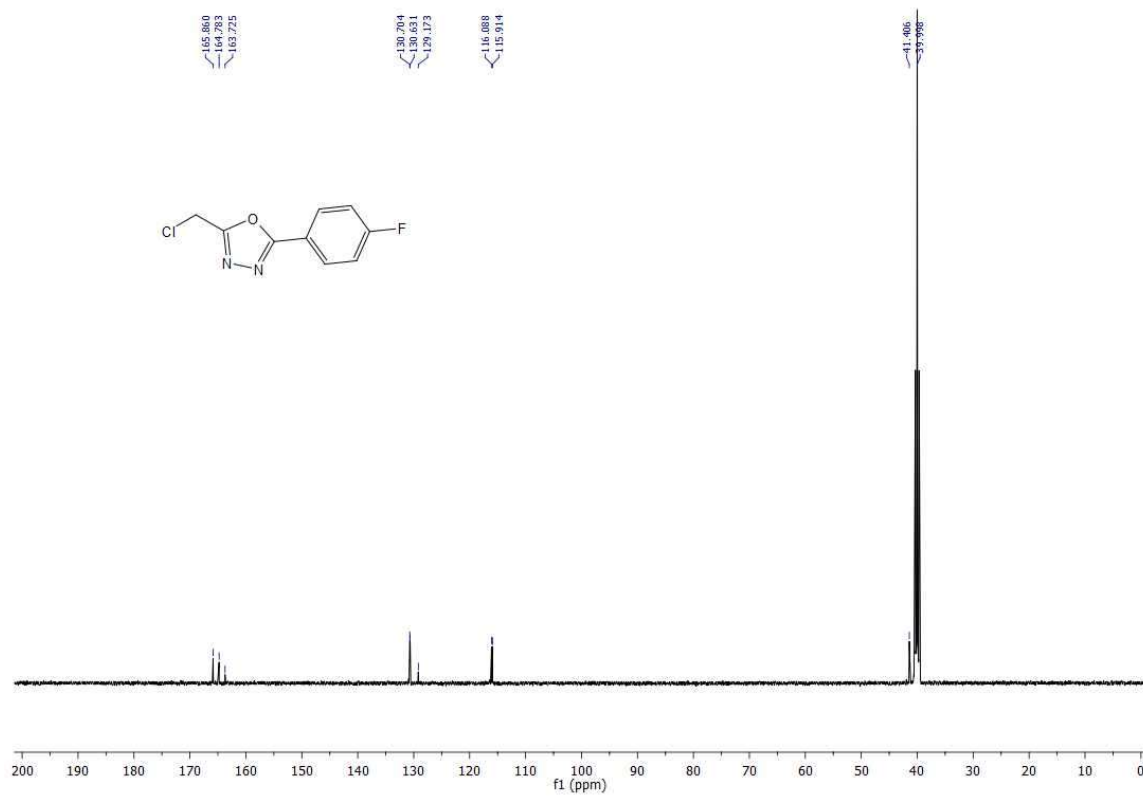


Figure 8. 37. ¹³C NMR spectra of target compound 8l.

¹H NMR, ¹³C NMR, HRMS, and HPLC spectra of the representative final compounds

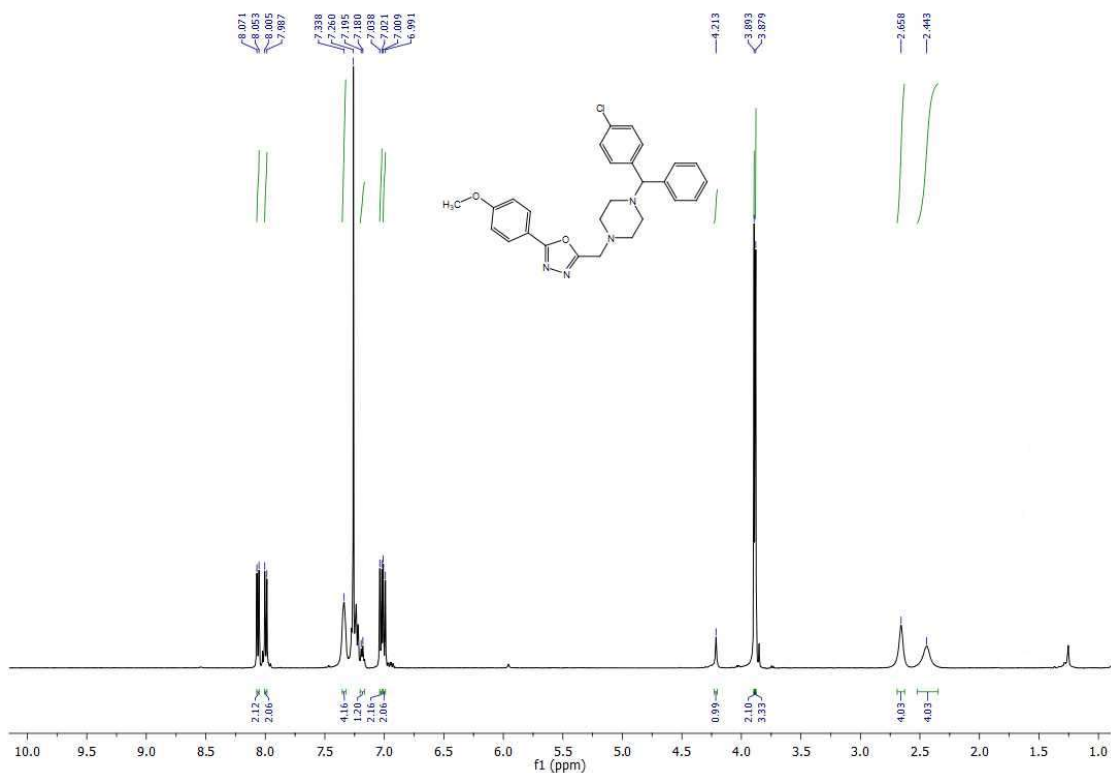


Figure 8. 38. ¹H NMR spectra of target compound 9b.

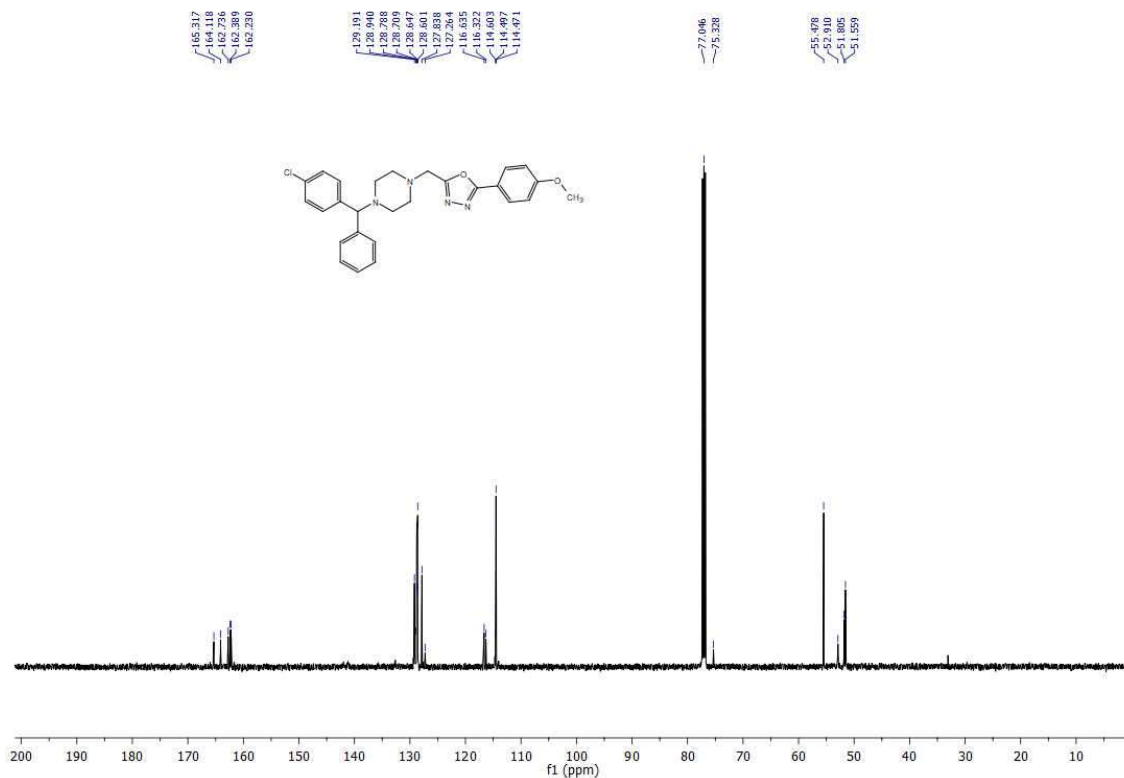


Figure 8. 39. ¹³C NMR spectra of target compound 9b.

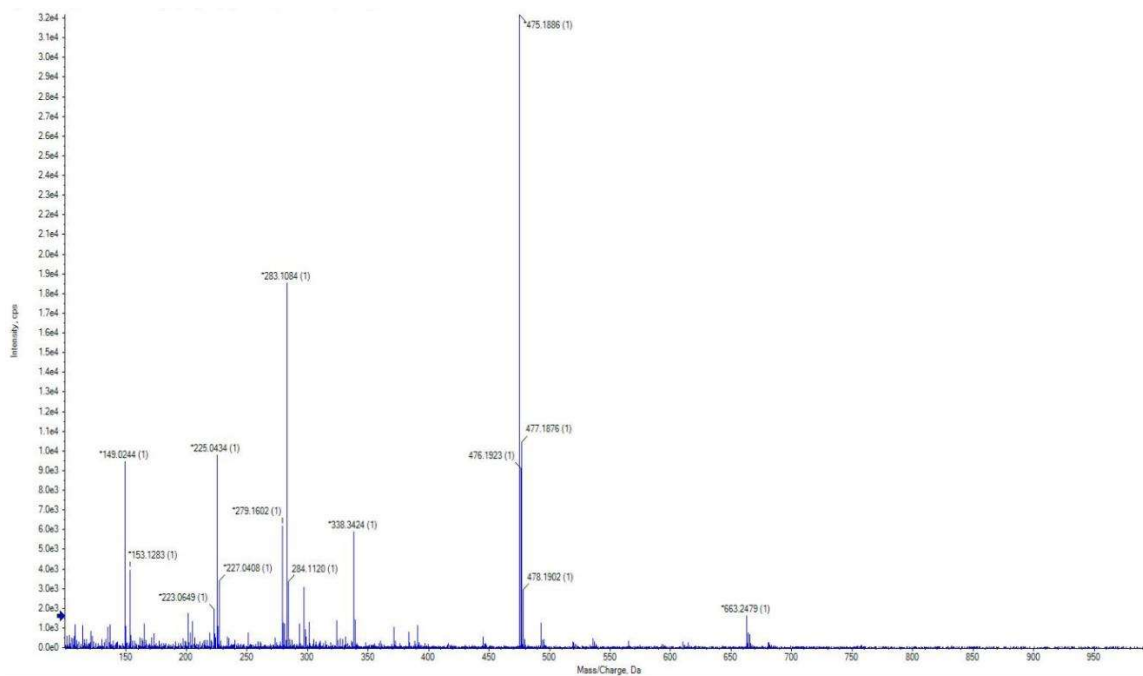


Figure 8. 40. HRMS [M + H]⁺spectra of target compound 9b.

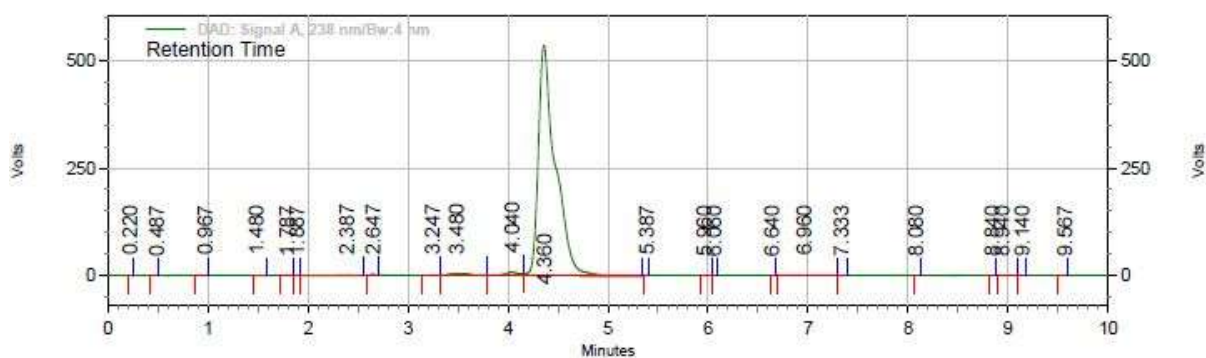


Figure 8. 41. HPLC chromatogram of target compound 9b

Percentage purity of compound 9b:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5μm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 μl
- ✓ Detection range: λ_{max} = 254

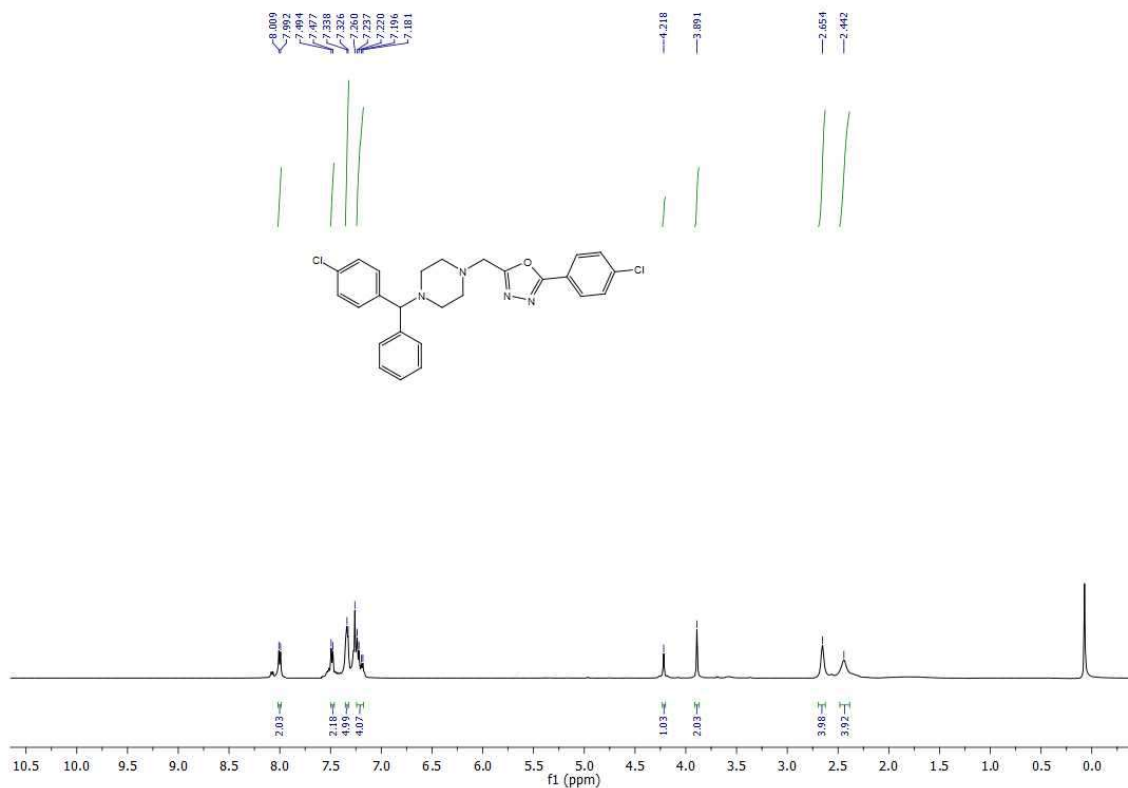


Figure 8. 42. ¹H spectra of target compound 9d

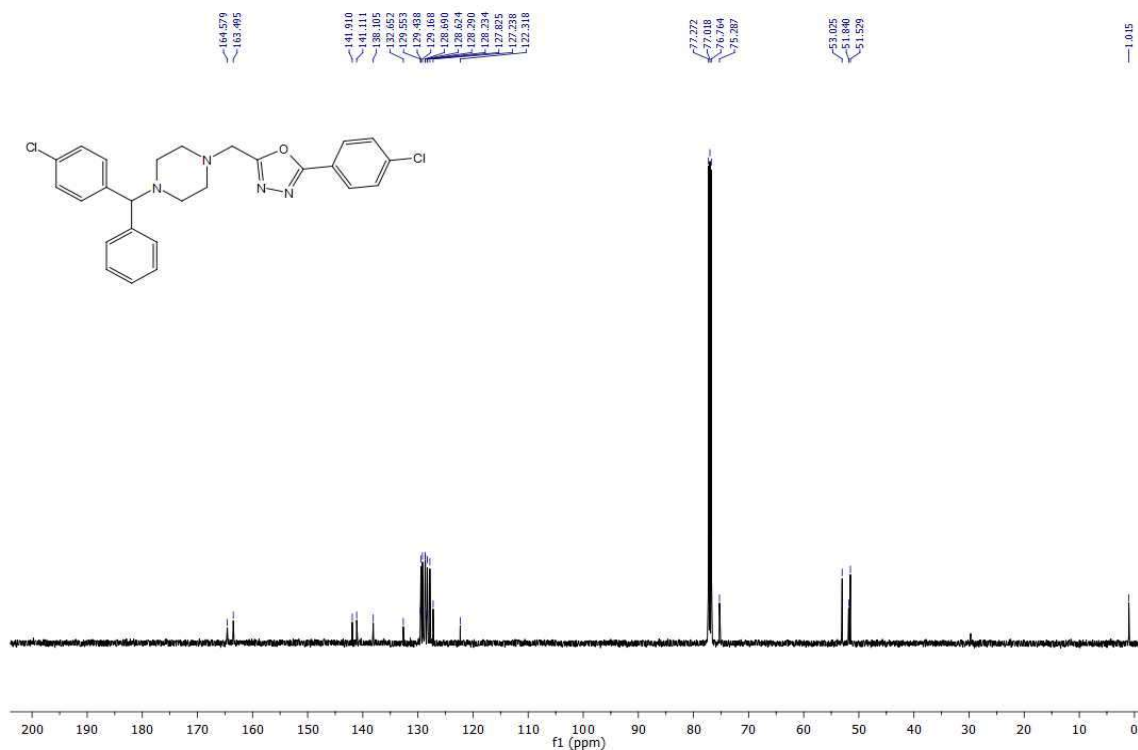


Figure 8. 43. ¹³C spectra of target compound 9d

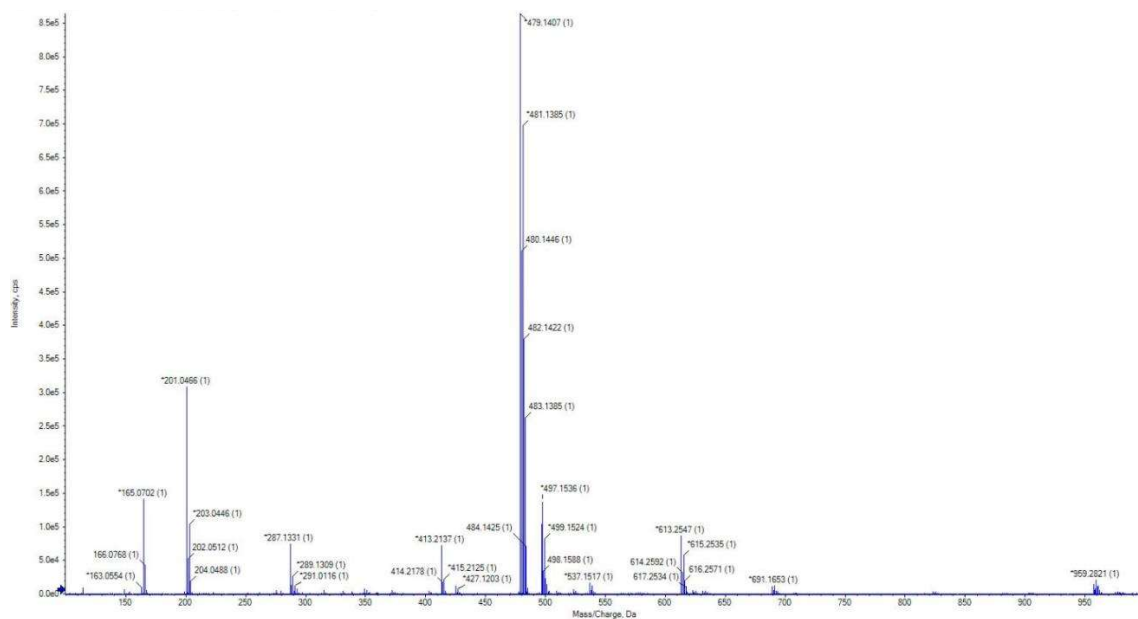


Figure 8. 44. HRMS $[M + H]^+$ spectra of target compound 9d.

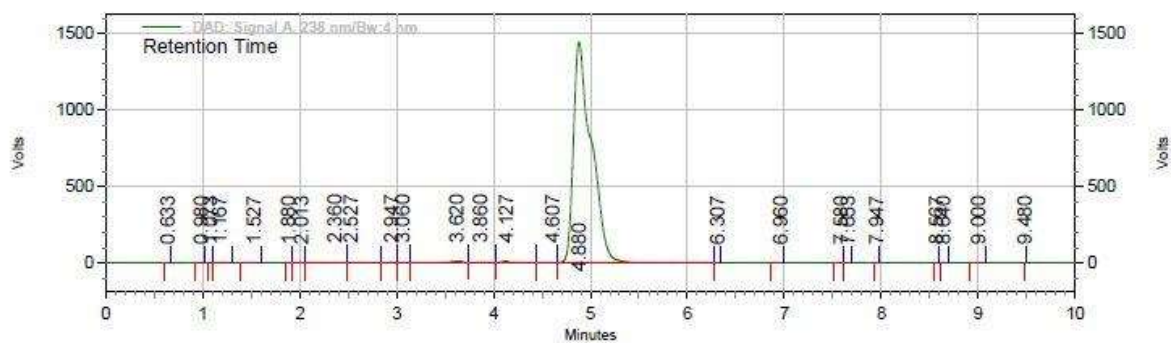


Figure 8. 45. HPLC chromatogram of target compound 9d

Percentage purity of compound 9d:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C_{18} 250 \times 4.6mm, 5 μ m Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 μ l
- ✓ Detection range: $\lambda_{max} = 254$

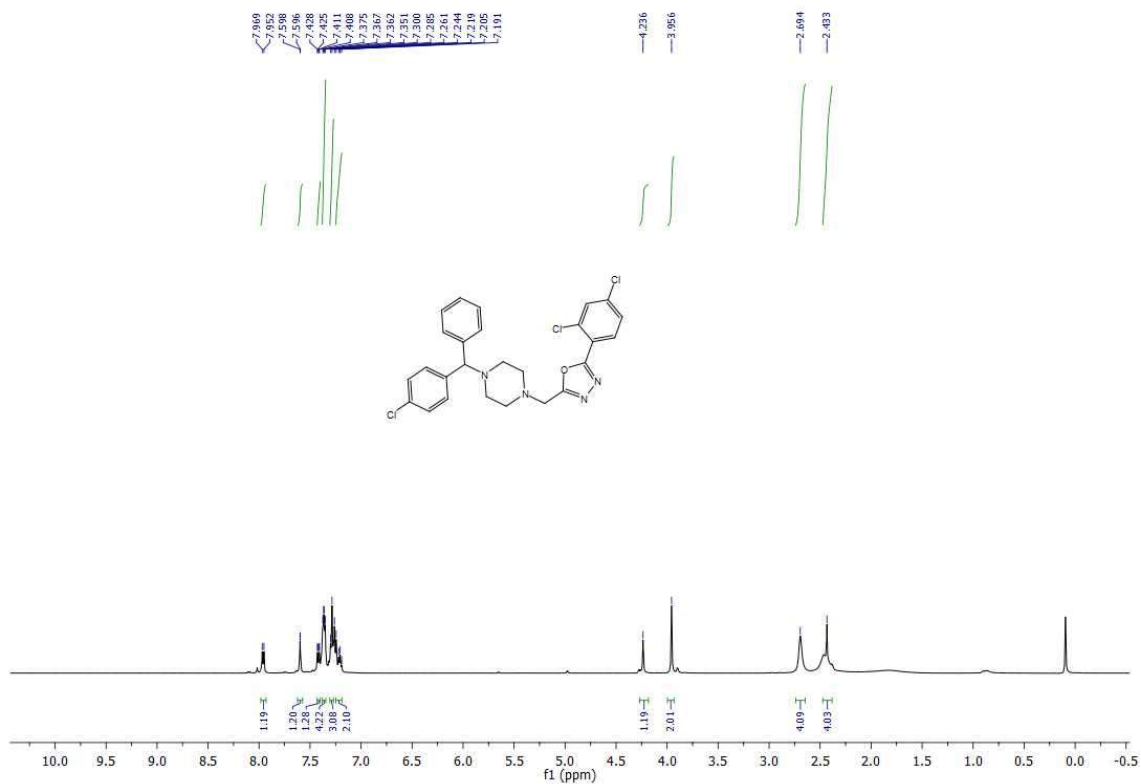


Figure 8. 46. ¹H spectra of target compound 9e

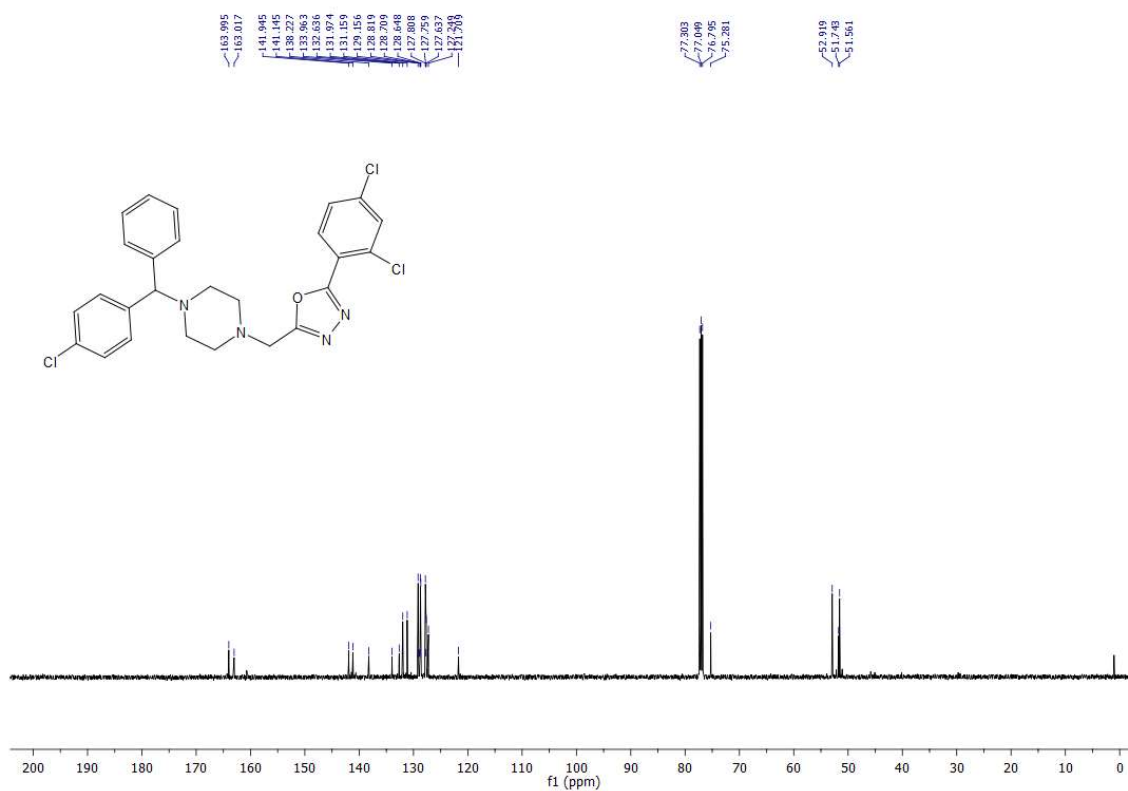


Figure 8. 47. ¹³C spectra of target compound 9e

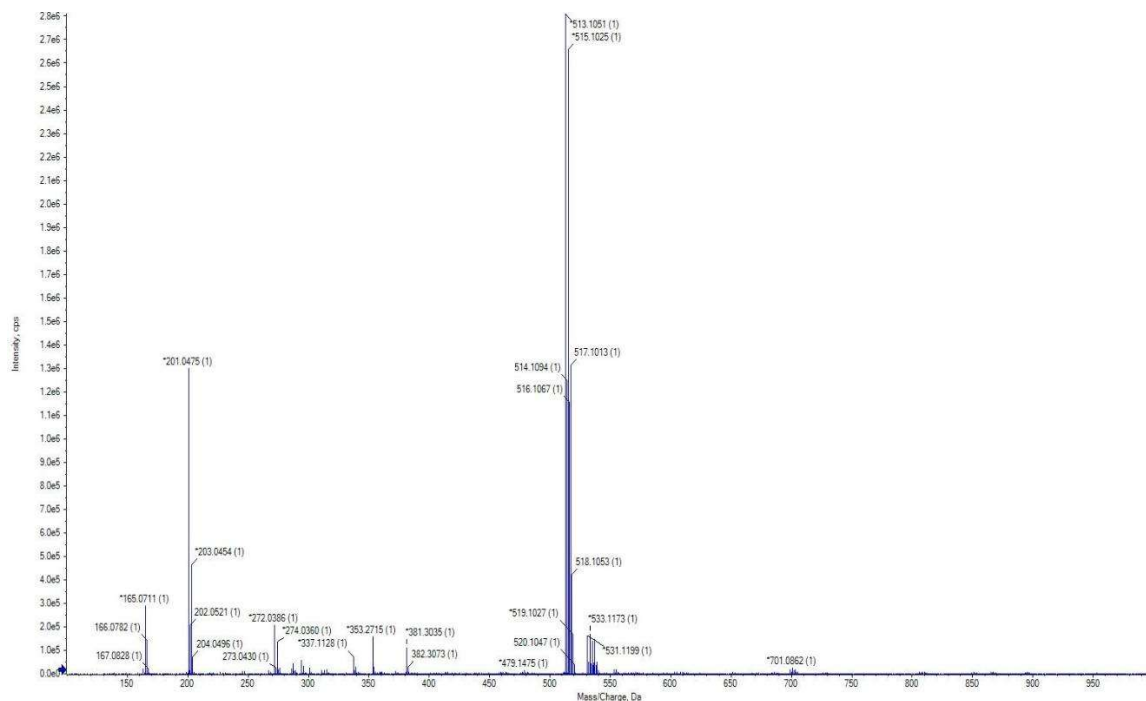


Figure 8. 48. HRMS spectra of target compound 9e

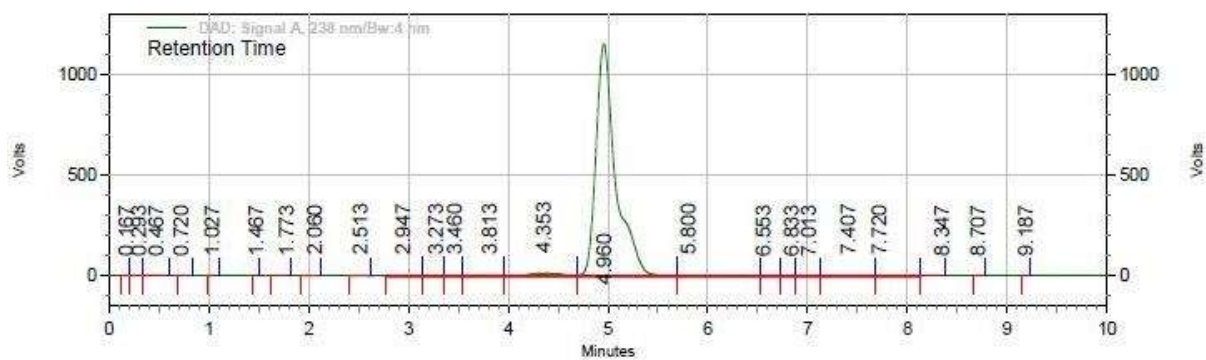


Figure 8. 49. HPLC chromatogram of target compound 9e

Percentage purity of compound 9e:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5µm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 µl
- ✓ Detection range: λ_{max} = 254

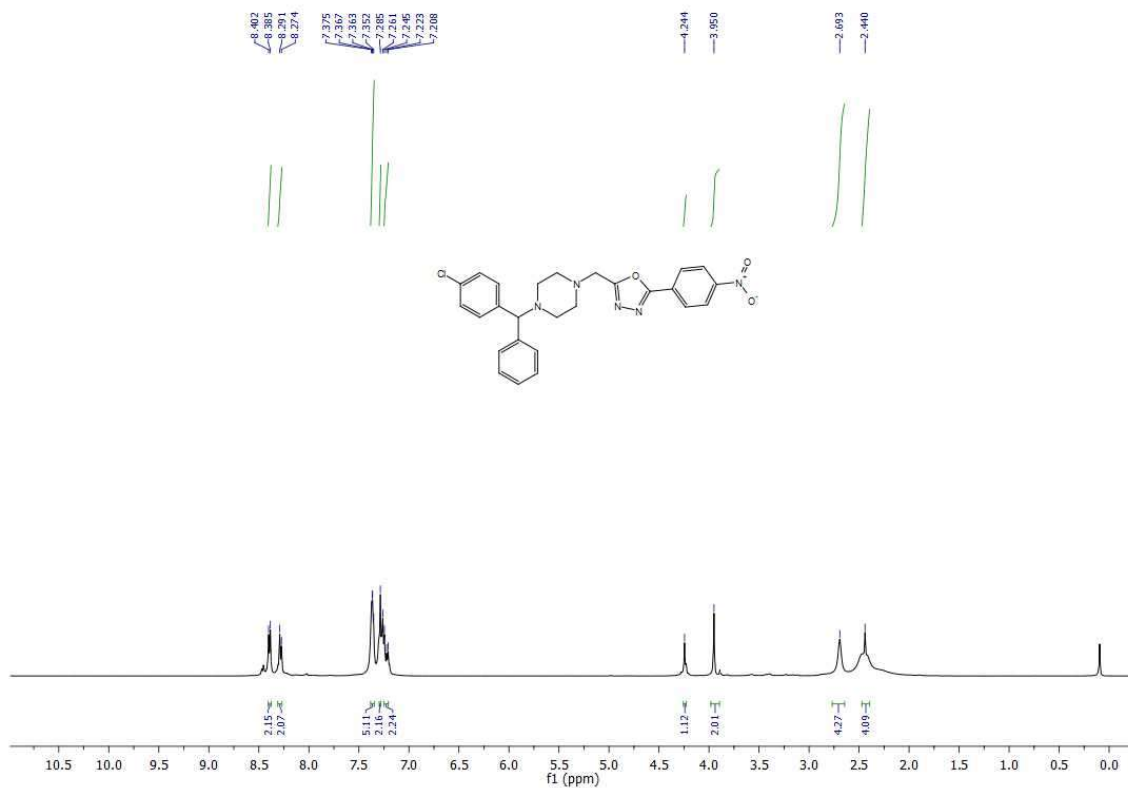


Figure 8. 50. ¹H spectra of target compound 9g

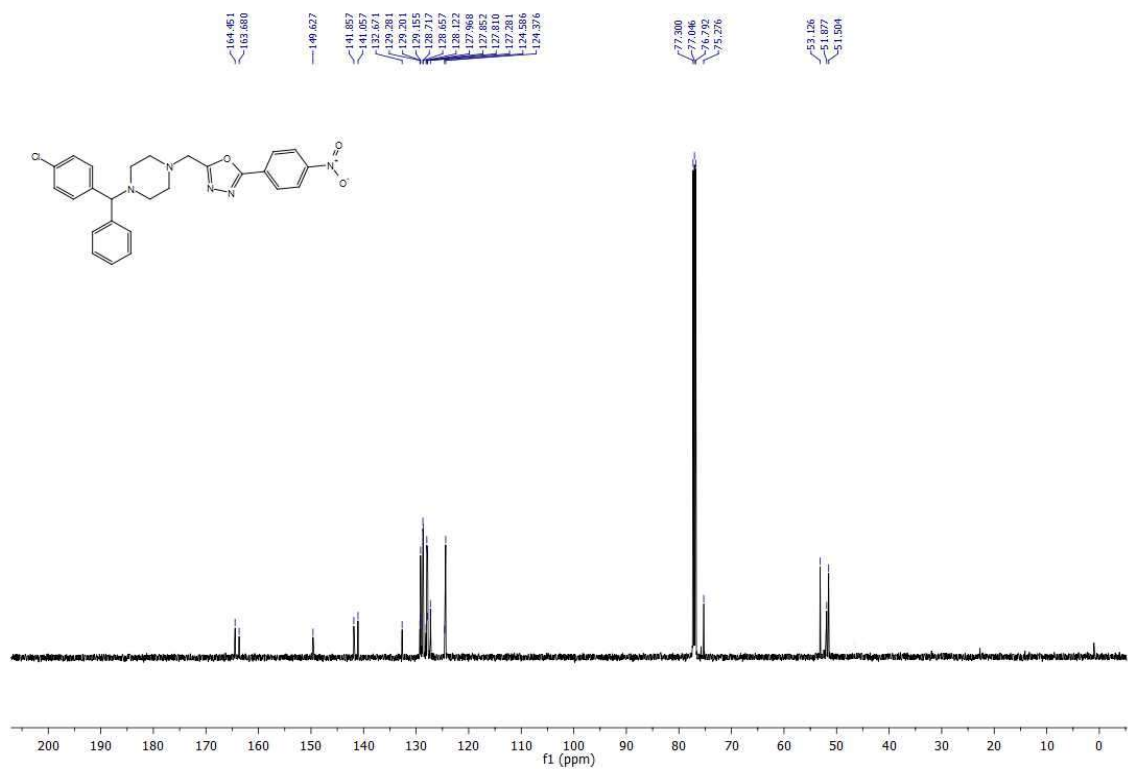


Figure 8. 51. ¹³C spectra of target compound 9g

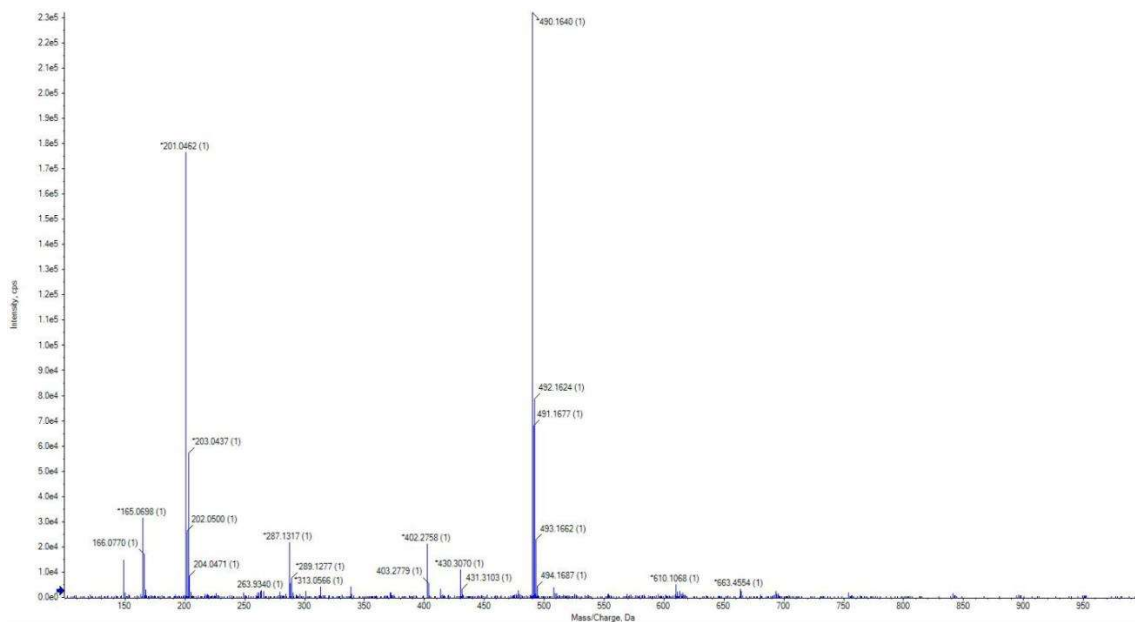


Figure 8. 52. HRMS spectra of target compound 9g

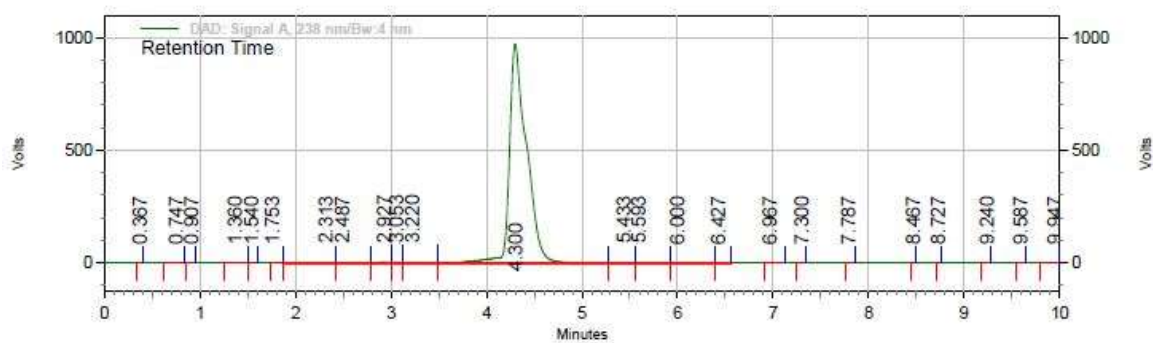


Figure 8. 53. HPLC chromatogram of target compound 9g

Percentage purity of compound 9g:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5μm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 μl
- ✓ Detection range: λ_{max} = 254

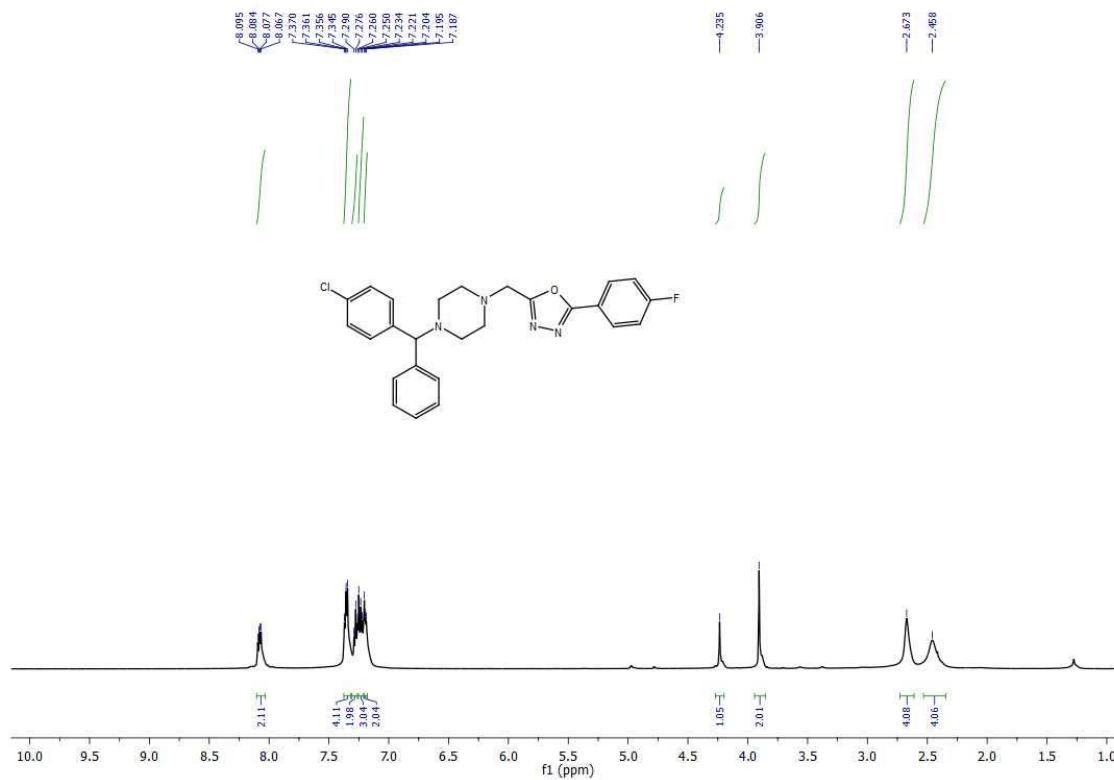


Figure 8. 54. ¹H spectra of compound 9l

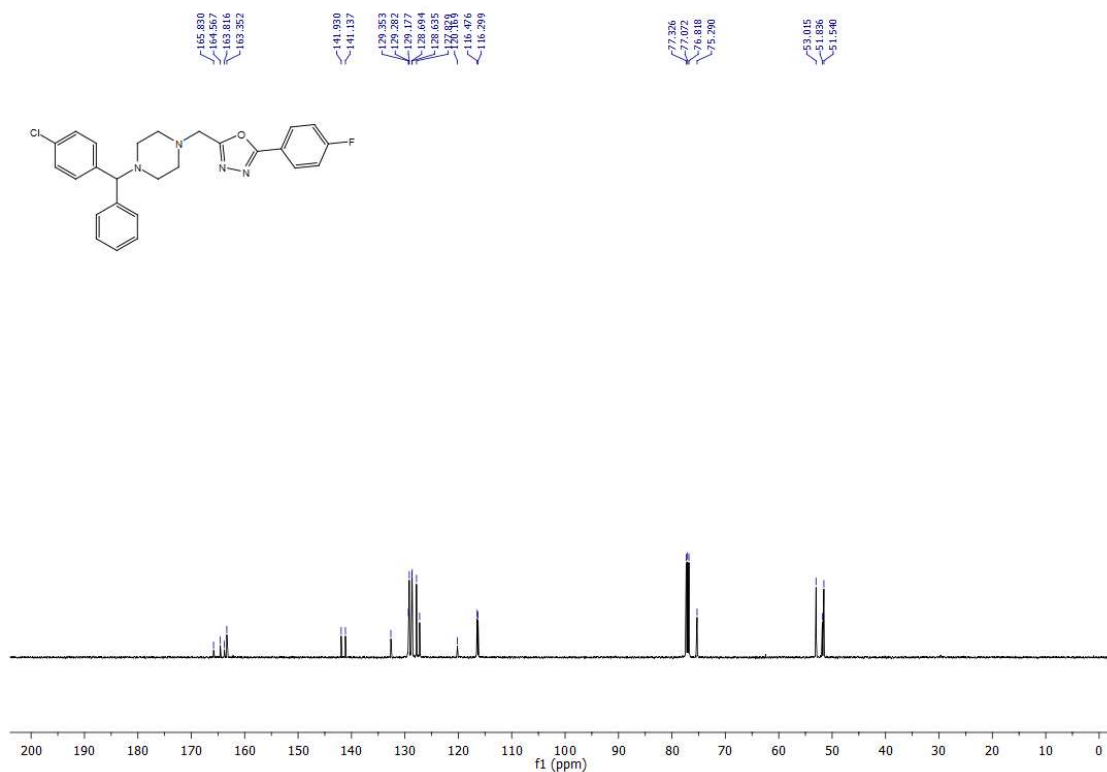


Figure 8. 55. ¹³C spectra of compound 9l

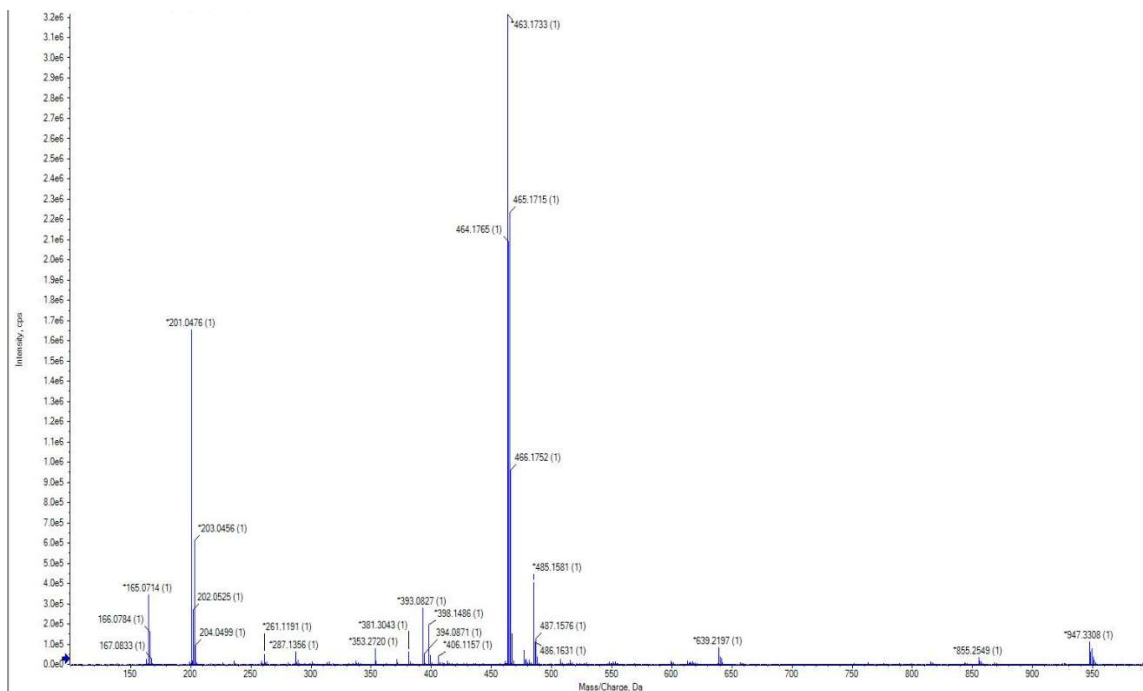


Figure 8. 56. HRMS spectra of target compound 9I

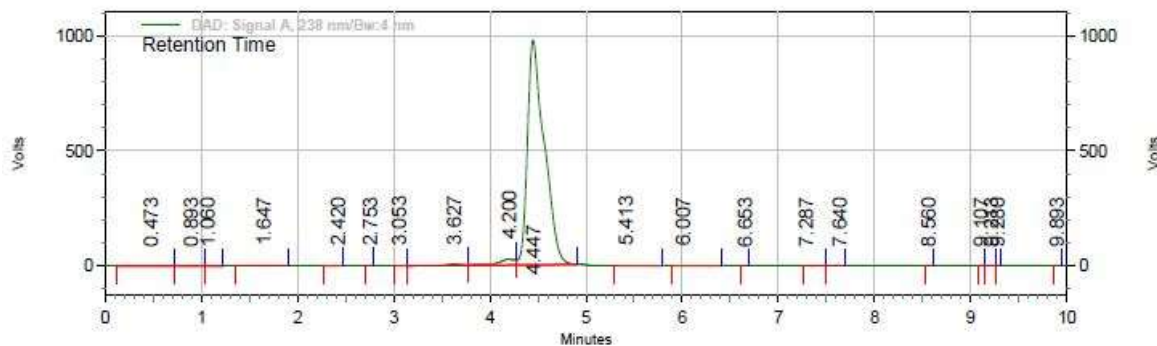


Figure 8. 57. HPLC chromatogram of target compound 9I

Percentage purity of compound 9I:

- ✓ Determined using the Agilent 1200 Infinity high-performance liquid chromatography (HPLC) system, USA.
- ✓ Column: Quasar C₁₈ 250 × 4.6mm, 5μm Cat. No. N9308801
- ✓ Mobile phase: Methanol (90): Water (10)
- ✓ Flow rate: 1ml/min.
- ✓ Sample volume: 20 μl
- ✓ Detection range: λ_{max} = 254

¹H NMR spectra of each of the corresponding final compounds

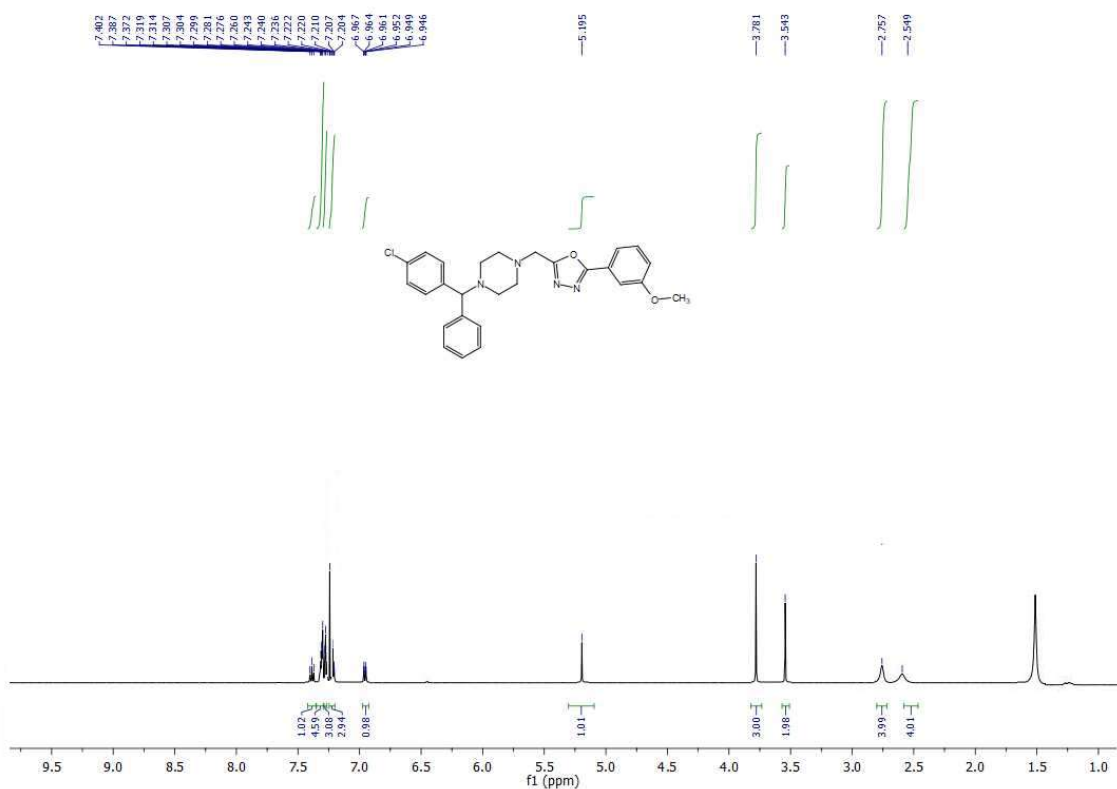


Figure 8. 58. ¹H spectra of compound 9a

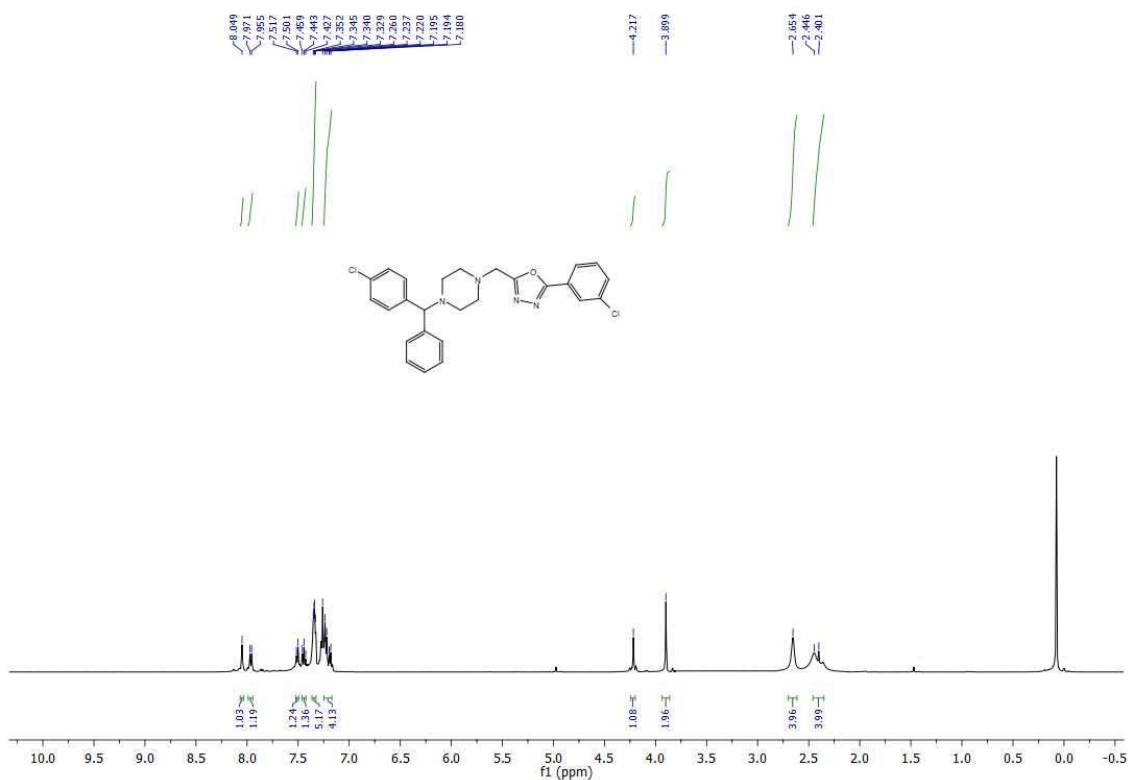


Figure 8. 59. ¹H spectra of compound 9c

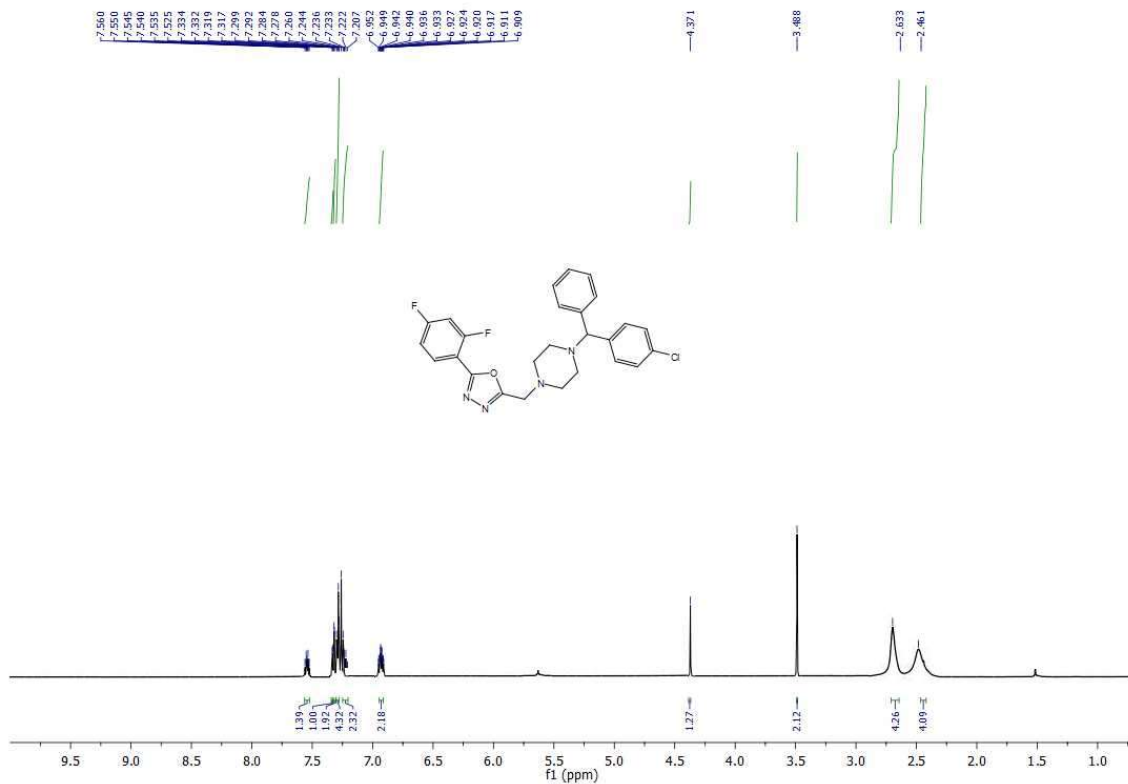


Figure 8. 62. ¹H spectra of compound 9i

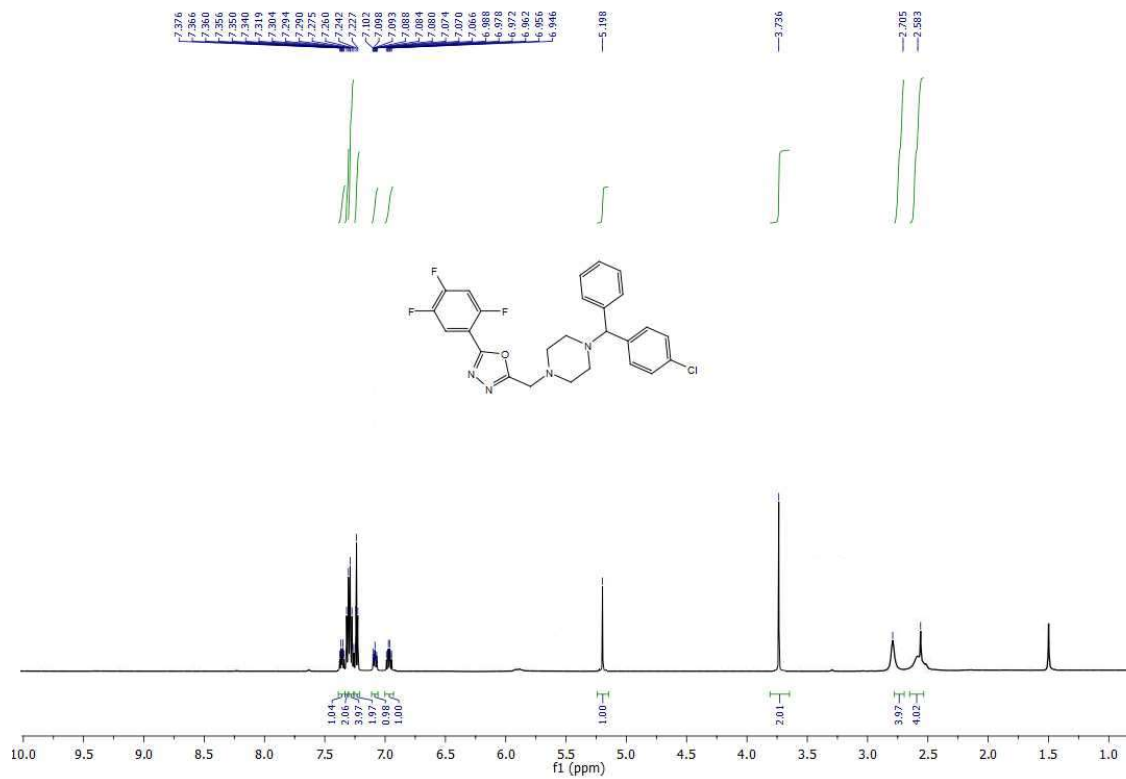


Figure 8. 63. ¹H spectra of compound 9j

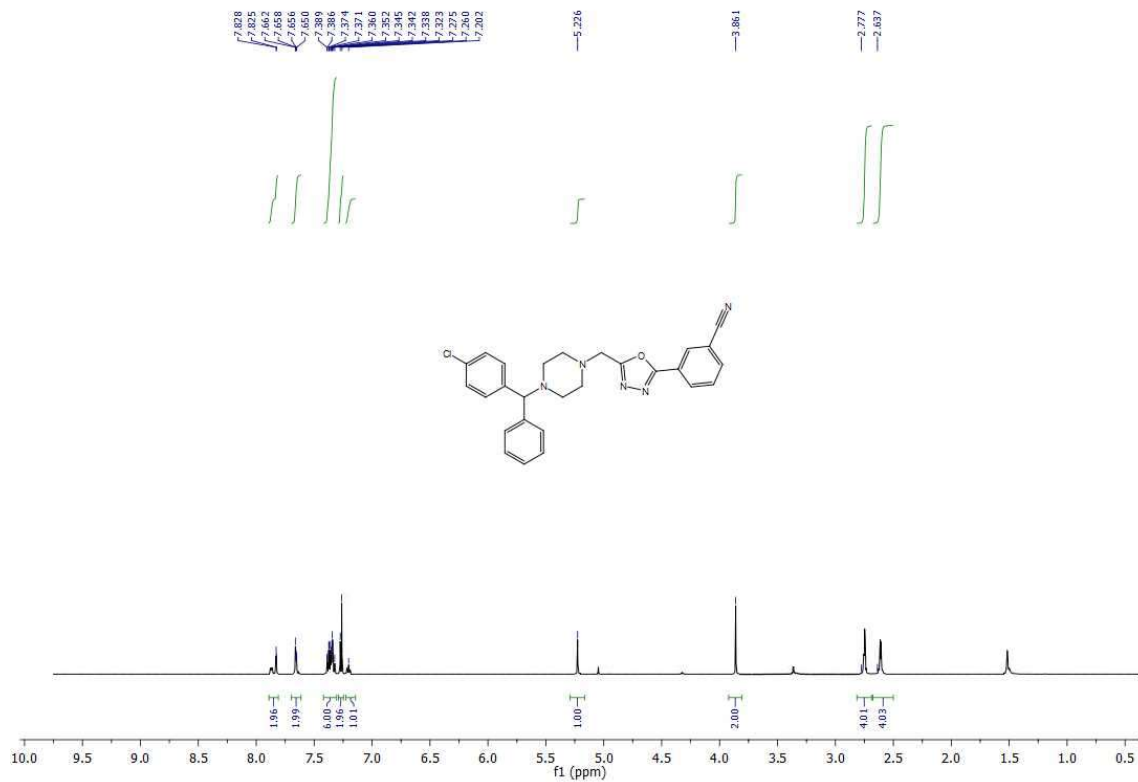


Figure 8. 66. ¹H spectra of compound 9n

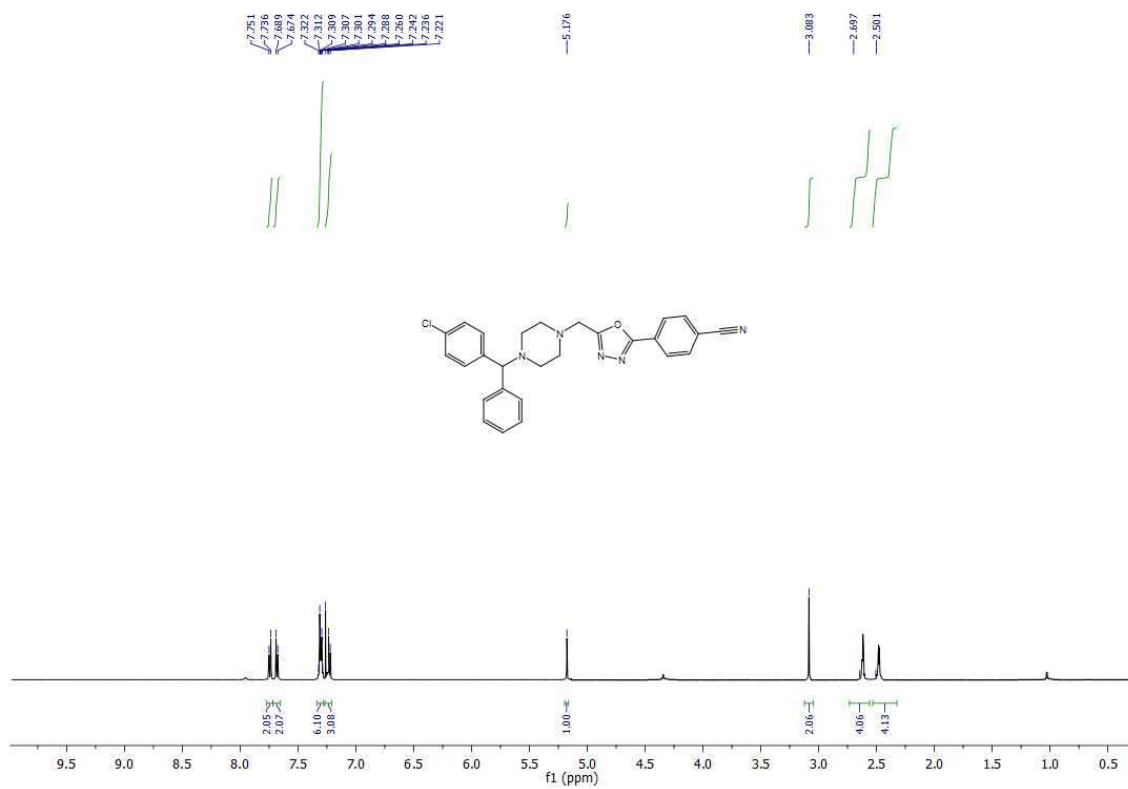


Figure 8. 67. ¹H spectra of compound 9o

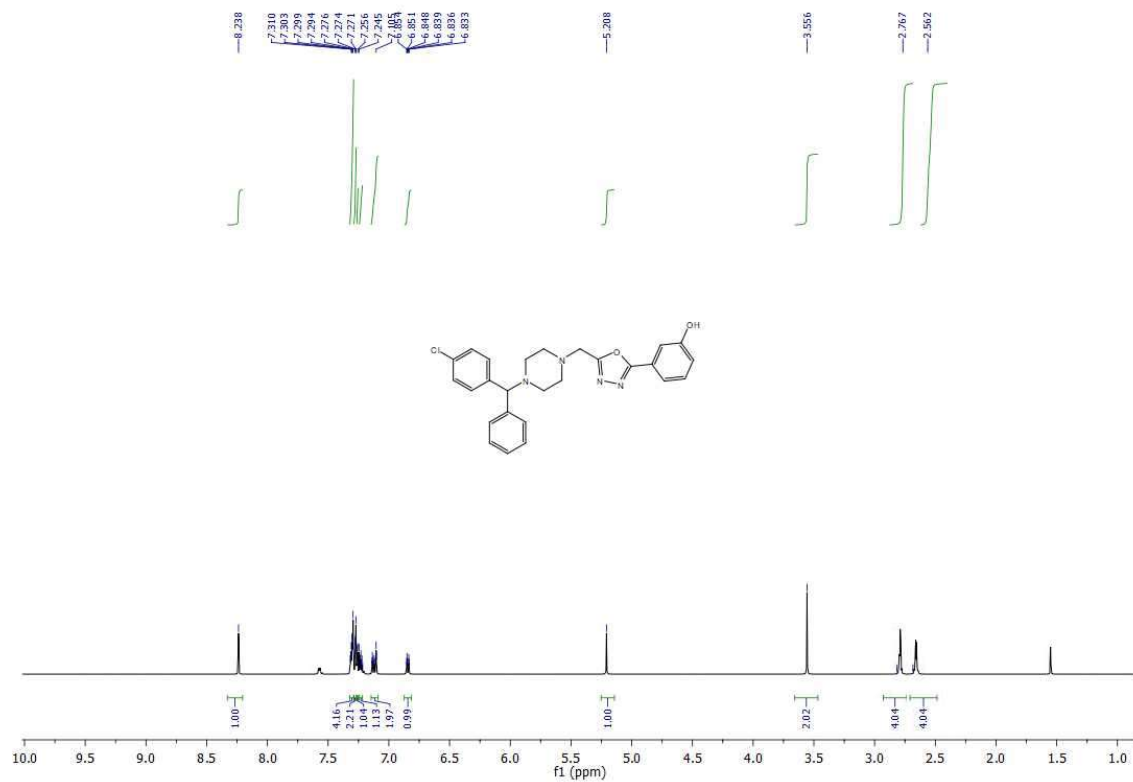


Figure 8. 69. ¹H spectra of compound 9q

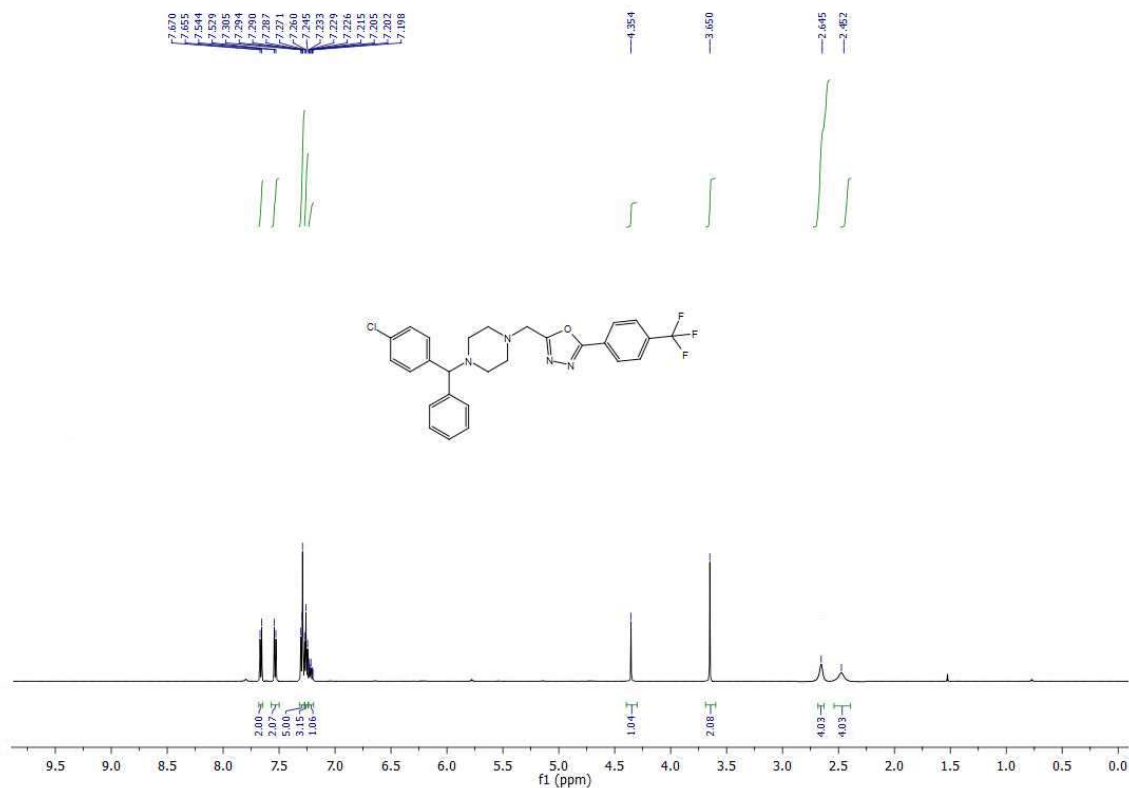


Figure 8. 70. ¹H spectra of compound 9s

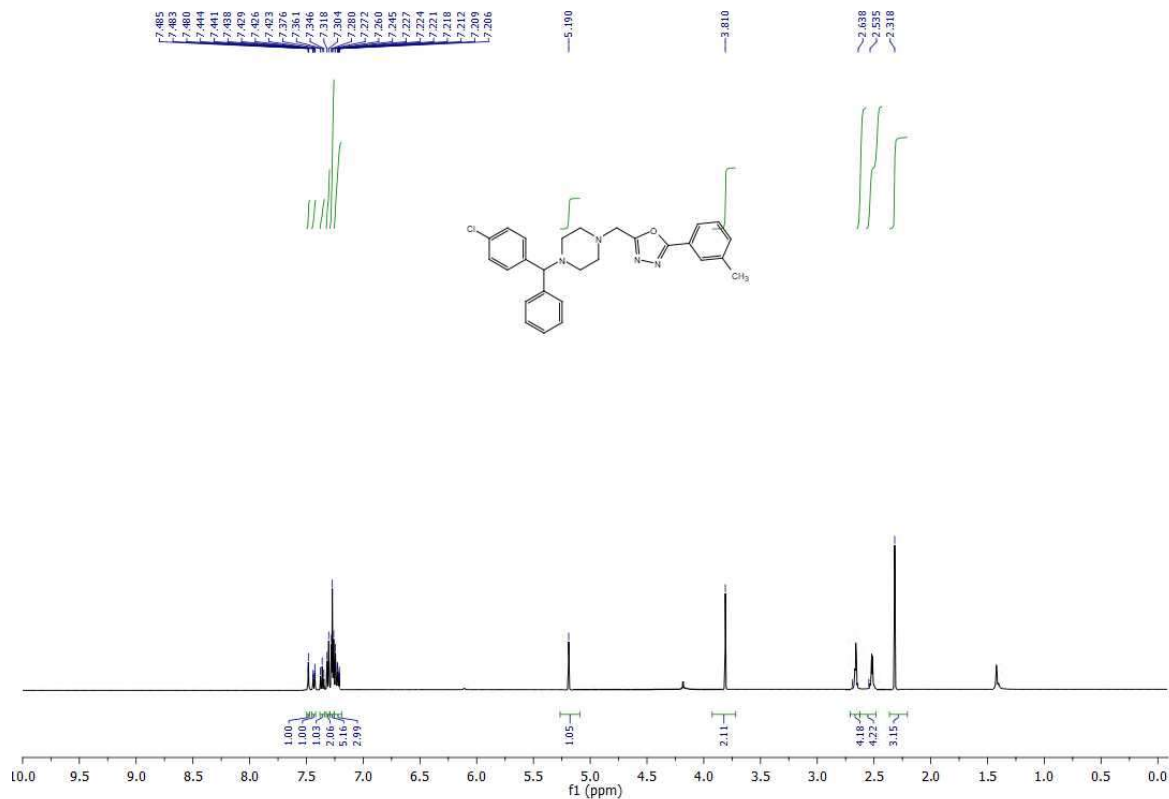


Figure 8. 71. ¹H spectra of compound 9t

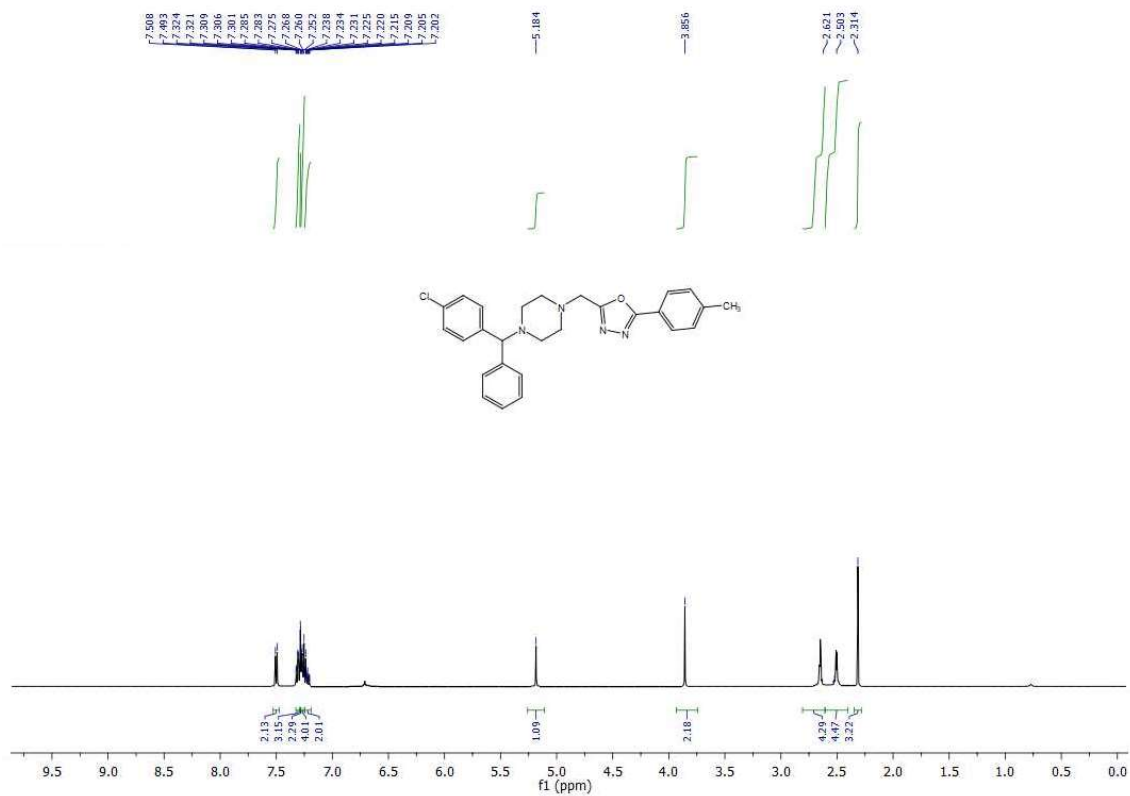


Figure 8. 72. ¹H spectra of compound 9u

**Lists of Research Papers
&
Presentation**

PUBLICATIONS FROM Ph.D. DISSERTATION

- **Saraf, P.**, Bhardwaj, B., Verma, A., Siddiqui, M. A., Verma, H., Kumar, P., ... & Shrivastava, S. K. (2024). Design, synthesis, and evaluation of benzhydrylpiperazine-based novel dual COX-2/5-LOX inhibitors with anti-inflammatory and anti-cancer activity. *RSC Medicinal Chemistry*. (IF: 4.1)
- **Saraf, P.**, Tripathi, P. N., Tripathi, M. K., Tripathi, A., Verma, H., Waiker, D. K., & Shrivastava, S. K. (2022). Novel 5, 6-diphenyl-1, 2, 4-triazine-3-thiol derivatives as dual COX-2/5-LOX inhibitors devoid of cardiotoxicity. *Bioorganic Chemistry*, 129, 106147. (IF: 5.1)

OTHER PUBLICATIONS

- Tripathi, N., **Saraf, P.**, Bhardwaj, N., Shrivastava, S. K., & Jain, S. K. (2024). Identifying inflammation-related targets of natural lactones using network pharmacology, molecular modeling and in vitro approaches. *Journal of Biomolecular Structure and Dynamics*, 1-16. (IF: 5.2)
- Verma, A., Waiker, D. K., Singh, N., Roy, A., Singh, N., **Saraf, P.**, ... & Shrivastava, S. K. (2024). Design, Synthesis, and Biological Investigation of Quinazoline Derivatives as Multitargeting Therapeutics in Alzheimer's Disease Therapy. *ACS Chemical Neuroscience*, 15(4), 745-771. (IF: 4.1)
- Singh, A., Verma, A., Bhardwaj, B., **Saraf, P.**, Kumar, H., Jain, N., ... & Shrivastava, S. K. (2024). Structure-Guided Design, Synthesis, and Biological Evaluation of Peripheral Anionic Site Selective and Brain Permeable Novel Oxadiazole-Piperazine Conjugates against Alzheimer's Disease with Antioxidant Potential. *ACS omega*, 9(16), 18169-18182. (IF: 4.1)

- Verma, A., Waiker, D. K., Singh, N., Singh, A., Saraf, P., Bhardwaj, B., ... & Shrivastava, S. K. (2024). Lead optimization based design, synthesis, and pharmacological evaluation of quinazoline derivatives as multi-targeting agents for Alzheimer's disease treatment. *European Journal of Medicinal Chemistry*, 271, 116450. (IF:6.7)
- Waiker, D. K., Verma, A., Saraf, P., TA, G., Krishnamurthy, S., Chaurasia, R. N., & Shrivastava, S. K. (2023). Development and Evaluation of Some Molecular Hybrids of N-(1-Benzylpiperidin-4-yl)-2-((5-phenyl-1,3,4-oxadiazol-2-yl) thio) as Multifunctional Agents to Combat Alzheimer's Disease. *ACS omega*, 8(10), 9394-9414. (IF: 4.1)
- Kiran, P. V. R., Waiker, D. K., Verma, A., Saraf, P., Bhardwaj, B., Kumar, H., & Shrivastava, S. K. (2023). Design and development of benzyl piperazine linked 5-phenyl-1, 2, 4-triazole-3-thione conjugates as potential agents to combat Alzheimer's disease. *Bioorganic Chemistry*, 139, 106749. (IF: 5.1)
- Verma, A., Waiker, D. K., Bhardwaj, B., Saraf, P., & Shrivastava, S. K. (2022). The molecular mechanism, targets, and novel molecules in the treatment of Alzheimer's disease. *Bioorganic Chemistry*, 119, 105562. (IF: 5.1)
- Shrivastava, S. K., Sinha, O., Kumar, M., Waiker, D. K., Verma, A., Tripathi, P. N., ... & Saraf, P. (2022). Synthesis, characterization, and biological evaluation of some novel γ -aminobutyric acid aminotransferase (GABA-AT) inhibitors. *Medicinal Chemistry Research*, 31(9), 1594-1610. (IF: 2.6)
- Shrivastava, S. K., Nivrutti, A. A., Bhardwaj, B., Waiker, D. K., Verma, A., Tripathi, P. N., & Saraf, P. (2022). Drug reposition-based design, synthesis, and biological evaluation of dual inhibitors of acetylcholinesterase and β -Secretase for treatment of Alzheimer's disease. *Journal of Molecular Structure*, 1262, 132979. (IF: 3.8)

- Tripathi, A., Choubey, P. K., Sharma, P., Seth, A., **Saraf, P.**, & Shrivastava, S. K. (2020). Design, synthesis, and biological evaluation of ferulic acid based 1, 3, 4-oxadiazole hybrids as multifunctional therapeutics for the treatment of Alzheimer's disease. *Bioorganic chemistry*, 95, 103506. (IF: 5.1)

CONFERENCES

- Presented poster in the international conference **GP2A 2024** (Group for the Promotion of Pharmaceutical Chemistry in Academia) at the University of Coimbra, Portugal, Europe.
- Presented poster in international conference **SKICON, 2023**, held at Shivajirao Kadam Institute of Pharmaceutical Education & Research, Indore, M.P.
- Presented poster in the institute day held at IIT (BHU), Varanasi, during February 16-18, 2018.
- Presented poster in the international conference **Emerging Trends in Drug Discovery and Development (ETDDD)**, 2018 held at IIT (BHU).
- Attended **Spirit 17th- National Seminar on Programmatic Approach toward Pharmaceutical Transcendence**, 2017 held at IIT (BHU), Varanasi.
- Participated in Diamond Jubilee Celebrations & **International Conference** on 'Industry Interaction: **Global Pharma Prospects** held at Dr. HS Gour University, Sagar in 2016.
- Volunteered **National seminar** on Emerging frontier & challenges in nanotechnology & Drug Design held at Dr. HS Gour University (**EFCNDD-2015**).
- Presented poster in All India Seminar on **Biotechnology and Human Health**, 2014.
- Presented Poster on the **Medicinal value of Isabgol plant** in **National seminar, 2013** held at SIPS, Sagar.