

GREEN APPROACH: A SIMPLE ONE-POT SYNTHESIS OF PYRANOPYRAZOLES SCAFFOLD

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EXPERIMENTAL

Material and Methods

All reagents and chemicals were of analytical grade and used without further purification. The progress of the reaction was monitored on TLC. All the products were confirmed by comparing their melting points, IR, and ¹H NMR data with literature data.

General procedure for the synthesis of substituted pyranopyrazole (5a-j)

To a round bottom flask substituted aromatic aldehyde (1mmol), malononitrile (1mmol), ethyl acetoacetate (1mmol), and hydrazine hydrate (1 mmol) in toluene under reflux conditions for the time as specified in **Figure 3**. The advancement of the reaction was tracked through TLC (10% ethyl acetate: *n*-hexane). The resulting product was combined with water and ethyl acetate (1:4). The combined solvent extracts underwent vacuum concentration. Subsequently, the compounds underwent recrystallization in ethanol to yield the purified product (**5a-j**).

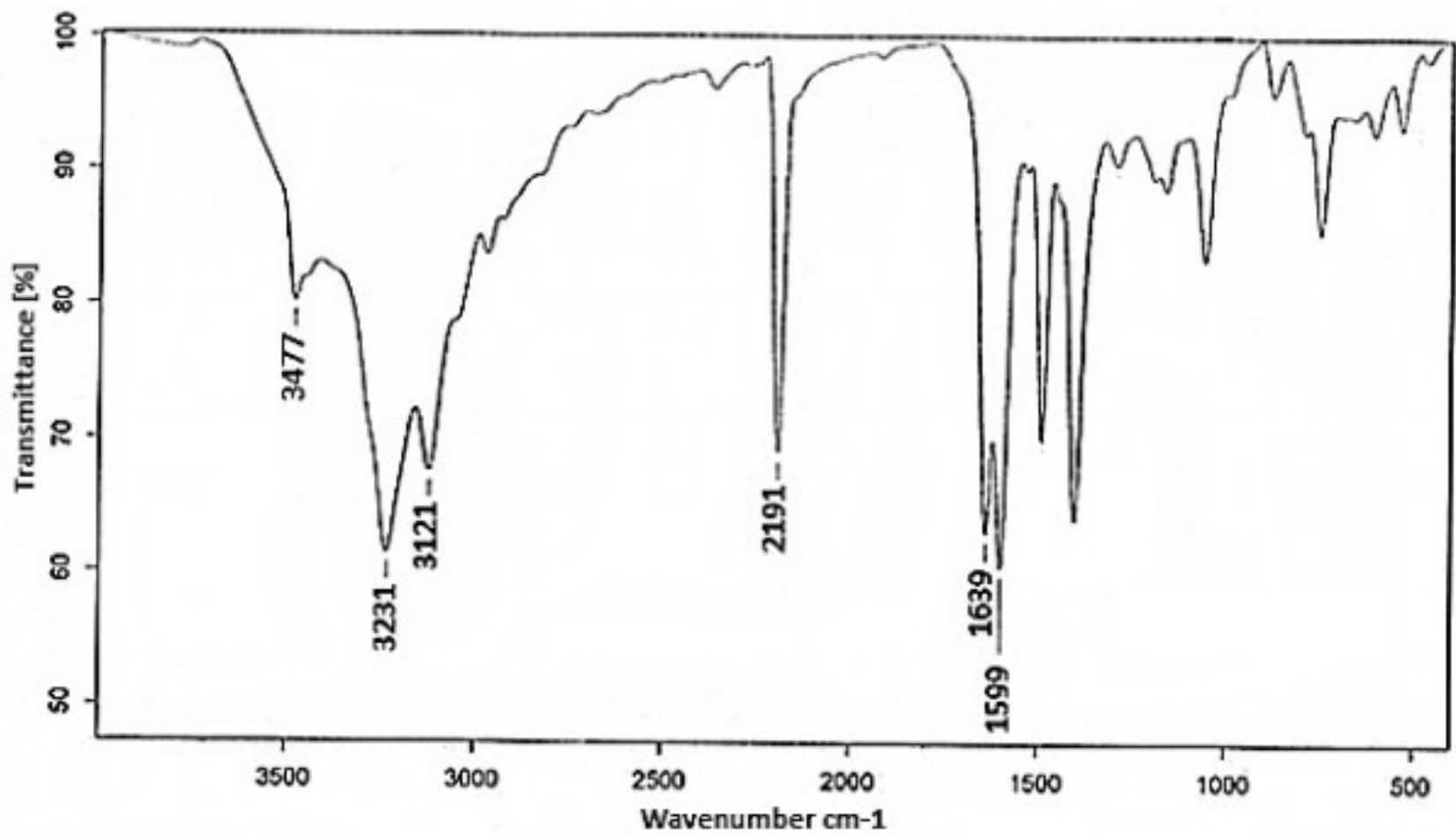


Figure 1. IR spectrum of compound 5a

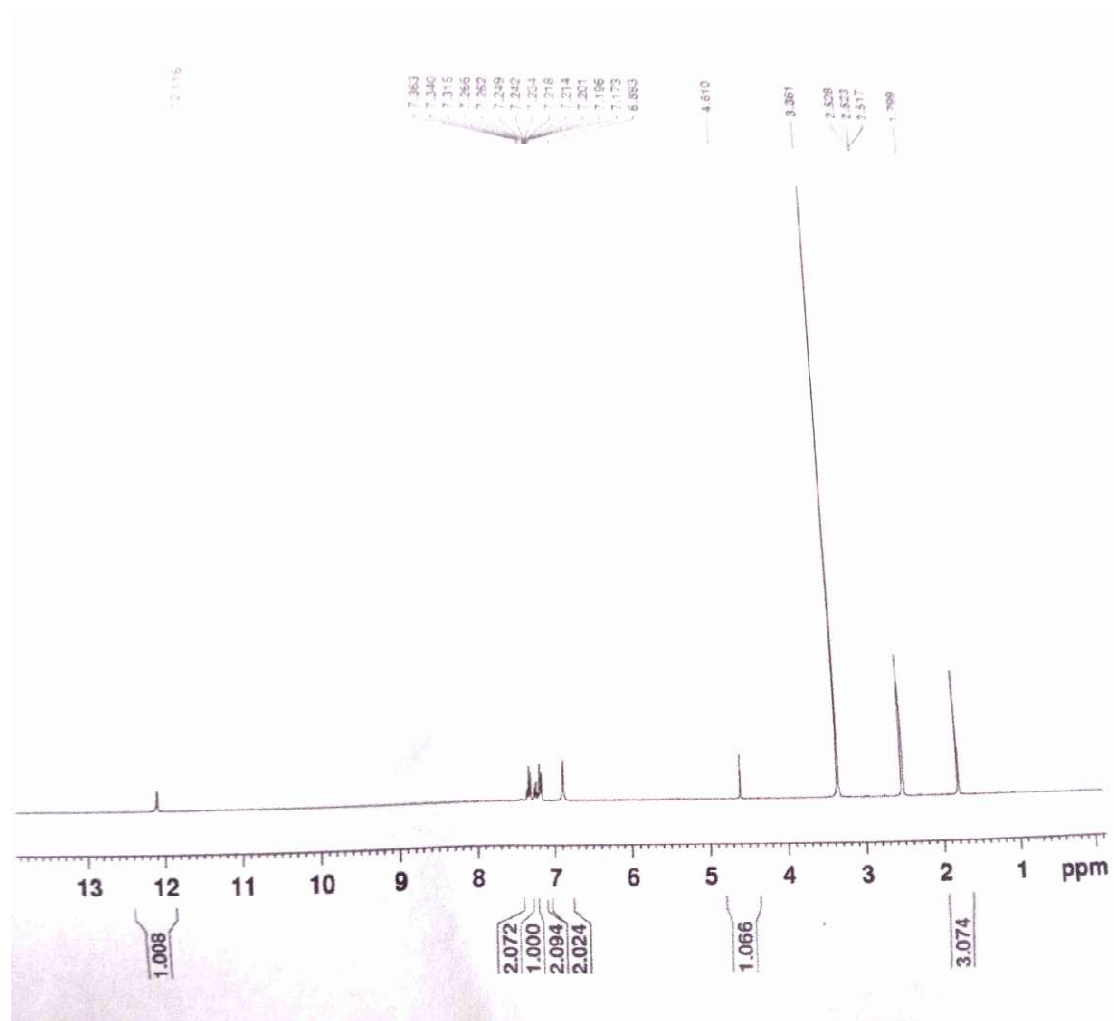


Figure 2. ^1H NMR spectrum of compound 5a

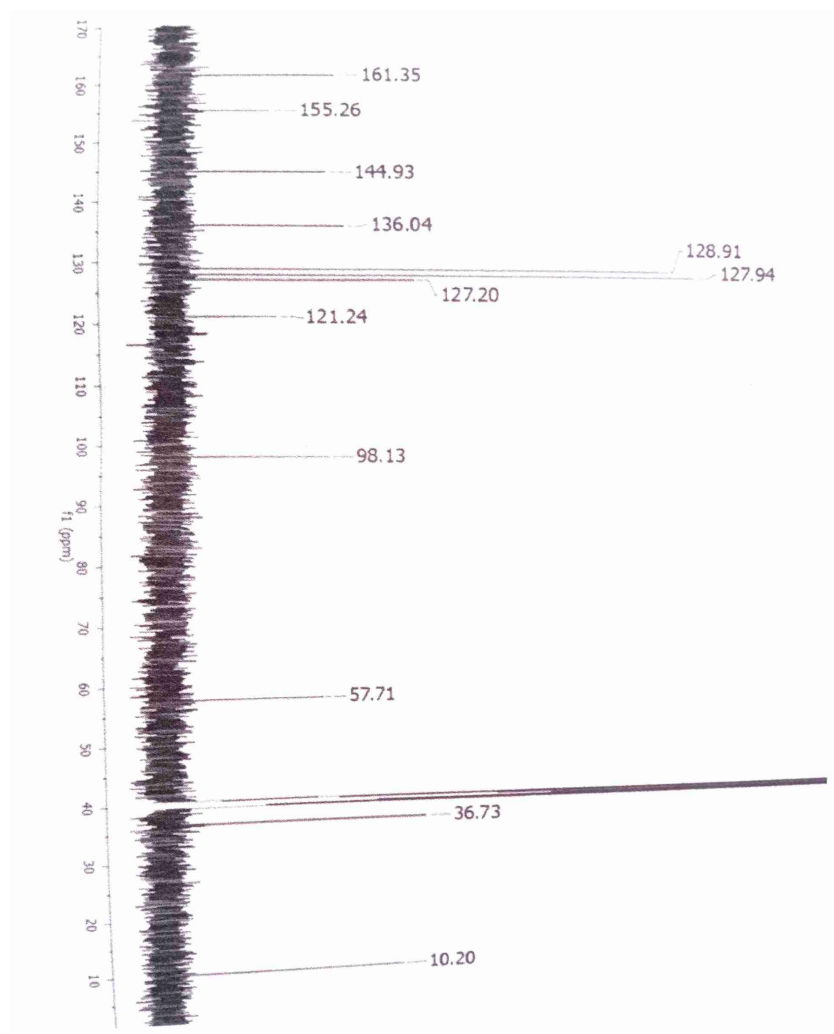


Figure 3. ^{13}C NMR spectrum of compound 5a

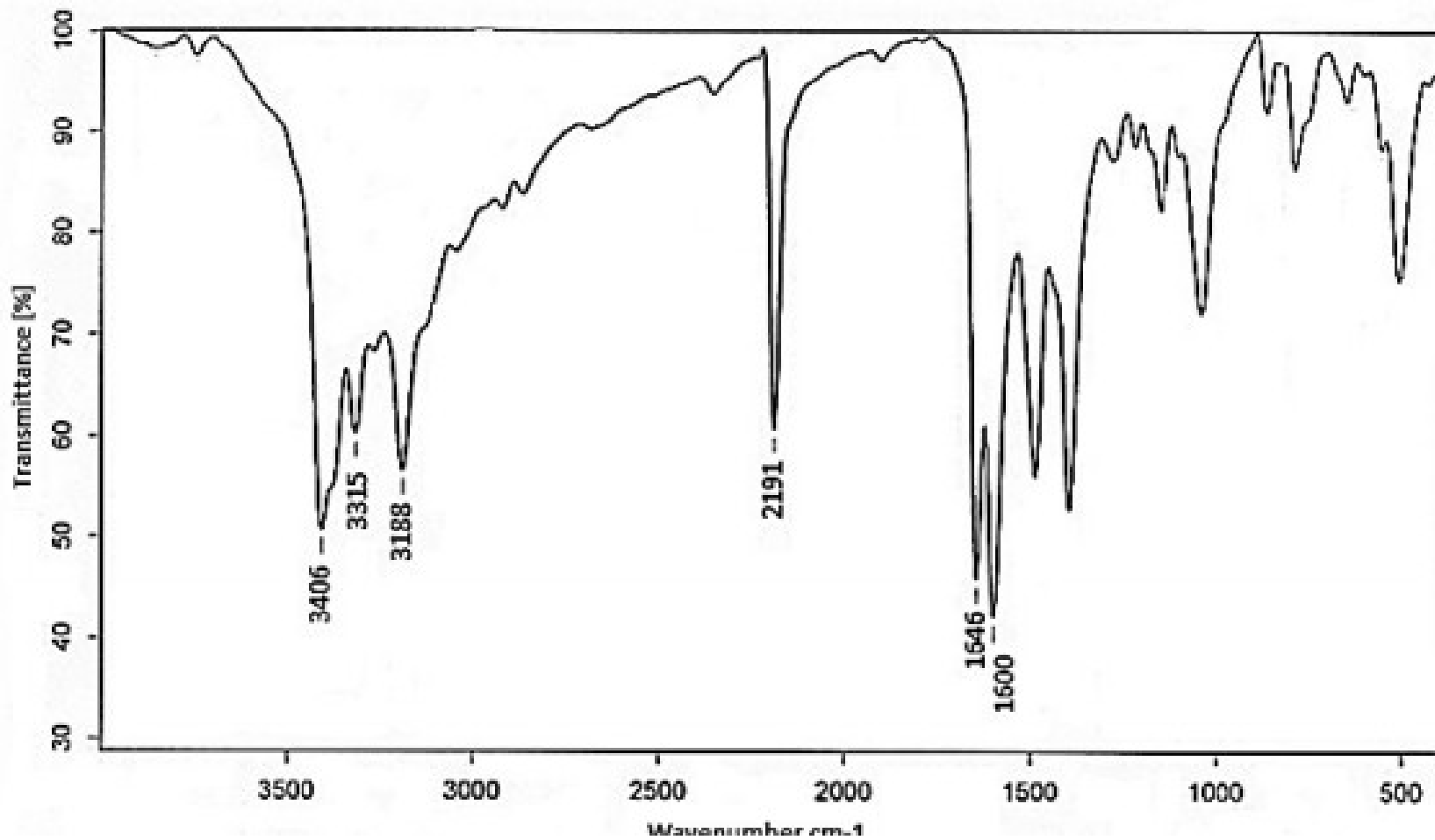


Figure 4. IR spectrum of compound 5b

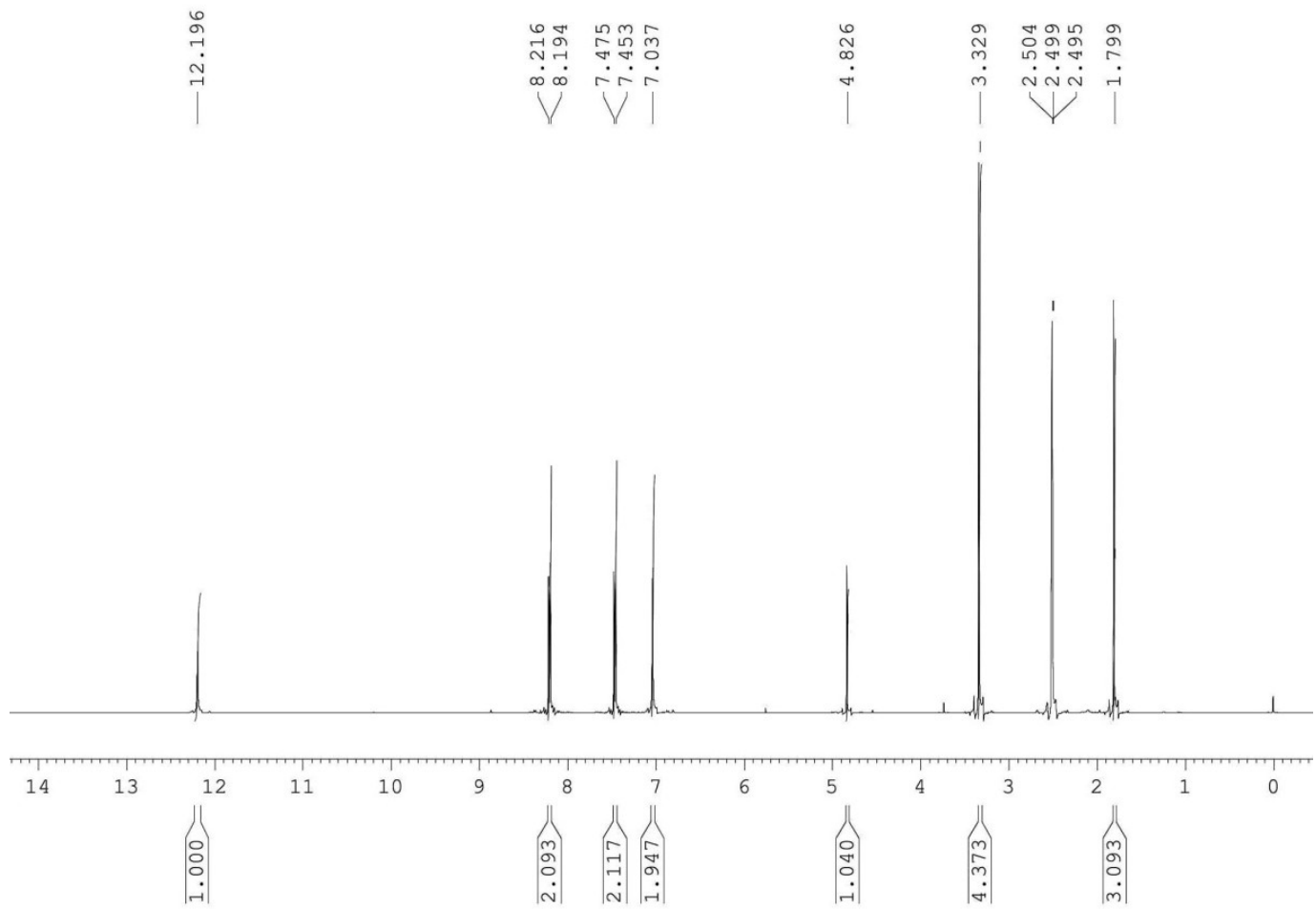


Figure 5. ¹H NMR spectrum of compound **5b**

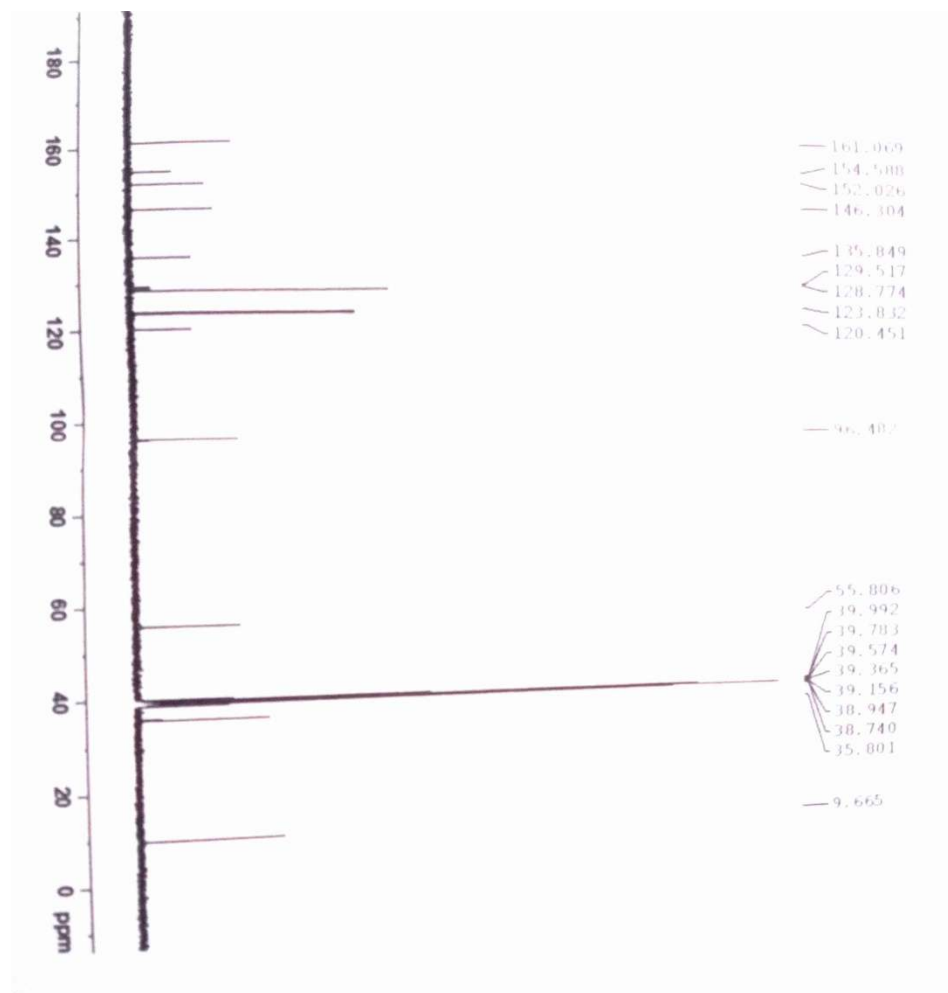


Figure 6. ^{13}C NMR spectrum of compound **5b**

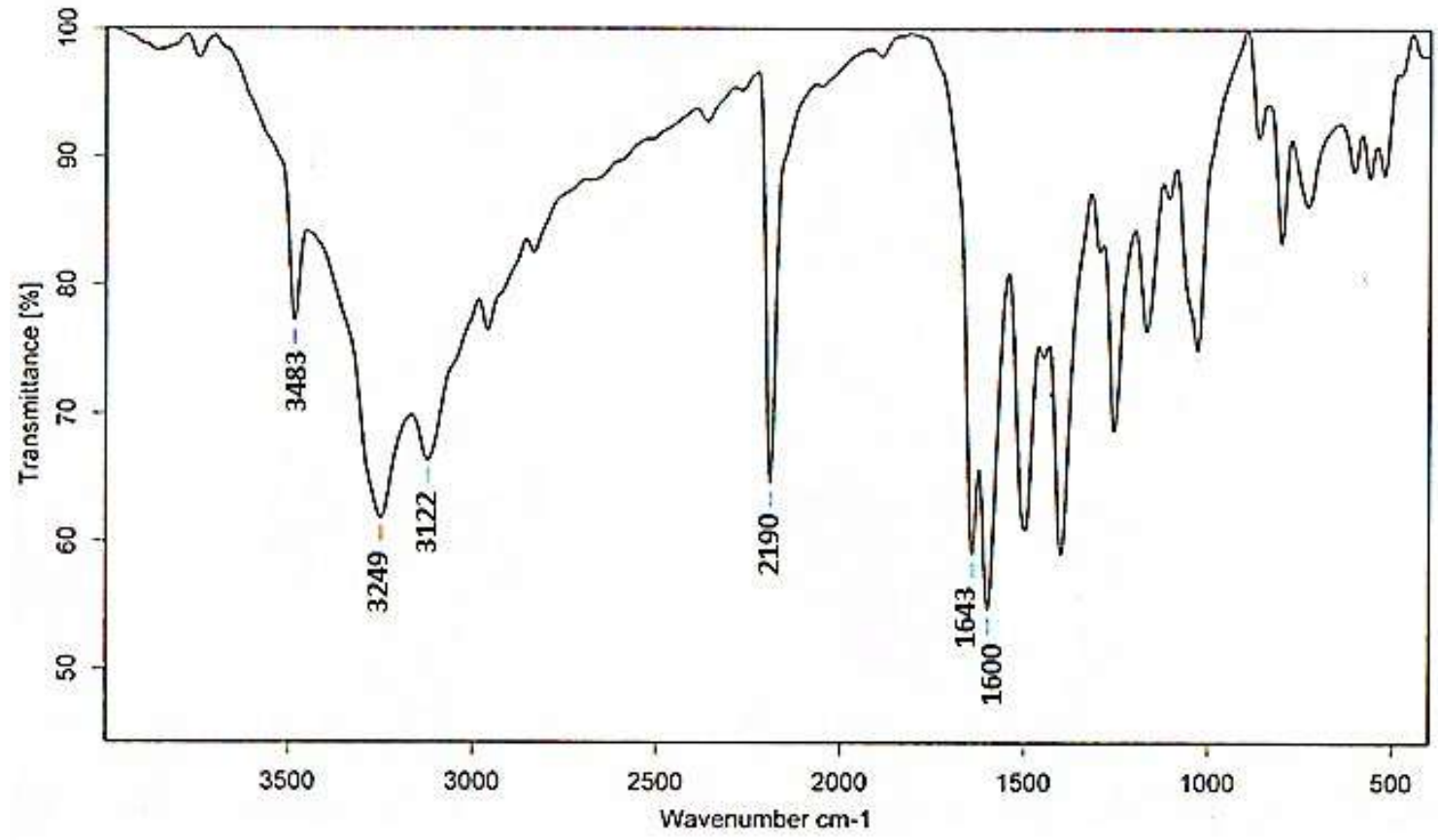


Figure 7. IR spectrum of compound 5c

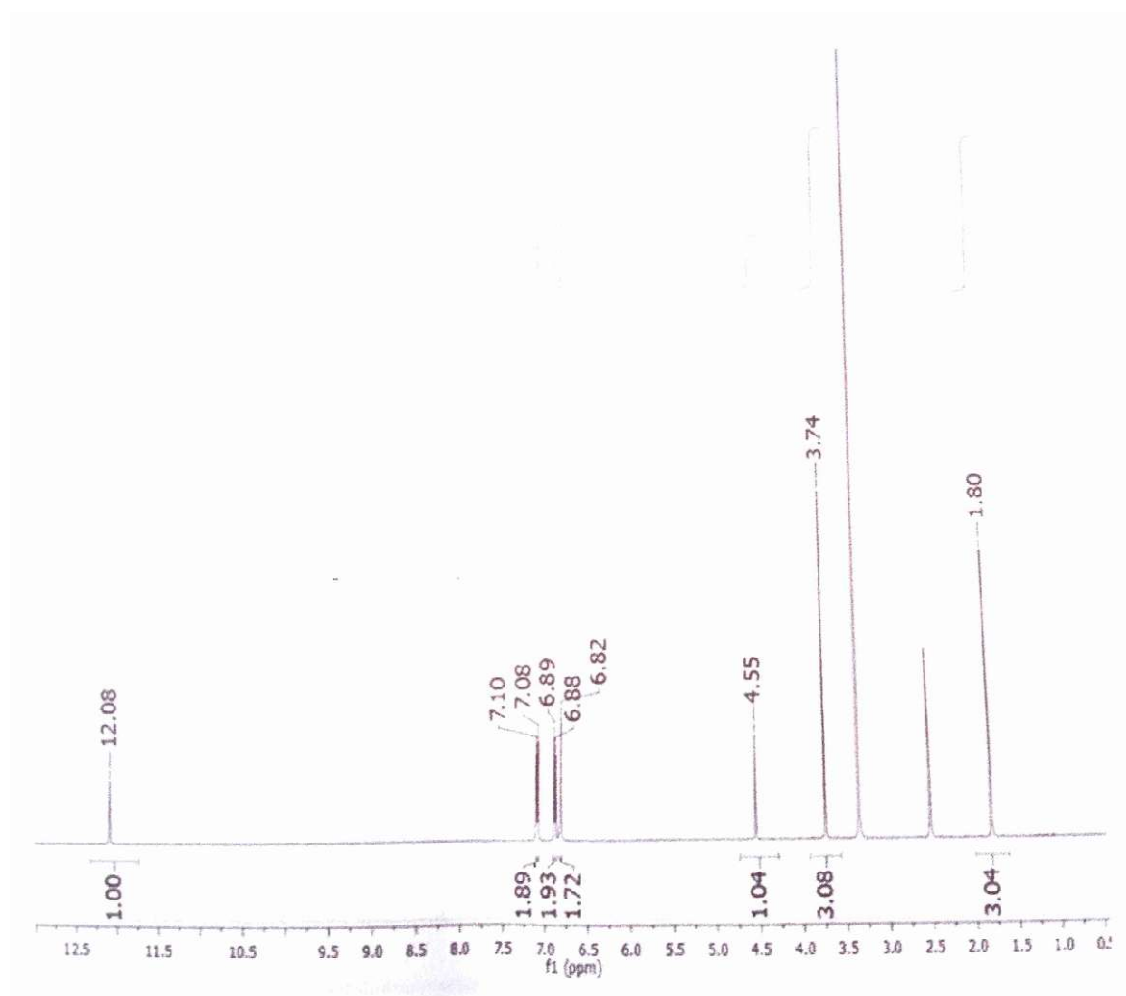


Figure 8. ^1H NMR spectrum of compound 5c

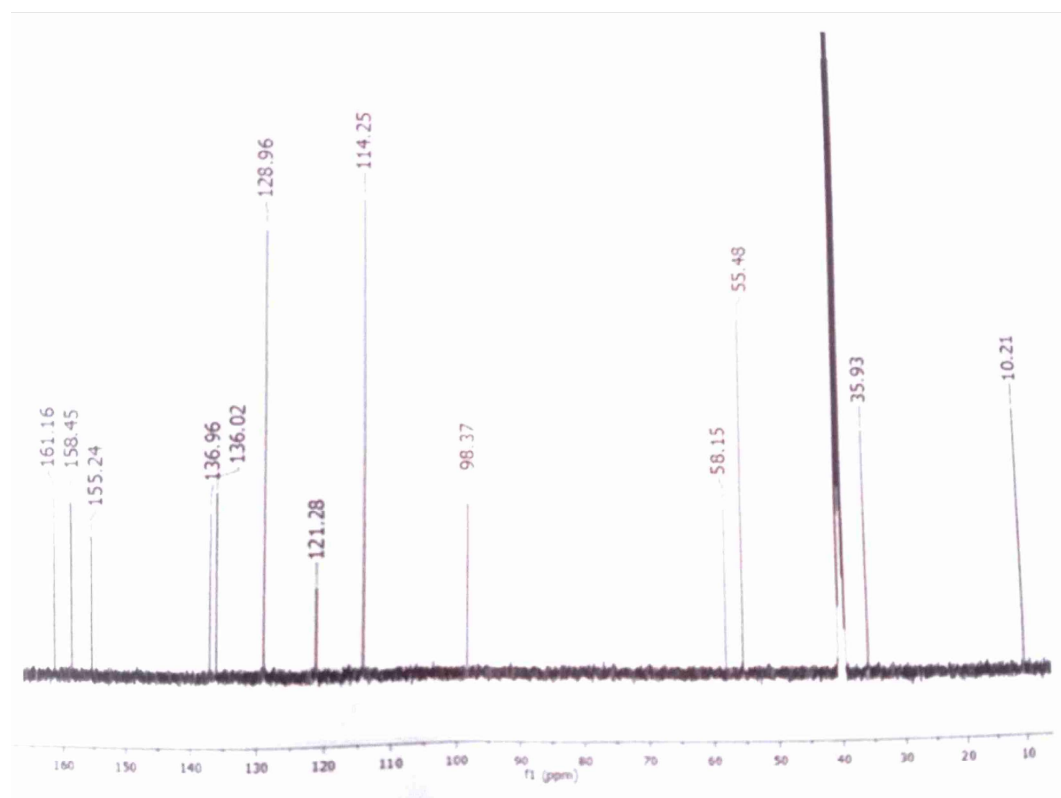


Figure 9. ^{13}C NMR spectrum of compound 5c

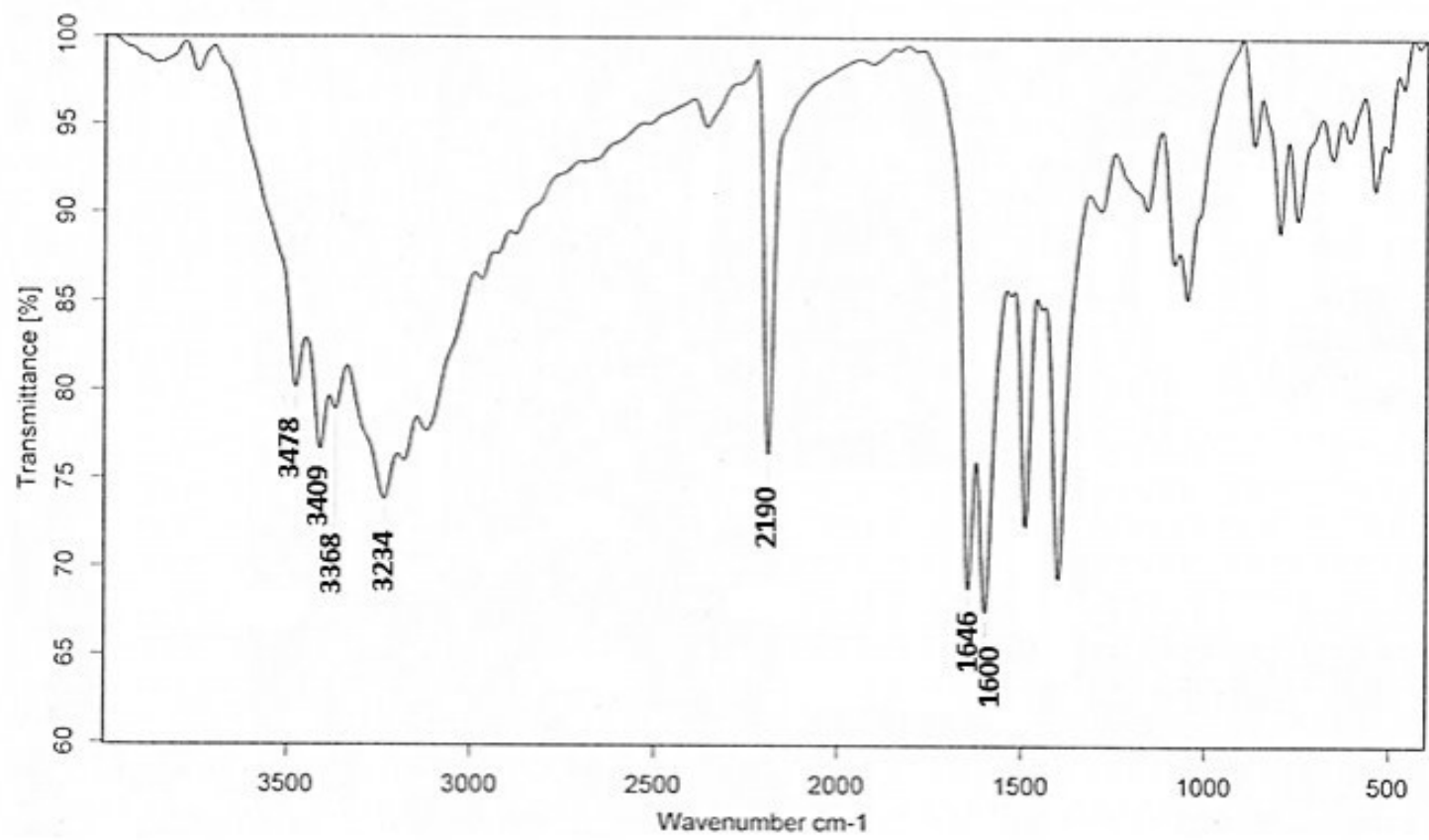


Figure 10. IR spectrum of compound **5d**

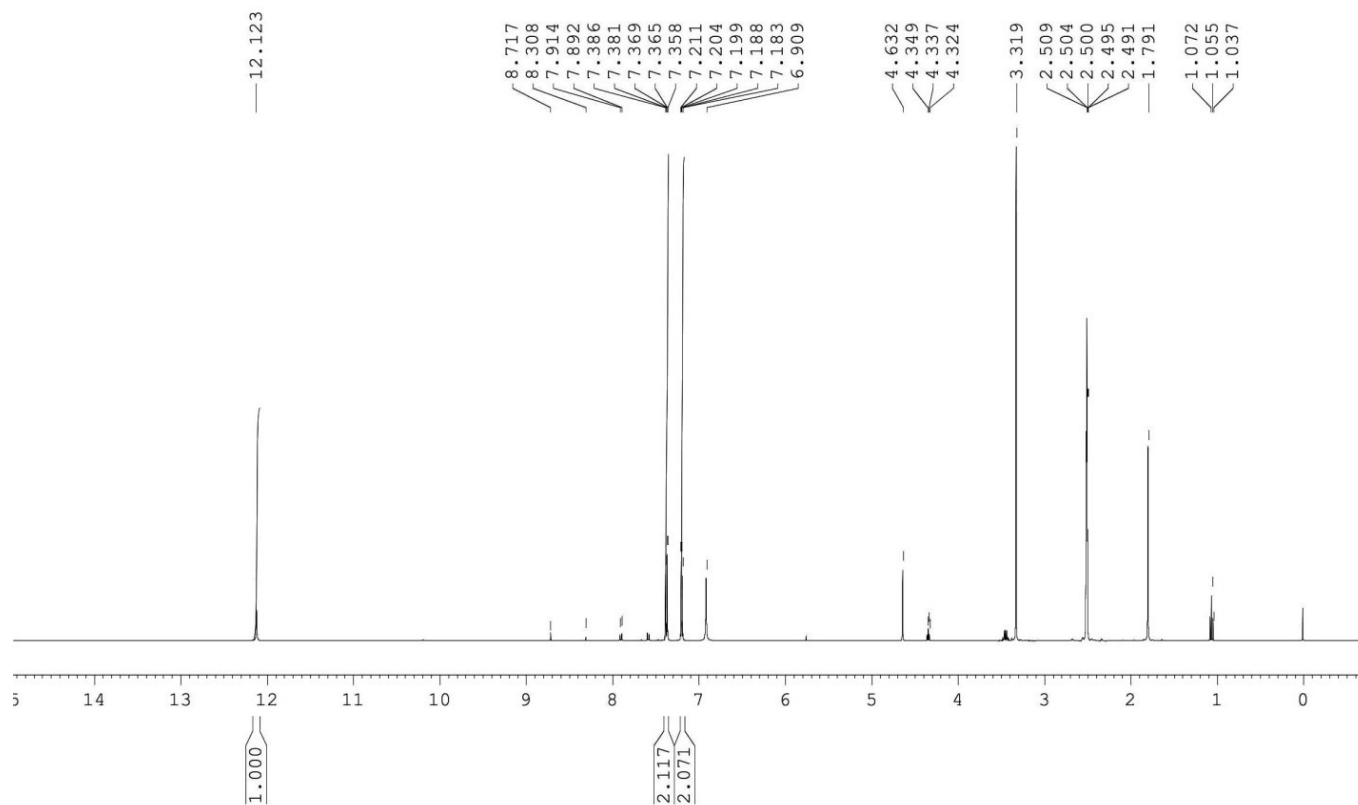


Figure 11. ¹H NMR spectrum of compound 5d

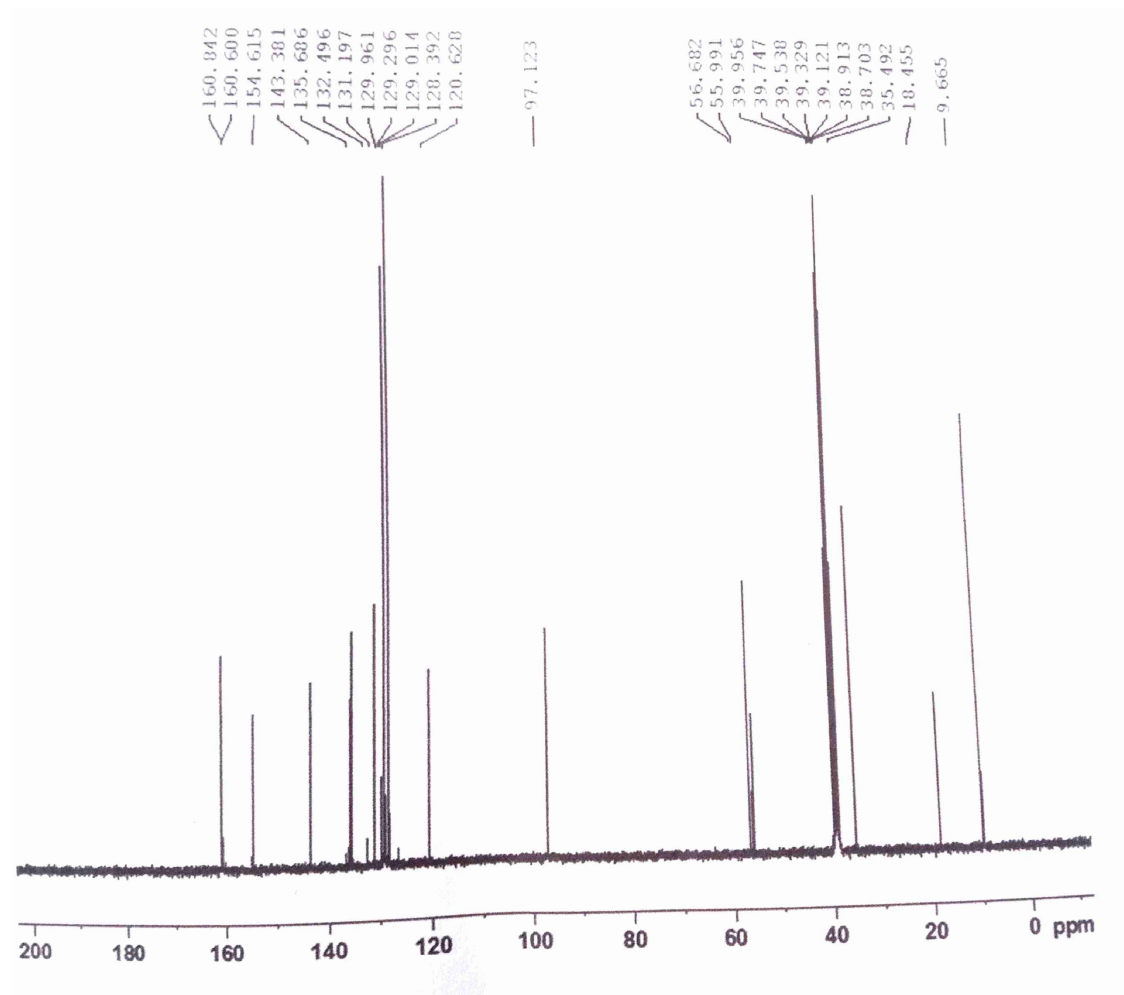


Figure 12. ^{13}C NMR spectrum of compound 5d

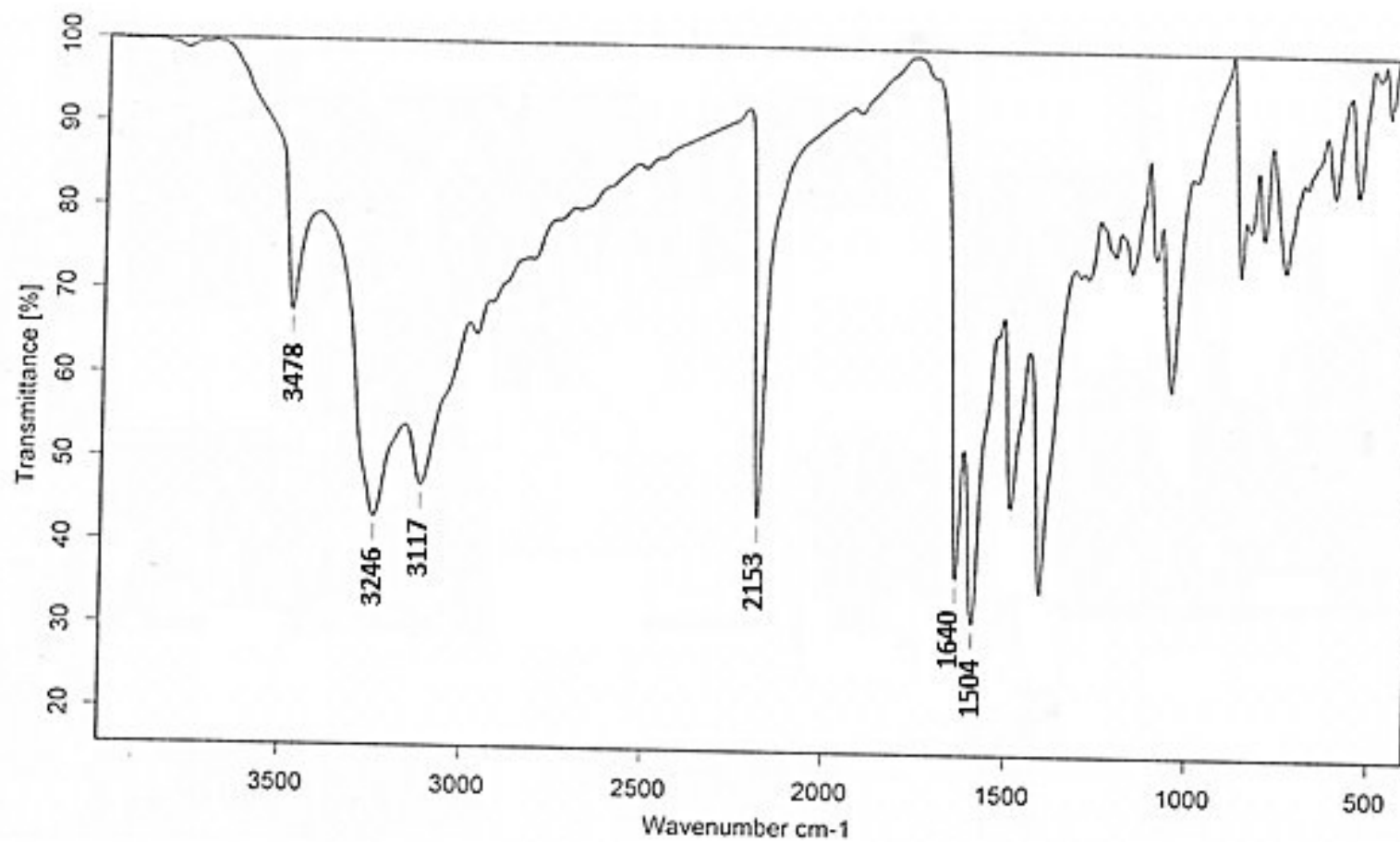


Figure 13. IR spectrum of compound 5e

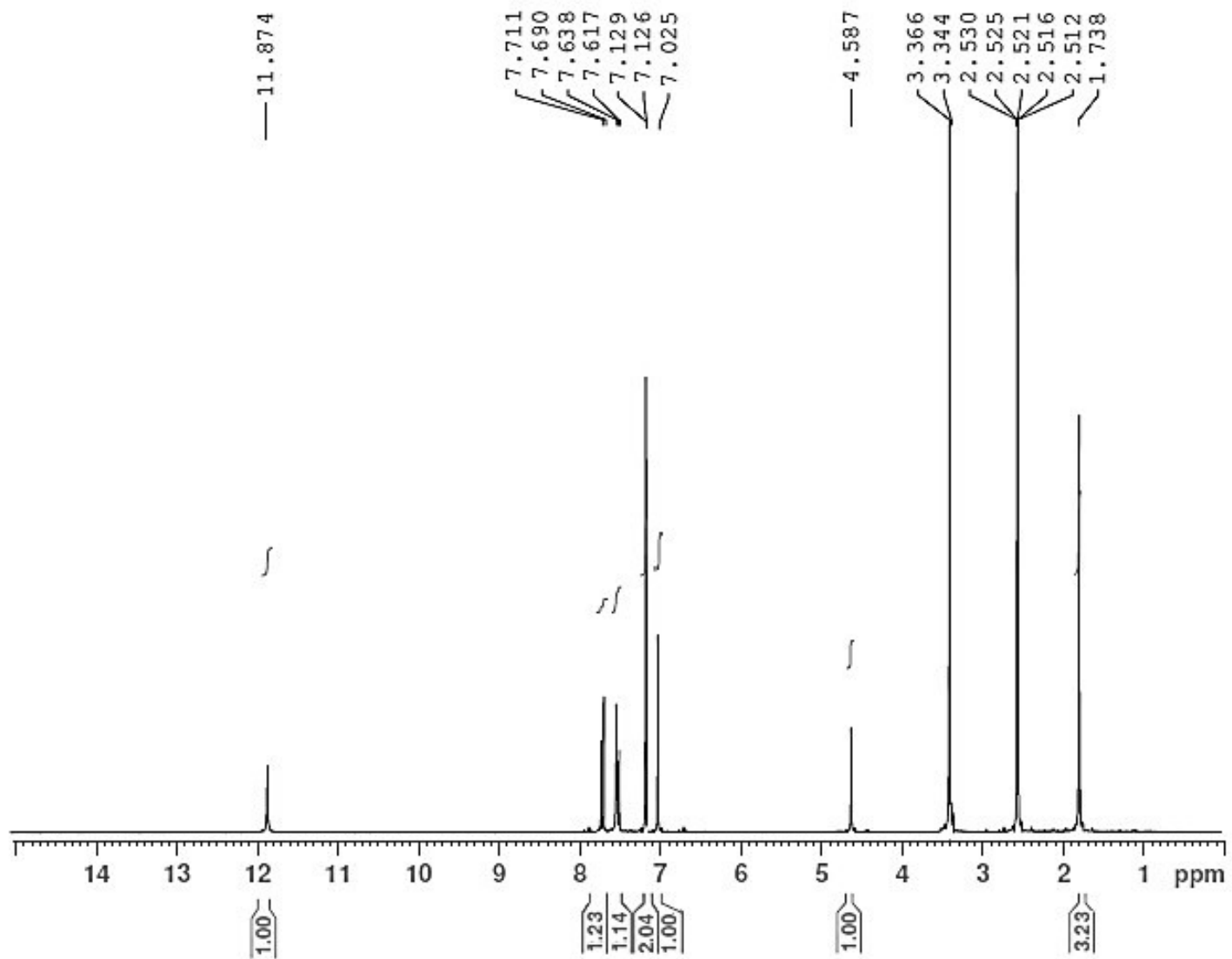


Figure 14. ¹H NMR spectrum of compound 5e

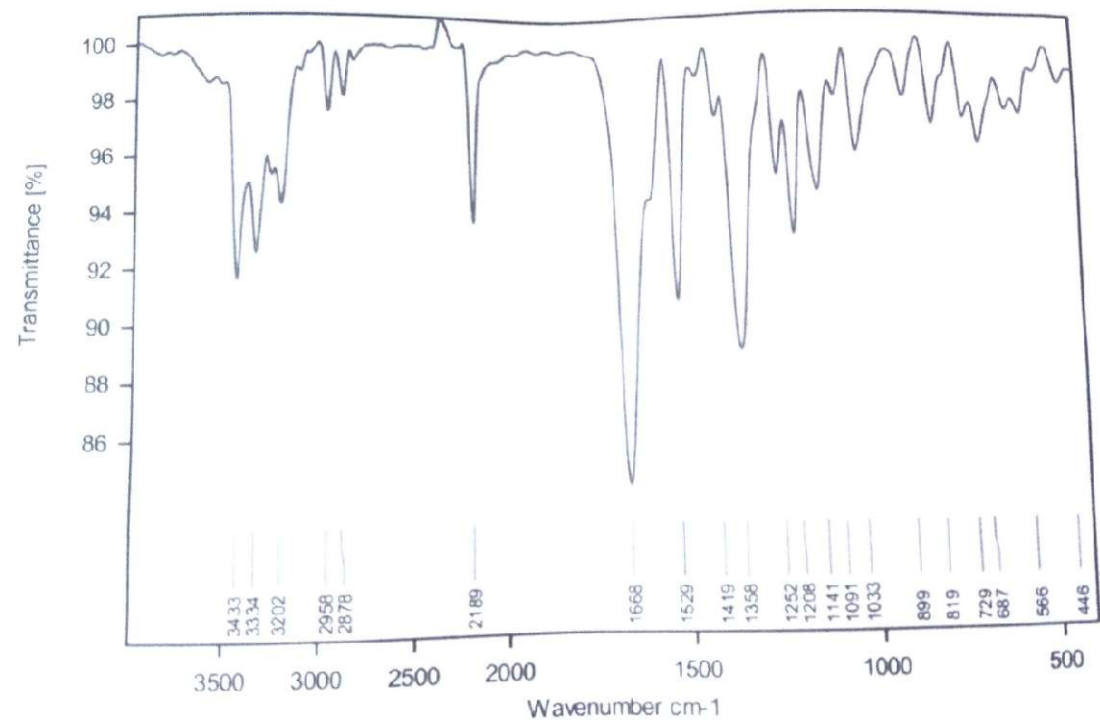


Figure 15. IR spectrum of compound **5f**

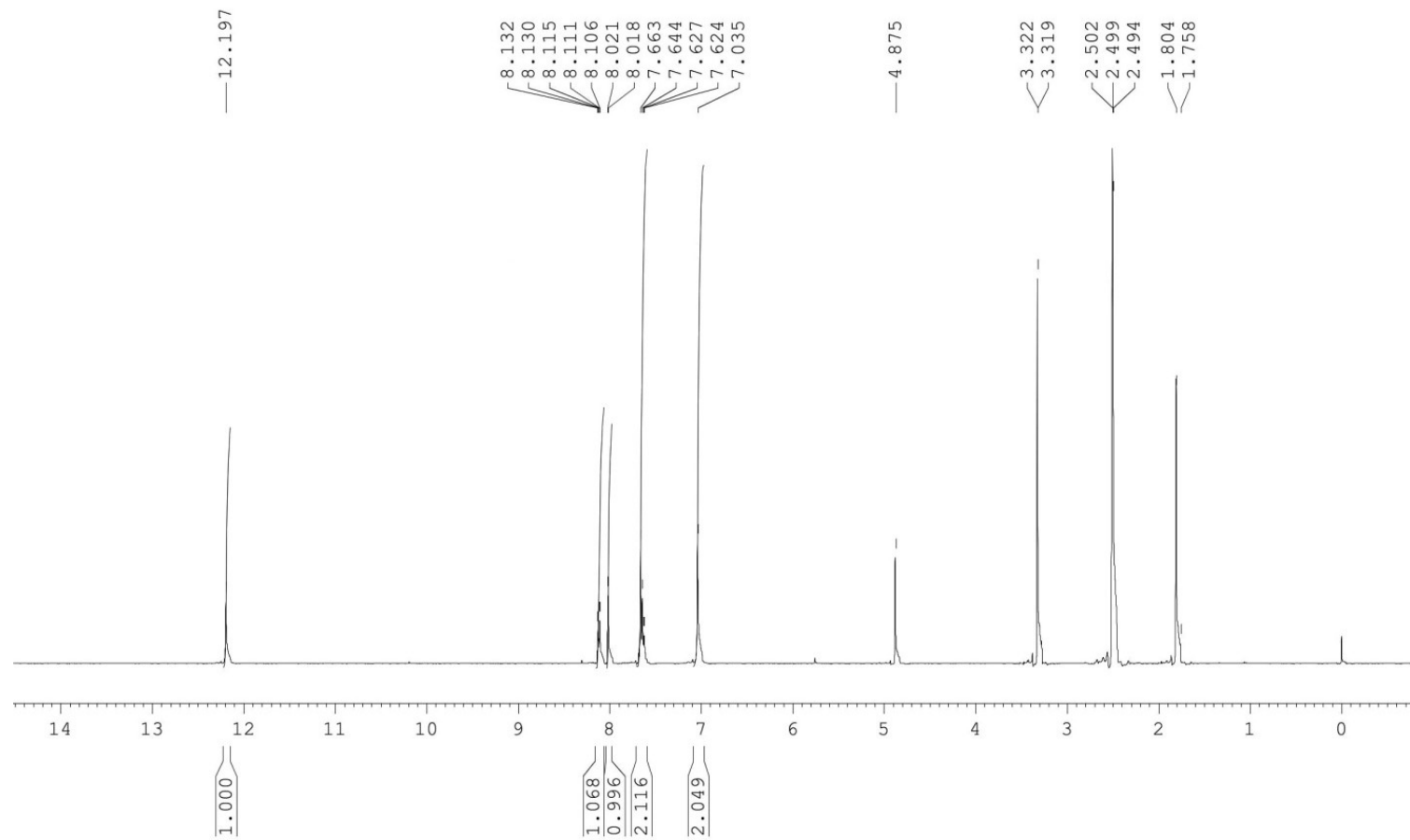


Figure 16. ^1H NMR spectrum of compound **5f**

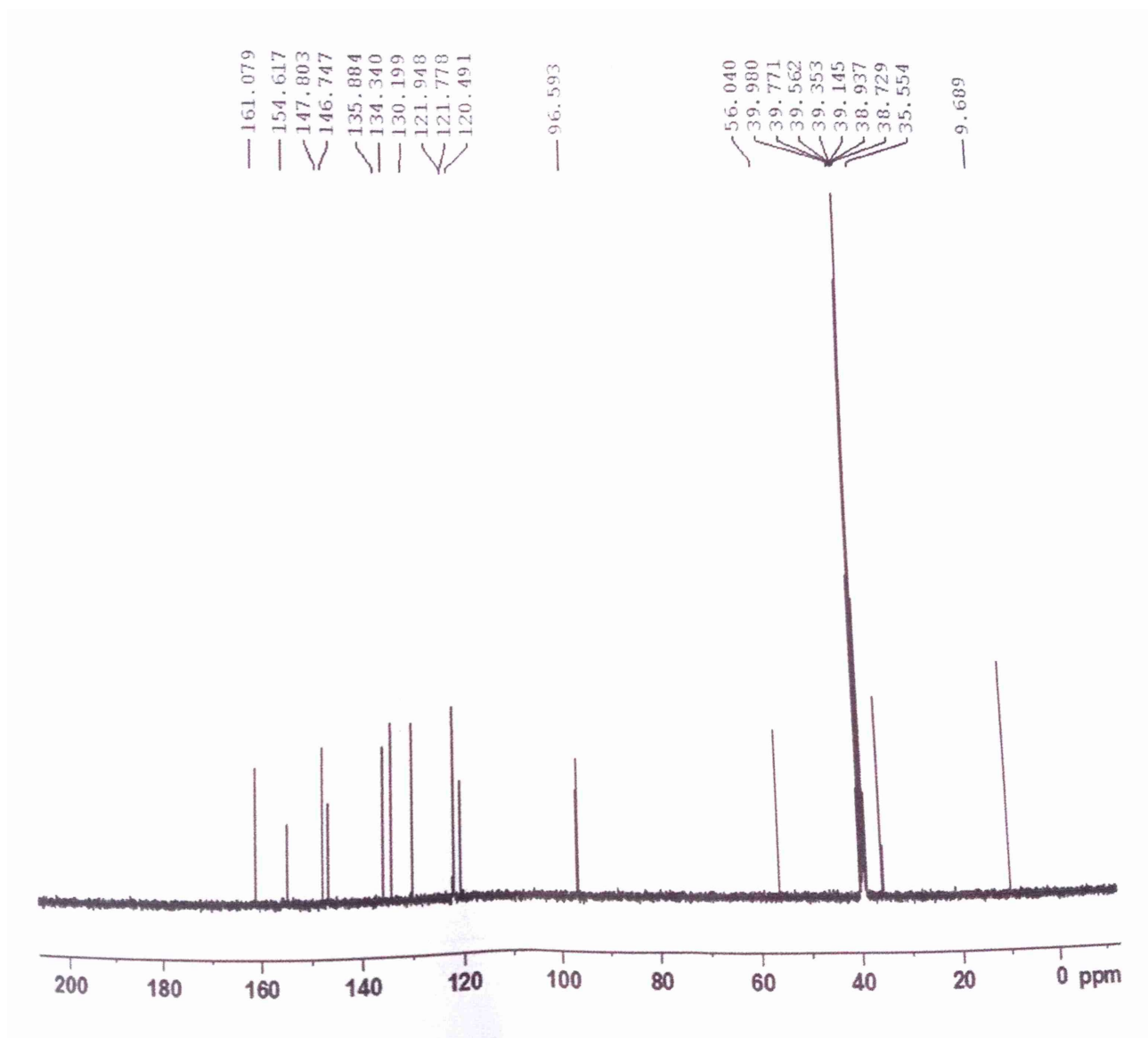


Figure 17. ^{13}C NMR spectrum of compound **5f**

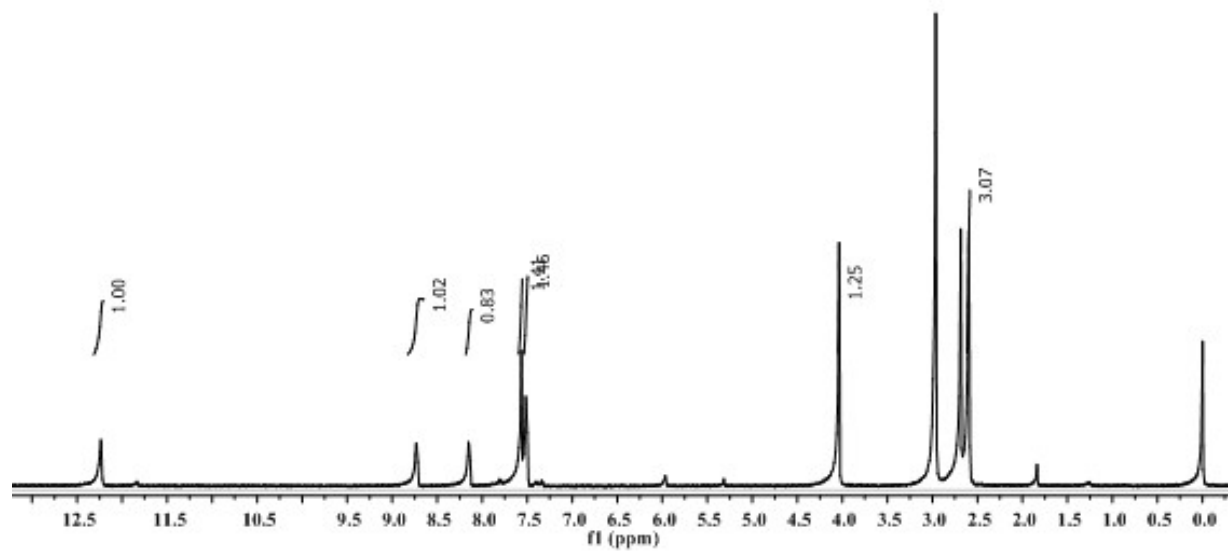


Figure 18. ^1H NMR spectrum of compound **5g**

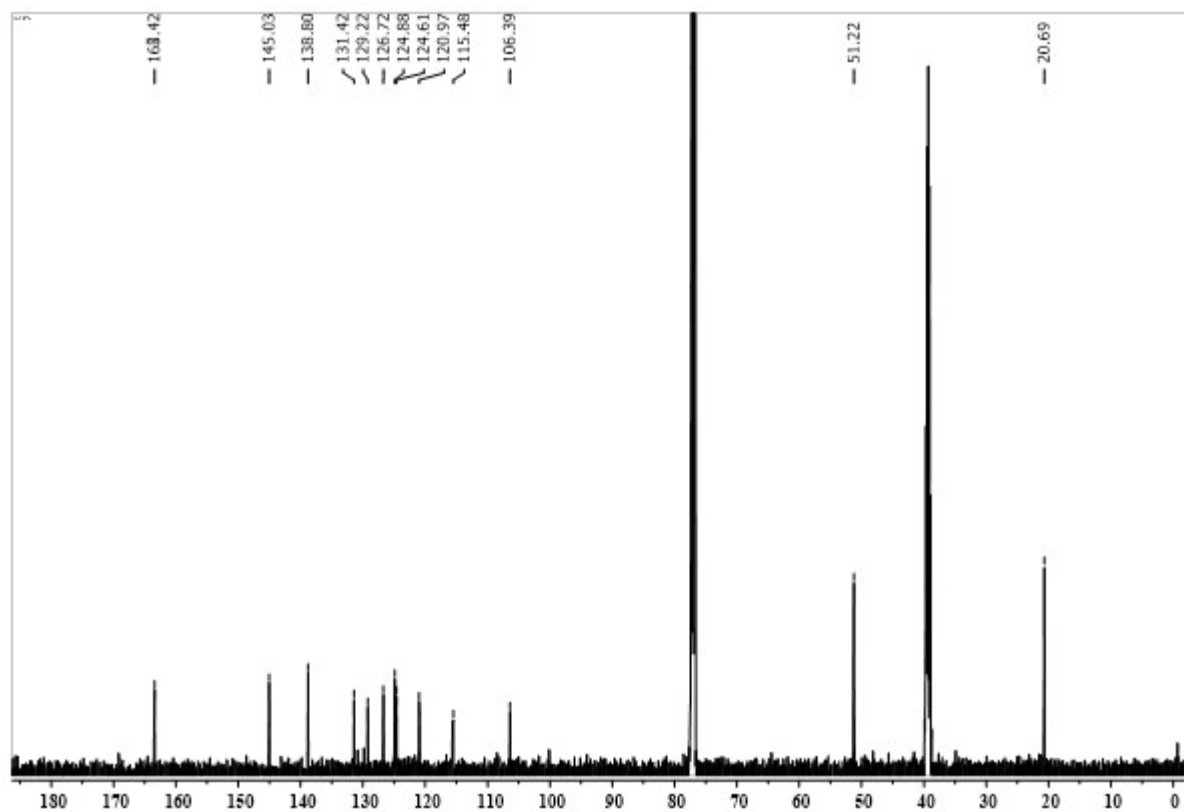


Figure 19. ^{13}C NMR spectrum of compound **5g**

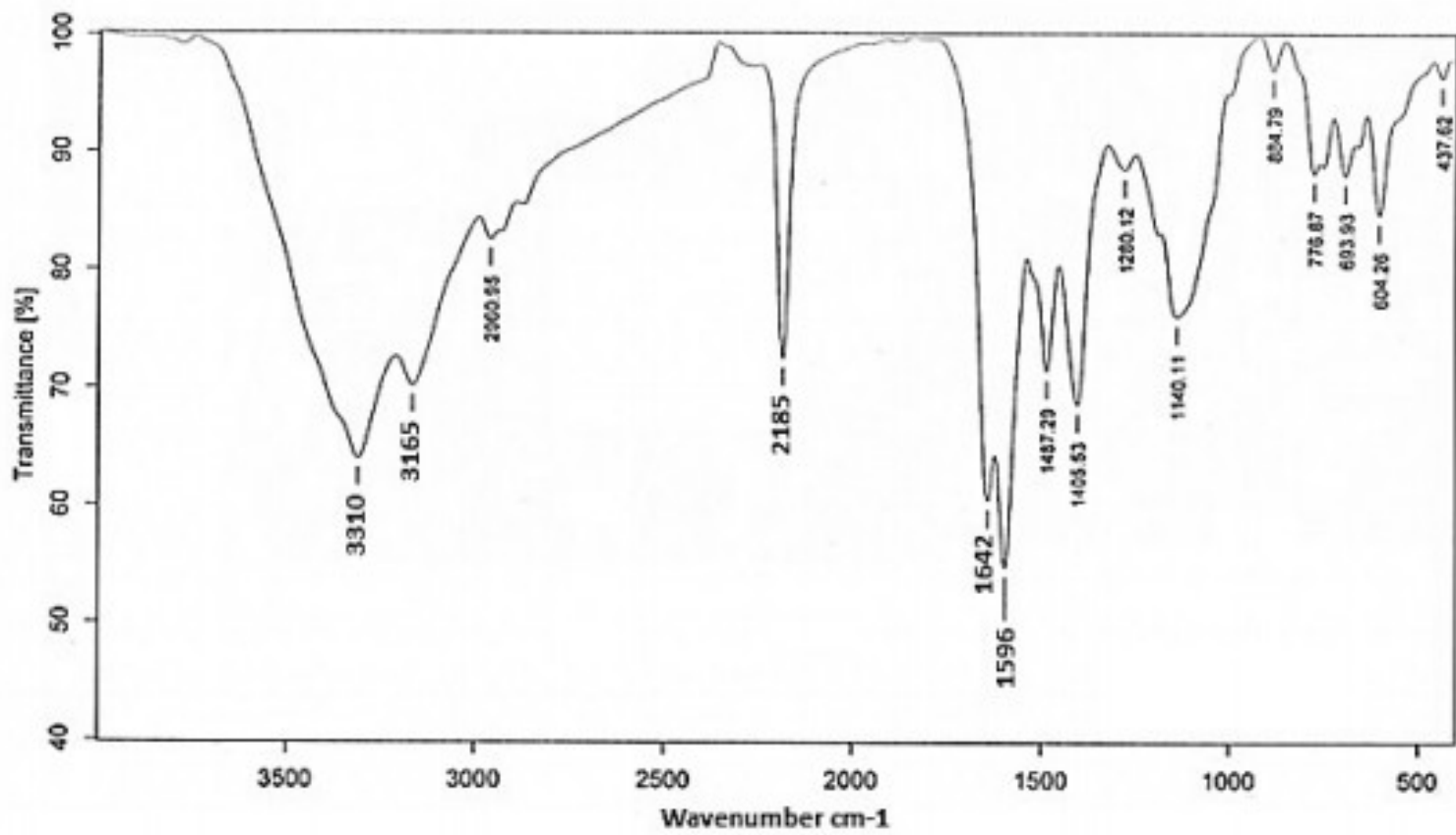


Figure 20. IR spectrum of compound 5h

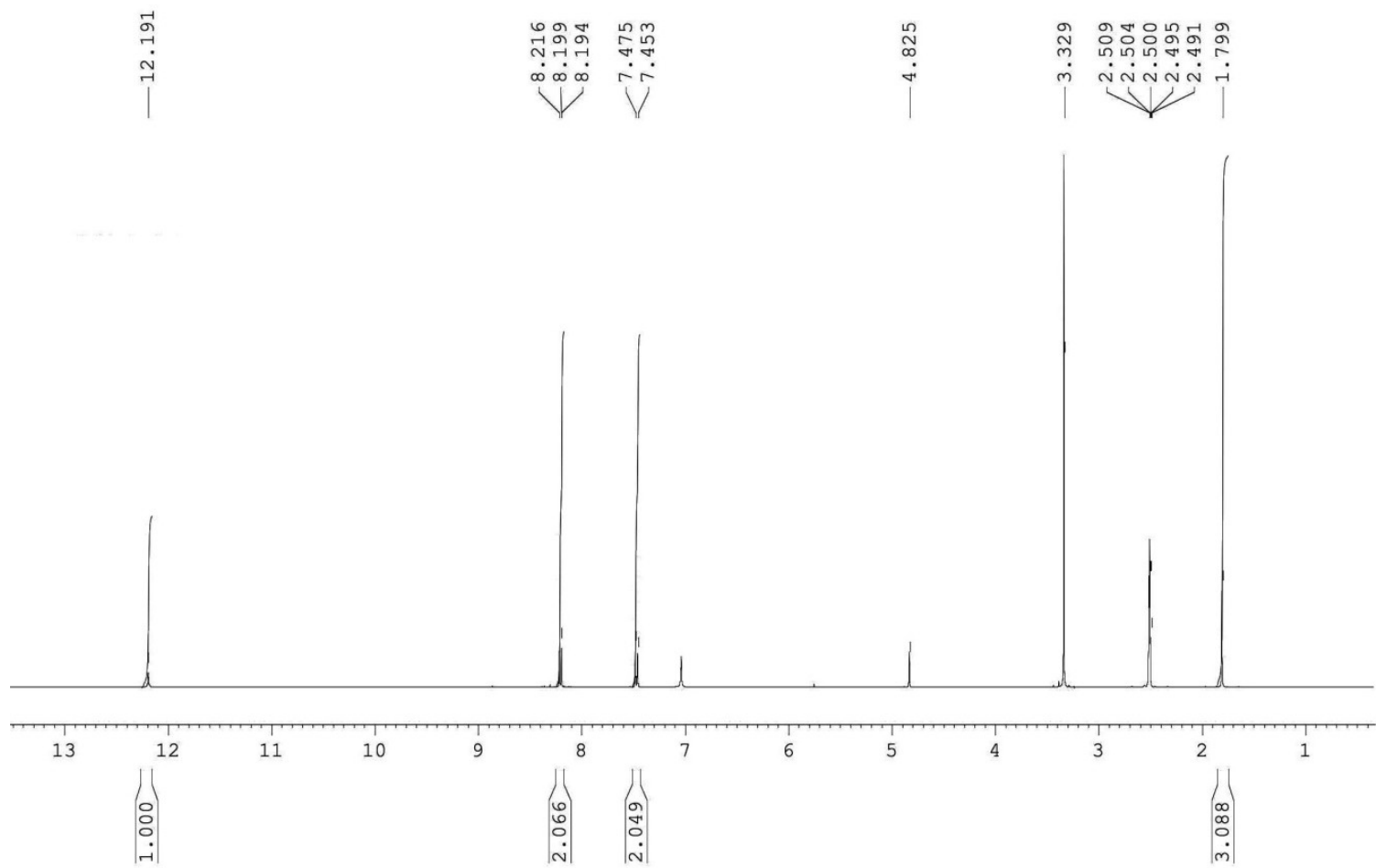


Figure 21. ^1H NMR spectrum of compound **5h**

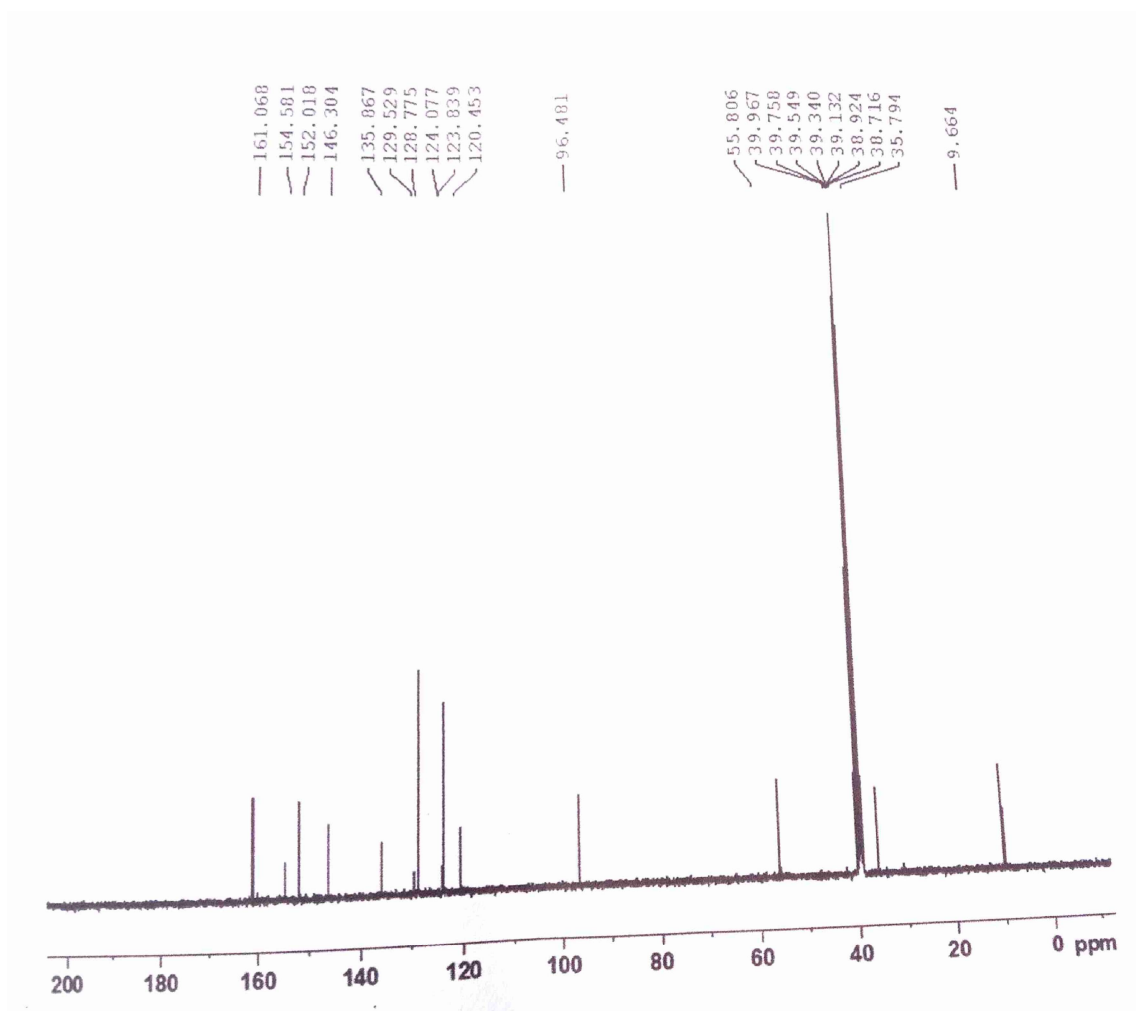


Figure 22. ^{13}C NMR spectrum of compound 5h

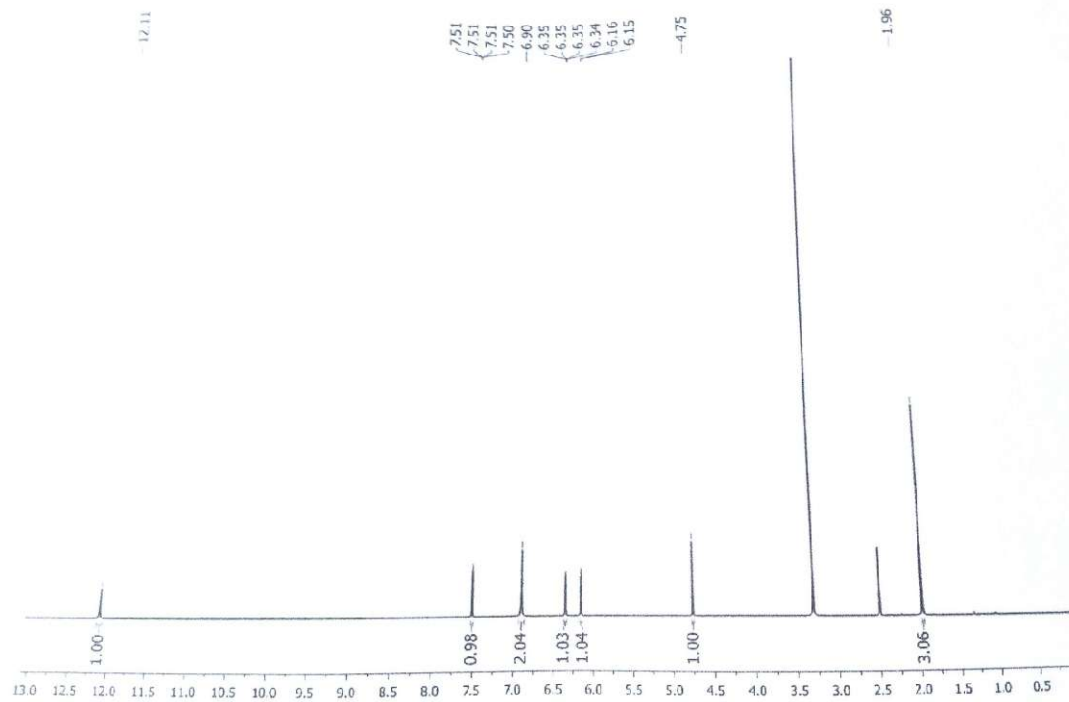


Figure 23. ^1H NMR spectrum of compound **5i**

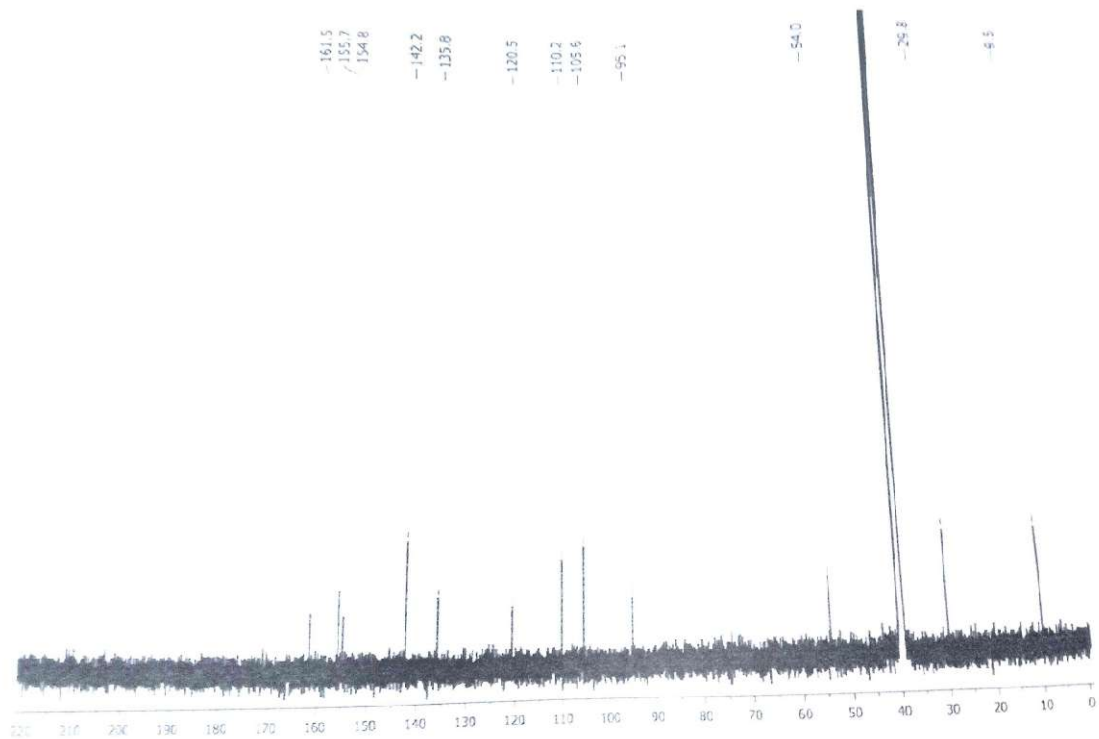


Figure 24. ^{13}C NMR spectrum of compound **5i**

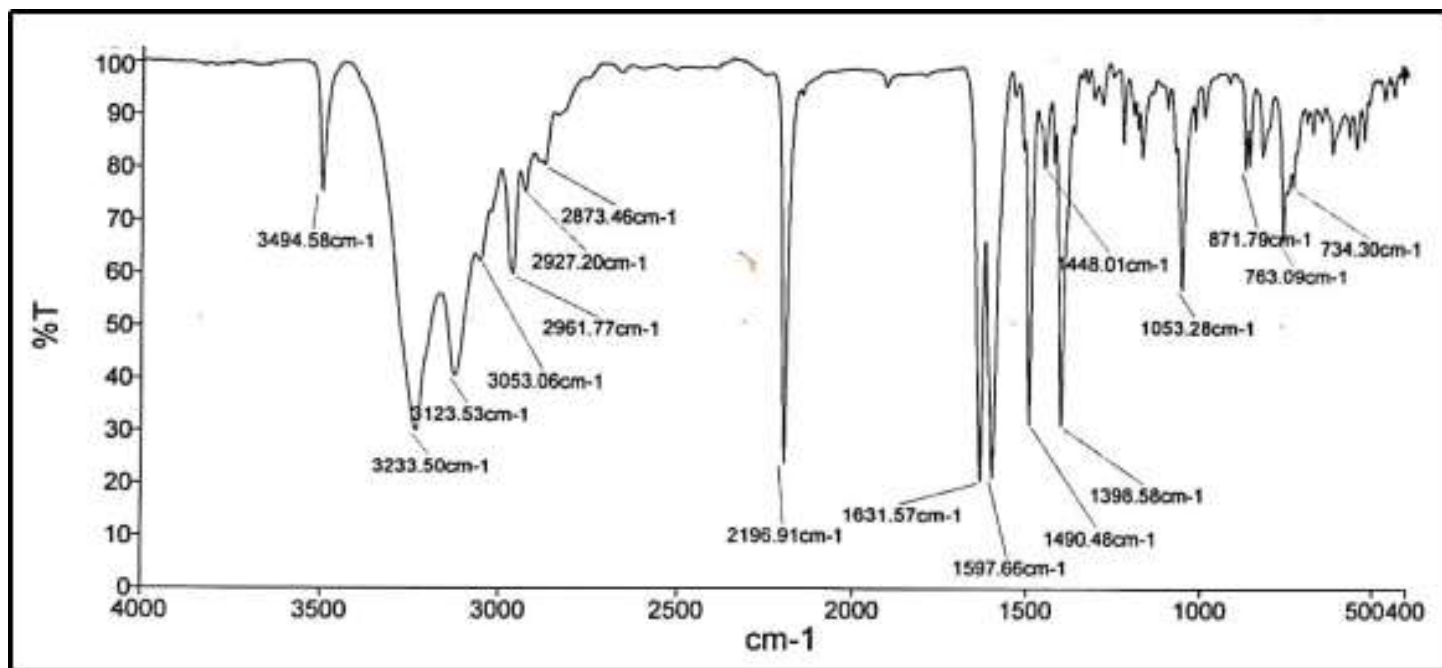


Figure 25. IR spectrum of compound 5j

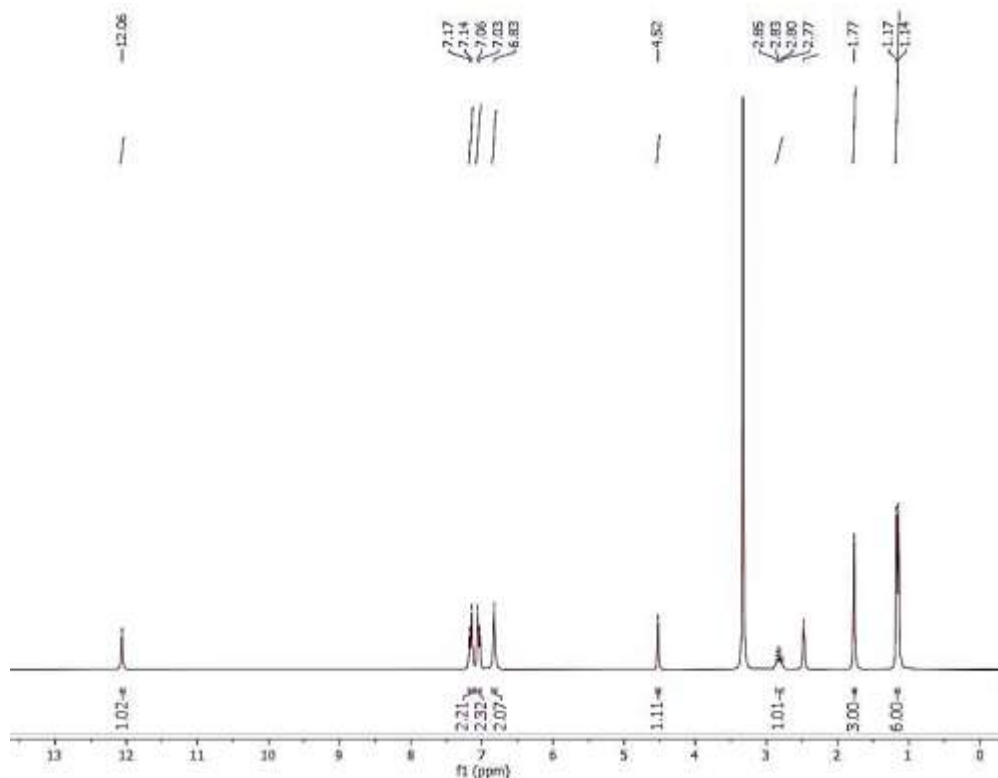


Figure 26. ^1H NMR spectrum of compound **5j**