

SUPPLEMENTARY MATERIALS

Microwave Assisted Synthesis and Antimicrobial Potential of Quinoline-Based 4-Hydrazide-Hydrazone Derivatives

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Synthesis of 2-propylquinoline-4-carboxylic acid (1). Isatin (30.00 g, 204 mmol) was added to a solution of potassium hydroxide (11.42 g, 204 mmol) in H₂O (300 mL) with continuous stirring for 30 minutes at room temperature until all the solutes have dissolved, resulting in a yellowish coloured solution. Pentan-2-one (43.37 mL, 408 mmol) was added slowly to the yellowish solution and resulting mixture was heated under reflux at a temperature of 80 °C while stirring for about 9 h on a heating mantle. The reaction mixture was cooled on ice bath in a round bottom flask, and then acidified using drop-wise addition of concentrated HCl to achieve a pH of 1-2. Upon cooling, the precipitate formed was filtered using suction filtration, washed with water to remove inorganic salt and air-dried to afford 2-propylquinoline-4-carboxylic acid (1).

Synthesis of ethyl 2-propylquinoline-4-carboxylate (2). The precursor compound (1) formed was put into a round bottomed flask and 25.5 mL of freshly distilled absolute ethanol was added into the round bottom flask, while stirring for about 10 mins, thereafter concentrated sulphuric acid (3.0 mL) was added down the walls of the flask in a drop-wise manner, with the reaction mixture still stirring using a magnetic stirrer, 3 boiling stones were added to the reaction mixture to avoid bumping. The reaction mixture was then heated under reflux for 1 h at a temperature of 60-80 °C. Upon the completion of reaction, the resulting mixture was cooled for 30 mins, and poured into a separatory funnel containing 50 mL of water, the round bottom flask was rinsed with 25 mL of water and was added to the content in separatory funnel. It was subsequently extracted with 35 mL of diethyl ether in two portions. The ether layer was combined and dried over anhydrous Na₂SO₄. It was filtered and evaporated to dryness to afford ethyl 2-propylquinoline-4-carboxylate (2).

Synthesis of 2-propylquinoline-4-carbohydrazide (3). Ethyl 2-propylquinoline-4-carboxylate 2 (3 g, 12.34 mmol) was dissolved in 20 mL of ethanol under continuous stirring at room temperature until complete dissolution was achieved. Hydrazine hydrate (0.74 g, 14.81 mmol, 1.20 eq.) was then added drop-wisely to the solution above over a period of 5 min after which the mixture was heated under reflux for 1 h. It was allowed to cool and the precipitate which crystallized out was filtered by suction to afford 2-propylquinoline-4-carbohydrazide (3).

Antibacterial sensitivity testing of compounds. All the synthesized benzimidazole templates and gentamicin were screened for antibacterial activity on the targeted organisms mentioned above using agar well diffusion method [25]. The medium employed was diagnostic sensitivity test agar (Biotech Ltd). With the aid of a sterile 1 mL pipette, about 0.2 mL of the broth culture of test organism was added to 18 mL sterile molten diagnostic sensitivity test agar (Biotech Ltd) which had already cooled down to 45 °C. This was well mixed and poured into previously sterilized petri dishes, which had been properly labeled according to the test organisms. The medium was then allowed to set. With the aid of a sterile cork borer, the required numbers of holes were bored into the medium. The wells were made of about 5 mm to the edge of the plate. The wells were then filled up aseptically with the solution of the compound in DMSO using Pasteur pipettes. Gentamicin was used as the standard antibacterial agent at a concentration of 1000 µg/mL. The plates were allowed to stand for about 1 h on the bench for proper diffusion of the antibacterial agents into the medium and then incubated uprightly at 37 °C for 24 h. Care was taken not to stockpile the plates. Clear zones of inhibition (Z.O.I.) in millimeters (mm) indicated the relative susceptibility of the bacteria to the compounds and Gentamicin clinical reference.

Minimum inhibitory concentration (MIC). The Minimum Inhibitory Concentration (MIC) was done using the method of Russel and Furr [25]. Based on the level of resistance of some organisms and large zones of inhibition experienced in others, Minimum Inhibitory Concentration (MIC) was selectively done for three gram positive and three gram negative bacterial strains. Different concentrations (0.39, 0.78, 1.56, 3.13, 6.25, 12.50, 25.00 and 50.00 $\mu\text{g/mL}$) of the compounds and standard were prepared using a two fold dilution which was prepared in a sterile plate with the aid of sterile pipette and then mixed with 18 mL of molten nutrient agar. This was then allowed to set. The surface of the nutrient agar plate was allowed to dry before streaking with overnight broth cultures of the bacterial strains. The plates were then labeled accordingly and incubated at 37 °C for up to 72 h. They were subsequently examined for the presence or absence of growth. The lowest concentration preventing the growth of bacteria was taken as the Minimum Inhibitory Concentration of the compounds. This procedure was likewise repeated for the Gentamycin (standard). To ensure that the solvent had no effect on the bacterial growth, a control was performed at the test medium supplemented with DMSO at the same dilutions as used in the experiments.

Spectral data

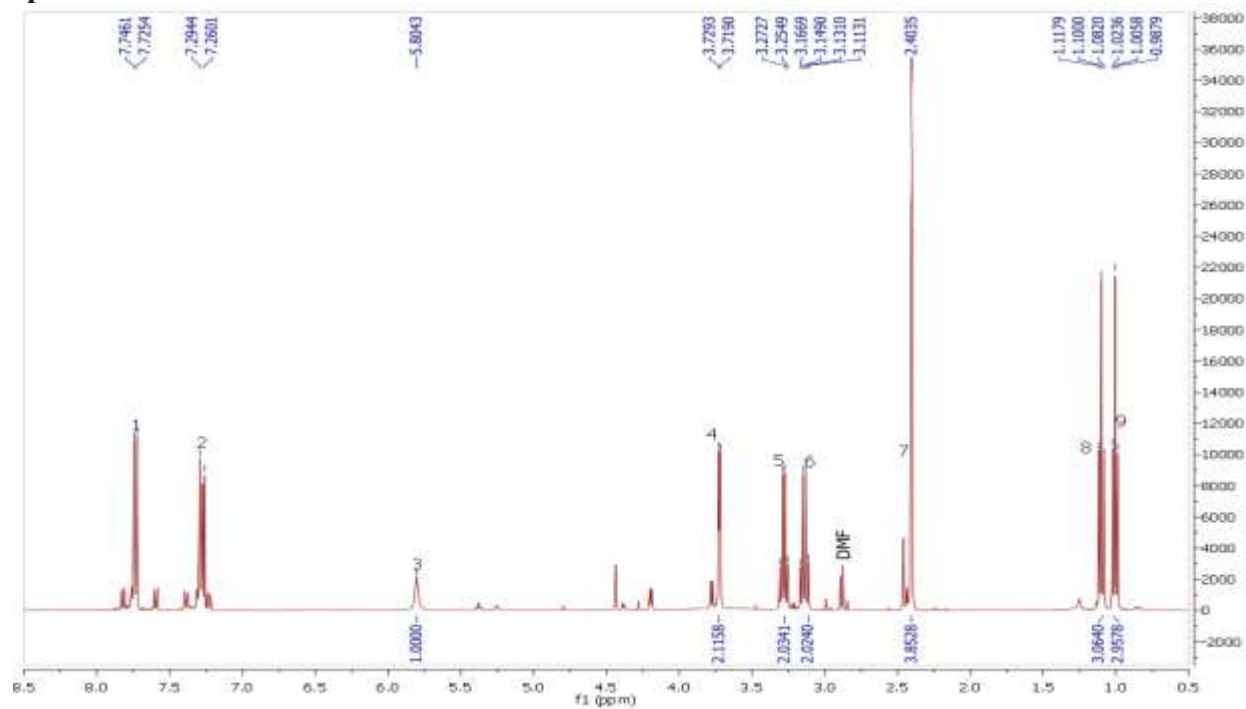


Figure S1. ¹H NMR spectrum of *N'*-(butan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4a**

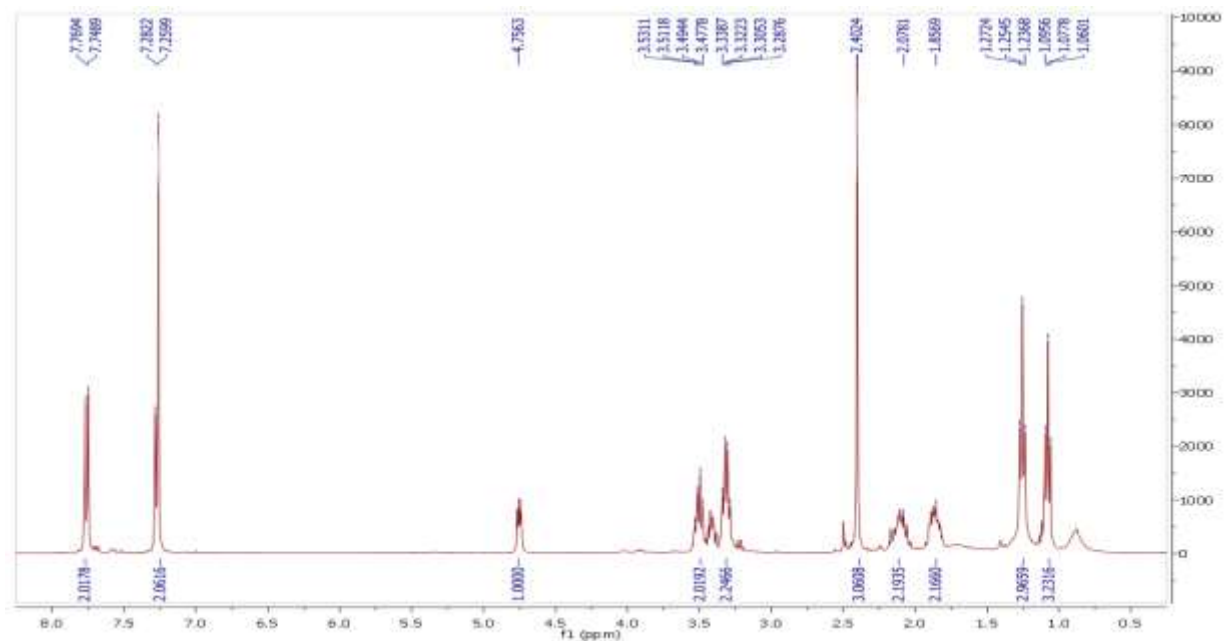


Figure S2. ¹H NMR spectrum of *N'*-(pentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4b**.

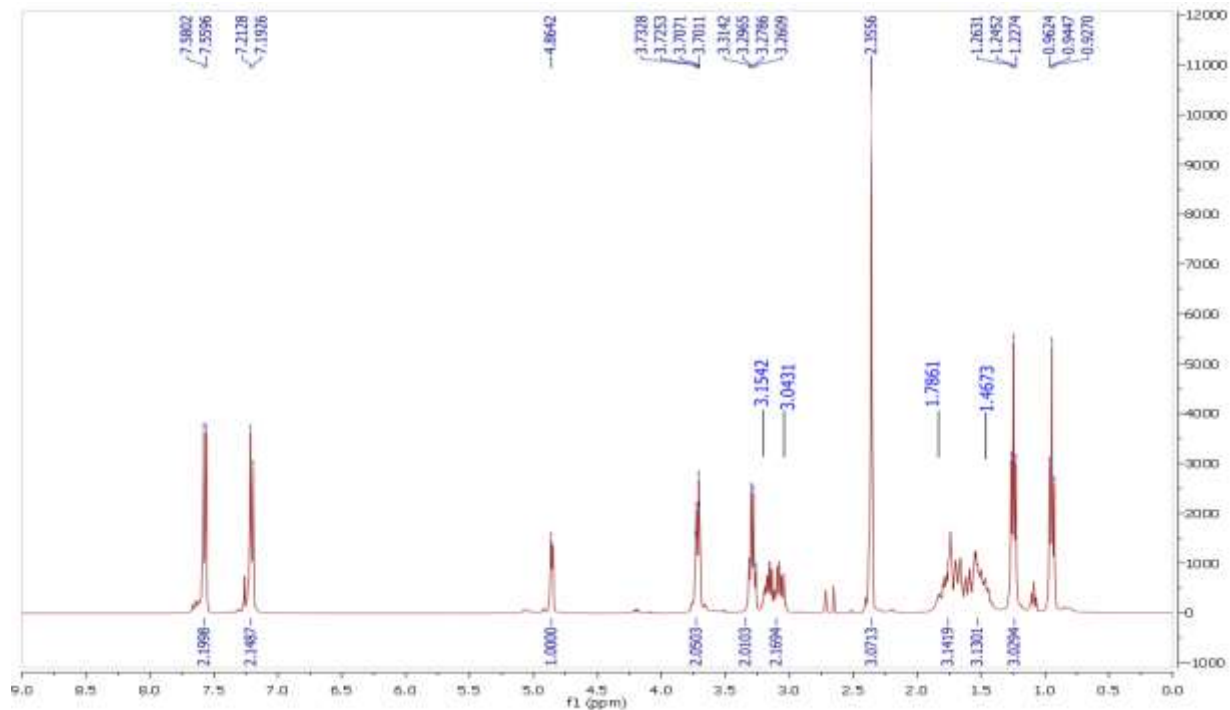


Figure S3. ^1H NMR spectrum of N' -(hepta-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4c**

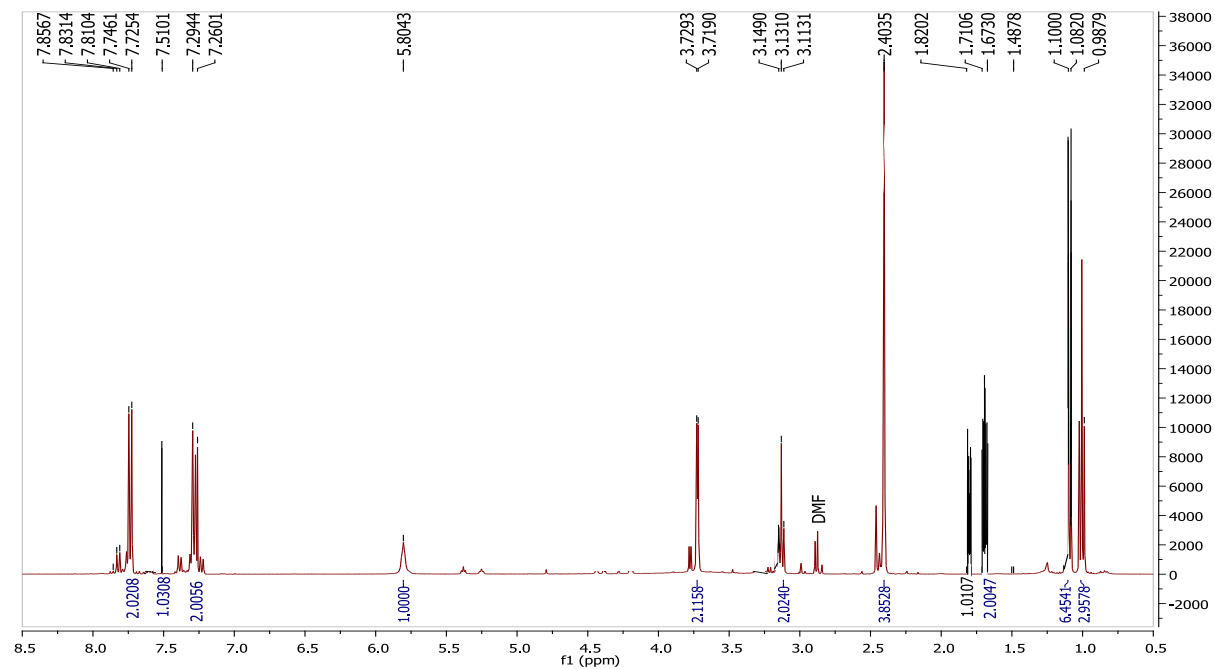


Figure S4. ^1H NMR spectrum of N' -(4-Methylpentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4d**

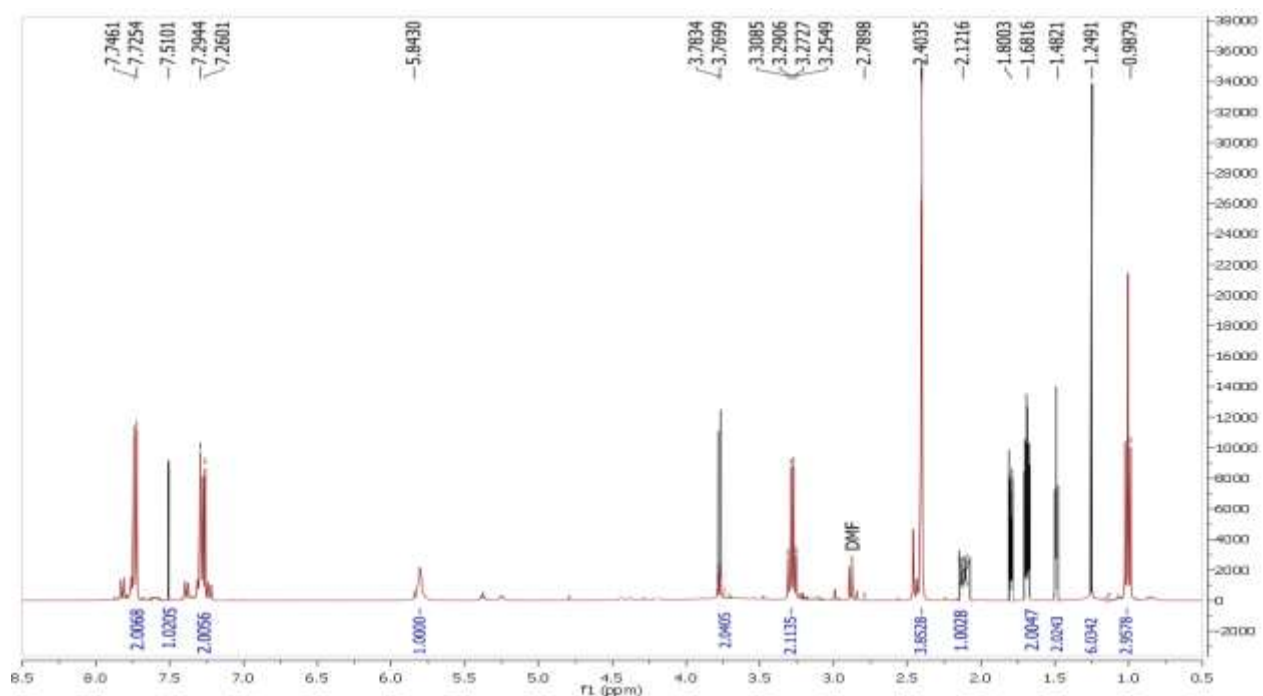


Figure S5. ^1H NMR spectrum of 2-propyl-(N' -(1,7,7-trimethylbicyclo[2.2.1]heptan-2-ylidene)quinoline-4-carbohydrazide, **4e**

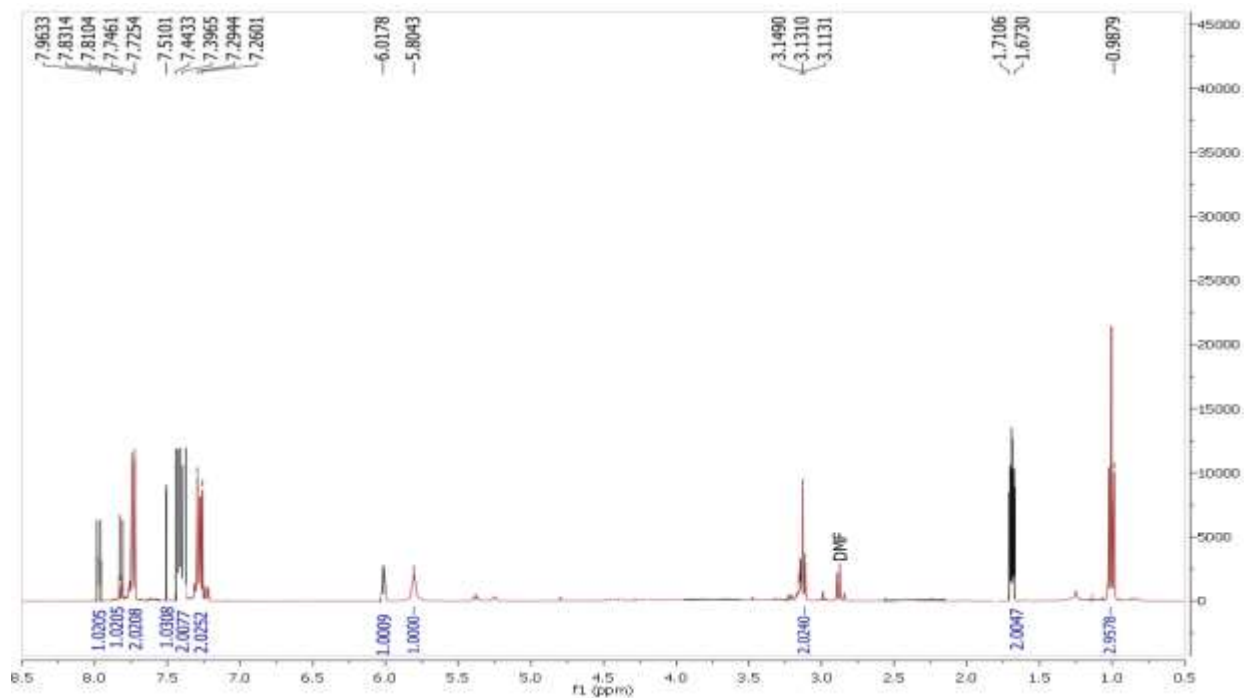


Figure S6. ^1H NMR spectrum of N' -(2-oxoindolin-3-ylidene)-2-propylquinoline-4-carbohydrazide, **4f**

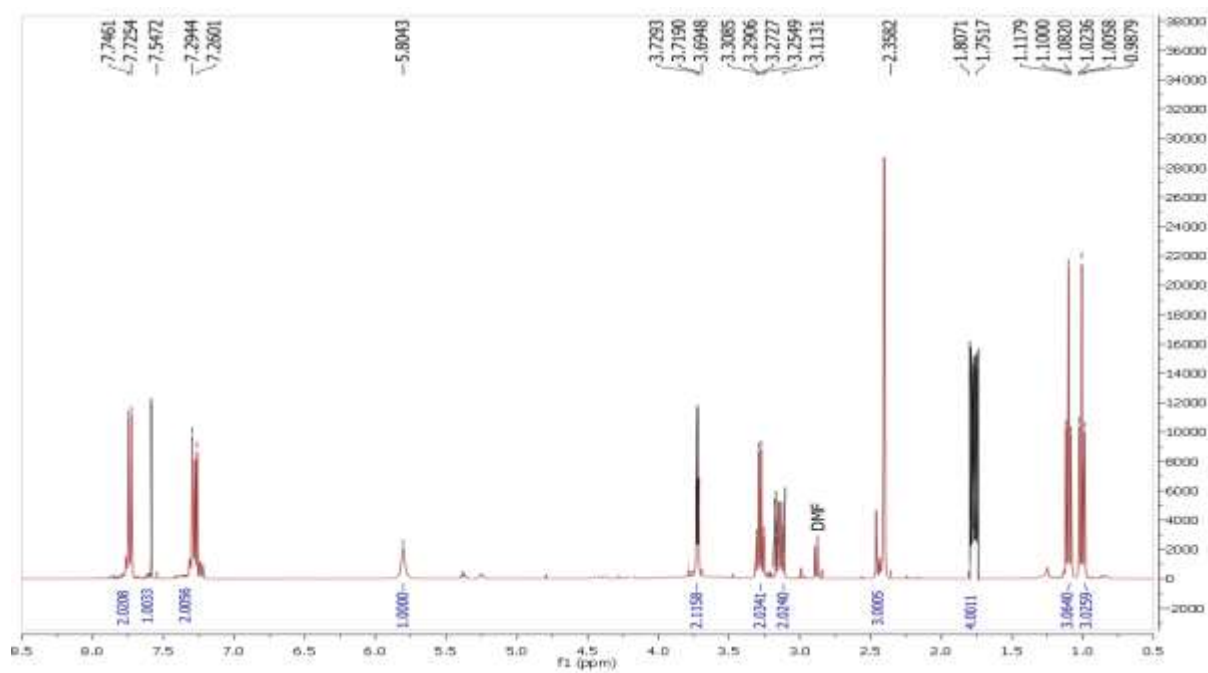


Figure S7. ¹H NMR spectrum of *N'*-(3-oxoheptan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4g**

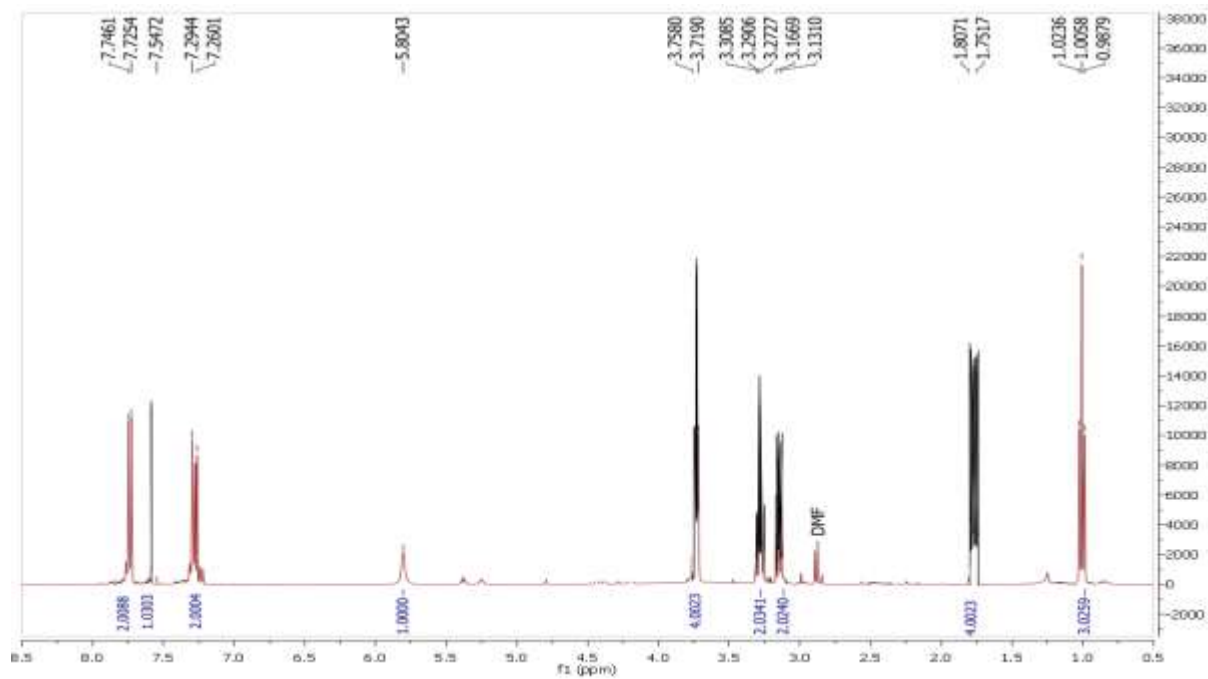


Figure S8. ¹H NMR spectrum of *N'*-cyclopentylidene-2-propylquinoline-4-carbohydrazide, **4h**

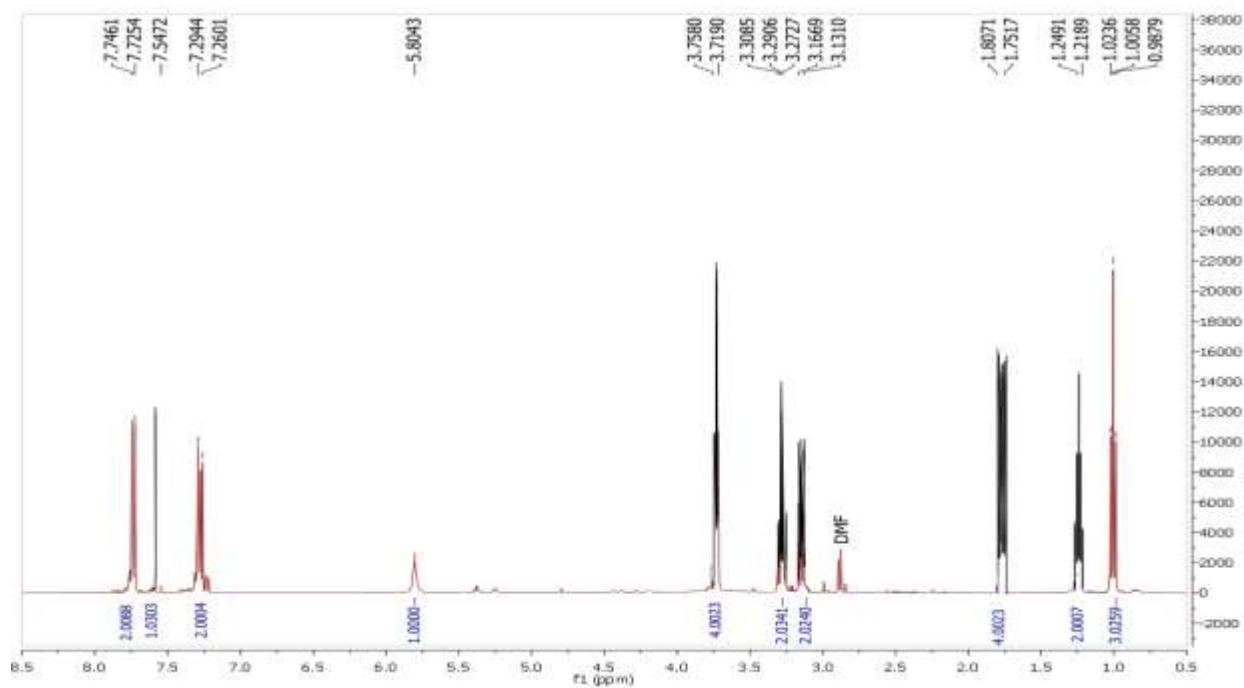


Figure S9. ^1H NMR spectrum of *N'*-cyclohexylidene-2-propylquinoline-4-carbohydrazide, **4i**

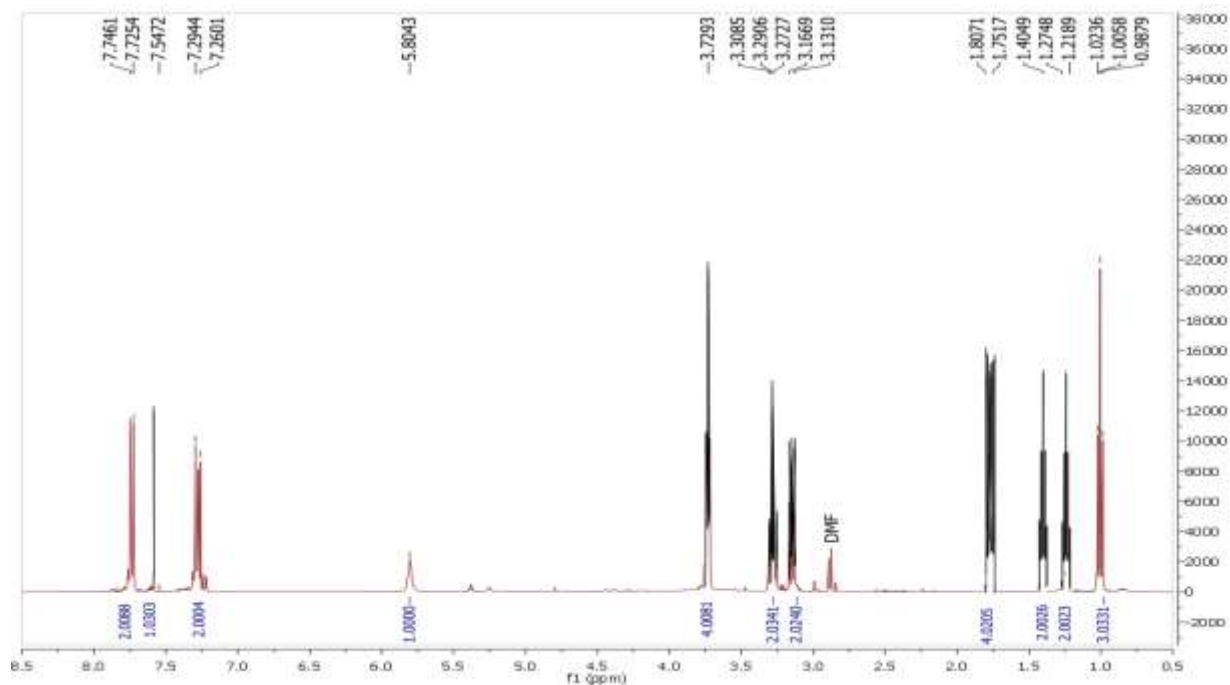


Figure S10. ^1H NMR spectrum of *N'*-cycloheptylidene-2-propylquinoline-4-carbohydrazide, **4j**

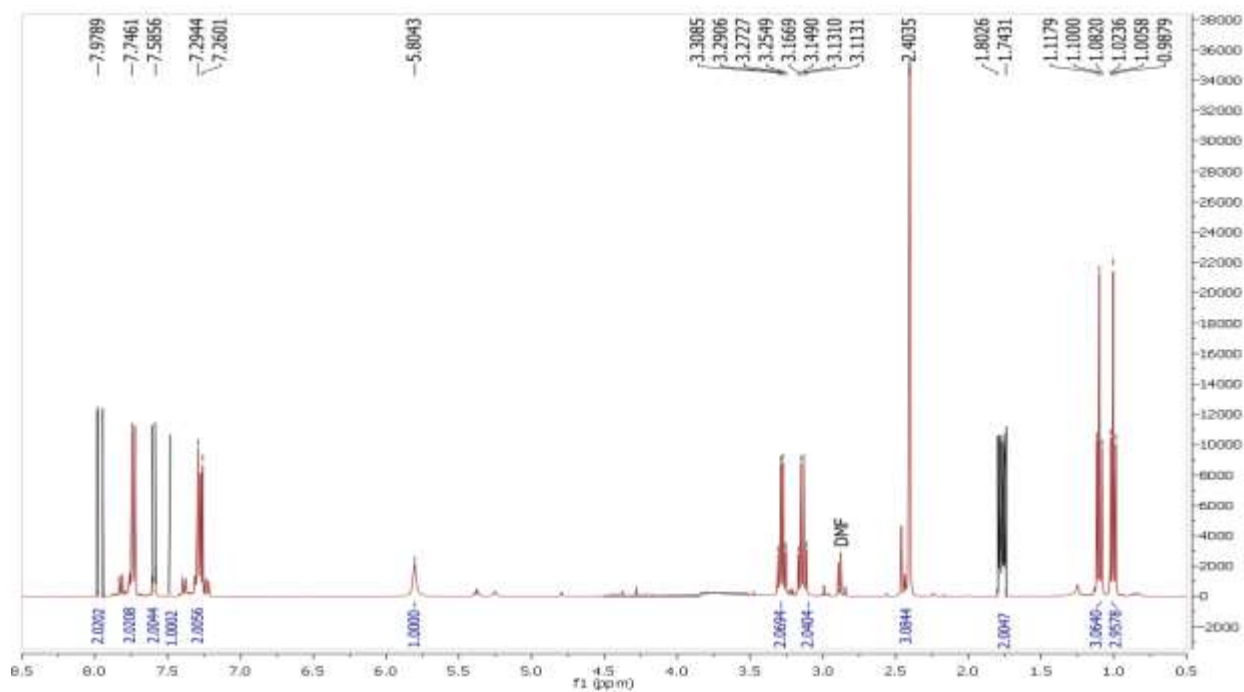


Figure S11. ¹H NMR spectrum of *N'*-(1-(4-ethylphenyl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4k**

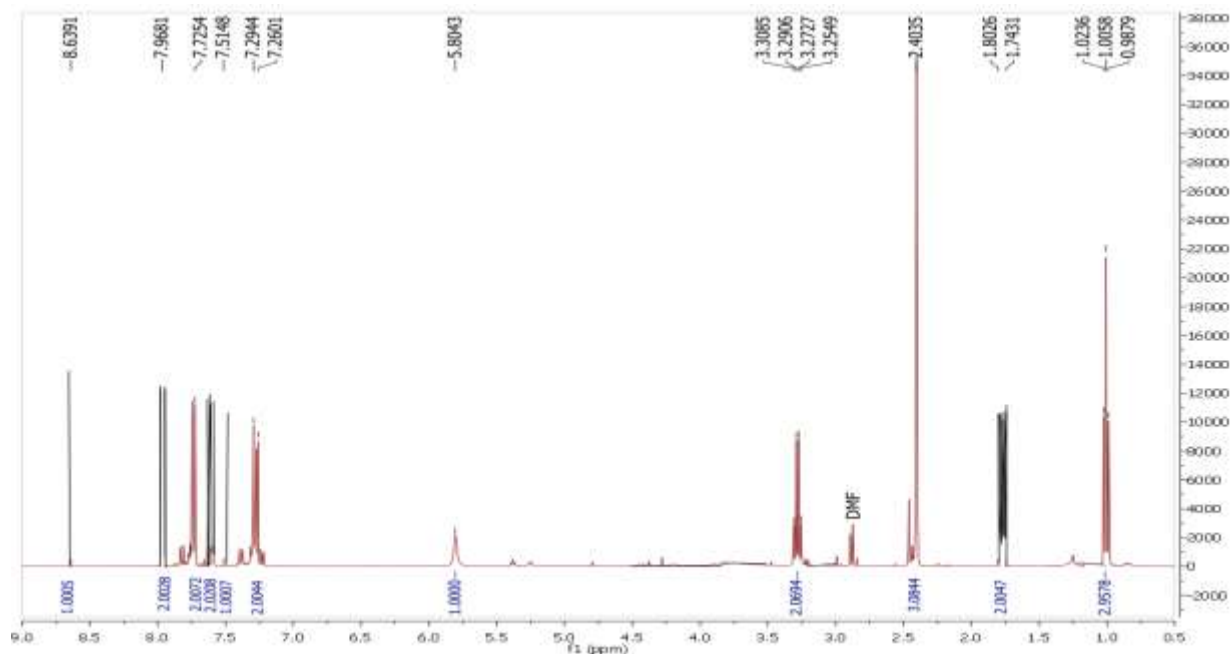


Figure S12. ¹H NMR spectrum of *N'*-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4l**

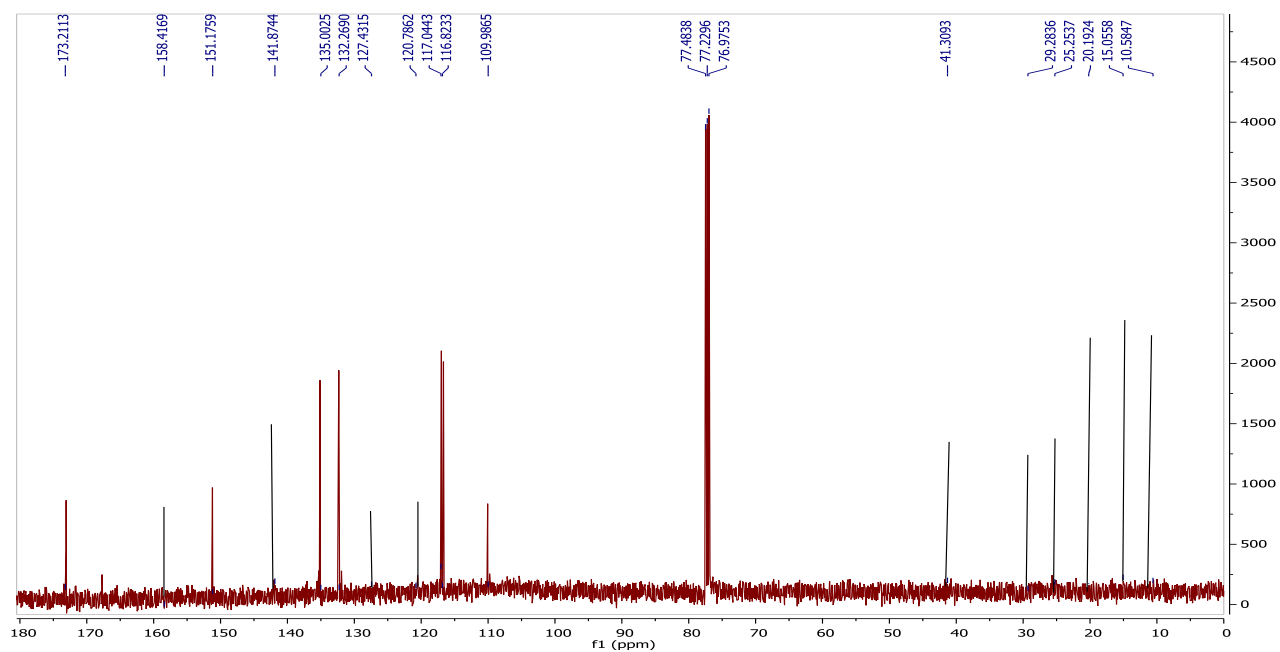


Figure S13. ^{13}C NMR spectrum of N' -(butan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4a**

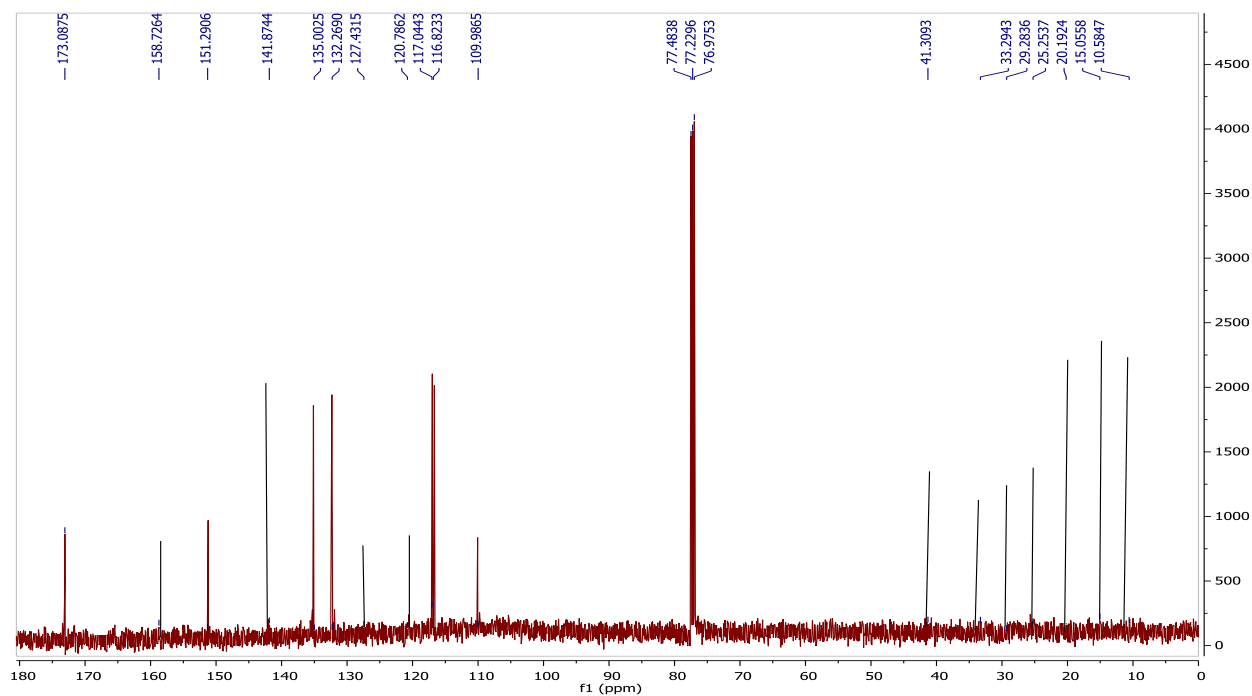


Figure S14. ^{13}C NMR spectrum of N' -(pentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4b**.

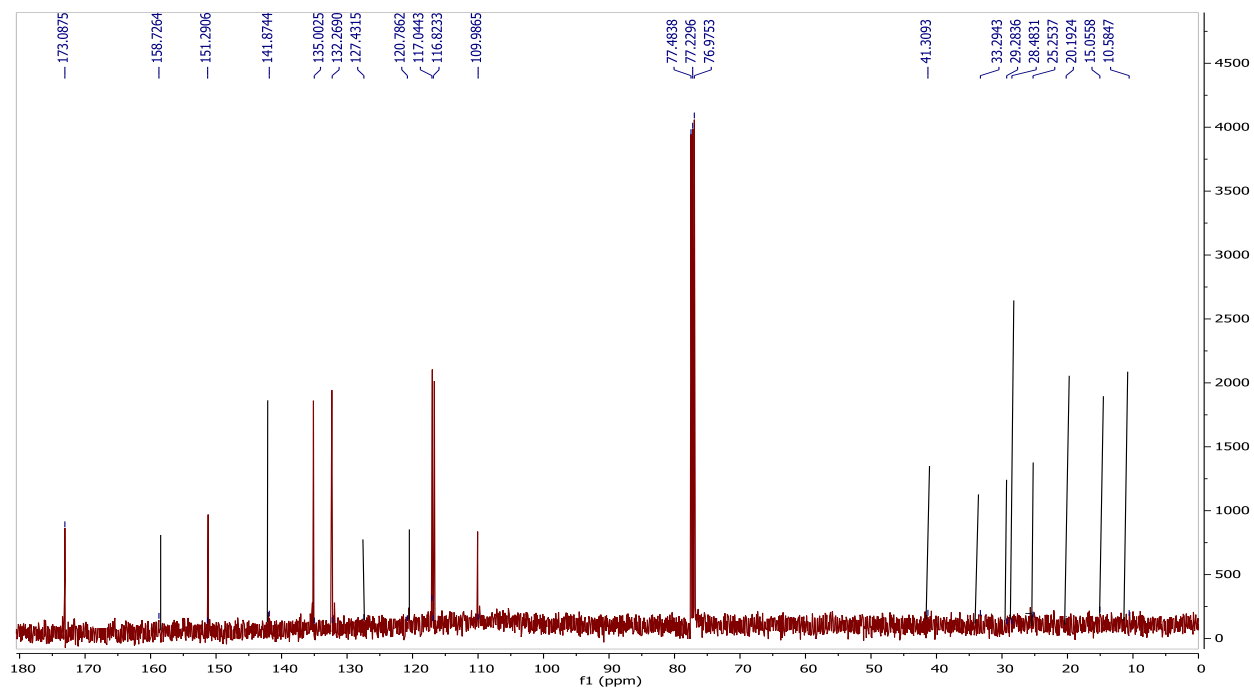


Figure S15. ^{13}C NMR spectrum of N' -(hepta-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4c**

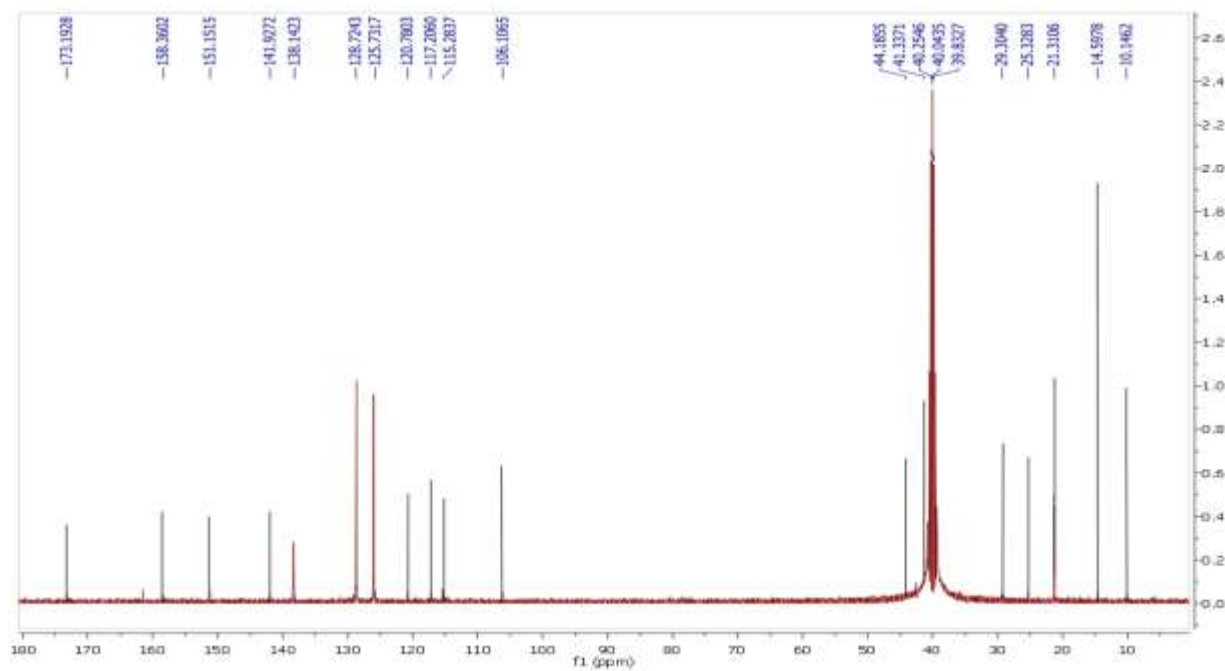


Figure S16. ^{13}C NMR spectrum of N' -(4-Methylpentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4d**

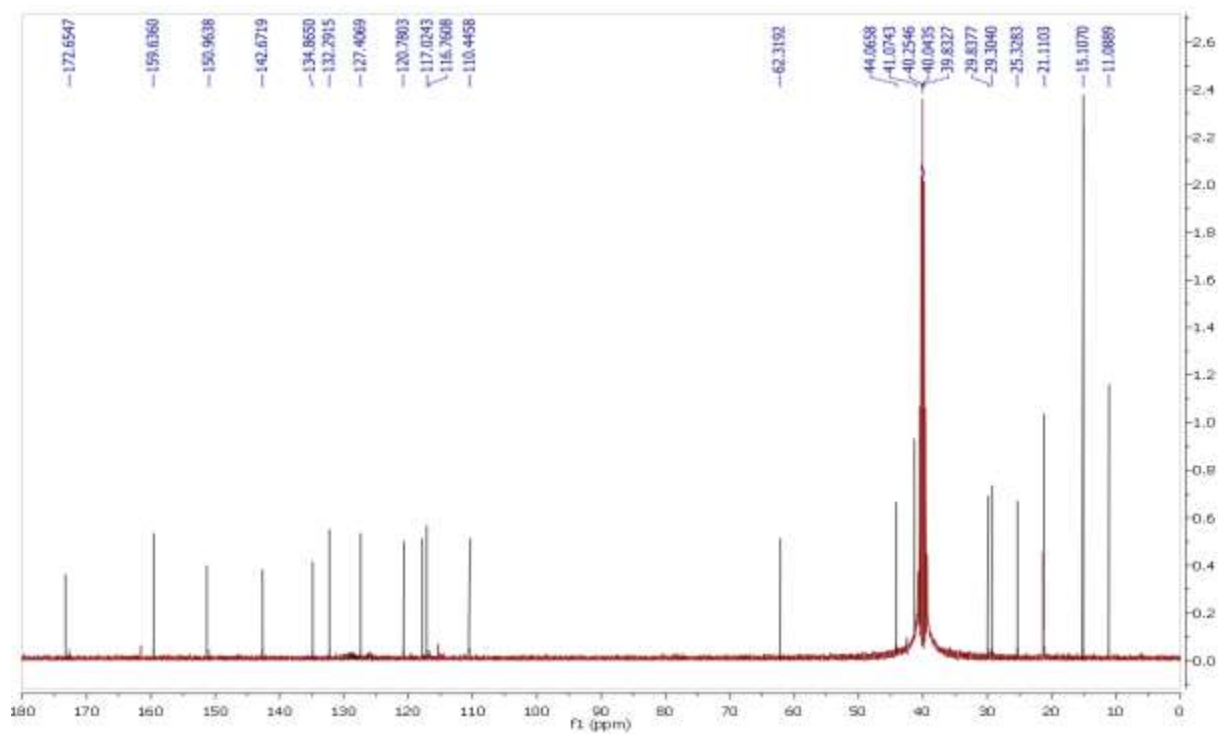


Figure S17. ^{13}C NMR spectrum of 2-propyl-(N' -(1,7,7-trimethylbicyclo[2.2.1]heptan-2-ylidene)quinoline-4-carbohydrazide, **4e**

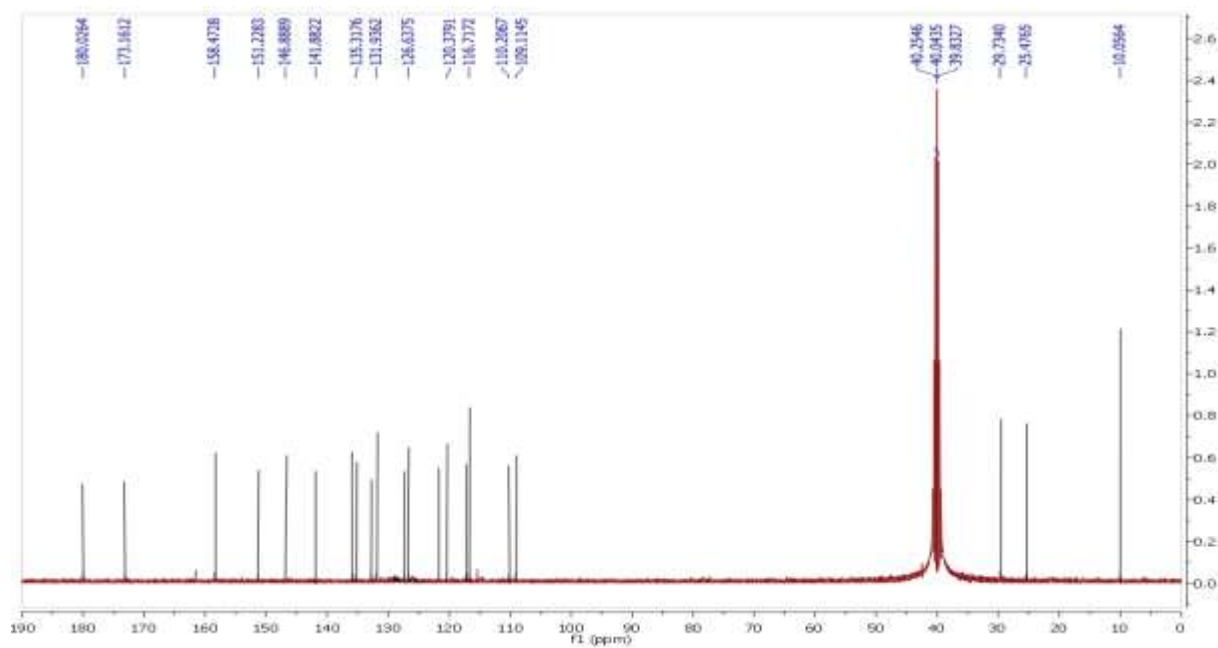


Figure S18. ^{13}C NMR spectrum of N' -(2-oxoindolin-3-ylidene)-2-propylquinoline-4-carbohydrazide, **4f**

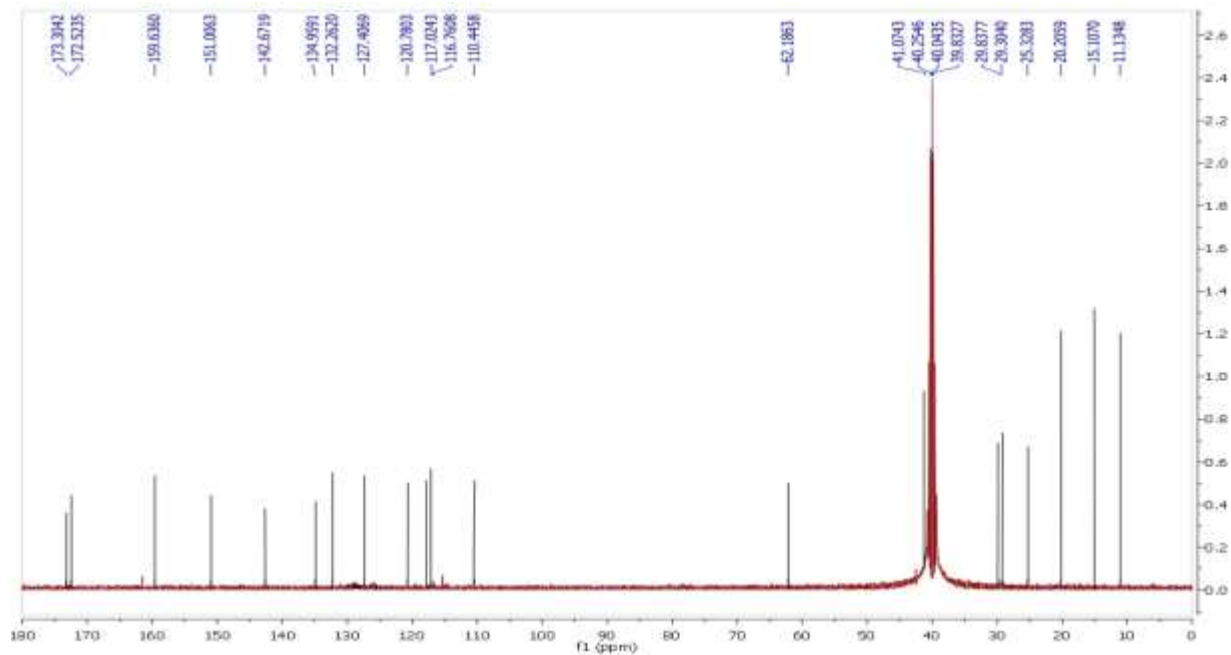


Figure S19. ^{13}C NMR spectrum of *N'*-(3-oxoheptan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4g**

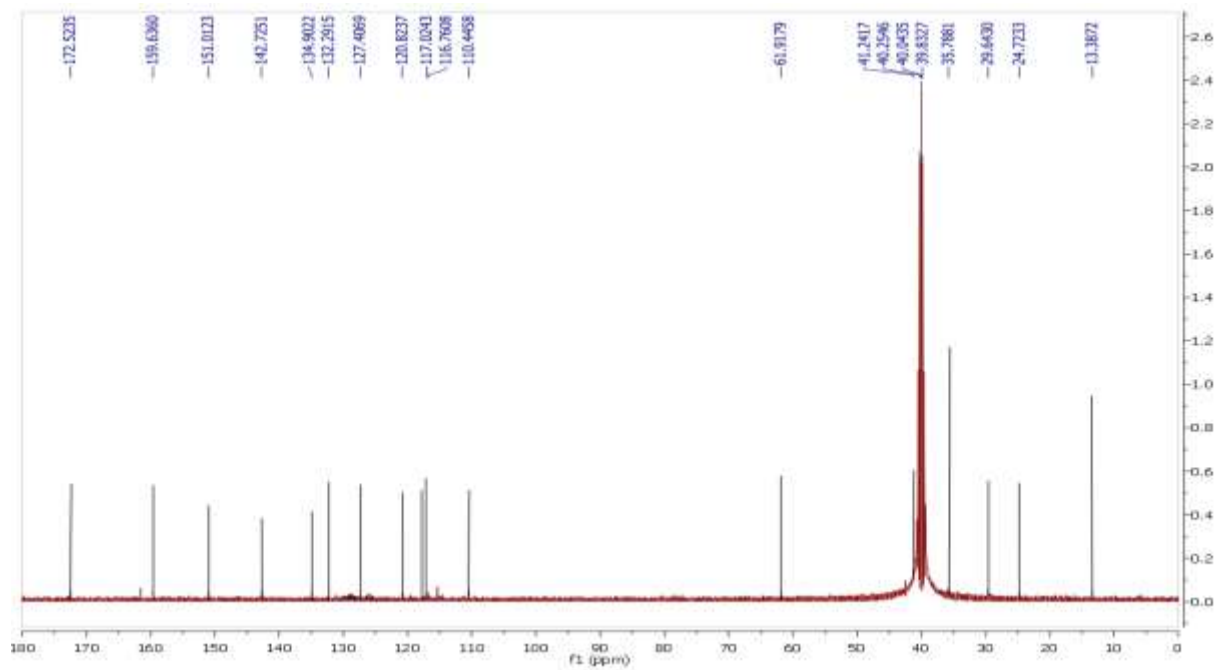


Figure S20. ^{13}C NMR spectrum of *N'*-cyclopentylidene-2-propylquinoline-4-carbohydrazide, **4h**

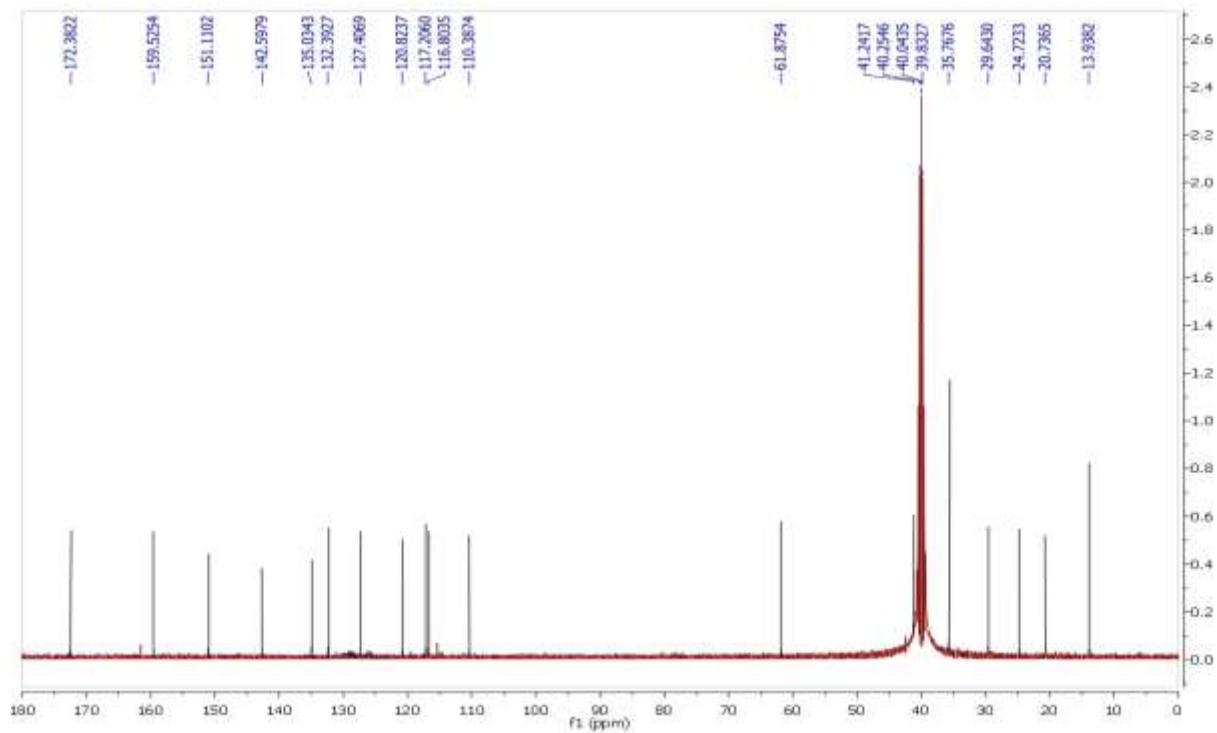


Figure S21. ^{13}C NMR spectrum of *N'*-cyclohexylidene-2-propylquinoline-4-carbohydrazide, **4i**

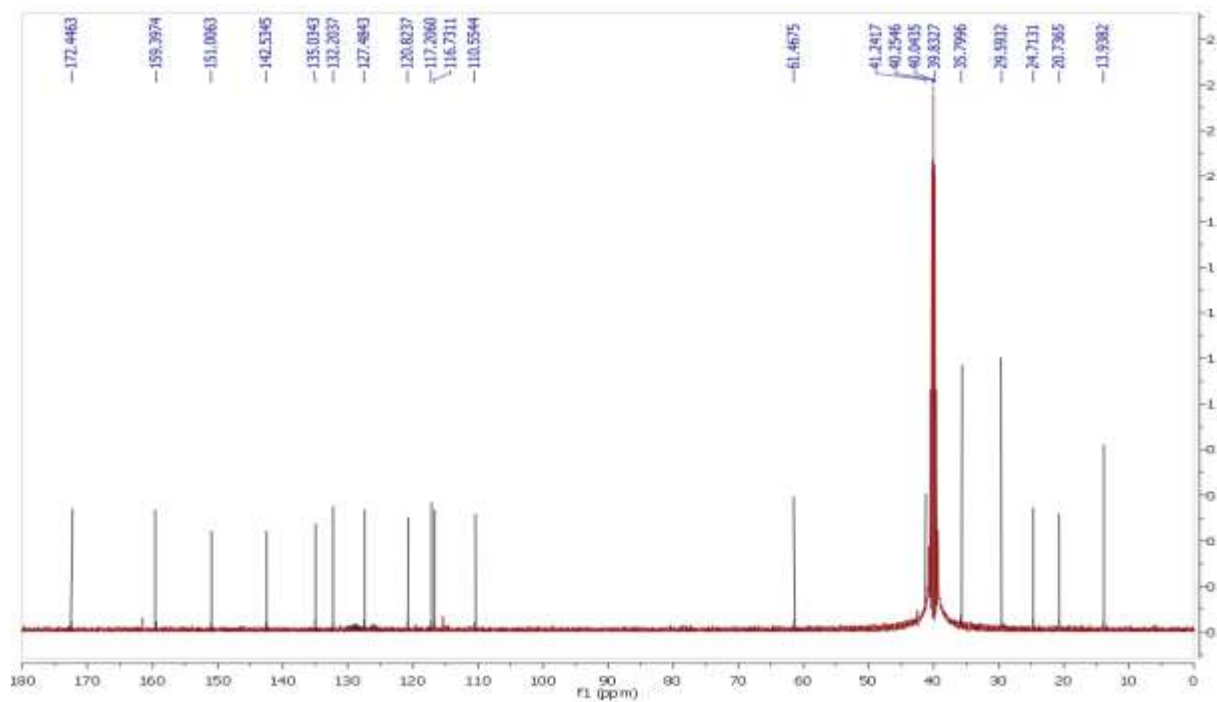


Figure S22. ^{13}C NMR spectrum of *N'*-cycloheptylidene-2-propylquinoline-4-carbohydrazide, **4j**

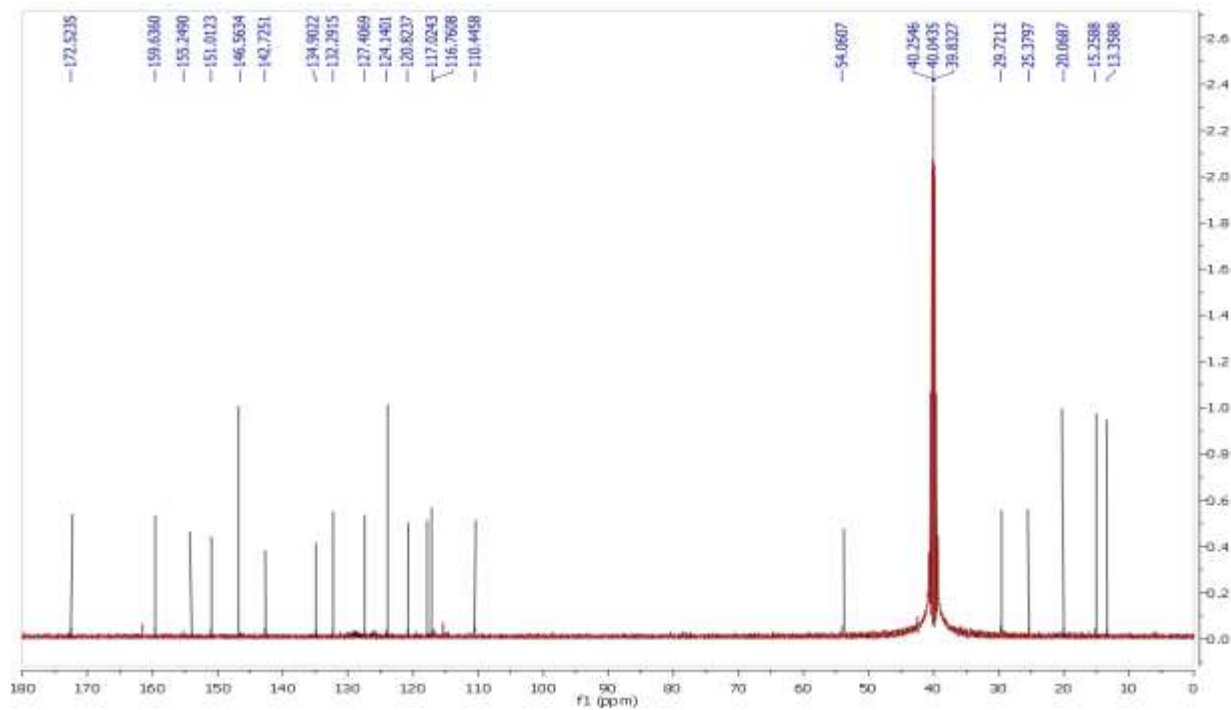


Figure S23. ^{13}C NMR spectrum of *N'*-(1-(4-ethylphenyl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4k**

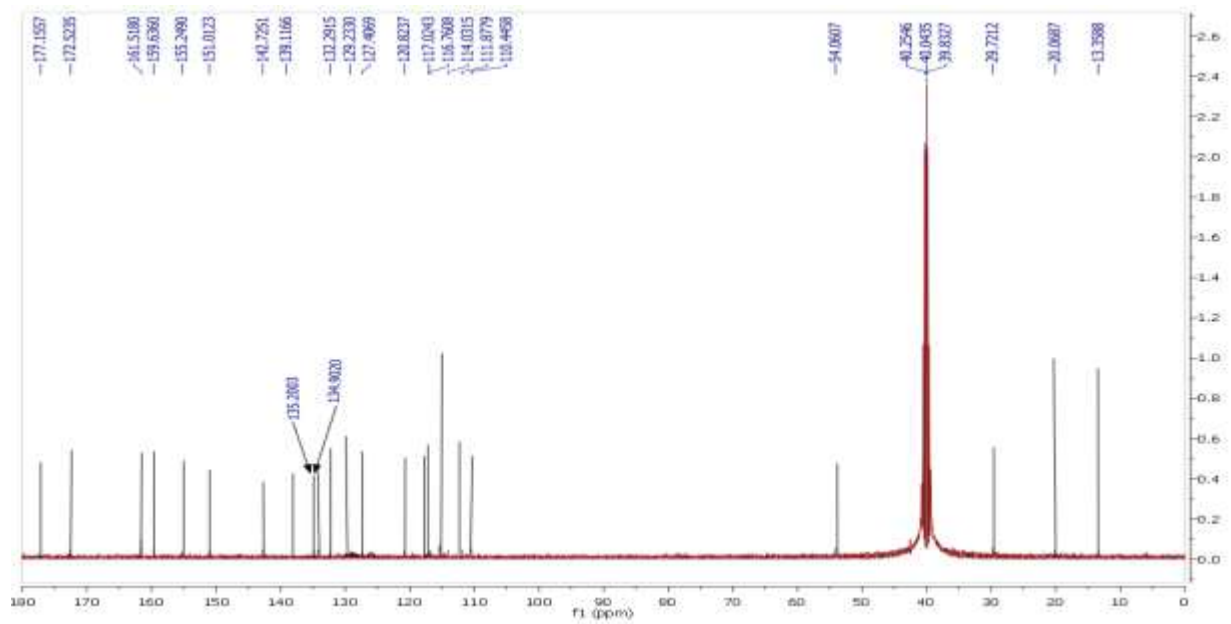


Figure S24. ^{13}C NMR spectrum of *N'*-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4l**

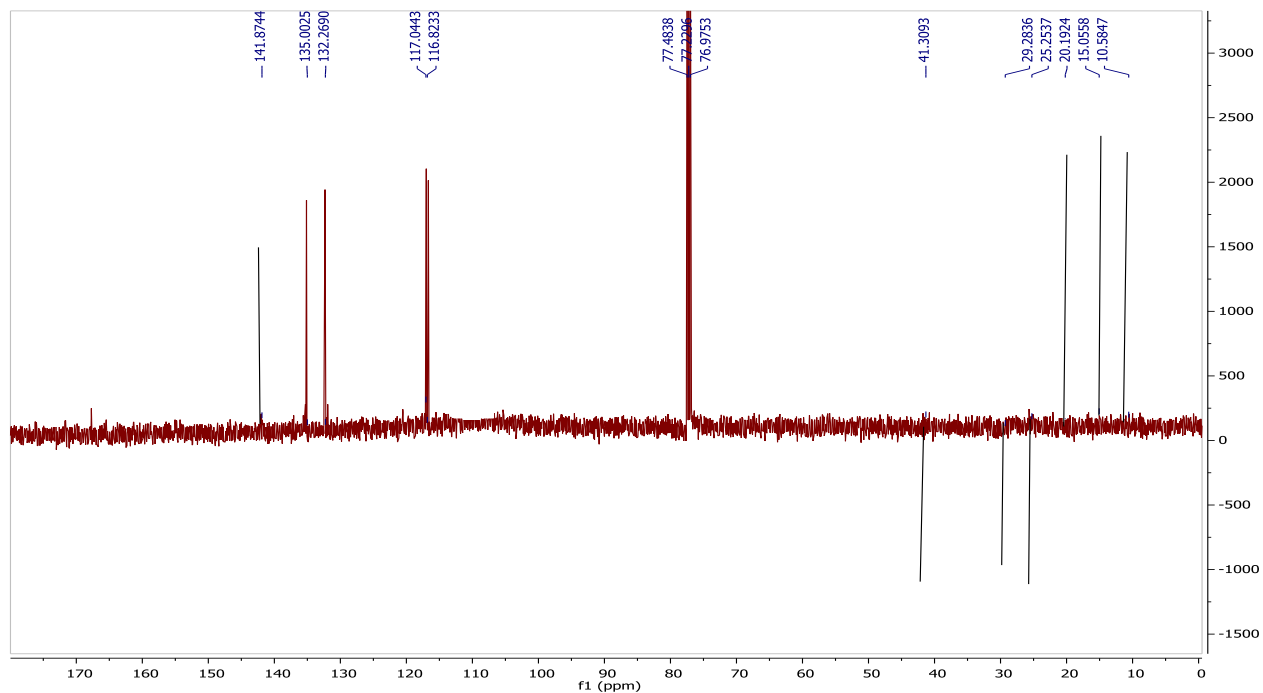


Figure S25. DEPT 135 spectrum of *N'*-(butan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4a**

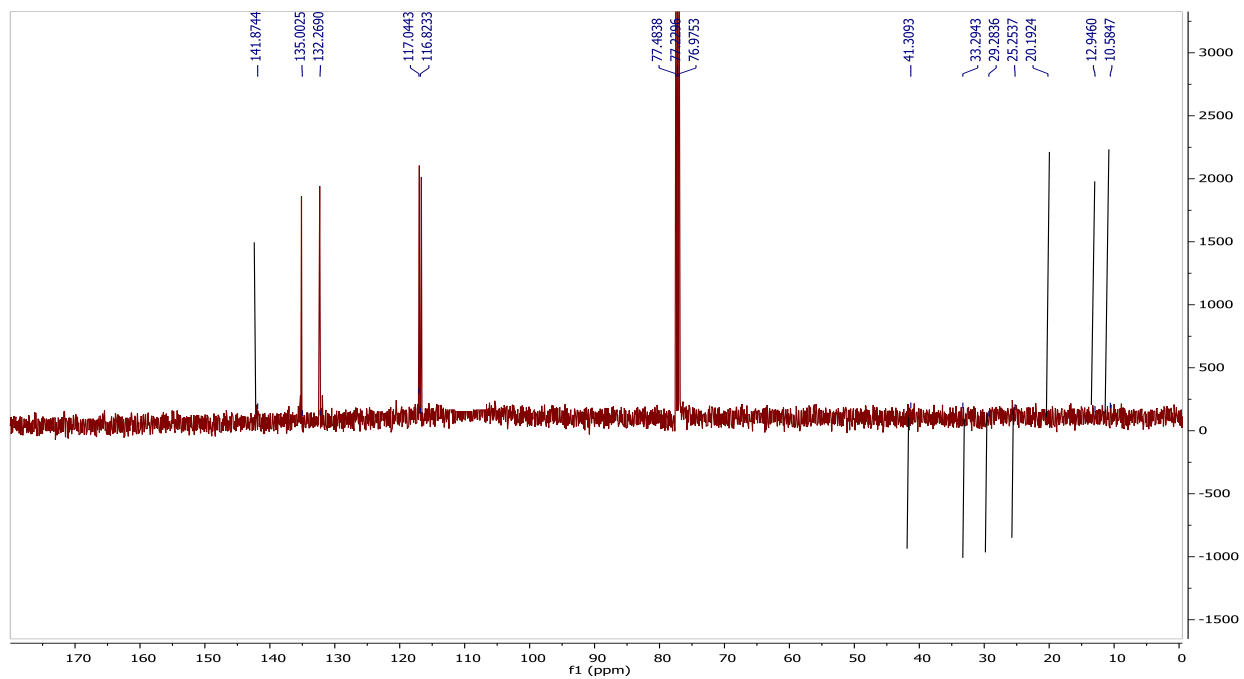


Figure S26. DEPT 135 spectrum of *N'*-(pentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4b**.

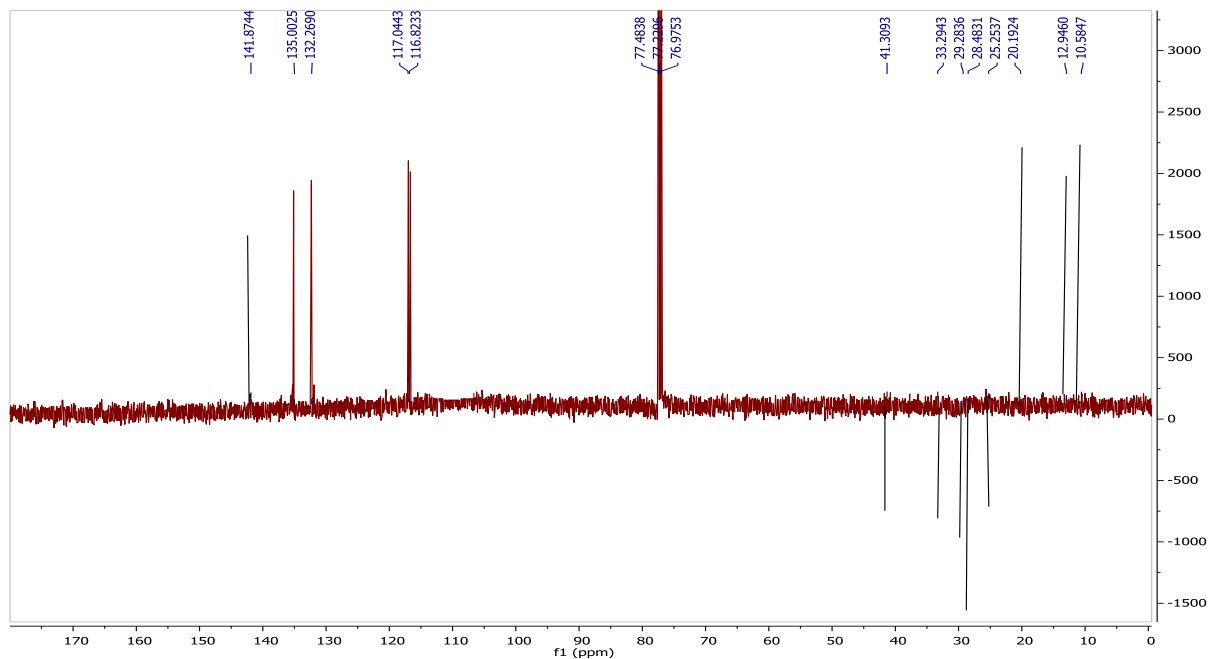


Figure S27. DEPT 135 spectrum of *N'*-(hepta-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4c**

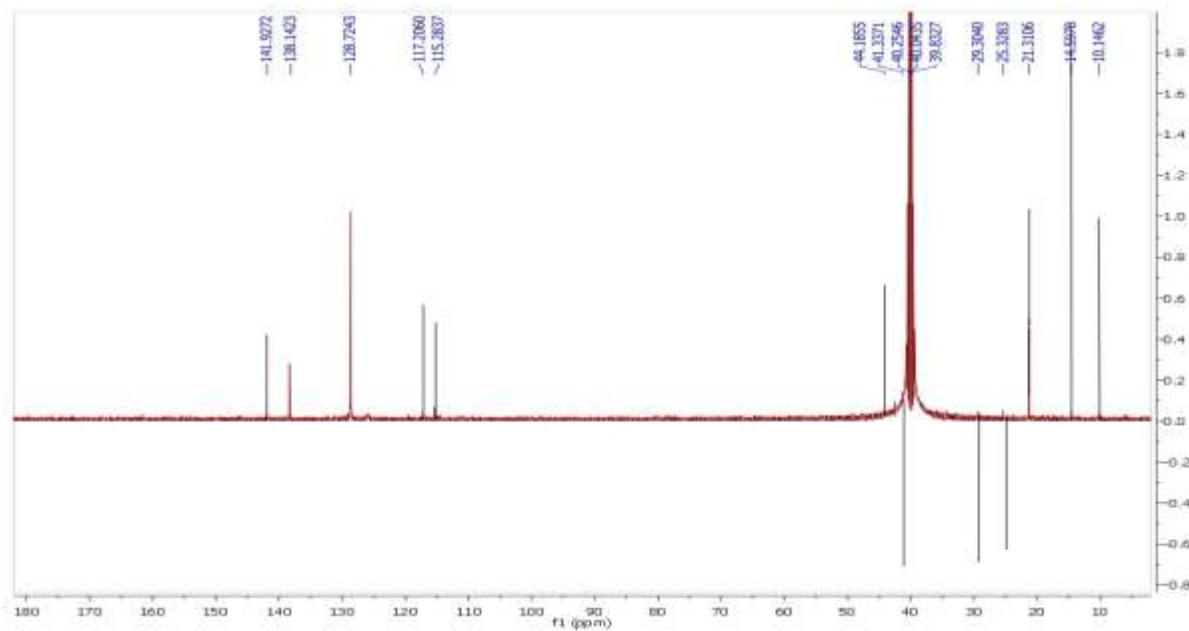


Figure S28. DEPT 135 spectrum of *N'*-(4-methylpentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4d**

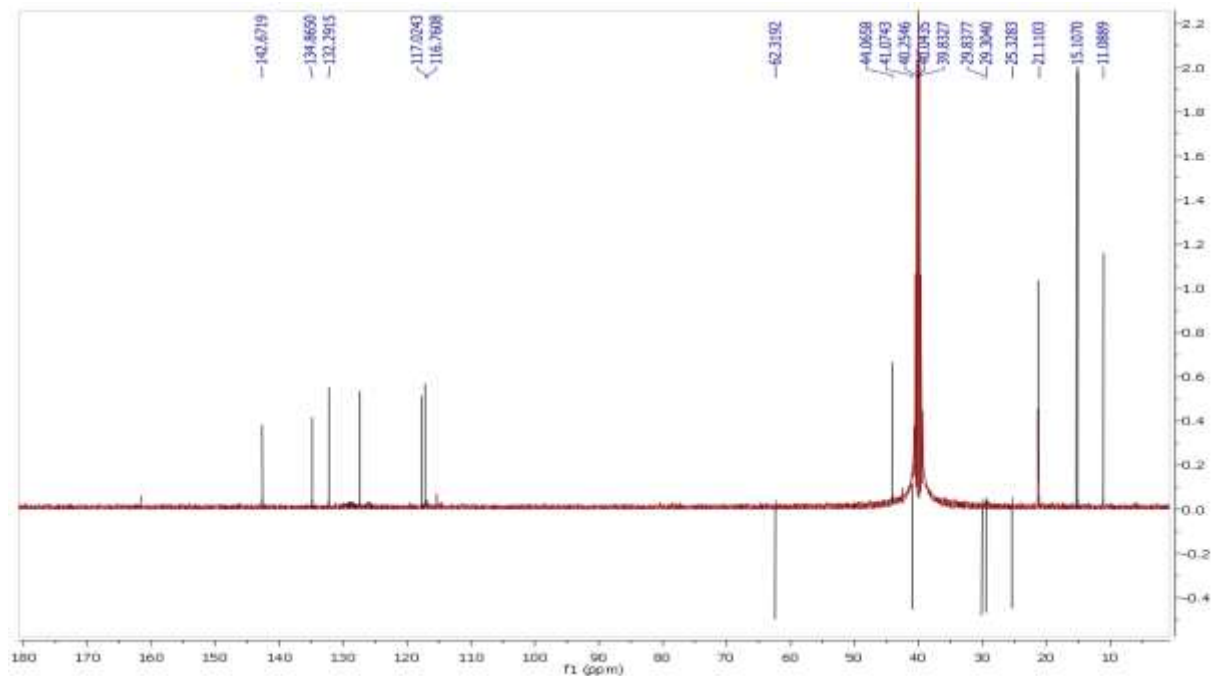


Figure S29. DEPT 135 spectrum of 2-propyl-(*N'*-(1,7,7-trimethylbicyclo[2.2.1]heptan-2-ylidene)quinoline-4-carbohydrazide, **4e**

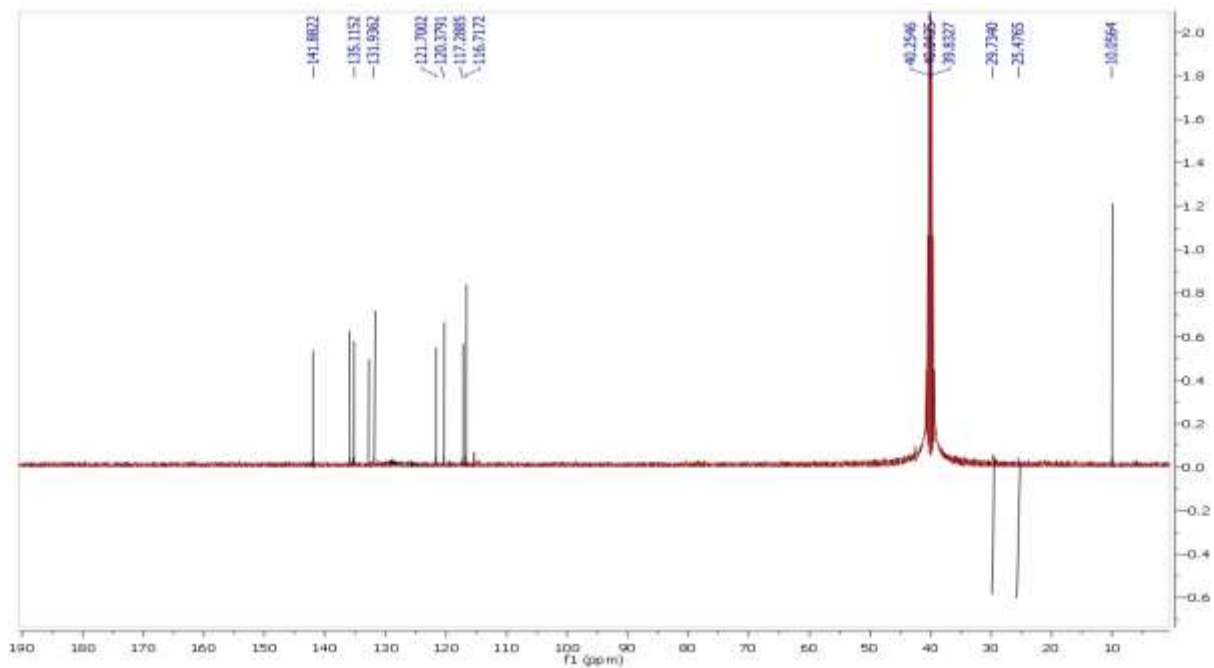


Figure S30. DEPT 135 spectrum of *N'*-(2-oxoindolin-3-ylidene)-2-propylquinoline-4-carbohydrazide, **4f**

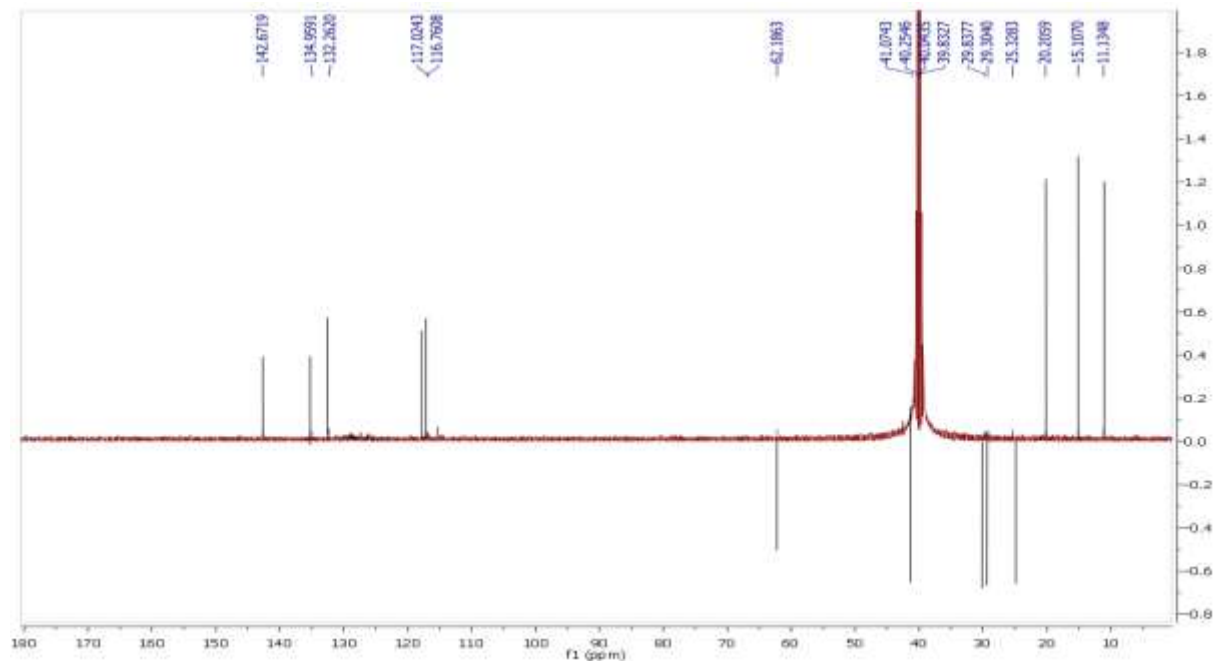


Figure S31. DEPT 135 spectrum of *N'*-(3-oxoheptan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4g**

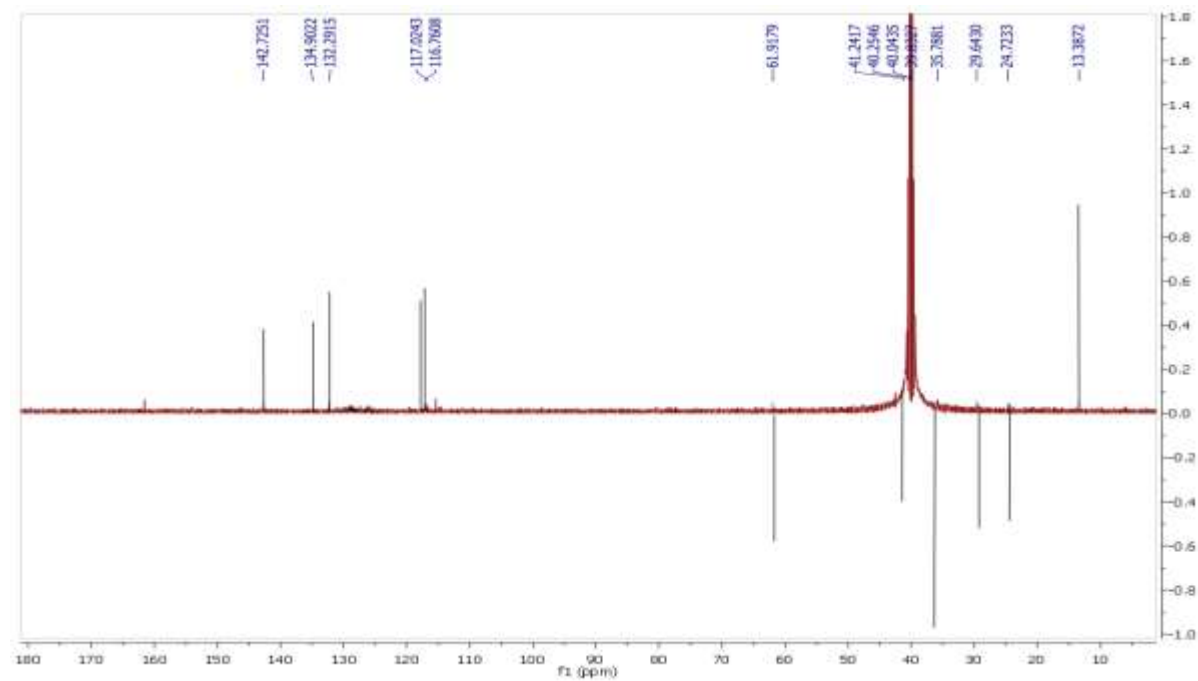


Figure S32. DEPT 135 spectrum of *N'*-cyclopentylidene-2-propylquinoline-4-carbohydrazide, **4h**

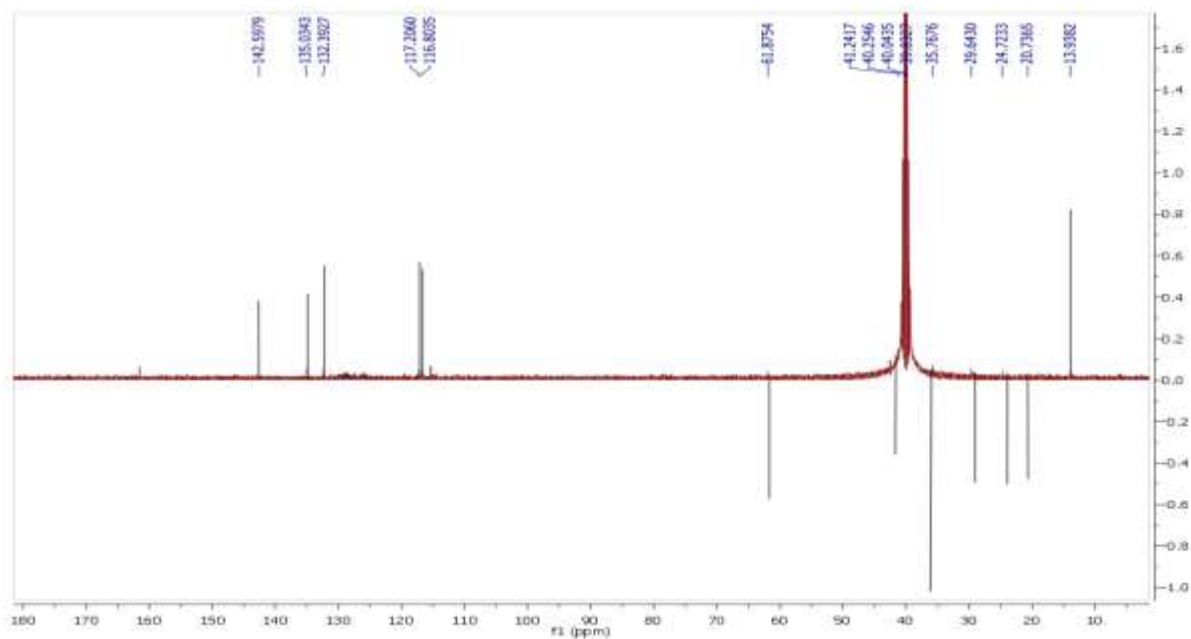


Figure S33. DEPT 135 spectrum of *N'*-cyclohexylidene-2-propylquinoline-4-carbohydrazide, **4i**

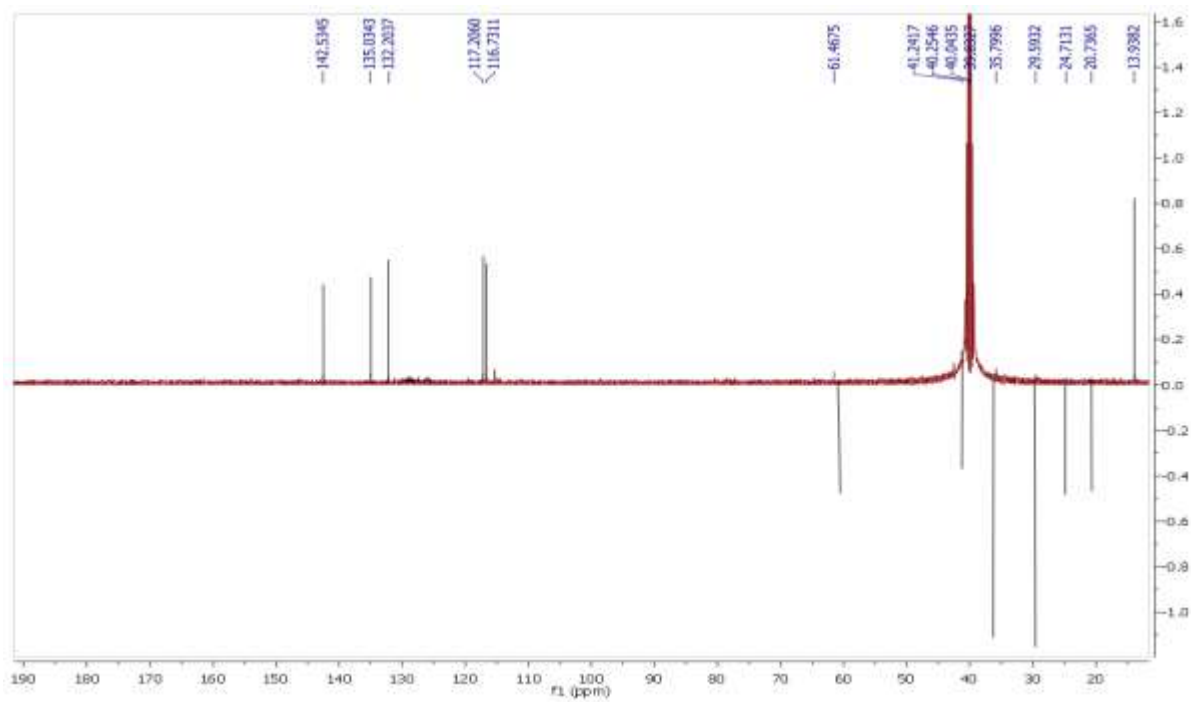


Figure S34. DEPT 135 spectrum of *N'*-cycloheptylidene-2-propylquinoline-4-carbohydrazide, **4j**

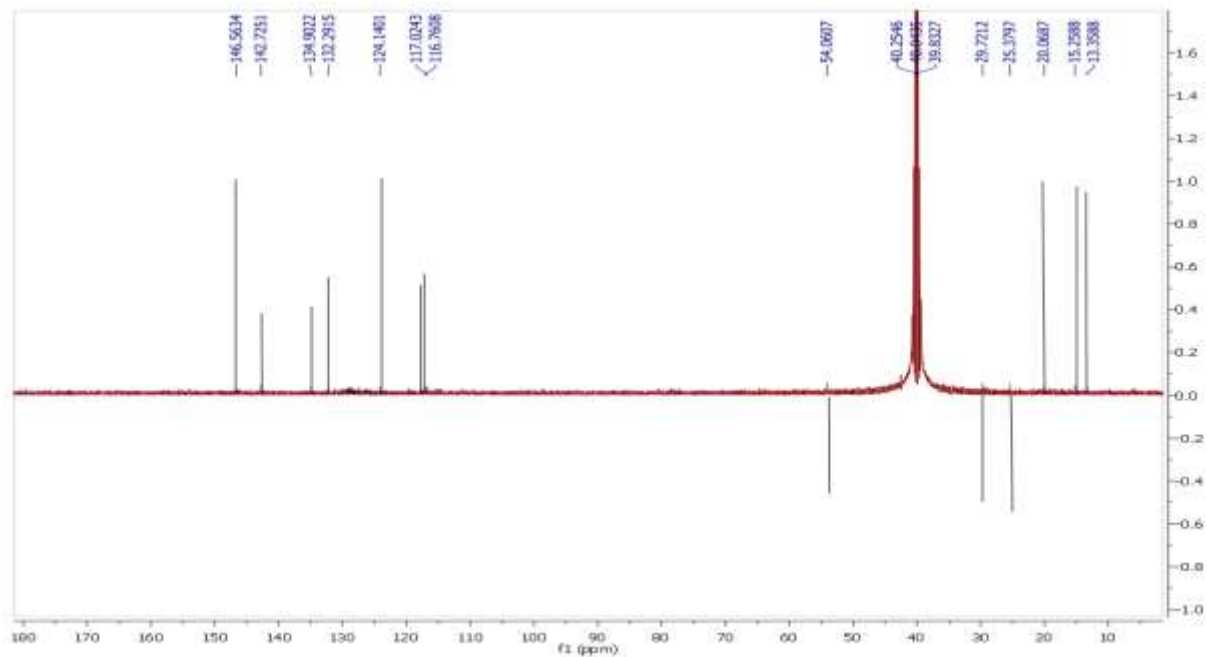


Figure S35. DEPT 135 spectrum of *N'*-(1-(4-ethylphenyl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4k**

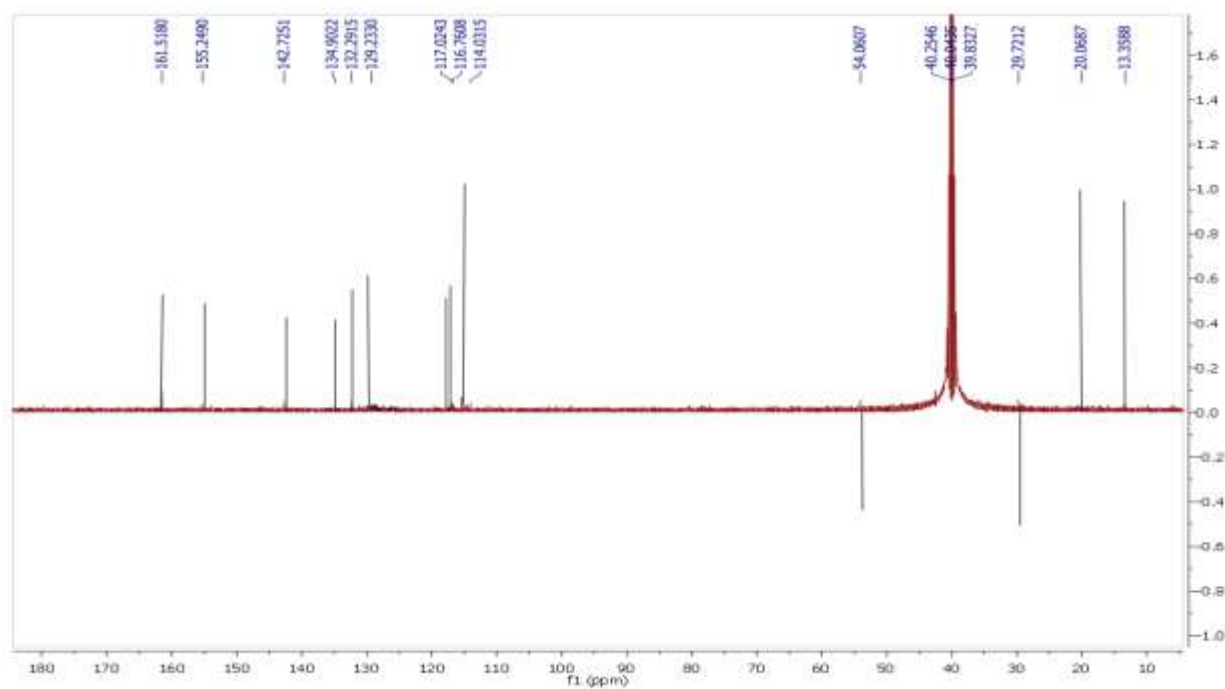


Figure S36. DEPT 135 spectrum of *N'*-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4l**

TEST SETUP
 GENESYS 10S v1.200 2L7J355002

Scanning 9:21 26Feb16
 Test Name QH BUTANON [Saved]
 Measurement Mode Absorbance
 Start Wavelength 200nm
 Stop Wavelength 600nm
 Sample Positioner 1-Cell Platform
 Scan Speed Fast
 ID# (0=OFF) Off
 Auto Print Off

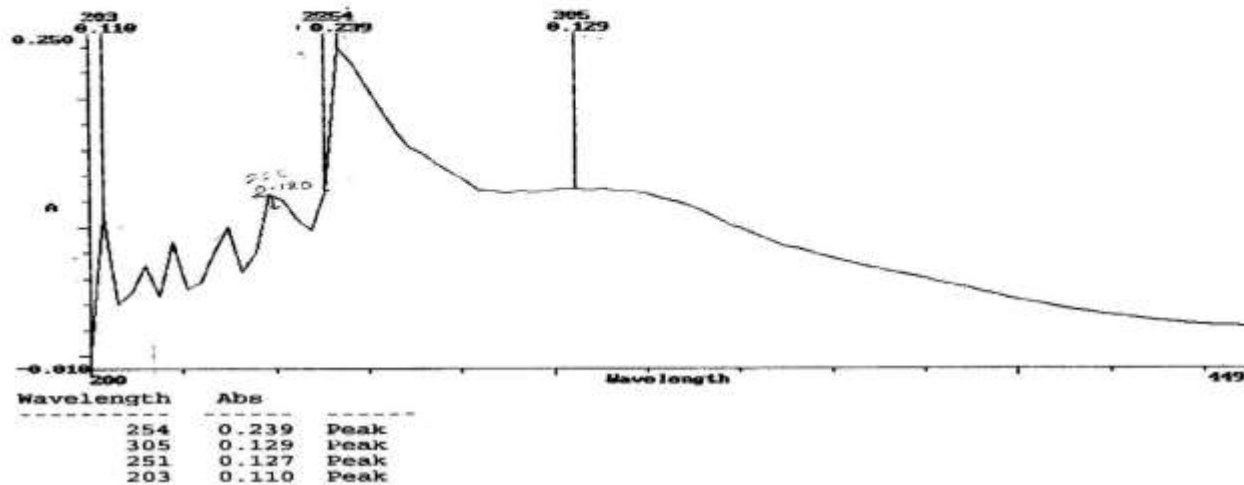


Figure S37. UV-Visible spectrum of *N'*-(butan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4a**

TEST SETUP
 GENESYS 10S v1.200 2L7J355002

Scanning 9:49 26Feb16
 Test Name QH 2_P [Saved]
 Measurement Mode Absorbance
 Start Wavelength 200nm
 Stop Wavelength 600nm
 Sample Positioner 1-Cell Platform
 Scan Speed Fast
 ID# (0=OFF) Off
 Auto Print Off

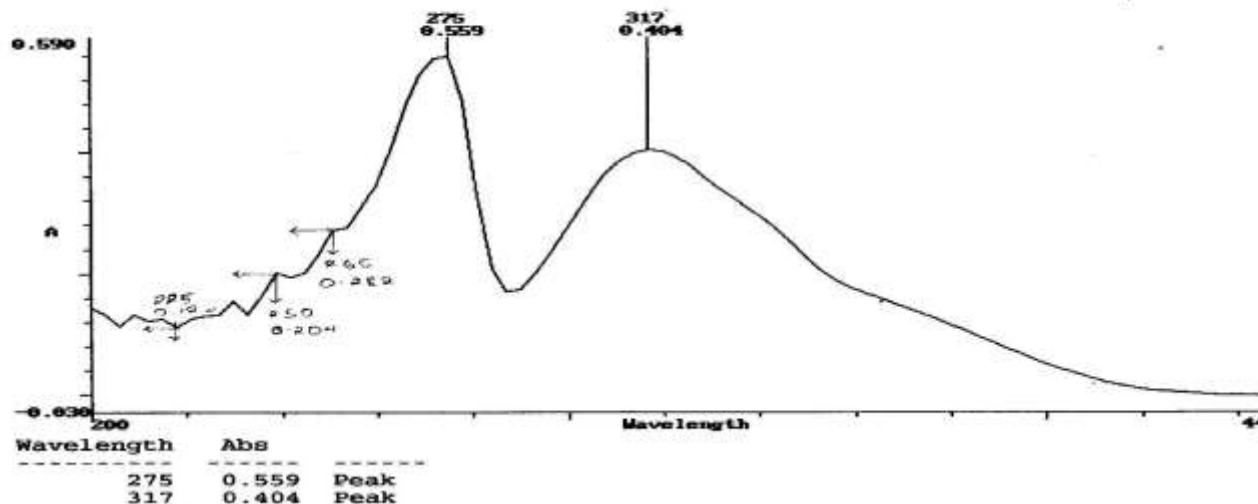


Figure S38. UV-Visible spectrum of *N'*-(pentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4b**.

TEST SETUP
 GENESYS 10S v1.200 2L7J355002

Scanning 9:53 26Feb16
 Test Name QH 2_H[Saved]
 Measurement Mode Absorbance
 Start Wavelength 200nm
 Stop Wavelength 600nm
 Sample Positioner 1-Cell Platform
 Scan Speed Fast
 ID# (0=OFF) Off
 Auto Print Off

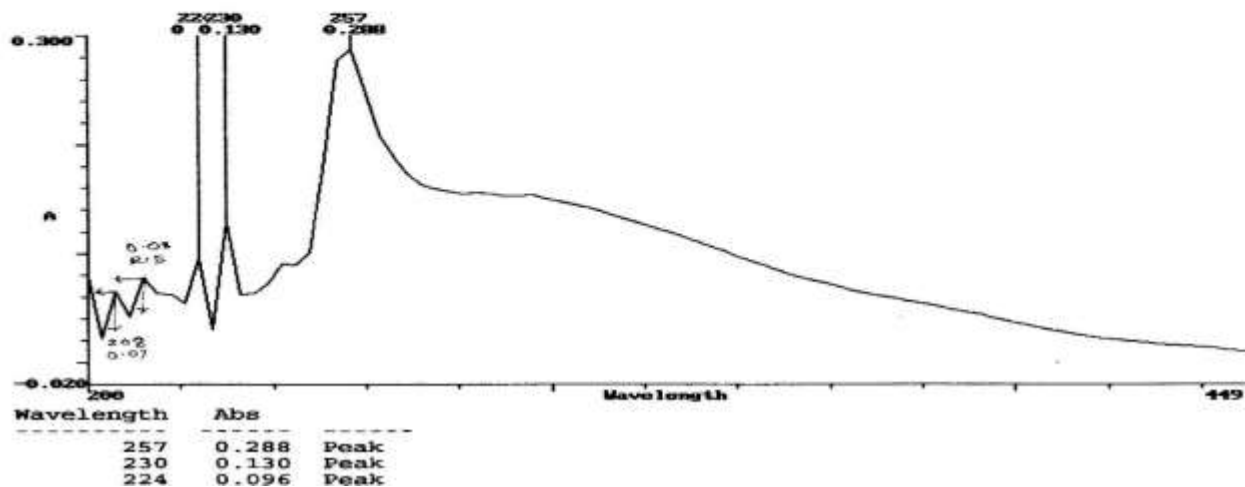


Figure S39. UV-Visible spectrum of *N'*-(hepta-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4c**

TEST SETUP
 GENESYS 10S v1.200 2L7J355002

Scanning 10:18 26Feb16
 Test Name QH 4_M_2_P[Saved]
 Measurement Mode Absorbance
 Start Wavelength 200nm
 Stop Wavelength 600nm
 Sample Positioner 1-Cell Platform
 Scan Speed Fast
 ID# (0=OFF) Off
 Auto Print Off

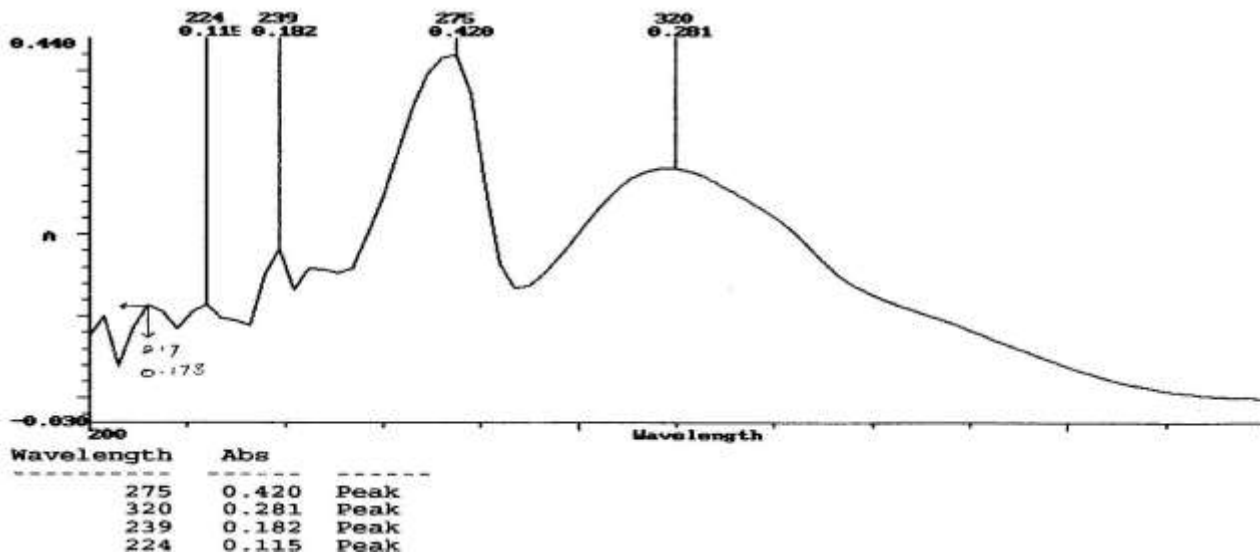


Figure S40. UV-Visible spectrum of *N'*-(4-methylpentan-2-ylidene)-2-propyl quinoline-4-carbohydrazide, **4d**

TEST SETUP
GENESYS 10S v1.200 2L7J355002

Scanning 14:45 29Feb16
Test Name QH Ca [Saved]
Measurement Mode Absorbance
Start Wavelength 200nm
Stop Wavelength 600nm
Sample Positioner 1-Cell Platform
Scan Speed Fast
ID# (0-OFF) Off
Auto Print Off

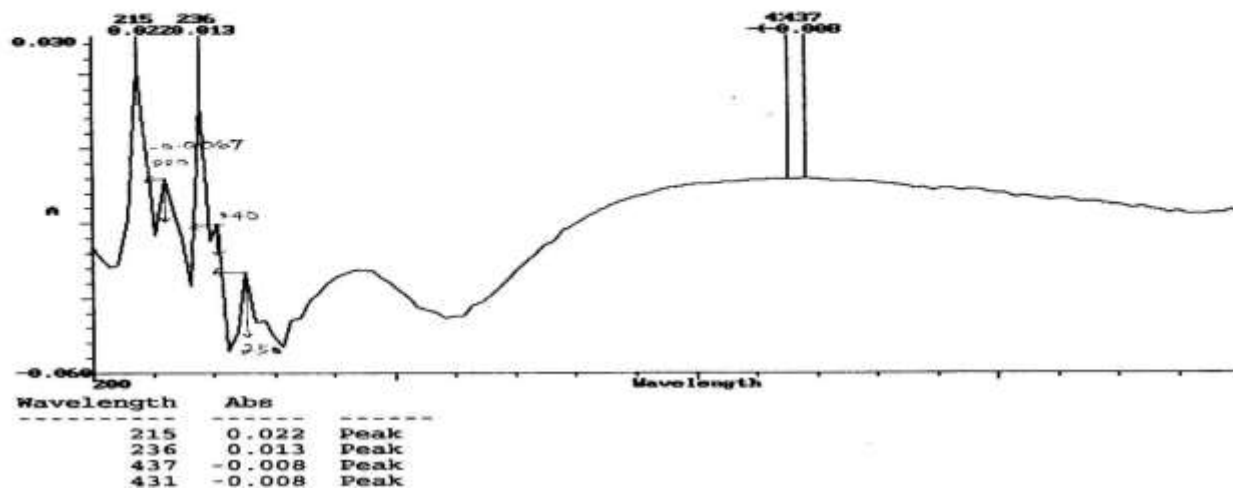


Figure S41. UV-Visible spectrum of 2-propyl-(*N'*-(1,7,7-trimethylbicyclo[2.2.1]heptan-2-ylidene)quinoline-4-carbohydrazide, **4e**

TEST SETUP
GENESYS 10S v1.200 2L7J355002

Scanning 10:27 26Feb16
Test Name QH 2S_H_D [Saved]
Measurement Mode Absorbance
Start Wavelength 200nm
Stop Wavelength 600nm
Sample Positioner 1-Cell Platform
Scan Speed Fast
ID# (0-OFF) Off
Auto Print Off

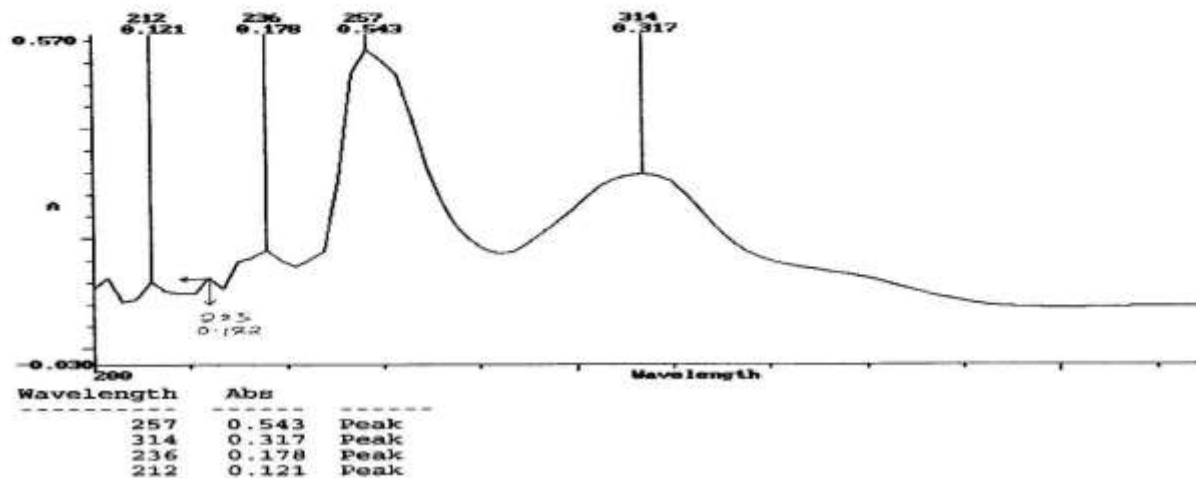


Figure S42. UV-Visible spectrum of *N'*-(2-oxoindolin-3-ylidene)-2-propylquinoline-4-carbohydrazide, **4f**

TEST SETUP
GENSYS 10S v1.200 2L7J355002

Scanning 10:46 26Feb16
Test Name QH 2 3_HD[Saved]
Measurement Mode Absorbance
Start Wavelength 200nm
Stop Wavelength 600nm
Sample Positioner 1-Cell Platform
Scan Speed Fast
ID# (0=OFF) Off
Auto Print Off

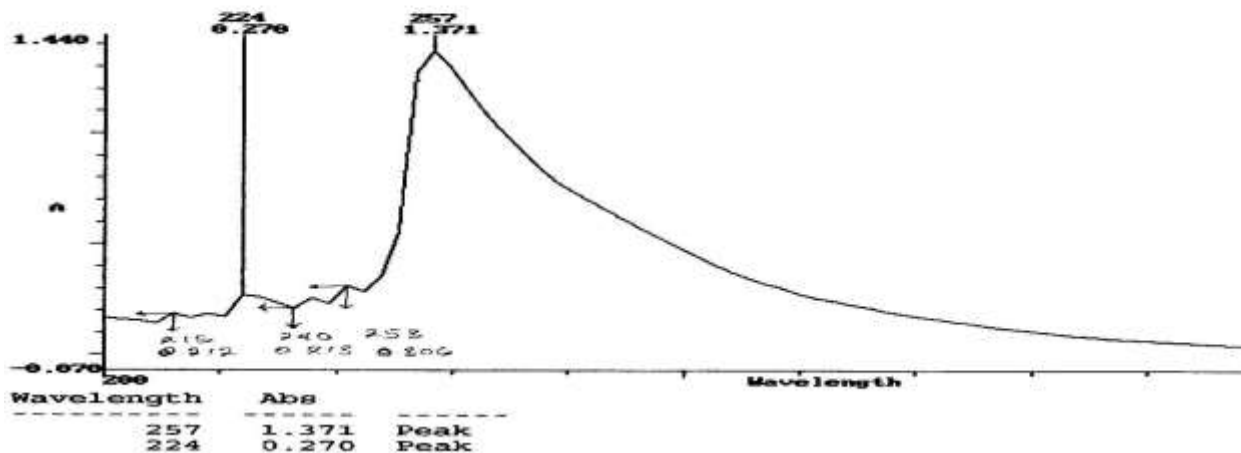


Figure S43. UV-Visible spectrum of *N'*-(3-oxoheptan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4g**

Scanning
Test Name QH Cy_P[Saved]
Measurement Mode Absorbance
Start Wavelength 200nm
Stop Wavelength 600nm
Sample Positioner 1-Cell Platform
Scan Speed Fast
ID# (0=OFF) Off
Auto Print Off

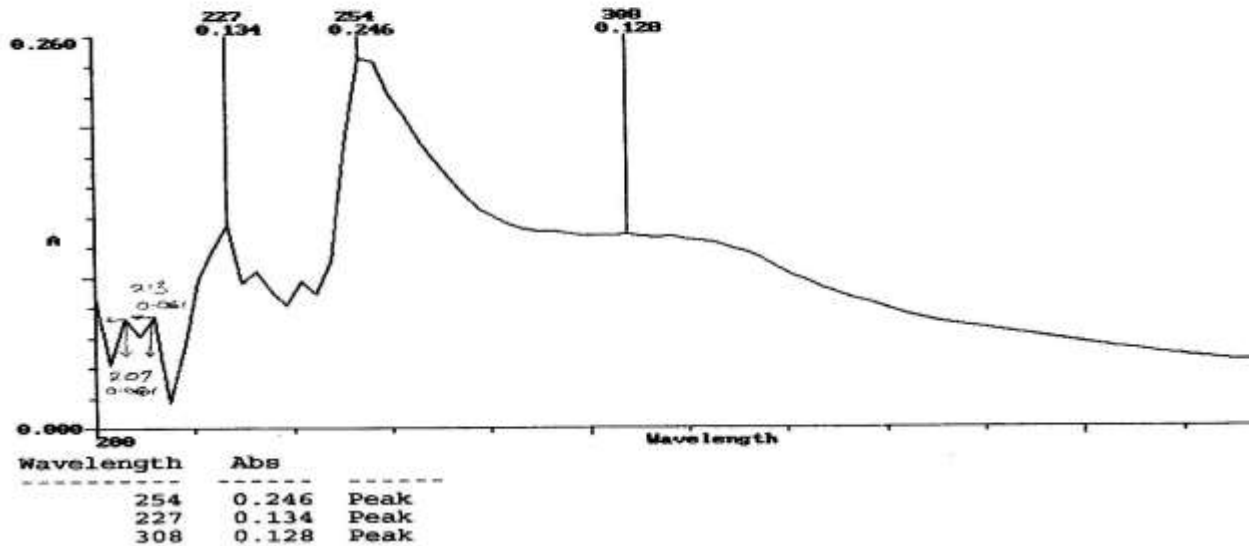


Figure S44. UV-Visible spectrum of *N'*-cyclopentylidene-2-propylquinoline-4-carbohydrazide, **4h**

TEST SETUP
GENESYS 10S v1.200 2L7J355002

Scanning 10:52 26Feb16
Test Name QH Cy_R[Saved]
Measurement Mode Absorbance
Start Wavelength 200nm
Stop Wavelength 600nm
Sample Positioner 1-Cell Platform
Scan Speed Fast
ID# (0=OFF) Off
Auto Print Off

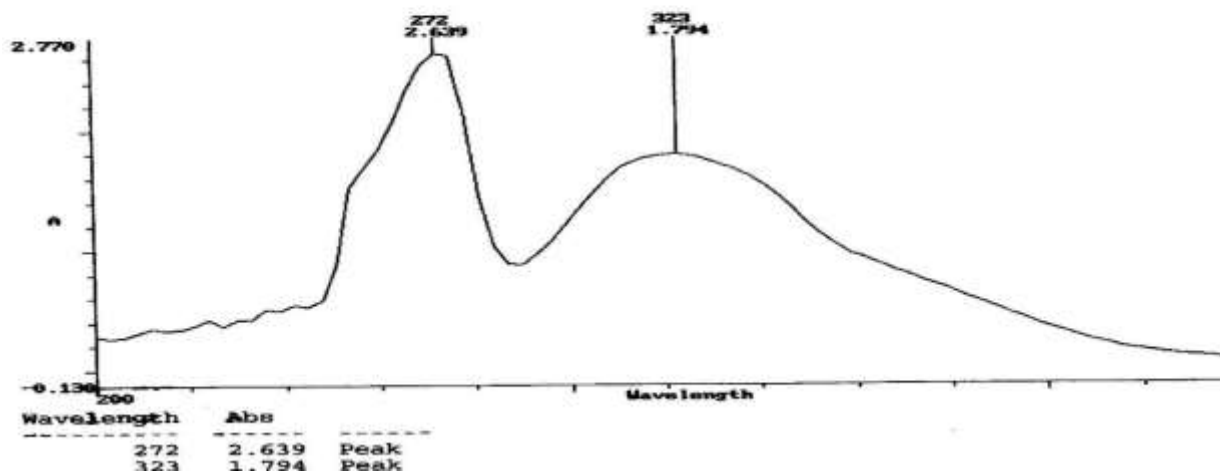


Figure S45. UV-Visible spectrum of *N'*-cyclohexylidene-2-propylquinoline-4-carbohydrazide, **4i**

TEST SETUP
GENESYS 10S v1.200 2L7J355002

Scanning 13:16 3Feb16
Test Name GROUP 2J[Saved]
Measurement Mode Absorbance
Start Wavelength 200nm
Stop Wavelength 600nm
Sample Positioner 1-Cell Platform
Scan Speed Fast
ID# (0=OFF) Off
Auto Print Off

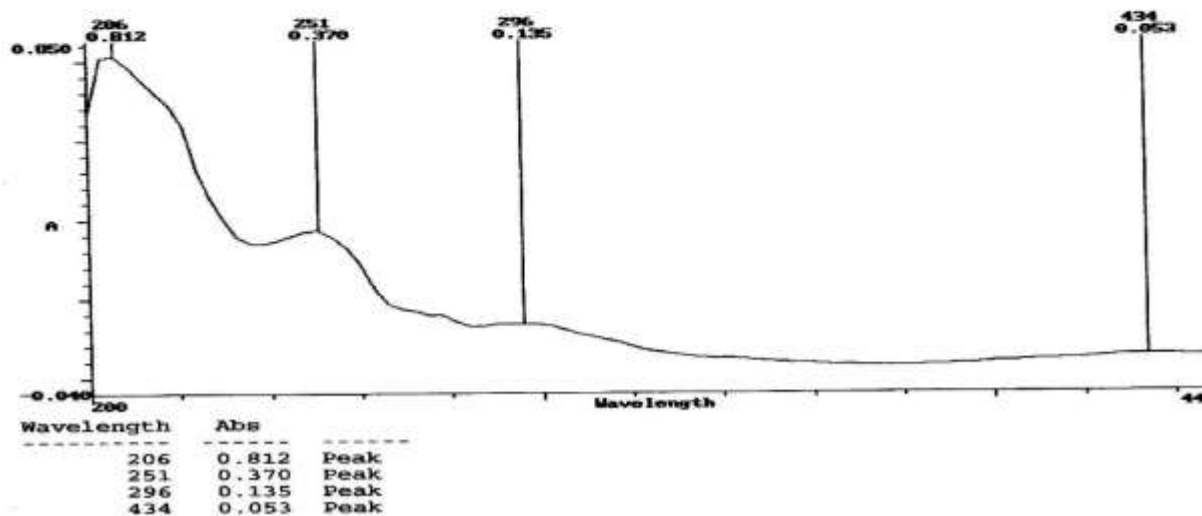


Figure S46. UV-Visible spectrum of *N'*-cycloheptylidene-2-propylquinoline-4-carbohydrazide, **4j**

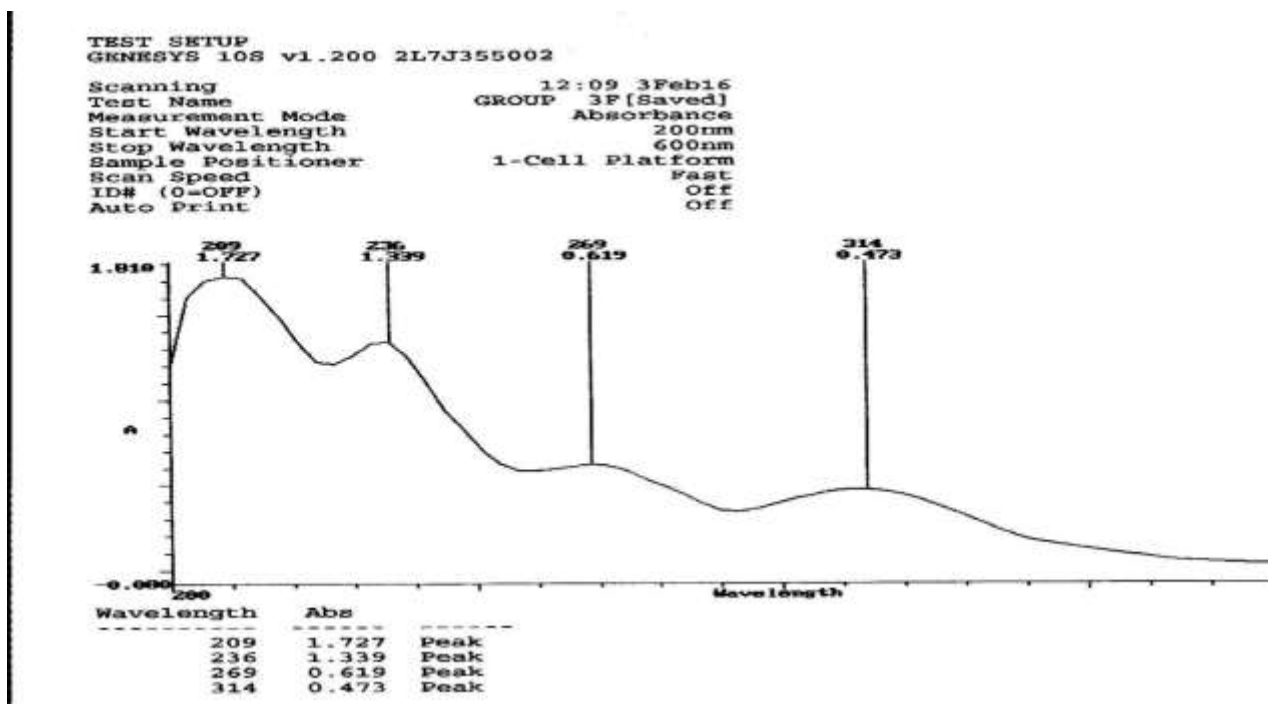


Figure S47. UV-Visible spectrum of *N'*-(1-(4-ethylphenyl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4k**

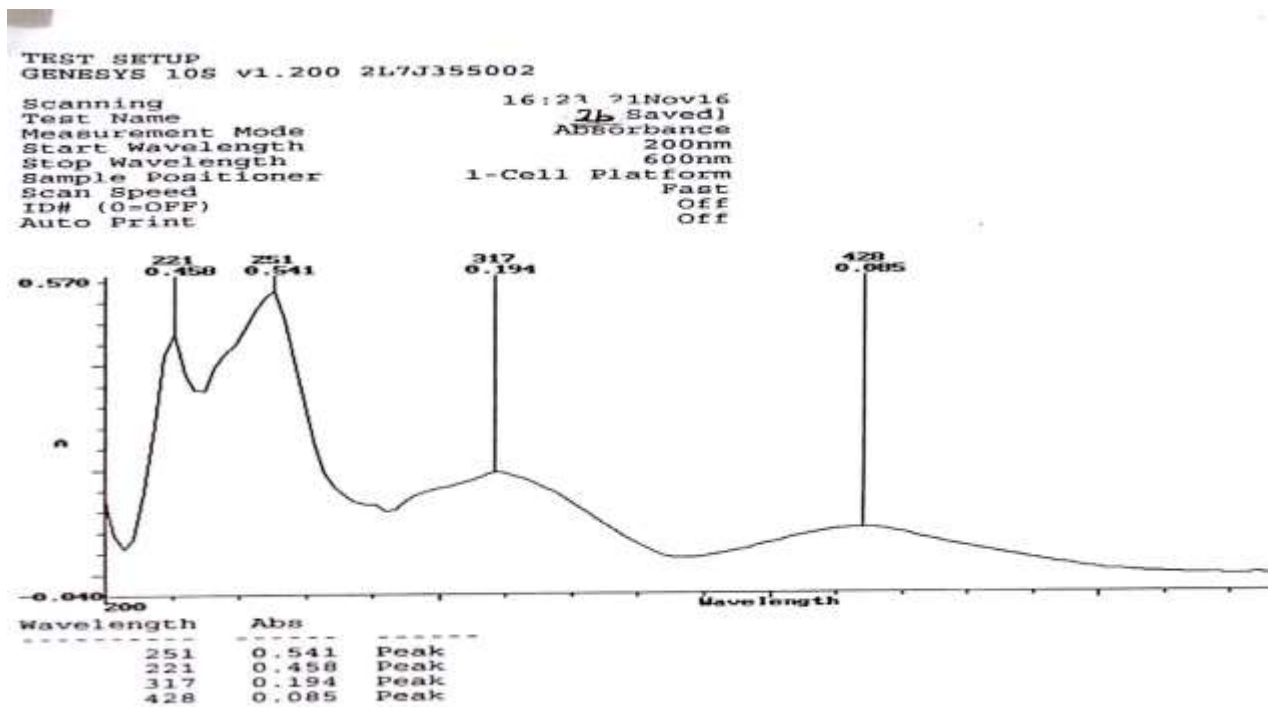


Figure S48. UV-Visible spectrum of *N'*-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2-propylquinoline-4-carbohydrazide, **4l**

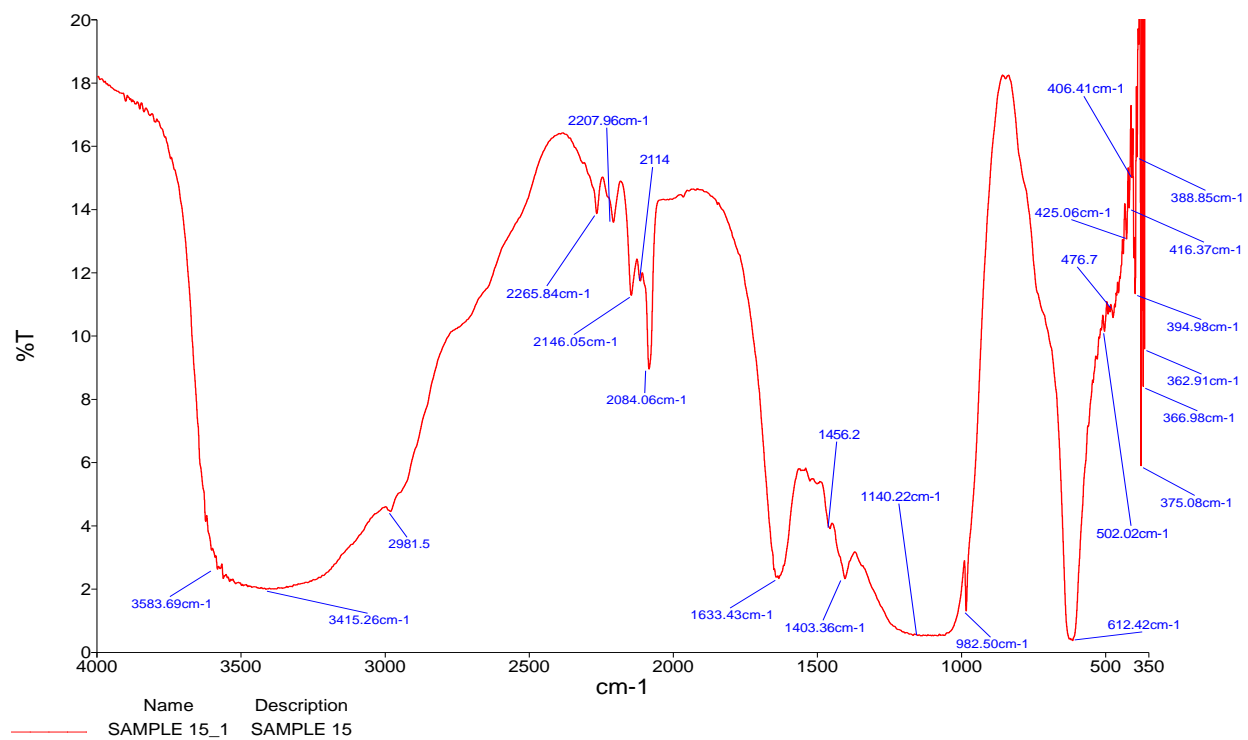


Figure S49. FT-IR spectrum of *N'*-(butan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4a**

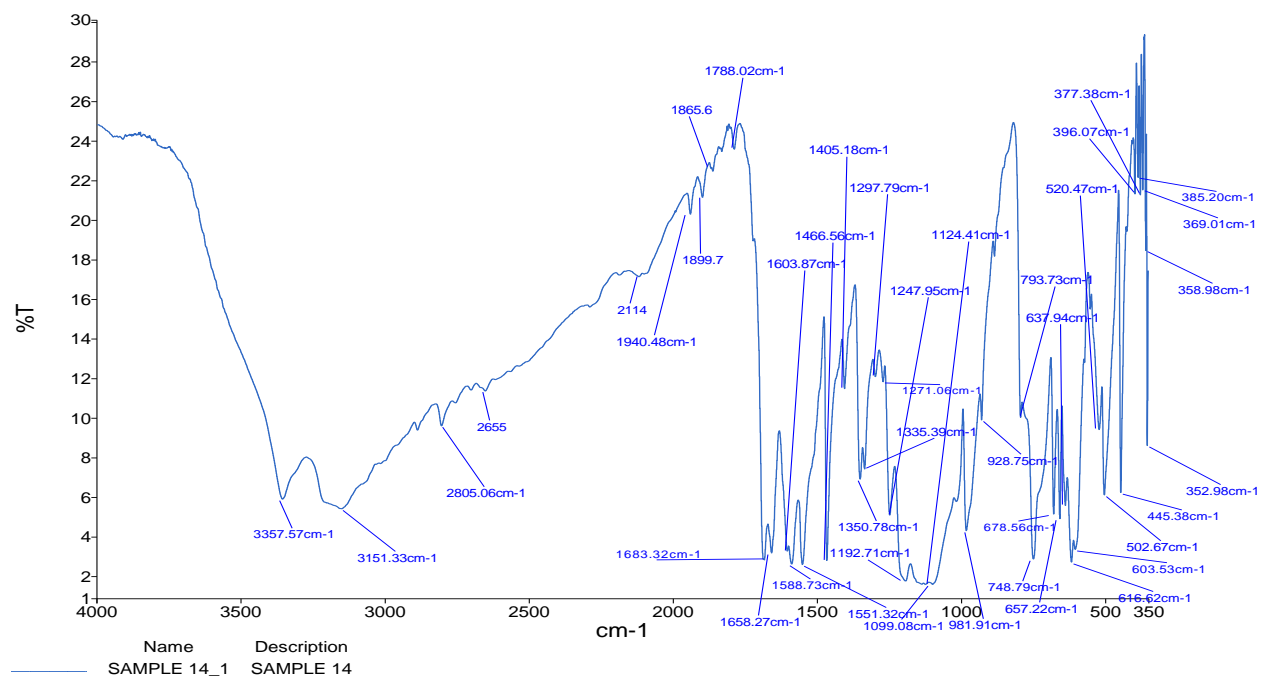


Figure S50. FT-IR spectrum of *N'*-(pentan-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4b**.

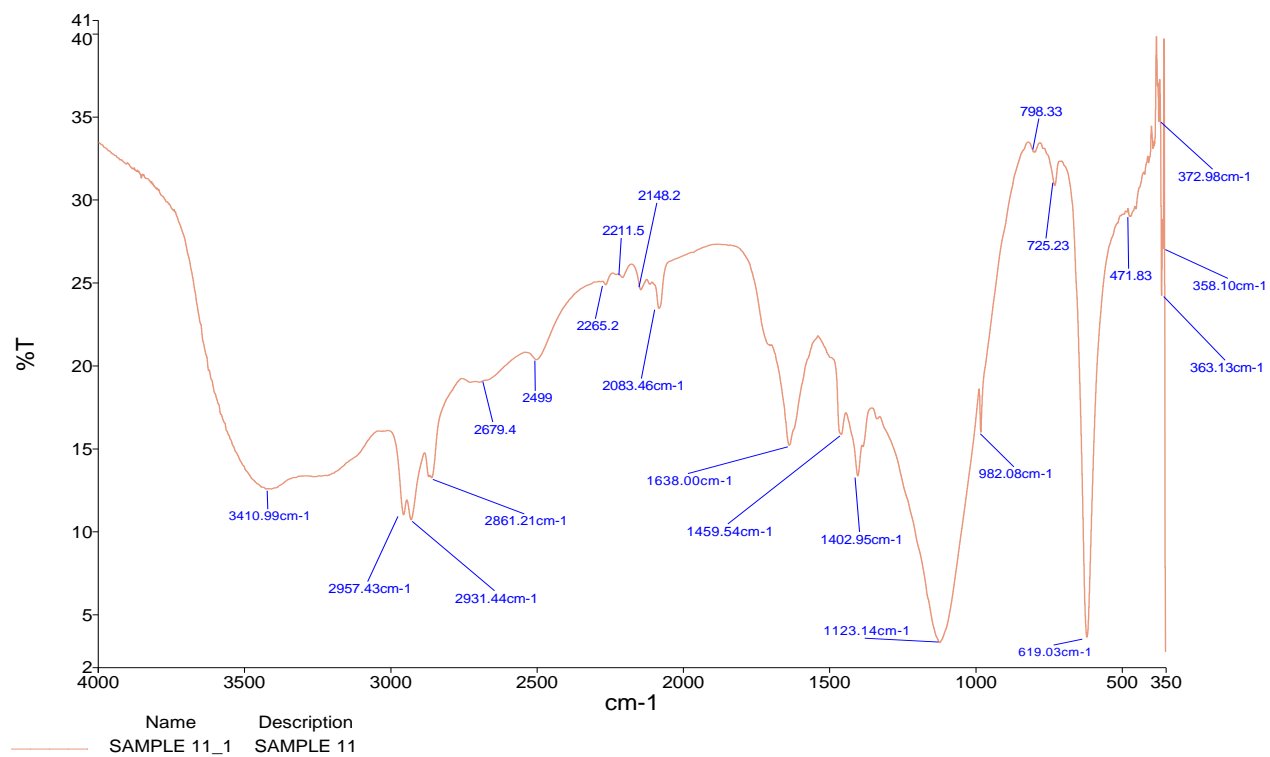


Figure S51: FT-IR spectrum of *N'*-(hepta-2-ylidene)-2-propylquinoline-4-carbohydrazide, **4c**