Application of TMU-17-UR as a pillar-layered MOFs as a hydrogen bonding catalyst for the preparation of pyrazolo[3,4-*b*]quinolines

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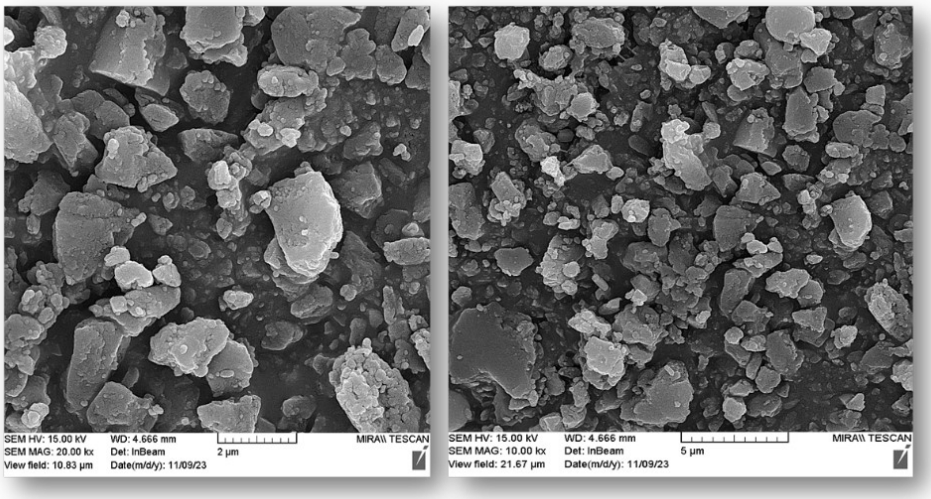


Figure S1: Scanning electron microscope (SEM) images of TMU-17-UR as an efficient H-bond pillar-layered MOFs-based catalyst.

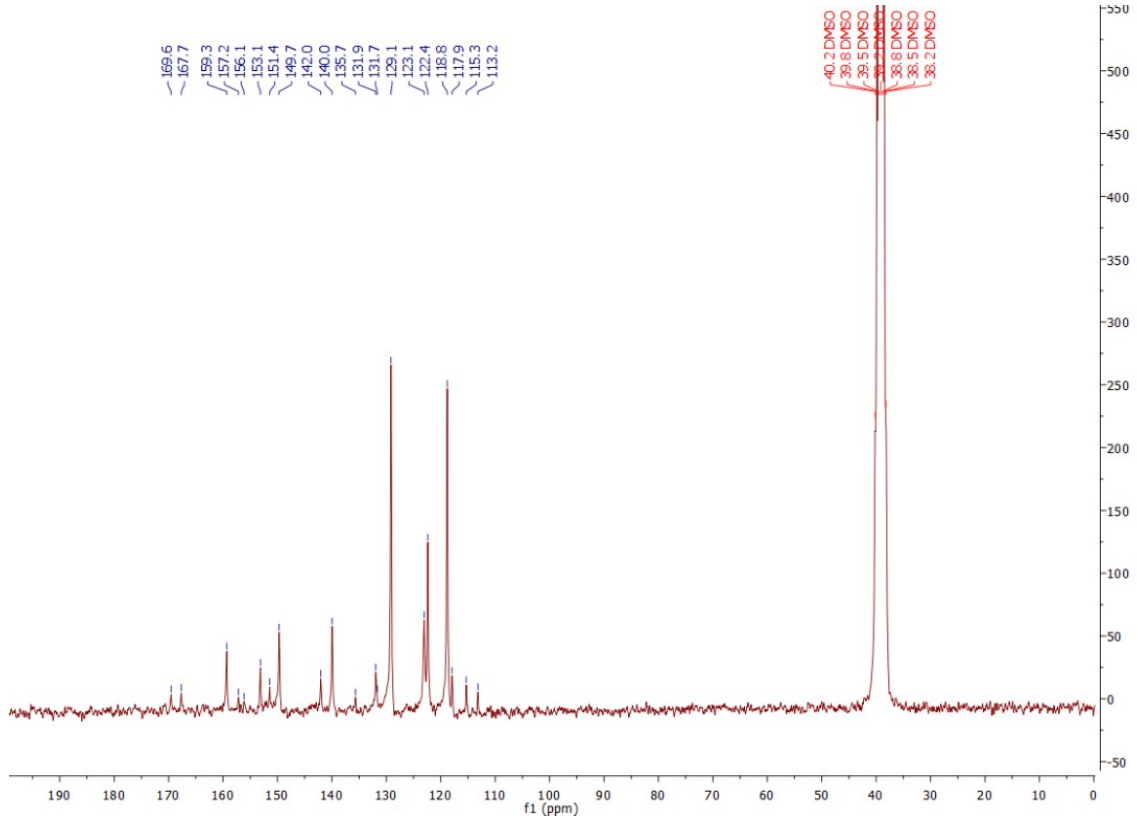
Materials and methods

Indole (C_8H_7N, 99%), acetic anhydride ($C_4H_6O_3$, 95%), cyano acetic acid ($C_3H_3NO_2$, 99%), hydrazine (N_2H_4, 80%), *P*-TSA ($C_7H_8O_3S$, 99%), 5,5-dimethylcyclohexane-1,3-dione (Sigma-Aldrich, 99%), 1-(4-chlorophenyl)ethan-1-one (Merk, 98%), bromine (Sigma-Aldrich, 99%), potassium cyanide (Merk, 97%), pyridine-4-carboxaldehyde (C_6H_5NO, Sigma-Aldrich), formic acid (Merk, 98%), $Zn(NO_3)_2{\cdot}6H_2O$ (Sigma-Aldrich, 99%), 2-amino terephthalic acid (NH_2-BDC, 95%), phenyl isocyanate (PhNCO, 98%) and various aromatic aldehyde derivatives (95%) were purchased from Merck and Sigma-Aldrich. Furthermore, *N*, *N*-dimethylformamide (DMF, 99%), ethanol (EtOH, 99%), methanol (MeOH, 99%), acetonitrile (CH_3CN, 99%) and other solvents were purchased from commercial sources without further purification.

Characterization

The X-ray powder diffraction (XRD) technique with a model device PHILIPS PW1730 (Netherlands), was used to characterize the crystal plates of the catalyst. The FT-IR technique model device (PerkinElmer Spectrum Version 10.02.00) was used to identify the functional groups of the different stages of the desired catalyst. Moreover, the morphology of the different stages of catalyst was characterized using a scanning electron microscope (SEM) technique as well as energy-dispersive spectroscopy (EDS) and elemental mapping was carried out by the model ZEISS Sigma VP (German). In addition, the thermal and chemical stability of the catalyst was determined using a thermogravimetry/differential thermal analysis (TG/DTA) technique (BAHR STA 503). Finally, the Brunauer-Emmett-Teller (BET, BELSORP-mini-II)

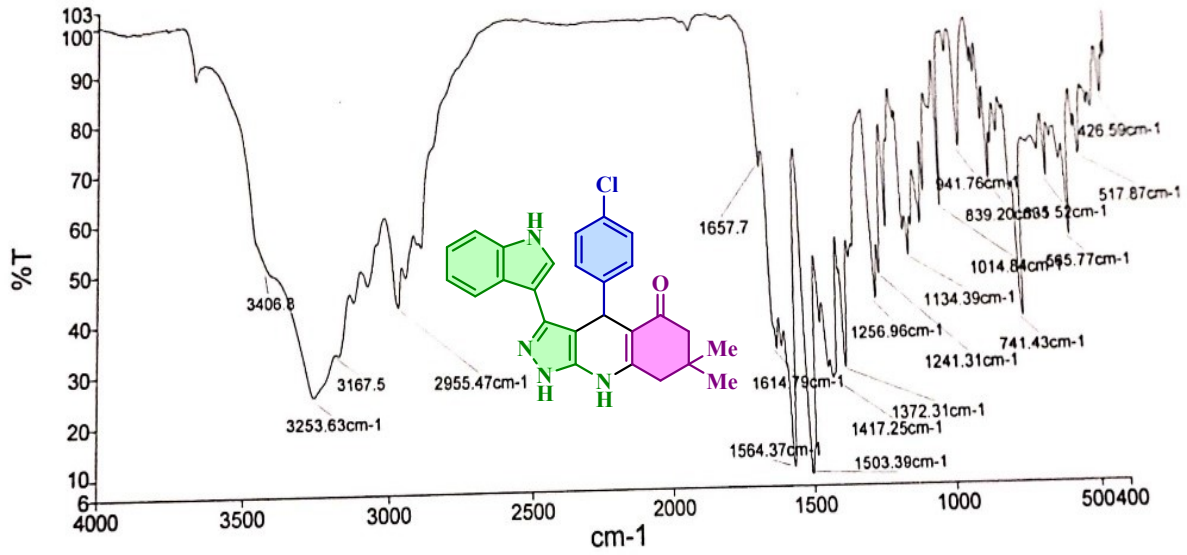
British Journal of Haematology (BJH) technique with a model device BELSORP-mini-II was utilized to determine the surface area and pore size of the synthesized catalyst.



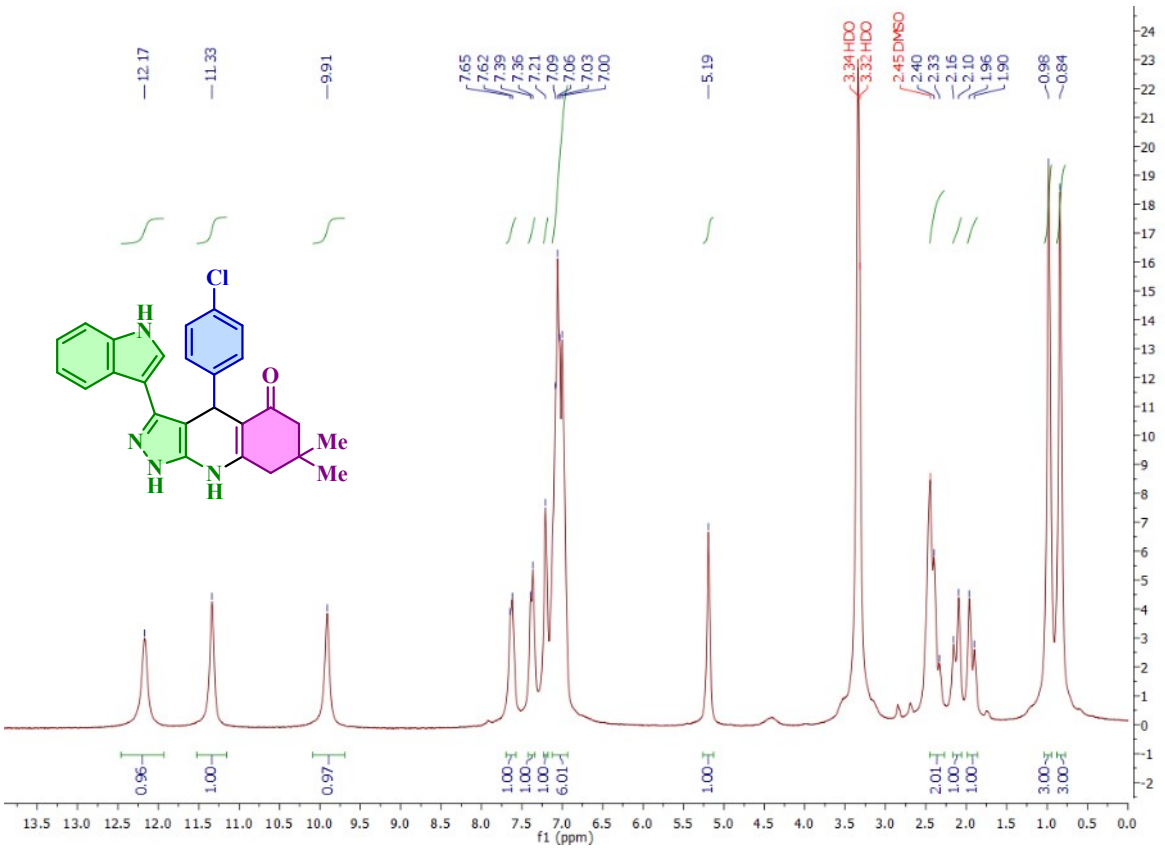
^{13}C-NMR spectrum of TMU-17-UR.

Spectral data of TMU-17-UR as a catalyst:

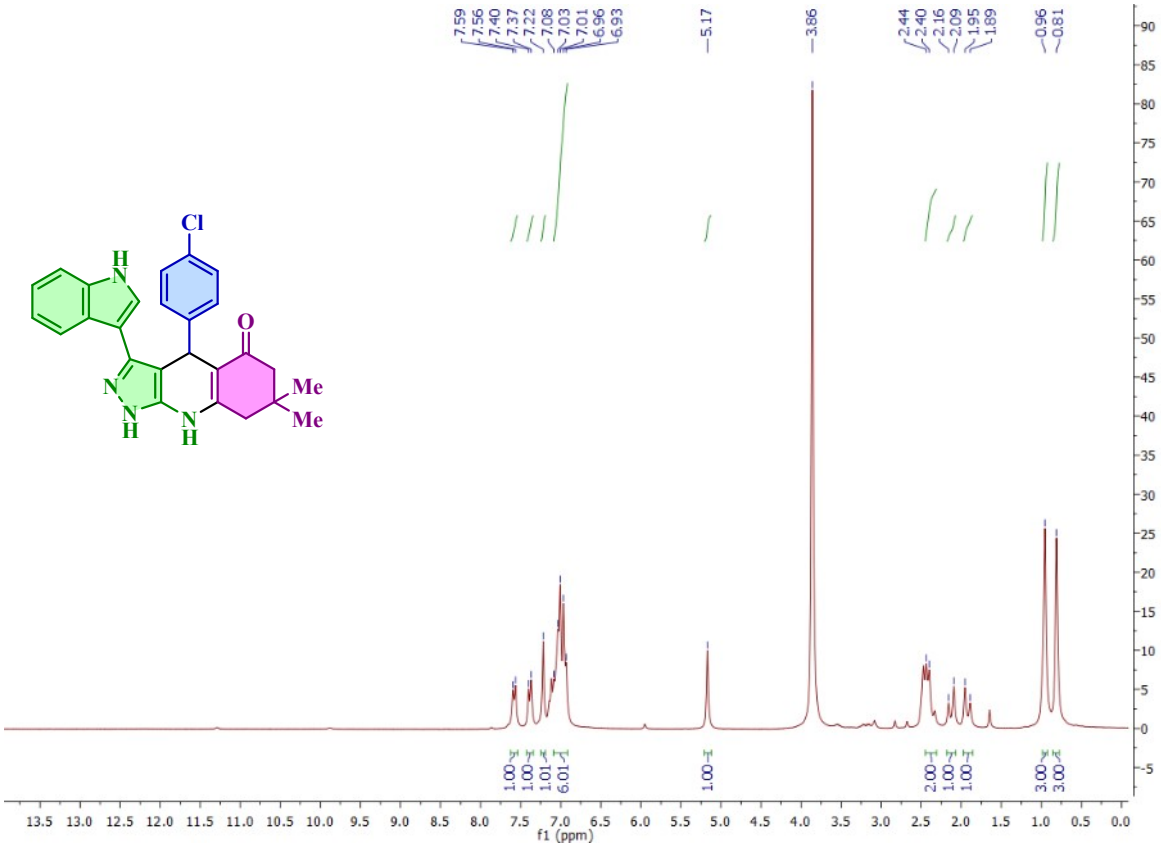
^{13}C-NMR (63 MHz, DMSO-d_6) δ_{ppm} 169.6, 167.7, 159.3, 157.2, 156.1, 153.1, 151.4, 149.7, 142.0, 140.0, 135.7, 131.9, 131.7, 129.1, 123.1, 122.4, 118.8, 117.9, 115.3, 113.2.



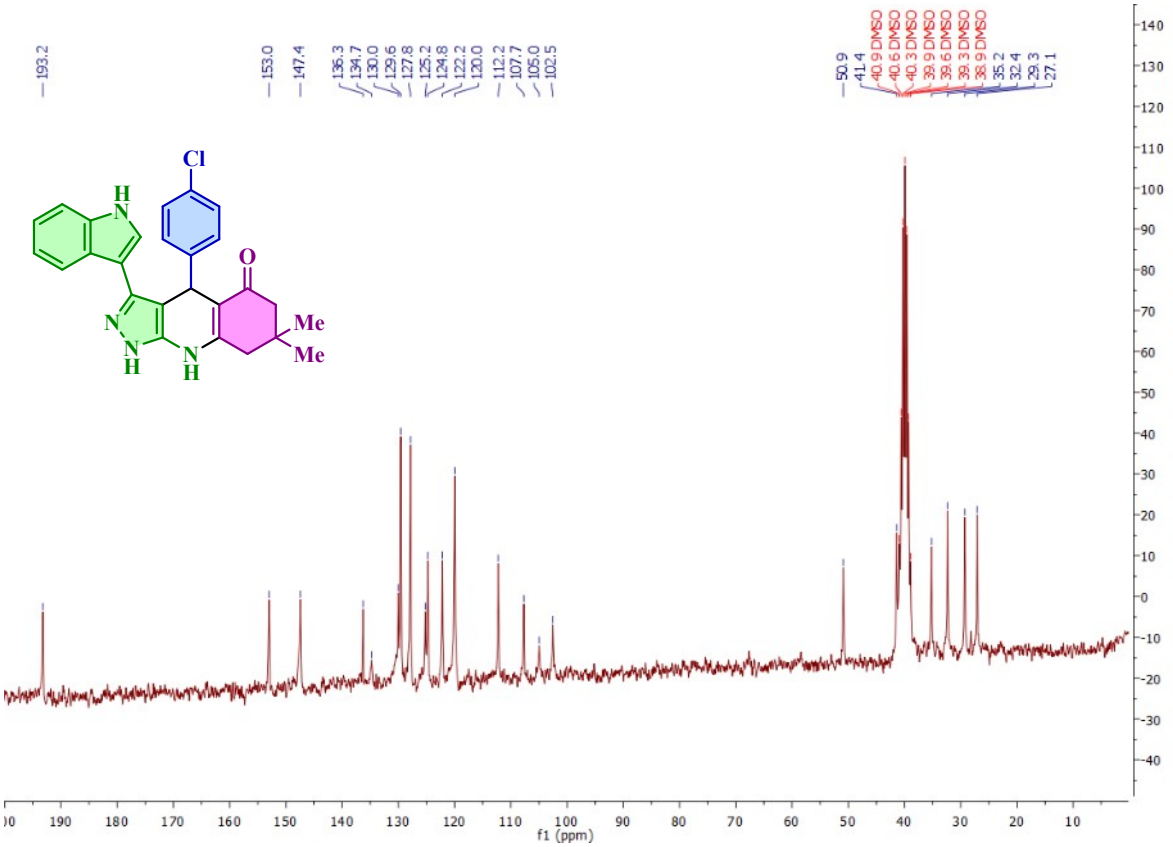
FT-IR spectrum of 4-(4-chlorophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC1).



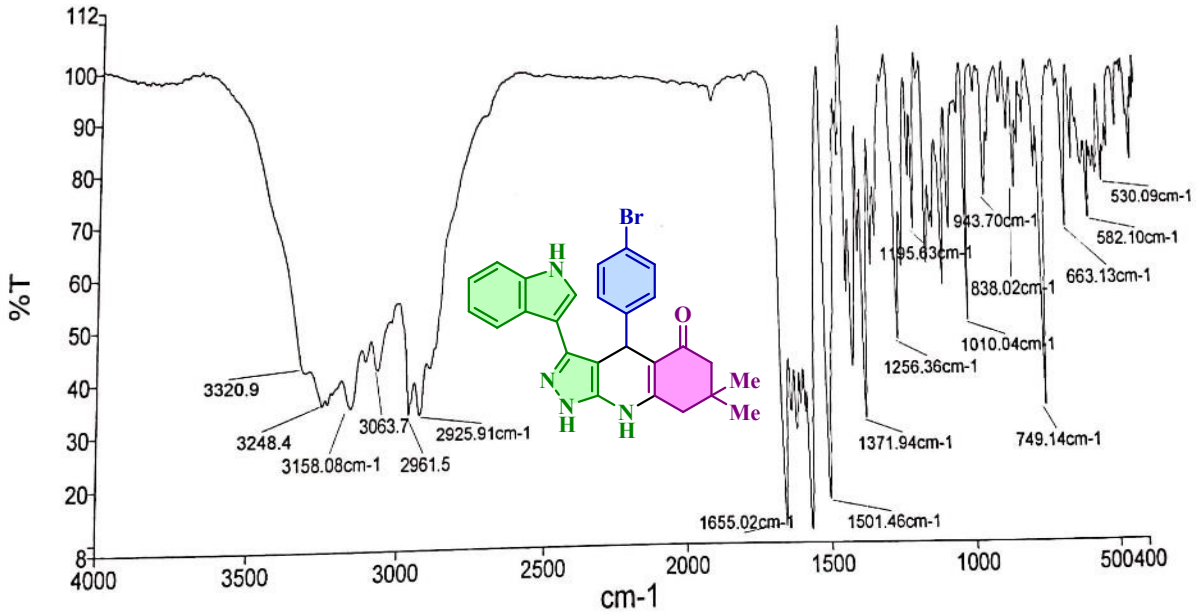
[1]H-NMR spectrum of 4-(4-chlorophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC1).



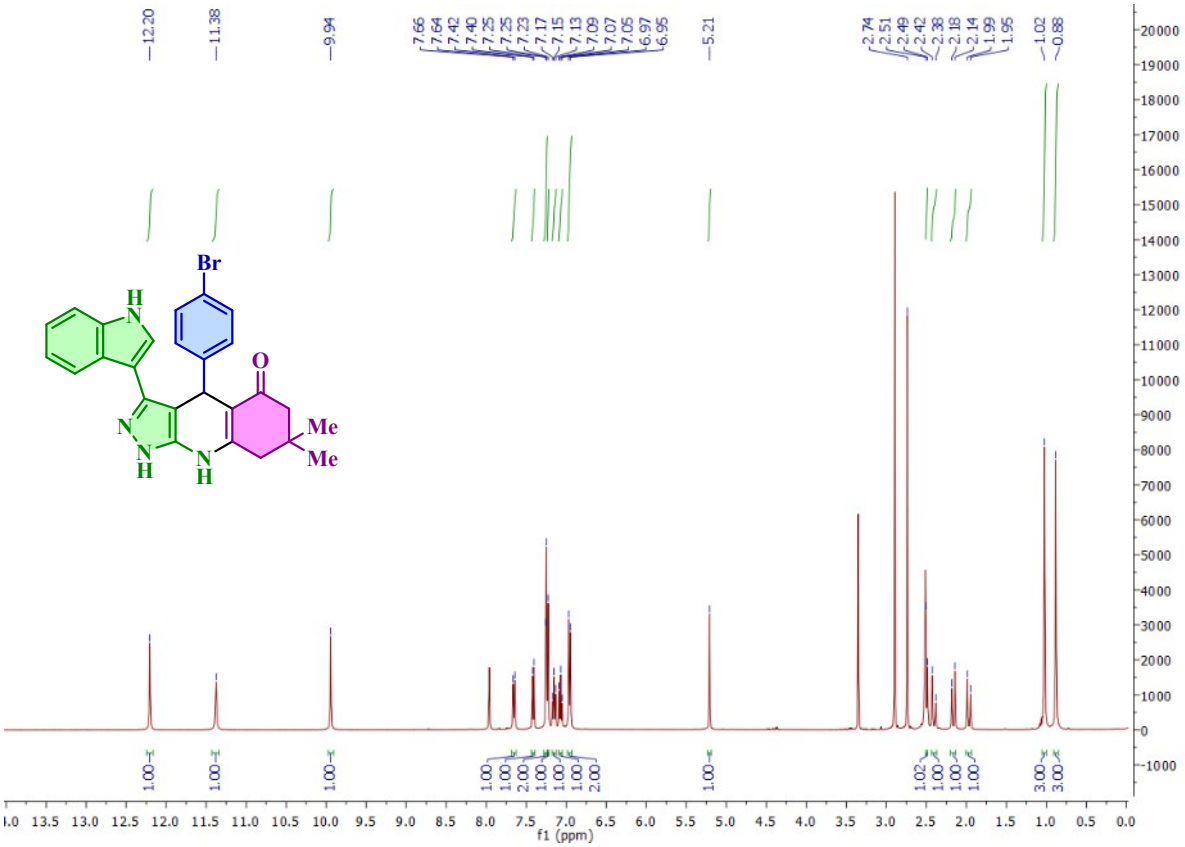
D_2O exchanging spectrum of 4-(4-chlorophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC1).



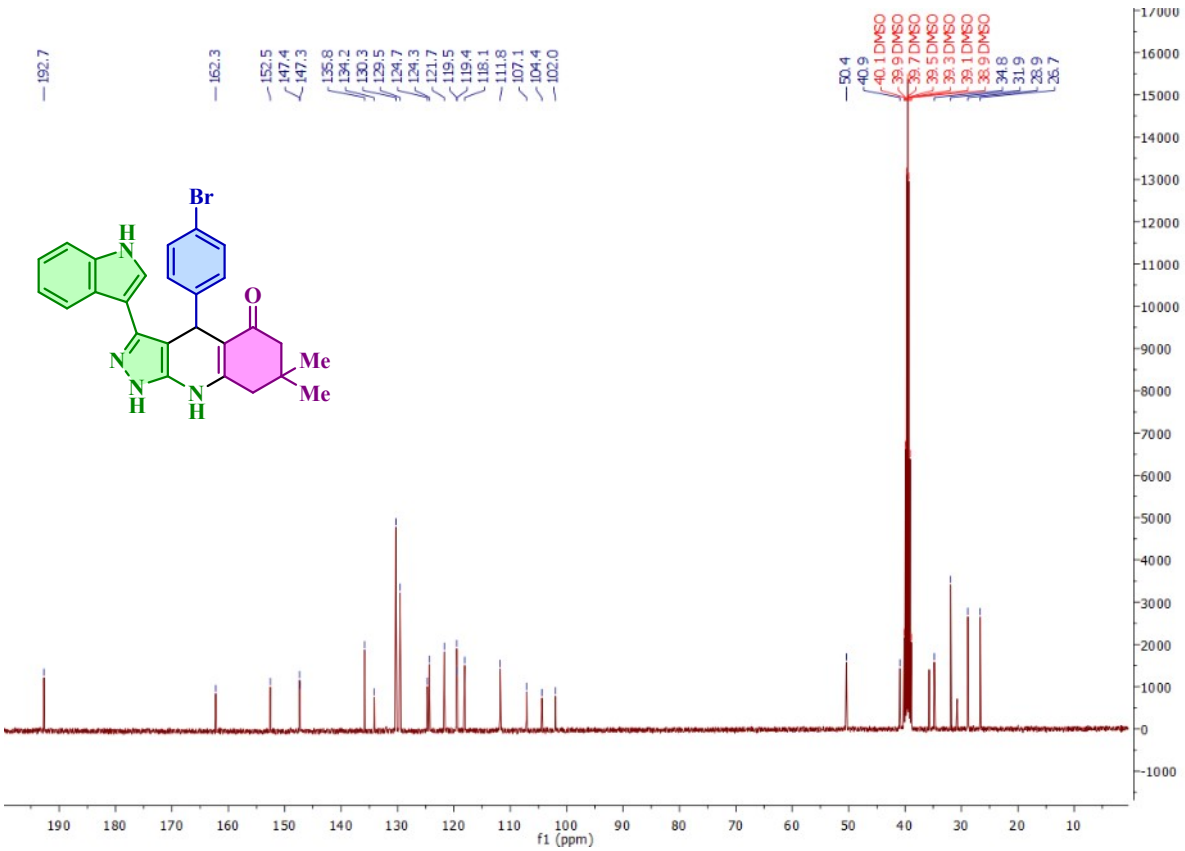
^{13}C-NMR spectrum of 4-(4-chlorophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC1).



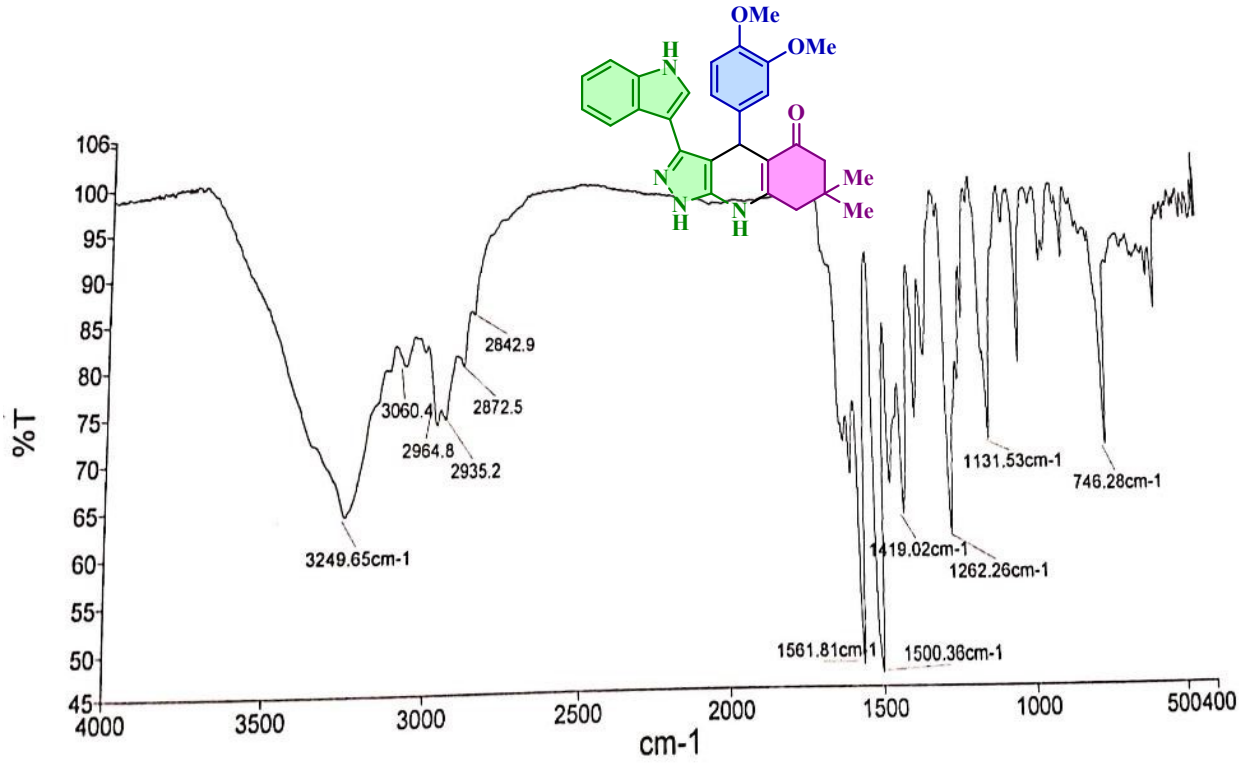
FT-IR spectrum of 4-(4-bromophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC2).



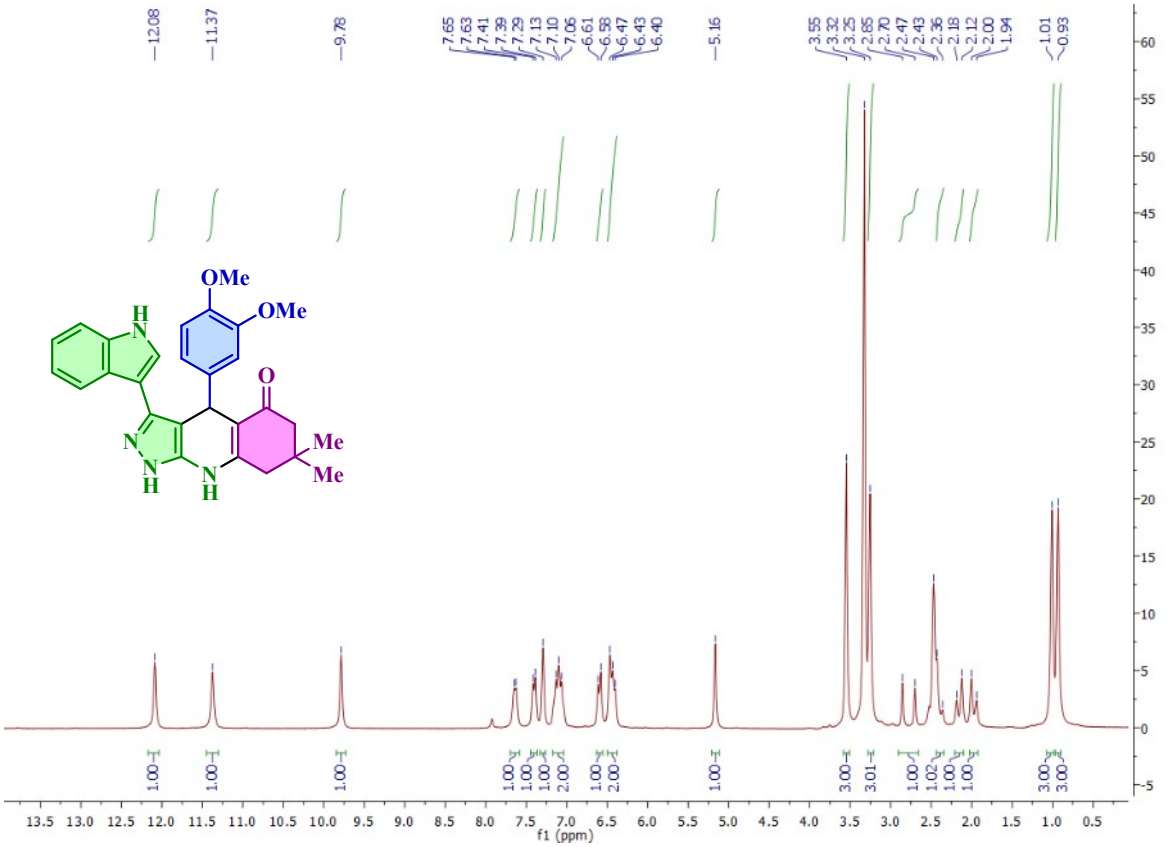
[1]H-NMR spectrum of 4-(4-bromophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC2).



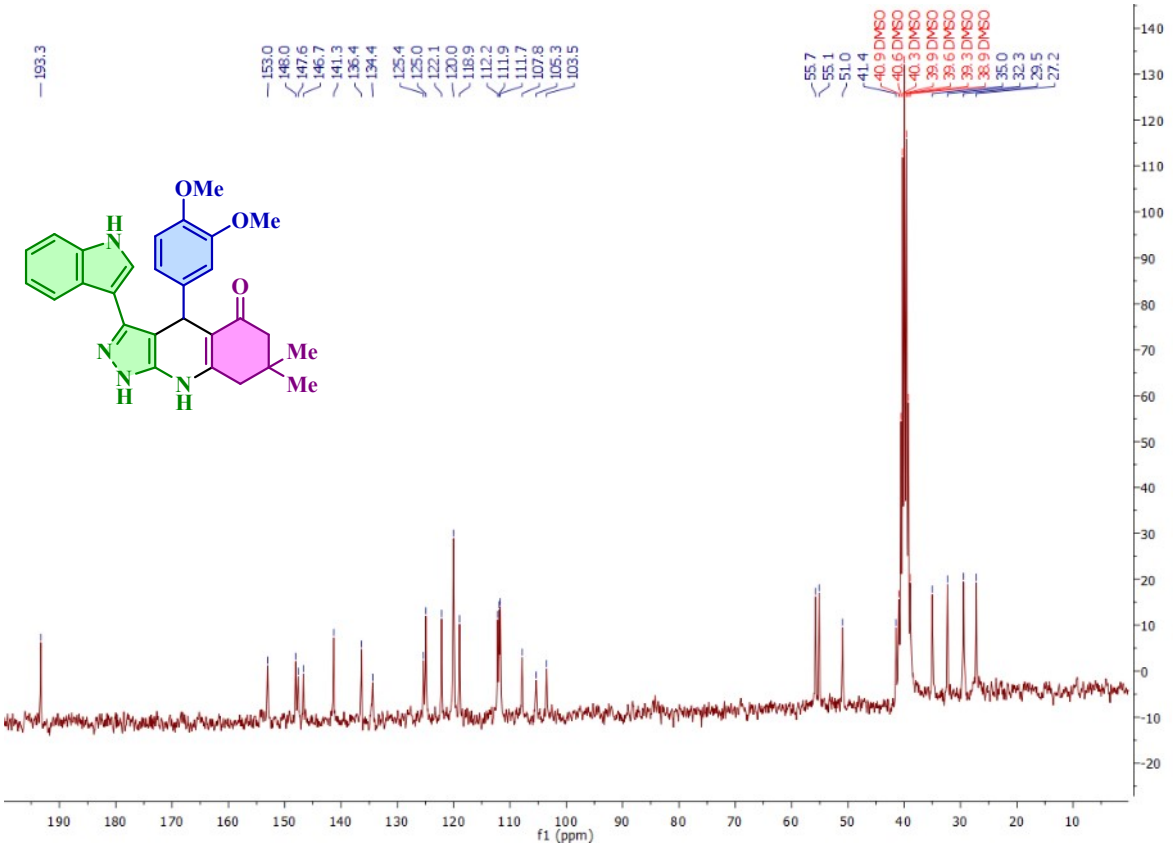
^{13}C-NMR spectrum of 4-(4-bromophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC2).



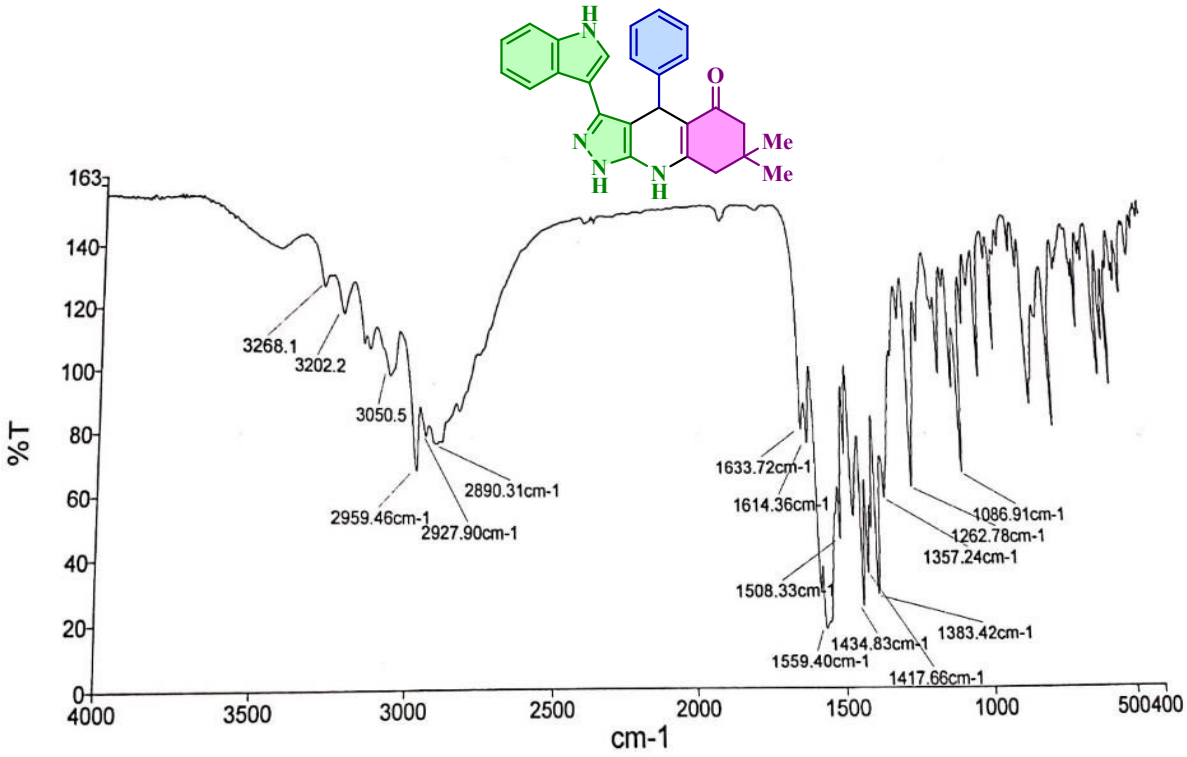
FT-IR spectrum of 4-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC3).



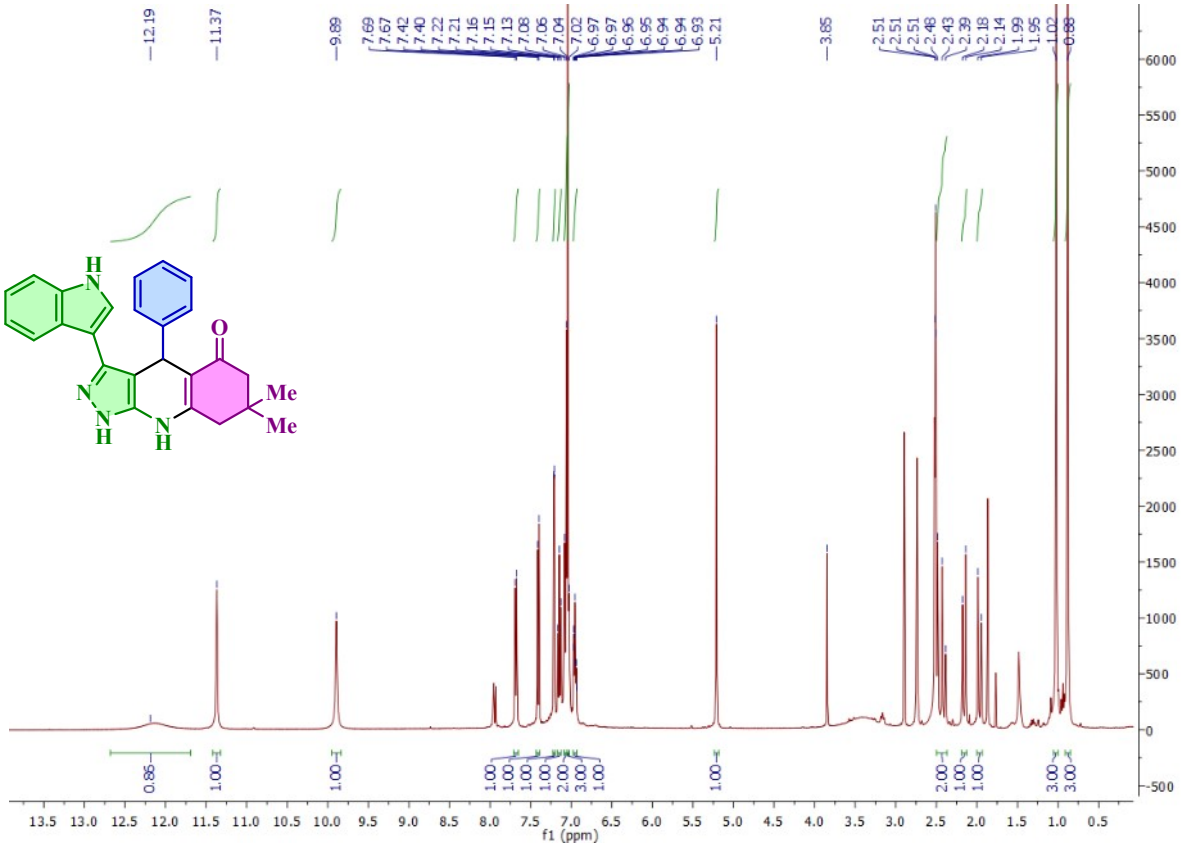
[1]H-NMR spectrum of 4-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC3).



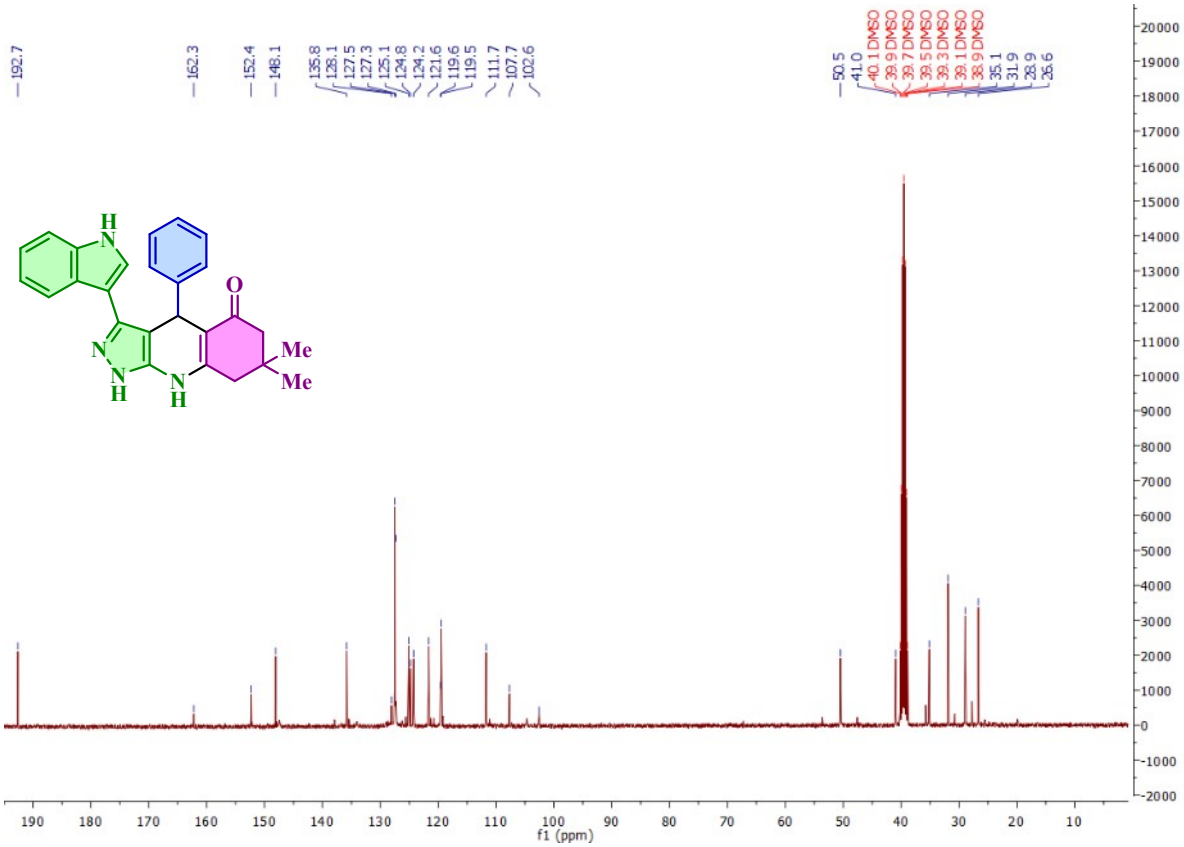
[13]C-NMR spectrum of 4-(3,4-dimethoxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC3).



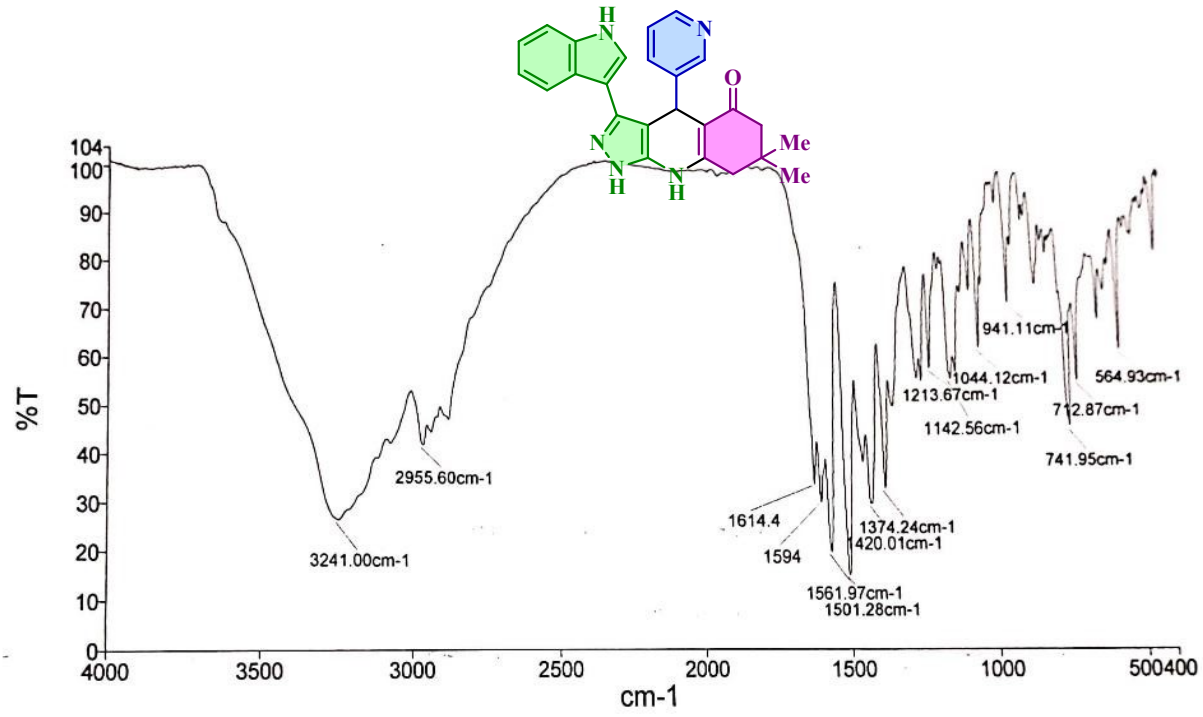
FT-IR spectrum of 3-(1H-indol-3-yl)-7,7-dimethyl-4-phenyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC4).



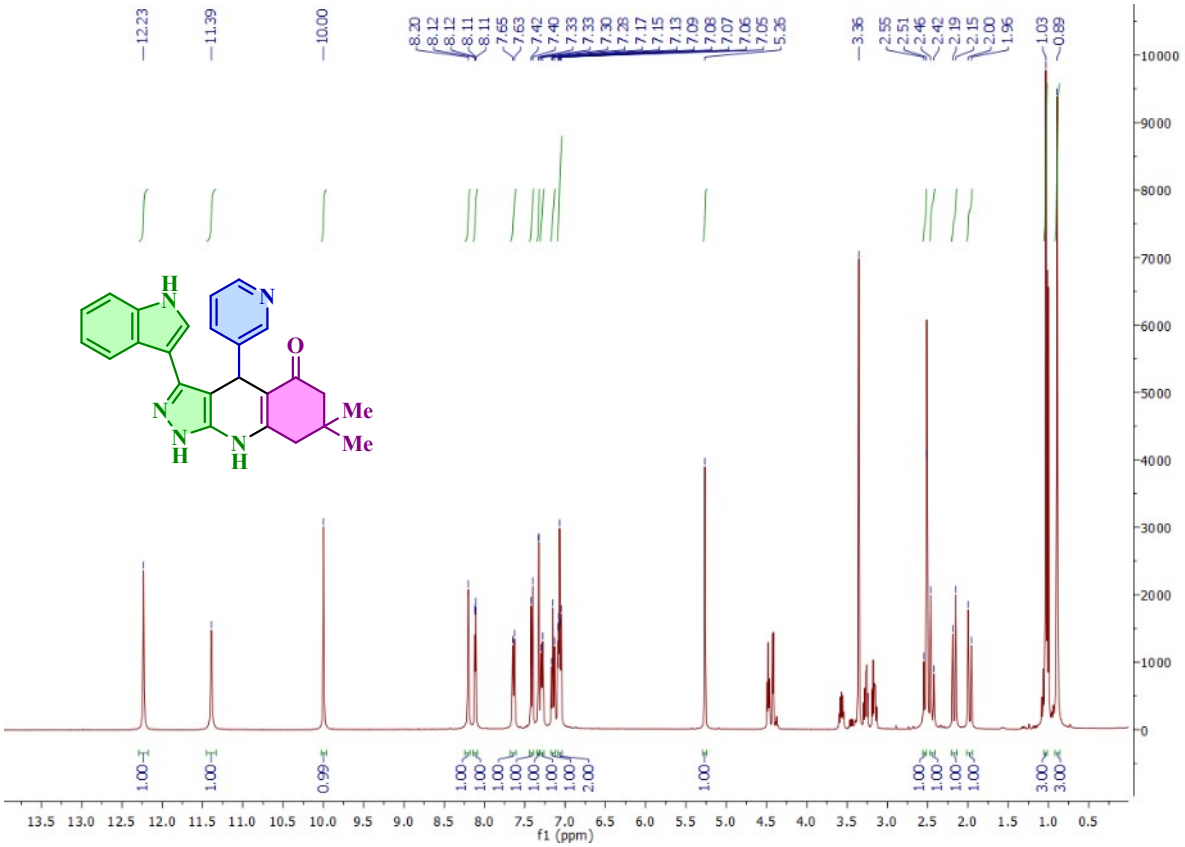
[1]H-NMR spectrum of 3-(1H-indol-3-yl)-7,7-dimethyl-4-phenyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC4).



^{13}C-NMR spectrum of 3-(1H-indol-3-yl)-7,7-dimethyl-4-phenyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC4).



FT-IR spectrum of 3-(1H-indol-3-yl)-7,7-dimethyl-4-(pyridin-3-yl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC5).



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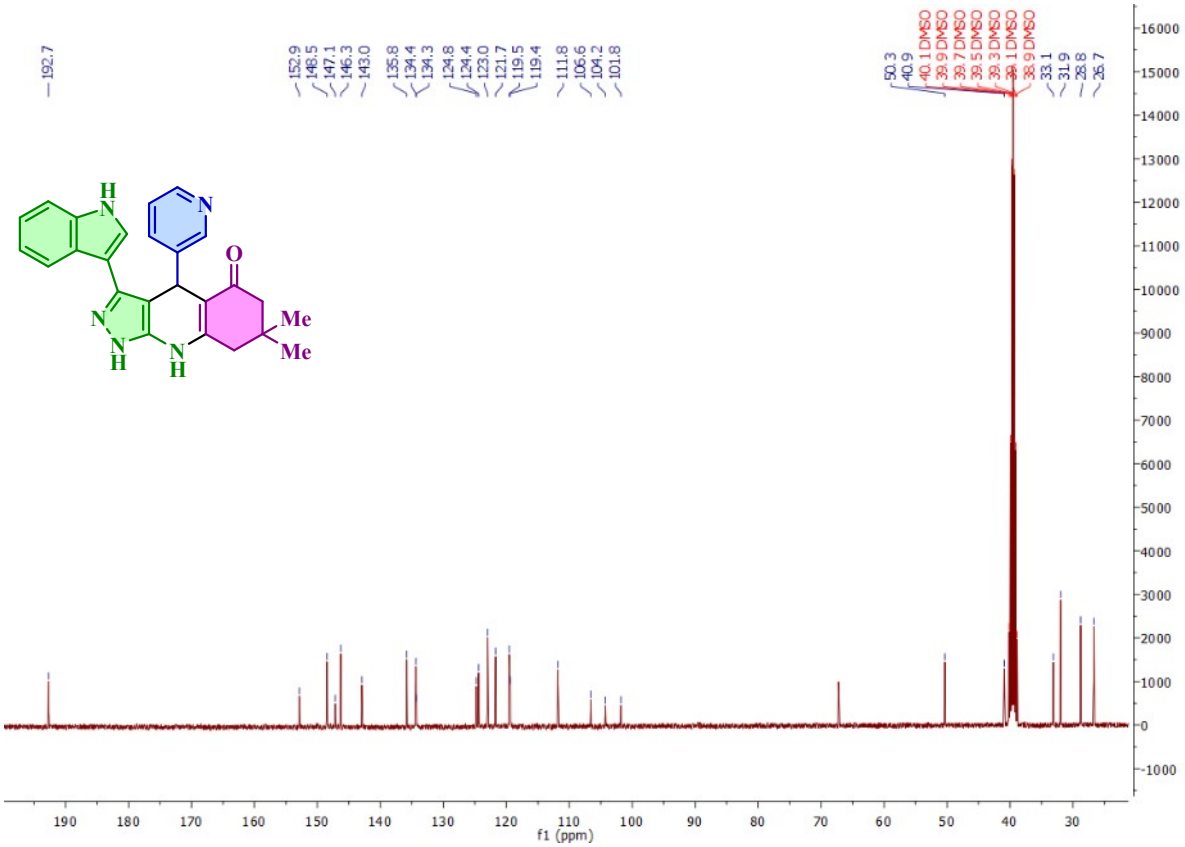
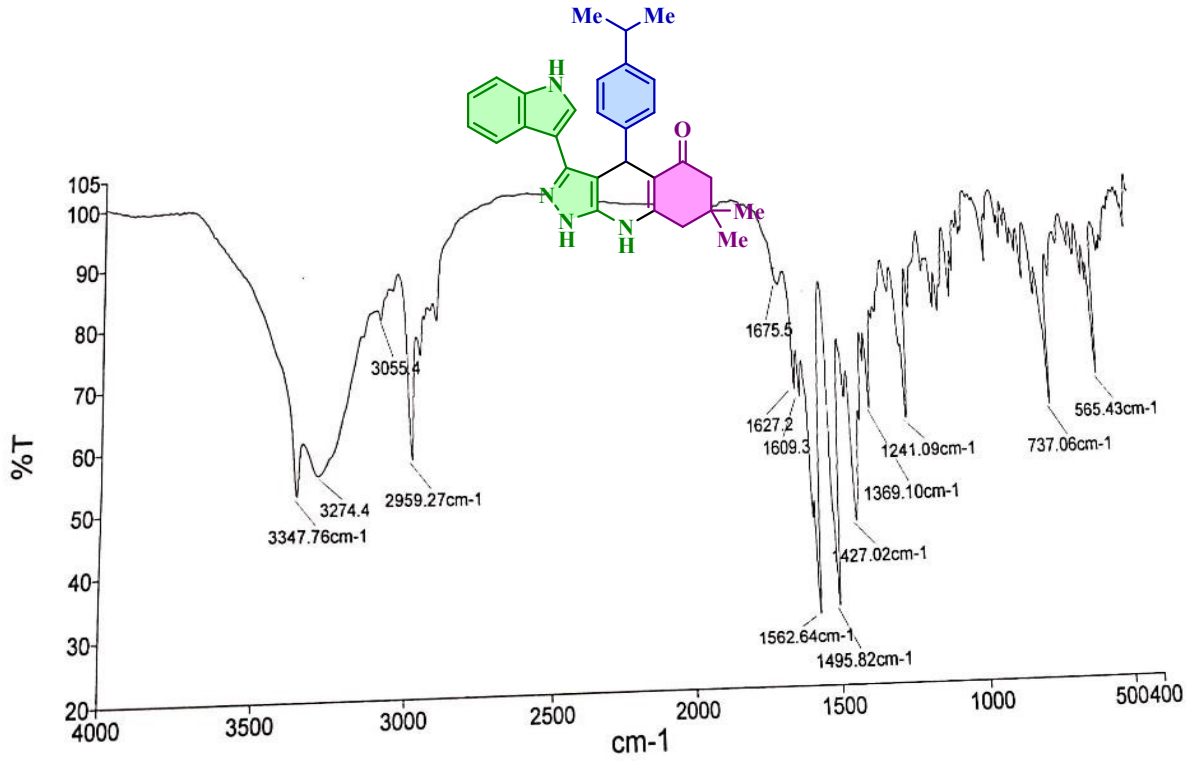
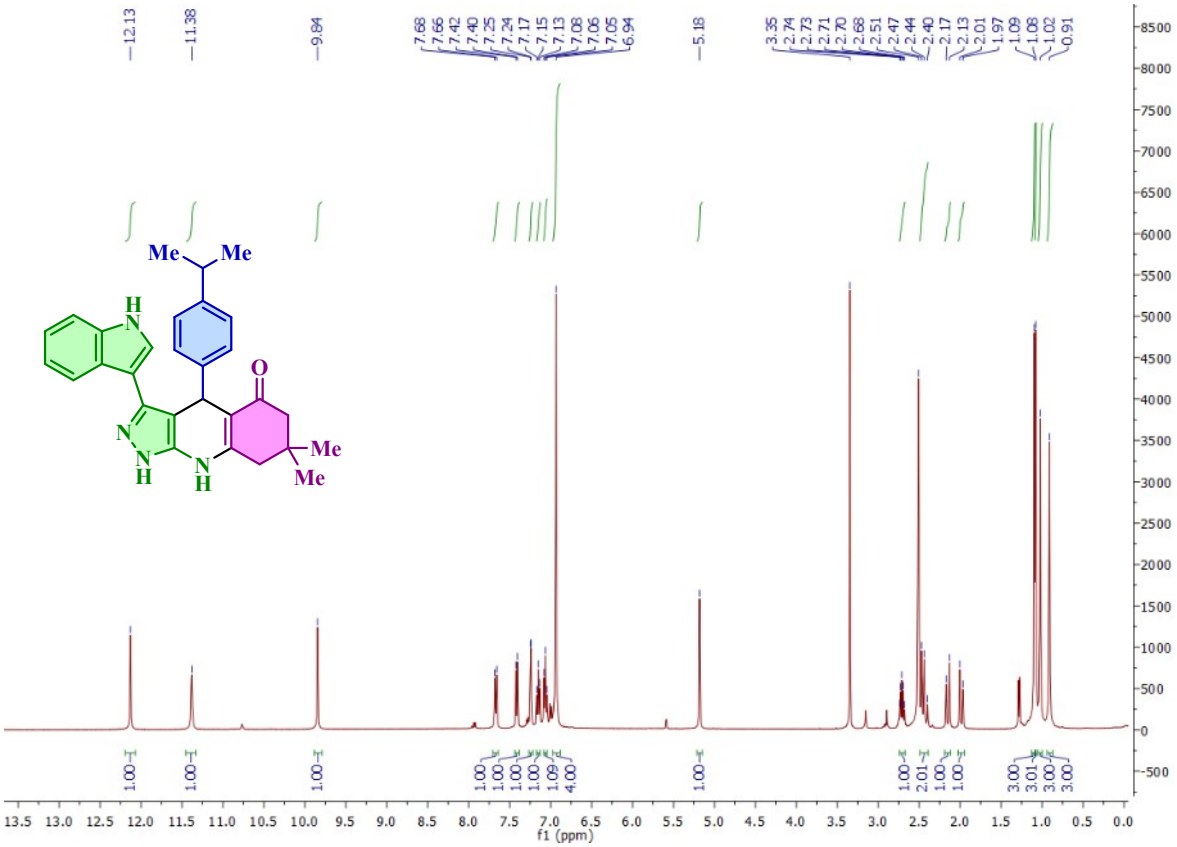


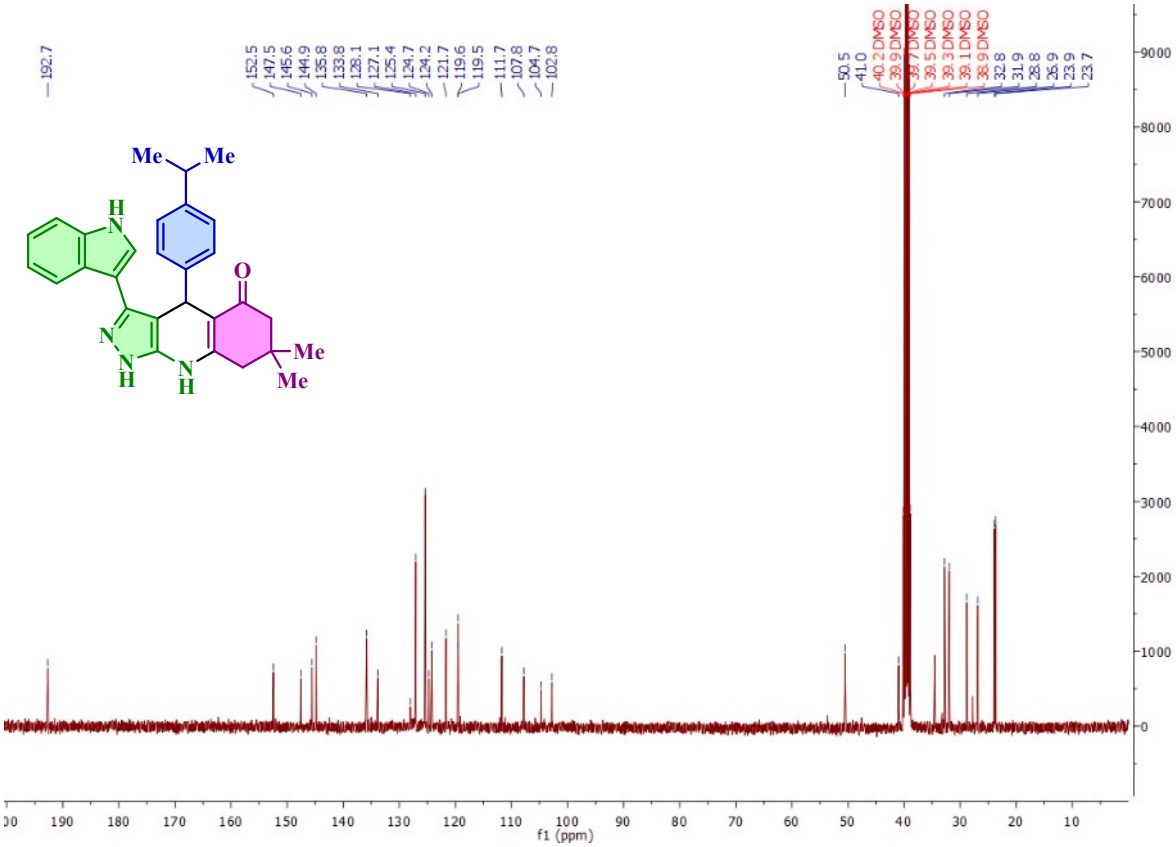
^{13}C-NMR spectrum of 3-(1H-indol-3-yl)-7,7-dimethyl-4-(pyridin-3-yl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC5).



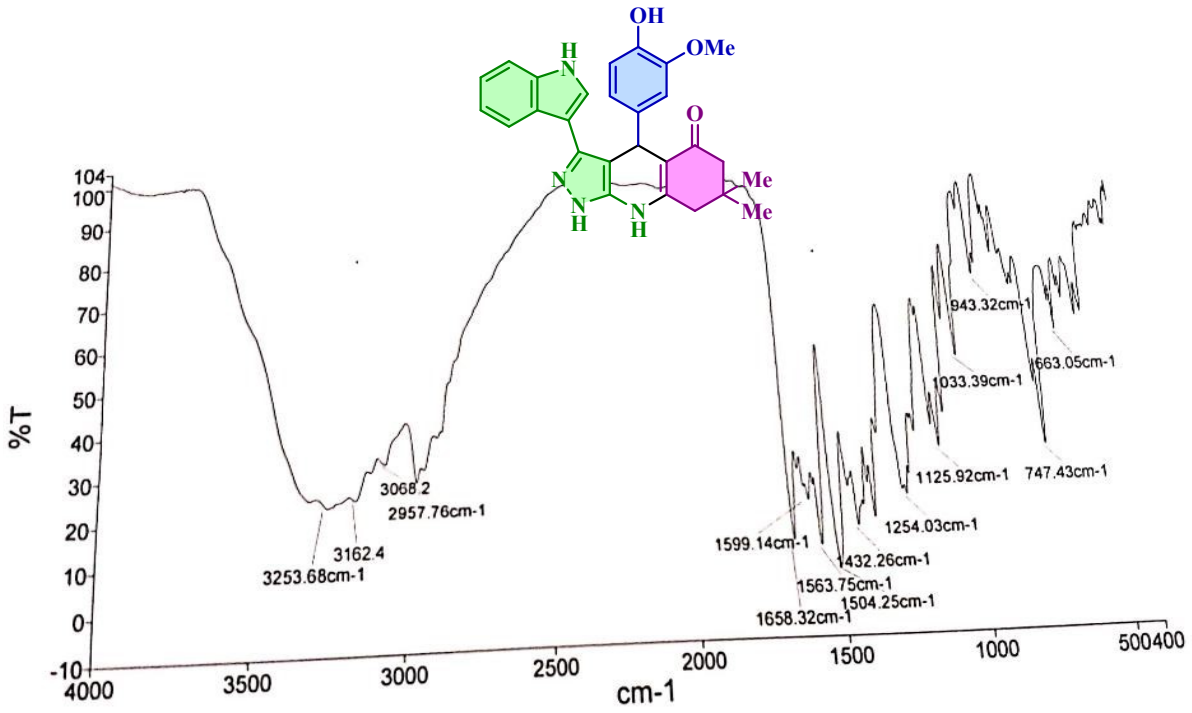
FT-IR spectrum of 3-(1H-indol-3-yl)-4-(4-isopropylphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC6).



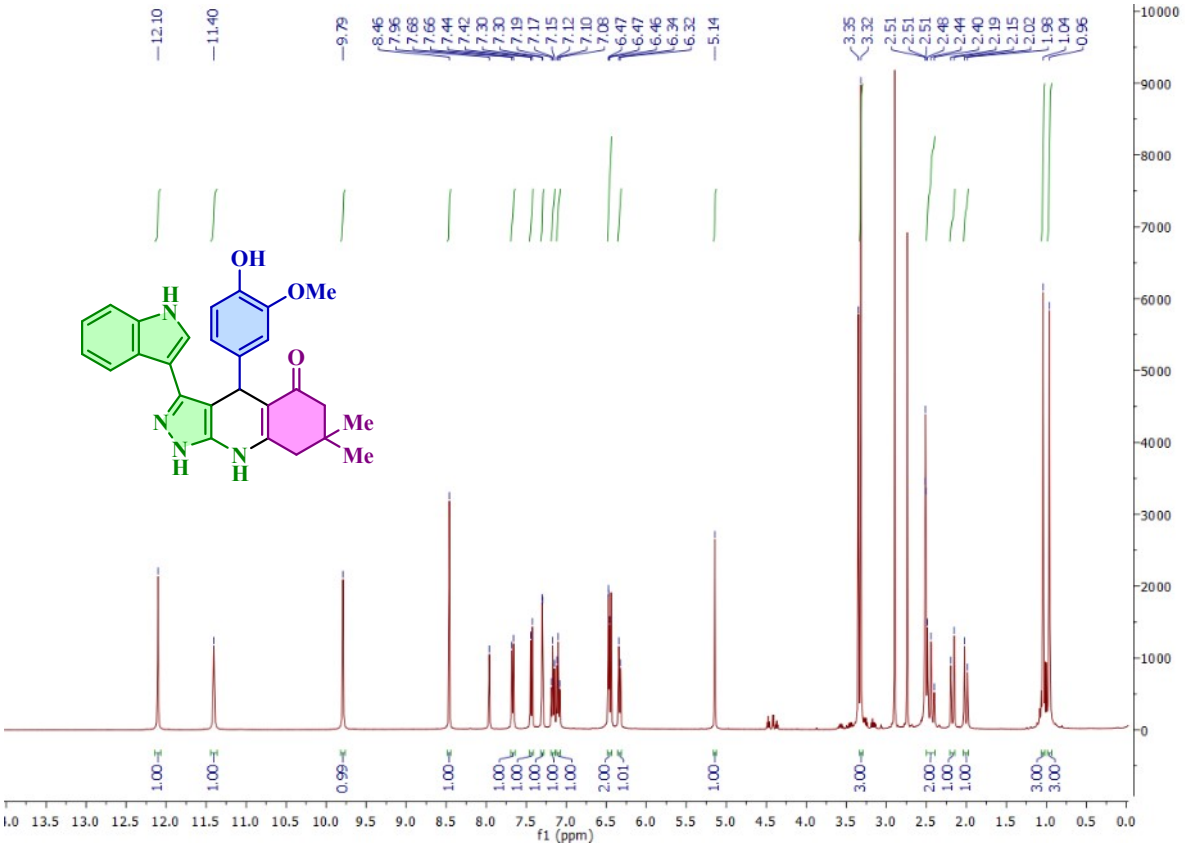
[1]H-NMR spectrum of 3-(1H-indol-3-yl)-4-(4-isopropylphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC6).



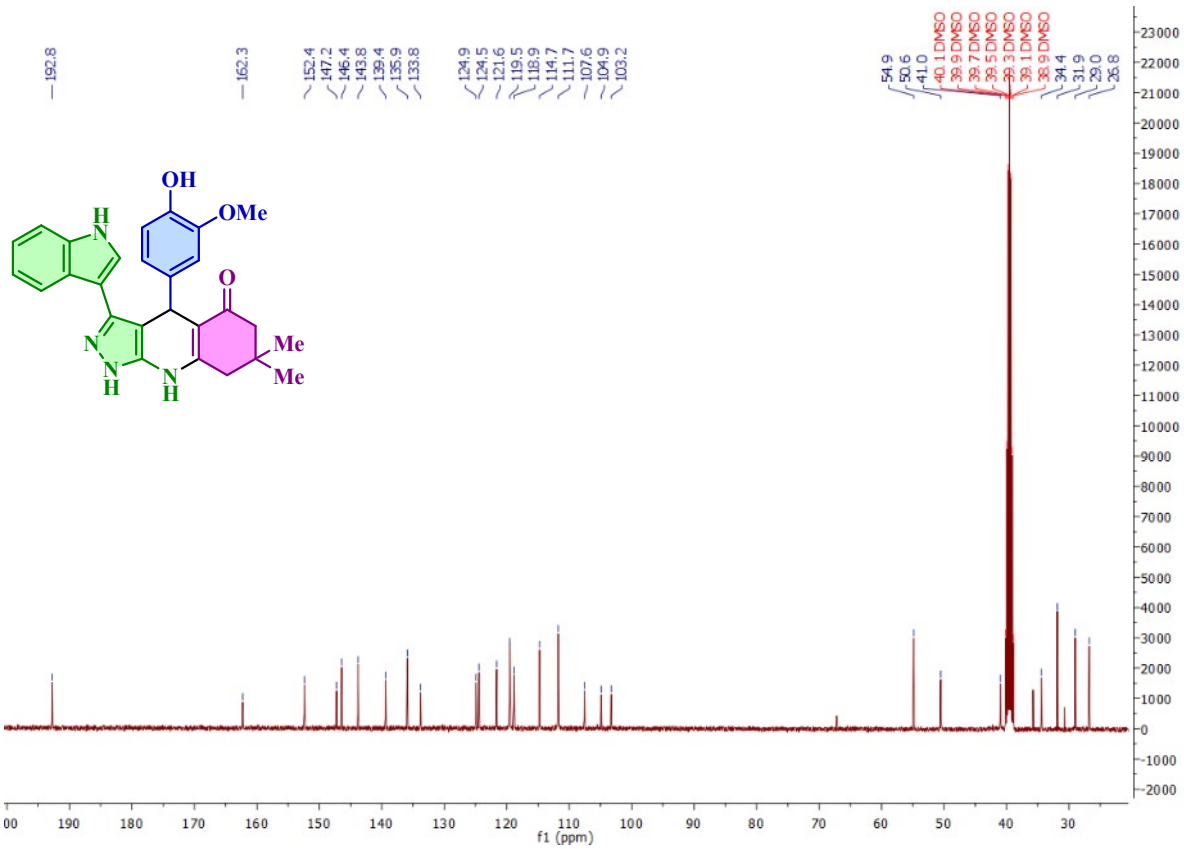
^{13}C-NMR spectrum of 3-(1H-indol-3-yl)-4-(4-isopropylphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC6).



FT-IR spectrum of 4-(4-hydroxy-3-methoxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC7).



[1]H-NMR spectrum of 4-(4-hydroxy-3-methoxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC7).



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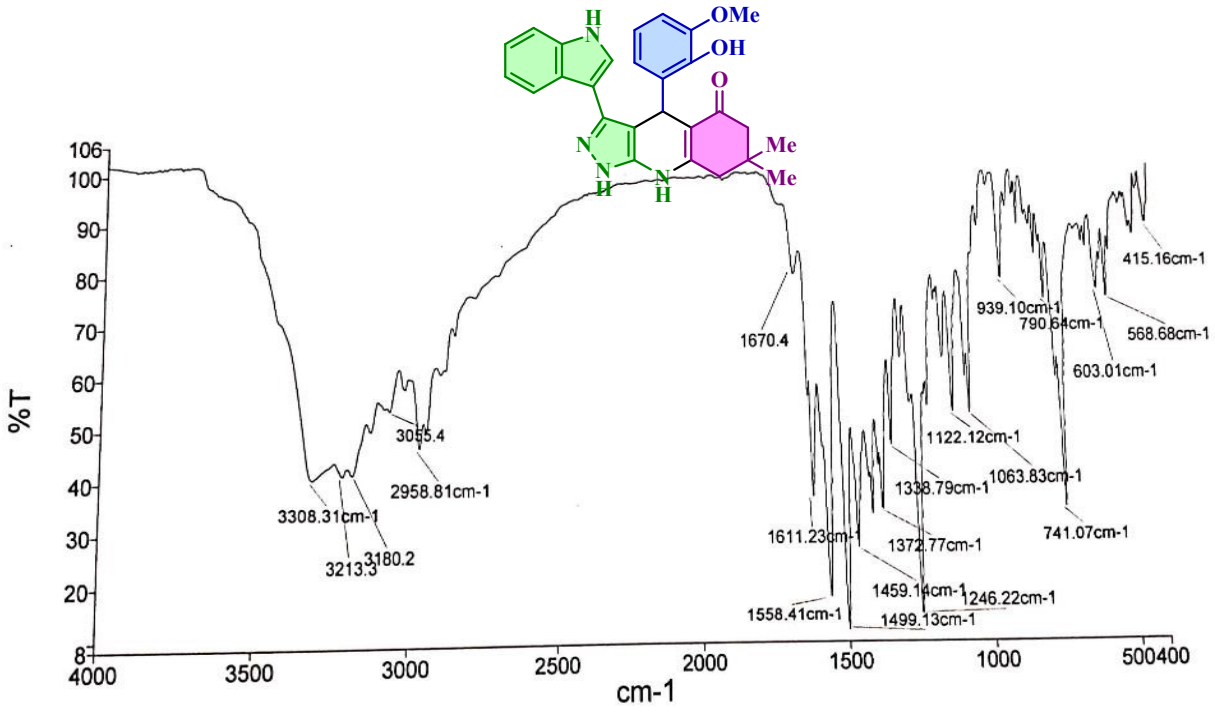
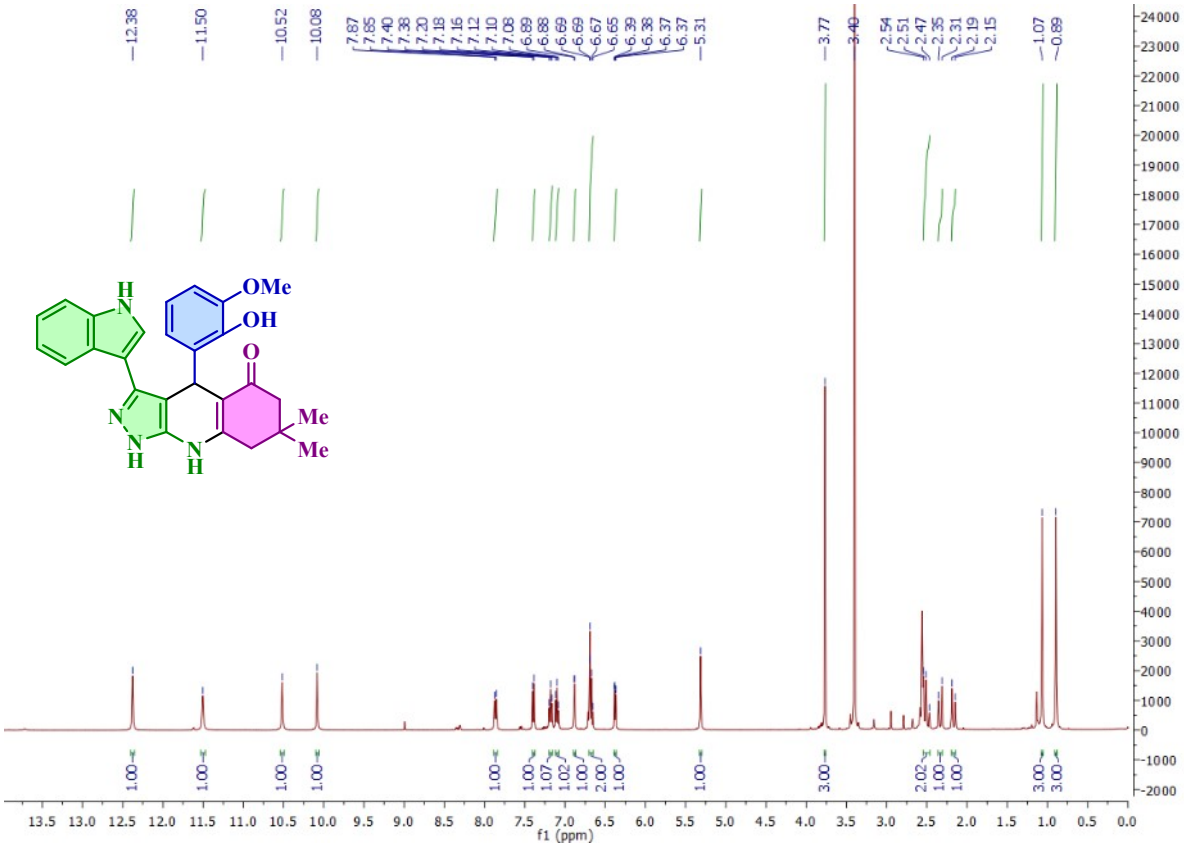


FT-IR spectrum of 4-(2-hydroxy-3-methoxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC8).



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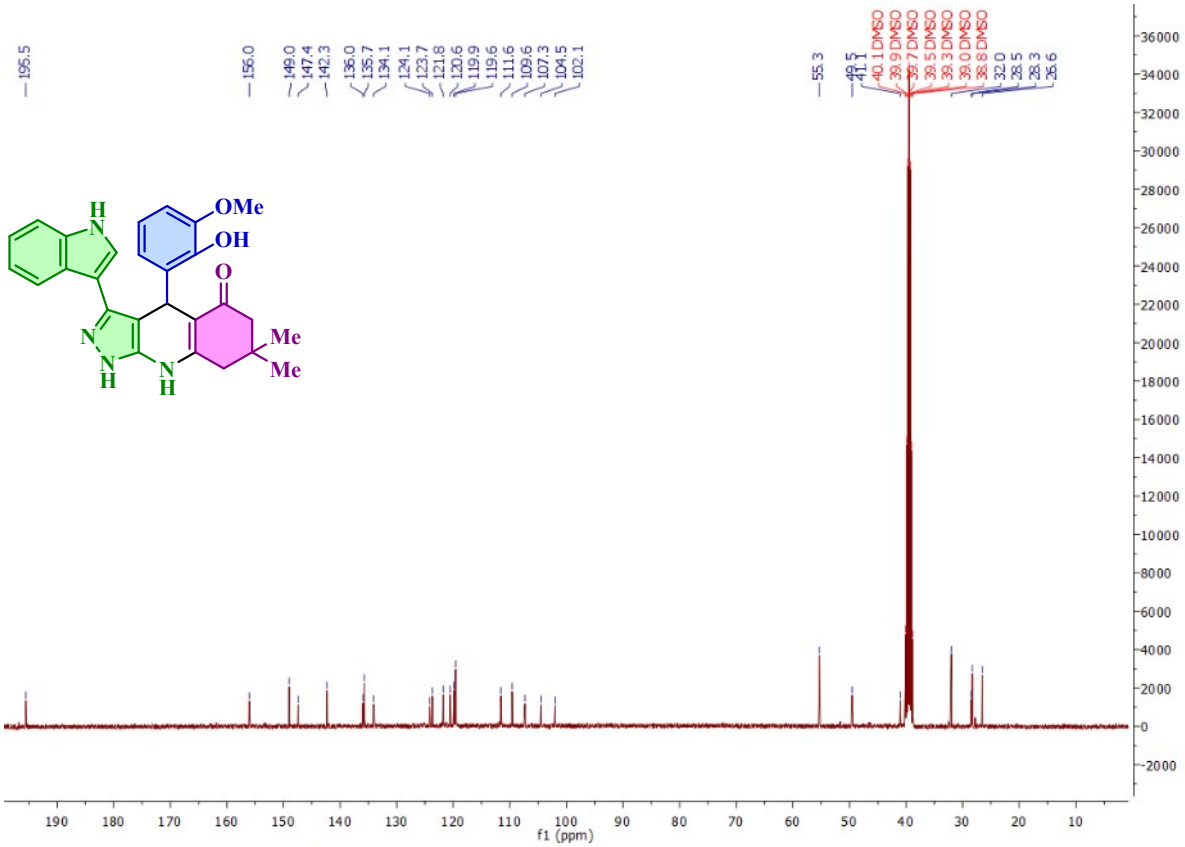

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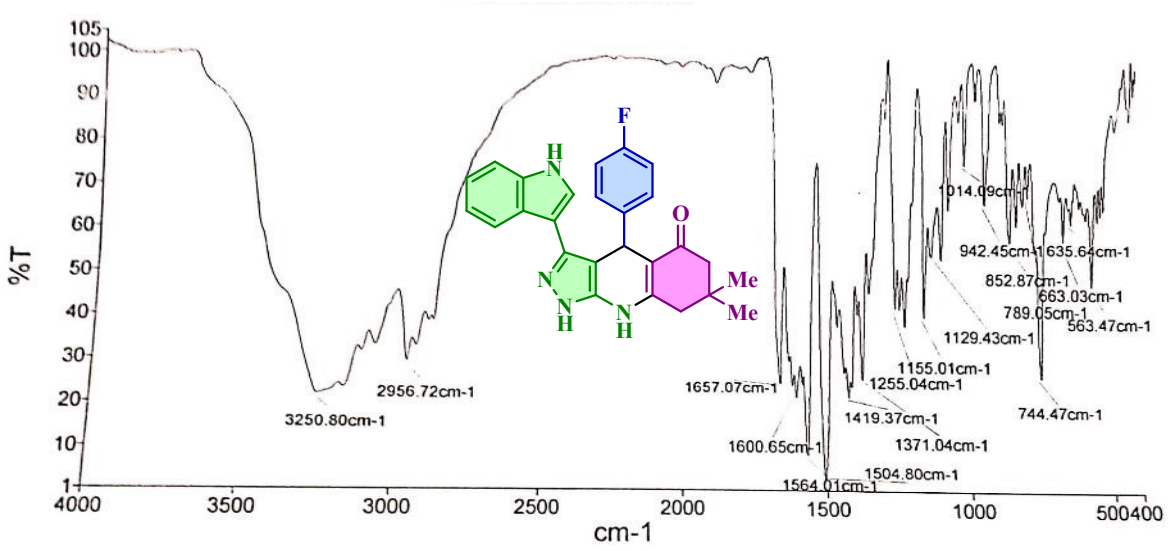
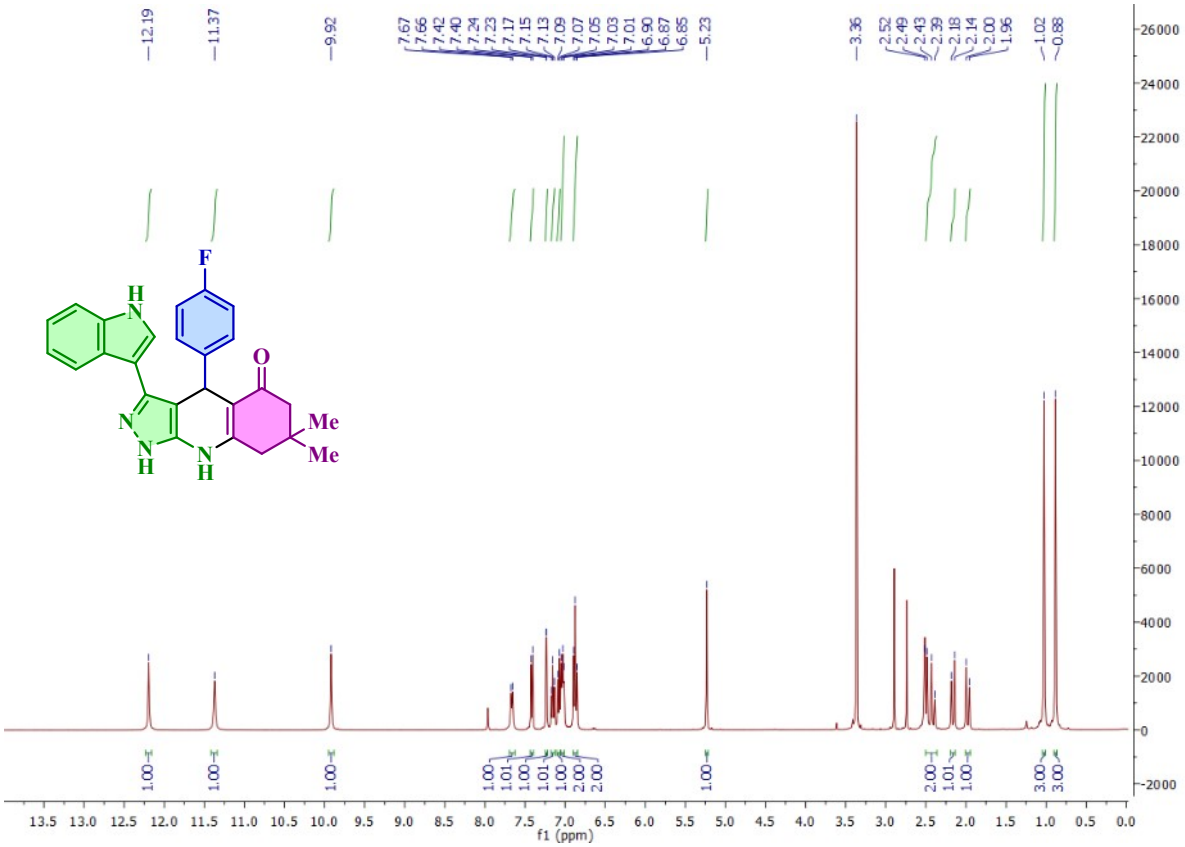


FT-IR spectrum of 4-(4-fluorophenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC9).



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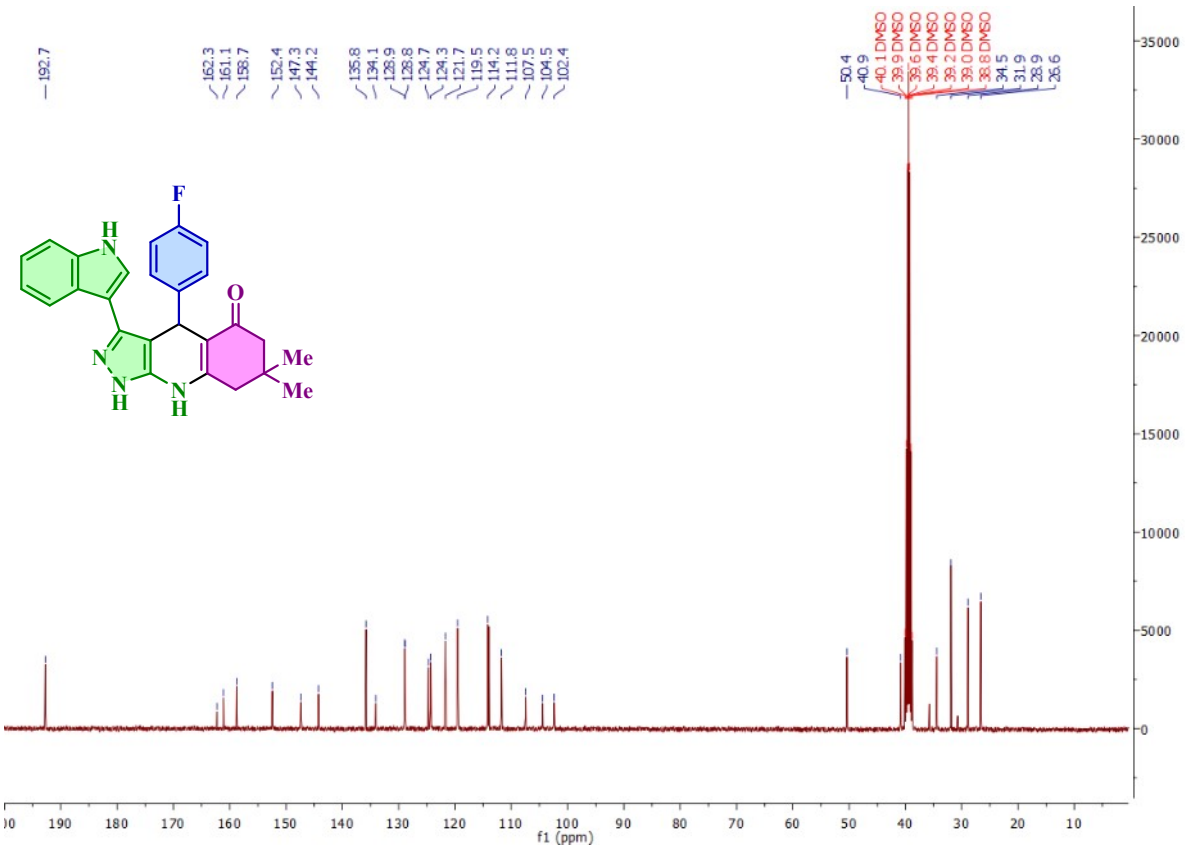

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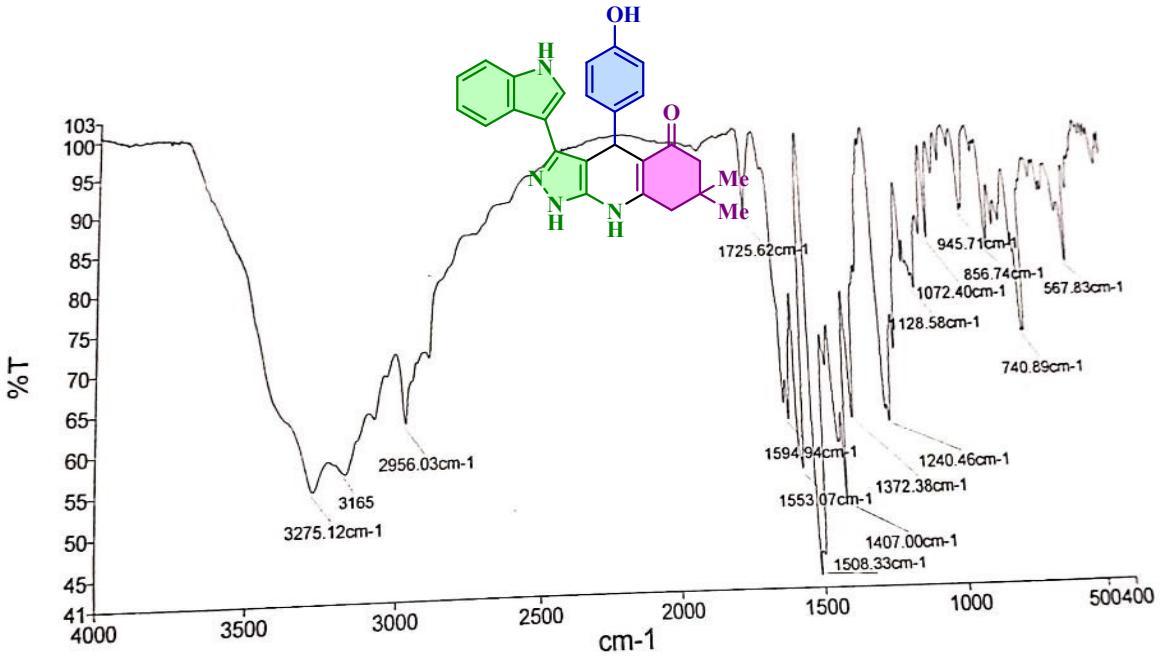
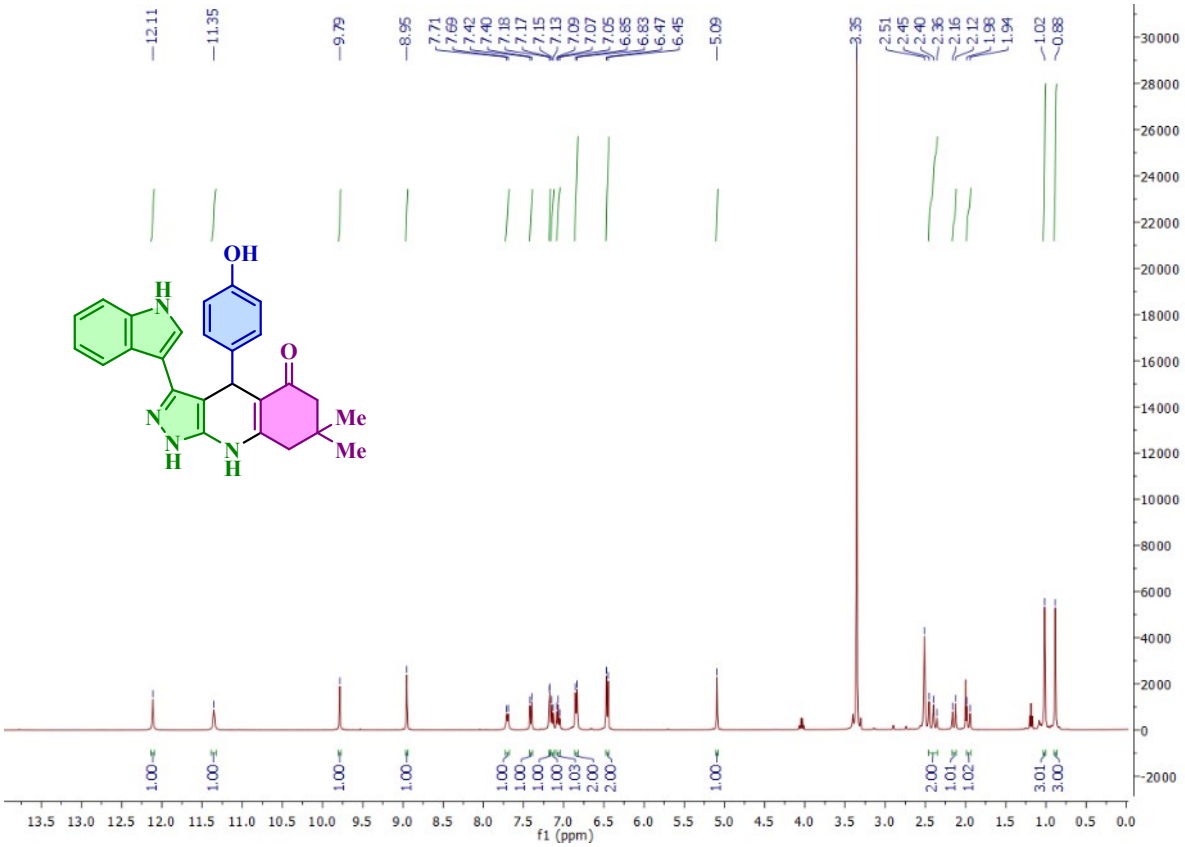


FT-IR spectrum of 4-(4-hydroxyphenyl)-3-(1H-indol-3-yl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FC10).



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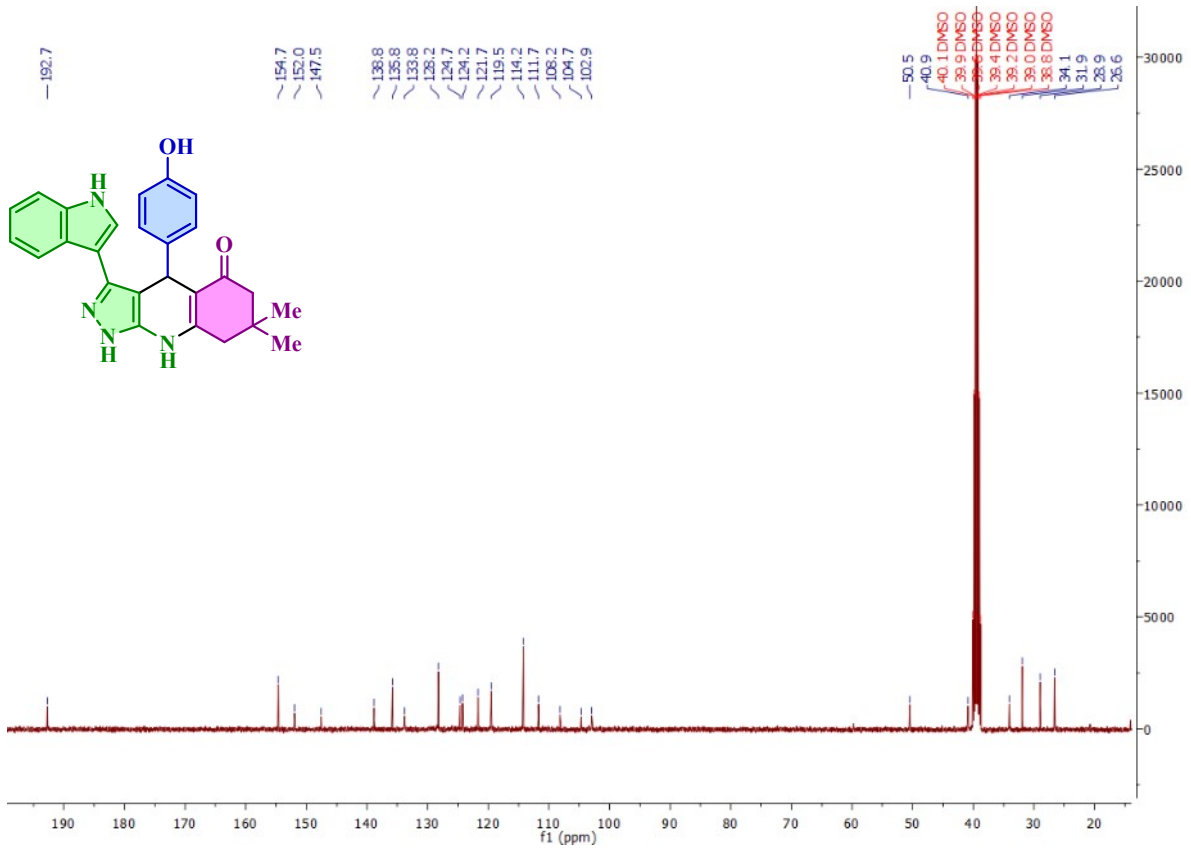

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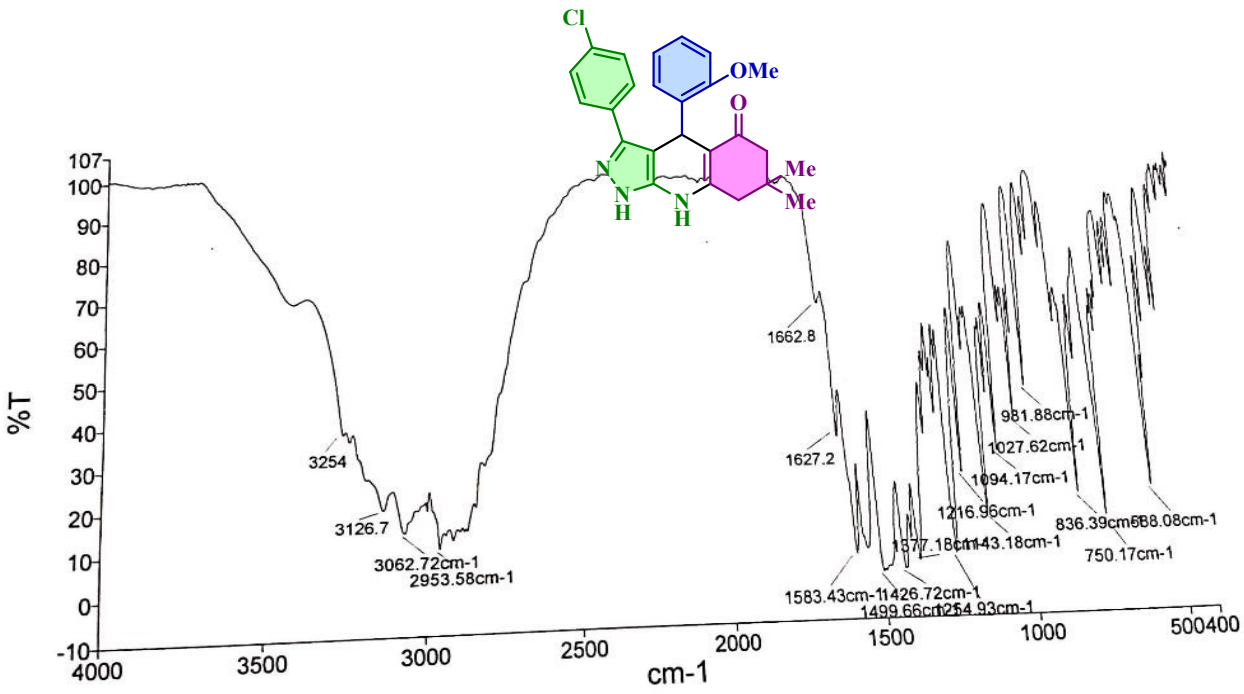
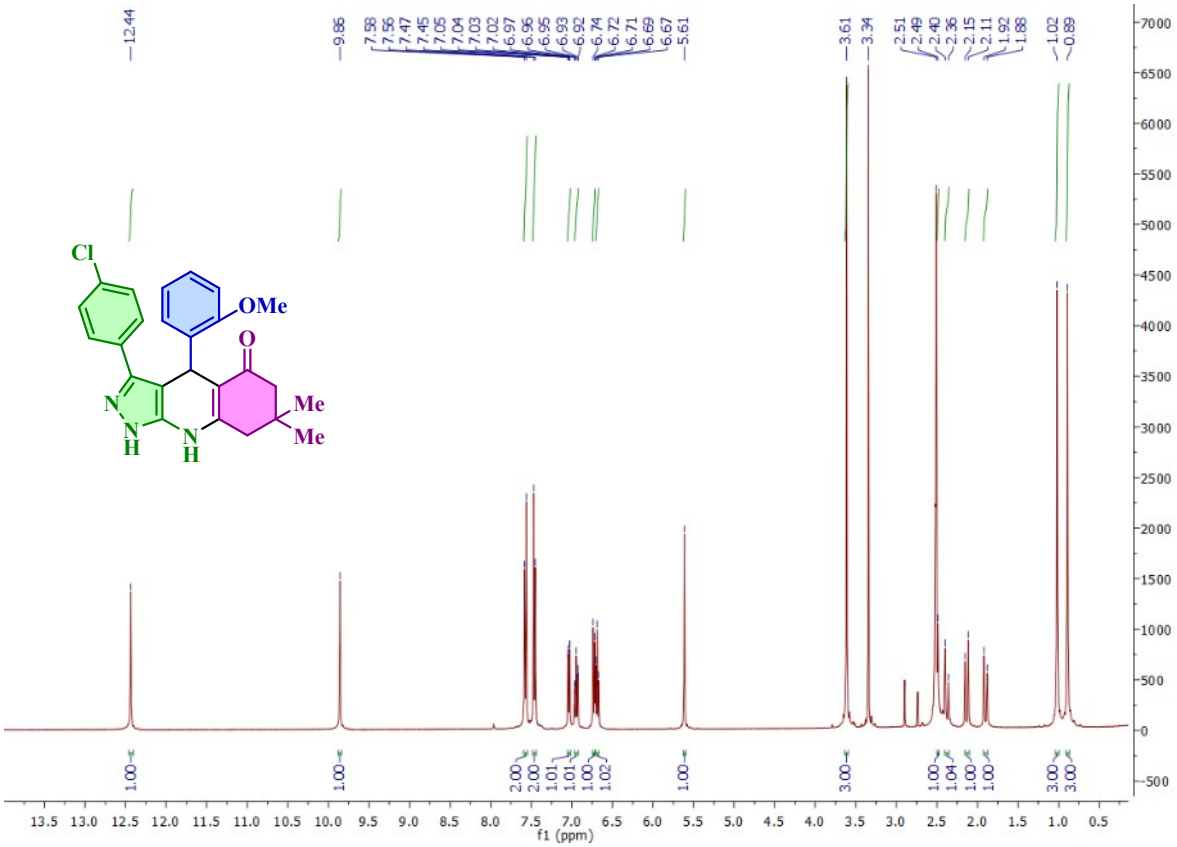
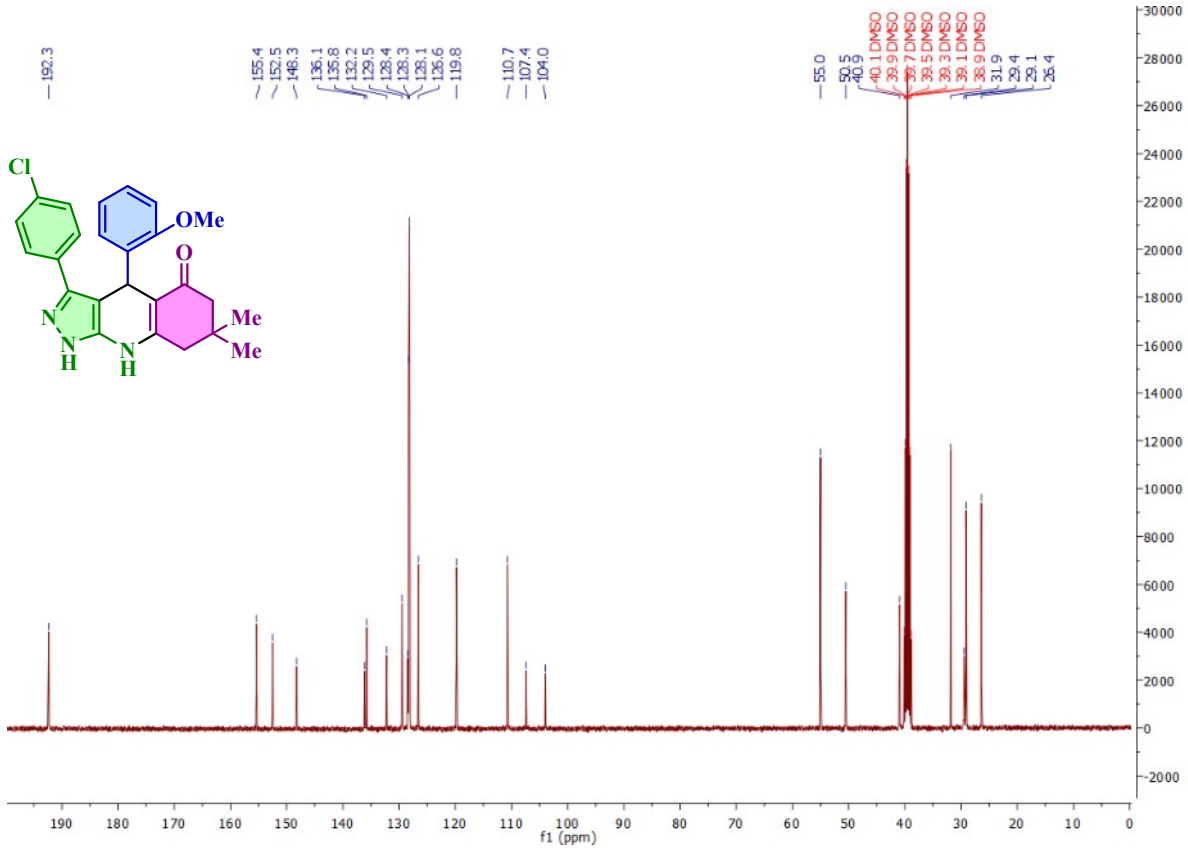


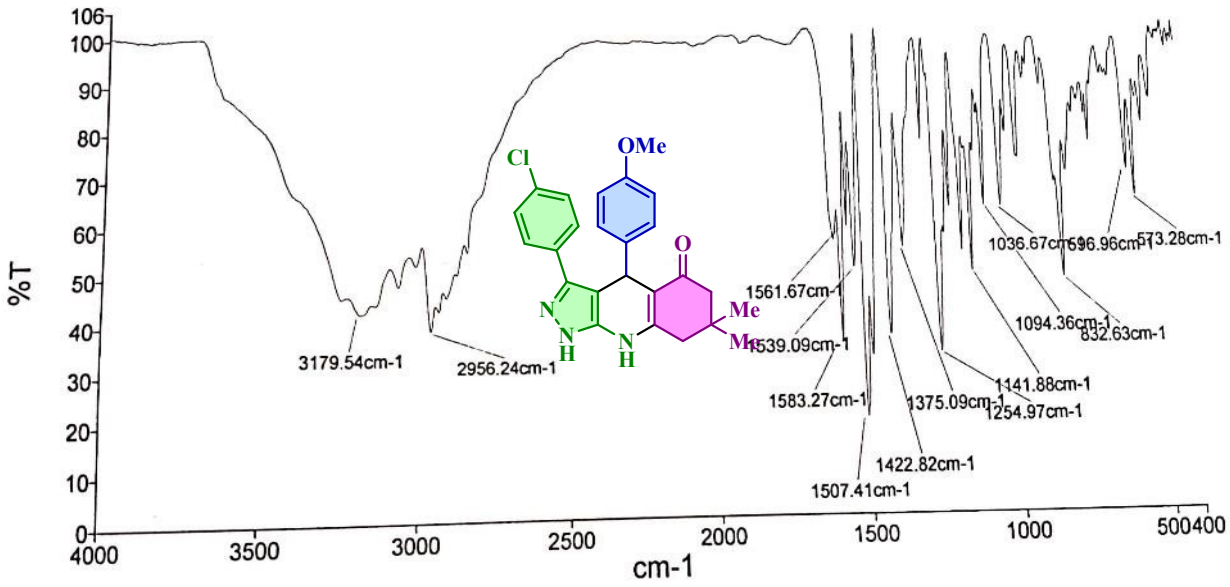
FT-IR spectrum of 3-(4-chlorophenyl)-4-(2-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD1).



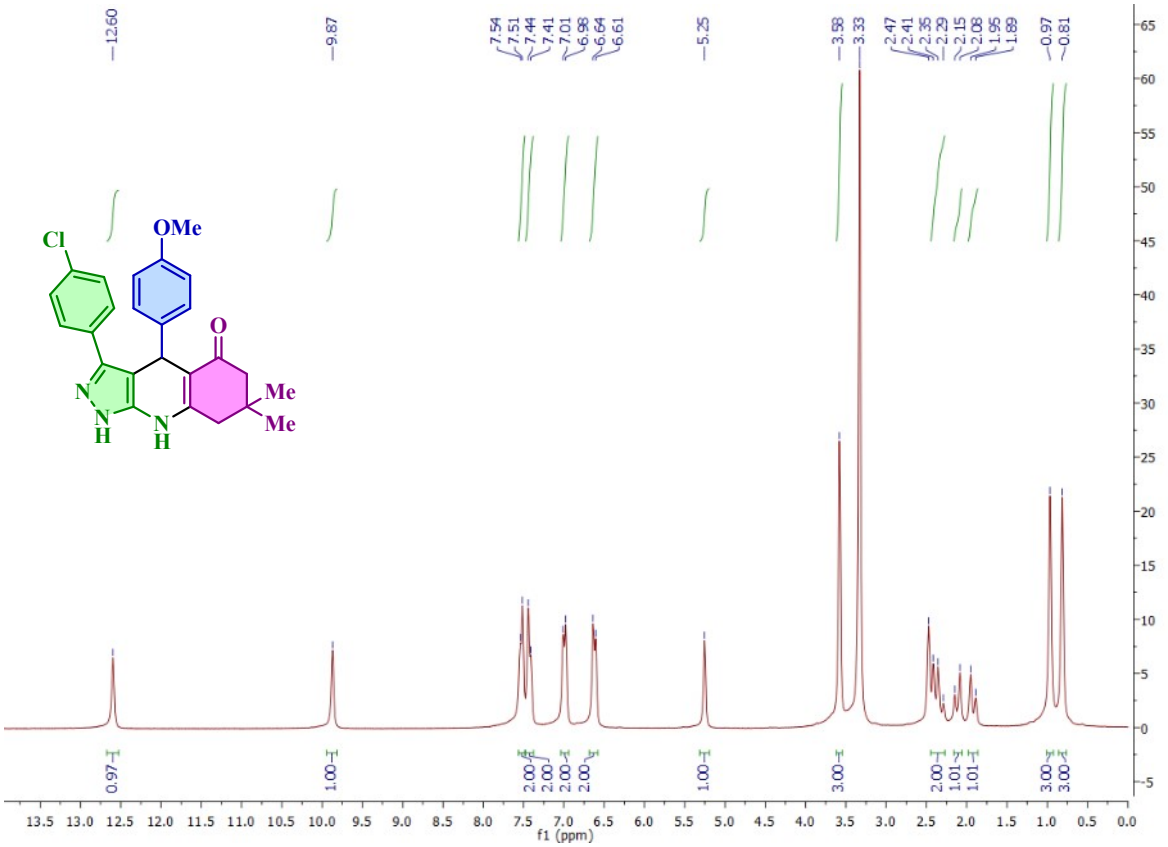
[1]H-NMR spectrum of 3-(4-chlorophenyl)-4-(2-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD1).



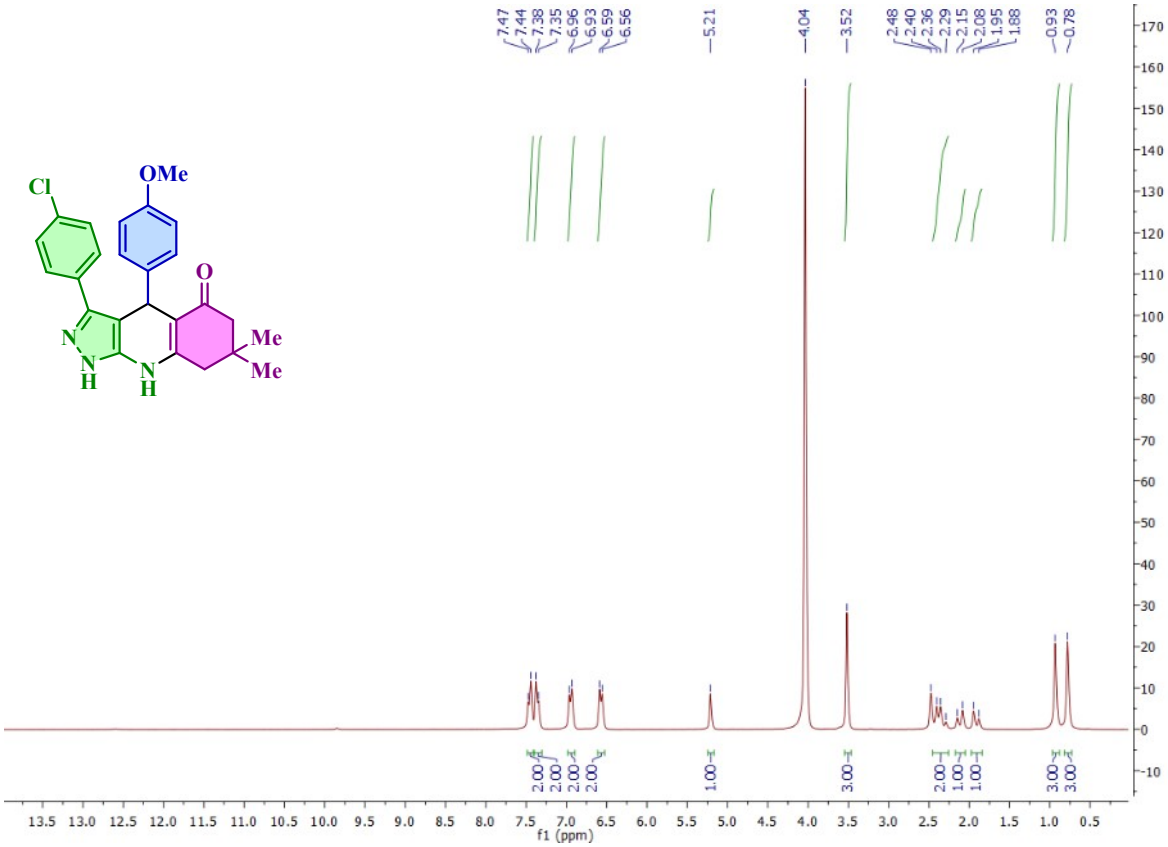
^{13}C-NMR spectrum of 3-(4-chlorophenyl)-4-(2-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD1).



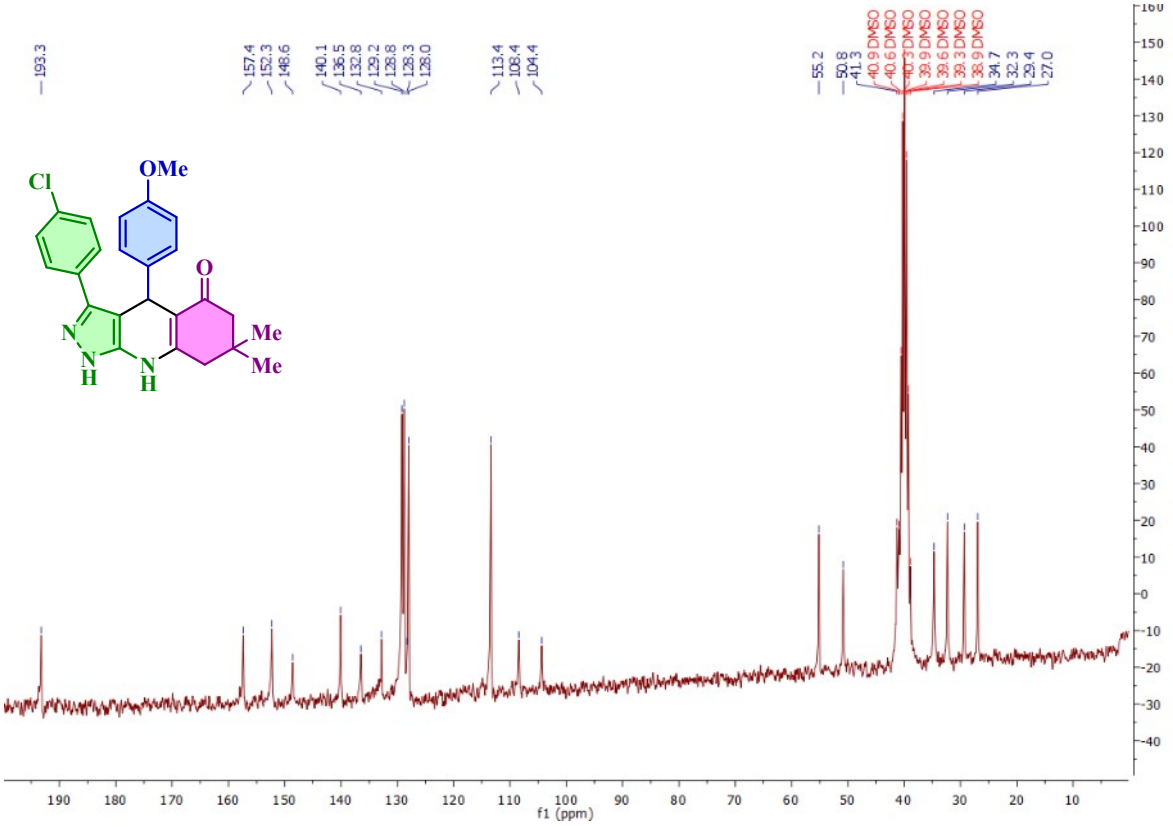
FT-IR spectrum of 3-(4-chlorophenyl)-4-(4-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD2).



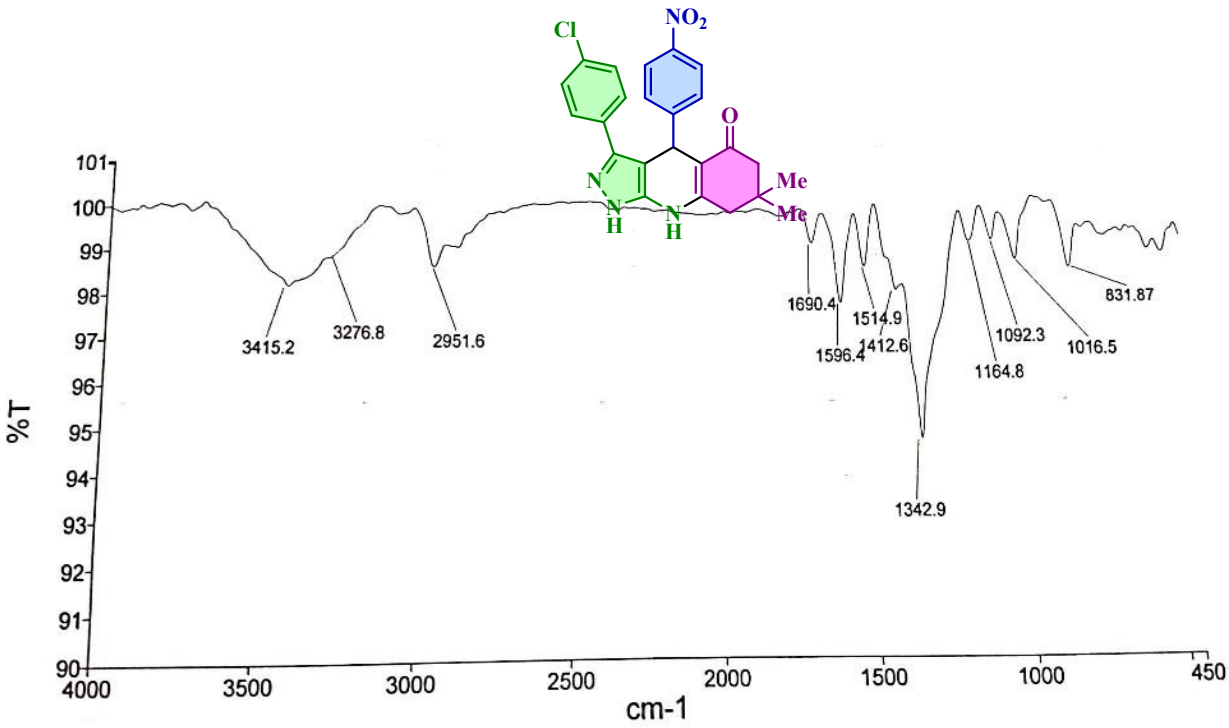
[1]H-NMR spectrum of 3-(4-chlorophenyl)-4-(4-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD2).



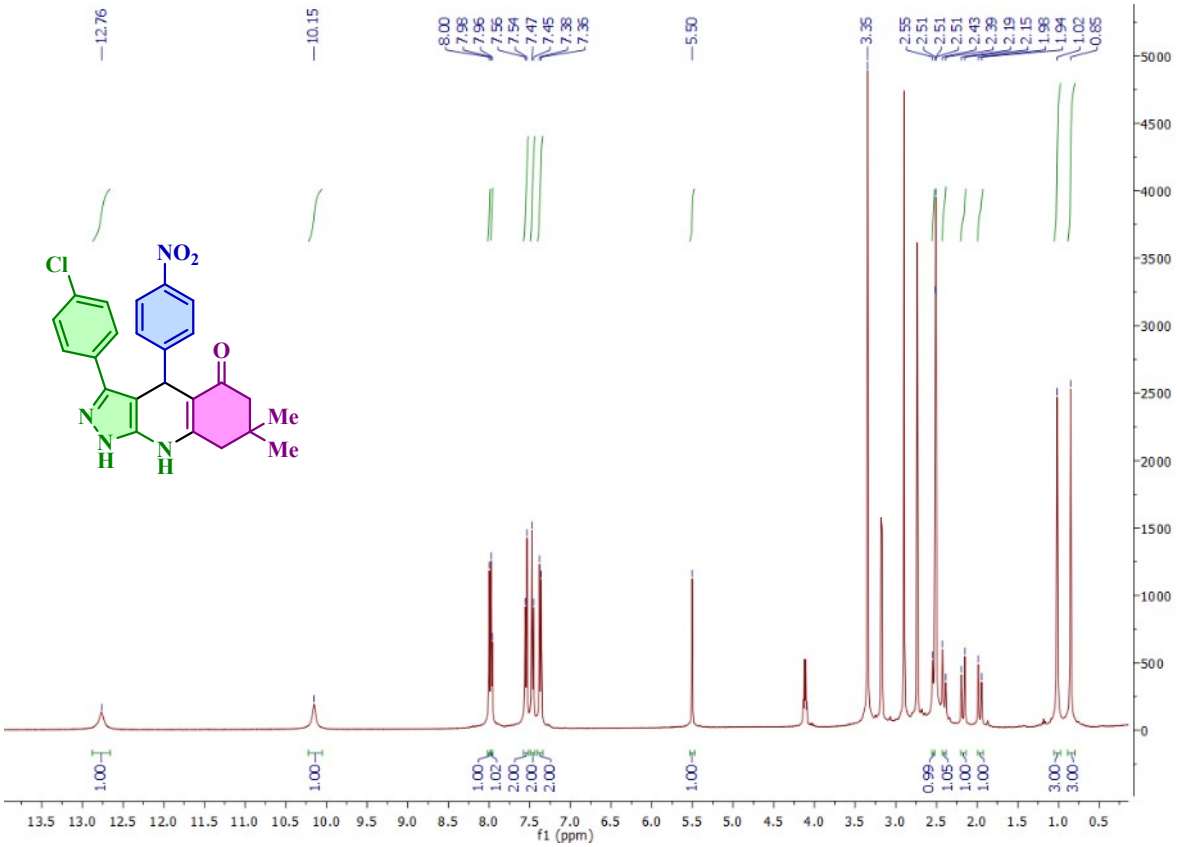
D2O exchanging spectrum of 3-(4-chlorophenyl)-4-(4-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD2).



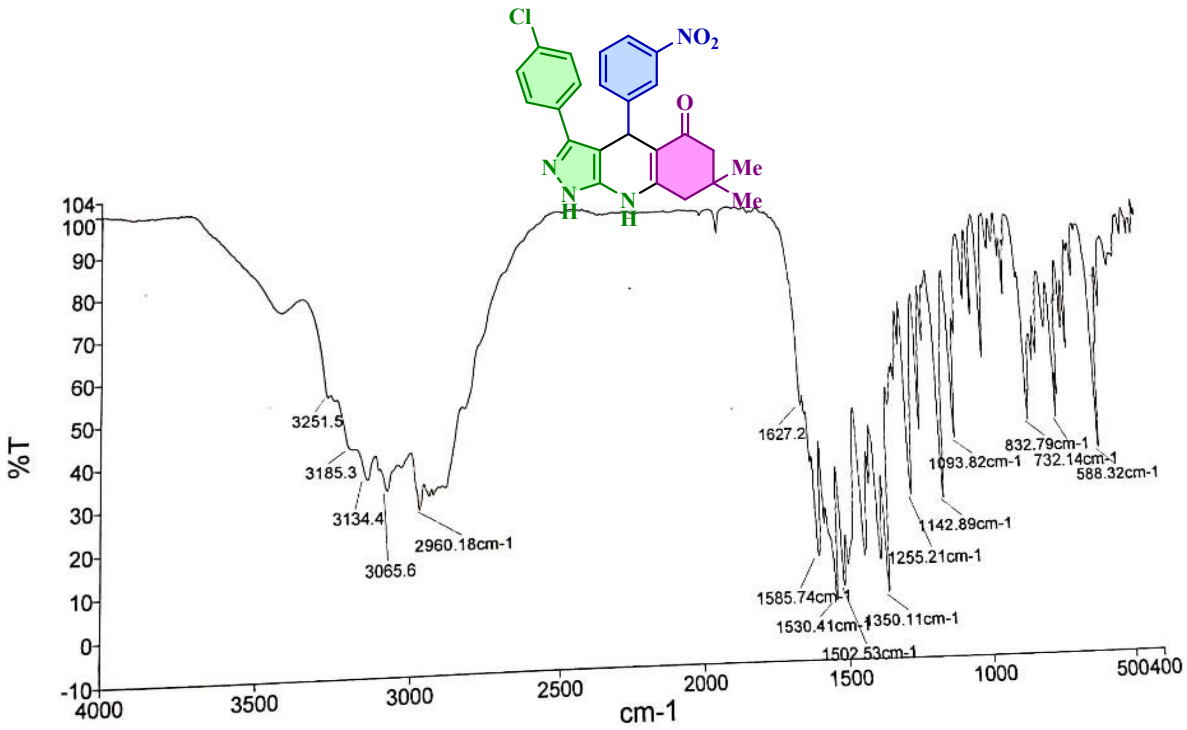
^{13}C-NMR spectrum of 3-(4-chlorophenyl)-4-(4-methoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD2).



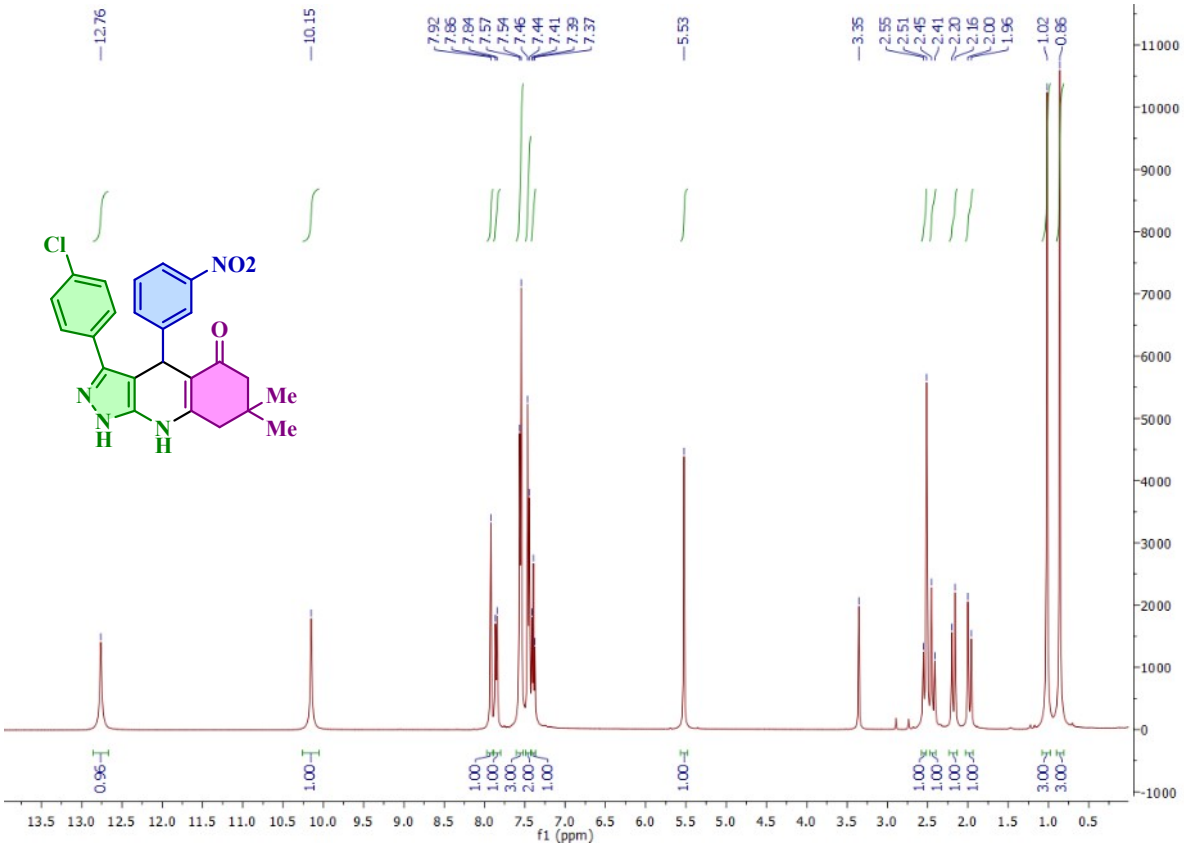
FT-IR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(4-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD3).



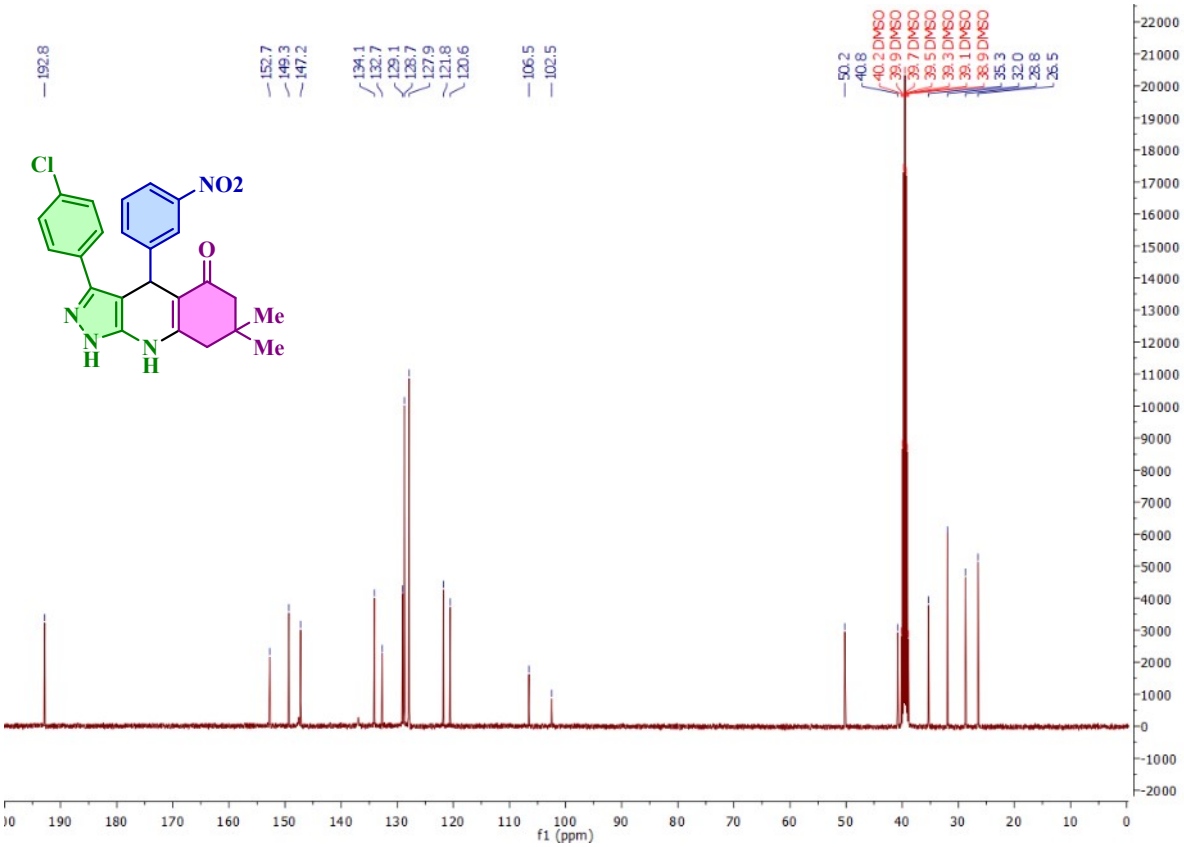
[1]H-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(4-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD3).



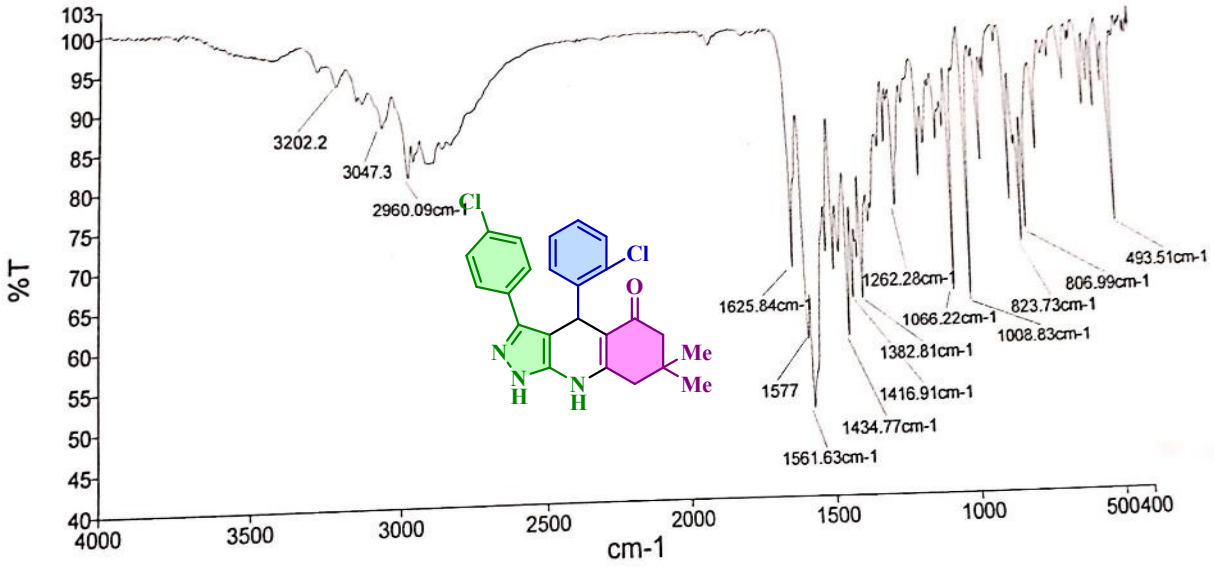
FT-IR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(3-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD4).



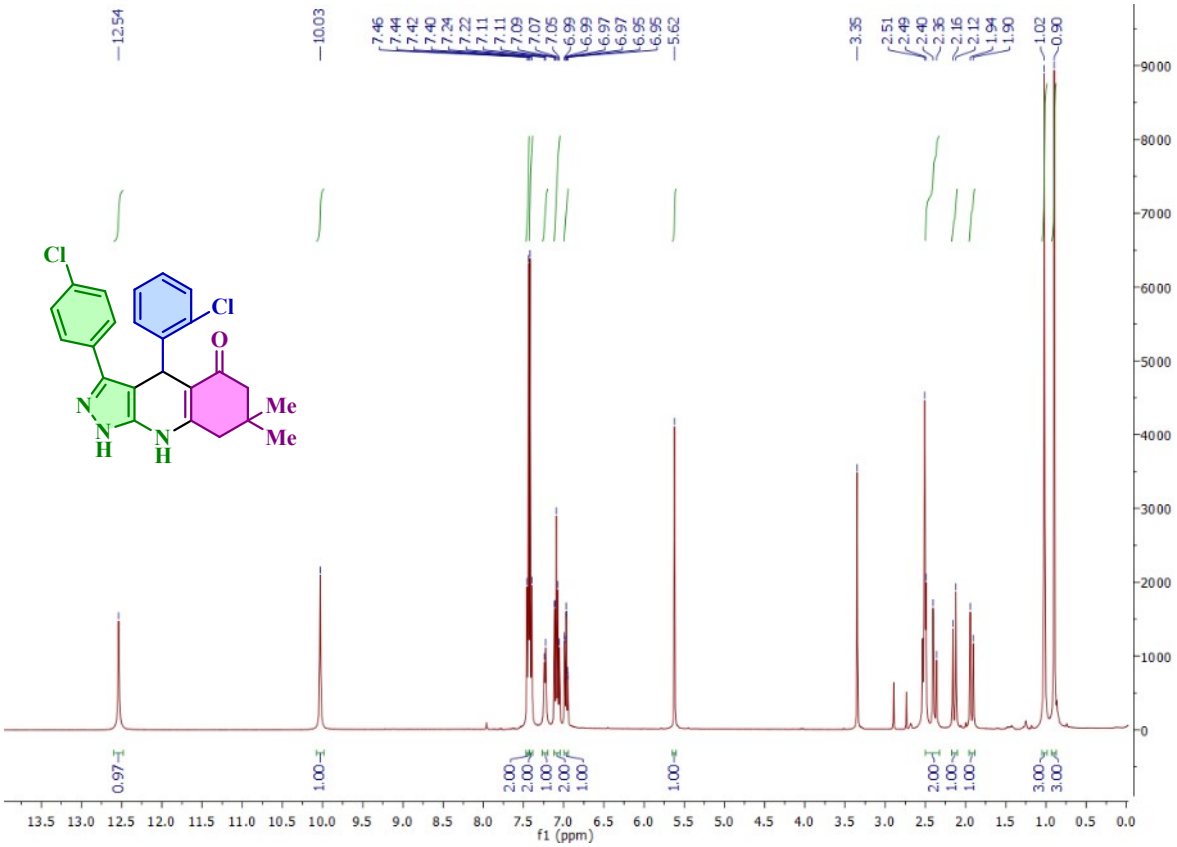
[1]H-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(3-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD4).



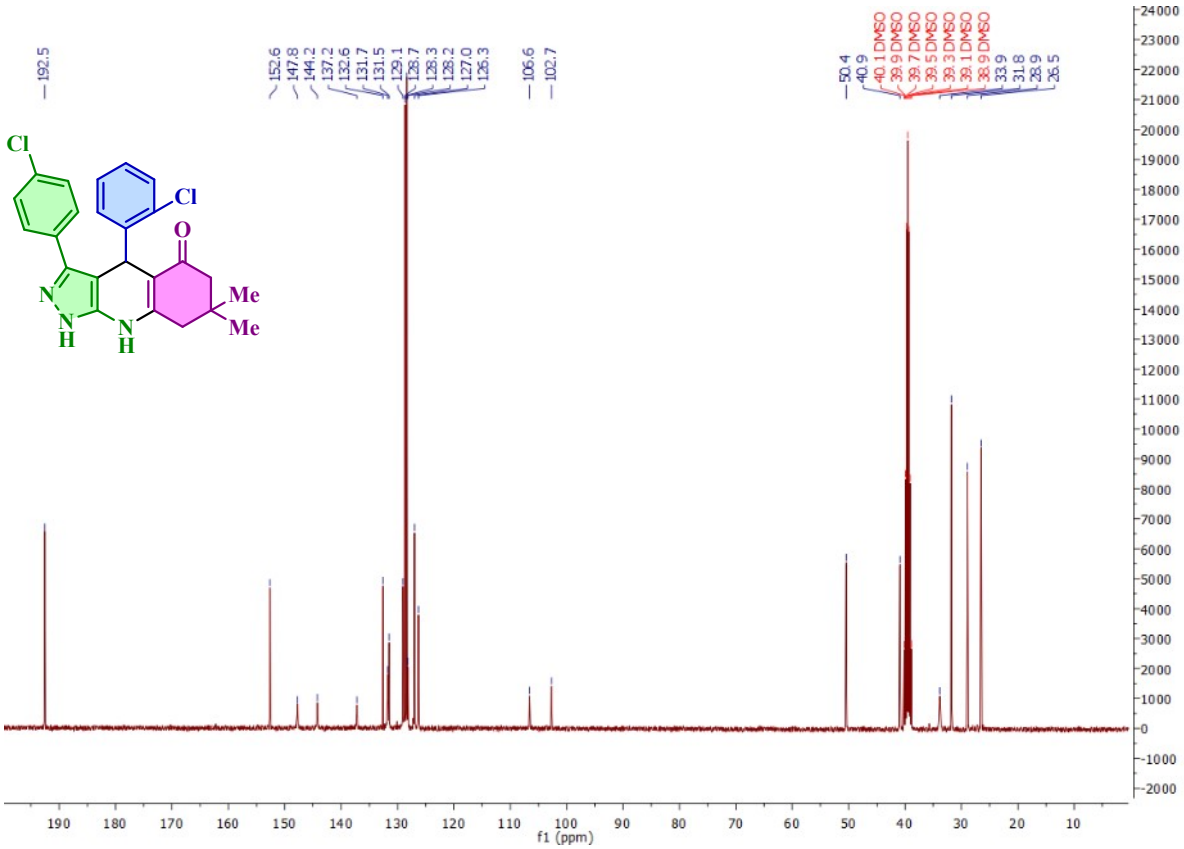
^{13}C-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(3-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD4).



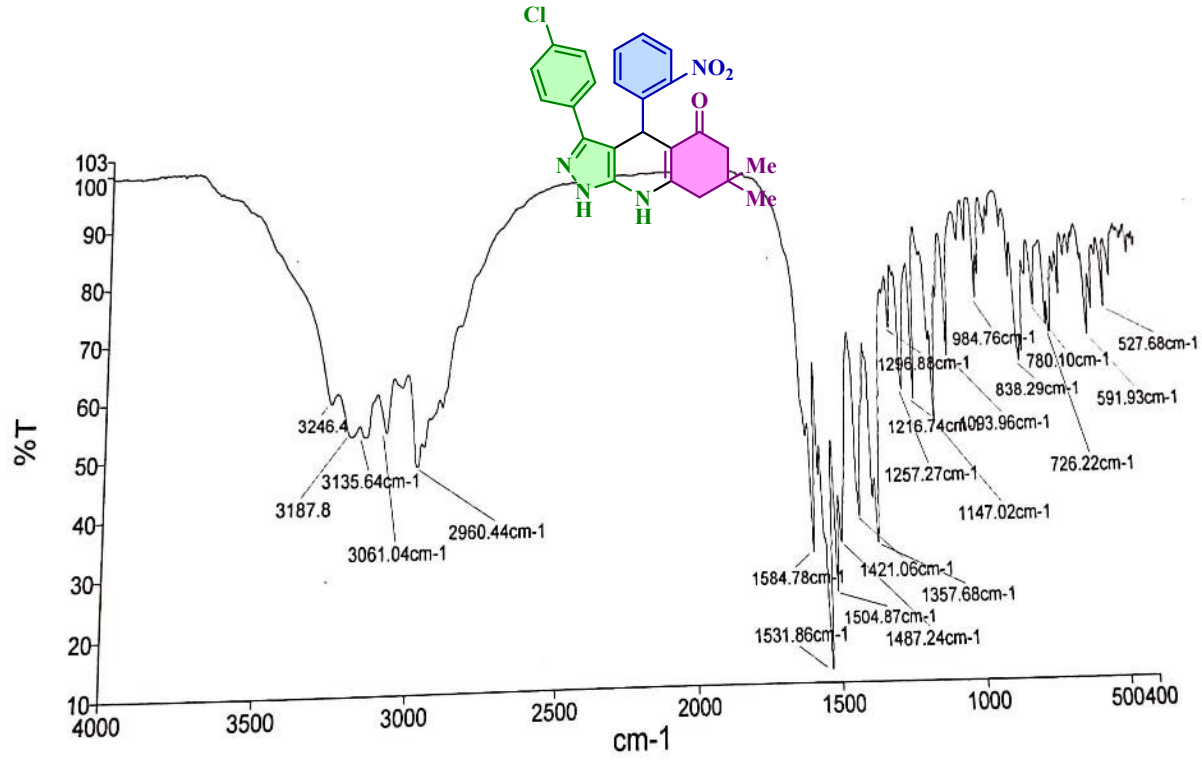
FT-IR spectrum of 4-(2-chlorophenyl)-3-(4-chlorophenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD5).



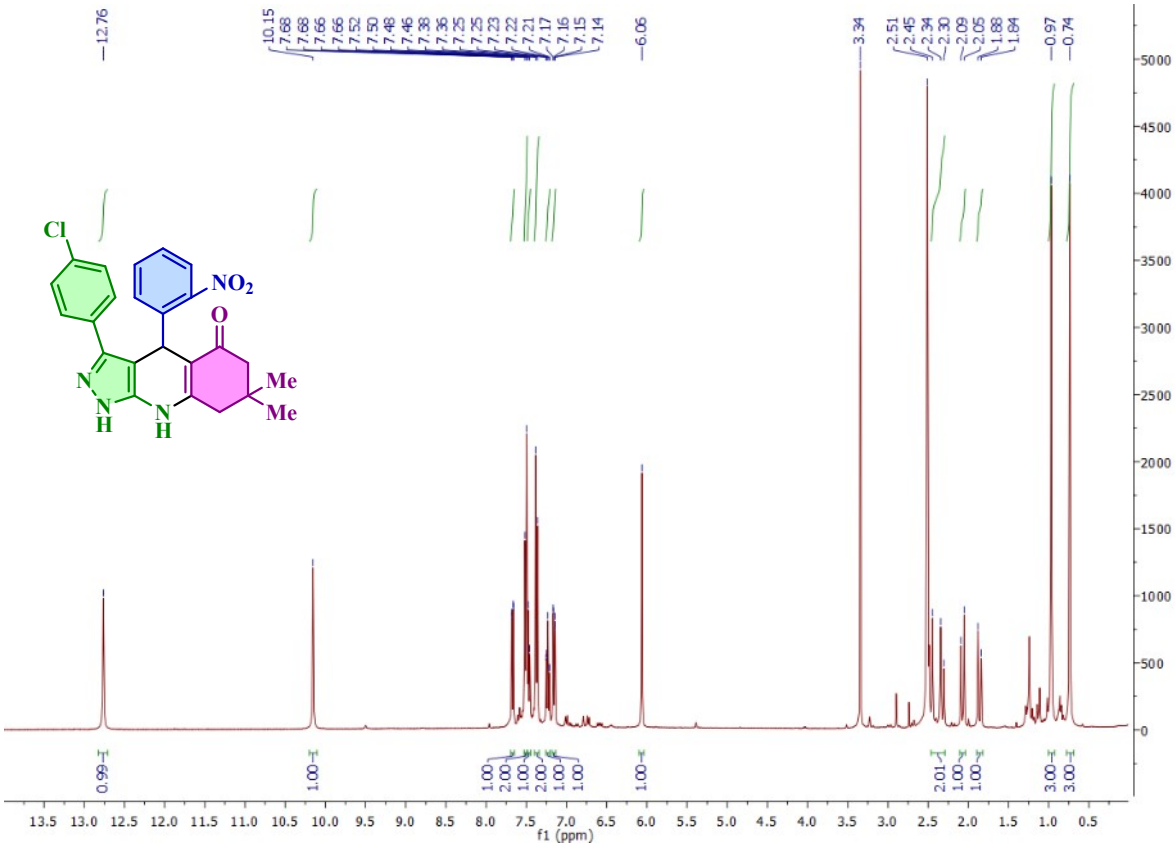
[1]H-NMR spectrum of 4-(2-chlorophenyl)-3-(4-chlorophenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD5).



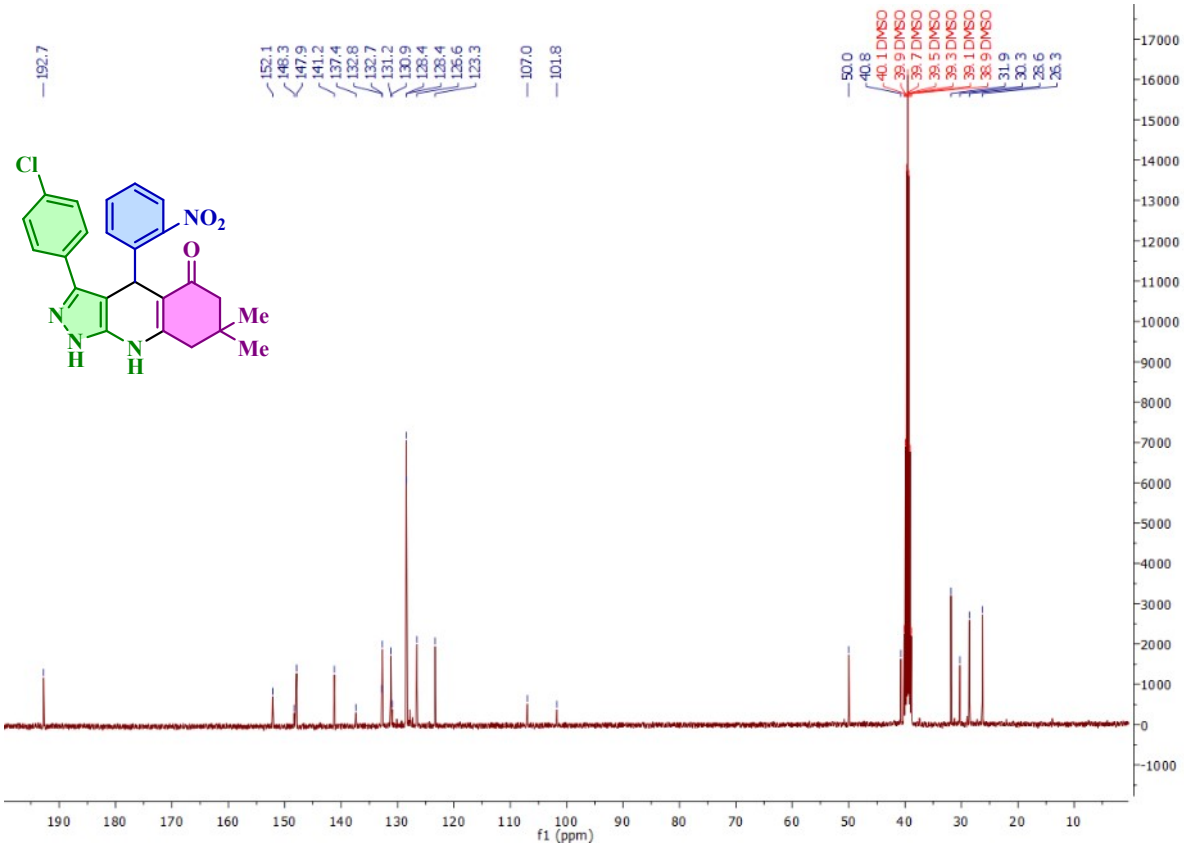
^{13}C-NMR spectrum of 4-(2-chlorophenyl)-3-(4-chlorophenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD5).



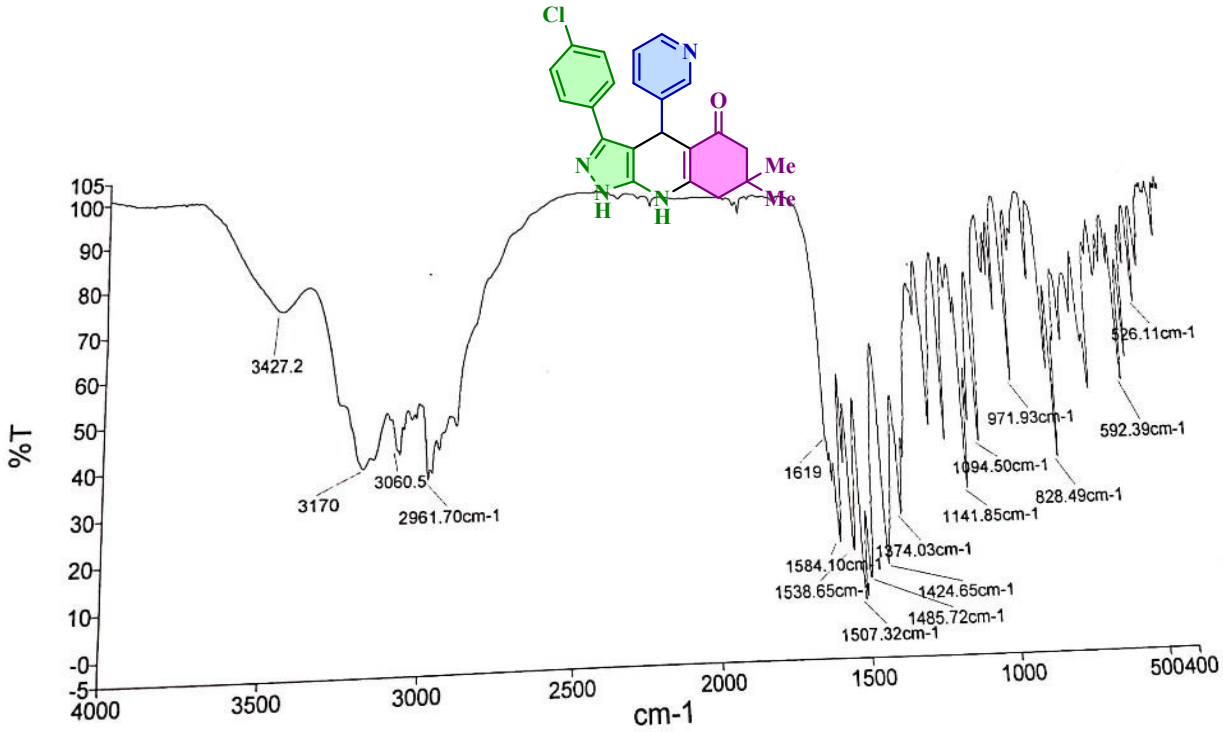
FT-IR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(2-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD6).



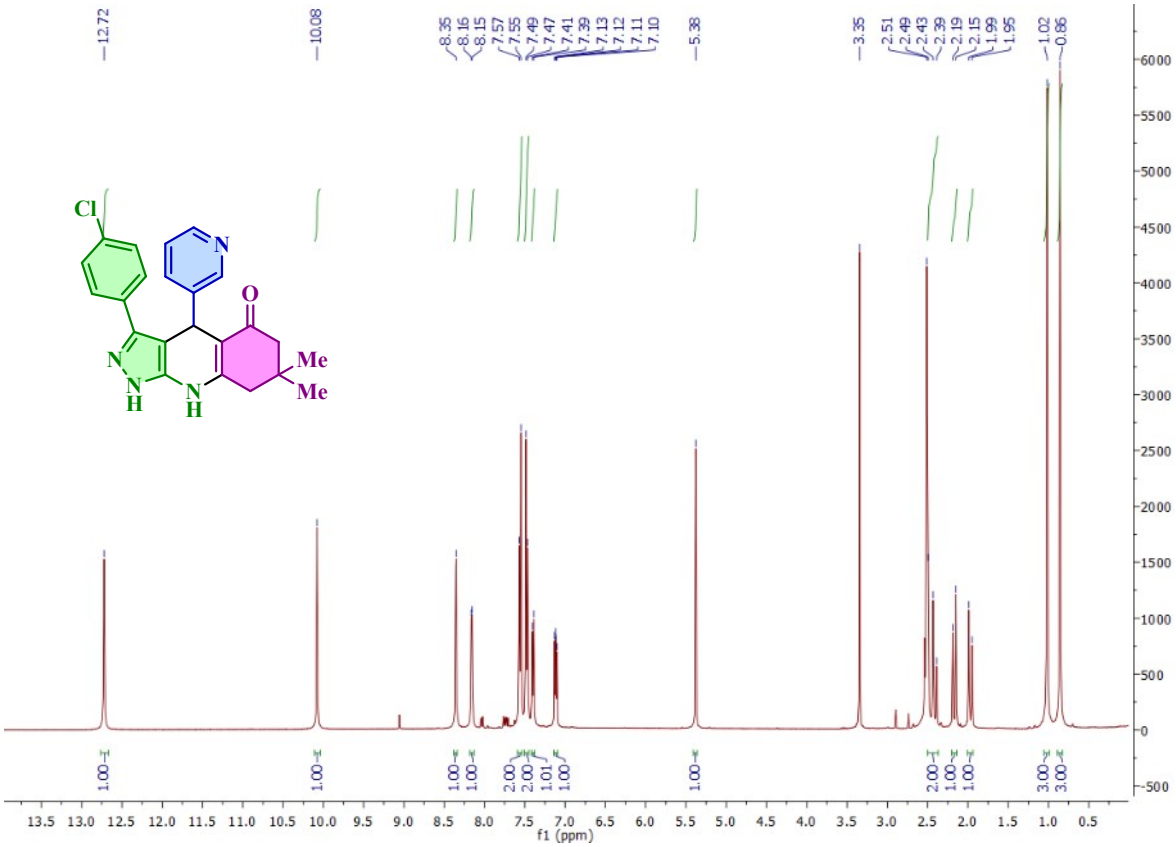
[1]H-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(2-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD6).



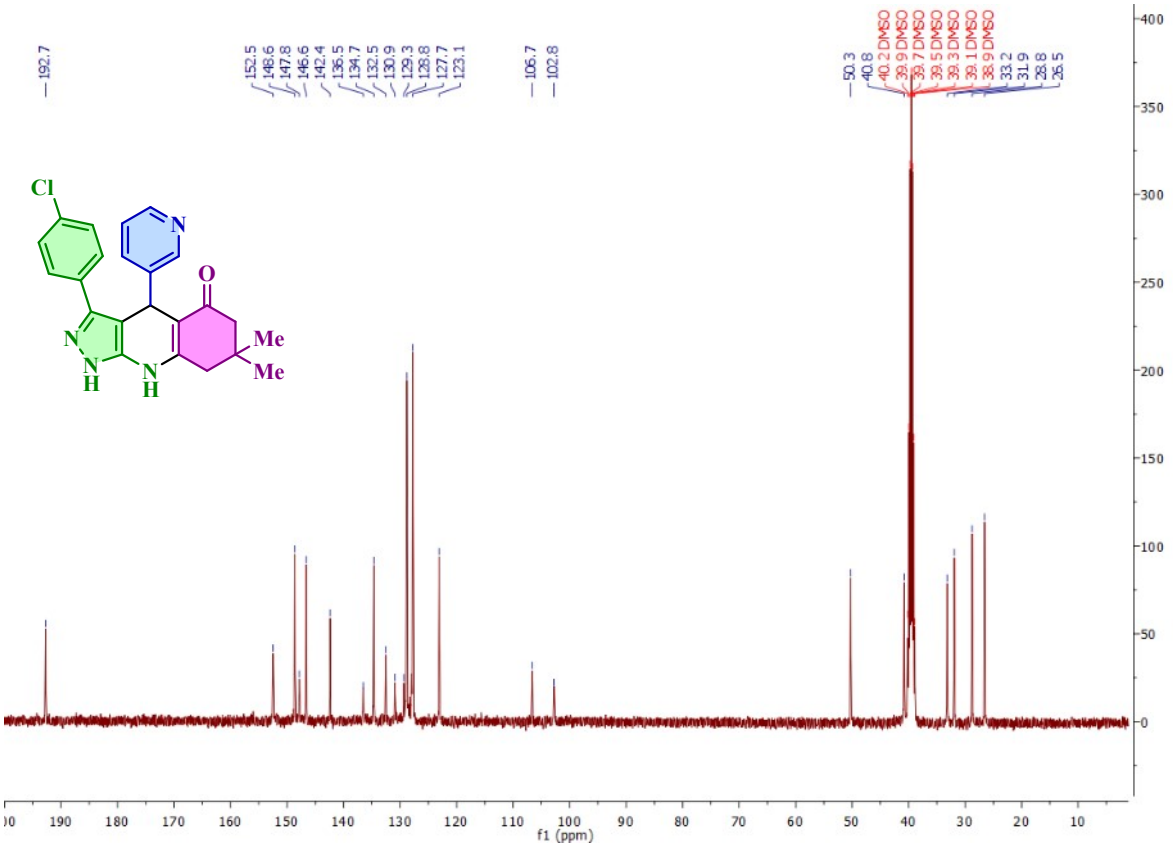
^{13}C-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(2-nitrophenyl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD6).



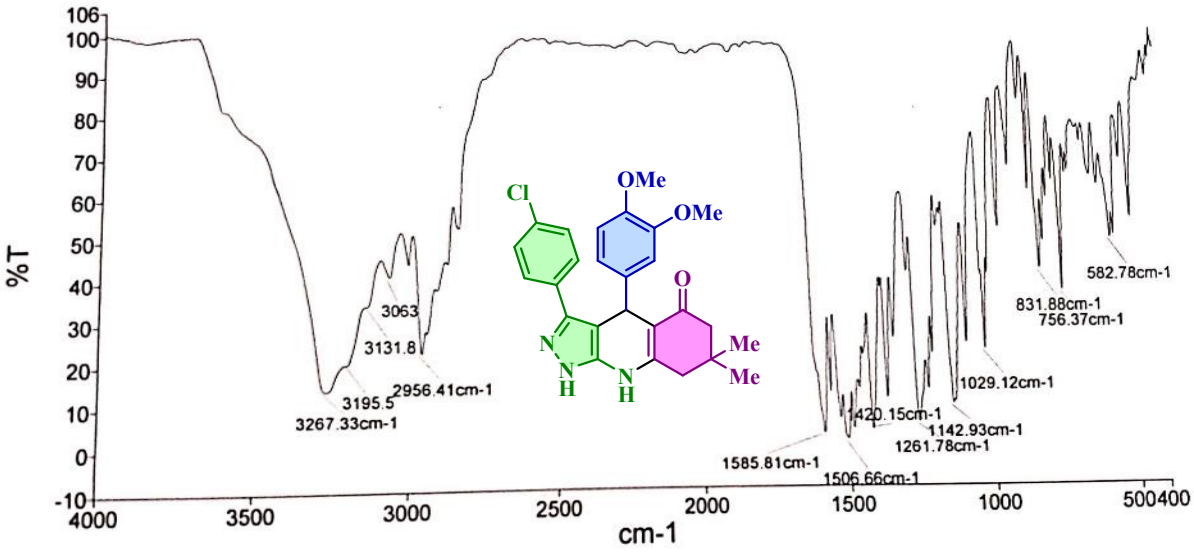
FT-IR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(pyridin-3-yl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD7).



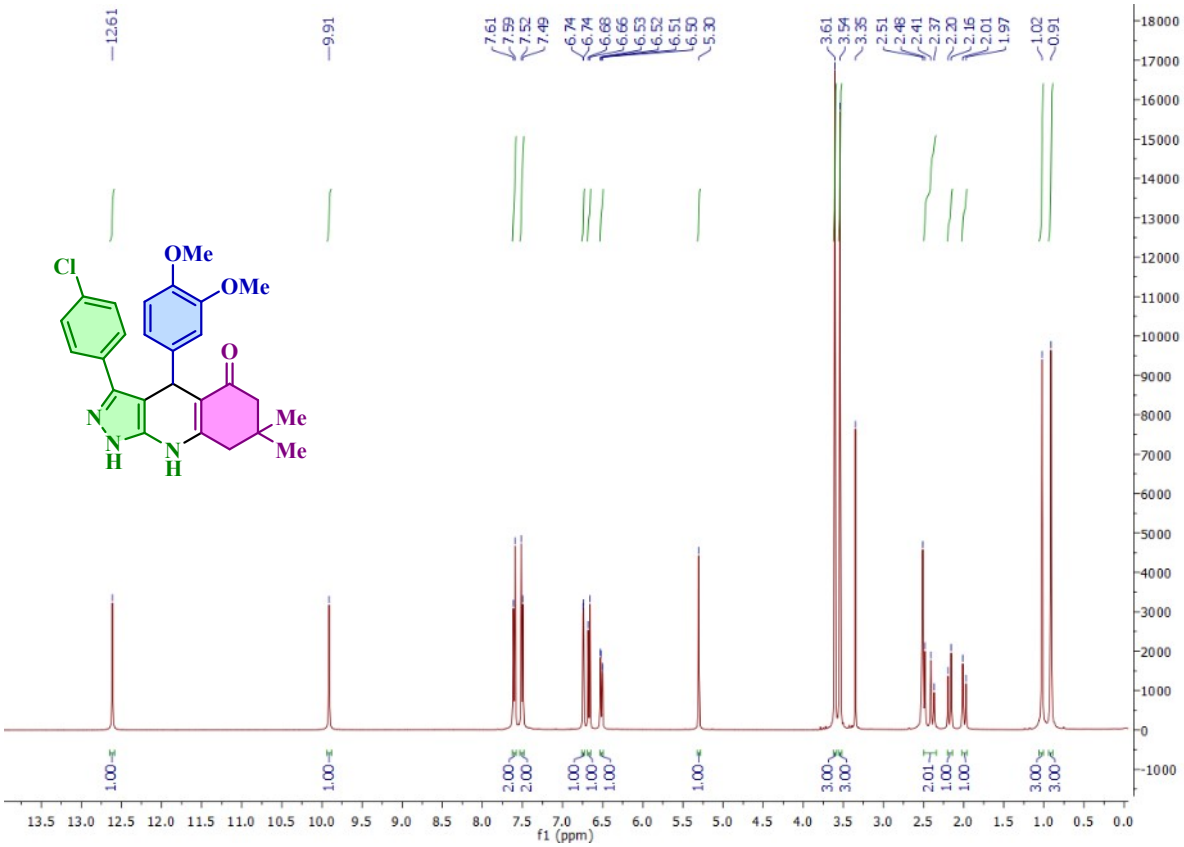
[1]H-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(pyridin-3-yl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD7).



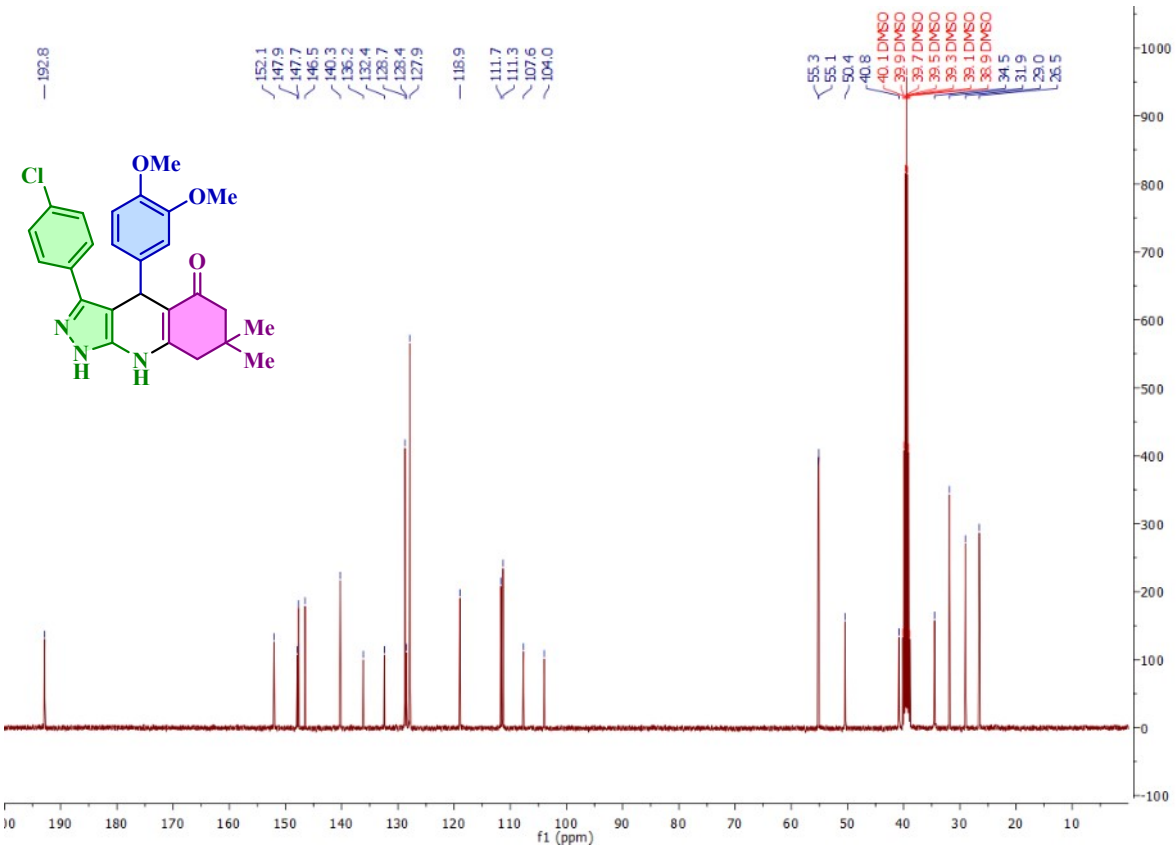
^{13}C-NMR spectrum of 3-(4-chlorophenyl)-7,7-dimethyl-4-(pyridin-3-yl)-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD7).



FT-IR spectrum of 3-(4-chlorophenyl)-4-(3,4-dimethoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD8).



[1]H-NMR spectrum of 3-(4-chlorophenyl)-4-(3,4-dimethoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD8).



^{13}C-NMR spectrum of3-(4-chlorophenyl)-4-(3,4-dimethoxyphenyl)-7,7-dimethyl-1,4,6,7,8,9-hexahydro-5H-pyrazolo[3,4-b]quinolin-5-one (FD8).