SUPPORTING DATA

β-cyclodextrin: a green supramolecular catalyst assisted eco-friendly one-pot three-component synthesis of biologically active substituted pyrrolidine-2-one

Subhankar Paul[a], Sharmistha Das[a], Bijeta Mitra[a], Gyan Chandra Pariyar[b], and Pranab Ghosh[a*]

[a]Department of Chemistry, University of North Bengal, District-Darjeeling, West Bengal, India.

[b]Department of Food Technology, University of North Bengal, District-Darjeeling, West Bengal, India.

Tel.: +91 (0353) 2776381; fax: +91 (0353) 2699001.

E-mail address: pizy12@yahoo.com (P. Ghosh).

Table of contents		**Page no.**

I. General Information

All the easily available reagents, precursors and solvents used for our research work were purchased from the commercial suppliers and were used without further purification. The solvents used for TLC and column chromatography i.e., petroleum ether and ethyl acetate were used after purification by distillation. ^{1}H-NMR, ^{13}C-NMR, and ^{19}F-NMR were recorded using 400 MHz, 100 MHz and 376 MHz respectively on Bruker AV 400 NMR spectrometer and Bruker AV 300 NMR Spectrometer using TMS (Tetramethyl silane) as the internal standard. The splitting patterns of protons were described as s (singlet), d (doublet), t (triplet) and m (multiples).

II. General procedure for the synthesis of Methyl/Ethyl 2-aryl-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4a-4i) & (6a-6e) and Methyl/Ethyl 1-aryl-4-hydroxy-5-oxo-2-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (5a-5d) & (7a-7b)

β-Cyclodextrin (10 mg) and 2 ml of water-ethanol (1:1 ratio) were added to the mixture of aldehydes (1 mmol), amines (1 mmol) and dimethylacetylenedicarboxylate (1.2 mmol) or diethylacetylenedicarboxylate (1.2 mmol) contained in a reaction vessel. The resulting reaction mixture was then stirred in a magnetic stirrer at room temperature for 8 h in open air. The progress of the reaction was monitored by TLC using petroleum ether and ethyl acetate as eluent solvent in silica bed on aluminium sheet. After completion of the reaction as indicated by TLC, a momentous amount of water and ethyl acetate were added to the reaction mixture and extracted thrice by ethyl acetate with the help of separating funnel. The extracted ethyl acetate solution was then passed through the bed of anhydrous sodium sulphate (Na_2SO_4) to make it a completely water free organic solution and evaporated to a significant amount of volume reduction and the crude product or sometimes the crystal product obtained was then purified by filtering and washing it with ethyl acetate and pet ether mixture. The products obtained were known compounds and identified through ^{1}H-NMR, ^{13}C-NMR and ^{19}F-NMR spectroscopies.

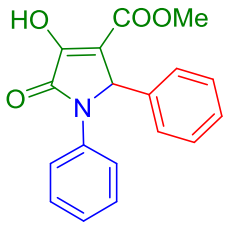
III. Characterization data of the synthesized derivatives of Methyl/Ethyl 2-aryl-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4a-4i) & (6a-6e) and Methyl/Ethyl 1-aryl-4-hydroxy-5-oxo-2-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (5a-5d) & (7a-7b)

Methyl 4-hydroxy-5-oxo-1,2-diphenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4a):

pale white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.562 (s, 3H), 6.048 (s, 1H), 7.194-7.261 (m, 8H), 7.541 (d, 2H, J = 7.6 Hz), 11.746 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.570, 61.003, 112.399, 122.976, 125.816, 128.107, 128.381, 128.730, 129.127, 136.695, 136.943, 152.996, 162.920, 164.403.



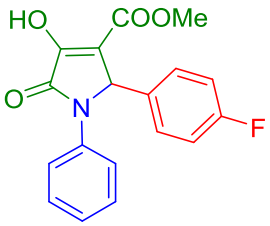
Methyl 2-(4-fluorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4b):

white crystal;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.571 (s, 3H), 6.086 (s, 3H), 6.965-7.095 (m, 3H), 7.202-7.341 (m, 4H), 7.480-7.599 (m, 2H), 11.798 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.589, 60.231, 112.096, 115.473, 115.688, 123.051, 125.892, 129.161, 130.187, 130.268, 133.165, 136.553, 153.160, 162.920, 163.212, 164.320;

^{19}F-NMR (376MHz, DMSO-d_6) δ(ppm): -114.264.



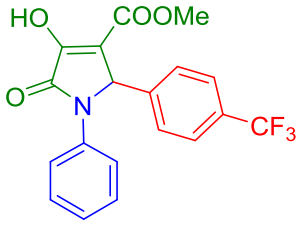
Methyl 4-hydroxy-5-oxo-1-phenyl-2-(4-(trifluoromethyl) phenyl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4c):

white crystal;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.566 (s, 3H), 6.191 (s, 1H), 7.065-7.102 (m, 1H), 7.265-7.304 (m, 2H), 7.494-7.620 (m, 6H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.479, 60.315, 110.747, 122.848, 123.134, 125.597, 125.936, 128.286, 128.596, 129.075, 129.231, 136.605, 142.556, 154.639, 163.232, 164.878;

^{19}F-NMR (376MHz, DMSO-d_6) δ(ppm): -60.998.

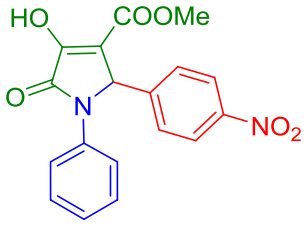


Methyl 4-hydroxy-2-(4-nitrophenyl)-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4d):

pale yellow solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.483 (s, 3H), 6.098 (s,1H), 7.026-7.063 (m, 1H), 7.236-7.275 (m, 2H), 7.516-7.599 (m, 4H), 8.040 (d, 2H, J = 8.4 Hz);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 50.621, 59.853, 122.489, 123.629, 125.467, 129.145, 129.395, 137.246, 147.135, 147.950, 164.154, 167.084.

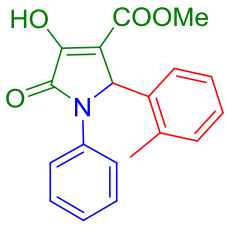


Methyl 4-hydroxy-5-oxo-1-phenyl-2-(o-tolyl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4e):

pale yellow solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 2.555 (s, 3H), 3.579 (s, 3H), 6.260 (s, 1H), 7.000-7.109 (m, 5H), 7.266-7.305 (m, 2H), 7.416-7.561 (m, 2H), 11.684 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 19.520, 51.592, 57.046, 113.167, 123.287, 125.276, 126.133, 126.617, 128.007, 129.228, 130.859, 134.953, 136.881, 137.810, 153.269, 163.106, 164.732.

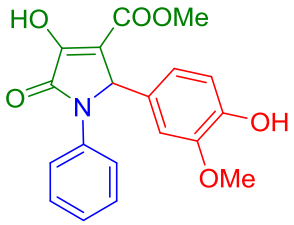


Methyl 4-hydroxy-2-(4-hydroxy-3-methoxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4f):

pale yellow solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.335 (s, 3H), 3.657 (s, 3H), 5.946 (s, 1H), 6.574-6.642 (m, 2H), 6.768 (d, 1H, J = 8.0 Hz), 7.091 (t, 1H, J = 7.2 Hz), 7.269-7.308 (m, 2H), 7.552 (d, 2H, J = 8 Hz), 8.911 (s, 1H), 11.558 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.535, 56.100, 60.642, 112.224, 112.506, 115.700, 120.689, 121.478, 123.234, 125.759, 127.270, 128.495, 129.043, 136.840, 146.638, 147.686, 152.715, 163.023, 164.299.

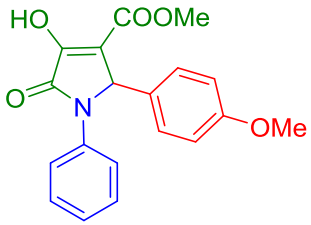


Methyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4g):

pale white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.566 (s, 3H), 3.623 (s, 3H), 5.994 (s, 1H), 6.742 (d, 2H, J = 8.4 Hz), 7.049-7.152 (m, 3H), 7.246-7.285 (m, 2H), 7.534 (d, 2H, J = 8 Hz);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.560, 55.347, 60.498, 112.462, 114.110, 123.019, 125.754, 128.483, 129.109, 129.279, 136.710, 152.795, 159.182, 162.966, 164.338.

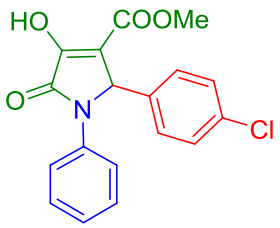


Methyl 2-(4-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4h):

white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.511 (s, 3H), 6.022 (s, 1H), 7.060 (t, 1H, J = 7.2 Hz), 7.172-7.328 (m, 6H), 7.540-7.589 (m, 2H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.204, 60.173, 109.223, 122.851, 125.738, 128.612, 129.137, 129.983, 132.565, 136.825, 137.310, 156.909, 163.769, 165.740.

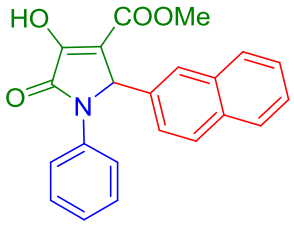


Methyl 4-hydroxy-2-(naphthalen-2-yl)-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4i):

white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.539 (s, 3H), 6.228 (s, 1H), 6.997-7.034 (m, 1H), 7.216-7.274 (m, 3H), 7.426-7.448 (m, 2H), 7.597 (d, 2H, J = 7.6 Hz), 7.720-7.785 (m, 2H), 7.831-7.854 (m, 1H), 11.848 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.589, 61.185, 112.229, 123.072, 124.878, 125.871, 126.691, 127.940, 128.197, 128.473, 129.127, 133.052, 134.415, 136.703, 153.224, 162.991, 164.485.

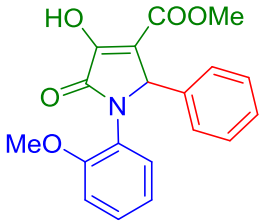


Methyl 4-hydroxy-1-(2-methoxyphenyl)-5-oxo-2-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (5a):

pale white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.565 (s, 3H), 3.755 (s, 3H), 5.742 (s, 1H), 6.761-6.834 (m, 1H), 7.006-7.213 (m, 8H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 51.536, 55.649, 56.170, 62.562, 111.023, 112.105, 112.844, 114.304, 116.674, 120.699, 121.305, 124.496, 128.125, 128.468, 128.606, 129.407, 136.684, 153.839, 155.162, 163.047, 164.419.

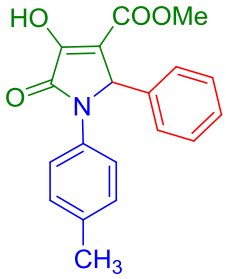


Methyl 4-hydroxy-5-oxo-2-phenyl-1-(p-tolyl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (5b):

pale yellow solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 2.232 (s, 3H), 3.597 (s, 3H), 6.014 (s, 1H), 7.070 (d, 2H, J = 8.4 Hz), 7.127-7.239 (m, 5H), 7.427 (d, 2H, J = 8.4 Hz), 11.723 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 20.874, 51.530, 61.122, 112.234, 122.998, 128.102, 128.343, 128.712, 129.588, 134.189, 135.125, 137.033, 153.138, 162.939, 164.261.

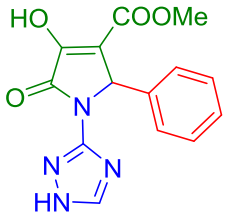


Methyl 4-hydroxy-5-oxo-2-phenyl-1-(1*H*-1,2,4-triazol-3-yl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (5c):

white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.526 (s, 3H), 6.350 (s, 1H), 7.209-7.369 (m, 5H), 7.718 (s, 1H), 11.689 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 52.253, 53.656, 59.317, 98.725, 127.375, 128.854, 129.135, 139.934, 141.102, 146.795, 151.028, 163.670, 164.415.

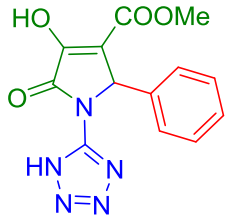


Methyl 4-hydroxy-5-oxo-2-phenyl-1-(1*H*-tetrazol-5-yl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (5d):

white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 3.535 (s, 3H), 6.761 (s, 1H), 7.300-7.415 (m, 5H), 12.232 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 52.487, 53.848, 58.784, 99.918, 127.622, 129.452, 139.429, 139.940, 148.604, 163.304, 163.931.

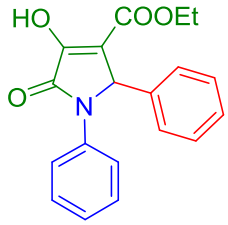


Ethyl 4-hydroxy-5-oxo-1,2-diphenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (6a):

white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.028 (t, 3H, J = 7 Hz), 3.893-3.951 (m, 2H), 5.876 (s, 1H), 7.025-7.258 (m, 8H), 7.555 (d, 2H, J = 8 Hz);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.601, 58.834, 60.255, 60.781, 122.594, 125.173. 127.690, 128.075, 128.310, 128.969, 137.538, 139.488, 163.745, 166.990.

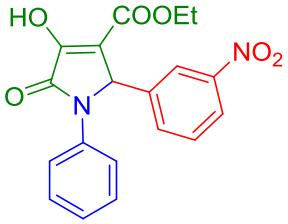


Ethyl 4-hydroxy-2-(3-nitrophenyl)-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (6b):

pale yellow solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.018 (t, 3H, *J* = 7 Hz), 3.881-3.973 (m, 2H), 6.123 (s, 1H), 7.006-7.043 (m, 1H), 7.467 (t, 1H, *J* = 8 Hz), 7.587-7.665 (m, 3H) 7.975 (d, 1H, *J* = 8 Hz), 8.143 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.625, 58.911, 59.771, 122.584, 122.866, 123.245, 125.434, 129.164, 130.013, 134.498, 137.205, 147.819, 163.652.

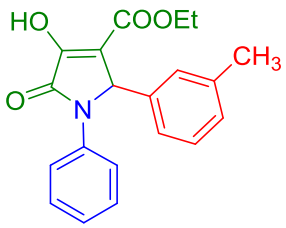


Ethyl 4-hydroxy-5-oxo-1-phenyl-2-(m-tolyl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (6c):

pale yellow solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.029 (t, 3H, *J* = 7 Hz), 2.159 (s, 3H), 3.873-3.936 (m, 2H), 5.807 (s, 1H), 6.880-7.039 (m, 5H), 7.209-7.248 (m, 2H), 7.548 (d, 2H, *J* = 8 Hz);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.661, 21.424, 58.727, 60.716, 106.156, 122.498, 125.091, 125.354, 128.149, 128.378, 128.964, 137.204, 137.668, 139.604, 160.324, 163.863, 167.207.

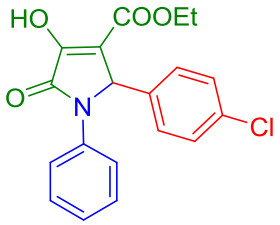


Ethyl 2-(4-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (6d):

white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.080 (t, 3H, J = 7 Hz), 3.860-4.002 (m, 2H), 5.926 (s, 1H), 7.013-7.050 (m, 1H), 7.172-7.282 (m, 6H), 7.547 (d, 2H, J = 8 Hz);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.671, 59.001, 60.024, 106.459, 122.644, 125.358, 128.367, 129.047, 130.026, 132.148, 137.289, 138.577, 159.877, 163.741, 166.825.

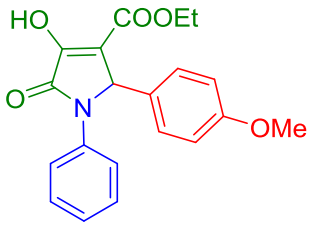


Ethyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (6e):

pale white solid;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.037 (t, 3H, J = 7 Hz), 3.622 (s, 3H), 3.887-3.937 (m, 2H), 5.813 (s, 1H), 6.709 (d, 2H, J = 8.4 Hz), 6.995-7.032 (m, 1H), 7.091-7.115 (m, 2H), 7.210-7.249 (m, 2H), 7.542 (d, 2H, J = 8.4 Hz);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.749, 55.310, 58.708, 60.224, 113.676, 122.605, 125.021, 128.913, 129.157, 131.310, 137.637, 158.690, 163.866, 167.060.

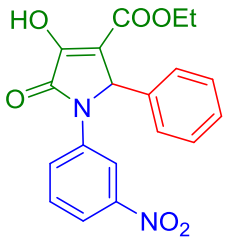


Ethyl 4-hydroxy-1-(3-nitrophenyl)-5-oxo-2-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (7a):

pale white crystal;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.068 (t, 3H, J = 7 Hz), 3.951-4.089 (m, 2H), 6.206 (s, 1H), 7.135-7.238 (m, 3H), 7.315 (d, 2H, J = 7.6 Hz), 7.533-7.574 (m, 1H), 7.895-7.963 (m, 2H), 8.606 (s, 1H), 11.877 (s, 1H);

^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.421, 60.284, 60.950, 113.338, 116.765, 120.142, 128.271, 128.633, 128.858, 130.581, 136.381, 137.839, 148.268, 152.542, 162.277, 164.976.

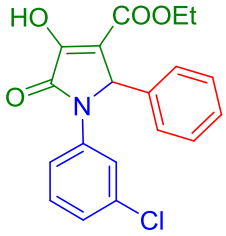


Ethyl 1-(3-chlorophenyl)-4-hydroxy-5-oxo-2-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate (7b):

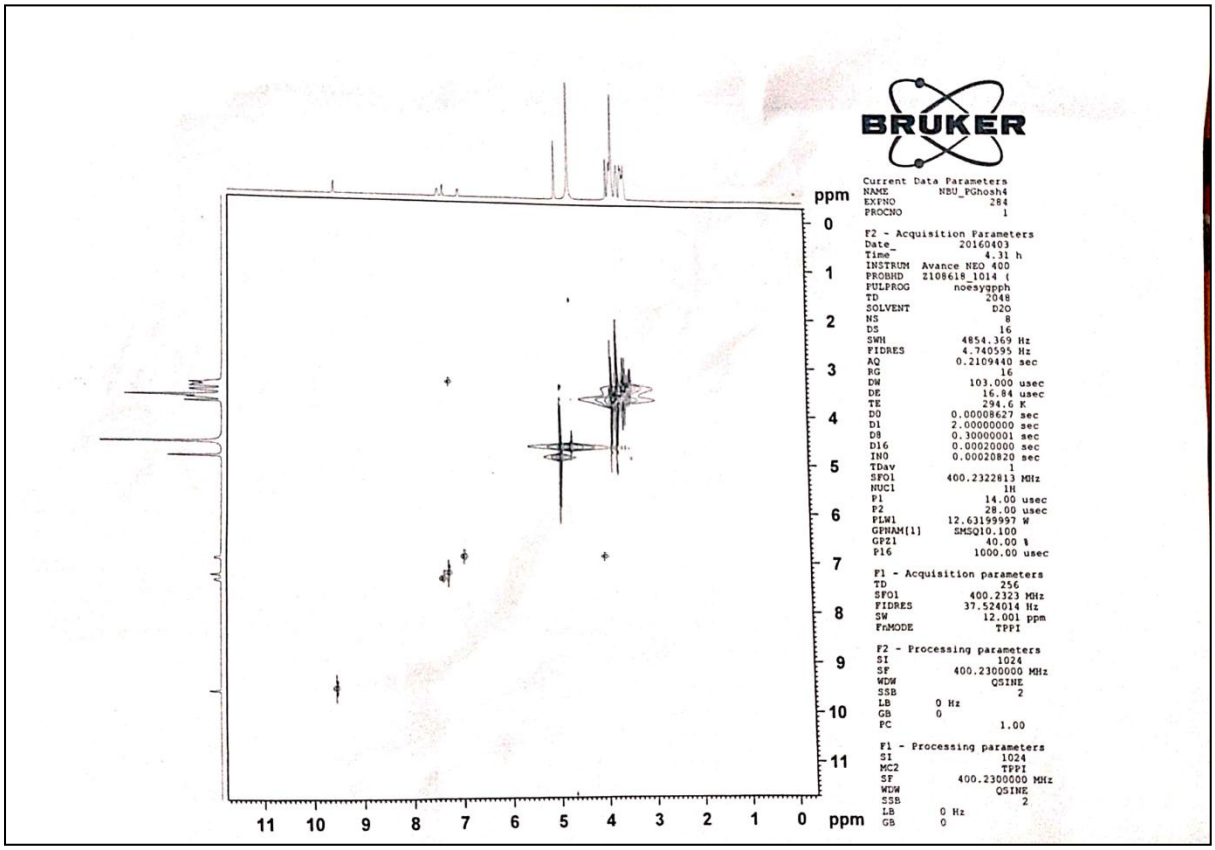
pale white crystal;

^{1}H-NMR (400 MHz, DMSO-d_6) δ(ppm): 1.058 (t, 3H, J = 7 Hz), 3.952-4.059 (m, 2H), 6.097 (s, 1H), 7.110-7.303 (m, 7H), 7.508 (d, 1H, J = 8 Hz) 7.747 (s, 1H) 11.783 (s, 1H);

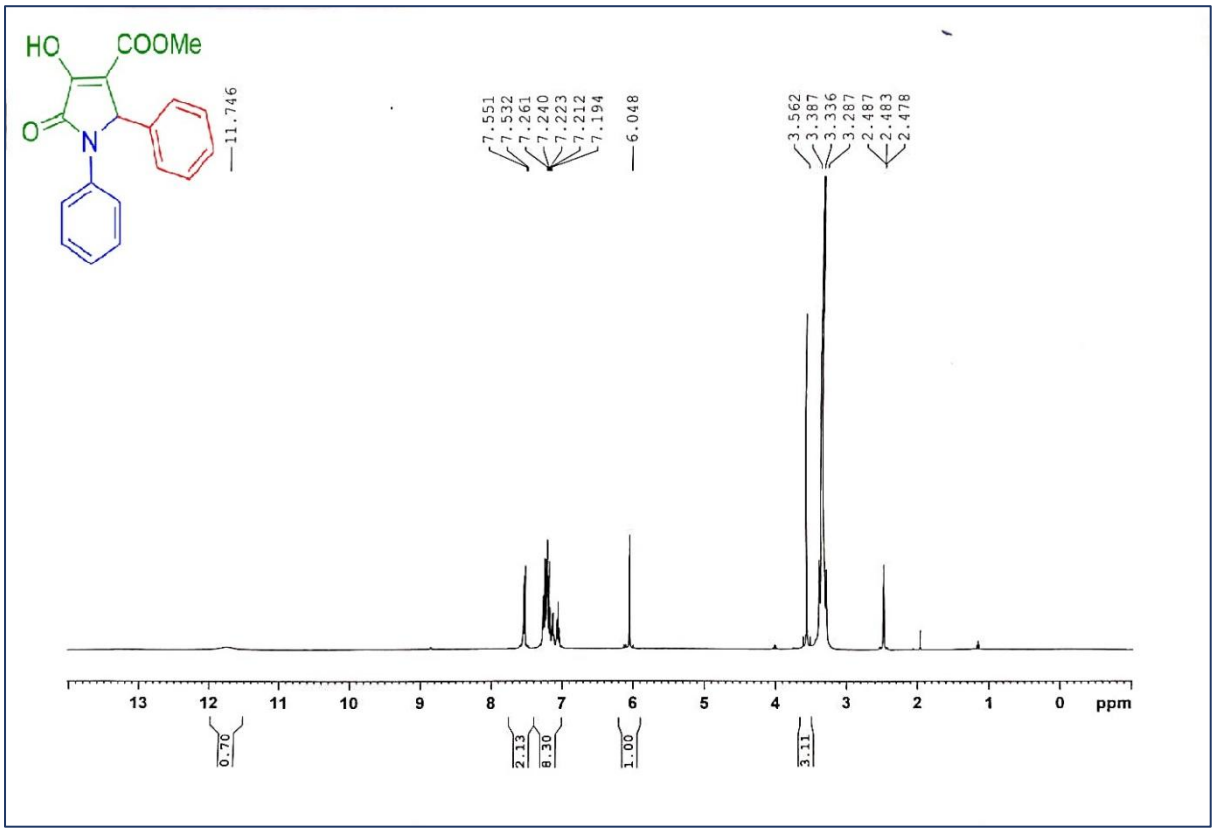
^{13}C-NMR (100 MHz, DMSO-d_6) δ(ppm): 14.428, 60.210, 60.920, 113.036, 121.151, 122.406, 125.548, 128.211, 128.502, 128.755, 130.744, 133.469, 136.673, 138.129, 152.724, 162.326, 164.659.

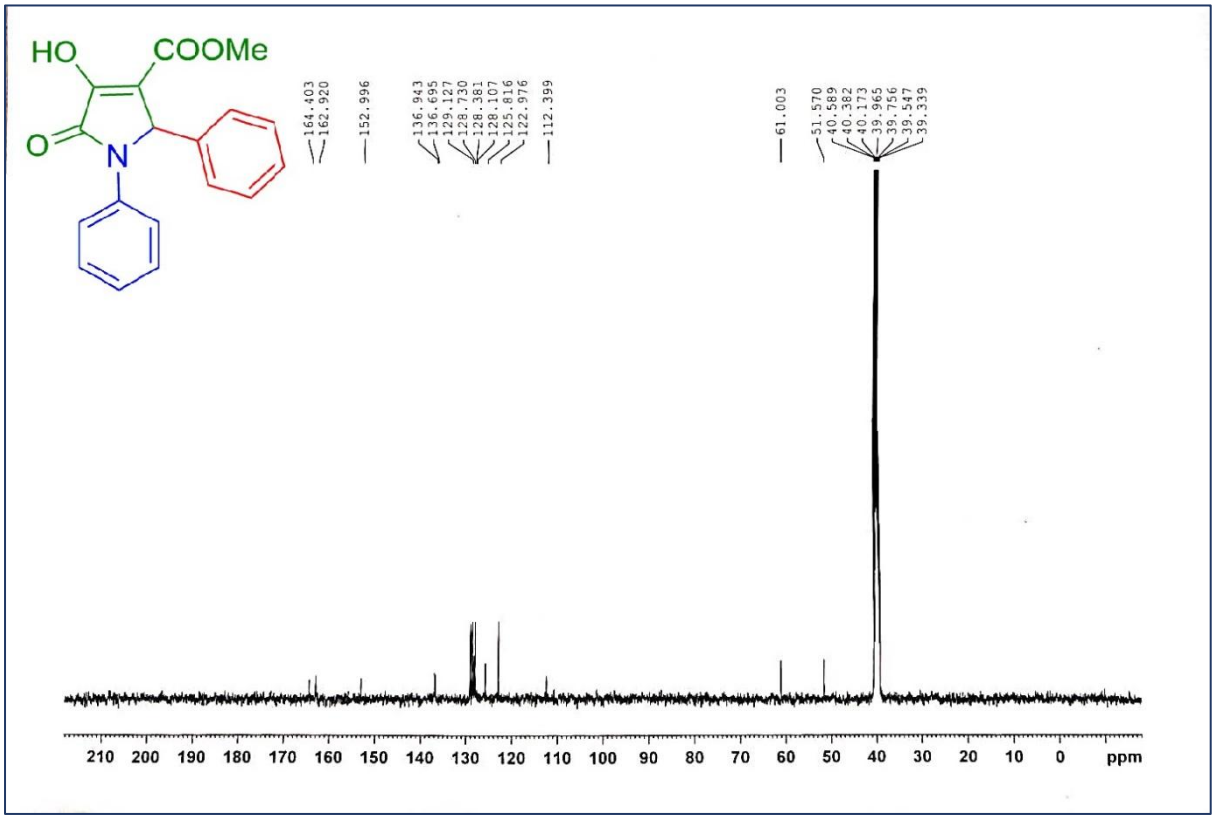


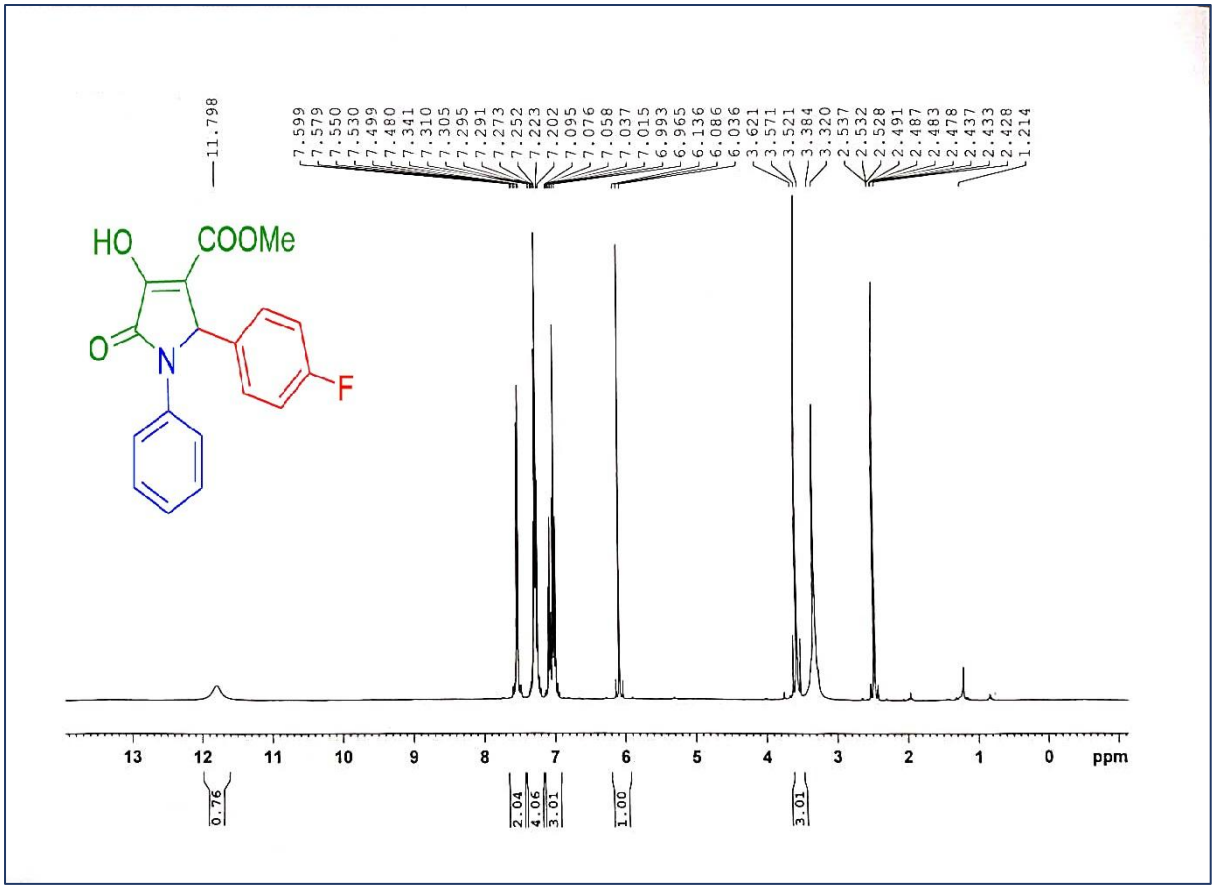
IV. Scanned copy of NOSEY NMR spectra of inclusion complex of aldehydes and β-CD

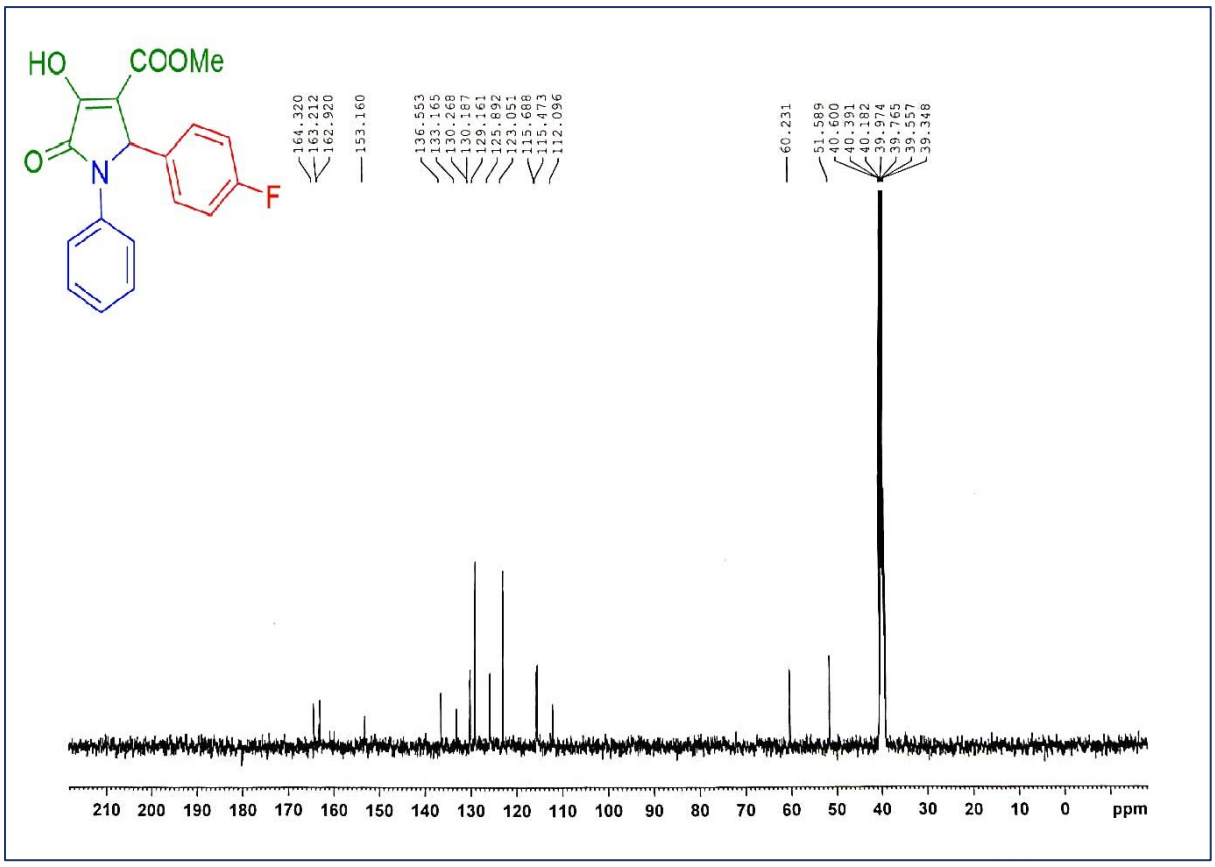


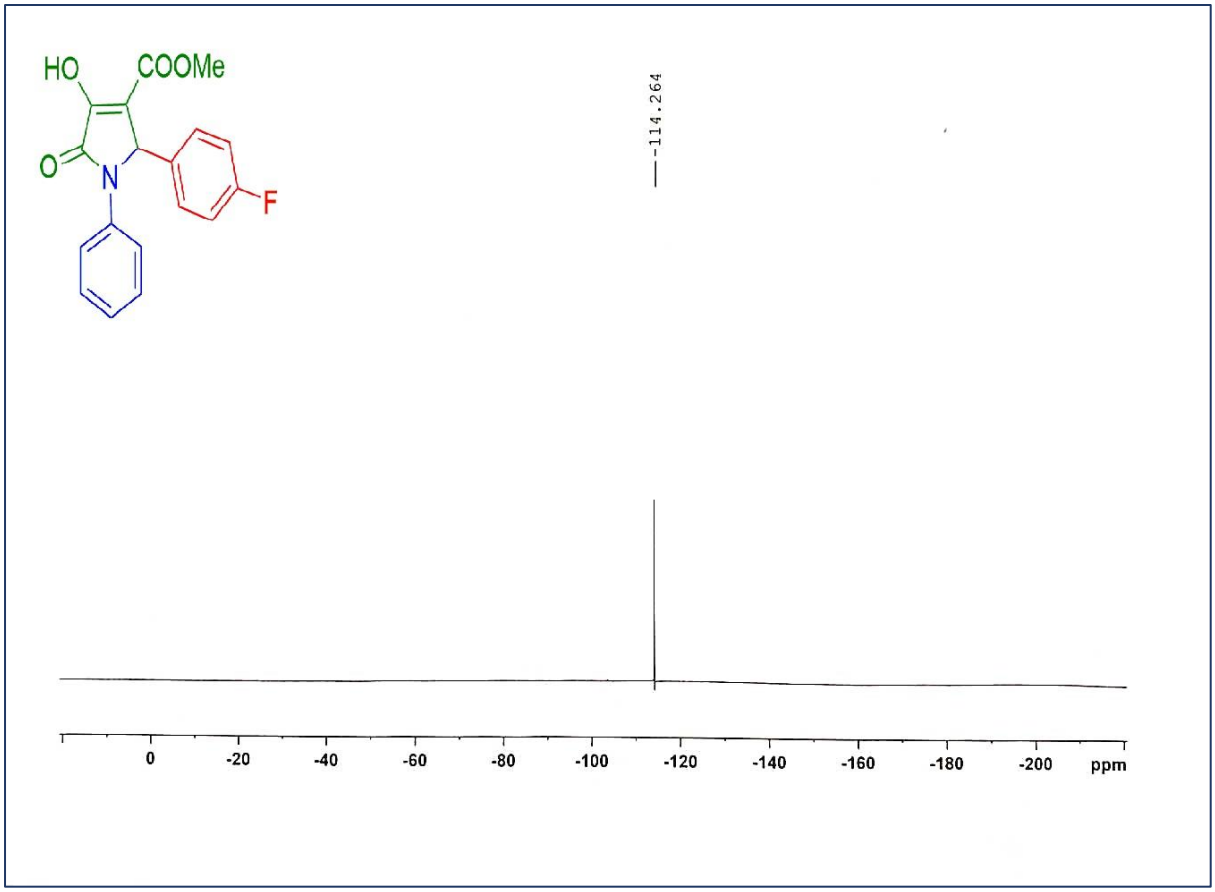
V. Scanned copies of the ^{1}H, ^{13}C and ^{19}F NMR of synthesised derivatives

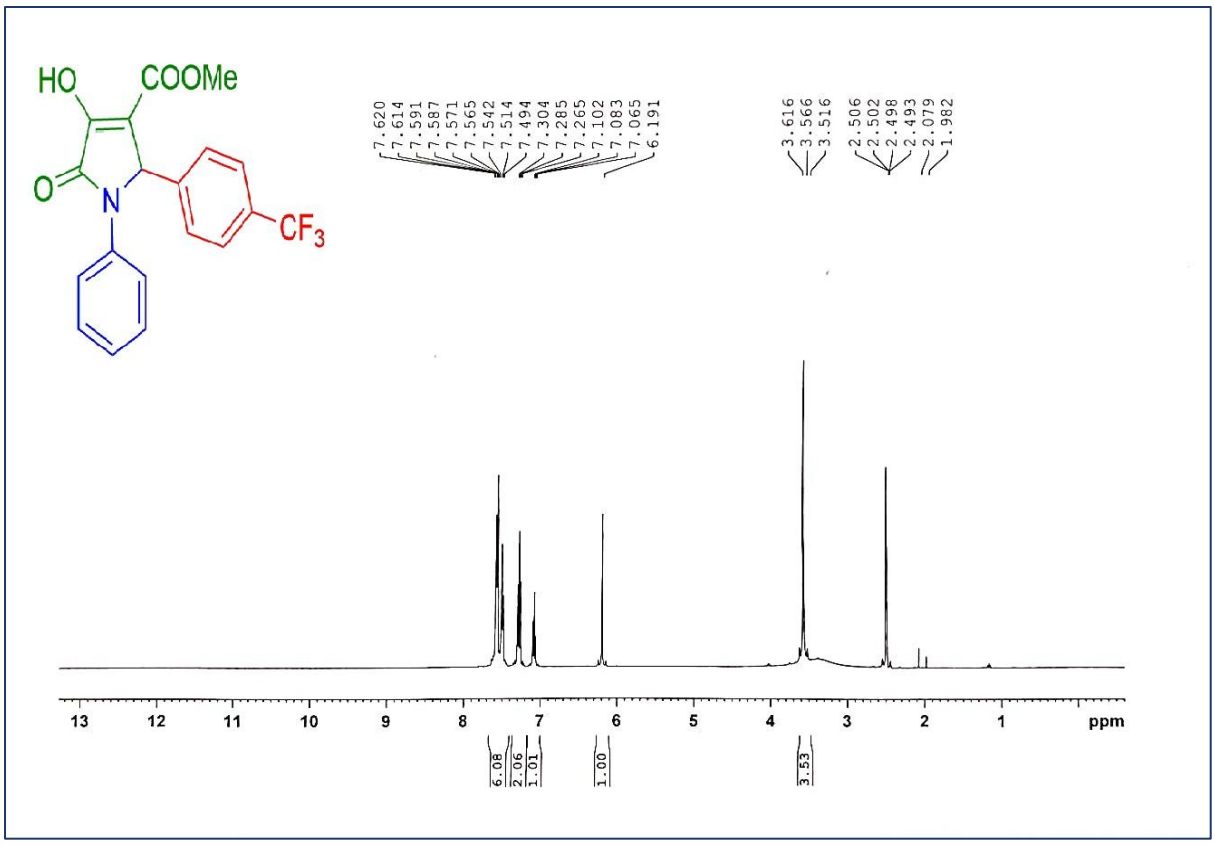


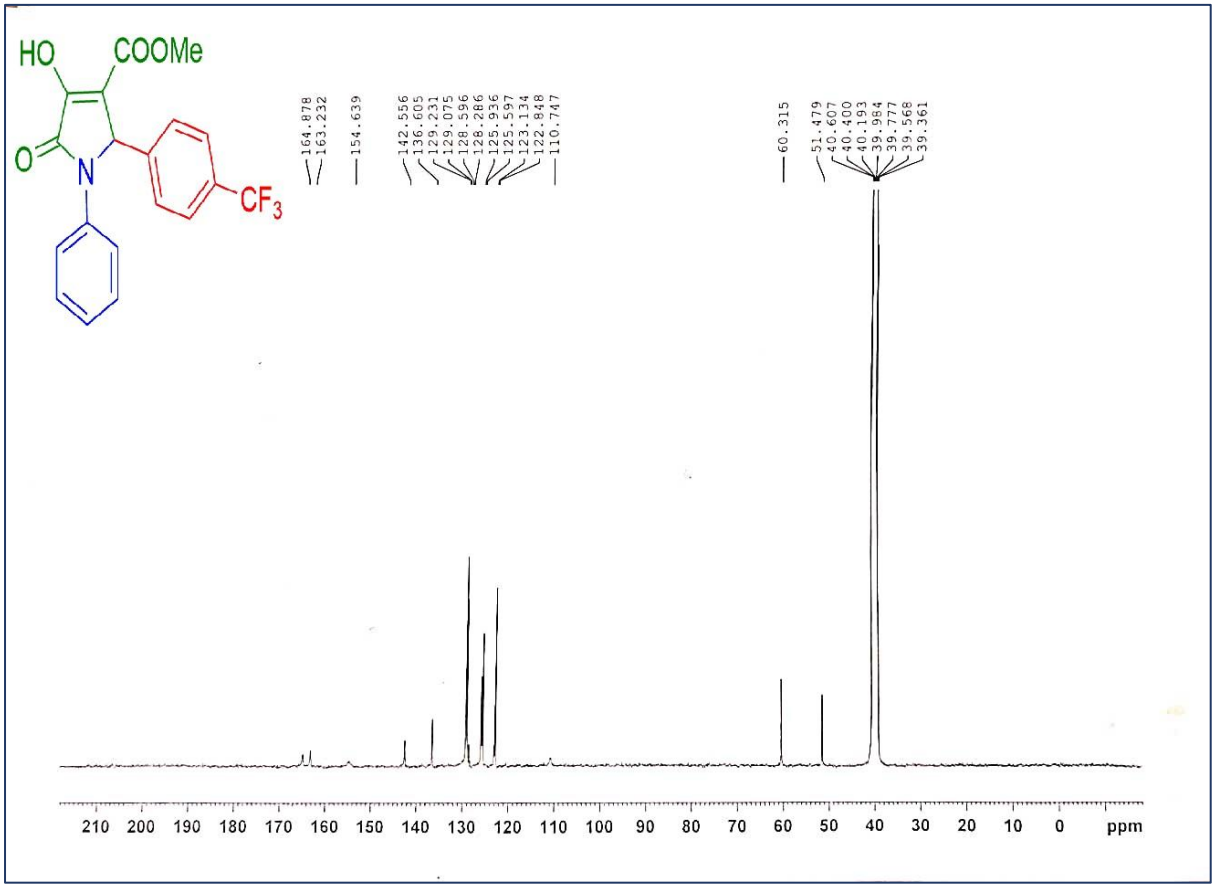


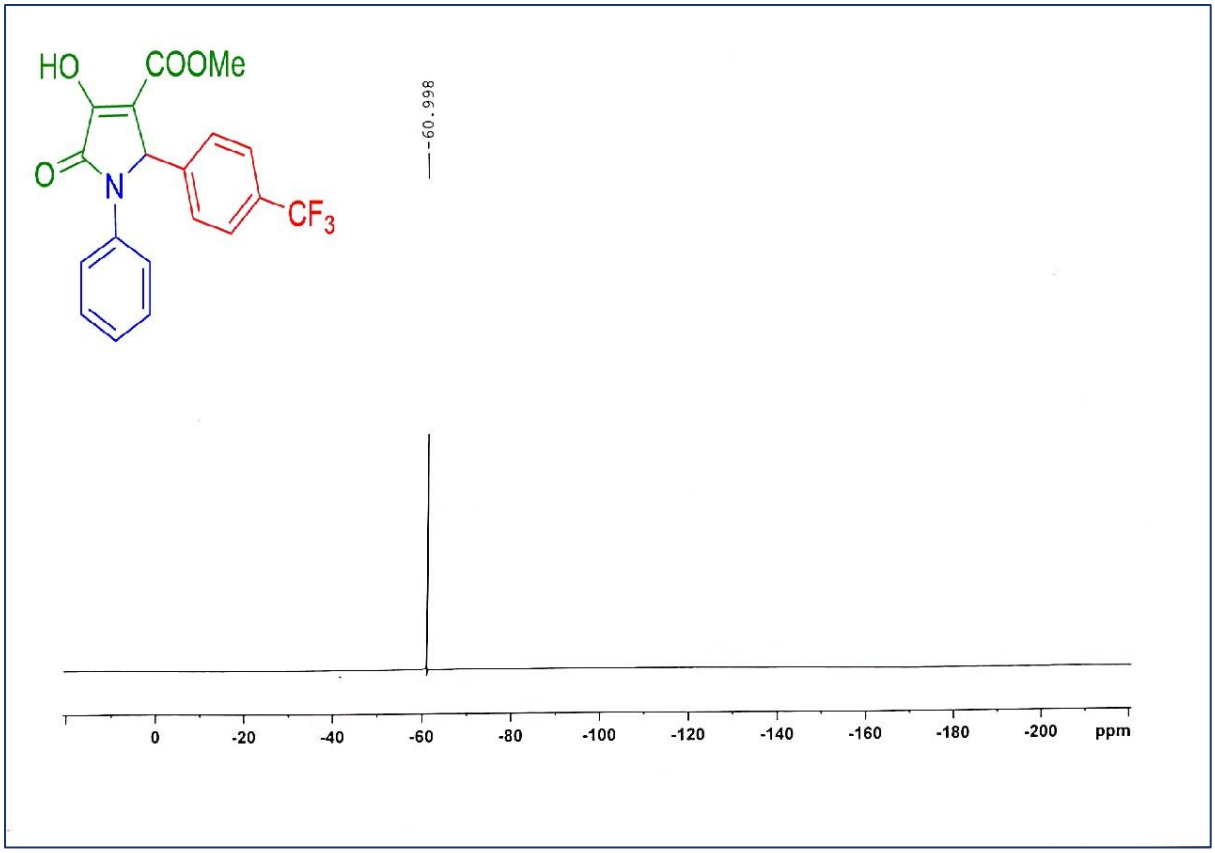

HO
COOMe
O
N
F
11.798
7.599
7.579
7.550
7.530
7.499
7.480
7.341
7.310
7.305
7.295
7.291
7.273
7.252
7.223
7.202
7.095
7.076
7.058
7.037
7.015
6.993
6.965
6.136
6.086
6.036
3.621
3.571
3.521
3.384
3.320
2.537
2.532
2.528
2.491
2.487
2.483
2.478
2.437
2.433
2.428
1.214
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.76
2.04
4.06
3.01
1.00
3.01

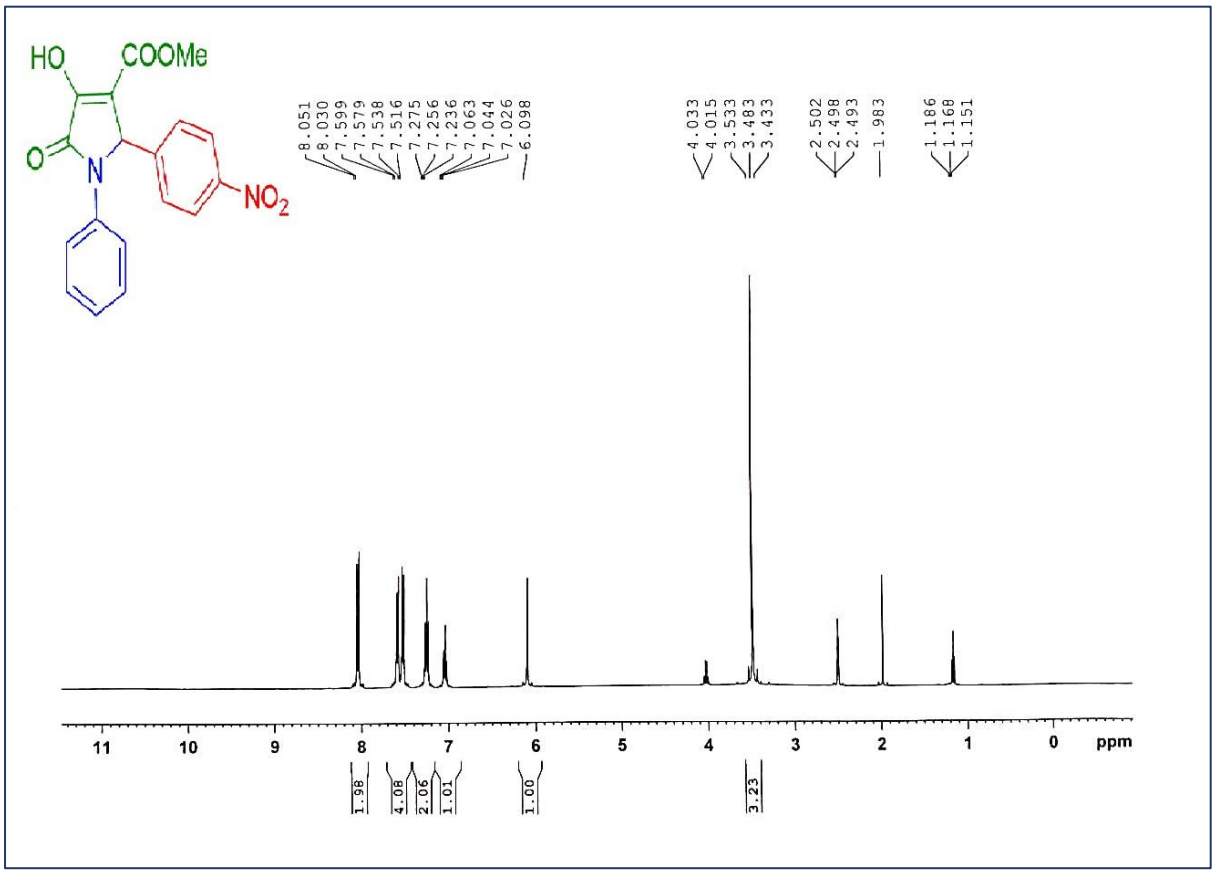

HO
COOMe
O
N
F
164.320
163.212
162.920
153.160
136.553
133.165
130.268
130.187
129.161
125.892
123.051
115.688
115.473
112.096
60.231
51.589
40.600
40.391
40.182
39.974
39.765
39.557
39.348
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

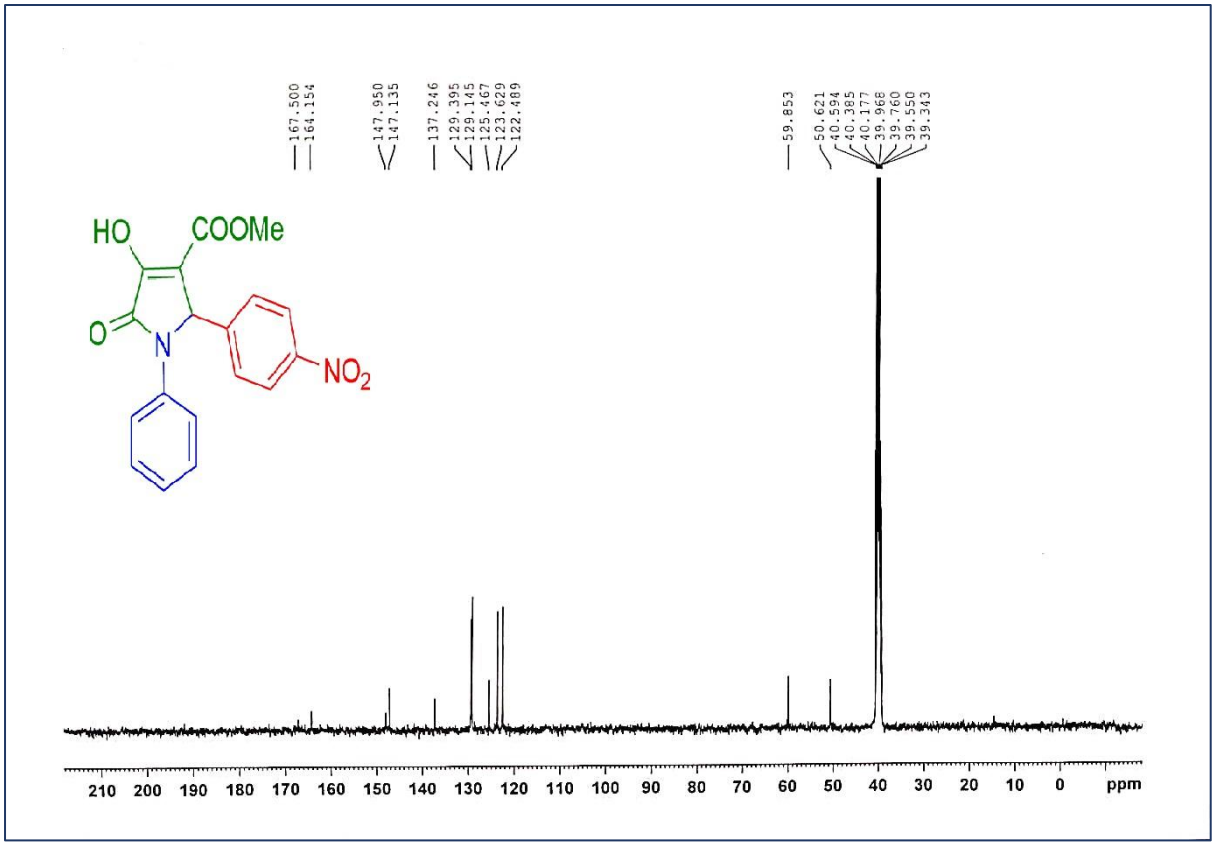

HO
COOMe
O
N
F
-114.264
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

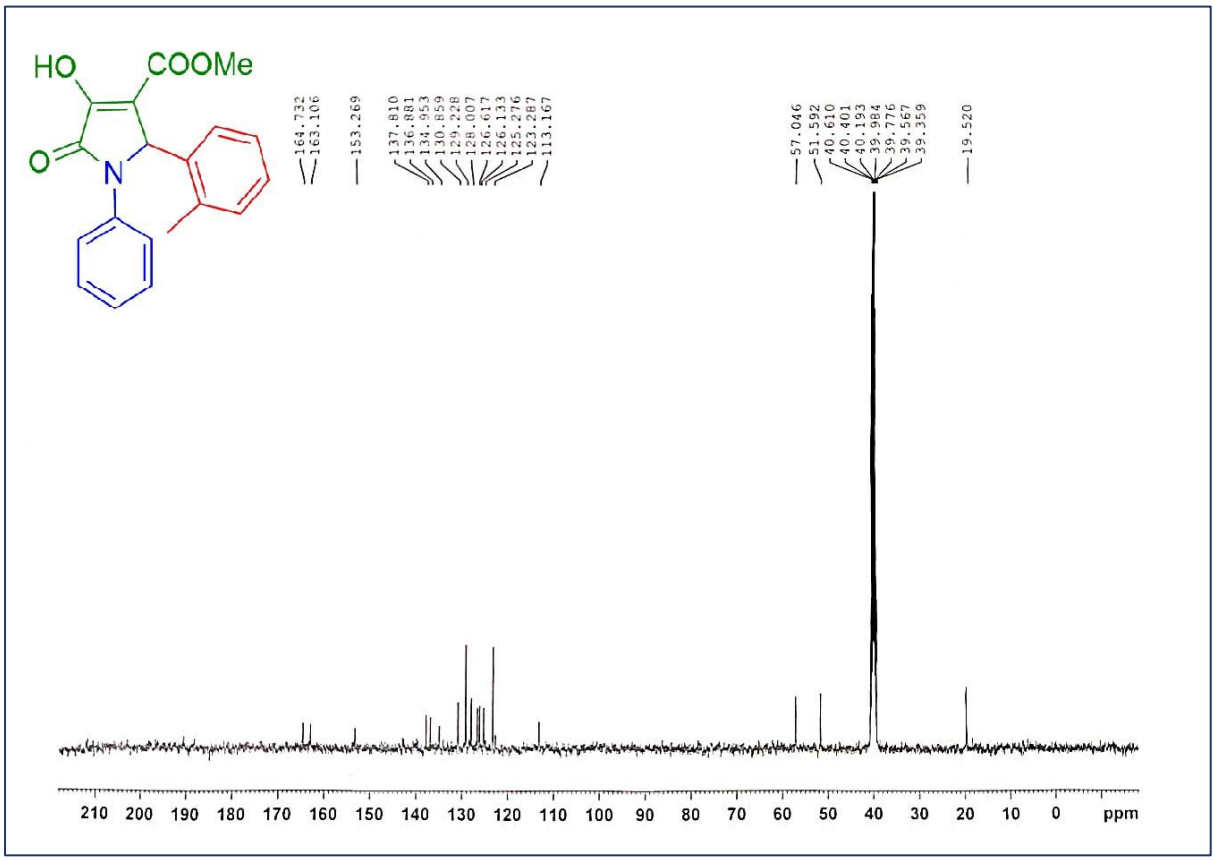

HO
COOMe
O
N
CF3
7.620
7.614
7.591
7.587
7.571
7.565
7.542
7.514
7.494
7.304
7.285
7.265
7.102
7.083
7.065
6.191
3.616
3.566
3.516
2.506
2.502
2.498
2.493
2.079
1.982
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
6.08
2.06
1.01
1.00
3.53

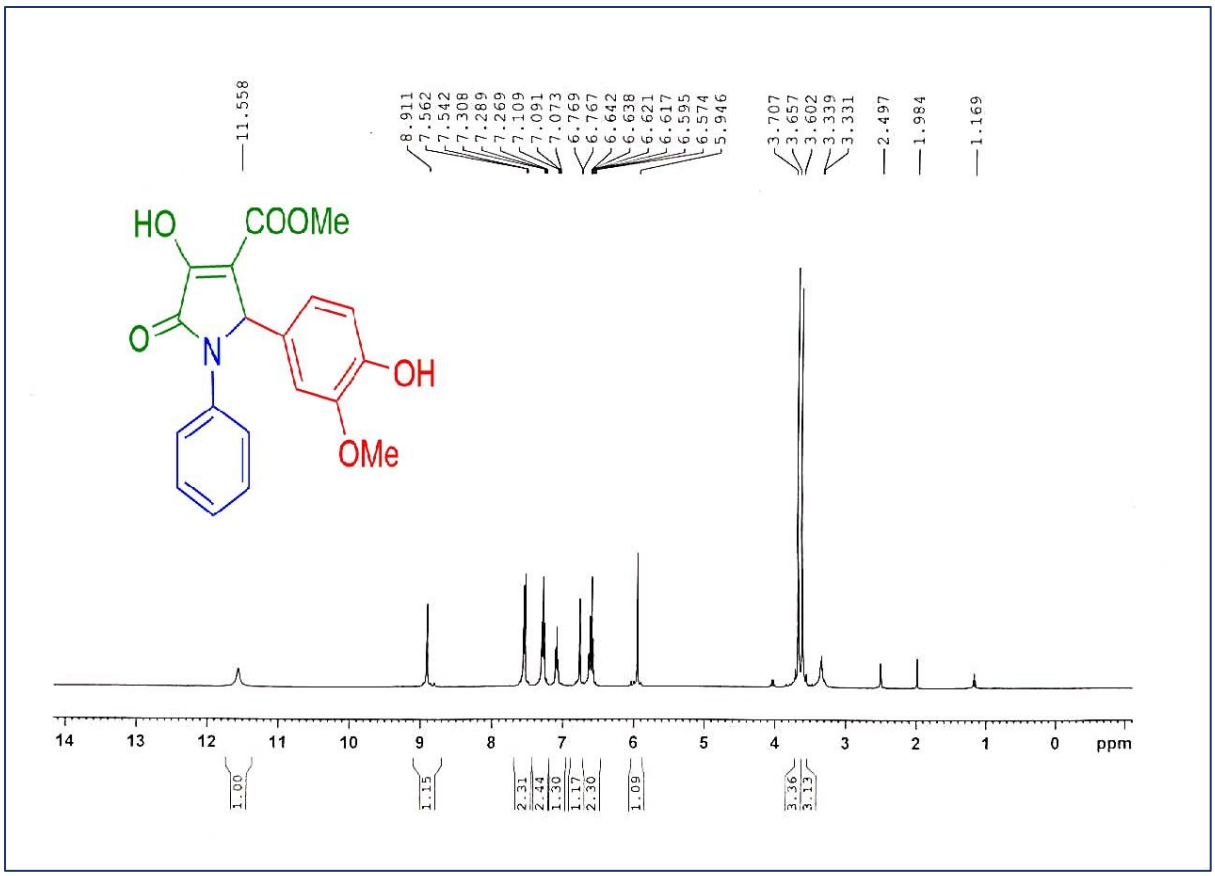

HO
COOMe
O
N
CF3
164.878
163.232
154.639
142.556
136.605
129.231
129.075
128.596
128.286
125.936
125.597
123.134
122.848
110.747
60.315
51.479
40.607
40.400
40.193
39.984
39.777
39.568
39.361
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

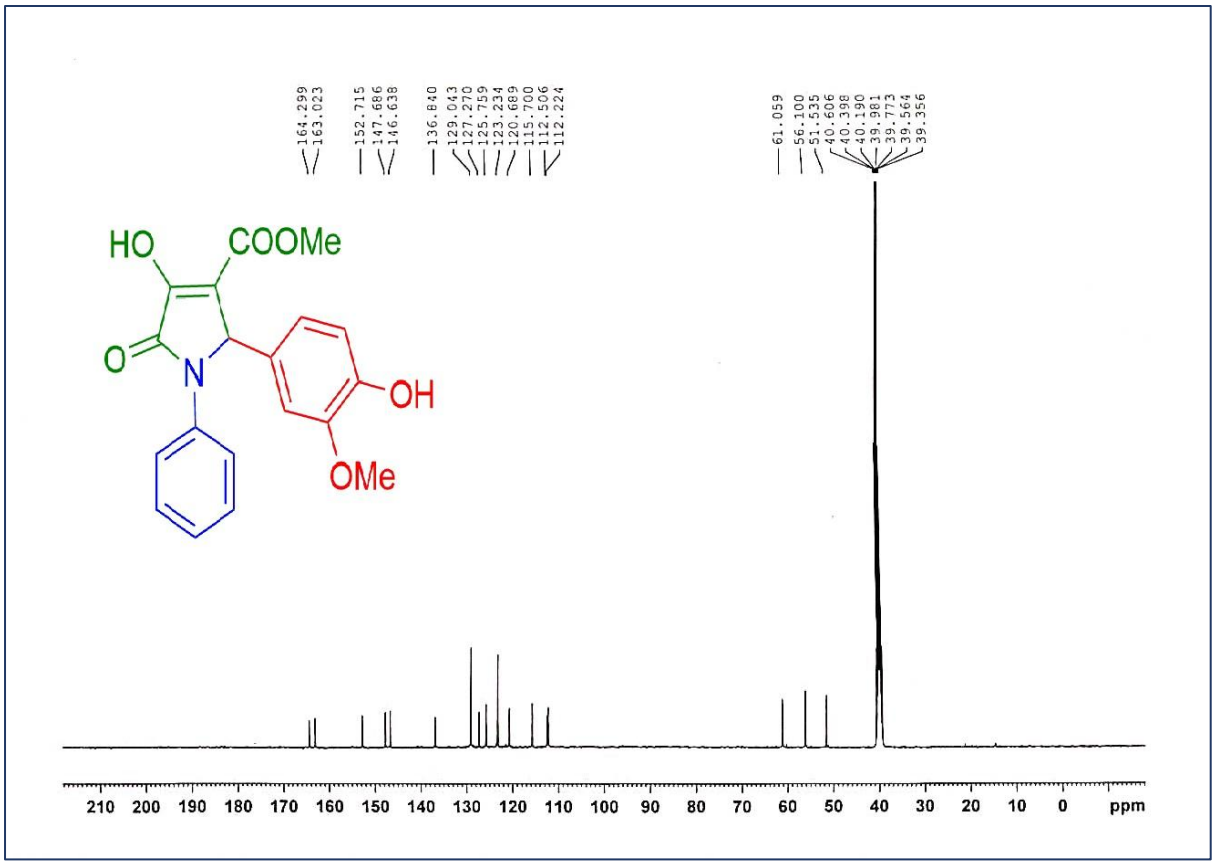

HO
COOMe
O
N
CF3
-60.998
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

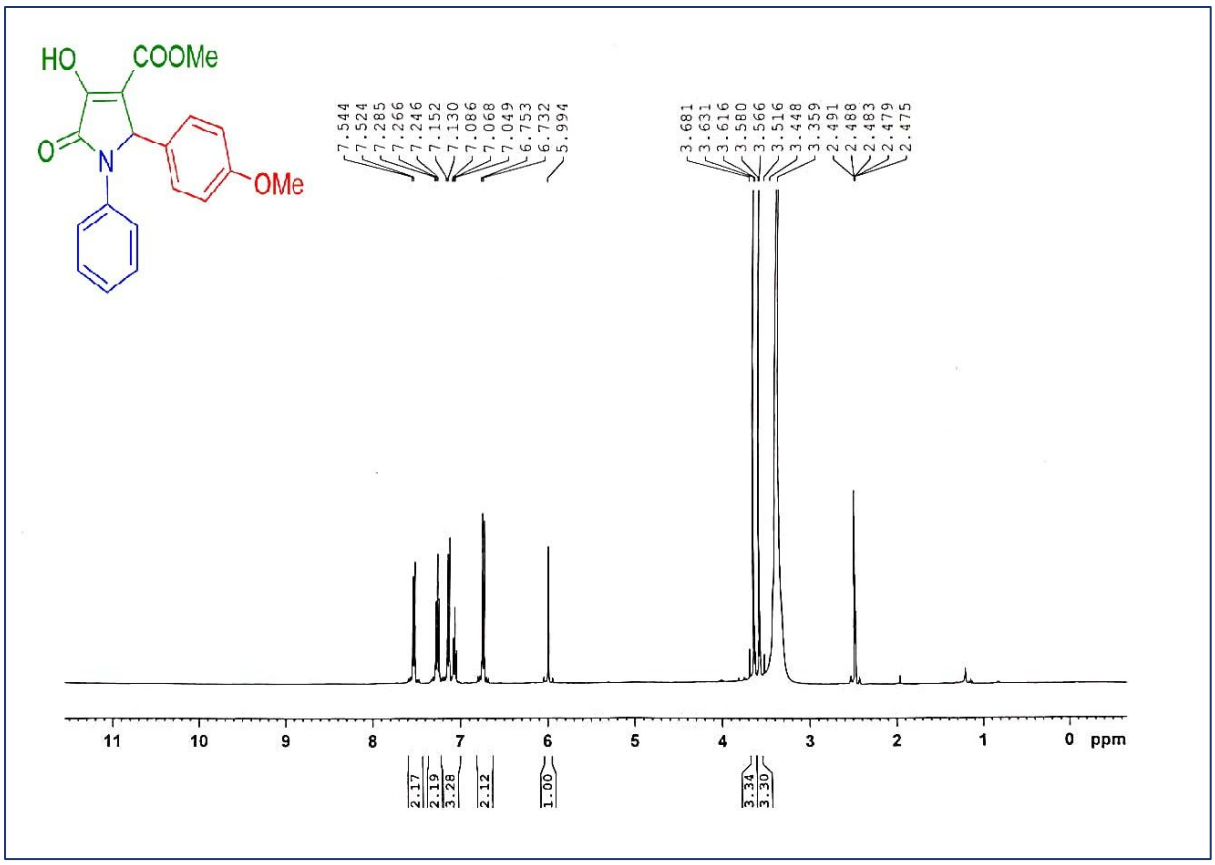

HO
COOMe
O
N
NO2
8.051
8.030
7.599
7.579
7.538
7.516
7.275
7.256
7.236
7.063
7.044
7.026
6.098
4.033
4.015
3.533
3.483
3.433
2.502
2.498
2.493
1.983
1.186
1.168
1.151
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
4.08
2.06
1.01
1.00
3.23

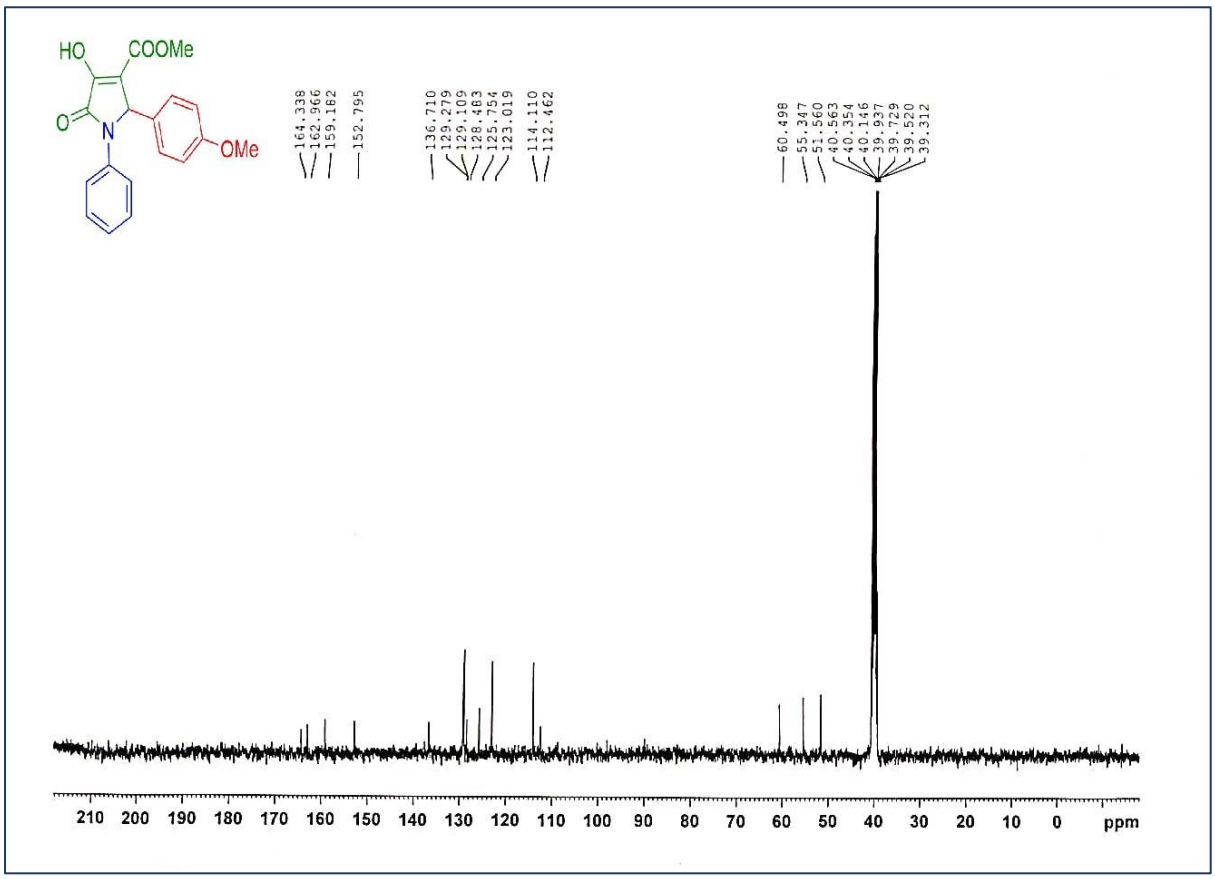

167.500
164.154
147.950
147.135
137.246
129.395
129.145
125.467
123.629
122.489
59.853
50.621
40.594
40.385
40.177
39.968
39.760
39.550
39.343
HO
COOMe
O
N
NO2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

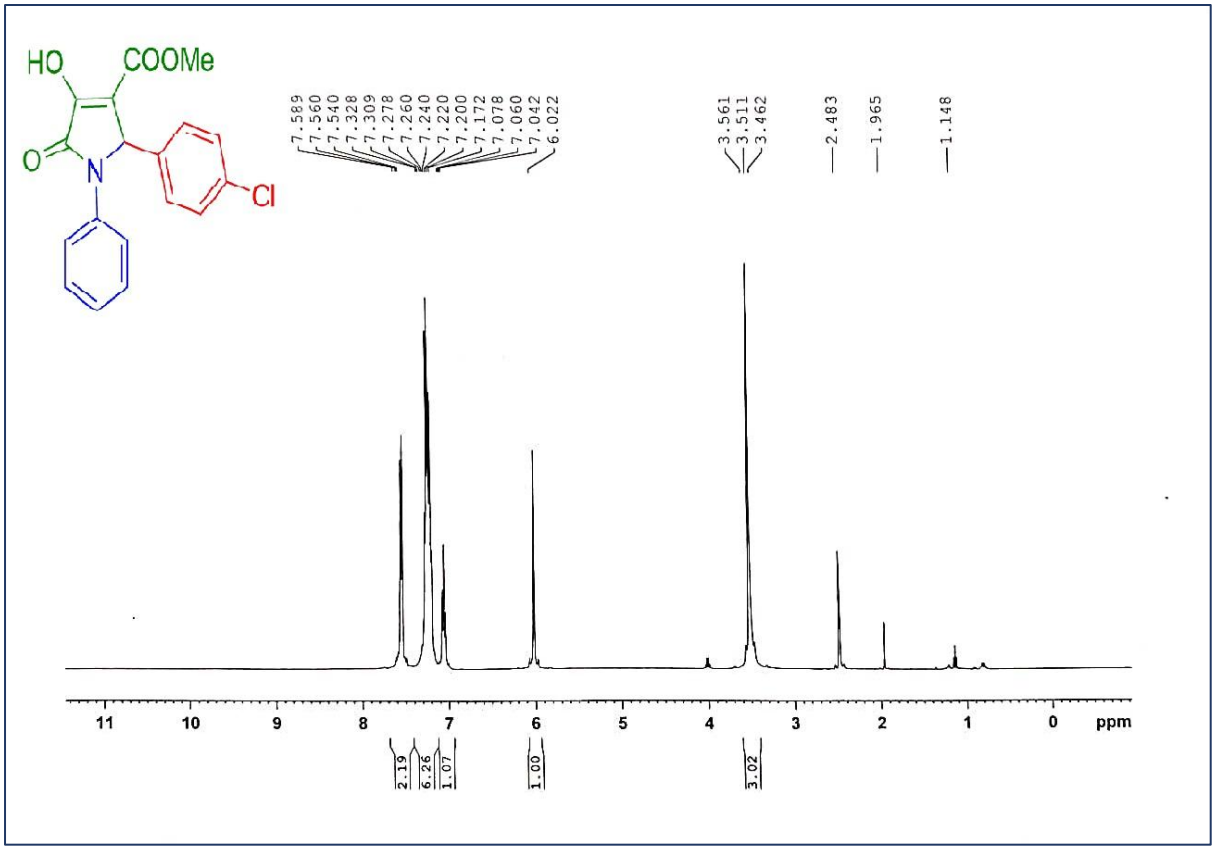

HO
COOMe
O
N
164.732
163.106
153.269
137.810
136.881
134.953
130.859
129.228
128.007
126.617
126.133
125.276
123.287
113.167
57.046
51.592
40.610
40.401
40.193
39.984
39.776
39.567
39.359
19.520
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

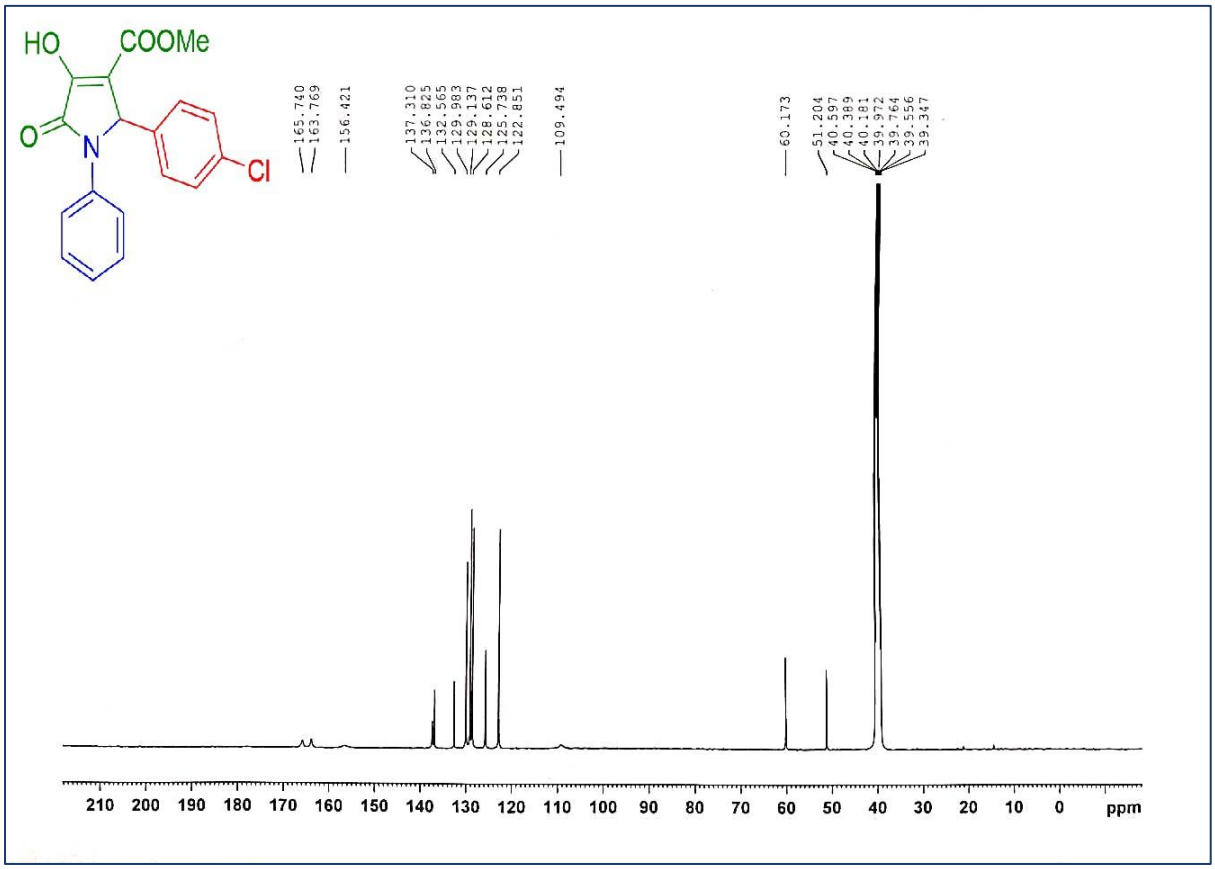

11.558
8.911
7.562
7.542
7.308
7.289
7.269
7.109
7.091
7.073
6.769
6.767
6.642
6.638
6.621
6.617
6.595
6.574
5.946
3.707
3.657
3.602
3.339
3.331
2.497
1.984
1.169
HO
COOMe
O
N
OH
OMe
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.15
2.31
2.44
1.30
1.17
2.30
1.09
3.36
3.13

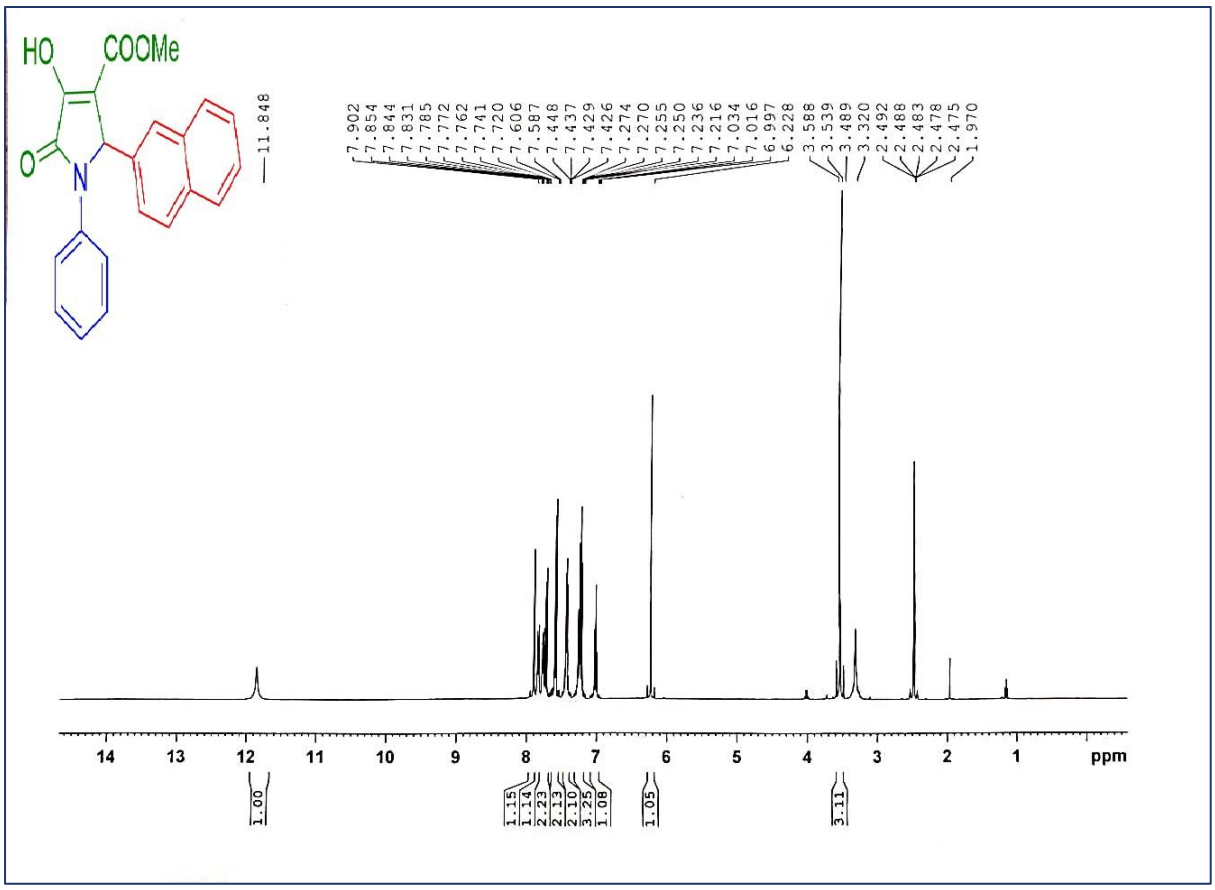

164.299
163.023
152.715
147.686
146.638
136.840
129.043
127.270
125.759
123.234
120.689
115.700
112.506
112.224
61.059
56.100
51.535
40.606
40.398
40.190
39.981
39.773
39.564
39.356
HO
COOMe
O
N
OH
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

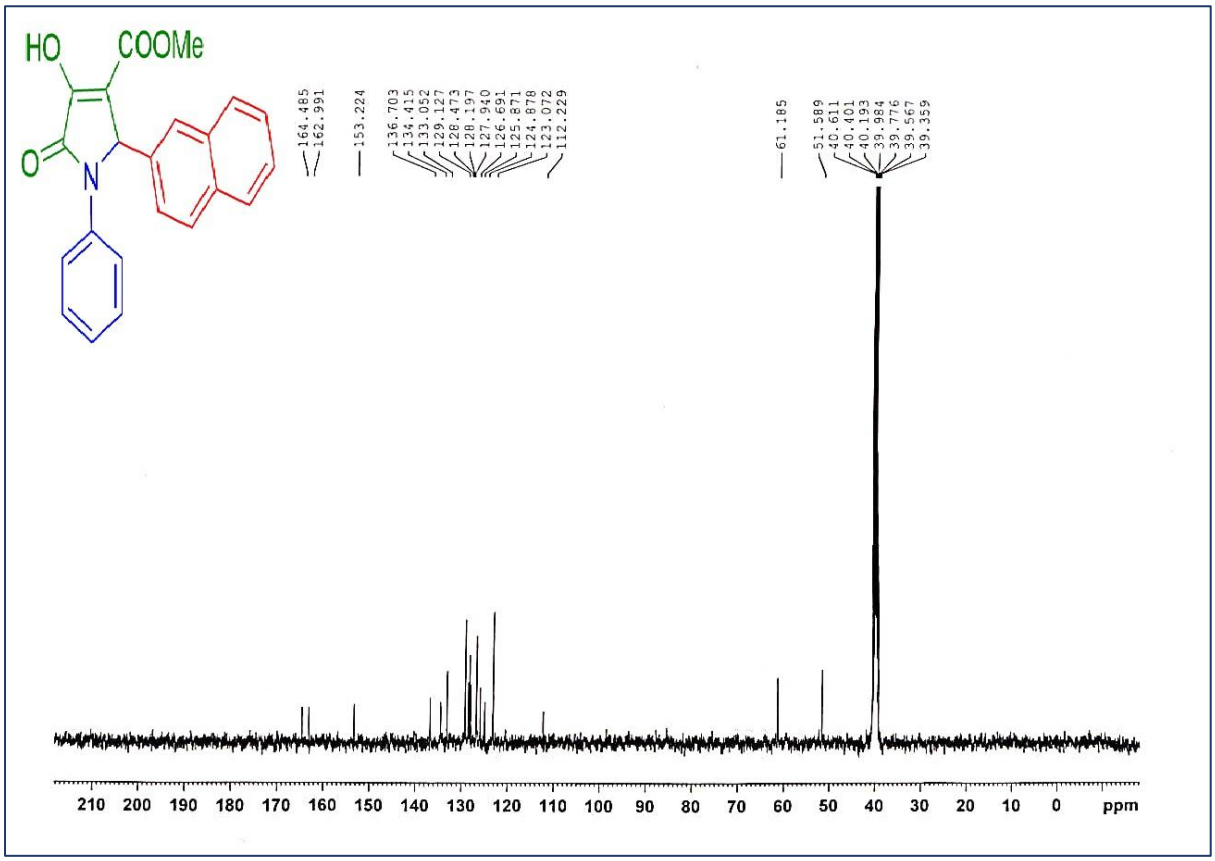

HO
COOMe
O
N
OMe
7.544
7.524
7.285
7.266
7.246
7.152
7.130
7.086
7.068
7.049
6.753
6.732
5.994
3.681
3.631
3.616
3.580
3.566
3.516
3.448
3.359
2.491
2.488
2.483
2.479
2.475
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.17
2.19
3.28
2.12
1.00
3.34
3.30

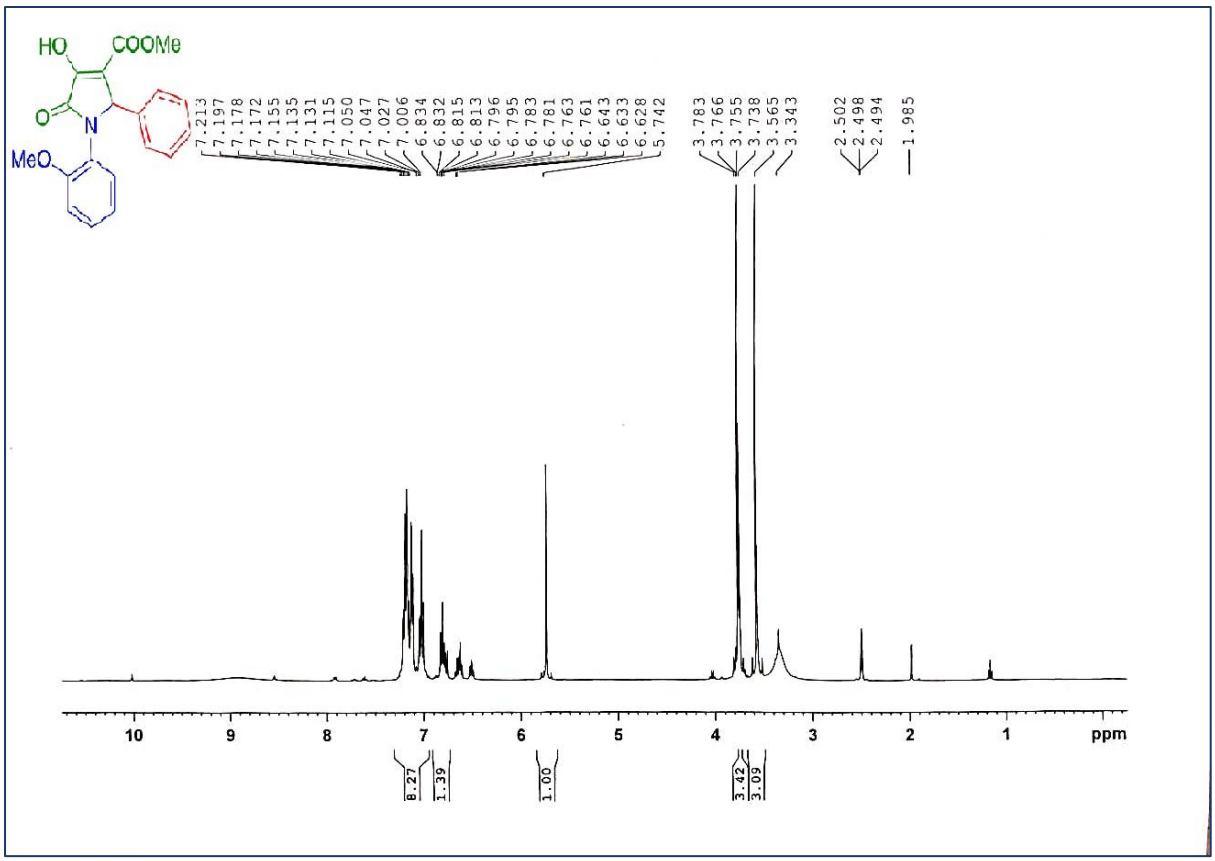

HO
COOMe
O
N
OMe
164.338
162.966
159.182
152.795
136.710
129.279
129.109
128.483
125.754
123.019
114.110
112.462
60.498
55.347
51.560
40.563
40.354
40.146
39.937
39.729
39.520
39.312
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

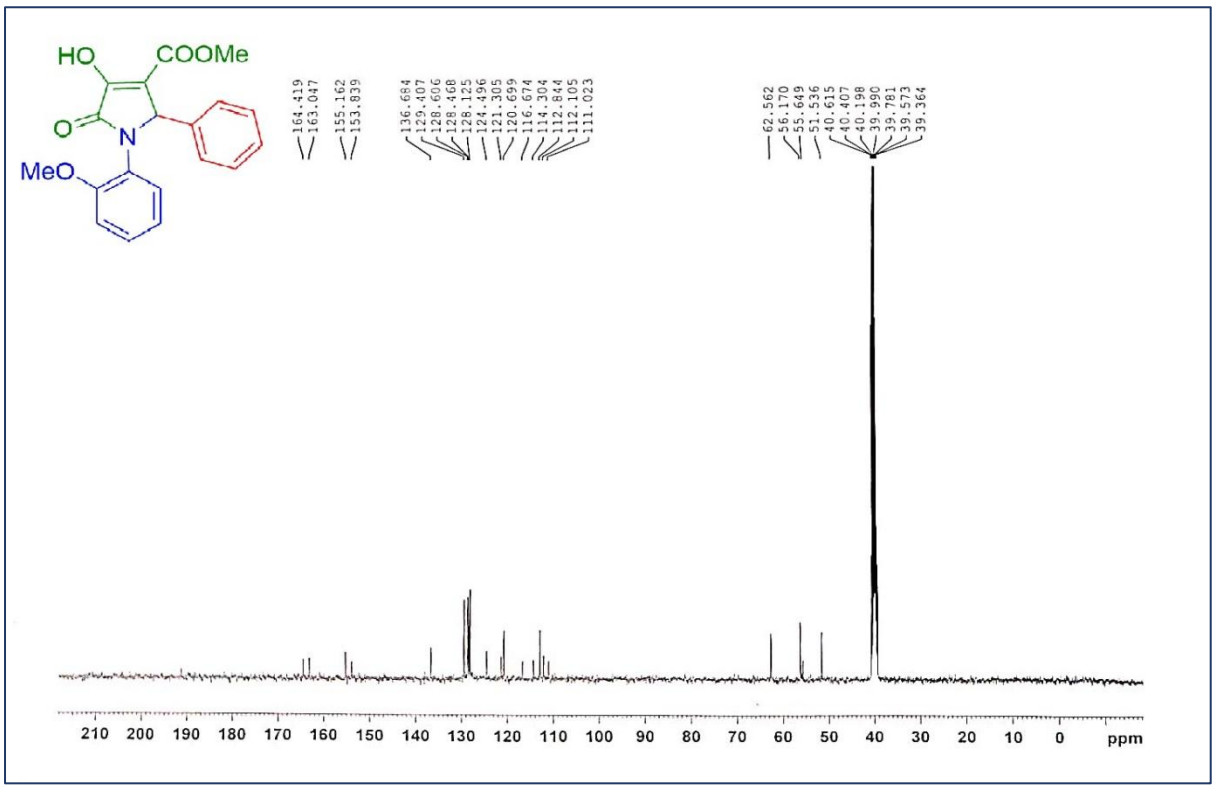

HO
COOMe
O
N
Cl
7.589
7.560
7.540
7.328
7.309
7.278
7.260
7.240
7.220
7.200
7.172
7.078
7.060
7.042
6.022
3.561
3.511
3.462
2.483
1.965
1.148
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.19
6.26
1.07
1.00
3.02

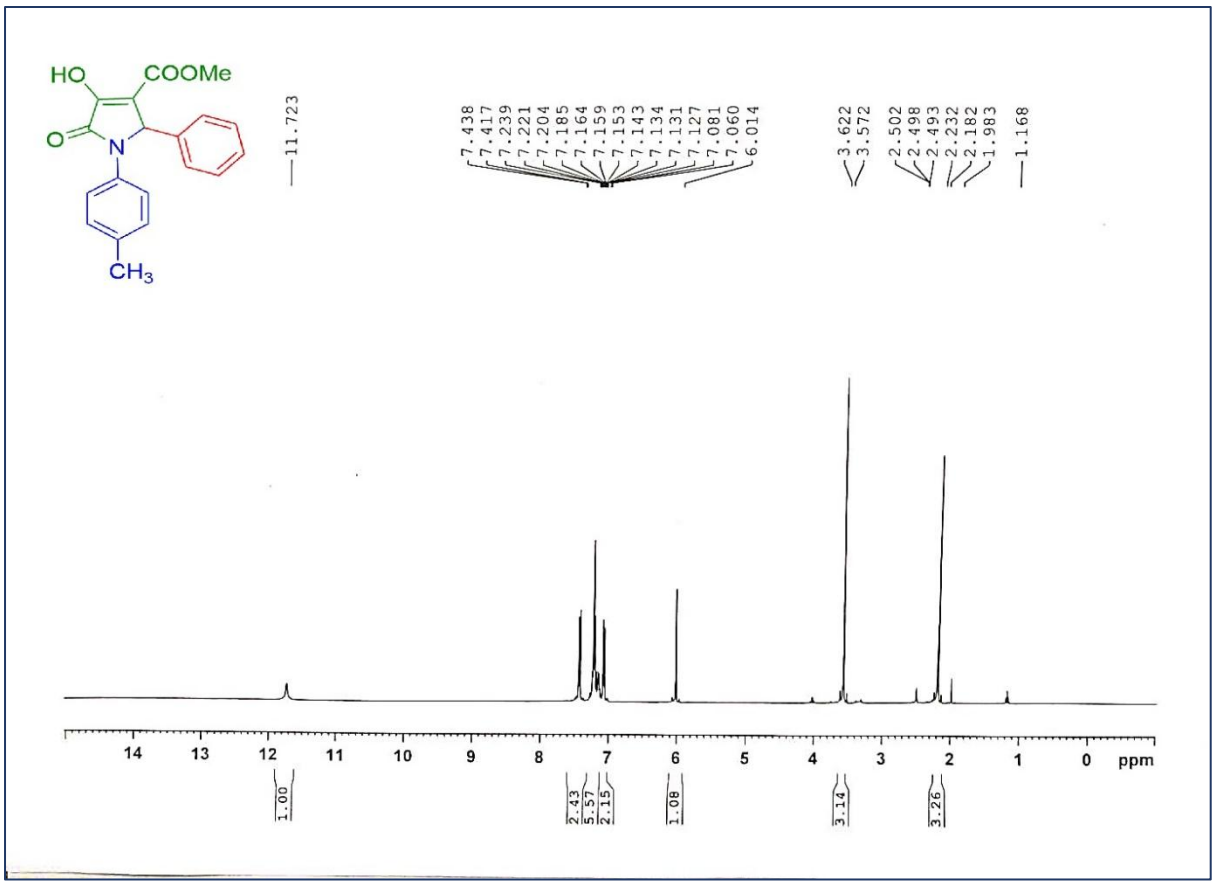

HO
COOMe
O
N
Cl
165.740
163.769
156.421
137.310
136.825
132.565
129.983
129.137
128.612
125.738
122.851
109.494
60.173
51.204
40.597
40.389
40.181
39.972
39.764
39.556
39.347
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

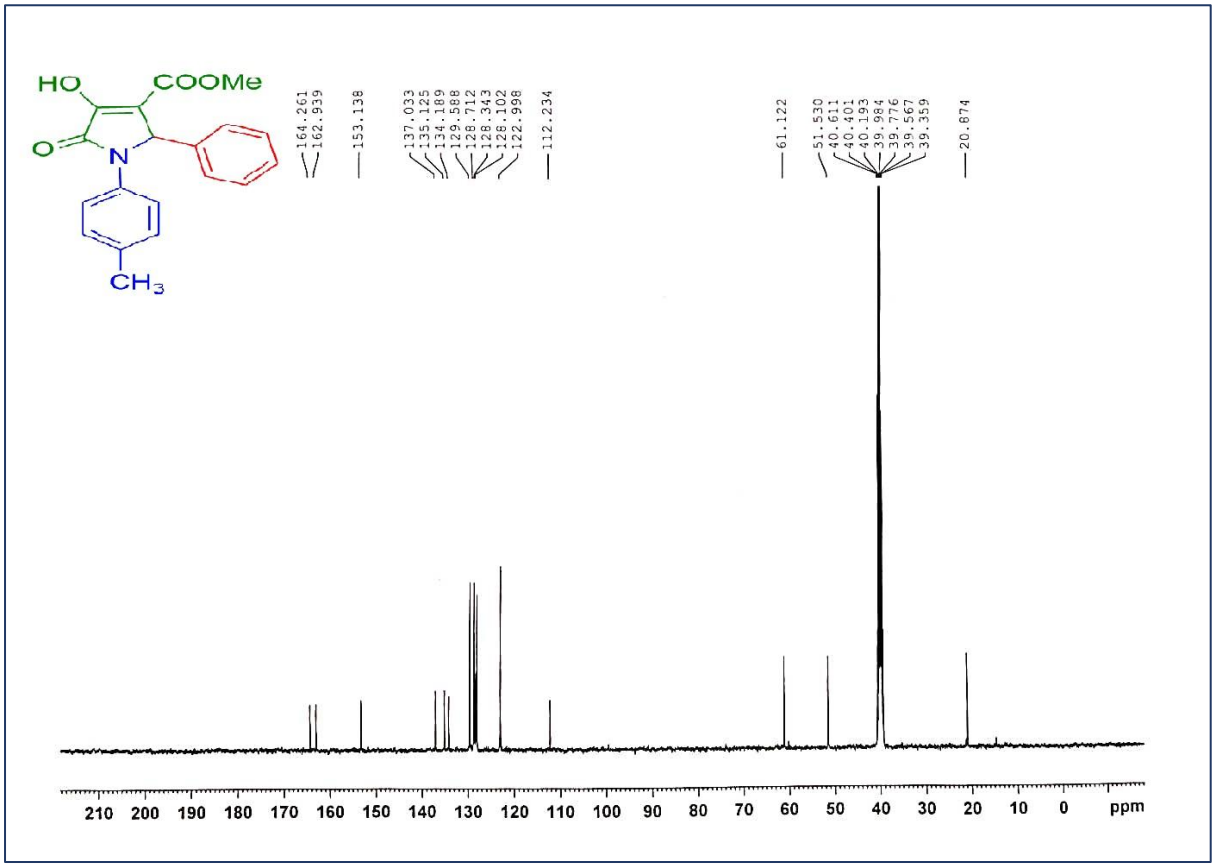

HO
COOMe
O
N
11.848
7.902
7.854
7.844
7.831
7.785
7.772
7.762
7.741
7.720
7.606
7.587
7.448
7.437
7.429
7.426
7.274
7.270
7.255
7.250
7.236
7.216
7.034
7.016
6.997
6.228
3.588
3.539
3.489
3.320
2.492
2.488
2.483
2.478
2.475
1.970
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.15
1.14
2.23
2.13
2.10
3.25
1.08
1.05
3.11

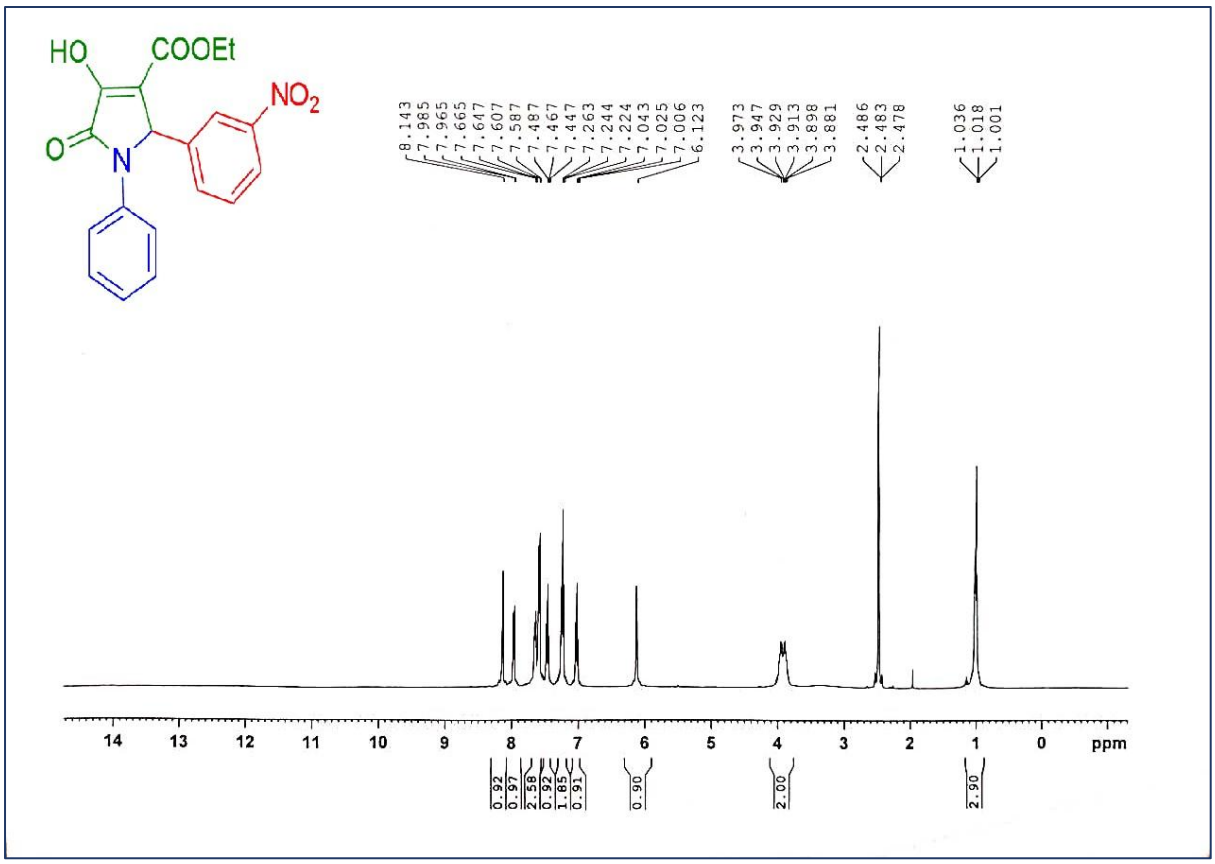

HO
COOMe
O
N
164.485
162.991
153.224
136.703
134.415
133.052
129.127
128.473
128.197
127.940
126.691
125.871
124.878
123.072
112.229
61.185
51.589
40.611
40.401
40.193
39.984
39.776
39.567
39.359
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

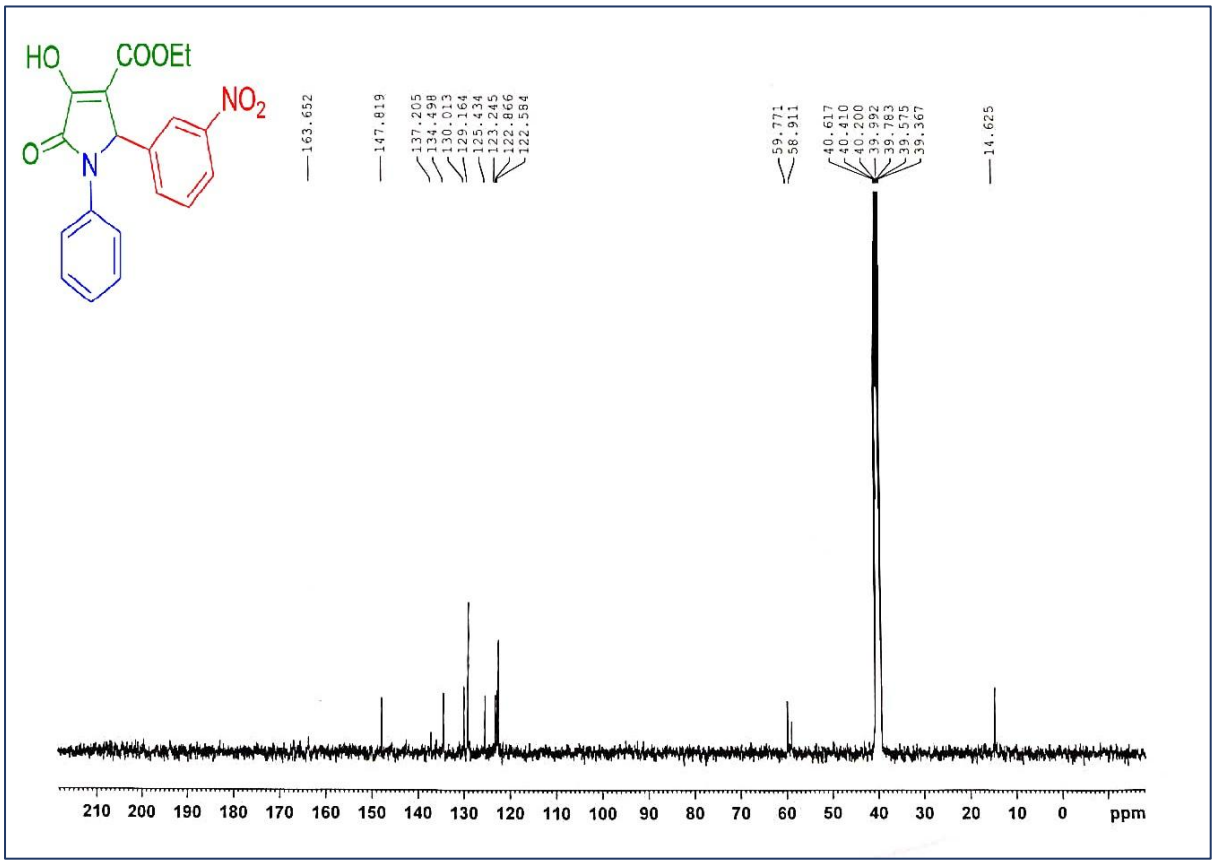

HO
COOMe
O
N
MeO
7.213
7.197
7.178
7.172
7.155
7.135
7.131
7.115
7.050
7.047
7.027
7.006
6.834
6.832
6.815
6.813
6.796
6.795
6.783
6.781
6.763
6.761
6.643
6.633
6.628
5.742
3.783
3.766
3.755
3.738
3.565
3.343
2.502
2.498
2.494
1.985
10
9
8
7
6
5
4
3
2
1
ppm
8.27
1.39
1.00
3.42
3.09

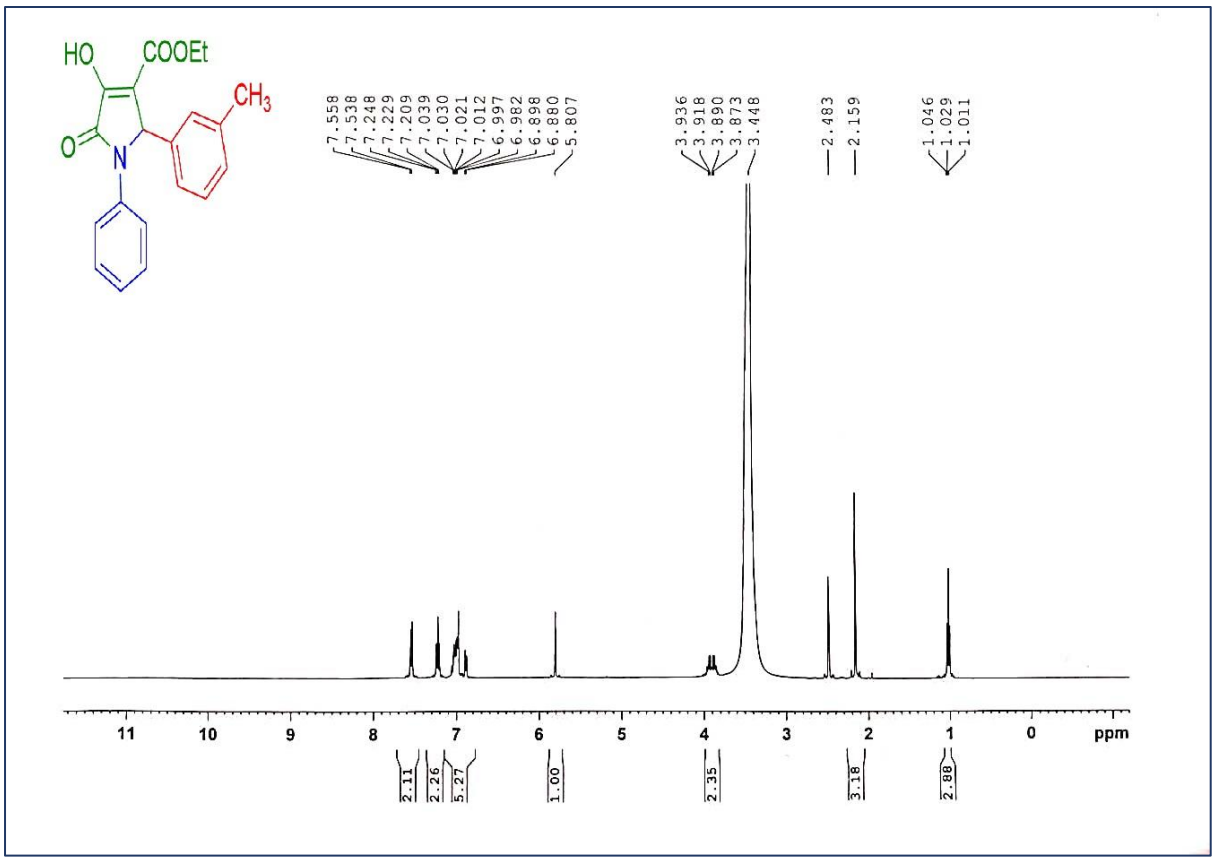

HO
COOMe
O
N
MeO
164.419
163.047
155.162
153.839
136.684
129.407
128.606
128.468
128.125
124.496
121.305
120.699
116.674
114.304
112.844
112.105
111.023
62.562
56.170
55.649
51.536
40.615
40.407
40.198
39.990
39.781
39.573
39.364
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

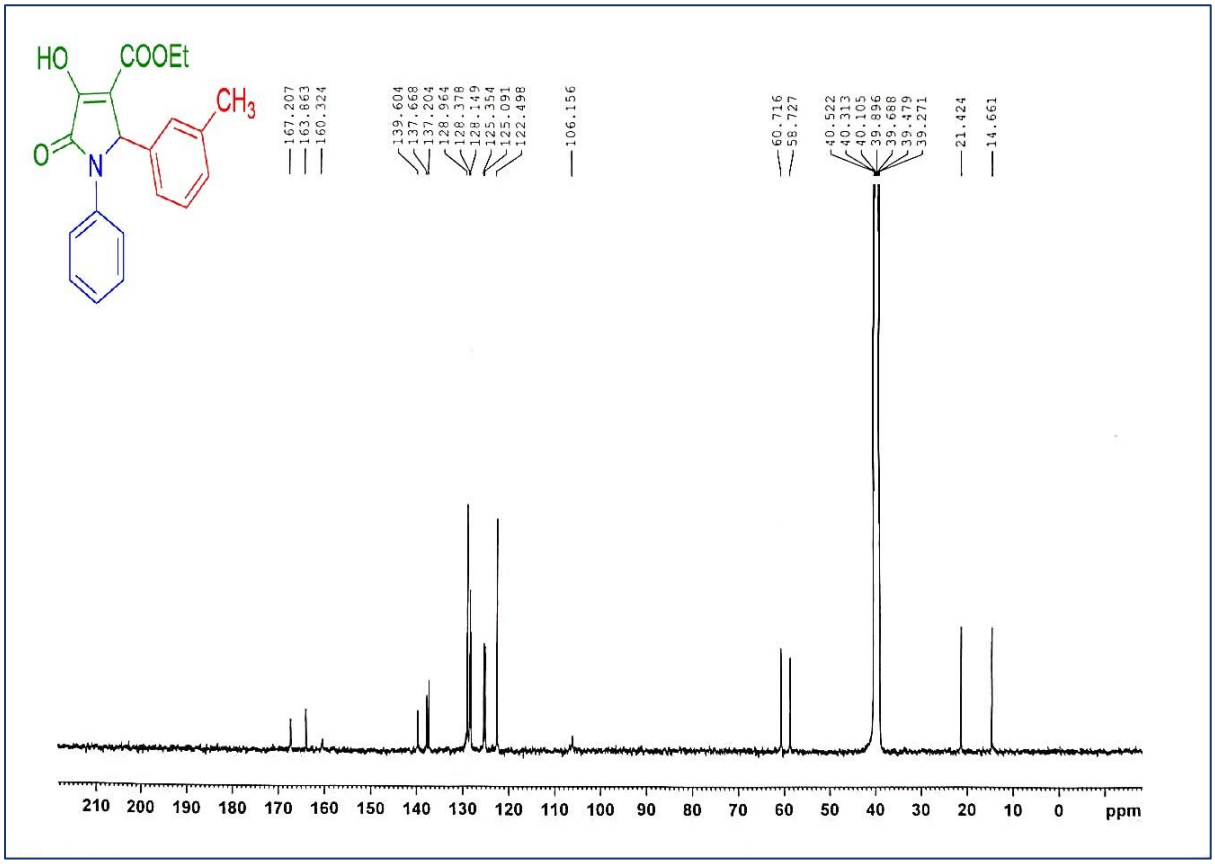

HO
COOMe
O
N
CH3
11.723
7.438
7.417
7.239
7.221
7.204
7.185
7.164
7.159
7.153
7.143
7.134
7.131
7.127
7.081
7.060
6.014
3.622
3.572
2.502
2.498
2.493
2.232
2.182
1.983
1.168
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.43
5.57
2.15
1.08
3.14
3.26

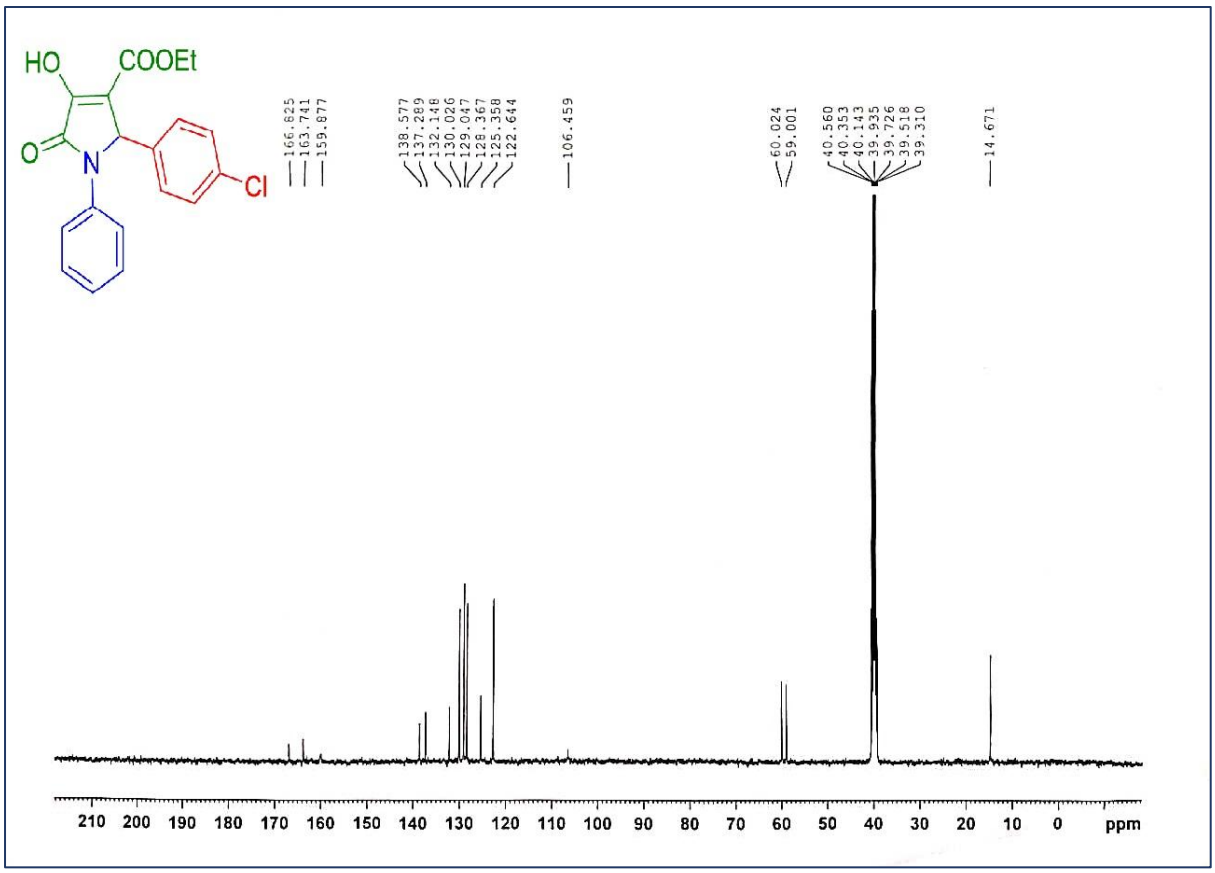

HO
COOMe
O
N
CH3
164.261
162.939
153.138
137.033
135.125
134.189
129.588
128.712
128.343
128.102
122.998
112.234
61.122
51.530
40.611
40.401
40.193
39.984
39.776
39.567
39.359
20.874
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

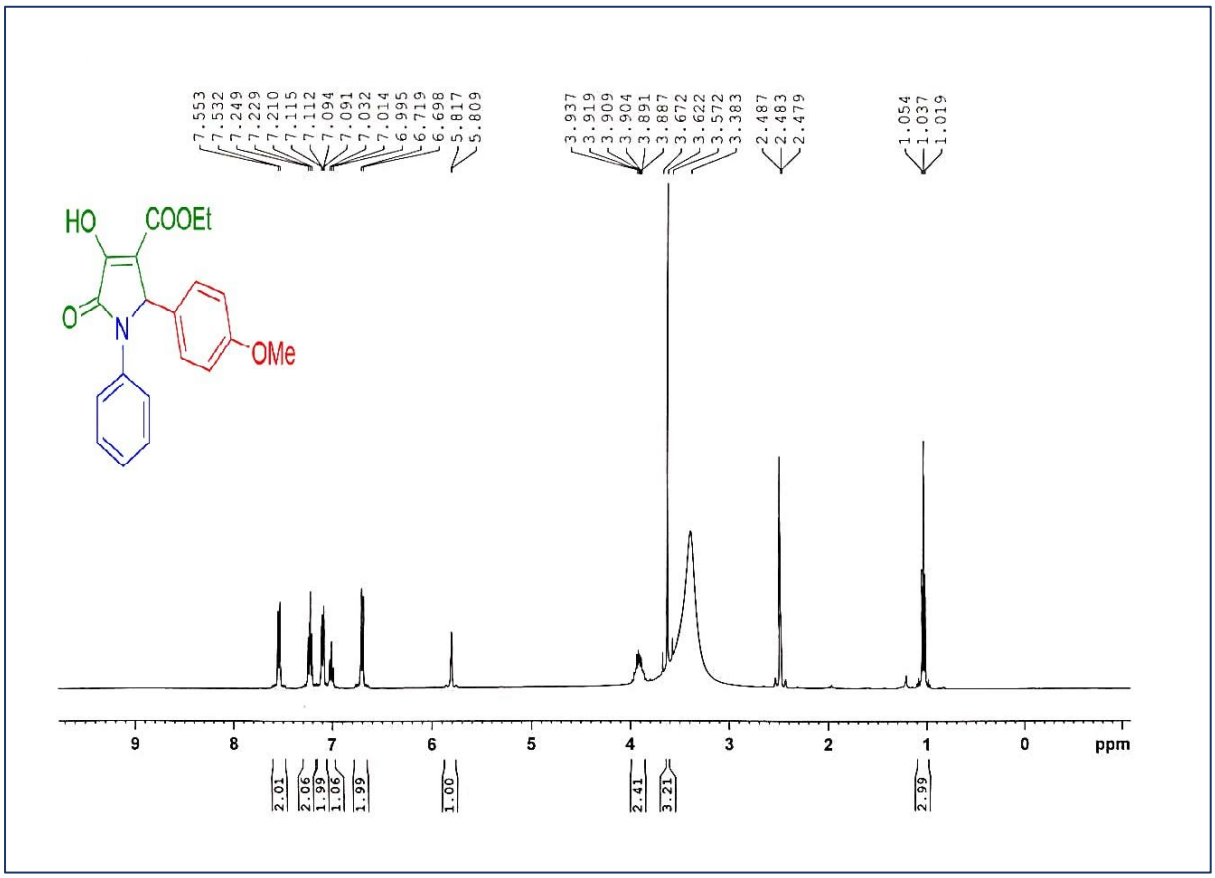

HO
COOEt
NO2
O
N
8.143
7.985
7.965
7.665
7.647
7.607
7.587
7.487
7.467
7.447
7.263
7.244
7.224
7.043
7.025
7.006
6.123
3.973
3.947
3.929
3.913
3.898
3.881
2.486
2.483
2.478
1.036
1.018
1.001
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.92
0.97
2.58
0.92
1.85
0.91
0.90
2.00
2.90

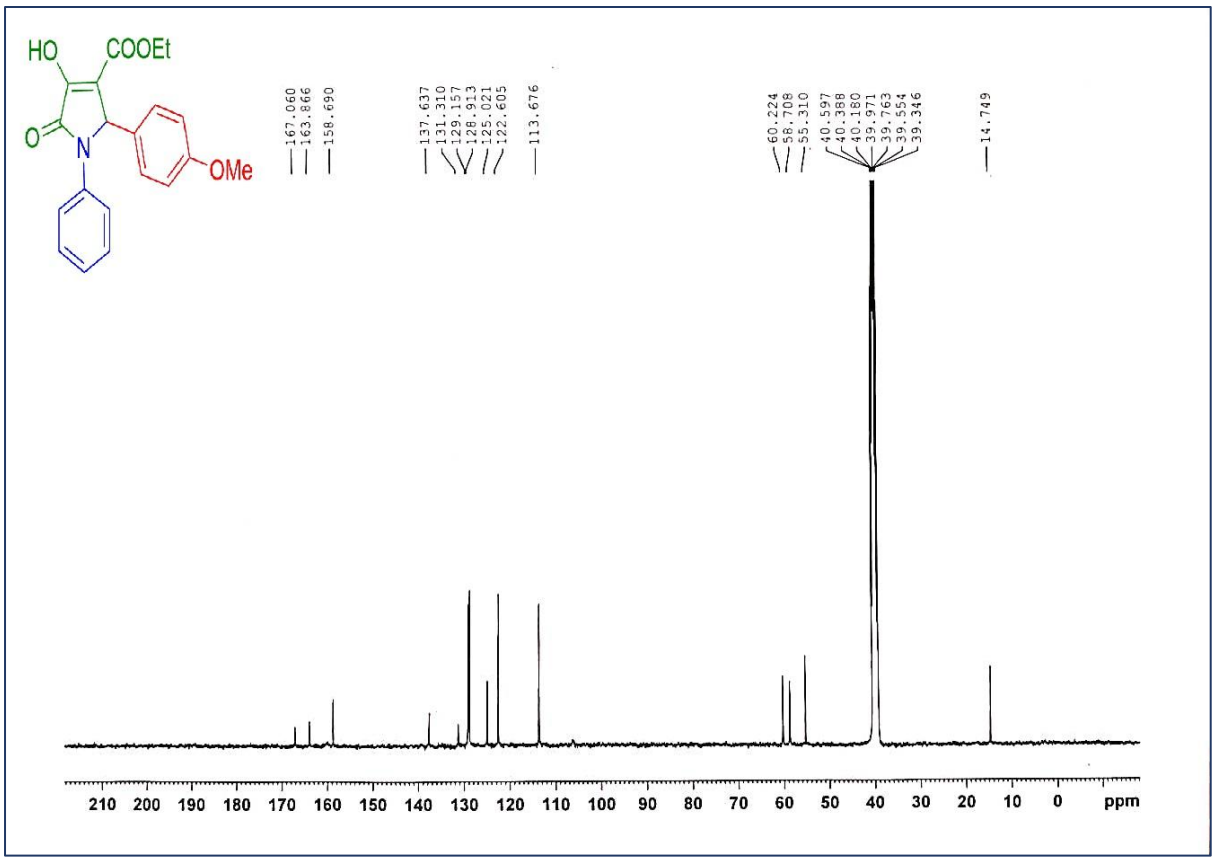

HO
COOEt
NO2
O
N
163.652
147.819
137.205
134.498
130.013
129.164
125.434
123.245
122.866
122.594
59.771
58.911
40.617
40.410
40.200
39.992
39.783
39.575
39.367
14.625
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

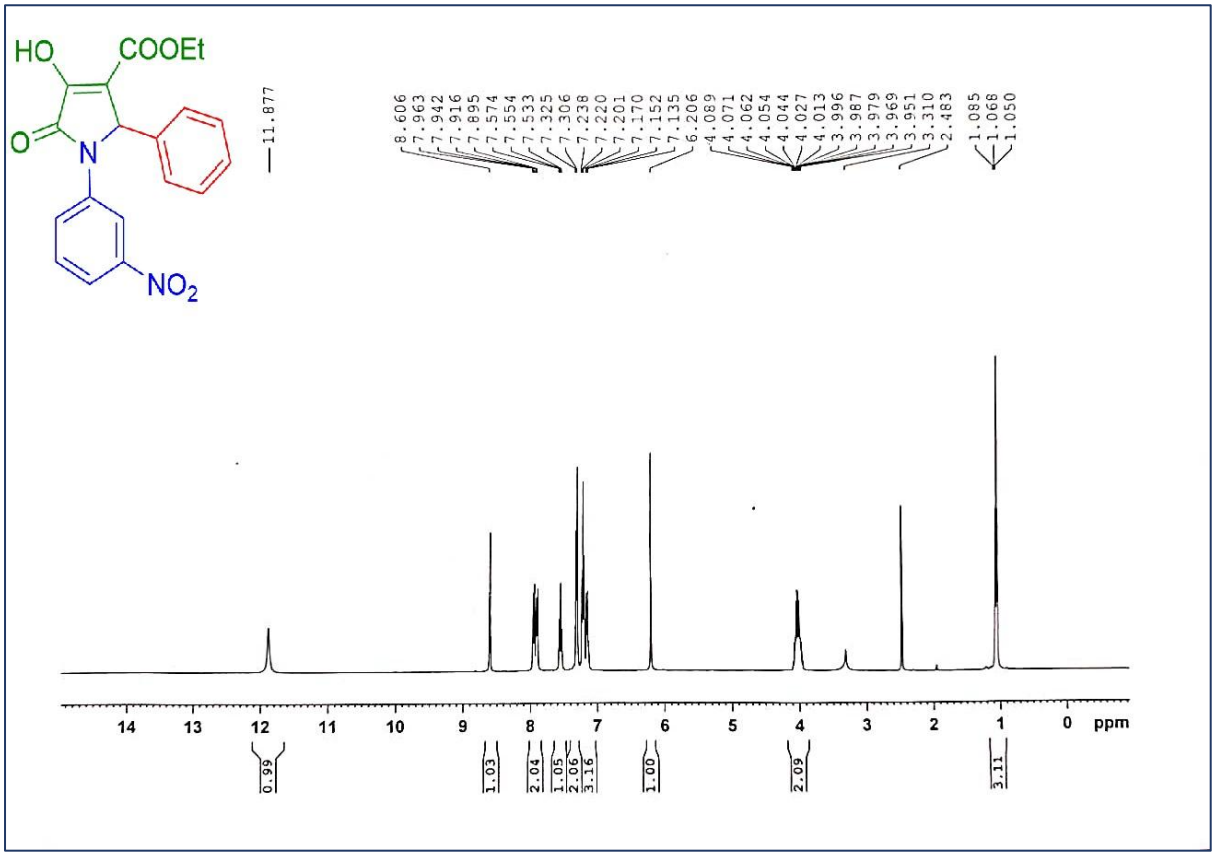

HO
COOEt
CH3
O
N
7.558
7.538
7.248
7.229
7.209
7.039
7.030
7.021
7.012
6.997
6.982
6.898
6.880
5.807
3.936
3.918
3.890
3.873
3.448
2.483
2.159
1.046
1.029
1.011
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.11
2.26
5.27
1.00
2.35
3.18
2.88

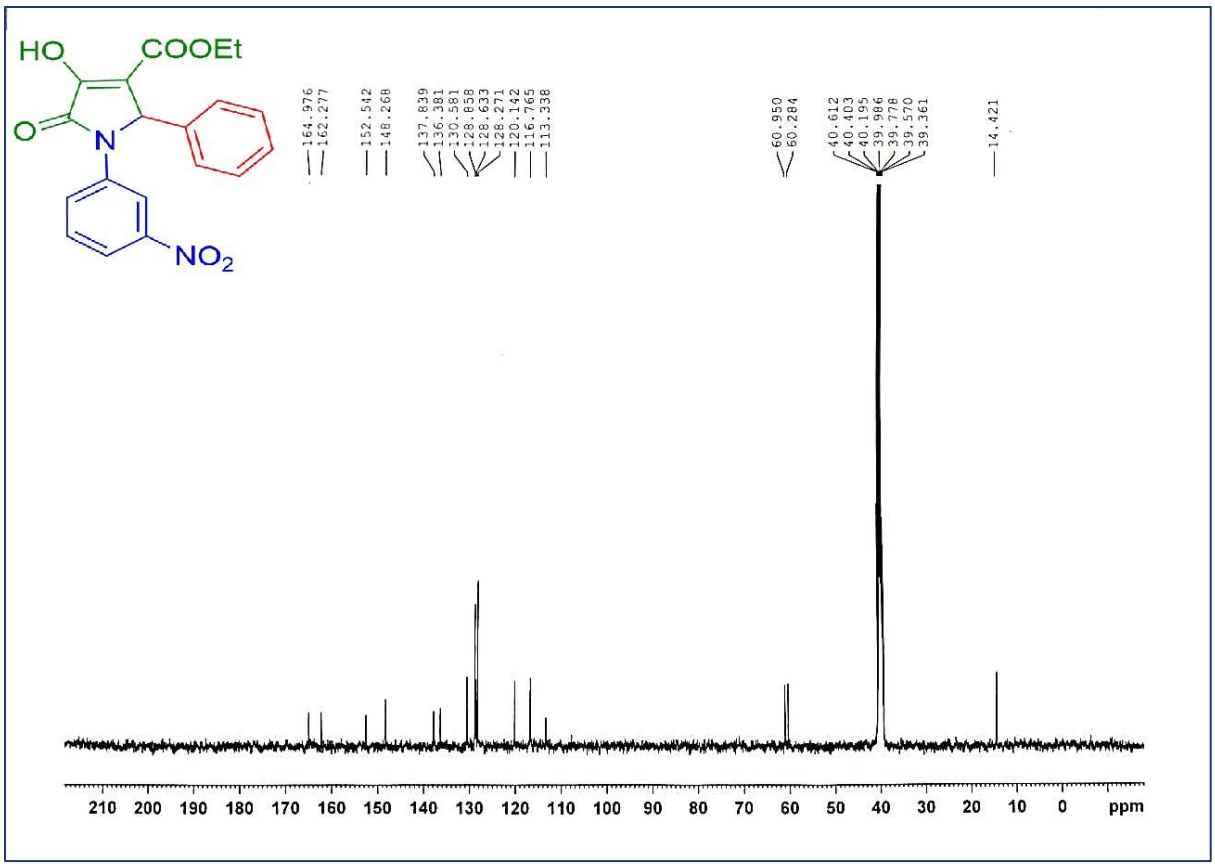

HO
COOEt
CH3
O
N
167.207
163.863
160.324
139.604
137.668
137.204
128.964
128.378
128.149
125.354
125.091
122.498
106.156
60.716
58.727
40.522
40.313
40.105
39.896
39.688
39.479
39.271
21.424
14.661
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

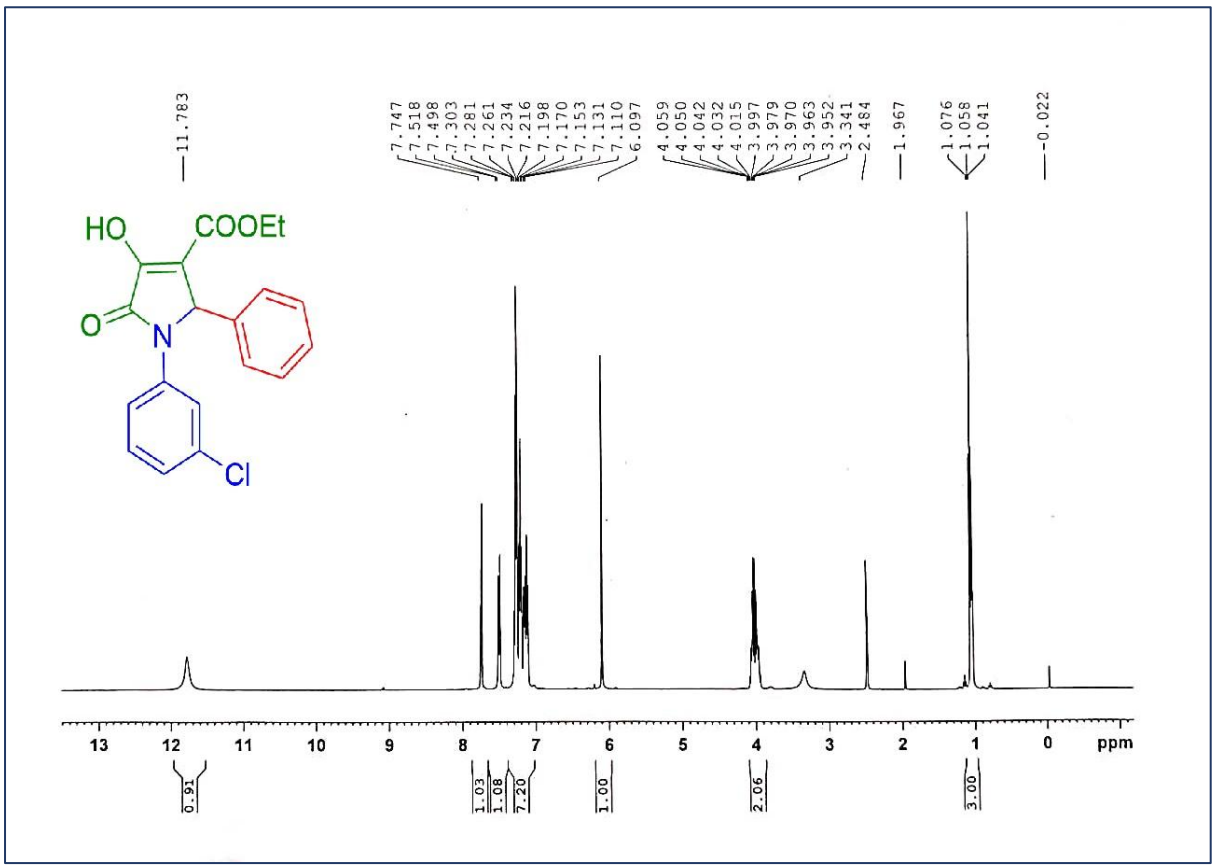

HO
COOEt
O
N
Cl
166.625
163.741
159.877
138.577
137.289
132.148
130.026
129.047
128.367
125.358
122.644
106.459
60.024
59.001
40.560
40.353
40.143
39.935
39.726
39.518
39.310
14.671
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

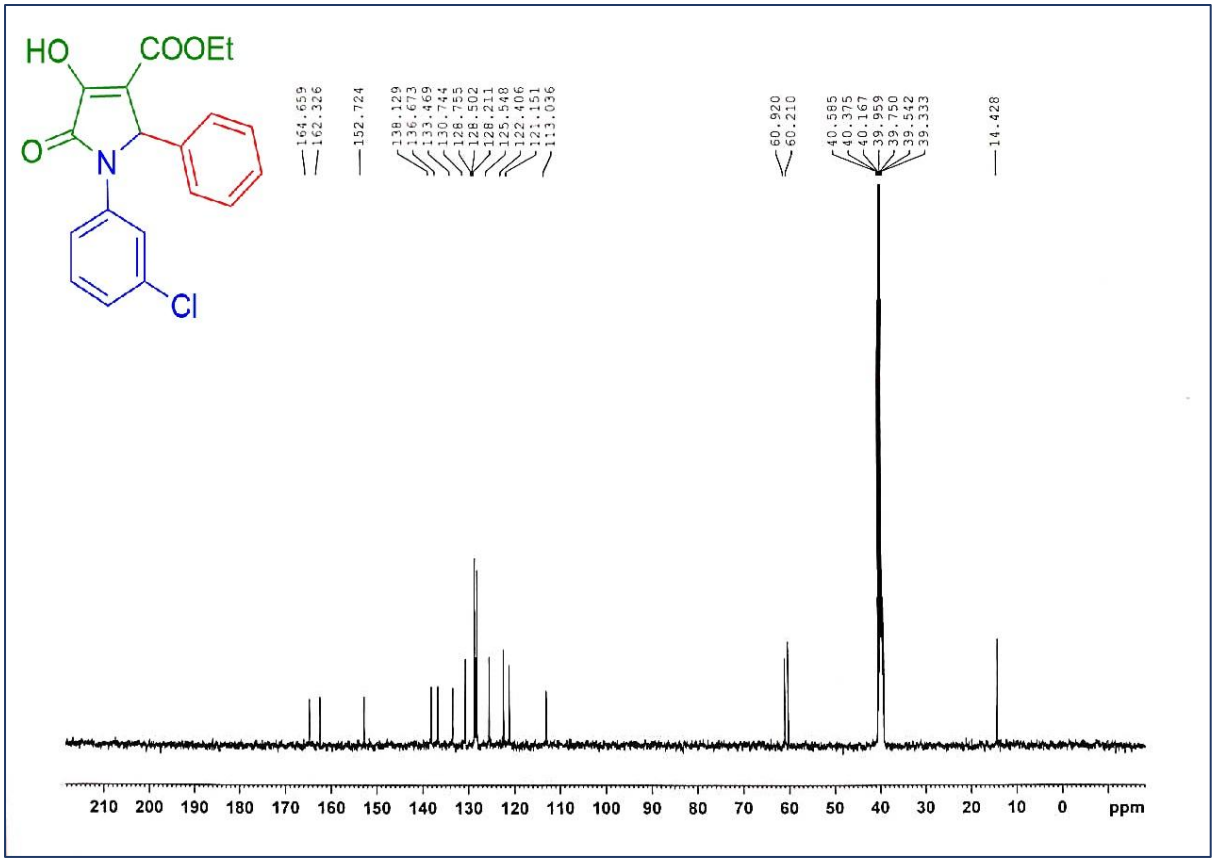

7.553
7.532
7.249
7.229
7.210
7.115
7.112
7.094
7.091
7.032
7.014
6.995
6.719
6.698
5.817
5.809
3.937
3.919
3.909
3.904
3.891
3.887
3.672
3.622
3.572
3.383
2.487
2.483
2.479
1.054
1.037
1.019
HO
COOEt
O
N
OMe
9 8 7 6 5 4 3 2 1 0 ppm
2.01
2.06
1.99
1.06
1.99
1.00
2.41
3.21
2.99


HO
COOEt
O
N
OMe
167.060
163.866
158.690
137.637
131.310
129.157
128.913
125.021
122.605
113.676
60.224
58.708
55.310
40.597
40.388
40.180
39.971
39.763
39.554
39.346
14.749
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


HO
COOEt
O
N
NO2
11.877
8.606
7.963
7.942
7.916
7.895
7.574
7.554
7.533
7.325
7.306
7.238
7.220
7.201
7.170
7.152
7.135
6.206
4.089
4.071
4.062
4.054
4.044
4.027
4.013
3.996
3.987
3.979
3.969
3.951
3.310
2.483
1.085
1.068
1.050
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
1.03
2.04
1.05
2.06
3.16
1.00
2.09
3.11


HO
COOEt
O
N
NO2
164.976
162.277
152.542
148.268
137.839
136.381
130.581
128.858
128.633
128.271
120.142
116.765
113.338
60.950
60.284
40.612
40.403
40.195
39.986
39.778
39.570
39.361
14.421
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.783
7.747
7.518
7.498
7.303
7.281
7.261
7.234
7.216
7.198
7.170
7.153
7.131
7.110
6.097
4.059
4.050
4.042
4.032
4.015
3.997
3.979
3.970
3.963
3.952
3.341
2.484
1.967
1.076
1.058
1.041
0.022
HO
COOEt
O
N
Cl
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.91
1.03
1.08
7.20
1.00
2.06
3.00


HO
COOEt
O
N
Cl
164.659
162.326
152.724
138.129
136.673
133.469
130.744
128.755
128.502
128.211
125.548
122.406
121.151
113.036
60.920
60.210
40.585
40.375
40.167
39.959
39.750
39.542
39.333
14.428
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm