An economical synthesis of benzodiazepine derivatives using activated carbon of Thymus plant (ACT) modified IRMOF-3 core-shell as a potential ecofriendly catalyst

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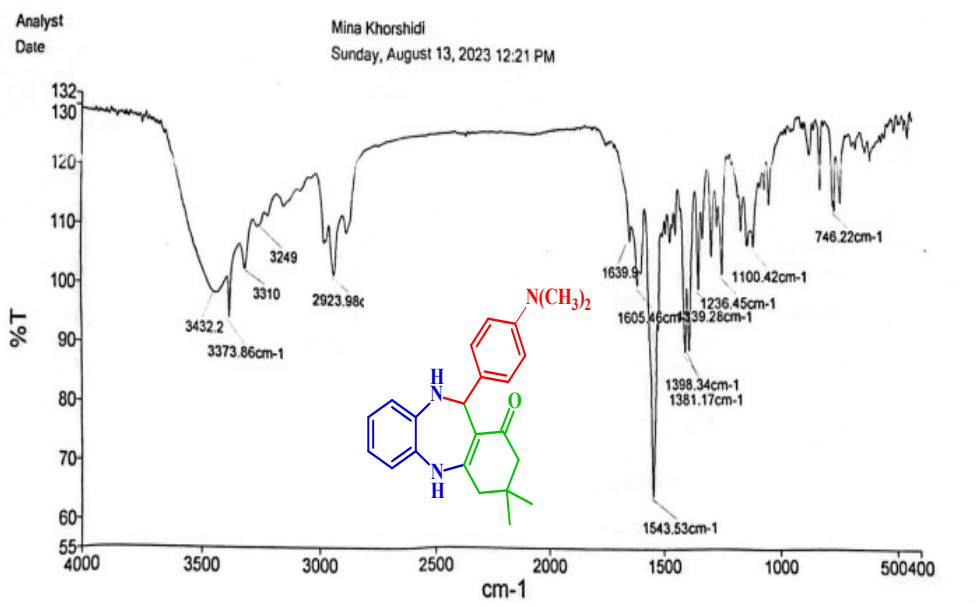
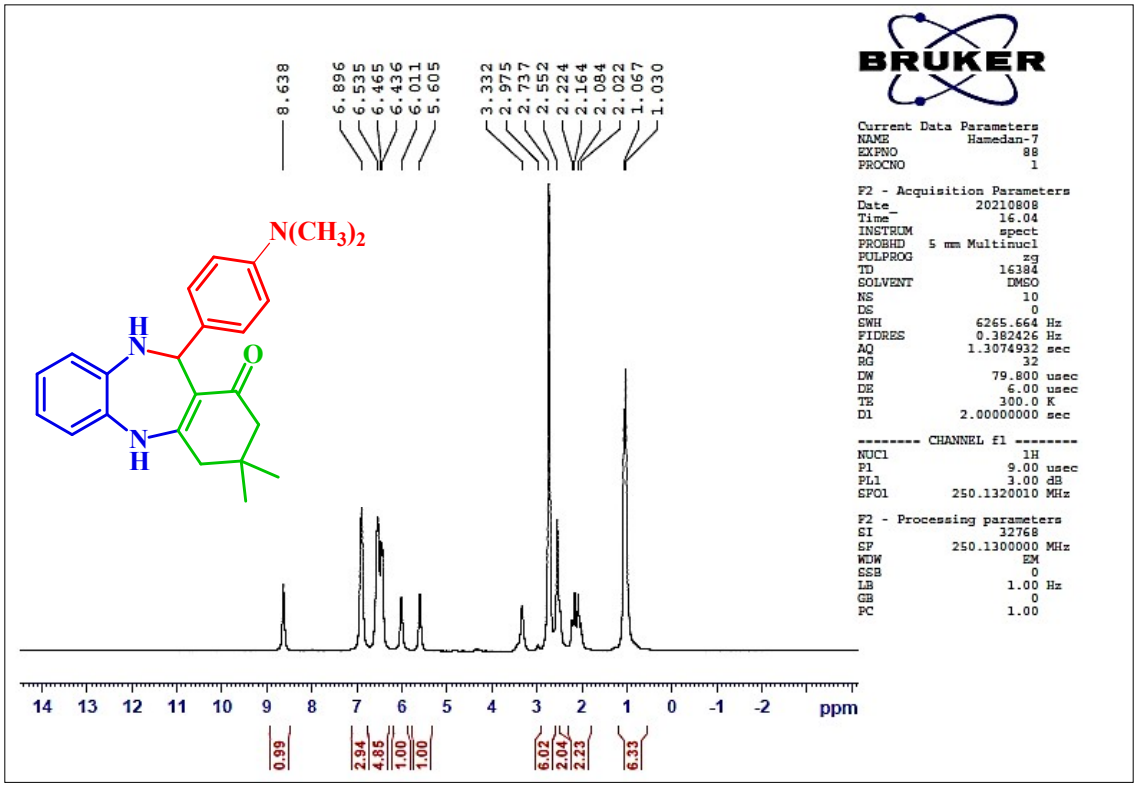
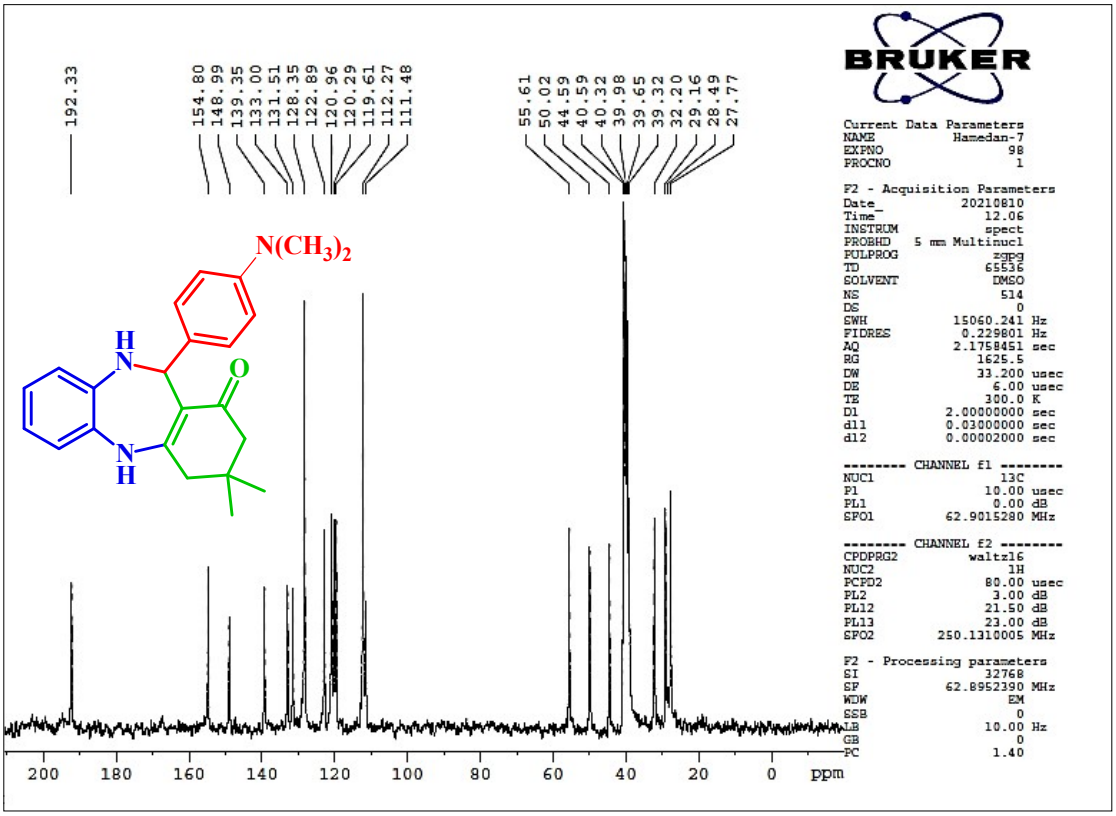
Sample characterization

The samples were pressed using the KBr pellet method and analyzed at a constant temperature of 20°C, a humidity of 35%, and a scanning range of 400-4000 cm^{-1} using a Perkin-Elmer Spectrum infrared spectrometer. Field emission scanning electron microscopy was applied to examine the surface morphology of ACT and ACT@IRMOF-3 (with an accelerating voltage of around 20 KV FE-SEM QUANTA FEG250, Republic of Czech). Transmission electron microscopy (TEM; JEOL, JEM 2100, 120 KV acceleration voltages, Japan) was applied to analyze the particle size. 1H NMR and 13CNMR were measured in DMSO-d_6 using TMS as an internal standard on a Bruker 250 MHz spectrometer. The thermal stability of ACT and ACT@IRMOF-3 samples was investigated using a Perkin Elmer TGA/DTA instrument at a temperature of 25-600 °C. Specific surface areas of catalysts were measured by N2 physisorption at liquid nitrogen temperature using a Micromeritics Tristar 3000 surface area analyzer and the standard multipoint BET analysis method. Samples were degassed in flowing N2 for 12 h at 200 °C before N_2 physisorption measurements. The specific surface areas were evaluated using the Brunauer-Emmett-Teller (BET) method in the p/p_0 range of 0.05 - 0.2.

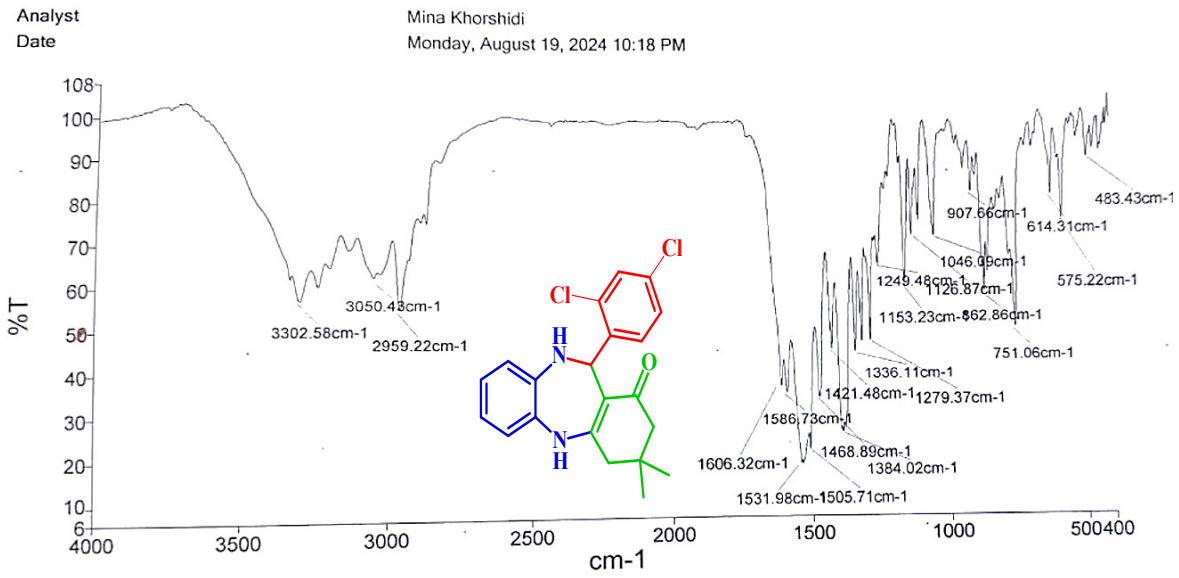
^{1}H NMR (250 MHz, DMSO) δ 1.03 (s, 3H, CH_3), 1.07 (s, 3H, CH_3), 2.02 (q, J = 15.0 Hz, 2H, -CH_2), 2.55 (s, 2H, -CH_2-C=O), 2.74 (s, $(CH_3)_2$N), 5.60 (s, 1H, C-H), 6.01 (s, NH), 6.44-6.90 (m, 8H Ar), 8.64 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 27.8, 28.8, 32.2, 44.6, 50.0, 55.6, 111.5, 112.3, 119.6, 120.3, 120.9, 122.9, 128.3, 131.5, 133.0, 139.3, 149.0, 154.8, 192.3.

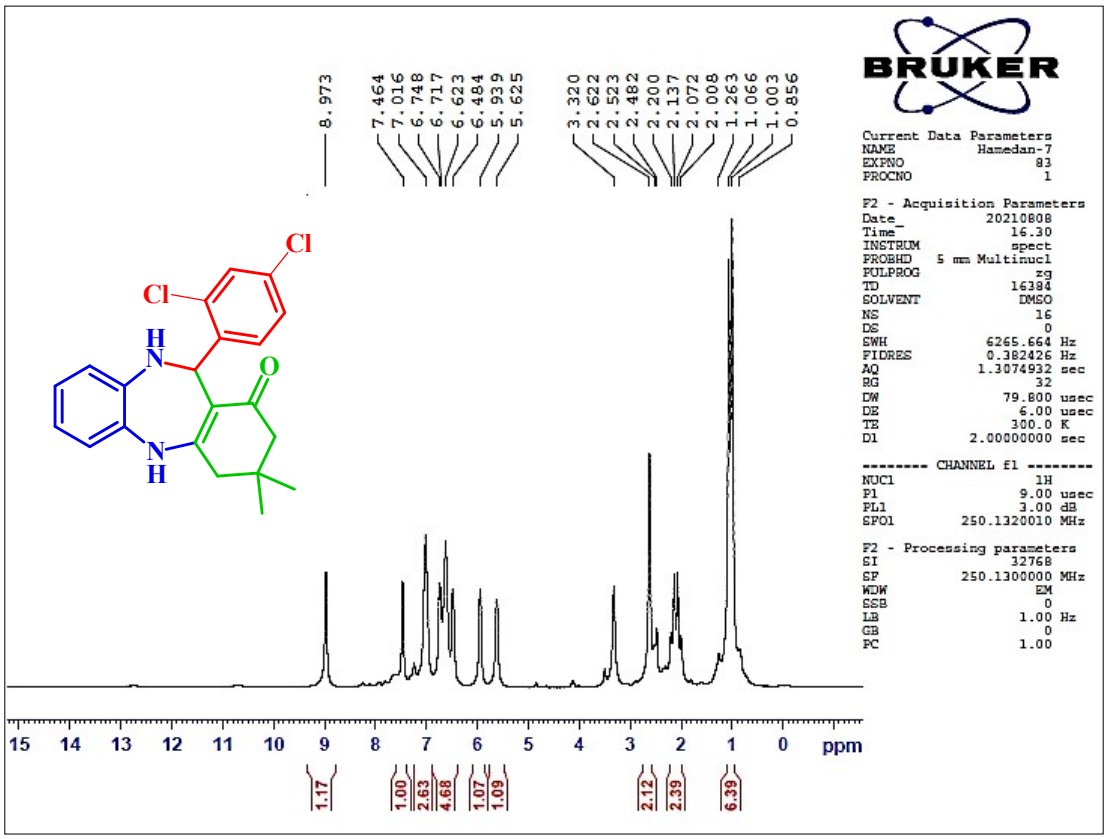






^{1}H NMR (250 MHz, DMSO) δ 1.00 (s, 3H, CH_3), 1.07 (s, 3H, CH_3), 2.10 (q, J = 16.2 Hz, 2H, -CH_2), 2.62 (s, 2H, -CH_2-C=O), 5.62 (s, 1H, C-H), 5.94 (s, 1H Ar), 6.48-7.02 (m, 6H Ar), 7.46 (s, NH), 8.97 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 28.3, 32.2, 44.5, 49.8, 54.6, 109.1, 120.6, 121.3, 123.6, 126.8, 129.1, 132.0, 132.2, 134.6, 137.7, 140.6, 156.0, 192.5.





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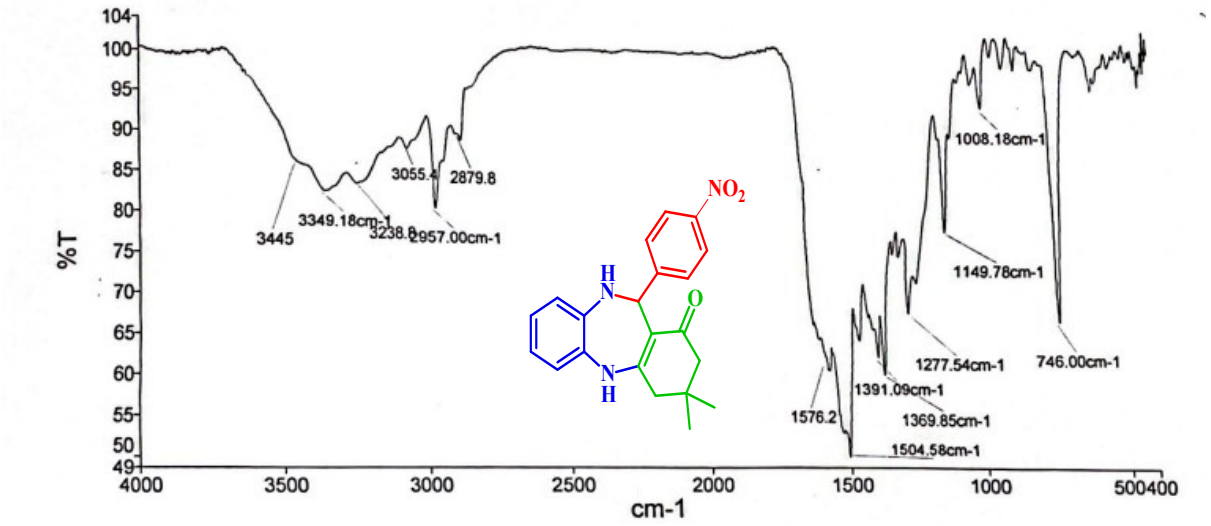
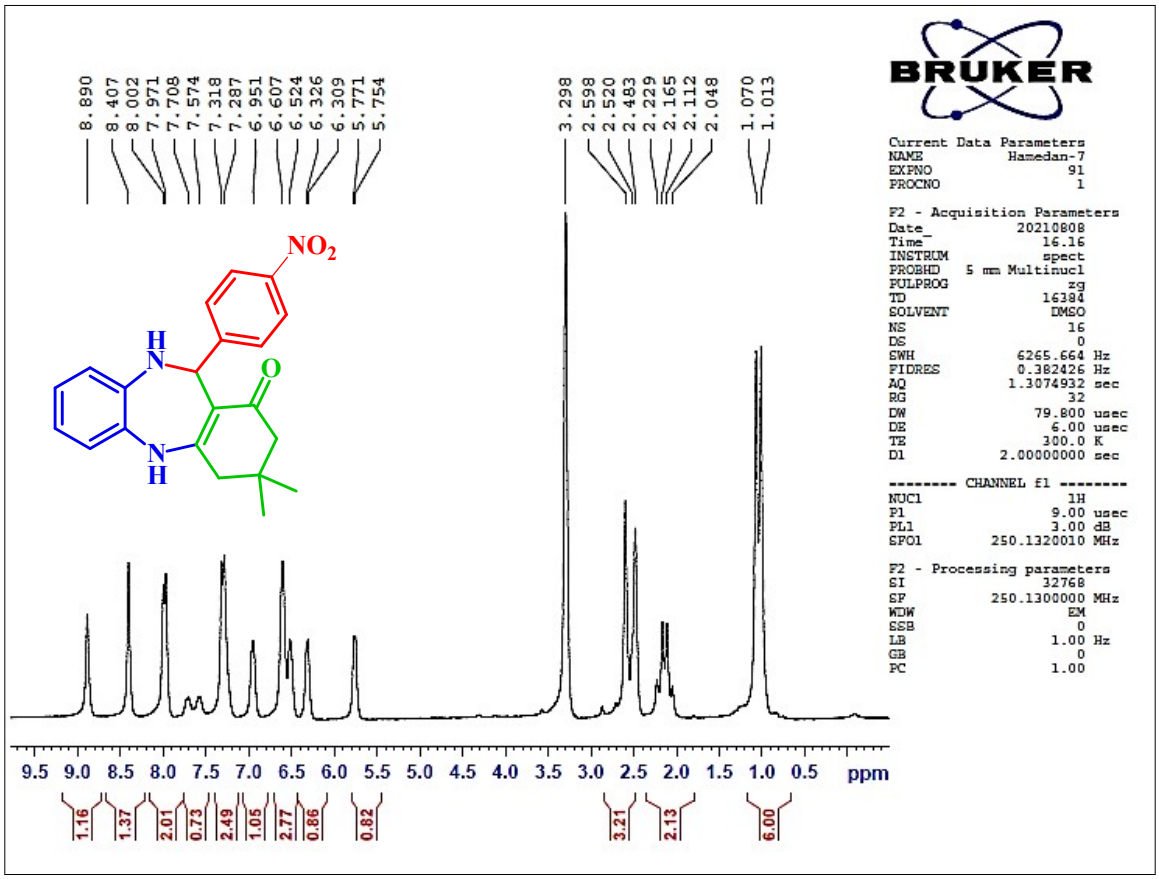
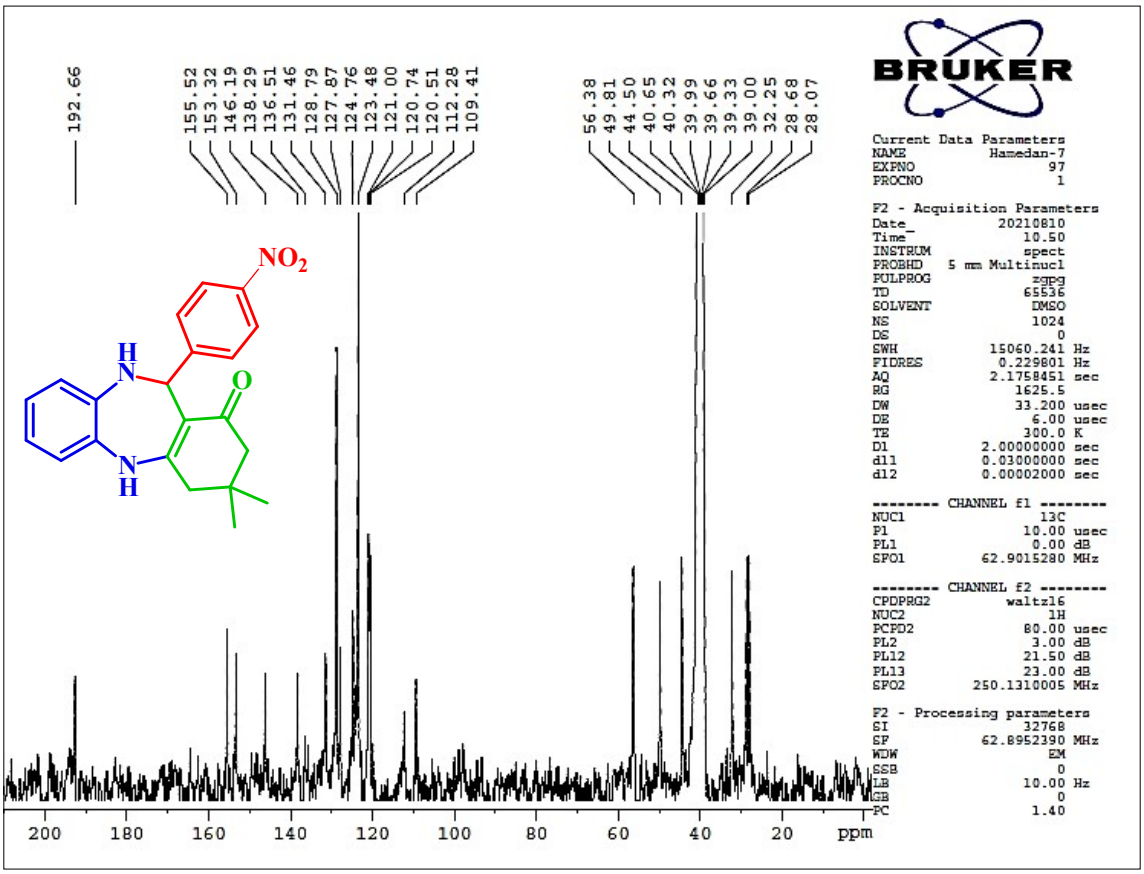
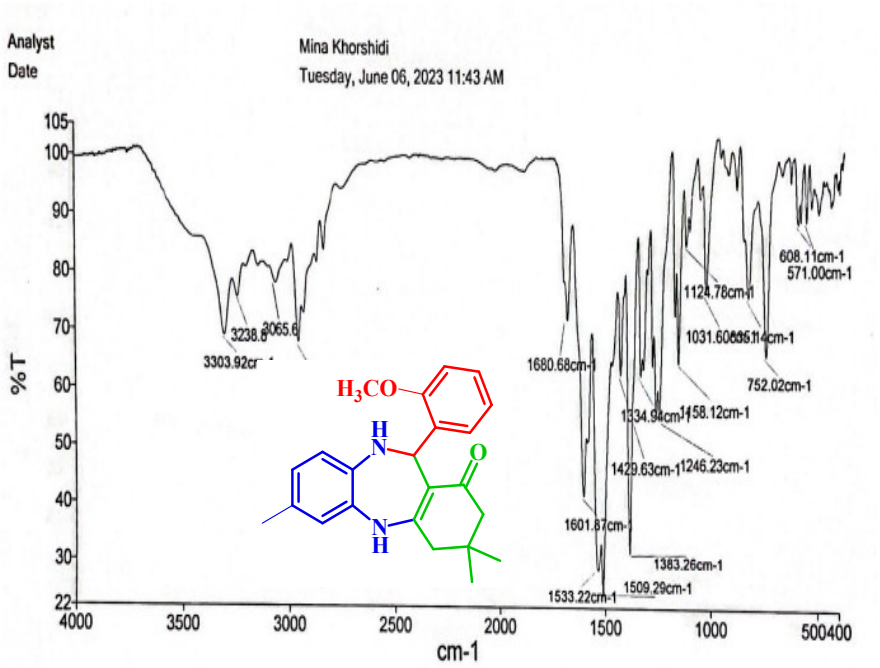
^{13}C NMR (63 MHz, DMSO) δ 28.4, 32.2, 44.5, 49.8, 56.4, 109.4, 112.3, 120.7, 123.5, 124.8, 127.9, 128.8, 131.5, 138.3, 146.2, 153.3, 155.5, 192.7.







^{1}H NMR (250 MHz, DMSO) δ 1.06 (s, 6H, 2CH_3), 1.96 (s, 3H, CH_3), 2.11 (s, 2H, -CH_2), 2.54 (s, 2H, -CH_2-C=O), 3.88 (s, OCH_3), 5.26 (s, 1H), 5.91-6.88 (m, 8H Ar), 7.00 (s, NH), 8.72 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 20.6, 28.5, 32.2, 44.6, 50.0, 52.7, 55.9, 109.3, 110.9, 119.8, 120.3, 120.9, 123.8, 126.9, 128.1, 129.1, 131.8, 136.3, 138.7, 155.8, 157.5, 192.2.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Mina Khorshidi
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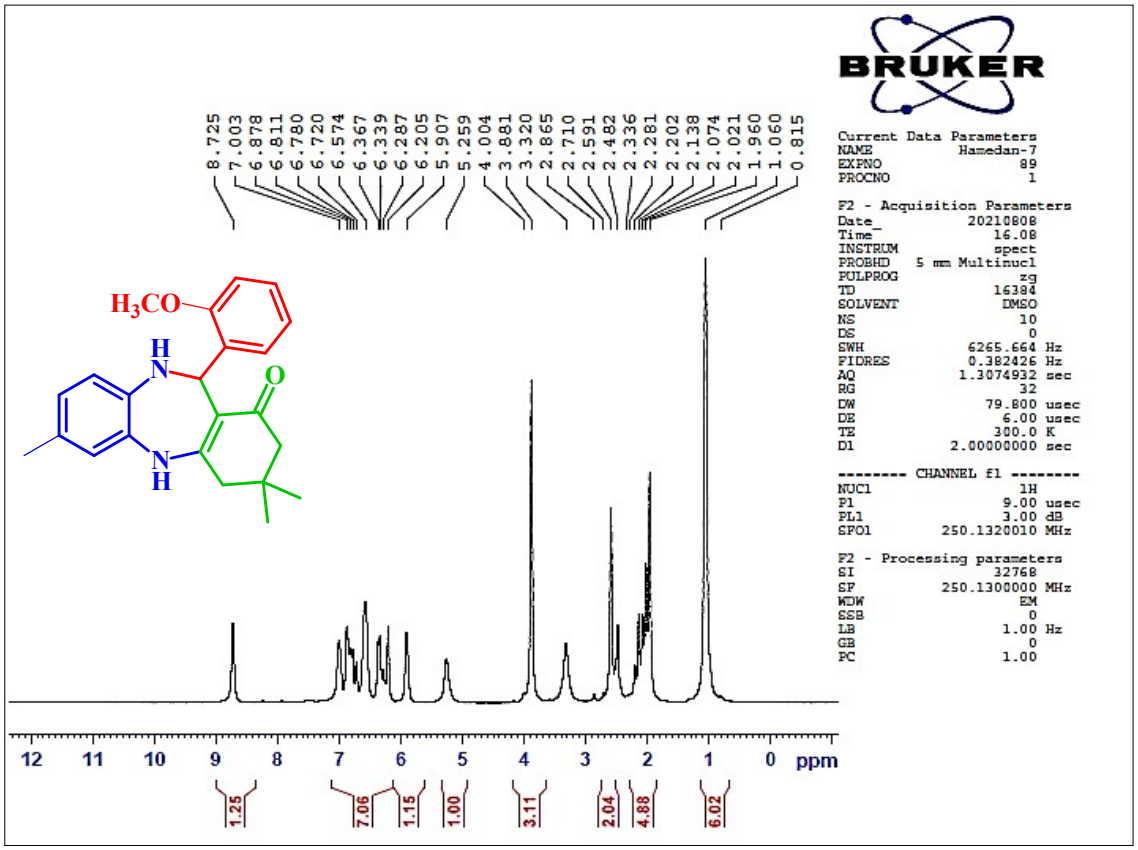
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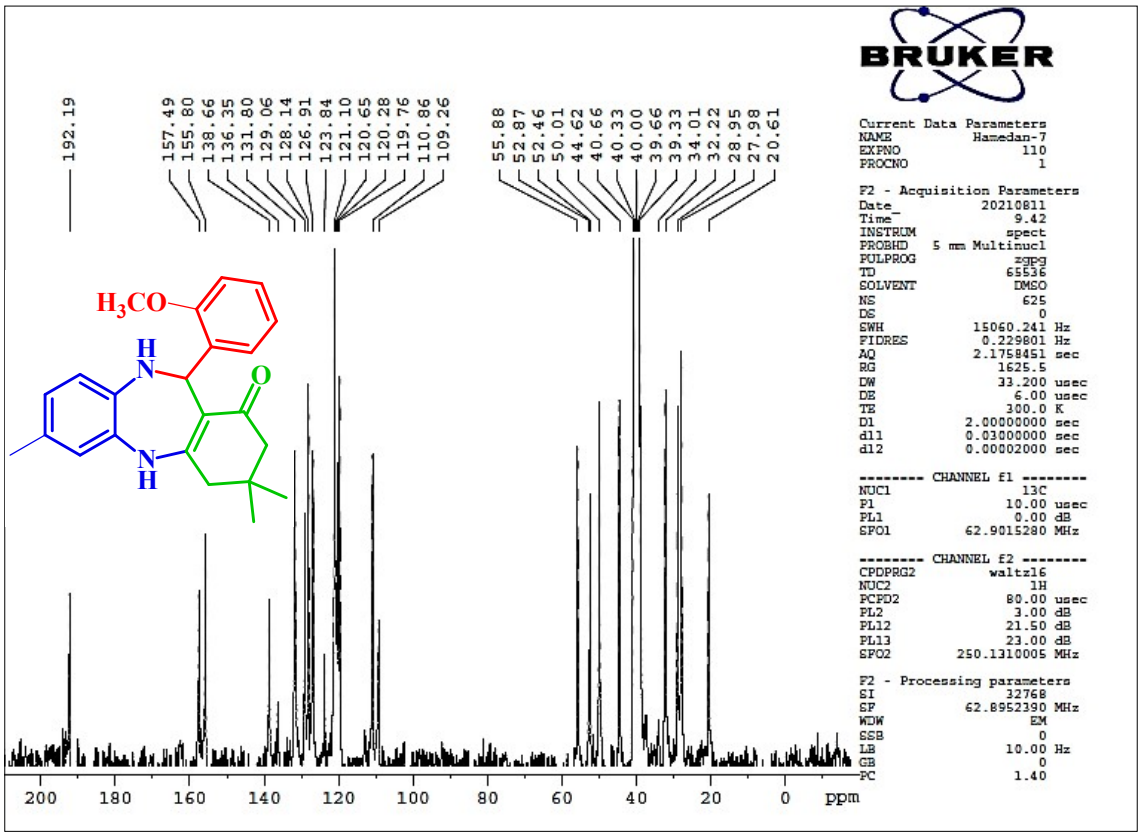
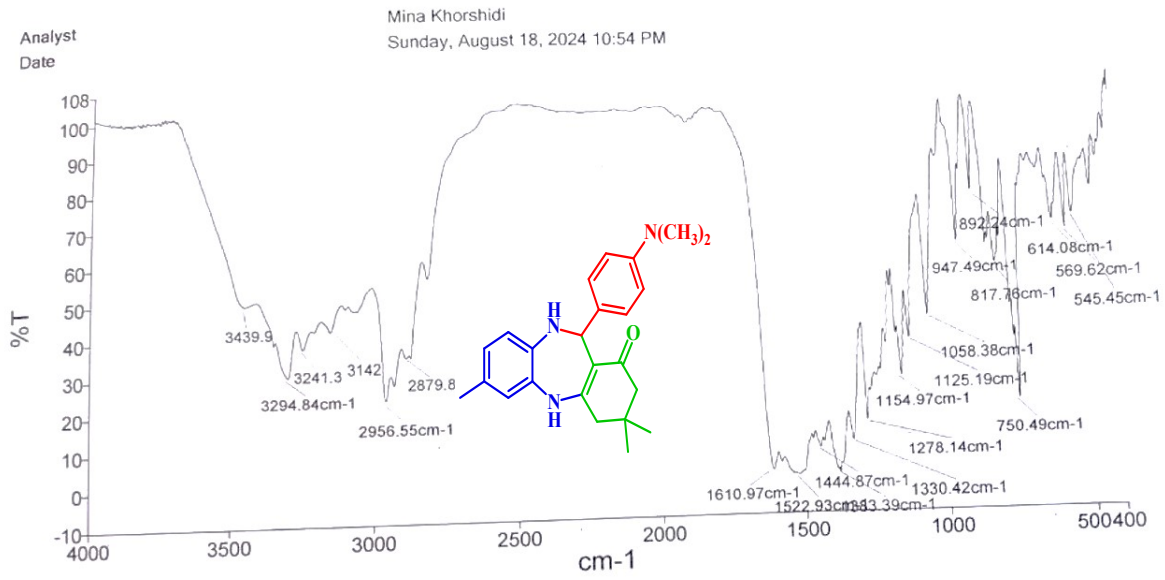


^{1}H NMR (250 MHz, DMSO) δ 1.02 (s, 3H, CH_3), 1.06 (s, 3H, CH_3), 2.03 (s, 3H, CH_3), 2.18 (s, 2H, -CH_2), 2.53 (s, 2H, -CH_2-C=O), 2.74 (s, $(CH_3)_2$N), 5.59 (s, 1H, C-H), 5.87-6.81 (m, 7H Ar), 6.91 (s, NH), 8.60 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 20.7, 27.7, 29.2, 32.2, 44.6, 50.0, 55.5, 111.2, 111.9, 120.7, 123.7, 128.4, 129.0, 131.6, 133.1, 136.9, 139.1, 149.0, 154.7, 192.1.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Mina Khorshidi
Sunday, August 18, 2024 10:54 PM
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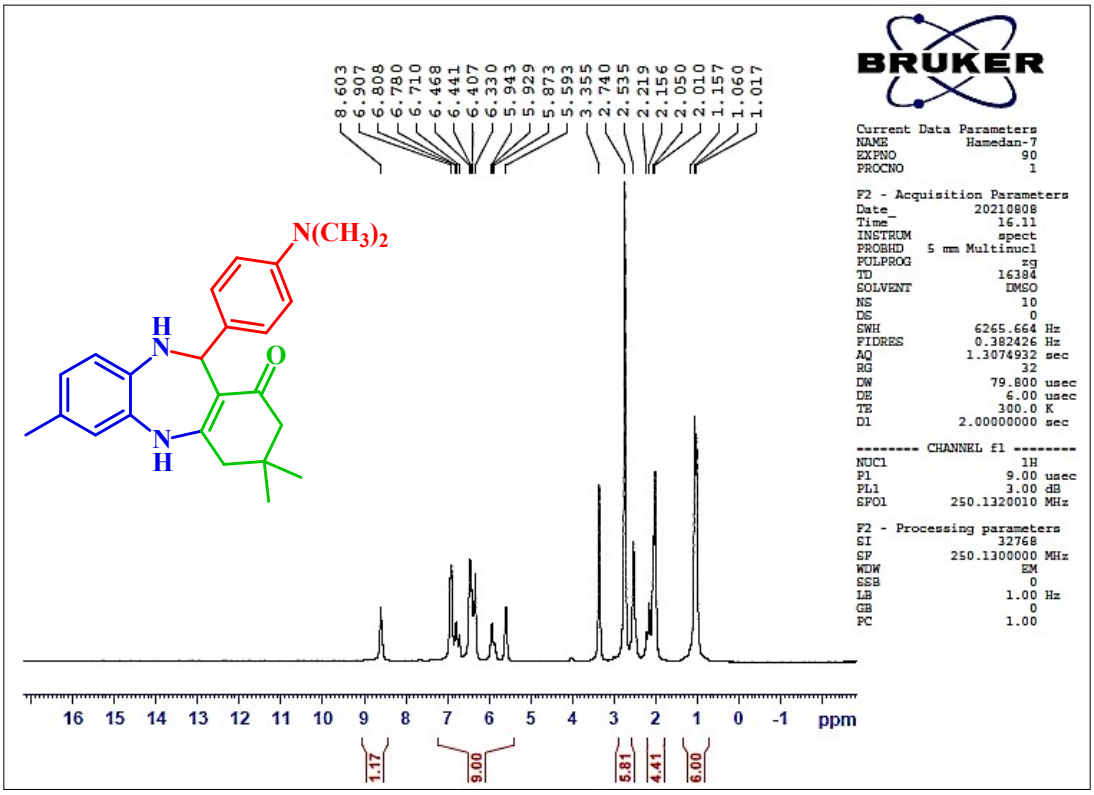
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TE 300.0 K
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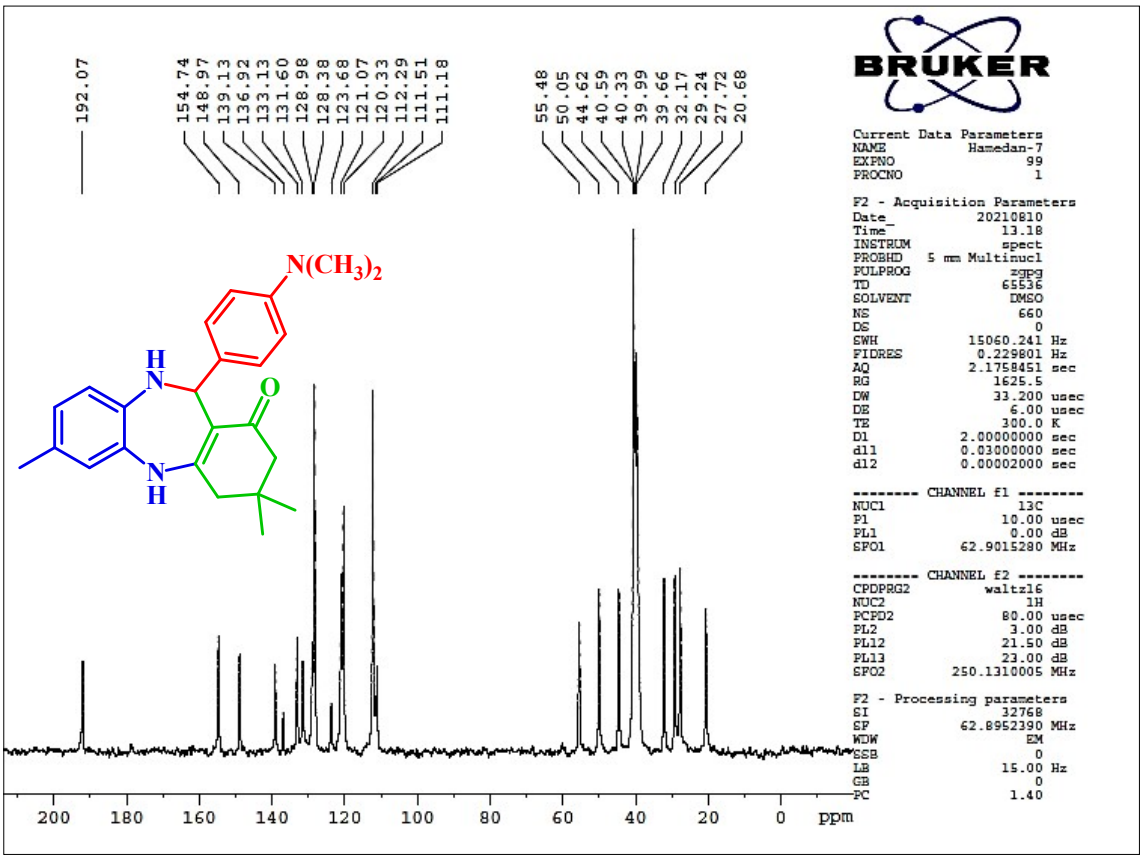
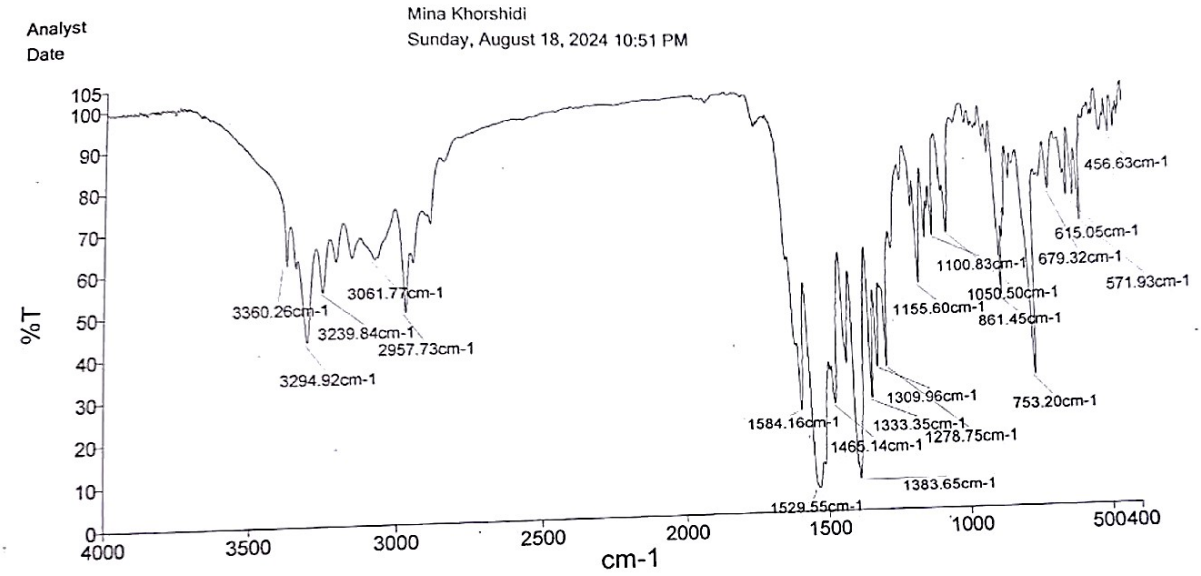


^{1}H NMR (250 MHz, DMSO) δ 0.99 (s, 3H, CH_3), 1.05 (s, 3H, CH_3), 2.04 (s, 3H, CH_3), 2.48 (s, 2H, -CH_2), 2.54 (s, 2H, -CH_2-C=O), 5.64 (s, 1H, CH), 6.08-7.08 (m, 7H Ar), 7.14 (s, NH), 8.75 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 20.6, 28.4, 32.2, 44.5, 49.9, 55.7, 109.8, 120.8, 124.0, 128.1, 129.0, 129.5, 130.8, 132.1, 136.3, 138.5, 144.3, 155.3, 192.3.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Mina Khorshidi
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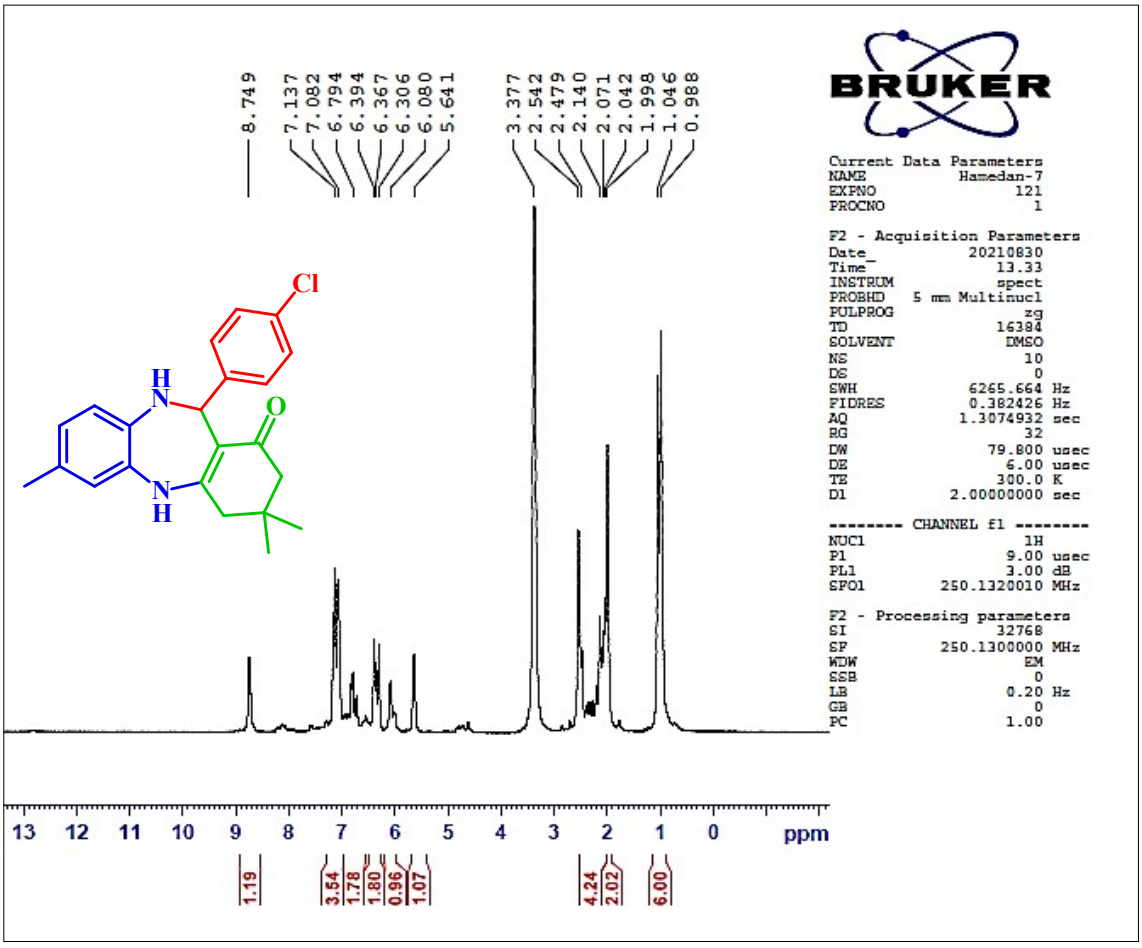
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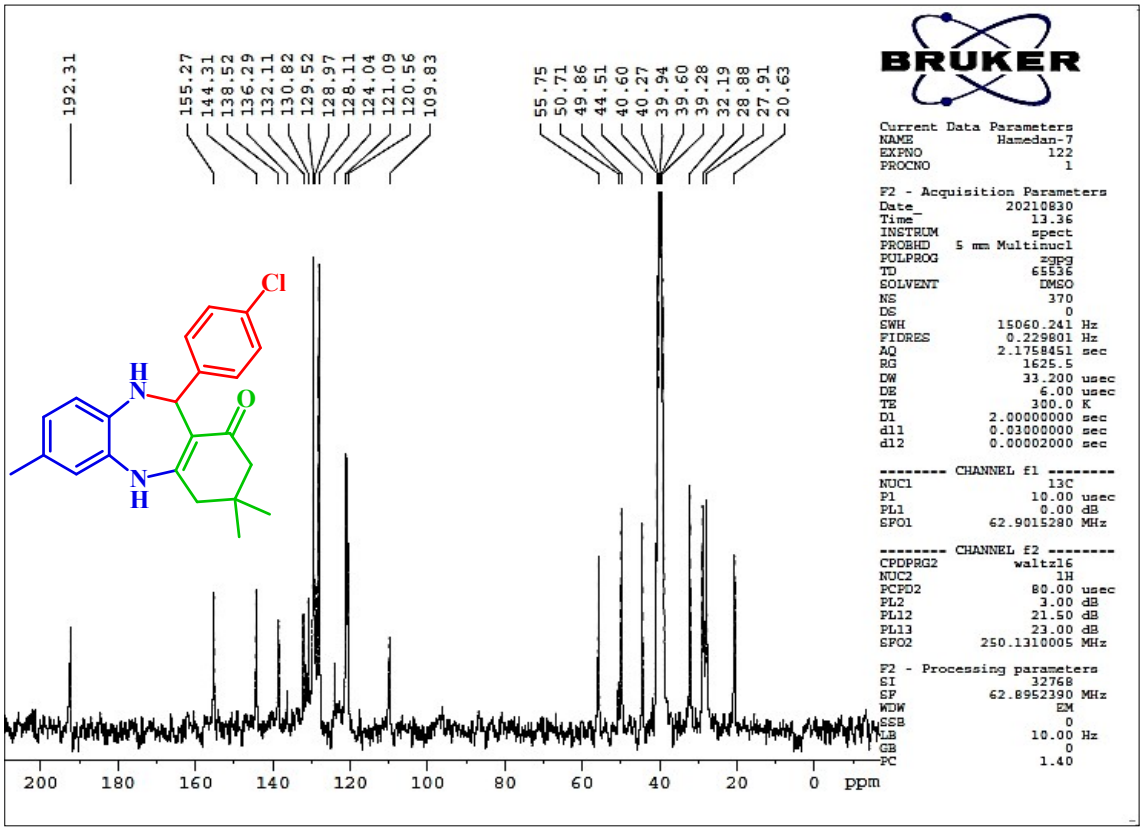
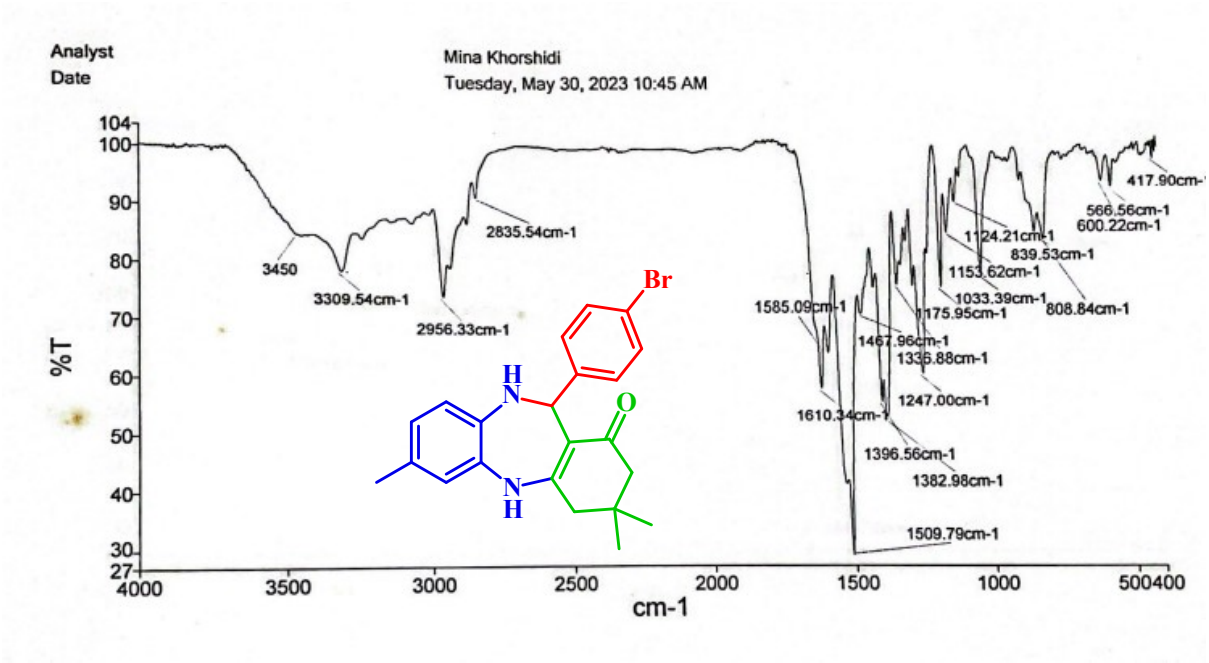


^{1}H NMR (250 MHz, DMSO) δ 0.99 (s, 3H, CH_3), 1.05 (s, 3H, CH_3), 2.04 (s, 3H, CH_3), 2.18 (s, 2H, -CH_2), 2.52 (s, 2H, -CH_2-C=O), 5.65 (s, 1H, C-H), 6.10-6.74 (m, 3H Ar), 6.82 (s, NH), 7.01-7.30 (m, 4H Ar), 8.77 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 20.7, 28.4, 32.2, 44.5, 49.9, 55.8, 109.8, 119.4, 120.8, 124.1, 129.0, 130.0, 131.0, 132.1, 136.3, 138.5, 144.8, 155.3, 192.3.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Mina Khorshidi
Tuesday, May 30, 2023 10:45 AM
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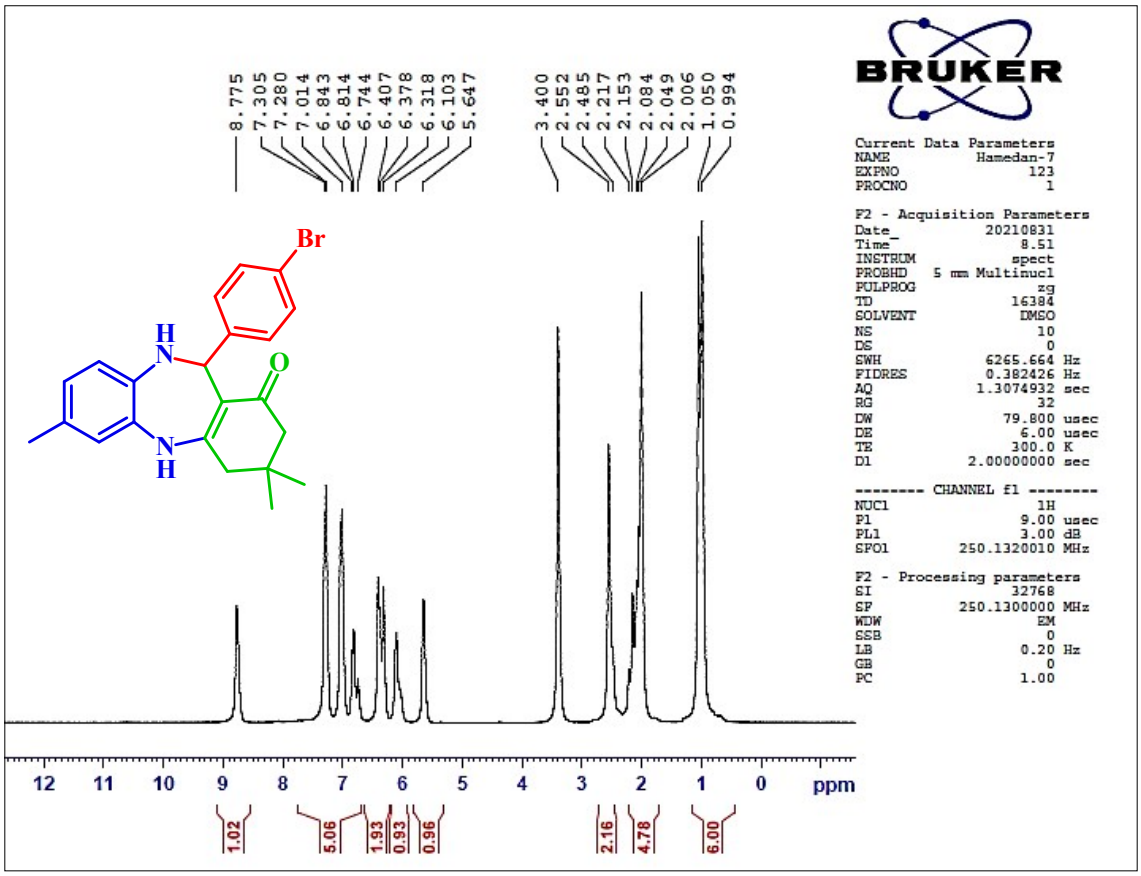
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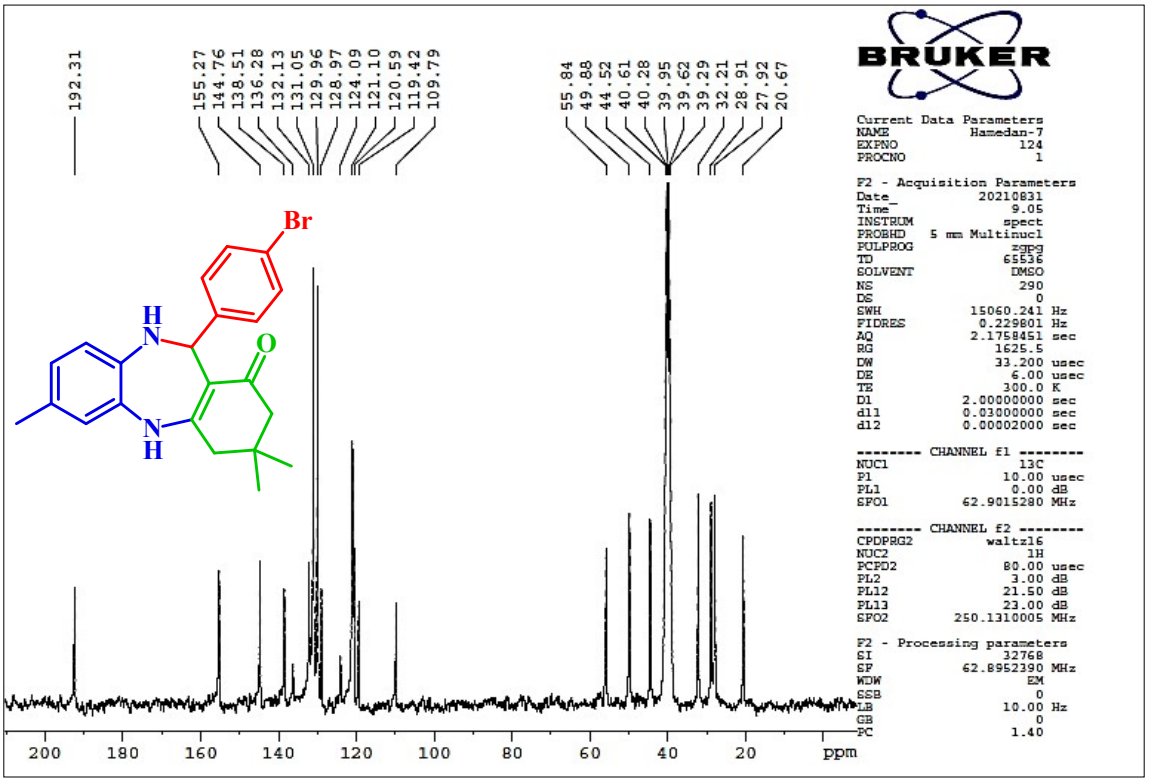
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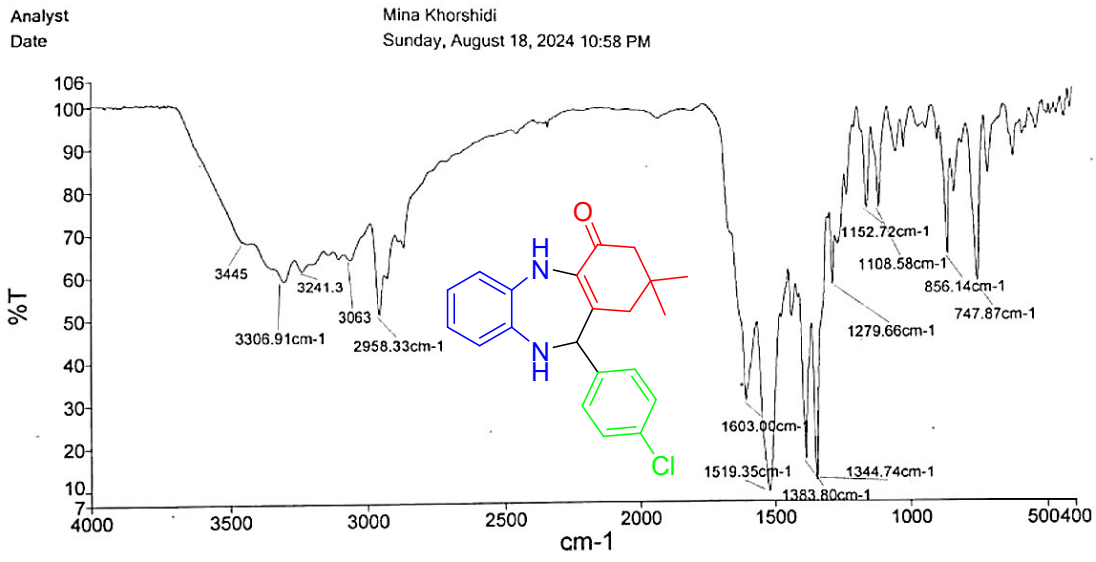
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Mina Khorshidi
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Sunday, August 18, 2024 10:58 PM
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2958.33cm-1
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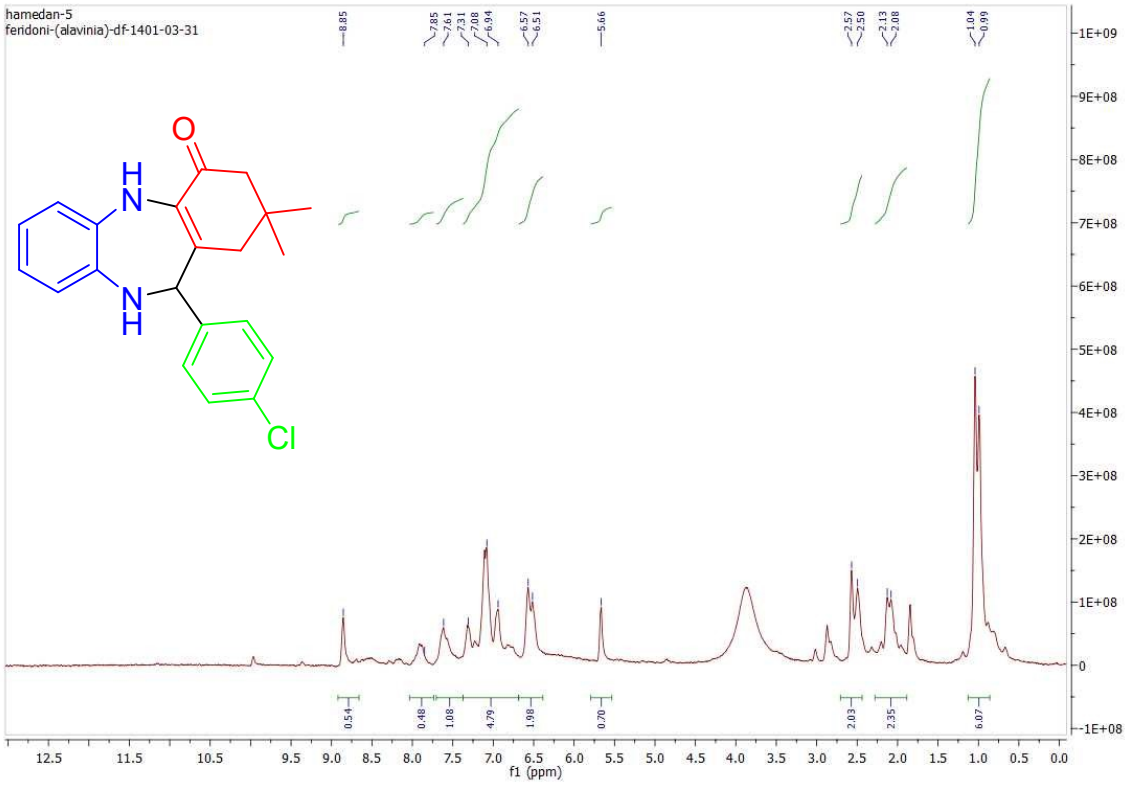
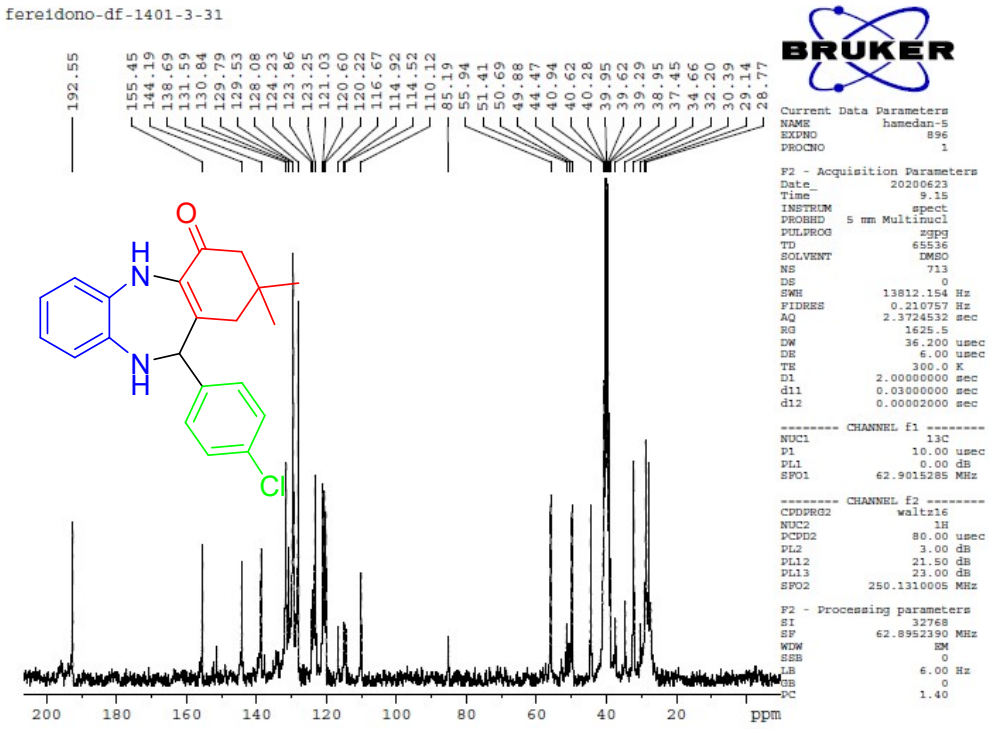
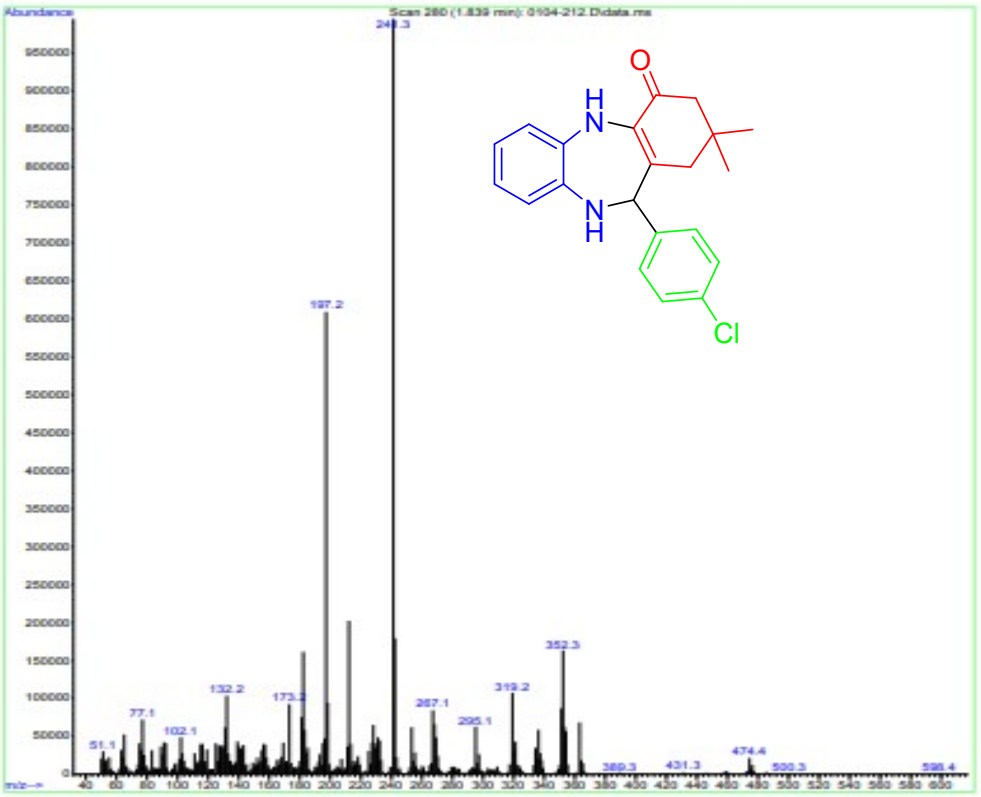
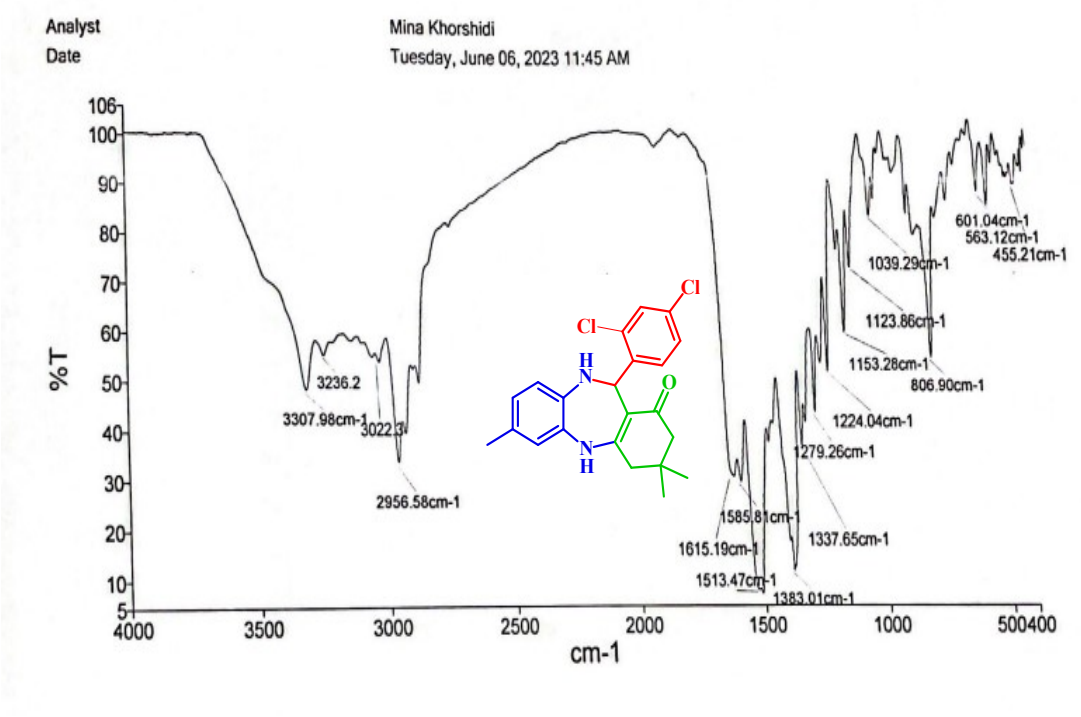
H
Cl

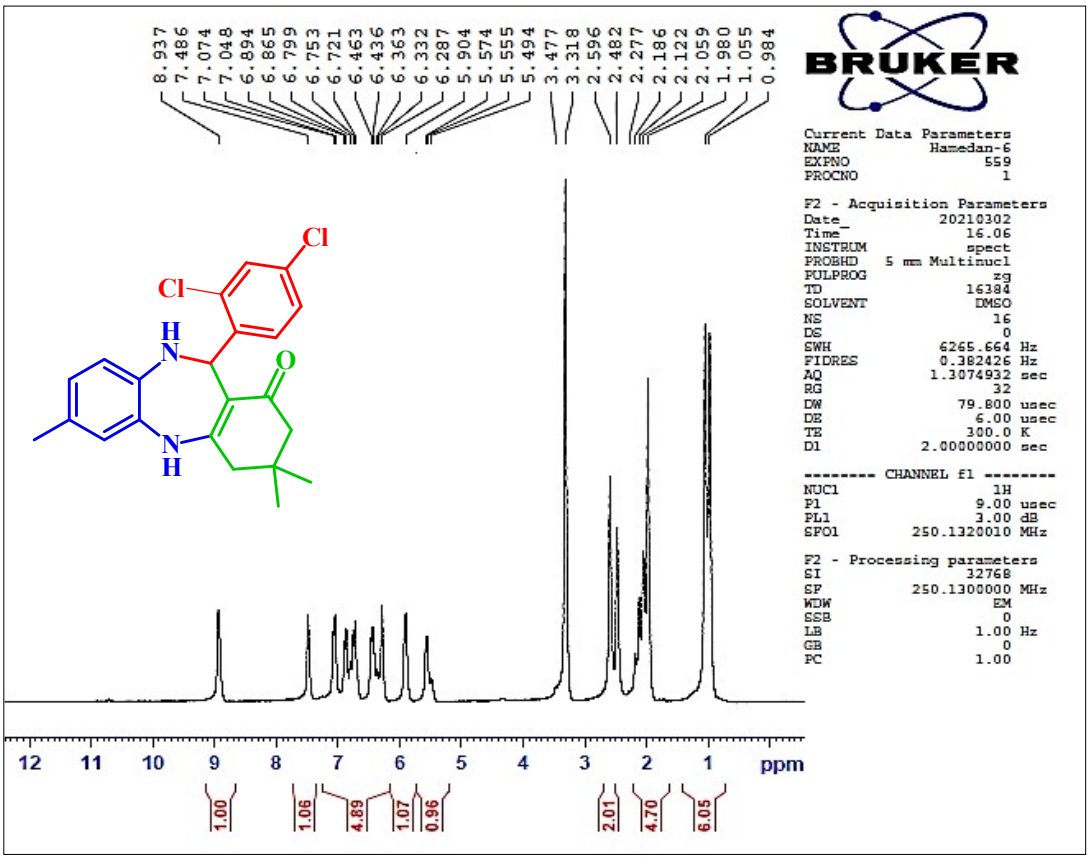

hamedan-5
feridoni-(alavinia)-df-1401-03-31
f1 (ppm)

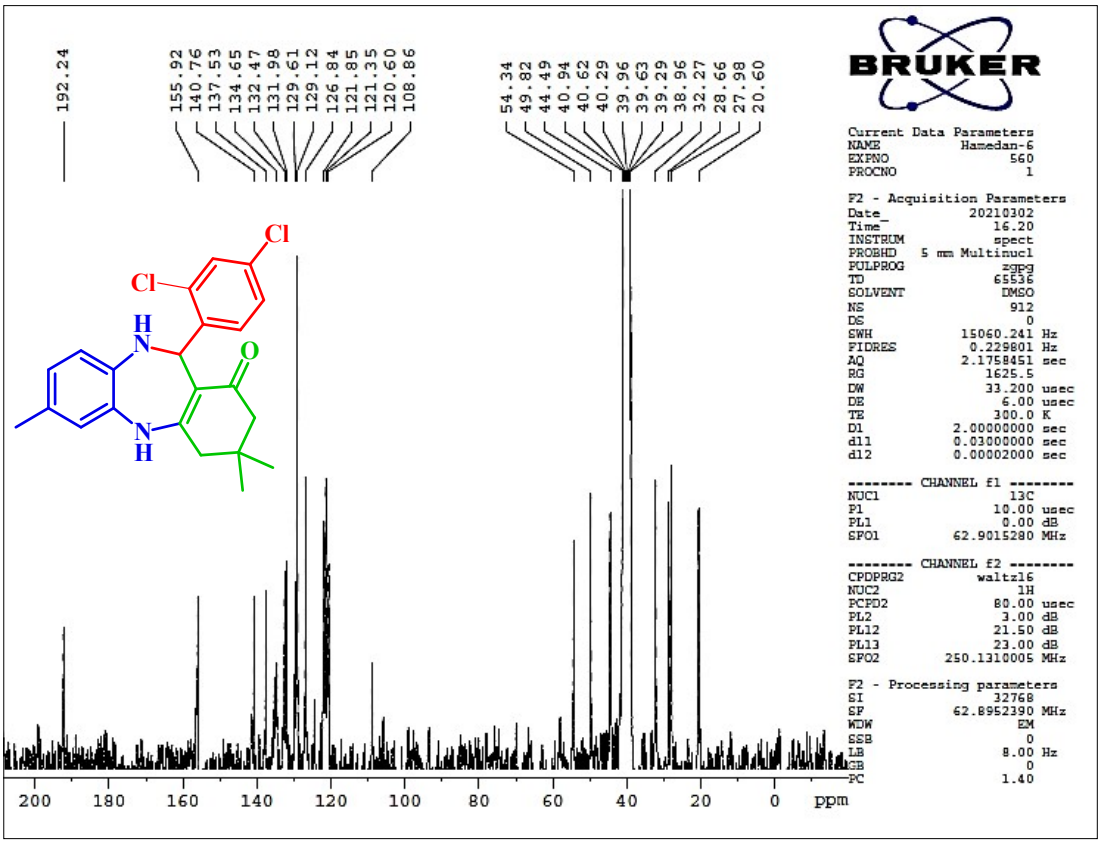

fereidono-df-1401-3-31
ppm


Abundance
Scan 280 (1.839 min): 0104-212.D\data.ms
248.3
197.2
132.2
173.2
77.1
102.1
51.1
287.1
295.1
319.2
352.3
389.3
431.3
474.4
590.3
598.4
m/z-->
O
H
N
N
H
Cl

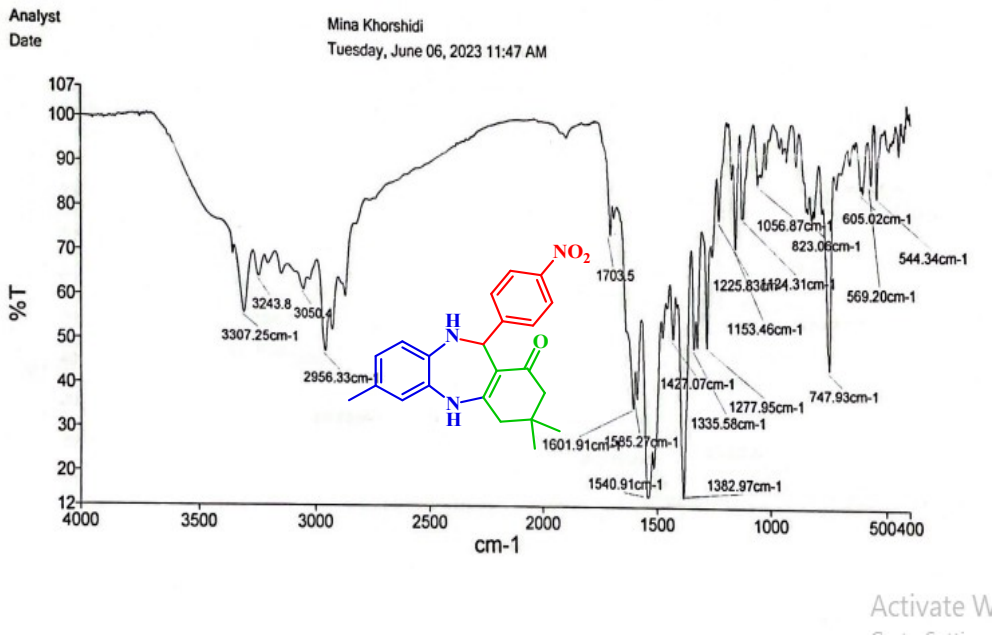
^{1}H NMR (250 MHz, DMSO) δ 0/98 (s, 3H, CH_3), 1.05 (s, 3H, CH_3), 1.98 (s, 3H, -CH_3), 2.09 (s, 2H, -CH_2), 2.60 (s, 2H, -CH_2-C=O), 5.50 (s, 1H, C-H), 5.90-6.46 (m, 4H Ar), 6.72-7.07 (m, 3H Ar), 7.49 (s, NH), 8.94 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 20.6, 28.3, 32.3, 44.5, 49.8, 54.3, 108.9, 120.6, 121.3, 121.8, 126.8, 129.1, 129.6, 130.1, 132.0, 132.5, 134.6, 137.5, 140.8, 155.9, 192.2.

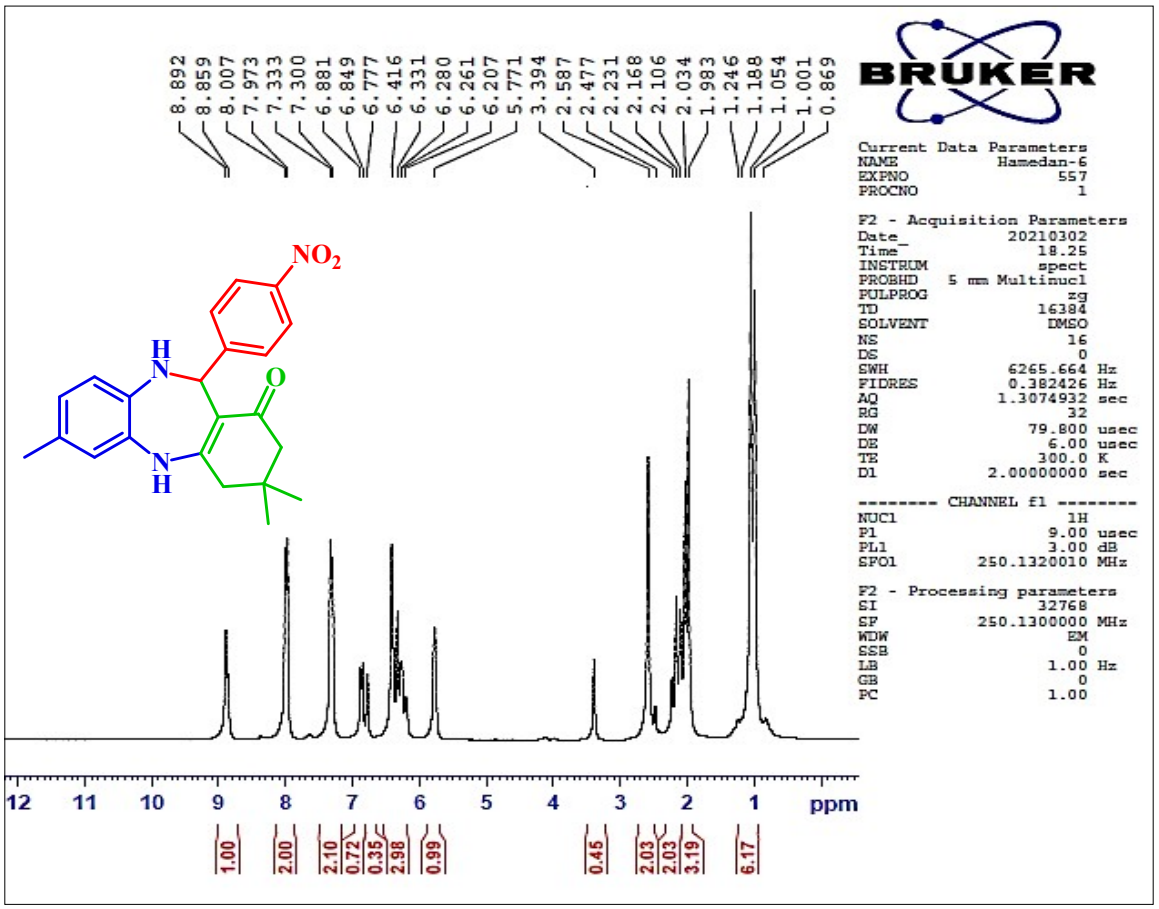


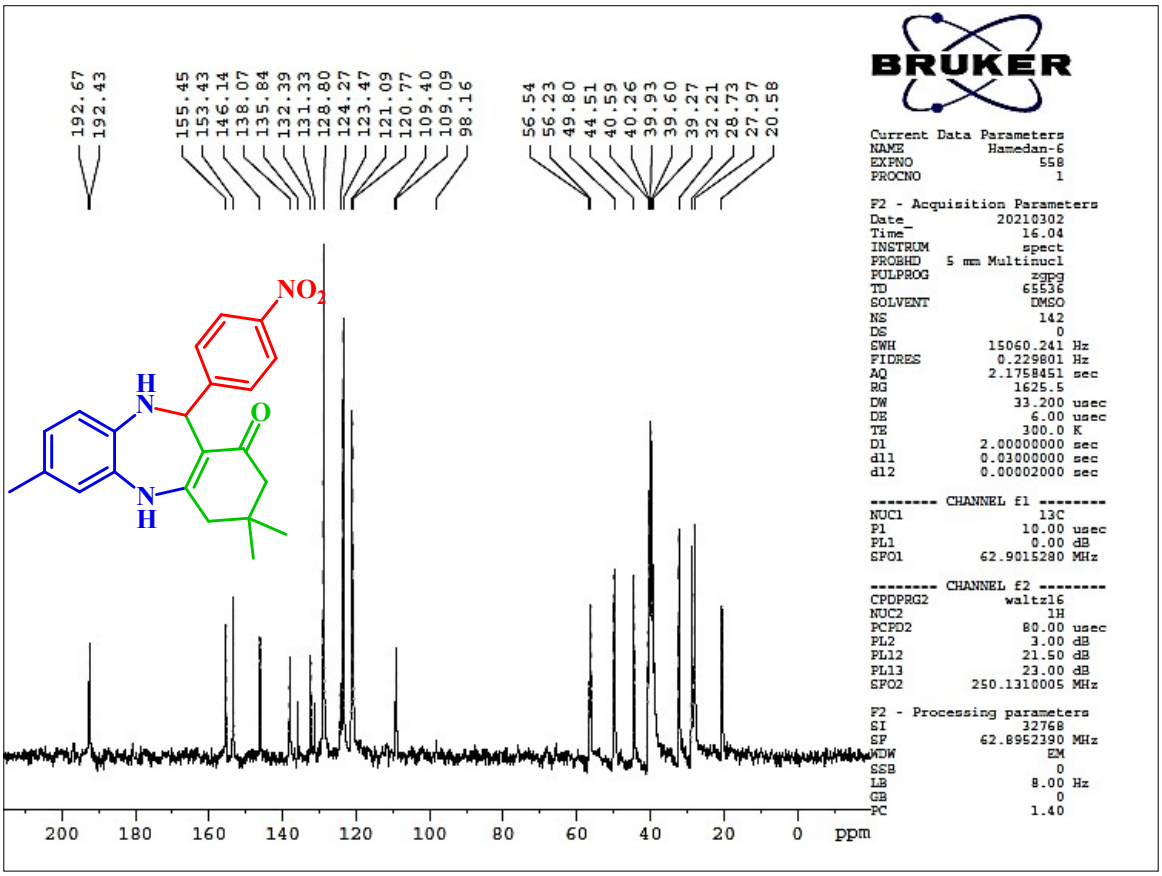

8.937
7.486
7.074
7.048
6.894
6.865
6.799
6.753
6.721
6.463
6.436
6.363
6.332
6.287
5.904
5.574
5.555
5.494
3.477
3.318
2.596
2.482
2.277
2.186
2.122
2.059
1.980
1.055
0.984
Cl
Cl
H
N
O
N
H
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.06
4.89
1.07
0.96
2.01
4.70
6.05
BRUKER
Current Data Parameters
NAME Hamedan-6
EXPNO 559
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210302
Time 16.06
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT DMSO
NS 16
DS 0
SWH 6265.664 Hz
FIDRES 0.382426 Hz
AQ 1.3074932 sec
RG 32
DW 79.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
-------- CHANNEL f1 --------
NUC1 1H
P1 9.00 usec
PL1 3.00 dB
SFO1 250.1320010 MHz
F2 - Processing parameters
SI 32768
SF 250.1300000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

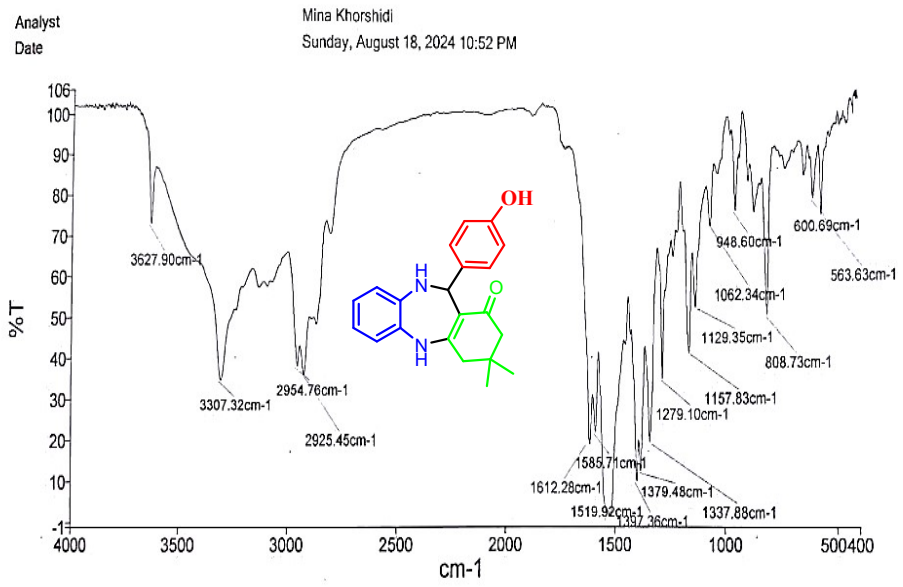

192.24
155.92
140.76
137.53
134.65
132.47
131.98
129.61
129.12
126.84
121.85
121.35
120.60
108.86
54.34
49.82
44.49
40.94
40.62
40.29
39.96
39.63
39.29
38.96
32.27
28.66
27.98
20.60
Cl
Cl
H
N
O
N
H
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME Hamedan-6
EXPNO 560
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210302
Time 16.20
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 912
DS 0
SWH 15060.241 Hz
FIDRES 0.229801 Hz
AQ 2.1758451 sec
RG 1625.5
DW 33.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
-------- CHANNEL f1 --------
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 62.9015280 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 21.50 dB
PL13 23.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952390 MHz
WDW EM
SSB 0
LB 8.00 Hz
GB 0
PC 1.40

^{1}H NMR (250 MHz, DMSO) δ 1.00 (s, 3H, CH_3), 1.05 (s, 3H, CH_3), 1.98 (s, 3H, -CH_3), 2.21 (q, J = 15.5 Hz, 2H, -CH_2), 2.59 (s, 2H, -CH_2-C=O), 5.77 (s, 1H, C-H), 6.21-6.42 (m, 3H Ar), 6.86 (s, NH), 7.32 (d, J = 8.2 Hz, 2H), 7.99 (d, J = 8.2 Hz, 2H), 8.87 (s, NH). ^{13}C NMR (63 MHz, DMSO) δ 20.6, 28.3, 32.2, 44.5, 49.8, 56.4, 98.2, 109.2, 120.9, 123.9, 128.8, 131.3, 132.4, 135.8, 138.1, 146.1, 153.4, 155.4, 192.5.




8.892
8.859
8.007
7.973
7.333
7.300
6.881
6.849
6.777
6.416
6.331
6.280
6.261
6.207
5.771
3.394
2.587
2.477
2.231
2.168
2.106
2.034
1.983
1.246
1.188
1.054
1.001
0.869
NO2
H
N
O
N
H
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.00
2.10
0.72
0.35
2.98
0.99
0.45
2.03
2.03
3.19
6.17
BRUKER
Current Data Parameters
NAME Hamedan-6
EXPNO 557
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210302
Time 18.25
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT DMSO
NS 16
DS 0
SWH 6265.664 Hz
FIDRES 0.382426 Hz
AQ 1.3074932 sec
RG 32
DW 79.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
-------- CHANNEL f1 --------
NUC1 1H
P1 9.00 usec
PL1 3.00 dB
SFO1 250.1320010 MHz
F2 - Processing parameters
SI 32768
SF 250.1300000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00


192.67
192.43
155.45
153.43
146.14
138.07
135.84
132.39
131.33
128.80
124.27
123.47
121.09
120.77
109.40
109.09
98.16
56.54
56.23
49.80
44.51
40.59
40.26
39.93
39.60
39.27
32.21
28.73
27.97
20.58
NO2
H
N
O
N
H
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME Hamedan-6
EXPNO 558
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210302
Time 16.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 142
DS 0
SWH 15060.241 Hz
FIDRES 0.229801 Hz
AQ 2.1758451 sec
RG 1625.5
DW 33.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
-------- CHANNEL f1 --------
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 62.9015280 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 21.50 dB
PL13 23.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952390 MHz
WDW EM
SSB 0
LB 8.00 Hz
GB 0
PC 1.40


Analyst
Mina Khorshidi
Date
Sunday, August 18, 2024 10:52 PM
%T
cm-1
OH
H
N
H
N
O
3627.90cm-1
3307.32cm-1
2954.76cm-1
2925.45cm-1
1612.28cm-1
1585.71cm-1
1519.92cm-1
1397.36cm-1
1379.48cm-1
1337.88cm-1
1279.10cm-1
1157.83cm-1
1129.35cm-1
1062.34cm-1
948.60cm-1
808.73cm-1
600.69cm-1
563.63cm-1