

Graphene oxide functionalized tannic acid- trimethylolpropane triglycidyl ether nanoparticles: a versatile network nanocatalyst for synthesis of 4-substituted-1,5-benzodiazepine derivatives

Alireza Gharehkhani, Maryam Hajjami*, Reza Hazbavi

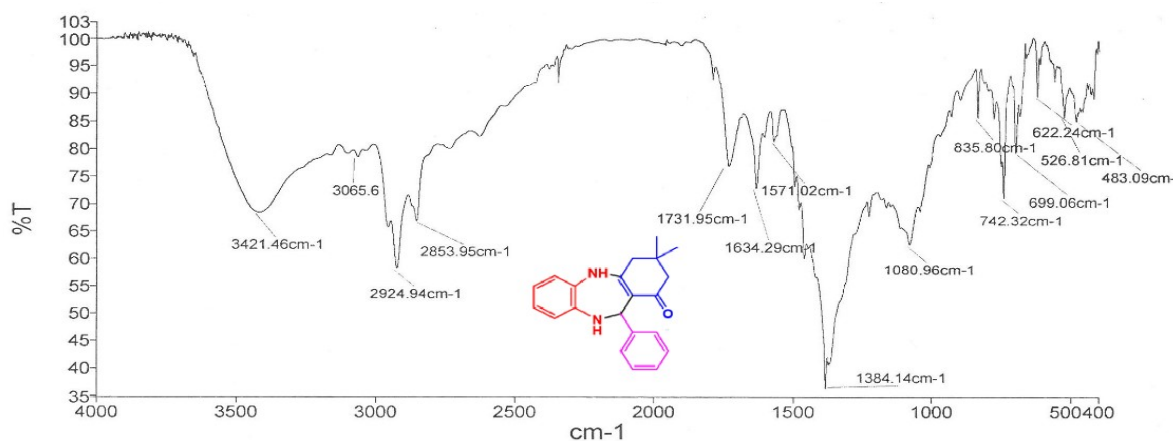
Department of Organic Chemistry, Faculty of Chemistry and Petroleum Sciences, Bu-Ali Sina University, Postal code 6517838683,

11-(4-chlorophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one:

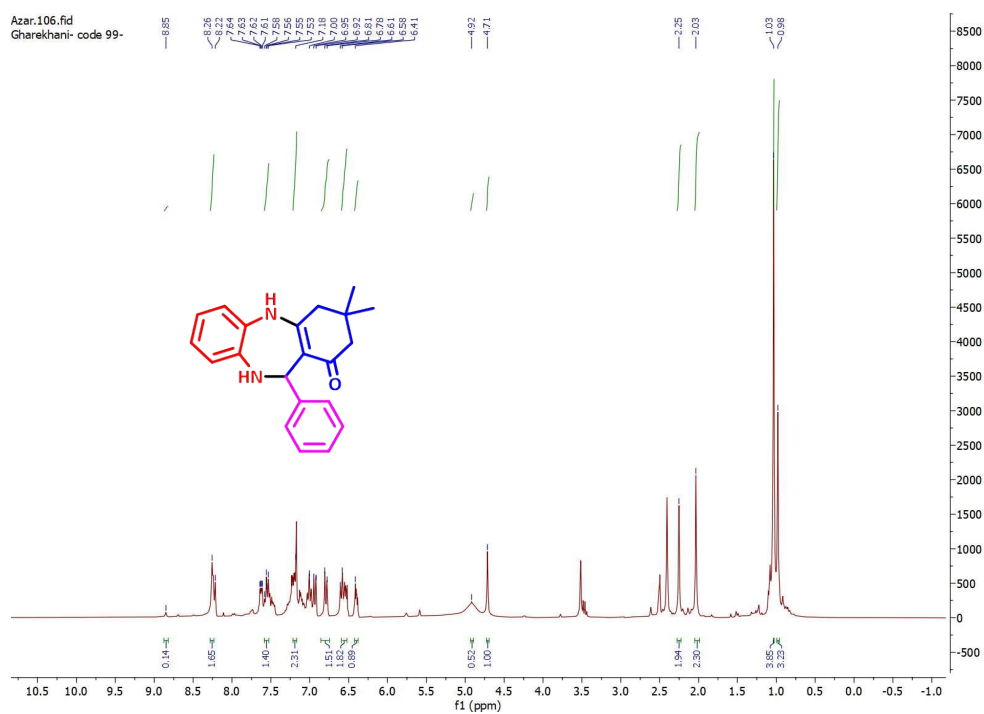
FT-IR (KBr, cm⁻¹): 471, 748, 1044, 1093, 1151, 1335, 1384, 1531, 1603, 1629, 2872, 2957, 3055, 3414.

¹H NMR (400 MHz, DMSO-d₆): δ ¹ 12.73 (s, 1H), 7.90 (d, *J* = 7.3 Hz, 2H), 7.63 (s, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.22 (s, 3H), 6.65 (s, 1H), 5.42 (s, 1H), 3.53 (s, 2H), 2.62 (s, 2H), 1.48 (s, 3H), 1.22 (s, 3H).

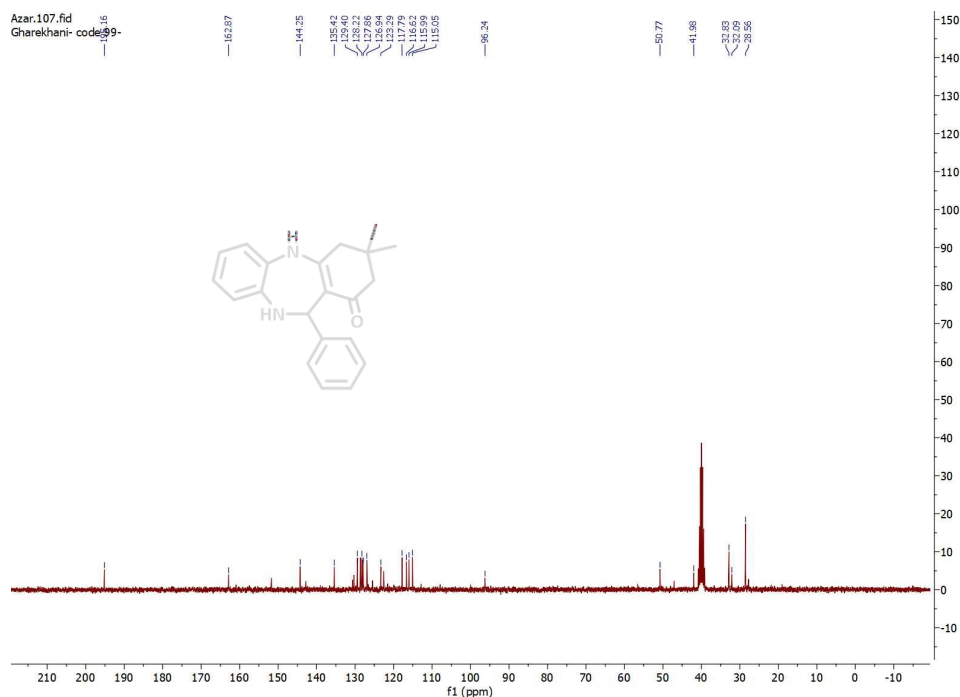
^{13}C NMR (75 MHz, DMSO) δ 195.1, 162.8, 144.2, 135.4, 129.4, 128.2, 127.8, 126.9, 123.2, 117.7, 116.6, 115.9, 115.0, 96.2, 50.7, 41.9, 32.8, 32.0, 28.5.



FT-IR spectrum of the compound 3,3-dimethyl-11-phenyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



^1H NMR spectrum of the compound 3,3-dimethyl-11-phenyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



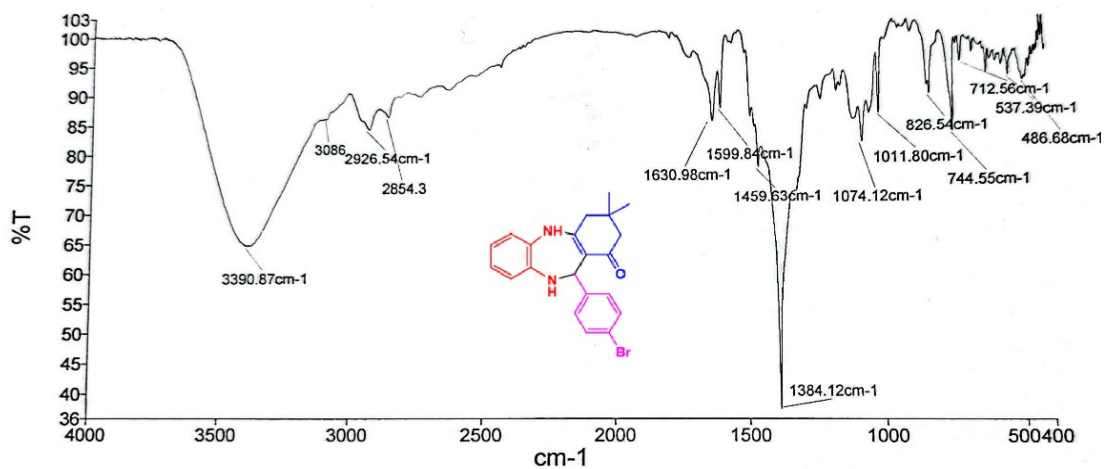
^{13}C NMR spectrum of the compound 3,3-dimethyl-11-phenyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one

11-(4-bromophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one:

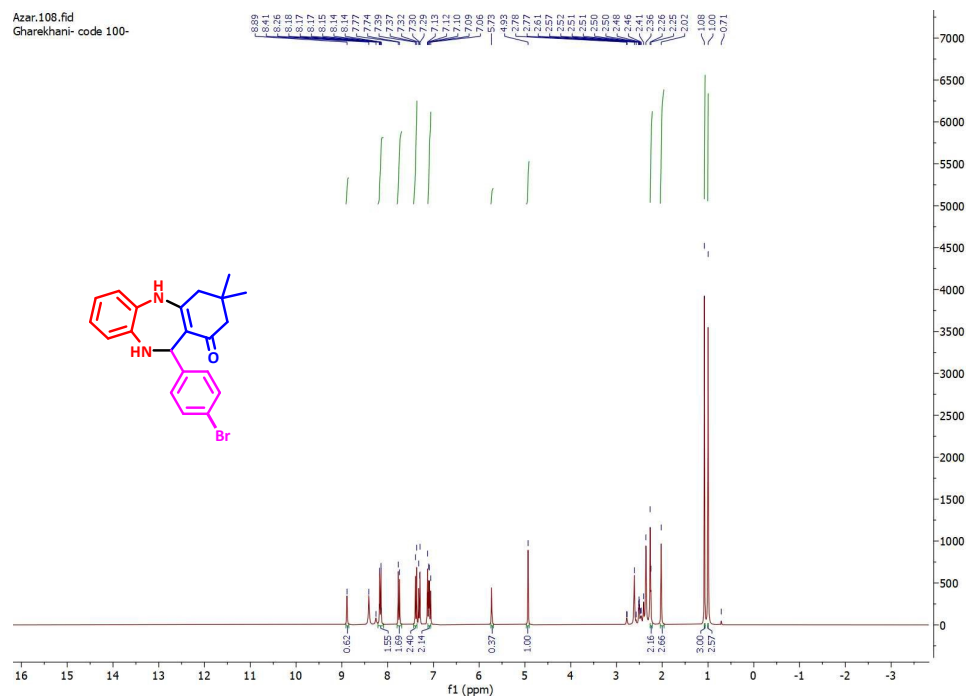
FT-IR (KBr, cm^{-1}): 486, 537, 712, 744, 826, 1011, 1074, 1384, 1459, 1599, 1630, 2854, 2926, 3086, 3390.

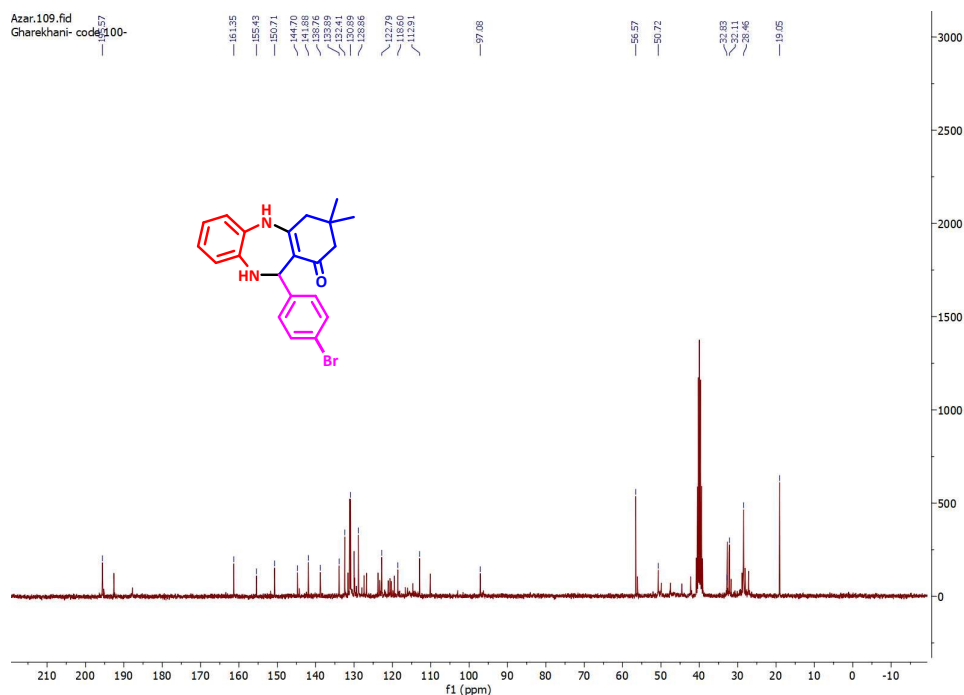
^1H NMR (300 MHz, DMSO) δ 8.89 (s, 1H), 8.21 – 7.06 (m, 8H), 4.93 (s, 1H), 2.25 (s, 2H), 2.02 (s, 3H), 1.08 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (75 MHz, DMSO) δ 195.1, 162.8, 144.2, 135.4, 129.4, 128.2, 127.8, 126.9, 123.2, 117.7, 116.6, 115.9, 115.0, 96.2, 50.7, 41.9, 32.8, 32.0, 28.5.



FT-IR spectrum of the compound 11-(4-bromophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one





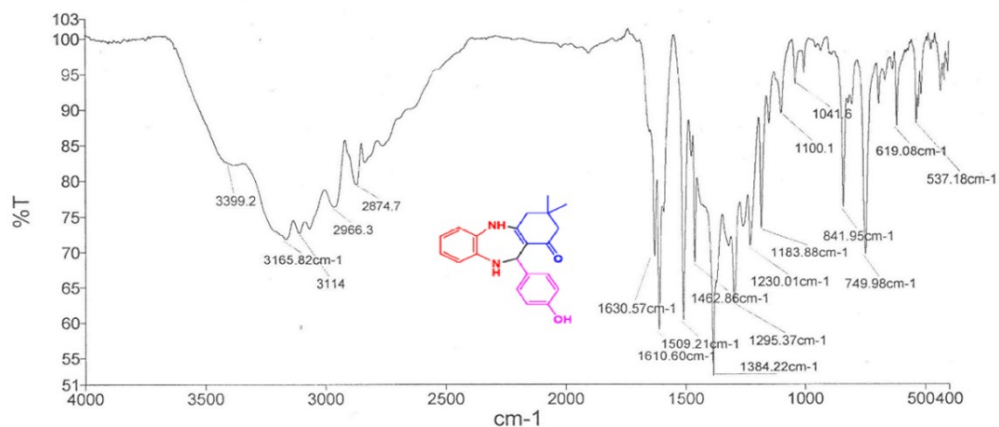
^{13}C NMR spectrum of the compound 11-(4-bromophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one

11-(4-hydroxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one:

FT-IR (KBr, cm^{-1}): 537, 619, 749, 841, 1041, 1100, 1183, 1230, 1295, 1384, 1462, 1509, 1610, 1630, 2874, 2966, 3114, 3165, 3399.

^1H NMR (300 MHz, DMSO) δ 9.07 (s, 1H), 8.74 (s, 1H), 7.56-6.53 (m, 8H), 4.88 (s, 1H), 2.35 (s, 2H), 2.02 (s, 2H), 1.03 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (75 MHz, DMSO) δ 195.1, 161.2, 159.6, 152.2, 144.2, 133.9, 129.4, 128.6, 127.8, 122.0, 121.5, 115.9, 114.8, 97.0, 56.5, 50.7, 32.8, 28.4, 19.0.



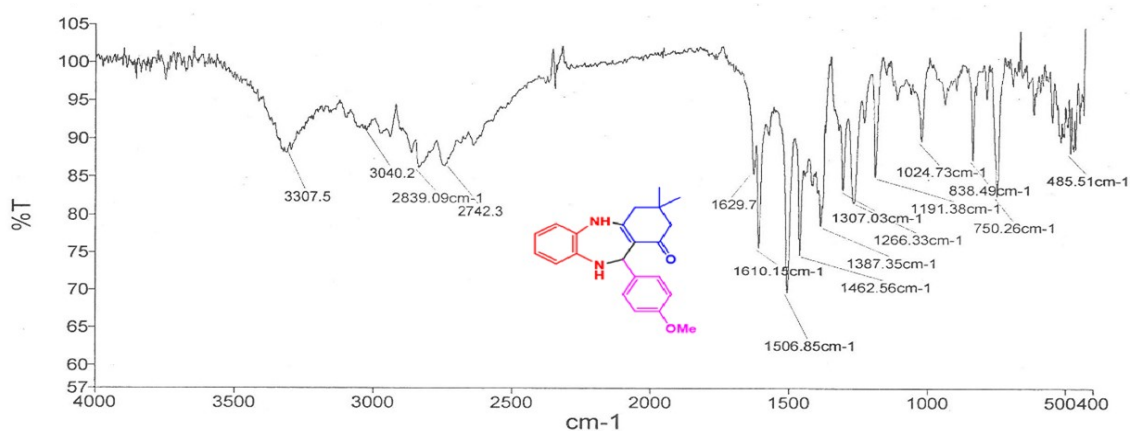
FT-IR spectrum of the compound 11-(4-hydroxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one

11-(4-methoxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one:

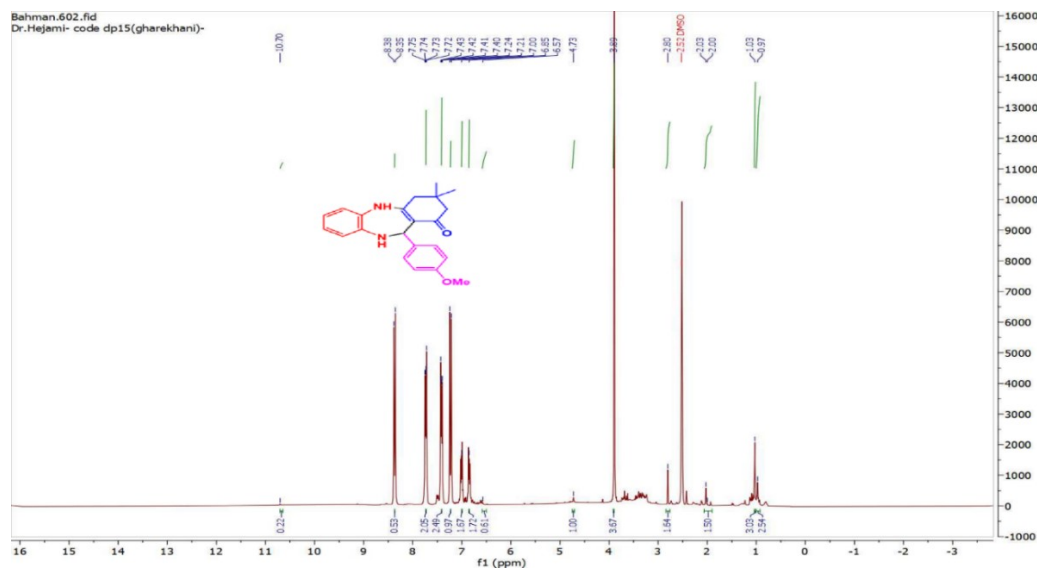
FT-IR (KBr, cm^{-1}): 485, 750, 838, 1024, 1191, 1266, 1307, 1387, 1462, 1506, 1610, 1629, 2742, 2839, 3040, 3307.

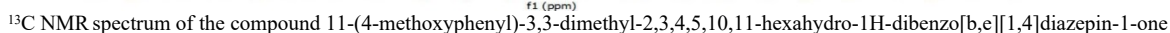
^1H NMR (400 MHz, DMSO-d_6): δ 10.70 (s, 1H), 7.73 - 6.85 (m, 8H), 6.57 (s, 1H), 4.73 (s, 1H), 3.89 (s, 4H), 2.80 (s, 2H), 2.03 (s, 2H), 1.03 (s, 3H), 0.97 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 162.7, 149.9, 134.7, 129.9, 127.8, 125.6, 122.9, 120.6, 118.1, 114.5, 114.0, 63.5, 56.1, 40.6, 32.8, 28.5, 12.8.



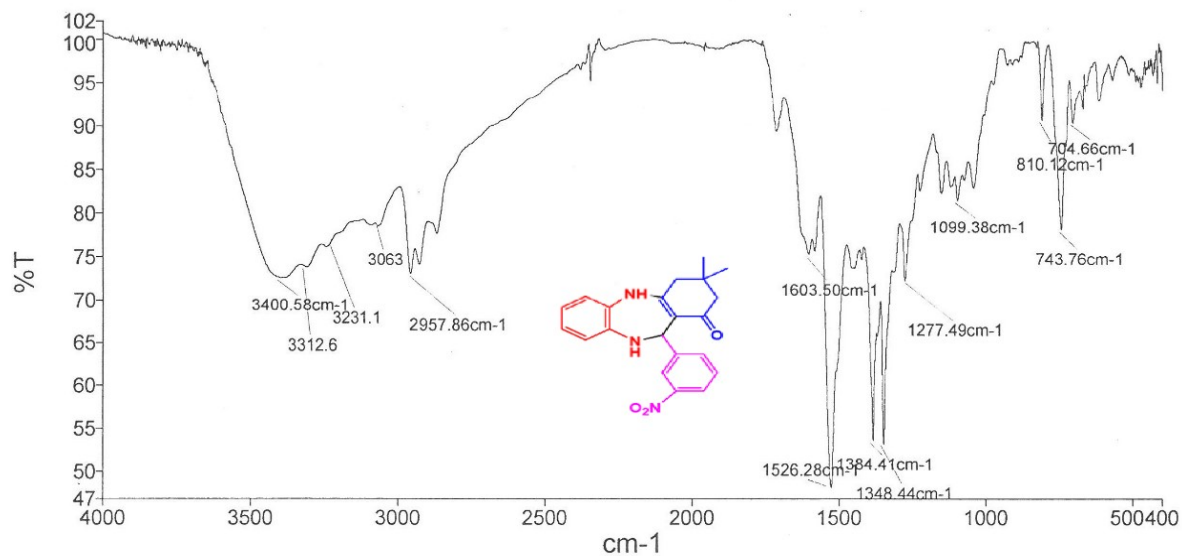
FT-IR spectrum of the compound 11-(4-methoxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



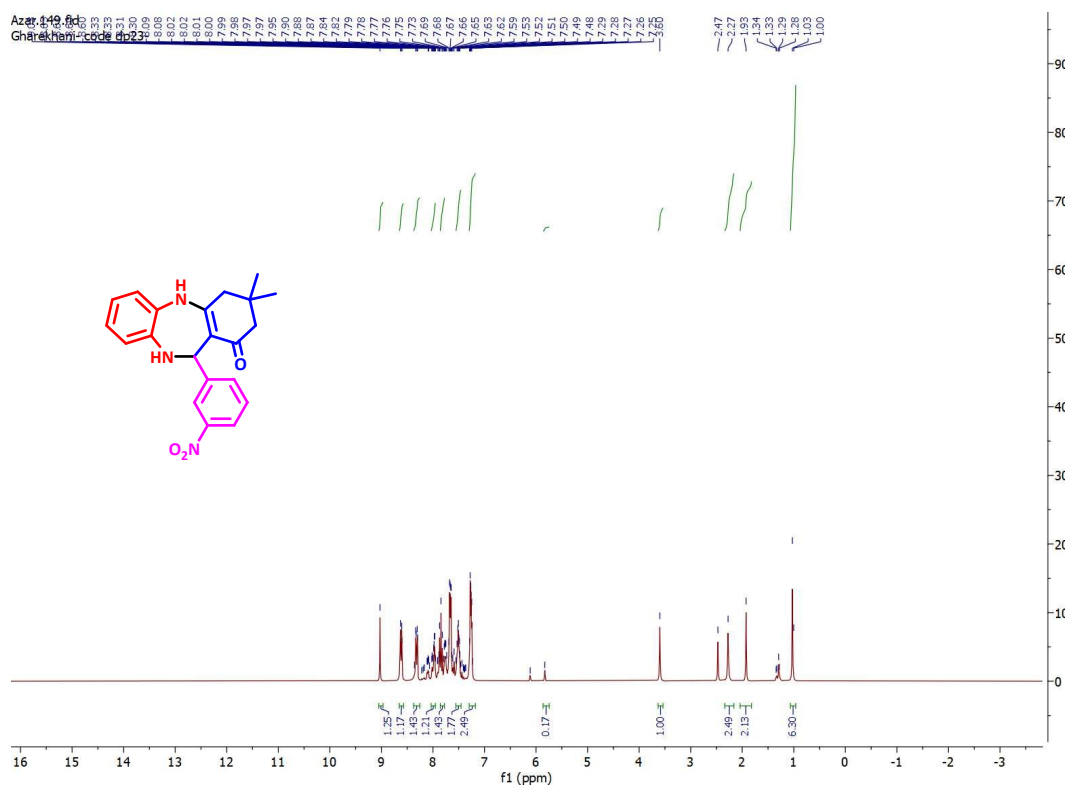


FT-IR (KBr, cm⁻¹):704, 743, 810, 1099, 1277, 1248, 1384, 1426, 1603, 2957, 3063, 3231, 3312 3400.

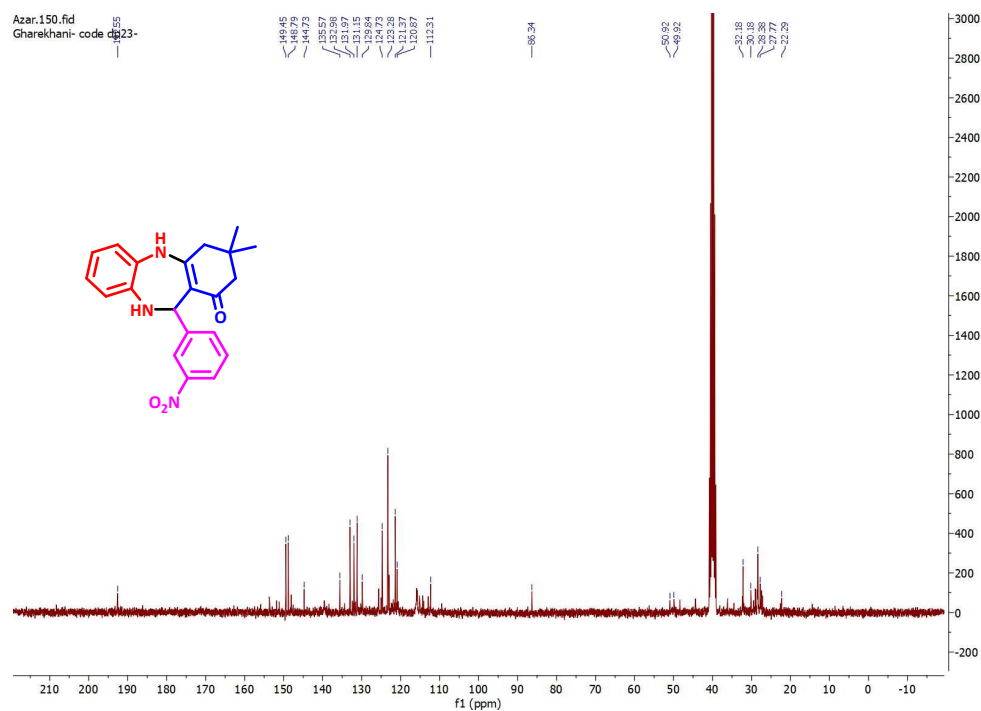
¹³C NMR (75 MHz, DMSO) δ 192.5, 149.4, 148.7, 135.5, 132.9, 131.9, 131.1, 129.8, 124.7, 123.7, 123.2, 121.3, 120.8, 112.3, 86.3, 50.9, 32.1, 28.3, 27.7, 22.2.



FT-IR spectrum of the compound 3,3-dimethyl-11-(3-nitrophenyl)-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



^1H NMR spectrum of the compound 3,3-dimethyl-11-(3-nitrophenyl)-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



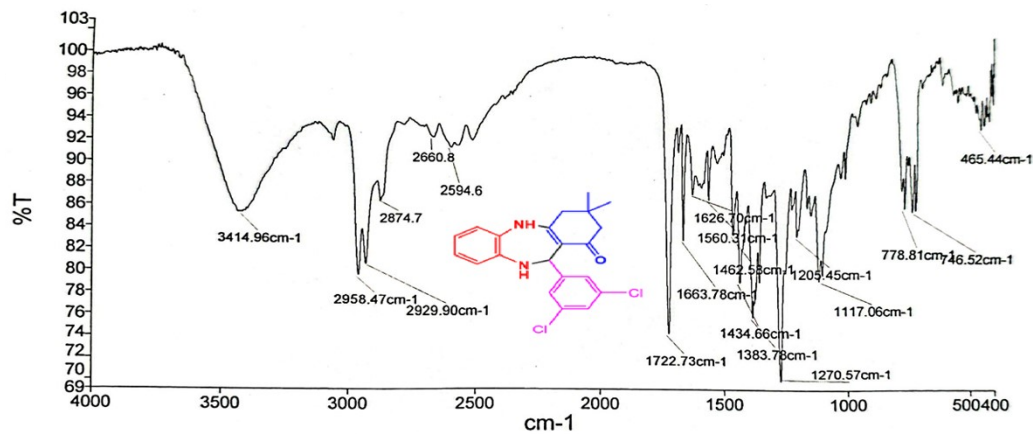
^{13}C NMR spectrum of the compound 3,3-dimethyl-11-(3-nitrophenyl)-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one

11-(3,5-dichlorophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one:

FT-IR (KBr, cm^{-1}): 465, 778, 1117, 1205, 1270, 1383, 1434, 1462, 1560, 1626, 1663, 1722, 2594, 2660, 2874, 2929, 2958, 3414.

^1H NMR (500 MHz, DMSO) δ 9.01 (s, 1H), 7.63- 6.37 (m, 7H), 4.68 (s, 1H), 2.39 (s, 3H), 2.00 (s, 2H), 1.01 (s, 3H), 0.96 (s, 3H).

^{13}C NMR (126 MHz, DMSO) δ 194.6, 146.6, 143.7, 134.8, 132.3, 130.5, 128.2, 127.8, 127.4, 127.3, 122.8, 121.0, 117.3, 116.1, 115.5, 114.6, 50.2, 32.3, 31.4, 31.0, 27.9.



133.6, 133.1, 132.9, 132.8, 132.7, 132.6, 132.5, 132.4, 132.3, 132.2, 132.1, 132.0, 131.9, 131.8, 131.7, 131.6, 131.5, 131.4, 131.3, 131.2, 131.1, 131.0, 130.9, 130.8, 130.7, 130.6, 130.5, 130.4, 130.3, 130.2, 130.1, 130.0, 129.9, 129.8, 129.7, 129.6, 129.5, 129.4, 129.3, 129.2, 129.1, 129.0, 128.9, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4, 127.3, 127.2, 127.1, 127.0, 126.9, 126.8, 126.7, 126.6, 126.5, 126.4, 126.3, 126.2, 126.1, 126.0, 125.9, 125.8, 125.7, 125.6, 125.5, 125.4, 125.3, 125.2, 125.1, 125.0, 124.9, 124.8, 124.7, 124.6, 124.5, 124.4, 124.3, 124.2, 124.1, 124.0, 123.9, 123.8, 123.7, 123.6, 123.5, 123.4, 123.3, 123.2, 123.1, 123.0, 122.9, 122.8, 122.7, 122.6, 122.5, 122.4, 122.3, 122.2, 122.1, 122.0, 121.9, 121.8, 121.7, 121.6, 121.5, 121.4, 121.3, 121.2, 121.1, 121.0, 120.9, 120.8, 120.7, 120.6, 120.5, 120.4, 120.3, 120.2, 120.1, 120.0, 119.9, 119.8, 119.7, 119.6, 119.5, 119.4, 119.3, 119.2, 119.1, 119.0, 118.9, 118.8, 118.7, 118.6, 118.5, 118.4, 118.3, 118.2, 118.1, 118.0, 117.9, 117.8, 117.7, 117.6, 117.5, 117.4, 117.3, 117.2, 117.1, 117.0, 116.9, 116.8, 116.7, 116.6, 116.5, 116.4, 116.3, 116.2, 116.1, 116.0, 115.9, 115.8, 115.7, 115.6, 115.5, 115.4, 115.3, 115.2, 115.1, 115.0, 114.9, 114.8, 114.7, 114.6, 114.5, 114.4, 114.3, 114.2, 114.1, 114.0, 113.9, 113.8, 113.7, 113.6, 113.5, 113.4, 113.3, 113.2, 113.1, 113.0, 112.9, 112.8, 112.7, 112.6, 112.5, 112.4, 112.3, 112.2, 112.1, 112.0, 111.9, 111.8, 111.7, 111.6, 111.5, 111.4, 111.3, 111.2, 111.1, 111.0, 110.9, 110.8, 110.7, 110.6, 110.5, 110.4, 110.3, 110.2, 110.1, 110.0, 109.9, 109.8, 109.7, 109.6, 109.5, 109.4, 109.3, 109.2, 109.1, 109.0, 108.9, 108.8, 108.7, 108.6, 108.5, 108.4, 108.3, 108.2, 108.1, 108.0, 107.9, 107.8, 107.7, 107.6, 107.5, 107.4, 107.3, 107.2, 107.1, 107.0, 106.9, 106.8, 106.7, 106.6, 106.5, 106.4, 106.3, 106.2, 106.1, 106.0, 105.9, 105.8, 105.7, 105.6, 105.5, 105.4, 105.3, 105.2, 105.1, 105.0, 104.9, 104.8, 104.7, 104.6, 104.5, 104.4, 104.3, 104.2, 104.1, 104.0, 103.9, 103.8, 103.7, 103.6, 103.5, 103.4, 103.3, 103.2, 103.1, 103.0, 102.9, 102.8, 102.7, 102.6, 102.5, 102.4, 102.3, 102.2, 102.1, 102.0, 101.9, 101.8, 101.7, 101.6, 101.5, 101.4, 101.3, 101.2, 101.1, 101.0, 100.9, 100.8, 100.7, 100.6, 100.5, 100.4, 100.3, 100.2, 100.1, 100.0, 99.9, 99.8, 99.7, 99.6, 99.5, 99.4, 99.3, 99.2, 99.1, 99.0, 98.9, 98.8, 98.7, 98.6, 98.5, 98.4, 98.3, 98.2, 98.1, 98.0, 97.9, 97.8, 97.7, 97.6, 97.5, 97.4, 97.3, 97.2, 97.1, 97.0, 96.9, 96.8, 96.7, 96.6, 96.5, 96.4, 96.3, 96.2, 96.1, 96.0, 95.9, 95.8, 95.7, 95.6, 95.5, 95.4, 95.3, 95.2, 95.1, 95.0, 94.9, 94.8, 94.7, 94.6, 94.5, 94.4, 94.3, 94.2, 94.1, 94.0, 93.9, 93.8, 93.7, 93.6, 93.5, 93.4, 93.3, 93.2, 93.1, 93.0, 92.9, 92.8, 92.7, 92.6, 92.5, 92.4, 92.3, 92.2, 92.1, 92.0, 91.9, 91.8, 91.7, 91.6, 91.5, 91.4, 91.3, 91.2, 91.1, 91.0, 90.9, 90.8, 90.7, 90.6, 90.5, 90.4, 90.3, 90.2, 90.1, 90.0, 89.9, 89.8, 89.7, 89.6, 89.5, 89.4, 89.3, 89.2, 89.1, 89.0, 88.9, 88.8, 88.7, 88.6, 88.5, 88.4, 88.3, 88.2, 88.1, 88.0, 87.9, 87.8, 87.7, 87.6, 87.5, 87.4, 87.3, 87.2, 87.1, 87.0, 86.9, 86.8, 86.7, 86.6, 86.5, 86.4, 86.3, 86.2, 86.1, 86.0, 85.9, 85.8, 85.7, 85.6, 85.5, 85.4, 85.3, 85.2, 85.1, 85.0, 84.9, 84.8, 84.7, 84.6, 84.5, 84.4, 84.3, 84.2, 84.1, 84.0, 83.9, 83.8, 83.7, 83.6, 83.5, 83.4, 83.3, 83.2, 83.1, 83.0, 82.9, 82.8, 82.7, 82.6, 82.5, 82.4, 82.3, 82.2, 82.1, 82.0, 81.9, 81.8, 81.7, 81.6, 81.5, 81.4, 81.3, 81.2, 81.1, 81.0, 80.9, 80.8, 80.7, 80.6, 80.5, 80.4, 80.3, 80.2, 80.1, 80.0, 79.9, 79.8, 79.7, 79.6, 79.5, 79.4, 79.3, 79.2, 79.1, 79.0, 78.9, 78.8, 78.7, 78.6, 78.5, 78.4, 78.3, 78.2, 78.1, 78.0, 77.9, 77.8, 77.7, 77.6, 77.5, 77.4, 77.3, 77.2, 77.1, 77.0, 76.9, 76.8, 76.7, 76.6, 76.5, 76.4, 76.3, 76.2, 76.1, 76.0, 75.9, 75.8, 75.7, 75.6, 75.5, 75.4, 75.3, 75.2, 75.1, 75.0, 74.9, 74.8, 74.7, 74.6, 74.5, 74.4, 74.3, 74.2, 74.1, 74.0, 73.9, 73.8, 73.7, 73.6, 73.5, 73.4, 73.3, 73.2, 73.1, 73.0, 72.9, 72.8, 72.7, 72.6, 72.5, 72.4, 72.3, 72.2, 72.1, 72.0, 71.9, 71.8, 71.7, 71.6, 71.5, 71.4, 71.3, 71.2, 71.1, 71.0, 70.9, 70.8, 70.7, 70.6, 70.5,

133-6.134.fid
Mr.A.Gharakhani
Sample: 104dp- 13Cnmr in DMSO

Chemical structure of the compound (104dp-):

CN1C(=O)C2=C(C1)C(=C(C=C2)C3=CC(=CC=C3)N)C4=CC(=CC=C4)N

13C NMR peaks (ppm):

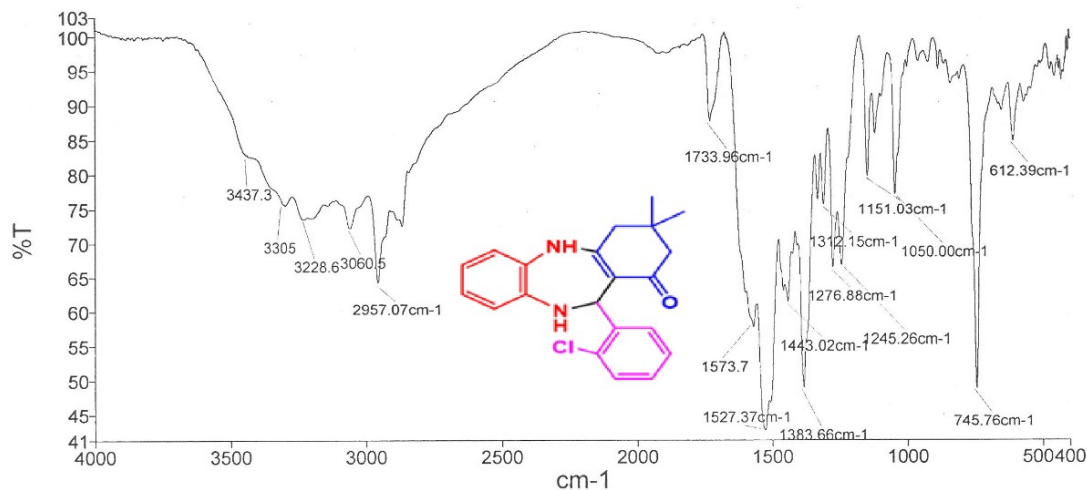
- 146.69
- 143.73
- 134.82
- 133.59
- 128.28
- 127.45
- 127.32
- 125.83
- 117.37
- 116.11
- 114.63
- 50.28
- 32.32
- 31.42
- 31.09
- 27.95

FT-IR (KBr, cm⁻¹): 612, 745, 1050, 1151, 1245, 1312, 1383, 1443, 1527, 1573, 1733, 2957, 3060, 3228,

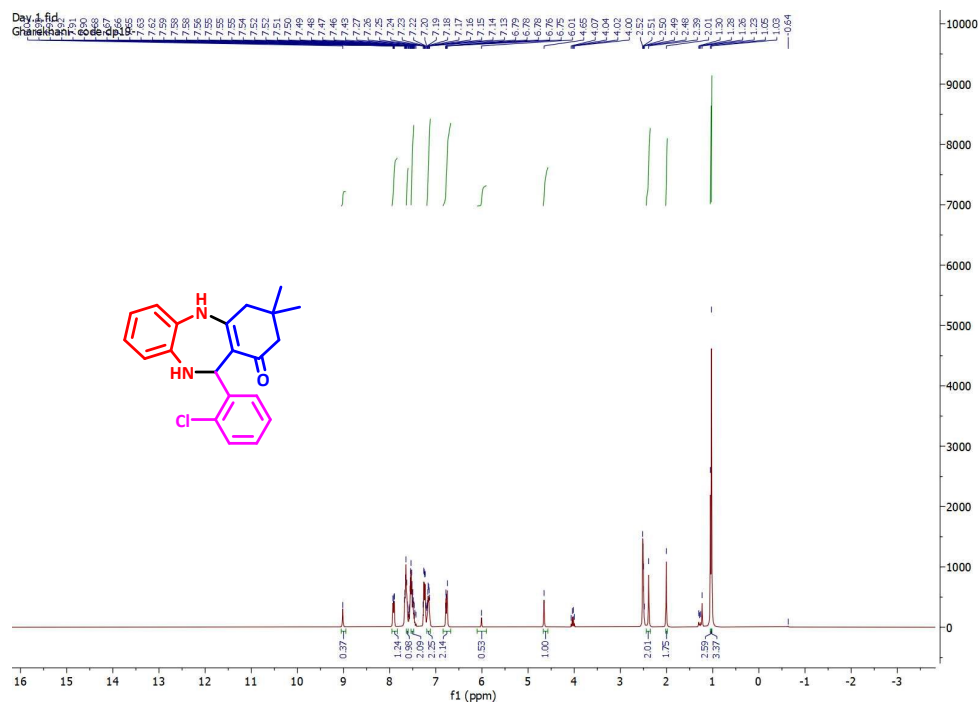
2205, 3437.

¹H NMR (300 MHz, DMSO) δ 9.0 (s, 1H), 7.95 – 6.68 (m, 8H), 6.01 (s, 1H), 4.65 (s, 1H), 2.39 (s, 2H), 2.01 (s, 2H), 1.05 (s, 3H), 1.03 (s, 3H).

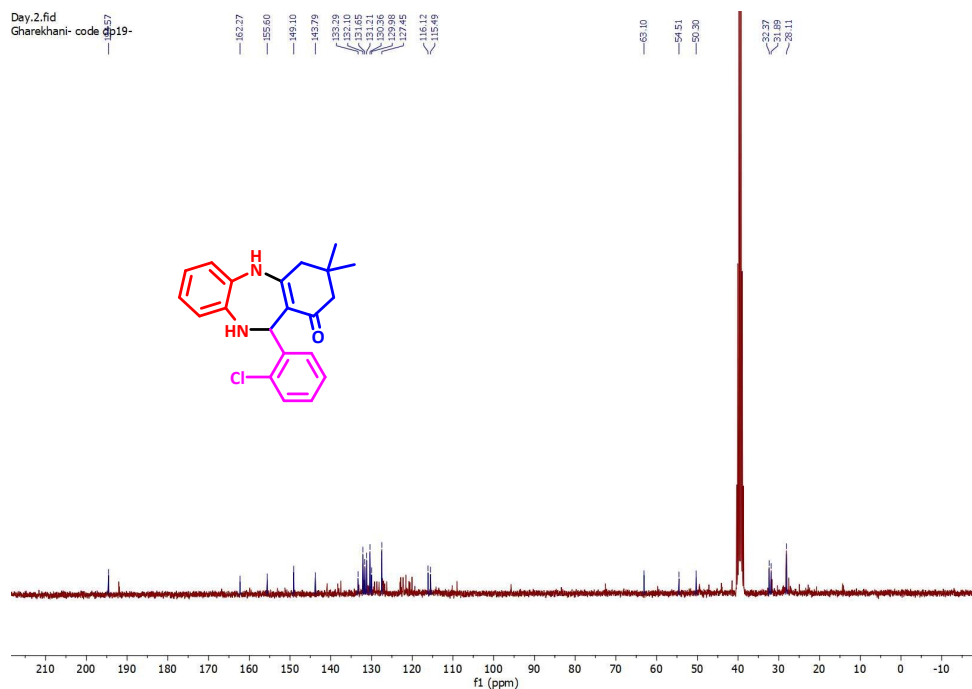
¹³C NMR (75 MHz, DMSO) δ 194.5, 162.2, 155.6, 149.1, 143.7, 133.2, 132.1, 131.6, 131.2, 130.3, 129.9, 127.4, 116.1, 115.4, 63.1, 54.5, 50.3, 32.3, 31.8, 28.1.



FT-IR spectrum of the compound 11-(2-chlorophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



¹H NMR spectrum of the compound 11-(2-chlorophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one



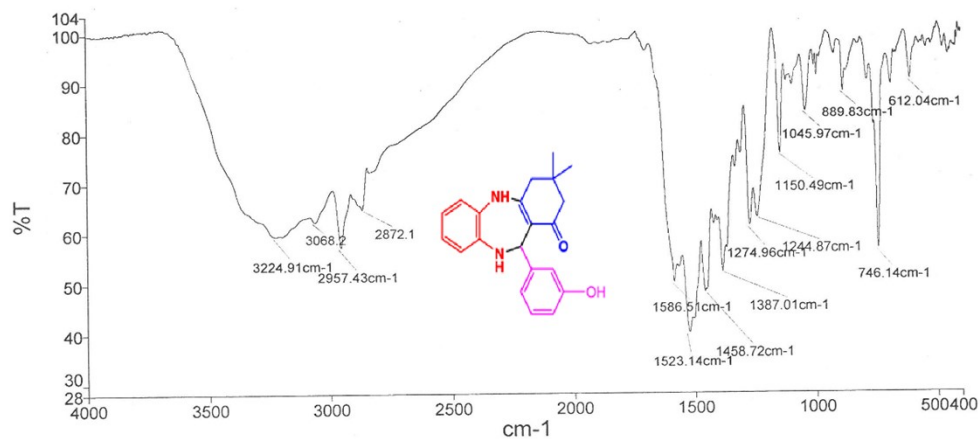
¹³C NMR spectrum of the compound 11-(2-chlorophenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-one

11-(3-hydroxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-One

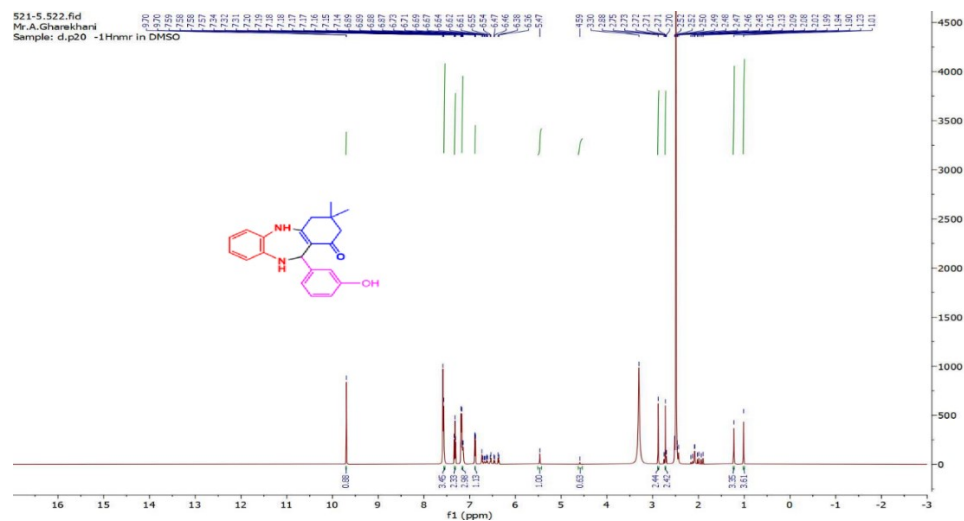
FT-IR (KBr, cm⁻¹): 612, 746, 889, 1045, 1150, 1244, 1274, 1387, 1458, 1523, 1586, 2872, 2957, 3068, 3224.

¹H NMR (300 MHz, DMSO) δ 9.76 (s, 1H), 8.20 (s, 1H), 7.62 – 6.57 (m, 8H), 4.66 (s, 1H), 2.50 (s, 2H), 2.01 (s, 2H), 1.03 (s, 3H), 0.98 (d, J = 4.2 Hz, 3H).

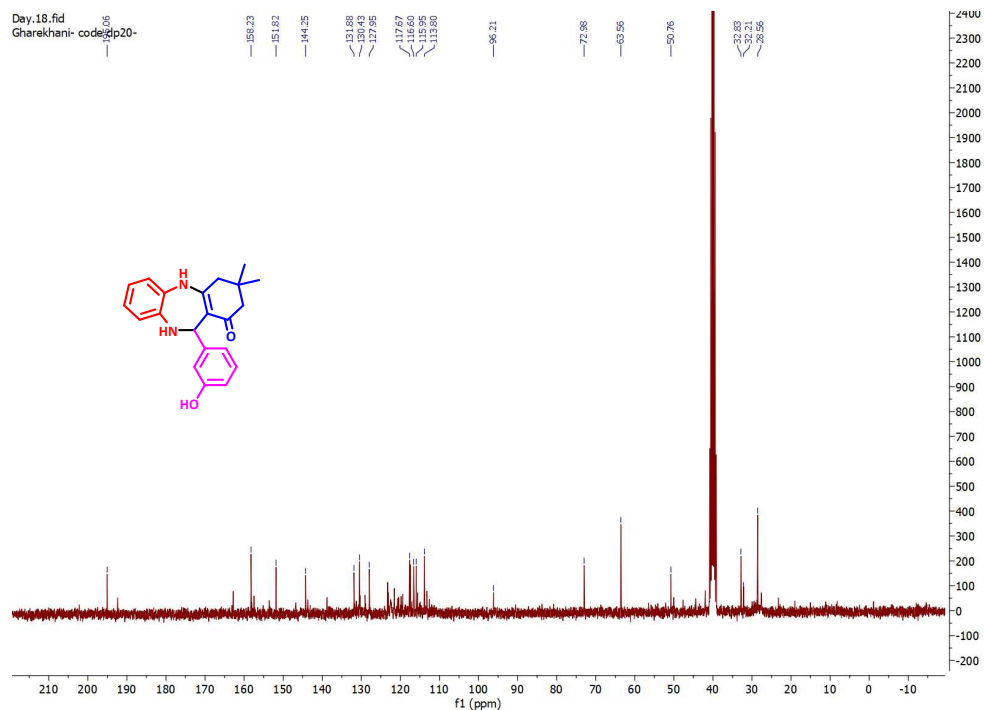
¹³C NMR (75 MHz, DMSO) δ 195.0, 158.2, 151.8, 144.2, 131.8, 130.4, 127.9, 117.6, 116.6, 115.9, 113.8, 96.2, 72.9, 63.5, 50.7, 32.8, 32.2, 28.5.



FT-IR spectrum of the compound 11-(3-hydroxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-One



¹H NMR spectrum of the compound 11-(3-hydroxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-One



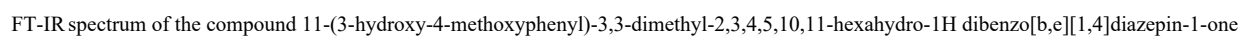
¹³C NMR spectrum of the compound 11-(3-hydroxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H-dibenzo[b,e][1,4]diazepin-1-One

11-(3-hydroxy-4-methoxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H dibenzo[b,e][1,4]diazepin-1-one:

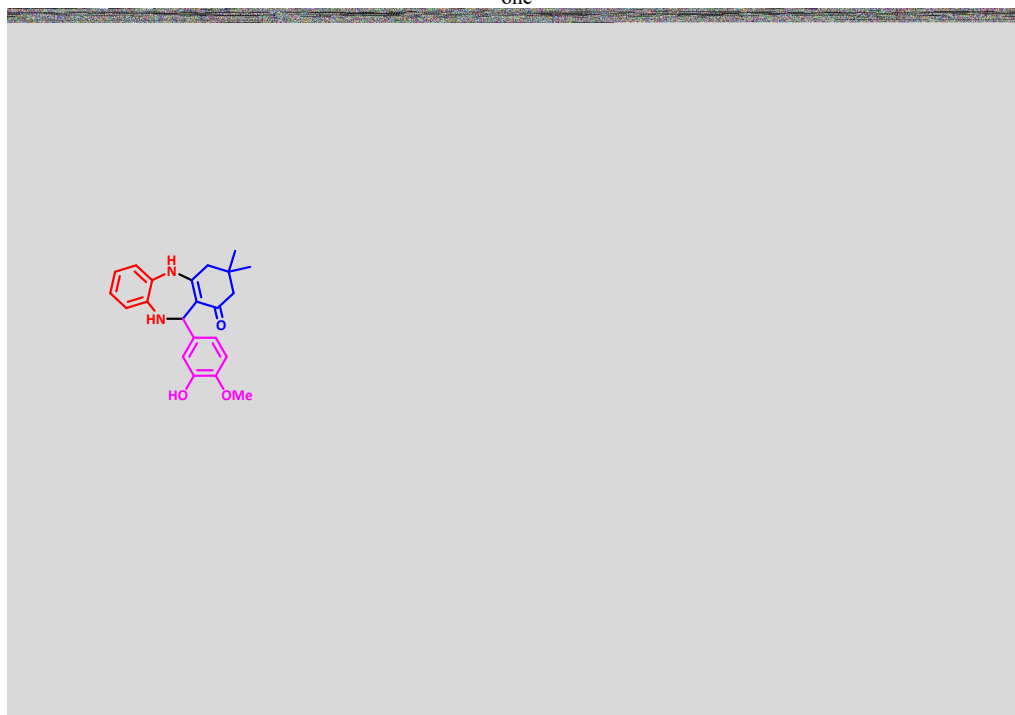
FT-IR (KBr, cm⁻¹): 611, 746, 1034, 1150, 1273, 1386, 1452, 1503, 1566, 2872, 2957, 3228, 3315.

¹H NMR (500 MHz, DMSO) δ 8.40 (s, 1H), 7.78- 6.53 (m, 7H), 4.91 (s, 1H), 3.88 (s, 3H), 2.34 (s, 2H), 2.01 (s, 3H), 1.06 (s, 3H), 1.05 (s, 3H).

¹³C NMR (126 MHz, DMSO) δ 194.8, 160.9, 151.8, 148.5, 147.8, 133.4, 122.7, 121.6, 119.7, 115.6, 115.3, 114.9, 110.3, 96.5, 56.0, 50.2, 32.3, 31.4, 28.5, 18.5.



¹H NMR spectrum of the compound 11-(3-hydroxy-4-methoxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H dibenzo[b,e][1,4]diazepin-1-one



¹³C NMR spectrum of the compound 11-(3-hydroxy-4-methoxyphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1H dibenzo[b,e][1,4]diazepin-1-one