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$MnFe_2O_4$@NH_2@2AB-Ni: A novel, highly active, stable and magnetically recoverable nanocatalyst and use of this heterogeneous catalyst in green synthesis of spirooxindoles in water

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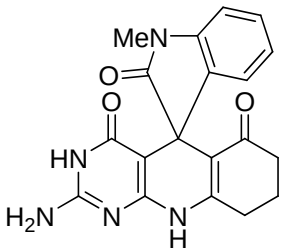
Experimental Part

General. The chemicals used in this work were obtained from Fluka and Merck and were used without purification. Melting points were measured on an Electrothermal 9200 apparatus. IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FT-IR spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. ^{1}H and ^{13}C NMR spectra were obtained on solutions in DMSO-d_6 using tetramethylsilane as internal reference. X-ray diffraction (XRD) pattern of the as-synthesized material was obtained using a Holland Philips Xpert X-ray powder diffraction (XRD) diffractometer (CuK, radiation, λ= 0.154056 nm), at a scanning speed of 2°/min from 10° to 100° (2θ). The nanocatalyst was determined using a KYKY EM-3200 Scanning Electron Microscope (SEM) operated at a 26 kV accelerating voltage. Thermogravimetric/differential thermal analyses (TG/DTA) was performed on a Thermal Analyzer with a heating rate of 20 °C min^{-1} over a temperature range of 25–800 °C under flowing compressed N_2. The content of nickel in the heterogenized catalyst was determined by VISTA-PRO, CCD simultaneous ICP analyser. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

Typical procedure for the preparation of spirooxindoles 4(a-f) and 8(a-h).

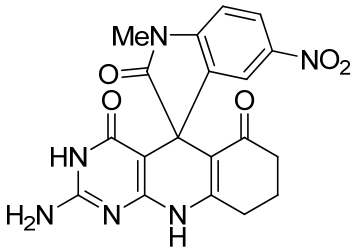
A mixture of isatin **1** (1 mmol), cyclohexane-1,3-dione **2** (1 mmol), 2,6-diaminopyrimidin-4(3*H*)-one **3** or barbituric acid **7** (1 mmol), catalyst (10 mg) and water (5 mL) was added in a round-bottomed flask and stirred at 90 ºC. After completion of the reaction (monitored by TLC) the catalyst was easily separated from the reaction mixture with an external magnet. After separation of catalyst, the reaction mixture was filtered and the precipitate washed with water and recrystallized by EtOH to afford the pure product.

2'-Amino-1-methyl-8',9'-dihydro-3'*H*-spiro[indoline-3,5'-pyrimido[4,5-*b*]quinoline]-2,4',6'(7'*H*,10'*H*)-trione (4a).



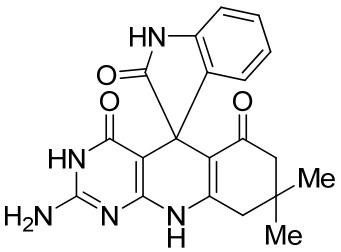
Cream powder (79%); mp >300 ˚C dec. IR (KBr) (ν_{max}/ cm^{-1}): 3438, 3288, 2933, 1677, 1652. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 1.79 (2H, brs, CH_2), 2.03 (2H, brs, CH_2), 2.52 (2H, brs, CH_2), 3.04 (3H, s, NCH_3), 6.40 (2H, s, NH_2), 6.75-7.07 (4H, m, H-Ar), 9.55 (1H, s, NH), 10.12 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 21.2, 26.6, 27.3, 37.5, 48.4, 91.8, 106.8, 110.5, 121.4, 122.7, 127.3, 136.6, 145.1, 153.8, 154.4, 154.6, 160.4, 179.5, 193.4.

2'-Amino-1-methyl-5-nitro-8',9'-dihydro-3'*H*-spiro[indoline-3,5'-pyrimido[4,5-*b*] quinoline]-2,4',6'(7'*H*,10'*H*)-trione (4b).



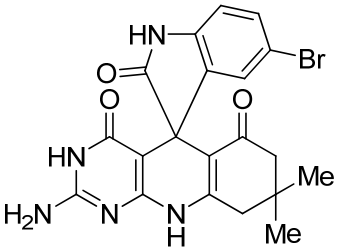
Cream powder (80%); mp >300 ˚C dec. IR (KBr) (ν_{max}/ cm^{-1}): 3299, 3167, 2944, 1688, 1669. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 1.81-1.82 (2H, m, CH_2), 2.07-2.11 (2H, m, CH_2), 2.55-2.59 (2H, m, CH_2), 3.16 (3H, s, NCH_3), 6.53 (2H, s, NH_2), 7.02-8.17 (3H, m, H-Ar), 9.80 (1H, s, NH), 10.28 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 21.0, 26.9, 27.2, 37.2, 48.5, 90.8, 106.8, 109.4, 117.8, 125.2, 137.5, 142.3, 151.4, 154.1, 154.9, 155.7, 160.6, 180.3, 193.9.

2'-Amino-8',8'-dimethyl-8',9'-dihydro-3'*H*-spiro[indoline-3,5'-pyrimido[4,5-*b*]quinoline] -2,4',6'(7'*H*,10'*H*)-trione (4c).



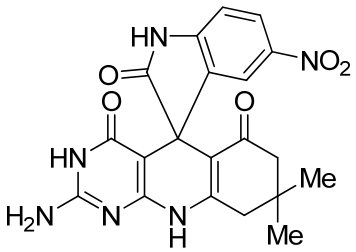
Cream powder (96%); mp >300 ˚C dec. IR (KBr) (ν_{max}/ cm^{-1}): 3334, 3189, 2938, 1697, 1655. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 0.90 (3H, s, CH_3), 0.98 (3H, s, CH_3), 1.82-2.08 (2H, m, CH_2), 2.32-2.50 (2H, m, CH_2), 6.35 (2H, s, NH_2), 6.57-6.99 (4H, m, H-Ar), 9.45 (1H, s, NH), 9.92 (1H, s, NH), 10.24 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 26.9, 28.9, 32.3, 48.8, 51.0, 92.1, 108.1, 109.3, 120.6, 122.8, 127.0, 137.3, 143.6, 152.2, 153.9, 154.5, 160.5, 180.7, 193.1.

2'-Amino-5-bromo-8',8'-dimethyl-8',9'-dihydro-3'*H*-spiro[indoline-3,5'-pyrimido[4,5-*b*] quinoline]-2,4',6'(7'*H*,10'*H*)-trione (4d).



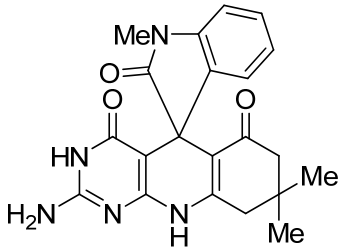
Cream powder (94%); mp >300 ˚C dec. IR (KBr) (ν_{max}/ cm^{-1}): 3416, 3164, 2959, 1701, 1645. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 0.91 (3H, s, CH_3), 0.97 (3H, s, CH_3), 1.88-2.06 (2H, m, CH_2), 2.41 (2H, brs, CH_2), 6.42 (2H, s, NCH_2), 6.54-7.15 (3H, m, H-Ar), 9.55 (1H, s, NH), 10.12 (1H, s, NH), 10.31 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 27.3, 28.5, 32.3, 49.2, 50.9, 91.5, 108.7, 110.0, 112.2, 125.4, 129.7, 139.8, 143.2, 152.8, 154.0, 154.7, 160.5, 180.3, 193.3.

2'-Amino-8',8'-dimethyl-5-nitro-8',9'-dihydro-3'*H*-spiro[indoline-3,5'-pyrimido[4,5-*b*] quinoline]-2,4',6'(7'*H*,10'*H*)-trione (4e).



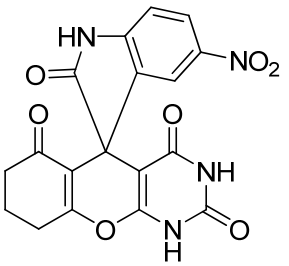
Cream powder (93%); mp >300 ˚C dec. IR (KBr) (ν_{max}/ cm^{-1}): 3427, 3185, 2923, 1720, 1662. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 0.92 (3H, s, CH_3), 0.97 (3H, s, CH_3), 1.90-2.06 (2H, m, CH_2), 2.47 (2H, brs, CH_2), 6.49 (2H, s, NH_2), 6.80-8.03 (3H, m, H-Ar), 9.70 (1H, s, NH), 10.38 (1H, s, NH), 10.79 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 27.4, 28.3, 32.4, 49.0, 50.7, 91.0, 108.0, 108.3, 117.8, 125.1, 138.3, 141.7, 150.6, 153.5, 154.2, 154.8, 160.7, 181.4, 193.6.

2'-Amino-1,8',8'-trimethyl-8',9'-dihydro-3'*H*-spiro[indoline-3,5'-pyrimido[4,5-*b*] quinoline]-2,4',6'(7'*H*,10'*H*)-trione (4f).



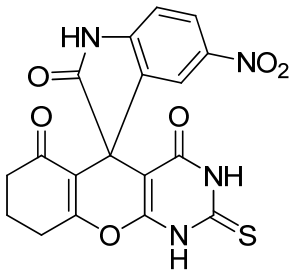
Cream powder (92%); mp >300 ˚C dec. IR (KBr) (ν_{max}/ cm^{-1}): 3330, 3176, 2940, 1691, 1652. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 0.89 (3H, s, CH_3), 0.97 (3H, s, CH_3), 1.81-2.05 (2H, m, CH_2), 2.34-2.49 (2H, m, CH_2), 3.05 (3H, s, NCH_3), 6.40 (2H, s, NH_2), 6.86-7.07 (4H, m, H-Ar), 9.51 (1H, s, NH), 10.13 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 26.6, 27.0, 28.9, 32.3, 48.3, 50.9, 91.8, 106.8, 109.1, 121.4, 122.5, 127.3, 136.4, 145.1, 152.4, 153.9, 154.6, 160.4, 179.4, 193.1.

5'-Nitro-8,9-dihydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2,2',4,6 (1*H*,3*H*,7*H*)-tetraone (8a)



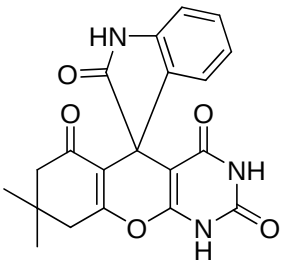
Colorless crystals (92%); mp 278-280 ˚C dec. IR (KBr) (ν_{max} /cm^{-1}): 3224, 1734, 1687, 1622. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 1.92 (2H, brs, CH_2), 2.25 (2H, brs, CH_2), 2.72 (2H, brs, CH_2), 6.91 (1H, d, $^{3}J_{HH}$ = 8.4 Hz, H-Ar), 8.05 (1H, s, H-Ar), 8.09 (1H, d, $^{3}J_{HH}$ = 8.4 Hz, H-Ar), 11.11 (1H, s, NH), 11.16 (1H, s, NH), 12.31 (1H, brs, NH).^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 20.0, 27.3, 37.0, 45.6, 88.6, 108.8, 113.6, 119.6, 126.1, 135.0, 142.3, 149.4, 150.9, 153.8, 162.2, 166.7, 179.0, 196.0.

5'-Nitro-2-thioxo-2,3,8,9-tetrahydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2',4,6(1*H*,7*H*)-trione (8b)



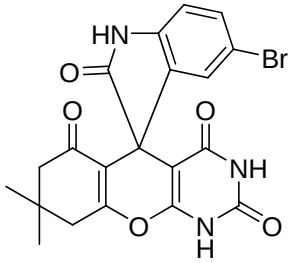
Colorless crystals (88%); mp >300 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3363, 3060, 1717, 1683, 1622. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 1.69-1.75 (2H, brs, CH_2), 2.25 (2H, brs, CH_2), 2.72 (2H, brs, CH_2), 6.91 (1H, d, $^{3}J_{HH}$ = 6.0 Hz, H-Ar), 8.08-8.09 (2H, m, H-Ar), 11.20 (1H, s, NH), 12.48 (1H, s, NH), 13.75 (1H, brs, NH).^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 19.9, 27.3, 37.0, 45.6, 93.2, 108.8, 113.4, 119.9, 126.2, 134.4, 142.4, 150.9, 153.2, 159.8, 166.6, 174.1, 175.5, 195.9.

8,8-Dimethyl-8,9-dihydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2,2',4,6(1*H*,3*H*,7*H*)-tetraone (8c)



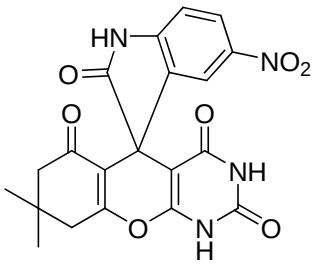
White powder (96%); mp >300 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3337, 3265, 1746, 1664, 1620. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 0.97 (3H, s, CH_3), 1.03 (3H, s, CH_3), 2.07, 2.10 (2H, AB_q, $^{3}J_{AB}$ = 15.9 Hz, CH_2), 2.55, 2.66 (2H, AB_q, $^{3}J_{AB}$ = 17.6 Hz, CH_2), 6.71 (1H, d, $^{3}J_{HH}$ = 7.8 Hz H-Ar), 6.78 (1H, t, $^{3}J_{HH}$ = 7.5 Hz H-Ar), 6.97 (1H, d, $^{3}J_{HH}$ = 7.5 Hz, H-Ar), 6.08 (1H, t, $^{3}J_{HH}$ = 7.5 Hz, H-Ar), 10.36(1H, s, NH), 11.01(1H, s, NH), 12.19(1H, brs, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 27.1, 28.2, 32.2, 45.5, 50.8, 89.6, 109.0, 113.6, 121.3, 123.3, 128.3, 134.0, 144.3, 149.4, 153.5, 161.9, 163.6, 178.2, 195.3.

5'-Bromo-8,8-dimethyl-8,9-dihydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2,2',4,6(1*H*,3*H*,7*H*)-tetraone (8d)



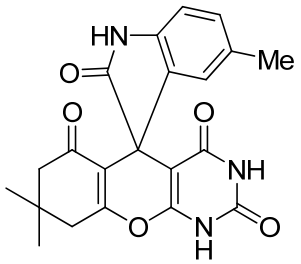
Colorless crystals (94%); mp >300 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3162, 3121, 3049, 1739, 1724, 16581, 1617. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 0.99 (3H, s, CH_3), 1.02 (3H, s, CH_3), 2.09-2.21 (2H, m, CH_2), 2.60 (2H, brs, CH_2), 6.67 (1H, d, $^{3}J_{HH}$ = 8.7 Hz, H-Ar), 7.23-7.25 (2H, m, H-Ar), 10.52 (1H, s, NH), 11.05 (1H, s, NH), 12.23 (1H, brs, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 27.5, 27.8, 32.2, 45.7, 50.8, 89.0, 110.7, 112.9, 113.0, 126.3, 130.9, 136.4, 143.7, 149.5, 153.7, 162.0, 164.0, 177.9, 195.6.

8,8-Dimethyl-5'-nitro-8,9-dihydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2,2',4,6(1*H*,3*H*,7*H*)-tetraone (8e)



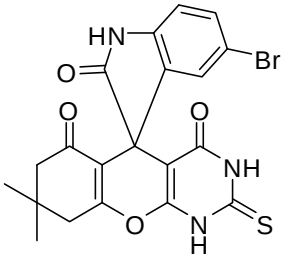
Colorless crystals (96%); mp 290-292 ˚C dec. IR (KBr) (ν_{max} /cm^{-1}): 3041, 2957, 1730, 1691, 1632. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 0.99 (3H, s, CH_3), 1.02 (3H, s, CH_3), 2.167 (2H, brs, CH_2), 2.64 (2H, brs, CH_2), 6.91 (1H, d, $^{3}J_{HH}$ = 8.5 Hz, H-Ar), 8.04 (1H, s, H-Ar), 8.04-8.11 (1H, m, H-Ar), 11.10 (1H, s, NH), 11.16(1H, s, NH), 12.31(1H, brs, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 27.6, 27.7, 32.3, 45.6, 50.6, 88.5, 108.8, 112.6, 119.3, 126.1, 135.0, 142.3, 149.4, 151.0, 154.0, 162.2, 164.7, 178.9, 195.9.

5',8,8-Trimethyl-8,9-dihydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2,2',4,6 (1*H*,3*H*,7*H*)-tetraone (8f)



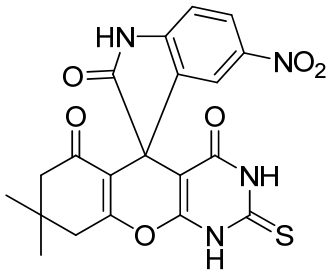
Colorless crystals (95%); mp 290-292 ˚C dec. IR (KBr) (ν_{max} /cm^{-1}): 3368, 3178, 2962, 1724, 1680, 1622. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 0.98 (3H, s, CH_3), 1.02 (3H, s, CH_3), 2.06-2.21 (2H, m, CH_2), 2.59-2.61 (2H, m, CH_2), 3.66 (3H, s, CH_3), 6.59 (1H, d, $^{3}J_{HH}$ = 7.8 Hz, H-Ar), 7.81 (1H, s, H-Ar), 7.87 (1H, d, $^{3}J_{HH}$ = 7.8 Hz, H-Ar), 10.26 (1H, s, NH), 11.01 (1H, s, NH), 12.19 (1H, s, NH) . ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 19.0, 21.1, 27.3, 28.0, 32.0, 45.5, 49.9, 89.7, 108.7, 113.6, 124.0, 128.5, 130.0, 134.1, 141.8, 149.5, 153.4, 161.9, 163.5, 178.1, 195.3.

5'-Bromo-8,8-dimethyl-2-thioxo-2,3,8,9-tetrahydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2',4,6(1*H*,7*H*)-trione (8g)

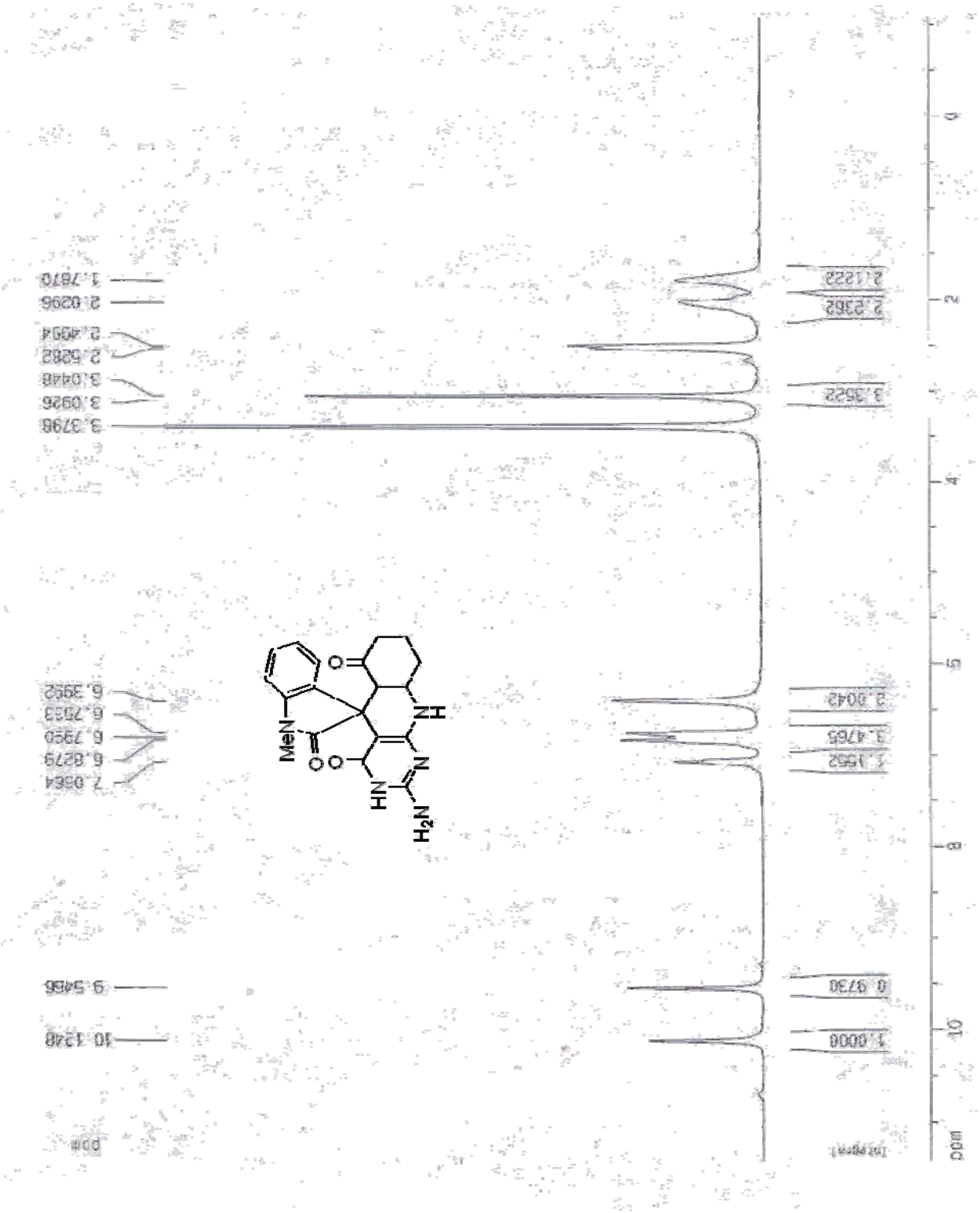


Colorless crystals (90%); mp 280-282 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3060, 2967, 1711, 1708. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 0.99 (3H, s, CH_3), 1.02 (3H, s, CH_3), 2.16 (2H, brs, CH_2), 2.60 (2H, brs , CH_2), 6.67 (1H, d, $^{3}J_{HH}$ = 8.1 Hz, H-Ar), 7.25 (1H, d, $^{3}J_{HH}$ = 7.2 Hz, H-Ar), 7.33 (1H, s, H-Ar), 10.58 (1H, s, NH), 12.45 (1H, s, NH), 13.68 (1H, brs, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 27.5, 27.8, 32.3, 45.7, 50.7, 93.7, 110.8, 112.9, 113.0, 126.6, 131.1, 135.8, 143.7, 153.1, 159.7, 164.0, 174.1, 177.4, 195.5.

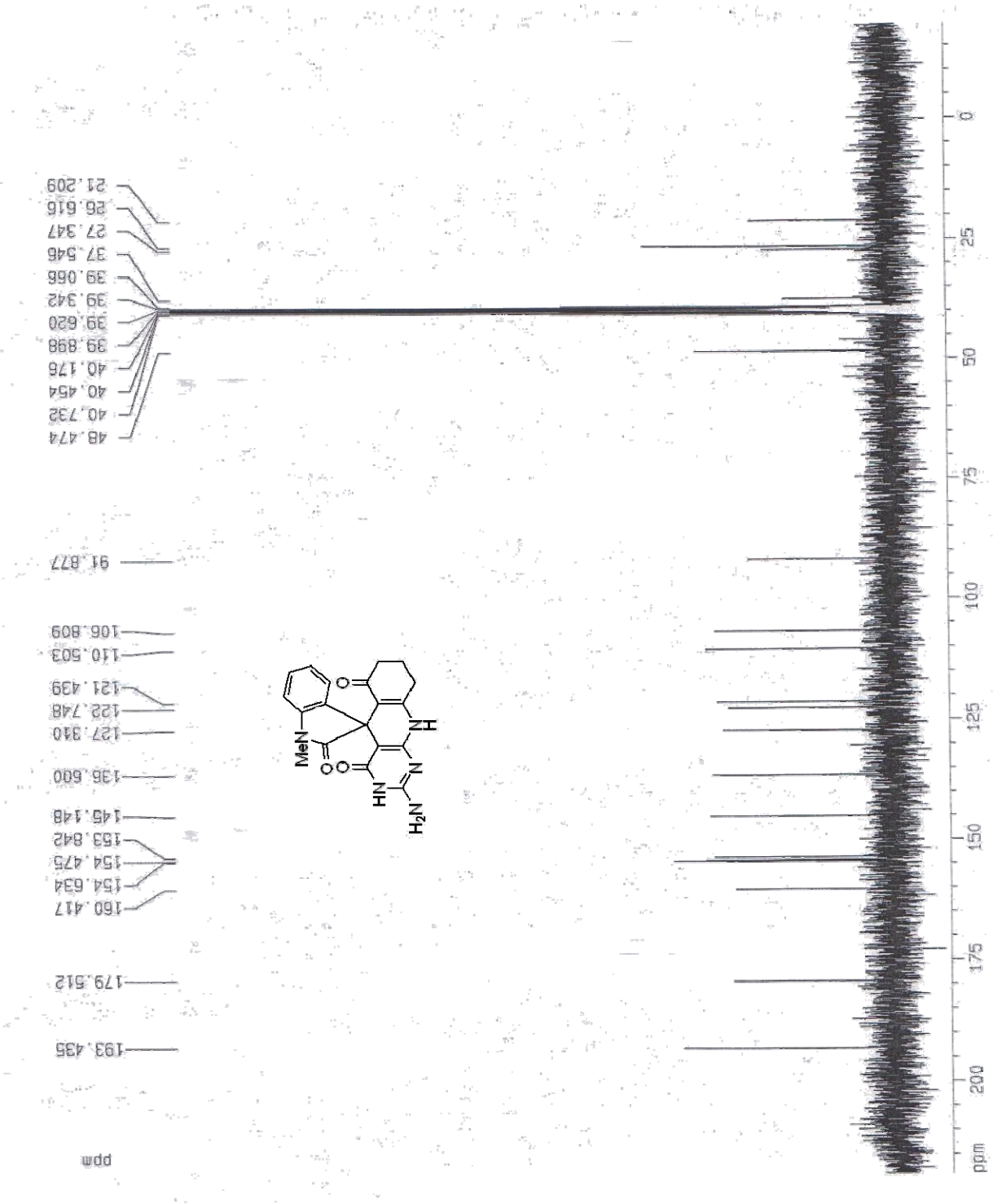
8,8-Dimethyl-5'-nitro-2-thioxo-2,3,8,9-tetrahydrospiro[chromeno[2,3-*d*]pyrimidine-5,3'-indoline]-2',4,6(1*H*,7*H*)-trione (8h)



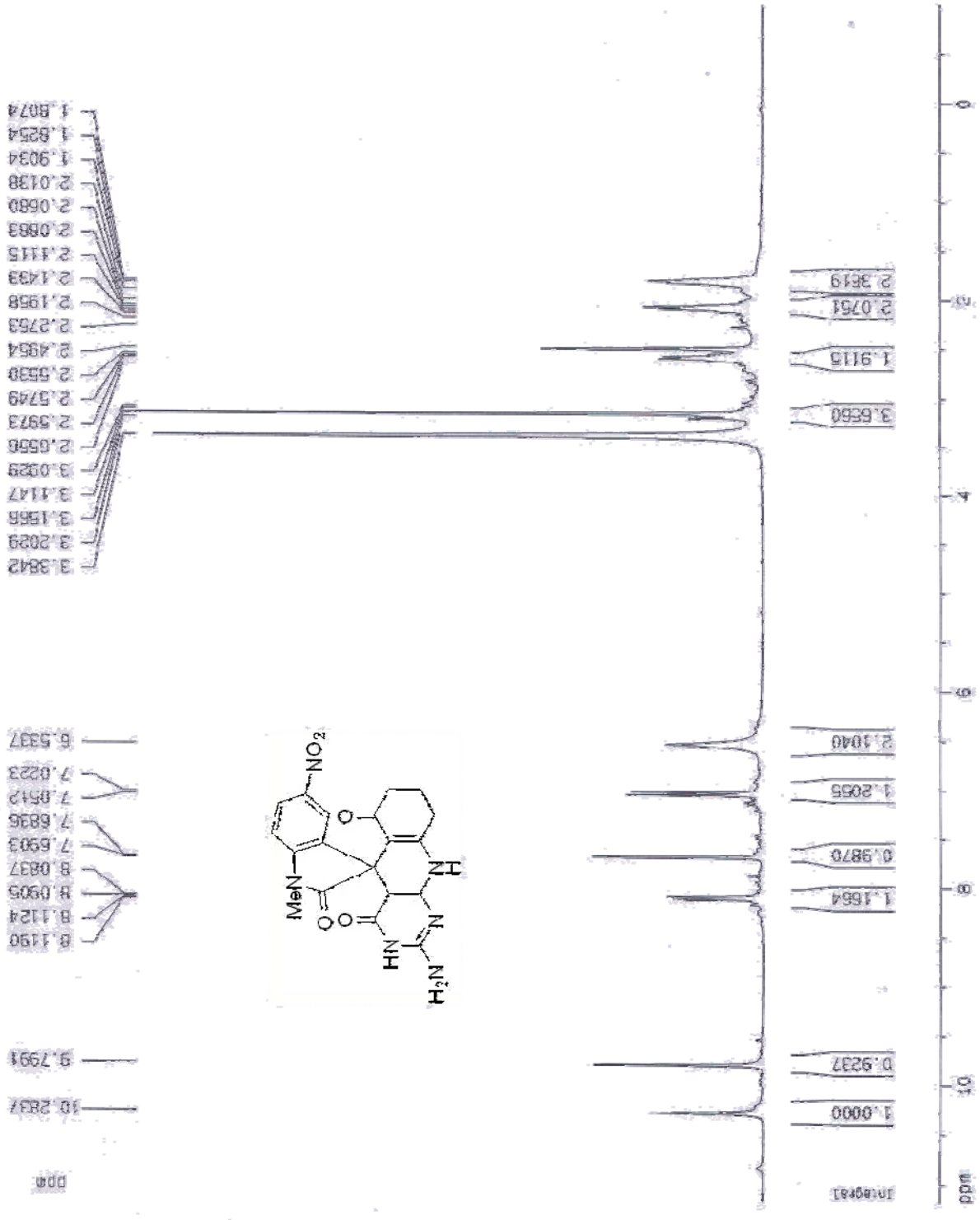
Colorless crystals (92%); mp >300 ˚C. IR (KBr) (ν_{max} /cm^{-1}): 3383, 3337, 2962, 1730, 1684, 1627. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ_H (ppm) 1.00-1.07 (6H, m, 2CH_3), 2.17 (2H, brs, CH_2), 2.63 (2H, brs, CH_2), 6.93 (1H, d, $^{2}J_{AB}$ = 8.1 Hz, H-Ar), 8.09-8.14 (2H, m, H-Ar), 11.21 (1H, s, NH), 12.48 (1H, s, NH), 13.76 (1H, brs, NH) . ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ_C (ppm) 19.0, 27.6, 32.3, 45.5, 50.5, 56.5, 93.2, 108.9, 112.4, 119.7, 126.2, 134.4, 142.4, 150.9, 153.4, 160.0, 164.7, 174.2, 195.8.


MeN
O
O
O
NH
N
HN
H_2N
1.7870
2.0296
2.4094
2.5282
3.0448
3.0926
3.3798
6.3992
6.7533
6.7950
6.8279
7.0564
9.5466
10.1248
1.0000
0.9730
1.1562
3.4765
2.0043
3.3522
2.2362
2.1222
ppm

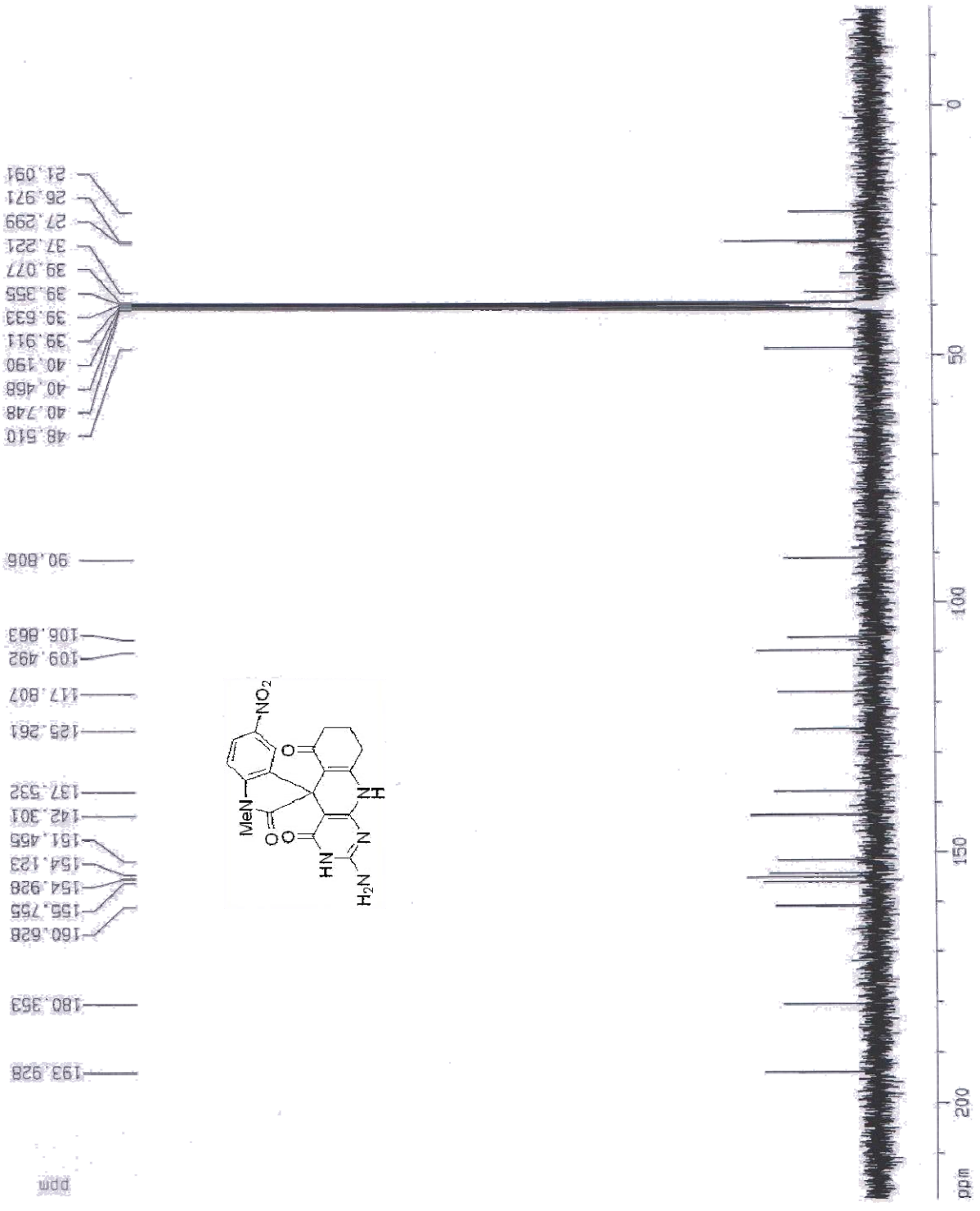
^{1}H NMR of **4a**


21.209
26.616
27.347
37.546
39.066
39.342
39.620
39.898
40.176
40.454
40.732
48.474
91.877
106.809
110.503
121.439
122.748
127.310
136.600
145.148
153.842
154.475
154.634
160.417
179.512
193.435
MeN
HN
NH
N
H2N
O
O
O
0
25
50
75
100
125
150
175
200
ppm

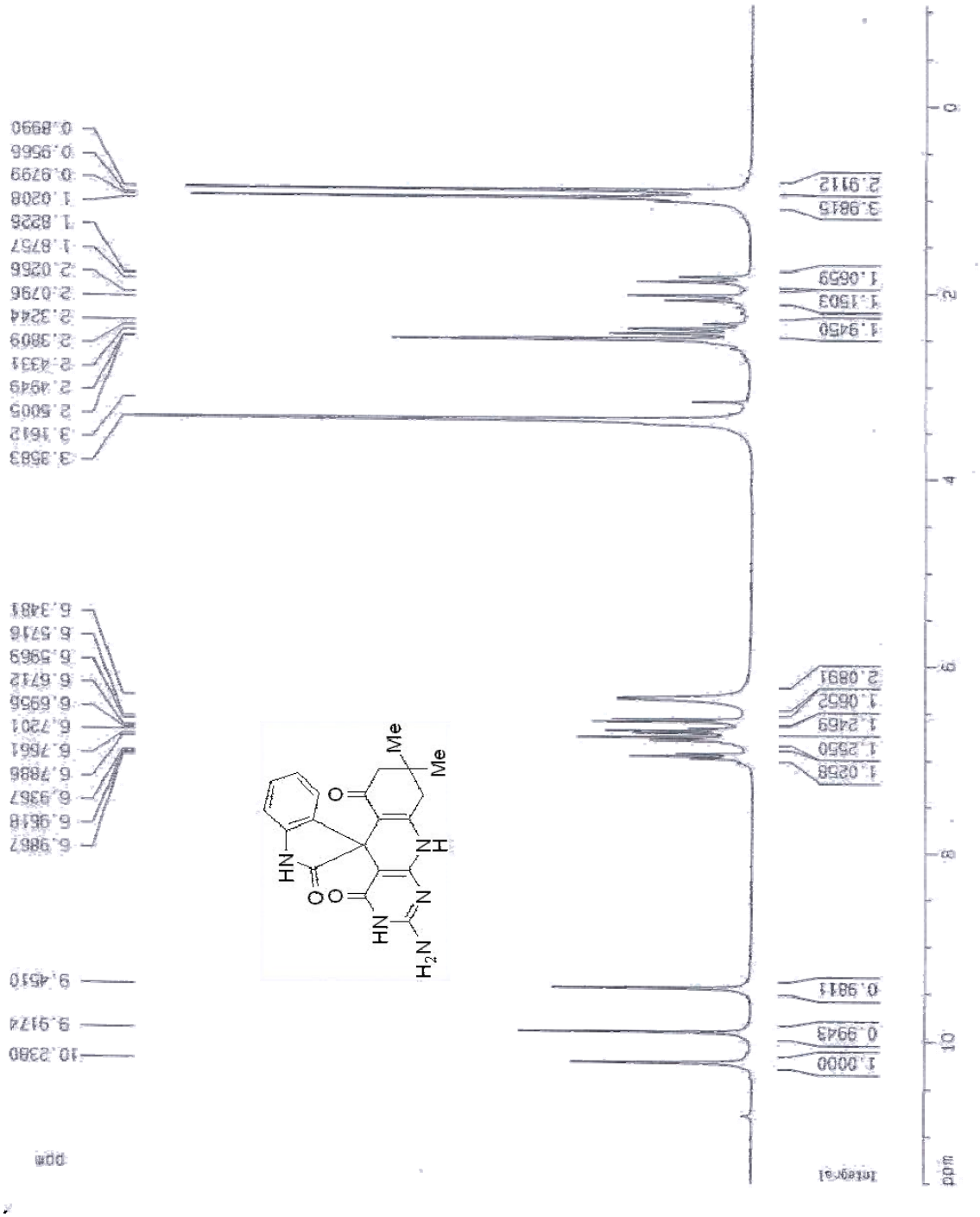
^{13}C NMR of **4a**



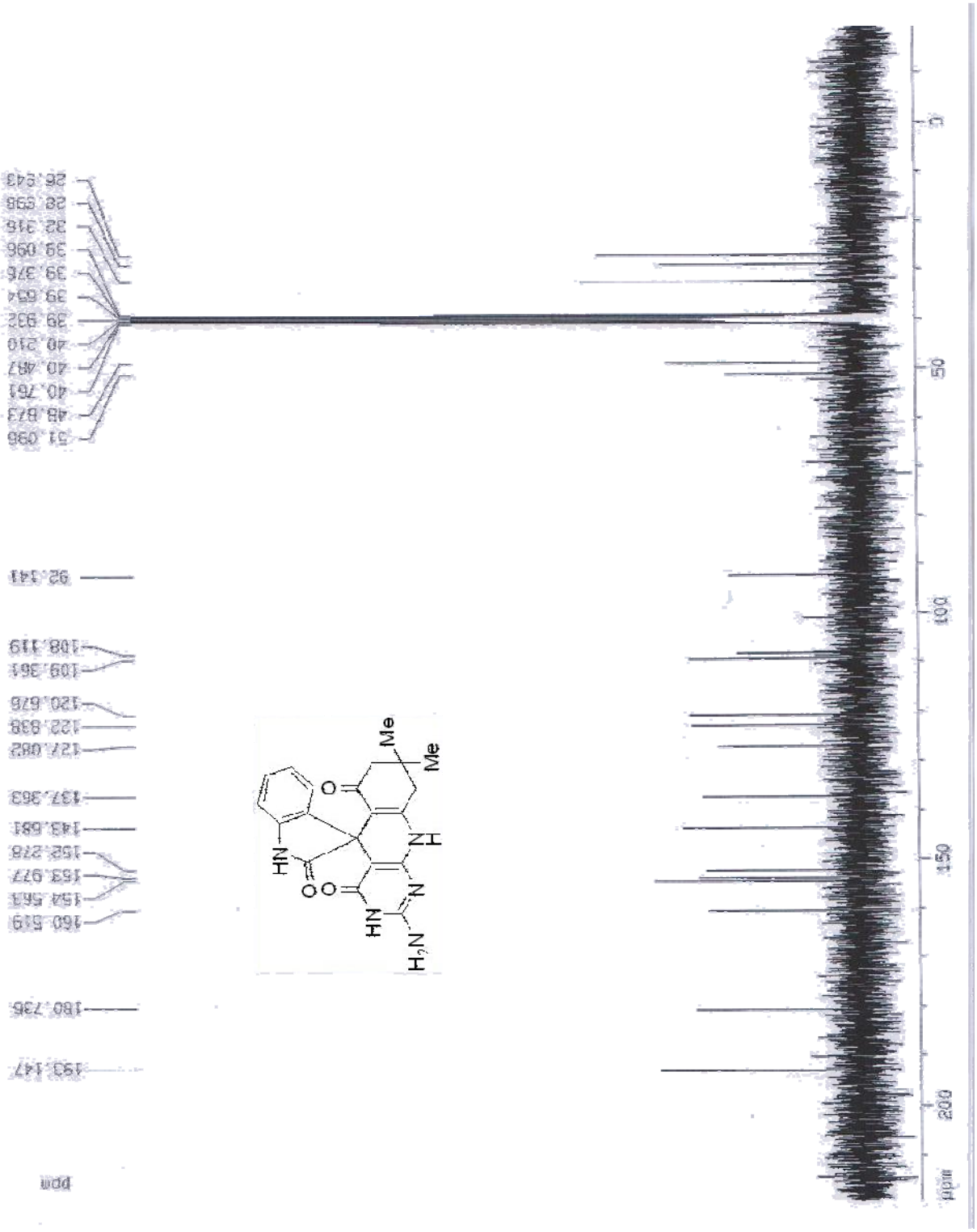
^{1}H NMR of **4b**


21.091
26.971
27.299
37.221
39.077
39.355
39.633
39.911
40.190
40.468
40.748
48.510
90.806
106.863
109.492
117.807
125.261
137.532
142.301
151.465
154.123
154.928
156.755
160.628
180.353
193.928
ppm
0
50
100
150
200
NO2
MeN
O
O
O
HN
N
NH
H2N

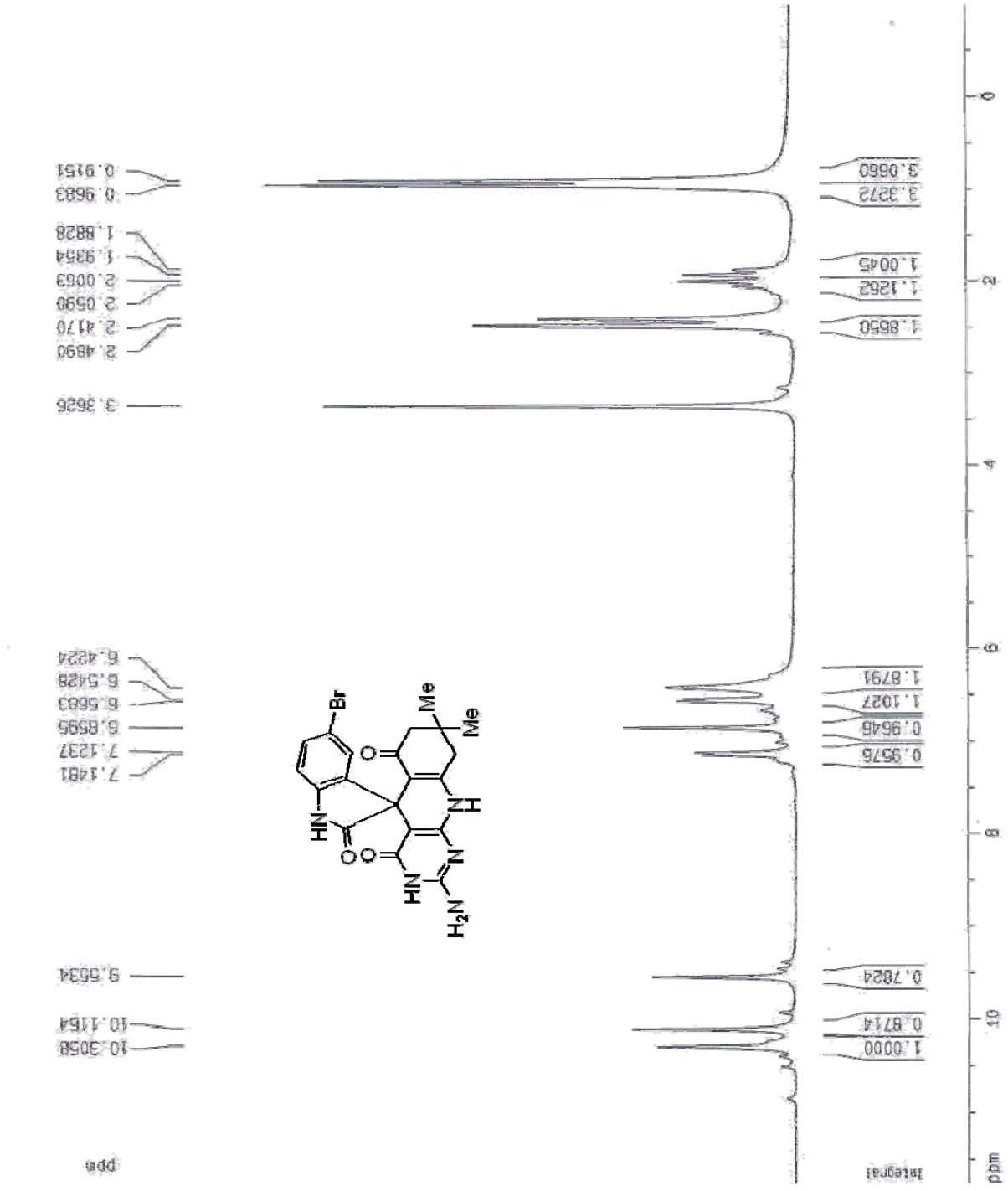
^{13}C NMR of **4b**



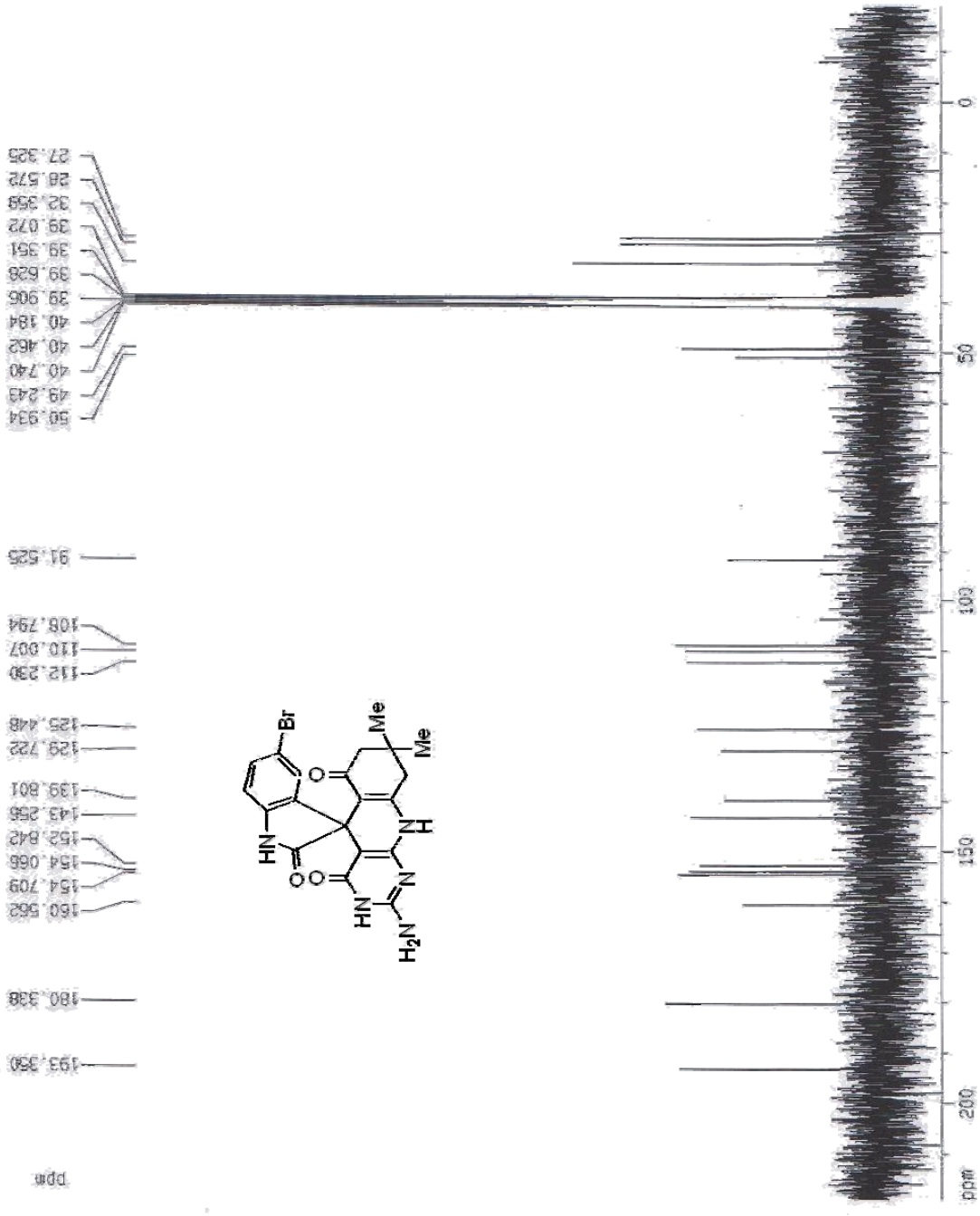
^{1}H NMR of **4c**


26.543
28.598
32.316
39.096
39.376
39.634
39.932
40.210
40.487
40.761
48.873
51.096
92.341
108.119
109.361
120.676
122.838
127.082
137.363
143.681
152.276
153.977
154.561
160.519
180.736
193.147
ppm
Me
Me
O
HN
NH
O
O
HN
N
H2N
0
50
100
150
200
ppm

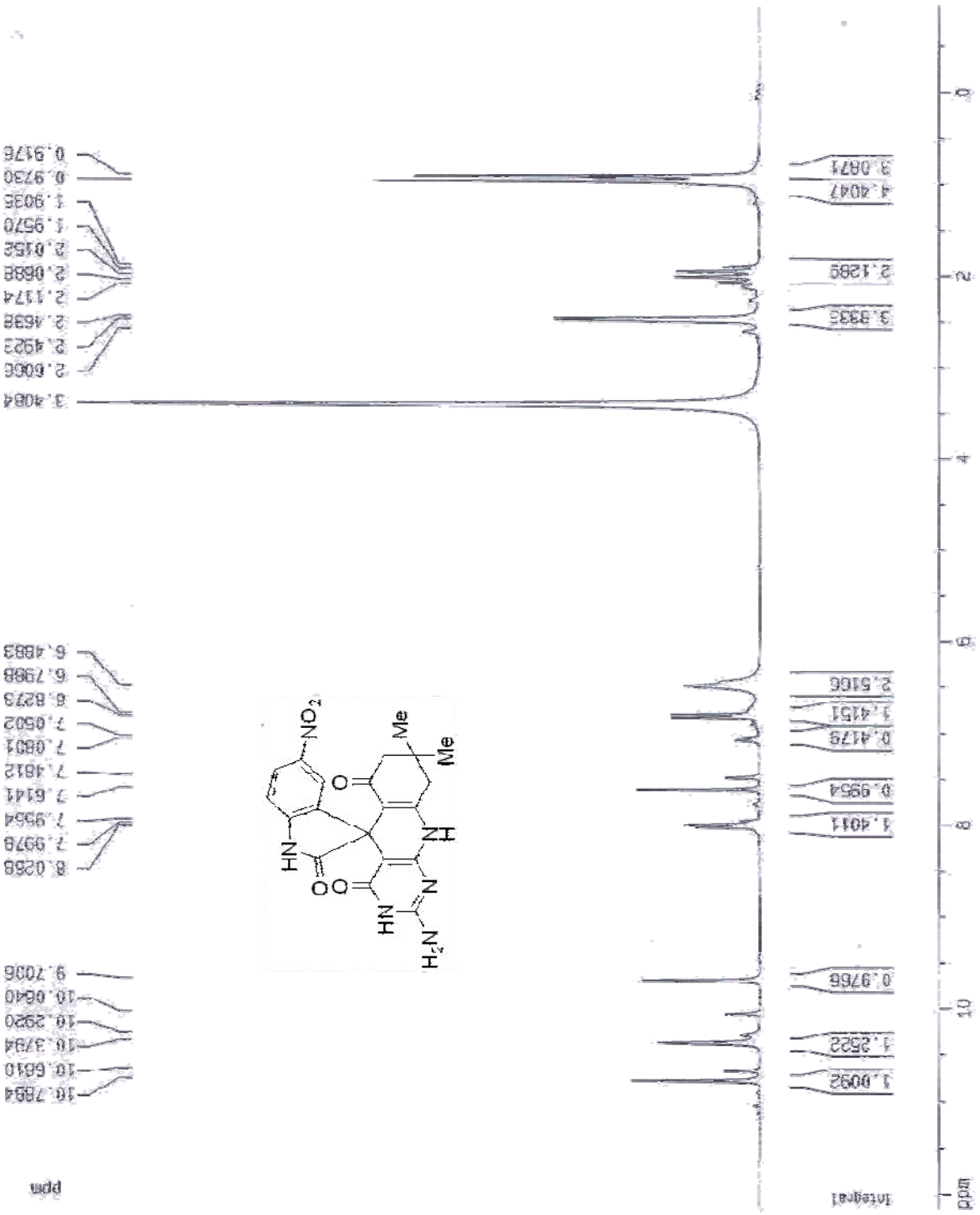
^{13}C NMR of **4c**


Br
Me
Me
O
NH
NH
O
O
HN
N
H_2N
0.9151
0.9683
1.8828
1.9354
2.0063
2.0590
2.4170
2.4890
3.3626
6.4224
6.5428
6.5663
6.8595
7.1237
7.1481
9.5534
10.1154
10.3058
3.0660
3.3272
1.0045
1.1262
1.8550
1.8791
1.1027
0.9646
0.9576
0.7824
0.8714
1.0000
integral
ppm

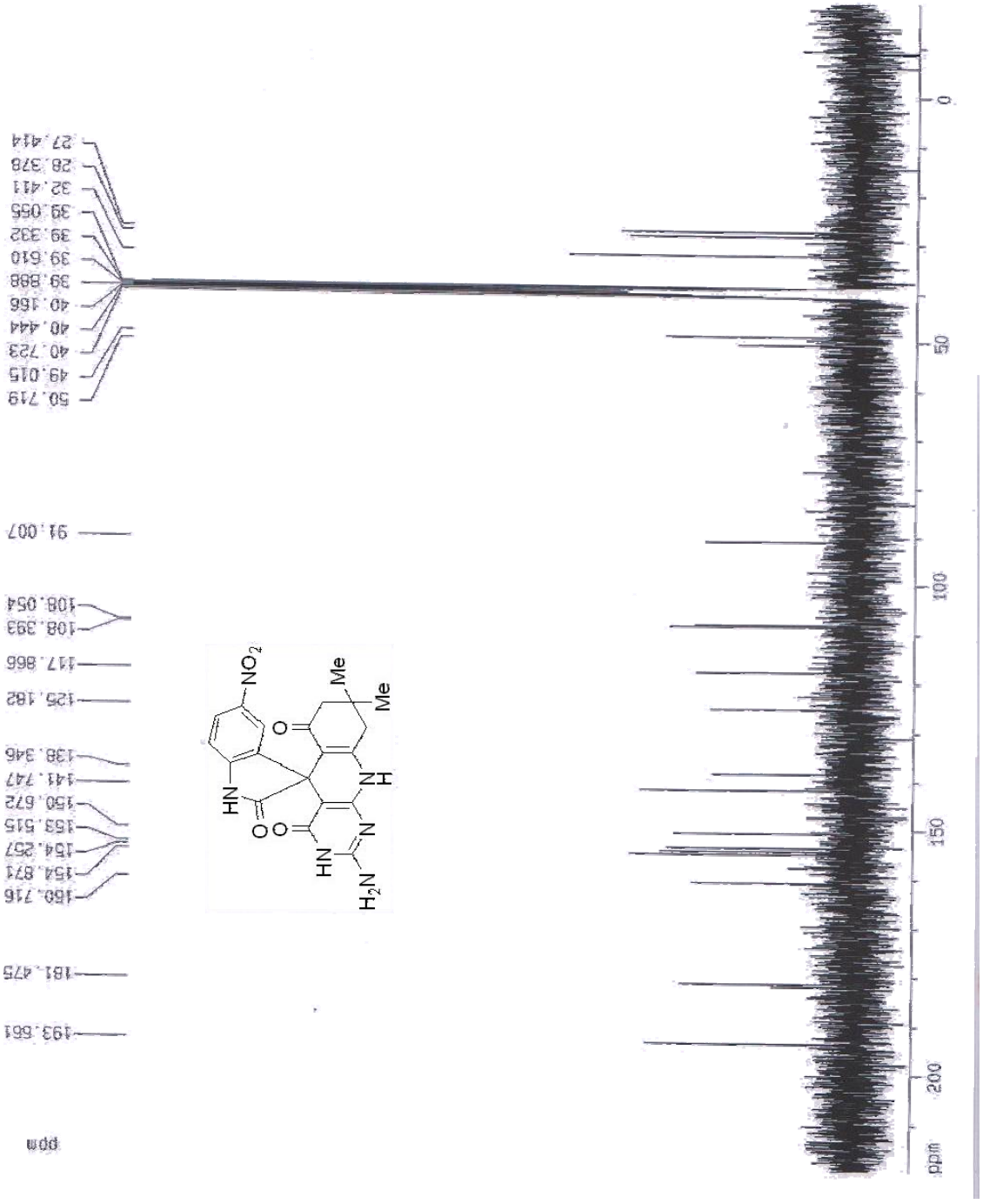
^{1}H NMR of **4d**


27.325
28.572
32.359
39.072
39.351
39.628
39.906
40.184
40.462
40.740
49.243
50.934
91.525
108.794
110.007
112.230
125.448
129.722
139.801
143.256
152.842
154.066
154.709
160.562
180.338
193.350
ppm
Br
Me
Me
O
O
O
HN
HN
N
H
N
H2N
0
50
100
150
200
ppm

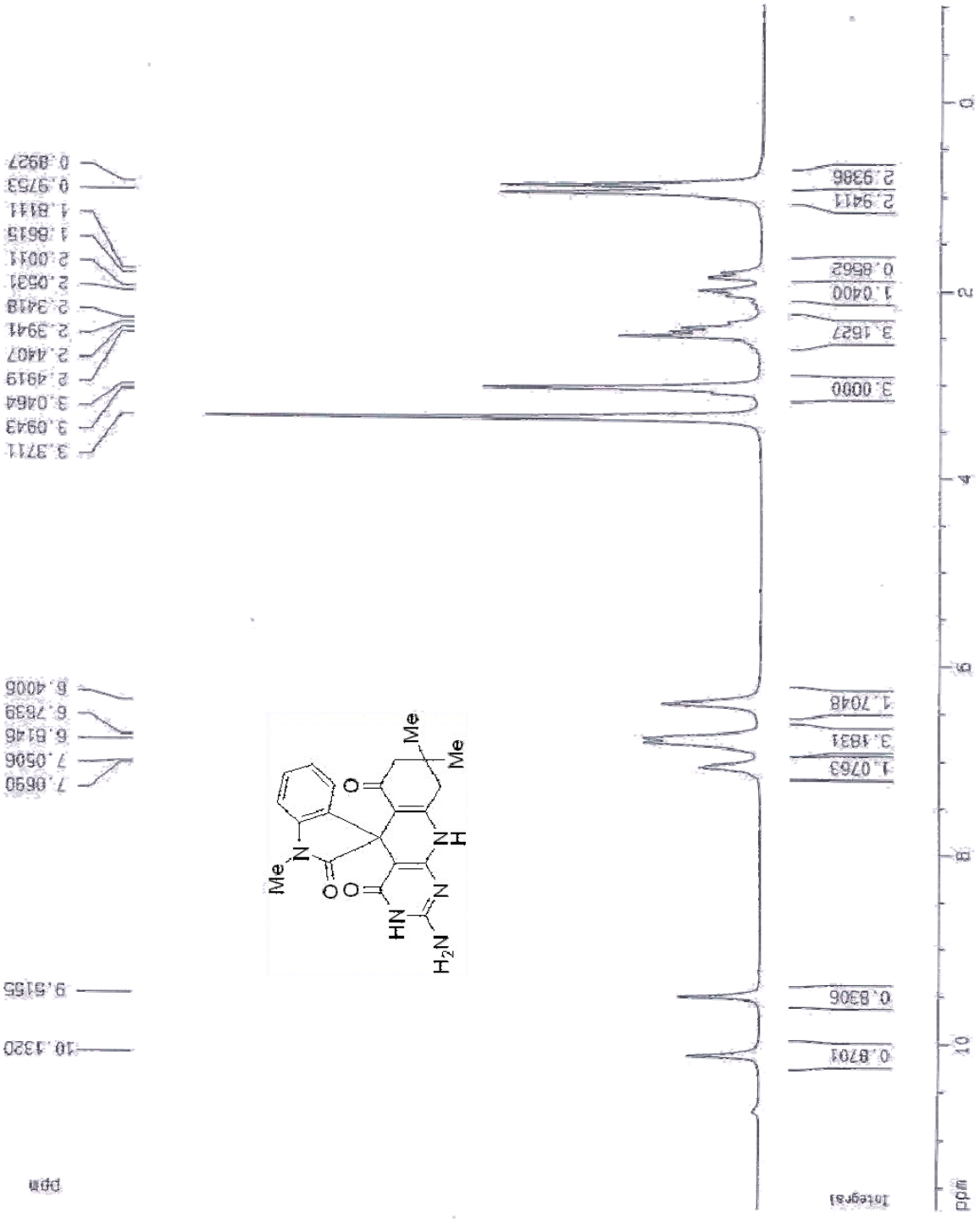
^{13}C NMR of **4d**


ppm
Integral
0
2
4
6
8
10
3.0871
4.4047
2.1285
3.8333
2.5166
1.4151
0.4179
0.9954
1.4011
0.9766
1.2522
1.0092
9.7036
10.0640
10.2920
10.3794
10.6010
10.7884
8.0258
7.9978
7.9564
7.6141
7.4812
7.0801
7.0502
6.8273
6.7988
6.4883
3.4084
2.6066
2.4923
2.4638
2.1174
2.0688
2.0152
1.9570
1.9035
0.9730
0.9376

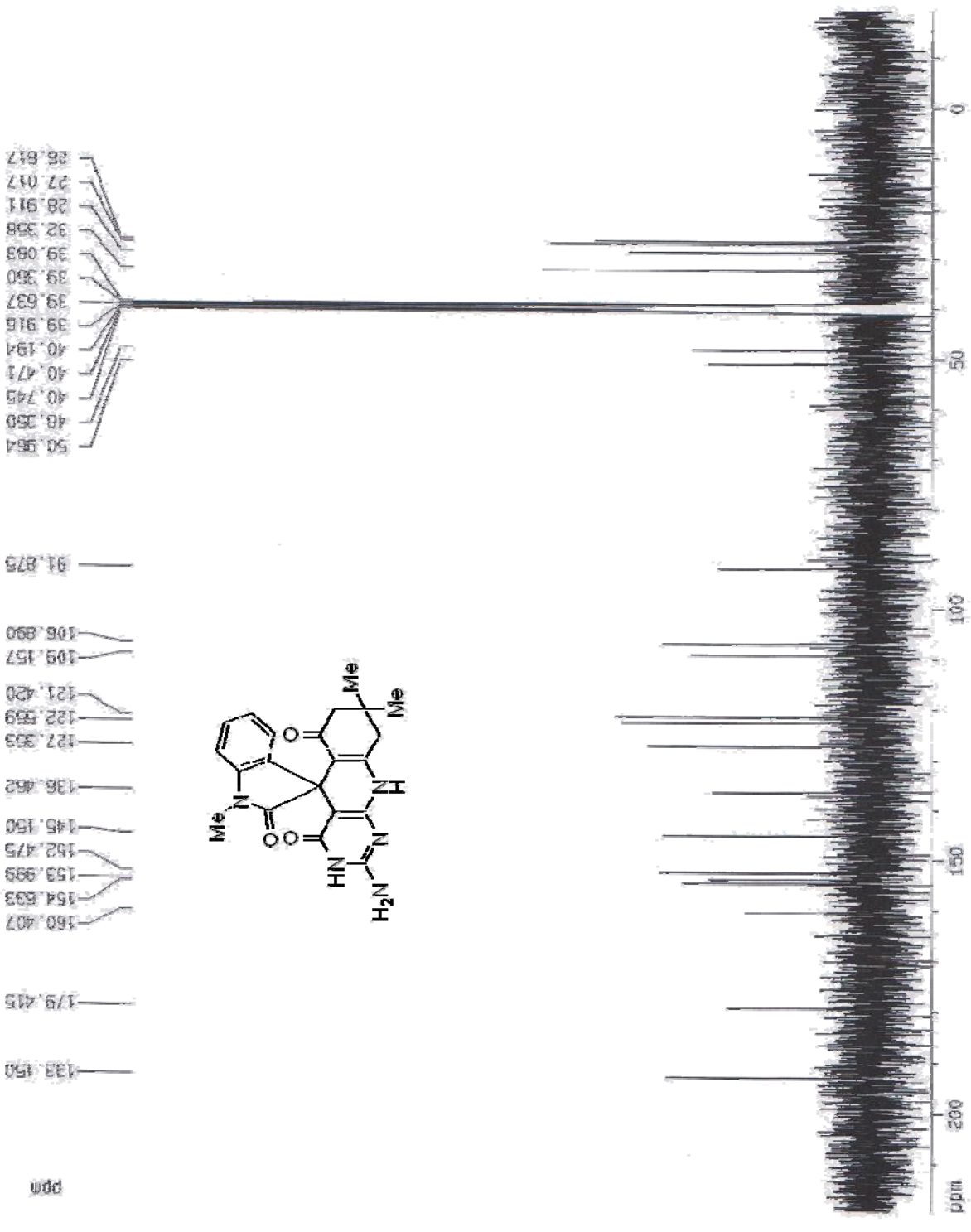
^{1}H NMR of **4e**


27.414
28.378
32.411
39.055
39.332
39.610
39.888
40.166
40.444
40.723
49.015
50.719
91.007
108.054
108.393
117.866
125.182
138.346
141.747
150.672
153.515
154.257
154.871
160.716
181.475
193.661
ppm
0
50
100
150
200
ppm
NO2
Me
Me
O
HN
O
O
HN
N
H
N
H2N

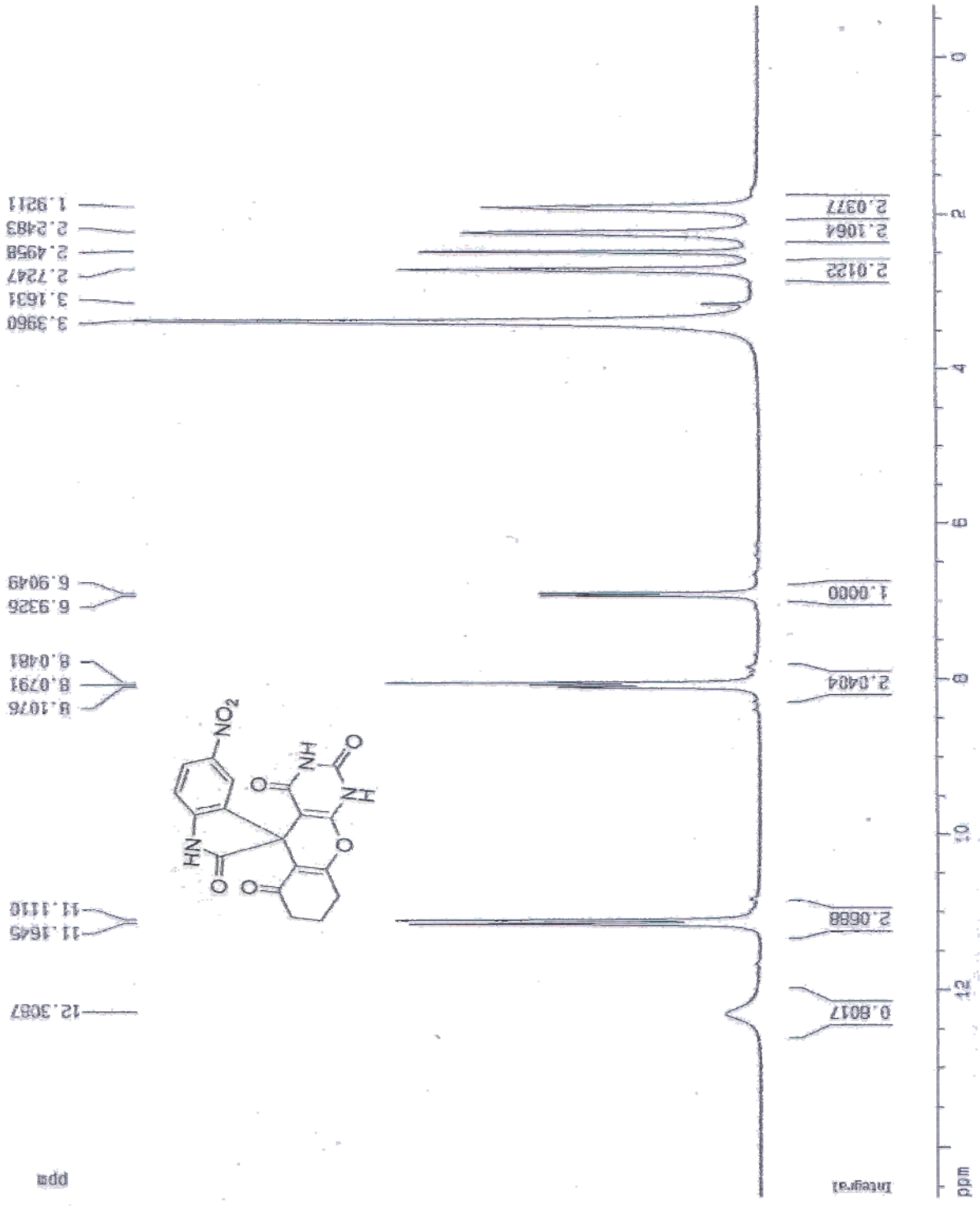
^{13}C NMR of **4e**


Me
Me
O
N
H
Me
N
O
O
N
HN
H_2N
0.8927
0.9753
1.8141
1.8615
2.0011
2.0531
2.3418
2.3941
2.4407
2.4919
3.0464
3.0943
3.3711
6.4005
6.7639
6.8146
7.0506
7.0690
9.5155
10.1320
2.9386
2.9411
0.8562
1.0400
3.1627
3.0000
1.7048
3.1831
1.0763
0.8306
0.8701
ppm
Integral

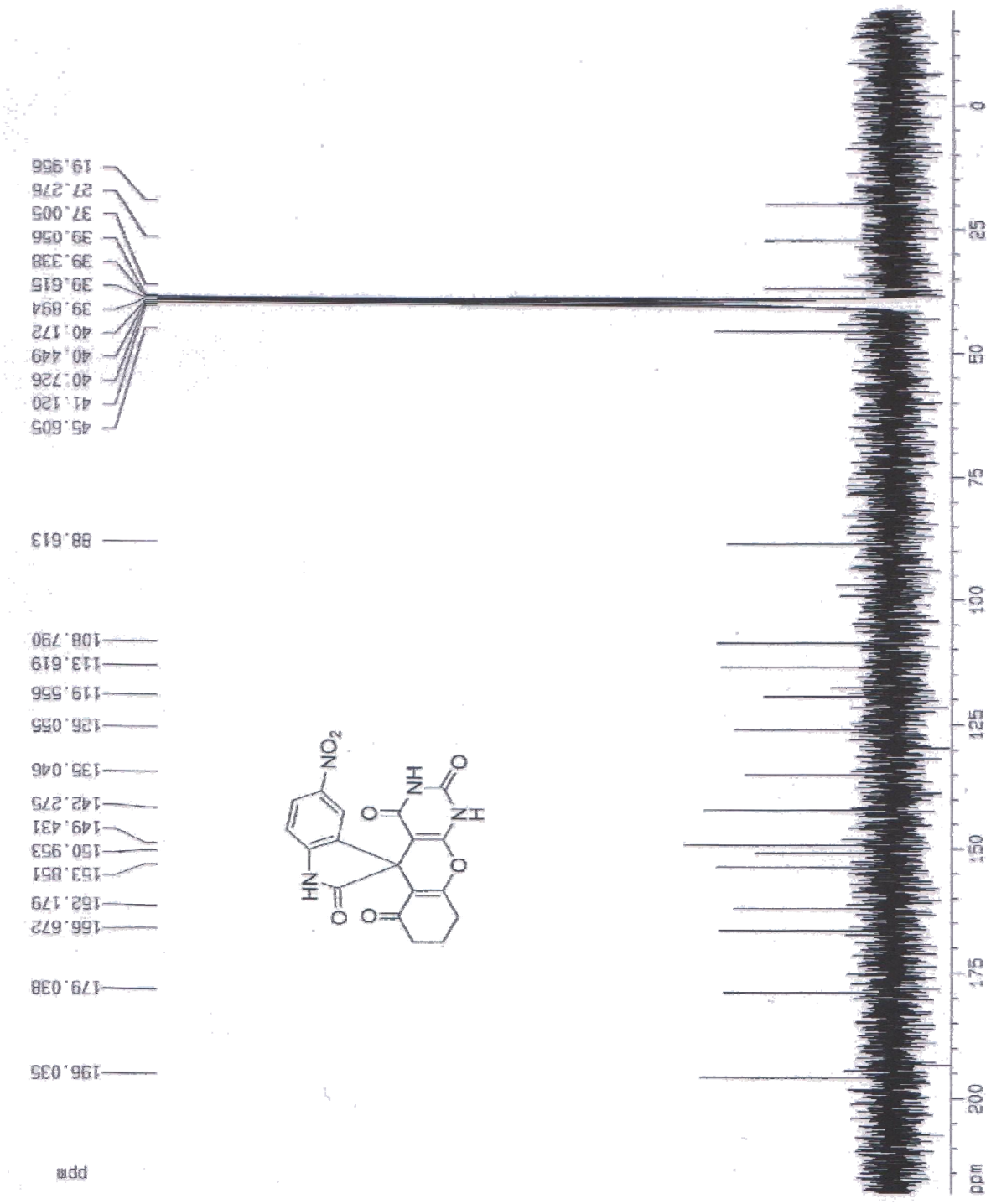
^{1}H NMR of **4f**


26.817
27.017
28.911
32.358
39.083
39.360
39.637
39.916
40.194
40.471
40.745
48.350
50.964
91.875
106.890
109.157
121.420
122.559
127.353
136.462
145.150
152.475
153.989
154.533
160.407
179.415
193.150
ppm
0
50
100
150
200
Me
Me
Me
O
O
O
N
N
H
N
HN
H2N

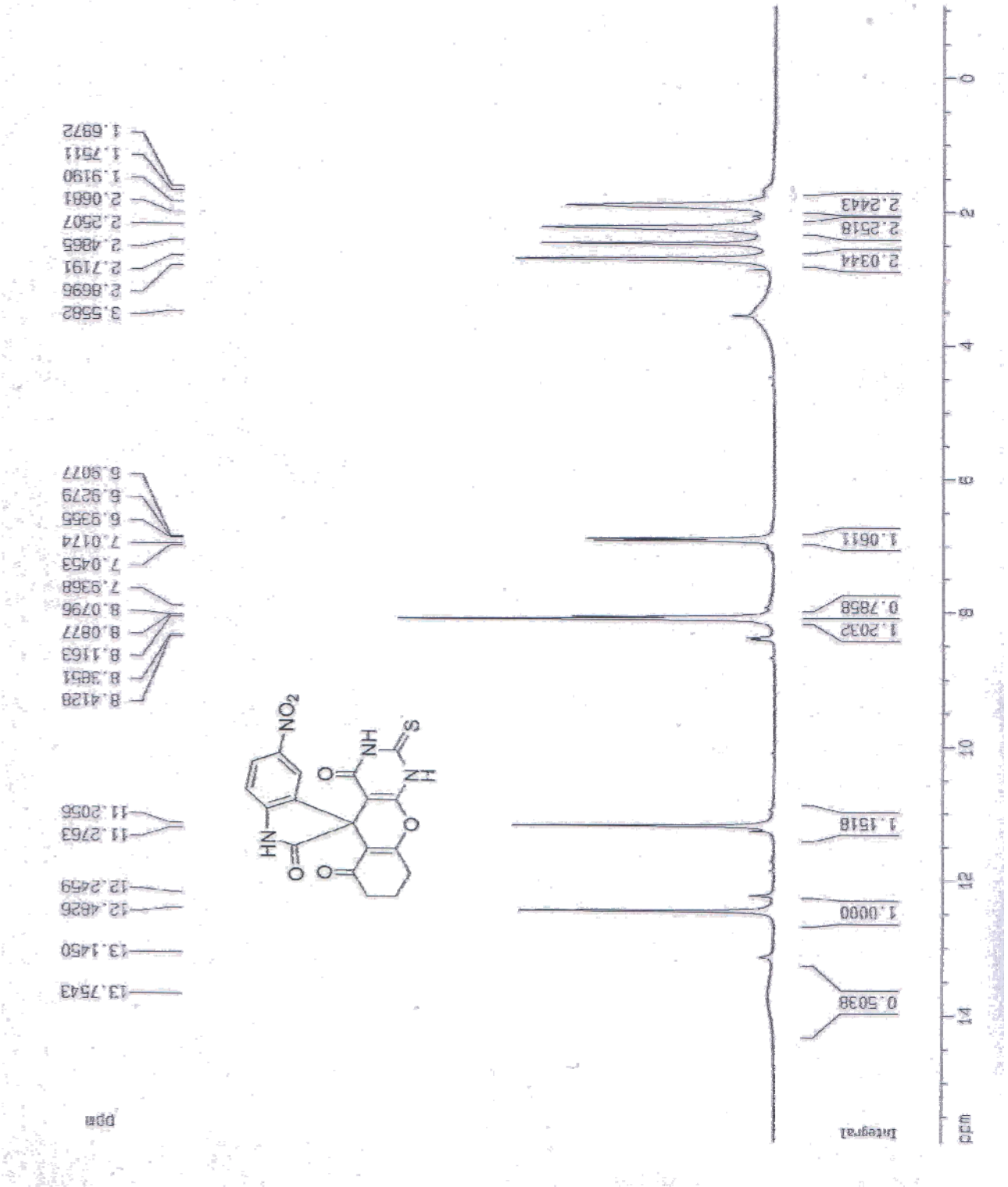
^{13}C NMR of **4f**


ppm
Integral
12.3087
11.1118
11.1645
8.1076
8.0791
8.0481
6.9326
6.9049
3.3960
3.1631
2.7247
2.4958
2.4833
1.9211
0.8017
2.0688
2.0404
1.0000
2.0122
2.1064
2.0377

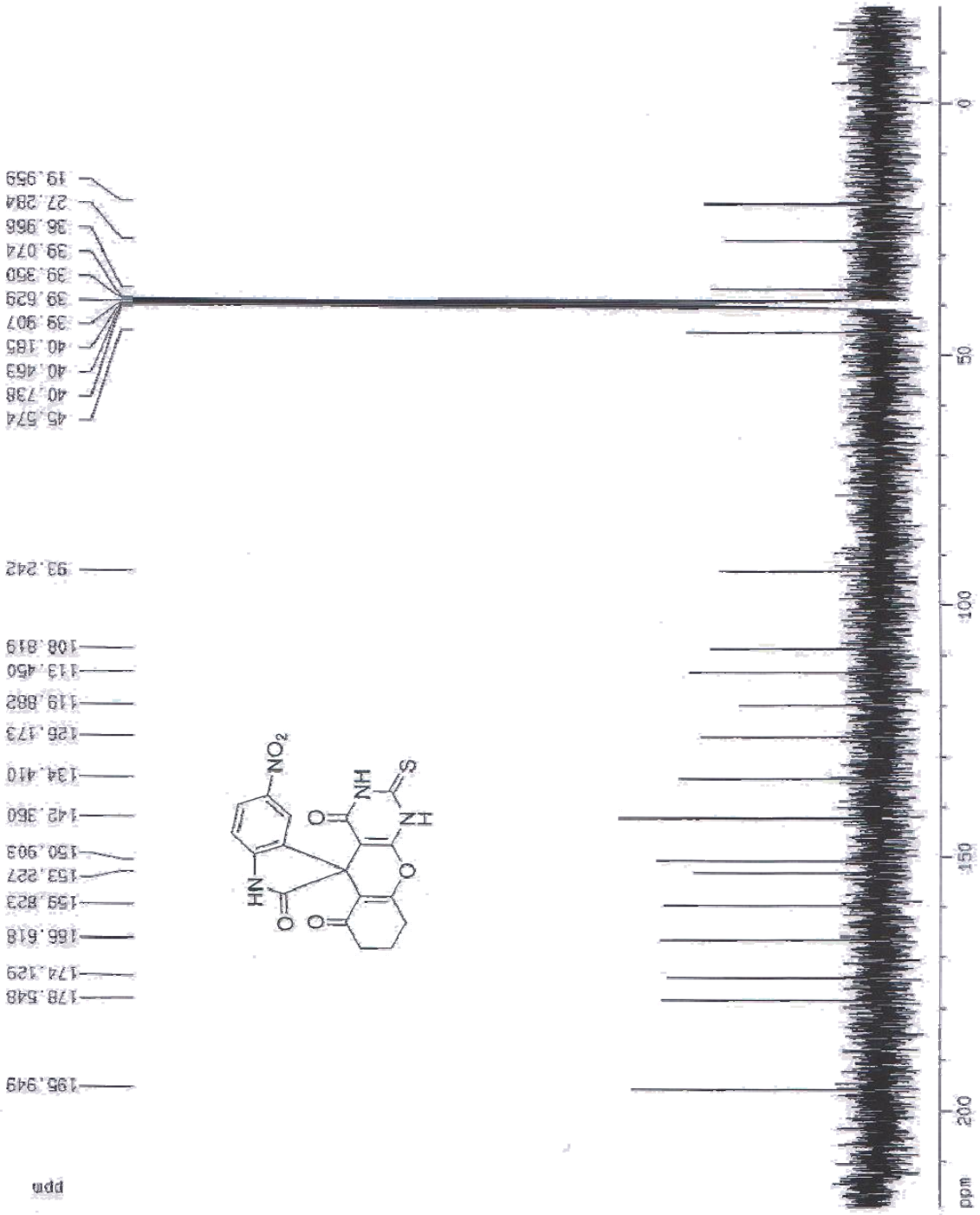
^{1}H NMR of **8a**


19.956
27.276
37.005
39.056
39.338
39.615
39.894
40.172
40.449
40.726
41.120
45.605
88.613
108.790
113.619
119.556
126.055
135.046
142.275
149.431
150.953
153.851
162.179
166.672
179.038
196.035
ppm
0
25
50
75
100
125
150
175
200
NO2
NH
O
HN

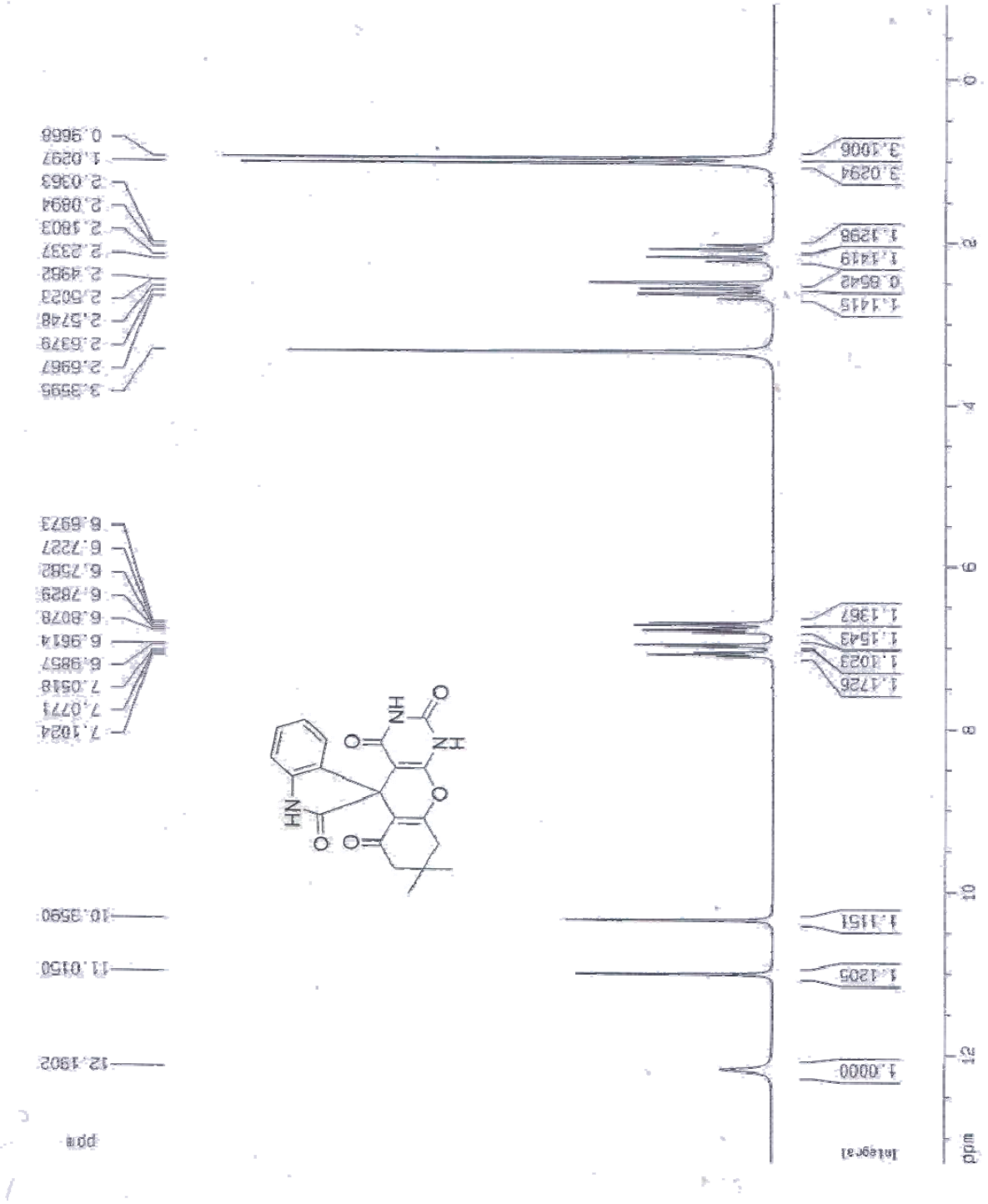
^{13}C NMR of **8a**



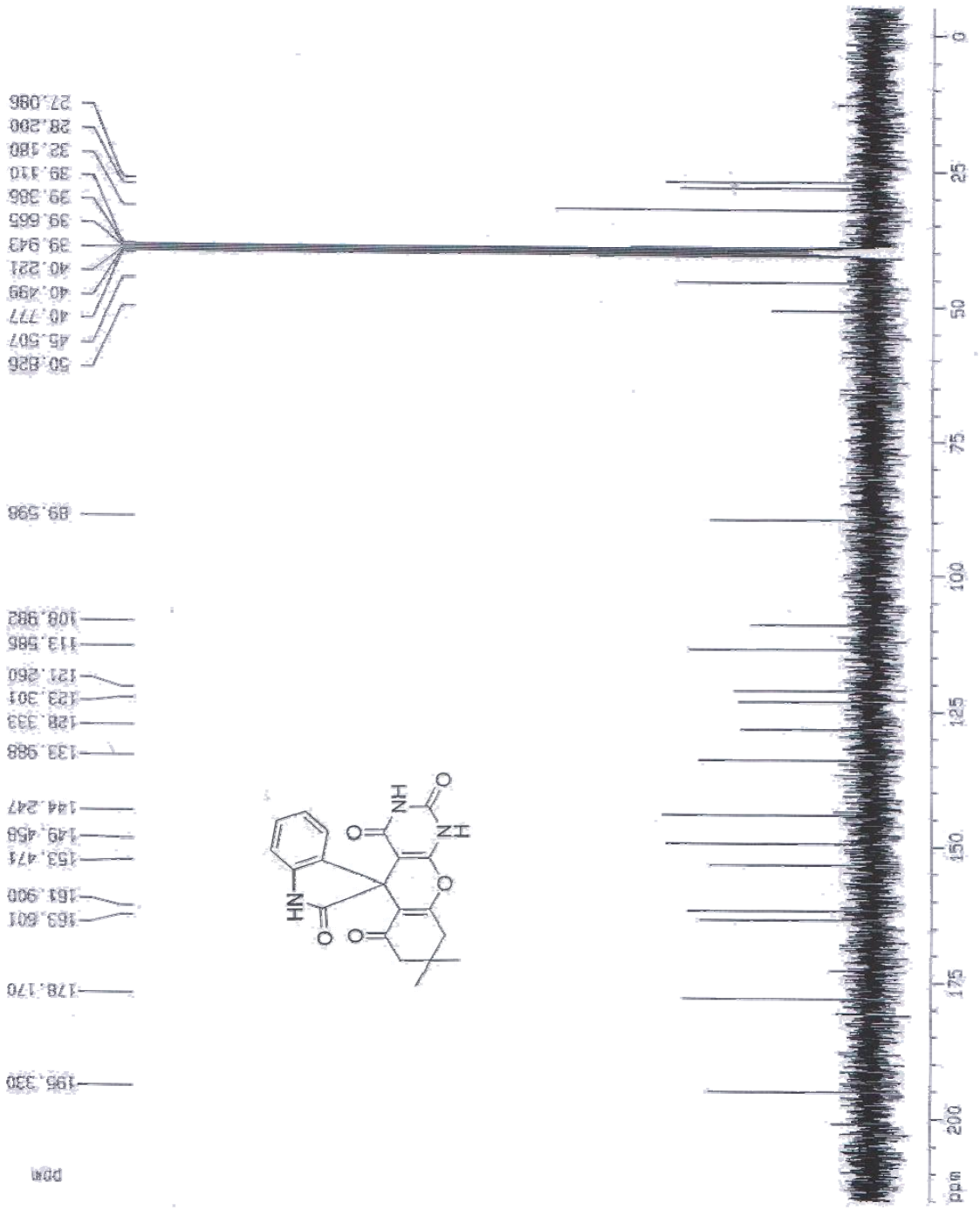
^{1}H NMR of **8b**


19.959
27.284
36.966
39.074
39.350
39.629
39.907
40.185
40.463
40.738
45.574
93.242
108.819
113.450
119.882
126.173
134.410
142.360
150.903
153.227
159.823
166.618
174.129
178.548
195.949
ppm
0
50
100
150
200
NO2
NH
S
HN
O
HN
O
O
O

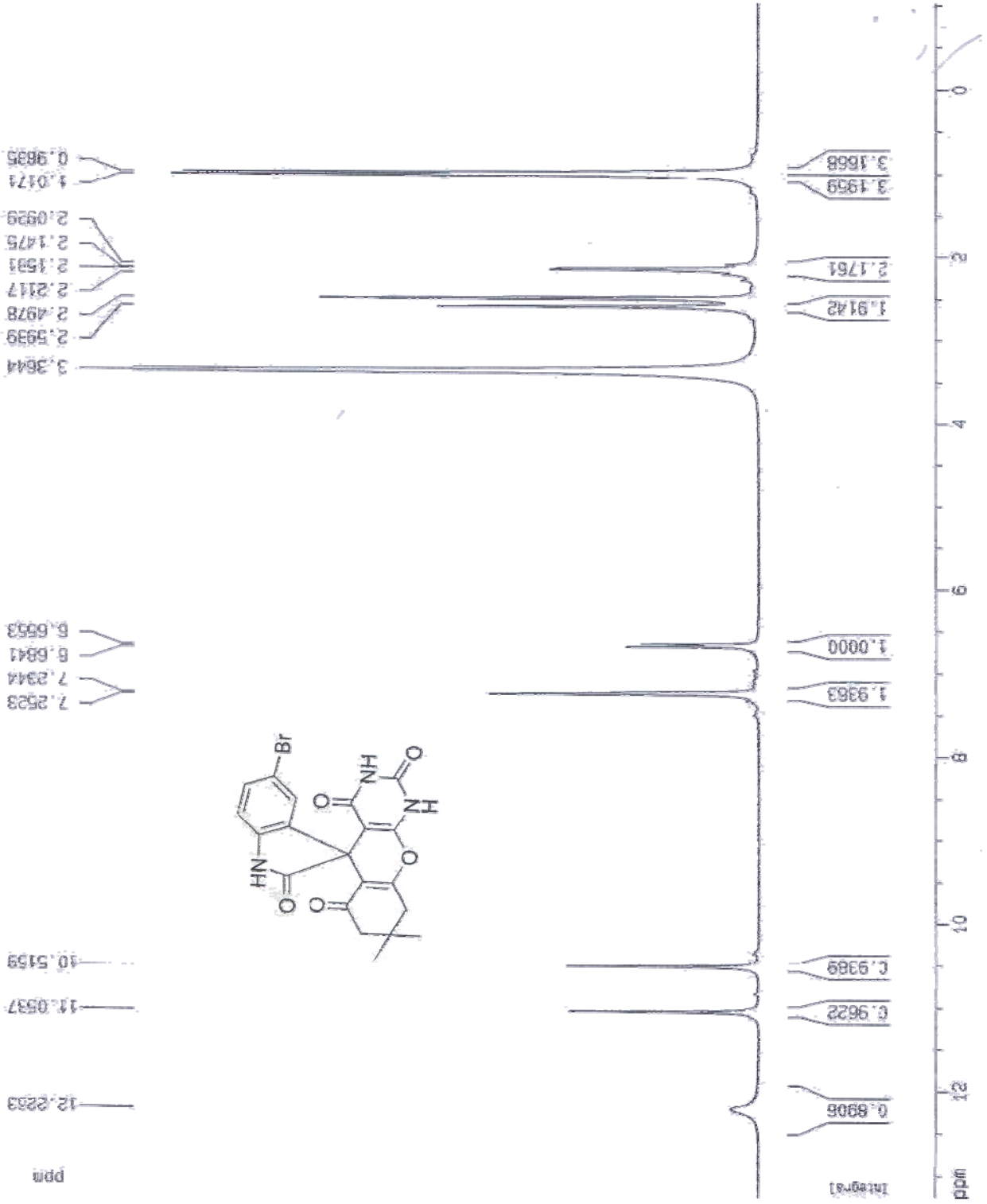
^{13}C NMR of **8b**



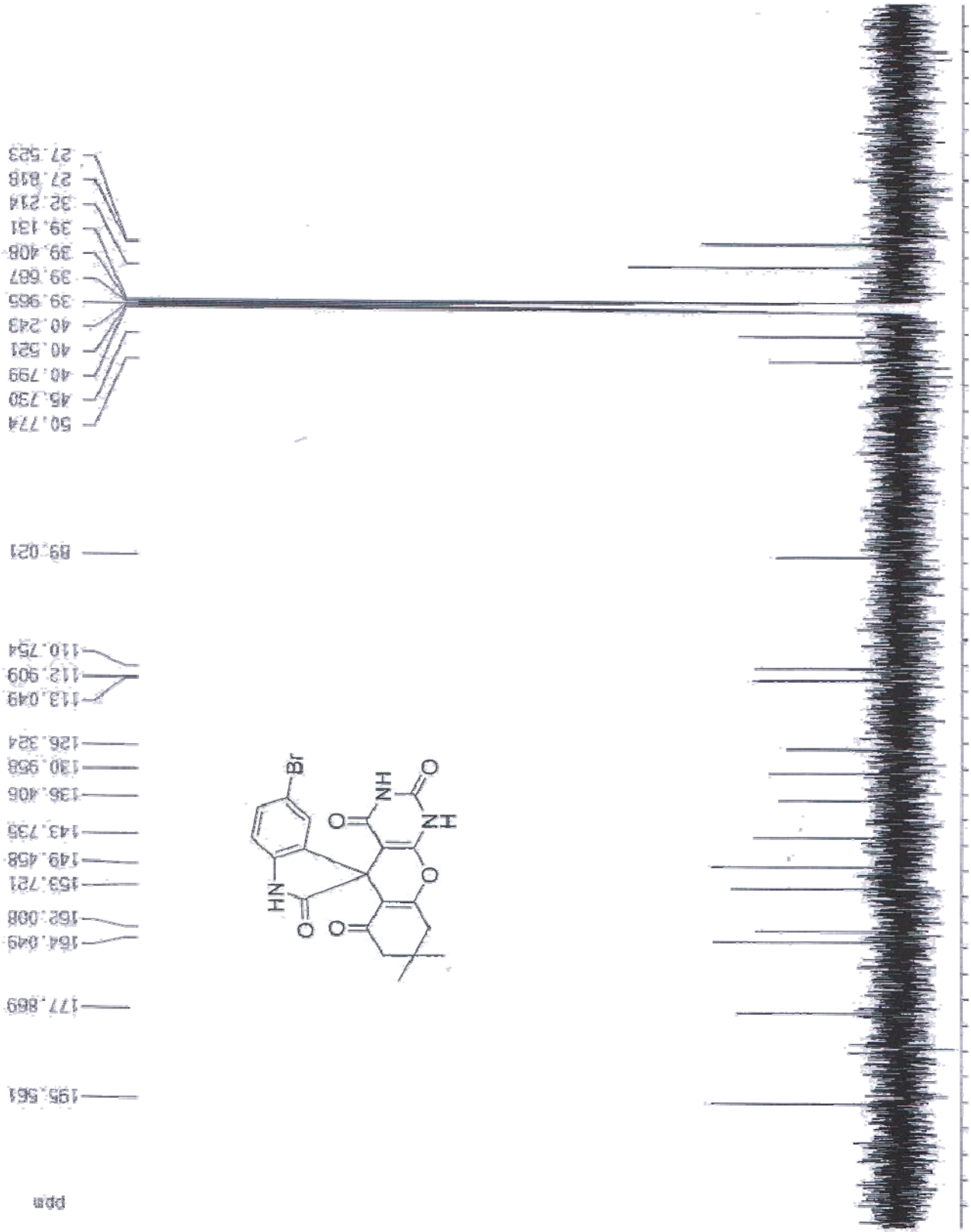
^{1}H NMR of 8c


27.086
28.200
32.180
39.110
39.366
39.665
39.943
40.221
40.499
40.777
45.507
50.826
89.598
108.982
113.586
121.260
123.301
128.333
133.988
144.247
149.458
153.474
161.900
163.601
178.170
195.330
ppm
0
25
50
75
100
125
150
175
200
NH
O
HN
O
O
NH
O

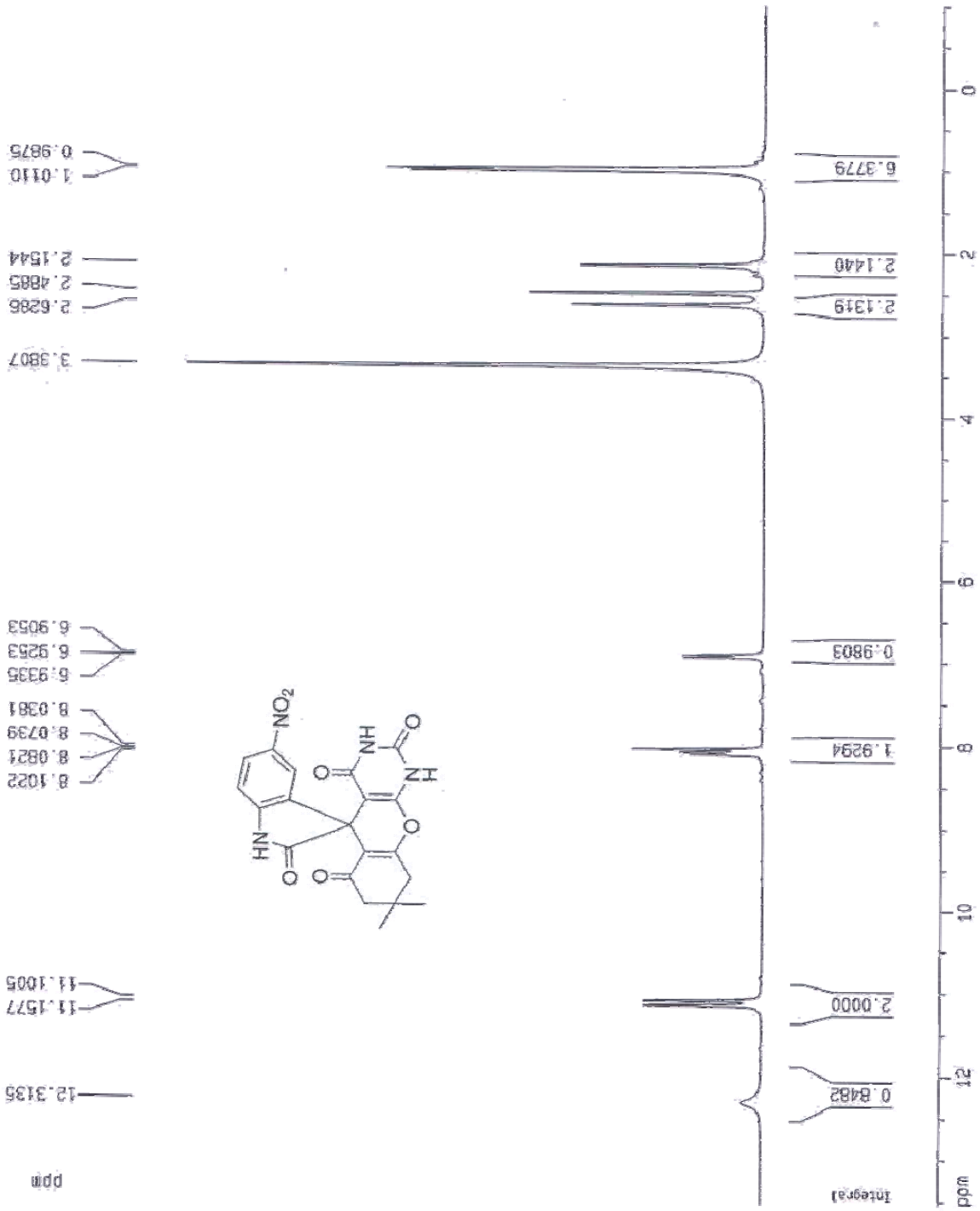
^{13}C NMR of **8c**


0.9835
1.0171
2.0929
2.1475
2.1581
2.2117
2.4978
2.5939
3.3644
7.2523
7.2344
6.8841
6.8653
10.5159
11.0537
12.2253
ppm
3.1568
3.1959
2.1761
1.9142
1.0000
1.9363
0.9306
0.9622
0.8906
Integral
Br
HN
O
NH
O
O
HN
O
O
0
2
4
6
8
10
12
ppm

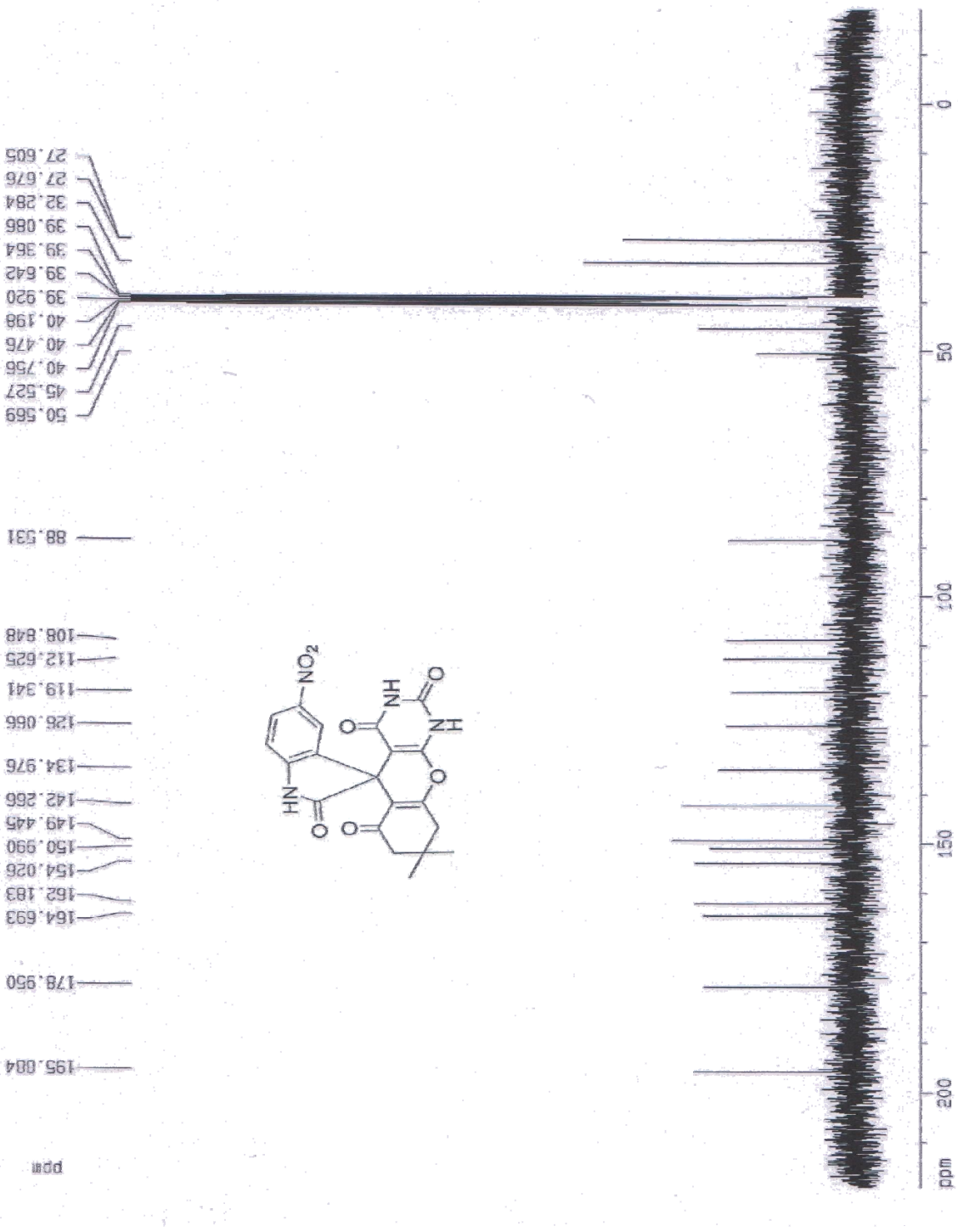
^{1}H NMR of **8d**


27.523
27.818
32.214
39.131
39.408
39.687
39.965
40.243
40.521
40.799
45.730
50.774
89.021
110.754
112.909
113.049
126.324
130.958
136.406
143.735
149.458
153.721
162.008
164.049
177.869
195.561
ppm
Br
NH
O
NH
O
O
NH
O
O

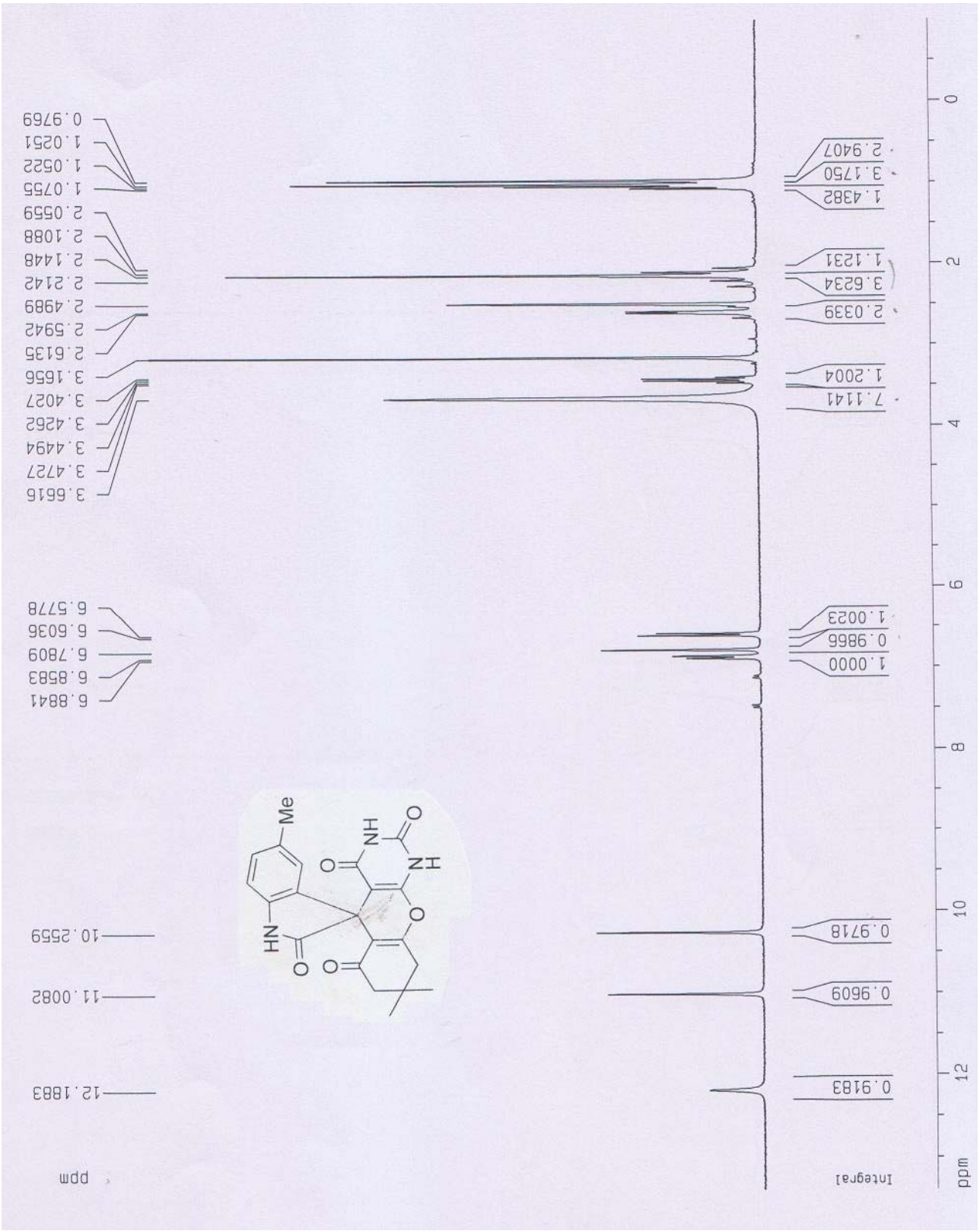
^{13}C NMR of **8d**


0.9875
1.0110
2.1544
2.4885
2.6286
3.3807
6.9053
6.9253
6.9335
8.0381
8.0739
8.0821
8.1022
11.1005
11.1577
12.3135
6.3779
2.1440
2.1319
0.9803
1.9294
2.0000
0.8482
ppm
Integral
NO2
NH
O
HN

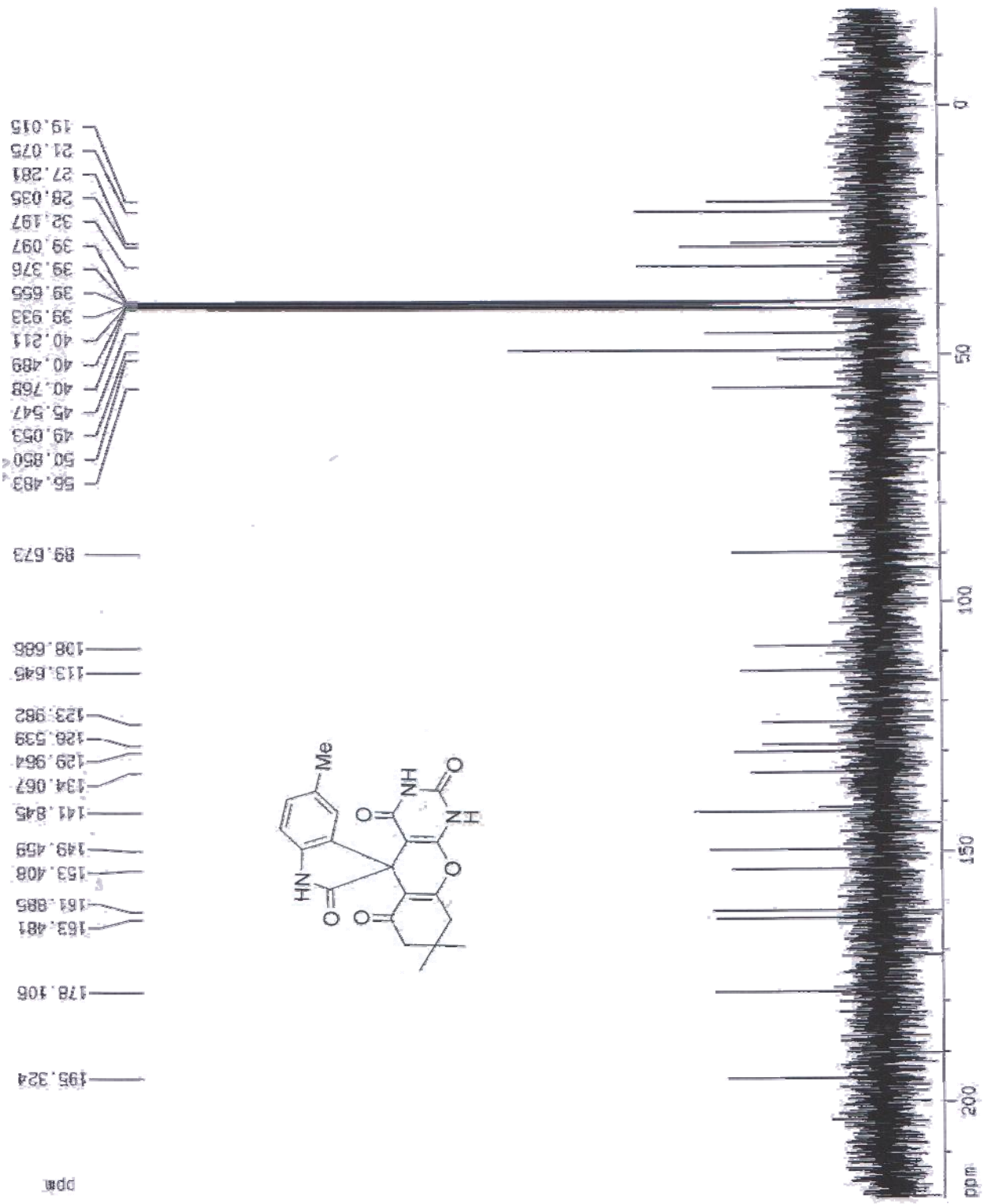
^{1}H NMR of **8e**


27.605
27.676
32.284
39.086
39.364
39.642
39.920
40.198
40.476
40.756
45.527
50.569
88.531
108.848
112.625
119.341
126.066
134.976
142.266
149.445
150.990
154.026
162.183
164.693
178.950
195.004
ppm
0
50
100
150
200
NO2
NH
O
HN

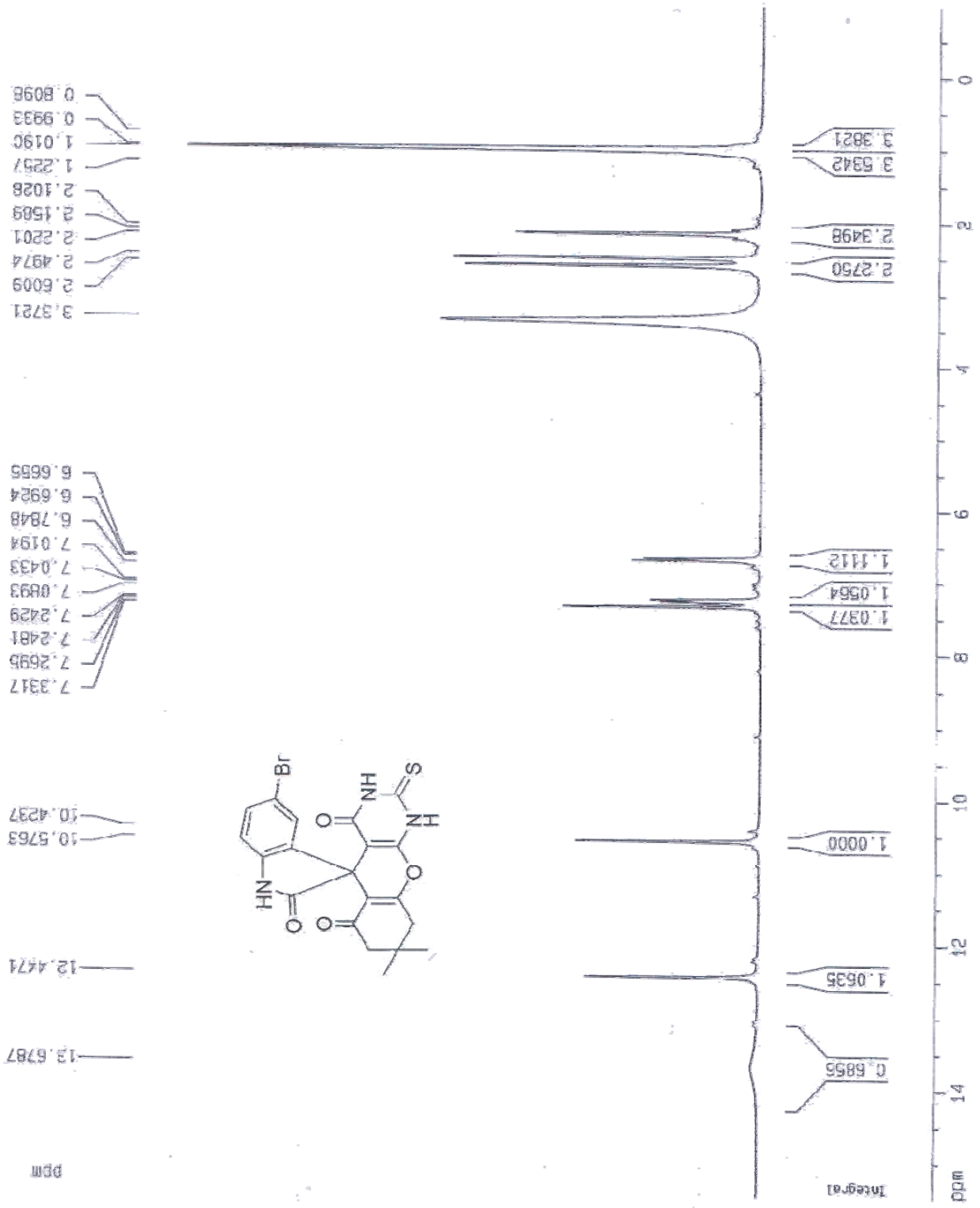
^{13}C NMR of **8e**


ppm
12.1883
11.0082
10.2559
6.8841
6.8583
6.7809
6.6036
6.5778
3.6616
3.4727
3.4494
3.4262
3.4027
3.1656
2.6135
2.5942
2.4989
2.2142
2.1448
2.1088
2.0559
1.0755
1.0522
1.0251
0.9769
Integral
0.9183
0.9609
0.9718
1.0000
0.9866
1.0023
7.1141
1.2004
2.0339
3.6234
1.1231
3.1750
1.4382
2.9407
Me
NH
O
NH
O
O
NH
O
O
O
ppm

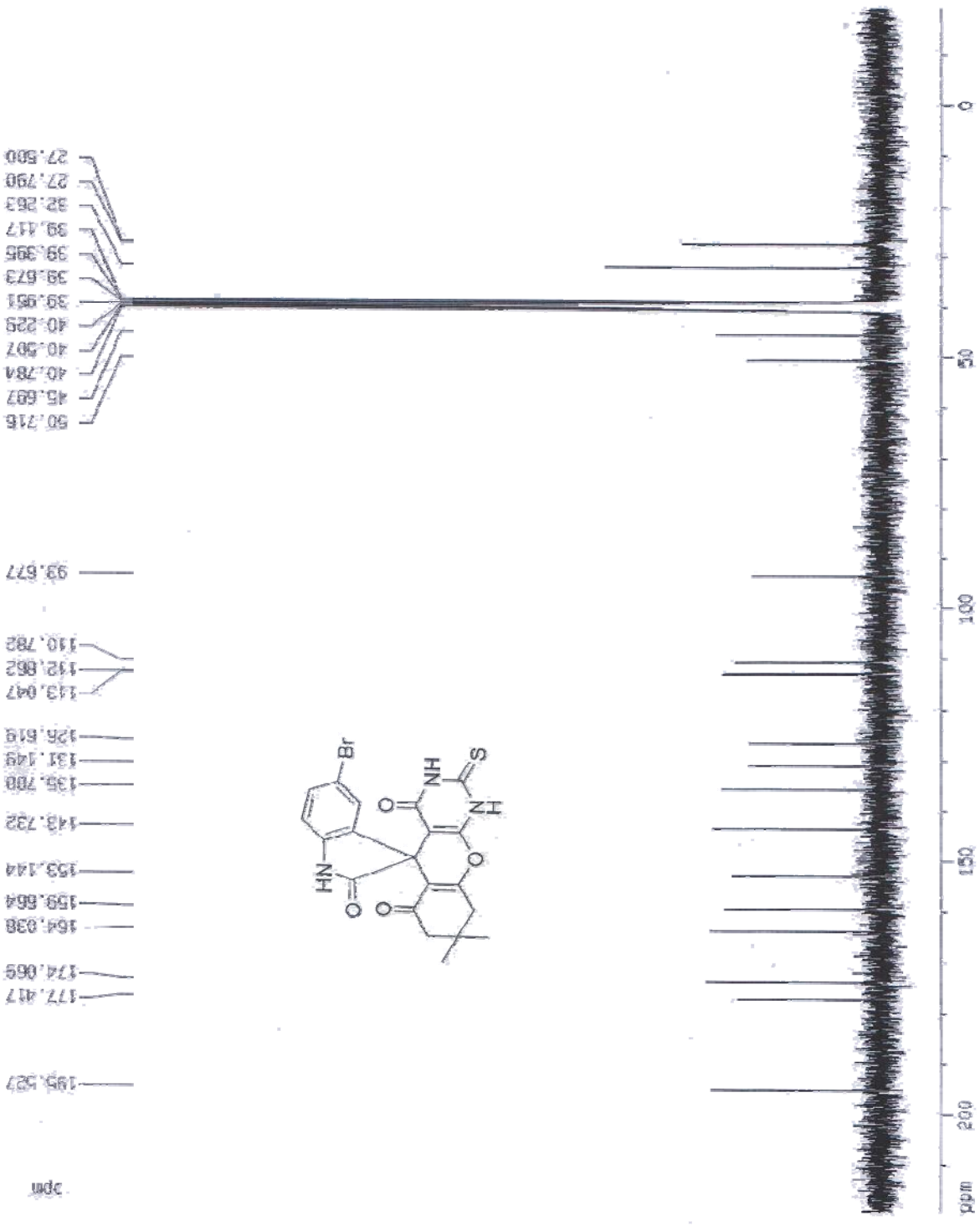
^{1}H NMR of **8f**


19.015
21.075
27.281
28.035
32.197
39.097
39.376
39.655
39.933
40.211
40.489
40.768
45.547
49.053
50.850
56.483
89.673
108.686
113.645
123.982
126.539
129.964
134.067
141.845
149.459
153.408
161.885
163.481
178.105
195.324
ppm
0
50
100
150
200
Me
NH
O
HN
O
O
O

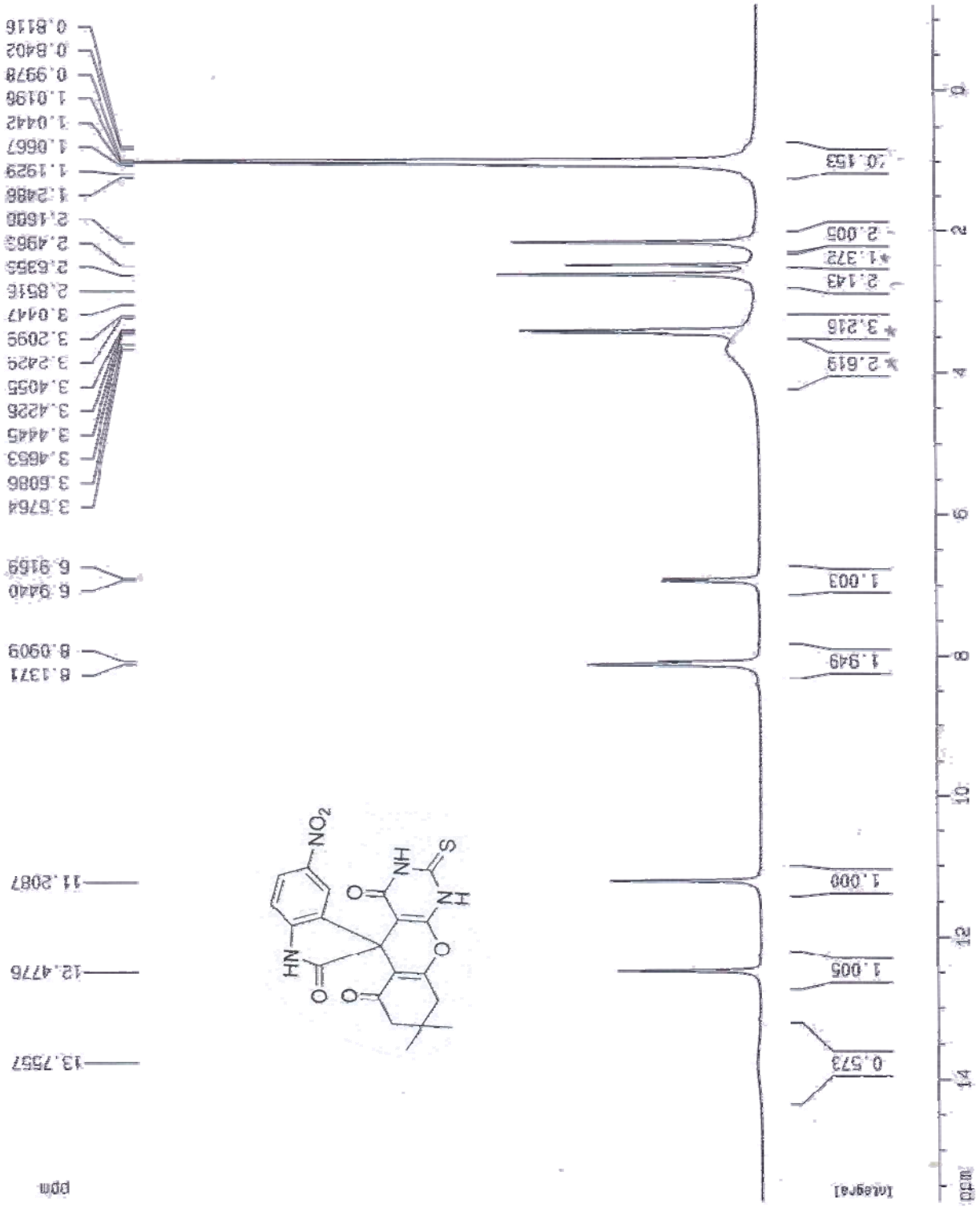
^{13}C NMR of **8f**


0.8098
0.9933
1.0190
1.2257
2.1028
2.1509
2.2201
2.4974
2.6009
3.3721
7.3317
7.2695
7.2481
7.2429
7.0893
7.0433
7.0194
6.7848
6.6924
6.6655
10.4237
10.5763
12.4474
13.6787
PPM
3.3821
3.5342
2.3498
2.2750
1.1412
1.0564
1.0377
1.0000
1.0635
0.6855
Integral
0
2
4
6
8
10
12
14
ppm
Br
NH
S
NH
O
O
NH
O
O

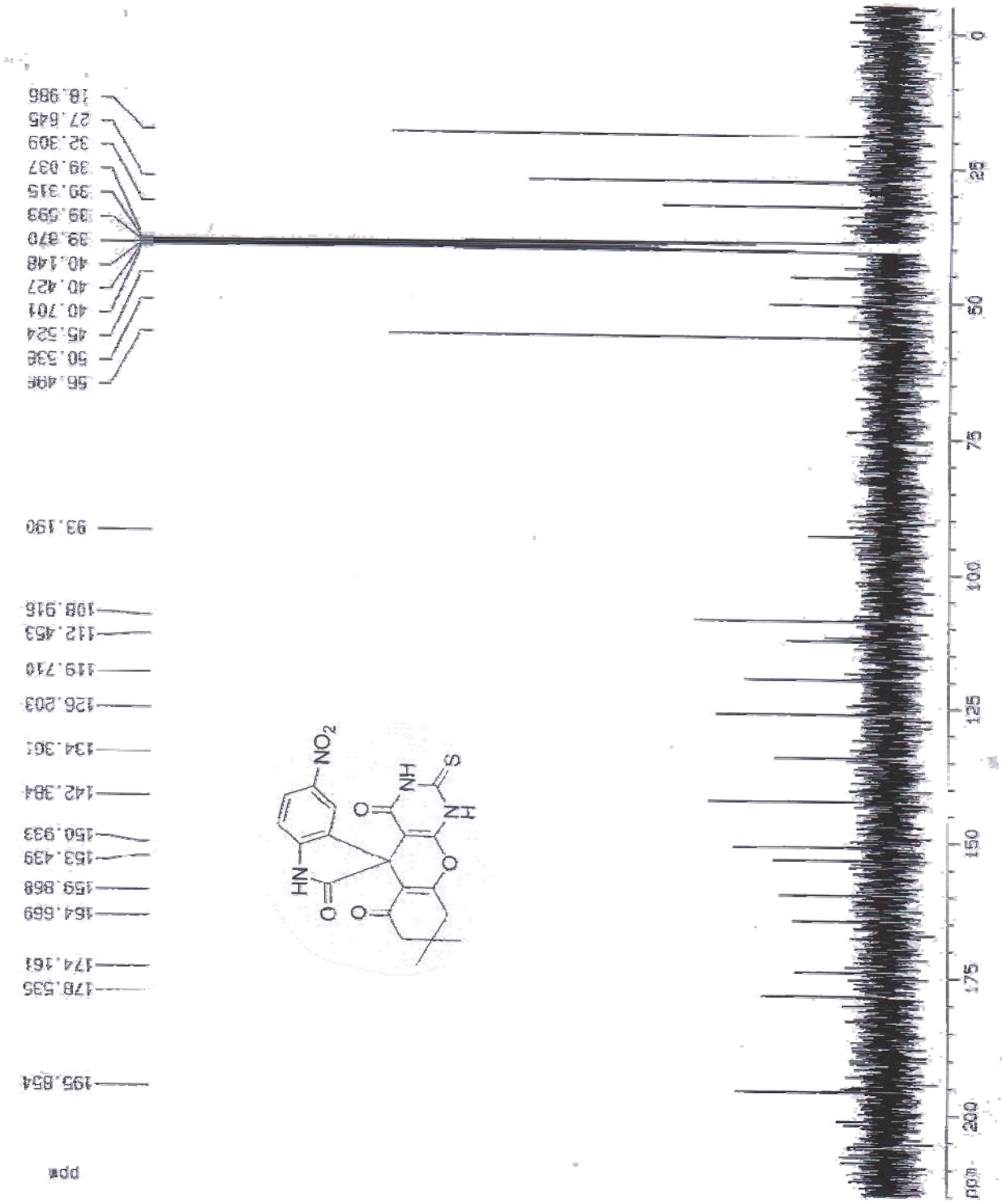
^{1}H NMR of **8g**


27.500
27.790
32.263
39.417
39.395
39.673
39.951
40.229
40.507
40.784
45.687
50.716
93.677
110.782
112.862
113.047
125.619
131.149
135.788
143.732
153.144
159.664
164.038
174.069
177.417
195.527
Br
NH
S
NH
O
NH
O
O
O
0
50
100
150
200
ppm

^{13}C NMR of **8g**


0.8116
0.8402
0.9978
1.0196
1.0442
1.0667
1.1929
1.2488
2.1688
2.4963
2.6355
2.8516
3.0447
3.2099
3.2429
3.4055
3.4226
3.4445
3.4653
3.5086
3.5764
6.9169
6.9440
8.0909
8.1371
11.2087
12.4776
13.7557
ppm
20.153
2.005
1.372
2.143
3.216
2.619
1.003
1.949
1.000
1.005
0.573
Integral
NO2
NH
S
NH
O
O
O
NH
O

^{1}H NMR of **8h**


18.986
27.645
32.309
39.037
39.315
39.599
39.870
40.148
40.427
40.701
45.524
50.536
56.496
93.190
108.916
112.453
119.710
126.203
134.361
142.384
150.933
153.439
159.868
164.669
174.161
178.535
195.854
ppm
0
25
50
75
100
125
150
175
200
NO2
NH
S
N
H
O
HN
O
O

^{13}C NMR of **8h**