Electronics supplementary information

Nanodomain cubic cuprous oxide as reusable catalyst in one-pot synthesis of 3-alkyl/aryl-3-(pyrrole-2-yl)-2-phenyl-2,3-dihydro-isoindolinones and 3-alkyl/aryl-3-(indole-3-yl)-2-phenyl-2,3-dihydro-isoindolinones in aqueous medium

Swarbhanu Sarkar,[a,$] Nivedita Chatterjee,[a,$] Manas Roy,[b,$] Rammyani Pal,[a,$] Sabyasachi Sarkar,[c] Asish Kumar Sen,[a,*]

[a]*Chemistry Division, CSIR-Indian Institute of Chemical Biology, 4 Raja S. C. Mullick Road, Jadavpur, Kolkata-700 032, India.*

[b]*Department of Chemistry, Indian Institute of Technology, Kanpur-208016, India.*

[c]*Department of Chemistry, Bengal Engineering and Science University, Shibpur, Howrah-711103, India.*

[*]*Corresponding author. Fax: +91 33 24735197; Tel: +91 33 24995806.*

E-Mail: asishksen@yahoo.com; aksen@iicb.res.in (A. K. Sen)

[$]*Contributed equally to the work*

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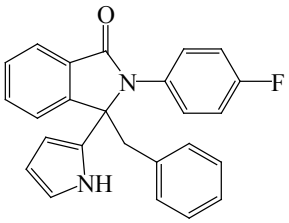
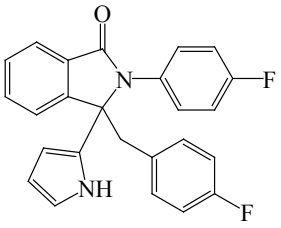
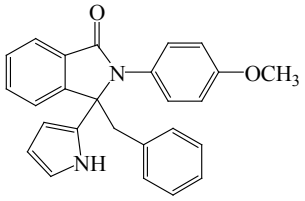
Compound 5a: ^{1}H NMR (DMSO-d_6; 300 MHz) : δ = 3.59 (d, 1 H, *J* = 14.1 Hz), 3.95 (d, 1 H, *J* = 14.1 Hz), 6.08 (s, 1H), 6.40-6.46 (m, 3 H), 6.70 (s, 1 H), 6.93-7.06 (m, 5 H), 7.18 (t, 2 H, *J* = 9 Hz), 7.43-7.47 (m, 2 H), 7.57-7.65 (m, 2H), 10.74 (s, 1 H) ppm; ^{13} C NMR (DMSO-d_6; 75 MHz) : δ = 69.07, 106.87, 107.88, 114.94, 115.24, 119.44, 123.10, 123.24, 126.31, 126.41, 126.68, 127.38, 128.42, 130.00, 130.59, 130.71, 132.25, 133.43, 134.24, 147.95, 157.79, 161.01, 166.93 ppm; HRMS (ESI): m/z calcd for $C_{25}H_{19}FN_2O$ $[M+Na]^+$405.1379; found: 405.1384.



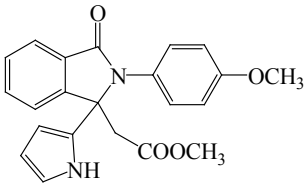
Compound 5b: ^{1}H NMR (DMSO-d_6; 300 MHz) : δ = 3.59 (d, 1 H, *J* = 14.1 Hz), 3.93 (d, 1 H, *J* = 14.1 Hz), 6.09 (s, 1H), 6.39-6.49 (m, 3 H), 6.71 (s, 1H), 6.80 (t, 2 H, *J* = 8.7 Hz), 6.99-7.04 (m, 2 H), 7.18 (t, 2 H, *J* = 8.7 Hz), 7.46 (t, 2 H, *J* = 7.2 Hz), 7.59-7.65 (m, 2 H), 10.75 (s, 1H) ppm; ^{13} C NMR (DMSO-d_6; 75 MHz) : δ = 68.97, 106.89, 107.85, 114.03, 114.31, 115.01, 115.31, 119.46, 123.11, 123.21, 126.19, 126.30, 128.50, 130.42, 130.46, 130.49, 130.54, 131.68, 131.79, 132.31, 133.34, 133.37, 147.73, 166.86 ppm; HRMS (ESI): m/z calcd for $C_{25}H_{18}N_2F_2O$ $[M+Na]^+$423.1285; found: 423.1292.



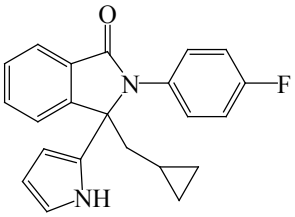
Compound 5c: ^{1}H NMR (DMSO-d_6; 300 MHz) : δ = 3.54 (d, 1 H, *J* = 14.1 Hz), 3.75 (s, 3 H), 3.91 (d, 1 H, *J* = 14.1 Hz), 6.07 (s, 1H), 6.34 (s, 1H), 6.50 (d, 2 H, *J* = 7.5 Hz), 6.69 (s, 1H), 6.83-7.03 (m, 7 H), 7.43 (t, 2 H, *J* = 6.9 Hz), 7.54-7.59 (m, 2 H), 10.69 (s, 1H) ppm; ^{13} C NMR (DMSO-d_6; 75 MHz) : δ = 55.17, 68.92, 106.86, 107.70, 113.49, 119.23, 122.92, 123.24, 126.18, 126.56, 127.31, 128.26, 129.67, 130.07, 130.89, 130.97, 131.87, 134.45, 147.87, 156.76, 166.71 ppm; HRMS (ESI): m/z calcd for $C_{26}H_{22}N_2O_2$ $[M+Na]^+$417.1579; found: 417.1573.



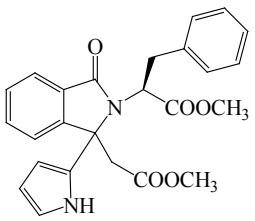
Compound 5d: ^{1}H NMR (DMSO-d_6; 600 MHz) : δ = 3.07 (d, 1 H, *J* = 9 Hz), 3.28 (s, 3H), 3.59 (d, 1 H, *J* = 6 Hz), 3.71 (s, 3H), 5.95 (s, 1H), 6.08 (s, 1H), 6.65-6.71 (m, 3 H), 6.84-6.86 (m, 2 H), 7.32 (d, 1 H, *J* = 3 Hz), 7.48-7.56 (m, 2 H), 7.75 (d, 1 H, *J* = 3 Hz), 10.66 (s, 1H) ppm; ^{13} C NMR (DMSO-d_6; 150 MHz) : δ = 37.94, 51.56, 55.61, 66.38, 107.83, 114.25, 119.88, 123.04, 123.37, 128.74, 128.77, 129.01, 129.90, 132.03, 132.41, 147.90, 158.38, 167.65, 169.08 ppm; HRMS (ESI): m/z calcd for $C_{22}H_{20}N_2O_4$ [M+Na]+399.1321; found: 399.1325.



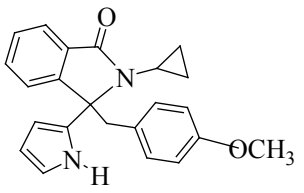
Compound 5e: ^{1}H NMR (DMSO-d_6; 300 MHz) : δ = 5.99 (s, 1H), 6.12 (s, 1H), 6.66 (s, 1H), 6.95-6.99 (m, 2 H), 7.16 (t, 2 H, *J* = 8.7 Hz), 7.30 (d, 1 H, *J* = 7.5 Hz), 7.50-7.63 (m, 2 H), 7.81 (d, 1 H, *J* = 7.2 Hz), 10.66 (s, 1H) ppm; ^{13} C NMR (DMSO-d_6; 75 MHz) : δ = 3.37, 4.64, 38.16, 69.17, 106.82, 107.57, 115.15, 119.17, 122.93, 123.16, 127.48, 127.59, 128.23, 130.54, 130.87, 132.15, 132.99, 133.03, 148.84, 158.28, 167.24 ppm; HRMS (ESI): m/z calcd for $C_{22}H_{19}N_2FO$ [M+Na]+369.1379; found: 369.1386.



Compound 5f: ^{1}H NMR (DMSO-d_6; 300 MHz) : δ = 3.23 (s, 3H), 3.36-3.63 (m, 7 H), 4.64 (t, 1 H, *J* = 7.5 Hz), 5.86 (s, 1H), 5.93 (s, 1H), 6.48 (d, 2 H, *J* = 7.2 Hz), 6.56 (d, 1 H, J = 7.5 Hz), 6.69 (s, 1H), 6.99-7.48 (m, 5 H), 7.64 (d, 1 H, *J* = 7.5 Hz), 10.59 (s, 1H) ppm; ^{13} C NMR (DMSO-d_6; 75 MHz) : δ = 35.17, 41.21, 51.65, 57.06, 67.71, 107.09, 109.13, 119.02, 122.60, 124.41, 126.56, 127.54, 128.05, 128.26, 128.46, 129.57, 130.04, 130.92, 132.04, 135.66, 138.26, 146.58, 167.16, 169.92 ppm; HRMS (ESI): m/z calcd for $C_{25}H_{24}N_2O_5$ [M+Na]+455.1583; found: 455.1587.

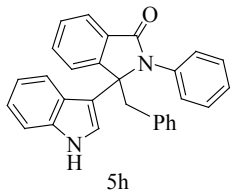


Compound 5g: ^{1}H NMR ($CDCl_3$; 300 MHz): 0.27-0.36 (m, 1 H), 0.48-0.58 (m, 1 H), 0.83-1.19 (m, 2 H), 2.59-2.66 (m, 1 H), 3.64 (s, 1H), 3.68-3.83 (m, 2H), 6.20 (s, 1 H), 6.47-6.57 (m, 5H), 6.77 (s, 1 H), 6.96-7.00 (m, 2H), 7.20-7.26 (m, 1H), 7.34-7.39 (m, 1H), 8.93 (1H, brs) ppm; ^{13}C NMR ($CDCl_3$; 75 MHz) : δ = 2.51, 5.84, 24.02, 39.96, 55.01, 69.03,



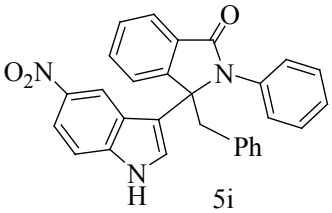
107.04, 108.14, 113.17, 113.26, 118.84, 122.47, 122.74, 126.20, 128.30, 130.81, 130.97, 131.26, 131.45, 148.11, 158.30, 170.15 ppm; HRMS (ESI): m/z calcd for $C_{23}H_{22}N_2O_2$ [M+Na]+381.1579; found: 381.1585.

Compound 5h: ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.85 (d, 1H, *J* = 14.1 Hz), 4.11 (d, 1H, *J* = 14.1 Hz), 6.48 (d, 2H, *J* = 7.2 Hz), 6.56 (d, 1H, *J* = 8.1 Hz), 6.68 (t, 1H, *J* = 7.8 Hz), 6.93-7.14 (m, 5 H), 7.24 (d, 4H, *J* = 3.9 Hz), 7.35-7.47 (m, 3H), 7.54-7.64 (m,2H), 7.92 (s,1H), 11.34 (s,1H) ppm; ESI MS: $[M+Na]^+$ 437.48



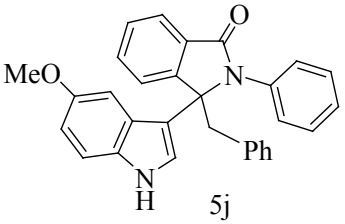
5h

Compound 5i: ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.82 (d, 1H, *J* = 13.5 Hz), 3.94 (d, 1H, *J* = 13.8 Hz), 6.55 (d, 2H, *J* = 7.5 Hz), 6.99 (t, 2H, *J* = 7.8 Hz), 7.07-7.22 (m, 6H), 7.39 (d, 2H, *J* = 9 Hz), 7.48-7.53 (m, 2H), 7.60 (d, 2H, *J* = 7.0 Hz), 7.91-7.94 (m, 1H), 8.03 (dd, 1H, *J* = 2.1, 8.5 Hz), 8.85 (brs, 1H) ppm; ESI MS: $[M+H]^+$ 460.74



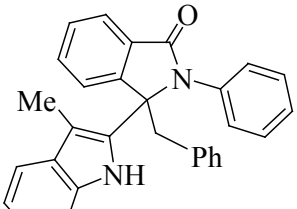
5i

Compound 5j: ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.43(s, 3H), 3.77 (d, 1H, *J* = 13.8 Hz), 3.91 (d, 1H, *J* = 13.8 Hz), 6.11 (s, 1H), 6.53 (d, 2H, *J* = 7.2 Hz), 6.76 (dd, 1H, *J* = 2.4, 9 Hz), 6.98 (t, 2H, *J* = 7.3 Hz), 7.05-7.10 (m, 3H), 7.13-7.29 (m, 5H), 7.42-7.54 (m, 3H), 7.85 (d, 1H, *J* = 7.5 Hz), 8.23 (brs, 1H) ppm; ESI MS: $[M+H]^+$ 445.63

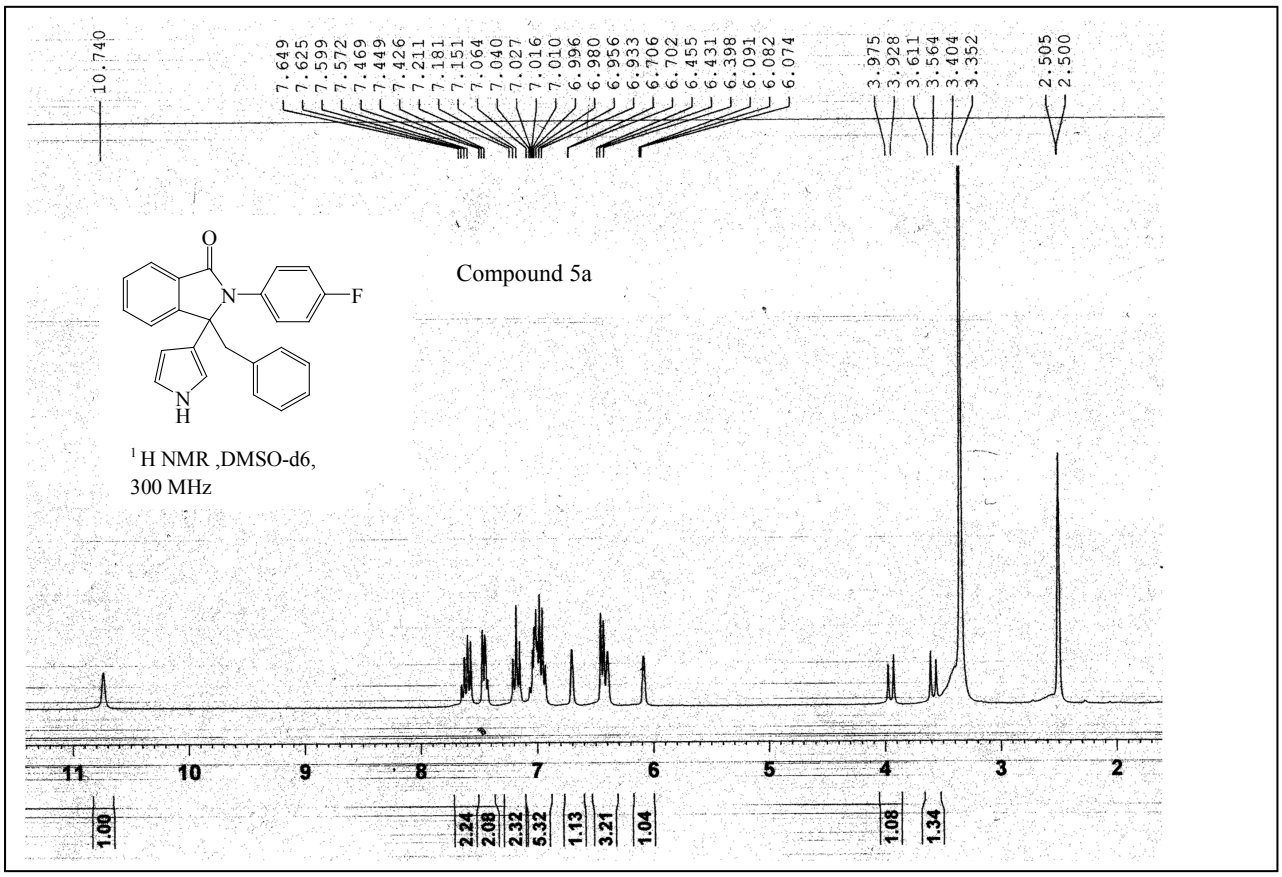


5j

Compound 5k: ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.24 (s, 3H), 3.88 (d, 1H, *J* = 13.5 Hz), 4.09 (d, 1H, *J* = 13.5 Hz), 6.38 (d, 2H, *J* = 7.2 Hz), 6.94 (t, 2H, *J* = 7.5 Hz), 7.07 (t, 1H, *J* = 7.2 Hz), 7.17-7.28 (m, 8H), 7.37-7.45 (m, 2H), 7.54-7.60 (m, 2H), 7.73 (d, 1H, *J* = 7.5 Hz), 8.30 (brs, 1H) ppm; ESI MS: $[M+Na]^+$ 451.39



5K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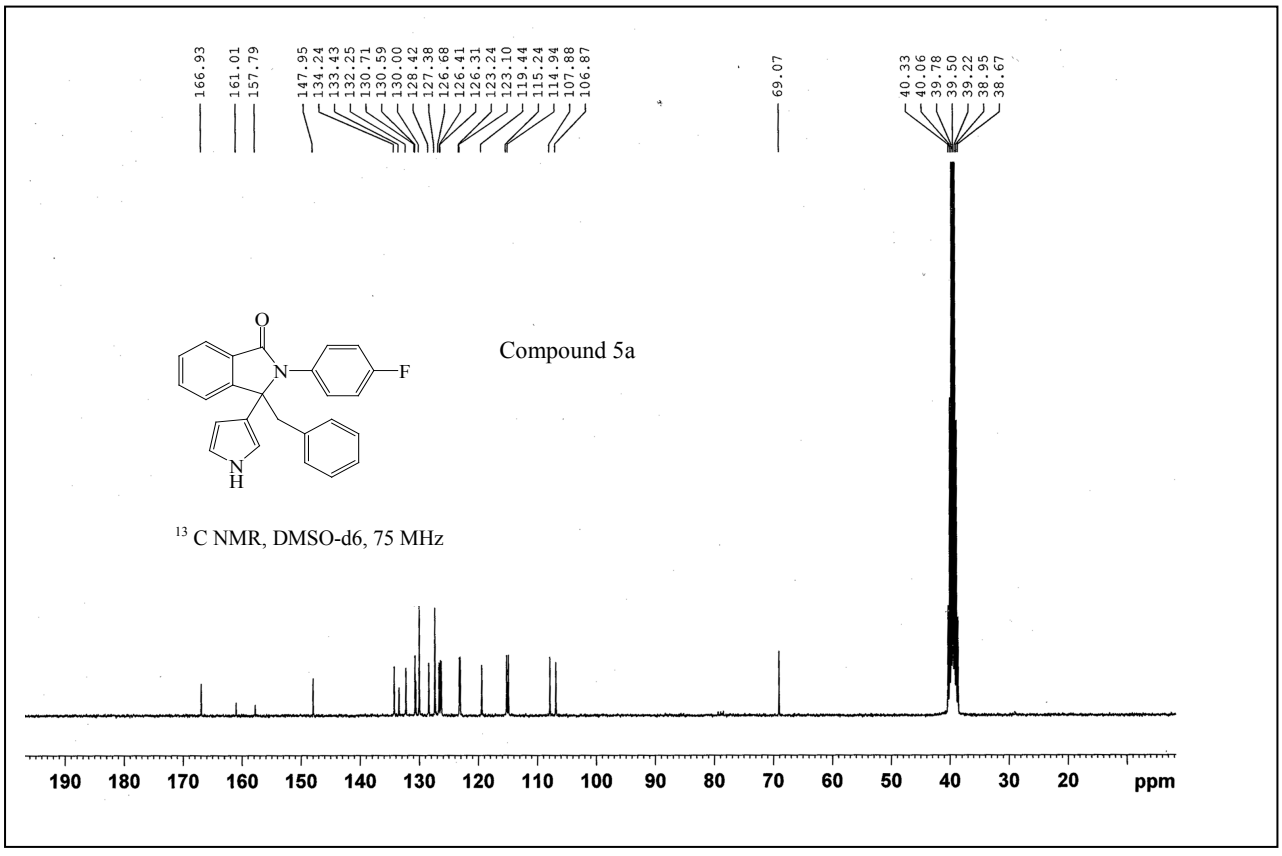
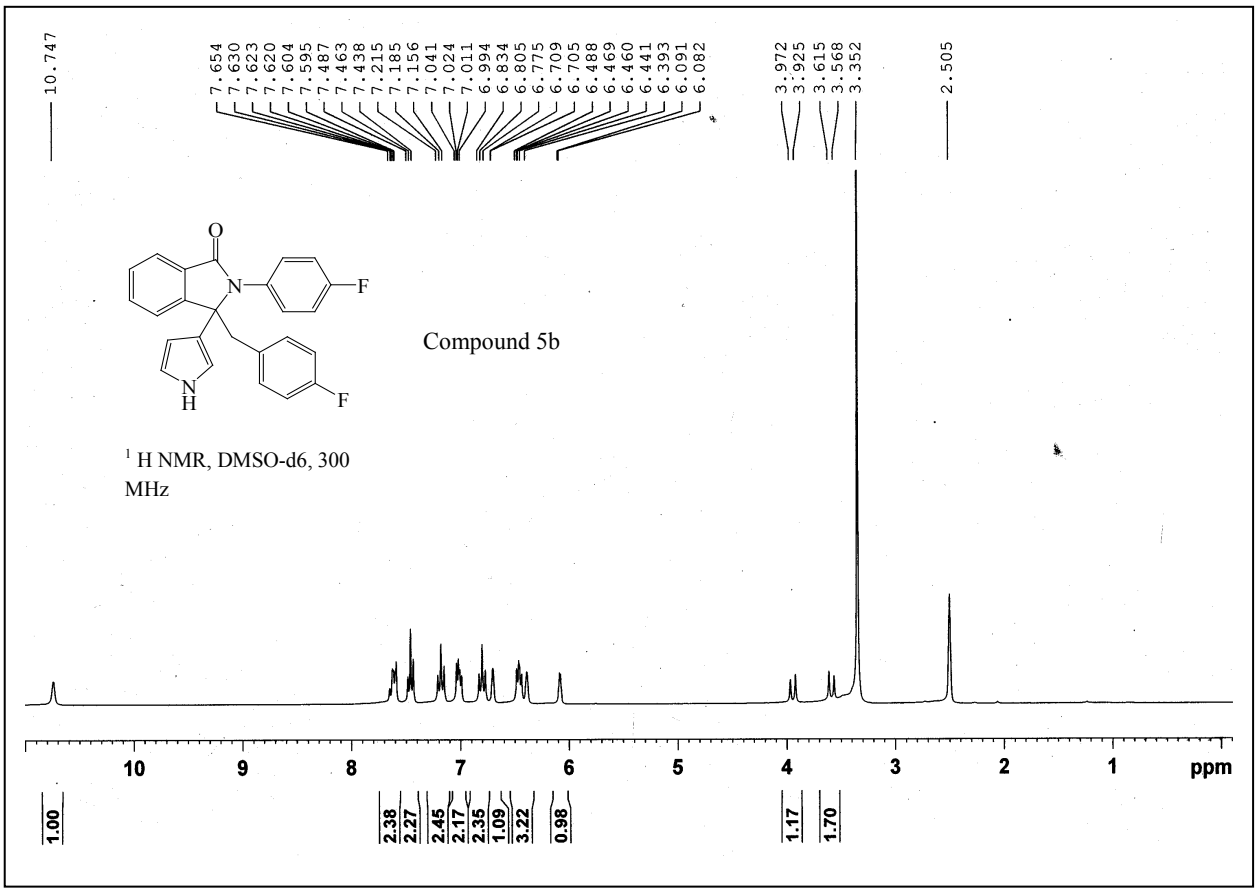
7.649
7.625
7.599
7.572
7.469
7.449
7.426
7.211
7.181
7.151
7.064
7.040
7.027
7.016
7.010
6.996
6.980
6.956
6.933
6.706
6.702
6.455
6.431
6.398
6.091
6.082
6.074
3.975
3.928
3.611
3.564
3.404
3.352
2.505
2.500
O
N
F
N
H
Compound 5a
1 H NMR ,DMSO-d6,
300 MHz
11
10
9
8
7
6
5
4
3
2
1.00
2.24
2.08
2.32
5.32
1.13
3.21
1.04
1.08
1.34


166.93
161.01
157.79
147.95
134.24
133.43
132.25
130.71
130.59
130.00
128.42
127.38
126.68
126.41
126.31
123.24
123.10
119.44
115.24
114.94
107.88
106.87
69.07
40.33
40.06
39.78
39.50
39.22
38.95
38.67
O
N
F
N
H
Compound 5a
13 C NMR, DMSO-d6, 75 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.623
7.620
7.604
7.595
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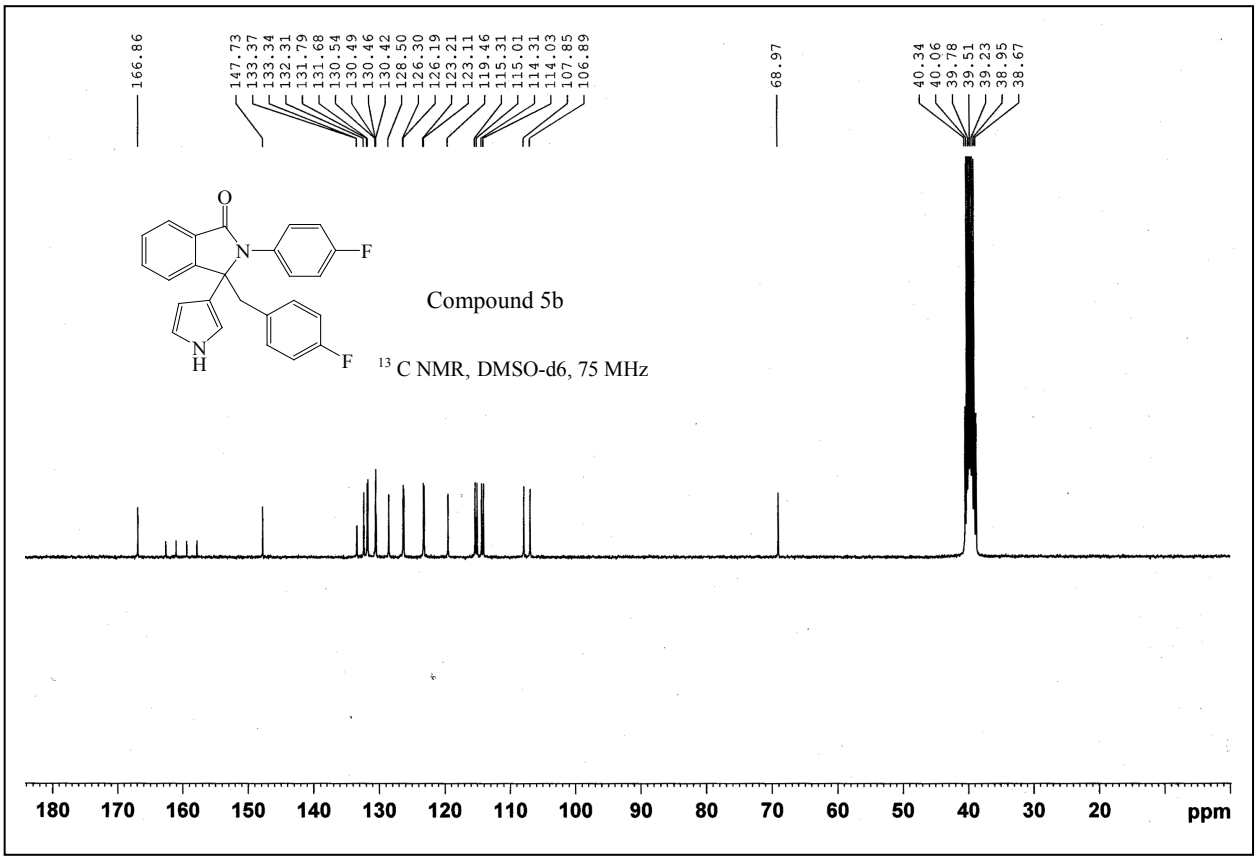
7.156
7.041
7.024
7.011
6.994
6.834
6.805
6.775
6.709
6.705
6.488
6.469
6.460
6.441
6.393
6.091
6.082
3.972
3.925
3.615
3.568
3.352
2.505
Compound 5b
1 H NMR, DMSO-d6, 300 MHz
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.38
2.27
2.45
2.17
2.35
1.09
3.22
0.98
1.17
1.70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133.34
132.31
131.79
131.68
130.54
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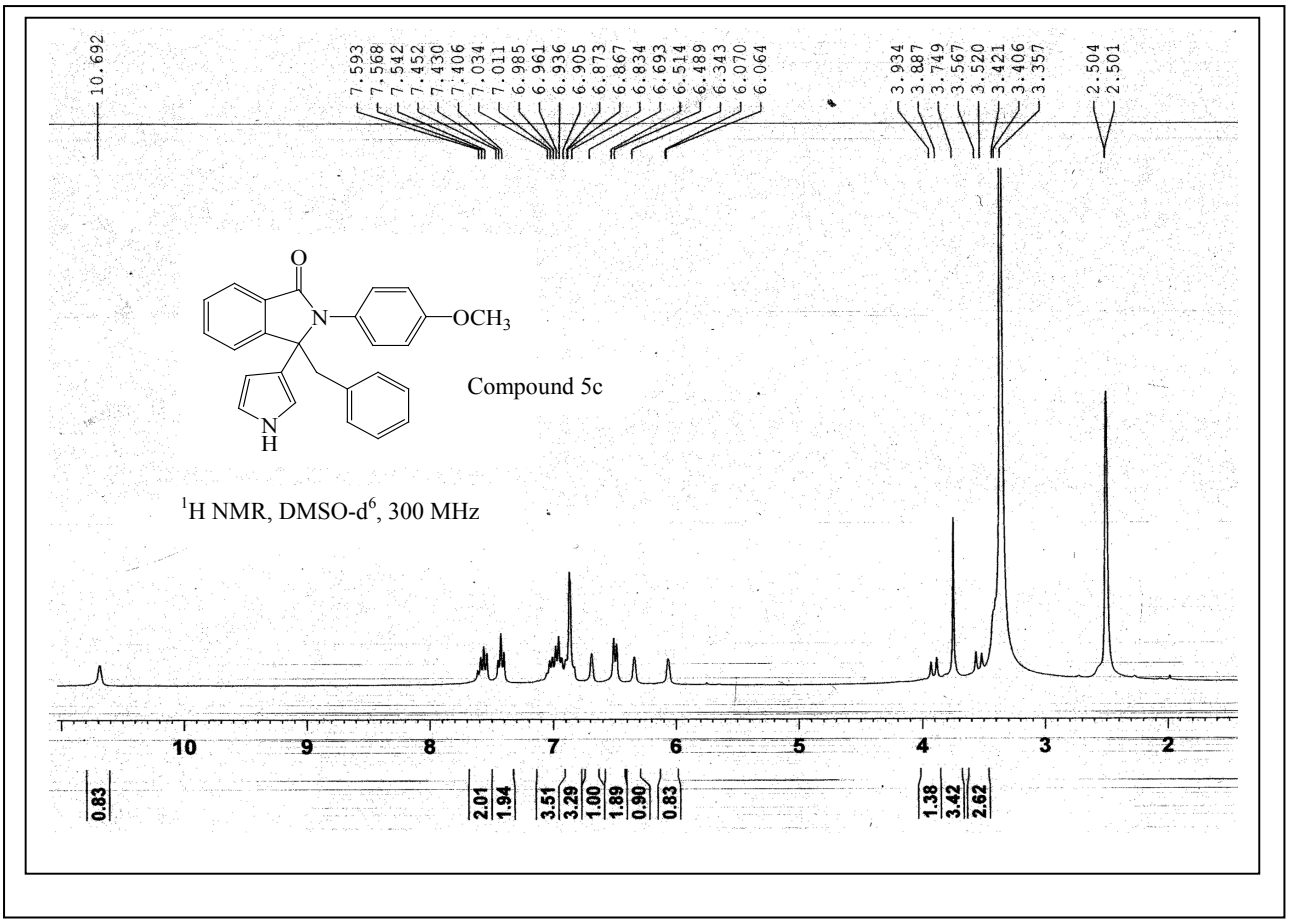
130.46
130.42
128.50
126.30
126.19
123.21
123.11
119.46
115.31
115.01
114.31
114.03
107.85
106.89
68.97
40.34
40.06
39.78
39.51
39.23
38.95
38.67
Compound 5b
13 C NMR, DMSO-d6, 75 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.568
7.542
7.452
7.430
7.406
7.034
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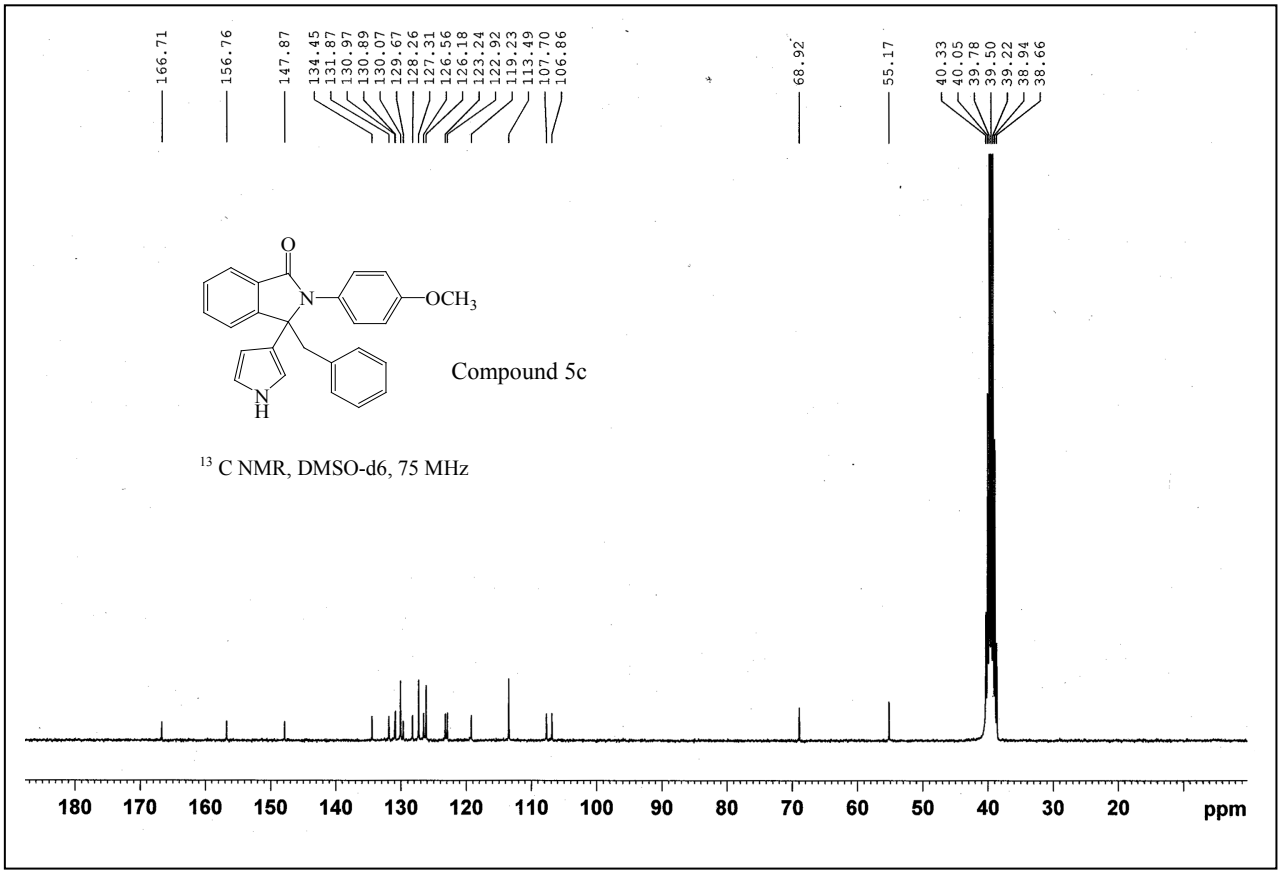
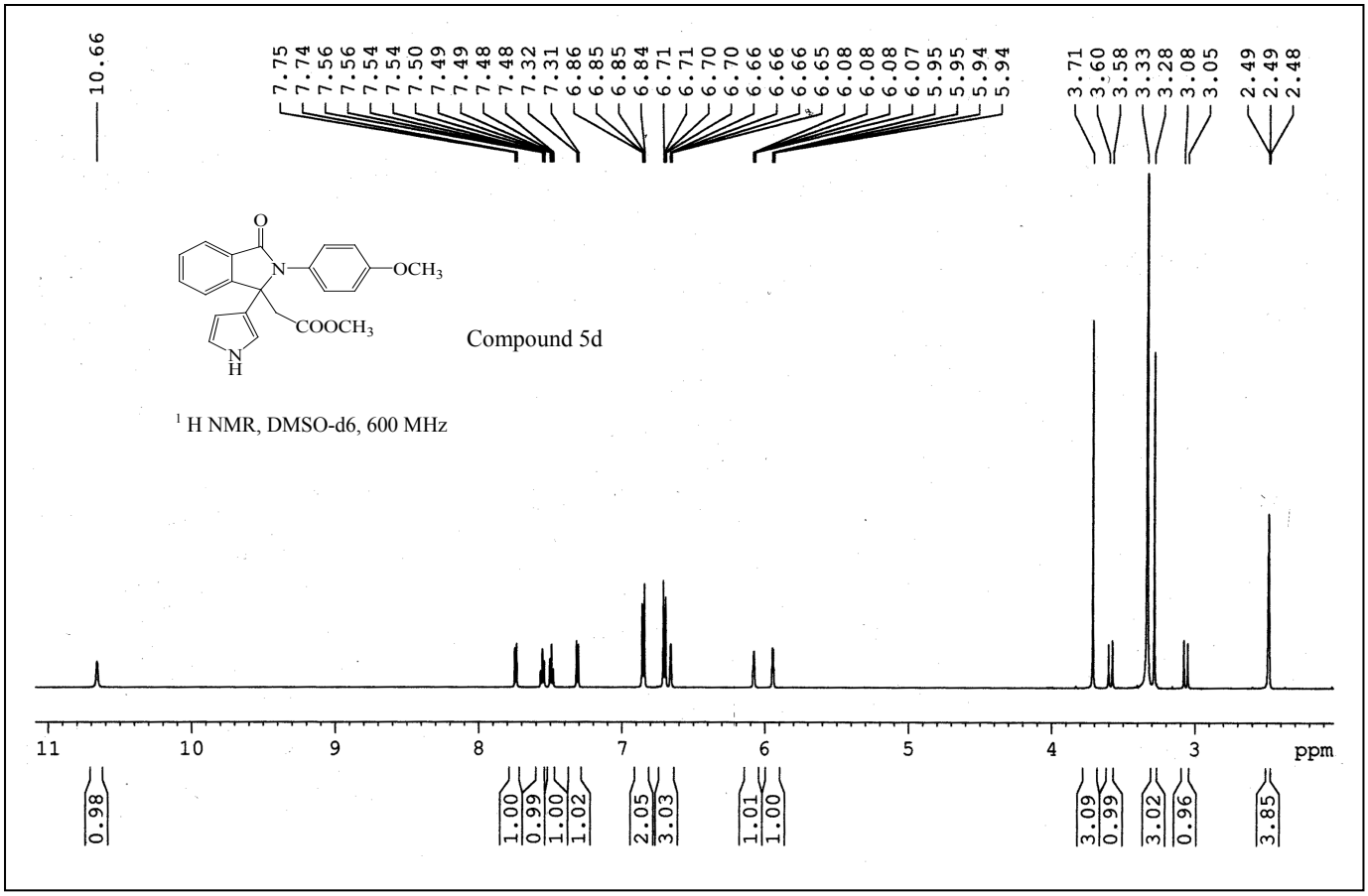
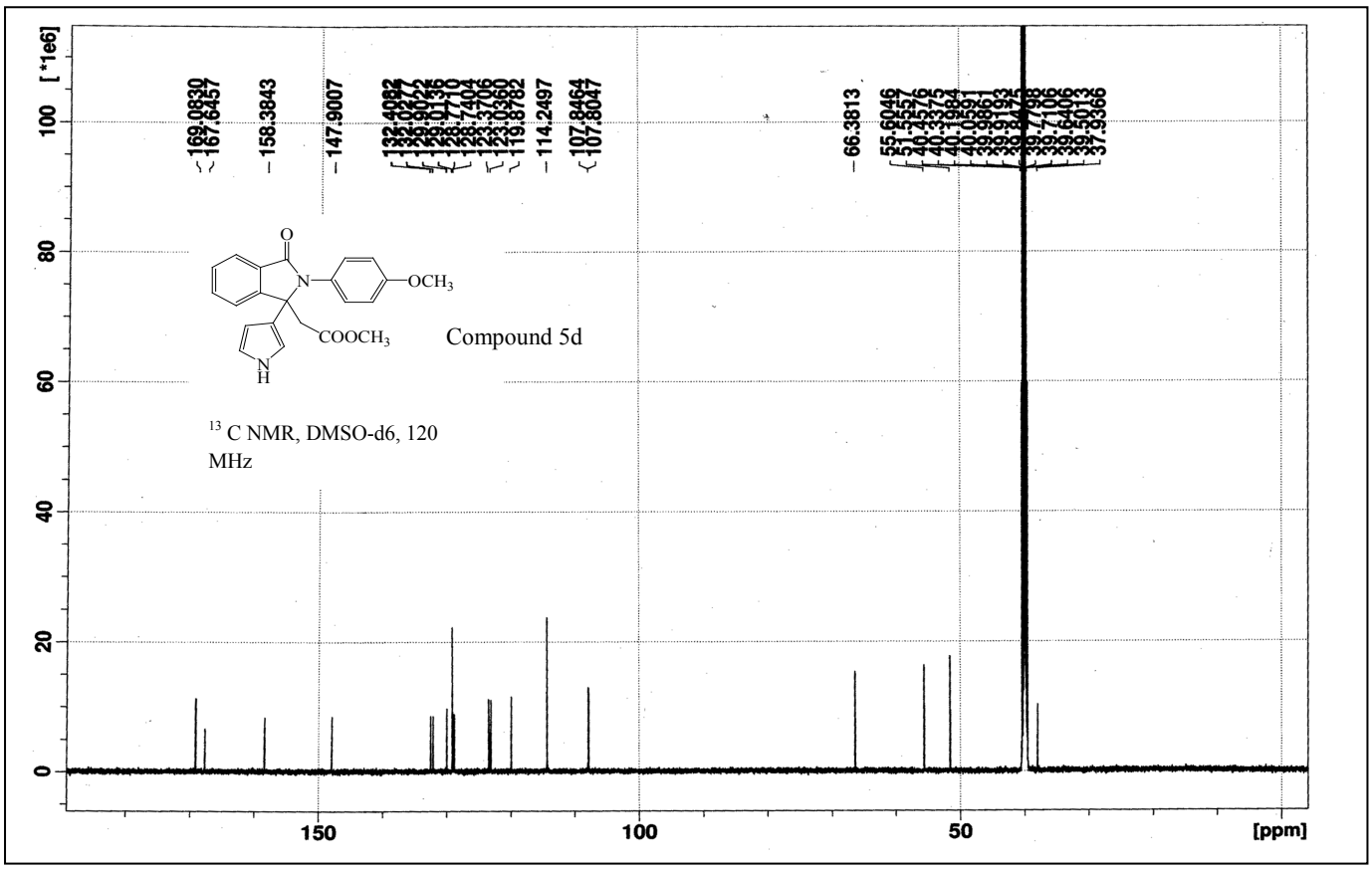
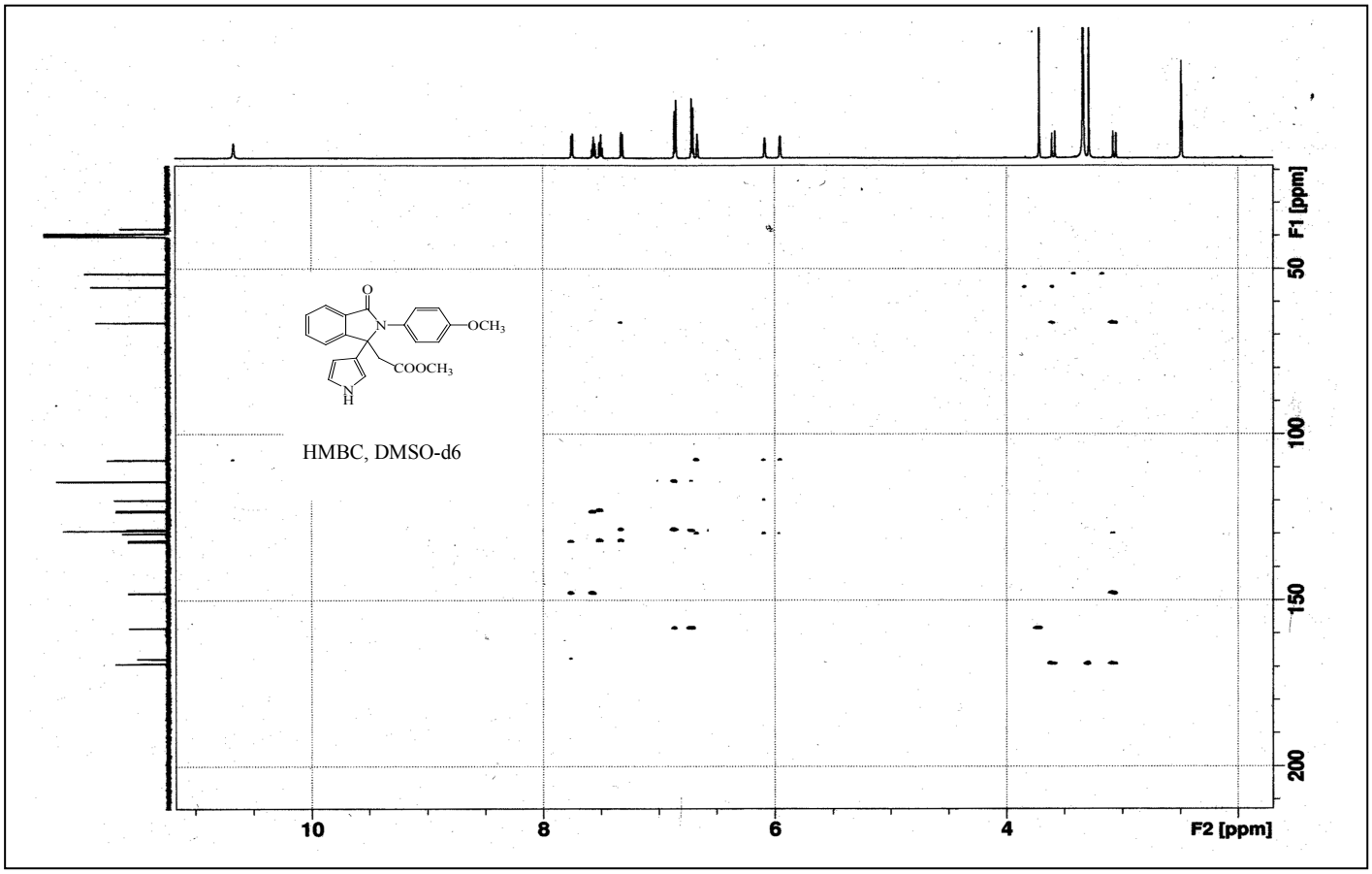
6.985
6.961
6.936
6.905
6.873
6.867
6.834
6.693
6.514
6.489
6.343
6.070
6.064
3.934
3.887
3.749
3.567
3.520
3.421
3.406
3.357
2.504
2.501
O
N
OCH3
N
H
Compound 5c
1H NMR, DMSO-d6, 300 MHz
10
9
8
7
6
5
4
3
2
0.83
2.01
1.94
3.51
3.29
1.00
1.89
0.90
0.83
1.38
3.42
2.62

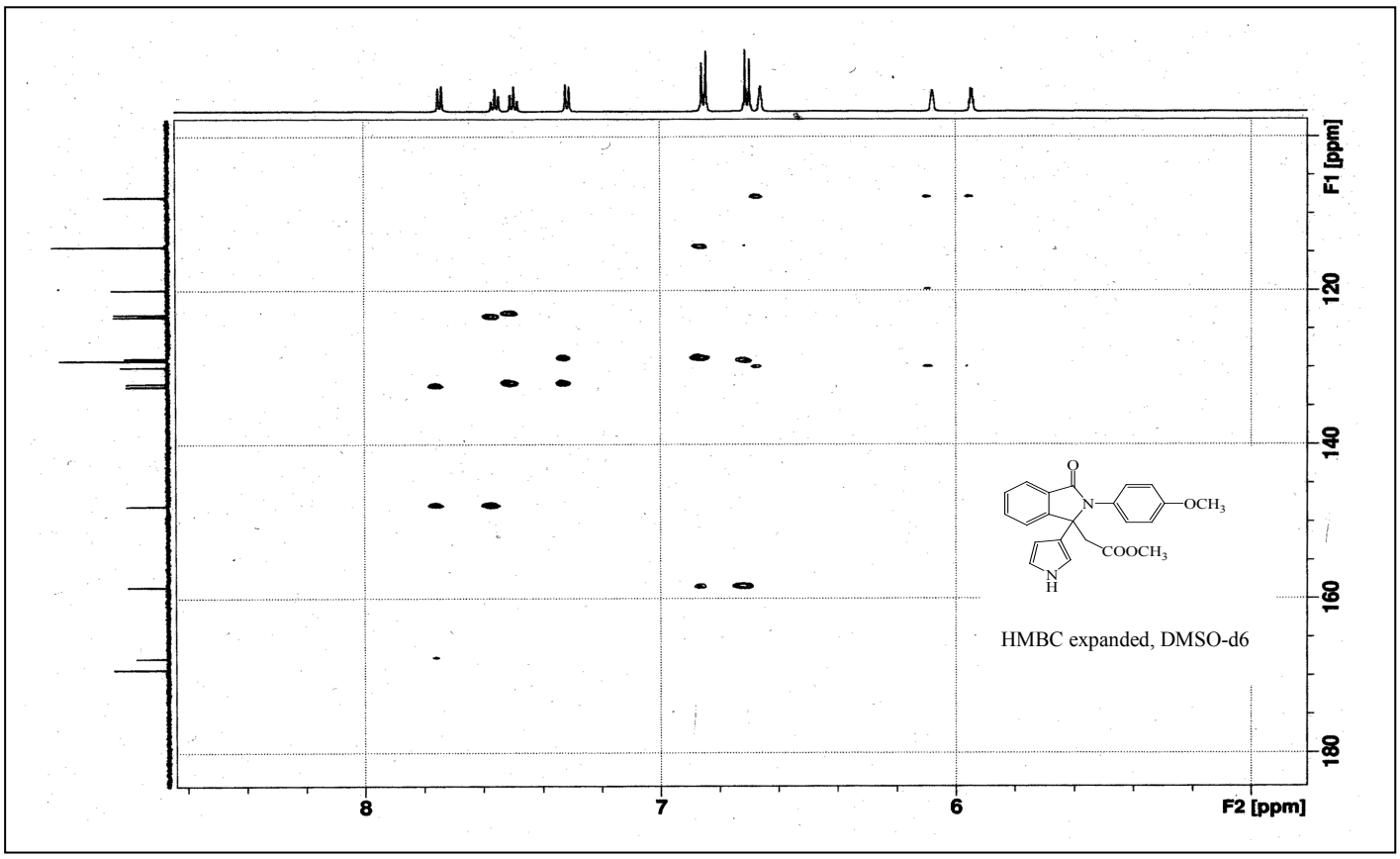

166.71
156.76
147.87
134.45
131.87
130.97
130.89
130.07
129.67
128.26
127.31
126.56
126.18
123.24
122.92
119.23
113.49
107.70
106.86
68.92
55.17
40.33
40.05
39.78
39.50
39.22
38.94
38.66
O
N
OCH3
N
H
Compound 5c
13 C NMR, DMSO-d6, 75 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



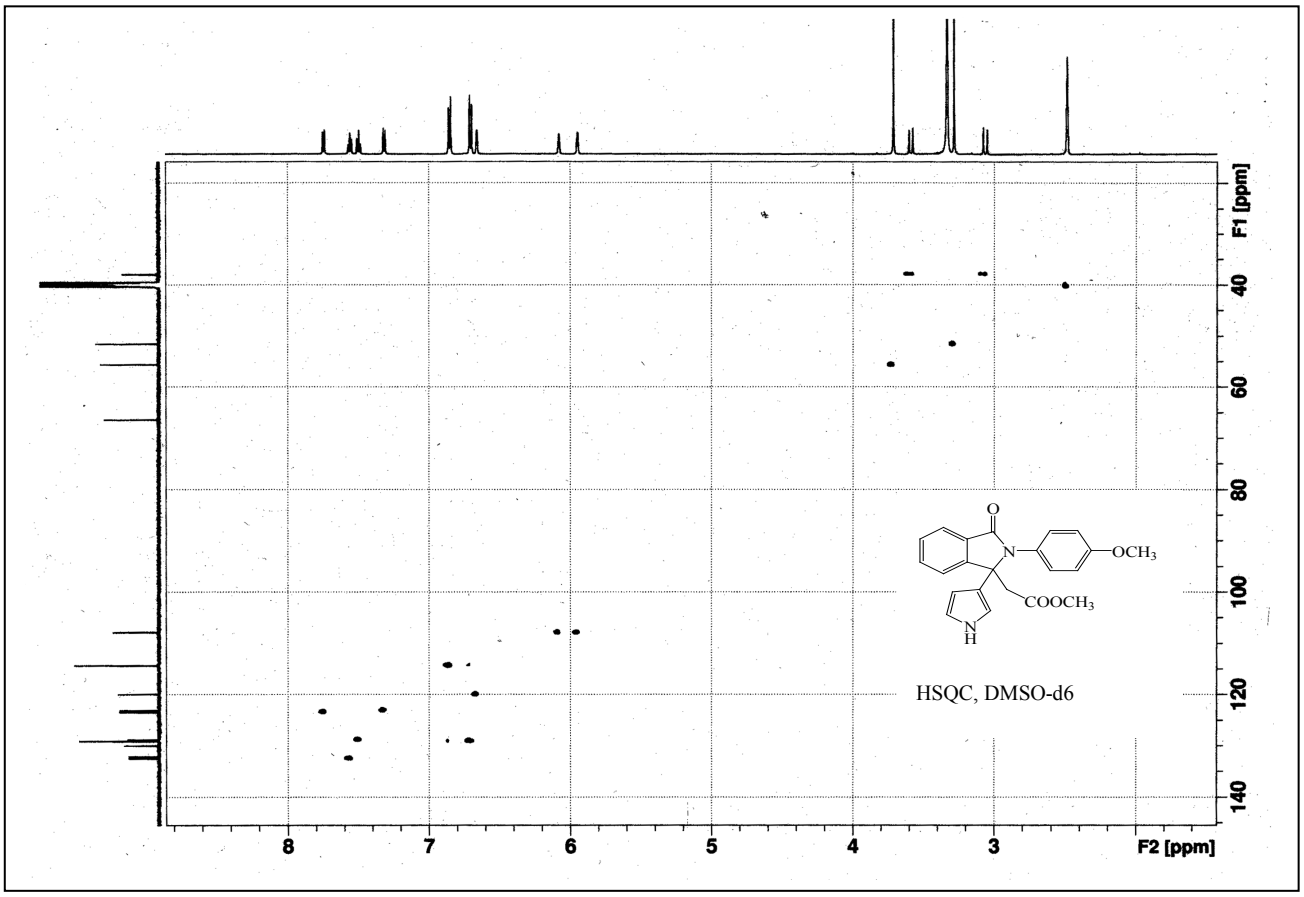




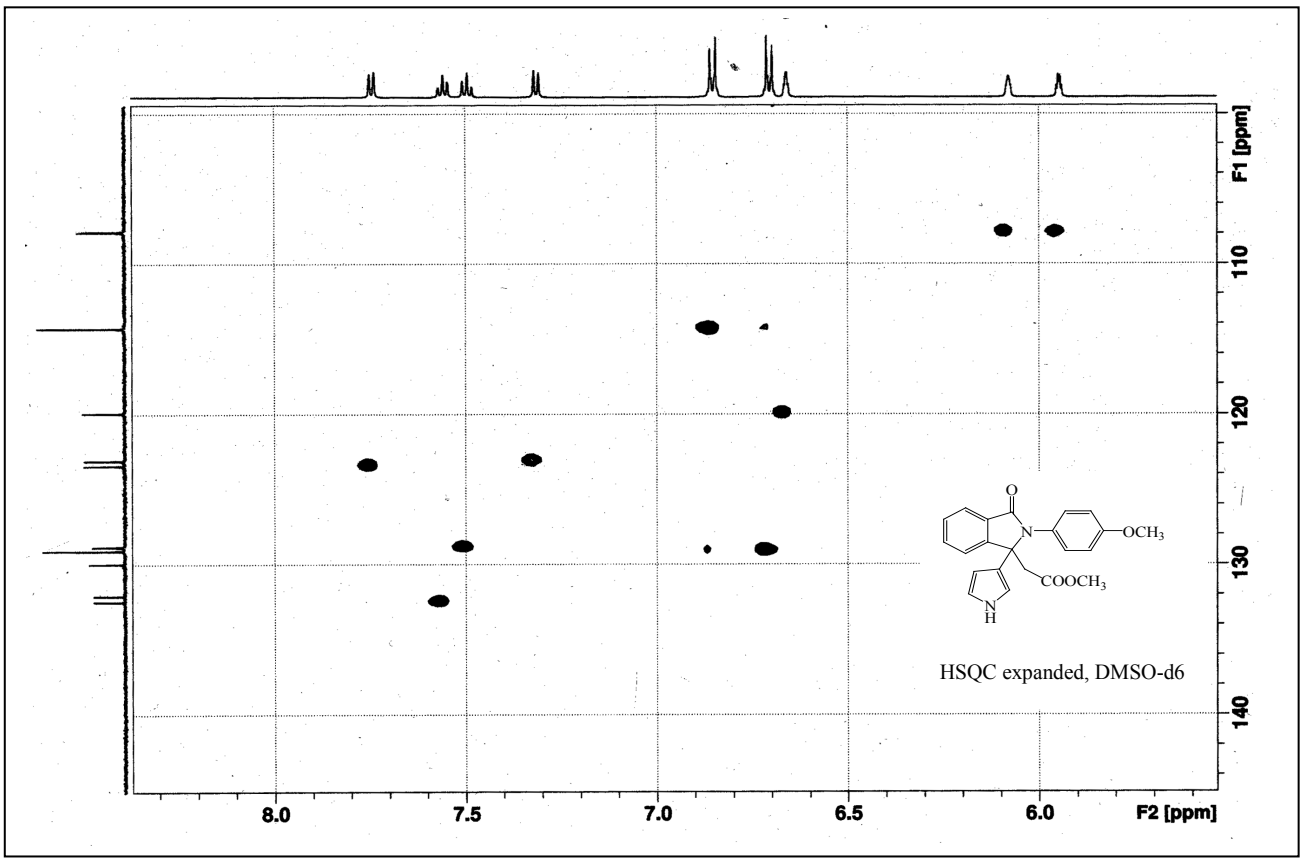
HMBC, DMSO-d6



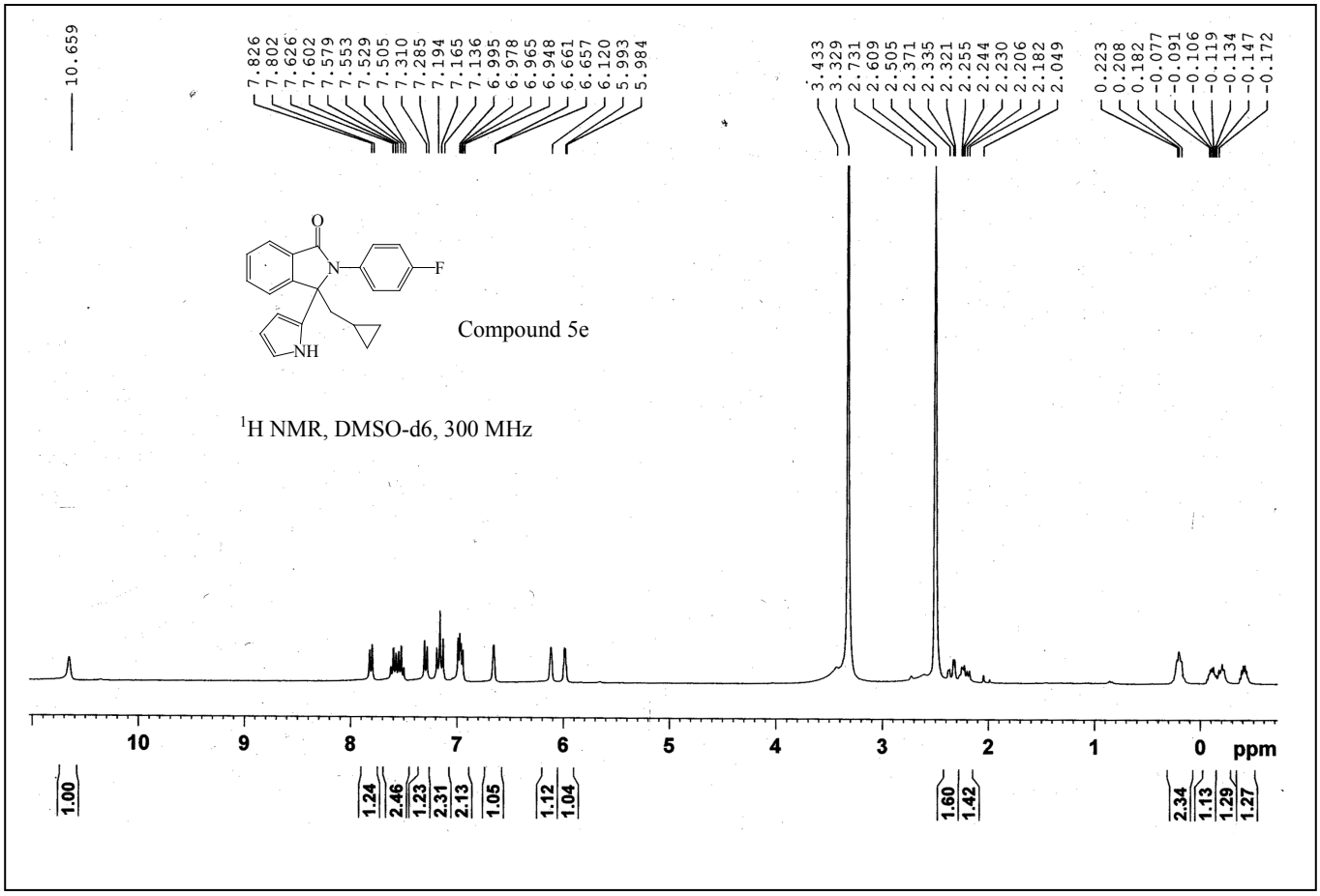
HMBC expanded, DMSO-d6

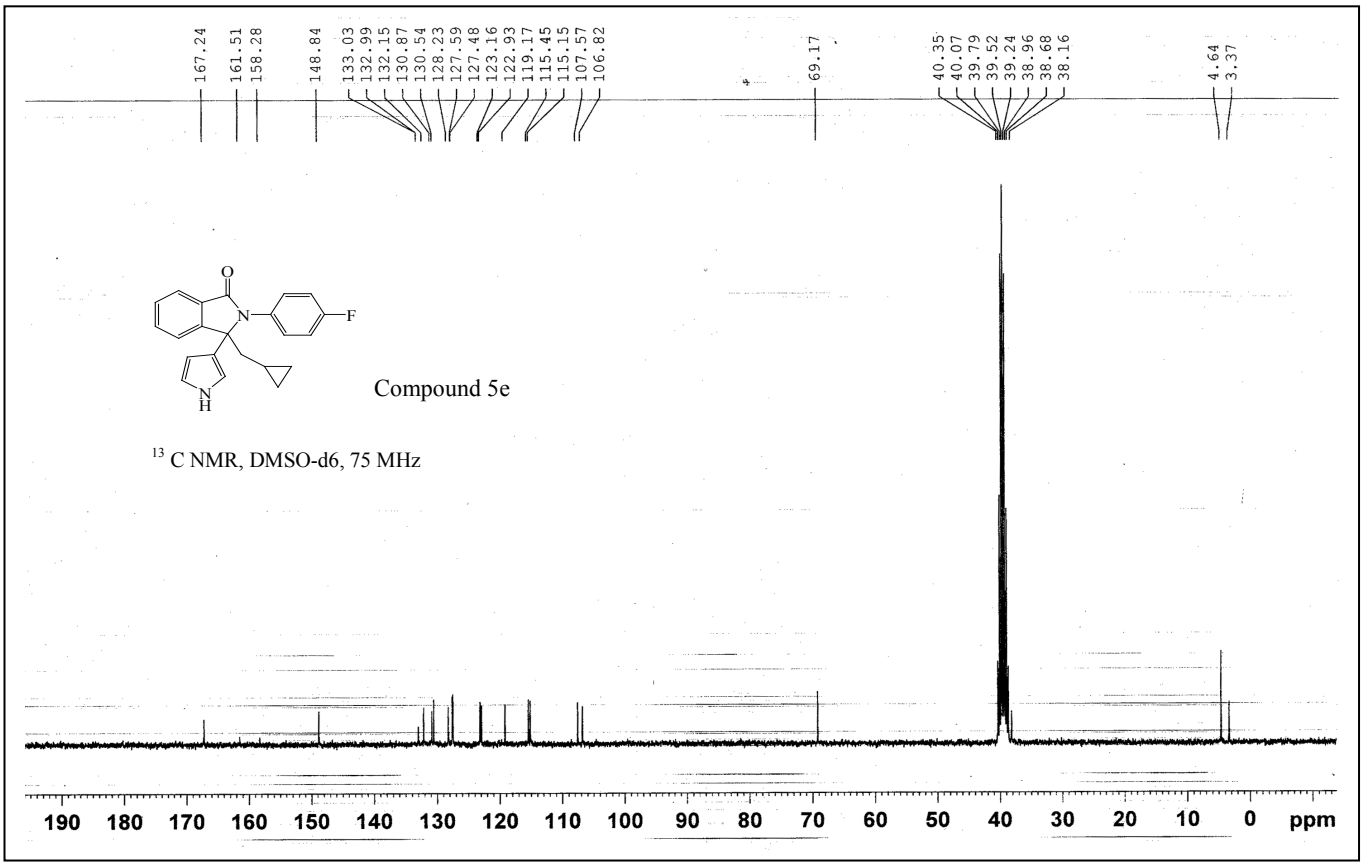


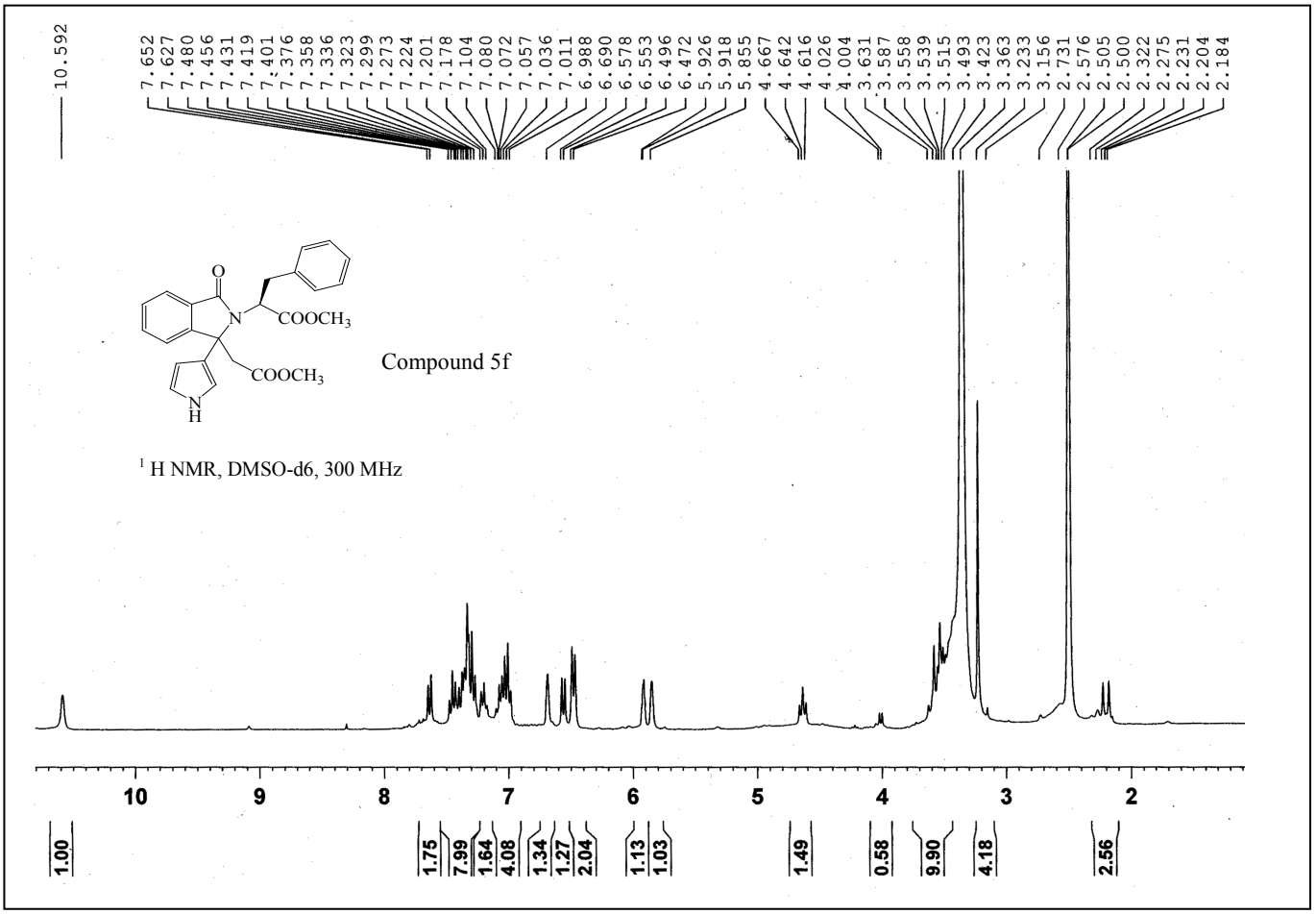
HSQC, DMSO-d6

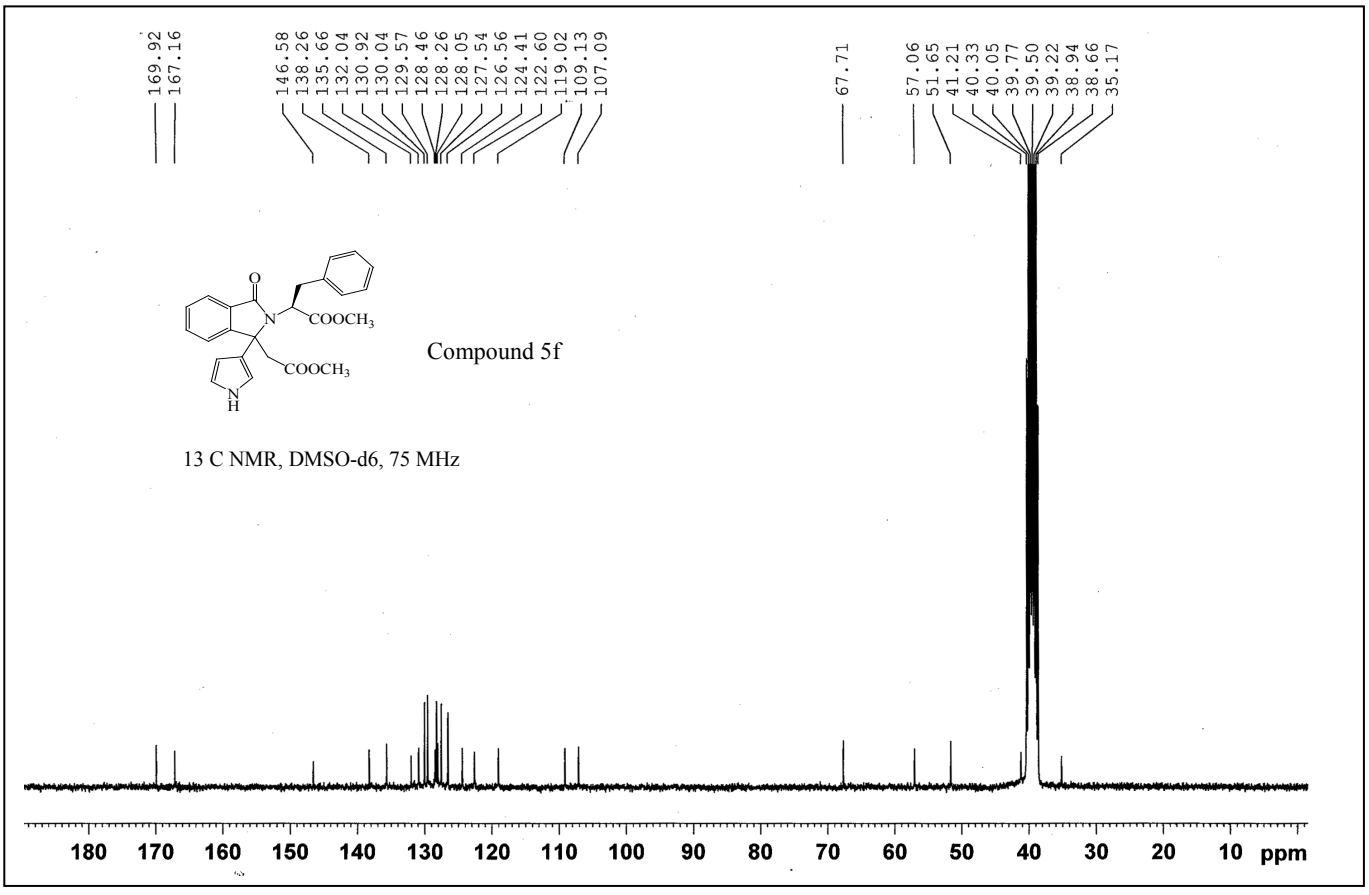


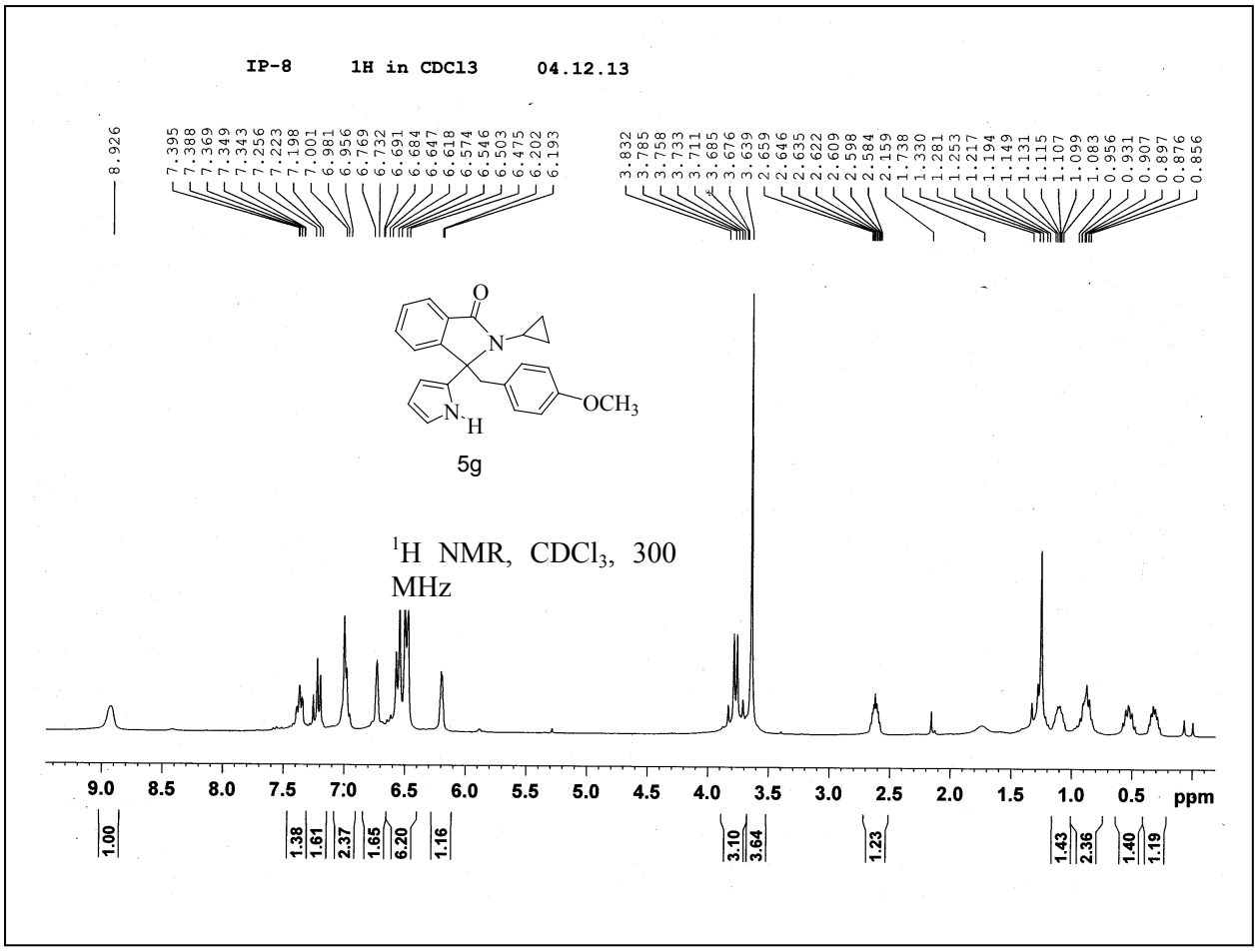
HSQC expanded, DMSO-d6

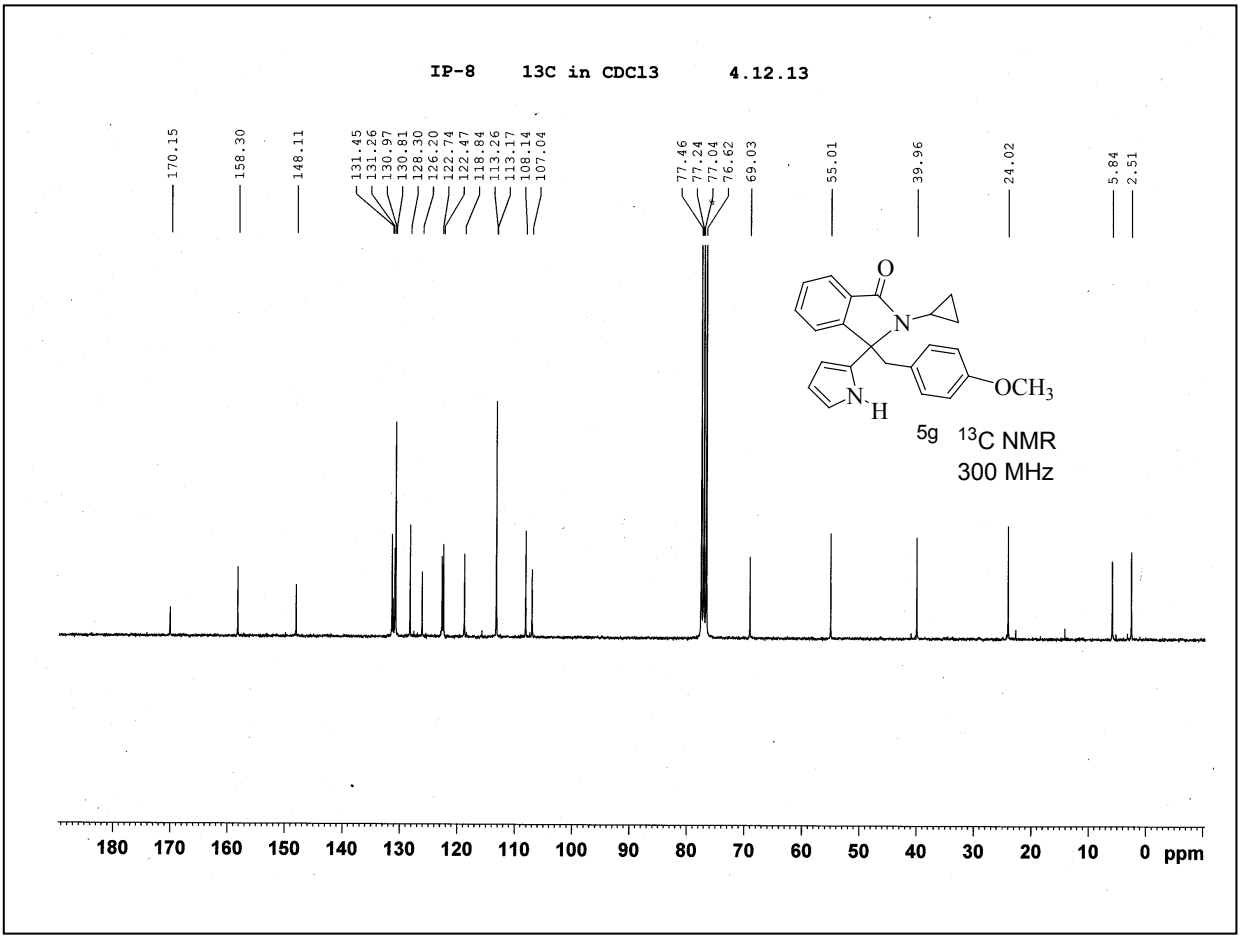


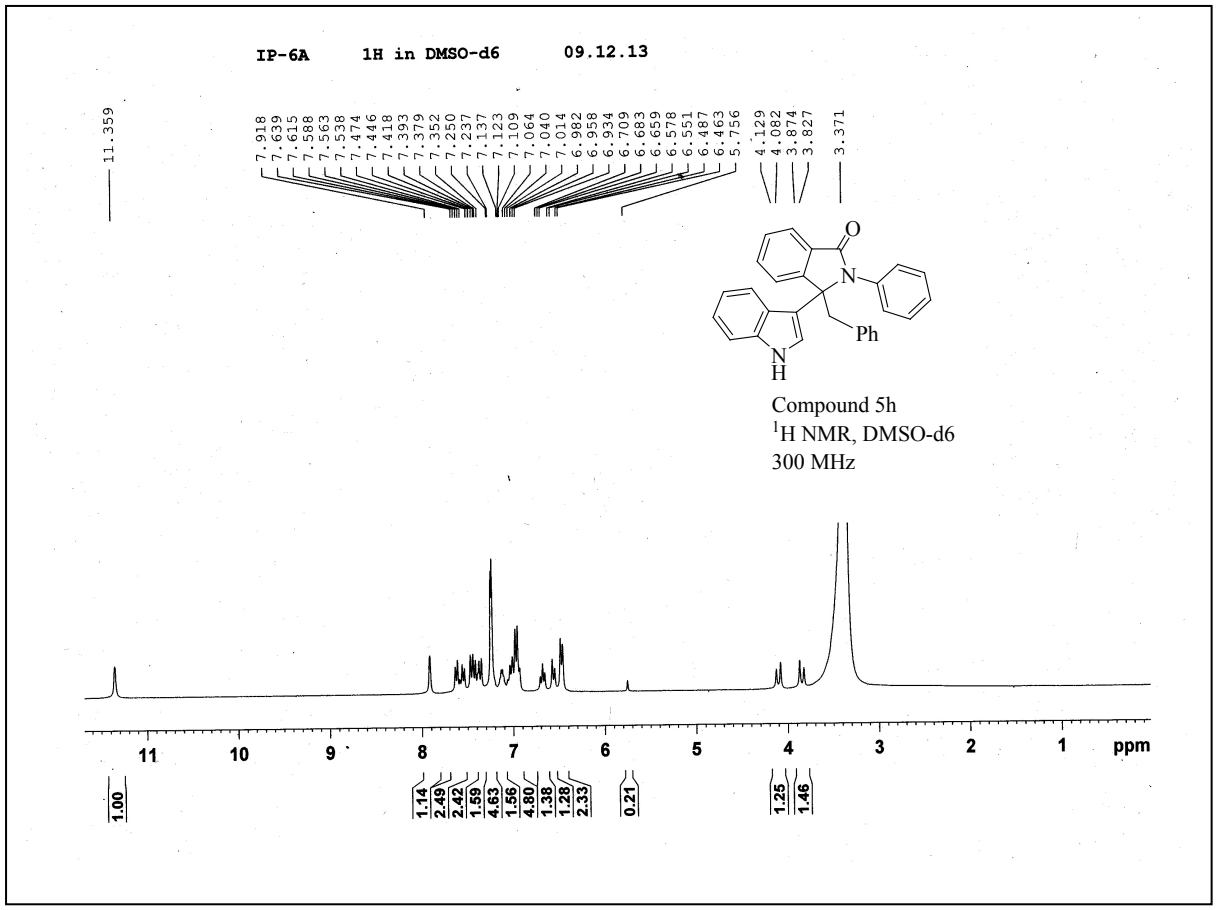



10.592
7.652
7.627
7.480
7.456
7.431
7.419
7.401
7.376
7.358
7.336
7.323
7.299
7.273
7.224
7.201
7.178
7.104
7.080
7.072
7.057
7.036
7.011
6.988
6.690
6.578
6.553
6.496
6.472
5.926
5.918
5.855
4.667
4.642
4.616
4.026
4.004
3.631
3.587
3.558
3.539
3.515
3.493
3.423
3.363
3.233
3.156
2.731
2.576
2.505
2.500
2.322
2.275
2.231
2.204
2.184
O
N
COOCH3
COOCH3
N
H
Compound 5f
1 H NMR, DMSO-d6, 300 MHz
10
9
8
7
6
5
4
3
2
1.00
1.75
7.99
1.64
4.08
1.34
1.27
2.04
1.13
1.03
1.49
0.58
9.90
4.18
2.56

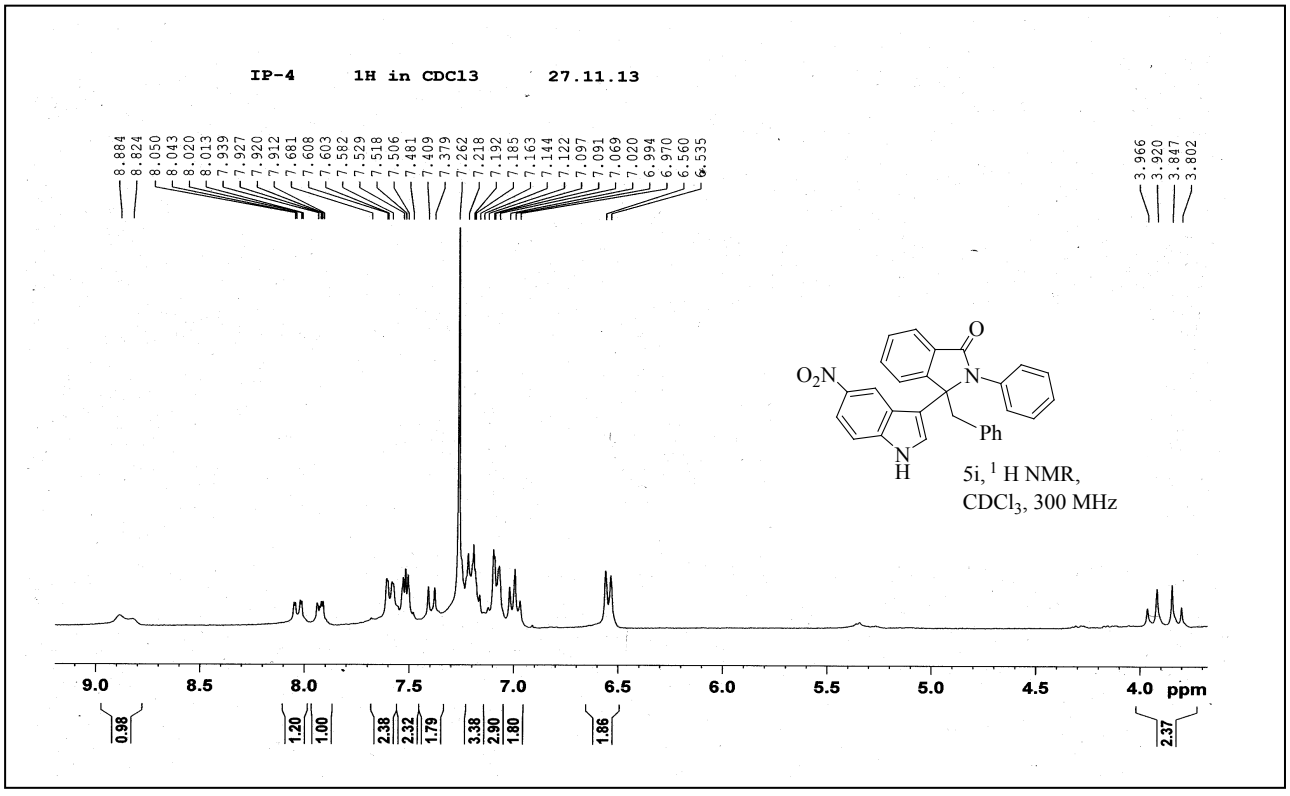

169.92
167.16
146.58
138.26
135.66
132.04
130.92
130.04
129.57
128.46
128.26
128.05
127.54
126.56
124.41
122.60
119.02
109.13
107.09
67.71
57.06
51.65
41.21
40.33
40.05
39.77
39.50
39.22
38.94
38.66
35.17
O
N
COOCH3
COOCH3
N
H
Compound 5f
13 C NMR, DMSO-d6, 75 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



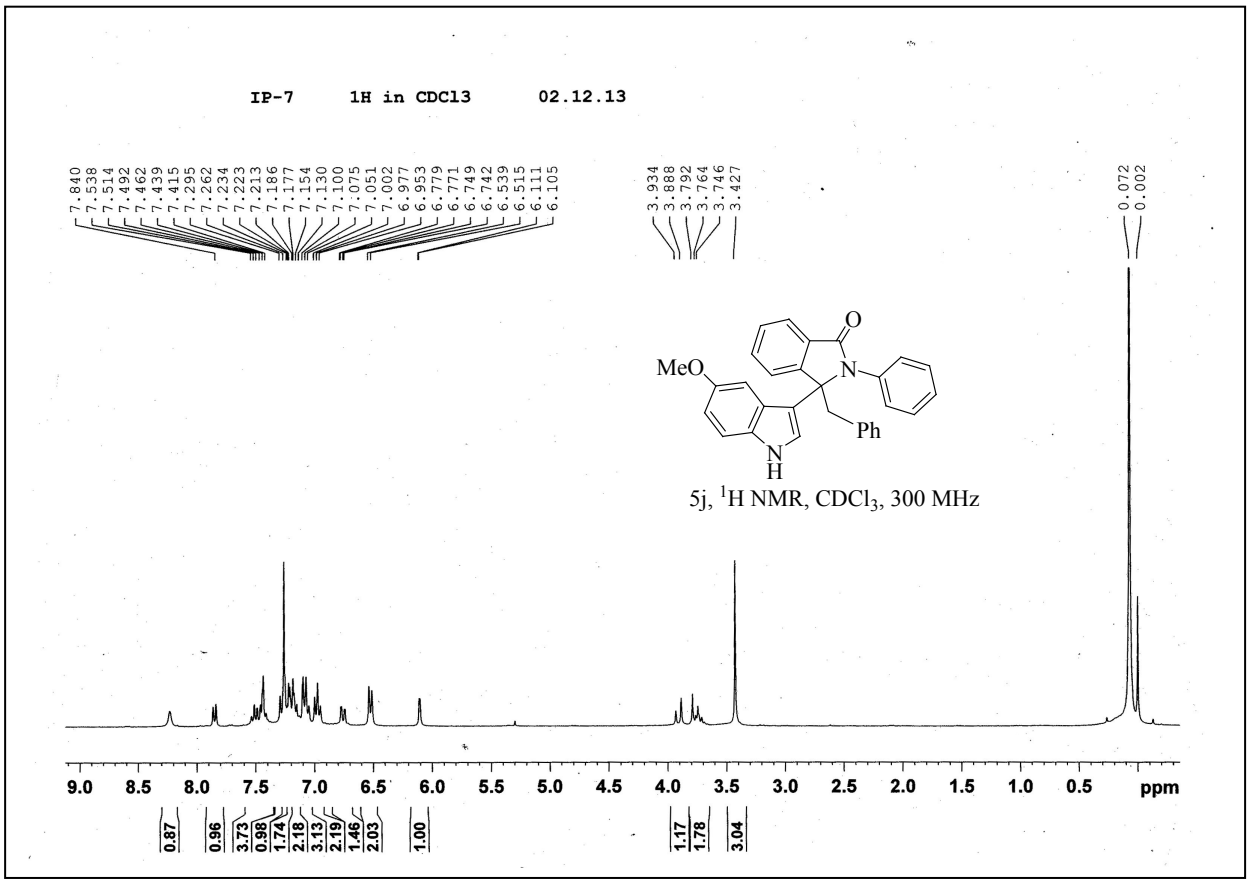


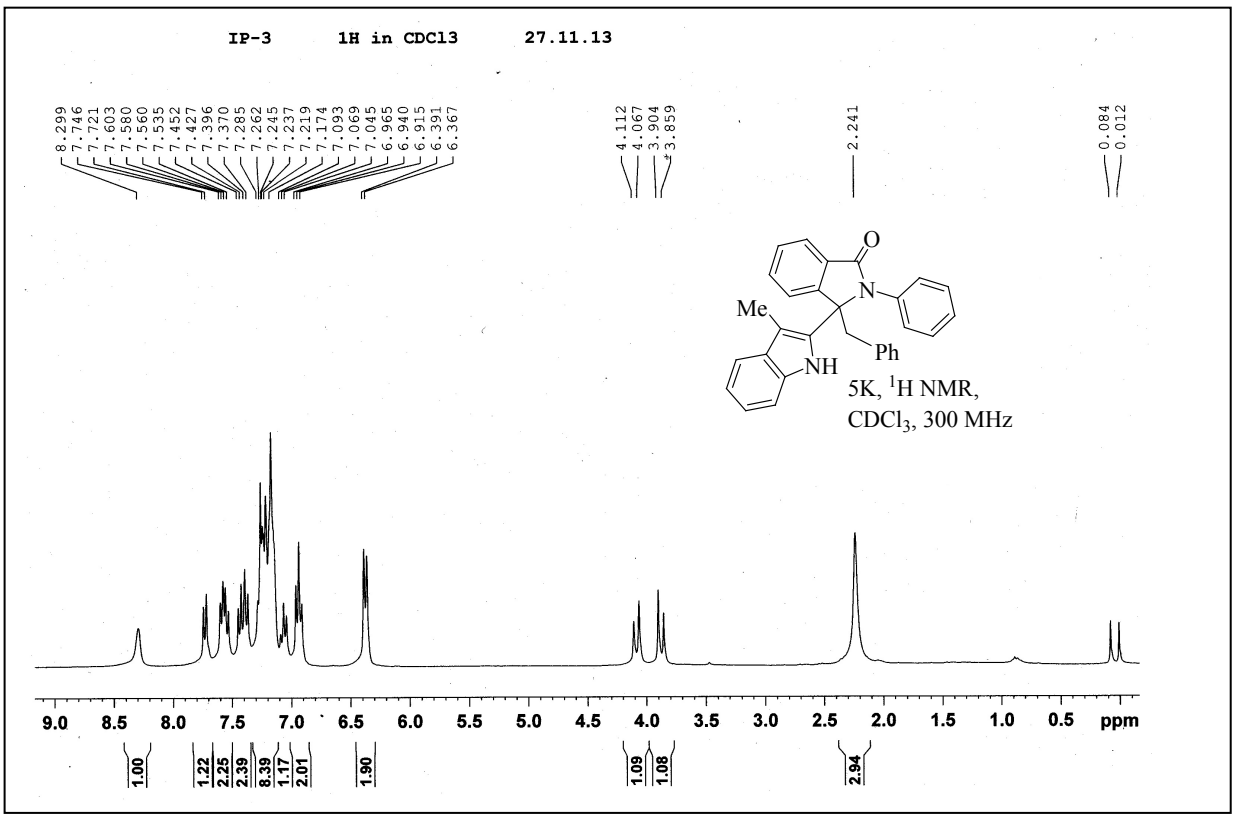


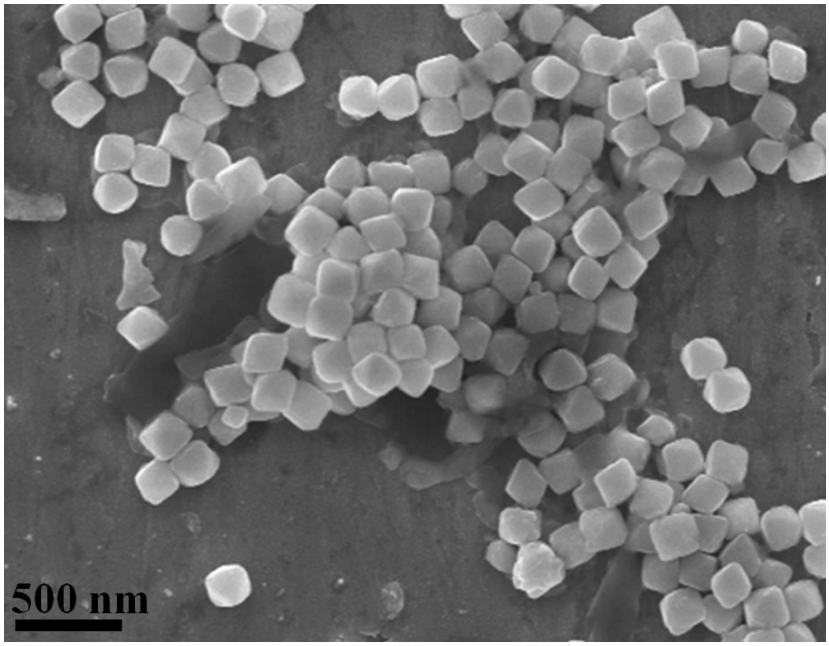
Compound 5h
^{1}H NMR, DMSO-d6
300 MHz



5i, ^{1} H NMR,
$CDCl_3$, 300 MHz


IP-7 1H in CDCl3 02.12.13
MeO
Ph
5j, ^{1}H NMR, $CDCl_3$, 300 MHz


IP-3 1H in CDCl3 27.11.13
Me
Ph
5K, ^{1}H NMR,
$CDCl_3$, 300 MHz



SEM image of the cuprous oxide nanoparticle in 500 nm scale bar

Crystal Data for **5a:**

Single crystal data for compound **5a**: $C_{25}H_{19}FN_2O$ (Mol Wt: 342.42, triclinic, space group P-1, unit cell parameters: a) 9.384(2), b) 10.889(3), c) 11.239(3), alpha 65.601(4), beta 82.825(3), gamma 74.204(2) dcalcd) 1.262 gcm^{-3}. Diffraction data were measured with MoK\a (0.71073 Å) radiation at 296 K using Kappa Apex 2 .The structure were solved direct methods using the SHELXL-97 program. Refinements of F^2^ were carried out against all reflection using SHELXL – 97. The non-hydrogen atoms were refined with anisotropic thermal parameters.The hydrogen atoms were included in geometric position and given thermal parameters equivalent to 1.2 times those of the atom to which they were attached. The final R-value were R1) 0.0643, and wR2) 0.1765. These data can be obtained free of charge via http://www.ccdc.am.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2! EZ, UK; Fax: +44-1223-336033; or deposit@ccdc.cam.ac.uk) have been deposited at the Cambridge Crystallographic Data Centre. **CCDC** 967591 (Deposition No.) contains the supplementary crystallographic data for this paper). Johnson, C.K. ORTERP II, Report ORNL-5138, Oak Ridge, National Laboratory, Tennessee, USA, 1976.