Supplementary Information

Sulfonated carbon/nano-metal oxide composites: a novel and recylable solid acid catalyst for organic synthesis in benign reaction media

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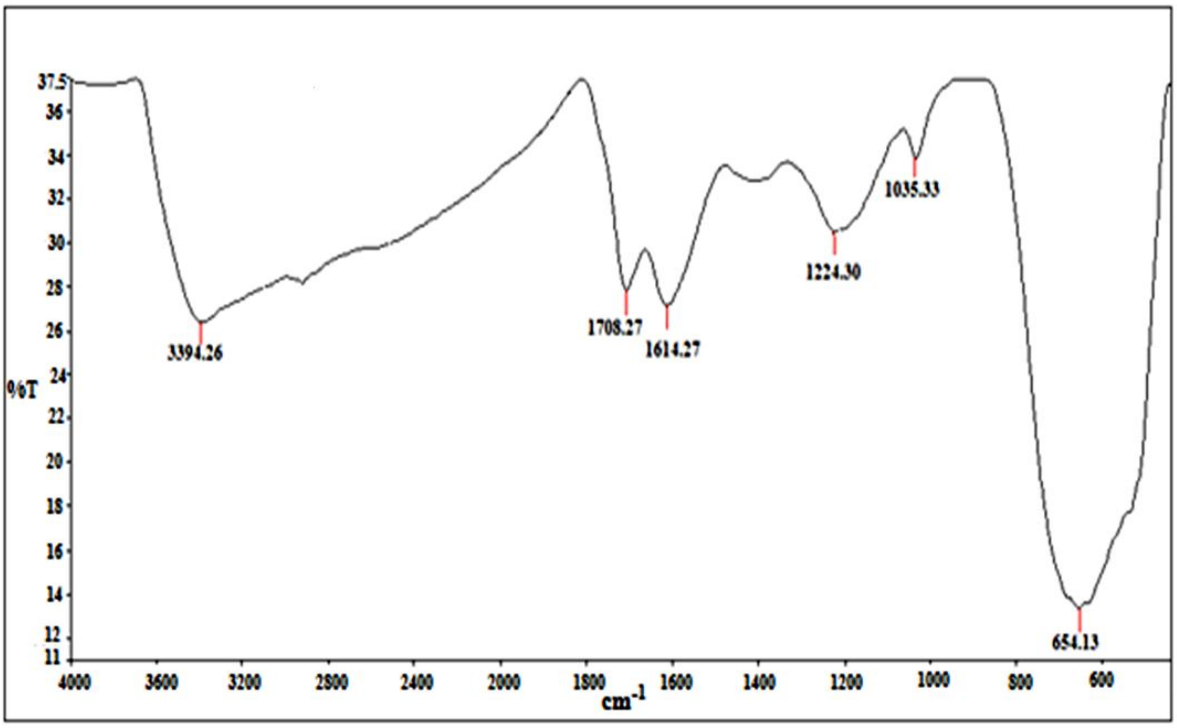
37.5
36
34
32
30
28
26
24
22
20
18
16
14
12
11
%T
3394.26
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1614.27
1224.30
1035.33
654.13
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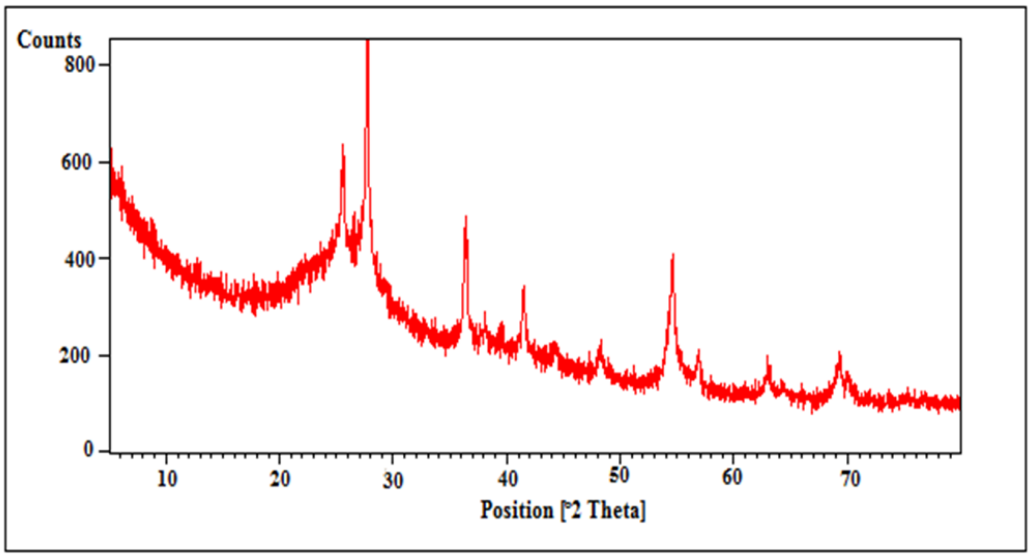
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
cm-1

Figure S2



Pos. [°2Th.]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Area [cts*°2Th.]
25.5514	0.9261	3.48338	36.35	116.06
27.6624	0.4960	3.22216	100.00	176.08
36.3124	0.3584	2.47201	51.15	68.99
41.4409	0.4419	2.17716	27.23	44.66
44.1210	1.3959	2.05093	5.33	25.71
48.2128	0.4711	1.88599	12.36	21.90
54.4965	0.6725	1.68244	53.20	131.64
56.7784	0.5042	1.62012	14.57	27.28
62.9520	0.5253	1.47527	11.55	23.54
69.1979	0.9739	1.35657	14.28	50.90

Figure S3

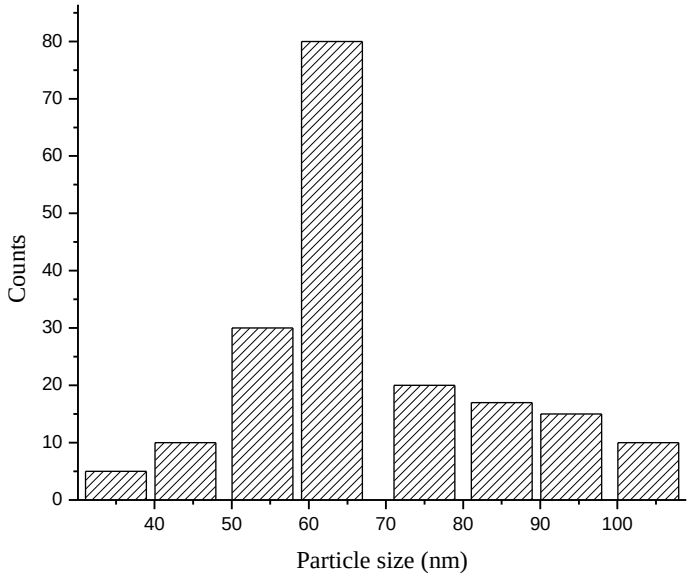


Figure S4

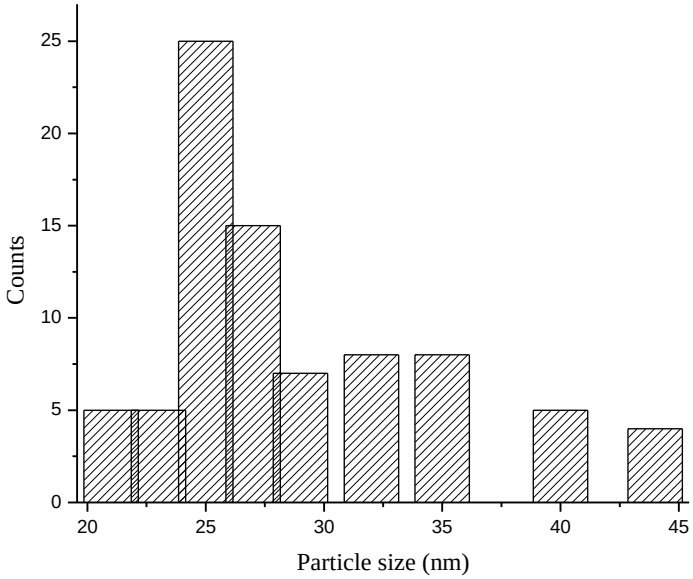


Figure S5

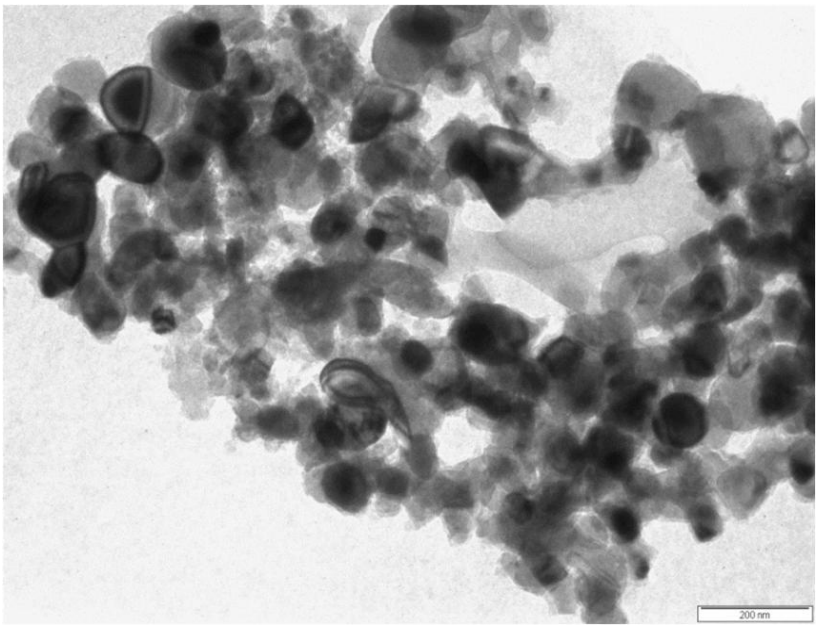


Figure S6

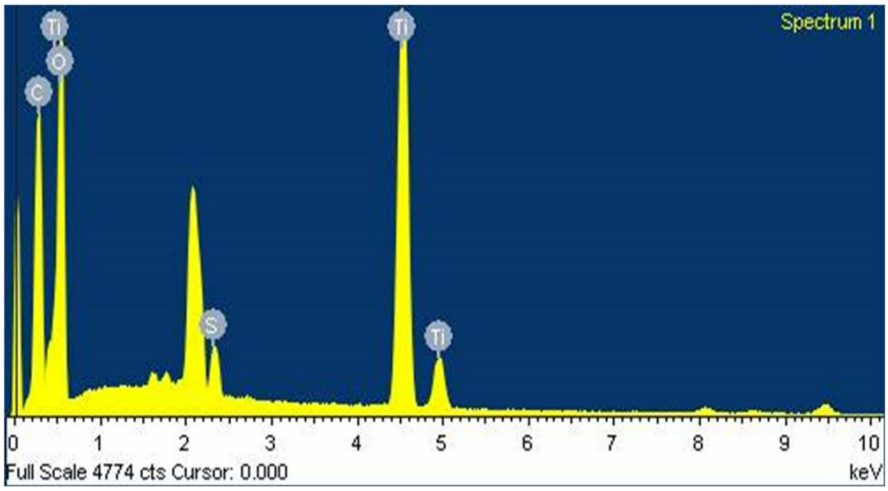
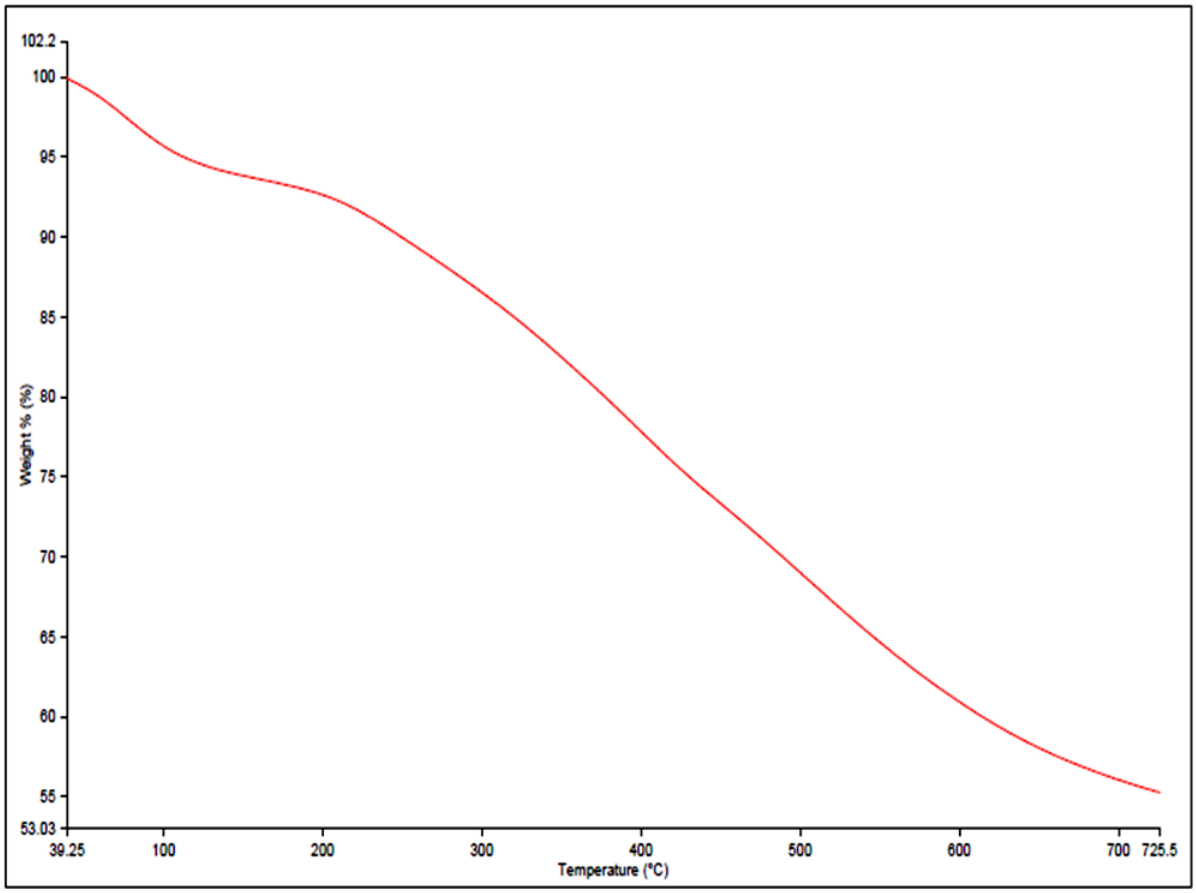
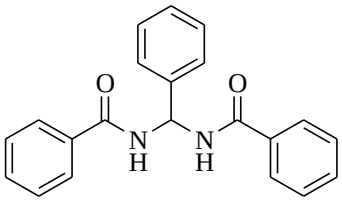


Figure S7


102.2
100
95
90
85
80
75
70
65
60
55
53.03
Weight % (%)
39.25
100
200
300
400
500
600
700
725.5
Temperature (°C)

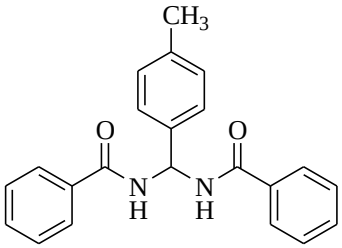
Spectral data of products 3a-o

***N,N'*-Benzylidenebisbenzamide (3a)**



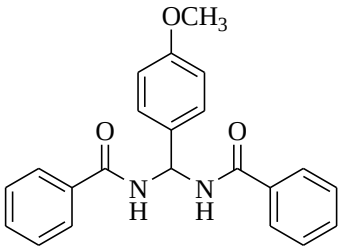
White solid; m.p./lit. m.p. 236-237/237-238 ^{o}C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3270, 3021, 2967, 2771, 1648, 1532, 1271, 1140, 1052, 803 and 694; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.03-7.07 (t, J = 8 Hz, 1H, CH), 7.31-7.35 (t, J = 8 Hz, 1H, ArH), 7.38–7.42 (t, J = 8 Hz, 2H, ArH), 7.48-7.51 (t, J = 8 Hz, 6H, ArH), 7.55-7.59 (t, J = 8 Hz, 2H, ArH), 7.91-7.93 (d, J = 8 Hz, 4H, ArH), 9.04-9.06 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 59.1, 126.9, 127.9, 128.2, 128.8, 132.1, 134.1, 140.73, 166.0; MS (LC-MS): 331.14 [M]$^{+}$.

***N,N'*-(4-Methylbenzylidene)bisbenzamide (3b)**



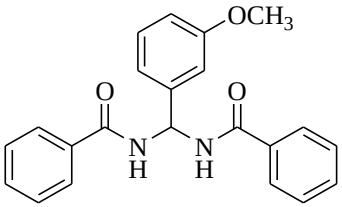
White solid; m.p./lit. m.p. 245-246/242-243 ^{o}C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3252, 3079, 2924, 2846, 1643, 1510, 1349, 1282, 1142, 1057, 821, 701; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.32 (s, 3H, CH_3), 7.00-7.04 (t, J = 8 Hz, 1H, CH), 7.20-7.22 (d, J = 8 Hz, 2H, ArH), 7.37-7.39 (d, J = 8 Hz, 2H, ArH), 7.49-7.60 (m, 6H, ArH), 7.91-7.93 (d, J = 8Hz, 4H, ArH), 8.99 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 21.2, 58.9, 126.8, 127.9, 128.8, 129.3, 132.0, 134.3, 137.3, 137.8, 165.9; MS (LC-MS): 345.16 [M]$^{+}$.

***N,N'*-(4-Methoxybenzylidene)bisbenzamide (3c)**



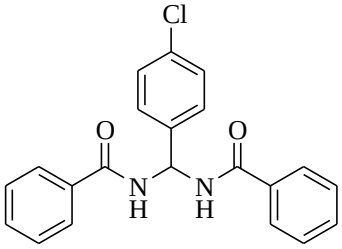
White solid; m.p./lit. m.p. 230-232/232-234 °C[2]; FTIR (KBr, ν_{max} in cm^{-1}): 3258, 3089, 2924, 2852, 1653, 1542, 1336, 1273, 1150, 1058, 828, 693; ^{1}H NMR (400 MHz, $CDCl_3$+DMSO-d_6): δ 3.71 (s, 3H, OCH_3), 6.97-7.01 (t, J = 8 Hz, 1H, CH), 7.35-7.59 (m, 10H, ArH), 7.82-7.84 (d, J = 8 Hz, 4H, ArH), 8.59-8.61 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$+DMSO-d_6): δ 55.3, 58.9, 113.7, 127.4, 128.4, 131.6, 132.6, 134.1, 159.1, 166.2; MS (LC-MS): 361.15 $[M]^+$.

***N,N'*-(3-Methoxybenzylidene)bisbenzamide (3d)**



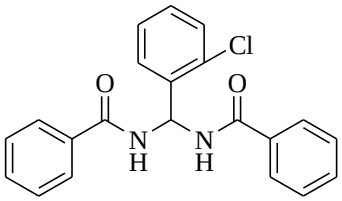
White solid; m.p./lit. m.p. 185-187/188–190 °C[3]; FTIR (KBr, ν_{max} in cm^{-1}): 3280, 3079, 2981, 2773, 1646, 1520, 1276, 1140, 1078, 831, 680; ^{1}H NMR (400 MHz, $CDCl_3$+DMSO-d_6): δ 3.74 (s, 3H, OCH_3), 6.84-6.88 (t, J = 8 Hz, 1H, CH), 7.01-7.08 (m, 3H, ArH), 7.30-7.34 (t, J = 8 Hz, 1H, ArH), 7.51-7.59 (m, 6H, ArH), 7.90-7.91 (d, J = 8 Hz, 4H, ArH), 9.04-9.06 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$+DMSO-d_6): δ 55.5, 58.9, 112.8, 119.1, 127.9, 128.6, 129.8, 131.9, 134.3, 142.3, 159.7, 166.0; MS (LC-MS): 361.14 $[M]^+$.

***N,N'*-(4-Chlorobenzylidene)bisbenzamide (3e)**



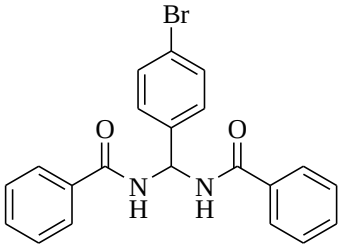
White solid; m.p./lit. m.p. 228-229/230-232 ^{o}C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3261, 3086, 2983, 2775, 1649, 1546, 1344, 1276, 1141, 1087, 825, 692; ^{1}H NMR (400 MHz, DMSO-d_6): δ 6.99-7.03 (t, J = 8 Hz, 1H, CH), 7.46-7.60 (m, 10H, ArH), 7.92-7.94 (d, J = 8 Hz, 4H, ArH), 9.08-9.10 (d, 2H, J = 8 Hz, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 58.8, 127.9, 128.0, 128.6, 128.7, 128.8, 129.0, 132.1, 132.7, 134.1, 139.6, 166.2; MS (LC-MS): 366 $[M]^+$, 368 $[M+2]^+$.

***N,N'*-(2-Chlorobenzylidene)bisbenzamide (3f)**



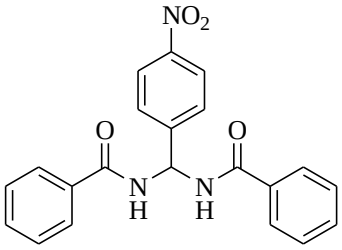
White solid; m.p./lit. m.p. 245-246/242-244 ^{o}C[4]; FTIR (KBr, ν_{max} in cm^{-1}): 3280, 3082, 2914, 2776, 1649, 1509, 1345, 1272, 1223, 1052, 830 and 705; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.16-7.20 (t, J = 8 Hz, 1H, CH), 7.38-7.40 (t, J = 4 Hz, 2H, ArH), 7.46-7.50 (t, J = 8 Hz, 5H, ArH), 7.54-7.58 (t, J = 4 Hz, 2H, ArH), 7.66-7.68 (d, J = 8 Hz, 1H, ArH), 7.93-7.95 (d, J = 8Hz, 4H, ArH), 9.11-9.13 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 57.7, 127.5, 128.1, 128.7, 128.9, 129.9, 130.0, 132.0, 132.9, 134.2, 166.5; MS (LC-MS): 366 $[M]^+$, 368 $[M+2]^+$.

***N,N'*-(4-Bromobenzylidene)bisbenzamide (3g)**



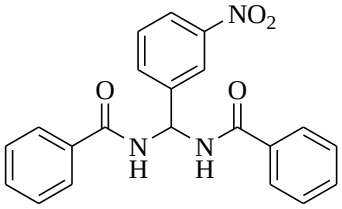
White solid; m.p./lit. m.p. 255-257/259-260 °C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3285, 3072, 2974, 2749, 1638, 1542, 1480, 1345, 1272, 1202, 1142, 1078, 824, 798, 570; ^{1}H NMR (400MHz, DMSO-d_6): δ 6.72-6.76 (t, J = 8 Hz, 1H, CH), 7.40-7.59 (m, 10H, ArH), 7.88-7.90 (d, J = 8 Hz, 4H, ArH), 8.15-8.17 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 57.4, 121.3, 127.2, 128.9, 129.4, 131.8, 132.1, 134.2, 142.9, 168.1; MS (LC-MS): 410 $[M]^+$, 412 $[M+2]^+$.

***N,N'*-(4-Nitrobenzylidene)bisbenzamide (3h)**



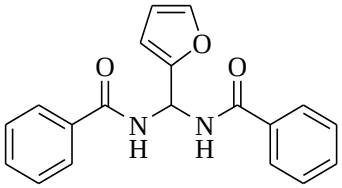
White solid; m.p./lit. m.p. 267-269/265-267 °C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3305, 3080, 2974, 2875, 1690, 1610, 1350, 1262, 1198, 1142, 1078, 870, 798, 719, 570; ^{1}H NMR (400MHz, DMSO-d_6): δ 7.07-7.11 (t, J = 8 Hz, 1H, CH), 7.49-7.60 (m, 6H, ArH), 7.76-7.78 (d, J = 8 Hz, 2H, ArH), 7.94-7.96 (d, J = 8 Hz, 4H, ArH), 8.27-8.29 (d, J = 8 Hz, 2H, ArH), 9.24-9.26 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 56.6, 123.9, 124.0, 128.0, 128.5, 128.8, 133.4, 147.0, 147.5, 166.3; MS (LC-MS): 376.13 $[M]^+$.

***N,N'*-(3-Nitrobenzylidene)bisbenzamide (3i)**



White solid; m.p./lit. m.p. 195-196/190-192 °C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3284, 3058, 2836, 1662, 1540, 1481, 1344, 1272, 1278, 1201, 1135, 1076, 885, 792, 651, 576; ^{1}H NMR (400MHz, DMSO-d_6): δ 7.07-7.10 (t, J = 8 Hz, 1H, CH), 7.49-7.60 (m, 6H, ArH), 7.69-7.73 (t, J = 8 Hz, 1H, ArH), 7.93-7.96 (m, 5H, ArH), 8.21-8.23 (d, J = 8 Hz, 1H, ArH), 8.34 (s, 1H, ArH), 9.26-9.28 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 59.3, 121.8, 123.2, 128.0, 128.8, 130.4, 132.2, 134.0, 142.8, 148.2, 166.3; MS (LC-MS): 376.12 $[M]^+$.

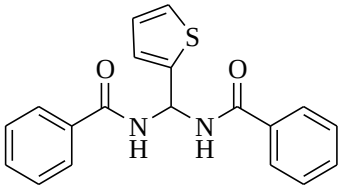
***N,N'*-(Furan-2-ylmethylene)bisbenzamide (3j)**



Yellowish solid; m.p./lit. m.p. 202-204/206-208 °C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3277, 3050, 2928, 1652, 1548, 1488, 1340, 1277, 1201, 1137, 1067, 800, 706, 650; ^{1}H NMR (400MHz,

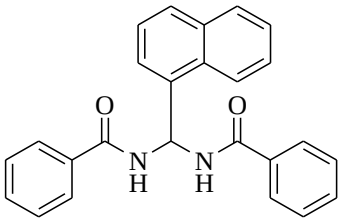
DMSO-d_6): δ 7.01-7.05 (t, J = 8 Hz, 1H, CH), 7.26-7.50 (m, 9H, ArH), 7.86-7.88 (d, J = 8 Hz, 4H, ArH), 8.96-8.98 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 59.1, 119.8, 126.7, 127.8, 128.9, 130.3, 134.2, 140.8, 144.5, 152.9, 165.8; MS (LC-MS): 321.12 $[M]^+$.

***N,N'*-(Thiophen-2-ylmethylene)bisbenzamide (3k)**



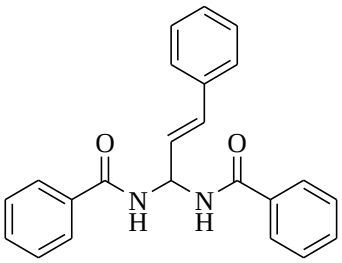
Yellowish solid; m.p./lit. m.p. 214-215/208-210 ^oC[2]; FTIR (KBr, ν_{max} in cm^{-1}): 3263, 3061, 2925, 2842, 1635, 1530, 1516, 1348, 1239, 1148, 1036, 937, 814, 702; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.00-7.05 (t, J = 8 Hz, 1H, CH), 7.14-7.16 (d, J = 8 Hz, 1H, ArH), 7.23-7.27 (t, J = 8 Hz, 1H, ArH), 7.49-7.60 (m, 7H, ArH) , 7.90-7.92 (d, J = 8 Hz, 4H, ArH), 9.15-9.17 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 55.7, 125.4, 125.9, 127.4, 127.9, 128.9, 132.2, 134.1, 166.0; MS (LC-MS): 337.10 $[M]^+$.

***N,N'*-(Napthylidene)bisbenzamide (3l)**



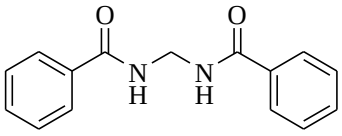
White solid; m.p. 286-288 ^oC; FTIR (KBr, ν_{max} in cm^{-1}): 3276, 3145, 2908, 1664, 1567, 1521, 1369, 1276, 1174, 1087, 981, 894, 607, 570; ^{1}H NMR (400 MHz, $CDCl_3$+DMSO-d_6): δ 7.36-7.52 (m, 9H, CH and ArH), 7.23-7.81 (m, 4H, ArH), 7.89-7.91 (d, J = 8 Hz, 4H, ArH), 8.13-8.17 (t, J = 8 Hz, 1H, ArH), 8.92-8.94 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$+DMSO-d_6): δ 56.9, 123.5, 123.8, 125.3, 125.9, 126.7, 127.8, 128.3, 128.7, 130.9, 131.6, 133.8, 134.2, 166.2; MS (LC-MS): 381.16 $[M]^+$.

***N,N'*-(3-Phenyl-2-ene-propylene)bisbenzamide (3m)**



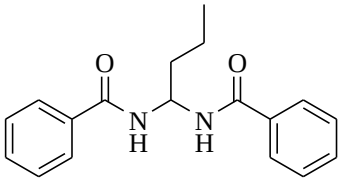
Brownish solid; m.p./lit. m.p. 195-198/199-201 ^{o}C[5]; FTIR (KBr, ν_{max} in cm^{-1}): 3274, 3237, 2986, 2922, 1647, 1543, 1504, 1484, 1419, 1299, 1146, 1079, 1032, 756, 690; ^{1}H NMR (400 MHz, $CDCl_3$+DMSO-d_6): δ 6.65 (m, 2H, CH), 6.69-6.72 (d, J = 12 Hz, 1H, CH), 7.28 -7.32 (t, J = 8 Hz, 1H, ArH), 7.35 (m, 2H, ArH), 7.48 (m, 4H, ArH), 7.56-7.60 (t, J = 8 Hz, 2H, ArH), 7.86-7.90 (t, J = 8 Hz, 2H, ArH), 7.92-7.94 (d, J = 8 Hz, 4H, ArH), 8.87-7.89 (d, J = 8Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$+DMSO-d_6): δ 56.3, 58.4, 118.0, 126.7, 126.8, 126.9, 127.6, 127.8, 128.1, 128.2, 128.3, 128.4, 128.5, 128.7, 143.6, 166.02, 166.6, 168.9; MS (LC-MS): 357.16 $[M]^{+}$.

***N,N'*-Methylene bisbenzamide (3n)**



White solid; m.p./lit. m.p. 215-216/216-218 ^{o}C[1]; FTIR (KBr, ν_{max} in cm^{-1}): 3376, 3233, 3171, 2978, 2925, 1651, 1577, 1526, 1482, 1405, 1293, 1142, 1076, 1024, 978, 892, 819, 712; ^{1}H NMR (400MHz, DMSO-d_6): δ 4.85-4.89 (m, 2H, CH_2), 7.44-7.57 (m, 6H, ArH), 7.89-7.92 (t, J = 8 Hz, 4H, ArH), 9.08-9.10 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 31.2, 127.9, 128.7, 131.9, 134.7, 166.2; MS (LC-MS): 255.11 $[M]^{+}$.

***N,N'*-(*n*-Propylmethylene)bisbenzamide (3o)**

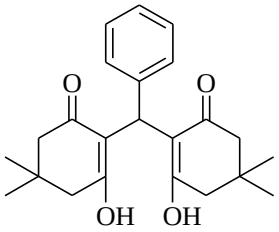


Yellowish solid; m.p./lit. m.p. 166-168/171-172 ^{o}C[5]; FTIR (KBr, ν_{max} in cm^{-1}): 3370, 3239, 3172, 2971, 2923, 1652, 1571, 1521, 1481, 1402, 1295, 1143, 1078, 1025, 979, 894, 817,

711; ^{1}H NMR (400 MHz, DMSO-d_6): δ 0.86-0.89 (t, J = 8 Hz, 3H, CH_3), 1.34-1.42 (m, 2H, CH_2), 1.83-1.89 (q, J = 8 Hz, 2H, CH_2) 5.70-5.78 (quin, J = 8 Hz, 1H, CH), 7.40-7.54 (m, 6H, ArH), 7.87-7.89 (d, J = 8 Hz, 4H), 8.02-8.04 (d, J = 8 Hz, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 13.7, 29.7, 35.8, 49.0, 127.9, 128.6, 131.6, 134.7, 168.7; MS (LC-MS): 297.16 $[M]^+$.

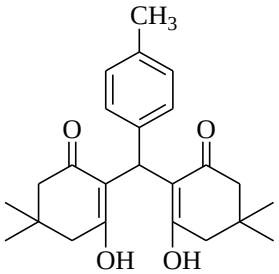
Spectral data of products 5a-j

2,2'-(Phenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5a)



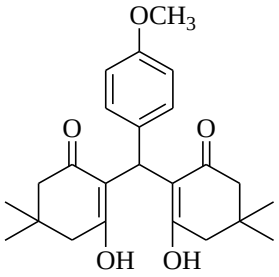
White solid; m.p./lit. m.p. 198-199/199-201 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3436, 2871, 2968, 1724, 1592, 796, 684; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.13 (s, 6H, 2×CH_3), 1.26 (s, 6H, 2×CH_3), 2.36-2.43 (m, 8H, 4×CH_2), 5.57 (s, 1H, CH), 7.11-7.13 (d, J = 8 Hz, 2H, ArH) 7.18-7.22 (t, J = 8 Hz, 1H, ArH), 7.28-7.32 (t, J = 8 Hz, 2H, ArH), 11.94 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.5, 31.4, 32.6, 46.4, 46.9, 115.5, 125.8, 126.7, 128.2, 137.9, 189.4, 190.4; MS (ESI): 369.10 $[M]^+$.

2,2'-(4-Methylphenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5b)



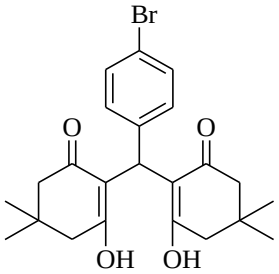
White solid; m.p./lit. m.p. 140-142/141-143 °C[7]; FTIR (KBr, ν_{max} in cm^{-1}): 3433, 2863, 2961, 1724, 1506, 769, 657; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.07 (s, 6H, 2×CH_3), 1.24 (s, 6H, 2×CH_3), 2.27 (s, 3H, CH_3), 2.39-2.44 (m, 8H, 4×CH_2), 5.49 (s, 1H, CH), 6.98-7.00 (d, J = 8 Hz, 2H, ArH), 7.08-7.10 (d, J = 8 Hz, 2H, ArH), 11.93 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.2, 30.9, 31.4, 32.7, 46.4, 54.1, 57.3, 115.7, 128.7, 126.6, 128.9, 134.9, 135.2, 189.2, 192.3; MS (ESI): 383.13 $[M]^+$.

2,2'-(4-Methoxyphenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5c)



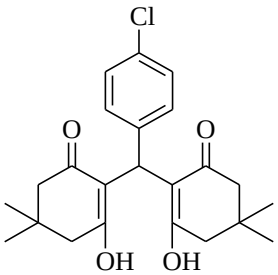
White solid; m.p./lit. m.p. 149-150/146-148 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3449, 2865, 2956, 1602, 775, 680; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.01 (s, 6H, 2×CH_3), 1.12 (s, 6H, 2×CH_3), 2.21–2.47 (m, 8H, 4×CH_2), 3.75 (s, 3H, OCH_3), 4.75 (s, 1H, CH), 6.76-6.79 (d, J = 8 Hz, 2H, ArH), 7.21-7.23 (d, J = 8 Hz, 2H, ArH), 11.94 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.2, 31.3, 32.0, 40.9, 50.8, 55.1, 113.5, 113.6, 115.8, 127.8, 129.3, 136.5, 182.0, 196.5; MS (ESI): 399.14 $[M]^+$.

2,2'-(4-Bromophenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5d)



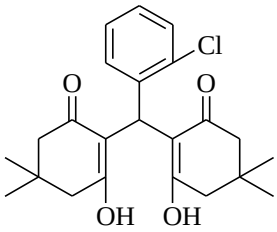
White solid; m.p./lit. m.p. 220-222/221-223 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3432, 2859, 2958, 1597, 764, 672; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.12 (s, 6H, 2×CH_3), 1.24 (s, 6H, 2×CH_3), 2.31–2.50 (m, 8H, 4×CH_2), 5.47 (s, 1H, CH), 6.97-6.99 (d, J = 8 Hz, 2H, ArH), 7.39-7.41 (d, J = 8 Hz, 2H, ArH), 11.91 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.4, 29.6, 31.4, 32.5, 46.4, 47.0, 115.3, 119.6, 128.6, 131.3, 137.3, 189.4, 190.7; MS (ESI): 448 $[M]^+$, 450 [M+2].

2,2'-(4-Chlorophenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5e)



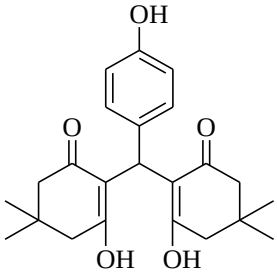
White solid; m.p./lit. m.p. 146-148/145-147 °C[7]; FTIR (KBr, ν_{max} in cm^{-1}): 3431, 2860, 2952, 1601, 789, 632; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.13 (s, 6H, 2×CH_3), 1.24 (s, 6H, 2×CH_3), 2.32–2.51 (m, 8H, 4×CH_2), 5.50 (s, 1H, CH), 7.03-7.05 (d, J = 8 Hz, 2H, ArH), 7.25-7.27 (d, J = 8 Hz, 2H, ArH), 11.91 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.4, 29.6, 31.4, 32.4, 46.4, 47.0, 115.4, 128.2, 128.4, 131.6, 136.7, 189.5, 190.7; MS (ESI): 403 $[M]^+$, 405 $[M+2]^+$.

2,2'-(2-Chlorophenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5f)



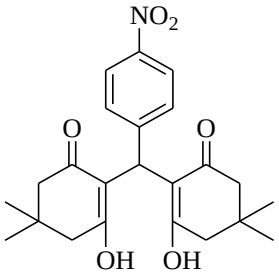
White solid; m.p./lit. m.p. 199-201/202-204 °C[7]; FTIR (KBr, ν_{max} in cm^{-1}): 3431, 2856, 2952, 1596, 786, 694; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.08 (s, 6H, 2×CH_3), 1.16 (s, 6H, 2×CH_3), 2.19–2.48 (m, 8H, 4×CH_2), 5.64 (s, 1H, CH), 7.15-7.18 (t, J = 8 Hz, 1H, ArH), 7.21–7.25 (t, J = 8 Hz, 1H, ArH), 7.31-7.33 (d, J = 8 Hz, 1H, ArH), 7.39-7.41 (d, J = 8 Hz, 1H, ArH), 11.91 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.9, 28.8, 31.5, 32.1, 46.5, 47.0, 115.7, 126.4, 127.6, 129.4, 130.3, 133.5, 136.6, 189.7; MS (ESI): 403 $[M]^+$, 405 $[M+2]^+$.

2,2'-(4-Hydroxyphenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5g)



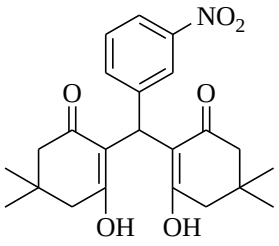
White solid, m.p./lit. m.p. 200-202/201-203 °C[7]; FTIR (KBr, ν_{max} in cm^{-1}): 3439, 2867, 2963, 1594, 768, 687; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.12 (s, 6H, 2×CH_3), 2.01 (s, 6H, 2×CH_3), 2.24–2.53 (m, 8H, 4×CH_2), 5.69 (s, 1H, CH), 7.17-7.19 (d, J = 8 Hz, 2H, ArH), 6.84-6.86 (d, J = 8 Hz, 2H, ArH), 10.49 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.5, 28.9, 41.2, 47.8, 52.6, 111.8, 116.7, 129.6, 130.2, 133.2, 154.5, 180.6, 196.4; MS (ESI): 385 $[M]^+$.

2,2'-(4-Nitrophenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5h)



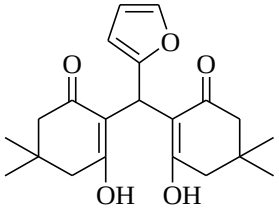
White solid; m.p./lit. m.p. 194-196/195-197 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3447, 2875, 2959, 1598, 769, 658; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.01 (s, 6H, 2×CH_3), 1.14 (s, 6H, 2×CH_3), 2.17–2.47 (m, 8H, 4×CH_2), 5.54 (s, 1H, CH), 7.48-7.50 (d, *J* = 8Hz, 2H, ArH), 8.10-8.12 (d, *J* = 8Hz, 2H, ArH), 11.91 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.2, 32.1, 33.2, 46.7, 50.1, 114.6, 123.5, 128.6, 146.5, 188.9, 190.8; MS (ESI): 414 $[M]^+$.

2,2'-(3-Nitrophenylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5i)



White solid; m.p./lit. m.p. 200-202/201-203 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3411, 2884, 2964, 1599, 775, 659; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.14 (s, 6H, 2×CH_3), 1.29 (s, 6H, 2×CH_3), 2.33–2.54 (m, 8H, 4×CH_2), 5.56 (s, 1H, CH), 7.41–7.48 (m, 2H, ArH), 8.02 (s, 1H, ArH), 8.05-8.07 (d, *J* = 8 Hz, 1H, ArH), 11.90 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.7, 30.9, 31.4, 32.9, 46.5, 46.9, 114.8, 121.2, 122.4, 129.3, 132.9, 140.7, 148.4, 189.6, 191.1; MS (ESI): 414 $[M]^+$.

2,2'-(Furan-2-ylmethylene)bis(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (5j)

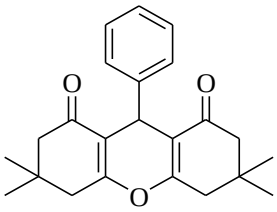


White solid; m.p./lit. m.p. 141-143/142-144 °C[8]; FTIR (KBr, ν_{max} in cm^{-1}): 3434, 2855, 2959, 1601, 769, 684; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.84 (s, 6H, 2×CH_3), 2.16 (s, 6H, 2×CH_3),

2.21–2.47 (m, 8H, 4×CH_2), 4.41 (s, 1H, CH), 5.87-5.89 (d, *J* = 8 Hz, 1H, ArH), 6.17-6.21 (t, *J* = 8 Hz 1H, ArH), 7.23-7.25 (d, *J* = 8 Hz, 1H, ArH), 12.37 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.5, 29.4, 31.3, 46.7, 52.4, 106.9, 111.2, 115.6, 131.1, 143.2, 154.5, 189.5, 192.6; MS (ESI): 359 $[M]^+$.

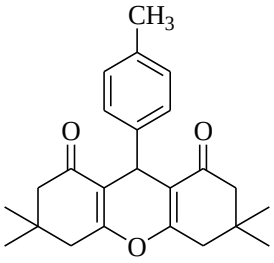
Spectral data of compounds 6a-j

3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxo-octahydroxanthene (6a)



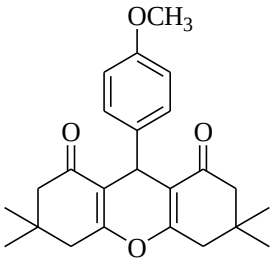
White solid; m.p./lit. m.p. 199-200/203-204 ^oC[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3350, 2980, 1725, 1699, 1520, 1360, 1260, 1195, 843; ^{1}H NMR (400 MHz, $CDCl_3$): δ1.01 (s, 6H, 2×CH_3), 1.12 (s, 6H, 2×CH_3), 2.17-2.28 (q, 4H, 2×CH_2), 2.49 (s, 4H, 2×CH_2), 4.77 (s, 1H, CH), 7.10-7.14 (t, *J* = 8 Hz, 1H, ArH), 7.22-7.25 (t, *J* = 8Hz, 2H, ArH), 7.30-7.32 (d, *J* = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.3, 31.8, 32.2, 40.9, 50.8, 115.7, 126.4, 128.0, 128.4, 143.9, 162.4, 196.7; MS (ESI): 351.19 $[M]^+$.

3,3,6,6-Tetramethyl-9-(4-Tolyl)-1,8-dioxo-octahydroxanthene (6b)



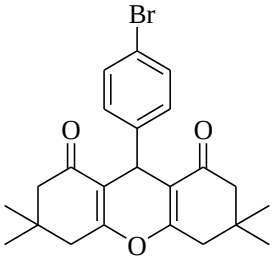
White solid; m.p./lit. m.p. 205-206/208-209 ^oC[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3498, 2933, 1719, 1645, 1577, 1279, 1384, 1166, 850; ^{1}H NMR (400 MHz, $CDCl_3$): δ1.02 (s, 6H, 2×CH_3), 1.11 (s, 6H, 2×CH_3), 2.18-2.27 (q, 4H, 2×CH_2), 2.30 (s, 3H, CH_3), 2.48 (s, 4H, 2×CH_2), 4.76 (s, 1H, CH), 7.02-7.04 (d, *J* = 8Hz, 2H, ArH), 7.18-7.20 (d, *J* = 8Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 21.0, 27.4, 29.3, 31.4, 32.2, 40.9, 50.8, 115.8, 128.2, 128.8, 135.8, 141.2, 162.1, 196.4; MS (ESI): 365.21 $[M]^+$.

3,3,6,6-Tetramethyl-9-(4-Methoxyphenyl)-1,8-dioxo-octahydroxanthene (6c)



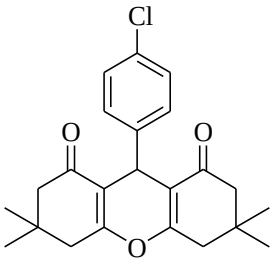
White solid; m.p./lit. m.p. 246-248/242-244 ^{o}C[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3025, 2980, 1685, 1513, 1450, 1375, 1260, 1170, 1032, 840; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.03 (s, 6H, 2×CH_3), 1.09 (s, 6H, 2×CH_3), 2.16-2.27 (q, 4H, 2×CH_2), 2.46 (s, 4H, 2×CH_2), 3.75 (s, 3H, OCH_3), 4.70 (s, 1H, CH), 6.78-6.80 (d, J = 8 Hz, 2H, ArH), 7.21-7.23 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.3, 30.9, 32.2, 40.9, 50.8, 55.1, 113.5, 115.8, 129.3, 136.5, 157.9, 162.1, 196.5; MS (ESI): 381.20 $[M]^{+}$.

3,3,6,6-Tetramethyl-9-(4-Bromoophenyl)-1,8-dioxo-octahydroxanthene (6d)



White solid; m.p./lit. m.p. 238-240/240-241 ^{o}C[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3498, 2933, 2929, 1719, 1645, 1577, 1384, 1166, 840; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.02 (s, 6H, 2×CH_3), 1.11 (s, 6H, 2× CH_3), 2.16-2.27 (q, 4H, 2×CH_2), 2.48 (s, 4H, 2×CH_2), 4.74 (s, 1H, CH), 7.19–7.28 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.3, 31.5, 32.2, 40.8, 50.7, 115.3, 128.2, 129.8, 132.0, 142.7, 162.4, 196.3; MS (ESI): 429 $[M]^{+}$, 431 $[M+2]^{+}$.

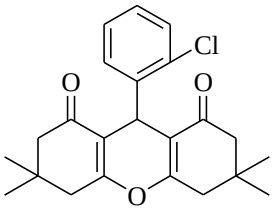
3,3,6,6-Tetramethyl-9-(4-Chlorophenyl)-1,8-dioxo-octahydroxanthene (6e)



White solid; m.p./lit. m.p. 235-236/237-238 ^{o}C[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3496, 2956, 2957, 1718, 1643, 1578, 1386, 1165, 840; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.01 (s, 6H, CH_3), 1.13

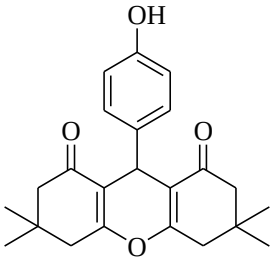
(s, 6H, CH_3), 2.17-2.28 (q, 4H, CH_2), 2.48 (s, 4H, CH_2), 4.73 (s, 1H, CH), 7.19-7.26 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.3, 31.5, 32.2, 40.8, 50.6, 115.3, 128.1, 129.7, 132.0, 142.6, 162.7, 196.4; MS (ESI): 386 $[M]^+$, 388 $[M+2]^+$.

3,3,6,6-Tetramethyl-9-(2-Chlorophenyl)-1,8-dioxo-octahydroxanthene (6f)



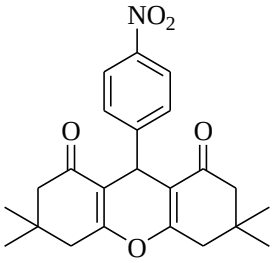
White solid, m.p./lit. m.p. 220-222/225-226 ^oC[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3392, 2960, 1719, 1664, 1595, 1380, 1166, 850, 650; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.04 (s, 6H, 2×CH_3), 1.12 (s, 6H, 2×CH_3), 2.16-2.27 (q, 4H, 2×CH_2), 2.47 (s, 4H, 2×CH_2), 5.02 (s, 1H, CH), 7.06-7.10 (t, *J* = 8 Hz, 1H, ArH), 7.17-7.21 (t, *J* = 8 Hz, 1H, ArH), 7.24-7.26 (d, *J* = 8 Hz, 1H, ArH), 7.45-7.47 (d, *J* = 8 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.4, 29.3, 31.9, 40.9, 50.7, 113.6, 126.2, 128.1, 130.4, 133.0, 139.8, 141.5, 163.0, 196.6; MS (ESI): 386 $[M]^+$, 388 $[M+2]^+$.

3,3,6,6-Tetramethyl-9-(4-Hydroxyphenyl)-1,8-dioxo-octahydroxanthene (6g)



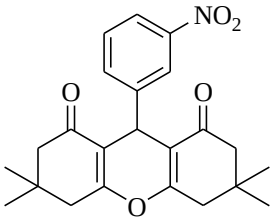
White solid; 253-255/250-251 ^oC[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3694, 3565, 2972, 1665, 1508, 1355, 1169, 842, 760, 690; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.05 (s, 6H, 2 ×CH_3), 1.11 (s, 6H, 2×CH_3), 2.12-2.25 (q, 4H, 2×CH_2), 2.49 (s, 4H, 2×CH_2), 4.69 (s, 1H, CH), 4.89 (s, OH, exchangeable with D_2O), 6.60-6.62 (d, *J* = 8Hz, 2H, ArH), 7.11-7.13 (d, *J* = 8Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.5, 29.3, 30.9, 32.4, 40.8, 50.8, 115.2, 115.8, 129.4, 135.8, 154.5, 162.3, 197.1; MS (ESI): 367.19 $[M]^+$.

3,3,6,6-Tetramethyl-9-(4-Nitrophenyl)-1,8-dioxo-octahydroxanthene (6h)



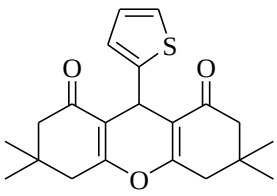
White solid; m.p./lit. m.p. 243-246/250-251 ^{o}C[9]; FTIR (KBr, ν_{max} in cm^{-1}): 3151, 2972, 1665, 1508, 1355, 1169, 842, 760, 690; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.02 (s, 6H, 2×CH_3), 1.14 (s, 6H, 2×CH_3), 2.17-2.30 (q, 4H, 2×CH_2), 2.52 (s, 4H, 2×CH_2), 4.85 (s, 1H, CH), 7.48-7.51 (d, J = 8 Hz, 2H, ArH), 8.11-8.13 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.3, 29.4, 32.2, 32.3, 41.1, 50.6, 114.7, 123.6, 129.2, 146.5, 151.7, 162.9, 196.4; MS (ESI): 396.18 $[M]^{+}$.

3,3,6,6-Tetramethyl-9-(3-Nitrophenyl)-1,8-dioxo-octahydroxanthene (6i)



White solid; m.p./lit. m.p. 169-170/170-172 ^{o}C[9]; FTIR (KBr, ν_{max} in cm^{-1}): 2960, 2873, 1680, 1593, 1377, 1251, 1157, 1042, 842, 665; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.01 (s, 6H, 2×CH_3), 1.13 (s, 6H, 2×CH_3), 2.16-2.31 (q, 4H, 2×CH_2), 2.48 (s, 4H, 2×CH_2), 4.81 (s, 1H, CH), 7.41-7.45 (t, J = 8 Hz, 1H, ArH), 7.81-7.83 (d, J = 8Hz, 1H, ArH), 7.98-8.02 (t, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.4, 29.4, 32.1, 32.3, 40.8, 50.6, 114.5, 121.7, 122.5, 128.8, 146.3, 148.3, 163.0, 196.4; MS (ESI): 396.18 $[M]^{+}$.

3,3,6,6-Tetramethyl-9-(2-Thienyl)-1,8-dioxo-octahydroxanthene (6j)

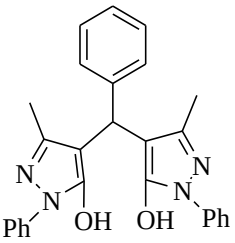


Brown solid; m.p./lit. m.p. 167-168/164-165 ^{o}C[10]; FTIR (KBr, ν_{max} in cm^{-1}): 2955, 2896, 2871, 1659, 1590, 1371, 1039, 857, 659; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.02 (s, 6H, 2×CH_3),

1.11 (s, 6H, 2×CH_3), 2.14-2.28 (q, 4H, 2×CH_2), 2.53 (s, 4H, 2×CH_2), 4.91 (s, 1H, CH), 6.78 (m, 1H, ArH), 7.82-7.97 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.4, 29.4, 32.1, 32.3, 40.8, 50.6, 114.55, 121.7, 122.5, 128.8, 146.3, 148.3, 163.0, 196.4; MS (ESI): 357.15 $[M]^+$.

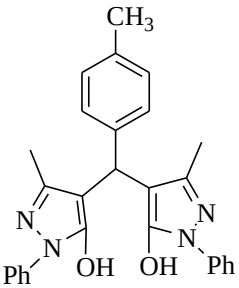
Spectral data of 7a-j

4,4′-(Phenylmethylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7a)



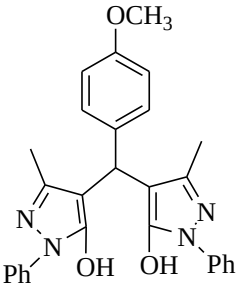
White solid; m.p./lit. m.p. 170−172/171−172 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3515, 3129, 3081, 2919, 1603, 1584, 1414, 1316, 1286, 1126, 1027, 760; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.10 (s, 3H, CH_3), 2.19 (s, 3H, CH_3), 4.79 (s, 1H, CH), 7.10-7.30 (m, 11H, ArH), 7.59-7.61 (d, J = 8 Hz, 4H, ArH), 13.34 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 11.9, 34.0, 121.2, 126.1, 126.4, 127.2, 128.3, 128.9, 137.1, 140.4, 146.7; MS (ESI): 437 $[M]^+$.

4,4′-[(4-Methylphenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7b)



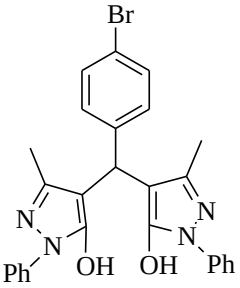
White solid; m.p./lit. m.p. 199-201/201-202 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3455, 3137, 3084, 2975, 1602, 1582, 1486, 1455, 1369, 1282, 1184, 1052, 758, 691; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.17 (s, 3H, CH_3), 2.19 (s, 3H, CH_3), 2.30 (s, 3H, CH_3), 4.82 (s, 1H, CH), 7.03-7.17 (m, 6H, ArH), 7.31-7.35 (t, J = 8 Hz, 4H, ArH), 7.65-7.67 (d, J = 8 Hz, 4H, ArH), 12.92 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 11.9, 20.9, 30.2, 121.1, 122.8, 126.0, 127.0, 128.9, 129.1, 135.9, 137.5, 142.0, 146.7; MS (ESI): 451 $[M]^+$.

4,4′-[(4-Methoxyphenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7c)



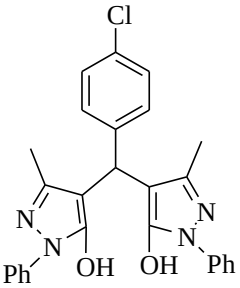
Pale yellow solid; m.p./lit. m.p. 143-145/145-147 °C[11]; FTIR (KBr, ν_{max} in cm^{-1}): 3439, 3065, 2955, 2925, 1599, 1503, 1415, 1222, 1157, 805, 753, 691; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.02 (s, 3H, CH_3), 2.06 (s, 3H, CH_3), 3.75 (s, 3H, OCH_3), 4.72 (s, 1H, CH), 6.78-6.80 (d, J = 8 Hz, 2H, ArH), 7.10-7.15 (m, 4H, ArH), 7.26-7.30 (m, 4H, ArH), 7.58-7.60 (d, J = 8 Hz, 4H, ArH), 13.22 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 11.9, 32.9, 55.0, 106.5, 114.3, 119.3, 121.3, 126.2, 128.2, 128.9, 132.4, 136.7, 137.0, 146.5, 158.0; MS (ESI): 471 $[M]^+$.

4,4′-[(4-Bromophenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7d)



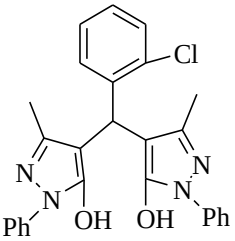
White solid; m.p./lit. m.p. 180−182/181−183 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3440, 3062, 2921, 1597, 1501, 1485, 1404, 1260, 1185, 834, 750, 687; ^{1}H NMR (400 MHz, $CDCl_3$+DMSO-d_6): δ 2.29 (s, 3H, CH_3), 2.50 (s, 3H, CH_3), 4.79 (s, 1H, CH), 7.11-7.15 (t, J = 8 Hz, 4H, ArH), 7.28-7.33 (m, 6H, ArH), 7.65-7.67 (d, J = 8 Hz, 4H, ArH), 13.48 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$+DMSO-d_6): δ 13.0, 33.5, 121.1, 126.3, 126.8, 127.4, 128.8, 129.3, 131.1, 140.9, 146.1; MS (ESI): 515 $[M]^+$, 517 $[M+2]^+$.

4,4′-[(4-Chlorophenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7e)



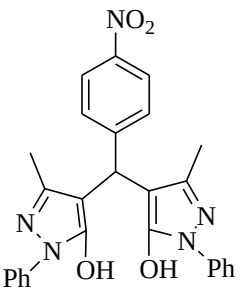
White solid, m.p./lit. m.p. 207-208/207-209 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3443, 3066, 2977, 1600, 1580, 1487, 1373, 1296, 1198, 1087, 810, 690; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.19 (s, 3H, CH_3), 2.22 (s, 3H, CH_3), 4.83 (s, 1H, CH), 7.17-7.25 (m, 6H, ArH), 7.34-7.38 (t, J = 8 Hz, 4H, ArH), 7.66-7.68 (d, J = 8 Hz, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.1, 29.5, 121.4, 126.2, 127.2, 128.5, 128.7, 129.0, 133.9, 137.0, 141.9, 146.8; MS (ESI): 471 $[M]^+$, 473 $[M+2]^+$.

4,4′-[(2-Chlorophenyl]methylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7f)



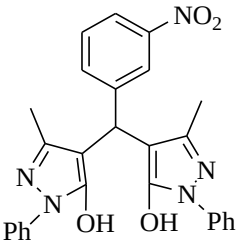
White solid; m.p./lit. m.p. 237-239/236-237 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3456, 3072, 2967, 1605, 1587, 1482, 1376, 1276, 1193, 1084, 822, 696; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.26 (s, 3H, CH_3), 2.30 (s, 3H, CH_3), 5.08 (s, 1H, CH), 7.27−7.29 (m, 2H), 7.41−7.43 (m, 8H), 7.75−7.77 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 47.9, 121.5, 126.2, 126.8, 127.0, 129.0, 129.6, 133.5, 143.8, 155.6. MS (ESI): 471 [M], 473 $[M+2]^+$.

4,4′-[(4-Nitrophenyl]methylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7g)



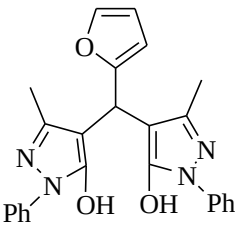
Pale yellow solid; m.p./lit. m.p. 228-230/230-232 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3435, 3067, 2922, 1601, 1582, 1479, 1346, 1296, 1106, 1070, 983, 822, 690; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.35 (s, 6H, 2×CH_3), 5.16 (s, 1H, CH), 7.24−7.28 (t, J = 8 Hz, 2H, ArH), 7.43−7.47 (t, J = 8 Hz, 4H, ArH), 7.51−7.53 (d, J = 8 Hz, 2H, ArH), 7.69−7.71 (d, J = 8 Hz, 4H, ArH), 8.17−8.19 (d, J = 8 Hz, 2H, ArH), 13.89 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, DMSO-d_6): δ 12.3, 31.5, 121.0, 123.9, 124.3, 126.7, 129.2, 129.8, 146.4, 146.8, 150.8; MS (ESI): 482 $[M]^+$.

4,4′-[(3-Nitrophenyl]methylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7h)



Pale yellow solid; m.p./lit. m.p. 230-232/232-234 °C[12]; FTIR (KBr, ν_{max} in cm^{-1}): 3427, 3150, 3106, 2921, 1599, 1408, 1346, 1276, 1026, 902, 734; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.21 (s, 3H, CH_3), 2.33 (s, 3H, CH_3), 4.89 (1H, s, CH), 7.16-7.20 (t, J = 8 Hz, 2H, ArH), 7.33-7.37 (t, J = 8 Hz, 4H, ArH), 7.44-7.47 (d, J = 8 Hz, 1H, ArH), 7.63-7.65 (d, J = 8 Hz, 5H, ArH), 8.07-8.09 (d, J = 8 Hz, 2H), 12.84 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.0, 29.8, 121.5, 121.7, 122.4, 126.5, 129.0, 129.3, 133.7, 134.1, 136.9, 140.1, 142.9, 146.5, 148.4; MS (ESI): 482 $[M]^+$.

4,4′-(Furan-2-ylmethylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7i)



White solid, m.p./lit. m.p. 190-191/189-191 °C[11]; FTIR (KBr, ν_{max} in cm^{-1}): 3442, 3064, 2943, 2921, 1589, 1513, 1413, 1225, 1156, 803, 757, 692; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.30 (s, 3H, CH_3), 2.51 (s, 3H, CH_3), 4.98 (s, 1H, CH), 6.11-6.12 (d, J = 4 Hz, 1H, ArH), 6.34-6.37 (t, J = 4 Hz, 1H, ArH), 7.24-7.28 (t, J = 8 Hz, 2H, ArH), 7.43-7.51 (m, 5H, ArH), 7.69-7.71 (d, J = 8 Hz, 4H, ArH), 13.85 (bs, OH, exchangeable with D_2O); ^{13}C NMR (100

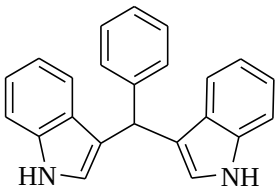
MHz, DMSO-d_6): δ 11.9, 28.8, 106.6, 110.9, 120.9, 126.0, 129.6, 142.2, 146.5, 154.4; MS (ESI): 427 $[M]^+$.

4,4′-(Thiophen-2-ylmethylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (7j)

White solid; m.p./lit. m.p. 191-194/190-192 °C[13]; FTIR (KBr, ν_{max} in cm^{-1}): 3456, 3072, 2967, 1604, 1586, 1488, 1371, 1270, 1195, 1083, 822, 694; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.32 (s, 3H, CH_3), 2.51 (s, 3H, CH_3), 5.13 (s, 1H, CH), 6.75-6.76 (d, J = 4 Hz, 1H, CH), 6.90-6.92 (m, 1H, ArH), 7.24-7.30 (m, 3H, ArH), 7.43-7.47 (t, J = 8 Hz, 4H, ArH), 7.70-7.72 (d, J = 8 Hz, 4H, ArH), 14.02 (bs, OH, exchangeable with D_2O); ^{13}C NMR (400 MHz, DMSO-d_6): δ 12.0, 30.2, 121.0, 121.2, 124.5, 124.6, 126.2, 127.2, 129.4, 146.3, 147.9; MS (ESI): 443 $[M]^+$.

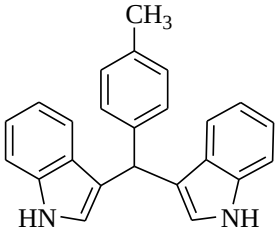
Spectral data of products 8a-8j

Bis(3-indolyl)phenylmethane (8a)



Pink solid, m.p./lit. m.p. 140-142/142-144 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3410, 3054, 2956, 2854, 1454, 743; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.86 (s, 1H, CH), 6.66 (s, 2H, ArH), 6.94 (t, J = 7.6 Hz, 2H, ArH), 7.32-7.07 (m, 9H, ArH), 7.93 (bs, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 31.6, 110.9, 111.9, 118.4, 119.5, 121.2, 124.0, 126.3, 127.1, 128.5, 128.6, 137.0, 145.2; MS (ESI): 323 $[M]^+$.

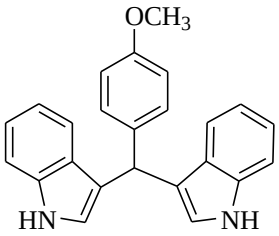
Bis(3-indolyl)-4-methylphenylmethane (8b)



Pale white solid m.p./lit. m.p. 96-98/98-100 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3409, 3052, 2950, 2920, 2856, 1455, 1416, 1337, 1092, 771, 742; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.35 (s, 3H, CH_3), 5.88 (s, 1H, CH), 6.69 (s, 2H, ArH), 7.01-7.05 (t, J = 8 Hz, 2H, ArH), 7.10-7.12 (d, J =

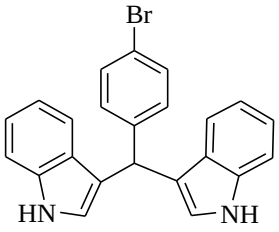
8 Hz, 2H, ArH), 7.17-7.21 (t, *J* = 8 Hz, 2H, ArH), 7.25-7.27 (d, *J* = 8 Hz, 2H, ArH), 7.37-7.43 (m, 4H, ArH), 7.93 (bs, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.3, 39.8, 110.9, 119.2, 119.9, 121.9, 123.5, 127.1, 128.6, 128.9, 135.5, 136.7, 140.9; MS (ESI): 337 $[M]^+$.

Bis(3-indolyl)-4-methoxyphenylmethane (8c)



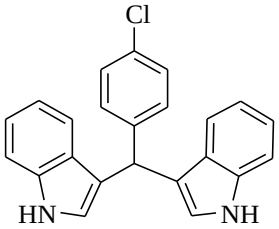
Pink solid; m.p./lit. m.p. 184-186/ 185-187 °C[14]; FTIR (KBr, ν_{max} in cm^{-1}): 3412, 3055, 2957, 1509, 1456, 1271, 1093, 745; ^1H NMR (400 MHz, $CDCl_3$): δ 3.74 (s, 3H, OCH_3), 5.87 (s, 1H, CH), 6.68 (s, 2H, ArH), 6.91-6.93 (d, *J* = 8 Hz, 2H, ArH), 7.02-7.06 (t, *J* = 8 Hz, 2H, ArH), 7.16-7.7.20 (t, *J* = 8 Hz, 2H, ArH), 7.21-7.42 (m, 6H, ArH), 7.97 (bs, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 37.6, 57.4, 112.4, 113.7, 119.4, 122.6, 123.4, 126.8, 132.5, 137.9, 159.7; MS (ESI): 353 $[M]^+$.

Bis(3-indolyl)-4-bromophenylmethane (8d)



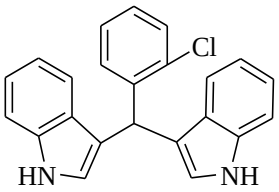
Pink solid; m.p./lit. m.p. 79-81/80-82 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3412, 3050, 2950, 2914, 2849, 1456, 1418, 1383, 1337, 1095, 741; ^1H NMR (400 MHz, $CDCl_3$): δ 5.92 (s, 1H, CH), 6.69 (s, 2H, ArH), 7.03-7.07 (t, *J* = 8 Hz, 2H, ArH), 7.19-7.43 (m, 10H, ArH), 7.96 (bs, 2H, NH, exchangeable with D_2O);.^{13}C NMR (100 MHz, $CDCl_3$): δ 30.2, 111.0, 112.1, 119.2, 119.9, 121.9, 122.1, 123.6, 126.1, 128.2, 128.7, 131.3, 136.7; MS (ESI): 401 $[M]^+$, 403 $[M+2]^+$.

Bis(3-indolyl)-4-chlorophenylmethane (8e)



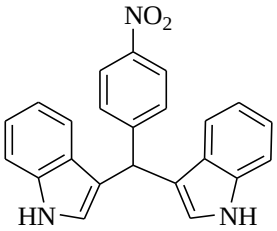
Pink solid; m.p./lit. m.p. 78-79/80-81 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3411, 3055, 2957, 2921, 1456, 1417, 1337, 742; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.89 (s, 1H, CH), 6.68 (s, 2H, ArH), 7.02-7.06 (t, *J* = 8 Hz, 2H, ArH), 7.21-7.23 (t, *J* = 8 Hz, 2H, ArH), 7.25-7.40 (m, 8H, ArH), 7.99 (bs, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 39.7, 111.1, 111.3, 119.2, 119.3, 119.8, 122.0, 123.6, 126.8, 128.3, 130.0, 131.8, 136.7; MS (ESI): 357 $[M]^+$, 359 $[M+2]^+$.

Bis(3-indolyl)-2-chlorophenylmethane (8f)



White solid; m.p./lit. m.p. 71-73/72-74 ^oC[14]; FTIR (KBr, ν_{max} in cm^{-1}): 3409, 3053, 2957, 2927, 2869, 1455, 1477, 1337, 1093, 741; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.37 (s, 1H, CH), 6.66 (s, 2H, ArH), 7.02-7.06 (t, *J* = 8 Hz, 2H, ArH), 7.11-7.46 (m, 10H, ArH), 7.98 (bs, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 36.7, 111.1, 118.3, 119.2, 119.8, 122.0, 123.8, 126.6, 127.0, 127.5, 129.4, 130.3, 133.9, 136.7, 141.4; MS (ESI): 357 $[M]^+$, 359 $[M+2]^+$.

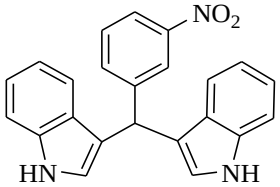
Bis(3-indolyl)-4-nitrophenylmethane (8g)



Yellow solid; m.p./lit. m.p. 217-219/ 218-220 °C[6]; FTIR (KBr, ν_{max} in cm^{-1}): 3456, 3423, 3386, 3052, 1507, 1456, 1339, 746; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.73 (s, 1H, CH), 6.48 (s, 2H, ArH), 6.68-6.71 (t, *J* = 8 Hz, 2H, ArH), 6.85-6.89 (t, *J* = 8 Hz, 2H, ArH), 7.04-7.06

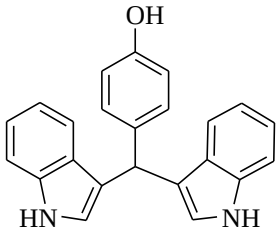
(d, *J* = 8 Hz, 2H, ArH), 7.14-7.16 (d, *J* = 8 Hz, 2H, ArH), 7.27-7.29 (d, *J* = 8 Hz, 2H, ArH), 7.85-7.87 (d, *J* = 8 Hz, 2H, ArH), 9.83 (bs, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 39.9, 111.4, 116.9, 118.6, 119.0, 121.3, 123.2, 123.9, 126.4, 129.3, 136.8, 146.0, 152.6; MS (ESI): 368 $[M]^+$.

Bis(3-indolyl)-3-nitrophenylmethane (8h)



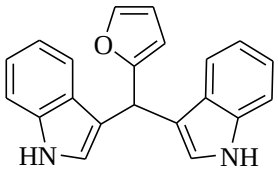
Yellow solid, m.p./lit. m.p. 260-262/262-266 ^oC[15]; FTIR (KBr, ν_{max} in cm^{-1}): 3410, 3055, 1525, 1456, 1348, 743; ^1H NMR (400 MHz, $CDCl_3$): δ 6.02 (s, 1H, CH), 6.70 (s, 2H, ArH), 7.03-7.07 (t, *J* = 8 Hz, 2H, ArH), 7.20-7.24 (t, *J* = 8 Hz, 2H, ArH), 7.37-7.47 (m, 5H, ArH), 7.71-7.73 (d, *J* = 8 Hz, 1H, ArH), 8.08-8.12 (t, *J* = 8 Hz, 2H, ArH), 8.24 (bs, 2H, NH, exchangeable with D_2O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 40.1, 111.2, 111.3, 119.5, 121.5, 122.3, 123.6, 123.7, 126.6, 129.1, 134.9, 136.9, 146.4, 148.5; MS (ESI): 368 $[M]^+$.

Bis(3-indolyl)-4-hydroxyphenylmethane (8i)



Pink solid, m.p./lit. m.p. 120-122/ 122-124 °C[15]; FTIR (KBr, ν_{max} in cm^{-1}): 3450, 3409, 3054, 2955, 1617, 1455, 1093, 747; ^1H NMR (400 MHz, $CDCl_3$): δ 5.78 (s, 1H, CH), 6.89 (s, 2H, ArH), 7.03-7.07 (t, *J* = 8 Hz, 2H, ArH), 7.21-7.23 (d, *J* = 8 Hz, 2H, ArH), 7.27-7.34 (m, 8H, ArH), 7.87 (bs, 2H, NH, exchangeable with D_2O), 8.57 (bs, OH, exchangeable with D_2O), ^{13}C NMR (100 MHz, $CDCl_3$): δ 37.6, 110.9, 114.2, 119.7, 121.2, 123.7, 124.9, 126.8, 129.4, 138.7, 146.3, 148.5.; MS (ESI): 339 $[M]^+$.

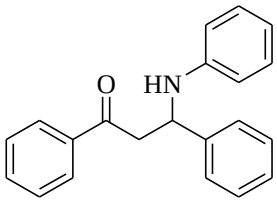
Bis(3-indolyl)-2-furylmethane (8j)



Black solid; m.p./lit. m.p. 316-318/322-325 °C[14]; FTIR (KBr, ν_{max} in cm^{-1}): 3407, 3115, 3052, 1455, 1418, 1337, 1093, 1008, 783, 741; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.99 (s, 1H, CH), 6.93 (s, 2H, ArH), 7.09-7.43 (11H, m, ArH), 8.05 (bs, NH, exchangeable with D_2O), ^{13}C NMR (100 MHz, $CDCl_3$): δ 34.8, 106.5, 110.2, 111.3, 112.2, 118.0, 119.3, 119.7, 121.7, 124.3, 126.3, 135.9, 142.0;. MS (ESI): 313 $[M]^+$.

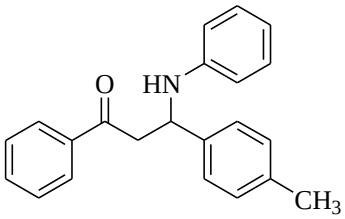
Spectral data of products 12a-o

1,3-Diphenyl-3-(Phenylamino)propan-1-one (12a)



Colorless solid; m.p./lit. m.p. 165-168/169–170 °C[16]; FTIR (KBr, ν_{max} in cm^{-1}): 3384, 3017, 2399, 1671, 1600, 1509, 1449, 1417, 1291, 1214, 1077, 1002, 856, 756, 668; ^{1}H NMR (400 MHz, $CDCl_3$ + DMSO-d_6): δ 3.55-3.61 (m, 2H, CH_2), 4.51 (bs, 1H, NH, exchangeable with D_2O), 4.94-4.97 (t, J = 8 Hz, 1H, CH), 5.75-5.77 (d, J = 8 Hz, 1H, ArH), 6.47-6.49 (d, J = 8 Hz, 1H, ArH), 6.92-6.96 (t, J = 8 Hz, 2H, ArH), 7.11-7.15 (t, J = 8 Hz, 2H, ArH), 7.21-7.25 (t, J = 8 Hz, 2H, ArH), 7.39-7.42 (m, 5H, ArH), 7.51-7.55 (t, J = 8 Hz, 1H, ArH), 7.88-7.89 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$ + DMSO-d_6): δ 46.7, 53.6, 113.3, 116.6, 126.2, 127.0, 128.2, 128.7, 128.9, 133.2, 136.6, 144.0, 147.9, 197.6; MS (ESI): 302 $[M]^+$.

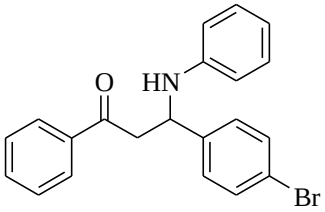
3-(4-Methylphenyl)-1-phenyl-3-(phenylamino)propan-1-one (12b)



White solid; m.p./lit. m.p. 128-129/131-132 °C[17]; FTIR (KBr, cm^{-1}): 3394, 3019, 1681, 1601, 1504, 1448, 1214, 1010, 755, 695, 666 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.34 (s, 3H, CH_3), 3.45-3.52 (m, 2H, CH_2), 4.55 (bs, 1H, NH, exchangeable with D_2O), 4.99-5.02 (t, J = 4 Hz, 1H, CH), 6.58-6.60 (d, J = 8 Hz, 2H, ArH), 6.67-6.70 (t, J = 8 Hz, 1H, ArH), 7.10–7.17 (m, 4H, ArH), 7.35-7.37 (d, J = 8 Hz, 2H, ArH), 7.46-7.50 (t, J = 8Hz, 2H, ArH), 7.57-7.61 (t, J = 8 Hz, 1H, ArH), 7.94-7.96 (d, J = 8Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ

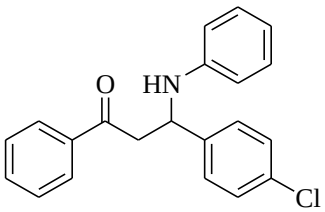
21.2, 46.1, 54.7, 113.7, 117.7, 126.2, 128.2, 128.7, 129.1, 129.5, 133.3, 136.8, 136.9, 139.9, 147.2, 198.4; MS (ESI): 316 $[M]^+$.

3-(4-Bromophenyl)-1-phenyl-3-(phenylamino)propan-1-one (12c)



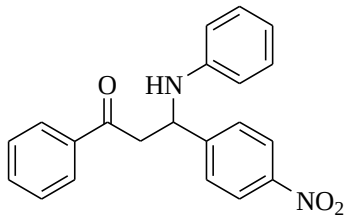
Colorless solid; m.p./lit. m.p. 128-120/130-131 ^oC[18]; FTIR (KBr, ν_{max} in cm^{-1}): 3394, 3019, 1681, 1601, 1504, 1448, 1214, 1010, 755, 695, 666; ^1H NMR (400 MHz, $CDCl_3$): δ 3.45-3.52 (m, 2H, CH_2), 4.60 (bs, 1H, NH, exchangeable with D_2O), 4.97-5.01 (t, J = 8 Hz, 1H, CH), 6.54-6.56 (d, J = 8 Hz, 2H, ArH), 6.69-6.72 (t, J = 8 Hz, 1H, ArH), 7.12-7.16 (t, J = 8 Hz, 2H, ArH), 7.45–7.94 (m, 7H, ArH), 8.03-8.05 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 45.6, 54.28, 114.1, 118.6, 122.3, 124.2, 127.8, 128.8, 129.2, 131.5, 132.0, 132.4, 143.5, 146.2, 190.4; MS (ESI): 380 $[M]^+$, 382 $[M+2]^+$.

3-(4-Chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one (12d)



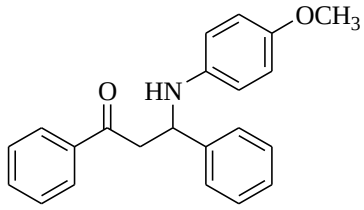
Colorless solid; m.p./lit. m.p. 113-114/117-118 ^oC[16]; FTIR (KBr, ν_{max} in cm^{-1}): 3399, 3019, 2399, 1682, 1601, 1504, 1448, 1407, 1215, 1013, 825, 756, 690; ^1H NMR (400 MHz, $CDCl_3$): δ 3.45-3.49 (m, 2H, CH_2), 4. 59 (bs, 1H, NH, exchangeable with D_2O), 4.98-5.02 (t, J = 8 Hz, 1H, CH), 7.41-7.43 (d, J = 8 Hz, 2H, ArH), 7.52-7.56 (t, J = 8 Hz, 1H, ArH), 7.60-7.63 (t, J = 4 Hz, 2H, ArH), 7.77–7.81 (m, 7H, ArH), 8.03–8.05 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.7, 55.8, 113.8, 122.4, 124.2, 128.1, 128.5, 128.7, 129.2, 129.6.0, 132.9, 133.3, 136.4, 138.0, 190.3; MS (ESI): 336 $[M]^+$, 338 $[M+2]^+$.

3-(4-Nitrophenyl)-1-phenyl-3-(phenylamino)propan-1-one (12e)



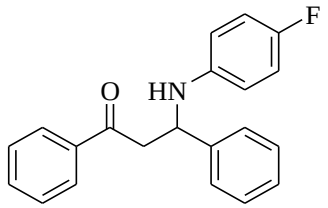
Creamish solid; m.p./lit. m.p. 105-106/108-109 ^{o}C[17]; FTIR (KBr, ν_{max} in cm^{-1}): 3396, 1670, 1578, 1510, 1300, 730, 681; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.44-3.54 (m, 2H, CH_2), 4.72 (bs, 1H, NH, exchangeable with D_2O), 5.11-5.14 (t, J = 8 Hz, 1H, CH), 7.54–7.87 (m, 10H, ArH), 8.05-8.07 (d, J = 8 Hz, 2H, ArH), 8.30-8.32 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 31.3, 54.2, 113.9, 121.1, 124.2, 125.7, 127.4, 128.3, 128.6, 128.8, 128.9, 133.4, 137.5, 141.0, 141.5, 148.6, 189.6, 208.6; MS (ESI): 347 $[M]^{+}$.

3-[(4-Methoxyphenyl)amino]-1,3-diphenylpropan-1-one (12f)



Yellow solid; m.p./lit. m.p. 120-121/124-126 ^{o}C[19]; FTIR (KBr, ν_{max} in cm^{-1}): 3376, 1683, 1576, 1515, 1299, 729, 670; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.44-3.50 (m, 2H, CH_2). 3.71 (s, 3H, OCH_3), 4.45 (bs, 1H, NH, exchangeable with D_2O), 5.03-5.07 (t, J = 8 Hz, 1H, CH), 6.54-6.56 (d, J = 8 Hz, 2H, ArH), 6.70-6.72 (d, J = 8 Hz, 2H, ArH), 7.24–7.61 (m, 8H, ArH), 7.93-7.95 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.6, 55.7, 114.4, 114.7, 115.3, 122.2, 126.4, 127.3, 128.2, 128.7, 128.8, 133.4, 136.6, 141.2, 143.3, 152.4, 158.7, 198.6; MS (ESI): 332 $[M]^{+}$.

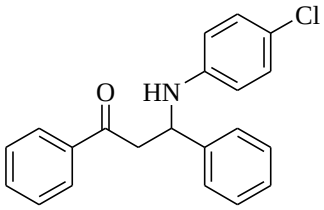
3-[(4-Fluorophenyl)amino]-1,3-diphenylpropan-1-one (12g)



Creamish solid; m.p./lit. m.p. 144-146/149-151 ^{o}C[17]; FTIR (KBr, ν_{max} in cm^{-1}): 3380, 1698, 1590, 1514, 1220, 730, 684; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.44-3.50 (m, 2H, CH_2), 4.50 (s,

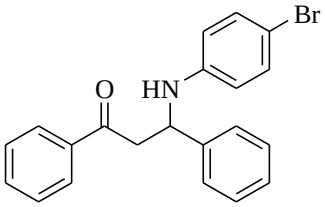
1H, NH, exchangeable with D_2O), 4.93-4.96 (t, *J* = 8 Hz, 1H, CH), 6.49–6.53 (m, 2H, ArH), 6.79-6.83 (t, *J* = 8 Hz, 2H, ArH), 7.25-7.27 (d, *J* = 8 Hz, 1H, ArH), 7.34-7.37 (t, *J* = 8 Hz, 2H, ArH), 7.44-7.49 (m, 4H, ArH), 7.58-7.61 (t, *J* = 8 Hz, 1H, ArH), 7.93-7.95 (d, *J* = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.4, 55.3, 114.8, 114.9, 115.4, 115.6, 126.3, 127.4, 128.9, 128.7, 128.9, 133.6, 136.7, 142.9, 198.4; MS (ESI): 320 $[M]^+$.

3-(4-Chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one (12h)



Colorless solid; m.p./lit. m.p. 168-169/170–171 ^oC[19]; FTIR (KBr, ν_{max} in cm^{-1}): 3399, 3018, 2399, 1682, 1601, 1504, 1448, 1407, 1215, 1013, 825, 756, 690; ^{1}H NMR (400 MHz, $CDCl_3$+ DMSO-d_6): δ 3.54-3.61 (m, 2H, CH_2), 4.49 (bs, 1H, NH, exchangeable with D_2O), 4.91-4.95 (t, *J* = 8 Hz, 1H, CH), 5.97-5.99 (d, *J* = 8 Hz, 1H, ArH), 6.43-6.45 (d, *J* = 8 Hz, 2H, ArH), 6.88-6.90 (d, *J* = 8 Hz, 2H, ArH), 7.12-7.15 (t, *J* = 8 Hz, 1H, ArH), 7.21-7.25 (t, *J* = 8 Hz, 2H, ArH), 7.36–7.44 (m, 3H, ArH), 7.51-7.53 (d, *J* = 8 Hz, 1H, ArH), 7.87–7.89 (d, *J* = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$+DMSO-d_6): δ 46.7, 53.6, 114.4, 120.2, 126.6, 127.1, 128.1, 128.6, 128.7, 128.8, 133.3, 136.9, 143.5, 146.6, 197.2; MS (ESI): 336 $[M]^+$, 338 $[M+2]^+$.

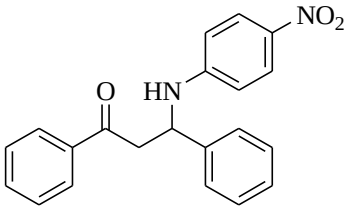
3-(4-Bromophenylamino)-1,3-diphenylpropan-1-one (12i)



Colorless solid; m.p./lit. m.p. 182-183/180-181^oC[16]; FTIR (KBr, ν_{max} in cm^{-1}): 3368, 3019, 1665, 1593, 1576, 1504, 1447, 1407, 1312, 1283, 1070, 916, 848; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.44-3.51 (m, 2H, CH_2), 4.56 (bs, 1H, NH, exchangeable with D_2O), 5.00-5.03 (t, *J* = 8 Hz, 1H, CH), 6.57-6.59 (d, *J* = 8 Hz, 2H, ArH), 6.68-6.71 (t, *J* = 8Hz, 1H, ArH), 7.10-7.14 (t, *J* = 8Hz, 2H, ArH), 7.32–7.87 (m, 7H, ArH), 7.91-7.93 (d, *J* = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.5, 55.1, 113.8, 117.9, 121.6, 126.3, 127.4, 128.5, 128.8,

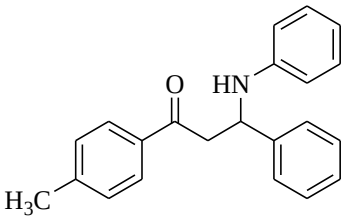
129.0, 129.2, 129.7, 130.5, 130.8, 132.0, 142.7, 145.4, 146.6, 197.3; MS (ESI): 380 $[M]^+$, 382 $[M+2]^+$.

3-(4-Bromophenylamino)-1,3-diphenylpropan-1-one (12j)



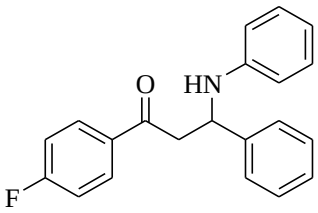
Yellow solid; m.p./lit. m.p. 182–183/184–186 ^oC[19]; FTIR (KBr, ν_{max} in cm^{-1}): 3380, 3012, 1610, 1591, 1571, 1503, 1456, 1401, 1324, 1230, 1056, 976, 832; ^1H NMR (400 MHz, $CDCl_3$): δ 3.54-3.56 (m, 2H, CH_2), 4.64 (bs, 1H, NH, exchangeable with D_2O), 5.01-5.05 (t, J = 8 Hz, 1H, CH), 6.65-6.67 (d, J = 8 Hz, 2H, ArH), 6.80-6.82 (d, J = 8 Hz, 2H, ArH), 7.23-7.27 (t, J = 8 Hz, 2H, ArH), 7.30-7.34 (t, J = 8 Hz, 2H, ArH), 7.45-7.51 (m, 3H, ArH), 7.56-7.80 (t, J = 8 Hz, 1H, ArH), 7.92-7.94 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 47.2, 54.4, 114.8, 115.7, 125.8, 126.8, 127.8, 128.6, 128.9, 129.7, 133.0, 133.5, 136.1, 136.6, 141.8, 144.8, 197.9; MS (ESI): 347.14 $[M]^+$.

3-(Phenylamino)-1-(4-methylphenyl)-3-phenyl-propan-1-one (12k)



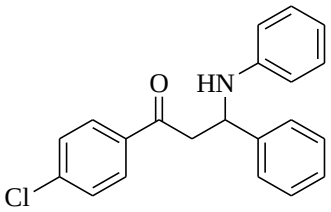
Creamish solid; m.p./lit. m.p. 142-144/139-140 ^oC[17]; FTIR (KBr, ν_{max} in cm^{-1}): 3384, 3028, 2984, 2966, 2846, 1679, 1625, 1520, 1410, 1349, 1267, 1145, 824, 741; ^1H NMR (400 MHz, $CDCl_3$): δ 2.41 (s, 3H, CH_3), 3.43-3.47 (m, 2H, CH_2), 4.43 (bs, 1H, NH, exchangeable with D_2O), 4.92-4.94 (t, J = 8 Hz, 1H, CH), 6.45-6.47 (d, J = 8 Hz, 2H, ArH), 6.76-6.78 (d, J = 8 Hz, 2H, ArH), 7.13-7.18 (m, 8H, ArH), 7.78-7.80 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.5, 55.1, 74.2, 112.6, 119.2, 128.0, 128.1, 129.6, 132.5, 142.1, 143.4, 195.8; MS (ESI): 316 $[M]^+$.

3-(Phenylamino)-1-(4-fluorophenyl)-3-phenyl-propan-1-one (12l)



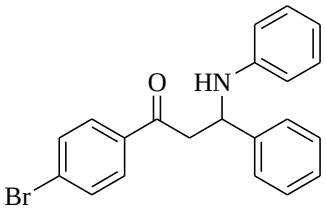
Colorless solid; m.p/lit. m.p. 115-116/119–120 ^{o}C[16]; FTIR (KBr, ν_{max} in cm^{-1}): 3397, 3024, 2987, 2964, 2859, 1681, 1600, 1507, 1406, 1342, 1246, 1157, 835, 759; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.45-3.57 (m, 2H, CH_2), 4.60 (bs, 1H, NH, exchangeable with D_2O), 5.00-5.04 (t, J = 8 Hz, 1H, CH), 6.59–6.72 (m, 3H, ArH), 7.05–7.19 (m, 4H), 7.22-7.32 (m, 3H, ArH), 7.43–7.45 (d, J = 8 Hz, 2H, ArH), 7.86-7.94 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.0, 54.7, 113.9, 115.8, 115.9, 117.9, 126.3, 127.3, 128.7, 129.1, 130.7, 130.9, 133.1, 142.9, 146.6, 163.5, 194.1; MS (ESI): 320 $[M]^{+}$.

3-(Phenylamino)-1-(4-chlorophenyl)-3-phenyl-propan-1-one (12m)



Colorless solid; m.p./lit. m.p. 115-116 ^{o}C/119-120 ^{o}C[16]; FTIR (KBr, ν_{max} in cm^{-1}): 3377, 3019, 2922, 2399, 1918, 1683, 1601, 1589, 1504, 1431, 1358, 1215, 1177, 1094, 832, 755, 699; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.49-3.54 (m, 2H, CH_2), 4.58 (bs, 1H, NH, exchangeable with D_2O), 4.98-4.51 (t, J = 8 Hz, 1H, CH), 6.52-6.71 (m, 3H, ArH), 7.02-7.09 (m, 2H, ArH), 7.19-7.42 (m, 7H, ArH), 7.78-7.80 (d, J = 8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 42.5, 51.0, 113.4, 118.9, 120.4, 121.6, 128.5, 129.7, 129.9, 131.9, 136.6, 139.1, 141.4, 143.5, 189.6; MS (ESI): 336 $[M]^{+}$, 338 $[M+2]^{+}$.

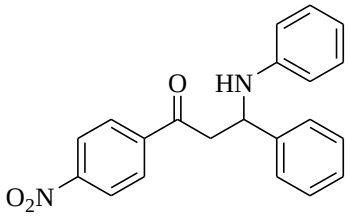
3-(Phenylamino)-1-(4-bromophenyl)-3-phenyl-propan-1-one (12n)



White solid; m.p./lit. m.p. 140-142/143-145 ^{o}C[20]; FTIR (KBr, cm^{-1}): 3376, 1686, 1591, 1504, 1299, 735, 693; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.44-3.51 (m, 2H, CH_2), 4.53 (bs, 1H, NH, exchangeable with D_2O), 5.00-5.03 (t, J = 4 Hz, 1H, CH), 6.57-6.59 (d, J = 8 Hz, 1H, ArH),

6.68-6.71 (t, *J* = 8 Hz, 1H, ArH), 7.10-7.14 (t, *J* = 8 Hz, 1H, ArH), 7.24–7.26 (d, *J* = 8 Hz, 1H, ArH), 7.32-7.36 (t, *J* = 8 Hz, 1H, ArH), 7.43-7.49 (m, 3H, ArH), 7.59-7.61 (d, *J* = 8 Hz, 1H, ArH), 7.67-7.69 (d, *J* = 8 Hz, 2H, ArH), 7.76-7.78 (d, *J* = 8 Hz, 2H, ArH), 7.91-7.93 (d, *J* = 8 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.5, 54.7, 113.6, 117.9, 121.6, 126.3, 127.4, 127.5, 128.5, 128.8, 129.0, 129.2, 129.7, 130.0, 130.8, 131.9, 132.0, 142.6, 145.6, 146.6, 197.6; MS (ESI): 380.2 $[M]^+$, 382.01$[M+2]^+$.

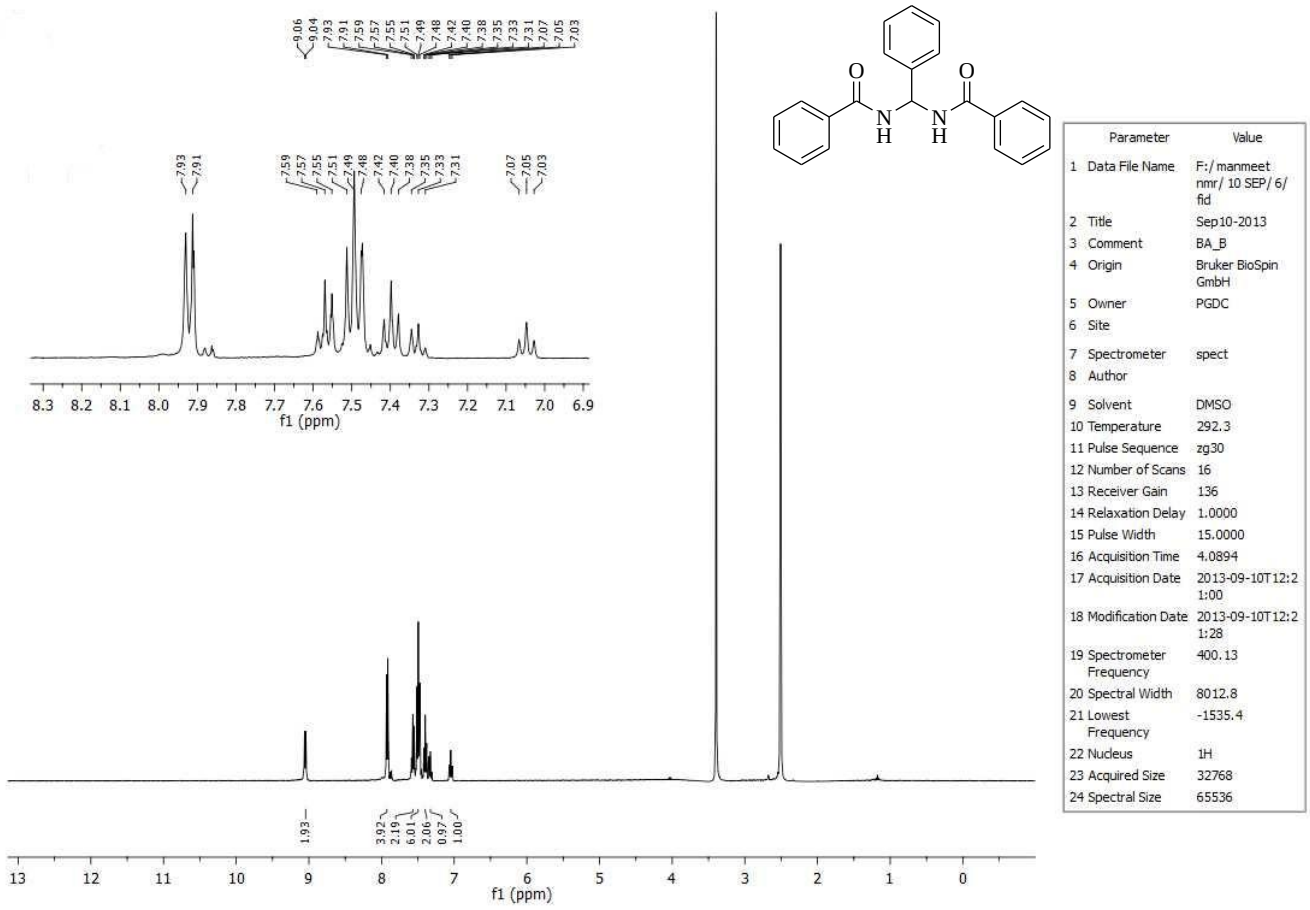
3-(Phenylamino)-1-(4-nitrophenyl)-3-phenyl-propan-1-one (12o)



Yellow solid; m.p./lit. m.p. 149-151/146-148 °C[21]; FTIR (KBr, ν_{max} in cm^{-1}): 3409, 1679, 1610, 1520, 1520, 1432, 1389; ^{1}H NMR (400 MHz; $CDCl_3$): δ 3.51-3.64 (m, 2H, CH_2), 4.64 (bs, 1H, NH, exchangeable with D_2O), 5.05-5.08 (t, *J* = 4 Hz, 1H, CH), 6.59-6.61 (d, *J* = 8 Hz, 1H, ArH), 6.69-6.93 (t, *J* = 8 Hz, 1H, ArH), 7.11-7.15 (t, *J* = 8 Hz, 1H, ArH), 7.24-7.26 (d, *J* = 8 Hz, IH, ArH), 7.32-7.36 (t, *J* = 8 Hz, 1H, ArH), 7.47-7.52 (m, 4H, ArH), 7.68-7.72 (t, *J* = 8 Hz, 1H, ArH), 8.16-8.18 (d, *J* = 8 Hz, 2H, ArH), 8.37-8.39 (d, *J* = 8Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.6, 50.9, 113.8, 118.1, 121.3, 126.3, 127.6, 128.7, 128.9, 129.1, 129.2, 129.4, 131.3, 134.3, 141.1, 142.3, 143.1, 146.9, 150.1, 150.4, 196.9; MS (ESI): 347.14 $[M]^+$.

Spectral data of some selected compounds

^{1}H spectra of 3a



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8	Author	
9	Solvent	DMSO
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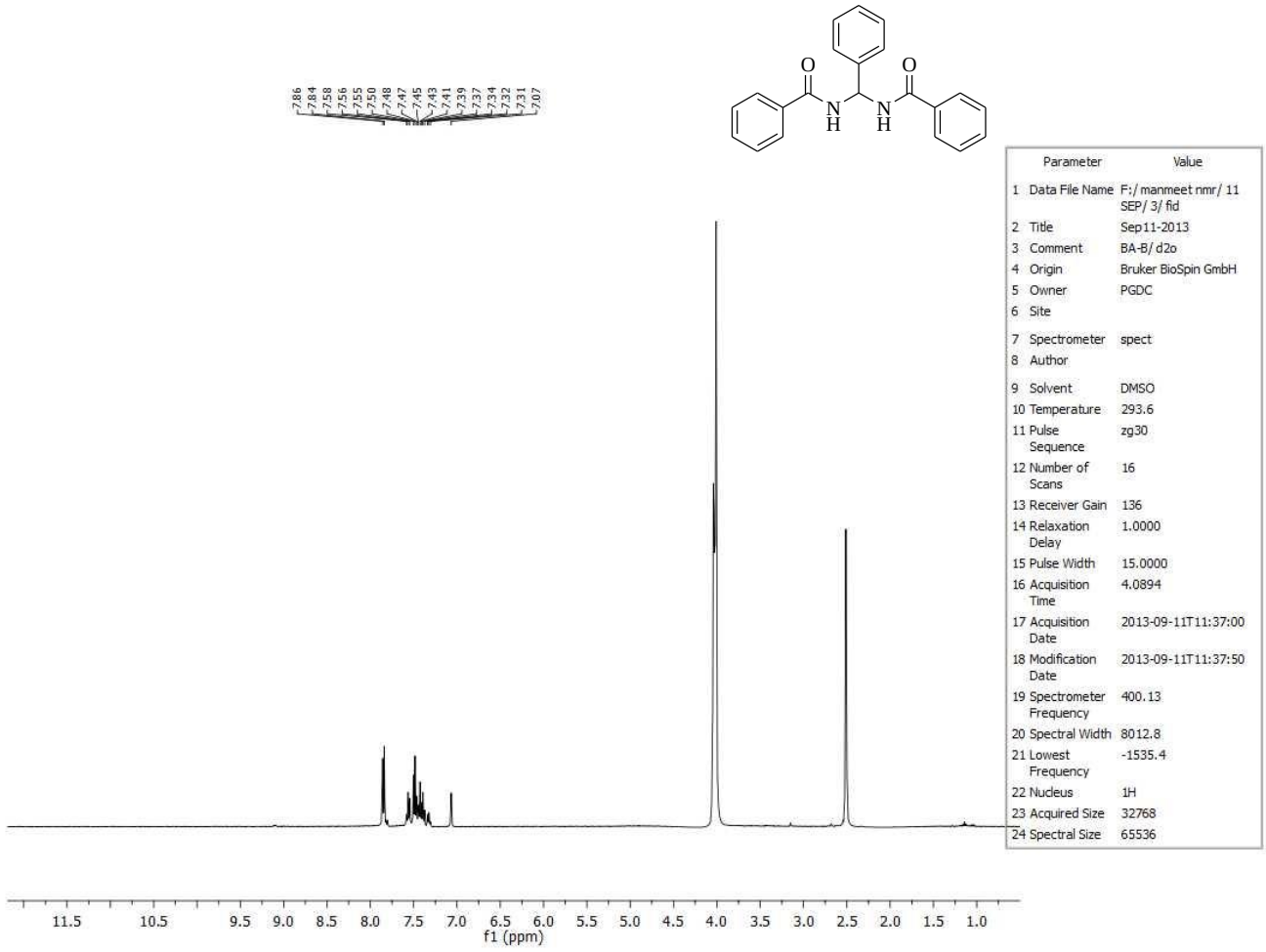
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	136
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2013-09-10T12:21:00
18	Modification Date	2013-09-10T12:21:28
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 3a



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7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	293.6
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	136
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
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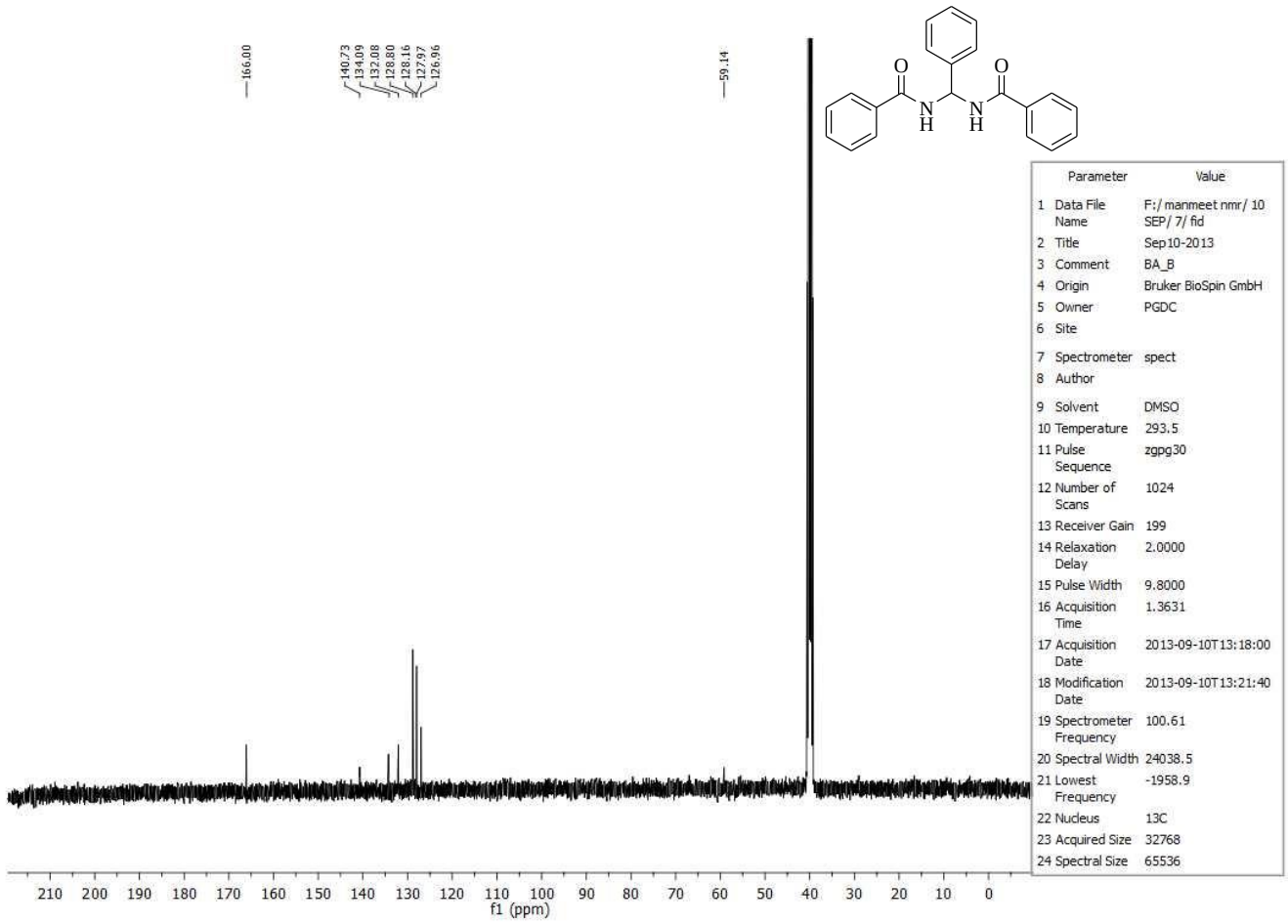
18	Modification Date	2013-09-11T11:37:50
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 3a



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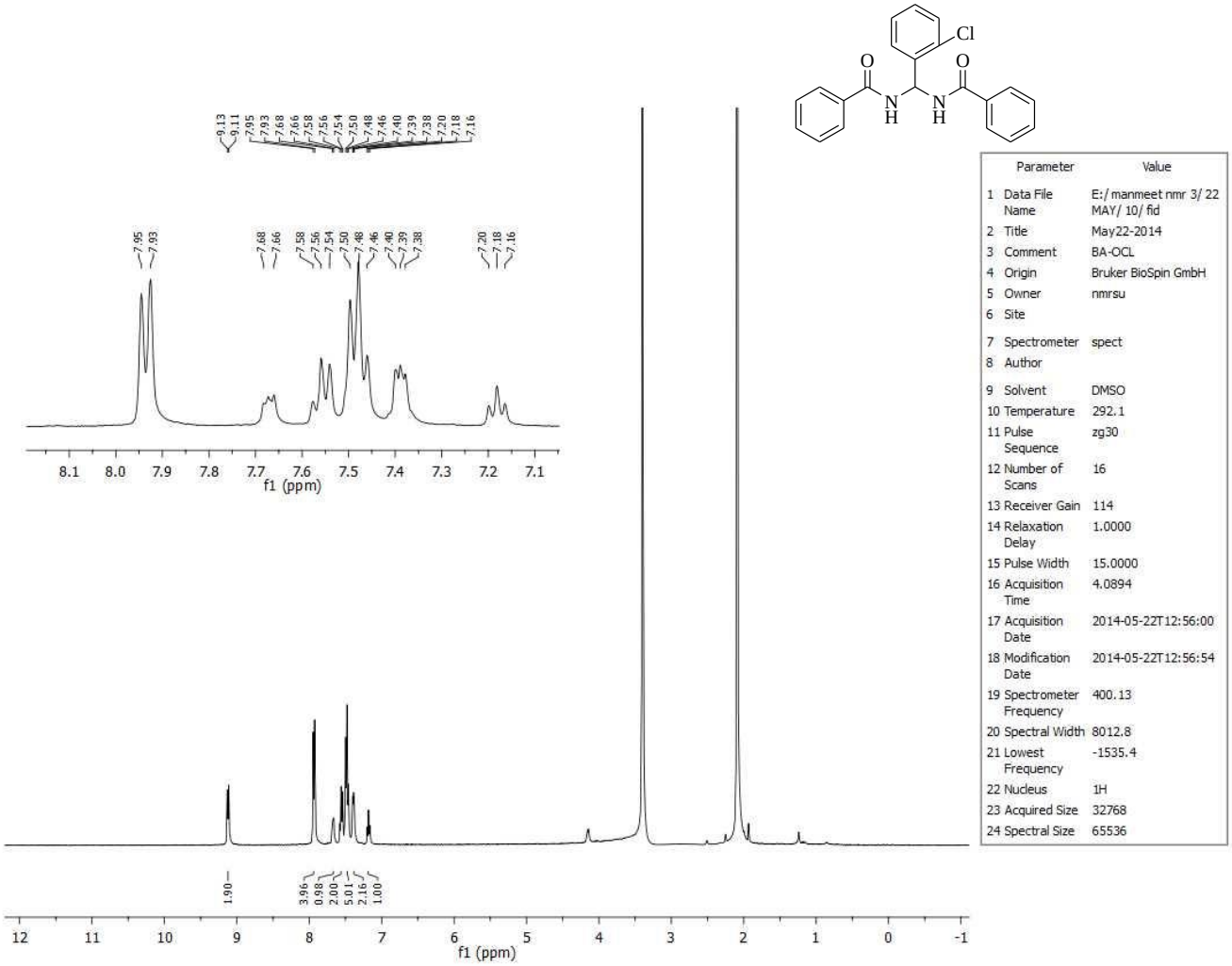
5	Owner	PGDC
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	293.5
11	Pulse Sequence	zgpg30
12	Number of Scans	1024
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2013-09-10T13:18:00
18	Modification Date	2013-09-10T13:21:40
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 3f



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5	Owner	nmrsu
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8	Author	
9	Solvent	DMSO
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13	Receiver Gain	114
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
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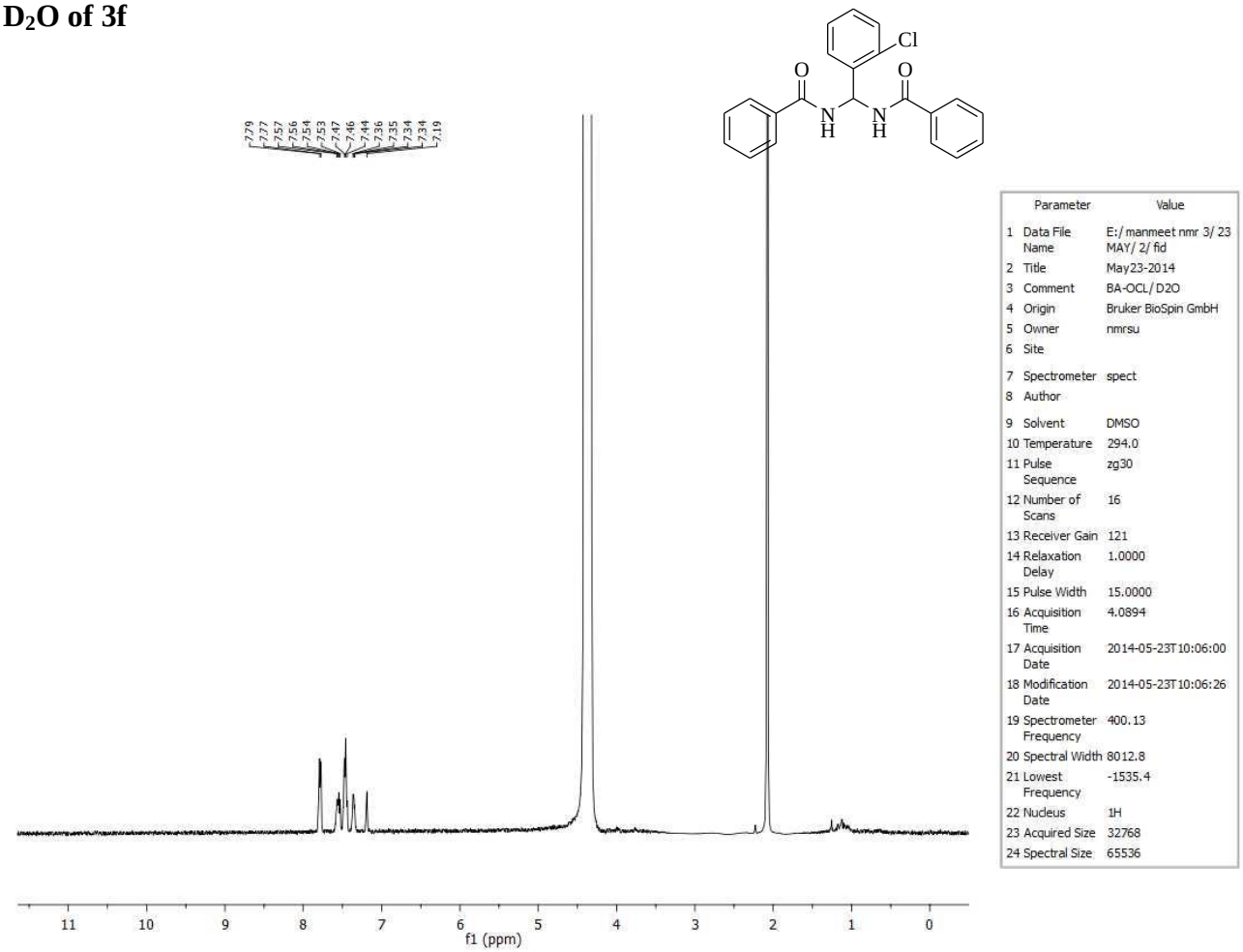
18	Modification Date	2014-05-22T12:56:54
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 3f



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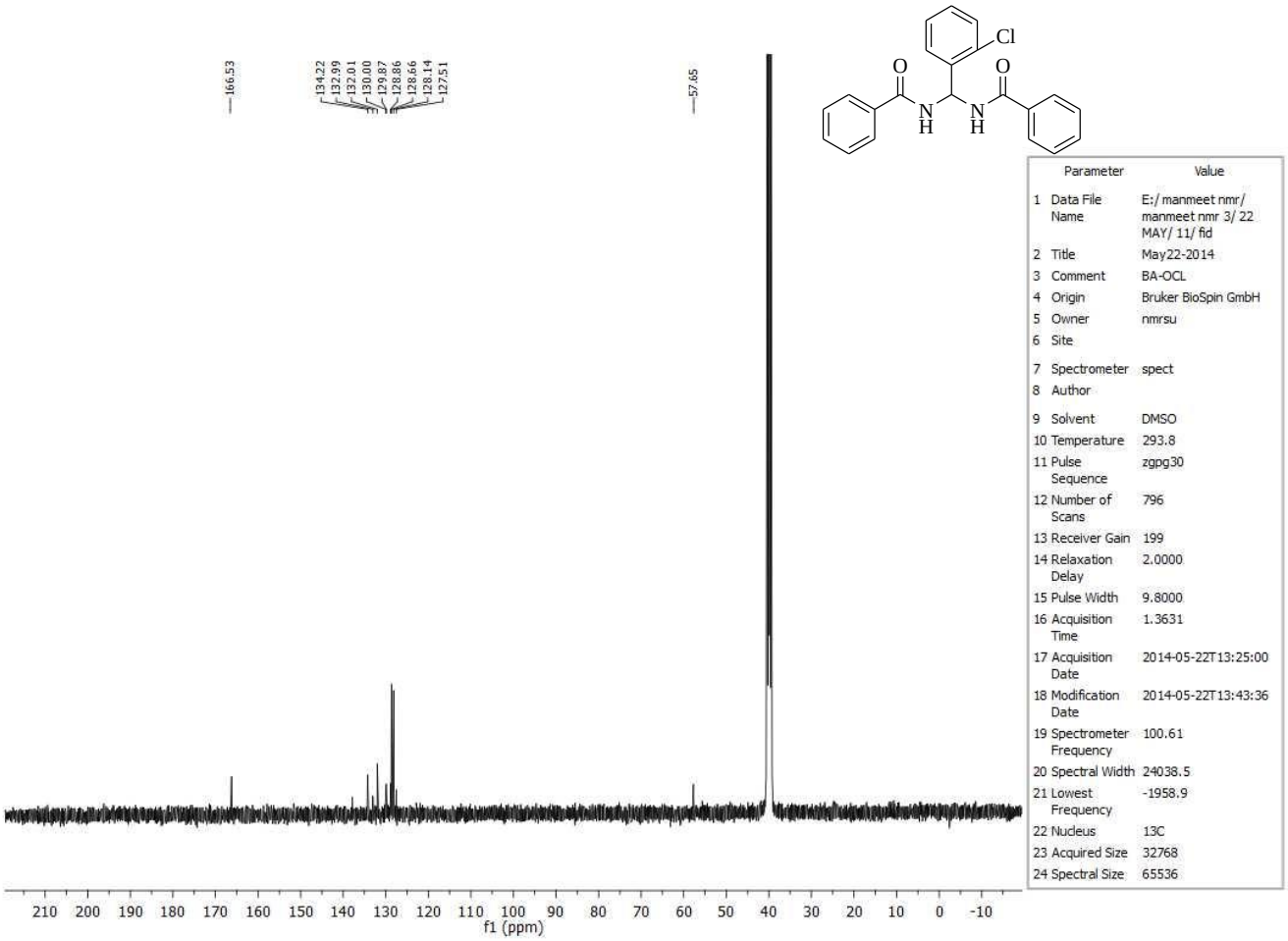
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	294.0
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	121
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-23T10:06:00
18	Modification Date	2014-05-23T10:06:26
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 3f



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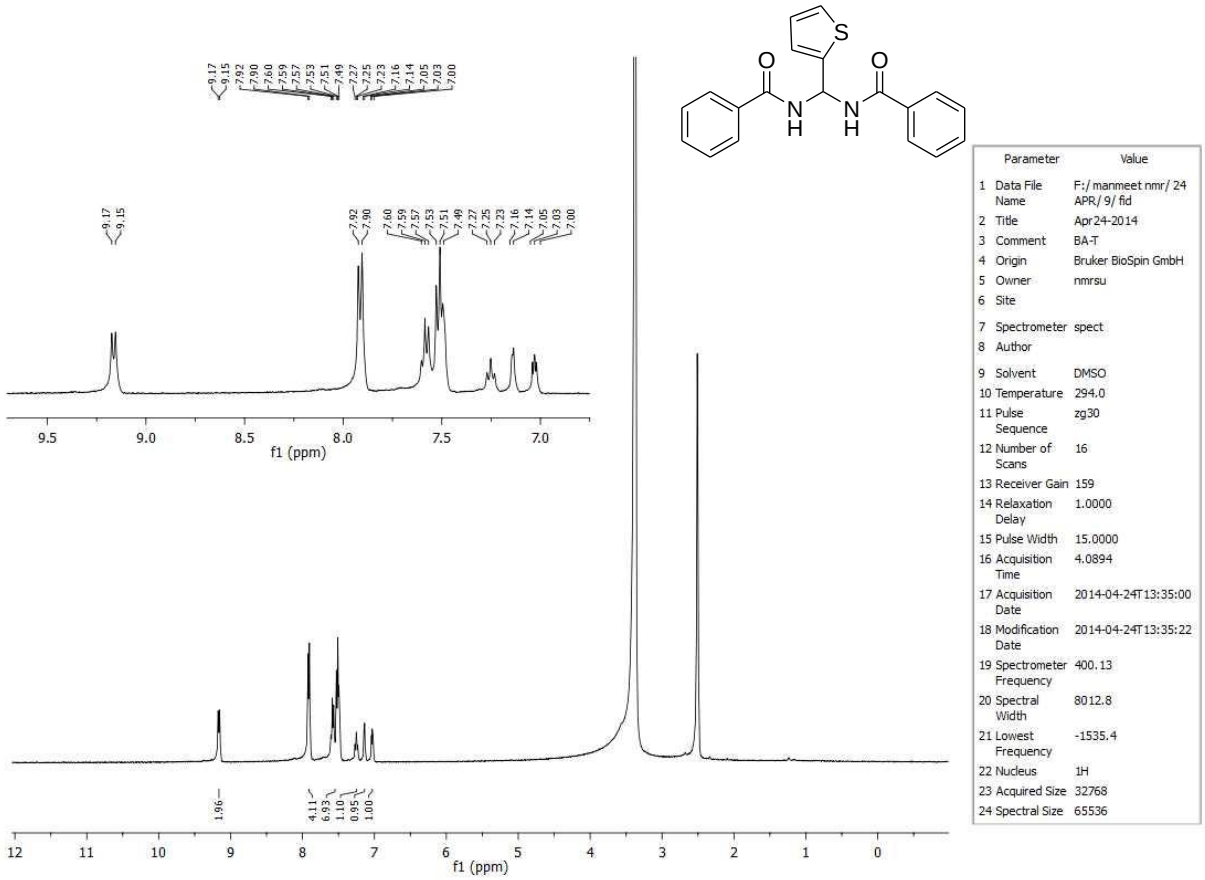
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	293.8
11	Pulse Sequence	zgpg30
12	Number of Scans	796
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-05-22T13:25:00
18	Modification Date	2014-05-22T13:43:36
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 3k



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8	Author	
9	Solvent	DMSO
10	Temperature	294.0
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12	Number of Scans	16
13	Receiver Gain	159
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
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20	Spectral Width	8012.8
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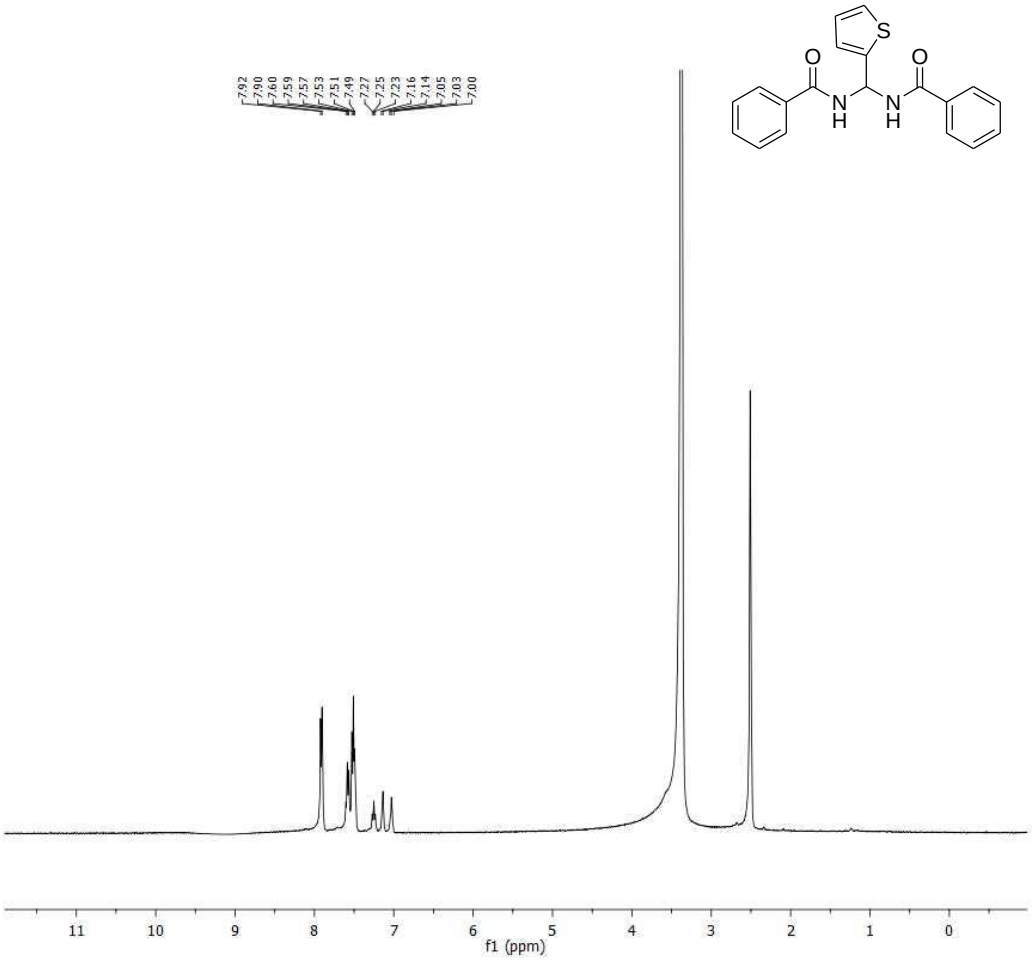
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 3k



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8	Author	
9	Solvent	DMSO
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16	Acquisition Time	4.0894
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19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
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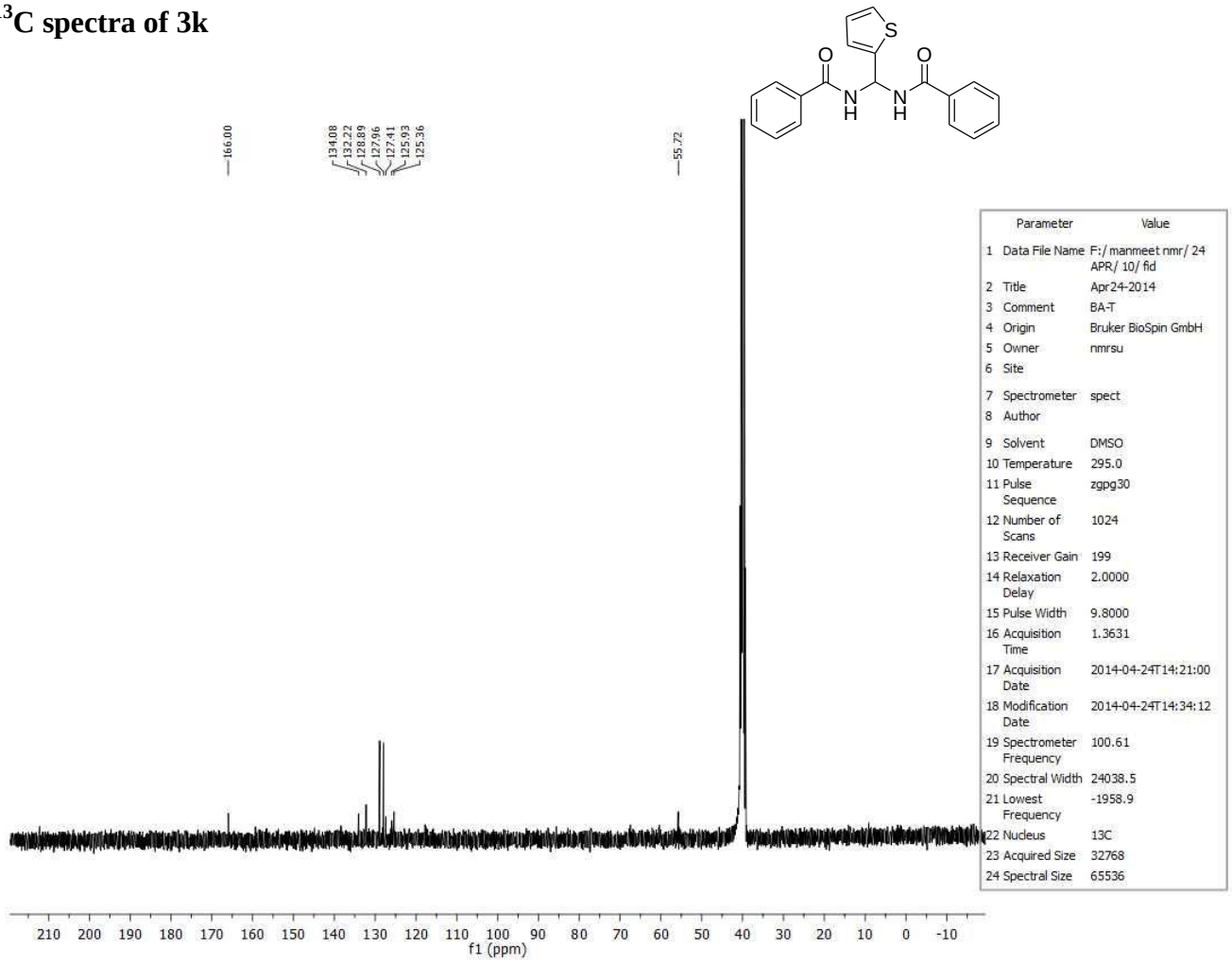
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 3k



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8	Author	
9	Solvent	DMSO
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11	Pulse Sequence	zgpg30
12	Number of Scans	1024
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
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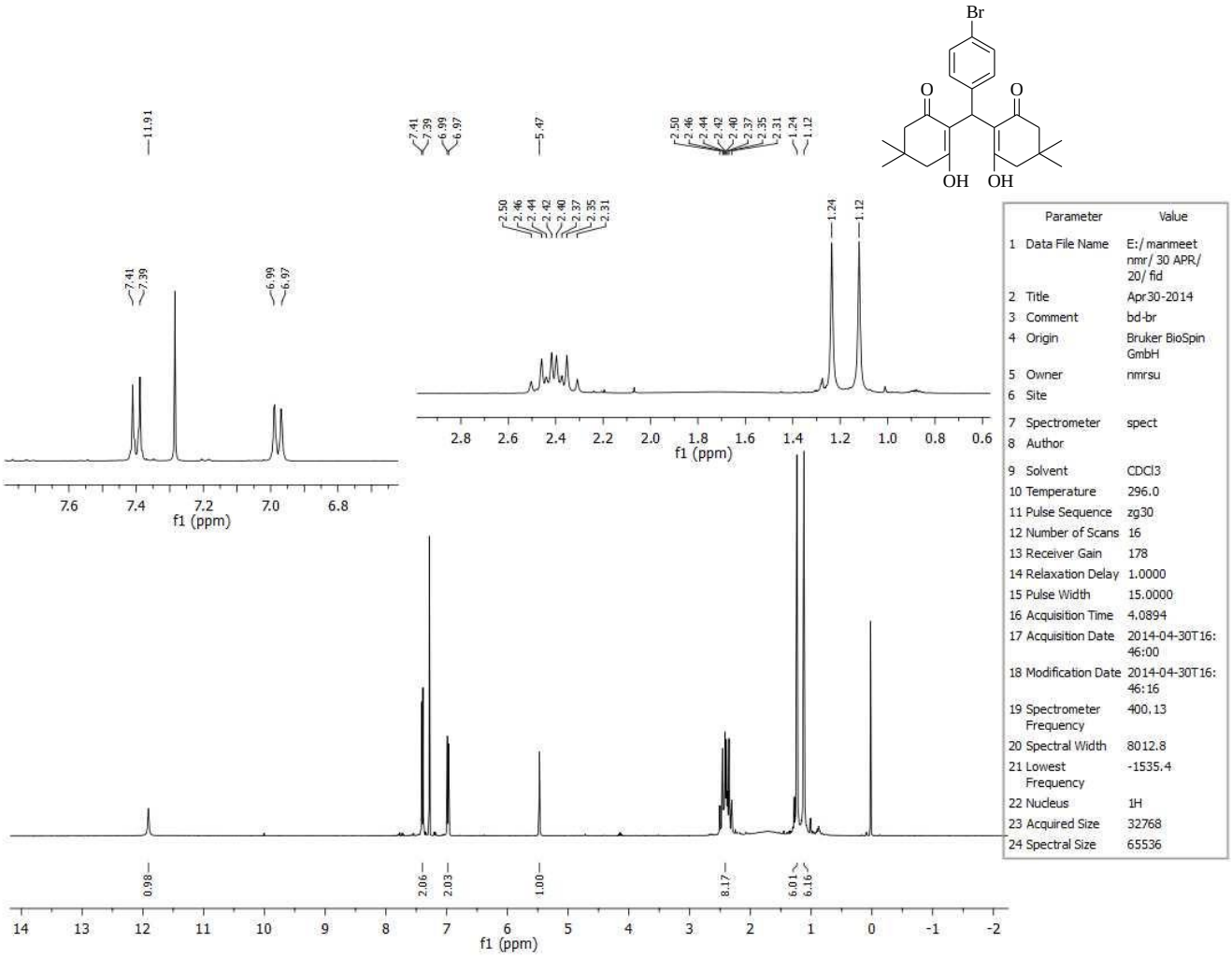
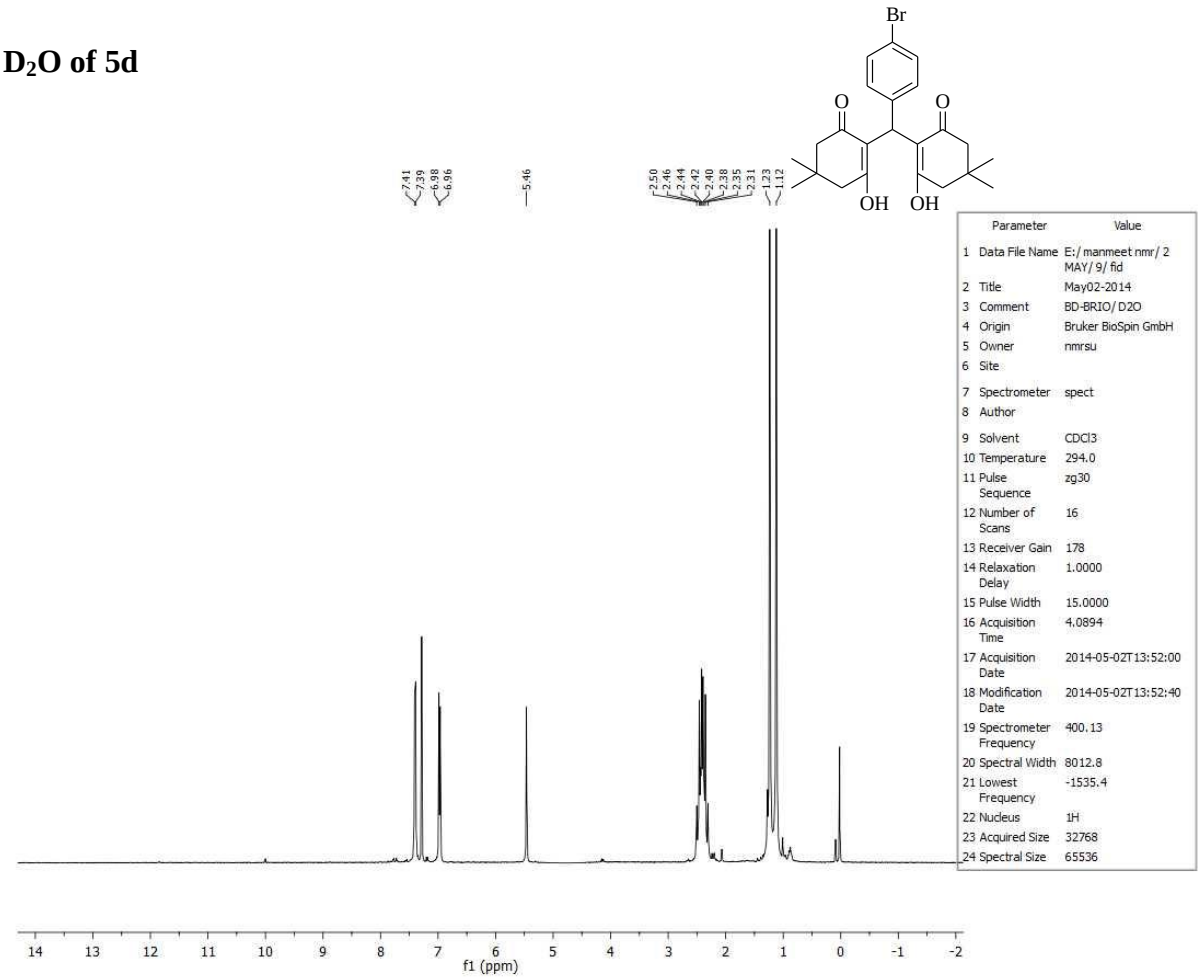
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 5d



D_2O of 5d



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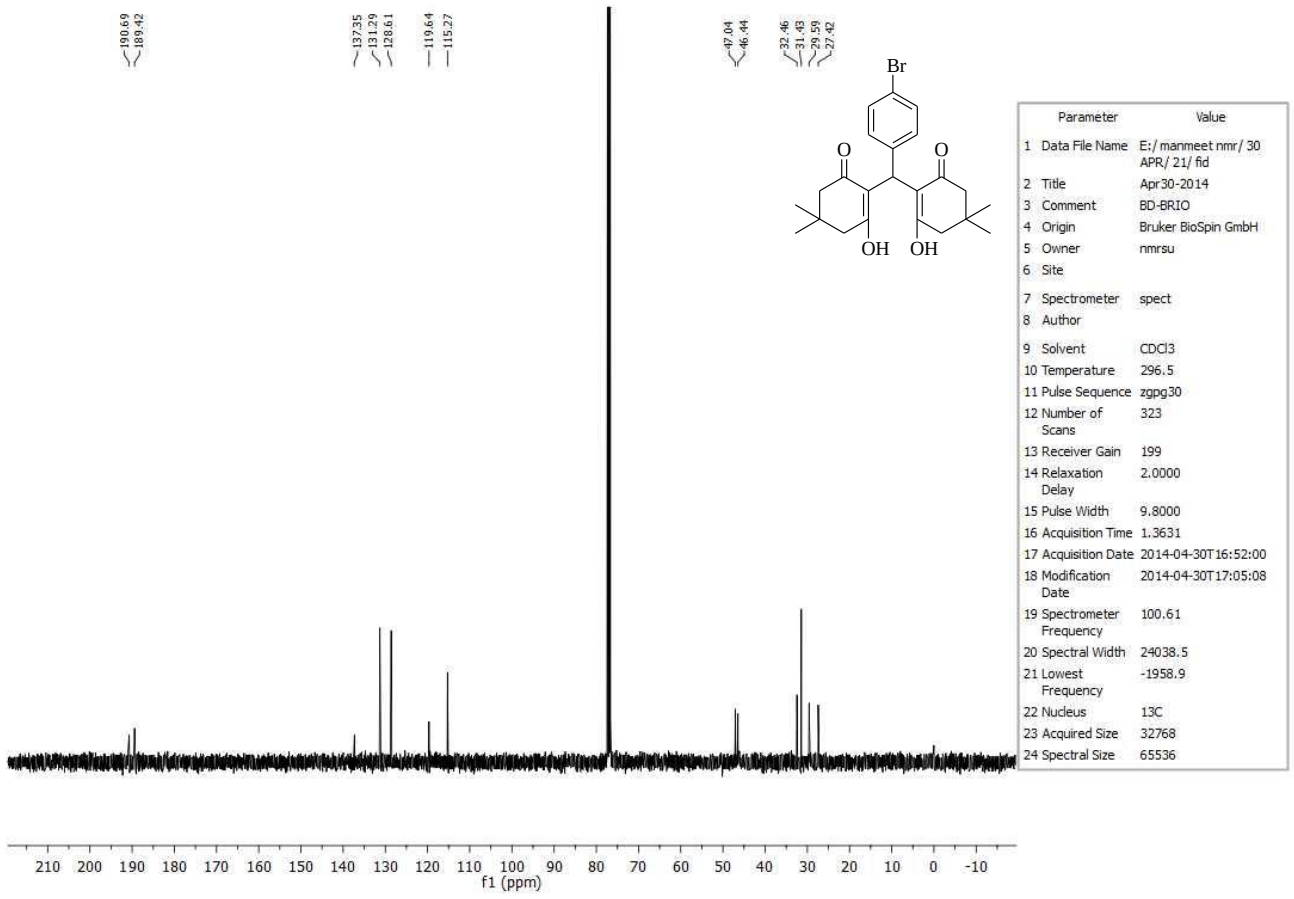
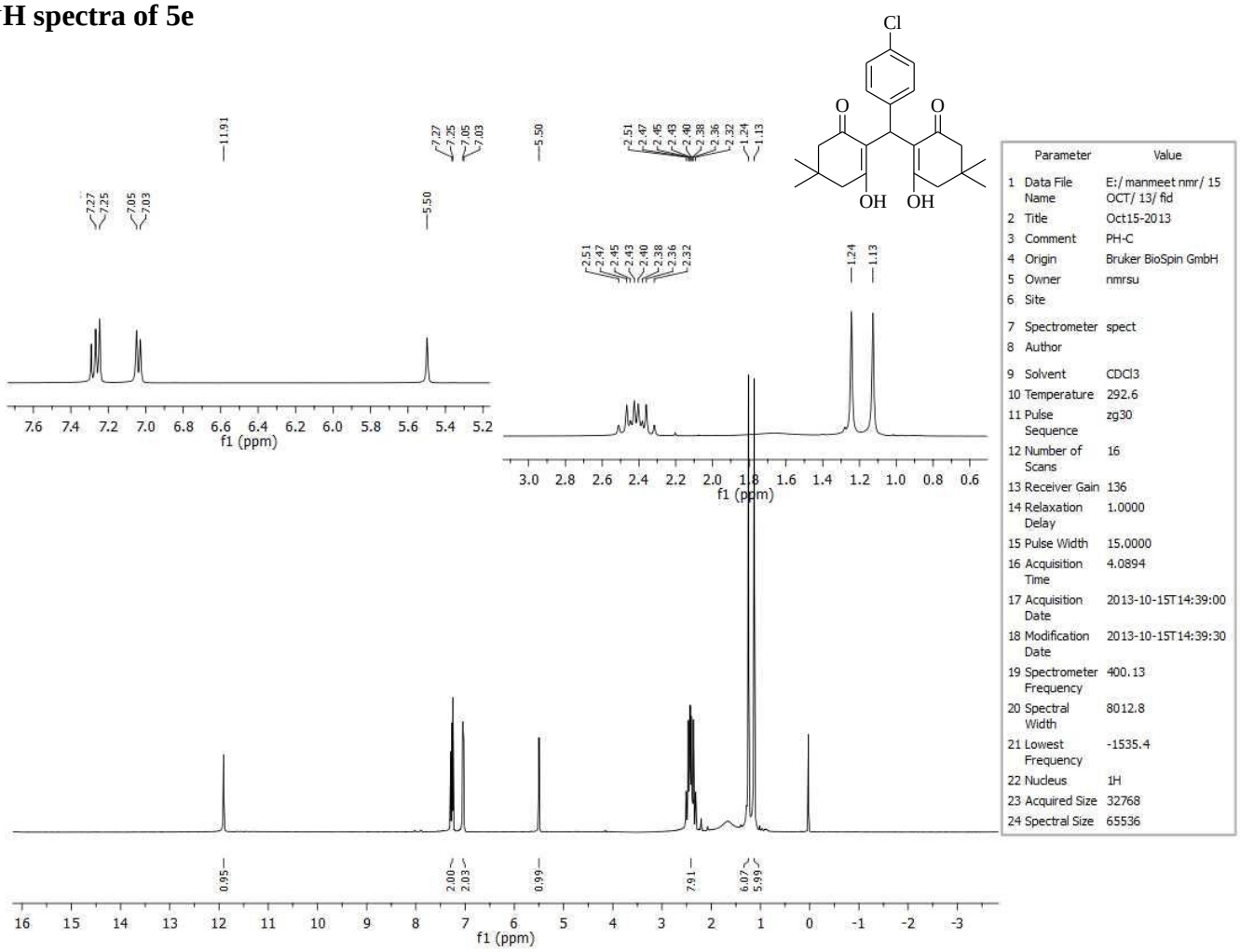
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.0
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	178
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-02T13:52:00
18	Modification Date	2014-05-02T13:52:40
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 5d



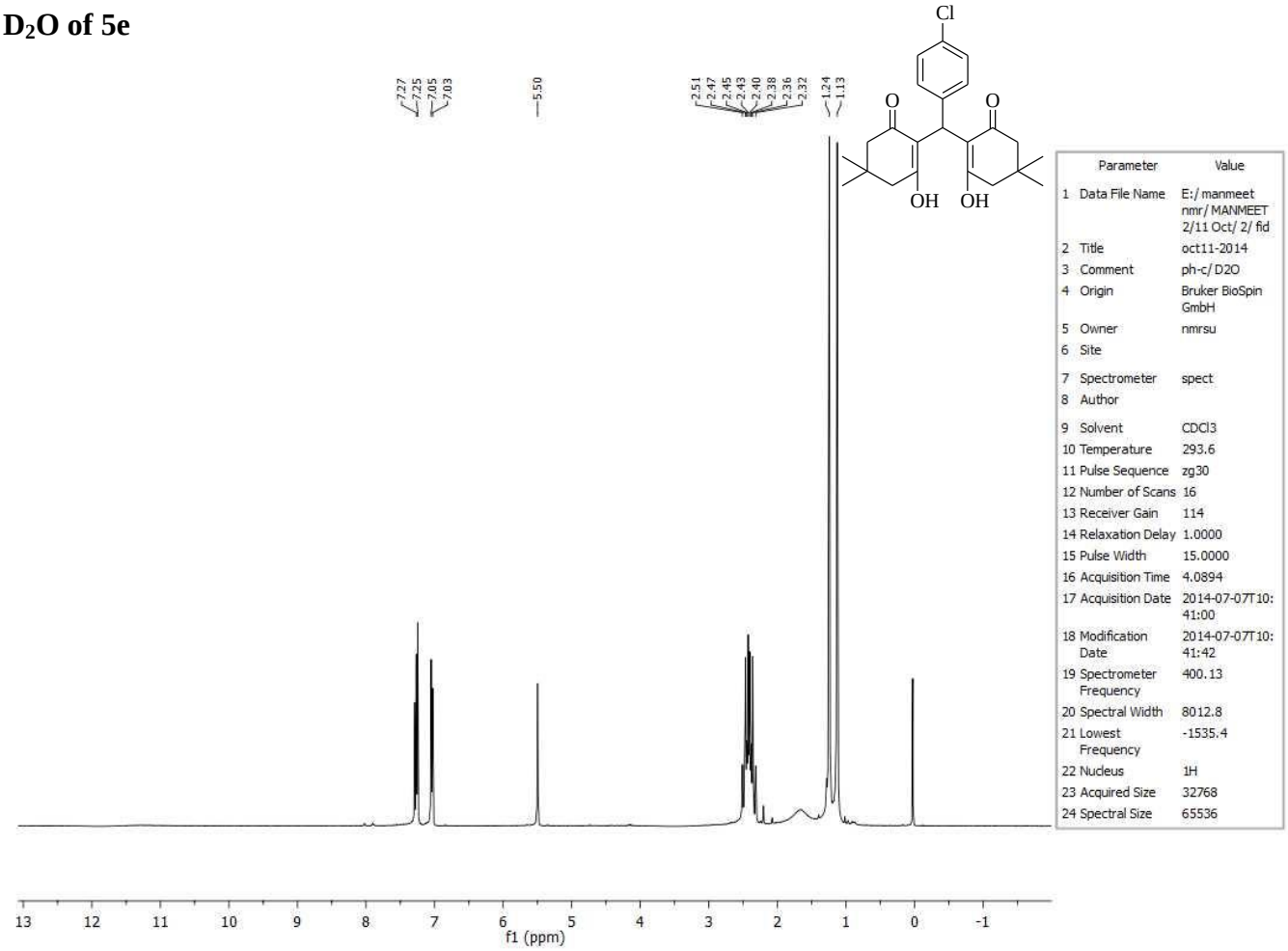
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 30 APR/ 21/ fid
2	Title	Apr30-2014
3	Comment	BD-BRIO
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	296.5
11	Pulse Sequence	zgpg30
12	Number of Scans	323
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-04-30T16:52:00
18	Modification Date	2014-04-30T17:05:08
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 5e



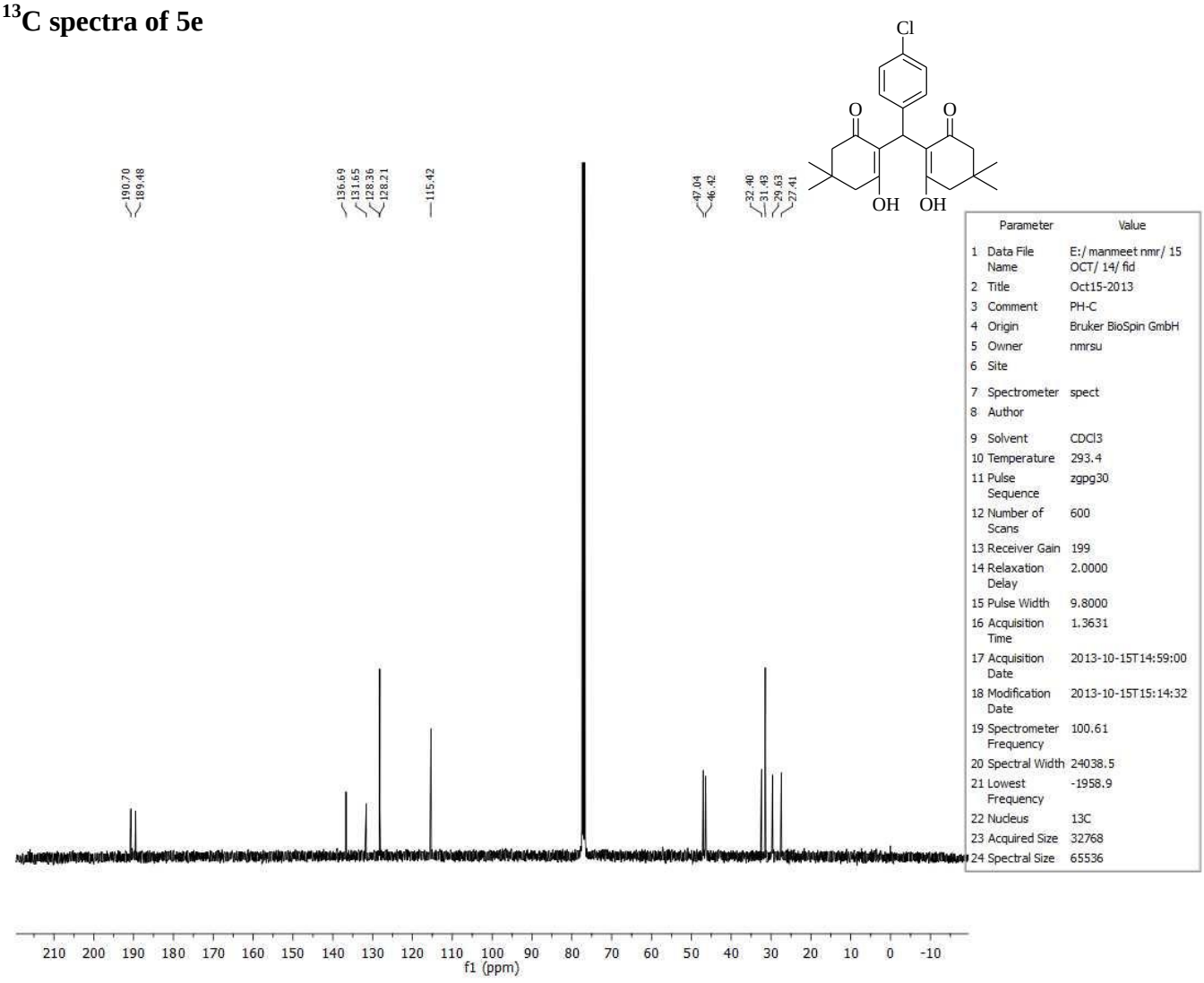
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 15 OCT/ 13/ fid
2	Title	Oct15-2013
3	Comment	PH-C
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	292.6
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	136
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2013-10-15T14:39:00
18	Modification Date	2013-10-15T14:39:30
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 5e

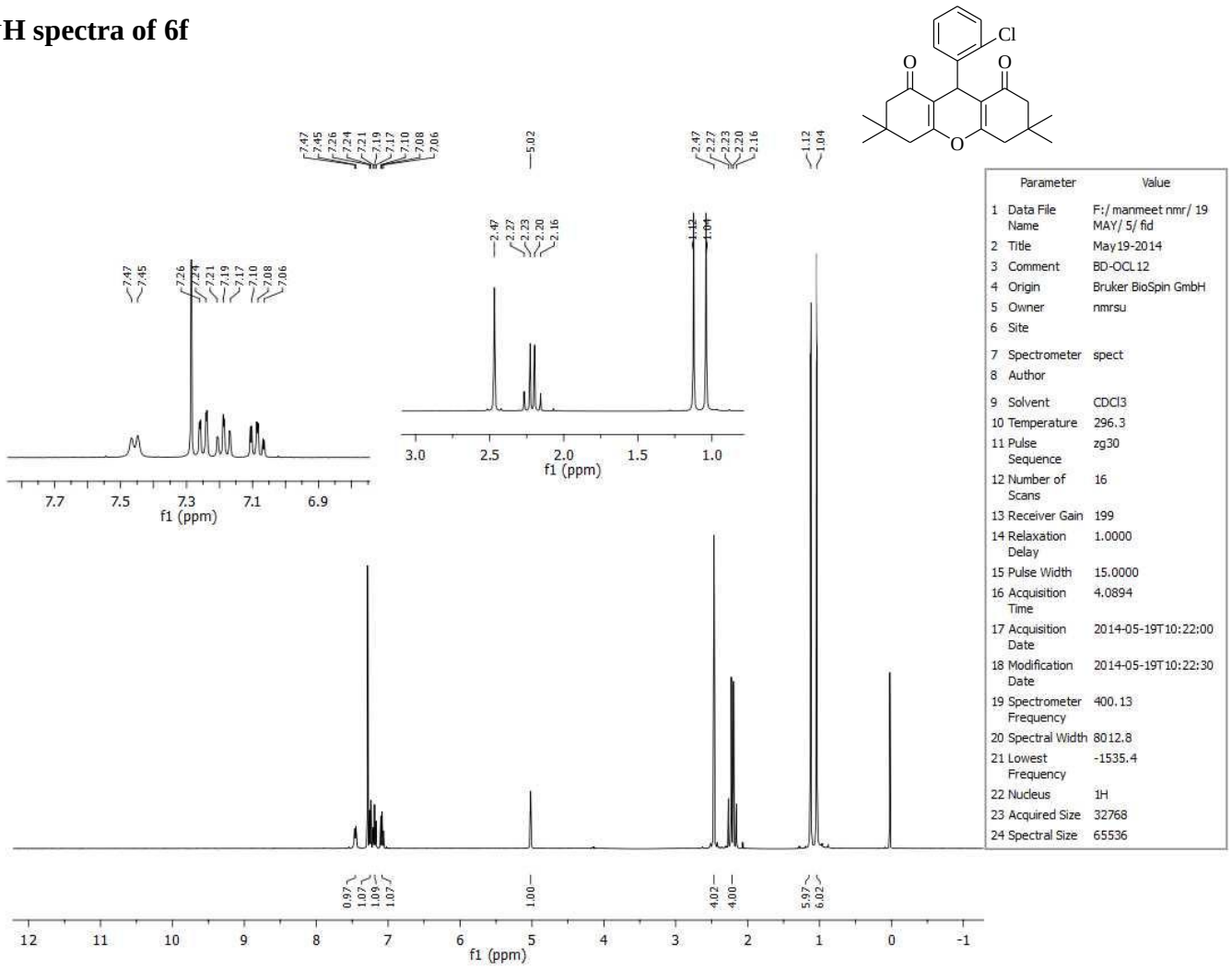


	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ MANMEET 2/11 Oct/ 2/ fid
2	Title	oct11-2014
3	Comment	ph-c/ D2O
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	293.6
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	114
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-07-07T10: 41:00
18	Modification Date	2014-07-07T10: 41:42
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 5e

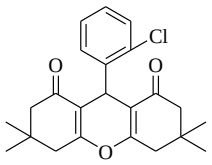


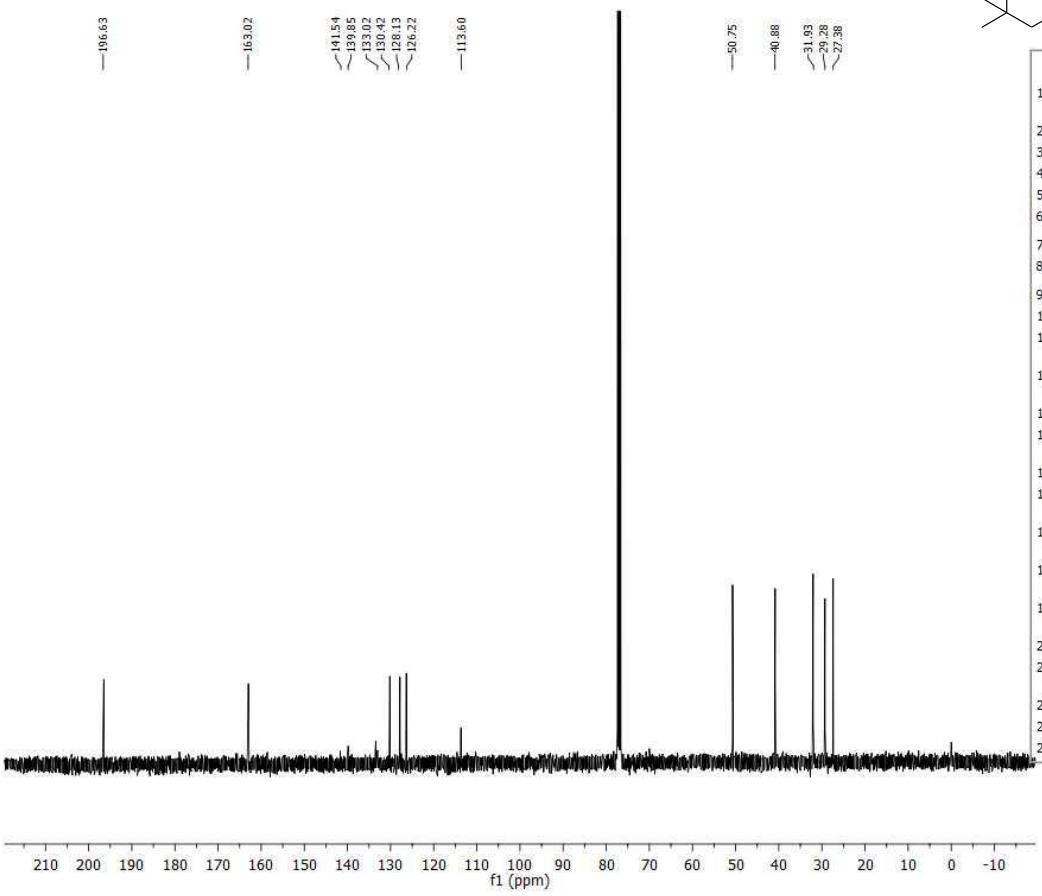
^{1}H spectra of 6f



	Parameter	Value
1	Data File Name	F:/ manmeet nmr/ 19 MAY/ 5/ fid
2	Title	May19-2014
3	Comment	BD-OCL12
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	296.3
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	199
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-19T10:22:00
18	Modification Date	2014-05-19T10:22:30
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

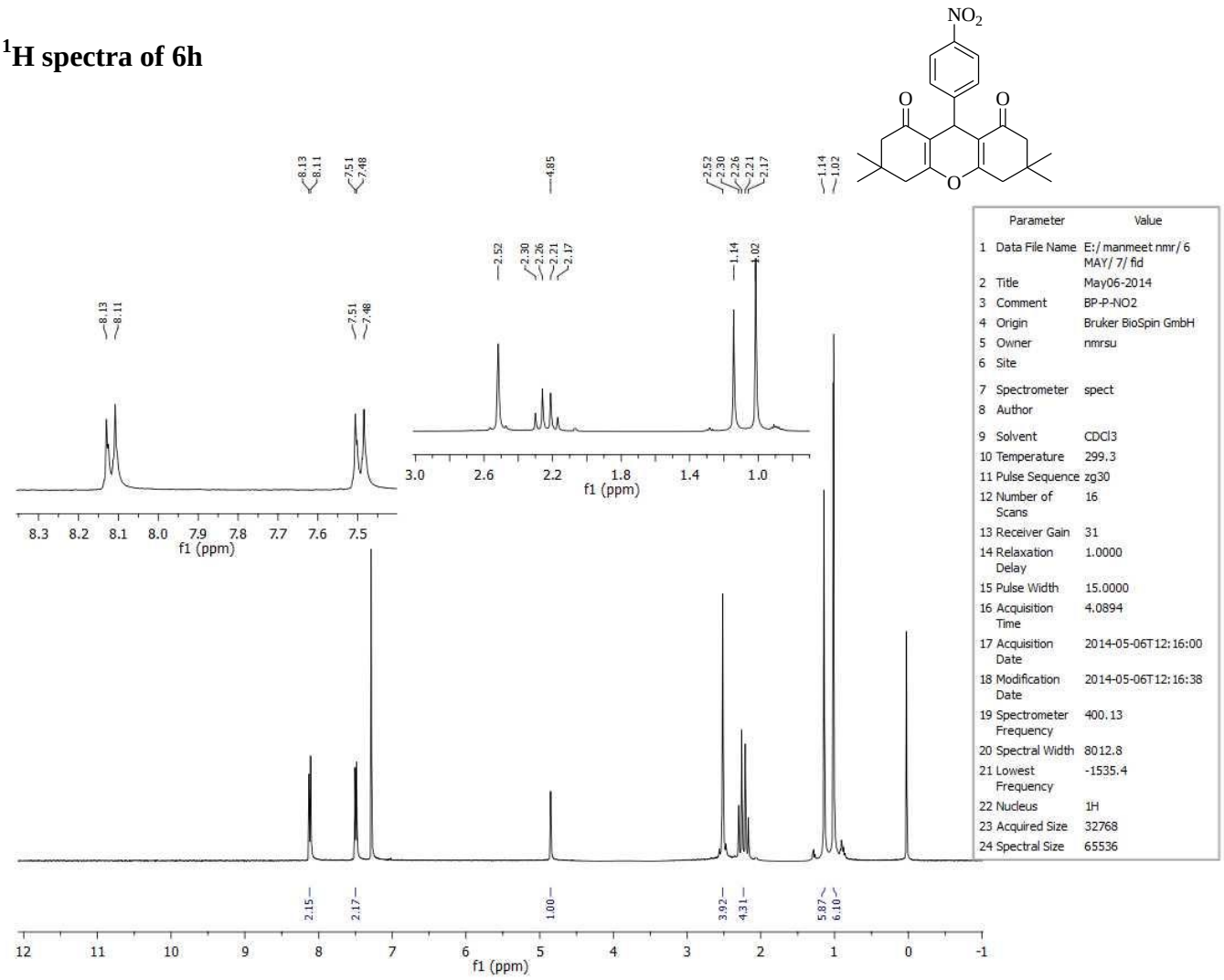
^{13}C spectra of 6f



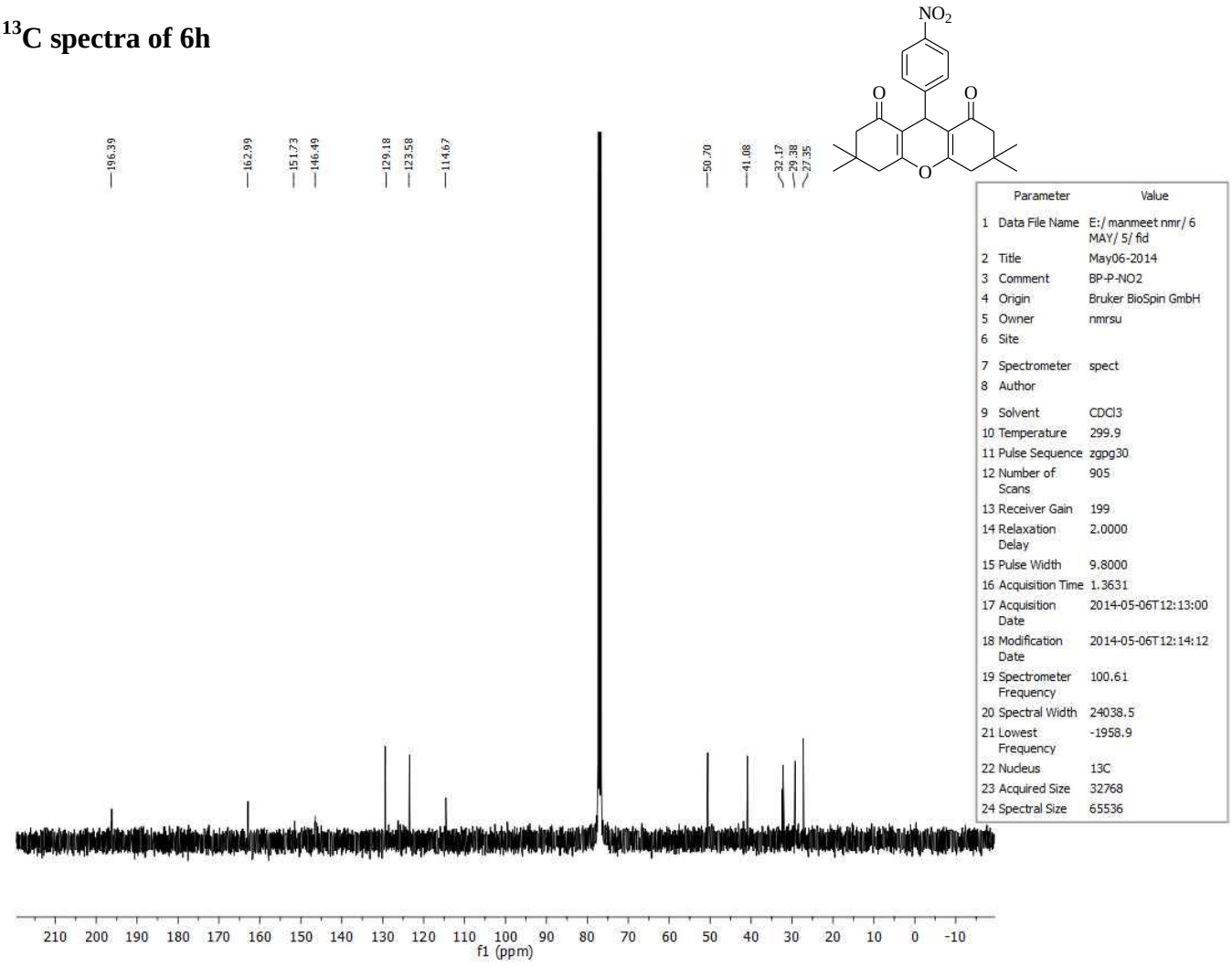


	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 19 MAY/ 6/ fid
2	Title	May19-2014
3	Comment	BD-OCL12
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	296.9
11	Pulse Sequence	zgpg30
12	Number of Scans	734
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-05-19T11:04:00
18	Modification Date	2014-05-19T11:04:50
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 6h

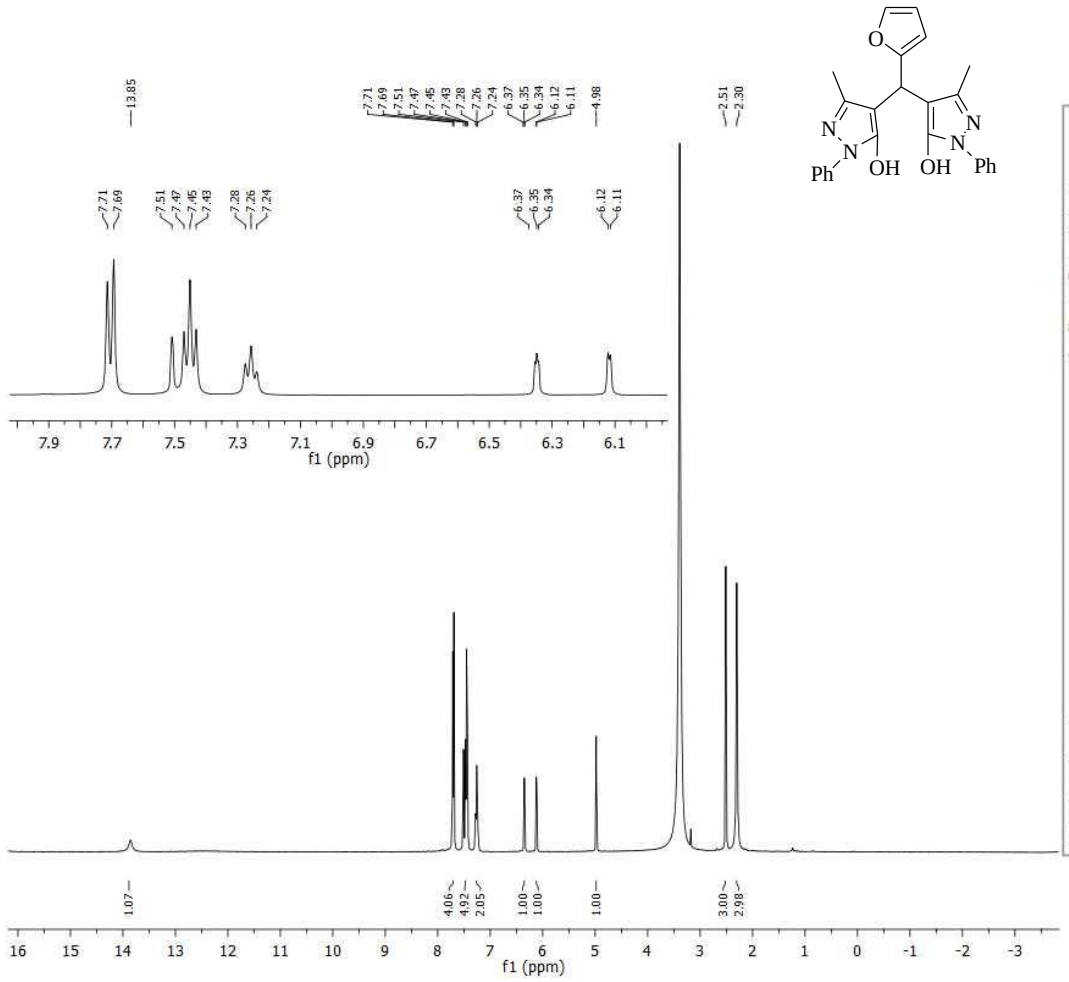


^{13}C spectra of 6h



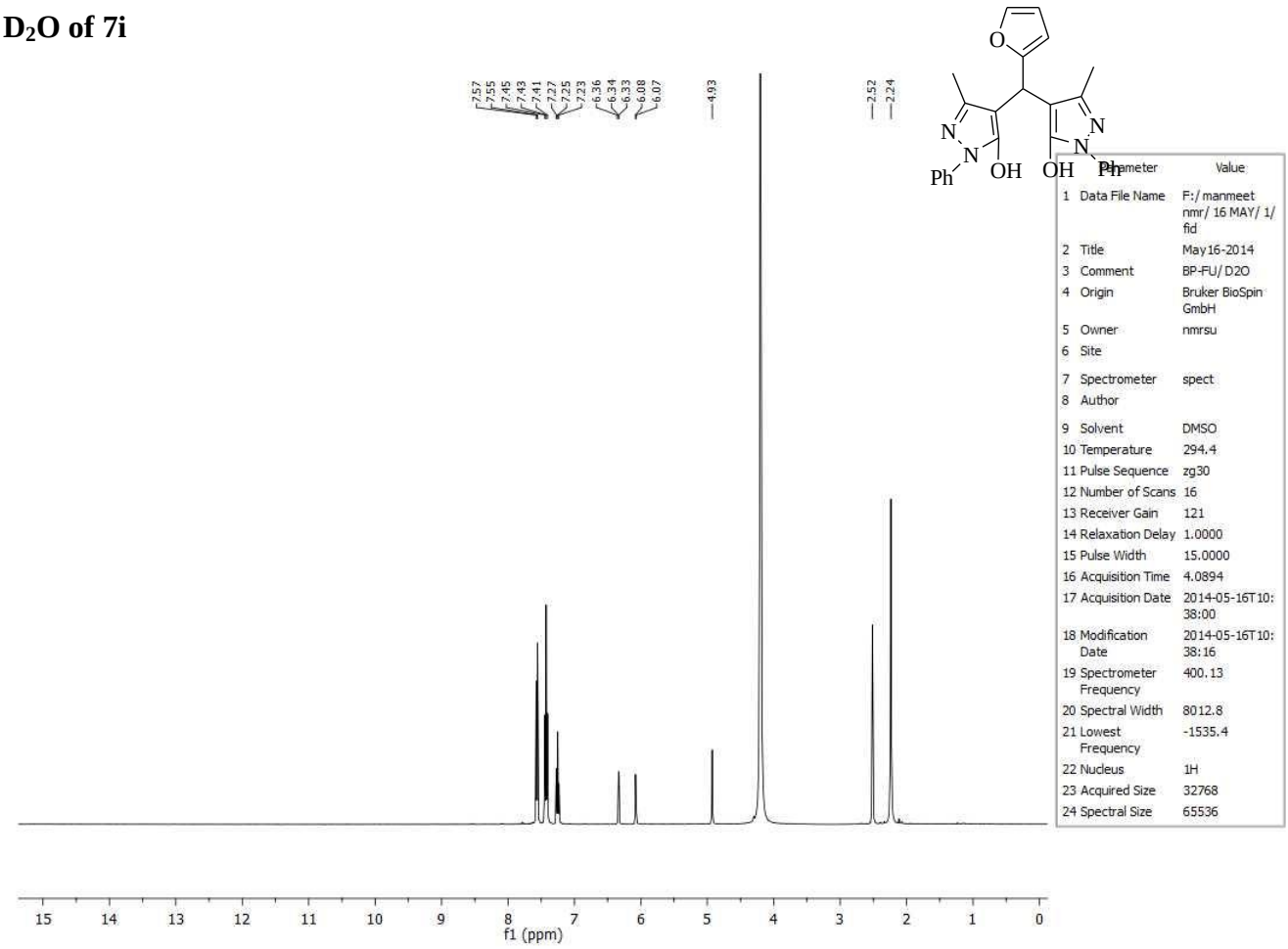
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 6 MAY/ 5/ fid
2	Title	May06-2014
3	Comment	BP-P-NO2
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	299.9
11	Pulse Sequence	zgpg30
12	Number of Scans	905
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-05-06T12:13:00
18	Modification Date	2014-05-06T12:14:12
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 7i



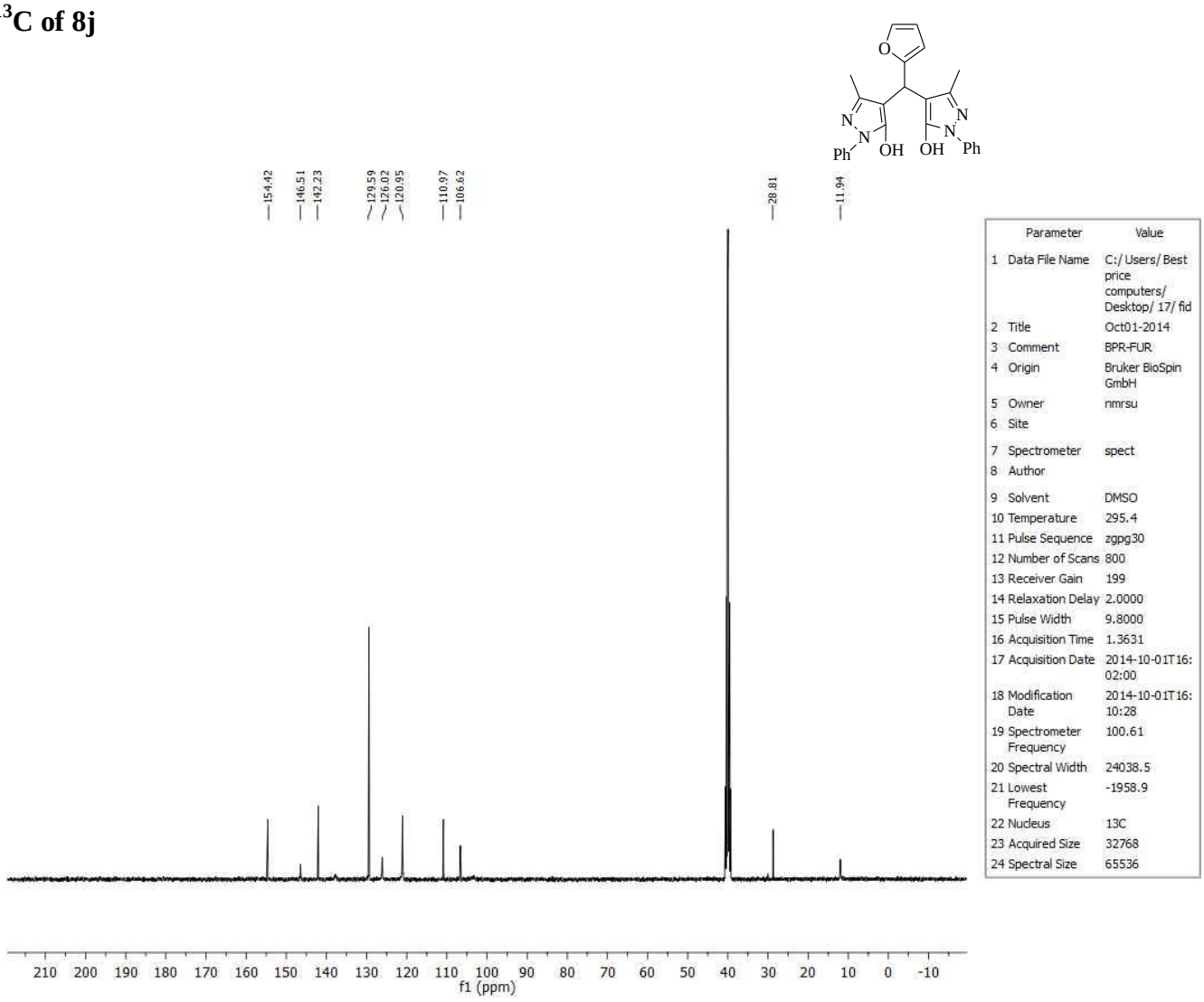
	Parameter	Value
1	Data File Name	F:/ manmeet nmr/ 15 MAY/ 24/ fid
2	Title	May15-2014
3	Comment	BP-FU
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	294.9
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	121
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-15T15:07:00
18	Modification Date	2014-05-15T15:07:40
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 7i



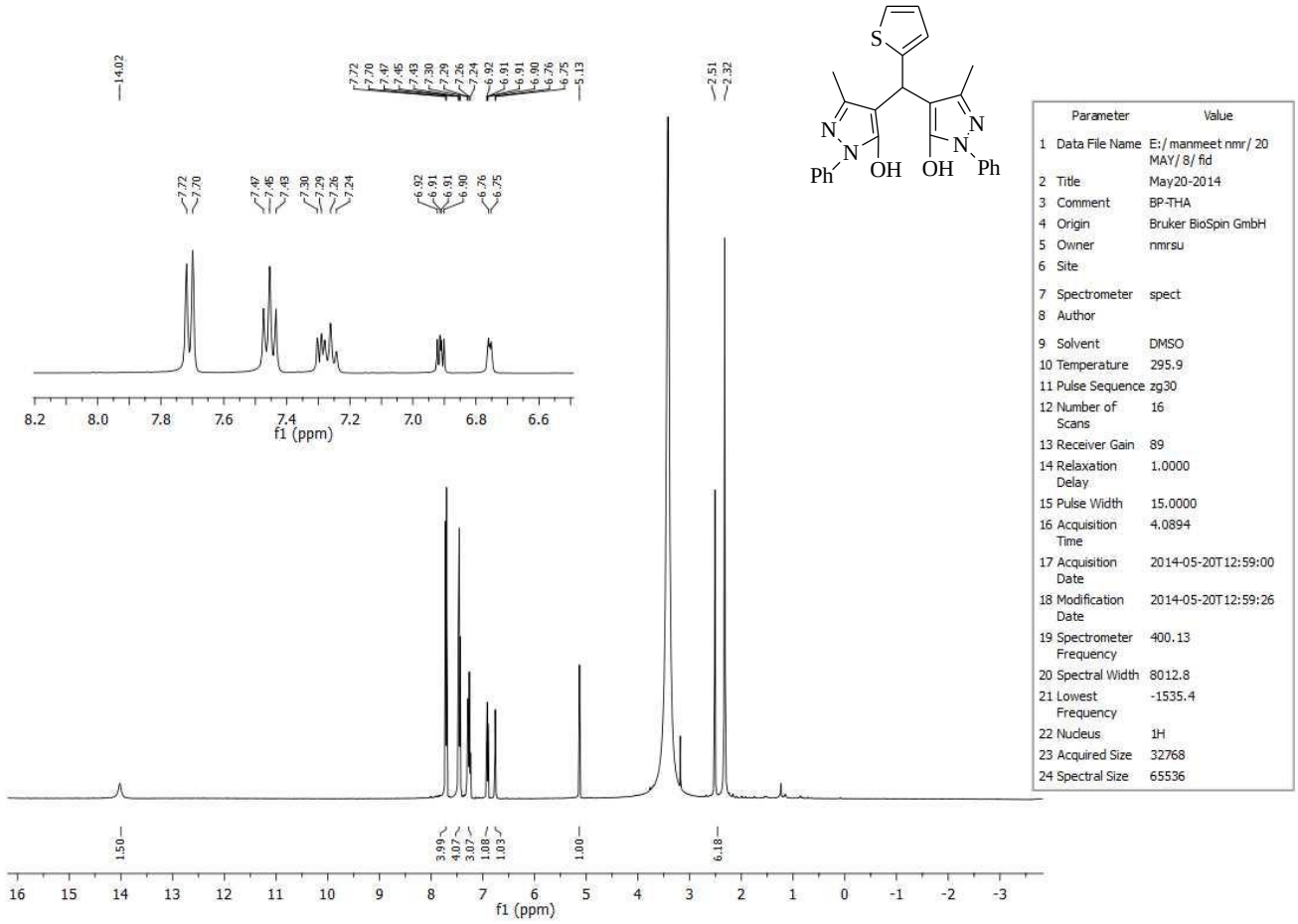
	Parameter	Value
1	Data File Name	F:/ manmeet nmr/ 16 MAY/ 1/ fid
2	Title	May16-2014
3	Comment	BP-FU/ D2O
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	294.4
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	121
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-16T10:38:00
18	Modification Date	2014-05-16T10:38:16
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C of 8j



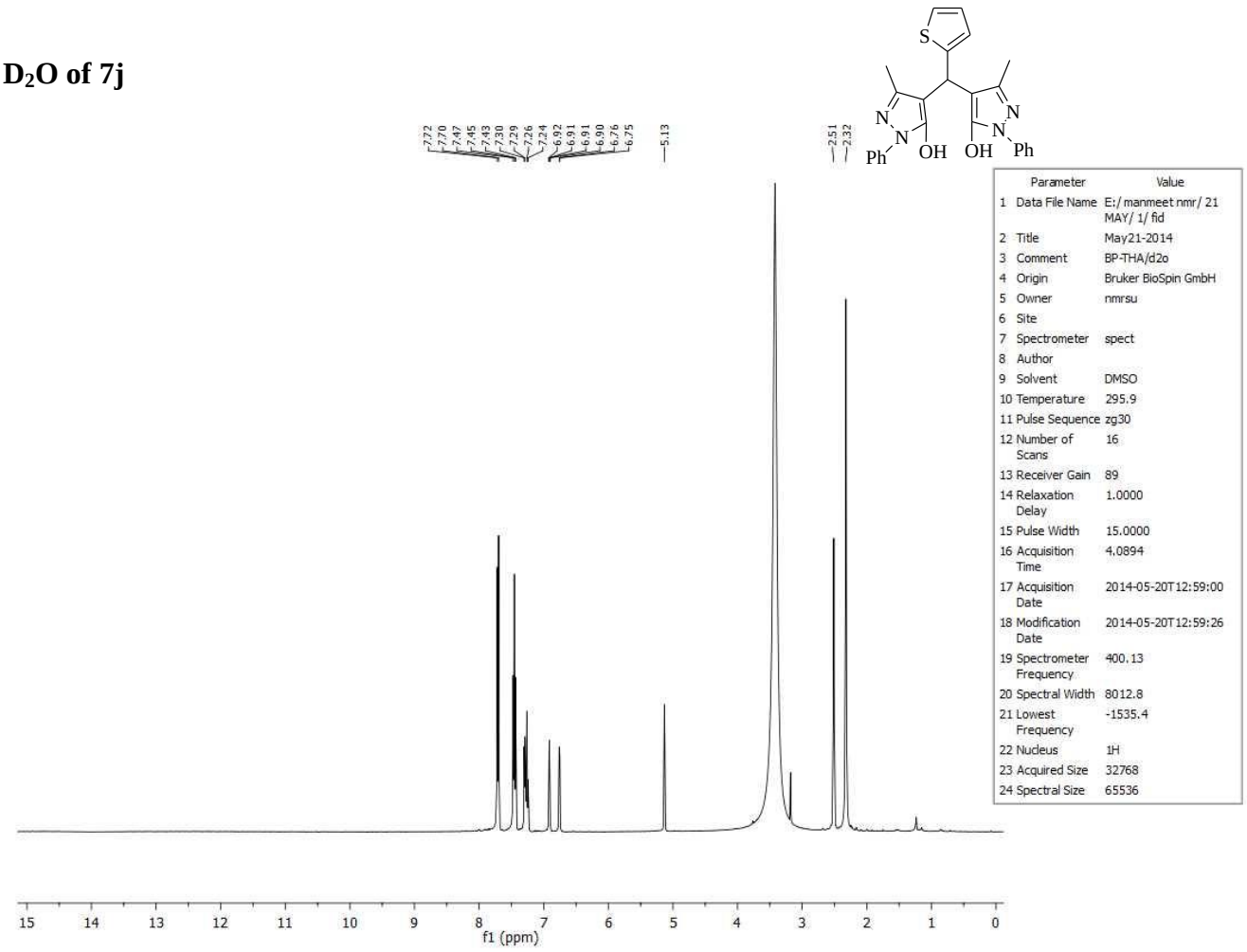
	Parameter	Value
1	Data File Name	C:/ Users/ Best price computers/ Desktop/ 17/ fid
2	Title	Oct01-2014
3	Comment	BPR-FUR
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	295.4
11	Pulse Sequence	zgpg30
12	Number of Scans	800
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-10-01T16:02:00
18	Modification Date	2014-10-01T16:10:28
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 7j

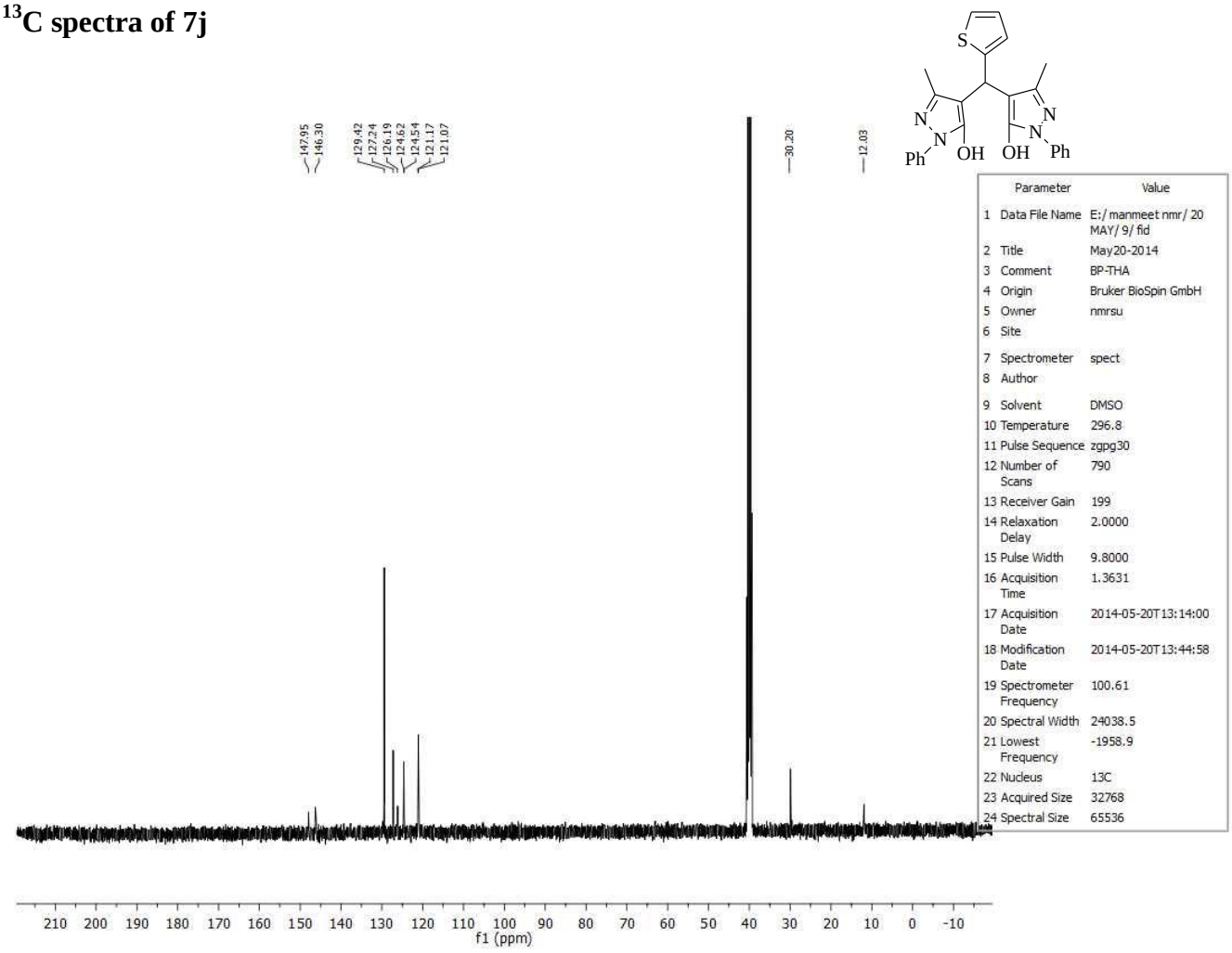


	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 20 MAY/ 8/ fid
2	Title	May20-2014
3	Comment	BP-THA
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	295.9
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	89
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-20T12:59:00
18	Modification Date	2014-05-20T12:59:26
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 7j

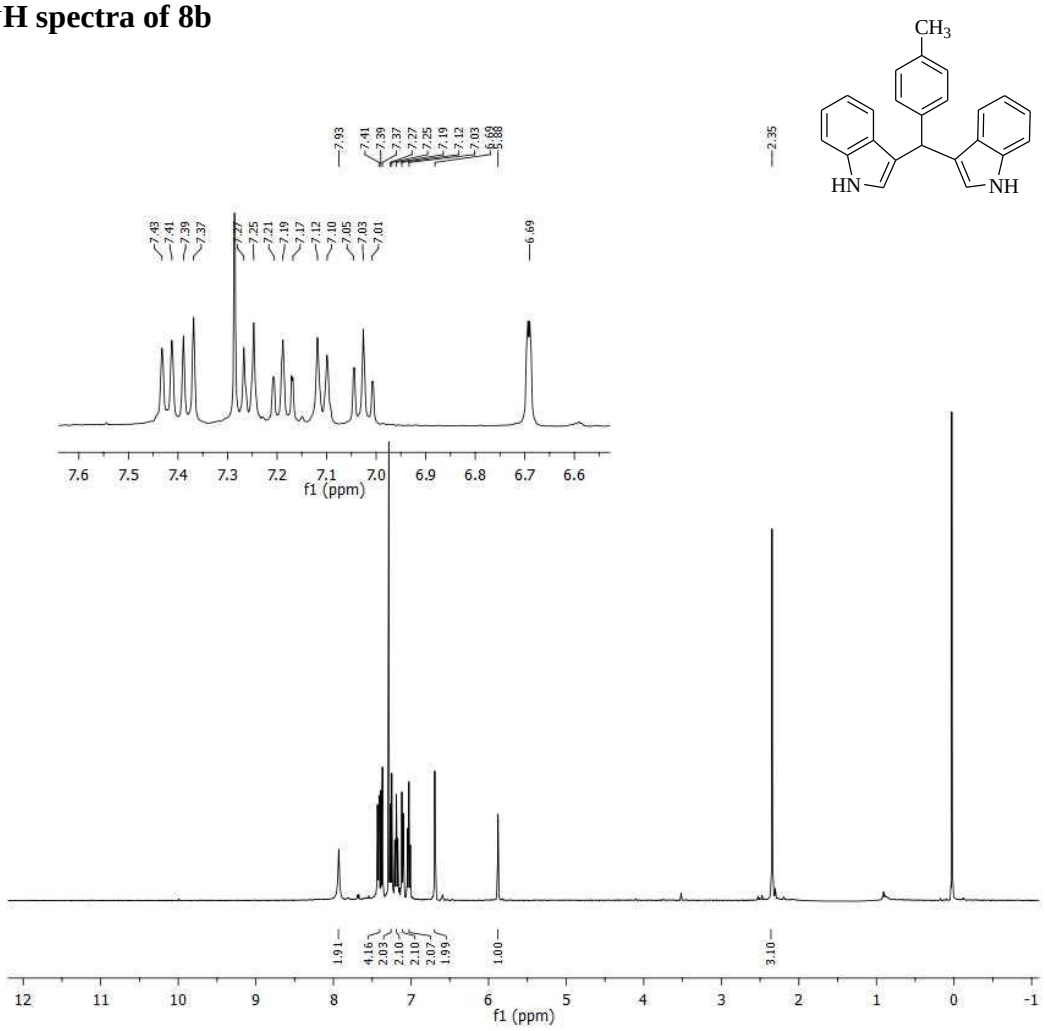

7.72
7.70
7.47
7.45
7.43
7.30
7.29
7.26
7.24
6.92
6.91
6.91
6.90
6.76
6.75
5.13
2.51
2.32
S
N
N
Ph
OH
OH
N
N
Ph
Parameter Value
1 Data File Name E:/manmeet nmr/21 MAY/1/fid
2 Title May21-2014
3 Comment BP-THA/d2o
4 Origin Bruker BioSpin GmbH
5 Owner nmrsu
6 Site
7 Spectrometer spect
8 Author
9 Solvent DMSO
10 Temperature 295.9
11 Pulse Sequence zg30
12 Number of Scans 16
13 Receiver Gain 89
14 Relaxation Delay 1.0000
15 Pulse Width 15.0000
16 Acquisition Time 4.0894
17 Acquisition Date 2014-05-20T12:59:00
18 Modification Date 2014-05-20T12:59:26
19 Spectrometer Frequency 400.13
20 Spectral Width 8012.8
21 Lowest Frequency -1535.4
22 Nucleus 1H
23 Acquired Size 32768
24 Spectral Size 65536
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

^{13}C spectra of 7j



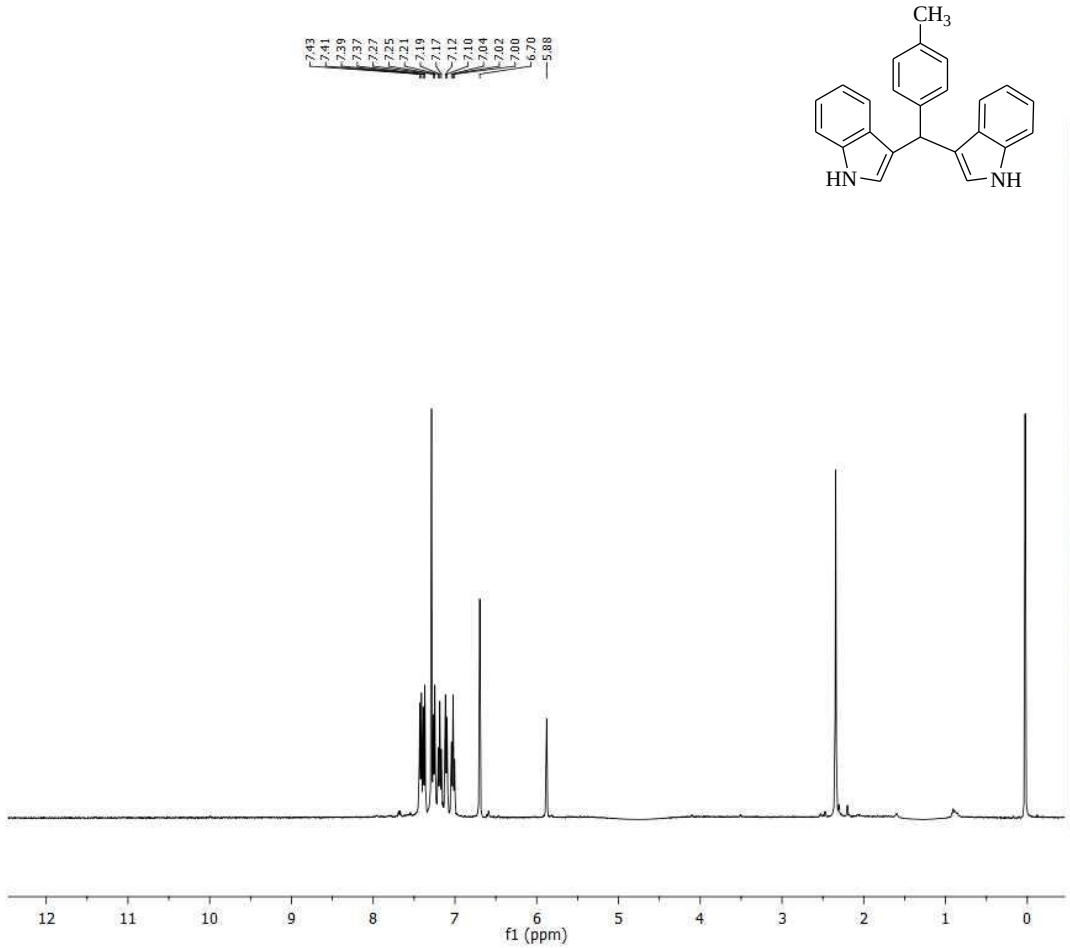
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 20 MAY/ 9/ fid
2	Title	May20-2014
3	Comment	BP-THA
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	296.8
11	Pulse Sequence	zgpg30
12	Number of Scans	790
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-05-20T13:14:00
18	Modification Date	2014-05-20T13:44:58
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 8b



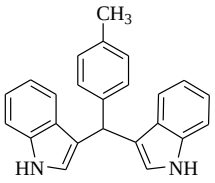
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 8 MAY/ 3/ fid
2	Title	May08-2014
3	Comment	BI-P-CH3
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.1
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	199
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-08T11:04:00
18	Modification Date	2014-05-08T11:04:14
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

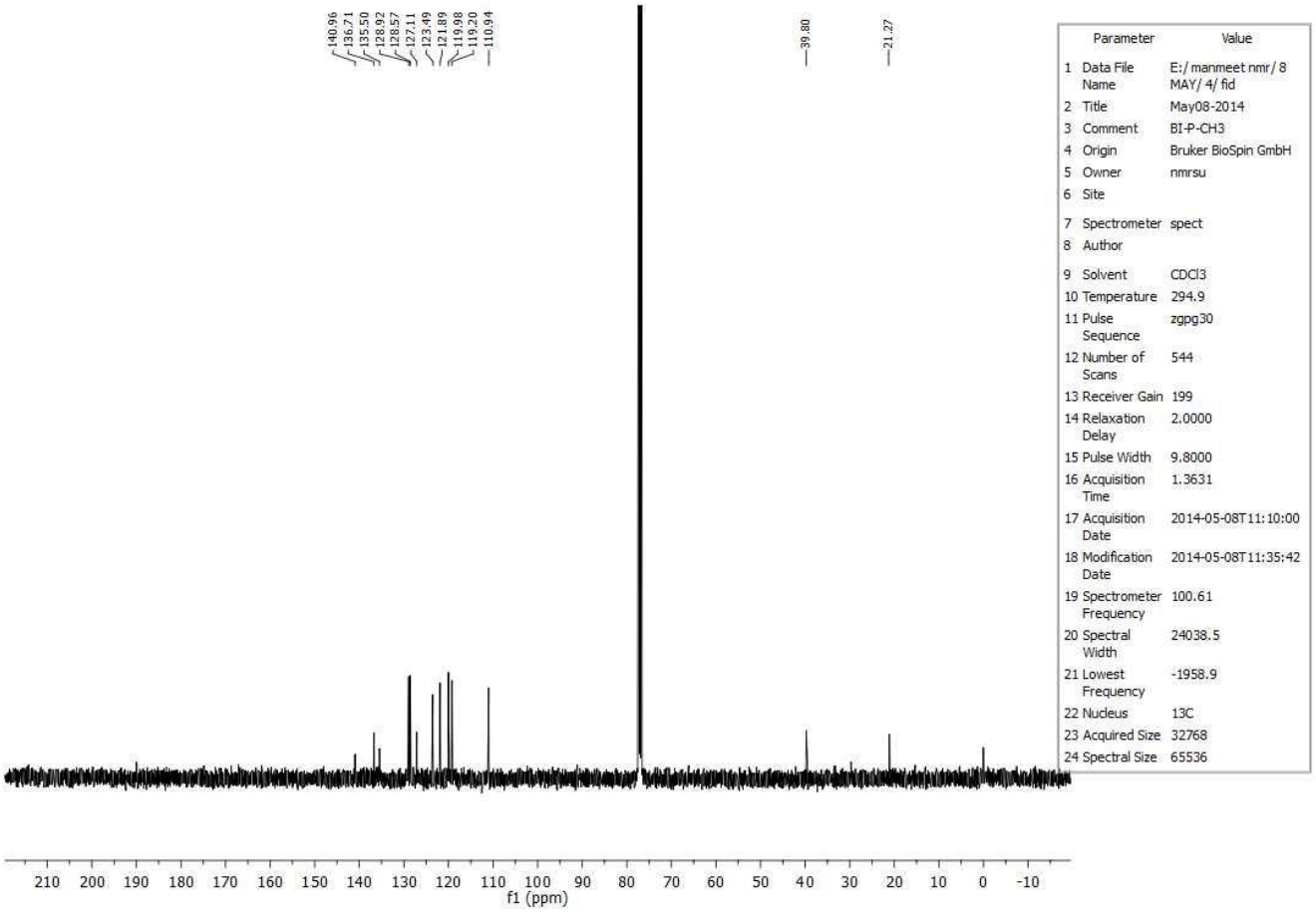
D_2O of 8b



	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 9 MAY/ 26/ fid
2	Title	May09-2014
3	Comment	BI-P-CH3
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	295.5
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	199
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-05-09T16:58:00
18	Modification Date	2014-05-09T16:58:30
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

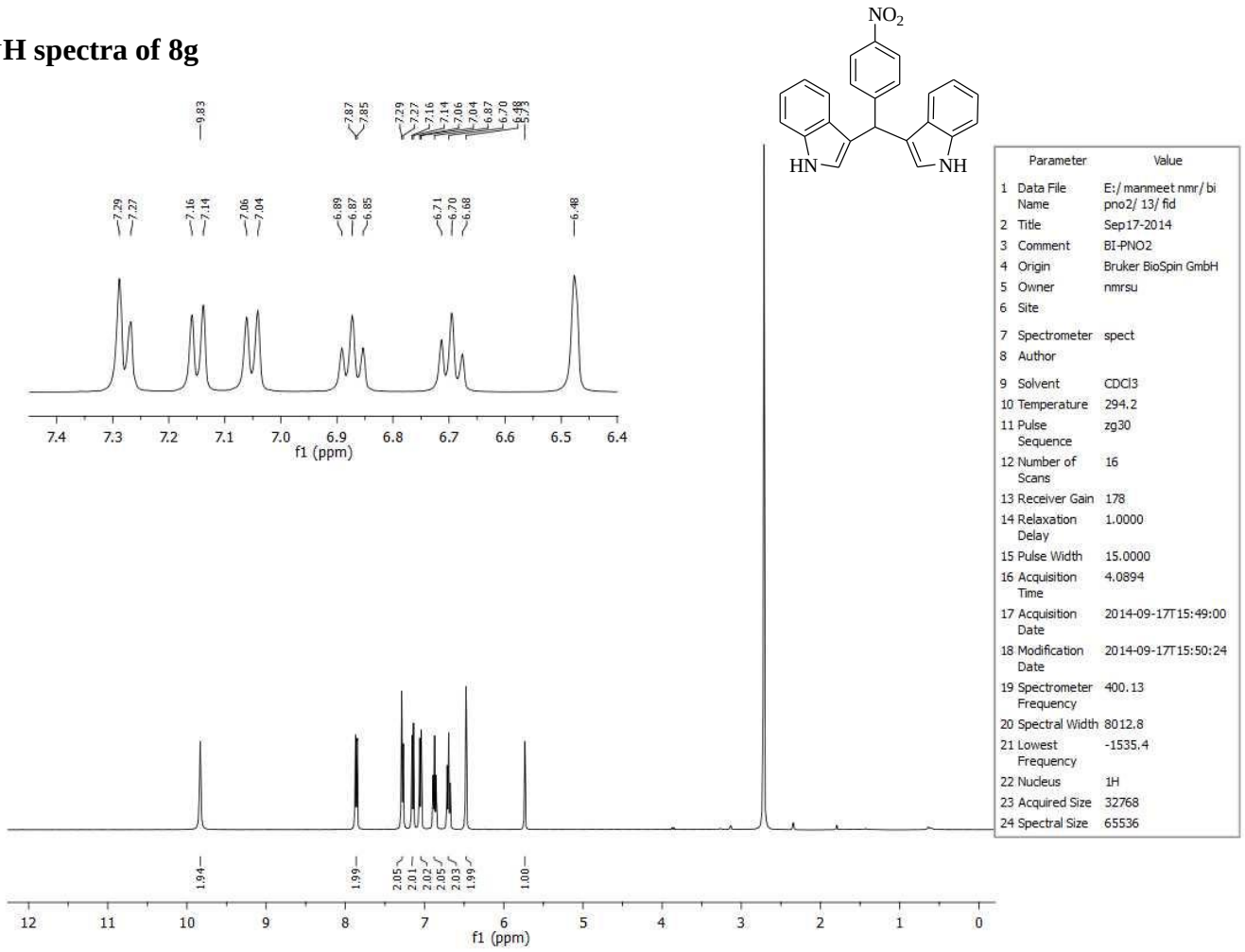
^{13}C spectra of 8b





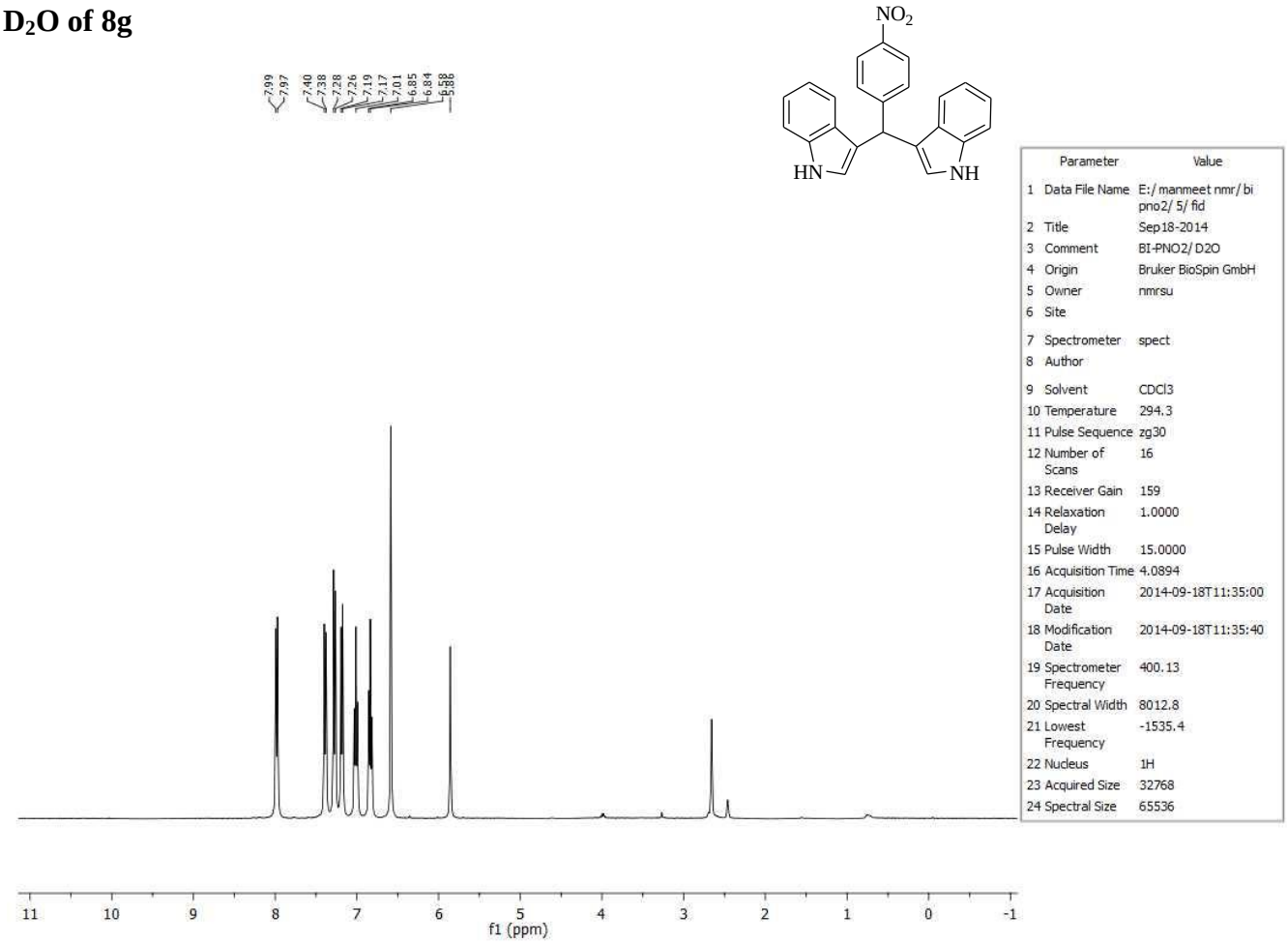
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ 8 MAY/ 4/ fid
2	Title	May08-2014
3	Comment	BI-P-CH3
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.9
11	Pulse Sequence	zgpg30
12	Number of Scans	544
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-05-08T11:10:00
18	Modification Date	2014-05-08T11:35:42
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 8g



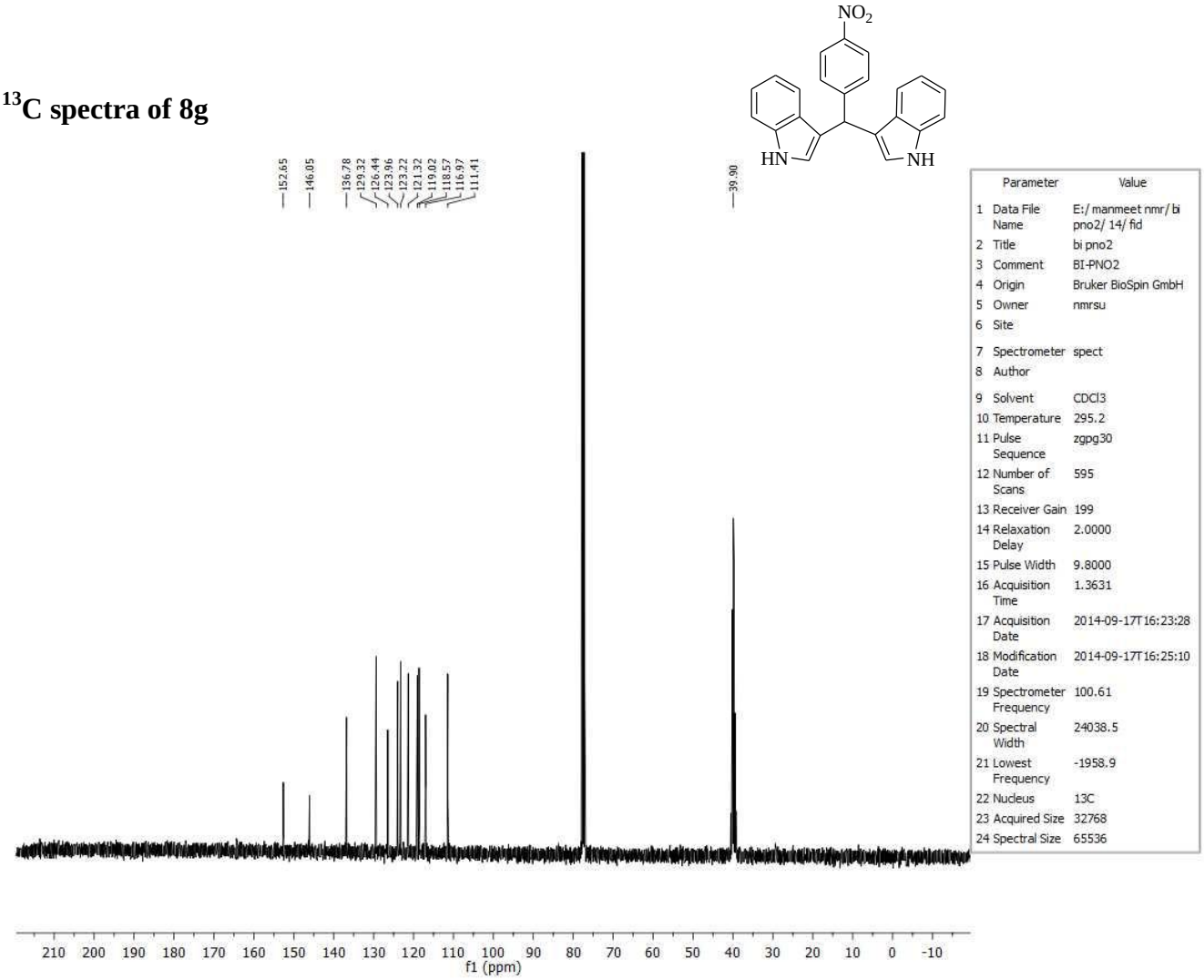
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ bi pno2/ 13/ fid
2	Title	Sep17-2014
3	Comment	BI-PNO2
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.2
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	178
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-09-17T15:49:00
18	Modification Date	2014-09-17T15:50:24
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 8g



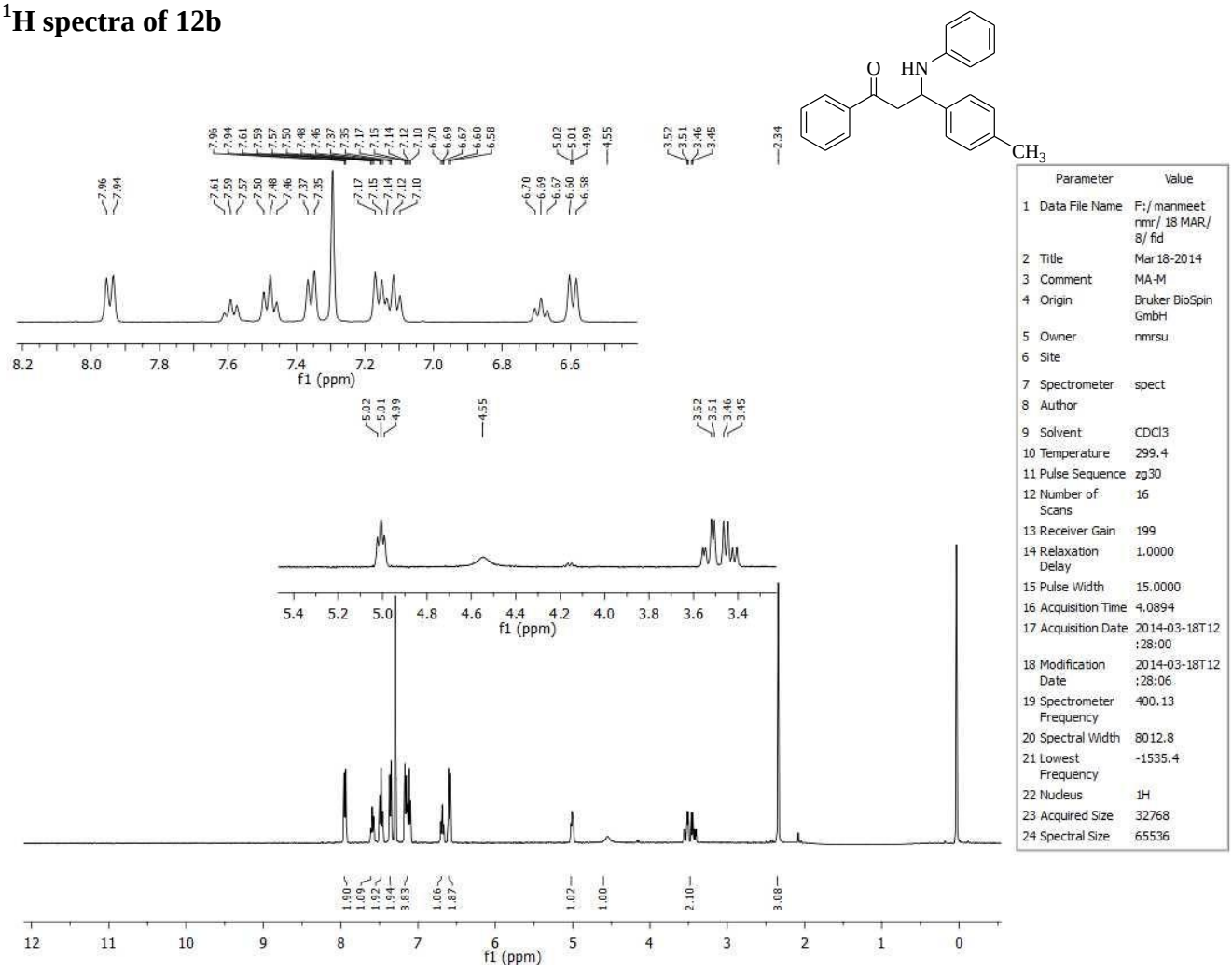
	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ bi pno2/ 5/ fid
2	Title	Sep18-2014
3	Comment	BI-PNO2/ D2O
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.3
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	159
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-09-18T11:35:00
18	Modification Date	2014-09-18T11:35:40
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 8g



	Parameter	Value
1	Data File Name	E:/ manmeet nmr/ bi pno2/ 14/ fid
2	Title	bi pno2
3	Comment	BI-PNO2
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	295.2
11	Pulse Sequence	zgpg30
12	Number of Scans	595
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-09-17T16:23:28
18	Modification Date	2014-09-17T16:25:10
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 12b



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2	Title	Mar18-2014
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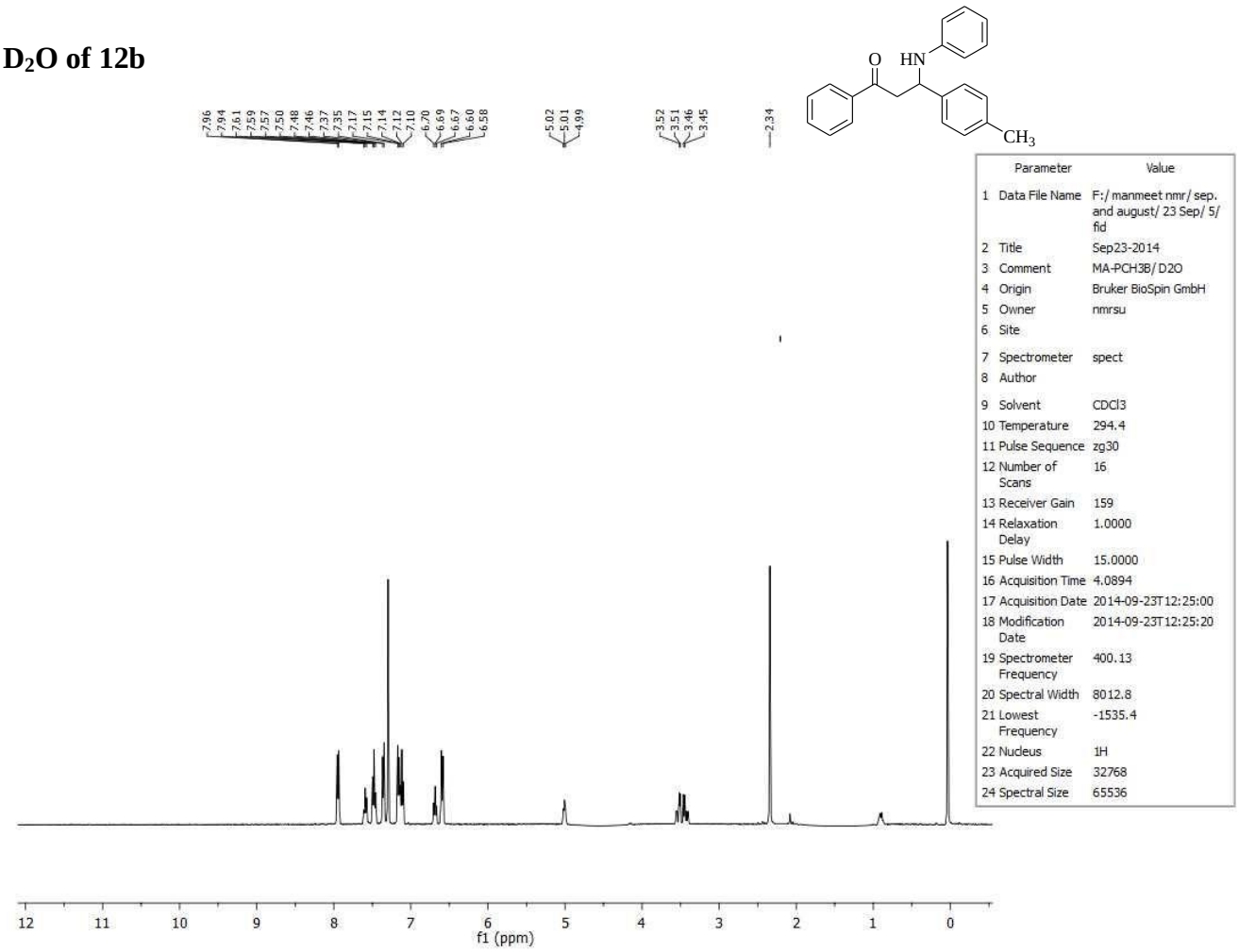
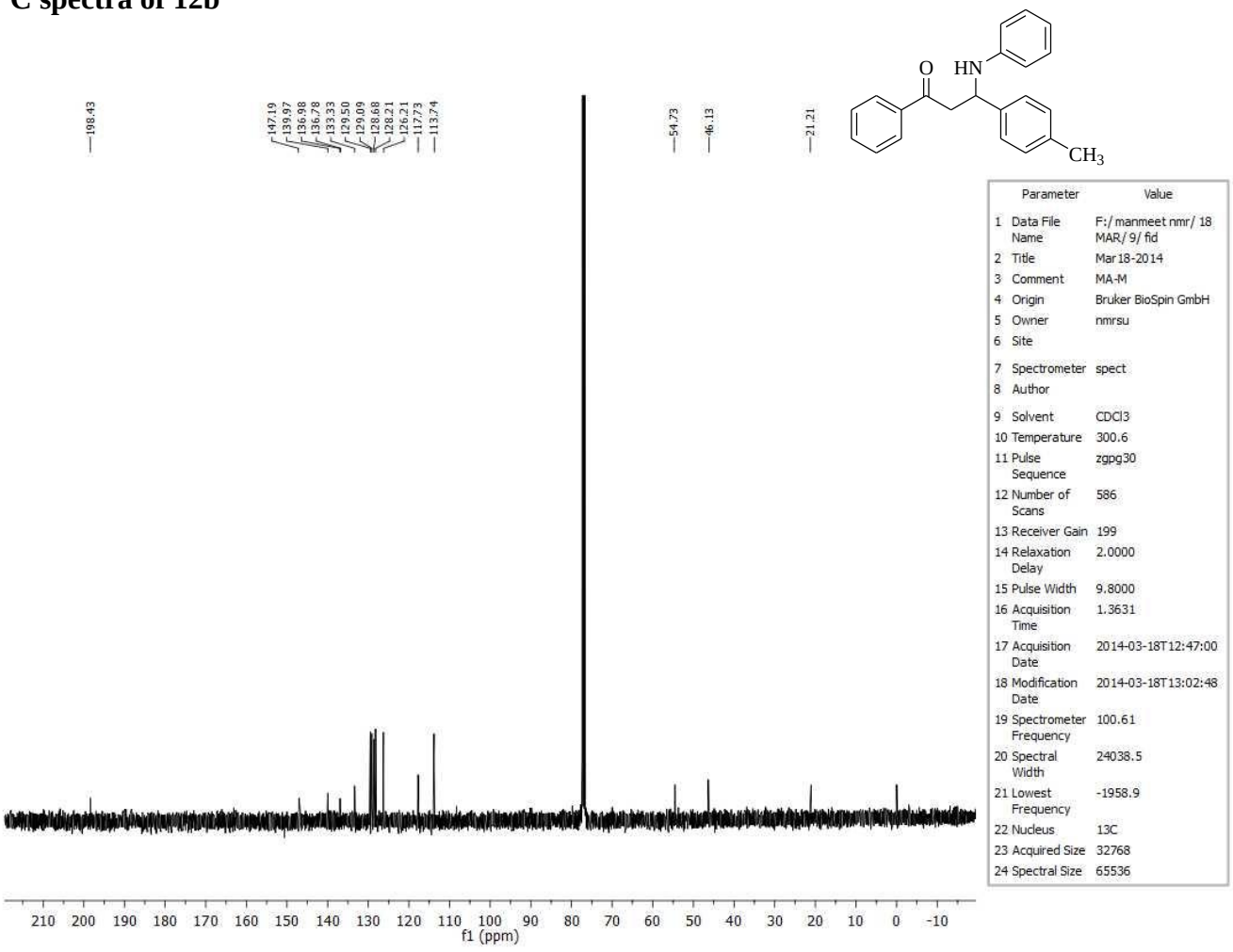
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	299.4
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	199
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-03-18T12 :28:00
18	Modification Date	2014-03-18T12 :28:06
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 12b


7.96
7.94
7.61
7.59
7.57
7.50
7.48
7.46
7.37
7.35
7.17
7.15
7.14
7.12
7.10
6.70
6.69
6.67
6.60
6.58
5.02
5.01
4.99
3.52
3.51
3.46
3.45
2.34
O
HN
CH3
Parameter Value
1 Data File Name F:/ manmeet nmr/ sep. and august/ 23 Sep/ 5/ fid
2 Title Sep23-2014
3 Comment MA-PCH3B/ D2O
4 Origin Bruker BioSpin GmbH
5 Owner nmrsu
6 Site
7 Spectrometer spect
8 Author
9 Solvent CDCl3
10 Temperature 294.4
11 Pulse Sequence zg30
12 Number of Scans 16
13 Receiver Gain 159
14 Relaxation Delay 1.0000
15 Pulse Width 15.0000
16 Acquisition Time 4.0894
17 Acquisition Date 2014-09-23T12:25:00
18 Modification Date 2014-09-23T12:25:20
19 Spectrometer Frequency 400.13
20 Spectral Width 8012.8
21 Lowest Frequency -1535.4
22 Nucleus 1H
23 Acquired Size 32768
24 Spectral Size 65536
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

^{13}C spectra of 12b



	Parameter	Value
1	Data File Name	F:/ manmeet nmr/ 18 MAR/ 9/ fid
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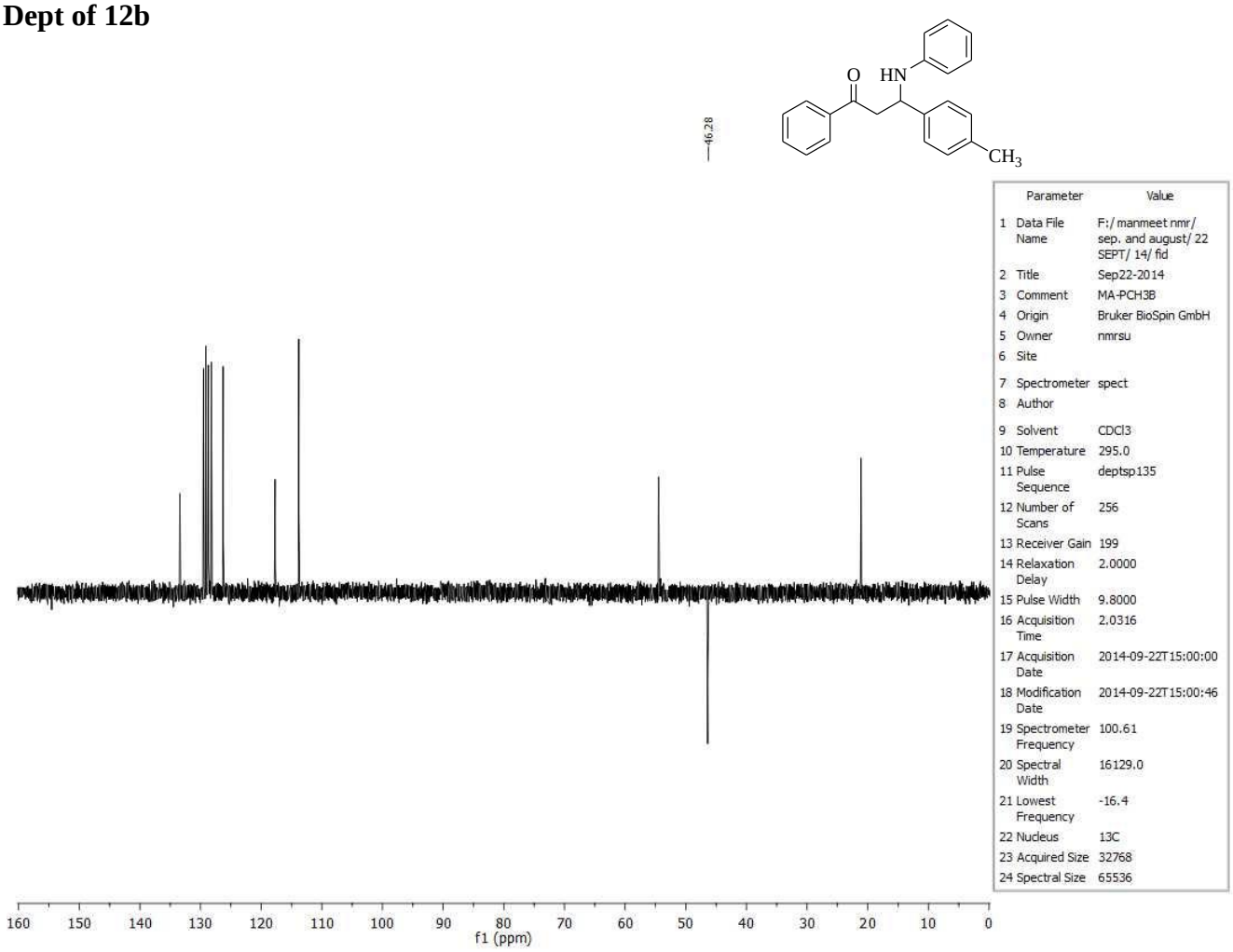
3	Comment	MA-M
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	300.6
11	Pulse Sequence	zgpg30
12	Number of Scans	586
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-03-18T12:47:00
18	Modification Date	2014-03-18T13:02:48
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

Dept of 12b



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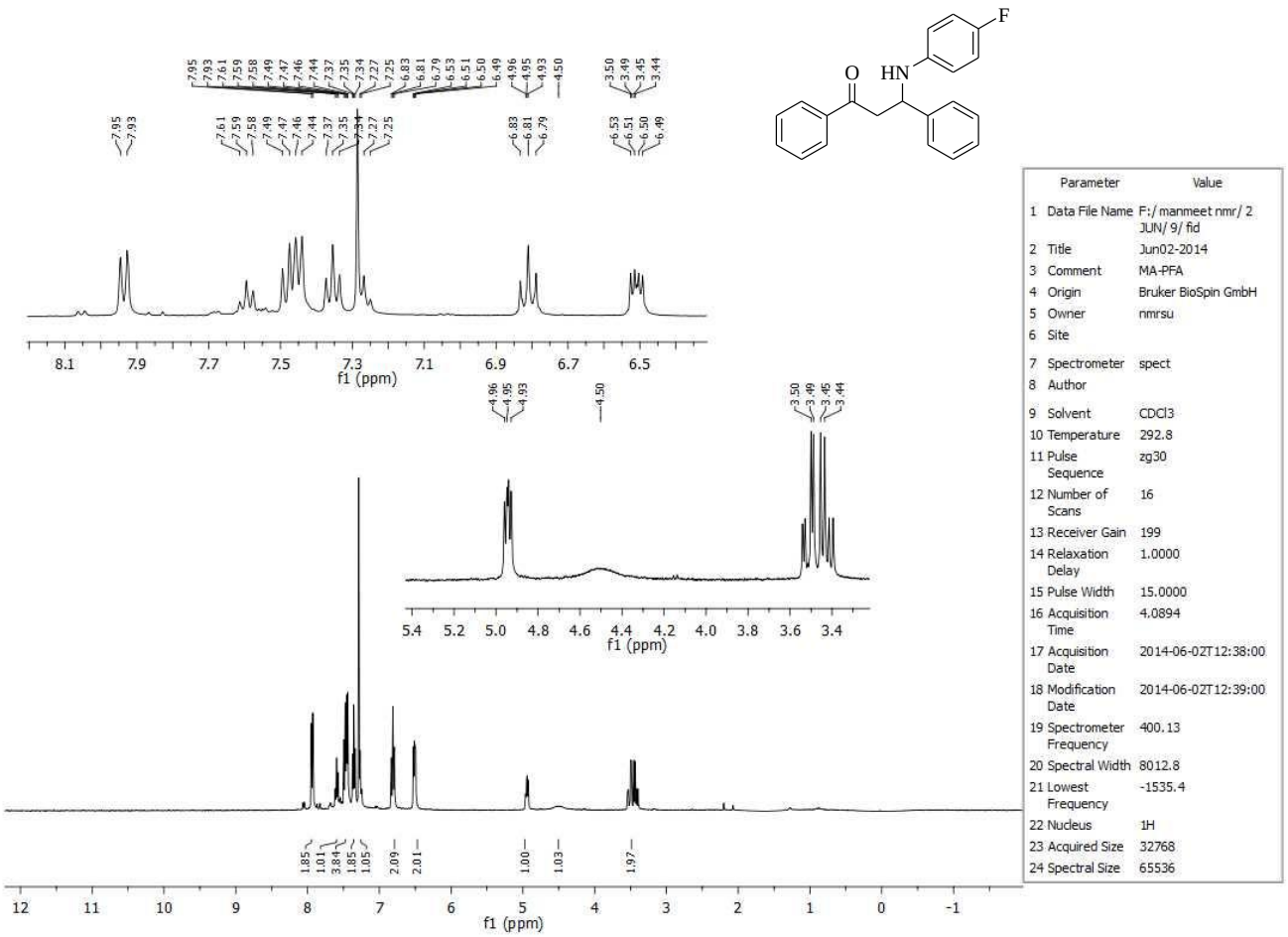
1	Data File Name	F:/ manmeet nmr/ sep. and august/ 22 SEPT/ 14/ fid
2	Title	Sep22-2014
3	Comment	MA-PCH3B
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	295.0
11	Pulse Sequence	deptsp135
12	Number of Scans	256
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	2.0316
17	Acquisition Date	2014-09-22T15:00:00
18	Modification Date	2014-09-22T15:00:46
19	Spectrometer Frequency	100.61
20	Spectral Width	16129.0
21	Lowest Frequency	-16.4
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 12g



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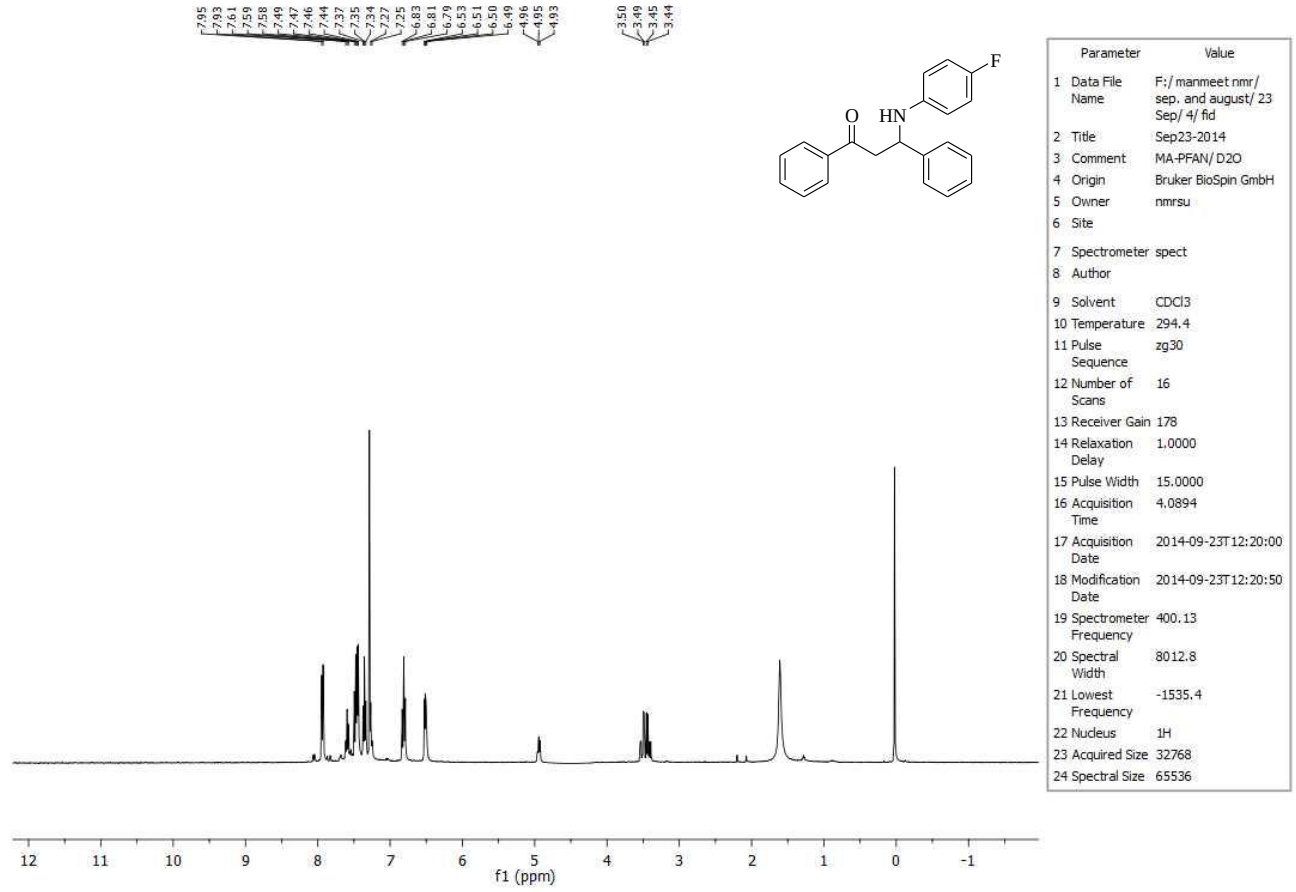
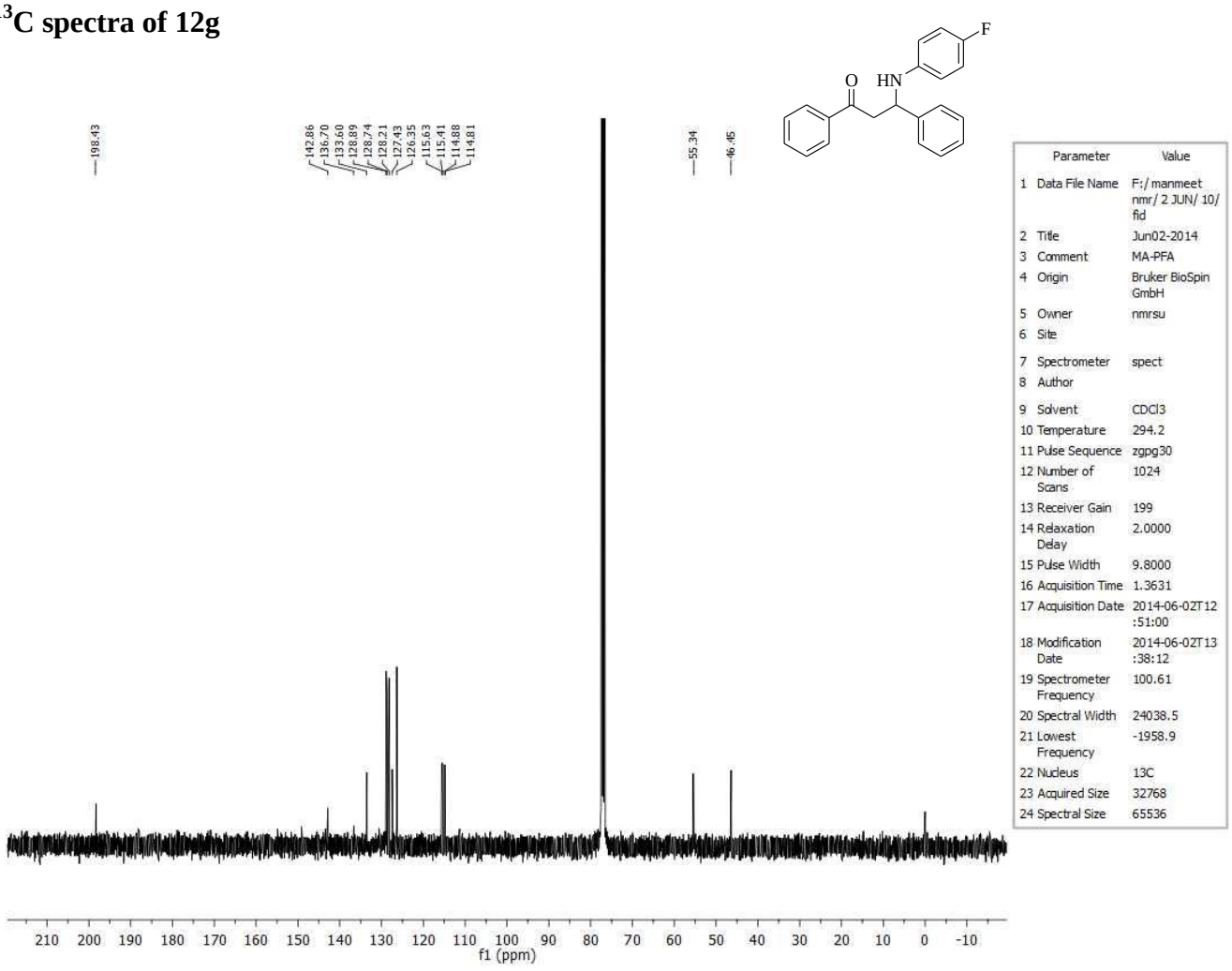
1	Data File Name	F:/ manmeet nmr/ 2 JUN/ 9/ fid
2	Title	Jun02-2014
3	Comment	MA-PFA
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	292.8
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	199
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-06-02T12:38:00
18	Modification Date	2014-06-02T12:39:00
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 12g



	Parameter	Value
1	Data File Name	F:/ manmeet nmr/ sep. and august/ 23 Sep/ 4/ fid
2	Title	Sep23-2014
3	Comment	MA-PFAN/ D2O
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.4
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	178
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-09-23T12:20:00
18	Modification Date	2014-09-23T12:20:50
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 12g



	Parameter	Value
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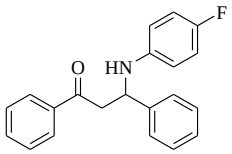
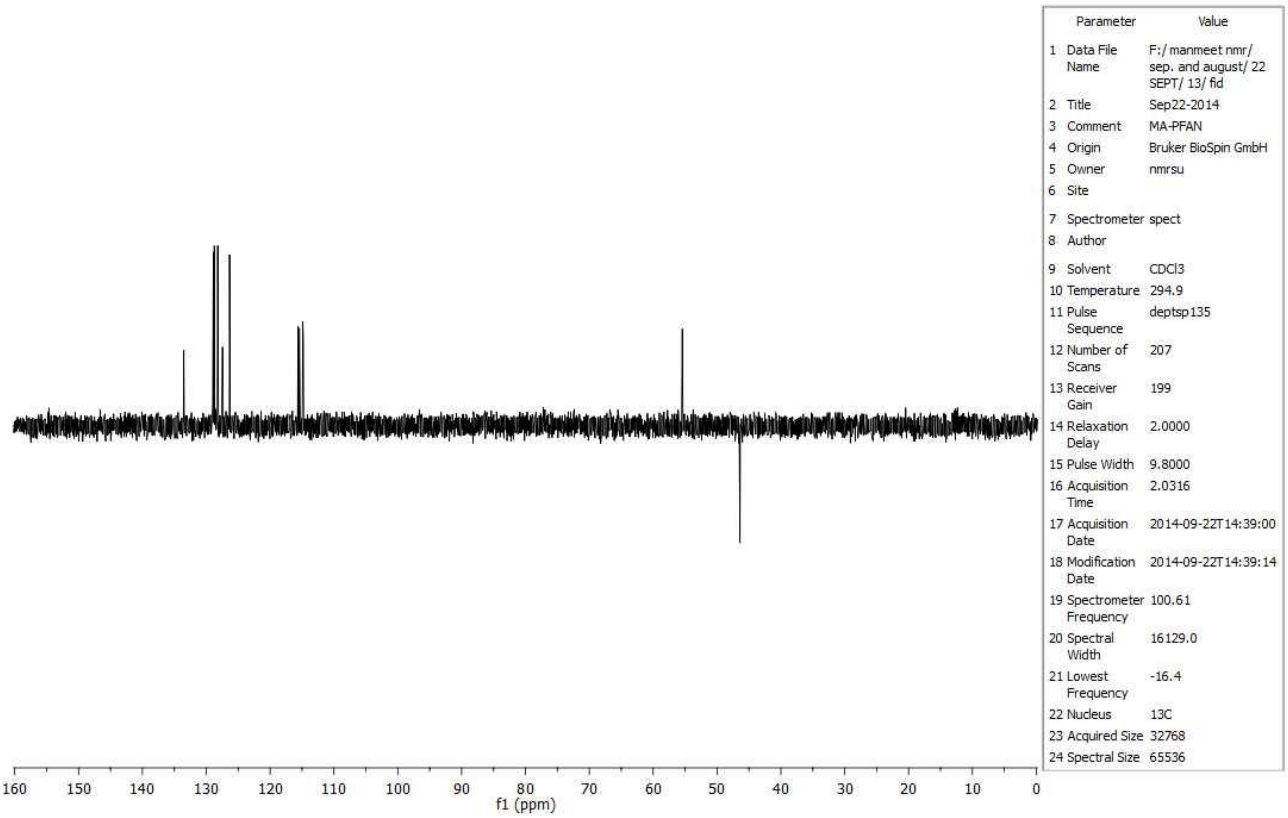
2	Title	Jun02-2014
3	Comment	MA-PFA
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.2
11	Pulse Sequence	zgpg30
12	Number of Scans	1024
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-06-02T12 :51:00
18	Modification Date	2014-06-02T13 :38:12
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

Dept of 12g





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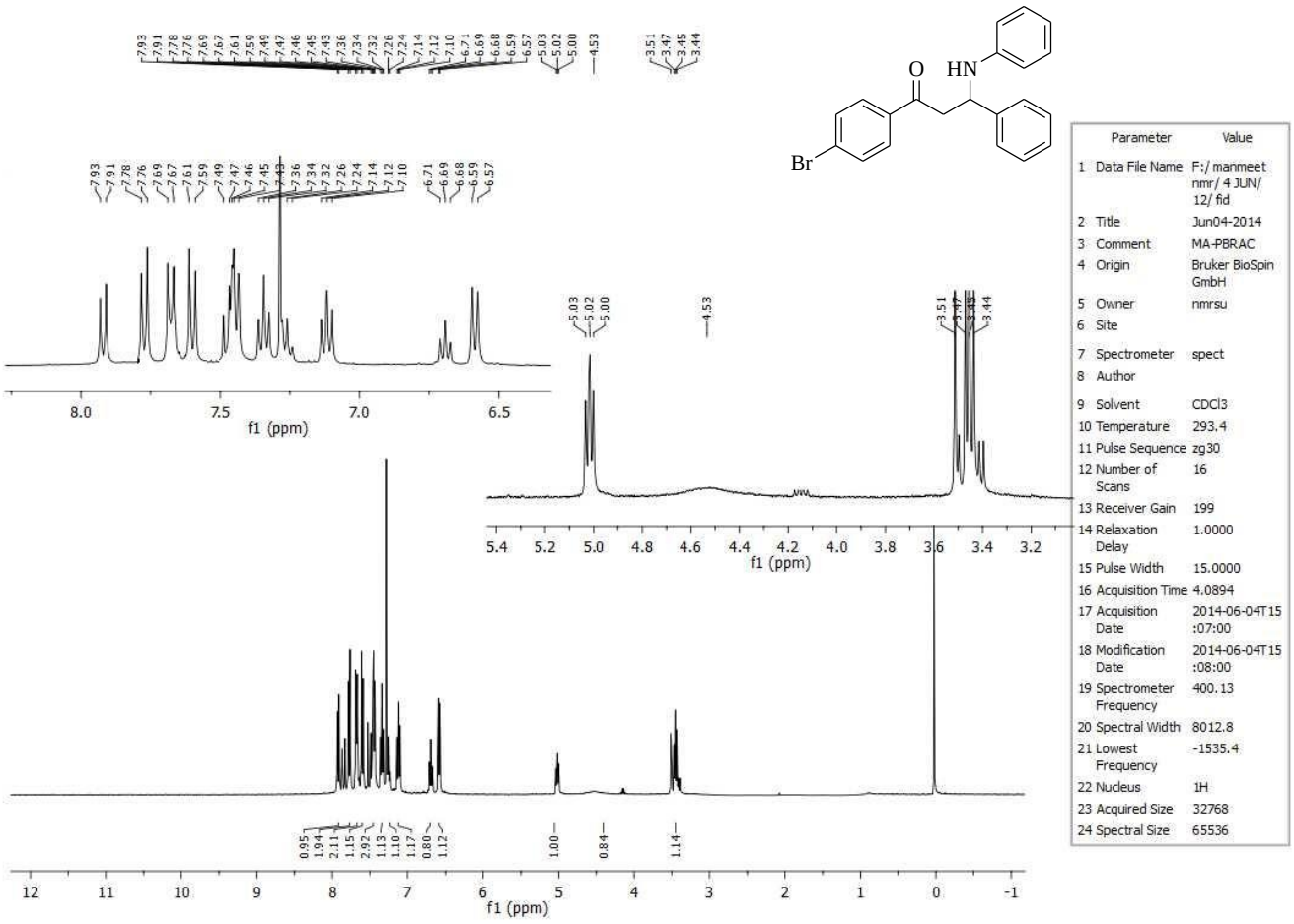
3	Comment	MA-PFAN
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.9
11	Pulse Sequence	deptsp135
12	Number of Scans	207
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	2.0316
17	Acquisition Date	2014-09-22T14:39:00
18	Modification Date	2014-09-22T14:39:14
19	Spectrometer Frequency	100.61
20	Spectral Width	16129.0
21	Lowest Frequency	-16.4
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

^{1}H spectra of 12n



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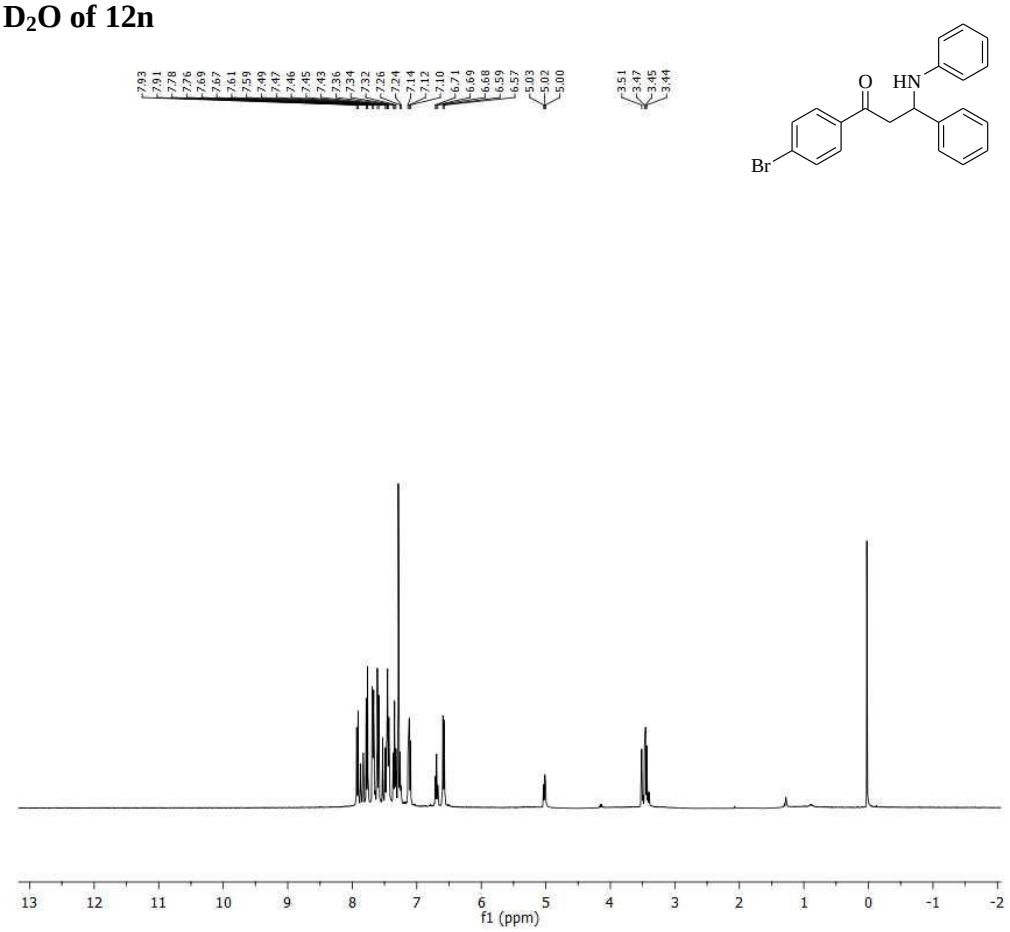
1	Data File Name	F:/ manmeet nmr/ 4 JUN/ 12/ fid
2	Title	Jun04-2014
3	Comment	MA-PBRAC
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	293.4
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	199
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-06-04T15 :07:00
18	Modification Date	2014-06-04T15 :08:00
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

D_2O of 12n



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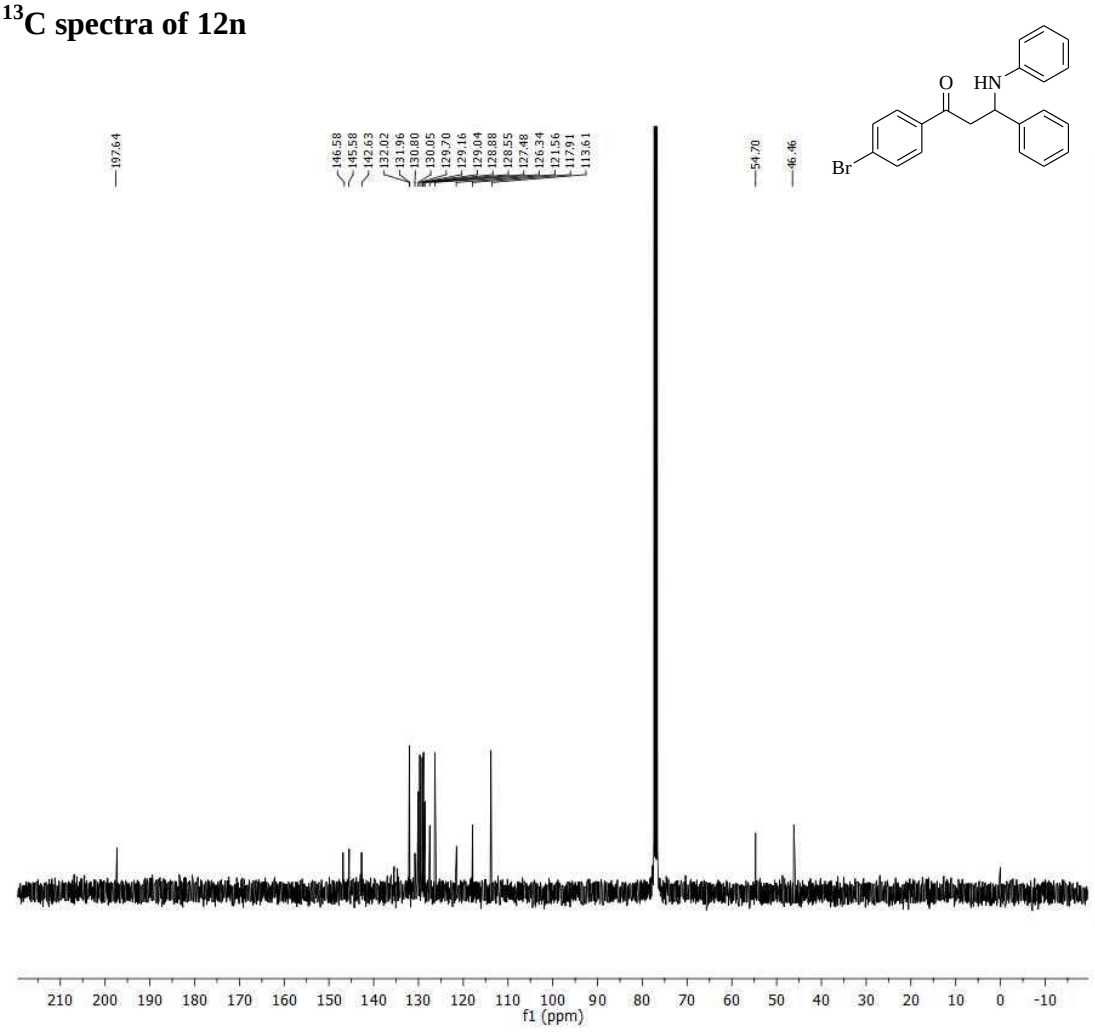
3	Comment	MA-PBRAC/ D2O
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.3
11	Pulse Sequence	zg30
12	Number of Scans	16
13	Receiver Gain	136
14	Relaxation Delay	1.0000
15	Pulse Width	15.0000
16	Acquisition Time	4.0894
17	Acquisition Date	2014-09-24T11:06:00
18	Modification Date	2014-09-24T11:06:16
19	Spectrometer Frequency	400.13
20	Spectral Width	8012.8
21	Lowest Frequency	-1535.4
22	Nucleus	1H
23	Acquired Size	32768
24	Spectral Size	65536

^{13}C spectra of 12n



	Parameter	Value
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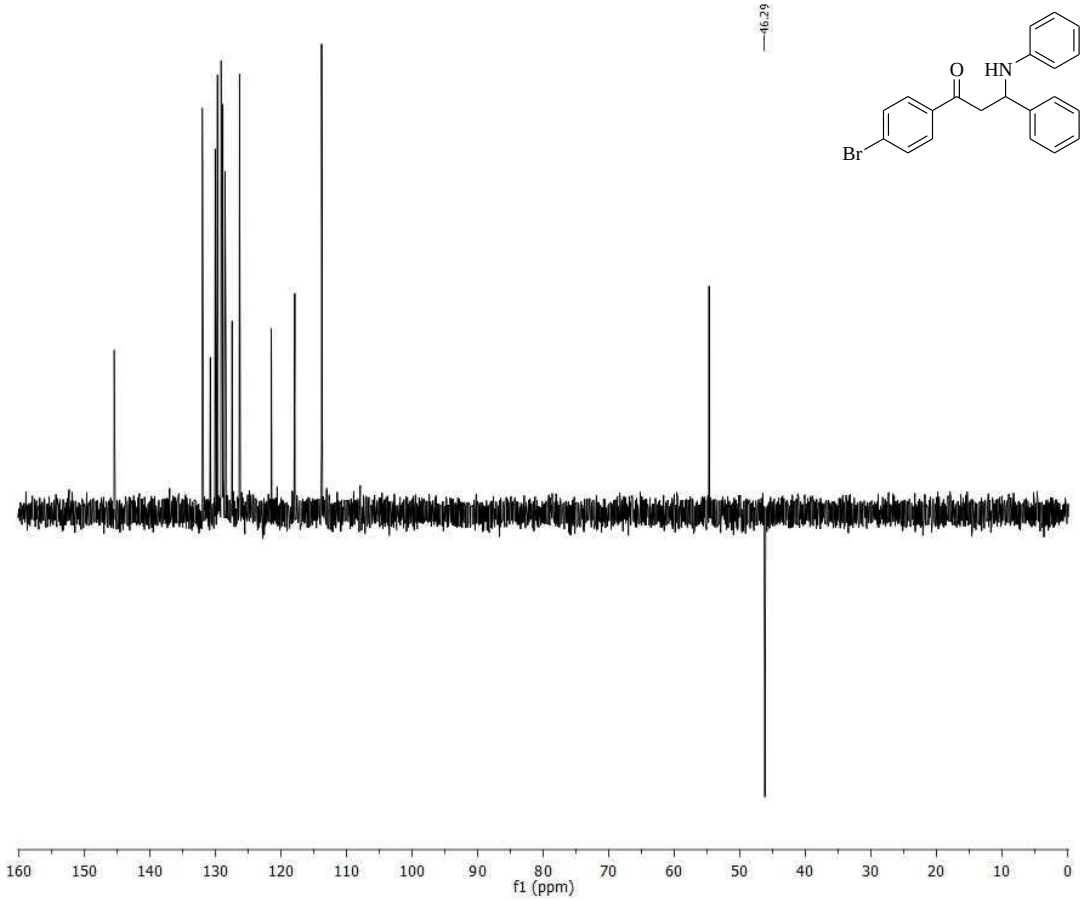
2	Title	Jun04-2014
3	Comment	MA-PBRAC
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.8
11	Pulse Sequence	zgpg30
12	Number of Scans	491
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	1.3631
17	Acquisition Date	2014-06-04T15:36:00
18	Modification Date	2014-06-04T15:36:46
19	Spectrometer Frequency	100.61
20	Spectral Width	24038.5
21	Lowest Frequency	-1958.9
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

Dept of 12n



	Parameter	Value
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5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	295.2
11	Pulse Sequence	deptsp135
12	Number of Scans	204
13	Receiver Gain	199
14	Relaxation Delay	2.0000
15	Pulse Width	9.8000
16	Acquisition Time	2.0316
17	Acquisition Date	2014-09-23T15: 09:17
18	Modification Date	2014-09-23T15: 09:30
19	Spectrometer Frequency	100.61
20	Spectral Width	16129.0
21	Lowest Frequency	-16.4
22	Nucleus	13C
23	Acquired Size	32768
24	Spectral Size	65536

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