Supporting Information

Solvent- and catalyst-free synthesis of 2,3-dihydro-1*H*-benzo[*d*]imidazoles

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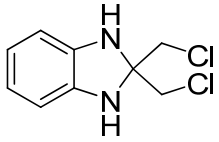
I. General information and experimental procedures

Solvents were dried by the standard procedures. ^{1}H and ^{13}C NMR spectra were determined in $CDCl_3$ or DMSO-d_6 on a Varian-Inova 400MHz spectrometer and chemical shifts were reported in ppm from internal TMS (δ). High resolution mass spectra were recorded on a MicroMass TOF mass spectrometer (EI). Column chromatography was performed with 200-300 mesh silica gel using flash column techniques. All of the reagents were used directly as obtained commercially unless otherwise noted.

Phenylenediamine (1 mmol) and 1,3-dichloroacetone (2 mmol) were conducted into a mortar and ground at 20 ℃ until the end of the completion (monitored by TLC). For some *o*-phenylenediamines with electron-withdrawing groups, the reactants need to be put into a tube and heated at 60 ℃ in an oil bath. After diluted with a bit of acetone, the residue was purified directly by column chromatography to afford the desired compounds **3**.

II. Compound analytical data

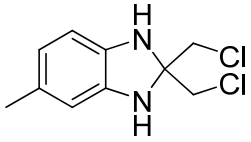
2,2-Bis(chloromethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (3a)



3a

Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.79 (s, 4H, $2CH_2$), 4.38 (s, br s, 2H, NH), 6.56-6.70 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.9, 82.1, 110.1, 121.2, 138.3; HRMS: calcd for $C_9H_{10}Cl_2N_2$, 216.0221 [M^+], found 216.0093.

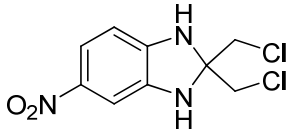
2,2-Bis(chloromethyl)-5-methyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (3b)



3b

Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.22 (s, 3H, CH_3), 3.80 (s, 4H, $2CH_2$), 4.30 (s, 2H, 2NH), 6.44 (s, 1H, ArH), 6.50 (s, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.6, 46.9, 82.2, 110.2, 111.2, 121.2, 131.1, 135.9, 138.7; HRMS: calcd for $C_{10}H_{12}Cl_2N_2$, 230.0378 [M^+], found 230.0378.

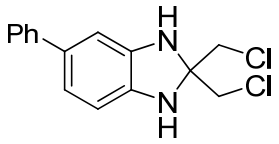
2,2-Bis(chloromethyl)-5-nitro-2,3-dihydro-1*H*-benzo[*d*]imidazole (3c)



3c

Colorless solid, m.p. 136-138 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.81 (s, 4H, $2CH_2$), 4.69-5.09 (br s, 2H, 2NH), 6.46 (d, 1H, J = 8.4 Hz, ArH), 7.33 (d, 1H, J = 2.4 Hz, ArH), 7.70-7.73 (m, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.78, 83.53, 103.9, 106.1, 120.0, 138.3, 141.5, 144.6; HRMS: calcd for $C_9H_9Cl_2N_3O_2$, 261.0072 [M^+], found 261.0064; Elemental analysis (%) calcd for $C_9H_9Cl_2N_3O_2$: C, 41.24; H, 3.46; N, 16.03; found: C, 41.06; H, 3.48; N, 15.85.

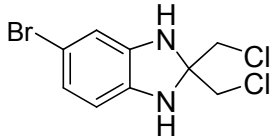
2,2-Bis(chloromethyl)-5-phenyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (3d)



3d

Colorless solid, m.p. 105-107 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.76 (s, 4H, $2CH_2$), 4.39 (s, 2H, 2NH), 6.56 (d, 1H, J = 10.4 Hz, ArH), 6.74 (s, 1H, ArH), 6.90 (d, 1H, J = 10.0 Hz, ArH), 7.17-7.47 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 44.4, 80.0, 106.3, 107.4, 117.6, 124.4, 124.5, 126.6, 132.1, 135.3, 136.4, 139.3; HRMS: calcd for $C_{15}H_{14}Cl_2N_2$, 292.0534 [M^+], found 292.0523.

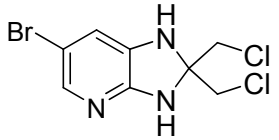
5-Bromo-2,2-bis(chloromethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (3e)



3e

Colorless solid, m.p. 107-109 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 4H, $2CH_2$), 4.46 (s, 2H, 2NH), 6.41 (d, 1H, J = 8.4 Hz, ArH), 6.66-6.79 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.8, 82.7, 110.8, 112.8, 113.6, 123.4, 137.5, 139.9; HRMS: calcd for $C_9H_9BrCl_2N_2$, 293.9326 [M^+], found 293.9283.

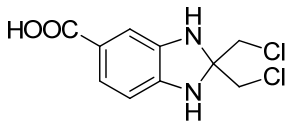
6-Bromo-2,2-bis(chloromethyl)-2,3-dihydro-1*H*-imidazo[4,5-*b*]pyridine (3f)



3f

Colorless solid, m.p. 90-92 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.72 (s, 4H, $2CH_2$), 6.42 (s, 1H, NH), 7.49-7.66 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 39.7, 106.6, 109.1, 112.5, 115.9, 136.4, 138.3, 142.0; HRMS: calcd for $C_8H_8BrCl_2N_3$, 294.9279 [M^+], 260.9491 [M^+-Cl+1], found 260.9489 [M^+-Cl+1].

2,2-Bis(chloromethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole-5-carboxylic acid (3g)

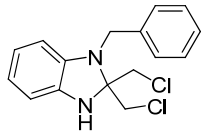


3g

Colorless solid, m.p. 142-144 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.76 (s, 4H, $2CH_2$), 6.32-7.18 (m, 3H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 48.2, 82.6, 104.4, 106.3, 119.7, 122.7, 139.2, 144.1, 167.9; HRMS: calcd for $C_{10}H_{10}Cl_2N_2O_2$, 260.0119 [M^+], found

260.0115.

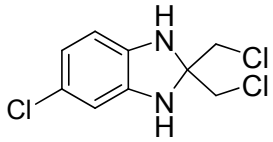
1-Benzyl-2,2-bis(chloromethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (3h)



3h

Colorless solid, m.p. 96-98 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.80-3.84 (AB coupling, J_1 = 11.7 Hz, J_2 = 8.0 Hz, 4H, $2CH_2$), 4.47 (s, H, NH), 4.52 (s, 2H, CH_2), 6.09-6.11 (m, 1H, ArH), 6.58-6.59 (m, 1H, ArH), 7.23-7.38 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.7, 48.1, 84.7, 105.5, 108.2, 118.7, 120.3, 126.6, 127.3, 128.7, 136.6, 138.3, 140.7; HRMS: calcd for $C_{16}H_{16}Cl_2N_2$, 306.0661 [M^+], found 306.0677.

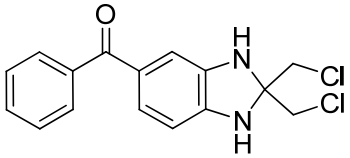
2,2-Bis(chloromethyl)- 5-chloro-2,3-dihydro-1*H*-benzo[*d*]imidazole (3i)



3i

Colorless solid, m.p. 50-52 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 4H, $2CH_2$), 4.42 (br s, 2H, 2NH), 6.44-6.64 (m, 3H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 46.7, 82.8, 110.0, 110.2, 120.3, 125.8, 136.69, 139.6; HRMS: calcd for $C_9H_9Cl_3N_2$, 249.9831 [M^+], found 249.98.

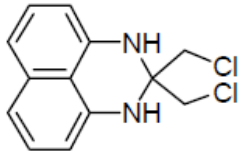
(2,2-Bis(chloromethyl)-5-benzoyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (3j)



3j

Colorless solid, m.p. 156-158 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.83 (s, 4H, $2CH_2$), 4.55-5.00 (br s, 2H, 2NH), 6.51(d, 1H, J = 7.6 Hz, ArH), 7.21(d, 1H, J = 10.4 Hz, ArH) 7.13 (s, 1H, ArH), 7.43-7.73 (m, 5H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 48.2, 82.5, 103.5, 103.9, 105.5, 125.6, 126.6, 127.9, 128.6, 130.7, 139.3, 139.5, 144.8, 193.8; HRMS: calcd for $C_{16}H_{14}Cl_2N_2O$, 320.0483, found 320.0468.

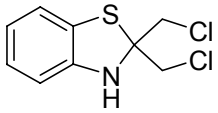
2,2-Bis(chloromethyl)-2,3-dihydro-1H-perimidine(3k)



3k

Colorless solid, m.p. 126-128 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.79(s, 4H, $2CH_2$), 4.73(br s, 2H, 2NH), 6.55(d, 2H, J_2 = 7.1Hz, ArH), 7.28-7.29(m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 107.1, 112.8, 118.7, 127.7, 134.8, 137.7. HRMS: calcd for $C_{13}H_{12}Cl_2N_2$ 266.0378[M^+], found 266.0367.

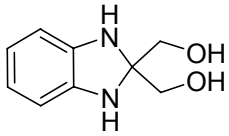
2,2-Bis(chloromethyl)-2,3-dihydrobenzo[d]thiazole(3l)



3l

Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.03 (br s, 1H, NH), 4.34(s, 4H, $2CH_2$), 7.35-6.64(m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): 46.1, 79.3, 110.5, 120.9, 122.2, 123.8, 126.0, 144.8. HRMS: calcd for $C_9H_9Cl_2NS$ 232.9833 [M^+], found 232.9836.

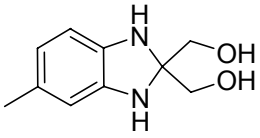
2,2-Bis(hydroxymethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (4a)



4a

Colorless solid, m.p. 73-75 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.40 (d, 4H, J = 5.1Hz, $2CH_2$), 4.56 (t, 2H, J = 5.2Hz, 2OH), 5.28 (br s, 2H, 2NH), 6.28-6.37 (m, 4H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 63.6, 82.9, 107.1, 117.8, 140.7; HRMS: calcd for $C_9H_{12}N_2O_2$, 180.0899 [M^+], found 180.0891.

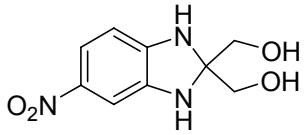
2,2-Bis(hydroxymethyl)-5-methyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (4b)



4b

Colorless solid, m.p. 74-76 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.07 (s, 3H, CH_3), 3.39 (s, 4H, $2CH_2$), 4.60 (s, 2H, 2OH), 5.24 (br s, 2H, 2NH) 6.14-6.18 (m, 3H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 20.8, 63.6, 83.0, 107.3, 108.5, 117.7, 126.6, 138.3, 141.2; HRMS: calcd for $C_{10}H_{14}N_2O_2$ 194.1055 [M^+], found 194.1055.

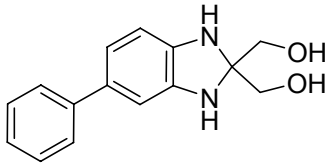
2,2-Bis(hydroxymethyl)-5-nitro-2,3-dihydro-1*H*-benzo[*d*]imidazole (4c)



4c

Colorless solid, m.p. 132-133 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.43 (d, J = 4.0 Hz, 4H, 2CH_2), 4.91 (br s, 2H, 2OH), 6.29 (s, 1H, NH), 6.85 (s, 1H, NH), 6.15-7.53 (m, 3H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 64.1, 85.3, 98.6, 101.8, 119.0, 137.4, 141.2, 148.9; HRMS: calcd for $C_9H_{11}N_3O_4$ 225.0750 [M^+], found 223.3504[M^+-2].

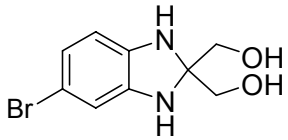
2,2-Bis(hydroxymethyl)-5-phenyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (4d)



4d

Colorless solid, m.p. 123-126 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 3.42 (s, 4H, 2CH_2), 4.74 (br, 2H, 2OH), 5.56-5.63 (br s, 2H, 2NH), 6.33 (d, 1H, J_1 = 8.0Hz, ArH), 6.56 (d, 1H, ArH), 6.67 (dd, 1H, J_1 = 8.0 H_Z, J_2 = 1.6 Hz, ArH), 7.17-7.46 (m, 5H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 63.9, 83.3, 105.3, 106.8, 116.7, 125.5, 125.6, 128.4, 130.1, 140.8, 141.6, 141.6; HRMS: calcd for $C_{15}H_{16}N_2O_2$, 256.1212 [M^+], found 256.1212.

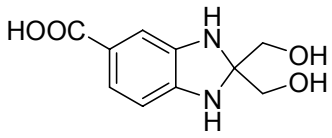
2,2-Bis(hydroxymethyl)-5-bromo-2,3-dihydro-1*H*-benzo[*d*]imidazole (4e)



4e

Colorless solid, m.p. 108-110 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.40 (s, 4H, 2CH_2), 4.46 (br s, 2H, 2OH), 5.56 (s, 1H, NH), 5.74 (s, 1H, NH), 6.12-6.44 (m, 3H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 63.8, 83.8, 107.4, 108.4, 108.8, 119.2, 140.3, 143.0; HRMS: calcd for $C_9H_{11}BrN_2O_2$ 258.0004 [M^+], found 257.9987.

2,2-Bis(hydroxymethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole-5-carboxylic acid(4g)

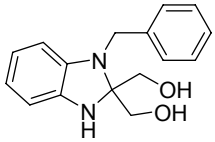


4g

Colorless solid, m.p. 161-163 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.42 (s, 4H, 2CH_2), 4.68 (br, 2H, 2OH), 5.60 (br s, 1H, NH), 6.29 (br s, 1H, NH), 6.21-7.13 (m, 3H, ArH), 11.76 (br, 1H, COOH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 63.9, 83.8, 104.1, 106.5, 118.9,

122.5, 140.4, 145.8, 167.8.

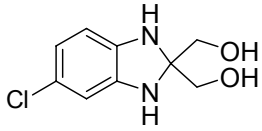
1-Benzyl-2,2-bis(hydroxymethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (4h)



4h

Colorless solid, m.p. 118-120 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.30 (br s, 3H, 2OH, NH), 3.61-3.70 (AB coupling, J_1 = 11.4 Hz, J_2 = 14.9 Hz, 4H, $2CH_2$), 4.34 (s, 2H, CH_2), 6.16-6.65 (m, 4H, ArH), 7.27-7.37 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 47.9, 63.3, 86.5, 106.0, 110.6, 118.7, 121.6, 127.2, 127.8, 129.2, 138.1, 139.3, 143.0; HRMS: calcd for $C_{16}H_{18}N_2O_2$, 270.1368 [M^+], found 270.1381.

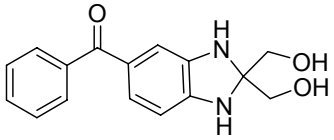
2,2-Bis(hydroxymethyl)- 5-chloro-2,3-dihydro-1*H*-benzo[*d*]imidazole (4i)



4i

Colorless solid, m.p. 72-74 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.38 (s, 4H, $2CH_2$), 4.75 (br s, 2H, 2OH), 5.60-5.61 (br s, 2H, 2NH), 6.16-6.30 (m, 3H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 63.7, 79.2, 84.1, 106.1, 106.7, 116.3, 121.1, 140.0, 142.8; HRMS: calcd for $C_9H_{11}ClN_2O_2$ 214.0509 [M^+], found 214.0808.

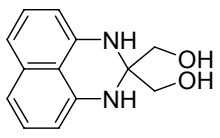
2,2-Bis(hydroxymethyl)-5-benzoyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (4j)



4j

Colorless solid, m.p. 132-135 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.45 (d, 4H, J = 5.3 Hz, $2CH_2$), 4.75 (br s, 2H, 2OH), 5.82 (s, 1H, NH), 6.22-6.87 (m, 3H, ArH), 6.74 (s, 1H, NH), 7.45-7.57 (m, 5H, ArH); ^{1}H NMR (400 MHz, DMSO-d_6, D_2O exchange): δ 3.43 (s, 4H, $2CH_2$), 6.21-6.86 (m, 3H, ArH), 7.46-7.55 (m, 5H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 64.0, 79.1, 84.0, 103.1, 105.5, 125.8, 125.9, 127.9, 128.5, 130.5, 139.7, 140.8, 146.7, 193.5; HRMS: calcd for $C_{16}H_{16}N_2O_3$, 284.1161, found 249.1003 [M^+-2OH-1H].

2,2-Bis(hydroxymethyl)-2,3-dihydro-1H-perimidine(4k)

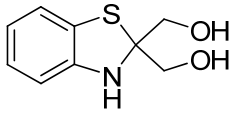


4k

Colorless solid, m.p. 156-158 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.46 (d, 4H, J = 5.3Hz,

$2CH_2$), 4.78 (br s, 2H, , 2NH), 6.08 (br s, 2H, 2OH), 6.48 (d, 2H, J = 7.3Hz, ArH), 6.89 (d, 2H, J = 8.0Hz, ArH), 7.10 (t, 2H, J = 7.7Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 62.3, 68.1, 104.2, 111.4, 114.4, 126.9, 134.0, 140.7. HRMS: calcd for $C_{13}H_{14}N_2O_2$ 230.1055[M^+], found 230.1060.

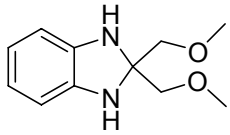
2,2-Bis(hydroxymethyl)-2,3-dihydrobenzo[d]thiazole(4l)



4l

Colorless solid, m.p. 108-110 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.68 (dd, 2H, J_1 = 5.4Hz, J_2 = 10.7Hz, CH_2), 3.59 (dd, 2H, J_1 = 5.9Hz, J_2 = 10.7Hz, CH_2) , 5.05 (t, 2H, J = 5.4Hz, 2OH), 6.15(s, 1H, NH), 6.52-6.50(m, 2H, ArH), 6.79 (t, 1H, J = 7.6Hz, ArH), 6.92 (d, 1H, J = 7.7Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): 40.1, 64.2, 82.2, 109.0, 118.0, 121.1, 124.7, 124.8, 147.3. HRMS: calcd for $C_9H_{11}NO_2S$ 197.0511[M^+], found 197.0526.

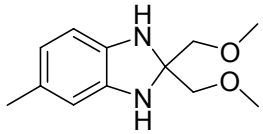
2,2-Bis(methoxymethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (5a)



5a

Colorless solid, m.p. 78-80 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.40 (s, 6H, $2CH_3$), 3.49 (s, 4H, $2CH_2$) 4.19 (br s, 2H, 2NH), 6.56-6.67 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 59.8, 75.0, 80.9, 110.8, 120.9, 140.3; HRMS: calcd for $C_{11}H_{16}N_2O_2$ 208.1212 [M^+], found 208.1221.

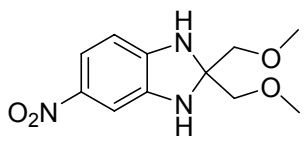
2,2-Bis(methoxymethyl)-5-methyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (5b)



5b

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.12 (s, 3H, CH_3), 3.39 (s, 6H, $2CH_3$), 3.47 (s, 2H, $2CH_2$), 5.24 (br s, 2H, 2NH), 6.43-6.51 (m, 3H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.5, 59.7, 75.0, 81.0, 110.8, 111.6, 120.8, 130.6, 137.8, 140.8; HRMS: calcd for $C_{12}H_{18}N_2O_2$ 222.1368 [M^+], found 222.1400.

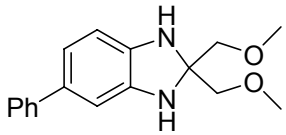
2,2-Bis(methoxymethyl)-5-nitro-2,3-dihydro-1*H*-benzo[*d*]imidazole (5c)



5c

Colorless solid, m.p. 128-130 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.58 (s, 6H, $2CH_3$), 4.85 (s, 2H, $2CH_2$), 8.19-9.02 (m, 3H, ArH), 9.15 (br s, 2H, 2NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.0, 59.9, 74.5, 123.5, 124.1, 12.3, 131.2, 144.6, 146.9; HRMS: calcd for $C_{11}H_{15}N_3O_4$ 253.1063 [M^+], found 189.0535 (M^+-$2OCH_3$-2H) .

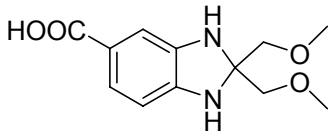
2,2-Bis(methoxymethyl)-5-phenyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (5d)



5d

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.42 (s, 6H, $2CH_3$), 3.52 (s, 4H, $2CH_2$), 4.28 (br s, 2H, 2NH), 6.22-6.91 (m, 3H, ArH), 7.24-7.51 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 59.9, 75.1, 81.3, 109.6, 110.6, 113.1, 120.0, 126.7, 127.1, 129.0, 134.4; HRMS: calcd for $C_{15}H_{16}N_2O_2$ 284.1525 [M^+], found 284.0046.

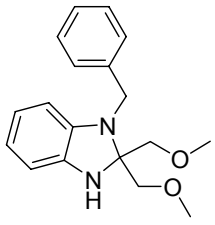
2,2-Bis(methoxymethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazol-5-carboxylic acid (5g)



5g

Colorless solid, m.p. 116-119 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.29 (s, 6H, $2CH_3$), 3.34 (s, 4H, $2CH_2$), 5.96 (br s, 1H, NH), 6.61 (br s, 1H, NH), 6.20-7.13 (m, 3H, ArH), 11.79 (br s, 1H, COOH); ^{1}H NMR (400 MHz, DMSO-d_6 + D_2O): δ 3.27 (s, 6H, $2CH_3$), 3.33 (s, 4H, $2CH_2$), 6.20-7.13 (m, 3H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 58.9, 75.2, 82.0, 104.1, 106.3, 119.0, 122.5, 140.1, 145.3, 167.9; HRMS: calcd for $C_{12}H_{16}N_2O_4$ 252.1110 [M^+], found 252.1117.

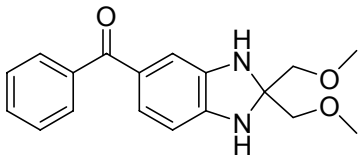
1-Benzyl-2,2-bis(methoxymethyl)-2,3-dihydro-1*H*-benzo[*d*]imidazole (5h)



5h

Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.33 (s, 6H, $2CH_3$), 3.54-3.68 (AB coupling, J_1 = 9.4 Hz, J_2 = 21.0 Hz, 4H, $2CH_2$), 4.33 (br s, H, NH), 4.46 (s, 2H, CH_2), 5.99-6.52 (m, 3H, ArH), 7.23-7.38 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 48.6, 59.7, 74.4, 105.3, 109.1, 113.1, 118.1, 120.5, 127.0 , 127.1, 128.8, 138.5; HRMS: calcd for: $C_{18}H_{22}O_2N_2$ 298.1681 [M^+], found 298.1696.

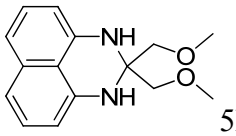
2,2-Bis(methoxymethyl)-5-benzoyl-2,3-dihydro-1*H*-benzo[*d*]imidazole (5j)



5j

Red oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.36 (s, 6H, $2CH_3$), 3.47 (s, 4H, $2CH_2$), 4.54 (br s, 1H, NH), 4.82 (br s, 1H, NH), 6.39-7.14 (m, 3H, ArH), 7.38-7.69 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 59.8, 75.0, 81.7, 106.7, 110.3, 127.4, 128.3, 129.4, 129.8, 131.5, 139.6, 140.0, 145.4, 195.9; HRMS: calcd for $C_{18}H_{20}N_2O_3$ 312.1474 [M^+], found 312.1469.

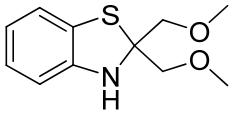
2,2-Bis(methoxymethyl)-2,3-dihydro-1H-perimidine(5k)



5k

Colorless solid, m.p. 114-116 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.40(s, 6H, $2CH_3$), 3.51(s, 4H, $2CH_2$), 4.78(br s, 2H, 2NH), 6.52 (d, 2H, J = 7.3Hz, ArH), 7.16-7.28 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): 59.8, 67.6, 73.5, 106.3, 113.0, 117.6, 127.5, 134.9, 139.4. HRMS: calcd for $C_{15}H_{18}N_2O_2$ 258.1368 [M^+], found 258.1371.

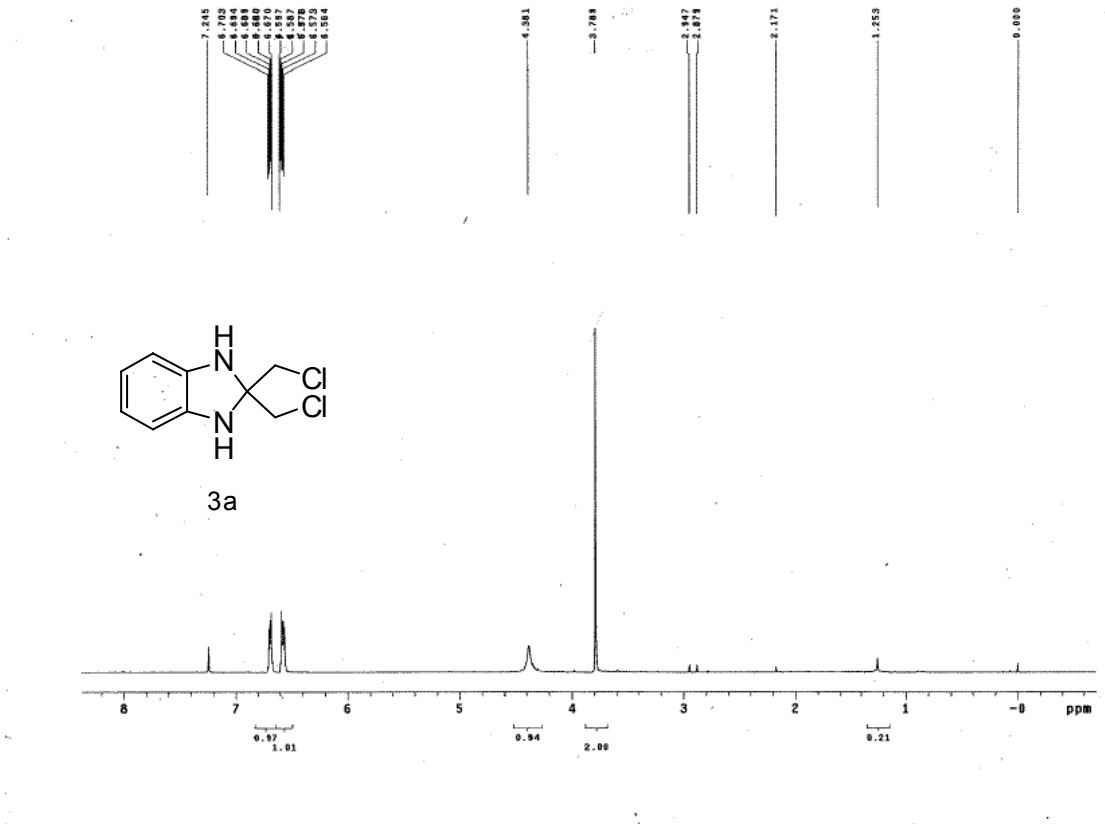
2,2-Bis(methoxymethyl)-2,3-dihydrobenzo[d]thiazole(5l)

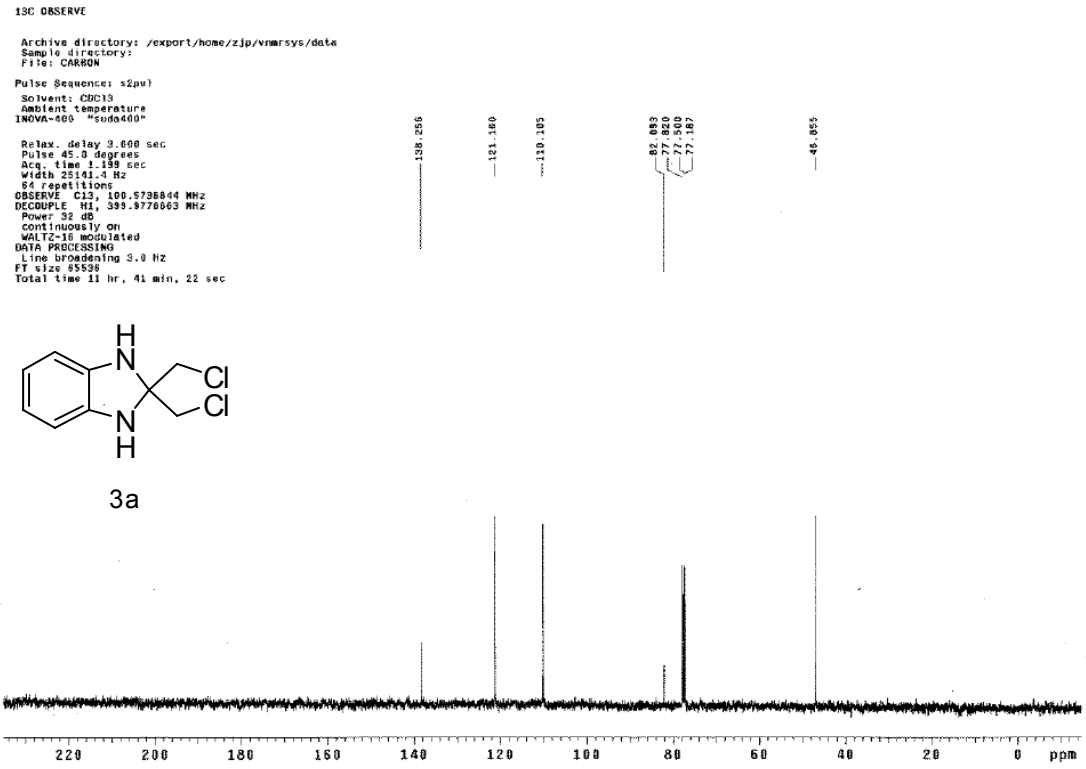


5l

Colorless solid, m.p. 74-76 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.41(s, 6H, $2CH_3$), 3.74-3.62(m, 4H, $2CH_2$), 4.51(br s, 1H, NH), 6.61 (d, 1H, J = 7.8Hz, ArH), 6.71 (t, 1H, J = 7.5Hz, ArH), 6.89 (t, 1H, J = 8.2Hz, ArH), 7.01 (d, 1H, J = 7.6Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): 59.9, 75.4, 78.5, 111.4, 121.0, 122.5, 125.8, 126.6, 146.3. HRMS: calcd for $C_{15}H_{17}NO_2S$ 225.0823[M^+], found 225.0819.

III. ^{1}H, ^{13}C NMR and HRMS spectra

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Sample directory:
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Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
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Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25141.4 Hz
64 repetitions
OBSERVE C13, 100.5736844 MHz
DECOUPLE H1, 399.9776063 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 11 hr, 41 min, 22 sec
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110.105
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77.820
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Cl
Cl
N
H
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40
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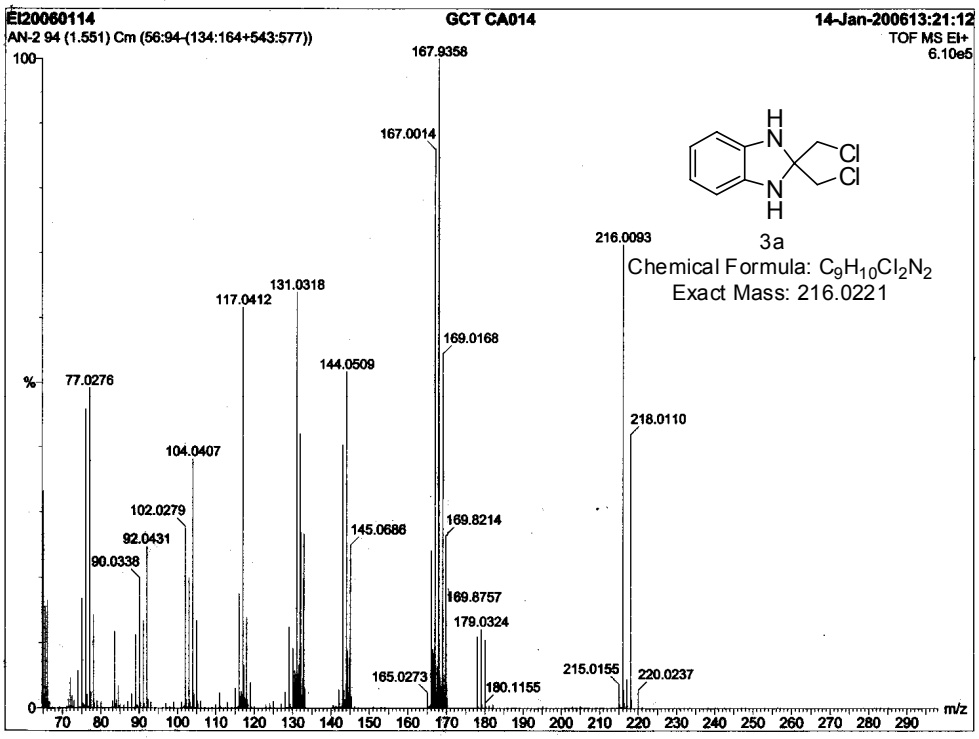
0
ppm


EI20060114
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14-Jan-200613:21:12
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%
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167.0014
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Cl
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H
3a
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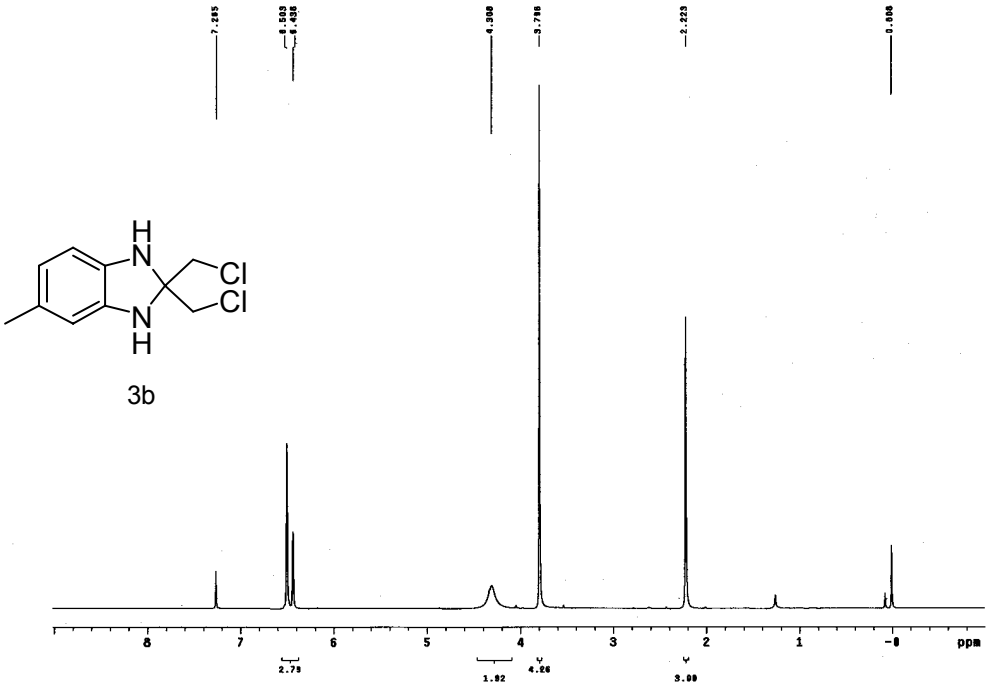
m/z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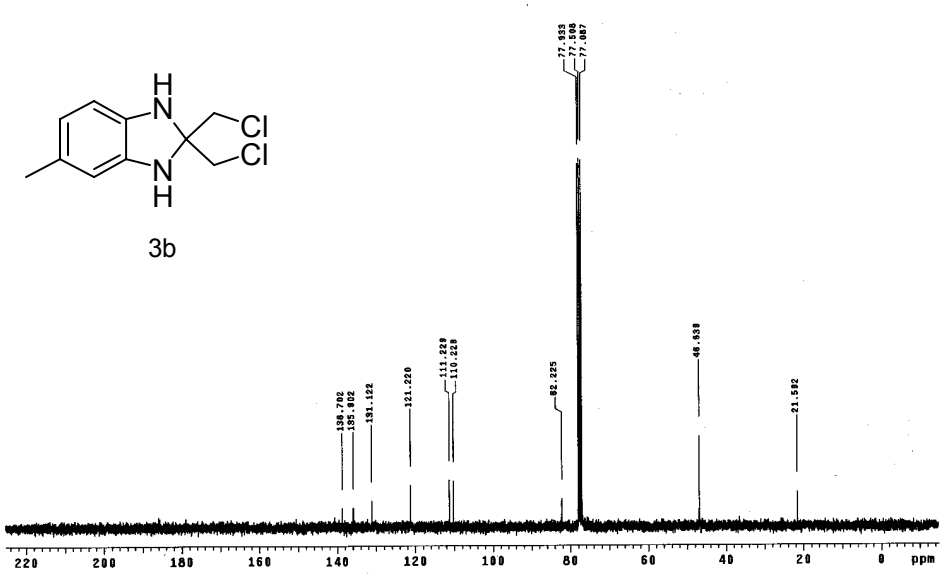
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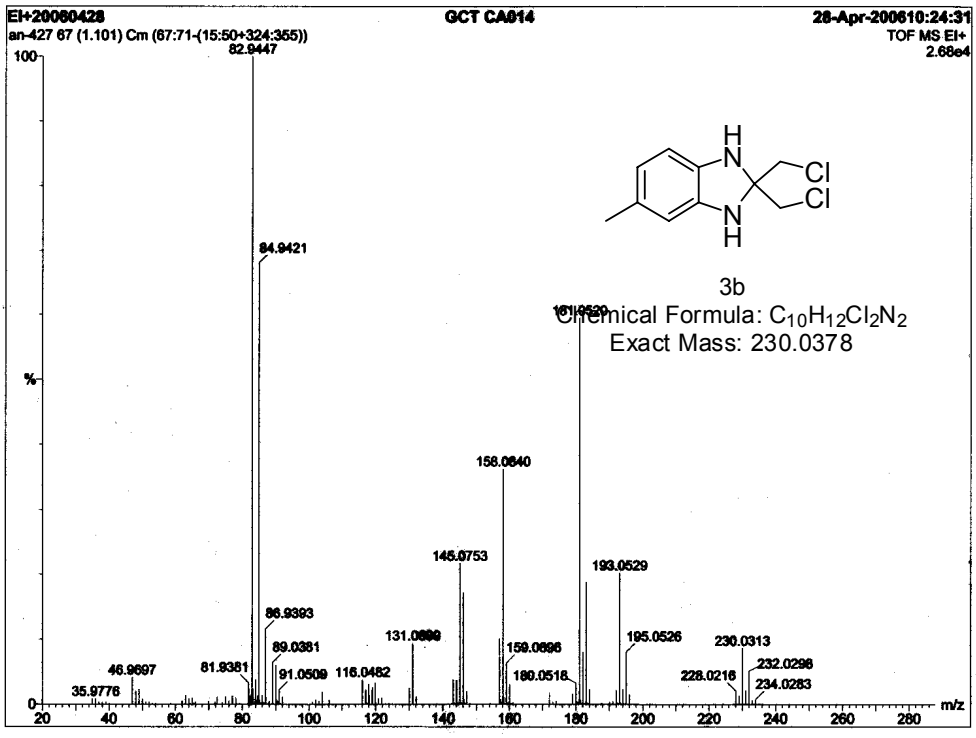
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28-Apr-200610:24:31
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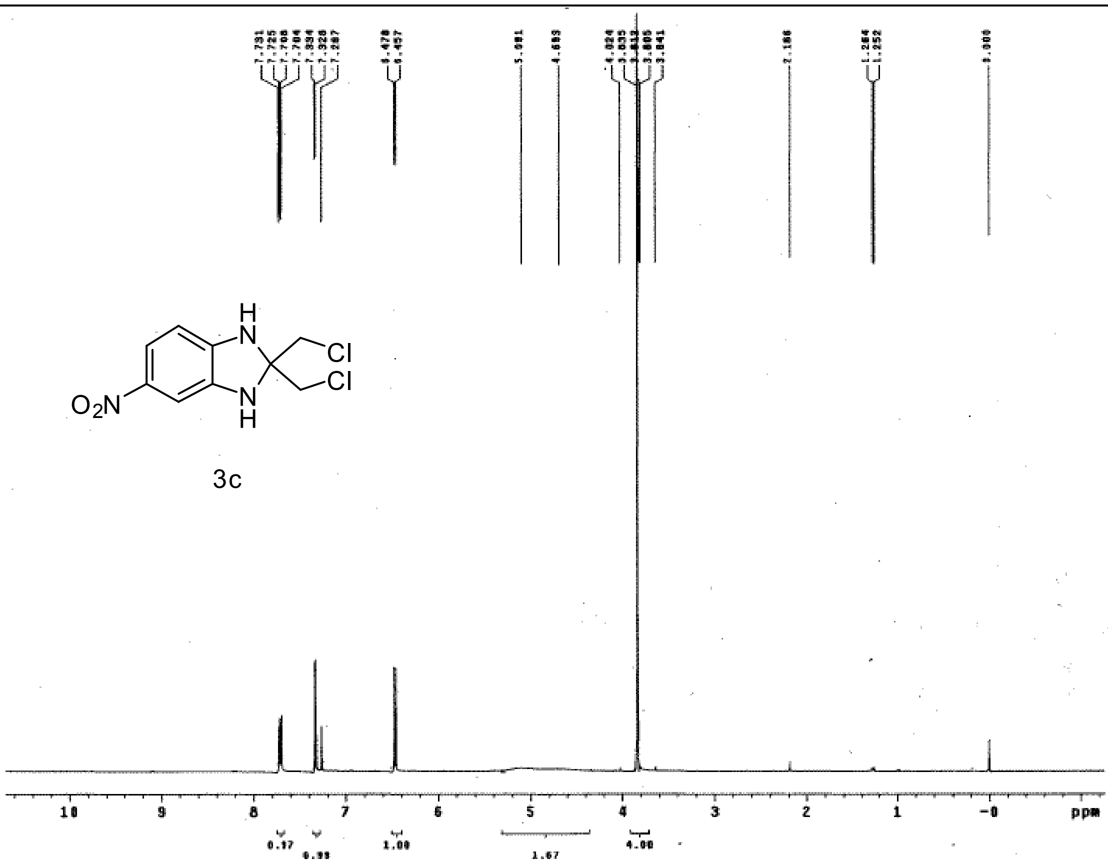
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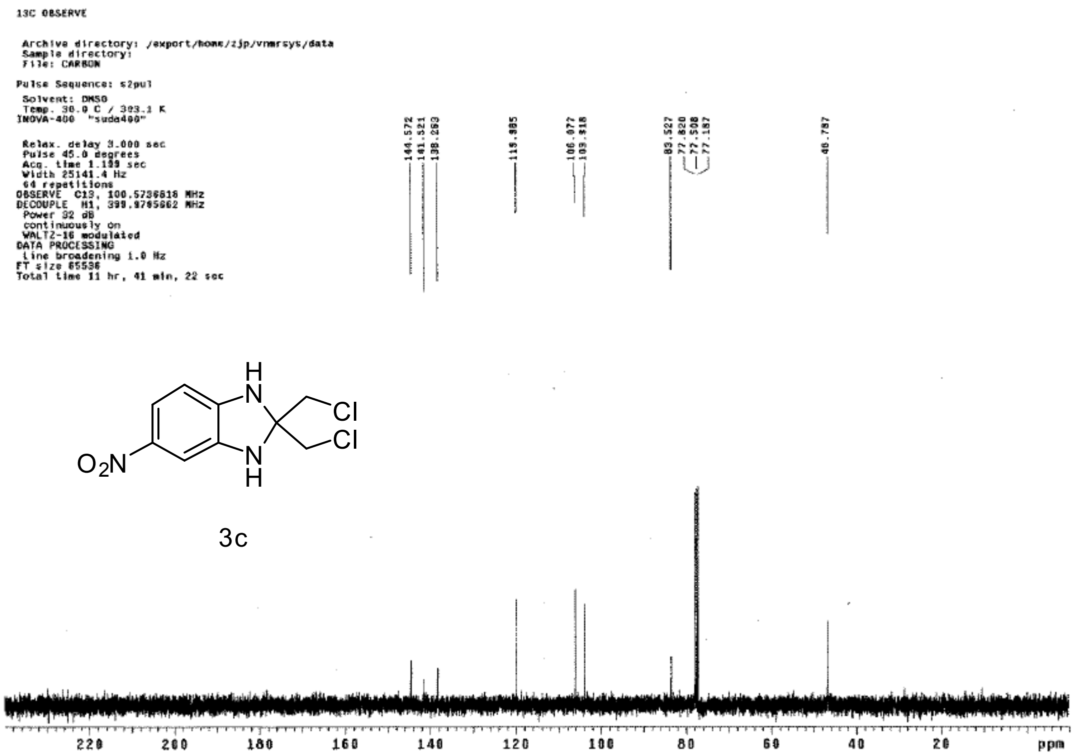
13C OBSERVE
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Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Temp. 30.0 C / 303.1 K
INOVA-400 "suda400"
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25141.4 Hz
64 repetitions
OBSERVE C13, 100.5736818 MHz
DECOUPLE H1, 399.9785862 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 41 min, 22 sec
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160
140
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80
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40
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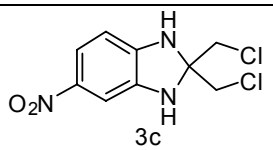
ppm



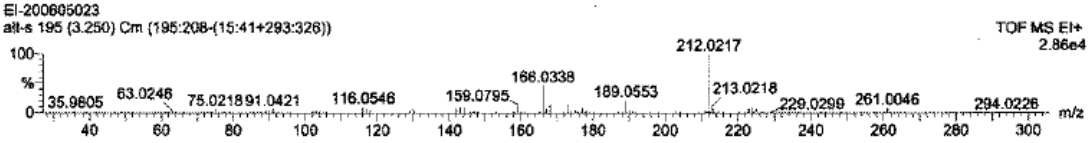
3c

Chemical Formula: $C_9H_9Cl_2N_3O_2$
Exact Mass: 261.0072

Multiple Mass Analysis: 21 mass(es) processed
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
796 formula(e) evaluated with 30 results within limits (up to 50 closest results for each mass)

EI-200605023
alt-s 195 (3.250) Cm (195:208-(15:41+293:326))



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		75.0195	2.3	31.3	1.5	2	C H3 N2 O2
91.0421	5.66	91.0422	-0.1	-1.1	5.0	1	C6 H5 N
		91.0382	3.9	43.1	1.0	2	C H5 N3 O2
116.0546	9.93	116.0586	-4.0	-34.3	2.0	2	C4 H8 N2 O2
		116.0500	4.6	39.4	6.5	1	C8 H6 N
117.0523	5.53	117.0538	-1.5	-13.0	2.0	1	C3 H7 N3 O2
130.0654	7.13	130.0657	-0.3	-2.1	6.5	2	C9 H8 N
		130.0617	3.7	28.8	2.5	1	C4 H8 N3 O2
142.0558	7.35	142.0531	2.7	19.0	8.0	1	C9 H6 N2
143.0619	9.95	143.0609	1.0	6.8	7.5	1	C9 H7 N2
144.0712	10.14	144.0687	2.5	17.0	7.0	1	C9 H8 N2
159.0795	14.57	159.0796	-0.1	-0.9	7.0	1	C9 H9 N3
166.0338	46.00	166.0323	1.5	9.0	2.0	2	C5 H9 N2 O2 37Cl
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		166.0383	-4.5	-27.3	1.5	1	C4 H9 N3 O2 35Cl
167.0295	6.05	167.0276	1.9	11.6	2.0	3	C4 H8 N3 O2 37Cl
		167.0316	-2.1	-12.4	6.0	2	C9 H8 N 37Cl
		167.0264	3.1	18.8	5.5	1	C9 H8 O 35Cl
		167.0250	4.5	26.8	6.0	4	C7 H6 N3 35Cl
168.0300	14.36	168.0329	-2.9	-17.0	5.5	3	C7 H7 N3 35Cl
		168.0268	3.2	18.9	6.0	2	C8 H7 N2 37Cl
		168.0342	-4.2	-25.0	5.0	1	C9 H9 O 35Cl
173.0611	15.54	173.0589	2.2	12.6	8.0	1	C9 H7 N3 O
177.0431	7.43	---					
189.0553	21.19	189.0538	1.5	7.8	8.0	1	C9 H7 N3 O2
212.0217	100.00	212.0227	-1.0	-4.6	6.5	1	C8 H7 N3 O2 35Cl
213.0218	12.15	213.0245	-2.7	-12.6	6.5	1	C9 H8 N2 O2 37Cl
223.0187	6.46	223.0149	3.8	17.2	8.0	1	C9 H6 N3 O2 35Cl
224.0256	10.03	224.0227	2.9	13.0	7.5	1	C9 H7 N3 O2 35Cl
225.0266	6.43	225.0305	-3.9	-17.4	7.0	1	C9 H8 N3 O2 35Cl
261.0046	7.68	---					

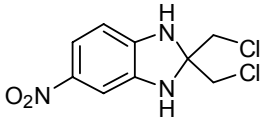
Eager 200 Summarize Results

Method Name : Test Eager 200 (Channel B)
Method Filename : CHN0511.MTH
Company name : SuzhouUniversity
Operator ID : WB

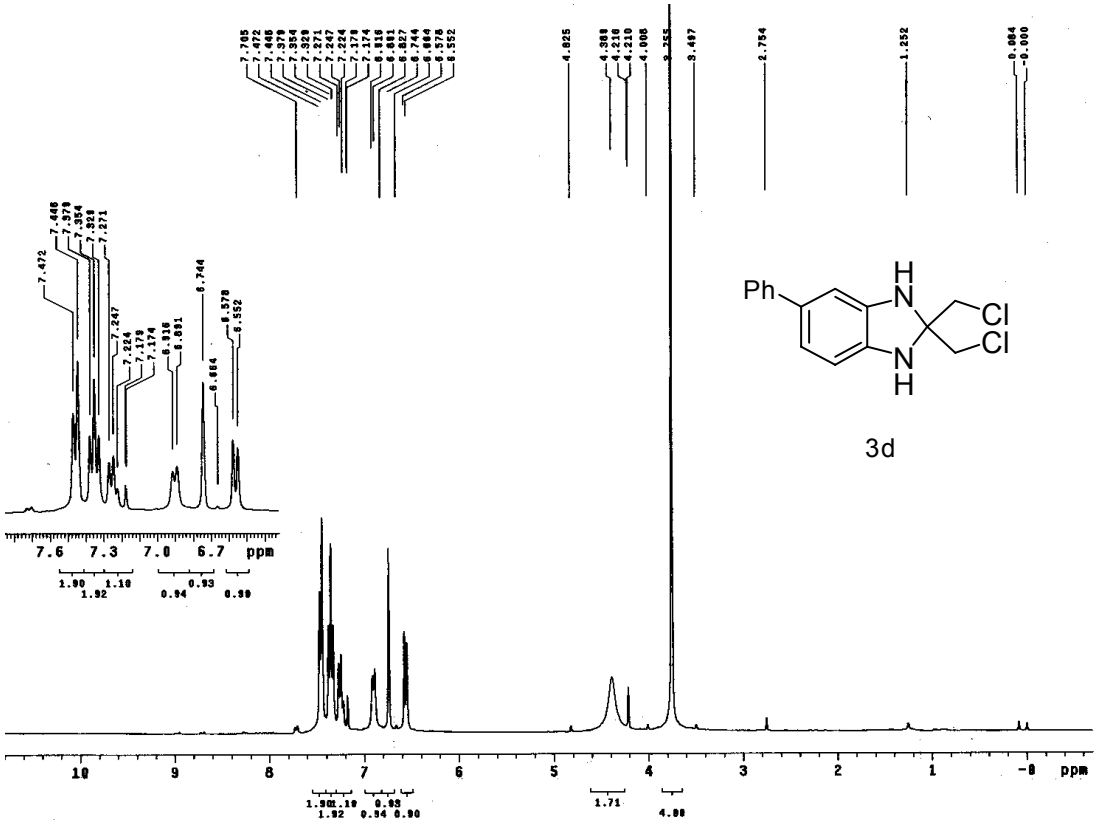
Group No : 1 Sample Name	Element % Nitrogen	Carbon	Hydrogen
an5-15	15.84960079	41.05747604	3.478757858

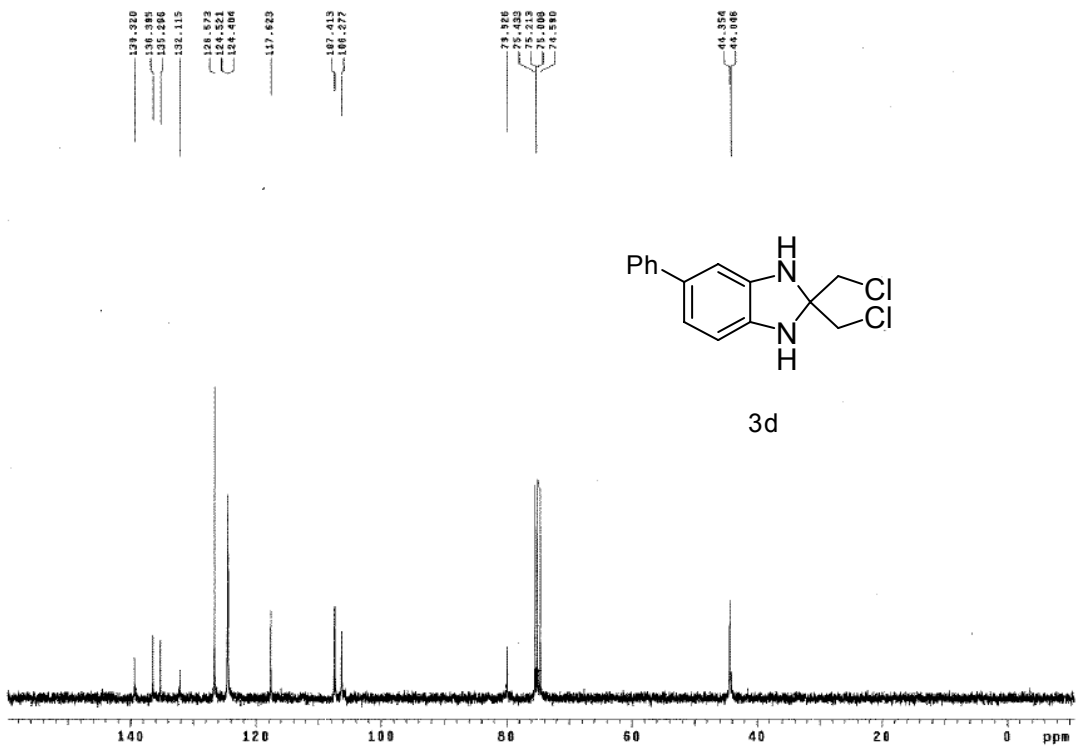
1 Sample(s) in Group No : 1

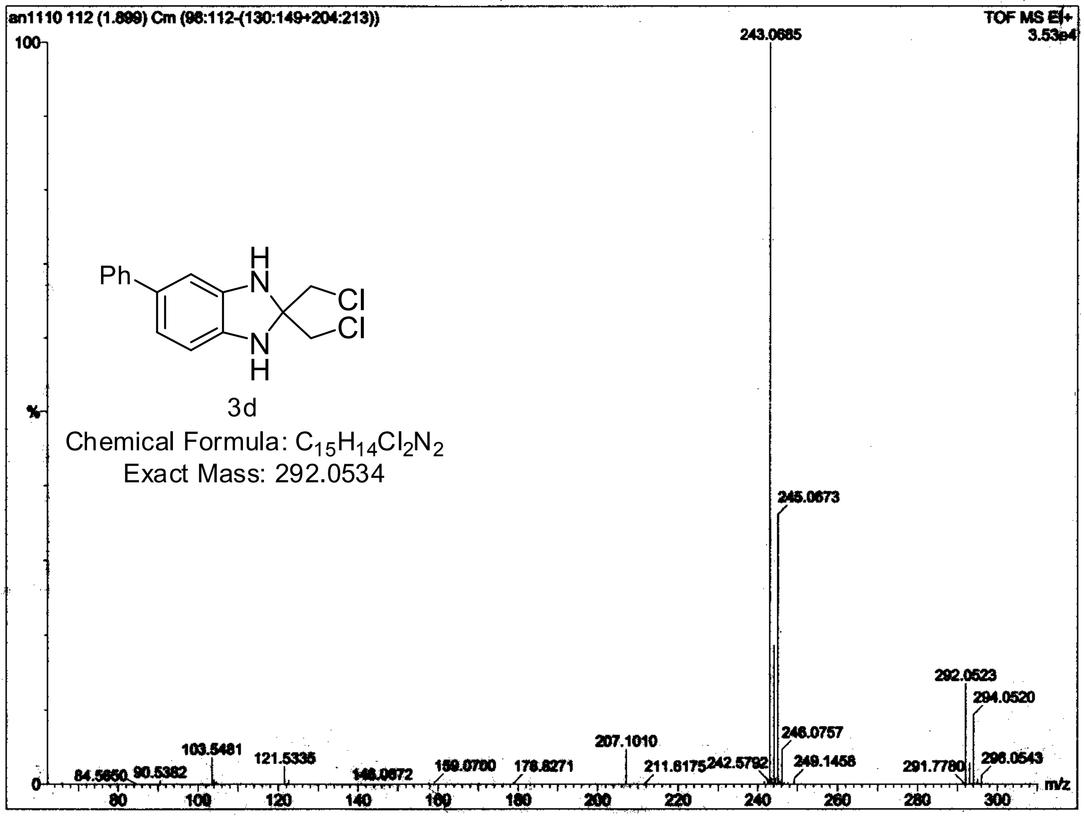
Component Name	Average	Std. Dev.
Nitrogen	15.8496	0.0000000
Carbon	41.05748	0.0000000
Hydrogen	3.478758	0.0000000

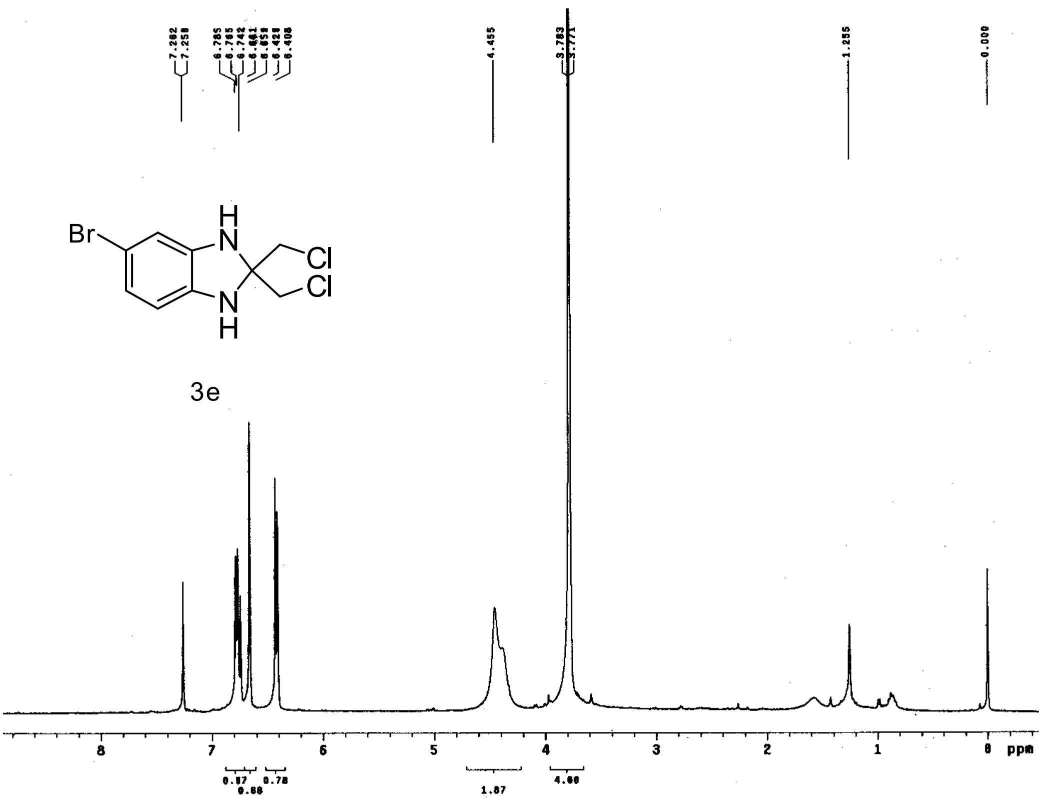


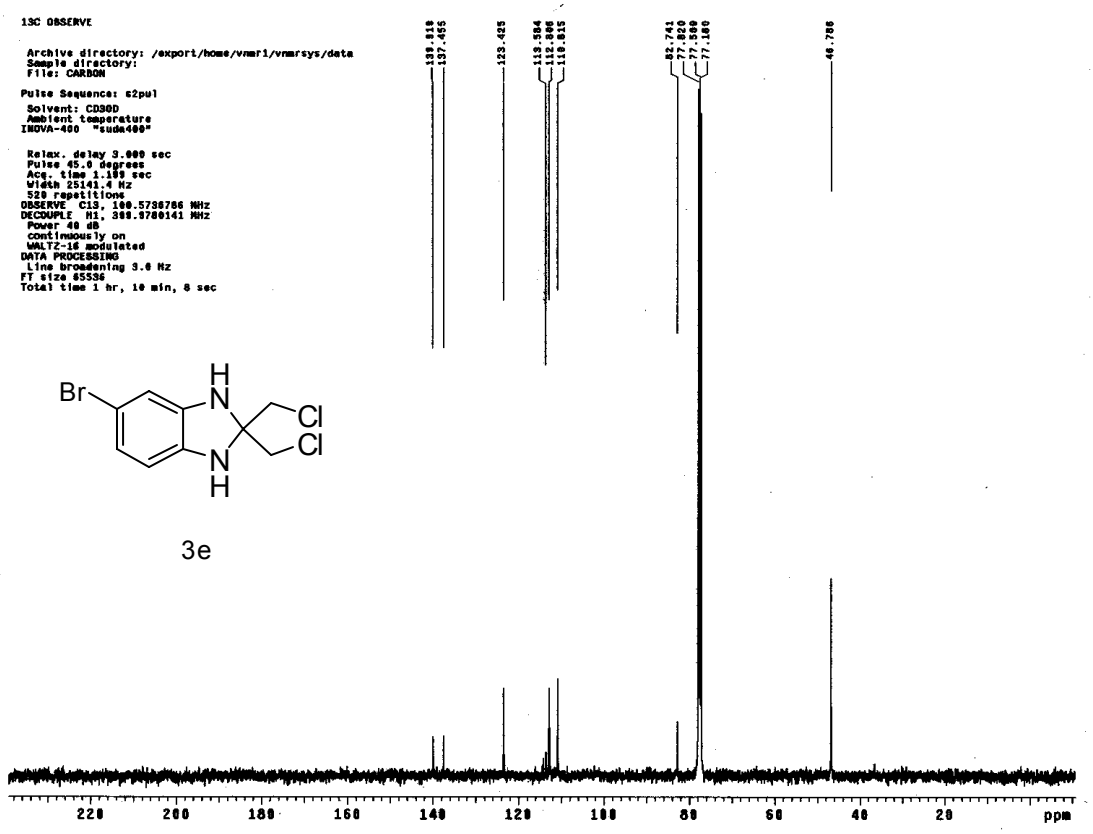
3c

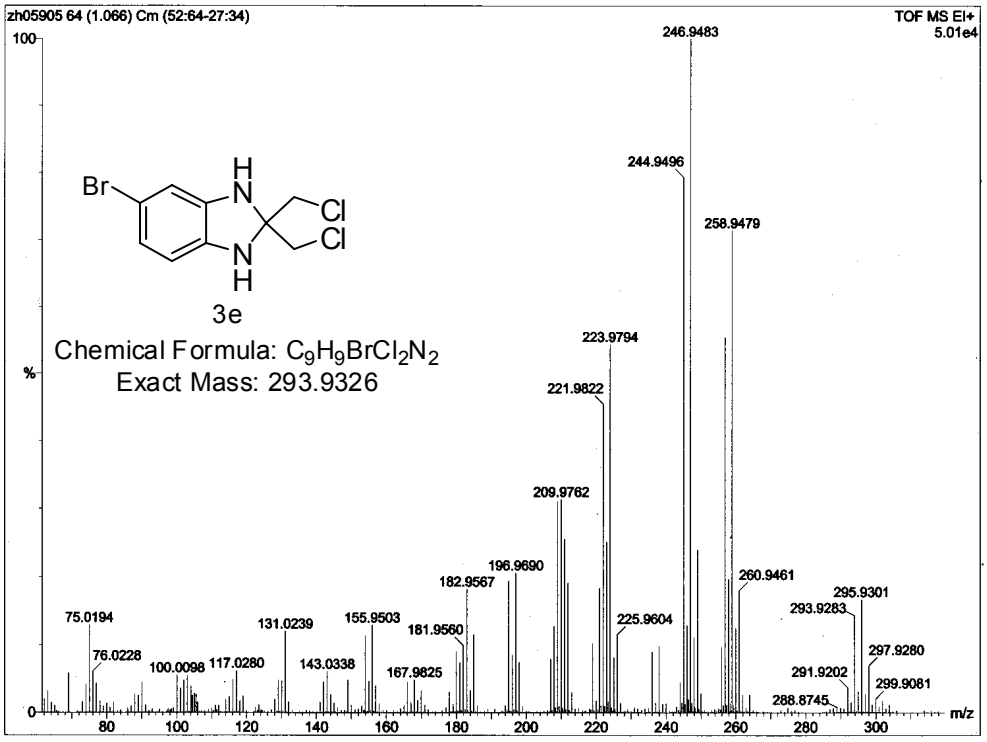

Ph
H
N
N
H
Cl
Cl
3d
ppm

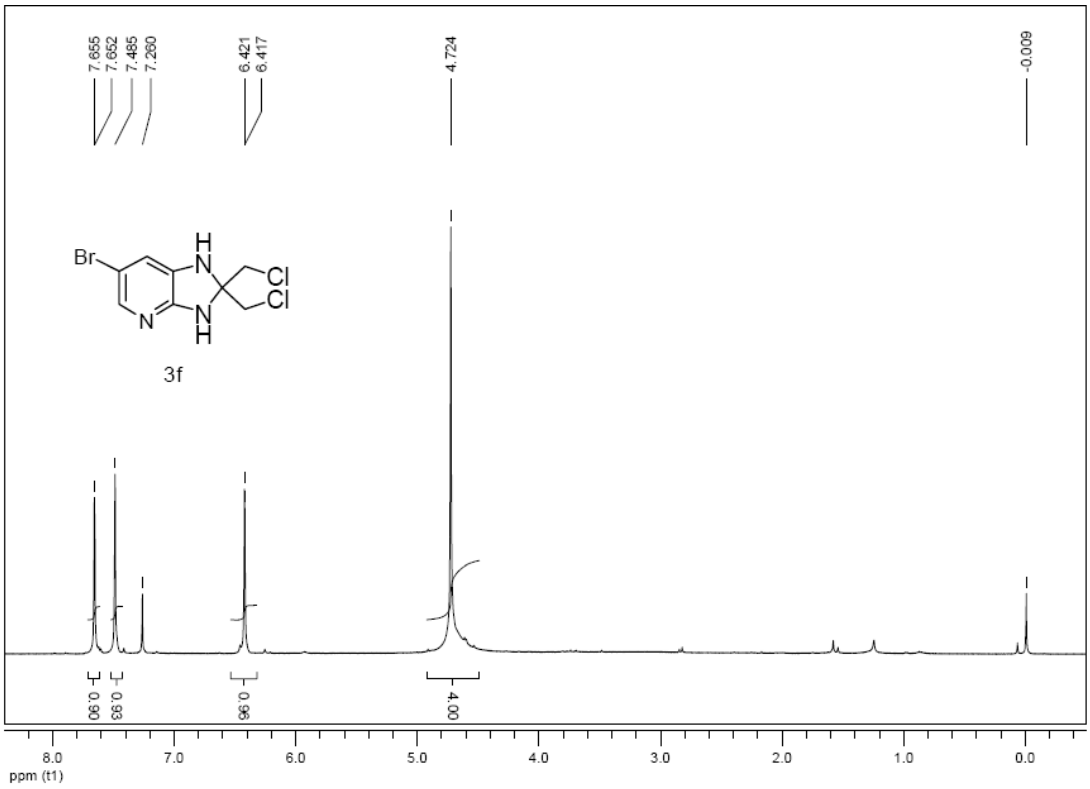

Ph
H
N
N
H
Cl
Cl
3d
ppm

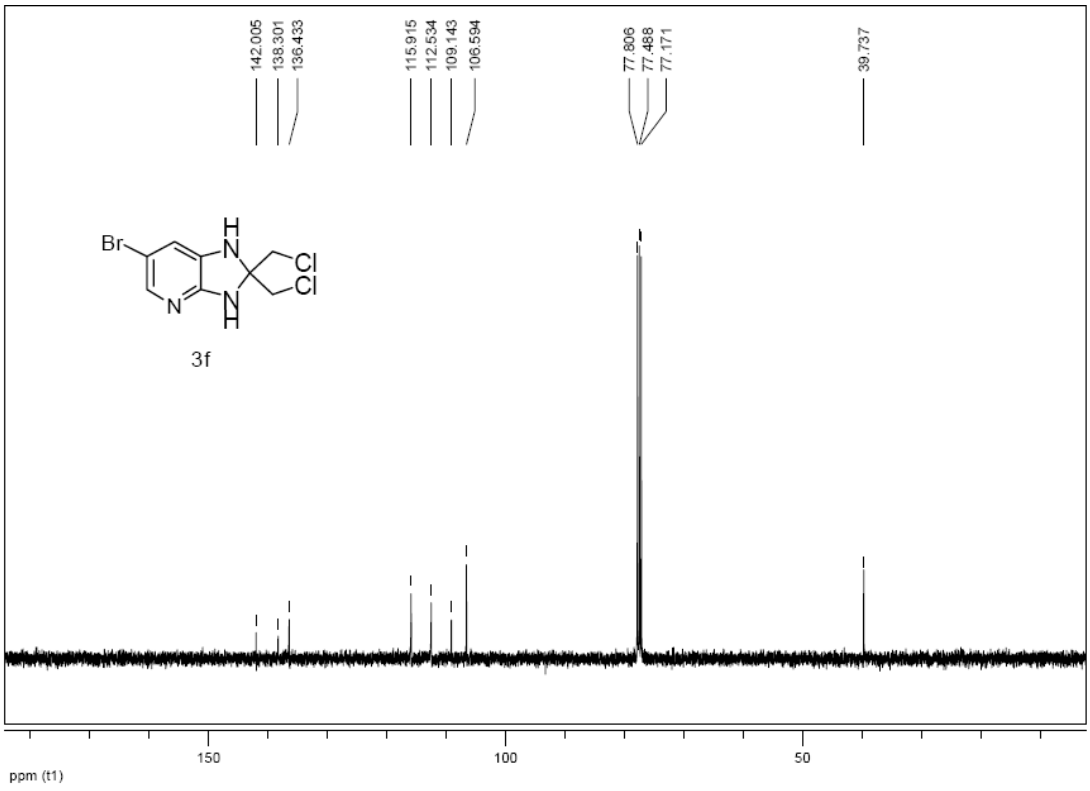

an1110 112 (1.899) Cm (98:112-(130:149+204:213))
TOF MS EI+
3.53e4
243.0685
245.0673
292.0523
294.0520
246.0757
207.1010
103.5481
121.5335
84.5650
90.5382
148.0672
159.0700
178.8271
211.6175
242.5792
249.1458
291.7780
296.0543
m/z
%
Ph
N
H
Cl
3d
Chemical Formula: $C_{15}H_{14}Cl_2N_2$
Exact Mass: 292.0534

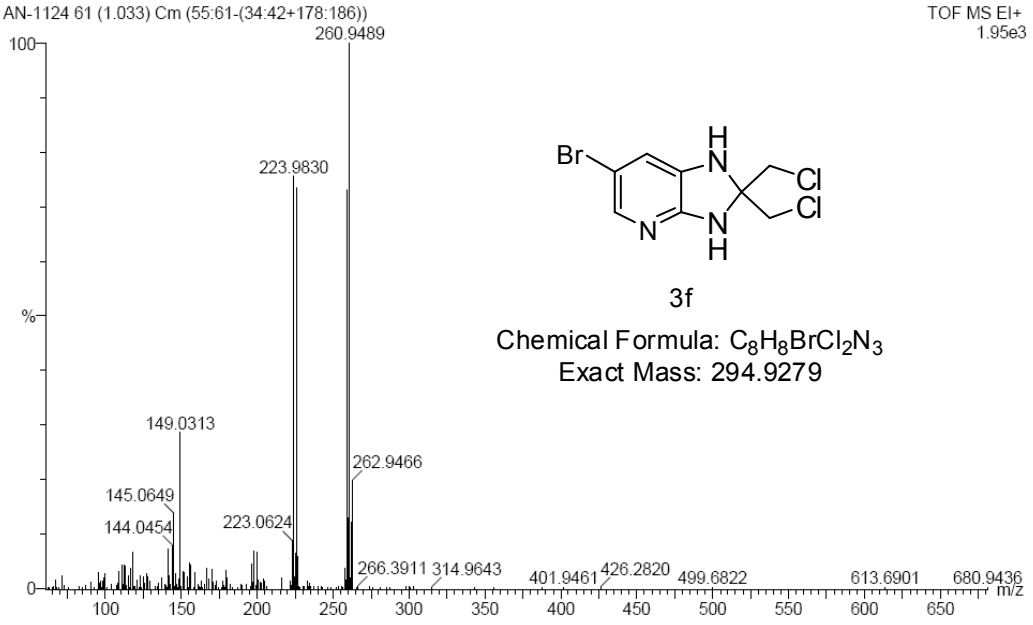

7.282
7.258
6.785
6.765
6.742
6.661
6.659
6.428
6.406
4.455
3.783
3.771
1.255
0.000
Br
N
H
Cl
3e
ppm
0.97
0.88
0.78
1.87
4.00


1SC OBSERVE
Archive directory: /export/home/vmsr1/vmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CD3OD
Ambient temperature
INOVA-400 "suda400"
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.189 sec
Width 25141.4 Hz
520 repetitions
OBSERVE C13, 100.5736786 MHz
DECOUPLE H1, 399.9780141 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 1 hr, 10 min, 8 sec
139.818
137.455
123.425
113.584
112.806
110.815
82.741
77.820
77.560
77.180
46.786
Br
H
N
N
H
Cl
Cl
3e
220
200
180
160
140
120
100
80
60
40
20
ppm


zh05905 64 (1.066) Cm (52:64-27:34)
TOF MS EI+
5.01e4
100
%
0
Br
H
N
N
H
Cl
Cl
3e
Chemical Formula: C9H9BrCl2N2
Exact Mass: 293.9326
246.9483
244.9496
258.9479
223.9794
221.9822
209.9762
196.9690
182.9567
260.9461
295.9301
293.9283
225.9604
75.0194
131.0239
155.9503
181.9560
76.0228
100.0098
117.0280
143.0338
167.9825
297.9280
291.9202
299.9081
288.8745
80
100
120
140
160
180
200
220
240
260
280
300
m/z

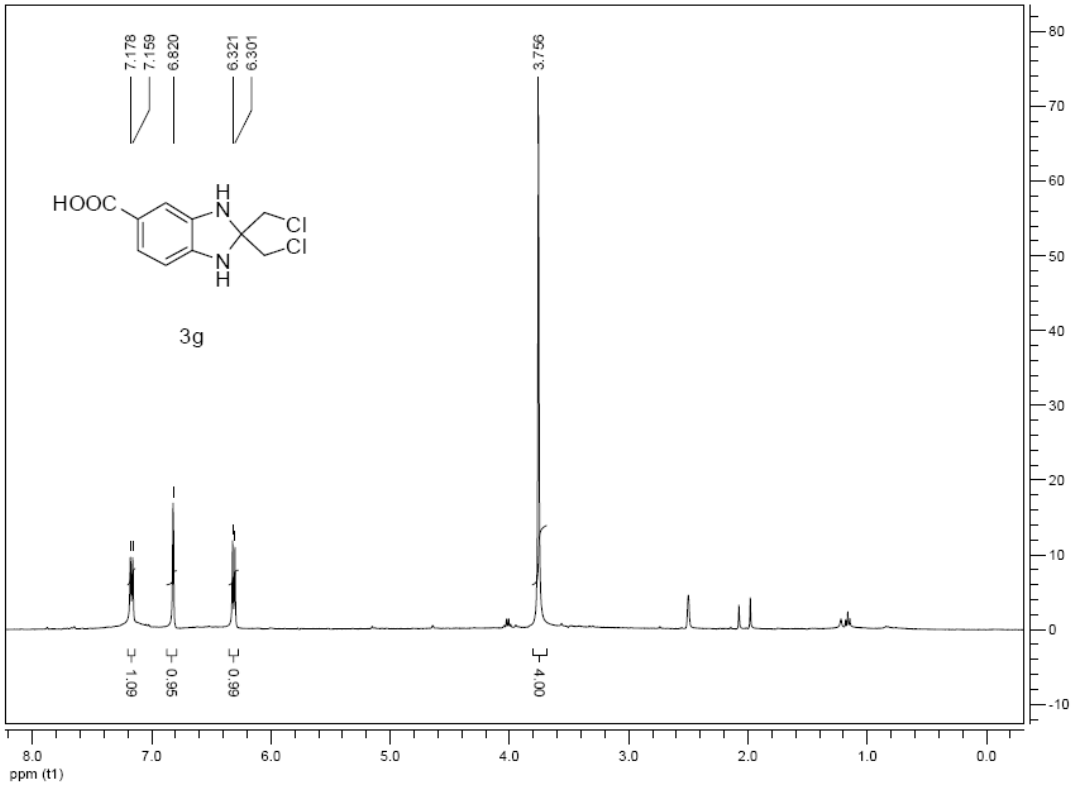

7.655
7.652
7.485
7.260
6.421
6.417
4.724
-0.009
Br
H
N
Cl
Cl
N
N
H
3f
0.90
0.93
0.96
4.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

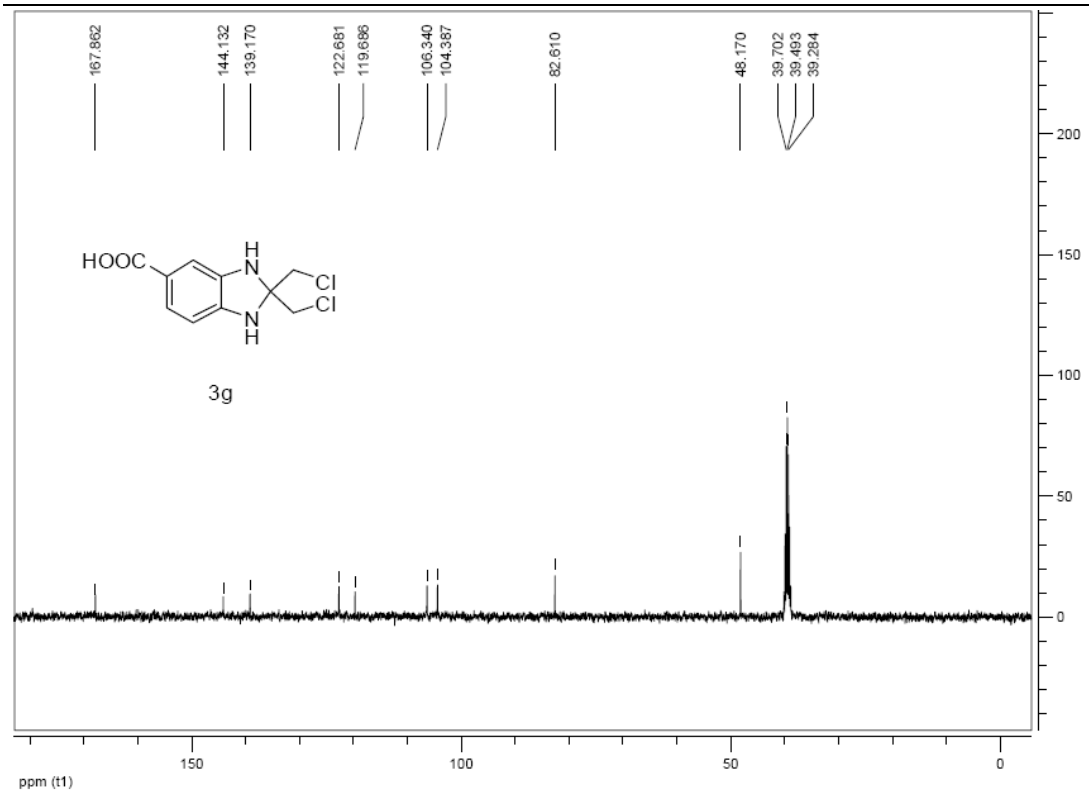

142.005
138.301
136.433
115.915
112.534
109.143
106.594
77.806
77.488
77.171
39.737
Br
H
N
Cl
Cl
N
N
H
3f
150
100
50
ppm (t1)

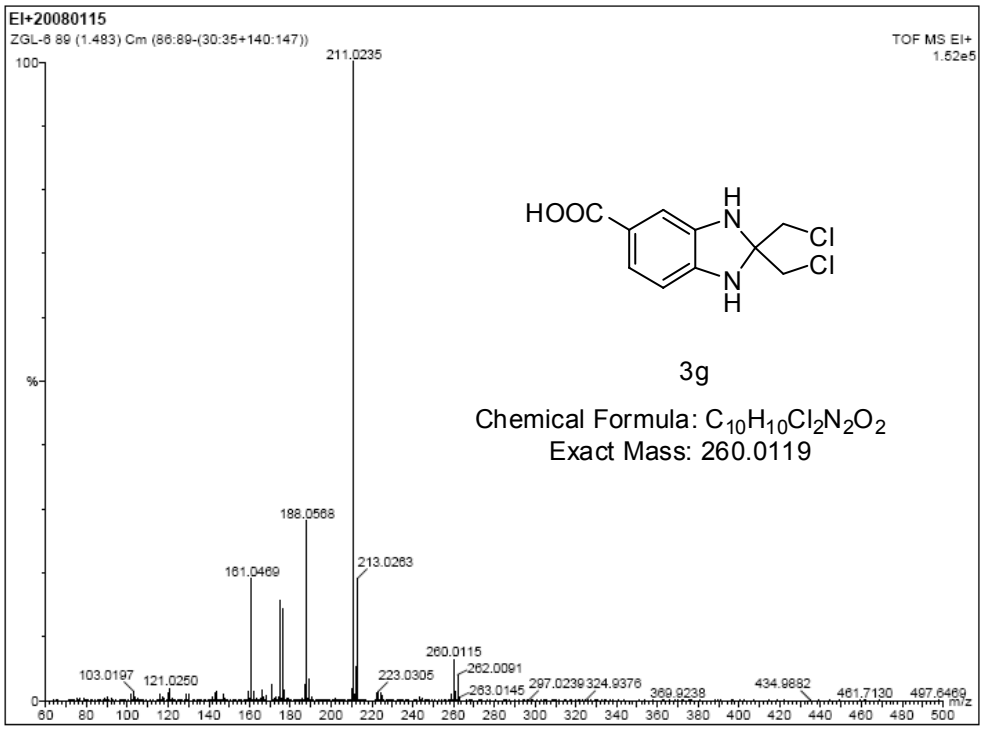


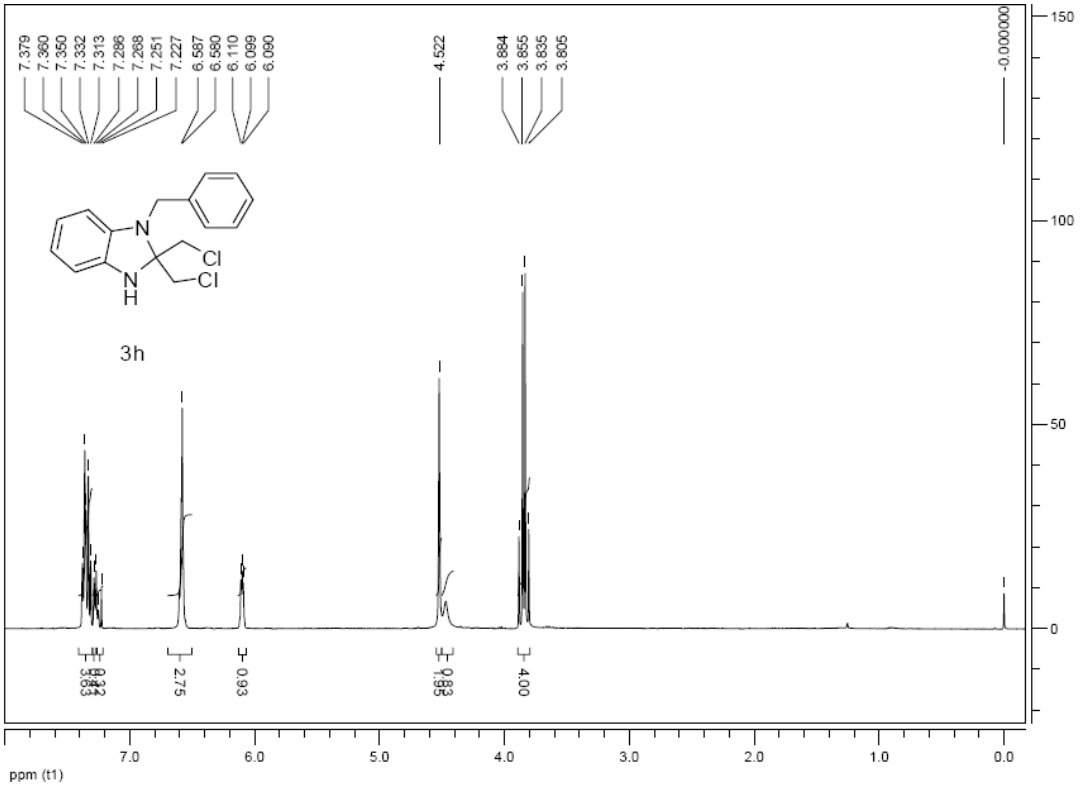
3f

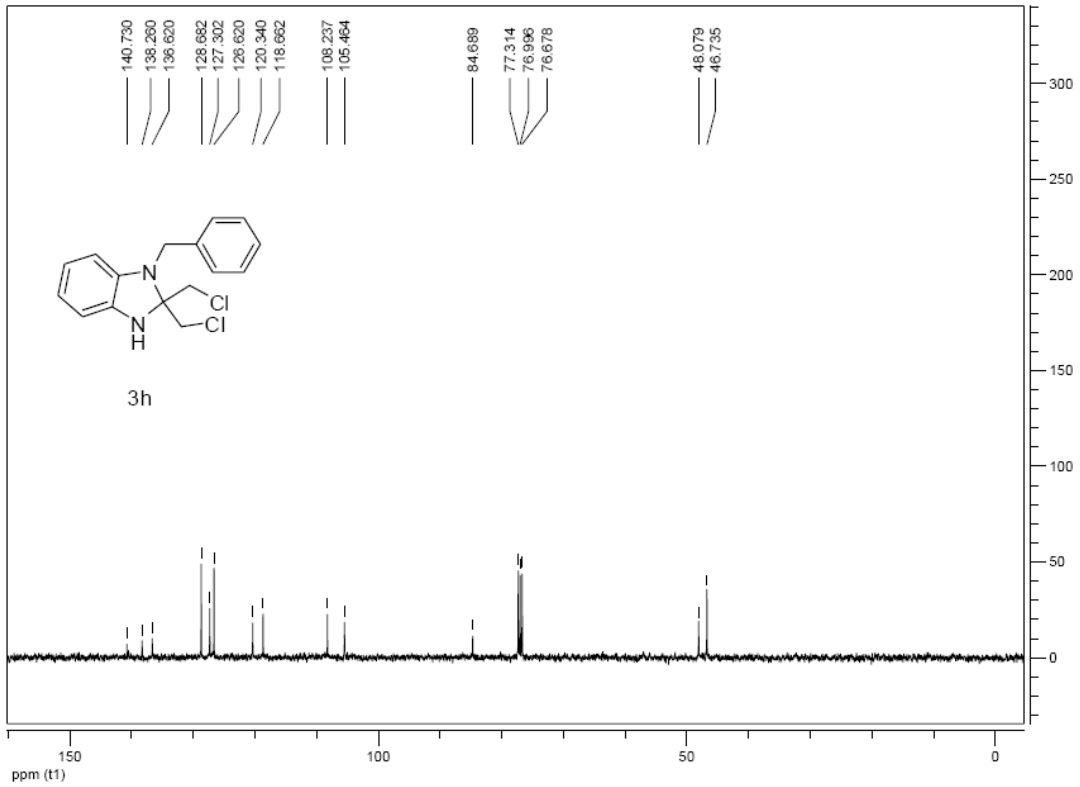
Chemical Formula: $C_8H_8BrCl_2N_3$
Exact Mass: 294.9279

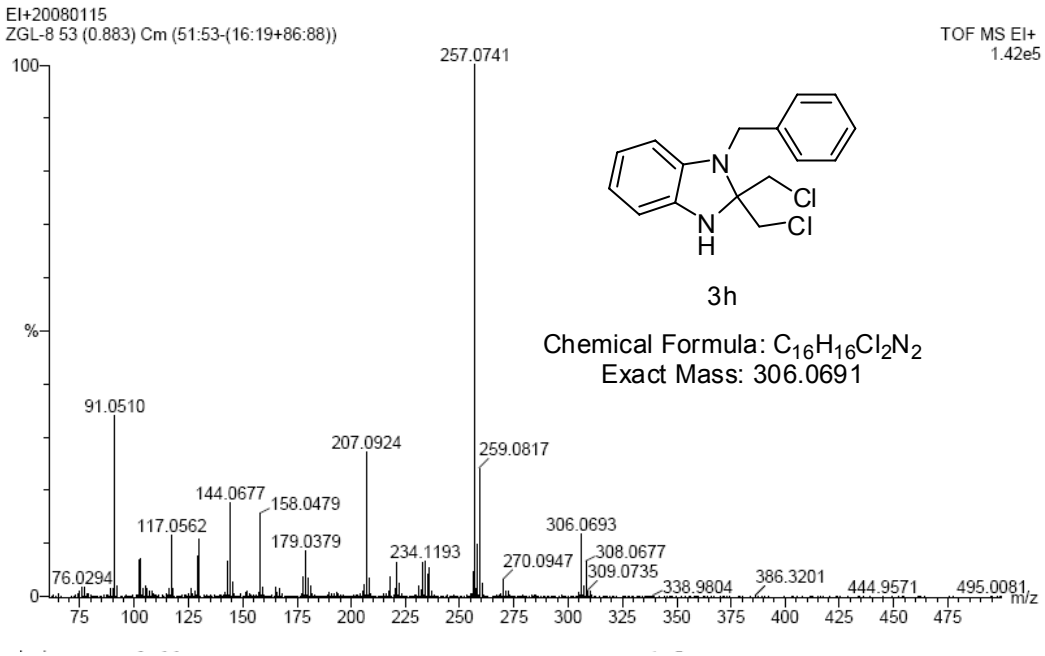



167.862
144.132
139.170
122.681
119.686
106.340
104.387
82.610
48.170
39.702
39.493
39.284
HOOC
H
N
Cl
Cl
N
H
3g
200
150
100
50
0
150
100
50
0
ppm (t1)


EI+20080115
ZGL-6 89 (1.483) Cm (86:89-(30:35+140:147))
TOF MS EI+
1.52e5
100
%
0
211.0235
188.0568
161.0469
213.0283
260.0115
262.0091
103.0197
121.0250
223.0305
263.0145
297.0239
324.9376
369.9238
434.9882
461.7130
497.6469
m/z
60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500
HOOC
H
N
Cl
Cl
N
H
3g
Chemical Formula: C10H10Cl2N2O2
Exact Mass: 260.0119

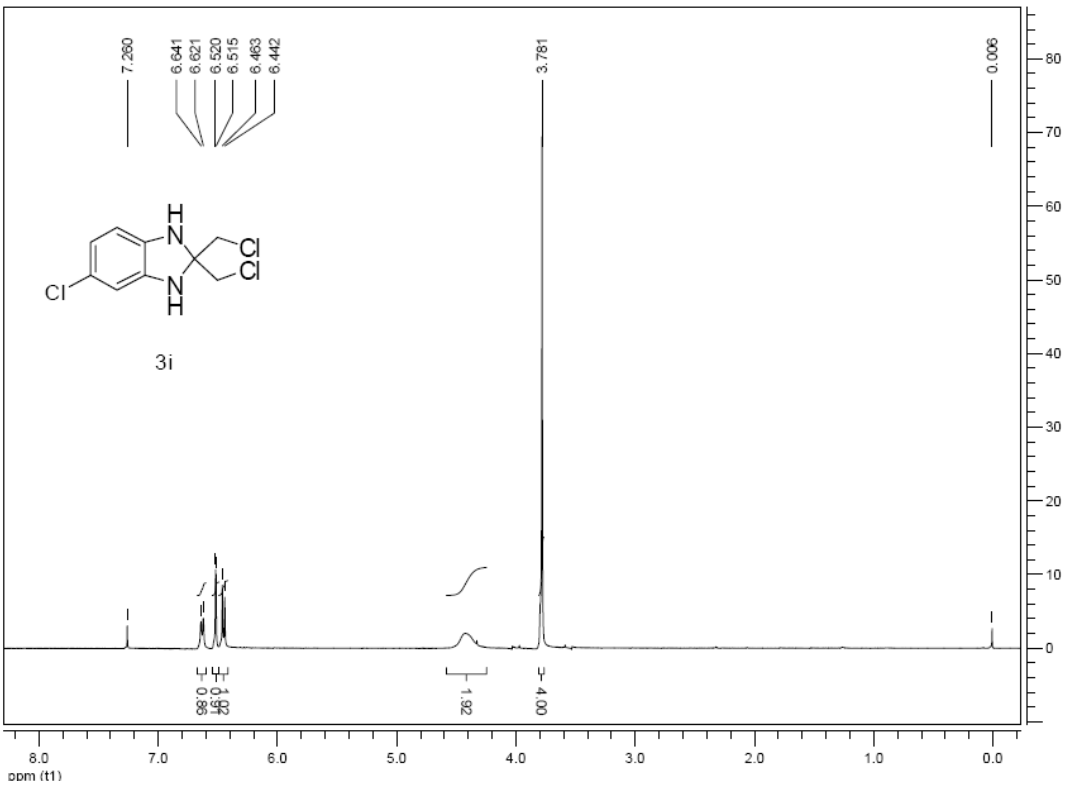

7.379
7.360
7.350
7.332
7.313
7.286
7.268
7.251
7.227
6.587
6.580
6.110
6.099
6.090
4.522
3.884
3.855
3.835
3.805
-0.000000
N
H
Cl
Cl
3h
2.75
0.93
1.95
4.00
ppm (t1)


140.730
138.260
136.620
128.682
127.302
126.620
120.340
118.662
106.237
105.464
84.689
77.314
76.996
76.678
48.079
46.735
N
H
Cl
Cl
3h
ppm (t1)

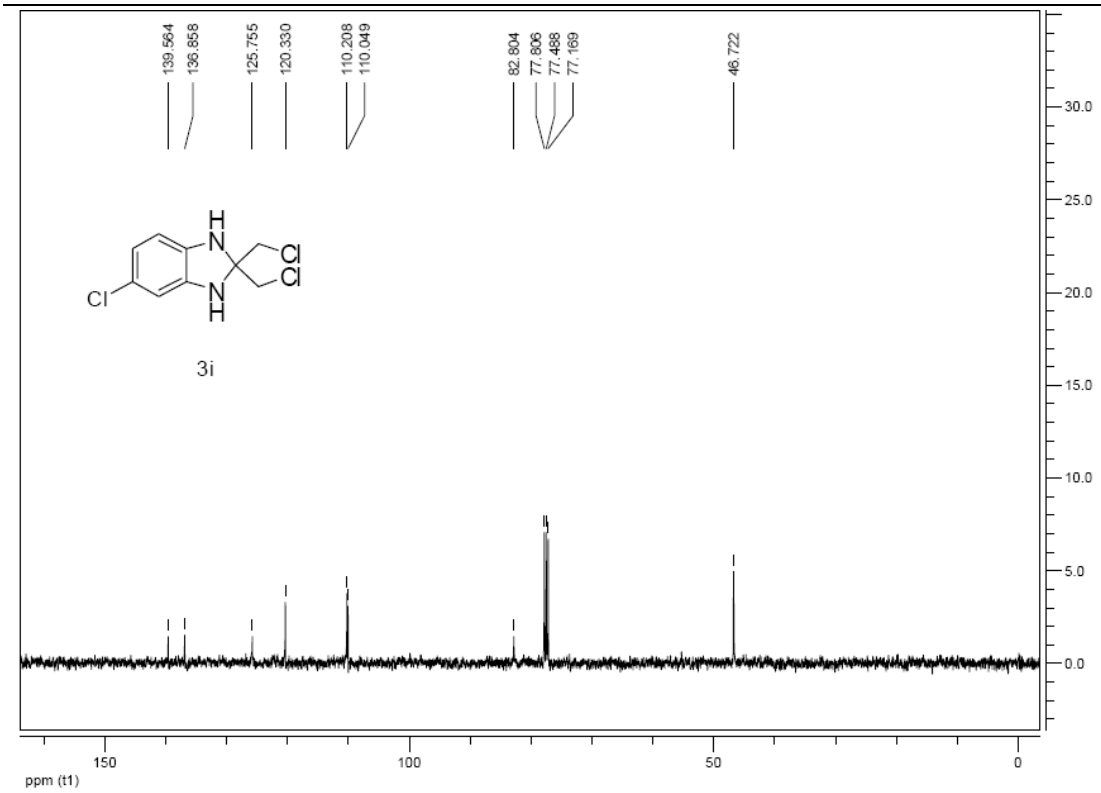


3h

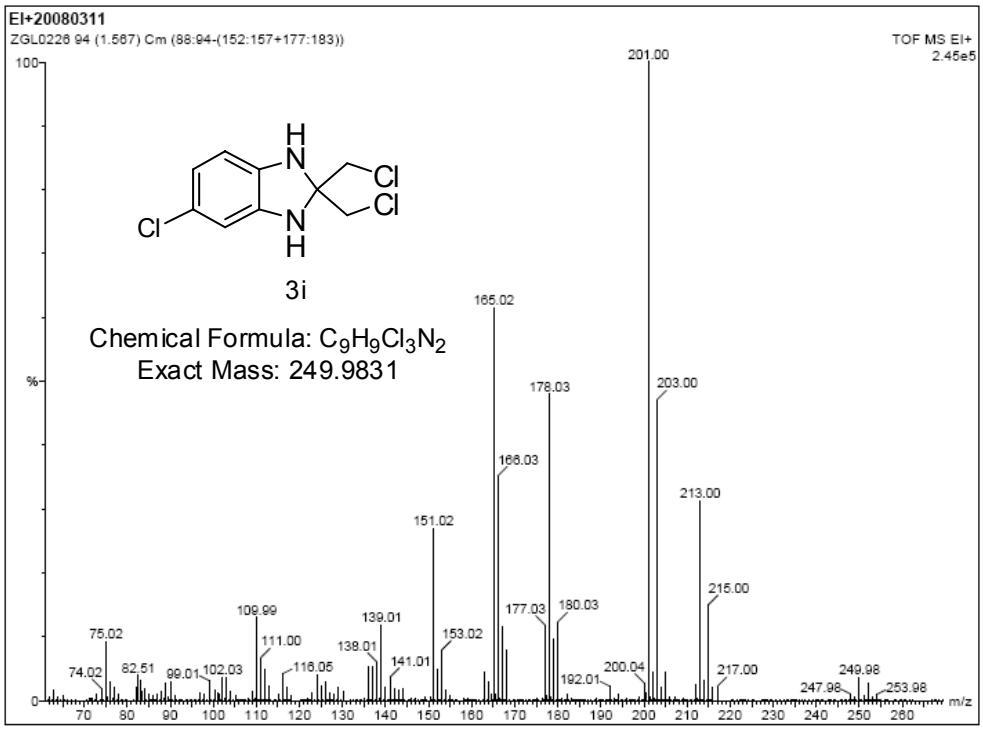
Chemical Formula: $C_{16}H_{16}Cl_2N_2$
Exact Mass: 306.0691



3i

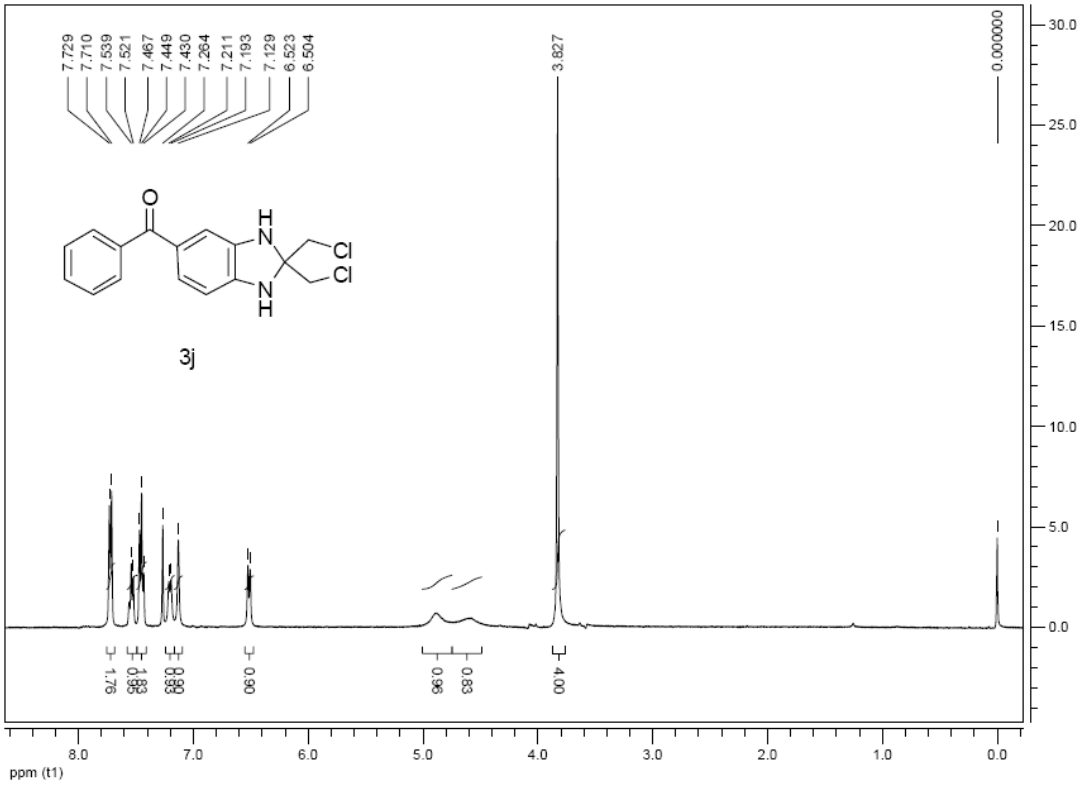


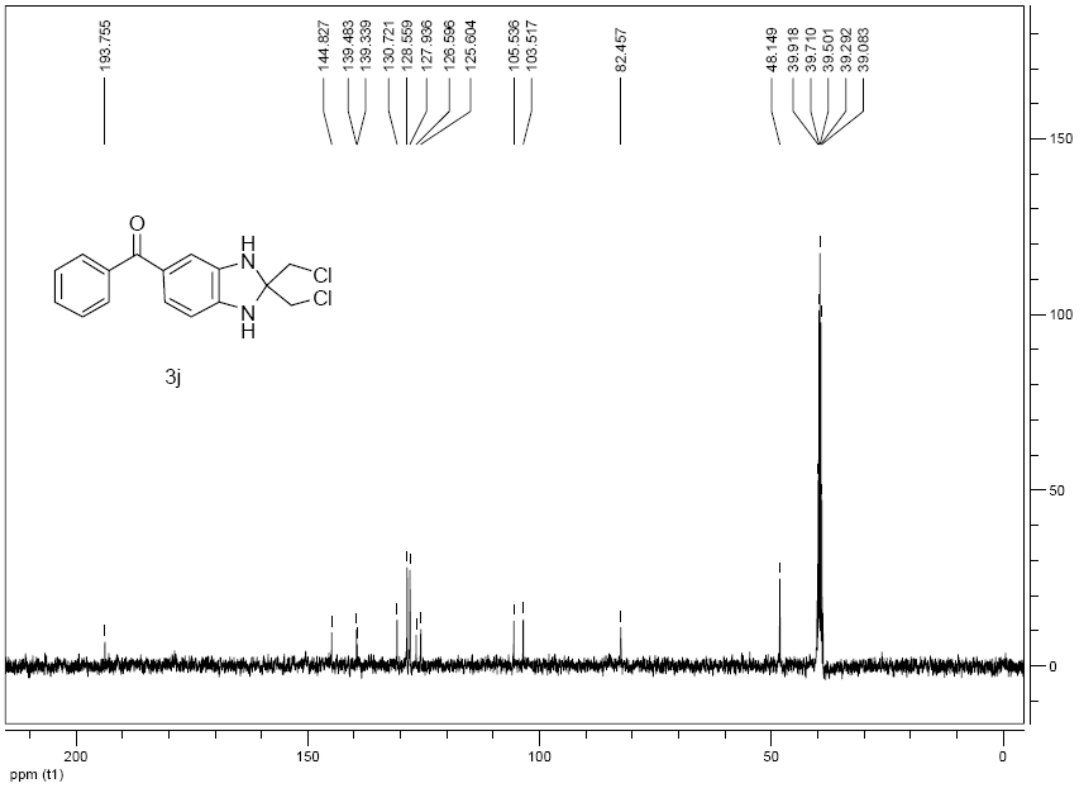
3i

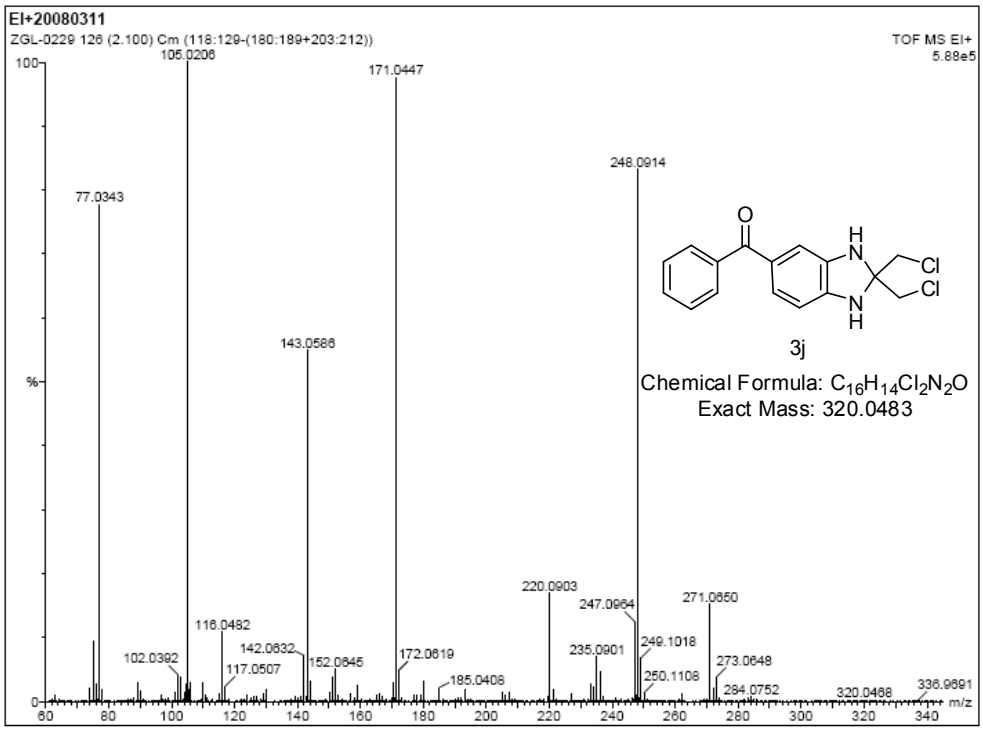


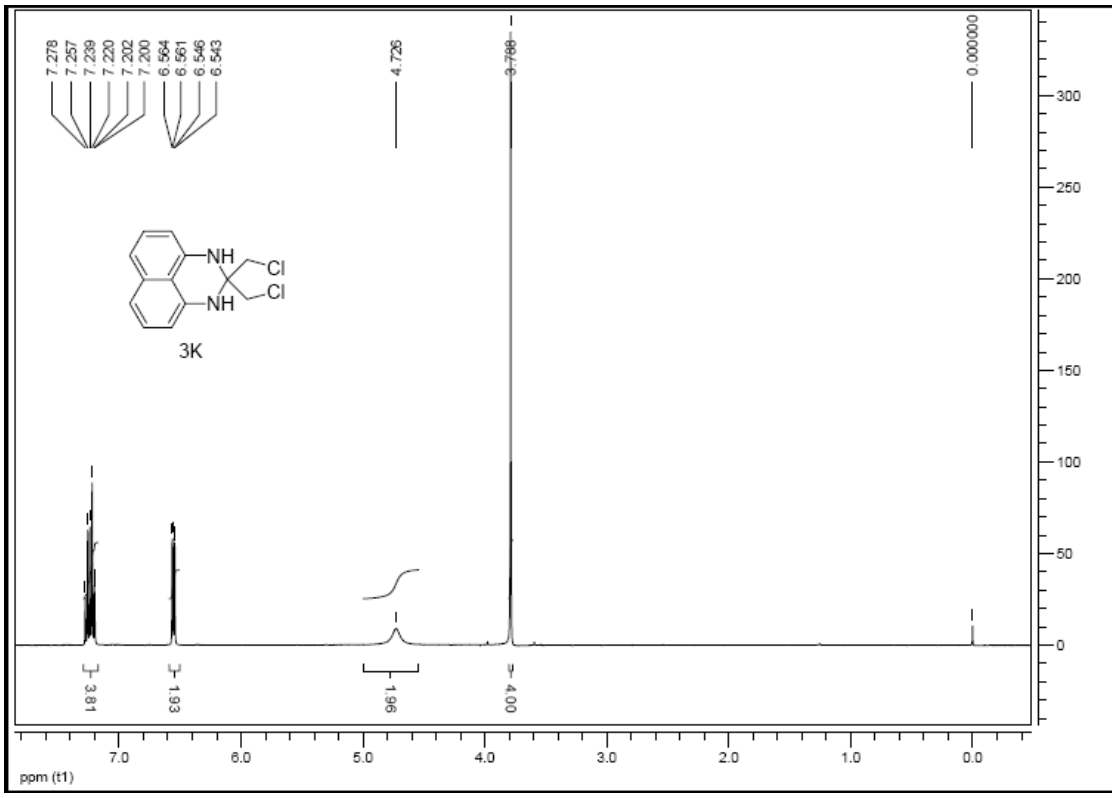
3i

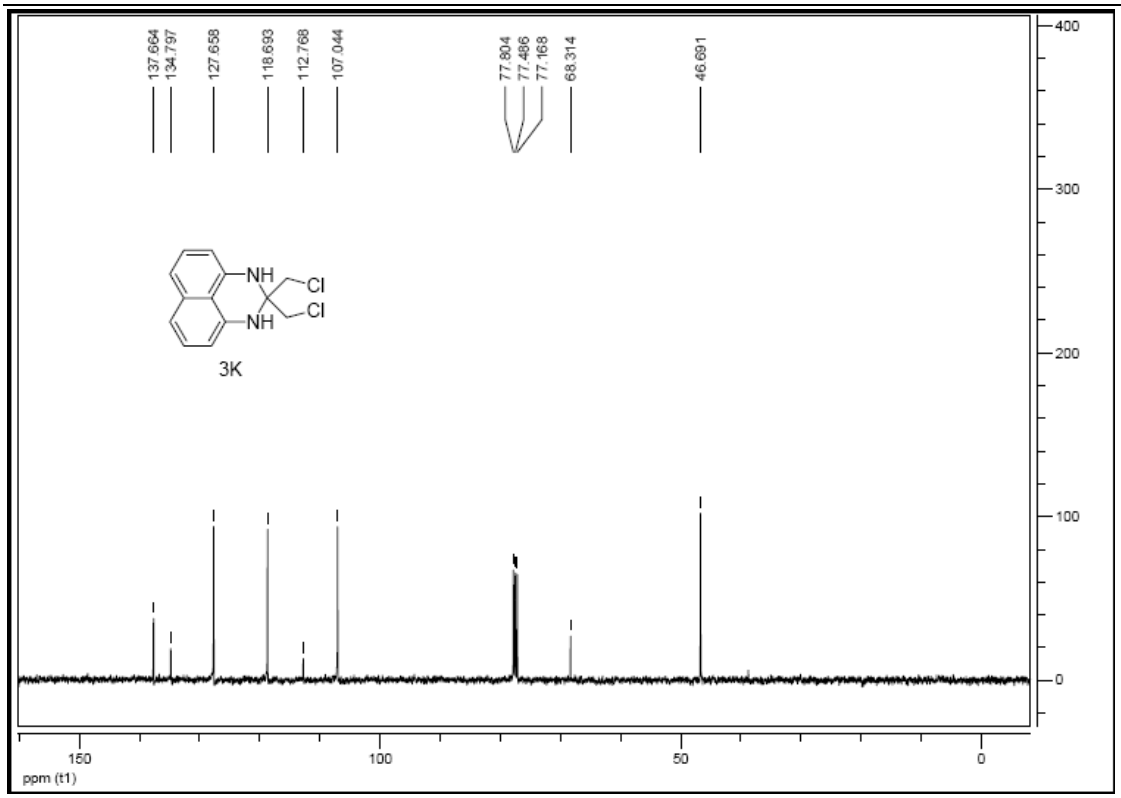
Chemical Formula: $C_9H_9Cl_3N_2$
Exact Mass: 249.9831

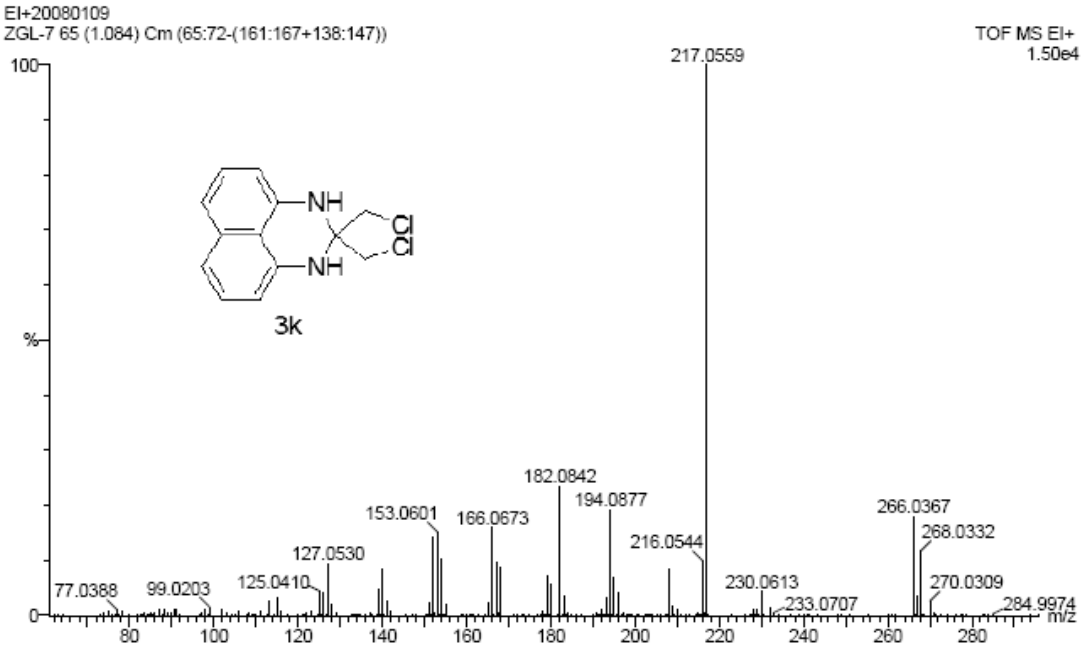

7.729
7.710
7.539
7.521
7.467
7.449
7.430
7.264
7.211
7.193
7.129
6.523
6.504
3.827
0.000000
O
H
N
Cl
Cl
N
H
3j
1.76
1.96
1.83
0.88
0.88
0.90
0.96
0.83
4.00
ppm (t1)

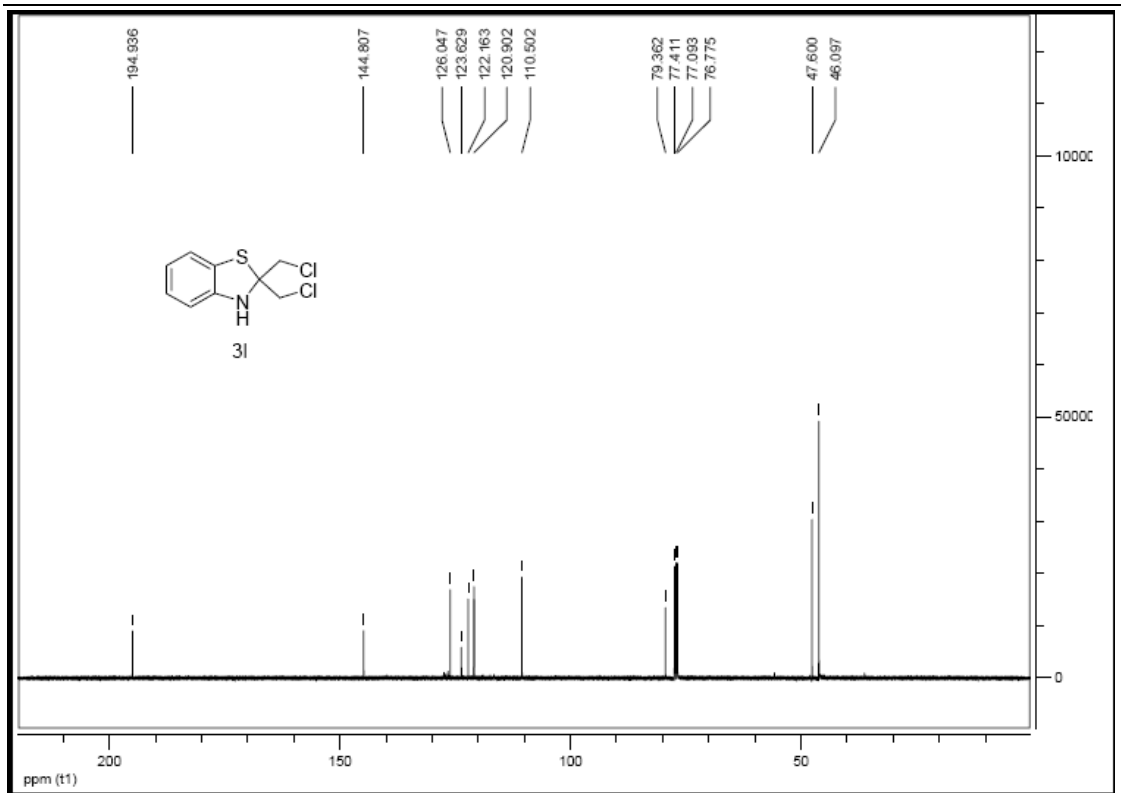

193.755
144.827
139.483
139.339
130.721
128.559
127.936
126.596
125.604
105.536
103.517
82.457
48.149
39.918
39.710
39.501
39.292
39.083
O
H
N
Cl
Cl
N
H
3j
ppm (t1)

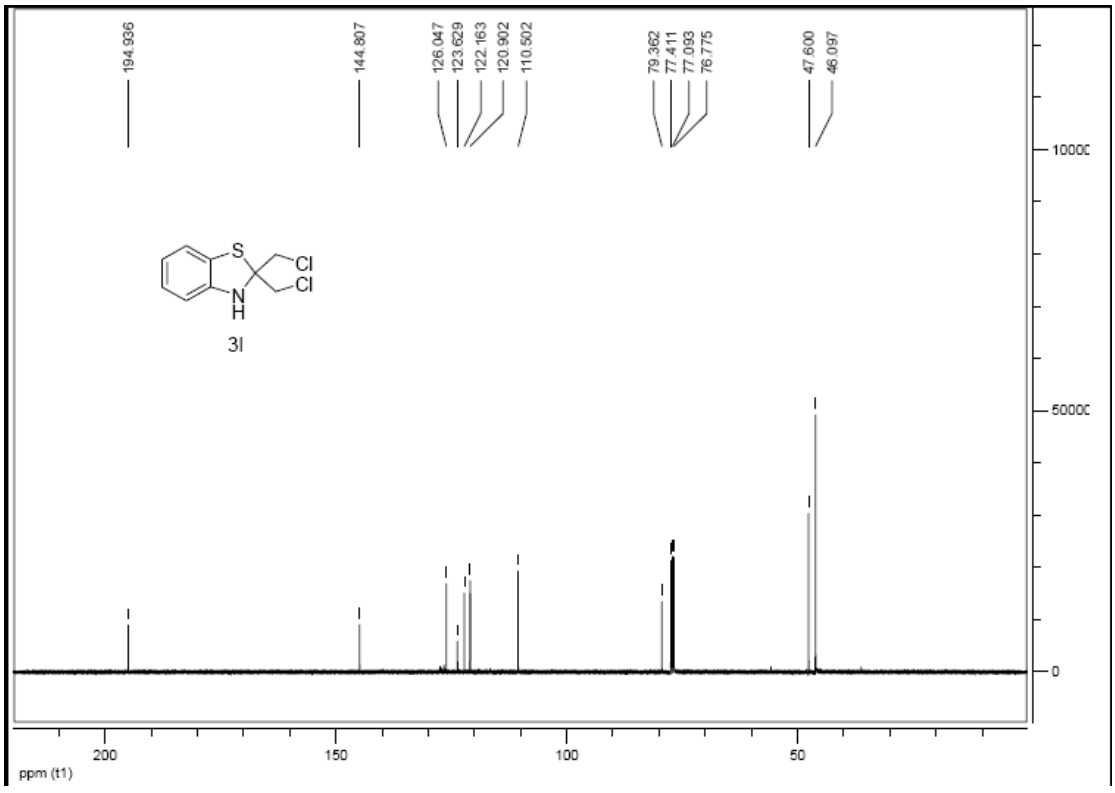

EI+20080311
ZGL-0229 126 (2.100) Cm (118:129-(180:189+203:212))
TOF MS EI+
5.88e5
105.0206
171.0447
248.0914
77.0343
143.0586
220.0903
247.0964
271.0850
116.0482
142.0632
249.1018
102.0392
117.0507
152.0646
172.0619
235.0901
273.0648
185.0408
250.1108
284.0752
320.0468
336.9691
m/z
3j
Chemical Formula: C16H14Cl2N2O
Exact Mass: 320.0483

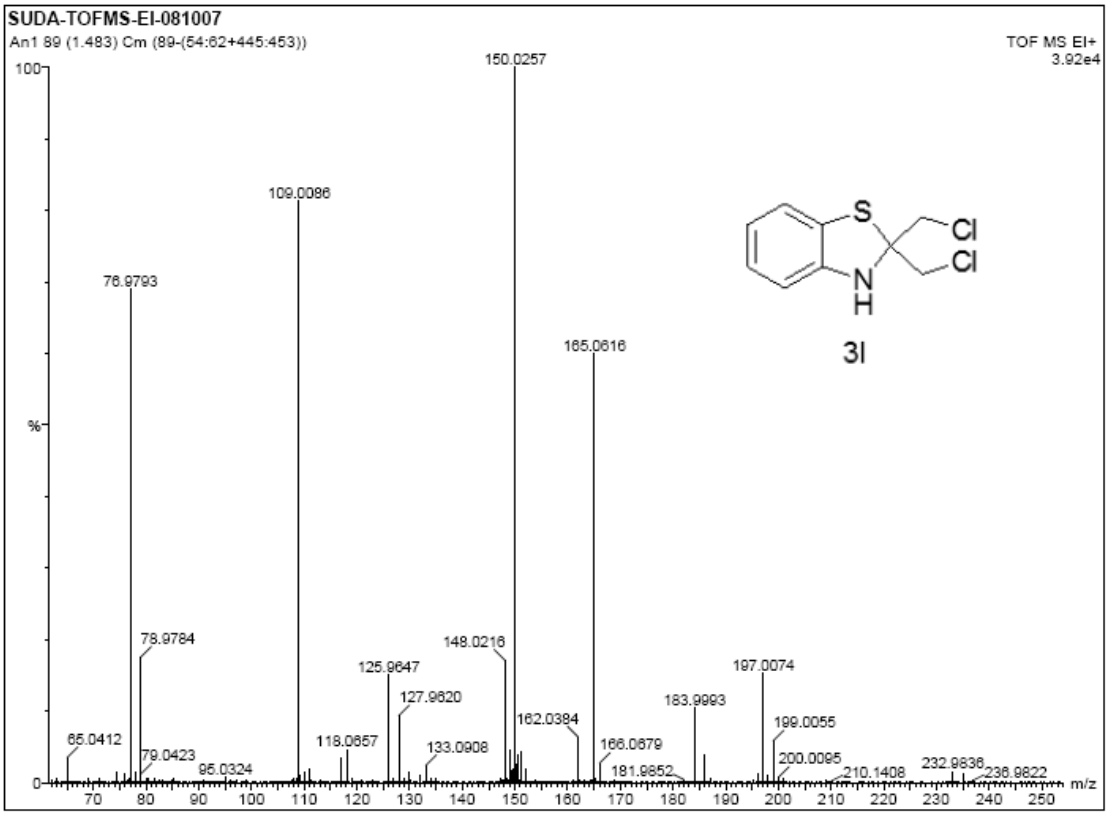

7.278
7.257
7.239
7.220
7.202
7.200
6.564
6.561
6.546
6.543
4.726
3.788
0.000000
3K
3.81
1.93
1.96
4.00
ppm (t1)

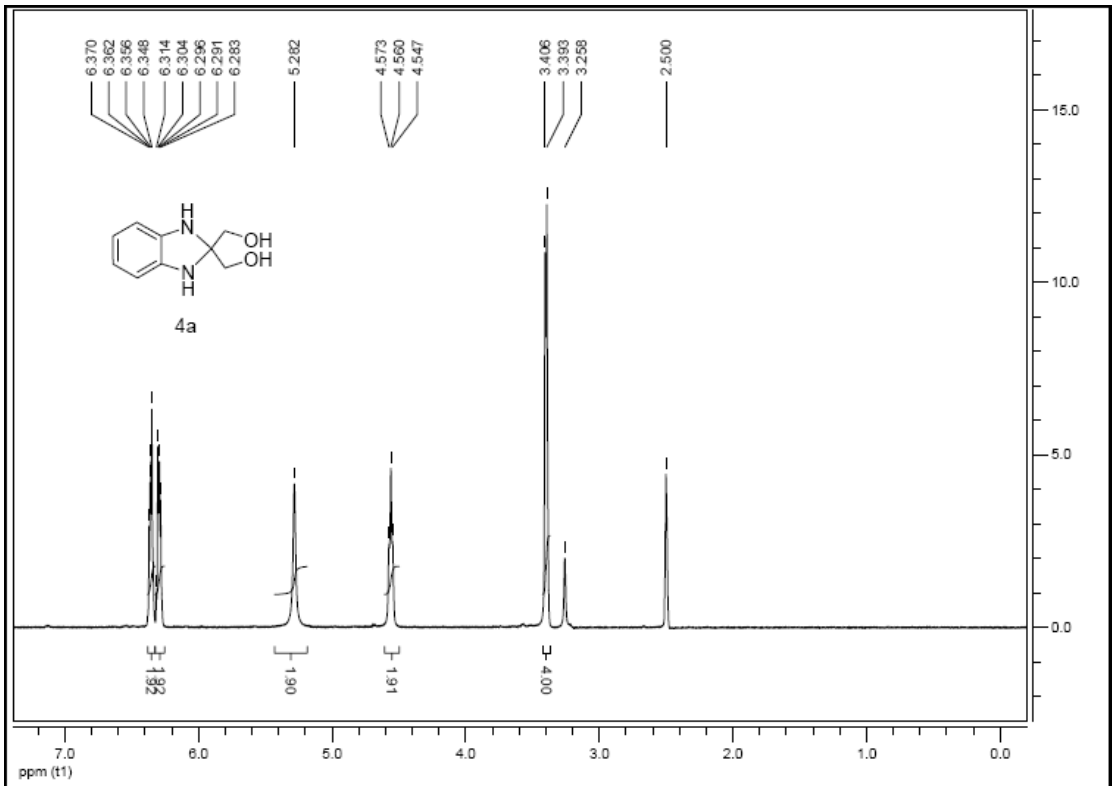

137.664
134.797
127.658
118.693
112.768
107.044
77.804
77.486
77.168
68.314
46.691
NH
Cl
Cl
NH
3K
400
300
200
100
0
150
100
50
0
ppm (t1)

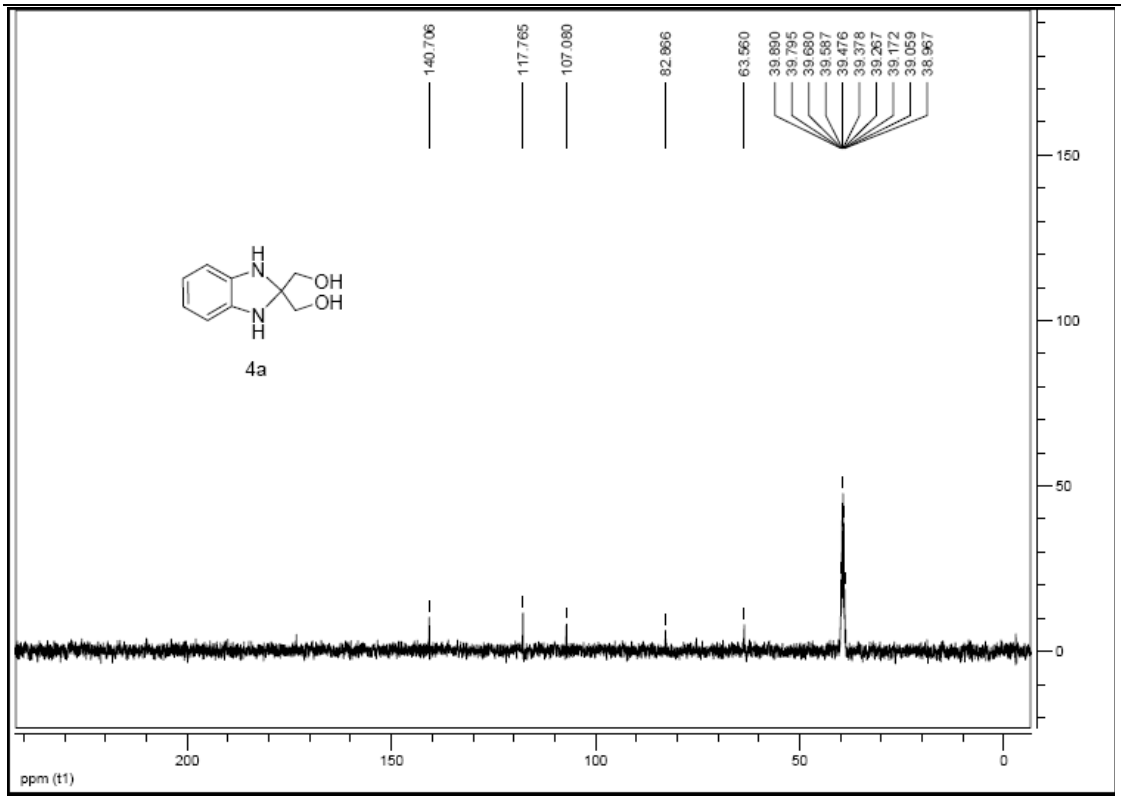

EI+20080109
ZGL-7 65 (1.084) Cm (65:72-(161:167+138:147))
TOF MS EI+
1.50e4
100
%
0
217.0559
182.0842
194.0877
153.0601
166.0673
266.0367
268.0332
216.0544
127.0530
125.0410
77.0388
99.0203
230.0613
270.0309
233.0707
284.9974
NH
Cl
Cl
NH
3k
80
100
120
140
160
180
200
220
240
260
280
m/z

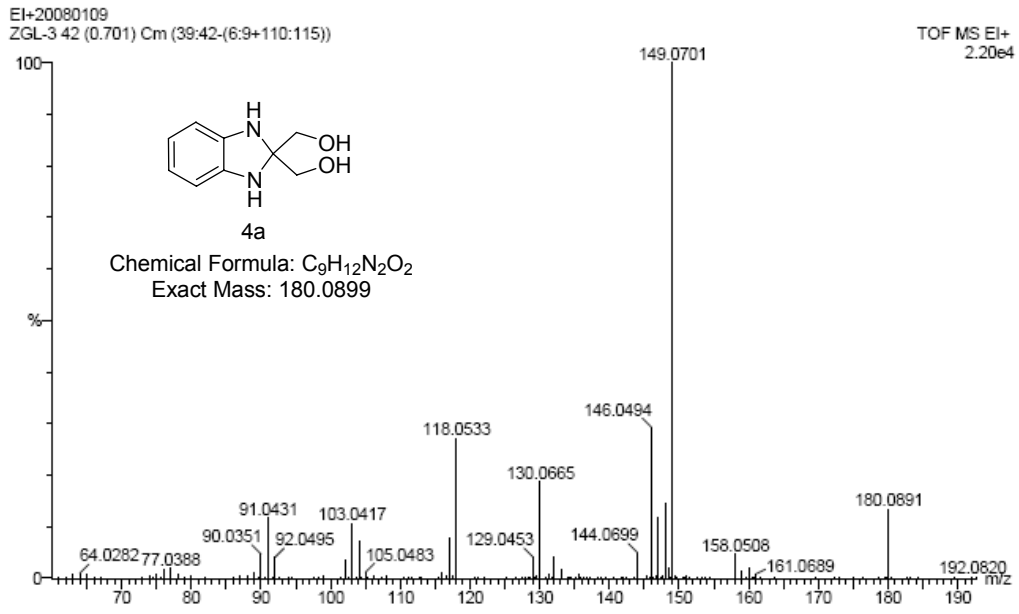

194.936
144.807
126.047
123.629
122.163
120.902
110.502
79.362
77.411
77.093
76.775
47.600
46.097
10000
50000
0
200
150
100
50
ppm (t1)
S
Cl
Cl
N
H
3l

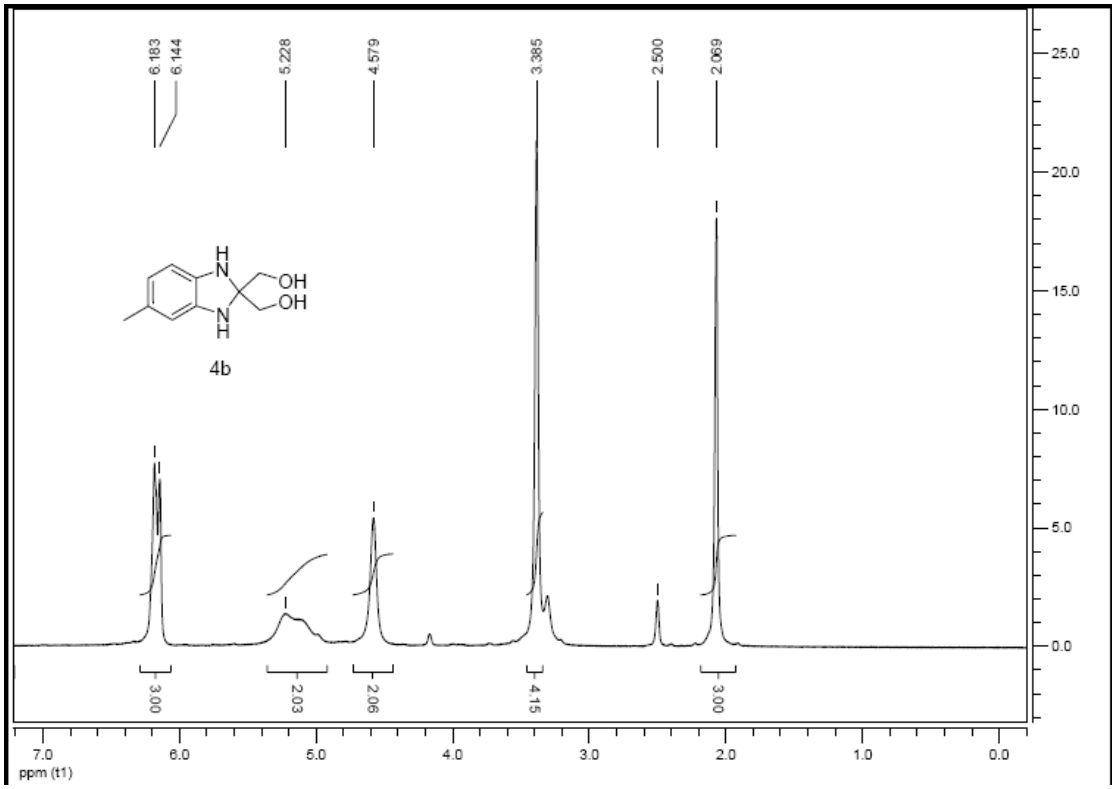

194.936
144.807
126.047
123.629
122.163
120.902
110.502
79.362
77.411
77.093
76.775
47.600
46.097
10000
50000
0
200
150
100
50
ppm (t1)
S
Cl
Cl
N
H
3l

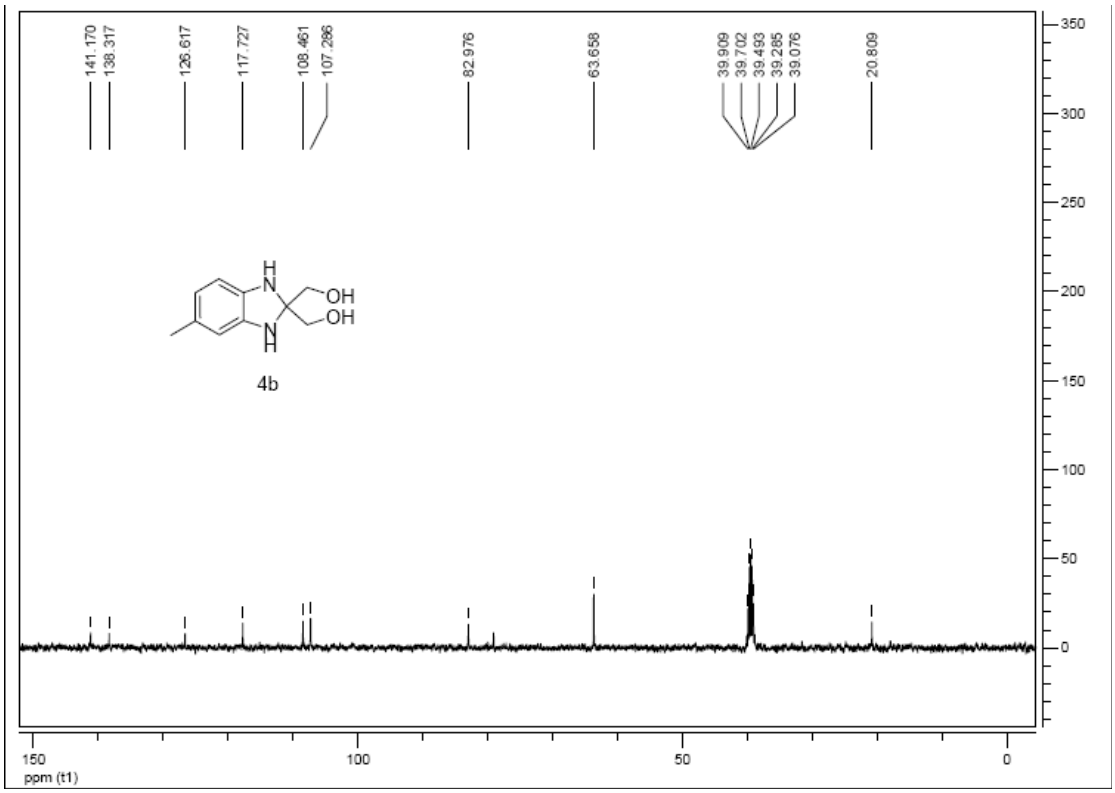

SUDA-TOFMS-EI-081007
An1 89 (1.483) Cm (89-(54:62+445:453))
TOF MS EI+
3.92e4
150.0257
109.0086
76.9793
165.0616
78.9784
148.0216
125.9647
197.0074
127.9820
183.9993
162.0384
199.0055
65.0412
118.0857
133.0908
166.0679
79.0423
200.0095
95.0324
181.9852
210.1408
232.9836
238.9822
m/z
%
S
Cl
Cl
N
H
3l

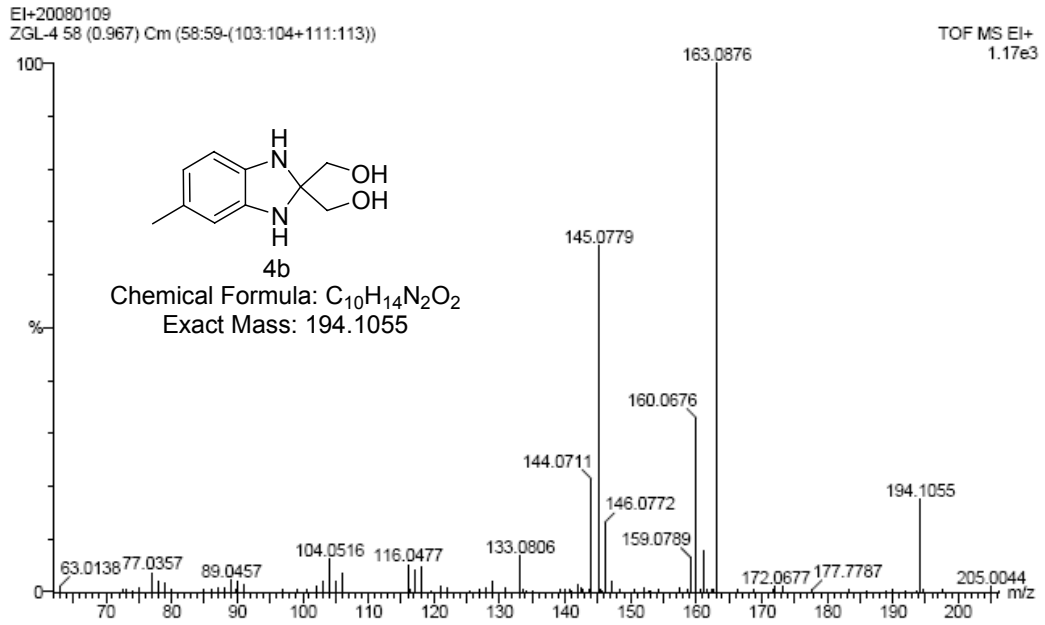

6.370
6.362
6.356
6.348
6.314
6.304
6.296
6.291
6.283
5.282
4.573
4.560
4.547
3.406
3.393
3.258
2.500
H
N
OH
OH
N
H
4a
1.00
1.90
1.91
4.00
ppm (t1)

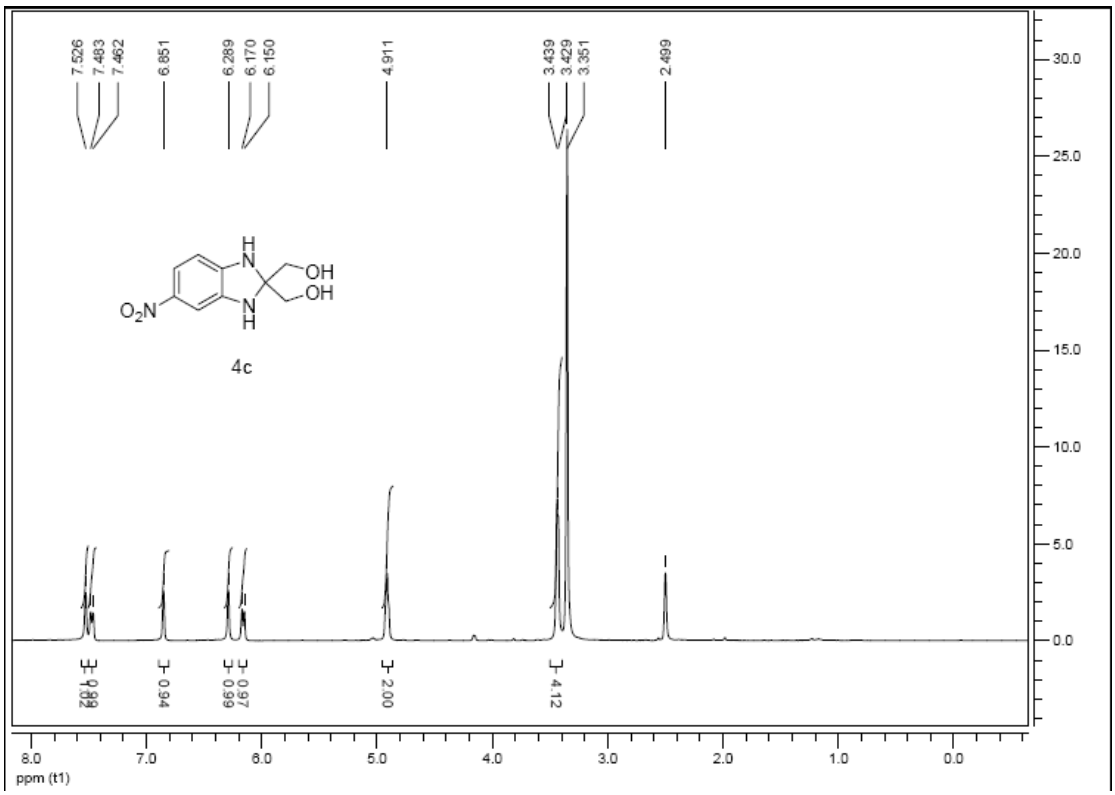

140.706
117.765
107.080
82.866
63.560
39.890
39.795
39.680
39.587
39.476
39.378
39.267
39.172
39.059
38.967
150
100
50
0
200
150
100
50
0
ppm (t1)
H
N
OH
OH
N
H
4a

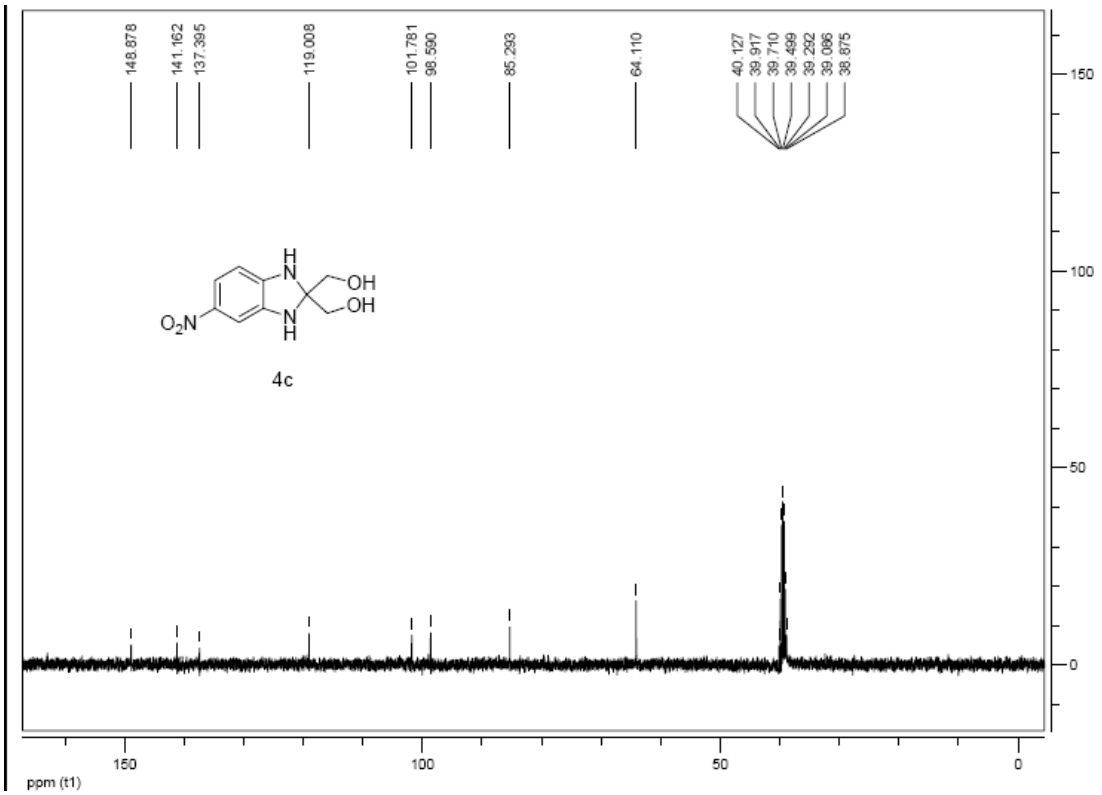

EI+20080109
ZGL-3 42 (0.701) Cm (39:42-(6:9+110:115))
TOF MS EI+
2.20e4
100
%
0
149.0701
146.0494
118.0533
130.0665
180.0891
91.0431
103.0417
90.0351
92.0495
129.0453
144.0699
158.0508
64.0282
77.0388
105.0483
161.0689
192.0820
70
80
90
100
110
120
130
140
150
160
170
180
190
m/z
H
N
OH
OH
N
H
4a
Chemical Formula: C9H12N2O2
Exact Mass: 180.0899

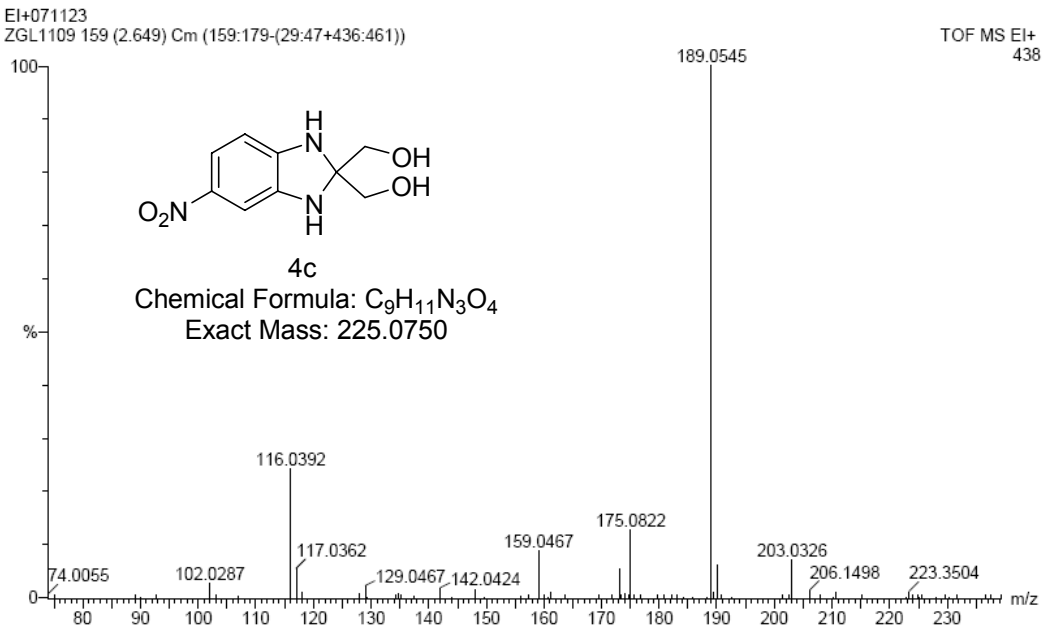

6.183
6.144
5.228
4.579
3.385
2.500
2.069
4b
3.00
2.03
2.06
4.15
3.00
ppm (t1)

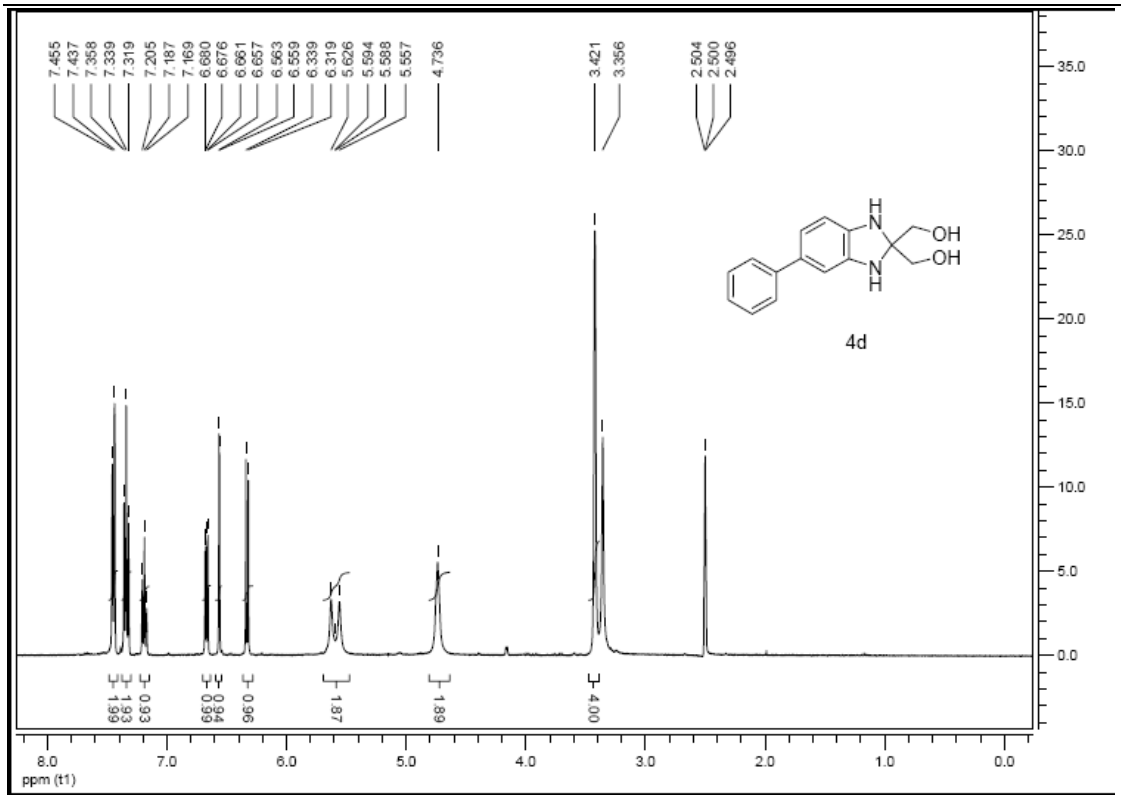

141.170
138.317
126.617
117.727
108.461
107.286
82.976
63.658
39.909
39.702
39.493
39.285
39.076
20.809
4b
ppm (t1)

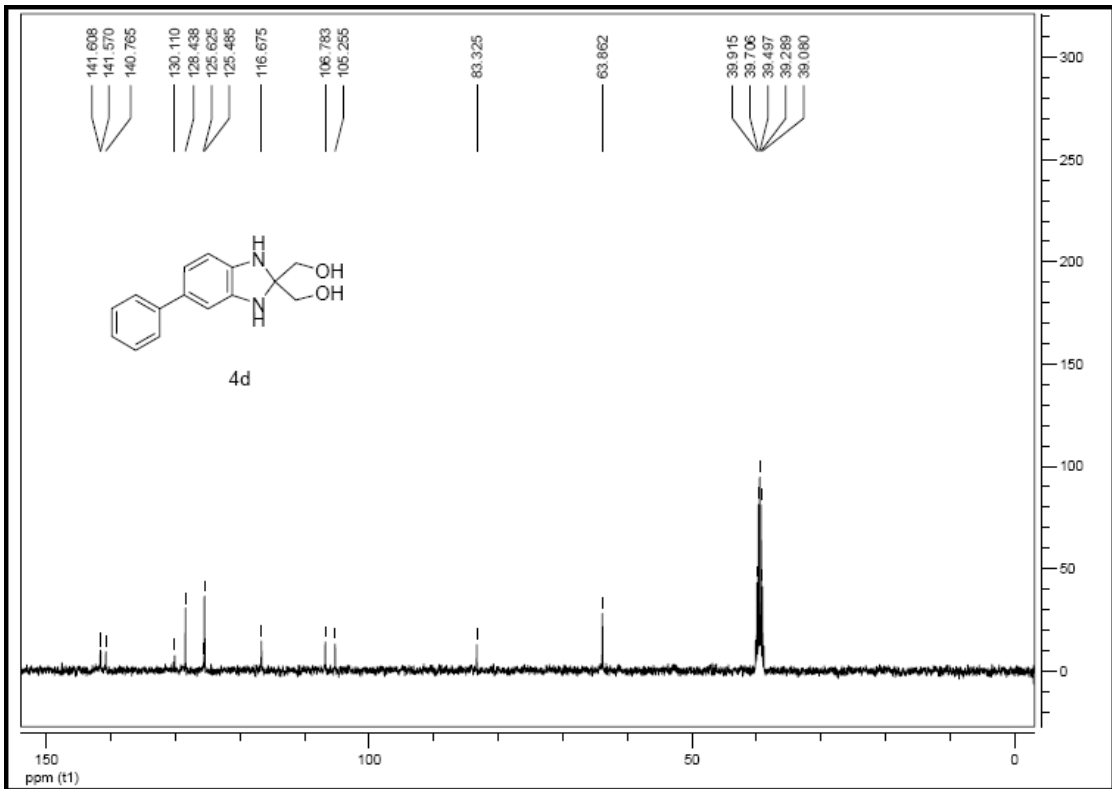

EI+20080109
ZGL-4 58 (0.967) Cm (58:59-(103:104+111:113))
TOF MS EI+
1.17e3
100
%
0
163.0876
145.0779
160.0676
144.0711
146.0772
194.1055
159.0789
104.0516
116.0477
133.0806
63.0138
77.0357
89.0457
172.0677
177.7787
205.0044
m/z
70
80
90
100
110
120
130
140
150
160
170
180
190
200
4b
Chemical Formula: C10H14N2O2
Exact Mass: 194.1055

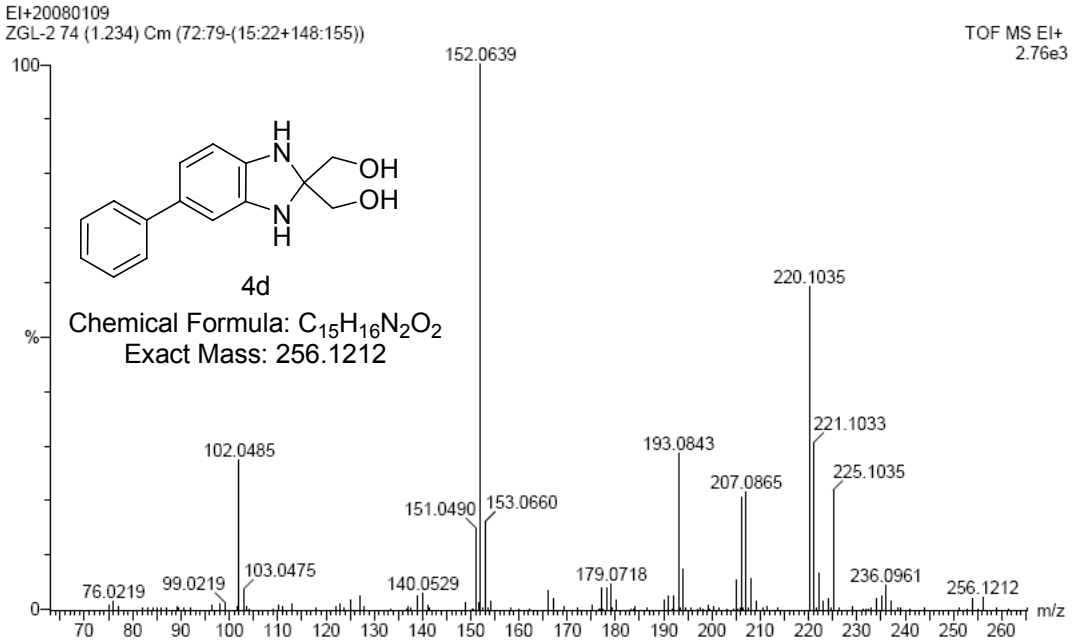

7.526
7.483
7.462
6.851
6.289
6.170
6.150
4.911
3.439
3.429
3.351
2.499
4c
1.00
0.94
0.99
0.97
2.00
4.12
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
30.0
25.0
20.0
15.0
10.0
5.0
0.0

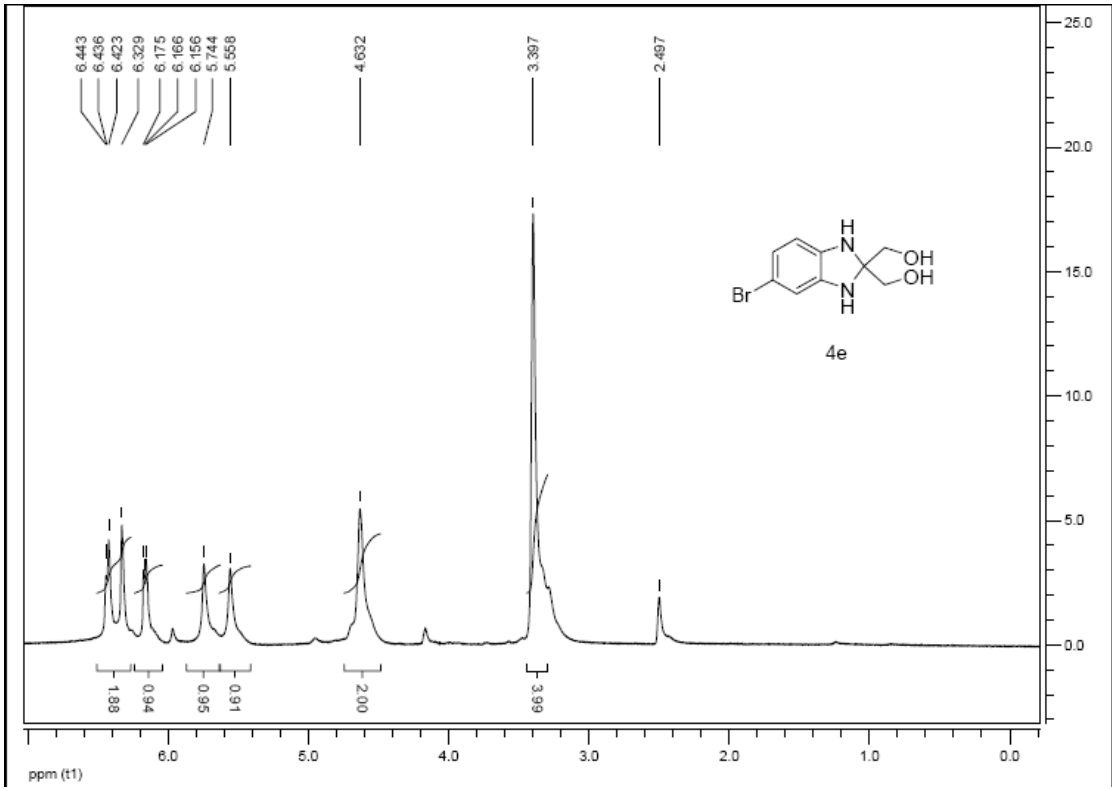

148.878
141.162
137.395
119.008
101.781
98.590
85.293
64.110
40.127
39.917
39.710
39.499
39.292
39.086
38.875
150
100
50
0
ppm (t1)
4c

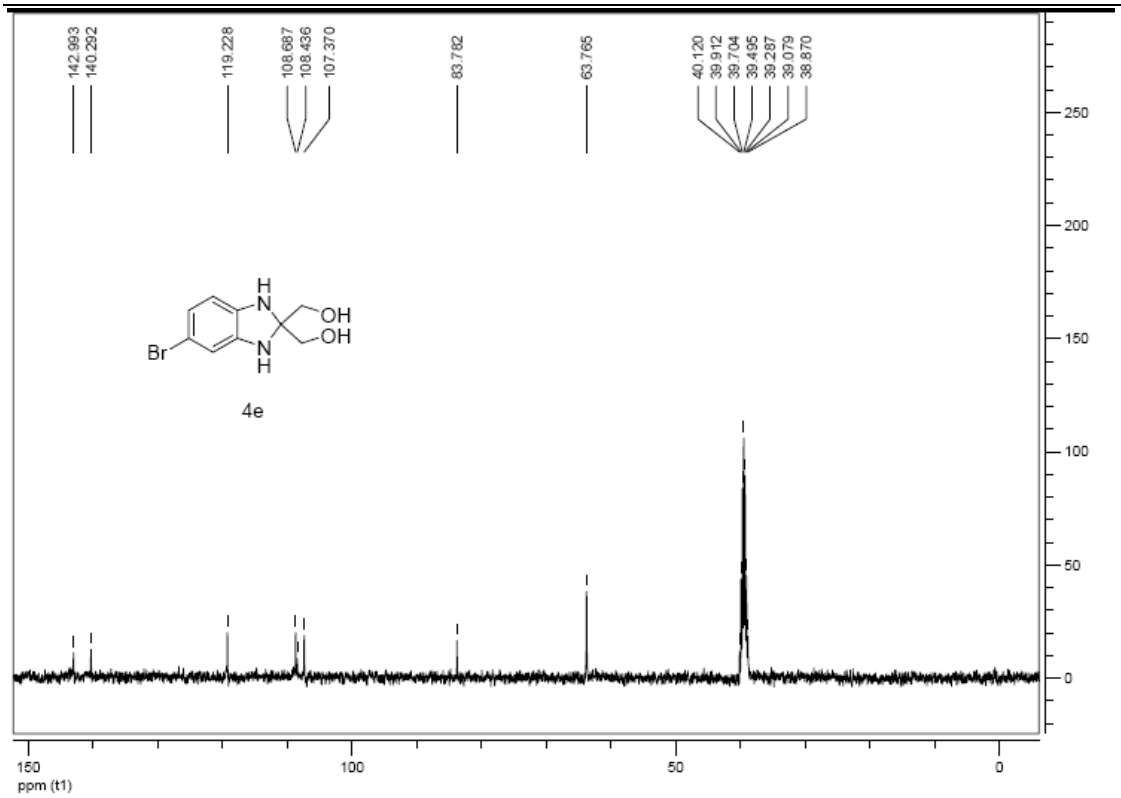

EI+071123
ZGL1109 159 (2.649) Cm (159:179-(29:47+436:461))
TOF MS EI+
438
189.0545
4c
Chemical Formula: C9H11N3O4
Exact Mass: 225.0750
116.0392
117.0362
159.0467
175.0822
203.0326
74.0055
102.0287
129.0467
142.0424
206.1498
223.3504
m/z

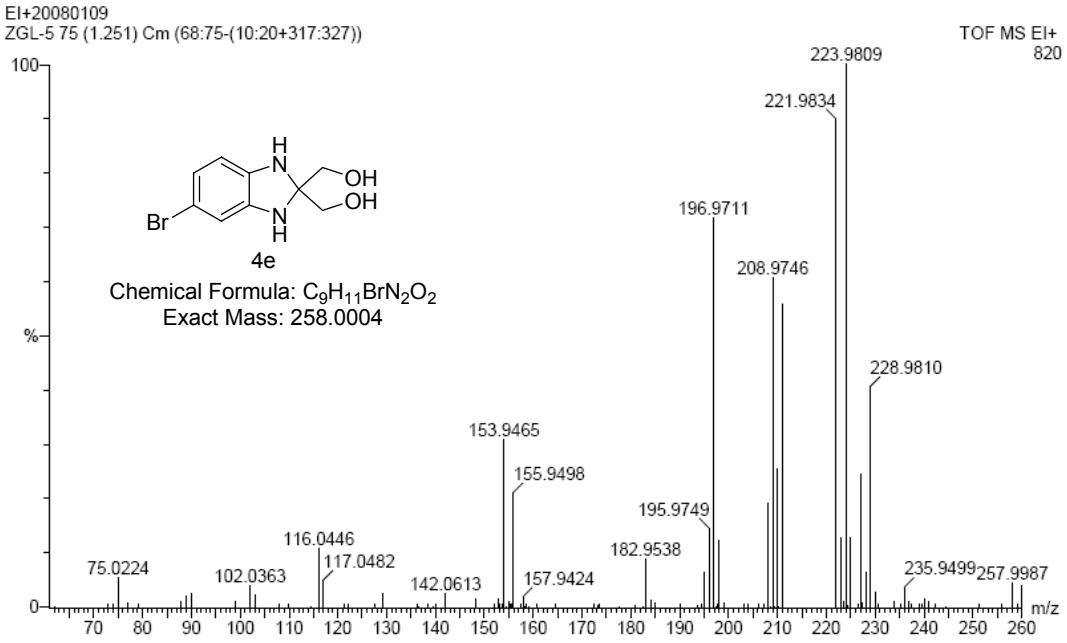

7.455
7.437
7.358
7.339
7.319
7.205
7.187
7.169
6.680
6.676
6.661
6.657
6.563
6.559
6.339
6.319
5.626
5.594
5.588
5.557
4.736
3.421
3.356
2.504
2.500
2.496
OH
OH
4d
1.99
1.93
0.93
0.99
0.94
0.96
1.87
1.89
4.00
ppm (t1)

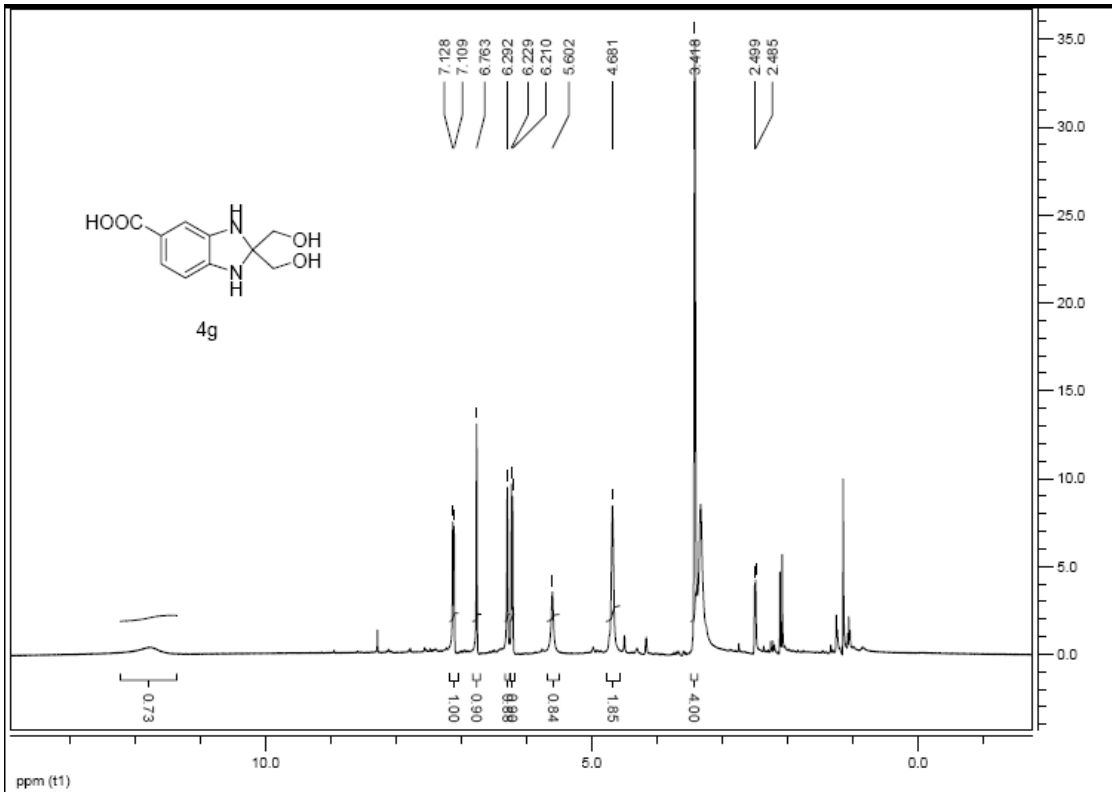

141.608
141.570
140.765
130.110
128.438
125.625
125.485
116.675
106.783
105.255
83.325
63.862
39.915
39.706
39.497
39.289
39.080
OH
OH
4d
ppm (t1)

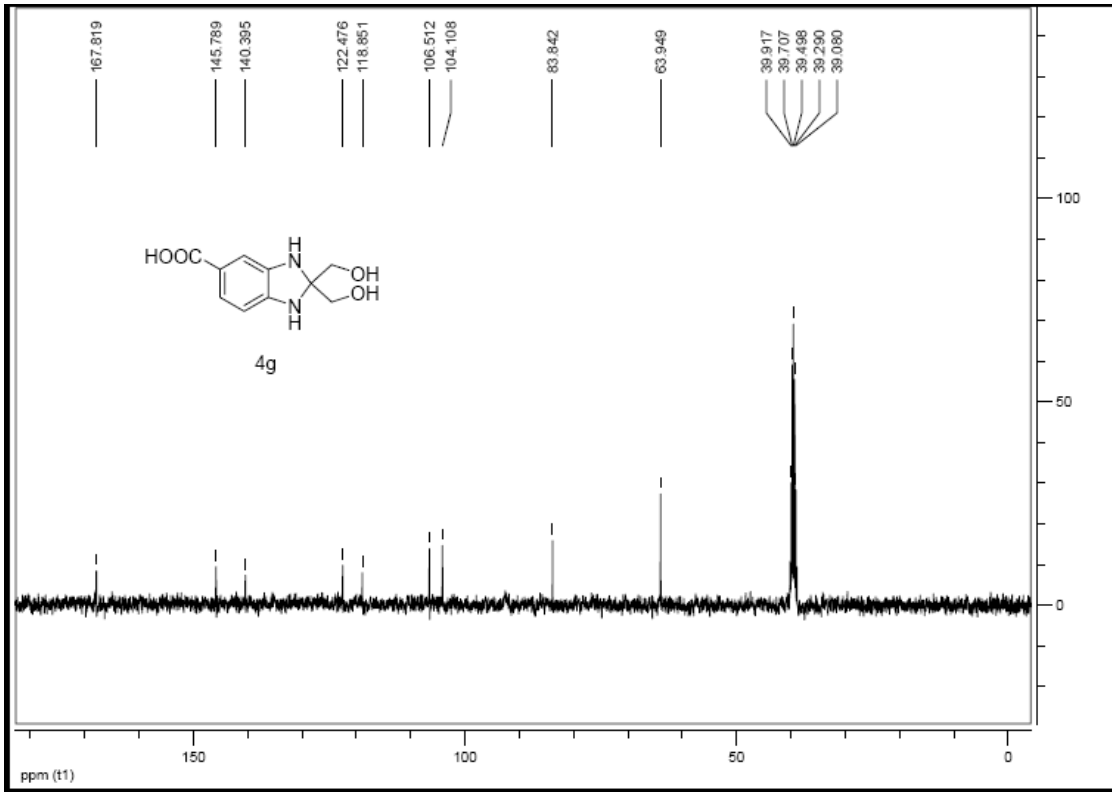

EI+20080109
ZGL-2 74 (1.234) Cm (72:79-(15:22+148:155))
TOF MS EI+
2.76e3
4d
Chemical Formula: C15H16N2O2
Exact Mass: 256.1212
152.0639
220.1035
221.1033
193.0843
207.0865
225.1035
102.0485
151.0490
153.0660
103.0475
76.0219
99.0219
140.0529
179.0718
236.0961
256.1212
m/z

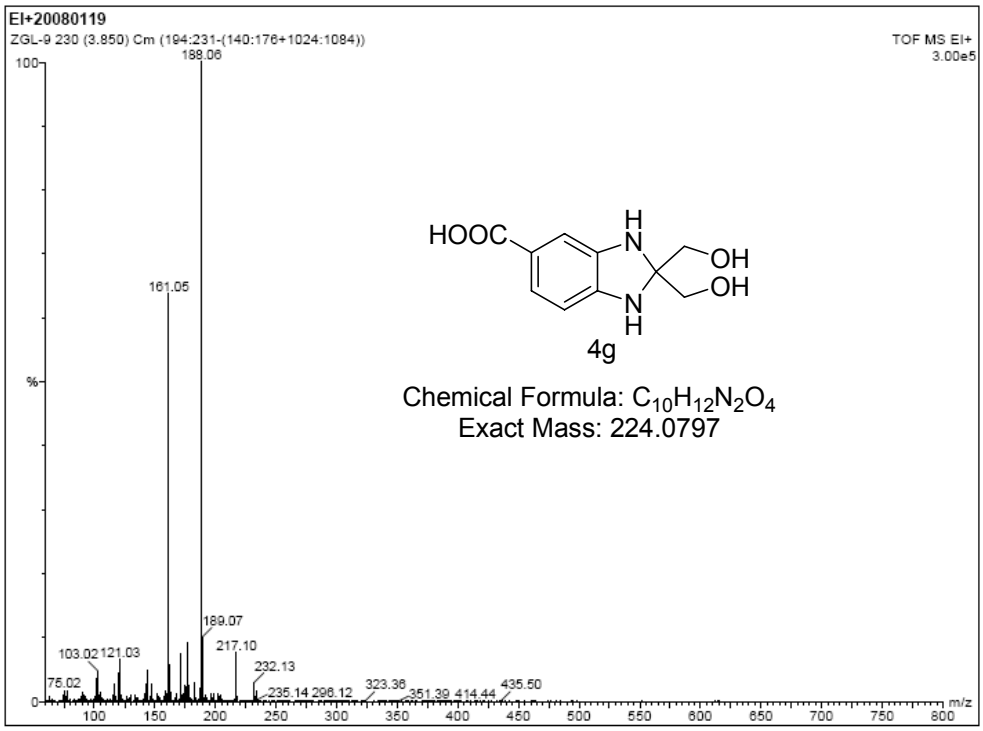

6.443
6.436
6.423
6.329
6.175
6.166
6.156
5.744
5.568
4.632
3.397
2.497
4e
1.88
0.94
0.95
0.91
2.00
3.69
ppm (t1)

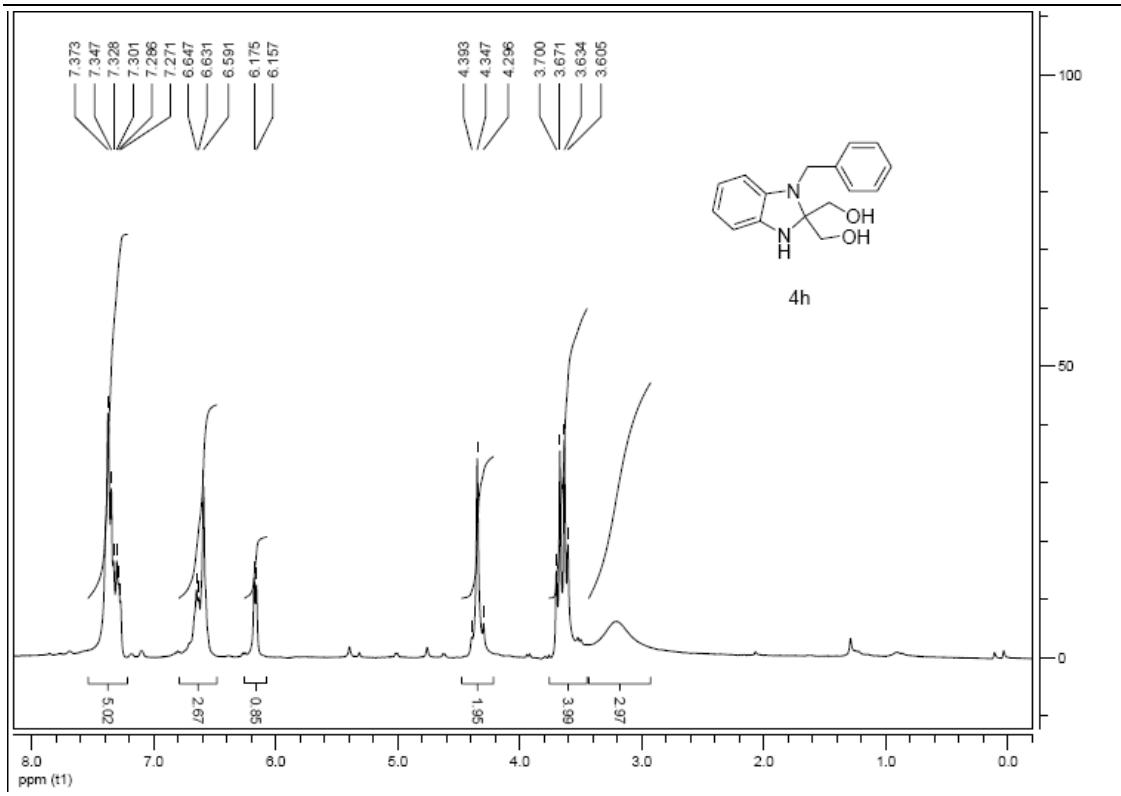

142.993
140.292
119.228
108.687
108.436
107.370
83.782
63.765
40.120
39.912
39.704
39.495
39.287
39.079
38.870
H
N
OH
OH
N
H
Br
4e
250
200
150
100
50
0
150
100
50
0
ppm (t1)

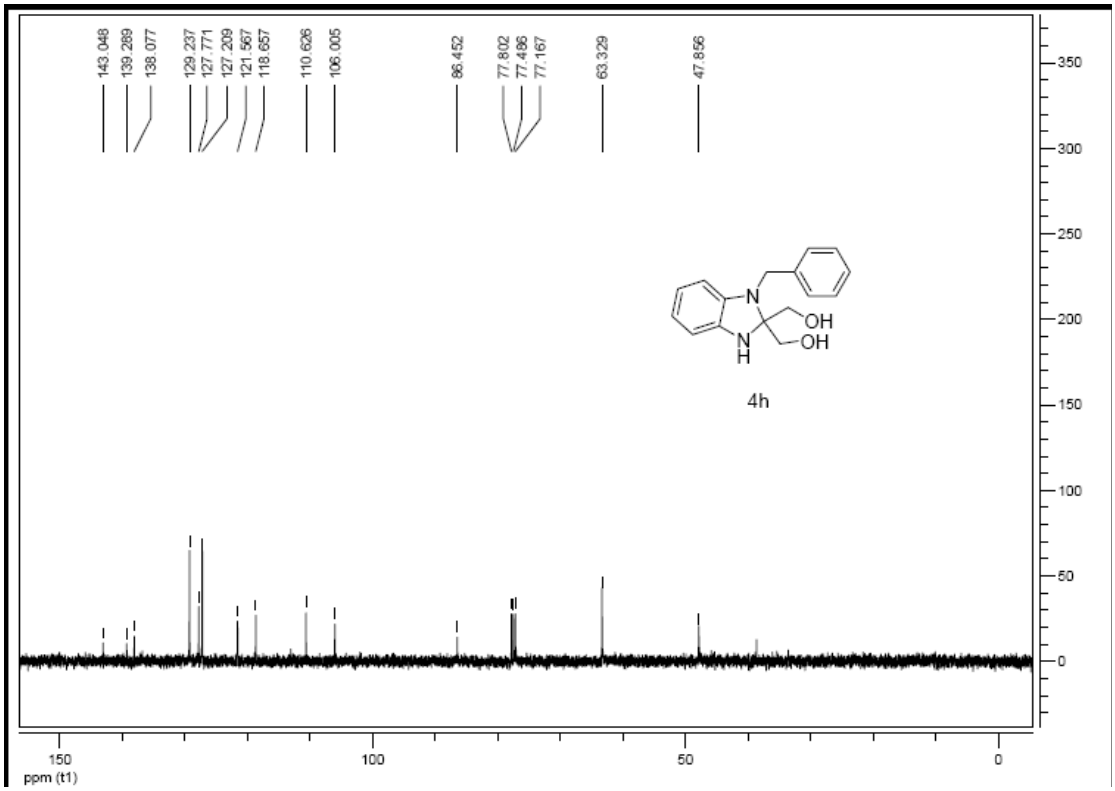

EI+20080109
ZGL-5 75 (1.251) Cm (68:75-(10:20+317:327))
TOF MS EI+
820
100
%
0
223.9809
221.9834
196.9711
208.9746
228.9810
153.9465
155.9498
195.9749
116.0446
117.0482
182.9538
75.0224
102.0363
142.0613
157.9424
235.9499
257.9987
70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260
m/z
H
N
OH
OH
N
H
Br
4e
Chemical Formula: C9H11BrN2O2
Exact Mass: 258.0004

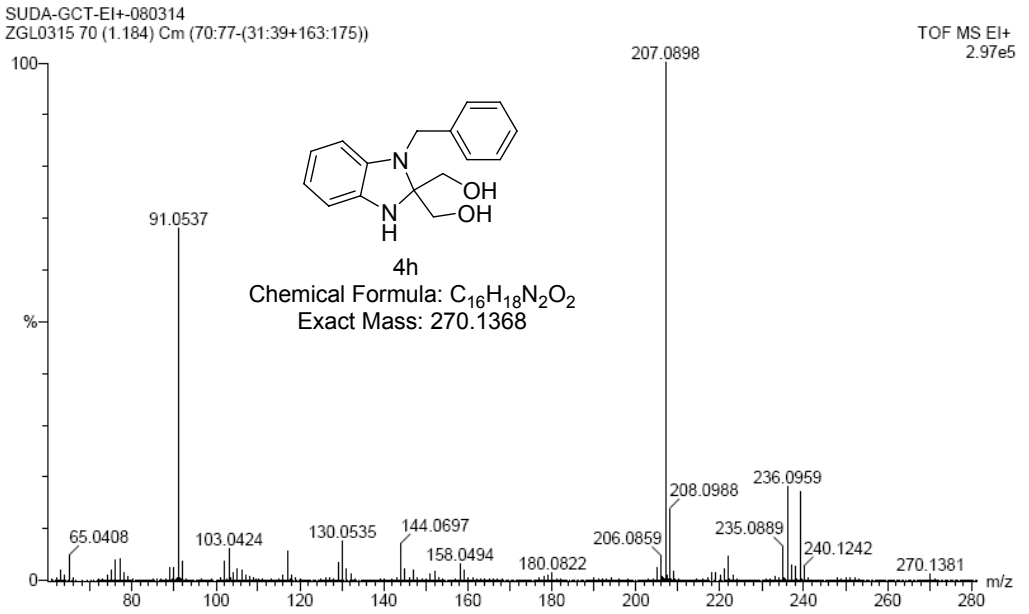

7.128
7.109
6.763
6.292
6.229
6.210
5.602
4.681
3.448
2.499
2.485
HOOC
OH
OH
4g
0.73
1.00
0.90
0.84
1.85
4.00
ppm (t1)

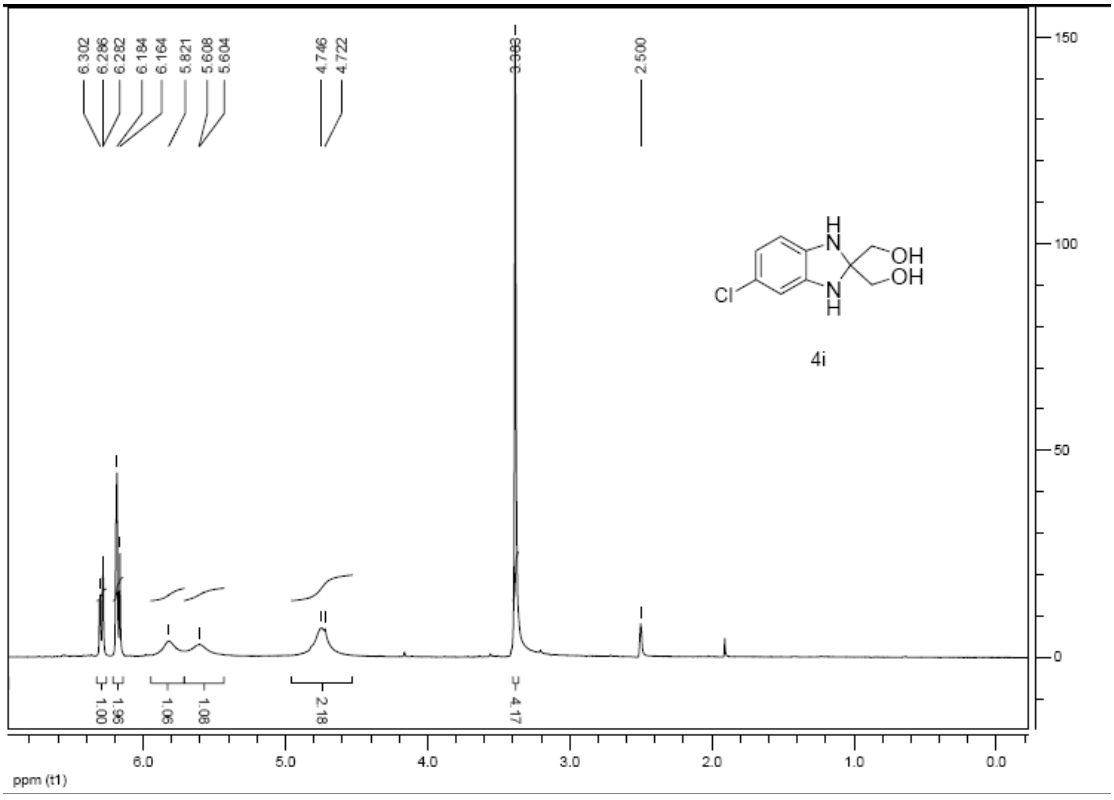

167.819
145.789
140.395
122.476
118.851
106.512
104.108
83.842
63.949
39.917
39.707
39.498
39.290
39.080
HOOC
OH
OH
4g
ppm (t1)

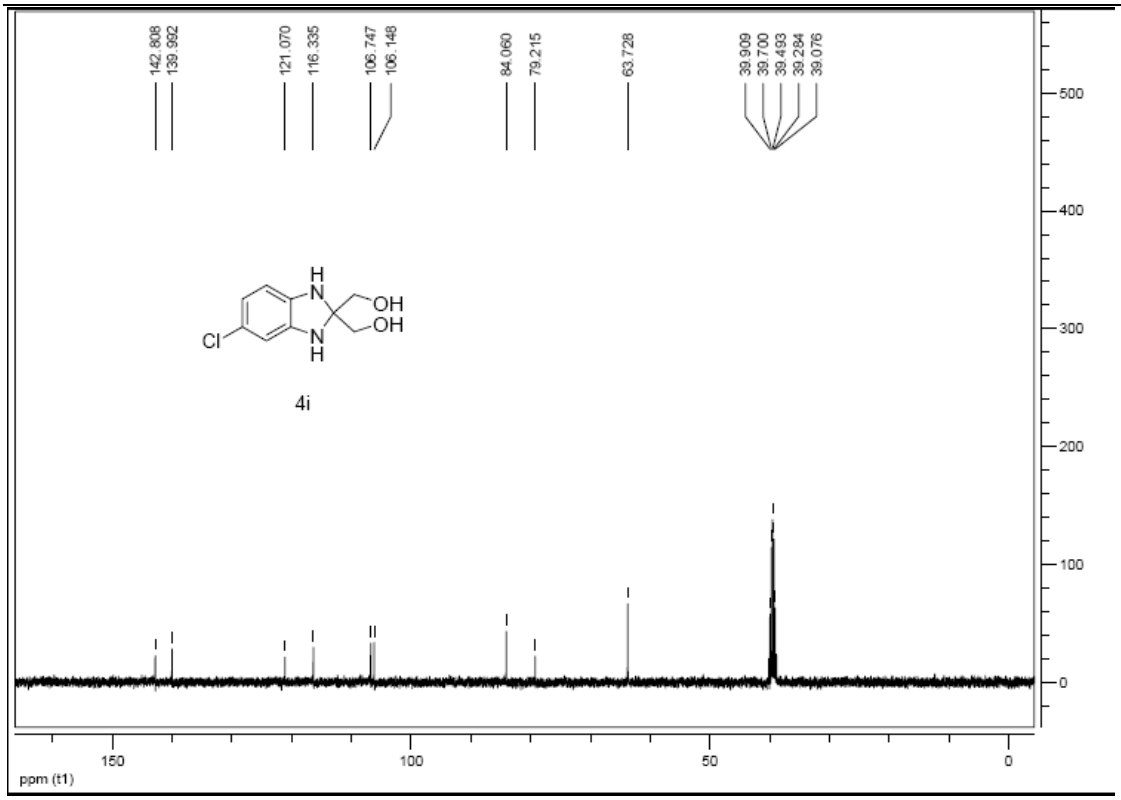

EI+20080119
ZGL-9 230 (3.850) Cm (194:231-(140:176+1024:1084))
TOF MS EI+
3.00e5
188.06
161.05
189.07
217.10
232.13
103.02
121.03
75.02
235.14
296.12
323.36
351.39
414.44
435.50
%
m/z
HOOC
H
N
OH
OH
N
H
4g
Chemical Formula: $C_{10}H_{12}N_2O_4$
Exact Mass: 224.0797

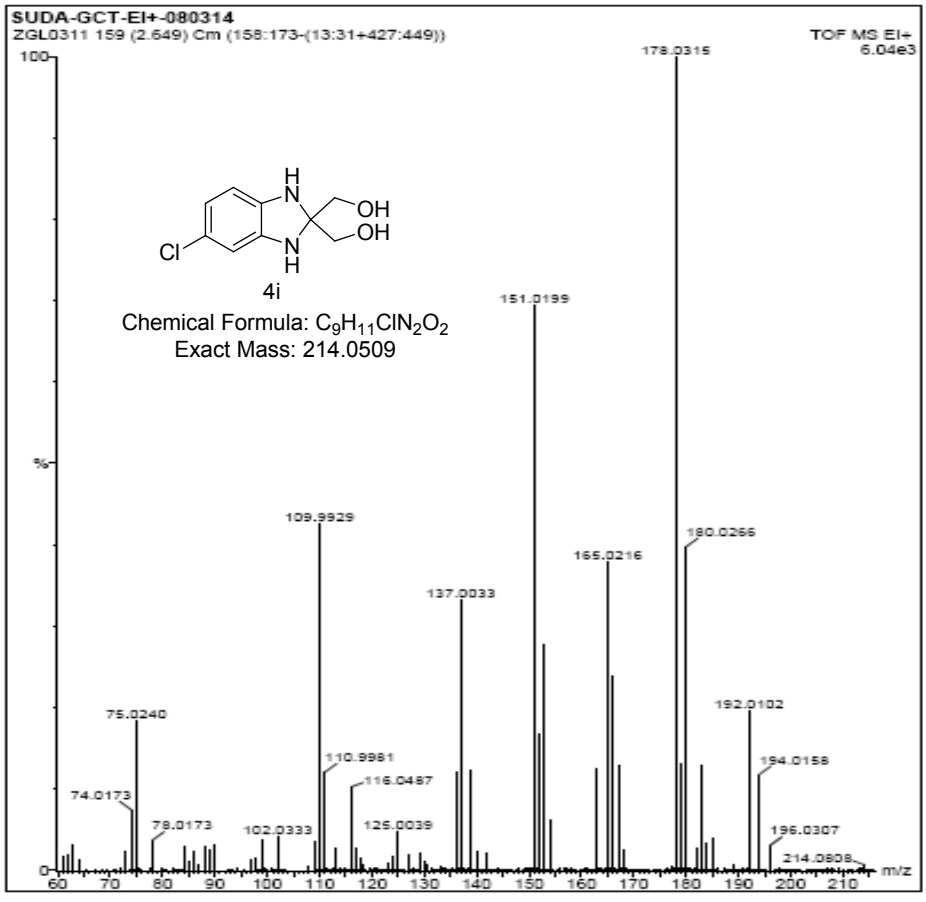

7.373
7.347
7.328
7.301
7.286
7.271
6.647
6.631
6.591
6.175
6.157
4.393
4.347
4.296
3.700
3.671
3.634
3.605
OH
OH
N
H
4h
5.02
2.67
0.86
1.96
3.99
2.97
ppm (t1)


143.048
139.289
138.077
129.237
127.771
127.209
121.567
118.657
110.626
106.005
86.452
77.802
77.486
77.167
63.329
47.856
OH
OH
N
H
4h
ppm (t1)

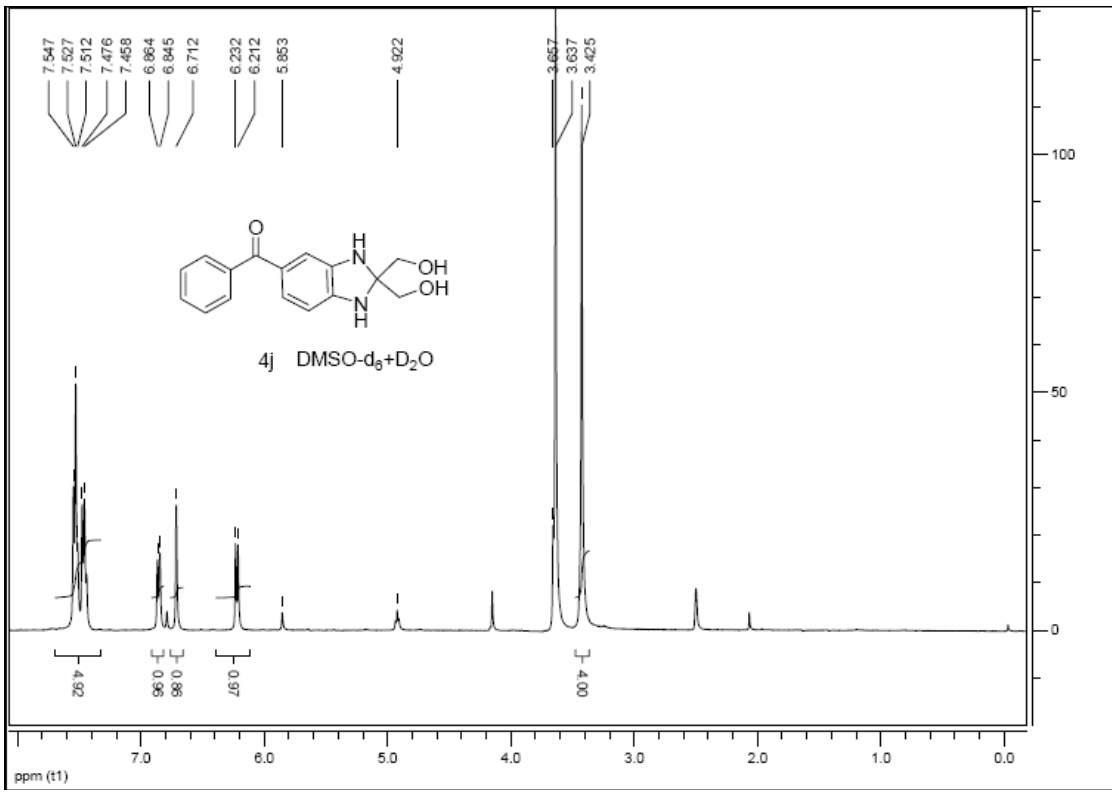

SUDA-GCT-EI+-080314
ZGL0315 70 (1.184) Cm (70:77-(31:39+163:175))
TOF MS EI+
2.97e5
207.0898
91.0537
4h
Chemical Formula: C16H18N2O2
Exact Mass: 270.1368
65.0408
103.0424
130.0535
144.0697
158.0494
180.0822
206.0859
208.0988
235.0889
236.0959
240.1242
270.1381
m/z

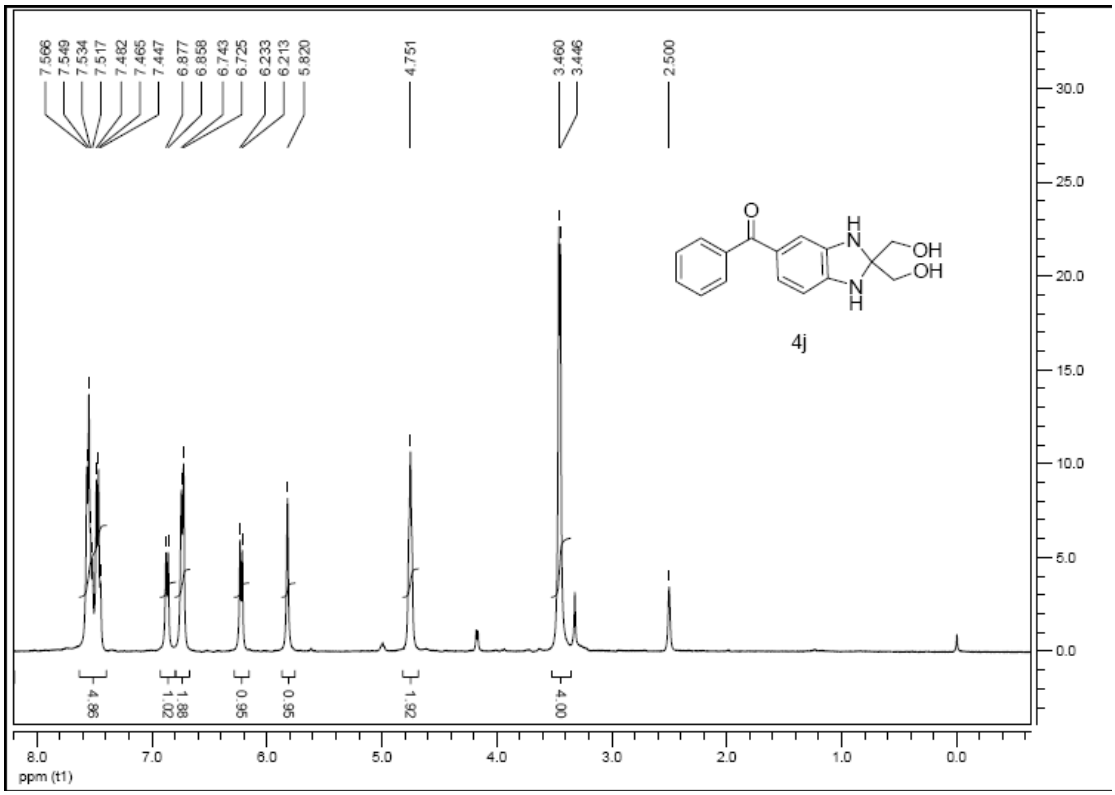

6.302
6.286
6.282
6.184
6.164
5.821
5.608
5.604
4.746
4.722
2.500
4i
ppm (t1)

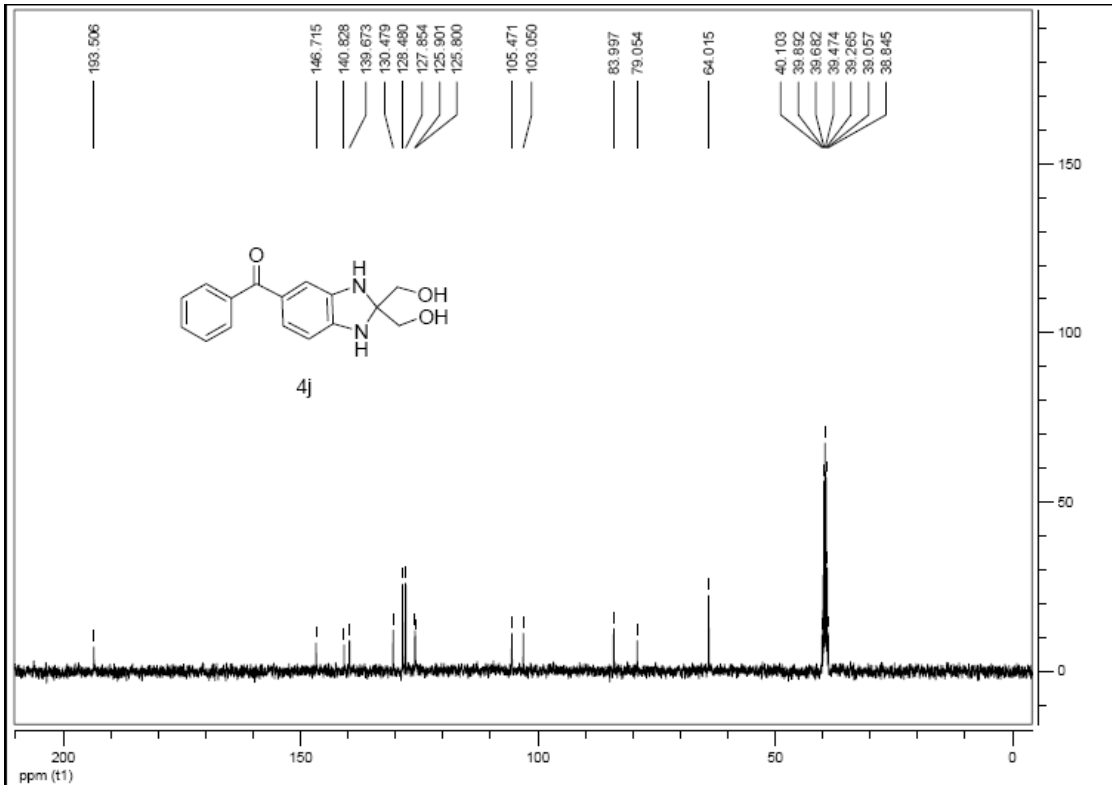

142.808
139.992
121.070
116.335
106.747
106.148
84.060
79.215
63.728
39.909
39.700
39.493
39.284
39.076
H
N
OH
OH
Cl
N
H
4i
500
400
300
200
100
0
150
100
50
0
ppm (t1)

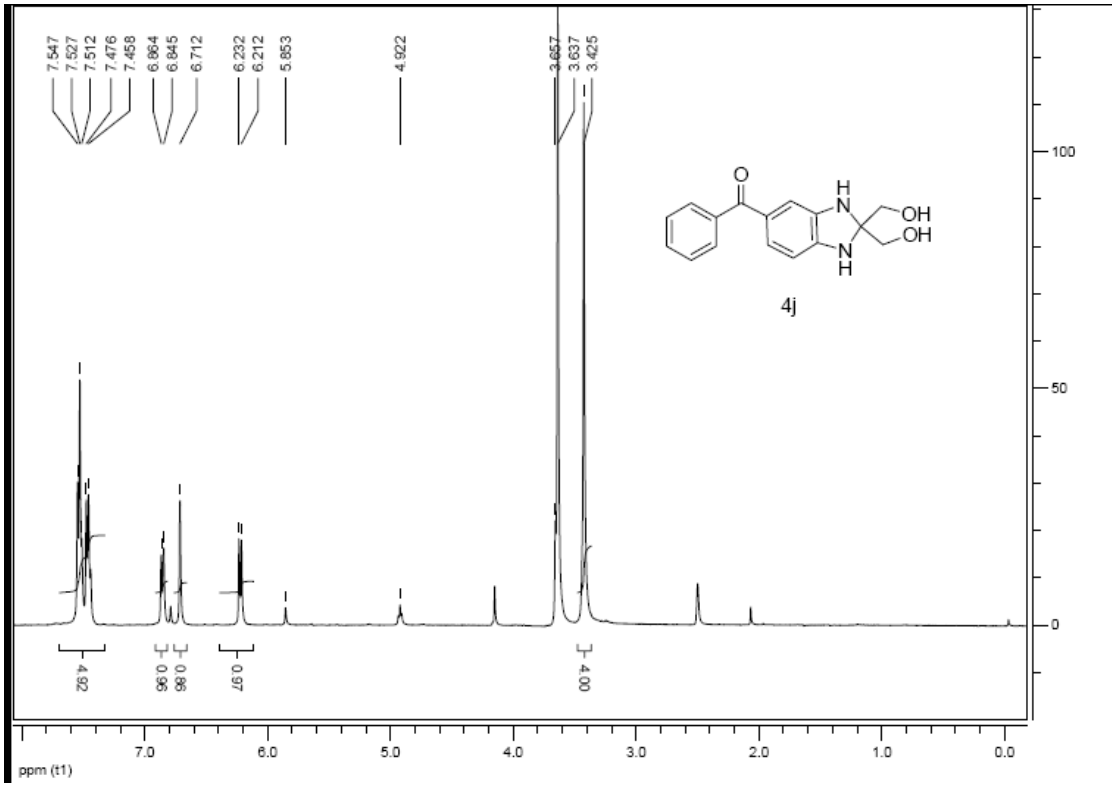

SUDA-GCT-EI+-080314
ZGL0311 159 (2.649) Cm (158:173-(13:31+427:449))
TOF MS EI+
6.04e3
100
%
0
178.0315
151.0199
109.9929
180.0266
165.0216
137.0033
192.0102
75.0240
110.9981
116.0487
194.0158
74.0173
78.0173
102.0333
125.0039
196.0307
214.0808
m/z
60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210
H
N
OH
OH
Cl
N
H
4i
Chemical Formula: C9H11ClN2O2
Exact Mass: 214.0509

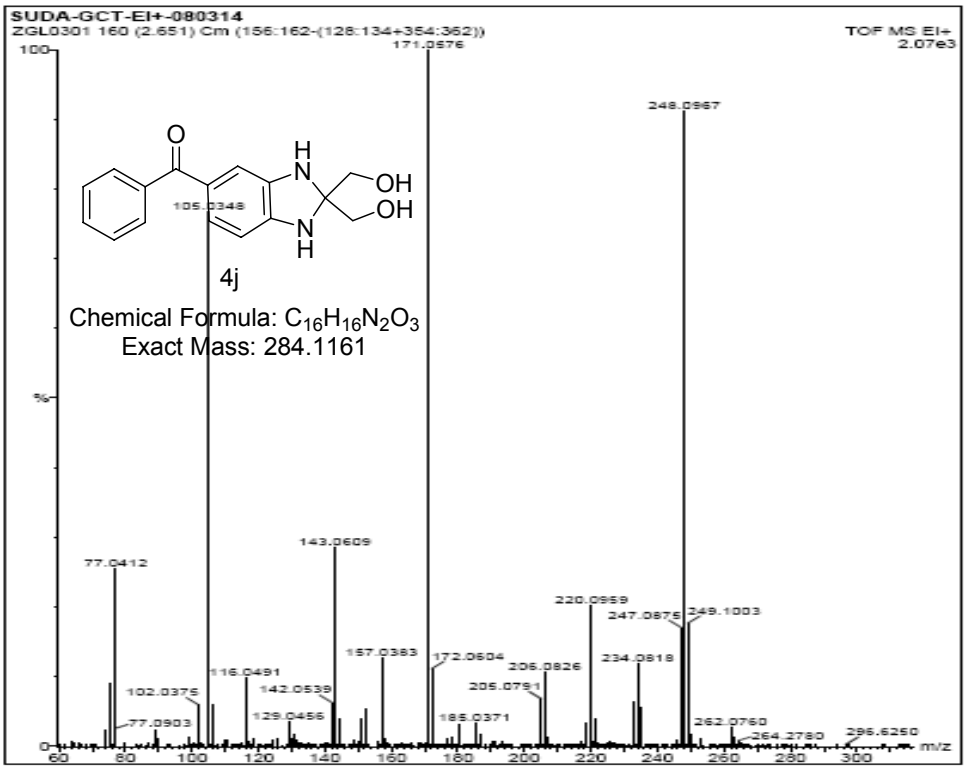
c

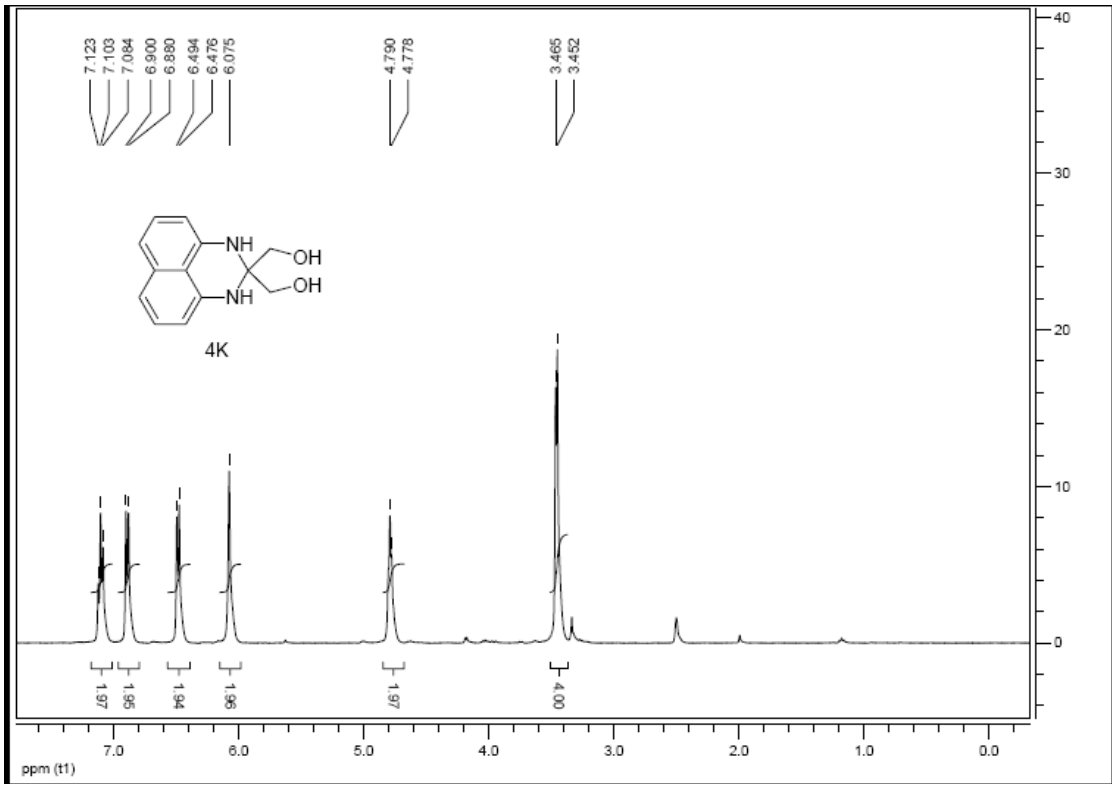

7.547
7.527
7.512
7.476
7.458
6.864
6.845
6.712
6.232
6.212
5.853
4.922
3.657
3.637
3.425
4j DMSO-d6+D2O
4.92
0.96
0.86
0.97
4.00
ppm (t1)

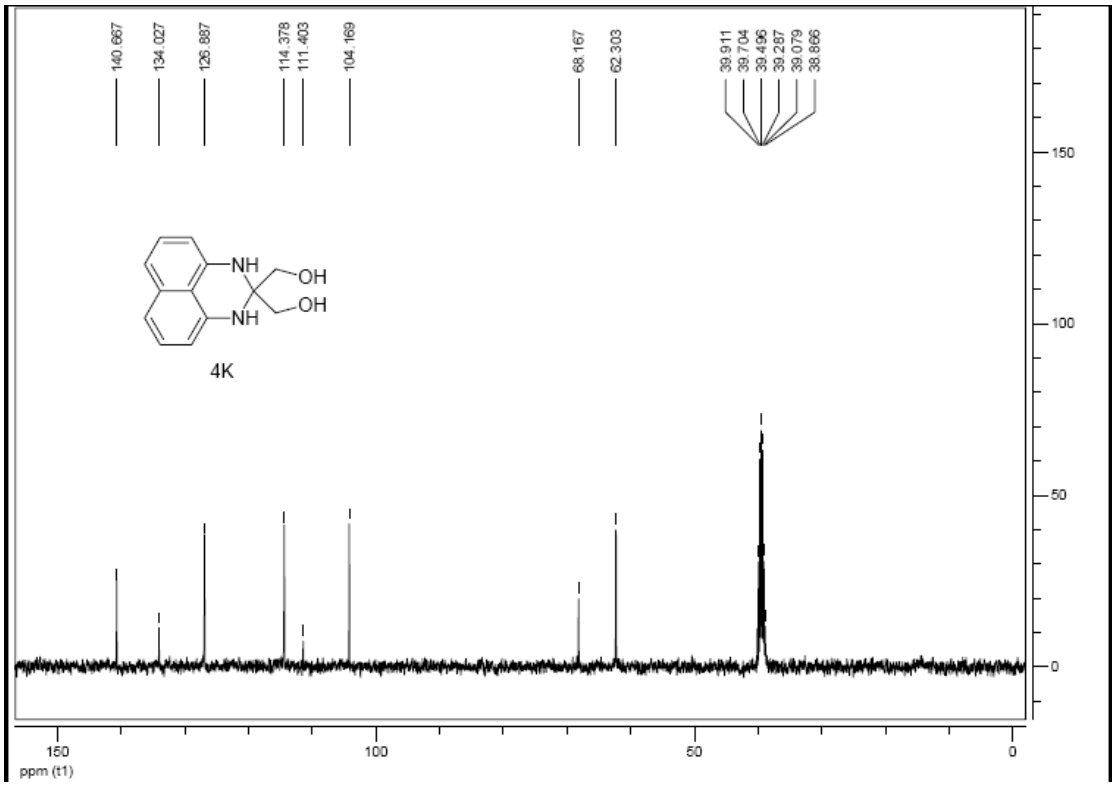

7.566
7.549
7.534
7.517
7.482
7.465
7.447
6.877
6.858
6.743
6.725
6.233
6.213
5.820
4.751
3.460
3.446
2.500
4j
4.86
1.02
0.88
0.96
0.96
1.92
4.00
ppm (t1)

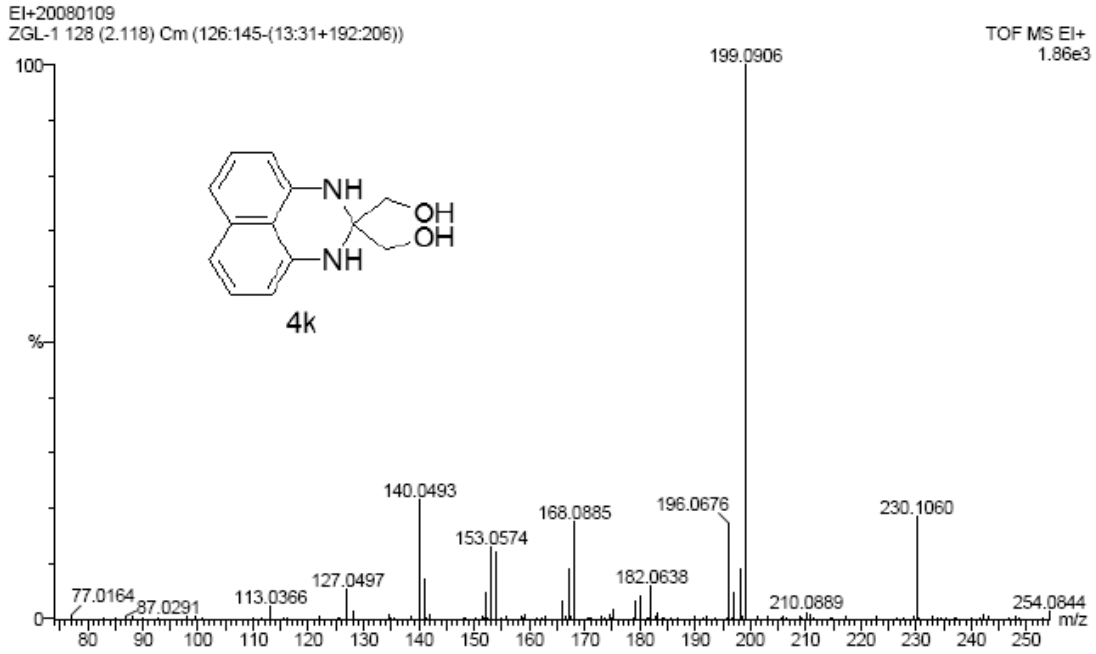

193.506
146.715
140.828
139.673
130.479
128.480
127.854
125.901
125.800
105.471
103.050
83.997
79.054
64.015
40.103
39.892
39.682
39.474
39.265
39.057
38.845
O
H
N
OH
OH
N
H
4j
150
100
50
0
200
150
100
50
0
ppm (t1)

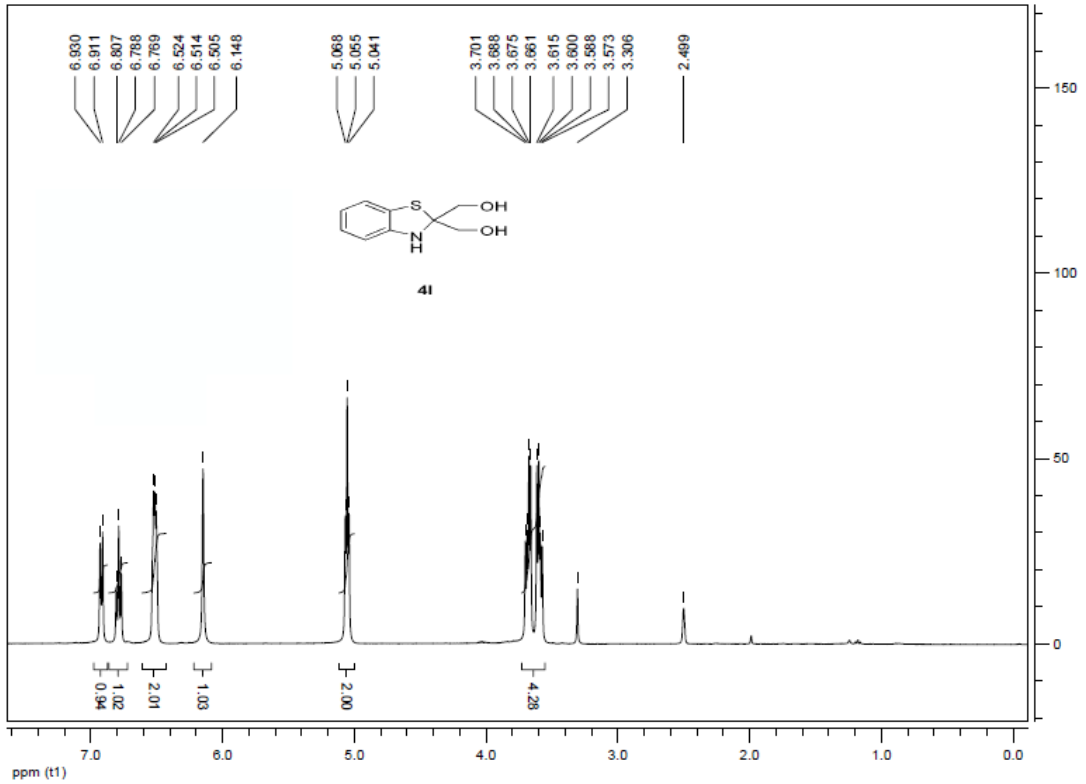

7.547
7.527
7.512
7.476
7.458
6.864
6.845
6.712
6.232
6.212
5.853
4.922
3.657
3.637
3.425
O
H
N
OH
OH
N
H
4j
100
50
0
4.92
0.96
0.98
0.97
4.00
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

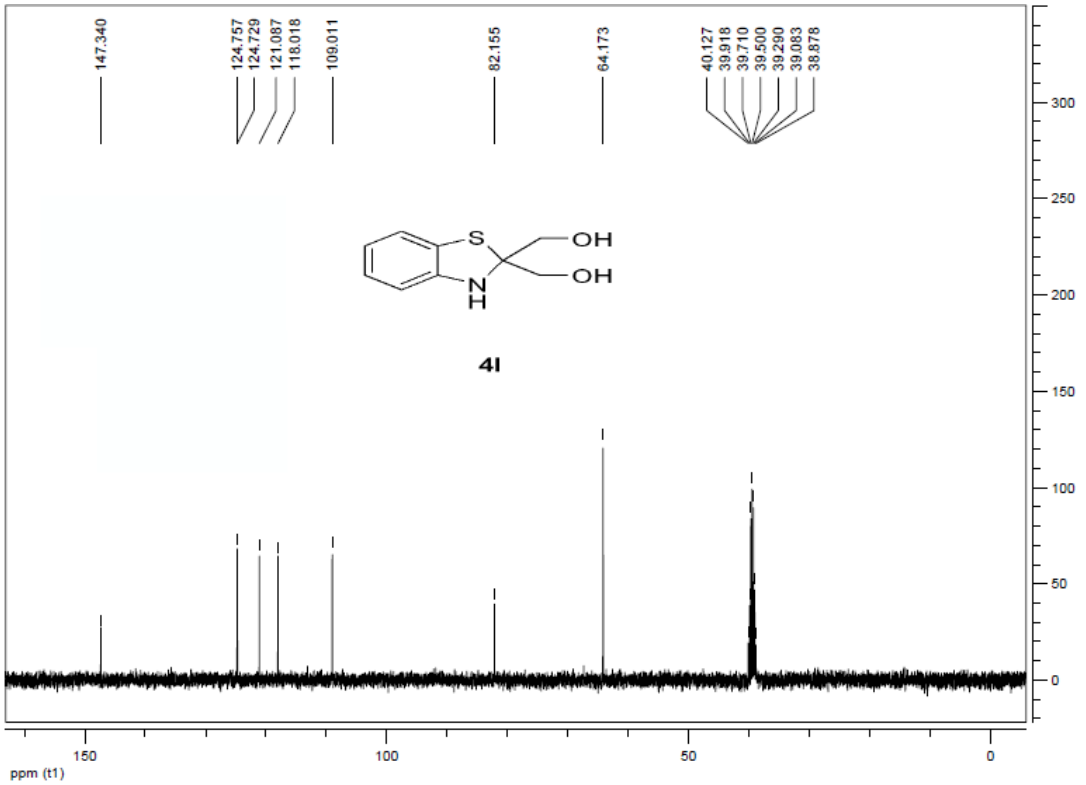

SUDA-GCT-EI+-080314
ZGL0301 160 (2.651) Cm (156:162-(128:134+354:362))
TOF MS EI+
2.07e3
100
%
0
171.0676
248.0967
105.0348
143.0609
77.0412
220.0959
247.0875
249.1003
157.0383
172.0604
206.0826
234.0818
116.0491
142.0539
205.0791
102.0375
129.0456
185.0371
77.0903
262.0760
264.2780
296.6250
60
60
100
120
140
160
180
200
220
240
260
280
300
m/z
O
H
N
OH
OH
N
H
4j
Chemical Formula: C16H16N2O3
Exact Mass: 284.1161

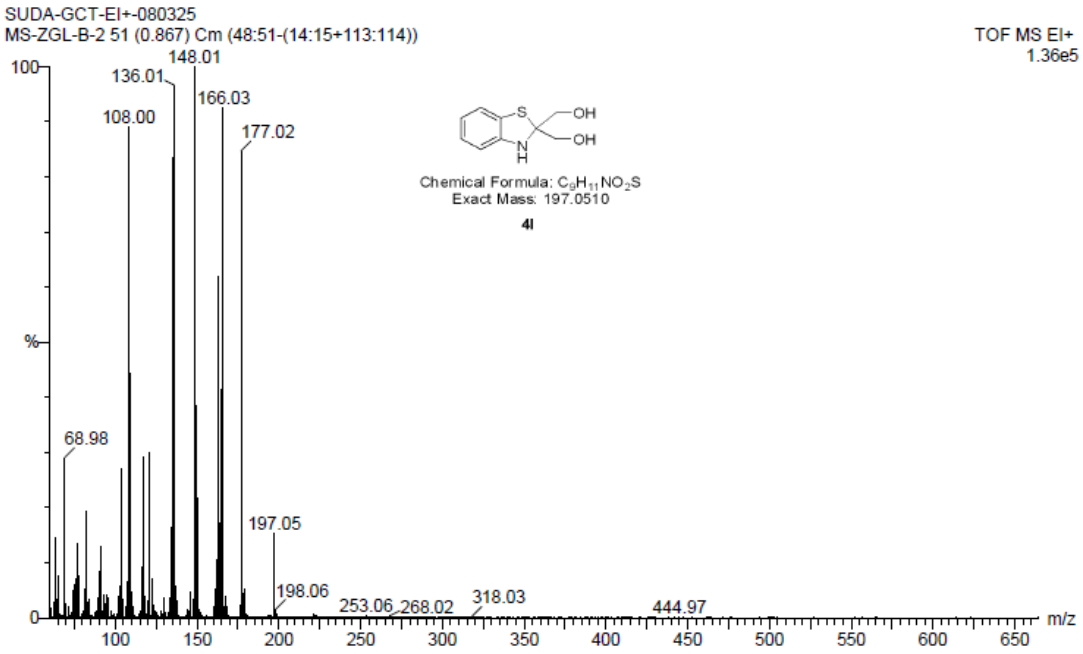

7.123
7.103
7.084
6.900
6.880
6.494
6.476
6.075
4.790
4.778
3.465
3.452
NH
OH
OH
NH
4K
1.97
1.96
1.94
1.96
1.97
4.00
ppm (t1)

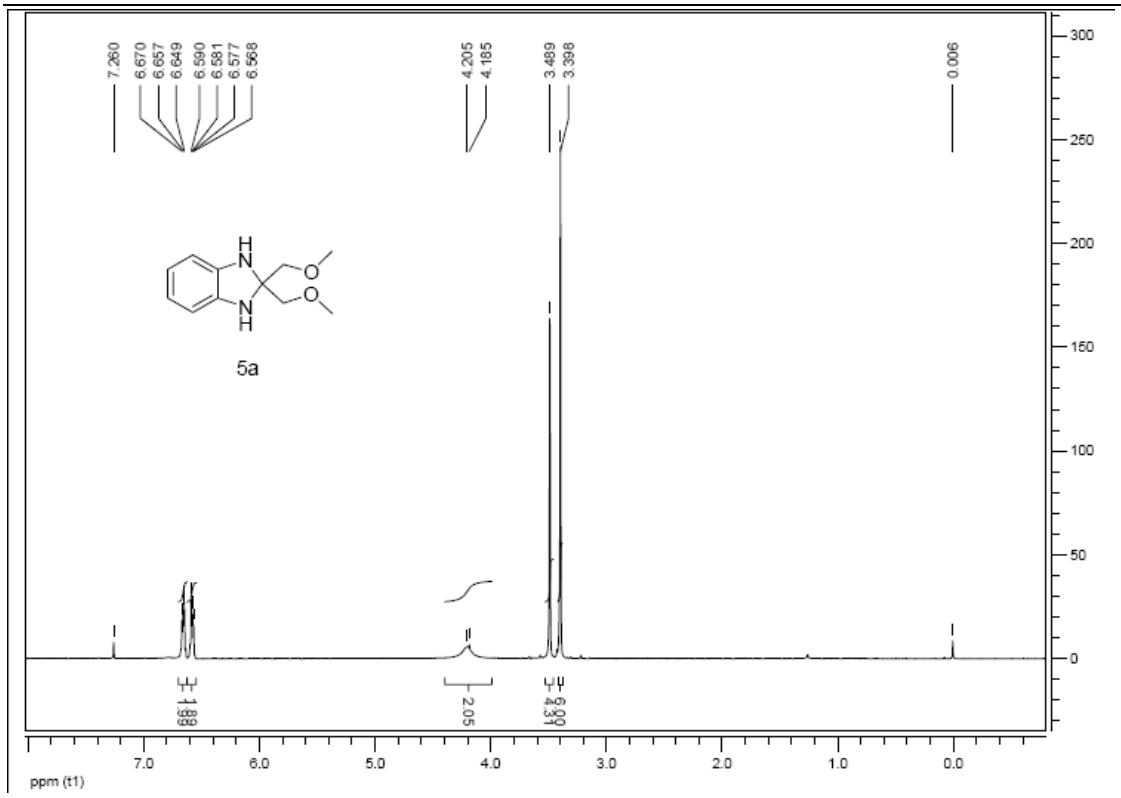

140.667
134.027
126.887
114.378
111.403
104.169
68.167
62.303
39.911
39.704
39.496
39.287
39.079
38.866
NH
OH
OH
NH
4K
ppm (t1)

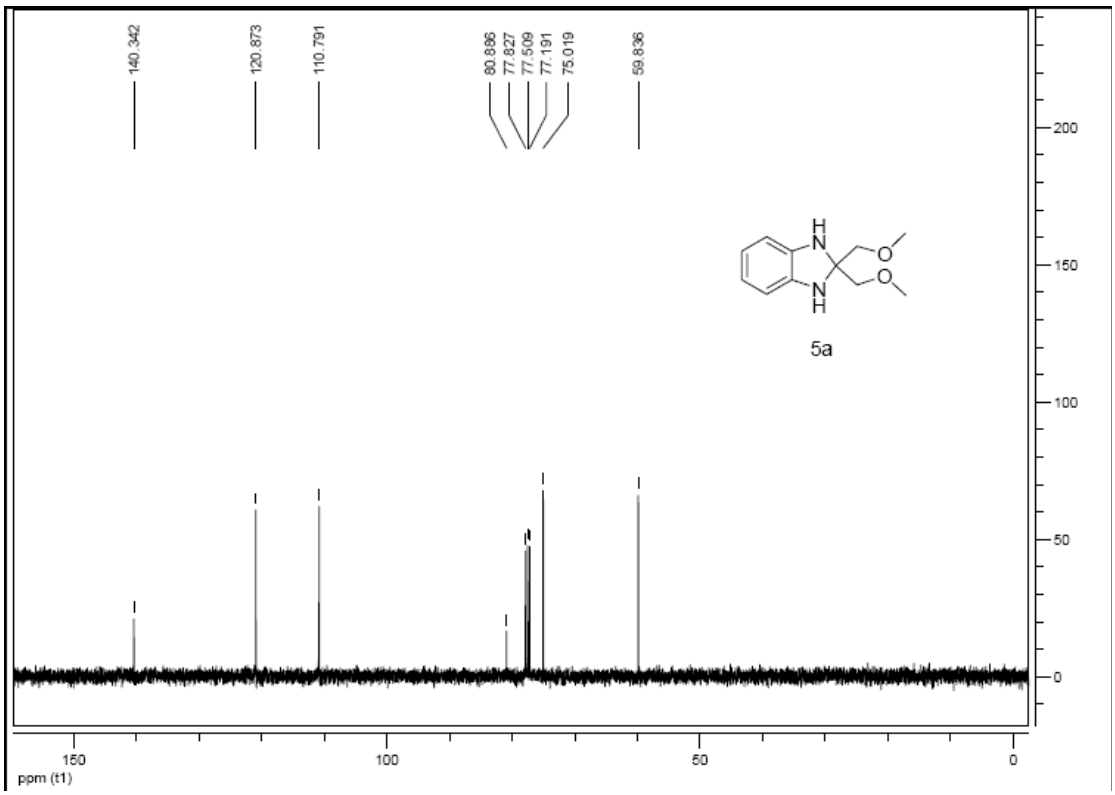

EI+20080109
ZGL-1 128 (2.118) Cm (126:145-(13:31+192:206))
TOF MS EI+
1.86e3
199.0906
NH
OH
OH
NH
4k
%
140.0493
168.0885
196.0676
230.1060
153.0574
182.0638
127.0497
77.0164
87.0291
113.0366
210.0889
254.0844
m/z

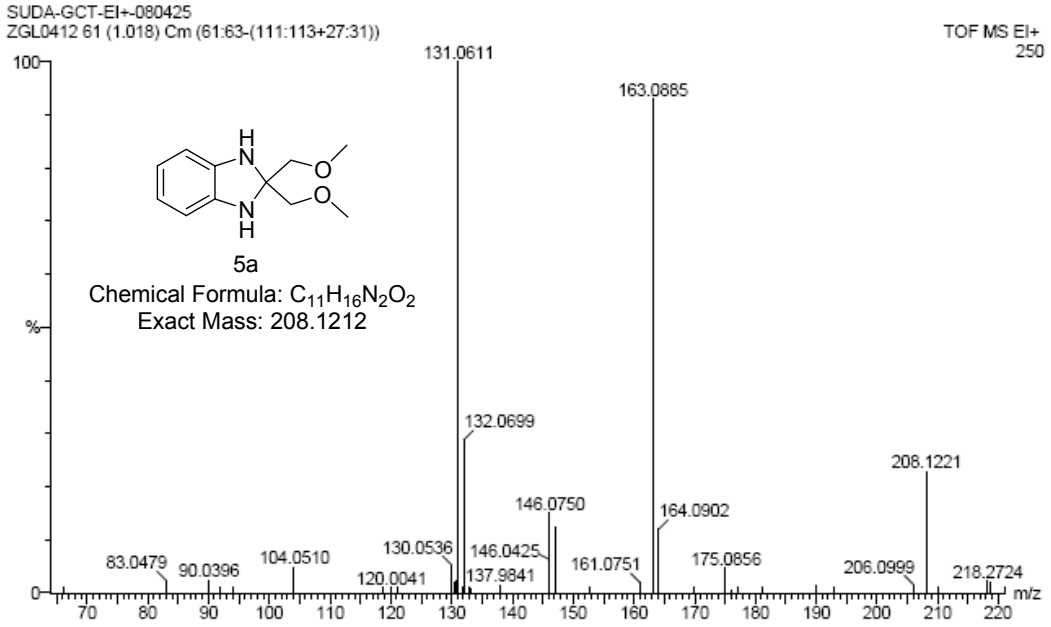

6.930
6.911
6.807
6.788
6.769
6.524
6.514
6.505
6.148
5.068
5.055
5.041
3.701
3.688
3.675
3.661
3.615
3.600
3.588
3.573
3.306
2.499
S
OH
N
H
OH
4l
0.94
1.20
2.01
1.03
2.00
4.28
ppm (t1)


147.340
124.757
124.729
121.087
118.018
109.011
82.155
64.173
40.127
39.918
39.710
39.500
39.290
39.083
38.878
S
OH
OH
N
H
4l
300
250
200
150
100
50
0
150
100
50
0
ppm (t1)


SUDA-GCT-EI+-080325
MS-ZGL-B-2 51 (0.867) Cm (48:51-(14:15+113:114))
TOF MS EI+
1.36e5
100
%
0
148.01
136.01
166.03
108.00
177.02
68.98
197.05
198.06
253.06
268.02
318.03
444.97
100
150
200
250
300
350
400
450
500
550
600
650
m/z
S
OH
OH
N
H
Chemical Formula: C9H11NO2S
Exact Mass: 197.0510
4l


7.260
6.670
6.657
6.649
6.590
6.581
6.577
6.568
4.205
4.185
3.489
3.398
0.006
5a
1.99
2.05
6.90
4.31
ppm (t1)

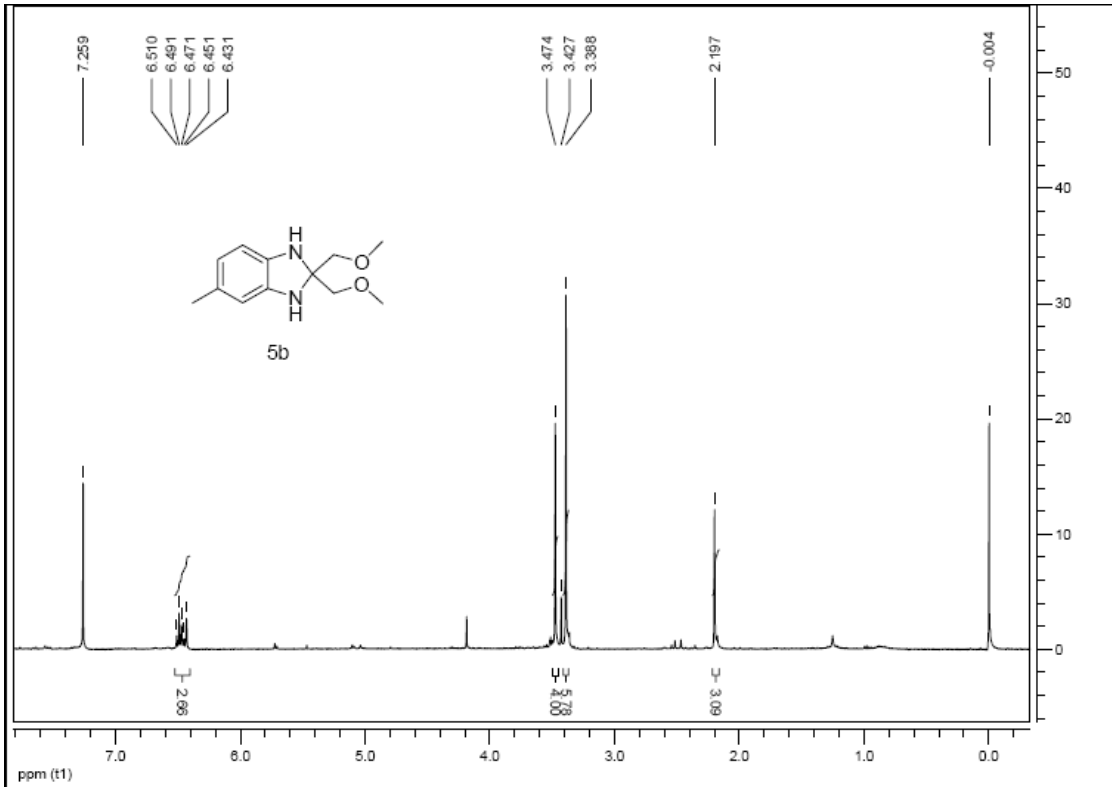

140.342
120.873
110.791
80.886
77.827
77.509
77.191
75.019
59.836
5a
ppm (t1)



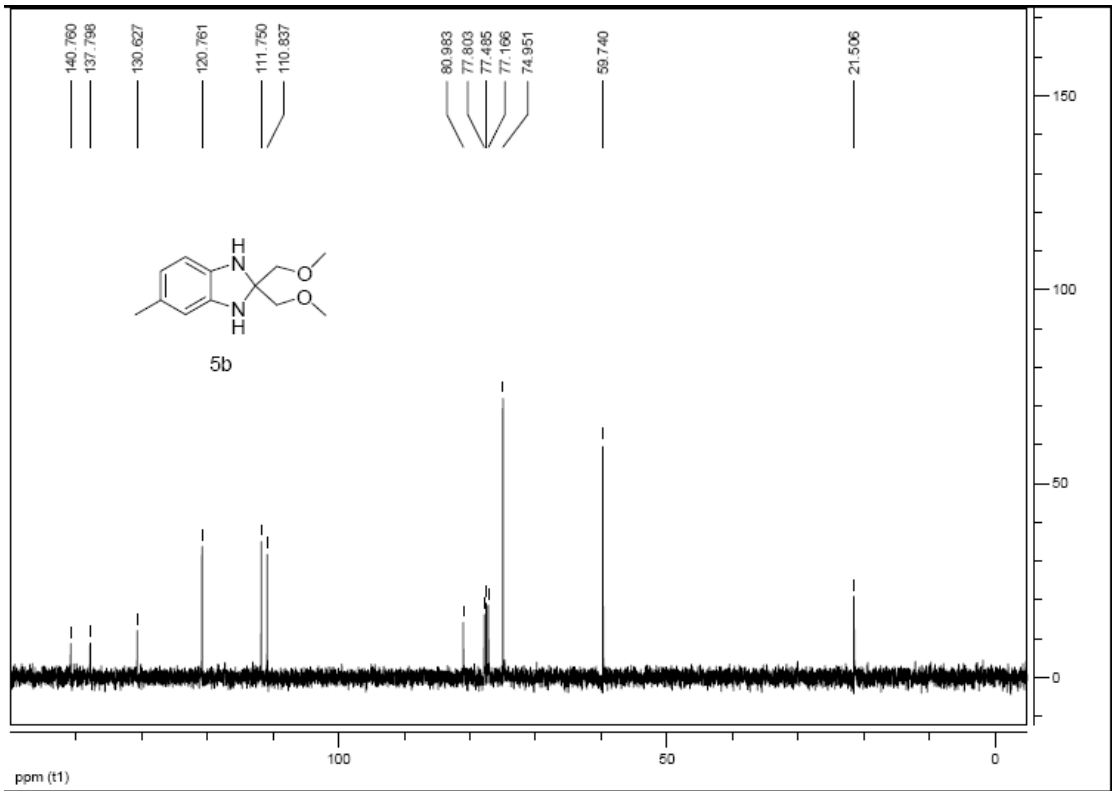
5a

Chemical Formula: $C_{11}H_{16}N_2O_2$

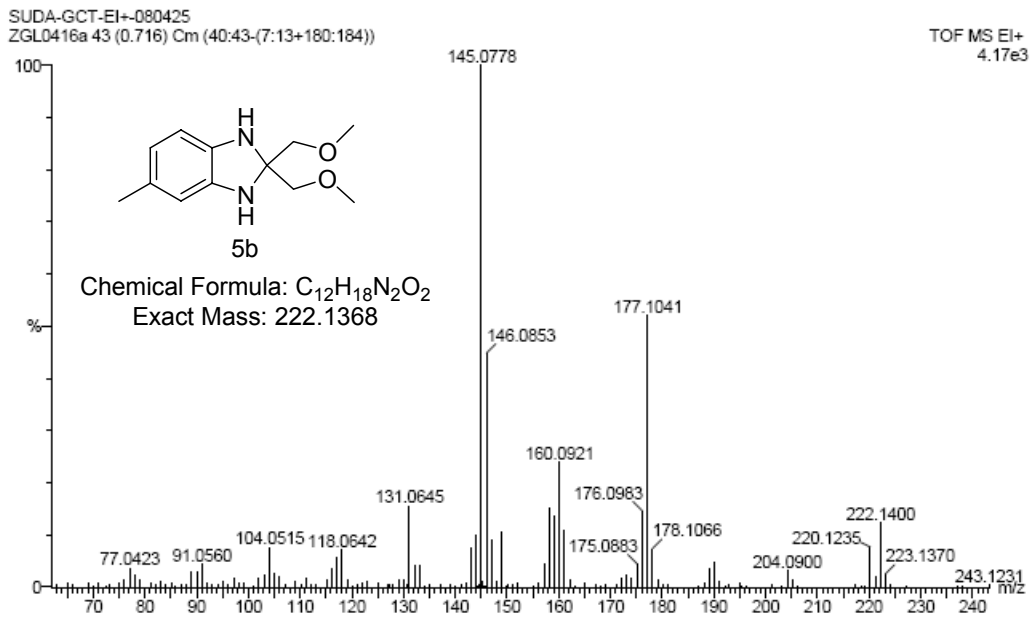
Exact Mass: 208.1212



5b

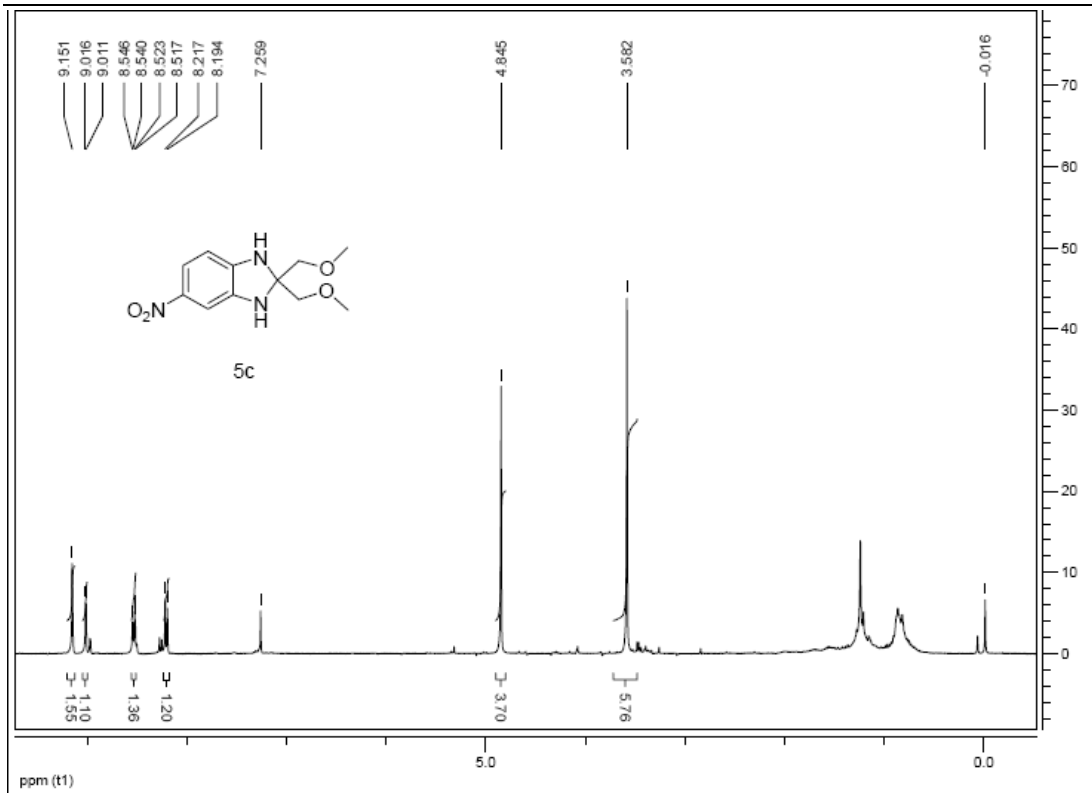


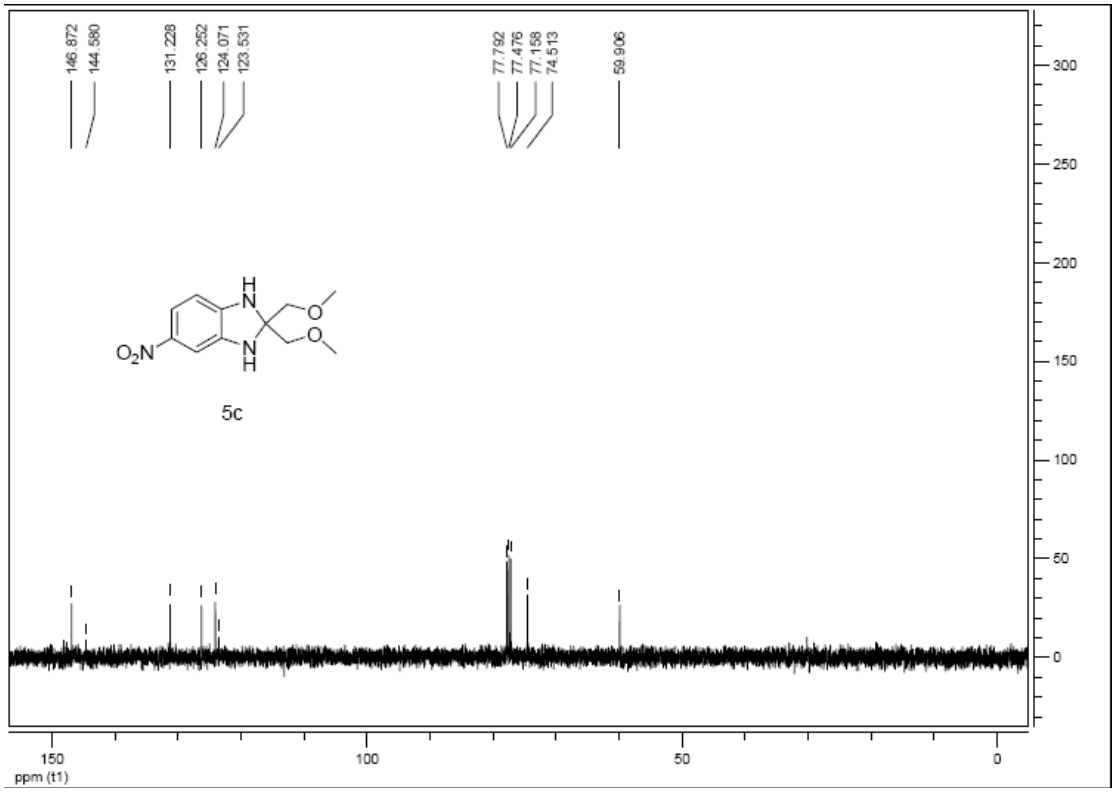
5b

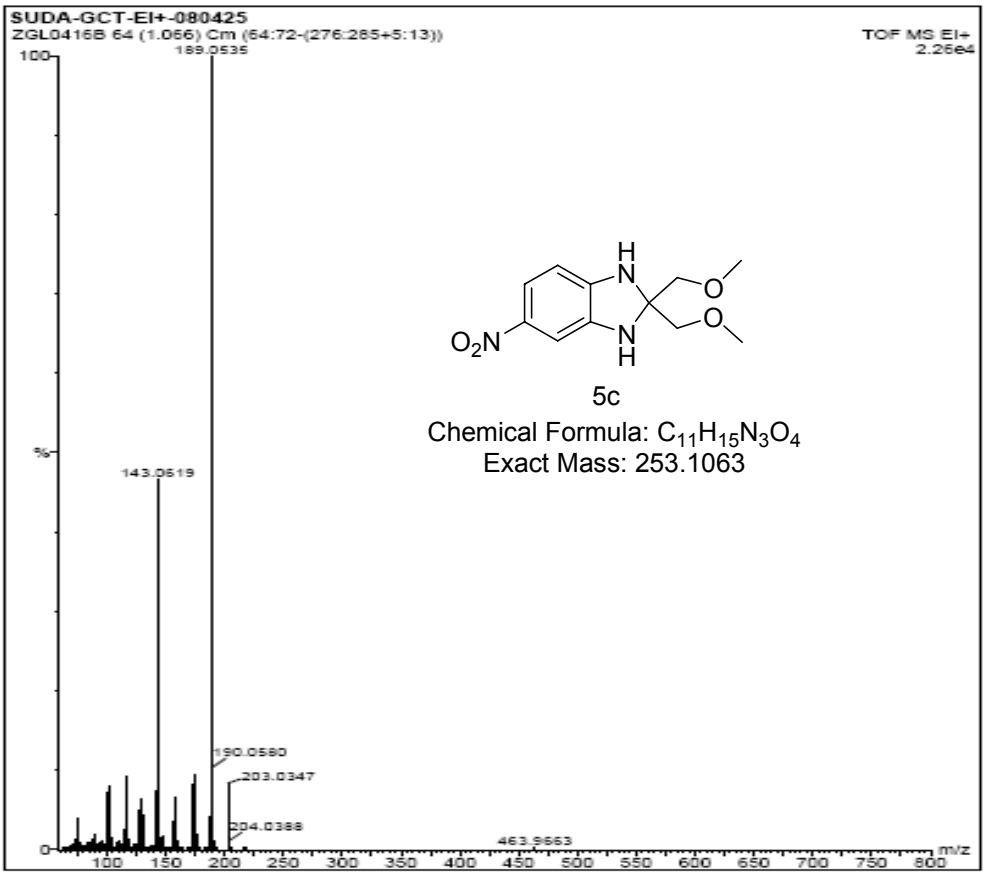


5b

Chemical Formula: $C_{12}H_{18}N_2O_2$
Exact Mass: 222.1368


9.151
9.016
9.011
8.546
8.540
8.523
8.517
8.217
8.194
7.259
4.845
3.582
-0.016
5c
1.55
1.10
1.36
1.20
3.70
5.76
5.0
0.0
ppm (t1)

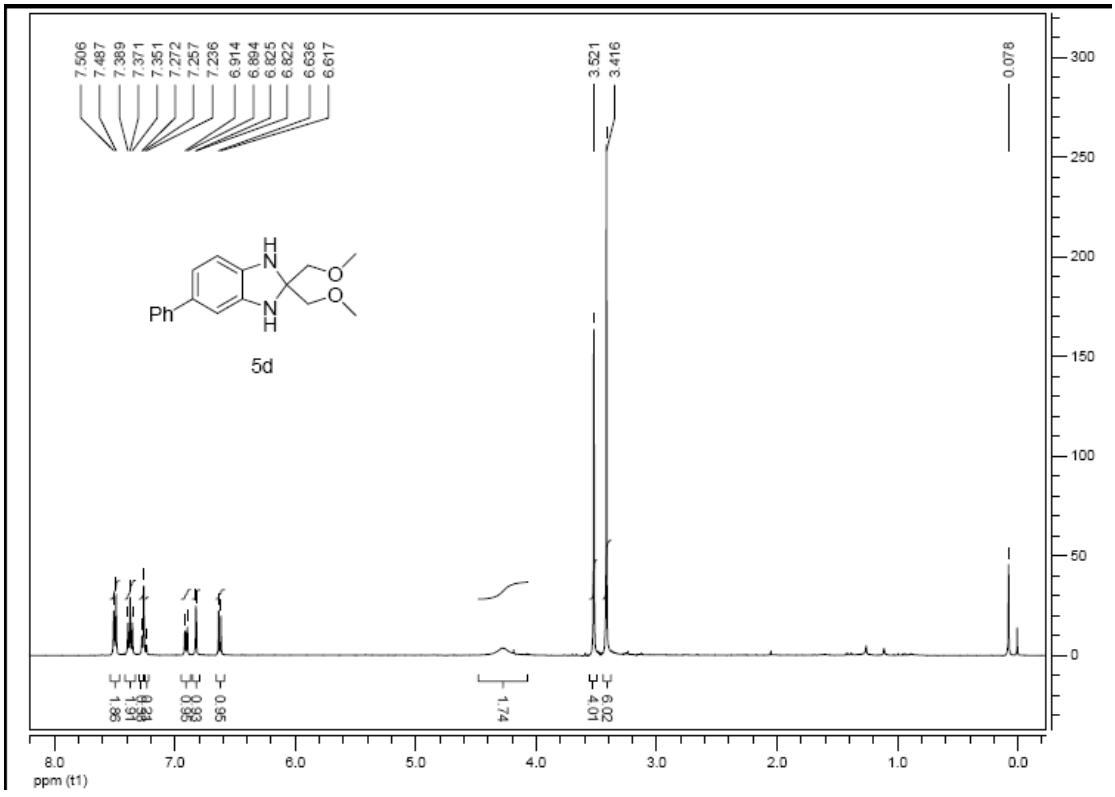

146.872
144.580
131.228
126.252
124.071
123.531
77.792
77.476
77.158
74.513
59.906
5c
150
100
50
0
ppm (t1)

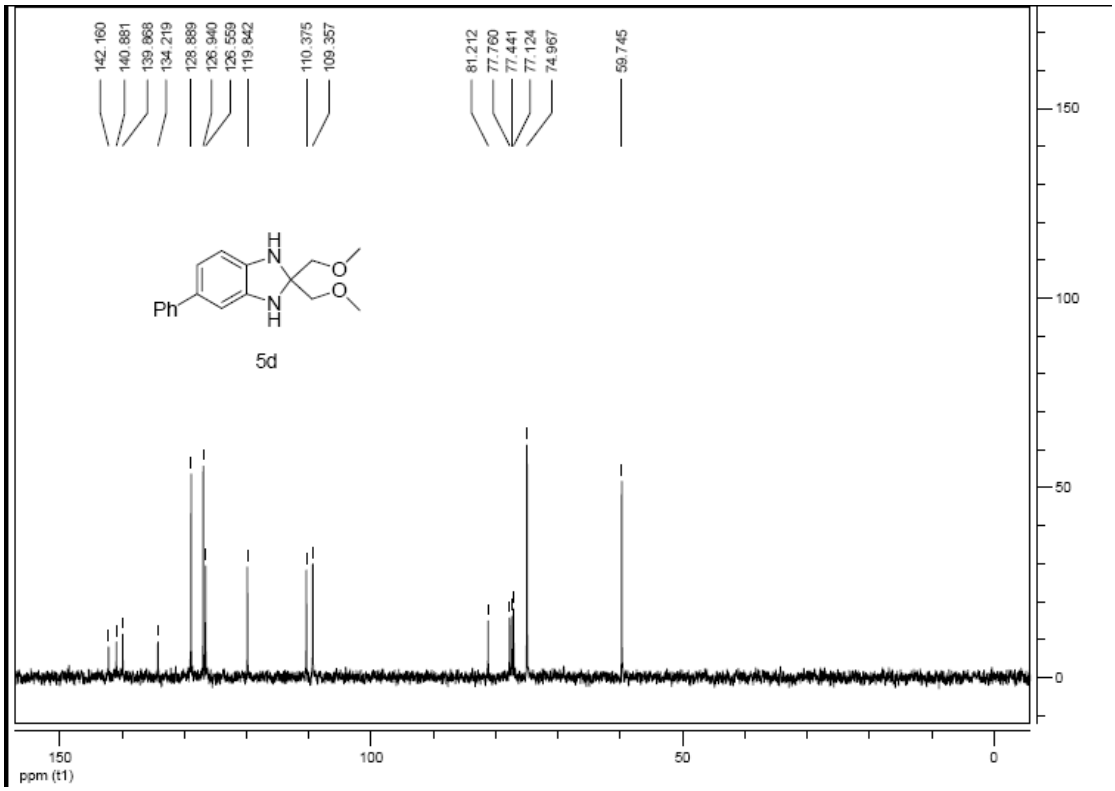


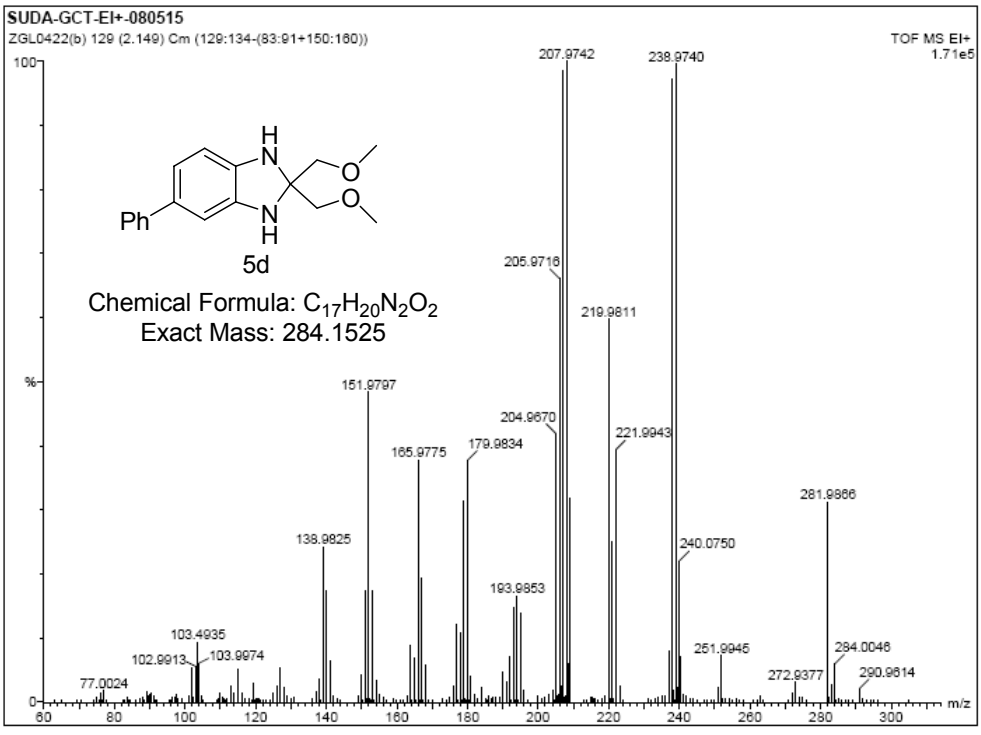
5c

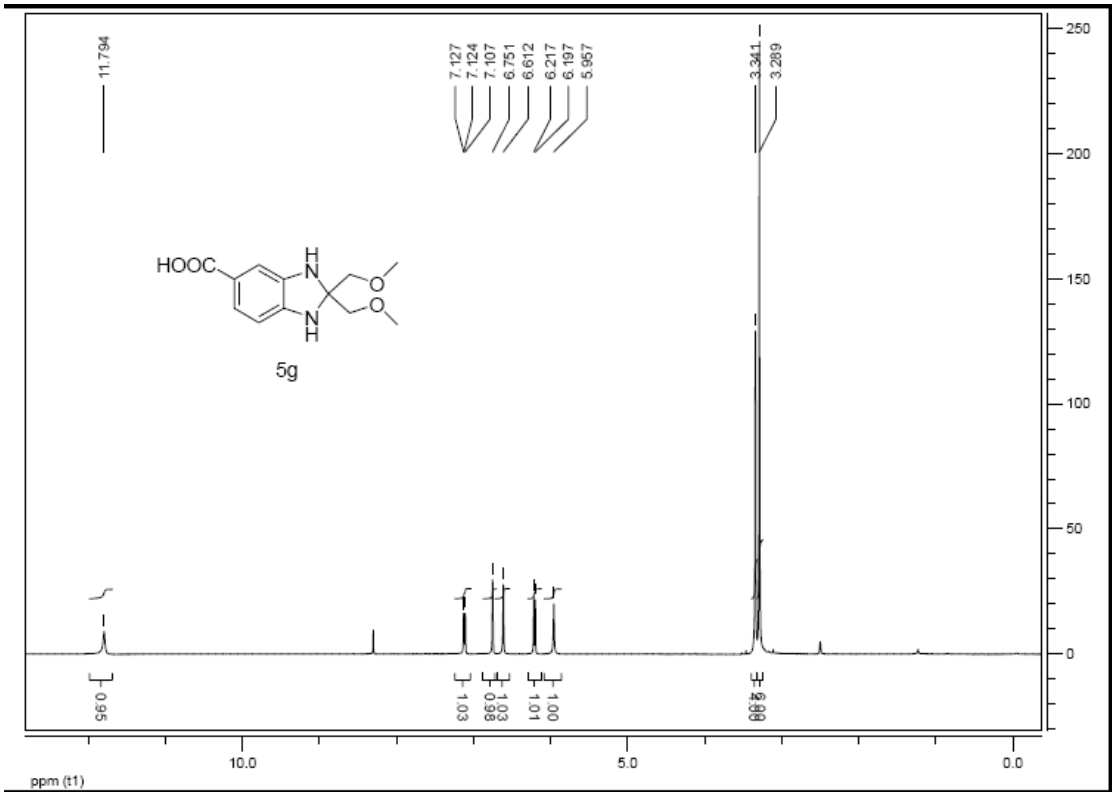
Chemical Formula: $C_{11}H_{15}N_3O_4$

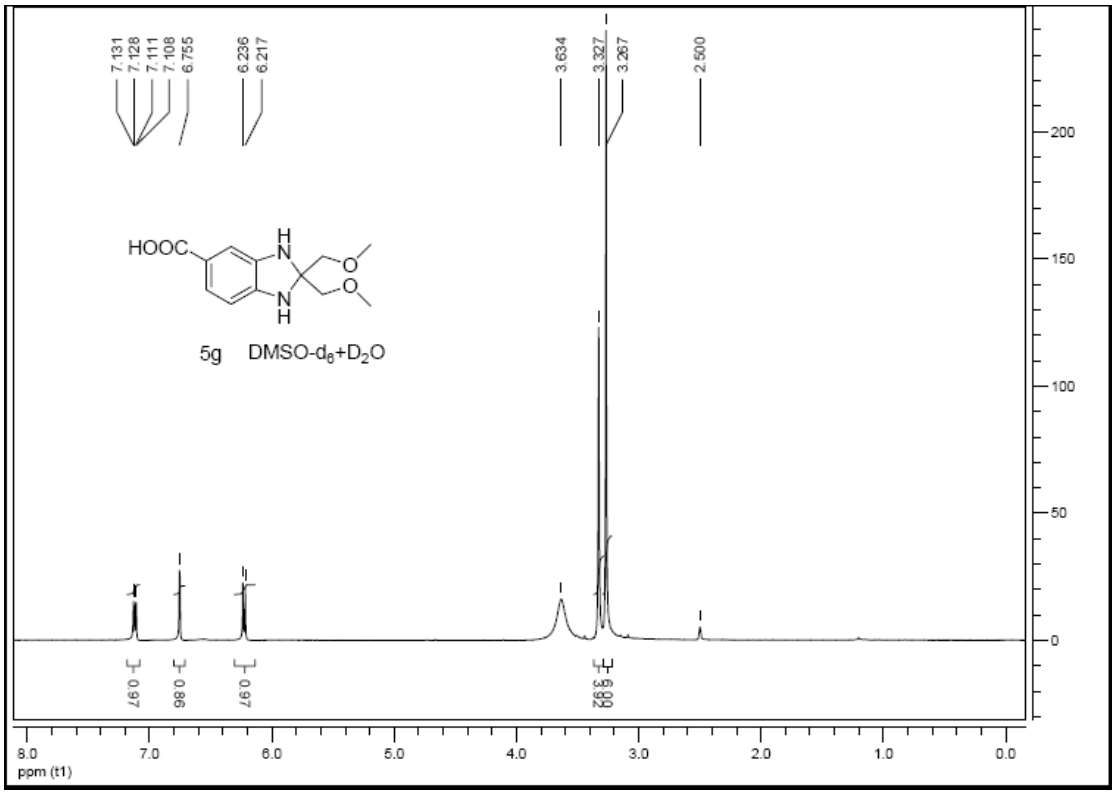
Exact Mass: 253.1063

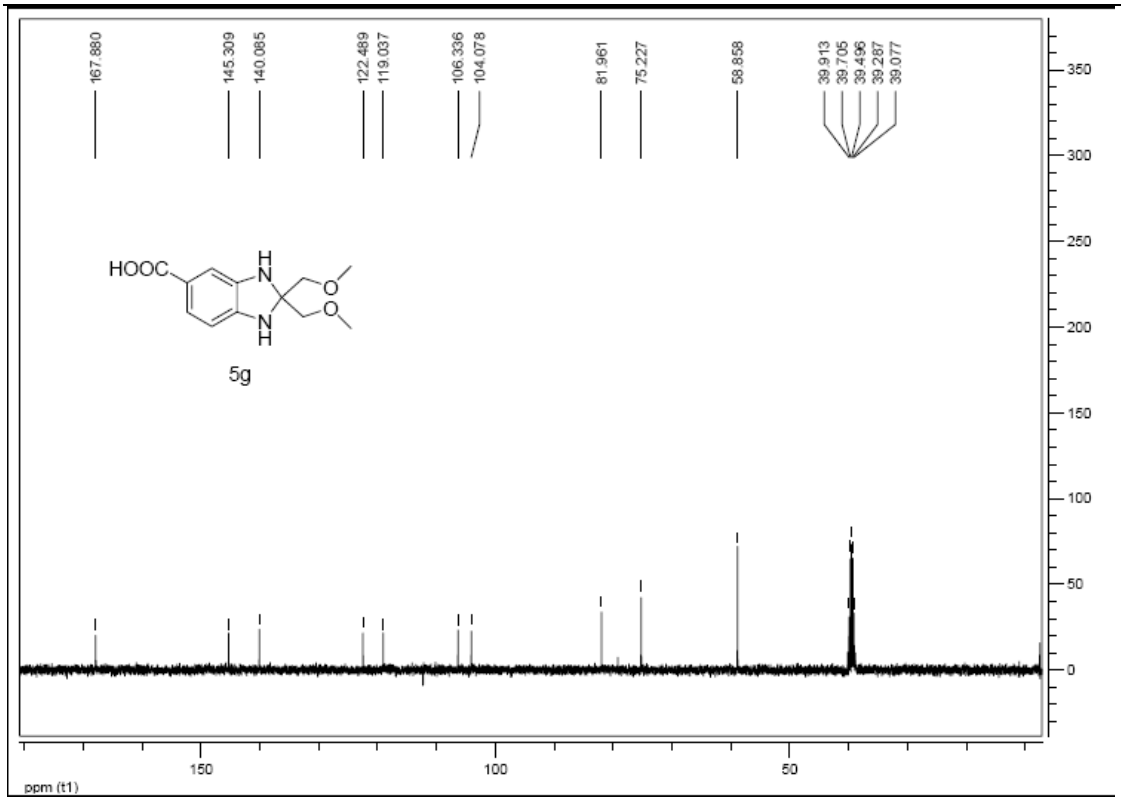


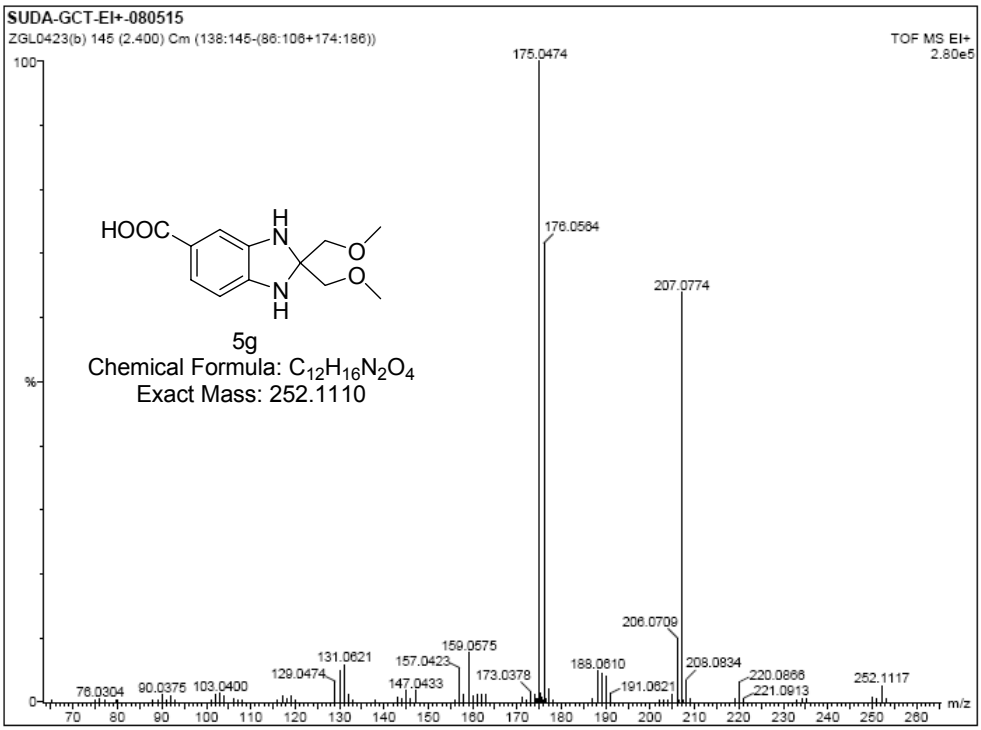

142.160
140.881
139.868
134.219
128.889
126.940
126.559
119.842
110.375
109.357
81.212
77.760
77.441
77.124
74.967
59.745
Ph
5d
150
100
50
0
ppm (t1)

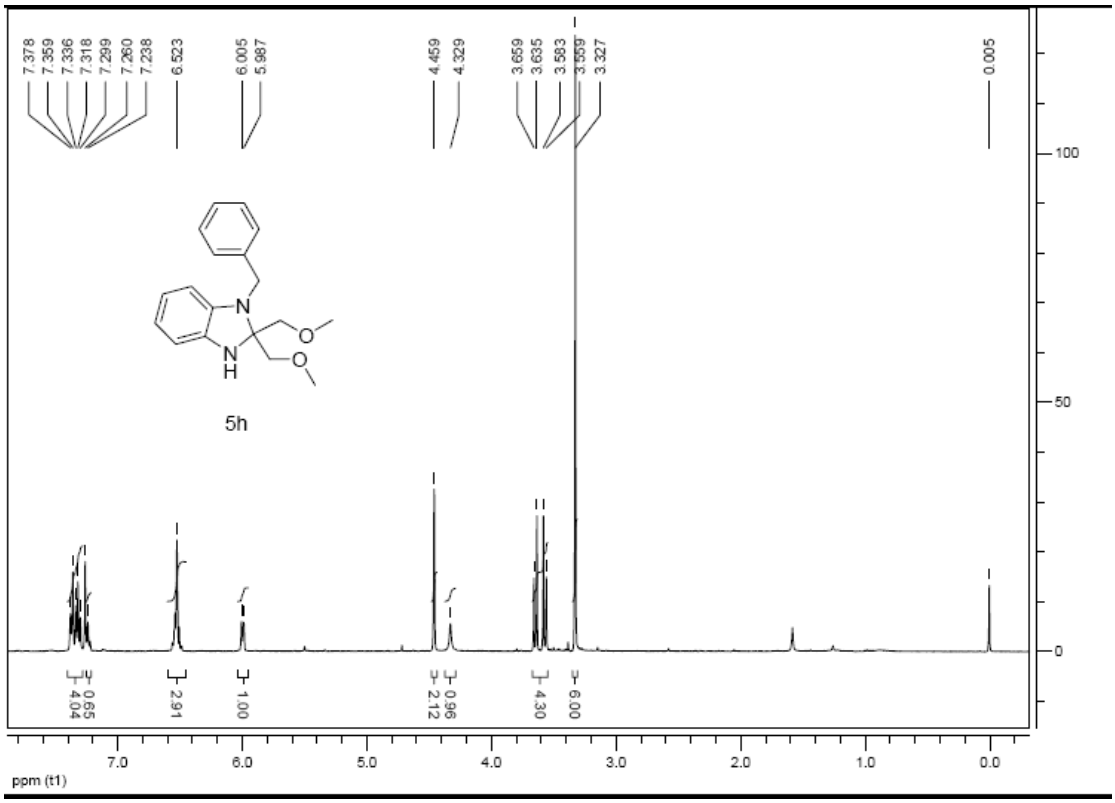

SUDA-GCT-EI+-080515
ZGL0422(b) 129 (2.149) Cm (129:134-(83:91+150:160))
TOF MS EI+
1.71e5
Ph
5d
Chemical Formula: C17H20N2O2
Exact Mass: 284.1525
207.9742
238.9740
205.9716
219.9811
151.9797
204.9670
221.9943
165.9775
179.9834
281.9866
138.9825
240.0750
193.9853
103.4935
102.9913
103.9974
251.9945
284.0046
77.0024
272.9377
290.9614
%
m/z

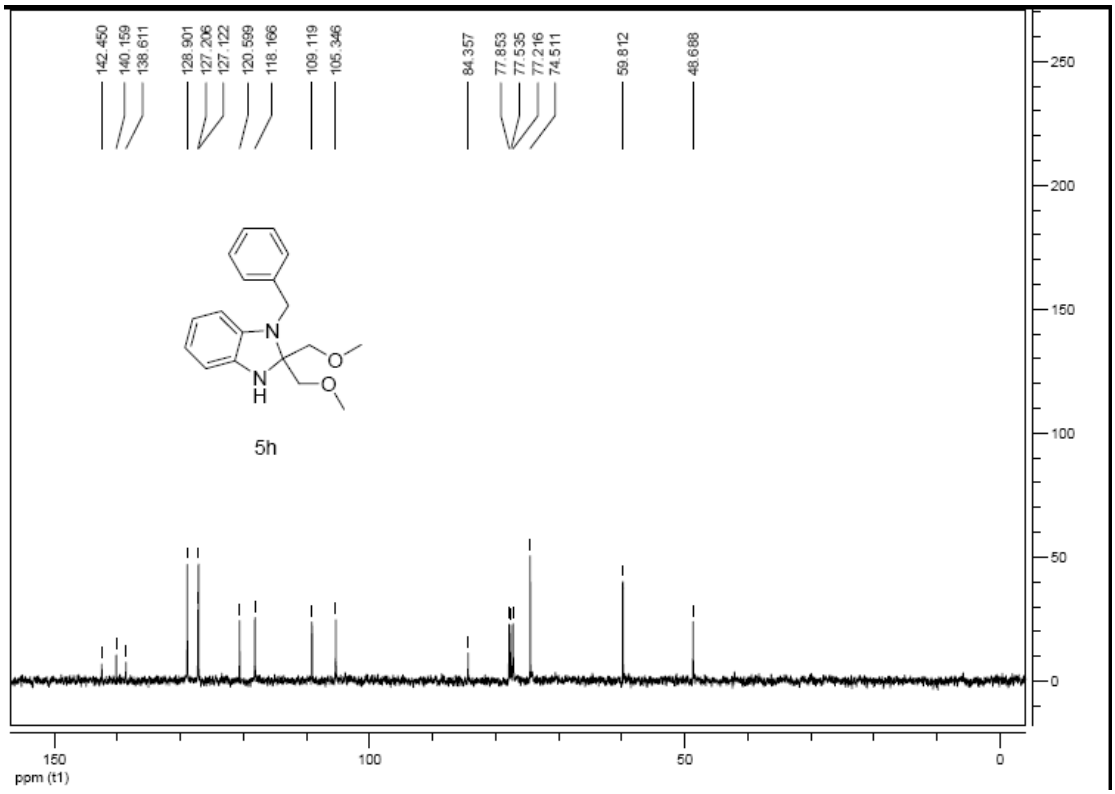

11.794
7.127
7.124
7.107
6.751
6.612
6.217
6.197
5.957
3.341
3.289
HOOC
5g
0.95
1.03
0.98
1.03
1.01
1.00
4.00
10.0
5.0
0.0
ppm (t1)

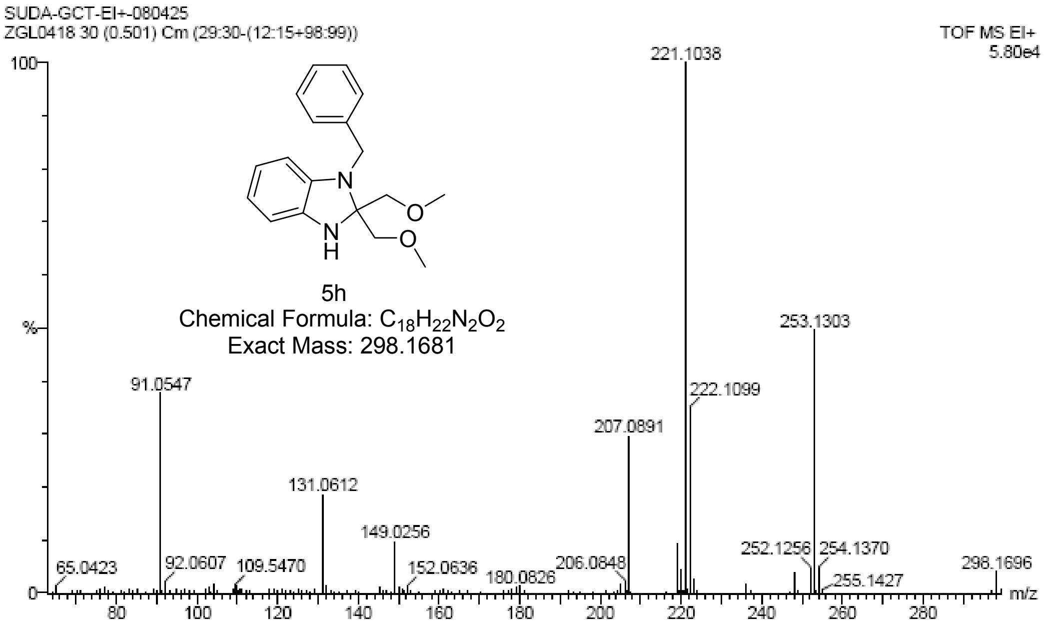

7.131
7.128
7.111
7.108
6.755
6.236
6.217
3.634
3.327
3.267
2.500
HOOC
5g DMSO-d6+D2O
0.97
0.98
0.97
3.92
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

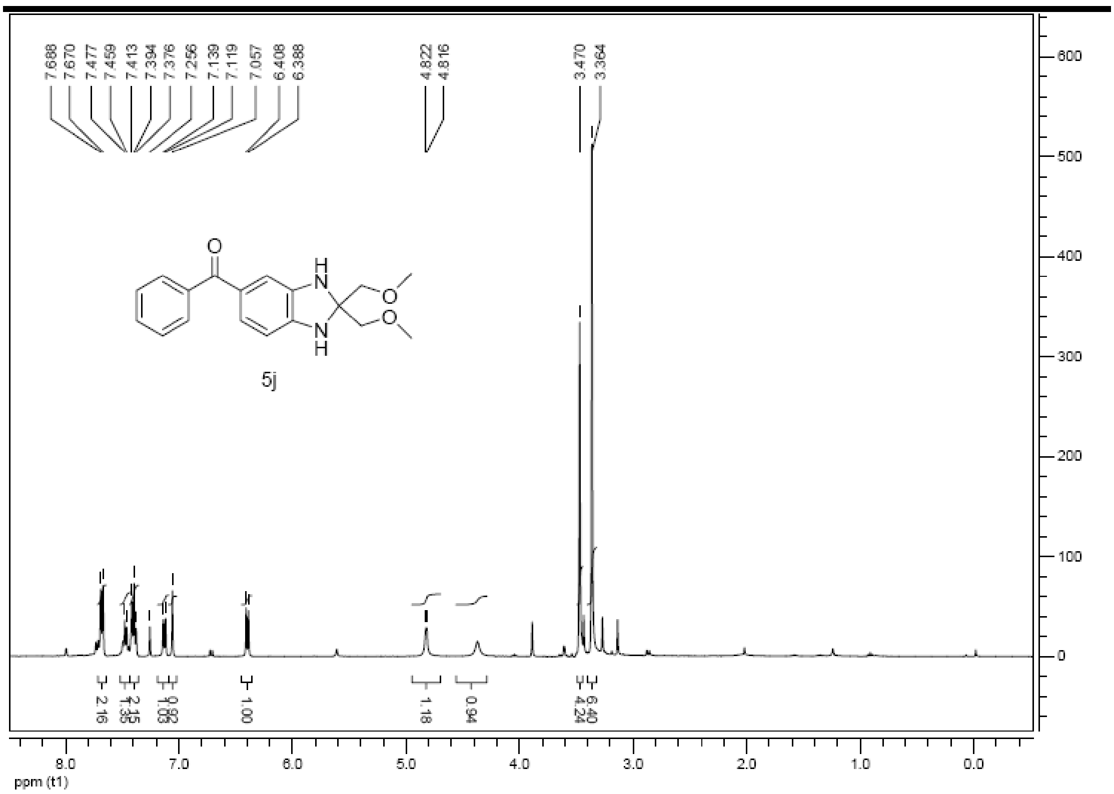

167.880
145.309
140.085
122.489
119.037
106.336
104.078
81.961
75.227
58.858
39.913
39.705
39.496
39.287
39.077
HOOC
H
N
O
O
N
H
5g
350
300
250
200
150
100
50
0
150
100
50
ppm (t1)

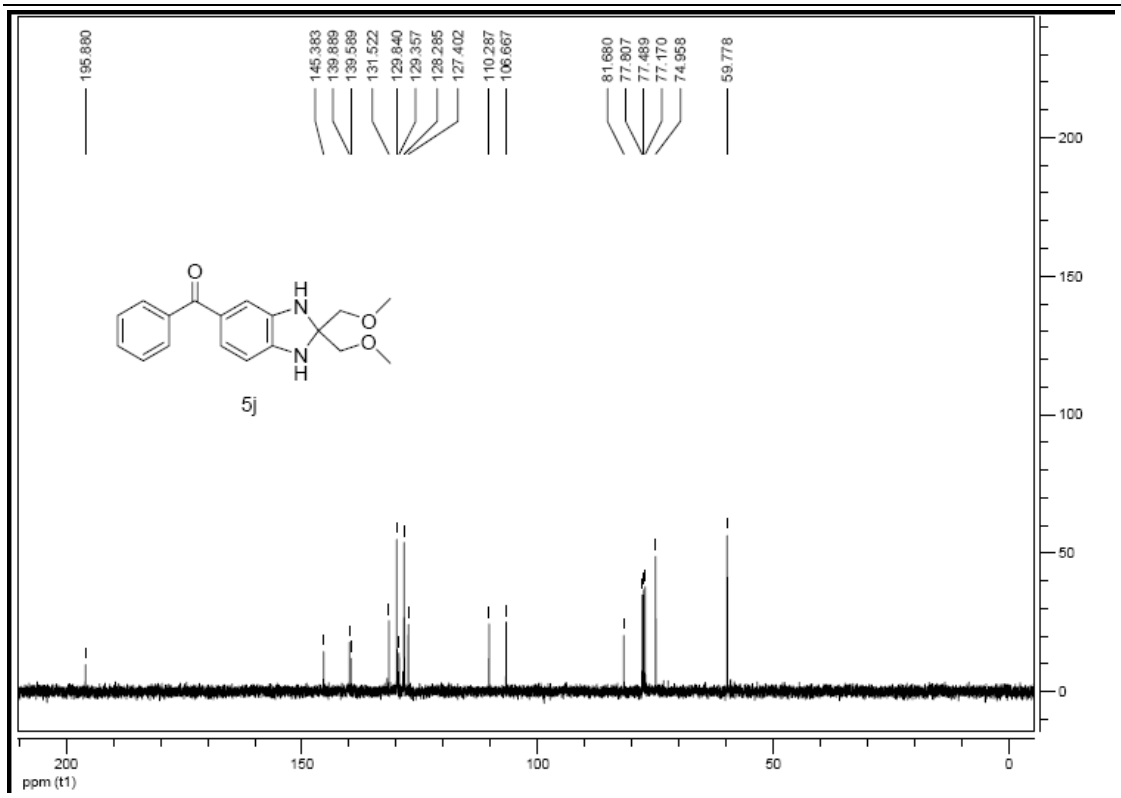

SUDA-GCT-EI+-080515
ZGL0423(b) 145 (2.400) Cm (138:145-(86:106+174:186))
TOF MS EI+
2.80e5
100
%
0
175.0474
176.0564
207.0774
206.0709
159.0575
131.0621
157.0423
129.0474
147.0433
173.0378
188.0610
208.0834
191.0621
220.0866
221.0913
252.1117
76.0304
90.0375
103.0400
m/z
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
HOOC
H
N
O
O
N
H
5g
Chemical Formula: C12H16N2O4
Exact Mass: 252.1110

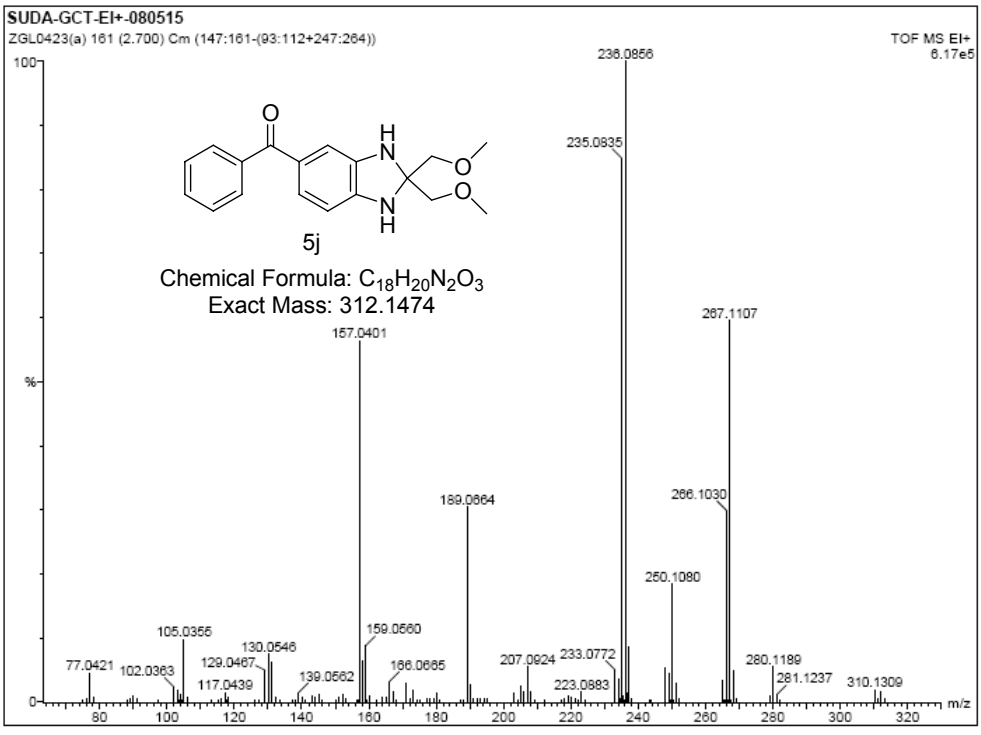

7.378
7.359
7.336
7.318
7.299
7.260
7.238
6.523
6.005
5.987
4.459
4.329
3.659
3.635
3.583
3.559
3.327
0.005
5h
0.55
4.04
1.91
1.00
2.12
0.96
4.30
6.00
ppm (t1)

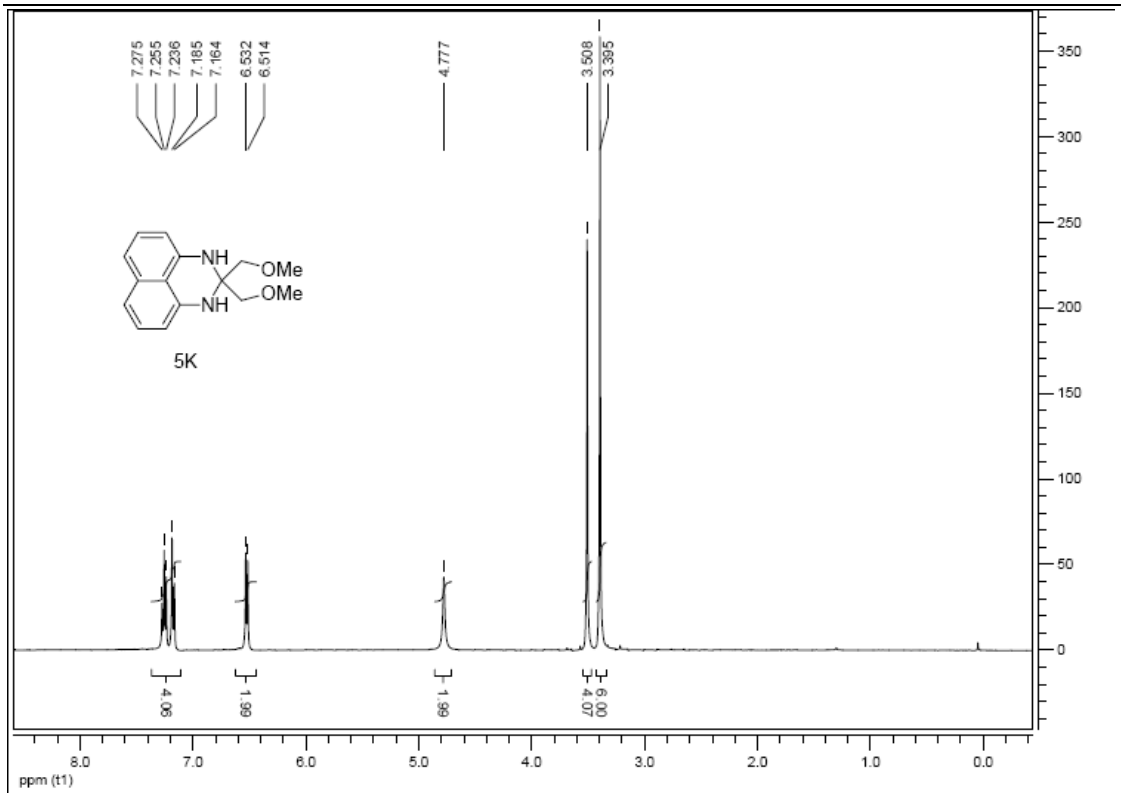

142.450
140.159
138.611
128.901
127.206
127.122
120.599
118.166
109.119
105.346
84.357
77.853
77.535
77.216
74.511
59.812
48.688
5h
ppm (t1)

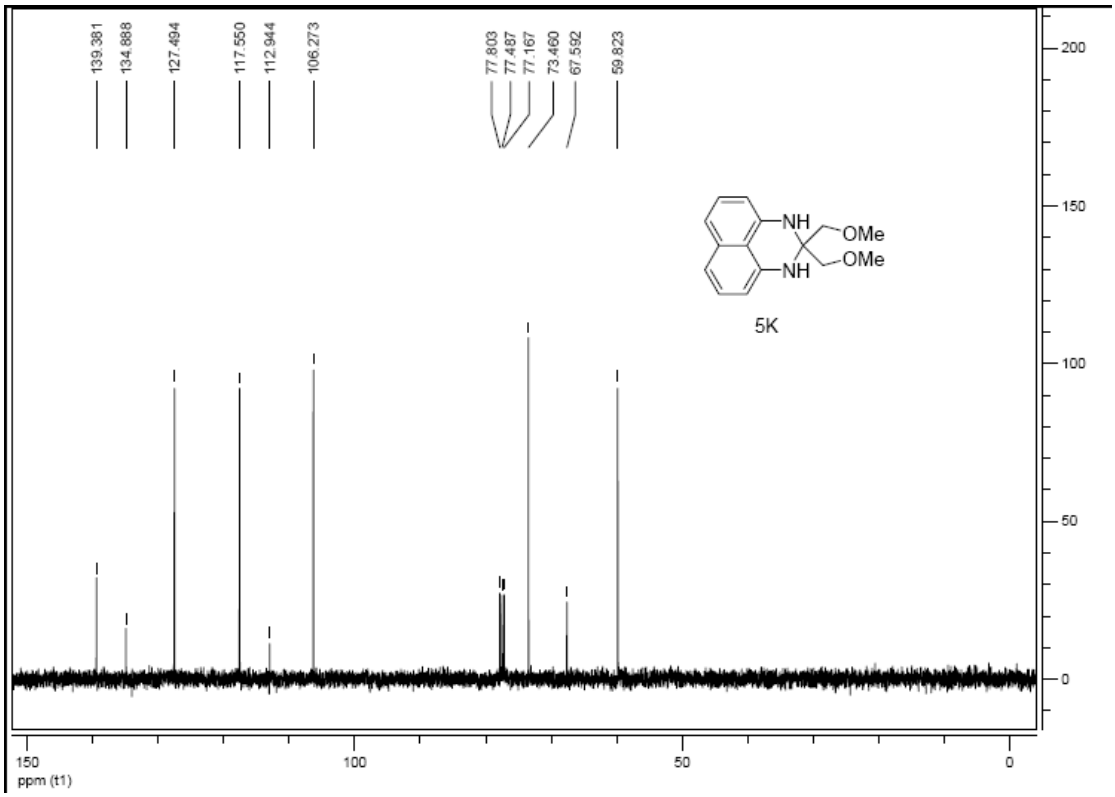

SUDA-GCT-EI+-080425
ZGL0418 30 (0.501) Cm (29:30-(12:15+98:99))
TOF MS EI+
5.80e4
5h
Chemical Formula: C18H22N2O2
Exact Mass: 298.1681
221.1038
253.1303
91.0547
222.1099
207.0891
131.0612
149.0256
252.1256
254.1370
298.1696
65.0423
92.0607
109.5470
152.0636
180.0826
206.0848
255.1427
m/z

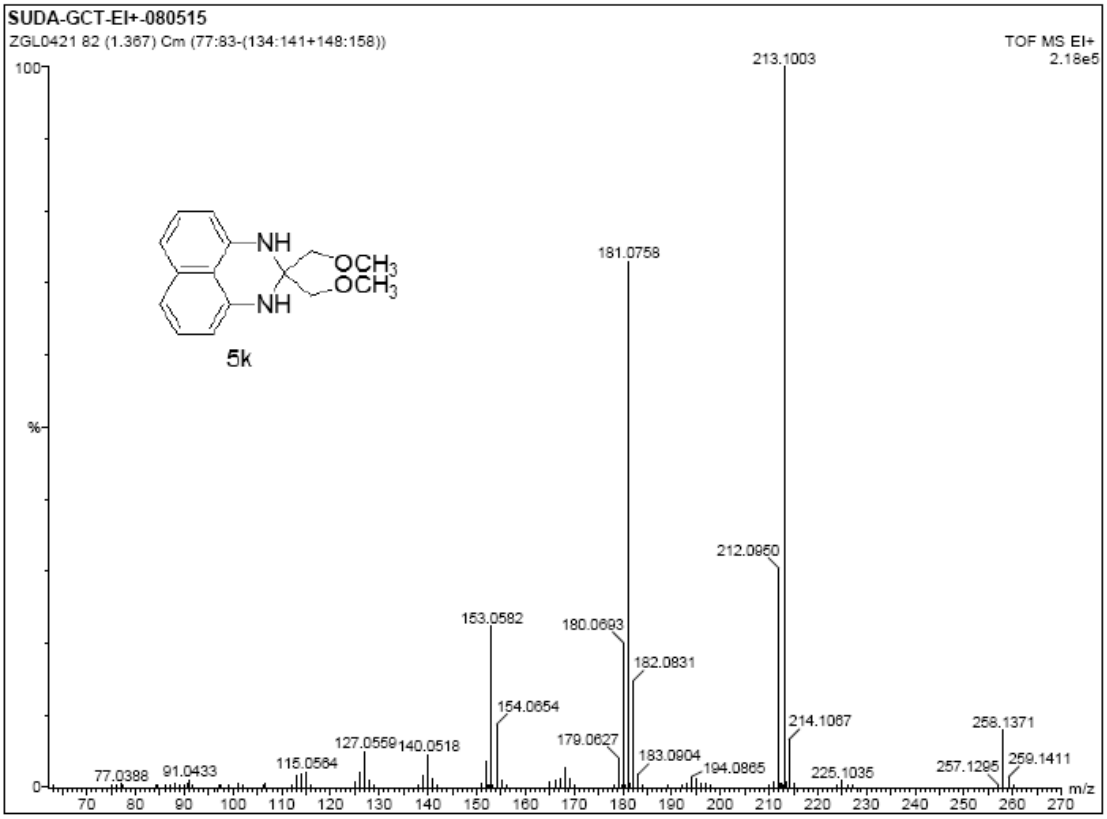

7.688
7.670
7.477
7.459
7.413
7.394
7.376
7.256
7.139
7.119
7.057
6.408
6.388
4.822
4.816
3.470
3.364
5j
2.16
2.15
1.35
1.05
0.93
1.00
1.18
0.94
6.40
4.24
ppm (t1)

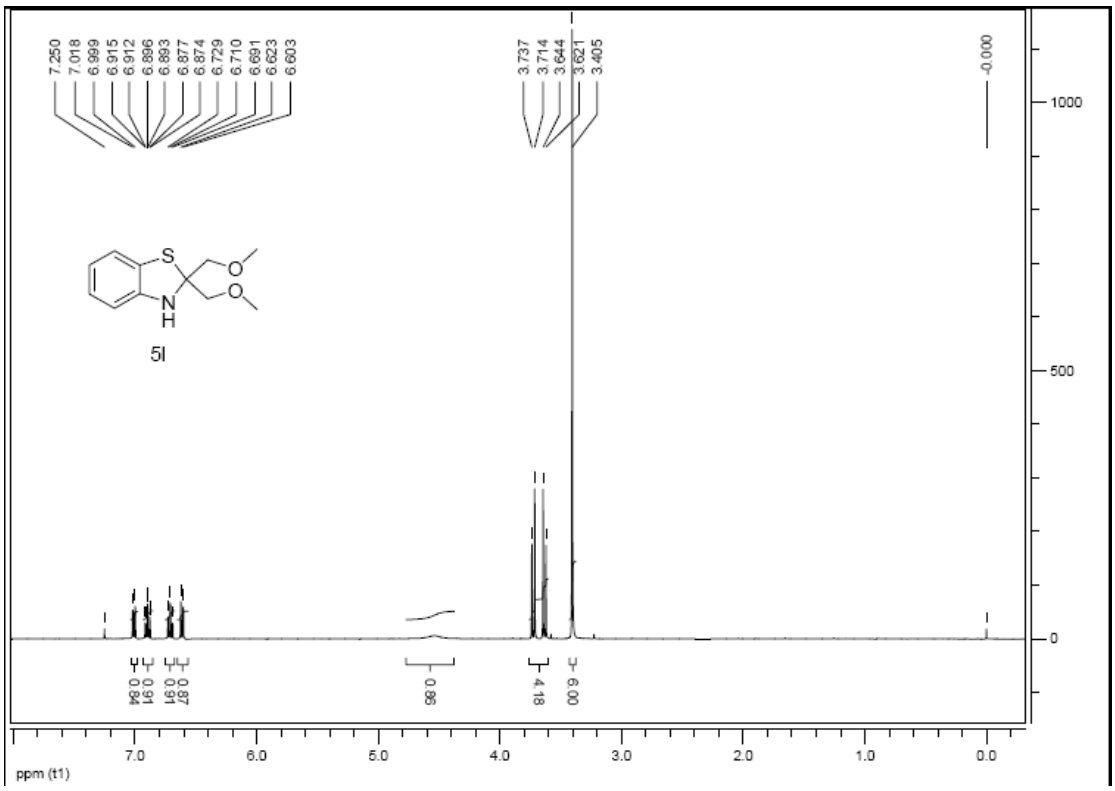

195.880
145.383
139.889
139.589
131.522
129.840
129.357
128.285
127.402
110.287
106.667
81.680
77.807
77.489
77.170
74.958
59.778
O
H
N
O
O
N
H
5j
200
150
100
50
0
ppm (t1)

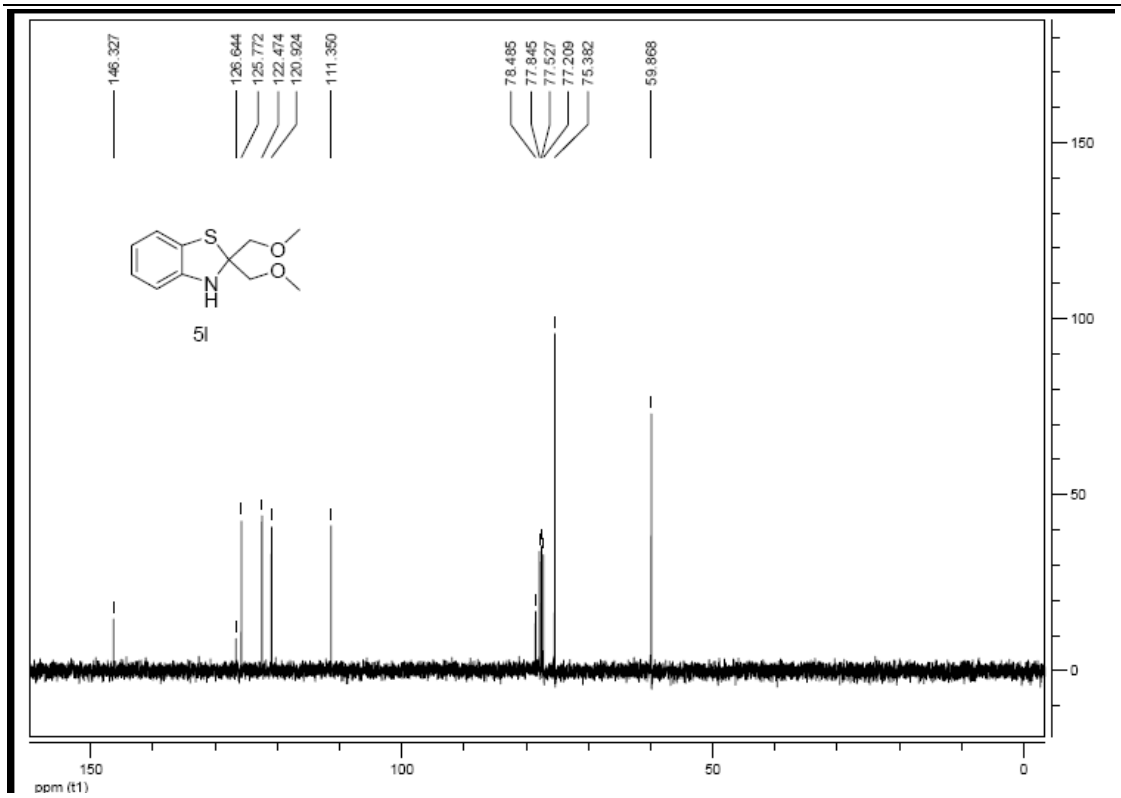

SUDA-GCT-EI+-080515
ZGL0423(a) 161 (2.700) Cm (147:161-(93:112+247:264))
TOF MS EI+
6.17e5
100
%
0
O
H
N
O
O
N
H
5j
Chemical Formula: C18H20N2O3
Exact Mass: 312.1474
236.0856
235.0835
267.1107
157.0401
189.0664
266.1030
250.1080
159.0560
105.0355
130.0546
77.0421
102.0363
129.0467
117.0439
139.0562
166.0665
207.0924
233.0772
223.0883
280.1189
281.1237
310.1309
80
100
120
140
160
180
200
220
240
260
280
300
320
m/z

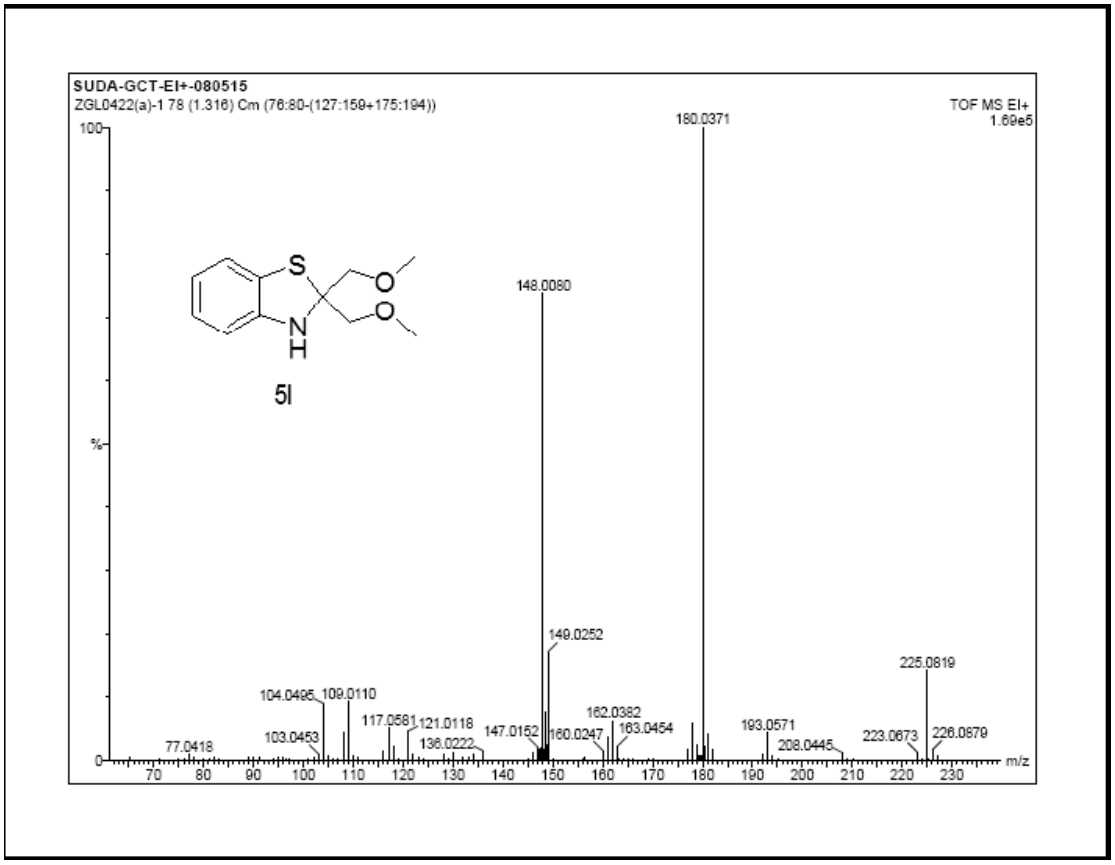

7.275
7.255
7.236
7.185
7.164
6.532
6.514
4.777
3.508
3.395
NH
OMe
OMe
NH
5K
4.06
1.99
1.99
4.07
6.00
ppm (t1)


139.381
134.888
127.494
117.550
112.944
106.273
77.803
77.487
77.167
73.460
67.592
59.823
NH
OMe
OMe
NH
5K
ppm (t1)

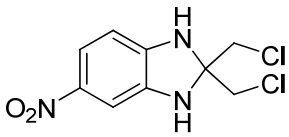

SUDA-GCT-EI+-080515
ZGL0421 82 (1.367) Cm (77:83-(134:141+148:158))
TOF MS EI+
2.18e5
100
%
0
213.1003
181.0758
212.0950
153.0582
180.0693
182.0831
154.0654
214.1067
258.1371
127.0559
140.0518
179.0627
183.0904
194.0865
225.1035
257.1295
259.1411
115.0564
77.0388
91.0433
m/z
NH
OCH3
OCH3
NH
5k


7.250
7.018
6.999
6.915
6.912
6.896
6.893
6.877
6.874
6.729
6.710
6.691
6.623
6.603
3.737
3.714
3.644
3.621
3.405
-0.000
1000
500
0
0.94
0.91
0.91
0.87
0.96
4.18
6.00
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
S
O
O
N
H
5l

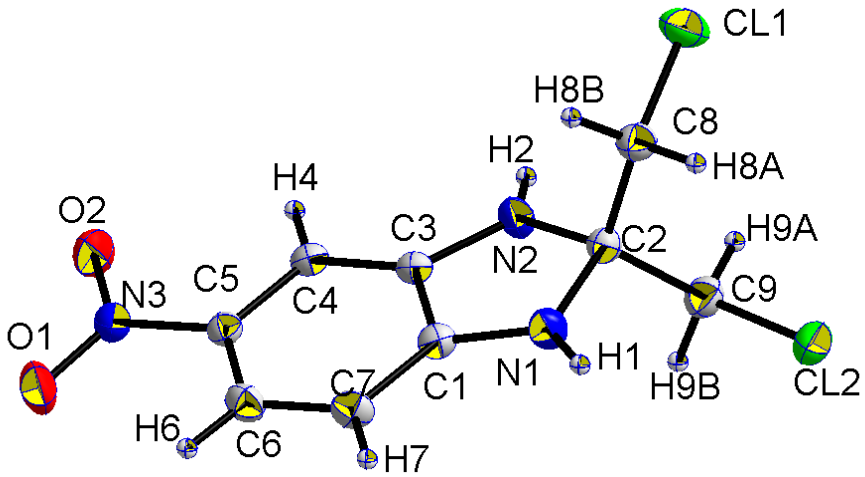

146.327
126.644
125.772
122.474
120.924
111.350
78.485
77.845
77.527
77.209
75.382
59.868
S
O
O
N
H
5l
150
100
50
0
ppm (t1)


SUDA-GCT-EI+-080515
ZGL0422(a)-1 78 (1.316) Cm (76:80-(127:159+175:194))
TOF MS EI+
1.69e5
180.0371
148.0080
149.0252
225.0819
104.0495
109.0110
117.0581
121.0118
162.0382
163.0454
147.0152
160.0247
193.0571
208.0445
223.0673
226.0879
103.0453
136.0222
77.0418
S
O
O
N
H
5l
100
%
0
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
m/z

IV. X-Ray crystal structures

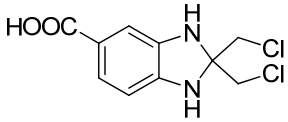


3c

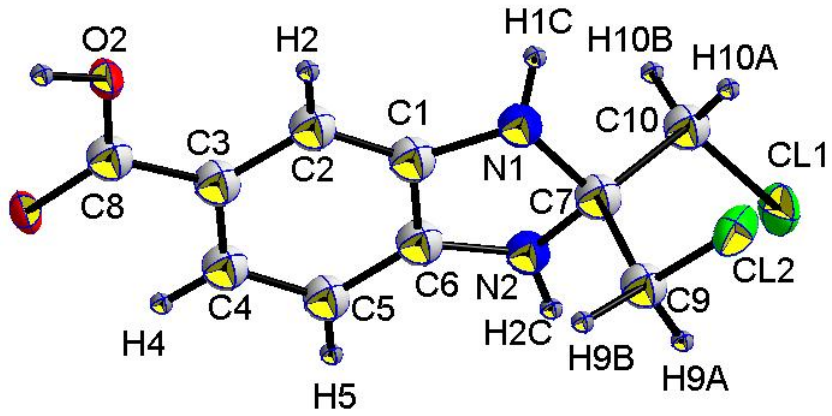


CCDC 793232

Formula: $C_9H_9Cl_2N_3O_2$; Unit cell parameters: a = 5.6284(2), b = 12.5878(4), c = 15.9350(4) Å; $\alpha = 90.00°$, $\beta = 98.913(2)°$, $\gamma = 90.00°$; space group P21/c.

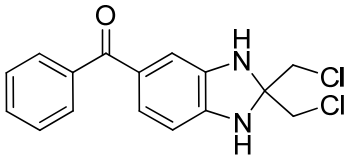


3g

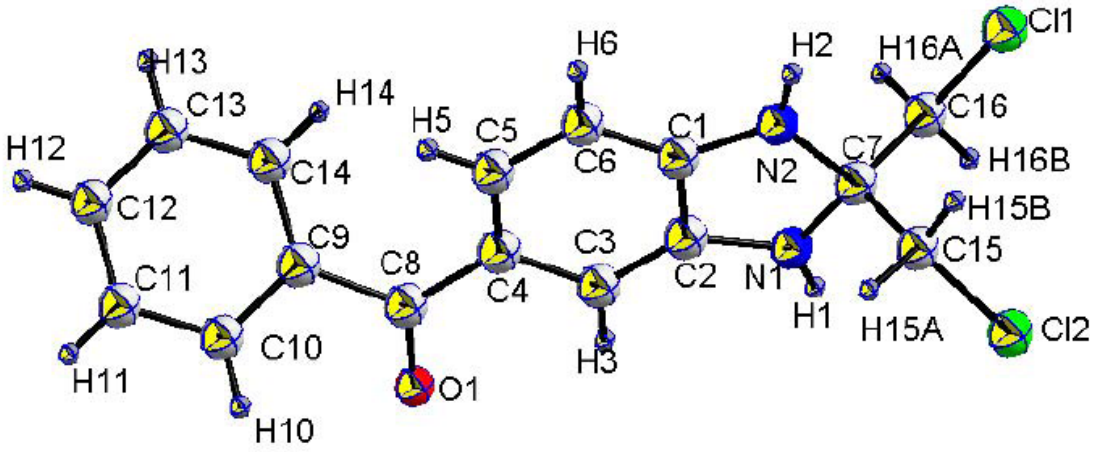


CCDC 793231

Formula: $C_{10}H_{10}Cl_2N_2O_2$; Unit cell parameters: a = 5.88980(10), b = 18.9022(4), c = 10.1073(2) Å; α = 90.00°, β = 105.3930(10)°, γ = 90.00° ; space group P21/c.



3j



CCDC 793230

Formula: C_{16} H_{14} Cl_2 N_2 O_1
Unit cell parameters: a 9.8379(6) b 12.4803(7) c 13.9555(8)alpha 108.764(2) beta 92.805(2) gamma 106.932(2); space group P-1