SUPPORTING INFORMATION

Reaction of 6-aminouracils with aldehydes in water as both solvent and reactant under $FeCl_3.6H_2O$ catalysis: towards 5-alkyl/arylidenebarbituric acids

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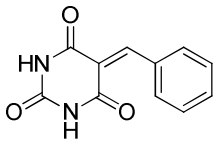
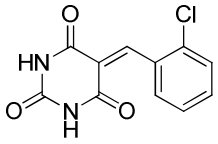
1. Compound Characterization data of 5-alkyl/arylidenebarbituric acids (4aa-4an and 4ba-4cd)



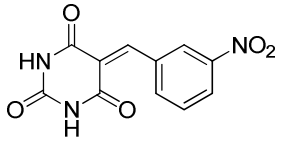
4aa

5-Benzylidenepyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (4aa)[38d]: Yield, 0.183 g (85%). White solid, mp 270-272 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 7.90-7.92 (m, 3H), 8.03- 8.05 (m, 2H), 8.25 (s, 1H), 11.22 (s, 1H), 11.38 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 119.2, 128.1, 129.3, 132.3, 133.2, 150.3, 154.8, 161.7, 163.5; IR (KBr)(ν_{max}/ cm^{-1}) 3244, 3153, 1751, 1692, 1665, 1621, 1545; MS (ESI): *m*/*z* calcd for $C_{11}H_8N_2O_3$: 216.05; found 217.0 $[M + H]^+$, 239.0 $[M + Na]^+$; anal. calcd for $C_{11}H_8N_2O_3$: C, 61.11; H, 3.73; N, 12.96. Found: C, 61.31; H, 3.91; N 12.72%.



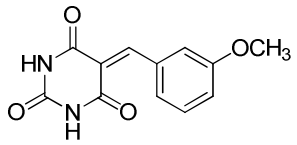
4ab

5-(2-Chlorobenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (4ab)[38d]: Yield, 0.20 g (80%). White solid, mp 250-252 °C (from EtOH). ^{1}H NMR (600 MHz, DMSO-d_6): δ (ppm) 7.36 (t, 1H, J = 7.8 Hz), 7.45-7.48 (m, 1H), 7.53 (d, 1H, J = 7.8 Hz), 7.73 (d, 1H, J = 7.2 Hz), 8.29 (s, 1H), 11.25 (s, 1H), 11.47 (s, 1H); ^{13}C NMR (150 MHz, DMSO-d_6): δ (ppm) 121.7, 126.3, 128.9, 131.9, 132.0, 132.3, 133.2, 149.9, 150.2, 160.9, 162.7; IR (KBr)(ν_{max}/ cm^{-1}) 3231, 3125, 1741, 1699, 1671, 1622, 1532; MS (ESI): *m*/*z* calcd for $C_{11}H_7ClN_2O_3$: 250.01; found 251.0 $[M + H]^+$, 273.0 $[M + Na]^+$; anal. calcd for $C_{11}H_7ClN_2O_3$: C, 52.71; H, 2.82; N, 11.18. Found: C, 52.91; H, 2.69; N 11.33%.



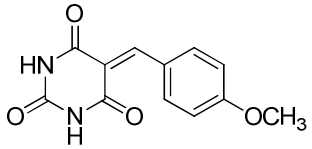
4ac

5-(3-Nitrobenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ac**)[30a]: Yield 0.211 g (81%). Brown solid, mp 243-245 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 7.70-8.31 (m, 4H), 8.93 (s, 1H), 11.32 (s, 1H), 11.47 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 122.0, 125.9, 126.5, 131.3, 135.3, 138.8, 147.5, 150.6, 151.6, 161.9, 163.2; IR (KBr)(ν_{max}/ cm^{-1}) 3267, 3143, 1733, 1696, 1670, 1622, 1565; MS (ESI): *m*/*z* calcd for $C_{11}H_7N_3O_5$: 261.04; found 262.0 $[M + H]^+$, 283.9 $[M + Na]^+$; anal. calcd for $C_{11}H_7N_3O_5$: C, 50.58; H, 2.70; N, 16.09. Found: C, 50.73; H, 2.91; N 15.81%.



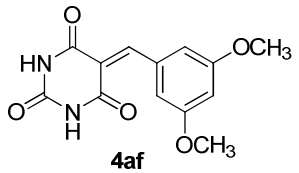
4ad

5-(3-Methoxybenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ad**): Yield, 0.233 g (95%). Yellow solid, mp 227-229 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.7 (s, 3H), 7.09-7.22 (m, 1H), 7.34-7.39 (m, 1H), 7.57 (d, 1H, J = 9 Hz), 7.82 (s, 1H), 8.23 (s, 1H), 11.25 (s, 1H), 11.40 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 49.2, 111.6, 112.5, 113.3, 120.1, 123.1, 127.8, 144.2, 148.5, 152.6, 155.7, 157.4; IR (KBr)(ν_{max}/ cm^{-1}) 3234, 3178, 1740, 1702, 1678, 1620, 1570; MS (ESI): *m*/*z* calcd for $C_{12}H_{10}N_2O_4$: 246.06; found 246.9 $[M + H]^+$, 269.1 $[M + Na]^+$; anal. calcd for $C_{12}H_{10}N_2O_4$: C, 58.54; H, 4.09; N 11.38. Found: C, 58.79; H, 3.93; N, 11.49%.



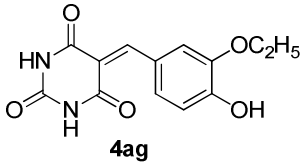
4ae

5-(4-Methoxybenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ae**)[38d]: Yield, 0.236 g (96%). Yellow solid, mp 277-279 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.85 (s, 3H), 7.03 (d, 2H, J = 9 Hz), 8.22 (s, 1H), 8.33 (d, 2H, J = 8.7 Hz), 11.18 (s, 1H), 11.31 (s, 1H). ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 49.7, 108.0, 109.5, 119.1, 131.5, 144.2, 149.0, 156.2, 157.5; IR (KBr)(ν_{max}/ cm^{-1}) 3245, 3127, 1765, 1689, 1660, 1623, 1545; MS (ESI): *m*/*z* calcd for $C_{12}H_{10}N_2O_4$: 246.06; found 247.0 $[M + H]^+$, 269.0 $[M + Na]^+$; anal. calcd for $C_{12}H_{10}N_2O_4$: C, 58.54; H, 4.09; N, 11.38. Found: C, 58.72; H, 3.99; N, 11.65%.



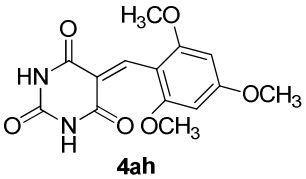
4af

5-(3,5-Dimethoxybenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4af**): Yield, 0.248 g (90%). Yellow solid, mp 235-237 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.74 (s, 6H), 6.68 (s, 1H), 7.33 (m, 2H), 8.17 (s, 1H), 11.22 (s, 1H), 11.39 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 55.4, 104.6, 111.0, 119.5, 134.2, 150.2, 154.6, 159.8, 161.6, 163.4; IR (KBr)(ν_{max}/ cm^{-1}) 3233, 3125, 1770, 1685, 1657, 1620, 1543; MS (ESI): *m*/*z* calcd for $C_{13}H_{12}N_2O_5$: 276.07; found 277.0 $[M + H]^+$, 299.1 $[M + Na]^+$; anal. calcd for $C_{13}H_{12}N_2O_5$: C, 56.52; H, 4.38; N, 10.14. Found: C, 56.73; H, 4.53; N, 10.02%.



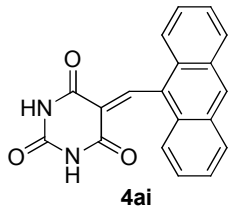
4ag

5-(3-Ethoxy-4-hydroxybenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ag**): Yield, 0.240 g (87%). Yellow solid, mp 251-253 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.31-1.36 (m, 3H), 4.03-4.08 (m, 2H), 6.86-6.90 (m, 1H), 7.70-7.73 (m, 1H), 8.20 (s, 1H), 8.48 (s, 1H), 10.52 (s, 1H), 11.12 (s, 1H), 11.24 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 8.6, 57.8, 107.9, 109.3, 112.8, 118.2, 126.7, 140.1, 144.2, 147.3, 150.0, 156.5, 158.3; IR (KBr)(ν_{max}/ cm^{-1}) 3564, 3244, 3152, 1778, 1696, 1651, 1627, 1538; MS (ESI): *m*/*z* calcd for $C_{13}H_{12}N_2O_5$: 276.07; found 299.1 $[M + Na]^+$; anal. calcd for $C_{13}H_{12}N_2O_5$: C, 56.52; H, 4.38; N, 10.14. Found: C, 55.72; H, 4.61; N 10.32%.



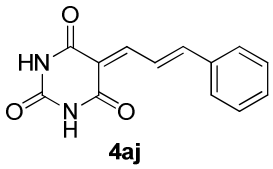
4ah

5-(2,4,6-Trimethoxybenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ah**): Yield, 0.278 g (91%). Yellow solid, mp 265-267 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.68 (s, 3H), 3.75 (s, 3H), 3.79 (s, 3H), 7.80 (m, 2H), 8.22 (s, 1H), 11.23 (s, 1H), 11.36 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 55.61, 59.9, 112.1, 117.0, 127.2, 141.3, 149.8, 151.5, 154.9, 163.3; IR (KBr)(ν_{max}/ cm^{-1}) 3256, 3145, 1771, 1698, 1661, 1622, 1525; MS (ESI): *m*/*z* calcd for $C_{14}H_{14}N_2O_6$: 306.09; found 306.9 $[M + H]^+$, 329.0 $[M + Na]^+$; anal. calcd for $C_{14}H_{14}N_2O_6$: C, 54.90; H, 4.61; N, 9.15. Found: C, 54.71; H, 4.74; N, 9.36%.



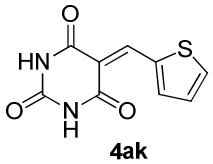
4ai

5-(Anthracen-10-ylmethylene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ai**)[39]: Yield, 0.246 g (78%). Red solid, mp > 300 °C (from EtOH). ^{1}H NMR (600 MHz, DMSO-d_6): δ (ppm) 7.49-7.55 (m, 4H), 7.93 (d, 2H, J = 8.4 Hz), 8.12 (d, 2H, J = 7.8 Hz), 8.56 (s, 1H), 8.98 (s, 1H), 11.07 (s, 1H), 11.52 (s, 1H); ^{13}C NMR (150 MHz, DMSO-d_6): δ (ppm) 125.5, 126.2, 127.6, 127.7, 128.6, 129.4, 130.5, 150.4, 151.2, 151.6, 160.4, 162.3, 167.7; IR (KBr)(ν_{max}/ cm^{-1}) 3245, 3182, 1743, 1695, 1678, 1620, 1570; MS (ESI): m/z calcd for $C_{19}H_{12}N_2O_3$: 316.08; found 317.0 $[M + H]^+$, 338.9 $[M + Na]^+$; anal. calcd for $C_{19}H_{12}N_2O_3$: C, 72.15; H, 3.82; N, 8.86. Found: C, 72.33; H, 3.94; N 8.58%.



4aj

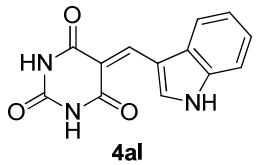
5-(3-Phenylallylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4aj**)[38e]: Yield, 0.220 g (91%). Yellow solid, mp 258-260 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 7.46 (m, 3H), 7.66-7.71 (m, 3H), 7.96-8.01 (m, 1H), 8.36-8.47 (m, 1H), 11.19 (s, 1H), 11.24 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 109.8, 118.3, 122.6, 123.3, 125.2, 129.2, 144.3, 146.7, 147.7, 157.0, 157.1; IR (KBr)(ν_{max}/ cm^{-1}) 3370, 3221, 1712, 1688, 1648, 1620, 1550; MS (ESI): m/z calcd for $C_{13}H_{10}N_2O_3$: 242.07; found 242.9 $[M + H]^+$, 264.9 $[M + Na]^+$; anal. calcd for $C_{13}H_{10}N_2O_3$: C, 64.46; H, 4.16; N, 11.56. Found: C, 64.68; H, 4.34; N, 11.28%.



4ak

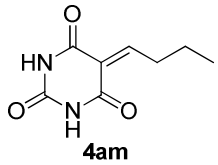
5-(Thiophen-2-ylmethylene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ak**)[38c]: Yield, 0.179 g (81%). Yellow solid, mp 269-271 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 7.33 (m, 1H), 8.16 (m, 1H), 8.26 (m, 1H), 8.54 (s, 1H), 11.25 (s, 1H), 11.29 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 105.6, 122.4, 130.3, 136.2, 139.8, 139.9, 144.3, 157.1, 157.5; IR (KBr)(ν_{max}/ cm^{-1}) 3241, 3136, 1768, 1690, 1646, 1620; MS (ESI): m/z calcd for

$C_9H_6N_2O_3S$: 222.01; found 245.0 $[M + Na]^+$; anal. calcd for $C_9H_6N_2O_3S$: C, 48.64; H, 2.72; N, 12.61. Found: C, 48.86; H, 2.91; N, 12.36%.



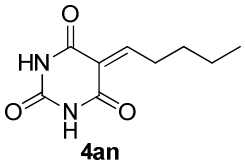
4al

5-((1*H*-Indol-2-yl)methylene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4al**)[1b]: Yield, 0.211 g (83%). Yellow solid, mp 190-192 °C (from EtOH). ^{1}H NMR (600 MHz, DMSO-d_6): δ (ppm) 7.32-7.33 (m, 2H), 7.58-7.60 (m, 1H), 7.87-7.88 (m, 1H), 8.71 (s, 1H), 9.50-9.51 (m, 1H), 11.03 (s, 1H), 11.11 (s, 1H), 12.73 (s, 1H). ^{13}C NMR (150 MHz, DMSO-d_6): δ (ppm) 108.6, 111.4, 113.1, 117.6, 122.6, 123.6, 129.1, 136.4, 139.7, 143.7, 150.3, 163.2, 164.5; IR (KBr)(ν_{max}/ cm^{-1}) 3245, 3167, 1772, 1694, 1654, 1621, 1533; MS (ESI): *m*/*z* calcd for $C_{13}H_9N_3O_3$: 255.06; found 255.9 $[M + H]^+$, 277.9 $[M + Na]^+$; anal. calcd for $C_{13}H_9N_3O_3$: C, 61.18; H, 3.55; N, 16.46. Found: C, 61.39; H, 3.39; N, 16.74%.



4am

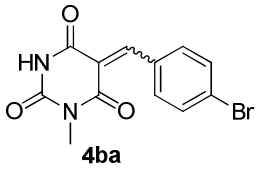
5-Butylidenepyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4am**): Yield, 0.136 g (75%). White solid, mp 223-225 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 0.71-0.92 (m, 3H), 1.22-1.27 (m, 2H), 1.68-1.70 (m, 2H), 7.83-7.87 (m, 1H), 11.66 (s, 1H), 11.70 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6 + $CDCl_3$): δ (ppm) 24.2, 25.4, 31.08, 135.9, 150.0, 151.2, 151.9, 164.2; IR (KBr)(ν_{max}/ cm^{-1}) 3225, 3133, 2924, 1745, 1683, 1621; MS (ESI): *m*/*z* calcd for $C_8H_{10}N_2O_3$: 182.07; found 182.9 $[M + H]^+$, 204.9 $[M + Na]^+$; anal. calcd for $C_8H_{10}N_2O_3$: C, 52.74; H, 5.53; N, 15.38. Found: C, 52.91; H, 5.33; N, 15.56%.



4an

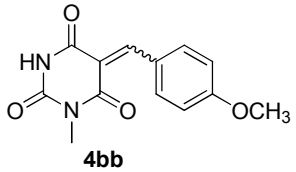
5-Pentylidenepyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4an**): Yield, 0.143 g (73%). Yellow solid, mp 243-245 °C (from EtOH). ^{1}H NMR (600 MHz, DMSO-d_6): δ (ppm) 0.69-0.71 (m, 3H), 0.75-0.88 (m, 6H), 7.88-7.90 (m, 1H), 11.63 (s, 1H), 11.66 (s, 1H); ^{13}C NMR (150 MHz, DMSO-d_6): δ (ppm)13.6, 14.6, 21.3, 33.5, 129.7, 149.2, 151.6, 162.0, 167.7; IR (KBr)(ν_{max}/ cm^{-1}) 3229, 3132, 2924, 1747, 1685, 1667, 1622; MS (ESI): *m*/*z* calcd for

$C_9H_{12}N_2O_3$: 196.08; found 196.9 $[M + H]^+$, 218.9 $[M + Na]^+$; anal. calcd for $C_9H_{12}N_2O_3$: C, 55.09; H, 6.16; N, 14.28. Found: C, 55.27; H, 5.97; N, 14.54%.



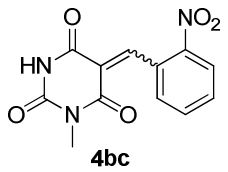
4ba

5-(4-Bromobenzylidene)-1-methylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ba**): Yield, 0.250 g (81%). White solid, mp 207-209 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.08/3.14 (s‡, 3H), 7.63-7.66 (m, 2H), 7.90-7.95 (m, 2H), 8.21/8.23 (s‡, 1H), 11.48/11.62 (s‡, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 26.7/27.3, 119.3, 125.4, 130.6/130.8, 131.5/131.8, 134.0/134.1, 150.0/150.1, 152.6/153.2, 160.1/160.7, 161.5/162.5; IR (KBr)(ν_{max}/ cm^{-1}) 3235, 1736, 1698, 1670, 1621, 1538; MS (ESI): *m*/*z* calcd for $C_{12}H_9BrN_2O_3$: 307.98; found 307.9 $[M + H]^+$, 329.9 $[M + Na]^+$; anal. calcd for $C_{12}H_9BrN_2O_3$: C, 46.63; H, 2.93; N, 9.06. Found: C, 46.83; H, 2.77; N, 9.32%.



4bb

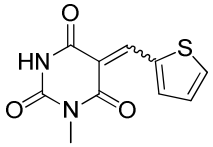
5-(4-Methoxybenzylidene)-1-methylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4bb**)[41]: Yield, 0.229 g (88%). Yellow solid, mp 243-245 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.11/3.13 (s‡, 3H), 3.83 (s, 3H), 7.01/7.04 (m‡, 2H), 8.21-8.33 (m, 3H), 11.36/11.48 (s‡, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 26.8/27.35, 55.27, 113.5, 115.13, 124.6/124.7, 136.9/137.0, 150.2, 154.6/155.2, 160.7/161.3, 162.2, 163.1/163.3; IR (KBr)(ν_{max}/ cm^{-1}) 3231, 1734, 1693, 1671, 1623, 1534; MS (ESI): *m*/*z* calcd for $C_{13}H_{12}N_2O_4$: 260.08; found 261.0 $[M + H]^+$, 283.0 $[M + Na]^+$; anal. calcd for $C_{13}H_{12}N_2O_4$: C, 60.00; H, 4.65; N, 10.76. Found: C, 60.23; H, 4.83; N, 10.51%.



4bc

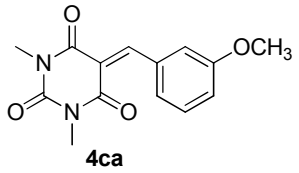
5-(2-Nitrobenzylidene)-1-methylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4bc**): Yield, 0.214 g (78%). Yellow solid, mp 192-194 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 2.97/3.16 (s‡, 3H), 7.51-7.54 (m, 1H), 7.66-7.68 (m, 1H), 7.74-7.79 (m, 1H), 8.20-8.24 (m, 1H), 8.61/8.63 (s‡, 1H), 11.45/11.69 (s‡, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm)

26.5/27.1, 119.9, 123.6, 129.5, 129.7/129.8, 131.2/131.5, 133.3, 145.6/145.8, 150.1, 152.4/152.7, 159.7/160.4, 160.9/161.8; IR (KBr)(ν_{max}/ cm^{-1}) 3234, 1731, 1697, 1668, 1621, 1531; MS (ESI): *m*/*z* calcd for $C_{12}H_9N_3O_5$: 275.05; found 275.9 $[M + H]^+$, 297.9 $[M + Na]^+$; anal. calcd for $C_{12}H_9N_3O_5$: C, 52.37; H, 3.30; N, 15.27. Found: C, 52.62; H, 3.11; N 15.41%.



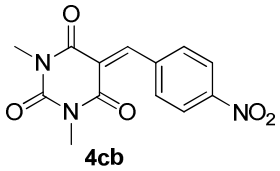
4bd

1-Methyl-5-(thiophen-2-ylmethylene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4bd**)[16a]: Yield, 0.189 g (80%). Red solid, mp 278-280 °C (from EtOH). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 3.14 (s, 3H), 7.31-7.35 (m, 1H), 8.16-8.17 (m, 1H), 8.25-8.28 (m, 1H), 8.56/8.58 (s‡, 1H), 11.48/11.51 (s‡, 1H). ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 26.4/27.1, 111.0, 128.0, 135.7/135.9, 141.8, 145.4/145.6, 146.0, 150.1, 161.5/161.9, 162.8; IR (KBr)(ν_{max}/ cm^{-1}) 3236, 1736, 1698, 1665, 1620; MS (ESI): *m*/*z* calcd for $C_{10}H_8N_2O_3S$: 236.03; found 237.0 $[M + H]^+$, 259.0 $[M + Na]^+$; anal. calcd for $C_{10}H_8N_2O_3S$: C, 50.84; H, 3.41; N, 11.86. Found: C, 51.02; H, 3.49; N, 11.58%.



4ca

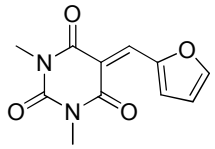
5-(3-methoxybenzylidene)-1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4ca**)[8b]: Yield, 0.233 g (85%). Yellow solid, mp 133-135 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.35 (s, 3H), 3.40 (s, 3H), 3.85 (s, 3H), 7.05 (d, 1H, J = 9.0 Hz), 7.33 (t, 1H, J = 7.8 Hz), 7.54 (d, 1H, J = 7.5 Hz), 7.77 (s, 1H), 8.51 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 28.4, 29.0, 55.3, 117.5, 119.3, 126.5, 129.1, 133.6, 151.1, 159.0, 160.2, 162.4; IR (KBr)(ν_{max}/ cm^{-1}) 1736, 1695, 1667, 1620, 1533; MS (ESI): *m*/*z* calcd for $C_{14}H_{14}N_2O_4$: 274.10; found 275.0 $[M + H]^+$, 297.0 $[M + Na]^+$; anal. calcd for $C_{14}H_{14}N_2O_4$: C, 61.31; H, 5.14; N, 10.21. Found: C, 61.53; H, 5.01; N, 10.49%.



4cb

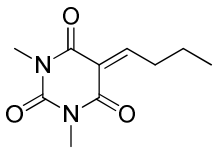
1,3-Dimethyl-5-(4-nitrobenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4cb**): Yield, 0.245 g (85%). White solid, mp 198-201 °C (from EtOH). ^{1}H NMR (300 MHz, $CDCl_3$): δ

(ppm) 3.35 (s, 3H), 3.44 (s, 3H), 7.93 (d, 2H, J = 8.7 Hz), 8.27 (d, 2H, J = 8.7 Hz), 8.57 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 28.5, 29.1, 120.7, 123.1, 132.1, 139.0, 148.9, 150.8, 155.3, 159.7, 161.4; IR (KBr)(ν_{max}/ cm^{-1}) 1733, 1690, 1665, 1621,1531; MS (ESI): m/z calcd for $C_{13}H_{11}N_3O_5$: 289.07; found 289.9 $[M + H]^+$, 311.9 $[M + Na]^+$; anal. calcd for $C_{13}H_{11}N_3O_5$: C, 53.98; H, 3.83; N, 14.53. Found: C, 54.21; H, 3.67; N, 14.71%.



4cc

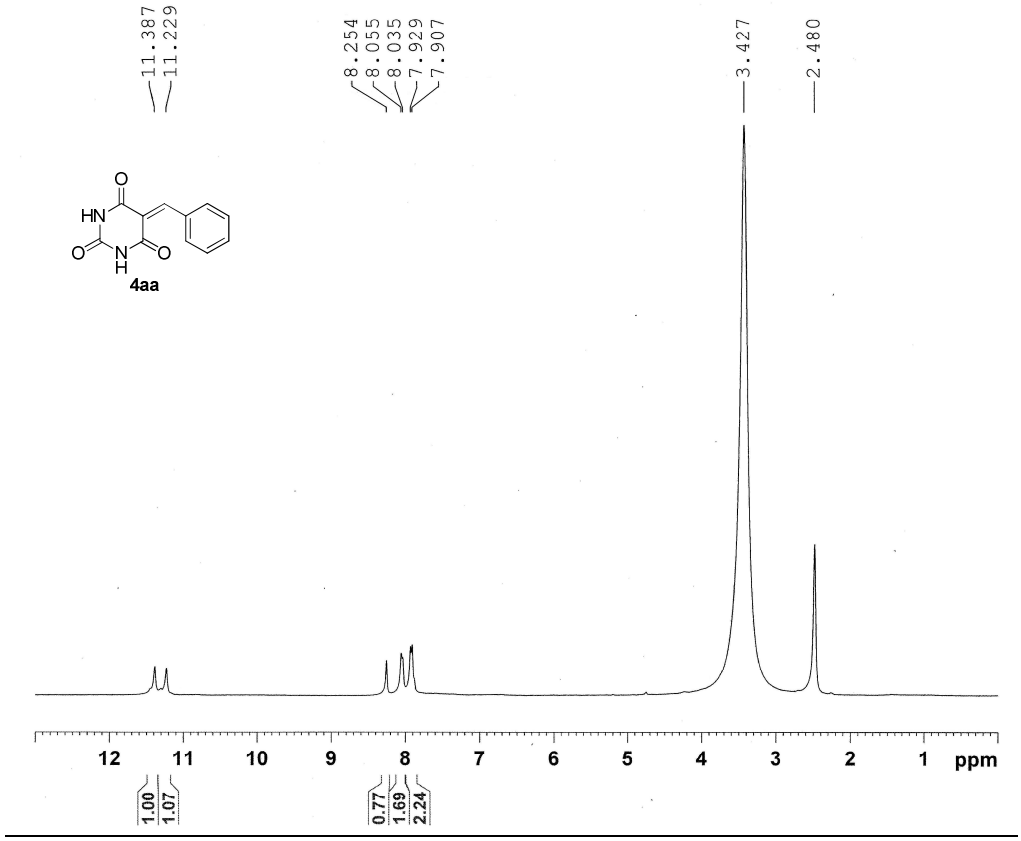
5-(Furan-2-ylmethylene)-1,3-dimethylpyrimidine-2,4,6-(1*H*,3*H*,5*H*)-trione (**4cc**)[16a]: Yield, 0.182 g (78%). Red solid, mp 201-203 °C (from EtOH). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 3.39 (s, 3H), 3.40 (s, 3H), 6.73-6.74 (m, 1H), 7.85 (m, 1H), 8.42 (s, 1H), 8.62-8.63 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 28.1, 28.9, 111.2, 115.1, 128.0, 140.9, 150.4, 151.0, 151.3, 160.7, 162.4; IR (KBr)(ν_{max}/ cm^{-1}) 1739, 1695, 1662, 1622; MS (ESI): m/z calcd for $C_{11}H_{10}N_2O_4$: 234.06; found 256.9, $[M + Na]^+$; anal. calcd for $C_{11}H_{10}N_2O_4$: C, 56.41; H, 4.30; N, 11.96. Found: C, 56.63; H, 4.14; N, 11.67%.

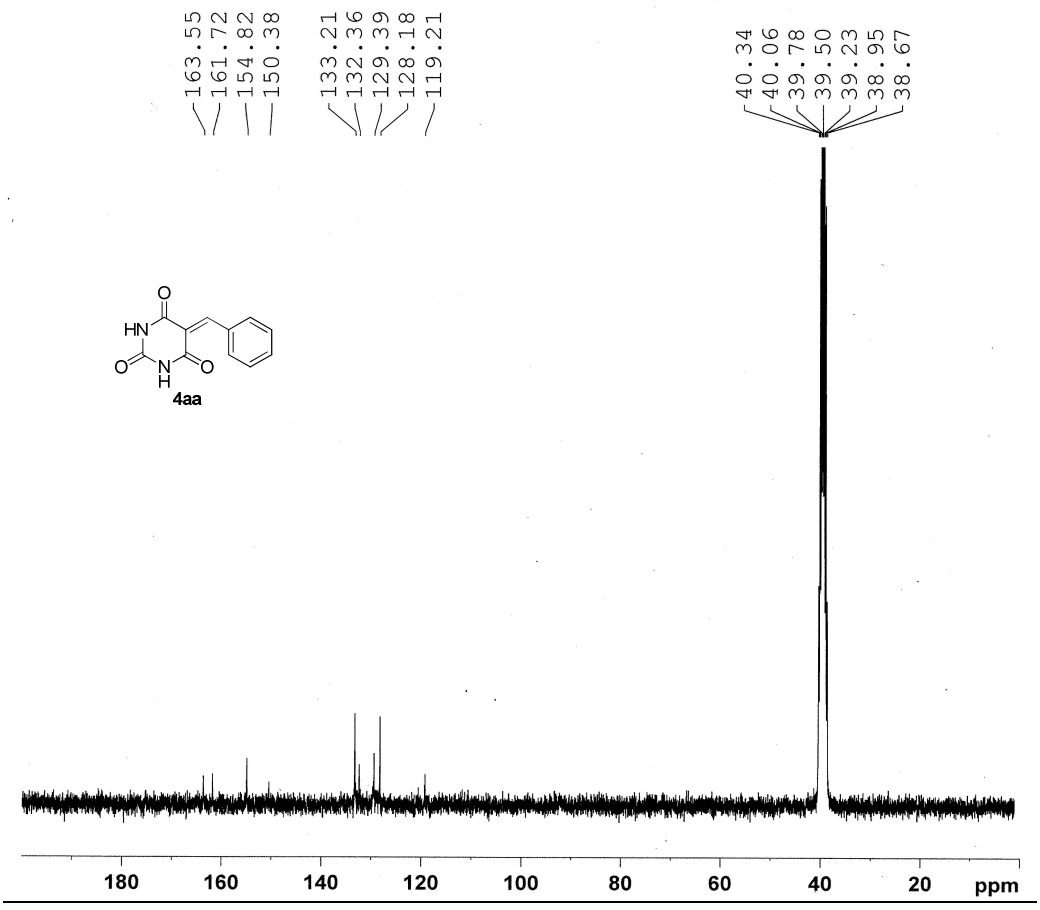


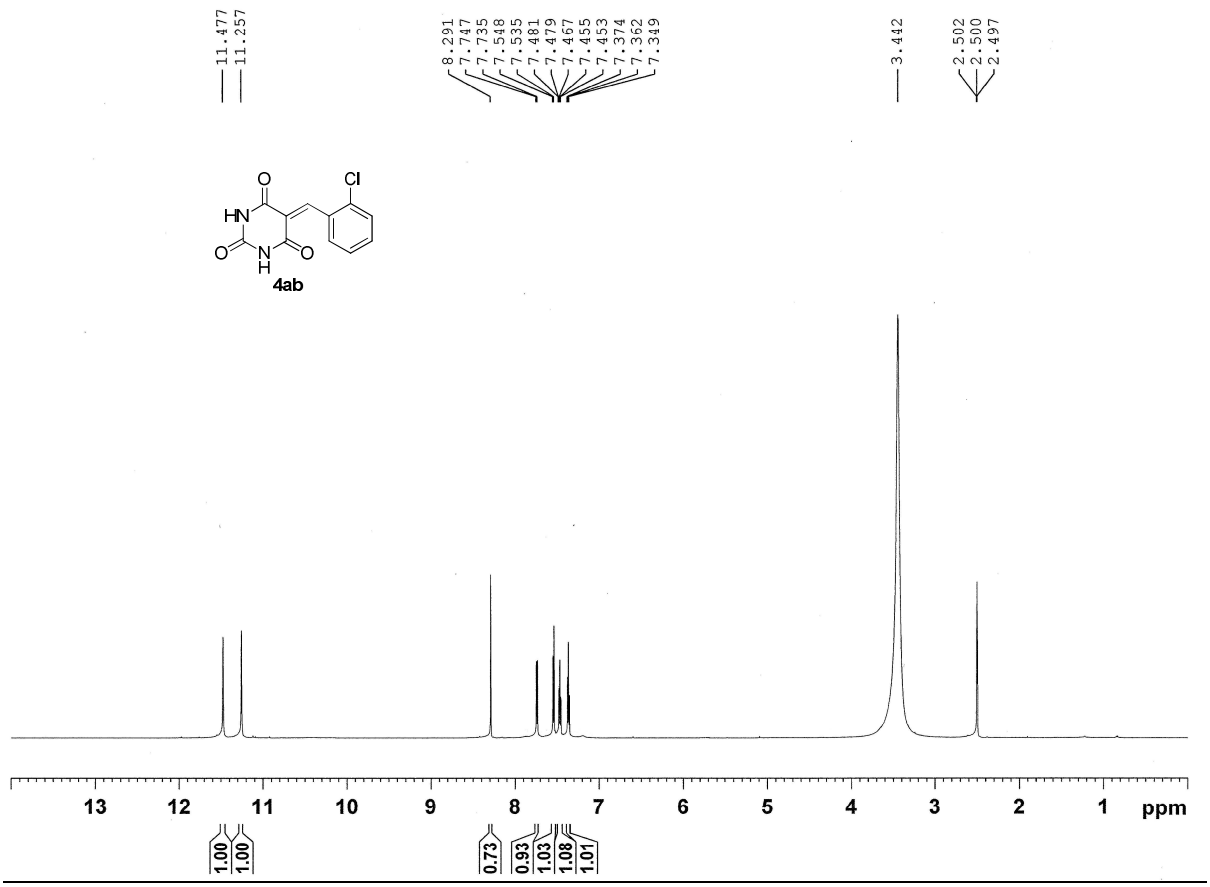
4cd

5-Butylidene-1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (**4cd**): Yield, 0.149 g (71%). Red solid, mp 143-145 °C (from EtOH). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 0.87-0.98 (m, 3H), 1.36-1.48 (m, 2H), 1.61-1.67 (m, 2H), 3.31 (s, 6H), 4.83-4.86 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 11.1, 13.7, 20.9, 28.6, 125.6, 150.7, 151.0, 163.3, 164.8; IR (KBr)(ν_{max}/ cm^{-1}) 3220, 3136, 2924, 1747, 1680, 1669, 1629; MS (ESI): m/z calcd for $C_{10}H_{14}N_2O_3$: 210.10; found 232.9 $[M + Na]^+$; anal. calcd for $C_{10}H_{14}N_2O_3$: C, 57.13; H, 6.71; N, 13.33. Found: C, 57.34; H, 6.54; N, 13.61%.

2. ^{1}H- and ^{13}-C NMR Spectra of 4aa-4an and 4ba-4bh.



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7.735
7.548
7.535
7.481
7.479
7.467
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7.453
7.374
7.362
7.349
3.442
2.502
2.500
2.497
Cl
HN
O
N
H
4ab
13
12
11
10
9
8
7
6
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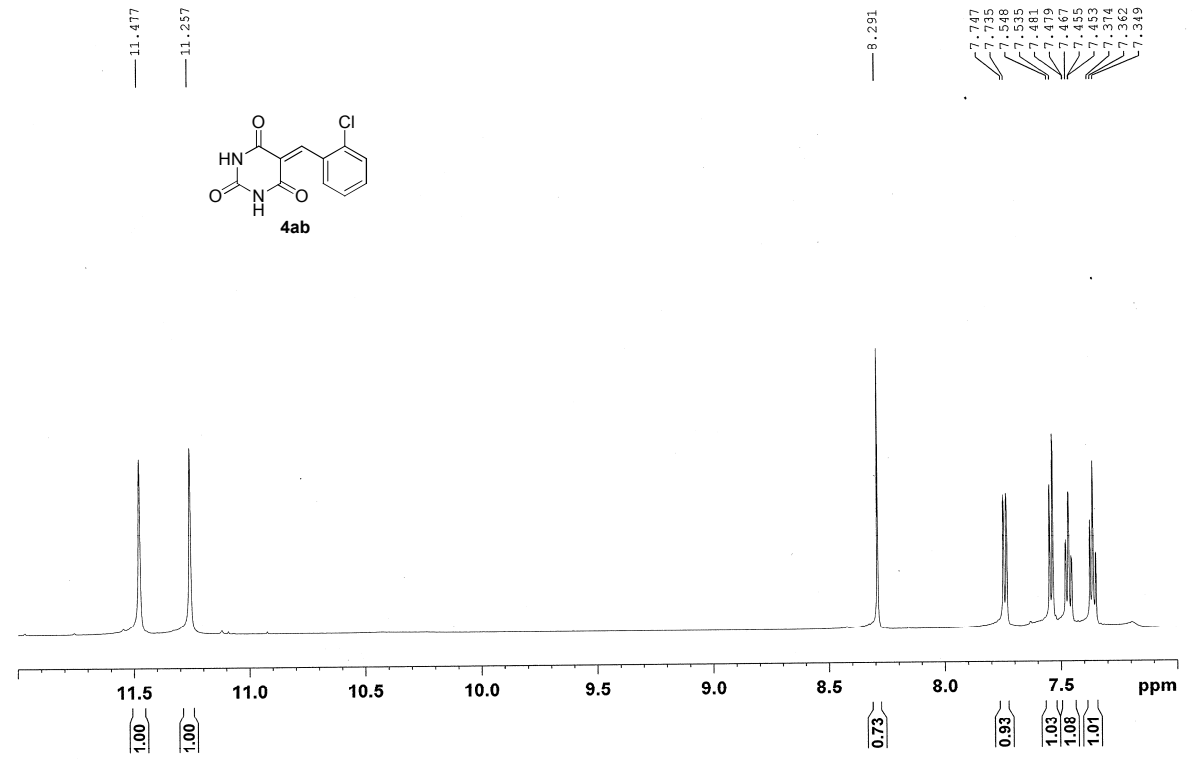
1.01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8.291
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7.349
Cl
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O
N
H
4ab
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10.0
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8.0
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ppm
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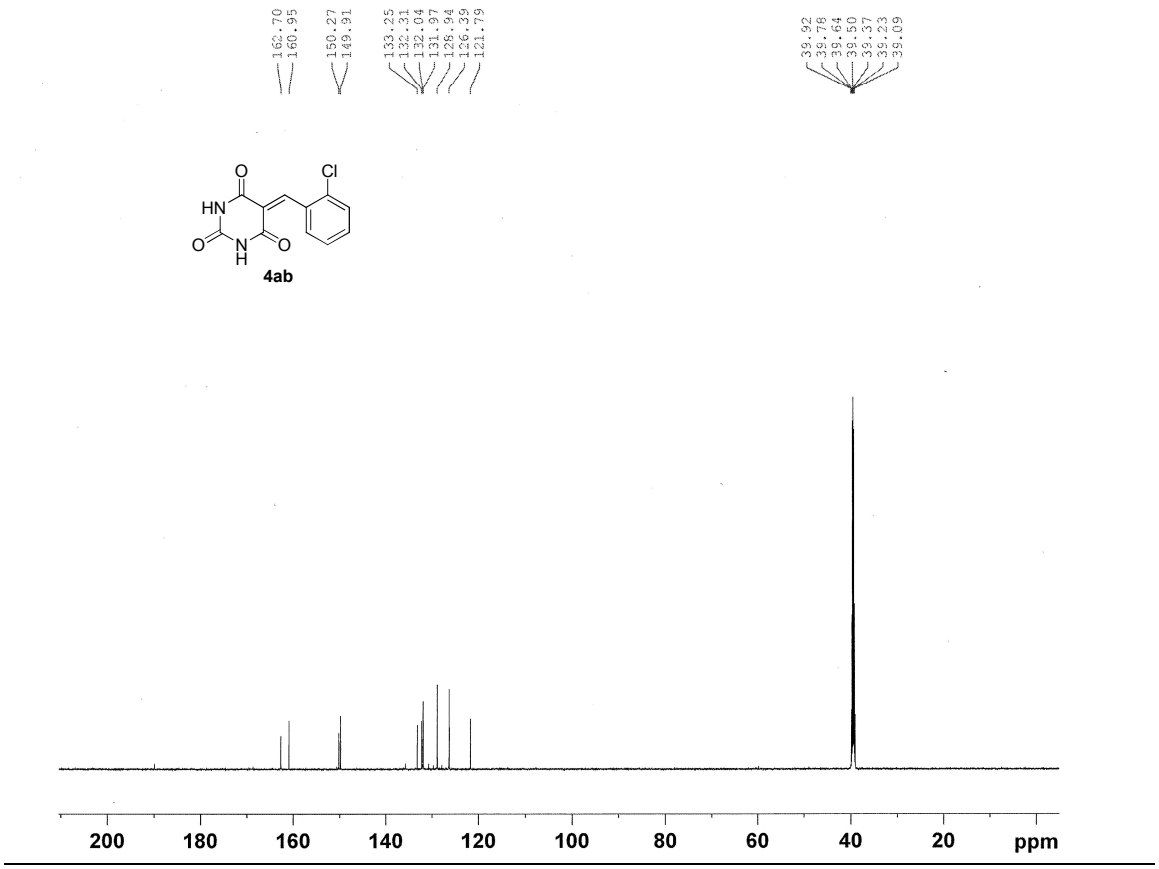
1.08
1.01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39.64
39.50
39.37
39.23
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N
H
O
Cl
4ab
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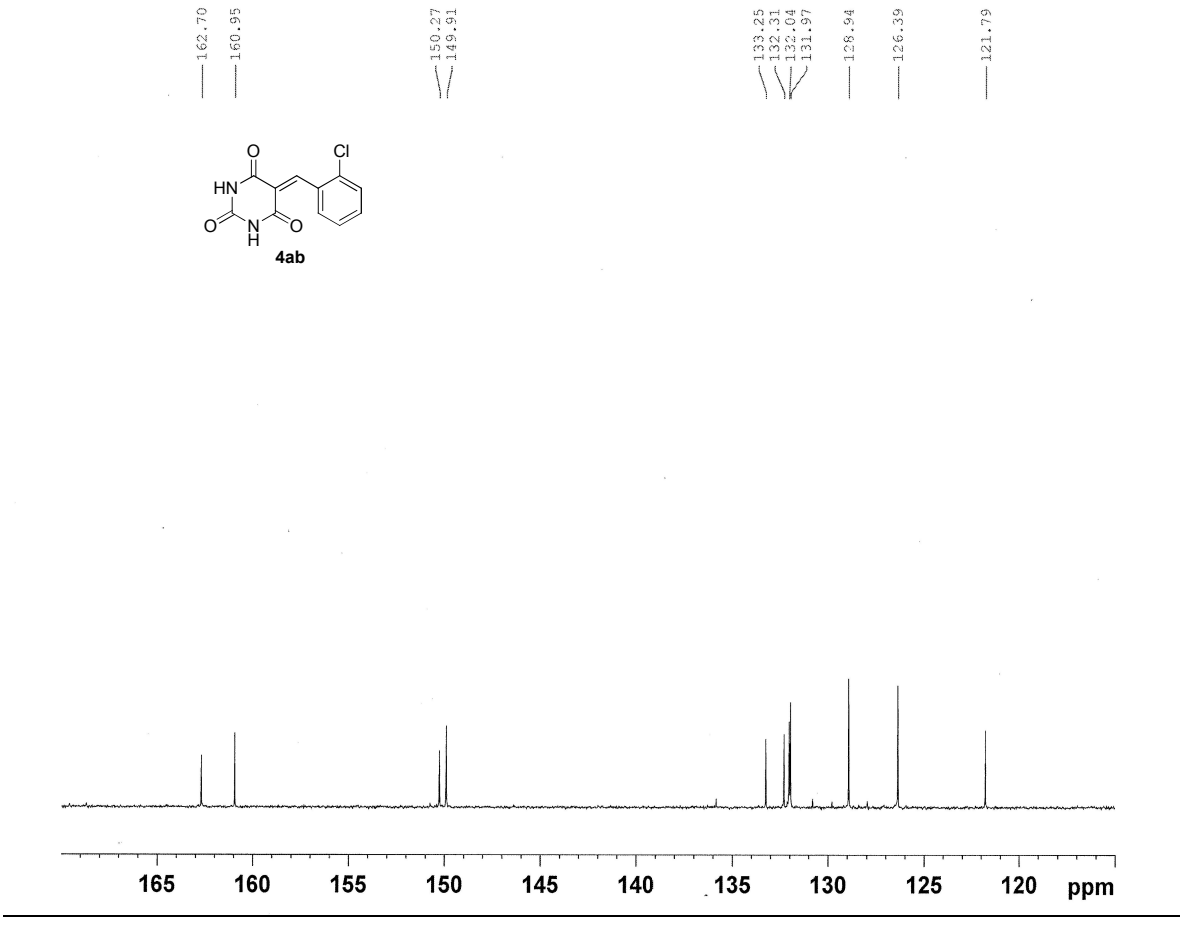
140
120
100
80
60
40
20
ppm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149.91
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N
H
O
Cl
4ab
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150
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135
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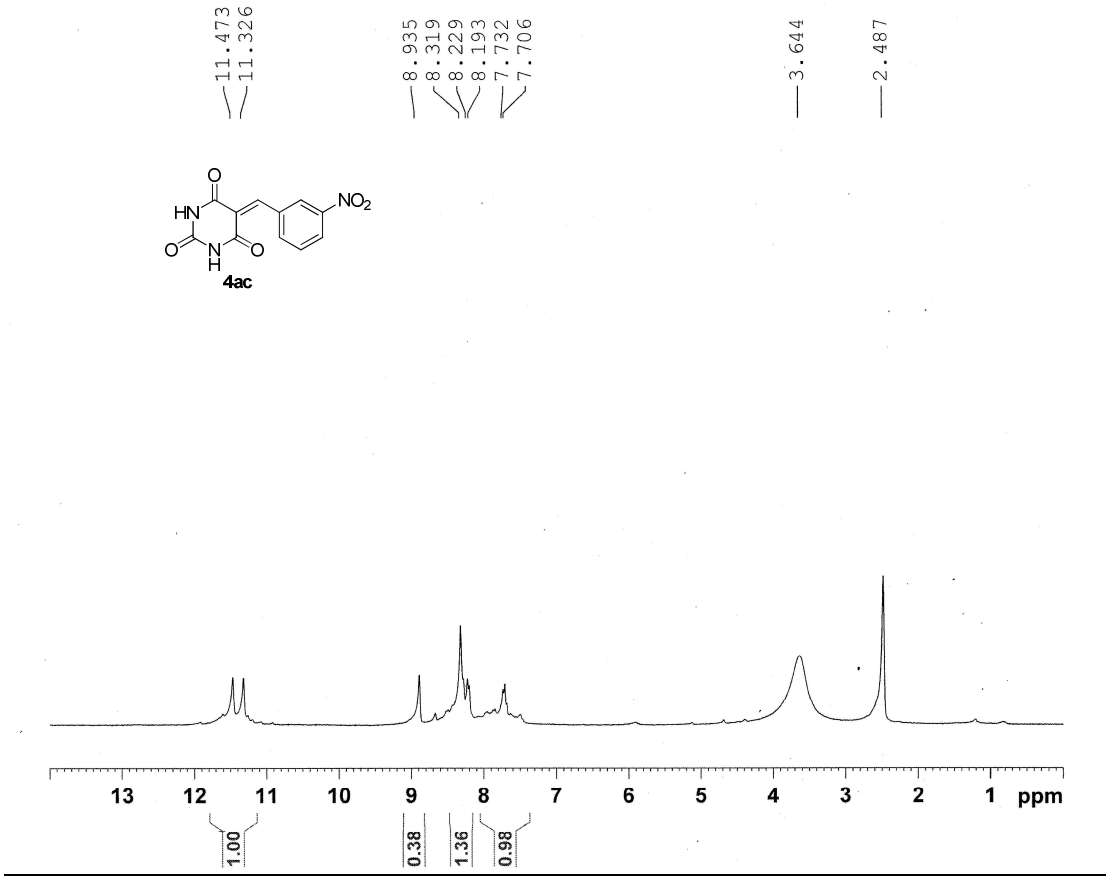
120
ppm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H
4ac
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3
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ppm
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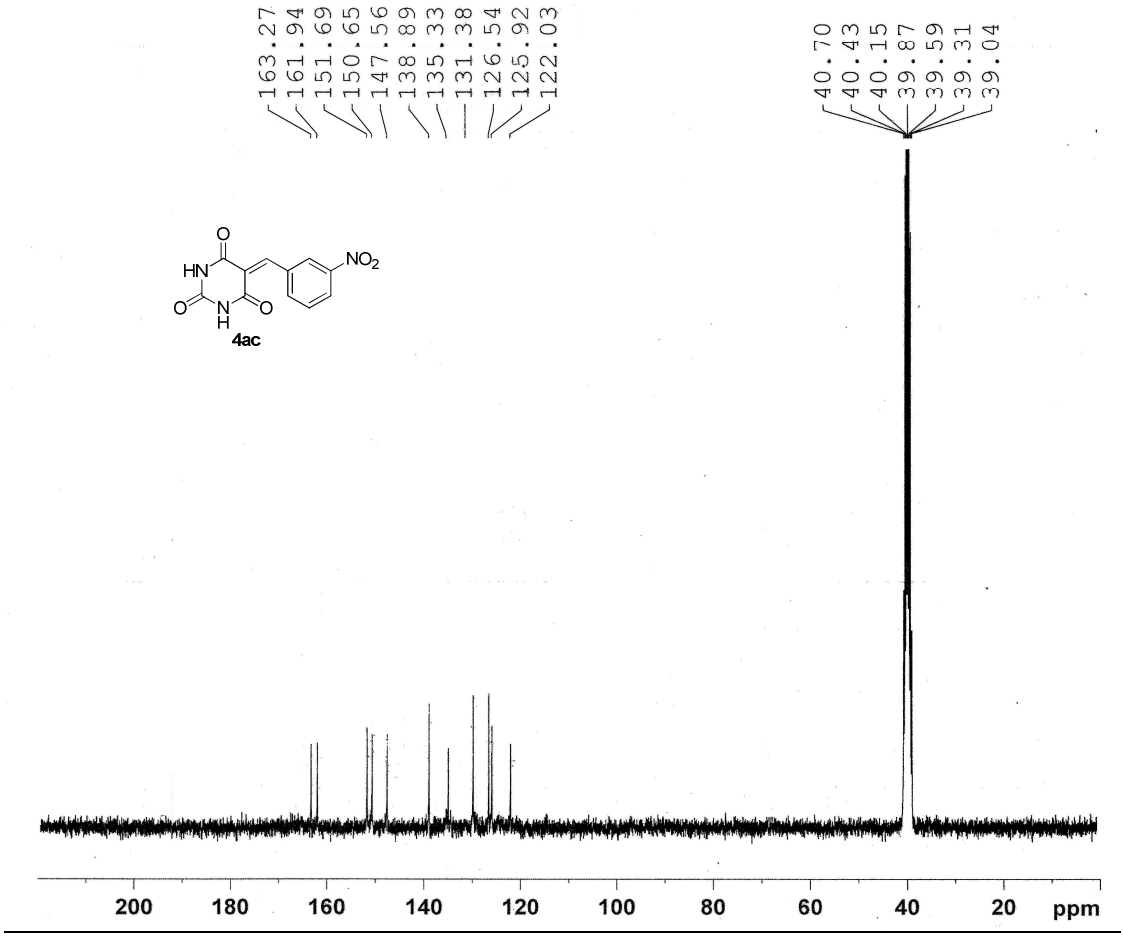
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40.15
39.87
39.59
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HN
O
N
H
4ac
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140
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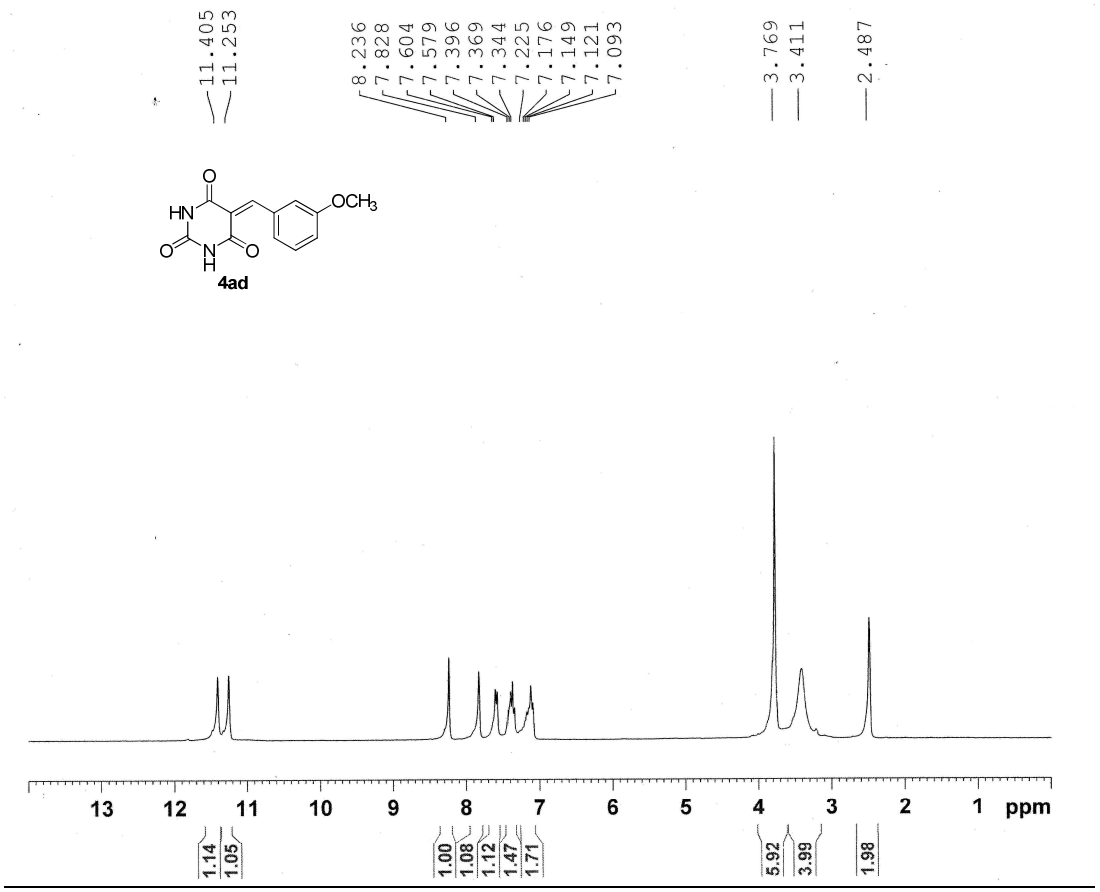
80
60
40
20
ppm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7.121
7.093
3.769
3.411
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O
N
H
4ad
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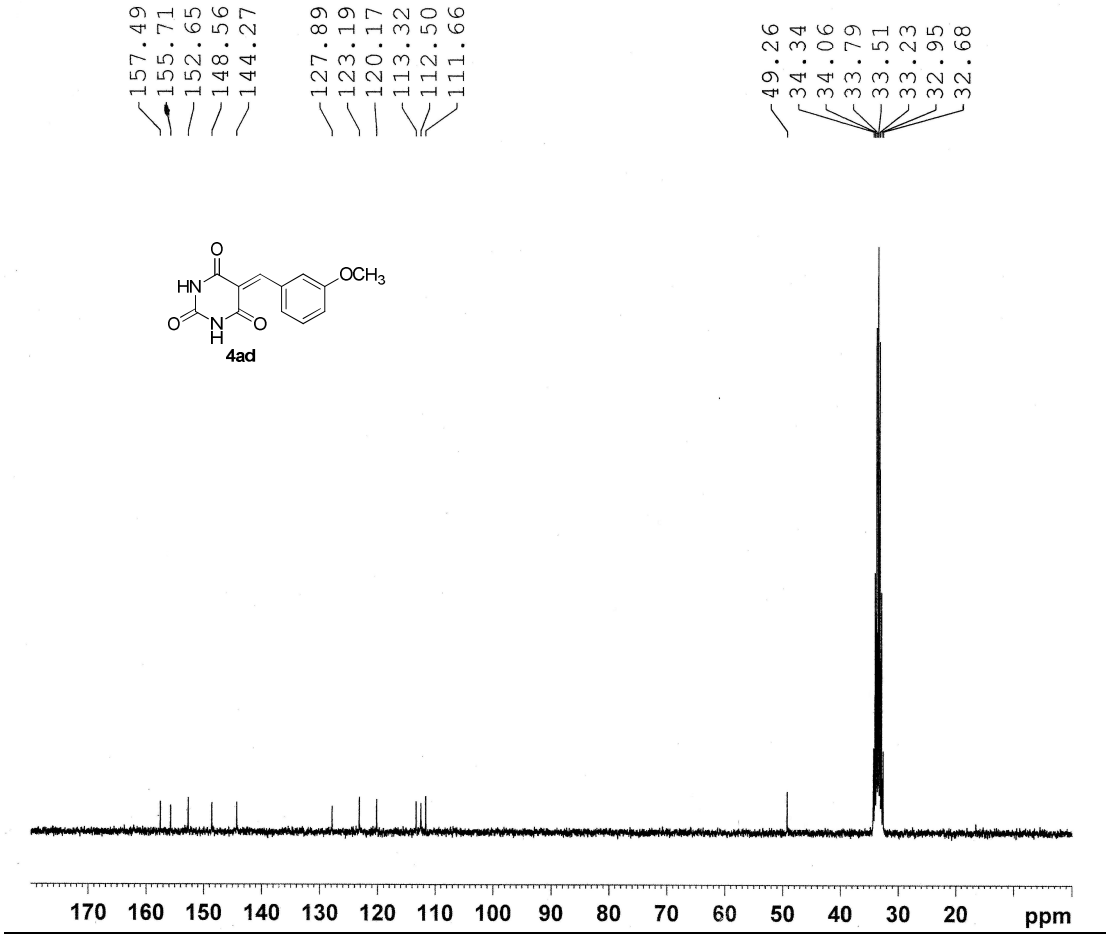
1.98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120.17
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34.34
34.06
33.79
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H
4ad
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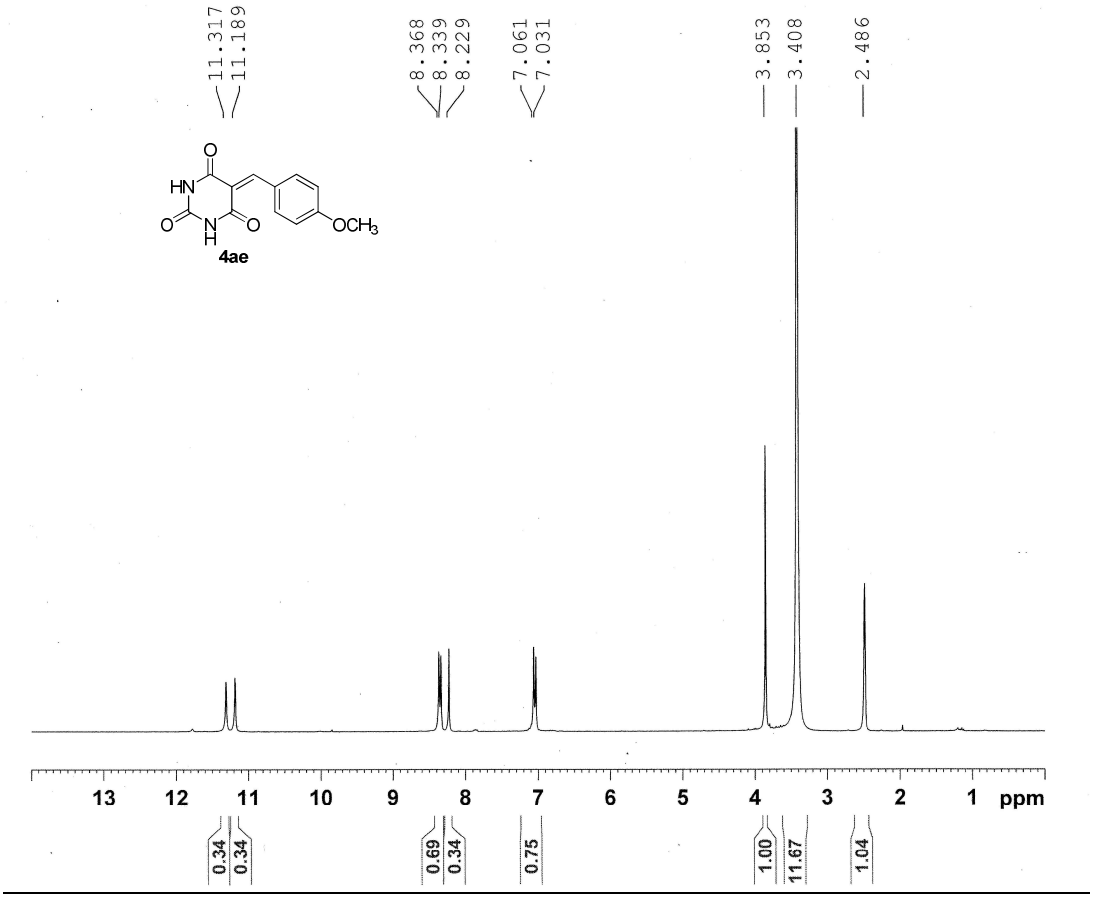
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0.34
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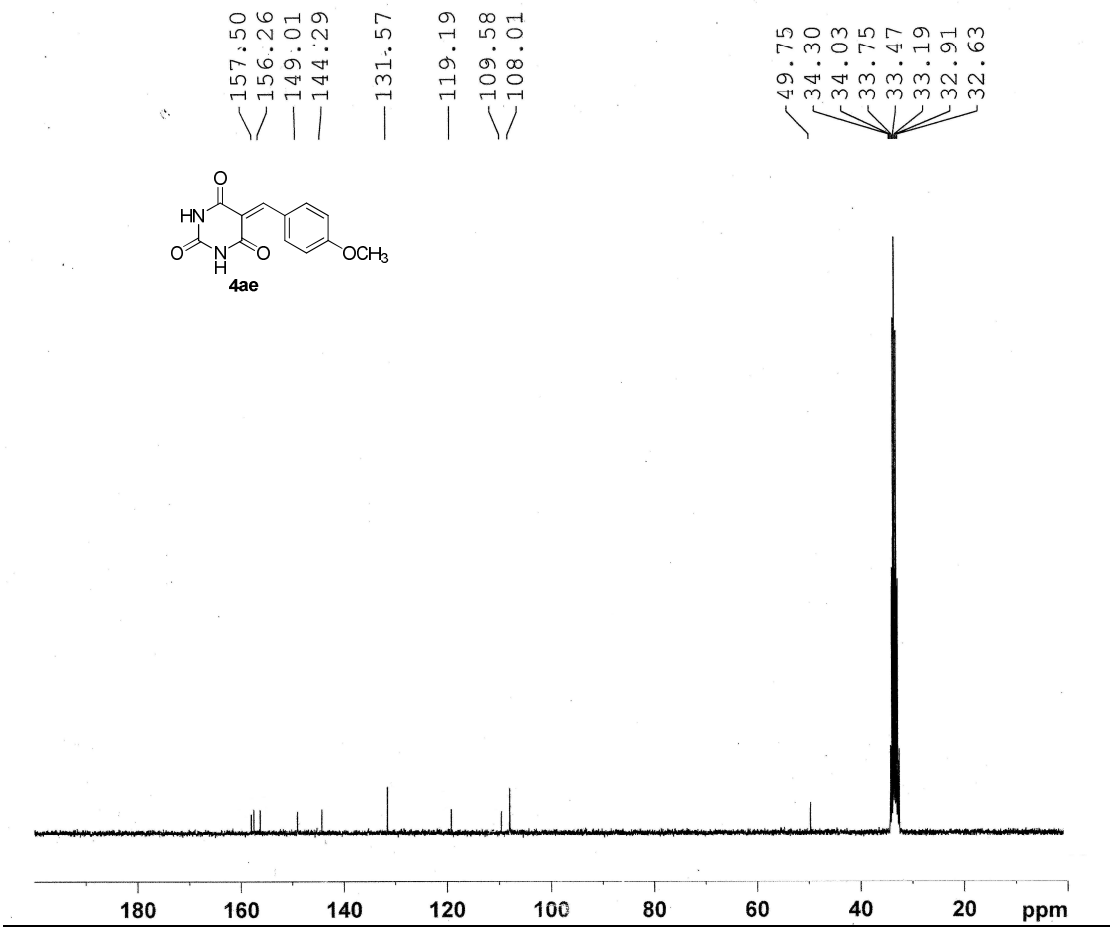
1.00
11.67
1.04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32.91
32.63
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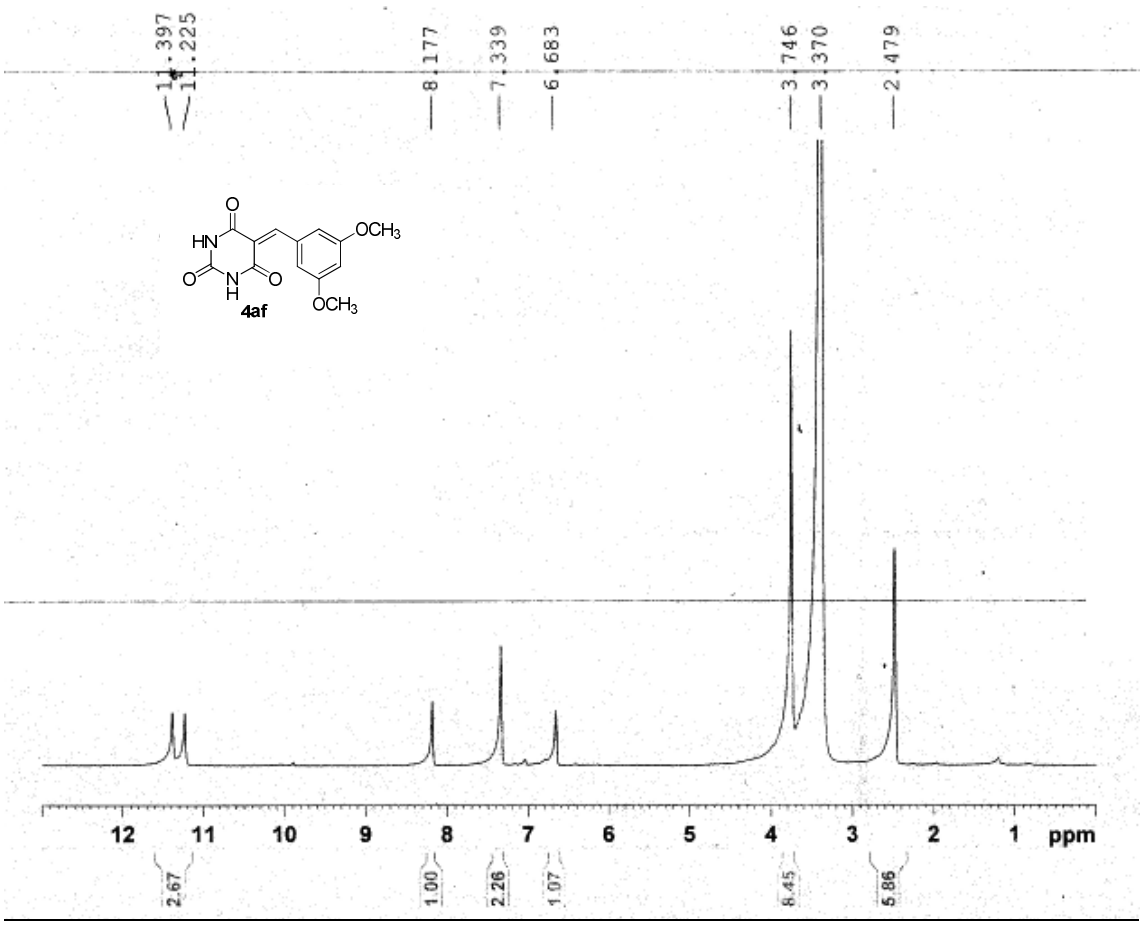
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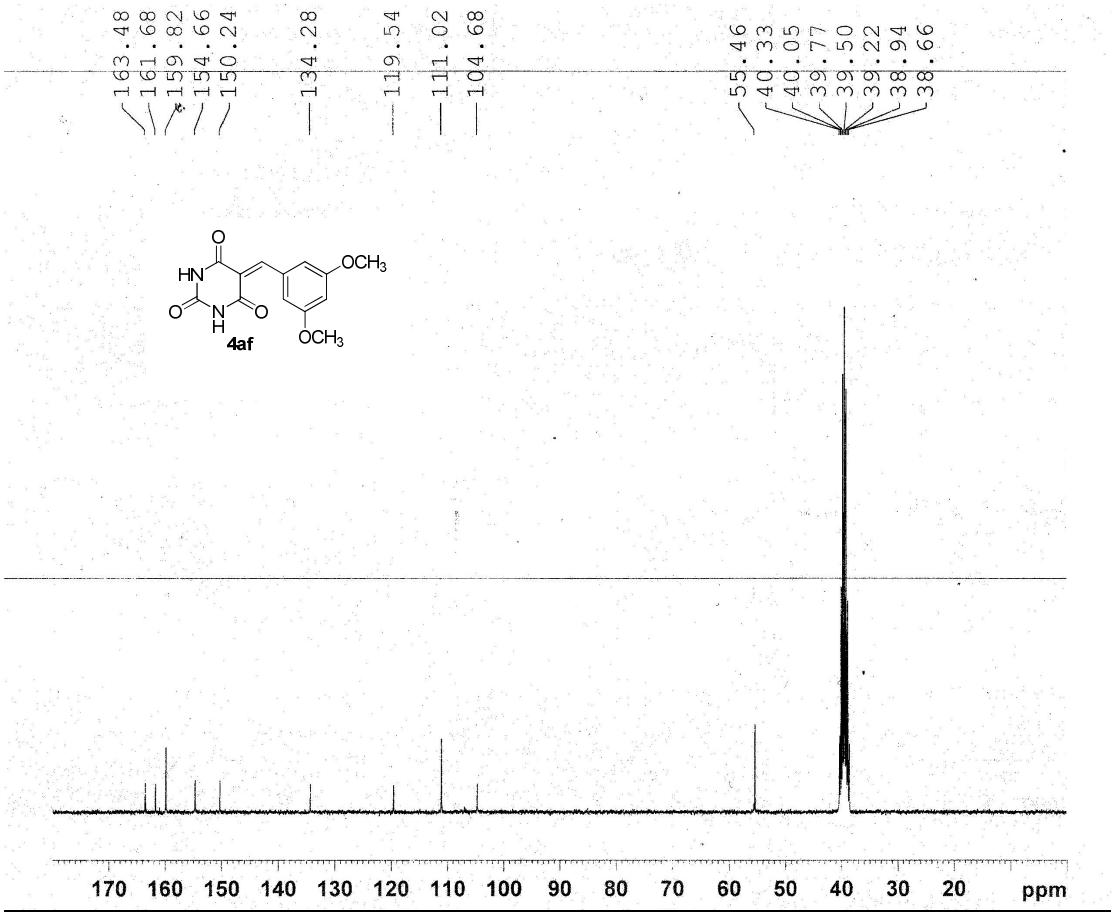
5.86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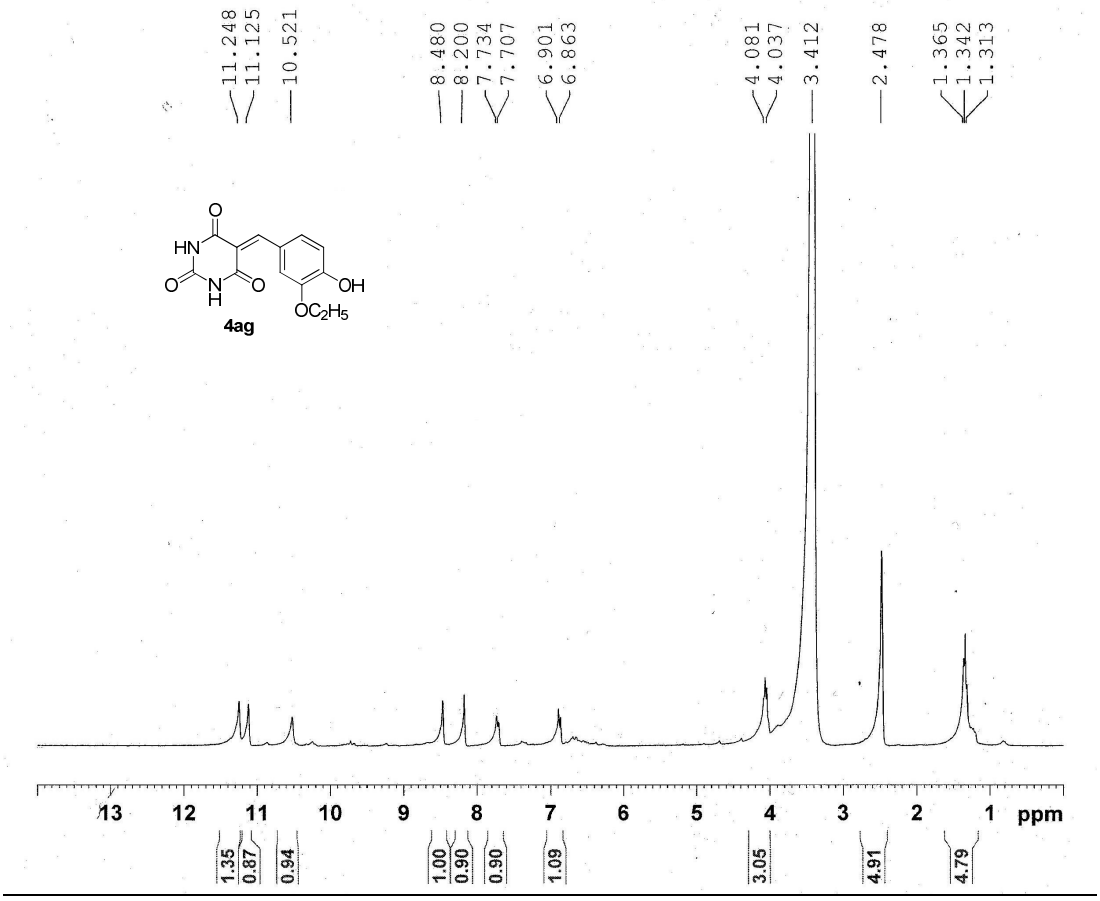
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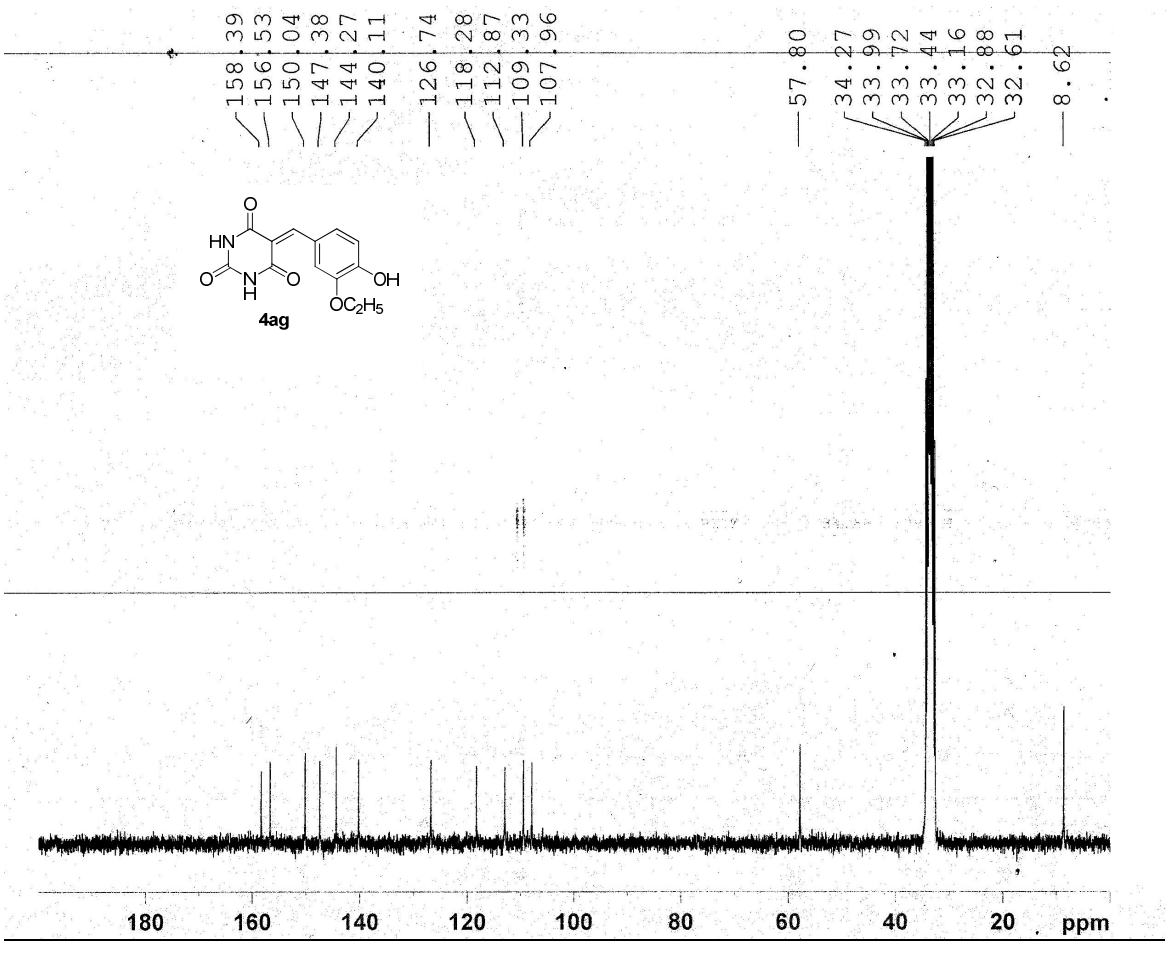
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32.61
8.62
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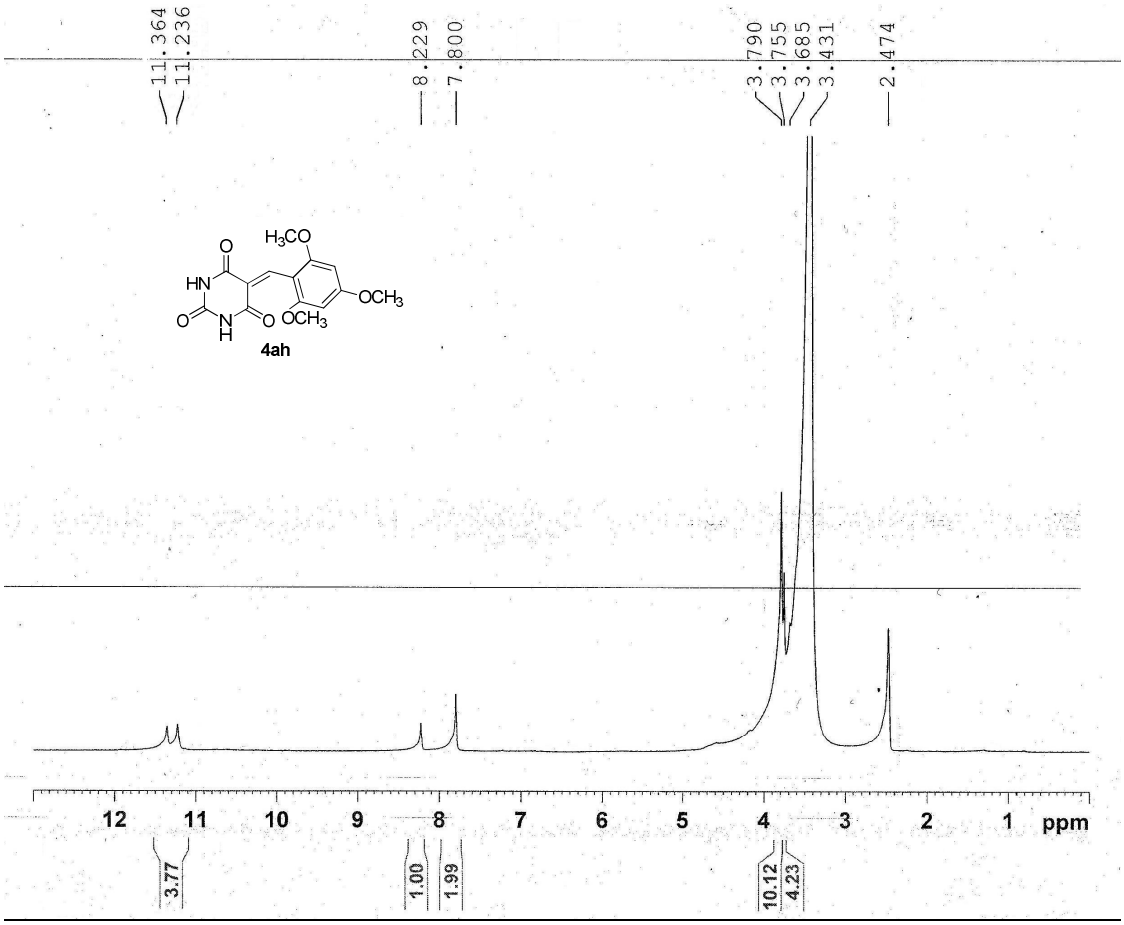
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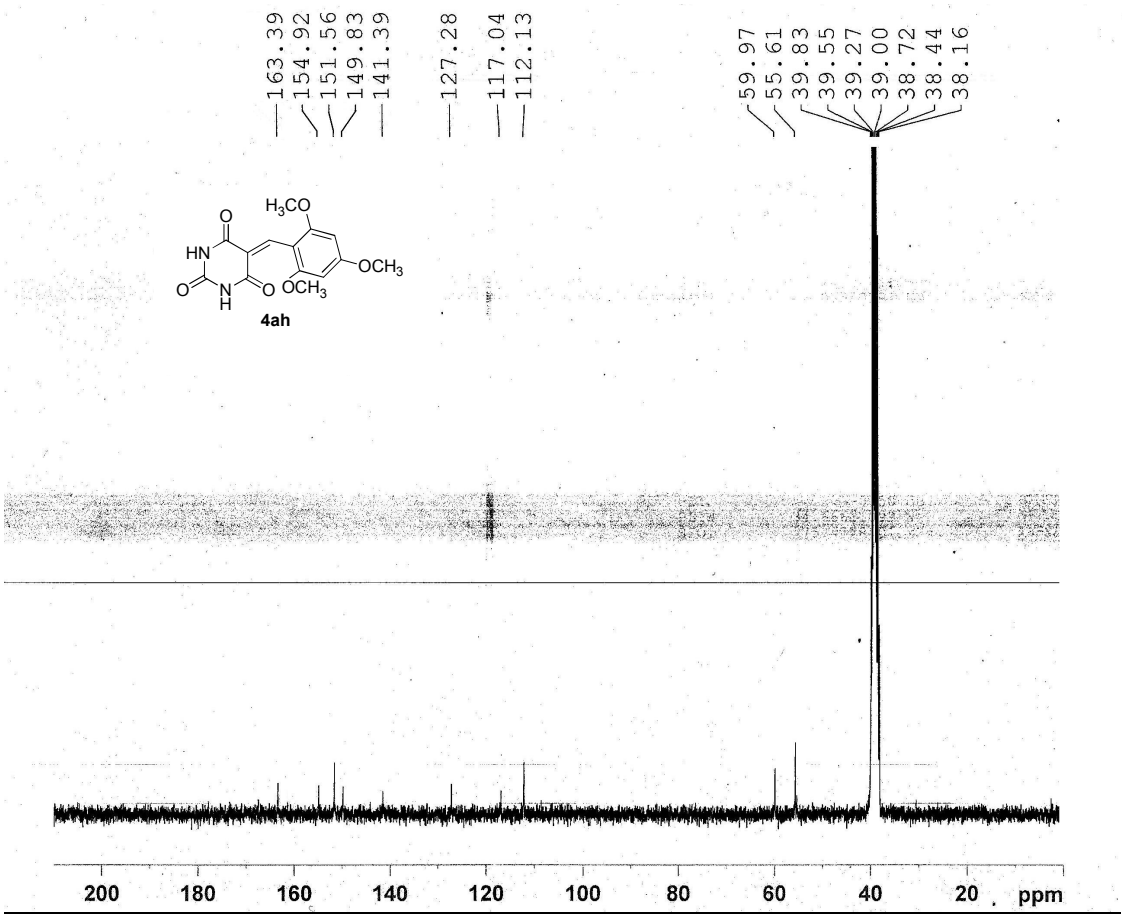
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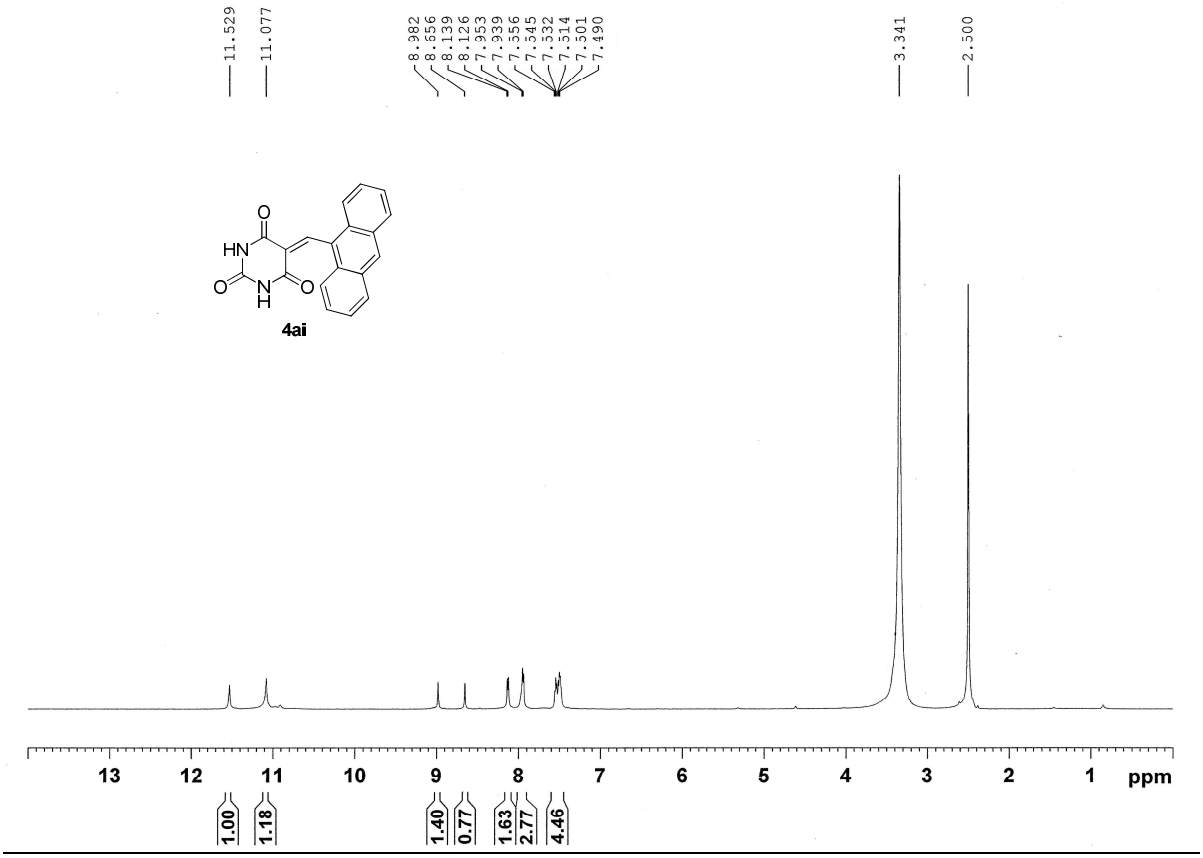
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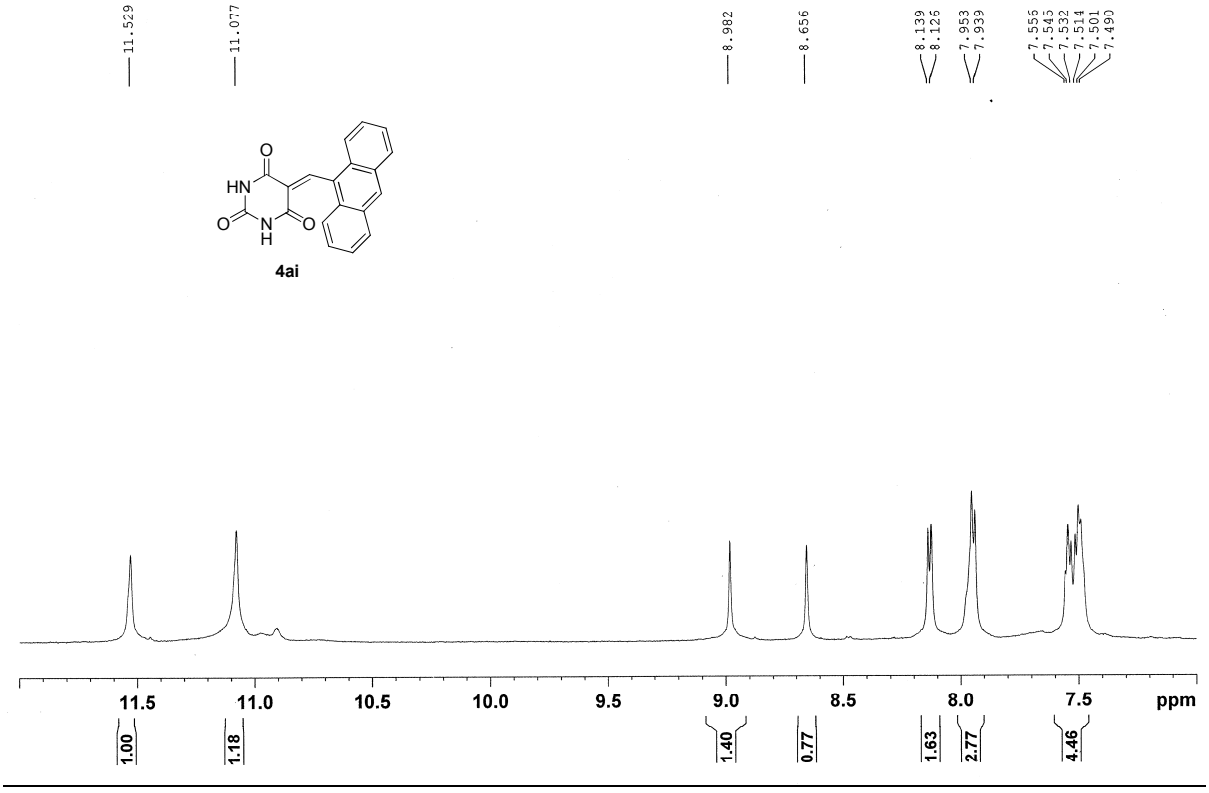
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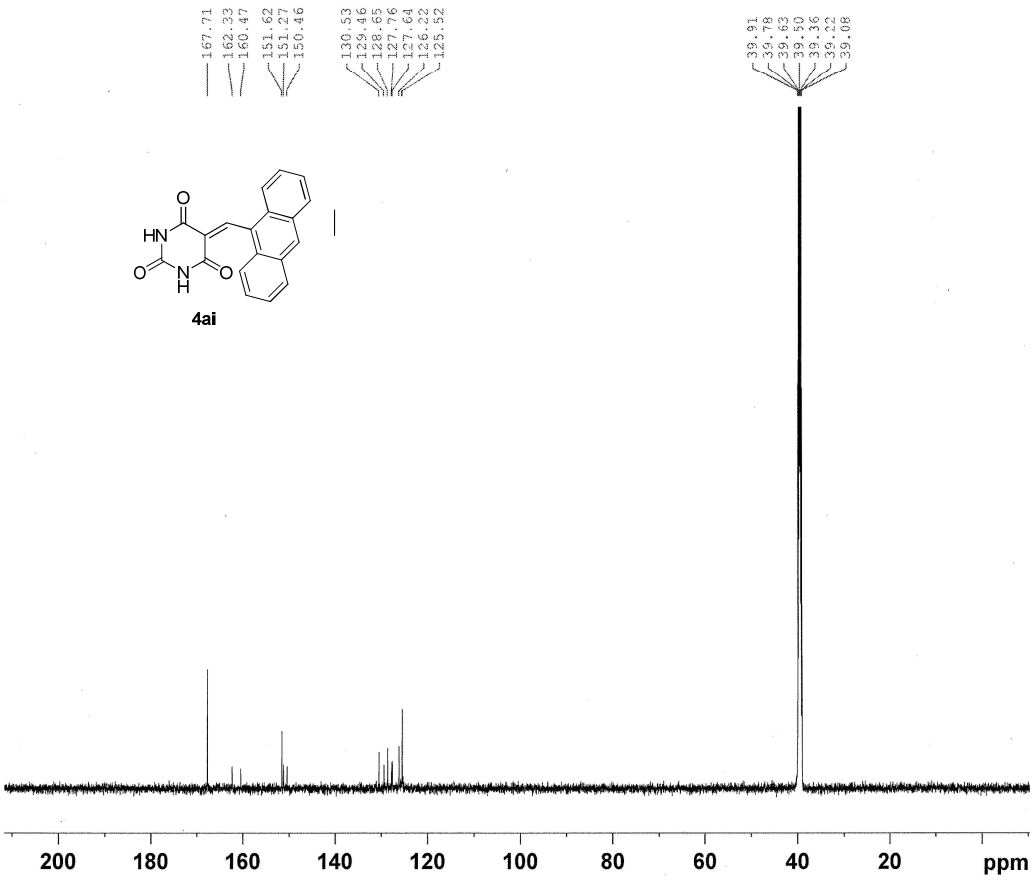
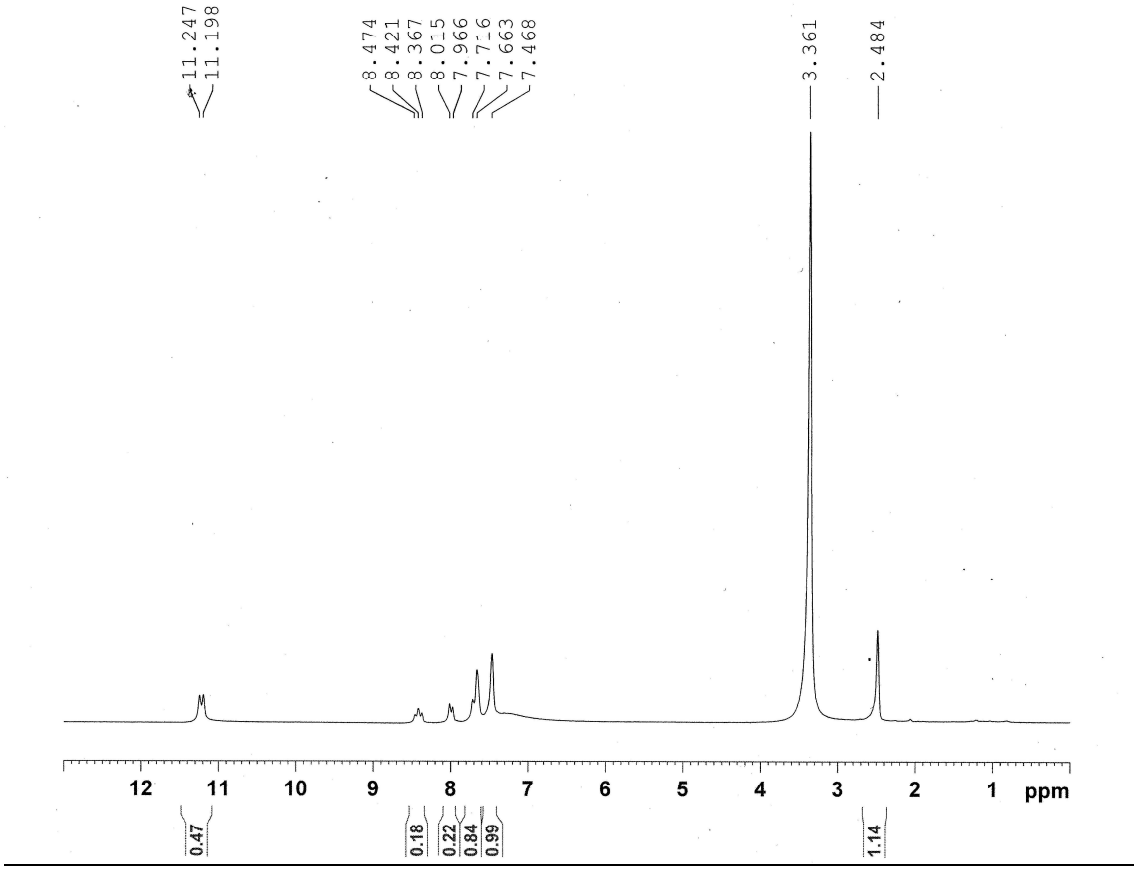
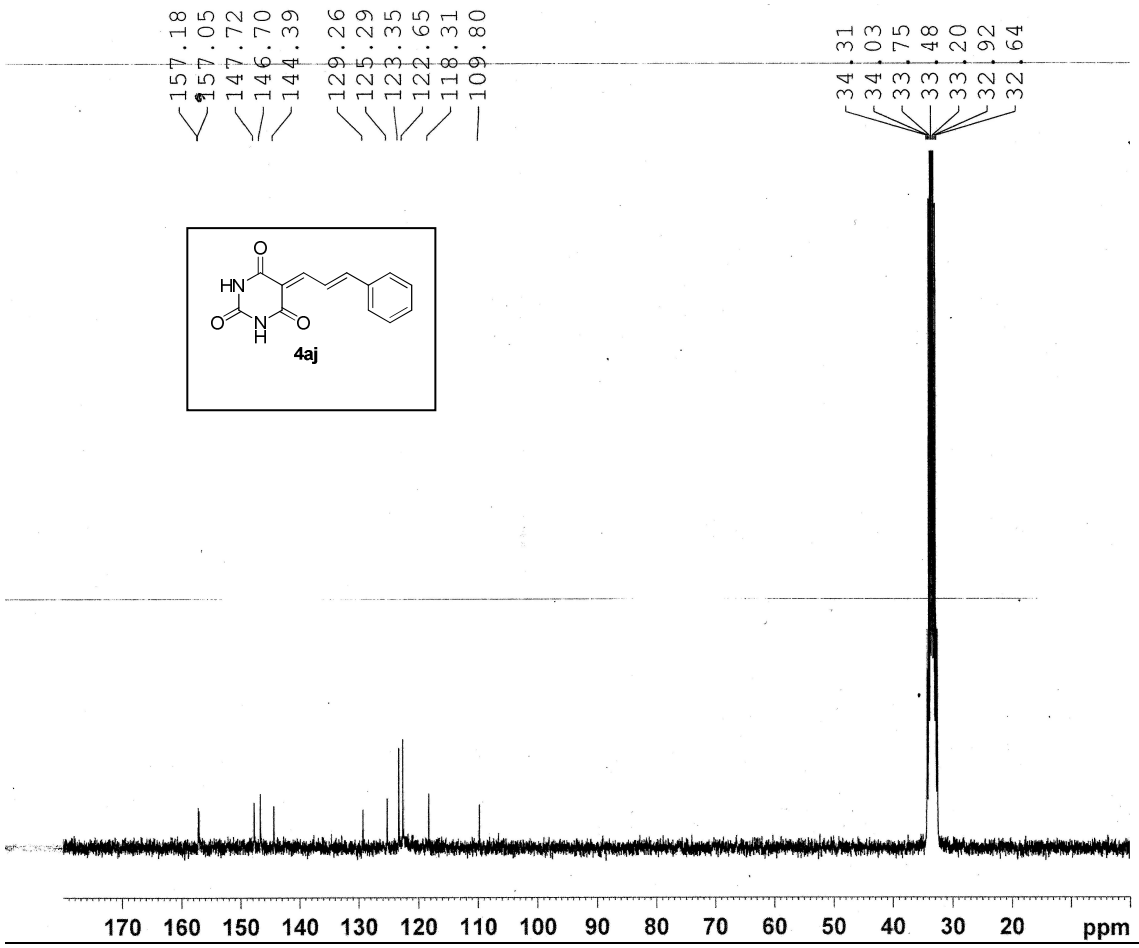
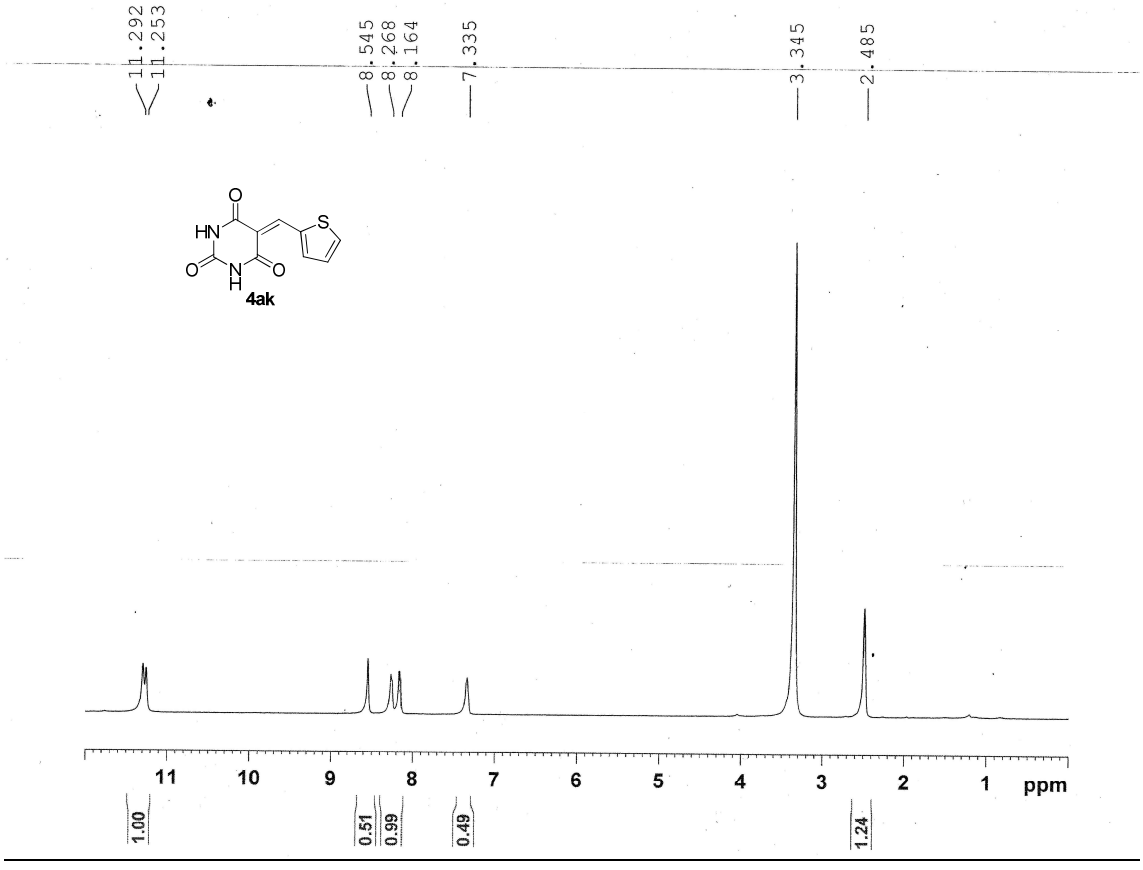
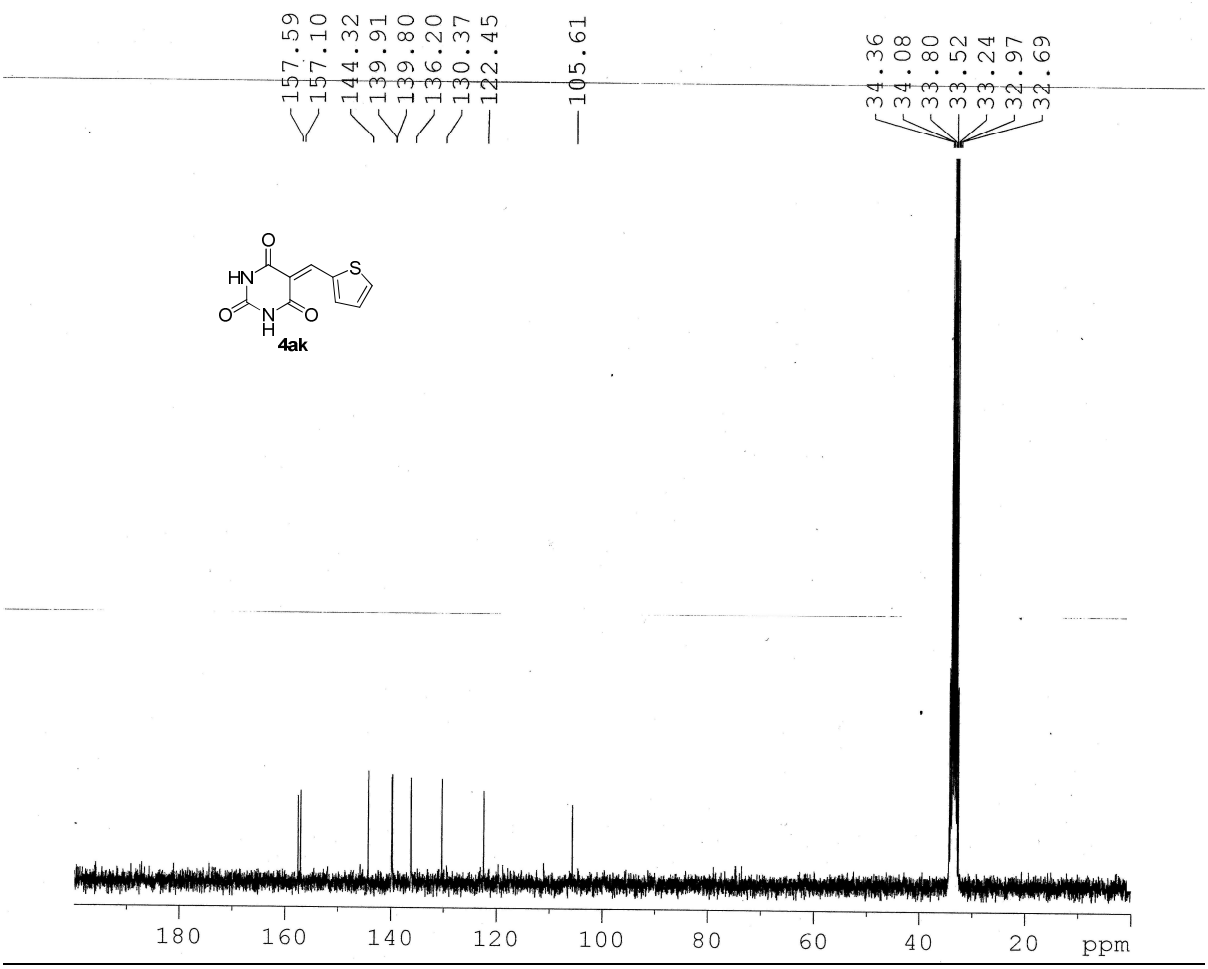
2.77
4.46

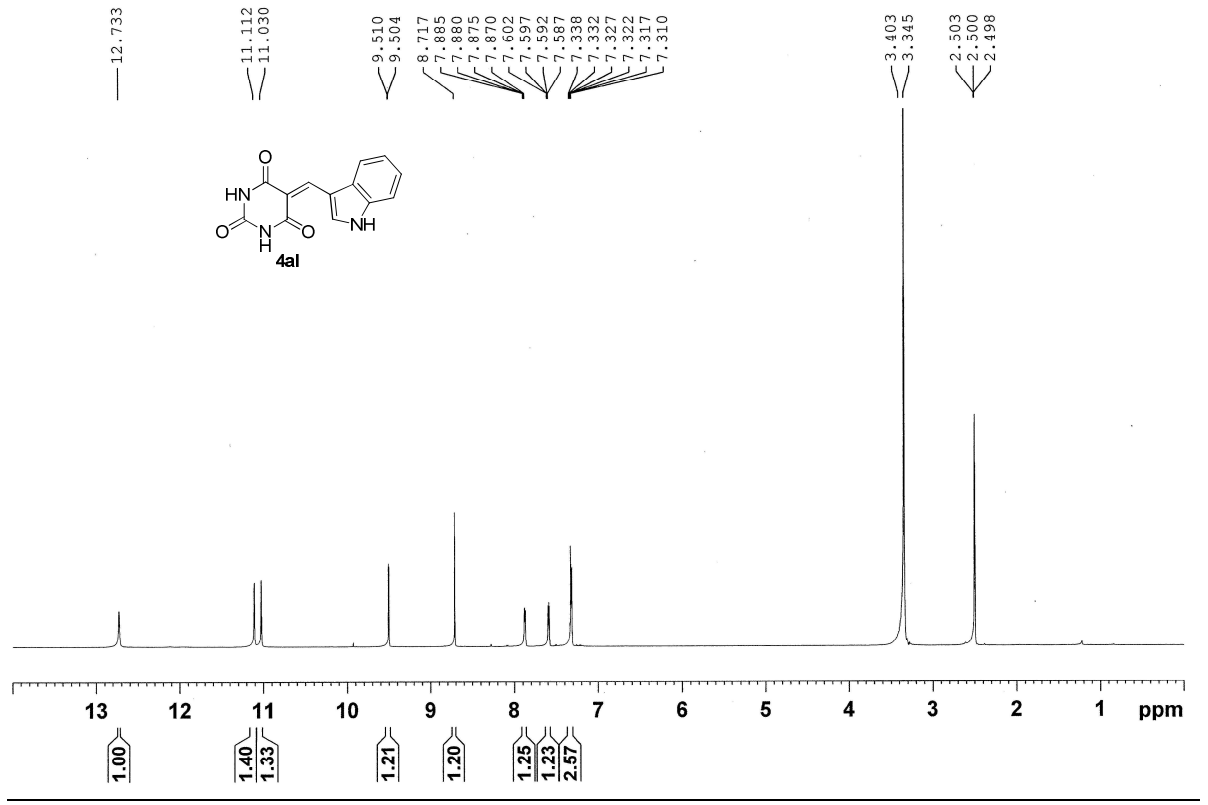

167.71
162.33
160.47
151.62
151.27
150.46
130.53
129.46
128.65
127.76
127.64
126.22
125.52
39.91
39.78
39.63
39.50
39.36
39.22
39.08
4ai
200
180
160
140
120
100
80
60
40
20
ppm

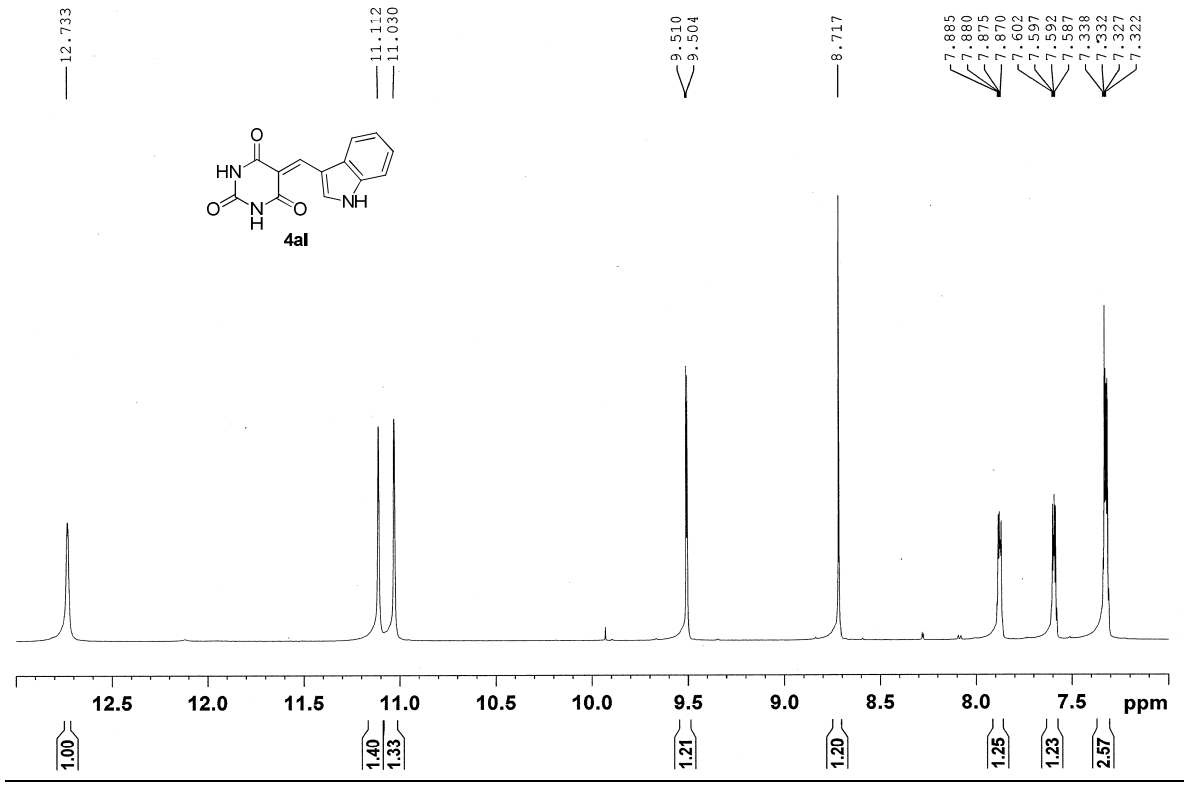

11.247
11.198
8.474
8.421
8.367
8.015
7.966
7.716
7.663
7.468
3.361
2.484
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.47
0.18
0.22
0.84
0.99
1.14

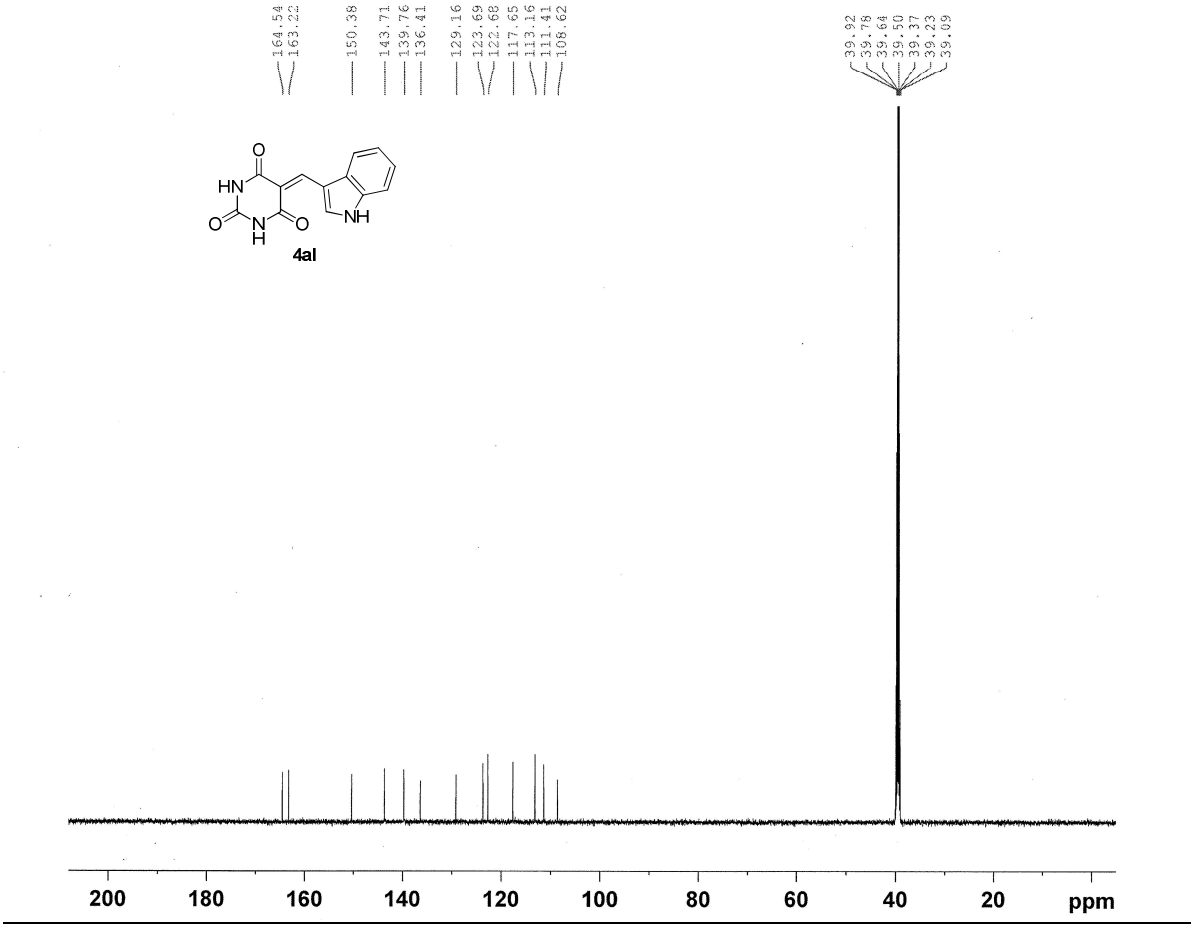

157.18
157.05
147.72
146.70
144.39
129.26
125.29
123.35
122.65
118.31
109.80
34.31
34.03
33.75
33.48
33.20
32.92
32.64
4aj
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

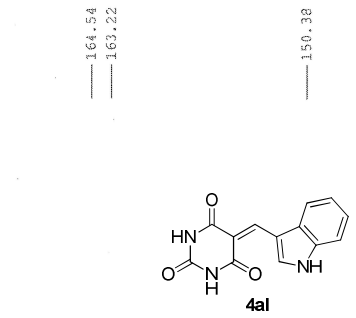

11.292
11.253
8.545
8.268
8.164
7.335
3.345
2.485
4ak
11 10 9 8 7 6 5 4 3 2 1 ppm
1.00
0.51
0.99
0.49
1.24

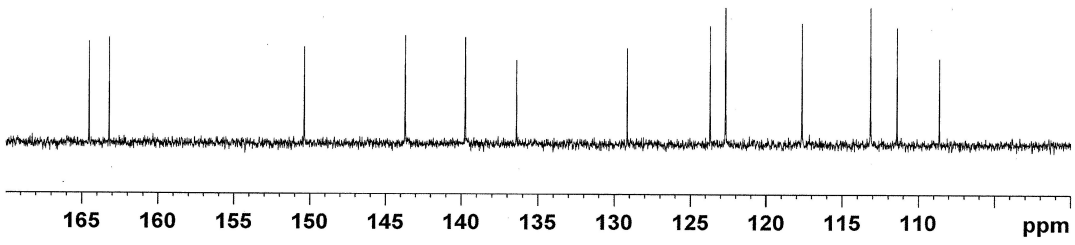

157.59
157.10
144.32
139.91
139.80
136.20
130.37
122.45
105.61
34.36
34.08
33.80
33.52
33.24
32.97
32.69
4ak
180
160
140
120
100
80
60
40
20
ppm

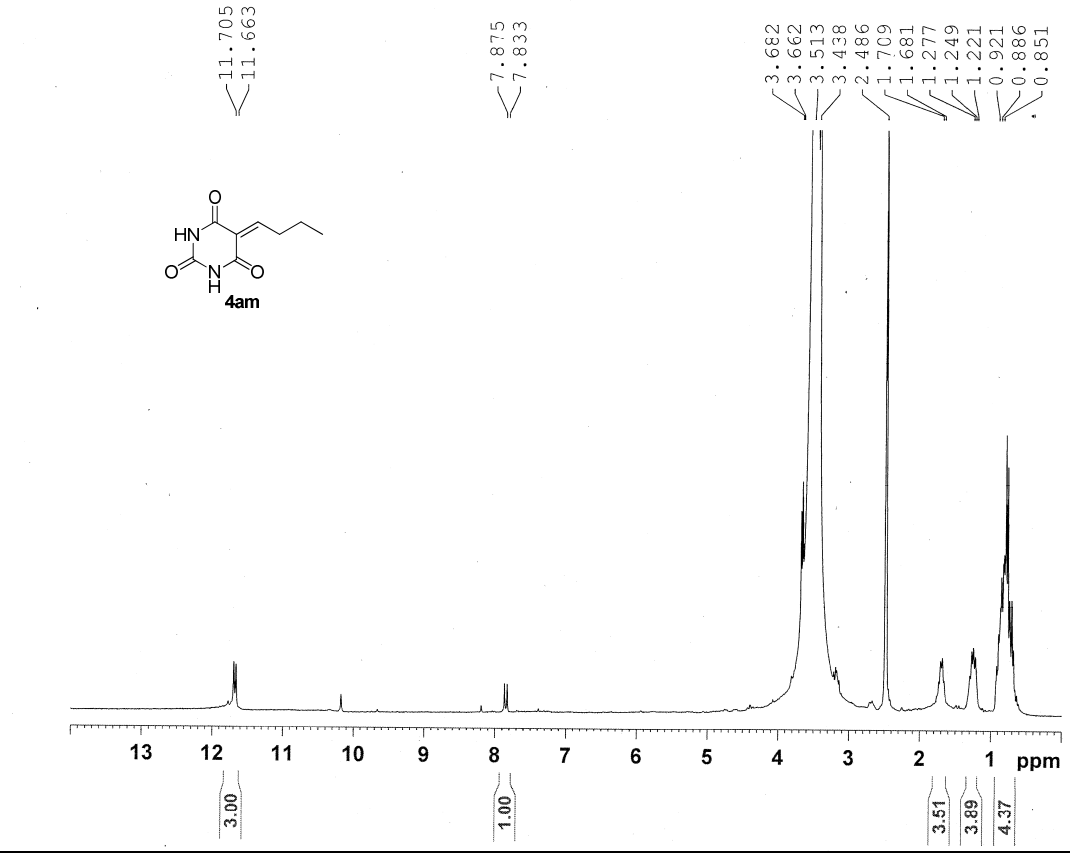

12.733
11.112
11.030
9.510
9.504
8.717
7.885
7.880
7.875
7.870
7.602
7.597
7.592
7.587
7.338
7.332
7.327
7.322
7.317
7.310
3.403
3.345
2.503
2.500
2.498
4al
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.40
1.33
1.21
1.20
1.25
1.23
2.57

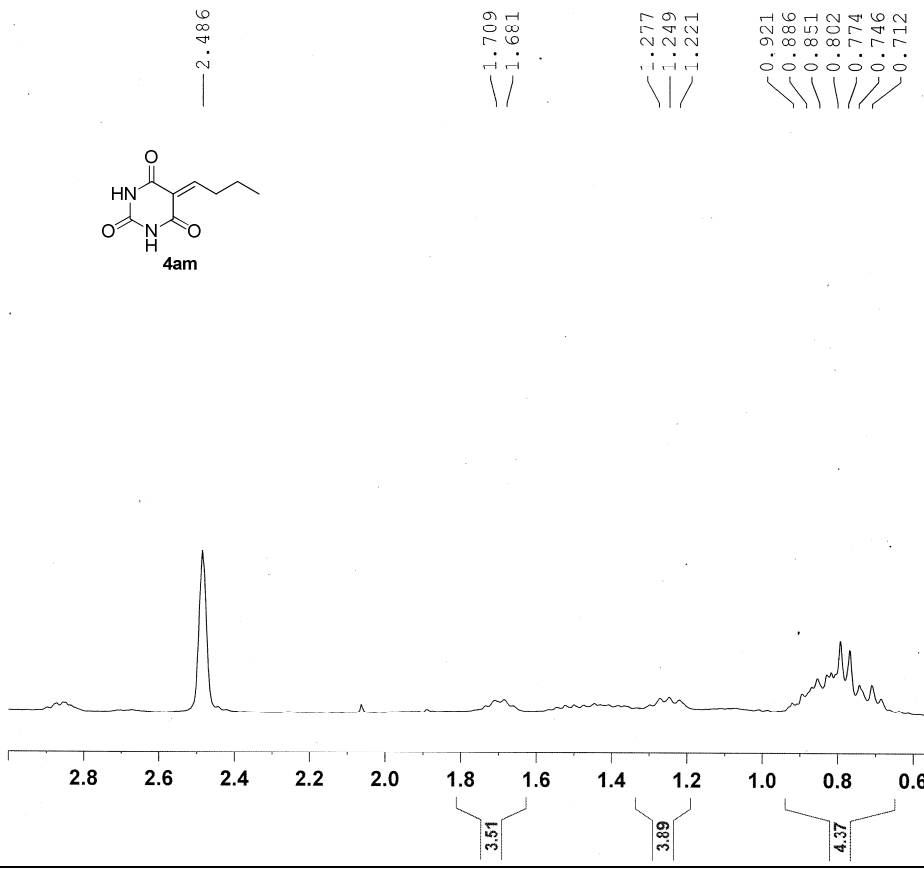

12.733
11.112
11.030
9.510
9.504
8.717
7.885
7.880
7.875
7.870
7.602
7.597
7.592
7.587
7.338
7.332
7.327
7.322
4al
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
ppm
1.00
1.40
1.33
1.21
1.20
1.25
1.23
2.57

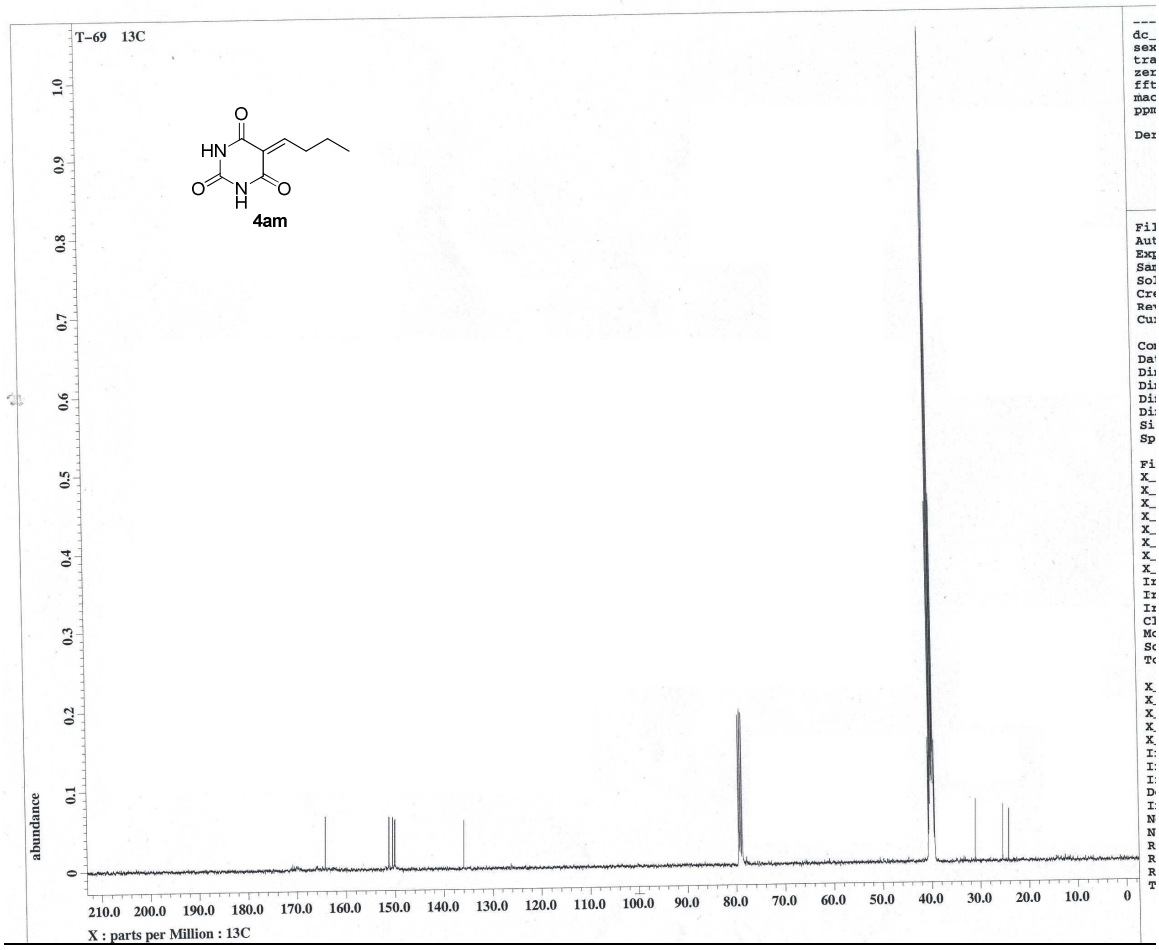

164.54
163.22
150.38
143.71
139.76
136.41
129.16
123.69
122.68
117.65
113.16
111.41
108.62
39.92
39.78
39.64
39.50
39.37
39.23
39.09
4al
200
180
160
140
120
100
80
60
40
20
ppm

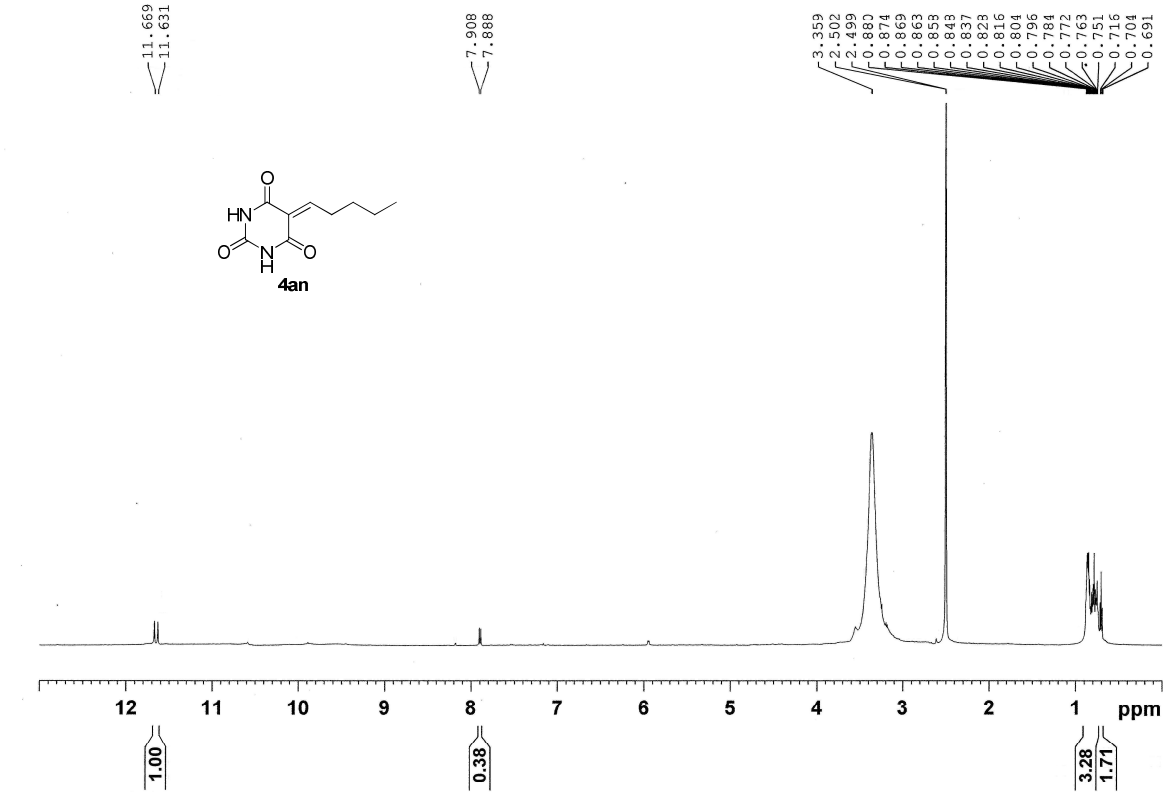

164.54
163.22
150.38
143.71
139.76
136.41
129.16
123.69
122.68
117.65
113.16
111.41
108.62
HN
O
N
H
O
O
NH
4al

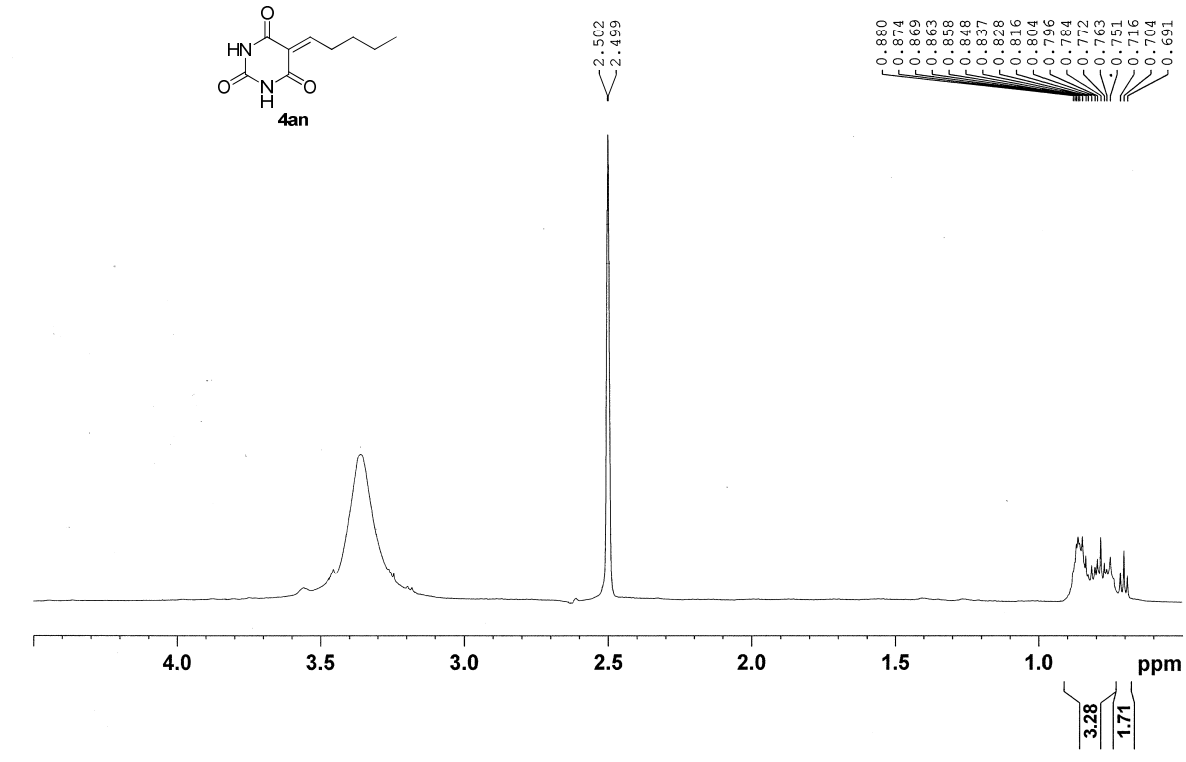

165
160
155
150
145
140
135
130
125
120
115
110
ppm

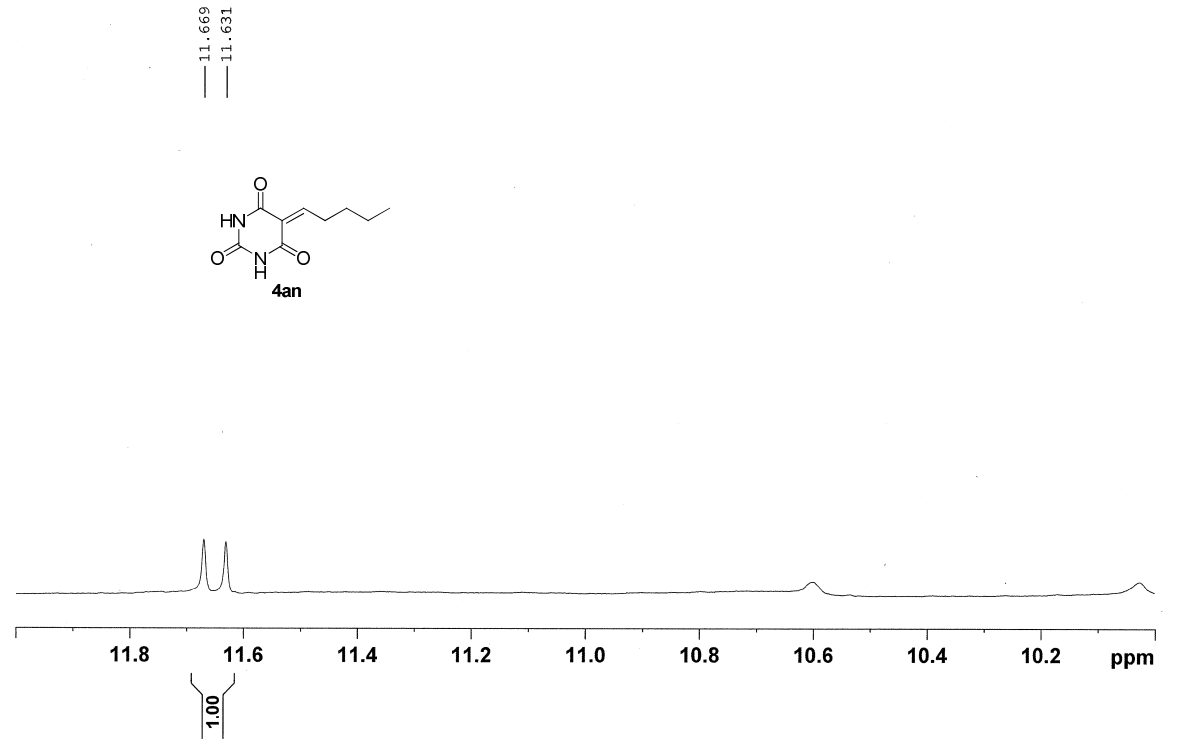

11.705
11.663
7.875
7.833
3.682
3.662
3.513
3.438
2.486
1.709
1.681
1.277
1.249
1.221
0.921
0.886
0.851
HN
O
N
H
O
O
4am
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
3.00
1.00
3.51
3.89
4.37

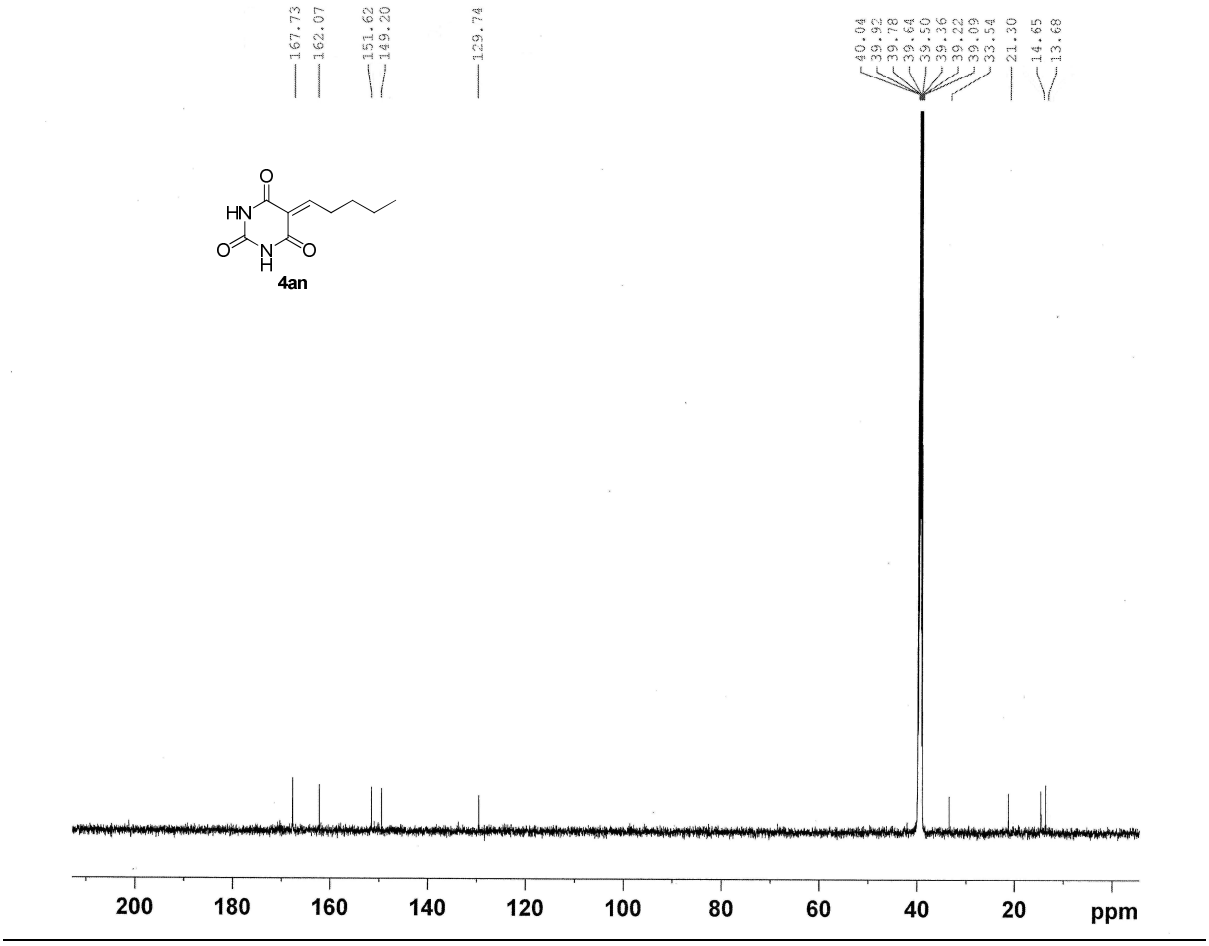

2.486
1.709
1.681
1.277
1.249
1.221
0.921
0.886
0.851
0.802
0.774
0.746
0.712
4am
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
ppm
3.51
3.89
4.37

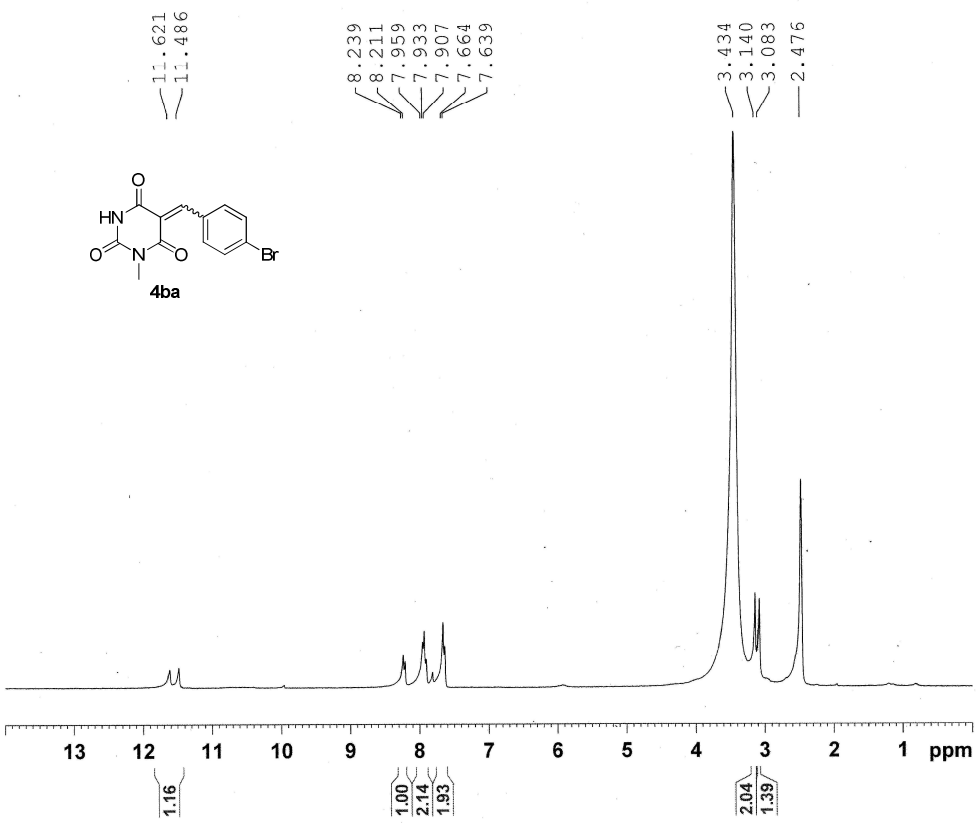

T-69 13C
4am
abundance
X : parts per Million : 13C

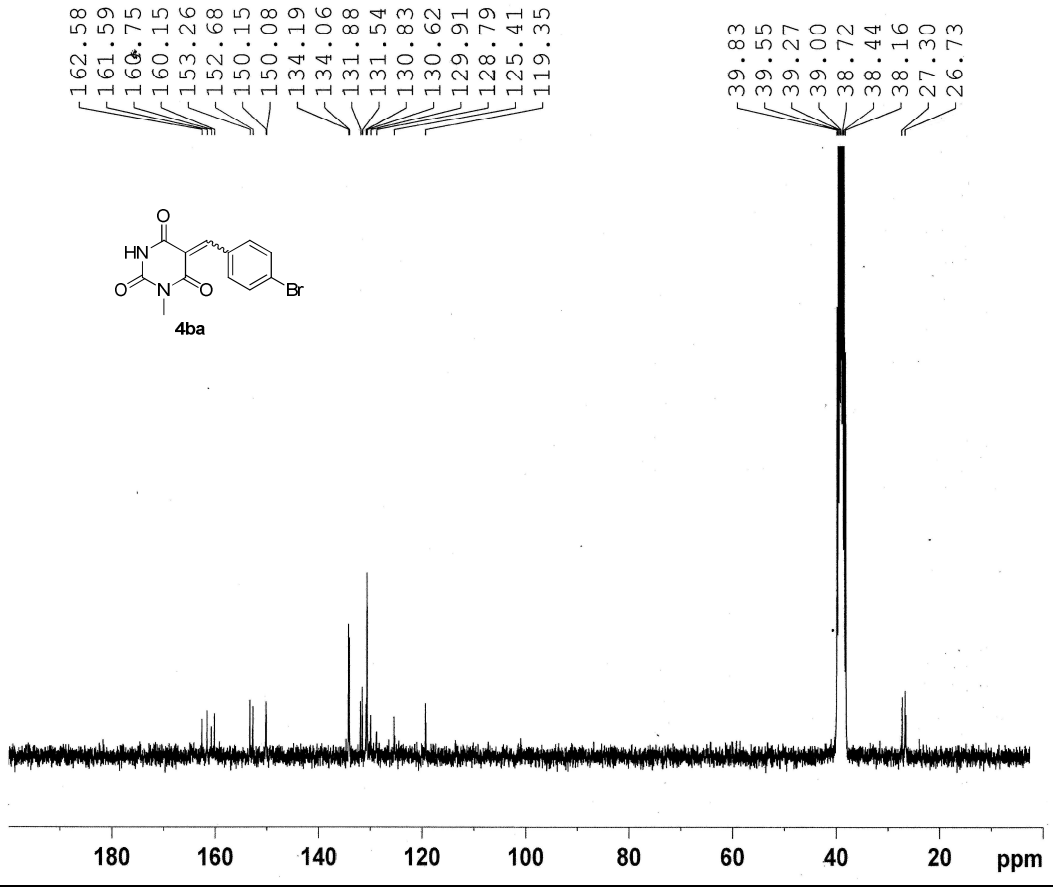

11.669
11.631
7.908
7.888
3.359
2.502
2.499
0.880
0.874
0.869
0.863
0.858
0.843
0.837
0.823
0.816
0.804
0.796
0.784
0.772
0.763
0.751
0.716
0.704
0.691
4an
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.38
3.28
1.71

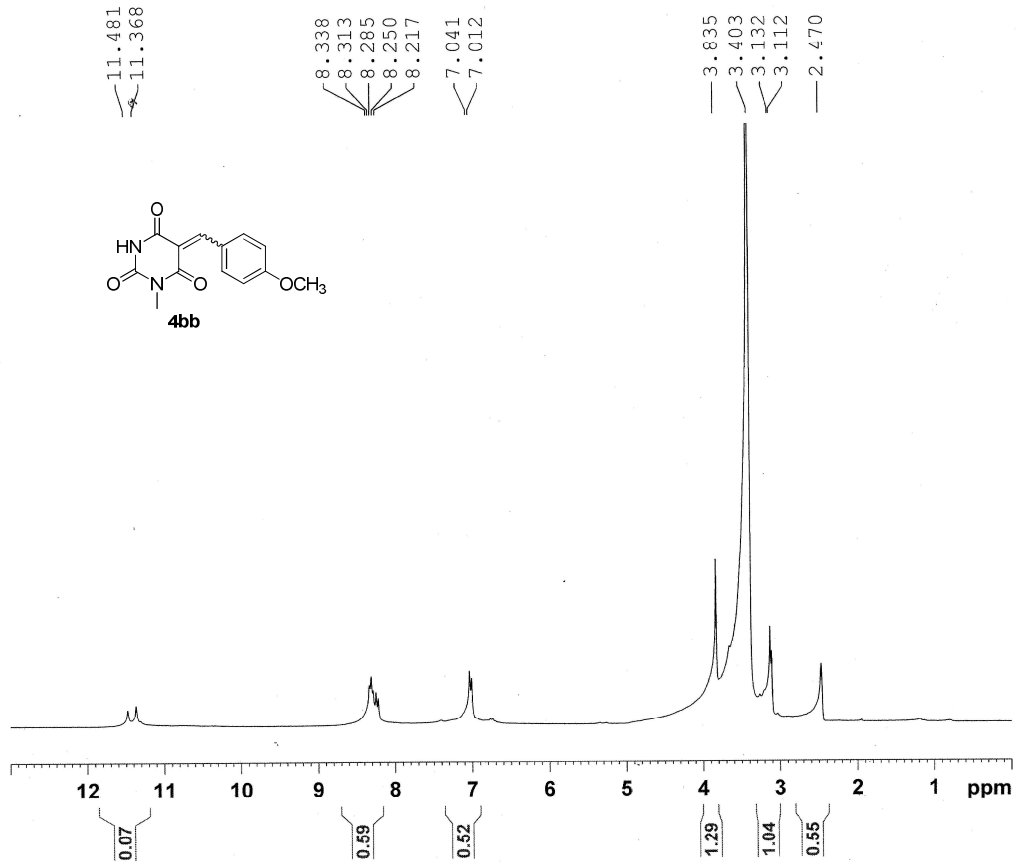

4an
2.502
2.499
0.880
0.874
0.869
0.863
0.858
0.848
0.837
0.828
0.816
0.804
0.796
0.784
0.772
0.763
0.751
0.716
0.704
0.691
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
3.28
1.71

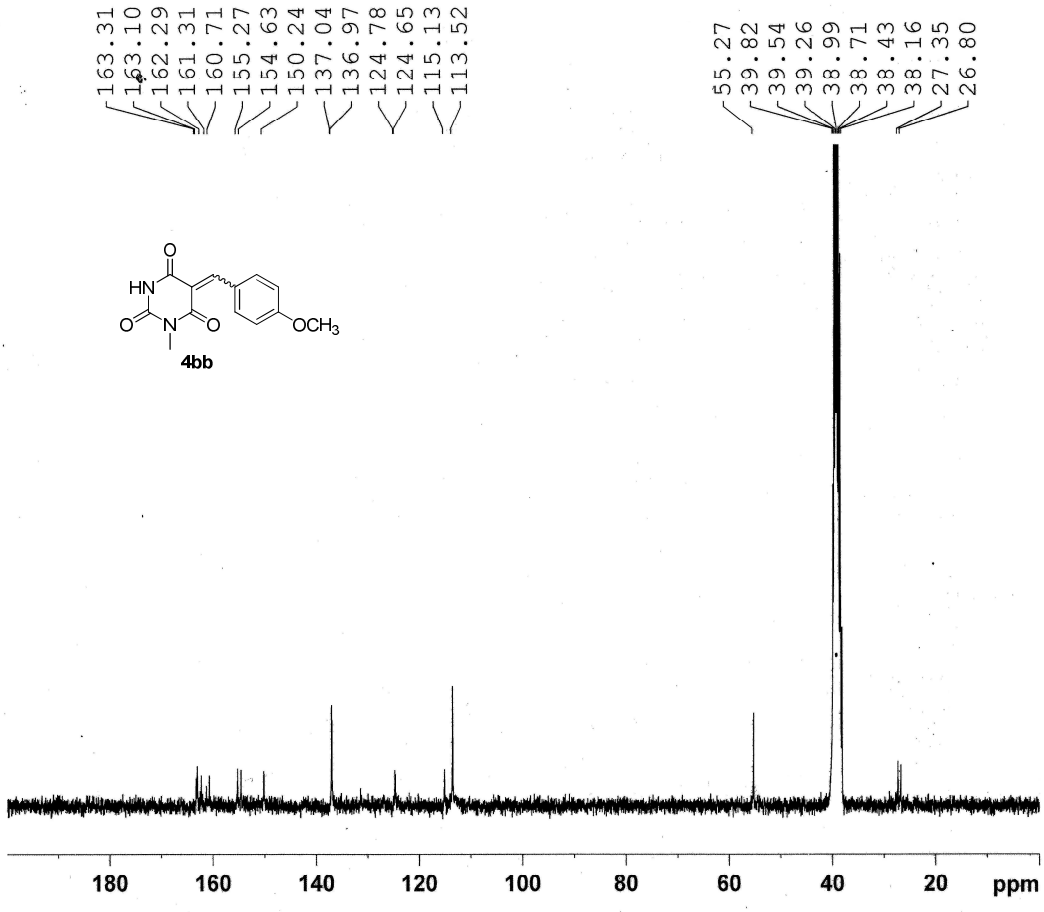

11.669
11.631
4an
11.8
11.6
11.4
11.2
11.0
10.8
10.6
10.4
10.2
ppm
1.00

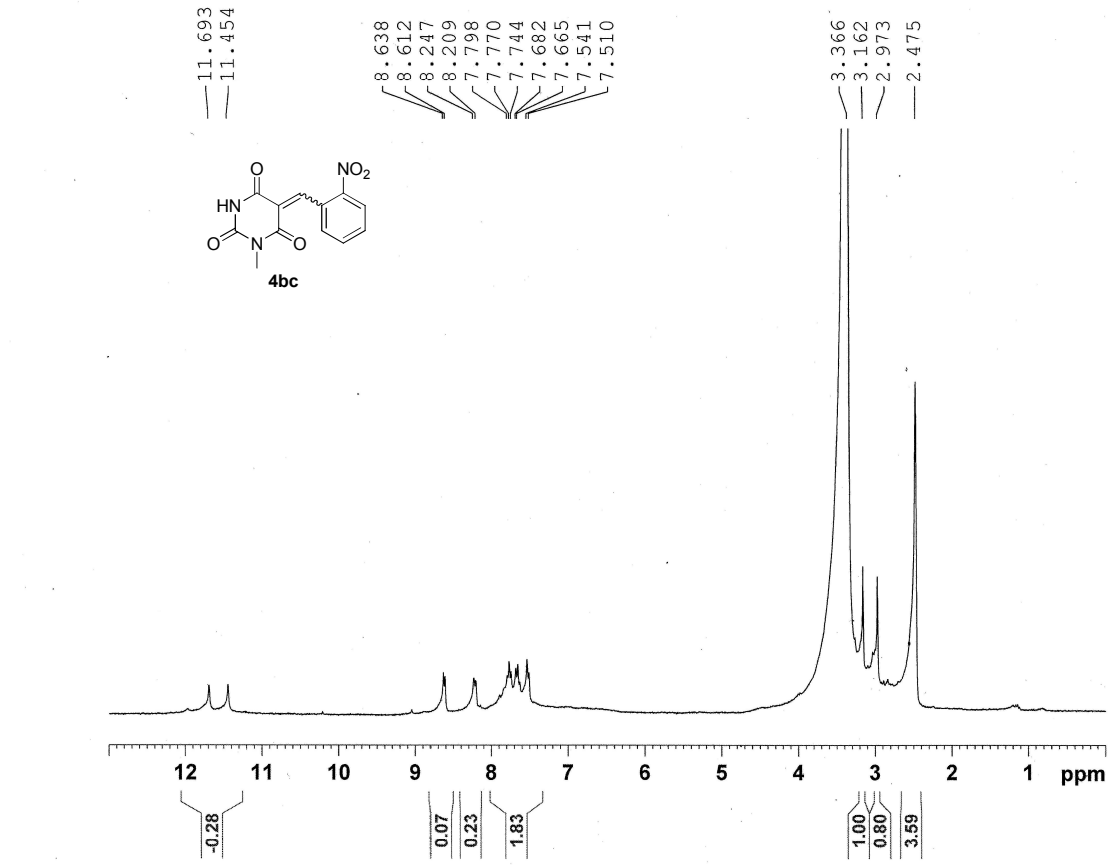

167.73
162.07
151.62
149.20
129.74
40.04
39.92
39.78
39.64
39.50
39.36
39.22
39.09
33.54
21.30
14.65
13.68
4an
200
180
160
140
120
100
80
60
40
20
ppm

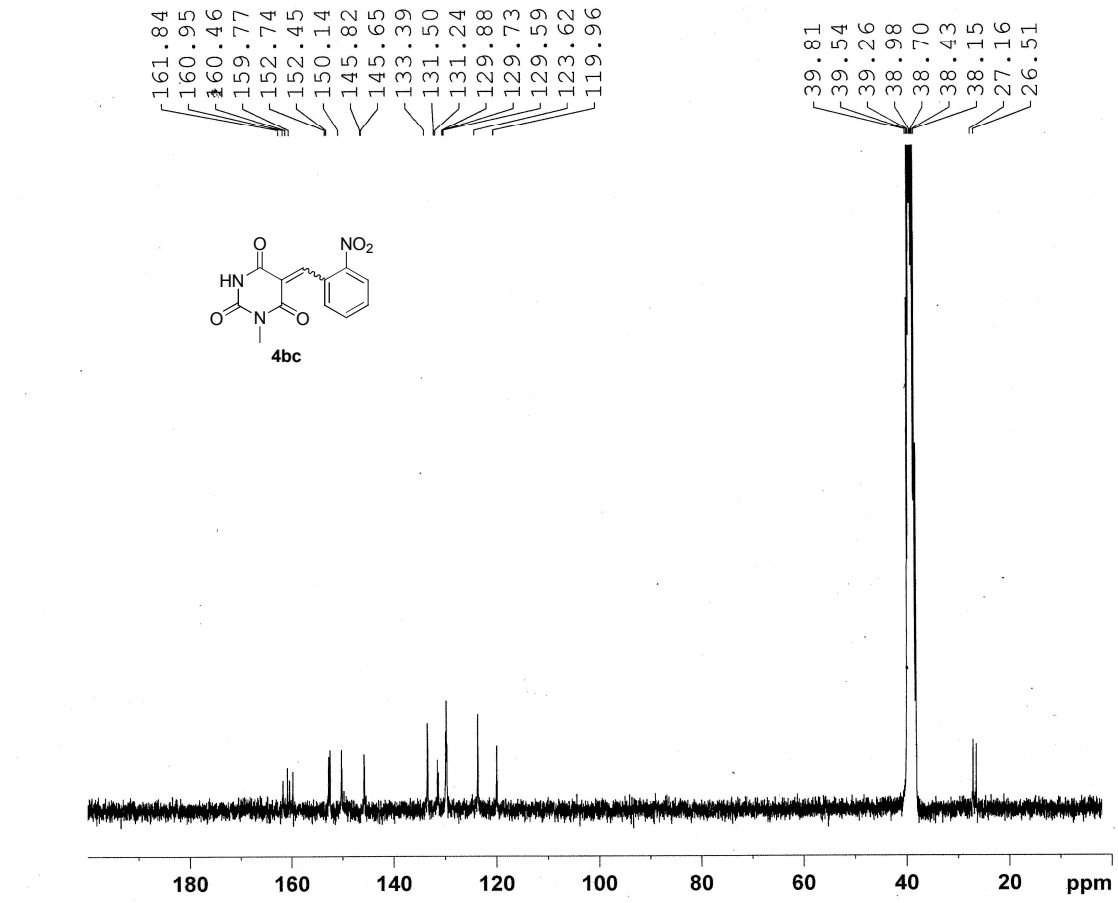

11.621
11.486
8.239
8.211
7.959
7.933
7.907
7.664
7.639
3.434
3.140
3.083
2.476
HN
O
N
O
O
Br
4ba
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.16
1.00
2.14
1.93
2.04
1.39

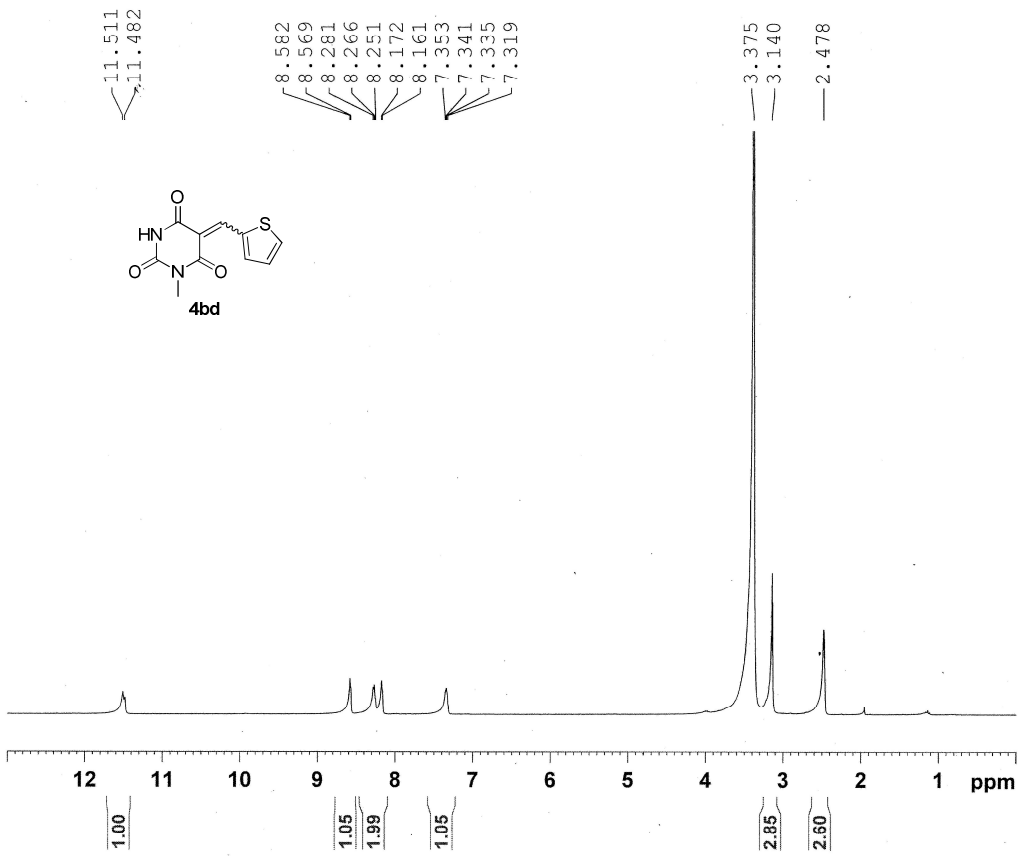

162.58
161.59
160.75
160.15
153.26
152.68
150.15
150.08
134.19
134.06
131.88
131.54
130.83
130.62
129.91
128.79
125.41
119.35
39.83
39.55
39.27
39.00
38.72
38.44
38.16
27.30
26.73
HN
O
N
O
O
Br
4ba
180
160
140
120
100
80
60
40
20
ppm

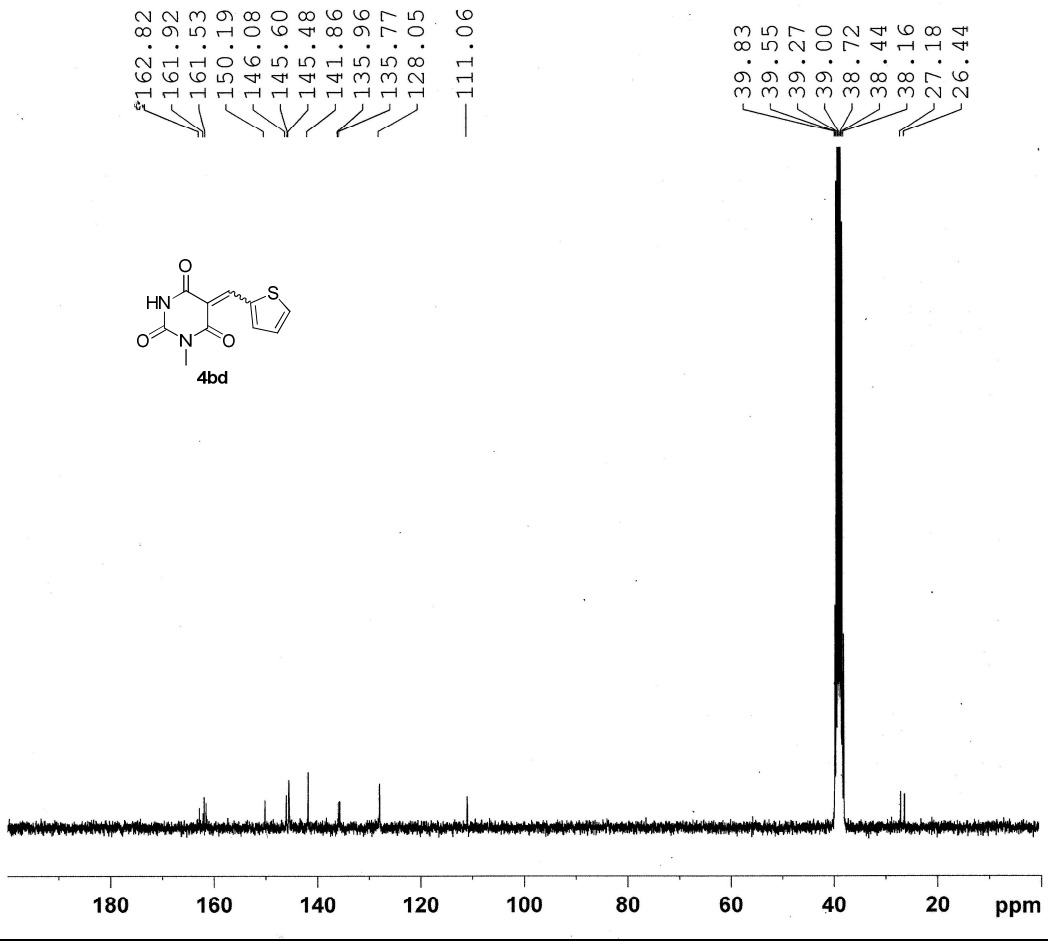

11.481
11.368
8.338
8.313
8.285
8.250
8.217
7.041
7.012
3.835
3.403
3.132
3.112
2.470
HN
O
N
OCH3
4bb
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.07
0.59
0.52
1.29
1.04
0.55

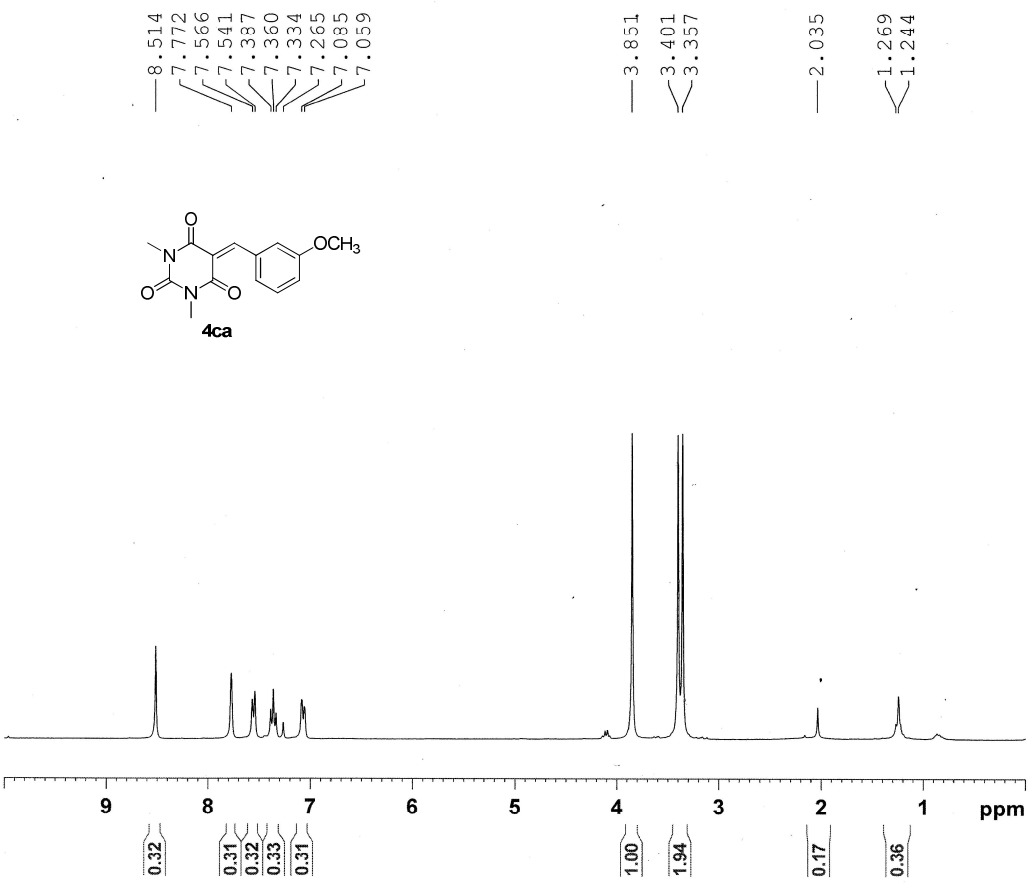

163.31
163.10
162.29
161.31
160.71
155.27
154.63
150.24
137.04
136.97
124.78
124.65
115.13
113.52
55.27
39.82
39.54
39.26
38.99
38.71
38.43
38.16
27.35
26.80
HN
O
N
OCH3
4bb
180
160
140
120
100
80
60
40
20
ppm

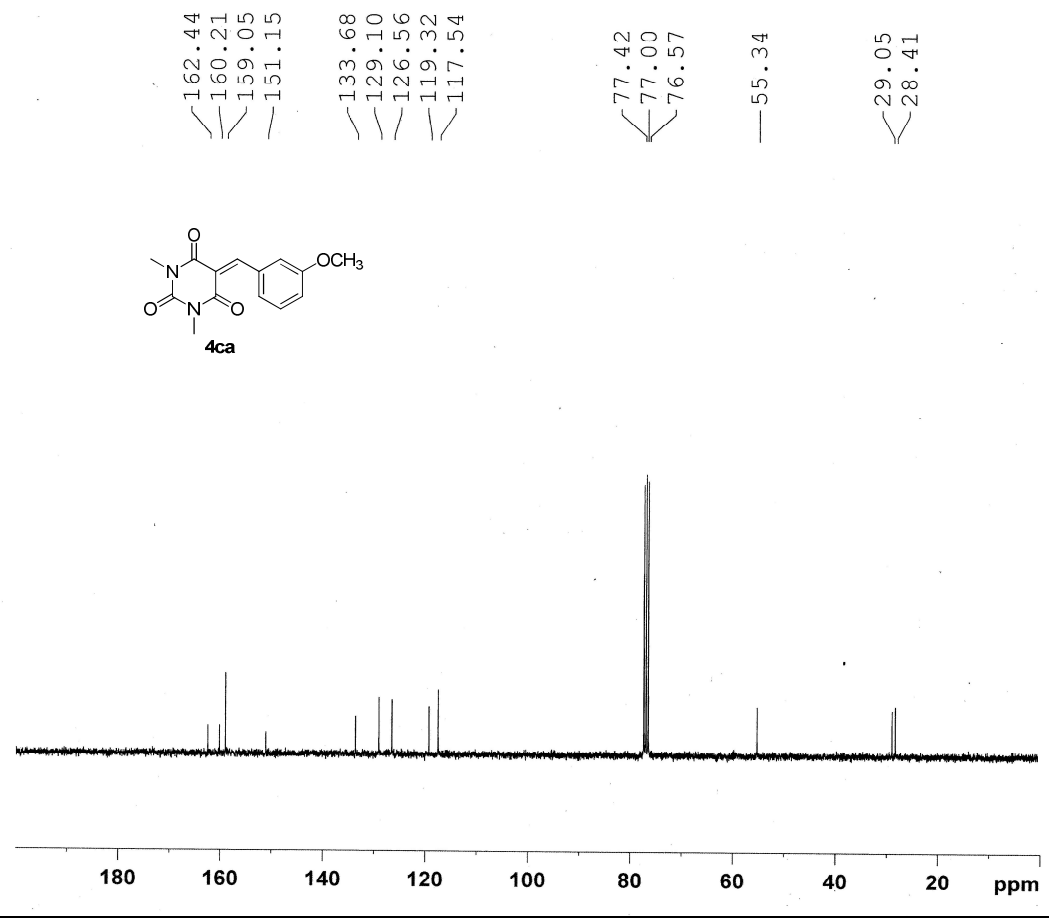

11.693
11.454
8.638
8.612
8.247
8.209
7.798
7.770
7.744
7.682
7.665
7.541
7.510
3.366
3.162
2.973
2.475
O
NO2
HN
O
N
O
4bc
12
11
10
9
8
7
6
5
4
3
2
1
ppm
-0.28
0.07
0.23
1.83
1.00
0.80
3.59

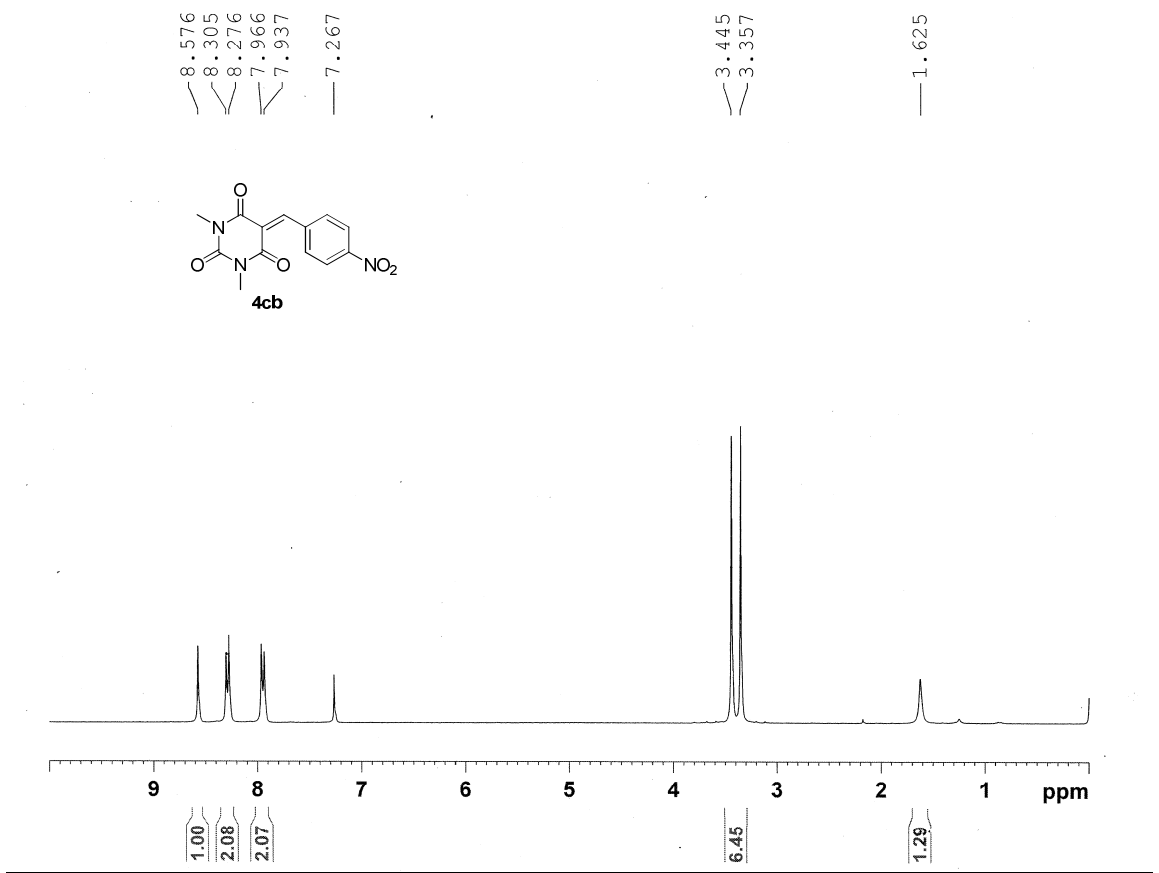

161.84
160.95
160.46
159.77
152.74
152.45
150.14
145.82
145.65
133.39
131.50
131.24
129.88
129.73
129.59
123.62
119.96
39.81
39.54
39.26
38.98
38.70
38.43
38.15
27.16
26.51
O
NO2
HN
O
N
O
4bc
180
160
140
120
100
80
60
40
20
ppm

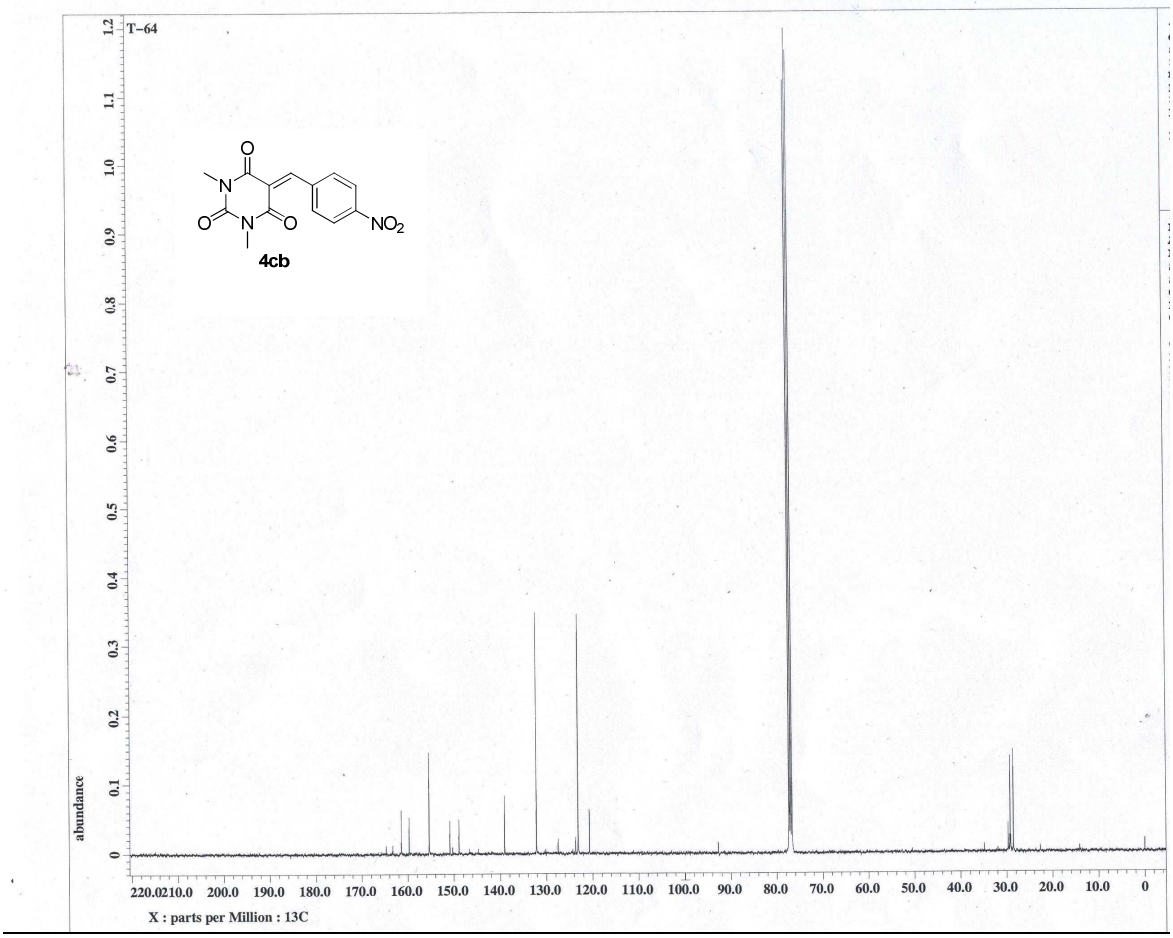

11.511
11.482
8.582
8.569
8.281
8.266
8.251
8.172
8.161
7.353
7.341
7.335
7.319
3.375
3.140
2.478
4bd
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.05
1.99
1.05
2.85
2.60

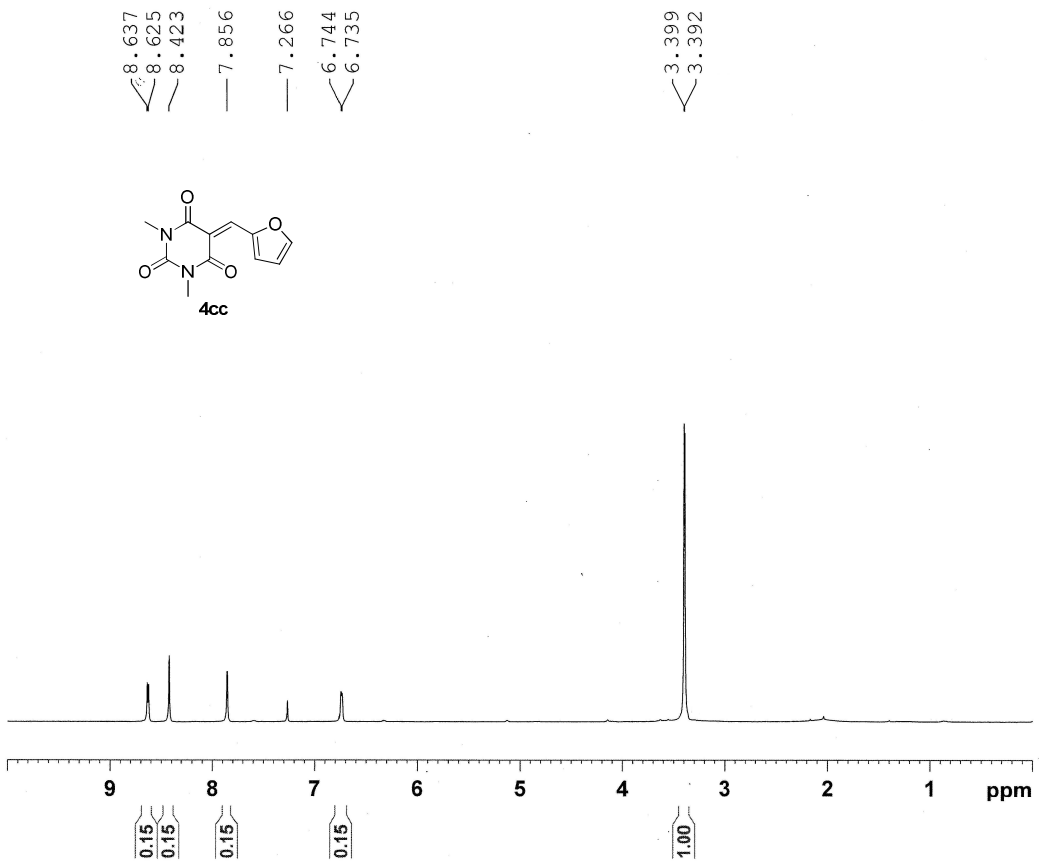

162.82
161.92
161.53
150.19
146.08
145.60
145.48
141.86
135.96
135.77
128.05
111.06
39.83
39.55
39.27
39.00
38.72
38.44
38.16
27.18
26.44
4bd
180
160
140
120
100
80
60
40
20
ppm

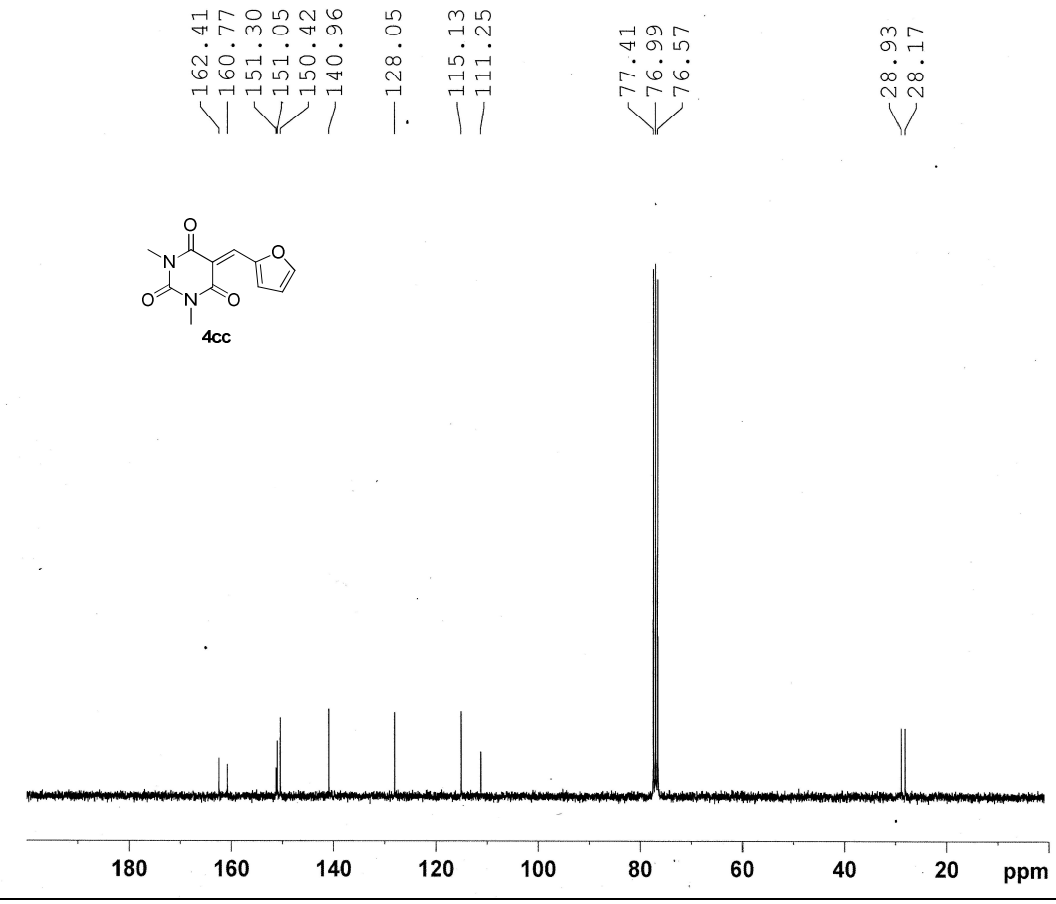

8.514
7.772
7.566
7.541
7.387
7.360
7.334
7.265
7.085
7.059
3.851
3.401
3.357
2.035
1.269
1.244
OCH3
4ca
9
8
7
6
5
4
3
2
1
ppm
0.32
0.31
0.32
0.33
0.31
1.00
1.94
0.17
0.36

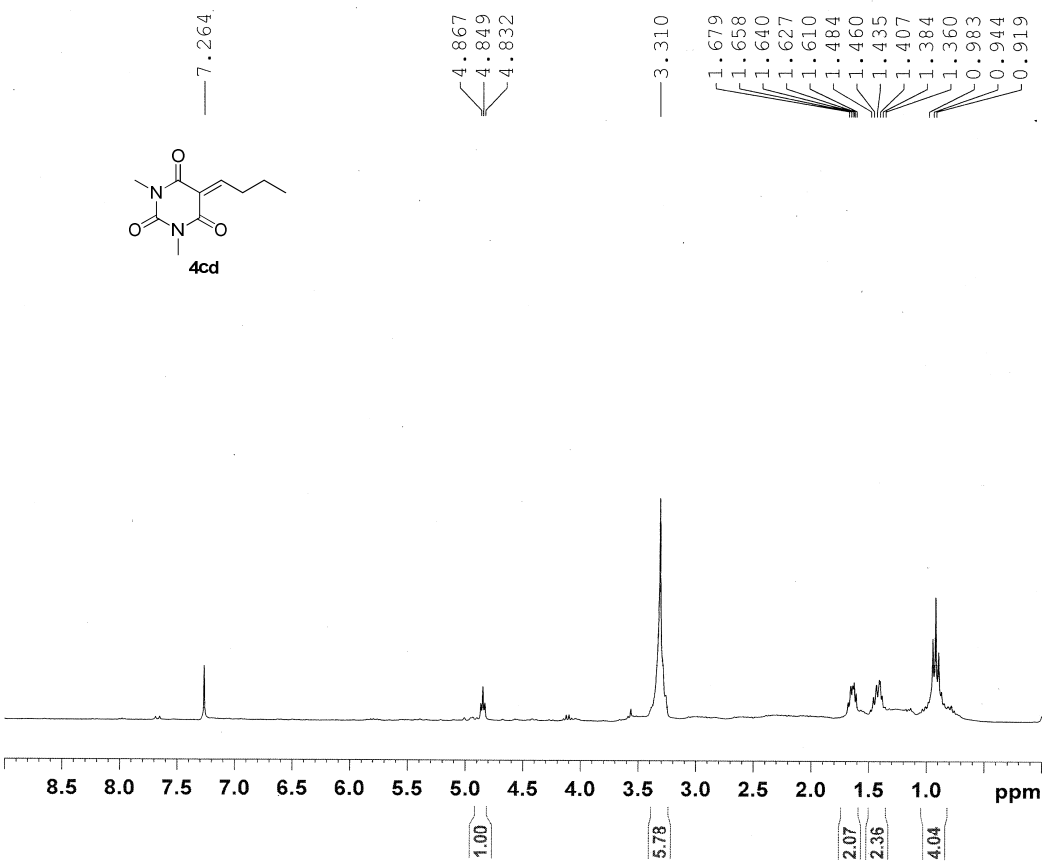

162.44
160.21
159.05
151.15
133.68
129.10
126.56
119.32
117.54
77.42
77.00
76.57
55.34
29.05
28.41
OCH3
4ca
180
160
140
120
100
80
60
40
20
ppm

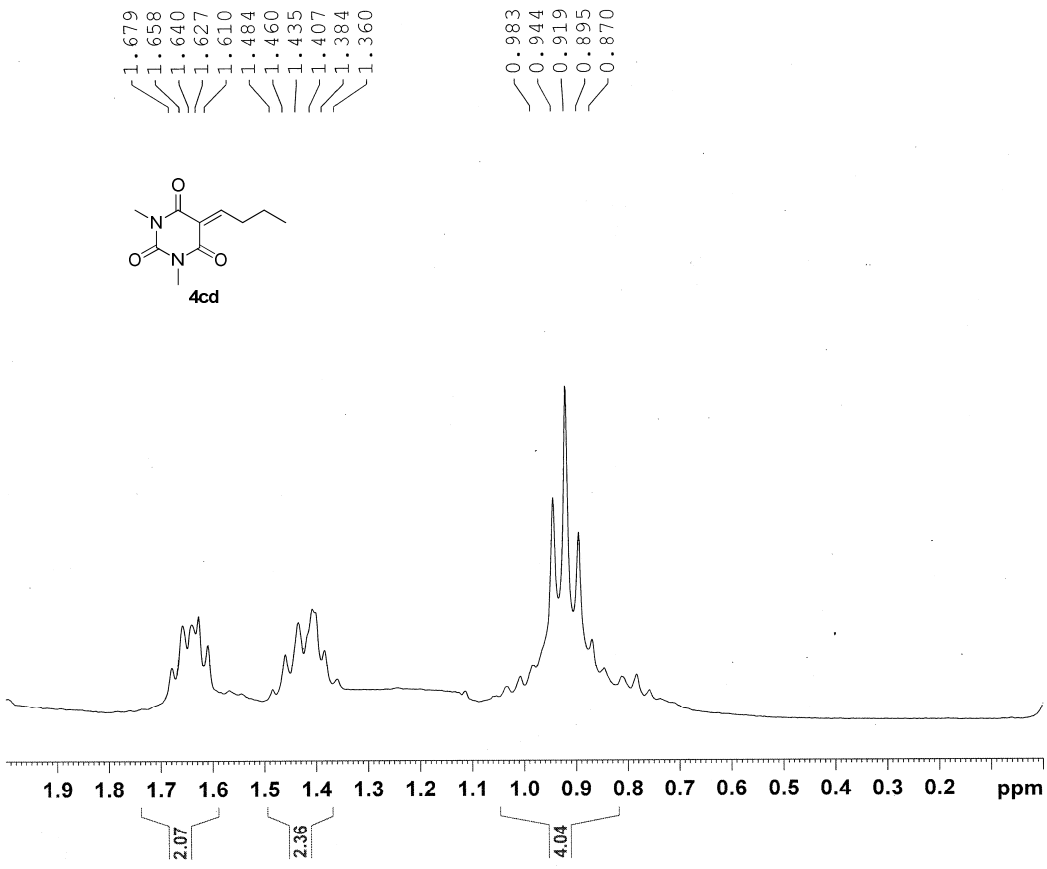

8.576
8.305
8.276
7.966
7.937
7.267
3.445
3.357
1.625
4cb
9
8
7
6
5
4
3
2
1
ppm
1.00
2.08
2.07
6.45
1.29

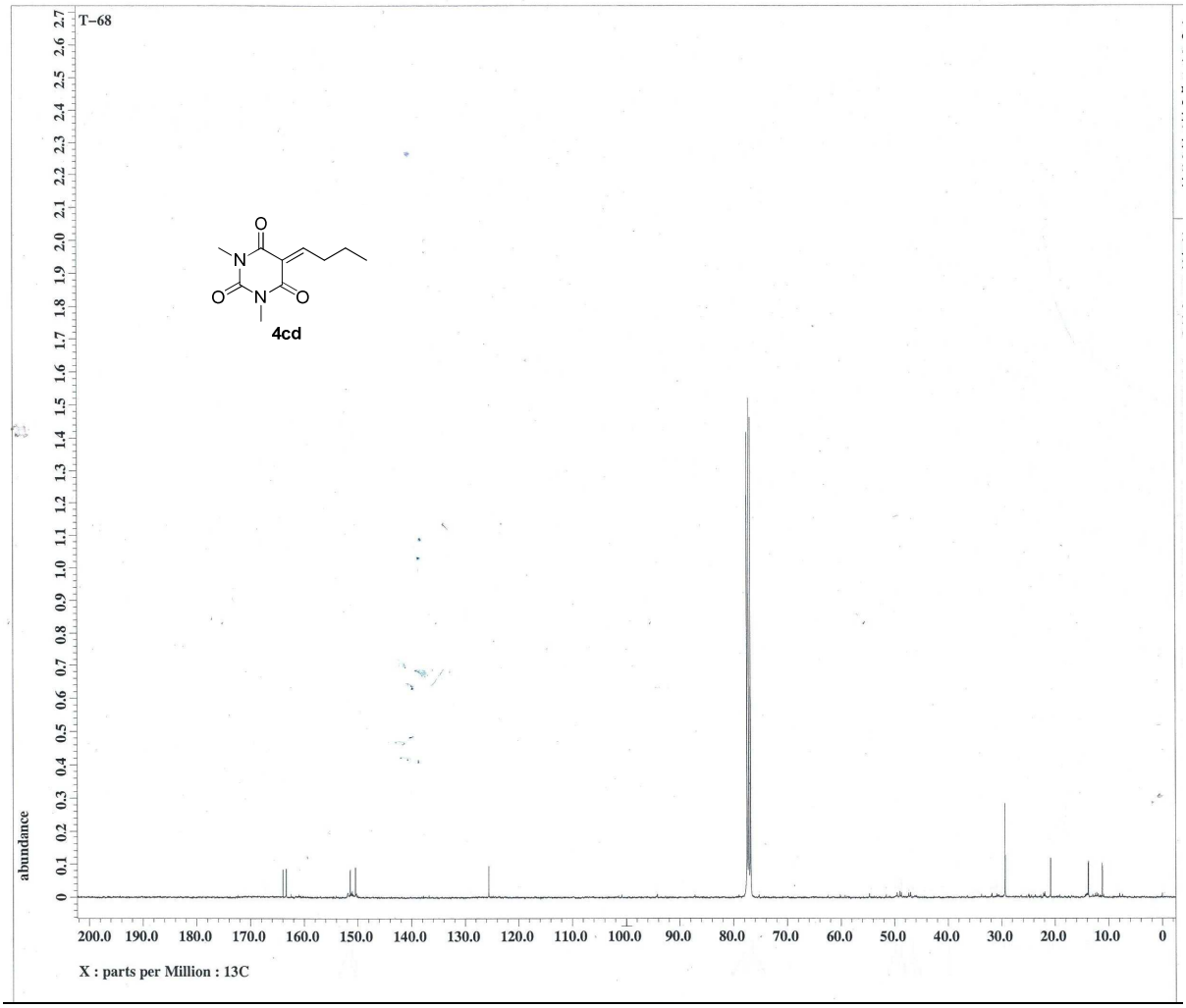

T-64
4cb
abundance
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : 13C


8.637
8.625
8.423
7.856
7.266
6.744
6.735
3.399
3.392
4cc
9
8
7
6
5
4
3
2
1
ppm
0.15
0.15
0.15
0.15
1.00


162.41
160.77
151.30
151.05
150.42
140.96
128.05
115.13
111.25
77.41
76.99
76.57
28.93
28.17
4cc
180
160
140
120
100
80
60
40
20
ppm


7.264
4.867
4.849
4.832
3.310
1.679
1.658
1.640
1.627
1.610
1.484
1.460
1.435
1.407
1.384
1.360
0.983
0.944
0.919
4cd
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
5.78
2.07
2.36
4.04


1.679
1.658
1.640
1.627
1.610
1.484
1.460
1.435
1.407
1.384
1.360
0.983
0.944
0.919
0.895
0.870
4cd
1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 ppm
2.07
2.36
4.04


T-68
4cd
abundance
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0 2.1 2.2 2.3 2.4 2.5 2.6 2.7
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : 13C