Formation of unexpected α-amino amidine through three-component 'UGI condensation reaction'

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General information:

Melting points were determined on a Büchi melting point apparatus and are uncorrected. IR spectra were recorded on Perkin-Elmer 281 IR spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on Varian 400 spectrometer TMS as internal reference; chemical shifts (δ scale) are reported in parts per million (ppm). ^{1}H NMR Spectra are reported in the order: multiplicity, coupling constant (J value) in hertz (Hz) and no of protons; signals were characterized as s (singlet), d (doublet), t (triplet), m (multiplet), and dd (doublet of doublet), bs (broad). HRMS spectrometry data was collected on Agilent Technologies 6520 Accurate-Mass Q-TOF LC/MS Elemental analyses were carried out using Perkin-Elmer 2400 Series II CHNS/O analyzer at the Department of Chemistry, Indian Institute of Technology, Guwahati. The X-ray crystal structures were determined with a Siemen P-4 diffractometer.

N-tert-Butyl-N',2-diphenyl-2-(phenyl amino)acetamidine (**5a**): Yield = 92%, white solid, mp 134 °C, IR (KBr) ν_{max} 3389, 3027, 2963, 2906, 1634, 1601, 1591, 1485, 1310, 1253, 1220, 1185, 1166, 1070 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.22 (m, 5H), 7.14 (dd, *J* = 2 Hz, *J* = 8 Hz, 2H), 7.03(t, *J* = 8 Hz, 2H), 6.86-6.81 (m, 2H), 6.70 (d, *J* = 7.6 Hz, 2H), 6.48 (d, *J* = 7.2 Hz, 2H), 5.90 (bs, 1H, NH), 4.92 (s, 1H), 3.71 (bs, 1H, NH), 1.46 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 154.0, 150.7, 147.3, 140.0, 129.4, 128.9, 128.6, 128.4, 128.2, 122.4, 121.3, 119.5, 113.9, 60.0, 51.0, 28.5. Anal. Calcd for $C_{24}H_{27}N_3$ (357.49): C, 80.63; H, 7.61; N, 11.75. Found: C, 80.54; H, 7.53; N, 11.66.

2-(4-Chlorophenylamino)-N-tert-butyl-2-(4-bromophenyl)-N'-(4-chlorophenyl)acetamidine (**5b**): Yield = 85%, pale yellow solid, mp 190 °C, IR (KBr) ν_{max} 3382, 2963, 1640, 1485, 1259, 1084 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* =8.8 Hz, 2H), 7.03 (dd, *J* = 4.0 Hz, *J* = 8.8 Hz, 4H), 6.57 (d, *J* = 8.8 Hz, 2H), 6.38 (d, *J* = 8.4 Hz, 2H), 5.79 (bs, 1H, NH), 4.79 (s, 1H), 3.68 (bs, 1H, NH), 1.43 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.5, 149.1, 145.3, 138.3, 135.1, 132.4, 129.8, 129.5, 128.8, 126.7, 124.8, 123.5, 115.0, 59.4, 51.4, 28.5.

Anal. Calcd for $C_{24}H_{24}BrCl_2N_3$ (505.28): C, 57.05; H, 4.79; N, 8.32. Found: C, 56.92; H, 4.70; N, 8.21. HRMS (ESI) calcd for $C_{24}H_{24}BrCl_2N_3$ [M + H] 506.0581 found 506.0600.

2-(4-Bromophenylamino)-N-tert-butyl-N',2-bis(4-bromophenyl)acetamidine (**5c**): Yield = 77%, pale yellow solid, mp 182 °C, IR (KBr) ν_{max} 3381, 2965, 2901, 1641, 1591, 1581, 1506, 1488, 1388, 1253, 1180, 1164, 1070 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.52 (d, *J* = 8.8 Hz, 2H), 6.33 (d, *J* = 8.4 Hz, 2H), 5.77 (bs, 1H, NH), 4.78 (s, 1H), 3.71 (bs, 1H, NH), 1.42 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.4, 149.5, 145.7, 138.2, 132.4, 131.7, 129.8, 124.0, 122.9 (2C), 115.4, 114.3, 111.9, 59.3, 51.4, 28.4. Anal. Calcd for $C_{24}H_{24}Br_3N_3$ (594.18): C, 48.51; H, 4.07; N, 7.07. Found: C, 48.42; H, 3.98; N, 6.97.

2-(4-Chlorophenylamino)-N-tert-butyl-N',2-bis(4-chlorophenyl)acetamidine (**5d**): Yield = 93%, white solid, mp 180 °C, IR (KBr) ν_{max} 3384, 2967, 2901, 1641, 1491, 1482, 1284, 1253, 1180, 1088 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$): δ 7.28 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.8 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 6.57 (d, *J* = 8.8 Hz, 2H), 6.38 (d, *J* = 8.4 Hz, 2H), 5.80 (bs, 1H, NH), 4.80 (s, 1H), 3.68 (bs, 1H, NH), 1.43 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.6, 149.1, 145.4, 137.8, 134.8, 129.6, 129.5,129.4, 128.8, 126.7, 124.8, 123.6, 115.0, 59.4, 51.4, 28.5. Anal. Calcd for $C_{24}H_{24}Cl_3N_3$ (460.83): C, 62.55; H, 5.25; N, 9.12. Found: C, 62.44; H, 5.17; N, 9.02.

2-(4-Chlorophenylamino)-N-tert-butyl-N'-(4-chlorophenyl)-2-(4-fluorophenyl)acetamidine (**5e**): Yield = 89 %, white solid, mp 153° C, IR (KBr) ν_{max} 3408, 3386, 2964, 2924, 1634, 1601, 1509, 1494, 1483, 1230, 1181, 1157, 1086 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.19 (d, *J* = 9.2 Hz, 2H), 7.25-7.10 (m, 2H), 7.03-6.97 (m, 4H), 6.58 (d, *J* = 8.8 Hz, 2H), 6.37 (d, *J* = 8.8 Hz, 2H), 5.83 (bs, 1H, NH), 4.80 (d, *J* = 1.6 Hz, 1H), 3.69 (bs, 1H, NH), 1.43 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.9, 149.1, 145.5, 135.1, 130.0, 129.5, 128.7, 126.6, 124.7, 123.5, 116.2, 116.0, 115.0, 59.4, 51.3, 28.5. Anal. Calcd for $C_{24}H_{24}Cl_2FN_3$ (444.37): C, 64.87; H, 5.44; N, 9.46. Found: C, 64.76; H, 5.38; N, 9.34.

2-(4-Bromophenylamino)-N-tert-butyl-N'-(4-bromophenyl)-2-(4-nitrophenyl)acetamidine (**5f**): Yield = 76%, yellow solid, mp 218 °C, IR (KBr) ν_{max} 3384, 2960, 1634, 1522, 1492, 1479, 1347,

1182 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.8 Hz, 2H), 7.37-7.32 (m, 4H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.55 (d, *J* = 8.8 Hz, 2H), 6.33 (d, *J* = 8.8 Hz, 2H), 5.77 (bs, 1H, NH), 4.99 (d, *J* = 1.6 Hz, 1H), 3.73 (bs, 1H, NH), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 152.7, 149.3, 148.0, 145.9, 145.4, 132.5, 131.9, 129.3, 124.4, 123.8, 115.6, 114.6, 112.4, 59.2, 51.6, 28.4. Anal. Calcd for $C_{24}H_{24}Br_2N_4O_2$ (560.28): C, 51.45; H, 4.32; N, 10.00. Found: C, 51.32; H, 4.26; N, 9.90.

2-(4-Bromophenylamino)-N-tert-butyl-N'-(4-bromophenyl)-2-(3,4,5-trimethoxyphenyl) acetamidine (**5g**): Yield = 82%, white solid, mp 215°C, IR (KBr) ν_{max} 3382, 3362, 2958, 2935, 1637, 1595, 1508, 1481, 1329, 1131 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 8.8 Hz, 2H), 6.36 (d, *J* = 8.4 Hz, 2H), 6.32 (s, 2H), 5.89 (bs, 1H, NH), 4.71 (s, 1H), 3.85 (s, 3H), 3.76 (s, 7H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.9, 153.6, 149.9, 146.1, 138.3, 134.6, 132.4, 131.6, 124.3, 115.5, 114.1, 111.8, 105.1, 61.2, 60.5, 56.3, 51.3, 28.6. Anal. Calcd for $C_{27}H_{31}Br_2N_3O_3$ (605.36): C, 53.57; H, 5.16; N, 6.94. Found: C, 53.42; H, 5.08; N, 6.85.

2-(4-Bromophenylamino)-N-tert-butyl-N'-(4-bromophenyl)-2-(2-nitrophenyl)acetamidine (**5h**): Yield = 75%, light yellow, mp 198°C, IR (KBr) ν_{max} 3357, 3298, 2969, 2956, 1615, 1580, 1520, 1480, 1347, 1258, 1181, 1071 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.8 Hz, 1H), 7.61-7.60 (m, 2H), 7.50-7.45 (m, 1H), 7.34 (d, *J* = 8.8 Hz, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 6.55 (d, *J* = 8.8 Hz, 2H), 6.16 (d, *J* = 8.8 Hz, 2H), 5.98 (bs, 1H, NH), 5.34 (d, *J* = 3.2 Hz, 1H), 3.94 (bs, 1H, NH), 1.45 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.4, 149.3, 149.0, 145.8, 133.7, 133.0, 132.4, 131.7, 130.0, 129.8, 125.0, 123.4, 115.7, 114.4, 112.3, 56.1, 51.6, 28.4. Anal. Calcd for $C_{24}H_{24}Br_2N_4O_2$ (560.28): C, 51.45; H, 4.32; N, 10.00. Found: C, 51.34; H, 4.25; N, 9.90.

N'-(4-Bromophenyl)-2-(4-bromophenylamino)-N-tert-butyl-2-(2-chlorophenyl)acetamidine (**5i**): Yield = 65 %, white solid, mp 166°C, IR (KBr) ν_{max} 3373, 2966, 1719, 1669, 1633, 1588, 1492, 1373, 1212, 1127, 1071, 1033 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 6.4 Hz, 1H), 7.34-7.29 (m, 5H), 7.11 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 8.8 Hz, 2H), 6.27 (d, *J* = 8.8 Hz, 2H), 5.89 (bs, 1H, NH), 5.16 (s, 1H), 3.65 (bs, 1H, NH), 1.47 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.7, 149.6, 146.3, 136.7, 134.9, 132.3, 131.8, 131.6, 130.2, 129.1, 127.4, 123.6, 115.4, 114.2,

111.9, 57.1, 51.4, 28.5. Anal. Calcd for $C_{24}H_{24}Br_2ClN_3$ (549.73): C, 52.44; H, 4.40; N, 7.64. Found: C, 52.38; H, 4.28; N, 7.52.

2-(4-Chlorophenylamino)-N-tert-butyl-N'-(4-chlorophenyl)-2-(2,4-dimethoxyphenyl)acetamidine(**5j**): Yield = 78%, white solid, mp 140°C, IR (KBr) ν_{max} 3358, 2962, 2838, 1614, 1587, 1509, 1493, 1482, 1290, 1207, 1171, 1098, 1032 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.4 Hz, 1H), 7.17 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 6.56 (d, *J* = 9.2 Hz, 2H), 6.47 (dd, *J* = 2.4 Hz, *J* = 8.4 Hz, 1H), 6.33 (d, *J* = 8.4 Hz, 2H), 6.28 (d, *J* = 2 Hz, 1H),5.92 (bs, 1H, NH), 5.05 (s, 1H), 3.82 (s, 3H), 3.64 (bs, 1H, NH), 3.42 (s, 3H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 161.3, 158.0, 155.3, 149.8, 146.3, 129.2, 129.0, 128.3, 126.0, 124.0, 123.6, 120.1, 115.0, 104.6, 98.5, 55.5, 55.4, 53.4, 51.1, 28.5. Anal. Calcd for $C_{26}H_{29}Cl_2N_3O_2$ (486.43): C, 64.20; H, 6.01; N, 8.64. Found: C, 64.08; H, 5.93; N, 8.56.

2-(4-Chlorophenylamino)-N-tert-butyl-2-(2-chlorophenyl)-N'-(4-chlorophenyl)acetamidine (**5k**): Yield = 87 %, white solid, mp 188°C, IR (KBr) ν_{max} 3401, 3371, 3045, 2961, 1634, 1599, 1513, 1494, 1482, 1182, 1089 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 6.4 Hz, 1H), 7.31-7.27 (m, 3H), 7.19 (dd, *J* = 2 Hz, *J* = 6.8 Hz, 2H), 6.97 (dd, *J* = 2.4 Hz, *J* = 6.8 Hz, 2H), 6.59 (dd, *J* = 2.4 Hz, *J* = 7.2 Hz, 2H), 6.31 (dd, *J* = 2 Hz, *J* = 6.4 Hz, 2H), 5.90 (bs, 1H, NH), 5.16 (d, *J* = 2.0 Hz, 1H), 3.63 (bs, 1H, NH), 1.47 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 153.9, 149.1, 145.8, 136.8, 134.9, 130.2, 129.4, 129.1, 128.7 (2C), 127.4, 126.6, 124.7, 123.2, 115.0, 57.2, 51.4, 28.5. Anal. Calcd for $C_{24}H_{24}Cl_3N_3$ (460.83): C, 62.55; H, 5.25; N, 9.12. Found: C, 62.44; H, 5.16; N, 9.02.

N-tert-Butyl-N'-(4-chlorophenyl)-2-(4-chlorophenylamino)2(3hydroxyphenyl)acetamidine (**5l**): Yield = 82 %, yellow solid, mp 192°C, IR (KBr) ν_{max} 3384, 2964, 2924, 1630, 1597, 1586, 1493, 1484, 1454, 1251, 1089 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.17-7.10 (m, 3H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.74 (d, *J* = 7.6 Hz, 1H), 6.65 (d, *J* = 7.2 Hz, 1H), 6.56 (s, 1H), 6.53 (d, *J* = 8.4 Hz, 2H), 6.40 (d, *J* = 8.4 Hz, 2H), 5.84 (bs, 1H, NH), 4.70 (s, 1H), 3.76 (bs, 1H, NH), 3.45 (bs, 1H, OH), 1.41 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 156.6, 154.3, 149.0, 145.5, 140.8, 130.4, 129.4, 128.6, 126.7, 124.5, 123.8, 120.0, 115.8, 115.0, 114.9, 60.0, 51.3, 28.5. Anal. Calcd for $C_{24}H_{25}Cl_2N_3O$ (442.38): C, 65.16; H, 5.70; N, 9.50. Found: C, 65.04; H, 5.58; N, 9.42.

N-Cyclohexyl-N',2-diphenyl-2-(phenyl amino)acetamidine (**5m**): Yield = 85%, white solid, mp 148°C, IR (KBr) ν_{max} 3373, 3238, 3023, 2928, 2853, 1621, 1589, 1517, 1495, 1484, 1313, 1259, 1199 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.28-7.22 (m, 5H), 7.12-7.05 (m, 4H), 6.85-6.80 (m, 2H), 6.69 (d, *J* = 8.0 Hz, 2H), 6.50 (d, *J* = 7.2 Hz, 2H), 5.97 (bs, 1H, NH), 4.97 (s, 1H), 3.94 (bs, 1H, NH), 2.13-2.05 (m, 2H), 1.45-1.40 (m, 4H), 1.30-1.07 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 155.2, 150.6, 147.2, 139.6, 129.3, 128.8, 128.5, 128.3, 128.1, 122.8, 121.6, 119.4, 113.8, 59.8, 48.6, 33.1, 32.6, 25.9, 25.0, 24.8. Anal. Calcd for $C_{26}H_{29}N_3$ (383.53): C, 81.42; H, 7.62; N, 10.96. Found: C, 81.32; H, 7.54; N, 10.88.

N',2-bis(4-Chlorophenyl)-2-(4-chlorophenylamino)-N-cyclohexylacetamidine (**5n**): Yield = 90%, pale yellow, mp 148°C, IR (KBr) ν_{max} 3255, 2934, 2855, 1640, 1600, 14292, 1256, 1091, 1013 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$/DMSO): δ 7.25 (d, *J* = 7.2 Hz, 2H), 7.18 (dd, *J* = 7.2 Hz, *J* = 19.6 Hz, 4H), 6.99 (d, *J* = 6.8 Hz, 2H), 6.70 (d, *J* = 6.8 Hz, 2H), 6.56 (d, *J* = 7.6 Hz, 2H), 4.97 (s, 1H), 3.79 (bs, 1H, NH), 3.20 (bs, 1H, NH), 1.98 (bs, 1H), 1.74-1.53 (m, 5H), 1.36-1.15 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 161.5, 142.8, 132.8, 132.5, 131.8, 131.4, 128.1, 128.0, 127.5, 127.4, 126.7, 121.4, 113.5, 56.2, 50.7, 29.2, 23.0, 22.6 Anal. Calcd for $C_{26}H_{26}Cl_3N_3$ (486.86): C, 64.14; H, 5.38; N, 8.63 Found: C, 64.05; H, 5.29; N, 8.57.

N'-(4-chlorophenyl)-2-((4-chlorophenyl)amino)-N-cyclohexyl-2-phenylacetimidamide (**5o**): Yield = 80%, White solid, mp 178 °C, ^1H NMR (400 MHz, $CDCl_3$): δ 7.31-7.26 (m, 3H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.10 (d, *J* = 6.8 Hz, 2H), 6.99 (d, *J* = 8 Hz, 2H), 6.55 (d, *J* = 8.4 Hz, 2H), 6.38 (d, *J* = 8.4 Hz, 2H), 5.93 (d, *J* = 7.6 Hz, 1H), 4.83 (bs, *NH*, 1H), 3.85 (bs, *NH*, 1H), 2.11-1.97 (m, 2H), 1.72-1.61 (m, 3H), 1.45-1.03 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 155.3, 148.9, 145.5, 138.8, 129.3, 129.1, 128.8, 128.6, 128.0, 126.9, 124.4, 124.0, 114.9, 59.9, 48.8, 33.1, 32.6, 25.8, 25.0, 24.9. Anal. Calcd for $C_{26}H_{27}Cl_2N_3$: C, 69.02; H, 6.02; N, 9.29 Found: C, 68.94; H, 5.92; N, 9.21.

N-cyclohexyl-N'-(3,4-dimethylphenyl)-2-((3,4-dimethylphenyl)amino)-2-(4-fluorophenyl) acetimidamide (**5p**): Yield = 76%, Pale yellow solid, mp 175 °C, ^1H NMR (400 MHz, $CDCl_3$): δ 7.10-7.06 (m, 2H), 6.98-6.91 (m, 2H), 6.80 (d, *J* = 7.6 Hz, 1H), 6.50 (d, *J* = 2Hz, 1H), 6.43 (dd, *J* = 2.4, *J* = 8 Hz, 1H), 6.25-6.22 (m, 2H), 5.93 (d, *J* = 8.4 Hz, 1H), 4.88 (bs, *NH*, 1H), 3.93-3.91 (m, 1H), 3.53 (bs, *NH*, 1H), 2.22 (s, 3H), 2.17 (s, 3H), 2.12 (s, 3H), 2.02 (s, 3H), 1.72-1.69 (m,

1H), 1.65-1.57 (m, 3H), 1.46-1.34 (m, 2H), 1.26-1.04 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 155.6, 148.1, 145.4, 137.5, 136.4, 136.1, 130.4, 130.0 (2C), 129.7, 129.4, 127.6, 124.2, 119.8, 115.7, 115.5, 111.4, 59.5, 48.6, 33.3, 32.7, 26.0, 25.2, 25.0, 20.1, 19.8, 19.1, 18.9. Anal. Calcd for $C_{30}H_{36}FN_3$: C, 78.74; H, 7.93; N, 9.18 Found: C, 78.63; H, 7.82; N, 9.05.

N'-(4-chlorophenyl)-2-((4-chlorophenyl)amino)-2-(4-fluorophenyl)-N-(2-morpholinoethyl) acetimidamide (**5q**): Yield = 35%, brown solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18-7.16 (m, 4H), 7.06-7.00 (m, 2H), 6.55 (d, *J* = 8.8 Hz, 2H), 6.42 (d, *J* = 8.4 Hz, 2H), 4.89 (s, 1H), 3.85 (bs, *NH*, 1H), 3.49-3.44 (m, 4H), 3.30 (bs, *NH*, 1H), 2.65-2.63 (m, 2H), 2.48-2.46 (m, 2H), 2.40-2.30 (m, 4H). Anal. Calcd for $C_{26}H_{27}Cl_2FN_4O$: C, 62.28; H, 5.43; N, 11.17 Found: C, 62.15; H, 5.32; N, 11.05.

Crystal data

Table 1. Crystal data and structure refinement for **5b**.

Identification code	**5b**	
Empirical formula	C24 H24 Br Cl2 N3	
Formula weight	505.27	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pna2(1)	
Unit cell dimensions	a = 13.3588(10) Å	α= 90°.
	b = 10.7992(8) Å	β= 90°.
	c = 16.6412(12) Å	γ = 90°.
Volume	2400.7(3) $Å^3$	
Z	4	
Density (calculated)	1.398 Mg/m^3	
Absorption coefficient	1.950 mm^{-1}	
F(000)	1032	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.25 to 28.44°.	
Index ranges	-17<=h<=17, -14<=k<=14, -22<=l<=22	
Reflections collected	29277	
Independent reflections	5976 [R(int) = 0.0367]	
Completeness to theta = 28.44°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5976 / 1 / 271	
Goodness-of-fit on F^2	0.993	

Final R indices [I>2sigma(I)]	R1 = 0.0323, wR2 = 0.0665
R indices (all data)	R1 = 0.0668, wR2 = 0.0770
Absolute structure parameter	0.008(7)
Largest diff. peak and hole	0.269 and -0.357 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	4652(1)	3718(1)	2216(1)	71(1)
Cl(1)	13586(1)	6656(1)	1408(1)	82(1)
Cl(2)	6574(1)	9235(1)	-1600(1)	64(1)
C(1)	6689(2)	6878(3)	-1060(2)	51(1)
C(2)	7211(2)	7887(2)	-1328(1)	44(1)
C(3)	8231(2)	7846(2)	-1372(2)	51(1)
C(4)	8741(2)	6779(3)	-1172(2)	50(1)
C(5)	8228(2)	5727(2)	-918(1)	40(1)
C(6)	7198(2)	5801(3)	-864(2)	49(1)
C(7)	6181(2)	5266(3)	1581(2)	56(1)
C(8)	7095(2)	5497(2)	1228(2)	50(1)
C(9)	7736(2)	4545(2)	1008(1)	40(1)
C(10)	7421(2)	3337(2)	1157(2)	55(1)
C(11)	6517(2)	3100(2)	1513(2)	56(1)
C(12)	5908(2)	4069(3)	1722(2)	49(1)
C(13)	10453(2)	5073(2)	1303(1)	42(1)
C(14)	10936(2)	5464(3)	617(2)	60(1)
C(15)	11904(2)	5941(3)	655(2)	65(1)
C(16)	12380(2)	6030(2)	1377(2)	54(1)
C(17)	11910(2)	5667(2)	2067(2)	56(1)
C(18)	10960(2)	5189(2)	2032(1)	52(1)
C(19)	8504(3)	1823(3)	-1111(2)	80(1)
C(20)	9812(3)	888(3)	-247(2)	70(1)
C(21)	10289(3)	2498(3)	-1263(2)	86(1)
C(22)	8772(2)	4840(2)	671(1)	41(1)
C(23)	8950(2)	4148(2)	-125(1)	41(1)
C(24)	9481(2)	2069(2)	-677(2)	51(1)
N(1)	9344(2)	3001(2)	-43(1)	46(1)
N(2)	8733(2)	4598(2)	-816(1)	47(1)
N(3)	9505(2)	4524(2)	1287(1)	48(1)

Table 3. Bond lengths [Å] and angles [°] for **5b**.

Br(1)-C(12)	1.906(3)
Cl(1)-C(16)	1.748(3)
Cl(2)-C(2)	1.747(3)
C(1)-C(2)	1.369(4)
C(1)-C(6)	1.385(4)
C(2)-C(3)	1.365(4)
C(3)-C(4)	1.379(4)
C(4)-C(5)	1.392(4)
C(5)-C(6)	1.381(4)
C(5)-N(2)	1.404(3)
C(7)-C(12)	1.363(4)
C(7)-C(8)	1.378(4)
C(8)-C(9)	1.387(3)
C(9)-C(10)	1.393(3)
C(9)-C(22)	1.527(4)
C(10)-C(11)	1.370(4)
C(11)-C(12)	1.370(4)
C(13)-C(14)	1.377(4)
C(13)-C(18)	1.395(3)
C(13)-N(3)	1.398(3)
C(14)-C(15)	1.394(4)
C(15)-C(16)	1.362(4)
C(16)-C(17)	1.366(4)
C(17)-C(18)	1.371(4)
C(19)-C(24)	1.514(4)
C(20)-C(24)	1.527(4)
C(21)-C(24)	1.527(4)
C(22)-N(3)	1.458(3)
C(22)-C(23)	1.540(3)
C(23)-N(2)	1.282(3)
C(23)-N(1)	1.352(3)
C(24)-N(1)	1.471(3)

C(2)-C(1)-C(6)	119.6(3)
C(3)-C(2)-C(1)	120.1(2)
C(3)-C(2)-Cl(2)	119.9(2)
C(1)-C(2)-Cl(2)	120.0(2)
C(2)-C(3)-C(4)	120.4(3)
C(3)-C(4)-C(5)	120.8(3)
C(6)-C(5)-C(4)	117.6(2)
C(6)-C(5)-N(2)	121.4(2)
C(4)-C(5)-N(2)	120.6(2)
C(5)-C(6)-C(1)	121.5(3)
C(12)-C(7)-C(8)	118.9(3)
C(7)-C(8)-C(9)	121.6(2)
C(8)-C(9)-C(10)	117.5(2)
C(8)-C(9)-C(22)	120.1(2)
C(10)-C(9)-C(22)	122.3(2)
C(11)-C(10)-C(9)	121.2(2)
C(10)-C(11)-C(12)	119.4(3)
C(7)-C(12)-C(11)	121.4(3)
C(7)-C(12)-Br(1)	119.9(2)
C(11)-C(12)-Br(1)	118.7(2)
C(14)-C(13)-C(18)	117.7(2)
C(14)-C(13)-N(3)	122.6(2)
C(18)-C(13)-N(3)	119.6(2)
C(13)-C(14)-C(15)	120.8(3)
C(16)-C(15)-C(14)	119.9(3)
C(15)-C(16)-C(17)	120.4(3)
C(15)-C(16)-Cl(1)	118.9(2)
C(17)-C(16)-Cl(1)	120.6(2)
C(16)-C(17)-C(18)	119.8(2)
C(17)-C(18)-C(13)	121.4(2)
N(3)-C(22)-C(9)	107.60(19)
N(3)-C(22)-C(23)	112.80(19)
C(9)-C(22)-C(23)	110.80(19)
N(2)-C(23)-N(1)	121.8(2)
N(2)-C(23)-C(22)	123.6(2)

N(1)-C(23)-C(22)	114.6(2)
N(1)-C(24)-C(19)	110.8(2)
N(1)-C(24)-C(21)	109.8(2)
C(19)-C(24)-C(21)	111.0(3)
N(1)-C(24)-C(20)	105.7(2)
C(19)-C(24)-C(20)	109.1(3)
C(21)-C(24)-C(20)	110.3(3)
C(23)-N(1)-C(24)	127.1(2)
C(23)-N(2)-C(5)	123.2(2)
C(13)-N(3)-C(22)	121.5(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	52(1)	83(1)	77(1)	16(1)	10(1)	-4(1)
Cl(1)	51(1)	76(1)	121(1)	24(1)	-21(1)	-2(1)
Cl(2)	75(1)	55(1)	63(1)	6(1)	-9(1)	14(1)
C(1)	36(1)	60(2)	55(2)	4(1)	-7(1)	0(1)
C(2)	49(2)	48(2)	35(1)	-2(1)	-4(1)	4(1)
C(3)	55(2)	45(2)	54(2)	4(1)	7(1)	-8(1)
C(4)	38(1)	53(2)	59(2)	0(1)	7(1)	-2(1)
C(5)	45(2)	42(1)	32(1)	-2(1)	-3(1)	-1(1)
C(6)	46(2)	48(2)	54(2)	8(1)	-2(1)	-7(1)
C(7)	55(2)	50(2)	62(2)	-6(1)	5(1)	5(1)
C(8)	52(2)	35(1)	65(2)	-1(1)	1(1)	-2(1)
C(9)	46(2)	37(1)	36(1)	-1(1)	2(1)	-1(1)
C(10)	63(2)	40(1)	61(2)	2(1)	12(1)	5(1)
C(11)	60(2)	41(2)	66(2)	6(1)	8(1)	-7(1)
C(12)	48(2)	57(2)	41(1)	4(1)	4(1)	-5(1)
C(13)	53(2)	37(1)	36(1)	-1(1)	-3(1)	4(1)
C(14)	61(2)	77(2)	40(1)	13(1)	-6(1)	-11(2)
C(15)	61(2)	83(2)	50(2)	20(1)	1(2)	-4(2)
C(16)	51(2)	43(2)	69(2)	7(1)	-13(2)	6(1)
C(17)	67(2)	57(2)	44(2)	-5(1)	-16(1)	2(1)
C(18)	62(2)	57(2)	38(2)	0(1)	-2(1)	-1(1)
C(19)	87(2)	63(2)	89(2)	-22(2)	-33(2)	7(2)
C(20)	97(3)	49(2)	65(2)	-9(1)	-9(2)	21(2)
C(21)	97(3)	85(2)	76(2)	-7(2)	33(2)	13(2)
C(22)	50(2)	35(1)	38(1)	1(1)	0(1)	0(1)
C(23)	41(1)	41(1)	41(1)	-2(1)	2(1)	1(1)
C(24)	61(2)	43(2)	47(2)	-8(1)	-4(1)	7(1)
N(1)	55(1)	46(1)	38(1)	-2(1)	-2(1)	11(1)
N(2)	56(2)	47(1)	38(1)	0(1)	1(1)	9(1)
N(3)	58(1)	48(1)	36(1)	7(1)	-2(1)	-8(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5b**.

	x	y	z	U(eq)
H(1A)	5996	6914	-1009	61
H(3A)	8584	8542	-1539	62
H(4A)	9435	6762	-1206	60
H(6A)	6838	5114	-691	59
H(7A)	5758	5914	1721	67
H(8A)	7287	6312	1135	60
H(10A)	7833	2679	1012	66
H(11A)	6318	2289	1612	67
H(14A)	10612	5410	124	71
H(15A)	12225	6199	188	78
H(17A)	12233	5744	2559	67
H(18A)	10647	4937	2503	63
H(19A)	8291	2563	-1382	120
H(19B)	8003	1579	-729	120
H(19C)	8599	1173	-1497	120
H(20A)	10437	1034	21	106
H(20B)	9894	235	-633	106
H(20C)	9314	654	140	106
H(21A)	10074	3244	-1525	129
H(21B)	10404	1866	-1658	129
H(21C)	10898	2655	-974	129
H(22A)	8811	5732	568	49
H(1B)	9534	2795	432	55
H(3C)	9352	3987	1648	57

Table 1. Crystal data and structure refinement for **5n**

Identification code	**5n**	
Empirical formula	C26 H26 Cl3 N3	
Formula weight	486.85	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 12.9999(9) Å	α= 90°.
	b = 11.9658(10) Å	β= 92.120(4)°.
	c = 16.7478(12) Å	γ = 90°.
Volume	2603.4(3) $Å^3$	
Z	4	
Density (calculated)	1.242 Mg/m^3	
Absorption coefficient	0.370 mm^{-1}	
F(000)	1016	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	1.95 to 26.49°.	
Index ranges	-13<=h<=15, -13<=k<=12, -20<=l<=20	
Reflections collected	21584	
Independent reflections	4970 [R(int) = 0.0450]	
Completeness to theta = 26.49°	92.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4970 / 0 / 289	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0875, wR2 = 0.2237	
R indices (all data)	R1 = 0.1786, wR2 = 0.2754	
Largest diff. peak and hole	0.594 and -0.509 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5n**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	4674(1)	7238(2)	10673(1)	130(1)
Cl(3)	7714(1)	1262(3)	6988(2)	182(1)
N(1)	3100(2)	5619(3)	7519(2)	55(1)
N(2)	3443(3)	2564(3)	7596(2)	58(1)
C(13)	3194(3)	3627(3)	7954(2)	56(1)
C(14)	3021(3)	4578(4)	7349(2)	53(1)
N(3)	2756(3)	4243(3)	6608(2)	66(1)
C(4)	3471(3)	5952(3)	8288(2)	56(1)
C(15)	4442(3)	2286(4)	7433(2)	60(1)
Cl(2)	-461(2)	3228(2)	10016(2)	215(2)
C(3)	4506(4)	6150(4)	8425(3)	74(1)
C(7)	2264(4)	3495(4)	8455(3)	65(1)
C(16)	4715(4)	1163(4)	7372(3)	74(1)
C(6)	3181(4)	6566(4)	9628(3)	81(2)
C(21)	2640(4)	4960(4)	5907(2)	66(1)
C(1)	4222(5)	6734(4)	9758(3)	84(2)
C(12)	2351(5)	3621(4)	9277(3)	91(2)
C(8)	1320(4)	3263(4)	8117(4)	80(2)
C(5)	2812(4)	6187(4)	8892(3)	69(1)
C(19)	6195(5)	2758(7)	7199(3)	103(2)
C(17)	5706(5)	863(6)	7224(4)	101(2)
C(18)	6454(5)	1661(8)	7147(4)	106(2)
C(20)	5197(4)	3075(5)	7335(3)	78(1)
C(22)	3402(5)	4616(6)	5302(4)	114(2)
C(10)	608(7)	3320(6)	9420(6)	117(2)
C(26)	1593(4)	4951(6)	5574(3)	107(2)
C(23)	3273(5)	5276(7)	4527(4)	130(3)
C(2)	4882(4)	6524(5)	9155(3)	89(2)
C(9)	456(5)	3154(5)	8610(5)	108(2)
C(11)	1522(8)	3535(6)	9756(4)	122(2)

C(25)	1483(5)	5615(7)	4792(4)	130(3)
C(24)	2214(6)	5215(6)	4189(3)	118(2)

Table 3. Bond lengths [Å] and angles [°] for **5n.**

Cl(1)-C(1)	1.730(5)
Cl(3)-C(18)	1.736(6)
N(1)-C(14)	1.280(5)
N(1)-C(4)	1.416(5)
N(2)-C(15)	1.377(5)
N(2)-C(13)	1.449(5)
C(13)-C(7)	1.505(6)
C(13)-C(14)	1.536(6)
C(14)-N(3)	1.337(5)
N(3)-C(21)	1.458(5)
C(4)-C(3)	1.377(6)
C(4)-C(5)	1.379(6)
C(15)-C(20)	1.377(6)
C(15)-C(16)	1.395(6)
Cl(2)-C(10)	1.745(7)
C(3)-C(2)	1.376(6)
C(7)-C(8)	1.362(7)
C(7)-C(12)	1.386(7)
C(16)-C(17)	1.368(7)
C(6)-C(1)	1.377(7)
C(6)-C(5)	1.383(6)
C(21)-C(26)	1.453(7)
C(21)-C(22)	1.500(7)
C(1)-C(2)	1.372(7)
C(12)-C(11)	1.370(9)
C(8)-C(9)	1.424(8)
C(19)-C(18)	1.359(9)
C(19)-C(20)	1.378(8)
C(17)-C(18)	1.372(9)
C(22)-C(23)	1.524(8)
C(10)-C(11)	1.321(10)
C(10)-C(9)	1.378(10)
C(26)-C(25)	1.533(7)

C(23)-C(24)	1.471(8)
C(25)-C(24)	1.490(9)

C(14)-N(1)-C(4)	119.9(3)
C(15)-N(2)-C(13)	121.4(3)
N(2)-C(13)-C(7)	109.6(3)
N(2)-C(13)-C(14)	114.0(3)
C(7)-C(13)-C(14)	110.2(3)
N(1)-C(14)-N(3)	121.0(4)
N(1)-C(14)-C(13)	124.4(4)
N(3)-C(14)-C(13)	114.6(4)
C(14)-N(3)-C(21)	125.9(4)
C(3)-C(4)-C(5)	118.2(4)
C(3)-C(4)-N(1)	119.8(4)
C(5)-C(4)-N(1)	121.7(4)
C(20)-C(15)-N(2)	122.7(4)
C(20)-C(15)-C(16)	117.8(4)
N(2)-C(15)-C(16)	119.5(4)
C(2)-C(3)-C(4)	121.1(5)
C(8)-C(7)-C(12)	118.3(5)
C(8)-C(7)-C(13)	121.3(4)
C(12)-C(7)-C(13)	120.3(5)
C(17)-C(16)-C(15)	120.7(5)
C(1)-C(6)-C(5)	119.6(5)
C(26)-C(21)-N(3)	111.9(4)
C(26)-C(21)-C(22)	111.7(4)
N(3)-C(21)-C(22)	109.4(4)
C(2)-C(1)-C(6)	119.7(5)
C(2)-C(1)-Cl(1)	120.9(5)
C(6)-C(1)-Cl(1)	119.3(5)
C(11)-C(12)-C(7)	122.4(6)
C(7)-C(8)-C(9)	119.7(6)
C(4)-C(5)-C(6)	121.1(4)
C(18)-C(19)-C(20)	120.9(6)
C(16)-C(17)-C(18)	120.7(6)

C(19)-C(18)-C(17)	119.2(5)
C(19)-C(18)-Cl(3)	120.9(6)
C(17)-C(18)-Cl(3)	119.9(6)
C(15)-C(20)-C(19)	120.7(6)
C(21)-C(22)-C(23)	112.3(5)
C(11)-C(10)-C(9)	122.8(7)
C(11)-C(10)-Cl(2)	119.5(8)
C(9)-C(10)-Cl(2)	117.7(7)
C(21)-C(26)-C(25)	112.4(5)
C(24)-C(23)-C(22)	111.9(5)
C(1)-C(2)-C(3)	120.1(5)
C(10)-C(9)-C(8)	118.0(6)
C(10)-C(11)-C(12)	118.7(7)
C(24)-C(25)-C(26)	111.7(6)
C(23)-C(24)-C(25)	109.6(5)

Symmetry transformations used to generate equivalent atoms:

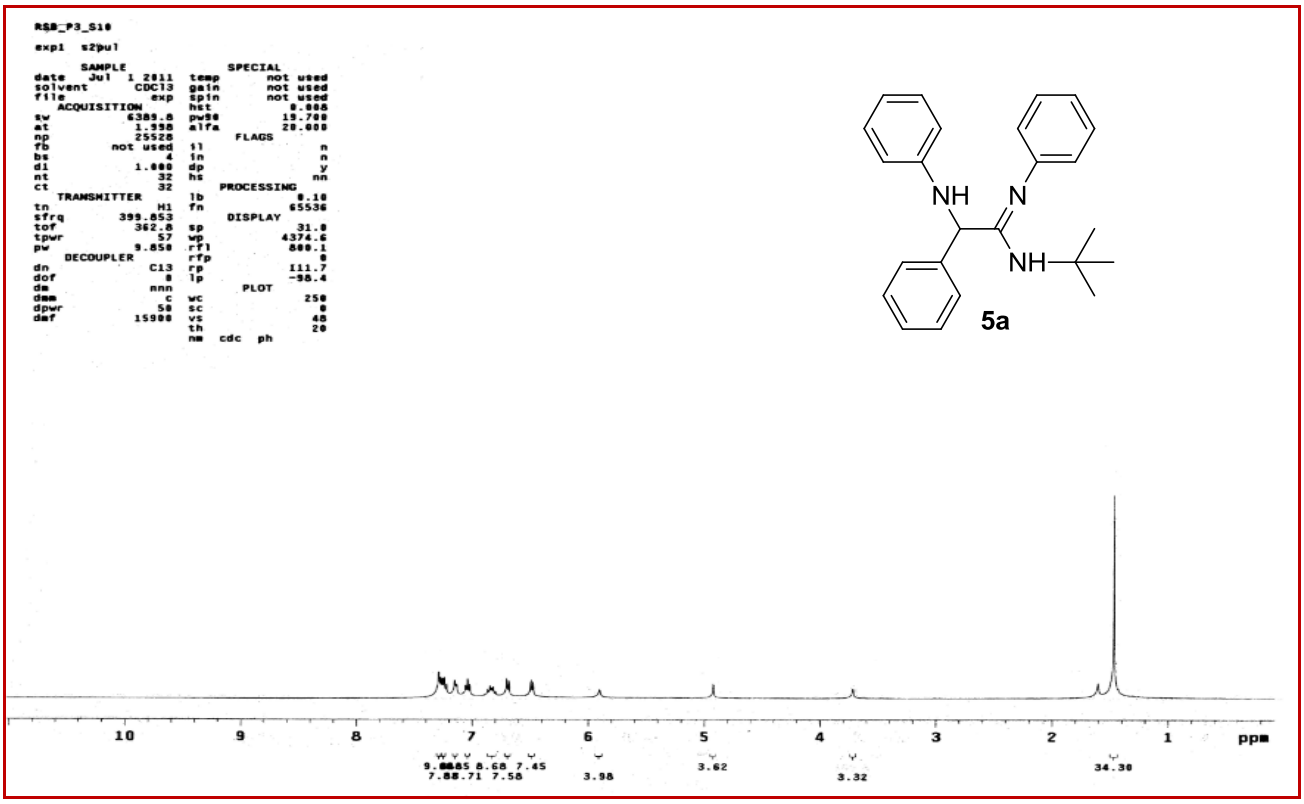
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5n**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	141(2)	167(2)	78(1)	-35(1)	-43(1)	38(1)
Cl(3)	78(1)	282(3)	189(2)	40(2)	51(1)	54(2)
N(1)	57(2)	51(2)	57(2)	1(2)	-1(2)	-2(2)
N(2)	50(2)	50(2)	73(2)	-3(2)	-1(2)	-3(2)
C(13)	58(3)	52(3)	57(2)	2(2)	-2(2)	-1(2)
C(14)	45(2)	59(3)	56(3)	4(2)	-1(2)	-1(2)
N(3)	87(3)	50(2)	58(2)	3(2)	-14(2)	-7(2)
C(4)	62(3)	50(3)	56(3)	2(2)	-2(2)	0(2)
C(15)	61(3)	68(3)	53(3)	8(2)	3(2)	1(3)
Cl(2)	193(3)	207(3)	257(3)	1(2)	160(3)	-17(2)
C(3)	61(3)	90(4)	72(3)	-16(3)	1(2)	-2(3)
C(7)	72(3)	52(3)	71(3)	5(2)	14(3)	5(2)
C(16)	63(3)	75(4)	86(3)	4(3)	9(3)	8(3)
C(6)	87(4)	96(4)	61(3)	1(3)	5(3)	14(3)
C(21)	88(3)	56(3)	54(3)	2(2)	-6(2)	1(2)
C(1)	97(4)	87(4)	65(3)	-9(3)	-16(3)	18(3)
C(12)	112(5)	85(4)	77(4)	11(3)	18(3)	7(3)
C(8)	66(3)	68(3)	107(4)	-5(3)	18(3)	-1(3)
C(5)	65(3)	78(3)	65(3)	4(3)	1(2)	4(2)
C(19)	64(4)	147(6)	97(4)	41(4)	18(3)	-13(4)
C(17)	83(4)	113(5)	109(4)	2(4)	18(3)	30(4)
C(18)	60(4)	160(7)	99(4)	22(4)	28(3)	32(4)
C(20)	69(3)	87(4)	79(3)	13(3)	11(3)	-7(3)
C(22)	84(4)	160(6)	97(4)	29(4)	15(3)	5(4)
C(10)	116(6)	95(5)	144(7)	3(5)	69(6)	-1(4)
C(26)	77(4)	154(6)	89(4)	45(4)	-4(3)	8(4)
C(23)	110(5)	194(8)	87(4)	46(5)	22(4)	-5(5)
C(2)	70(3)	114(5)	83(4)	-19(3)	-14(3)	4(3)
C(9)	86(4)	75(4)	164(7)	3(4)	28(5)	-7(3)
C(11)	150(7)	122(6)	97(5)	13(4)	56(5)	0(5)

C(25)	90(4)	183(7)	114(5)	72(5)	-16(4)	-2(5)
C(24)	146(6)	142(6)	64(4)	17(4)	-14(4)	-36(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5n**.

	x	y	z	U(eq)
H(2A)	2957	2096	7482	70
H(13A)	3775	3839	8313	67
H(3A)	2643	3540	6540	79
H(3B)	4958	6028	8016	89
H(16A)	4219	612	7432	89
H(6A)	2728	6706	10034	97
H(21A)	2804	5726	6074	80
H(12A)	2996	3768	9513	109
H(8A)	1239	3176	7566	96
H(5A)	2107	6089	8803	83
H(19A)	6697	3303	7142	123
H(17A)	5874	112	7175	121
H(20A)	5032	3830	7360	94
H(22A)	3316	3826	5187	136
H(22B)	4093	4726	5525	136
H(26A)	1137	5267	5960	128
H(26B)	1382	4184	5475	128
H(23A)	3452	6052	4628	156
H(23B)	3743	4985	4141	156
H(2B)	5585	6636	9241	107
H(9A)	-192	2976	8392	130
H(11A)	1601	3627	10306	146
H(25A)	784	5543	4575	155
H(25B)	1610	6399	4901	155
H(24A)	2051	4450	4041	141
H(24B)	2150	5674	3713	141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date Jul 1 2011
solvent CDCl3
file exp
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sw 6389.8
at 1.998
np 25528
fb not used
bs 4
di 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp 31.0
wp 4374.6
rfl 880.1
rfp 0
rp 111.7
lp -98.4
PLOT
wc 250
sc 0
vs 48
th 20
nm cdc ph
NH
N
NH
5a
10
9
8
7
6
5
4
3
2
1
ppm
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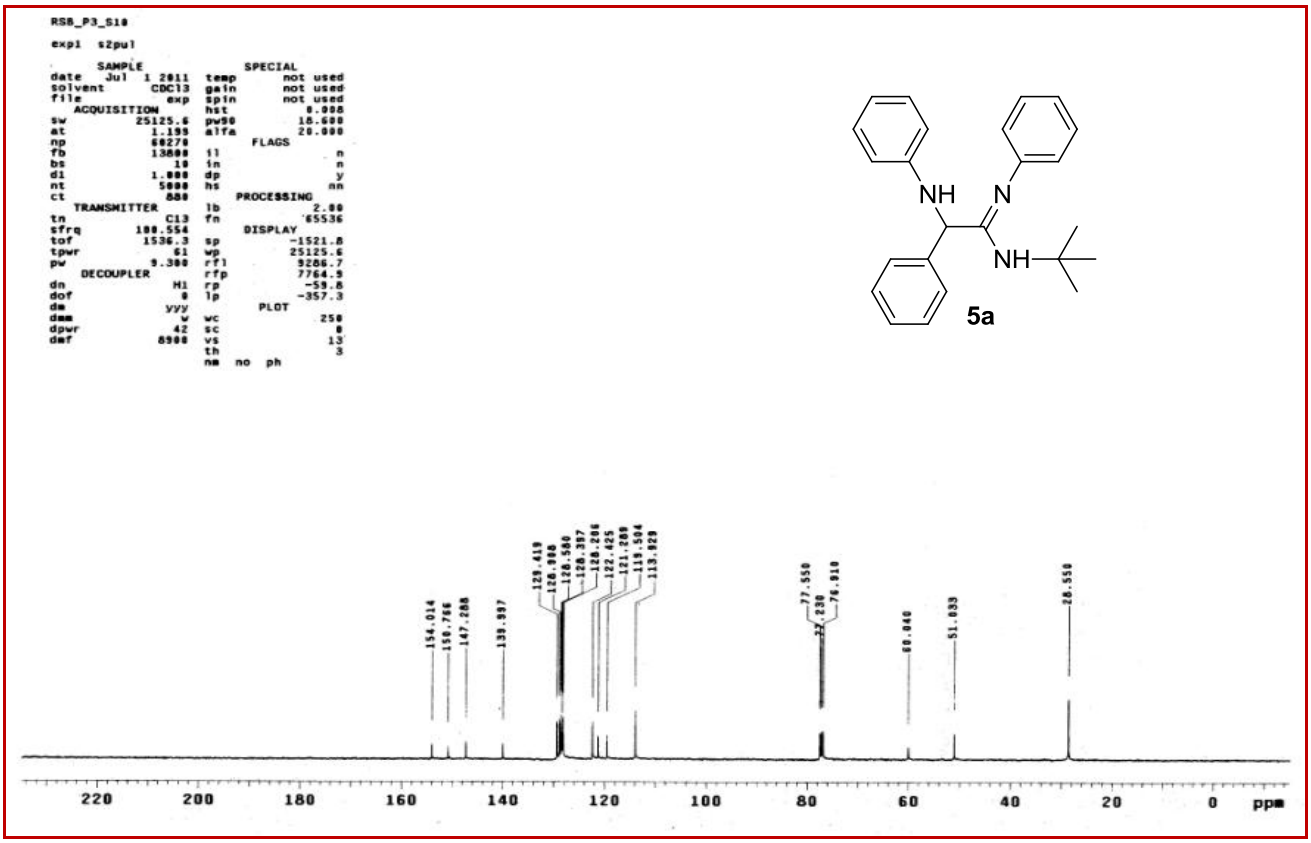
8.85
8.68
7.88
7.71
7.58
7.45
3.98
3.62
3.32
34.30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date Jul 1 2011
solvent CDCl3
file exp
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at 1.195
np 60270
fb 13888
bs 16
di 1.000
nt 5888
ct 880
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 10.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -1521.8
wp 25125.6
rfl 9286.7
rfp 7764.5
rp -59.8
lp -357.3
PLOT
wc 250
sc 0
vs 13
th 3
nm no ph
NH
N
NH
5a
154.014
150.766
147.288
139.997
129.419
128.998
128.580
128.397
128.206
122.425
121.788
119.504
113.929
77.550
77.230
76.910
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51.033
28.550
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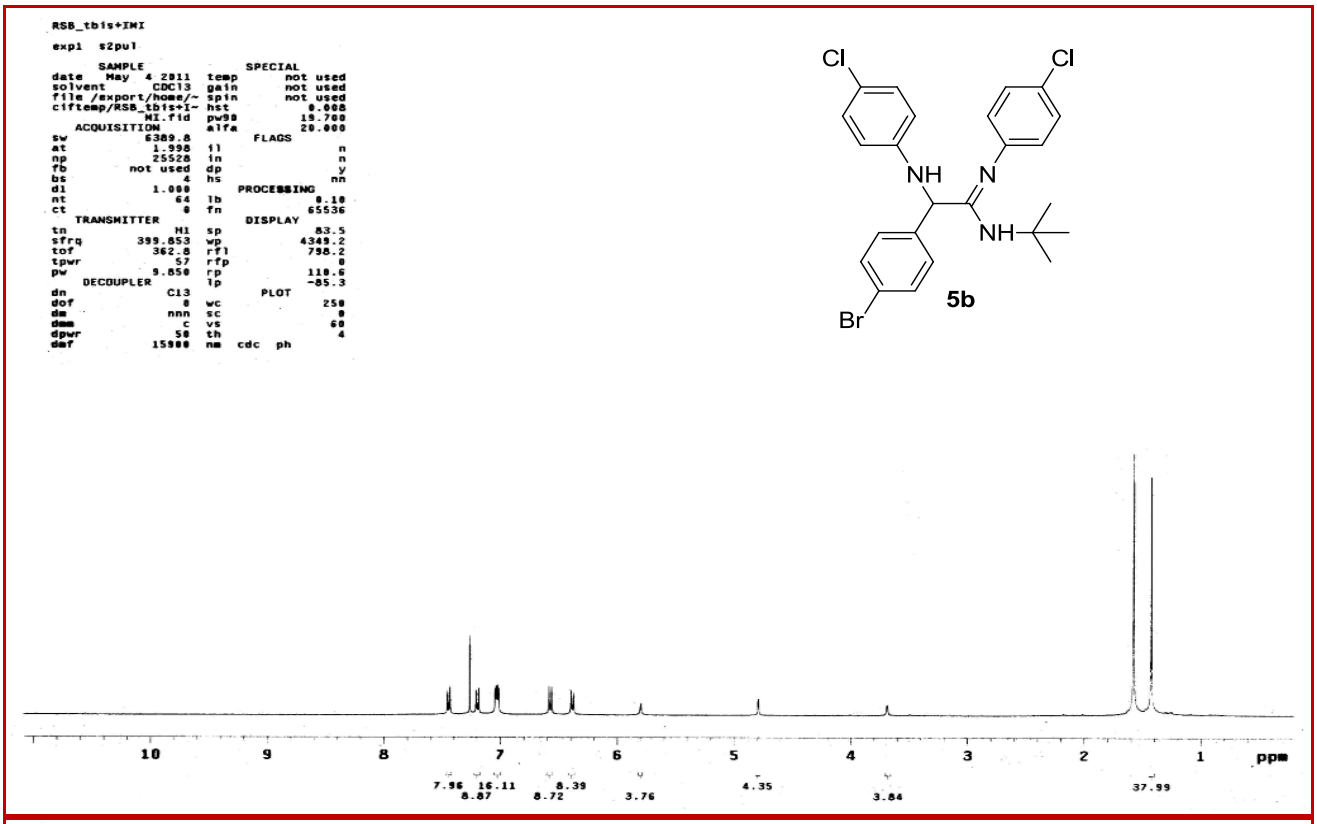
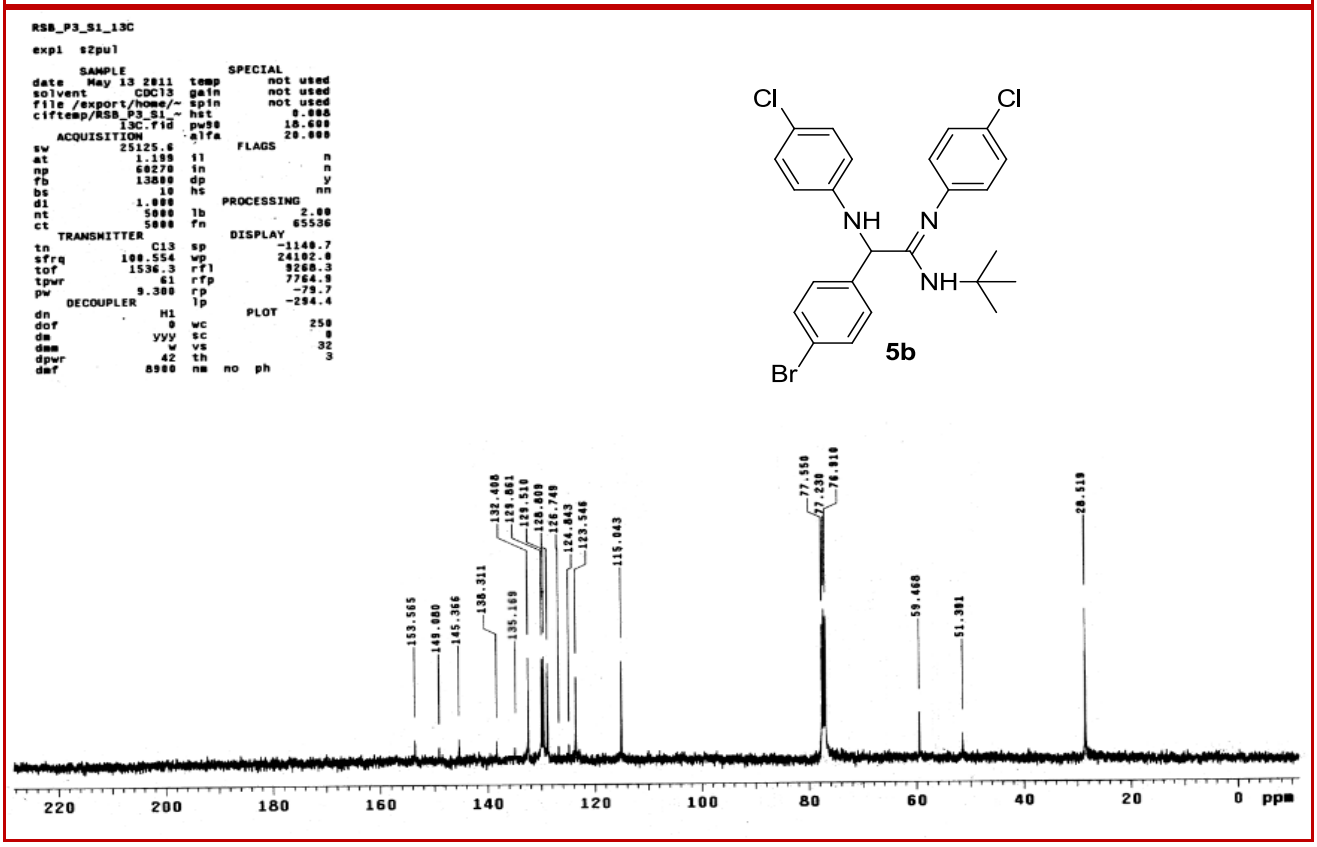
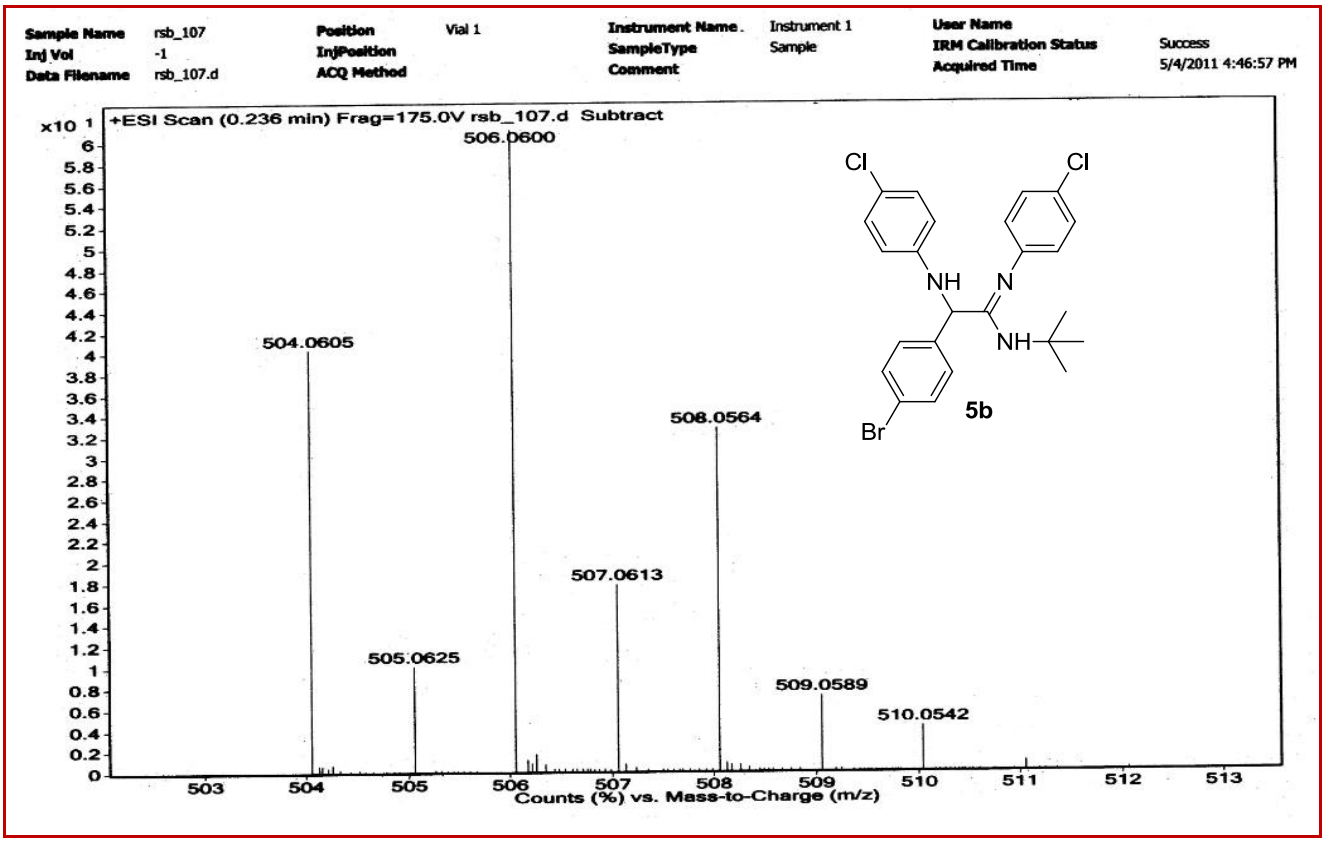
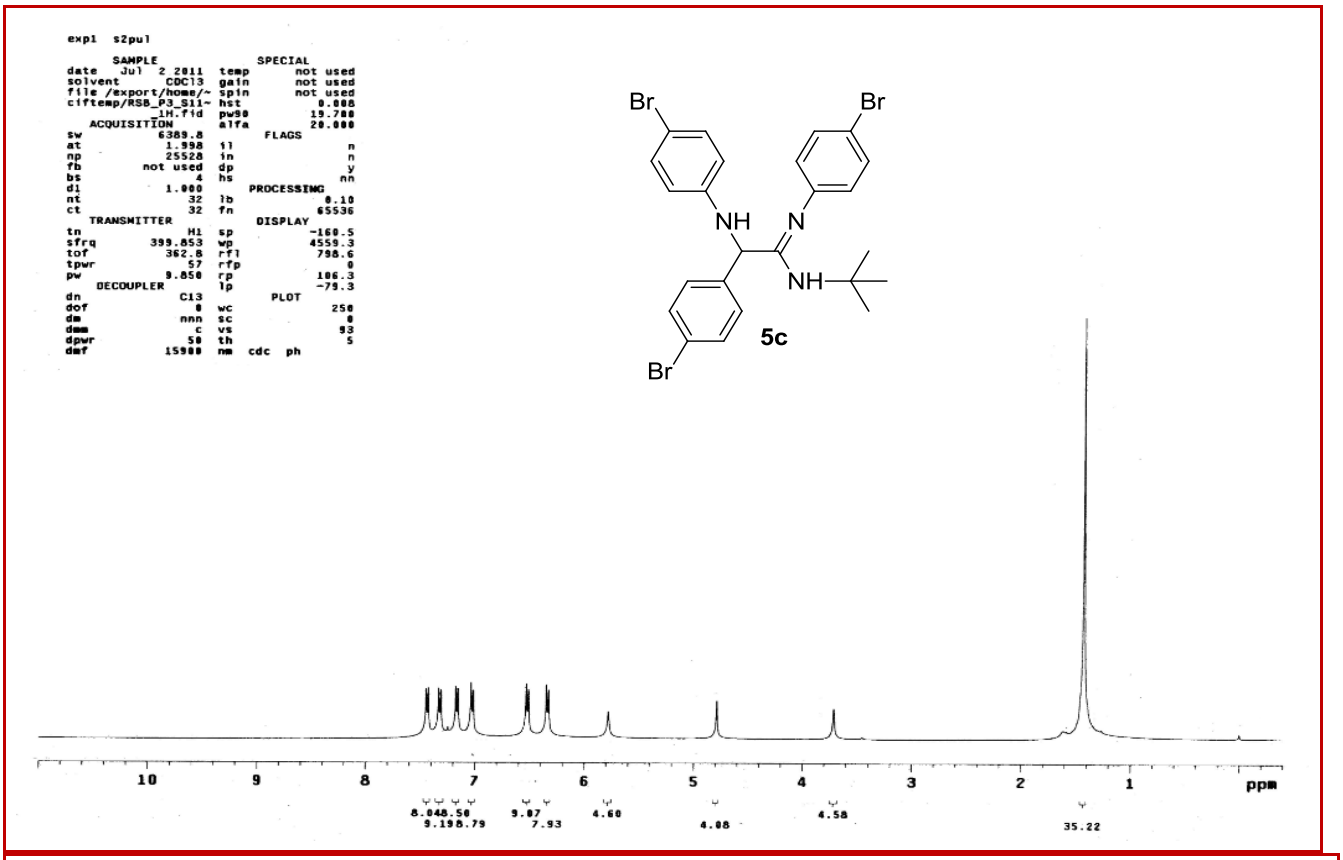
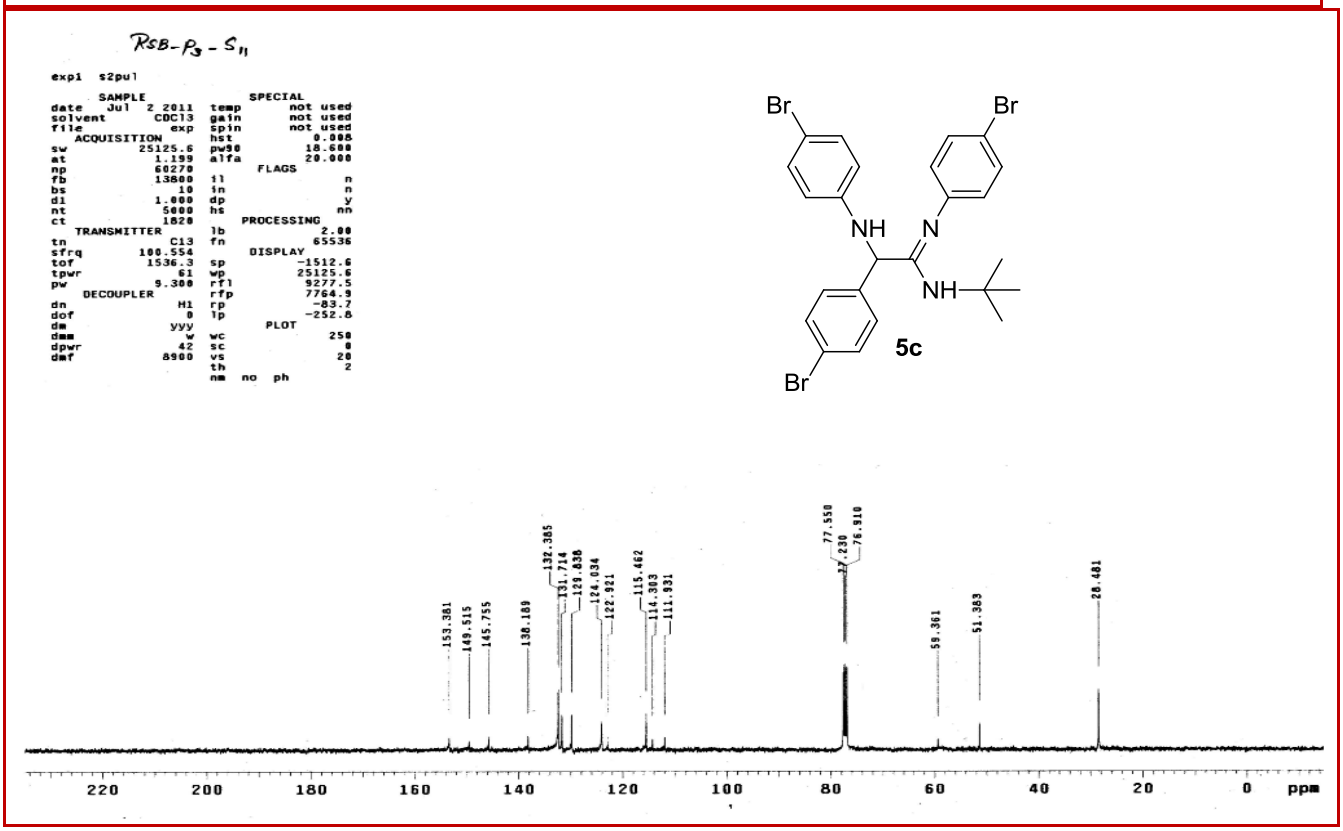
200
180
160
140
120
100
80
60
40
20
0
ppm

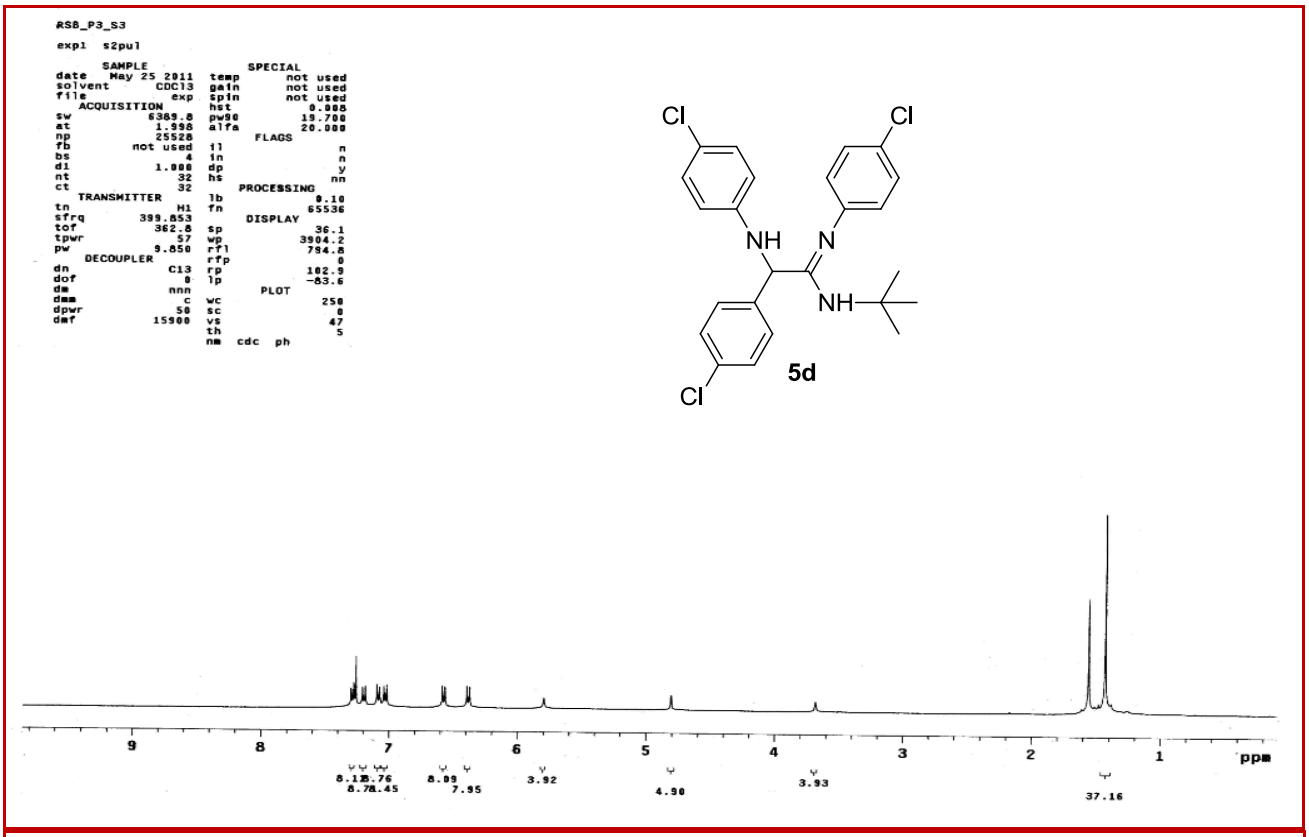


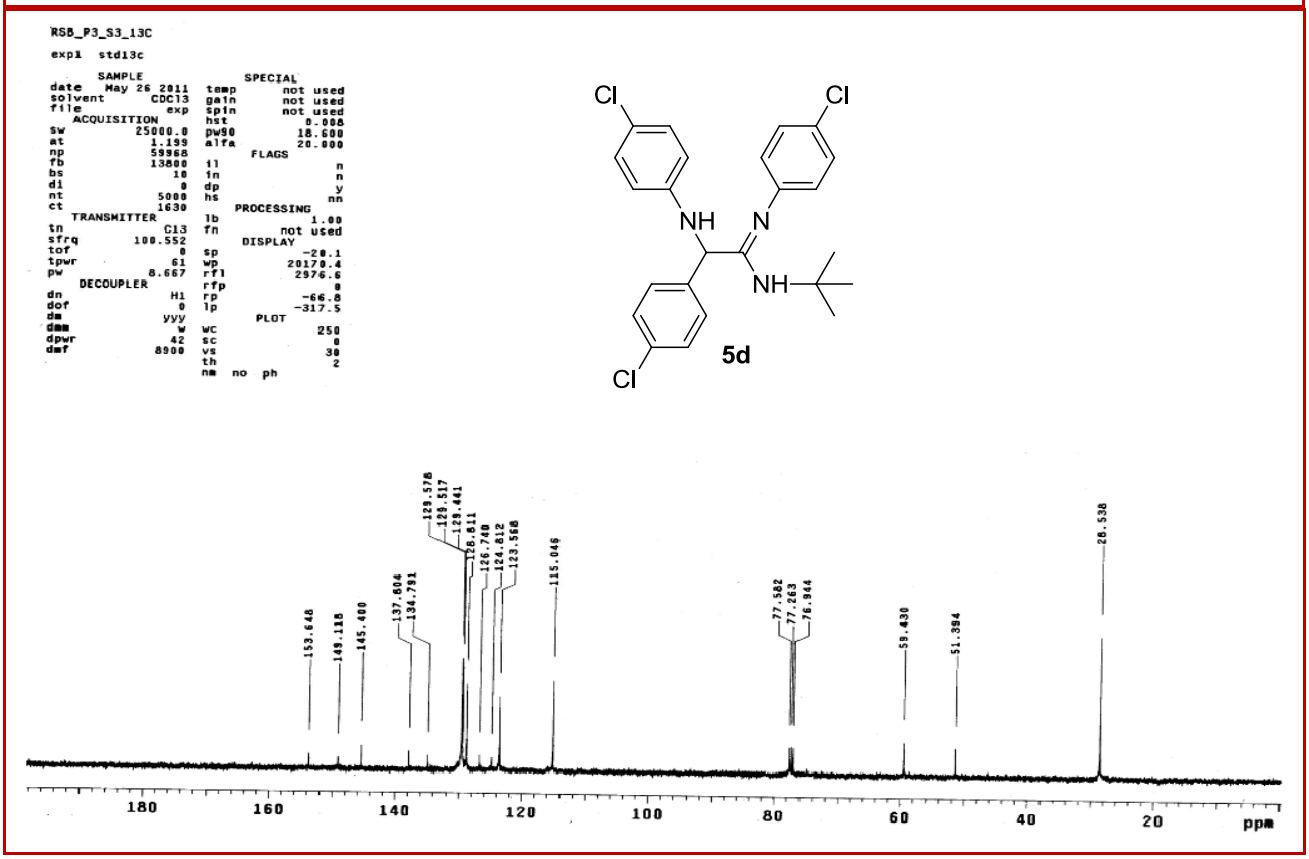


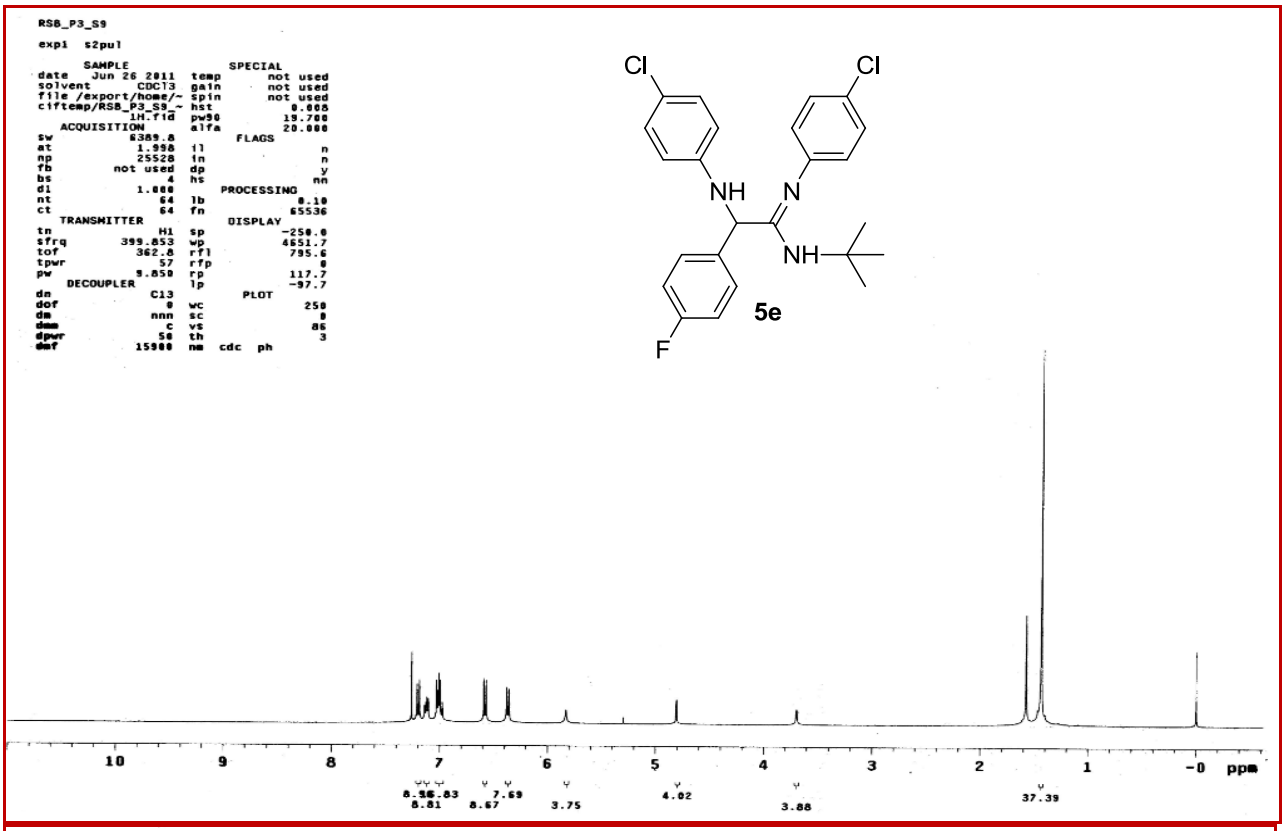


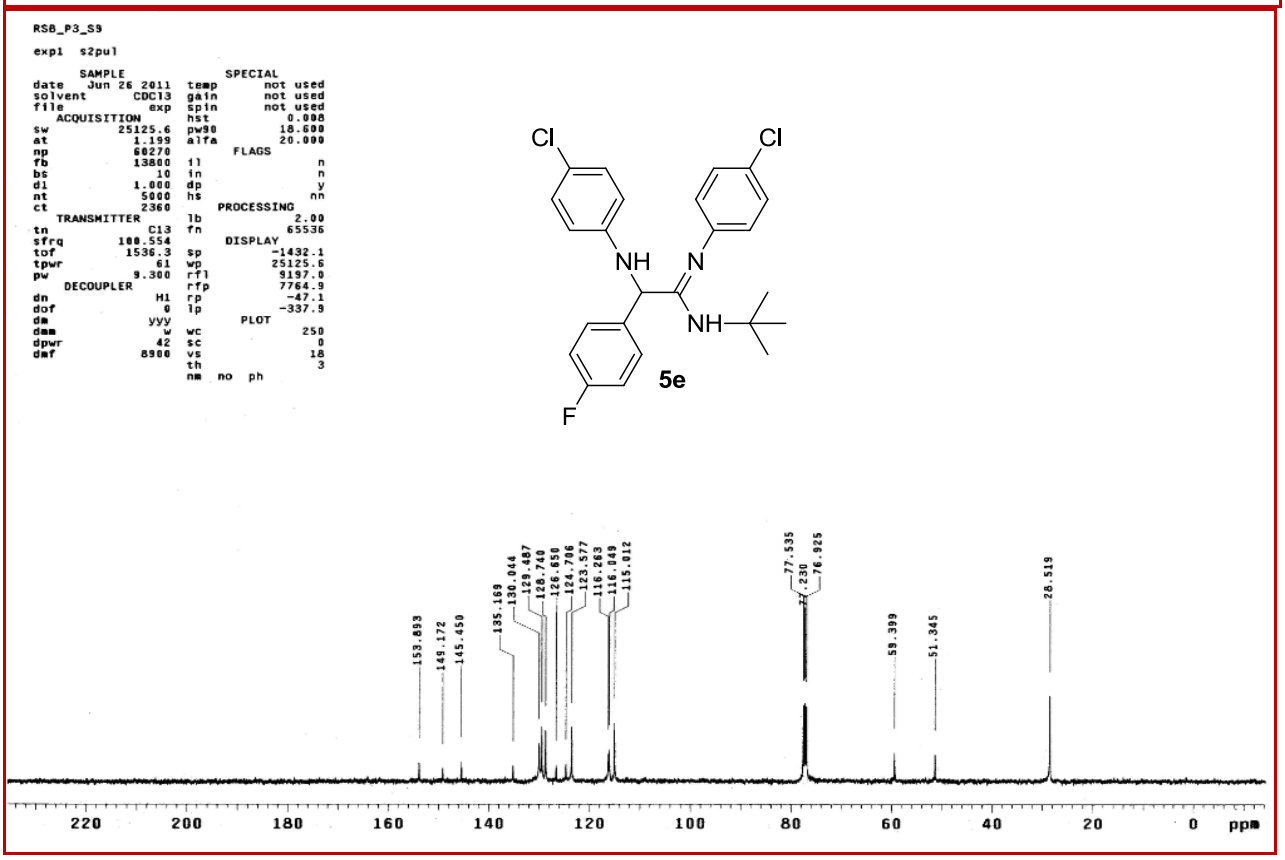


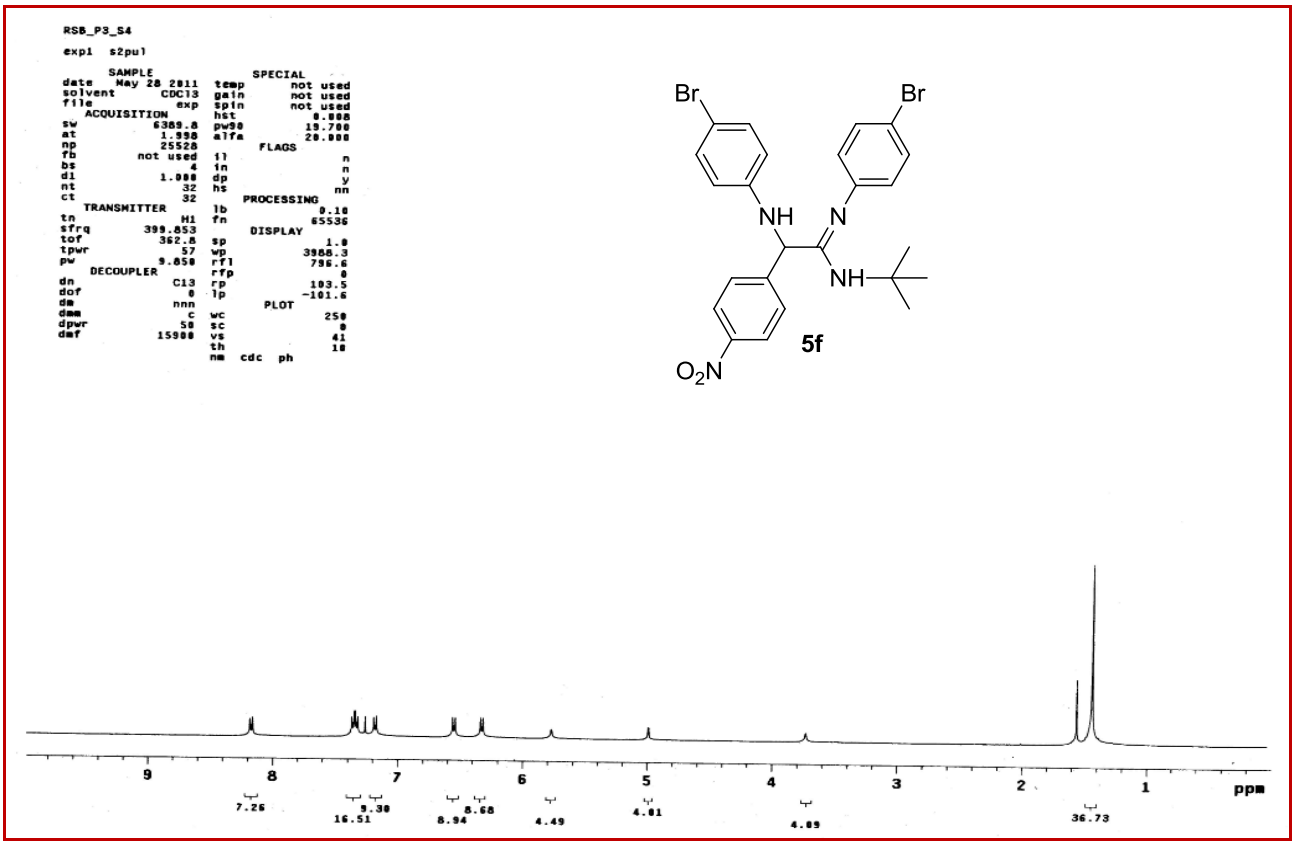








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date May 28 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 58
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.000
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp 1.0
wp 3988.3
rfl 796.6
rfp 0
rp 103.5
lp -101.6
PLOT
wc 250
sc 0
vs 41
th 10
nm cdc ph
Br
Br
NH
N
NH
O2N
5f
9
8
7
6
5
4
3
2
1
ppm
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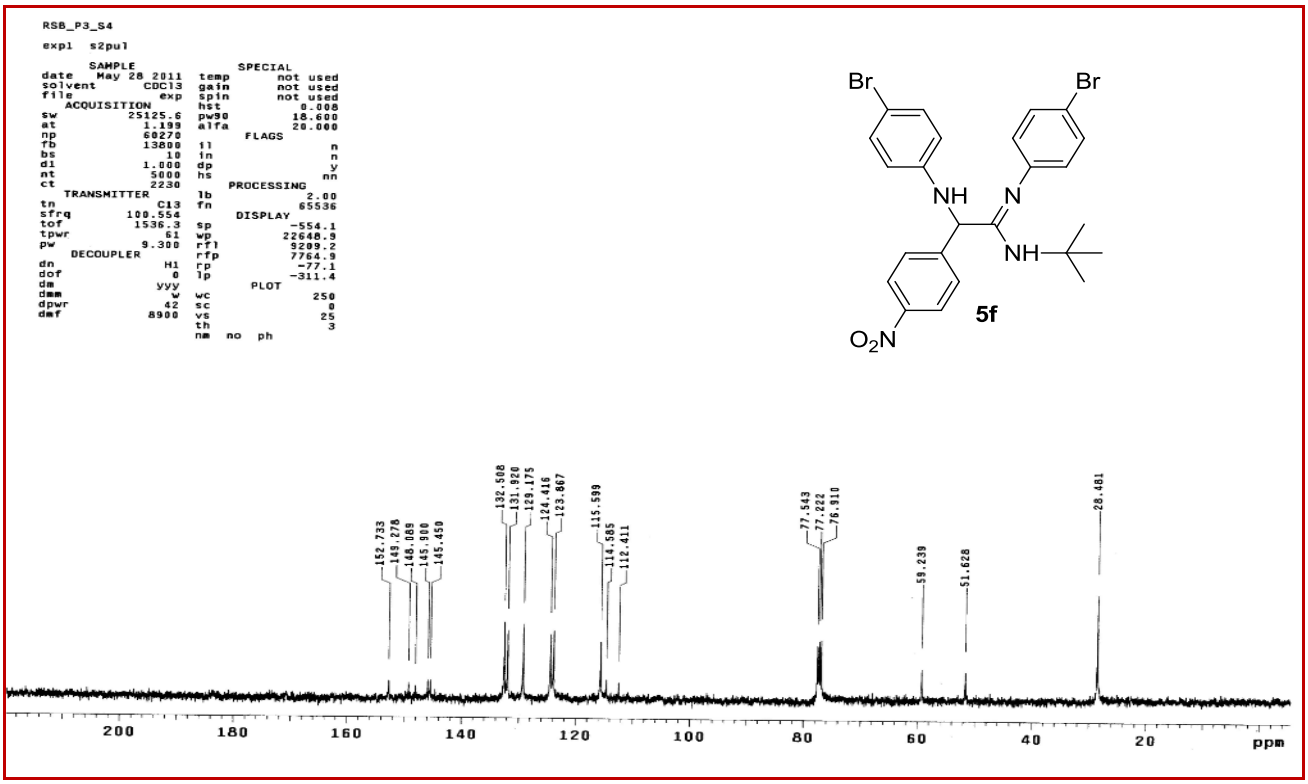
9.30
8.94
8.68
4.49
4.01
4.89
36.73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expl s2pul
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date May 28 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 2230
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.000
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -554.1
wp 22648.9
rfl 9209.2
rfp 7764.9
rp -77.1
lp -311.4
PLOT
wc 250
sc 0
vs 25
th 3
nm no ph
Br
Br
NH
N
NH
O2N
5f
152.733
149.278
148.089
145.900
145.450
132.508
131.920
129.175
124.416
123.867
115.599
114.585
112.411
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59.239
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140
120
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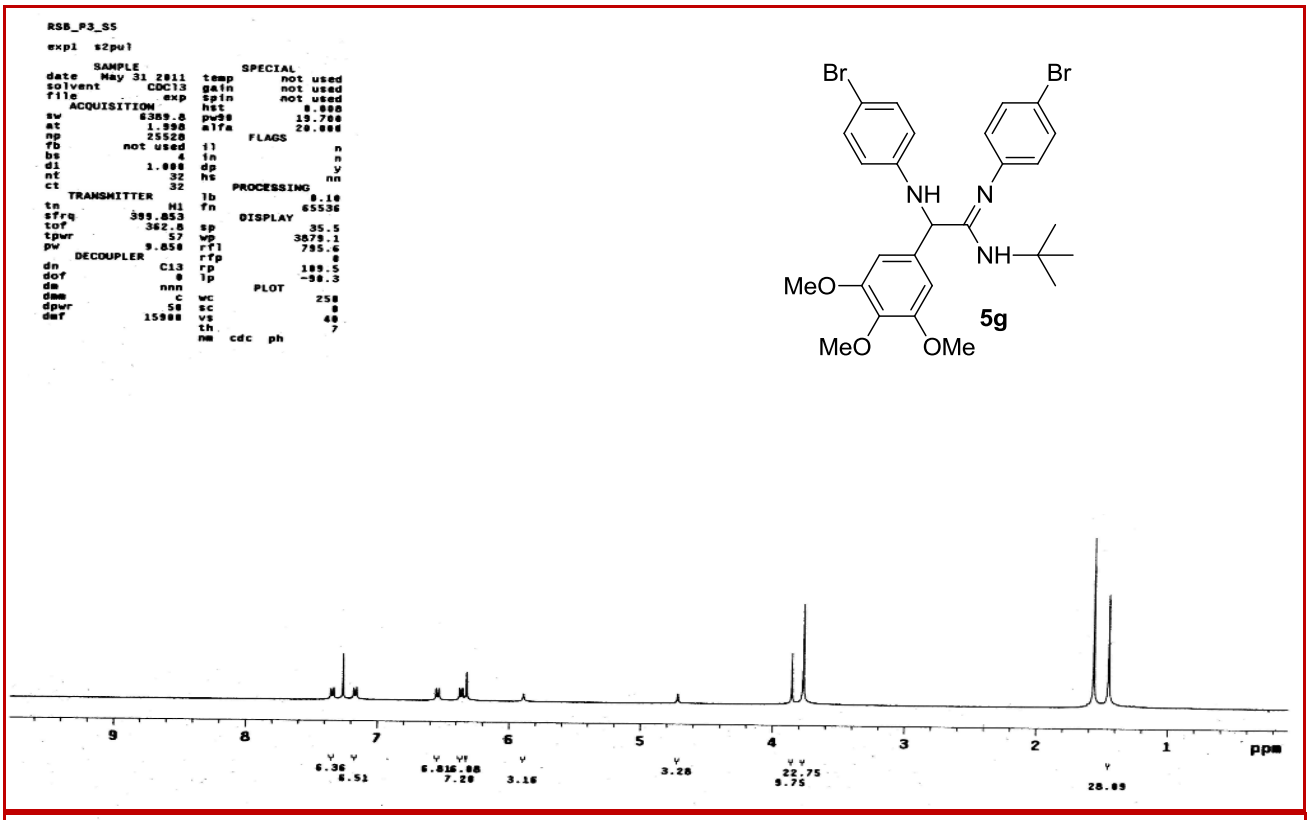
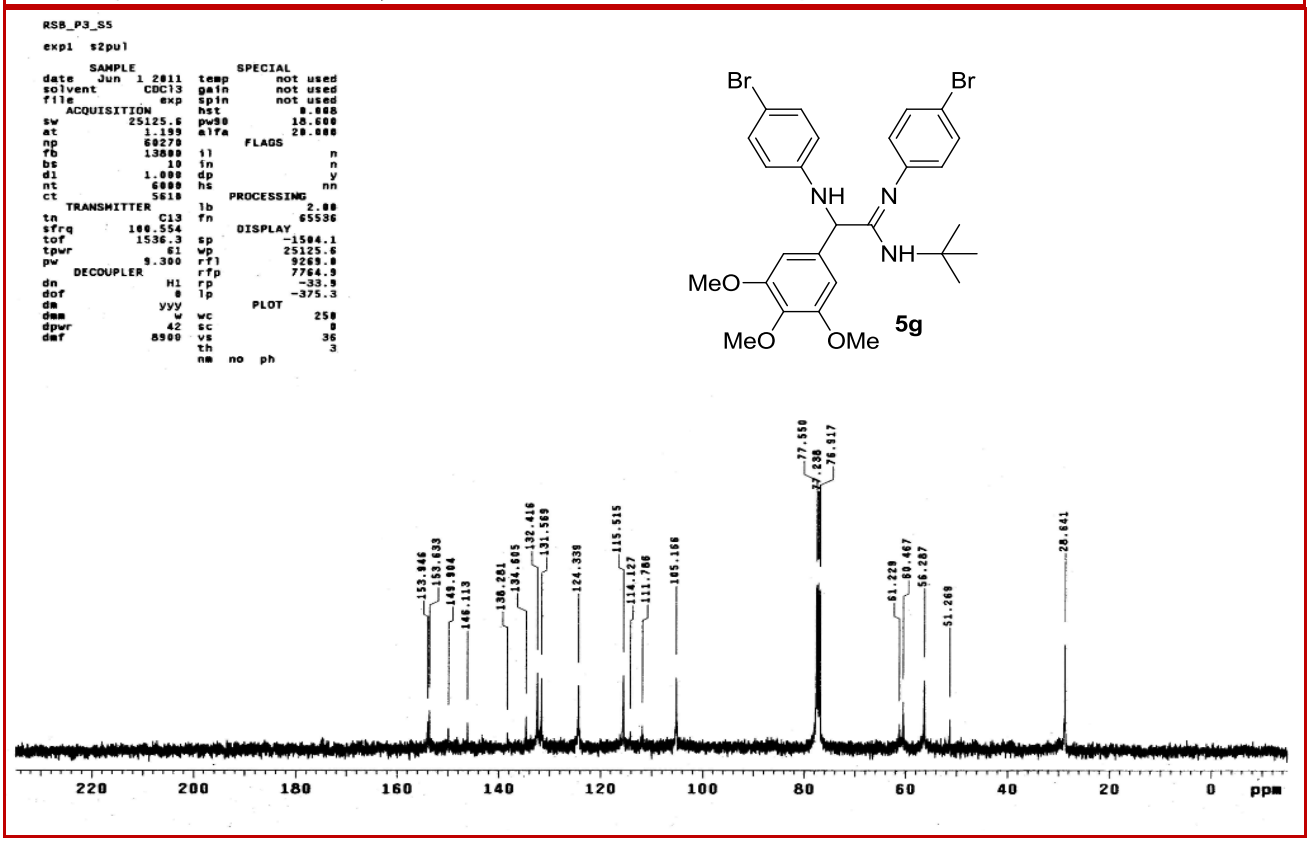
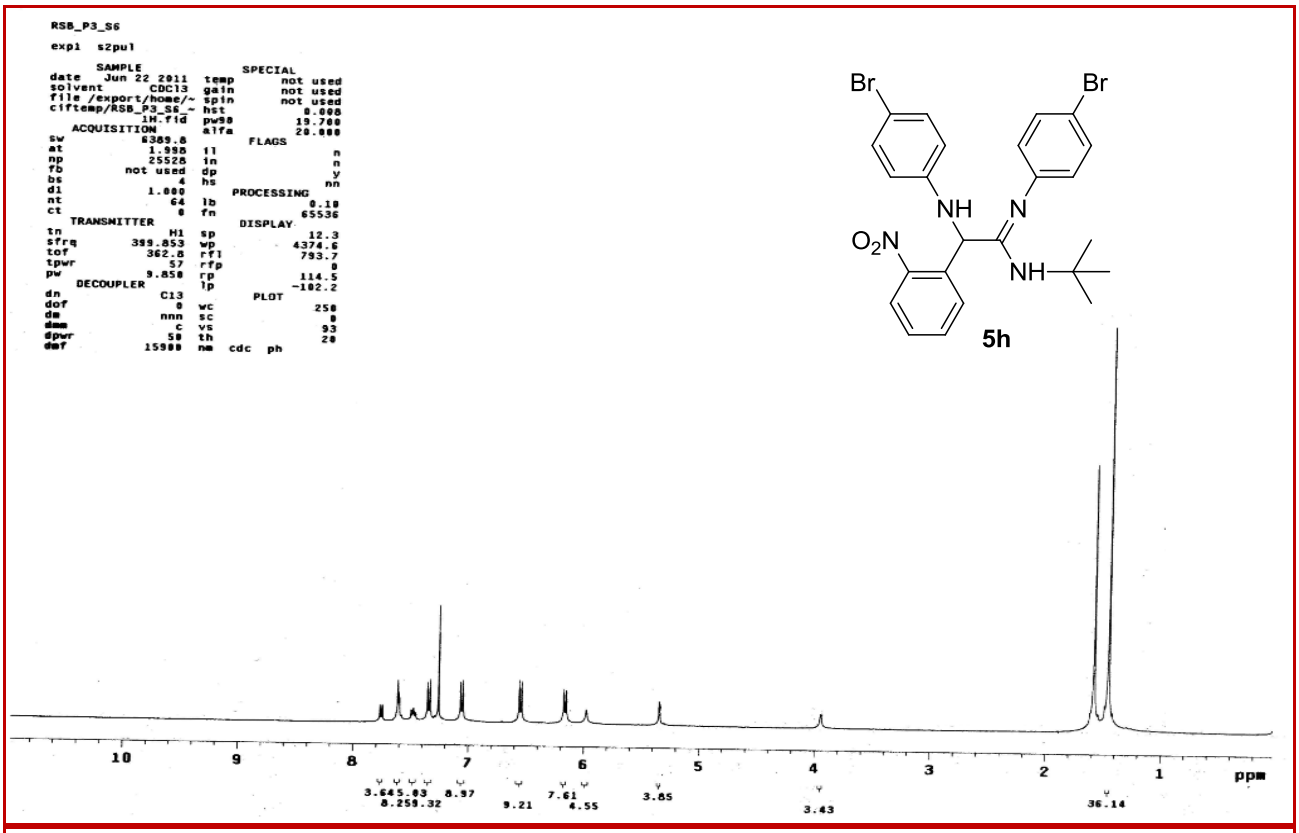
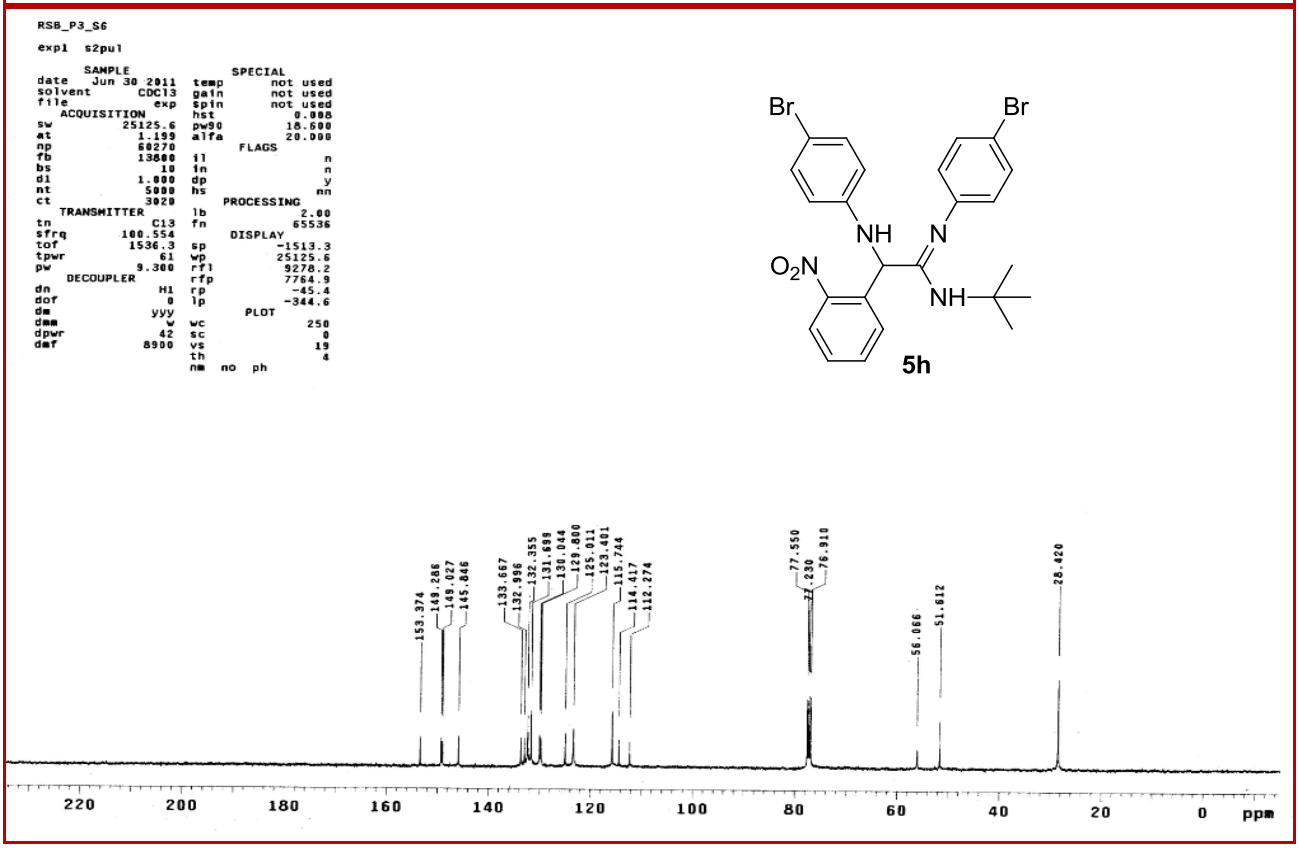
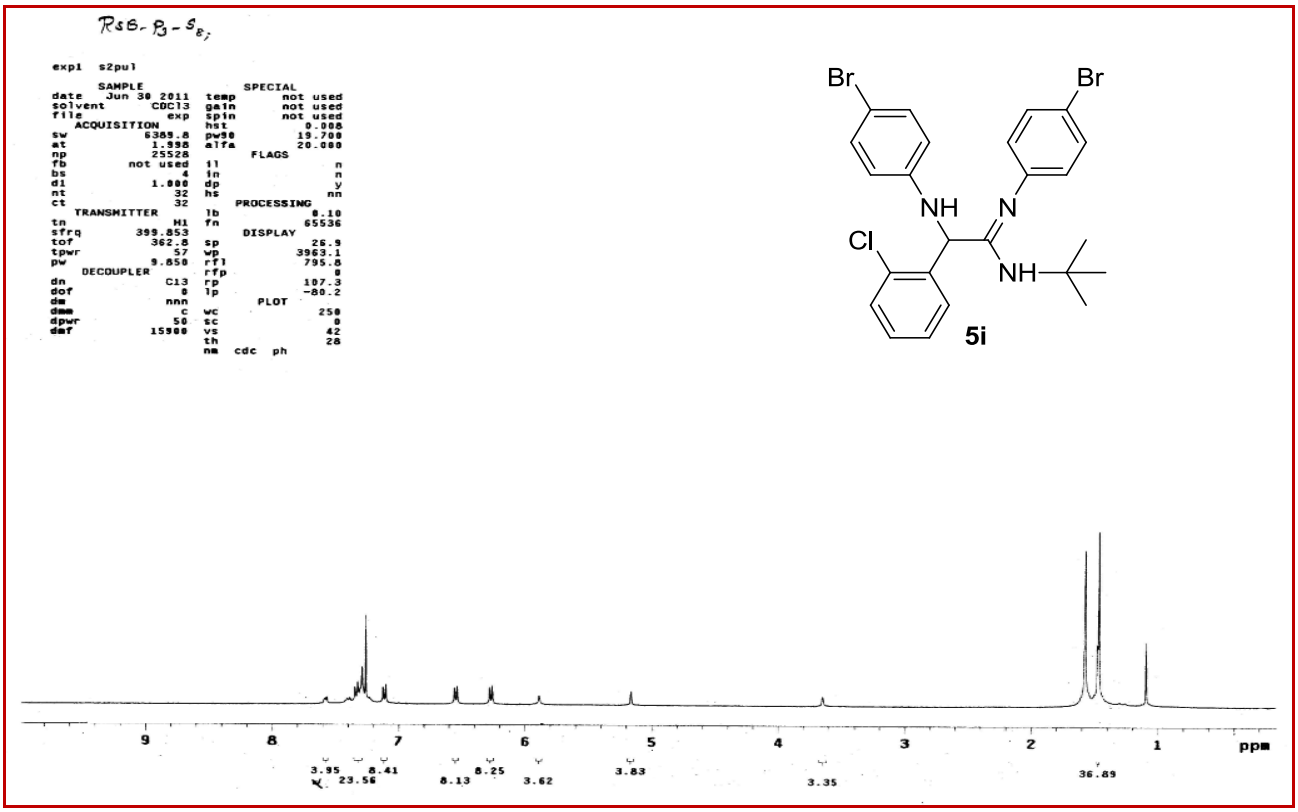
80
60
40
20
ppm

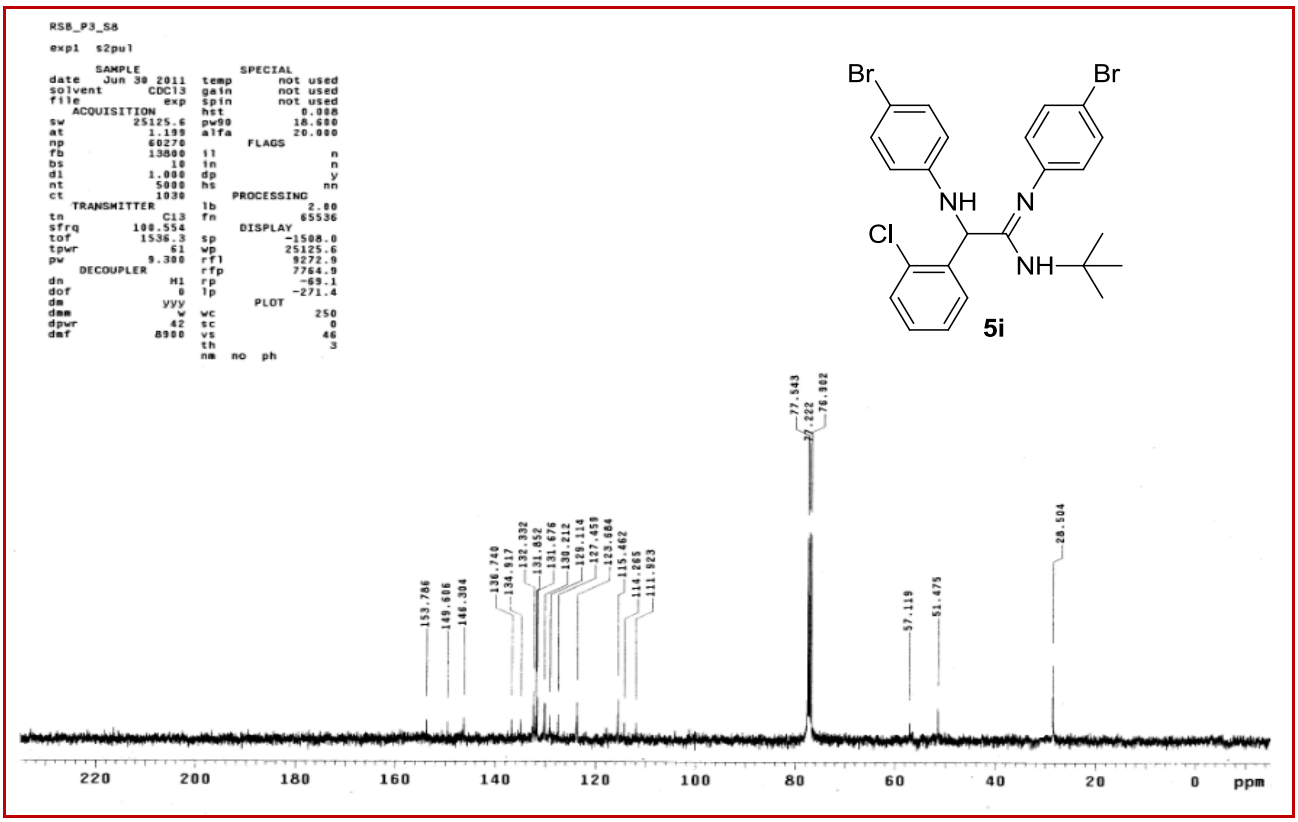


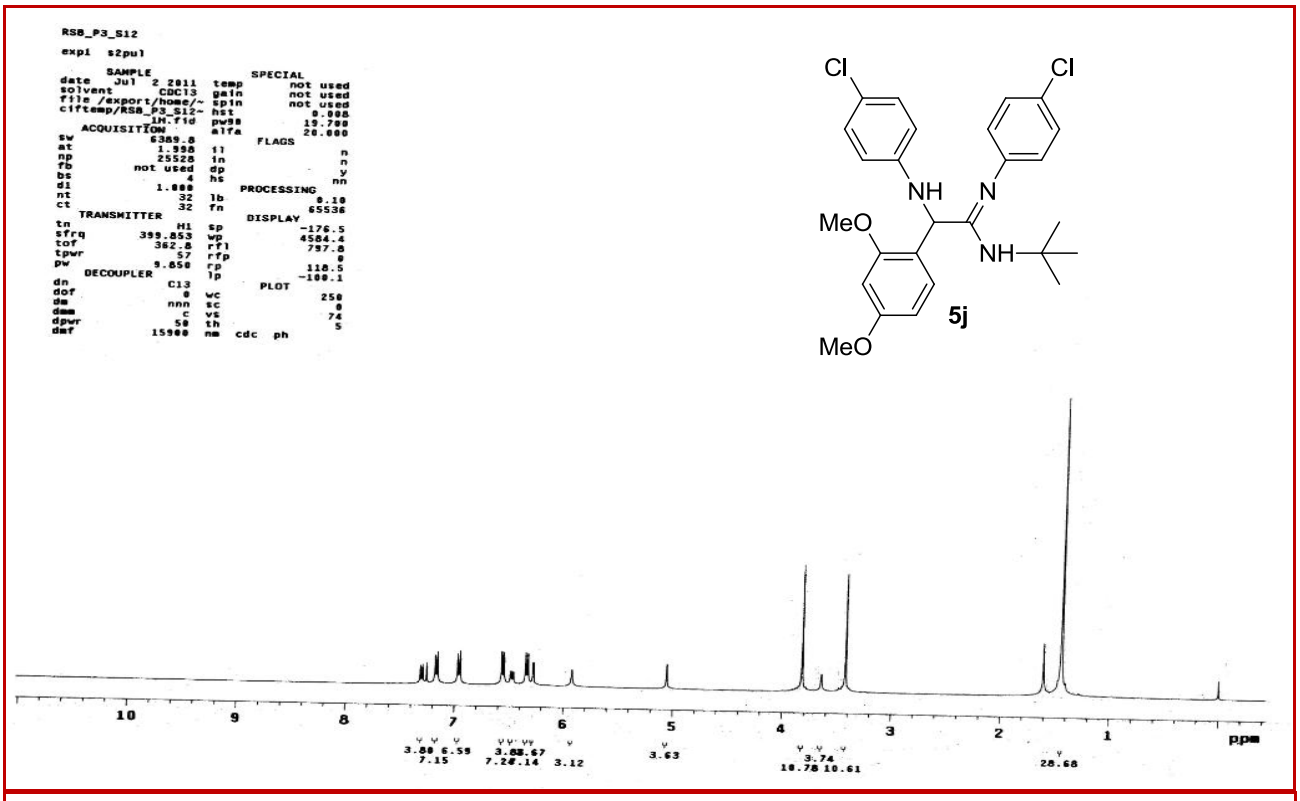








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date Jul 2 2011
solvent CDCl3
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ciftemp/RS8_P3_S12~
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ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -176.5
wp 4584.4
rfl 797.8
rfp 0
rp 118.5
lp -100.1
PLOT
wc 250
sc 0
vs 74
th 5
nm cdc ph
Cl
Cl
NH
N
MeO
NH
MeO
5j
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3.88.67
7.26.14
3.12
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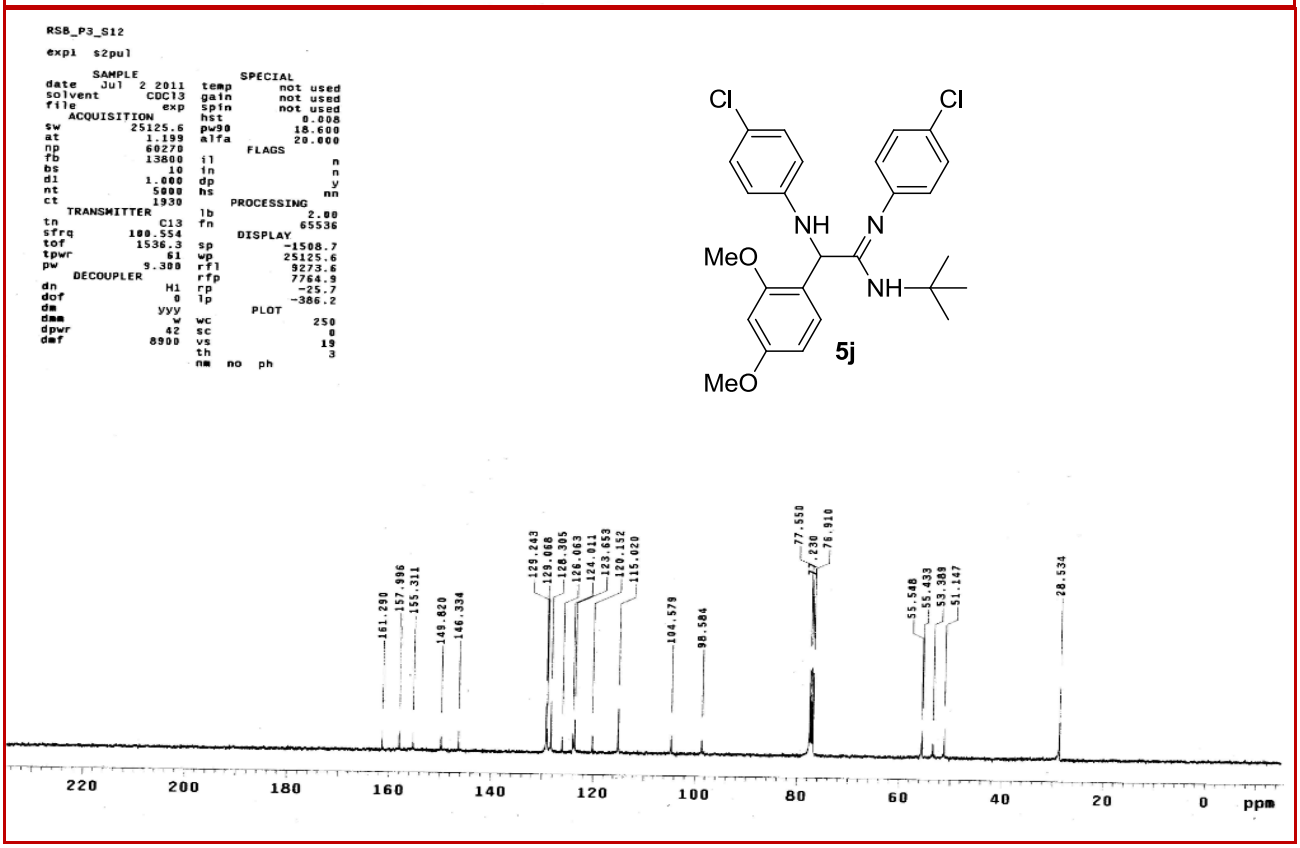
10.78 10.61
28.68
ppm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date Jul 2 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 1930
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -1508.7
wp 25125.6
rfl 9273.6
rfp 7764.9
rp -25.7
lp -386.2
PLOT
wc 250
sc 0
vs 19
th 3
nm no ph
Cl
Cl
NH
N
MeO
NH
MeO
5j
161.290
157.996
155.311
149.820
146.334
129.243
129.060
128.305
126.063
124.011
123.653
120.152
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98.584
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77.230
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55.433
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51.147
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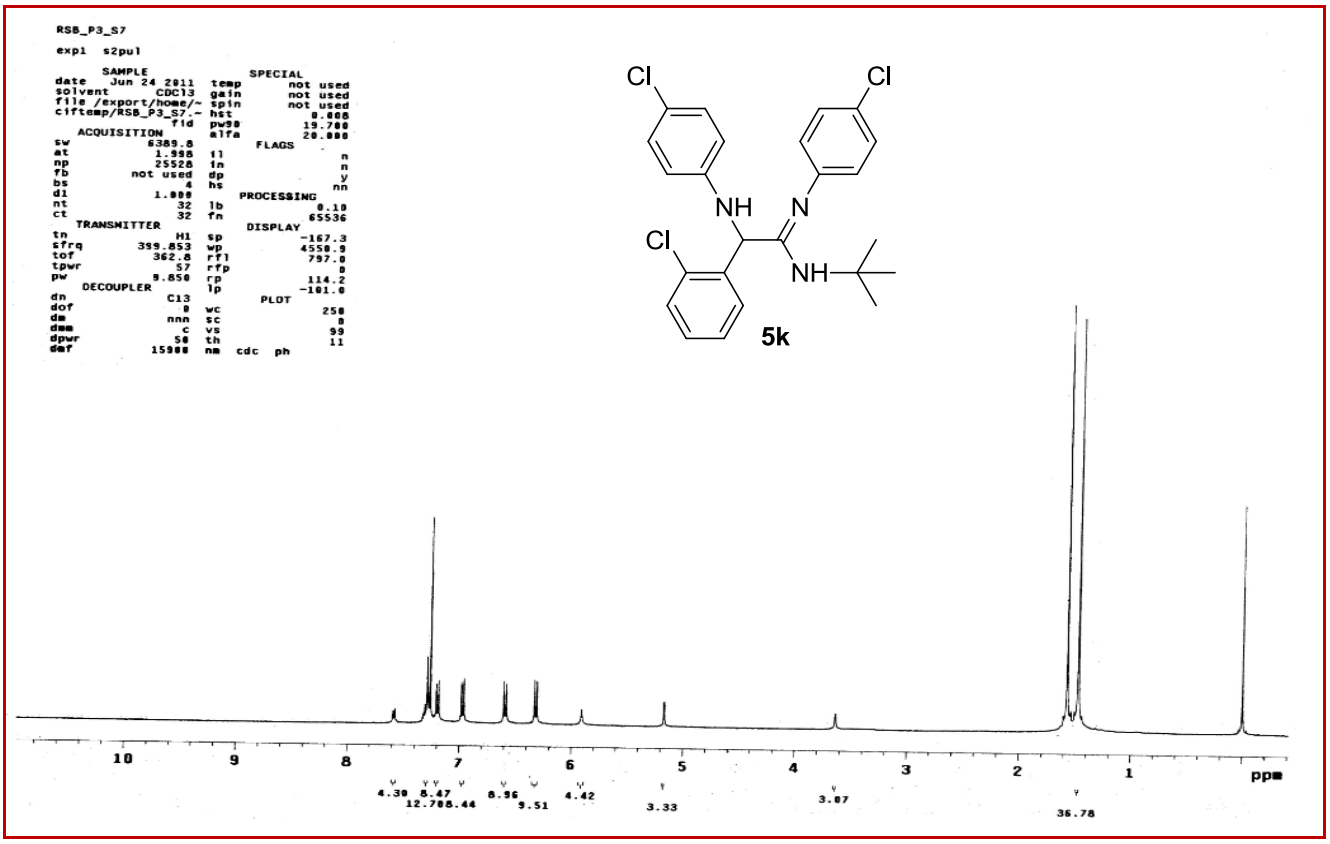
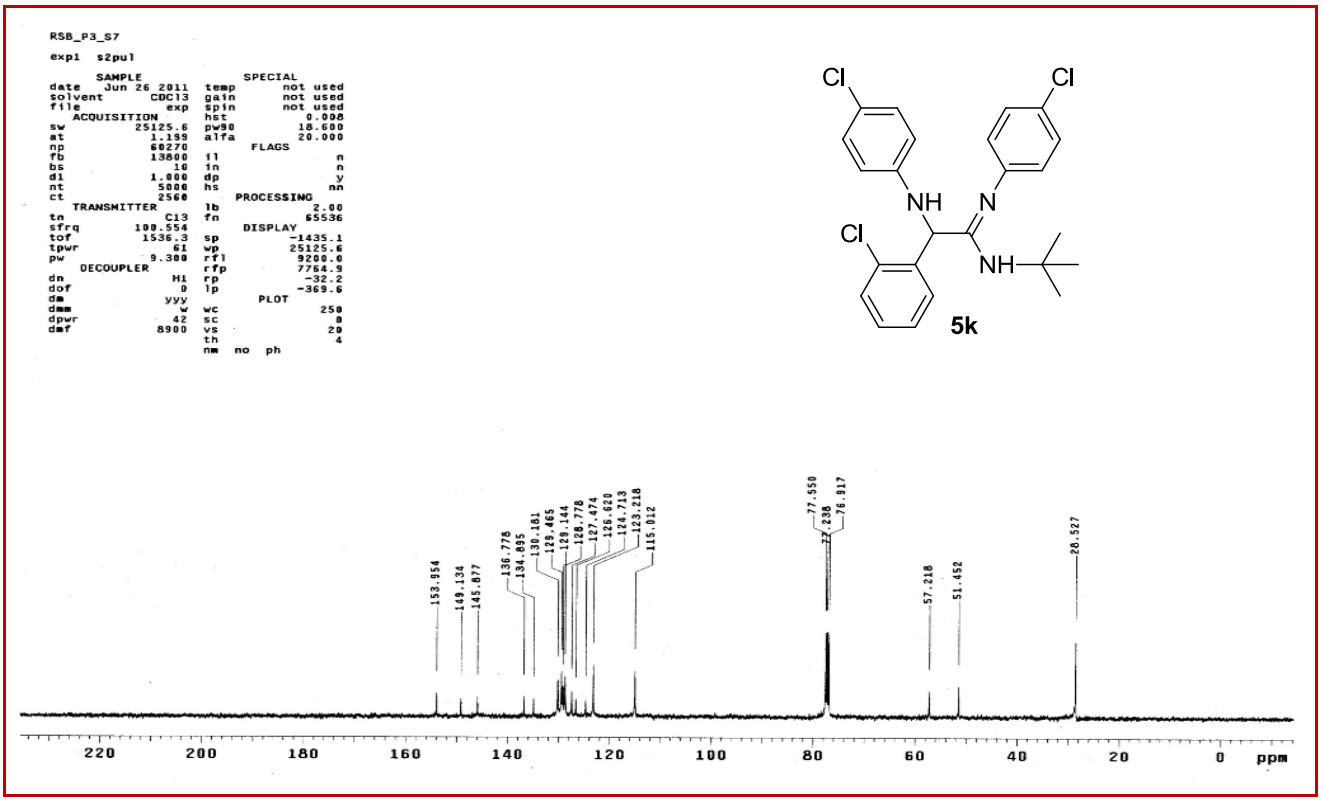
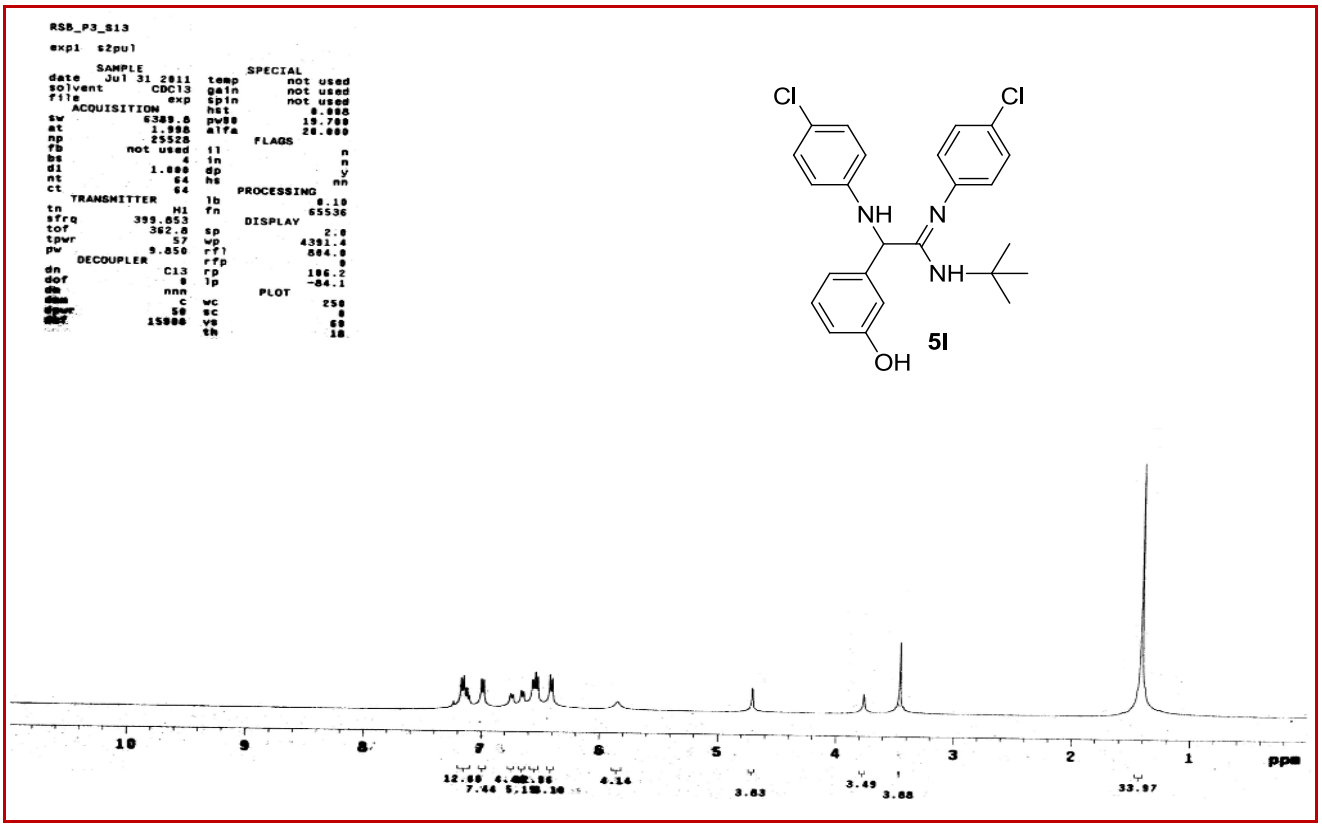
ppm



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date Jul 31 2011
solvent CDCl3
file exp
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at 1.998
np 25528
fb not used
bs 4
dl 1.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 399.853
tof 382.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.000
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp 2.8
wp 4391.4
rfl 864.8
rfp 0
rp 186.2
lp -84.1
PLOT
wc 250
sc 0
vs 68
th 18
Cl
Cl
NH
N
NH
OH
5l
10
9
8
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6
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3
2
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ppm
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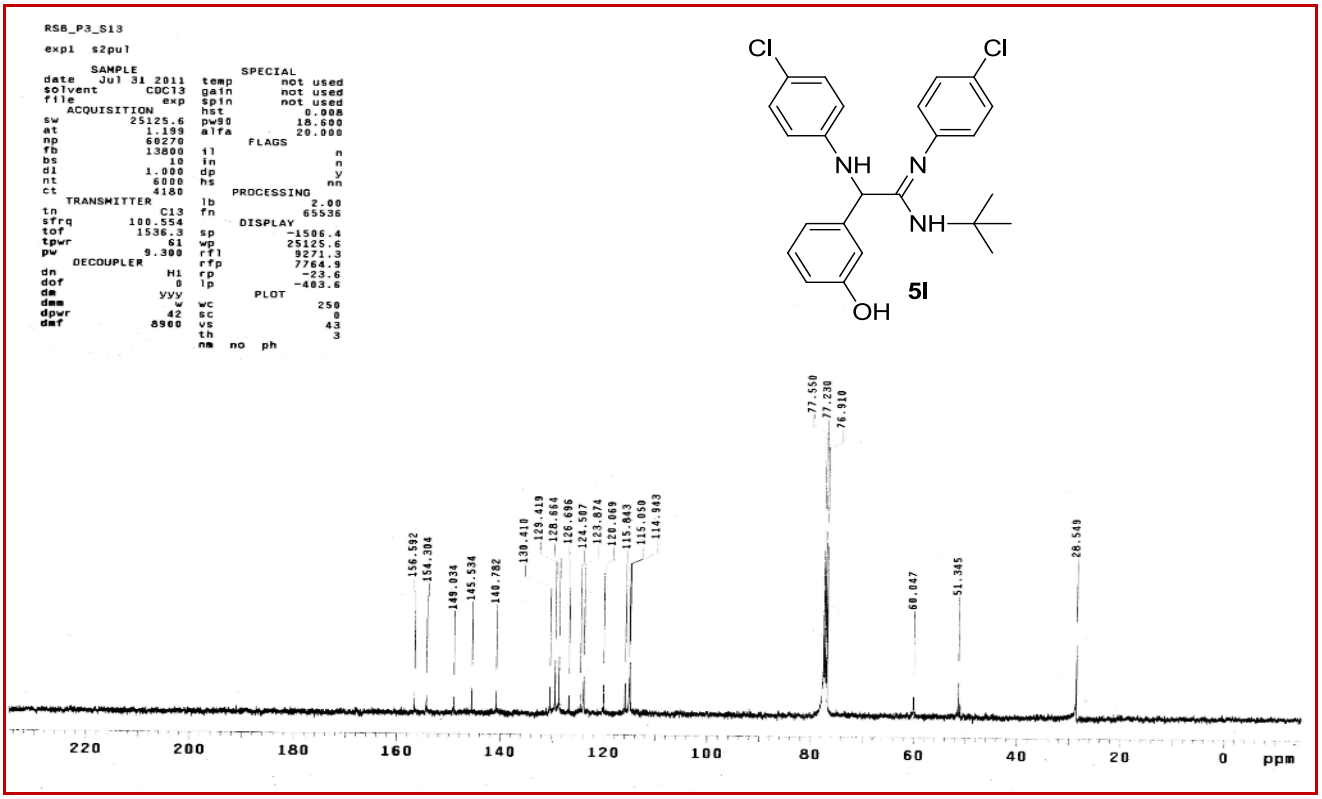
3.88
33.97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date Jul 31 2011
solvent CDCl3
file exp
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at 1.199
np 60270
fb 13800
bs 10
dl 1.000
nt 6000
ct 4180
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -1506.4
wp 25125.6
rfl 9271.3
rfp 7764.9
rp -23.6
lp -403.6
PLOT
wc 250
sc 0
vs 43
th 3
nm no ph
Cl
Cl
NH
N
NH
OH
5l
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77.230
76.910
156.592
154.304
149.034
145.534
140.782
130.410
129.419
128.664
126.696
124.587
123.874
120.069
115.843
115.050
114.943
69.047
51.345
28.549
220
200
180
160
140
120
100
80
60
40
20
0
ppm