Electronic Supplementary Material (ESI) for RSC Advances

Supporting information for

Synthesis of phenothiazines from cyclohexanones and 2-aminobenzenethiols under transition-metal-free conditions

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General information:

All reactions were carried out under an atmosphere of oxygen unless otherwise noted. Flash chromatography was performed on neutral aluminum oxide (100-200 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument internally referenced to tetramethylsilane (TMS) or acetone signals. Mass spectra was measured on an Agilent 5975 GC-MS instrument (EI). The new compounds were characterized by ^{1}H NMR, ^{13}C NMR, MS and HRMS. The structure of known compounds were further corroborated by comparing their ^{1}H NMR, ^{13}C NMR data and MS data with those of literature. All reagents were obtained from commercial suppliers and used without further purification.

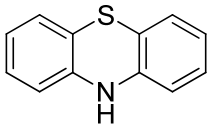
General procedure (for 0.2 mmol scale):

Potassium iodide (3.4 mg, 0.02 mmol), (benzylsulfonyl)benzene (**A-1**, 4.6 mg, 0.02 mmol) were added to a 10 mL reaction tube. The sealed tube was purged with oxygen for three times and was added 2-aminobenzenethiol (**1a**, 21.4 μL, 0.2 mmol), cyclohexanone (**2a**, 31 μL, 0.3 mmol) and chlorobenzene (0.8 mL) by syringe. The reaction vessel was stirred at 140 ^{o}C for 24 h. After cooling to room temperature, the volatiles were removed under reduced pressure. The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to afford the desired product **3a** as pale yellow solid (28.3 mg, 71% yield).

General procedure (for 0.5 mmol scale):

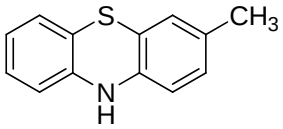
Potassium iodide (8.3 mg, 0.05 mmol), (benzylsulfonyl)benzene (**A-1**, 11.5 mg, 0.05 mmol) were added to a 10 mL reaction tube. The sealed tube was purged with oxygen for three times and was added 2-aminobenzenethiol (**1a**, 53.3 μL, 0.5 mmol), cyclohexanone (**2a**, 77.5 μL, 0.75 mmol) and chlorobenzene (2.0 mL) by syringe. The reaction vessel was stirred at 140 ^{o}C. After 4 h, 8 h and 12 h, the reaction tube was flushed with oxygen and the reaction mixture was stirred for 24 h. After cooling to room temperature, the volatiles were removed under reduced pressure. The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give the desired product **3a** as pale yellow solid (70 mg, 70% yield).

10*H*-Phenothiazine (3a, CAS: 92-84-2)[1]



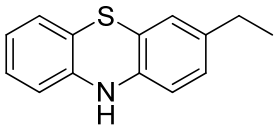
^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.82 (brs, 1H), 7.00-6.93 (m, 4H), 6.79 (t, J = 7.0 Hz, 2H), 6.71 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.6, 128.5, 127.4, 123.1, 118.6, 115.6; MS (EI) *m/z* (%) 199 (100), 167, 154, 140, 77.

3-Methyl-10*H*-phenothiazine (3b, CAS: 3939-47-7**)**[2]



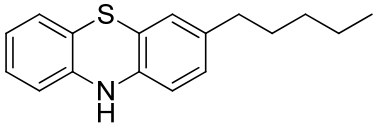
The reaction was conducted with 2-aminobenzenethiol (**1a**, 21.3 μL, 0.2 mmol) and 4-methylcyclohexanone (**2b**, 36.8 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3b** as yellow solid (35.4 mg, 83%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.74 (brs, 1H), 6.97-6.93 (m, 2H), 6.77-6.60 (m, 5H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.8, 143.8, 141.0, 141.0, 132.3, 128.9, 128.3, 127.6, 127.3, 122.7, 118.3, 115.4, 20.5; MS (EI) *m/z* (%) 213 (100), 198, 180, 167, 90.

3-Ethyl-10*H*-phenothiazine (3c, CAS: 54027-87-1)[3]



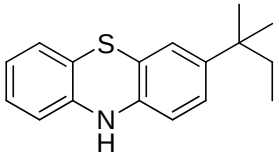
The reaction was conducted with 2-aminobenzenethiol (**1a**, 21.3 μL, 0.2 mmol) and 4-ethylcyclohexanone (**2c**, 42.3 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 50:1) to give **3c** as yellow solid (34.5 mg, 76%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.76 (brs, 1H), 6.95 (t, J = 5.8 Hz, 2H), 6.84-6.63 (m, 5H), 2.47 (m, 2H), 1.14 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.8, 141.2, 139.1, 128.3, 127.8, 127.3, 126.6, 122.7, 118.5, 118.4, 115.5, 115.4, 28.6, 16.3; MS (EI) *m/z* (%) 227, 212 (100), 180, 167, 106.

3-Pentyl-10*H*-phenothiazine (3d, CAS: 93730-07-5)[3]



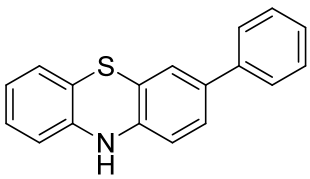
The reaction was conducted with 2-aminobenzenethiol (**1a**, 21.3 μL, 0.2 mmol) and 4-pentylcyclohexanone (**2d**, 57.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 50:1) to give **3d** as yellow solid (37.8 mg, 70%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.76 (brs, 1H), 6.98-6.93 (m, 2H), 6.84-6.63 (m, 5H), 2.45 (m, 2H), 1.54 (m, 2H), 1.30 (m, 4H), 0.88 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.8, 141.2, 137.6, 128.3, 127.3, 127.1, 127.0, 122.7, 118.5, 118.3, 115.9, 115.4, 35.6, 32.2, 32.1, 23.3, 14.5; MS (EI) *m/z* (%) 269, 212 (100), 180, 167, 152.

3-*tert*-Pentyl-10*H*-phenothiazine (3e)



The reaction was conducted with 2-aminobenzenethiol (**1a**, 21.3 μL, 0.2 mmol) and 4-(tert-pentyl)cyclohexanone (**2e**, 54.9 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3e** as yellow solid (43.0 mg, 80%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.77 (brs, 1H), 6.97-6.93 (m, 4H), 6.78-6.66 (m, 3H), 1.59 (m, 2H), 1.21 (s, 6H), 0.66 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 144.2, 143.8, 140.9, 128.3, 127.3, 126.0, 124.8, 122.7, 118.6, 118.0, 115.4, 115.1, 38.1, 37.4, 28.9, 9.6; HRMS (ESI, *m/z*): calcd for $C_{17}H_{19}NS$ $[M]^+$ 269.1233, found 269.1232.

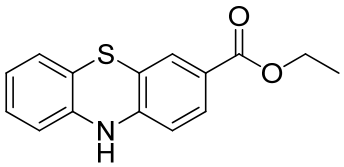
3-Phenyl-10*H*-phenothiazine (3f, CAS: 4018-68-2)[4]



The reaction was conducted with 2-aminobenzenethiol (**1a**, 21.3 μL, 0.2 mmol) and 4-phenylcyclohexanone (**2f**, 52.2 mg, 0.3 mmol). The residue was purified by column

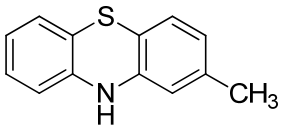
chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3f** as yellow solid (44.6 mg, 81%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.97 (brs, 1H), 7.58 (d, J = 6.4 Hz, 2H), 7.41-7.25 (m, 5H), 6.99 (t, J = 8.2 Hz, 2H), 6.81-6.73 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.2, 142.8, 141.1, 136.1, 129.8, 128.5, 127.9, 127.8, 127.4, 127.1, 125.6, 123.2, 119.2, 118.3, 115.9, 115.7; MS (EI) *m/z* (%) 275 (100), 243, 152, 114, 77.

Ethyl 10*H*-phenothiazine-3-carboxylate (3g)



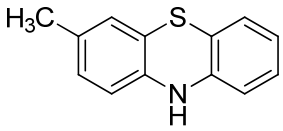
The reaction was conducted with 2-aminobenzenethiol (**1a**, 21.3 μL, 0.2 mmol) and ethyl 4-oxocyclohexanecarboxylate (**2g**, 47.8 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3g** as yellow solid (39.0 mg, 72%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 8.30 (brs, 1H), 7.62 (d, J = 7.6 Hz, 1H), 7.52 (s, 1H), 7.01 (d, J = 6.8 Hz, 1H), 6.95 (d, J = 6.8 Hz, 1H), 6.85 (d, J = 6.8 Hz, 1H), 6.73 (d, J = 6.8 Hz, 2H), 4.27 (m, 2H), 1.33 (t, J = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 166.1, 147.4, 147.4, 141.8, 130.4, 128.7, 128.4, 127.4, 125.1, 124.0, 117.9, 116.0, 114.8, 61.3, 14.8; HRMS (ESI, *m/z*): calcd for $C_{15}H_{13}NO_2S$ $[M]^+$ 271.0662, found 271.0660.

2-Methyl-10*H*-phenothiazine (3j, CAS: 5828-51-3)[1]



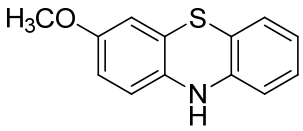
The reaction was conducted with 2-aminobenzenethiol (**1a,** 21.3 μL, 0.2 mmol) and 3-methylcyclohex-2-enone (**2j**, 34.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3j** as yellow solid (22.2 mg, 52%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.76 (brs, 1H), 6.98-6.93 (m, 2H), 6.83-6.78 (m, 2H), 6.71 (d, J = 7.6 Hz, 1H), 6.63 (d, J = 6.8 Hz, 1H), 6.55 (s, 1H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.6, 143.4, 138.3, 128.3, 127.3, 127.1, 123.8, 122.9, 118.9, 116.4, 115.6, 115.2, 21.2; MS (EI) *m/z* (%) 213 (100), 198, 180, 167, 152.

3-Methyl-10*H*-phenothiazine (3k, CAS: 3939-47-7**)**[2]



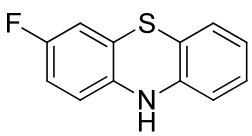
The reaction was conducted with 2-amino-5-methylbenzenethiol (**1b**, 27.8 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3k** as yellow solid (26.4 mg, 62%). This product is same as **3b**. ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.74 (brs, 1H), 6.97-6.93 (m, 2H), 6.77-6.60 (m, 5H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.8, 143.8, 141.0, 141.0, 132.3, 128.9, 128.3, 127.6, 127.3, 122.7, 118.3, 115.4, 20.5; MS (EI) *m/z* (%) 213 (100), 198, 180, 167, 90.

3-Methoxy-10*H*-phenothiazine (3l, CAS: 1771-19-3**)**[1]



The reaction was conducted with 2-amino-5-methoxybenzenethiol (**1c**, 31.1 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 10:1) to give **3l** as white solid (26.6 mg, 58%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.65 (brs, 1H), 6.98-6.94 (m, 2H), 6.78-6.59 (m, 5H), 3.71 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 156.6, 144.2, 137.0, 128.5, 127.3, 122.6, 119.7, 118.0, 116.2, 115.4, 114.0, 112.8, 56.1; MS (EI) *m/z* (%) 229 (100), 214, 186, 154, 128.

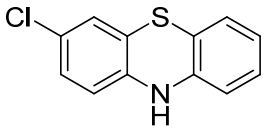
3-Fluoro-10*H*-phenothiazine (3m, CAS: 397-59-1**)**[5]



The reaction was conducted with 2-amino-5-fluorobenzenethiol (**1d**, 28.6 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 50:1) to give **3m** as white solid (33.0 mg, 76%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.90 (brs, 1H), 7.01-6.97 (m, 2H), 6.81-6.73 (m, 5H);

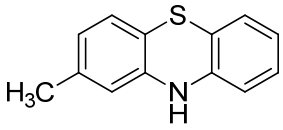
^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 159.4 (d, J = 237.1 Hz), 143.6, 140.0, 128.8, 127.4, 123.2, 120.5 (d, J = 6.8 Hz), 117.5, 116.1 (d, J = 8.0 Hz),115.6, 114.6 (d, J = 22.6 Hz), 114.0 (d, J = 25.1 Hz); MS (EI) *m/z* (%) 217 (100), 185, 172, 157, 91.

3-Chloro-10*H*-phenothiazine (3n, CAS: 1207-99-4**)**[4]



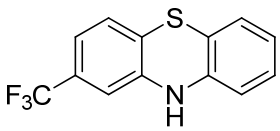
The reaction was conducted with 2-amino-5-chlorobenzenethiol (**1e**, 31.9 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3n** as yellow solid (35.0 mg, 75%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.99 (brs, 1H), 7.01-6.95 (m, 4H), 6.82 (m, 1H), 6.71 (d, J = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.0, 142.5, 128.8, 128.2, 127.4, 127.0, 126.6, 123.4, 120.7, 117.6, 116.5, 115.8; MS (EI) *m/z* (%) 233 (100), 198, 188, 166, 154.

2-Methyl-10*H*-phenothiazine (3o, CAS: 5828-51-3**)**[1]



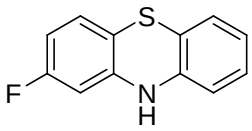
The reaction was conducted with 2-amino-4-methylbenzenethiol (**1f**, 27.8 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3o** as yellow solid (27.7 mg, 65%). This product is same as **3j**. ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.76 (brs, 1H), 6.98-6.93 (m, 2H), 6.83-6.78 (m, 2H), 6.71 (d, J = 7.6 Hz, 1H), 6.63 (d, J = 6.8 Hz, 1H), 6.55 (s, 1H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 143.6, 143.4, 138.3, 128.3, 127.3, 127.1, 123.8, 122.9, 118.9, 116.4, 115.6, 115.2, 21.2; MS (EI) *m/z* (%) 213 (100), 198, 180, 167, 152.

2-(Trifluoromethyl)-10*H*-phenothiazine (3p, CAS: 92-30-8**)**[1]



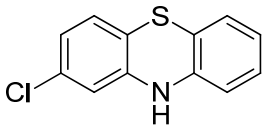
The reaction was conducted with 2-amino-4-(trifluoromethyl)benzenethiol hydrochloride (**1g**, 45.9 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether) to give **3p** as yellow solid (32.0 mg, 60%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 8.11 (brs, 1H), 7.13-6.96 (m, 5H), 6.85 (m, 1H), 6.72 (d, J = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 144.0, 142.4, 130.2 (q, J = 31.9 Hz), 129.0, 127.8, 127.4, 125.3 (q, J = 271.3 Hz), 124.0 (q, J = 2.0 Hz),123.8, 119.3 (q, J = 4.0 Hz), 117.5, 115.9, 115.5 (q, J = 3.8 Hz); MS (EI) *m/z* (%) 267 (100), 247, 235, 222, 91.

2-Fluoro-10*H*-phenothiazine (3q, CAS: 397-58-0)[1]



The reaction was conducted with 2-amino-4-fluorobenzenethiol (**1h**, 28.6 mg, 0.2 mmol), and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 50:1) to give **3q** as yellow solid (25.2 mg, 58%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 8.07 (brs, 1H), 7.02-6.96 (m, 3H), 6.83 (m, 1H), 6.72 (d, J = 6.0 Hz, 1H), 6.59-6.51 (m, 2H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 163.8 (d, J = 240.5 Hz), 145.4 (d, J = 10.5 Hz), 142.6, 128.6, 128.4 (d, J = 9.7 Hz), 127.4, 123.6, 118.6, 115.8, 113.8, 109.3 (d, J = 22.5 Hz), 102.8 (d, J = 26.1 Hz); MS (EI) *m/z* (%) 217 (100), 185, 172, 157, 91.

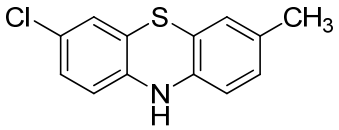
2-Chloro-10*H*-phenothiazine (3r, CAS: 92-39-7)[1]



The reaction was conducted with 2-amino-4-chlorobenzenethiol (**1i**, 31.9 mg, 0.2 mmol) and cyclohexanone (**2a**, 31.0 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3r** as yellow solid (32.7 mg,

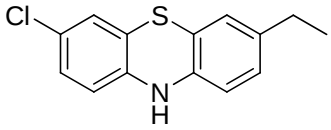
70%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 8.02 (brs, 1H), 7.02-6.95 (m, 3H), 6.83-6.70 (m, 4H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 144.9, 142.6, 133.5, 128.7, 128.4, 127.4, 123.6, 122.6, 118.2, 117.6, 115.9, 115.2; MS (EI) *m/z* (%) 233 (100), 198, 188, 166, 154.

3-Chloro-7-methyl-10*H*-phenothiazine (3t, CAS: 35565-23-2)[6]



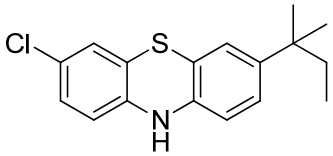
The reaction was conducted with 2-amino-5-chlorobenzenethiol (**1e**, 31.9 mg, 0.2 mmol) and 4-methylcyclohexanone (**2b**, 36.8 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3t** as yellow solid (35.0 mg, 71%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.86 (brs, 1H), 6.99-6.97 (m, 2H), 6.83-6.78 (m, 2H), 6.68 (d, J = 8.0 Hz, 1H), 6.61 (d, J = 7.6 Hz, 1H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 142.8, 142.8, 140.6, 132.9, 129.3, 128.1, 127.7, 126.6, 120.7, 117.5, 116.3, 115.6, 20.5; MS (EI) *m/z* (%) 247 (100), 232, 212, 178, 168.

3-Chloro-7-ethyl-10*H*-phenothiazine (3u, CAS: 106837-73-4)[7]



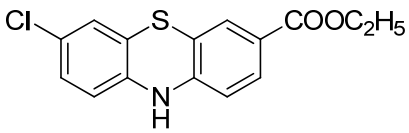
The reaction was conducted with 2-amino-5-chlorobenzenethiol (**1e**, 31.9 mg, 0.2 mmol) and 4-ethylcyclohexanone (**2c**, 42.3 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3u** as yellow solid (39.7 mg, 76%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.89 (brs, 1H), 6.97 (s, 2H), 6.87-6.82 (m, 2H), 6.70-6.63 (m, 2H), 2.48 (m, 2H), 1.14 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 142.8, 140.8, 139.6, 128.1, 128.1, 126.7, 126.6, 120.7, 117.5, 117.5, 116.4, 115.7, 28.6, 16.2; MS (EI) *m/z* (%) 261 (100), 246, 210, 167, 123.

3-Chloro-7-*tert*-pentyl-10*H*-phenothiazine (3v)



The reaction was conducted with 2-amino-5-chlorobenzenethiol (**1e**, 31.9 mg, 0.2 mmol) and 4-(tert-pentyl)cyclohexanone (**2e**, 54.9 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3v** as yellow solid (49.0 mg, 81%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 7.91 (brs, 1H), 6.98-6.93 (m, 4H), 6.71-6.65 (m, 2H), 1.59 (m, 2H), 1.21 (s, 6H), 0.66 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 144.7, 142.8, 140.4, 128.1, 126.7, 126.6, 126.4, 124.9, 120.8, 117.2, 116.4, 115.4, 38.2, 37.4, 28.9, 9.6; HRMS (ESI, *m/z*): calcd for $C_{17}H_{18}ClNS$ $[M]^+$ 303.0843, found 303.0843

Ethyl 7-chloro-10*H*-phenothiazine-3-carboxylate (3w)



The reaction was conducted with 2-amino-5-chlorobenzenethiol (**1e**, 31.9 mg, 0.2 mmol) and ethyl 4-oxocyclohexanecarboxylate (**2g**, 47.8 μL, 0.3 mmol). The residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/EtOAc = 20:1) to give **3w** as yellow solid (52.0 mg, 85%). ^{1}H NMR (400 MHz, CD_3COCD_3, ppm) δ 8.41 (brs, 1H), 7.63 (d, J = 7.2 Hz, 1H), 7.53 (s, 1H), 7.03-7.01 (m, 2H), 6.73 (m, 2H), 4.27 (m, 2H), 1.33 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3, ppm) δ 165.9, 146.9, 140.8, 130.7, 128.4, 128.0, 128.0, 126.7, 125.5, 120.2, 117.4, 117.0, 115.1, 61.3, 14.8; HRMS (ESI, *m/z*): calcd for $C_{15}H_{13}ClNO_2S$ $[M+H]^+$ 306.0350, found 306.0350

References

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[3] Takada, A.; Nishimura, H. *Chem. Pharm. Bull.* **1962**, *10*, 1.

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[5] Craig, C.; Warburton. *Aust. J Chem.* **1956**, *9*, 294.

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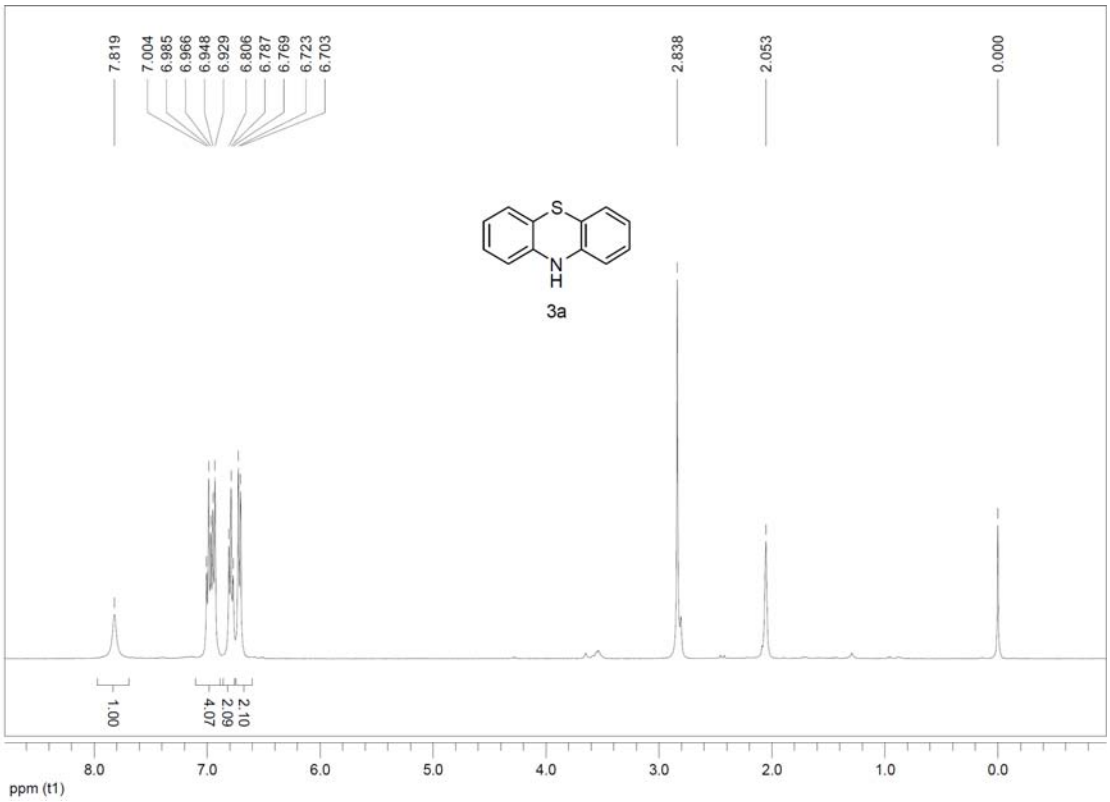
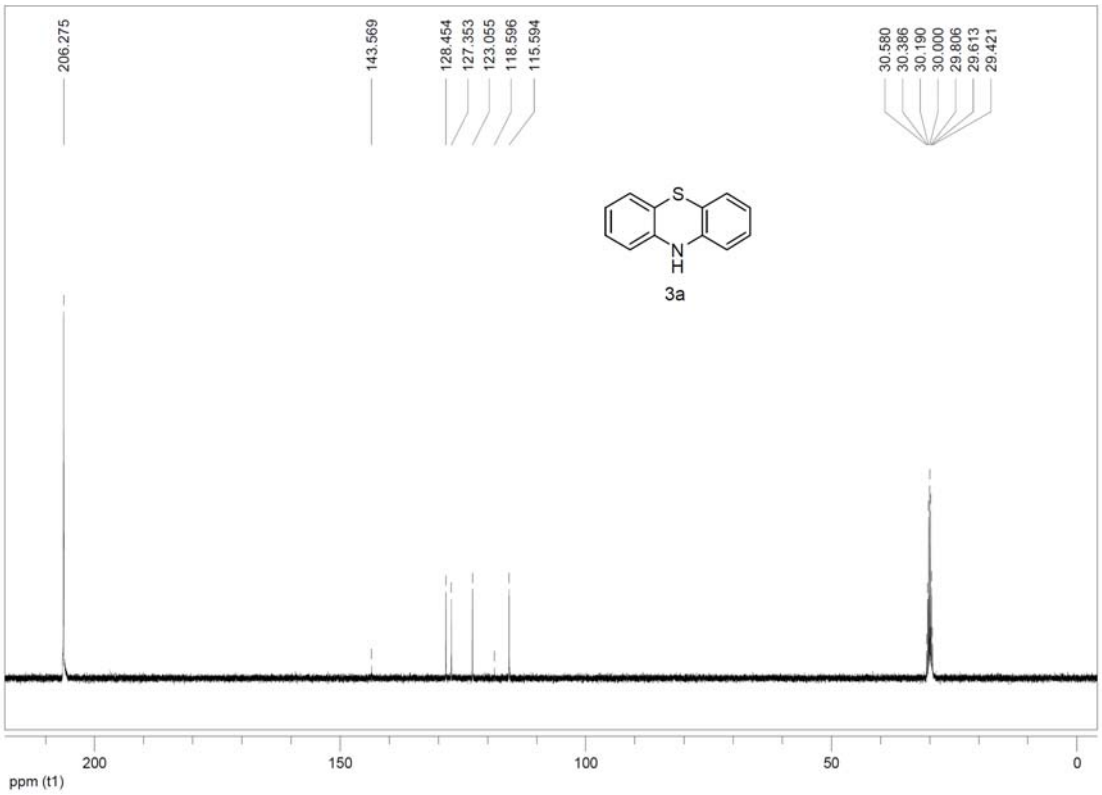
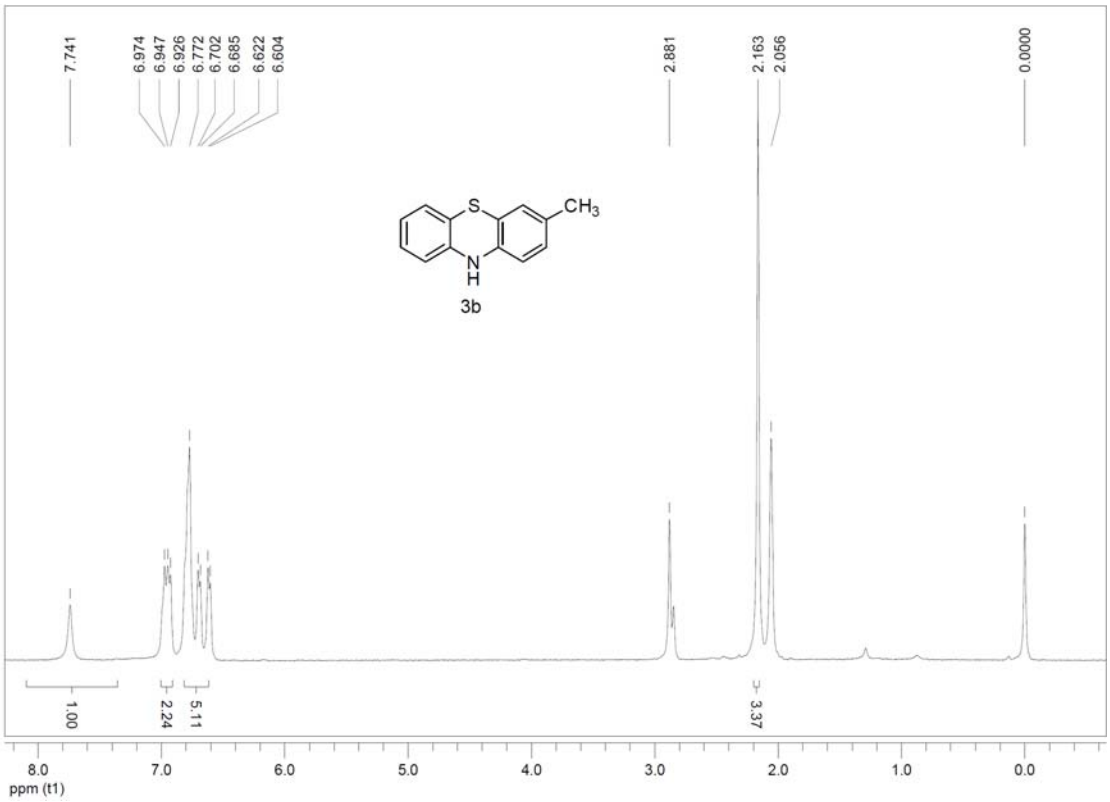
^{1}H NMR and ^{13}C NMR spectra



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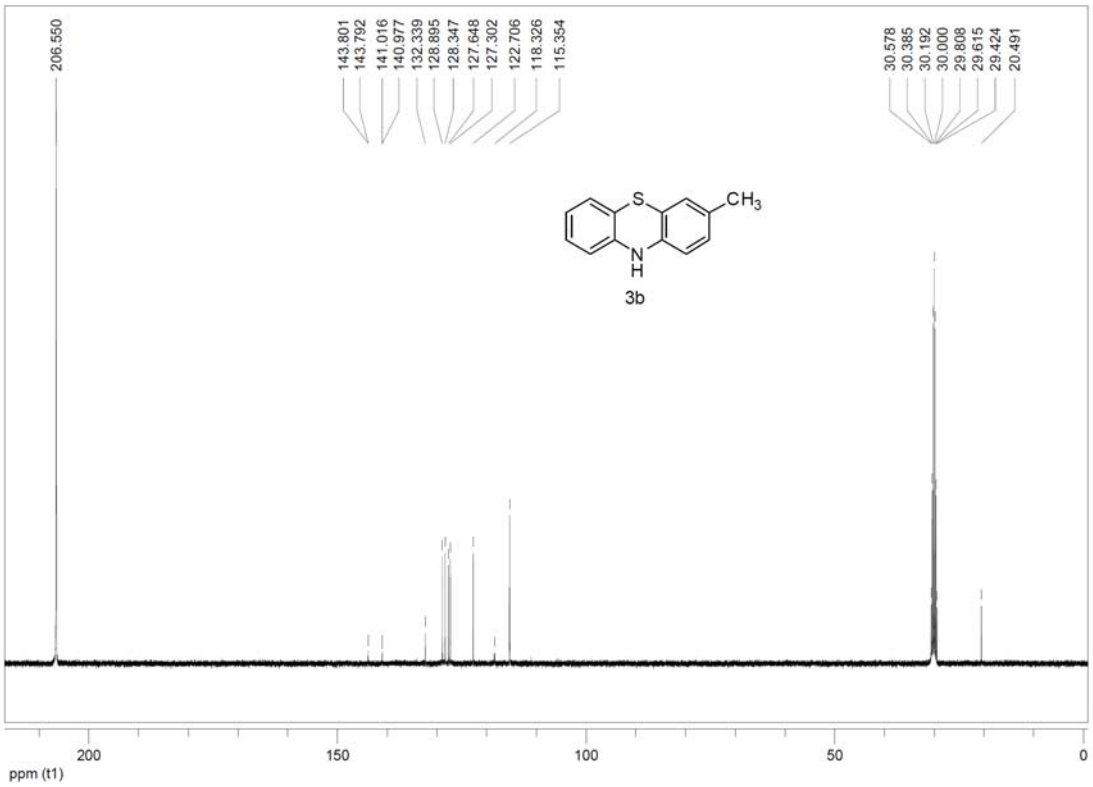
6.947
6.926
6.772
6.702
6.685
6.622
6.604
2.881
2.163
2.056
0.0000
S
CH3
N
H
3b
1.00
2.24
5.11
3.37
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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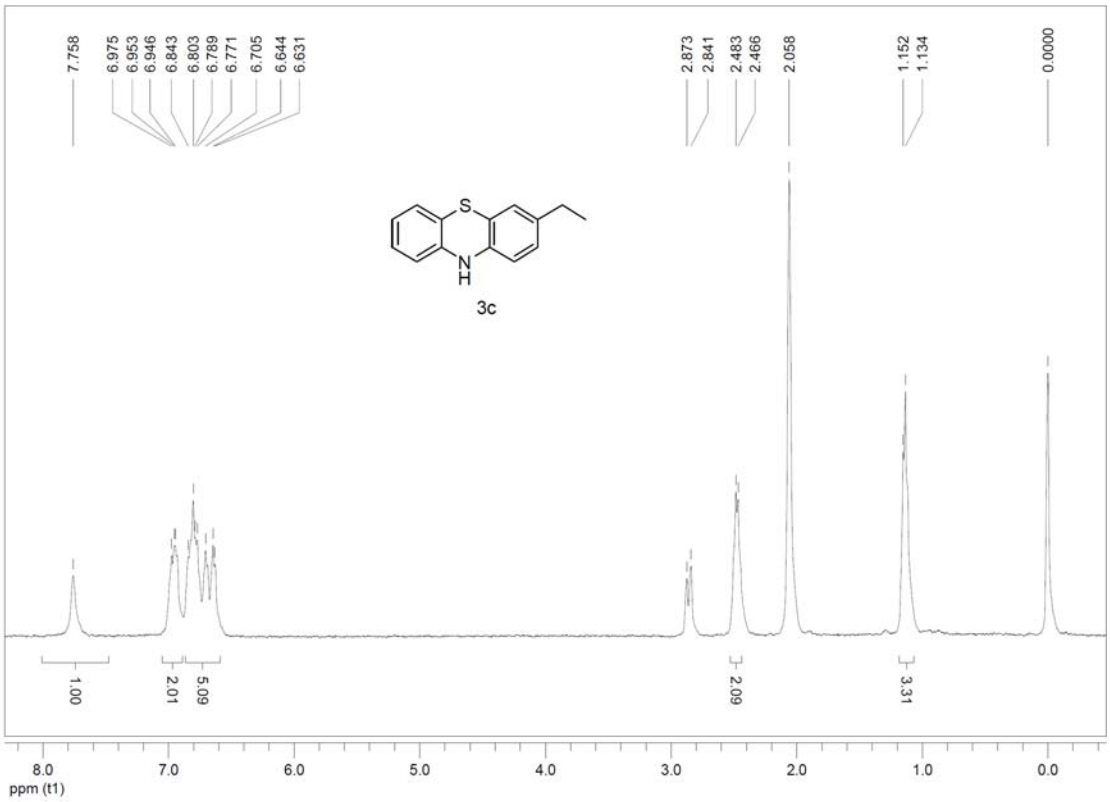
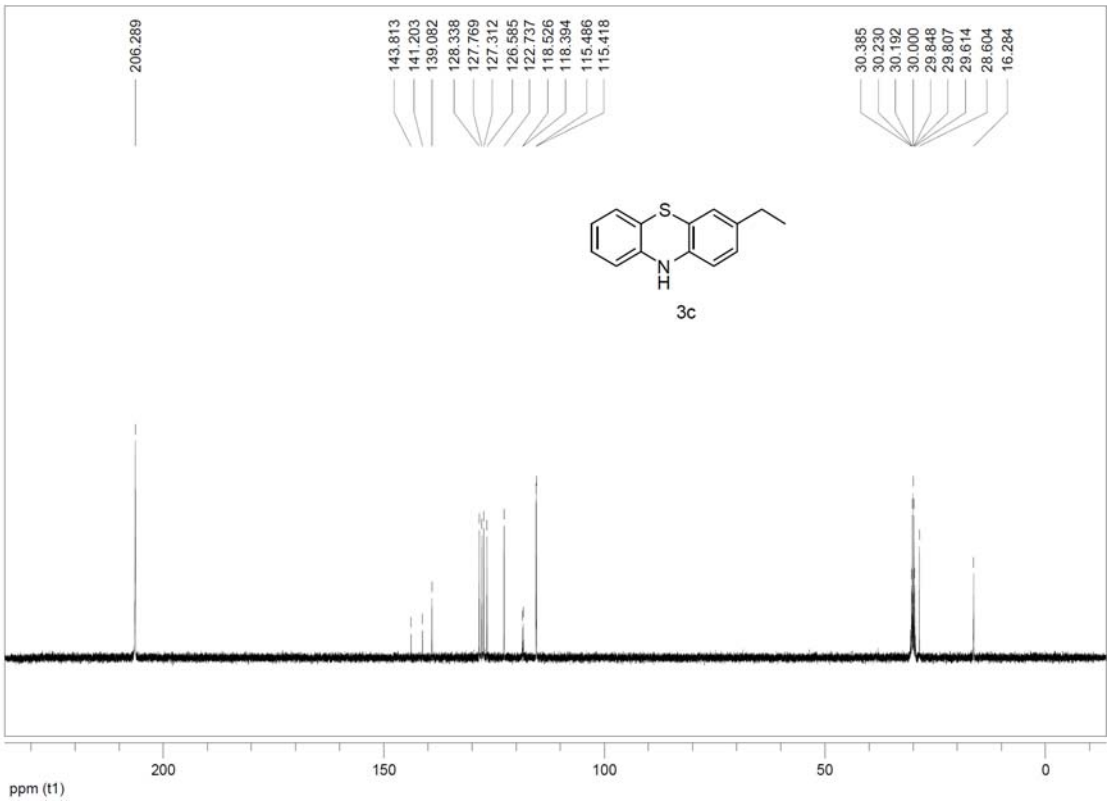
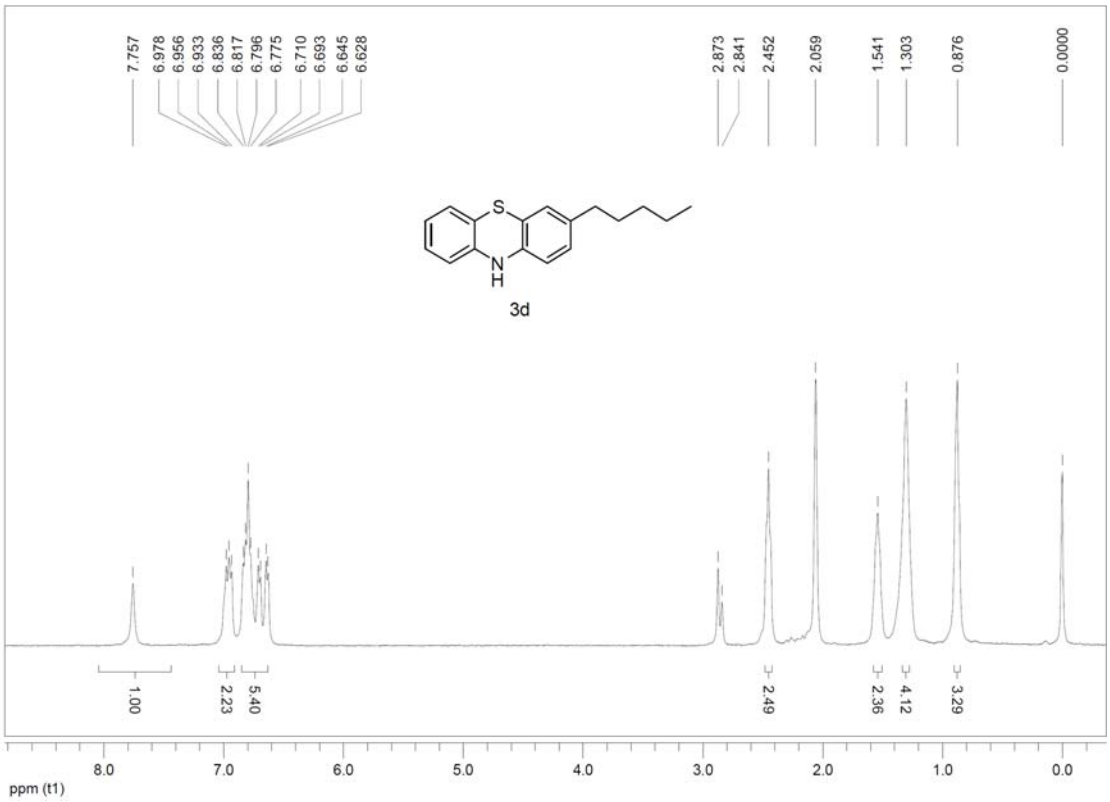
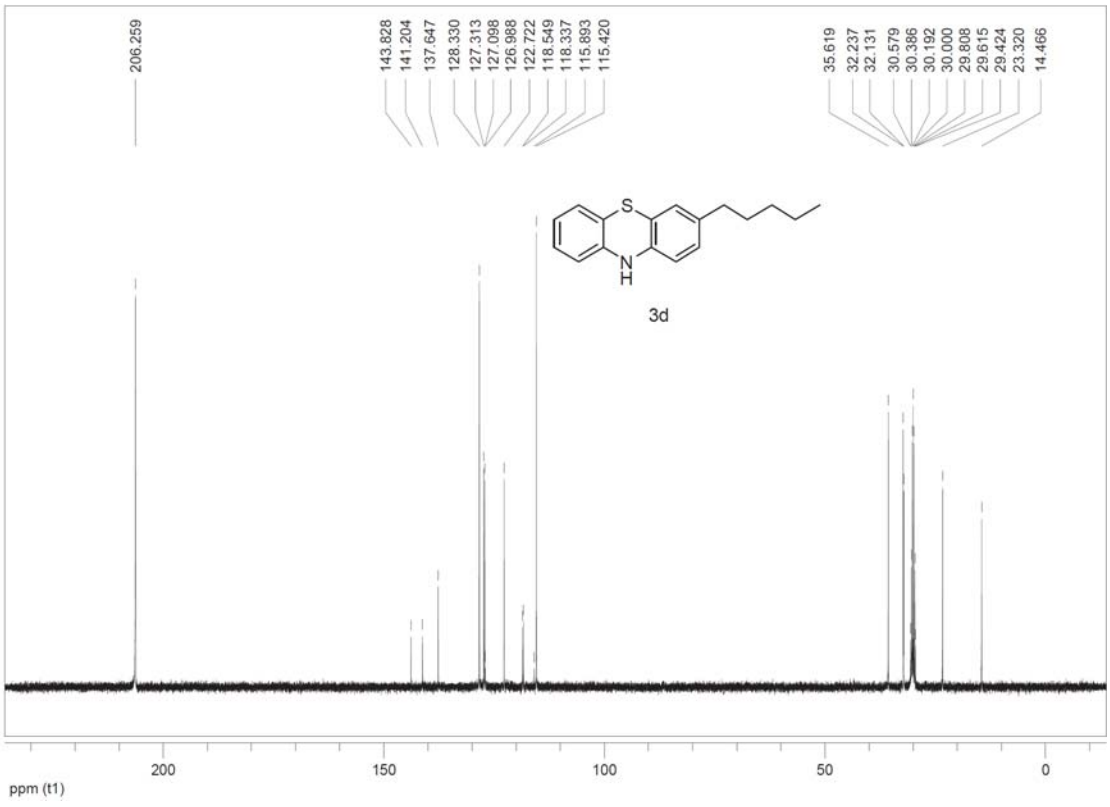
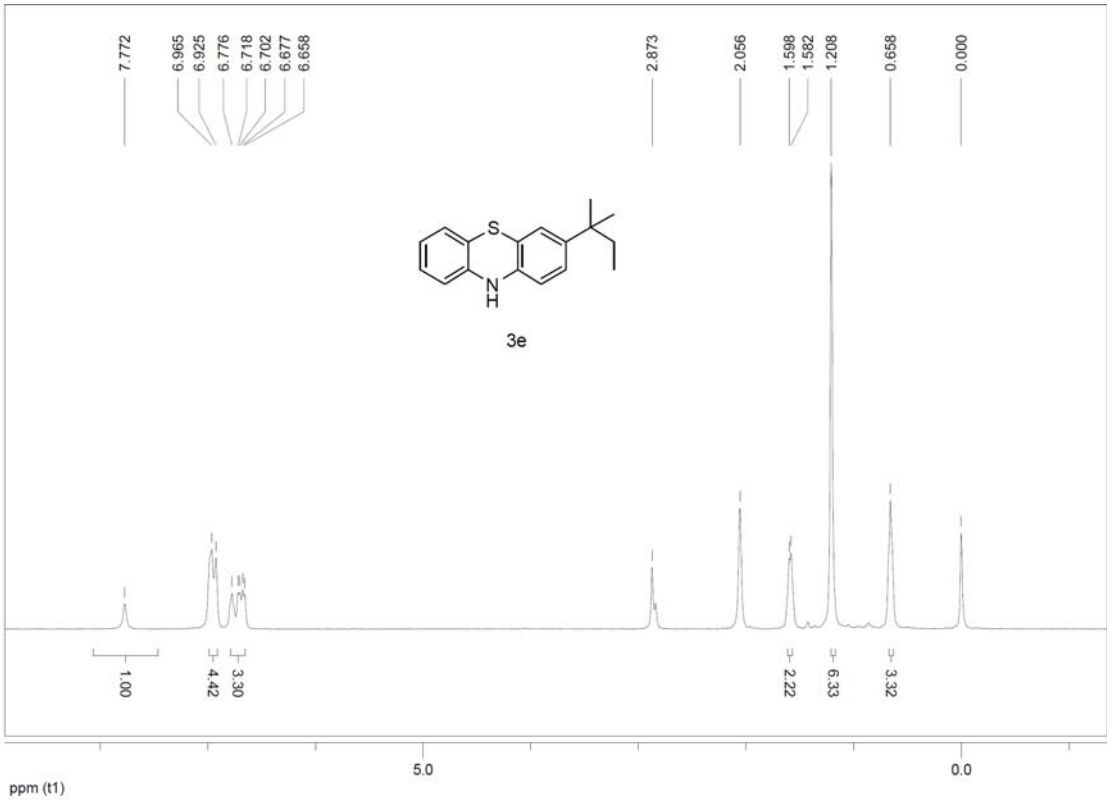
143.801
143.792
141.016
140.977
132.339
128.895
128.347
127.648
127.302
122.706
118.326
115.354
30.578
30.385
30.192
30.000
29.808
29.615
29.424
20.491
S
CH3
N
H
3b
200
150
100
50
0
ppm (t1)







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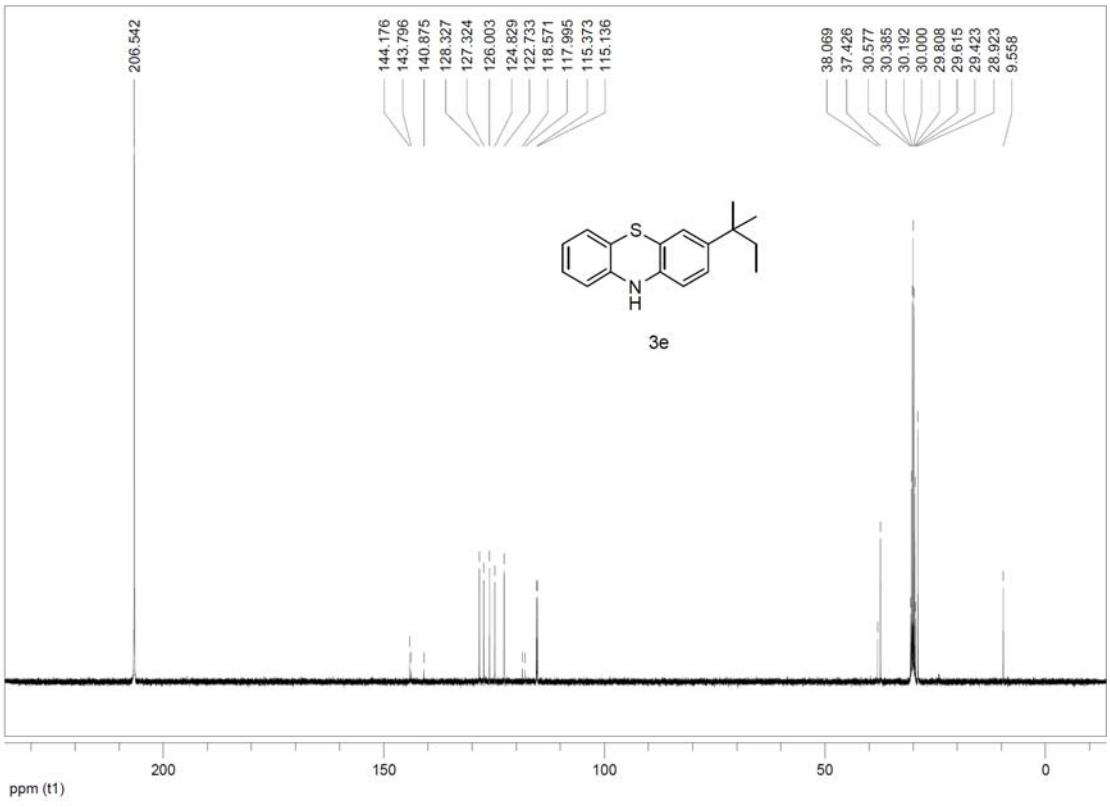
6.925
6.776
6.718
6.702
6.677
6.658
2.873
2.056
1.598
1.582
1.208
0.658
0.000
3e
1.00
4.42
3.30
2.22
6.33
3.32
5.0
0.0
ppm (t1)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124.829
122.733
118.571
117.995
115.373
115.136
38.069
37.426
30.577
30.385
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30.000
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29.615
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3e
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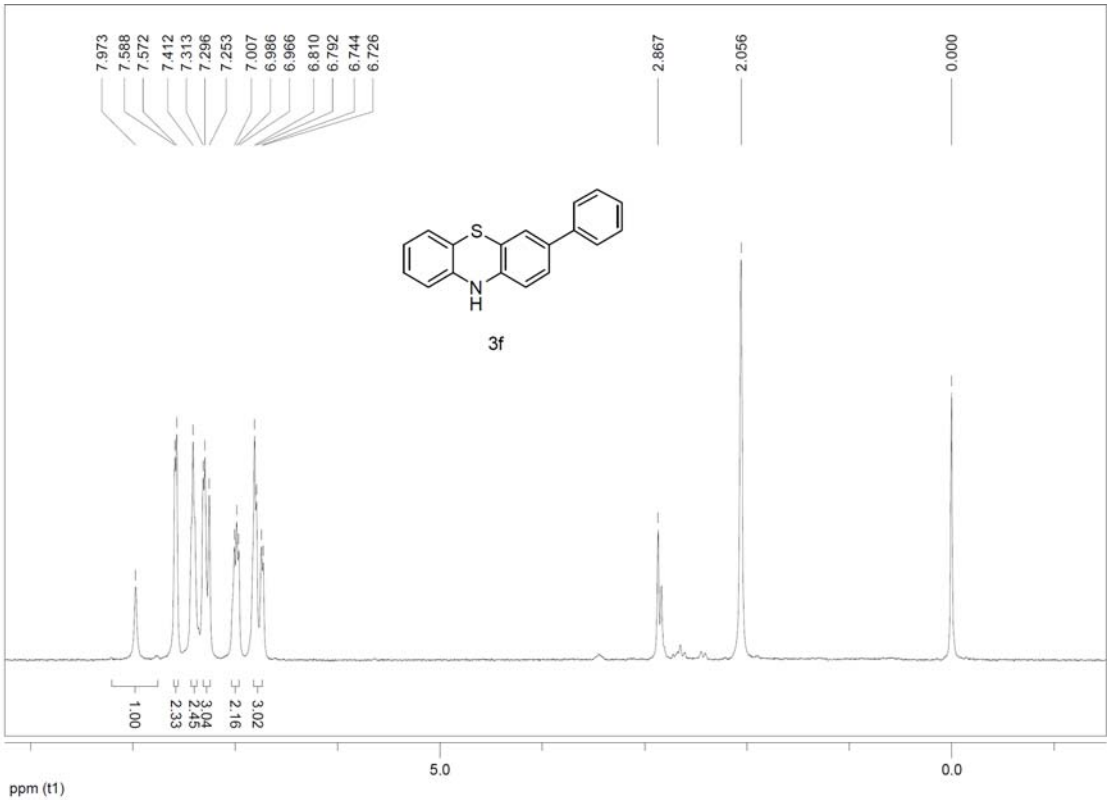
150
100
50
0
ppm (t1)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6.966
6.810
6.792
6.744
6.726
2.867
2.056
0.000
S
N
H
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2.33
2.45
3.04
2.16
3.02
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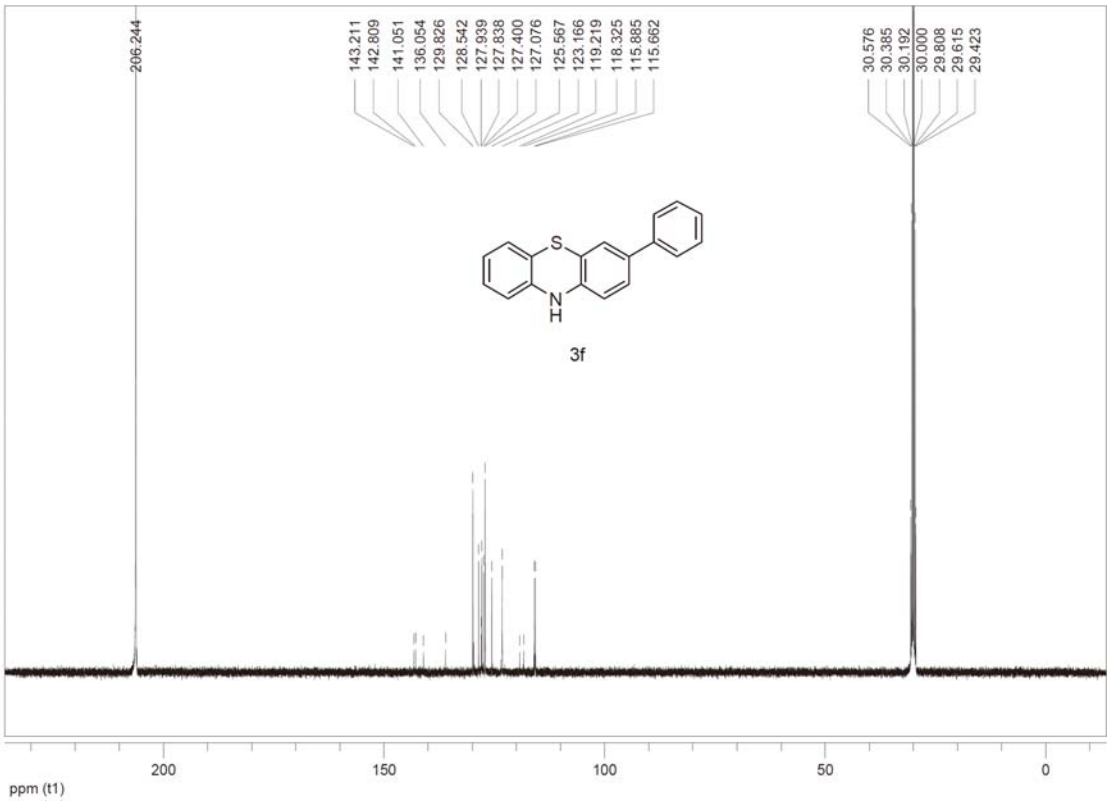
0.0
ppm (t1)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142.809
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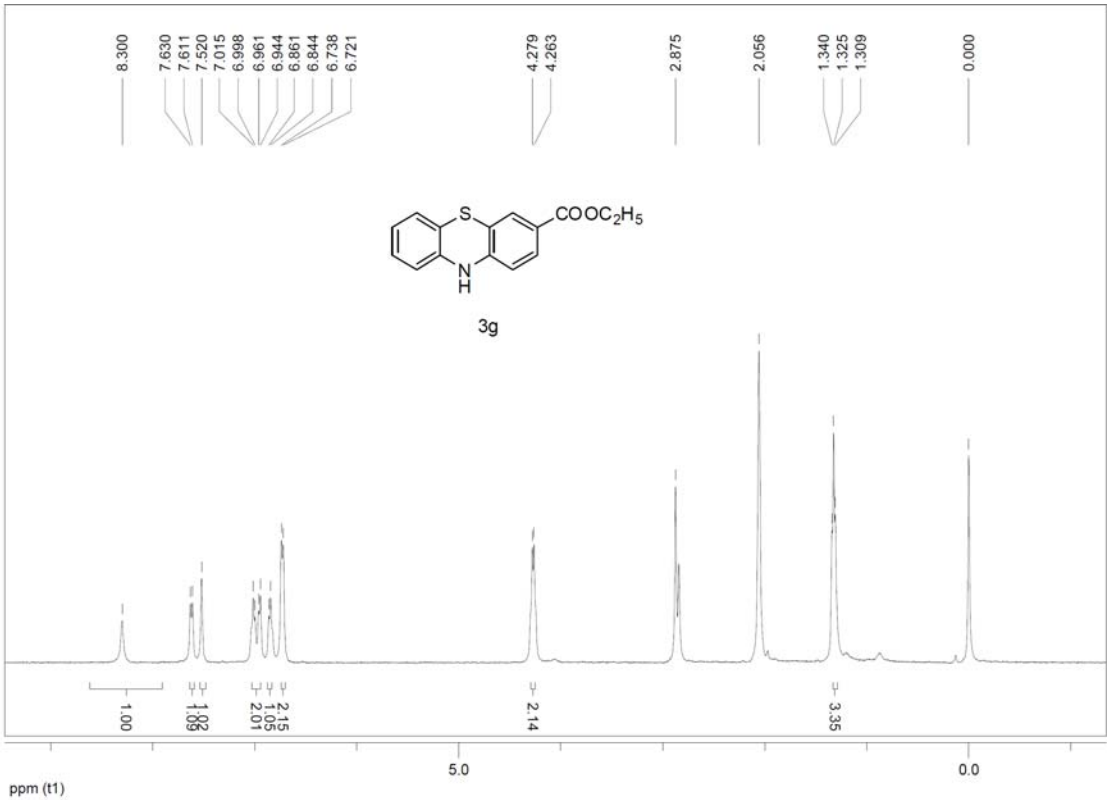
136.054
129.826
128.542
127.939
127.838
127.400
127.076
125.567
123.166
119.219
118.325
115.885
115.662
30.576
30.385
30.192
30.000
29.808
29.615
29.423
S
N
H
3f
200
150
100
50
0
ppm (t1)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7.611
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7.015
6.998
6.961
6.944
6.861
6.844
6.738
6.721
4.279
4.263
2.875
2.056
1.340
1.325
1.309
0.000
COOC2H5
3g
1.00
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1.09
2.01
1.05
2.15
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3.35
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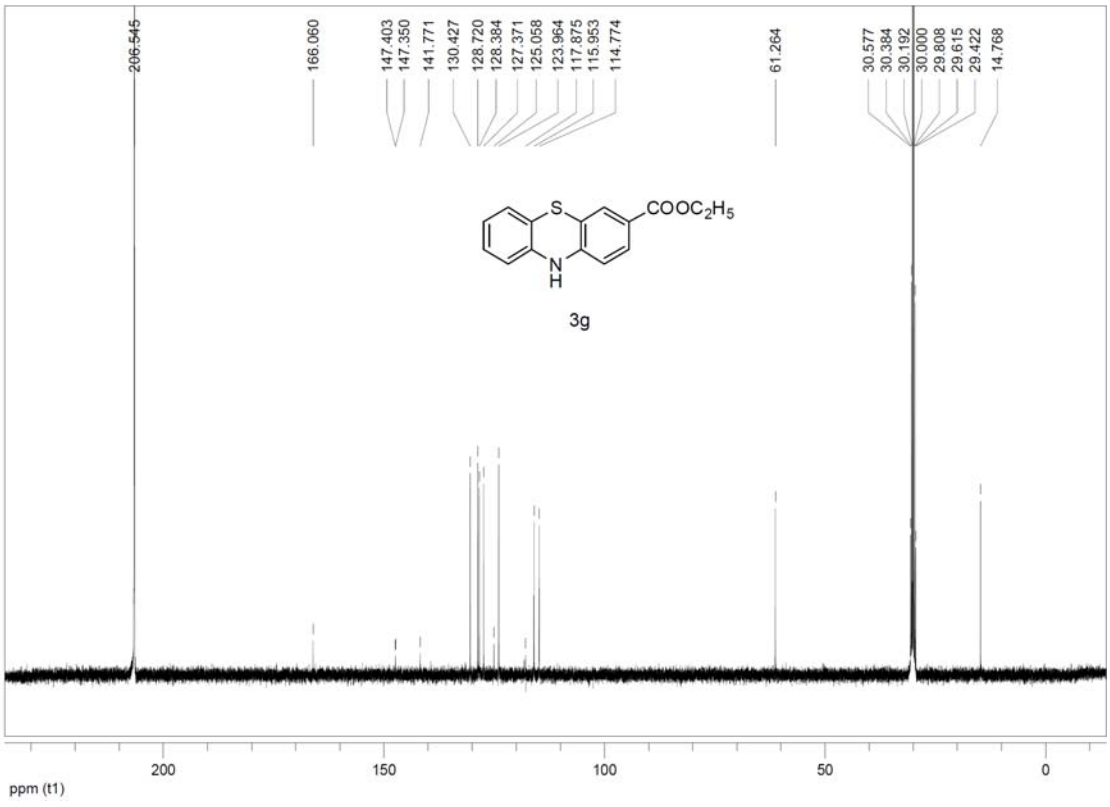
0.0
ppm (t1)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147.350
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130.427
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128.384
127.371
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115.953
114.774
61.264
30.577
30.384
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29.615
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COOC2H5
3g
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50
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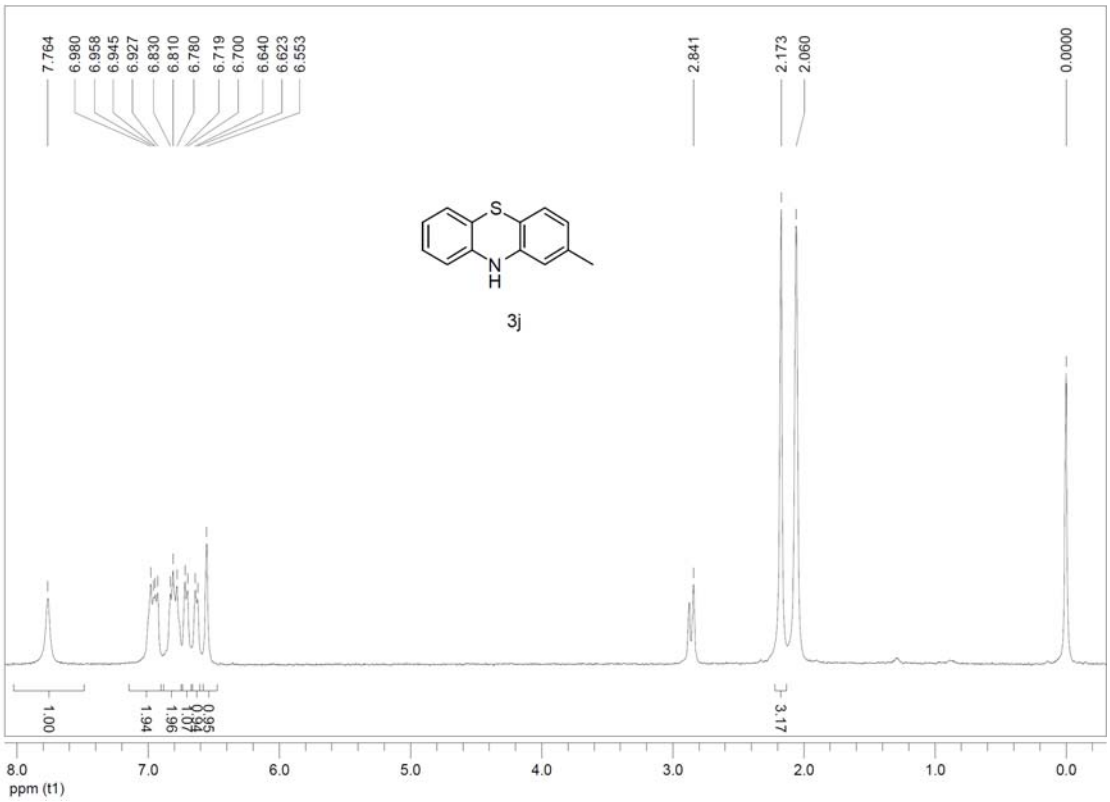
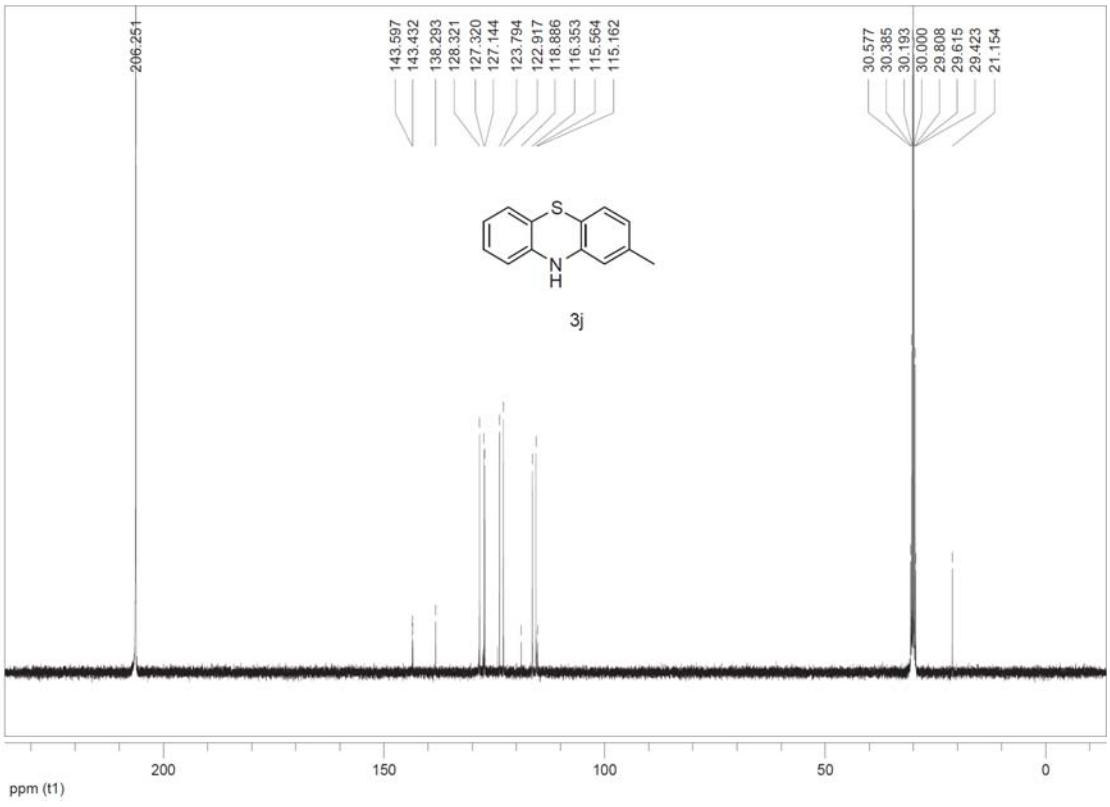
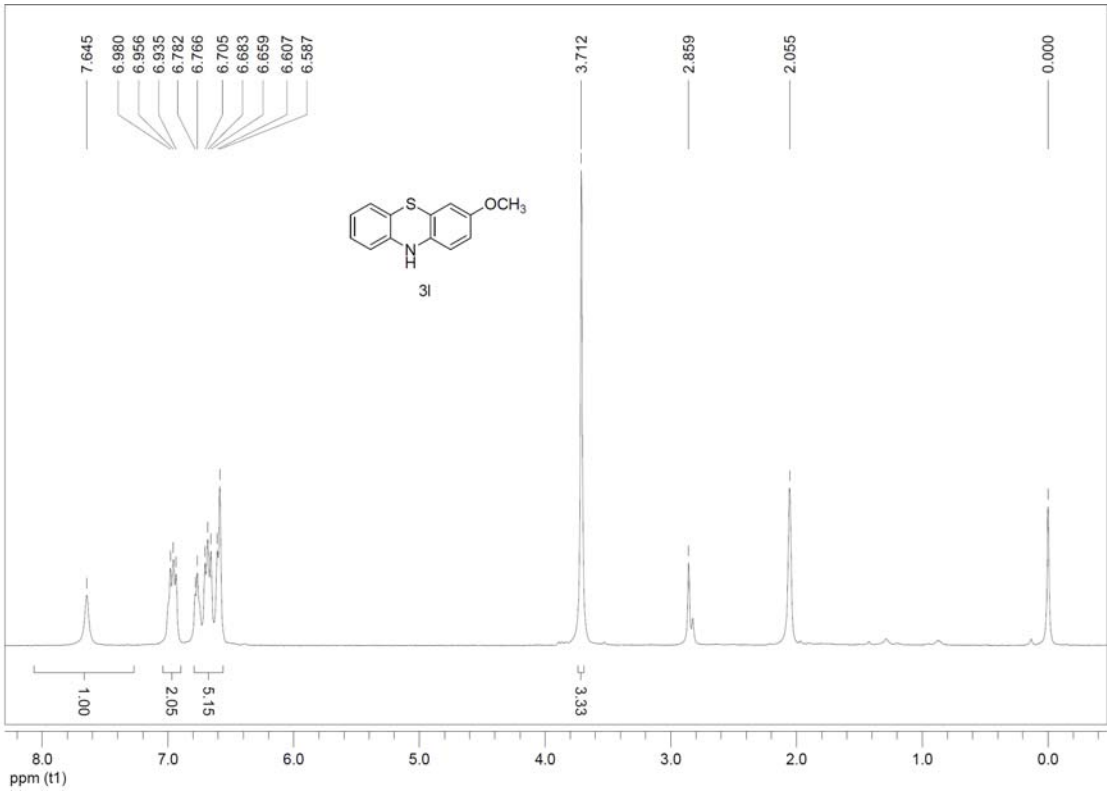
ppm (t1)



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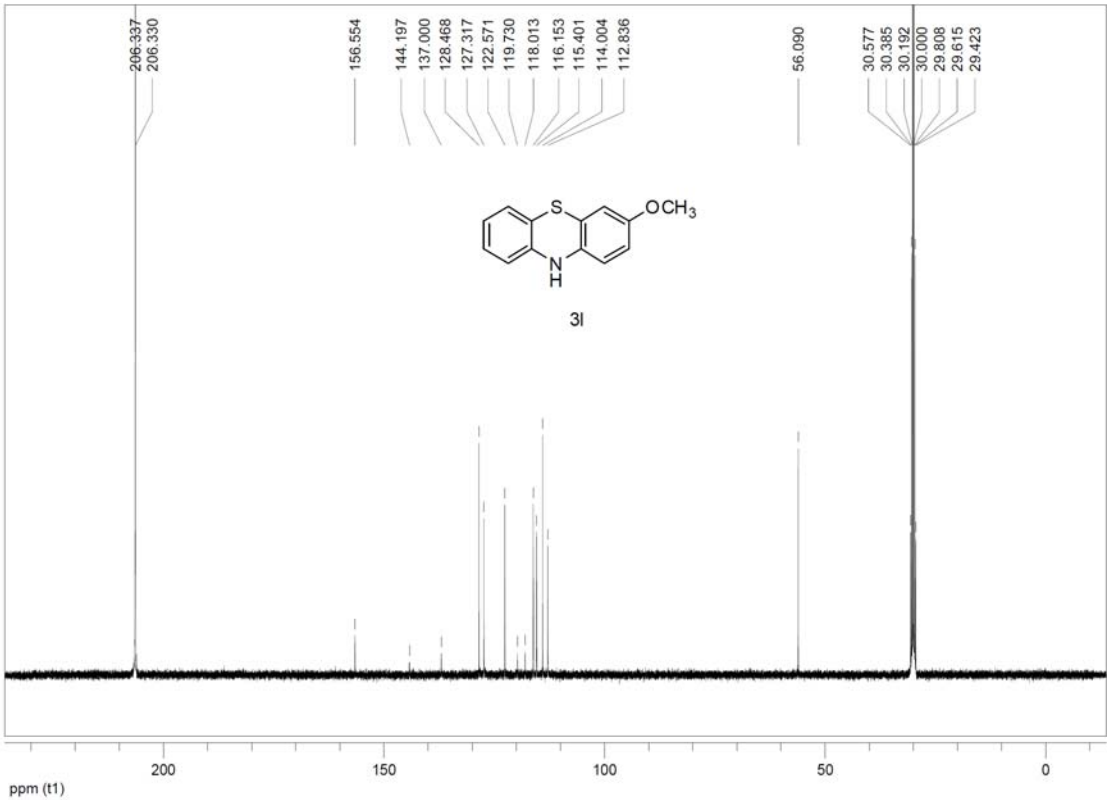
6.956
6.935
6.782
6.766
6.705
6.683
6.659
6.607
6.587
3.712
2.859
2.055
0.000
OCH3
S
N
H
3l
1.00
2.05
5.15
3.33
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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118.013
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115.401
114.004
112.836
56.090
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30.385
30.192
30.000
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29.615
29.423
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S
N
H
3l
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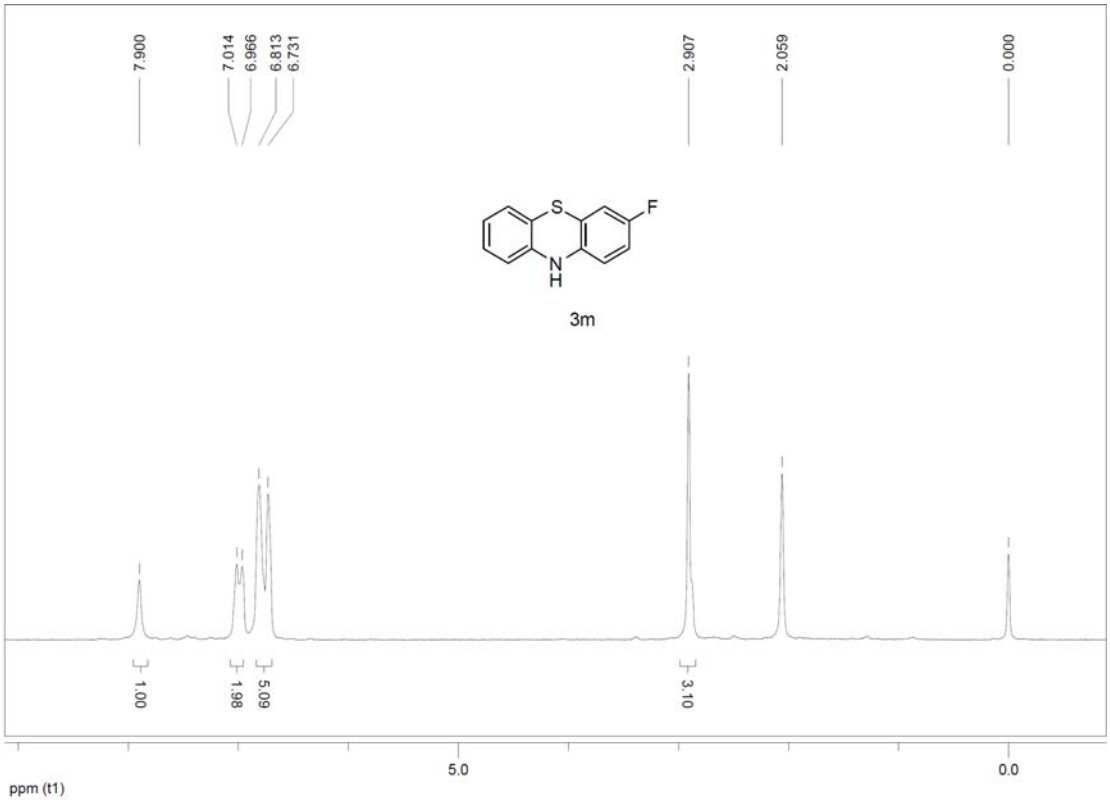
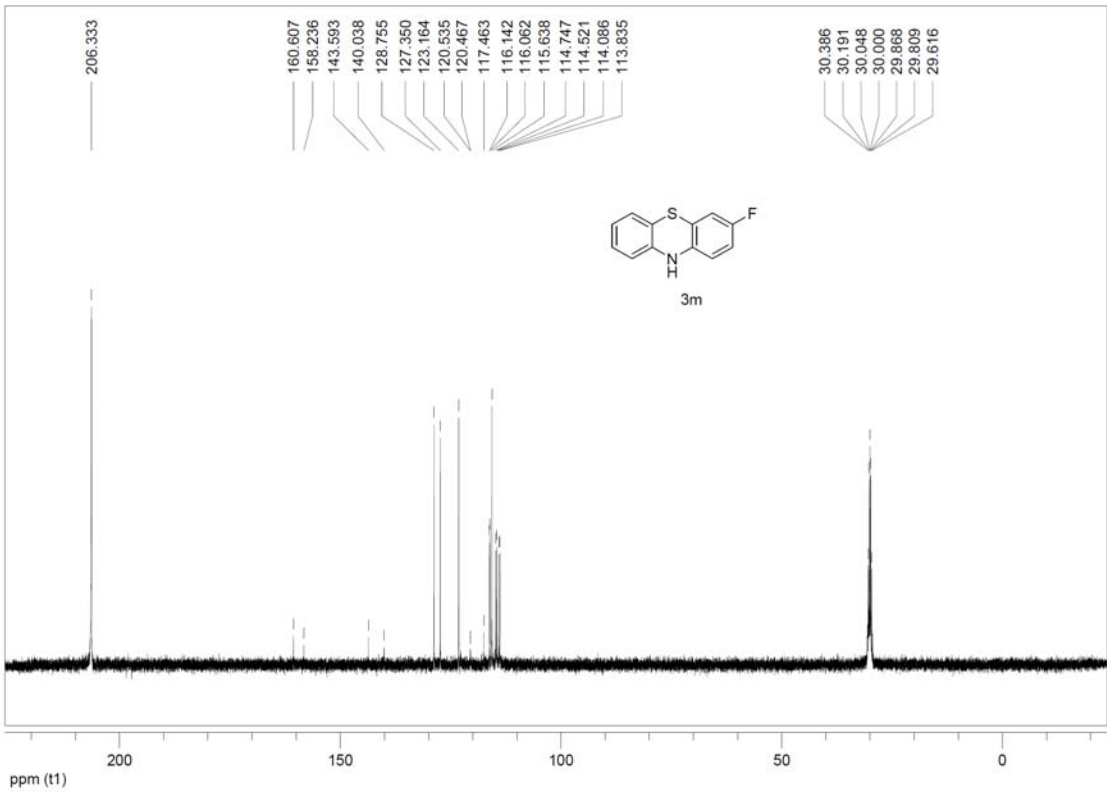
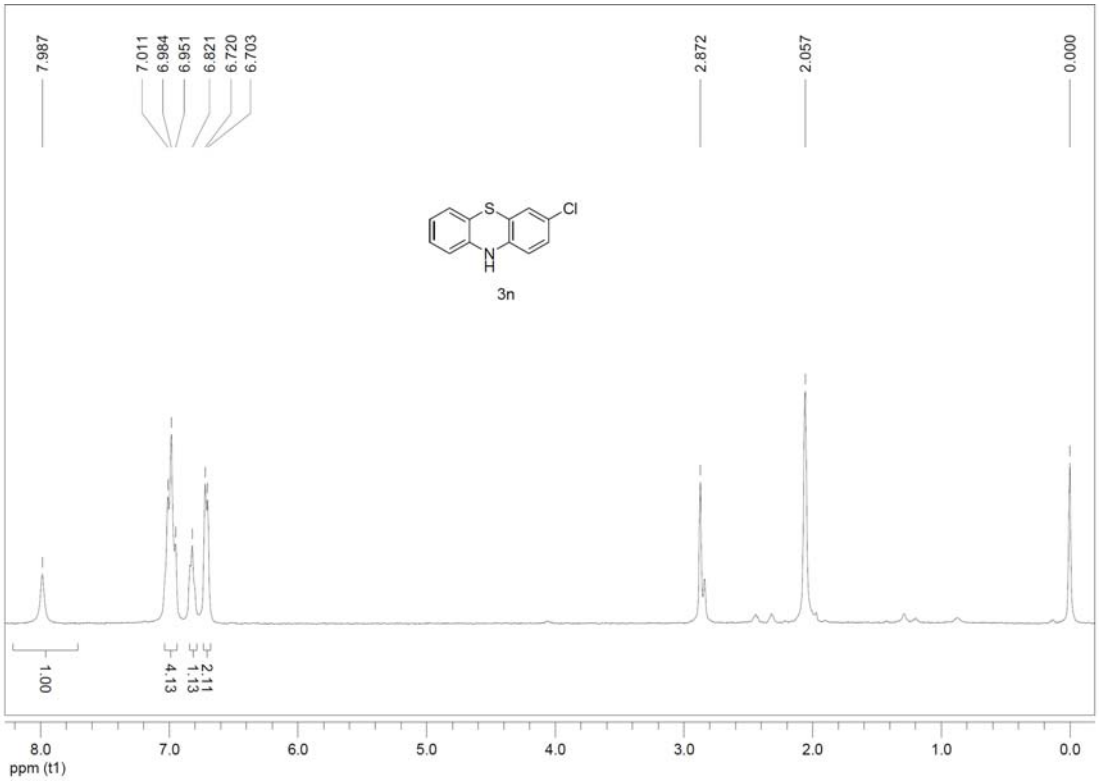
100
50
0
ppm (t1)



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Cl
N
H
3n
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4.13
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7.0
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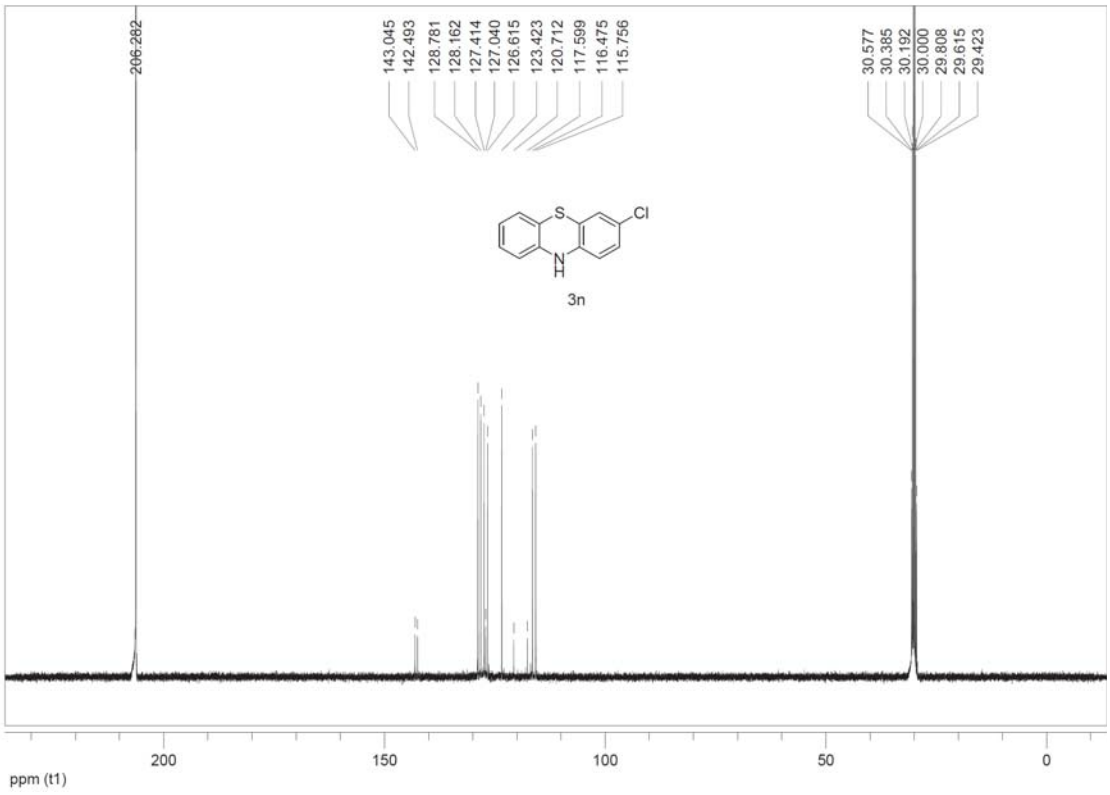
ppm (t1)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30.385
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30.000
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29.615
29.423
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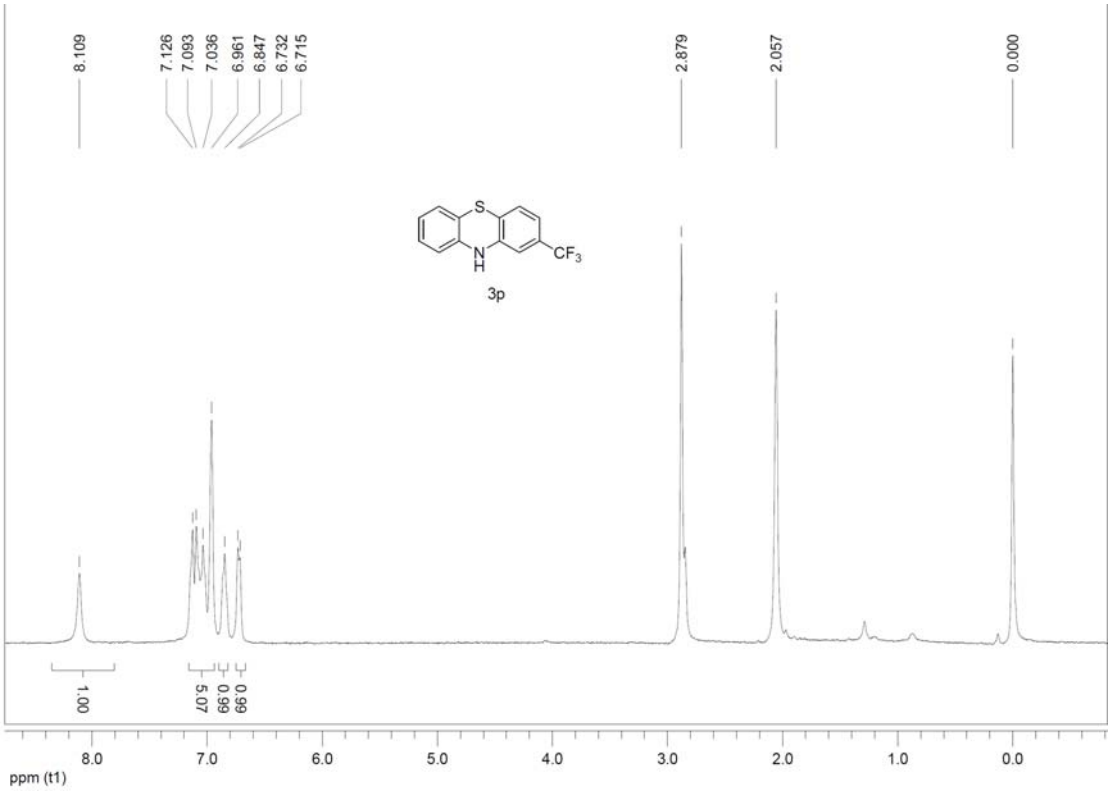
50
0
ppm (t1)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7.036
6.961
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6.715
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2.057
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1.00
5.07
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5.0
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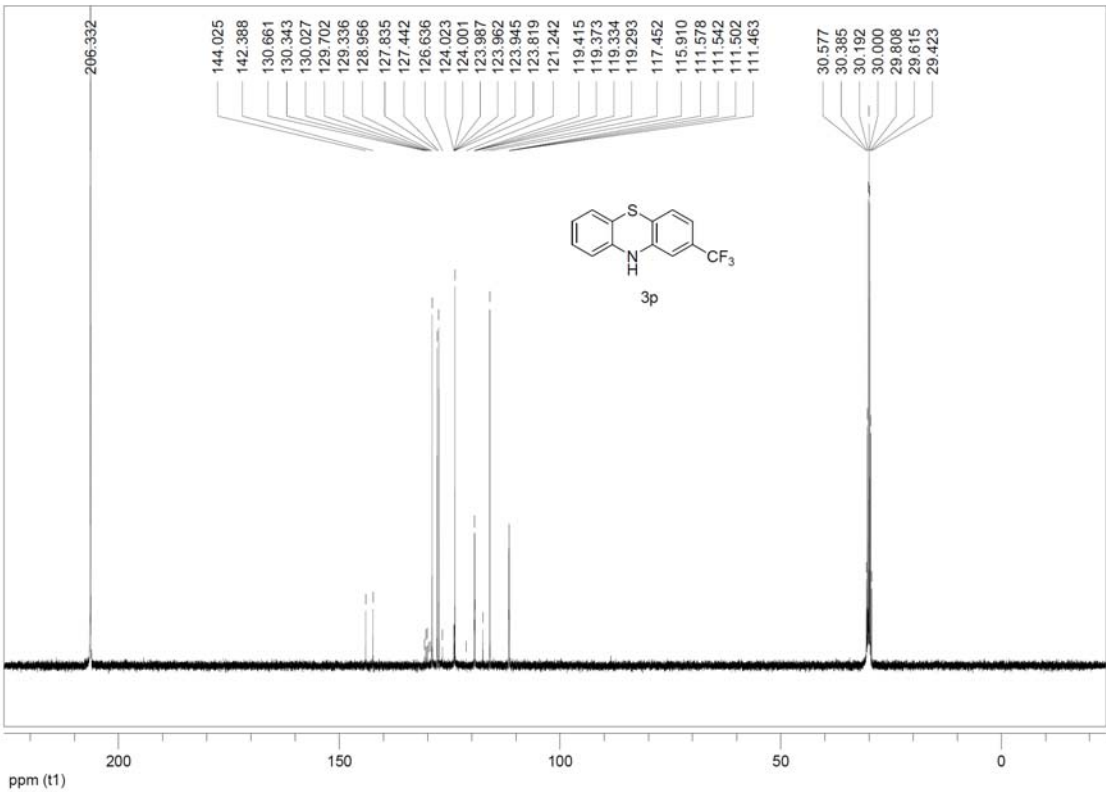
2.0
1.0
0.0
ppm (t1)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127.442
126.636
124.023
124.001
123.987
123.962
123.945
123.819
121.242
119.415
119.373
119.334
119.293
117.452
115.910
111.578
111.542
111.502
111.463
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29.423
3p
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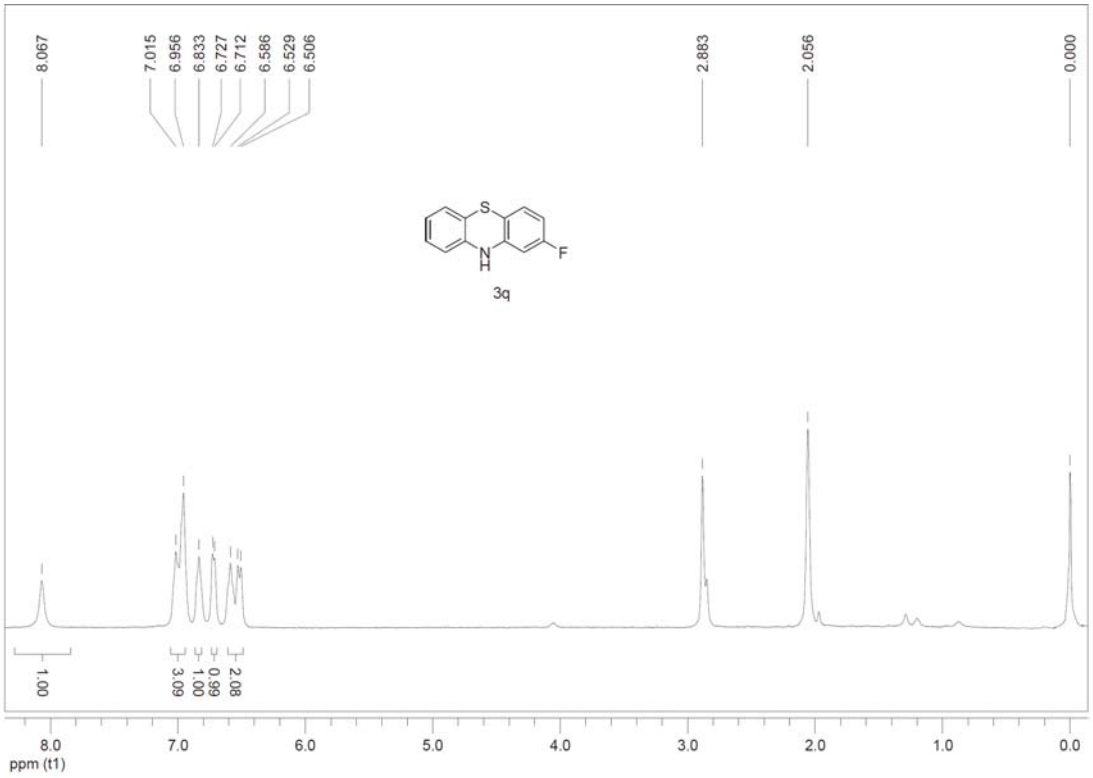
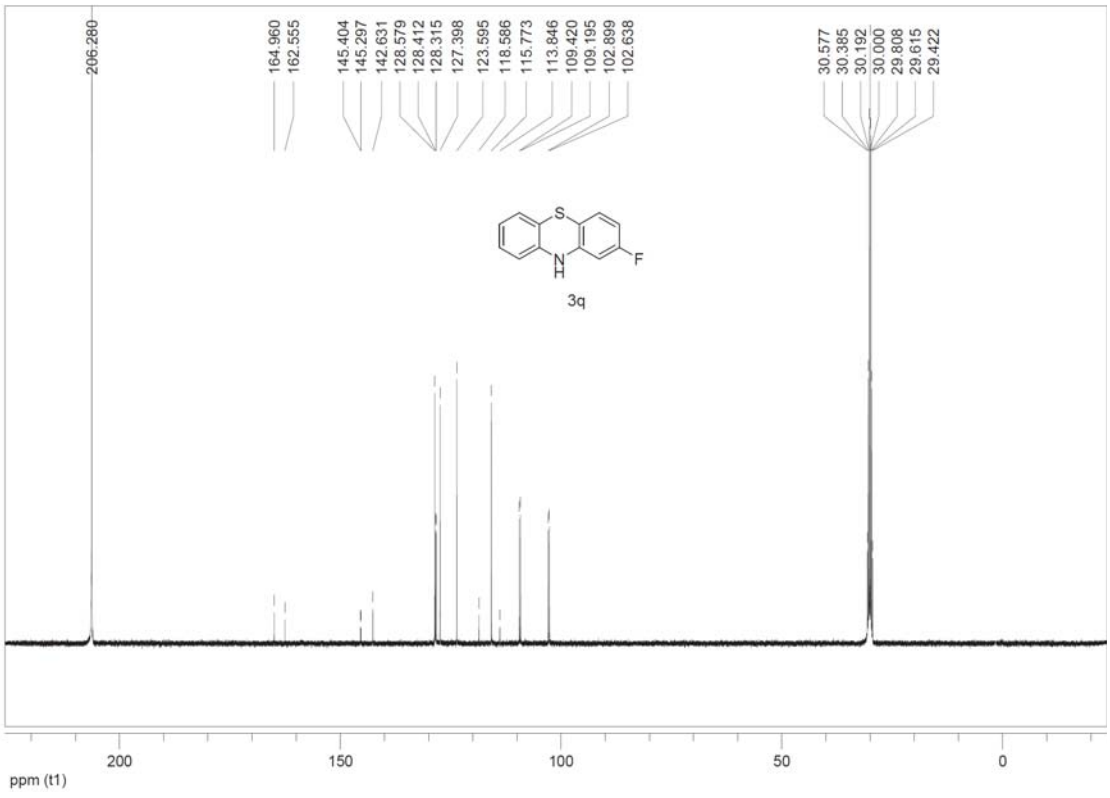
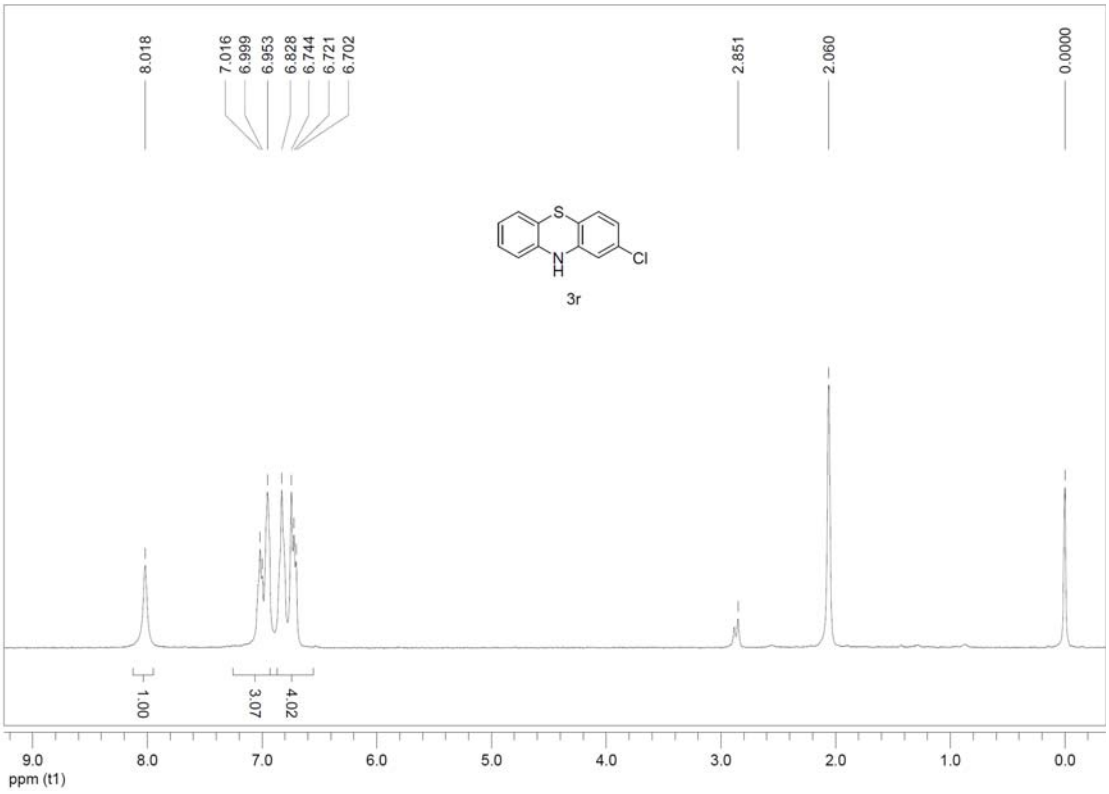
150
100
50
0
ppm (t1)



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6.721
6.702
2.851
2.060
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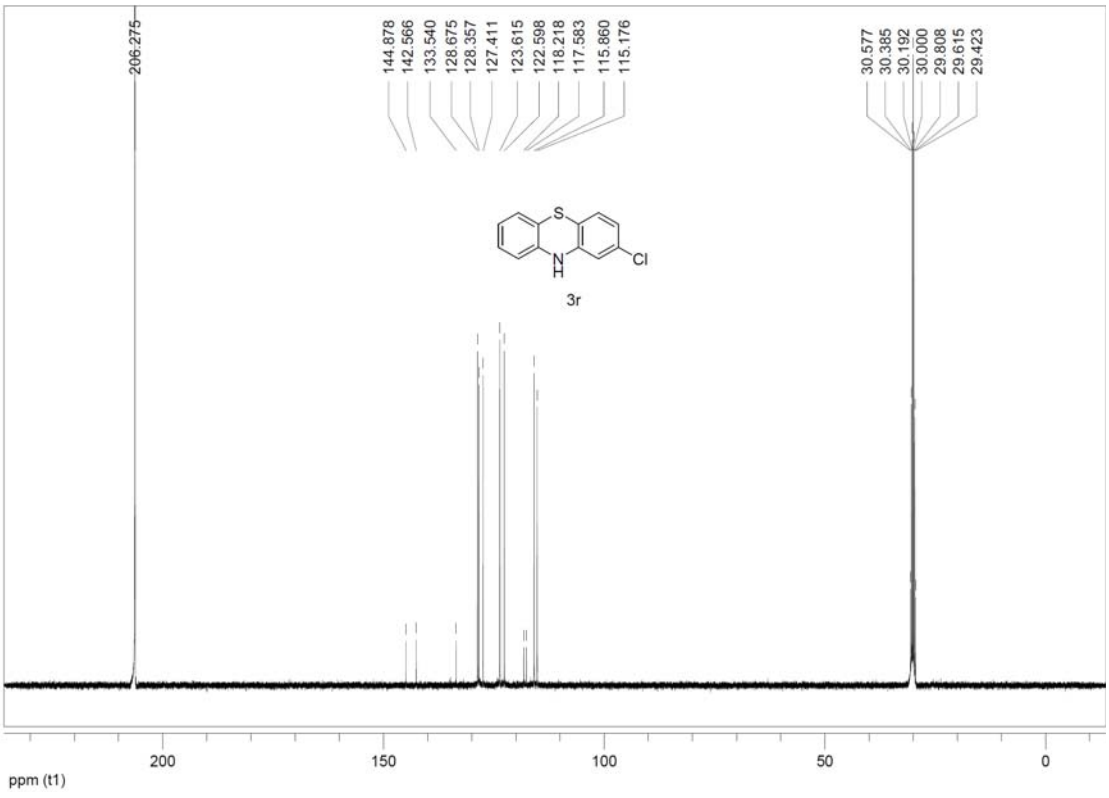
N
H
Cl
3r
1.00
3.07
4.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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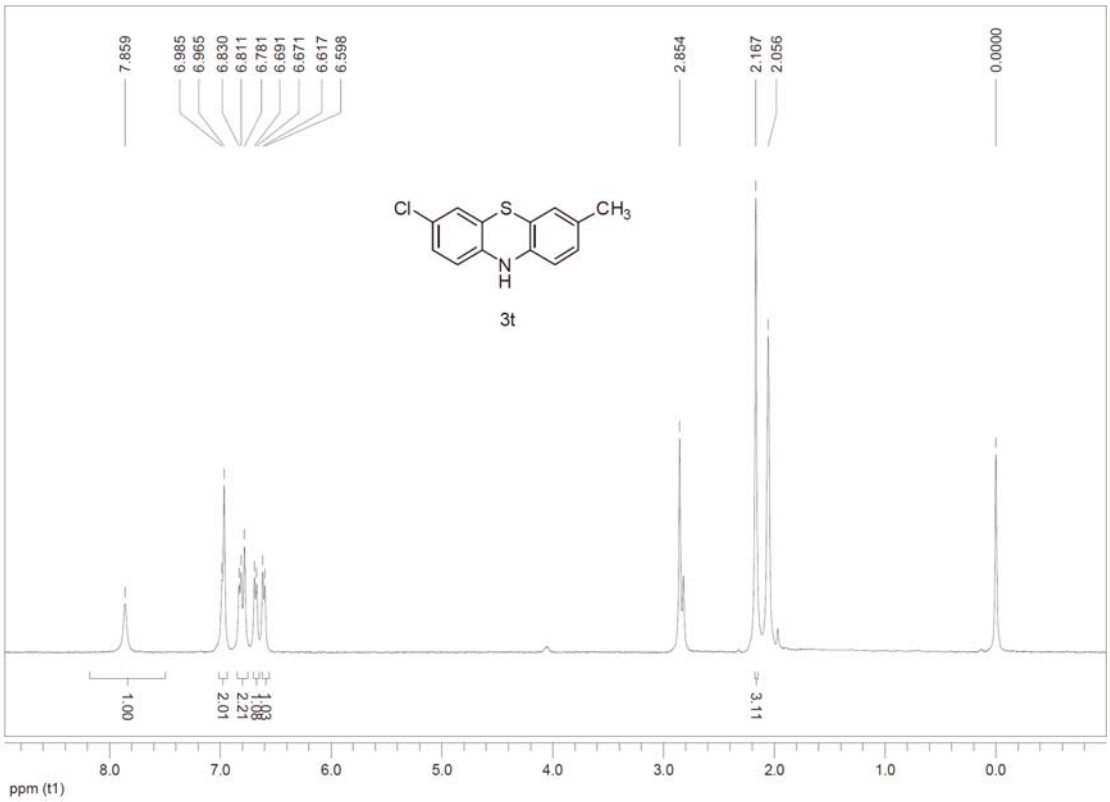
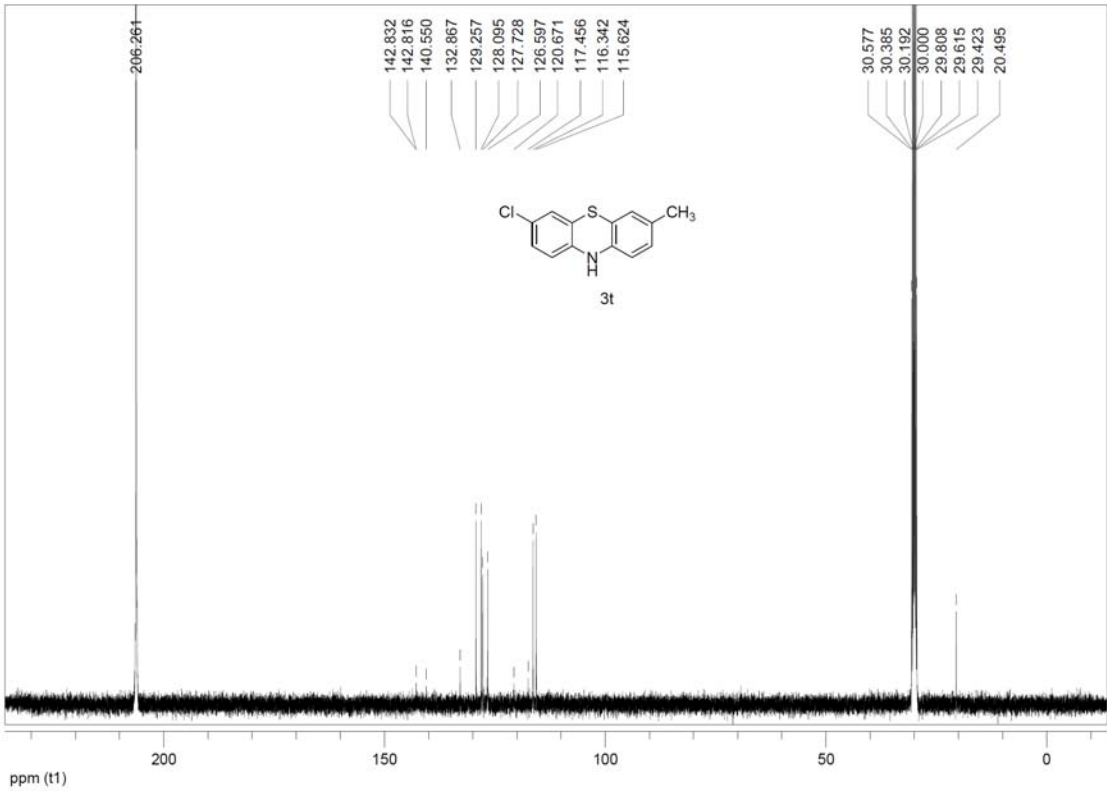
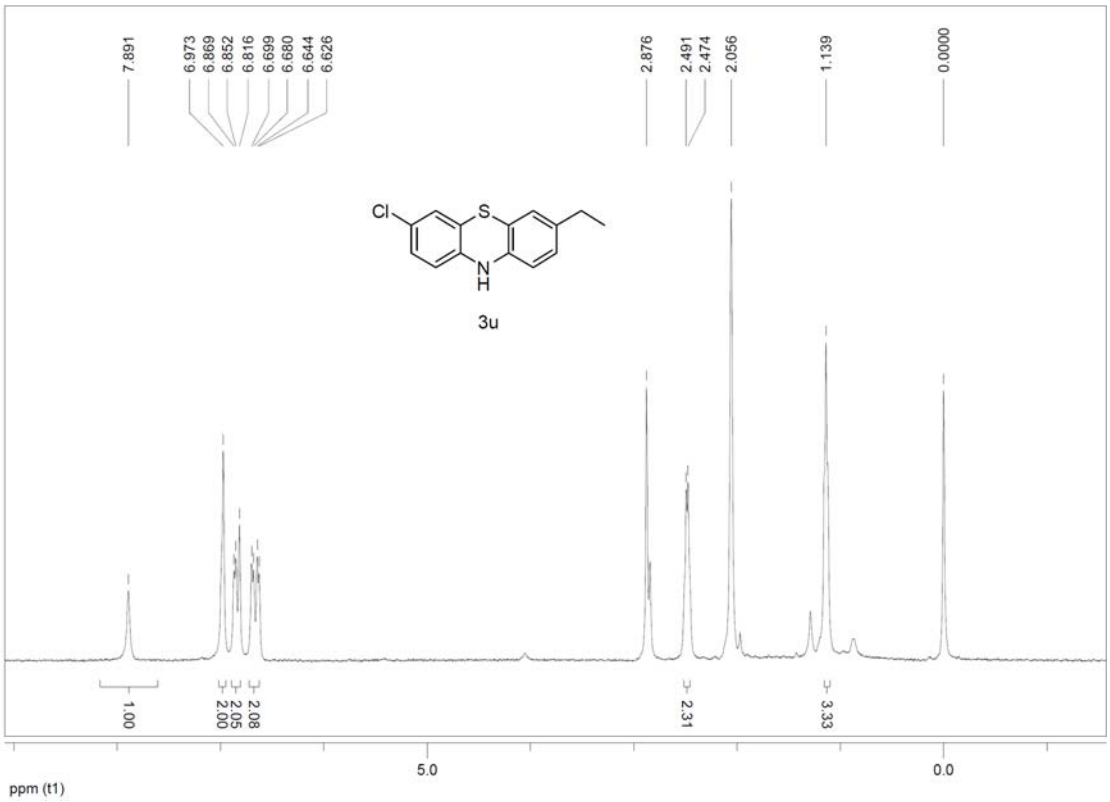
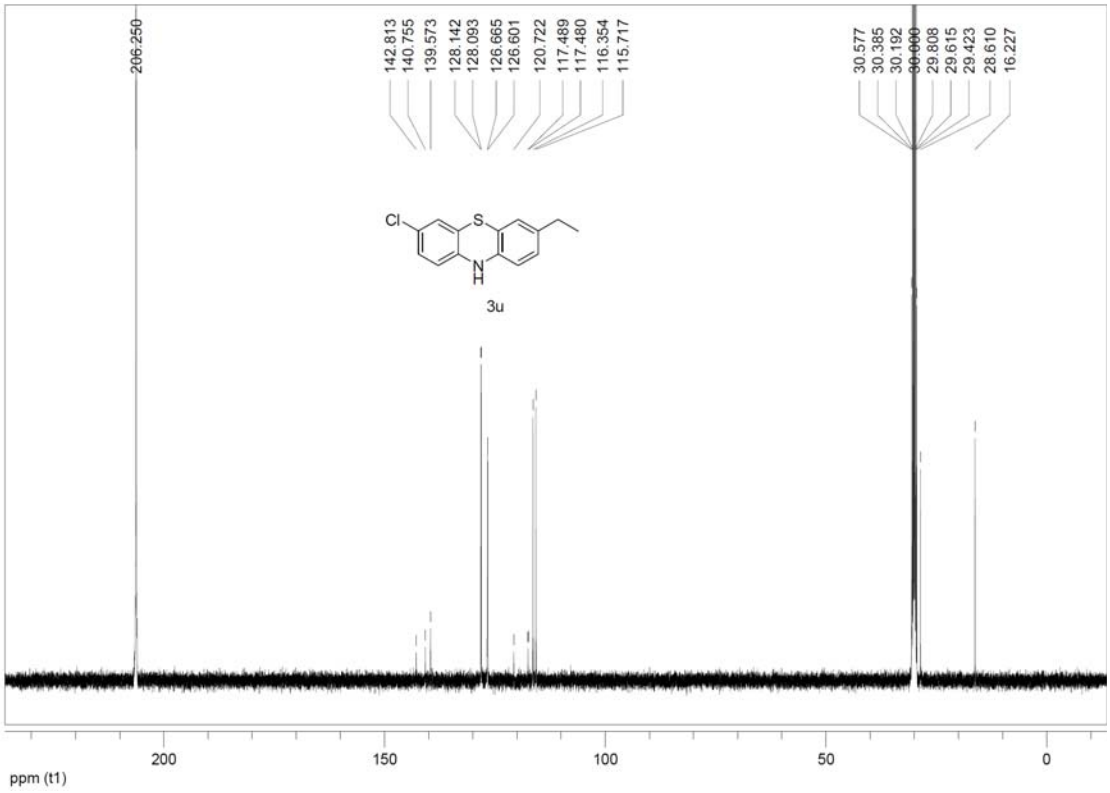
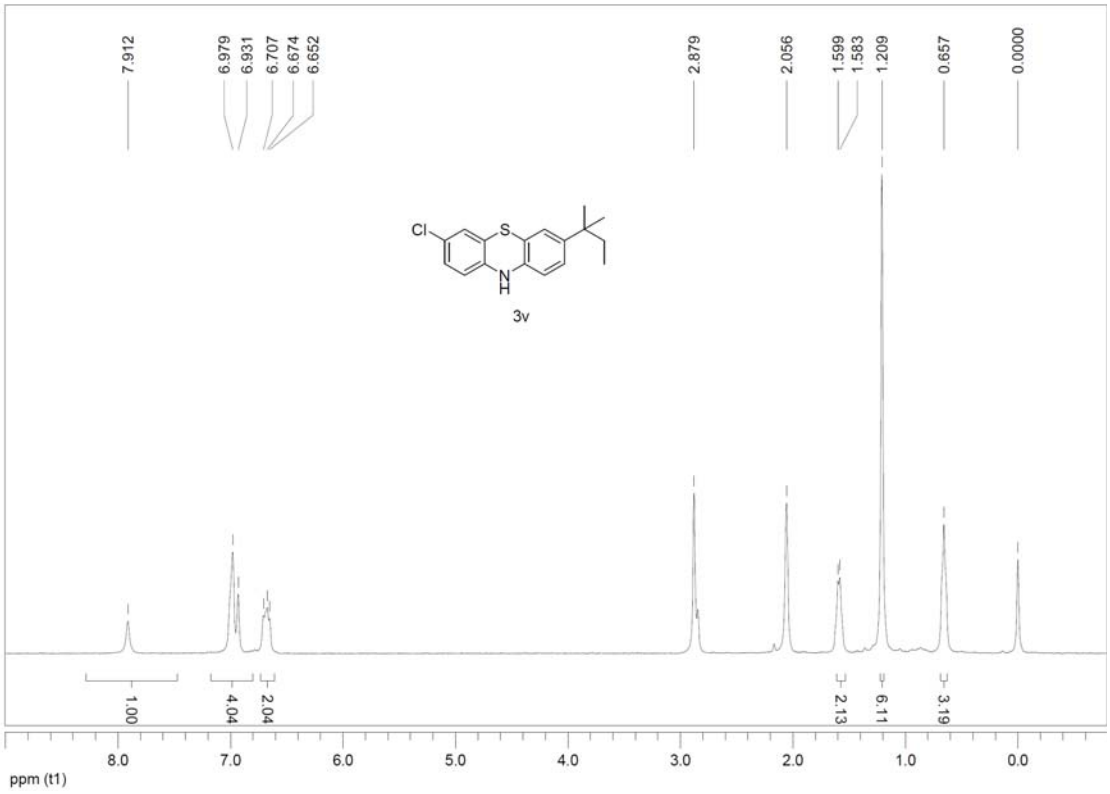
128.357
127.411
123.615
122.598
118.218
117.583
115.860
115.176
30.577
30.385
30.192
30.000
29.808
29.615
29.423
S
N
H
Cl
3r
200
150
100
50
0
ppm (t1)

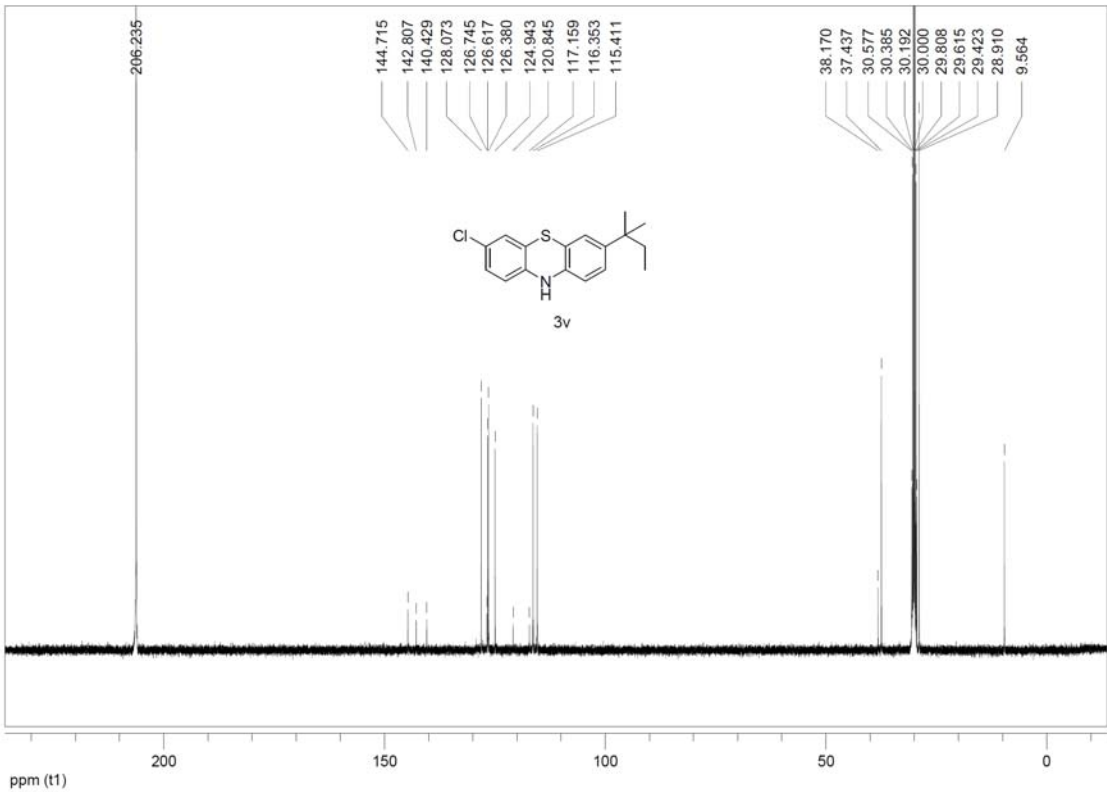

7.859
6.985
6.965
6.830
6.811
6.781
6.691
6.671
6.617
6.598
2.854
2.167
2.056
0.0000
Cl
S
CH3
N
H
3t
1.00
2.01
2.21
1.08
1.03
3.11
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

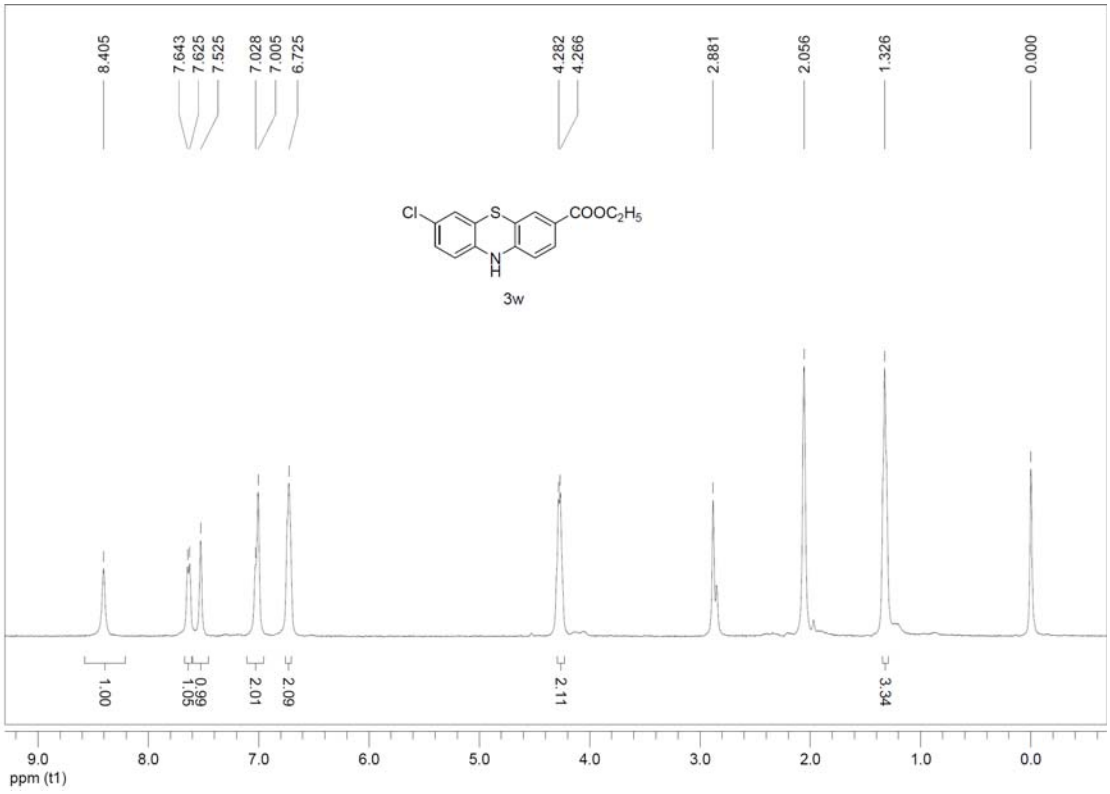

206.261
142.832
142.816
140.550
132.867
129.257
128.095
127.728
126.597
120.671
117.456
116.342
115.624
30.577
30.385
30.192
30.000
29.808
29.615
29.423
20.495
Cl
S
CH3
N
H
3t
200
150
100
50
0
ppm (t1)

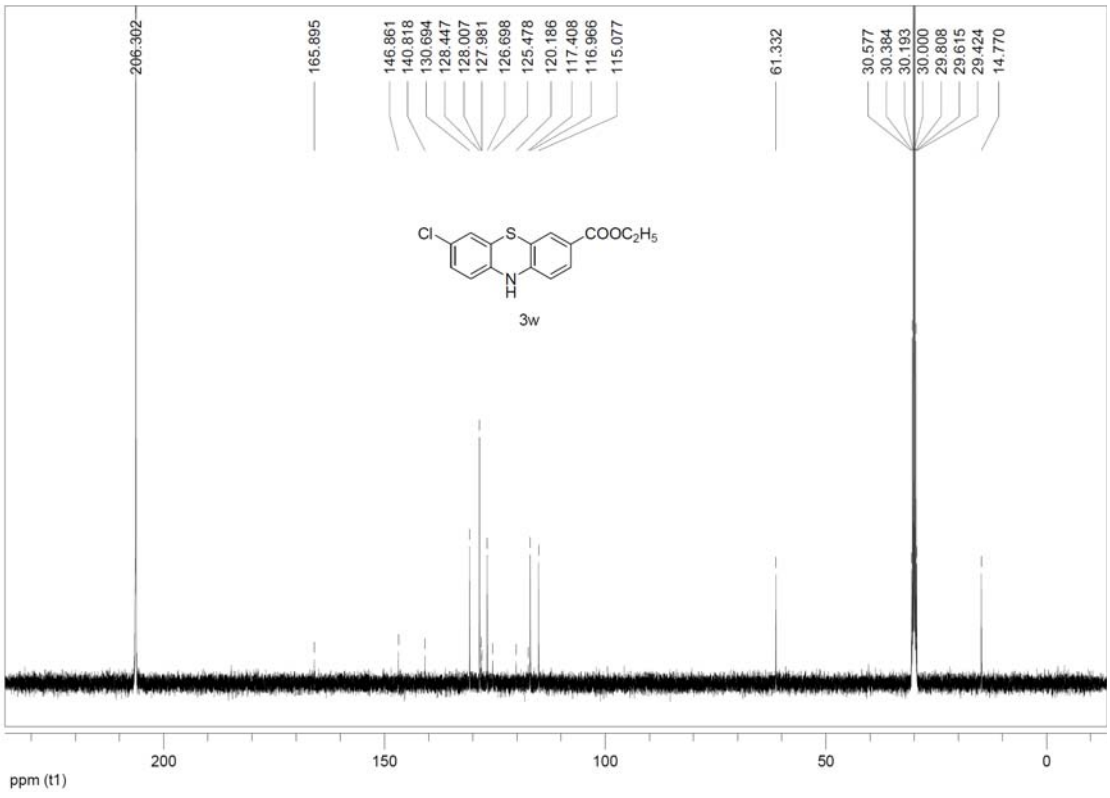

7.891
6.973
6.869
6.852
6.816
6.699
6.680
6.644
6.626
2.876
2.491
2.474
2.056
1.139
0.0000
Cl
S
N
H
3u
1.00
2.00
2.05
2.08
2.31
3.33
5.0
0.0
ppm (t1)


206.250
142.813
140.755
139.573
128.142
128.093
126.665
126.601
120.722
117.489
117.480
116.354
115.717
30.577
30.385
30.192
30.000
29.808
29.615
29.423
28.610
16.227
Cl
S
N
H
3u
200
150
100
50
0
ppm (t1)





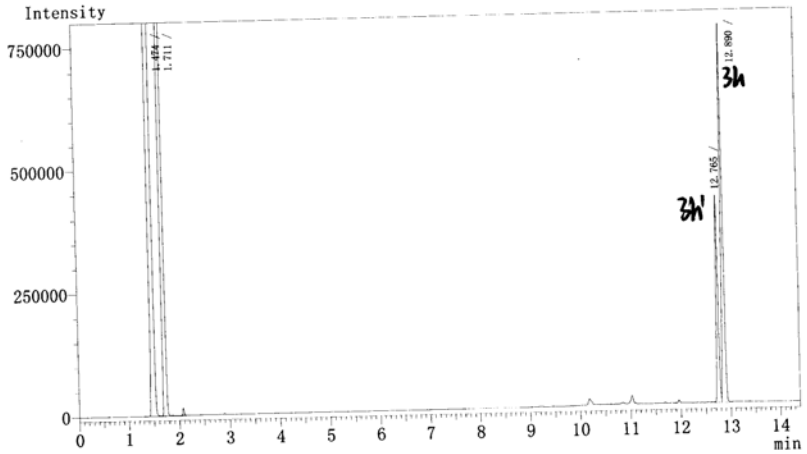




E:\ﾁﾎﾔﾆｷ藕ｷﾔ琚豺ﾏｵﾁﾐ\882.gcd

采集日期和时间	: 2013-1-28 10:23:55
用户名	: Admin
样品瓶号	: 1
样品名称	:
样品 ID	:
样品类型	: 未知样
进样量	:
内标量	:
数据名	: E:\廖云峰\吩噻嗪系列\882.gcd
方法文件名	: E:\20121025\250.gcm

GC analysis for 3h and 3h'



峰号	保留时间	面积	面积%	峰高
1	1.474	229359006	82.5355	156679875
2	1.711	45267813	16.2898	30650080
3	12.765	1100909	0.3962	418084
4	12.890	2163663	0.7786	758688
全部		277891391	00.0000	188506727

1:2

1 / 1