Supplementary Information

Design and synthesis of novel class of CK2 inhibitors: application of copper- and gold-catalysed cascade reactions for fused nitrogen heterocycles

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Methyl 6-{7-(methylsulfonyl)dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl}nicotinate (11b). By use of the procedure for the synthesis of **11a**, **9** (50 mg, 0.21 mmol) and methyl 6-bromonicotinate (**10b**) (55 mg, 0.26 mmol) were converted to the title compound **11b** (50.9 mg, 65%) as a pale yellow solid: mp 240–241 °C; IR (neat): ν_{max}/cm^{-1} 1710 (C=O), 1282 (OCH_3); δ_H (400 MHz; $CDCl_3$; Me_4Si) 3.14 (3H, s), 3.99 (3H, s), 6.99 (1H, dd, *J* 3.9, 0.7 Hz), 7.02 (1H, dd, *J* 3.9, 0.7 Hz), 7.52 (1H, d, *J* 8.5 Hz), 7.73 (1H, d, *J* 3.7 Hz), 8.06 (1H, d, *J* 3.7 Hz), 8.45 (1H, dd, *J* 8.5, 2.2 Hz), 9.25 (1H, d, *J* 2.2 Hz), 9.44 (1H, s). δ_C (125 MHz; $CDCl_3$) 40.7, 52.4, 107.5, 107.8, 110.0, 111.7, 122.1, 126.4, 126.9, 129.3, 129.7, 139.8, 146.2, 147.6, 150.9, 154.7, 165.2; HRMS (FAB) *m/z* Calc. for $C_{17}H_{15}N_4O_4S$ (MH^+) 371.0809, found 371.0811.

Methyl 2-{7-(methylsulfonyl)dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl}isonicotinate (11c). By use of the procedure for the synthesis of **11a**, **9** (52 mg, 0.22 mmol) and methyl 2-bromoisonicotinate (**10c**) (57 mg, 0.26 mmol) were converted to the title compound **11c** (69.5 mg, 85%) as a white solid: mp 205–206 °C; IR (neat): ν_{max}/cm^{-1} 1720 (C=O), 1284 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si) 3.13 (3H, s), 4.02 (3H, s), 6.97–6.99 (2H, m), 7.71–7.72 (2H, m), 8.01 (1H, s), 8.06 (1H, d, *J* 3.4 Hz), 8.74 (1H, d, *J* 5.2 Hz), 9.37 (1H, s); δ_C (125 MHz; $CDCl_3$) 40.6, 53.0, 107.0, 107.0, 110.1, 112.5, 119.1, 126.4, 126.9, 129.5, 129.6, 140.1, 146.0, 147.4, 149.8, 153.0, 165.0; HRMS (FAB) *m/z* Calc. for $C_{17}H_{15}N_4O_4S$ (MH^+) 371.0809, found 371.0803.

Methyl 2-{7-(methylsulfonyl)dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl}nicotinate (11d). By use of the procedure for the synthesis of **11a**, **9** (50 mg, 0.22 mmol) and methyl 2-bromonicotinate (**10d**) (55 mg, 0.26 mmol) were converted to the title compound **11d** (48.5 mg, 62%) as a yellow solid: mp 230–231 °C; IR (neat): ν_{max}/cm^{-1} 1726 (C=O), 1289 (OCH_3); δ_H (400 MHz; $CDCl_3$; Me_4Si) 3.09 (3H, s), 3.65 (3H, s), 6.94 (1H, d, *J* 3.7 Hz), 6.98 (1H, d, *J* 3.9 Hz), 7.44 (1H, dd, *J* 7.8, 4.9 Hz), 7.68 (1H, d, *J* 3.9 Hz), 7.70 (1H, d, *J* 3.7 Hz), 8.34 (1H, dd, *J* 7.8, 2.0 Hz), 8.40 (1H, s), 8.75 (1H, dd, *J* 4.9, 2.0 Hz); δ_C (100 MHz; $CDCl_3$) 40.5, 52.7, 103.7, 105.3, 110.2, 121.4, 121.8, 125.9, 127.3,

129.2, 132.6, 140.8, 145.8, 146.9, 149.6, 151.9, 166.0; HRMS (FAB) *m/z* Calc. for $C_{17}H_{15}N_4O_4S$ (MH^+) 371.0809, found 371.0811.

6-{Dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl}nicotinic acid (5b). By use of the procedure for the synthesis of **5a**, **11b** (48 mg, 0.13 mmol) was converted to the title compound **5b** as an NH_3 salt (20.4 mg, 53%). Pale yellow solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1596 (C=O); δ_H (500 MHz; DMSO-d_6; Me_4Si) 6.56 (1H, s), 6.82 (1H, d, *J* 3.4 Hz), 7.66 (1H, s), 7.81 (2H, d, *J* 8.0 Hz), 7.98 (1H, d, *J* 3.4 Hz), 8.05 (1H, s), 8.16 (2H, d, *J* 8.0 Hz), 11.03 (1H, s); δ_C (125 MHz; DMSO-d_6) 100.5, 104.9, 107.4, 111.8, 122.6, 125.5, 126.8, 128.3, 129.8, 139.6, 144.5, 144.9, 149.8, 154.7, 166.0; HRMS (FAB) *m/z* Calc. for $C_{15}H_9N_4O_2$ $[M - H]^-$ 277.0731, found 277.0747.

2-{Dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl}isonicotinic acid (5c). By use of the procedure for the synthesis of **5a**, **11c** (69 mg, 0.19 mmol) was converted to the title compound **5c** as an NH_3 salt (17.8 mg, 32%). Pale yellow solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1592 (C=O); δ_H (500 MHz; DMSO-d_6; Me_4Si) 6.53–6.55 (1H, br m), 6.81 (1H, d, *J* 4.0 Hz), 7.64 (1H, d, *J* 5.0 Hz), 7.66–7.67 (1H, m), 8.09 (1H, s), 8.35 (1H, d, *J* 4.0 Hz), 8.63 (1H, d, *J* 5.0 Hz), 8.88 (1H, s), 11.23 (1H, s); δ_C (125 MHz; DMSO-d_6) 100.4, 103.7, 105.8, 112.1, 119.0, 125.5, 126.7, 128.5, 129.6, 144.2, 144.7, 147.3, 148.5, 152.7, 166.2; HRMS (FAB) *m/z* Calc. for $C_{15}H_9N_4O_2$ $[M - H]^-$ 277.0731, found 277.0722.

2-{Dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl}nicotinic acid (5d). By use of the procedure for the synthesis of **5a**, **11d** (48 mg, 0.13 mmol) was converted to the title compound **5d** as an NH_3 salt (6.0 mg, 16%). Yellow solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1591 (C=O); δ_H (500 MHz; DMSO-d_6; Me_4Si) 6.47–6.49 (1H, br m), 6.59 (1H, d, *J* 3.4 Hz), 7.30 (1H, dd, *J* 7.2, 4.6 Hz), 7.56–7.57 (1H, m), 7.91–7.93 (2H, m), 8.19 (1H, s), 8.46 (1H, dd, *J* 4.6, 1.7 Hz), 11.03 (1H, s); δ_C (125 MHz; DMSO-d_6) 100.2, 101.9, 102.9, 105.0, 120.8, 126.2, 126.4, 128.9, 131.2, 138.0, 143.7,

144.2, 146.7, 147.7, 168.3; HRMS (FAB) *m/z* Calc. for $C_{15}H_9N_4O_2$ $[M - H]^-$ 277.0731, found 277.0722.

6-{Dipyrrolo[3,2-*b*:2',3'-*e*]pyridin-1(7*H*)-yl)picolinic acid (5e). By use of the procedure for the synthesis of **11a**, **9** (50 mg, 0.22 mmol) and methyl 6-bromopicolinate (**10e**) (55 mg, 0.26 mmol) were converted to the title compound **11e** (21.2 mg, containing inseparable impurities, <27%) as a pale yellow solid. This crude product was used for the next reaction without further purifications. δ_H (400 MHz; $CDCl_3$; Me_4Si) 3.20 (3H, s), 4.13 (3H, s), 6.97-7.00 (2H, m), 7.61–7.65 (1H, m), 7.74 (1H, d, *J* 3.9 Hz), 8.00–8.02 (3H, m), 9.82 (1H, s); HRMS (FAB) *m/z* Calc. for $C_{17}H_{15}N_4O_4S$ (MH^+) 371.0809, found 371.0803.

By use of the procedure for the synthesis of **5a**, **11e** (21 mg, crude) was converted to the title compound **5e** as an NH_3 salt (5.6 mg, 9% from **9**). Pale yellow solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1592 (C=O); δ_H (500 MHz; DMSO-d_6; Me_4Si) 6.48–6.50 (1H, br m), 6.78 (1H, d, *J* 3.4 Hz), 7.60–7.61 (1H, m), 7.72–7.73 (2H, m), 7.90–7.93 (1H, m), 8.32 (1H, d, *J* 3.4 Hz), 9.34 (1H, s), 11.59 (1H, s); δ_C (125 MHz; DMSO-d_6) 100.20, 104.96, 105.39, 111.97, 118.88, 125.76, 126.79, 128.14, 128.20, 129.52, 138.71, 144.27, 144.60, 151.53, 168.01; HRMS (FAB) *m/z* Calc. for $C_{15}H_9N_4O_2$ $[M - H]^-$ 277.0731, found 277.0748.

Methyl 4-ethynyl-3-(phenylethynyl)benzoate (17a). By use of the procedure for the synthesis of **17c**, **15** (319 mg, 1.03 mmol) and ethynylbenzene (**16a**) (0.124 mL, 1.13 mmol) were converted to the title compound **17a** (168 mg, 63%) as a pale brown solid: mp 71–72 °C; IR (neat): ν_{max}/cm^{-1} 3262 (C≡CH), 2222 (C≡C), 1715 (C=O), 1254 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si) 3.51 (1H, s), 3.94 (3H, s), 7.36–7.37 (3H, m), 7.57–7.60 (3H, m), 7.93 (1H, dd, *J* 8.0, 1.7 Hz), 8.21 (1H, d, *J* 1.7 Hz); δ_C (125 MHz; $CDCl_3$) 52.4, 81.6, 83.9, 87.0, 94.4, 122.8, 126.7, 128.4 (2C), 128.6, 128.7, 128.8, 130.1, 131.8 (2C), 132.6, 132.8, 165.9; HRMS (FAB) *m/z* Calc. for $C_{18}H_{13}O_2$ (MH^+) 261.0910, found 261.0901.

Methyl 4-ethynyl-3-[(4-methoxyphenyl)ethynyl]benzoate (17b). By use of the procedure for the synthesis of **17c**, **15** (636 mg, 2.00 mmol) and 1-ethynyl-4-methoxybenzene (**16b**) (0.30 mL, 2.20 mmol) were converted to the title compound **17b** (115 mg, 55%) as colourless crystals: mp 133–134 °C; IR (neat): ν_{max}/cm^{-1} 3252 (C≡CH), 2209 (C≡C), 1729 (C=O), 1286, 1247 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si) 3.50 (1H, s), 3.83 (3H, s), 3.93 (3H, s), 6.88–6.90 (2H, m), 7.50–7.52 (2H, m), 7.58 (1H, d, *J* 8.0 Hz), 7.90 (1H, dd, *J* 8.0, 1.7 Hz), 8.18 (1H, d, *J* 1.7 Hz); δ_C (125 MHz; $CDCl_3$) 52.4, 55.3, 81.7, 83.7, 85.9, 94.6, 114.1 (2C), 114.9, 127.0, 128.2, 128.4, 130.1, 132.6, 132.6, 133.3 (2C), 160.1, 165.9; HRMS (FAB) *m/z* Calc. for $C_{19}H_{14}O_3$ (M^+) 290.0943, found 290.0942.

Methyl 4-ethynyl-3-(pent-1-ynyl)benzoate (17d). By use of the procedure for the synthesis of **17c**, **15** (358 mg, 1.15 mmol) and 1-pentyne (**16d**) (0.10 mL, 1.26 mmol) were converted to the title compound **17d** (50.5 mg, 19%) as a white solid: mp 69–70 °C; IR (neat): ν_{max}/cm^{-1} 3240 (C≡CH), 2230 (C≡C), 1709 (C=O), 1228 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si) 1.09 (3H, t, *J* 7.4 Hz), 1.63–1.70 (2H, m), 2.46 (2H, t, *J* 6.9 Hz), 3.42 (1H, s), 3.91 (3H, s), 7.53 (1H, d, *J* 8.0 Hz), 7.86 (1H, dd, *J* 8.0, 1.7 Hz), 8.08 (1H, d, *J* 1.7 Hz); δ_C (125 MHz; $CDCl_3$) 13.5, 21.6, 22.0, 52.3, 78.5, 81.8, 83.2, 96.0, 127.5, 127.9, 128.6, 130.0, 132.5, 133.0, 166.0; HRMS (FAB) *m/z* Calc. for $C_{15}H_{15}O_2$ (MH^+) 227.1067, found 227.1066.

Dimethyl 2-(4-methoxybenzyl)-4-phenyl-2,3-dihydro-1*H*-benzo[*g*]indazole-1,7-dicarboxylate (20a). By use of the procedure for the synthesis of **20b**, **17a** (50 mg, 0.192 mmol) was converted to the title compound **20a** [20.0 mg, 22% (51% rsm)] as a yellow oil: IR (neat): ν_{max}/cm^{-1} 1719, 1610 (C=O), 1264 (OCH_3); δ_H (400 MHz; $CDCl_3$; Me_4Si, 50 °C) 3.74 (3H, s), 3.78–3.81 (5H, br m), 3.99 (3H, s), 4.34 (2H, br s), 6.75 (2H, d, *J* 8.5 Hz), 7.19 (2H, d, *J* 8.5 Hz), 7.39–7.48 (5H, m), 7.84 (1H, s), 8.03–8.10 (2H, m), 8.64 (1H, s); δ_C (100 MHz; $CDCl_3$) 52.3, 53.6, 55.2, 57.6, 61.7, 113.5 (2C),

125.1, 125.6, 126.0, 127.5, 127.6, 127.9, 128.1 (2C), 128.2, 128.9 (2C), 130.0, 131.0 (2C), 131.3, 133.6, 136.5, 137.4, 139.1, 157.0, 159.1, 167.1; HRMS (FAB) *m/z* Calc. for $C_{29}H_{26}N_2O_5$ (M^+) 482.1842, found 482.1841.

Dimethyl 2-(4-methoxybenzyl)-4-(thiophen-2-yl)-2,3-dihydro-1*H*-benzo[*g*]indazole-1,7-dicarboxylate (20d). By use of the procedure for the synthesis of **20b**, **17c** (30 mg, 0.113 mmol) was converted to the title compound **20d** [13.0 mg, 24% (50% rsm)] as a brown oil: IR (neat): ν_{max}/cm^{-1} 1718, 1598 (C=O), 1262 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si, 50 °C) 3.76 (3H, s), 3.80 (3H, s), 3.83 (2H, s), 3.99 (3H, s), 4.48 (2H, br s), 6.78 (2H, d, *J* 8.6 Hz), 7.12–7.14 (1H, m), 7.19 (1H, dd, *J* 3.4, 1.1 Hz), 7.23 (2H, d, *J* 8.6 Hz), 7.38 (1H, dd, *J* 5.2, 1.1 Hz), 7.98–8.01 (2H, m), 8.05–8.07 (1H, m), 8.63 (1H, s); δ_C (100 MHz; $CDCl_3$) 52.3, 53.6, 55.2, 57.9, 61.9, 113.5 (2C), 125.1, 125.7, 126.0, 126.1 (2C), 126.4, 127.8, 128.1, 128.2, 129.0, 129.1, 131.1 (2C), 131.2, 133.5, 137.7, 141.3, 157.0, 159.2, 167.0; HRMS (FAB) *m/z* Calc. for $C_{27}H_{25}N_2O_5S$ (MH^+) 489.1479, found 489.1476.

Dimethyl 2-(4-methoxybenzyl)-4-propyl-2,3-dihydro-1*H*-benzo[*g*]indazole-1,7-dicarboxylate (20f). By use of the procedure for the synthesis of **20b**, **17d** (30 mg, 0.133 mmol) was converted to the title compound **20f** (15.2 mg, 25%) as a yellow oil. In this case, the reaction was carried out with IPrAuCl (8.3 mg, 13.3 μmol), and AgOTf (3.4 mg, 13.3 μmol), and after portionwise addition of a hydrazine **18** (11 mg, 0.053 mmol × 3) and paraformaldehyde **19a** (3.0 mg, 0.11 mmol as HCHO × 3) at 35 °C over 6 h, the mixture was stirred at 80 °C for 3 h: IR (neat): ν_{max}/cm^{-1} 1719, 1610 (C=O), 1255 (OCH_3); δ_H (400 MHz; $CDCl_3$; Me_4Si, 50 °C) 0.98 (3H, t, *J* 7.3 Hz), 1.63–1.73 (2H, m), 2.65 (2H, t, *J* 7.6 Hz), 3.78 (3H, s), 3.79 (3H, s), 3.83 (2H, s), 3.97 (3H, s), 4.24 (2H, br s), 6.81 (2H, d, *J* 8.5 Hz), 7.26 (2H, d, *J* 8.5 Hz), 7.59 (1H, s), 8.00–8.01 (2H, m), 8.56 (1H, s); δ_C (100 MHz; $CDCl_3$) 13.8, 23.1, 35.4, 52.2, 53.5, 55.2, 56.3, 61.9, 113.6 (2C), 124.2, 125.3, 125.5, 127.00, 127.04, 128.4,

130.81, 130.84, 131.1 (2C), 133.6, 136.3, 136.4, 156.9, 159.2, 167.2; HRMS (FAB) *m/z* Calc. for $C_{26}H_{29}N_2O_5$ (MH^+) 449.2071, found 449.2075.

Dimethyl 3-isopropyl-2-(4-methoxybenzyl)-4-(4-methoxyphenyl)-2,3-dihydro-1*H*-benzo[*g*]indazole-1,7-di carboxylate (20c). By use of the procedure for the synthesis of **20e**, **17b** (29 mg, 0.10 mmol) was converted to the title compound **20c** (40.0 mg, 72%) as a pale yellow solid: mp 209–210 °C; IR (neat): ν_{max}/cm^{-1} 1717, 1609 (C=O), 1250 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si) 0.48 (3H, d, *J* 6.9 Hz), 0.50 (3H, d, *J* 6.9 Hz), 1.31–1.35 (1H, m), 3.79 (3H, s), 3.81 (1H, d, *J* 12.6 Hz), 3.83 (3H, s), 3.87 (3H, s), 3.98 (3H, s), 4.12 (1H, d, *J* 12.6 Hz), 4.16 (1H, d, *J* 4.0 Hz), 6.85 (2H, d, *J* 8.6 Hz), 6.97 (2H, d, *J* 8.6 Hz), 7.33–7.36 (4H, m), 7.70 (1H, s), 8.01–8.06 (2H, m), 8.61 (1H, s); δ_C (125 MHz; $CDCl_3$) 16.2, 20.1, 31.0, 52.2, 53.3, 55.2, 55.3, 62.7, 72.4, 113.4 (2C), 114.2 (2C), 124.5, 125.3, 125.9, 127.3, 128.0, 128.7, 129.2 (2C), 131.1, 131.5 (2C), 131.9, 132.2, 133.6, 136.8, 137.1, 157.2, 159.1, 159.2, 167.2; HRMS (FAB) *m/z* Calc. for $C_{33}H_{35}N_2O_6$ (MH^+) 555.2490, found 555.2493.

4-(4-Methoxyphenyl)-1*H*-benzo[*g*]indazole-7-carboxylic acid (6b). By use of the procedure for the synthesis of **6a**, **20b** (15 mg, 0.029 mmol) was converted to the title compound **6b** (4.7 mg, 50%) as a white solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1707 (C=O), 1248 (OCH_3); δ_H (500 MHz; DMSO-d_6; Me_4Si) 3.86 (3H, s), 7.15 (2H, d, *J* 8.6 Hz), 7.76 (1H, s), 7.80 (2H, d, *J* 8.6 Hz), 8.14 (1H, d, *J* 8.6 Hz), 8.31 (1H, s), 8.55 (1H, d, *J* 8.6 Hz), 8.71 (1H, s); δ_C (125 MHz; DMSO-d_6) 55.2, 114.5 (2C), 119.1, 120.1, 120.9, 122.0 (2C), 125.8, 128.7, 129.4 (2C), 130.7, 131.3, 131.5, 133.1 (2C), 159.2, 167.4; HRMS (FAB) *m/z* Calc. for $C_{19}H_{13}N_2O_3$ $[M - H]^-$ 317.0932, found 317.0920.

4-(Thiophen-2-yl)-1*H*-benzo[*g*]indazole-7-carboxylic acid (6d). By use of the procedure for the synthesis of **6a**, **20d** (27 mg, 0.055 mmol) was converted to the title compound **6d** (6.2 mg, 38%) as a white solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1690 (C=O); δ_H (500 MHz; DMSO-d_6; Me_4Si)

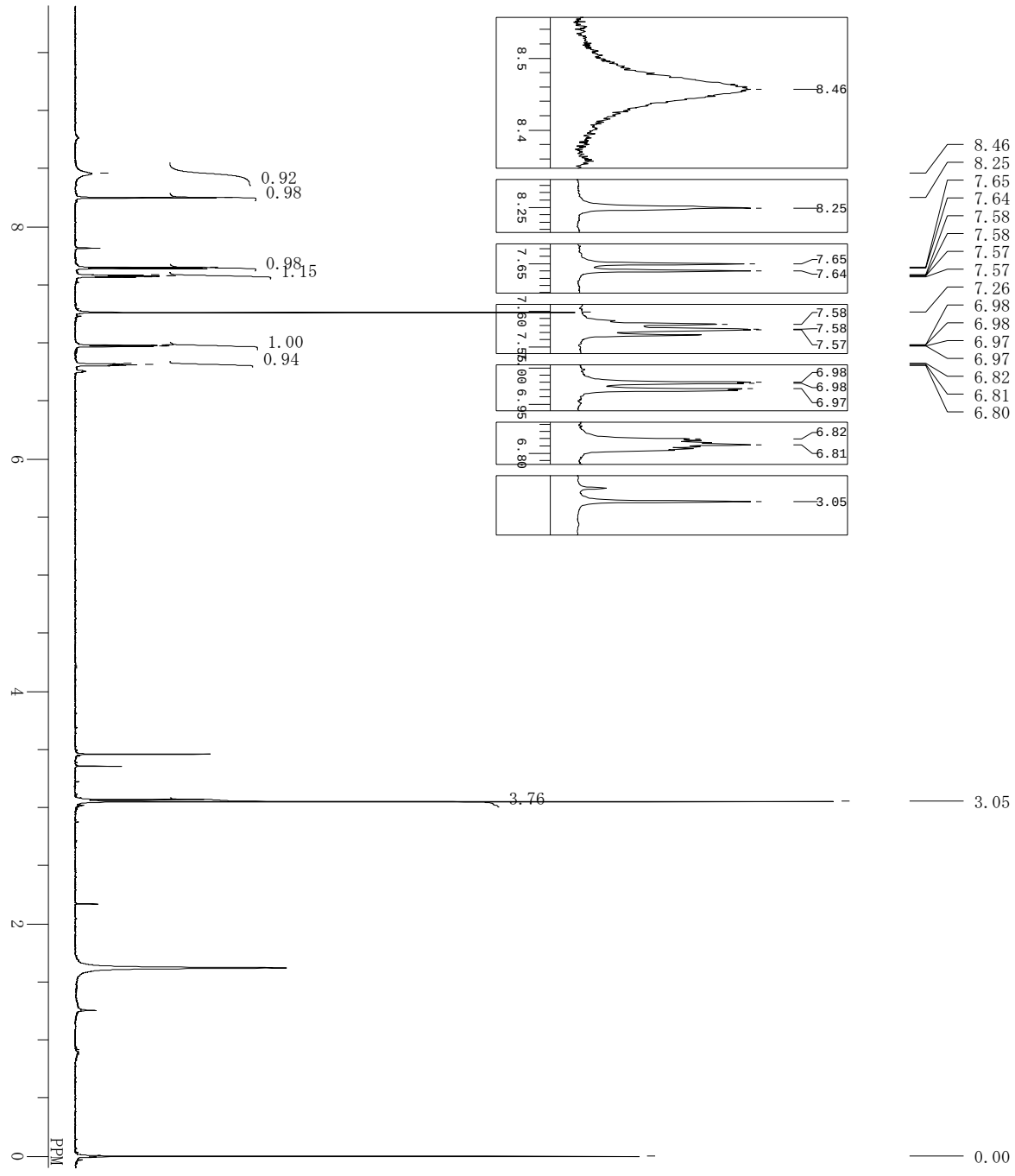
7.28–7.29 (1H, m), 7.73 (1H, d, *J* 5.2 Hz), 7.82 (1H, d, *J* 3.4 Hz), 7.98 (1H, s), 8.15 (1H, d, *J* 8.6 Hz), 8.54 (1H, d, *J* 8.6 Hz), 8.57 (1H, br s), 8.73 (1H, s); δ_C (125 MHz; DMSO-d_6) 118.0, 119.8, 121.2, 122.0, 122.3, 126.1, 126.18, 126.20, 126.6, 128.5, 129.0, 130.8, 131.28, 131.32, 141.2, 167.3; HRMS (FAB) *m/z* Calc. for $C_{16}H_9N_2O_2S$ $[M - H]^-$ 293.0390, found 293.0386.

4-Propyl-1*H*-benzo[*g*]indazole-7-carboxylic acid (6f). By use of the procedure for the synthesis of **6a**, **20f** (15 mg, 0.033 mmol) was converted to the title compound **6f** (4.0 mg, 47%) as a white solid: mp >300 °C; IR (neat): ν_{max}/cm^{-1} 1689 (C=O); δ_H (500 MHz, DMSO-d_6; Me_4Si) 0.96 (3H, t, *J* 7.2 Hz), 1.75–1.82 (2H, m), 2.92 (2H, t, *J* 7.4 Hz), 7.31 (1H, s), 8.05 (1H, d, *J* 8.4 Hz), 8.21 (1H, s), 8.34 (1H, d, *J* 8.4 Hz), 8.44 (1H, s); δ_C (125 MHz; DMSO-d_6) 13.9, 22.9, 35.0, 120.0 (2C), 120.3, 120.9 (2C), 126.1, 129.1, 131.6, 132.7, 133.4, 135.0, 168.4; HRMS (FAB) *m/z* Calc. for $C_{15}H_{13}N_2O_2$ $[M - H]^-$ 253.0983, found 253.0988.

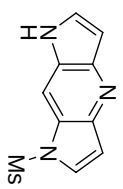
3-Isopropyl-4-(4-methoxyphenyl)-1*H*-benzo[*g*]indazole-7-carboxylic acid (6c). By use of the procedure for the synthesis of **21e**, **20c** (30 mg, 0.054 mmol) was converted to the title compound **21c** (23.4 mg, 100%) as white solid: mp 113–114 °C; IR (neat): ν_{max}/cm^{-1} 1755, 1717 (C=O), 1267, 1247 (OCH_3); δ_H (500 MHz; $CDCl_3$; Me_4Si) 1.14 (6H, d, *J* 6.9 Hz), 2.86–2.91 (1H, m), 3.91 (3H, s), 4.00 (3H, s), 4.20 (3H, s), 7.01 (2H, d, *J* 8.6 Hz), 7.38 (2H, d, *J* 8.6 Hz), 7.62 (1H, s), 8.17 (1H, dd, *J* 9.2, 1.7 Hz), 8.65 (1H, d, *J* 1.7 Hz), 9.11 (1H, d, *J* 9.2 Hz); δ_C (125 MHz; $CDCl_3$) 21.8 (2C), 27.0, 52.3, 55.1, 55.3, 113.5 (2C), 122.9, 123.1, 125.3, 126.2, 128.2, 128.3, 130.3 (2C), 131.0, 131.7, 133.1, 134.6, 138.9, 152.6, 158.0, 159.5, 166.9; HRMS (FAB) *m/z* Calc. for $C_{25}H_{25}N_2O_5$ (MH^+) 433.1758, found 433.1770.

By use of the procedure for the synthesis of **6e**, **21c** (18 mg, 0.042 mmol) was converted to the title compound **6c** (11.6 mg, 77%) as white solid: mp >300 °C IR (neat): ν_{max}/cm^{-1} 1688 (C=O), 1245 (OCH_3); δ_H (500 MHz; DMSO-d_6; Me_4Si, 80 °C) 1.10 (6H, d, *J* 6.9 Hz), 2.91–2.97 (1H, m), 3.85 (3H, s), 7.06 (2H, d, *J* 8.6 Hz), 7.35 (1H, s), 7.43 (2H, d, *J* 8.6 Hz), 8.10 (1H, d, *J* 8.6 Hz), 8.53 (1H,

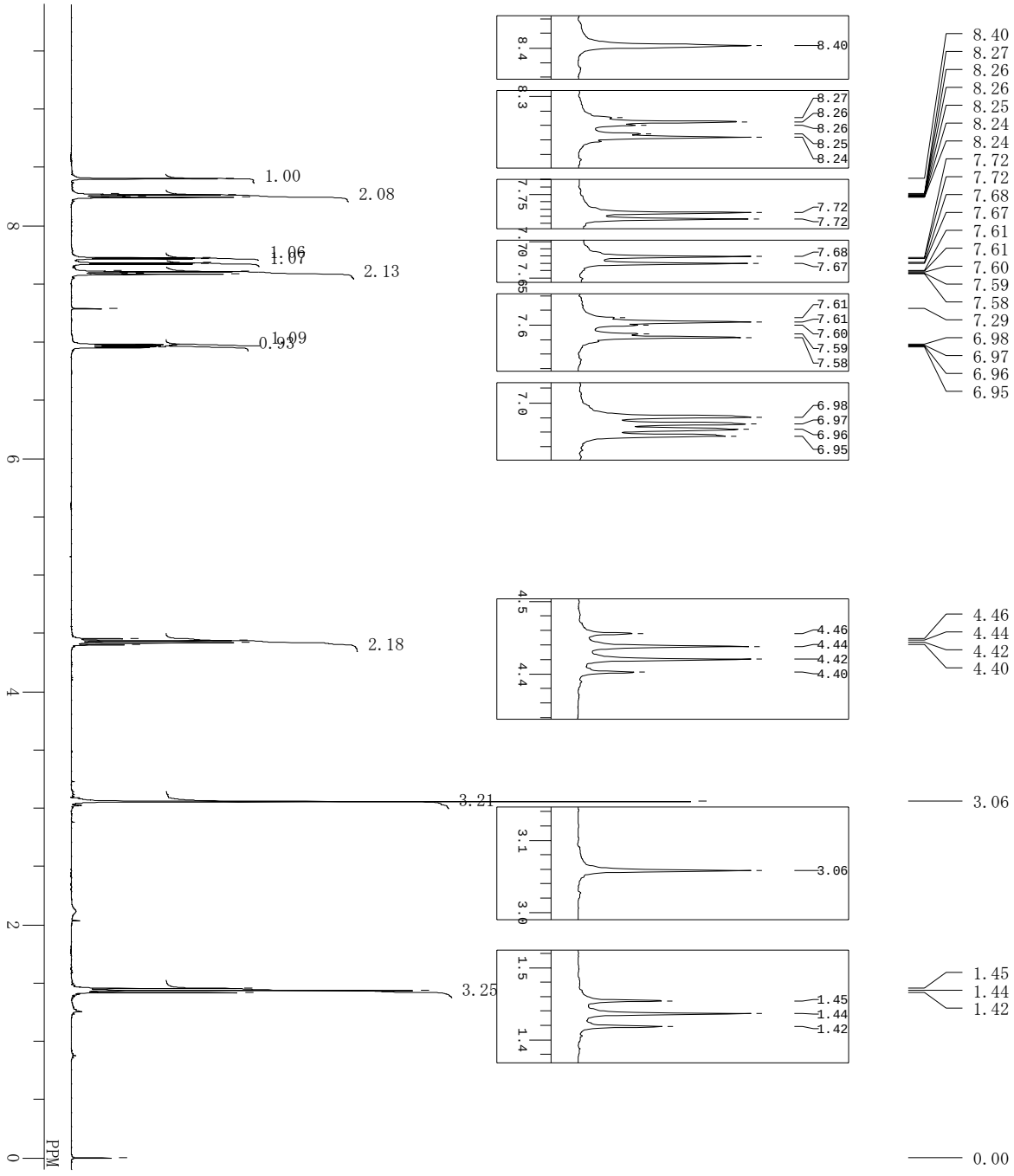
d, *J* 8.6 Hz), 8.59 (1H, s); δ_C (125 MHz; DMSO-d_6) 22.6 (2C), 26.3, 55.1, 113.5 (2C), 116.1, 121.9, 122.0, 122.3, 125.6, 128.4, 130.0 (2C), 130.4, 130.9, 132.3, 135.3, 138.7, 150.7, 158.9, 167.4; HRMS (FAB) *m/z* Calc. for $C_{22}H_{19}N_2O_3$ $[M - H]^-$ 359.1401, found 359.1391.



1H-NMR (CDCl3) δ :
3.05 (s, 3H),
6.80-6.82 (m, 1H),
6.97 (dd, J = 3.8, 0.6 Hz, 1H),
7.57-7.59 (m, 1H),
7.65 (d, J = 3.8 Hz, 1H),
8.25 (br s, 1H),
8.46 (s, 1H).

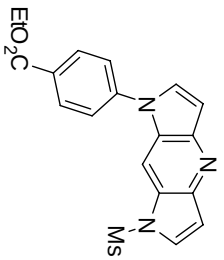


6

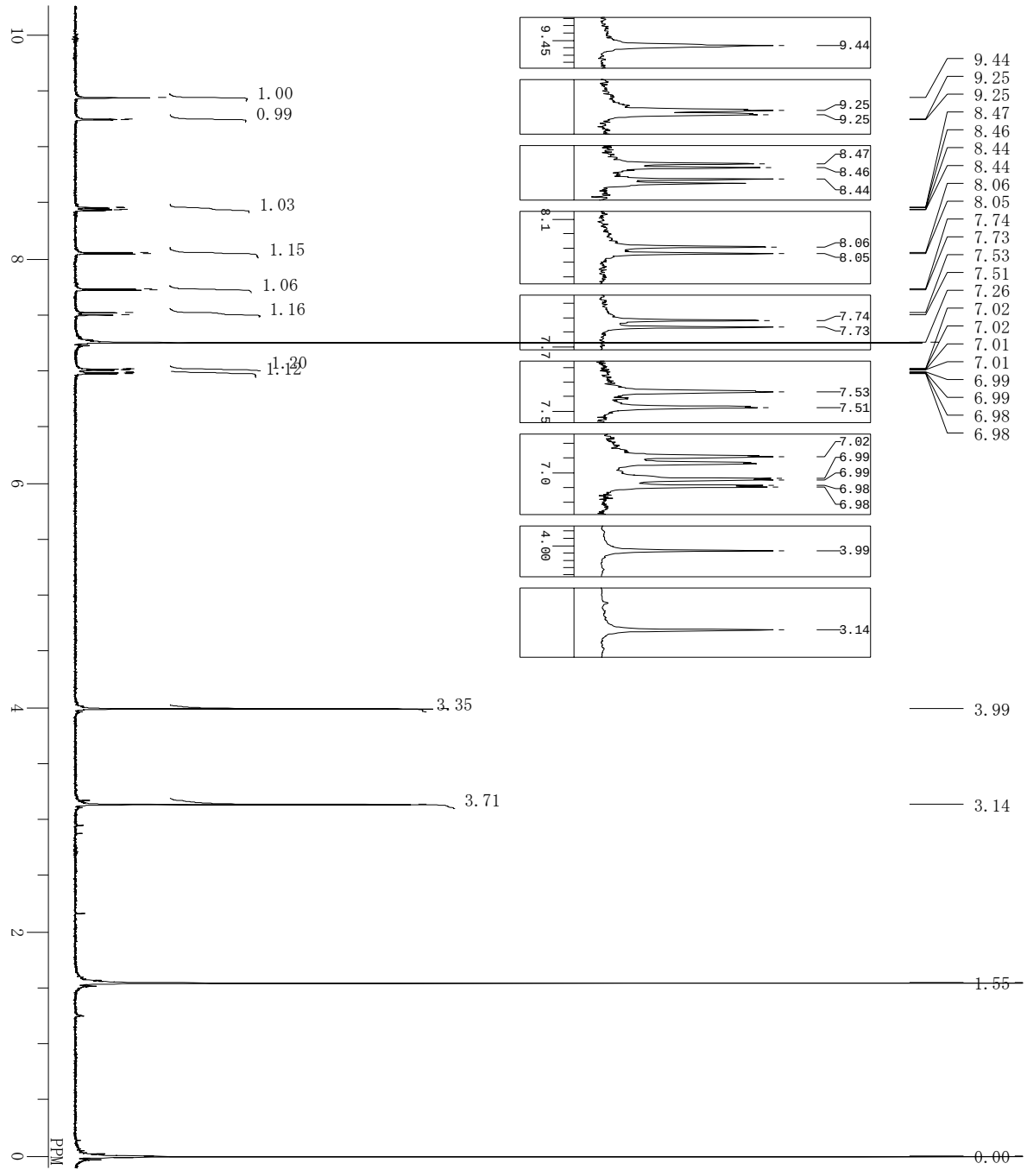


DFILE 100727-652 ys233 fine.als
COMNT
DATIM Tue Jul 27 10:03:38 2010
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 2
4.1001 sec
ACQTM 2.9000 sec
PD 5.50 usec
PW1
IRNUC 1H
CTEMP 25.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14

1H-NMR (CDCl3) δ :
1.44 (t, J = 7.1 Hz, 3H),
3.06 (s, 3H),
4.43 (q, J = 7.1 Hz, 2H),
6.96 (d, J = 3.7 Hz, 1H),
6.98 (d, J = 3.7 Hz, 1H),
7.58-7.61 (m, 2H),
7.68 (d, J = 3.7 Hz, 1H),
7.72 (d, J = 3.7 Hz, 1H),
8.24-8.27 (m, 2H),
8.40 (s, 1H).

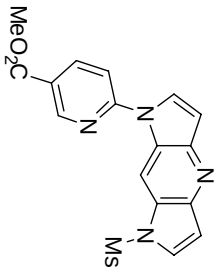


11a

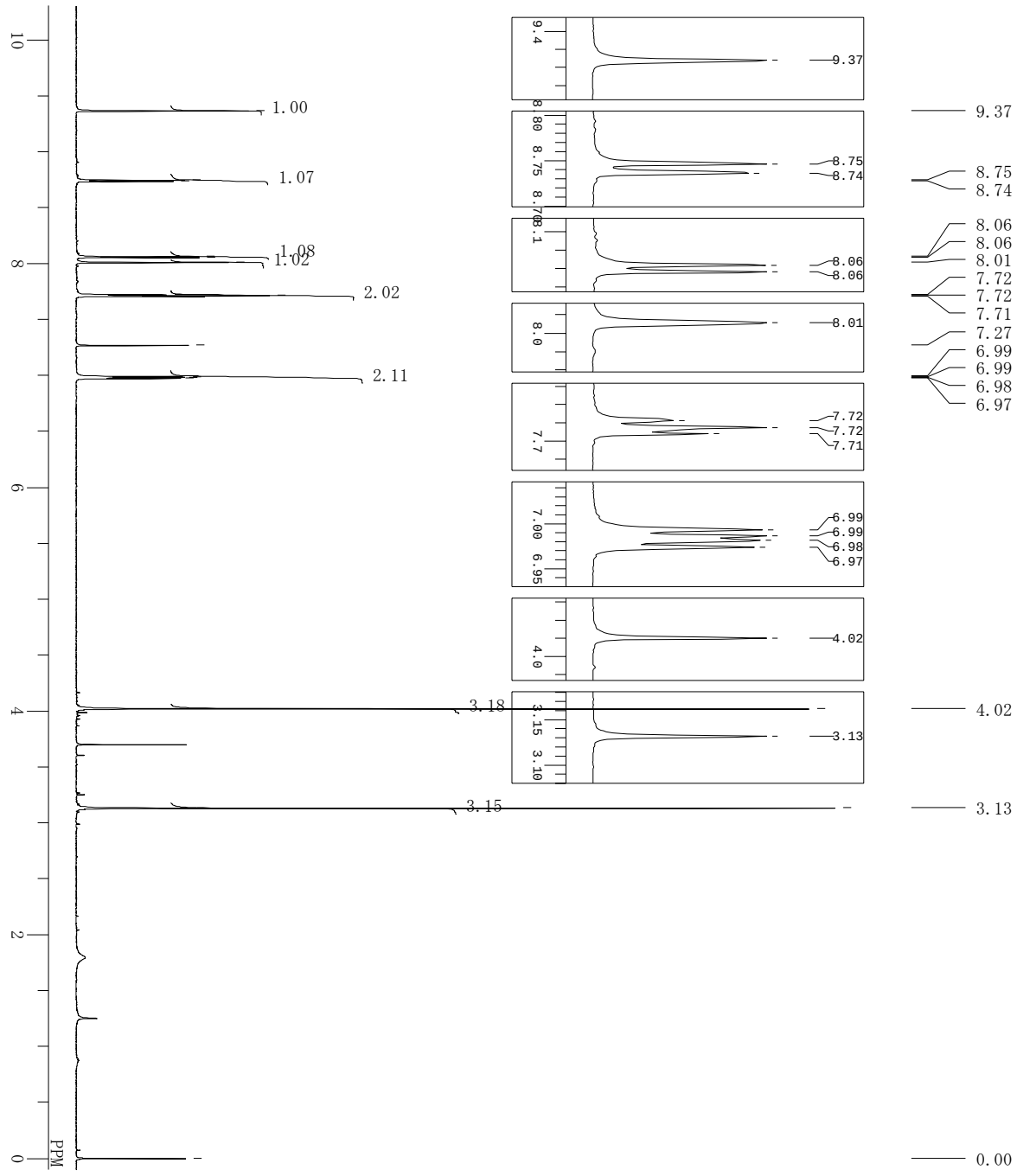


DFILE 100730-655 ys236 fine.als
COMNT
DATIM Fri Jul 30 21:07:04 2010
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 8
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

1H-NMR (CDCl3) δ :
3.14 (s, 3H),
3.99 (s, 3H),
6.99 (dd, J = 3.9, 0.7 Hz, 1H),
7.02 (dd, J = 3.9, 0.7 Hz, 1H),
7.52 (d, J = 8.5 Hz, 1H),
7.73 (d, J = 3.7 Hz, 1H),
8.06 (d, J = 3.7 Hz, 1H),
8.45 (dd, J = 8.5, 2.2 Hz, 1H),
9.25 (d, J = 2.2 Hz, 1H),
9.44 (s, 1H).

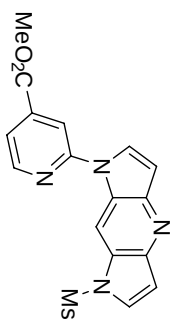


11b

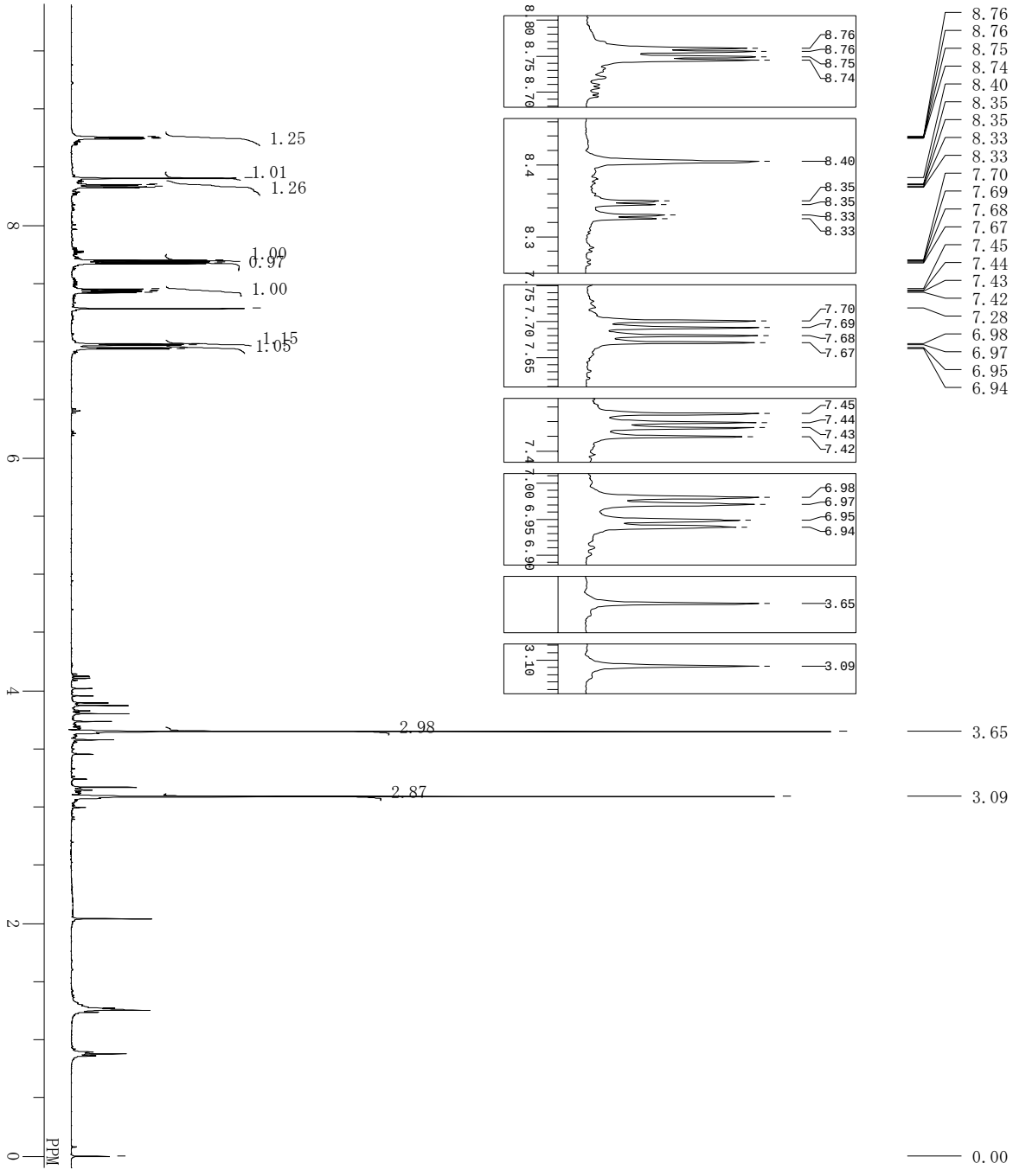


DFILE 101104-G156 ys242-fine.als
COMNT single_pulse
DATIM 04-11-2010 10:59:56
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 1
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.35 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

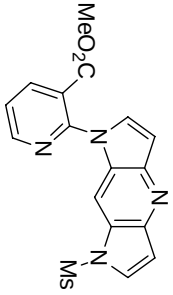
1H-NMR (CDCl3) δ :
3.13 (s, 3H),
4.02 (s, 3H),
6.97-6.99 (m, 2H),
7.71-7.72 (m, 2H),
8.01 (s, 1H),
8.06 (d, J = 3.4 Hz, 1H),
8.74 (d, J = 5.2 Hz, 1H),
9.37 (s, 1H).



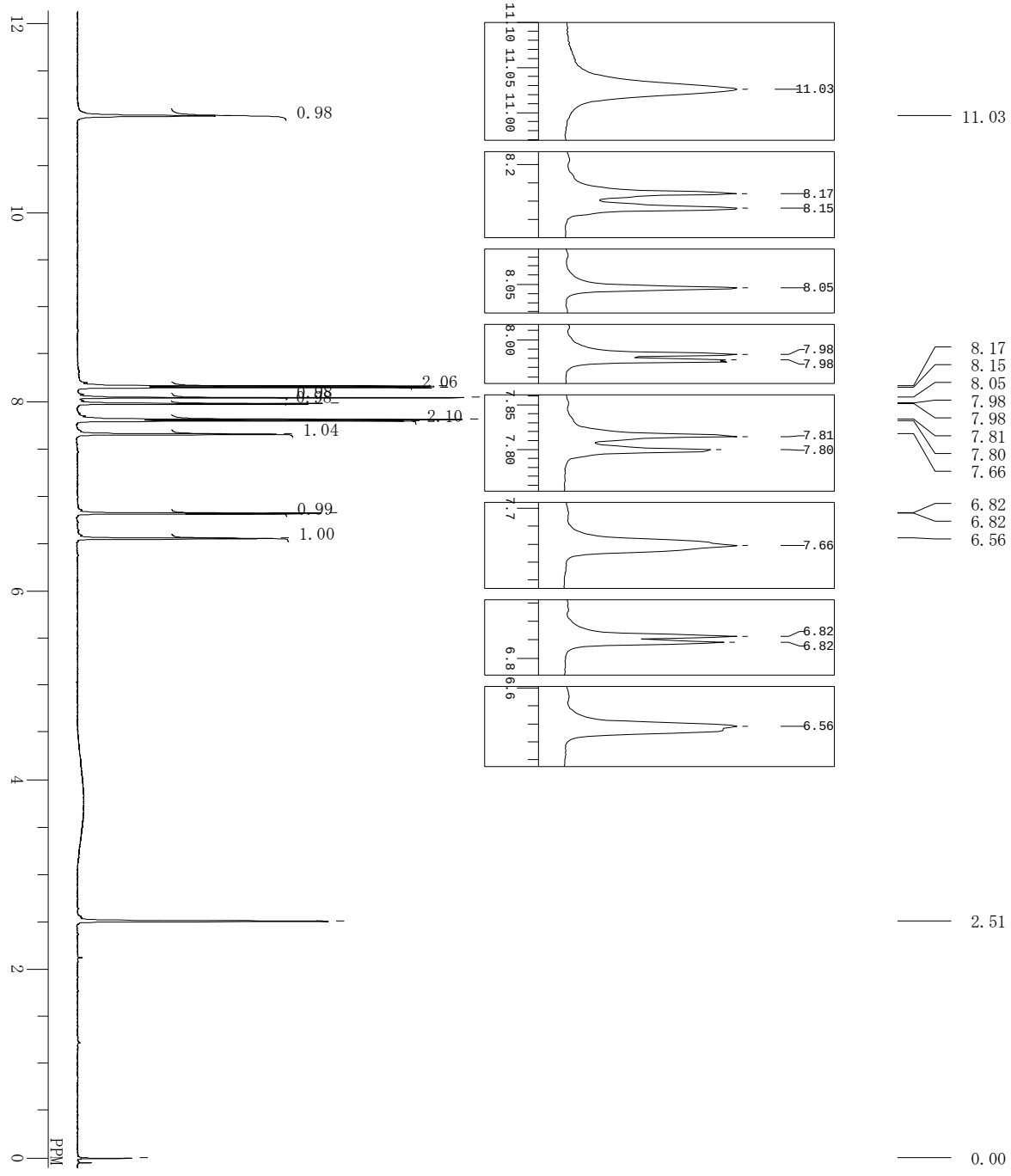
11c



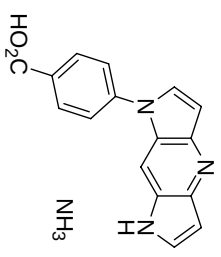
1H-NMR (CDCl3) δ :
3.09 (s, 3H),
3.65 (s, 3H),
6.94 (d, J = 3.7 Hz, 1H),
6.98 (d, J = 3.9 Hz, 1H),
7.44 (dd, J = 7.8, 4.9 Hz, 1H),
7.68 (d, J = 3.9 Hz, 1H),
7.70 (d, J = 3.7 Hz, 1H),
8.34 (dd, J = 7.8, 2.0 Hz, 1H),
8.40 (s, 1H),
8.75 (dd, J = 4.9, 2.0 Hz, 1H).



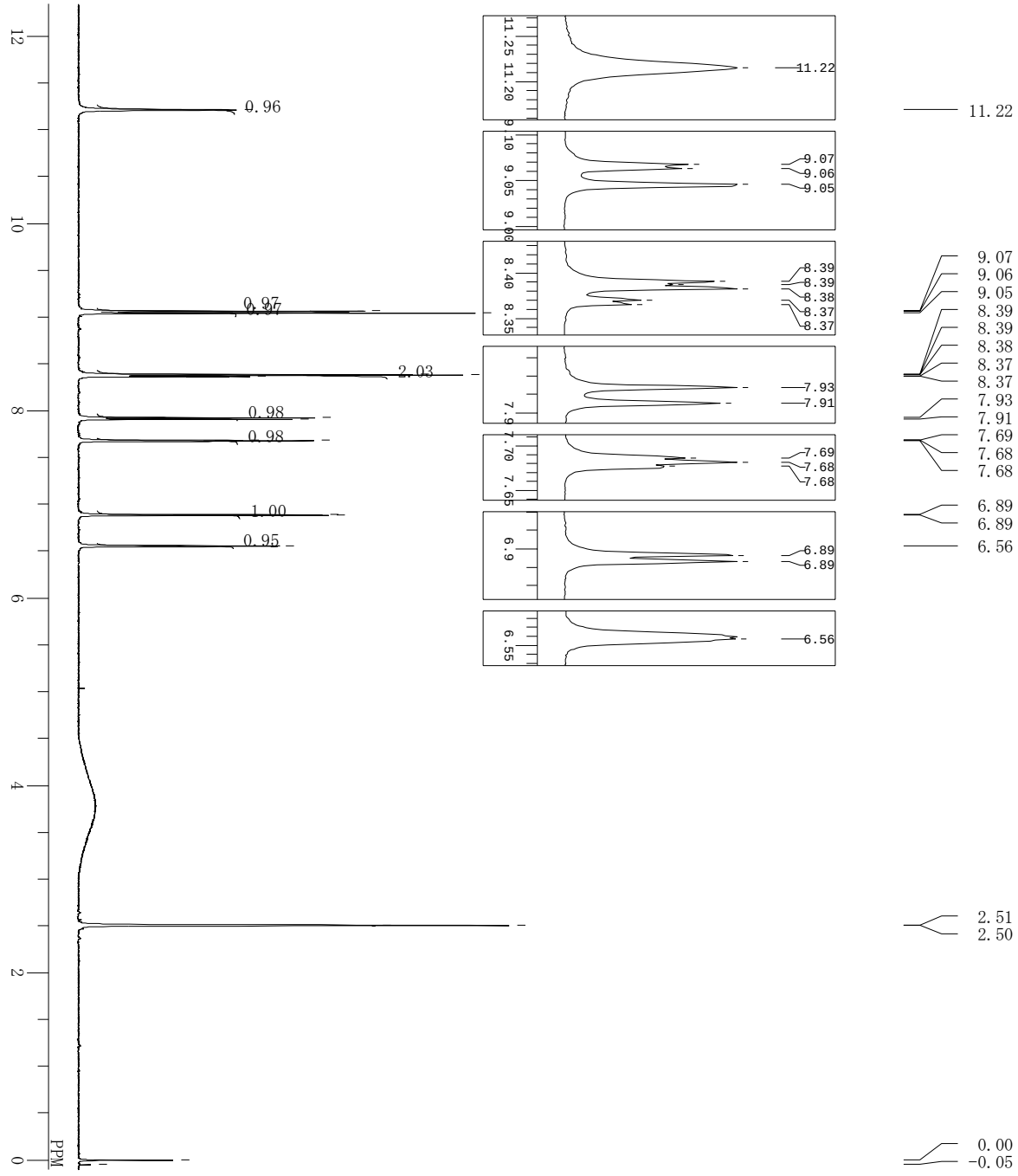
11d



1H-NMR (DMSO-D6) δ :
6.56 (s, 1H),
6.82 (d, J = 3.4 Hz, 1H),
7.66 (s, 1H),
7.81 (d, J = 8.0 Hz, 2H),
7.98 (d, J = 3.4 Hz, 1H),
8.05 (s, 1H),
8.16 (d, J = 8.0 Hz, 2H),
11.03 (s, 1H).

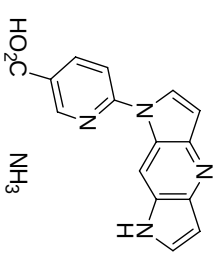


5a

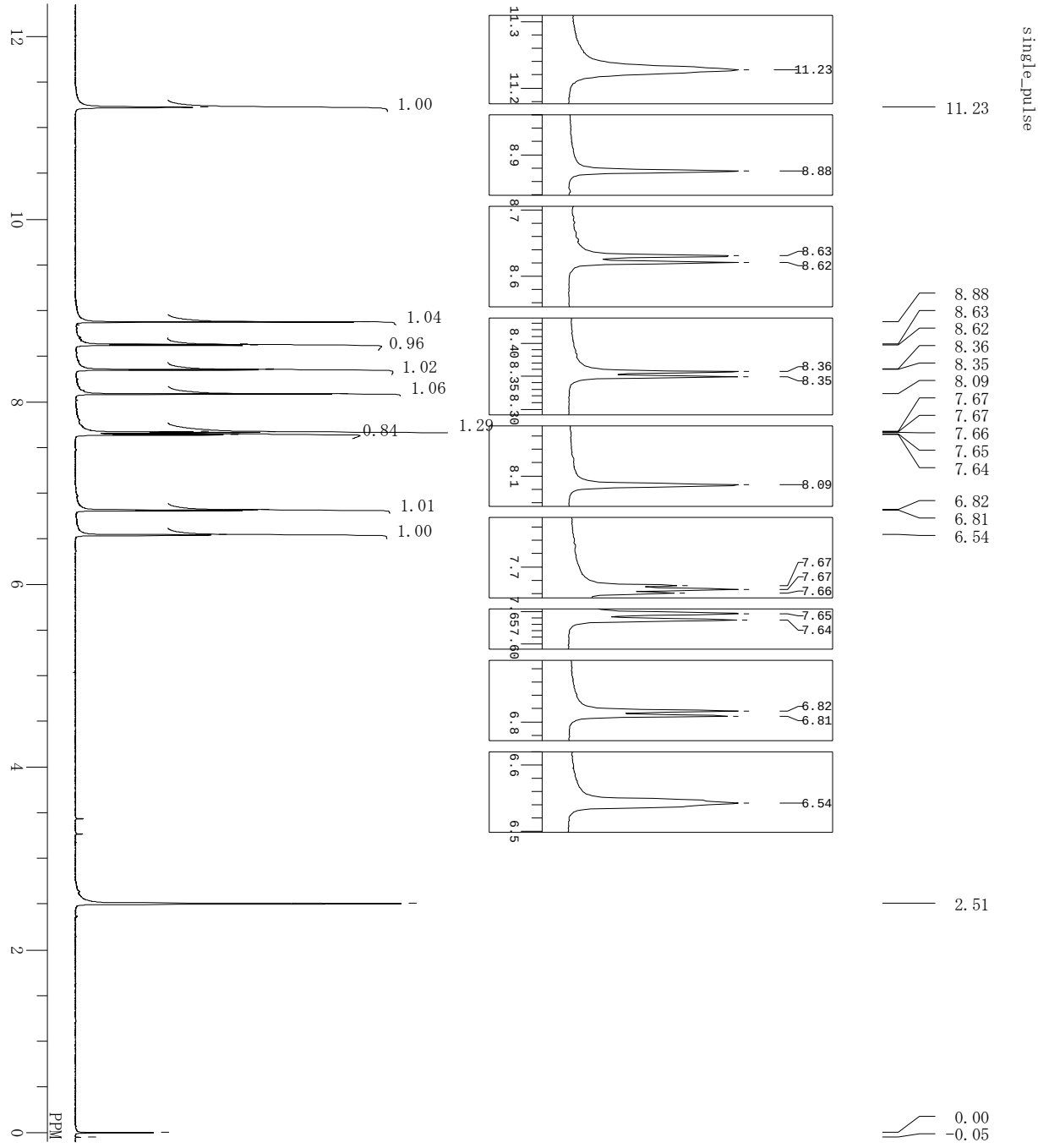



```
DFILE  100806-664 ys238-fine.als
COMNT  single_pulse
DATIM  07-08-2010 00:24:42
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ  500.16 MHz
OBSET    2.41 KHz
OBFIN    6.01 Hz
POINT  13107
FREQU  7507.39 Hz
SCANS  2
ACQTM  1.7459 sec
PD     5.0000 sec
PW1    6.35 usec
IRNUC  1H
CTEMP  29.1 c
SLVNT  DMSO
EXREF  0.00 ppm
BF     0.12 Hz
RGAIN  44
```

1H-NMR (DMSO-D6) δ :
6.55-6.57 (br m, 1H),
6.89 (d, J = 3.4 Hz, 1H),
7.68-7.69 (m, 1H),
7.92 (d, J = 8.6 Hz, 1H),
8.37-8.39 (m, 2H),
9.05 (s, 1H),
9.07 (d, J = 2.3 Hz, 1H),
11.22 (s, 1H).

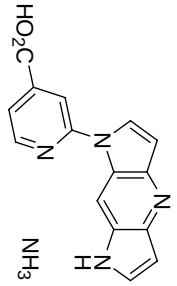


5b

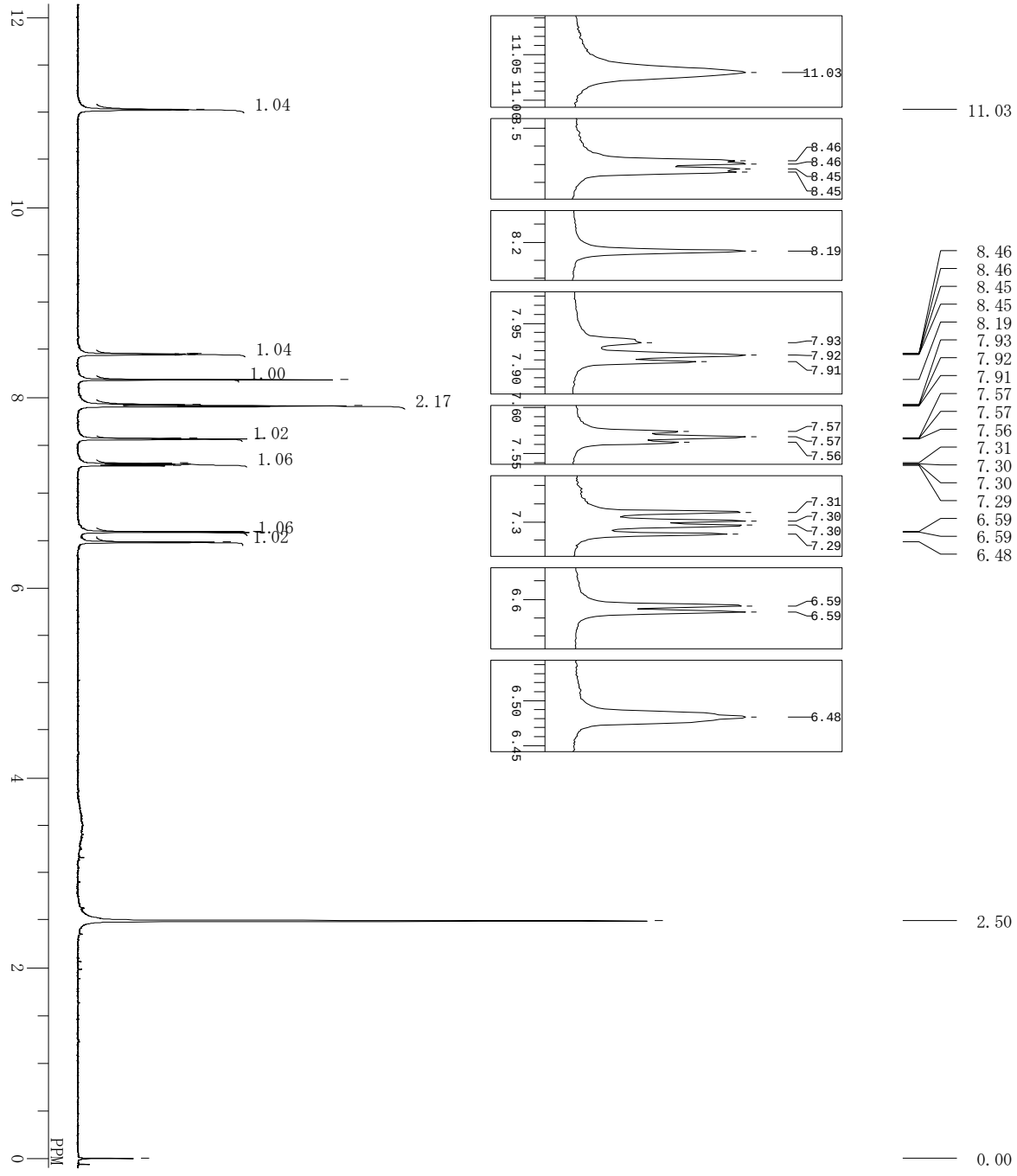


DFILE 110112-ys246NH3-1.als
COMNT single_pulse
DATIM 12-01-2011 17:31:59
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 4
1.7459 sec
ACQTM 5.0000 sec
PD 6.50 usec
PW1
IRNUC 1H
CTEMP 24.4 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

1H-NMR (DMSO-D6) δ :
6.53-6.55 (br m, 1H),
6.81 (d, J = 4.0 Hz, 1H),
7.64 (d, J = 5.0 Hz, 1H),
7.66-7.67 (m, 1H),
8.09 (s, 1H),
8.35 (d, J = 4.0 Hz, 1H),
8.63 (d, J = 5.0 Hz, 1H),
8.88 (s, 1H),
11.23 (s, 1H).

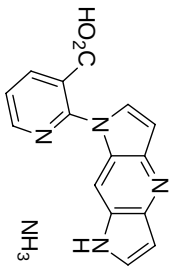


5c

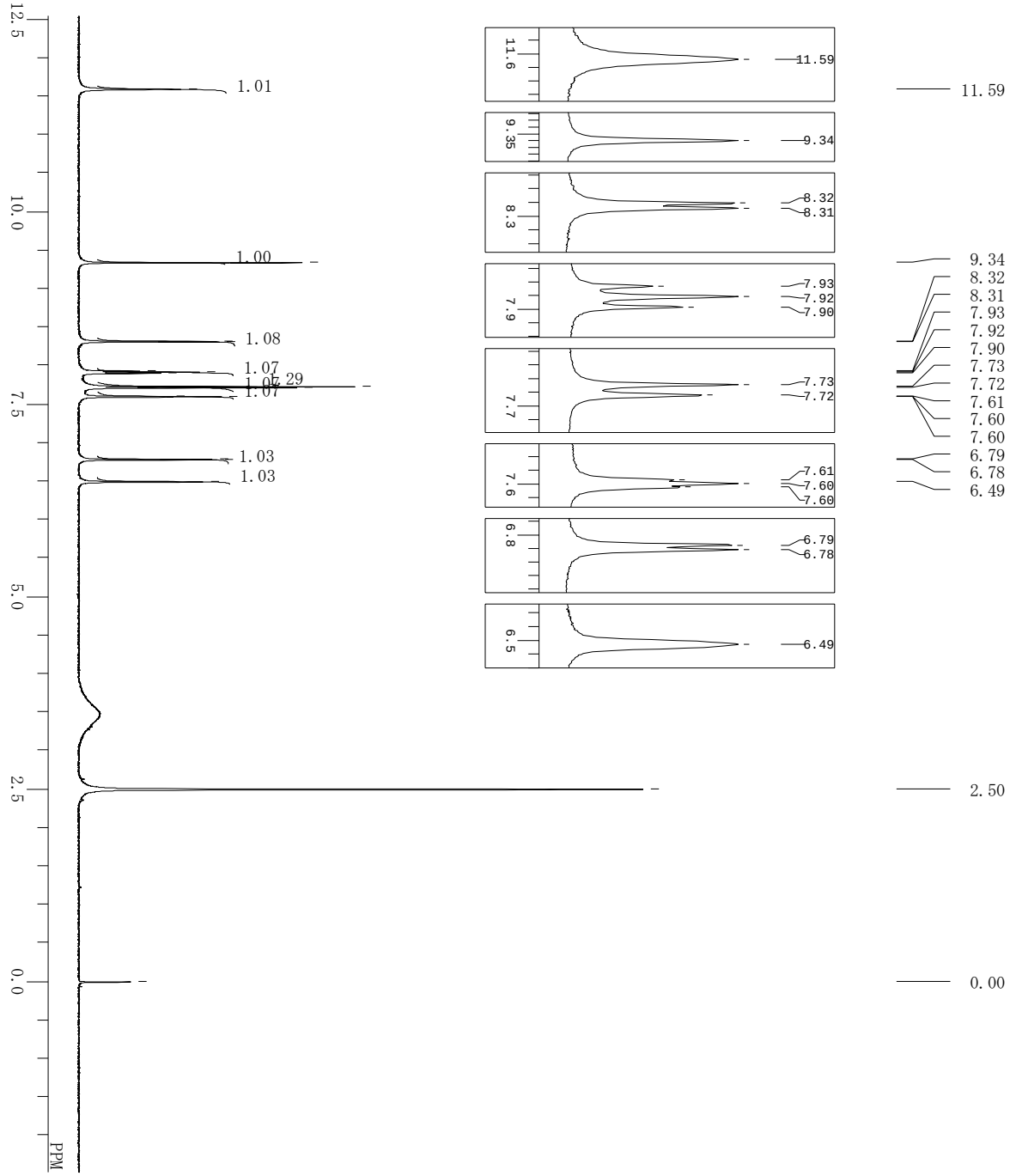



```
DFILE  110115-G162 ys247NH3-1.als
COMNT  single_pulse
DATIM  15-01-2011 15:23:26
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ  500.16 MHz
OBSET    2.41 KHz
OBFIN    6.01 Hz
POINT   13107
FREQU   7507.39 Hz
SCANS       4
          1.7459 sec
ACQTM     5.0000 sec
PD          6.50 usec
PW1
IRNUC  1H
CTEMP     25.7 c
SLVNT  DMSO
EXREF     0.00 ppm
BF        0.12 Hz
RGAIN       50
```

1H-NMR (DMSO-D6) δ :
6.47-6.49 (br m, 1H),
6.59 (d, J = 3.4 Hz, 1H),
7.30 (dd, J = 7.2, 4.6 Hz, 1H),
7.56-7.57 (m, 1H),
7.91-7.93 (m, 2H),
8.19 (s, 1H),
8.46 (dd, J = 4.6, 1.7 Hz, 1H),
11.03 (s, 1H).

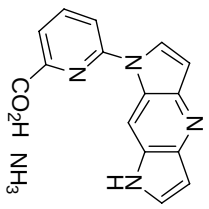


5d

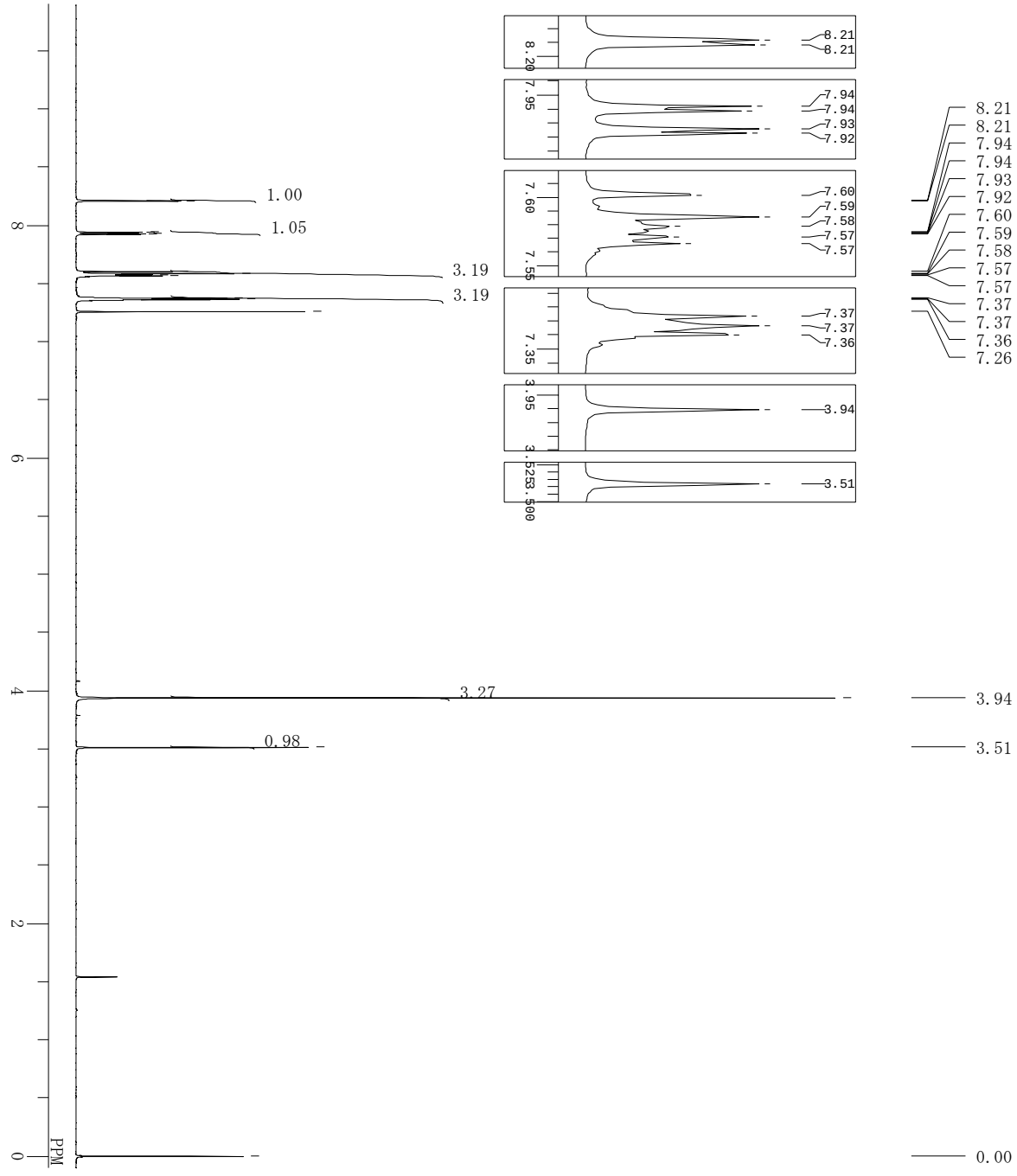


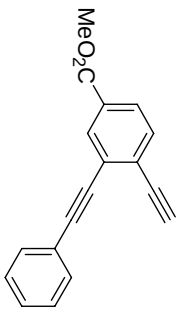
DFILE 110113-G158 ys243NH3-fine.als
COMNT single_pulse
DATIM 13-01-2011 09:51:47
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 24.1 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

1H-NMR (DMSO-D6) δ :
6.48-6.50 (br m, 1H),
6.78 (d, J = 3.4 Hz, 1H),
7.60-7.61 (m, 1H),
7.72-7.73 (m, 2H),
7.90-7.93 (m, 1H),
8.32 (d, J = 3.4 Hz, 1H),
9.34 (s, 1H),
11.59 (s, 1H).



5e

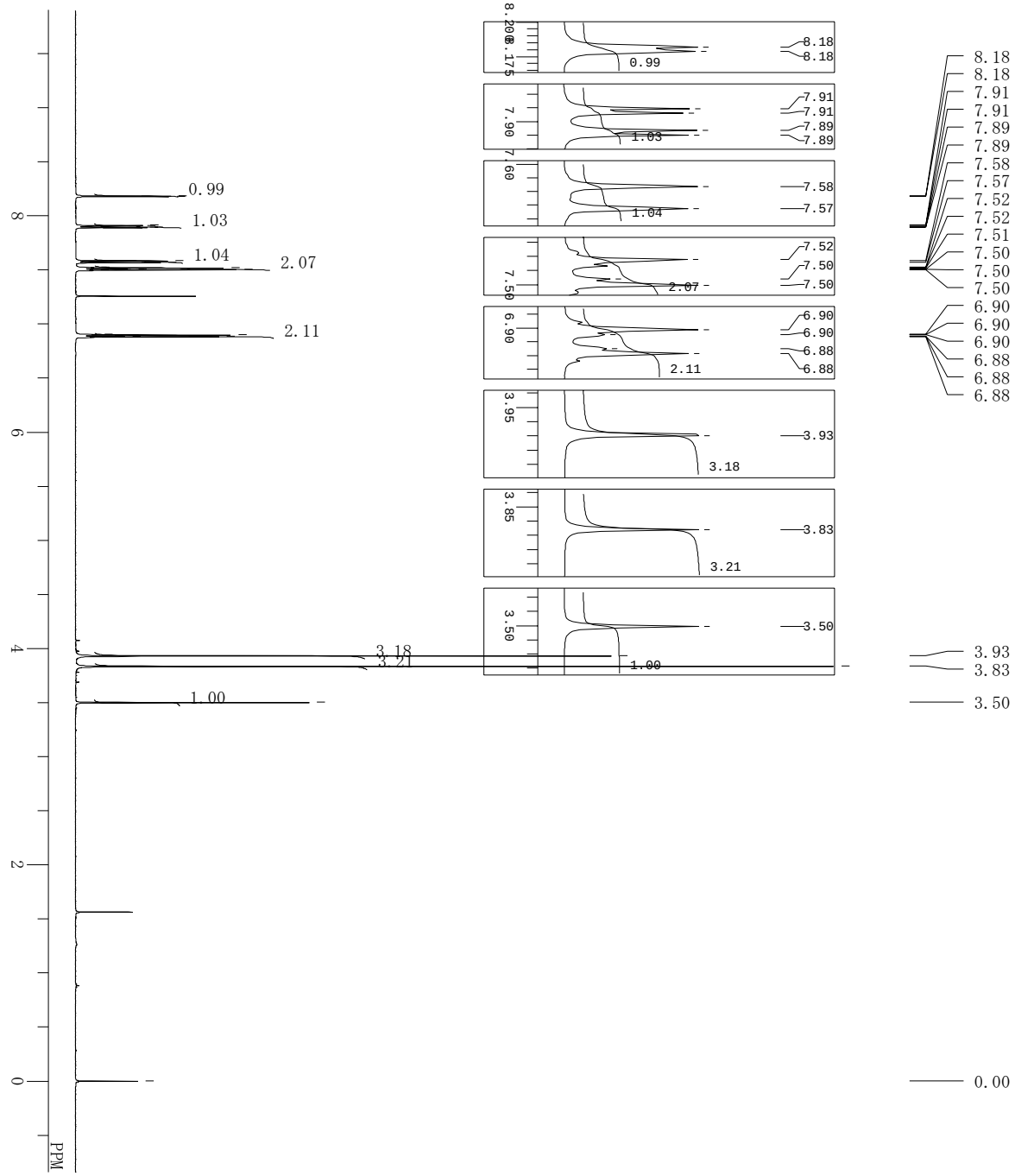




17a

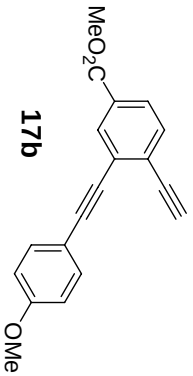
DFILE 120124-B57-1.als
COMNT single_pulse
DATIM 19-01-2012 17:49:44
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

1H-NMR (CDCl3) δ :
3.51 (s, 1H),
3.94 (s, 3H),
7.36-7.37 (m, 3H),
7.57-7.60 (m, 3H),
7.93 (dd, J = 8.0, 1.7 Hz, 1H),
8.21 (d, J = 1.7 Hz, 1H).

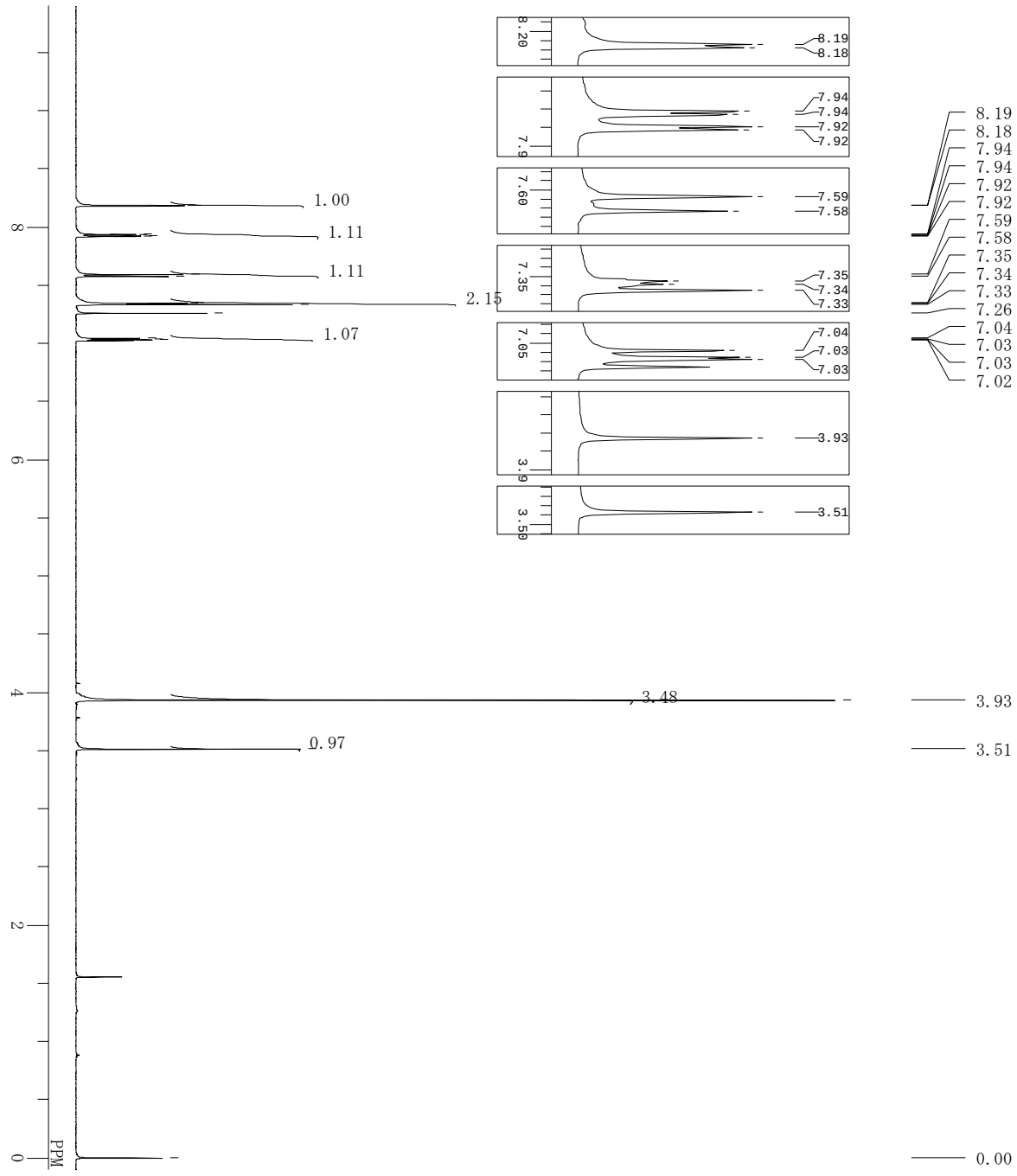


DFILE A195-1.als
COMNT single_pulse
DATIM 16-11-2011 11:03:05
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48

1H-NMR (CDCl3) δ :
3.50 (1H, s),
3.83 (3H, s),
3.93 (3H, s),
6.88-6.90 (2H, m),
7.50-7.52 (2H, m),
7.58 (1H, d, J = 8.0 Hz),
7.90 (1H, dd, J = 8.0, 1.7 Hz),
8.18 (1H, d, J = 1.7 Hz).

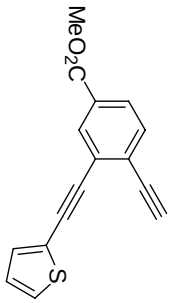


17b

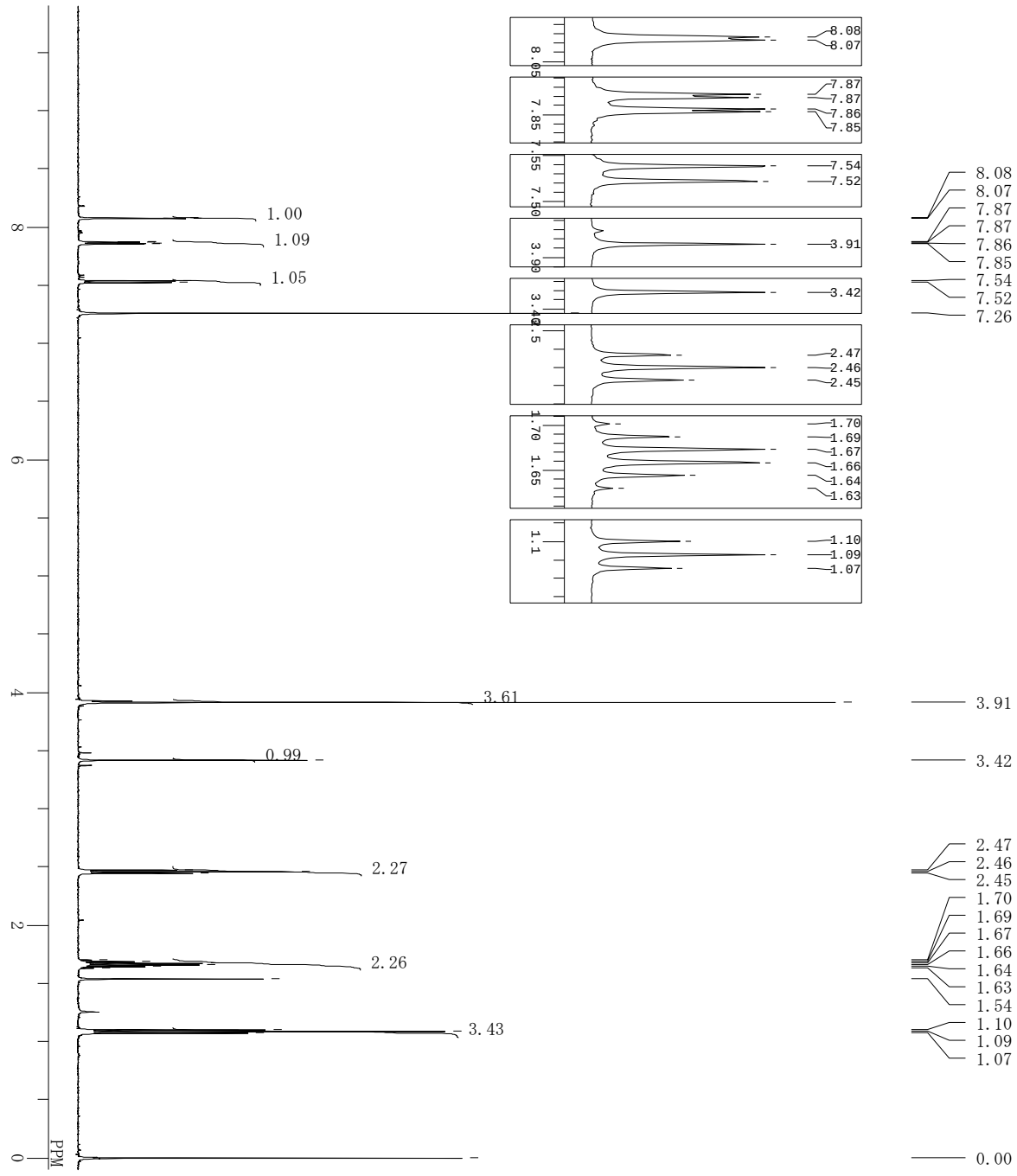


DFILE 120125-ysa210-1.als
COMNT single_pulse
DATIM 25-01-2012 11:05:38
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48

1H-NMR (CDCl3) δ :
3.51 (s, 1H),
3.93 (s, 3H),
7.02-7.04 (m, 1H),
7.33 (s, 1H),
7.34-7.35 (m, 1H),
7.58 (d, J = 8.0 Hz, 1H),
7.93 (dd, J = 8.0, 1.7 Hz, 1H),
8.18 (d, J = 1.7 Hz, 1H).

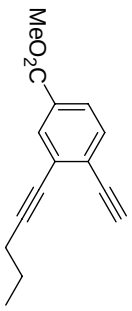


17c

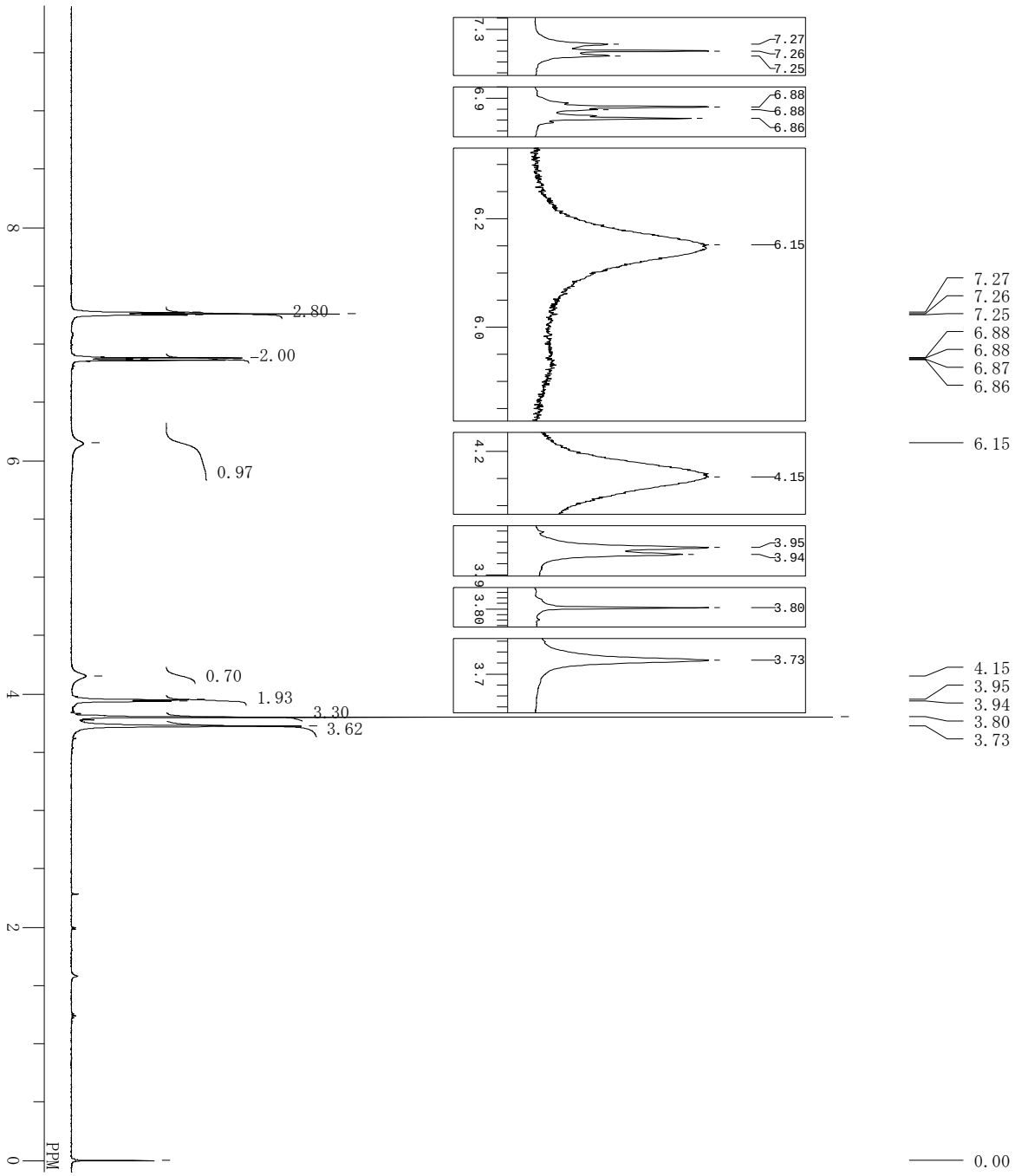


DFILE 120124-NB56-2-1.als
COMNT single_pulse
DATIM 20-01-2012 14:53:04
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

1H-NMR (CDCl3) δ :
1.09 (t, J = 7.4 Hz, 3H),
1.63-1.70 (m, 2H),
2.46 (t, J = 6.9 Hz, 2H),
3.42 (s, 1H),
3.91 (s, 3H),
7.53 (d, J = 8.0 Hz, 1H),
7.86 (dd, J = 8.0, 1.7 Hz, 1H),
8.08 (d, J = 1.7 Hz, 1H).

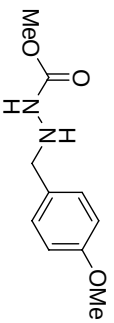


17d

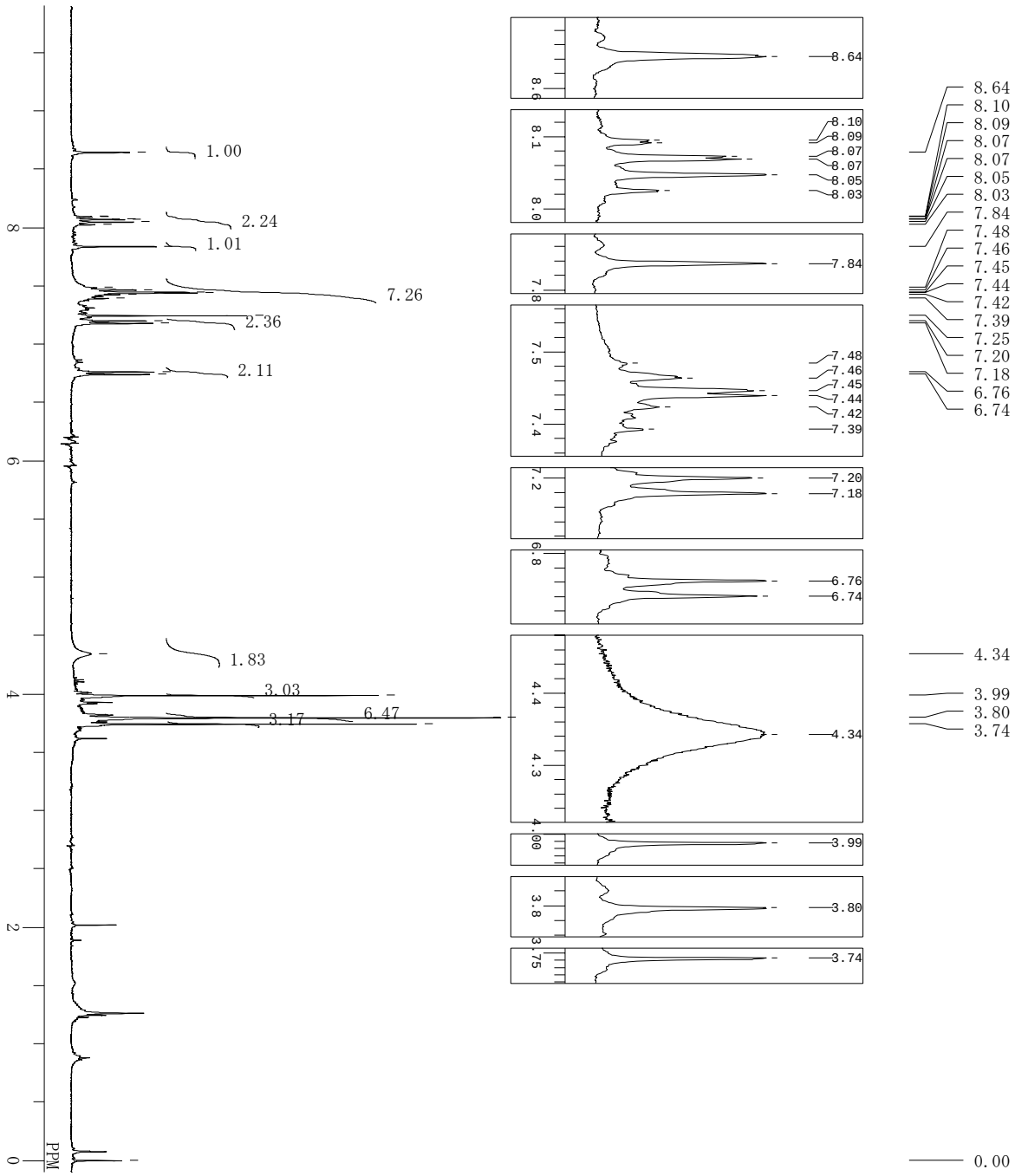


DFILE 111206-I181 ysa171.als
COMNT
DATIM Tue Dec 06 15:04:35 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 8
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

1H-NMR (CDCl3) δ :
3.73 (br s, 3H),
3.80 (s, 3H),
3.94 (d, J = 4.9 Hz, 2H),
4.15 (br s, 1H),
6.15 (br s, 1H),
6.86-6.88 (m, 2H),
7.25-7.27 (m, 3H).

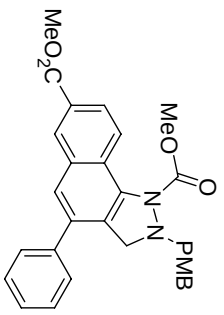


18

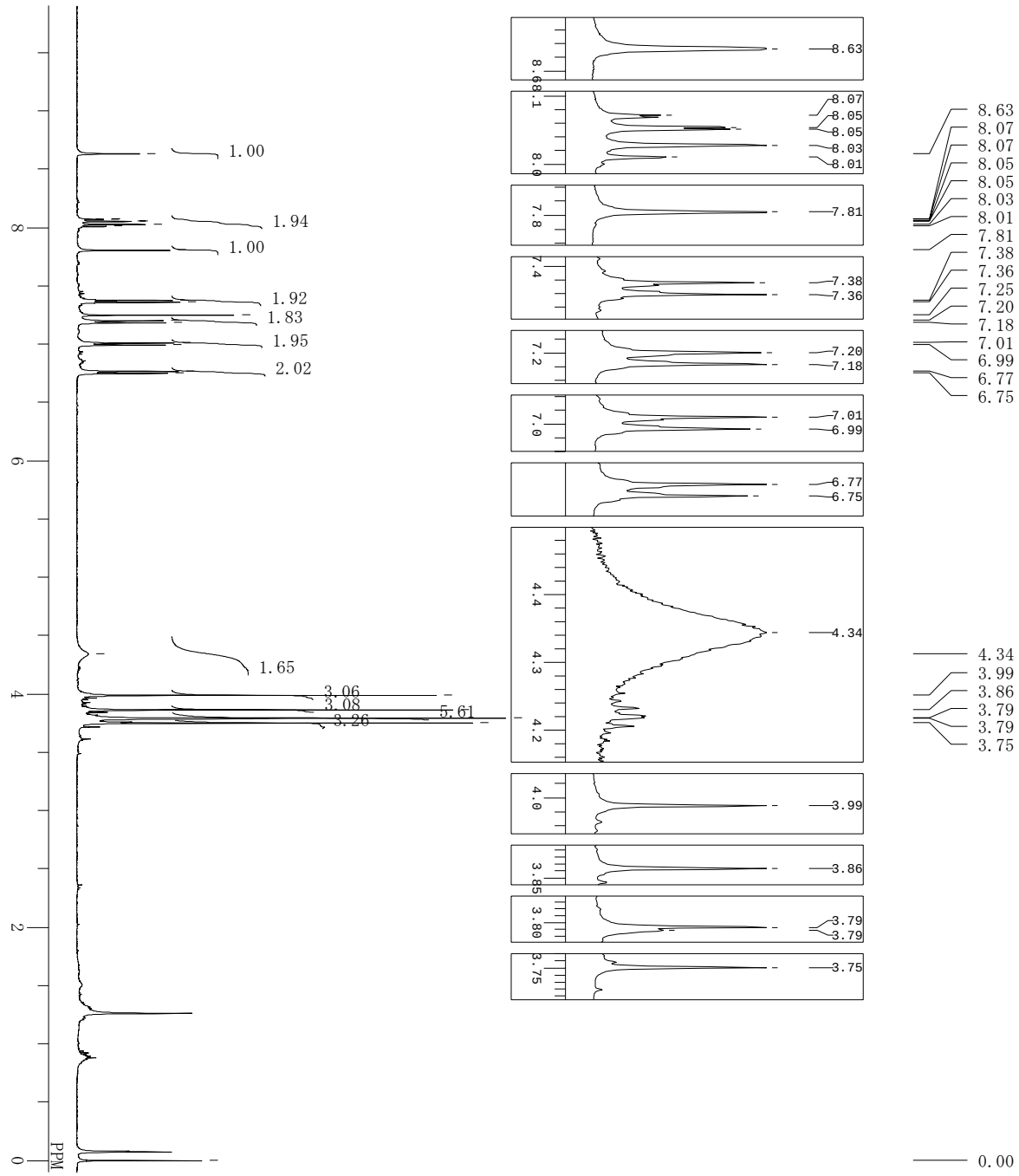


DFILE 120122-J18-ysa223-50d.als
COMNT
DATIM Sun Jan 22 13:41:57 2012
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 2
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 50.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18

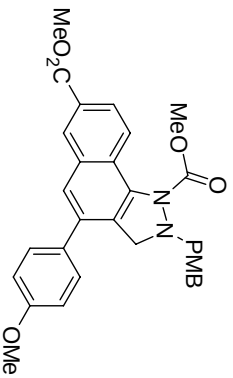
1H-NMR (CDCl3) δ :
3.74 (s, 3H),
3.78-3.81 (br m, 5H),
3.99 (s, 3H),
4.34 (br s, 2H),
6.75 (d, J = 8.5 Hz, 2H),
7.19 (d, J = 8.5 Hz, 2H),
7.39-7.48 (m, 5H),
7.84 (s, 1H),
8.03-8.10 (m, 2H),
8.64 (s, 1H).



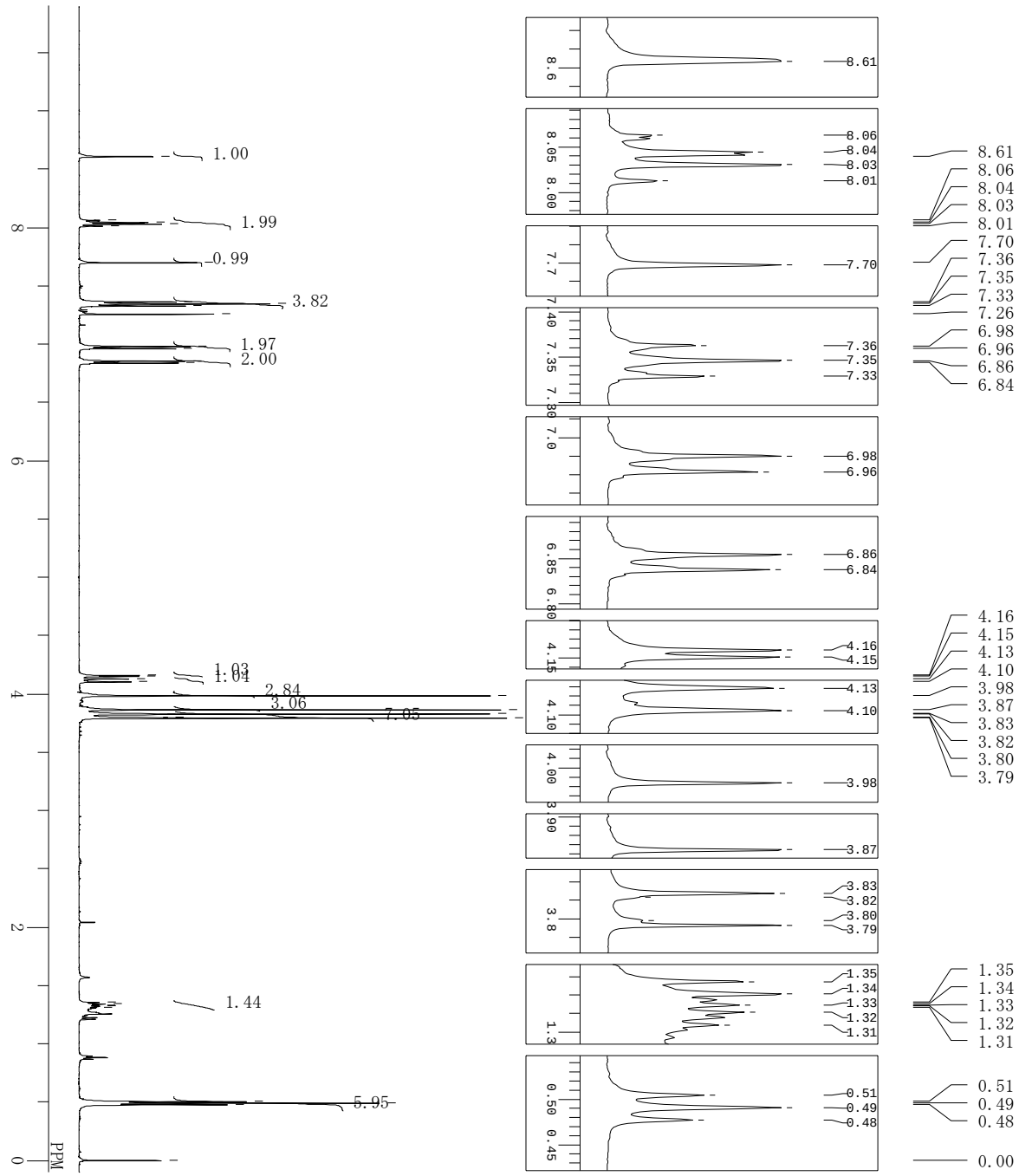
20a



1H-NMR (CDCl3) δ :
3.75 (s, 3H),
3.79 (br s, 2H),
3.79 (s, 3H),
3.86 (s, 3H),
3.99 (s, 3H),
4.34 (br s, 2H),
6.76 (d, J = 8.6 Hz, 2H),
7.00 (d, J = 8.6 Hz, 2H),
7.19 (d, J = 8.6 Hz, 2H),
7.37 (d, J = 8.6 Hz, 2H),
7.81 (s, 1H),
8.01-8.07 (m, 2H),
8.63 (s, 1H).

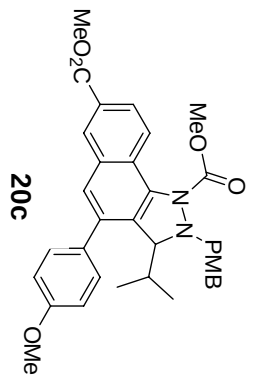


20b

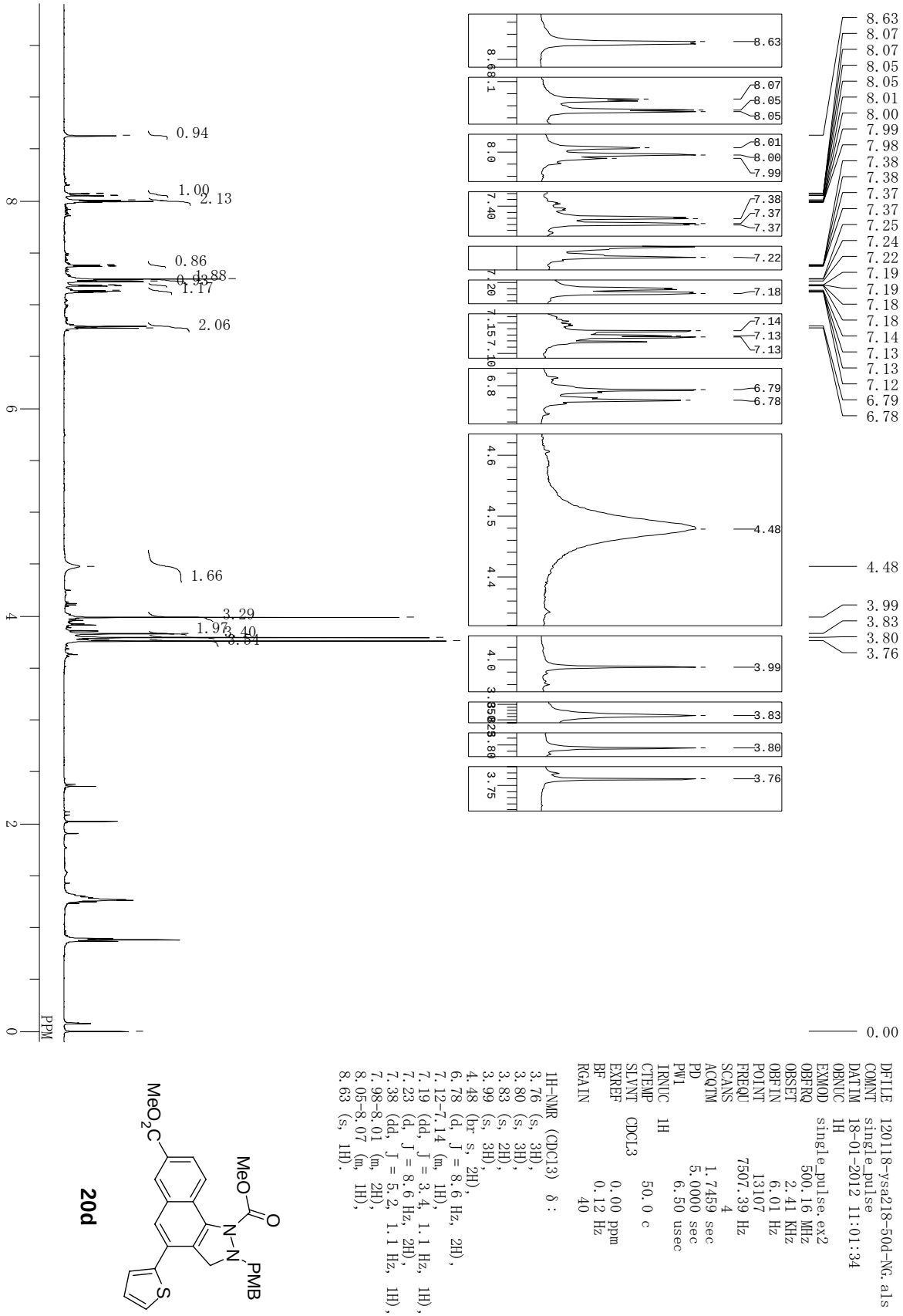


DFILE 111027-1120-ysa181-good.als
COMNT single_pulse
DATIM 27-10-2011 14:38:20
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
1.7459 sec
ACQTM 5.0000 sec
PD 6.50 usec
PW1
IRNUC 1H
CTEMP 25.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

1H-NMR (CDCl3) δ : 0.49 (t, J = 6.9 Hz, 6H), 1.31-1.35 (m, 1H), 3.79 (s, 3H), 3.81 (d, J = 12.6 Hz, 1H), 3.83 (s, 3H), 3.87 (s, 3H), 3.98 (s, 3H), 4.12 (d, J = 12.6 Hz, 1H), 4.16 (d, J = 4.0 Hz, 1H), 6.85 (d, J = 8.6 Hz, 2H), 6.97 (d, J = 8.6 Hz, 2H), 7.33-7.36 (m, 4H), 7.70 (s, 1H), 8.01-8.06 (m, 2H), 8.61 (s, 1H).



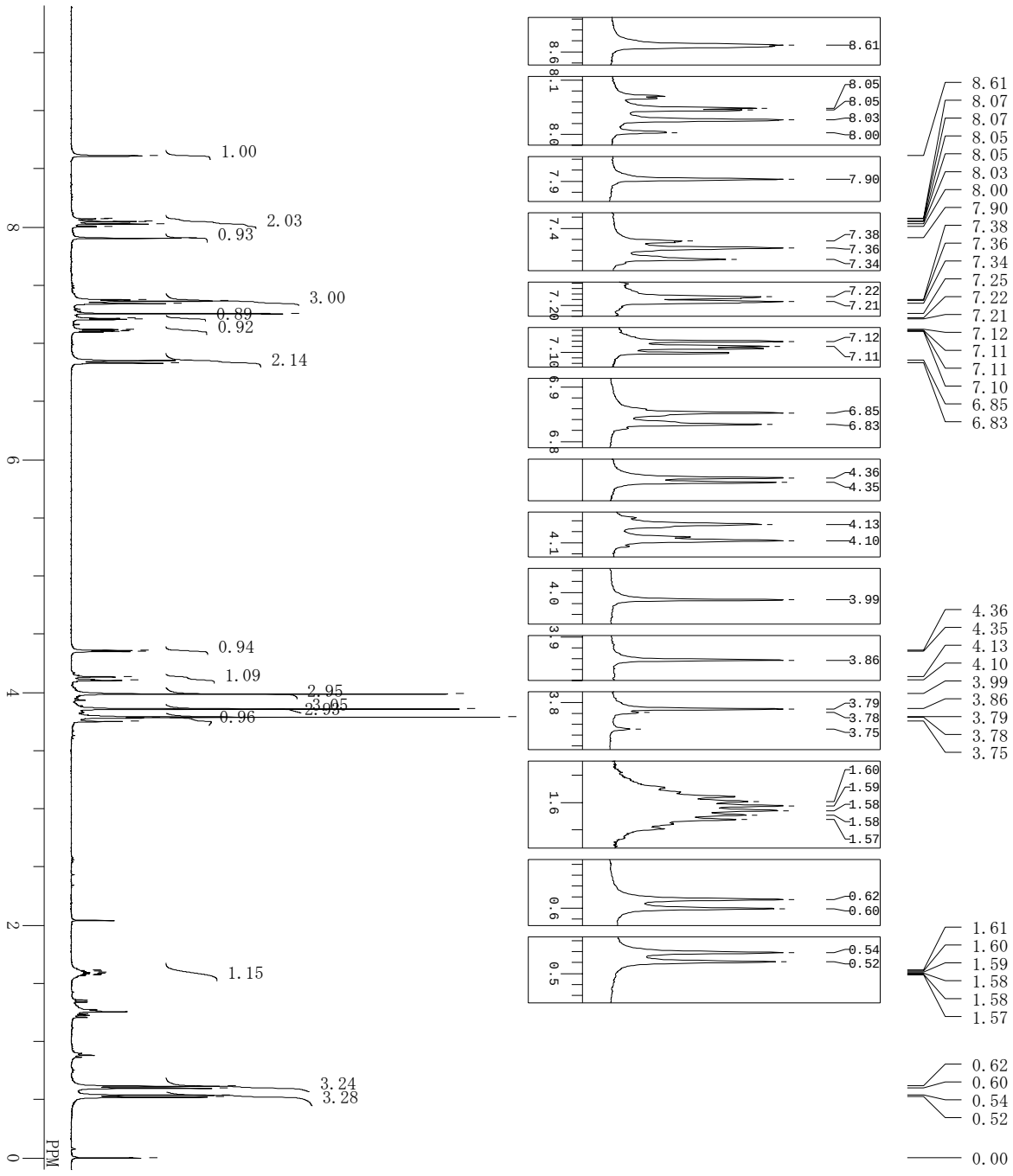
20c



DFILE 120118-ysa218-50d-NG.als
COMNT single_pulse
DATIM 18-01-2012 11:01:34
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 50.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

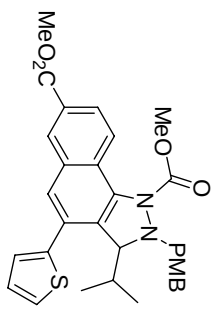
1H-NMR (CDCl3) δ :
3.76 (s, 3H),
3.80 (s, 3H),
3.83 (s, 2H),
3.99 (s, 3H),
4.48 (br s, 2H),
6.78 (d, J = 8.6 Hz, 2H),
7.12-7.14 (m, 1H),
7.19 (dd, J = 3.4, 1.1 Hz, 1H),
7.23 (d, J = 8.6 Hz, 2H),
7.38 (dd, J = 5.2, 1.1 Hz, 1H),
7.98-8.01 (m, 2H),
8.05-8.07 (m, 1H),
8.63 (s, 1H).

20d

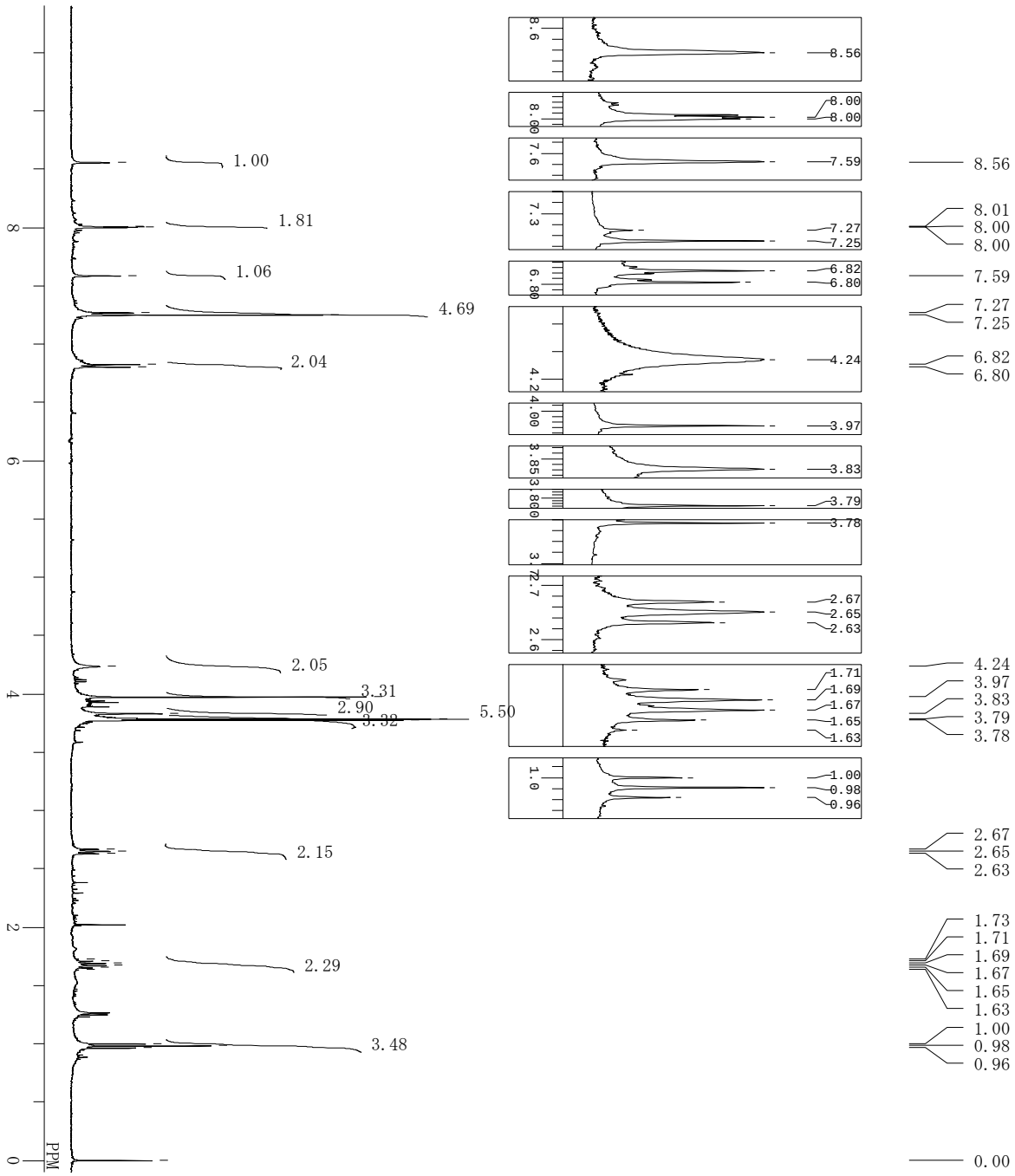


DFILE 111211-I191-ysa211-boo.als
COMNT
DATIM Sun Dec 11 13:41:14 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 2
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 24.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

1H-NMR (CDCl3) δ :
0.53 (d, J = 6.8 Hz, 3H),
0.61 (d, J = 6.8 Hz, 3H),
1.55-1.63 (m, 1H),
3.77 (d, J = 12.2 Hz, 1H),
3.79 (s, 3H),
3.86 (s, 3H),
3.99 (s, 3H),
4.12 (d, J = 12.2 Hz, 1H),
4.36 (d, J = 3.4 Hz, 1H),
6.84 (d, J = 8.5 Hz, 2H),
7.11 (dd, J = 4.8, 3.4 Hz, 1H),
7.21 (d, J = 3.7 Hz, 1H),
7.34-7.38 (m, 3H),
7.90 (s, 1H),
8.00-8.07 (m, 2H),
8.61 (s, 1H).

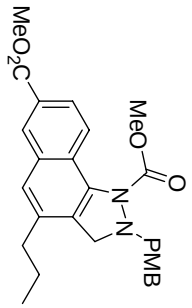


20e

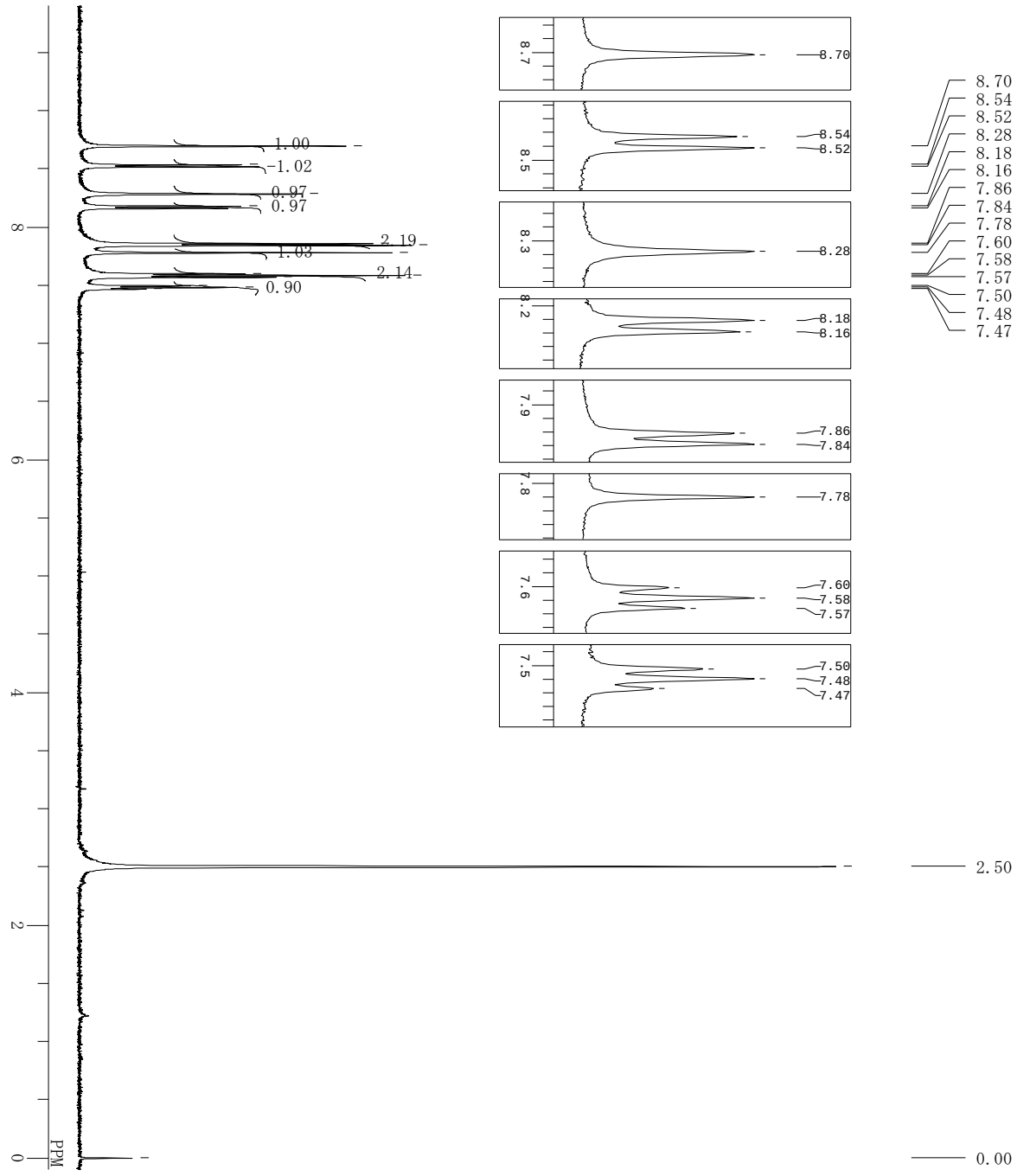


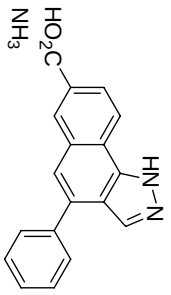
DFILE 120122-J20-ysa224.als
COMNT
DATIM Sun Jan 22 15:46:26 2012
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 4
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 50.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22

1H-NMR (CDCl3) δ :
0.98 (t, J = 7.3 Hz, 3H),
1.63-1.73 (m, 2H),
2.65 (t, J = 7.6 Hz, 2H),
3.78 (s, 3H),
3.79 (s, 3H),
3.83 (s, 2H),
3.97 (s, 3H),
4.24 (br s, 2H),
6.81 (d, J = 8.5 Hz, 2H),
7.26 (d, J = 8.5 Hz, 2H),
7.59 (s, 1H),
8.00-8.01 (m, 2H),
8.56 (s, 1H).

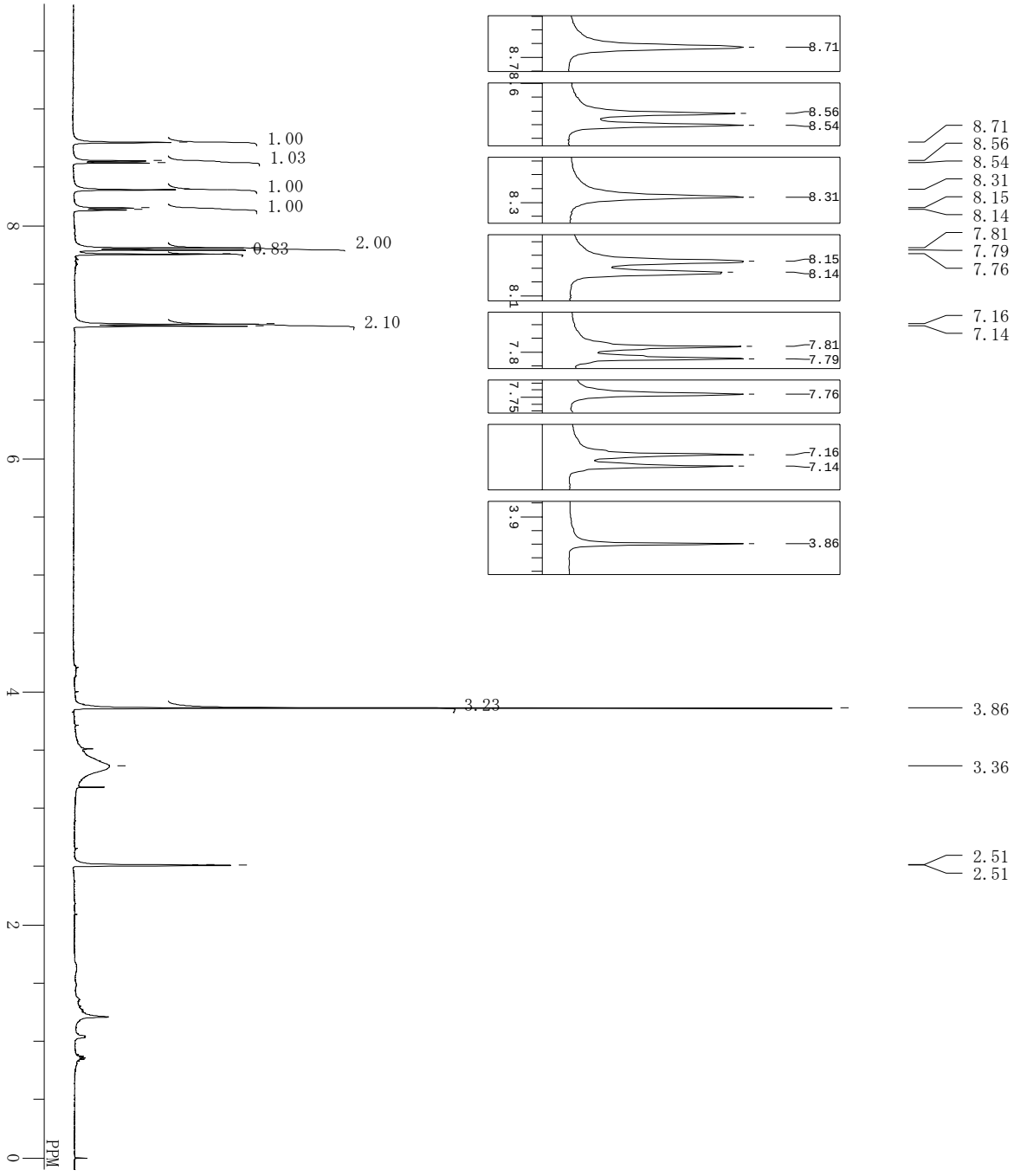


20f


single_pulse
8.70
8.54
8.52
8.28
8.18
8.16
7.86
7.84
7.78
7.60
7.58
7.57
7.50
7.48
7.47
2.50
0.00
1.00
1.02
0.97
0.97
2.19
1.03
2.14
0.90
PPM
DFILE 110127-H26 ysa95NH3-fine.als
COMNT single_pulse
DATIM 27-01-2011 17:55:47
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.8 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48
1H-NMR (DMSO-D6) δ :
7.48 (dd, J = 7.6, 7.6 Hz, 1H),
7.58 (dd, J = 7.6, 7.6 Hz, 2H),
7.78 (s, 1H),
7.85 (d, J = 7.6 Hz, 2H),
8.17 (d, J = 8.6 Hz, 1H),
8.28 (s, 1H),
8.53 (d, J = 8.6 Hz, 1H),
8.70 (s, 1H).

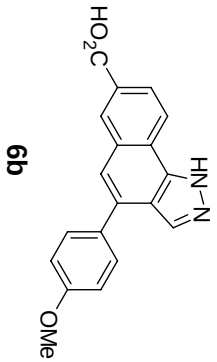

HO2C
NH3
HN–N

6a

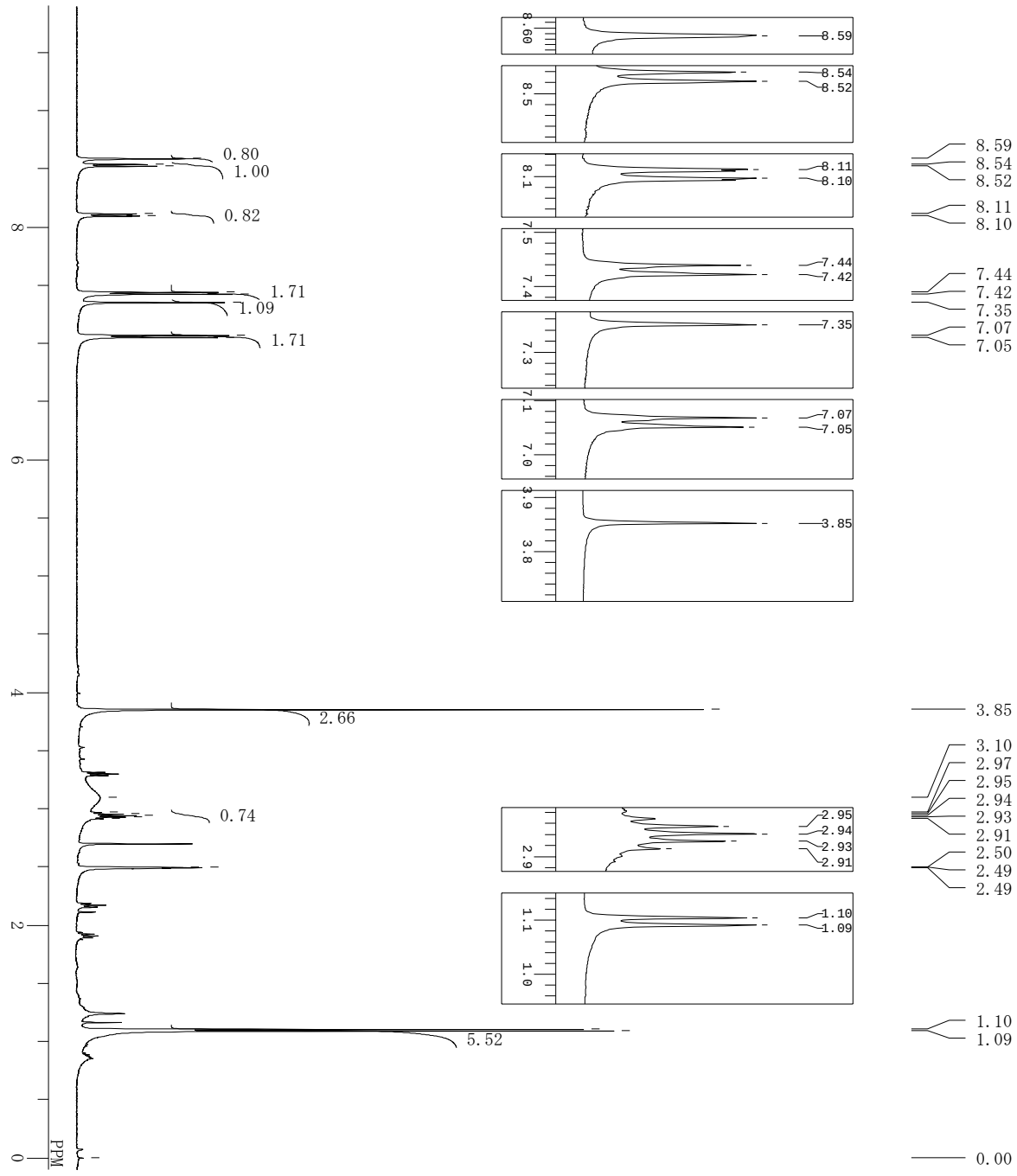


DFILE 111214-I192-ysa212 DMSO-great.e
COMNT single_pulse
DATIM 14-12-2011 18:18:49
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 26.0 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

1H-NMR (DMSO-D6) δ :
3.86 (s, 3H),
7.15 (d, J = 8.6 Hz, 2H),
7.76 (s, 1H),
7.80 (d, J = 8.6 Hz, 2H),
8.14 (d, J = 8.6 Hz, 1H),
8.31 (s, 1H),
8.55 (d, J = 8.6 Hz, 1H),
8.71 (s, 1H).

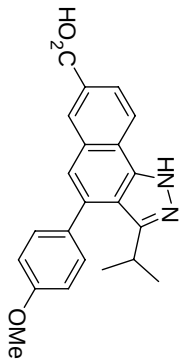


6b

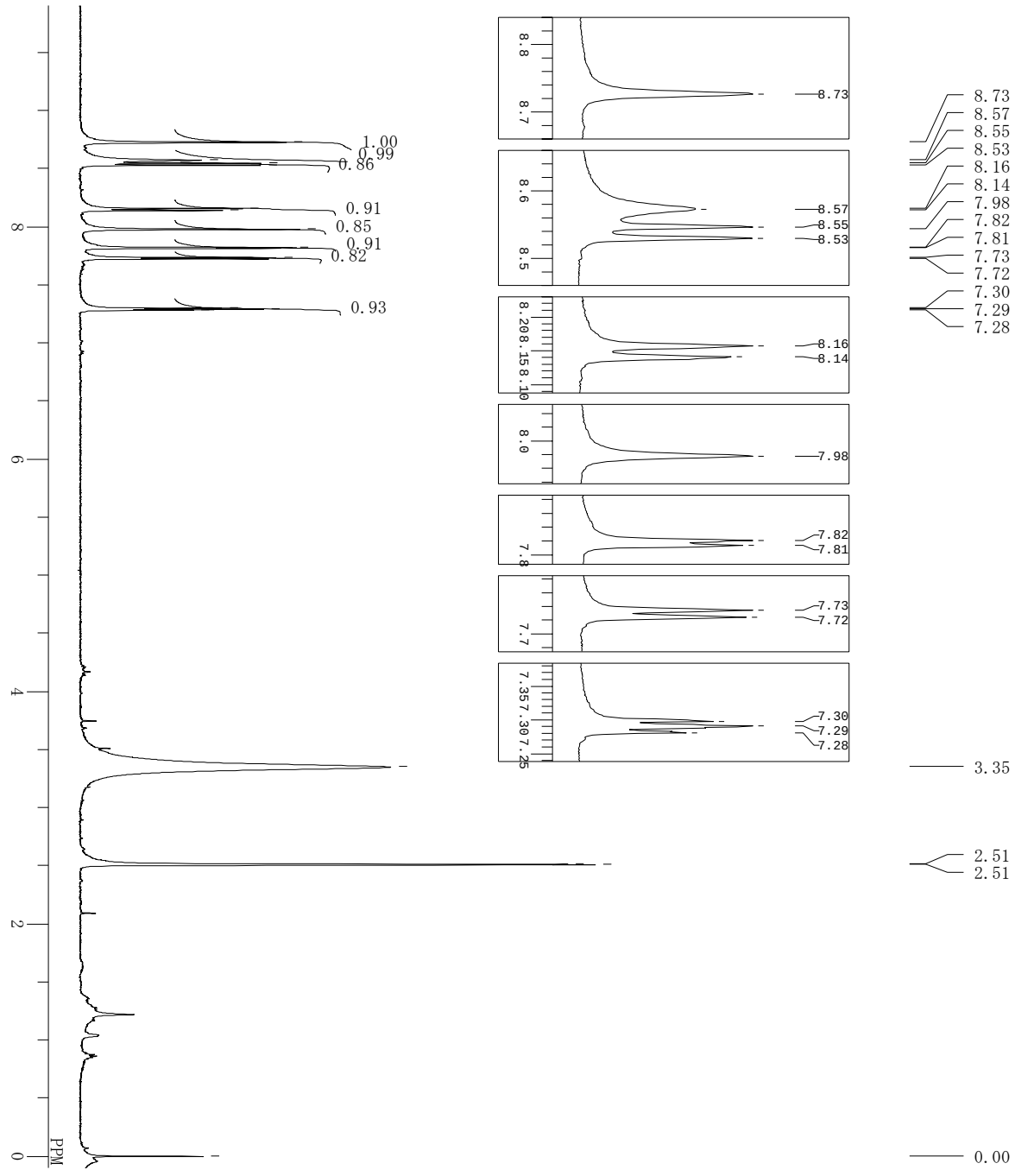


DFILE 111214-1140-ysa184 DMSO80-1.als
COMNT single_pulse
DATIM 14-12-2011 10:33:54
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 80.0 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

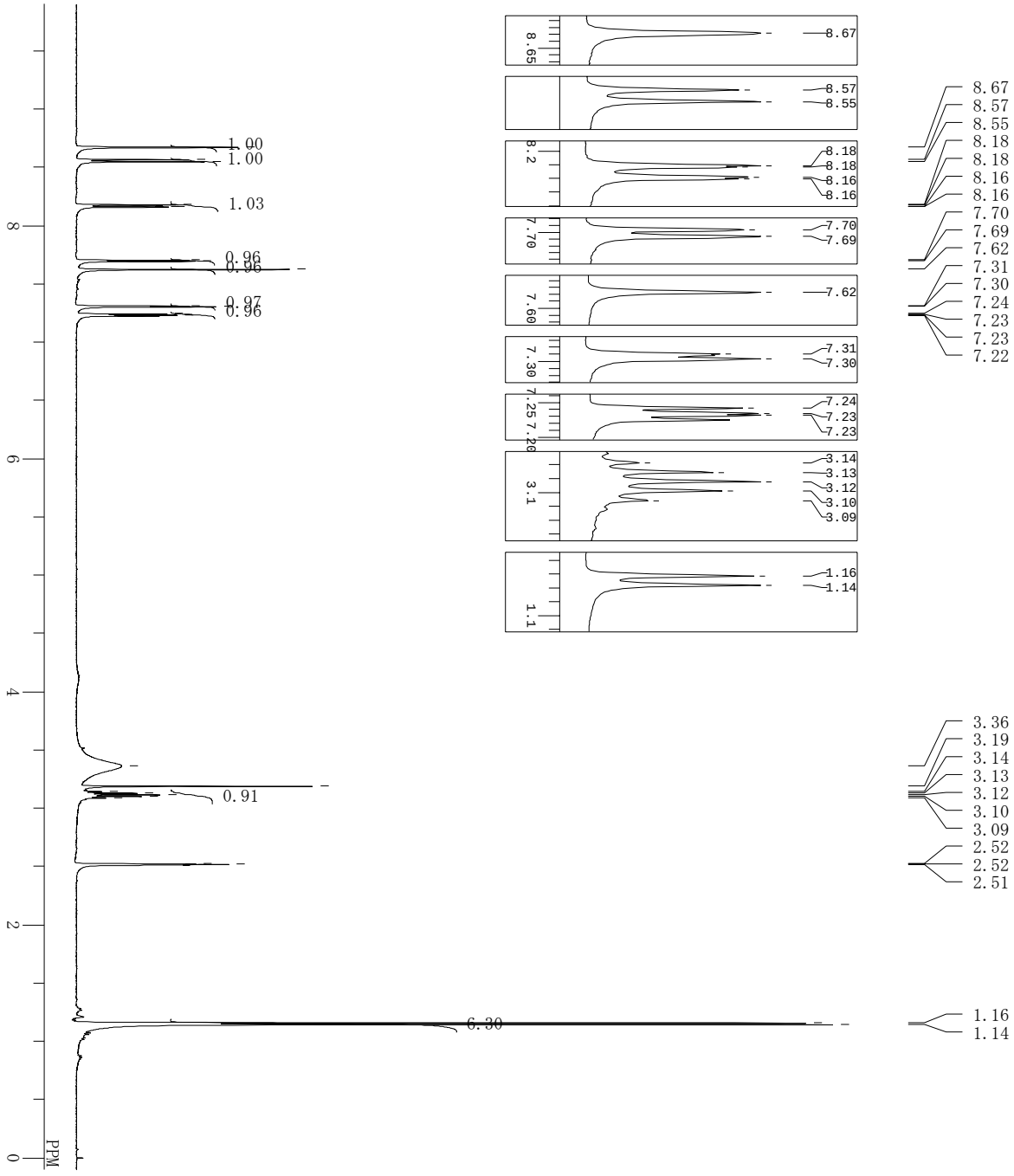
1H-NMR (DMSO-D6) δ : 1.10 (d, J = 6.9 Hz, 6H), 2.91-2.97 (m, 1H), 3.85 (s, 3H), 7.06 (d, J = 8.6 Hz, 2H), 7.35 (s, 1H), 7.43 (d, J = 8.6 Hz, 2H), 8.10 (d, J = 8.6 Hz, 1H), 8.53 (d, J = 8.6 Hz, 1H), 8.59 (s, 1H).



6c

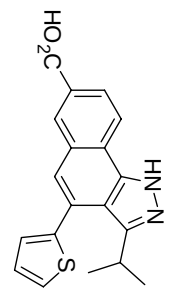

single_pulse
8.73
8.57
8.55
8.53
8.16
8.14
7.98
7.82
7.81
7.73
7.72
7.30
7.29
7.28
3.35
2.51
2.51
0.00
1.00
0.99
0.86
0.91
0.85
0.91
0.82
0.93
PPM
DFILE 111213-I193-ysa213 DMSO great.e
COMNT single_pulse
DATIM 13-12-2011 21:53:04
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
1H-NMR (DMSO-D6) δ :
7.28-7.29 (m, 1H),
7.73 (d, J = 5.2 Hz, 1H),
7.82 (d, J = 3.4 Hz, 1H),
7.98 (s, 1H),
8.15 (d, J = 8.6 Hz, 1H),
8.54 (d, J = 8.6 Hz, 1H),
8.57 (br s, 1H),
8.73 (s, 1H).

6d

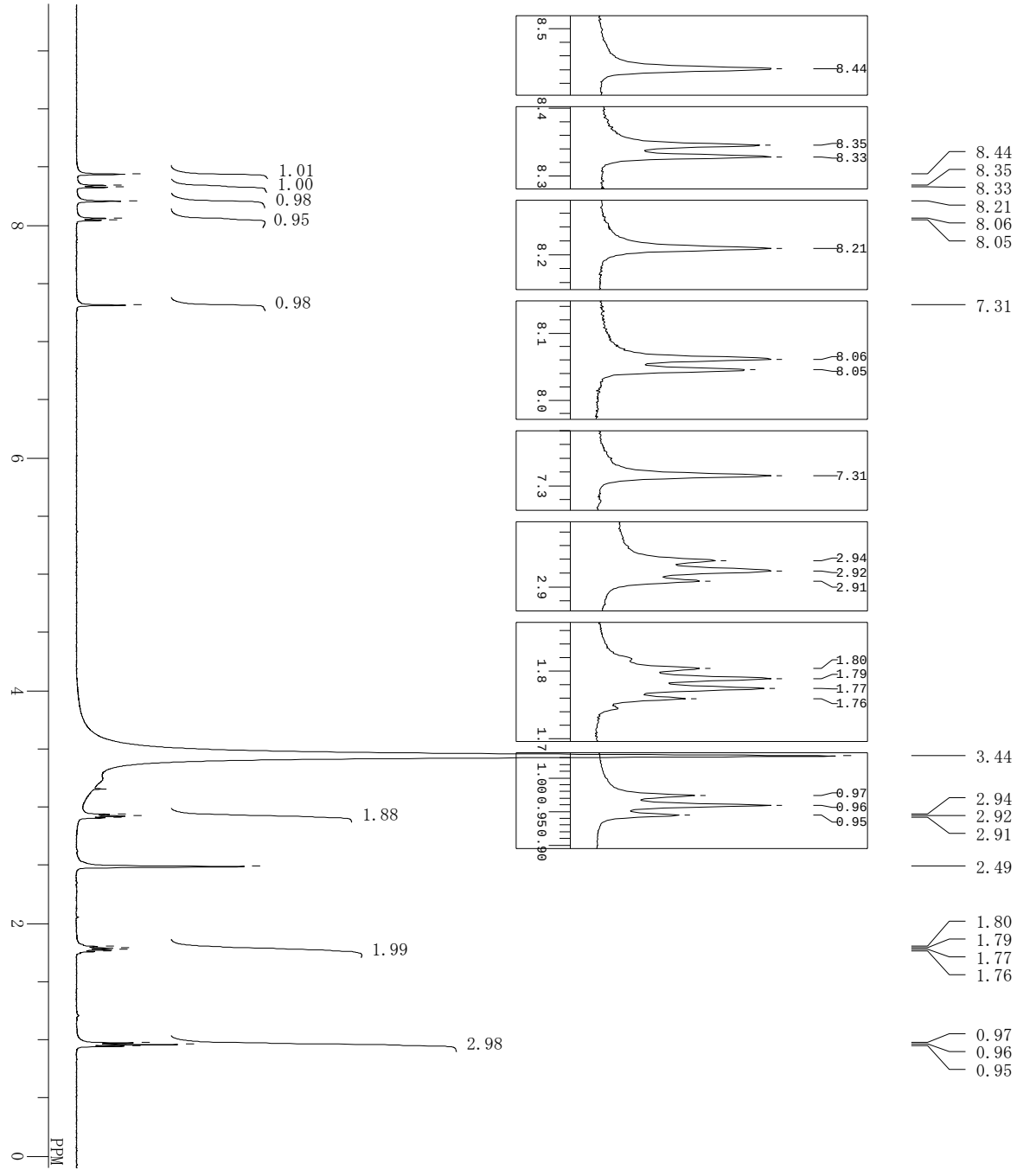


DFILE 111214-I196-ysa215 DMSO-boo.als
COMNT single_pulse
DATIM 14-12-2011 22:32:26
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT DMSO
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

1H-NMR (DMSO-D6) δ :
1.15 (d, J = 6.9 Hz, 6H),
3.09-3.14 (m, 1H),
7.23 (dd, J = 4.9, 3.7 Hz, 1H),
7.31 (d, J = 3.7 Hz, 1H),
7.62 (s, 1H),
7.70 (d, J = 4.9 Hz, 1H),
8.17 (d, J = 8.6 Hz, 1H),
8.56 (d, J = 8.6 Hz, 1H),
8.67 (s, 1H).

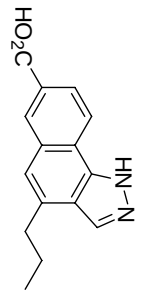


6e

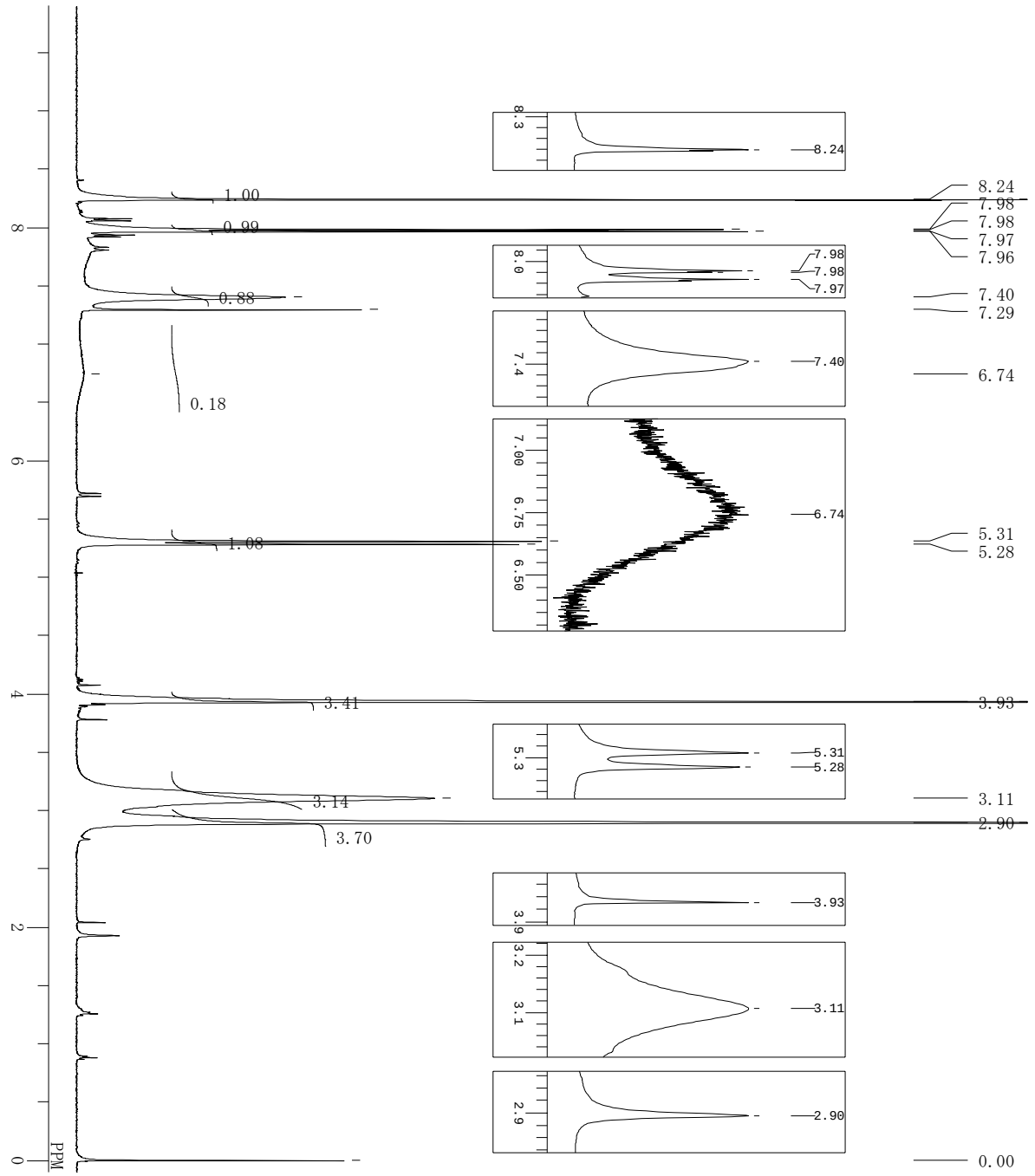


DFILE 120122-ysa100-1.als
COMNT single_pulse
DATIM 22-01-2012 20:59:48
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
1.7459 sec
ACQTM 5.0000 sec
PD 6.50 usec
PW1
IRNUC 1H
CTEMP 26.6 c
SLVNT DMSO
EXREF 2.49 ppm
BF 0.12 Hz
RGAIN 44

1H-NMR (DMSO-D6) δ :
0.96 (t, J = 7.2 Hz, 3H),
1.75-1.82 (m, 2H),
2.92 (t, J = 7.4 Hz, 2H),
7.31 (s, 1H),
8.05 (d, J = 8.4 Hz, 1H),
8.21 (s, 1H),
8.34 (d, J = 8.4 Hz, 1H),
8.44 (s, 1H).

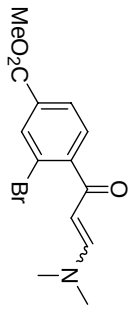


6f



DFILE 1201250-ysa63-1.als
COMNT single_pulse
DATIM 25-01-2012 13:31:55
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38

1H-NMR (CDCl3) δ :
2.90 (s, 4H),
3.11 (br s, 3H),
3.93 (s, 3H),
5.30 (d, J = 12.6 Hz, 1H),
6.74 (br s, 1H),
7.40 (br s, 1H),
7.97 (dd, J = 8.0, 1.7 Hz, 1H),
8.24 (d, J = 1.7 Hz, 1H).



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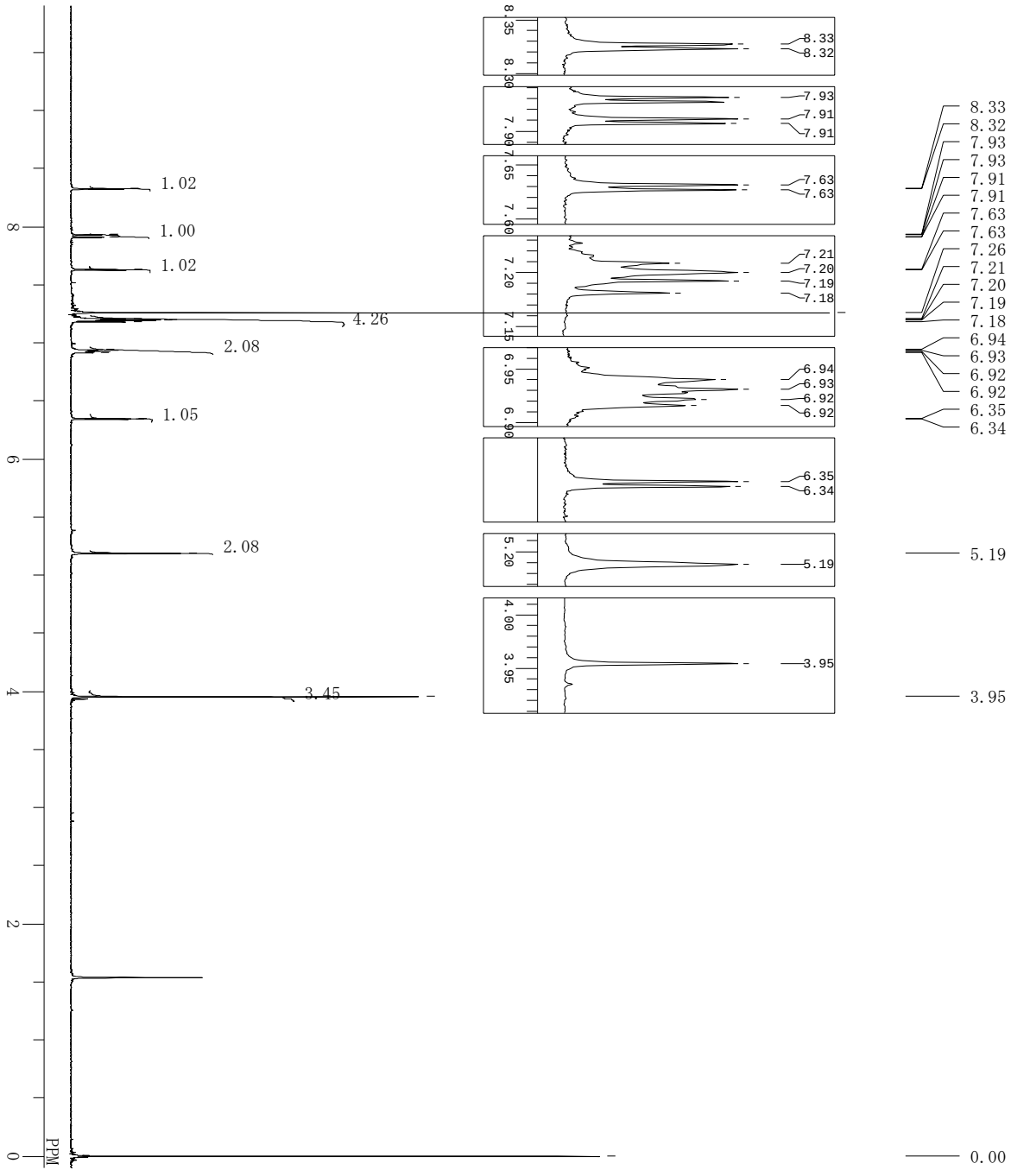
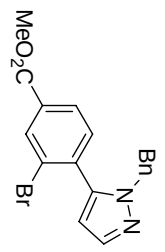


DFILE 101228-H17 ysa64-fine.als
COMNT
DATIM Tue Dec 28 14:44:21 2010
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 8
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 24

1H-NMR (CDCl3) δ :
3.95 (s, 3H),
5.19 (s, 2H),
6.35 (d, J = 1.7 Hz, 1H),
6.92-6.94 (m, 2H),
7.18-7.21 (m, 4H),
7.63 (d, J = 1.7 Hz, 1H),
7.92 (dd, J = 7.9, 1.7 Hz, 1H),
8.33 (d, J = 1.7 Hz, 1H).



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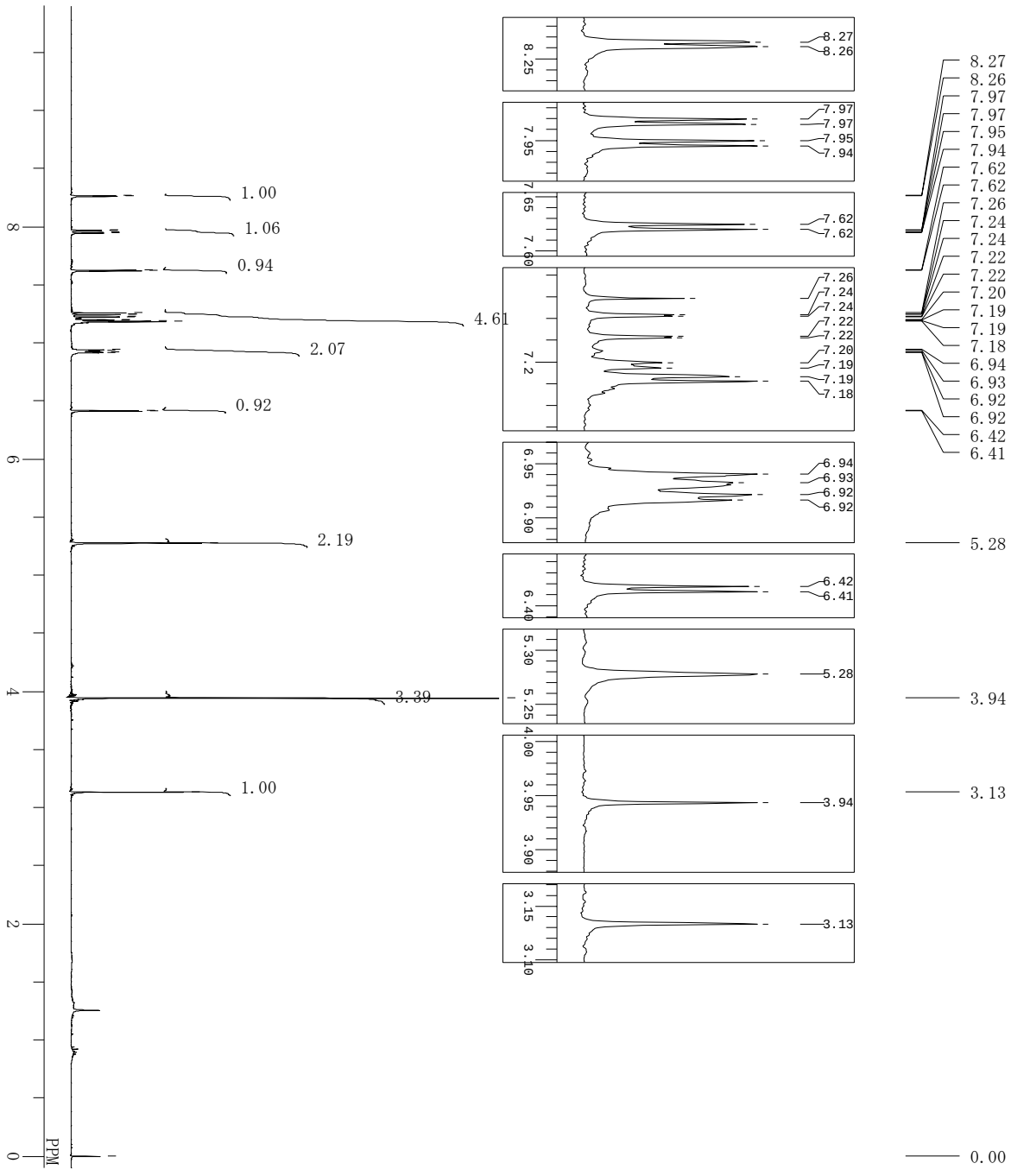
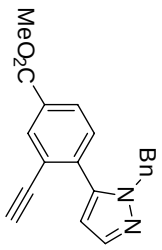


DFILE 110105-H21 ysa92 fine.als
COMNT
DATIM Wed Jan 05 11:41:21 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 1
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16

1H-NMR (CDCl3) δ :
3.13 (s, 1H),
3.94 (s, 3H),
5.28 (s, 2H),
6.42 (d, J = 2.0 Hz, 1H),
6.92-6.94 (m, 2H),
7.18-7.26 (m, 4H),
7.62 (d, J = 2.0 Hz, 1H),
7.96 (dd, J = 8.0, 2.0 Hz, 1H),
8.26 (d, J = 1.7 Hz, 1H).



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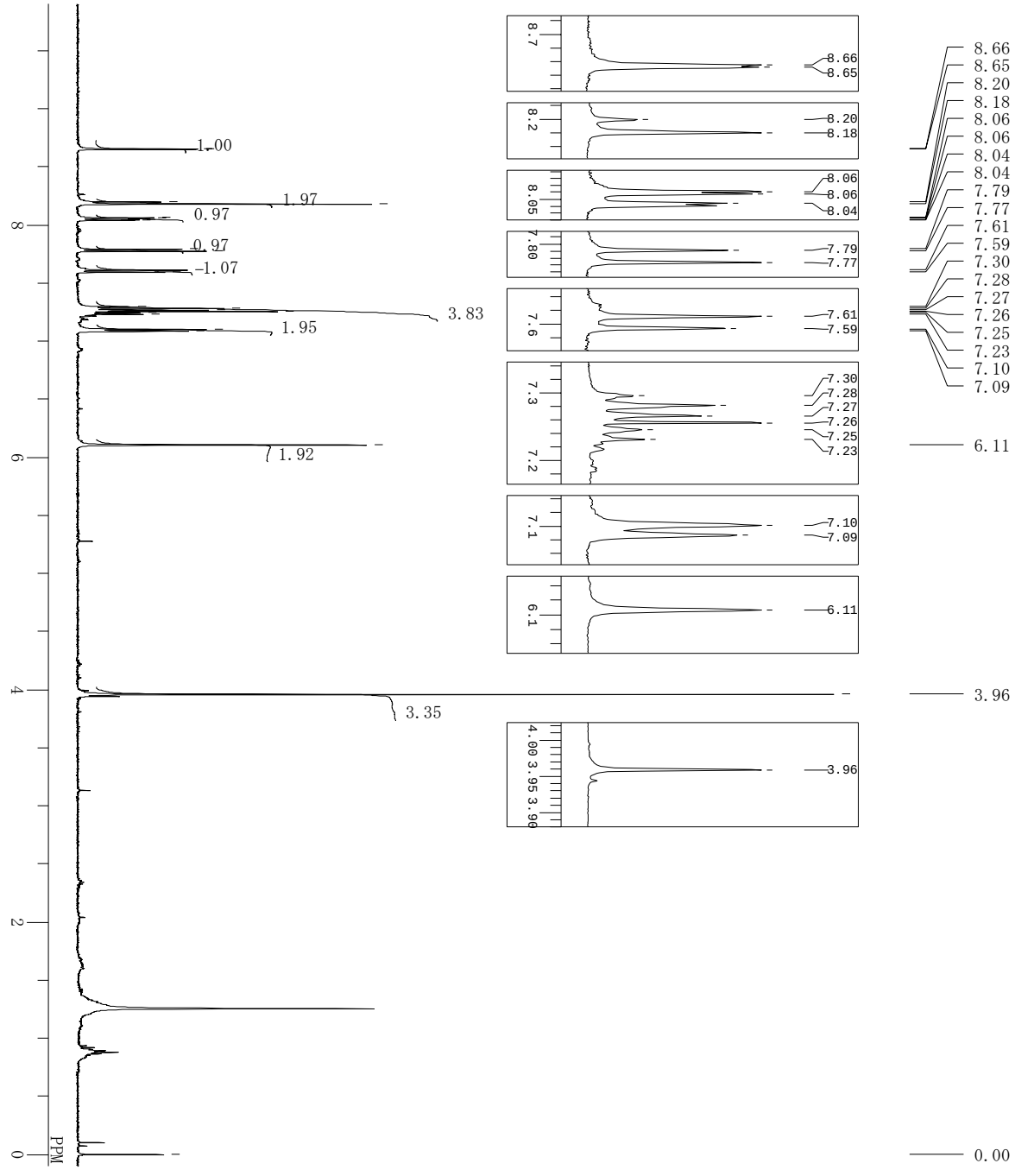
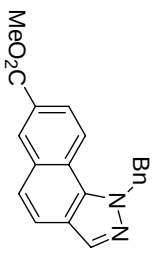
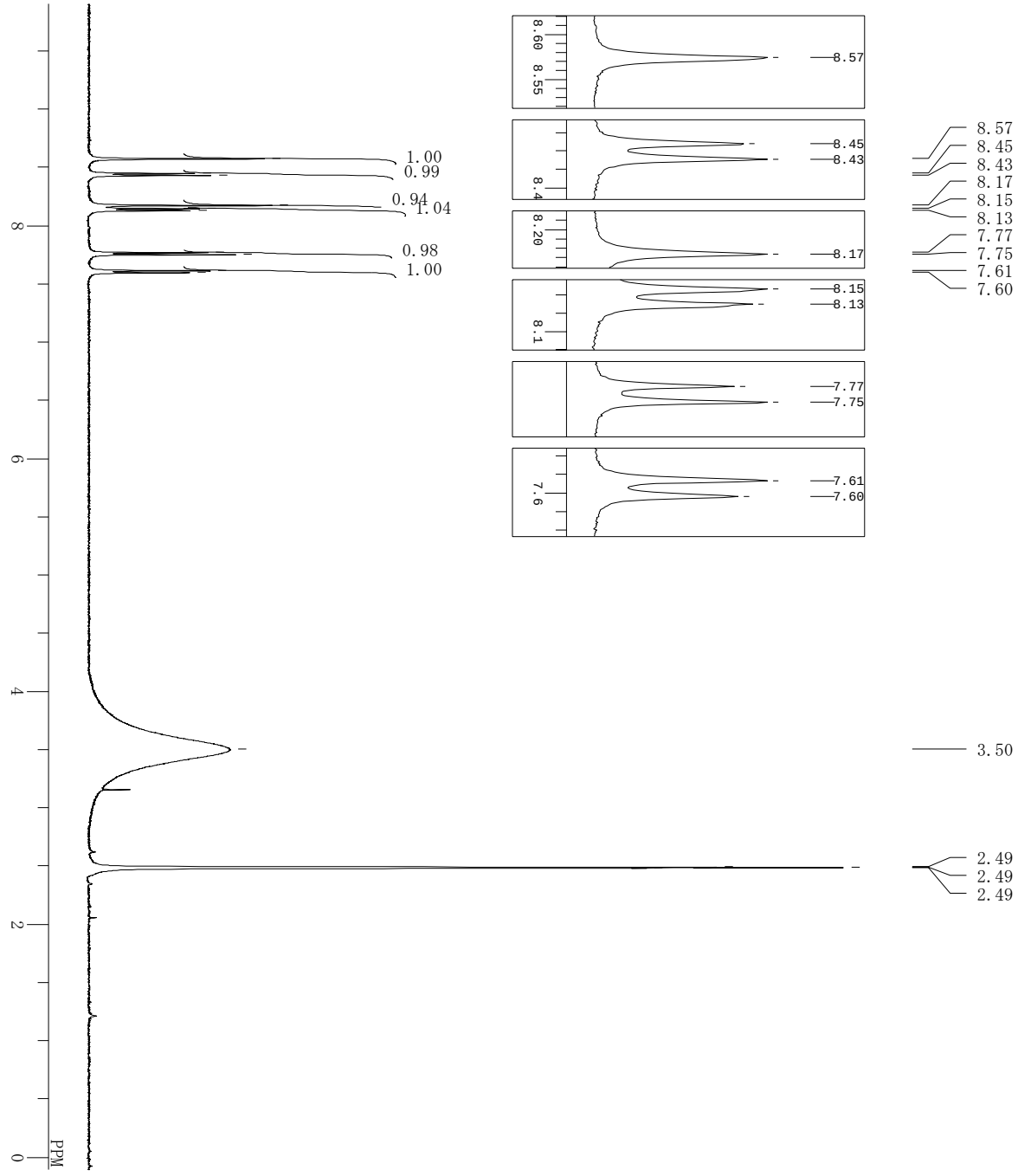


DFILE 110121-H38 ysa97-fine.als
COMNT single_pulse
DATIM 21-01-2011 10:25:36
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 2
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54

1H-NMR (CDCl3) δ :
3.96 (s, 3H),
6.11 (s, 2H),
7.09 (d, J = 7.4 Hz, 2H),
7.23-7.30 (m, 3H),
7.60 (d, J = 9.2 Hz, 1H),
7.78 (d, J = 9.2 Hz, 1H),
8.05 (dd, J = 8.6, 1.7 Hz, 1H),
8.18 (s, 1H),
8.20 (s, 1H),
8.65 (d, J = 1.7 Hz, 1H).

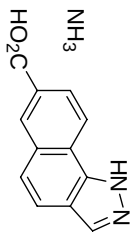


21g



DFILE 120122-ysa99-NH3-1.als
COMNT single_pulse
DATIM 22-01-2012 13:14:42
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 26.7 c
SLVNT DMSO
EXREF 2.49 ppm
BF 0.12 Hz
RGAIN 50

1H-NMR (DMSO-D6) δ :
7.60 (d, J = 8.6 Hz, 1H),
7.76 (d, J = 8.6 Hz, 1H),
8.14 (d, J = 8.6 Hz, 1H),
8.17 (s, 1H),
8.44 (d, J = 8.6 Hz, 1H),
8.57 (s, 1H).



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