

Supporting Information for

Palladium-Catalyzed C-H Bond Functionalization of C6-Arylpurines

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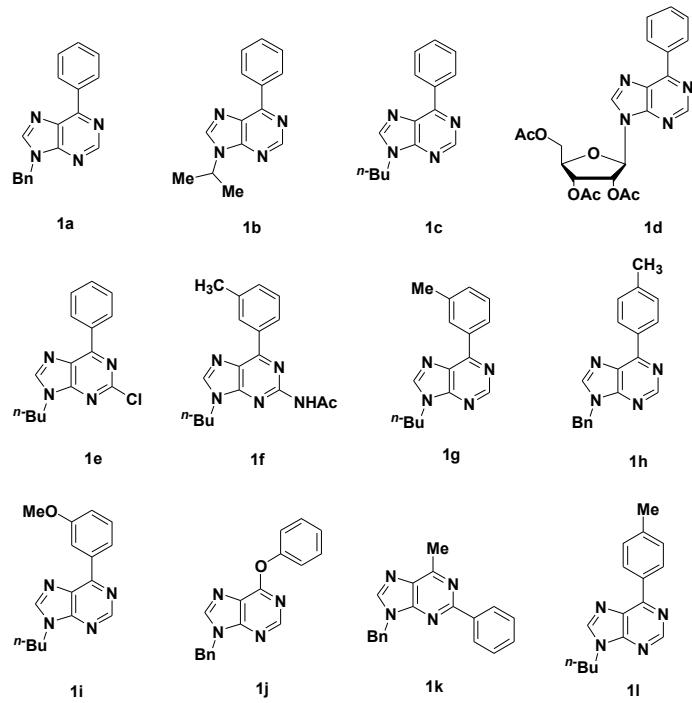
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General Information: NMR spectra were recorded with a 400 MHz spectrometer for ^1H NMR, 100 MHz for ^{13}C NMR. Chemical shifts δ are given in ppm relative to the residual proton signals of the deuterated solvent CDCl_3 for ^1H and ^{13}C NMR. High resolution mass spectra were taken with a 3000 mass spectrometer, using Waters Q-TofMS/MS system. For column chromatography silica gel (200-300 mesh) was used as the stationary phase. All reactions were monitored by thin layer chromatography (TLC). All reagents and solvents were purchased from commercial sources and purified commonly before used.

Synthesis of Starting Materials

Starting materials **1a-i¹**, **1l¹**, **1j²** and **1k^{1,3}** were synthesized according to the corresponding reference.



General procedure for the purine-directed Pd-catalyzed C_{Ar}-H bonds acetoxylation

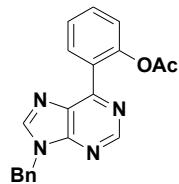
In a 35 mL tube containing a stir bar, C6-aryl purine (**1a-1j**) or **1l** (0.3 mmol), Pd(OAc)₂ (33 mg, 0.015 mmol), PhI(OAc)₂ (0.144 mg, 0.45 mmol) were dissolved in Ac₂O (1 mL) and AcOH (1 mL). The tube was sealed with a Teflon lined cap and the reaction mixture was stirred at 120 °C for 3-15 h until TLC showed the reaction almost proceeded completely. The resulting mixture was cooled to room temperature. The solvent was removed by distillation under vacuum and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc) to afford the corresponding acetoxylated product.

General procedure for the purine-directed diverse C-H bond functionalizations

In a 35 mL tube containing a stir bar, **1c** or **1g**, **1i**, **1l** (0.3 mmol), Pd(OAc)₂ (33 mg, 0.015 mmol), PhI(OAc)₂ (0.144 mg, 0.45 mmol) or NBS (0.45 mmol), NCS (0.45 mmol) were dissolved in solvent as indicated in Table 3. The tube was sealed with a Teflon lined cap and the reaction mixture was stirred at 100 °C for 3-15 h until TLC showed the reaction almost proceeded completely. The resulting mixture was cooled to room temperature. The solvent was removed by distillation under vacuum and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc) to afford the corresponding product.

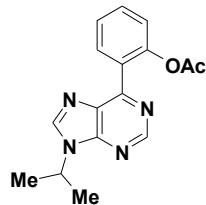
Characterization of compounds

Acetic acid 2-(9-benzyl-9*H*-purin-6-yl)-phenyl ester (**2a**)



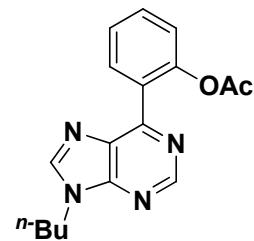
¹H NMR (400 MHz, CDCl₃) δ 9.05 (s, 1H), 8.21-8.19 (m, 1H), 8.07 (s, 1H), 7.55-7.51 (m, 1H), 7.44-7.26 (m, 7H), 5.47 (s, 2H), 2.19 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.6, 154.5, 152.3, 152.2, 148.8, 144.4, 135.1, 132.5, 131.7, 131.3, 129.2, 128.6, 128.0, 127.8, 126.1, 123.9, 47.3, 21.3; HRMS: calcd for C₂₀H₁₆N₄NaO₂ [M + Na]⁺ 367.1171, found 367.1173.

Acetic acid 2-(9-isopropyl-9*H*-purin-6-yl)-phenyl ester (**2b**)



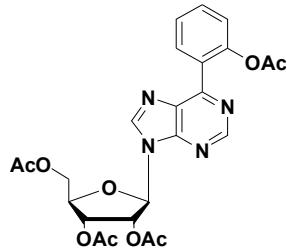
¹H NMR (400 MHz, CDCl₃) δ 9.00 (s, 1H), 8.18 (dd, *J* = 8.0, 1.2 Hz, 1H), 8.16 (s, 1H), 7.53 (t, *J* = 1.2 Hz, 1H), 7.43 (td, *J* = 7.2, 0.8 Hz, 1H), 7.28 (d, *J* = 0.8 Hz, 1H), 4.97 (quint, *J* = 6.8 Hz, 1H), 2.19 (s, 1H), 1.68 (d, *J* = 6.8 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 169.6, 154.4, 151.7, 148.8, 142.3, 132.5, 132.3, 131.1, 128.1, 126.0, 123.9, 47.4, 22.5, 21.2; HRMS: calcd for C₁₆H₁₆N₄NaO₂ [M + Na]⁺ 319.1171, found 319.1176.

Acetic acid 2-(9-butyl-9*H*-purin-6-yl)-phenyl ester (**2c**)



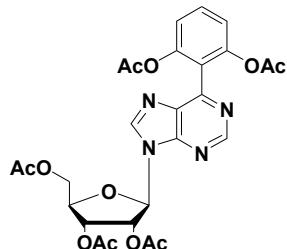
¹H NMR (400 MHz, CDCl₃) δ 9.03 (s, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 8.09 (s, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 8.4 Hz, 1H), 4.32 (t, *J* = 7.2 Hz, 2H), 2.20 (s, 3H), 1.98-1.90 (m, 2H), 1.45-1.35 (m, 2H), 1.07-0.95 (m, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 169.6, 152.0, 148.8, 132.4, 131.2, 126.1, 123.9, 43.8, 31.9, 21.2, 19.9, 13.5; HRMS: calcd for C₁₇H₁₈N₄NaO₂ [M + Na]⁺ 333.1327, found 333.1328.

**(2R,3R,4R,5R)-2-(acetoxymethyl)-5-(6-(2-acetoxyphenyl)-9H-purin-9-yl)tetrahydrofuran-3,4-diyli
diacetate (2da)**



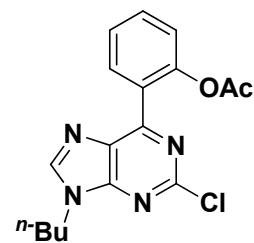
¹H NMR (400 MHz, CDCl₃) δ 9.03 (s, 1H), 8.25 (s, 1H), 8.15 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.56-7.53 (m, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 1H), 6.27 (d, *J* = 5.2 Hz, 1H), 6.01 (t, *J* = 5.2 Hz, 1H), 5.71 (t, *J* = 5.2 Hz, 1H), 4.49-4.38 (m, 3H), 2.18 (s, 3H), 2.16 (s, 3H), 2.12 (s, 3H), 2.10 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.3, 169.6, 169.4, 155.1, 152.4, 151.6, 148.8, 142.9, 132.5, 131.5, 127.7, 126.1, 123.9, 86.5, 80.4, 73.0, 70.6, 63.0, 21.2, 20.7, 20.5, 20.4; HRMS: calcd for C₂₄H₂₄N₄NaO₉ [M + Na]⁺ 535.1441, found 535.1454.

**(2R,3R,4R,5R)-2-(acetoxymethyl)-5-(6-(2,6-diacetoxyphenyl)-9H-purin-9-yl)tetrahydrofuran-3,4-diyli
diacetate (2db)**



¹H NMR (400 MHz, CDCl₃) δ 9.05 (s, 1H), 8.22 (s, 1H), 7.55 (t, *J* = 8.4 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 6.24 (d, *J* = 5.2 Hz, 1H), 5.97 (t, *J* = 5.2 Hz, 1H), 5.72 (t, *J* = 5.2 Hz, 1H), 4.50-4.38 (m, 3H), 2.17 (s, 3H), 2.11 (s, 3H), 2.10 (s, 3H), 2.01 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 170.3, 169.6, 169.4, 168.7, 152.3, 152.1, 151.2, 149.3, 143.3, 133.7, 130.7, 121.4, 120.9, 86.8, 80.3, 73.1, 70.5, 62.9, 20.9, 20.7, 20.5, 20.4; HRMS: calcd for C₂₆H₂₆N₄NaO₁₁ [M + Na]⁺ 593.1496, found 593.1496.

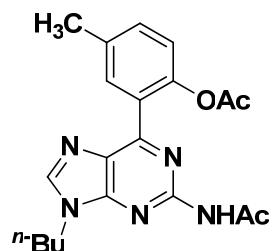
Acetic acid 2-(2-chloro-9-butyl-9H-purin-6-yl)-phenyl ester (2e)



¹H NMR (400 MHz, CDCl₃) δ 8.37 (dd, *J* = 8.0, 1.2 Hz, 1H), 8.07 (s, 1H), 7.58-7.53 (m, 1H), 7.47-7.41 (m, 1H), 7.26 (d, *J* = 7.6 Hz, 1H), 4.27 (t, *J* = 7.2 Hz, 2H), 2.30 (s, 3H), 1.95-1.88 (m, 2H), 1.45-1.35 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.8, 155.8, 153.9, 153.6, 149.1, 145.2, 132.9, 131.9, 130.7, 126.8, 126.1, 124.2, 43.9, 31.8, 21.2, 19.9, 13.5; HRMS: calcd for C₁₇H₁₇ClN₄NaO₂ [M +

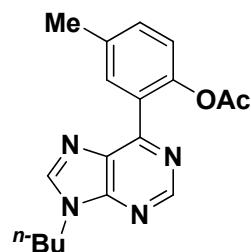
$\text{Na}]^+$ 367.0938, found 367.0932.

Acetic acid 2-(2-acetylamino-9-butyl-9*H*-purin-6-yl)-4-methyl-phenyl ester (2f)



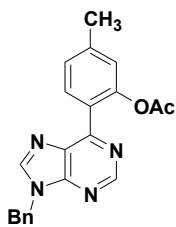
^1H NMR (400 MHz, CDCl_3) δ 8.25 (br, 1H), 7.97 (s, 1H), 7.77 (d, $J = 2.0$ Hz, 1H), 7.32 (dd, $J = 8.4, 1.6$ Hz, 1H), 7.14 (d, $J = 8.0$ Hz, 1H), 4.22 (t, $J = 7.2$ Hz, 2H), 2.59 (s, 3H), 2.42 (s, 3H), 2.11 (s, 3H), 1.95-1.86 (m, 2H), 1.41-1.35 (m, 2H), 0.98 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 155.6, 152.9, 152.4, 146.4, 144.1, 135.8, 132.3, 132.0, 128.5, 127.5, 123.4, 43.6, 31.7, 25.1, 21.2, 20.9, 19.9, 13.5; HRMS: calcd for $\text{C}_{20}\text{H}_{23}\text{N}_5\text{NaO}_3$ $[\text{M} + \text{Na}]^+$ 404.1699, found 404.1695.

Acetic acid 2-(9-butyl-9*H*-purin-6-yl)-4-methyl-phenyl ester (2g)



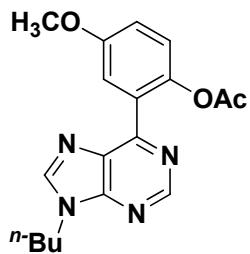
^1H NMR (400 MHz, CDCl_3) δ 9.00 (s, 1H), 8.08 (s, 1H), 7.93 (s, 1H), 7.32 (dd, $J = 8.0, 2.0$ Hz, 1H), 7.15 (d, $J = 8.0$ Hz, 1H), 4.29 (t, $J = 7.2$ Hz, 2H), 2.43 (s, 3H), 2.13 (s, 3H), 1.96-1.88 (m, 2H), 1.43-1.33 (m, 2H), 0.97 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.7, 154.5, 152.0, 152.0, 146.6, 144.6, 135.7, 132.6, 131.9, 131.8, 127.7, 123.5, 43.8, 31.9, 21.3, 21.0, 19.9, 13.5; HRMS: calcd for $\text{C}_{18}\text{H}_{21}\text{N}_4\text{O}_2$ $[\text{M} + \text{H}]^+$ 325.1665, found 325.1662.

Acetic acid 2-(9-benzyl-9*H*-purin-6-yl)-5-methyl-phenyl ester (2h)



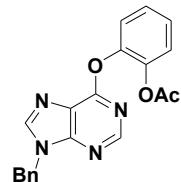
^1H NMR (400 MHz, CDCl_3) δ 9.03 (s, 1H), 8.15 (d, $J = 8.0$ Hz, 1H), 8.07 (s, 1H), 7.40-7.31 (m, 5H), 7.23 (d, $J = 8.0$ Hz, 1H), 7.08 (s, 1H), 5.47 (s, 2H), 2.44 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 154.6, 152.2, 152.1, 148.7, 144.3, 142.2, 135.1, 132.4, 131.5, 129.4, 128.6, 127.8, 126.9, 125.0, 124.4, 47.3, 21.4, 21.3; HRMS: calcd for $\text{C}_{21}\text{H}_{18}\text{N}_4\text{NaO}_2$ $[\text{M} + \text{Na}]^+$ 381.1327, found 381.1330.

Acetic acid 2-(9-butyl-9*H*-purin-6-yl)-4-methoxy-phenyl ester (2i)



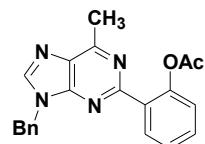
¹H NMR (400 MHz, CDCl₃) δ 9.01 (s, 1H), 8.08 (s, 1H), 7.73 (d, *J* = 3.2 Hz, 1H), 7.18 (d, *J* = 8.8 Hz, 1H), 7.05 (dd, *J* = 8.8, 3.2 Hz, 1H), 4.31 (t, *J* = 7.2 Hz, 2H), 3.87 (s, 3H), 2.14 (s, 3H), 1.97-1.89 (m, 2H), 1.44-1.35 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.9, 157.1, 154.2, 152.1, 151.9, 144.6, 142.3, 131.8, 128.6, 124.6, 117.1, 116.7, 55.7, 43.8, 31.9, 21.2, 19.9, 13.5; HRMS: calcd for C₁₈H₂₀N₄NaO₃ [M + Na]⁺ 363.1433, found 363.1428.

Acetic acid 2-(9-benzyl-9*H*-purin-6-yloxy)-phenyl ester (2j)



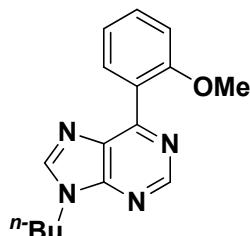
¹H NMR (400 MHz, CDCl₃) δ 8.54 (s, 1H), 8.00 (s, 1H), 7.39-7.25 (m, 9H), 5.46 (s, 2H), 2.03 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 168.3, 159.3, 153.3, 152.2, 143.7, 143.1, 142.6, 134.9, 129.2, 128.7, 127.9, 126.6, 126.5, 123.8, 123.7, 121.1, 47.7, 20.6. HRMS: calcd for C₂₀H₁₇N₄O₃ [M + H]⁺ 361.1301, found 361.1301.

Acetic acid 2-(9-benzyl-6-methyl-9*H*-purin-2-yl)-phenyl ester (2k)



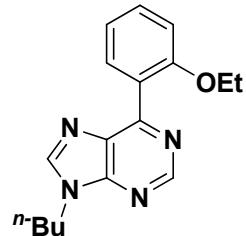
¹H NMR (400 MHz, CDCl₃) δ 8.22 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.99 (s, 1H), 7.49-4.45 (m, 1H), 7.41-7.33 (m, 4H), 7.31-7.29 (m, 2H), 7.16 (d, *J* = 8.0 Hz, 1H), 5.46 (s, 2H), 2.90 (s, 3H), 2.21 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.8, 158.9, 157.9, 149.0, 143.9, 135.9, 132.0, 131.4, 130.6, 129.1, 128.5, 127.8, 126.2, 123.7, 47.0, 21.2, 19.7; HRMS: calcd for C₂₁H₁₈N₄NaO₂ [M + Na]⁺ 381.1327, found 381.1327.

9-butyl-6-(2-methoxy-phenyl)-9*H*-purine (3a)



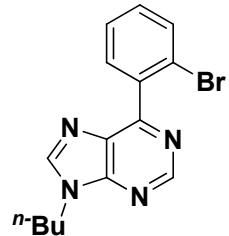
¹H NMR (400 MHz, CDCl₃) δ 9.06 (s, 1H), 8.05 (s, 1H), 7.65 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.47 (td, *J* = 8.4, 1.6 Hz, 1H), 7.13-7.08 (m, 2H), 4.31 (t, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 1.98-1.91 (m, 2H), 1.46-1.37 (m, 2H), 0.99 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 157.5, 156.5, 152.2, 151.7, 144.3, 132.6, 131.7, 131.3, 124.9, 120.7, 111.9, 55.9, 43.7, 31.9, 19.9, 13.5; HRMS: calcd for C₁₆H₁₉N₄O [M + H]⁺ 383.1559, found 383.1556.

6-(2-Ethoxy-phenyl)-9-butyl-9*H*-purine (3b)



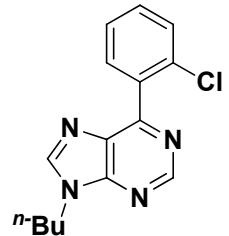
¹H NMR (400 MHz, CDCl₃) δ 9.04 (s, 1H), 8.05 (s, 1H), 7.64 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.46-7.42 (m, 1H), 7.11-7.05 (m, 2H), 4.31 (t, *J* = 7.2 Hz, 2H), 4.10 (q, *J* = 7.2 Hz, 2H), 1.98-1.90 (m, 2H), 1.45-1.36 (m, 2H), 1.22 (t, *J* = 7.2 Hz, 3H), 0.98 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 156.9, 156.7, 152.2, 151.5, 144.0, 132.5, 131.5, 131.2, 125.2, 120.6, 113.0, 64.3, 43.7, 31.9, 19.9, 14.7, 13.5; HRMS: calcd for C₁₇H₂₁N₄O [M + H]⁺ 297.1715, found 297.1717.

6-(2-bromophenyl)-9-butyl-9*H*-purine (3c)



¹H NMR (400 MHz, CDCl₃) δ 9.09 (s, 1H), 8.11 (s, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.38-7.34 (m, 1H), 4.34 (t, *J* = 7.2 Hz, 2H), 2.00-1.93 (m, 2H), 1.47-1.38 (m, 2H), 1.00 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 157.2, 152.1, 152.0, 144.9, 136.5, 133.6, 132.0, 131.7, 130.9, 127.4, 122.0, 43.9, 31.9, 19.9, 13.5; HRMS: calcd for C₁₅H₁₅BrN₄Na [M + Na]⁺ 353.0378, found 353.0370.

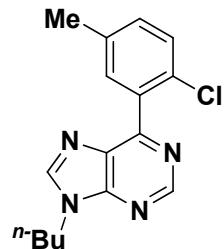
6-(2-chlorophenyl)-9-butyl-9*H*-purine (3d)



¹H NMR (400 MHz, CDCl₃) δ 9.08 (s, 1H), 8.10 (s, 1H), 7.68-7.65 (m, 1H), 7.57-7.54 (m, 1H), 7.44-7.41

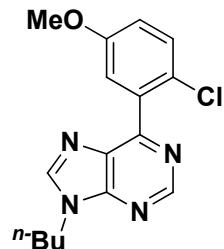
(m, 2H), 4.32 (t, $J = 7.2$ Hz, 2H), 1.98-1.91 (m, 2H), 1.46-1.36 (m, 2H), 0.98 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.0, 152.2, 151.9, 144.9, 134.6, 132.9, 132.3, 131.7, 130.8, 130.4, 126.8, 43.8, 31.9, 19.9, 13.5; HRMS: calcd for $\text{C}_{15}\text{H}_{15}\text{ClN}_4\text{Na}$ [$\text{M} + \text{Na}^{+}$] 309.0883, found 309.0880.

6-(2-chloro-5-methylphenyl)-9-butyl-9*H*-purine (3e)



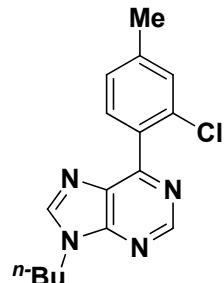
^1H NMR (400 MHz, CDCl_3) δ 9.07 (s, 1H), 8.10 (s, 1H), 7.45 (d, $J = 1.6$ Hz, 1H), 7.42 (d, $J = 8.0$ Hz, 1H), 7.23 (dd, $J = 8.4, 1.6$ Hz, 1H), 4.32 (t, $J = 7.2$ Hz, 2H), 2.38 (s, 3H), 1.98-1.91 (m, 2H), 1.45-1.36 (m, 2H), 0.98 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.3, 152.2, 151.9, 144.9, 136.8, 134.3, 132.3, 132.1, 131.6, 130.1, 129.9, 43.8, 31.9, 20.9, 19.9, 13.5; HRMS: calcd for $\text{C}_{16}\text{H}_{18}\text{ClN}_4$ [$\text{M} + \text{H}^{+}$] 301.1220, found 301.1222.

6-(2-chloro-5-methoxyphenyl)-9-butyl-9*H*-purine (3f)



^1H NMR (400 MHz, CDCl_3) δ 9.08 (s, 1H), 8.11 (s, 1H), 7.44 (d, $J = 8.8$ Hz, 1H), 7.17 (d, $J = 3.2$ Hz, 1H), 6.98 (dd, $J = 8.8, 3.2$ Hz, 1H), 4.32 (t, $J = 7.2$ Hz, 2H), 3.82 (s, 3H), 1.99-1.91 (m, 2H), 1.46-1.37 (m, 2H), 0.99 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.2, 155.9, 152.2, 151.9, 145.0, 135.2, 132.2, 131.2, 124.3, 117.1, 116.5, 55.6, 43.8, 31.9, 19.9, 13.5; HRMS: calcd for $\text{C}_{16}\text{H}_{18}\text{ClN}_4\text{O}$ [$\text{M} + \text{H}^{+}$] 317.1169, found 317.1162.

6-(2-chloro-4-methylphenyl)-9-butyl-9*H*-purine (3g)



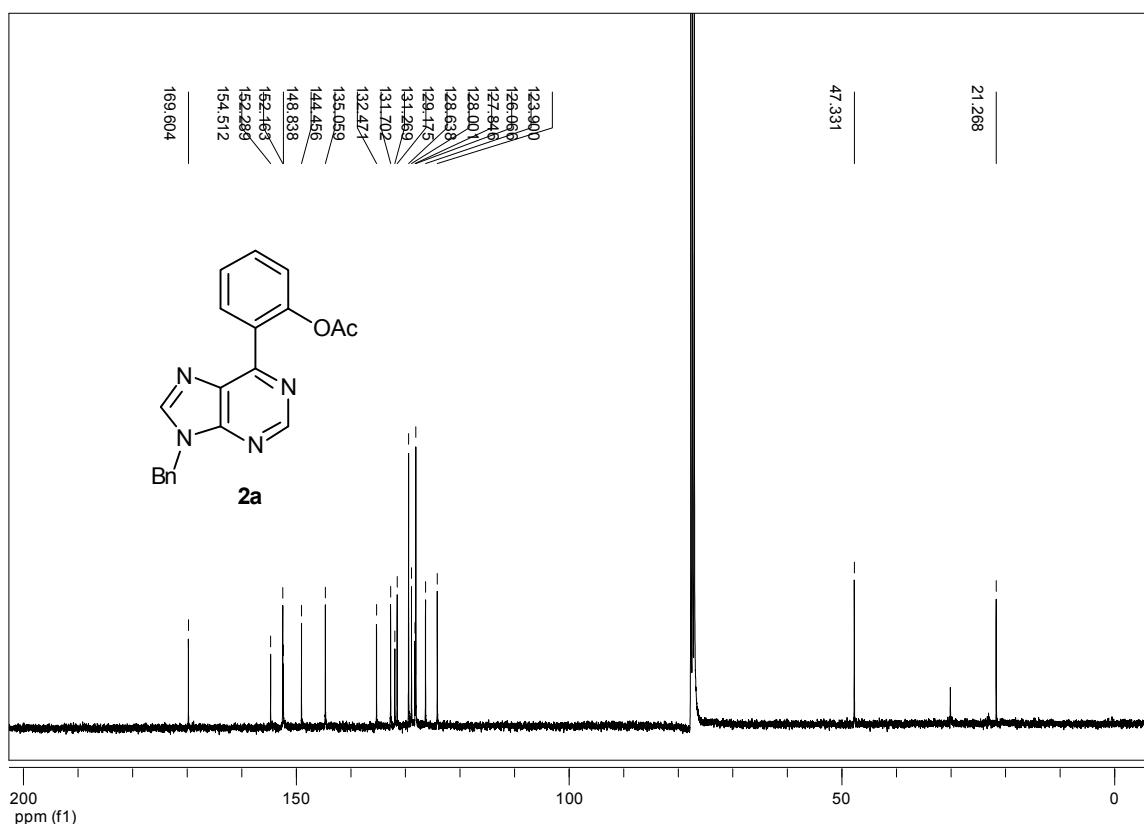
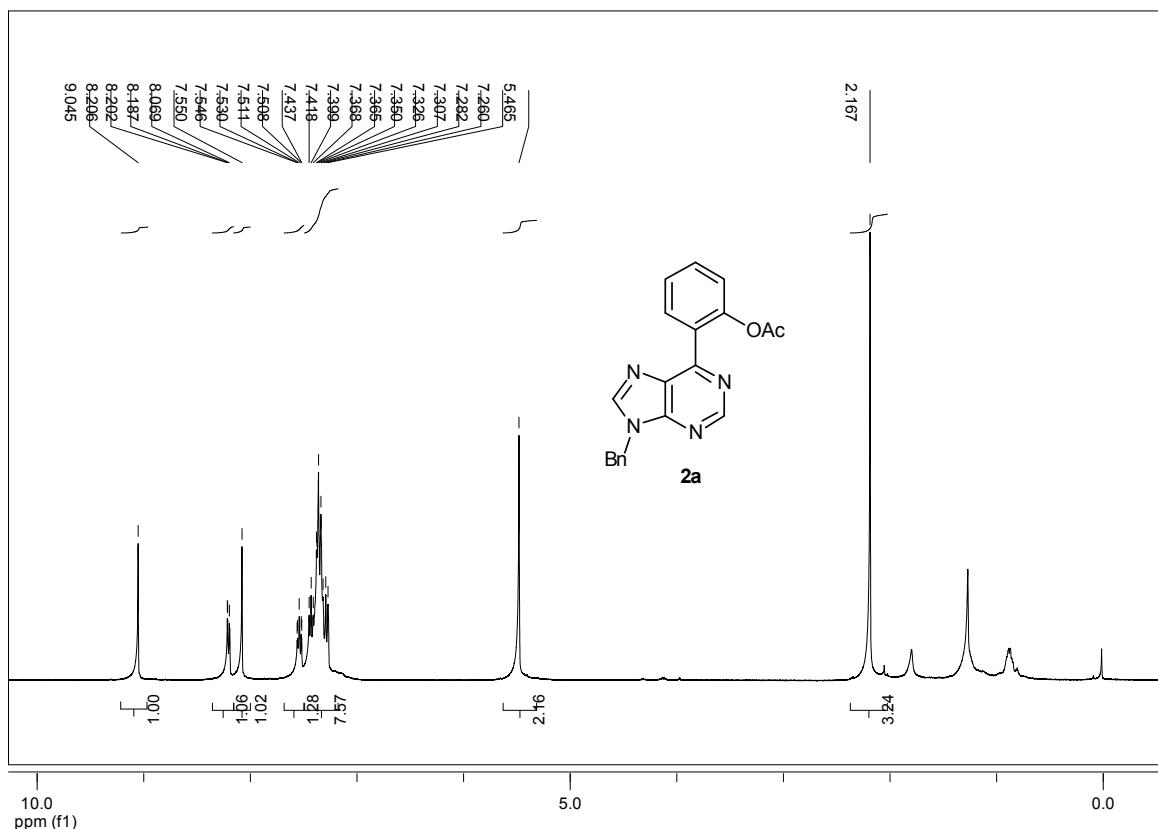
^1H NMR (400 MHz, CDCl_3) δ 9.07 (s, 1H), 8.08 (s, 1H), 7.57 (d, $J = 8.0$ Hz, 1H), 7.38 (s, 1H), 7.22 (d, $J =$

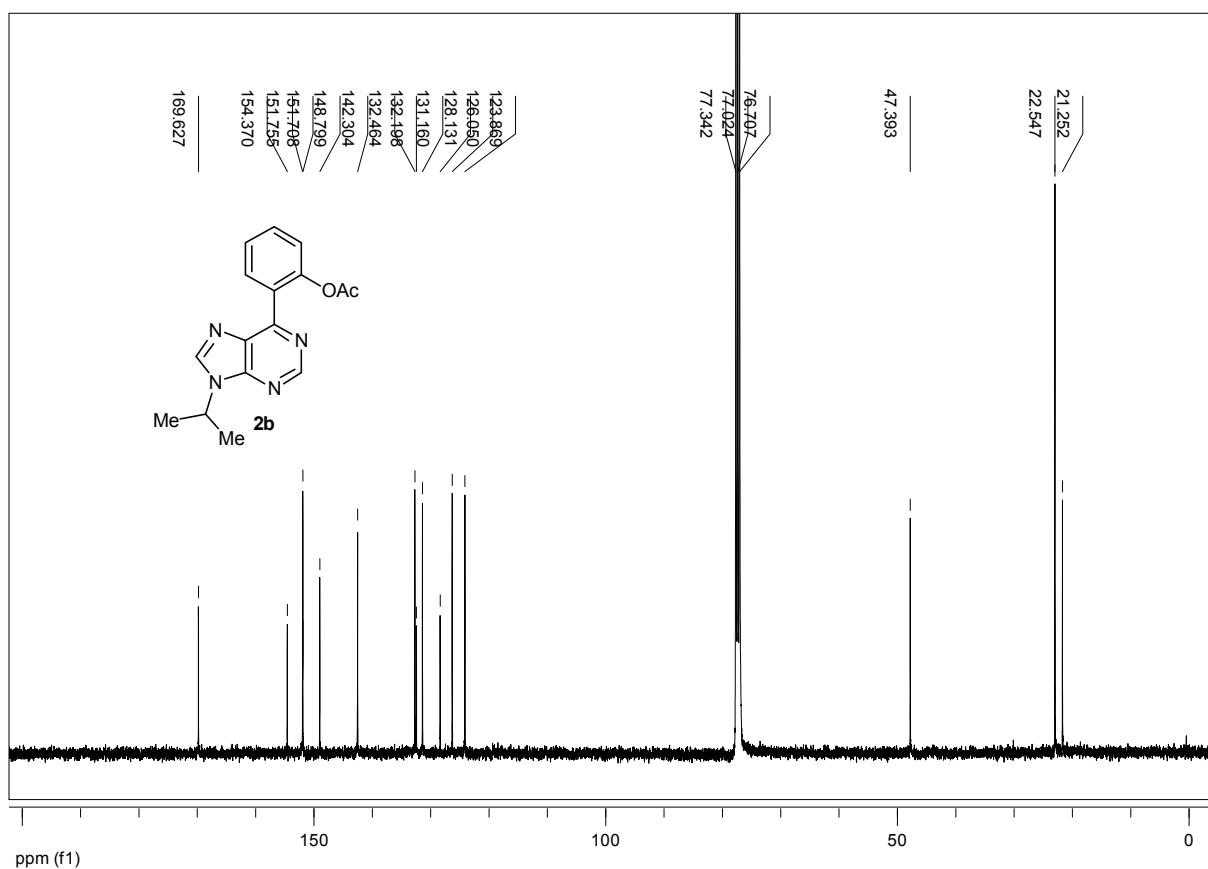
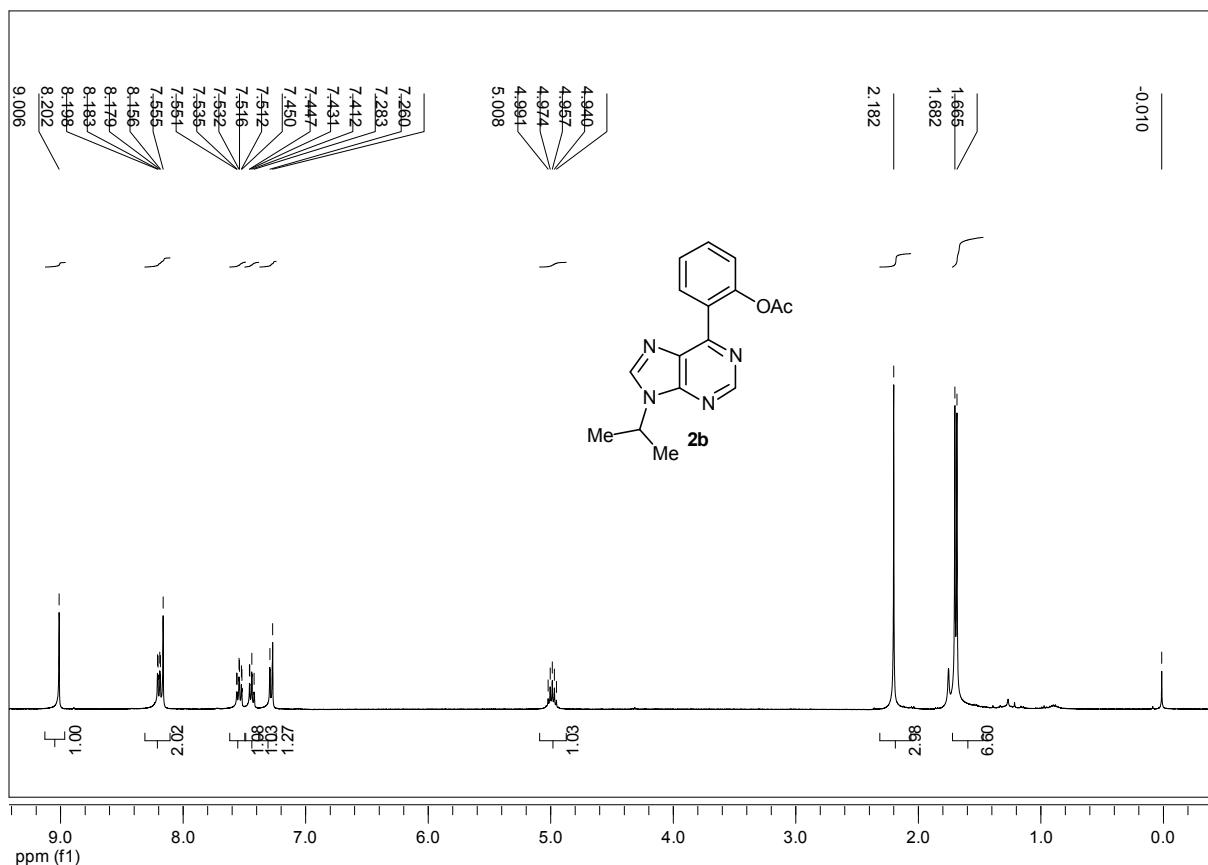
8.0 Hz, 1H), 4.31 (t, J = 7.2 Hz, 2H), 2.40 (s, 3H), 1.98-1.91 (m, 2H), 1.46-1.36 (m, 2H), 0.98 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.2, 152.2, 151.9, 144.8, 141.3, 132.6, 132.3, 131.6, 131.6, 130.9, 127.7, 43.8, 31.9, 21.1, 19.9, 13.5; HRMS: calcd for $\text{C}_{16}\text{H}_{18}\text{ClN}_4$ [$\text{M} + \text{H}^+$] 301.1220, found 301.1215.

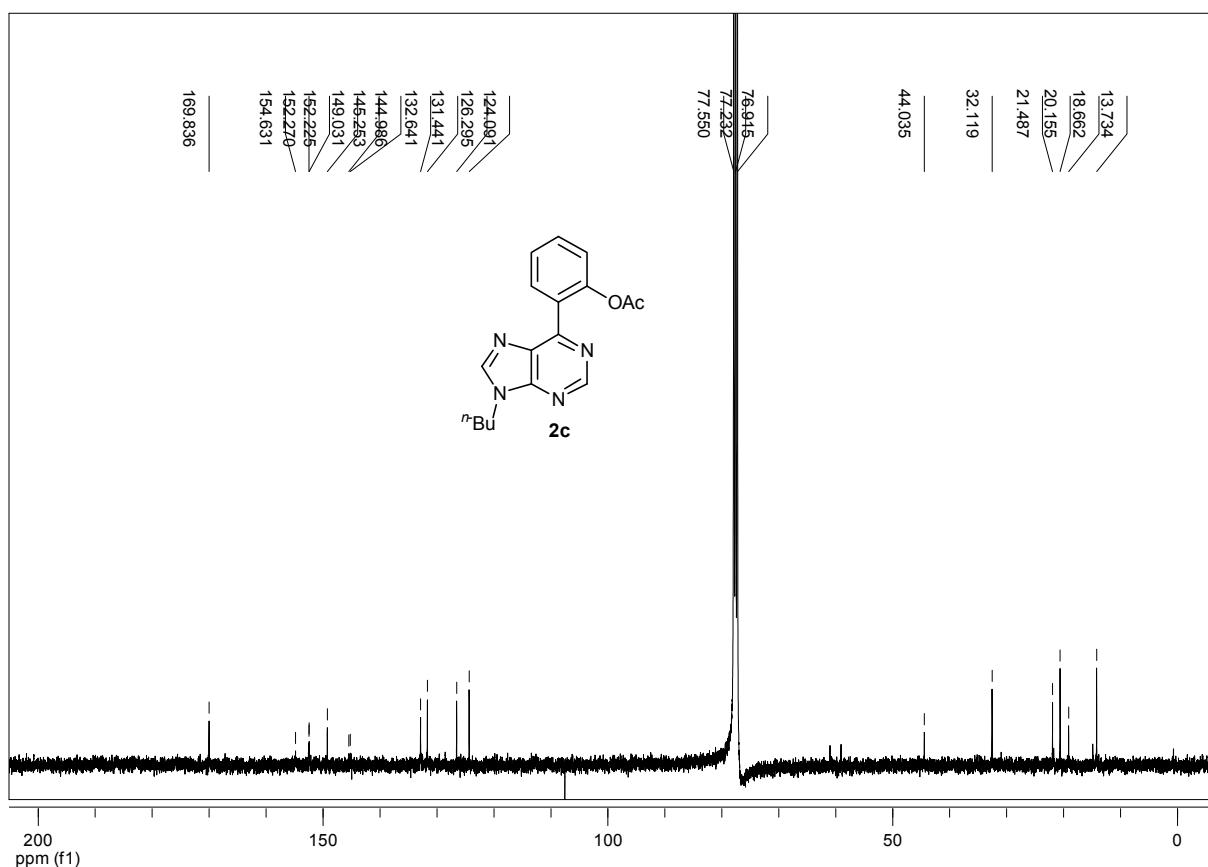
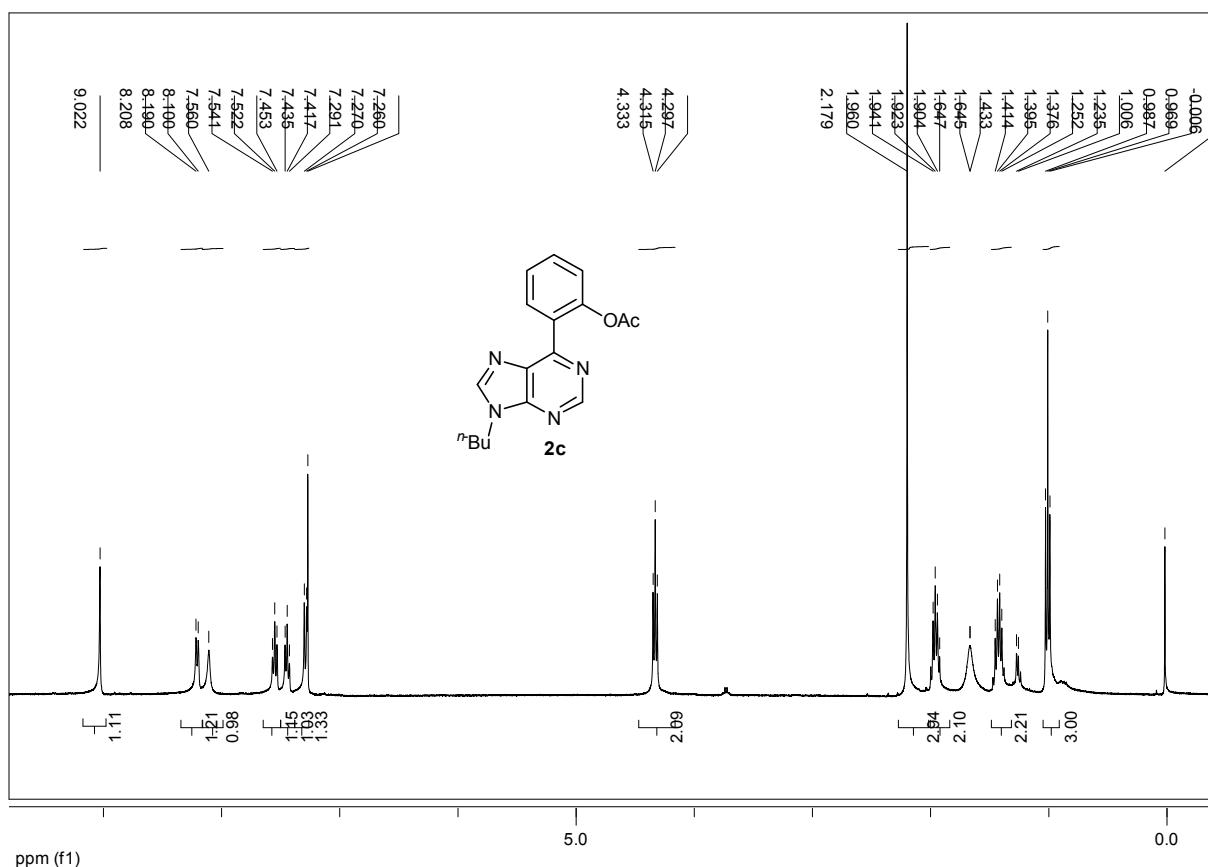
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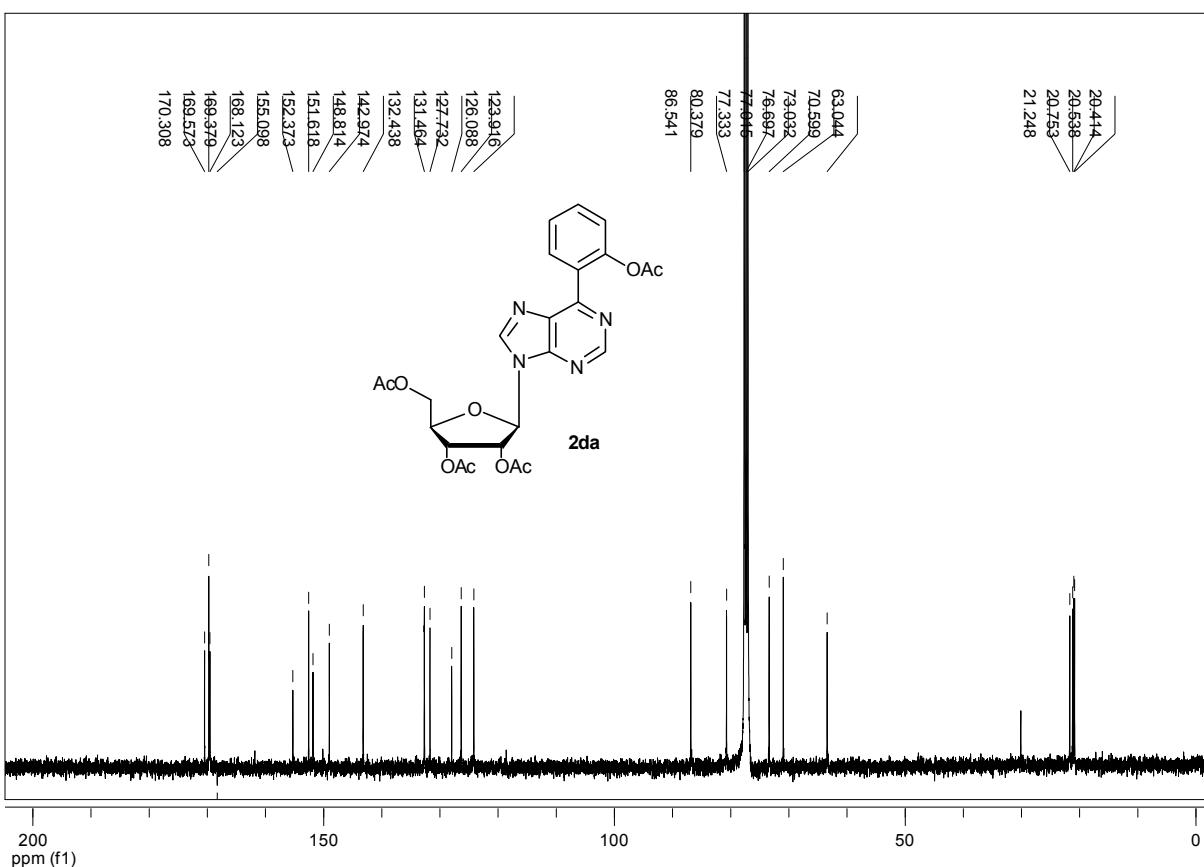
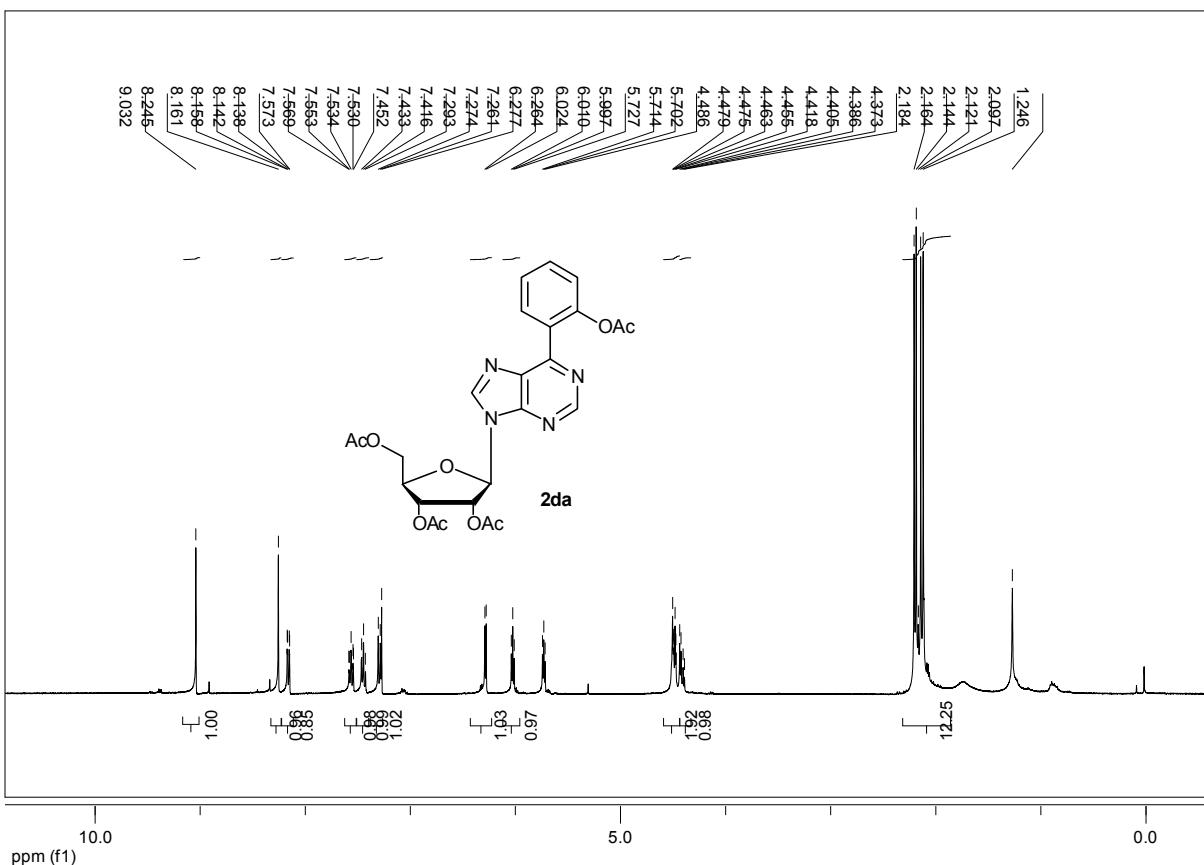
1. a) I. Černá, R. Pohl, B. Klepetářová and M. Hocek, *J. Org. Chem.*, **2008**, *73*, 9048; b) M. K. Lakshman, J. H. Hilmer, J. Q. Martin, J. C. Keeler, Y. Q. V. Dinh, F. N. Ngassa and L.;M. Russon, *J. Am. Chem. Soc.*, **2001**, *123*, 779.
2. L.-K. Huang, Y.-C. Cherng, Y.-R. Cheng, J.-P. Jang, Y.-L. Chao and Y.-J. Cherng, *Tetrahedron*, **2007**, *63*, 5323.
3. G. R. Qu, Z. J. Mao, H. Y. Niu, D. C. Wang, C. Xia and H. M. Guo, *Org. Lett.*, **2009**, *11*, 1745.

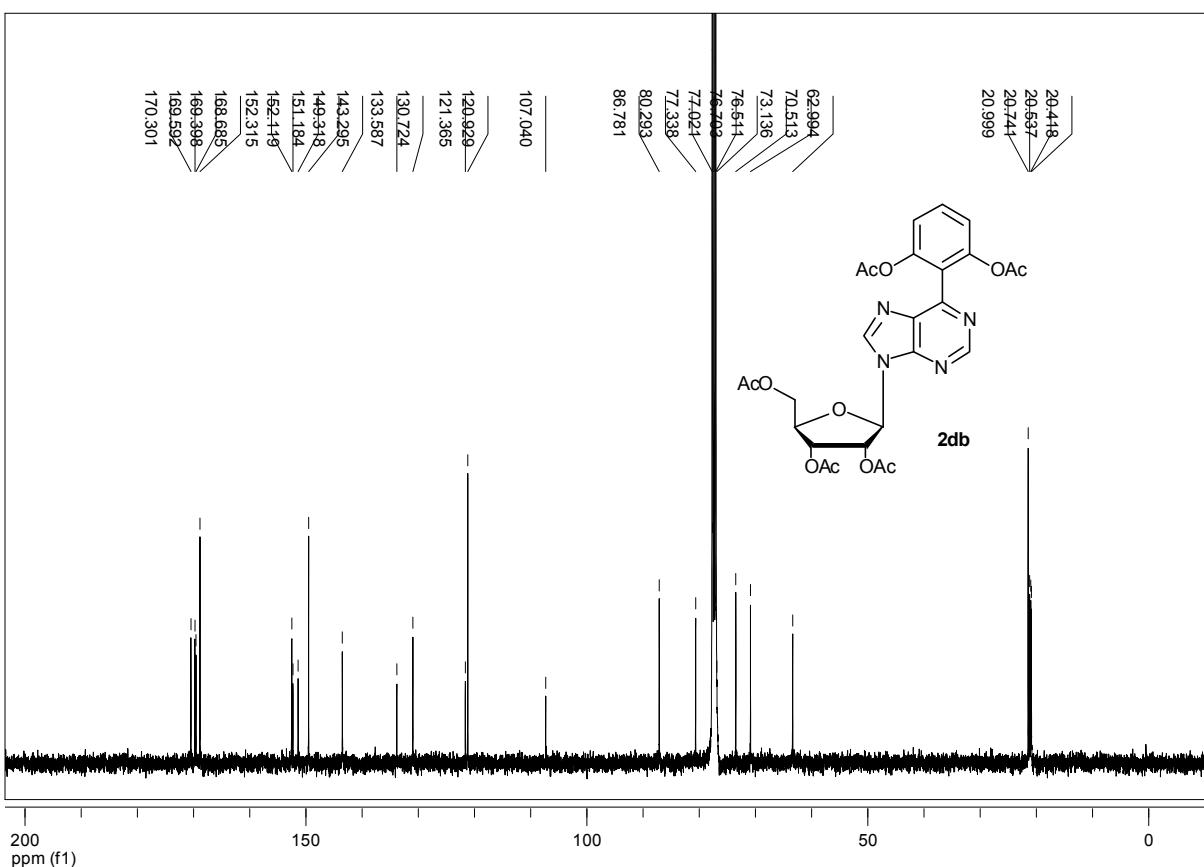
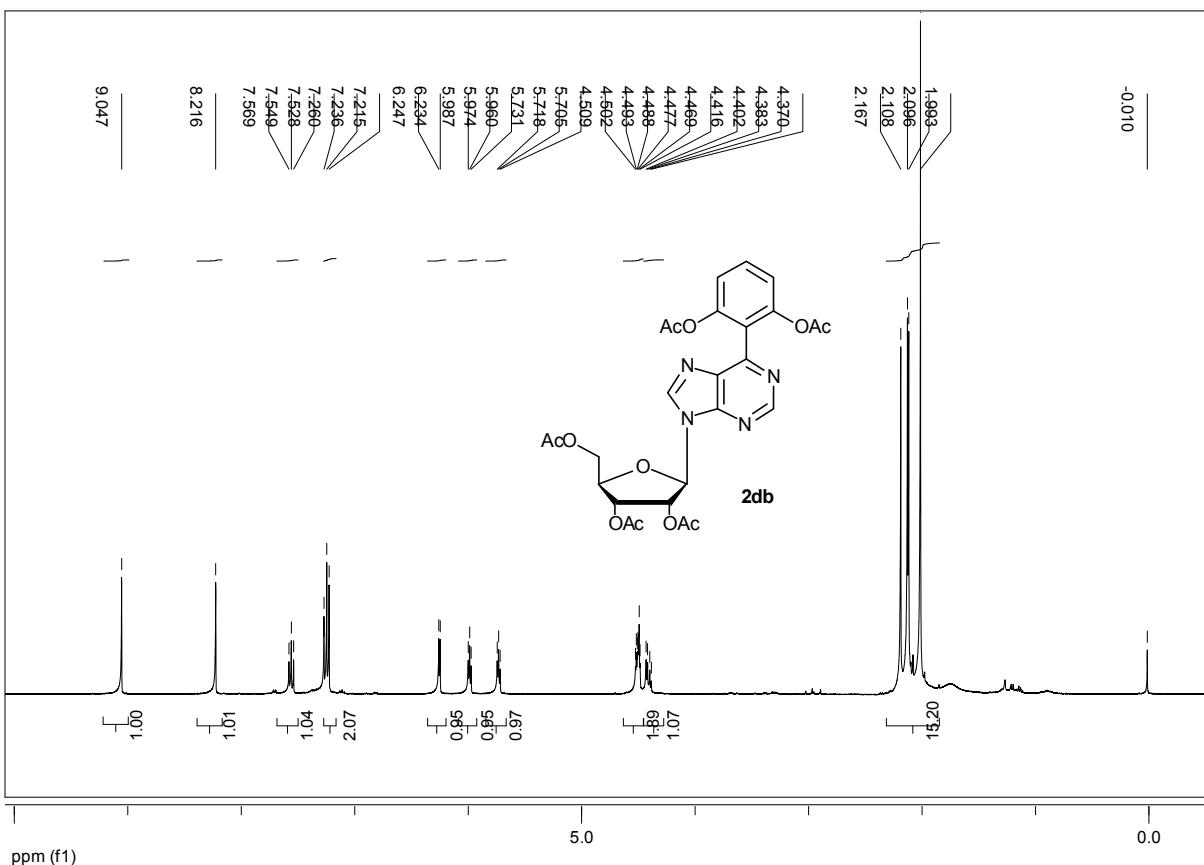
Copies of ^1H and ^{13}C NMR spectra

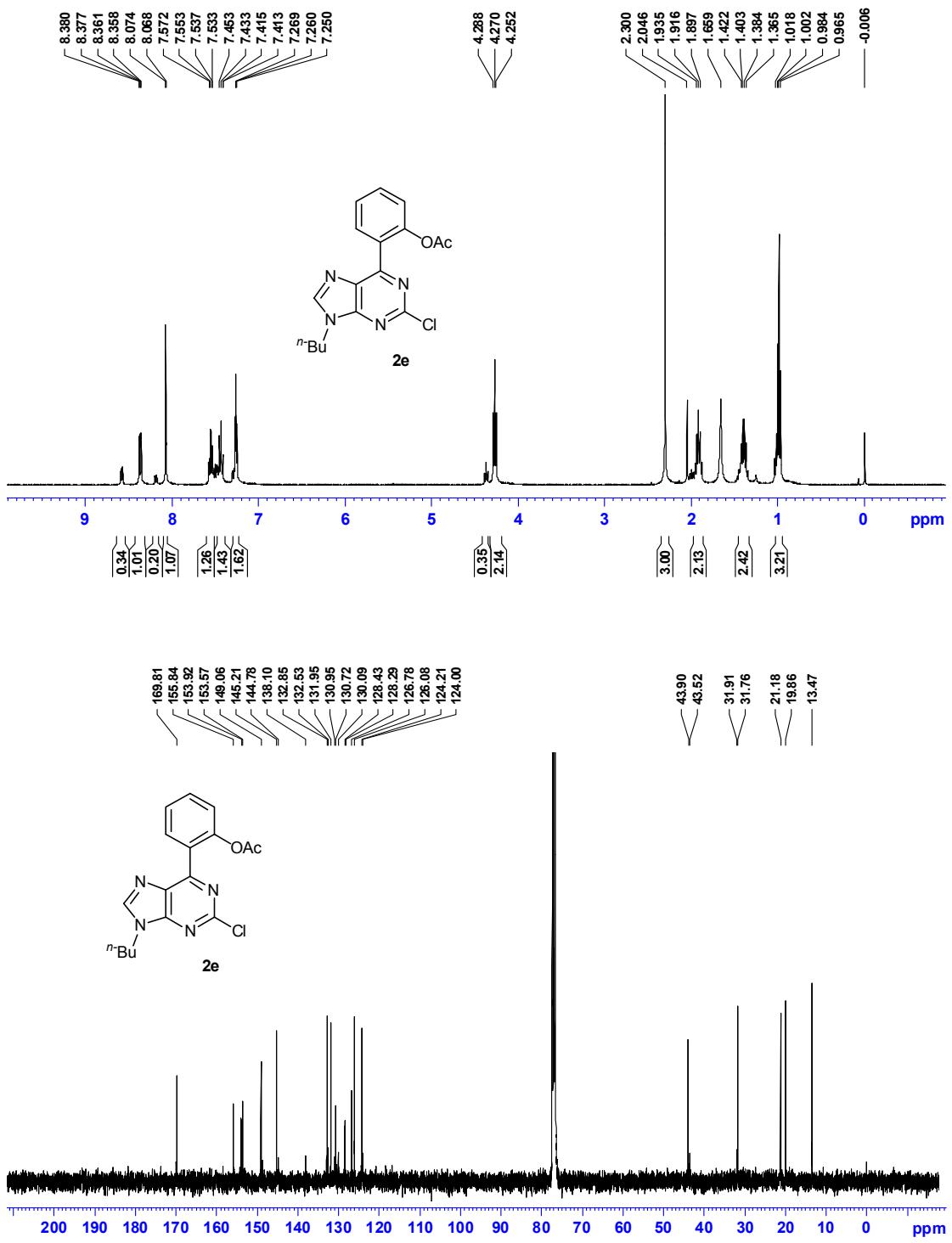


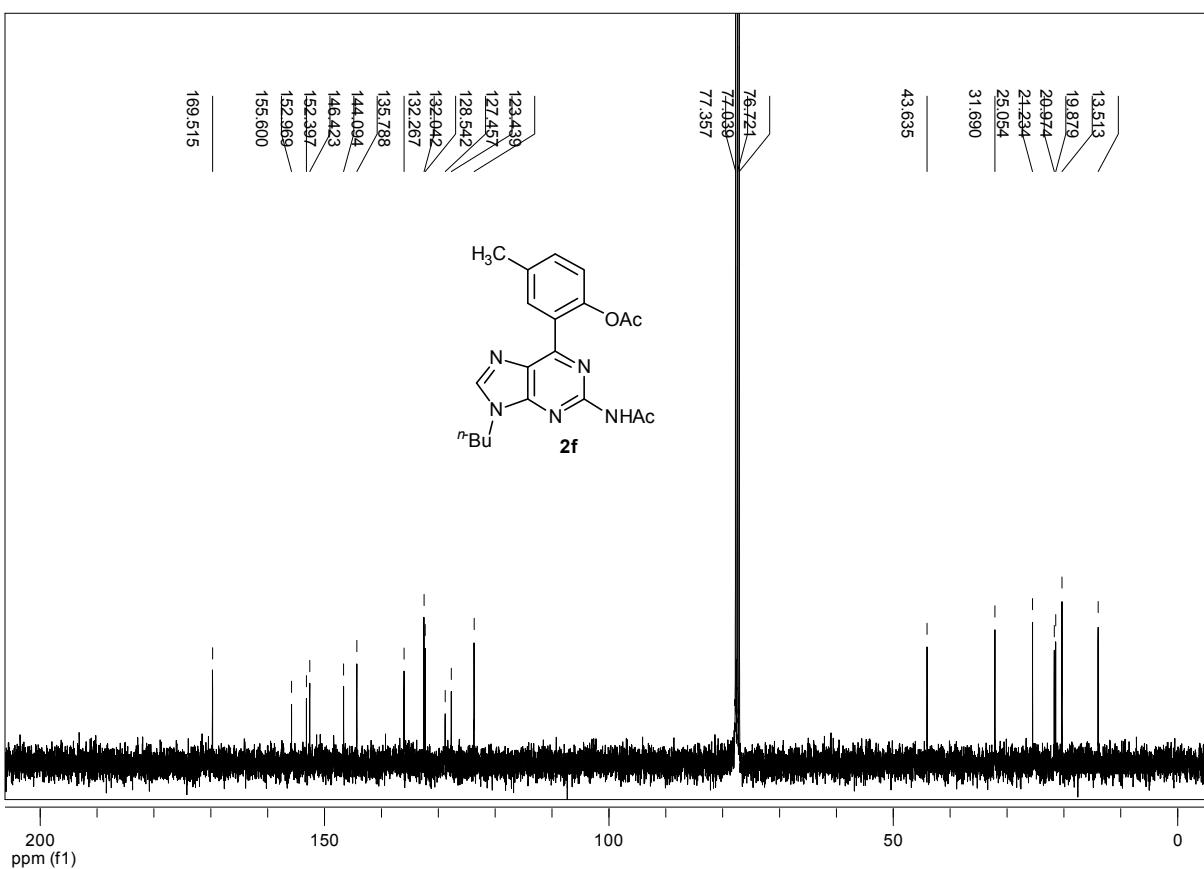
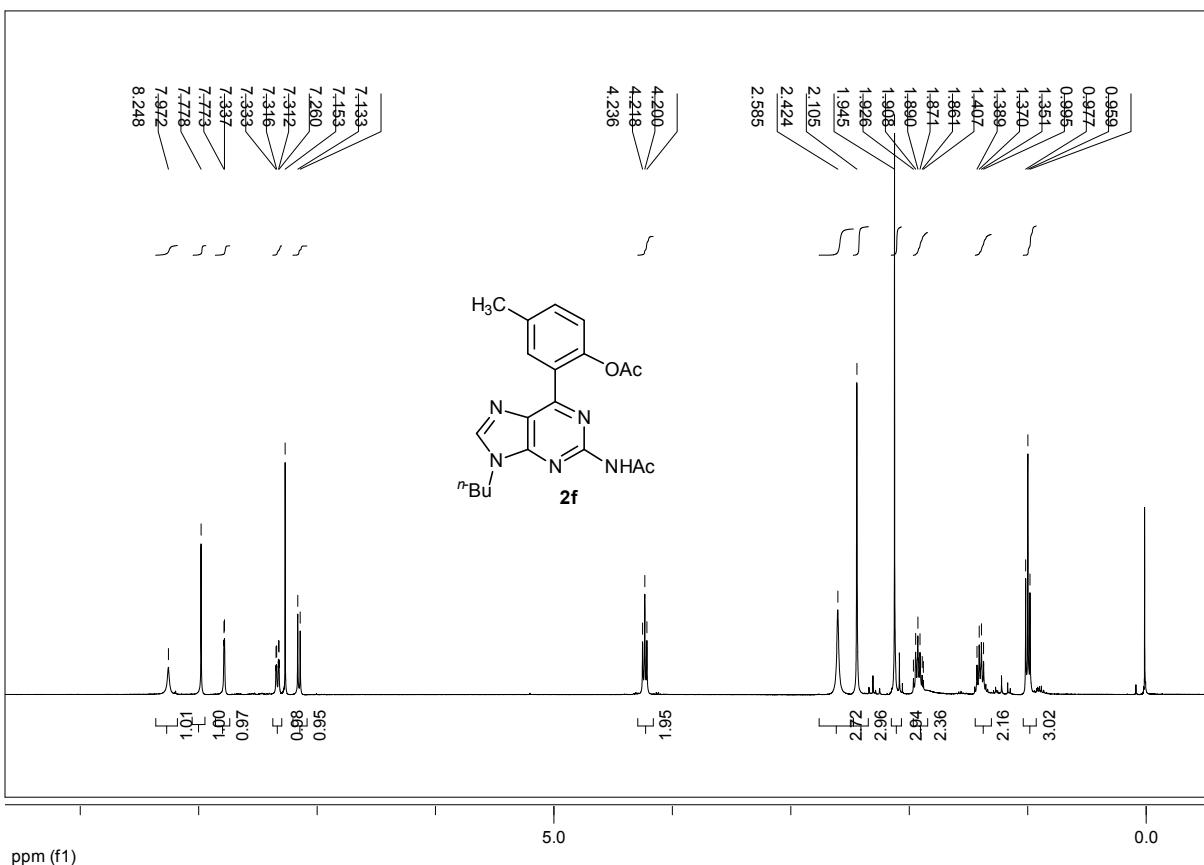


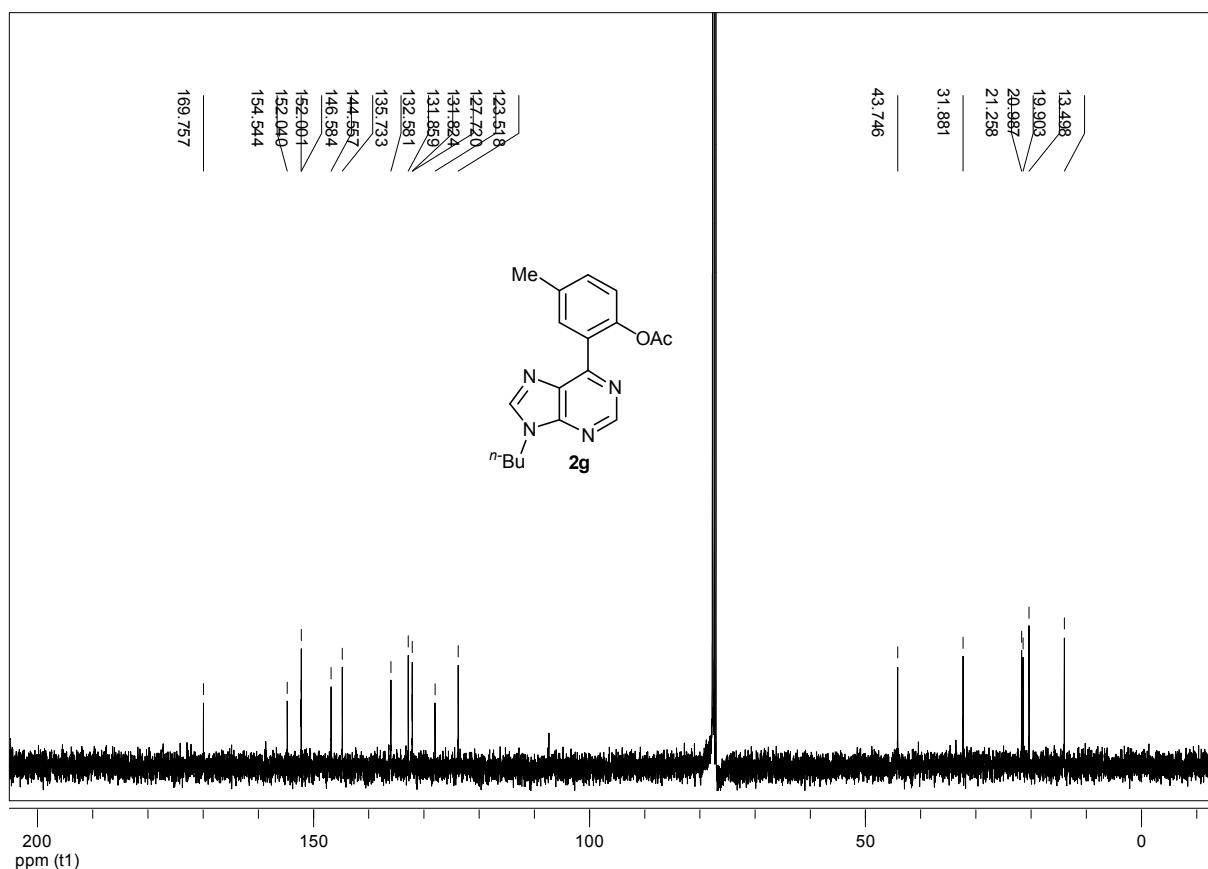
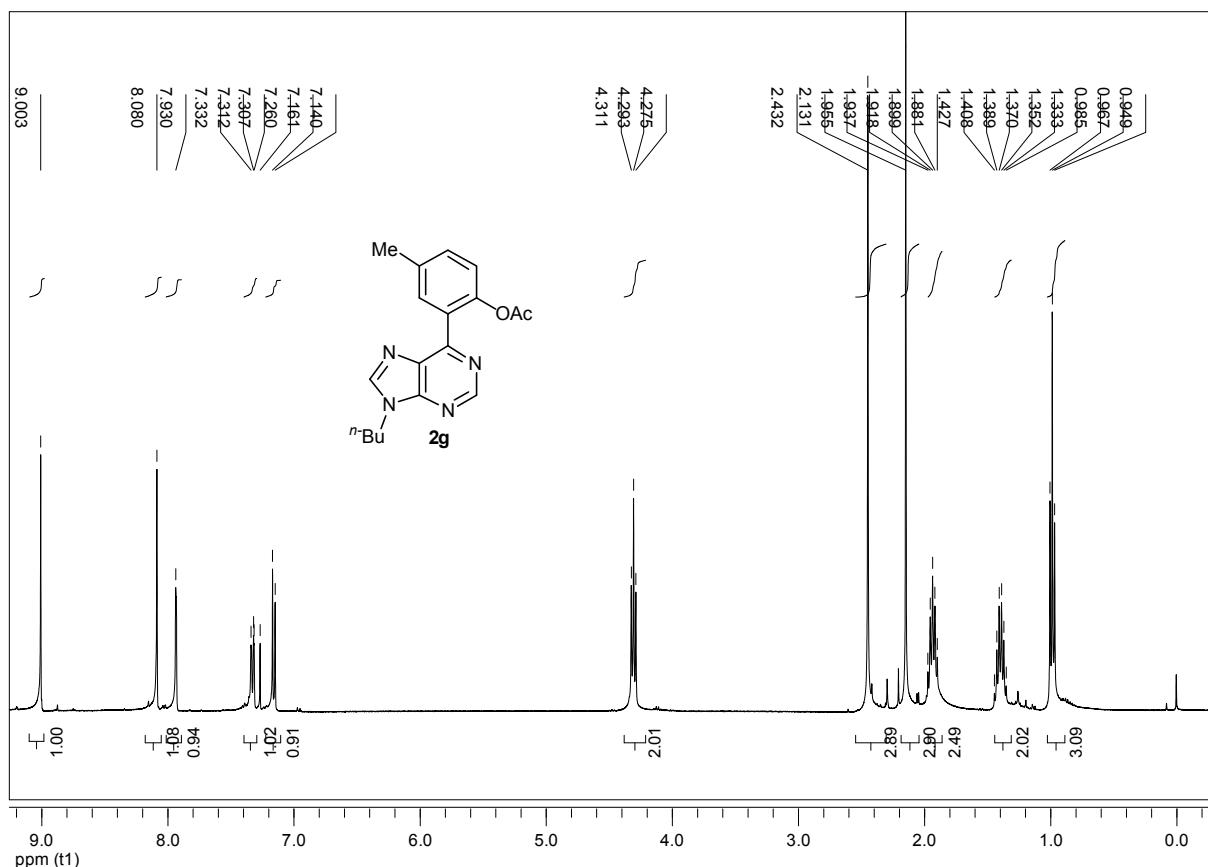


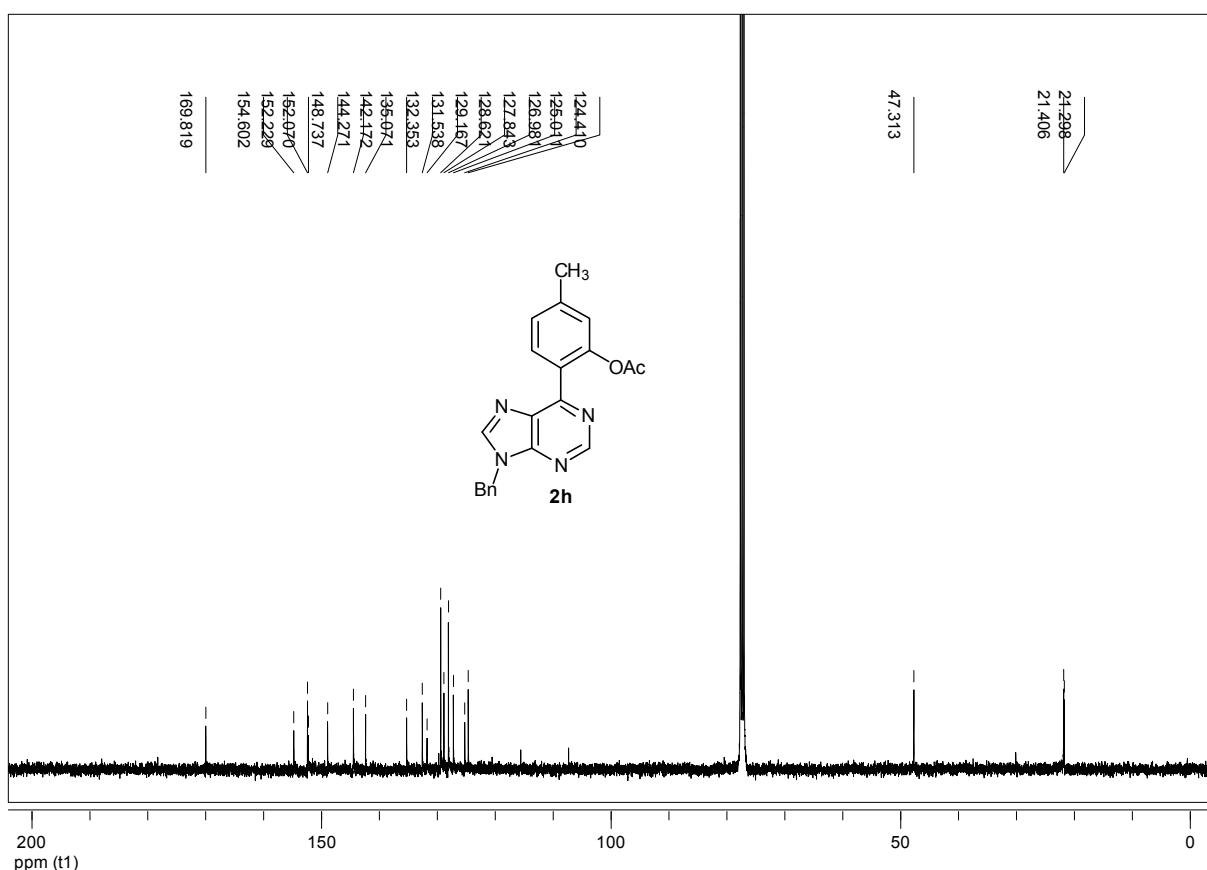
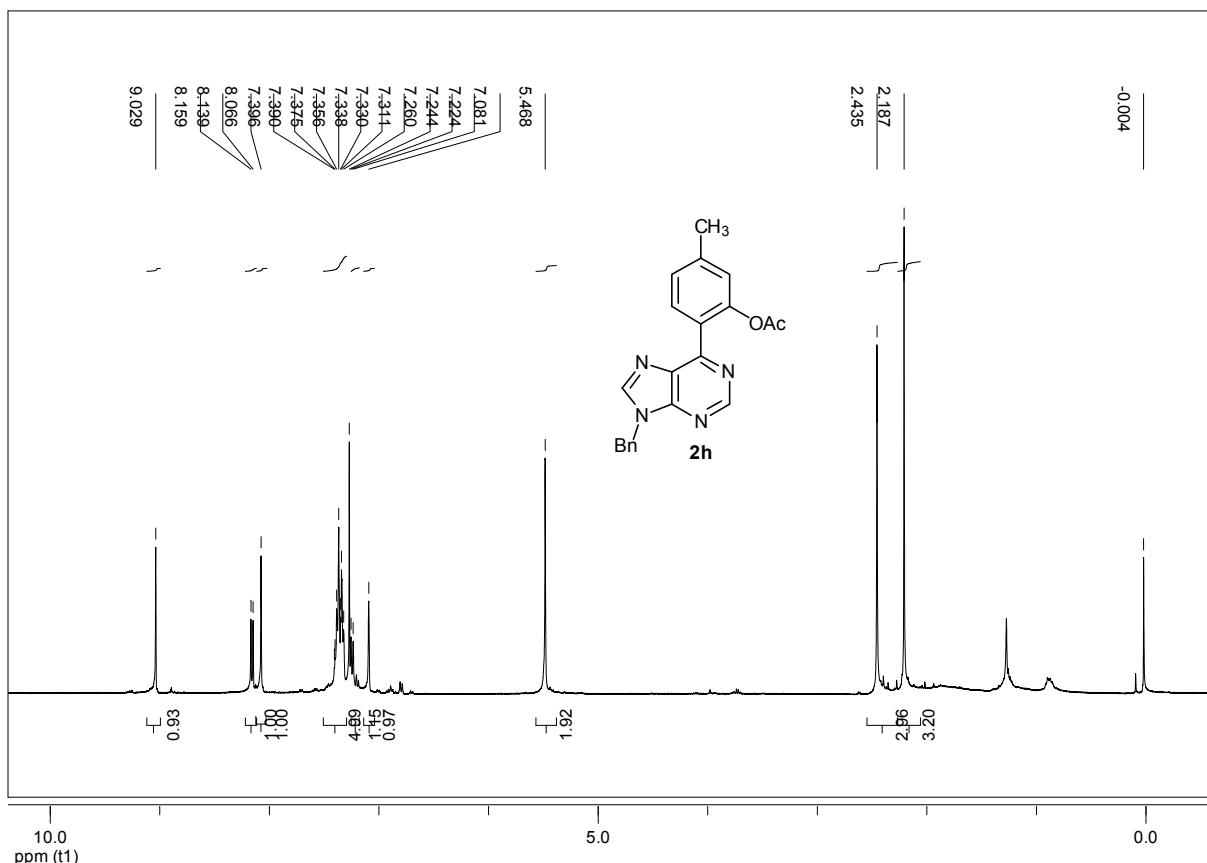


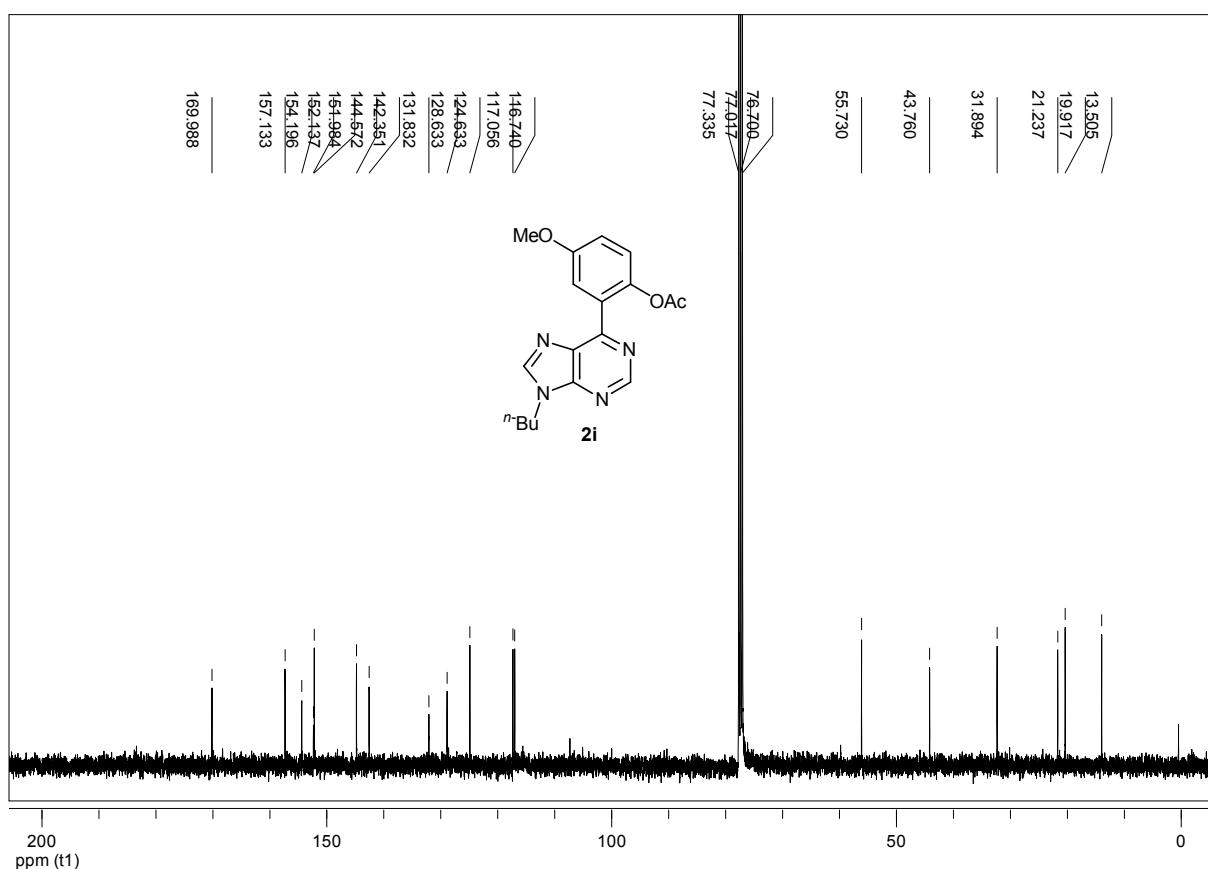
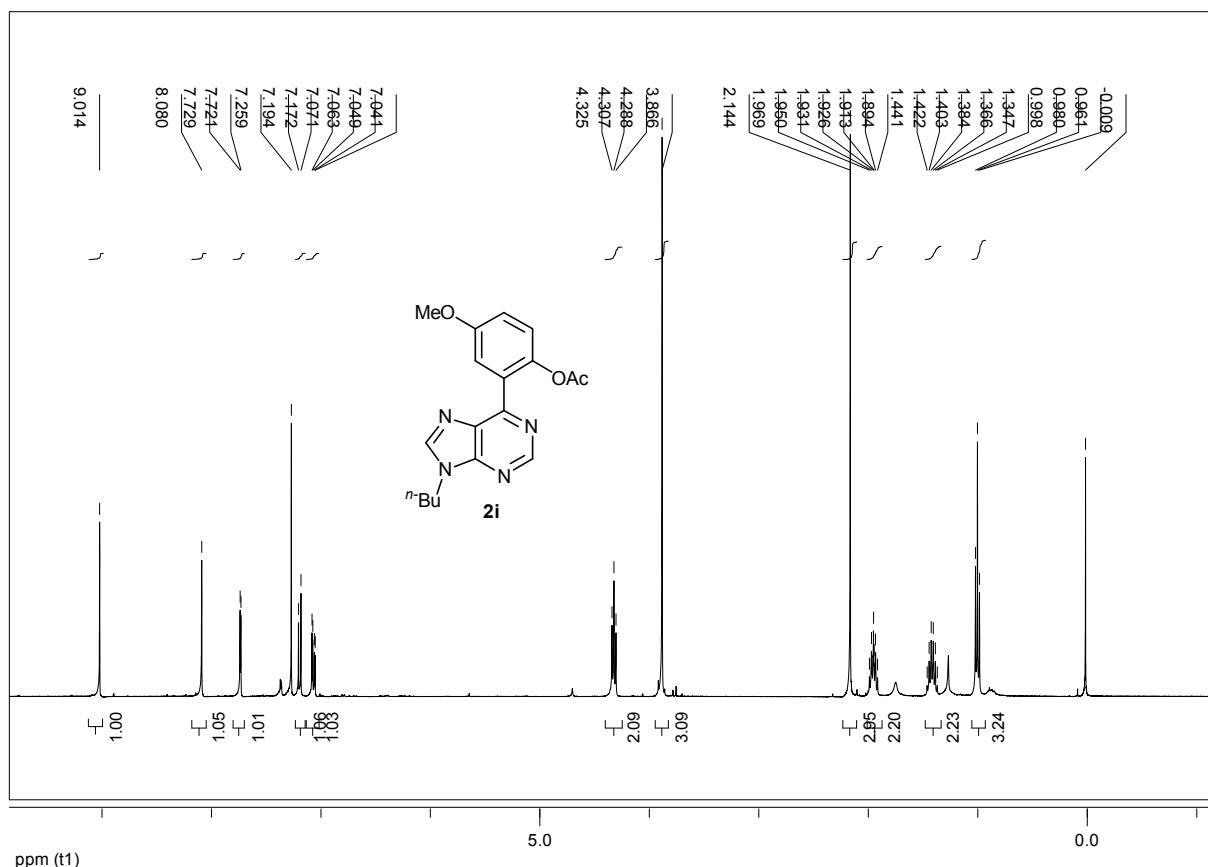


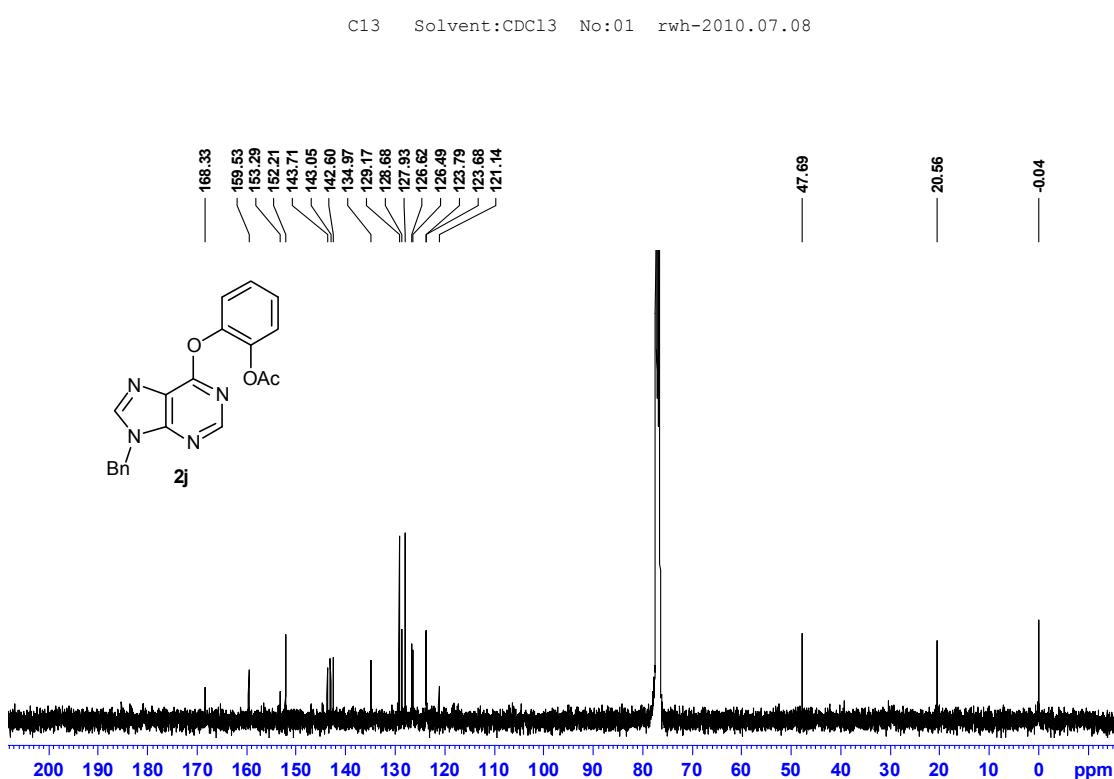
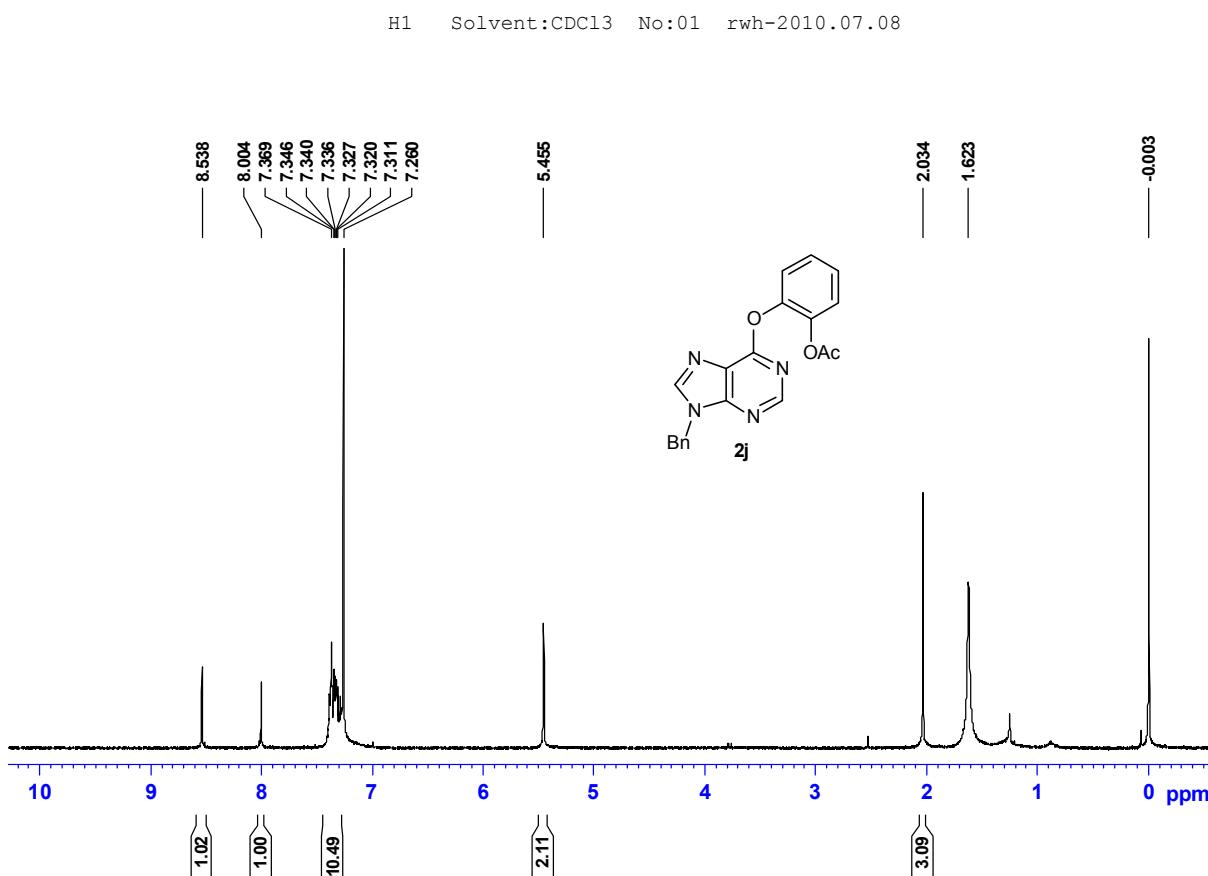


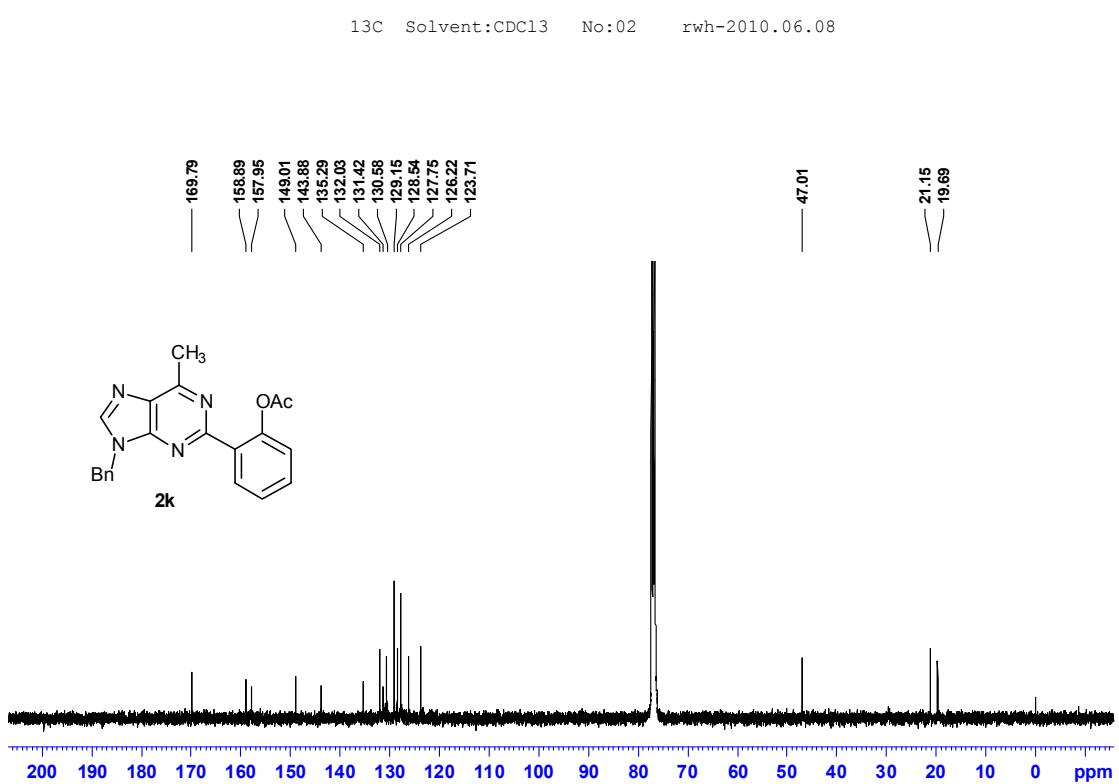
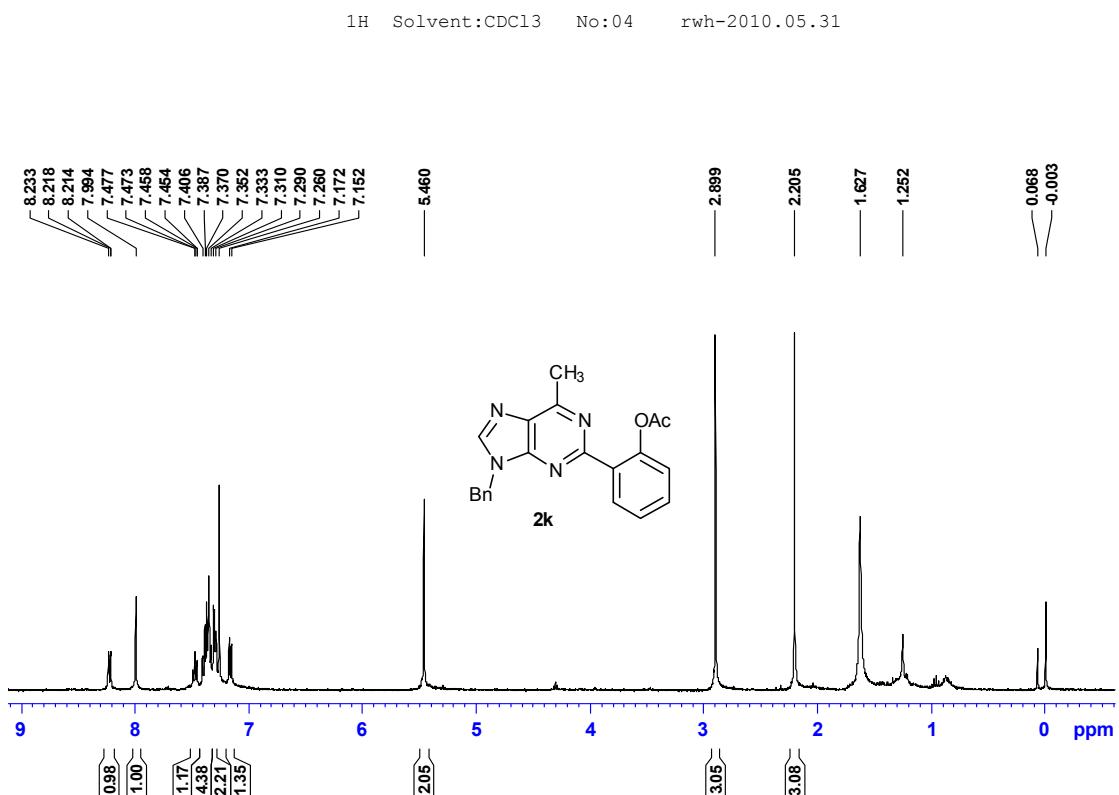




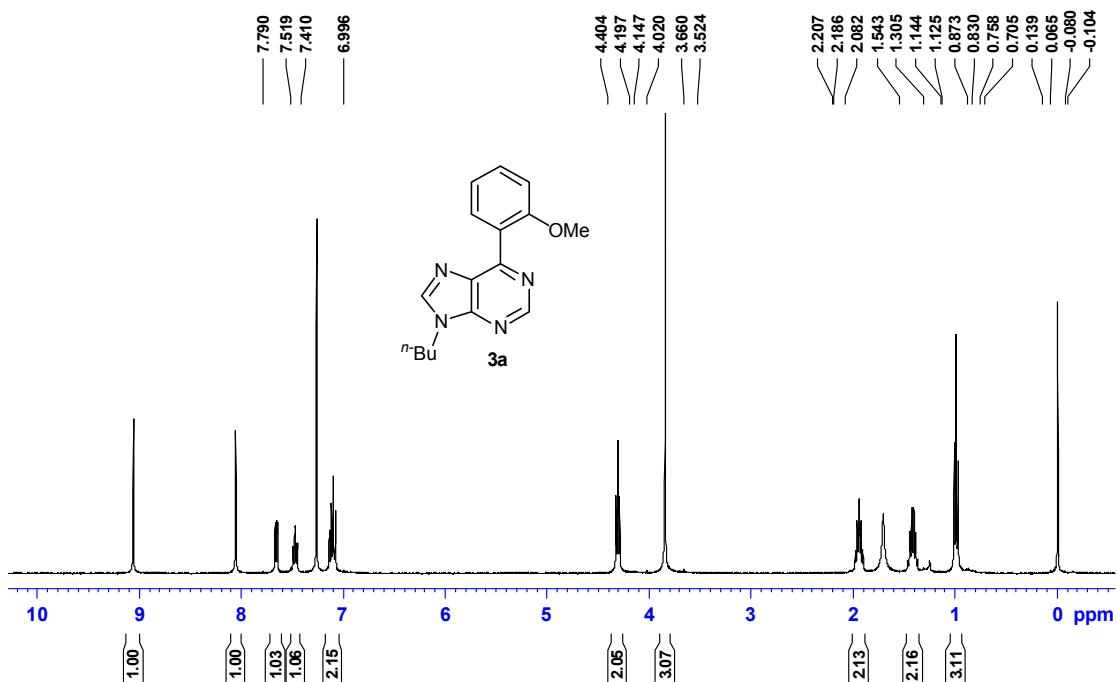




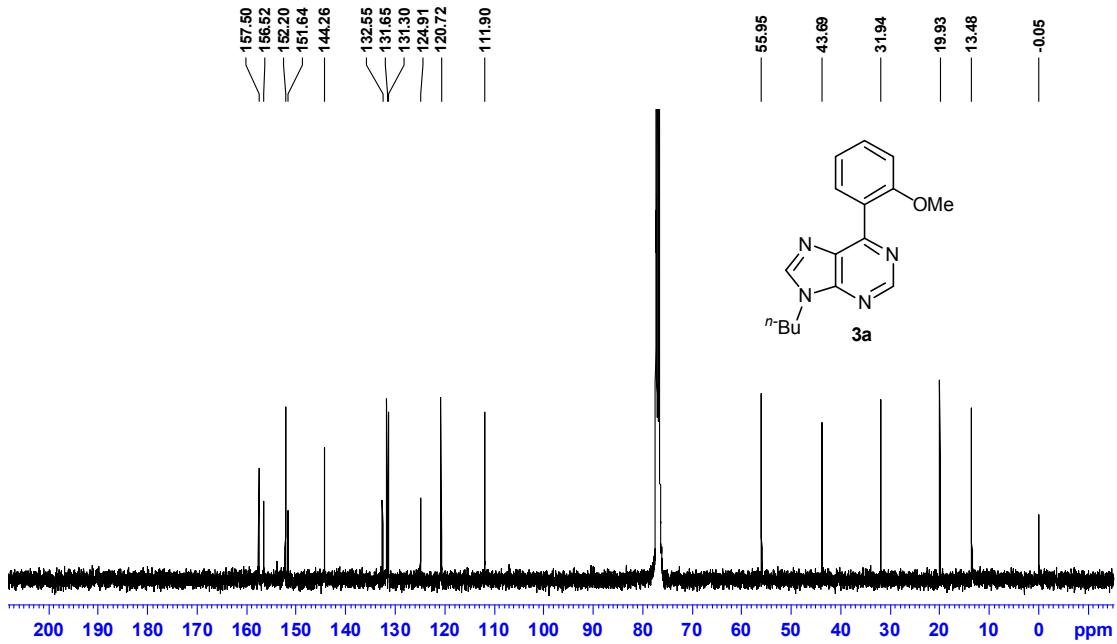


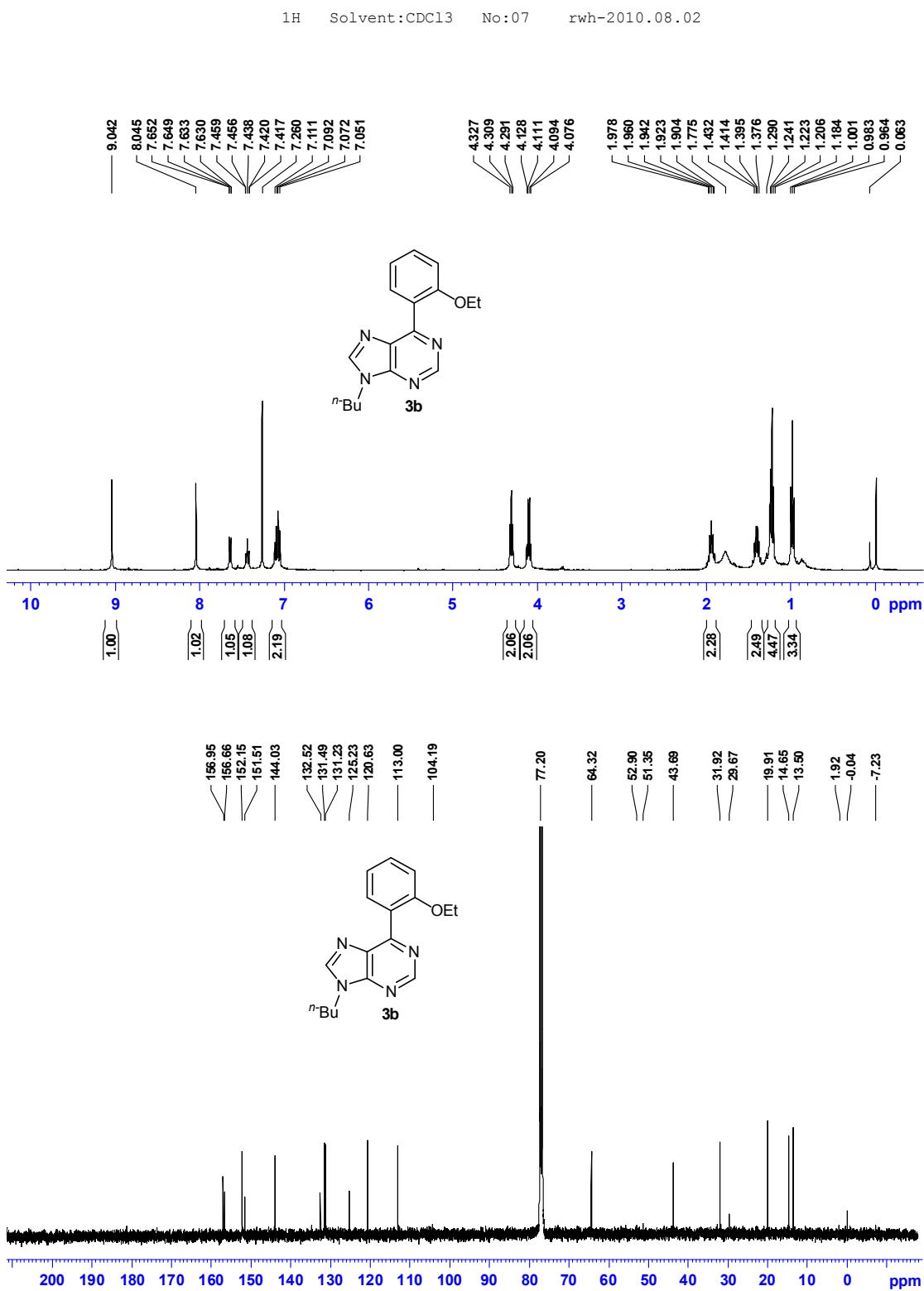


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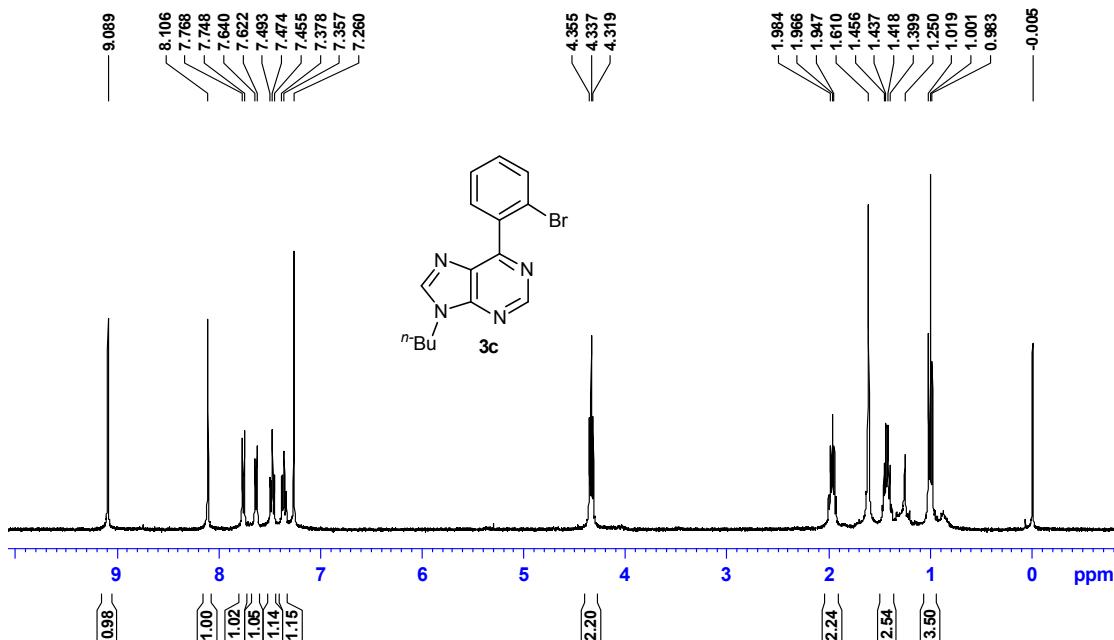


C13 Solvent:CDCl₃ No:02 rwh-2010.07.08





1H Solvent:CDCl₃ No:05 rwh-2010.05.31



13C Solvent:CDCl₃ No:23 (01) rwh-2010.06.11

