Synthesis of *N*² 2'-Deoxyguanosine Adducts Formed by 1-Nitropyrene

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Supporting Information

General procedures

All starting materials, reagents and solvents were of commercial grade and used as such unless otherwise specified. Anhydrous solvents were purchased from Aldrich and THF was dried by standard methods. NMR (¹H and ¹³C) spectra were recorded on a Bruker AC-400 spectrometer. Samples prepared for NMR analysis were dissolved in CDCl₃. Chemical shifts are reported in δ ppm relative to TMS in the proton spectra and to the deuterated solvent in the carbon spectra. LCMS (Electrospray ionization) was recorded on a Micromass Quattro II triple quadrupole mass spectrometer in acetonitrile mobile phase and the cone voltage set to 30 V. High resolution MS spectra were obtained either from Nebraska Center for Mass Spectrometry, University of Nebraska, Lincoln or Yale Cancer Center Mass Spectrometry Source. Thin-layer chromatography (TLC) was performed on silica gel sheets containing fluorescent indicator. Components were visualized by UV light. Chromatographic separations were carried out on silica gel 60 M (230-400 mesh). Preparative TLC was performed on fluorescent silica gel glass backed plates (250 μm). All experiments dealing with moisture and air-sensitive compounds were conducted under dry nitrogen. All new products showed a single spot on TLC analysis, after purification.

Scheme S₁

Scheme S₂

Experimental Details:

The syntheses of compounds **1-3** are based on literature procedures and all the compounds showed satisfactory spectral characteristics.

Typical procedure for coupling of **2** with bromoaromatics:

Synthesis of 2-(1-pyrenyl)-O⁶-benzyl-3',5'-bis-O-(*tert*-butyldimethylsilyl)-2'-deoxyguanosine (**4**):

Compound **2** (0.1 g, 0.17 mmol), 1-bromopyrene (0.06 g, 0.21 mmol), Cs₂CO₃ (0.08 g, 0.23 mmol), Pd(OAc)₂ (0.004 g, 0.01 mmol) and BINAP (0.08 g, 0.12 mmol) were weighed out in a vial and flushed with argon. Anhydrous toluene (1.5 mL) was added and the flask was refilled with argon before it was sealed. The mixture was allowed to stir on a sand bath at 80°C for 8-16 hours. It was subsequently filtered through a celite pad, concentrated under reduced pressure, and chromatographed with 8 % ethyl acetate in hexanes.

R_f = 0.35 in 20% ethyl acetate in hexanes.

¹H NMR: 0.068 (s, 6H; Si-Me), 0.070 (s, 6H; Si-Me), 0.90 (s, 9H; Me₃C), 0.91 (s, 9H; Me₃C), 2.34 (m, 1H; H-2'a), 2.56 (m, 1H; H-2'b), 3.77 (m, 1H; H-5'), 3.81 (m, 1H; H-5'), 3.97 (m, 1H; H-4'), 4.38 (m, 1H; H-3'), 5.49 (m, 2H; CH₂Ph), 6.32 (t, *J* = 6.5 Hz, 1H; H-1'), 7.22-7.73 (m, 5H; phenyl), 7.99 (s, 1H; H-8), 8.01-8.25 (m, 9H; pyrene), 8.55 (m, 1H; pyrene).

¹³C NMR: -4.79, -4.66, 17.99, 18.43, 25.76, 25.98, 41.17, 62.90, 65.36, 68.19, 72.00, 76.71, 84.03, 87.79, 116.75, 121.05, 121.31, 124.65, 125.05, 126.06, 126.14, 126.98, 127.31, 127.40, 127.64, 127.88, 127.95, 128.23, 128.26, 128.56, 131.59, 136.38, 138.26, 140.94, 156.71, 160.68.

HRFAB MS: 785.3777 (M⁺) (Calcd. C₄₅H₅₅N₅O₄Si₂: 785.3793)

Synthesis of 2-(8-nitro-1-pyrenyl)-*O*⁶-benzyl-3', 5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxyguanosine (5):

R_f = 0.25 in 20% ethyl acetate in hexanes.

¹H NMR: 0.09 (s, 3H; Si-Me), 0.094 (s, 3H; Si-Me), 0.10 (s, 3H; Si-Me), 0.11 (s, 3H; Si-Me), 0.92 (s, 9H; Me₃C), 0.93 (s, 9H; Me₃C), 2.42 (m, 1H; H-2'a), 2.58 (m, 1H; H-2'b), 3.80 (dq, *J* = 6.2, 12.3 Hz, 2H; H-5'), 4.02(q, *J* = 3.4 Hz, 1H; H-4'), 4.58 (m, 1H; H-3'), 5.60 (dd, *J* = 12.3, 14.0 Hz, 2H; CH₂Ph), 6.44 (t, *J* = 6.5 Hz, 1H; H-1'), 7.29 (m, 3H; phenyl), 7.41 (m, 2H; phenyl) 7.79 (s, 1H; H-8), 8.03 (d, *J* = 8.9 Hz, 1H), 8.06 (s, 1H), 8.14 (d, *J* = 8.6 Hz, 1H), 8.22 (d, *J* = 8.6 Hz, 1H), 8.28 (d, *J* = 8.2 Hz, 1H), 8.52 (d, *J* = 9.6 Hz, 1H), 8.69 (d, *J* = 8.6 Hz, 1H), 8.83 (d, *J* = 8.6 Hz, 1H), 8.98 (d, *J* = 9.6 Hz, 1H).

¹³C NMR: -4.7, -4.65, 18.0, 18.4, 25.8, 25.99, 41.4, 62.9, 68.4, 72.0, 76.8, 83.9, 87.8, 117.2, 120.9, 121.0, 121.6, 123.1, 124.0, 124.8, 125.2, 125.4, 126.9, 128.0, 128.3, 128.4, 130.9, 135.6, 135.9, 136.3, 138.6, 142.0, 153.2, 155.7, 160.7.

HRFAB MS: 831.3748 (M⁺ + H) (Calcd. C₄₅H₅₅N₆O₆Si₂: 831.3722).

Synthesis of 2-(6-nitro-1-pyrenyl)-*O*⁶-benzyl-3', 5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxyguanosine (6):

R_f = 0.2 in 20% ethyl acetate in hexanes.

¹H NMR: 0.081 (s, 3H; Si-Me), 0.083 (s, 3H; Si-Me), 0.089 (s, 3H; Si-Me), 0.098 (s, 3H; Si-Me), 0.90 (s, 9H; Me₃C), 0.91 (s, 9H; Me₃C), 2.40 (m, 1H; H-2'a), 2.57 (m, 1H; H-2'b), 3.79 (m, 2H; H-5'), 4.01(q, *J* = 3.4 Hz, 1H; H-4'), 4.58 (m, 1H; H-3'), 5.61 (m, 2H; CH₂Ph), 6.41 (t, *J* = 6.8 Hz, 1H; H-1'), 7.27(m, 3H; phenyl), 7.37 (m, 2H; phenyl) 7.72 (s, 1H), 8.06 (d, *J* = 9.2 Hz, 1H; H-8), 8.07 (s, 1H), 8.08 (d, *J* = 8.2 Hz, 1H), 8.24 (d, *J* = 8.6 Hz, 1H), 8.25 (d, *J* = 9.2 Hz, 1H), 8.39 (d, *J* = 9.2 Hz, 1H), 8.63 (d, *J* = 8.9 Hz, 1H), 8.74 (d, *J* = 8.6 Hz, 1H), 8.81 (d, *J* = 9.6 Hz, 1H).

¹³C NMR: -5.4, -5.3, -4.8, -4.6, 17.99, 18.4, 25.8, 25.99, 41.4, 62.9, 68.4, 72.0, 76.7, 83.9, 87.8, 120.1, 121.4, 121.99, 123.0, 123.5, 124.1, 125.4, 125.6, 126.3, 126.4, 127.6, 127.99, 128.0, 128.4, 131.5, 135.1, 135.9, 136.3, 138.6, 142.6, 153.2, 155.9, 160.7.

HRFAB MS: 831.3720 (M⁺ + H) (Calcd. C₄₅H₅₅N₆O₆Si₂: 831.3722).

Synthesis of 2-((4-phenyl)phenyl)-*O*⁶-benzyl-3', 5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxyguanosine (7):

R_f = 0.4 in 40% ethyl acetate in hexanes.

¹H NMR: 0.081 (s, 3H; Si-Me), 0.085 (s, 3H; Si-Me), 0.11 (s, 6H; Si-Me), 0.90 (s, 9H; Me₃C), 0.91 (s, 9H; Me₃C), 2.43 (m, 1H; H-2'a), 2.57 (m, 1H; H-2'b), 3.81 (m, 2H; H-5'), 4.04 (q, *J* = 3.4 Hz, 1H; H-4'), 4.60 (m, 1H; H-3'), 4.71 (s, 2H; H-3'), 5.65 (dd, 2H; *J* = 12.3, 14.0 Hz, CH₂Ph), 6.43 (t, *J* = 6.5 Hz, 1H; H-1'), 7.10 (s, 1H), 7.30-7.39 (m, 7H), 7.43 (t, 2H), 7.45 (d, 2H), 7.56-7.62 (dd, 5H), 7.70-7.73 (d, 2H), 8.02 (s, 1H).

¹³C NMR: -4.54, -4.42, 18.22, 18.64, 25.98, 26.19, 41.65, 63.14, 65.57, 68.48, 72.30, 76.91, 84.18, 88.0, 116.72, 119.22, 126.87, 126.96, 127.18, 127.65, 127.84, 128.21, 128.26, 128.63, 128.76, 128.96, 135.06, 136.63, 138.37, 139.49, 141.04, 153.38, 155.64, 160.75.

HRFAB MS: 737.3781 (M⁺) (Calcd. C₄₁H₅₅N₅O₄Si₂: 737.3793).

Synthesis of 2-(9-anthracyl)-*O*⁶-benzyl-3',5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxyguanosine (8):

R_f = 0.35 in 40% ethyl acetate in hexanes.

¹H NMR : 0.075 (s, 3H; Si-Me), 0.085 (s, 3H; Si-Me), 0.10 (s, 3H; Si-Me), 0.11 (s, 3H; Si-Me), 0.90 (s, 9H; Me₃C), 0.91 (s, 9H; Me₃C), 2.43 (m, 1H; H-2'a), 2.57 (m, 1H; H-2'b), 3.85 (m, 2H; H-5'), 4.1 (q, *J* = 3.2 Hz, 1H; H-4'), 4.66 (m, 1H; H-3'), 5.48 (dd, 2H, *J* = 12.4, 14.2 Hz, CH₂Ph), 6.46 (t, *J* = 6.4 Hz, 1H; H-1'), 7.28-7.35 (m, 5H), 7.51 (m, 4H) 7.64 (m, 1H), 7.93 (d, 1H), 8.08 (d, 1H), 8.20 (dd, 1H), 8.24 (s, 1H), 8.33 (m, 1H), 8.50 (s, 1H), 8.63 (d, 1H).

¹³C NMR: -5.45, -5.35, -4.78, -4.65, 18.00, 18.42, 25.78, 25.90, 25.99, 40.82, 62.85, 63.65, 65.30, 67.72, 71.96, 76.72, 83.89, 87.73, 116.29, 124.16, 125.35, 125.79, 126.00, 126.96, 127.57, 127.59, 127.78, 128.30, 128.49, 128.53, 128.55, 128.95, 129.45, 130.77, 131.99, 135.99, 137.82, 153.67, 158.75, 160.64.

HRFAB MS: 761.3761 (M⁺) (Calcd. C₄₃H₅₅N₅O₄Si₂: 761.3793).

Synthesis of 2-(1-naphthyl)-*O*⁶-benzyl-3', 5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxy-guanosine (9):

$R_f = 0.3$ in 20% ethyl acetate in hexanes.

¹H NMR: 0.077 (s, 3H; Si-Me), 0.09 (s, 3H; Si-Me), 0.10 (s, 3H; Si-Me), 0.11 (s, 3H; Si-Me), 0.91 (s, 9H; Me₃C), 0.92 (s, 9H; Me₃C), 2.43 (m, 1H; H-2'a), 2.57 (m, 1H; H-2'b), 3.75 (m, 2H; H-5'), 3.98 (q, $J = 3.4$ Hz, 1H; H-4'), 4.55 (m, 1H; H-3'), 5.49 (dd, 2H, $J = 12.0, 14.0$ Hz, CH₂Ph), 6.37 (t, $J = 6.2$ Hz, 1H; H-1'), 7.25-7.37 (m, 6H), 7.47 (m, 3H), 7.51 (d, 1H), 7.64 (d, 1H), 7.97 (s, 1H), 8.05 (m, 2H).

¹³C NMR: -4.55, -4.42, 18.22, 18.63, 25.99, 26.19, 41.37, 63.13, 65.56, 68.34, 72.28, 76.92, 84.20, 88.00, 116.79, 118.87, 121.58, 124.13, 125.97, 126.01, 126.04, 127.19, 127.55, 128.10, 128.49, 128.53, 128.76, 128.85, 134.49, 135.10, 136.64, 138.41, 156.87, 160.79.

HRFAB MS: 712.3684 (M^+) (Calcd. C₃₉H₅₄N₅O₄Si₂: 712.3715).

Synthesis of 2-(6-chrysenyl)-*O*⁶-benzyl-3',5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxyguanosine (10):

$R_f = 0.4$ in 20% ethyl acetate in hexanes.

¹H NMR: 0.070 (s, 3H; Si-Me), 0.081 (s, 3H; Si-Me), 0.09 (s, 3H; Si-Me), 0.10 (s, 3H; Si-Me), 0.89 (s, 9H; Si-Me), 0.90 (s, 9H; Si-Me), 2.48 (m, 1H; H-2'a), 2.68 (m, 1H; H-2'b), 3.75 (m, 2H; H-5'), 4.01 (q, $J = 3.0$ Hz, 1H; H-4'), 4.55 (m, 1H; H-3'), 4.71 (s, 1H), 5.54 (dd, 2H, $J = 12.2, 14.2$ Hz, CH₂Ph), 6.47 (t, $J = 6.4$ Hz, 1H; H-1'), 7.20-7.22 (m, 3H), 7.34-7.38 (m, 7H), 7.49 (m, 2H), 7.62 (m, 2H), 7.64 (m, 1H), 7.69 (t, 1H), 7.97 (m, 2H), 8.04 (s, 1H), 8.23 (d, 1H), 8.70 (d, 1H), 8.75 (d, 1H), 8.87 (d, 1H), 9.47 (s, 1H).

¹³C NMR: -4.8, -4.7, 17.98, 18.4, 25.7, 25.9, 25.97, 41.1, 62.8, 65.3, 68.3, 71.7, 76.7, 83.9, 87.8, 113.6, 116.7, 121, 121.9, 123.2, 123.9, 126.2, 126.4, 126.5, 126.7, 126.98, 127.6, 127.9, 128.2, 128.4, 128.5, 128.6, 131.4, 132.4, 138.3, 140.9, 153.3, 156.85, 160.75.

HRFAB MS: 811.3986 (M^+) (Calcd. C₄₇H₅₇N₅O₄Si₂: 811.3949).

Typical procedure for coupling of **3** with aromatic amines:

Synthesis of 2-(1-pyrenyl)-*O*⁶-benzyl-3',5'-bis-*O*-(*tert*-butyldimethylsilyl)-2'-deoxy-guanosine (**4**):

Compound **3** (0.16 g, 0.25 mmol), 1-aminopyrene (0.076 g, 0.35 mmol), Cs₂CO₃ (0.114 g, 0.35 mmol), Pd(OAc)₂ (0.006 g, 0.025 mmol) and BINAP (0.03 g, 0.037 mmol) were weighed out in a vial and flushed with argon. Anhydrous toluene (1.5 mL) was added and the flask was refilled with argon before it was sealed. The mixture was allowed to stir on a sand bath at 80°C for 8-16 hours. It was subsequently filtered through a celite pad, concentrated under reduced pressure and chromatographed with 8-10 % ethyl acetate in hexanes. The spectral features are identical as described earlier. Compounds **5**, **7** and **10** were synthesized in the same fashion all of which showed identical spectral features as described earlier for the coupling from **2**.

General procedure for deprotection:

Synthesis of 2-(8-nitro-1-pyrenyl)-*O*⁶-benzyl-2'-deoxyguanosine (**11**):

To a stirred solution of **5** (100 mg, 0.12 mmol) in THF (3 mL) was added tetrabutylammonium fluoride (0.36 mmol, 1.0 M solution in THF). The mixture was stirred at room temperature for 1.5 h, after which the solvent was removed under reduced pressure. The crude product was purified by chromatography on silica, with 4% methanol in chloroform. The product **11** (67 mg, 95% yield) was isolated as pale yellow oil.

R_f = 0.4 (10% methanol in chloroform)

¹H NMR (DMSO-*d*₆): 2.29 (m, 1H; H-2'a), 2.68 (m, 1H; H-2'b), 3.47 (m, 2H; H-5'), 3.83 (m, 1H; H-4'), 4.32 (m, 1H; H-3'), 4.86 (m, 1H, CH₂-OH), 5.26 (m, 1H, CH-OH), 5.48 (s, 2H; CH₂Ph), 6.33 (t, *J* = 6.5 Hz, 1H; H-1'), 7.29 (m, 3H; phenyl), 7.34 (m, 2H; phenyl), 8.16-8.73 (m, 9H; H-8 and pyrene), 10.11 (s, 1H, NH).

¹³C NMR: 23.4, 24.5, 49.47, 55.8, 61.3, 62.4, 62.5, 71.4, 83.8, 88.5, 119.4, 119.5, 122.0, 123.1, 125.1, 125.4, 125.5, 125.6, 125.7, 125.8, 125.9, 127.4, 127.9, 128.0, 131.8, 131.9, 132.9, 137.2, 150.4, 150.6, 169.9.

HRES MS: 625.1819 (M⁺+Na) (Calcd. C₃₃H₂₆N₆O₆Na: 625.1812).

Synthesis of 2-(6-nitro-1-pyrenyl)-O⁶-benzyl-2'-deoxyguanosine (12):

R_f = 0.4 (10% methanol in chloroform)

¹H NMR (DMSO-*d*₆): 2.30 (m, 1H; H-2'a), 2.65 (m, 1H; H-2'b), 3.49 (m, 2H; H-5'), 3.85 (m, 1H; H-4'), 4.34 (m, 1H; H-3'), 4.89 (m, 1H, CH₂-OH), 5.28 (m, 1H, CH-OH), 5.51 (s, 2H; CH₂Ph), 6.33 (t, *J* = 6.5 Hz, 1H; H-1'), 7.28 (m, 3H; phenyl), 7.34 (m, 2H; phenyl), 8.18-8.65 (m, 9H; H-8 and pyrene), 10.08 (s, 1H, NH).

¹³C NMR: 14.5, 26.6, 62.4, 62.53, 68.3, 71.6, 84.0, 88.6, 116.8, 120.0, 123.8, 124.3, 124.6, 125.1, 125.3, 126.6, 126.7, 127.4, 128.5, 128.9, 129.2, 129.3, 132.5, 135.8, 137.2, 138.1, 140.4, 142.6, 154.4, 157.2, 160.7.

HRES MS: 603.1995 (M⁺+H) (Calcd. C₃₃H₂₇N₆O₆: 603.1992).

Synthesis of 2-(8-acetamido-1-pyrenyl)-2'-deoxyguanosine (13):

Hydrogen gas was bubbled through a solution of **11** (60 mg, 0.1 mmol) and 5% Pd/C (2 mg) in 1:1 THF/methanol (5 mL) for 2 hours. The mixture was then stirred at room temperature overnight under mild pressure of hydrogen gas. The catalyst was removed by filtration. The filtrate was evaporated. The crude product was acetylated without purification by adding acetic anhydride (0.12 mL, 1.26 mmol), after dissolving the product in DCM (12 mL) and stirring for 5 h. Following workup the compound was purified by chromatography on silica, with 8% methanol in chloroform which gave 37 mg (71% yield) of the product **13** as a yellow oil.

R_f = 0.4 (20% methanol in chloroform)

¹H NMR (DMSO-*d*₆): 2.48 (m, 1H; H-2'a), 2.50 (s, 3H, CO-CH₃), 2.60 (m, 1H; H-2'b), 3.16 (m, 2H; H-5'), 3.74 (m, 1H; H-4'), 4.23 (m, 1H; H-3'), 4.80 (m, 1H, CH₂-OH), 5.18 (m, 1H, CH-OH), 6.10 (t, *J* = 6.5 Hz, 1H; H-1'), 8.04-8.37 (m, 8H; H-8 and pyrene), 8.63 (d, 1H,), 9.39 (br, 1H, NH-COCH₃), 10.31 (s, 1H, NH), 11.10 (br, 1H, N¹-H).

HRFAB MS: 524.1787 (M⁺) (Calcd. C₂₈H₂₄N₆O₅: 524.1808).

Synthesis of 2-(6-acetamido-1-pyrenyl)-2'-deoxyguanosine (14):

R_f = 0.4 (30% methanol in dichloromethane)

¹H NMR (DMSO-*d*₆): 2.28 (m, 1H; H-2'a), 2.49 (s, 3H, CO-CH₃), 2.53 (m, 1H; H-2'b), 3.33 (m, 2H; H-5'), 3.73 (m, 1H; H-4'), 4.22 (m, 1H; H-3'), 4.90 (m, 1H, CH₂-OH), 5.20 (m, 1H, CH-OH), 6.09 (t, *J* = 6.5 Hz, 1H; H-1'), 8.01-8.37 (m, 8H; H-8 and pyrene), 8.62 (d, 1H,), 9.80 (br, 1H, NH-COCH₃), 10.32 (br, 1H, NH), 11.20 (br, 1H, N¹-H).

¹³C NMR: 24.5, 49.5, 55.8, 62.4, 62.5, 71.6, 83.8, 88.5, 119.4, 119.5, 122.0, 123.1, 125.2, 125.4, 125.5, 125.6, 125.7, 125.9, 127.4, 127.9, 128.0, 131.8, 131.9, 132.9, 137.2, 150.4, 150.6, 169.9.

HRES MS: 547.1702 (M⁺ + Na) (Calcd. C₂₈H₂₄N₆O₅Na: 547.1706).

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F2P -0.500 ppm
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Current Data Parameters

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PROCNO 1

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RG 143.7
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DE 127.50 usec
TE 300.0 K
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***** CHANNEL f1 *****

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F2 - Processing parameters

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WDW EN
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters

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Current Data Parameters
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 PROCNO 1

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 DE 127.50 usec
 TE 300.0 K
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----- CHANNEL f1 -----
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 PL1 -4.00 dB
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F2 - Processing parameters
 SI 16384
 SF 400.1300133 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 FIP 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06625 Hz/cm

Current Data Parameters
NAME 1c051603-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030516
Time 15.09
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TO 16384
SOLVENT DMSO
NS 16
DS 2
SWH 5602.241 Hz
FIDRES 0.341934 Hz
AQ 1.4623220 sec
RG 362
DM 89.250 usec
DE 127.50 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.50 usec
PL1 -3.00 dB
SF01 400.1452008 MHz

F2 - Processing parameters
S1 32768
SF 400.1430049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 4401.57 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.57500 ppm/cm
HZCM 230.08223 Hz/cm

Current Data Parameters

NAME 1c052103-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030521
Time 18.47
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 16384
SOLVENT DMSO
NS 32
DS 2
SWH 5602.241 Hz
FIDRES 0.341934 Hz
AQ 1.4623220 sec
RG 645.1
DM 89.250 usec
DE 127.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 6.50 usec
PL1 -3.00 dB
SFO1 400.1452008 MHz

F2 - Processing parameters

SI 32768
SF 400.1430057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 12.000 ppm
F1 4801.72 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPHMC 0.62500 ppm/cm
HZCM 250.08535 Hz/cm

Current Data Parameters
 NAME 1c053003-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20030531
 Time 17 04
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 16384
 SOLVENT DMSO
 NS 16
 DS 2
 SMH 5602.241 Hz
 FIDRES 0.341934 Hz
 AQ 1.4623220 sec
 RG 362
 DM 89.250 usec
 DE 127.50 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 6.50 usec
 PL1 -3.00 dB
 SF01 400.1452008 MHz

F2 - Processing parameters

SI 32768
 SF 400.1430049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 11.000 ppm
 F1 4401.57 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCN 0.57500 ppm/cm
 HZCN 230.08223 Hz/cm

Current Data Parameters

NAME lc061003-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030611
Time 0.28
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zg30
TD 16384
SOLVENT DMSO
NS 128
DS 2
SWH 5602.241 Hz
FIDRES 0.341934 Hz
AQ 1.4623220 sec
RG 2896.3
DM 89.250 usec
DE 127.50 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.50 usec
PL1 -3.00 dB
SF01 400.1452008 MHz

F2 - Processing parameters

SI 32768
SF 400.1430052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 12.000 ppm
F1 4801.72 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.62500 ppm/cm
HZCM 250.08939 Hz/cm

Aromatic region blow-up of the NOESY Spectra of 5

Cosy correlation of 5

Oc1nc2c(ncn2COCc3ccccc3)c4ccc5c6ccc(cc5c1)cc([N+](=O)[O-])cc6 [illegible]

Aromatic region blow-up of NOESY correlation of **6**