Efficient Access to Cyclic Ureas Via Pd-Catalyzed Cyclization

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Supporting Information

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General considerations: Screening reactions were conducted under a nitrogen atmosphere in 8 mL vials equipped with magnetic stir-bars and septa. Larger scale preparative reactions were conducted in standard RB-flasks under a nitrogen atmosphere. HPLC grade solvents were used with no additional purification/drying. NMR spectra were recorded using a Bruker DPX-400 spectrometer; ^1H NMR recorded at 400 MHz and ^{13}C recorded at 100 MHz. TLC was performed using 2.5 cm $\times$ 7.5 cm EMD glass-backed plates (silica gel 60, F₂₅₄, 250 μm thickness). Flash column chromatography was performed using EMD silica gel 60, 230-400 mesh. HPLC was performed on an Agilent 1100 Series machine using an Agilent Zorbax (Eclipse XDB C8) column (4.6 mm $\times$ 150 mm), 0.65 mL/min flowrate, 205 nm detection, 30 $^\circ\text{C}$ column temperature; mobile phase MeCN/0.1% aq. H₃PO₄ 10/90 for 3 min then ramp to 60/40 over 10 min then ramp to 95/5 over 2 min then 95/5 for a further 4 min.

General procedure A

Reductive alkylation step: To a RB-flask equipped with magnetic stir-bar and temperature probe was charged the arylamine (100 mol%) and the carbonyl compound (110 mol%) followed by *i*-PrOAc (12 mL/g of arylamine) and trifluoroacetic acid (200 mol%). Sodium triacetoxyborohydride (120 mol%) was added as a solid over 1 min, normally accompanied by an increase in temperature to ~ 40 $^\circ\text{C}$. After 10 min agitation, the mixture was usually homogeneous and LC analysis indicated complete consumption of the arylamine. A solution of 10 wt% aqueous NaOH was added to the mixture until pH 8-9. The phases were allowed to separate and the organic phase washed with brine then dried over Na₂SO₄, filtered and concentrated to afford the crude product.¹

Urea formation step: To a RB-flask equipped with a magnetic stir-bar and temperature probe was charged with THF (< 50 ppm H₂O, 8 mL/mL of CSI) then chlorosulfonyl isocyanate (CSI) (120 mol%) was added at room temperature. The mixture was cooled to

¹ Analytical data for purified reductively alkylated products can be found in the preceding paper. For the present work, the crude products were used directly in the subsequent urea formation.

-10 °C. A solution of reductively alkylated amine (100 mol%) in THF : *i*-PrOAc (1 : 1) (< 500 ppm H₂O, 5 mL/g of amine) and added over a 20 min period. An exotherm was observed during this addition (max. temp. 2 °C). After 10 min and with the batch temperature returned to -10 °C, water (0.5 mL/g of substrate) was added dropwise over a 10 min period. A second exotherm was observed during the water addition (max. temp. 17 °C). The mixture was allowed to warm to rt and aged for 30 min. The mixture was treated with 10% aq. NaOH until pH 8-9 and the separated organic phase was washed with brine. The work-up was conducted at 50 °C to maintain solubility of the product. Azeotropic drying with *i*-PrOAc was conducted at atmospheric pressure under constant volume conditions until the water content was < 250 ppm by KF titration (THF was also removed during this process). The urea was isolated by crystallization.

4-[1-(2-Chloro-pyridin-3-yl)ureido]piperidine-1-carboxylic acid ethyl ester (3a):

Following general procedure A, **3a** was obtained as an off-white solid (85%): ¹H NMR (400 MHz, DMSO) δ 0.73-1.41 (br m, 2H), 1.10 (t, *J* = 7.2 Hz, 3H), 1.67-2.00 (br m, 2H), 2.65-2.93 (br m, 2H), 3.84-4.05 (m, 4H), 4.23-4.36 (m, 1H), 5.84 (s, 2H), 7.45 (dd, *J* = 7.6, 4.8 Hz, 1H), 7.76 (dd, *J* = 7.6, 1.6 Hz, 1H), 8.37 (dd, *J* = 4.8, 1.6 Hz, 1H); ¹³C {¹H} NMR (100 MHz, DMSO) δ 14.8, 30.0 and 31.7 (br, rotamers), 43.2, 54.2, 61.0, 124.3, 133.9, 142.2, 149.0, 152.3, 154.7, 156.5. HRMS calcd. for C₁₄H₁₉ClN₄O₃ [M+H]⁺ 327.12184, found 327.12253.

3b

4-[1-(2-Chloro-phenyl)ureido]piperidine-1-carboxylic acid ethyl ester (3b):

Following general procedure A, **3b** was obtained as an off-white solid (82%): ^1H NMR (400 MHz, DMSO) δ 0.74-0.96 (br m, 1H), 1.06 (t, J = 7.2 Hz, 3H), 1.16-1.35 (br m, 1H), 1.62-1.97 (br m, 2H), 2.66-2.91 (br m, 2H), 3.82-4.04 (m, 4H), 4.21-4.36 (m, 1H), 5.50 (br s, 2H), 7.22-7.27 (m, 1H), 7.31-7.37 (m, 2H), 7.49-7.55 (m, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 15.0, 30.0 and 31.9 (br, rotamers), 43.4, 54.1, 61.1, 128.6, 130.1, 130.9, 133.3, 135.1, 136.9, 154.8, 156.9. HRMS calcd. for $\text{C}_{15}\text{H}_{20}\text{ClN}_3\text{O}_3$ $[\text{M}+\text{H}]^+$ 326.12660, found 326.12676.

3c

4-[1-(2-Bromo-phenyl)ureido]piperidine-1-carboxylic acid ethyl ester (3c):

Following general procedure A, **3c** was obtained as an off-white solid (84%): ^1H NMR (400 MHz, DMSO) δ 0.75-0.95 (m, 1H), 1.67 (t, J = 7.2 Hz, 3H), 1.21-1.38 (m, 1H), 1.68-1.80 (m, 1H), 1.88-1.98 (m, 1H), 2.60-2.91 (br m, 2H), 3.84-4.04 (m, 4H), 4.18-4.29 (m, 1H), 5.46 (br s, 2H), 7.23-7.30 (m, 2H), 7.39 (dt, J = 8.4, 2.0 Hz, 1H), 7.69 (dd, J = 8.4, 2.0 Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 15.0, 31.1 (br d), 43.4, 54.3, 61.1, 126.6, 129.3, 130.3, 133.2, 134.1, 138.6, 154.8, 156.7. HRMS calcd. for $\text{C}_{15}\text{H}_{20}\text{BrN}_3\text{O}_3$ $[\text{M}+\text{H}]^+$ 370.07608, found 370.07653.

4-[1-(2-Iodo-phenyl)ureido]piperidine-1-carboxylic acid ethyl ester (3d): Following general procedure A, **3d** was obtained as an off-white solid (81%): ^1H NMR (400 MHz, DMSO) δ 0.74-0.88 (m, 1H), 1.03-1.15 (m, 3H), 1.28-1.42 (m, 1H), 1.72-1.81 (m, 1H), 1.94-2.03 (m, 1H), 2.61-2.90 (br m, 2H), 3.80-4.02 (m, 4H), 4.14-4.26 (m, 1H), 5.39 (br s, 2H), 7.07 (td, $J = 8.0, 1.2$ Hz, 1H), 7.23 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.40 (td, $J = 8.0, 1.2$ Hz, 1H), 7.91 (dd, $J = 8.0, 1.2$ Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 15.0, 30.2 and 32.1 (rotamers), 43.4 and 43.5 (rotamers), 54.4, 61.1, 105.4, 130.0, 130.2, 132.0, 140.4, 142.3, 154.9, 156.5. HRMS calcd. for $\text{C}_{15}\text{H}_{20}\text{IN}_3\text{O}_3$ $[\text{M}+\text{H}]^+$ 418.06221, found 418.06301.

1-(2-Chloro-pyridin-3-yl)-1-(4-fluoro-benzyl)urea (3e): Following general procedure A, **3e** was obtained as an off-white solid (88%): ^1H NMR (400 MHz, DMSO) δ 4.71 (br, 2H), 6.05 (s, 2H), 7.08 (t, $J = 8.8$ Hz, 2H), 7.24 (td, $J = 8.0, 6.0$ Hz, 2H), 7.33 (dd, $J = 8.0, 4.4$ Hz, 1H), 7.45 (dd, $J = 8.0, 2.0$ Hz, 1H), 8.29 (dd, $J = 4.4, 2.0$ Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 50.8, 115.4 (d, $J_{\text{CF}} = 20.1$ Hz), 124.3, 130.8 (d, $J_{\text{CF}} = 10.1$ Hz), 134.5 (d, $J_{\text{CF}} = 10.1$ Hz), 136.2, 141.0, 148.7, 150.6, 157.2, 161.8 (d, $J_{\text{CF}} = 241.0$ Hz). HRMS calcd. for $\text{C}_{13}\text{H}_{11}\text{ClFN}_3\text{O}$ $[\text{M}+\text{H}]^+$ 280.06474, found 280.06392.

3f

1-Benzyl-1-(2-chloro-pyridin-3-yl)urea (3f): Following general procedure A, **3f** was obtained as an off-white solid (86%): ^1H NMR (400 MHz, DMSO) δ 3.91-5.64 (br, 2H), 6.02 (s, 2H), 7.10-7.32 (m, 6H), 7.41 (d, J = 7.6, 1.2 Hz, 1H), 8.25 (d, J = 4.4, 1.2 Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 51.6, 124.3, 127.7, 128.7 (2C), 136.4, 138.4, 140.9, 148.7, 150.6, 157.3. HRMS calcd. for $\text{C}_{13}\text{H}_{12}\text{ClN}_3\text{O}$ $[\text{M}+\text{H}]^+$ 262.07417, found 262.07341.

3g

1-(2-Chloro-pyridin-3-yl)-1-cyclohexylurea (3g): Following general procedure A, **3g** was obtained as an off-white solid (86%): ^1H NMR (400 MHz, DMSO) δ 0.75-0.98 (m, 2H), 1.03-1.21 (m, 1H), 1.25-1.43 (m, 2H), 1.51-1.61 (m, 1H), 1.63-1.78 (m, 2H), 1.91-2.07 (m, 2H), 4.32 (tt, J = 12.4, 3.6 Hz, 1H), 4.53 (s, 2H), 7.33 (dd, J = 8.0, 4.4 Hz, 1H), 7.61 (dd, J = 8.0, 2.0 Hz, 1H), 8.40 (dd, J = 4.4, 2.0 Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 25.3, 25.8, 30.5 and 33.0 (br, rotamers), 56.6, 123.1, 133.8, 141.0, 149.1, 153.0, 156.4. HRMS calcd. for $\text{C}_{12}\text{H}_{16}\text{ClN}_3\text{O}$ $[\text{M}+\text{H}]^+$ 254.10547, found 254.10667.

3h

1-(2-Chloro-pyridin-3-yl)-1-cyclopentylurea (3h): Following general procedure A, **3h** was obtained as an off-white solid (83%): ^1H NMR (400 MHz, DMSO) δ 0.81-1.32 (br m, 2H), 1.32-1.50 (m, 4H), 1.67-1.95 (br m, 2H), 4.34-4.46 (m, 1H), 5.77 (s, 2H), 7.42 (dd, $J = 7.6, 4.4$ Hz, 1H), 7.72 (dd, $J = 7.6, 2.0$ Hz, 1H), 8.33 (dd, $J = 4.4, 2.0$, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 22.6, 30.0 (br), 58.3, 124.4, 134.7, 142.3, 148.9, 152.5, 157.2. HRMS calcd. for $\text{C}_{11}\text{H}_{14}\text{ClN}_3\text{O}$ $[\text{M}+\text{H}]^+$ 240.08982, found 240.08965.

3i

1-(2-Chloro-pyridin-3-yl)-1-cyclohexylmethylurea (3i): Following general procedure A, **3i** was obtained as an off-white solid (85%): ^1H NMR (400 MHz, DMSO) δ 0.74-0.93 (m, 2H), 0.99-1.17 (m, 3H), 1.20-1.35 (m, 1H), 1.45-1.74 (m, 5H), 3.29 (s, 2H), 5.82 (s, 2H), 7.43 (dd, $J = 8.0, 4.2$ Hz, 1H), 7.77 (dd, $J = 8.0, 2.0$ Hz, 1H), 8.30 (dd, $J = 4.8, 2.0$ Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 25.9, 26.5, 30.8, 37.1, 54.5, 124.6, 137.5, 141.1, 148.4, 150.5, 157.4. HRMS calcd. for $\text{C}_{13}\text{H}_{18}\text{ClN}_3\text{O}$ $[\text{M}+\text{H}]^+$ 268.12112, found 268.1217.

1-(2-Chloro-pyridin-3-yl)-1-(4-methoxy-benzyl)urea (3j): Following general procedure A, **3j** was obtained as an off-white solid (%): ^1H NMR (400 MHz, DMSO) δ 3.66 (s, 3H), 3.80-5.35 (br, 2H), 5.98 (br s, 2H), 6.77 (d, $J = 8.4$ Hz, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 7.28 (dd, $J = 8.0, 4.8$ Hz, 1H), 7.35 (dd, $J = 8.0, 2.0$ Hz, 1H), 8.24 (dd, $J = 4.8, 2.0$ Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 50.9, 55.5, 114.1, 124.3, 130.2 (2C), 136.3, 141.1, 148.6, 150.7, 157.3, 158.9. HRMS calcd. for $\text{C}_{14}\text{H}_{14}\text{ClN}_3\text{O}_2$ $[\text{M}+\text{H}]^+$ 292.08473, found 292.08587.

1-(2-Chloro-phenyl)-1-(4-methoxy-benzyl)urea (3k): Following general procedure A, **3k** was obtained as an off-white solid (85%): ^1H NMR (400 MHz, DMSO) δ 3.69 (s, 3H), 3.89-4.59 (br, 1H), 4.65-5.35 (br, 1H), 5.69 (s, 2H), 6.80 (d, $J = 8.4$ Hz, 2H), 6.96 (dd, $J = 8.0, 2.0$ Hz, 1H), 7.09 (d, $J = 8.4$ Hz, 2H), 7.19-7.31 (m, 2H), 7.51 (dd, $J = 8.0, 1.2$ Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 51.0, 55.3, 113.9, 128.3, 129.5, 129.9, 130.6, 130.7, 132.0, 133.2, 139.3, 157.4, 158.7. HRMS calcd. for $\text{C}_{15}\text{H}_{15}\text{ClN}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 291.0900, found 291.0904.

3l

1-Benzyl-1-(2-chloro-4-methyl-phenyl)urea (3l): Following general procedure A, **3l** was obtained as an off-white solid (80%): ^1H NMR (400 MHz, DMSO) δ 2.26 (s, 3H), 3.88-4.38 (br, 1H), 4.96-5.38 (br, 1H), 5.69 (br s, 2H), 6.86 (d, J = 8.0 Hz, 1H), 7.01 (d, J = 8.0 Hz, 1H), 7.12-7.29 (m, 5H), 7.34 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 20.7, 51.6, 127.3, 128.5, 128.6, 129.0, 131.0, 131.5, 132.7, 136.6, 138.8, 139.4, 157.6. HRMS calcd. for $\text{C}_{15}\text{H}_{15}\text{ClN}_2\text{O}$ $[\text{M}+\text{H}]^+$ 275.09457, found 275.09479.

3m

1-Benzyl-1-(2-chloro-4-fluoro-phenyl)urea (3m): Following general procedure A, **3m** was obtained as an off-white solid (81%): ^1H NMR (400 MHz, DMSO) δ 3.97-4.35 (br, 1H), 5.01-5.31 (br, 1H), 5.83 (br s, 2H), 7.00 (dd, J = 8.4, 6.0 Hz, 1H), 7.10 (dd, J = 8.4, 2.8 Hz, 1H), 7.17-7.29 (m, 5H), 7.50 (dd, J = 8.4, 2.8 Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 51.6, 115.4 (d, J_{CF} = 20.1 Hz), 118.0 (d, J_{CF} = 30.2 Hz), 127.4, 128.5, 128.6, 133.3 (d, J_{CF} = 10.1 Hz), 134.5 (d, J_{CF} = 10.1 Hz), 135.9, 138.5, 157.5, 161.2 (d, J_{CF} = 251.5 Hz). HRMS calcd. for $\text{C}_{14}\text{H}_{12}\text{ClFN}_2\text{O}$ $[\text{M}+\text{H}]^+$ 279.06950, found 279.07002.

General procedure B

Pd-Catalyzed urea cyclization with dppb: To a Morton-type flask equipped with stir-bar, reflux condenser and temperature probe was charged NaHCO₃ (300 mol%), urea (100 mol%), and *i*-PrOH (750-1500 ppm H₂O, 10 mL/g substrate). The heterogeneous mixture was agitated and purged with N₂ using an M-fritted gas dispersion tube. After 30 min, Pd(OAc)₂ (1 mol%) and dppb (2 mol%) were added and the N₂ purge continued for a further 30 min. The resulting pink mixture was heated to 83 °C (reflux) for 24 h. After this time, LC analysis of the yellow mixture indicated > 99% conversion. Atmospheric pressure distillation was initiated and continued until ~ 60% of *i*-PrOH had been removed. *i*-PrOAc was added to achieve an ~ 2 : 1 ratio of *i*-PrOAc : *i*-PrOH (the *i*-PrOH aids solubility during workup). Water (3 mL/g substrate) was added and the temperature was maintained at 60 °C throughout workup to ensure solubility. After 15 min agitation, the separated organic phase was washed with brine. Azeotropic drying with *i*-PrOAc was conducted at atmospheric pressure under constant volume conditions until the water content was < 150 ppm measured by KF titration (*i*-PrOH was also removed during this process). The product was isolated by crystallization.

General procedure C

Pd-Catalyzed urea cyclization with X-Phos: To a Morton-type flask equipped with stir-bar, reflux condenser and temperature probe was charged NaHCO₃ (300 mol%), urea (100 mol%), and *i*-PrOH (750-1500 ppm H₂O, 10 mL/g substrate). The heterogeneous mixture was agitated and purged with N₂ using an M-fritted gas dispersion tube. After 30 min, Pd(OAc)₂ (1 mol%) and X-Phos (3 mol%) were added and the N₂ purge continued for a further 30 min. The resulting mixture was heated to 83 °C (reflux) for 24 h. After this time, LC analysis of the yellow mixture indicated > 99% conversion. Atmospheric pressure distillation was initiated and continued until ~ 60% of *i*-PrOH had been removed. *i*-PrOAc was added to achieve an ~ 2 : 1 ratio of *i*-PrOAc : *i*-PrOH (the *i*-PrOH aids solubility during workup). Water (3 mL/g substrate) was added and the temperature was maintained at 60 °C throughout workup to ensure solubility. After 15 min agitation, the separated organic phase was washed with brine. Azeotropic drying with *i*-PrOAc was conducted at atmospheric pressure under constant volume conditions until the water

content was < 150 ppm measured by KF titration (*i*-PrOH was also removed during this process). The product was isolated by crystallization.

4-(2-Oxo-2,3-dihydroimidazo[4,5-*b*]pyridin-1-yl)piperidine-1-carboxylic acid ethyl ester (4a): Following general procedure B, **4a** was obtained as an off-white solid (96%): ¹H NMR (400 MHz, DMSO) δ 1.18 (t, *J* = 7.2 Hz, 3H), 1.65-1.76 (m, 2H), 2.06-2.22 (m, 2H), 2.78-3.03 (br m, 2H), 4.04 (q, *J* = 7.2 Hz, 2H), 4.08-4.20 (m, 2H), 4.30-4.41 (m, 1H), 6.95 (dd, *J* = 7.6, 5.2 Hz, 1H), 7.54 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.87 (dd, *J* = 5.2, 1.2 Hz, 1H), 11.47 (br, 1H); ¹³C {¹H} NMR (100 MHz, DMSO) δ 14.9, 29.0, 43.5, 50.2, 61.1, 115.0, 116.7, 123.6, 140.0, 143.8, 153.5, 154.9. HRMS calcd. for C₁₄H₁₈N₄O₃ [M+H]⁺ 291.14517, found 291.14598.

4-(2-Oxo-2,3-dihydrobenzoimidazol-1-yl)piperidine-1-carboxylic acid ethyl ester (4b)²: Following general procedure C, **4b** was obtained as an off-white solid (89%): ¹H NMR (400 MHz, DMSO) δ 1.20 (t, *J* = 7.2 Hz, 3H), 1.62-1.73 (m, 2H), 2.12-2.28 (m,

² Gustin, D. J.; Sehon, C. A.; Wei, J.; Cai, H.; Meduna, S. P.; Khatuya, H.; Sun, S.; Gu, Y.; Jiang, W.; Thurmond, R. L.; Karlsson, L.; Edwards, J. P. *Biorog. Med. Chem. Lett.* **2005**, *15*, 1687-1692.

2H), 2.78-3.03 (br m, 2H), 4.07 (q, $J = 7.2$ Hz, 2H), 4.09-4.18 (m, 2H), 4.27-4.40 (m, 1H), 6.93-6.99 (m, 3H), 7.18-7.25 (m, 1H), 10.82 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 14.9, 28.9, 43.4, 50.2, 61.1, 108.8, 109.2, 120.7, 120.9, 128.7, 129.6, 154.0, 154.9. HRMS calcd. for $\text{C}_{15}\text{H}_{19}\text{N}_3\text{O}_3$ $[\text{M}+\text{H}]^+$ 290.14992, found 290.14957.

1-(4-Fluoro-benzyl)-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one (4c): Following general procedure B, **4c** was obtained as an off-white solid (95%): ^1H NMR (400 MHz, DMSO) δ 5.00 (s, 2H), 6.96 (dd, $J = 8.0, 5.2$ Hz, 1H), 7.11-7.19 (m, 2H), 7.34-7.41 (m, 3H), 7.89 (dd, $J = 5.2, 1.6$ Hz, 1H), 11.64 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 42.8, 114.4, 115.8 (d, $J_{\text{CF}} = 30.2$ Hz), 117.0, 124.3, 130.0 (d, $J_{\text{CF}} = 10.1$ Hz), 133.3, 140.5, 143.9, 154.1, 161.9 (d, $J_{\text{CF}} = 241.5$ Hz). HRMS calcd. for $\text{C}_{13}\text{H}_{10}\text{FN}_3\text{O}$ $[\text{M}+\text{H}]^+$ 244.08807, found 244.08968.

1-Benzyl-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one (4d)³: Following general procedure B, **4d** was obtained as an off-white solid (95%): ^1H NMR (400 MHz, DMSO) δ 5.01 (s, 2H), 6.92 (dd, $J = 8.0, 5.6$ Hz, 1H), 7.18-7.26 (m, 1H), 7.27-7.34 (m, 5H), 7.89 (d, $J = 5.6$

³ Meanwell, N.; Sit, S. Y.; Gao, J.; Wong, H. S.; Gao, Q.; St. Laurent, D. R.; Balasubramanian, N. *J. Org. Chem.* **1995**, *60*, 1565-1582.

Hz, 1H), 11.68 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 43.5, 114.4, 117.0, 124.4, 127.8, 127.9, 129.0, 137.1, 140.5, 143.9, 154.1. HRMS calcd. for $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 226.09749, found 226.09812.

1-Cyclohexyl-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one (4e): Following general procedure B, **4e** was obtained as an off-white solid (93%): ^1H NMR (400 MHz, DMSO) δ 1.15-1.43 (m, 3H), 1.56-1.73 (m, 3H), 1.74-1.85 (m, 2H), 1.91-2.08 (m, 2H), 4.06-4.19 (m, 1H), 6.94 (dd, $J = 8.0, 5.2$ Hz, 1H), 7.56 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.86 (dd, $J = 5.2, 1.2$ Hz, 1H), 11.49 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 25.0, 25.8, 29.9, 51.8, 115.2, 116.6, 123.5, 139.8, 143.8, 153.4. HRMS calcd. for $\text{C}_{12}\text{H}_{15}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 218.12879, found 218.13001.

1-Cyclopentyl-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one (4f): Following general procedure B, **4f** was obtained as an off-white solid (94%): ^1H NMR (400 MHz, DMSO) δ 1.54-1.67 (m, 2H), 1.77-2.02 (6H), 4.62-4.75 (m, 1H), 6.96 (dd, $J = 8.0, 5.2$ Hz, 1H), 7.42 (d, $J = 8.0$ Hz, 1H), 7.88 (d, $J = 5.2$ Hz, 1H), 11.49 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 24.7, 28.7, 52.6, 114.8, 116.7, 123.3, 139.9, 143.9, 153.6. HRMS calcd. for $\text{C}_{11}\text{H}_{13}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 204.11314, found 204.11401.

1-Cyclohexylmethyl-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one (4g)³: Following general procedure B, **4g** was obtained as an off-white solid (95%): ¹H NMR (400 MHz, DMSO) δ 0.84-0.99 (m, 2H), 1.02-1.17 (m, 3H), 1.45-1.64 (m, 5H), 1.66-1.79 (m, 1H), 3.57 (d, *J* = 7.6 Hz, 2H), 6.95 (dd, *J* = 8.0, 4.8 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 4.8 Hz, 1H), 11.45 (s, 1H); ¹³C {¹H} NMR (100 MHz, DMSO) δ 25.6, 26.3, 30.6, 37.1, 46.3, 114.4, 117.0, 125.3, 140.2, 143.9, 154.3. HRMS calcd. for C₁₃H₁₇N₃O [M+H]⁺ 232.14444, found 232.14495.

1-(4-Methoxy-benzyl)-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one (4h): Following general procedure B, **4h** was obtained as an off-white solid (95%): ¹H NMR (400 MHz, DMSO) δ 3.70 (s, 3H), 4.92 (s, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.94 (dd, *J* = 8.0, 5.2 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.34 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.88 (dd, *J* = 5.2, 1.2 Hz, 1H), 11.60 (s, 1H); ¹³C {¹H} NMR (100 MHz, DMSO) δ 43.0, 55.4, 114.4, 114.1, 116.9, 124.3, 129.0, 129.3, 140.4, 143.9, 154.1, 159.0. HRMS calcd. for C₁₄H₁₃N₃O₂ [M+H]⁺ 256.1086, found 256.1087.

1-(4-Methoxy-benzyl)-1,3-dihydrobenzoimidazol-2-one (4i): Following general procedure C, **4i** was obtained as an off-white solid (92%): ^1H NMR (400 MHz, DMSO) δ 3.68 (s, 3H), 4.91 (s, 2H), 6.86 (d, J = 8.0 Hz, 2H), 6.91-7.04 (m, 4H), 7.26 (d, J = 8.0 Hz, 2H), 10.91 (br, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 43.0, 55.4, 108.4, 109.2, 114.3, 120.8, 121.3, 128.7, 129.2, 129.5, 130.3, 154.7, 158.9. HRMS calcd. for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 255.1134, found 255.1130.

1-Benzyl-5-methyl-1,3-dihydrobenzoimidazol-2-one (4j)⁴: Following general procedure C, **4j** was obtained as an off-white solid (90%): ^1H NMR (400 MHz, DMSO) δ 2.23 (s, 3H), 4.93 (s, 2H), 6.70 (d, J = 8.0 Hz, 1H), 6.78 (s, 1H), 6.83 (d, J = 8.0 Hz, 1H), 7.15-7.22 (m, J = 8.0 Hz, 1H), 7.23-7.31 (m, 4H), 10.85 (br, 1H); ^{13}C $\{^1\text{H}\}$ NMR (100 MHz, DMSO) δ 21.4, 43.7, 108.3, 109.9, 121.5, 127.7, 127.8, 128.4, 128.9, 129.0, 130.6, 137.8, 155.0. HRMS calcd. for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ 239.11789, found 239.11756.

⁴ Kametani, T.; Shio, M. *J. Heterocyclic Chem.* **1970**, 7, 831-834.

1-Benzyl-5-fluoro-1,3-dihydrobenzoimidazol-2-one (4k)⁵: Following general procedure C, **4k** was obtained as an off-white solid (91%): ¹H NMR (400 MHz, DMSO) δ 4.96 (s, 2H), 6.73 (dd, J = 8.8, 2.4 Hz, 1H), 6.83 (dd, J = 8.8, 2.4 Hz, 1H), 6.94 (dd, J = 8.8, 4.8 Hz, 1H), 7.16-7.24 (m, 1H), 7.25-7.30 (m, 4H), 11.10 (br, 1H); ¹³C {¹H} NMR (100 MHz, DMSO) δ 43.8, 97.5 (d, J_{CF} = 28.2 Hz), 107.2 (d, J_{CF} = 24.1 Hz), 108.9 (d, J_{CF} = 9.0 Hz), 126.9, 127.8, 127.9, 129.1, 129.5 (d, J_{CF} = 13.1 Hz), 137.5, 155.2, 158.3 (d, J_{CF} = 234.4 Hz). HRMS calcd. for C₁₄H₁₁FN₂O [M+H]⁺ 243.09282, found 243.09200.

⁵ Orjales, A.; Mosquera, R.; Labeaga, L.; Rhodes, R. *J. Med. Chem.* **1997**, *40*, 586-593.

Current Data Parameters
NAME 224591-077-2
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060306
Time 10.38
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 57
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.42553 ppm/cm
HZOM 170.26810 Hz/cm

Current Data Parameters
NAME 224591-077-2
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060306
Time 10.45
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 360
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW DM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
HZOM 866.27899 Hz/cm

Current Data Parameters
NAME 224591-062-3
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051228
Time 13.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 128
DM 76.000 usec
DE 6.00 usec
TE 300.8 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.870207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3598.83 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHOM 0.38298 ppm/cm
HZOM 153.14171 Hz/cm

Current Data Parameters
NAME 224591-062-3
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051228
Time 13.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 363
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 299.8 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 0.00 Hz
PPHOM 8.51024 ppm/cm
HZOM -866.73247 Hz/cm

Current Data Parameters
NAME 224591-062-1
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051228
Time 11.08
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 256
DM 76.000 usec
DE 6.00 usec
TE 300.5 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3998.70 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.42553 ppm/cm
HZOM 170.15747 Hz/cm

Current Data Parameters
NAME 224591-062-1
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051228
Time 11.15
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 334
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW DM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
HZOM 866.72247 Hz/cm

Current Data Parameters
NAME 224591-062-2
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051228
Time 11.19
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 256
DM 76.000 usec
DE 6.00 usec
TE 300.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3998.70 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.42553 ppm/cm
HZOM 170.15747 Hz/cm

Current Data Parameters
NAME 224591-062-2
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051228
Time 11.36
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 4
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
HZOM 866.73247 Hz/cm

Current Data Parameters
NAME 224591-015-1
EXPNO 1
PROCNO 1

nmr400a h-1

F2 - Acquisition Parameters
Date_ 20051110
Time 8.45
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 101.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.252014 MHz

F2 - Processing parameters
SI 16384
SF 400.250593 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1 10.000 ppm
F1 4002.50 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.31918 Hz/cm

Current Data Parameters
NAME 224591-015-1
EXPNO 2
PROCNO 1

nmr400a h-1 decoupled c-13

F2 - Acquisition Parameters
Date_ 20051110
Time 8.47
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 168
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.653973 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.252012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1 200.000 ppm
F1 20128.59 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 850.15385 ppm/cm
HZCM 850.15385 Hz/cm

Current Data Parameters
NAME 224591-014-2
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051114
Time 8.35
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 80.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3998.70 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.42953 ppm/cm
HZCM 170.15747 Hz/cm

Current Data Parameters
NAME 224591-014-2
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051114
Time 8.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 151
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 8.51664 ppm/cm
HZCM 855.72247 Hz/cm

Current Data Parameters
NAME 224591-013-1
EXPNO 1
PROCNO 1

nmr400a h-1

F2 - Acquisition Parameters
Date_ 20051110
Time 8.27
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 80.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.252014 MHz

F2 - Processing parameters
SI 16384
SF 400.250593 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1 10.000 ppm
F1 4002.50 Hz
F2 -0.000 ppm
F2 -0.00 Hz
PRWCH 0.42653 ppm/cm
HZCM 170.31918 Hz/cm

Current Data Parameters
NAME 224591-013-1
EXPNO 2
PROCNO 1

nmr400a h-1 decoupled c-13

F2 - Acquisition Parameters
Date_ 20051110
Time 8.32
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 183
DS 0
SWH 26315.789 Hz
FIDRES 0.401947 Hz
AQ 1.2462340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6259737 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.252012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1 200.000 ppm
F1 20128.59 Hz
F2 -0.000 ppm
F2 -0.00 Hz
PRWCH 0.42653 ppm/cm
HZCM 856.53583 Hz/cm

156.4
153.0
149.1
141.0
133.8
123.1
77.4
77.1
76.7
56.6
33.0
30.5
25.8
25.3

Current Data Parameters
NAME 224591-022-1
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051114
Time 10.11
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 71.8
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3998.70 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPM0H 0.42953 ppm/cm
HZCM 170.15747 Hz/cm

Current Data Parameters
NAME 224591-022-1
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051114
Time 10.15
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 236
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPM0H 0.42953 ppm/cm
HZCM 170.15747 Hz/cm

Current Data Parameters
NAME 224591-021-1
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051114
Time 8.27
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 101.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SFO1 399.8724592 Mhz

F2 - Processing parameters
SI 16384
SF 399.8700207 Mhz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1 10.000 ppm
F1 3998.70 Hz
F2 -0.000 ppm
F2 -0.00 Hz
PPMCM 0.42953 ppm/cm
HZCM 170.15747 Hz/cm

3i

Current Data Parameters
NAME 224591-021-1
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051114
Time 8.33
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 210
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SFO1 100.5584112 Mhz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SFO2 399.8719994 Mhz

F2 - Processing parameters
SI 32768
SF 100.5473900 Mhz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1 200.000 ppm
F1 20109.48 Hz
F2 -0.000 ppm
F2 -0.00 Hz
PPMCM 0.5012244 ppm/cm
HZCM 855.12244 Hz/cm

3i

Current Data Parameters
NAME 224591-016-2
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051114
Time 8.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 80.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3998.70 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPM0M 0.42953 ppm/cm
HZCM 170.15747 Hz/cm

Current Data Parameters
NAME 224591-016-2
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051114
Time 8.47
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 161
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPM0M 0.41095 ppm/cm
HZCM 855.75247 Hz/cm

Current Data Parameters
NAME 224591-041-1
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20051212
Time 15.12
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 256
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHOM 0.38298 ppm/cm
H2OM 153.24127 Hz/cm

3k

Current Data Parameters
NAME 224591-041-1
EXPNO 3
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060207
Time 10.07
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 151
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
H2OM 86.27899 Hz/cm

3k

Current Data Parameters
NAME 224591-059-4-1
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20051221
Time 11.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 40.3
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHOM 0.38298 ppm/cm
H2OM 153.24127 Hz/cm

Current Data Parameters
NAME 224591-059-4-1
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20051221
Time 11.53
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 217
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 0.00 Hz
PPHOM 8.51064 ppm/cm
H2OM 866.27899 Hz/cm

Current Data Parameters
NAME 224591-059-1-1
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20051221
Time 10.19
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 114
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.1000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.38298 ppm/cm
HZCM 153.24127 Hz/cm

Current Data Parameters
NAME 224591-059-1-1
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20051221
Time 10.23
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 257
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.1000000 sec
d11 0.0300000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.000 MHz
PPMCM 8.51064 ppm/cm
HZCM 866.27899 Hz/cm

162.4
159.9
157.5
138.5
135.9
134.5
134.4
133.3
133.2
128.6
128.5
127.4
118.1
117.8
115.5
115.3

51.6
40.5
40.3
40.1
39.9
39.7
39.5
39.3

Current Data Parameters
NAME 224591-057-2
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060306
Time 10.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 35.9
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.42553 ppm/cm
H2OM 170.26810 Hz/cm

Current Data Parameters
NAME 224591-057-2
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060306
Time 10.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 544
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW DM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
H2OM 86.27899 Hz/cm

Current Data Parameters
NAME 224591-071-1
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060207
Time 10.13
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 574.7
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.55319 ppm/cm
HZCM 221.34851 Hz/cm

Current Data Parameters
NAME 224591-071-1
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060207
Time 10.25
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 321
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 14596.5
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.1322007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 8.51064 ppm/cm
HZCM 846.27899 Hz/cm

Current Data Parameters
NAME 224591-017-3-3
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060214
Time 10.53
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 256
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.55319 ppm/cm
H2OM 221.34851 Hz/cm

Current Data Parameters
NAME 224591-017-3-2
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060206
Time 15.48
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 210
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.1322007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
H2OM 806.27889 Hz/cm

163.1
160.7
154.1
143.9
140.5
133.3
130.0
129.9
124.3
117.0
115.9
115.7
114.4

42.8
40.3
40.0
39.8
39.6
39.4

Current Data Parameters
NAME 224591-143-2-1
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060302
Time 9.48
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 57
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.55319 ppm/cm
HZCM 221.34851 Hz/cm

Current Data Parameters
NAME 224591-143-2-1
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060302
Time 9.52
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 251
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 200.000 ppm
F1P 20122.55 Hz
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
HZCM 846.27899 Hz/cm

Current Data Parameters
NAME 224591-017-1-2
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060207
Time 9.22
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 40.3
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.55319 ppm/cm
HZOM 221.34851 Hz/cm

Current Data Parameters
NAME 224591-017-1-2
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060207
Time 9.24
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 183
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

153.4
143.8
139.8
123.5
116.6
115.2

51.8
40.5
40.3
40.1
39.9
39.7
39.5
39.2
29.9
26.8
25.0

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
HZOM 866.27899 Hz/cm

Current Data Parameters
NAME 224591-017-6-2
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060207
Time 9.31
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 90.5
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.95319 ppm/cm
HZCM 221.34851 Hz/cm

Current Data Parameters
NAME 224591-017-6-2
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060207
Time 9.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 223
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

153.6
143.9
139.9
123.3
116.7
114.8

52.6
40.5
40.3
40.1
39.9
39.7
39.5
39.3
28.7
24.7

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.1322007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.95 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51004 ppm/cm
HZCM 866.27899 Hz/cm

Current Data Parameters
NAME 224591-017-5-1
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051116
Time 10.08
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6576.947 Hz
FIDRES 0.230774 Hz
AQ 2.4904180 sec
RG 90.5
DM 76.000 usec
DE 6.00 usec
TE 299.9 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.870207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 24.00 cm
F1P 13.000 ppm
F1 5198.31 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.55319 ppm/cm
H2OM 221.20470 Hz/cm

Current Data Parameters
NAME 224591-017-5-1
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051116
Time 10.13
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 185
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.1 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.46 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
H2OM 86.572247 Hz/cm

154.3
143.9
140.2
125.3
117.0
114.4

46.3
40.7
40.5
40.2
40.0
39.8
39.6
39.4
37.1
30.6
26.3
25.6

Current Data Parameters
NAME 224591-017-4-3
EXPNO 1
PROCNO 1

nmr400c h1

F2 - Acquisition Parameters
Date_ 20060206
Time 15.03
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 256
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 0.55319 ppm/cm
HZOM 221.34851 Hz/cm

Current Data Parameters
NAME 224591-017-4
EXPNO 2
PROCNO 1

c-13 nmr400c

F2 - Acquisition Parameters
Date_ 20060207
Time 8.48
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 155
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPHOM 8.51064 ppm/cm
HZOM 856.27899 Hz/cm

159.0
154.1
143.9
140.4
129.3
129.0
124.3
116.9
114.4
114.4

55.4
43.0
40.5
40.3
40.1
39.7
39.5
39.2
21.9

Current Data Parameters
NAME 224591-046-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051215
Time 14.53
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 64
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SF01 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHOM 0.38298 ppm/cm
H2DM 153.24127 Hz/cm

nmr400c h1

Current Data Parameters
NAME 224591-046-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051215
Time 14.57
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 171
DS 0
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485108 sec
RG 8192
DM 19.050 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SF01 100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.1322007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 0.00 Hz
PPHOM 8.51064 ppm/cm
H2DM 846.27889 Hz/cm

c-13 nmr400c

Current Data Parameters
NAME 224591-061-4
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051228
Time 13.58
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.230774 Hz
AQ 2.4904180 sec
RG 181
DM 76.000 usec
DE 6.00 usec
TE 300.3 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 13.000 ppm
F1 5198.31 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.55319 ppm/cm
HZCM 221.20470 Hz/cm

Current Data Parameters
NAME 224591-061-4
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051228
Time 14.04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 116
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 299.9 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 8.51064 ppm/cm
HZCM 865.72247 Hz/cm

155.0
137.8
130.6
129.0
128.9
128.4
127.8
127.8
121.5
109.9
108.3
43.7
40.7
40.4
40.2
40.0
39.8
39.6
39.4
21.5

Current Data Parameters
NAME 224591-061-1
EXPNO 1
PROCNO 1

nmr400b h-1

F2 - Acquisition Parameters
Date_ 20051228
Time 13.46
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6576.947 Hz
FIDRES 0.230774 Hz
AQ 2.4904180 sec
RG 161.3
DM 76.000 usec
DE 6.00 usec
TE 299.8 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SF01 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3598.83 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHOM 0.38298 ppm/cm
H2OM 153.14171 Hz/cm

Current Data Parameters
NAME 224591-061-1
EXPNO 2
PROCNO 1

nmr400b c-13 (h-1 decoupled)

F2 - Acquisition Parameters
Date_ 20051228
Time 13.55
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 336
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 299.8 K
D1 0.10000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.60 usec
PL1 5.00 dB
SF01 100.5584112 MHz

----- CHANNEL f2 -----
DPPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 22.00 dB
SF02 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 0.00 Hz
PPHOM 8.51064 ppm/cm
H2OM 85.73247 Hz/cm