New and Practical Method for Synthesis of 1- and 1,3-Substituted Xanthines

Ilia A. Zavialov,* Vilas H. Dahanukar, Hoa Nguyen, Cecilia Orr and David R. Andrews

Department of Synthetic Chemistry, Schering-Plough Research Institute, 1011 Morris

Avenue, Union, NJ 07083

ilia.zavialov@spcorp.com

Supporting Information

	Page
I General Procedures	S2
II Experimental Procedures	S2
III Spectroscopic Data	S3
IV References	S9
V ¹ H and ¹³ C NMR Spectra	S9

General Procedures

Unless otherwise noted, all reactions were performed under the positive nitrogen pressure. Carbamates **2** were either purchased directly from Aldrich, Acros, TCI America or prepared using standard literature procedures.¹ All other reagents and solvents were purchased from Aldrich and used as received.

Preparation of 1-PMB-4-amino-5-ethoxycarbonylimidazole (1): Prepared analogously to the literature procedure for 1-benzyl derivative.²

Typical procedure for the preparation of 1-substituted-7-PMB xanthines (7): A three-necked flask equipped with a mechanical stirrer, thermocouple, condenser and nitrogen inlet was charged with 4-amino-5-ethoxycarbonyl-7-PMB-imidazole **1** (27.5 g, 0.1 mol), carbamate **2c** (12.4 g, 0.12 mol) and diglyme (150 mL). The well agitated mixture was heated to 70-80 C and a solution of potassium tert-butoxide (16.8 g, 0.15 mol, double the amount for O-aryl carbamates) in diglyme (100 mL) was added dropwise over a period of 30 minutes. Reaction was agitated for an additional 1 hour (12 hours for **7g**) and cooled to 20-25 C. Precipitated xanthine potassium salt **6b** was isolated by filtration, washed on the filter with THF (2x40 mL) and dried under vacuum to give the off-white product which can be used directly in the next step. Conversion of **6b** to **7b** was performed by dissolving **6b** solid in 10% NaOH (100 mL) and adjusting the pH of the solution to 6-7 with AcOH keeping the reaction temperature below 30 C. Precipitated

solid was collected by filtration, washed with water (3x100 mL) and dried under vacuum with nitrogen sweep to give xanthine **7b** (23.5 g, 82.0% yield) as off-white solid.

Typical procedure for the preparation of 1,3-substituted-7-PMB xanthines (9) via alkylation of potassium salts (6): A three-necked flask equipped with a mechanical stirrer, thermocouple, condenser and nitrogen inlet was charged with solid xanthine potassium salt **6f** (35.0 g, 0.1 mol), 2-(Boc-amino)-ethyl bromide (26.9 g, 0.12 mol), tetrabutylammonium bromide (1.6 g, 5 mmol), and dry THF (300 mL). The well agitated reaction mixture was heated to reflux (62-65 C) and agitated for 2 hours. Upon cooling and solvent removal, analytically pure product **9f** (41.0 g, 90.0% yield) was isolated by chromatography (silicagel, EtOAc:hexanes 1:1 vol/vol).

Typical procedure for the preparation of 1,3-substituted-7-PMB xanthines (9) via alkylation of xanthines (7): A three-necked flask equipped with a mechanical stirrer, thermocouple, condenser and nitrogen inlet was charged with solid xanthine **7g** (32.8 g, 0.1 mol), potassium carbonate (20.7 g, 0.15 mol), tetrabutylammonium bromide (1.6 g, 5 mmol), dry acetonitrile (300 mL), and bromoacetonitrile (14.4 g, 0.12 mol). The well agitated reaction mixture was heated to reflux (78-80 C) and agitated for 2 hours. Upon cooling and solvent removal, analytically pure product **9g** (31.6 g, 86.0% yield) was isolated by chromatography (silicagel, EtOAc:hexanes 1:1 vol/vol).

Spectroscopic data

1-[(4-methoxyphenyl)methyl]-4-amino-5-ethoxycarbonylimidazole (1): m.p. 109-111 °C, UV/vis (acetonitrile/water) λ_{max} 228.6 (2.0), 272.2 (1.6) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.30 (m, 2H), 7.18 (m, 2H), 6.91 (m, 2H), 5.32 (s, 2H), 4.32 (q, $J=7.1$ Hz, 2H), 3.85 (s, 3H), 1.37 (t, $J=7.1$ Hz, 3H); ^{13}C NMR (100 Mhz, CDCl_3): δ 161.0, 159.1, 138.9, 128.5, 128.4, 114.0, 101.8, 59.5, 55.1, 50.0, 14.4; HRMS (FAB) calcd for $\text{C}_{14}\text{H}_{18}\text{N}_3\text{O}_3$ $[\text{MH}]^+$ 276.1348, found 276.1351.

1-methyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7a): m.p. 201-203 °C, UV/vis (acetonitrile/water) λ_{max} 228.0 (3.0), 273.0 (1.0) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.52 (s, 1H), 7.25 (m, 2H), 6.83 (m, 2H), 5.35 (s, 2H), 3.74 (s, 3H), 3.34 (s, 3H); ^{13}C NMR (100 Mhz, CDCl_3): δ 158.9, 155.2, 151.1, 147.8, 142.4, 129.2, 129.0, 114.0, 105.6, 55.1, 48.3, 26.8; HRMS (FAB) calcd for $\text{C}_{14}\text{H}_{15}\text{N}_4\text{O}_3$ $[\text{MH}]^+$ 287.1144, found 287.1148.

1-ethyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7b): m.p. 209-211 °C, UV/vis (acetonitrile/water) λ_{max} 228.0 (3.0), 273.0 (1.0) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.58 (s, 1H), 7.28 (m, 2H), 6.87 (m, 2H), 5.39 (s, 2H), 4.02 (q, $J=7.0$ Hz, 2H), 3.78 (s, 3H), 1.22 (t, $J=7.0$ Hz, 3H); ^{13}C NMR (100 Mhz, DMSO-d_6): δ 159.0, 155.0, 150.8, 148.1, 142.5, 129.2, 129.0, 114.0, 105.7, 55.1, 48.4, 34.8, 13.2; HRMS (FAB) calcd for $\text{C}_{15}\text{H}_{17}\text{N}_4\text{O}_3$ $[\text{MH}]^+$ 301.1301, found 301.1297.

1-butyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7c): m.p. 183-185 °C, UV/vis (acetonitrile/water) λ_{max} 228.0 (3.0), 273.0 (1.0) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.60 (s, 1H), 7.32 (m, 2H), 6.91 (m, 2H), 5.43 (s, 2H), 4.0 (t, $J=7.4$ Hz, 2H), 3.82 (s, 3H), 1.67 (m, 2H), 1.42 (m, 2H), 0.98 (t, $J=7.3$ Hz, 3H); ^{13}C NMR (100

Mhz, CDCl₃): δ 159.9, 155.7, 151.5, 147.4, 140.5, 129.9, 128.8, 114.5, 106.9, 55.3, 50.0, 40.6, 30.1, 20.2, 13.8; HRMS (FAB) calcd for C₁₇H₂₁N₄O₃ [MH]⁺ 329.1614, found 329.1611.

1-allyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7d): m.p. 190-192 °C, UV/vis (acetonitrile/water) λ_{max} 202.9 (2.3), 225.0 (1.4), 271.0 (0.8) nm; ¹H NMR (400 MHz, CDCl₃): δ 7.55 (s, 1H), 7.28 (m, 2H), 6.85 (m, 2H), 5.90 (m, 1H), 5.39 (s, 2H), 5.25 (m, 1H), 5.16 (m, 1H), 4.58 (d, J= 5.5 Hz, 2H), 3.79 (s, 3H); ¹³C NMR (100 Mhz, CDCl₃): δ 159.8, 155.3, 151.2, 147.6, 140.7, 132.1, 129.9, 126.7, 117.3, 114.4, 106.8, 55.2, 49.9, 42.6; HRMS (FAB) calcd for C₁₆H₁₇N₄O₃ [MH]⁺ 313.1301, found 313.1295.

1-benzyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7e): m.p. 213-215 °C, UV/vis (acetonitrile/water) λ_{max} 208.7 (3.0), 271.0 (1.5) nm; ¹H NMR (400 MHz, CDCl₃): δ 7.50 (s, 1H), 7.45 (d, J=8.5 Hz, 2H), 7.30 (m, 5H), 6.87 (d, J=8.5 Hz, 2H), 5.39 (s, 2H), 5.15 (s, 2H), 3.79 (s, 3H); ¹³C NMR (100 Mhz, DMSO-d₆): δ 158.9, 155.0, 150.9, 148.0, 142.8, 137.8, 129.2, 126.9, 128.2, 127.3, 126.9, 114.0, 105.6, 55.1, 48.4, 42.8; HRMS (FAB) calcd for C₂₀H₁₉N₄O₃ [MH]⁺ 363.1457, found 363.1463.

1-cyclopropyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7f): m.p. 161-164 °C, UV/vis (acetonitrile/water) λ_{max} 227.0 (3.0), 274 (1.2); ¹H NMR (400 MHz, CDCl₃): δ 7.49 (s, 1H), 7.28 (m, 2H), 6.82 (m, 2H), 5.35 (s, 2H), 3.75 (s, 3H), 2.62 (m, 1H), 1.15 (m, 2H), 0.80 (br s, 2H); ¹³C NMR (100 Mhz, CDCl₃): δ 159.8, 156.8, 152.4, 147.6, 140.5, 129.7, 126.9, 114.4, 107.0, 55.2, 49.9, 24.3, 8.6; HRMS (FAB) calcd for C₁₆H₁₇N₄O₃ [MH]⁺ 313.1301, found 313.1298.

1-*tert*-butyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7g):

m.p. 162-164 °C, UV/vis (acetonitrile/water) λ_{max} 208.7 (3.0), 274.5 (1.3) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.41 (s, 1H), 7.30 (d, $J=8.6$ Hz, 2H), 6.87 (d, $J=8.6$ Hz, 2H), 5.34 (s, 2H), 3.79 (s, 3H), 1.73 (s, 9H); ^{13}C NMR (100 Mhz, CDCl_3): δ 159.8, 158.2, 152.9, 146.7, 140.1, 129.7, 127.1, 114.4, 107.9, 62.1, 55.3, 49.7, 30.2; HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{21}\text{N}_4\text{O}_3$ $[\text{MH}]^+$ 329.1614, found 329.1608.

1-phenyl-3,7-dihydro-7-[(4-methoxyphenyl)methyl]-1H-Purine-2,6-dione (7h): m.p.

240-242 °C, UV/vis (acetonitrile/water) λ_{max} 204.0 (2.6), 225.0 (1.6), 271.0 (1.0) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.60 (s, 1H), 7.38-7.45 (m, 3H), 7.25 (d, $J=8.5$ Hz, 2H), 7.21 (m, 2H), 6.83 (d, $J=8.5$ Hz, 2H), 5.32 (s, 2H), 3.74 (s, 3H); ^{13}C NMR (100 Mhz, CDCl_3): δ 159.9, 155.7, 151.4, 147.8, 140.9, 134.8, 130.0, 129.5, 129.3, 128.8, 128.7, 126.6, 114.5, 55.3, 50.1; HRMS (FAB) calcd for $\text{C}_{19}\text{H}_{17}\text{N}_4\text{O}_3$ $[\text{MH}]^+$ 349.1301, found 349.1306.

1-methyl-3-[2-(acetoxylethyl)-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-

2,6-dione (9a): m.p. 123 - 125 °C, UV/vis (acetonitrile/water) λ_{max} 225.0 (0.6), 273.4 (0.4) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.48 (s, 1H), 7.28 (m, 2H), 6.88 (m, 2H), 5.40 (s, 2H), 4.37 (m, 4H), 3.77 (s, 3H), 3.37 (s, 3H), 1.93 (s, 3H); ^{13}C NMR (100 Mhz, CDCl_3): δ 170.6, 159.7, 155.1, 151.2, 148.4, 140.5, 129.5, 127.2, 114.3, 106.8, 61.1, 55.1, 49.7, 42.1, 27.8, 20.7; HRMS (FAB) calcd for $\text{C}_{18}\text{H}_{21}\text{N}_4\text{O}_5$ $[\text{MH}]^+$ 373.1512, found 373.1502.

1-ethyl-3-(N-phenylpropionamide-2-yl)-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-

1H-Purine-2,6-dione (9b): m.p. 72-75 °C, UV/vis (acetonitrile/water) λ_{max} 215.0 (3.1), 230.0 (3.0), 271.0 (1.7) nm; ^1H NMR (400 MHz, CDCl_3): δ 8.12 (s, 1H), 7.46 (m, 3H),

7.25-7.31 (m, 4H), 7.06 (m, 1H), 6.87 (m, 2H), 5.64 (q, 1H), 5.38 (m, 2H), 4.11 (q, 2H), 3.75 (s, 3H), 1.82 (d, 3H), 1.23 (t, 3H); ^{13}C NMR (100 Mhz, CDCl_3): δ 167.6, 159.6, 154.5, 150.8, 147.6, 140.4, 137.6, 129.7, 128.6, 126.9, 124.1, 120.0, 114.1, 107.3, 55.1, 54.5, 49.7, 36.5, 14.6, 13.0; HRMS (FAB) calcd for $\text{C}_{24}\text{H}_{26}\text{N}_5\text{O}_4$ $[\text{MH}]^+$ 448.1985, found 448.1977.

1-butyl-3-(1-ethylpropyl)-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-2,6-dione (9c): m.p. 5-8 °C, UV/vis (acetonitrile/water) λ_{max} 216.9 (3.0), 275.7 (1.7) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.42 (m, 1H), 7.28 (m, 2H), 6.85 (m, 2H), 5.40 (s, 2H), 3.97 (t, $J=7.5$ Hz, 2H), 3.75 (s, 3H), 2.18 (m, 2H), 1.80 (m, 2H), 1.59 (m, 2H), 1.33 (m, 2H), 0.9 (t, $J=7.4$ Hz, 3H), 0.8 (t, $J=7.4$ Hz, 6H); ^{13}C NMR (100 Mhz, CDCl_3): δ 159.6, 155.1, 140.0, 129.6, 127.2, 114.1, 60.0, 55.0, 49.4, 41.0, 29.9, 24.8, 20.0, 13.6, 10.9; HRMS (FAB) calcd for $\text{C}_{22}\text{H}_{31}\text{N}_4\text{O}_3$ $[\text{MH}]^+$ 399.2396, found 399.2389.

1-allyl-3-benzyl-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-2,6-dione (9d): m.p. 103-105 °C, UV/vis (acetonitrile/water) λ_{max} 202.9 (2.4), 271.0 (0.9) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.50 (m, 3H), 7.28 (m, 5H), 6.85 (m, 2H), 5.91 (m, 1H), 5.40 (s, 2H), 5.28 (s, 2H), 5.18 (m, 2H), 4.62 (m, 2H), 3.80 (s, 1H); ^{13}C NMR (100 Mhz, CDCl_3): δ 159.5, 154.6, 150.8, 148.5, 140.5, 136.2, 132.1, 129.7, 128.5, 128.3, 127.6, 127.0, 117.2, 114.2, 106.8, 55.1, 49.7, 46.4, 43.2; HRMS (FAB) calcd for $\text{C}_{23}\text{H}_{31}\text{N}_4\text{O}_3$ $[\text{MH}]^+$ 403.1770, found 403.1773.

1-benzyl-3-(ethoxycarbonylmethyl)-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-2,6-dione (9e): m.p. 20-22 °C, UV/vis (acetonitrile/water) λ_{max} 218.1 (3.2), 272.2 (2.5) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.39 (m, 3H), 7.20 (m, 5H), 6.82 (m, 2H),

5.33 (s, 2H), 5.12 (s, 2H), 4.72 (s, 2H), 4.12 (q, J=7.1 Hz, 2H), 3.73 (s, 3H), 1.18 (t, J=7.1 Hz, 3H); ^{13}C NMR (100 Mhz, CDCl_3): δ 167.3, 159.2, 154.4, 150.8, 147.7, 140.6, 136.7, 129.2, 127.9, 127.8, 126.9, 126.8, 113.8, 106.3, 61.2, 54.7, 49.2, 43.9, 43.4, 13.6; HRMS (FAB) calcd for $\text{C}_{24}\text{H}_{25}\text{N}_4\text{O}_5$ $[\text{MH}]^+$ 449.1825, found 449.1806.

1-cyclopropyl-3-[2-(Boc-amino)ethyl]-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-2,6-dione (9f): m.p. 151-153 °C, UV/vis (acetonitrile/water) λ_{max} 213.4 (3.0), 274.5 (1.5) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.48 (s, 1H), 7.29 (m, 2H), 6.85 (m, 2H), 5.35 (s, 2H), 4.19 (t, 2H), 3.04 (s, 3H), 3.45 (br, 2H), 2.65 (m, 1H), 1.30 (s, 9H), 1.15 (m, 2H), 0.78 (m, 2H); ^{13}C NMR (100 Mhz, CDCl_3): δ 159.6, 156.1, 155.8, 140.4, 129.6, 127.1, 114.2, 78.9, 55.1, 49.7, 42.6, 39.2, 28.1, 24.8, 8.7; HRMS (FAB) calcd for $\text{C}_{19}\text{H}_{24}\text{N}_5\text{O}_8$ $[\text{MH}]^+$ 456.2247, found 456.2253.

1-tert-butyl-3-cyanomethyl-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-2,6-dione (9g): m.p. 106-108 °C, UV/vis (acetonitrile/water) λ_{max} 219.3 (3.2), 274.5 (2.0) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.50 (s, 1H), 7.29(m, 2H), 6.90 (m, 2H), 5.39 (s, 2H), 4.90 (s, 2H), 3.81 (s, 3H), 1.75 (s, 9H); ^{13}C NMR (100 Mhz, CDCl_3): δ 159.5, 156.8, 151.2, 145.8, 140.3, 129.5, 127.0, 114.5, 114.1, 107.9, 63.0, 55.1, 49.5, 30.2, 29.8; HRMS (FAB) calcd for $\text{C}_{19}\text{H}_{22}\text{N}_5\text{O}_3$ $[\text{MH}]^+$ 368.1723, found 368.1720.

1-phenyl-3-(1-cyclohexene-2-yl)-7-[(4-methoxyphenyl)methyl]-3,7-dihydro-1H-Purine-2,6-dione (9h): m.p. 15-18 °C, UV/vis (acetonitrile/water) λ_{max} 207.6 (2.6), 275.7 (1.0) nm; ^1H NMR (400 MHz, CDCl_3): δ 7.50 (s, 1H), 7.42 (m, 2H), 7.38 (m, 1H), 7.27 (m, 2H), 7.20 (m, 2H), 6.81 (m, 2H), 5.80 (br m, 1H), 5.62 (m, 1H), 5.45 (br, 1H), 5.32 (s, 2H), 3.72 (s, 3H), 2.38 (m, 1H), 2.11 (br, 1H), 1.91 (m, 3H), 1.70 (m, 1H); ^{13}C

NMR (100 Mhz, CDCl₃): δ 159.7, 155.1, 150.7, 148.9, 140.5, 135.4, 129.7, 129.2, 128.8, 128.6, 128.4, 127.3, 127.1, 114.2, 107.4, 55.1, 53.1, 49.6, 26.0, 24.0, 22.0; HRMS (FAB) calcd for C₂₅H₂₅N₄O₃ [MH]⁺ 429.1927, found 429.1932.

1-phenyl-3,7-dihydro-1H-Purine-2,6-dione (10): compound is described in the literature;² m.p. dec. > 350 °C, ¹H NMR (400 MHz, DMSO-d₆): δ 8.02 (s, 1H), 7.33 (m, 5H); ¹³C NMR (100 Mhz, DMSO-d₆): δ 155.1, 151.1, 147.6, 141.0, 136.2, 129.3, 128.8, 127.8, 106.6; HRMS (FAB) calcd for C₁₁H₉N₄O₂ [MH]⁺ 229.0726, found 229.0730.

1-methyl-3-[2-(acetoxy)ethyl]-3,7-dihydro-1H-Purine-2,6-dione (11): compound is described in the literature;³ m.p. 197-198 °C, ¹H NMR (400 MHz, CDCl₃): δ 7.88 (s, 1H), 4.42 (br, 4H), 3.40 (s, 3H), 1.95 (s, 3H); HRMS (FAB) calcd for C₁₀H₁₃N₄O₄ [MH]⁺ 253.0937, found 253.0940.

References

1. Thavonekham, B. *Synthesis* **1997**, 1189, and refs. cited therein.
2. Bridson, P.K.; Wang, X. *Synthesis* **1995**, 7, 855.
3. Wells, J.N.; Garst, J.E.; Kramer, G.L. *J. Med. Chem.* **1981**, 24, 954.

¹H and ¹³C NMR Spectra

Current Data Parameters
 NAME Apr19-2004
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040419
 Time 11.05
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 287.4
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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

NUC1 1H
 P1 10.00 usec
 PL1 -1.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1299509 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 FIP 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 200.08500 Hz/cm

Current Data Parameters
NAME Apr19-2004
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040419
Time 11.32
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 6192
DN 19.990 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 19.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127801 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
EX 20.00 cm
F4P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
P0MCM 10.00000 ppm/cm
HZCM 1006.12775 Hz/cm

Current Data Parameters

NAME Feb28-2001
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010228
Time 11:28
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.145 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DM 50.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1 1H
P1 10.00 usec
PL1 -1.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300291 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMM 0.50000 ppm/cm
HZCM 200.06502 Hz/cm

Current Data Parameters
 NAME Feb28-2001
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010301
 Time 0.59
 INSTRUM spect
 PULPROG 5 mm QNP 1H
 TO 799530
 SOLVENT CDC13
 NS 2048
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.363387 Hz
 AQ 1.3042164 sec
 RG 8192
 DN 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.00 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 17.00 dB
 PL13 19.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6132918 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.66 Hz
 F2P 0.000 ppm
 F2 10.00000 ppm/cm
 FPMCM 1006.13287 Hz/cm

Current Data Parameters

NAME Oct10-2002
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters

Date_ 20021010
Time 11.49
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT COCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1 1H
P1 10.00 usec
PL1 -1.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300169 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
FIP 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPNOM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

Current Data Parameters
 NAME Oct10-2002
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021011
 Time 3.33
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 5120
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 8192
 DM 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.00 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CDPPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 17.00 dB
 PL13 19.00 dB
 SF02 400.1318005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6128113 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.56 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 10.00000 ppm/cm
 HZCM 1006.12805 Hz/cm

Current Data Parameters
NAME Feb27-2001
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010227
Time 10.54
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65535
SOLVENT CDCl3
NS 8
DS 2
SWH 8276.145 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2 0.000 ppm
F2 0.00 Hz
PPHCH 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

Current Data Parameters
NAME Feb27-2001
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010228
Time 21.00
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 4096
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 19.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCK 10.00000 ppm/cm
HZCM 1006.12769 Hz/cm

Current Data Parameters
NAME 78118-47
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010321
Time 7.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 8250.825 Hz
FIDRES 0.251795 Hz
AQ 1.9857908 sec
RG 456.1
DM 50.500 Hz
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 12.90 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300177 MHz
WDW ra
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
P1MCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

Current Data Parameters
 NAME Mar21-2001
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010322
 Time 1.42
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 7168
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.383367 Hz
 AQ 1.3042164 sec
 RG 4096
 DM 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.00 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 17.00 dB
 PL13 19.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127776 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PRACH 10.00000 ppm/cm
 HZCM 1008.12775 Hz/cm

42.566
 49.911
 59.220

76.589
 77.001
 77.199

100.797
 117.412
 117.708
 129.459
 129.459
 132.140
 140.673
 147.508
 157.298
 157.521
 159.803

Current Data Parameters
 NAME Apr09-2001
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010409
 Time 16.25
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.145 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 812.7
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 -1.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300174 MHz
 NDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPBPM 0.50000 ppm/cm
 HZCM 200.09502 Hz/cm

Current Data Parameters

NAME Feb28-2001
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010228
Time 11:21
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1 1H
P1 10.00 usec
PL1 -1.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SF 32768
SF 400.1300289 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters

CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06502 Hz/cm

Current Data Parameters
NAME Feb28-2001
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010228
Time 22.59
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 25125.629 Hz
FIDRES 0.363387 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 8.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0300000 sec
d12 0.0000200 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 3.00 dB
SF01 100.6237858 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 19.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127763 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMC 10.00000 ppm/cm
HZCM 1006.12775 Hz/cm

Current Data Parameters
NAME Mar14-2001
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010314
Time 15.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.145 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -1.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.728 ppm
F1 3892.60 Hz
F2P -0.068 ppm
F2 -27.19 Hz
PPMCM 0.48981 ppm/cm
HZCM 195.98923 Hz/cm

Current Data Parameters	
NAME	Mar14-2001
EXPNO	21
PROCNO	1

[illegible]

```
===== CHANNEL f1 =====
NUC1      13C
P1        11.00 usec
PL1       3.00 dB
SF01     100.6237959 MHz
```

```

CHANNEL 12
wz1z15
1H
80.00 usec
-1.00 dB
17.00 dB
19.00 dB
400.131505 MHz

```

F2 - Processing parameters	
SI	32768
SF	100.6127740 MHz
MDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters	
CX	20.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCH	10.00000 ppm/cm
Hz/cm	1000.00000 Hz/cm

Current Data Parameters

NAME Mar15-2001
 EXPNO 30
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20010315
 Time 18.56
 INSTRUM spect
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 574.7
 GM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

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

NUC1 1H
 P1 10.00 usec
 PL1 -1.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300329 MHz
 WDW EM
 SSF 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCH 0.50000 ppm/cm
 HZCH 200.06502 Hz/cm

Current Data Parameters
NAME Mar15-2001
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010316
Time 1.59
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7168
DS 4
SWH 25125.629 Hz
FIDRES 0.38387 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 3.00 dB
SF01 100.627955 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 19.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127732 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
FIP 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCH 10.00000 ppm/cm
HZCM 1006.12769 Hz/cm

Current Data Parameters

NAME N_Me_xanthine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031017
Time 13.46
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.251795 Hz
AQ 1.9857908 sec
RG 256
DM 60.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec

***** CHANNEL f1 *****

MUC1 1H
P1 12.90 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 16384
SF 400.1300157 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

10 NMR plot parameters

CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

Current Data Parameters

NAME N_Me_xanthine
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031017
Time 13.55
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT DMS
NS 274
DS 2
SWH 31746.031 Hz
FIDRES 0.404406 Hz
AQ 1.0322419 sec
RG 2048
DM 15.750 usec
DE 6.00 usec
TE 300.0 K
O1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SF01 100.6254358 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 21.00 dB
SF02 400.1315005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127825 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.56 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCH 10.00000 ppm/cm
HZCM 1006.12787 Hz/cm

NAME	80531-116
EXPNO	1
PROCNO	1

Date_ 20031023
Time 16.27
INSTRUM spect

INSTRUM	5 mm QNP 1H	spec
PROBHD	ZQ30	
PULPRG	32768	
TD	COC13	
SOLVENT	16	
NS	0	
DS	8250 825 Hz	
SWH	0.251795 Hz	
FIDRES	1.9857908 sec	
Acq	456.1	
RG	60.600 usec	
DW	6.00 usec	
DE	300.0 K	
TE	2.00000000 sec	
D1		

```
===== CHANNEL f1 =====
```

NUC1	1H
P1	12.90 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

SI	16384
SF	400.1300177 MHz
WDW	no

NUM	NO
BSS	0
LB	0.00 Hz
GB	0
PC	1.00

CX	20.00	cm
F1P	10.000	ppm
F1	4001.30	Hz
F2P	0.000	ppm
F2	0.00	Hz
PPMCM	0.50000	ppm/cm
HZCM	200.08500	Hz/cm

Current Data Parameters

NAME 80531-124
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031105
Time 14.31
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8250.825 Hz
FIDRES 0.251795 Hz
AQ 1.9857908 sec
RG 40.3
DM 60.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

***** CHANNEL f1 *****

NUC1 1H
P1 12.90 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters

SI 16384
SF 400.1300177 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCH 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

Current Data Parameters

NAME 80531-124
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031105
Time 14.36
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 414
DS 2
SWH 31746.031 Hz
FIDRES 0.484405 Hz
AQ 1.0322419 sec
RG 4096
DM 15.750 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SF01 100.6254358 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 21.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127863 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
FIP 200.000 ppm
F1 20122.56 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCA 10.00000 ppm/cm
HZCM 1006.12787 Hz/cm

Current Data Parameters

NAME 79956-25
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20021002
Time 16.12
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.251795 Hz
AQ 1.9857908 sec
RG 322.5
DW 60.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

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

NUC1 1H
P1 12.90 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 16384
SF 400.1300127 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

Current Data Parameters
NAME 79556-26
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021002
Time 16.18
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 388
DS 2
SMH 31746.031 Hz
FIDRES 0.484406 Hz
AQ 1.0322419 sec
RG 2048
DW 15.750 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SF01 100.6254356 MHz

===== CHANNEL f2 =====
CPDPRG2 zgpg30
NUC2 1H
P2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 21.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127932 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
FIP 200.000 ppm
F1 20122.56 Hz
F2 0.000 ppm
F2 0.00 Hz
PRMCM 10.00000 ppm/cm
HZCM 1006.12793 Hz/cm

Current Data Parameters
 NAME Oct14-2003
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031014
 Time 15.40
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 181
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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

NUC1 1H
 P1 10.00 usec
 PL1 -1.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300387 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 200.06502 Hz/cm

2

Current Data Parameters

NAME 75956-31
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20021009
Time 18.32
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.251795 Hz
AQ 1.9857908 sec
RG 181
DM 60.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec

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

NUC1 1H
P1 12.90 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 16384
SF 400.1300177 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
FIP 10.000 ppm
F1 4001.30 Hz
F2 0.000 ppm
F2 0.00 Hz
PPMCH 0.50000 ppm/cm
HZCM 200.08500 Hz/cm

[illegible]

Current Data Parameters

NAME Oct28-2003
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031028
Time 15.21
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1 1H
P1 10.00 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300391 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ID NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06502 Hz/cm

Current Data Parameters

NAME Oct28-2003
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031028
Time 15.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 6192
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 19.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127886 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
FAP 200.000 ppm
F1 20122.56 Hz
F2 0.000 ppm
F2 0.00 Hz
PPMCH 10.00000 ppm/cm
HZOM 1006.12787 Hz/cm

22.019
24.011
26.016

49.644
53.138
55.123

76.681
77.318
77.600

107.381
114.192
127.109
127.321
129.385
128.637
128.823
129.158
129.668
135.401
140.497
148.913
150.712
155.079
159.084

ppm

ppm