Scope and Limitations of the Nitro-Mannich Reaction for the Stereoselective Synthesis of 1,2-Diamines.

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General experimental.

For all non-aqueous chemistry, glassware was rigorously flame-dried and an inert (N₂) atmosphere was maintained throughout. Thin layer chromatography was carried out using Merck 5549 60F silica-aluminium plates with potassium permanganate for visualization. For flash column chromatography, BDH 60 silica gel was used and direct column loading was employed in all cases. All imines and β -nitro amines were used immediately, or stored under N₂ in the freezer for the minimum amount of time. Unless stated otherwise, compound data shows good concordance with literature values. NMR data was collected at 400 or 500MHz. Data was manipulated directly from the spectrometer or via a networked PC with Win-NMR software. All samples were analysed in C₆D₆ unless otherwise stated. Reference values for residual solvents were taken as δ = 7.17 (C₆D₆), 7.27 (CDCl₃) and 2.50 (d⁶-DMSO) for ¹H-NMR; δ = 128.06 (C₆D₆) and 77.16 (CDCl₃) for ¹³C-NMR. Multiplicities for coupled signals are denoted thus: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants (J) are given in Hz. Mass spectrometric high-resolution data are quoted to four decimal places (0.1 mDa) with error limits for acceptance of ± 5.0 ppm (defined as calcd./found mass 10^{-6}). Infrared data was collected using sodium chloride discs. Both oils and solids were analysed neat. All solvents and reagents were used as supplied or purified using standard techniques.

General procedure for synthesis of imines. Rigorously dried 4Å molecular sieves (1.0 g per mmol) were added to a solution of amine in DCM (5 mL per mmol) under N₂ at rt. and after a period of 5 min carbonyl compound (1 equiv.) was added. The mixture was left to stir for 14 h at rt before filtration through celite and removal of solvents *in vacuo*, to yield the crude imine. Imines were judged >95% pure by ¹H NMR, but were purified if possible.

10a. Prepared using general procedure above, 2-methoxybenzylamine (4.00 g, 29.10 mmol) and benzaldehyde (2.94 mL, 29.10 mmol) gave crude **10a** (5.46 g, 98%) as a colourless oil: IR $\nu_{\max}$ (film) 2835, 1645, 1492, 1243, 1030, 751, 694cm⁻¹; ¹H NMR δ 3.33 (3H, s), 4.95 (2H, s), 6.58 (1H, d, *J* 7.6), 6.94 (1H, t, *J* 1.2), 7.13 (4H, m), 7.51 (1H, d, *J* 7.5), 7.77 (2H, dd, *J* 7.6, 2.0), 8.15 (1H, s); ¹³C NMR δ 54.9, 59.6, 110.4, 120.9, 128.1, 128.3, 128.7, 128.9, 129.7, 130.6, 137.3, 157.5, 161.6; m/z (EI+) 225 (59%, M⁺), 196 (16%), 121 (100%), 91 (92%), 65 (13%); HRMS C₁₅H₁₅NO calcd. 225.1154, found 225.1152.

10b. Prepared using general procedure above, 4-methoxybenzylamine (4.00 g, 29.1 mmol) and benzaldehyde (2.94 mL, 29.10 mmol) gave crude **10b** (5.24 g, 94%) as a colourless oil. All spectral data was in agreement with that in the literature.¹

10c. Prepared using general procedure above except using neutral alumina as dessicant, 4-methoxyphenylamine (10.00 g, 81.2 mmol) and benzaldehyde (8.20 mL, 81.2 mmol) gave crude **10c** (14.41 g, 84%) as a grey platelets mp 65-7 °C (lit. mp 65 °C). All spectral data was in agreement with that in the literature.²

10d. Prepared using general procedure above, 2-methylbenzylamine (274 mg, 2.00 mmol) and benzaldehyde (144 mg, 2.00 mmol) gave crude PMP protected **10d** (380 mg, 99%) as a clear, pale-yellow oil; IR $\nu_{\max}$ (film) 3062, 2844, 1702, 1646, 1540, 750 cm^{-1} ; ^1H NMR δ 2.21 (3H, s), 4.59 (2H, s), 7.07 – 7.16 (6H, m), 7.37 (1H, m), 7.73 (2H, m), 8.00 (1H, s); ^{13}C NMR δ 19.3, 63.0, 126.4, 127.4, 128.1, 128.9, 129.6, 130.4, 130.7, 136.3, 137.1, 138.3, 161.4; m/z (EI+) 209 (41%, M^+), 208 (25% $\text{M}^+ - \text{H}^+$), 105 (100%, CH_2ArMe^+), 77 (26%, Ph^+); HRMS $\text{C}_{15}\text{H}_{15}\text{N}$ calcd. 209.1205, found 209.1211.

12. Prepared using general procedure above, 2-methoxybenzylamine (274 mg, 2.00 mmol) and 2-furaldehyde (0.17 mL, 2.00 mmol) gave crude **12** (430 mg, 99%) as a clear, pale-yellow oil; IR $\nu_{\max}$ (film) 2835, 1589, 1491, 1438, 1244, 1049, 753 cm^{-1} ; ^1H NMR δ 3.27 (3H, s), 4.85 (2H, s), 5.99 (1H, d, J 1.7), 6.54 (1H, d, J 8.2), 6.62 (1H, d, J 3.3), 6.90 (1H, t, J 8.2), 7.02 (1H, d, J 1.7), 7.09 (1H, t, J 8.1), 7.45 (1H, d, J 8.2), 8.01 (1H, s); ^{13}C NMR δ 54.9, 59.8, 110.4, 111.8, 112.3, 120.9, 127.8, 128.3, 129.9, 144.3, 150.7, 153.2, 157.5; m/z (CI^+) 216 (100%, MH^+), 186 (51%), 121 (98%), 109 (20%), 91 (72%); HRMS $\text{C}_{13}\text{H}_{14}\text{NO}_2$ (MH^+) calcd. 216.1025, found 216.1020.

13. Prepared using general procedure above, 2-methoxybenzylamine (274 mg, 2.00 mmol) and 2-thiophencarboxaldehyde (0.19 mL, 2.00 mmol) gave crude **13** (460 mg, >99%) as a clear, pale-yellow oil; IR $\nu_{\max}$ (film) 2835, 1633, 1587, 1491, 1432, 1242, 1030, 754 cm^{-1} ; ^1H NMR δ 3.29 (3H, s), 4.88 (2H, s), 6.54 (1H, d, J 8.2), 6.65 (1H, m), 6.79 (1H, d, J 5.0), 6.88 (2H, m), 7.10 (1H, t, J 8.0), 7.49 (1H, d, J 7.5), 8.12 (1H, s); ^{13}C -NMR δ 54.8, 59.0, 109.8, 120.9, 127.3, 127.8, 128.3, 128.9, 129.7, 130.3, 143.7, 155.0, 157.5; m/z (EI+) 231 (46%, M^+), 202 (40%), 125 (18%), 121 (100%, $\text{C}_8\text{H}_9\text{O}$), 91 (100%), 65 (17%); HRMS $\text{C}_{13}\text{H}_{13}\text{NOS}$ calcd. 231.0718, found 231.0720.

14. Prepared using general procedure above, 2-methoxybenzylamine (685 mg, 5.00 mmol) and 2-pyrrolecarboxaldehyde (476 mg, 5.00 mmol) gave **14** (1.07 g, 99%) as a pale-yellow solid. Recrystallisation from hot EtOAc/petroleum ether gave **14** (0.95 g, 89%) as a clear crystalline solid mp 97.0 – 99.0 °C; IR $\nu_{\max}$ (solid) 1639, 1490, 1461, 1422, 1243, 1028, 755 cm^{-1} ; ^1H NMR δ 3.24 (3H, s),

4.77 (2H, s), 6.16 (1H, t, $J = 2.5$), 6.36 (2H, m), 6.51 (1H, d, $J 8.1$), 6.85 (1H, t, $J 7.5$), 7.09 (1H, t, $J 7.8$), 7.28 (1H, d, $J 7.4$); ^{13}C NMR δ 54.8, 59.0, 109.6, 110.4, 115.3, 120.9, 122.8, 128.3, 128.5, 129.8, 130.7, 153.7, 157.5; m/z (EI^+) 215 (10%, MH^+), 214 (57%, M^+), 183 (7%, $\text{M}^+ - \text{OMe}$), 121 (65%, CH_2ArOMe), 107 (11%, $\text{C}_7\text{H}_9\text{N}$); HRMS $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$ calcd. 214.11061, found 214.10991; Anal. calcd. for $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$: C, 72.87; H, 6.59; N, 13.07; found C, 72.88; H, 6.63; N, 13.14%.

15. Prepared using general procedure above, 2-methoxybenzylamine (685 mg, 5.00 mmol) and 2-*N*-Methylpyrrole-carboxaldehyde (546 mg, 5.00 mmol) gave crude **15** (1.14 g, 99%) as a clear oil; IR ν_{max} (film) 2835, 1641, 1588, 1490, 1426, 1241, 1030, 753 cm^{-1} ; ^1H NMR δ 3.37 (3H, s), 3.62 (3H, s), 4.84 (2H, s), 6.16 (1H, m), 6.30 (1H, s), 6.48 (1H, m), 6.61 (1H, d, $J 7.2$), 6.95 (1H, t, $J 7.4$), 7.14 (1H, t, $J 7.5$), 7.48 (1H, d, $J 7.4$), 8.12 (1H, s); ^{13}C NMR δ 36.7, 54.9, 62.8, 108.3, 110.3, 117.9, 120.9, 128.0, 128.3, 129.3, 129.4, 130.5, 154.0, 157.4; m/z (EI^+) 228 (100%, M^+), 121 (51%, CH_2ArOMe), 94 (13%, $\text{C}_6\text{H}_8\text{N}$), 91 (44%); HRMS $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}$ calcd. 228.12627, found 228.12527.

16. Prepared using general procedure above except reaction performed at $-78\text{ }^\circ\text{C}$, 2-methoxybenzylamine (685 mg, 5.00 mmol) and hexanal (501 mg, 5.00 mmol) gave crude **16** (1.10 g, 99%) as a clear oil. The imine was used immediately as rapid decomposition was noted at $25\text{ }^\circ\text{C}$; IR ν_{max} (film) 2928, 2856, 1670, 1601, 1588, 1492, 1438, 1243, 1031, 753 cm^{-1} ; ^1H NMR δ 0.81 (3H, t, $J 6.8$), 1.20 (4H, m), 1.44 (2H, p, $J 7.4$), 2.15 (2H, m), 3.33 (3H, s), 4.76 (2H, s), 6.57 (1H, d, $J 8.0$), 6.95 (1H, t, $J 7.2$), 7.12 (1H, t, $J 8.4$), 7.53 (2H, m); ^{13}C NMR δ 14.3, 22.9, 25.9, 31.8, 36.2, 54.7, 59.8, 110.2, 120.8, 127.9, 129.0, 129.4, 157.4, 165.1; m/z (EI^+) 219 (5%, M^+), 163 (53%, $\text{M}^+ - \text{Bu}$), 136 (26%, NCH_2ArOMe), 132 (51%), 121 (83%, CH_2ArOMe); HRMS $\text{C}_{14}\text{H}_{21}\text{NO}$ calcd. 219.1623, found 219.1614.

17. Prepared using general procedure above, 2-methoxybenzylamine (274 mg, 2.00 mmol) and isobutyraldehyde (144 mg, 2.00 mmol) gave crude **17** (380 mg, >99%) as a clear, pale-yellow oil; IR ν_{max} (film) 2691, 1670, 1601, 1588, 1491, 1463, 1242, 1031, 752 cm^{-1} ; ^1H NMR δ 1.01 (6H, d, $J 6.9$), 2.33 (1H, sep, $J 6.9$), 3.34 (3H, s), 4.75 (2H, s), 6.57 (1H, dd, $J 8.1, 0.9$), 6.95 (1H, td, $J 7.5, 1.1$), 7.12 (1H, td, $J 7.5, 1.1$), 7.49 (2H, m); ^{13}C NMR δ 19.4, 34.3, 54.8, 59.4, 110.3, 120.9, 127.8, 129.1, 129.3, 157.4, 169.6, m/z (EI^+) 191 (6%, M^+), 148 (23%, $\text{M}^+ - \text{iPr}$), 121 (100%, $\text{C}_8\text{H}_9\text{O}$), 91 (62%, C_7H_7), 51 (50%); HRMS $\text{C}_{12}\text{H}_{17}\text{NO}$ calcd. 191.1310, found 191.1316.

18. A solution of 2-methoxybenzylamine (678 mg, 5.00 mmol) in acetone (20 mL) was stirred with $4\text{ \AA}$ molecular sieves (5.00g) for 18 h. Filtration (celite) and removal of solvent in *vacuo* gave crude **18**

(885 mg, >99%) as a clear oil. Material contained <5% residual 2-methoxybenzylamine, attempted purification by chromatography or treatment with solid supported acids gave starting materials; IR $\nu_{\max}$ (film) 2940, 1665, 1491, 1241, 1029, 754 cm^{-1} ; ^1H NMR δ 1.24 (3H, s), 1.90 (3H, s), 3.36 (3H, s), 4.58 (2H, s), 6.61 (1H, d, J 8.0), 7.00 (1H, t, J 7.6), 7.14 (1H, t, J 7.5), 7.82 (1H, d, J 7.9); ^{13}C NMR δ 18.0, 29.3, 49.9, 54.8, 110.1, 120.8, 127.9, 129.1, 129.9, 157.3, 166.3; m/z (EI^+) 178 (6%, MH^+), 177 (35%, M^+), 146 (20%, $\text{M}^+ - \text{OMe}$), 121 (100%, CH_2ArOMe); HRMS $\text{C}_{11}\text{H}_{15}\text{NO}$ calcd. 177.1154, found 177.1160.

General procedure for reduction of nitro group³ and subsequent formation of imidazolidinone.

A triple evacuation/ N_2 fill was carried out on a suspension of samarium metal (40 mesh, 5.26 g, 35.0 mmol) and 1,2-diiodo-ethane (9.16 g, 32.5 mmol) in a Schlenk tube. The mixture was diluted with THF (50 mL) and stirred for 1 h under N_2 . A further 150 mL THF was added and the mixture stirred for 2 h until an intense, deep blue solution was obtained. Crude β -nitro-amine (5.00 mmol) in MeOH/THF (1:1, 30 mL) was added and the mixture stirred for 16 h at rt. A solution of oxalic acid (8.60 g) in water (100 mL) was added and the mixture filtered (celite) and the organics removed in *vacuo*. The resulting aqueous phase was basified to >pH12 (2 M, NaOH) and extracted with EtOAc. The combined organics were washed with satd. aq. $\text{Na}_2\text{S}_2\text{O}_3$ and satd. brine before drying (MgSO_4) and evaporation to yield crude, mono-protected 1,2-diamine.

A solution of phosgene (20% in toluene, 2.65 mL, 5.00 mmol) was added drop wise to a solution of the crude diamine from above in CH_2Cl_2 (30 mL) and Et_3N (1.50 mL, 10.0 mmol) at -20°C . After addition the mixture was allowed to warm to 25°C over 30 min, 5M NaOH (20 mL) was added, the organic phase separated, dried (MgSO_4) and evaporated to a yellow/brown highly viscous oil. After dissolving in the minimum amount of hot EtOAc/petroleum ether, the product imidazolidinone was crystallised and obtained as a solid. Further recrystallisation (EtOAc/petroleum ether mixtures) to remove the minor diastereoisomer was carried out where indicated. Spectroscopic data.

cis-20a. Prepared using general procedure above, crude **11a** (5.00 mmol, *anti/syn* 90/10) gave after crystallisation from hot EtOAc/petroleum diastereomerically pure **10a** (1.14 g, 74%) as colourless needles. Further recrystallisation afforded colourless needles suitable for single crystal X-ray structure determination; mp $136.0\text{--}138.0^\circ\text{C}$; IR $\nu_{\max}$ (solid) 1740, 1687, 1460, 1233, 1030, 762, 704 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.83 (3H, t, J 7.5), 1.12 (2H, m), 3.64 (3H, s), 3.88 (2H, m), 4.62 (1H, d, J 8.7), 4.88 (1H, d, J 15.3), 5.47 (1H, br s), 6.87 (1H, d, J 8.0), 6.97 (1H, t, J 7.5), 7.16–7.36 (6H, m); ^{13}C NMR (CDCl_3) δ 10.9, 25.0, 39.8, 55.1, 56.8, 63.2, 110.2, 120.5, 125.6, 128.0, 128.2, 128.3, 128.6, 129.9,

136.4, 157.1, 162.6; m/z (EI^+) 311 (15%, MH^+), 310 (68%, M^+), 189 (16%, $M^+ - CH_2ArOMe$); HRMS $C_{19}H_{22}N_2O_2$ calcd. 310.1681, found 310.1677; Anal. calcd. for $C_{19}H_{22}N_2O_2$: C, 73.52; H, 7.14; N, 9.03; found C, 73.66; H, 7.20; N, 8.91.

trans-20a. Prepared using general procedure above, crude **11a** (5.00 mmol, *anti/syn* 25/75) gave after crystallisation from hot EtOAc/petroleum ether **20a** as a yellow solid (701 mg, 71%, *trans/cis* 70/30). Further recrystallisation afforded diastereomerically pure **trans-20a** (405 mg, 41 %) as colourless needles; mp 121.0 – 123.0 °C; IR ν_{max} (film) 3225, 2964, 2241, 1693, 1492, 1460, 1244, 732 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.78 (3H, t, J 6.9), 1.35 – 1.53 (2H, m), 3.30 (1H, q, J 6.4), 3.55 (3H, s), 3.76 (1H, d, J 15.5), 3.93 (1H, d, J 6.2), 4.65 (1H, d, J 15.3), 5.58 (1H, br s), 6.68 (1H, d, J 8.2), 6.78 (1H, t, J 7.4), 7.06 (1H, d, J 7.4), 7.10 – 7.26 (6H, m); ^{13}C NMR ($CDCl_3$) δ 9.7, 28.2, 39.8, 55.1, 60.7, 65.4, 110.2, 120.4, 124.9, 127.3, 128.0, 128.3, 128.7, 129.8, 140.4, 157.6, 161.9; m/z (EI^+) 311 (26%, MH^+), 310 (50%, M^+), 189 (16%); HRMS $C_{19}H_{22}N_2O_2$ calcd. 310.1681, found 310.1684; Anal. calcd. for $C_{19}H_{22}N_2O_2$: C, 73.52; H, 7.14; N, 9.03; found C, 73.46; H, 7.13; N, 9.13.

20b. Prepared using general procedure above, crude **19b** (5.00 mmol, *anti/syn* 90/10) gave after crystallisation from hot EtOAc/petroleum ether diastereomerically pure **20b** as a yellow solid (1.30 g, 87%). Further recrystallisation afforded colourless needles suitable for single crystal X-ray structure determination; mp 138.0 – 140.0 °C; IR ν_{max} (solid) 1686, 1491, 1456, 1237, 751 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.81 (3H, t, J 7.4), 1.22 (2H, p, J 7.4), 3.73-3.77 (4H, m), 3.84 (1H, d, J 15.3), 4.63 (1H, d, J 8.2), 4.77 (1H, d, J 15.3), 4.90 (1H, br-s), 6.22 (1H, d, J 3.2), 6.37 (1H, m), 6.82 (1H, d, J 8.2), 6.90 (1H, t, J 7.4), 7.21 (2H, m), 7.39 (1H, s); ^{13}C NMR ($CDCl_3$) δ 10.7, 24.4, 40.0, 55.3, 56.4, 57.3, 109.4, 110.3, 120.5, 125.5, 128.6, 128.7, 129.8, 142.5, 150.5, 157.6, 161.7; m/z (EI^+) 300 (43%, M^+), 219 (26%), 179 (47%), 121 (100%, $M^+ - CH_2ArOMe$); HRMS $C_{17}H_{20}N_2O_3$ calcd. 300.1474, found 300.1460; Anal. calcd. for $C_{17}H_{20}N_2O_3$: C, 67.97; H, 6.72; N, 9.33; found C, 67.63; H, 6.69; N, 9.08.

20c. Prepared using general procedure above, crude **19c** (5.00 mmol, *anti/syn* 80/20) gave after crystallisation from hot EtOAc/petroleum ether diastereomerically pure **20c** as a yellow solid (932 mg, 59%). Further recrystallisation afforded colourless needles suitable for single crystal X-ray structure determination; mp 156.0 – 158.0 °C; IR ν_{max} (solid) 3225, 1684, 1460, 721 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.89 (3H, t, J 7.4), 1.32 (2H, m), 3.80 (4H, m), 3.95 (1H, d, J 15.3), 4.82 (1H, d, J 15.3), 4.90 (1H, d, J 8.0), 5.24 (1H, br-s), 6.88-7.09 (4H, m), 7.22-7.38 (3H, m); ^{13}C NMR ($CDCl_3$) δ 10.9, 24.2, 39.9, 55.2, 57.0, 59.0, 110.2, 120.5, 125.6, 126.1, 126.6, 128.3, 129.8, 130.0, 139.6, 157.6, 161.8; m/z (EI^+) 316

(40%, M^+), 195 (11%), 179 (14%), 138 (100%); HRMS $C_{17}H_{20}N_2O_2S$ calcd. 316.1245, found 316.1253; Anal. calcd. for $C_{17}H_{20}N_2O_2S$: C, 64.53; H, 6.37; N, 8.85 found C, 64.32; H, 6.42; N, 8.48.

20e. Prepared using general procedure above, crude **19e** (5.00 mmol, *anti/syn* 75/25) gave after crystallisation from hot EtOAc/petroleum diastereomerically pure **20e** as a yellow solid (1.09 g, 69%). Further recrystallisation afforded colourless needles suitable for single crystal X-ray structure determination; mp 171.8 – 174.2 °C; IR $\nu_{\max}$ (solid) 3221, 1690, 1460, 1234, 1028, 774, 721 cm^{-1} ; 1H NMR (d^6 -DMSO @ 95 °C) δ 0.71 (3H, t, J 7.4), 1.11 (2H, m), 3.38 (3H, s), 3.70 (4H, m), 3.78 (1H, d, J 15.5), 4.50 (1H, d, J 15.5), 4.70 (1H, d, J 6.5), 5.89 (1H, m), 5.98 (1H, t, J 3.0), 6.39 (1H, br-s), 6.63 (1H, t, J 2.5), 6.92 (2H, m), 7.12 (1H, d, J 7.2), 7.23 (1H, t, J 8.2); ^{13}C NMR ($CDCl_3$) δ 10.9, 25.3, 33.9, 40.7, 55.5, 55.8, 56.5, 107.7, 109.4, 110.7, 120.8, 122.5, 125.6, 127.1, 129.0, 130.6, 158.2, 162.2; m/z (EI^+) 313 (43%, M^+), 162 (20%), 135 (100%); HRMS $C_{18}H_{23}N_3O_2$ calcd. 313.1790, found 313.1800; Anal. calcd. for $C_{18}H_{23}N_3O_2$: C, 68.97; H, 7.40; N, 13.41; found C, 68.77; H, 7.33; N, 13.32.

20f. Prepared using general procedure above, crude **19f** (5.00 mmol, *anti/syn* 90/10) gave after purification by flash column chromatography (2:1 hexane:EtOAc) diastereomerically pure **20f** as a clear oil (1.26 g, 83%); IR $\nu_{\max}$ (film) 3237, 2933, 1697, 1492, 1460, 1243, 1104, 1029, 754 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.87 (3H, t, J 7.0), 0.96 (3H, t, J 7.4), 1.23 (6H, m), 1.40-1.69 (4H, m), 3.46 (2H, m), 3.83 (3H, s), 4.26 (1H, d, J 15.1), 4.57 (1H, d, J 15.1), 4.99 (1H, br-s), 6.85 (1H, dd, J 8.2, 0.9), 6.94 (1H, td, J 7.5, 1.0), 7.23 (1H, td, J 7.6, 0.9), 7.30 (1H, dd, J 7.5, 0.8); ^{13}C NMR ($CDCl_3$) δ 10.8, 14.1, 22.5, 22.6, 25.5, 27.1, 32.1, 39.5, 55.4, 55.5, 58.8, 110.2, 120.7, 125.9, 128.3, 129.5, 157.3, 162.9; m/z (EI^+) 305 (10%, MH^+), 304 (41%, M^+), 233 (56%, $M^+ - n$ -pen); HRMS $C_{18}H_{28}N_2O_2$ calcd. 304.2151, found 304.2156

20g. Prepared using general procedure above, crude **19g** (5.00 mmol, *anti/syn* 50/50) gave after purification by flash column chromatography (2:1 hexane:EtOAc) diastereomerically pure: (**4R***, **5S***)-*cis*-**20g** as a white solid (358 mg, 26%). Further recrystallisation (EtOAc/petroleum) afforded colourless needles suitable for single crystal X-ray structure determination; mp 155.0 – 157.0 °C; IR $\nu_{\max}$ (solid) 1683, 1467, 1241, 1030, 760 cm^{-1} ; 1H NMR ($CDCl_3$) δ 1.01 (3H, t, J 7.0), 1.08 (6H, dd, J 6.9, 2.9), 1.67 (2H, pent, J 7.2), 2.10 (1H, m), 3.48 (1H, dd, J 8.0, 3.0), 3.67 (1H, q, J 6.8), 3.89 (3H, s), 4.35 (1H, d, J 16.0), 4.87 (1H, br-s), 4.90 (1H, d, J 16.0), 6.93 (1H, d, J 8.2), 7.00 (1H, t, J 7.6), 7.33 (2H, m); ^{13}C NMR ($CDCl_3$) δ 11.6, 17.6, 21.2, 23.1, 28.2, 40.9, 55.3, 57.5, 62.4, 110.3, 120.6, 126.0, 128.2, 128.9, 157.3, 163.3; m/z (EI^+) 276 (10%, M^+), 234 (14%, $MH^+ - iPr$), 233 (58%, $M^+ - iPr$);

HRMS $C_{16}H_{24}N_2O_2$ calcd. 276.18378, found 276.18312. Anal. calcd. for $C_{16}H_{24}N_2O_2$: C, 69.52; H, 8.76; N, 10.14; found C, 69.48; H, 8.70; N, 10.01.

(4S*, 5S*)-trans-20g as a white solid (344 mg, 25%) mp 126.0-128.0 °C; IR $\nu_{\max}$ (solid) 1683, 1460, 1240, 1032, 761 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.77 (9H, m), 1.29 (1H, m), 1.99 (2H, dqd, J 10.4, 6.9, 3.4), 2.96 (1H, t, J 3.6), 3.18 (1H, br.qd, J 5.7), 3.74 (3H, s), 4.05 (1H, d, J 15.6), 4.69 (2H, m), 6.78 (1H, d, J 8.0), 6.86 (1H, t, J 7.4), 7.17 (2H, m); ^{13}C NMR ($CDCl_3$) δ 9.4, 15.2, 17.8, 28.1, 30.6, 38.9, 51.3, 55.4, 64.6, 110.3, 120.7, 125.6, 128.5, 129.6, 157.6, 161.3; m/z (EI^+) 276 (38%, M^+), 234 (37%, $MH^+ - ^iPr$), 233 (70%, $M^+ - ^iPr$); HRMS $C_{16}H_{24}N_2O_2$ calcd. 276.18378, found 276.18325.

N-(2-methoxybenzyl)-iso-propylamine. By product (76%) from reduction of **19h**. IR $\nu_{\max}$ (film) 3417, 2964, 1601, 1241, 1030, 752 cm^{-1} ; 1H NMR ($CDCl_3$) δ 1.16 (6H, d, J 6.2), 1.85 (1H), 2.87 (1H, hept, J 6.2), 3.86-3.96 (5H, m), 6.93 (1H, d, J 7.9), 6.99 (1H, t, J 7.6), 7.29-7.35 (2H, m); ^{13}C NMR ($CDCl_3$) δ 23.0, 47.0, 47.7, 55.4, 110.3, 120.5, 128.2, 128.8, 129.9, 157.8; m/z (EI^+) 179 (13%, M^+), 178 (10% $M^+ - H$), 164 (53%, $M^+ - Me$), 121 (100%); HRMS $C_{11}H_{17}NO$ calcd. 179.1310, found 179.1317.

¹ Arrowsmith, J.E.; Cook, M.J. *J. Chem. Soc. Perkin Trans. I* **1979**, 2364.

² Shindo, M. Koga, K.; Tomioka, K. *Tetrahedron* **1994**, 50, 4429.

³ Sturgess, M.A.; Yarberry, D.J. *Tetrahedron Lett.* **1993**, 34, 4743-6.

Current Data Parameters

NAME	EXPNO	PROCNO
GPH304	1	1

F2 - Acquisition Parameters

Date_	Time	INSTRUM	PROBHD	PULPROG	TD	SOLVENT	NS	DS	SWH	FIDRES	AQ	RG	DW	DE	TE	D1
20021113	11.33	av400	5 mm BBO BB-1H	zg30	32768	CSD6	8	2	4189.272 Hz	0.146157 Hz	3.4210291 sec	25.4	104.400 usec	5.00 usec	300.0 K	1.00000000 sec

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

NUC1	P1	PL1	SFO1
¹ H	7.50 usec	4.00 dB	400.1322007 MHz

F2 - Processing parameters

SI	SF	WDW	SSB	LB	GB	PC
32768	400.1300439 MHz	no	0	0.00 Hz	0	1.00

1D NMR plot parameters

CX	CY	FLP	F1	F2	PEPCM	HZCM
30.00 cm	19.00 cm	9.456 ppm	3783.60 Hz	-0.253 ppm	-101.23 Hz	0.32364 ppm/cm
						129.49617 Hz/cm

ppm

161.660
157.467
137.271
133.939
130.884
130.581
129.975
129.628
128.928
128.711
128.629
128.304
128.192
128.063
127.822
127.095
120.921
110.374

62.036
59.612
54.906

Current Data Parameters
NAME: GPH304
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters

Date_: 20021113
Time: 11.52
INSTRUM: av400
PROBHD: 5 mm BBO BB-1H
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 384
DS: 2
SWH: 25125.629 Hz
FIDRES: 0.76673 Hz
AQ: 0.652132 sec
RG: 1024.3
DE: 19.900 usec
TE: 300.0 K
D1: 1.00000000 sec
d11: 0.03000000 sec
d12: 0.00020000 sec
NUC1: 13C
P1: 13.00 usec
PL1: -0.10 dB
SFO1: 100.6273939 MHz
CPCPGC2: waltz16
NUC2: 1H
PCPD2: 90.00 usec
PL2: 4.00 dB
PL12: 25.58 dB
PL13: 29.00 dB
SFO2: 400.131605 MHz

F2 - Processing parameters

SI: 32768
SF: 100.6127456 MHz
WDW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters

CA: 30.00 cm
CY: 19.00 cm
F1P: 245.000 ppm
F1: 24650.12 Hz
F2P: -20.000 ppm
F2: -2012.25 Hz
PPMKM: 8.83333 ppm/cm
HZCM: 888.74591 Hz/cm

Current Data Parameters
NAME GRH563
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031003
Time 13.59
INSTRUM spect
PROBHD 5 mm BBO BP-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 0.00 dB
SFO1 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300467 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
FIP 10.508 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PWCN 0.3667 ppm/cm
HZCM 146.71434 Hz/cm

Current Data Parameters
NAME: GPH563
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20031003
Time: 14.16
INSTRUM: av400
PROBHD: 5 mm BBO BB-1H
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 320
DS: 2
SWH: 25125.629 Hz
FIDRES: 0.766773 Hz
AQ: 0.6521312 sec
RG: 11585.2
DW: 19.900 usec
DE: 20.00 usec
TE: 300.0 K
D1: 1.00000000 sec
d11: 0.03000000 sec
d12: 0.00020000 sec
NUC1: 13C
P1: 7.55 usec
PL1: 6.00 dB
SE01: 100.6231959 MHz
CDEPRG2: waltz16
NUC2: 1H
PCPD2: 94.00 usec
P12: 0.00 dB
P13: 22.00 dB
P14: 26.00 dB
SF02: 400.1316005 MHz

F2 - Processing parameters
SI: 32768
SF: 100.6127448 MHz
WDW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CA: 30.00 cm
CY: 44.86 cm
F1P: 245.000 ppm
F1: 24650.12 Hz
F2P: -20.000 ppm
F2: -2012.25 Hz
PPMCM: 8.8333 ppm/cm
HZCM: 888.74591 Hz/cm

Current Data Parameters

NAME	gph339
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030127
Time	13.26
INSTRUM	DRX500
PROBHD	5 mm MUltiNUC
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.18299 Hz
AQ	2.7329011 sec
RG	35.9
DW	83.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	13.30 usec
PL1	-3.00 dB
SFO1	500.1327507 MHz

F2 - Processing parameters

SI	16384
SF	500.1300535 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
FLP	9.000 ppm
F1	4501.17 Hz
F2p	-0.200 ppm
F2	-100.03 Hz
PPMCM	0.30667 ppm/cm
HZCM	153.37321 Hz/cm

Current Data Parameters
NAME gpm339
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030127
Time 13:49
INSTRUM DEX300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.465356 sec
RG 3649.1
RW 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
F1 9.50 usec
P1 5.00 dB
SE01 125.7715724 MHz
CDEPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SF02 500.1327507 MHz

F2 - Processing parameters
SI 16384
SF 125.7577609 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 ppm
F1 30010.65 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
PPMCM 8.8333 ppm/cm
HZCM 1110.86023 Hz/cm

Current Data Parameters
NAME GRH351
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030214
Time 14:06
INSTRUM DRAGON
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12531.328 Hz
FIDRES 0.191213 Hz
AQ 2.6193564 sec
RG 90.5
DM 39.900 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -3.00 dB
SFO1 500.135014 MHz

F2 - Processing parameters
SI 16384
SF 500.1300580 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1p 9.000 ppm
F1 4501.17 Hz
F2p -0.200 ppm
F2 -100.03 Hz
PPMCK 0.30667 ppm/cm
HZCK 153.37321 Hz/cm

Current Data Parameters
NAME GPR351
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030214
Time 14.38
INSTRUM DRX500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 704
DS 2
SWH 35211.270 Hz
FIDRES 1.074623 Hz
AQ 0.463356 sec
RG 7298.2
DM 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 9.30 usec
PL1 9.00 dB
SFO1 125.771574 MHz
CPEPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 500.1327507 MHz

F2 - Processing parameters
SI 16384
SF 125.757609 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 ppm
F1 30810.65 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
PPMCM 8.8333 ppm/cm
HZCM 1110.86023 Hz/cm

GPH362-imine

Current Data Parameters
 NAME GPH362
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030220
 Time 14.07
 INSTRUM DEK500
 PROBRD 5 mm Multis
 PULPROG zgpg30
 ID 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 12531.328 Hz
 FIDRES 0.191213 Hz
 AQ 2.6149364 sec
 RG 45.3
 DM 39.300 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.30 usec
 PL1 -3.00 dB
 SFO1 500.135014 MHz

F2 - Processing parameters
 SI 16384
 SF 500.1300510 MHz
 KW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CK 30.00 cm
 FIP 10.000 ppm
 F1 5001.30 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.34000 ppm/cm
 HCM 170.04422 Hz/cm

GPH362-imine

Current Data Parameters

NAME	GR339x
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030310
Time	10.43
INSTRUM	av400
PROBHD	5 mm BBI 1H-BB
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.14657 Hz
AQ	3.4210291 sec
RG	16
DM	104.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
PL	7.62 usec
PL1	3.00 dB
SFO1	400.1322067 MHz

F2 - Processing parameters

SI	16384
SE	400.1300405 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	13.88 cm
FIP	10.000 ppm
F1	4001.30 Hz
F2P	-0.200 ppm
F2	-80.03 Hz
PPMCM	0.34000 ppm/cm
HZCM	136.04420 Hz/cm

Current Data Parameters
NAME GPH38x
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030310
Time 10.55
INSTRUM 5 mm BB1 1H-BB
PROBHD zgpg30
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 10327.3
DW 19.900 usec
DE 20.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec
NUC1 13C
P1 12.88 usec
P11 1.00 dB
SFO1 100.6237959 MHz
CPOPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
P12 3.00 dB
PL12 25.00 dB
PL13 29.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127468 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CT 19.00 cm
F1P 2483.000 ppm
F1 24690.12 Hz
F2P -20.00 ppm
F2 -2012.25 Hz
PCKM 8.83333 ppm/cm
HZCM 888.74591 Hz/cm

Current Data Parameters

NAME	GRH529
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030731
Time	10.41
INSTRUM	DMX500
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	12531.328 Hz
FIDRES	0.191213 Hz
AQ	2.6149364 sec
RG	50.8
DM	39.900 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	13.30 usec
PL1	-3.00 dB
SFO1	500.135014 MHz

F2 - Processing parameters

SI	16384
SP	500.1300572 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

ID NMR plot parameters

CX	30.00 cm
FLP	8.500 ppm
F1	4251.10 Hz
F2	-0.280 ppm
F2	-100.03 Hz
EPHOC	0.23000 ppm/cm
HZCM	145.03712 Hz/cm

Current Data Parameters
NAME GPR529
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030731
Time 11.12
INSTRUM DUK300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 704
DS 2
SWH 35211.270 Hz
FIDRES 1.07453 Hz
AQ 0.463356 sec
RG 3649.1
DE 14.200 usec
TE 300.0 K
D1 1.0000000 sec
d11 0.0300000 sec
d12 0.0002000 sec
NUC1 13C
P1 9.50 usec
PL1 9.00 dB
SFO1 125.7715724 MHz
CPCPGC2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 500.1327507 MHz

F2 - Processing parameters
SI 16384
SF 125.757566 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 PPM
F1 30810.65 Hz
F2P -20.000 PPM
F2 -2515.16 Hz
PPMCM 8.8333 PPM/cm
HZCM 1110.86023 Hz/cm

Current Data Parameters

NAME	gary1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020927
Time	13.37
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	4
DS	2
SWH	4789.272 Hz
FIDRES	0.146197 Hz
AQ	3.4210291 sec
RG	4.0
DW	104.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	7.56 usec
PL1	4.00 dB
SFO1	400.132007 MHz

F2 - Processing parameters

SI	32768
SF	400.130039 MHz
WDW	NO
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	19.00 cm
FLP	10.000 ppm
F1	400.30 Hz
F2P	-9.200 ppm
F2	-80.003 Hz
PPMCM	0.34000 ppm/cm
HZCM	136.04420 Hz/cm

Current Data Parameters

NAME	EXPNO	PROCNO
9ph272	1	1

F2 - Acquisition Parameters

Date_	Time
20020927	13:27

1D NMR parameters

Parameter	Value
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	394
DS	2
SWH	25175.629 Hz
FIDRES	0.166773 Hz
AQ	0.6521352 sec
RG	13004
DW	19.900 usec
DE	20.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.0002000 sec
NUC1	¹³ C
F1	8.50 usec
P11	-0.10 dB
SFO1	100.6237959 MHz
CPDPRG2	waltz16
NUC2	¹ H
PCPD2	90.00 usec
PL2	2.50 dB
PL12	25.50 dB
PL13	29.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

Parameter	Value
SI	32768
SF	100.6127425 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

Parameter	Value
CX	30.00 cm
CY	19.00 cm
F1P	245.000 ppm
F1	24650.12 Hz
F2P	-20.000 ppm
F2	-2012.25 Hz
PPMKM	8.8333 ppm/cm
HZCM	888.74585 Hz/cm

Current Data Parameters

NAME	GPH314
EXNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030305
Time	14.38
INSTRUM	DRX500
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	12531.328 Hz
FIDRES	0.191213 Hz
AQ	2.6149364 sec
RG	16
RG	16
DW	39.900 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	¹ H
PL1	13.30 usec
PL1	-2.00 dB
SFO1	500.135014 MHz

F2 - Processing parameters

SF	500.130026 MHz
SI	16384
SE	no
MSB	0
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
FLP	10.000 ppm
F1	5001.30 Hz
F2P	-0.200 ppm
F2	-100.03 Hz
PPMCM	0.3400 ppm/cm
HZCM	170.04422 Hz/cm

Current Data Parameters
 NAME: GPH374
 EXPNO: 2
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20030305
 Time: 14.56
 INSTRUM: DRX500
 PROBHD: 5 mm MUltiNu
 PULPROG: zgpg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 256
 DS: 2
 SWH: 35211.210 Hz
 FIDRES: 1.074563 Hz
 AQ: 0.465356 sec
 RG: 36492.1
 RW: 14.200 usec
 DE: 5.50 usec
 TE: 300.0 K
 D1: 1.00000000 sec
 d11: 0.03000000 sec
 d12: 0.00002000 sec
 NUC1: 13C
 P1: 9.50 usec
 PL1: 5.00 dB
 SFO1: 125.715124 MHz
 CPOPRG2: waltz16
 NUC2: 1H
 PCPD2: 115.00 usec
 PL2: -3.00 dB
 PL12: 16.00 dB
 PL13: 16.00 dB
 SFO2: 500.1327507 MHz

F2 - Processing parameters
 SI: 16384
 SF: 125.7577566 MHz
 WDW: EM
 SSB: 0
 LB: 3.00 Hz
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 30.00 cm
 F1P: 245.000 ppm
 F1: 30810.65 Hz
 F2P: -20.000 ppm
 F2: -2515.16 Hz
 PEWCM: 8.83333 ppm/cm
 HZCM: 1110.86023 Hz/cm

Current Data Parameters
NAME GPR398
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030317
Time 16.00
INSTRUM DRX500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 448
DS 2
SWH 35211.270 Hz
FIDRES 1.074565 Hz
AQ 0.465356 sec
RG 3649.1
DW 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 9.50 usec
PL1 5.00 dB
SF01 125.7715724 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PE12 16.00 dB
PL13 16.00 dB
SF02 500.1327507 MHz

F2 - Processing parameters
SI 16384
SE 125.771568 MHz
FDM EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 ppm
F1 30810.65 Hz
F2P -20.000 ppm
F2 -2515.15 Hz
PFGMH 8.83333 ppm/cm
HZCM 1110.86023 Hz/cm

Current Data Parameters
NAME: GPH564
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters

Date_: 20031002
Time: 14.20
INSTRUM: av400
PROBHD: 5 mm BBO BB-1H
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 192
DS: 2
SWH: 25125.629 Hz
FIDRES: 0.766773 Hz
AQ: 0.6521332 sec
RG: 11585.2
DM: 19.900 usec
DE: 20.00 usec
TE: 300.0 K
D1: 1.00000000 sec
d11: 0.03000000 sec
d12: 0.00002000 sec
NUC1: 13C
F1: 125 usec
P1: 6.00 dB
SF01: 100.6237959 MHz
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 94.00 usec
PL2: 0.00 dB
PL12: 22.00 dB
PL13: 26.00 dB
SF02: 400.1316005 MHz

F2 - Processing parameters

SF: 32768
SF: 100.6127290 MHz
WDW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CX: 30.00 cm
CY: 90.65 cm
F1P: 245.000 ppm
F1: 24650.12 Hz
F2P: -20.000 ppm
F2: -2012.25 Hz
PPMCK: 8.8333 ppm/cm
HZCM: 888.74579 Hz/cm

Current Data Parameters
NAME: GP4413
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20030328
Time: 14.43
INSTRUM: DRX300
PROBHD: 5 mm Multinu
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 0
SWH: 12531.328 Hz
FIDRES: 0.191213 Hz
AQ: 2.619394 sec
RG: 45.3
DM: 39.900 usec
DE: 4.50 usec
TE: 300.0 K
D1: 1.00000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 13.30 usec
PL1: -3.00 dB
SFO1: 500.1355014 MHz

F2 - Processing parameters
SI: 16384
SF: 500.1300516 MHz
WDW: no
SSB: 0
TB: 0.00 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 30.00 cm
F1P: 10.000 ppm
F1: 5001.30 Hz
F2P: -0.200 ppm
F2: -100.03 Hz
PPMCK: 0.34000 ppm/cm
HZCM: 170.04422 Hz/cm

Current Data Parameters

NAME	VALUE
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
20030328	15.42
INSTRUM	DRX300
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	832
DS	2
SWH	35211.270 Hz
FIDRES	1.07463 Hz
AQ	0.463356 sec
RG	3649.1
DM	14.200 usec
DE	5.50 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00020000 sec
NUC1	¹³ C
P1	9.50 usec
PL1	0.00 dB
SFO1	125.7715724 MHz
CPDPRG2	waltz16
NUC2	¹ H
PCPD2	115.00 usec
PL2	-3.00 dB
PL12	16.00 dB
PL13	16.00 dB
SFO2	500.1327507 MHz

F2 - Processing parameters

SI	Value
SF	125.77157588 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

Parameter	Value
CX	30.00 cm
FI1P	245.000 ppm
F1	30910.65 Hz
F2P	-20.000 ppm
F2	-2515.16 Hz
PEMCM	8.83333 ppm/cm
HZCM	110.86023 Hz/cm

Current Data Parameters
 NAME: 19c
 EXPNO: 1
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_: 20030405
 Time: 13.06
 INSTRUM: av400
 PROBHD: 5 mm BBO BB-1H
 PULPROG: zg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 16
 DS: 2
 SWH: 4789.272 Hz
 FIDRES: 0.146157 Hz
 AQ: 3.4210291 sec
 RG: 90.5
 DW: 104.400 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1: 1H
 P1: 7.50 usec
 PL1: -1.00 dB
 SF01: 400.1322007 MHz
 F2 - Processing parameters
 SI: 16384
 SF: 400.1300398 MHz
 KW: 0
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 1.00
 ID NMR plot parameters
 CX: 30.00 cm
 CY: 14.91 cm
 PIP: 10.000 ppm
 FI: 4001.30 Hz
 F2P: -0.208 ppm
 F2: -80.03 Hz
 PPMCM: 0.34600 ppm/cm
 HZCM: 136.04420 Hz/cm

Current Data Parameters

NAME	VALUE
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Parameter	Value
Date_	20030405
Time	13.33
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	576
DS	2
SWH	25175.629 Hz
FIDRES	0.166773 Hz
AQ	0.6321332 sec
RG	10321.3
DM	19.900 usec
DE	20.00 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.03000000 sec
d13	0.00002000 sec
NUC1	¹³ C
PC1	7.47 usec
PL1	6.00 dB
SFO1	100.6237959 MHz
CPDPRG2	waltz16
NUC2	¹ H
PCPD2	98.00 usec
PL2	-1.00 dB
PL12	21.50 dB
PL13	26.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

Parameter	Value
SI	32768
SF	100.6127440 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

Parameter	Value
CX	30.00 cm
CY	121.17 cm
FLP	245.000 ppm
F1	24650.12 Hz
F2p	-20.000 ppm
F2	-2012.25 Hz
PRCKM	8.8333 ppm/cm
HZCM	888.74591 Hz/cm

Current Data Parameters

NAME	VALUE
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
20030423	13.31

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

NAME	VALUE
NUC1	¹ H
P1	13.30 usec
PL1	-3.00 dB
SFO1	500.135014 MHz

F2 - Processing parameters

NAME	VALUE
SI	16384
SE	500.1300523 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

NAME	VALUE
CX	30.00 cm
FLP	10.000 ppm
F1	5001.30 Hz
F2P	-0.200 ppm
F2	-100.03 Hz
PPMCK	0.34000 ppm/cm
HZCK	170.04422 Hz/cm

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

NAME	VALUE
DS	0
SWH	12531.328 Hz
FIDRES	0.191213 Hz
AQ	2.6149364 sec
RG	45.3
DW	39.900 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

Current Data Parameters
NAME GPR430
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030423
Time 14.15
INSTRUM DRX300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 960
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.465356 sec
RG 3649.1
DM 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d12 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 9.50 usec
PL1 5.00 dB
SFO1 125.7715724 MHz
CPCPRG2 waltz16
NUC2 1H
PCPR2 115.00 usec
PL2 -3.00 dB
P12 16.00 dB
P13 16.00 dB
SFO2 500.1327507 MHz

F2 - Processing parameters
SI 16384
SF 125.7577588 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 ppm
F1 30810.65 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
PWCW 8.83333 ppm/cm
H2OM 1110.86023 Hz/cm

Current Data Parameters

NAME	VALUE
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
20030802	14.46
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	40.3
DW	104.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
F1	7.30 usec
PL1	0.00 dB
SFO1	400.1322007 MHz

F2 - Processing parameters

SI	16384
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY <th>19.00 cm</th>	19.00 cm
FLP <th>10.500 ppm</th>	10.500 ppm
F1 <th>4201.37 Hz</th>	4201.37 Hz
F2 <th>-0.500 ppm</th>	-0.500 ppm
FREQM <th>0.36667 ppm/cm</th>	0.36667 ppm/cm
HZCM <th>146.71454 Hz/cm</th>	146.71454 Hz/cm

Current Data Parameters
NAME GPR31
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080802
Time 13.13

INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 576
DS 2
SWH 25175.629 Hz
FIDRES 0.166773 Hz
AQ 0.6521352 sec
RG 16384
RG 19.900 usec
DE 20.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec
NUC1 13C
F1 7.55 usec
P1 6.00 dB
SFO1 100.6257959 MHz
CPCPG2 waitz16
NUC2 1H
PCPD2 94.00 usec
P12 0.00 dB
P13 22.00 dB
P14 26.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127631 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 245.000 ppm
F1 24650.13 Hz
F2P -20.000 ppm
F2 -2012.26 Hz
PPMPPM 8.83333 ppm/cm
HZCM 888.74609 Hz/cm

Current Data Parameters
 NAME: GPH273
 EXPNO: 1
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_: 20020930
 Time: 13.46
 INSTRUM: av400
 PROBHD: 5 mm BBO BB-1H
 PULPROG: zg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 16
 DS: 2
 SWH: 4789.272 Hz
 FIDRES: 0.146157 Hz
 AQ: 3.4210291 sec
 RG: 256
 DW: 104.400 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1: 1H
 P1: 7.50 usec
 PL1: 4.00 dB
 SFO1: 400.1322007 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 400.1300402 MHz
 WDW: no
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 1.00
 ID NMR plot parameters
 CX: 30.00 cm
 CY: 19.00 cm
 P1P: 8.000 ppm
 F1: 3201.04 Hz
 F2P: -0.200 ppm
 FZ: -80.03 Hz
 PPMCM: 0.27333 ppm/cm
 HZCM: 109.36887 Hz/cm

ppm

Current Data Parameters
NAME GPH273a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021002

Time 9.12
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 320
DS 2
SWH 25125.629 Hz
FIDRES 0.166773 Hz
AQ 0.6521332 sec
RG 16384
DW 19.900 usec
DE 20.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
F1 22.00 usec
PL1 -0.10 dB
SFO1 100.6237959 MHz
CPEPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 2.90 dB
PL12 25.50 dB
PL13 29.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127425 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 19.00 cm
F1P 245.000 ppm
F1 24650.12 Hz
F2P -20.000 ppm
F2 -2012.25 Hz
PCWCM 8.8333 ppm/cm
HZCM 888.74565 Hz/cm

GPH548-nitro-amine crude

Current Data Parameters
 NAME GPH548
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030912
 Time 8:52
 INSTRUM av400
 PROSED 5 mm BBO BB-1H
 PULPROG zg30
 ID 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 57
 DM 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.30 usec
 PL1 0.00 dB
 SFO1 400.1322007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300000 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 CY 15.69 cm
 F1P 9.500 ppm
 F1 3801.24 Hz
 F2P -0.100 ppm
 F2 -40.01 Hz
 PPMCK 0.32000 ppm/cm
 HZCM 128.04161 Hz/cm

GPH548-nitro-amine crude

Current Data Parameters

NAME	EXPNO	PROCNO
GPH548	2	1

F2 - Acquisition Parameters

Date_	Time
20030912	9.24

INSTRUM av400

PROBHD 5 mm BBO BB-1H

PULPROG zgpg30

TD 32768

SOLVENT CHCl3

NS 640

DS 2

SWH 25125.629 Hz

FIDRES 0.766773 Hz

AQ 0.6521332 sec

RG 16384

DM 19.960 usec

DE 20.00 usec

TE 300.0 K

D1 1.00000000 sec

d11 0.03000000 sec

d12 0.00020000 sec

NUC1 13C

P1 7.55 usec

PL1 6.00 dB

SFO1 100.6237959 MHz

CDPRG2 WALTZ16

NUC2 1H

PCPD2 94.00 usec

PL2 0.00 dB

PL12 22.00 dB

PL13 26.00 dB

SFO2 400.1316085 MHz

F2 - Processing parameters

SI	SE
32768	100.6127433 MHz

MDM EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX	CY	F1P	F1	F2P	F2	PPMCM	HZCM
30.00 cm	106.76 cm	245.000 ppm	24650.12 Hz	-20.000 ppm	-2012.25 Hz	8.83333 ppm/cm	888.74585 Hz/cm

Current Data Parameters
NAME GRH393
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030315
Time 12.25
INSTRUM DRX500
PROBHD 5 mm NUC131
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12531.328 Hz
FIDRES 0.191213 Hz
AQ 2.6149364 sec
RG 40.3
DM 39.900 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -3.00 dB
SFO1 500.135014 MHz

F2 - Processing parameters
SI 16384
SF 500.1300304 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.280 ppm
F2 -100.03 Hz
FREQ 0.34000 ppm/cm
HZCM 170.04420 Hz/cm

Current Data Parameters

NAME	GFH393
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030315
Time	12:57
INSTRUM	DRX500
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	32768
SOLVENT	DMSO
NS	704
DS	2
SWH	35211.270 Hz
FIDRES	1.074563 Hz
AQ	0.4653556 sec
RG	7296.2
DW	14.200 usec
DE	5.50 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00020000 sec
NUC1	¹³ C
P1	9.50 usec
PL1	5.00 dB
SFO1	125.7715724 MHz
CPDPRG2	waltz16
NUC2	¹ H
PCPD2	115.00 usec
PL2	-3.00 dB
PL12	16.00 dB
PL13	16.00 dB
SFO2	500.1321507 MHz

F2 - Processing parameters

SI	16384
SF	125.757877 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
F1P	245.000 ppm
F1	30610.66 Hz
F2P	-20.000 ppm
F2	-2515.16 Hz
PPMCM	8.63333 ppm/cm
HZCM	1110.86047 Hz/cm

Current Data Parameters
NAME GPR306
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021123
Time 19.57
INSTRUM DRX500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1152
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.465356 sec
RG 7298.2
DM 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 ¹³C
P1 9.50 usec
PL1 5.00 dB
SFO1 125.7715724 MHz

F2 - Processing parameters
SI 16384
SF 125.7577920 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 30.00 cm
FLP 245.000 ppm
F1 30810.66 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
FREQM 8.83335 ppm/cm
HZCM 1110.86047 Hz/cm

Current Data Parameters
NAME gpb347b
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030213
Time 13:58
INSTRUM DRX500
PROBHD 5 mm Maltinn
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 960
DS 2
SWH 35211.270 Hz
FIDRES 1.07453 Hz
AQ 0.4653536 sec
RG 7295.2
DM 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d12 0.03000000 sec
d13 0.0002000 sec
NUC1 13C
P1 9.50 usec
PL1 5.00 dB
SF01 125.7715724 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SF02 500.1327507 MHz

F2 - Processing parameters
SI 16384
SF 125.7717688 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 ppm
F1 30810.66 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
PPMCM 8.8333 ppm/cm
HZCM 1110.86047 Hz/cm

Current Data Parameters
NAME GPH364
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030326
Time 16.08
INSTRUM DVS500
PROBHD 5 mm MUltiNUC
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12531.328 Hz
FIDRES 0.191213 Hz
AQ 2.6149364 sec
RG 71.8
DM 39.900 usec
DE 4.50 usec
TE 306.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -3.00 dB
SFO1 500.1355014 MHz

F2 - Processing parameters
SI 16384
SF 500.1299859 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
FIP 10.000 ppm
F1 5001.39 Hz
F2P -0.200 ppm
F2 -100.63 Hz
PWCNM 0.34000 ppm/cm
HZCM 170.04419 Hz/cm

GPH364-urea

Current Data Parameters
NAME GPH364
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030326
Time 15.55
INSTRUM DRX500
PROBHD 5 mm Multino
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 384
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.465356 sec
RG 7298.2
DM 14.200 usec
DE 3.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec
NPO1 13C
P1 9.50 usec
PI1 5.00 dB
SFO1 125.7715724 MHz
CQDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 500.1327507 MHz

F2 - Processing Parameters

SI 16384
SF 125.7577898 MHz
WDW EX
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CK 30.00 cm
F1P 245.000 ppm
F1 30810.66 Hz
F2P 20.000 ppm
F2 -2515.16 Hz
PPMCM 8.83333 ppm/cm
HSCM 1110.86047 Hz/cm

Current Data Parameters

NAME	VALUE
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Parameter	Value
Date_	20030731
Time	16:24
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	D2O
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	724.1
DM	104.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

Parameter	Value
NUC1	¹ H
PL1	7.30 usec
PL1	0.00 dB
SE01	400.1322007 MHz

F2 - Processing parameters

Parameter	Value
SI	16384
SP	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

Parameter	Value
CX	30.00 cm
CY	17.94 cm
FLP	10.500 ppm
F1	4201.37 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.3667 ppm/cm
HZCM	146.71434 Hz/cm

Current Data Parameters
NAME GRH460
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030627
Time 12.46
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2112
DS 2
SWH 25125.629 Hz
FIDRES 0.766713 Hz
AQ 0.652132 sec
RG 16384
DM 19.906 usec
DE 20.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 7.47 usec
P11 6.00 dB
SFO1 100.623199 MHz
CPOPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 -1.00 dB
PL12 21.50 dB
PL13 26.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.617290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
C1 48.12 cm
F1P 245.000 ppm
F1 24650.12 Hz
F2P -20.000 ppm
F2 -2012.25 Hz
FPPCKM 8.83333 ppm/cm
HZCM 888.74519 Hz/cm

Current Data Parameters

NAME	gms2ang
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030812
Time	8.57
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	18
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	80.6
DW	104.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	7.30 usec
PL1	0.00 dB
SFO1	400.1322007 MHz

F2 - Processing parameters

SI	16384
SE	400.1300000 MHz
WIM	no
SSB	0
GB	0.00 Hz
CB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	19.00 cm
X1P	10.500 ppm
F1	4201.37 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.3667 ppm/cm
HZCM	146.71434 Hz/cm

Current Data Parameters
NAME: GPH332
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20030122
Time: 13.39
INSTRUM: DEK500
PROBHD: 5 mm Multinu
PULPROG: zg30
TD: 32768
SOLVENT: CDCl3
NS: 16
DS: 0
SWH: 5995.204 Hz
FIDRES: 0.182859 Hz
AQ: 2.7329011 sec
RG: 71.8
DM: 83.400 usec
DE: 4.50 usec
TE: 300.0 K
D1: 1.00000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 13.30 usec
PL1: -3.00 dB
SFO1: 500.1327507 MHz

F2 - Processing parameters
SI: 16384
SF: 500.1299869 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 30.00 cm
F1P: 8.562 ppm
F1: 4282.33 Hz
F2P: -0.137 ppm
F2: -68.68 Hz
PRCKM: 0.28399 ppm/cm
HZCM: 145.03369 Hz/cm

Current Data Parameters
NAME: CPH332
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20030122
Time: 14.06
INSTRUM: DRX500
PROBHD: 5 mm Multinu
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 576
DS: 2
SWH: 35211.270 Hz
FIDRES: 1.074563 Hz
AQ: 0.465356 sec
RG: 7298.2
BW: 14.200 usec
DE: 5.50 usec
TE: 300.0 K
D1: 1.0000000 sec
d11: 0.0300000 sec
d12: 0.0002000 sec
NUC1: ¹³C
P1: 9.50 usec
PL1: 5.00 dB
SFO1: 125.7715724 MHz
CPDPRG2: waltz16
NUC2: ¹H
PCPD2: 115.00 usec
F22: 3.00 dB
PL2: 16.00 dB
PL13: 16.00 dB
SFO2: 500.1327507 MHz

F2 - Processing parameters
SI: 16384
SF: 125.757888 MHz
KOW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 30.00 cm
FIP: 245.000 ppm
F1: 30810.66 Hz
F2P: -20.000 ppm
F2: -2515.16 Hz
FREQM: 8.85335 ppm/cm
H2CM: 1110.86047 Hz/cm

Current Data Parameters

NAME	VALUE
NAME	GP1320
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
20021213	9.12

INSTRUM DRX500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 101.6
DM 83.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

NUC1	1H
P1	13.30 usec
PL1	-3.00 dB
SFO1	500.1327507 MHz

F2 - Processing parameters

SI	16384
SF	500.1300612 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CK	30.00 cm
F1P	8.000 ppm
F1	4001.04 Hz
F2P	-0.160 ppm
F2	-79.79 Hz
PPMCM	0.27198 ppm/cm
HZCM	136.02759 Hz/cm

Current Data Parameters

NAME	GFH320
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20021211
Time	14.51
INSTRUM	DRX300
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	832
DS	2
SWH	35211.270 Hz
FIDRES	1.074543 Hz
AQ	0.463356 sec
RG	1298.2
DM	14.200 usec
DE	5.50 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.03000000 sec
d12	0.00082000 sec
NUC1	¹³ C
P1	9.50 usec
P11	3.00 dB
SFO1	125.717174 MHz
CPDPRG2	waltz16
NUC2	¹ H
PCPD2	115.00 usec
P12	-3.00 dB
P12	16.00 dB
P13	16.00 dB
SFO2	500.1327507 MHz

F2 - Processing Parameters

SI	16384
SF	125.7577791 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
F1P	173.316 ppm
F1	21795.81 Hz
F2P	-10.709 ppm
F2	-1346.73 Hz
PCNCH	6.13416 ppm/cm
HZCM	771.41803 Hz/cm

GPH547-amine

Current Data Parameters

NAME	GPH547
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030915
Time	11.43
INSTRUM	DRX500
PROBHD	5 mm MSL100
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	12531.328 Hz
FIDRES	0.191213 Hz
AQ	2.6149364 sec
RG	161.3
DM	39.900 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
PI	13.30 usec
PT1	-3.00 dB
ST01	500.1355014 MHz

F2 - Processing parameters

SI	16384
SP	500.1299869 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
FLP	9.000 ppm
F1	4501.17 Hz
F2P	-0.200 ppm
F2	-100.03 Hz
PPMCM	0.30667 ppm/cm
HZCM	153.37318 Hz/cm

GP547-amine

Current Data Parameters
NAME GP547
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20080915
Time 12.09
INSTRUM DR2500
PROBHD 5 mm WALTIMU
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 576
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.4653556 sec
RG 7298.2
DM 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec
NUC1 13C
P1 9.50 usec
PL1 5.00 dB
SFO1 125.757794 MHz
CDEPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 500.1327507 MHz

F2 - Processing parameters

SI 16384
SF 125.757791 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 30.00 cm
F1P 245.000 ppm
F1 30810.66 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
PEWCM 8.83333 ppm/cm
HZCM 1110.86035 Hz/cm

GPH419-urea

163.503
152.237
142.767
142.510
110.486
108.058
106.708
77.408
77.156
76.901
60.652
58.652
55.927
54.422
31.892
29.825
28.382
24.352
10.690
9.844

Current Data Parameters
NAME GPH419
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030403
Time 13.45
INSTRUM DRX-500
PROBHD 5 mm Multin
ZPP930
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 768
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.463356 sec
RG 1298.2
DM 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 9.50 usec
PL1 5.00 dB
SFO1 125.7715724 MHz
CFOREQ2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 500.1327507 MHz

F2 - Processing Parameters
SI 16384
SF 125.757791 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 245.000 ppm
F1 3082.066 Hz
F2P -20.000 ppm
F2 -2515.16 Hz
PPWCM 8.83333 ppm/cm
HZCM 1110.86035 Hz/cm

Current Data Parameters
NAME GPI424
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030410
Time 14.45
INSTRUM DRX500
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12531.328 Hz
FIDRES 0.191213 Hz
AQ 2.6149364 sec
RG 181
DM 39.900 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -3.00 dB
SFO1 500.135014 MHz

F2 - Processing parameters
SI 16384
SF 500.1300259 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
FIP 10.000 ppm
F1 5001.30 Hz
F2P -0.200 ppm
F2 -100.03 Hz
PRCM 0.34000 ppm/cm
HZCM 110.04420 Hz/cm

Current Data Parameters
 NAME: CPH424
 EXPNO: 2
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20030408
 Time: 14.33
 INSTRUM: av400
 PROBHD: 5 mm BBO BB-1H
 PULPROG: zgpg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 384
 DS: 2
 SWH: 25125.623 Hz
 FIDRES: 0.166713 Hz
 AQ: 0.6521332 sec
 RG: 16384
 DW: 19.909 usec
 DE: 20.00 usec
 TE: 300.0 K
 D1: 1.00000000 sec
 d11: 0.03000000 sec
 d12: 0.00002000 sec
 NUC1: 13C
 P1: 7.47 usec
 PL1: 6.00 dB
 SFO1: 100.6231959 MHz
 CPOPRG2: waltz16
 NUC2: 1H
 PCEPD2: 98.00 usec
 PL2: -1.00 dB
 PL12: 21.50 dB
 PL13: 26.00 dB
 SFO2: 400.1316005 MHz

F2 - Processing parameters
 SI: 32768
 SF: 100.6127616 MHz
 FWH: EM
 SSB: 0
 LB: 3.00 Hz
 GB: 0
 PC: 1.40

1D NMR plot parameters
 CX: 30.00 cm
 CY: 19.00 cm
 F1P: 245.000 ppm
 F1: 24650.13 Hz
 F2P: -20.000 ppm
 F2: -2012.26 Hz
 PPMCM: 8.83333 ppm/cm
 HZCM: 888.74609 Hz/cm

Current Data Parameters
 NAME gar12ang
 EXNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030812
 Time 9.02
 INSTRUM av400
 PROBD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 362
 DW 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 7.30 usec
 PL1 0.00 dB
 SFO1 400.1322067 MHz

F2 - Processing parameters
 SI 16384
 SF 400.130050 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 30.00 cm
 CY 19.00 cm
 F1 34803 ppm
 F2 3922.50 Hz
 FZ 0.041 ppm
 E2 16.23 Hz
 PPMCM 0.32542 ppm/cm
 HZCM 130.20901 Hz/cm

164.487

77.478
77.160
76.841

57.777
56.187

31.822
29.613
26.076
22.686
22.456
14.004
10.846

Current Data Parameters
NAME GPH537
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030808
Time 13.13
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 128
DS 2
SWH 25175.629 Hz
FIDRES 0.166773 Hz
AQ 0.632132 sec
RG 16384
DW 19.900 usec
DE 20.00 usec
TE 300.0 K
D1 1.0000000 sec
d11 0.0300000 sec
d12 0.0002000 sec
NUC1 13C
F1 7.55 usec
P1 6.00 dB
SFO1 100.623199 MHz
CPCPRG2 waltz16
NUC2 1H
PCPD2 94.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 26.00 dB
SFO2 400.131605 MHz

F2 - Processing parameters
SI 32768
SF 100.6127654 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 11.88 cm
F1P 245.000 ppm
F1 24650.13 Hz
F2P -20.000 ppm
F2 -2012.26 Hz
PPMCM 8.8333 ppm/cm
HZCM 888.74609 Hz/cm

Current Data Parameters
NAME CPH528
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030729
Time 10.32
INSTRUM DRASCO
PROBHD 5 mm MSL100
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1152
DS 2
SWH 35211.270 Hz
FIDRES 1.074563 Hz
AQ 0.4653556 sec
RG 7298.2
DW 14.200 usec
DE 5.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 5.00 dB
SFO1 125.7715724 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 500.1327507 MHz

F2 - Processing parameters
SI 16384
SF 125.7578070 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1 245.000 ppm
F1 30810.66 Hz
F2 20.000 ppm
F2 -2313.18 Hz
FPCW 8.8333 ppm/cm
HZCM 1110.8606 Hz/cm

Stereoview for **10a**

Stereoview for **10c**

Stereoview for **10e**

Stereoview for **20g**