Supporting Information

Enantioselective Total Synthesis of the Securinega Alkaloid (-)-

Secu'amamine A

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General Methods. All non-aqueous reactions were carried out in oven- or flame-dried glassware under an argon atmosphere. All chemicals were purchased from commercial vendors and used as is, unless otherwise specified. Anhydrous tetrahydrofuran (THF) and dichloromethane (CH₂Cl₂) were obtained from a solvent purification system (Glass Contour). Reactions were magnetically stirred and monitored by thin layer chromatography (TLC) with 250 μ m EMD 60 F₂₅₄ precoated silica gel plates. Flash column chromatography was performed using EMD silica gel 60 (230-400 mesh). ¹H and ¹³C NMR spectral data were recorded on Bruker DPX-300, CDPX-300, or DRX 400 MHz spectrometers. Chemical shifts are reported relative to chloroform (δ 7.24), dichloromethane (δ 5.32), benzene (δ 7.15) for ¹H NMR and chloroform (δ 77.0), dichloromethane (δ 53.8), benzene (δ 128.0) for ¹³C NMR. Optical rotations were measured on a Rudoph Research Analytical Autopol II digital polarimeter.



Synthesis of N-Trityl Ester 33. To a solution of D-proline (15.00 g, 130.30 mmol) in methanol (150 mL) was slowly added thionyl chloride (19.0 mL, 260.5 mmol) at 0 °C. When the addition was complete, the ice bath was removed. The reaction mixture was stirred for an additional 12 h at rt before the solvent and other volatile compounds were removed in vacuo. The resulting oil was dissolved in CHCl₃ (200 mL), and Et₃N (55 mL, 394.6 mmol) was added followed by trityl chloride (34.50 g, 123.80 mmol). The reaction mixture was stirred for 12 h at rt and hydrolyzed with a 2:1 mixture of saturated aqueous NH₄Cl solution and NH₄OH (28% in water). The aqueous layer was extracted with CH₂Cl₂. The combined organic extracts were dried over MgSO₄ and concentrated. The solid obtained was dissolved in THF and filtered. The filtrate was concentrated under reduced pressure. After recrystallization of the residue from Et₂O, compound **33** (40.6 g, 84%) was obtained as a slightly yellow solid: $\left[\alpha\right]_{D}^{20} = +40.1$ (c 2.74, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.62-7.59 (m, 6H), 7.33-7.27 (m, 6H), 7.22-7.16 (m, 3H), 3.97 (dd, J = 8.9, 2.5 Hz, 1H), 3.73 (s, 3H), 3.50-3.46 (m, 1H), 2.92-2.89 (m, 1H), 1.70-1.50 (m, 2H), 1.14-0.95 (m, 2H); ¹³C NMR (75 MHz, CDCl3) δ 177.1, 144.7, 129.2, 127.6, 126.1, 77.4, 62.7, 51.5, 49.9, 31.2, 24.2; HRMS-ES (m/z): $[M + Na]^+$ calcd for C₂₅H₂₅NO₂Na, 394.1783; found, 394.1793.



Synthesis of N-Trityl Alcohol 34. A solution of ester 33 (39.00 g, 105.00 mmol) in dry THF (150 mL) was added slowly via an addition funnel to a slurry of LiAlH₄ (3.19 g, 84.00 mmol) in dry THF (50 mL) at 0 °C. After the addition was complete, the reaction mixture was allowed to warm to rt and stirred for 12 h. The reaction mixture was cooled to 0 °C, and then carefully quenched with water (3.2 mL), 15% aqueous NaOH (3.2 mL), and water (9.6 mL). After being stirred for 30 min, the mixture was diluted with EtOAc, filtered through Celite and the phases were separated. The aqueous phase was saturated with NaCl and extracted with EtOAc. The combined organic phases were dried over MgSO₄, and concentrated to yield the title compound as a white foam (34.26 g, 95%), which was used without further purification. For analytical purposes, alcohol 34 was purified by flash column chromatography on silica gel (hexanes/EtOAc/Et₃N, 9/1/0.1) to give a white foam: $[\alpha]_{D}^{20} = -37.7$ (c 2.17, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.65-7.62 (m, 6H), 7.34-7.28 (m, 6H), 7.25-7.22 (m, 3H), 3,70-3.66 (m, 1H), 3.62 (d, J = 7.1Hz, 1H), 3,57-3.54 (m, 1H), 3.27-3.24 (m, 1H), 3.10-3.04 (m, 1H), 2.40 (br s, 1H), 1.48-1.44 (m, 2H), 1.10-1.02 (m, 1H), 0.70-0.63 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 145.0, 129.5, 127.5, 126.1, 77.6, 65.7, 61.1, 50.9, 29.0, 24.1; HRMS-ES (*m/z*): [M + Na]⁺ calcd for C₂₄H₂₅NONa, 366.1834; found, 366.1824.



Synthesis of N-Trityl Aldehyde 11. To a solution of (COCl)₂ (13.0 mL, 148.5 mmol) in CH₂Cl₂ (120 mL) cooled at -78 °C was added dropwise a solution of DMSO (17.6 mL, 247.5 mmol) in CH₂Cl₂ (70 mL) via an addition funnel. After the addition was complete, the reaction was stirred for 30 min before a solution of the alcohol 34 (34.00 g, 99.00 mmol) in CH₂Cl₂ (90 mL) was added dropwise at -78 °C. After stirring the mixture for 1.5 h at this temperature, Et₃N (55.2 mL, 396.0 mmol) was added. The reaction mixture was further stirred for 1.5 h at -78 °C. and then guenched with a 2:1 mixture of a saturated aqueous NH₄Cl solution and NH₄OH (28% in water). The layers were separated, and the aqueous layer was extracted with CH₂Cl₂. The combined organic extracts were dried over MgSO₄ and concentrated under reduced pressure. The solid obtained was dissolved in THF and filtered. The filtrate was concentrated in vacuo to give, after recrystallization from ether, the title compound (30.40 g, 90%) as a slightly yellow solid: $\left[\alpha\right]_{D}^{20}$ = +12.5 (c 2.55, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 9.77 (d, *J* = 2.8 Hz, 1H), 7.50-7.47 (m, 6H), 7.21-7.16 (m, 6H), 7.12-7.07 (m, 3H), 3.71-3.65 (m, 1H), 3.24-3.16 (m, 1H), 2.88-2.80 (m, 1H), 1.55-1.47 (m, 1H), 1.38-1.30 (m, 1H), 1.07-1.00 (m, 1H), 0.74-0.69 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 204.4, 144.4, 129.4, 127.7, 126.4, 76.9, 50.5, 28.0, 24.2; HRMS-ES (m/z): $[M + Na]^+$ calcd for C₂₄H₂₃NONa, 364.1677; found, 364.1657.



Synthesis of Alcohol 13. Magnesium turnings (5.991 g, 246.540 mmol) were flame dried, cooled under argon and then immersed in THF (20 mL). Two drops of 1,2-dibromomethane were added to the mixture which was then heated at reflux for 15 min. The heating was stopped and a solution of vinyl bromide **12** (24.00 g, 61.63 mmol) in THF (50 mL) was added dropwise via an addition funnel to keep a gentle reflux. After the addition was complete, the reaction mixture was stirred further at reflux for 30 min and then cooled to rt.

The resulting Grignard reagent was added via cannula to a solution of the aldehyde **11** (14.028 g, 41.088 mmol) in THF (50 mL) at -78 °C. The reaction mixture was stirred for 4 h at -78 °C and then quenched by the addition of saturated aqueous NH₄Cl (5 mL). The solution was diluted with EtOAc and H₂O. The aqueous phase was saturated with NaCl and the organic phase was separated. The aqueous layer was further extracted with EtOAc. The combined organic phases were dried over MgSO₄ and concentrated *in vacuo*. Purification of the residue via flash chromatography on silica gel (hexanes/EtOAc/Et₃N, 9.5/0.5/0.1) afforded the desired alcohol **13** (25.449 g, 95%) as a white foam: $[\alpha]_{D}^{20} = +24.5$ (c 5.88, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.74 (d, *J* = 6.6 Hz, 4H), 7.64 (d, *J* = 7.8 Hz, 6H), 7.54-7.45 (m, 6H), 7.39-7.21 (m, 9H), 5.23 (s, 1H), 4.91 (s, 1H), 4.45 (s, 1H), 3.71-3.67 (m, 1H), 3.63 (t, *J* = 7.1 Hz, 2H), 3.36-3.27 (m, 2H), 3.14-3.07 (m, 1H), 2.21-2.11 (m, 1H), 2.02-1.95 (m, 1H), 1.70-1.59 (m, 1H), 1.43-1.35 (m, 1H), 1.14 (s, 9H), 1.08-1.01 (m, 1H), 0.39-0.26 (m, 1H); ¹³C NMR (75 MHz, CDCl₃)

δ 145.4, 144.5, 135.5, 135.4, 133.8, 129.7, 129.5, 127.5, 127.4, 126.2, 110.2, 78.0, 75.6, 62.6, 62.4, 52.9, 36.2, 26.8, 25.2, 24.8, 19.1; HRMS-ES (m/z): $[M + H]^+$ calcd for C₄₄H₅₀NO₂Si, 652.3611; found, 652.3618.



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Synthesis of MOM Ether 14. To a solution of the alcohol 13 (25.00 g, 38.35 mmol) in DCM (200 mL) at rt was added DIPEA (33.4 mL, 191.9 mmol) followed by MOMBr (12.5 mL, 153.4 mmol). The reaction mixture was then heated at 50 °C and stirred for 24 h. The reaction mixture was concentrated under reduced pressure and the residue obtained was dissolved in THF and filtered. The filtrate was then concentrated and the resulting oil was subjected to silica gel chromatography (hexanes/EtOAc/Et₃N, 8/2/0.1) to give the title compound (24.00 g, 90%) as a slightly yellow oil: $[\alpha]_D^{20} = +38.8$ (c 5.73 , CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.75-7.71 (m, 4H), 7.67 (d, J = 7.2 Hz, 6H), 7.52-7.47 (m, 6H), 7.30 (t, J = 7.6 Hz, 6H), 7.21 (t, J = 7.2 Hz, 3H), 5.08 (s, 1H), 4.90 (d, J = 6.5 Hz, 1H), 4.87 (s, 1H), 4.81 (d, J = 6.5 Hz, 1H), 4.77 (s, 1H), 3.72 (s, 3H), 3.64 (t, J = 7.0 Hz, 2H), 3.56 (d, J = 8.1 Hz, 1H), 3.49-3.36 (m, 1H), 3.13-3.05 (m, 1H), 1.97-1.90 (m, 1H), 1.83-1.72 (m, 1H), 1.71-1.64 (m, 1H), 1.47-1.38 (m, 1H), 1.13 (s, 9H), 0.88-0.77 (m, 1H), 0.34-0.22 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 145.35, 144.15, 135.54, 135.51, 133.95, 133.87, 129.66, 129.53, 127.57, 127.47, 126.00, 111.47, 94.95,

82.69, 78.42, 62.84, 62.10, 55.84, 51.83, 36.16, 26.77, 25.34, 24.13, 19.15; HRMS-ES (m/z): $[M + H]^+$ calcd for C₄₆H₅₄NO₃Si, 696.3873; found, 696.3914.



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Synthesis of Alcohol 35. To a solution of silvl ether 14 (23.00 g, 33.05 mmol) in THF (100 mL) was added TBAF (1 M solution in THF, 49.6 mL, 49.6 mmol) at rt. After 12 h at rt, the reaction mixture was diluted with saturated aqueous NH₄Cl (60.0 mL) and then EtOAc and H_2O . NaCl was added to saturate the aqueous phase and the organic phase was separated. The aqueous phase was extracted with EtOAc. The combined organic extracts were dried over MgSO₄ and concentrated in *vacuo*. The crude material was purified by silica gel flash chromatography ((hexanes/EtOAc/Et₃N, 9/1/0.1) to give the title compound (14.370 g, 95%) as a slightly yellow oil: $\left[\alpha\right]_{D}^{20} = +28.4$ (c 9.5, CHCl₃); ¹H NMR (300 MHz, CD₂Cl₂) δ 7.64-7.59 (m, 6H), 7.31-7.25 (m, 6H), 7.22-7.13 (m, 3H), 5.08 (t, J = 1.6 Hz, 1H), 4.90 (d, J = 0.8 Hz, 1H), 4.87 (d, J = 6.5 Hz, 1H), 4.73 (d, J =6.6 Hz, 1H), 4.68 (s, 1H), 3.61 (s, 3H), 3.50-3.46 (m, 3H), 3.42-3.32 (m, 1H), 3.06-2.98 (m, 1H), 1.99-1.89 (m, 1H), 1.68-1.58 (m, 3H), 1.40-1.29 (m, 1H), 0.77-0.69 (m, 1H), 0.32-0.22 (m, 1H); ¹³C NMR (75 MHz, CD₂Cl₂) δ 145.8, 145.2, 130.1, 127.9, 126.4, 112.5, 95.8, 83.0, 78.8, 63.2, 61.1, 56.1, 52.1, 37.2, 25.7, 24.4; HRMS-ES (*m/z*): $[M + H]^+$ calcd for C₃₀H₃₆NO₃, 458.2695; found, 458.2722.



Synthesis of Carbamate Alcohol 15. To a solution of alcohol 35 (14.15 g, 30.92 mmol) in MeOH (200 mL) was added AcOH (14.0 mL). After the mixture was stirred at rt for 12 h, TEA (40 mL) was added followed by (Boc)₂O (10.12 g, 46.38 mmol). The reaction mixture was stirred for an additional 6h at rt and the solvent was removed under reduced pressure. Purification of the residue via flash chromatography on silica gel (hexanes/EtOAc/Et₃N, 6/4/0.1) afforded the title compound (8.680 g, 89%) as a colorless oil: $[\alpha]_D^{20} = +125.2$ (c 6.15, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 5.16 (s, 0.5H), 5.07 (s, 0.5H), 5.00 (s, 1H), 4.51 (d, J = 6.7 Hz, 1H), 4.45 (d, J = 6.6 Hz, 1H), 4.32 (d, J = 5.0 Hz, 0.5 H), 3.94 (br s, 0.5 H), 3.82-3.70 (m, 3H), 3.45 (m, 0.5H), 3.34 (m, 0.5H), 3.29 (s, 1.5H), 2.07-1.93 (m, 2H), 1.71-1.66 (m, 2H), 1.45 (s, 4.5H), 1.38 (s, 4.5 H) ; ¹³C NMR (75 MHz, CDCl₃) δ 154.4, 153.9, 143.4, 143.2, 112.7, 112.2, 94.3, 94.2, 79.3, 79.0, 77.8, 77.3, 60.5, 60.1, 58.3, 58.2, 55.4, 55.2, 55.0, 46.9, 46.8, 36.0, 35.4, 28.2, 24.8, 24.7, 23.8, 23.5; HRMS-ES (*m*/z): $[M + H]^+$ calcd for C₁₆H₃₀NO₅, 316.2124; found, 316.2108.



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Synthesis of Aldehyde 16. To a solution of the carbamate alcohol 15 (6.200 g, 19.660 mmol) in CH₂Cl₂ (200 mL) was added NaHCO₃ (7.430 g, 88.470 mmol) followed

by Dess-Martin periodinane (12.50 g, 29.49 mmol). After 4 h, 10% $Na_2S_2O_3$ aqueous solution (200 mL) was added to the reaction mixture. After the solids dissolved, the mixture was extracted with CH₂Cl₂. The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo* to give a pale yellow oil. The aldehyde obtained was unstable and decomposed during chromatographic purification and therefore was used in crude form.



Synthesis of (*E*)-Enone 18. To a suspension of NaH (60% dispersion in mineral oil, 1.415 g, 35.390 mmol) in THF (15 mL) was slowly added a solution of the phosphonate 17 (9.364 g, 39.320 mmol) in THF (50 mL) at rt. After 45 min at rt, a solution of the crude aldehyde 16 (6.161 g, 19.660 mmol) in THF (40 mL) was added. The reaction mixture was stirred for 1.5 h and quenched with saturated aqueous NH₄Cl (5 mL). The resulting cloudy solution was diluted with EtOAc and H₂O. The aqueous phase was saturated with NaCl and the organic phase was separated. The aqueous phase was then extracted with EtOAc. The combined organic layers were dried over Na₂SO₄ and concentrated. The crude residue was chromatographed over silica gel to give the title compound (5.850 g, 70% based on carbamate alcohol 15) as a slightly yellow oil: $[\alpha]_{D}^{20} = +87.5$ (c 6.40, CHCl₃); ¹H NMR (400 MHz , CDCl₃) δ 6.93-6.71 (m, 1H), 6.17 (d, J = 15.9 Hz, 1H), 5.20 (d, J = 26.3 Hz, 1H), 4.95 (d, J = 19.2 Hz, 1H), 4.54-4.45 (m, 3H), 3.93 (br s, 0.5H), 3.79 (br s, 0.5H), 3.67 (s, 3H), 3.54 (br s, 0.5H), 3.39 (br s, 0.5H), 3.02 (m, 0.5H), 2.95-2.88 (m, 3.5H), 2.65-2.59 (m, 2H), 2.01-

1.95 (m, 2H), 1.76-1.69 (m, 2H), 1.48 (s, 4.5H), 1.45 (s, 4.5H); ¹³C NMR (75 MHz, CDCl₃) δ 197.96, 197.62g, 173.24, 154.44, 153.98, 144.57, 143.82, 143.75, 143.17, 131.43, 131.25, 113.64, 94.56, 94.30, 79.56, 78.99, 78.15, 77.72, 58.55, 58.45, 55.44, 55.36, 51.67, 47.01, 35.55, 35.27, 34.62, 34.40, 28.43, 27.69, 27.61, 25.18, 24.84, 24.16, 23.72; HRMS-ES (*m/z*): [M + H]⁺ calcd for C₂₂H₃₆NO₇, 426.2492; found, 426.2468.



Synthesis of Indolizidines 24 and 23. To a solution of the enone 18 (71.0 mg, 0.167 mmol) in CH₂Cl₂ (6 mL) was added TFA (0.26 mL, 3.34 mmol). After the reaction mixture was stirred for 6 h at rt, the solvent was removed under reduced pressure and the resulting residue was evacuated under high vacuum for 1 h. The residue was then dissolved in CH₂Cl₂ (10 mL) and cooled to -78 °C. DIPEA (0.29 mL, 1.67 mmol) was slowly added along the inner wall of the flask. After 4 h at this temperature, the mixture was warmed to rt and the solvent was removed *in vacuo*. Purification of the residue by silica gel chromatography (hexanes/EtOAc/Et₃N, 4/6/0.1) afforded the major conjugate addition product 24 (32.0 mg, 59 %) and the minor isomer 23 (6.0 mg, 11%).

Major isomer **24** (pale yellow oil): $[\alpha]_D^{20} = +86.9$ (c 3.50, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 5.02 (s, 1H), 4.80 (d, J = 1.6 Hz, 1H), 4.64 (dd, J = 17.9 Hz, 6.8 Hz, 2H), 3.71 (d, J = 8.9 Hz, 1H), 3.63 (s, 3H), 3.56 (dd, J = 12.4 Hz, 5.8 Hz, 1H), 3.37 (s, 3H), 2.85 (td, J = 8.8 Hz, 3.6 Hz, 1H), 2.71-2.66 (m, 2H), 2.55-2.45 (m, 6H), 2.39 (dd, J = 1.6 Hz, 1H), 2.55-2.45 (m, 6H), 2.39 (dd, J = 1.6 Hz, 1H), 2.55-2.45 (m, 6H), 2.39 (dd, J = 1.6 Hz, 1H), 3.67 (m, 2H), 2.55-2.45 (m, 6H), 2.39 (dd, J = 1.6 Hz, 1H), 2.55-2.45 (m, 6H), 2.39 (dd, J = 1.6 Hz, 1H), 2.55-2.45 (m, 6H), 2.39 (dd, J = 1.6 Hz, 1H), 3.67 (m, 2H), 2.55-2.45 (m, 6H), 2.59 (m, 2H), 2.55-2.45 (m, 2H

16.0 Hz, 7.2 Hz, 1H), 2.13 (dd, J = 13.3 Hz, 1.9 Hz, 1H), 2.02-1.93 (m, 1H), 1.85-1.76 (m, 1H), 1.72-1.56 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 208.3, 173.1, 143.5, 108.7, 95.7, 79.9, 61.6, 55.8, 51.7, 51.3, 49.1, 38.1, 37.6, 37.5, 28.9, 27.6, 21.3; HRMS-ES (*m/z*): $[M + H]^+$ calcd for C₁₇H₂₈NO₅, 326.1907; found, 326.1946.

Minor isomer **23** (pale yellow oil): ¹H NMR (400 MHz, CDCl₃) δ 4.91 (d, J = 1.6 Hz, 1H), 4.78 (d, J = 1.7 Hz, 1H), 4.66 (dd, J = 11.4 Hz, 6.7 Hz, 2H), 3.79 (d, J = 9.0 Hz, 1H), 3.64 (s, 3H), 3.37 (s, 3H), 3.02 (td, J = 8.3 Hz, 2.3 Hz, 1H), 2.82-2.69 (m, 3H), 2.64-2.53 (m, 3H), 2.48-2.35 (m, 2H), 2.09-1.90 (m, 4H), 1.79-1.60 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 207.3, 173.5, 145.9, 107.0, 96.4, 80.5, 69.7, 58.8, 56.2, 52.2, 51.7, 48.3, 40.6, 38.4, 29.3, 28.0, 21.4; HRMS-ES (m/z): $[M + H]^+$ calcd for C₁₇H₂₈NO₅, 326.1907; found, 326.1964.



Isomerization of 24 to 23. To a solution of the major conjugate addition product **24** (15.0 mg, 0.0461 mmol) in CH_2Cl_2 (5 mL) was added basic alumina (EMD Chemicals TLC/GL AL OX60 F254, 2.00 g). The heterogenous mixture was stirred for 5 min and the solvent was removed *in vacuo*. The remaining solid was evacuated under high vacuum. After 12 h at rt, the alumina was washed with 10% isopropanol in CHCl₃ and filtered. The elutant was concentrated and the residue was purified by silica gel

chromatography (hexanes/EtOAc/Et₃N, 4/6/0.1) to afford the minor conjugate addition product **23** (12.0 mg, 80%).



Synthesis of Diketo Ester 25. To a solution of the major indolizidine 24 (36.0 mg, 0.111 mmol) in THF (6 mL) and H₂O (2 mL) at 0 °C was added OsO₄ (4 wt% solution in water, 0.21 mL, 0.033 mmol). After the mixture was stirred for 15 min at this temperature, a solution of NaIO₄ (118.7 mg, 0.555 mmol) in H₂O (3 mL), and a solution of NMO (65.0 mg, 0.555 mmol) in H₂O (3 mL) were added. The reaction mixture was stirred for another 5 h at 0 °C. The mixture was then partitioned between EtOAc and H₂O and the aqueous phase was saturated with NaCl. The organic layer was separated and the aqueous layer was extracted with EtOAc. The combined organic phases were dried over Na₂SO₄ and concentrated. The residue was purified by silica gel chromatography (hexanes/EtOAc/Et₃N, 4/6/0.1) to give compound 25 (23.0 mg, 63%) as a colorless oil: $\left[\alpha\right]_{D}^{20}$ = +88.4 (c 1.90, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 4.71 (d, J = 7.0 Hz, 1H), 4.62 (d, J = 7.0 Hz, 1H), 3.98-3.89 (m, 1H), 3.95 (d, J = 9.4 Hz, 1H), 3.62 (s, 3H), 3.36 (s, 3H), 2.98-2.86 (m, 2H), 2.81 (dd, J = 13.4 Hz, 6.4 Hz, 1H), 2.74-2.61 (m, 3H), 2.60-2.48 (m, 3H), 2.39 (dd, J = 16.5 Hz, 8.7 Hz, 1H), 2.20 (dd, J = 13.3 Hz, 2.0 Hz, 1H), 2.13-2.04 (m, 1H), 1.96-1.87 (m, 1H), 1.85-1.73 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 207.6, 206.9, 172.9, 96.1, 81.0, 62.4, 55.9, 52.9, 51.8, 49.4, 43.5, 40.8, 38.0, 30.0, 27.5, 22.1; HRMS-ES (m/z): $[M + H]^+$ calcd for C₁₆H₂₆NO₆, 328.1760; found, 328.1779.



Synthesis of Tetracyclic γ -Lactone 26. To a solution of diketo ester 25 (95.0 mg, 0.290 mmol) in MeOH (20 mL) was added NaOMe (24.0 mg, 0.444 mmol). The solution was stirred for 12 h at rt and then quenched with saturated aqueous NH₄Cl (1.0 mL). The methanol solvent was removed under reduced pressure. The cloudy aqueous solution was diluted with H₂O and extracted with 15% isopropanol in CHCl₃. The combined organic phases were dried over Na₂SO₄ and concentrated. Purification of the residue by chromatography on silica gel (hexanes/EtOAc/Et₃N, 3/7/0.1) yielded compound **26** (64.0 mg, 75 %) as a colorless oil: $[\alpha]_{D}^{20} = +12.6$ (c 1.90, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 4.74 (d, *J* = 6.8 Hz, 1H), 4.61 (d, *J* = 6.8 Hz, 1H), 3.64 (d, *J* = 9.1 Hz, 1H), 3.58-3.53 (m, 1H), 3.47 (t, *J* = 10.6 Hz, 1H), 3.34 (s, 3H), 2.91-2.68 (m, 3H), 2.55 (d, *J* = 18.3 Hz, 1H), 2.48-2.33 (m, 3H), 2.26-2.19 (m, 1H), 2.11-2.03 (m, 1H), 1.93-1.84 (m, 1H), 1.81-1.67 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 207.4, 173.9, 97.4, 86.8, 79.9, 59.4, 56.1, 48.8, 48.7, 47.1, 36.6, 35.5, 29.8, 28.6, 21.5; HRMS-ES (*m*/*z*): $[M + H]^+$ calcd for C₁₅H₂₂NO₅, 296.1498; found, 296.1515.

By further elution of the silica gel column with EtOAc/isopropanol/Et₃N (9.5/0.5/0.1), hydroxy ester **28** (14.2 mg, 15%) was obtained as a pale yellow oil: ¹H NMR (400 MHz, CDCl₃) δ 4.73 (d, J = 5.9 Hz, 1H), 4.46 (d, J = 5.9 Hz, 1H), 4.34 (s, 1H), 3.67 (s, 3H), 3.45 (d, J = 2.8 Hz, 1H), 3.41 (s, 3H), 3.22 (d, J = 9.8 Hz, 1H), 3.15 (dd, J = 10.1 Hz, 2.7 Hz, 1H), 2.93 (dd, J = 16.8 Hz, 10.1 Hz, 1H), 2.82 (td, J = 8.1 Hz,

4.1 Hz, 1H), 2.78 (dt, J = 16.5 Hz, 2.0 Hz, 1H), 2.69 (dd, J = 16.8 Hz, 2.9 Hz, 1H), 2.67-2.61 (m, 1H), 2.36-2.27 (m, 2H), 2.19 (dd, J = 12.9 Hz, 2.7 Hz, 1H), 2.13 (dt, J = 12.1 Hz, 3.0 Hz 1H), 1.93-1.83 (m, 1H), 1.85-1.75 (m, 1H), 1.72-1.63 (m, 1H), 1.46-1.39 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 208.3, 174.2, 98.7, 89.6, 73.8, 59.4, 58.6, 56.1, 51.7, 50.7, 48.4, 41.0, 40.4, 29.4, 28.5, 21.3; HRMS-ES (m/z): $[M + H]^+$ calcd for C₁₆H₂₆NO₆, 328.1760; found, 328.1756.



Transformation of Hydroxyl Ester 28 to Lactone 26. To a solution of hydroxy ester **28** (6.0 mg, 0.0306 mmol) in MeOH (5 mL) was added NaOMe (20.0 mg, 0.370 mmol). After stirring the mixture for 36 h at rt, the reaction was quenched with saturated aqueous NH₄Cl (1 mL). The solvent was removed under reduced pressure and the aqueous solution was diluted with H₂O and extracted with 15% isopropanol in CHCl₃. The combined organic phases were dried over Na₂SO₄ and concentrated. Purification of the residue by chromatography on silica gel (hexanes/EtOAc/Et₃N, 3/7/0.1) yielded lactone **26** (4.9 mg, 90 %).



29

Synthesis of Enol Triflate 29. A solution of ketone 26 (95.0 mg, 0.322 mmol) in THF (10.0 mL) was added dropwise to a solution of KHMDS (0.5 M in toluene, 1.29 mL, 0.65 mmol) in THF (2 mL) at -78 °C. After 1 h at this temperature, a solution of Nphenyltriflimide (138.0 mg, 0.386 mmol) in THF (2 mL) was added slowly to the mixture. The reaction mixture was stirred at -78 °C for another 12 h and then guenched with saturated aqueous NH₄Cl (1 mL). The solution was diluted with EtOAc and H₂O and the aqueous phase was saturated with NaCl. The organic phase was then separated and the aqueous layer was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4 and concentrated. The residue was purified by silica gel column chromatography (hexanes/EtOAc/Et₃N, 6/4/0.1) to give enol triflate **29** (117.0 mg, 85%) as a colorless oil: $\left[\alpha\right]_{D}^{20} = -10.7$ (c 3.55, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 5.93 (d, J = 6.4 Hz, 1H), 4.74 (d, J = 6.8 Hz, 1H), 4.62 (d, J = 6.8 Hz, 1H), 3.79-3.75 (m, 1H), 3.64 (d, J = 9.5 Hz, 1H), 3.54 (t, J = 10.5 Hz, 1H), 3.36 (s, 3H), 2.91 (dd, J = 8.4 Hz, 2.7 Hz, 1H), 2.85 (dd, J= 17.8 Hz, 9.6 Hz, 1H), 2.63 (dd, J = 17.6 Hz, 11.9 Hz, 1H), 2.38 (dd, J = 16.5 Hz, 8.3 Hz, 1H), 2.32-2.24 (m, 1H), 2.08 (dd, J = 12.4 Hz, 3.1 Hz, 1H), 2.05-1.99 (m, 1H), 1.91-1.83 (m, 1H), 1.79 (dd, J = 12.3 Hz, 3.0 Hz, 1H), 1.77-1.68 (m, 1H), 1.67-1.58 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 172.9, 149.7, 124.8, 120.6, 116.3, 115.4, 112.1, 97.3, 85.9, 78.8, 58.8, 56.0, 49.5, 48.6, 37.2, 32.9, 33.8, 27.8, 21.8; HRMS-ES (m/z): $[M + H]^+$ calcd for C₁₆H₂₁NO₇SF₃, 428.0991; found, 428.0967.



Synthesis of Alkene 30. To a solution of enol triflate 29 (71.0 mg, 0.166 mmol), DIPEA (0.12 mL, 0.689 mmol), Pd(OAc)₂ (0.4 mg, 0.002 mmol) and PPh₃ (1.0 mg, 0.004 mmol) in DMF (1.5 mL) was added formic acid (22.9 mg, 0.498 mmol). The solution was stirred at 60 °C for 1 h. During this period, the mixture became black. After cooling, the mixture was diluted with EtOAc and washed with brine. The organic phase was dried over Na_2SO_4 and concentrated. The residue was purified by silica gel column chromatography (hexanes/EtOAc/Et₃N, 3/7/0.1) to give alkene **30** (44.1 mg, 95%) as a colorless oil: $[\alpha]_{D}^{20} = -104.4$ (c 0.90, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 5.99 (dd, J = 9.7 Hz, 3.7 Hz, 1H), 5.82-5.78 (m, 1H), 4.75 (d, J = 6.7 Hz, 1H), 4.60 (d, J = 6.7 Hz, 1H), 3.61 (d, J = 9.4 Hz, 1H), 3.57 (dd, J = 5.7 Hz, 2.9 Hz, 1H), 3.34 (s, 3H), 3.20-3.09 (m, 1H), 2.88 (td, J = 8.5 Hz, 3.0 Hz, 1H), 2.73 (dd, J = 17.4 Hz, 9.3 Hz, 1H), 2.42-2.21 (m, 3H), 2.01 (dd, J = 12.3 Hz, 2.8 Hz, 1H), 1.96-1.95 (m, 1H), 1.89-1.81 (m, 1H), 1.75 $(dd, J = 12.0 Hz, 3.0 Hz, 1H), 1.71-1.57 (m, 2H); {}^{13}C NMR (75 MHz, CDCl₃) \delta 175.0,$ 130.8, 124.4, 97.5, 85.7, 79.8, 58.8, 55.9, 50.8, 48.5, 34.9, 34.7, 33.7, 27.7, 21.8; HRMS-ES (m/z): $[M + H]^+$ calcd for C₁₆H₂₂NO₄, 280.1549; found, 280.1552.



Synthesis of Selenide 31. To a solution of the lactone 30 (21.0 mg, 0.0752 mmol) in THF (3 mL) at -78 °C was added LDA (2.0 M in heptane/THF/ethylbenzne, 0.15 mL, 0.30 mmol). After stirring the mixture for 1 h at this temperature, a solution of PhSeCl (28.8 mg, 0.150 mmol) in THF (2 mL) was slowly added. The reaction mixture was stirred at -78 °C for another 12 h and then guenched with saturated aqueous NH₄Cl (1.0 mL). The mixture was diluted with EtOAc and H₂O, and NaCl was then added to saturate the aqueous phase. The organic phase was separated and the aqueous phase was extracted with EtOAc. The combined organic extracts were dried over Na₂SO₄ and concentrated. The residue was subjected to silica gel column chromatography (hexanes/EtOAc/Et₃N, 3/7/0.1) to provide the selenide (28.0 mg, 86%) as a colorless oil: $\left[\alpha\right]_{D}^{20} = -127.1$ (c 1.40, CHCl₃); ¹H NMR (300 MHz, CDCl₃) & 7.72-7.68 (m, 2H), 7.38-7.26 (m, 3H), 6.06 (dd, J = 9.7 Hz, 3.7 Hz, 1H), 5.84 (ddd, J = 9.6 Hz, 5.7 Hz, 1.8 Hz, 1H), 4.48 (d, J = 6.8 Hz, 1H), 4.15 (d, J = 6.8 Hz, 1H), 3.63 (d, J = 12.1 Hz, 1H), 3.57 (dt, J = 6.0 Hz, 3.0 Hz, 1H), 3.50 (d, J = 9.5 Hz, 1H), 3.20 (s, 3H), 3.06 (ddd, J = 12.1 Hz, 3.7 Hz, 2.0 Hz, 1H), 2.86(td, J = 8.5 Hz, 3.2 Hz, 1H), 2.36 (dd, J = 16.4 Hz, 8.6 Hz, 1H), 2.16 (td, J = 8.8 Hz, 6.4Hz, 1H), 1.99 (dd, J = 12.1 Hz, 2.9 Hz, 1H), 1.94-1.88 (m, 1H), 1.82-1.76 (m, 1H), 1.68 (dd, J = 11.9 Hz, 3.0 Hz, 1H), 1.64-1.52 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 173.8, 136.7, 129.5, 129.2, 129.0, 125.9, 125.0, 97.0, 84.7, 79.5, 58.8, 55.7, 51.0, 48.5, 44.1,

41.3, 34.0, 27.7, 21.8; HRMS-ES (m/z): $[M + H]^+$ calcd for C₂₁H₂₆NO₄Se, 436.1027; found, 436.1019.



32

Synthesis of Diene Lactone 32. To a solution of selenide 31 (28.0 mg, 0.065 mmol) in MeOH (8 mL) and H₂O (4 mL) was added NaHCO₃ (11.0 mg, 0.131 mmol) followed by NaIO₄ (69.0 mg, 0.323 mmol) at rt. The reaction mixture was stirred for 1 h at this temperature and then methanol was removed in vacuo. The resulting cloudy solution was diluted with H₂O and EtOAc. The aqueous phase was saturated with NaCl and the organic phase was separated. The aqueous layer was subsequently extracted with EtOAc and 15% isopropanol in CHCl₃. The combined organic extracts were dried over Na₂SO₄ and the solvent was removed under reduced pressure. The crude material obtained was purified by silica gel column chromatography (hexanes/EtOAc/Et₃N, 3/7/0.1) to yield diene lactone **32** (15.0 mg, 84%) as a colorless oil: $\left[\alpha\right]_{\rm D}^{20} = -468.6$ (c 0.70, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 6.77 (d, J = 9.5 Hz, 1H), 6.14 (dd, J = 9.5Hz, 5.5 Hz, 1H), 5.83 (s, 1H), 4.75 (d, J = 6.9 Hz, 1H), 4.56 (d, J = 7.0 Hz, 1H), 3.88 (dd, J = 5.5 Hz, 2.8 Hz, 1H), 3.62 (d, J = 9.6 Hz, 1H), 3.33 (s, 3H), 2.98 (td, J = 8.6 Hz, 3.3 Hz, 1H), 2.54-2.43 (m, 2H), 2.33 (dd, J = 11.6 Hz, 2.8 Hz, 1H), 2.10-1.98 (m, 1H), 1.97 $(dd, J = 11.6 Hz, 3.2 Hz, 1H), 1.91-1.84 (m, 1H), 1.74-1.65 (m, 2H); {}^{13}C NMR (75 MHz, 1.65 MHz), 1.91-1.84 (m, 1H), 1.74-1.65 (m, 2H); {}^{13}C NMR (75 MHz), 1.91-1.84 (m, 1H), 1.74-1.65 (m, 2H); {}^{13}C NMR (75 MHz), 1.91-1.84 (m, 1H), 1.74-1.65 (m, 2H); {}^{13}C NMR (75 MHz), 1.91-1.84 (m, 1H), 1.74-1.65 (m, 2H); {}^{13}C NMR (75 MHz), 1.91-1.84 (m, 1H), 1.91-1.84 (m, 1H), 1.91-1.84 (m, 2H); {}^{13}C NMR (m, 2H); {}^{13}C NMR$ CDCl₃) δ 171.9, 162.0, 133.5, 124.3, 114.3, 98.1, 86.7, 80.4, 59.1, 56.2, 51.9, 48.5, 37.5, 28.2, 22.1; HRMS-ES (m/z): $[M + H]^+$ calcd for C₂₁H₂₀NO₄, 278.1392; found, 278.1385.



Synthesis of (-)-Secu'amamine A (5). To a solution of diene lactone 32 (14.0 mg, 0.050 mmol) in MeOH (6 mL) was added conc. HCl (0.60 mL). The reaction mixture was then stirred for 5 h at 60 °C. After cooling the mixture to rt, saturated aqueous NaHCO₃ (3 mL) was added and methanol was removed under reduced pressure. The resulting cloudy solution was then diluted with H₂O and 15% isopropanol in CHCl₃. The aqueous phase was saturated with NaCl, and the organic phase was separated. The aqueous layer was extracted with 15% isopropanol in CHCl₃. The combined organic layers were dried over Na₂SO₄ and concentrated. Purification of the residue via silica gel column chromatography (hexanes/EtOAc/Et₃N, 1/9/0.1) gave (-)-Secu'amamine A (5) (11.0 mg, 93 %) as a colorless oil: $[\alpha]_{D}^{20} = -511.3$ (c 0.15, CHCl₃); reported rotation: $\left[\alpha\right]_{D}^{20} = -479$ (c 0.15, CHCl₃); X-Ray quality crystals were prepared via slow evaporation from deuteriobenzene; ¹H NMR (400 MHz, CDCl₃) δ 6.76 (d, J = 9.6 Hz, 1H), 6.16 (dd, J = 9.5 Hz, 5.6 Hz, 1H), 5.83 (s, 1H), 3.90 (dt, J = 5.5 Hz, 2.8 Hz, 1H), 3.70 (d, J = 9.6Hz, 1H), 2.98 (td, J = 8.6 Hz, 3.7 Hz, 1H), 2.68 (br s, 1H), 2.60-2.47 (m, 2H), 2.34 (dd, J) = 11.6 Hz, 2.8 Hz, 1H), 2.09-2.03 (m, 1H), 1.98 (dd, J = 11.6 Hz, 3.2 Hz, 1H), 1.94-1.86 (m, 1H), 1.78-1.72 (m, 1H), 1.68-1.60 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 172.42, 162.08, 133.99, 124.34, 114.06, 87.05, 74.80, 59.53, 52.12, 48.60, 36.88, 28.24, 22.19; HRMS-ES (m/z): $[M + H]^+$ calcd for C₁₃H₁₆NO₃, 234.1130; found, 234.1150.

NMR Data of (-)-Secu'amamine A (5) in Deuteriobenzene: ¹H NMR (400 MHz, C₆D₆) δ 6.07 (d, J = 9.5 Hz, 1H), 5.54 (s, 1H), 5.43 (dd, J = 9.5 Hz, 5.6 Hz, 1H), 3.50 (d, J = 9.6 Hz, 1H), 3.18 (dt, J = 5.5 Hz, 3.0 Hz, 1H), 2.90 (br s, 1H), 2.53 (td, J = 8.3 Hz, 3.7 Hz, 1H), 2.38 (dt, J = 9.5 Hz, 7.0 Hz, 1H), 2.13 (td, J = 8.5 Hz, 7.1 Hz, 1H), 2.05 (dd, J = 11.4 Hz, 2.8 Hz, 1H), 1.96-1.88 (m, 1H), 1.65-1.54 (m, 1H), 1.52 (dd, J = 11.4 Hz, 3.9 Hz, 1H), 1.49-1.42 (m, 1H), 1.40-1.34 (m, 1H); ¹³C NMR (75 MHz, C₆D₆) δ 172.31, 161.95, 133.54, 124.15, 114.82, 87.03, 75.42, 60.10, 52.30, 48.82, 37.14, 28.91, 22.77.



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PL-Jan10-07 - 4 1 F2 - Acquisition Parameters 20080110 14.32 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 47 4 18796.992 Hz 0.286819 Hz 1.7433076 sec 1024 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 13C 5.40 usec -6.00 dB 75.4106357 MHz ======== CHANNEL f2 ========== waltz16 1H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz F2 - Processing parameters 32768 75.4023780 MHz ЕΜ 0 1.00 Hz 0 1.40 10 NMA plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42615 Hz/cm

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ũ đ d	145.377 144.477 135.467 135.467 135.440 123.758 123.758 123.758 127.773 127.128 127.128 127.128 127.128 126.181	$\begin{array}{c} 77.995 \\ 77.425 \\ 77.000 \\ 76.576 \\ 76.576 \\ 75.561 \\ 62.649 \\ 62.400 \\ 62.400 \\ 52.891 \end{array}$	Current D NAME EXPNO PROCNO F2 - Acqu Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES
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ppm 200 175			EX F1P F1 F2P F2 PPMCM HZCM

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> Data Parameters PL-Jan 15-07 3 1 Quisition Parameters 20080115 21,30 spect 5 mm GNP 1H/1 zgpg30 65536 COC13 62 4 18796,992 Hz 0.286819 Hz 1.7433076 sec 1024 26.600 usec 6.00 usec 300.0 K 2,00000000 sec 0.03000000 sec 0.00002000 Sec ==== CHANNEL {1 ============== 130 5.40 usec -6.0<u>0</u> dB 75.4106357 MHz ===== CHANNEL f2 ============== waltz16 18 115.00 usec 0.00 d8 20.00 dB 20.00 dB 299.8711995 MHz ocessing parameters 32768 75.4023918 MHz ΕM 0 1.00 Hz 0 1.40 lot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42627 Hz/cm

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Current Data Parameters PL-Ju124-07 2 F2 - Acquisition Parameters 20070724 15.13 spect 5 mm Multinu zg30 65536 CDC13 16 2 6172.839 Hz 0.094190 Hz 5.3084660 sec 71.B 81.000 usec 6.00 usec 300.0 K 1.00000000 sec ========= CHANNEL f1 ======== 1H 9.60 usec -6.00 dB 300.1318534 MHz Processing parameters 32768 300.1300000 MHz no 0 0.00 Hz 0 1.00 10 NMR plot parameters 20.00 cm 11.000 ррт 3301.43 Hz -1.000 ppm -300.13 Hz 0.50000 ppm/cm 180.07800 Hz/cm



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		NUC1 P1 PL1 SF01 CPDPRG2 NUC2 PCPD2 PL2 PL12 PL13 SF02
		F2 - Pro SI SF WDW SSB LB GB PC 10 NMR p CX
1 <u>1</u> 	 0 	F1 F2P F2P F2 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2

Current Data Parameters PL-Ju124-07 З quisition Parameters 20070724 15.20 spect 5 mm Multinu zgpg30 65536 CDC13 103 4 18832.393 Hz 0.287360 Hz 1.7400308 sec 16384 26.550 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 11.80 usec 0.00 dB 75.4760200 MHz ===== CHANNEL f2 ============== waltz16 1H 110.00 usec 0.00 dB 17.50 dB 17.50 dB 300.1312005 MHz ocessing parameters 32768 75.46775B3 MHz EМ 0 1.00 Hz 0 1.40 plot parameters 20.00 cm 219.454 ppm 16539.10 Hz . -19.167 ppm -1446.52 Hz 11.91610 ppm/cm 899.28101 Hz/cm

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Current Data Parameters PL-Ju121-07 F2 - Acquisition Parameters 20070721 17.35 spect 5 mm QNP 1H/1 **zg**30 65536 CD5CJ5 16 5 6172.839 Hz 0.094190 Hz 5.3084660 sec 114 81.000 usec 5.00 usec 300.0 K 1.00000000 sec ========== CHANNEL f1 ======== 1H 11.70 usec 0.00 dB 299,8718518 MHz - Processing parameters 32768 299.8700143 MHz no 0 0.00 Hz 0 1.00 1D NMR plot parameters 20,00 cm 11.000 ppm 3298.57 Hz ~1.000 ppm -299.87 Hz 0.60000 ppm/cm 179,92201 Hz/cm





95.827	83.022 78.838 63.180 61.113 54.523 54.163 53.801 53.126 53.126 53.126 53.126 53.126 53.173 53.173 53.173 53.173 53.173 53.173 53.173 53.173	



Current Data Parameters PL-Ju121-07 2 PROCNO 1 F2 - Acquisition Parameters 20070721 17.55 INSTRUM spect PROBHD 5 mm GNP 1H/1 PULPROG zgpg30 65536 CD2C12 SOLVENT 162 4 18796.992 Hz 0.286819 Hz FIORES 1,7433076 sec 362 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0,03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz waltzi6 ίH 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz F2 - Processing parameters 32768 75.4023468 MHz ЕM 0 1.00 Hz 0 1.40 1D NMR plot parameters 20.00 cm 234.574 ppm 17687.44 Hz -14.715 ppm -1109.55 Hz 12.46446 ppm/cm 939.84955 Hz/cm

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Current Data Parameters PL-Ju127-07 F2 - Acquisition Parameters 20070727 15.19 spect 5 mm BBI 1H-B zg30 65536 CDC13 16 2 8278,146 Hz 0.126314 Hz 3.9584243 sec 143.7 60.400 usec 5.00 usec 300.0 K 1.00000000 sec 1H 6.45 usec 0.00 dB 400.1324710 MHz F2 - Processing parameters 32768 400.1300166 MHz ΠQ 0 0.00 Hz 0 1.00 1D NMR plot parameters 20.00 cm 11.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm

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					P1 PL1 SF01 SF01 SF02 PCPD2 PL2 PL12 PL12 PL13 SF02 F2 - Pr SI SF WDW SSB LB GB PC 1D NMR CX F1P F1 F2P
0	75	50	25	0	F2 PPMCM HZCM

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Data Parameters PL-Sep10-07 2 1 equisition Parameters 20070910 10.40 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 112 4 18796.992 Hz 0.286819 Hz 1.7433076 sec 2048 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 Usec -6.00 dB 75.4106357 MHz ----- CHANNEL f2 ========= waltz16 1H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz rocessing parameters 32768 75.4023878 MHz ЕМ 0 1,00 Hz 0 1.40 plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42627 Hz/cm

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Current Data Parameters PL-Sep28-07 3 F2 - Acquisition Parameters 20070928 23.16 spect 5 mm QNP 1H/1 zg30 65536 CDC13 16 2 6172.839 Hz 0.094190 Hz 5.3084660 sec 181 81.000 usec 6.00 usec 300.0 К 1.00000000 sec 1H 11.70 usec 0.00 dB 299.8718518 MHz Processing parameters 32768 299.8700161 MHz nα 0 0.00 Hz 0 1.00 10 NMR plot parameters 20.00 cm 11.000 ppm 3298.57 Hz -1.000 ppm -299.87 Hz 0.60000 ppm/cm 179.92201 Hz/cm

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Current Data Parameters PL-Aug21-07 2 F2 - Acquisition Parameters 20070822 0.00 spect 5 mm BBI 1H-B zg30 65536 CDC13 16 2 8278.146 Hz 0.126314 Hz 3.9584243 sec 45.3 60.400 usec 6.00 usec 300.0 K 1.00000000 sec 1H 6.45 usec 0.00 dB 400.1324710 MHz - Processing parameters 32768 400.1300000 MHz no 0 0.00 Hz 0 1.00 10 NMR plot parameters 20.00 cm 11.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm

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Current Data Parameters PL-Aug23-07 Э 1 F2 - Acquisition Parameters 20070823 16.39 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 1400 4 18796.992 Hz 0.286819 Hz 1.7433076 sec 1024 25.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz waltz16 **1**H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz F2 - Processing parameters 32768 75.4023792 MHz EM 0 . .-1.00 Hz 0 1.40 10 NMR plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42621 Hz/cm



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								k k						-							SF WDW SSB LB GB PC 1D NMF CX	
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it Data Parameters PL-Feb05-08 З 1 cquisition Parameters 20080205 12.55 spect M 5 mm BBI 1H-B zg30 G 65536 CDC13 16 2 8278.146 Hz 3.9584243 sec 28.5 . 60.400 usec 6.00 usec 300.0 K 1.00000000 sec ====== CHANNEL f1 ======== **1**H 6.45 usec 0.00 dB 400.1324710 MHz Processing parameters 32768 400.1300174 MHz no 0 0.00 Hz 0 1.00 4R plot parameters 👘 20,00 cm 11.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm



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Current Data Parameters PL-Oct07-07 5 1 F2 - Acquisition Parameters 20071008 0.01 spect 5 mm QNP 1H/1 zgpg30 65536 COC13 1113 4 18796.992 Hz 0.286819 Hz 1.7433076 sec 2048 25.600 usec 5.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz waltz16 1H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz F2 - Processing parameters 32768 75.4023769 MHz ЕМ 0 1.00 Hz 0 **i**.40 1D NMA plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42615 Hz/cm





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Current Data Parameters PL-Oct08-67 NAME EXPNO 2 PROCNO 1 F2 - Acquisition Parameters 20071006 Date_ Time 18,1B INSTRUM spect PROEHO 5 mm 683 1H-6 PULPAGG TO inv4gptp 2040 C0C13 SOLVENT ns Os Swh 4 ę0 2620.545 Hz 1.279563 Hz 0.3908064 sec FIDAES 40 AC: 7293.2 190,800 vsec ÐW Œ 6.00 usec 300.0 K 145.0000000 0.00000300 Sec 1.99631298 Sec 0.00344626 Sec 0.00172414 Sec 0.03000000 Sec CNS12 **d**0 D1 02 04 011 0.00000300 sec d13 D16 0.00020000 sec 0.00052414 sec d 20 d21 0.00224428 sec INO 0.00001490 sec ETCECTETETET CHANGE [] ETECETETETE NJC1 ۶H P1 7.‡0 u≤ec p2 14.20 uSec PLS 0.00 dB 5701 400.1317545 MHz ganp 130 17.00 usec CPDPAG2 NUC2 ₽₹ p4 34.00 usec PCP02 64.**0**0 usec PL2 -6.00 69 PL12 6.30 08 SF02 100.6202332 MHz GRADIENT CHANNEL STATES GPNAM) 5INE,100 51NE.100 GPNAH2 5INE.:00 GPNAH3 GPX 17,00 🕱 20.00 🕱 GPX2 GFX3 25.00 X GPY1 17.00 X GPY2 20.00 X GPY3 25.00 % GPZ1 17.00 X GPZ2 20.00 X GPZ3 25.00 X P16 1000.00 usec F: - Acquisition parameters X630 2 τD 255 SF01 100.5202 NHz FIDRES 65.541107 Hz 166.751 ppp 51 f2 - Processing parameters 21 2046 SF 400.1300000 MHz hDhi GSINE 556 L0 66 - 2 0.00 Hz 0 FC 1.00 F1 - Processing parameters 51 MC2 57 MOM S58 L6 68 1024 TPPE 100.5127290 MHz GSENE 2 0.00 Hz Û 20 NHA plot parameters 20,00 cm 15,00 cm CX2 CX1 f 2P2.0 5.962 ppm F2LQ F2PHI 2365,39 Hz 1.331 ppm 532.59 Hz F 2H 3 134.353 ppm F 1950 13517.58 Hz FļLŪ 7.326 ppm 737.07 Hz 0.23153 ppm/cm F IPil1 F 1H! F2PPMCM F2HZCM 92.54037 H2/CA 165 6

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Corrept Data Parameters PL-OctOB-07 NAME EXPN0 4 PROCNO 1 F2 - Acquisition Parameters 20071008 Oate_ Time 21.16 INSTRUM spect PROSHD 5 mm 861 iH-8 PULPROG ajb.cosygsoftp 2048 TD SOL VENT NS COC) 3 4 **D**5 6 SWH 2520.545 Hz 1,279563 Hz FIDRES 0.3908084 sec AG 5792.6 RG j90.800 usec CN 6.00 usec DE 300.0 K ٦E 0.00000300 sec 00 Di 4.00000000 sec 0.00000300 sec di3 D16 0.00020000 Sec 02D G.00120300 sec ≩N0 0.00015020 sec -----CHANNEL (S SERVERSE NBCS ĩН 7.10 usec P1 14.20 usec 65 0.00 dB ₽Lí \$F01 400.1317545 KHz ======= GRADIEN; CHANNEL ====== P\$6 1000.00 usec Fi - Accuisition parameters NDO 2 650 ťD 5F D 1 400.1318 MHz FIDRES 4.031608 Hz 6.549 ppm SY F2 - Processing parameters 2048 51 400.1300000 MBz SF SINE KDK 558 0 0,00 Hz LB 68 0 1.40 PC F1 - Processing parameters 1024 TPP1 51 MC2 SF 400.1300000 MHz WDW SINE SSB 0 ĽΒ 0.00 Hz 6B 0 20 NMR plot parameters 15.00 cm CXS 15.00 cm CXi 6.211 ppm 2485.20 Hz F2PL0 F2L0 1.146 ppm 458.37 Hz 6.496 ppm ES54J F2H1 FJPLO 2599.08 Hz 1.245 ppm FILO FiPHI 498.04 Hz F 1H1 0.33769 ppm/cm 136.12186 Hz/cm 0.35000 ppm/cm F2PPMCM F 2HZCM F 1PPMCM Hz/Gr

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Current Data Parameters PL-Oct06-07 NAME EXPN0 PROCNO 1 F2 - Acquisition Parameters 20071006 Qate_ Time 19.00 INSTRUM spect PROBHŌ 5 mm 881 1H-8 PULPROS inv4gslplrnd 2048 τD CDC13 SOLVENT NS θ D5 SKH 16 2620.545 Hz FIDRES 1.279563 Hz AQ 0.3908064 sec RG 15364 190.800 usec DW: DE 6.00 usec TE CNST2 300.0 K 145.0000000 dO 0.00000300 sec 01 1.50000000 sec 0.00344828 sec d2 95 0.06500000 sec d13 0.00000300 sec D16 0.00020000 sec 0.00002240 sec INO ======= CHANNEL f1 =========== NUC1 114 7.10 usec P٩ 14.20 usec p2 0.00 dB PLi SFO 400.1317546 MHz ------ CHANNEL 12 FERRESEERE NUC2 13C P3 17.00 usec -6.00 dB PL2 100.6227903 MHz SF02 ********** GRADIENT CHANNEL ****** P16 1000.00 usec F1 - Acquisition parameters NDQ 2 ΤŌ 512 SF01 100.6228 MHz 43,596539 Hz FIDRES 221.**833** ppm SN F2 - Processing parameters SI 204B 40D.1301597 MHz SF SINE WDX SS6 Q LÐ 0.00 Hz GB Q РÇ 1.40 F1 - Processing parameters 1024 SI NC2 0F SF 100.6126736 MHz KDW SINE S58 Ð LB 0.00 Hz GB. 0 20 NMA plot parameters CX2 20.00 cm CXS 17.00 cm F2PLO 5,869 ppm F2L0 2348.52 Hz 0.746 ppm ES5HI 298.66 Hz F2HI FIPLO 211.261 ppm 21255.57 Hz FILO -8,860 ppm F1PHI -891.47 Hz Fiki 0.25615 ppm/cm F2PPMCM 102.49300 Hz/cm L5HZCM F FPMCN 94834 ppm/c

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PL-0ct08-07 5 11 -F2 - Acquisition Parameters 20071009 0.31 spect 5 mm 88I 1H-8 noesygptp 2048 CDC13 8 8 2620.545 Hz 1.279563 Hz 0.3908084 sec 35,9 190.800 usec 6.00 usec 300.0 K 0.00000300 sec 4.00000000 sec 0.8000001 sec 0.00020000 sec 0.39880002 sec 0.00019080 sec 1H 7.10 usec 14.20 usec 0.00 dB 400.1317546 MHz ========= GRADIENT CHANNEL ======== sine.100 sine,100 0.00 % 0.00 % 0.00 % 0.00 % 40.00 % -40.00 % 1000.00 usec F2 - Processing parameters 2048 400.1300092 MHz QSINE 2 0.00 Hz 0 **i**.00 1D NMA plot parameters 20.00 cm **ii**.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm



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PL-Oct08-07 5 5 F2 - Acquisition Parameters 20071009 0.31 spect 5 mm 88I 1H~B noesygptp 2048 CDC13 8 8 2620.545 Hz 1.279563 Hz 0.3908084 sec 35.9 190.800 usec 6.00 usec 300.0 K 0.00000300 sec 4.00000000 sec 0.8000000i sec 0.00020000 sec 0.39880002 sec 0.00019080 sec 1H7.10 usec 14.20 usec 0.00 dB 400.1317546 MHz ========= GRADIENT CHANNEL ======== sine.100 sine.100 0.00 % 0.00 % 0.00 % 0.00 % 40.00 % -40.00 % 1000.00 usec F2 - Processing parameters 2048 400.1300092 MHz QSINE 2 0.00 Hz 0 1.00 1D NMR plot parameters 20.00 cm 11.000 ppm 4401.43 Hz -i.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm

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PL-0ct08-07 5 29 F2 - Acquisition Parameters 20071009 0.31 spect 5 mm 881 1H-B noesygptp 2048 CDC13 8 8 2620.545 Hz 1.279563 Hz 0.3908084 sec 35.9 190.800 usec 5.00 usec 300.0 K 0.00000300 sec 4.00000000 sec 0.80000001 sec 0.00020000 sec 0.39880002 sec 0.00019080 sec 1H 7.10 usec 14.20 usec 0.00 dB 400.1317546 MHz ========== GRADIENT CHANNEL ======== sine.100 sine.100 0.00 % 0.00 % 0.00 % 0.00 % 40.00 % -40.00 % 1000.00 usec F2 - Processing parameters 2048 400.1300092 MHz Q\$INE 2 0.00 Hz 0 1.00 1D NMR plot parameters 20,00 cm 11.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm





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> Current Data Parameters PL-Feb02-08 F2 - Acquisition Parameters 20080202 14.08 spect 5 mm QNP 1/1 zg30 65536 COC13 16 2 6172,839 Hz 0.094190 Hz · 5.3084660 sec 128 81.000 usec 6.00 usec 300.0 K 1.00000000 sec ======== CHANNEL f1 ========= 1H 11.70 usec 0.00 dB 299.8718518 MHz Processing parameters 32768 299.8700161 MHz na 0 0.00 Hz 0 1.00 10 NMR plot parameters 20.00 cm 11.000 ppm 3298.57 Hz -1.000 ppm -299.87 Hz 0.60000 ppm/cm 179.92201 Hz/cm



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Current Data Parameters PL-Feb02-08 NAME EXPNO 2 PROCNO 1 F2 - Acquisition Parameters Date_ 20080202 14.14 Time INSTRUM spect PROBHO 5 mm GNP 1H/1 PULPROG zgpg30 65536 SOLVENT CDC]3 55 4 SWH 18796.992 Hz FIDAES 0.286819 Hz 1.7433076 sec 1024 25.600 Usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec ========= CHANNEL f1 ============== 13C NUCI 5.40 usec Ρ1 PL1 -6.00 d8 75.4106357 MHz SF01 CPOPRG2 waltz16 NUC2 1H 115.00 usec PCPD2 0.00 dB PL2 PL 12 20.00 dB PL13 20.00 dB SF02 299.8711995 MHz F2 - Processing parameters SI 32768 SF 75.4023410 MHz WDW Ем 0 558 1.00 Hz L8 0 G8 1.40 ΡÇ 1D NMR plot parameters 20.00 cm СХ FiΡ 215.000 ppm 16211.50 Hz Fi -5.000 ppm F2P F2 -377.01 Hz PPMCM 11.00000 ppm/cm HZCM. 829.42578 Hz/cm

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																					Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AG RG DW DE TE D1
				منغ	L																NUC1 P1 PL1 SF01 F2 - Pr SI SF WDW SSB LB GB PC 1D NMR CX F1P
	0.0984	0.0743	0.9837	0.0604	2.1654	3.1396 // 2 8787 //		1. 3002	3.6634	2.7819	1.1257	1.1102///	<u>1.3880/</u>								F1 F2P F2 PPMCM HZCM
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Current Data Parameters PL-Feb10-08 2 1 cquisition Parameters 20080210 15.43 spect 5 mm GNP 1H/1 zg30 6 65536 . CDC13 16 2 6172.839 Hz 0.094190 Hz 5.3084660 sec 114 81.000 usec 6.00 usec 300.0 K 1.00000000 sec ===== CHANNEL fi ======= 1H 11.70 usec 0.00 dB 299.8718518 MHz rocessing parameters 32768 299.8700159 MHz រាល 0 0.00 Hz 0 1.00 plot parameters 20.00 cm 11.000 ppm 3298.57 Hz -1.000 ppm -299.87 Hz 0.60000 ppm/cm 179.92201 Hz/cm

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PL-Feb10-08 3 1 F2 - Acquisition Parameters 20080210 15.50 spect 5 mm GNP 1H/1 zgpg30 65536 CDC13 119 4 18795.992 Hz 0.286819 Hz 1,7433076 sec 2048 25.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz ========= CHANNEL f2 ============== waltz16 **1**H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz F2 - Processing parameters 32768 75.4023769 MHz ΕM 0 1.00 Hz 0 1.40 1D NMR plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42615 Hz/cm

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Current Data Parameters PL-Feb15-08 F2 - Acquisition Parameters 20080215 10.05 spect 5 mm QNP 1H/1 zg30 65536 CDC13 16 2 6172.839 Hz 0.094190 Hz 5.3084660 sec 203.2 81.000 usec 6.00 usec 300.0 K 1.00000000 sec 1H 11.70 usec 0.00 dB 299.8718518 MHz F2 - Processing parameters 32768 299.8700157 MHz no 0 0.00 Hz 0 1.00 1D NMR plot parameters 20.00 cm 11.000 ppm 3298.57 Hz -1.000 ppm -299.87 Hz 0.60000 ppm/cm 179.92201 Hz/cm

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77.427	59.362 56.076 56.076 48.772 48.730			Current NAME EXPNO PROCNO F2 - ACC Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG DW DE TE D1 D11 D12 ======= NUC1 P1 PL1 SF01 ======= NUC1 P1 PL1 SF01 ======= CPDPRG2 NUC2 PCPD2 PL2 PL12 PL13 SF02 F2 - Pro SI SF WDW SSB LB GB PC 1D NMR p CX F1P F1
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Data Parameters PL-Nov18-07 1 equisition Parameters · 20071118 22.56 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 2160 4 18796.992 Hz 0.286819 Hz 1.7433076 sec 1024 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 13C • 5.40 usec -6.00 dB 75.4106357 MHz waltz16 1H 115.00 usec 0.00 dB 20.**00** dB 20.00 dB 299.8711995 MHz ocessing parameters 32766 75.4023746 MHz ΕM 0 1.00 Hz 0 1.40 plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42615 Hz/cm

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Current Data Parameters NAME PL-Mar30-08 73739 75739 75758 7577 75759 75559 75559 71737 73414 71737 73415 71737 73415 71737 73415 71737 73415 71737 73415 71737 73415 71737 71737 73415 71737 71747 7 EXPNO PROCNO •• F2 - Acquisition Parameters \mathcal{D} \circ 50080330 Date_ Time 20.41 INSTRUM spect 5 mm 88I 1H-B PROBHO PULPAOG zg30 65536 ΤD CDC13 SOLVENT 16 NS DS 2 SWH 8278.146 Hz 0.126314 Hz FIDRES 3,9584243 sec AG RG 114 DW 60.400 usec <u>-</u> DE 6.00 usec ΤE 300.0 K Di 1.00000000 sec ========= CHANNEL f1 ======== NUC 1 1H 2 6.45 usec P1 0.00 dB PL1 SF01 400.1324710 MHz F2 - Processing parameters SÏ 32768 400.1300169 MHz SF WDW no SS8 0 LB 0.00 Hz GB 0 PC 1.00 1D NMR plot parameters СX 20.00 cm F1P 11.000 ppm **F** 1 4401.43 Hz F2P -1.000 ppm F2 -400.13 Hz 0.60000 ppm/cm PPMCM 240.07800 Hz/cm

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Data Parameters PL-Mar14-08 2 1 quisition Parameters 20080314 12.45 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 514 4 18796.992 Hz 0,286819 Hz i.7433076 sec 1625.5 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz waltz16 1H 115.00 Usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz rocessing parameters 32768 75,4023763 MHz ΕM 0 1.00 Hz 0 1.40 plot parameters -20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm B29.42615 Hz/cm

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nt Data Parameters PL-Dec 19-07 Acquisition Parameters 20071219 17.33 spect JM 5 mm 881 1H-B)G zg30 24576 CDC13 T 16 . 0 8278.146 Hz 0.336839 Hz 1.4844404 sec 322.5 6.00 usec 300.0 K 1.00000000 sec ====== CHANNEL f1 ======== iΗ 6.45 usec 0.00 dB 400.1324710 MHz Processing parameters 32768 400.1300174 MHz no 0 0.00 Hz 0 1.00 plot parameters 20.00 cm 11.000 ppm 4401.43 Hz ~1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07802 Hz/cm

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				PL1 SF01

rent Data Parameters PL-Dec 19-07 2 1 Acquisition Parameters 20071220 9.46 spect 5 mm Multinu ROG zgpg30 65536 CDC13 9747 4 18832.393 Hz 0.287360 Hz 1.7400308 sec 16384 26.550 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec ======= CHANNEL f1 ============= 130 11.80 usec 0.00 dB 75.4760200 MHz RG2 waltz16 1H 110.00 usec 0.00 dB 17.50 dB 17.50 dB 300.1312005 MHz Processing parameters 32768 75.4677509 MHz EM 0 1.00 Hz 0 1.40 IMA plot parameters 20.00 cm 215.000 ppm 16225.57 Hz ~5.000 ppm -377.34 Hz 11.00000 ppm/cm 830.14526 Hz/cm

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Data Parameters PL-Mar08-08 quisition Parameters 20080308 13.17 spect 5 mm GNP 1H/1 zg30 65536 CDC13 16 2 6172.839 Hz 0.094190 Hz 5.30B4660 sec 228.1 81.000 usec 6.00 usec 300.0 K 1.00000000 sec ===== CHANNEL f1 ======= 1H 11.70 usec 0.00 dB 299.8718518 MHz rocessing parameters 32768 299.8700161 MHz no 0 0.00 Hz 0 1.00 plot parameters 20.00 cm 11.000 ppm 3298.57 Hz -1.000 ppm -299.87 Hz . 0.60000 ppm/cm 179.92201 Hz/cm

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Current Data Parameters NAME PL-Dec29-07 EXPNO 2 PROCNO 3 F2 - Acquisition Parameters Date_ 20071229 13.53 Time INSTRUM spect PROBHD 5 mm GNP 1H/1 PULPROG zgpg30 TD 65536 SOLVENT CDC13 1377 NS 0S 4 SWH 18796.992 Hz FIDRES 0.2868**19** Hz 1.7433076 sec AQ 1024 RG 26.600 usec DW DE 6.00 usec ΤE 300.0 К D1 2.00000000 sec D11 0.03000000 sec D12 0.00002000 sec 130 NUC1 5.40 usec Ρ1 PL1 -6.00 dB SF01 75.4106357 MHz CPOPRG2 waltz16 NUC2 1H 115.00 usec PCPD2 0.00 dB PL2 20.00 dB PL12 20.00 dB PL13 SF02 299.8711995 MHz F2 - Processing parameters SI 32768 SF 75.4023757 MHz WDW ΕM \$\$B 0 LВ 1.00 Hz 0 GΒ PC 1.40 1D NMR plot parameters 20.00 cm СХ F1P 234.1**9**0 ppm F1 17658.51 Hz F2P -15.099 ppm F2 -1138.48 Hz PPMCM 12.46446 ppm/cm 75 50 25 100 HŻĊM 939.84961 Hz/cm

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Data Parameters PL-Jan04-07 equisition Parameters 20080104 23.05 spect 5 mm QNP 1H/1 zg30 65536 CDC13 16 2 6172.839 Hz 0.094190 Hz 5.3084660 sec 256 81.000 usec 6.00 usec 300.0 K 1.00000000 sec ===== CHANNEL f1 ======= íΗ 11.70 usec 0.00 dB 299.8718518 MHz . rocessing parameters 32768 299.8700159 MHz 10 0 0.00 Hz 0 1.00 plot parameters 20.00 cm 11.000 ppm . 3298.57 Hz -i.000 ppm -299.87 Hz 0.60000 ppm/cm 179.92201 Hz/cm

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				D12 ======= NUC1 P1 PL1 SF01 =======
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Data Parameters PL-Jan03-07 3 1 quisition Parameters 20080103 21.14 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 2556 4 18796.992 Hz 0.286B19 Hz 1.7433076 sec 512 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz ----- CHANNEL f2 ------waltz16 1H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz rocessing parameters 32768 75.4023757 MHz ΕM 0 . 1.00 Hz 0 1.40 plot parameters 20.00 cm 215.000 ppm 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42615 Hz/cm

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nt Data Parameters, PL-Jan06-0入 Acquisition Parameters 20080107 0.19 spect 5 mm QNP 1H/1 zg30)G 65536 . CDC13 16 2 6172.839 Hz 0.094190 Hz 5.3084660 sec 458.1 81.000 usec 6.00 usec 300.0 К 1.00000000 sec ====== CHANNEL f1 ======== . 1H 11.70 usec 0.00 dB 299.8718518 MHz . Processing parameters 32768 299.8700163 MHz no 0 0.00 Hz 0 1.00 plot parameters 20.00 cm 11.000 ppm 3298.57 Hz -1.000 ppm -299.87 Hz 0.60000 ppm/cm (-179.92201 Hz/cm

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Data Parameters PL-Jan06-07 - 7 1 quisition Parameters 20080107 2.40 spect 5 mm QNP 1H/1 zgpg30 65536 CDC13 2024 4 18796.992 Hz 0.286819 Hz 1.7433076 sec 1024 25.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz ===== CHANNEL f2 ========== waltz16 1H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz ocessing parameters 32768 75.4023746 MHz ЕМ 0 1,00 Hz 0 1.40 plot parameters 20.00 cm 215.000 ррт 16211.51 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42615 Hz/cm .

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Current Data Parameters PL-Jan07-08 F2 - Acquisition Parameters 20080108 14.41 spect 5 mm BBI 1H-B zg30 65536 CDC13 15 2 8278.146 Hz 0.126314 Hz 3.9584243 sec 362 60.400 usec 6.00 usec 300.0 K 1.00000000 sec ========== CHANNEL f1 ========= 1H 6.45 usec 0.00 dB 400.1324710 MHz F2 - Processing parameters 32768 400.1300176 MHz no 0 0.00 Hz 0 1.00 1D NMR plot parameters 20.00 cm 11.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07800 Hz/cm

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Current Data Parameters PL-Apr05-08 З F2 - Acquisition Parameters 20080405 16.56 spect 5 mm BBI 1H-B zg30 65536 C6D6 16 2 8278.146 Hz 0.126314 Hz 3.9584243 sec 362 60.400 usec 6.00 usec 300.0 K 1.00000000 sec ========= CHANNEL f1 ======== 1H 6.45 usec 0.00 dB 400.1324710 MHz Processing parameters 32768 400.1300436 MHz no 0 0.00 Hz 0 1.00 iD NMR plot parameters 20.00 cm 11.000 ppm 4401.43 Hz -1.000 ppm -400.13 Hz 0.60000 ppm/cm 240.07802 Hz/cm

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 75.418	60.03		 28.911	Current NAME EXPNO PROCNO F2 - Ac Date_ Time INSTRUM PROBHO PULPROG TD SOLVENT NS OS SWH FIDRES AQ RG OW DE TE D1 D11 D12 ======= NUC1 P1 PL1 SF01
		-		CPDPRG2 NUC2 PCPD2 PL2 PL12 PL13 SF02 F2 - Pri
				SI SF WDW SSB LB GB PC
75		50	25	1D NMA (CX F1P F1 F2P F2 PPMCM HZCM

Data Parameters PL-Apr06-08 1 1 cquisition Parameters 20080406 11,15 spect 5 mm GNP 1H/1 zgpg30 65536 C6D6 1191 4 18796,992 Hz 0.286819 Hz 1.7433076 sec 2048 26.600 usec 6.00 usec 300.0 K 2.00000000 sec 0.03000000 sec 0.00002000 sec 130 5.40 usec -6.00 dB 75.4106357 MHz waltz16 1H 115.00 usec 0.00 dB 20.00 dB 20.00 dB 299.8711995 MHz rocessing parameters 32768 75.4023410 MHz ЕM 0 1.00 Hz 0 1.40 plot parameters 20.00 cm 215.000 ppm 16211.50 Hz -5.000 ppm -377.01 Hz 11.00000 ppm/cm 829.42578 Hz/cm

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Current Data Parameters PL-Apr05-06 NAME EXPNO з PROENC 3 F2 - Acquisition Parameters 20060405 Date_ Time 18,12 INSTRUM spect Paobho Pulpaog To 5 ma 861 1H-6 inv49ptp 2048 SOLVENT C616 NS DS SWH FIDRES 4 69 2053.061 Hz 1.292497 Hz A۵ 0.3598596 sec 18390.4 HE 175.200 USEC DN DE 6,00 esec Ī€ 300,0 K CNS12 145.0000000 d0 D1 0.00000300 sec 1.59631259 sec d2 d4 0.00344628 sec 0.00172414 sec dii 0.03000000 sec d13 016 0.00000300 sec 0.00020000 sec d50 0.00052414 sec 0.00224428 sec 0.00001490 sec d21 IND NUC1 P1 p2 PL1 18 5.81 usec 13.52 USec 0.00 dB 5F01 400.1316713 MHz ----- CHANNEL 12 ARCHIGE CPDPAG2 garp 13C NUC2 17.00 usec PЭ 34.00 USec ₽¢ PCPD2 64.00 usec FL2 -5.00 58 PL12 5.30 dB 5F02 100.6202332 MHz GPNAH1 SINE.100 STNE, 100 GPNAH2 GPNAH3 STNE . 100 17.00 % CPX1 GPX2 20.00 1 6PX3 25.00 X 6PY1 17.00 % GPY2 20,00 % 6PY3 25.00 X GPZi 17.00 % 6PZ2 20.00 X GPZ3 25.00 % FiĘ 1000.00 usec F1 - Acquisition parameters КОФ 70 4 256 SF01 100.6202 MHz FIDAES 65.541107 Hz SK 166.751 ppm F2 - Processing parameters SI SF 2048 400.1300000 MHz kok SSB LB GB PC **OSINE** - 2 0.00 Hz 0 1.00 F\$ - Processing parameters si NC2 SF NDN SSE LB 1024 1PP1 100.6127290 MHz OS1NE - 2 0,00 Hz GE 0 20 NMA plot parameters CX2 20,00 сл CX1 15.00 cm F2PLD 7.743 ppn FELO 3090.20 Hz F2PH 0,611 ppm F2HI 244.40 Hz FIPLD 157.957 ppm Filū 15893.45 Hz F3PHI -6,797 ррм F\$HI -285.07 Hz FEPPINCK 0.35562 ppe/cm F2HZCM 142.69406 Hz/cm F SPPRCK 11.11756 ppm/cm FIRZEM 1116.55624 Hz/cm

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Current Data Parameters PL-Apr05-08 NAME EXPNO 5 PROCND 1 F2 - Acquisition Parameters 20080406 Date_ 0.49 Time INSTRUM spect PA08H0 5 mm 8BI 1H-B PULPROG ajb.cosygsmftp ΤD 2048 SOLVENT C605 NS 4 DS θ SWH 2853.801 Hz 1.393497 Hz FIORES 0.3588596 sec AG 2895.3 ЯG 175,200 usec DW ۵E 6.00 Usec 300.0 K ΤĘ 0.00000300 sec 00 4,00000000 sec D1 d13 0,00000300 sec 016 0.00020000 sec 0.00120300 sec ď20 0.00009360 sec INO NUC1 1H Ρ1 6.81 usec 13.62 usec P2 PL1 0.00 **d**8 400.1316713 MHz SFD1 P16 1000.00 usec Fi - Acquisition parameters NDÓ 2 650 TD SF01 400.1317 MHz FIDRES 8.218277 Hz 13.350 ppm SH F2 - Processing parameters SI 2048 ŞF 400.1300000 MHz KDK ŞINE SSB 0 0.00 Hz L**B** 68 0 i.00 PC F1 - Processing parameters 1024 Sī MC2 SF TPPI 400,1300000 MHz WDW SINE SSB 0 LB 0.00 Hz G₿ 0 2D NMR plot parameters CX5 15.00 cm CX1 15.00 cm 7,548 ppm F2PL0 F2LO 3020.25 Hz F2PHI 0,642 ppm F2HI 256.94 Hz F1PL0 7.814 ppm 3126.80 Hz FilO -1.546 ppm FIPHI F1HI -618.78 Hz 0.46040 ppm/cm F2PPMCM 184.22026 Hz/cm F2HZCM 0.62406 ppm/cm F 1PPMCM 249.70508 Hz/cm F 1HZCM

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Current Data Parameters PL-Apr05-08 NAME EXPNO PROCNO 1 F2 - Acquisition Parameters Date_ 20080405 Time 5.00 INSTRUM spect PROBHD 5 mm 881 1H-8 PULPAOG inv4gslp]rnd 2048 TD SOLVENT C606 NS B DS 16 SWH 2853,86; Hz FIORES 1.393497 Hz 0.3568596 sec AO 8192 RG 175.200 usec DK DE 6.00 usec ΤE 300.0 K CNST2 145.0000000 0.00000300 sec 60 1.50000000 sec 01 0.00344628 sec d2 06 0.06500000 sec d13 0.00000300 sec **016** 0,0002000D sec 0.00002240 sec INO ========= CHANNEL f1 ============== NUC1 1H 6.01 Usec P۱ 13.62 usec p2 PL1 SF01 0.00 dB 400.1316713 MHz ERFERENCE CHANNEL 12 =============== NUC2 130 P3 17.00 usec -6.**00** d8 PL2 SF02 100.6227903 NHz F==F====== GRADIENT CHANNEL ======== P16 1000.00 usec F1 - Acquisition parameters NDO 2 512 TD SF01 100.6228 MHz FIDRES 43.596539 Hz 221.833 ppm SW F2 - Processing parameters ŞI SF WOW 2046 400.1301597 MHz SINE SSB 0 LB 0.00 Hz GB 0 1,40 PC Fi - Processing parameters SI 1024 QF MC2 100.6126736 MHz SF NDX SSB SINE 0 LB 0.0D Hz GB Ū 20 NMR plot parameters CX5 20.00 cm CX1 17.00 Cm F2PL0 7.344 ppm F2L0 2938.57 Hz E S D H I 0.212 ppm F2HI 84.69 Hz FIPLO 211.478 ppm F1L0 21277.37 Hz -10.377 ppm F1PHI -1044.06 Hz F1HI 0.35662 ppm/cm F2PPMCM F2HZCM 142.694D6 Hz/cm 13.05030 ppm/cm F1PPMCM F1HZCM 1313.02515 Hz/cm

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Current Data Parameters PL-Apr05-08 4 11 F2 - Acquisition Parameters 20080405 18.54 spect 5 mm BBI 1H-B noesygptp 2048 C606 8 8 2853.881 Hz 1.393497 Hz 0.3588596 sec 128 175.200 usec 6.00 usec 300.0 К 0.00000300 sec 4.00000000 sec 0.80000001 sec 0.00020000 sec 0.39880002 sec 0.00012495 sec ========= CHANNEL f1 =========== 1H 6.81 usec 13.62 usec 0.00 dB 400.1316713 MHz GRADIENT CHANNEL ====== sine.100 siле.**100** 0.00 % 0.00 % 0.00 % 0.00 % 40.00 % -40.00 % 1000,00 usec F2 - Processing parameters 2048 400.1300092 MHz QSINE 2 0.00 Hz 0 1.00 iD NMR plot parameters 20.00 cm 7.000 ppm 2800.91 Hz 1.000 ppm 400.13 Hz 0.30000 ppm/cm 120.03900 Hz/cm