Electronic Supplementary Information

Total Synthesis of (-)-20-Epiuleine via Stereocontrolled One-pot Asymmetric Azaelectrocyclization Followed by Novel 1,4-Addition Reaction

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General Procedure

All commercially available reagents were used without further purification. All solvents were used after distillation. Tetrahydrofuran, diethyl ether and toluene were refluxed over and distilled from sodium. Dichloromethane was refluxed over and distilled from P₂O₅. Dimethylformamide (DMF) was distilled from CaH₂. Preparative separation was usually performed by column chromatography on silica gel. The ¹H NMR and ¹³C NMR spectra were recorded using a 400 MHz spectrometer, and chemical shifts were represented as δ-values relative to the internal standard TMS. The IR spectra were recorded by a FT-IR spectrometer. The high-resolution mass spectra (HRMS) were measured by an ESI-TOF MS.

Ethyl (2S)-1-[(1S,2R)-2-hydroxy-7-isopropylindan-1-yl]-2-phenyl-1,2,5,6-tetrahydropyridine-4-carboxylate (27a):

To a solution of aminoacetal (359 mg, 0.616 mmol) in THF (6 ml) were added sodium borohydride (114 mg, 3.01 mmol) and a trifluoroborane ethereate complex (0.076 ml, 0.616 mmol) at 0 °C. After the mixture was stirred at 0 °C for 30 min, H₂O was added, and the resulting mixture was extracted with ether. The organic layers were combined, washed with brine, dried over MgSO₄, filtered, and concentrated in vacuo to give the alcohol product 27a (81 mg, 90%) as yellow amorphous: IR (neat, cm⁻¹) 2959, 2360, 1690, 1238, 1087, 697; ¹H NMR (400 MHz, CDCl₃, 50 °C) δ 7.06-7.34 (m, 7H), 6.81-6.92 (br s, 1H), 6.70-6.81 (br s, 1H), 4.73-5.15 (br s, 1H), 4.41-4.58 (br s, 1H), 4.28 (d, 1H, J = 6.6 Hz), 4.14-4.24 (m, 2H), 2.87-3.13 (br s, 4H), 2.38-2.74 (br m, 3H), 1.27 (t, 3H, J = 7.1 Hz), 1.14-1.24 (br s, 3H), 0.80-1.00 (br s, 3H); ¹³C NMR (100 MHz, CDCl₃, 50 °C) δ 166.9, 147.7, 140.6, 129.4, 128.9, 128.3, 127.8, 123.4, 122.1, 60.8, 41.2, 26.2, 24.4, 22.9, 14.2, 5.1; ESI HRMS m/z calcld for C₂₆H₂₃N₂O₃ (M+Na)⁺ 428.2202, found 428.2213.

Ethyl (2S,3S)-(−)-3-ethyl-2-phenyl-piperidine-4-carboxylate (29a):

To a solution of 27a (100 mg, 0.247 mmol) in ether (2.5 ml) was slowly added ethyl magnesium bromide (4.9 ml, 4.9 mol, 1.0 M in ether) at 0 °C. After the mixture was stirred for 20 min, H₂O and a 1 N HCl solution were carefully added, and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated in vacuo to give the crude 28a.

To a solution of the crude piperidine and n-propylamine (0.18 ml, 2.22 mmol) in chloroform (2.5 ml) was added lead tetraacetate (438 mg, 0.99 mmol) at -50 °C. After the mixture was stirred for 15 min, it was added to an ice-water bath. The organic layers were combined, washed with water, dried over MgSO₄, filtered and concentrated in vacuo to give the crude products. Column chromatography on silica gel (from 0% to 1.2% methanol in chloroform) gave 29a (53 mg, 82% for 2 steps) in a 2.7 : 1 (0.73 : 0.27) mixture of C2β and C2α stereoisomers as a yellow amorphous solid: IR (neat, cm⁻¹) 2937, 1726, 1455, 1156, 753, 701; ¹H NMR (400 MHz, CDCl₃) δ 7.24-7.38 (m, 5H), 4.05-4.23 (m, 2.27H), 3.40 (d, 0.73H, J = 10.3 Hz), 3.14-3.25 (m, 1H), 3.00 (dd, 0.27H, J = 4.4, 3.9 Hz), 2.95 (ddd, 0.27H, J = 11.9, 11.9, 3.4 Hz), 2.74-2.82 (m, 0.73H), 2.43-2.51 (m, 0.73H), 1.82-2.02 (m, 0.27H, J = 10.31 (m, 5H), 0.73 (t, 0.81H, J = 7.3 Hz), 0.67 (t, 2.19H, J = 7.6 Hz); major isomer ¹³C NMR (100 MHz, CDCl₃) δ 175.3, 142.9, 128.4, 127.9, 127.5, 65.1, 60.2, 47.0, 43.8, 30.3, 22.6, 14.2, 9.4; minor isomer ¹³C NMR (100 MHz, CDCl₃) δ 174.4, 143.7, 128.3, 127.8, 127.2, 61.8, 59.8, 45.3, 42.4, 40.2, 28.9, 22.3, 14.3, 11.5; ESI HRMS m/z calcld for C₁₆H₂₃N₂O₃ (M+H)⁺ 262.1807, found 262.1795.

Ethyl (2S)-1-[(1S,2R)-2-hydroxy-7-isopropylindan-1-yl]-2-(thiophen-3-yl)-1,2,5,6-tetrahydropyridine-4-carboxylate (27b):

To a solution of aminoacetal derivative of thiophene (449 mg, 1.10 mmol) in acetonitrile (11 ml) were added sodium cyanoborohydride (344 mg, 5.48 mmol) and a 2 N HCl solution at 0 °C. After the mixture was stirred at 0 °C for 30 min, a saturated aqueous NaHCO₃ solution was added, and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated in vacuo to give the crude products. Column chromatography on silica gel (from 9.1% to 25% ethyl acetate in hexane) gave 27b (421 mg, 93%) as a yellow amorphous solid: IR (KBr disk, cm⁻¹) 2956, 2360, 1690, 1252, 775; ¹H NMR (400 MHz, CDCl₃, 50 °C) δ 7.24-7.31 (br s, 1H), 7.19 (dd, 1H, J = 7.6, 7.6 Hz), 6.88-7.10 (m, 4H), 6.80 (s, 1H), 4.78-5.04 (br s, 1H), 4.35 (d, 1H, J = 6.6 Hz), 4.14-4.26 (m, 2H), 2.65-3.07 (br m, 3H), 2.80-2.48 (br m, 4H), 1.28 (t, 3H, J = 7.1 Hz), 1.05-1.18 (br d, 3H, J = 5.3 Hz), 0.80-0.97 (br d, 1H, J =...
5.3 Hz); $^{13}$C NMR (100 MHz, CDCl$_3$, 50 °C) $\delta$ 166.8, 147.6, 139.8, 129.1, 128.3, 125.7, 124.0, 123.3, 122.1, 60.5, 41.0, 26.2, 24.8, 22.8, 14.2, 7.1; ESI HRMS m/z calcd for C$_2$H$_2$N$_2$O$_2$S$_2$ (M+Na$^+$) 434.1766, found 434.1767.

**Ethyl (2S,3S)-(+)-3-ethyl-2-((thiophen-3-yl)-piperidine-4-carboxylate (29b):**

To a solution of 27b (100 mg, 0.242 mmol) in ether (2.5 ml) was slowly added ethyl magnesium bromide (4.9 ml, 4.9 mmol, 1.0 M in ether) at 0 °C. After the mixture was stirred for 20 min, H$_2$O and a 1 N HCl solution were carefully added, and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO$_4$, filtered and concentrated in vacuo to give the crude 28b.

To a solution of crude piperidine and n-propylamine (0.18 ml, 2.19 mmol) in chloroform (12 ml) was added lead tetraacetate (431 mg, 0.97 mmol) at room temperature. After the mixture was stirred for 28 h, a saturated aqueous NaHCO$_3$ solution was added, and the resulting mixture was extracted with chloroform. The organic layers were combined, washed with water, dried over MgSO$_4$, filtered and concentrated in vacuo to give the crude products. Column chromatography on silica gel (from 0% to 1.3% methanol in chloroform) gave 29b (53 mg, 75% for 2 steps) in a 3:8 : 1 (0.79 : 0.21) mixture of C$_2$β and C$_2$α stereoisomers as a yellow amorphous solid: IR (KBr disk, cm$^{-1}$) 3424, 2964, 2932, 2361, 1719, 1157, 756; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.24-7.29 (m, 1H), 7.05-7.10 (m, 1H), 7.00-7.04 (m, 1H), 4.08-4.26 (m, 2.21H), 3.57 (d, 0.79H, J = 10.3 Hz), 3.06-3.20 (m, 1H), 2.86-2.99 (m, 0.42H), 2.75 (ddd, 0.79H, $J = 11.4, 11.4, 3.7$ Hz), 2.44 (ddd, 0.79H, $J = 11.2, 11.2, 4.8$ Hz), 1.74-1.94 (m, 3H), 1.06-1.34 (m, 5H) 0.78 (t, 3H, $J = 7.6$ Hz), 0.68 (t, 3H, $J = 7.6$ Hz); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 174.4, 144.9, 59.9, 56.7, 44.9, 42.0, 40.2, 28.0, 22.8, 14.2, 11.6; ESI HRMS m/z calcld for C$_4$H$_7$N$_2$O$_2$S$_2$ (M+Na$^+$) 398.2096, found 398.2105.

**Ethyl (15,2R)-cis-1-[(2S)-4-Acetyl-2-phenyl-1,2,5,6-tetrahydropyridin-1-yl]-7-isopropylindan-2-ol (27c):**

To a solution of 27d (155 mg, 0.369 mmol) in dichloromethane (1.8 ml) were added ethyl vinyl ether (0.176 ml, 1.843 ml) and PPTS (9 mg, 0.0369 mmol) at room temperature. After the mixture was stirred at reflux for 20 h, a saturated aqueous NH$_4$Cl solution was added, and the resulting mixture was extracted with chloroform. The organic layers were combined, washed with brine, dried over MgSO$_4$, filtered and concentrated in vacuo to give the ethoxyethyl protected alcohol product (153 mg, 85%) as a yellow amorphous solid.

To a solution of ethoxyethyl protected alcohol (634 mg, 1.286 mmol) in THF (12.8 ml) was added methyl magnesium chloride (1.72 ml, 5.147 mmol) at 0 °C. After the mixture was stirred at 0 °C for 30 min, then added to H$_2$O, the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO$_4$, filtered and concentrated in vacuo to give the ketone product (539 mg, 94%) as a white amorphous solid.

To a solution of the ketone (124 mg, 0.277 mmol) in MeOH (2.8 ml) was added a 2 N HCl solution (2.8 ml) at room temperature. After the mixture was stirred at room temperature for 28 h, a saturated aqueous NaHCO$_3$ solution was added, and the resulting mixture was extracted with chloroform. The organic layers were combined, washed with brine, dried over MgSO$_4$, filtered and concentrated in vacuo to give 27c (100 mg, 96%) as a white amorphous solid: IR (KBr disk, cm$^{-1}$) 3457, 2960, 2940, 1650, 1267, 1088; $^1$H NMR (400 MHz, CDCl$_3$, 50 °C) $\delta$ 7.00-7.14 (m, 1H), 6.80-6.93 (br s, 1H), 6.58-6.67 (br s, 1H), 4.77-5.40 (br s, 1H), 4.42-4.60 (br s, 1H), 4.28 (d, 1H, $J = 6.6$ Hz), 2.85-3.12 (br s, 3H), 2.26-2.80 (br m, 4H), 2.25 (s, 3H), 1.00-1.03 (br m, 3H), 0.80-0.87 (br s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$, 50 °C) $\delta$ 198.4, 147.6, 141.7, 140.9, 136.8, 136.7, 129.3, 128.9, 128.4, 127.9, 123.4, 122.0, 62.7, 41.1, 29.0, 25.0, 24.9, 24.3, 23.3, 23.0, 15.2; ESI HRMS m/z calcld for C$_{25}$H$_{26}$N$_2$O$_2$ (M+Na$^+$) 398.2096, found 398.2105.

**(2S,3S)-4-Acetyl-3-ethyl-2-phenyl-piperidine (29c), and 2-[(2S)-2-phenyl,1,2,5,6-tetrahydro-benzothiazine-4-yl]-butan-2-ol (30c):**
To a solution of 27c (100 mg, 0.266 mmol) in ether (2.6 ml) was slowly added ethyl magnesium bromide (5.3 ml, 5.3 mmol, 1.0 M in ether) at 0 °C. After the mixture was stirred for 20 min, water was carefully added followed by 1 N hydrochloric acid, and extracted with ethyl acetate. The organic layers were washed with brine, dried over MgSO₄, filtered and concentrated in vacuo to give the crude product.

To a solution of the crude piperidine and n-propylamine (0.20 ml, 2.40 mmol) in chloroform (2.6 ml) was added lead tetraacetate (472 mg, 1.07 mmol) at -50 °C. After the mixture was stirred for 15 min, it was added to an ice-1N aqueous sodium hydroxide solution. The resulting mixture was filtered, and extracted with chloroform. The organic layers were combined, washed with water, dried over MgSO₄, filtered and concentrated in vacuo to give the crude products. Column chromatography on silica gel (from 0% to 1.2% methanol in chloroform) gave 29c (53 mg, 41% for 2 steps) in a 2.2 : 1 (0.69 : 0.31) mixture of C2β and C2α stereoisomers as a yellow oil and 30c (16 mg, 26%) in a 1:1 mixture of stereoisomers as a yellow oil. Data for 29c: IR (KBr disk, cm⁻¹) 2960, 2360, 2340, 1709, 1354, 756; ¹H NMR (400 MHz, CDCl₃) δ 7.21-7.40 (m, 5H), 4.21 (d, 0.31H, J = 9.6 Hz), 3.39 (d, 0.69H, J = 10.1 Hz), 3.09-3.24 (m, 0.1H), 2.84-2.93 (m, 0.62H), 2.59 (ddd, 0.69H, J = 11.9, 11.7, 2.5 Hz), 2.20 (q, 3H, 0.68), 1.66-2.05 (m, 3H), 0.68 (t, 0.93H, J = 7.6 Hz), 0.62 (t, 2.07H, J = 7.6 Hz); major isomer ¹³C NMR (100 MHz, CDCl₃) δ 211.6, 142.7, 128.4, 127.9, 127.6, 65.1, 54.6, 46.7, 43.2, 29.9, 28.4, 22.5, 9.6; minor isomer ¹³C NMR (100 MHz, CDCl₃) δ 211.2, 143.6, 143.6, 128.3, 127.8, 127.2, 61.4, 45.4, 41.9, 30.5, 27.6, 21.7, 11.9; ESI HRMS m/z calcd for C₁₃H₁₇N₂O₂ (M+H)⁺ 232.1701, found 232.1690. Data for 30c: IR (KBr disk, cm⁻¹) 3420, 2940, 2930, 1650, 1456, 760; ¹H NMR (400 MHz, CDCl₃) δ 7.36-7.22 (m, 5H), 5.78-5.72 (br m, 1H), 4.55-4.51 (br m, 1H), 3.19-3.09 (br m, 1H), 2.97-2.88 (br m, 1H), 2.30-2.12 (m, 2H), 1.68-1.58 (m, 2H), 1.34-1.30 (m, 3H), 0.91-0.82 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 7143.1, 142.4, 142.3, 128.5, 127.9, 127.9, 127.5, 127.0, 122.1, 121.7, 75.0, 58.4, 58.3, 42.0, 41.7, 32.9, 32.8, 27.1, 27.0, 24.9, 24.7, 8.3, 8.1; ESI HRMS m/z calcd for C₁₃H₂₇N₂O₄ (M+H)⁺ 232.1701, found 232.1700.

(2S)-1-[[1S,2R]-2-hydroxy-7-isopropylindan-1-yl]-N-methoxy-N-methyl-2-phenyl-1,2,5,6-tetrahydropyridine-4-carboxyamid e (27d):

To a suspension of Weinreb amide (100 mg, 0.372 mmol) and molecular sieve 4A (372 mg) in DMF (2.0 ml) was added cis-1-amino-7-isopropylindan-2-ol (9) (75 mg, 0.390 mmol) at room temperature, and then the mixture was stirred for 10 min at this temperature. After the mixture was added lithium chloride (39 mg, 0.929 mmol), tri(2-furyl)phosphine (28 mg, 0.199 mmol) and tris(dibenzylideneacetone)dipalladium(0) (27 mg, 0.029 mmol) at room temperature, the mixture was stirred for 10 min at this temperature then a solution of phenylvinylstannane (219 mg, 0.558 mmol) in DMF (0.6 ml) was added to this suspension. After the reaction mixture was stirred at 80 °C for 3 h, a 10% aqueous NH₃ solution was added, and the resulting mixture was extracted with ether. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated in vacuo to give the crude aminocetal products. Column chromatography on silica gel (from 33% to 50% ethyl acetate in hexane) gave the aminocetal (136 mg, 87%) as a yellow foam: IR (neat, cm⁻¹) 2960, 2360, 1620, 1161, 1021, 747; ¹H NMR (400 MHz, CDCl₃) δ 7.30-7.43 (m, 5H), 7.14 (dd, 1H, J = 7.6, 7.6 Hz), 6.94-7.01 (m, 2H), 6.16 (d, 1H, J = 1.6 Hz), 4.95-4.98 (m, 1H), 4.89 (d, 1H, J = 5.7 Hz), 4.46 (d, 1H, J = 5.7 Hz), 4.07-4.04 (m, 1H), 3.62 (3H), 3.22 (3H), 3.02-3.19 (m, 2H), 2.79-2.83 (m, 1H), 2.65-2.75 (m, 1H), 2.61 (q, 1H, J = 6.9, 6.6 Hz), 0.99 (d, 3H, J = 6.6 Hz), 0.56 (d, 3H, J = 6.9 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 169.4, 143.1, 142.1, 136.3, 132.0, 129.3, 128.7, 128.3, 127.8, 126.4, 123.4, 121.6, 86.5, 75.0, 74.3, 61.5, 61.2, 39.6, 33.8, 28.0, 26.7, 23.4, 22.8; ESI HRMS m/z calcd for C₂₆H₃₂N₂O₃ (M+Na)⁺ 441.2154, found 441.2153.

To a solution of the aminocetal (136 mg, 0.323 mmol) in acetonitrile (3.2 ml) was added sodium cyanoborohydride (101 mg, 1.617 mmol) at 0 °C. After the mixture was stirred at 0 °C for 1 h, H₂O was added, and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated in vacuo to give the alcohol product 27d (118 mg, 87%) as yellow amorphous solid: IR (neat, cm⁻¹) 2960, 2360, 1618, 1382, 1087, 703; ¹H NMR (400 MHz, CDCl₃, 50 °C) δ 7.00-7.51 (m, 7H), 6.74-6.92 (br m, 1H), 6.07-6.15 (br s, 1H), 4.38-4.57 (br s, 1H), 4.28 (d, 1H, J = 6.6 Hz), 3.58-3.65 (m, 4H), 2.86-3.30 (br m, 7H), 2.30-2.86 (br m, 3H), 1.16-1.32 (br s, 3H), 0.82-1.10 (br s, 3H); ¹³C NMR (100 MHz, CDCl₃, 50 °C) δ 170.5, 147.6, 141.4, 136.9, 134.3, 129.1, 129.0, 128.7, 128.3, 127.7, 127.3, 122.1, 70.1, 63.8, 62.3, 61.2, 41.0, 33.6, 32.1, 28.0, 24.5, 22.9; ESI HRMS m/z calcd for C₂₆H₃₂N₂O₃ (M+Na)⁺ 443.2311, found 443.2307.
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![Chemical structure diagram]

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Filename = 13C N-methyl isomer-1
Author = delta
Experiment = single_pulse_dec
Sample_id = S#498967
Solvent = CHLOROFORM-D
Creation_time = 18-FEB-2008 14:02:42
Revision_time = 18-FEB-2008 14:06:07
Current_Time = 24-AUG-2010 18:52:50
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX400M
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 115.0
Total_scans = 115.0
X_90_width = 9.6[us]
X_acq_time = 1.04333312[s]
X_angle = 45[deg]
X_atn = 7.8[db]
X_pulse = 4.8[us]
Irr_atn_dec = 21.4[db]
Irr_atn_noe = 21.4[db]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 5[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.04333312[s]
Temp_get = 24.2[DC]
Filename = 13C alcohol isomer-2.
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 23-APR-2010 13:53:58
Revision_time = 24-AUG-2010 18:57:19
Current_Time = 24-AUG-2010 18:58:27
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX400M
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.9584665[Hs]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 304
Total_scans = 304
X_90_width = 9.2[us]
X_acq_time = 1.04333312[s]
X_angle = 45[deg]
X_atn = 6.6[dB]
X_pulse = 4.6[us]
Irr_atn_dec = 22.2[dB]
Irr_atn_noe = 22.2[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 5[s]
Recvr_gain = 58
Relaxation_delay = 5[s]
Repetition_time = 6.04333312[s]
Temp_get = 24.6[dC]
Filename = 1H Aldehyde isomer-3.
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 26-APR-2010 08:25:08
Revision_time = 24-AUG-2010 19:06:04
Current_Time = 24-AUG-2010 19:06:46
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX400M
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 4.36731904[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 4[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 0.22897343[Hz]
X_sweep = 7.5030012[kHz]
X_90_width = 10.5[us]
X_acq_time = 4.36731904[s]
X_angle = 45[deg]
X_atn = 1.4[dB]
X_pulse = 5.25[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 32
Relaxation_delay = 1[s]
Repetition_time = 5.36731904[s]
Temp_get = 23.4[°C]
Filename = 13C Aldehyde isomer-2
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 26-APR-2010 08:50:57
Revision_time = 24-AUG-2010 19:08:13
Current_Time = 24-AUG-2010 19:08:50
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX400M
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 221
Total_scans = 221
X_90_width = 9.2[us]
X_acq_time = 1.04333312[s]
X_angle = 45[deg]
X_atn = 4.6[db]
X_pulse = 4.6[us]
Irr_atn_dec = 22.2[db]
Irr_atn_noe = 22.2[db]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 5[s]
Recvr_gain = 54
Relaxation_delay = 5[s]
Repetition_time = 6.04333312[s]
Temp_get = 24[dC]
Filename = 1H alcohol-3.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 27-FEB-2008 12:22:16
Revision_time = 29-AUG-2010 17:31:53
Current_time = 25-AUG-2010 17:32:29
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX400M
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 4.36731904[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 32768
X_prescans = 1
X_resolution = 0.22897343[Hz]
X_sweep = 7.5030012[kHz]
Cliped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_angle = 45[deg]
X_atn = 2.8[dB]
X_pulse = 5.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 20
Relaxation_delay = 5[s]
Repetition_time = 9.36731904[s]
Temp_get = 22.5[dC]
Filename = 1H aldehyde-3.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 3-MAR-2008 12:26:44
Revision_time = 25-AUG-2010 17:35:15
Current_Time = 25-AUG-2010 17:35:26
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX400M
Spectrometer = DELTA2 NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 4.36731904[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 0[ppm]
X_points = 12288
X_prescans = 1
X_resolution = 0.22897343[Hz]
X_sweep = 7.5030012[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 11.2[us]
X_acq_time = 4.36731904[s]
X_angle = 45[deg]
X_atn = 3.8[dB]
X_pulse = 5.6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 24
Relaxation_delay = 5[s]
Repetition_time = 9.36731904[s]
Temp_get = 23.2[dC]
Filename = 13C ketone-1.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 54391142
Solvent = CHLOROFORM-D
Creation_time = 1-FEB-2008 11:28:12
Revision_time = 1-FEB-2008 11:30:27
Current_Time = 25-AUG-2010 17:50:56
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECK400M
Spectrometer = DELTA2 NMR
Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 354
Total_scans = 354
X_90_width = 9.6[us]
X_acq_time = 1.04333312[s]
X_angle = 45[deg]
X_atn = 7.8[dB]
X_pulse = 4.8[us]
Irr_atn_dec = 21.4[dB]
Irr_atn_noe = 21.4[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 5[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.04333312[s]
Temp_get = 24.2[dC]