Catalytic Asymmetric Formation of β -Sultams

Supporting Information

Marian Zajac, and René Peters*

*Laboratorium für Organische Chemie, ETH Zürich, Hönggerberg HCI E 111, Wolfgang-Pauli-Str. 10,
CH-8093 Zürich, Switzerland*

peters@org.chem.ethz.ch

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Experimental

Except as otherwise indicated, all reactions were carried out in oven dried glassware under a positive pressure of nitrogen or argon. Dichloromethane, toluene and THF were purified by distillation and dried by a passage through activated alumina under nitrogen atmosphere. For work-up procedures and flash-chromatography, distilled technical grade-solvents were used. Diisopropylethylamine (Acros) was distilled over CaH_2 . Ethyl glyoxylate (Fluka, 50% solution in toluene) was fractionally distilled under reduced pressure from P_2O_5 . Imines **6a**^[1,2] and **6b**,^[3] sulfonylchloride **3d**^[4] and the modified cinchona alkaloids **4c**^[5] (MeQ), **4d**^[6] (BnQ), **4e**^[7] (TMSQ), and **4g**^[8] (Q₂PYR) were prepared according to know literature procedures. All other laboratory chemicals were purchased from *ABCR*, *Acros*, *Aldrich*, *Fluka*, *J.T. Baker* or *Merck* and were used without purification.

Unless otherwise indicated, all liquids were added *via* syringe, solids were added neat against an argon flow. For long-time additions of reagents, syringe pumps *Thermo Orion M361* and *kdScientific* were used in combination with Hamilton glass syringes. Solvents were removed at a heating-bath temperature of 40 °C and 600 - 30 mbar pressure by rotary evaporation. Non-volatile compounds were dried *in vacuo* at 0.01 mbar over P_2O_5 . Except as otherwise indicated, yields refer to purified compounds and are calculated in mol% of the used starting material. Reactions were magnetically stirred and monitored by thin layer-chromatography (TLC) using silica gel plates from *Merck* (silica gel 60 F₂₅₄). Visualization occurred by fluorescence quenching under UV light and by staining with aqueous KMnO_4 / NaOH solution. Purification by flash chromatography was performed on silica gel 60 Å, 32-62, provided by *Fluka*, using a forced flow of eluent at 0.2-0.4 bar pressure air.

NMR-spectra were recorded on a *Varian Gemini 300* and a *Varian Mercury 300* spectrometer operating at 300 MHz (¹H) and 75 MHz (¹³C) or by the NMR service of the Laboratory of

Organic Chemistry at ETHZ on a *Bruker DRX400* spectrometer operating at 400 MHz (^1H) and 100 MHz (^{13}C). Chemical shifts δ are referred in terms of ppm and J -coupling constants are given in Hz. Abbreviations for multiplicity are as follows: *s* (singlet), *d* (doublet), *t* (triplet), *q* (quadruplet), *m* (multiplet), *b* (broad signal). IR-spectra were recorded on a *Perkin Elmer Spectrum One FT-IR* with an *Universal ATR Sampling Accessory* and the signals are given by wave numbers (cm^{-1}). Optical rotation was measured on a *Jasco DIP-100 digital Polarimeter* operating at the sodium D line with a 100 mm path length cell. Melting points were measured using a *Büchi 535* melting point apparatus in open glass capillaries and are uncorrected. Mass spectra were obtained from the ETH Zürich MS Service. High resolution EI mass spectra were performed on a *Micromass AutoSpec Ultima* and were calibrated with perfluorotributylamine (PFTBA) prior to data acquisition. High resolution ESI mass spectra were performed on an *Ion Spec Ultima 2 FTICR*, HI-RES MALDI spectra were recorded using an *Ion Spec Ultima HR FT-ICR MS MALDI-FT-ICR MS* employing DCTB or 3-hydroxy-2-pyridinecarboxylic acid as matrix. Combustion analysis was performed by the Mikroelementaranalytisches Laboratorium at ETH Zürich. The single crystal X-ray structure analysis was performed by Paul Seiler at ETH Zürich.

General procedure for the formation of 4-alkyl-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidine-1,1-dioxides (2a-f, GP1)

A Schlenk tube was charged with imine **6a** (50 mg, 0.166 mmol, 1.0 equiv.) and the corresponding cinchona alkaloid derivative **4** (0.017 mmol, 0.1 equiv.), evacuated and filled with nitrogen. A solution was prepared by addition of dry DCM (1 mL) which was stirred for 5 min at room temperature and subsequently cooled to $-80\text{ }^\circ\text{C}$. After 10 min, DIPEA (diisopropylethylamine, 72 μL , 0.414 mmol, 2.5 equiv.) was added dropwise, followed after additional 5 min by the indicated sulfonyl chloride **3a-f** (0.332 mmol, 2.0 equiv., neat or as a solution in DCM (0.5 mL, for sulfonylchlorides **3d** and **3e**)). The reaction mixture was stirred at $-80\text{ }^\circ\text{C}$ for 20 hours. After this time, the reaction was quenched with 0.1 M aqueous HCl (1 mL) and the mixture was warmed up to room temperature, diluted with ethylacetate (10 mL) and washed with 0.1 M HCl (10 mL) and brine (10 mL). The organic phase was dried over Na_2SO_4 , filtered and evaporated *in vacuo* to give the corresponding crude product. The yields were determined by $^1\text{H-NMR}$ using nitromethane as internal standard.

(3*R*,4*R*)-4-Ethyl-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidine-1,1-dioxide (2a)

β -Sultam **2a** was prepared according to GP1, using cinchonidine (**4b**, 6 mg, 0.017 mmol, 0.1 equiv.) as catalyst and *n*-propylsulfonylchloride **3a** (37 μL , 0.332 mmol, 2.0 equiv.). The product

was obtained as slightly yellow solid (0.129 mmol, yield: 78%, *ee* = 81%, *dr* = 19:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min). An analytical sample was obtained by crystallization from cyclohexane/ethylacetate 1:1.

C₁₂H₁₄Cl₃NO₄S₂, MW: 406.72 g/mol. **Mp**: 184.9-185.6 °C. **[α]_D^{24.6°C}** (c = 1.485, CHCl₃) = +56.3 ± 0.3 (sample with *ee* = 82% after recrystallization from ethylacetate / cyclohexane 1:1). **¹H NMR (400 MHz, CDCl₃, 22 °C)**: δ = 7.92 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.37 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 4.77 (*d*, *J* = 9.1, 1 H, CH-CCl₃); 4.68 (*ddd*, *J* = 12.4, 9.1, 3.6, 1 H, CH-CH₂); 2.63-2.51 (*m*, 1 H, CH₂); 2.47 (*s*, 3 H, CH₃-Ph); 2.31-2.21 (*m*, 1 H, CH₂); 1.17 (*t*, *J* = 7.3, 3 H, CH₂-CH₃). **¹³C NMR (100 MHz, CDCl₃, 22 °C)**: δ = 145.8; 134.5; 129.8; 128.6; 95.3; 80.8; 65.3; 21.8; 18.6; 13.3. **IR (ATR, neat)**: ν = 2955, 2936, 1372, 1362, 1199, 1176, 1157, 1085, 1015, 932, 802. **HRMS (EI)** *m/z*: Calc. for [M - CCl₃]⁺: 288.0359. Found: 288.0356 (18.7%). **Anal. Calcd. for C₁₂H₁₄Cl₃NO₄S₂**: C, 35.44; H, 3.47; N, 3.44. Found: C, 35.57; H, 3.57; N, 3.41.

(3*R*,4*R*)-4-Propyl-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidinedi-1,1-dioxide (2b)

β-Sultam **2b** was prepared according to GP1, using quinine **4a** (6 mg, 0.017 mmol, 0.1 equiv.) as catalyst and *n*-butylsulfonylchloride **3b** (43 μL, 0.332 mmol, 2.0 equiv.). The product was obtained as slightly yellow solid (0.134 mmol, yield: 81%, *ee* = 91%, *dr* = 18:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 97:3 *n*-hexane/*i*PrOH, 1.0 mL/min). An analytical sample was obtained by crystallization from cyclohexane/ethylacetate (1:1).

C₁₃H₁₆Cl₃NO₄S₂, MW: 420.76 g/mol. **Mp**: 162.0-162.5 °C. $[\alpha]_D^{23.9^\circ C}$ (c = 1.015, CHCl₃) = +89.0 ± 0.2 (sample with *ee* = 99% after recrystallization from ethylacetate / cyclohexane 1:1). **¹H NMR (400 MHz, CDCl₃, 22 °C)**: δ = 7.92 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.37 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 4.79-4.70 (*m*, 2 H, CH₂-CH-CH-CCl₃); 2.56-2.46 (*m*, 1 H, CH₂); 2.47 (*s*, 3 H, CH₃-Ph); 2.22-2.13 (*m*, 1 H, CH₂); 1.65-1.54 (*m*, 1 H, CH₂); 1.52-1.39 (*m*, 1 H, CH₂); 1.01 (*t*, *J* = 7.3, 3 H, CH₂-CH₃). **¹³C NMR (100 MHz, CDCl₃, 22 °C)**: δ = 145.8; 134.6; 129.8; 128.6; 95.3; 79.0; 65.2; 26.6; 22.3; 21.8; 13.3. **IR (ATR, neat)**: ν = 2990, 2971, 1465, 1357, 1342, 1194, 1169, 1158, 1086, 1035, 950, 803. **HRMS (EI) *m/z***: Calc. for [M – CCl₃]⁺: 302.0515. Found: 302.0514 (19.9%). **Anal. Calcd. for C₁₃H₁₆Cl₃NO₄S₂**: C, 37.14; H, 3.83; N, 3.33. Found: C, 37.32; H, 4.01; N, 3.24.

(3*R*,4*R*)-4-(2-Chloroethyl)-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidine-1,1-dioxide (2c)

β-Sultam **2c** was prepared according to GP1, using quinine **4a** (6 mg, 0.017 mmol, 0.1 equiv.) as catalyst and 3-chloropropylsulfonylchloride **3c** (40 μL, 0.332 mmol, 2.0 equiv.). The product was obtained as slightly yellow solid (0.129 mmol, yield: 78%, *ee* = 94%, *dr* = 11:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 85:15 *n*-hexane/EtOH, 1.0 mL/min). An analytical sample was obtained by crystallization from cyclohexane/ethylacetate (1:1).

C₁₂H₁₃Cl₄NO₄S₂, MW: 441.18 g/mol. **Mp**: 178.0-178.4 °C. $[\alpha]_D^{24.4^\circ C}$ (c = 0.610, CHCl₃) = +97.3 ± 0.3 (sample with *ee* = 99% after recrystallization from ethylacetate / cyclohexane 1:1). **¹H NMR (400 MHz, CDCl₃, 22 °C)**: δ = 7.93 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.39 (*d*, *J* = 8.4, 2 H, CH_{Ph});

5.05 (*ddd*, $J = 11.4, 9.1, 3.0$, 1 H, CH-CH_2); 4.82 (*d*, $J = 9.1$, 1 H, CH-CCl_3); 3.81-3.76 (*m*, 1 H, $\text{CH}_2\text{-Cl}$); 3.61-3.55 (*m*, 1 H, $\text{CH}_2\text{-Cl}$); 3.08-2.99 (*m*, 1 H, CH_2); 2.78-2.69 (*m*, 1 H, CH_2); 2.48 (*s*, 3 H, $\text{CH}_3\text{-Ph}$). ^{13}C NMR (100 MHz, CDCl_3 , 22 °C): $\delta = 146.1$; 134.1; 129.9; 128.7; 95.0; 74.9; 64.7; 41.9; 27.9; 21.8. IR (ATR, neat): $\nu = 2933, 1367, 1357, 1348, 1181, 1192, 1163, 1084, 1025, 930, 829, 813, 799$. HRMS (EI) m/z : Calc. for $[\text{M}]^+$: 438.9035. Found: 438.9038 (0.3%). Anal. Calcd. for $\text{C}_{12}\text{H}_{13}\text{Cl}_4\text{NO}_4\text{S}_2$: C, 32.67; H, 2.97; N, 3.17. Found: C, 32.90; H, 3.01; N, 3.15.

(3*R*,4*R*)-4-(Phenylmethyl)-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidine-1,1-dioxide (2d)

β -Sultam **2d** was prepared according to GP1, using quinine **4a** (6 mg, 0.017 mmol, 0.1 equiv.) as catalyst and 2-phenylethylsulfonylchloride **3d** (68 mg, 0.332 mmol, 2.0 equiv.) as solution in DCM (0.5 mL). The product was obtained as slightly yellow solid (0.156 mmol, yield: 94%, *ee* = 94%, *dr* = 21:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min). An analytical sample was obtained by crystallization from 1:1 cyclohexane/ethylacetate mixture.

$\text{C}_{17}\text{H}_{16}\text{Cl}_3\text{NO}_4\text{S}_2$, MW: 468.80 g/mol. Mp: 174.8-175.5 °C. $[\alpha]_D^{25.3^\circ\text{C}}$ ($c = 1.295$, CHCl_3) = +78.3 $\pm$ 0.2 (sample with *ee* = 99% after recrystallization from ethylacetate / cyclohexane 1:1). ^1H NMR (400 MHz, CDCl_3 , 22 °C): $\delta = 7.92$ (*d*, $J = 8.4$, 2 H, CH_{Ph}); 7.38-7.16 (*m*, 7 H, CH_{Ph}); 4.99 (*ddd*, $J = 12.0, 9.1, 3.1$, 1 H, CH-CH_2); 4.84 (*d*, $J = 9.1$, 1 H, CH-CCl_3); 3.79 (*dd*, $J = 14.9, 12.0$, 1 H, $\text{CH}_2\text{-Ph}$); 3.61 (*dd*, $J = 14.9, 3.1$, 1 H, $\text{CH}_2\text{-Ph}$); 2.47 (*s*, 3 H, $\text{CH}_3\text{-Ph}$). ^{13}C NMR (100 MHz, CDCl_3 , 22 °C): $\delta = 145.9$; 135.5; 134.4; 129.9; 129.3; 129.3; 128.7; 95.0; 80.3; 65.3;

30.8; 21.8. **IR (ATR, neat):** ν = 3034, 2932, 1498, 1459, 1440, 1372, 1360, 1347, 1204, 1191, 1177, 1159, 1082, 1023, 908, 820, 812, 793. **HRMS (ESI) m/z :** Calc. for $[\text{MNa}]^+$: 489.9479. Found: 489.9486. **Anal. Calcd. for $\text{C}_{17}\text{H}_{16}\text{Cl}_3\text{NO}_4\text{S}_2$:** C, 43.55; H, 3.44; N, 2.99. Found: C, 43.54; H, 3.52; N, 2.96.

(3*R*,4*R*)-4-(2-(4-Methoxyphenoxy)-ethyl)-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidine-1,1-dioxide (2e)

β -Sultam **2e** was prepared according to GP1, using quinine **4a** (6 mg, 0.017 mmol, 0.1 equiv.) as catalyst and 2-(4-methoxyphenoxy)-propylsulfonylechloride **3e** (88 mg, 0.332 mmol, 2.0 equiv.) as solution in DCM (0.5 mL). The product was obtained as slightly yellow solid (0.158 mmol, yield: 95%, *ee* = 87%, *dr* = 13:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 65:35 *n*-hexane/EtOH, 1.0 mL/min). An analytical sample was obtained by crystallization from cyclohexane/ethylacetate (1:1).

$\text{C}_{19}\text{H}_{20}\text{Cl}_3\text{NO}_6\text{S}_2$, MW: 528.86 g/mol. **Mp:** 199.0-199.4 °C. **$[\alpha]_D^{24.7^\circ\text{C}}$** (*c* = 0.970, CHCl_3) = -48.1 ± 0.9 (sample with *ee* = 90% after recrystallization from ethylacetate / cyclohexane 1:1).

^1H NMR (400 MHz, CDCl_3 , 22 °C): δ = 7.94 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.37 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 6.86-6.80 (*m*, 4 H, CH_{Ph}); 5.02 (*ddd*, *J* = 11.8, 9.2, 2.6, 1 H, CH-CH_2); 4.81 (*d*, *J* = 9.2, 1 H, CH-CCl_3); 4.10-4.05 (*m*, 1 H, $\text{CH}_2\text{-O}$); 4.01-3.96 (*m*, 1 H, $\text{CH}_2\text{-O}$); 3.76 (*s*, 3 H, $\text{CH}_3\text{-O}$); 3.05-2.96 (*m*, 1 H, CH_2); 2.81-2.73 (*m*, 1 H, CH_2); 2.47 (*s*, 3 H, $\text{CH}_3\text{-Ph}$). **^{13}C NMR (100 MHz, CDCl_3 , 22 °C):** δ = 154.5; 152.1; 145.8; 134.5; 129.9; 128.6; 115.6; 114.7; 95.4; 76.3; 65.6; 64.8; 55.7; 25.1; 21.8. **IR (ATR, neat):** ν = 2950, 2935, 1509, 1376, 1365, 1230, 1193, 1169,

1053, 1030, 936, 810. **HRMS (ESI) m/z :** Calc. for $[MNa]^+$: 549.9690. Found: 549.9701. A satisfactory microanalysis could not be obtained due to solvent inclusion.

(3*S*,4*S*)-4-Methyl-2-(*p*-toluenesulfonyl)-3-(trichloromethyl)-1,2-thiazetidine-1,1-dioxide (2f)

β -Sultam **2f** was prepared according to GP1, using Q₂PYR **4g** (15 mg, 0.017 mmol, 0.1 equiv.) as catalyst and ethylsulfonylchloride **3f** (29 μ L, 0.332 mmol, 2.0 equiv.). The product was obtained as colorless solid (0.133 mmol, yield: 80%, *ee* = 80%, *dr* = 17:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 80:20 *n*-hexane/EtOH, 1.0 mL/min). An analytical sample was obtained by crystallization from 1:1 cyclohexane/ethylacetate. The assignment of the (*S,S*)-configuration of **2f** is based on a comparison of the CD spectra of **2f** and **2b**. The *syn*-configuration of **2f** was determined by ¹H-NMR using NOE-experiments.

C₁₁H₁₂Cl₃NO₄S₂, **MW:** 392.71 g/mol. **Mp:** 178.5-179.3 °C. **$[\alpha]_D^{23.4^\circ C}$** (*c* = 0.450, CHCl₃) = -48.3 ± 0.7 (sample with *ee* = 98% after recrystallization from ethylacetate / cyclohexane 1:1).

¹H NMR (400 MHz, CDCl₃, 22 °C): δ = 7.93 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.37 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 4.87 (*qd*, *J* = 9.1, 7.4, 1 H, CH-CH₃); 4.78 (*d*, *J* = 9.1, 1 H, CH-CCl₃); 2.47 (*s*, 3 H, CH₃-Ph); 1.87 (*d*, *J* = 7.4, 3 H, CH-CH₃). **¹³C NMR (100 MHz, CDCl₃, 22 °C):** δ = 145.9; 134.4; 129.8; 128.7; 95.2; 73.0; 64.8; 21.8; 9.7. **IR (ATR, neat):** ν = 2963, 1364, 1204, 1178, 1163, 1039, 1001, 926, 828, 805, 785. **HRMS (EI) m/z :** Calc. for $[M - CCl_3]^+$: 274.0202. Found: 274.0201 (18.8%). **Anal. Calcd. for C₁₁H₁₂Cl₃NO₄S₂:** C, 33.64; H, 3.08; N, 3.57. Found: C, 33.46; H, 3.03; N, 3.55.

General procedure for the formation of 3-alkyl-3-octylsulfamoyl-2-(*p*-toluenesulfonylamino)propionic acid ethyl esters (**14a-d**, GP2)

In a Schlenk tube, a solution of quinine **4a** (6 mg, 0.020 mmol, 0.1 equiv.) in dry DCM (0.5 mL) was cooled to -80°C . After 10 min, DIPEA (diisopropylethylamine, 85 μL , 0.490 mmol, 2.5 equiv.) was added dropwise, followed after additional 5 min by the requisite sulfonylchloride **3a-d** (0.392 mmol, 2.0 equiv., neat or as a solution in DCM (0.25 mL) in the case of sulfonylchloride **3d**). To this mixture, imine **6b** (50 mg, 0.196 mmol, 1 equiv.) was added as a solution in DCM (0.5 mL) over a period of 10 hours using a syringe pump. After complete addition the reaction mixture was stirred at -80°C for additional 10 hours and finally *n*-octylamine (65 μL , 0.392 mmol, 2.0 equiv.) was added at the same temperature. The mixture was allowed to warm up to room temperature and was stirred for additional 4 hours. It was subsequently diluted with ethylacetate (10 mL) and washed successively with 0.1 M aqueous HCl (10 mL) and brine (10 mL). The organic phase was dried over Na_2SO_4 , filtered and evaporated *in vacuo*. The crude product was purified by flash-chromatography on silica gel (ethylacetate/cyclohexane,1:6).

(2*R*,3*R*)-3-Octylsulfamoyl-2-(*p*-toluenesulfonylamino)pentanoic acid ethyl ester (**14a**)

Sulfonamide **14a** was prepared according to GP2, using *n*-propylsulfonylchloride **3a** (44 μ L, 0.392 mmol, 2.0 equiv.). The product was obtained as colorless solid (53 mg, 0.108 mmol, yield: 55%, *ee* = 80%, *dr* = 11:1). The *ee* values were determined by HPLC (Chiralcel OD-H 25+15 cm, 98:2 *n*-hexane/EtOH, 0.7 mL/min).

C₂₂H₃₈N₂O₆S₂, MW: 490.69 g/mol. **Mp**: 124.8-125.3 °C. $[\alpha]_D^{26.4^\circ C}$ (*c* = 0.865, CHCl₃) = +10.4 $\pm$ 0.7 (sample with *ee* = 80%). **¹H NMR (300 MHz, CDCl₃, 22 °C)**: δ = 7.73 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.28 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 5.74 (*d*, *J* = 9.6, 1 H, Ts-NH-CH); 4.39 (*t*, *J* = 5.9, 1 H, NH-CH₂); 4.27 (*dd*, *J* = 9.6, 2.8, 1 H, CH-NHTs); 4.08-3.86 (*m*, 2 H, CH₂-O); 3.55-3.48 (*m*, 1 H, CH-CH₂); 3.04 (*q*, *J* = 6.8, 2 H, CH₂-NH); 2.41 (*s*, 3 H, CH₃-Ph); 2.06-1.82 (*m*, 2 H, CH₂-CH); 1.59-1.44 (*m*, 2 H, CH₂-CH₂NH); 1.26 (*bs*, 10 H, CH₂(Octyl)); 1.09 (*m*, 6 H, 2xCH₃); 0.87 (*t*, *J* = 7.2, 3 H, CH₃(Octyl)). **¹³C NMR (75 MHz, CDCl₃, 22 °C)**: δ = 169.6; 144.0; 137.0; 129.8; 127.5; 66.0; 62.7; 54.6; 43.9; 31.9; 30.6; 29.3; 26.7; 22.8; 21.7; 20.1; 14.2; 13.9; 11.6. **IR (ATR, neat)**: ν = 3308, 2928, 2855, 1749, 1716, 1460, 1437, 1336, 1318, 1290, 1166, 1132, 1093, 1081, 1032, 912, 811. **HRMS (MALDI) *m/z***: Calc. for [MH]⁺: 491.2244. Found: 491.2243. A satisfactory microanalysis could not be obtained due to solvent inclusion.

(2*R*,3*R*)-3-Octylsulfamoyl-2-(*p*-toluenesulfonylamino)hexanoic acid ethyl ester
(14b)

Sulfonamide **14b** was prepared according to GP2, using *n*-butylsulfonylchloride **3b** (52 μ L, 0.392 mmol, 2.0 equiv.). The product was obtained as colorless solid (50 mg, 0.100 mmol, yield: 51%, *ee* = 86%, *dr* = 11:1). The *ee* values were determined by HPLC (Chiralcel OD-H 25+15 cm, 95:5 *n*-hexane/EtOH, 0.7 mL/min).

C₂₃H₄₀N₂O₆S₂, MW: 504.70 g/mol. **Mp**: 130.1-130.8 °C. $[\alpha]_D^{26.7^\circ\text{C}}$ (c = 0.860, CHCl₃) = +9.3 ± 0.4 (sample with *ee* = 86%). **¹H NMR (300 MHz, CDCl₃, 22 °C)**: δ = 7.73 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.29 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 5.73 (*d*, *J* = 9.6, 1 H, Ts-NH-CH); 4.28 (*t*, *J* = 5.9, 1 H, NH-CH₂); 4.23 (*dd*, *J* = 9.6, 2.8, 1 H, CH-NHTs); 4.10-3.90 (*m*, 2 H, CH₂-O); 3.64-3.58 (*m*, 1 H, CH-CH₂); 3.05 (*q*, *J* = 6.8, 2 H, CH₂-NH); 2.42 (*s*, 3 H, CH₃-Ph); 1.90-1.80 (*m*, 2 H, CH₂-CH); 1.60-1.44 (*m*, 2 H, CH₂-CH₂NH); 1.46-1.32 (*m*, 2 H, CH₂-CH₃); 1.27 (*b*, 10 H, CH₂(Octyl)); 1.10 (*t*, *J* = 7.2, 3 H, CH₃-CH₂O); 0.93 (*t*, *J* = 7.2, 3 H, CH₃); 0.87 (*t*, *J* = 6.8, 3 H, CH₃(Octyl)). **¹³C NMR (75 MHz, CDCl₃, 22 °C)**: δ = 169.4; 143.7; 136.7; 129.5; 127.2; 63.8; 62.4; 54.7; 43.6; 31.7; 30.3; 29.0; 28.3; 26.4; 22.5; 21.4; 19.9; 14.0; 13.6; 13.5. **IR (ATR, neat)**: ν = 3305, 2925, 2855, 1749, 1716, 1440, 1327, 1306, 1286, 1165, 1135, 1092, 1032, 811. **HRMS (MALDI) *m/z***: Calc. for [MH]⁺: 527.2220. Found: 527.2215. **Anal. Calcd. for C₂₃H₄₀N₂O₆S₂**: C, 54.73; H, 7.99; N, 5.55. Found: C, 54.99; H, 7.87; N, 5.57.

(2*R*, 3*R*)-3-Octylsulfamoyl-4-phenyl-2-(*p*-toluenesulfonylamino)butyric acid ethyl ester (14d)

Sulfonamide **14d** was prepared according to GP2, using 2-phenylethylsulfonylchloride **3d** (81 mg, 0.392 mmol, 2.0 equiv.) as a solution in DCM (0.25 mL). After *n*-octylamine addition, the reaction mixture was stirred for only 1.5 hours. The product was obtained as colorless solid (43 mg, 0.078 mmol, yield: 40%, *ee* = 78%, *dr* = 17:1). The *ee* values were determined by HPLC (Chiralcel AD-H 25 cm, 90:10 *n*-hexane/EtOH, 1.0 mL/min).

C₂₇H₄₀NO₆S₂, MW: 552.76 g/mol. **Mp**: 98.6-99.5 °C. $[\alpha]_D^{26.9^\circ\text{C}}$ (c = 0.585, CHCl₃) = +31.9 ± 0.5 (sample with *ee* = 78%). **¹H NMR (300 MHz, CDCl₃, 22 °C)**: δ = 7.70 (*d*, *J* = 8.1, 2 H, CH_{Ph}); 7.38-7.25 (*m*, 7H, CH_{Ph}); 5.67 (*d*, *J* = 9.3, 1 H, Ts-NH-CH); 4.19 (*dd*, *J* = 9.3, 2.5, 1 H, CH-NHTs); 4.05-3.88 (*m*, 2 H, CH₂-O, NH-CH₂); 3.72 (*m*, 1 H, CH-CH₂); 3.29 (*dd*, *J* = 14.3, 6.2, 1 H, CH₂-Ph); 3.17 (*dd*, *J* = 14.3, 8.1, 1 H, CH₂-Ph); 3.06-2.94 (*m*, 1 H, CH₂-NH); 2.87-2.76 (*m*, 1 H, CH₂-NH); 2.41 (*s*, 3 H, CH₃-Ph); 1.43-1.29 (*m*, 2 H, CH₂-CH₂NH); 1.24 (*b*, 10 H, CH₂(Octyl)); 1.07 (*t*, *J* = 6.8, 3 H, CH₃-CH₂O); 0.88 (*t*, *J* = 6.8, 3 H, CH₃(Octyl)). **¹³C NMR (75 MHz, CDCl₃, 22 °C)**: δ = 169.2; 144.0; 136.9; 136.8; 129.8; 129.4; 129.2; 127.6; 65.3; 62.8; 55.6; 43.7; 33.3; 31.9; 30.3; 29.3; 26.6; 22.8; 21.7; 14.3; 13.9. **IR (ATR, neat)**: ν = 3336, 2925, 2854, 1721, 1715, 1443, 1408, 1328, 1278, 1165, 1149, 1092, 1028. **HRMS (ESI) *m/z***: Calc. for [MNa]⁺: 575.2220. Found: 575.2219. **Anal. Calcd. for C₂₇H₄₀NO₆S₂**: C, 58.67; H, 7.29; N, 5.07. Found: C, 58.38; H, 7.22; N, 4.95.

(2*R*, 3*R*)-1,1,1-Trichloro-2-(*p*-toluenesulfonylamino)pentane-3-sulfonic acid phenethyl ester (9)

To a stirred solution of β-sultam **2a** (50 mg, 0.123 mmol, 1.0 equiv., crystallized from ethylacetate / cyclohexane 1:1, *ee* = 85%, *dr* > 100:1) in DCM (0.5 mL) at room temperature, 2-phenylethylalcohol (29 μL, 0.246 mmol, 2.0 equiv.) and triethylamine (17 μL, 0.246 mmol, 1.0 equiv.) were added as a solution in DCM (0.5 mL) and the solution was stirred for 14 hours. After this time, the mixture was diluted with ethylacetate (10 mL) and washed with 0.1 M aqueous HCl (10 mL) and brine (10 mL). The organic phase was dried over Na₂SO₄, filtered and evaporated *in vacuo*. The crude product was purified by flash chromatography on silica gel

(ethylacetate/cyclohexane, 1:4) to furnish the pure product **9** as colorless solid (55 mg, 0.105 mmol, yield: 85%, *ee* = 83%, *dr* > 100:1). The *ee* values were determined by HPLC (Chiralcel AD-H, 93:7 *n*-hexane/EtOH, 1.0 mL/min).

C₂₀H₂₄Cl₃NO₅S₂, MW: 528.90 g/mol. **Mp**: 145.3-145.7 °C. $[\alpha]_D^{26.1^\circ\text{C}}$ (*c* = 0.285, CHCl₃) = +19.3 ± 1.0 (sample with *ee* = 90% after recrystallization from ethylacetate / cyclohexane 1:1). **¹H NMR (300 MHz, CDCl₃, 22 °C)**: δ = 7.81 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.35-7.22 (*m*, 7 H, CH_{Ph}); 5.32 (*d*, *J* = 10.6, 1 H, NH); 5.16 (*dd*, *J* = 10.6, 1.2, 1 H, CH-NH); 4.49 (*t*, *J* = 7.2, 2 H, CH₂-O); 3.64 (*m*, 1 H, CH-CH₂); 3.09 (*t*, *J* = 7.2, 2 H, CH₂-Ph); 2.42 (*s*, 3 H, CH₃-Ph); 1.92-1.80 (*m*, 2 H, CH₂-CH); 1.09 (*t*, *J* = 7.5, 3 H, CH₃). **¹³C NMR (75 MHz, CDCl₃, 22 °C)**: δ = 143.8; 137.7; 136.0; 129.4; 128.9; 128.6; 127.2; 127.0; 101.2; 70.3; 66.8; 63.8; 35.8; 21.7; 21.2; 12.8. **IR (ATR, neat)**: ν = 3291, 2972, 2926, 1456, 1340, 1318, 1233, 1151, 1140, 1094, 951, 928, 902, 853. **HRMS (MALDI) *m/z***: Calc. for [MNa]⁺: 550.0054. Found: 550.0053. **Anal. Calcd. for C₂₀H₂₄Cl₃NO₅S₂**: C, 45.42; H, 4.57; N, 2.65. Found: C, 45.70; H, 4.75; N, 2.57.

(1*R*,2*R*)-N-[2-Benzylsulfamoyl-1-(trichloromethyl)-butyl]-*p*-toluenesulfonamide (10**)**

To a stirred solution of β-sultam **2a** (50 mg, 0.123 mmol, 1.0 equiv., ethylacetate / cyclohexane 1:1, *ee* = 85%, *dr* > 100:1) in DCM (0.5 mL) at room temperature, benzylamine (13 μL, 0.123 mmol, 1.0 equiv.) was added as a solution in DCM (0.5 mL) and the solution was stirred for 14 hours. The solvent was then evaporated *in vacuo* to furnish the pure product **10** as colorless solid (61 mg, 0.118 mmol, yield: 96%, *ee* = 85%, *dr* > 100:1). The *ee* values were determined by HPLC (Chiralcel AD-H, 93:7 *n*-hexane/EtOH, 1.0 mL/min).

C₁₉H₂₃Cl₃N₂O₄S₂, MW: 513.89 g/mol. **Mp**: 171.0-171.3 °C. $[\alpha]_D^{25.9^\circ C}$ (c = 0.595, CHCl₃) = -26.7 ± 0.6 (sample with *ee* = 80%). **¹H NMR (300 MHz, CDCl₃, 22 °C)**: δ = 7.78 (*d*, *J* = 8.4, 2 H, CH_{Ph}); 7.43-7.25 (*m*, 7H, CH_{Ph}); 5.52 (*dd*, *J* = 6.8, 5.0, 1 H, CH-NHTs); 5.32 (*d*, *J* = 10.6, 1 H, NH); 5.10 (*d*, *J* = 10.0, 1 H, NH); 4.56 (*dd*, *J* = 14.3, 7.2, 1 H, CH₂-Ph); 4.40 (*dd*, *J* = 14.3, 4.7, 1 H, CH₂-Ph); 3.70 (*m*, 1 H, CH-CH₂); 2.42 (*s*, 3 H, CH₃-Ph); 2.05-1.93 (*m*, 2 H, CH₂-CH); 1.27 (*t*, *J* = 7.5, 3 H, CH₃-CH₂). **¹³C NMR (75 MHz, CDCl₃, 22 °C)**: δ = 144.2; 137.4; 136.8; 129.5; 128.7; 127.8; 127.4; 101.0; 67.7; 66.3; 48.1; 21.7; 20.9; 13.3. **IR (ATR, neat)**: ν = 3299, 3239, 2963, 2923, 1454, 1322, 1159, 1132, 1089, 1073, 923. **HRMS (MALDI) *m/z***: Calc. for [MNa]⁺: 535.0057. Found: 535.0056. **Anal. Calcd. for C₁₉H₂₃Cl₃N₂O₄S₂**: C, 44.41; H, 4.51; N, 5.45. Found: C, 44.27; H, 4.60; N, 5.38.

(1*R*,2*R*)-N-[2-(Pyrrolidine-1-sulfonyl)-1-(trichloromethyl)butyl]-*p*-toluenesulfonamide (11)

To a stirred solution of β-sultam **2a** (100 mg, 0.246 mmol, 1.0 equiv., crystallized from ethylacetate / cyclohexane 1:1, *ee* = 85%, *dr* > 100:1) in DCM (1 mL) at room temperature, pyrrolidine (21 μL, 0.246 mmol, 1.0 equiv.) was added as a solution in DCM (0.5 mL) and the solution was stirred for 14 hours. The solvent was then evaporated *in vacuo* to furnish the pure product **11** as colorless solid (114 mg, 0.239 mmol, yield: 97%, *ee* = 85%, *dr* > 100:1). The *ee* values were determined by HPLC (Chiralcel AD-H, 93:7 *n*-hexane/EtOH, 1.0 mL/min).

C₁₆H₂₃Cl₃N₂O₄S₂, MW: 477.85 g/mol. **Mp**: 170.5-171.1 °C. $[\alpha]_D^{25.6^\circ C}$ (c = 1.510, CHCl₃) = +37.1 ± 0.2 (sample with *ee* = 80%). **¹H NMR (300 MHz, CDCl₃, 22 °C)**: δ = 7.85 (*d*, *J* = 8.1, 2

H, CH_{Ph}); 7.28 (*d*, $J = 8.1$, 2 H, CH_{Ph}); 5.37 (*d*, $J = 10.3$, 1 H, NH); 5.06 (*dd*, $J = 10.3$, 0.9, 1 H, CH-NH); 3.57-3.43 (*m*, 5 H, CH-CH₂, CH₂-N); 2.42 (*s*, 3 H, CH₃-Ph); 1.99-1.88 (*m*, 4 H, CH₂CH₂-N); 1.86-1.76 (*m*, 2 H, CH₂-CH); 1.14 (*t*, $J = 7.5$, 3 H, CH₃). ¹³C NMR (75 MHz, CDCl₃, 22 °C): $\delta = 143.9$; 138.4; 129.6; 127.5; 102.2; 66.9; 64.3; 48.8; 26.2; 21.8; 20.5; 13.5. IR (ATR, neat): $\nu = 3258$, 2972, 2883, 1459, 1325, 1314, 1159, 1133, 1091, 1013, 984, 915. HRMS (MALDI) *m/z*: Calc. for [MNa]⁺: 499.0057. Found: 499.0052. Anal. Calcd. for C₁₆H₂₃Cl₃N₂O₄S₂: C, 40.22; H, 4.85; N, 5.86. Found: C, 40.49; H, 4.91 N, 5.88.

(1*R*,2*R*)-N-[1-(Dichloromethyl)-2-(pyrrolidine-1-sulfonyl)-butyl]-*p*-toluenesulfonamide (12)

To a stirred solution of sulfonamide **11** (40 mg, 0.084 mmol, 1.0 equiv., *ee* = 80%, *dr* > 100:1) in THF (1 mL) under argon at room temperature, tributyltin hydride (68 μ L, 0.251 mmol, 3.0 equiv.) was added as a solution in THF (1 mL). The mixture was subsequently stirred at 60 °C for 14 hours. After this time, the solvent was removed *in vacuo*, the residue was dissolved in acetonitrile (6 mL) and washed three times with *n*-hexane (10 mL) to remove all tin derivatives. The crude product was purified by flash chromatography on silica gel (ethylacetate/cyclohexane, 1:4) to furnish the pure product **12** as colorless oil (26 mg, 0.059 mmol, yield: 70%).

C₁₆H₂₄Cl₂N₂O₄S₂, MW: 442.06 g/mol. $[\alpha]_D^{27.0^\circ\text{C}}$ (*c* = 0.730, CHCl₃) = -2.8 ± 0.3 (sample with *ee* = 80%). ¹H NMR (300 MHz, CDCl₃, 22 °C): $\delta = 7.79$ (*d*, $J = 8.2$, 2 H, CH_{Ph}); 7.32 (*d*, $J = 8.2$, 2 H, CH_{Ph}); 6.24 (*d*, $J = 3.0$, 1 H, CHCl₂); 5.25 (*d*, $J = 8.8$, 1 H, NH); 4.29 (*ddd*, $J = 8.8$, 4.4, 3.0, 1 H, CH-NH); 3.44-3.28 (*m*, 4 H, CH₂-N); 3.24-3.18 (*m*, 1 H, CH-CH₂); 2.43 (*s*, 3 H, CH₃-Ph); 2.09-1.89 (*m*, 2 H, CH₂-CH); 1.94-1.90 (*m*, 4 H, CH₂CH₂-N); 1.14 (*t*, $J = 7.4$, 3 H, CH₃). ¹³C

NMR (75 MHz, CDCl₃, 22 °C): δ = 144.0; 136.7; 129.5; 127.5; 73.8; 64.9; 59.8; 48.4; 25.9; 21.5; 20.1; 13.1. **IR (ATR, neat):** ν = 3280, 2970, 2882, 1450, 1318, 1197, 1158, 1140, 1090, 1069, 1011, 910. **HRMS (ESI) m/z :** Calc. for [MNa]⁺: 465.0447. Found: 465.0443. A satisfactory microanalysis could not be obtained due to solvent inclusion.

(1*R*,2*R*)-N-[1-(Chloromethyl)-2-(pyrrolidine-1-sulfonyl)-butyl]-*p*-toluenesulfonamide (13)

To a reaction flask charged with AIBN (7 mg, 0.042 mmol, 0.5 equiv.), a solution of sulfonamide **11** (40 mg, 0.084 mmol, 1.0 equiv., *ee* = 80%, *dr* > 100:1) in toluene (4 mL) and a solution of tributyltin hydride (135 μ L, 0.502 mmol, 6.0 equiv.) in toluene (1 mL) were successively added under argon at room temperature. The mixture was stirred at 60 °C for 4 hours. After this time, the solvent was removed *in vacuo*, the residue was dissolved in acetonitrile (10 mL) and washed three times with *n*-hexane (20 mL) to remove all tin derivatives. The crude product was purified by flash-chromatography on silica gel (1:5 ethylacetate/cyclohexane) to furnish the pure product **13** as colorless oil (22 mg, 0.054 mmol, yield: 64%).

C₁₆H₂₅ClN₂O₄S₂, MW: 408.96 g/mol. $[\alpha]_D^{25.3^\circ\text{C}}$ (*c* = 0.400, CHCl₃) = -17.7 ± 0.6 (sample with *ee* = 80%). **¹H NMR (300 MHz, CDCl₃, 22 °C):** δ = 7.79 (*d*, *J* = 8.1, 2 H, CH_{Ph}); 7.32 (*d*, *J* = 8.1, 2 H, CH_{Ph}); 5.75 (*d*, *J* = 8.1, 1 H, NH); 3.87-3.77 (*m*, 2 H, CH-NH, CH₂Cl); 3.67-3.59 (*m*, 1 H, CH₂Cl); 3.36-3.20 (*m*, 5 H, CH₂-N); 2.43 (*s*, 3 H, CH₃-Ph); 1.94-1.84 (*m*, 5 H, CH₂CH₂-N, CHH-CH); 1.80-1.65 (*m*, 1 H, CHH-CH); 1.08 (*t*, *J* = 7.5, 3 H, CH₃). **¹³C NMR (75 MHz, CDCl₃, 22 °C):** δ = 144.0; 137.2; 129.8; 127.7; 65.1; 55.5; 48.1; 43.5; 26.1; 21.7; 20.4; 12.3. **IR**

(ATR, neat): ν = 3314, 2970, 2930, 2878, 1450, 1426, 1335, 1319, 1306, 1284, 1198, 1160, 1141, 1122, 1092, 1070, 1015, 960, 940, 908. HRMS (ESI) m/z : Calc. for $[\text{MNa}]^+$: 431.0837. Found: 431.0836. Anal. Calcd. for $\text{C}_{16}\text{H}_{25}\text{ClN}_2\text{O}_4\text{S}_2$: C, 46.99; H, 6.16; N, 6.85. Found: C, 46.69; H, 6.57; N, 6.51.

(1*R*,2*R*)-N-[2-Octylsulfamoyl-1-(trichloromethyl)-propyl]-*p*-toluenesulfonamide (15)

Sulfonamide **15** was prepared by modified GP1 using Q₂PYR **4g** (15 mg, 0.017 mmol, 0.1 equiv.) and ethylsulfonylchloride **3f** (29 μL , 0.332 mmol, 2.0 equiv.). After 20 hours at -80°C , *n*-octylamine (55 μL , 0.332 mmol, 2.0 equiv.) was added dropwise. The reaction mixture was allowed to warm up to room temperature and was stirred for additional 4 hours. Prior to workup, the mixture was diluted with ethylacetate (10 mL) and then washed with aqueous 0.1 M HCl (10 mL) and brine (10 mL). The organic phase was dried over Na_2SO_4 , filtered and evaporated *in vacuo*. The crude product was purified by flash chromatography on silica gel (ethylacetate/hexane, 1:7). The product was obtained as colorless oil (64 mg, 0.123 mmol, yield: 74%, *ee* = 80%, *dr* = 17:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 95:5 *n*-hexane/*i*PrOH, 1.0 mL/min).

$\text{C}_{19}\text{H}_{31}\text{Cl}_3\text{N}_2\text{O}_4\text{S}_2$, MW: 521.96 g/mol. $[\alpha]_D^{24.8^\circ\text{C}}$ (c = 0.250, CHCl_3) = -27.7 ± 0.6 (sample with *ee* = 80%). ^1H NMR (300 MHz, CDCl_3 , 22°C): δ = 7.80 (*d*, J = 8.1, 2 H, CH_{Ph}); 7.30 (*d*, J = 8.1, 2 H, CH_{Ph}); 5.16 (*m*, 2 H, *NH*); 5.05 (*d*, J = 10.6, 1 H, *CH-NHTs*); 3.94 (*q*, J = 7.2, 1 H, *CH-CH*₃); 3.37-3.15 (*m*, 2 H, *CH*₂-*NH*); 2.42 (*s*, 3 H, *CH*₃-Ph); 1.60 (*d*, J = 7.2, 3 H, *CH*₃); 1.65-1.50

(*m*, 2 H, CH₂-CH₂NH); 1.27 (*b*, 10 H, CH₂(*octyl*)); 0.87 (*t*, *J* = 6.8, 3 H, CH₃(*octyl*)). ¹³C NMR (75 MHz, CDCl₃, 22 °C): δ = 144.2; 136.8; 129.5; 127.4; 101.1; 67.2; 59.5; 44.8; 31.8; 31.1; 29.2; 26.5; 22.7; 21.7; 14.2; 11.6. IR (ATR, neat): ν = 3282, 2954, 2925, 2855, 1447, 1329, 1306, 1159, 1140, 1091, 1063 1018, 905. HRMS (ESI) *m/z*: Calc. for [MNa]⁺: 543.0683. Found: 543.0684. Anal. Calcd. for C₁₉H₃₁Cl₃N₂O₄S₂: C, 43.72; H, 5.99; N, 5.37. Found: C, 43.99; H, 6.05; N, 5.44.

Deuteration experiment

A Schlenk tube was charged with quinine **4a** (6 mg, 0.017 mmol, 0.05 equiv.), evacuated and filled with nitrogen. A solution was prepared by addition of dry DCM (1 mL) which was then cooled to –80 °C. After 10 min, DIPEA (diisopropylethylamine, 72 μL, 0.414 mmol, 1.25 equiv.) and MeOD (41 μL, 0.996 mmol, 3.0 equiv.) were added dropwise, followed after additional 5 min by *n*-propylsulfonylchloride **3a** (37 μL, 0.332 mmol, 1.0 equiv.). The reaction mixture was stirred at –80 °C for 20 hours. After this time, the reaction was quenched with 0.1 M aqueous HCl (1 mL) and the mixture was warmed up to room temperature, diluted with ethylacetate (10 mL) and washed with 0.1 M HCl (10 mL) and brine (10 mL). The organic phase was dried over Na₂SO₄, filtered and evaporated *in vacuo*. Sulfonylchloride **3a** was recovered unchanged and only traces of α-deuterated ester were detected.

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Current Data Parameters
 NAME g1342
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 PROCNO 1

Zajac/Peters MZ112 OPR:PZ
 400 MHz 1H-NMR

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 DW 78.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.20 usec
 PL1 -4.00 dB
 SFO1 400.1325608 MHz

F2 - Processing parameters
 SI 65536
 SF 400.130082 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
 CX 40.00 cm
 PIP 9.500 ppm
 F1 3861.24 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.25000 ppm/cm
 HZCM 100.03250 Hz/cm

Current Data Parameters
 NAME g1342
 EXFNO 2
 PROCNO 1

Zajac/Peters MZ112 OPR:PZ
 100MHz 13C-BB-NMR

F2 - Acquisition Parameters
 Date_ 20061024
 Time 13.40
 INSTRUM DRX400
 PROBHD 5 mm BBO BB-
 PULPROG zgpg30
 TD 68804
 SOLVENT CDCl3
 NS 2000
 DS 0
 SWH 26246.719 Hz
 FIDRES 0.381471 Hz
 AQ 1.3107652 sec
 RG 8192
 DW 19.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 d12 0.60002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.60 usec
 PL1 1.00 dB
 SFO1 100.6241989 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -4.00 dB
 PL12 18.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127712 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
 CX 40.00 cm
 PIP 232.000 ppm
 F1 23342.16 Hz
 F2P -8.000 ppm
 F2 -804.91 Hz
 PPMCM 6.00000 ppm/cm
 HZCM 603.67670 Hz/cm

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

Mercury-300BB "gem4oc"

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

128 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 7 min, 2 sec

Current Data Parameters
NAME gl343
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061026
Time 9.04
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 68804
SOLVENT CDCl3
NS 1214
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 16384
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 40.00 cm
PIF 232.000 ppm
P1 23342.16 Hz
P2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cm

Zajac/Peters MZ125 OPR:FZ
100MHz 13C-BB-NMR

STANDARD 1H OBSERVE

Sample directory: MZ131

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

File: MZ131.1H

INOVA-500 "nmroc"

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

128 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 7 min, 2 sec

Current Data Parameters
NAME g1344
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061026
Time 10.24
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg45_cpd
TD 68804
SOLVENT CDCl3
NS 1200
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 11585.2
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127708 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FIP 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cm

Zajac/Peters MZ131 OPR:PZ
100MHz 13C-BB-NMR

STANDARD 1H OBSERVE

Sample directory: MZ145

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

File: MZ145.1H

INOVA-500 "nmroc"

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

128 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 7 min, 2 sec

Current Data Parameters
NAME g1346
EXPNO 2
PROCNO 1F2 - Acquisition Parameters
Date_ 20061030
Time 16.12
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 68804
SOLVENT CDCl₃
NS 14000
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 8192
EW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHzF2 - Processing parameters
SI 65536
SF 100.6127680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.401D NMR plot parameters
CX 40.00 cm
F1P 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cmZajac/Peters MZ145 OPR:PZ
100MHz 13C-BB-NMR

Current Data Parameters
NAME g1345
EXPNO 1
PROCNO 1

Zajac/Peters MZ152 OPR:PZ
400 MHz 1H-NMR

F2 - Acquisition Parameters
Date_ 20061026
Time 12.23
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 362
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 -4.00 dB
SFO1 400.1325608 MHz

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW DO
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.500 ppm
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.03250 Hz/cm

Current Data Parameters
NAME g1345
EXPNO 2
PROCNO 1

Zajac/Peters MZ152 OPR:PZ
100MHz 13C-BB-NMR

F2 - Acquisition Parameters
Date_ 20061026
Time 16.14
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg45_cpd
TD 68804
SOLVENT CDCl3
NS 6000
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 8192
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

ppm

154.50
152.06
145.82

134.53
129.87

115.61

95.42

77.01

64.81

55.72

25.10
21.77

-0.02

===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127693 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cm

Current Data Parameters
NAME g1211
EXPNO 1
PROCNO 1

Zajac/Peters MZ17 OPR:PZ
400 MHz 1H-NMR

F2 - Acquisition Parameters
Date_ 20051207
Time 10.26
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 287.4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 -4.00 dB
SFO1 400.1325608 MHz

F2 - Processing parameters
SI 65536
SF 400.1300085 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FIP 9.500 ppm
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.03250 Hz/cm

Current Data Parameters
NAME g1211
EXPNO 2
PROCNO 1

Zajac/Peters MZ17 OPR:PZ
100MHz 13C-BB-NMR

F2 - Acquisition Parameters
Date_ 20051207
Time 11.05
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg45 cpd
TD 68804
SOLVENT CDCl3
NS 1000
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 8192
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127693 MHz
WDW BM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FIP 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cm

Current Data Parameters
NAME F0480
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070111
Time 16.41
INSTRUM DRX500
PROBHD 5 mm BBI 1H-
PULPROG noemul
TD 90112
SOLVENT CDCl3
NS 320
DS 4
SWH 8090.615 Hz
FIDRES 0.089784 Hz
AQ 5.5689716 sec
RG 800
DN 61.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
D20 0.07000000 sec
L4 30
NUC1 1H
P1 7.10 usec
PL1 4.00 dB
SFO1 500.0332000 MHz
FQ2LIST f2list.2
NUC2 1H
PL2 120.00 dB
PL14 74.00 dB
SFO2 500.0332000 MHz

F2 - Processing parameters
SI 65536
SF 500.0300558 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
FIP 9.500 ppm
F1 4750.29 Hz
F2P -0.500 ppm
F2 -250.02 Hz
PFMCM 0.25000 ppm/cm
HZCM 125.00751 Hz/cm

Current Data Parameters
NAME F0480
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070111
Time 16.44
INSTRUM DRX500
PROBHD 5 mm BBI 1H-
PULPROG noemul
TD 90112
SOLVENT CDCl3
NS 320
DS 4
SWH 8090.615 Hz
FIDRES 0.089784 Hz
AQ 5.5689716 sec
RG 800
DN 61.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
D20 0.07000000 sec
L4 30
NUC1 1H
P1 7.10 usec
PL1 4.00 dB
SFO1 500.0332000 MHz
FQ2LIST f2list.3
NUC2 1H
PL2 120.00 dB
PL14 74.00 dB
SFO2 500.0332000 MHz

F2 - Processing parameters
SI 65536
SF 500.0300558 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
FIP 9.500 ppm
F1 4750.29 Hz
F2P -0.500 ppm
F2 -250.02 Hz
PFMCM 0.25000 ppm/cm
HZCM 125.00751 Hz/cm

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

Mercury-300BB "gem4oc"

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

16 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 52 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

File: MZ171-1.C13

GEMINI-300BB "gem3oc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

14184 repetitions

OBSERVE C13, 75.4560752 MHz

DECOUPLE H1, 300.0848347 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 9 hr, 59 min, 59 sec

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

GEMINI-300BB "gem3oc"

Pulse 29.5 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

32 repetitions

OBSERVE H1, 300.0836333 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 45 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

File: MZ172-1.c13

GEMINI-300BB "gem3oc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

14788 repetitions

OBSERVE C13, 75.3779672 MHz

DECOUPLE H1, 299.7740804 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 10 hr, 8 sec

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

Mercury-300BB "gem4oc"

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

128 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 7 min, 2 sec

13C OBSERVE

Sample directory: MZ173-3.C13

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

File: MZ173-3.C13

INOVA-500 "nmroc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

12888 repetitions

OBSERVE C13, 75.4560752 MHz

DECOUPLE H1, 300.0848347 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 9 hr, 59 min, 47 sec

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

Mercury-300BB "gem4oc"

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

64 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 3 min, 31 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

File: MZ154-4.C13

GEMINI-300BB "gem3oc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

14188 repetitions

OBSERVE C13, 75.4102323 MHz

DECOUPLE H1, 299.9024328 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 10 hr, 8 sec

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

GEMINI-300BB "gem3oc"

Pulse 29.5 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

64 repetitions

OBSERVE H1, 300.0836333 MHz

DATA PROCESSING

FT size 32768

Total time 3 min, 31 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

File: MZ153-4.C13

GEMINI-300BB "gem3oc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

14896 repetitions

OBSERVE C13, 75.4102323 MHz

DECOUPLE H1, 299.9024328 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 10 hr, 30 min, 4 sec

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

Mercury-300BB "gem4oc"

11

Pulse 18.0 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

128 repetitions

OBSERVE H1, 299.9012510 MHz

DATA PROCESSING

FT size 32768

Total time 7 min, 2 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

File: MZ155-4.c13

GEMINI-300BB "gem3oc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

12292 repetitions

OBSERVE C13, 75.4560752 MHz

DECOUPLE H1, 300.0848347 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 8 hr, 39 min, 58 sec

STANDARD 1H OBSERVE

Sample directory: MZ183-4CHR
File: /export/home/locnmr/vnmrsys/exp1/text

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
User: rzajac
Mercury-300BB "gem5oc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
64 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 19 sec

13C OBSERVE

Sample directory: MZ183-4CHR.C13

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
User: rzajac
File: MZ183-4CHR.C13
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
19720 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 13 hr, 20 min, 0 sec

STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

GEMINI-300BB "gem3oc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
128 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 7 min, 2 sec

13C OBSERVE

Sample directory: MZ184-12.C13

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: rzajac

File: MZ184-12.C13

INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
13968 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 9 hr, 59 min, 47 sec

STANDARD 1H OBSERVE

Sample directory: MZ48-87CHR

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

File: MZ48-87CHR.1H

INOVA-500 "nmroc"

Pulse 29.5 degrees

Acq. time 3.138 sec

Width 5099.4 Hz

32 repetitions

OBSERVE H1, 300.0836333 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 45 sec

¹³C OBSERVE

Sample directory: MZ48-87.C13

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: rzajac

File: MZ48-87.C13

INOVA-500 "nmroc"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 20000.0 Hz

9456 repetitions

OBSERVE C13, 75.4911544 MHz

DECOUPLE H1, 300.2242455 MHz

Power 35 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 65536

Total time 6 hr, 39 min, 50 sec

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ112.rac.ODH 80-20
 iPrOH 20min.met
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 80-20 iPrOH
 20min.met
 Acquired: 8/7/2006 6:28:15 PM
 Printed: 2/2/2007 4:36:30 PM

UV Results

Retention Time	Area	Area %	Height	Height %
11.270	30213612	50.15	1268432	55.55
13.347	30029833	49.85	1014854	44.45

Totals	60243445	100.00	2283286	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ112-77.ODH 80-20
 iPrOH 20min.met
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 80-20 iPrOH
 20min.met
 Acquired: 8/7/2006 6:49:35 PM
 Printed: 2/2/2007 4:37:05 PM

UV Results

Retention Time	Area	Area %	Height	Height %
11.213	105786134	90.63	4384551	91.98
13.390	10941758	9.37	382513	8.02

Totals	116727892	100.00	4767064	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ125.rac.ODH 97-3
 iPrOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 97-3 iPrOH
 60min.met
 Acquired: 12/22/2006 3:12:50 PM
 Printed: 2/2/2007 3:21:47 PM

UV Results

Retention Time	Area	Area %	Height	Height %
30.463	34366012	49.45	453835	51.28
34.153	35127954	50.55	431131	48.72

Totals	69493966	100.00	884966	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ125-11.ODH 97-3
 iPrOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 97-3 iPrOH
 60min.met
 Acquired: 5/17/2006 3:11:14 AM
 Printed: 2/2/2007 3:22:12 PM

UV Results

Retention Time	Area	Area %	Height	Height %
31.393	2807024	4.64	41061	5.81
34.813	57718131	95.36	665619	94.19

Totals	60525155	100.00	706680	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ131-1.rac.ODH 85-15
 EtOH 30min.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 85-15 EtOH
 30min.met
 Acquired: 8/24/2006 4:22:45 PM
 Printed: 2/2/2007 3:22:43 PM

UV Results

Retention Time	Area	Area %	Height	Height %
12.240	33789892	49.91	1348405	52.24
13.340	33918315	50.09	1232727	47.76

Totals	67708207	100.00	2581132	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ131-21.ODH 85-15
 EtOH 30min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 85-15 EtOH
 30min.met
 Acquired: 8/30/2006 1:53:56 PM
 Printed: 2/2/2007 3:23:04 PM

UV Results

Retention Time	Area	Area %	Height	Height %
12.913	399926	3.05	16080	3.53
14.037	12731043	96.95	439056	96.47

Totals	13130969	100.00	455136	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ145.rac.ODH 80-20
 iPrOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 80-20 iPrOH
 60min.met
 Acquired: 5/27/2006 3:55:14 PM
 Printed: 2/2/2007 3:24:01 PM

UV Results

Retention Time	Area	Area %	Height	Height %
16.017	20986713	49.76	452060	56.08
20.677	21186254	50.24	354012	43.92

Totals	42172967	100.00	806072	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ145-4.ODH 80-20
 iPrOH 30min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 80-20 iPrOH
 30min.met
 Acquired: 8/30/2006 11:41:30 AM
 Printed: 2/2/2007 3:24:34 PM

UV Results

Retention Time	Area	Area %	Height	Height %
16.380	26489694	97.00	601104	97.48
21.177	819652	3.00	15548	2.52

Totals	27309346	100.00	616652	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ152.rac.ODH 65-35
 EtOH 30min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 65-35 EtOH
 30min.met
 Acquired: 8/24/2006 2:14:04 PM
 Printed: 2/2/2007 3:25:05 PM

UV Results

Retention Time	Area	Area %	Height	Height %
11.007	36596375	50.06	1069601	61.82
17.050	36503362	49.94	660711	38.18

Totals	73099737	100.00	1730312	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ152-3.ODH 65-35 EtOH
 30min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 65-35 EtOH
 30min.met
 Acquired: 8/30/2006 2:52:49 PM
 Printed: 2/2/2007 3:25:27 PM

UV Results

Retention Time	Area	Area %	Height	Height %
11.400	46637440	93.42	1511646	95.80
17.977	3285140	6.58	66211	4.20

Totals	49922580	100.00	1577857	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ17.rac.ODH 80-20 EtOH
 30min.met
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 80-20 EtOH
 30min.met
 Acquired: 8/1/2006 10:35:20 PM
 Printed: 2/2/2007 3:15:18 PM

UV Results

Retention Time	Area	Area %	Height	Height %
16.627	8092648	49.98	220007	55.07
20.407	8098158	50.02	179529	44.93

Totals	16190806	100.00	399536	100.00
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2f

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ17-11.ODH 80-20 EtOH
 30min.met
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 80-20 EtOH
 30min.met
 Acquired: 8/1/2006 9:32:49 PM
 Printed: 2/2/2007 3:16:04 PM

UV Results

Retention Time	Area	Area %	Height	Height %
16.653	4321679	9.76	117880	11.89
20.273	39953682	90.24	873675	88.11

Totals	44275361	100.00	991555	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ171.rac.ODHx2 98-2 EtOH
 80min 0.7ml.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODHx2 98-2 EtOH 80min
 0.7ml.met
 Acquired: 1/23/2007 11:10:04 PM
 Printed: 2/2/2007 3:39:57 PM

UV Results

Retention Time	Area	Area %	Height	Height %
47.810	43731734	49.65	465735	61.80
55.673	44349874	50.35	287939	38.20

Totals	88081608	100.00	753674	100.00
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14a

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ171-2.ODHx2 98-2 EtOH
 80min 0.7ml.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODHx2 98-2 EtOH 80min
 0.7ml.met
 Acquired: 1/24/2007 12:31:24 AM
 Printed: 2/2/2007 3:40:26 PM

UV Results

Retention Time	Area	Area %	Height	Height %
47.780	58406644	89.80	611609	93.11
57.407	6634894	10.20	45255	6.89

Totals	65041538	100.00	656864	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ172.rac.ODHx2 95-5 EtOH
 30min 0.7ml.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODHx2 95-5 EtOH 30min
 0.7ml.met
 Acquired: 1/24/2007 4:49:32 PM
 Printed: 2/2/2007 3:40:58 PM

UV Results

Retention Time	Area	Area %	Height	Height %
20.290	50956542	49.63	1551822	62.06
22.750	51710787	50.37	948578	37.94

Totals	102667329	100.00	2500400	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ172-2.ODHx2 95-5 EtOH
 30min 0.7ml.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODHx2 95-5 EtOH 30min
 0.7ml.met
 Acquired: 1/25/2007 3:10:19 PM
 Printed: 2/2/2007 3:41:23 PM

UV Results

Retention Time	Area	Area %	Height	Height %
20.250	182974229	93.00	5174939	95.13
23.270	13771530	7.00	264719	4.87

Totals	196745759	100.00	5439658	100.00
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14b

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ173.rac.ADH 90-10 EtOH 80min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 90-10 EtOH 80min.met
 Acquired: 6/28/2006 11:55:02 AM
 Printed: 2/2/2007 3:42:08 PM

UV Results

Retention Time	Area	Area %	Height	Height %
23.033	133618638	48.09	2720808	54.91
31.090	144228605	51.91	2233779	45.09
Totals	277847243	100.00	4954587	100.00

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ173-3.ADH 90-10 EtOH 60m.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 90-10 EtOH 80min.met
 Acquired: 2/1/2007 9:25:24 PM
 Printed: 2/2/2007 3:42:37 PM

UV Results

Retention Time	Area	Area %	Height	Height %
22.730	46066050	88.76	932980	90.94
30.897	5832976	11.24	92928	9.06
Totals	51899026	100.00	1025908	100.00

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ154.rac.ADH 93-7 EtOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 93-7 EtOH 60min.met

Acquired: 9/11/2006 8:58:27 PM
 Printed: 2/2/2007 3:28:01 PM

UV Results

Retention Time	Area	Area %	Height	Height %
13.343	35074483	49.92	1141488	54.71
15.877	35185108	50.08	944793	45.29

Totals	70259591	100.00	2086281	100.00
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9

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ154-7.ADH 93-7 EtOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 93-7 EtOH 60min.met

Acquired: 9/13/2006 11:53:21 AM
 Printed: 2/2/2007 3:28:56 PM

UV Results

Retention Time	Area	Area %	Height	Height %
13.357	21608987	91.35	732233	92.69
15.970	2045644	8.65	57707	7.31

Totals	23654631	100.00	789940	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ153.rac.ADH 93-7 EtOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 93-7 EtOH 60min.met

Acquired: 9/11/2006 8:19:45 PM
 Printed: 2/2/2007 3:26:14 PM

UV Results

Retention Time	Area	Area %	Height	Height %
15.497	50186059	50.11	891068	69.54
22.193	49966543	49.89	390232	30.46

Totals	100152602	100.00	1281300	100.00
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10

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ153-6.ADH 93-7 EtOH 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 93-7 EtOH 60min.met

Acquired: 9/13/2006 10:52:07 AM
 Printed: 2/2/2007 3:27:20 PM

UV Results

Retention Time	Area	Area %	Height	Height %
15.677	23704058	92.34	394262	97.03
23.763	1967678	7.66	12067	2.97

Totals	25671736	100.00	406329	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ155.rac.ADH 93-7 EtOH
 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 93-7 EtOH 60min.met

Acquired: 9/11/2006 9:52:59 PM
 Printed: 2/2/2007 3:29:25 PM

UV Results

Retention Time	Area	Area %	Height	Height %
10.987	19867589	51.40	789033	55.29
12.887	18782914	48.60	638065	44.71

Totals	38650503	100.00	1427098	100.00
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11

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ155-6.ADH 93-7 EtOH
 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ADH 93-7 EtOH 60min.met

Acquired: 9/27/2006 4:39:42 PM
 Printed: 2/2/2007 3:30:13 PM

UV Results

Retention Time	Area	Area %	Height	Height %
10.567	207514554	92.71	7954468	93.36
12.660	16326928	7.29	566160	6.64

Totals	223841482	100.00	8520628	100.00
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Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ48.rac.ODH 95-5 iPrOH
 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 95-5 iPrOH
 60min.met
 Acquired: 1/31/2007 12:34:13 AM
 Printed: 2/2/2007 3:20:19 PM

UV Results

Retention Time	Area	Area %	Height	Height %
28.593	52680295	45.78	411518	50.73
32.427	62402700	54.22	399610	49.27

Totals	115082995	100.00	811128	100.00
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15

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\MZ48-69.ODH 95-5 iPrOH
 60min.met.dat
 Method: C:\EZChrom Elite\Enterprise\Projects\Sulfonamides\Data\paper\ODH 95-5 iPrOH
 60min.met
 Acquired: 5/17/2006 1:08:41 AM
 Printed: 2/2/2007 3:20:43 PM

UV Results

Retention Time	Area	Area %	Height	Height %
27.817	24686553	9.95	272317	12.61
31.200	223537894	90.05	1888039	87.39

Totals	248224447	100.00	2160356	100.00
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