Asymmetric Construction of 3-Vinylidene-Pyrrolidine Derivatives Containing Allene Moiety via Ag(I)/TF-BiphamPhos-Catalyzed 1,3-Dipolar Cycloaddition of Azomethine Ylides with Diethyl 2-(3,3-Diphenylpropa-1,2-dienylidene) Malonate

Zhi-Yong Xue, Xin Fang, and Chun-Jiang Wang*

E-mail: cjwang@whu.edu.cn

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General Remarks

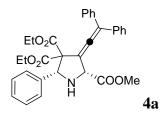
¹H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration). ¹³C NMR spectra were recorded on a VARIAN Mercury 100 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Diastereomeric ratios were determined from crude ¹H NMR or HPLC analysis. Enantiomeric ratios were determined by HPLC, using a chiralcel AD-H column, a chiralpak AS-H column with hexane and *i*-PrOH as solvents. The absoluted configurations of the products were determined as (2*R*,5*R*) by X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results. Catalyst **1a-e¹** and diethyl 2-(3,3-diphenylpropa-1,2-dienylidene) malonate **2**² were prepared according to the literature.

General Procedure for the synthesis of *a*-imino esters

To a suspension of the corresponding amino acid ester hydrochloride (12 mmol) and MgSO₄ (16.0 mmol) in CH₂Cl₂ (20 mL) was added Et₃N (2.0 mL, 14 mmol). The mixture was stirred at room temperature for 1h, and then the corresponding aldehyde (10.0 mmol) was added. The reaction was stirred at room temperature overnight, and then the resulting precipitate was removed by filtration. The filtrate was washed once with water (30 mL), the aqueous phase was extracted once with CH₂Cl₂(15 mL) and the combined organic phase was washed with brine 3 times, dried over MgSO₄ and concentrated. The resulting iminoesters were used in 1,3-dipolar cycloadditions without further purification.

General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with diethyl 2-(3,3-diphenylpropa-1,2-dienylidene)malonate Catalyzed by Ag(I)/TF-BiphamPhos 1e Complex

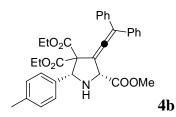
Under argon atmosphere (*S*)-TF-BiphamPhos **1e** (6.0 mg, 0.0075 mmol) and AgOAc (1.2 mg, 0.007 mmol) were dissolved in 2mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.40 mmol), K_2CO_3 (0.04 mmol) and diethyl 2-(3,3-diphenylpropa-1,2-dienylidene)malonate (0.23 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.



(2R,5R)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-phenylpyrrolidine-

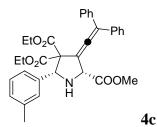
2,4,4-tricarboxylate

i-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.06 and 6.05 min.



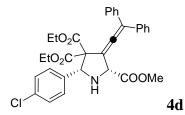
(2*R*,5*R*)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-p-tolylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 92% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}_{D} = -71.2$ (*c* 1.5, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.34-7.32 (m, 12H), 7.16-7.13 (m, 2H), 5.25 (s, 1H), 4.76 (s, 1H), 4.10-4.07 (m, 1H), 3.99-3.96 (m, 1H), 3.88-3.83 (m, 1H), 3.74-3.63 (m, 1H), 3.54 (s, 3H), 2.33 (s, 3H), 0.96-0.81 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.39, 13.51, 21.02, 29.60, 52.34, 61.43, 61.86, 68.24, 68.61, 107.31, 116.19, 126.79, 127.64, 127.79, 128.13, 128.25, 128.40, 128.80, 132.86, 135.45, 135.61, 137.71, 167.87, 168.39, 170.25, 200.44; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₃H₃₃NO₆+H⁺: 540.2381, found 540.2394. The product was analyzed to determine the enantioselectivity of the reaction: 90% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 10.27 and 15.85 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-m-tolylpyrrolidine-2,4,4- tricarboxylate

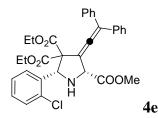
The title compound was prepared according to the general procedure as described above in 90% yield. It was purified by flash chromatography to afford yellow oil. [α]²⁵_D = -53.5 (*c* 0.7, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.34-7.19 (m, 13H), 7.12-7.09 (m, 1H), 5.25 (s, 1H), 4.77 (s, 1H), 4.13-4.07 (m, 1H), 3.99-3.93 (m, 1H), 3.88-3.82 (m, 1H), 3.72-3.62 (m, 1H), 3.55 (s, 3H), 2.35 (s, 3H), 0.96-0.87 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 12.40, 13.53, 21.42, 52.41, 61.45, 61.91, 68.33, 68.56, 107.16, 116.32, 124.16, 127.49, 127.69, 127.82, 128.05, 128.14, 128.30, 128.43, 128.86, 135.41, 135.58, 135.73, 137.68, 167.87, 168.31, 170.22, 200.52; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 696 cm⁻¹. HRMS Calcd. For C₃₃H₃₃NO₆+H⁺: 540.2381, found 540.2388.. The product was analyzed to determine the enantioselectivity of the reaction: 92% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.93 and 13.18 min.



(2R,5R)-4,4-diethyl 2-methyl 5-(4-chlorophenyl)-3-(2,2-diphenylvinylidene)

pyrrolidine-2,4,4-tricarboxylate

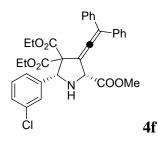
The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}_{D} = -46.4$ (*c* 0.7, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45-7.32 (m, 13H), 7.27-7.26 (m, 1H), 5.24 (s, 1H), 4.76 (s, 1H), 4.14-4.08 (m, 1H), 3.99-3.93 (m, 1H), 3.90-3.84 (m, 1H), 3.75-3.69 (m, 1H), 3.55 (s, 3H), 0.96-0.83 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.45, 52.40, 61.59, 61.80, 62.04, 67.60, 68.42, 106.91, 116.41, 127.73, 127.85, 128.13, 128.28, 128.43, 133.90, 134.69, 135.33, 135.48, 167.69, 168.33, 170.19, 200.53; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 696 cm⁻¹. HRMS Calcd. For C₃₂H₃₀ClNO₆+H⁺: 560.1834, found 560.1841. The product was analyzed to determine the enantioselectivity of the reaction: 93% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 11.30 and 15.26 min.



(2R,5S)-4,4-diethyl 2-methyl 5-(2-chlorophenyl)-3-(2,2-diphenylvinylidene)

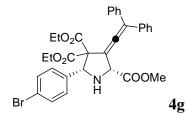
pyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 90% yield. It was purified by flash chromatography to afford yellow oil; $[\alpha]^{25}_{D} = -131.8$ (*c* 1.6, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.60-7.57 (m, 1H), 7.44-7.20 (m, 13H), 5.75 (s, 1H), 4.79 (s, 1H), 4.12-4.06 (m, 1H), 4.03-3.95 (m, 1H), 3.89-3.83 (m, 1H), 3.69-3.62 (m, 1H), 3.58 (s, 3H), 1.01-0.84 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.28, 13.53, 29.57, 52.37, 61.43, 61.83, 62.04, 64.78, 68.27, 106.17, 116.53, 126.59, 127.67, 127.78, 128.22, 128.34, 128.60, 128.80, 129.17, 129.62, 134.39, 135.29, 135.65, 167.15, 167.98, 170.77, 200.93; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 696 cm⁻¹. HRMS Calcd. For C₃₂H₃₀ClNO₆+H⁺: 560.1834, found 560.1839. The product was analyzed to determine the enantioselectivity of the reaction: 92% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 10.18 and 12.26 min.



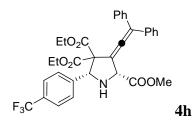
(2*R*,5*R*)-4,4-diethyl 2-methyl 5-(3-chlorophenyl)-3-(2,2-diphenylvinylidene) pyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. It was purified by flash chromatography to afford yellow oil. [α]²⁵_D = -50.9 (*c* 1.7, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.49 (s, 1H), 7.40-7.26 (m, 13H), 5.23 (s 1H), 4.76 (s, 1H), 4.15-4.09 (m, 1H), 4.00-3.96 (m, 1H), 3.94-3.86 (m, 1H), 3.77-3.71 (m, 1H), 3.55 (s, 3H), 0.94-0.83 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.45, 13.51, 29.60, 52.43, 61.65, 61.80, 62.07, 67.66, 68.45, 106.82, 116.50, 125.41, 127.24, 127.76, 127.88, 128.13, 128.22, 128.31, 128.43, 129.41, 134.11, 135.30, 135.52, 138.29, 167.66, 168.24, 170.16, 200.59; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 696 cm⁻¹. HRMS Calcd. For $C_{32}H_{30}CINO_6+H^+$: 560.1834, found 560.1840. The product was analyzed to determine the enantioselectivity of the reaction: 90% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.98 and 12.93 min.



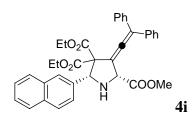
(2*R*,5*R*)-4,4-diethyl 2-methyl 5-(4-bromophenyl)-3-(2,2-diphenylvinylidene) pyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 89% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}{}_{D} = -31.3$ (*c* 2.1, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.49-7.46 (m, 2H), 7.39-7.26 (m, 12H), 5.24 (s 1H), 4.78 (s, 1H), 4.14-4.07 (m, 1H), 3.99-3.95 (m, 1H), 3.93-3.84 (m, 1H), 3.76-3.70 (m, 1H), 3.55 (s, 3H), 0.96-0.80 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.45, 52.37, 61.59, 61.74, 62.01, 67.63, 68.33, 106.85, 116.41, 122.05, 127.70, 127.85, 128.10, 128.28, 128.40, 128.74, 131.18, 135.21, 135.45, 167.63, 168.27, 170.13, 200.53; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₂H₃₀BrNO₆+H⁺: 604.1329, found 604.1325. The product was analyzed to determine the enantioselectivity of the reaction: 91% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 12.50 and 17.62 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-(4-(trifluoromethyl) phenyl)pyrrolidine-2,4,4-tricarboxylate

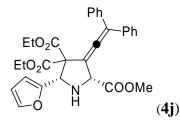
The title compound was prepared according to the general procedure as described above in 88% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}_{D} = -52.2$ (*c* 1.9, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.58-7.54 (m, 5H), 7.32-7.18 (m, 9H), 5.21 (s, 1H), 4.68 (s, 1H), 4.07-4.01 (m, 1H), 3.92-3.86 (m, 1H), 3.78-3.72 (m, 1H), 3.63-3.57 (m, 1H), 3.47 (s, 3H), 0.86 (t, *J* = 7.2, 3H), 0.77 (t, *J* = 7.2, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.30, 13.48, 52.43, 61.63, 61.78, 62.14, 67.71, 68.47, 106.81, 116.59, 124.97, 125.02, 127.52, 127.79, 127.91, 128.14, 128.33, 128.40, 128.46, 135.27, 135.47, 140.41, 167.58, 168.30, 170.19, 200.59; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₃H₃₀F₃NO₆+H⁺: 594.2098, found 594.2119. The product was analyzed to determine the enantioselectivity of the reaction: 93% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.75 and 9.92 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-(naphthalen-2-yl) pyrrolidine-2,4,4-tricarboxylate

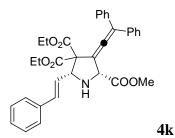
The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}{}_{D} = -24.8$ (*c* 1.6, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.92 (s, 1H), 7.82-7.79 (m, 3H), 7.63-7.48 (m, 1H), 7.48-7.26 (m, 11H), 5.44 (s, 1H), 4.82 (s, 1H),

4.15-4.10 (m, 1H), 4.01-3.96 (m, 1H), 3.79-3.71 (m, 1H), 3.65-3.57 (m, 4H), 0.95 (t, J = 6.9, 3H), 0.73 (t, J = 7.2, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.28, 13.51, 52.38, 61.43, 61.97, 68.41, 68.71, 107.31, 116.32, 125.23, 125.66, 126.01, 127.44, 127.67, 127.82, 128.01, 128.13, 128.28, 128.42, 132.97, 133.03, 133.61, 135.39, 135.58, 167.83, 168.45, 170.30, 200.49; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₆H₃₃NO₆+H⁺: 576.2381, found 576.2397. The product was analyzed to determine the enantioselectivity of the reaction: 91% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 9.38 and 12.62 min.



(2*R*,5*S*)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-(furan-2-yl) pyrrolidine-2,4,4-tricarboxylate

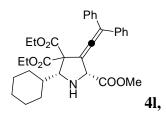
The title compound was prepared according to the general procedure as described above in 82% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}_{D} = -38.2$ (*c* 1.4, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.38-7.33 (m, 10H), 6.44 (s, 1H), 6.36 (s, 1H), 5.20 (s, 1H), 4.71 (s, 1H), 4.09-3.93(m, 4H), 3.54(s, 3H), 1.09 (t, *J* = 6.9, 3H), 0.95 (t, *J* = 6.9, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.56, 13.62, 52.41, 61.78, 61.97, 63.54, 67.05, 106.30, 108.70, 110.52, 116.36, 127.70, 127.84, 128.14, 128.20, 128.28, 128.40, 128.45, 135.42, 135.55, 142.22, 149.02, 167.69, 167.89, 170.02, 200.67; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₀H₂₉NO₇+H⁺: 516.2017, found 516.2027. The product was analyzed to determine the enantioselectivity of the reaction: 90% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 9.36 and 10.93 min.



(2R,5R,E)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-5-styrylpyrrolidine-

2,4,4-tricarboxylate

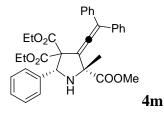
The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}_{D} = -50.3$ (*c* 0.5, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.40-7.30 (m, 15H), 6.78 (d, *J* = 16.2, 1H), 6.38-6.31 (m, 1H), 4.74 (s, 1H), 4.35 (d, *J* = 5.4, 1H), 4.13-4.05 (m, 4H), 3.54(s, 3H), 1.09 (t, *J* = 7.2, 3H), 0.99 (t, *J* = 7.2, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.89, 14.24, 52.69, 62.11, 62.22, 62.43, 67.05, 67.28, 67.75, 106.69, 116.50, 124.49, 126.79, 127.96, 128.08, 128.42, 128.57, 128.70, 128.76, 129.07, 132.73, 135.85, 136.74, 168.22, 168.36, 170.46, 200.93; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₄H₃₃NO₆+H⁺: 552.2381, found 552.2400. The product was analyzed to determine the enantioselectivity of the reaction: 91% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 12.68 and 19.14 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 5-cyclohexyl-3-(2,2-diphenylvinylidene) pvrrolidine-2,4,4-tricarboxylate

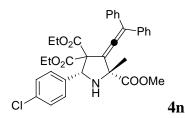
The title compound was prepared according to the general procedure as described above in 82% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}{}_{D} = -64.5$ (*c* 1.4, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.36-7.29 (m,

10H), 4.66 (s, 1H), 4.22-4.14 (m, 2H), 4.06-3.93 (m, 2H), 3.82 (d, J = 8.7, 1H), 3.51 (s, 3H), 2.05-1.65 (m, 11H), 0.90-0.74 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.62, 13.86, 14.04, 22.62, 26.03, 26.19, 29.29, 29.61, 30.99, 31.14, 31.86, 39.94, 52.21, 61.52, 61.66, 61.95, 66.48, 71.63, 109.08, 116.36, 127.61, 127.73, 128.25, 128.37, 135.61, 168.25, 168.59, 170.33, 199.49; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₂H₃₇NO₆+H⁺: 532.2694, found 532.2706. The product was analyzed to determine the enantioselectivity of the reaction: 88% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 6.08 and 10.61 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 3-(2,2-diphenylvinylidene)-2-methyl-5phenylpyrrolidine-2,4,4-tricarboxylate

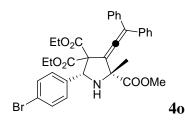
The title compound was prepared according to the general procedure as described above in 88% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_{D}$ = +20.0 (*c* 0.3, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.47-7.45 (m, 2H), 7.35-7.25 (m, 13H), 5.40 (s, 1H), 4.09-4.03 (m, 1H), 3.91-3.79 (m, 2H), 3.71-3.63 (m, 1H), 3.47 (s, 3H), 1.77 (s, 3H), 0.92-0.85 (m, 6H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 13.70, 22.93, 26.79, 29.94, 32.17, 52.93, 61.75, 62.20, 66.78, 67.97, 69.78, 113.02, 116.60, 127.21, 127.91, 128.06, 128.29, 128.48, 128.54, 128.79, 135.61, 136.03, 168.06, 168.76, 173.26, 201.20; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₃H₃₃NO₆+H⁺: 540.2381, found 540.2380. The product was analyzed to determine the enantioselectivity of the reaction: 93% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.09 and 6.38 min.



(2R,5R)-4,4-diethyl 2-methyl 5-(4-chlorophenyl)-3-(2,2-diphenylvinylidene)-2-

methylpyrrolidine-2,4,4-tricarboxylate

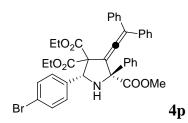
The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}{}_{D} = -15.6 \ (c \ 0.7, \ CHCl_3); {}^{1}H \ NMR \ (CDCl_3, \ TMS, \ 300 \ MHz) \ \delta \ 7.43-7.26 \ (m, 14H), 5.35 \ (s, 1H), 4.06-4.04 \ (m, 1H), 3.91-3.79 \ (m, 2H), 3.74-3.72 \ (m, 1H), 3.46 \ (s, 3H), 1.75 \ (s, 3H), 0.94-0.84 \ (m, 6H); {}^{13}C \ NMR \ (CDCl_3, \ TMS, 100 \ MHz) \ \delta \ 13.49, 26.61, 52.63, 61.57, 62.01, 65.90, 67.63, 69.45, 112.61, 116.41, 127.69, 127.82, 127.96, 127.99, 128.22, 128.28, 128.31, 128.34, 128.45, 128.53, 128.56, 129.01, 133.90, 134.69, 135.33, 135.48, 167.64, 168.51, 173.00, 200.93; IR \ (KBr) v \ 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 \ cm^{-1}$. HRMS Calcd. For $C_{33}H_{32}CINO_6+H^+$: 574.1991, found 574.1983. The product was analyzed to determine the enantioselectivity of the reaction: 95% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220 \ nm$); $t_r = 5.05 \ and 6.43 \ min$.



(2*R*,5*R*)-4,4-diethyl 2-methyl 5-(4-bromophenyl)-3-(2,2-diphenylvinylidene)-2-methylpyrrolidine-2,4,4-tricarboxylate

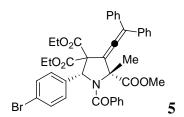
The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}{}_{D} = -16.1 \ (c \ 1.0, \ CHCl_3); {}^{1}H \ NMR \ (CDCl_3, \ TMS, \ 300 \ MHz) \ \delta \ 7.48-7.45 \ (m, 2H), \ 7.37-7.26 \ (m, \ 12H), \ 5.33 \ (s, \ 1H), \ 4.06-4.03 \ (m, \ 1H), \ 3.92-3.84 \ (m, \ 2H), \ 3.72-3.71 \ (m, \ 1H), \ 3.46 \ (s, \ 3H), \ 1.75 \ (s, \ 3H), \ 0.94-0.83 \ (m, \ 6H); {}^{13}C \ NMR \ (CDCl_3, \ CDCl_3, \ MR)$

TMS, 100 MHz) δ 13.46, 26.57, 52.60, 61.56, 62.00, 65.94, 67.60, 69.38, 112.58, 116.39, 122.04, 127.67, 127.81, 127.98, 128.25, 128.31, 128.49, 128.59, 128.75, 131.23, 135.21, 135.30, 135.44, 167.60, 168.47, 172.97, 200.91; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₃H₃₂BrNO₆+H⁺: 618.1486, found 618.1488. The product was analyzed to determine the enantioselectivity of the reaction: 96% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.03 and 8.03 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 5-(4-bromophenyl)-3-(2,2-diphenylvinylidene)-2phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 82% yield. It was purified by flash chromatography to afford yellow oil. $[\alpha]^{25}_{D} = +46.4$ (*c* 1.7, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.58-7.56 (m, 2H), 7.47-7.35 (m, 17H), 4.97 (s, 1H), 4.13-4.10 (m, 1H), 4.03-4.00 (m, 1H), 3.80-3.77 (m, 1H), 3.67-3.61 (m, 1H), 3.48 (s, 3H), 0.96 (d, *J* = 6.9 Hz, 3H), 0.88 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 13.40, 13.54, 53.05, 61.69, 62.03, 65.43, 69.89, 74.53, 110.18, 116.09, 121.90, 127.47, 127.75, 127.90, 127.96, 128.04, 128.19, 128.36, 128.39, 128.75, 128.86, 131.10, 135.26, 135.30, 135.39, 139.38, 168.02, 168.36, 171.06, 202.15; IR (KBr) v 3338, 2981, 2925, 1736, 1254, 1201, 1050, 770, 696 cm⁻¹. HRMS Calcd. For C₃₈H₃₄BrNO₆+H⁺: 680.1642, found 680.1648. The product was analyzed to determine the enantioselectivity of the reaction: 96% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.98 and 16.31 min.



(2*R*,5*R*)-4,4-diethyl 2-methyl 1-benzoyl-5-(4-bromophenyl)-3-(2,2-diphenylvinylidene)-2-phenylpyrrolidine-2,4,4-tricarboxylate

The compound **40** (0.23mmol) was dissolved in 2mL CH₂Cl₂ at room temperature, and then benzoyl chloride (0.46 mmol) and TEA (0.46 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), It was purified by flash chromatography to afford white solid, 98% yield. $[\alpha]^{25}_{D}$ = -193.4 (*c* 1.0, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.55-7.53 (m, 2H), 7.45-7.16 (m, 10H), 7.01 (m, 2H), 6.01 (s 1H), 4.23-4.16 (m, 2H), 3.66-3.57 (m, 1H), 3.53 (s, 3H), 3.49-3.41 (m, 1H), 1.95 (s, 3H), 1.06 (t, *J* = 6.9 Hz, 3H), 0.68 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 13.53, 13.89, 24.24, 52.76, 62.11, 63.45, 67.35, 67.85, 69.45, 106.66, 118.12, 122.45, 126.48, 128.34, 128.51, 128.68, 128.98, 129.74, 130.74, 131.21, 135.06, 135.34, 136.91, 164.93, 169.30, 169.89, 170.65, 202.73; IR (KBr) v 3425, 1719, 1261 cm⁻¹. HRMS Calcd. For C₄₀H₃₆BrNO₇+Na⁺: 744.1567, found 744.1576. The product was analyzed to determine the enantioselectivity of the reaction: 96% ee, determined by HPLC (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.80 and 9.87 min.

The relative and absolute configuration of (2R,5R)-5 were determined by X-ray diffraction analysis

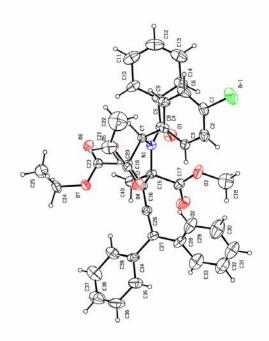
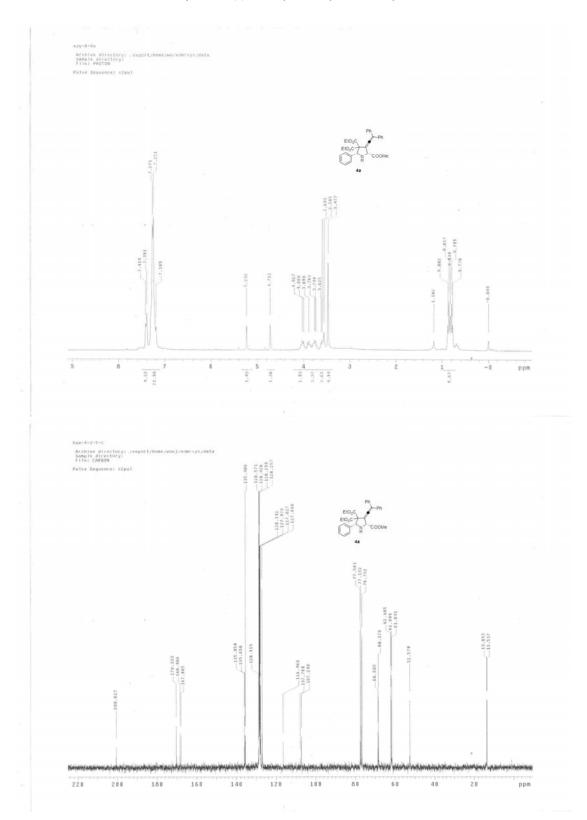


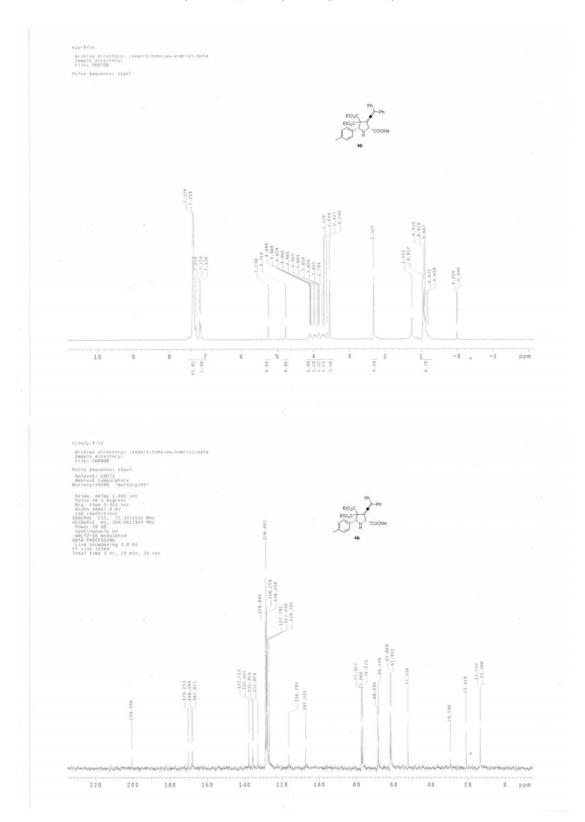
Figure 1. X-ray Structure of (2*R*,5*R*)-5

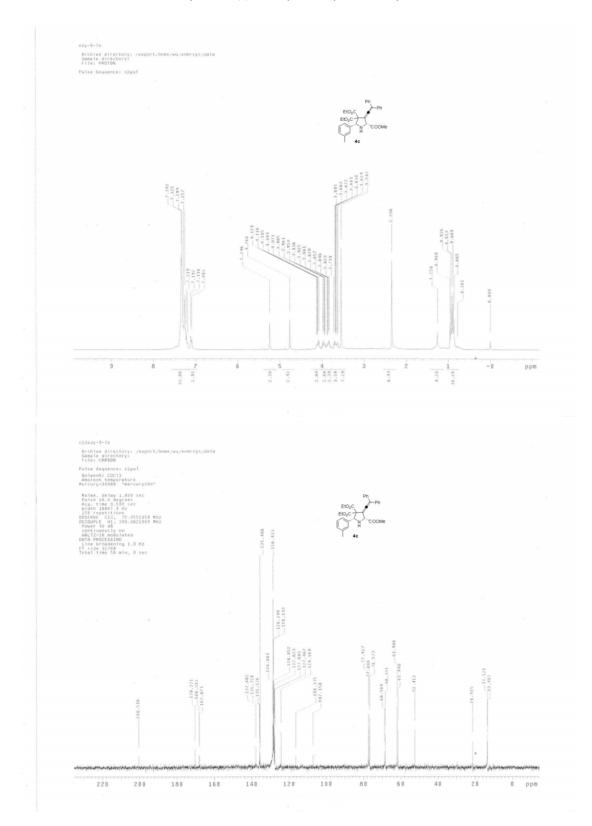
Crystal data for (2R,5R)-**5**: C₄₀H₃₆BrNO₇, M_r = 722.60, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 13.323(2), b = 15.987(3), c = 17.448(3) Å, V = 3716.3(11)Å³, Z = 4, 3536 unique reflections, final $R_1 = 0.0503$ and $wR_2 = 0.1444$ for 4470 observed [$I > 2\sigma(I)$] reflections. Flack $\chi = 0.008(18)$ CCDC 809653 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

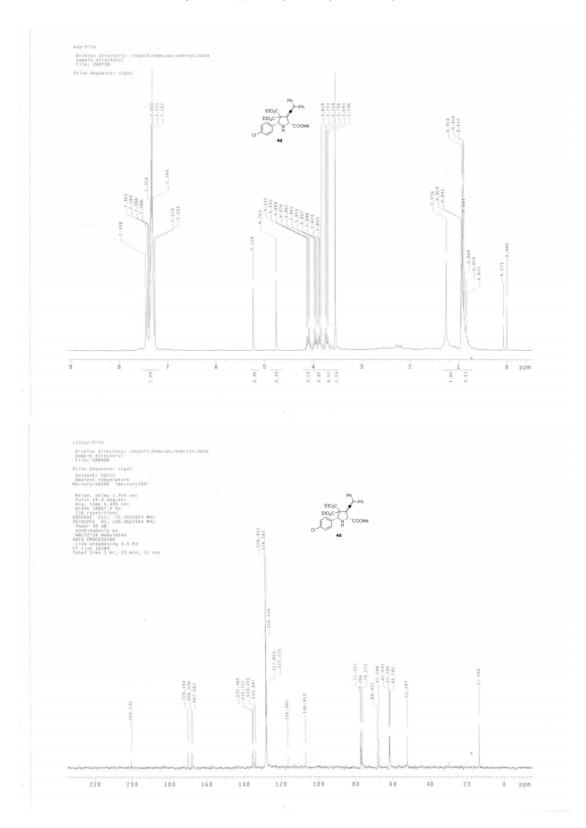
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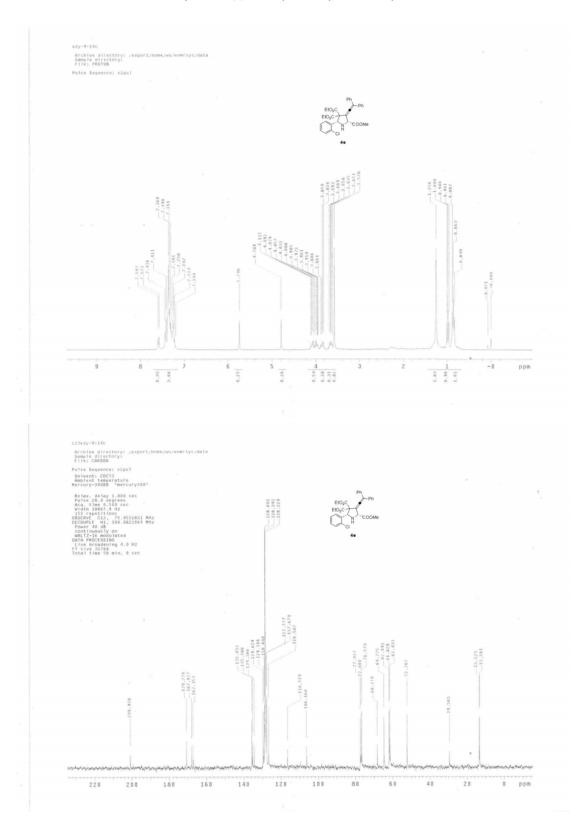
- 1. Wang, C.-J.; Liang, G.; Xue, Z.-Y.; Gao, F J. Am. Chem. Soc. 2008, 130, 17250
- 2. (a) Ratts, K. W.; Partos, R. D. J. Am. Chem. Soc. 1969, 91, 6112. (b) Browne, N. R.;
- Brown, R. F. C.; Eastwood, F. W.; Fallon, G. D. Aust. J. Chem. 1987, 40, 1675.

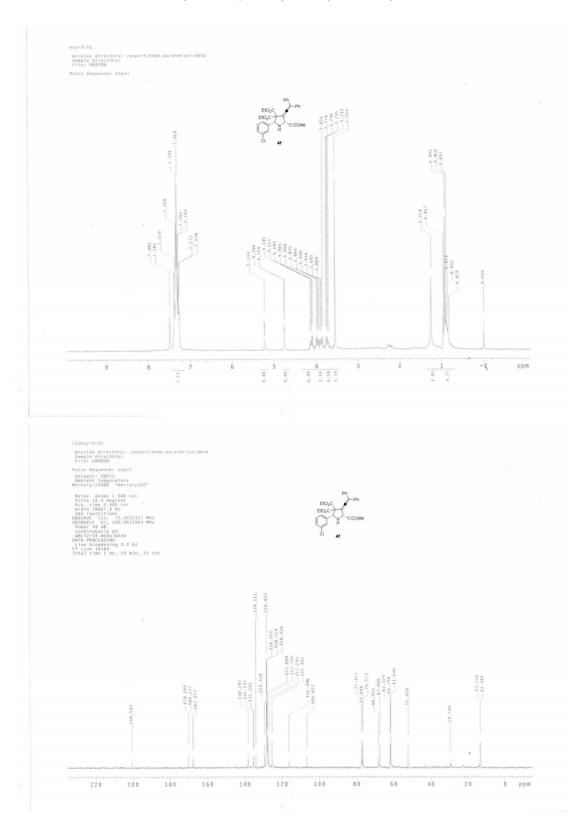


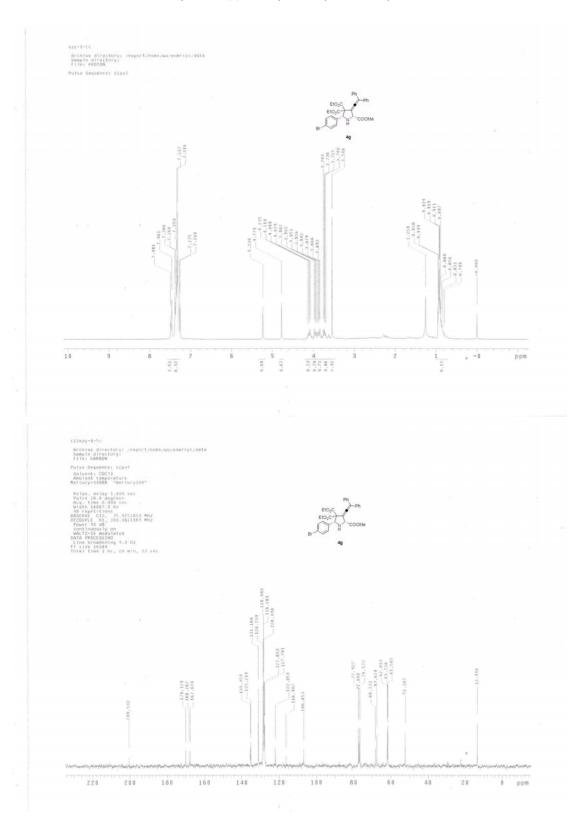


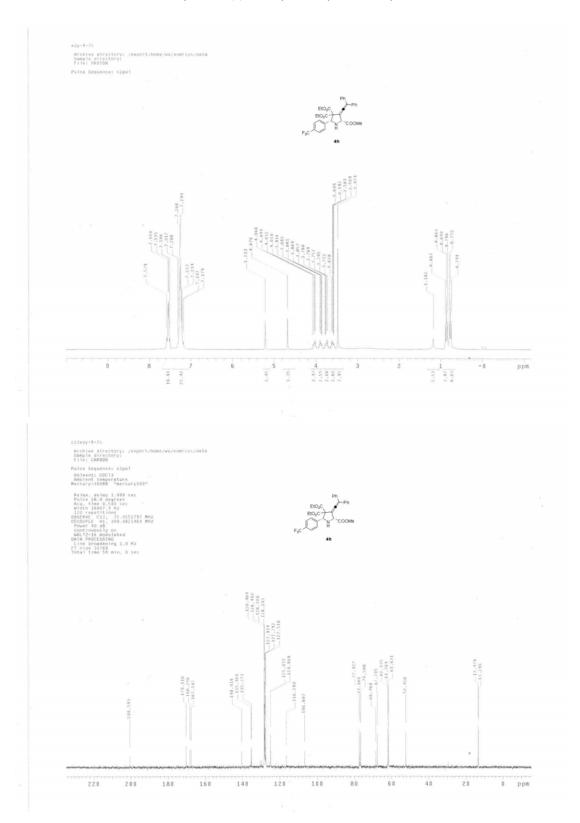


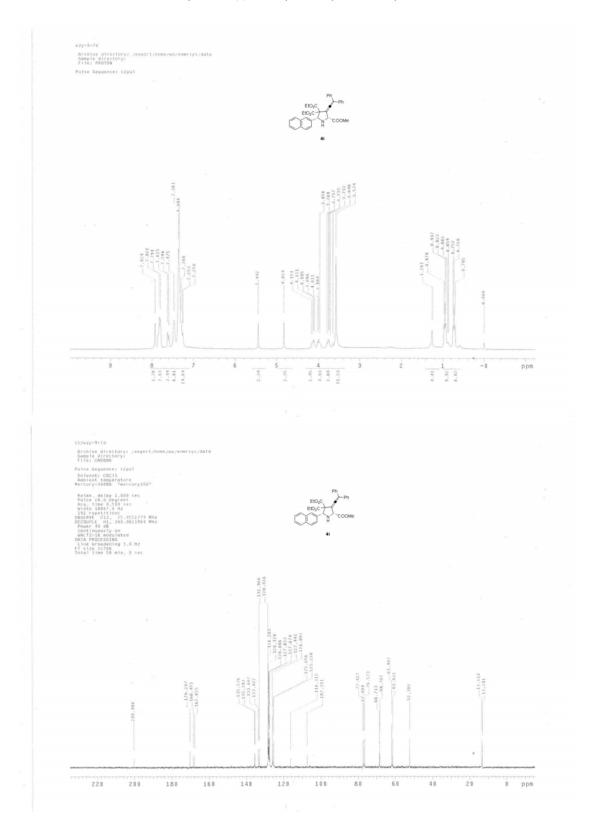


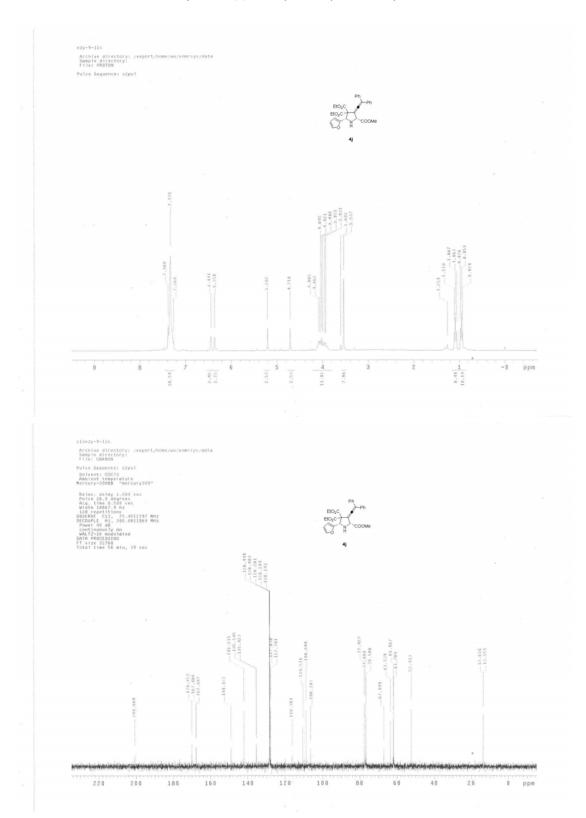


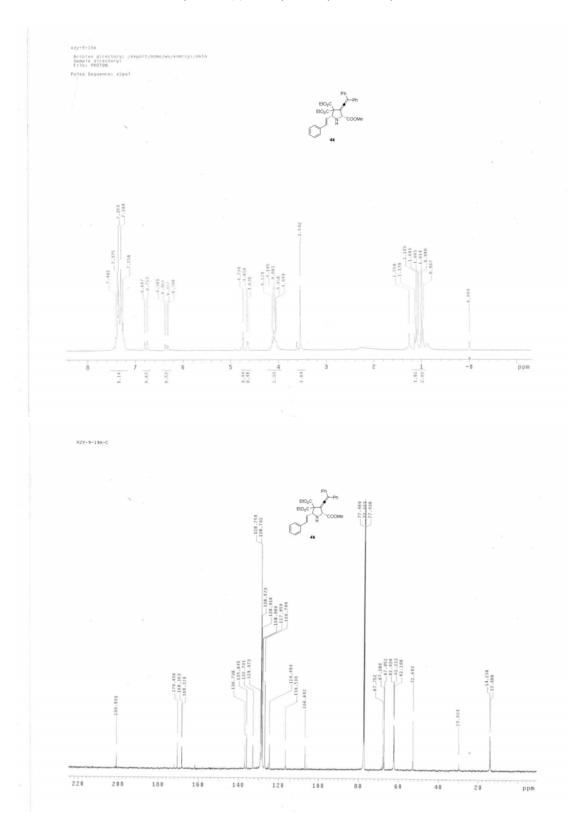


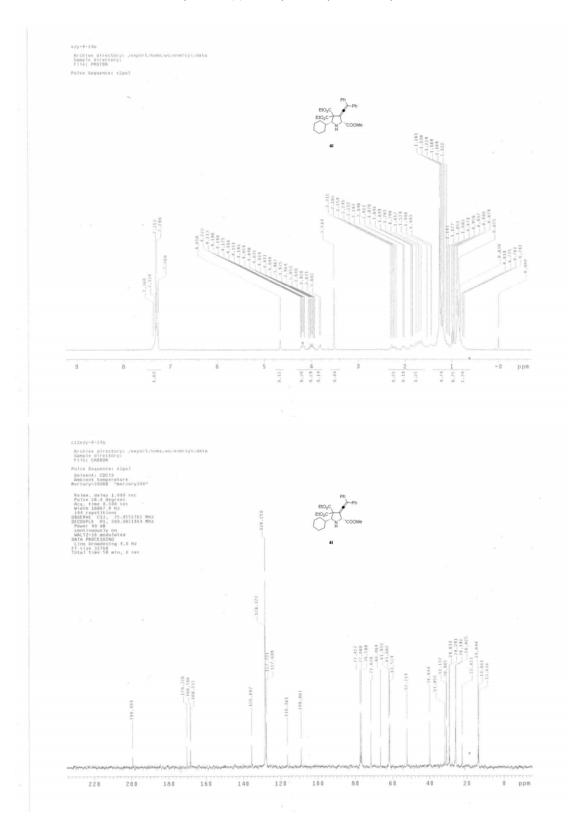


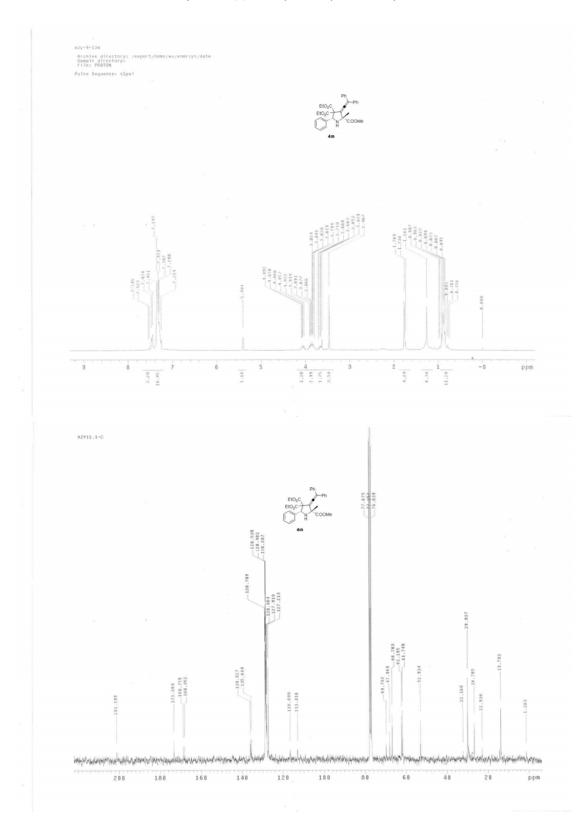


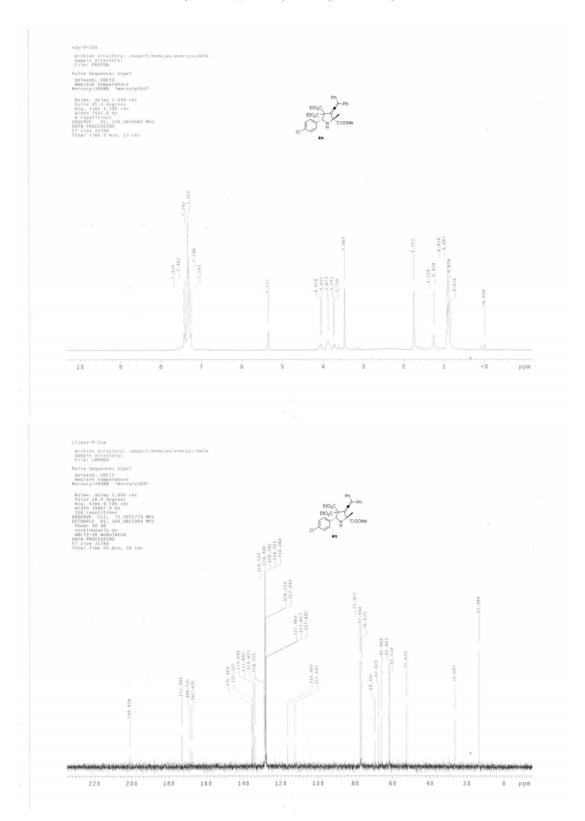


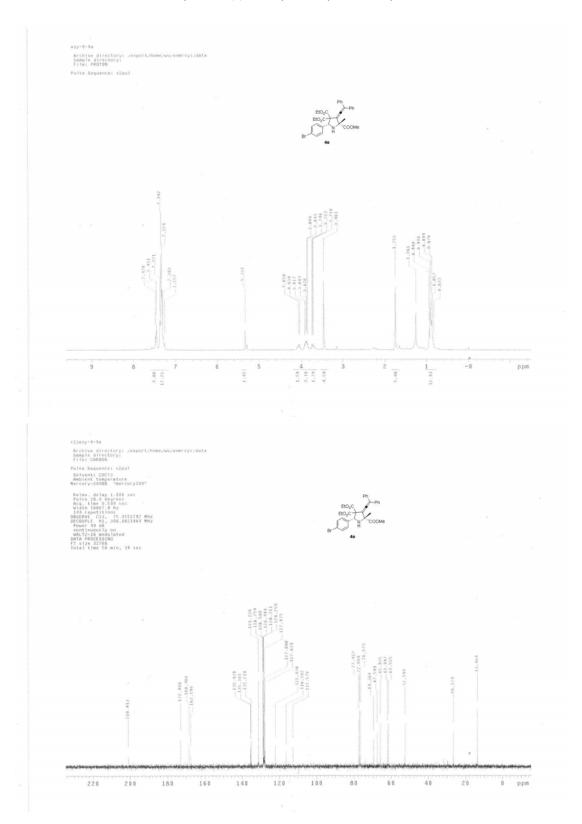


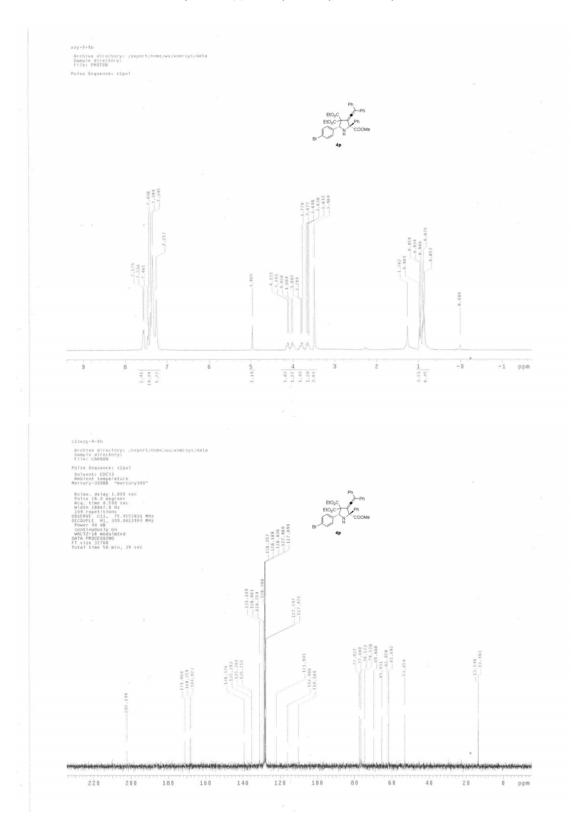


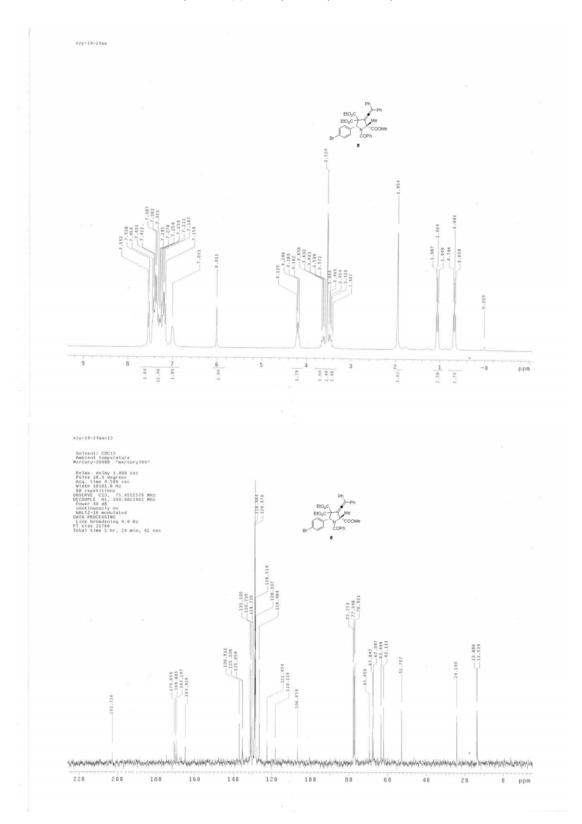


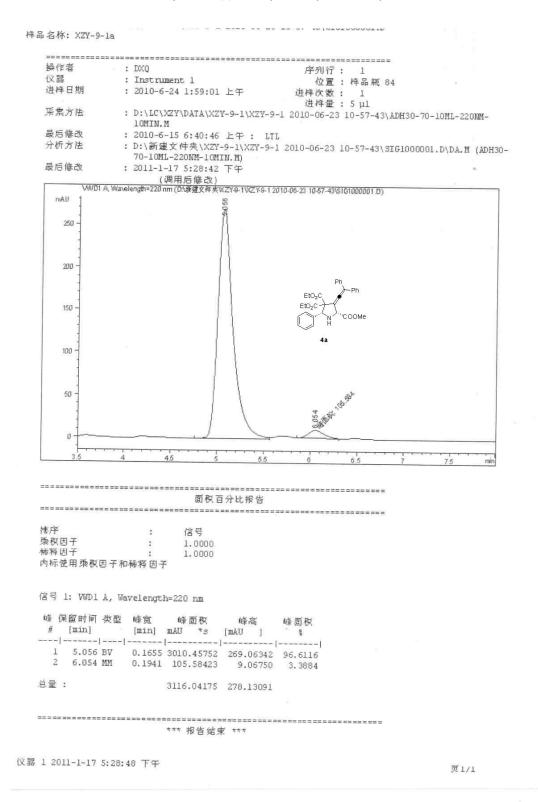


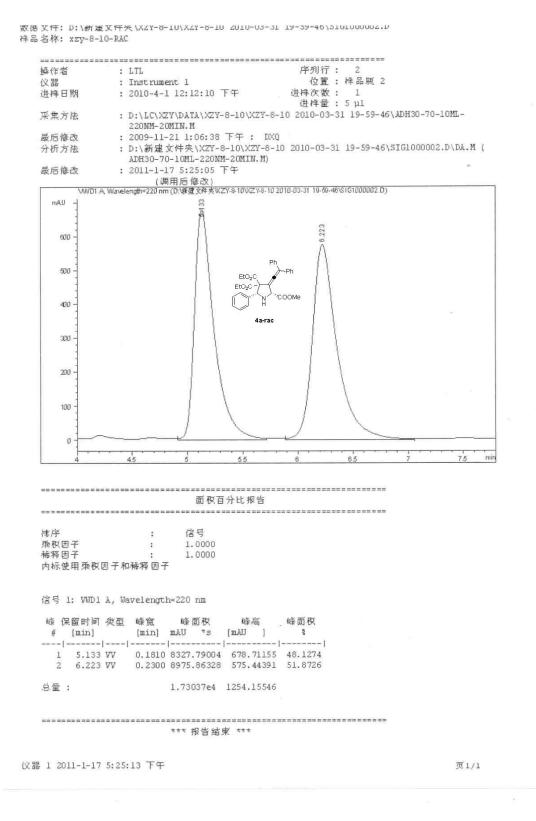












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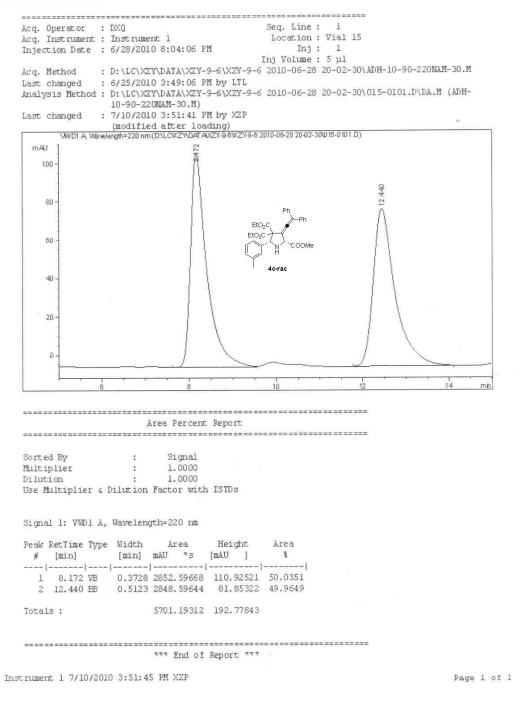
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Darg life D:/Pr/VSI/DHIK/VSI-2-//VSI-2-/ 5010-00-20 50 04 41/000 01010 Sample Name: xzy-9-7E Seq. Line : Acq. Operator : LTL 1 Location : Vial 85 Acq. Instrument : Instrument 1 Inj: 1 Inj Volume: 5 µl Injection Date : 6/30/2010 8:05:57 PM : D:\LC\XZY\DATA\XZY-9-7\XZY-9-7 2010-06-30 20-04-41\ADH-10-90-220NAM-30.M Acq. Method Last changed : 6/25/2010 3:49:06 PM by LTL Analysis Method : D:\LC\XZY\DATA\XZY-9-7\XZY-9-7 2010-06-30 20-04-41\085-0101.D\DA.M (ADH-10-90-220NAM-30.M) : 7/10/2010 3:58:28 PM by XZP Last changed (modified after loading) (W/D1 A, Wavelergth=220 nm (D\LCWZY\DATAVZY\97 \XZY\97 2010-06:30 20-04-41\085-0101.D) Lesi Stale 4604 mAU 300 250 EtO₂ 200 -150 100 10.180 Eles 50 0 min 10 14 Area Percent Report Sorted By Signal 5 1.0000 Multiplier . 1.0000 Di lution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] m&U *s [mAU] d'h -------1-1 8.934 MM 0.3424 6375.19141 310.27399 95.8019 2 13.183 MM 0.4684 279.36490 9.93954 4.1981 1 6654.55630 320.21352 Totals : *** End of Report ***

Instrument 1 7/10/2010 3:58:32 PM XZP

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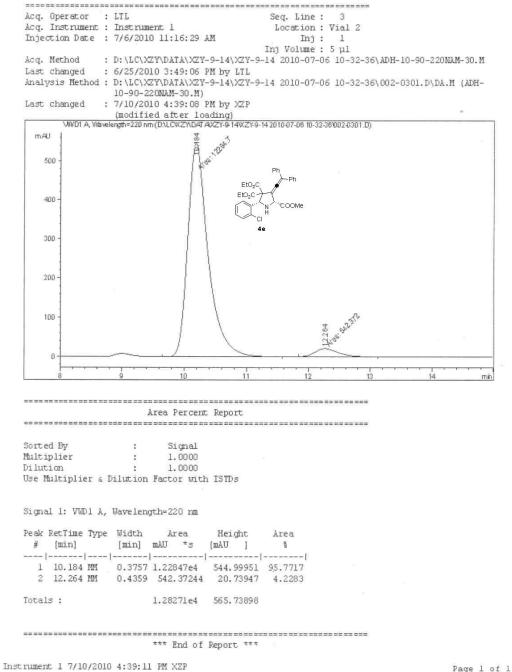
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm ppe Width Area Height Area
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm ppe Width Area Height Area [min] mAU *s [mAU] %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] []- 1 11.301 M	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm ppe Width Area Height Area [min] mAU *s [mAU] %
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] []- 1 11.301 M	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm ype Width Area Height Area [min] mAU *s [mAU] %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty # [min] 	Area Percent Report : 1.0000 : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm 7pe Width Area Height Area [min] mAU *s [mAU] *
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	Area Percent Report : 1.0000 : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm 7pe Width Area Height Area [min] mAU *s [mAU] *
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm ype Width Area Height Area [min] mAU *s [mAU] %

DEER LITE D:/PC/YYI/DEIE/YYI-A-9/YYI-A-9 YOIO-00-79 IO-92-91/001-0101'D Sample Name: xzy-9-3a Acq. Operator : LTL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Inj: 1 Inj Volume: 5 µl Injection Date : 6/25/2010 5:01:06 PM Acq. Method : D:\LC\XZY\DATA\XZY-9-3\XZY-9-3 2010-06-25 16-59-51\ADH-10-90-220NAM-30.M Last changed : 6/25/2010 3:49:06 PM by LTL Analysis Method : D:\LC\XZY\DATA\XZY-9-3\XZY-9-3 2010-06-25 16-59-51\001-0101.D\DA.M (ADH-10.00 2000W S 20 N) 10-90-220NAM-30.M) : 7/10/2010 3:30:46 PM by XZP Last changed (modified after loading) (WVD1 A Wavelergth=220 nm(D\LCXZYDATAVZY-9.3 XZY9.3 2010-06-25 16:69-510201-0101.D) mAU 4 EtO₂C 80 EtO₂C 15.818 70 CL 4d-rad 60 50 40 30 20 10 Ð 17 min 14 15 18 10 11 12 -----Area Percent Report Sorted By 5 Signal 1.0000 Multiplier : 1,0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] 뤕 ----1 11.714 BB 0.3956 2392.79663 90.15250 50.0689 2 15.818 EB 0.5417 2386.20850 65.91983 49.9311 Totals : 4779.00513 156.07233 *** End of Report *** .

Instrument 1 7/10/2010 3:30:50 PM XZP

DACA LITE D:/PC/YSI/NWIW/YSI-A-14/YSI-A-14 SOIO-0/-OD IO-35-30/005-0301'N Sample Name: XZY-9-14C

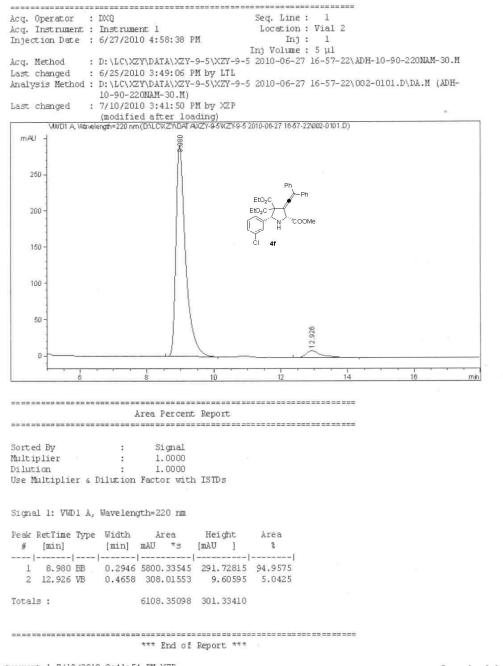


Data File D:\LC\XZY\DATA\XZY-9-13\XZY-9-13 2010-07-05 17-03-27\003-0101.D Sample Name: xzy-9-13E

Acg. Operator	• TMC	Seq. Line		
	: Instrument 1		: Vial 3	
	: 7/5/2010 5:04:44 PM		: 1	
Hijecoron bace	. ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,,	Inj Volume		
Acg. Method	: D:\LC\XZY\DATA\XZY-9-			\ADH-10-90-220NAM-40.M
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MIGT AS TO ME CHOO	10-90-220NAM-40.M)	10,721 2 10 2010 07	00 17 00 17	1000 offerin laure (res
Last changed	: 7/10/2010 4:20:57 PM	htt Y7D		
hase changed	(modified after loadi			*
VI0/D1 & 108	avelength=220 nm (DALCWZYADAT AVZ		03-27/003-0101.D)	
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Sorted By	: Signal			
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	4 Dilution Factor with I	STDs		
ees investigated		110 B.B. (70)		
Signal 1: VAD1	A, Wavelength=220 nm			
		Weight Turn		
Peak RetTime Ty		Height Area		
# [min]		IAU] %		
1 10.242 MM	0.3894 1131.38806	48.42767 48.1329		
2 12.343 MM	0.4908 1219.15991	41.40055 51.8671		
Totals :	2350.54797	89.82822		
	*** End of Re			
	state of the			
	010 4.01.00 NV V2D			

Instrument 1 7/10/2010 4:21:00 PM XZP

Data File D:\LC\XZY\DATA\XZY-9-5\XZY-9-5 2010-06-27 16-57-22\002-0101.D Sample Name: xzy-9-5b

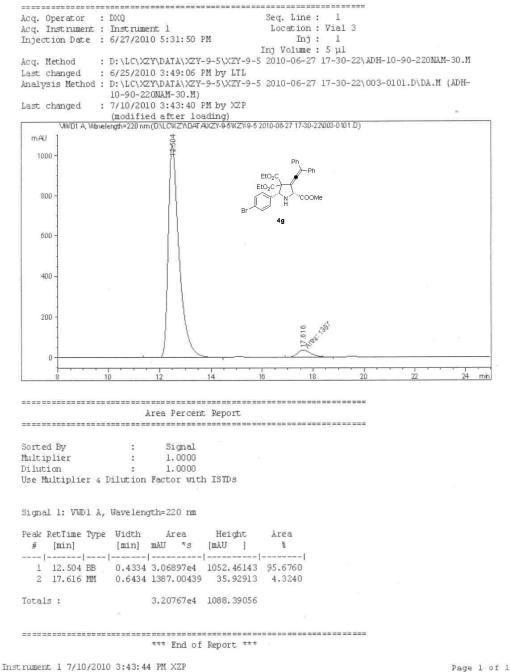


Instrument 1 7/10/2010 3:41:54 PM XZP

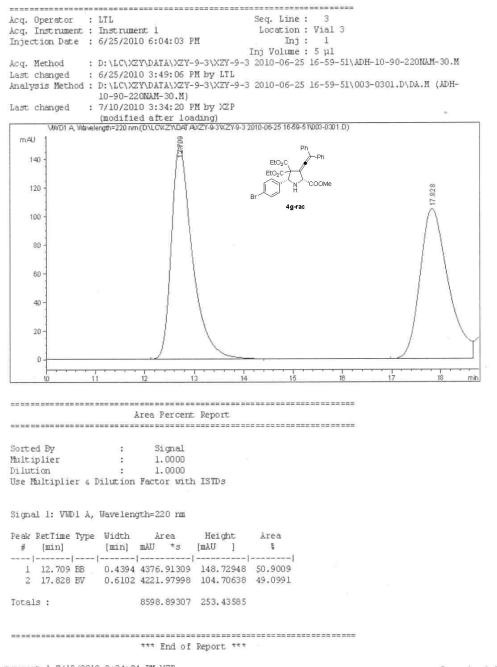
Data File D:\LC\XZY\DATA\XZY-9-3\XZY-9-3 2010-06-25 16-59-51\002-0201.D Sample Name: xzy-9-3b

Acq. Operator : LTL	Seq. Line : 2	
Acq. Instrument : Inst		
Injection Date : 6/25		
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	C\XZY\DATA\XZY-9-3\XZY-9-3 2010-06-25 16-59-51\ADH-10-90-220NAM-30	. 11
	/2010 3:49:06 PM by LTL	H-
	C\XZY\DATA\XZY-9-3\XZY-9-3 2010-06-25 16-59-51\002-0201.D\DA.M (AD) 0-220NAM-30.M)	
	/2010 3:32:39 PM by XZP	
	ified after loading)	
VM/D1 A, Wavelength=2	20 nm (DALCWZYADAT AWZY-9-3WZY-9-3 2010-06-25 16-59-51/002-0201.D)	
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Comment is DIUD 1 1 Treese	lowerth-220 mm	
Signal 1: VWD1 A, Wave	rengen=220 mm	
Peak RetTime Type Wid	th Area Height Area	
	n] mAU *s [mAU] %	
1 8.970 VV 0.2		
	430 1521.76160 51.18403 49.8069	
Totals :	3055.32068 127.36186	
	*** End of Report ***	
1 1		
rument 1 7/10/2010 3:3	2:43 PM XZP Pa	ge 1 of

Data File D:\LC\XZY\DATA\XZY-9-5\XZY-9-5 ZUIU-U6-27 17-30-22\UU3-UIU1.D Sample Name: xzy-9-5c



Data File D:\LC\XZY\DAT&\XZY-9-3\XZY-9-3 2010-06-25 16-59-51\003-0301.D Sample Name: xzy-9-3c



Instrument 1 7/10/2010 3:34:24 PM XZP

DACH LITE D:/PC/VSI/DMIM/VVI-A-//VVI-A-/ SOIO-00-20 12-50-20/005 010115 Sample Name: xzy-9-7C Seq. Line : 1 Acq. Operator : LTL Acq. Instrument : Instrument 1 Location : Vial 82 Inj: 1 Inj Volume: 5 µl Injection Date : 6/30/2010 3:28:31 PM
 Inf
 Volume : 5 µI

 Acq. Method
 : D:\LC\XZY\DATA\XZY-9-7\XZY-9-7 2010-06-30 15-26-50\ADH-10-90-220NAM-30.M

 Last changed
 : 6/25/2010 3:49:06 PM by LTL

 Analysis Method
 : D:\LC\XZY\DATA\XZY-9-7\XZY-9-7 2010-06-30 15-26-50\082-0101.D\DA.M (ADH-10-90-220NAM-30.M)
: 7/10/2010 3:53:37 PM by XZP Last changed (modified after loading) WWD1 A Wavelergt=220 nm(D\LCWZYDATAVZY-9.7 XZY-9.7 2010-06:30 15-26-50/082-0101.D) No. Construction of the second mAU \$752 120 EtO₂0 EtO₂C 100 1COOMe 4h 80 60 40 20 920 Û 10 3Ì min] Area Percent Report Signal 1.0000 Sorted By : : Multiplier 1,0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] 12 - | ------ | ------ | -----------8.752 MM 0.3051 2581.28760 141.00961 96.5078 9.920 MM 0.3149 93.40594 4.94408 3.4922 1 2 Totals : 2674.69353 145.95369 *** End of Report ***

Instrument 1 7/10/2010 3:53:41 PM XZP

NACE LITE D:/PC/YYI/NETE/YYI-A-0/YYI-A-0 YOTO-00-70 I/-50-40/010-05011 Sample Name: xzy-9-6c Acq. Instrument : Instrument 1 Location . Mark Location : Vial 13 Inj : l Inj Volume : 5 μl Injection Date : 6/28/2010 5:59:28 PM Ing Volume : 5 µl Acq. Method : D:\LC\XZY\DATA\XZY-9-6\XZY-9-6 2010-06-28 17-26-43\ADH-10-90-220NAM-30.M Last changed : 6/25/2010 3:49:06 PM by LTL Analysis Method : D:\LC\XZY\DATA\XZY-9-6\XZY-9-6 2010-06-28 17-26-43\013-0201.D\DA.M (ADH-10-90-220NAM-30.M) Last changed : 7/10/2010 3:49:16 PM by XZP (modified after loading) (WVD1 A Wavelergth=220 nm(D\LCXZYDATAVZY9-6 02010-06-28 17-26-43013-0201.D) mAU 243 3.692 80 -Ph EtO₂C EtO₂C COOME Ň Í 60 F₂C 4h-rac 40 20 0 10 11 min Area Percent Report Sorted By : Signal 1.0000 Multiplier : 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area [mAU] # [min] [min] mAU *s 2 ----. | ------ | ------- | ------- | ------- | 8.543 VV 0.2972 1879.28027 94.03332 50.0727 9.692 VB 0.3329 1873.81970 84.04836 49.9273 1 2 Totals : 3753.09998 178.08168

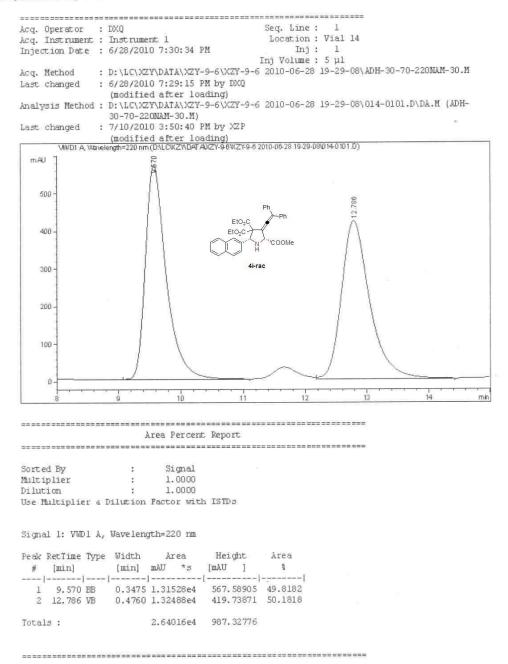
*** End of Report ***

Instrument 1 7/10/2010 3:49:20 PM XZP

Data File D:\LC\XZY\DATA\XZY-9-7\XZY-9-7 2010-06-30 17-14-44\084-0201.D Sample Name: XZY-9-7D

	r : LTL ent : Instrum	ent l		Seq. Line : Location : V:	2 ial 84		
	te : 6/30/20			Inj :	1		
				nj Volume : 5			UN 20 M
Acq. Method Last changed		ZY\DATA\XZY- 10 10:42:51		2010-06-30 17	-14-44\ADE	1-30-70-2201	1AM-30.M
	hod : D:\LC\X			2010-06-30 17	-14-44\084	-0201.D\DA.	M (ADH-
÷		20NAM-30.M)					and arrestation
Last changed		10 3:55:50 F					
W/D1	(IIIOdilii) A. Wavelength=220 nr	ed after los m(DALCWZADATA		0-06-30 17-14-4408	40201.D)		
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Sorted By Multiplier	:	Signal 1.0000					
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Sorted By Multiplier Dilution Use Multipli	:	Signal 1.0000 1.0000 Factor with					
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Sorted By Multiplier Dilution Use Multipli Signal 1: VW	: er & Dilution Dl A, Wavelen Type Width	Signal 1.0000 1.0000 Factor with gth=220 nm	ISTDs	Årea 8			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min]	er & Dilution Dl A, Wavelen Type Width [min]	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s	Height [mAU]	årea 8			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min] 	: er & Dilution Dl A, Wavelen Type Width [min] !! EB 0.3718	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s [] 2.21678e4	Height [mAU] 890.86017 9	لاتوم ما 95.6337			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min]	: er & Dilution Dl A, Wavelen Type Width [min] !! EB 0.3718	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s	Height [mAU] 890.86017 9	årea 8			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min] 	: er & Dilution Dl A, Wavelen Type Width [min] !! EB 0.3718	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s [] 2.21678e4	Height [mAU] 31.03508	لاتوم ما 95.6337			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min] 	: er & Dilution Dl A, Wavelen Type Width [min] !! EB 0.3718	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s [] 2.21678e4 1012.11017	Height [mAU] 31.03508	لاتوم ما 95.6337			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min] 1 9.376 2 12.624 Totals :	: er & Dilution Dl A, Wavelen Type Width [min] EB 0.3718 MM 0.5435	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s 1 2.21678e4 1012.11017 2.31799e4	Height [mAU] 31.03508 921.89524	لاتوم ما 95.6337 4.3663			
Sorted By Multiplier Dilution Use Multipli Signal 1: VW Peak RetTime # [min] 1 9.376 2 12.624 Totals :	: er & Dilution Dl A, Wavelen Type Width [min] !! EB 0.3718	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s 1 2.21678e4 1012.11017 2.31799e4	Height [mAU] 890.86017 31.03508 921.89524	لاتوم ما 95.6337 4.3663			

Data File D:\LC\XZY\DATA\XZY-9-6\XZY-9-6 2010-06-28 19-29-08\014-0101.D Sample Name: xzy-9-6d



Instrument 1 7/10/2010 3:50:44 FM XZP

Data File D:\LC\XZY\DATA\XZY-9-12\TMC-2-76 2010-07-03 10-45-25\UU2-U3U1.D Sample Name: XZY-9-12C

Acq. Instr				Seq. Line :	3	
AUG. LINCL	tor : TMC ument : Instru	ment 1		Location :		
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milection	vale . 11012	10 12,19,30 P	**	Inj Volume :		
less Hables		THO DATE THO	2 261 740 2			H-10-90-220NAM-30.M
Acq. Metho				-76 2010-07-03	10-43-23/203	1-10-50-220MAII 50.11
Last chang	ed : 6/25/2	010 3:49:06 P	T DY LIL	76 2010 07 02	10 45 251 00	2-0301.D\DA.M (ADH-
Analysis M			9-12/110-2	-/6 2010-07-03	10-40-20100	2-0501. DIDA.M (AMI-
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		Area Percent	Report			14 min
6		Area Percent	Report			14 min
		Area Percent	Report			14 min
Sorted By		Area Percent Signal	Report			14 min
Sorted By Multiplier		Area Percent Signal 1.0000	Report			14 min
Sorted By Multiplier Dilution	:	Area Percent Signal 1,0000 1,0000	Report			14 min
Sorted By Multiplier Dilution		Area Percent Signal 1,0000 1,0000	Report			14 min
Sorted By Multiplier Dilution	:	Area Percent Signal 1,0000 1,0000	Report			14 min
Sorted By Multiplier Dilution Use Multip	:	Area Percent Signal 1.0000 1.0000 pn Pactor with	Report			14 min
Sorted By Multiplier Dilution Use Multip Signal 1: Peak RetTi # [mir	: ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ;	Area Percent Signal 1.0000 1.0000 on Factor with ength=220 nm Area m&U *s	: Report n ISTDs Height [mAU]	Àrea §		14 min
Sorted By Multiplier Dilution Use Multip Signal 1: Peak RetTi # [mir 1 9.3	: ; ;)lier & Dilutio VUD1 Å, Wavelo me Type Width	Area Percent Signal 1.0000 1.0000 on Factor with ength=220 nm h Area mAU *s 	Report I ISTDs Height [mAU] 603.49316	للاوم ۱ ۱ا ۱ 94.9261		14 min
Sorted By Multiplier Dilution Use Multip Signal 1: Peak RetTi # [mir 1 9.3	: ;; ;; ;; ;; ;; ;; ;; ;; ;; ;; ;; ;; ;;	Area Percent Signal 1.0000 1.0000 on Factor with ength=220 nm h Area mAU *s 	Report I ISTD's Height [mAU] 603.49316 26.83401	Area § 94.9261 5.0739		14 min
Sorted By Multiplier Dilution Use Multip Signal 1: Peak RetTi # [mir 1 9.3 2 10.9 Totals :	: : : : : : : : : : : : : :	Area Percent Signal 1.0000 1.0000 on Factor with ength=220 nm Area MAU *s 1.24431e4 31 665.09320 1.31082e4	Height [mAU] 603.49316 26.83401 630.32718	Àrea § 11 94.9261 5.0739		14 min

NGCS RITE D:/PC/YSI/DEIE/YSI-A-TI/YSI-A-TI SOTO-O/-OS O2-41-54/002 OTOI'D Sample Name: xzy-9-11C Location : Vial 3 Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\XZY\DATA\XZY-9-11\XZY-9-11 2010-07-02 09-41-24\ADH-10-90-220NAM-30.M Last changed : 6/25/2010 3:49:06 PM by LTL Analysis Method : D:\LC\XZY\DATA\XZY-9-11\XZY-9-11 2010-07-02 09-41-24\003-0101.D\DA.M (ADH-10-90-220NAM-30.M) Last changed : 7/10/2010 4:09:17 PM by XZP (modified after loading) WWD1A Wavelength=220 nm/04/2020 Seq. Line : 1 mAU 350 10.952 300 EtO₂0 EtO₂C 250 Ló 4j-rac 200 150 100 50 0 11 14 min 10 Area Percent Report Sorted By : Signal 1.0000 Multiplier : 1.0000 Dilution 8 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] - | ------ | ------- | ------1 9.383 EV 0.3072 7341.60010 354.46844 49.8463 2 10.952 VB 0.3592 7386.87305 305.55832 50.1537 1 1.47285e4 660.02676 Totals : _____ *** End of Report ***

Instrument 1 7/10/2010 4:09:20 PM XZP

Data File D:\LC\XZY\DATA\XZY-9-19\XZY-9-19 2010-07-08 15-29-47\UUZ-U1U1.D Sample Name: XZY-9-19A

Acq. Operator : Acq. Instrument :					
		Seq. Lin			
			n: Vial 2		
injection Date :	7/8/2010 3:31:20 F		j: 1		
and the second second	D. 11 CI 10000 D 1011 1070	Inj Volum	e; ο μι 7-09 15-20-47\ λτ	H-30-70-220%	11 M-30 M
		7-9-19\XZY-9-19 2010-0	7-00 13-29-477AL	/11-30-70-2201	MI- 30.11
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	30-70-220NAM-30.M)				
Last changed :	7/10/2010 4:42:26				
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Acq. Operator	: LTL	Seq. Line : 2 Location : Vial 2
	: Instrument 1 : 7/7/2010 8:47:45 AM	Inj: 1
IIIJecciul Dace	. 1/1/2010 0.47.45 AI	Inj Volume : 5 µl
Acg. Method	: D:\LC\XZY\DATA\XZY-9-1	16\XZY-9-16 2010-07-07 08-35-21\ADH-30-70-220NAM-30.N
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	30-70-220NAM-30.M)	
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Data File D:\LC\XZY\DATA\XZY-9-14\XZY-9-14 2010-07-06 10-32-36\001-0201.D Sample Name: XZY-9-14B

	ator : LTL Seq. Line : 2
	nument : Instrument 1 Location : Vial 1
Injection	Date : 7/6/2010 10:44:58 AM Inj : 1
2 221027	Inj Volume : 5 µl
Acq. Meth	
Last chan	ged : 6/25/2010 3:49:06 PM by LTL
Analysis	Method : D:\LC\XZY\DATA\XZY-9-14\XZY-9-14 2010-07-06 10-32-36\001-0201.D\DA.M (ADH-
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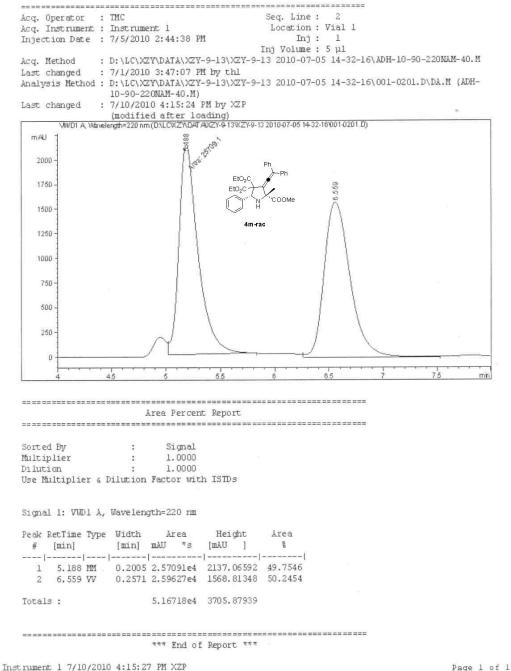
Data File D:\LC\X2Y\DATA\X2Y-9-13\X2Y-9-13 2010-07-05 15-15-21\002-0101.D Sample Name: D

Acq. Operator :	TMC	2	Seq. Line : 1	
Acq. instrument :	Instrument 1		Location : Vial 2	
	7/5/2010 3:16:43 P		Inj: 1	
			nj Volume : 5 µl	
	D:\LC\XZY\DATA\XZY	-9-13\XZY-9-13	3 2010-07-05 15-15-21\AD	H-10-90-220NAM-40.M
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Data File D:\LC\XZY\DATA\XZY-9-14\XZY-9-14 2010-07-07 09-35-47\UU3-0101.D Sample Name: XZY-9-14A

	or : LTL Seq. Line : 1	
	ment : Instrument 1 Location : Vial 3	
Injection Dat	ate : 7/7/2010 9:37:26 AM Inj : 1	
900 - 6 6 6 6 7 7 7 9 7 6 7 7 7	Inj Volume : 5 µl	
Acq. Method	: D:\LC\XZY\DATA\XZY-9-14\XZY-9-14 2010-07-07 09-35-47\ADH-10-90-	-220NAM-30.M
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	10-90-220NAM-30.M)	Sector Contractor
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Data File D:\LC\XZY\DATA\XZY-9-13\XZY-9-13 2010-07-05 14-32-16\001-0201.D Sample Name: XZY-9-13A



Data File D:\LC\XZY\DATA\XZY-9-12\TMC-2-76 2010-07-03 10-45-25\001-0201.D Sample Name: XZY-9-12A

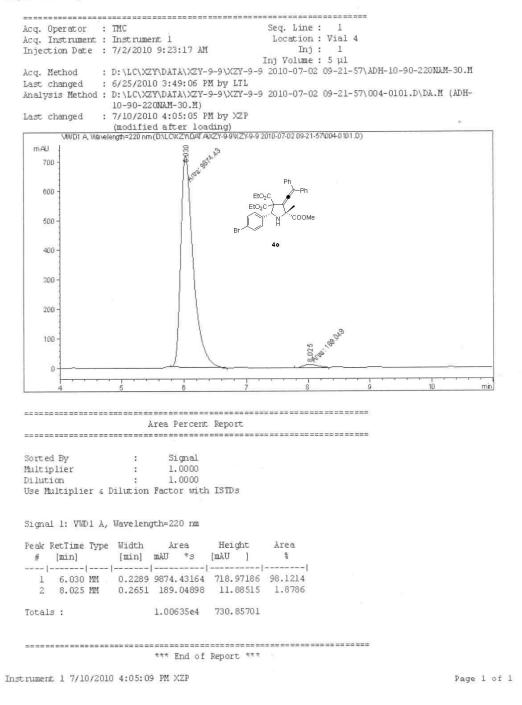
Acg. Instrument	: TMC Seq. Line : 2
	: Instrument l Location : Vial 1
Injection Date	: 7/3/2010 11:48:21 AM Inj: 1
less Methods	Inj Volume : 5 µl
Acq. Method	: D:\LC\TMC\DATE\TMC-2-76\TMC-2-76 2010-07-03 10-45-25\ADH-10-90-220NAM-30.M
Last changed	: 6/25/2010 3:49:06 PM by LTL 1 : D:\LC\XZY\DATA\XZY-9-12\TMC-2-76 2010-07-03 10-45-25\001-0201.D\DA.M (ADH-
Anarysis neonoc	10-90-220NAM-30.M)
Last changed	: 12/2/2010 10:18:38 AM by dxq
	(modified after loading)
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Data File D:\LC\XZY\DATA\XZY-9-11\XZY-9-11 2010-07-02 08-54-39\002-0101.D Sample Name: xzy-9-11A

Area Percent Report Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 A, Wavelength=220 nm Peak RetTime Type Width Area # [min] [min] min] [min] mAU 1 5.768 MM 0.2199 3735.38379 2 7.355 MM 0.2724 3686.45801								
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ker, Method : D: LCV2ZYDATAX027-9-11/XZY-9-11 2010-07-02 08-54-39\ADH-10-90-220NAM-30.N Last changed : 6/25/2010 3:49:06 PH by LTL Analyzis Method : D: LCVZYDATAX027-9-11/XZY-9-11 2010-07-02 08-54-39\002-0101.D\DA.M (ADH- 10-90-220NAM-30.N) Last changed : 7/10/2010 4:08:11 PH by XZP modelfield after loading) W014 (Museumofield active loading) w014 (Museumofield active loading) may be a standard active loading by the standa	Injection Dat	e : //2/201	U 8:55:00 AM					
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rument 1 7/10/2010 4:08:15 FM XZP Page 1	Sorted By Multiplier Dilution Use Multiplie Signal 1: VWD Peak RetTime # [min] 	: : : : : : : : : : : : : :	Area Percent Signal 1.0000 1.0000 h Factor with ngth=220 nm Area mAU *s 	Height [mAU] 263.09082 225.51962 508.61044	Area § 			8.5 mi
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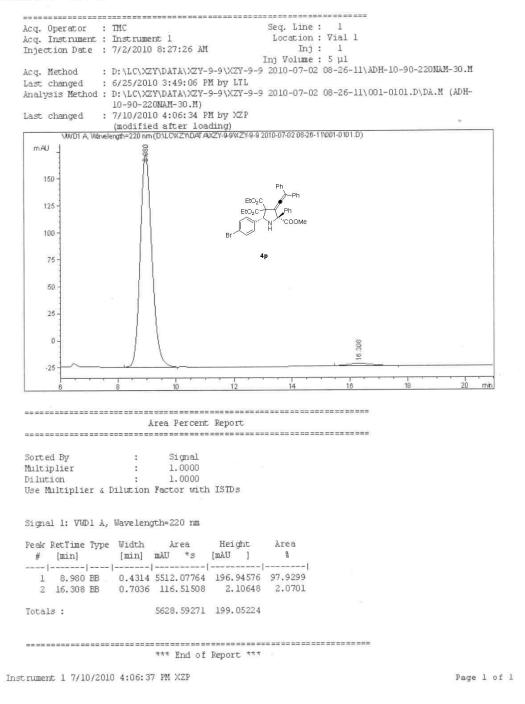
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Data File D:\LC\XZY\DATA\XZY-9-8\XZY-9-8 2010-06-29 19-09-46\031-0101.D Sample Name: xzy-9-8a

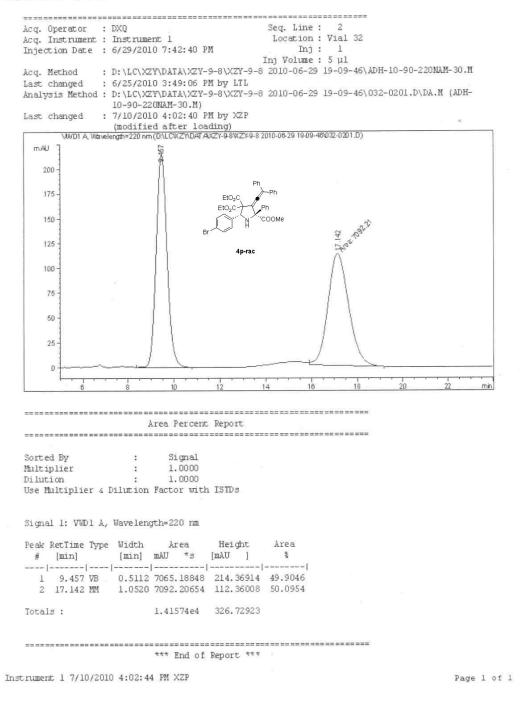
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Sorted H Multipl: Dilution Use Mult Signal . Peak Ret # [n 	By ier n tiplier & 1: VWD1 Å, tTime Type min] 	i i Dilution Waveleng Width [min] -1	Signal 1.0000 Factor with pth=220 nm Area mAU *s	Report ISTDs Height [mAU] 	årea % 50.5861		- <u>1</u> - <u>9</u>	âm.
Sorted H Multipl: Dilution Use Mult Signal . Peak Ret # [n 	By ier n tiplier & 1: VWD1 Å, tTime Type min] 	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	Signal 1.0000 1.0000 Factor with gth=220 nm Area mAU *s 	Report ISTDs Height [mAU] 	årea % 50.5861		- <u>1</u> - <u>0</u>	mit
Sorted H Multipl: Dilution Use Mult Signal . Peak Ret # [n 	By ier n tiplier a 1: VWD1 Å, tTime Type min] 5.939 MM 7.866 MM	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	Signal 1.0000 1.0000 Factor with pth=220 nm Area mAU *s 	Report ISTDs Height [mAU] 	årea % 50.5861		- I	mit
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Sorted H Multipl: Dilution Use Mult Signal : Peak Ret # [p 	Ey ier n tiplier & tTime Type min] 	: : Dilution , Waveleng e Width [min] -] 0.2266 0.3010	rea Percent Signal 1.0000 1.0000 Factor with Area MAU *s 5291.31348 5168.69629 1.04600e4	Report Height [mAU] 389.24362 286.20123 675.44485	Àrea % 50.5861 49.4139			min
Sorted H Multipl: Dilution Use Mult Signal : Peak Ret # [p 	Ey ier n tiplier & tTime Type min] 	: : Dilution , Waveleng e Width [min] -] 0.2266 0.3010	Signal 1.0000 1.0000 Factor with pth=220 nm Area mAU *s 5291.31348 5168.69629 1.04600e4	Report Height [mAU] 389.24362 286.20123 675.44485	Àrea % 		- <u>1</u> - <u>9</u>	min
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Data File D:\LC\XZY\DATA\XZY-9-9\XZY-9-9 2010-07-02 08-26-11\001-0101.D Sample Name: xzy-9-9B

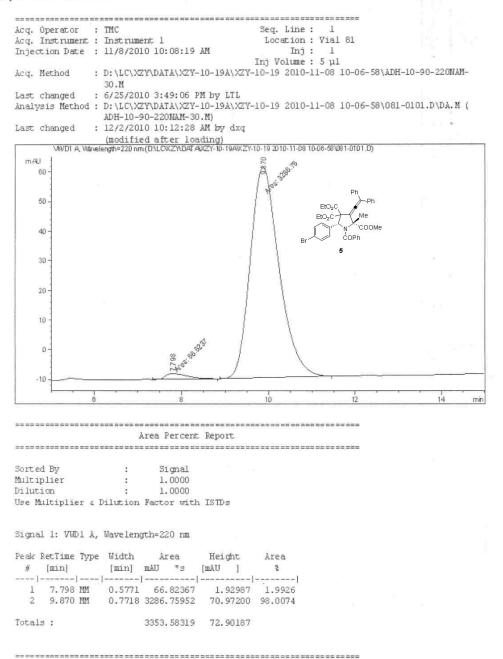


63

Data File D:\LC\XZY\DATA\XZY-9-8\XZY-9-8 2010-06-29 19-09-46\032-0201.D Sample Name: xzy-9-8c



Data File D:\LU\XZY\DATA\XZY-10-19A\XZY-10-19 Z010-11-08 10-05-58\001-0101.D Sample Name: XZY-10-19A



Instrument 1 12/2/2010 10:12:32 AM dxq

Data File D:\LC\XZY\DATA\XZY-10-13\XZY-10-13 2010-10-28 15-26-46\072-0201.D Sample Name: XZY-10-13Å

cq. Instrument njection Date	TMC Seq. Line : 2 Instrument 1 Location : Vial 72 10/28/2010 3:39:20 PM Inj : 1 Inj Volume : 5 µl	
cq. Method	D:\LC\XZY\DATA\XZY-10-13\XZY-10-13 2010-10-28 15-26-46\ADH-10-90-220NAM-85.M	
ast changed nalysis Method	7/29/2010 7:59:04 PM by dxq D:\LC\XZY\DATA\XZY-10-13\XZY-10-13 2010-10-28 15-26-46\072-0201.D\DA.M (
0.72	ADH-10-90-220NAM-85.M) 12/2/2010 10:05:51 AM by dxq (modified after loading)	
AND THE R.	length=220 nm (D/LCWZY/DAT AWZY-10-13/VZY-10-13 2010-10-28 15-26-46/072-0201 D)	
mAU]		
200 -	EtO ₂ C EtO ₂ C Me C COOME	
150 -	Hr COPh 5-rac	
100 -		
51 (s) 81		
50 -		
o		
1	4 6 8 10 12	mi
	Årea Percent Report	
Sorted By Multiplier Dilution Jse Multiplier 6	: Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs	
ignal 1: WWDI ۸	Wavelength=220 nm	
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Peak RetTime Typ # [min] 1 6.313 VV	e Width Area Height Area [min] mAU *s [mAU] %	
Peak RetTime Typ # [min] 1 6.313 VV	E Width Area Height Area [min] mAU *s [mAU] % 	

Instrument 1 12/2/2010 10:05:56 AM dxq