Supporting Information

Palladium-catalyzed asymmetric coupling cyclization of terminal γ-allenols with

aryl iodides

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General Information. All reactions were carried out in oven dried Schlenk tubes. $Pd(dba)_2$ was purchased from Aladdin; (R,R)-DACH Phenyl Trost Ligand L1 (98%) glove was purchased from Stream Chemicals and kept in box: (S)-tetrahydrofuran-2-carboxylic acid (97%) was purchased from Accela; K₃PO₄ was purchased from Acros and kept in glove box; 3 Å molecular sieves were purchased from Alfa Aesar and kept in glove box after activation (activated at 450 °C for 10 h in Muffle furnace; after cooling to 200 °C, transferred to the glove box to allow to cool to room temperature for use). CH₃CN, (CH₃)₃CCN, Et₃N, DMF and CH₃(CH₂)₃CN were dried over CaH₂ and distilled freshly before use. 1,4-Dioxane and toluene were dried over sodium wire and distilled freshly before use. Other reagents were used without further treatment. All the temperatures are referred to the oil baths used.

Experimental details and analytical data

1. Preparation of (R)-2-(1-phenylvinyl)tetrahydrofuran 2a (xx-5-141)



Typical procedure I: To a dry Schlenk tube were added 4,5-hexadien-1-ol **1a** (98.2 mg, 1 mmol), freshly distilled CH₃CN (6 mL), and (CH₃)₃CCN (4 mL). The solution was frozen with a liquid nitrogen bath, degassed for ten minutes and then thawed under argon. This cycle was repeated for three times. To another dry Schlenk tube were sequentially added K₃PO₄ (318.3 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.7 mg, 0.05 mmol), and (*R*,*R*)-DACH Phenyl Trost Ligand (51.9 mg, 0.075 mmol) in a glove box. This Schlenk tube was then taken out to the bench, which was followed by the addition of the above-mentioned degassed solution. The resulting mixture was then stirred at room temperature for 13 min, then frozen with a liquid nitrogen bath and iodobenzene (170 μ L, d = 1.81 g/cm³, 305.9 mg, 1.5 mmol) was added under argon. After repeating evacuating and refilling with argon for three times, the resulting mixture was stirred at 90 °C under the argon atmosphere with an

argon balloon. When the reaction was complete as monitored by TLC, it was filtered and washed with ethyl ether. The solvent was evaporated under vacuum, and the residue was purified by chromatography on silica gel (eluent: petroleum ether:ethyl ether = 20:1) to afford (*R*)-**2a** (136.0 mg, 78%) as an oil; 92% ee determined by HPLC analysis (AD-H column, rate = 0.8 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 230 nm, $t_R(\text{minor}) = 5.3$ min, $t_R(\text{major}) = 6.6$ min); $[\alpha]^{20}_D = -8.9$ (c = 2.62, ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 7.50-7.22 (m, 5 H, Ar-H), 5.37 (s, 1 H, one proton form H₂C=C), 5.30 (s, 1 H, one proton form H₂C=C), 4.87 (t, *J* = 6.9 Hz, 1 H, CHO), 4.10-3.97 (m, 1 H, one proton from OCH₂), 3.95-3.82 (m, 1 H, one proton from OCH₂), 2.18-2.01 (m, 1 H, one proton from CH₂CH₂), 1.98-1.82 (m, 2 H, two protons from CH₂CH₂), 1.71-1.55 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 149.6, 139.9, 128.2, 127.4, 126.7, 111.6, 80.0, 68.4, 31.7, 25.5; IR (neat, cm⁻¹): 2975, 2867, 1631, 1494, 1443, 1060; MS (70 ev, EI) *m/z* (%): 174 (M⁺, 100.00); HRMS Calcd for C₁₂H₁₄O (M⁺): 174.1045, Found: 174.1044.

The absolute configuration of the product 2a was determined to be *R* by comparison of the t_R of the authentic (*S*)-2a, which was in situ prepared by the Wittig methylation of (*S*)-phenyl(tetrahydrofuran-2-yl)methanone¹ derived from the reaction between (*S*)-tetrahydrofuran-2-carboxylic acid and phenylmagnesium bromide.²

PhBr + Mg
$$\xrightarrow{\text{THF}}$$
 PhMgBr \xrightarrow{O}_{O} \xrightarrow{O}_{O} \xrightarrow{O}_{O} Ph
THF, 0 °C 1 h 51% , 96% ee

To a 100 mL 3-necked flask equipped with a condenser and an addition funnel were added magnesium turnings (1.0208 g, 42 mmol) and anhydrous THF (40 mL) under N_2 . A solution of bromobenzene (5.4951 g, 35 mmol) in anhydrous THF (10 mL) was added through the addition funnel. The Grignard reaction was initiated with the addition of approximately 5 mL of the bromobenzene solution and a few crystal of iodine. The remaining bromobenzene solution was then added and the reaction was heated under reflux for 1 hour. Then it was cooled to 0 °C and a solution of (*S*)-tetrahydrofuran-2-carboxylic acid (1.1610 g, 10 mmol) in anhydrous THF (12.5

mL) was added in one portion with stirring. After 30 minutes at 0 °C, the reaction was warmed up to room temperature and quenched with aqueous hydrochloric acid (1 N, 10 mL). The organic layer was separated from the aqueous layer, which was extracted with ethyl ether $(3 \times 20 \text{ mL})$. The organic layer was washed with brine and dried over anhydrous magnesium sulfate. Filtration, evaporation and column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 5/1 to 3/1) afforded (S)-phenyl(tetrahydrofuran-2-yl)methanone (910.2 mg, 51%, 96% ee) (HPLC conditions: As-H column, rate = 0.7 mL/min, eluent: hexane/*i*-PrOH = 70:30, λ = 214 nm, $t_{\rm R}(\text{major}) = 7.2 \text{ min}, t_{\rm R}(\text{minor}) = 14.8 \text{ min});$ liquid; $[\alpha]_{\rm D}^{20} = +1.4 (c = 1.07, c = 1.07)$ CHCl₃); ¹H NMR (300 MHz, CDCl₃) & 8.04-7.90 (m, 2 H, Ar-H), 7.58-7.36 (m, 3 H, Ar-H), 5.29-5.16 (m, 1 H, OCH), 4.04-3.86 (m, 2 H, OCH₂), 2.33-2.17 (m, 1 H, one proton from CH₂CH₂), 2.14-2.00 (m, 1 H, one proton from CH₂CH₂), 1.99-1.84 (m, 2 H, two protons from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 198.5, 134.8, 133.1, 128.5, 128.4, 79.8, 69.2, 29.1, 25.4; IR (neat, cm⁻¹): 2976, 2952, 2873, 1689, 1597, 1580, 1449, 1226, 1178, 1100, 1058, 1003; MS (70 ev, EI) m/z (%): 176 (M⁺, 0.50), 71 (100); Anal. Calcd for C₁₁H₁₂O₂: C, 74.98; H, 6.86. Found: C, 74.81; H, 6.91.

Ph₃PCH₃Br + *n*-BuLi
$$\frac{\text{THF}}{-78 \,^{\circ}\text{C}} \xrightarrow[-78 \,^{\circ}\text{C}]{} \text{THF} \xrightarrow[-78 \,^{\circ}\text{C} \text{ to rt}]{} \text{S}^{\circ}\text{D} \text{S}^{\circ}\text{C} \text{ to rt}} \xrightarrow[-78 \,^{\circ}\text{C} \text{ to rt}]{} \text{S}^{\circ}\text{D} \text{S}^{\circ}\text{C} \text{ to rt}$$

To a slurry of $(C_6H_5)_3PCH_3Br$ (625.9 mg, 1.8 mmol) in THF (8 mL) cooled to -78 °C was added *n*-C₄H₉Li (2.5 M in hexane, 0.72 mL, 1.8 mmol). The resulting mixture was stirred at this temperature for 10 min. To this bright yellow solution was added a solution of (*S*)-phenyl(tetrahydrofuran-2-yl)methanone (308.8 mg, 1.8 mmol) in THF (8 mL). After being stirred at room temperature for 9.5 h, the solution was quenched with an aqueous solution of hydrochloric acid (1 N, 10 mL). The organic layer was separated and the aqueous layer was extracted with ethyl ether (3 × 10 mL). The combined organic layer was washed with brine and dried over anhydrous magnesium sulfate. Filtration, evaporation and column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 10/1) afforded (*S*)-**2a** (242.3 mg, 79%, 96% ee)

(HPLC conditions: AD-H column, rate = 0.8 mL/min, eluent: hexane/*i*-PrOH = 95:5, $\lambda = 230$ nm, $t_{\rm R}$ (major) = 5.5 min, $t_{\rm R}$ (minor) = 6.8 min); liquid; $[\alpha]^{20}{}_{\rm D}$ = +8.5 (c = 2.52, ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 7.48-7.20 (m, 5 H, Ar-H), 5.36 (dd, J = 1.5, 1.2 Hz, one proton form H₂C=C), 5.29 (s, 1 H, one proton form H₂C=C), 4.85 (t, J = 6.9 Hz, 1 H, CHO), 4.01 (dt, J = 8.1, 6.6 Hz, 1 H, one proton from OCH₂), 3.87 (dt, J = 8.1, 6.9 Hz, 1 H, one proton from OCH₂), 2.13-1.98 (m, 1 H, one proton from CH₂CH₂), 1.98-1.83 (m, 2 H, two protons from CH₂CH₂), 1.70-1.55 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 149.5, 139.8, 128.2, 127.4, 126.6, 111.5, 79.9, 68.3, 31.6, 25.5; IR (neat, cm⁻¹): 2987, 2850, 2867, 1715, 1495, 1445, 1080, 1061, 1028; MS (70 ev, EI) m/z (%): 174 (M⁺, 66.49), 71 (100); HRMS Calcd for C₁₂H₁₄O (M⁺): 174.1045, Found: 174.1044.

The following compounds **2b-2l** in Table 2 and Table 3 were prepared according to **Typical Procedure I**.

All the racemic products were also prepared according to this procedure in the absence of the chiral ligand and replacing $Pd(dba)_2$ with $Pd(PPh_3)_4$ in CH_3CN .

2. Preparation of (R)-2-(1-(4-methoxyphenyl)vinyl)tetrahydrofuran 2b (xx-6-20)



The reaction of **1a** (98.1 mg, 1.0 mmol), CH₃CN (4.8 mL), (CH₃)₃CCN (3.2 mL), K₃PO₄ (318.4 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.7 mg, 0.050 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (51.8 mg, 0.075 mmol), and 1-iodo-4-methoxybenzene (351.1 mg, 1.5 mmol) afforded (*R*)-**2b** (167.9 mg, 82%, 88% ee) (eluent: petroleum ether/ethyl ether = 20/1 to 10/1) (HPLC conditions: AD-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 230 nm, *t*_R(minor) = 12.2 min, *t*_R(major) = 13.9 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.38-7.28 (m, 2 H, Ar-H), 6.90-6.82 (m, 2 H, Ar-H), 5.29 (dd, *J* = 1.8, 1.5 Hz, 1 H, one proton from

H₂C=C), 5.24-5.22 (m, 1 H, one proton from H₂C=C), 4.83 (t, J = 6.9 Hz, 1 H, OCH), 4.02 (dt, J = 8.4, 6.6 Hz, 1 H, one proton from OCH₂), 3.88 (dt, J = 8.1, 7.0 Hz, 1 H, one proton from OCH₂), 3.81 (s, 3 H, CH₃), 2.15-2.02 (m, 1 H, one proton from CH₂CH₂), 1.96-1.85 (m, 2 H, two protons from CH₂CH₂), 1.71-1.57 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 159.0, 148.7, 132.2, 127.7, 113.5, 110.1, 80.0, 68.4, 55.1, 31.6, 25.5; IR (neat, cm⁻¹): 2973, 2869, 1608, 1510, 1461, 1245, 1179, 1106, 1061, 1032; MS (70 ev, EI) m/z (%): 204 (M⁺, 36.16), 133 (100); HRMS Calcd for C₁₃H₁₆O₂ (M⁺): 204.1150, Found: 204.1151.

3. Preparation of (*R*)-2-(1-(3,5-dimethylphenyl)vinyl)tetrahydrofuran 2c (xx-6-96)



The reaction of **1a** (98.0 mg, 1.0 mmol), CH₃CN (4.8 mL), (CH₃)₃CCN (3.2 mL), K₃PO₄ (318.3 mg, 1.5 mmol), 3 Å molecular sieve (400.1 mg), Pd(dba)₂ (28.8 mg, 0.050 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (51.9 mg, 0.075 mmol), and 1-iodo-3,5-dimethylbenzene (217 μ L, d = 1.608 g/cm³, 348.9 mg, 1.5 mmol) afforded (*R*)-**2c** (153.0 mg, 76%, 89% ee) (eluent: petroleum ether/ethyl ether = 30/1) (HPLC conditions: AD-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 230 nm, *t*_R(minor) = 7.5 min, *t*_R(major) = 9.0 min); liquid; ¹H NMR (300 MHz, CDCl₃) & 6.99 (s, 2 H, Ar-H), 6.93 (s, 1 H, Ar-H), 5.32 (dd, *J* = 1.8, 1.5 Hz, 1 H, one proton from H₂C=), 5.26 (s, 1 H, one proton from H₂C=), 4.85 (t, *J* = 7.1 Hz, 1 H, OCH), 4.03 (dt, *J* = 8.1, 6.8 Hz, 1 H, one proton from OCH₂), 3.88 (dt, *J* = 8.1, 7.1 Hz, 1 H, one proton from CH₂CH₂); 1.97-1.85 (m, 2 H, two protons from CH₂CH₂), 1.71-1.57 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) & 149.8, 139.9, 137.5, 129.0, 124.5, 111.0, 80.0, 68.3, 31.7, 25.5, 21.3; IR (neat, cm⁻¹): 2974, 2948, 2917, 2865, 1598, 1459, 1068; MS (70 ev, EI) *m/z* (%): 202 (M⁺, 76.16), 71 (100); HRMS Calcd

for C₁₄H₁₈O (M⁺): 202.1358, Found: 202.1359.

4. Preparation of (*R*)-2-(1-(4-*N*,*N*-diethylaminocarbonylphenyl)vinyl)tetrahydrofuran 2d (xx-6-43)



The reaction of 1d (98.2 mg, 1.0 mmol), CH₃CN (3.6 mL), (CH₃)₃CCN (2.4 mL), K₃PO₄ (318.5 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.7 mg, 0.050 mmol), (R,R)-DACH Phenyl Trost Ligand (51.8 mg, 0.075 mmol), and N,N-diethyl-4-iodobenzamide (454.8 mg, 1.5 mmol) afforded (R)-2d (205.0 mg, 75%, 90% ee) (eluent: petroleum ether/ethyl acetate = 3/1 to 2/1) (HPLC conditions: OJ-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 90:10, λ = 230 nm, $t_{\rm R}$ (major) = 14.4 min, $t_{\rm R}$ (minor) = 15.5 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.43-7.36 (m, 2 H, Ar-H), 7.35-7.27 (m, 2 H, Ar-H), 5.38 (dd, J = 1.8, 1.5 Hz, 1 H, one proton from $H_2C=$), 5.31 (s, 1 H, one proton from $H_2C=$), 4.82 (t, J=7.1 Hz, 1 H, OCH), 4.00 (dt, J = 8.1, 6.6 Hz, 1 H, one proton from OCH₂), 3.86 (dt, J = 8.1, 6.9 Hz, 1 H, one proton from OCH₂), 3.70-3.10 (br, 4 H, 2×CH₂), 2.14-1.98 (m, 1 H, one proton from CH_2CH_2 , 1.96-1.80 (m, 2 H, two protons from CH_2CH_2), 1.70-1.52 (m, 1 H, one proton from CH₂CH₂), 1.40-0.90 (br, 6 H, $2 \times CH_3$); ¹³C NMR (75.4 MHz, CDCl₃) δ 170.8, 148.6, 140.4, 136.0, 126.5, 126.1, 112.2, 79.7, 68.2, 43.1, 39.0, 31.4, 25.3, 13.9, 12.7; IR (neat, cm⁻¹): 2973, 2935, 2874, 1627, 1458, 1426, 1287, 1092, 1059, 1019; MS (70 ev, EI) m/z (%): 273 (M⁺, 37.38), 201 (100); HRMS Calcd for C₁₇H₂₃NO₂ (M⁺): 273.1729, Found: 273.1730.

5. Preparation of (*R*)-2-(1-(3-*N*,*N*-diethylaminocarbonylphenyl)vinyl)tetrahydrofuran 2e (xx-6-58)

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The reaction of 1d (98.0 mg, 1.0 mmol), CH₃CN (3.6 mL), (CH₃)₃CCN (2.4 mL), K₃PO₄ (318.3 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.7 mg, 0.050 mmol), (R,R)-DACH Phenyl Trost Ligand (51.9 mg, 0.075 mmol), and N,N-diethyl-3-iodobenzamide (454.8 mg, 1.5 mmol) afforded (R)-2e (235.7 mg, 86%, 91% ee) (eluent: petroleum ether/ethyl acetate = 5/1 to 3/1) (HPLC conditions: OD-H column, rate = 1.0 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 214 nm, $t_{\rm R}$ (major) = 24.2 min, $t_{\rm R}$ (minor) = 25.9 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.45-7.25 (m, 4 H, Ar-H), 5.40 (s, 1 H, one proton from $H_2C=$), 5.32 (s, 1 H, one proton from $H_2C=$), 4.83 (t, J = 6.9 Hz, 1 H, OCH), 4.05-3.94 (m, 1 H, one proton from OCH₂), 3.94-3.82 (m, 1 H, one proton from OCH₂), 3.70-3.10 (br, 4 H, 2×CH₂), 2.15-2.00 (m, 1 H, one proton from CH₂CH₂), 1.98-1.80 (m, 2 H, two protons from CH₂CH₂), 1.70-1.56 (m, 1 H, one proton from CH₂CH₂), 1.40-0.96 (br, 6 H, $2 \times CH_3$); ¹³C NMR (75.4 MHz, CDCl₃) § 171.0, 148.8, 139.9, 137.1, 128.2, 127.3, 125.2, 124.5, 112.3, 79.8, 68.3, 43.2, 39.1, 31.5, 25.4, 14.1, 12.8; IR (neat, cm⁻¹): 2973, 2946, 2871, 1629, 1458, 1432, 1381, 1283, 1220, 1098, 1062; MS (70 ev, EI) m/z (%): 273 (M⁺, 31.34), 201 (100); HRMS Calcd for C₁₇H₂₃NO₂ (M⁺): 273.1729, Found: 273.1728.

6. Preparation of (R)-2-(1-(4-iodophenyl)vinyl)tetrahydrofuran 2f (xx-6-40)



The reaction of **1d** (98.1 mg, 1.0 mmol), CH₃CN (2.4 mL), (CH₃)₃CCN (1.6 mL), K₃PO₄ (318.5 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.8 mg, 0.050 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (51.7 mg, 0.075 mmol), and 1,4-diiodobenzene (395.8 mg, 1.2 mmol) afforded (*R*)-**2f** (179.3 mg, 60%, 88% ee)

(eluent: petroleum ether/ethyl ether = 40/1 to 20/1) (HPLC conditions: OJ-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 98:2, λ = 230 nm, t_R (major) = 13.4 min, t_R (minor) = 14.8 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.69-7.60 (m, 2 H, Ar-H), 7.17-7.09 (m, 2 H, Ar-H), 5.37 (dd, J = 1.2, 0.9 Hz, 1 H, one proton from H₂C=), 5.29 (dd, J = 1.2, 0.9 Hz, 1 H, one proton from H₂C=), 4.78 (t, J = 6.9 Hz, 1 H, OCH), 4.00 (dt, J = 8.1, 6.6 Hz, 1 H, one proton from OCH₂), 3.87 (dt, J = 8.1, 7.1 Hz, 1 H, one proton from OCH₂), 2.12-2.00 (m, 1 H, one proton from CH₂CH₂), 1.96-1.81 (m, 2 H, two protons from CH₂CH₂), 1.70-1.52 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 148.5, 139.3, 137.2, 128.6, 112.4, 93.0, 79.8, 68.4, 31.5, 25.5; IR (neat, cm⁻¹): 2973, 2947, 2865, 1484, 1385, 1178, 1065, 1054, 1004; MS (70 ev, EI) m/z (%): 300 (M⁺, 34.41), 71 (100); HRMS Calcd for C₁₂H₁₃OI (M⁺): 300.0011, Found: 300.0012.

7. Preparation of (R)-2-(1-(4-bromophenyl)vinyl)tetrahydrofuran 2g (xx-6-34)



The reaction of **1a** (98.1 mg, 1.0 mmol), CH₃CN (2.4 mL), (CH₃)₃CCN (1.6 mL), K₃PO₄ (318.3 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.8 mg, 0.050 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (51.9 mg, 0.075 mmol), and 1-bromo-4-iodobenzene (424.2 mg, 1.5 mmol) afforded (*R*)-**2g** (197.1 mg, 78%, 85% ee) (eluent: petroleum ether/ethyl acetate = 40/1 to 20/1) (HPLC conditions: OJ-H column, rate = 0.6 mL/min, eluent: hexane/*i*-PrOH = 98:2, λ = 214 nm, *t*_R(major) = 10.5 min, *t*_R(minor) = 11.9 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.54-7.38 (m, 2 H, Ar-H), 7.32-7.20 (m, 2 H, Ar-H), 5.42-5.34 (m, 1 H, one proton from H₂C=), 5.29 (s, 1 H, one proton from H₂C=), 4.79 (t, *J* = 6.8 Hz, 1 H, OCH), 4.06-3.94 (m, 1 H, one proton from OCH₂), 3.92-3.80 (m, 1 H, one proton from OCH₂), 2.16-2.00 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 148.4, 138.7, 131.3, 128.4, 121.4, 112.4, 79.9, 68.4, 31.5, 25.5; IR (neat, cm⁻¹): 2975, 2948, 2868, 1630, 1588, 1487, 1390, 1179, 1073, 1058, 1008; MS (70 ev, EI) m/z (%): 254 [M⁺(⁸¹Br), 12.86], 252 [M⁺(⁷⁹Br), 13.32], 71 (100); HRMS Calcd for C₁₂H₁₃O⁷⁹Br (M⁺): 252.0150, Found: 252.0151.

8. Preparation of (R)-2-(1-(4-acetylphenyl)vinyl)tetrahydrofuran 2h (xx-6-25)



The reaction of 1d (98.2 mg, 1.0 mmol), CH₃CN (3.6 mL), (CH₃)₃CCN (2.4 mL), K₃PO₄ (318.5 mg, 1.5 mmol), 3 Å molecular sieve (400 mg), Pd(dba)₂ (28.7 mg, 0.050 mmol), (R,R)-DACH Phenyl Trost Ligand (51.9 mg, 0.075 mmol), and 1-(4-iodophenyl)ethanone (369.0 mg, 1.5 mmol) afforded (R)-2h (156.7 mg, 72%, 87% ee) (eluent: petroleum ether/ethyl acetate = 20/1 to 10/1) (HPLC conditions: AS-H column, rate = 1.0 mL/min, eluent: hexane/*i*-PrOH = 98:2, λ = 214 nm, $t_{\rm R}({\rm minor}) = 21.1 {\rm min}, t_{\rm R}({\rm major}) = 23.6 {\rm min});$ liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.95-7.87 (m, 2 H, Ar-H), 7.52-7.43 (m, 2 H, Ar-H), 5.46 (dd, J = 1.5, 1.2 Hz, 1 H, one proton from $H_2C=$), 5.38 (s, 1 H, one proton from $H_2C=$), 4.85 (t, J = 6.9 Hz, 1 H, OCH), 4.01 (dt, J = 8.1, 6.6 Hz, 1 H, one proton from OCH₂), 3.88 (dt, J = 8.1, 7.1 Hz, 1 H, one proton from OCH₂), 2.59 (s, 3 H, CH₃), 2.16-2.02 (m, 1 H, one proton from CH₂CH₂), 1.98-1.84 (m, 2 H, two protons from CH₂CH₂), 1.68-1.53 (m, 1 H, one proton from CH₂CH₂); ¹³C NMR (75.4 MHz, CDCl₃) δ 197.6, 148.7, 144.6, 136.1, 128.3, 126.9, 113.6, 79.8, 68.4, 31.6, 26.5, 25.5; IR (neat, cm⁻¹): 2975, 2872, 1681, 1604, 1405, 1358, 1266, 1187, 1057; MS (70 ev, EI) m/z (%): 216 (M⁺, 65.87), 71 (100); HRMS Calcd for $C_{14}H_{16}O_2$ (M⁺): 216.1150, Found: 216.1151.

9. Preparation of (R)-2-(1-(4-ethoxycarbonylphenyl)vinyl)tetrahydrofuran 2i (xx-6-98) Electronic Supplementary Material (ESI) for Chemical Communications This journal is © The Royal Society of Chemistry 2013



The reaction of **1a** (49.0 mg, 0.5 mmol), CH₃CN (1.8 mL), (CH₃)₃CCN (1.2 mL), K₃PO₄ (159.1 mg, 0.75 mmol), 3 Å molecular sieve (200 mg), Pd(dba)₂ (14.4 mg, 0.025 mmol), (R,R)-DACH Phenyl Trost Ligand (25.8 mg, 0.0375 mmol), and ethyl 4-iodobenzoate (125 μ L, d = 1.66 g/cm³, 207.1 mg, 0.75 mmol) afforded the mixture of 2i and dba (eluent: petroleum ether/ethyl acetate = 10:1), which couldn't be separated via column chromatography on silica gel. Then MeOH (3 mL) and NaBH₄ (3.0 mg, 0.08 mmol) were added and the resulting mixture was stirred at RT for half an hour. The solvent was evaporated under vacuum, and the residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to afford (R)-2i (81.8 mg, 67%, 89% ee) (HPLC conditions: AS-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 98:2, λ = 230 nm, $t_{\rm R}$ (minor) = 15.9 min, $t_{\rm R}$ (major) = 19.0 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 8.04-7.94 (m, 2 H, Ar-H), 7.50-7.40 (m, 2 H, Ar-H), 5.45 (dd, J = 1.6, 1.6 Hz, 1 H, one proton from H₂C=), 5.38 (s, 1 H, one proton from H₂C=), 4.86 (t, J = 6.9 Hz, 1 H, OCH), 4.37 (q, J = 7.2 Hz, 2 H, $COOCH_2$, 4.02 (dt, J = 8.1, 6.6 Hz, 1 H, one proton from OCH_2), 3.93-3.83 (m, 1 H, one proton from OCH₂), 2.16-2.02 (m, 1 H, one proton from CH₂CH₂), 1.98-1.84 (m, 2 H, two protons from CH_2CH_2), 1.68-1.53 (m, 1 H, one proton from CH_2CH_2), 1.39 $(t, J = 7.2 \text{ Hz}, 3 \text{ H}, \text{CH}_3)$; ¹³C NMR (75.4 MHz, CDCl₃) δ 166.3, 148.7, 144.3, 129.43, 129.36, 126.6, 113.3, 79.7, 68.4, 60.8, 31.5, 25.5, 14.2; IR (neat, cm⁻¹): 2980, 2870, 1714, 1608, 1367, 1273, 1181, 1101, 1060, 1019; MS (70 ev, EI) m/z (%): 246 (M⁺, 15.95), 71 (100); HRMS Calcd for $C_{15}H_{18}O_3(M^+)$: 246.1256, Found: 246.1257.

10. Preparation of (*R*)-3,3-dimethyl-2-(1-phenylvinyl)tetrahydrofuran 2j (xx-6-99)

Electronic Supplementary Material (ESI) for Chemical Communications This journal is © The Royal Society of Chemistry 2013



The reaction of **1b** (63.2 mg, 0.5 mmol), CH₃CN (1.2 mL), (CH₃)₃CCN (0.8 mL), K₃PO₄ (159.2 mg, 0.75 mmol), 3 Å molecular sieve (200 mg), Pd(dba)₂ (14.4 mg, 0.025 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (25.8 mg, 0.0375 mmol), and iodobenzene (85 μ L, d = 1.81 g/cm³, 153.9 mg, 0.75 mmol) afforded (*R*)-**2j** (79.2 mg, 78%, 88% ee) (eluent: petroleum ether/ethyl ether = 30/1) (HPLC conditions: AD-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 230 nm, *t*_R(minor) = 7.7 min, *t*_R(major) = 12.7 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.38-7.21 (m, 5 H, Ar-H), 5.39 (dd, *J* = 2.1, 1.5 Hz, 1 H, one proton from H₂C=), 5.25 (dd, *J* = 2.1, 0.9 Hz, 1 H, one proton from H₂C=), 4.59-4.55 (m, 1 H, OCH), 4.06-3.89 (m, 2 H, OCH₂), 1.91-1.72 (m, 2 H, CH₂), 0.80 (s, 3 H, CH₃), 0.76 (s, 3 H, CH₃); ¹³C NMR (75.4 MHz, CDCl₃) δ 147.5, 141.2, 128.1, 127.3, 127.2, 113.5, 87.4, 65.5, 41.9, 41.4, 26.6, 22.7; IR (neat, cm⁻¹): 2961, 2933, 2871, 1632, 1574, 1494, 1464, 1386, 1163, 1113, 1064, 1030, 1004; MS (70 ev, EI) *m/z* (%): 202 (M⁺, 75.38), 70 (100); HRMS Calcd for C₁₄H₁₈O (M⁺): 202.1358, Found: 202.1359.

11. Preparation of (*R*)-4,4-dimethyl-2-(1-phenylvinyl)tetrahydrofuran 2k (xx-6-97)



The reaction of **1c** (62.8 mg, 0.5 mmol), CH₃CN (1.8 mL), (CH₃)₃CCN (1.2 mL), K₃PO₄ (159.2 mg, 0.75 mmol), 3 Å molecular sieve (200 mg), Pd(dba)₂ (14.6 mg, 0.025 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (25.8 mg, 0.0375 mmol), and iodobenzene (85 μ L, d = 1.81 g/cm³, 153.9 mg, 0.75 mmol) afforded (*R*)-**2k** (82.0 mg, 81%, 85% ee) (eluent: petroleum ether/ethyl ether = 30/1) (HPLC conditions: AD-H S12

column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 230 nm, $t_R(minor)$ = 8.0 min, $t_R(major)$ = 9.5 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.41-7.21 (m, 5 H, Ar-H), 5.44 (dd, *J* = 1.5, 1.5 Hz, 1 H, one proton from H₂C=), 5.27 (dd, *J* = 1.2, 1.2 Hz, 1 H, one proton from H₂C=), 5.03-4.95 (m, 1 H, OCH), 3.65 (d, *J* = 7.8 Hz, 1 H, one proton from OCH₂), 3.61 (d, *J* = 8.4 Hz, 1 H, one proton from OCH₂), 1.92 (dd, *J* = 12.3, 7.2 Hz, 1 H, one proton from CH₂), 1.48 (dd, *J* = 12.2, 9.2 Hz, 1 H, one proton from CH₂), 1.14 (s, 3 H, CH₃), 1.06 (s, 3 H, CH₃); ¹³C NMR (75.4 MHz, CDCl₃) δ 149.9, 139.9, 128.2, 127.4, 126.7, 110.9, 80.4, 80.1, 47.1, 39.9, 26.5, 26.2; IR (neat, cm⁻¹): 2957, 2869, 1495, 1465, 1368, 1285, 1059, 1028; MS (70 ev, EI) *m/z* (%): 202 (M⁺, 50.07), 103 (100); HRMS Calcd for C₁₄H₁₈O (M⁺): 202.1358, Found: 202.1359.

12. Preparation of (*R*)-2,2-dimethyl-5-(1-phenylvinyl)tetrahydrofuran 21 (xx-6-100)



The reaction of **1d** (63.3 mg, 0.5 mmol), CH₃CN (2.0 mL), (CH₃)₃CCN (1.35 mL), K₃PO₄ (159.3 mg, 0.75 mmol), 3 Å molecular sieve (200 mg), Pd(dba)₂ (14.4 mg, 0.025 mmol), (*R*,*R*)-DACH Phenyl Trost Ligand (25.8 mg, 0.0375 mmol), and iodobenzene (85 μ L, d = 1.81 g/cm³, 153.9 mg, 0.75 mmol) afforded (*R*)-**2l** (80.5 mg, 79%, 86% ee) (eluent: petroleum ether/ethyl ether = 30/1) (HPLC conditions: AD-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 230 nm, *t*_R(minor) = 7.8 min, *t*_R(major) = 9.9 min); liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.42-7.22 (m, 5 H, Ar-H), 5.45 (dd, *J* = 1.8, 1.5 Hz, 1 H, one proton from H₂C=), 5.30-5.25 (m, 1 H, one proton from H₂C=), 4.97-4.88 (m, 1 H, OCH), 2.23-2.10 (m, 1 H, one proton from CH₂CH₂), 1.82-1.63 (m, 3 H, three protons from CH₂CH₂), 1.35 (s, 3 H, CH₃), 1.32 (s, 3 H, CH₃); ¹³C NMR (75.4 MHz, CDCl₃) δ 150.3, 140.2, 128.1, 127.3, 126.8, 111.5, 81.3, 79.6, 38.3, 32.6, 28.8, 28.0; IR (neat, cm⁻¹): 2969, 2869, 1495, 1459, 1379,

1248, 1147, 1075, 1051; MS (70 ev, EI) *m/z* (%): 202 (M⁺, 66.02), 81 (100); HRMS Calcd for C₁₄H₁₈O (M⁺): 202.1358, Found: 202.1356.

13. One-Gram-Scale-Preparation of (R)-2-(1-(4'-bromobiphenyl-4-yl)vinyl)-

tetrahydrofuran 2m (xx-6-86)



The reaction of **1a** (1.0013 g, 10.2 mmol), CH₃CN (37 mL), (CH₃)₃CCN (25 mL), $K_{3}PO_{4}$ (3.2480 g, 15.3 mmol), 3 Å molecular sieve (4.0803 g), Pd(dba)₂ (293.2 mg, 0.51 mmol), (R,R)-DACH Phenyl Trost Ligand (528.2 mg, 0.765 mmol), and 4-bromo-4'-iodobiphenyl (4.3940 g, 12.2 mmol) afforded (R)-2m and dba (eluent: petroleum ether/ethyl acetate = 20:1 to 10:1), which couldn't be separated via column chromatography on silica gel. Then MeOH (25 mL) and NaBH₄ (100.0 mg, 2.6 mmol) were added and the resulting mixture was stirred at RT for half an hour. The solvent was evaporated under vacuum, and the residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) to afford (R)-2m (2.8632 g, 85%, 90% ee) (HPLC conditions: IC column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 95:5, λ = 214 nm, $t_{\rm R}$ (minor) = 14.3 min, $t_{\rm R}$ (major) = 15.1 min); solid; melting point: 98 °C (petroleum/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 7.78-7.30 (m, 8 H, Ar-H), 5.40 (s, 1 H, one proton from $H_2C=$), 5.36 (s, 1 H, one proton from H₂C=), 4.88 (t, 1 H, J = 6.9 Hz, OCH), 4.12-3.98 (m, 1 H, one proton from OCH₂), 3.98-3.81 (m, 1 H, one proton from OCH₂), 2.20-2.02 (m, 1 H, one proton from CH₂CH₂), 2.00-1.84 (m, 2 H, two protons from CH₂CH₂), 1.72-1.57 (m, 1 H, one proton from CH₂CH₂); 13 C NMR (75.4 MHz, CDCl₃) δ 148.8, 139.5, 139.1, 138.9, 131.8, 128.4, 127.1, 126.6, 121.5, 111.8, 79.8, 68.4, 31.7, 25.5; IR (neat, cm⁻¹): 3037, 2951, 2861, 1479, 1191, 1107, 1061, 1035; MS (70 ev, EI) m/z (%): 330 $(M^{+}(^{81}Br), 25.56), 328 (M^{+}(^{79}Br), 25.33), 71 (100);$ Anal. Calcd for $C_{18}H_{17}BrO$: C, 65.67; H, 5.20. Found: C, 65.70; H, 5.23.

References:

- (1) Daniel J.; Lindsay A.; Jon T. Org. Lett. 2012, 14, 378.
- (2) Eric, J.; Jeffrey, A. J. Heterocyclic Chem. 1995, 32, 109.





				,	
Operator:dell	Timebase:U-3000	Sequence:LSH	2		

Page 1-1 2010-3-27 3:52 上台

Sample Name: Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-5-141 419 unknown test test 2010-3-27 : 9.45	3:37			Inj Ci W Bi Di Si Si	lection Volume: hannel: 'avelength: andwidth: ilution Factor: ample Weight: ample Amount:	20.0 UV_VIS_ 230 n.a. 1.0000 1.0000 1.0000
600 LSH #415			X	x-5-141			UV_VIS_1 WVL:230 nm
500 400 300	Ph					2 - 6.573	
200							
100							
0					1 - 5.533		
1							i

No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре
	min		mAU	mAU*min	%		
1	5.53	n.a.	25.389	2.747	3.94	n.a.	BMB
2	6.57	n.a.	528.895	66.969	96.06	n.a.	BMB
Total:			554.284	69.715	100.00	0.000	

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

Page 1-1 2010-3-28 5:02 下台

Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	421 unknown test test 2010-3-27 5:08 7.08			Channel: Channel: Waveleng Bandwidth Dilution Fa Sample W Sample A	th: n: actor: /eight: mount:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
400 LSH #417 MAU		XX-	5-30			UV_VIS_1 WVL:230 nm
350 300 250 0					1 - 5.447	2 - 6.467
00-						

No.	Ret.Time	Peak Na	ame Height	Area	Rel.Area	Amount	Туре
	min		mAU	mAU*min	%		
1	5.45	n.a.	336.012	37.272	49.62	n.a.	BMB
2	6.47	n.a.	300.891	37.848	50.38	n.a.	BMB
Total:			636.902	75.120	100.00	0.000	
			`				

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)





XX-6-89



实验时间: 2012-12-25, 14:18:02 报告时间: 2012-12-25, 14:40:12 谱图文件:F:\slf\xiexi\2012-12-25\XX-6-89\新建文件夹\XX-6-89-As-h-70+30-0.7-214.org

XX-6-87

实验时间: 2012-12-25, 13:04:08 报告时间: 2012-12-25, 14:19:49 谱图文件:F:\slf\xiexi\2012-12-25\新建文件夹\XX-6-87-As-h-70+30-0.7-214.org

实验内容简介: As-h 70:30 214nm 0.7ml/min



峰号	峰名	保留时间	峰高	峰面积	含量
1		7.223	1023824.438	11555903.000	49.1567
2		14.657	409319.656	11952417.000	50.8433
总计			1433144.094	23508320.000	100.0000





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216 xx-6-90 AD-H-95	-5-0.8-230		
Sample Name:	xx-6-90	Injection Volume:	20.0
Sample Type:	unknown	Wavelength:	214
Control Program:	test	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2012-12-20 9:10	Sample Weight:	1.0000
Run Time (min):	10.18	Sample Amount:	1.0000



No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре	
	min		mAU	mAU*min	%			_
1	5.52	n.a.	181.700	18.336	97.98	n.a.	BMB	
2	6.85	n.a.	3.135	0.378	2.02	n.a.	BMB	
Total:			184.835	18.714	100.00	0.000		

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

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217 xx-5-30 AD-H-95-	5-0.8-230		
Sample Name:	xx-5-30	Injection Volume:	20.0
Sample Type:	unknown	Wavelength:	214
Control Program:	test	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2012-12-20 9:21	Sample Weight:	1.0000
Run Time (min):	7.94	Sample Amount:	1.0000



No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре	
	min		mAU	mAU*min	%			
1	5.44	n.a.	460.444	47.093	49.94	n.a.	BMB	
2	6.75	n.a.	385.432	47.208	50.06	n.a.	BMB	_
Total:			845.876	94.301	100.00	0.000		

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)





148 xx-6-20 AD-H-95-5-0.5-230 Sample Name: xx-6-20 Injection Volume: 20.0 Vial Number: 282 Channel: UV_VIS_1 Sample Type: unknown Wavelength: 230 Control Program: test Bandwidth: n.a. Quantif. Method: test Dilution Factor: 1.0000 Recording Time: 2012-6-18 23:28 Sample Weight: 1.0000 Run Time (min): 15.41 Sample Amount: 1.0000 600 xx #148 mAU xx-6-20 UV_VIS_1 WVL:230 nm 2 - 13.853 500-OCH3 400 300 200 100-1 - 12.200 0 -100 mir 2.0 4.0 6.0 8.0 10.0 12.0 14.0 15.4

No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре
	min		mAU	mAU*min	%		
1	12.20	n.a.	40.454	9.025	6.20	n.a.	BMB
2	13.85	<u>n.a</u> .	539.533	136.533	93.80	n.a.	BMB
Total:			579.987	145.558	100.00	0.000	

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

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No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	12.19	n.a.	613.214	139.691	49.80	n.a.	BMB
2	13.83	n.a.	552.193	140.787	50.20	n.a.	BMB
Total:			1165.407	280.478	100.00	0.000	

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

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Page 1-1 2013-1-15 10:22 上午

240 xx-6-96 AD-H-95	5-5-0.5-230			
Sample Name: Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-6-96 374 unknown test test 2013-1-14 21:00 10.53		Injection Volume: Channel: Wavelength: Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
900 xx #240 mAU 800 700 600		xx-6-96	2	UV_VIS_1 WVL:230 nm - 8.987

No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	7.47	n.a.	55.294	7.821	5.49	n.a.	BMB
2	8.99	n.a.	806.216	134.644	94.51	n.a.	BMB
Total:			861.510	142.465	100.00	0.000	

5.0

6.0

7.0

4.0

1 - 7.467

8.0

9.0

default/Integration

500-

400-

300-

200-

0

-100

2.0

3.0

1.0

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

min

10.5

300- 200- 100- - 100- 0- - 100- 0- - 100- 0- - 100- 0- - 100- - - 100- - - 100- - - -	, min) 11.
	min
300- 200- 100- 0-	
300- 200- 100-	
300-	
200-	
300-	
400- 0	
500-	
600-	
700 mAU V 1 - 7.440	VVL:230 nm
700 xx #242 xx-6-13	UV_VIS_1
Run Time (min): 11.29 Sample Amount:	1.0000
Quantif. Method: test Dilution Factor: Constraint Sample Weight: Const	1.0000 1.0000
Control Program: test Bandwidth:	230 n.a.
Vial Number: 376 Channel: Vial Number: 476 Channel:	UV_VIS_1

561.231

1196.614

50.49

100.00

94.738

187.636

default/Integration

2

Total:

n.a.

8.96

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

n.a.

0.000

BMB




Operator:dell	Timebase:U-3000	Sequence:xx
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185 x OJ-F	x -6-43 I-90-10-0	0.5-230					
Sample Vial Nun Sample Control I Quantif. Recordir Run Tim	Name: nber: Type: Program: Method: ng Time: ne (min):	xx-6-43 319 unknown test test 2012-7-1 19:07 16.99			Injection Volu Channel: Wavelength: Bandwidth: Dilution Fact Sample Weig Sample Amo	ume: or: ght: bunt:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
800 xx f mA 700 600 500 400 200 200	1185 U Co	CONEt2	xx-6-43			1 - 14	UV_VIS_1 WVL:230 nm 1.420
-100 0.0	2.0	4.0 6.0	8.0	10.0	12.0	14.0	17.0
No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре

No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Type
	min		mAU	mAU*min	%		
1	14.42	n.a.	723.462	211.462	95.24	n.a.	BMB
2	15.52	n.a.	35.527	10.557	4.76	n.a.	BMB
Total:	1010		758.989	222.019	100.00	0.000	

default/Integration

Operator:dell Timebase:U-3000 Sequence:xx

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186 xx-6-42				
OJ-H-90-10-	-0.5-230			
Sample Name: Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-6-42 320 unknown test test 2012-7-1 19:24 18.19		Injection Volume: Channel: Wavelength: Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
450 xx #186 [modified mAU 400 C 350 200 250 200 250 200 250 200 250 200 250 200 250 200 250 200 250 25	1 by dell] ONEt2	xx-6-42	1 - 14.4	UV VIS 1 WVL:230 nm 53 2-15.520
-50	4.0 6.0	8.0 10.0	12.0 14.0	16.0 18.

No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	гуре
	min		mAU	mAU*min	%		
1	14.45	n.a.	389.630	112.154	50.12	n.a.	BMB*
2	15.52	n.a.	362.201	111.598	49.88	n.a.	BMB*
Total	10.02		751.831	223.752	100.00	0.000	
Total.							

default/Integration

Chromeleon (c) Dionex 1996-2006 Version 6.80 SR5 Build 2413 (137116)

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xx-6-58-od-h-95-5-1-214

实验时间: 2012/7/17,13:18:23 谱图文件:D:\zhuguangjiong\xx\20120717\xx-6-58-od-95-5-1-214.org 报告时间: 2012/7/17, 15:07:55

实验内容简介: od-h 95/5 1ml/min 214nm



		分	· 析结果表			
峰号	峰名	保留时间	峰高	峰面积	含量	
1		24.212	919309.313	30821372.000	95.4200	
2		25.855	40718.965	1479367.375	4.5800	
总计			960028.277	32300739.375	100.0000	

PDF 文件使用 "pdfFactorv Pro" 试用版本创建 www.fineprint.cn

xx-6-52-od-h-95-5-1-214

实验时间: 2012/7/17,12:32:03 谱图文件:D:\zhuguangjiong\xx\20120717\xx-6-52-od-95-5-1-214.org 报告时间: 2012/7/17, 15:06:39

实验内容简介: od-h 95/5 1ml/min 214nm



		分	· 析结果表		
峰号	峰名	保留时间	峰高	峰面积	含量
1		24.835	528172.000	17470038.000	49.9039
2		26.257	466627.875	17537294.000	50.0961
总计			994799.875	35007332.000	100.0000

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Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-6-40 310 unknown test test 2012-6-30 19:45 16.21		Injection Volume: Channel: Wavelength: Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
800 <u>xx #176</u> mAU 700- 600- 500- 400- 300-		xx-6-40	1 - 13	UV VIS 1 WVL:230 nm 3.360
200-				I

No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре
	min		mAU	mAU*min	%		
1	13.36	n.a.	742.529	186.322	93.90	n.a.	BMB
2	14.83	n.a.	45.100	12.098	6.10	n.a.	BMB
Total:			787.629	198.421	100.00	0.000	

default/Integration

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175 xx-6-36 OJ-H-98-2-	0.5-230		
Sample Name: Vial Number: Sample Type:	xx-6-36 309	Injection Volume: Channel: Wavelength:	20.0 UV_VIS_1 230
Control Program:	test	Bandwidth:	n.a.
Quantif. Method: Recording Time: Run Time (min):	test 2012-6-30 19:27 17.24	Dilution Factor: Sample Weight: Sample Amount:	1.0000 1.0000 1.0000



No.	Ret.Time		Peak Name	Height	Area	Rel.Area	Amount	Туре
	min			mAU	mAU*min	%		
1	13.48	n.a.		477.285	118.492	49.99	n.a.	BMB
2	14.99	n.a.		430.028	118.542	50.01	n.a.	BMB
Total:				907.313	237.035	100.00	0.000	

default/Integration





xx-6-34

实验时间: 2012-06-28,13:17:07 报告时间: 2012-06-28,13:20:28 谱图文件:F:\slf\xiexi\2012-06-28\XX-6-34\新建文件夹(2)\XX-6-34-0J-h-98-2-0.6-214.org

实验内容简介: 0J-H 98:2 214nm 0.6ml/min



峰号	峰名	保留时间	峰高	峰面积	含量	
1		10.490	709267.063	7867080.500	92.4552	
2		11.857	39949.730	641992.688	7.5448	
			749216.793	8509073.188	100.0000	

xx-6-35

实验时间: 2012-06-28,12:26:16 报告时间: 2012-06-28,12:27:30 谱图文件:F:\slf\xiexi\2012-06-28\XX-6-35\新建文件夹\XX-6-35-0J-h-98-2-0.6-214.org

实验内容简介: 0J-H 98:2 214nm 0.6ml/min



峰号	峰名	保留时间	峰高	峰面积	含量	
1		10.475	253820.375	2833843.000	50.0083	
2		11.925	225483.984	2832906.000	49.9917	
总计			479304.359	5666749.000	100.0000	





总计



159327.811

4864330.219

100.0000

xx-6-25-as-h-98-2-1-214

报告时间: 2012-06-21, 13:07:25

实验时间: 2012-06-21,12:03:52 请揭文件:D:\zhuguangjiong\xx\20120621\xx-6-25-as-h-98-2-1-214..org

PDF 文件使用 "pdfFactory Pro" 试用版本创建 www.fineprint.cn

xx-5-152-as-h-98-2-1-214

实验内容简介: as-h 98/2 1ml/min 214nm



			DIFARIA			
峰号	峰名	保留时间	峰高	峰面积	含量	
1		22.273	446157.281	13546459.000	50.0151	_
2		24.955	405394.844	13538300.000	49.9849	
总计			851552.125	27084759.000	100.0000	

PDF 文件使用 "pdfFactory Pro" 试用版本创建 www.fineprint.cn





Operator:dell	Timebase:U-3000	Sequence:xx
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251 xx-6-98 AS-H-98-2-0.5-230								
Sample Name:	xx-6-98	Injection Volume:	20.0					
Vial Number:	385	Channel:	UV_VIS_1					
Sample Type:	unknown	Wavelength:	230					
Control Program:	test	Bandwidth:	n.a.					
Quantif. Method:	test	Dilution Factor:	1.0000					
Recording Time:	2013-1-18 14:14	Sample Weight:	1.0000					
Run Time (min):	21.25	Sample Amount:	1.0000					



No.	Ret.Time	Pea	ak Name	Height	Area	Rel.Area	Amount	Туре
	min			mAU	mAU*min	%		
1	15.87	n.a.		14.276	5.831	5.65	n.a.	BMB
2	19.01	n.a.		166.523	97.442	94.35	n.a.	BMB
Total:				180.799	103.273	100.00	0.000	

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Operator:dell Timebase:U-3000 Sequence:xx

Sample Name: Vial Number: Sample Type: Control Prograi Quantif. Methoo Recording Time Run Time (min)	xx-6-6 386 unkno n: test d: test e: 2013- : 21.21	51 own 1-18 14:3	6			Injec Cha Wav Ban Diluu Sar Sar	ction Volume nnel: velength: dwidth: tion Factor: nple Weight: nple Amount	9: 2 U 2 n 1 : 1	0.0 V_VIS_1 30 .a. .0000 .0000 .0000
900 xx #252				xx-6-6	1			l	JV_VIS_1
_mAU							1 - 15.587	W	/L:230 nm
800	CO ₂ Et								
700	/ >								
	//							2 -	18.720
600	-							Λ	
500- Ó									
400-									
300-									
-									
200-									
100-									
1]	
0							J		
-100									min
0.0 2.0	4.0	6.0	8.0	10.0	12.0	14.0	16.0	18.0	21.

No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре
	min		mAU	mAU*min	%		
1	15.59	n.a.	844.202	379.464	49.24	n.a.	BMB
2	18.72	n.a.	614.462	391.202	50.76	n.a.	BMB
Total:			1458.664	770.665	100.00	0.000	

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253 xx-6-99

ample Name: ial Number: ample Type: ontrol Program: uantif. Method: ecording Time: un Time (min):	xx-6-99 387 unknown test test 2013-1-1 13.54	n 8 22:17			Injec Chai Wav Bane Dilut Sam Sam	tion Volu nnel: delength: dwidth: dion Facto ple Weig pple Amo	or: ht: unt:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
700 xx #253 mAU			 xx-6-99					UV_VIS_1 WVL:230 nm
600								2 - 12.7
500-	SU							
400-								
300-								
200-								
100-				1 - 7.1 A	733			
0			 	/\				
-								

No.	Ret.Time	Peak	Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	7 73	n.a.		58.386	8.239	5.95	n.a.	BMB
2	12.71	n.a.		587.951	130.235	94.05	n.a.	BMB
Total:				646.336	138.474	100.00	0.000	
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Operat	or:dell	Timebase:U-3000	Sequence:xx
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254 xx-5-186 ЛD-H-95-5-0.5-230							
Sample Name: Vial Number:	xx-5-186 388	Injection Volume: Channel:	20.0 UV_VIS_1				
Sample Type:	unknown	Wavelength:	230				
Control Program:	test	Bandwidth:	n.a.				
Quantif. Method:	test	Dilution Factor:	1.0000				
Recording Time:	2013-1-18 22:30	Sample Weight:	1.0000				
Run Time (min):	25.27	Sample Amount:	1.0000				



No.	Ret.Time	Peak Name	Height	Area	Rel.Area	Amount	Туре	
	min		mAU	mAU*min	%			
1	7.73	n.a.	634.642	91.349	49.45	n.a.	BMB	
2	12.73	n.a.	423.268	93.378	50.55	n.a.	BMB	
Total:			1057.910	184.727	100.00	0.000		

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244 xx-6-97 AD-H-9:	5-5-0.5-230		
Sample Name: Vial Number: Sample Type:	xx-6-97 378 unknown	Injection Volume: Channel: Wavelength:	20.0 UV_VIS_1 230
Control Program: Quantif. Method: Recording Time:	test test 2013-1-16 15:27	Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	n.a. 1.0000 1.0000 1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	7.97	n.a.	54.792	8.028	7.51	n.a.	BMB*
2	9.53	n.a.	572.657	98.793	92.49	n.a.	BMB*
Total:			627.449	106.821	100.00	0.000	
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Operator:dell	Timebase:U-3000	Sequence:xx
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245 xx-5-18	7		
AD-H-95	-5-0.5-230		
Sample Name: Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-5-187 379 unknown test test 2013-1-16 15:39 10.12	Injection Volume: Channel: Wavelength: Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000



No.	Ret.Time	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	7.97	n.a.	1713.890	267.882	49.50	n.a.	BMB
2	9.51	n.a.	1544.236	273.298	50.50	n.a.	BMB
Total:	0.01		3258.126	541.180	100.00	0.000	

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default/Integration





Sample Name: Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-6-100 380 unknown test test 2013-1-17 23:22 10.65		Injection Volume: Channel: Wavelength: Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	20.0 UV_VIS_1 230 n.a. 1.0000 1.0000 1.0000
120 <u>xx #246</u> mAU 100-		xx-6-100		UV VIS 1 WVL:230 nm 2 - 9.895
60- 40-				
20-			1 - 7.807	

No.	Ret.Time		Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	7.81	na		10.342	1.506	7.04	n.a.	BMB
2	9.89	n a		111.361	19.884	92.96	n.a.	BMB
Total:	0.00	n.a.		121.703	21.391	100.00	0.000	

5.0

3.0

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2.0

1.0

4.0

6.0

7.0

8.0

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min 10.6

9.0

Operator:dell Timebase:U-3000 Sequence:xx

246 xx-6-100

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Operator:dell Timebase:U-3000	Sequence:xx
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247 xx-5-16 AD-H-95	3 5-5-0.5 - 230		
Sample Name:	xx-5-166	Injection Volume:	20.0
Vial Number:	381	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	230
Control Program:	test	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2013-1-17 23:34	Sample Weight:	1.0000
Run Time (min):	24.44	Sample Amount:	1.0000



No.	Ret.Time	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре
1	7.81	n.a.	603.933	90.184	49.96	n.a.	BMB
2	9.91	n.a.	503.330	90.316	50.04	n.a.	BMB
Total:			1107.263	180.499	100.00	0.000	
Total.							

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default/Integration




Operator:dell Timebase:U-3000 Sequence:xx



No.	Ret.Time	Peak Name	Height	Area mAU*min	Rel.Area %	Amount	Туре
L	min		12 794	3.211	5.05	n.a.	BMB
1	14.29	n.a.	229 344	60.313	94.95	n.a.	BMB
2	15.07	n.a	242 138	63 524	100.00	0.000	
Total:			242.100	00.02			

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default/Integration

Operator:dell Timebase:U-3000 Sequence:xx

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220 xx-6-78 IC -95-5-0.	5-214		
Sample Name: Vial Number: Sample Type: Control Program: Quantif. Method: Recording Time: Run Time (min):	xx-6-78 354 unknown test test 2012-12-20 21:23 16.07	Injection Volume: Channel: Wavelength: Bandwidth: Dilution Factor: Sample Weight: Sample Amount:	20.0 UV_VIS_1 214 n.a. 1.0000 1.0000



No.	Ret.Time	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Туре	
1	14.24	n.a.	194.525	48.005	49.81	n.a.	BMB	
2	15.02	n.a.	185.926	48.373	50.19	n.a.	BMB	_
Total:			380.451	96.378	100.00	0.000		
Total.								-

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