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## Supporting Information

Development of Enantioselective Palladium-Catalyzed Alkene Carboalkoxylation Reactions for the Synthesis of Tetrahydrofurans Brett A. Hopkins, Zachary J. Garlets, and John P. Wolfe*

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## Supporting Information

## Experimental procedures and characterization data for new compounds.

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General: Reactions were carried out under nitrogen in flame-dried glassware. Tris(dibenzylideneacetone)dipalladium was purchased from Strem Chemical Co. and used without further purification. Dichloromethane and toluene were purified using a GlassContour solvent system. Anhydrous dioxane was purchased from Acros Organics
in a sure seal bottle and used as received. All other solvents and aryl halides were purchased from commercial sources and used as received. 1-(But-3-en-1-yl)cyclopentan-1-ol (1a), ${ }^{[1]} \quad$ 2,5-dimethylhex-5-en-2-ol (1d) ${ }^{[1]}$ and (+)-(1S,2R)-2-phenylcyclohexan-1-ol, ${ }^{[2]}$ 4-methyl-2,2-diphenylpent-4-en-1-ol (5), ${ }^{[3]}$ and ligands L1-L6 ${ }^{[4]}$ were synthesized according to literature procedures. 4-Penten-1-ol (1b) was purchased from commercial sources and was used without further purification. Yields refer to isolated compounds that are estimated to be $\geq 95 \%$ pure as judged by ${ }^{1} \mathrm{H}$ NMR or GC analysis unless stated otherwise. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 2 and 3 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those in the manuscript.

## Synthesis of Substrates:



1,1-Diphenylpent-4-en-1-ol (1c). ${ }^{[5]}$ A flame dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with 4-pentenoyl chloride (5 $\mathrm{mmol}, 0.55 \mathrm{~mL}$ ) and diethyl ether ( 50 mL ). The mixture was cooled to $0{ }^{\circ} \mathrm{C}$ in an ice bath for five min and then $\mathrm{PhMgBr}(20 \mathrm{~mL}, 20 \mathrm{mmol}, 1 \mathrm{M}$ in THF) was added dropwise to the flask. The resulting mixture was warmed to rt and stirred for 12 h , then the flask was cooled to $0{ }^{\circ} \mathrm{C}$ in an ice bath and slowly quenched with saturated aqueous ammonium chloride ( 10 mL ). The mixture was transferred to a separatory funnel, the layers were separated, and the aqueous layer was extracted with ethyl acetate (3 x 25 mL . The organic layers were combined, dried over anhydrous sodium sulfate, filtered,
and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel to afford the title compound ( $864 \mathrm{mg}, 72 \%$ ) as a colorless oil. ${ }^{1} \mathrm{H} \operatorname{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.43(\mathrm{~d}, J=7.4 \mathrm{~Hz}, 4 \mathrm{H}), 7.33(\mathrm{t}, J=8.1 \mathrm{~Hz}, 4 \mathrm{H}), 7.24$ (t, J = 6.6 Hz, 2 H), 6.85-6.78 (m, 2 H ), 5.06-4.96 (m, 2 H ), 2.44-2.38 (m, 2 H ), 2.18 (s, 1 H), 2.12-2.04 (m, 2 H). Spectroscopic data was consistent with that previously reported in the literature. ${ }^{[5]}$


4-Methyl-1,1-diphenylpent-4-en-1-ol (1e). A flame dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with $\mathrm{PhMgBr}(25 \mathrm{~mL}$, $25 \mathrm{mmol}, 1 \mathrm{M}$ in THF). The solution was cooled to $0^{\circ} \mathrm{C}$ in an ice bath for five min. In a separate flask ethyl 4-methylpent-4-enoate ${ }^{[6]}(1.0 \mathrm{~g}, 7 \mathrm{mmol})$ was dissolved in 20 mL anhydrous THF, and the resulting solution was added dropwise to the flask containing the cooled PhMgBr solution. The reaction mixture was then warmed to rt , stirred for 12 h , then was cooled to $0{ }^{\circ} \mathrm{C}$ in an ice bath and slowly quenched with saturated aqueous ammonium chloride ( 20 mL ). The resulting mixture was transferred to a separatory funnel, the layers were separated, and the aqueous layer was extracted with diethyl ether $(3 \times 25 \mathrm{~mL})$. The organic layers were combined, dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel to afford the title compound ( $1.54 \mathrm{~g}, 88 \%$ ) as a colorless oil. ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.43$ (dd, $J=8.31,0.98 \mathrm{~Hz}, 4 \mathrm{H}$ ), $7.34-7.31$ (m, 4H), 7.25-7.20 (m, 2 H ), 4.73 (s, 1 H ), 4.70 (s, 1 H ), 2.48-2.42 (m, 2 H ), 2.25 (s, br,

1 H ), 2.06-1.99 (m, 2 H ), $1.74(\mathrm{~s}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $175 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 147.1, 146.4, 128.4, 127.0, 126.2, 110.1, 78.5, 40.0, 32.2, 23.0; IR (film) 3469, 2932, $1446 \mathrm{~cm}^{-1}$; MS (EI) 252.1515 ( 252.1514 calcd for $\mathrm{C}_{18} \mathrm{H}_{20} \mathrm{O}, \mathrm{M}+$ ).

(E)-1,1-Diphenylhex-4-en-1-ol (1f). The title compound was prepared from PhMgBr (50 $\mathrm{mL}, 50 \mathrm{mmol}, 1 \mathrm{M}$ in THF) and (E)-ethyl hex-4-enoate ${ }^{[7]}(2.28 \mathrm{~g}, 16.0 \mathrm{mmol})$ using a procedure analogous to that described above for the synthesis of $\mathbf{1 e}$. This procedure afforded the title compound ( $1.23 \mathrm{~g}, 30 \%$ ) as a colorless solid, $\mathrm{mp} 53-54^{\circ} \mathrm{C}:{ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.40-7.43(\mathrm{~m}, 4 \mathrm{H}), 7.28-7.33(\mathrm{~m}, 4 \mathrm{H}), 7.20-7.24(\mathrm{~m}, 2 \mathrm{H}), 5.37-$ 5.51 (m, 2 H), 2.33-2.38(m, 2 H), 2.23 (s, 1 H), 1.96-2.03 (m, 2 H), 1.63 (dd, J = 5.9, $1.0 \mathrm{~Hz}, 3 \mathrm{H}$ ); ${ }^{13} \mathrm{C}$ NMR ( $175 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 147.2, 131.3, 128.3, 127.0, 126.2, 125.7, 78.6, 41.7, 27.3, 18.1; IR (film) 3556, 2958, $1446 \mathrm{~cm}^{-1}$; MS (EI) 252.1510 (252.1514 calcd for $\mathrm{C}_{18} \mathrm{H}_{20} \mathrm{O}, \mathrm{M}+$ ).


3-(Cyclohex-1-en-1-yl)-1,1-diphenylpropan-1-ol (1g). The title compound was prepared from PhMgBr ( $11 \mathrm{~mL}, 11 \mathrm{mmol}, 1 \mathrm{M}$ in THF) and 3-(cyclohex-1-en-1-yl)-1-phenylpropan-1-one ${ }^{[8]}(1.2 \mathrm{~g}, 5.5 \mathrm{mmol})$ using a procedure analogous to that described above for the synthesis of $\mathbf{1 e}$. This procedure afforded the title compound ( 600 mg ,
$37 \%$ ) as a colorless oil. ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.39-7.49(\mathrm{~m}, 4 \mathrm{H}), 7.28-7.34(\mathrm{~m}$, 4 H), 7.18-7.24 (m, 2 H), 5.41 (s, 1 H), 2.38-2.45 (m, 2 H), 2.37 (s, 1 H), 1.88-2.03 (m, 6 H ), 1.48-1.67 (m, 4 H ); ${ }^{13} \mathrm{C}$ NMR ( $175 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 147.2,138.2$, 128.3, 126.9, 126.2, 121.6, 78.7, $39.8,32.5,28.7,25.4,23.1,22.7$; IR (film) $3467,2923,1446 \mathrm{~cm}^{-1}$; MS (EI) 292.1823 (292.1827 calcd for $\mathrm{C}_{21} \mathrm{H}_{24} \mathrm{O}, \mathrm{M}+$ ).

## Synthesis of ligand L7.


(-)-(1S,2R)-2-[(1,1'-Biphenyl)-4-yl]cyclohexan-1-ol (S1). A flame-dried 2-neck round bottom flask equipped with a stirbar and a reflux condenser was cooled under a stream of nitrogen and charged with magnesium turnings ( $1.76 \mathrm{~g}, 72 \mathrm{mmol}$ ) and THF ( 50 mL ). A solution of 4-bromobiphenyl ( $11.65 \mathrm{~g}, 50 \mathrm{mmol}$ ) in THF ( 15 mL ) was slowly added. The reaction mixture began to rapidly reflux, and the reaction temperature was controlled by placing the flask in an ice bath until reflux subsided. Once the magnesium turnings had disappeared, the reaction mixture was cooled to $-20^{\circ} \mathrm{C}$ for 10 min then $\mathrm{CuCl}(8 \mathrm{~mol} \%)$ was added to the reaction mixture immediately followed by the addition of cyclohexene oxide ( $3.36 \mathrm{~mL}, 33.3 \mathrm{mmol}$ ) as a solution in THF ( 7 mL ). The resulting mixture was allowed to slowly warm to rt and stirred for 4 h . The mixture was then cooled to $0{ }^{\circ} \mathrm{C}$ and quenched with saturated ammonium chloride $(1 \mathrm{~mL} / \mathrm{mmol}$ cyclohexene oxide). The mixture was filtered through a pad of celite, and transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with ethyl acetate (3x). The combined organic layers were then dried over sodium
sulfate, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel to afford $4.00 \mathrm{~g}(48 \%)$ of $( \pm)$-S1 as a white solid.

A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with PS 30 Amano Lipase (14.8 mg), (土)-S1 (3.74 g, 14.8 mmol ) and tert-butyl methyl ether ( 45 mL ). Neat vinyl acetate ( $13.6 \mathrm{~mL}, 148 \mathrm{mmol}$ ) was then added and the resulting mixture was stirred at rt until one enantiomer of the alcohol had been consumed as judged by chiral HPLC analysis (3 days). The mixture was then filtered through a fritted funnel and the enzyme was washed with diethyl ether and then recycled for future use (if desired). The resulting solution was concentrated in vacuo and the crude product was purified by flash chromatography on silica gel to afford 1.72 g (46\%) of the title compound as a white solid, mp $122-125{ }^{\circ} \mathrm{C}$. This material was judged to be >99:1 er by chiral HPLC analysis (Chiracel OJH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 4 \%$ IPA/Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=21.8$ and 25.0 min ). $[\alpha]^{23}{ }_{\mathrm{D}}-13.99$ (c 3.38, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H} \operatorname{NMR}\left(700 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.62-7.57(\mathrm{~m}, 4 \mathrm{H}), 7.45(\mathrm{t}, \mathrm{J}=7.7 \mathrm{~Hz}, 2 \mathrm{H})$, 7.37-7.34 (m, $3 H$ ), 3.72 (td, $J=10.0,4.2 \mathrm{~Hz}, 1 \mathrm{H}), 2.51(\mathrm{td}, J=11.1,3.6 \mathrm{~Hz}, 1 \mathrm{H}), 2.1-$ $2.15(\mathrm{~m}, 1 \mathrm{H}), 1.95-1.88(\mathrm{~m}, 2 \mathrm{H}), 1.81$ (app. d, $J=13.2 \mathrm{~Hz}, 1 \mathrm{H}), 1.64-1.35(\mathrm{~m}, 5 \mathrm{H})$; ${ }^{13} \mathrm{C} \operatorname{NMR}(175 \mathrm{MHz} \mathrm{CDCl} 3) ~ \delta 142.6,141.1,140.0,128.9,128.5,127.7,127.3,127.2$, 74.6, 53.1, 34.5, 33.5, 26.2, 25.3; IR (film) 3548, 2919, $1490 \mathrm{~cm}^{-1}$; MS (ESI+) 270.1850 (270.1852 calcd for $\mathrm{C}_{18} \mathrm{H}_{20} \mathrm{O}, \mathrm{M}+\mathrm{NH}_{4}{ }^{+}$).

(+)-(1S,2R,3aS,8aS)-6-\{[-2-([1,1'-Biphenyl]-4-yl)cyclohexyl]oxy\}-2',2'-dimethyl-4,4,8,8-Tetraphenyltetrahydro-[1,3]dioxolo[4,5-e][1,3,2]dioxaphosphepine

The ligand was prepared according to a previously reported procedure for the synthesis of chiral phosphites. ${ }^{[4]}$ A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with (1S,2R)-2-([1,1'-biphenyl]-4-yl)cyclohexan-1-ol ( $255 \mathrm{mg}, 1.01 \mathrm{mmol}$ ), and dry dichloromethane ( 2 mL ). Neat $\mathrm{PCl}_{3}$ (86 $\mu \mathrm{L}, 1.01 \mathrm{mmol}$, was added and the resulting mixture was allowed to stir for 1 h at rt . After this time, anhydrous $\mathrm{NEt}_{3}(0.56 \mathrm{~mL}, 4.04 \mathrm{mmol})$ was added dropwise and the mixture was stirred at rt for 30 min . A solution of $(S, S)$-TADDOL ( $450 \mathrm{mg}, 0.963 \mathrm{mmol}$ ) in dichloromethane ( 2 mL ) was added, and the reaction mixture was stirred at rt for 12 h. The mixture was then diluted with diethyl ether ( 20 mL ) and then filtered through celite. The solvent was evaporated in vacuo and the crude product was purified by flash chromatography on silica gel to afford 520 mg (72\%) of the title compound as a white foamy solid, $\mathrm{mp} 115-118{ }^{\circ} \mathrm{C} .[\alpha]^{23} \mathrm{D}+130.0\left(c 5.81, \mathrm{CH}_{2} \mathrm{Cl}_{2}\right) ;{ }^{1} \mathrm{H}$ NMR (700 MHz, $\left.\mathrm{CDCl}_{3}\right) \delta 7.51(\mathrm{~d}, J=7.9 \mathrm{~Hz}, 2 \mathrm{H}), 7.38-7.02(\mathrm{~m}, 27 \mathrm{H}), 4.94-4.90(\mathrm{~m}, 1 \mathrm{H}), 4.88(\mathrm{~d}, \mathrm{~J}=$ 8.4 Hz, 1 H), 4.56 (app. qd, $J=9.9,3.7 \mathrm{~Hz}, 1 \mathrm{H}), 2.71-2.66(\mathrm{~m}, 1 \mathrm{H}), 2.28$ (app. d, $J=$ 13.8 Hz, 1 H), 1.95 (app. d, $J=13.2 \mathrm{~Hz}, 1 \mathrm{H}), 1.81-1.73(\mathrm{~m}, 2 \mathrm{H}), 1.69-1.50(\mathrm{~m}, 2 \mathrm{H})$, 1.41-1.31 (m, 2 H ), 1.19 (s, 3 H ), 0.30 (s, 3 H ); ${ }^{13} \mathrm{C}$ NMR (175 MHz CDCl ${ }_{3}$ ) $\delta 146.6$, $146.1,143.0,141.8,141.25,141.23,139.4,129.4,129.0,128.92,128.89,128.6,128.0$, $127.8,127.54,127.48,127.45,127.38,127.34,127.30,127.18,127.15,127.12,127.06$, $126.9,112.0,82.9,82.7,82.6,82.12,82.10,81.92,81.88,78.10,78.09,51.39,51.37$, $35.7,33.8,27.6,26.0,25.5,25.3$ (due to the complexity of the spectra all the peaks are listed without assigning C-P couplings); ${ }^{31} \mathrm{P}$ NMR (202 MHz CDCl ${ }_{3}$ ) $\delta 140.6$; IR (film)

2932, 1486, $1447 \mathrm{~cm}^{-1}$; MS (ESI+) 747.3224 (747.3234 calcd for $\mathrm{C}_{49} \mathrm{H}_{47} \mathrm{O}_{5} \mathrm{P}, \mathrm{M}+\mathrm{H}^{+}$).

## General procedure for asymmetric Pd-catalyzed carboalkoxylation reactions. A

 flame-dried Schlenk tube equipped with a stirbar was cooled under a stream of nitrogen and charged with $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(2 \mathrm{~mol} \%), \mathrm{L} 7(5 \mathrm{~mol} \%)$, the alcohol substrate (1.0 equiv), and $\mathrm{NaO}^{t} \mathrm{Bu}$ (1.50-2.0 equiv). The flask was purged with $\mathrm{N}_{2}$ then the aryl or alkenyl halide (1.40-2.0 equiv), and dioxane or toluene ( 0.10 M ) was added. The resulting mixture was heated to $90^{\circ} \mathrm{C}$ with stirring until the starting material had been consumed as judged by TLC analysis (ca. 12 h ). The reaction mixture was then cooled to rt , saturated aqueous ammonium chloride ( $6 \mathrm{~mL} / \mathrm{mmol}$ substrate) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate ( $3 \times 5 \mathrm{~mL}$ ) then the combined organic layers were dried over anhydrous $\mathrm{Na}_{2} \mathrm{SO}_{4}$, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel. (+)-(S)-2-(Naphthalen-2-ylmethyl)-1-oxaspiro[4.4]nonane (2a). The general procedure was employed for the coupling of 1-(but-3-en-1-yl)cyclopentan-1-ol (28.0 mg, 0.20 mmol ) and 2-bromonaphthalene ( $75.0 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathrm{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $31.1 \mathrm{mg}, 58 \%, 10: 1$ regioselectivity) as a colorless oil: $[\alpha]^{23}{ }_{\mathrm{D}}+12.4(c 2.1$, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.85-7.79(\mathrm{~m}, 3 \mathrm{H}), 7.67(\mathrm{~s}, 1 \mathrm{H}), 7.49-7.35(\mathrm{~m}, 3$
H), 4.29-4.22 (m, 1H), 3.15 (dd, $J=13.6,5.0 \mathrm{~Hz}, 1 \mathrm{H}), 2.87(\mathrm{dd}, J=13.3,7.7 \mathrm{~Hz}, 1 \mathrm{H})$, 1.95-1.87(m, 1 H$), 1.86-1.49(\mathrm{~m}, 11 \mathrm{H}) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(125 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 136.6,133.7$, $132.3,128.4,127.84,127.79,127.75,127.7,91.6,79.2,43.0,39.4,38.6,36.6,24.2 ;$ IR (film) 2953, 2361, 2338, $1508 \mathrm{~cm}^{-1} . \mathrm{MS}(\mathrm{CI}) 267.1743$ (267.1743 calcd for $\mathrm{C}_{19} \mathrm{H}_{22} \mathrm{O}, \mathrm{M}+$ $\mathrm{H}^{+}$). The enantiopurity was determined to be 89:11 er by chiral HPLC analysis (Chiralcel OJH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 1 \% \mathrm{IPA} /$ Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=10.5$ and 12.8 $\min )$.

(+)-(S)-2-(Naphthalen-2-ylmethyl)-1-oxaspiro[4.4]nonane (2b). The general procedure was employed for the coupling of 1-(but-3-en-1-yl)cyclopentan-1-ol ( 28 mg , 0.20 mmol ) and 4-bromobenzophenone ( $94 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathrm{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$, and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $34.6 \mathrm{mg}, 53 \%, 6: 1$ regioselectivity) as a clear oil: $[\alpha]^{23}{ }_{\mathrm{D}}+21.9$ (c 1.77, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H}$ NMR (500 MHz, $\left.\mathrm{CDCl}_{3}\right) \delta 7.80-7.77(\mathrm{~m}, 2 \mathrm{H}), 7.74(\mathrm{~d}, \mathrm{~J}=8.1 \mathrm{~Hz}, 2 \mathrm{H})$, $7.57(\mathrm{t}, J=7.3 \mathrm{~Hz}, 1 \mathrm{H}), 7.49-7.45(\mathrm{~m}, 2 \mathrm{H}), 7.34(\mathrm{~d}, J=8.3 \mathrm{~Hz}, 2 \mathrm{H}), 4.19$ (app. quint, $J=6.6 \mathrm{~Hz}, 1 \mathrm{H}), 3.01(\mathrm{dd}, J=13.4,5.6 \mathrm{~Hz}, 1 \mathrm{H}), 2.80(\mathrm{dd}, J=13.4,6.8 \mathrm{~Hz}, 1 \mathrm{H}), 1.96-$ $1.90(\mathrm{~m}, 1 \mathrm{H}), 1.83-1.48(\mathrm{~m}, 11 \mathrm{H}) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(125 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 196.5,144.1,137.9$, $132.2,130.1,129.9,129.4,128.3,128.2,127.6,91.5 .78 .5,42.7,39.2,38.4,36.4,31.3$, 24.0, 23.9; IR (film) 2959, 1655, 1606, $1277 \mathrm{~cm}^{-1}$. MS (CI) 321.1848 (321.1849 calcd for $\left.\mathrm{C}_{22} \mathrm{H}_{24} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}\right)$. The enantiopurity was determined to be $82: 18$ er by chiral HPLC
analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 1 \% \mathrm{IPA} /$ Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}$, $R T=17.4$ and 18.4 min$)$.

${ }^{(+)-(S)-2-(N a p h t h a l e n-2-y l m e t h y l) t e t r a h y d r o f u r a n t e t r a h y d r o f u r a n ~(2 c) . ~ T h e ~ g e n e r a l ~}$ procedure was employed for the coupling of pent-4-en-1-ol ( $17 \mathrm{mg}, 0.20 \mathrm{mmol}$ ) and 2bromonaphthalene ( $58 \mathrm{mg}, 0.28 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}$, 0.004 mmol ) and $\mathbf{L 7}(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$, and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound (9.9 $\mathrm{mg}, 23 \%$ ) as a light yellow oil. $[\alpha]^{23}{ }_{\mathrm{D}}=+2.1\left(\mathrm{c} 0.95, \mathrm{CH}_{2} \mathrm{Cl}_{2}\right) ;{ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.83-7.75(\mathrm{~m}, 3 \mathrm{H}), 7.68(\mathrm{~s}, 1 \mathrm{H}), 7.48-7.36(\mathrm{~m}, 3 \mathrm{H}), 4.18$ (app. quint, $J=6.5 \mathrm{~Hz}, 1$ H), 3.95-3.89 (m, 1 H), 3.79-3.73 (m, 1 H ), 3.08 (dd, $J=13.5,6.4 \mathrm{~Hz}, 1 \mathrm{H}$ ), 2.92 (dd, $J$ $=13.7,6.4 \mathrm{~Hz}, 1 \mathrm{H}), 1.98-1.82(\mathrm{~m}, 3 \mathrm{H}), 1.66-1.57(\mathrm{~m}, 1 \mathrm{H})$. Other spectroscopic data matched those previously reported. ${ }^{[1]}$ The enantiopurity was determined to be 58:42 er by chiral HPLC analysis (Chiralcel OJH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 1 \%$ IPA/Hexanes, 1.50 $\mathrm{mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=19.8$ and 26.1 min ).

(+)-(S)-5-(Naphthalen-2-ylmethyl)-2,2-diphenyItetrahydrofuran (2d). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol ( $48 \mathrm{mg}, 0.20$ mmol ) and 2-bromonaphthalene ( $58 \mathrm{mg}, 0.28 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathbf{L 7}(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of
$90^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound (48.7mg, $67 \%$ ) as a white solid, $\mathrm{mp} 83-86{ }^{\circ} \mathrm{C} .[\alpha]^{23}{ }_{\mathrm{D}}+29.6\left(c 4.24, \mathrm{CH}_{2} \mathrm{Cl}_{2}\right.$ ); ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.86(\mathrm{~d}, J=7.2 \mathrm{~Hz}, 1 \mathrm{H}), 7.81(\mathrm{~d}, J=8.6 \mathrm{~Hz}, 2 \mathrm{H}), 7.74(\mathrm{~s}$, $1 \mathrm{H}), 7.55-7.44(\mathrm{~m}, 7 \mathrm{H}), 7.37-7.31(\mathrm{~m}, 4 \mathrm{H}), 7.28-7.21(\mathrm{~m}, 2 \mathrm{H}), 4.53$ (app. quint, $J=$ $6.7 \mathrm{~Hz}, 1 \mathrm{H}), 3.34(\mathrm{dd}, J=13.6,6.0 \mathrm{~Hz}, 1 \mathrm{H}), 3.02(\mathrm{dd}, J=13.6,7.0 \mathrm{~Hz}, 1 \mathrm{H}), 2.71-2.64$ $(\mathrm{m}, 1 \mathrm{H}), 2.58-2.51(\mathrm{~m}, 1 \mathrm{H}), 2.03-1.95(\mathrm{~m}, 1 \mathrm{H}), 1.89-1.81(\mathrm{~m}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125 $\left.\mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 147.4,146.9,136.6,133.7,132.3,128.31,128.25,128.2,127.88$, 127.87, 127.8, 127.6, 126.76, 126.74, 126.1, 126.0, 125.4, 88.5, 79.9, 42.8, 38.8, 31.0; IR (film) 2934, 1601, $1446 \mathrm{~cm}^{-1} . \mathrm{MS}(\mathrm{Cl}) 365.1899$ (365.1900 calcd for $\mathrm{C}_{27} \mathrm{H}_{24} \mathrm{O}, \mathrm{M}+$ $\mathrm{H}^{+}$). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \% \mathrm{IPA} /$ Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=5.2$ and 6.3 min$)$. When 2.0 equiv of $\mathrm{H}_{2} \mathrm{O}$ was added with toluene as solvent the enantiopurity was determined to be 96:4 er.


## (+)-(S)-\{4-[(5,5-diphenyltetrahydrofuran-2-yl)methyl]phenyl\}(phenyl)methanone

(2e). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1ol ( $48 \mathrm{mg}, 0.20 \mathrm{mmol}$ ) and 4-bromobenzophenone ( $94 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathrm{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90{ }^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $52 \mathrm{mg}, 62 \%$ ) as a colorless oil. $[\alpha]^{23}{ }_{\mathrm{D}}+18.9$ (c 2.40, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H} \operatorname{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.82-7.78(\mathrm{~m}, 2 \mathrm{H}), 7.75(\mathrm{~d}, \mathrm{~J}=8.1 \mathrm{~Hz}, 2 \mathrm{H})$,
$7.59(\mathrm{t}, J=7.2 \mathrm{~Hz}, 1 \mathrm{H}), 7.51-7.40(\mathrm{~m}, 6 \mathrm{H}), 7.38(\mathrm{~d}, J=8.1 \mathrm{~Hz}, 2 \mathrm{H}), 7.31-7.25(\mathrm{~m}, 4$ H), 7.22-7.17 (m, 2 H), 4.42 (app. quint, $J=6.6 \mathrm{~Hz}, 1 \mathrm{H}$ ), 3.16 (dd, $J=13.6,6.6 \mathrm{~Hz}, 1$ H), 2.91 (dd, J = 13.7, 6.4 Hz, 1 H ), 2.68-2.61 (m, 1 H ), 2.54-2.47 (m, 1 H ), 2.03-1.96 ( $\mathrm{m}, 1 \mathrm{H}$ ), 1.81-1.73 (m, 1 H ); ${ }^{13} \mathrm{C} \operatorname{NMR}\left(125 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta$ 196.7, 147.2, 146.7, 144.2, 138.0, 135.7, 132.4, 130.4, 130.2, 129.5, 128.40, 128.35, 128.2, 126.83, 126.80, 126.0, 125.9, 88.6, 79.5, 42.7, 38.7, 31.2; IR (film) 2362, 1654, $1446 \mathrm{~cm}^{-1}$. MS (CI) 419.2006 (419.2006 calcd for $\mathrm{C}_{30} \mathrm{H}_{26} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 92:8 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 5 \%$ IPA/Hexanes, 1.00 $\mathrm{mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=9.8$ and 11.7 min$)$. When 2.0 equiv of $\mathrm{H}_{2} \mathrm{O}$ was added with toluene as solvent the enantiopurity was determined to be 95:5 er (an unknown product co-eluted when water was used with this reaction see spectra for product $\mathbf{2 e}$ below.)


## $(+)-(S)-5-[(6-m e t h o x y n a p h t h a l e n-2-y l) m e t h y l]-2,2-d i p h e n y l t e t r a h y d r o f u r a n ~(2 f) . ~ T h e ~$

 general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol ( 48 mg , 0.20 mmol ) and 2-bromo-6-methoxynaphthalene ( $85 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathbf{L 7}(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $52 \mathrm{mg}, 66 \%$ ) as a white solid, $\mathrm{mp} 93-96{ }^{\circ} \mathrm{C}$. $[\alpha]^{23}{ }_{\mathrm{D}}+29.8$ (c 5.19, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.69$ (app. dd, $J=8.6,3.2 \mathrm{~Hz}, 2 \mathrm{H}$ ), $7.65(\mathrm{~s}$, 1 H ), 7.53-7.46 (m, 4 H ), 7.69 (d, J = $8.4 \mathrm{~Hz}, 1 \mathrm{H}$ ), 7.36-7.28 (m, 4 H ), 7.27-7.13 (m, 4 H), 4.50 (app. quint, $J=6.7 \mathrm{~Hz}, 1 \mathrm{H}$ ), 3.94 (s, 3H), 3.29 (dd, $J=13.7,5.9 \mathrm{~Hz}, 1 \mathrm{H}$ ), 2.97(dd, $J=13.7,7.1 \mathrm{~Hz}, 1 \mathrm{H}), 2.69-2.61(\mathrm{~m}, 1 \mathrm{H}), 2.55-2.48(\mathrm{~m}, 1 \mathrm{H}), 2.00-1.93(\mathrm{~m}, 1 \mathrm{H})$, 1.87-1.79 (m, 1 H ); ${ }^{13} \mathrm{C} \operatorname{NMR}\left(125 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 157.4,147.4,146.9,134.2,133.3$, 129.2, 128.6, 128.3, 128.2, 127.7, 126.74, 126.72, 126.1, 126.0, 118.8, 105.8, 88.5 , 80.0, 55.4, 42.6, 38.8, 30.9; IR (film) 2937, 1605, $1448 \mathrm{~cm}^{-1} . \mathrm{MS}$ (CI) 395.2004 (395.2006 calcd for $\mathrm{C}_{28} \mathrm{H}_{26} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \%$ IPA/Hexanes, 1.00 $\mathrm{mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=7.5$ and 8.9 min$)$.


## (+)-(S)-5-(4-Methoxybenzyl)-2,2-diphenyltetrahydrofuran (2g). The general

 procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol ) and 4-bromoanisole ( $46 \mu \mathrm{~L}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}$ ( $3.7 \mathrm{mg}, 0.004 \mathrm{mmol}$ ) and $\mathrm{L} 7\left(7.5 \mathrm{mg}, 0.010 \mathrm{mmol}\right.$ ), a reaction temperature of $90{ }^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $45.0 \mathrm{mg}, 65 \%$ ) as a colorless oil: $[\alpha]^{23}{ }_{\mathrm{D}}+24.5$ (c 2.00, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H}$ NMR (700 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 7.47-7.42(\mathrm{~m}, 4 \mathrm{H}), 7.30-7.26(\mathrm{~m}, 4 \mathrm{H}), 7.21-7.15(\mathrm{~m}, 4 \mathrm{H}), 6.82$ (d, $J=8.6 \mathrm{~Hz}, 2 \mathrm{H}$ ), 4.33 (app. quint, $J=6.6 \mathrm{~Hz}, 1 \mathrm{H}$ ), $3.79(\mathrm{~s}, 3 \mathrm{H}), 3.08$ (dd, $J=13.6$, $5.8 \mathrm{~Hz}, 1 \mathrm{H}), 2.74(\mathrm{dd}, \mathrm{J}=13.7,7.2 \mathrm{~Hz}, 1 \mathrm{H}), 2.64-2.59(\mathrm{~m}, 1 \mathrm{H}), 2.50-2.45(\mathrm{~m}, 1 \mathrm{H})$, 1.95-1.89 (m, 1 H ), 1.76-1.70 (m, 1 H ); ${ }^{13} \mathrm{C}$ NMR (175 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 158.2,147.4$, $146.9,131.1,130.5,128.3,128.1,126.73,126.70,126.1,126.0,113.9,88.4,80.2,55.4$, 41.8, 38.8, 30.9; IR (film) 2936, 1606, $1512 \mathrm{~cm}^{-1}$. MS (CI) 345.1847 (345.1855 calcd for $\mathrm{C}_{24} \mathrm{H}_{24} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 94:6 er by chiral HPLCanalysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \%$ IPA/Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}$, $R T=7.5$ and 8.9 min$)$.

$(+)-(S)$-1-Benzyl-5-[(5,5-diphenyltetrahydrofuran-2-yl)methyl]-1H-indole (2h). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol ( 48 mg , 0.20 mmol ) and 1-benzyl-5-bromo-1H-indole ( $103 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathbf{L 7}(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $63.5 \mathrm{mg}, 72 \%$ ) as a colorless oil: $[\alpha]^{23} \mathrm{D}+14.7$ (c 6.00 , $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H}$ NMR ( $700 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.54-7.46(\mathrm{~m}, 5 \mathrm{H}), 7.33-7.25(\mathrm{~m}, 7 \mathrm{H}), 7.23-$ 7.18 (m, 3 H ), $7.15-7.08$ ( $\mathrm{m}, 4 \mathrm{H}$ ), $6.50(\mathrm{~d}, \mathrm{~J}=3.1 \mathrm{~Hz}, 1 \mathrm{H}$ ), 5.30 ( $\mathrm{s}, 2 \mathrm{H}$ ), 4.44 (app. quint, $J=6.7 \mathrm{~Hz}, 1 \mathrm{H}$ ), $3.30(\mathrm{dd}, J=13.5,5.4, \mathrm{~Hz} 1 \mathrm{H}), 2.88(\mathrm{dd}, J=13.5,7.9 \mathrm{~Hz}, 1 \mathrm{H})$, 2.65-2.60 (m, 1 H), 2.55-2.50 (m, 1 H), 1.95-1.89 (m, 1 H), 1.84-1.78 (m, 1 H); ${ }^{13} \mathrm{C}$ NMR (175 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 147.5,147.0,137.8,135.3,130.0,129.0,128.9,128.3,128.1$, 127.7, 127.0, 126.68, 126.66, 126.13, 126.08, 123.7, 121.4, 109.5, 101.5, 88.4, 80.9, 50.3, 42.8, 38.9, 30.9; IR (film) 2923, 1485, $1446 \mathrm{~cm}^{-1}$. MS (CI) 444.2319 (444.2322 calcd for $\mathrm{C}_{32} \mathrm{H}_{29} \mathrm{NO}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 93:7 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \%$ IPA/Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254$ $\mathrm{nm}, \mathrm{RT}=10.8$ and 21.3 min ).


## (+)-(S)-Phenyl\{4-[(2,5,5-trimethyltetrahydrofuran-2-yl)methyl]phenyl\}methanone

(2j). The general procedure was employed for the coupling of 2,5-dimethylhex-5-en-2ol $^{1}$ ( $26 \mathrm{mg}, 0.20 \mathrm{mmol}$ ) and 4-bromobenzophenone ( $94 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathrm{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90{ }^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $51.0 \mathrm{mg}, 77 \%$ ) as a colorless oil: $[\alpha]^{23}{ }_{\mathrm{D}}+3.48$ (c 7.10, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H} \operatorname{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.76-7.82(\mathrm{~m}, 2 \mathrm{H}), 7.73(\mathrm{~d}, \mathrm{~J}=8.1 \mathrm{~Hz}, 2 \mathrm{H})$, $7.54-7.60(\mathrm{~m}, 1 \mathrm{H}), 7.47(\mathrm{t}, J=10.0 \mathrm{~Hz}, 2 \mathrm{H}), 7.35(\mathrm{~d}, J=8.1 \mathrm{~Hz}, 2 \mathrm{H}), 2.85(\mathrm{~s}, 2 \mathrm{H})$, 1.95-2.03 (m, 1 H), 1.76-1.85 (m, 2 H), 1.56-1.66 (m, 1 H), $1.25(\mathrm{~d}, \mathrm{~J}=2.9 \mathrm{~Hz}, 6 \mathrm{H})$, 1.14 (s, 3 H ); ${ }^{13} \mathrm{C}$ NMR (175 MHz, $\mathrm{CDCl}_{3}$ ) $\delta$ 196.8, 143.9, 138.1, 135.6, 132.3, 130.8, 130.1, 129.9, 128.4, 83.3, 81.7, 48.5, 38.6, 36.7, 29.9, 29.4, 28.6; IR (film) 2966, 1654, $1277 \mathrm{~cm}^{-1}$; MS (ESI+) 309.1847 (309.1849 calcd for $\mathrm{C}_{21} \mathrm{H}_{24} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 38:62 er by chiral HPLC analysis (Chiralcel ADH, 25 $\mathrm{cm} \times 4.6 \mathrm{~mm}, 2 \% \mathrm{IPA} /$ Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 195 \mathrm{~nm}, \mathrm{RT}=10.1$ and 10.8 min$)$.


## (+)-(S)-\{4-[(2-Methyl-5,5-diphenyltetrahydrofuran-2

$\mathbf{y l}) m e t h y l] p h e n y l\}(p h e n y l) m e t h a n o n e(2 k)$. The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol (51 mg, 0.20 mmol ) and 4-
bromobenzophenone ( $94 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7$ $\mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathrm{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound (74.8 mg, 86\%) as a colorless solid, mp 89-91 ${ }^{\circ} \mathrm{C}:[\alpha]^{23} \mathrm{D}+20.9\left(c 6.30, \mathrm{CH}_{2} \mathrm{Cl}_{2}\right) ;{ }^{1} \mathrm{H}$ NMR (500 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 7.79-7.86(\mathrm{~m}, 2 \mathrm{H}), 7.72(\mathrm{~d}, \mathrm{~J}=8.1 \mathrm{~Hz}, 2 \mathrm{H}), 7.58-7.64(\mathrm{~m}, 1$ H), 7.46-7.54 (m, 6 H), 7.26-7.40(m, 6H), 7.16-7.25 (m, 2H), 3.03 (d, J = $13.2 \mathrm{~Hz}, 1$ $\mathrm{H}), 2.91(\mathrm{~d}, \mathrm{~J}=13.2 \mathrm{~Hz}, 1 \mathrm{H}), 2.62-2.75(\mathrm{~m}, 2 \mathrm{H}), 2.09(\mathrm{~m}, 1 \mathrm{H}), 1.86(\mathrm{~m} 1 \mathrm{H}), 1.31(\mathrm{~s}, 3$ $\mathrm{H}) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(175 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 196.7,148.2,147.7,143.8,138.0,135.6,132.3$, 130.6, 130.1, 129.9, 128.4, 128.1, 128.1, 126.7, 126.5, 126.0, 125.8, 88.7, 84.6, 48.5, 38.4, 37.4, 27.2; IR (film) 2966, 1654, $1277 \mathrm{~cm}^{-1}$; MS (ESI+) 433.2160 (433.2162 calcd for $\mathrm{C}_{31} \mathrm{H}_{28} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \%$ IPA/Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 275 \mathrm{~nm}$, $R T=15.4$ and 18.1 min$).$


## (+)-(S)-1-Benzyl-5-[(2-methyl-5,5-diphenyltetrahydrofuran-2-yl)methyl]-1H-indole

(2I). The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol (51 mg, 0.20 mmol ) and 1-benzyl-5-bromo-1H-indole (103 mg, $0.36 \mathrm{mmol})$ using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and L 7 (7.5 $\mathrm{mg}, 0.010 \mathrm{mmol}$ ), a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h . This procedure afforded the title compound ( $80.5 \mathrm{mg}, 88 \%$ ) as a colorless solid, mp 127-128 ${ }^{\circ} \mathrm{C}:[\alpha]^{23} \mathrm{D}+22.6\left(c 6.91, \mathrm{CH}_{2} \mathrm{Cl}_{2}\right) ;{ }^{1} \mathrm{H} \operatorname{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta=7.43-7.51(\mathrm{~m}, 5 \mathrm{H})$,
$7.22-7.33(\mathrm{~m}, 10 \mathrm{H}), 7.10-7.20(\mathrm{~m}, 5 \mathrm{H}), 7.08(\mathrm{~d}, J=3.2 \mathrm{~Hz}, 1 \mathrm{H}), 7.04(\mathrm{dd}, J=8.4,1.6$ Hz, 1 H), $6.46(\mathrm{~d}, J=3.2 \mathrm{~Hz}, 1 \mathrm{H}), 5.29(\mathrm{~s}, 2 \mathrm{H}), 3.04(\mathrm{~d}, J=13.4 \mathrm{~Hz}, 1 \mathrm{H}), 2.90(\mathrm{~d}, J=$ $13.4 \mathrm{~Hz}, 1 \mathrm{H}), 2.62-2.66(\mathrm{~m}, 2 \mathrm{H}), 2.06-2.13(\mathrm{~m}, 1 \mathrm{H}), 1.67-1.75(\mathrm{~m}, 1 \mathrm{H}), 1.26(\mathrm{~s}, 3 \mathrm{H})$; ${ }^{13} \mathrm{C}$ NMR (125 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 148.5,148.1,137.8,135.3,129.8,128.9,128.7,128.3$, $128.1,128.0,127.7,127.0,126.5,126.4,126.2,126.0,124.8,122.5,109.1,101.5,88.4$, 85.4, 50.2, 48.5, 38.8, 36.9, 27.1; IR (film) 2924, 1485, $1447 \mathrm{~cm}^{-1}$; MS (ESI+) 458.2478 (458.2478 calcd for $\mathrm{C}_{33} \mathrm{H}_{31} \mathrm{NO}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be $96: 4$ er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 1 \%$ IPA/Hexanes, 1.00 $\mathrm{mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=11.6$ and 30.3 min ).

(+)-(S)-4-\{4-[(2-Methyl-5,5-diphenyltetrahydrofuran-2-yl)methyl]phenyl\}morpholine
$(2 m)$. The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol ( $51 \mathrm{mg}, 0.20 \mathrm{mmol}$ ) and 4-(4-bromophenyl)morpholine ( 87 mg , $0.36 \mathrm{mmol})$ using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and L 7 (7.5 $\mathrm{mg}, 0.010 \mathrm{mmol}$ ), a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $68.0 \mathrm{mg}, 82 \%$ ) as a colorless solid, $\mathrm{mp} 143-145{ }^{\circ} \mathrm{C}:[\alpha]^{23} \mathrm{D}+28.8\left(c 6.70, \mathrm{CH}_{2} \mathrm{Cl}_{2}\right) ;{ }^{1} \mathrm{H}$ NMR $\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.47$ (dd, $J=12.8,7.5 \mathrm{~Hz}, 4 \mathrm{H}), 7.22-7.33(\mathrm{~m}, 4 \mathrm{H}), 7.13-7.19(\mathrm{~m}, 2 \mathrm{H}), 7.12(\mathrm{~d}, J=8.3 \mathrm{~Hz}$, $2 \mathrm{H}), 6.79(\mathrm{~d}, J=8.3 \mathrm{~Hz}, 2 \mathrm{H}), 3.87(\mathrm{t}, J=4.7 \mathrm{~Hz}, 4 \mathrm{H}), 3.09-3.14(\mathrm{~m}, 4 \mathrm{H}), 2.88(\mathrm{~d}, J=$ 13.5 Hz, 1 H), 2.74 (d, J = $13.5 \mathrm{~Hz}, 1 \mathrm{H}$ ), 2.59-2.68 (m, 2 H), 1.98-2.03 (m, 1 H), 1.69$1.74(\mathrm{~m}, 1 \mathrm{H}), 1.23(\mathrm{~s}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(125 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 149.7,148.4,148.0,131.3$,
$130.3,128.1,128.0,126.5,126.5,126.1,125.9,115.4,88.4,85.1,67.1,49.7,47.6$, 38.7, 37.0, 27.0; IR (film) 2966, 1515, $1446 \mathrm{~cm}^{-1}$; MS (ESI+) 414.2427 (414.2428 calcd for $\mathrm{C}_{28} \mathrm{H}_{31} \mathrm{NO}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be $93: 7$ er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \% \mathrm{IPA} /$ Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 210 \mathrm{~nm}$, $R T=8.7$ and 10.7 min$)$.

(+)-(S)-4-[(2-Methyl-5,5-diphenyltetrahydrofuran-2-yl)methyl]benzonitrile (2n). The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1ol (51 mg, 0.20 mmol ) and 4-bromobenzonitrile ( $66 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathrm{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90{ }^{\circ} \mathrm{C}$ and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $27.0 \mathrm{mg}, 38 \%$ ) as a colorless solid, mp $100-104{ }^{\circ} \mathrm{C}:[\alpha]^{23}{ }_{\mathrm{D}}$ +23.9 (c 2.00, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H} \operatorname{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.47(\mathrm{~d}, \mathrm{~J}=8.1 \mathrm{~Hz}, 2 \mathrm{H}), 7.38-$ $7.42(\mathrm{~m}, 4 \mathrm{H}), 7.27-7.30(\mathrm{~m}, 4 \mathrm{H}), 7.22-7.25(\mathrm{~m}, 2 \mathrm{H}), 7.13-7.22(\mathrm{~m}, 2 \mathrm{H}), 2.90-2.95(\mathrm{~d}$, $J=13.5 \mathrm{~Hz}, 1 \mathrm{H}), 2.79-2.85(\mathrm{~d}, J=13.5 \mathrm{~Hz}, 1 \mathrm{H}), 2.55-2.67(\mathrm{~m}, 2 \mathrm{H}), 1.96-2.02(\mathrm{~m}, 1$ $\mathrm{H}), 1.79-1.86(\mathrm{~m}, 1 \mathrm{H}), 1.23(\mathrm{~s}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125 MHz, $\left.\mathrm{CDCl}_{3}\right) \delta$ 148.1, 147.4, 144.2, 131.7, 131.4, 128.2, 128.2, 126.8, 126.6, 126.0, 125.7, 119.4, 110.1, 88.8, 84.3, 48.5, 38.2, 37.5, 27.3; IR (film) 2925, 2223, $1607 \mathrm{~cm}^{-1}$; MS (ESI+) 376.1670 (376.1670 calcd for $\mathrm{C}_{25} \mathrm{H}_{23} \mathrm{NO}, \mathrm{M}+\mathrm{Na}^{+}$). The enantiopurity was determined to be $87: 13$ er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 2 \%$ IPA/Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}$, $\lambda 195 \mathrm{~nm}, \mathrm{RT}=7.5$ and 8.4 min$)$.


## (+)-(R)-\{4-[(2-Methyl-4,4-diphenyItetrahydrofuran-2-

yl)methyl]phenyl\}(phenyl)methanone (6). The general procedure was employed for the coupling of 4-methyl-2,2-diphenylpent-4-en-1-ol ${ }^{[3]}$ ( $51 \mathrm{mg}, 0.20 \mathrm{mmol}$ ) and (4bromophenyl)(phenyl)methanone ( $94 \mathrm{mg}, 0.36 \mathrm{mmol}$ ) using a catalyst composed of $\mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.7 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathbf{L} 7(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$, and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound ( $73 \mathrm{mg}, 84 \%$ ) as a light yellow oil. $[\alpha]^{23}{ }_{\mathrm{D}}=+0.01$ (c $5.9, \mathrm{CH}_{2} \mathrm{Cl}_{2}$ ); ${ }^{1} \mathrm{H}$ NMR (500 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 7.57-7.62(\mathrm{~m}, 1 \mathrm{H}), 7.46-7.52(\mathrm{~m}, 2 \mathrm{H}), 7.26-7.36(\mathrm{~m}, 11 \mathrm{H}), 7.16-$ $7.22(\mathrm{~m}, 2 \mathrm{H}), 4.53(\mathrm{~d}, J=9.5 \mathrm{~Hz}, 1 \mathrm{H}), 4.39(\mathrm{~d}, J=9.5 \mathrm{~Hz}, 1 \mathrm{H}), 2.90(\mathrm{~d}, J=13.2 \mathrm{~Hz}, 1$ H), $2.81(\mathrm{~d}, J=12.7 \mathrm{~Hz}, 1 \mathrm{H}), 2.73(\mathrm{~d}, J=13.2 \mathrm{~Hz}, 1 \mathrm{H}), 2.61(\mathrm{~d}, J=12.7 \mathrm{~Hz}, 1 \mathrm{H}), 1.12$ (s, 3 H ); ${ }^{13} \mathrm{C}$ NMR (125 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 196.7,146.5,143.4,138.0,135.7,132.4,130.6$, 130.1, 130.0, 128.6, 128.5, 128.4, 127.3, 126.4, 126.4, 83.7, 75.5, 56.5, 50.3, 47.8, 26.9; IR (film) 2926.7, 2247, 1654, $1276 \mathrm{~cm}^{-1}$; MS (ESI+) 433.2164 (433.2162 calcd for $\mathrm{C}_{31} \mathrm{H}_{28} \mathrm{O}_{2}, \mathrm{M}+\mathrm{H}^{+}$). The enantiopurity was determined to be 51:49 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 5 \% \mathrm{IPA} /$ Hexanes, $1.00 \mathrm{~mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}$, $R T=15.6$ and 21.3 min ).

## Determination of absolute configuration:

Product 2i was synthesized according to general procedure D. The optical rotation of this compound $\left([\alpha]^{23}{ }_{\mathrm{D}}+4.54\left(c 0.22, \mathrm{CHCl}_{3}\right)\right.$ ); was compared with that in the literature ${ }^{[9]}$ $\left(\mathrm{lit}[\alpha]^{23} \mathrm{D}+8.30\left(c \quad 0.6, \mathrm{CHCl}_{3}\right)\right)$. Both compounds were dextrorotatory, thus $\mathbf{2 i}$ was assigned the $(S)$ configuration on this basis.

(+)-(S,E)-5-[3-(4-Methoxyphenyl)allyl]-2,2-diphenyltetrahydrofuran (2i): The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol ( $48 \mathrm{mg}, 0.20$ mmol ) and (E)-1-(2-bromovinyl)-4-methoxybenzene ( $85 \mathrm{mg}, 0.40 \mathrm{mmol}$ ) using a catalyst composed of $2 \mathrm{~mol} \% \mathrm{Pd}_{2}(\mathrm{dba})_{3}(3.6 \mathrm{mg}, 0.004 \mathrm{mmol})$ and $\mathbf{L 7}(7.5 \mathrm{mg}, 0.010 \mathrm{mmol})$, a reaction temperature of $90^{\circ} \mathrm{C}$ and a reaction time of 12 h . This procedure afforded the title compound (14 mg, $18 \%$ ) as a colorless oil. $[\alpha]^{23}{ }_{\mathrm{D}}+4.54\left(c \quad 0.22, \mathrm{CHCl}_{3}\right)$; lit ${ }^{[9]}[\alpha]^{23}{ }_{\mathrm{D}}$ +8.30 (c 0.6, $\mathrm{CHCl}_{3}$ ); ${ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.46(\mathrm{~d}, \mathrm{~J}=7.5 \mathrm{~Hz}, 4 \mathrm{H}), 7.33-7.23$ (m, 6 H), 7.22-7.15 (m, 2 H), 6.83 (d, J = $8.8 \mathrm{~Hz}, 2 H$ ), $6.40(\mathrm{~d}, \mathrm{~J}=16.0 \mathrm{~Hz}, 1 \mathrm{H}), 6.83$ (dt, J = 15.7, 7.0 Hz, 1 H), 4.31-4.21 (m, 1 H), 3.80 (s, 3H), 2.70-2.58 (m, 2 H), 2.57$2.40(\mathrm{~m}, 2 \mathrm{H}), 2.06-1.86(\mathrm{~m}, 1 \mathrm{H}), 1.81-1.70(\mathrm{~m}, 1 \mathrm{H})$. Other spectroscopic data matched that of the literature. ${ }^{[9]}$ The enantiopurity was determined to be 79:21 er by chiral HPLC analysis (Chiralcel ADH, $25 \mathrm{~cm} \times 4.6 \mathrm{~mm}, 0.5 \%$ IPA/Hexanes, 1.00 $\mathrm{mL} / \mathrm{min}, \lambda 254 \mathrm{~nm}, \mathrm{RT}=17.5$ and 18.8 min$).$

## Screen of Chiral TADDOL-Derived Phosphite Ligands:



## References

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[2] B. E. Carpenter, I. R. Hunt, B. A. Keay, Tetrahedron: Asymmetry 1996, 7, 3107.
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Levêque, F. Mazé, S. Rosset, Eur. J. Org. Chem. 2000, 4011.
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Chemler, Angew. Chem. 2014, 126, 6501; Angew. Chem. Int. Ed. 2014, 53, 6383.


 study ouner garistaz Study owner krohem

Plot coste 2015-05-14




## 人 Agilent Technologies

## Sample Name <br> Sale collected 2016-05-14 <br> Fube sequence CARBON

Temperature 26
study owner garietuz
arintuz






## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. Hopkins 12 -arylcyclohexanols\RACcyclohexanol-BAH-7-176-1.00mL_min-4.00\%IPA-OJH.Icd

| Acquired by | : Admin |
| :--- | :--- |
| Sample Name | $:$ RACcyclohexanol-BAH-7-176-1.00mL_min-4.00\%IPA-OJH |
| Sample ID | $\vdots$ |
| Tray\# | $: 1$ |
| Vail \# | $: 1$ |
| Injection Volume | $\vdots 1$ uL |
| Data File Name | $:$ RACcyclohexanol-BAH-7-176-1.00mL_min-4.00\%IPA-OJH.Icd |
| Method File Name | :Cyclic Urea Method.Icm |
| Batch File Name | $\vdots$ |
| Report File Name | Default.Icr |
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| Data Processed | $: 6 / 17 / 2014$ 4:54:01 PM |

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PeakTable
PDA Ch1 254 nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 21.833 | 38905320 | 645897 | 49.439 | 55.274 |
| 2 | 24.956 | 39788133 | 522646 | 50.561 | 44.726 |
| Total |  | 78693453 | 1168542 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:\...|Data\B. Hopkins\2-arylcyclohexanolsICHIRAL-cyclohexanol-BAH-9-62-1.00mL_min-4.00\%IPA-OJH-3days.Icd
Acquired by : Admin
Sample Name
: CHIRAL-cyclohexanol-BAH-9-62-1.00mL_min-4.00\%IPA-OJH-3days
Sample ID
$\therefore 1$
Tray\#
: 1
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
: 1 uL
: CHIRAL-cyclohexanol-BAH-9-62-1.00mL_min-4.00\%IPA-OJH-3days.Icd
: Cyclic Urea Method.Icm
:
: Default.lcr
: 2/9/2015 10:31:38 AM
: 2/9/2015 11:41:41 AM
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1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 21.535 | 10056711 | 144136 | 99.784 | 99.956 |
| 2 | 25.931 | 21749 | 63 | 0.216 | 0.044 |
| Total |  | 10078460 | 144200 | 100.000 | 100.000 |




| Agilent Technologies | Sample Name <br> Date collected | 2015-03-16 | Pulse sequence <br> Solvent cdol3 | CARBON | Temperature 22 <br> Operator |
| :--- | :--- | :--- | :--- | :--- | :--- |




| Agilent Technologies | Sample Name <br> Date collected 2015-03-21 | Pulse sequence Solvent cdcl3 | Carbon | Temperature 22 Operator bahopki | Study owner Printed from | bahopki <br> kr.chem.Isa.umich.edu-vnmrs500 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |



## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalB. HopkinsISprio-RAC-BAH-8-92(1)-1.00\%IPA-1.00mL_min-OJH1.Icd

<Chromatogram>

C:ILabSolutions\DatalB. Hopkins\Sprio-RAC-BAH-8-92(1)-1.00\%IPA-1.00mL_min-OJH1.Icd
mAU


1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254 nm 4 nm

| Peak | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | :---: | ---: | ---: | ---: |
| 1 | 10.418 | 1241027 | 40824 | 49.700 | 53.782 |
| 2 | 12.802 | 1256030 | 35082 | 50.300 | 46.218 |
| Totail |  | 2497057 | 75905 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:L...IData\B. Hopkinsicyclopentylfused (3018-15 and on)\CHIRAL-BAH-9-147(2)-1.00\%IPA-1.00mL_min-OJH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed

CHIRAL-BAH-9-147(2)-1.00\%IPA-1.00mL_min-OJH
$\therefore$
$: 1$
1 uL
CHIRAL-BAH-9-147(2)-1.00\%IPA-1.00mL_min-OJH.Icd Cyclic Urea Method.Icm

Default.Icr 4/10/2015 10:45:11 PM 4/10/2015 11:20:21 PM
<Chromatogram>





## ==== Shimadzu LCsolution Analysis Report ====

C:I...IDatalB. Hopkinslcyclopentylfused (3018-15 and on)\RAC-BAH-9-58(5)-1.00\%IPA-1.00mL_min-ADH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed

RAC-BAH-9-58(5)-1.00\%IPA- 1.00 mL _min-ADH
$: 1$
$: 1$
: 1 uL
RAC-BAH-9-58(5)-1.00\%IPA-1.00mL_min-ADH.Icd Cyclic Urea Method.Icm

Default.Icr 5/8/2015 1:22:52 PM 5/8/2015 1:50:25 PM
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1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254 nm 4 nm

| Peak | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | :--- | ---: | ---: | ---: |
| 1 | 17.333 | 14045763 | 531427 | 49.132 | 53.520 |
| 2 | 18.224 | 14541794 | 461515 | 50.868 | 46.480 |
| Total |  | 28587558 | 992942 | 100.000 | 100.000 |

==== Shimadzu LCsolution Analysis Report ====

| Acquired by | : Admin |
| :---: | :---: |
| Sample Name | : CHIRAL-BAH-9-180(3)-1.00\%IPA-1.00mL_min-ADH |
| Sample ID |  |
| Tray\# | : 1 |
| Vail \# | : 1 |
| Injection Volume | : 1 uL |
| Data File Name | : CHIRAL-BAH-9-180(3)-1.00\%IPA-1.00mL_min-ADH.Icd |
| Method File Name | : Cyclic Urea Method.Icm |
| Batch File Name |  |
| Report File Name | : Default.lcr |
| Data Acquired | : 5/8/2015 2:54:37 PM |
| Data Processed | : 5/8/2015 3:18:40 PM |

<Chromatogram>


1 PDA Multi 1/254nm 4nm
PeakTable
PDA Ch1 254 nm 4 nm

| Peak | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 17.430 | 860939 | 30219 | 18.094 | 18.474 |
| 2 | 18.385 | 3897330 | 13358 | 81.96 | 81.526 |
| Totail |  | 4758269 | 163577 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

| C:ILabSolutions\DatalB. Hopkinslother THFSIRAC-BAH-9-149(1)-1.00\%IPA-1.50mL_min-OJH.Icd |  |
| :---: | :---: |
| Sample Name | : RAC-BAH-9-149(1)-1.00\%IPA-1.50mL_min-OJH |
| Sample ID |  |
| Tray\# | : 1 |
| Vail \# | : 1 |
| Injection Volume | : 1 uL |
| Data File Name | RAC-BAH-9-149(1)-1.00\%IPA-1.50mL_min-OJH.Icd |
| Method File Name | : Cyclic Urea Method.Icm |
| Batch File Name |  |
| Report File Name | Default.lcr |
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| Data Processed | : 4/12/2015 11:53:14 AM |

<Chromatogram>


## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalB. Hopkinslother THFSICHIRAL-BAH-9-166-1.00\%IPA-1.50mL_min-OJH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
: Admin
CHIRAL-BAH-9-166-1.00\%IPA-1.50mL_min-OJH
$: 1$
$: 1$
CHIRAL-BAH-9-166-1.00\%IPA-1.50mL_min-OJH.Icd
Cyclic Urea Method.Icm
Default.Icr
5/5/2015 6:02:55 PM
:5/5/2015 7:12:57 PM
<Chromatogram>
C:ILabSolutions\Data\B. Hopkinslother THFSICHIRAL-BAH-9-166-1.00\%IPA-1.50mL_min-OJH.Icd
mAU


1 PDA Multi $1 / 230 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 230 nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 19.272 | 16768556 | 273470 | 58.206 | 64.431 |
| 2 | 25.328 | 12040354 | 150966 | 41.794 | 35.569 |
| Total |  | 28808910 | 424436 | 100.000 | 100.000 |



2d

$\qquad$



## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalB. Hopkinsigemdiphenyl thflRAC-BAH-9-27-2.00\%IPA-1.00mL_min-ADH-2.Icd
Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired Admin
RAC-BAH-9-27-2.00\%IPA-1.00mL_min-ADH-2 1
$: 1$
$: 1 \mathrm{uL}$
: RAC-BAH-9-27-2.00\%IPA-1.00mL_min-ADH-2.Icd
Cyclic Urea Method.Icm

Data Processed

> Default.Icr
> 4/6/2015 11:36:45 AM
<Chromatogram>

C:ILabSolutions\DatalB. Hopkinsigemdiphenyl thflRAC-BAH-9-27-2.00\%IPA-1.00mL_min-ADH-2.Icd
mAU


2d

1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable

| PDACh1 254 nm 4nm |  |  |  |  |  |
| ---: | ---: | ---: | ---: | ---: | ---: |
| Peak $\#$ | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| 1 | 4.873 | 742501 | 75958 | 50.586 | 52.078 |
| 2 | 6.031 | 72592 | 6989 | 49.414 | 47.922 |
| Iotal |  | 1467793 | 145854 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. Hopkinsigemdiphenyl thfICHIRAL-BAH-9-144(1)-2.00\%IPA-1.00mL_min-ADH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed

Admin
CHIRAL-BAH-9-144(1)-2.00\%IPA-1.00mL_min-ADH
$: 1$
$: 1$
$: 1$
$: 1 u L$
$: 1 \mathrm{uL}$
: CHIRAL-BAH-9-144(1)-2.00\%IPA-1.00mL_min-ADH.Icd
: Cyclic Urea Method.Icm
: Default.Icr
4/7/2015 2:28:04 PM $: 4 / 7 / 2015$ 2:43:36 PM
<Chromatogram>


1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254 nm 4 nm

| Peakł | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 5.218 | 11628104 | 1328650 | 95.102 | 95.642 |
| 2 | 6.325 | 598911 | 60539 | 4.898 | 4.358 |
| Total |  | 12227015 | 1389189 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-152(1)-2.00\%IPA-1.00mL_min-ADH-2.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed

CHIRAL-BAH-9-152(1)-2.00\%IPA-1.00mL_min-ADH-2
$\vdots 1$
$: 1$
: 1 uL
CHIRAL-BAH-9-152(1)-2.00\%IPA-1.00mL_min-ADH-2.Icd Cyclic Urea Method.Icm

Default.lcr 4/16/2015 1:32:51 PM : $4 / 16 / 2015$ 1:50:10 PM
<Chromatogram>

C:ILabSolutions\Data\B. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-152(1)-2.00\%IPA-1.00mL_min-ADH-2.Icd mAU


1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254 nm 4nm

| Peak $\#$ | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 5.220 | 5795128 | 672196 | 95.568 | 96.400 |
| 2 | 6.345 | 268775 | 2501 | 4.432 | 3.600 |
| Totai |  | 6063903 | 697297 | 100.000 | 100.000 |




Data file /home/bahopki/vnmrsys/data/BAH-9-178-2proton.fid $\quad$ Plot date 2015-05-08




## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalB. Hopkinsigemdiphenyl thfIRAC-BAH-9-163(2)-5.00\%IPA-1.00mL_min-ADH.Icd
Acquired by Sample Name Sample ID
Tray\#
Vail \#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
RAC-BAH-9-163(2)-5.00\%IPA-1.00mL_min-ADH
$\vdots 1$
$: 1$
RAC-BAH-9-163(2)-5.00\%IPA-1.00mL_min-ADH.Icd
: Cyclic Urea Method.Icm
: Default.Icr
4/22/2015 11:43:27 PM : 4/23/2015 12:14:45 AM


2e

## <Chromatogram>



1 PDA Multi 1/254nm 4nm
PeakTable
PDA Ch1 254 nm 4 nm

| Peal\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | :--- | ---: | ---: | ---: |
| 1 | 9.987 | 18652646 | 1152095 | 50.859 | 54.585 |
| 2 | 11.867 | 18022436 | 958560 | 49.141 | 45.415 |
| Tota. |  | 36675082 | 2110655 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-171(1)-5.00\%IPA-1.00mL_min-ADH.Icd
Acquired by Sample Name Sample ID
Tray\#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired Admin
CHIRAL-BAH-9-171(1)-5.00\%IPA-1.00mL_min-ADH

Data Processed
$: 1$
$: 1$
: 1 uL
: CHIRAL-BAH-9-171(1)-5.00\%IPA-1.00mL_min-ADH.Icd Cyclic Urea Method.lcm

Default.Icr
: 5/2/2015 12:02:09 PM $: 5 / 2 / 2015$ 12:44:56 PM
<Chromatogram>


1 PDA Multi 1/254nm 4nm
PeakTable
PDACh1 254 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.734 | 8178331 | 542062 | 93.068 | 94.082 |
| 2 | 11.585 | 609165 | 34098 | 6.932 | 5.918 |
| Totai |  | 8787496 | 576160 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutionsIData\B. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-165(4)-5.00\%IPA-1.00mL_min-ADH.Icd

Acquired by
Sample Name Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed

Admin
CHIRAL-BAH-9-165(4)-5.00\%IPA-1.00mL_min-ADH
$: 1$
$: 1$
: CHIRAL-BAH-9-165(4)-5.00\%IPA-1.00mL_min-ADH.Icd
: Cyclic Urea Method.Icm
Default.Icr
4/24/2015 7:47:58 AM
: 4/24/2015 8:04:49 AM
<Chromatogram>


1 PDA Multi 1/254nm 4nm
PeakTable
PDA Ch1 254 nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.837 | 17433060 | 1144879 | 94.713 | 95.438 |
| 2 | 11.730 | 973158 | 54726 | 5.287 | 4.562 |
| Tota. |  | 18406218 | 1199605 | 100.000 | 100.000 |




## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutionsIData\B. Hopkinsigemdiphenyl thfIRAC-BAH-9-142(1)-2.00\%IPA-1.00mL_min-ADH.Icd
Acquired by Sample Name Sample ID
Tray\#
Vail \#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed : Admin
RAC-BAH-9-142(1)-2.00\%IPA-1.00mL_min-ADH
+
$\therefore 1$
$: 1$
$: 1$
$: 1$ uL
: 1 uL
: RAC-BAH-9-142(1)-2.00\%IPA-1.00mL_min-ADH.Icd Cyclic Urea Method.Icm

Default.Icr
4/6/2015 1:02:20 PM 4/6/2015 1:28:36 PM

<Chromatogram>
$2 f$
C:ILabSolutionsIDatalB. HopkinsIgemdiphenyl thfiRAC-BAH-9-142(1)-2.00\%IPA-1.00mL_min-ADH.Icd
mAU


1 PDA Multi $1 / 254 n m 4 n m$
PeakTable
PDACh1 254 nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 7.454 | 3362119 | 291487 | 50.829 | 53.807 |
| 2 | 8.805 | 3252515 | 250237 | 49.171 | 46.193 |
| Totai |  | 6614634 | 541724 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

| C:ILabSoluti Acquired by | atalB. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-144(2): Admin |
| :---: | :---: |
| Sample Name | : CHIRAL-BAH-9-144(2)-2.00\%IPA-1.00mL_min-ADH |
| Sample ID |  |
| Tray\# | : 1 |
| Vail \# | : 1 |
| Injection Volume | : 1 uL |
| Data File Name | : CHIRAL-BAH-9-144(2)-2.00\%IPA-1.00mL_min-ADH.Icd |
| Method File Name | : Cyclic Urea Method.lcm |
| Batch File Name |  |
| Report File Name | : Default.lcr |
| Data Acquired | : 4/7/2015 3:49:10 PM |
| Data Processed | : 4/7/2015 4:59:12 PM |

## <Chromatogram>



1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PDA Ch1 254 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 7.495 | 6299925 | 527840 | 94.803 | 95.510 |
| 2 | 8.872 | 345355 | 24817 | 5.197 | 4.490 |
| Totail |  | 6645280 | 552657 | 100.000 | 100.000 |


$\qquad$



## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutionsIData\B. Hopkinsigemdiphenyl thfIRAC-BAH-9-158(1)-1.00\%IPA-1.00mL_min-ADH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
: Admin
: RAC-BAH-9-158(1)-1.00\%IPA-1.00mL_min-ADH
$: 1$
$\vdots 1$
: 1 uL
: RAC-BAH-9-158(1)-1.00\%IPA-1.00mL_min-ADH.Icd Cyclic Urea Method.Icm

Default.Icr
4/20/2015 11:12:08 PM 4/20/2015 11:25:00 PM

<Chromatogram>


1 PDA Multi 1/230nm 4nm
PeakTable
PDA Ch1 230 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 6.922 | 16482730 | 762059 | 50.931 | 47.384 |
| 2 | 8.115 | 15880069 | 846210 | 49.069 | 52.616 |
| Total |  | 32362798 | 1608269 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutionsIData\B. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-170(I)-1.00\%IPA-1.0mL_min-ADH.Icd
Acquired by Admin
Sample Name
Sample ID
Tray\#
Injection Volume
Data File Name
CHIRAL-BAH-9-170(I)-1.00\%IPA-1.0mL_min-ADH
$: 1$
$: 1$

Method File Name
Batch File Name
Report File Name
Data Acquired
: 1 uL
CHIRAL-BAH-9-170(I)-1.00\%IPA-1.0mL_min-ADH.Icd
Cyclic Urea Method.Icm

Data Processed

$$
\begin{aligned}
& \text { Default.Icr } \\
& \text { 5/1/2015 6:05:32 PM }
\end{aligned}
$$

$$
\begin{aligned}
& 5 / 1 / 2015 \text { 6:05:32 PM } \\
& : 5 / 1 / 2015 \text { 6:46:19 PM }
\end{aligned}
$$

## <Chromatogram>

C:ILabSolutionsIDatalB. HopkinsIgemdiphenyl thfichIRAL-BAH-9-170(I)-1.00\%IPA-1.0mL_min-ADH.Icd
mAU


1 PDA Multi $1 / 230 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 230 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 7.076 | 15706142 | 769699 | 94.696 | 93.385 |
| 2 | 8.215 | 879721 | 54526 | 5.304 | 6.615 |
| Totail |  | 16585863 | 824224 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. Hopkinsigemdiphenyl thflCHIRAL-BAH-9-158(3)-1.00\%IPA-1.00mL_min-ADH.Icd
Acquired by Admin
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
CHIRAL-BAH-9-158(3)-1.00\%IPA-1.00mL_min-ADH
$\vdots$
$: 1$
CHIRAL-BAH-9-158(3)-1.00\%IPA-1.00mL_min-ADH.Icd
: Cyclic Urea Method.Icm
Default.Icr
: 4/21/2015 12:21:25 AM 4/21/2015 12:31:07 AM

## <Chromatogram>



1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable

| PDACh1 254 nm 4 nm |  |  |  |  |  |
| ---: | ---: | ---: | ---: | ---: | ---: |
| Peal | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| 1 | 6.990 | 891515 | 4479 | 95.481 | 94.667 |
| 2 | 8.204 | 42192 | 2521 | 4.519 | 5.333 |
| Totail |  | 933707 | 47270 | 100.000 | 100.000 |





## ==== Shimadzu LCsolution Analysis Report ====

## C:ILabSolutionsIData\B. Hopkinsigemdiphenyl thfIRAC-BAH-9-167(1)-2.00\%IPA-1.00mL_min-ADH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
RAC-BAH-9-167(1)-2.00\%IPA-1.00mL_min-ADH

Method File Name
Batch File Name
Report File Name
Data Acquired
$\vdots 1$
$: 1$
: RAC-BAH-9-167(1)-2.00\%IPA-1.00mL_min-ADH.Icd
: Cyclic Urea Method.Icm

Data Processed

> Default.Icr
> 5/3/2015 9:57:24 PM
: 5/3/2015 10:28:12 PM
<Chromatogram>


1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254 nm 4 nm

| Pealł | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | :---: | ---: | ---: | ---: |
| 1 | 10.836 | 1143534 | 65081 | 49.978 | 65.593 |
| 2 | 21.333 | 1144518 | 34138 | 50.022 | 34.407 |
| Total |  | 2288053 | 99219 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. HopkinsIgemdiphenyl thflCHIRAL-BAH-9-175(2)-2.00\%IPA-1.00mL_min-ADH.Icd
Acquired by
: Admin
Sample Name : CHIRAL-BAH-9-175(2)-2.00\%IPA-1.00mL_min-ADH
Sample ID
$\div$
Tray\# $\quad: 1$
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
$: 1$ uL
: CHIRAL-BAH-9-175(2)-2.00\%IPA-1.00mL_min-ADH.Icd
: Cyclic Urea Method.Icm
: Default.lcr
<Chromatogram>

C:ILabSolutions\DatalB. HopkinsIgemdiphenyl thflCHIRAL-BAH-9-175(2)-2.00\%IPA-1.00mL_min-ADH.Icd
mAU
(
1 PDA Multi 1/254nm 4nm

| PeakTable |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PDA Ch1 254nm 4nm |  |  |  |  |  |
| Peak\# | Ret. Time | Area | Height | Area \% | Height \% |
| 1 | 10.632 | 10096553 | 593722 | 93.160 | 96.529 |
| 2 | 21.141 | 741340 | 21347 | 6.840 | 3.471 |
| Total |  | 10837893 | 615069 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. HopkinsIgemdiphenyl thflRAC-BAH-9-138(1)-1.00\%IPA-0.50mL_min-ADH.Icd
Acquired by
: RAC-BAH-9-138(1)-1.00\%IPA-0.50mL_min-ADH
Sample Name
Sample ID
: 1
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
$: 1$
: 1 uL
: RAC-BAH-9-138(1)-1.00\%IPA-0.50mL_min-ADH.Icd
: Cyclic Urea Method.Icm
Default.|cr
: 4/3/2015 2:58:02 PM
Data Processed $\quad: 4 / 3 / 2015$ 3:22:05 PM

## <Chromatogram>



1 PDA Multi 1/254nm 4nm
PeakTable
PeakTable
PDA Ch1 254 nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | :--- | ---: | ---: | ---: |
| 1 | 17.433 | 11809441 | 465170 | 48.852 | 54.004 |
| 2 | 18.786 | 12364382 | 396186 | 51.148 | 45.996 |
| Total |  | 24173823 | 861356 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\B. HopkinsIgemdiphenyl thflCHIRAL-BAH-9-138(2)-1.00\%IPA-0.50mL_min-ADH.Icd
Acquired by : Admin
Sample Name : CHIRAL-BAH-9-138(2)-1.00\%IPA-0.50mL_min-ADH
Sample ID
Tray\# : 1
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
: 1
: 1 uL
: CHIRAL-BAH-9-138(2)-1.00\%IPA-0.50mL_min-ADH.Icd
Cyclic Urea Method.Icm

Data Processed
: Default.lcr
: 4/3/2015 3:26:59 PM
<Chromatogram>

C:ILabSolutions\Data\B. Hopkins\gemdiphenyl thflCHIRAL-BAH-9-138(2)-1.00\%IPA-0.50mL_min-ADH.Icd
mAU


1 PDA Multi $1 / 254 \mathrm{~nm} 4 n m$
PeakTable
PDA Ch1 254 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 17.493 | 9690833 | 388542 | 79.180 | 86.888 |
| 2 | 18.827 | 2548227 | 58633 | 20.820 | 13.112 |
| Total |  | 12239059 | 447175 | 100.000 | 100.000 |





## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac GIRAC-ZJG-2-189C-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min-ADH.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name Batch File Name
Report File Name
Data Acquired
Data Processed
: Admin
RAC-ZJG-2-189C-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min
$: 1$
$: 1$
: 1 uL
RAC-ZJG-2-189C-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min-ADH.Icd
: Cyclic Urea Method.lcm
Default.lcr
4/29/2015 2:38:55 PM
4/29/2015 2:57:12 PM
<Chromatogram>


## ==== Shimadzu LCsolution Analysis Report ====

| C:ILabSolu <br> Acquired by | IDatalZac GICHIRAL-ZJG-2-197-4bromobenzophenon-2.00\%IPA-01.0mL_minADH.Icd : Admin |
| :---: | :---: |
| Sample Name | : CHIRAL-ZJG-2-197-4bromobenzophenon-2.00\%IPA-01.0mL_minADH |
| Sample ID |  |
| Tray\# | : 1 |
| Vail \# | : 1 |
| Injection Volume | : 1 uL |
| Data File Name | : CHIRAL-ZJG-2-197-4bromobenzophenon-2.00\%IPA-01.0mL_minADH.Icd |
| Method File Name | : Cyclic Urea Method.lcm |
| Batch File Name |  |
| Report File Name | : Default.lcr |
| Data Acquired | : 5/2/2015 11:08:59 AM |
| Data Processed | : 5/2/2015 11:24:17 AM |

## <Chromatogram>



1 PDA Multi 1/195nm 4nm
PeakTable
PDA Ch1 195 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 10.101 | 10171837 | 637898 | 37.522 | 45.663 |
| 2 | 10.851 | 16936870 | 759064 | 62.478 | 54.337 |
| Total |  | 27108707 | 1396962 | 100.000 | 100.000 |






## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac G\RAC-ZJG-2-201A-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min1.Icd
Acquired by : Admin
Sample Name
: RAC-ZJG-2-201-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min1
Sample ID
$: 1$
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
: 1 uL
: RAC-ZJG-2-201A-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min1.Icd
Batch File Name
Report File Name
Data Acquired
:
5/14/2015 5:38:49 PM
Data Processed

$$
: 5 / 14 / 2015 \text { 6:13:17 PM }
$$

## <Chromatogram>

> C:ILabSolutions\Data\Zac GIRAC-ZJG-2-201A-1-DPEPhos-4bromobenzophenone-2.00\%IPA-01.0mL_min1.Icd mAU
> 1 PDA Multi 1/275nm 4nm
> PeakTable

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac GICHIRAL-ZJG-2-208B-Bligand-4bromobenzophenone-2.00\%IPA-01.0mL_min-ADH.Icd Acquired by : Admin Sample Name
: CHIRAL-ZJG-2-208B-Bligand-4bromobenzophenone-2.00\%IPA-01.0mL_mi
Sample ID
$\vdots 1$
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
$: 1$
: CHIRAL-ZJG-2-208B-Bligand-4bromobenzophenone-2.00\%IPA-01.0mL_min-ADH.Icd
Batch File Name
Report File Name
Data Acquired
: Cyclic Urea Method.lcm
: Default.Icr
: 5/14/2015 4:44:49 PM
Data Processed
: 5/14/2015 5:15:41 PM

## <Chromatogram>








## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\Zac GIRAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00\%IPA-01.0mL_min1.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
: Admin
:RAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00\%IPA-01.0mL_min1
$\vdots 1$
$: 1$
: 1 uL
RAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00\%IPA-01.0mL_min1.Icd
: Cyclic Urea Method.lcm

: Default.lcr
5/16/2015 11:18:00 AM
5/16/2015 12:05:19 PM

## <Chromatogram>

C:ILabSolutions\Data\Zac GIRAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00\%IPA-01.0mL_min1.Icd
mAU

1 PDA Multi $1 / 254 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 254 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 11.505 | 6336323 | 221869 | 49.463 | 80.036 |
| 2 | 27.434 | 6473788 | 55341 | 50.537 | 19.964 |
| Total |  | 12810111 | 277211 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac GICHIRAL-ZJG-3-6-A-Bligand-bromobnindole-1.0\%IPA-01.0mL_minAD1.Icd

Acquired by
Sample Name
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
: Admin
: CHIRAL-ZJG-3-6-A-Bligand-bromobnindole-1.0\%IPA-01.0mL_minAD1
$\vdots 1$
$: 1$
: 1 uL
: CHIRAL-ZJG-3-6-A-Bligand-bromobnindole-1.0\%IPA-01.0mL_minAD1.Icd
: Cyclic Urea Method.Icm
: Default.lcr
: 5/16/2015 12:06:37 PM
: 5/16/2015 1:06:37 PM
<Chromatogram>


1 PDA Multi 1/254nm 4nm
PDA Ch1 254 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 11.592 | 30684412 | 846134 | 95.635 | 98.815 |
| 2 | 30.326 | 1400511 | 10144 | 4.365 | 1.185 |
| Total |  | 32084923 | 856277 | 100.000 | 100.000 |




## Agilemt Technologies

Sample Name $\quad$ Puase sequence CARBON $\quad$ Temperature 2026
study ouncer garistux Study own
Prirted tom ariotar Sole coliected 2015-05-14 Solvent osola Fomperature arieta. ca.umloh.edu-wnerce 600


## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac GIRAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00\%IPA-01.0mL_min2.Icd Acquired by : Admin
Sample Name
: RAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00\%IPA-01.0mL_min
Sample ID
$\vdots 1$
Tray\#
Vail \#
Injection Volume
Data File Name
Method File Name
Batch File Name
Report File Name
Data Acquired
$: 1$ uL
: RAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00\%IPA-01.0mL_min2.Icd
: Cyclic Urea Method.lcm

Data Processed
: 5/14/2015 5:17:10 PM
: 5/14/2015 5:34:30 PM

## <Chromatogram>

C:ILabSolutions\DatalZac GIRAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00\%IPA-01.0mL_min2.Icd mAU


1 PDA Multi 1/210nm 4nm
PeakTable
PDA Ch1 210 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 8.637 | 10540544 | 739139 | 49.688 | 55.597 |
| 2 | 10.569 | 10672771 | 590323 | 50.312 | 44.403 |
| Total |  | 21213314 | 1329462 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac GICHIRAL-ZJG-2-205-1-4bromobenzomorpoli-2.00\%IPA-01.0mL_min.Icd
Acquired by Admin
Sample Name
CHIRAL-ZJG-2-205-1-4bromobenzomorpoli-2.00\%IPA-01.0mL_min
Sample ID
Tray\#
Vail \#
Injection Volume
Data File Name Method File Name
Batch File Name
Report File Name
Data Acquired
Data Processed
: 1
$: 1 \mathrm{uL}$
: CHIRAL-ZJG-2-205-1-4bromobenzomorpoli-2.00\%IPA-01.0mL min.Icd
: Cyclic Urea Method.lcm
: Default.lcr
5/11/2015 12:11:49 PM
5/11/2015 12:32:52 PM
<Chromatogram>





## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\DatalZac G\RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00\%IPA-01.0mL_min.Icd

| Acquired by | $:$ Admin |
| :--- | :--- |
| Sample Name | $:$ RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00\%IPA-01.0mL_min. |
| Sample ID | $\vdots 1$ |
| Tray\# | $: 1$ |
| Vail \# | $: 1$ uL |
| Injection Volume | $:$ RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00\%IPA-01.0mL_min.lcd |
| Data File Name | $:$ Cyclic Urea Method.Icm |
| Method File Name |  |
| Batch File Name | Default.lcr |
| Report File Name | $: 5 / 12 / 20153: 11: 17$ PM |
| Data Acquired | $: 5 / 12 / 20153: 20: 54$ PM |
| Data Processed |  |

## <Chromatogram>



C:ILabSolutionsIDataZZac G|RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00\%IPA-01.0mL min.lcd

1 PDA Multi $1 / 195 \mathrm{~nm} 4 \mathrm{~nm}$
PeakTable
PDA Ch1 195 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 7.467 | 10329364 | 884476 | 49.713 | 52.115 |
| 2 | 8.348 | 10448562 | 812688 | 50.287 | 47.885 |
| Total |  | 20777926 | 1697164 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions\Data\Zac GICHIRAL-ZJG-3-2-Bligand-bromobenzonitr-2.00\%IPA-01.0mL_min1ADH.Icd

Acquired by Sample Name Sample ID Tray\#
Vail \# Injection Volume Data File Name Method File Name Batch File Name Report File Name Data Acquired Data Processed
: Admin
RAC-ZJG-3-2-DPEPhos-bromobenzonitr-2.00\%IPA-01.0mL_min1ADH
$\vdots 1$
: 1
: 1 uL
: CHIRAL-ZJG-3-2-Bligand-bromobenzonitr-2.00\%IPA-01.0mL_min1ADH.Icd
Cyclic Urea Method.Icm
Default.lcr
5/13/2015 4:39:14 PM
: 5/13/2015 4:56:56 PM

## <Chromatogram>



1 PDA Multi 1/195nm 4nm
PeakTable
PDA Ch1 195 nm 4nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 7.484 | 20895001 | 1791445 | 86.563 | 87.549 |
| 2 | 8.371 | 3243362 | 254773 | 13.437 | 12.451 |
| Total |  | 24138363 | 2046218 | 100.000 | 100.000 |




## ==== Shimadzu LCsolution Analysis Report ====

C:ILabSolutions IDatalZac GITHFs\RAC-ZJG-3-96-DPEPhos-benzophenone-5.0\%IPA-1.0mL_minADH.Icd

| Acquired by | $:$ Admin |
| :--- | :--- |
| Sample Name | $:$ RAC-ZJG-3-96-DPEPhos-benzophenone-5.0\%IPA-1.0mL_minADH.Icd |
| Sample ID | $\vdots$ |
| Tray\# | $: 1$ |
| Vail \# | $: 1$ |
| Injection Volume | $: 1$ uL |
| Data File Name | $:$ RAC-ZJG-3-96-DPEPhos-benzophenone- $5.0 \%$ IPA-1.0mL_minADH.Icd |
| Method File Name | $:$ Cyclic Urea Method.Icm |
| Batch File Name | $\vdots$ |
| Report File Name | $:$ Default.Icr |
| Data Acquired | $: 8 / 14 / 2015$ 11:04:13 AM |
| Data Processed | $: 8 / 14 / 2015$ 11:29:30 AM |

<Chromatogram>


1 PDA Multi 1/254nm 4nm
PeakTable
PDA Ch1 254 nm 4 nm

| Peak\# | Ret. Time | Area | Height | Area $\%$ | Height $\%$ |
| ---: | ---: | :--- | ---: | ---: | ---: |
| 1 | 15.632 | 12769339 | 498328 | 49.746 | 57.006 |
| 2 | 21.332 | 12899764 | 375837 | 50.254 | 42.994 |
| Total |  | 25669103 | 874165 | 100.000 | 100.000 |

## ==== Shimadzu LCsolution Analysis Report ====

| Acquired by | : Admin |
| :---: | :---: |
| Sample Name | : CHIRAL-ZJG-3-97-1st-L7-benzophenone-5.0\%IPA-1.0mL_minADH.Icd |
| Sample ID | : |
| Tray\# | : 1 |
| Vail \# | : 1 |
| Injection Volume | : 1 uL |
| Data File Name | : CHIRAL-ZJG-3-97-1st-L7-benzophenone-5.0\%IPA-1.0mL_minADH.Icd |
| Method File Name | : Cyclic Urea Method.Icm |
| Batch File Name | : |
| Report File Name | : Default.lcr |
| Data Acquired | : 8/14/2015 11:33:50 AM |
| Data Processed | : 8/14/2015 11:59:52 AM |

<Chromatogram>


