Palladium/H⁺-Cocatalyzed Kinetic Resolution of tertiary Propargylic Alcohols

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General Information. NMR spectra were taken with an Agilent-400 spectrometer (400 MHz for ¹H NMR and 100 MHz for ¹³C NMR) in CDCl₃. Chemical shifts were recorded in ppm in relative to the TMS in CDCl₃ and coupling constants were reported in Hz. All reactions were carried out in flame-dried Schlenk tubes. $[PdCl(\pi-allyl)]_2$ was purchased from J&K Chemicals; (R) and (S)-DTBM-Segphos were purchased from Strem Chemicals Inc.; (PhO)₂POOH was purchased from Energy Chemical and purified through stirring with 1 N HCl, extracting with dichloromethane, and removing the solvent under vacuum; Anhydrous MeOH was purchased from Sinopharm Chemical Reagent Co., Ltd; Petroleum ether (b.p. 60-90 °C) was purchased from Shanghai Titan Scientific Co., Ltd. Toluene was dried over sodium wire with benzophenone as the indicator and distilled freshly before use. The reaction should be conducted in a hood working efficiently due to the toxicity of CO gas. All the temperatures are referred to the oil baths used. The NMR yields of allenoates and propargylic alcohols were determined by ¹H NMR analysis using 1,3,5-trimethylbenzene or dibromomethane as the internal standard. The starting propargylic alcohols were synthesized according to the reported procedures.¹ The apparatus used in this study is shown as follows:



Chinese and English comparison table for the HPLC spectra (Translations have been made in all the HPLC spectra presented here)

Chinese words	Corresponding English words		
实验内容简介	Brief introduction of experimental conditions		
色谱图	Chromatogram Figure		
电压 (mV)	Voltage (mV)		
时间 (min)	Time (min)		
峰号	Peak No.		
峰名	Peak name		
保留时间	Retention time		
峰高	Peak height		
峰面积	Peak area		
含量	Content		

Scheme S1 Known approaches to optically active tertiary propargylic alcohols-Part I a) Terminal alkyne approaches



Scheme S2 Known approaches to optically active tertiary propargylic alcohols-Part II



b) Alkynyl ketone approach

Calculation of selectivity factor s^2

In the kinetic resolution process, the selectivity factor *s* is related to the rate constants of the reaction of *R*- and *S*-enantiomers, k_R and k_{S} , respectively, by $s = k_R/k_S$, for $k_R > k_S$.



Basically, *s* can be established by the following equation for a first order reaction, in which C is the conversion and ee is the enatiomeric excess value of recovered substrate:

$$s = \frac{\text{Ln} [(1-C)(1-ee)]}{\text{Ln} [(1-C)(1+ee)]}$$

Besides, C can be calculated from the enatiomeric excess values of recovered substrate and product, ee and ee', respectively, through the equation C = ee/(ee+ee') if (*R*)-1a was stereospecifically transferred to (*S*_a)-2a and (*S*)-1a was completely transferred to (*R*_a)-2a in this kinetic resolution process.

However, in the current study, when (*R*)-1a was introduced into the standard reaction conditions with 98% ee, the product was obtained with only 90% ee, which makes the equation mentioned before not suitable here, thus, the conversion was determined by ¹H NMR analysis.



HO Ph (±)-1	[(π-allyl) (<i>R</i>)-DTBM- (PhO) ₂ I – <i>n</i> -Bu a 50	PdCl] ₂ (2 m Segphos (6 POOH (5 m OH (8 equiv. Solvent O balloon 0 °C, 10 h	ol%) 5 mol%) ol%)) Ph [*]	COOMe + n-Bu (S _a)- 2a	HO Ph (S)-1	<u>≕</u> — <i>n</i> -Bu a
		(S_a)	(S _a)-2a Recovery of		of 1a	
entry	Solvent	Yield	ee	NMR Yield	ee	s ^e
		/ % ^b	/ % ^c	/ % ^d	/ % ^c	
1	toluene	60	48	36 (35)	99	16.9
2	1,4-dioxane	21	49	74 (73)	25	7.6
3	CH ₃ CN	-	-	100 (-)	-	-
4	DME	-	-	100 (-)	-	-
5	THF	-	-	99 (-)	-	-
6 ^{<i>f</i>}	toluene	-	-	100 (-)	-	-

Table S1 Solvent effect in this kinetic resolution process ^a

^{*a*} Reaction conditions: (±)-1a (0.2 mmol), $[(\pi-allyl)PdCl]_2$ (2 mol%), (*R*)-DTBM-Segphos (6 mol%), (PhO)₂POOH (5 mol%), and MeOH (8 equiv.) in toluene (2 mL) at 50 °C under 1 atm of CO unless otherwise noted, DME = 1,2-dimethoxyethane, THF = tetrahydrofuran. ^{*b*} Isolated yields. ^{*c*} ee values of 2,3-allenoates and chiral propargylic alcohols were determined by HPLC. ^{*d*} NMR yields were determined by ¹H NMR analysis using 1,3,5-trimethylbenzene as the internal standard, the data in the parenthesis are isolated yields based on (±)-1a. ^{*e*} *s* = $k_{fast}/k_{slow} = ln[(recovery)(1-ee)]/ln[(recovery)(1+ee)], (recovery) is the recovery of 1a$ determined by ¹H NMR analysis, ee is the enantionmeric excesses value of recoveredsubstrate. ^{*f* $} 4 mol% PdCl₂ was used instead of 2 mol% [(<math>\pi$ -allyl)PdCl]₂.

$\stackrel{\text{HO}}{\longrightarrow}$ = r	[(π-allyl) (<i>R</i>)-DTBM- (PhO) ₂ I MeC	PdCl] ₂ (2 mol ^s Segphos (6 m POOH (5 mol ^s) DH (8 equiv.)	%) nol%) %)	COOMe	HO	<u>≕</u> — <i>n</i> -Bu	
Ph (<u>+</u>)- 1a	C	Foluene O balloon 10 h	Ph ⁽⁾ (S _a)	<i>n</i> -Bu)- 2a	Ph (<i>S</i>)-1	1a	
		(S_a) ·		2a Recovery			
entry	T / °C	Yield / % ^b	ee / % ^c	NMR Yield / % ^d	ee / % ^c	s ^e	
1	40	41	78	53 (50)	78	36.8	
2	45	52	54	39 (40)	91	11.4	
3 ^f	50	60	48	36 (35)	99	16.9	
4	55	72	38	14 (13)	99	5.1	

Table S2 Screening of reaction temperature in the kinetic resolution process ^a

а Reaction conditions: (±)-1a (0.5 mmol), $[(\pi-allyl)PdCl]_2$ (2 mol%). (R)-DTBM-Segphos (6 mol%), (PhO)₂POOH (5 mol%), and MeOH (8 equiv.) in toluene (2.5 mL) under 1 atm of CO unless otherwise noted. ^b Isolated yields. ^c ee values of 2,3-allenoates and chiral propargylic alcohols were determined by HPLC.^d NMR yields were determined by ¹H NMR analysis using 1,3,5-trimethylbenzene or dibromomethane as the internal standard, the data in the parenthesis are isolated yields based on (±)-1a. ^e $s = k_{fast}/k_{slow} = ln[(recovery)(1-ee)]/ln[(recovery)(1+ee)],$ (recovery) is the recovery of **1a** determined by ¹H NMR analysis, ee is the enantionmeric excesses value of recovered substrate.^f The reaction was carried out in 0.2 mmol scale.



Table S3 Screening of phosphoric acid and alcohol in the kinetic resolution process ^a

а Reaction mmol), conditions: (±)-1a (0.5) $[(\pi-allyl)PdCl]_2$ (2 mol%). (R)-DTBM-Segphos (6 mol%), (PhO)₂POOH (5 mol%), and MeOH (8 equiv.) in toluene (2.5 mL) at 50 °C under 1 atm of CO unless otherwise noted. ^b Isolated vields. ^c ee values of 2,3-allenoates and chiral propargylic alcohols were determined by HPLC. ^d NMR yields were determined by ¹H NMR analysis using 1,3,5-trimethylbenzene or dibromomethane as the internal standard, the data in the parenthesis are isolated yields based on (\pm) -1a. $e^{a} = k_{\text{fast}}/k_{\text{slow}}$ ln[(recovery)(1-ee)]/ln[(recovery)(1+ee)], (recovery) is the recovery of **1a** determined by ¹H NMR analysis, ee is the enantionmeric excesses value of recovered substrate. ^f The reaction was carried out in 0.2 mmol scale.

Experimental details and analytical data

1. Preparation of optically active tertiary propargylic alcohols and tetra-substituted allenoates

(1) Preparation of (S_a) -methyl 4-phenyl-2-butyl-2,3-pentadienoate $((S_a)$ -2a) (zwl-11-41-1) and (S)-2-phenyloct-3-yn-2-ol ((S)-1a) (zwl-11-41-2)



Typical Procedure: To a flame-dried Schlenk tube were added $[(\pi-allyl)PdCl]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (72.3 mg, 0.06 mmol), (PhO)₂POOH (12.4 mg, 0.05 mmol), (±)-**1a** (202.4 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) sequentially under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the liquid nitrogen bath was removed and the mixture was warmed up to room temperature and vigorously stirred at 50 °C with a balloon of CO for 10 h. The reaction mixture was then diluted with 5 mL of ethyl acetate, filtered through a short column silica gel (3.5 cm), eluted with ethyl acetate (20 mL), and concentrated. The residue was purified by column chromatography on silica gel to afford (*S*_a)-**2a** (144.2 mg, 59%) as an oil and (*S*)-**1a**³ (68.8 mg, 34%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 100/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)].

(*S*_a)-**2a**: 48% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 8.0 min, *t*_R (minor) = 9.3 min); [α]²²_D = +9.1 (*c* = 1.57, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.43-7.30 (m, 4 H, Ar-H), 7.28-7.20 (m, 1 H, Ar-H), 3.72 (s, 3 H, OCH₃), 2.35 (t, *J* = 7.6 Hz, 2 H, CH₂), 2.18 (s, 3 H, CH₃), 1.51-1.41 (m, 2 H, CH₂), 1.41-1.30 (m, 2 H, CH₂), 0.88 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 211.1, 167.8, 135.4, 128.5, 127.3,

125.9, 104.6, 102.0, 52.1, 30.2, 28.7, 22.3, 16.4, 13.9; MS (70 eV, EI) m/z (%): 244 (M⁺, 1.63), 143 (100); IR (neat): v = 2954, 2928, 2860, 1943, 1712, 1494, 1434, 1262, 1206, 1119, 1087, 1067, 1026 cm⁻¹; HRMS calcd for C₁₆H₂₀O₂ [M⁺]: 244.1463, found: 244.1466.

(*S*)-**1a**:³ 98% ee (HPLC conditions: OD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, $\lambda = 214$ nm, t_R (minor) = 22.9 min, t_R (major) = 26.0 min); $[\alpha]^{23}{}_D = -0.5$ (*c* = 1.10, CHCl₃) [90% ee, $[\alpha]^{30}{}_D = -0.3$ (c = 1.2, CHCl₃)]²; ¹H NMR (400 MHz, CDCl₃): $\delta = 7.71-7.62$ (m, 2 H, Ar-H), 7.41-7.31 (m, 2 H, Ar-H), 7.31-7.23 (m, 1 H, Ar-H), 2.38-2.22 (m, 3 H, OH and CH₂), 1.74 (s, 3 H, CH₃), 1.62-1.49 (m, 2 H, CH₂), 1.49-1.37 (m, 2 H, CH₂), 0.93 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): $\delta = 146.2$, 128.2, 127.5, 124.9, 85.7, 83.7, 70.0, 33.5, 30.7, 22.0, 18.4, 13.6; MS (70 eV, EI) *m*/*z* (%): 202 (M⁺, 1.18), 187 (100); IR (neat): *v* = 3393, 2958, 2931, 2872, 1493, 1447, 1365, 1327, 1232, 1180, 1093, 1063, 1027 cm⁻¹; Raman : *v* = 2240 cm⁻¹.

The following compounds were prepared according to Typical Procedure.

(2) Preparation of (S)-2-phenyloct-3-yn-2-ol ((S)-1a) (zwl-10-130)



The reaction of $[(\pi\text{-allyl})PdCl]_2$ (7.5 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1a** (202.2 mg, 1 mmol), MeOH (256.6 mg, 8.0 mmol), and toluene (5 mL) left (*S*)-**1a** (76.8 mg, 38%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 94% ee (HPLC conditions: OD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 21.2 min, t_R (major) = 24.9 min); $[\alpha]^{21}_D$ = -0.4 (*c* = 1.00, CHCl₃) [90% ee, $[\alpha]^{30}_D$ = -0.3 (*c* = 1.2, CHCl₃)]³; ¹H NMR (400 MHz, CDCl₃): δ = 7.69-7.62 (m, 2 H, Ar-H), 7.40-7.32 (m, 2 H, Ar-H), 7.32-7.24 (m, 1 H, Ar-H), 2.32-2.25 (m, 3 H, OH and CH₂), 1.74 (s, 3 H,

CH₃), 1.61-1.49 (m, 2 H, CH₂), 1.49-1.38 (m, 2 H, CH₂), 0.93 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 146.2, 128.1, 127.5, 124.9, 85.6, 83.6, 70.0, 33.5, 30.7, 22.0, 18.4, 13.6.



(3) Preparation of (S)-5-methyl-2-phenylhex-3-yn-2-ol ((S)-1b) (zwl-11-51)

The reaction of $[(\pi\text{-allyl})\text{PdCl}]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (72.4 mg, 0.06 mmol), (PhO)₂POOH (12.5 mg, 0.05 mmol), (±)-**1b** (188.4 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1b** (75.4 mg, 40%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 96% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, *t*_R (minor) = 8.1 min, *t*_R (major) = 10.1 min); $[\alpha]_{25}^{25}$ = +4.6 (*c* = 1.15, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.70-7.60 (m, 2 H, Ar-H), 7.41-7.31 (m, 2 H, Ar-H), 7.31-7.23 (m, 1 H, Ar-H), 2.72-2.58 (m, 1 H, CH), 2.43-2.32 (m, 1 H, OH), 1.73 (s, 3 H, CH₃), 1.21 (d, *J* = 7.2 Hz, 6 H, 2 x CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 146.2, 128.1, 127.4, 124.9, 91.0, 82.9, 69.9, 33.6, 22.9, 20.5; MS (70 eV, EI) *m/z* (%): 188 (M⁺, 1.70), 173 (100); IR (neat): *v* = 3292, 2972, 2926, 2872, 2241, 1447, 1366, 1318, 1234, 1186, 1089, 1048 cm⁻¹; HRMS calcd for C₁₃H₁₆O [M⁺]: 188.1201, found: 188.1203.





The reaction of $[(\pi-allyl)PdCl]_2$ (7.4 mg, 0.02 mmol), (*R*)-DTBM-Segphos (72.3 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-1c (236.6 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) left (*S*)-1c (104.1 mg, 44%) as an oil

[eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 90% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, *t*_R (minor) = 16.0 min, *t*_R (major) = 20.4 min); [α]²⁴_D = +0.7 (*c* = 1.69, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.69-7.59 (m, 2 H, Ar-H), 7.42-7.32 (m, 2 H, Ar-H), 7.32-7.22 (m, 1 H, Ar-H), 3.57 (t, *J* = 6.6 Hz, 2 H, CH₂), 2.38 (brs, 1 H, OH), 2.33 (t, *J* = 6.8 Hz, 2 H, CH₂), 1.98-1.83 (m, 2 H, CH₂), 1.80-1.60 (m, 5 H, CH₂ and CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 146.0, 128.2, 127.5, 124.9, 84.6, 84.4, 70.0, 44.5, 33.4, 31.5, 25.7, 18.0; MS (70 eV, EI) *m*/*z* (%): 238 (M⁺(³⁷Cl), 0.45), 236 (M⁺(³⁵Cl), 1.30), 221 (100); IR (neat): *v* = 3394, 2938, 2865, 2238, 1492, 1444, 1323, 1230, 1174, 1066 cm⁻¹; HRMS calcd for C₁₄H₁₇O³⁵Cl [M⁺]: 236.0968, found: 236.0970.

(5) Preparation of (S)-2-(4-chlorophenyl)oct-3-yn-2-ol ((S)-1d) (zwl-8-72)



The reaction of $[(\pi-allyl)PdCl]_2$ (7.5 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.8 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-1d (236.7 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) left (*S*)-1d (80.4 mg, 34%) as an oil [eluent: petroleum ether/ethyl acetate = 100/1 (1.5 L) to petroleum ether/ethyl acetate = 50/1]: 99% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 9.2 min, t_R (major) = 11.7 min); $[\alpha]^{20}_D$ = -0.6 (*c* = 1.20, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.61-7.54 (m, 2 H, Ar-H), 7.34-7.28 (m, 2 H, Ar-H), 2.42-2.38 (m, 1 H, OH), 2.27 (t, *J* = 7.2 Hz, 2 H, CH₂), 1.71 (s, 3 H, CH₃), 1.57-1.47 (m, 2 H, CH₂), 1.47-1.37 (m, 2 H, CH₂), 0.92 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 144.8, 133.2, 128.2, 126.5, 86.0, 83.3, 69.6, 33.6, 30.6, 22.0, 18.3, 13.6; MS (70 eV, EI) *m/z* (%): 238 (M⁺(³⁷Cl), 0.67), 236 (M⁺(³⁵Cl), 1.88), 221 (100); IR (neat): *v* = 3357, 2958, 2932, 2872, 1488, 1399, 1366, 1327, 1229, 1177, 1090, 1054, 1014 cm⁻¹; HRMS calcd for C₁₄H₁₇O³⁵Cl

[M⁺]: 236.0968, found: 236.0970.



The reaction of $[(\pi\text{-allyl})PdCl]_2$ (7.5 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1e** (309.1 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1e** (145.3 mg, 47%) as an oil [eluent: petroleum ether/ethyl acetate = 100/1 (1.5 L) to petroleum ether/ethyl acetate = 50/1]: 93% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 8.8 min, t_R (major) = 11.8 min); $[\alpha]^{20}_{D}$ = -1.6 (*c* = 1.21, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.55-7.49 (m, 2 H, Ar-H), 7.48-7.42 (m, 2 H, Ar-H), 2.43-2.35 (m, 1 H, OH), 2.25 (t, *J* = 7.2 Hz, 2 H, CH₂), 1.70 (s, 3 H, CH₃), 1.58-1.48 (m, 2 H, CH₂), 1.45-1.35 (m, 2 H, CH₂), 1.35-1.22 (m, 4 H, 2 x CH₂), 0.89 (t, *J* = 6.8 Hz, 3 H, CH₃ from *n*-C₆H₁₃); ¹³C NMR (100 MHz, CDCl₃): δ = 145.3, 131.2, 126.9, 121.4, 86.1, 83.2, 69.6, 33.6, 31.2, 28.5, 22.5, 18.7, 14.0; MS (70 eV, EI) *m*/*z* (%): 310 (M⁺(⁸¹Br), 2.31), 308 (M⁺(⁷⁹Br), 2.12), 293 (100); IR (neat): ν = 3385, 2955, 2929, 2858, 1681, 1589, 1485, 1466, 1394, 1365, 1271, 1228, 1177, 1074, 1010 cm⁻¹; HRMS calcd for C₁₆H₂₁O⁷⁹Br [M⁺]: 308.0776, found: 308.0773.

(7) Preparation of (S)-2-(2-methylphenyl)dec-3-yn-2-ol ((S)-1f) (zwl-8-187)



The reaction of $[(\pi\text{-allyl})PdCl]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1f** (244.1 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1f** (100.0 mg, 41%) as an oil [eluent: petroleum ether/ethyl acetate = 100/1 (1.5 L) to petroleum ether/ethyl acetate

= 50/1]: 91% ee (HPLC conditions: OJ-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 9.7 min, t_R (minor) = 10.4 min); $[\alpha]^{20}_D$ = -2.0 (*c* = 1.70, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.72-7.65 (m, 1 H, Ar-H), 7.20-7.12 (m, 3 H, Ar-H), 2.61 (s, 3 H, Ar-CH₃), 2.44 (s, 1 H, OH), 2.22 (t, *J* = 7.4 Hz, 2 H, CH₂), 1.79 (s, 3 H, CH₃), 1.56-1.45 (m, 2 H, CH₂), 1.45-1.20 (m, 6 H, 3 x CH₂), 0.88 (t, *J* = 7.2 Hz, 3 H, CH₃ from *n*-C₆H₁₃); ¹³C NMR (100 MHz, CDCl₃): δ = 142.8, 135.6, 132.2, 127.4, 125.6, 124.9, 85.3, 84.0, 69.7, 31.3, 31.1, 28.6, 28.5, 22.5, 21.2, 18.7, 14.0; MS (70 eV, EI) *m/z* (%): 244 (M⁺, 2.80), 229 (100); IR (neat): *v* = 3406, 2955, 2930, 2858, 1484, 1456, 1367, 1328, 1287, 1223, 1164, 1131, 1079, 1047 cm⁻¹; HRMS calcd for C₁₇H₂₄O [M⁺]: 244.1827, found: 244.1824.





The reaction of $[(\pi-\text{allyl})\text{PdCl}]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1g** (244.2 mg, 1 mmol), MeOH (256.4 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1g** (80.6 mg, 33%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 95% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 8.5 min, *t*_R (minor) = 10.7 min); $[\alpha]^{22}_{\text{D}}$ = -1.3 (*c* = 0.98, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.51-7.41 (m, 2 H, Ar-H), 7.29-7.19 (m, 1 H, Ar-H), 7.08 (d, *J* = 7.6 Hz, 1 H, Ar-H), 2.37 (s, 3 H, Ar-CH₃), 2.32 (brs, 1 H, OH), 2.27 (t, *J* = 7.2 Hz, 2 H, CH₂), 1.73 (s, 3 H, CH₃), 1.60-1.49 (m, 2 H, CH₂), 1.48-1.37 (m, 2 H, CH₂), 1.37-1.23 (m, 4 H, 2 x CH₂), 0.90 (t, *J* = 6.8 Hz, 3 H, CH₃ from *n*-C₆H₁₃); ¹³C NMR (100 MHz, CDCl₃): δ = 146.1, 137.8, 128.2, 128.1, 125.6, 122.0, 85.6, 83.8, 70.0, 33.5, 31.3, 28.6, 28.5, 22.5, 21.5, 18.37, 14.0; MS (70 eV, EI) *m*/*z* (%): 244 (M⁺, 2.27), 229 (100); IR (neat): *v* = 3403, 2955, 2929, 2858, 1607, 1485, 1457, 1365, 1329, 1255, 1199, 1158,

1084, 1062 cm⁻¹; HRMS calcd for C₁₇H₂₄O [M⁺]: 244.1827, found: 244.1825. (9) Preparation of (S)-2-(4-methylphenyl)oct-3-yn-2-ol ((S)-1h) (zwl-10-101)



The reaction of $[(\pi-\text{allyl})\text{PdCl}]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1h** (216.2 mg, 1 mmol), MeOH (256.2 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1h** (67.1 mg, 31%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 94% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 9.3 min, t_R (minor) = 13.0 min); $[\alpha]^{23}_{D}$ = -0.4 (*c* = 0.96, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.53 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.15 (d, *J* = 8.4 Hz, 2 H, Ar-H), 2.40-2.30 (m, 4 H, OH and Ar-CH₃), 2.27 (t, *J* = 7.2 Hz, 2 H, CH₂), 1.72 (s, 3 H, CH₃), 1.58-1.48 (m, 2 H, CH₂), 1.48-1.36 (m, 2 H, CH₂), 0.92 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 143.4, 137.1, 128.8, 124.9, 85.4, 83.9, 69.8, 33.4, 30.7, 21.9, 21.0, 18.4, 13.6; MS (70 eV, EI) *m*/*z* (%): 216 (M⁺, 2.20), 201 (100); IR (neat): v = 3384, 2957, 2930, 2863, 1509, 1456, 1405, 1365, 1327, 1234, 1184, 1089, 1052, 1020 cm⁻¹; HRMS calcd for C₁₅H₂₀O [M⁺]: 216.1514, found: 216.1516.

(10) Preparation of (S)-2-(2-naphthyl)oct-3-yn-2-ol ((S)-1i) (zwl-10-124)



The reaction of $[(\pi-\text{allyl})\text{PdCl}]_2$ (7.5 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1i** (252.5 mg, 1 mmol), MeOH (256.4 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1i** (75.8 mg, 30%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum

ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 98% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 22.0 min, t_R (minor) = 40.2 min); $[\alpha]^{23}_{D}$ = -11.8 (c = 1.55, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 8.11 (s, 1 H, Ar-H), 7.88-7.77 (m, 3 H, Ar-H), 7.77-7.70 (m, 1 H, Ar-H), 7.52-7.42 (m, 2 H, Ar-H), 2.47 (brs, 1 H, OH), 2.30 (t, J = 7.0 Hz, 2 H, CH₂), 1.82 (s, 3 H, CH₃), 1.63-1.51 (m, 2 H, CH₂), 1.51-1.39 (m, 2 H, CH₂), 0.94 (t, J = 7.2 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 143.5, 133.0, 132.7, 128.3, 128.0, 127.5, 126.1, 125.9, 123.6, 123.3, 85.9, 83.7, 70.1, 33.4, 30.7, 22.0, 18.4, 13.6; MS (70 eV, EI) m/z (%): 253 (M⁺+1, 2.05), 252 (M⁺, 7.42), 155 (100); IR (neat): ν = 3387, 3057, 2957, 2930, 2861, 1673, 1601, 1506, 1465, 1354, 1326, 1271, 1194, 1162, 1127, 1084, 1052, 1018 cm⁻¹; HRMS calcd for C₁₈H₂₀O [M⁺]: 252.1514, found: 252.1516.

(11) Preparation of (S)-2,4-diphenylbut-3-yn-2-ol ((S)-1j) (zwl-10-115)



The reaction of $[(\pi\text{-allyl})PdCl]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)_2POOH (12.5 mg, 0.05 mmol), (±)-**1j** (222.2 mg, 1 mmol), MeOH (256.4 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1j**² (73.3 mg, 33%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 96% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 17.2 min, t_R (minor) = 28.7 min); $[\alpha]^{23}_{D}$ = -7.2 (*c* = 1.23, CHCl₃) [96% ee, $[\alpha]^{30}_{D}$ = -7.4 (c = 1.3, CHCl₃)]²; ¹H NMR (400 MHz, CDCl₃): δ = 7.79-7.68 (m, 2 H, Ar-H), 7.55-7.44 (m, 2 H, Ar-H), 7.44-7.35 (m, 2 H, Ar-H), 7.35-7.26 (m, 4 H, Ar-H), 2.50 (s, 1 H, OH), 1.87 (s, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 145.6, 131.7, 128.4, 128.30, 128.26, 127.7, 125.0, 122.5, 92.4, 84.9, 70.3, 33.3; MS (70 eV, EI) *m/z* (%):

223 (M⁺+1, 4.20), 222 (M⁺, 30.84), 207 (100); IR (neat): v = 3298, 2978, 1597, 1488, 1442, 1402, 1365, 1270, 1212, 1140, 1088, 1073, 1050, 1027 cm⁻¹.





The reaction of $[(\pi\text{-allyl})PdCl]_2$ (7.5 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1k** (188.4 mg, 1 mmol), MeOH (256.6 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1k** (58.4 mg, 31%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 98% ee (HPLC conditions: AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.8 min, t_R (minor) = 7.2 min); $[\alpha]^{23}_{D}$ = -0.5 (*c* = 1.05, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.46-7.38 (m, 2 H, Ar-H), 7.35-7.27 (m, 3 H, Ar-H), 2.04 (s, 1 H, OH), 1.80-1.69 (m, 2 H, CH₂), 1.65-1.53 (m, 5 H, CH₃ and CH₂), 0.99 (t, *J* = 7.2 Hz, 3 H, CH₃ from *n*-Pr); ¹³C NMR (100 MHz, CDCl₃): δ = 131.6, 128.21, 128.18, 122.8, 92.9, 83.2, 68.6, 46.0, 29.8, 18.1, 14.2; MS (70 eV, EI) *m*/*z* (%): 188 (M⁺, 4.96), 145 (100); IR (neat): *v* = 3354, 2959, 2933, 2873, 1598, 1489, 1444, 1369, 1286, 1155, 1130, 1018 cm⁻¹; HRMS calcd for C₁₃H₁₆O [M⁺]: 188.1201, found: 188.1198.





The reaction of [(π-allyl)PdCl]₂ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1**l (216.3 mg, 1 mmol),

MeOH (256.4 mg, 8 mmol), and toluene (5 mL) left (*S*)-**11** (67.1 mg, 31%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 93% ee (HPLC conditions: IC column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 14.8 min, t_R (major) = 16.0 min); $[\alpha]^{22}_D$ = -1.0 (c = 1.00, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.46-7.38 (m, 2 H, Ar-H), 7.35-7.27 (m, 3 H, Ar-H), 2.11-2.02 (m, 1 H, OH), 1.82-1.67 (m, 2 H, CH₂), 1.67-1.50 (m, 5 H, CH₃ and CH₂), 1.42-1.30 (m, 4 H, 2 x CH₂), 0.91 (t, J = 7.0 Hz, 3 H, CH₃ from n-C₅H₁₁); ¹³C NMR (100 MHz, CDCl₃): δ = 131.6, 128.21, 128.18, 122.8, 93.0, 83.2, 68.7, 43.7, 31.9, 29.8, 24.4, 22.6, 14.0; MS (70 eV, EI) m/z (%): 216 (M⁺, 2.07), 145 (100); IR (neat): v = 3356, 2955, 2932, 2861, 1599, 1489, 1465, 1370, 1129, 1092, 1041 cm⁻¹; HRMS calcd for C₁₅H₂₀O [M⁺]: 216.1514, found: 216.1512.





The reaction of $[(\pi\text{-allyl})PdCl]_2$ (7.6 mg, 0.02 mmol), (*R*)-DTBM-Segphos (72.4 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-**1m** (196.1 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) left (*S*)-**1m** (51.0 mg, 26%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 96% ee (HPLC conditions: AD-H column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 16.4 min, t_R (minor) = 18.0 min); $[\alpha]^{25}_{D}$ = -0.3 (*c* = 1.20, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 2.19 (t, *J* = 6.8 Hz, 2 H, CH₂), 1.93-1.82 (brs, 1 H, OH), 1.72-1.57 (m, 2 H, CH₂), 1.54-1.21 (m, 13 H, CH₃ and 5 x CH₂), 1.00-0.83 (m, 6 H, 2 x CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 84.1, 83.7, 68.3, 43.9, 31.9, 30.8, 30.1, 24.4, 22.6, 21.9, 18.3, 14.0, 13.6; MS (ESI) *m/z* (%): 211 (M - OH+MeOH)⁺; IR (neat): *v* = 3367, 2957, 2932, 2862, 2238, 1461, 1371, 1329, 1130, 1055 cm⁻¹; HRMS calcd for C₁₃H₂₄O [M⁺]:

196.1827, found: 196.1830.





The reaction of $[(\pi-allyl)PdCl]_2$ (7.5 mg, 0.02 mmol), (S)-DTBM-Segphos (70.9 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (±)-1a (202.2 mg, 1 mmol), MeOH (256.1 mg, 8.0 mmol), and toluene (5 mL) left (R)-1a (60.7 mg, 30%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 98% ee (HPLC conditions: OD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, $t_{\rm R}$ (major) = 17.4 min, $t_{\rm R}$ (minor) = 19.3 min); $[\alpha]^{20}_{D}$ = +0.4 (c = 2.18, CHCl₃); ¹H NMR (400 MHz, CDCl₃); δ = 7.69-7.61 (m, 2 H, Ar-H), 7.39-7.31 (m, 2 H, Ar-H), 7.31-7.23 (m, 1 H, Ar-H), 2.33 (s, 1 H, OH), 2.28 (t, J = 7.2 Hz, 2 H, CH₂), 1.74 (s, 3 H, CH₃), 1.59-1.49 (m, 2 H, CH₂), 1.49-1.37 (m, 2 H, CH₂), 0.93 (t, J = 7.2 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR $(100 \text{ MHz}, \text{CDCl}_3)$: $\delta = 146.2, 128.2, 127.5, 124.9, 85.7, 83.7, 70.0, 33.5, 30.7, 22.0, 120.5, 12$ 18.4, 13.6; MS (70 eV, EI) m/z (%): 202 (M⁺, 1.33), 187 (100); IR (neat): v = 3386, 2958, 2931, 2862, 2238, 1447, 1365, 1327, 1231, 1179, 1093, 1062, 1027 cm⁻¹; Raman : v = 2240 cm⁻¹; HRMS calcd for C₁₄H₁₈O [M⁺]: 202.1358, found: 202.1361. (16) Preparation of (S_a) -methyl 2-propyl-4-phenyl-2,3-hexadienoate $((S_a)-2n)$ (zwl-8-173-1) and (S)-3-phenyloct-4-yn-3-ol ((S)-1n) (zwl-8-173-2)



The reaction of [(π-allyl)PdCl]₂ (7.4 mg, 0.02 mmol), (R)-DTBM-Segphos (70.9

mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (\pm)-**1n** (202.4 mg, 1 mmol), MeOH (256.1 mg, 8.0 mmol), and toluene (5 mL) afforded (S_a)-**2n** (154.0 mg, 63%) as an oil and left (*S*)-**1n** (48.6 mg, 24%) as an oil [eluent: petroleum ether/ethyl acetate = 100/1 (1.5 L) to petroleum ether/ethyl acetate = 50/1].

(*S*_a)-**2n**: 25% ee (HPLC conditions: IC column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, $\lambda = 214$ nm, *t*_R (major) = 6.5 min, *t*_R (minor) = 6.8 min); ¹H NMR (400 MHz, CDCl₃): $\delta = 7.41-7.28$ (m, 4 H, Ar-H), 7.28-7.20 (m, 1 H, Ar-H), 3.72 (s, 3 H, OCH₃), 2.54 (q, *J* = 7.2 Hz, 2 H, CH₂), 2.34 (t, *J* = 7.6 Hz, 2 H, CH₂), 1.57-1.43 (m, 2 H, CH₂), 1.15 (t, *J* = 7.6 Hz, 3 H, CH₃), 0.94 (t, *J* = 7.4 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 210.9, 168.0, 135.2, 128.5, 127.3, 126.2, 111.5, 103.8, 52.0, 31.1, 23.1, 21.5, 13.9, 12.3; MS (70 eV, EI) *m/z* (%): 245 (M⁺+1, 10.43), 244 (M⁺, 55.55), 229 (100); IR (neat): *v* = 2958, 2919, 2873, 1936, 1703, 1493, 1453, 1435, 1377, 1269, 1230, 1216, 1135, 1088, 1067, 1032 cm⁻¹; HRMS calcd for C₁₆H₂₀O₂ [M⁺]: 244.1463, found: 244.1466.

(*S*)-**1n**: 63% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, $\lambda = 214$ nm, t_R (minor) =6.2 min, t_R (major) = 7.5 min); ¹H NMR (400 MHz, CDCl₃): $\delta = 7.66-7.57$ (m, 2 H, Ar-H), 7.38-7.30 (m, 2 H, Ar-H), 7.30-7.22 (m, 1 H, Ar-H), 2.41-2.32 (m, 1 H, OH), 2.27 (t, *J* = 7.0 Hz, 2 H, CH₂), 2.02-1.80 (m, 2 H, CH₂), 1.64-1.52 (m, 2 H, CH₂), 1.02 (t, *J* = 7.4 Hz, 3 H, CH₃), 0.94 (t, *J* = 7.2 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): $\delta = 145.1$, 127.9, 127.4, 125.5, 86.6, 82.6, 73.9, 38.5, 22.1, 20.7, 13.5, 9.1; MS (ESI) *m*/*z* (%): 185 (M-OH)⁺; IR (neat): *v* = 3418, 2965, 2934, 2874, 2242, 1448, 1378, 1328, 1209, 1166, 1051, 1017 cm⁻¹; HRMS calcd for C₁₄H₁₈O [M⁺]: 202.1358, found: 202.1361.

(17) Preparation of (S_a) -ethyl 2-butyl-4-phenyl-2,3-pentadienoate $((S_a)$ -2aa) (zwl-11-18-1) and (S)-2-phenyloct-3-yn-2-ol ((S)-1a) (zwl-11-18-2)



The reaction of $[(\pi\text{-allyl})PdCl]_2$ (3.5 mg, 0.01 mmol), (*R*)-DTBM-Segphos (36.2 mg, 0.06 mmol), (PhO)₂POOH (6.4 mg, 0.05 mmol), (±)-**1a** (101.1 mg, 0.5 mmol), EtOH (184.1 mg, 4 mmol), and toluene (2.5 mL) afforded (S_a)-**2aa** (75.7 mg, 59%) as an oil and left (*S*)-**1a** (20.1 mg, 20%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)].

(*S*_a)-**2aa:** 41% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, *t*_R (minor) = 7.8 min, *t*_R (major) = 9.0 min; [α]²⁶_D = +28.2 (*c* = 1.17, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.44-7.28 (m, 4 H, Ar-H), 7.28-7.18 (m, 1 H, Ar-H), 4.19 (q, *J* = 7.2 Hz, 2 H, OCH₂), 2.34 (t, *J* = 7.4 Hz, 2 H, CH₃), 2.17 (s, 3 H, CH₃), 1.51-1.40 (m, 2 H, CH₂), 1.40-1.29 (m, 2 H, CH₂), 1.25 (t, *J* = 7.2 Hz, 3 H, CH₃), 0.88 (t, *J* = 7.4 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 211.1, 167.4, 135.7, 128.4, 127.2, 126.0, 104.5, 102.3, 60.8, 30.2, 28.7, 22.3, 16.4, 14.3, 13.9; MS (70 eV, EI) *m*/*z* (%): 258 (M⁺, 2.07), 143 (100); IR (neat): *v* = 2956, 2928, 2859, 1943, 1707, 1598, 1494, 1462, 1444, 1368, 1257, 1118, 1086, 1067, 1025 cm⁻¹; HRMS calcd for C₁₇H₂₂O₂ [M⁺]: 258.1620, found: 258.1623.

(*S*)-1a: 99% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, $\lambda = 214$ nm, t_R (major) = 13.7 min, t_R (minor) = 17.7 min); ¹H NMR (400 MHz, CDCl₃): $\delta = 7.69-7.62$ (m, 2 H, Ar-H), 7.40-7.32 (m, 2 H, Ar-H), 7.32-7.24 (m, 1 H, Ar-H), 2.33-2.24 (m, 3 H, OH and CH₂), 1.74 (s, 3 H, CH₃), 1.61-1.49 (m, 2 H, CH₂), 1.49-1.37 (m, 2 H, CH₂), 0.93 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu). For full characterization, see experiment No. 1. (1) at Page S9.

(18) Preparation of (S_a) -isopropyl 2-butyl-4-phenyl-2,3-pentadienoate $((S_a)$ -2ab) (zwl-11-19-1) and (S)-2-phenyloct-3-yn-2-ol ((S)-1a) (zwl-11-19-2)



The reaction of $[(\pi\text{-allyl})PdCl]_2$ (3.6 mg, 0.01 mmol), (*R*)-DTBM-Segphos (36.2 mg, 0.06 mmol), (PhO)₂POOH (6.4 mg, 0.05 mmol), (±)-**1a** (101.2 mg, 0.5 mmol), ^{*i*}PrOH (240.1 mg, 4 mmol), and toluene (2.5 mL) afforded (*S*_a)-**2ab** (46.3 mg, 34%) as an oil and left (*S*)-**1a** (31.4 mg, 31%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L) to petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)].

 (S_a) -**2ab**: 58% ee (HPLC conditions: Regis (*R*,*R*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.3 min, t_R (major) = 8.6 min; $[\alpha]^{26}_D$ = +51.2 (*c* = 1.20, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.43-7.29 (m, 4 H, Ar-H), 7.28-7.20 (m, 1 H, Ar-H), 5.13-4.99 (m, 1 H, OCH), 2.33 (t, *J* = 7.4 Hz, 2 H, CH₂), 2.17 (s, 3 H, CH₃), 1.50-1.38 (m, 2 H, CH₂), 1.38-1.28 (m, 2 H, CH₂), 1.28-1.17 (m, 6 H, 2 x CH₃), 0.88 (t, *J* = 7.4 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 211.1, 166.9, 135.9, 128.4, 127.2, 126.0, 104.4, 102.7, 68.1, 30.2, 28.6, 22.3, 21.9, 21.8, 16.4, 13.9; MS (70 eV, EI) *m/z* (%): 272 (M⁺, 1.62), 143 (100); IR (neat): *v* = 2957, 2928, 2860, 1944, 1704, 1494, 1463, 1373, 1260, 1105, 1066 cm⁻¹; HRMS calcd for C₁₈H₂₄O₂ [M⁺]: 272.1776, found: 272.1773.

(*S*)-1a: 99% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, $\lambda = 214$ nm, t_R (major) = 13.8 min, t_R (minor) = 18.4 min); ¹H NMR (400 MHz, CDCl₃): $\delta = 7.72$ -7.61 (m, 2 H, Ar-H), 7.42-7.32 (m, 2 H, Ar-H), 7.32-7.23 (m, 1 H, Ar-H), 2.35-2.20 (m, 3 H, OH and CH₂), 1.74 (s, 3 H, CH₃), 1.60-1.49 (m, 2 H, CH₂), 1.49-1.38 (m, 2 H, CH₂), 0.93 (t, *J* = 7.4 Hz, 3 H, CH₃ from *n*-Bu). For full characterization, see experiment No. 1. (1) at Page S9.

Synthesis of optically active 2,3-allenoates

(1) Preparation of (R_a) -methyl 4-phenyl-2-butyl-2,3-pentadienoate $((R_a)$ -2a) (zwl-11-65)



Following **Typical Procedure I**, the reaction of $[(\pi-\text{allyl})\text{PdCl}]_2$ (3.6 mg, 0.01 mmol), (*S*)-DTBM-Segphos (48.3 mg, 0.04 mmol), (PhO)₂POOH (12.4 mg, 0.05 mmol), (*S*)-**1a** (101.2 mg, 0.5 mmol, 98% ee), MeOH (128.4 mg, 4 mmol), and toluene (5 mL) afforded (R_a)-**2a** (105.1 mg, 86%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1]: 97% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.3 min, t_R (major) = 8.6 min); $[\alpha]^{20}_{\text{D}}$ = -35.8 (c = 1.25, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.44-7.29 (m, 4 H, Ar-H), 7.29-7.20 (m, 1 H, Ar-H), 3.72 (s, 3 H, OCH₃), 2.35 (t, J = 7.6 Hz, 2 H, CH₂), 2.18 (s, 3 H, CH₃), 1.52-1.41 (m, 2 H, CH₂), 1.41-1.30 (m, 2 H, CH₂), 0.88 (t, J = 7.4 Hz, 3 H, CH₃ from n-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 211.1, 167.8, 135.4, 128.5, 127.3, 125.9, 104.6, 102.0, 52.0, 30.2, 28.7, 22.3, 16.4, 13.8; MS (70 eV, EI) m/z (%): 244 (M⁺, 2.19), 143 (100); IR (neat): v = 2954, 2927, 2861, 1943, 1712, 1436, 1260, 1124, 1067 cm⁻¹; HRMS calcd for C₁₆H₂₀O₂ [M⁺]: 244.1463, found: 244.1460.

(2) Preparation of (R_a) -methyl 2-(4-chlorobutyl)-4-phenyl-2,3-pentadienoate $((R_a)-2c)$ (zwl-11-71)



Following Typical Procedure I, the reaction of $[(\pi-allyl)PdCl]_2$ (3.6 mg, 0.01

mmol), (*S*)-DTBM-Segphos (48.2 mg, 0.04 mmol), (PhO)₂POOH (12.4 mg, 0.05 mmol), (*S*)-**1c** (118.2 mg, 0.5 mmol, 96% ee), MeOH (128.6 mg, 4 mmol), and toluene (5 mL) afforded (R_a)-**2c** (119.7 mg, 86%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 100/1/1]: 96% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 9.4 min, t_R (major) = 10.6 min); $[\alpha]^{21}_D$ = -4.5 (c = 1.35, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.43-7.30 (m, 4 H, Ar-H), 7.30-7.21 (m, 1 H, Ar-H), 3.73 (s, 3 H, OCH₃), 3.50 (t, *J* = 6.6 Hz, 2 H, CH₂), 2.39 (t, *J* = 7.4 Hz, 2 H, CH₂), 2.19 (s, 3 H, CH₃), 1.87-1.75 (m, 2 H, CH₂), 1.70-1.56 (m, 2 H, CH₂); ¹³C NMR (100 MHz, CDCl₃): δ = 213.7, 166.5, 134.5, 132.9, 128.7, 128.30, 128.28, 127.8, 127.7, 126.1, 106.0, 104.3, 52.3, 16.3; MS (70 eV, EI) *m*/*z* (%): 280 (M⁺(³⁷Cl) , 1.75), 278 (M⁺(³⁵Cl) , 5.10), 143 (100); IR (neat): ν = 2950, 2864, 1942, 1710, 1493, 1436, 1251, 1099 cm⁻¹; HRMS calcd for C₁₆H₁₉O₂³⁵Cl [M⁺]: 278.1074, found: 278.1078.

(3) Preparation of (*R*_a)-methyl 2-(*n*-hexyl)-4-(4-bromophenyl)-2,3-pentadienoate ((*R*_a)-2e) (zwl-11-73)



Following **Typical Procedure I**, the reaction of $[(\pi\text{-allyl})PdCl]_2$ (3.6 mg, 0.01 mmol), (*S*)-DTBM-Segphos (48.2 mg, 0.04 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (*S*)-**1e** (154.4 mg, 0.5 mmol, 97% ee), MeOH (128.1 mg, 4 mmol), and toluene (5 mL) afforded (R_a)-**2e** (138.8 mg, 86%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 100/1/1]: 97% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.9 min, t_R (major) = 8.7 min); $[\alpha]^{21}_{D}$ = -18.0 (c = 1.89, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.45 (dt, J_1 = 8.9 Hz, J_2 = 2.2 Hz, 2 H, Ar-H), 7.23 (dt, J_1 = 8.8 Hz, J_2 = 2.3 Hz, 2 H, Ar-H), 3.73 (s, 3 H, OCH₃), 2.34 (t, J = 7.4 Hz, 2 H, CH₂), 2.15 (s, 3 H,

CH₃), 1.50-1.38 (m, 2 H, CH₂), 1.37-1.18 (m, 6 H, 3 x CH₂), 0.85 (t, J = 6.8 Hz, 3 H, CH₃ from n-C₆H₁₃); ¹³C NMR (100 MHz, CDCl₃): $\delta = 211.0$, 167.5, 134.5, 131.5, 127.5, 121.3, 103.9, 102.4, 52.1, 31.6, 28.9, 28.8, 27.9, 22.6, 16.3, 14.0; MS (70 eV, EI) m/z (%): 352 (M⁺(⁸¹Br), 1.27), 350 (M⁺(⁷⁹Br), 1.19), 142 (100); IR (neat): v = 1935, 1703, 1461, 1435, 1249, 1219, 1079, 1006 cm⁻¹; HRMS calcd for C₁₈H₂₃O₂⁷⁹Br [M⁺]: 350.0881, found: 350.0880.

(4) Preparation of (R_a) -methyl 2,4-diphenyl-2,3-pentadienoate $((R_a)-2j)$ (zwl-11-69)



Following **Typical Procedure I**, the reaction of $[(\pi\text{-allyl})PdCl]_2$ (3.6 mg, 0.01 mmol), (*S*)-DTBM-Segphos (48.2 mg, 0.04 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (*S*)-**1j** (111.0 mg, 0.5 mmol, 94% ee), MeOH (128.3 mg, 4 mmol), and toluene (5 mL) afforded (R_a)-**2j** (109.6 mg, 83%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 100/1/1]: 94% ee (HPLC conditions: PC-2 column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.6 min, t_R (major) = 8.8 min); $[\alpha]^{20}_D$ = +219.1 (c = 1.47, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.61-7.50 (m, 2 H, Ar-H), 7.50-7.40 (m, 2 H, Ar-H), 7.40-7.21 (m, 6 H, Ar-H), 3.80 (s, 3 H, OCH₃), 2.28 (s, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 213.6, 166.5, 134.5, 132.9, 128.7, 128.29, 128.27, 127.8, 127.6, 126.1, 106.0, 104.3, 52.3, 16.2; MS (70 eV, EI) m/z (%): 265 (M⁺+1, 16.85), 264 (M⁺, 75.94), 205 (100); IR (neat): v = 3026, 2950, 1931, 1715, 1491, 1436, 1270, 1241, 1194, 1169, 1019 cm⁻¹; HRMS calcd for C₁₈H₁₆O₂ [M⁺]: 264.1150, found: 264.1152.

(5) Preparation of (R_a) -methyl 2-phenyl-4-methyl-2,3-nonadienoate $((R_a)-2l)$ (zwl-11-70)



Following **Typical Procedure I**, the reaction of $[(\pi-\text{allyl})PdCl]_2$ (3.6 mg, 0.01 mmol), (*S*)-DTBM-Segphos (48.3 mg, 0.04 mmol), (PhO)₂POOH (12.4 mg, 0.05 mmol), (*S*)-**11** (108.0 mg, 0.5 mmol, 93% ee), MeOH (128.1 mg, 4 mmol), and toluene (5 mL) afforded (R_a)-**21** (105.7 mg, 82%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 100/1/1]: 92% ee (HPLC conditions: Regis (*S*,*S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (minor) = 11.3 min, t_R (major) = 12.3 min); $[\alpha]^{20}_{D}$ = +64.7 (c = 1.80, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.52-7.41 (m, 2 H, Ar-H), 7.37-7.27 (m, 2 H, Ar-H), 7.27-7.19 (m, 1 H, Ar-H), 3.78 (s, 3 H, OCH₃), 2.23-2.07 (m, 2 H, CH₂), 1.87 (s, 3 H, CH₃), 1.58-1.44 (m, 2 H, CH₂), 1.38-1.20 (m, 4 H, 2 x CH₂), 0.85 (t, J = 6.8 Hz, 3 H, CH₃ from n-Bu); ¹³C NMR (100 MHz, CDCl₃): δ = 209.4, 167.4, 134.0, 128.3, 128.1, 127.1, 105.1, 101.8, 52.0, 33.6, 31.3, 26.8, 22.4, 18.0, 14.0; MS (70 eV, EI) m/z (%): 259 (M⁺+1, 2.68), 258 (M⁺, 14.64), 143 (100); IR (neat): ν = 2926, 2858, 1948, 1715, 1435, 1272, 1196, 1175, 1021 cm⁻¹; HRMS calcd for C₁₇H₂₂O₂ [M⁺]: 258.1620, found: 258.1624.

2. Mechanistic studies





To a flame-dried Schlenk tube were added $[(\pi-allyl)PdCl]_2$ (3.6 mg, 0.01 mmol), (R)-DTBM-Segphos (36.0 mg, 0.03 mmol), (PhO)₂POOH (6.4 mg, 0.025 mmol), (S_a)-2a (122.0 mg, 0.5 mmol, 91% ee), MeOH (128.0 mg, 4 mmol), and toluene (2.5 mL) sequentially under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the liquid nitrogen bath was removed and the resulting mixture was warmed up to room temperature and vigorously stirred at 50 ^oC with a balloon of CO for 10 h. The reaction mixture was then diluted with ethyl acetate (5 mL), filtered through a short column silica gel (3.5 cm), eluted with ethyl acetate (20 mL), and concentrated. The residue was purified by column chromatography on silica gel to afford (S_a) -2a (117.0 mg, 96%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 100/1/1 (0.5 L)]: 91% ee (HPLC conditions: Regis (S,S) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, $t_{\rm R}$ (major) = 12.1 min, $t_{\rm R}$ (minor) = 14.4 min); ¹H NMR (400 MHz, CDCl₃): δ = 7.44-7.31 (m, 4 H, Ar-H), 7.30-7.22 (m, 1 H, Ar-H), 3.73 (s, 3 H, OCH₃), 2.35 (t, J = 7.4 Hz, 2 H, CH₂), 2.18 (s, 3 H, CH₃), 1.51-1.41 (m, 2 H, CH₂), 1.41-1.29 (m, 2 H, CH₂), 0.88 (t, J = 7.4 Hz, 3 H, CH₃ from *n*-Bu). For full characterization, see experiment No. 1. (1) at Page S8.

(2) Subjecting optically active allenoate to the reaction conditions applying(S)-DTBM-Segphos (zwl-11-67)



To a flame-dried Schlenk tube were added $[(\pi-allyl)PdCl]_2$ (3.6 mg, 0.01 mmol), (S)-DTBM-Segphos (36.0 mg, 0.03 mmol), (PhO)₂POOH (6.3 mg, 0.025 mmol), (S_a)-2a (122.0 mg, 0.5 mmol, 91% ee), MeOH (128.4 mg, 4 mmol), and toluene (2.5 mL) sequentially under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the liquid nitrogen bath was removed and the mixture was warmed up to room temperature and vigorously stirred at 50 °C with a balloon of CO for 10 h. The reaction mixture was then diluted with ethyl acetate (5 mL), filtered through a short column silica gel (3.5 cm), eluted with ethyl acetate (20 mL), and concentrated. The residue was purified by column chromatography on silica gel to afford (S_a)-2a (115.9 mg, 95%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 50/1/1 (0.5 L)]: 91% ee (HPLC conditions: Regis (S,S) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 7.6 min, $t_{\rm R}$ (minor) = 8.8 min); $[\alpha]^{24}_{\rm D}$ = +31.6 (c = 1.10, CHCl₃); ¹H NMR (400 MHz, $CDCl_3$): $\delta = 7.43-7.29$ (m, 4 H, Ar-H), 7.28-7.20 (m, 1 H, Ar-H), 3.72 (s, 3 H, OCH₃), 2.35 (t, J = 7.6 Hz, 2 H, CH₂), 2.18 (s, 3 H, CH₃), 1.52-1.41 (m, 2 H, CH₂), 1.41-1.29 (m, 2 H, CH₂), 0.88 (t, J = 7.4 Hz, 3 H, CH₃ from *n*-Bu). For full characterization, see experiment No. 1. (1) at Page S8.

(3) Subjecting optically active propargylic alcohol to the standard conditions (zwl-11-42)



To a flame-dried Schlenk tube were added $[(\pi-allyl)PdCl]_2$ (0.7 mg, 0.002 mmol), (R)-DTBM-Segphos (7.3 mg, 0.006 mmol), (PhO)₂POOH (1.2 mg, 0.005 mmol), (S)-1a (20.2 mg, 0.1 mmol, 98% ee), MeOH (25.7 mg, 0.8 mmol), and toluene (1 mL) sequentially under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the liquid nitrogen bath was removed and the resulting mixture was warmed up to room temperature and vigorously stirred at 50 °C with a balloon of CO for 2 h. The resulting mixture was then filtered through a short column of silica gel (3.5 cm), eluted with ethyl acetate (20 mL), and concentrated. The residue was purified by column chromatography on silica gel to afford (S)-1a (19.5 mg, 97%) as an oil [eluent: petroleum ether/ethyl ether/dichloromethane = 20/1/1 (0.5 L)]: 99% ee (HPLC conditions: OD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, $\lambda = 214$ nm, $t_{\rm R}$ (minor) = 22.6 min, $t_{\rm R}$ (major) = 25.3 min); ¹H NMR (400 MHz, CDCl₃): $\delta = 7.72-7.62$ (m, 2 H, Ar-H), 7.42-7.32 (m, 2 H, Ar-H), 7.32-7.23 (m, 1 H, Ar-H), 2.35-2.23 (m, 3 H, OH and CH₂), 1.74 (s, 3 H, CH₃), 1.60-1.49 (m, 2 H, CH₂), 1.49-1.38 (m, 2 H, CH₂), 0.93 (t, J = 7.4 Hz, 3 H, CH₃ from *n*-Bu). For full characterization, see experiment No. 1. (1) on page S9.

(4) Kinetic experiments (zwl-11-33)



To a flame-dried Schlenk tube were added $[(\pi-allyl)PdCl]_2$ (7.4 mg, 0.02 mmol), (*R*)-DTBM-Segphos (72.3 mg, 0.06 mmol), (PhO)₂POOH (12.6 mg, 0.05 mmol), (\pm)-**1a** (202.1 mg, 1 mmol), MeOH (256.1 mg, 8 mmol), and toluene (5 mL) sequentially under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the liquid nitrogen bath was removed and the mixture was warmed up to room temperature. Then the resulting mixture was stirred at 50 °C with a balloon of CO. 0.5 mL each of the aliquot was taken with an Ar-purged syringes after 1 h, 2 h, 3 h, 4 h, 5 h, 6 h. Each aliquot was immediately filtered through a short column silica gel (3.5 cm), eluted with ethyl acetate (20 mL), and concentrated. To the crude product was added 3.5 uL CH₂Br₂ as the internal standard. Then the mixture was analyzed by ¹H-NMR spectra to determine the recovery data of **1a**. All the data acquired were analyzed by Origin 8.0.

HO Ph (±)- 1a	[(π-allyI)PdC (<i>R</i>)-DTBM-Seg (PhO) ₂ POC <u>MeOH (</u> Toluene CO ba	CI] ₂ (2 mol%) pphos (6 mol%) DH (5 mol%) 8 equiv.) $a, 50 ^{\circ}\text{C}$ alloon (R_a) -2a	<i>l</i> e HO + <u>→</u> Ph (S)- 1 a
Entry	Time / h	NMR yield of 2a / %	Recovery of 1a / %
1	1	8	91
2	2	20	77
3	3	30	66
4	4	40	56
5	5	45	48
6	6	48	43

Table S4 Kinetic studies with the standard catalytic formula

Figure S1 Recovery of 1a vs. time



3. Synthetic applications

(1) Preparation of (2*R*)-2-phenyl-3-(*E*)-octen-2-ol ((2*R*)-*E*-3)⁴ (zwl-10-37)



To a flame-dried Schlenk tube were added (S)-1a (40.3 mg, 0.2 mmol, 98% ee) and ethyl ether (1 mL) under argon. The resulting mixture was then cooled down to -78 °C. To the reaction mixture was added a solution of Red-Al (3.5 M in toluene, 0.2 mL, 0.7 mmol) dropwise in 1 minute at -78 °C. Then the reaction mixture was stirred for 6 h at room temperature and subsequently quenched by dropwise addition of MeOH (2 mL) at -78 °C. To the mixture was added a saturated aqueous solution of potassium sodium tartrate (Rochelle's salt) (2 mL). After extraction with ethyl acetate (2 mL x 3), the organic layer was washed with brine (5 mL) and dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford (2R)-E-3⁵ (36.2 mg, 89%) as an oil [eluent: petroleum ether /ethyl ether/dichloromethane = 10/1/1]: 95% ee (HPLC conditions: AS-H column, hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 214 nm, $t_{\rm R}$ (minor) = 10.7 min, $t_{\rm R}$ (major) = 11.3 min); $[\alpha]^{20}_{\rm D}$ = +0.5 (c = 1.25, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.49-7.42 (m, 2 H, Ar-H), 7.38-7.29 (m, 2 H, Ar-H), 7.29-7.20 (m, 1 H, Ar-H), 5.78 (dt, J_1 = 15.6 Hz, J_2 = 1.2 Hz, 1 H, =CH), 5.67 (dt, J_1 = 15.6 Hz, J_2 = 6.5 Hz, 1 H, =CH), 2.11-2.01 (m, 2 H, CH₂), 1.91-1.84 (m, 1 H, OH), 1.63 (s, 3 H, CH₃), 1.42-1.25 (m, 4 H, 2 x CH₂), 0.89 (t, J = 7.0 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): $\delta = 147.3$, 136.8, 129.2, 128.1, 126.7, 125.2, 74.4, 31.9, 31.4, 29.9, 22.2, 13.9; MS (70 eV, EI) m/z (%): 204 (M⁺, 0.98), 147 (100); IR (neat): v = 3383, 2957, 2926, 2857, 1493, 1446, 1369, 1136, 1091, 1061, 1028 cm⁻¹. This reaction could not be monitored by TLC since the Rf values of starting material and product are the same. Prolonging reaction time would be necessary if the propargylic alcohol remains.



To a flame-dried Schlenk tube were added (S)-1a (40.4 mg, 0.2 mmol, 98% ee) and ethyl ether (1 mL) under argon. The resulting mixture was then cooled down to -78 °C. To the reaction mixture was added a solution of Red-Al (3.5 M in toluene, 0.2 mL, 0.7 mmol) dropwise in 1 minute at -78 °C. Then the cooling bath was removed and the mixture was stirred for 6 h at room temperature. After being cooled down to 0 °C. ethyl acetate (0.04 mL) was added and the resulting mixture was stirred at 0 °C for 0.5 h and then cooled down to -78 °C. A solution of I₂ (177.4 mg, 0.7 mmol) in THF (0.4 mL) was added dropwise in 1 minute. Then the reaction mixture was stirred for 12 h at 15 °C and quenched with dropwise addition of a saturated aqueous solution of NaS_2O_3 (5 mL) at 0 °C. After extraction with ethyl acetate (5 mL x 3), the organic layer was washed with brine (10 mL) and dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford (2R)-Z-4 (52.6 mg, 80%) as an oil [eluent: petroleum ether /ethyl acetate/dichloromethane = 40/1/1]: 96% ee (HPLC conditions: IC column, hexane/*i*-PrOH = 400/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 21.8 min, $t_{\rm R}$ (minor) = 25.4 min); $[\alpha]^{24}_{\rm D}$ = +0.3 (c = 1.00 , CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ = 7.52-7.40 (m, 2 H, Ar-H), 7.38-7.29 (m, 2 H, Ar-H), 7.29-7.21 (m, 1 H, Ar-H), 6.53 (s, 1 H, =CH), 3.19 (s, 1 H, OH), 2.55 (t, J = 7.2 Hz, 2 H, CH₂), 1.64 (s, 3 H, CH₃), 1.58-1.46 (m, 2 H, CH₂), 1.41-1.27 (m, 2 H, CH₂), 0.93 (t, J = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): $\delta = 147.1$, 140.3, 128.1, 126.8, 125.8, 108.6, 74.8, 46.4, 32.9, 31.5, 21.3, 13.8; MS (ESI) m/z (%): 185 (M - I - H₂O)⁺; IR (neat): v = 3444, 2956, 2928, 2858, 1627, 1492, 1446, 1370, 1336, 1113, 1090, 1065, 1028 cm⁻¹; HRMS calcd for C₁₄H₁₉OI [M⁺]: 330.0481, found: 330.0484. Prolonging reaction time at room temperature would be necessary before the addition of ethyl acetate if the propargylic alcohol remains.



(3) Preparation of (S_a) -N-methoxy-2-(2-phenyl-2,3-octadien-4-yl)benzamide $((S_a)$ -6a)⁶ (zwl-10-18)

To a Schlenk tube were added [Cp*RhCl₂]₂ (2.5 mg, 0.004 mmol), *N*-methoxybenzamide $5a^6$ (45.1 mg, 0.3 mmol), NaOAc (4.9 mg, 0.06 mmol), (S)-1a (40.6 mg, 0.2 mmol, 98% ee), and MeOH (1.2 mL) sequentially at room temperature. After being stirred for 72 h at room temperature, the reaction was complete as monitored by TLC. After filtration through a short column of silica gel (eluent: ethyl acetate 20 mL) and evaporation under reduced pressure, the crude product was purified by flash column chromatography on silica gel to afford (S_a) -6a (39.0 mg, 54%, 93% purity) as an oil [eluent: petroleum ether/ethyl acetate/dichloromethane = 10/1/0.5]: 94% ee (HPLC conditions: IC column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, $\lambda = 214 \text{ nm}, t_{\text{R}} \text{ (major)} = 10.4 \text{ min}, t_{\text{R}} \text{ (minor)} = 11.4 \text{ min}); [\alpha]^{26} = +309.4 (c = 1.67)$ CHCl₃); ¹H NMR (400 MHz, CDCl₃): $\delta = 8.83$ (brs, 1 H, NH), 7.55-7.11 (m, 9 H, Ar-H), 3.79 (s, 3 H, OCH₃), 2.59-2.28 (m, 2 H, CH₂), 2.18 (s, 3 H, CH₃), 1.57-1.43 (m, 2 H, CH₂), 1.43-1.31 (m, 2 H, CH₂), 0.87 (t, J = 7.4 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): $\delta = 203.7$, 167.5, 137.5, 137.2, 131.6, 130.6, 129.4, 128.8, 128.7, 128.33, 128.25, 127.1, 126.7, 125.6, 107.4, 102.6, 64.2, 33.8, 30.0, 22.3, 16.7, 13.8; MS (70 eV, EI) m/z (%): 336 (M⁺+1, 3.67), 335 (M⁺, 14.94), 260 (100); IR (neat): v = 3195, 2955, 2930, 2870, 2242, 1944, 1651, 1597, 1493, 1462, 1440, 1371,1299, 1027 cm⁻¹; HRMS calcd for $C_{22}H_{25}NO_2$ [M⁺]: 335.1885, found: 335.1886.
(4) Preparation of (S_a)-*N*-methoxy-4-bromo-2-(2-phenyl-2,3-octadien-4-yl)benzamide ((S_a)-6b)⁶ (zwl-10-50)



a Schlenk tube were added [Cp*RhCl₂]₂ (4.9 mg, 0.008 mmol), То *N*-methoxy-4-bromobenzamide $5b^6$ (69.2 mg, 0.3 mmol), NaOAc (5.0 mg, 0.06 mmol), (S)-1a (40.6 mg, 0.2 mmol, 98% ee), and MeOH (1.2 mL) sequentially at room temperature. After being stirred for 72 h at room temperature, the reaction was complete as monitored by TLC. After filtration through a short column of silica gel (eluent: ethyl acetate 20 mL) and evaporation under reduced pressure, the crude product was purified by flash column chromatography on silica gel to afford (S_a) -6b 52%, 91% purity) as an oil [eluent: petroleum ether/ethyl (47.3 mg, acetate/dichloromethane = 10/1/0.5]: 95% ee (HPLC conditions: IC column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R (major) = 11.6 min, t_R (minor) = 12.5 min); $\left[\alpha\right]_{D}^{26} = +198.4$ (c = 0.62, CHCl₃); ¹H NMR (400 MHz, CDCl₃): $\delta = 8.93$ (brs, 1 H, NH), 7.48 (s, 1 H, Ar-H), 7.43-7.23 (m, 6 H, Ar-H), 7.23-7.14 (m, 1 H, Ar-H), 3.77 (s, 3 H, OCH₃), 2.65-2.30 (m, 2 H, CH₂), 2.17 (s, 3 H, CH₃), 1.59-1.43 (m, 2 H, CH₂), 1.43-1.31 (m, 2 H, CH₂), 0.88 (t, J = 7.6 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (100 MHz, CDCl₃): $\delta = 203.9$, 166.6, 139.6, 136.8, 132.0 130.6, 130.2, 130.0, 128.4, 127.7, 127.0, 126.3, 125.7, 124.8, 106.4, 103.5, 64.3, 33.5, 29.9, 22.3, 16.6, 13.8; MS (70 eV, EI) m/z (%): 415 (M⁺(⁸¹Br), 9.12), 413 (M⁺(⁷⁹Br), 9.39), 43 (100); IR (neat): v = 3179, 2955, 2929, 2859, 1737, 1655, 1579, 1556, 1493, 1462, 1441,1372, 1240, 1079, 1028 cm⁻¹; HRMS calcd for C₂₂H₂₄NO₂⁷⁹Br [M⁺]: 413.0990, found: 413.0995.

References:

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zw1-11-41-1

报告时间: 2018-01-29, 14:52:40

实验时间: 2018-01-29,14:40:26 谱图文件:E:\data\zwl\zwl-11-41-1-whelk-s,s-200-1-1-214-18.1.29.org

实验内容简介; zw1-11-41-1-whe1k-s, s-200-1-1-214 Brief introduction of experimental conditions



分析结果表 10-2-

峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	n time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1		8.042	496307.219	7944497.000	73.9965
2		9.338	148625.797	2791813.250	26.0035
总计			644933.016	10736310.250	100.0000

zw1-9-139

报告时间: 2018-01-29, 14:50:14

实验时间: 2018-01-29, 14:21:05 谱图文件:E:\data\zwl\zwl-9-139-whelk-s, s-200-1-1-214-18, 1, 29, org

实验内容简介; zw1-9-139-whe1k-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name) 保留时间(Retent	tion time) 峰高(Peak height)	峰面积 (Peak	area) 含量 (Content)
1	8.290	500491.938	8495500.000	49.6469
2	9.772	434477.281	8616346.000	50.3531
总计		934969.219	17111846.000	100.0000





zw1-11-41-2

报告时间: 2018-01-29, 10:40:34

实验时间: 2018-01-29,10:08:44 谱图文件:E:\data\zwl\zwl-11-41-2-od-h-200-1-1-214-18,1.29.org

实验内容简介: zwl-11-41-2-od-h-200-1-1-214 Brief introduction of experimental conditions



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峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高 (Peak height)	峰面积(Peak area)	含量 (Content)
1		22.932	1823.750	47134.945	1.0650
2		25.695	28680.680	4378590.000	98.9350
总计			130504.429	4425724.945	100. 0000

zw1-10-177

实验时间: 2018-01-29,9:09:05
报告时间: 2018-01-29,9:42:01
谱图文件:E:\data\zwl\zwl-10-177-od-h-200-1-1-214-18.1.29.org
brief introduction of experimental conditions
实验内容简介:
zwl-10-177-od-h-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	on time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1		22.802	477322.750	15513478.000	49.9342
2		25.715	416485.000	15554379.000	50.0658
总计			893807.750	31067857.000	100.0000



S47



zw1-10-130

报告时间: 2017-10-29, 14:33:50

实验时间: 2017-10-29,14:01:42 谱图文件:E:\data\zwl\zwl-10-130-od-h-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd

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峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)) 峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1		21.162	1963. 569	56631.250	2.9201
2		24.888	52727.469	1882736.125	97.0799
总计			54691.038	1939367.375	100. 0000

zw1-9-184



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1		20.892 1	01460.320	3007729.750	49.6040
2		24.722	83459.898	3055757.750	50.3960
总计			184920.219	6063487.500	100.0000





zw1-11-51-whelk-r, r-100-1-1-214

报告时间: 2018-03-05, 19:29:56

实验时间: 2018-03-05, 17:54:12 谱图文件:D:\zhuguangjiong\zw1\20180305\zw1-11-51-whelk-r, r-100-1-1-214.org



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峰号(Peak No.)	峰名(Peak name) 保留时间(Reten	tion time) 峰高 (Peak height)	峰面积(Peak)	area) 含量 (Content)
1	8.100	10248.888	142264.516	2.1192
2	10.067	406127.938	6570812.500	97.8808
总计		416376.825	6713077.016	100.0000

zw1-11-47-whelk-r, r-100-1-1-214

报告时间: 2018-03-05, 19:26:45

实验时间: 2018-03-05, 17:34:13 谱图文件:D:\zhuguangjiong\zw1\20180305\zw1-11-47-whelkr, r-100-1-1-214.org



峰号(Peak No.)	峰名(Peak name)	保留时间(Retent	ion time) 峰高(Peak height)	峰面积(Peak	area) 含量(Content)
1		8.098	122880. 539	1531053.875	50.2793
2		9.798	84950.438	1514043. 500	49.7207
总计			207830.977	3045097.375	100.0000





zw1-11-53-whelk-r,r-100-1-1-214

报告时间: 2018-03-05, 19:33:41

实验时间: 2018-03-05,9:50:11 谱图文件:D:\zhuguangjiong\zw1\20180305\zw1-11-53-whelkr, r-100-1-1-214. org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)保留时间(Retention tir	me) 峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1	16.045	5581.602	136916.781	4.8496
2	20. 383	85107.570	2686343. 500	95.1504
总计		90689.172	2823260. 281	100.0000

zw1-8-119-whelk-r, r-100-1-1-214

报告时间: 2018-03-05, 19:31:22

实验时间: 2018-03-05, 10:46:32 谱图文件:D:\zhuguangjiong\zwl\20180305\zwl-8-119-whelkr, r-100-1-1-214. org



峰号(Peak No.)	峰名(Peak name) {	呆留时间(Rete	ntion time) 峰高 (Peak height)	峰面积(Peak	area) 含量 (Content)
1		15.348	310365.969	7532668.000	49.9841
2		19.582	228387.781	7537453.000	50.0159
总计			538753.750	15070121.000	100.0000





zw1-8-72-whe1k-200-1-1-214

实验时间: 2016-04-05, 12:08:52 报告时间: 2016-04-05, 14:57:10 谱图文件:D:\zhuguangjiong\zwl\20160405\zwl-8-72-whelk-200-1-1-214. org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time) 峰高(Peak height)	峰面积(Peak area)	含量(Content)
1		9.212	2170.168	32412.957	0.5475
2		11.678	317154. 500	5888037.500	99.4525
总计			319324.668	5920450.457	100.0000

zw1-8-19-whelk-200-1-1-214

实验时间: 2016-04-05, 12:42:42 报告时间: 2016-04-05, 14:55:18 谱图文件:D:\zhuguangjiong\zw1\20160405\zw1-8-19-whelk-200-1-1-214. org



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高(Peak height)	峰面积(Peak area)	含量(Content)
1		9.233 1	13819.242	1617175. 125	50.1100
2		11.752	89607.492	1610075. 500	49.8900
总计		1	203426. 734	3227250.625	100.0000





zw1-8-189-2-whe1k-200-1-1-214

报告时间: 2016-06-29, 15:33:13

实验时间: 2016-06-29, 15:02:56 谱图文件:D:\zhuguangjiong\zw1\20160628\zw1-8-189-2-whelk-200-1-1-214..org



峰号(Peak No.)	峰名(Peak name)保留时间(Retent	ion time) 峰高(Peak height)	峰面积(Peak	area) 含量(Content)
1	8.793	4195.861	115529.656	3.6861
2	11.807	150679.828	3018661.500	96.3139
总计		154875.689	3134191.156	100.0000

zw1-8-179-whe1k-200-1-1-214

实验时间: 2016-06-29,14:36:15 报告时间: 2016-06-29,15:32:21 谱图文件:D:\zhuguangjiong\zwl\20160628\zwl-8-179-whe1k-200-1-1-214..org



峰号(Peak No.)	峰名(Peak name) 保留时间	(Retention time) 峰高 (Peak height)	峰面积(Peak	area) 含量(Content)
1	9.017	225030.719	3837817.500	49.6912
2	11.882	208576.016	3885510.250	50. 3088
总计		433606.734	7723327.750	100.0000





zw1-8-187-2-oj-h-200-1-1-214

实验时间: 2016-06-29, 17:23:58 报告时间: 2016-06-29, 17:45:23 请图文件:D:\zhuguangjiong\zw1\20160628\zw1-8-187-2-oj-h-200-1-1-214..org



峰号(Peak No.)	峰名(Peak name) 保留时间(Retent	ion time) 峰高(Peak height)	峰面积(Peak	area) 含量(Content)
1	9.677	482537.094	6328731.000	95. 5790
2	10. 443	18904.176	292732.063	4. 4210
总计		501441.270	6621463.063	100.0000

zw1-8-177-oj-h-200-1-1-214

实验时间: 2016-06-29,16:56:01 报告时间: 2016-06-29,17:26:43 谱图文件:D:\zhuguangjiong\zwl\20160628\zwl-8-177-0j-h-200-1-1-214.org



峰号(Peak No.)	峰名(Peak name)	保留时间(Rete	ntion time) 峰高(Peak height)	峰面积(Peak a	area) 含量(Content)
1		9.605	216347.641	2735003.000	49.7589
2		10.343	200223. 797	2761504.250	50.2411
总计			416571.438	5496507.250	100.0000




实验时间: 2017-10-25、21:24:18 谱图文件:E:\data\zwl\zwl-10-129-whelk-s, s-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd 医rief introduction of experimental conditions 实验內容简介: zwl-10-129-whelk-s, s-200-1-1-214 报告时间: 2017-10-25, 21:38:47



峰号(Peak No.)	峰名(Peak name)	保留时间(Reten	tion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1		8.495	164122.281	2422488.750	97.3211
2		10.705	3391.566	66683.594	2.6789
总计			167513.847	2489172.344	100.0000

报告时间: 2017-10-25, 21:25:20

实验时间: 2017-10-25,21:10:46 谱图文件:E:\data\zwl\zwl-10-61-whelk-s,s-200-1-1-214-17.10.25.org 方法文件:E:\data\zwl\zwl.mtd

实验内容简介: zwl-10-61-whelk-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Reten	tion time) 峰高 (Peak height)	峰面积(Peak	area) 含量 (Content)
1		8.467	110892.078	1724765.625	50.0332
2		10.675	91118.008	1722478.000	49.9668
总计			202010.086	3447243.625	100.0000





S76

实验时间: 2017-10-14, 10:12:49 谱图文件:E:\data\zwl\zwl-10-101-whelk-s, s-200-1-1-214. org 方法文件:E:\data\zwl\zwl.mtd Sright Direction of experimental conditions 实验內容简介: zwl-10-101-whelk-s, s-200-1-1-214 报告时间: 2017-10-17, 14:08:31



峰号(Peak No.)	峰名(Peak name)	保留时间(Retent	tion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1		9.308	395926.438	6607310.000	97.1977
2		13.007	7925.763	190493.828	2.8023
总计			403852.200	6797803.828	100.0000

zw1-8-20

报告时间: 2017-10-14, 10:14:18

实验时间: 2017-10-14,9:54:41 请图文件:E:\data\zwl\zwl-8-20-whelk-s,s-200-1-1-214-17.10.14.org 方法文件:E:\data\zwl\zwl.mtd

Brief introduction of experimental conditions

实验内容简介: zw1-8-20 whe lk-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name) 保留	时间(Retention	time) 峰高(Peak height)	峰面积(Peak ar	ea) 含量 (Content)
1	ç	. 250	182265.719	3016561.000	50.0861
2	12	2. 888	130924.500	3006188.000	49.9139
总计			313190.219	6022749.000	100.0000





S80

报告时间: 2017-10-23, 21:20:54

实验时间: 2017-10-23,20:37:26 谱图文件:E:\data\zwl\zwl-10-124-whelk-s, s-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd

实验内容简介; zw1-10-124



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time) 峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1		21.993 1	001682.188	50106052.000	99. 1194
2		40.158	6194.592	445155.594	0.8806
总计		1	1007876. 779	50551207.594	100.0000

zw1-8-41

实验时间: 2017-10-23, 19:24:28 谱图文件:B:\data\zwl\zwl=8-41-whelk-s, s-200-1-1-214-17.10, 23.org 方法文件:E:\data\zwl\zwl.mtd Brief introduction of experimental conditions 报告时间: 2017-10-23, 20:39:15





峰号(Peak No.)	峰名(Peak name)	保留时间(Retention tim	e) 峰高(Peak height)	峰面积(Peak area	a) 含量 (Content)
1		22.320	167572.578	7365807.000	49.8083
2		39.767	93770. 930	7422519.500	50. 1917
总计			261343. 508	14788326.500	100.0000





实验时间: 2017-10-18, 19:19:10 谱图文件:E:\data\zwl\zwl-10-115-whelk-s, s-200-1-1-214. org 方法文件:E:\data\zwl\zwl.mtd 医rief introduction of experimental conditions 实验內容简介: zwl-10-T15-whelk-s, s-200-1-1-214 报告时间: 2017-10-18, 19:50:52



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time) 峰高(Peak height)	峰面积(Peak area) 含量 (Content)
1		17.217	162836.984	5330285.000	98.2242
2		28.675	1968.247	96368.727	1.7758
总计			164805.231	5426653.727	100.0000

zw1-7-28

报告时间: 2017-10-18, 18:49:50

实验时间: 2017-10-18, 18:17:25 谱图文件:E:\data\zwl\zwl-7-28-whelk-s, s-200-1-1-214-17, 10, 18, org 方法文件:E:\data\zwl\zwl.mtd

实验内容简介: xw1-7-28-whe1k-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	time) 峰高(Peak height)	峰面积(Peak;	area) 含量 (Content)
1		16.960	166608.766	5118363, 500	49.7273
2		27.185	84653.508	5174505.000	50.2727
总计			251262.273	10292868.500	100.0000





S88

报告时间: 2017-10-21, 10:23:23

实验时间: 2017-10-21,10:13:16 谱图文件:E:\data\zwl\zwl-10-118-ad-h-95-5-1-214.org 方法文件:E:\data\zwl\zwl.mtd

实验内容简介: zw1-10-118-ad-h-95-5-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高(Peak height)	峰面积(Peak area)	含量(Content)
1		6.787 2	263738.281	2291700.609	99.1329
2		7.212	1161.604	20045.147	0.8671
总计			264899.886	2311745. 756	100.0000

hcf-1-4

实验时间: 2017-10-21,9:52:30 谱图文件:E:\data\zwl\hcf-1-4-ad-h-95-5-1-214-17. 10.21. org 方法文件:E:\data\zwl\zwl.mtd 安验內容简介: hcf-1-4-ad-h-95-5-1-214 报告时间: 2017-10-21, 10:13:52



峰号(Peak No.)	峰名(Peak name)保留时间(Retent	ion time) 峰高(Peak height)	峰面积 (Peak	area) 含量 (Content)
1	6.803	530138.438	4628235.000	49.9353
2	7.230	503045.531	4640231.000	50.0647
总计		1033183.969	9268466.000	100.0000





报告时间: 2017-10-18, 20:56:37

实验时间: 2017-10-18,20:22:30 谱图文件:E:\data\zwl\zwl-10-116-IC-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd Seepa 容简介; zwl-10-116-ic-200-1-1-214





峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1		14.793	5519.999	105694.281	3. 5168
2		15.953 1	44096.719	2899716.500	96.4832
总计			149616.718	3005410. 781	100. 0000

实验时间: 2017-10-18,20:02:08 谱图文件:E:\data\zwl\zwl-10-63-IC-200-1-1-214-17.10.18.org 方法文件:E:\data\zwl\zwl.mtd 报告时间: 2017-10-18, 20:57:42

实验内容简介; zw1-10-63-1c-200-1-1-214



Э	Ð	T结	未	表	2
				1.00	-

		24 D1-M2			
峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time) 峰高 (Peak height)	峰面积(Peak area) 含量 (Content)
1		14.780	506005.750	9542713.000	49.9286
2		15.970	462844.156	9569991.000	50.0714
总计			968849.906	19112704.000	100.0000





zw1-11-25

实验时间: 2018-01-10,19:30:17 谱图文件:E:\data\zwl\zwl-11-25-ad-h-100-1-1-214-18.1.10.org 方法文件:E:\data\zwl\zwl.mtd 报告时间: 2018-01-10, 20:18:16 实验内容简介: zw1-11-25-ad-h-100-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)) 峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1		16.427	35815.621	763236. 188	98.0941
2		18.017	785.474	14828.752	1.9059
总计			36601.095	778064.939	100.0000

zw1-6-130

实验时间; 2018-01-10,20:16:41 谱图文件:E:\data\zwl\zwl-6-130-ad-h-100-1-1-214-18.1.10.org 方法文件:E:\data\zwl\zwl.mtd 报告时间: 2018-01-10, 20:38:18

实验内容简介; zw1=6=130=ad-h=100=1=1=214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)) 峰高(Peak height)	峰面积(Peak area) 含量 (Content)
1		16.670	20019.238	408204.313	50.4626
2		18.287	18316.678	400720.000	49.5374
总计			38335.916	808924.313	100.0000





实验时间: 2017-06-19,21:05:41 谱图文件:D:\data\zwl\zwl-10-24-od-h-200-1-1-214.org 报告时间: 2017-06-19, 21:33:07 which are the second s



峰号(Peak No.)	峰名(Peak name) 保留时间(Reten	tion time) 峰高(Peak height)	峰面积(Peak area) 含量 (Content	
1	17.352	314792. 344	6976912.500	99.1730
2	19.277	2288.860	58176.422	0.8269
总计		317081.204	7035088.922	100.0000

zw1-9-184





峰号(Peak No.)	峰名(Peak name)	保留时间(Reten	tion time) 峰高(Peak height)	峰面积(Peak ar	rea) 含量 (Content)
1		17.728	150981.813	3379982.000	49.9012
2		19.398	136656.313	3393361.250	50.0988
总计			287638.125	6773343.250	100.0000





zw1-8-173-1-ic-200-1-1-214

实验时间: 2016/6/17,13:06:41 报告时间: 2016/6/17,18:22:24 谱图文件:D:\zhuguangjiong\zwl\20160616\zwl-8-173-1-ie-200-1-1-214...org

实验内容简介:



峰号(Peak No.)	峰名(Peak name) 保留时间(Retent	ion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1	6.453	956853.938	7425737.000	62.5994
2	6.838	536793.813	4436579.000	37.4006
总计		1493647.750	11862316.000	100.0000

zw1-8-25-ic-200-1-1-214

实验时间: 2016/6/17,12:48:06 报告时间: 2016/6/17,18:21:40 谱图文件:D:\zhuguangjiong\zwl\20160616\zwl-8-25-ic-200-1-1-214..org

实验内容简介:



峰号(Peak No.)	峰名(Peak name) 保留时间(Retent	tion time) 峰高 (Peak height)	峰面积(Peak	area) 含量 (Content)
1	6.462	1121451. 125	8762396.000	49.8890
2	6.845	1028698.438	8801383.000	50.1110
总计		2150149.563	17563779.000	100.0000




zw1-8-173-2-whelk-200-1-1-214

报告时间: 2016/6/17, 18:20:38

实验时间: 2016/6/17,9:02:41 谱图文件:D:\zhuguangjiong\zw1\20160616\zw1-8-173-2-whelk-200-1-1-214..org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)	保留时间(Retent	tion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1		6.162	63770.379	796828.250	18.4311
2		7.488	295348.969	3526455.750	81.5689
总计			359119.348	4323284.000	100.0000

zw1-8-16-whelk-200-1-1-214

实验时间: 2016/6/17,9:23:33 报告时间: 2016/6/17,18:19:48 谱图文件:D:\zhuguangjiong\zw1\20160616\zw1-8-16-whelk-200-1-1-214..org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention tim	e) 峰高(Peak height)	峰面积(Peak area) 含量(Content)
1		6.105	437453. 531	5007110.500	49.8224
2		7.407	423874.688	5042801.500	50.1776
总计			861328.219	10049912.000	100.0000





zw1-11-18-1-whe1k-r, r-200-1-1-214

报告时间: 2018-01-19, 14:23:23

实验时间: 2018-01-09, 13:35:56 谱图文件:D:\zhuguangjiong\zwl\20180109\zwl-11-18-1whelk-r, r-200-1-1-214..org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	on time) 峰高(Peak height)	峰面积(Peak	area) 含量(Content)
1		7.765	155384.359	2273376.000	29.6115
2		9.015	361441.781	5403972.500	70.3885
总计			516826.141	7677348.500	100.0000

zw1-8-113-whelk-r, r-200-1-1-214

报告时间: 2018-01-19, 14:21:00

实验时间: 2018-01-09,13:06:36 谱图文件:D:\zhuguangjiong\zw1\20180109\zw1-8-113-whelkr, r-200-1-1-214.org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)保留时间(Retenti	ion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1	7.815	64914.402	905980.188	49.7961
2	9.065	61993. 223	913399. 500	50.2039
总计		126907.625	1819379.688	100.0000



zw1-11-18-2

实验时间: 2018-01-03, 16:10:23 谱图文件:E:\data\zwl\zwl-11-18-2-whelk-s, s-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd Brief introduction of experimental conditions 实验内容简介: zwl-11-18-2-whelk-s, s-200-1-1-214 报告时间: 2018-01-03, 16:41:02



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)) 峰高 (Peak height)	峰面积(Peak area)	含量 (Content)
1		13.685	129390. 258	4035790.750	99.7614
2		17.730	370.315	9653.605	0.2386
总计			129760. 572	4045444. 355	100.0000

zw1-10-177

分析结果表							
峰号 (Peak No.)	峰名 (Peak name) 保留时间 (Rete	ention time) 峰高(Peak height)	峰面积 (Peak	area) 含量 (Content)			
1	13.050	298480.719	13337632.000	50.2526			
2	16.995	370224.250	13203545.000	49.7474			
总计		668704.969	26541177.000	100.0000			





zw1-11-19-1-whe1k-r, r-200-1-1-214

报告时间: 2018-01-19, 14:24:17

实验时间: 2018-01-09, 15:37:43 谱图文件:D:\zhuguangjiong\zwl\20180109\zwl-11-19-1whelk-r, r-200-1-1-214..org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	time) 峰高(Peak height)	峰面积(Peak area) 含量(Content)
1		7.340	141313.141	2368081.750	21.2115
2		8.607	597790. 563	8796077.000	78.7885
总计			739103. 703	11164158.750	100.0000

zw1-8-114-whelk-r, r-200-1-1-214

报告时间: 2018-01-19, 14:21:53

实验时间: 2018-01-09, 15:09:31 谱图文件:D:\zhuguangjiong\zw1\20180109\zw1-8-114-whelkr, r-200-1-1-214.org

实验内容简介:



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention ti	me) 峰高(Peak height)	峰面积(Peak area) 含量(Content)
1		7.317	212267.281	3824037.000	50.0884
2		8.612	255875.906	3810538.000	49.9116
总计			468143.188	7634575.000	100.0000



zw1-11-19-2

实验时间: 2018-01-03,16:36:32 谱图文件:E:\data\zwl\zwl-11-19-2-whelk-s,s-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd 报告时间: 2018-01-03, 17:03:14

w验内容简介 zw1-11-19-2 whelk-s, s-200-1-1-214



峰号(Peak No.)	峰名 (Peak name) 保留时间 (Rete	ntion time) 峰高 (Peak height)	峰面积 (Peak	area) 含量 (Content)
1	13.847	139892.453	4895737.500	99.7662
2	18.433	423.490	11471.078	0.2338
总计		140315.943	4907208.578	100.0000

zw1-10-177

 实验时间: 2018-01-03, 14:58:04 诸图文件:E:\data\zwl\zwl-10-177-whelk-s, s-200-1-1-214-18.1.3.org 方法文件:E:\data\zwl\zwl.mtd 安验内容简介: zwl-10-177-whelk-s, s-200-1-1-214 报告时间: 2018-01-03, 15:56:13 Chromatogram Figure 色谱图处w1-10-177-whe1k-s, s-200-1-1-214-18, 1, 3, org) 1,000 950 900 850 650 650 650 550 550 550 550 300 250 250 250 250 250 250 0 0 0 0 HO -n-Bu -Ph (±)-1a -16.995 Voltage (mV) ∰ ₽ >13.050

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 时间 (min) Time (min)

峰号(Peak No.)	峰名(Peak name)	保留时间(Retent	tion time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1		13.050	298480.719	13337632.000	50.2526
2		16.995	370224.250	13203545.000	49.7474
总计			668704.969	26541177.000	100.0000





报告时间: 2018-04-12, 18:02:52

实验时间: 2018-04-12,17:48:02 谱图文件:E:\data\zwl\zwl-11-65-whelk-s,s-200-1-1-214-18.4.12.org

实验内容简介。 zw1-11-65-whe1k-s, s-200-1-1-214 Brief introduction of experimental conditions



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention til	me) 峰高 (Peak height)	峰面积 (Peak area) 含量 (Content
1		7.287	9732.876	158619.156	1.5224
2		8.635	412723.813	10260651.000	98.4776
总计			422456.688	10419270.156	100.0000

zw1-9-139

报告时间: 2018-04-12, 19:03:18

实验时间: 2018-04-12,18:50:54 谱图文件:E:\data\zwl\zwl-9-139-whelk-s,s-200-1-1-214-18.4.12.org

实验内容简介: zw1-9-139-whe1k-s, s-200-1-1-214 Brief introduction of experimental conditions



峰号(Peak No.)	峰名(Peak name) 保留时间(Retent	tion time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1	7.688	1155792.375	15057648.000	49.6680
2	8.850	841111.750	15258977.000	50.3320
总计		1996904.125	30316625.000	100.0000





実验时间: 2018-04-16, 21:55:07 谱图文件:E:\data\zwl\zwl-11-71-whelk-s, s-100-1-1-214-18, 4, 16-1. org 方法文件:E:\data\yaoyuan\0414. mtd ■ Brief introduction of experimental conditions 报告时间: 2018-04-17, 18:08:59 实验内容简介: zw1-11-71-whe1k-s, s-100-1-1-214 Chromatogram Figure 色谱图/zw1-11-71-whelk-s, s-100-1-1-214-18. 4. 16-1. org) 2,000 1,900 1,800 1,700 1,600 1,500 1,400 1,300 1,200 1,100 ♥ 1,000 ♥ 800 700 €00 500 500 100 100 2,000 COOMe -10.632 Ph (R_a)-2c CI 9.448 0 9 10 11 12 13 14 15 16 17 18 19 20 时间(min) <mark>Time (min)</mark> 0 2 3 4 5 6 8 1 7 分析结果表 ----.... ----

峰号(Peak No.)	峰名(Peak name) 保留时间(Reten	tion time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1	9.448	30770.469	534250, 500	2.0541
2	10.632	1185332.625	25474468.000	97.9459
总计		1216103.094	26008718.500	100.0000

报告时间: 2018-04-17, 18:08:23 Chromatogram Figure 色谱图 (zwl-8-145-whelk-s, s-100-1-1-214-18, 4, 16, org) 2,000 COOMe Ph - 9.413 (±)-2c ζI 10.677 0 9 10 11 12 13 14 15 16 17 18 19 20 时间(min) Time(min) 0 2 3 4 5 6 8 1 7 分析结果表 修西和 (Besterne) 今日 (Conten ... ----....

峰号(Peak No.)	峰名(Peak name) 保留时间(Retent	tion time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1	9.413	1151041.125	20616230.000	49.6995
2	10.677	985227.375	20865558.000	50.3005
总计		2136268.500	41481788.000	100.0000

zw1-8-145





实验时间: 2018-04-17, 17:03:47 语图文件:E:\data\zwl\zwl-11-73-whelk-s, s, -200-1-1-214-18.4. 17-1. org 方法文件:E:\data\yaoyuan\0414. mtd Brief introduction of experimental conditions

实验内容简介: zwl-11-73-whelk-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	time) 峰高(Peak height)	峰面积(Peak area) 含量 (Content)
1		7.920	13356.616	191971.297	1.7227
2		8.693	660292.250	10951710.000	98.2773
总计			673648.866	11143681.297	100.0000

zw1-8-200

报告时间: 2018-04-17, 16:26:10

实验时间: 2018-04-17,16:10:49 谱图文件:E:\data\zwl\zwl-8-200-whelk-s,s,-200-1-1-214-18.4.17.org 方法文件:E:\data\yaoyuan\0414.mtd

安验内容简介 zw1-8-200-whelk-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name) 保留时间(Reter	tion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1	8.132	36212.492	533018,250	49.8637
2	8.978	33533. 133	535932.188	50.1363
总计		69745.625	1068950.438	100.0000





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Project Name: defaults for copy Reported by User: Breeze user (Breeze)

	SAMPLE	INFORMATIO	NC
Sample Name:	zwl-11-69-pc-2-100-1-1-214	Acquired By:	Breeze
Sample Type:	末知	Date Acquirect	2018/4/17 19:43:56 CST
Vial:	999	Acq Method	zgj1001
Injection#.	71	Date Processed	2018/4/17 20:45:04 CST
Injection Volume	10.00 ul	Channel Name	W2489 ChA
Run Time	15.00 Minutes	Channel Desc.:	W2489 ChA 230nm
Column Type:		Sample Set Name	



Report Method: Individual Report ASC Page: 1 (共计 1) Printed 2018/4/17 20:45:27 PRC

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Project Name: defaults for copy Reported by User: Breeze user (Breeze)

		SAM	PLE	INFORMATIO	NC	
Sample Name: Sample Type: Vial: Injection #. Injection Volume: Run Time: Column Type:	zwl-7-135pc 求知 999 69 10.00 ul 15.00 Mnute	2-100-1-1-214 s		Acquired By: Date Acquirect Acq. Method Date Processed Channel Name Channel Desc.: Sample Set Name	Breeze 2018/4/17 18:59:03 CST 2018/4/17 20:44:39 CST W2489 ChA W2489 ChA 220nm	
190 180 170 160 150 0.40	Ph P (±)-2j	OOMe h		209.1	000	
0.20						
0.10 0.00 0.00	2.00	4.00	6.00	人 800 分中	10.00 12.00	14.00
0.10	2.00 RT Area (min) (@\$sec)	4.00 %Area Hsight	6.00 % Height	800 分钟	10.00 12.00	14.00
0.10	200 RT Area (min) (@\$sec) 1 7.532 15876841	4.00 %Area Height (%) 49.82 887215	6.00 % Height 59.22	。 《 》 》 後 申	10.00 12.00	14.00

Report Method: Individual Report ASC Page: 1 (共计 1) Printed 2018/4/17 20:45:15 PRC





报告时间: 2018-04-17, 18:11:30 <u>实验内容简介</u>: zwl-11-70-whelk-s, s-200-1-1-214 Chromatogram Figure 色谱图/zwl-11-70-whe1k-s, s-200-1-1-214-18, 4, 16-2, org) 2,000 1,900 1,800 1,700 1,600 1,500 1,400 1,300 1,200 1,100 1,200 1,100 1,200 1,000 1,000 1,200 0,00 1,000 0,000 0, 2,000 COOMe n-C5H11 Ph (R_a)-21 >12.285 +11.288 0 9 10 11 12 13 14 15 16 17 18 19 20 时间(min) Time(min) ō 2 3 4 5 6 8 1 分析结果表 修而积(Peak area) 含量 (Content) 14 17 10 1 AT IST IL A -.....

唯号(Peak No.)	唵名(Peak name) 保留时间(Retent	峰面积(Peak area) 含重 (Conten		
1	11.288	23293. 891	422110.094	4.2103
2	12.285	438309.594	9603543.000	95. 7897
总计		461603.484	10025653.094	100.0000

zw1-8-147

实验时间: 2018-04-16, 19:28:42 谱图文件:E:\data\zwl\zwl-8-147-whelk-s, s-200-1-1-214-18.4.16-1.org 方法文件:E:\data\yaoyuan\0414.mtd Brief introduction of experimental conditions 报告时间: 2018-04-20, 15:20:43 <u>实验内容简介</u> zw1-8-147-whe1k-s, s-200-1-1-214 Chromatogram Figure 色谱图(zw1-8-147-whe1k-s, s-200-1-1-214-18, 4, 16-1, org) 2,000 1,900 1,800 1,700 1,600 1,500 1,500 1,400 1,300 1,200 1,100 1,200 1,100 1,200 1,100 1,000 1,000 1,000 1,000 1,000 1,000 1,000 1,600 1,000 1,600 1,000 1,600 1,0000 1,000 1,000 1,000 1,0000 1,000 1,000 1,0000 1, COOMe n-C5H11 Ph (±)-**2l** 11.420 12.507 7 8 9 10 时间(min) Time (min) 13 1 2 3 4 5 11 12 14 15 6 分析结果表

峰号(Peak No.)	峰名(Peak name) 保留时间(Retenti	峰面积 (Peak area) 含量 (Content		
1	11.420	101879.672	2118019.500	50.0813
2	12. 507	91350.055	2111141.750	49.9187
总计		193229.727	4229161.250	100.0000


报告时间: 2018-01-04, 13:08:31

实验时间: 2018-01-03, 11:09:13 谱图文件:E:\data\zwl\zwl-11-22-whelk-s, s-200-1-1-214.org 方法文件:E:\data\zwl\zwl.mtd

 Brief introduction of experimental conditions

 实验内容简介:

 zwl-11-22-whelk-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time) 峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1		12.073	403891.813	11379891.000	95. 2917
2		14.383	12924.046	562267.938	4.7083
总计			416815.858	11942158.938	100.0000

实验时间: 2018-01-03,9:32:37 谱图文件:E:\data\zwl\zwl-9-139-whelk-s,s-200-1-1-214-18.1.3.org 方法文件:E:\data\zwl\zwl.mtd 报告时间: 2018-01-03,9:54:10 实验内容简介: zwl-9-139-whelk-s, s-200-1-1-214 Chromatogram Figure 色谱图 zw1-9-139-whe1k-s, s-200-1-1-214-18, 1, 3, org) 1,500 1,400 1,300 COOMe 1,200 1,100 Ph n-Bu 1,000 (±)-2a 900 Voltage (mV) 当日 800 11.742 13.852 700 Schme 2a

2 3 4 5 6

1

分析结果表 峰号(Peak No.) 峰名(Peak name) 保留时间(Retention time) 峰高(Peak height) 峰面积(Peak area) 含量 (Content) 619896.125 50.0764 11.742 14716571.000 1 2 13.852 599002.500 14671686.00049.9236 总计 1218898.625 29388257.000 100.0000

8

9 10 11 12 13 14 15 16 17 18 19 20 时间(min) Time (min)

zw1-9-139



报告时间: 2018-04-11, 19:28:52

实验时间: 2018-04-11, 16:46:54 谱图文件:E:\data\zwl\zwl-11-67-whelk-s, s-200-1-1-214-18.4.11. org

Brief introduction of experimental conditions

实验内容简介: zw1-11-67-whe1k-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention ti	ime) 峰高(Peak height)	峰面积(Peak area)	含量(Content)
1		7.598	741111.063	9224980.000	95. 3200
2		8.832	16986. 834	452923.063	4.6800
总计			758097.896	9677903.063	100.0000

zw1-9-139

实验时间: 2018-04-11,16:15:33 谱图文件:E:\data\zwl\zwl-9-139-whelk-s,s-200-1-1-214-18.4.11.org 报告时间: 2018-04-11, 16:32:17

Brief introduction of experimental conditions ¢ 实验内容简介: zw1-9-139-whe1k-s, s-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retent	ion time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1		7.770	502055. 188	7019085.000	49.5363
2		8.982	426594.656	7150499.500	50.4637
总计			928649.844	14169584.500	100.0000



实验时间: 2018-01-29, 10:37:25
 报告时间: 2018-01-29, 13:27:28
 诸图文件:E:\data\zwl\zwl-11-42-od-h-200-1-1-214-18. 1.29. org
 安验内容简介:
 zwl-11-42-od-h-200-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高(Peak height)	峰面积(Peak area)	含量(Content)
1		22. 583	1442.024	47599.824	0.4186
2		25. 327 3	326420.031	11324125.000	99. 5814
总计			327862.055	11371724.824	100.0000

zw1-10-177

生態时间: 2018-01-29,9:09:05 谱图文件:E:\data\zwl\zwl-10-177-od-h-200-1-1-214-18.1.29.org
Brief introduction of experimental conditions
実验内容简介: zwl-10-177-od-h-200-1-1-214
Corporations



峰号(Peak No.)	峰名(Peak name) 保留时间(Retention time)) 峰高(Peak height)	峰面积(Peak area)	含量 (Content)
1	22.802	477322.750	15513478.000	49.9342
2	25.715	416485.000	15554379.000	50.0658
总计		893807.750	31067857.000	100.0000





Parameter	Value (f2, f1)
Title	zwl-10-37-NOE
Origin	Varian
Solvent	odcl3
Temperature	26.0
Pulse Sequence	NOESY
Experiment	2D-NOESY
Number of Scans	16
Receiver Gain	38
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.1499
Acquisition Date	2017-08-23T16:41:01
Modification Date	2017-08-23T20:05:06
Spectrometer Frequency	(399.75, 399.75)
Spectral Width	(3255.2, 3255.2)
Lowest Frequency	(41.2, 41.2)
Nucleus	(IH, IH)
Acquired Size	(488, 200)
Spectral Size	(512, 512)



zw1-10-37-as-h-98-2-1-214

实验时间: 2017-07-06, 17:08:56 谱图文件:D:\zhuguangjiong\zwl\20170706\zwl-10-37-as-h-98-2-1-214. org



峰号(Peak No.)	峰名(Peak name)	保留时间(Reten	tion time) 峰高(Peak height)	峰面积(Peak	area) 含量 (Content)
1		10.717	9781.789	113525.742	2.6545
2		11.288	331645.469	4163275.250	97.3455
总计			341427.258	4276800.992	100.0000

zw1-10-19-rac-as-h-98-2-1-214

实验时间: 2017-07-06, 16:30:02 报告时间: 2017-07-06, 17:31:30 谱图文件:D:\zhuguangjiong\zwl\20170706\zwl-10-19-rac-as-h-98-2-1-214. org



峰号(Peak No.)	峰名(Peak name) 保	留时间(Retentio	n time) 峰高(Peak height)	峰面积(Peak area	a) 含量 (Content)
1		10.648	513380.281	6305159.500	49.4812
2		11.198	499607.563	6437378.000	50. 5188
总计			1012987.844	12742537.500	100.0000





实验时间: 2018-01-16, 20:51:53 谱图文件:E:\data\zwl\zwl-11-38-ic-400-1-1-214-18. 1. 16. org 方法文件:E:\data\zwl\zwl.mtd Brief introduction of experimental conditions 实验内容简介: zwl-11-38-ic-400-1-1-214 报告时间: 2018-01-16, 21:52:06



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention time)	峰高(Peak height)	峰面积(Peak area)	含量(Content)
1		21.822 8	595813.625	21841568.368	97.8551
2		25.390	9618.172	478748.476	2.1449
总计			605431.797	22320316.844	100.0000

zw1-10-30

实验时间: 2018-01-16,21:47:18 谱图文件:E:\data\zwl\zwl-10-30-ic-400-1-1-214-18.1.16.org 方法文件:E:\data\zwl\zwl.mtd 报告时间: 2018-01-16,22:16:32

实验内容简介: zw1-10-30-ic-400-1-1-214



峰号(Peak No.)	峰名(Peak name)	保留时间(Retention	time) 峰高(Peak height)	峰面积(Peak a	area) 含量 (Content)
1		21.392	275902.938	8311843. 500	50.0182
2		24.982	230701.609	8305795.000	49.9818
总计			506604.547	16617638.500	100.0000



S163





zw1-10-18-ic-90-10-1-214

实验时间: 2017-06-23, 11:03:40 报告时间: 2017-06-23, 14:33:20 谱图文件:D:\zhuguangjiong\zwl\20170622\zwl-10-18-ic-90-10-1-214...org



峰号(Peak No.)	峰名(Peak name)	保留时间(Retent	ion time) 峰高(Peak height)	峰面积(Peak	area) 含量(Content)
1		10.437	387483.156	6427971.500	97.2336
2		11.407	8051.422	182880.094	2.7664
总计			395534.579	6610851.594	100.0000

zw1-10-11-ic-90-10-1-214

实验时间: 2017-06-23, 11:20:28 报告时间: 2017-06-23, 14:32:09 谱图文件:D:\zhuguangjiong\zwl\20170622\zwl-10-11-ic-90-10-1-214...org



峰号(Peak No.)	峰名(Peak name)保留时间(Retent	ion time) 峰高 (Peak height)	峰面积(Peak area) 含量 (Content		
1	10. 398	961972.500	17275850.000	50.1575	
2	11. 365	887794.125	17167346.000	49.8425	
总计		1849766.625	34443196.000	100.0000	







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Project Name: defaults for copy Reported by User: Breeze user (Breeze)



					S	SAMF	LE	INFORMAT	101	١			
	Sample Name: Sample Type: Val: Injection # Injection Volume: Run Time: Column Type:		zwl-10:50ic:95:51-214 秋年 999 191 10:00 ul 20:00 Minutes				Acquired By: Date Acquired Acq. Mathcat Date Processed Channel Name Channel Desc.: Sample Set Name		Breeze 2017/7/13 17: zgj95 2017/7/13 18:2 W2489 ChA W2489 ChA 2				
AU	2:00 1:80 1:60 1:40 1:20 1:00 0:80 0:60 0:40 0:20 0:00		Br	(S _a)-1	N-OMe H n-Bu Ph Sb			2 0011	12.485	0			
	0.00	2.00		4.00	6.00		8.00	10.00 12 分钟	200	14.00	16.00	18.00	20.00
		Π	RT (min)	Area (@tsec)	%Area	Height (101)	% Height]					

Report Method: Individual Report HP Page: 1 (共计 1)

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Printed 2017/7/13 18:22:57 PRC

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Project Name: defaults for copy Reported by User: Breeze user (Breeze)



		SAM	PLE	INFORMAT	ION			
Sample Name Sample Type Viat: Injection# Injection Volume Run Time Column Type	zwl-1049ic-95551-214 秋年 9999 189 10.00 ul 65.00 Minutes			Acquired By: Date Acquired Acq. Method Date Processed Channel Name Channel Desc.: Sample Set Name:		Breeze 2017/7/13 16:00:50 CST 2017/7/13 16:43:49 CST W2459 ChA W2459 ChA 214mm		
1.80 1.60 1.40 1.20 0.80 0.60 0.40 0.20 0.00	Br (±)	Ph 6b			15921	۵		
0.00 2.00	9 4.00	6.00	8.00	10.00 1 分中	200	14.00	16.00	18.00
	RT Area	%Area Heigh	t %					

	RI (min)	Area (硼tsec)	%Area	Height (砌)	% Height
1	11.79£	33290825	49.53	1707801	51.27
2	12651	33919826	50.47	1623272	48.73

Report Method: Individual Report HP Page: 1(共计 1) Printed 2017/7/13 18:22:19 PRC