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Ir-catalyzed asymmetric hydrogenation of 3-arylindenones for the synthesis of chiral 3-arylindanones

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ABSTRACT

An efficient synthesis of chiral 3-arylindanones via iridium-catalyzed asymmetric hydrogenation of 3-arylindenones has been developed. The reaction showed good compatibility with various functional groups, delivering a variety of 3-arylindanones in excellent yields and with good enantioselectivities. The reaction was also carried out on a gram-scale, delivering the product in quantitative yield. In addition, the products can be easily derivatized and transformed into natural products and pharmaceutical agents.

1. Introduction

In drug discovery, indanones are considered to be privileged structures as these are present in numerous pharmaceuticals [1]. Among them, optically active 3-arylindanones are a core structural motif of many bioactive molecules and natural products, and are also very useful building blocks with applications in the pharmaceutical industry [2]. For example, (+)-isopaucifloral F, (−)-ampelopsin D, (+)-pallidol and (+)-laetevirenol A are resveratrol-derived natural products [3]; (+)-Brazilane and (+)-haematoxylane are used in traditional Chinese medicine for the treatment of convulsion, menstrual disorders and straumatic disease [4]; and (+)-indatraline is a famous pharmaceutical used for the treatment of depression (Fig. 1) [5]. Although the chiral 3-arylindanone motif is important, the methods for obtaining such structures are limited [6], and the reported strategies predominantly focus on the transition-metal catalyzed enantioselective intramolecular hydroacylation of 2-vinyl benzaldehydes (Scheme 1a) [7] and reductive Heck cyclization reactions of enones (Scheme 1b) [8]. Given the importance of this molecular structure, we postulated that the asymmetric hydrogenation of 3-arylindenones using our developed axis-unfixed biphenylphosphine-oxazoline/iridium catalysts could provide efficient access to chiral indanones (Scheme 1c).

Transition-metal catalyzed asymmetric hydrogenation reaction is a popular method for obtaining chiral molecules, as it is operationally simple and requires only low catalyst loadings [9]. However, to the best of our knowledge, there are no reports concerning transition-metal catalyzed asymmetric hydrogenation reactions for the synthesis of chiral 3-arylindanones. Our group developed an axis-unfixed biphenylphosphine-oxazoline/iridium catalyst which showed excellent reactivity and enantioselectivity for the asymmetric hydrogenation of a few types of C=C bonds [10,11], especially for the hydrogenation of five-membered exo-α,β-unsaturated compounds [11]. The carbonyl group is used as a coordinating functional group in the reaction and delivers the corresponding products bearing an α-chiral carbon to the carbonyl group [11]. In contrast to our previous work, 3-arylindenones are endo-α,β-unsaturated compounds, and possess a substituent group at the β-position. For such structures, maybe the carbonyl group could not be used as the coordinating functional group due to the rigidity of the system. Additionally, the C=C bond in such structures is electron-deficient, so the compound may not readily bind with the catalyst. To date, no catalytic system for the asymmetric hydrogenation of 3-arylindenones has been reported. The synthesis of 3-arylindanones with excellent enantioselectivities and yields via transition-metal catalyzed asymmetric hydrogenation is

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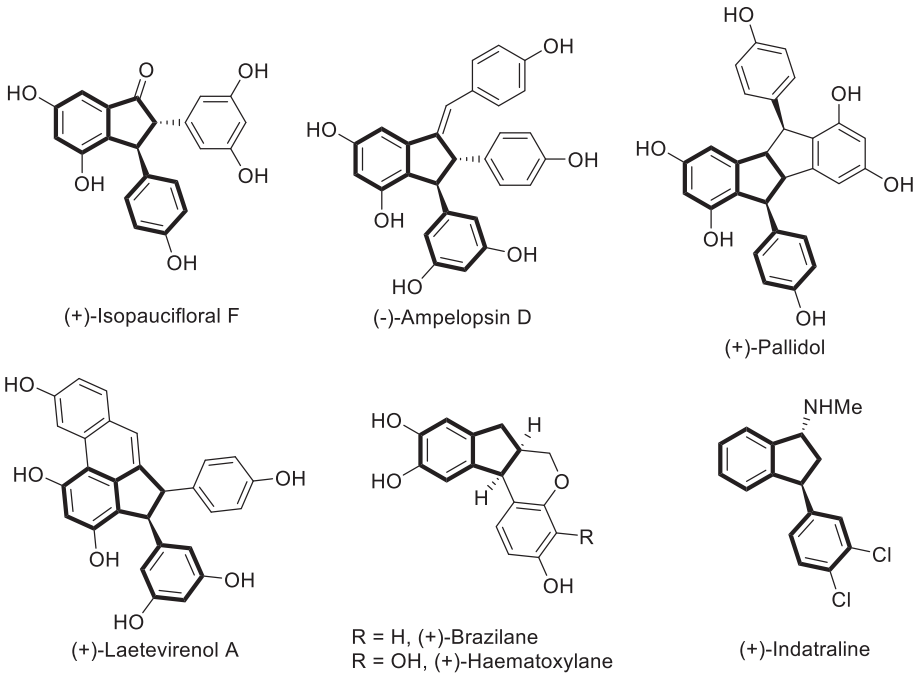
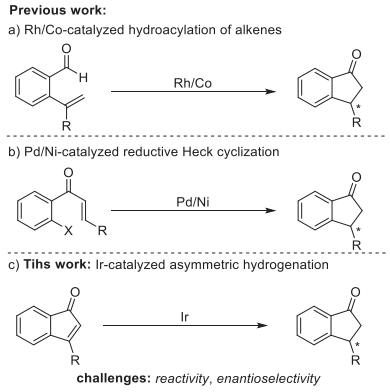


Fig. 1. Selected examples of natural products and pharmaceuticals bearing 3-arylindanones.



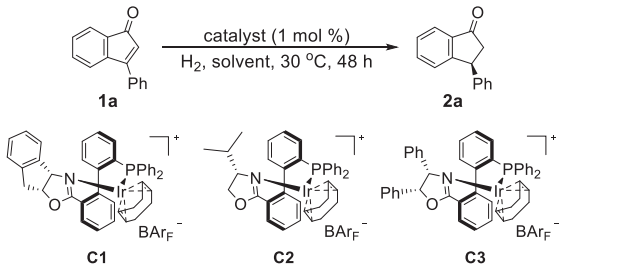
Scheme 1. Different methods for obtaining the chiral 3-aryl-indanones.

challenging.

2. Results and discussion

Based on our previous results [10,11], catalyst **C1** was selected and evaluated under asymmetric hydrogenation conditions for the reaction of 3-phenylindenone **1a** (Table 1). First, the solvent for the reaction was examined (entries 1–6). To our delight, the reaction proceeded very well when using toluene, dichloromethane, 1,2-dichloroethane or *o*-xylene as the reaction solvents, delivering the product **2a** in quantitative yields and with good enantioselectivities (entries 1–3 and 6). To improve the enantioselectivity of the product, various types of acids and bases were employed as additives and screened for the reaction, however better results were not obtained (entries 7–8). Next, the catalysts for the reaction were screened using *o*-xylene as the reaction solvent. Catalysts **C2** and **C3** provided the product **2a** with 78:22 and 93:7 er values, respectively (entries 9–10). 51% Conversion was obtained, when the hydrogen pressure was reduced to 30 bar (entry 11). The reaction solvent and temperature were also screened using **C3** as the catalyst, however no better results was obtained.

Table 1
Optimization of reaction conditions[a].

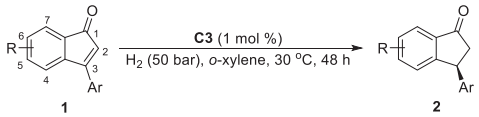


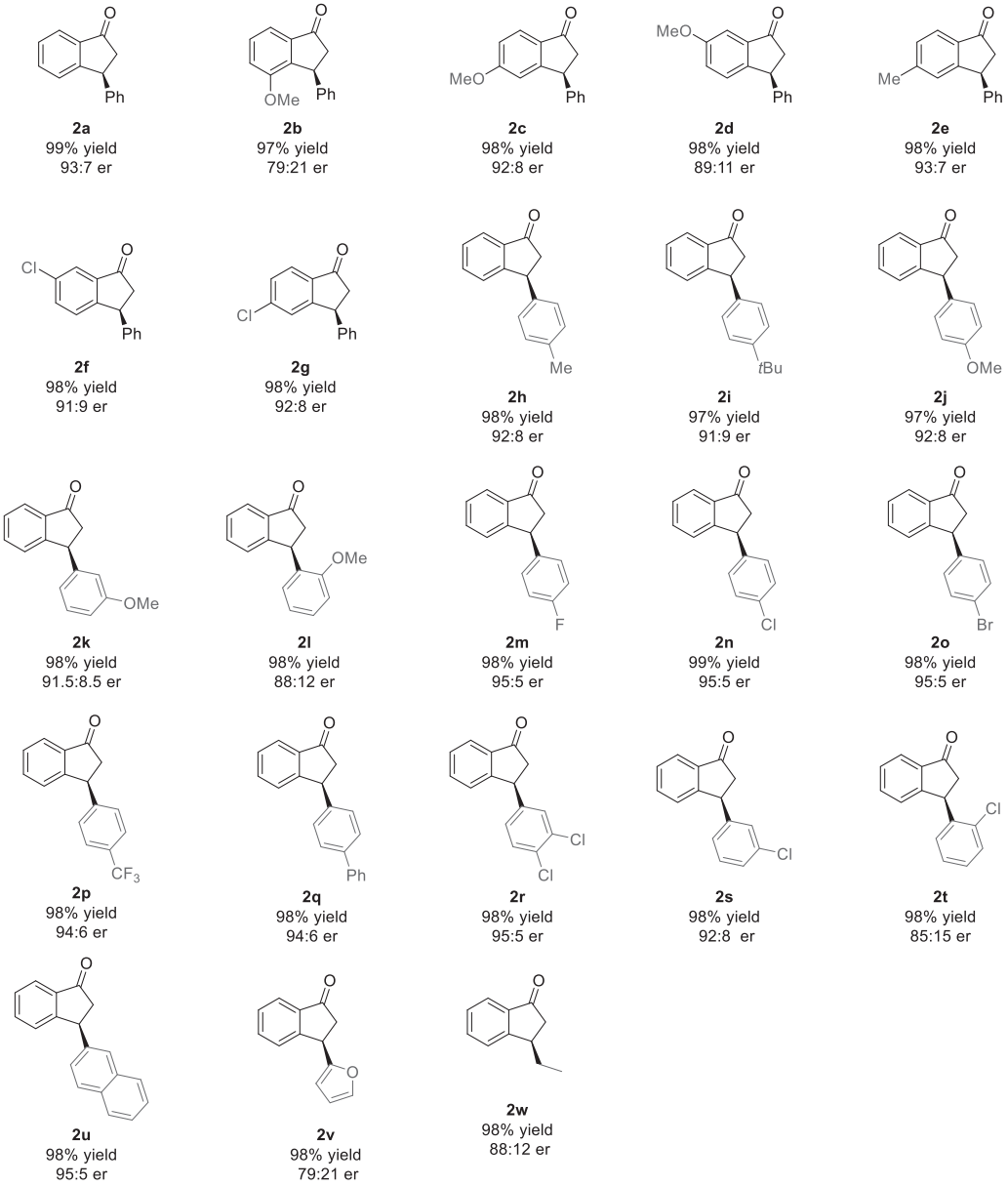
Entry	Catalyst	Solvent	Conv (%)[b]	Er[c]
1	**C1**	Toluene	99	90:10
2	**C1**	DCM	99	91:9
3	**C1**	DCE	99	88:12
4	**C1**	THF	50	86:14
5	**C1**	MeOH	14	91:9
6	**C1**	*o*-xylene	99	92:8
7[d]	**C1**	*o*-xylene	47	92:8
8[e]	**C1**	*o*-xylene	64	91:9
9	**C2**	*o*-xylene	99	78:22
10	**C3**	*o*-xylene	99	93:7
11[f]	**C3**	*o*-xylene	51	93:7

[a] Reaction conditions: 3-phenylindenone **1a** (0.2 mmol) and catalyst (1 mol %) were stirred under H_2 (50 bar) in solvent (2 mL) in an autoclave at 30 °C for 48 h.
[b] Determined by ^{1}H NMR of the crude product.
[c] Determined by HPLC analysis on chiral stationary phases.
[d] CH_3COONa was used as the additive.
[e] CH_3COOH was used as the additive.
[f] The pressure of hydrogen was 30 bar.

With the optimized reaction conditions in hand (Table 1, entry 10), we next examined the substrate scope of this asymmetric hydrogenation reaction (Table 2). First, 3-phenylindenones bearing different substituted groups on the indenone ring were tested. 3-

Table 2
Substrate scope[a,b].





[a]Reaction conditions: 3-arylindenone **2** (0.2 mmol) and catalyst **C3** (1 mol %) were stirred under H_2 (50 bar) in *o*-xylene (2 mL) in an autoclave at 30 °C for 48 h.

[b]Isolated yield; The er value was determined by HPLC analysis on chiral stationary phases.

Phenylindenones bearing an electron-withdrawing or electron-donating group at various positions of the indenones delivered the corresponding products with excellent yields and high enantioselectivities (**2b**-**2g**). Interestingly, methoxy substitution at the 4-position on the 3-phenylindenone gave the desired product **2b** in 97% yield, albeit with diminished enantioselectivity (79:21 er). It should be noted that the substrate bearing 5,6-dimethoxy groups on the 3-phenylindenone gave no product, and the starting material was recovered in quantitative yield. This may be ascribed to the two oxygens in the substrate coordinating to the catalyst, deactivating it. Next, the compatibility of 3-aryl substitutions for the synthesis of various 3-arylated indanones was examined. The transformation performs well with an electron-donating group at the *para*- or *meta*-position of the 3-phenyl ring, delivering the

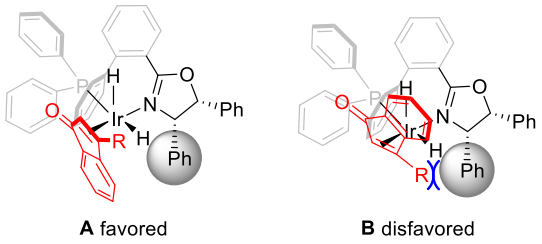


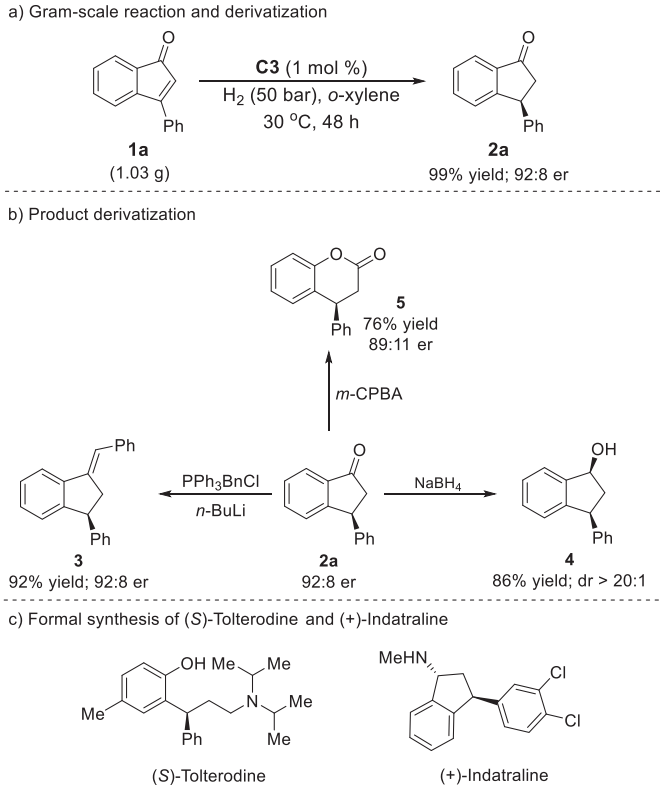
Fig. 2. Stereochemical model for the asymmetric hydrogenation of 3-arylindenones.

desired products in excellent yields and enantioselectivities (**2h**-**2k**). A substrate bearing a methoxy group at the *ortho*-position of 3-phenyl ring gave the product **2l** in 98% yield with a lower enantioselectivity (88:12 er). A variety of electron-withdrawing groups on the 3-phenyl ring were well tolerated under the reaction conditions, including fluoro, chloro, bromo, trifluoromethyl and phenyl groups, affording the corresponding products in excellent yields and good enantioselectivities (**2m**-**2s**). Similar to the results obtained for product **2l**, a substrate bearing a chloro substituent at the *ortho*-position of the 3-phenyl ring delivered the desired product **2t** in excellent yield but with a lower enantioselectivity (85:15 er), most likely due to the steric hindrance of the substrates. 2-Naphthyl substituted indenone afforded product **2u** in 98% yield and with 95:5 er. A heteroaryl ring 2-furyl substituted indenone delivered the desired product **2v** with moderate enantioselectivity. An alkyl-substituted indenone gave the product **2w** in 98% yield with 88:12 er. In addition, 2-methyl-3-phenylindenone was also synthesized, and tested under the reaction conditions, however, no reduced product was obtained. The absolute configuration of product **2a** was determined to be (*S*) by comparing the optical rotation value with previously reported data [8a]. The remainder of the products were assigned by analogy with product **2a**.

The observed stereochemical outcome of the reaction with the axis-unfixed biphenylphosphine-oxazoline/iridium catalyst can be rationalized based on favored and disfavored intermediates shown in Fig. 2 [11]. In the favored intermediate **A**, the R-group oriented upward, away from the phenyl group of the catalyst, thus leading to products with the *S*-configuration.

The asymmetric hydrogenation reaction can be performed on a gram-scale, delivering the corresponding product **2a** in quantitative yield with little erosion in enantioselectivity (Scheme 2a). The enantiomerically enriched 3-arylindanones can be derivatized into a variety of synthetically useful building blocks. For example, the carbonyl group in the product **2a** can be transformed into alkenyl and hydroxyl groups via a Wittig reaction and reduction reaction, delivering the corresponding product **3** and **4** in excellent yields, respectively. In addition, the product **2a** can also be transformed into lactone **5** in 76% yield with 89:11 er (Scheme 2b).

(*S*)-Tolterodine possesses spasmolytic activity against urinary disorders and intestinal spasms caused by various non-cholinergic mechanisms or where anti-muscarinc effects are not acceptable; its synthesis has attracted much attention [12,2b]. This new method provides an efficient formal synthesis of (*S*)-tolterodine, starting from the reduced product **2e** (Scheme 2c) [2b]. In addition, (+)-indatraline is a long-acting monoamine reuptake inhibitor and it has been subjected to several stereoselective synthesis [13]. The product **2r** was obtained in 95:5 er using our asymmetric hydrogenation conditions, which can be employed in the synthesis of (+)-indatraline via a reported method (Scheme 2c) [8c].



Scheme 2. Gram-scale reaction, product derivatization and formal synthesis of (*S*)-tolterodine and (+)-indatraline.

3. Conclusions

In summary, we have successfully developed an efficient method for the synthesis of enantiomerically enriched 3-arylindanones in excellent yields and good enantioselectivitites using an iridium-catalyzed asymmetric hydrogenation reaction employing our developed axis-unfixed biphenylphosphine-oxazoline ligand. The reaction shows excellent compatibility with various functional groups, and the reaction can be carried out on gram-scale with the product being easily derivatized. In addition, (*S*)-tolterodine and (+)-indatraline can be formally synthesized from the reduced products **2e** and **2r**, respectively.

4. Experimental sections

4.1. General experimental

All commercially available reagents were used as received. All asymmetric hydrogenation reactions were performed in autoclave under an atmosphere of hydrogen and the solvents were dried and distilled by standard procedures. ^{1}H, ^{13}C, and ^{19}F NMR spectra were obtained using Bruker Avance III HD 400 MHz NMR or Bruker Avance III HD 500 MHz NMR Spectrometer with TMS as an internal standard. Melting points were measured with SGW X-4 micro melting point apparatus. Enantioselectivities were determined by high performance liquid chromatography (HPLC) using Daicel CHIRALPAK OD-H and OJ-H columns with hexane/*i*PrOH as eluent. Optical rotations were measured on a Rudolph Research Analytical Autopol VI automatic polarimeter using a 50 mm path-length cell at 589 nm. High Resolution Mass Spectrometry (HRMS) analysis was carried out using an electrospray spectrometer Waters Micromass Q-TOF Premier Mass Spectrometer.

4.2. General procedure for the asymmetric hydrogenation of 3-arylindenones **1**

The catalyst **C3** (1 mol %), 3-arylindenone (**1**, 0.2 mmol) and anhydrous *o*-xylene (2 mL) were added to the hydrogenation tube and then the hydrogenation tube was charged in an autoclave. The autoclave was sealed and purged three times with hydrogen. The autoclave was charged with hydrogen to 50 bar, and the reaction mixture was stirred at 30 °C for 48 h. After releasing the hydrogen and removing the solvent, the mixture was purified by preparative TLC on silica gel to afford the pure product. The er values was determined by chiral HPLC.

4.3. Characterization data of products **2**

4.3.1. (S)-3-Phenyl-2,3-dihydro-1H-inden-1-one (**2a**) [8a]

Colorless solid (41.2 mg, 99% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85–7.78 (m, 1H), 7.55 (td, J = 7.5, 1.5 Hz, 1H), 7.40 (t, J = 7.5 Hz, 1H), 7.33–7.28 (m, 2H), 7.27–7.21 (m, 2H), 7.16–7.08 (m, 2H), 4.56 (dd, J = 8.0, 4.0 Hz, 1H), 3.22 (dd, J = 19.0, 8.0 Hz, 1H), 2.68 (dd, J = 19.5, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.0, 158.0, 143.7, 136.8, 135.1, 128.9, 127.9, 127.7, 127.0, 126.9, 123.4, 46.9, 44.5. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 11.9 min (minor), t_{R2} = 15.2 min (major)]; 93:7 er. [α]25D = +64.9 (c = 0.4, CH_2Cl_2).

4.3.2. (S)-4-Methoxy-3-phenyl-2,3-dihydro-1H-inden-1-one (**2b**) [8a]

Brown solid (46.2 mg, 97% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43–7.40 (m, 2H), 7.26–7.22 (m, 2H), 7.21–7.16 (m, 1H), 7.08–7.05 (m, 2H), 7.04 (dd, J = 6.0, 2.5 Hz, 1H), 4.65 (dd, J = 8.0, 2.5 Hz, 1H), 3.69 (s, 3H), 3.21 (dd, J = 19.5, 8.5 Hz, 1H), 2.63 (dd, J = 19.0, 2.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.5, 157.3, 145.7, 143.7, 138.5, 129.8, 128.4, 127.2, 126.4, 116.1, 115.1, 55.6, 47.3, 41.7. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 0.5 mL/min, 25 °C. t_{R1} = 15.4 min (major), t_{R2} = 19.6 min (minor)]; 79:21 er. [α]25D = −7.5 (c = 0.2, CH_2Cl_2).

4.3.3. (S)-5-Methoxy-3-phenyl-2,3-dihydro-1H-inden-1-one (**2c**) [8a]

White solid (46.7 mg, 98% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 8.4 Hz, 1H), 7.36–7.28 (m, 2H), 7.28–7.23 (m, 1H), 7.16–7.09 (m, 2H), 6.94 (dd, J = 8.4, 2.0 Hz, 1H), 6.66 (d, J = 1.6 Hz, 1H), 4.50 (dd, J = 8.0, 3.6 Hz, 1H), 3.79 (s, 3H), 3.21 (dd, J = 19.2, 8.0 Hz, 1H), 2.66 (dd, J = 18.8, 3.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 165.6, 160.9, 143.7, 130.2, 128.9, 127.7, 127.0, 125.1, 116.0, 109.8, 55.7, 47.1, 44.5. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 22.8 min (minor), t_{R2} = 24.7 min (major)]; 92:8 er. [α]25D = −10.0 (c = 0.2, CH_2Cl_2).

4.3.4. (S)-6-Methoxy-3-phenyl-2,3-dihydro-1H-inden-1-one (**2d**) [14]

White solid (46.7 mg, 98% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34–7.28 (m, 2H), 7.27–7.26 (m, 1H), 7.25–7.23 (m, 1H), 7.19–7.13 (m, 2H), 7.14–7.10 (m, 2H), 4.50 (dd, J = 8.0, 3.6 Hz, 1H), 3.79 (s, 3H), 3.21 (dd, J = 19.2, 8.0 Hz, 1H), 2.66 (dd, J = 18.8, 3.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0, 159.8, 150.9, 143.9, 138.0, 128.9, 127.6, 127.5, 126.9, 124.5, 104.4, 55.7, 47.6, 43.8. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 19.8 min (minor), t_{R2} = 24.8 min (major)]; 89:11 er. [α]25D = +43.4 (c = 0.2, CH_2Cl_2).

4.3.5. (S)-5-Methyl-3-phenyl-2,3-dihydro-1H-inden-1-one (**2e**) [14]

Yellow solid (43.6 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, J = 8.0 Hz, 1H), 7.32 (t, J = 7.5 Hz, 2H), 7.28–7.19 (m, 2H), 7.13 (d, J = 7.5 Hz, 2H), 7.05 (s, 1H), 4.51 (dd, J = 8.0, 4.0 Hz, 1H), 3.21 (dd, J = 19.0, 8.0 Hz, 1H), 2.68 (dd, J = 19.5, 4.0 Hz, 1H), 2.37 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.6, 158.5, 146.4, 143.8, 134.5, 129.2, 128.9, 127.6, 127.1, 126.9, 123.2, 47.0, 44.3, 22.1. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 0.5 mL/min, 25 °C. t_{R1} = 16.9 min (major), t_{R2} = 18.6 min (minor)]; 93:7 er. [α]25D = +20.3 (c = 0.1, CH_2Cl_2).

4.3.6. (S)-6-Chloro-3-phenyl-2,3-dihydro-1H-inden-1-one (**2f**) [14]

Colorless oil (47.6 mg, 98% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79–7.74 (m, 1H), 7.55–7.50 (m, 1H), 7.36–7.30 (m, 2H), 7.29–7.27 (m, 1H), 7.22 (d, J = 8.4 Hz, 1H), 7.14–7.08 (m, 2H), 4.55 (dd, J = 8.0, 4.0 Hz, 1H), 3.27 (dd, J = 19.2, 8.0 Hz, 1H), 2.73 (dd, J = 19.6, 4.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.6, 156.1, 143.2, 138.3, 135.2, 134.5, 129.1, 128.3, 127.7, 127.3, 123.3, 47.3, 44.2. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 0.5 mL/min, 25 °C. t_{R1} = 18.8 min (major), t_{R2} = 24.0 min (minor)]; 91:9 er. [α]25D = +55.6 (c = 0.2, CH_2Cl_2).

4.3.7. (S)-5-Chloro-3-phenyl-2,3-dihydro-1H-inden-1-one (**2g**) [14]

White solid (47.6 mg, 98% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75–7.70 (m, 1H), 7.40–7.36 (m, 1H), 7.36–7.30 (m, 2H), 7.29–7.26 (m, 1H), 7.24 (s, 1H), 7.13–7.08 (m, 2H), 4.53 (dd, J = 8.0, 3.6 Hz, 1H), 3.23 (dd, J = 19.6, 8.4 Hz, 1H), 2.71 (dd, J = 19.2, 4.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.5, 159.5, 142.9, 141.7, 135.2, 129.2, 128.9, 127.7, 127.4, 127.1, 124.7, 46.9, 44.3. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 11.8 min (minor), t_{R2} = 14.7 min (major)]; 92:8 er. [α]25D = −12.8 (c = 0.1, CH_2Cl_2).

4.3.8. (S)-3-(p-Tolyl)-2,3-dihydro-1H-inden-1-one (**2h**) [14]

Yellow oil (43.6 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, J = 8.0 Hz, 1H), 7.56 (t, J = 7.0 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 7.29–7.23 (m, 1H), 7.15–7.08 (m, 2H), 7.04–7.00 (m, 2H), 4.54 (dd, J = 8.0, 3.5 Hz, 1H), 3.22 (dd, J = 19.0, 8.0 Hz, 1H), 2.67 (dd, J = 19.0, 3.5 Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.2, 158.1, 140.6, 136.6, 136.5, 135.0, 129.5, 127.7, 127.4, 126.8, 123.3, 46.9, 44.0, 21.0. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 10.7 min (minor), t_{R2} = 17.3 min (major)]; 92:8 er. [α]25D = +109.0 (c = 0.2, CH_2Cl_2).

4.3.9. (S)-3-(tert-Butyl)phenyl)-2,3-dihydro-1H-inden-1-one (**2i**) [15]

Yellow solid (51.3 mg, 97% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, J = 7.5 Hz, 1H), 7.57 (t, J = 7.5 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 7.34–7.29 (m, 3H), 7.06 (d, J = 8.5 Hz, 2H), 4.56 (dd, J = 8.0, 3.5 Hz, 1H), 3.22 (dd, J = 19.0, 8.0 Hz, 1H), 2.70 (dd, J = 19.0, 4.0 Hz, 1H), 1.31 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.2, 158.1, 149.8, 140.5, 136.7, 135.0, 127.7, 127.2, 126.9, 125.7, 123.3, 46.8, 43.9, 34.4, 31.3. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 6.9 min (minor), t_{R2} = 8.8 min (major)]; 91:9 er. [α]25D = +35.6 (c = 0.6, CH_2Cl_2).

4.3.10. (S)-3-(4-Methoxyphenyl)-2,3-dihydro-1H-inden-1-one (**2j**) [8a]

Yellow solid (46.2 mg, 97% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, J = 7.5 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 7.27 (d, J = 6.0 Hz, 1H), 7.04 (d, J = 8.5 Hz, 2H), 6.85 (d, J = 9.0 Hz, 2H), 4.53 (dd, J = 8.0, 4.0 Hz, 1H), 3.79 (s, 3H), 3.21 (dd, J = 19.0, 8.0 Hz, 1H), 2.65 (dd, J = 19.5, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.4, 158.8, 158.5, 136.9, 136.0, 135.3, 128.8, 128.0, 127.0, 123.5, 114.5, 55.5, 47.2, 43.9. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 21.8 min (minor),

t_{R2} = 34.2 min (major)]; 92:8 er. [α]25D = +59.1 (c = 0.6, CH_2Cl_2).

*4.3.11. (S)-3-(3-Methoxyphenyl)-2,3-dihydro-1H-inden-1-one (**2k**) [15]*

Yellow oil (46.7 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, J = 8.0 Hz, 1H), 7.57 (t, J = 7.5 Hz, 1H), 7.42 (t, J = 7.5 Hz, 1H), 7.29 (d, J = 7.5 Hz, 1H), 7.23 (t, J = 7.5 Hz, 1H), 6.79 (dd, J = 8.0, 2.5 Hz, 1H), 6.72 (d, J = 8.0 Hz, 1H), 6.66 (s, 1H), 4.55 (dd, J = 8.0, 4.0 Hz, 1H), 3.76 (s, 3H), 3.22 (dd, J = 19.5, 8.0 Hz, 1H), 2.70 (dd, J = 19.5, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.2, 160.2, 158.0, 145.5, 137.0, 135.3, 130.2, 128.1, 127.1, 123.6, 120.2, 113.9, 112.2, 55.4, 46.9, 44.7. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 19.5 min (minor), t_{R2} = 24.6 min (major)]; 91.5:8.5 er. [α]25D = +51.6 (c = 0.5, CH_2Cl_2).

*4.3.12. (R)-3-(2-Methoxyphenyl)-2,3-dihydro-1H-inden-1-one (**2l**) [15]*

Brown oil (46.7 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, J = 7.5 Hz, 1H), 7.55 (t, J = 7.5 Hz, 1H), 7.43–7.34 (m, 1H), 7.29 (d, J = 7.5 Hz, 1H), 7.25–7.20 (m, 1H), 6.97–6.93 (m, 1H), 6.90–6.84 (m, 2H), 4.94–4.81 (m, 1H), 3.74 (s, 3H), 3.17 (dd, J = 19.0, 8.0 Hz, 1H), 2.69 (dd, J = 19.0, 3.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.7, 157.8, 157.2, 137.0, 134.6, 131.5, 128.3, 128.0, 127.4, 126.5, 123.2, 120.6, 110.8, 55.3, 45.1, 39.0. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 13.9 min (major), t_{R2} = 16.9 min (minor)]; 88:12 er. [α]25D = +29.1 (c = 0.5, CH_2Cl_2).

*4.3.13. (S)-3-(4-Fluorophenyl)-2,3-dihydro-1H-inden-1-one (**2m**) [8a]*

White solid (44.3 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, J = 8.0 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 7.43 (t, J = 7.5 Hz, 1H), 7.30–7.20 (m, 1H), 7.12–7.04 (m, 2H), 7.04–6.94 (m, 2H), 4.58 (dd, J = 8.0, 4.0 Hz, 1H), 3.23 (dd, J = 19.5, 8.0 Hz, 1H), 2.64 (dd, J = 19.5, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.9, 162.0, (d, J = 244.1 Hz), 157.9, 139.7 (d, J = 2.9 Hz), 136.9, 135.4, 129.3 (d, J = 7.9 Hz), 128.3, 127.0, 123.7, 116.0 (d, J = 21.2 Hz), 47.1, 43.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ −115.7. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 14.3 min (minor), t_{R2} = 16.8 min (major)]; 95:5 er. [α]25D = +37.9 (c = 0.3, CH_2Cl_2).

*4.3.14. (S)-3-(4-Chlorophenyl)-2,3-dihydro-1H-inden-1-one (**2n**) [8a]*

White solid (48.1 mg, 99% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 1H), 7.61–7.54 (m, 1H), 7.44 (t, J = 7.5 Hz, 1H), 7.31–7.21 (m, 3H), 7.06 (d, J = 8.4 Hz, 2H), 4.56 (dd, J = 8.0, 3.5 Hz, 1H), 3.23 (dd, J = 19.5, 8.5 Hz, 1H), 2.63 (dd, J = 19.0, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.1, 157.9, 142.8, 137.3, 135.8, 133.4, 129.6, 129.5, 128.7, 127.3, 124.1, 47.3, 44.4. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 16.4 min (minor), t_{R2} = 18.0 min (major)]; 95:5 er. [α]25D = +48.5 (c = 0.4, CH_2Cl_2).

*4.3.15. (S)-3-(4-Bromophenyl)-2,3-dihydro-1H-inden-1-one (**2o**) [16]*

White solid (56.3 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, J = 7.5 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 7.50–7.37 (m, 3H), 7.31–7.15 (m, 1H), 7.00 (d, J = 8.0 Hz, 2H), 4.55 (dd, J = 8.0, 4.0 Hz, 1H), 3.23 (dd, J = 19.5, 8.0 Hz, 1H), 2.63 (dd, J = 19.5, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.4, 157.2, 142.7, 136.7, 135.2, 132.0, 129.3, 128.1, 126.7, 123.5, 120.8, 46.6, 43.8. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 18.7 min (minor), t_{R2} = 20.6 min (major)]; 95:5 er. [α]25D = +44.0 (c = 0.4, CH_2Cl_2).

*4.3.16. (S)-3-(4-(Trifluoromethyl)phenyl)-2,3-dihydro-1H-inden-1-one (**2p**) [16]*

White solid (54.1 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, J = 7.5 Hz, 1H), 7.63–7.52 (m, 3H), 7.50–7.41 (m, 1H), 7.29–7.21 (m, 3H), 4.66 (dd, J = 8.0, 4.0 Hz, 1H), 3.27 (dd, J = 19.0, 8.0 Hz, 1H), 2.67 (dd, J = 19.0, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.1, 156.8, 147.7, 136.7, 135.2, 129.3 (q, J = 32.5 Hz), 128.2, 128.0, 126.7, 125.8 (q, J = 3.8 Hz), 124.0 (q, J = 272.5 Hz), 123.6, 46.5, 44.1. ^{19}F NMR (471 MHz, $CDCl_3$) δ - 62.5. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 0.5 mL/min, 25 °C. t_{R1} = 16.8 min (major), t_{R2} = 20.8 min (minor)]; 94:6 er. [α]25D = +32.0 (c = 0.6, CH_2Cl_2).

*4.3.17. (S)-3-([1,1′-Biphenyl]-4-yl)-2,3-dihydro-1H-inden-1-one (**2q**) [15]*

White solid (55.7 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, J = 7.5 Hz, 1H), 7.61–7.48 (m, 5H), 7.46–7.38 (m, 3H), 7.36–7.28 (m, 2H), 7.18 (d, J = 8.0 Hz, 2H), 4.61 (dd, J = 8.0, 4.0 Hz, 1H), 3.25 (dd, J = 19.5, 8.5 Hz, 1H), 2.72 (dd, J = 19.5, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.2, 158.1, 142.9, 140.8, 140.2, 137.0, 135.4, 129.0, 128.3, 128.2, 127.9, 127.6, 127.2, 127.1, 123.7, 47.1, 44.4. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 20.6 min (minor), t_{R2} = 24.5 min (major)]; 94:6 er. [α]25D = +56.2 (c = 0.3, CH_2Cl_2).

*4.3.18. (S)-3-(3,4-Dichlorophenyl)-2,3-dihydro-1H-inden-1-one (**2r**) [16]*

Yellow solid (54.3 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, J = 7.5 Hz, 1H), 7.61 (t, J = 7.5 Hz, 1H), 7.46 (t, J = 7.0 Hz, 1H), 7.38 (d, J = 8.0 Hz, 1H), 7.30–7.20 (m, 2H), 6.95 (dd, J = 8.5, 2.0 Hz, 1H), 4.55 (dd, J = 8.0, 4.0 Hz, 1H), 3.23 (dd, J = 19.5, 8.5 Hz, 1H), 2.62 (dd, J = 19.0, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.2, 156.8, 144.2, 137.0, 135.6, 133.2, 131.3, 131.1, 129.9, 128.6, 127.2, 126.9, 123.9, 46.7, 43.8. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 16.5 min (major), t_{R2} = 18.1 min (minor)]; 95:5 er. [α]25D = +38.2 (c = 0.5, CH_2Cl_2).

*4.3.19. (S)-3-(3-Chlorophenyl)-2,3-dihydro-1H-inden-1-one (**2s**) [17]*

White solid (47.6 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 7.44 (t, J = 7.5 Hz, 1H), 7.31–7.20 (m, 3H), 7.12 (s, 1H), 7.04–6.97 (m, 1H), 4.56 (dd, J = 8.0, 4.0 Hz, 1H), 3.23 (dd, J = 19.5, 8.0 Hz, 1H), 2.66 (dd, J = 19.0, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.3, 156.9, 145.7, 136.7, 135.2, 134.6, 130.1, 128.1, 127.7, 127.2, 126.7, 125.8, 123.5, 46.5, 44.0. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 0.5 mL/min, 25 °C. t_{R1} = 18.4 min (major), t_{R2} = 23.0 min (minor)]; 92:8 er. [α]25D = +41.4 (c = 0.6, CH_2Cl_2).

*4.3.20. (R)-3-(2-Chlorophenyl)-2,3-dihydro-1H-inden-1-one (**2t**) [17]*

Yellow solid (47.6 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, J = 7.5 Hz, 1H), 7.61 (t, J = 8.5 Hz, 1H), 7.48–7.40 (m, 2H), 7.38–7.31 (m, 1H), 7.23–7.11 (m, 2H), 6.87 (s, 1H), 5.12 (s, 1H), 3.31 (dd, J = 19.5, 8.5 Hz, 1H), 2.61 (d, J = 18.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.5, 156.5, 141.1, 137.2, 135.0, 134.0, 129.8, 128.4, 128.2, 128.0, 127.3, 126.8, 123.6, 45.3, 29.7. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 9.3min (major), t_{R2} = 10.7 min (minor)]; 85:15 er. [α]25D = −36.0 (c = 0.4, CH_2Cl_2).

*4.3.21. (S)-3-(Naphthalen-2-yl)-2,3-dihydro-1H-inden-1-one (**2u**) [8a]*

Yellow solid (54.3 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, J = 7.5 Hz, 1H), 7.82–7.74 (m, 3H), 7.66 (s, 1H), 7.56 (t,

J = 7.5 Hz, 1H), 7.51–7.39 (m, 3H), 7.31–7.24 (m, 1H), 7.13 (dd, J = 8.5, 1.5 Hz, 1H), 4.74 (dd, J = 8.0, 3.5 Hz, 1H), 3.29 (dd, J = 19.0, 8.0 Hz, 1H), 2.78 (dd, J = 19.0, 3.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.2, 158.1, 141.1, 137.1, 135.4, 133.7, 132.7, 129.2, 128.2, 127.9, 127.8, 127.2, 126.65, 126.63, 126.1, 125.7, 123.7, 46.9, 44.8. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 32.4 min (minor), t_{R2} = 45.2 min (major)]; 95:5 er. [α]25D = +95.3 (c = 0.3, CH_2Cl_2).

*4.3.22. (S)-3-(furan-2-yl)-2,3-dihydro-1H-inden-1-one (**2v**) [18]*

Yellow oil (38.8 mg, 98% yield). ^{1}H NMR (500 MHz, CDCl3) δ 7.80 (d, J = 7.5 Hz, 1H), 7.62 (td, J = 8.0, 1.5 Hz, 1H), 7.53 (dd, J = 8.0, 1.0 Hz, 1H), 7.44 (t, J = 7.0 Hz, 1H), 7.35 (dd, J = 2.0, 1.0 Hz, 1H), 6.32 (dd, J = 3.0, 2.0 Hz, 1H), 6.12 (d, J = 3.0 Hz, 1H), 4.69 (dd, J = 8.5, 4.0 Hz, 1H), 3.13 (dd, J = 19.0, 8.0 Hz, 1H), 2.88 (dd, J = 19.0, 4.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.1, 155.4, 154.9, 142.4, 136.6, 135.2, 128.5, 126.8, 123.9, 110.5, 106.0, 43.0, 37.9. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C, t_{R1} = 11.1 min (major), t_{R2} = 13.0 min (minor)]; 71:29 er. [α]25D = −7.4 (c = 0.3, $CHCl_3$).

*4.3.23. (R)-3-ethyl-2,3-dihydro-1H-inden-1-one (**2w**) [19]*

Yellow oil (31.4 mg, 98% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, J = 8.0 Hz, 1H), 7.61 (td, J = 7.5, 1.5 Hz, 1H), 7.52 (dd, J = 8.0, 1.0 Hz, 1H), 7.38 (t, J = 7.5 Hz, 1H), 3.39–3.25 (m, 1H), 2.86 (dd, J = 19.0, 7.5 Hz, 1H), 2.38 (dd, J = 19.0, 3.0 Hz, 1H), 2.03–1.92 (m, 1H), 1.62–1.47 (m, 1H), 0.99 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.5, 158.6, 136.8, 134.5, 127.4, 125.5, 123.4, 42.5, 39.5, 28.6, 11.5. HPLC [DAICEL CHIRALPAK OD-H, hexane/iPrOH = 95/5, 254 nm, 0.5 mL/min, 25 °C, t_{R1} = 11.0 min (minor), t_{R2} = 11.6 min (major)]; 88:12 er. [α]25D = −8.3 (c = 0.1, CH_2Cl_2).

4.4. Derivatization of the product

*4.4.1. (S,E)-1-benzylidene-3-phenyl-2,3-dihydro-1H-indene (**3**) [20]*

n-BuLi (0.4 mL, 1.6 M in hexane) was added dropwise to a suspension of benzyltriphenylphosphonium bromide (430.0 mg, 1.11 mmol) in anhydrous THF (20 mL). After the mixture was stirred at room temperature for 2 h, indanone (**2a**, 110.0 mg, 0.53 mmol) in THF (5 mL) was added. The reaction mixture was stirred at reflux for 24 h, then cooled to room temperature, quenched with water (5 mL), and extracted with *n*-hexane (30 mL × 4). The organic layers were combined, washed with water, dried (Na_2SO_4) and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (*n*-hexane) to give the corresponding product **3** (130.0 mg, 92% yield; 92:8 er) as a yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47–7.41 (m, 2H), 7.41–7.36 (m, 2H), 7.35–7.31 (m, 2H), 7.31–7.23 (m, 5H), 7.17–7.11 (m, 1H), 7.04–6.97 (m, 2H), 6.67 (s, 1H), 4.50–4.34 (m, 1H), 3.40 (ddd, *J* = 15.5, 8.0, 1.5 Hz, 1H), 2.96 (ddd, *J* = 15.5, 6.0, 2.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.7, 145.6, 142.0, 139.7, 138.3, 128.8, 128.73, 128.70, 128.66, 128.2, 127.0, 126.7, 126.5, 125.9, 124.4, 122.1, 48.9, 45.5. HPLC [DAICEL CHIRALPAK OJ-H, hexane/*i*PrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 9.1 min (minor), t_{R2} = 27.3 min (major)]; 92:8 er. [α]25D = +265.2 (c = 0.2, CH_2Cl_2).

*4.4.2. (1S,3S)-3-phenyl-2,3-dihydro-1H-inden-1-ol (**4**) [8a]*

To a solution of **2a** (112.0 mg, 0.54 mmol) in methanol (5 mL) was added $NaBH_4$ (24.0 mg, 0.64 mmol) at 0 °C. The mixture was stirred at the same temperature and monitored by TLC, after the reaction was completed, it was quenched with 20 mL of saturated aqueous $NaHCO_3$. The aqueous layer was extracted with EtOAc (15 mL × 4). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (2:1 hexanes/EA) to afford the product **4** (63.3 mg, 86% yield; >20:1 dr) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, J = 7.5 Hz, 1H), 7.35–7.28 (m, 3H), 7.27–7.22 (m, 4H), 6.95 (d, J = 7.5 Hz, 1H), 5.30 (t, J = 7.5 Hz, 1H), 4.19 (t, J = 8.5 Hz, 1H), 3.07–2.99 (m, 1H), 1.99–1.90 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.8, 145.4, 144.4, 128.8, 128.6, 128.5, 127.4, 126.8, 125.3, 123.9, 75.4, 48.5, 47.4. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 14.4 min (minor), t_{R2} = 17.9 min (major)]; 93:7 er. [α]25D = +16.1 (c = 0.1, CH_2Cl_2).

*4.4.3. (S)-4-phenylchroman-2-one (**5**) [8a]*

m-CPBA (200.0 mg, 1.34 mmol) and p-TsOH·H_2O (13.0 mg, 0.068 mmol) were added to a solution of **2a** (70 mg, 0.34 mmol) in CH_2Cl_2 (5 mL), and the mixture was stirred for 24 h under reflux. After cooling to room temperature, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ (5 mL) and $Na_2S_2O_3$ (5 mL). The aqueous layer was extracted with EtOAc (10 mL × 2), the combined organic layers was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (10:1 hexanes/EA) to afford the product **5** (57.9 mg, 76% yield) as a pale yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.27 (m, 4H), 7.19–7.11 (m, 3H), 7.09 (td, J = 7.5, 1.5 Hz, 1H), 6.98 (d, J = 8.0 Hz, 1H), 4.39–4.27 (m, 1H), 3.12–2.99 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.6, 151.6, 140.2, 129.1, 128.7, 128.3, 127.6, 127.5, 125.7, 124.6, 117.1, 40.6, 37.0. HPLC [DAICEL CHIRALPAK OJ-H, hexane/iPrOH = 95/5, 254 nm, 1.0 mL/min, 25 °C. t_{R1} = 11.7 min (minor), t_{R2} = 15.0 min (major)]; 89:11 er; [α]25D = +55.7 (c = 0.5, CH_2Cl_2).

Declaration of competing interest

The authors declare that they have no known competing finical interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tet.2021.132003.

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