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Synthesis of *rac*-α-aryl propionaldehydes *via* branched-selective hydroformylation of terminal arylalkenes using water-soluble Rh-PNP catalyst

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ABSTRACT

This work detailed the preparation of a class of water-soluble PNP ligands that differed by the nature of the substitute on phenyl ring of ligands. These ligands were incorporated into water-soluble rhodium-PNP complex catalysts that were used to regioselective hydroformylation of a series of terminal arylalkenes, providing efficient access to *rac*-α-aryl propionaldehydes in good to excellent yield (up to 97%) and branched-regioselectivity (up to 40:1 *b*/*l* ratio). Furthermore, gram-scale and diverse synthetic transformation demonstrated synthetic application of this methodology for non-steroidal antiinflammatory drugs.

rac-α-Arylaldehydes have been extensively used as important synthetic precursors for the profen family (α-arylpropionic acid derivatives), a class of non-steroidal anti-inflammatory drugs (NSAIDs) in pharmaceutical chemistry [1-5]. At present, more than 20 profen NSAIDs has been used in clinical for treating the heat of inflammation, pain, swelling and fever [6,7]. Some representative examples are illustrated in Fig. 1, such as ibuprofen (**1**) [8], ketoprofen (**2**) [9], flurbiprofen (**3**) [10], naproxen (**4**) [11] and loxoprofen (**5**) [12]. Undoubtedly, the development of efficient approaches towards *rac*-α-arylaldehydes is always a popular topic in both academic and industrial laboratories [13-17]. Traditional methods for synthesis of *rac*-α-arylaldehydes include the rearrangement *via* Darzen's glycidic esters [18,19] and Corey-Chaykovsk's epoxides [20] from aryl methylketones (Schemes 1a and b). However, these processes suffer from several drawbacks such as more complicated synthetic operations, harsh reaction conditions, and functional group incompatibility. Attractive alternatives have been developed using 1,1-arylmethyl alkenes as feedstocks (Scheme 1c) [21-23], including Che's Ru(IV) porphyrin-catalyzed aerobic oxidation *via* an epoxidation/isomerization tandem pathway [21], and Lahiri's catalytic PhIO oxidation by applying iron(II) dipic catalyst, *in situ* formed from $Fe(BF_4)_2{\cdot}6H_2O$ and pyridine-2,6-dicarboxylic acid [22], and Lei's anti-markovnikov oxygenation with H_2O as oxidant by a photoredox-metal dual catalysis [23]. However, protocols currently lack broad substrate scope due to the difficulties for access to 1,1-arylmethyl alkenes as special substrates.

The transition metal-catalyzed hydroformylation of terminal arylalkenes to arylaldehydes could provide an alternative, however typically only a small percent of the β-arylaldehyde products was formed alongside the α-arylaldehyde products. There are remarkable exception of Rh phosphine catalysts [24-35] including Wink's a cationic bis(dioxaphospholane)rhodium complex [24], Marchetti's Rh/pyridylphosphines pydiphos P-oxide complex [25], Amer's Rh/amino phosphine complex [26], Alper's Rh/diphenylphosphinoyl)phenylmethanol catalysis [27], Börner's Rh/diphosphate catalysis [28], Lerous's Rh/dibenzophosphole catalysis *etc.* [29], which form the β-aldehyde products with a good level of selectivity, but only for very limited substrates (Scheme 1d). Recent branched-selective hydroformylation of terminal arylalkenes has been reported using PPh_3-modified Fe as catalyst [30], but the limited success has been achieved toward this hydroformylation (Scheme 1d). Hydroformylations of terminal olefins were independently reported by Nozaki's group and Clarke's group, but the two methods were limited to the preparation of α-alkylaldehyde products [31,32]. Apart from this class of homogeneous catalysis, significant breakthrough have been achieved toward synthesis of β-arylaldehydes *via* biphasic Rh-

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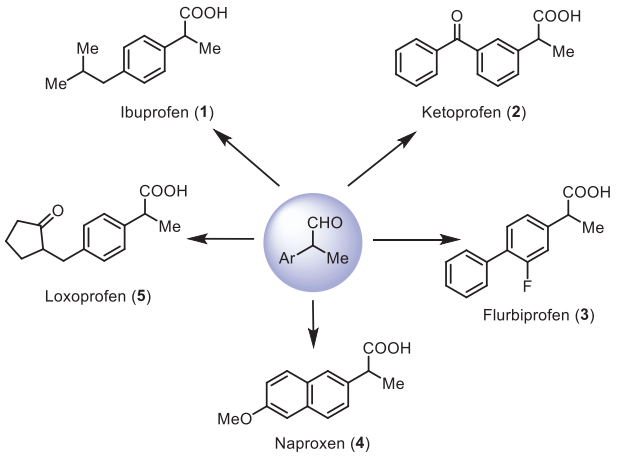
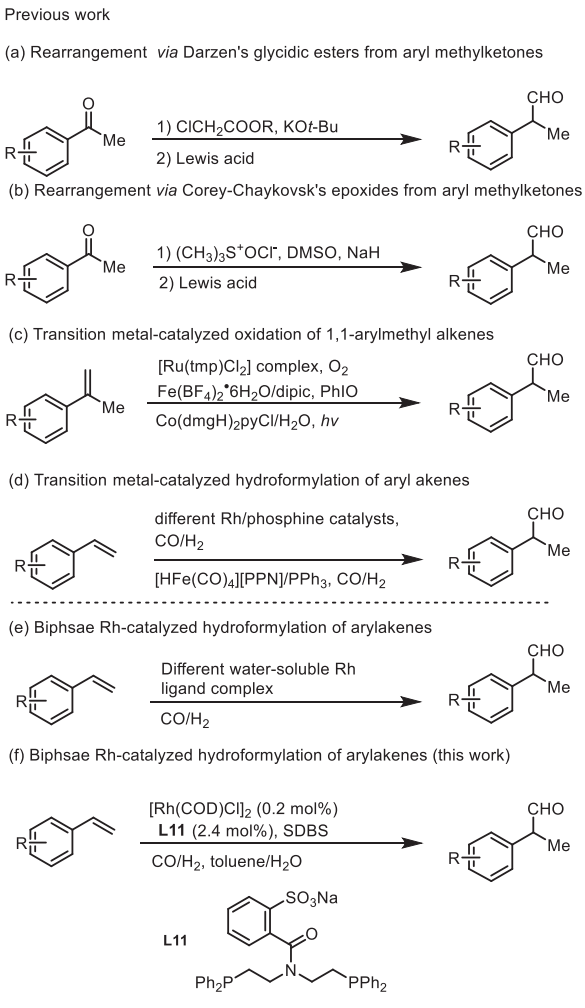


Fig. 1. Representative examples for synthesis of *rac*-profen family (**1**-**5**) from the corresponding *rac*-α-aryl propionaldehydes.



Scheme 1. Strategies to access *rac*-α-arylaldehydes.

catalyzed hydroformylation of terminal olefin using water-soluble poly(4-pentenoic acid (PPA)/bis(2-diphenylphosphino)ethyl) (DPPEA) or tri-*meta*-sulfonatophenylphosphine (TPPTS), or phenyl-β-D-glucopyranoside catalyst as ligands (Scheme 1e) [36-38]. Despite the obtained achievements in this field, the developing of a practical and efficient approach to access the *rac*-α-aryl propionaldehydes is still highly desirable from the perspective of green, step and

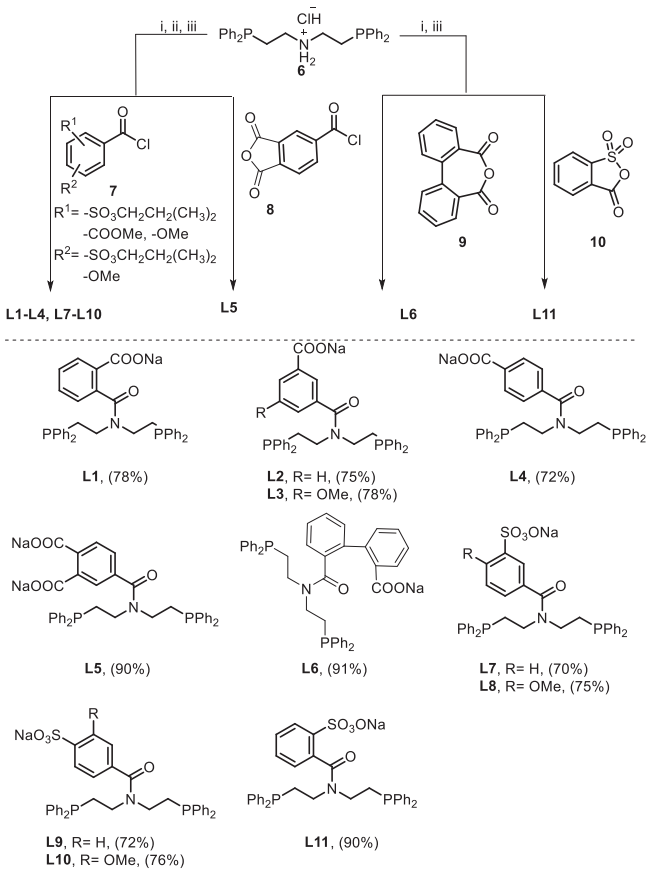


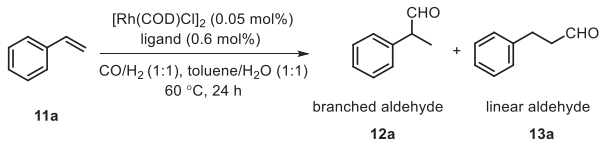
Fig. 2. Synthesis of water-soluble PNP ligands (**L1**-**L11**). (i) TEA, THF, r.t., 20-24 h; (ii) NaI, acetone, reflux, 20-30 h, or LiOH, THF/H_2O, 4-20 h; (iii) NaOH, THF/H_2O, 0.5-1 h.

atom economy [39]. Herein, we report the preparation of a series of novel water-soluble PNP ligands and their applications to the Rh-catalyzed branched-selective hydroformylation of terminal arylalkenes for the synthesis of *rac*-α-aryl propionaldehydes in aqueous biphasic catalytic system.

The known water-soluble PNP ligands **L5** and **L11** and new ligands **L1**-**L4** and **L6**-**L10** were prepared in high to excellent yields from bis(2-diphenylphosphinoethyl)amine hydrochloride (**6**) and appropriate carbonyl halides, and diphenic anhydride (**9**) and 2-sulfobenzoicanhydride (**10**) following a modified procedure developed by Whiteside and co-workers (Fig. 2) [40].

With this library of water-soluble PNP ligands in hand, we began our studies with an examination of their catalytic performance in Rh-catalyzed branched-selective hydroformylation of styrene **11a** as the benchmark substrate. As described in Table 1, all styrene hydrorformylation reactions were completed in 24 h toluene/H_2O (1:1) solvent at 3.0 MPa of syngas and 60 °C in the presence of 0.05 mol% $[Rh(COD)Cl]_2$ and 0.6 mol% ligand (Rh/**L** = 1:6). In all the excellent chemoselectivities were observed, and no hydrogenation product was detected *via* ^1H NMR analysis. The carbonated PNP ligands **L1**-**L6** were first examined. The ligands **L1** and **L2** bearing COONa at benzene's *ortho*- or *meta*-position, gave the branched aldehyde **12a** in moderate yields with good regioselectivities (*b*/*l* = 8.5:1 and 8.4:1, entries 1 and 2), respectively. When the ligand **L3** substituted with *meta*-OMe and *meta*-COONa groups on phenyl ring was employed, poor yield of **12a** was obtained, however, the *b*/*l* ratio in this instance was still exceptional (8.4:1, entry 3). Reaction conducted with the ligand **L4** possessing COONa group at *para*-position of phenyl ring, delivered branched aldehyde **12a** in only 42% yield and with lower regioselectivity (*b*/*l* = 6.5:1, entry 4).

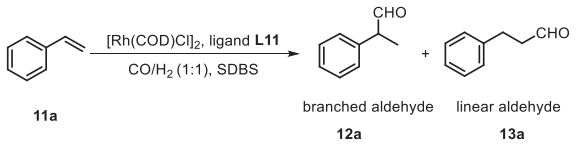
Table 1
Ligands screening for the Rh-catalyzed branched-selective hydroformylation of styrene.[a]



Entry	Ligand	t (h)	Yield of **11a** (%)[b]	*b*/*l* (**12a**/**13a**)[c]
1	**L1**	24	60	8.5:1
2	**L2**	24	49	8.4:1
3	**L3**	24	21	8.4:1
4	**L4**	24	42	6.5:1
5	**L5**	24	75	3.0:1
6	**L6**	24	84	5.4:1
7	**L7**	24	26	3.8:1
8	**L8**	24	76	3.2:1
9	**L9**	24	23	10.1:1
10	**L10**	24	44	4.8:1
11	**L11**	24	92	11.3:1
12	TPPTS	24	74	2.8:1
13[d]	**L11**	24	85	5.9:1
14[e]	**L11**	24	56	10.4:1
15[f]	**L11**	12	91	10.1:1
16[g]	**L11**	12	72	10.0:1
17	-	12	74	2.8:1

[a] Reaction conditions: Styrene **11a** (3.0 mmol), $[Rh(COD)Cl]_2$ (0.05 mol%), ligand (0.6 mol%), toluene (15 mL), H_2O (15 mL), syngas (CO/H_2 = 1:1, 3.0 MPa), 24 h.
[b] GC yield.
[c] Determined by GC.
[d] $Rh(acac)(CO)_2$ (0.1 mol%).
[e] $RhCl_3$ (0.1 mol%).
[f] 0.86 mol% sodium dodecylbenzenesulfonate (SDBS) was used.
[g] Absence of SDBS.

Table 2
Optimization of the Rh-catalyzed hydroformylation of styrene using **L11** as the ligand.[a]

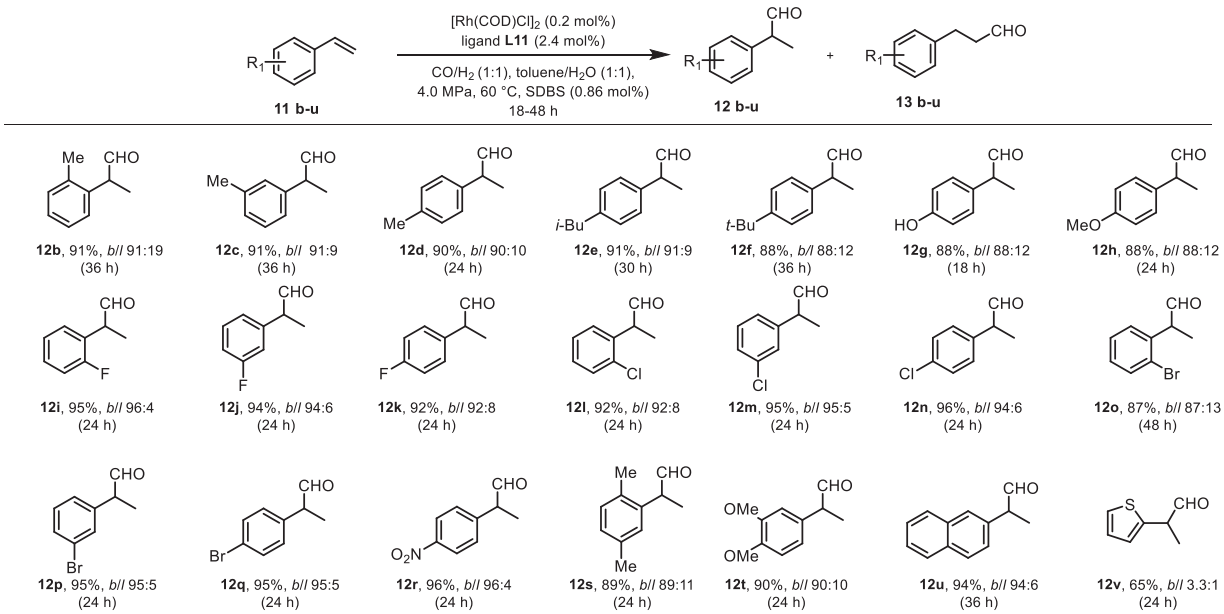


Entry	Rh/**L**	T (°C)	P (MPa)	Solvent	Yield of **12a** (%)[b]	*b*/*l* (**12a**/**13a**)[c]
1	1/6	60	3.0	Tol./H_2O	91	10.1:1
2[d]	1/6	25	3.0	Tol./H_2O	8	11.2:1
3	1/6	50	3.0	Tol./H_2O	70	11.1:1
4	1/6	70	3.0	Tol./H_2O	68	2.1:1
5	1/6	80	3.0	Tol./H_2O	60	1.6:1
6	1/3	60	3.0	Tol./H_2O	78	3.7:1
7	1/8	60	3.0	Tol./H_2O	47	9.7:1
8	1/6	60	2.0	Tol./H_2O	88	7.9:1
9	1/6	60	4.0	Tol./H_2O	92	11.3:1
10	1/6	60	4.0	DCM/H_2O	89	7.9:1
11	1/6	60	4.0	MTBE/H_2O	23	7.5:1
12	1/6	60	4.0	Et_2O/H_2O	19	8.2:1
13	1/6	60	4.0	hexane/H_2O	80	4.0:1
14	1/6	60	4.0	*c*-hexane/H_2O	71	5.1:1
15[e]	1/6	60	3.0	Tol./H_2O	88	8:1

[a] Reaction conditions: substrate (3.0 mmol), $[Rh(COD)Cl]_2$ (0.05 mol%), **L11**, organic solvent (15 mL)/H_2O (15 mL), syngas (CO/H_2 = 1:1), SDBS (0.86 mol%).
[b] GC yield.
[c] Determined by GC.
[d] 10% conversion.
[e] The second recycle.

The dicarboxylated PNP ligand **L5** gave better yield (75%, entry 5) compared with results of monocarboxylated PNP ligands (**L1-L4**), but with poor regioselectivity (*b*/*l* = 3.0:1). Moreover, the monocarboxylated PNP ligand **L6** with a biphenyl ring resulted in much higher yield (84%), albeit with a ratio of **12a**/**13a** of 5.4:1 (entry 6). The sulfonated PNP ligand **L7** with *meta*-SO_3Na gave poor yield and regioselectivity (26%, *b*/*l* = 3.8:1, entry 7) under identical conditions. The ligand **L8** bearing *meta*-SO_3Na and *para*-MeO groups of phenyl ring was shown to give superior yield of **12a**, but much worse regioselectivity (**12a**/**13a** = 3.2:1) was obtained (entry 8). A change of the position of SO_3Na group on ligand **L7** from *meta*- to *para*-position resulted in similar yield (entry 9), but high regioselectivity compared with the results of the corresponding sulfonated PNP ligand **L7-L10**. The use of ligand **L11** with *ortho*-SO_3Na group on phenyl ring afforded branched aldehyde **12a** in 92% yield with high regioselectivity (*b*/*l* = 11.3:1, entry 11). The well-known TPPTS only gave 74% yield of **12a**, with poor regioselectivity (*b*/*l* = 2.8:1, entry 12) under identical conditions. Other common Rh catalysts for hydroformylation, such as $Rh(acac)(CO)_2$ and $RhCl_3$, low regioselectivities and reactivities were respectively obtained (entries 13 and 14). Addition of SDBS (0.86 mol%) as the surfactant could short reaction time to 12 h, and provide branched **12a** in 91% yield and a 10.1:1 ratio of *b*/*l* (entry 15). Branched product was obtained in 72% yield with 10.0:1 ratio of *b*/*l* in absence of SDBS (Table 1, entry 16). The control experiment of absence of ligand was also carried out to give the product in 74% yield with 2.8:1 ratio *b*/*l* (entry 17). These results revealed the crucial role of these two types of ligand structures in controlling of yield and regioselectivity for this Rh-catalyzed hydroformylation reaction.

With the best ligand **L11**, a series of Rh-catalyzed styrene hydroformylation parameters were examined, including temperature, Rh/ ligand **L11** molar ratio, organic/aqueous biphasic system, and CO/H_2 pressure (Table 2). First, the temperature impacted on the activity and regioselectivity of the Rh-catalyzed styrene hydroformylation was investigated. Notably, the activity and regioselectivity of the reaction had obvious temperature effect. Lowering the temperature from 60 °C to 25 °C under standard condition (toluene/H_2O (1:1), 3.0 MPa CO/H_2 (1:1), 0.86 mol% SDBS) dramatically reduced the yield to 8% within 12 h (entry 1 *vs.* 2). Performing the reaction at 50 °C revealed an improved yield of 70% with good regioselectivity (*b*/*l* = 11.1:1, entry 3) with full conversion in 12 h. At temperature over 60 °C, the regioselectivity and yield for the branched **12a** was also significantly reduced (68%, *b*/*l* = 2.1:1 at 70 °C; 60%, *b*/*l* = 1.6:1 at 80 °C; entries 4 and 5). Increasing or decreasing the molar ratio of Rh/ligand **L11** distinctly impaired the yield and regioselectivity for this transformation. A decrease of the Rh ligand **L11** molar ratio from 1:6 to 1:3 led to low regioselectivity (*b*/*l* = 3.7:1, entry 1 *vs.* 6). Moreover, further increasing the molar ratio of Rh/ligand **L11** to 1:8 produced a significant rise in regioselectivity (*b*/*l* = 9.7:1), but with poor yield (47%, entry 7). Varying pressure of syngas (CO/H_2 = 1:1) did not retard the rate but did alter the selectivity towards branched-selective product. Reducing the syngas pressure to 2.0 MPa resulted in a decrease in branched aldehyde **12a** from *b*/*l* = 10.9:1 to 7.9:1 (entry 1 *vs.* 8). A slightly increase in yield and regioselectivity was observed when 4.0 MPa syngas pressure was used for this transformation (entry 9). In addition, the use of other organic/aqueous biphasic systems, suh as DCM/H_2O, MTBE/H_2O, hexane/H_2O, cyclohexane/H_2O, was found to be less effective than toluene/H_2O (entries 10-14). After reaction conditions were screened, the Rh-catalyzed hydroformylation reaction was performed using **L11** as a ligand under 4.0 MPa of syngas (CO/H_2, 1:1) at 60 °C in toluene/H_2O (1:1) biphasic system in the presence of 0.86 mol% SDBS. Optimized conversion, yield for branched aldehyde **12a**, and regioselectivity were achieved under this reaction conditions. The water-soluble Rh/**L11** catalyst can be readily recycled by simple phase separation and still gave 88% yield of **12a** in the second run (entry 15).

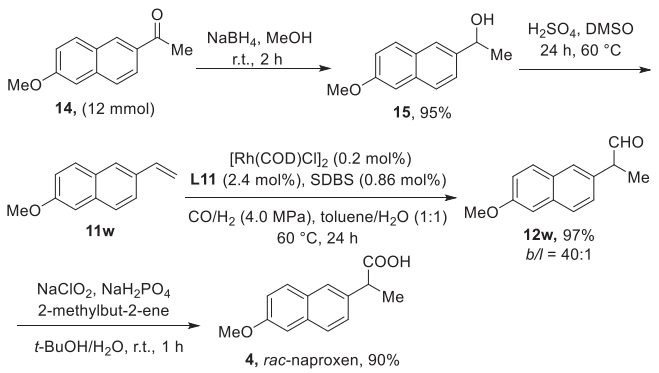


Scheme 2. Scope for the hydroformylation catalyzed by Rh/**L11**. Reaction conditions: Substrate (0.5 mmol), $[Rh(COD)Cl]_2$ (0.2 mol%), **L11** (2.4 mol%), toluene (1 mL), H_2O (1 mL), 60 °C, 18-48 h, syngas (CO/H_2 = 1), SDBS (0.86 mol%), all yields were isolated yield.

With the optimized reaction conditions, the suitability of this protocol was investigated with a range of terminal arylalkenes. As shown in Scheme 2, the mono-substituted phenyl terminal alkenes bearing electron-donating (methyl (**11b**-**11d**), *iso*-butyl (**11e**), *tert*-butyl (**11f**), hydroxyl (**11g**), methoxy (**11h**)), electron-withdrawing (fluro (**11i**-**11k**), chloro (**11l**-**11n**), bromo (**11o**-**11q**), and nitro (**11r**)) groups at any of the position of phenyl ring were hydroformylated efficiently to give the branched aldehydes (**12b**-**12r**) in 83%-94% yield with *b*/*l* = 6.9:1-26:4) with full conversion within 24-48 h. Generally, the mono-substituted phenyl terminal alkenes bearing electron-withdrawing groups exhibited higher reactivity and regioselectivity than their electron-donating groups (**11i**-**11r** *vs.* **11b**-**11h**). Moreover, several sensitive functional groups including chloro (**11l**-**11n**), bromo (**11o**-**11q**), and nitro (**11r**) were well tolerated, no hydrogenolysis or hydrogenation products was detected by GC analysis. Similarly, disubstituted phenyl termial alkenes with electron-donating (dimethyl **11s**, dimethoxy **11t**) groups were also smoothly hydroformylated into the branched products **12s**-**12t** in 86% and 90% yield with good regioselectivity (*b*/*l* = 8.1:1 and 9.1:1) resepectively. In addition to phenyl terminal alkenes, 2-naphthyl terminal alkene (**11u**) was also a suitable substrate for this hydroformylation reaction, delivering the branched product **12u** in 89% yield with *b*/*l* = 15.1:1. The heterocylcic olefin bearing thienyl ring also smoothly transformed to the corressponding product **12v** in 65% yield along with low selectivity (*b*/*l* = 3.3:1).

To further evaluate the efficiency and synthetic utility of this branched selective hydroformylation, we carried out the scale-up experiment using 6 mmol (1.10 g) of 6-methoxy-2-vinylnaphthalene **11w** [41], which could be easily prepared in two steps with an overall yield of 50% starting from commercially available 6-methoxy-2-acetonaphthalene (**14**). The branched-selectivehydroformylation of **11w** could be achieved using 0.2 mol% of the Rh/**L11** catalyst within 24 h under standard reaction conditions (4.0 MP of CO/H_2 (1:1)) at 60 °C in tulene/H_2O (1:1) to give the aldehyde **12w** in 97% yield with *b*/*l* = 40:1. Oxidation of crude product **12w** under Pinnick reaction conditions ($NaClO_2$, $KHPO_2$, 2-methyl-2-butene, *t*-BuOH, H_2O, 0 °C to r.t., 1 h) provided *rac*-naproxen (**4**) in 90% isolated yield (Scheme 3) [42].

In conclusion, we prepared a series of new water-soluble PNP rhodium catalysts and screened their catalytic performance in biphasic aqueous hydroformylation of vinyl arenes. A benzenesulfonates-containing PNP ligand **L11** gave the most efficient Rh complex catalyst, affording high regioselectivities towards α-aryl propionaldehydes from the branched-selective hydroformylation of various terminal arylalkenes. The exploration of substrate scope showed that the vinyl arenes with electron-withdrawing groups were usually more branched selective than those bearing electron-donating groups. Finally we demonstrated an aqueous route to the synthesis of anti-inflammatory drugs *rac*-naproxen in an efficient and green manner.



Scheme 3. Gram-scale synthesis of *rac*-naproxen (**4**).

Declaration of competing interest

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

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