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Heck-type reactions of allylic alcohols Part IV: (2-Substituted)-1-indanones *via* 5-*endo-trig* cyclizations☆

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Abstract

Various conditions have been tested to obtain efficiently 2-methyl-1-indanone *via* the Pd-catalyzed 5-*endo-trig* cyclization of 1-(*o*-bromophenyl)-2-methylprop-2-en-1-ol. High yield (97%) was obtained at 120 °C in DMF with $Pd(OAc)_2$/cinchonine as the catalytic system and $NaHCO_3$ as the base. Use of this procedure for the synthesis of other substituted indanones led to lower yields but replacing thermal heating by microwave heating improved greatly the results.

Keywords: Palladium; Catalysis; Heck reaction; Cyclization; Allylic alcohol; DMF; Cinchonine; Microwaves

1. Introduction

Over the last past years, we have studied the asymmetric protonation of prochiral enolic species with catalytic amounts of homochiral amino alcohols [1,2]. Since the Heck arylation of allylic alcohols can afford ketones *via* the formation of an enol as intermediate [3], we envisaged the synthesis of optically active ketones using this procedure. However, the Pd-catalyzed reactions of phenyl iodide with 3-methyl-3-buten-2-ol in the presence of homochiral amino alcohols led to very low ee's whatever the experimental conditions [4,5]. In the aim to obtain, as intermediate, a single enol configuration which, furthermore, would be rigid, we have subsequently examined the 5-*endo-trig* cyclization of 1-(*o*-bromophenyl)-2-methylprop-2-en-1-ol (**1a**) which possesses a prochiral allylic alcohol moiety to obtain 2-methyl-1-indanone (**2a**). When we started these studies [4], such a Pd-catalyzed cyclization was only reported from (*Z*)- and (*E*)-1-(*o*-bromo-phenyl)-oct-2-en-1-ols, in the absence of a chirality source, leading to 3-pentyl-1-indanone in 48–52% yields [6].[1] According to the Baldwin's rule, the 5-*endo-trig* cyclization is, furthermore, a disfavored reaction [7–9]. In the course of our studies, Pan and co-workers have however synthesized 1*H*-inden-1-ones from the Pd-catalyzed reaction of 1-(*o*-bromoaryl)-prop-2-en-1-ols under an air atmosphere [10]. According to these authors, this reaction involved the intramolecular Heck-type cyclization followed by the aerial oxidation of the resulting 1*H*-inden-1-ols. Our results are here reported.

2. Results and discussion

Preliminary experiments have been carried out at 120 °C in DMF using the prochiral substrate **1a**, $Pd(OAc)_2$ as the catalyst, $NaHCO_3$ as the base, and a catalytic amount of cinchonine or (+)*endo*-2-hydroxy-*endo*-3-aminobornane. The expected ketone, **2a**, was isolated in high yields but, unfortunately, was racemic (Table 1, runs 1 and 2). Lower reaction temperatures decrease the efficiency of the reaction without improving the optical activity. The use of (*R*)-BINAP instead

☆ For Part III, see Ref. [43].

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[1] Since the submission of the present study, new examples have been reported by Ray et al. [44].

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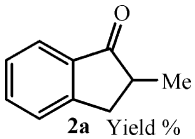
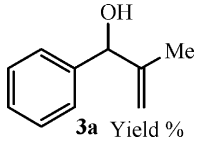
Table 1
Reactivity of 1-(*o*-bromophenyl)-2-methylprop-2-en-1-ol in DMF at 120 °C using 0.05 equiv. of $Pd(OAc)_2$

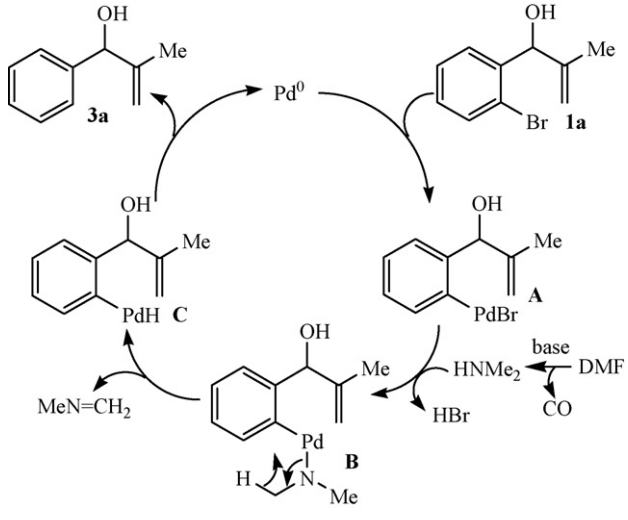
Run	Additive (0.1 equiv.)	Base (1.1 equiv.)	Time h	Conversion (%)	 **2a** Yield %	 **3a** Yield %
1	Cinchonine	$NaHCO_3$	19	100	97	0
2	HNBOH[a]	$NaHCO_3$	17	100	87	0
3	(*R*)-BINAP	$NaHCO_3$	17	100	37	53
4	PPh_3	$NaHCO_3$	22	100	80	0
5	None	$NaHCO_3$	16	95	86	0
6	None	$NaHCO_3$	22	100	82	0
7[b]	PPh_3	$NaHCO_3$	22	100	56	32
8	Cinchonine	Cs_2CO_3	8	100	62	22
9	Cinchonine	Na_2CO_3	22	100	93	0
10	Cinchonine	NaOAc	16	79	69	0
11	Cinchonine	TlOAc	16	86	30	42
12	Cinchonine	KF/Al_2O_3[c]	8	100	11	76
13	Cinchonine	NEt_3	22	90	83	0
14[d]	Cinchonine	Cy_2NMe	24	17	12	0
15	Cinchonine (1.2 equiv.)		19	37	30	0

[a] HNBOH: (+)*endo*-2-hydroxy-*endo*-3-aminobornane [13].
[b] Using $Pd_2(dba)_3{\cdot}CHCl_3$ as the catalyst.
[c] 500 mg/mmol; prepared as previously described [14].
[d] Reaction carried out at 100 °C.

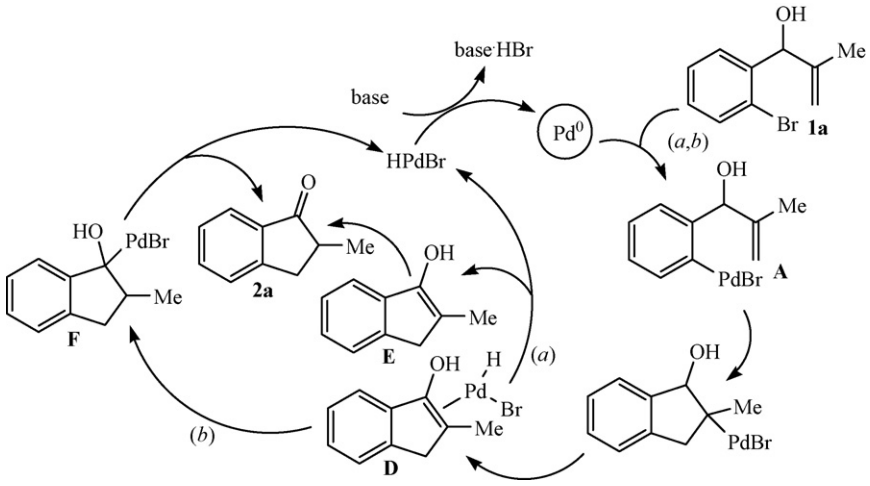
of the amino alcohol led to a mixture of **2a** and 1-phenyl-2-methylprop-2-en-1-ol (**3a**), **2a** being once more racemic (run 3). With PPh_3 as the ligand or in the absence of ligand, **2a** was selectively obtained when $Pd(OAc)_2$ was the catalyst (runs 4–6), while the $Pd_2(dba)_3{\cdot}CHCl_3/PPh_3$ system affords **2a** and **3a** (run 7). Complementary experiments using various inorganic and organic bases have shown the absence of enantio-induction by cinchonine, and the determining role of the base on both selectivity and efficiency of the reaction (runs 11–15). Thallium acetate and, in particular, KF/Al_2O_3 afforded **3a** as the main product (runs 11 and 12). Triethylamine yielded effectively **2a** (run 13) while *N*,*N*-dicyclohexylmethylamine and the use of only cinchonine led to low conversions (runs 14 and 15). Under all these conditions, we never detected the formation of 2-methyl-1*H*-inden-1-one or 2-methyl-1*H*-inden-1-ol. Given the results depicted in Table 1, it appears that the cinchonine/$NaHCO_3$ mixture is the most effective combination to obtain **2a**. We suspect that cinchonine can be a *N*,*O*- or *N*,*N*-bidentate ligand stabilizing palladium species [11]. Nevertheless, the oxidative addition reaction of the substrate to the Pd^0 species could occur from a monoligated palladium complex [12].

The formation of **3a** corresponds to the hydrogenolysis of the Ar–Br bond. We have recently reported [15] that, in the presence of an inorganic base, the hydrogenolysis is due to the decomposition of DMF [16] and reaction of the resulting dimethylamine with ArPdBr species [17] as depicted in Scheme 1. Intermediate **A**, formed by insertion of Pd^0 into the Ar–Br bond, reacts with dimethylamine to yield **B** that suffers a β–H elimination [18]. Reductive elimination of Pd^0 from the resulting ArPdH complex (**C**) gives **3a**. As previously observed [15], the efficiency of this hydrogenolysis process, that uses DMF as the hydrogen source, depends on the experimental conditions.

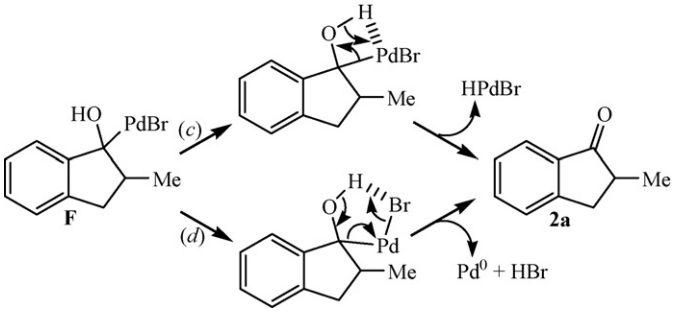
The obtention of racemic **2a** from reactions carried out in the presence of homochiral aminoalcohols could be due to the experimental conditions, namely basic conditions and high temperature, that could induce the racemisation of the optically active ketone. Furthermore, the absence of enantioselectivity could also be due to the mechanism of the Heck reaction. Let us to consider the Heck-type cyclization of **2a** (Scheme 2). The formation of enolic intermediate **D** is admitted for such a reaction [4,19]. The ketone could be obtained from **D** *via* either free prochiral enol **E** (path *a*) or addition/elimination of HPdBr (path *b*) but Smadja et al. have shown that path *a* is, at best, a minor reactive pathway [20]. According to studies on the Pd-induced domino reaction of benzyl β-ketoesters [21] carried



Scheme 1.



Scheme 2.

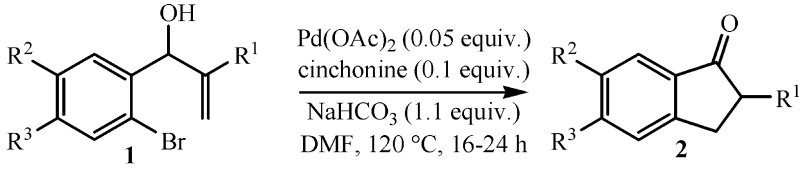


Scheme 3.

out in the course of the present work, the enantioselection step leading to chiral ketones from enolic species and homochiral amino alcohols occurs at the level of the free enol. The above comments are in agreement with the formation of racemic **2a**.

It seems of interest to point out that the Heck reaction is mechanistically germane to the mode of the Wacker oxidation of ethylene. Indeed, the Wacker process produces an enolic intermediate similar to **D**, and it has been shown that the β-H elimination product never leaves the coordination sphere of the palladium at this level, the formation of the aldehyde occurring through the addition/elimination of HPdX [22–24]. Recent calculations on the Wacker mechanism from Goddard and coworkers [25] led us to envisage a halide-mediated reductive elimination (Scheme 3, path *d*) rather than the usually accepted β-hydride elimination (Scheme 3, path *c*) for the **F** → **2a** step of the Heck reaction.

Since we have been able to carry out the 5-*endo-trig* cyclization of **1a** in high yields, this type of reaction was examined using other substrates under the experimental conditions of run 1, Table 1 (Eq. (1), Table 2). Changing the methyl substituent for a phenyl or an ethyl provided expected cyclized products **2b** and **2c** with fair yields, while the yield decreased to 42% in the absence of a substituent in this position. The cyclization of ethyl 2-((*o*-bromophenyl)(hydroxy)methyl)acrylate (**1e**) led to the concomitant cleavage of the C–CO_2Et bond to afford **2d**; this is likely due to the instability of β-ketoester **2e** under the experimental conditions [26]. A low yield of cyclized product **2f** was isolated from **1f** that has a methoxy substituent on the aromatic moiety. In the presence of two methoxy substituents or one nitro substituent on the aromatic moiety (**1g** and **1h** respectively), some conversion of the substrate was observed but without production of the expected cyclized product.



b: R^1 = Ph, R^2 = R^3 = H; **c**: R^1 = Et, R^2 = R^3 = H; **d**: R^1 = R^2 = R^3 = H; **e**: R^1 = CO_2Et, R^2 = R^3 = H; **f**: R^1 = Me, R^2 = OMe, R^3 = H; **g**: R^1 = Me, R^2 = R^3 = OMe; **h**: R^1 = Me, R^2 = NO_2, R^3 = H (1)

According to the literature, Heck reactions are often improved under microwave irradiation [27,28]. The efficiency of this activation method, that has seldom been used for Heck reactions of allylic alcohols [29], has been studied from our substrates (Table 3). The reactions have then be carried out under the conditions depicted in Eq. (1) except the use of microwave heating at 100 °C with a microwave power of 300 W for 0.5 h, instead of oil-bath heating at 120 °C for 16–24 h. As exemplified by the results compiled in Table 3, this procedure increased strongly the yields from **1b**, **1c**, **1d**, **1e** and **1f**. Moreover, **2g** was thus isolated in 61% yield while this compound was not isolated under thermal heating conditions. Nevertheless, the formation of **2h** remains precluded under these conditions.

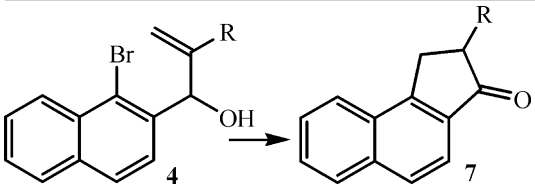
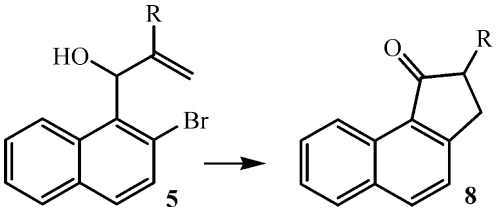
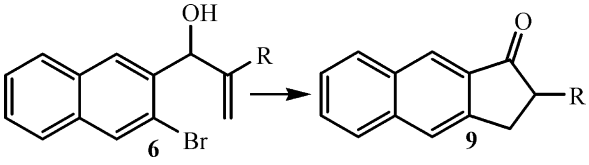
Table 2
Pd-catalyzed reaction of allylic alcohols **1b–1h** under the conditions depicted in Eq. (1)

Substrate	**1b**	**1c**	**1d**	**1e**	**1f**	**1g**	**1h**
Time (h)	16	24	24	21	24	24	24
Product (yield %)	**2b**, 67	**2c**, 56	**2d**, 42	**2d**, 47	**2f**, 12	**2g**, 0	**2h**, 0

Table 3
Pd-catalyzed reaction of allylic alcohols **1b–1h** under microwave irradiation

Substrate	**1a**	**1b**	**1c**	**1d**	**1e**	**1f**	**1g**	**1h**
Product (yield %)	**2a**, 92	**2b**, 90	**2c**, 93	**2d**, 86	**2d**, 86	**2f**, 84	**2g**, 61	**2h**, 0

Table 4
Pd-catalyzed reaction of naphthalene derivatives under thermal (Δ) and microwave (μw) conditions[a]

Reaction	R	Δ (yield %)	μw (yield %)
	Me: **a**	44	87
	Ph: **b**	0	63
	Me: **a**	56	92
	Ph: **b**	10	68
	Me: **a**	24	74
	Ph: **b**	0	23

[a] All reactions were carried out with relative amounts of reactants as those of Table 1, run 1 by heating with an oil-bath at 120 °C for 20 h (Δ) or microwaves at 100 °C (microwave power: 300 W) for 0.5 h (μw).

These results urge us to examine the 5-*endo-trig* cyclization of naphthalene derivatives **4**, **5** and **6** (R = Me (**a**) or Ph (**b**)) under both heating methods (Table 4). Under the thermal conditions, incomplete conversions were attained in 20 h; moderate yields of ketones **7a**, **8a** and **9a** were obtained from substrates with the methyl substituent while, with a phenyl substituent, the expected cyclized compound was isolated only from **5b**. Shifting to microwave conditions increased greatly the results, a low yield being obtained only from **6b**.

3. Conclusion

The Pd-catalyzed 5-*endo-trig* cyclization of 1-(*o*-bromoaryl)-2-substituted-prop-2-en-1-ols can efficiently proceed, particularly under microwave irradiation, using cinchonine as the ligand, sodium bicarbonate as the base and DMF as the solvent. The chemical yields are nevertheless sensitive to the substitution pattern of the substrate. The absence of optical activity for the cyclized products could be due to the required experimental conditions and/or to the nature of the intermediates.

4. Experimental

Substrates **1a–1d**, **1f–1h**, **4a**, **4b**, **5a**, **5b**, **6a** and **6b** have been prepared by addition of Grignard reagents to bromoarylaldehydes following a reported procedure [30] (see Supplementary Material for yields and characterization data). The synthesis of **1e** was carried out *via* a Baylis-Hillmann reaction as reported [31].

4.1. Cyclization procedure

To a mixture of $Pd(OAc)_2$ (11 mg, 0.05 equiv.), cinchonine (30 mg, 0.1 equiv.) and $NaHCO_3$ (92 mg, 1.1 equiv.) under an argon atmosphere was added a solution of the substrate (1 mmol) in DMF (2 mL). The mixture was heated either at 120 °C with an oil-bath for the time indicated in Tables, or at 100 °C for

0.5 h using a microwave apparatus (CEM-Discover, LabMate type) with a microwave power held at 300 W. After cooling to room temperature and addition of diethyl ether, the mixture was filtered over a small pad of Celite. The filtrate was successively washed with water and brine, then dried over $MgSO_4$. After elimination of the solvent, the product was isolated by flash-chromatography.

The NMR data of **2a** [32], **2b** [33], **2c** [34], **2d** [35], **2f** [36], **2g** [37], **3a** [38], **8a** [39], **8b** [40] and **9b** [41] were in agreement with those of literature.

2-Methyl-1,2-dihydrocyclopenta[*a*]naphthalen-3-one (**7a**): mp 70–71 °C (literature [42] 71–72 °C). ^{1}H NMR (250 MHz, $CDCl_3$): (1.29 (d, 3H, J = 8.2 Hz, CH_3), 2.70 (m, 1H, CH_2), 2.86 (dd, 1H, J = 17.6, 3.3 Hz, CH_2), 3.57 (m, 1H, CH), 7.46-7.59 (2H, H_{arom}), 7.62 (d, 1H, J = 8.6 Hz, H_{arom}), 7.68 (d, 1H, J = 8.6 Hz, H_{arom}), 7.81 (d, 1H, J = 7.6 Hz, H_{arom}), 7.90 (d, 1H, J = 7.6 Hz, H_{arom}). ^{13}C NMR (62.9 MHz, $CDCl_3$): (16.7, 33.4, 41.9, 119.8, 124.5, 127.1, 128.6, 128.9, 129.2, 130.4, 133.8, 136.7, 154.8, 209.4.

2-Phenyl-1,2-dihydrocyclopenta[*a*]naphthalen-3-one (**7b**): mp 65–67 °C. ^{1}H NMR (250 MHz, $CDCl_3$): (2.81 (dd, 1H, J = 14.3, 2.0 Hz, CH_2), 3.43 (dd, 1H, J = 14.3, 7.5 Hz, CH_2), 3.90 (dd, 1H, J = 7.5, 2.0 Hz, CH), 7.10–7.30 (5H, H_{arom}), 7.51–7.65 (2H, H_{arom}), 7.69 (t, 1H, J = 8.4 Hz, H_{arom}), 7.76 (t, 1H, J = 8.4 Hz, H_{arom}), 7.88 (d, 1H, J = 8.4 Hz, H_{arom}), 7.98 (d, 1H, J = 7.5 Hz, H_{arom}). ^{13}C NMR (62.9 MHz, $CDCl_3$): (34.5, 53.3, 120.1, 124.6, 126.1, 127.0, 127.6, 129.1, 129.5, 133.9, 136.9, 140.0, 155.2, 205.9.

2-Methyl-2,3-dihydrocyclopenta[*b*]naphthalen-1-one (**9a**): mp 88–89 °C. ^{1}H NMR (250 MHz, $CDCl_3$): (1.29 (d, 3H, J = 7.3 Hz, CH_3), 2.74 (m, 1H, CH_2), 2.82 (dd, 1H, J = 17.2, 5.0 Hz, CH_2), 3.48 (m, 1H, CH), 7.35–7.54 (2H, H_{arom}), 7.72–7.80 (2H, H_{arom}), 7.89 (d, 1H, J = 8.0 Hz, H_{arom}), 8.25 (s, 1H, H_{arom}). ^{13}C NMR (62.9 MHz, $CDCl_3$): (14.6, 32.8, 41.1, 122.9, 124.3, 126.0, 126.7, 128.6, 130.6, 132.4, 135.5, 144.5, 208.0.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molcata.2007.12.021.

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