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Pd-catalyzed carbonylation of α-arylvinyl bromides: Synthesis of 2-arylacrylic esters

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Abstract

(1,2-Dibromomoethyl)arenes, obtained from the bromination of styrene derivatives were regioselectivley dehydrobrominated to α-arylvinyl bromides in very good yields (82–94%). The vinyl bromides generated in situ underwent palladium-catalyzed carbonylation furnishing 2-arylacrylic esters, which are precursors for non-steroidal anti-inflammatory drugs. The best catalytic system is composed of $PdCl_2(PPh_3)_2$ (0.5–2 mol%), PPh_3 as phosphine ligand and *i*PrEtN as base. The carbonylation reaction can be carried out under mild conditions (10 atm of CO, 100 °C, 3.5 h) affording the esters in very good yields (88–95%). Moreover, we have developed a one-pot deydrobromination/carbonylation procedure.

Keywords: Carbonylation; Palladium; Vinyl bromides; 2-Arylacrylic esters; Anti-inflammatory drugs

1. Introduction

Pd-Catalyzed carbonylation reactions provide highly promising and eco-friendly routes for the synthesis of non-steroidal anti-inflammatory profen drugs [1–3]. The latter have as the common structural feature a 2-aryl-substituted propionic acid and they have been obtained in acid or ester forms by the hydroxy- or alkoxycarbonylation of vinyl aromatics [4–14], halides [15–18] and alcohols [19–21]. In this context, the Hoechst–Celanese process for the synthesis of Ibuprofen™ is considered to be one of the best examples of the role of catalysis in developing cleaner, environmentally benign routes for replacing stoichiometric organic synthesis [22]. This process employs three steps instead of the five in the original Boots process, with 100% atom-efficiency, where the key-step is the Pd-catalyzed carbonylation of 1-(*p*-isobutyphenyl)-ethanol [23]. On the other hand, Pd-catalyzed carbonylations of aryl acetylenes have been also used for the synthesis of 2-arylacrylic acids or esters [1–3,24–29]. These compounds are useful precursors for the enantioselective synthesis of 2-arylpropionic acids. For instance, in the Monsanto process [30,31], (*S*)-Naproxen™ is obtained in 100% yield and up to 98% ee from the asymmetric hydrogenation reaction of 2-(6′-methoxy-2′-naphthyl)acrylic acid catalyzed by a Ru-BINAP complex [32]. However, despite carbonylation being an efficient and regioselective method for preparing 2-arylacrylic acids, this reaction has the disadvantage of using expensive alkynes as starting materials. Alternatively, 1-bromo-1-(6′-methoxy-2′-naphthyl) has also been carbonylated to afford the unsaturated Naproxen™ precursor although here the vinyl bromide was also obtained from the hydrobromination of the corresponding alkyne [33]. Recently, we have used 1,2-dibromoethane to generate in situ vinyl bromide that was coupled with aryl boronic acids in the presence of a palladium catalyst to afford styrene derivatives [34]. The same protocol was successfully applied to the synthesis of geminal diarylethylenes by using (1,2-dibromoethyl)-benzene obtained from the bromination of styrene. Since functionalized styrenes are more accessible and cheaper than the corresponding alkynes, we decided to extend our dehydrobromination/Pd-catalyzed protocol to the synthesis of 2-arylacrylic esters. Herein we present a simple sequence starting from styrene derivatives (bromination/dehydrobromination/carbonylation), where the Pd-catalyzed reaction is the key step to afford 2-arylacrylic esters in very good yields (Scheme 1).

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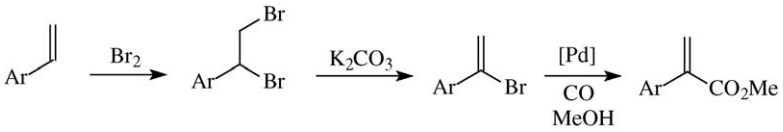


Scheme 1. Synthesis of 2-arylacrylic esters from styrene derivatives.

2. Experimental

2.1. General experimental procedures

Catalytic reactions were carried out under carbon monoxide pressure in a 100 ml-stainless steel autoclave. NMR spectra were recorded on a Varian VXR-200 or Varian XL 300. Infrared spectra were recorded on a Bomem B-102 spectrometer. Mass spectra were recorded on a GC/MS Shimadzu QP-5050 (EI, 70 eV).

Solvents were dried under adequate drying agents and distilled under argon prior to use. $PdCl(PPh_3)_2$ [35] and *p*-isobutylstyrene [36] were synthesized as described in the literature. 3-Vinylbenzophenone and 6-methoxy-2-vinyl-naphthalene were obtained form the coupling of the corresponding aryl bromides with ethylene under Heck conditions [3,37]. The (1,2-dibromomoethyl)arenes were obtained in high yields (89–100%) by bromination of the substituted styrenes at 0 °C using CCl_4 as solvent [38]. The other reagents were purchased from commercial sources and used without further purification.

2.2. Typical procedure for the deydrobromination reaction

A mixture of 1,2-dibromo-1-(*p*-isobutylphenyl)ethane (1.6 g, 5 mmol), K_2CO_3 (1.382 g, 10 mmol), THF (10 ml), and methanol (10 ml) was stirred under reflux for 1 h. The mixture was then allowed to cool to room temperature, taken up in ether (20 ml) and washed with aqueous KOH (10%, 5 ml) and brine (2 × 5 ml), and then dried over $MgSO_4$. After filtration, solvent was evaporated to give 1-bromo-1-(*p*-isobutylphenyl)ethene as an oil (1.12 g, 94%). ^{1}H NMR(200 MHz, $CDCl_3$) δ 0.92(6H, d, J = 6.59 Hz), 1.80–1.98 (1H, m), 2.48 (2H, d, J = 7.08 Hz), 5.73(1H, d, J = 1.46 Hz), 6.09 (1H, d, J = 1.46 Hz), 7.12 (2H, d, J = 8.06 Hz), 7.51 (2H, d, J = 8.11 Hz). ^{13}C NMR (50 MHz, $CDCl_3$) δ 22.59, 30.41, 45.25, 117.00, 127.26, 129.18, 131.34, 136.2, 141.3. IR (neat): 1611 cm^{-1}. GC-MS (IE 70 eV) *m/z* (%): 240(19, M + 2), 238(20, M), 197(32), 195(33), 159(100), 116(56), 115(61), 89(10).

2.3. General procedure for the optimization of the carbonylation reaction

A mixture of 1,2-dibromo-1-phenylethane (1 mmol), K_2CO_3 (0.138 g, 1 mmol), THF (6 ml), and methanol (6 ml) was stirred under reflux for 1 h. The mixture was then allowed to cool to room temperature, and analyzed by GC. After filtration, the solution was added to a 100 ml-stainless steel autoclave containing $PdCl_2(PPh_3)_2$, phosphine, base and solvent. The reactor was pressurized with 10 atm of CO and the reaction mixture was stirred at the desired temperature during the desired time. After cooling and releasing the excess carbon monoxide, the reaction mixture was analyzed by GC and GC-MS.

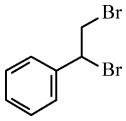
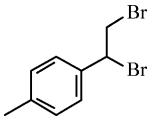
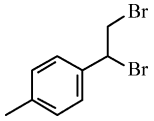
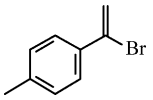
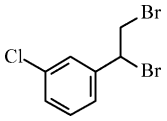
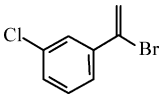
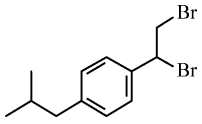
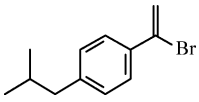
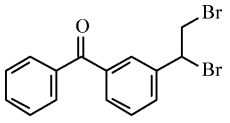
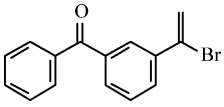
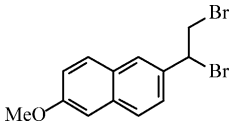
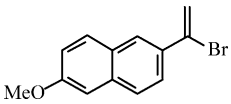
2.4. Typical procedure for the carbonylation reaction

In a 100 ml-stainless steel autoclave under argon were placed $PdCl_2(PPh_3)_2$ (56.1 mg, 0.08 mmol), PPh_3 (42.0 mg, 0.16 mmol), 1-bromo-1-(*p*-isobutylphenyl)ethene (956.6 mg, 4 mmol), methanol (8 ml) and THF (8 ml). The reactor was pressurized with 10 atm of CO and the reaction mixture was stirred at 100 °C for 3.5 h. After cooling and releasing the excess carbon monoxide, the reaction mixture was filtered over Celite and the solvent was evaporated. Distillation under reduced pressure in a Kugelrohr apparatus afforded the methyl 2-(*p*-isobutylphenyl)acrylate as an oil (94%). ^{1}H NMR(200 MHz, $CDCl_3$) δ 0.91 (6H, d, J = 6.59 Hz), 1.78–1.98 (1H, m), 2.48 (2H, d, J = 7.09 Hz), 3.82 (3H, s), 5.88(1H, d, J = 1,20 Hz), 6.31 (1H, d, J = 1.15 Hz), 7.13 (2H, d, J = 8.25 Hz), 7.33 (2H, d, J = 8.06 Hz). ^{13}C NMR (50 MHz, $CDCl_3$) δ 22.69, 30.48, 45.41, 52.45, 126.37, 128.21, 129.13, 134.3, 141.4, 142.13, 167.7. IR (neat): 1725, 1610 cm^{-1}. GC-MS (IE 70 eV) *m/z* (%): 218 (29, M), 176 (35), 175 (100), 145 (6), 121 (31), 117 (12), 116 (24), 115 (30).

3. Results and discussion

The α-arylvinyl bromides can readily be prepared from the corresponding substituted styrenes by a bromination/dehydrobromination procedure. The (1,2-dibromomoethyl) arenes were obtained in high yields (89–100%) by bromination of the substituted styrenes at 0 °C using CCl_4 as solvent. Dehydrohalogenation is generally carried out in solution where the most frequently used bases are KOH and NaOH. For instance, 1,2-dibromo-(*p*-isobutyl)ethane was dehydrobrominated with KOH/MeOH at 0 °C to give 1-bromo-1-(*p*-isobutylphenyl)ethene in 92% yield [39]. A set of experiments for the dehydrobromination of (1,2-dibromomoethyl)benzene was performed looking for reaction conditions that could be compatible for both dehydrobromination and carbonylation reactions. Among the bases (NEt_3, K_3PO_4, $NaHCO_3$, Na_2CO_3, K_2CO_3, *t*BuONa, NaOAc) and solvent media (MeOH, EtOH, iPrOH, THF, MeOH/THF, H_2O/THF, MeOH/toluene) examined, the best results were obtained using K_2CO_3 as base in a 1:1 mixture of THF/MeOH. Under the conditions studied the α-bromostyrene was always obtained with 95% regioselectivity. The dehydrobromination of a wide variety of (1,2-dibromomoethyl)arenes was carried out under optimized reaction conditions furnishing the corresponding α-arylvinyl bromides in very good yields (Table 1).

Table 1
Dehydrobomination of (1,2-dibromomoethyl)arenes[a]

Entry	Substrate	Product	Yield (%)[b]	α:β
1			90	95:5
2			82	92:8
3			89	97:3
4			94	91:9
5			92	99:1
6			94	100

[a] Reaction conditions: (1,2-dibromomoethyl)arene (5 mmol), K_2CO_3 (10 mmol), methanol (10 ml), THF (10 ml), 1 h, reflux.
[b] Isolated yield.

The palladium catalyzed alkoxycarbonylation of alkenyl halides was initially explored by Heck et al. [40]. Galamb and Alper have shown that bromostyrenes can be carbonylated to afford acids in good yields, and under mild conditions, using Pd(0) and phase transfer catalysis [41]. These reactions usually occur with predominant or complete retention of configuration. 1-Bromoalkenes are also converted into acids in reasonable yields, whereas 2-bromoalkenes are quite unreactive. The carbonylation of vinyl halides was used to produce the unsaturated precursor to Naproxen™ [33]. Thereafter, carbonylation of 1-bromo-1-(6-methoxy-2-naphthyl)ethene was carried out under 20 atm of CO in the presence of $Pd(PPh_3)_4$ (5 mol%) as catalyst, Et_3N as base and in a mixture of MeOH/THF at 70 °C for 21 h. Under these conditions a 96% conversion of vinyl bromide was observed and methyl 2-(6-methoxy-2-naphthyl)acrylate was isolated in 90% yield [33]. In order to improve these conditions and generalize for α-arylvinyl bromides, the carbonylation of α-bromostyrene was examined as a model reaction.

An initial screening was performed in order to determine the conditions for the optimization studies. In order to have a simple protocol α-bromostyrene was produced in situ from the dehydrobromination of (1,2-dibromomoethyl)benzene. At the end of the dehydrobromination reaction the mixture was filtered and the solution was added to a 100 ml-stainless steel autoclave for the carbonylation reaction studies. No conversion was observed in the absence of palladium compounds or in the presence of palladium compounds without phosphine ligand. Among the phosphine ligands examined (PPh_3, PCy_3, P(*o*-tol)$_3$, dppe, dppp, xantaphos, binap) triphenylphosphine gave the best results in terms of conversion and selectivity. As observed for the synthesis of the unsaturated precursor to Naproxen™ [33] $PdCl_2(PPh_3)_2$, an air-stable and easy to make complex, showed to be an excellent catalyst precursor for the carbonylation reaction. THF/MeOH solvent mixture used for the dehydrobromination reaction was also a good solvent for the carbonylation reaction under 10 atm of carbon monoxide.

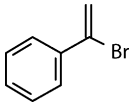
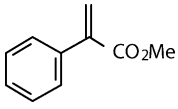
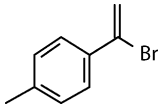
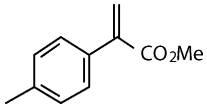
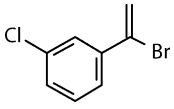
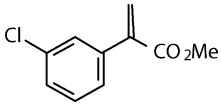
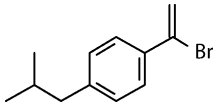
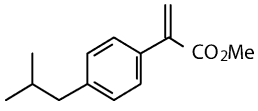
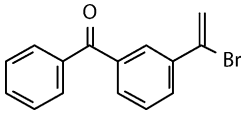
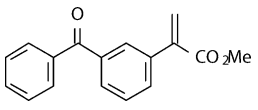
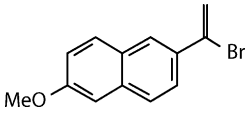
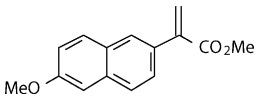
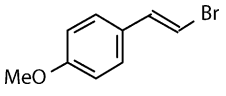
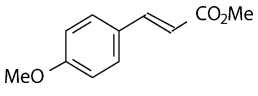
After the preliminary optimization a more detailed study in terms of base, palladium loading, temperature and time was performed (Table 2). The best base for the dehydrobromination step (K_2CO_3) gave poor results in terms of selectivity and yield (Table 2, entry 8). NaOAc (Table 2, entry 6) and *i*Pr_2EtN (Table 2, entry 10) gave the highest yields, but lower loadings of catalysts can be used in the presence of *i*Pr_2EtN (Table 2, entries 12–21). By increasing the reaction temperature to 100 °C reaction time can be shortened to 3.5 h. Therefore, the carbonylation of α-bromostyrene could be performed even with 0.5 mol catalyst loading in 95% isolated yield after 3.5 h (Table 2, entry 20).

Table 2
Pd-catalyzed carbonylation of α-bromostyrene[a]

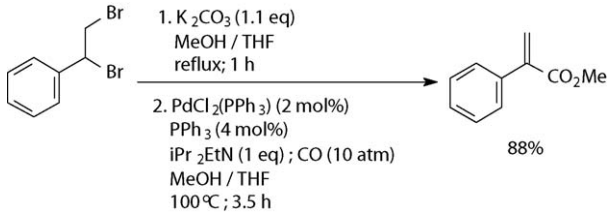
Entry	$PdCl_2(PPh_3)_2$ (mol%)	Base	Temperature (°C)	Time (h)	Conv. (%)	Yield[b] (%)
1	4	Et_3N	70	18	100	75
2	4	iPr_2NH	70	20	94	89
3	4	Et_2NH	70	20	100	55
4	4	$tBuNH_2$	70	18	100	64
5	4	$nBuNH_2$	70	20	100	56
6	4	iPr_2EtN	70	18	100	95
7	4	Me_2BnN	70	18	97	76
8	4	K_2CO_3	70	18	100	23
9	4	K_3PO_4	70	18	100	60
10	4	NaOAc	70	18	100	90
11	4	iPr_2EtN	70	3.5	40	30
12	4	iPr_2EtN	100	3.5	100	95
13	4	NaOAc	100	3.5	100	93
14	2	iPr_2EtN	100	3.5	100	95
15	2	NaOAc	100	3.5	100	86
16[c]	2	iPr_2EtN	100	3.5	100	96
17[c]	2	NaOAc	100	3.5	100	83
18[c]	1	iPr_2EtN	100	3.5	100	94
19[c]	1	NaOAc	100	3.5	63	50
20[c]	0.5	iPr_2EtN	100	3.5	100	95
21[c]	0.1	iPr_2EtN	100	3.5	39	38

[a] Reaction conditions: α-bromostyrene (1 mmol) in MeOH (6 ml)/THF (6 ml), base (1 mmol), $PdCl_2(PPh_3)_2/PPh_3$ ratio = 2, CO (10 atm), 70 °C.
[b] GC yields.
[c] Reaction conditions: α-bromostyrene (4 mmol) in MeOH (8 ml)/THF (8 mL), base (4 mmol), $PdCl_2(PPh_3)_2/PPh_3$ ratio = 2, CO (10 atm), 70 °C.

Table 3
Pd-catalyzed carbonylation of α-arylvinyl bromides[a]

Entry	Substrate	Product	Yield[b] (%)	α:β
1			95	95:5
2			92	92:8
3			88	97:3
4			94	91:9
5			90	99:1
6			90	100
7			92	100

[a] Reaction conditions: α-arylvinyl bromides (4 mmol), iPr_2EtN (4 mmol), $PdCl_2(PPh_3)_2$ (0.5 mol% for entry 1; 2 mol% for entries 2–7), $PdCl_2(PPh_3)_2/PPh_3$ ratio = 2, methanol (8 ml), THF (8 ml), 10 atm of CO, 100 °C, 3.5 h.
[b] Isolated yields.



Scheme 2. One-pot synthesis of methyl 2-phenylacrylate.

The best conditions so far developed for the α-bromostyrene were extended to a variety of vinyl bromides (Table 3), including the precursors for the Ibuprofen™, Ketoprofen™ and Naproxen™ (Table 3, entries 4, 5 and 6, respectively). Under these conditions, the reaction proceeds very smoothly and the esters were isolated in high yields. Furthermore the carbonylation reaction occurs without any isomerization. In fact, the regioselectivities are maintained in the carbonylation reaction since they were the same as those observed for the first dehydrobromination step (see Table 1). These results suggest that both the β- and α-positions can be carbonylated. Therefore, the carbonylation of *trans*-β-bromostyrene furnished selectively methyl *trans-p*-methoxycinnamate in 92% isolated yield (Table 3, entry 7).

Although we have not found a common base for these reactions, both occur in a basic media in a common solvent media (THF/MeOH) making a one-pot protocol possible (Scheme 2). Therefore, in a 100 ml-stainless steel autoclave were placed (1,2-dibromethyl)benzene, K_2CO_3, THF and methanol, and the mixture was stirred at 100 °C for 1 h. After cooling the reaction mixture, the reagents for the carbonylation were added [$PdCl_2(PPh_3)$, PPh_3, *i*$PrEt_2N$, THF, MeOH], and the mixture was stirred at 100 °C for 3.5 h under an atmosphere of carbon monoxide (10 atm). After work-up, methyl 2-phenylacrylate was obtained in 66% yield. It is worthwhile to mention that optimized reaction conditions for the dehydrobromination employs two equivalents of K_2CO_3 and this base gave poor results in the carbonylation reaction. Therefore, we could improve the yield by using only a slight excess of this base (1.1 eq) affording the methyl ester in 88% yield in a simple one-pot protocol.

4. Conclusions

In summary, we have found that 2-arylacrylic esters can be obtained via a three-step sequence (bromination/dehydrobromination/carbonylation) starting from styrene derivatives. (1,2-Dibromomoethyl)arenes, obtained from the bromination of styrene derivatives were regioselectivley converted to the α-arylvinyl bromides in very good yields. The vinyl bromides underwent palladium-catalyzed carbonylation under mild conditions. This method allows the selective preparation of a variety of 2-arylacrylic esters, known as precursors for non-steroidal anti-inflammatory drugs. Furthermore, we have demonstrated that the deydrobromination/carbonylation sequence can be carried out in a one-pot procedure.

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