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Microwave-assisted arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene with arylboronic acids

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Abstract—The palladium-catalyzed arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene with arylboronic acids was studied under controlled microwave irradiation conditions. A variety of different catalysts, bases, and solvents were explored in order to achieve optimum yields in the shortest possible reaction times. The best isolated yields were obtained using $Pd_2(dba)_3 \cdot CHCl_3/PPh_3$ as the catalytic system, potassium phosphate monohydrate as the base, and toluene/H_2O as a solvent system. Microwave irradiation using 5 mol % of the palladium catalyst for 90 s (max. temp 170 °C) generally afforded the cross-coupling products in good to excellent yields.

1. Introduction

Organoboron compounds are well known as versatile reagents in organic synthesis.[1–7] In this context, a significant number of transition metal-catalyzed carbon–carbon bond forming reactions employing arylboronic acids (Suzuki-coupling,[1] conjugate addition to enones,[2] addition to aldehydes,[3] allylic substitution,[4] and the cross-coupling reaction with alkenes or acid chlorides[5,6]) have been described and reviewed recently.[7]

In particular, the allylic substitution reaction is a powerful method used to construct new carbon–carbon bonds. Excellent selectivities have been obtained using palladium catalysts in combination with a range of chiral ligands.[8] Palladium(0) complexes have also been shown to catalyze a wide variety of synthetically useful substitutions of allylic substrates with carbon nucleophiles.[9] Rather surprisingly, however, only a few reports on the use of boronic acids as nucleophiles in palladium-catalyzed allylic substitution reactions have been published.[10–15] For example, the coupling of arylboronic acids with allyl bromides in the presence of a base in refluxing benzene has been reported.[10,11] Along similar lines, Hayashi et al.[12] described the reaction of phenylboronic acid with allylic acetates under basic conditions in water at room temperature using a resin-supported palladium catalyst. More recently, Balme and co-workers[13] achieved good results for the arylation of cinnamyl acetates with phenylboronic acid in methanol using dichlorobis(tri-2-furylphosphane) as the catalyst and potassium fluoride as the base. In related work, high conversions were achieved by Najera and co-workers in the aqueous cross-coupling of phenylboronic acid with allylic acetates using $PdCl_2$ complexes with amides of di-(2-pyridyl)methylamine as the catalyst in the presence of a phase-transfer catalyst.[15]

We herewith report an experimentally very simple and fast protocol for the palladium-catalyzed arylation of allylic acetates with a range of arylboronic acids. The new protocol makes use of controlled microwave heating,[16] uses a commercially available and inexpensive palladium source, and provides good product yields in extremely short reaction times (90 s).

2. Results and discussion

At the beginning of our work we decided to examine the effect of the catalyst system (Pd source and base) on the course of the allylic substitution reaction of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene **1** with 4-methoxyphenylboronic acid **2** (Scheme 1, Table 1).

The microwave-assisted arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene (**1**) with 4-methoxyphenylboronic acid

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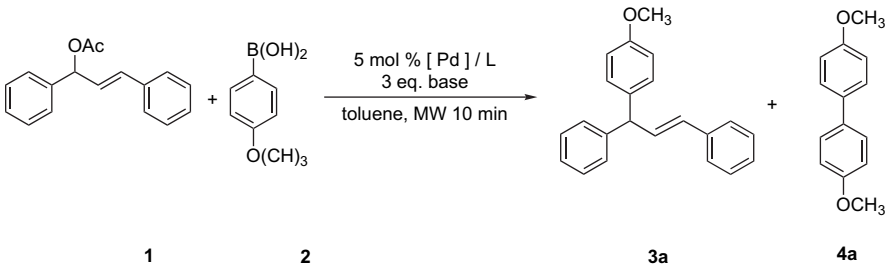


Scheme 1.

(**2a**) in toluene using $Pd(PPh)_3Cl_2/PPh_3$ as the catalytic system and K_3PO_4 as the base provided 52% of the desired product **3a** along with 35% of the homocoupling product of 4-methoxyphenylboronic acid, i.e., biphenyl **4a** (entry 1). Similar results were obtained when $Pd(OAc)_2/PPh_3$ was used as the catalytic system. The product of arylation was isolated in 58% yield and the homocoupling product was obtained in 18% yield (entry 2). The best yield (72%) of the desired arylation product **3a** was obtained using $Pd(PPh_3)_4/PPh_3$ as the catalyst. However, apart from the desired product **3a**, 26% of the homocoupling product **4a** were also isolated. Gratifyingly, a better selectivity was achieved using the $Pd_2(dba)_3\cdot CHCl_3/PPh_3$ as catalyst system. Here, the isolated yield for the cross-coupling product **3a** was 70%, while the homocoupling product **4a** was isolated in only 4% yield (entry 4). Using $Pd_2(dba)_3\cdot CHCl_3$ in combination with *o*-(di-*tert*-butylphosphino)biphenyl (2-DTBPB) decreased the yield of cross-coupling product **3a** to 21% (entry 5).

Table 1. Effect of the catalytic system (Pd/L=1:2) on the microwave-assisted arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene (**1**) with 4-methoxyphenylboronic acid (**2**) in toluene[a]

Entry	[Pd] source	Ligand	Base (3 equiv)	Yield of **3a**[b] (%)	Yield of **4a**[b] (%)
1	$Pd(PPh_3)_2Cl_2$	PPh_3	K_3PO_4	52	35
2	$Pd(OAc)_2$	PPh_3	K_3PO_4	58	18
3	$Pd(PPh_3)_4$	PPh_3	K_3PO_4	72	26
4	$Pd_2(dba)_3\cdot CHCl_3$	PPh_3	K_3PO_4	70	4
5	$Pd_2(dba)_3\cdot CHCl_3$	(2-DTBPB)[c]	K_3PO_4	21	4
6	$Pd_2(dba)_3\cdot CHCl_3$	(2-DTBPB)[c]	CsF	42	12
7	$Pd_2(dba)_3\cdot CHCl_3$	PPh_3	CsF	5	25

[a] Multimode microwave irradiation (Milestone Lavis 1100 reactor), 5 mol % of catalyst, 10 mol % of ligand, 1.42 equiv of boronic acid, 3 equiv of base, reaction time 10 min, max. temp 98 °C.
[b] Isolated yields of pure products after flash chromatography.
[c] 2-DTBPB=*o*-(di-*tert*-butylphosphino)biphenyl.

The same catalytic system in combination with CsF as the base gave similar low yields of the desired arylated product (entry 6). In addition, the use of $Pd_2(dba)_3\cdot CHCl_3/PPh_3$ in combination with CsF yielded unacceptable low product yields (entry 7).

Control experiments on the arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene (**1**) with 4-methoxyphenylboronic acid (**2a**) performed by conventional heating (98 °C, oil bath, 10 min) mimicking the microwave conditions reported in Table 1 (entry 4) provided significantly lower conversions. All the above mentioned microwave reactions were carried out under open vessel conditions in a multimode microwave Milestone Lavis 1100 reactor. Because of the low energy density in a multimode system[16,17] and the fact that toluene is a weak microwave absorber it was not possible to achieve a higher reaction temperature than 98 °C within the selected 10 min timeframe of the experiment. It was therefore of interest to see whether better results could be achieved in a closed vessel monomode system that allows superheating the reaction mixture to temperatures far above the boiling point of the solvent (110 °C).[16,17] Therefore, additional experiments were carried out in monomode reactor with sealed vessel capabilities (Table 2).

Interestingly, we found that the conversions in the palladium-catalyzed arylation (Scheme 1) were dependent on the quality of the potassium phosphate base, which is in accord with recently published data by Wawrzyniak and Heinicke.[18] In our hands, best results were typically with potassium phosphate monohydrate as the base in combination with toluene/H_2O mixture as the solvent. Regular toluene (technical grade), or toluene dried via azeotropic distillation gave comparatively lower yields (Table 2). Ultimately, the combination of a 24:1 toluene/water mixture and 3 equiv of commercially available (Aldrich) potassium

Table 2. Effect of temperature, base, and water on the arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene (**1**) with 4-methoxyphenylboronic acid (**2**) in toluene (Scheme 1)[a]

Entry	Toluene[b]	Base (3 equiv)	MW conditions[c]	Max. temp (°C)	Time (min)	Yield of **3a**[d] (%)
1	Moist	K_3PO_4	Open vessel	98	10	25
2	Moist	$K_3PO_4\cdot nH_2O$	Open vessel	98	10	70
3	Dry	$K_3PO_4\cdot nH_2O$	Sealed vessel	98	10	35
4	Moist	$K_3PO_4\cdot nH_2O$	Sealed vessel	98	10	65
5	Moist	$K_3PO_4\cdot H_2O$	Sealed vessel	130	10	47
6	Moist	$K_3PO_4\cdot H_2O$	Sealed vessel	170	5	61
7	With H_2O	$K_3PO_4\cdot H_2O$	Sealed vessel	170	1.5	72

[a] Microwave irradiation, 5 mol % $Pd_2(dba)_3\cdot CHCl_3$ as catalyst, 10 mol % of PPh_3 as ligand, 1.42 equiv of boronic acid, 3 equiv of base.
[b] Moist: technical grade; dry: dried via azeotropic distillation; with water: toluene/water=24:1.
[c] Open vessel: Milestone Lavis 1100 multimode reactor (max. power: 400 W); sealed vessel: Biotage Initiator single mode reactor (max. power: 300 W).
[d] Isolated yield of pure product after flash chromatography.

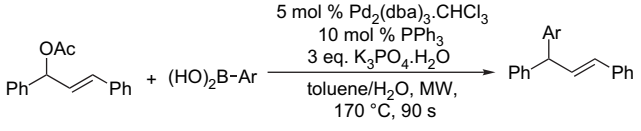
phosphate monohydrate provided the best and most reproducible results. Within only 90 s of total microwave irradiation time (max. temp 170 °C) a 72% isolated product yield of **3a** could be obtained (Table 2, entry 7). Under these conditions, only small amounts (4%) of the undesired homocoupling product **4a** could be detected. Reducing the catalyst loading to 2 mol % led to a lower isolated product yield (35%) after 90 s of irradiation.

Also of interest is the direct comparison of the results between open vessel and sealed vessel microwave irradiation. At the same measured reaction temperature of 98 °C and otherwise identical reaction conditions (moist toluene, 10 min reaction time), the outcome of the reaction is—within experimental error—identical (entry 2 versus entry 4).

Having optimized conditions for the rapid palladium-catalyzed cross-coupling of allylic acetates with phenylboronic acid in hand, we next wanted to test the applicability of this protocol and our catalytic system $Pd_2(dba)_3 \cdot CHCl_3$/PPh_3 for the arylation of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene **1** with a series of 10 different arylboronic acids (**2a**–**j**). The results are summarized in Table 3.

Without further optimization of the reaction conditions, all 10 arylboronic acids tested provided the anticipated cross-coupled products **3a**–**j** in moderate to excellent yields, ranging from 45 to 87%. Even sterically hindered boronic acids furnished the arylated products in acceptable yields (see entries 5 and 8). Also, both electron rich and electron poor (compare entries 4 and 6) boronic acids were equally suited as cross-coupling partners for the allylic acetate **1**.

Table 3. Palladium-catalyzed cross-coupling of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene with (**1**) different arylboronic acids[a]



Entry	Arylboronic acid	Product	Yield[b] (%)
1	Ar=4-(MeO)C_6H_4	**3a**	72
2	Ar=C_6H_5	**3b**	70
3	Ar=4-(Me)C_6H_4	**3c**	87
4	Ar=3-(Me)C_6H_4	**3d**	45
5	Ar=2-(Me)C_6H_4	**3e**	71
6	Ar=4-(CF_3)C_6H_4	**3f**	72
7	Ar=3-(CF_3)C_6H_4	**3g**	75
8	Ar=2-(CF_3)C_6H_4	**3h**	65
9	Ar=4-(Cl)C_6H_4	**3i**	57
10	Ar=1-naphthyl	**3j**	55

[a] 5 mol % $Pd_2(dba)_3 \cdot CHCl_3$, 10 mol % PPh_3, 3 equiv $K_3PO_4 \cdot H_2O$, toluene/H_2O (24:1), sealed vessel microwave irradiation, 170 °C, 1.5 min.
[b] Isolated yield of pure product after flash chromatography on silica gel using hexane/*t*-BuOMe (10:1) as the eluant.

3. Conclusion

In summary, we have discovered that the arylation of allylic acetates such as *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene **1** with arylboronic acids can be carried out very efficiently under controlled microwave heating conditions. Employing the commercially available and inexpensive catalytic system $Pd_2(dba)_3 \cdot CHCl_3$/PPh_3 in combination with potassium phosphate monohydrate as a base and toluene/water mixture as solvent, a range of boronic acids can be cross-coupled in high yields with the allylic acetate substrate. The very short reaction times of less than 2 min may allow the exploration of a variety of different structural classes based on this transformation.

4. Experimental

4.1. General

Microwave experiments were carried out either in the Milestone Lavis 1100 reactor (max. power setting 400 W) and in the Biotage Initiator reactor (max. power 300 W). All reactions were performed in an argon atmosphere. Products were analyzed by ^{1}H NMR, ^{13}C NMR, and GC–MS spectroscopy. The ^{1}H NMR and ^{13}C NMR spectra were measured at 300 MHz on a Varian Gemini spectrometer in $CDCl_3$ with tetramethylsilane as an internal standard. GC–MS measurements were performed on a gas chromatograph Trace GC 2000 Series Thermoquest CE Instruments with a flame ionization detector and a Voyager GC–MS Thermoquest Finnigan in SCAN-mode. Melting points were determined on a Kofler hot stage. Products **3a**–**c**, and **4a** were identified by comparison to authentic samples of the products.[19–21]

4.2. Reactions under microwave irradiation

A 5 mol % quantity of $Pd_2(dba)_3 \cdot CHCl_3$/PPh_3 catalyst and 10 mol % of PPh_3 were added to a solution of *rac*-(*E*)-3-acetoxy-1,3-diphenylprop-1-ene (**1**) (0.165 g, 0.7 mmol), the corresponding arylboronic acid (1 mmol, Table 3) and $K_3PO_4 \cdot H_2O$ (3 mmol) in toluene/water (24:1, v/v, 5 ml). The reaction mixture was heated by sealed vessel microwave irradiation in a Biotage Initiator microwave system for 1.5 min (max. temp 170 °C). The reaction mixture was then cooled and quenched with water (20 ml), and extracted into diethyl ether (15 ml). The organic layer was washed with brine, dried over anhydrous magnesium sulfate, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexane/*t*-BuOMe (10:1) as the eluant. The products **3a**–**c**, **4a** were found to be identical (^{1}H NMR data) with the data described in literature.[19–21] All new compounds (the products **3d**–**j**) were characterized by ^{1}H NMR and ^{13}C NMR spectroscopy, MS spectroscopy, and elemental analyses.

4.2.1. *rac*-(*E*)-1,3-Diphenyl-3-(3-tolyl)prop-1-ene [3d]. Colorless oil. ^{1}H NMR ($CDCl_3$, 300 Hz): δ 4.85 (d, J=7.8 Hz, 1H), 6.34 (d, J=15.6 Hz, 1H), 6.64 (dd, J=15.6, 7.8 Hz, 1H), 7.04 (m, 3H), 7.17–7.39 (m, 11H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 143.9, 143.7, 138.3, 137.5, 132.9, 131.5, 129.6, 128.9, 128.7, 128.6, 128.5, 127.5, 127.4, 126.6, 126.5, 125.9, 54.4, 21.7. GC–MS: 284 (100, M^+), 269 (43), 206 (50), 192 (70), 178 (40), 165 (32), 115 (29), 91 (21). Anal. Calcd for $C_{22}H_{20}$ (284.38): C, 92.91; H, 7.09. Found: C, 92.59; H, 7.30.

4.2.2. *rac*-(*E*)-1,3-Diphenyl-3-(2-tolyl)prop-1-ene [3e]. Colorless oil. ^{1}H NMR ($CDCl_3$, 300 Hz): δ 5.07 (d,

J=7.2 Hz, 1H); 6.20 (dd, J=15.6, 1.5 Hz, 1H), 6.66 (dd, J=15.6 Hz, 7.2 Hz, 1H), 7.16–7.38 (m, 14H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 143.0, 141.7, 137.5, 136.6, 132.7, 131.5, 130.7, 129.1, 128.7, 128.6, 128.5, 127.4, 126.7, 126.5, 126.4, 126.2, 50.5, 20.0. GC–MS: 284 (78, M^+), 269 (100), 206 (25), 191 (61), 178 (50), 165 (29), 115 (43), 91 (32). Anal. Calcd for $C_{22}H_{20}$ (284.38): C, 92.91; H, 7.09. Found: C, 93.03; H, 7.01.

4.2.3. *rac*-(*E*)-1,3-Diphenyl-3-[(4-trifluoromethyl)phenyl]prop-1-ene [3f]. Colorless oil. ^{1}H NMR ($CDCl_3$, 300 Hz): δ 4.94 (d, J=7.5 Hz, 1H), 6.35 (d, J=15.9 Hz, 1H), 6.64 (dd, J=15.9, 7.5 Hz, 1H), 7.21–7.49 (m, 12H), 7.57 (d, J=8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 147.8 (q, J=1.2 Hz), 142.0, 137.1, 132.4, 131.7, 129.2, 128.9, 128.8 (q, J=8.1 Hz), 128.8, 127.9, 127.0, 126.6, 125.6 (q, J=3.8 Hz), 124.5 (q, J=272 Hz), 54.2. GC–MS: 338 (90, M^+), 260 (100), 191 (50), 178 (45), 165 (33), 91 (27).

4.2.4. *rac*-(*E*)-1,3-Diphenyl-3-[(3-trifluoromethyl)phenyl]prop-1-ene [3g]. White crystals, mp 48–49 °C (from *n*-hexane). ^{1}H NMR ($CDCl_3$, 300 Hz): δ 4.95 (d, J=7.5 Hz, 1H), 6.36 (d, J=15.6 Hz, 1H), 6.64 (dd, J=15.6, 7.5 Hz, 1H), 7.21–7.50 (m, 14H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 144.5, 142.6, 136.9, 132.2, 132.1 (q, J=1.3 Hz), 131.5, 130.8 (q, J=32 Hz), 128.9, 128.7, 128.6, 127.6, 126.8, 126.4, 125.3 (q, J=3.6 Hz), 123.4 (q, J=3.7 Hz), 124.2 (q, J=271 Hz), 54.0. GC–MS: 338 (88, M^+), 260 (100), 191 (35), 178 (33), 115 (25), 91 (19).

4.2.5. *rac*-(*E*)-1,3-Diphenyl-3-[(2-trifluoromethyl)phenyl]prop-1-ene [3h]. White crystals, mp 49–51 °C (from *n*-hexane). ^{1}H NMR ($CDCl_3$, 300 Hz): δ 5.37 (d, J=7.5 Hz, 1H), 6.29 (d, J=15.9 Hz, 1H), 6.62 (dd, J=15.9, 7.5 Hz, 1H), 7.22–7.38 (m, 12H), 7.50 (t, J=15 Hz, 1H); 7.69 (d, J=7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 143.1, 142.3 (q, J=1.5 Hz), 137.0, 132.2, 131.9 (q, J=1.1 Hz), 131.7, 131.1, 128.6, 128.4, 127.5, 126.5, 126.4, 126.3, 125.9 (q, J=6.7 Hz), 124.5 (q, J=272 Hz), 48.7. GC–MS: 338 (100, M^+), 269 (22), 260 (80), 191 (33), 179 (30), 115 (26), 91 (22).

4.2.6. *rac*-(*E*)-1,3-Diphenyl-3-(4-chlorophenyl)prop-1-ene [3i]. White crystals, mp 62–63.5 °C (from *n*-hexane). ^{1}H NMR ($CDCl_3$, 300 Hz): δ 4.86 (d, J=7.2 Hz, 1H), 6.33 (d, J=15.9 Hz, 1H), 6.62 (dd, J=15.9, 7.2 Hz, 1H); 7.15–7.38 (m, 14H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 142.9, 141.9, 136.9, 132.2, 131.9, 131.7, 129.9, 128.5, 127.4, 126.6, 126.2. GC–MS: 304 (71, M^+), 269 (75), 226 (45), 191 (100), 178 (46), 165 (46), 115 (25), 91 (25). Anal. Calcd for $C_{21}H_{17}Cl$ (304.81): C, 82.74; H, 5.62; Cl, 11.63. Found: C, 82.94; H, 5.89; Cl, 10.93.

4.2.7. *rac*-(*E*)-1,3-Diphenyl-3-(1-naphthyl)prop-1-ene [3j]. Colorless oil. ^{1}H NMR ($CDCl_3$, 300 Hz): δ 5.66 (d, J= 7.2 Hz, 1H), 6.26 (dd, J=15.9, 1.2 Hz, 1H), 6.81 (dd, J= 15.9, 7.2 Hz, 1H), 7.20–7.45 (m, 14H), 7.78 (d, J=7.8 Hz, 1H), 7.85–7.88 (m, 1H), 8.03–8.06 (m, 1H). ^{13}C NMR ($CDCl_3$, 75 Hz): δ 143.3, 139.3, 137.5, 134.2, 132.8, 132.0, 131.9, 129.1, 128.9, 128.7, 127.6, 127.5, 126.7, 126.5, 126.2, 125.7, 125.6, 124.3, 50.3. GC–MS: 320 (100, M^+), 241 (42), 229 (99), 215 (36), 165 (30), 115 (25), 91 (22). Anal. Calcd for $C_{25}H_{20}$ (320.43): C, 93.71; H, 6.29. Found: C, 94.05; H, 6.39.

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