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ORIGINAL PAPER

Palladium-catalyzed arylations in 4-pyrone systems: 2,6-diaryl-3,5-dibromo-4-pyrones and kojic acid

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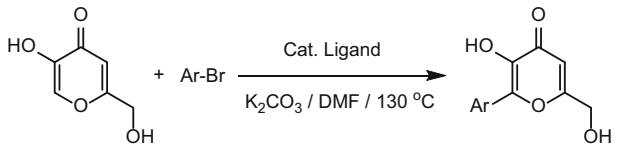
Abstract Palladium-catalyzed cross-coupling reactions of 2,6-diaryl-3,5-dibromo-4-pyrones with imidazole and benzimidazole in the presence of *N*,*N*′-dibutylbenzimidazol-2-ylidene as ligand resulted in the formation of the monosubstituted C- and N-arylated products. Kojic acid was also treated with some aryl halides at the same conditions, to afford C-6 arylated derivatives.

Graphical abstract



Keywords Palladium-catalyzed arylations · C–H activation · 2,6-Diaryl-4-pyrones · Kojic acid · Cross couplings

Introduction

Palladium-catalyzed cross-coupling reactions have arguably become among the most powerful and versatile tools for carbon–carbon bond formation [1–3]. Development of these processes by activation of aromatic C–H bond and enabling direct arylation reactions of aromatics and heteroaromatics have greatly facilitated the synthesis of more complex molecular systems from simpler ones [4–9]. Palladium complexes of N-heterocyclic carbenes (NHC) are potential catalytic species for the most challenging coupling reactions [10–12]. In this area, the catalysts containing benzimidazole ligand have been also well defined [13, 14]. Molecules bearing aryl-heteroaryl bonds which are accessible through above methodologies play a pivotal role in many of pharmaceuticals [15], optoelectronic materials [16], liquid crystals [17], and ligands for asymmetric catalysis [18]. 4-Pyrones and their natural and bioactive derivatives [19, 20] could be the central structural elements in such polyaromatic systems [21, 22]. Kojic acid (5-hydroxy-2-(hydroxymethyl)-4-pyrone) is a natural antioxidant and metabolic compound that is produced by several species of fungi. Its derivatives which are widely represented in various biologically relevant compounds have been the subject of several synthetic studies [23–25]. In connection with our studies directed toward the synthesis of various heterocyclic derivatives of 4-pyrones [26, 27], we planned the direct arylation of 4-pyrones to afford substituted molecules.

Due to the importance of C–H activation in the phenolic compounds [28], herein we report the palladium-catalyzed direct arylation reactions of kojic acid as a natural OH containing heterocycle. On the other hand, we have already reported the synthesis of 2,6-diaryl-3,5-dibromo-4-pyrones and their Pd catalyzed Heck reactions with alkyl acrylates [29]. Because of the presence of imidazole and benzimidazole nucleus in a large number of naturally occurring as well as biologically active molecules [30, 31], we decided to carry out the coupling reactions of these azoles with above dibromides to access new derivatives of 4-pyrones.

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Results and discussion

We started our experiments by the metal catalyzed reactions of kojic acid with aryl halides in the presence of a ligand and a base. Table 1 shows the results of treatment of bromobenzene with kojic acid at various conditions. Because this 4-pyrone derivative is likely to react via phenolic OH group (Ullman reaction) or C-6 carbon, we examined both Pd(II)- and Cu(I)-based reactions in the presence of a ligand as cocatalyst. As shown, the first reaction by employment of 2.5 mol% palladium chloride as catalyst, 5 mol% triphenylphosphine as ligand and K_2CO_3 in dry DMF, gave no notable product even after 24 h at 130 °C (entry 1). The use of *N*,*N*′-dibutylbenzimidazolium bromide (DBBIB) instead of PPh_3 resulted in the formation of only C-phenylated product **1a** after 5 h in 68 % yield (entry 2). Application of palladium acetate as catalyst under otherwise identical conditions produced the higher yield (entry 3). Furthermore, heating a mixture of reactants in an aqueous medium (H_2O: 1,4-dioxane 1:1) at reflux produced only trace amount of **1a** (entry 4). Treatment of kojic acid with 4-bromotoluene, 4-bromochlorobenzene and 1-bromonaphthalene under the optimized conditions (Table 1, entry 3) resulted in the related C-6 arylated derivatives **1b–1d** (Fig. 1). However, 4-bromonitrobenzene, 4-bromoanisole, and 4-bromo-*N*,*N*-dimethylaniline gave only the homocoupling products of these arylbromides at the same conditions, and no cross-coupling derivatives of 4-pyrone were observed. We also tested copper promoted reactions of these substrates in the presence of two ligands. But none of them leaded to any product (Table 1, entries 5 and 6).

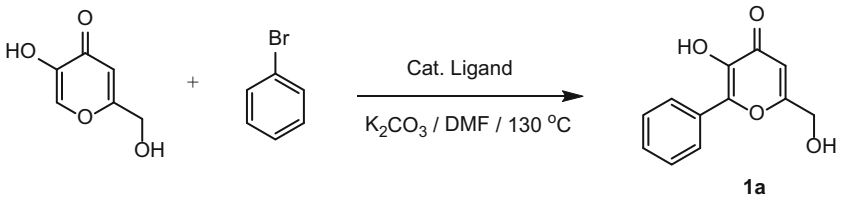
We then directed our efforts to the bromo-4-pyrone derivatives to evaluate their reactivity in direct reactions with NH containing heteroaryls. 3,5-Dibromo-2,6-diphenyl-4-pyrone (**2**), which was prepared by bromination of 2,6-diphenyl-4-pyrone with NBS/DMF [29], was chosen as halide partner in the catalytic reactions with imidazole. The results have been summarized in Table 2. As shown, treatment of dibromide **2** with 2 eq imidazole in the presence of Pd or Cu based catalytic systems gave the two monoarylated products **3a** and **3b**. In fact, the N–H group and C_2–H are the most active positions for the metal catalyzed arylation of imidazole and benzimidazole [32, 33]. Once again, the use of $Pd(OAc)_2$ with dibutylbenzimidazolium bromide as cocatalyst was more beneficial (entry 1). In all cases, the yields of two isomeric products were very similar and no disubstituted product was detected. Benzimidazole also reacted with dibromide **2** under the optimized conditions and the corresponding products **4a** and **4b** were obtained (Fig. 2). In a similar manner and under the same conditions, the substituted products **4c** and **4d** were synthesized by means of the reaction of 2,6-bis(*p*-tolyl)-3,5-dibromo-4-pyrone [29] with benzimidazole.

In conclusion, we synthesized some new coupling derivatives of 4-pyrones. Reaction of commercially available kojic acid with arylbromides in the presence of $Pd(OAc)_2$, *N*,*N*'-dibutylbenzimidazolium bromide and a base gave the C-6 arylated products. Coupling of imidazole and benzimidazole with 2,6-diaryl-3,5-dibromo-4-pyrones at the same conditions produced the C-3 monosubstituted products.

Experimental

Melting points were determined on a Electrothermal MEL-TEMP apparatus (model 1202D). FT-IR spectra were obtained with a Bruker Tensor 27 spectrometer. NMR

Table 1 Metal catalyzed reactions of kojic acid with bromobenzene

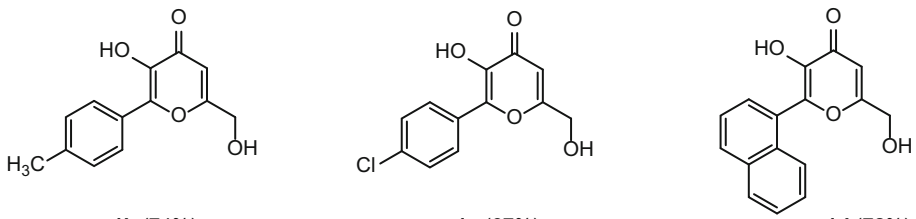


1a

Entry	Cat. (mol%)	Ligand (mol%)	Time/h	Solvent	Yield/%
1	$PdCl_2$ (2.5)	PPh_3 (5)	24	DMF	Trace
2	$PdCl_2$ (2.5)	DBBIB (5)	5	DMF	68
3	$Pd(OAc)_2$ (2.5)	DBBIB (5)	5	DMF	76
4	$Pd(OAc)_2$ (2.5)	DBBIB (5)	24	H_2O:dioxane (1:1)	Trace
5	CuI (5)	L-Proline (10)	24	DMF	–
6	CuI (5)	1,10-Phenantroline (10)	24	CH_3CN	–

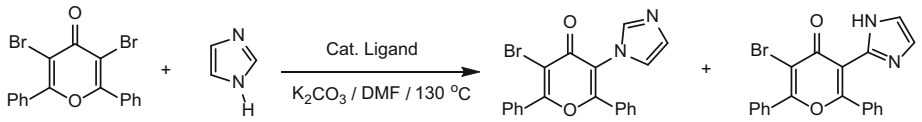
Conditions: kojic acid (2 mmol), aryl bromide (4 mmol), K_2CO_3 (2 mmol), 3 cm^3 solvent

Fig. 1 C-6 arylated derivatives of kojic acid



1b (74%) **1c** (67%) **1d** (72%)

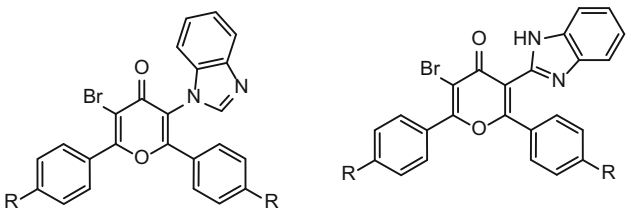
Table 2 Metal catalyzed reaction of imidazole with 3,5-dibromo-2,6-diphenyl-4-pyrone (**2**)



2 **3a** **3b**

Entry	Cat. (mol%)	Ligand (mol%)	Time/h	**3a**/%	**3b**/%
1	$Pd(OAc)_2$ (2.5)	DBBIB (5)	2	44	37
2	$PdCl_2$ (2.5)	DBBIB (5)	2	35	27
3	$Pd(OAc)_2$ (2.5)	PPh_3 (5)	24	32	33
4	CuI (5)	L-Proline (10)	24	30	25
5	CuI (5)	1,10-Phenantroline (10)	24	32	27

Conditions: dibromide **2** (2 mmol), imidazole (4 mmol), K_2CO_3 (4 mmol), 3 cm^3 DMF, 130 °C



4a: R=H (45%)
4c: R=CH_3 (42%)

4b: R=H (36%)
4d: R=CH_3 (39%)

Fig. 2 Coupling of benzimidazole with 2,6-diaryl-3,5-dibromo-4-pyrones

spectra were recorded with a Bruker Spectrospin Avance 400 spectrometer operating at 400 MHz (^{1}H NMR) and 100 MHz (^{13}C NMR), chemical shifts are given in parts per million (δ/ppm) relative to solvent peaks as standards ($CDCl_3$: 7.26 ppm (^{1}H), 76 ppm (^{13}C); DMSO-d_6: 2.50 ppm (^{1}H), 39.5 ppm (^{13}C)) or tetramethylsilane at 298 K. Elemental analysis was measured by Vario EL III apparatus (Elementar Co.). Preparative thin layer chromatographies (PLC) were done with prepared glass-backed plates (20 × 20 cm^2, 500 μ) using silica gel (Merck Kieselgel 60 $PF_{254+366}$).

General procedure for the synthesis of compounds 1a–1d

A mixture of 0.284 g kojic acid (2 mmol, 2 eq), arylbromide (4 mmol, 4 eq), 0.011 g palladium acetate (0.05 mmol), 0.031 g dibutylbenzimidazolium bromide (0.1 mmol, 0.1 eq), 0.276 g K_2CO_3 (2 mmol, 4 eq), and 3 cm^3 DMF was heated at 130 °C for 5 h. After cooling to 25 °C, 30 cm^3 water was added and the mixture was extracted with ethyl acetate (3 × 20 cm^3). The organic layers were combined, washed with 50 cm^3 water, the washed extracts were dried over Na_2SO_4, the dried extracts were filtered, and the filtrate was concentrated under vacuum. Purification of the product was done by preparative thin layer chromatography using acetone: *n*-hexane (1:3) to obtain products **1a**–**1d**.

3-Hydroxy-6-(hydroxymethyl)-2-phenyl-4H-pyran-4-one (**1a**, $C_{12}H_{10}O_4$)

Brown solid; yield 76 %; m.p.: 124–126 °C; FT-IR (KBr): $\bar{\nu}$ = 3300, 2923, 1647, 1615, 1572, 1215, 1108 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 4.43 (d, 2H, J = 4.1 Hz, benzylic CH_2), 5.76 (s, 1H, benzylic OH), 6.41 (s, 1H, pyrone H5), 7.44–7.51 (m, 3H, phenyl-H), 8.04 (d, 2H, J = 7.1 Hz, phenyl-H), 9.51 (s, 1H, pyrone-OH)

ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 59.6$ (CH_2-OH), 108.2, 126.8, 128.5, 129.3, 130.9, 142.3, 144.2, 167.6, 174.2 (pyrone C = O) ppm.

3-Hydroxy-6-(hydroxymethyl)-2-(4-methylphenyl)-4H-pyran-4-one (**1b**, $C_{13}H_{12}O_4$)
Brown solid; yield 74 %; m.p.: 142–144 °C; FT-IR (KBr): $\bar{\nu} = 3229, 2922, 1640, 1611, 1455, 1208, 1106, 797$ cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 2.35$ (s, 3H, CH_3), 4.42 (s, 2H, benzylic CH_2), 5.80 (s, 1H, benzylic OH), 6.39 (s, 1H, pyrone-H5), 7.31 (d, 2H, $J = 7.6$ Hz, phenyl-H), 7.93 (d, 2H, $J = 7.6$ Hz, phenyl-H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 21.1$, 59.8, 108.3, 126.9, 128.2, 129.2, 139.3, 142.0, 144.7, 167.6, 174.3 ppm.

2-(4-Chlorophenyl)-3-hydroxy-6-(hydroxymethyl)-4H-pyran-4-one (**1c**, $C_{12}H_9ClO_4$)
Brown solid; yield 67 %; m.p.: 173–175 °C; FT-IR (KBr): $\bar{\nu} = 3300, 2923, 1688, 1558, 1226, 1132$ cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 4.43$ (s, 2H, benzylic CH_2), 5.79 (s, 1H, benzylic OH), 6.41 (s, 1H, pyrone-H5), 7.60 (d, 2H, $J = 6.9$ Hz, phenyl-H), 8.06 (d, 2H, $J = 6.9$ Hz, phenyl-H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 59.3$, 111.2, 128.4, 130.2, 131.4, 135.6, 141.5, 146.5, 168.1, 173.2 ppm.

3-Hydroxy-6-(hydroxymethyl)-2-(naphthalene-1-yl)-4H-pyran-4-one (**1d**, $C_{16}H_{12}O_4$)
Brown solid; yield 72 %; m.p.: 158–160 °C; FT-IR (KBr): $\bar{\nu} = 3282, 3046, 2924, 1642, 1620, 1569, 1226, 1132$ cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 4.37$ (s, 2H, benzylic CH_2), 5.73 (s, 1H, benzylic OH), 6.51 (s, 1H, pyrone H5), 7.57–8.09 (m, 7H, naphthyl-H), 9.11 (s, 1H, pyrone-OH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 59.7$ (CH_2), 109.1, 125.2, 125.3, 126.3, 126.9, 127.9, 128.4, 128.6, 130.1, 130.3, 133.1, 142.9, 146.2, 168.1, 174.1 ppm.

General procedure for synthesis of compounds 3 and 4

A mixture of 2,6-diaryl-3,5-dibromo-4-pyrone (2 mmol, 2 eq), imidazole or benzimidazole (4 mmol, 4 eq), 0.011 g palladium acetate (0.05 mmol), 0.031 g dibutylbenzimidazolium bromide (0.1 mmol, 0.1 eq), 0.276 g K_2CO_3 (2 mmol, 4 eq), and 3 cm^3 DMF was heated at 130 °C for 2 h. After cooling, 30 cm^3 water was added and the mixture was extracted with ethyl acetate (3 × 20 cm^3). The organic layers were combined, washed with 50 cm^3 water, the washed extracts were dried over Na_2SO_4, the dried extracts were filtered, and the filtrate was dried over Na_2SO_4 and concentrated to dryness. The residue was purified by preparative thin layer chromatography using ethyl acetate: *n*-hexane (2:5) to obtain products **3** and **4**.

5-Bromo-2,6-diphenyl-3-(1H-imidazol-1-yl)-4H-pyran-4-one (**3a**, $C_{20}H_{13}BrN_2O_2$)
Yellow solid; yield 44 %; m.p.: 172–175 °C; FT-IR (KBr): $\bar{\nu} = 1685, 1468, 1263, 1017$ cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 7.23$ (d, 2H, $J = 8.7$ Hz, Ar–H), 7.46–7.53 (m, 4H, Ar–H), 7.56-7.63 (m, 4H, Ar–H), 7.94 (s, 1H, Im-H2), 8.25 (d, 2H, $J = 7.7$ Hz, Ar–H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 97.6, 119.4, 126.2, 126.6, 127.2, 127.8, 127.9, 128.9, 129.4, 129.6, 130.7, 131.0, 132.1, 137.3, 167.8, 177.9$ ppm.

5-Bromo-2,6-diphenyl-3-(1H-imidazol-2-yl)-4H-pyran-4-one (**3b**, $C_{20}H_{13}BrN_2O_2$)
Yellow solid; yield 37 %; m.p.: 198–200 °C; FT-IR (KBr): $\bar{\nu} = 3564, 1691, 1587, 1564, 1260, 1022$ cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 7.03$ (s, 1H, Im-H), 7.52–7.63 (m, 9H, Ar–H), 7.73 (s, 1H, Im-H), 8.21 (d, 2H, $J = 7.9$ Hz, Ar–H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 97.7, 119.7, 126.3, 127.2, 127.8, 128.0, 128.1, 129.4, 129.7, 130.7, 131.2, 132.1, 137.5, 167.9, 177.9$ ppm.

3-(1H-Benzimidazol-1-yl)-5-bromo-2,6-diphenyl-4H-pyran-4-one (**4a**, $C_{24}H_{15}BrN_2O_2$)
Yellow solid; yield 45 %; m.p.: 126 °C (decomp.), FT-IR (KBr): $\bar{\nu} = 3068, 1682, 1553, 1448, 1027$ cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 6.72$ (d, 1H, $J = 8.2$ Hz, Ar–H), 7.14–7.17 (m, 1H, Ar–H), 7.31–7.35 (m, 1H, Ar–H), 7.46–7.60 (m, 8H, Ar–H), 7.88 (d, 1H, $J = 8.1$ Hz, Ar–H), 8.13 (dd, 2H, $J = 7.4$, 1.1 Hz, Ar–H), 8.24 (s, 1H, benzimidazole-H2) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 98.2, 111.7, 119.8, 122.7, 123.2, 126.0, 127.1, 127.5, 127.7, 127.8, 127.9, 128.2, 129.7, 131.3, 132.1, 142.6, 143.0, 166.9, 178.7$ ppm.

3-(1H-Benzimidazol-2-yl)-5-bromo-2,6-diphenyl-4H-pyran-4-one (**4b**, $C_{24}H_{15}BrN_2O_2$)
Yellow solid; yield 36 %; m.p.: 212 °C (decomp.); FT-IR (KBr): $\bar{\nu} = 3429, 1681, 1551, 1448, 1262, 804, 686$ cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 6.74$ (d, 1H, $J = 8.0$ Hz, Ar–H), 7.11–7.15 (m, 1H, Ar–H), 7.29–7.31 (m, 1H, Ar–H), 7.49–7.67 (m, 8H, Ar–H), 7.88 (d, 1H, $J = 5.6$ Hz, benzimidazole-H), 8.11 (s, 1H, benzimidazole-H)), 8.27 (d, 2H, $J = 7.7$ Hz, Ar–H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 97.7, 110.5, 119.7, 122.2, 122.9, 125.3, 126.3, 127.2, 128.0, 128.2, 129.3, 130.1, 130.7, 132.1, 132.2, 138.2, 143.1, 143.3, 167.9, 177.9$ ppm.

3-(1H-Benzimidazol-1-yl)-2,6-bis(4-methylphenyl)-5-bromo-4H-pyran-4-one (**4c**, $C_{26}H_{19}BrN_2O_2$)
Yellow solid; yield 42 %; m.p.: 131 °C (decomp.); FT-IR (KBr): $\bar{\nu} = 3067, 2923, 1688, 1631, 1444, 1082$ cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 2.42$ (s, 3H, CH_3), 2.45 (s, 3H, CH_3), 6.77 (d, 1H, $J = 8.4$ Hz, Ar–H), 7.14–7.18 (m, 1H, Ar–H), 7.26–7.40 (m, 5H, Ar–H), 7.45 (d, 2H, $J = 8.0$ Hz, Ar–H), 7.88 (d, 1H, $J = 7.9$ Hz, Ar–H),

8.00 (d, 2H, $J = 8.1$ Hz, Ar–H), 8.20 (s, 1H, benzimidazol-H2) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 20.7$, 20.8, 110.6, 111.8, 119.7, 122.6, 123.1, 123.3, 124.9, 127.1, 127.3, 128.2, 128.7, 128.9, 129.3, 129.7, 142.1, 142.6, 143.1, 143.2, 166.9, 177.7 ppm.

3-(1H-Benzimidazol-2-yl)-2,6-bis(4-methylphenyl)-5-bromo-4H-pyran-4-one (**4d**, $C_{26}H_{19}BrN_2O_2$)
Yellow solid; yield 39 %; m.p.: 206 °C (decomp.); FT-IR (KBr): $\bar{\nu} = 3469$, 2923, 1687, 1579, 1088 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 2.45$ (s, 3H, CH_3), 2.48 (s, 3H, CH_3), 6.76 (d, 1H, $J = 7.9$ Hz, Ar–H), 7.11–7.18 (m, 1H, Ar–H), 7.26–7.31 (m, 4H, Ar–H), 7.38 (d, 2H, $J = 7.8$ Hz, Ar–H), 7.46 (d, 2H, $J = 7.6$ Hz, Ar–H), 7.92 (s, 1H, N–H), 8.16 (d, 2H, $J = 7.6$ Hz, Ar–H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 20.8$, 20.9, 96.9, 110.6, 119.6, 122.2, 122.9, 123.6, 127.1, 127.3, 128.5, 128.7, 128.9, 129.3, 129.7, 132.2, 138.2, 141.6, 143.2, 143.5, 167.9, 177.9 ppm.

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