Palladium-Catalyzed One-Pot Synthesis of 4-Aryl-1*H*-1,2,3-triazoles from *anti*-3-Aryl-2,3-dibromopropanoic Acids and Sodium Azide

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Abstract: 4-Aryl-1*H*-1,2,3-triazoles were synthesized from *anti*-3-aryl-2,3-dibromopropanoic acids and sodium azide by a one-pot method using *N*,*N*-dimethylformamide as solvent in the presence of tris(dibenzylideneacetone)dipalladium(0) [$Pd_2(dba)_3$] and Xantphos.

Key words: 1*H*-1,2,3-triazole, *anti*-3-aryl-2,3-dibromopropanoic acid, sodium azide, $Pd_2(dba)_3$, one-pot

1,2,3-Triazoles have found widespread applications in pharmaceuticals and agrochemicals.[1] The discovery[2] of copper(I)-catalyzed 1,3-dipolar cycloaddition of azides and terminal alkynes has further triggered the use of 1,2,3-triazoles in bioconjungation, drug discovery,[3] material science[4] and combinatorial chemistry.[5] In addition, a number of compounds containing 1,2,3-triazoles have shown a broad spectrum of biological activities; such compounds possess antibacterial,[6] herbicidal, fungicidal,[7] antiallergic,[8] and anti-HIV[9] properties.

The dipolar cycloaddition of terminal alkynes with organic azides, known as the Huisgen reaction, is arguably the most direct synthetic method for the generation of triazoles. The development of the copper(I)-catalyzed reaction between organic azides and terminal alkynes, which regioselectively provides 1,4-disubstituted 1,2,3-triazoles[2] under mild conditions, represented a definitive advance in triazole chemistry and has become the most practical and useful 'click' reaction.[10] The reaction is effective in the preparation of a wide variety of triazole-containing molecules;[11] however, it has a limitation in that inorganic azides are not good substrates. Consequently, 1*H*-1,2,3-triazoles, which also have a wide range of uses,[12] cannot be prepared directly using the click chemistry strategy, but instead require sequences involving deprotection steps and the employment of more elaborate azides.[13] Other routes to 1*H*-1,2,3-triazoles include dipolar cycloadditions between sodium azide and alkynes with an electron-withdrawing substituent,[1,14] the reaction of sodium azide with nitroalkenes,[15] and the rearrangement of propargyl azides.[16]

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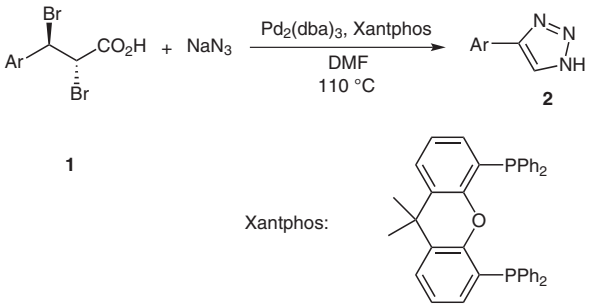
Recently, Barluenga and co-workers[17] reported a new methodology for the synthesis of 1*H*-triazoles from (*E*)-β-arylvinyl bromides and sodium azide catalyzed by a system involving tris(dibenzylideneacetone)dipalladium(0) [$Pd_2(dba)_3$] and Xantphos. This efficient transformation, afforded 4-aryl-1*H*-1,2,3-triazoles in near quantitative yields. Herein, we wish to report our results on the one-pot synthesis of 4-aryl-1*H*-1,2,3-triazoles from easily available *anti*-3-aryl-2,3-dibromopropanoic acids and sodium azide, catalyzed by $Pd_2(dba)_3$ and Xantphos (Scheme 1).



Scheme 1

We expected that (*Z*)-β-arylvinyl bromides, rather than the *E*-isomer, could be applicable as substrates for the palladium-catalyzed synthetic strategy developed by Barluenga.[17] Since (*Z*)-β-arylvinyl bromides could be readily obtained stereoselectively by simple debrominative decarboxylation of *anti*-3-aryl-2,3-dibromopropanoic acids in the presence of bases such as EtN_3, Na_2CO_3, $NaHCO_3$, K_2CO_3 and NaN_3,[18] we envisioned that *anti*-3-aryl-2,3-dibromopropanoic acids should constitute novel, simple starting substrates for the generation of 4-aryl-1*H*-1,2,3-triazoles upon treatment with sodium azide using the $Pd_2(dba)_3$–Xantphos system.

anti-3-Phenyl-2,3-dibromopropanoic acid (**1a**) was initially chosen as a model substrate using dioxane or DMSO as solvent and using the same amounts of $Pd_2(dba)_3$ and Xantphos as reported in the literature.[17] Much to our disappointment, however, treatment of a mixture of **1a** (1 mmol), sodium azide (4 mmol), $Pd_2(dba)_3$ (0.01 mmol, 1 mol%) and Xantphos (0.04 mmol, 4 mol%) in either dioxane or DMSO (3 mL) at 100 °C under a nitrogen atmosphere for 24 hours, only afforded **2a** in 23% (dioxane) and 20% (DMSO) yield (Table 1, entries 1 and 2).

Table 1 Transformation of **1a** into **2a** (Ar = Ph) under Various Conditions[a]

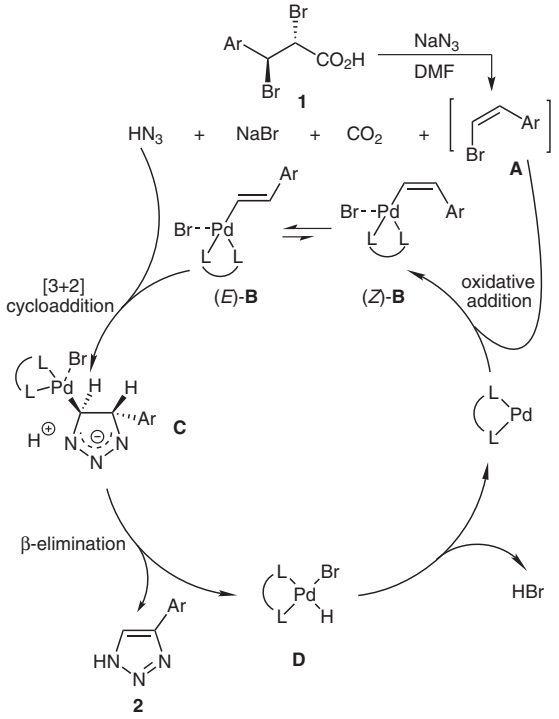
Entry	Solvent	Temp (°C)	Time (h)	Yield of **2a** (%)[b]
1	dioxane	100	24	23
2	DMSO	100	24	20
3	DMF	100	24	64
4	DMF	90	24	58
5	DMF	110	24	67
6	DMF	110	36	71
7	DMF	115	36	66
8	DMF	110	48	70
9[c]	DMF	110	36	49
10[d]	DMF	110	36	0
11[e]	DMF	110	36	trace

[a] Reaction conditions: **1a** (1 mmol), NaN_3 (4 mmol), $Pd_2(dba)_3$ (1 mol%), Xantphos (4 mol%), DMF (3 mL).
[b] Isolated yield based on **1a**.
[c] Use of NaN_3 (3 mmol).
[d] Without the use of $Pd_2(dba)_3$ and Xantphos.
[e] Use of Ph_3P (4 mol%) instead of Xantphos as the ligand.

Through an examination of the influence of solvents, it was found that DMF was the best solvent, producing **2a** in 64% yield (Table 1, entry 3). Encouraged by this result, we then investigated the effect of the temperature and reaction time; the results are also summarized in Table 1. It was pleasing to find that the desired product **2a** was obtained in a yield of 71% when the reaction was performed at 110 °C for 36 hours (Table 1, entry 6). Attempts to reduce the amount of sodium azide decreased the yield of **2a** dramatically (Table 1, entry 9). Test experiments carried out in the absence of $Pd_2(dba)_3$ and Xantphos resulted in recovered intermediate **A** (See Scheme 2 and Table 1, entry 10). Likewise, almost no conversion was observed when 4 mol% of triphenylphosphane was used as ligand instead of Xantphos (Table 1, entry 11). Hence, on the basis these experiments, it was concluded that the best conditions involved 1mol% of $Pd_2(dba)_3$ and 4 mol% Xantphos in the reaction system.

A range of *anti*-3-aryl-2,3-dibromopropanoic acids **1** gave, upon reaction under the optimized conditions, the corresponding 4-aryl-1*H*-1,2,3-triazoles **2** in good yields (Table 2). The starting *anti*-3-aryl-2,3-dibromopropanoic acids were easily prepared by bromination of the corresponding *trans*-α,β-unsaturated carboxylic acids. These results indicate that the present reaction can be used for the synthesis of 4-aryl-1*H*-1,2,3-triazoles carrying either an electron-donating group (Table 2, entries 2–4) or an electron-withdrawing group (Table 2, entries 5–8) in 58–71% yields. This method proved to be applicable to substrates bearing *ortho*-groups such as **1i** and **1j**, which gave the corresponding products **2i** and **2j** in 56% and 55% yields, respectively (Table 2, entries 9 and 10). The reaction of pyridyl-substituted substrate **1k** gave 1*H*-1,2,3-triazole **2k** in 61% yield (Table 2, entry 11). In the case of *anti*-3,3′-(1,4-phenylene)bis(2,3-dibromopropanoic acid) (**1l**), the di(1*H*-1,2,3-triazole) **2l** was isolated in a yield of 50% (Table 2, entry 12). *anti*-2,3-Dibromodecanoic acid **1m** was also tested as a model for 3-alkyl-2,3-dibromopropanoic acid. Unfortunately, almost no expected product was detected in the reaction system. The structure of 4-aryl-1*H*-1,2,3-triazoles **2a–l** were fully consistent with their ^1H NMR and MS data.[17,19–23]

The proposed pathways for the present debrominative decarboxylation and cycloaddition reactions are shown in Scheme 2. Debrominative decarboxylation of *anti*-3-aryl-2,3-dibromopropanoic acids **1** with sodium azide gives the (*Z*)-β-arylvinyl bromides **A**, which is immediately converted into the vinylpalladium complex (*Z*)-**B**. Subsequent [3+2] cycloaddition of the vinylpalladium complex (*E*)-**B**, a more stable complex formed from isomerization of (*Z*)-**B**, with HN_3, generated during the debrominative decarboxylation reaction of **1**, affords dihydrotriazolylpalladium complex **C**.[17] The latter complex would then undergo β-elimination to release the hydridopalladium complex **D** and the desired 4-aryl-1*H*-1,2,3-triazoles **2**. Finally, reductive elimination of hydridopalladium complex **D** releases HBr and regenerates the Pd(0) complex.



Scheme 2

Table 2 Synthesis of 4-Aryl-1*H*-1,2,3-triazoles **2** from *anti*-3-Aryl-2,3-dibromopropanoic Acids **1** and Sodium Azide[a]

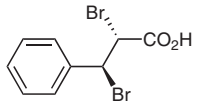
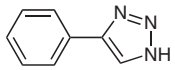
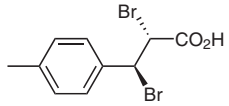
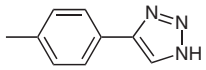
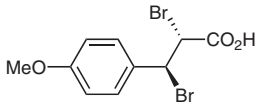
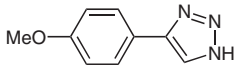
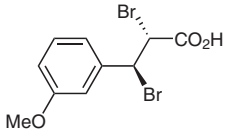
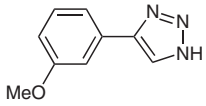
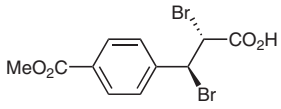
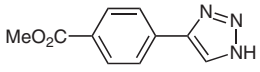
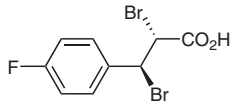
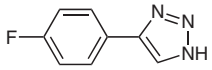
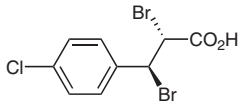
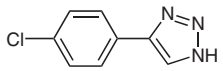
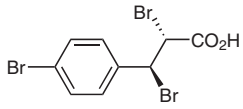
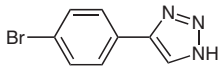
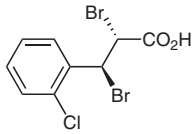
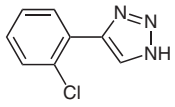
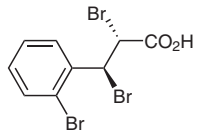
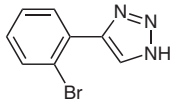
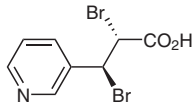
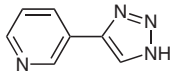
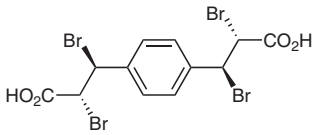
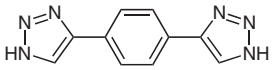
Entry	Substrate **1**	Product **2**	Yield of **2** (%)[b]
1	 **1a**	 **2a**	71
2	 **1b**	 **2b**	67
3	 **1c**	 **2c**	69
4	 **1d**	 **2d**	68
5	 **1e**	 **2e**	58
6	 **1f**	 **2f**	62
7	 **1g**	 **2g**	65
8	 **1h**	 **2h**	65
9	 **1i**	 **2i**	56
10	 **1j**	 **2j**	55
11	 **1k**	 **2k**	61

Table 2 Synthesis of 4-Aryl-1*H*-1,2,3-triazoles **2** from *anti*-3-Aryl-2,3-dibromopropanoic Acids **1** and Sodium Azide[a] (continued)

Entry	Substrate **1**	Product **2**	Yield of **2** (%)[b]
12	 **1l**	 **2l**	50

[a] Reaction conditions: **1a** (1 mmol), NaN_3 (4 mmol), $Pd_2(dba)_3$ (1 mol%), Xantphos (4 mol%), DMF (3 mL), 110 °C, 36 h.
[b] Isolated yield based on **1a**.

In summary, we have developed a facile one-pot method for the preparation of 4-aryl-1*H*-1,2,3-triazoles **2** from *anti*-3-aryl-2,3-dibromopropanoic acids **1** and sodium azide, catalyzed by $Pd_2(dba)_3$ and Xantphos, in moderate to high yields. The starting substrates **1** are readily available by bromination of the corresponding *trans*-α,β-unsaturated carboxylic acids. Further studies on mechanistic aspects of the present methodology are under investigation.

Melting points were recorded using a WRS-1B digital melting point apparatus and are uncorrected. ^{1}H NMR spectra were recorded using a Bruker AM-500 spectrometer in a mixture of DMSO-d_6 and $CDCl_3$ (1:1) with TMS as an internal standard. MS data were measured with a Varian-310 mass spectrometer. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with HuanghaiGF 254 silica gel coated plates. Column chromatography was carried out using 300–400 mesh silica gel at medium pressure. The synthesis of *anti*-3-aryl-2,3-dibromopropanoic acids was achieved as reported in our previous papers.[18m,p]

4-Aryl-1*H*-1,2,3-triazole (2); General Procedure

A reaction tube under nitrogen atmosphere was charged with *anti*-3-aryl-2,3-dibromopropanoic acid **1** (1 mmol), NaN_3 (260 mg, 4 mmol) and DMF (3 mL). After 30 min, Xantphos (23.1 mg, 0.04 mmol) and $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol) were added under nitrogen and the mixture was heated to 110 °C with stirring for 36 h. The mixture was allowed to cool to r.t. and then quenched with H_2O (10 mL). The mixture was extracted with EtOAc (3 × 10 mL) and the organic layer was washed with brine (5 mL). The combined organic layers were dried over Na_2SO_4, concentrated under reduced pressure and dried under high vacuum. Purification by column chromatography on silica gel (EtOAc–hexane) afforded the desired 4-aryl-1*H*-1,2,3-triazole **2**.

4-Phenyl-1*H*-1,2,3-triazole (2a)[17,19,21]

Yield: 103 mg (71%); white needles; mp 147.0–147.4 °C (EtOAc) (Lit.[19] 145.0–146.0 °C).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.32–7.44 (m, 3 H), 7.83–7.84 (m, 2 H), 8.23 (br s, 1 H).

MS (ESI): *m*/*z* = 145 [M$^+$].

4-*p*-Tolyl-1*H*-1,2,3-triazole (2b)[19,20,21]

Yield: 107 mg (67%); white needles; mp 157.0–157.3 °C (EtOAc) (Lit.[19] 150–152 °C, Lit.[20] 157–159 °C).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 2.36 (s, 3 H), 7.57 (d, *J* = 7.5 Hz, 2 H), 7.78 (d, *J* = 7.5 Hz, 2 H), 7.97 (br s, 1 H).

MS (ESI): *m*/*z* = 159 [M$^+$].

4-(4-Methoxyphenyl)-1*H*-1,2,3-triazole (2c)[17,21]

Yield: 121 mg (69%); white needles; mp 164.0–164.8 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 3.83 (s, 3 H), 6.96 (d, *J* = 7.3 Hz, 2 H), 7.74 (d, *J* = 7.3 Hz, 2 H), 7.91 (br s, 1 H).

MS (ESI): *m*/*z* = 175 [M$^+$].

4-(3-Methoxyphenyl)-1*H*-1,2,3-triazole (2d)[21]

Yield: 119 mg (68%); viscous oil.

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 3.32 (s, 3 H), 7.65–7.72 (m, 4 H), 8.18 (br s, 1 H).

MS (ESI): *m*/*z* = 175 [M$^+$].

Methyl 4-(1*H*-1,2,3-Triazol-4-yl)benzoate (2e)[21]

Yield: 118 mg (58%); white needles; mp 197.3–197.7 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 3.91 (s, 3 H), 7.87–8.39 (m, 5 H).

MS (ESI): *m*/*z* = 203 [M$^+$].

4-(4-Fluorophenyl)-1*H*-1,2,3-triazole (2f)[22]

Yield: 101 mg (62%); yellow needles; mp 172.1–172.5 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.14 (d, *J* = 8.5 Hz, 2 H), 7.83 (d, *J* = 8.5 Hz, 2 H), 7.94 (br s, 1 H).

MS (ESI): *m*/*z* = 163 [M$^+$].

4-(4-Chlorophenyl)-1*H*-1,2,3-triazole (2g)[21]

Yield: 117 mg (65%); white needles; mp 153.1–154.1 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.41 (d, *J* = 8.5 Hz, 2 H), 7.81 (d, *J* = 8.5 Hz, 2 H), 8.20 (br s, 1 H).

MS (ESI): *m*/*z* = 179 [M$^+$].

4-(4-Bromophenyl)-1*H*-1,2,3-triazole (2h)[21]

Yield: 146 mg (65%); white needles; mp 177.5–178.3 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.57 (d, *J* = 8.5 Hz, 2 H), 7.78 (d, *J* = 8.5 Hz, 2 H), 8.10 (br s, 1 H).

MS (ESI): *m*/*z* = 223 [M$^+$].

4-(2-Chlorophenyl)-1*H*-1,2,3-triazole (2i)[17]

Yield: 101 mg (56%); white needles; mp 105.1–105.7 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.31–7.60 (m, 4 H), 8.18 (br s, 1 H).

MS (ESI): *m*/*z* = 179 [M$^+$].

4-(2-Bromophenyl)-1*H*-1,2,3-triazole (2j)[17]

Yield: 123 mg (55%); white needles; mp 86.9–87.9 °C (EtOAc).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.26–7.99 (m, 4 H), 8.38 (br s, 1 H).

MS (ESI): *m*/*z* = 223 [M$^+$].

3-(1*H*-1,2,3-Triazol-4-yl)pyridine (2k)[21]
Yield: 89 mg (61%); white needles; mp 182.1–183.7 °C (MeOH).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.40–7.42 (m, 1 H), 8.07 (br s, 1 H), 8.16–8.18 (m, 1 H), 8.55 (br s, 1 H), 9.06 (s, 1 H).

MS (ESI): *m*/*z* = 146 [M$^+$].

1,4-Di(1*H*-1,2,3-triazol-4-yl)benzene (2l)[23]
Yield: 106 mg (50%); yellow needles; mp >300 °C (dec.) (MeOH).

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO-d_6): δ = 7.92 (s, 4 H), 8.06 (br s, 2 H).

MS (ESI): *m*/*z* = 212 [M$^+$].

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