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Reusable, Polystyrene-Resin-Supported, Palladium-Catalyzed, Atom-Efficient Cross-Coupling Reaction of Aryl Halides with Triarylbismuths

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A rapid, atom-efficient, cross-coupling reaction of triarylbismuths with aryl bromides or aryl iodides was reported, and the reaction involves the use of a catalytic amount of polystyrene-supported palladium in the presence of KF as base in DMSO at 105 °C in an open atmosphere. All three aryl groups of the triarylbismuths participated in the reaction and produced polyfunctional biaryls in excellent yields. The polymeric catalyst can be easily separated from the reaction mixture and reused more than 10 times without showing any obvious decrease in activity.

Introduction

The palladium-catalyzed cross-coupling reaction of electrophilic reagents with organometallic reagents is one of the most important synthetic methods for the construction of C–C bonds,[1] especially in the synthesis of biaryl compounds, which are important structural substructures in numerous natural products, polymers, agrochemicals, and pharmaceutical intermediates.[2] During the past decades, many palladium complexes have been used as homogeneous systems in these reactions, but most of them are expensive and air sensitive. Although these homogeneous catalysts always exhibit better activity and selectivity than heterogeneous ones,[3] heterogeneous catalysts have many advantages over homogeneous catalysts in industrial processes, such as recycling and lower cost. Thus, many polymer-supported[4] and inorganic solid-supported[5] (e.g., carbon, sol–gel, clays, and metal oxides) palladium catalysts have been reported. Among these supporters, cross-linked polystyrene resins,[6] which can bear different functional groups, provide a suitable supporter. Since the successful use of polystyrene-bound palladium catalysts in Suzuki cross-coupling reactions was reported,[7] cross-linked polystyrene supported palladium catalysts have been widely used in organic chemistry reactions.[8]

Up to the present, although various organometallic compounds such as organotin, organoboron, and organozinc compounds have been successfully utilized in cross-coupling reactions, organobismuth compounds have not been well studied.[9] Organobismuth compounds are normally nontoxic and potentially useful candidates for environmentally benign reagents, which is a major contemporary concern in the chemistry community.[10] Recently, some reactions involving organobismuth for C–C bond formation based on Pd-catalyzed cross-coupling have been reported.[11] Triarylbismuths, unlike organoboron, organotin, and other reagents, can react with three equivalents of an electrophilic reagent.[12] Among the limited successful examples for the construction of C–C bonds, long reaction times and harsh reaction conditions (such as under the protection of N_2 or under anhydrous and anaerobic conditions) were indispensable to accomplish the reaction. Furthermore, $Pd(PPh_3)_4$,[11b,12b] $Pd(OAc)_2$,[12a] or $PdCl_2$[12c] was used for the coupling reaction. These homogeneous catalysts are expensive or air sensitive, which limits their industrial applications and it is difficult to recycle and reuse these homogeneous catalysts due to their low stability toward oxidation.[13] These results stimulated us to pursue further research on these kinds of reactions. Herein, we report a highly efficient polystyrene-supported Pd^{II}-catalyzed protocol for an atom-efficient coupling of triarylbismuths with aryl iodides or aryl bromides in an open atmosphere. The methodology reported here has the following advantages: atom economical, short reaction time, broad substrate scope, simplicity of operation, as well as an easily recyclable catalyst with high efficiency.

Results and Discussion

The polystyrene-supported Pd^{II} catalyst with Pd/P ratio of 1:1 was prepared by a procedure described previously.[8a] In our initial efforts, we screened the cross-coupling reactivity of iodobenzene as a representative example by treating

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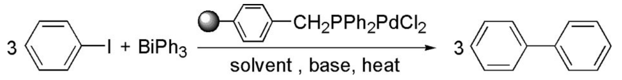
it with $BiPh_3$ and by using the polystyrene-supported Pd^{II} catalyst; we optimized the conditions in terms of solvent, base, and temperature. As shown in Table 1, solvents such as water, dichloromethane, acetonitrile, ethanol, PEG-400, ionic liquids, or dimethylformamide (DMF) provided poor or moderate yield of biphenyl (Table 1, Entries 1–8), and dimethyl sulfoxide (DMSO) was found to be the suitable solvent for the cross-coupling reaction (Table 1, Entry 9). In addition, the reactions carried out in the presence of different bases in DMSO revealed KF as a suitable base, giving 87% cross-coupling conversion (Table 1, Entry 9). Other bases including Na_2CO_3, K_2CO_3, K_3PO_4, $KHCO_3$, $NaHCO_3$, Et_3N, Al_2O_3, NaOH, and KOH provided poor conversion (Table 1, Entries 10–17). Further study on this reaction by varying the equivalents of KF revealed that 3.0 equivalents of base are necessary to obtain high yields of the cross-coupling products (Table 1, Entries 18–21). Furthermore, lowering or elevating the reaction temperature had no positive effect (Table 1, Entries 22–25). It is noteworthy that control reactions carried out without base (Table 1, Entry 26) or without the palladium catalyst (Table 1, Entry 27) delivered no cross-coupling product, indicating that the base and the catalyst are necessary for the cross-coupling reaction.

Strikingly, the cross-coupling reaction carried out in the presence of KF in DMSO with polystyrene-supported palladium catalyst (corresponding to 0.01 equiv. Pd with respect to $BiPh_3$) at 105 °C provided the highest cross-coupling conversion (Table 1, Entry 19). Furthermore, the atom efficiency of triphenylbismuth, for which all three phenyl groups efficiently coupled with 3 equiv. of electrophilic coupling partner, was thoroughly established.

Subsequently, we investigated the cross-coupling reaction of various triarylbismuths with a variety of electronically diverse aryl iodides. The reaction of various substituted aryl iodides with different triarylbismuths was found to be fast and efficient, furnishing good to high yields of the cross-coupling biphenyls in a short reaction time. As shown in Table 2, the reaction of both iodobenzene (Table 2, Entries 1–4) and electron-withdrawing aryl iodides (Table 2, Entries 5–14) afforded good to high yields of the cross-coupling products with different triarylbismuths. In particular, 1-chloro-2-iodobenzene, which has some discernible steric encumbrance, afforded the corresponding products in moderate yields (Table 2, Entries 15–18). Further studies indicated that the reactions of electron-donating 1-iodo-4-methoxybenzene furnished moderate yields of the products (Table 2, Entries 21 and 22). Also, it can be conducted from Table 2 that the reactivity of a range of triarylbismuths in the cross-coupling reaction was unaffected by a change in the electronics of the aryl rings in the triarylbismuths.

Table 1. Optimized conditions for the coupling of iodobenzene with triphenylbismuth.[a]



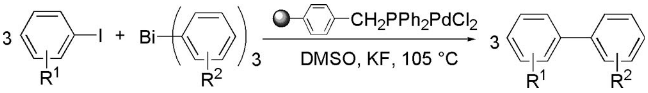
Entry	Base (mmol)	Solvent	T [°C]	Time [h]	Yield [%][b]
1	KF (3.5)	H_2O	105	24	0
2	KF (3.5)	H_2O	105	12	51[c]
3	KF (3.5)	CH_2Cl_2	105	5	66
4	KF (3.5)	CH_3CN	105	10	55
5	KF (3.5)	EtOH	105	10	65
6	KF (3.5)	PEG 400	105	2	64
7	KF (3.5)	$[Bmim]PF_6$	105	36	35
8	KF (3.5)	DMF	105	2	80
9	KF (3.5)	DMSO	105	0.25	87
10	K_2CO_3(3.5)	DMSO	105	0.5	81
11	K_3PO_4(3.5)	DMSO	105	1	84
12	$KHCO_3$ (3.5)	DMSO	105	2	59
13	$NaHCO_3$ (3.5)	DMSO	105	0.5	78
14	Et_3N (3.5)	DMSO	105	1	75
15	Al_2O_3 (3.5)	DMSO	105	1	35
16	NaOH (3.5)	DMSO	105	5	16
17	KOH (3.5)	DMSO	105	7	18
18	KF (4.0)	DMSO	105	0.25	80
19	**KF (3.0)**	**DMSO**	**105**	**0.25**	**94**
20	KF (2.5)	DMSO	105	0.25	86
21	KF (2.0)	DMSO	105	0.25	78
22	KF (3.0)	DMSO	115	0.25	88
23	KF (3.0)	DMSO	110	0.25	91
24	KF (3.0)	DMSO	100	0.25	90
25	KF (3.0)	DMSO	90	0.25	80
26	None	DMSO	105	24	0
27	KF (3.0)	DMSO	105	24	0[d]

[a] Reaction conditions: Iodobenzene (1.0 mmol), Ph_3Bi (0.34 mmol), solvent (3 mL), base, PS-supported Pd^{II} catalyst (corresponding to 0.01 equiv. Pd with respect to $BiPh_3$), stirring for the appropriate time. [b] Isolated yield. [c] 0.3 mmol TBAB (tetrabutylammonium bromide) was used. [d] Control reaction carried out without Pd catalyst.

To further elaborate the general reactivity of the present protocol with other electrophilic coupling reagents, we undertook the cross-coupling study with functionalized aryl bromides and the results are listed in Table 3. The reaction of 4-bromoacetophenone, 1-bromo-4-nitrobenzene, 4-bromobenzaldehyde, and 4-bromobenzonitrile as substrates with electron-withdrawing substituents furnished excellent yields of the functionalized biphenyls (Table 3, Entries 1–4). However, the cross-coupling of 1-bromo-4-chlorobenzene gave moderate yields of products (Table 3, Entries 5 and 6). Furthermore, bromobenzene substituted with electron-donating 4-methoxy and 4-methyl groups reacted inefficiently and gave poor yields of the corresponding cross-coupled products (Table 3, Entries 8 and 9). Notably, electron-donating substrates, which are known to show poor reactivity in this type of reaction, showed moderate reactivity with triarylbismuths.

The reusability and efficiency of the polystyrene-supported Pd^{II} catalyst were investigated on the model coupling reactions of iodobenzene with Ph_3Bi in the presence of KF in DMSO at 105 °C (Table 4). When the reaction was finished, the catalyst was recycled by filtering from the reaction mixture and washing with acetone (3 × 5 mL). After this step, the recycled catalyst was subjected to a second run by charging with the same substrates (iodobenzene, Ph_3Bi, DMSO, and KF). As shown in Table 4, high recyclable efficiency (the average isolated yield is 92% for 11 consecutive runs) of the catalyst was obtained, which demonstrates the practical recyclability of this catalyst and its potential use in large-scale processes.

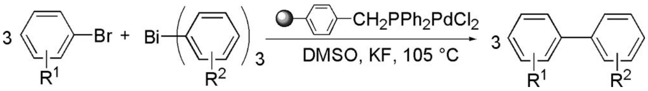
Table 2. Cross-coupling of aryl iodides with triarylbismuths.[a]



Entry	R^1	R^2	Time [min]	Yield [%][b]
1	H	H	15	94
2	H	4-Cl	15	88
3	H	4-CH_3	15	85
4	H	4-OCH_3	15	83
5	4-NO_2	H	15	96
6	4-NO_2	4-Cl	30	80
7	4-NO_2	4-F	20	82
8	4-NO_2	4-CH_3	20	91
9	4-NO_2	4-OCH_3	20	89
10	3-NO_2	H	20	92
11	3-NO_2	4-Cl	30	82
12	3-NO_2	4-F	20	80
13	3-NO_2	4-CH_3	20	92
14	3-NO_2	4-OCH_3	20	90
15	2-Cl	H	20	73
16	2-Cl	4-F	30	75
17	2-Cl	4-CH_3	30	78
18	2-Cl	4-OCH_3	30	78
19	3-CH_3	4-CH_3	40	80
20	3-CH_3	4-OCH_3	45	77
21	4-OCH_3	H	50	58
22	4-OCH_3	4-Cl	120	55

[a] Reaction conditions: aryl iodide (1.0 mmol), Ar_3Bi (0.34 mmol), DMSO (3 mL), KF (3.0 mmol), PS-supported Pd^{II} catalyst (corresponding to 0.01 equiv. Pd with respect to $BiAr_3$), stirring at 105 °C for the appropriate time. All products were characterized by melting point, IR, ^1H NMR, and ^{13}C NMR spectroscopy, and MS. [b] Isolated yield.

Table 3. Cross-coupling of aryl iodides with triarylbismuths.[a]



Entry	R^1	R^2	Time [min]	Yield [%][b]
1	4-$COCH_3$	H	30	91
2	4-NO_2	H	30	93
3	4-CHO	H	30	90
4	4-CN	H	40	92
5	4-Cl	H	50	76
6	4-Cl	4-Cl	120	70
7	H	H	60	65
8	4-OMe	H	120	43
9	4-CH_3	H	120	56

[a] Reaction conditions: aryl bromide (1.0 mmol), Ar_3Bi (0.34 mmol), DMSO (3 mL), KF (3.0 mmol), PS-supported Pd^{II} catalyst (corresponding to 0.01 equiv. Pd with respect to $BiAr_3$), stirring at 105 °C for the appropriate time. All products were characterized by melting point, IR, ^1H NMR, and ^{13}C NMR spectroscopy, and MS. [b] Isolated yield.

Table 4. Recycling of polymer-supported Pd^{II} catalyst.[a]

Entry	PS-supported Pd^{II}	Yield [%][b]
1	fresh	94
2	recycle 1	93
3	recycle 2	92
4	recycle 3	94
5	recycle 4	92
6	recycle 5	91
7	recycle 6	94
8	recycle 7	92
9	recycle 8	93
10	recycle 9	92
11	recycle 10	90

[a] Reaction conditions: Iodobenzene (1.0 mmol), Ph_3Bi (0.34 mmol), DMSO (3 mL), KF (3.0 mmol), PS-supported Pd^{II} catalyst (corresponding to 0.01 equiv. Pd with respect to $BiPh_3$), stirring at 105 °C for 15 min. [b] Isolated yield.

Conclusions

In conclusion, we have disclosed an efficient methodology for the synthesis of biaryl compounds that involves the cross-coupling of triarylbismuths with aryl bromides and aryl iodides catalyzed by polystyrene-supported palladium. The present protocol is highly efficient, as three equivalents of the aryl halide reacts with one equivalent of the triarylbismuth cleanly to provide a high yield of the cross-coupling product in short reaction time. In addition, the polystyrene-supported Pd^{II} catalyst can be easily recycled, and the efficiency of reusability of the catalyst for the coupling reactions did not decrease much over 10 runs.

Experimental Section

General Procedure for the Cross-Coupling Reactions of Ar_3Bi with Aryl Halides: To a round-bottomed flask charged with a mixture of KF (3.0 mmol), PS-supported Pd^{II} (corresponding to 1 mol-% palladium), and DMSO (3 mL) was added the aryl halide (1.0 mmol) and Ar_3Bi (0.34 mmol). The mixture was stirred at an oil bath temperature of 105 °C for the indicated time. After completion of the reaction as indicated by TLC, the mixture was cooled. Ethyl acetate (10 mL) and 1 M HCl (10 mL) were added, and the mixture was then filtered and the polymer catalyst was recycled by washing with acetone (3 × 5 mL). The residue was extracted with ethyl acetate (3 × 10 mL), and the combined ethyl acetate extract was washed with saturated NaCl, dried with anhydrous $MgSO_4$, filtered, and concentrated. The product was recrystallized from 95% ethanol or purified by column chromatography on silica gel (petroleum ether/ethyl acetate, 40:1) to give the analytically pure product.

PS-Supported Pd^{II} Catalyst Recycling Procedure: The recycled catalyst was subjected to a second run of cross-coupling reaction by charging with the same substrates (iodobenzene, Ph_3Bi, DMSO, and KF) without further addition of PS-supported Pd^{II}. The procedure is the same as that above.

Supporting Information (see footnote on the first page of this article): Experimental procedures, characterization data and copies of the original ^1H NMR and ^{13}C NMR spectra of compounds **1**–**28**.

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