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Synthesis of [60]Fullerene-Fused Spiroindanes by Palladium-Catalyzed Oxidative Annulation of [60]Fullerene with 2-Aryl Cyclic 1,3-Dicarbonyl Compounds

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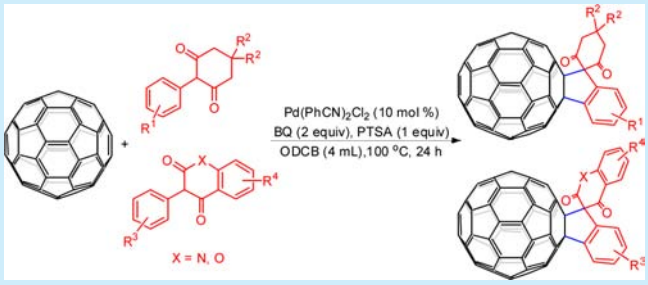
Supporting Information

ABSTRACT: A convenient and facile palladium-catalyzed reaction of [60]fullerene (C_{60}) with 2-aryl cyclic 1,3-dicarbonyl compounds via the enolate-directed sp^2 C−H activation and sp^3 C−H functionalization has been exploited to synthesize the novel and rare C_{60}-fused spiroindanes for the first time. This reaction is easy to perform with broad substrate scope and provides diversified products in 20−50% yields. A plausible reaction mechanism involving the palladium-catalyzed enolate-directed C−H activation and subsequent cyclization has been proposed, and the electrochemistry of the C_{60}-fused spiroindanes has also been investigated.



Owing to the potential applications of fullerene derivatives in materials science and biomedicine, numerous methodologies for chemical modification of fullerenes including various cycloadditions, radical additions, and nucleophilic additions have been developed during the last two decades.[1] Recently, the transition-metal-catalyzed methodology has been demonstrated as one of the most efficient approaches for the functionalization of fullerenes and offers more opportunities to synthesize novel functionalized fullerenes.[2] Our group has focused on developing new protocols to functionalize [60]fullerene (C_{60}) over the years. In 2009, we successfully introduced the palladium-catalyzed C−H activation strategy into fullerene chemistry and subsequently synthesized a series of novel [60]fullerene derivatives.[3] [60]Fullerene-fused heterocycles such as indolines,[3a] isoquinolinones,[4] azepines,[3b] tetrahydroisoquinolines,[3d] tetrahydrobenzazepines,[3f] sultones,[3c] and tetrahydrobenzooxepines/isochromans[3e] have been achieved by the palladium-catalyzed reactions of C_{60} with anilides, benzamides, *N*-sulfonyl-2-aminobiaryls, *N*-benzyl sulfonamides, *N*-(2-arylethyl) sulfonamides, arylsulfonic acids, and 2-phenylethyl/benzyl alcohols, respectively. [60]Fullerene-fused carbocycles such as dihydrophenanthrenes[3g] and tetralones[3h] have also been realized by the palladium-catalyzed reactions of C_{60} with 2-arylbenzoic acids and *sec*-alkyl aryl ketones, respectively. Despite the impressive progress in this field, there is still a demand for the synthesis of more novel fullerene derivatives through this powerful palladium-catalyzed C−H bond activation method. Recently, the Lam group reported the ruthenium-, palladium-, and rhodium-catalyzed reactions of alkynes/dienes with 2-aryl cyclic 1,3-dicarbonyls for the preparation of spiroindenes/spiroindanes.[5] Nevertheless, the reports on the synthesis of fullerene-fused indanes are still relatively deficient. In 1994, the Saunders group reported the synthesis of ^{3}He@C_{60}-fused indanes through the reaction of ^{3}He@C_{60} with the diradical generated by heating cyclopropa[b]naphthalene.[6a] In 2011, our group developed the $Mn(OAc)_3$-mediated radical reaction of C_{60} with 2-arylmalonates and 2-arylcyanoacetates to afford C_{60}-fused indane derivatives.[6b] Later, our group also revealed the synthesis of C_{60}-fused indanes through a Brønsted acid-promoted rearrangement reaction of C_{60}-fused tetrahydroisoquinolines and C_{60}-fused tetrahydrobenzazepines.[3d,f] In 2013, our group discovered a $FeCl_3$-mediated cyclization of C_{60} with *N*-benzhydryl sulfonamides to afford the C_{60}-fused indanes under high-speed vibration milling conditions.[6c] Herein, we report the synthesis of novel and rare C_{60}-fused spiroindanes by the palladium-catalyzed enolate-directed annulation of C_{60} with 2-aryl cyclic 1,3-dicarbonyl compounds. To the best of our knowledge, C_{60}-fused spiroindanes have not been synthesized until now. Compared with the previous work, the current strategy provides C_{60}-fused spiroindanes with broad substrate scope in a one-step procedure.

We began our exploration using 5,5-dimethyl-2-phenylcyclohexane-1,3-dione (**1a**) as the representative substrate to react with C_{60} to optimize the reaction conditions. The use of

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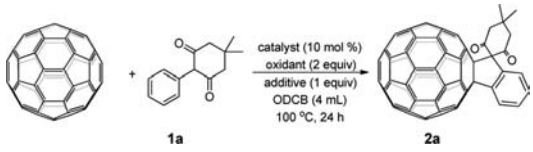
2 equiv of 1,4-benzoquinone (BQ) as the oxidant, 1 equiv of *p*-toluenesulfonic acid (PTSA) as the additive, and 10 mol % of $Pd(OAc)_2$ as the catalyst in 1,2-dichlorobenzene (ODCB) at 100 °C provided the desired product **2a** in 10% yield (Table 1, entry 1). The use of other Pd-catalysts such as

Table 1. Optimizing the Reaction Conditions for the Pd-Catalyzed Reaction of C_{60} with 1a[a]



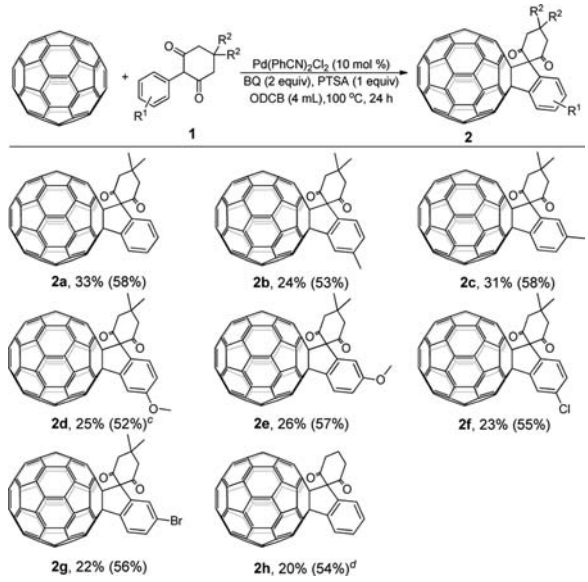
entry	catalyst	oxidant	additive	yield(%)[b]
1	$Pd(OAc)_2$	BQ	PTSA	10 (37)
2	$Pd(TFA)_2$	BQ	PTSA	10 (31)
3	$PdCl_2$	BQ	PTSA	11 (37)
4	$Pd(CH_3CN)_2Cl_2$	BQ	PTSA	27 (59)
5	$Pd(PhCN)_2Cl_2$	BQ	PTSA	33 (58)
6	$Pd(OTf)_2(MeCN)_4$	BQ	PTSA	22 (58)
7	$Pd(PhCN)_2Cl_2$	$Na_2S_2O_8$	PTSA	17 (39)
8	$Pd(PhCN)_2Cl_2$	NFSI	PTSA	20 (39)
9	$Pd(PhCN)_2Cl_2$	Oxone	PTSA	8 (20)
10	$Pd(PhCN)_2Cl_2$	$Cu(OAc)_2$	PTSA	trace
11	$Pd(PhCN)_2Cl_2$	AgOAc	PTSA	trace
12	$Pd(PhCN)_2Cl_2$	BQ	D-CSA	31 (49)
13	$Pd(PhCN)_2Cl_2$	BQ	TFA	26 (65)
14	$Pd(PhCN)_2Cl_2$	BQ	HOTf	23 (55)
15	$Pd(PhCN)_2Cl_2$	BQ	PivOH	28 (56)
16	$Pd(PhCN)_2Cl_2$	BQ	–	27 (52)
17[c]	$Pd(PhCN)_2Cl_2$	BQ	PTSA	trace
18[d]	$Pd(PhCN)_2Cl_2$	BQ	NaOAc	NP
19[d]	$Pd(PhCN)_2Cl_2$	BQ	K_2CO_3	NP
20[e]	$[RuCl_2(p\text{-cymene})]_2$	BQ	PTSA	8 (22)
21[e]	$[Cp^*RhCl_2]_2$	BQ	PTSA	6 (19)

[a]All the reactions were performed with 0.05 mmol of C_{60}, 0.10 mmol of **1a**, 0.10 mmol of oxidant, 0.005 mmol of catalyst, and 0.05 mmol of additive in 1,2-dichlorobenzene (4 mL) at 100 °C for 24 h. [b]Isolated yield. Values in parentheses were based on consumed C_{60}. [c]0.5 mL of PhCN was added. [d]2 equiv of additive was used. [e]5 mol % of catalyst.

$Pd(TFA)_2$ and $PdCl_2$ gave similar results (Table 1, entries 2 and 3). To our delight, $Pd(CH_3CN)_2Cl_2$ exhibited good efficiency to afford **2a** in 27% yield (Table 1, entry 4), and $Pd(PhCN)_2Cl_2$ gave the best result to provide product **2a** in 33% yield (Table 1, entry 5). $Pd(OTf)_2(MeCN)_4$, a cationic Pd(II) catalyst,[3h] decreased the yield of **2a** to 22% (Table 1, entry 6 versus entry 5). Further examination of other oxidants, including $Na_2S_2O_8$, *N*-fluorobenzenesulfonimide (NFSI), Oxone, $Cu(OAc)_2$, and AgOAc, indicated that BQ was still the most effective oxidant for this reaction (Table 1, entries 7–11). Replacing PTSA with D-camphorsulfonic acid (D-CSA), trifluoroacetic acid (TFA), trifluoromethanesulfonic acid (HOTf), and pivalic acid (PivOH) could not give a better yield (Table 1, entries 12–15). In addition, a lower yield (27%) of **2a** was obtained in the absence of PTSA (Table 1, entry 16 verus entry 5). Notably, only a trace amount of product **2a** was formed when 0.5 mL of PhCN was added as the cosolvent (Table 1, entry 17). No desired product **2a** was identified when the reaction was performed under basic conditions (Table 1, entries 18 and 19). Other metal catalysts such as $[RuCl_2(p\text{-cymene})]_2$ and $[Cp^*RhCl_2]_2$ were inferior compared with $Pd(PhCN)_2Cl_2$, affording **2a** in only 8% and 6% yields, respectively (Table 1, entries 20 and 21).

With the optimized reaction conditions in hand, the substrate scope was investigated by employing a wide array of 5,5-dimethyl-2-arylcyclohexane-1,3-diones. As shown in Scheme 1, different substituent groups on the phenyl ring

Scheme 1. Results for the Pd-Catalyzed Reaction of C_{60} with 2-Arylcyclohexane-1,3-diones 1a–h[a,b]



[a]Unless otherwise specified, all the reactions were performed with 0.05 mmol of C_{60}, 0.10 mmol of **1**, 0.005 mmol of $Pd(PhCN)_2Cl_2$, 0.10 mmol of BQ, 0.05 mmol of PTSA in 1,2-dichlorobenzene (4 mL) at 100 °C for 24 h. [b]Isolated yield. Values in parentheses were based on consumed C_{60}. [c]20 mol % of $Pd(PhCN)_2Cl_2$. [d]50 mol % of $Pd(PhCN)_2Cl_2$ for 5 h.

were tolerated in this transformation. Substrates with methyl, methoxy at the *meta*- or *para*-position of the phenyl ring could be smoothly transformed into the corresponding products **2b–e** in 24–31% yields. It is worth noting that halogens such as Cl and Br were also compatible, giving **2f** and **2g** in 23% and 22% yields, respectively, offering the opportunity for further synthetic transformations. 2-Phenylcyclohexane-1,3-dione, which was less reactive than its counterpart 5,5,-dimethyl-2-phenylcyclohexane-1,3-dione, required higher loading of the catalyst, i.e., 50 mol % $Pd(PhCN)_2Cl_2$, to provide product **2h** in 20% yield.

Next, we further extended the substrate scope to a range of 2-aryl cyclic 1,3-dicarbonyl compounds (Scheme 2). Gratifyingly, 1-methyl-3-phenylquinoline-2,4(1H,3H)-dione could be exploited to give the corresponding product **4a** in 50% yield. Various functional groups such as methyl, methoxy, and chloro on the phenyl ring were tolerated, affording the products **4b–d** in 29–46% yields. Substrate 3-phenylquinoline-2,4(1H,3H)-dione containing a free N–H bond also underwent oxidative annulation to give product **4e** in 28% yield, indicating that a methyl group on the nitrogen atom in the substrate was not necessary. Similarly, 1-isopropyl-3-phenylquinoline-2,4(1H,3H)-dione reacted to give product **4f** in 43% yield. Replacing the nitrogen atom with the oxygen atom could successfully provide **4g** in 22% yield. However, 3-

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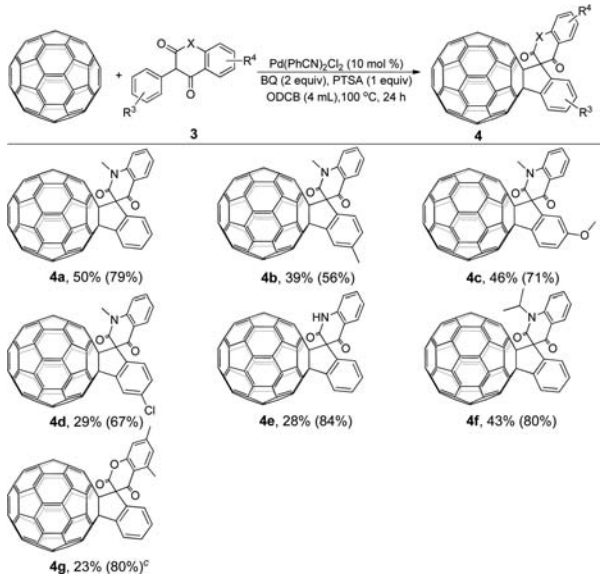
Scheme 2. Results for the Pd-Catalyzed Reaction of C_{60} with 2-Aryl Cyclic 1,3-Dicarbonyl Compounds 3a−g[a,b]



[a]Unless otherwise specified, all the reactions were performed with 0.05 mmol of C_{60}, 0.10 mmol of **3**, 0.005 mmol of $Pd(PhCN)_2Cl_2$, 0.10 mmol of BQ, and 0.05 mmol of PTSA in 1,2-dichlorobenzene (4 mL) at 100 °C for 24 h. [b]Isolated yield. Values in parentheses were based on consumed C_{60}. [c]20 mol % of $Pd(PhCN)_2Cl_2$ at 120 °C.

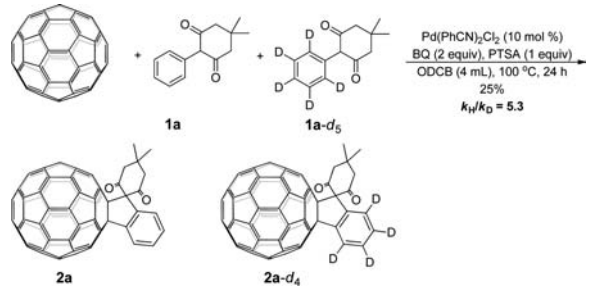
phenylpentane-2,4-dione, an acyclic substrate, was unreactive; Meldrum's acid derivatives such as 2,2-dimethyl-5-phenyl-1,3-dioxane-4,6-dione and barbituric acid derivatives such as 1,3-dimethyl-5-phenylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione were also inert under our reaction conditions.

The structures of C_{60}-fused spiroindanes **2a−h** and **4a−g** were unambiguously characterized by ESI HRMS, ^{1}H NMR, ^{13}C NMR, FT-IR, and UV−vis spectra. All of the mass spectra of these products gave the correct molecular ion peaks. The ^{13}C NMR spectra of **2a−h** clearly exhibited no more than 30 peaks in the range of 134−155 ppm for the sp^2-carbons of the C_{60} cage and two peaks in the 75−77 ppm range for the two sp^3-carbons of the C_{60} skeleton, consistent with the C_s symmetry of their molecular structures. The ^{13}C NMR spectra of **4a−g** exhibited at least 44 peaks in the range of 134−155 ppm for the sp^2-carbons of the fullerene cage and two peaks in the 75−79 ppm range for the two sp^3-carbons of the fullerene skeleton, consistent with the C_1 symmetry of their molecular structures. The UV−vis spectra of **2a−h** and **4a−g** displayed characteristic peaks at around 430 nm, which is a diagnostic absorption for 1,2-adducts of C_{60} with two carbon atoms attached to the C_{60} cage.

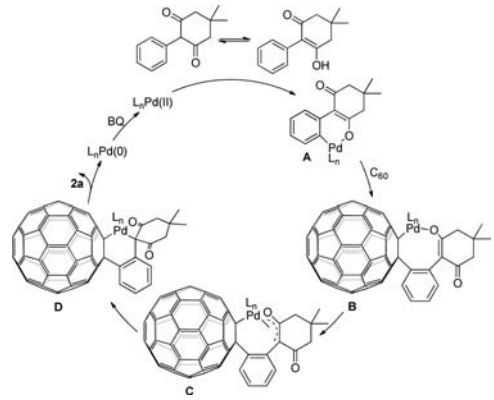
To gain insight into the reaction mechanism for the formation of C_{60}-fused spiroindanes **2**, a kinetic isotope effect (KIE) study was performed. Experiments revealed that the palladium-catalyzed reaction of C_{60} with 1 equiv of **1a** and 1 equiv of **1a**-d_5 exhibited a primary kinetic isotope effect k_H/k_D = 5.3, which indicated that the cleavage of C−H bond at the *ortho*-position was involved in the rate-determining step (Scheme 3).

On the basis of the above experimental results and previous literature,[3−5] a plausible reaction mechanism is proposed by using **1a** as the representative substrate (Scheme 4). Due to the fact that **1a** is a typical substrate for enol−keto

Scheme 3. Kinetic Isotope Effect Study for the Formation of C_{60}-Fused Spiroindane 2a



Scheme 4. Proposed Reaction Mechanism



tautomerism, the initial step should involve the palladium-catalyzed enolate-directed *ortho* C−H activation to form the six-membered palladacycle **A**.[5] Then, carbopalladation of C_{60} by the species **A** generates the intermediate **B**. Subsequently, the oxa-π-allylpalladacycle **C** is formed by rearrangement of the intermediate **B**. The oxa-π-allylpalladacycle **C** can further be transformed into the intermediate **D**. Finally, reductive elimination of **D** produces C_{60}-fused spiroindane **2a** and Pd(0). The Pd(0) species is reoxidized to a Pd(II) species by BQ to complete the catalytic cycle. Due to the low ratio of the enol form in substrates 3-phenylpentane-2,4-dione, 2,2-dimethyl-5-phenyl-1,3-dioxane-4,6-dione, and 1,3-dimethyl-5-phenylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, we can understand why they failed to provide the corresponding spiroindane products according to the proposed reaction mechanism.

The electrochemical properties of **2a−h** and **4a−g** along with C_{60} have been investigated by cyclic voltammetry (CV), and it was found that the obtained products exhibited irreversible redox processes probably due to the cleavage of the annulated C−C bond.[3h,7] However, the first redox process of **2a−h** and **4a−g** was reversible, and their first half-wave reduction potentials along with C_{60} are summarized in Table 2. As one of the C=C double bonds in fullerene derivatives is saturated, they usually show obvious cathodic shifts for the redox processes compared with the parent fullerene. However, the E_1 values of **2a−h** and **4a−g** were close to that of the pristine C_{60}, probably because the annulated carbon atom was connected with two electron-withdrawing carbonyl groups.

In summary, we report a novel palladium-catalyzed enolate-directed sp^2 C−H activation and sp^3 C−H functionalization of 2-aryl cyclic 1,3-dicarbonyl compounds with C_{60} to afford C_{60}-fused spiroindanes for the first time. The current protocol provides facile access to C_{60}-fused spiroindane derivatives via a

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Table 2. Half-Wave Reduction Potentials (V) of C_{60} and C_{60}-Fused Spiroindanes 2a–h and 4a–g[a]

compd	E_1	compd	E_1
C_{60}	−1.076	**2h**	−1.105
2a	−1.109	**4a**	−1.131
2b	−1.114	**4b**	−1.137
2c	−1.127	**4c**	−1.137
2d	−1.114	**4d**	−1.116
2e	−1.143	**4e**	−1.139
2f	−1.091	**4f**	−1.133
2g	−1.082	**4g**	−1.107

[a]Versus ferrocene/ferrocenium. Experimental conditions: 1 mM of compound and 0.1 M of *n*-Bu_4NClO_4 in anhydrous *o*-dichlorobenzene; reference electrode, SCE; working electrode, Pt; auxiliary electrode, Pt wire; scanning rate, 100 mV s^{-1}.

one-step procedure. A possible mechanism involving a palladium-catalyzed enolate-directed C–H activation and subsequent cyclization is proposed.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b01043.

Detailed experimental procedures and characterization data, the ^1H NMR and ^{13}C NMR spectra, and CVs of **2a–h** and **4a–g** (PDF)

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Notes

The authors declare no competing financial interest.

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