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Convergent Synthesis of Fluorene Derivatives by Rhodium-Catalyzed Stitching Reaction/Alkene Isomerization Sequence

Masaki Nishida^[a] and Ryo Shintani*^[a]

Abstract: A new and convergent synthetic method of fluorene derivatives has been developed by devising a rhodium-catalyzed stitching reaction/alkene isomerization sequence. The reactions proceed smoothly under mild conditions for a variety of substrate combinations, and extended π -conjugation systems are also readily accessible by utilizing this synthetic method. Optical properties of the obtained fluorene derivatives have also been examined.

Fluorenes constitute a structural motif often found in various functional π -conjugated materials such as light-emitting diodes, semiconductors, and chemosensors based on their optoelectronic properties.^[1] They are also used as ligands for transition metals in their anionic forms to provide effective catalysts for several reactions such as olefin polymerization.^[2] It is therefore highly important to develop synthetic methods that allow for a rapid access to diverse arrays of fluorenes and related compounds with high efficiency.

In addition to conventional synthetic approaches such as Friedel-Crafts cyclization^[3] and radical cyclization,^[4] transitionmetal-catalyzed cyclization involving C-H bond activation and subsequent C-C bond formation has recently been well investigated^[5] for both aromatic C-H bond activation^[6] and benzylic C-H bond activation.^[7] However, most of them are intramolecular processes based on the use of substituted biaryls or diarylmethanes,^[8] and it is more desirable to develop intermolecular reactions in view of convergence of the process and diversity of the accessible structures. In this regard, several intermolecular synthetic methods of fluorenes have been reported, including palladium-catalyzed tandem cross-coupling/cyclization reactions,[6d,g,7a,b] metal-catalyzed [2+2+2] cycloaddition of diynes and alkynes,^[9] and gold-catalyzed annulation of enynes and arylalkynes.^[10,11] Although some of them seem effective, they often suffer from limitations such as narrow substrate scope, high reaction temperature, and low product selectivity. As a new and complementary intermolecular approach, herein we describe the development of a rhodium-catalyzed stitching reaction/alkene isomerization sequence for the synthesis of fluorene derivatives from easily accessible substrate combinations.

Our synthetic strategy is illustrated in Scheme 1. Thus, we imagined that (benzo)fluorenes would be readily synthesized from 2-alkynylarylboronates I and 2-alkynylbezaldehydes (or β -alkynyl- α , β -enals) or their derivatives II as follows: a reaction of I with II in the presence of a rhodium(I) catalyst in a stitching manner^[12] would lead to initial product III through the formation of three consecutive carbon–carbon bonds via successive insertion of

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Scheme 1. Synthetic strategy of (benzo)fluorenes by rhodium-catalyzed stitching reaction followed by alkene isomerization (X = O, NR, etc.).

alkynes^[13] and an aldehyde (or its derivative),^[14] and subsequent isomerization of the diene moiety toward a thermodynamically more stable aromatic ring system accomplishes the formation of (benzo)fluorene **IV**.

Based on this synthetic strategy, we initially chose 2-((tertbutyldimethysilyl)ethynyl)phenylboronate 1a and 2-(1pentynyl)benzaldehyde (2a) as a model substrate combination for the rhodium-catalyzed stitching reaction [Eq. (1)]. After some investigation, we found that, by using [{Rh(OH)(cod)}2] (5 mol% Rh) as the catalyst with additional 1,5-cyclooctadiene (25 mol%) and H₂O (1.3 equiv) in THF, the reaction proceeded smoothly under mild conditions to give corresponding stitched product 3aa in 94% NMR yield.^[15] Treatment of crude **3aa** with *n*Bu₄NF led to clean formation of 11H-benzo[a]fluoren-5-ol 4aa through desilylation and alkene isomerization, and crude 4aa was further converted to corresponding acetate **5aa** for the ease of isolation (84% overall yield). It is worth noting that the first step of this reaction sequence represents a new entry of the rhodiumcatalyzed stitching reaction, which enables the formation of a 5,6fused carbocyclic system by a 6-membered ring-forming termination process using an aldehyde as the electrophilic component, instead of the previously reported 5-membered ring formation with an aryl bromide as the termination step.^[12]



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Scheme 2. Scope of rhodium-catalyzed stitching reaction followed by alkene isomerization.

We subsequently found that the present reaction sequence could be readily applied to the synthesis of various substituted fluorene derivatives as summarized in Scheme 2. For example, substituents on the alkyne of aldehydes 2 can be not only an alkyl or aryl group but also a silyl group, which is regarded as a terminal alkyne surrogate by treatment with nBu₄NF, to give corresponding benzofluorenes 5aa-5ad (42-86% vield). In addition, 2-(1pentynyl)benzaldehydes having various substituents as well as heteroaromatic aldehydes and an alkenyl aldehyde are applicable to give substituted benzofluorenes 5ae-5ai, heteroaryl-fused fluorenes 5aj and 4ak-4al, and alkyl-substituted fluorene 4am, respectively, in uniformly high yields (72-87% yield).^[16] With regard to the variation of arylboronates 1, several electronically different arylboronates including a naphthylboronate are well tolerated, giving corresponding products 5ba-5fa in 83-90% yield. By employing arylboronate 1b having a dimethylamino group and benzaldehyde 2h having a nitro group, donor-acceptor benzofluorene 5bh can also be synthesized (76% yield). Furthermore, substituents on the alkyne of arylboronates are not

limited to silyl groups, which will be removed by the treatment with nBu_4NF . Thus, phenyl and 2-naphthyl groups can be installed at 6-position by employing the corresponding arylboronates as shown in Eq. (2) (**4ga–4ha**, 72–78% yield). It is also possible to install substituents at this position by utilizing *ortho*-functionalization of the phenol moiety of obtained (benzo)fluorenes. For example, selective bromination of compound **4ak** proceeds smoothly by using 1.0 equiv of *N*-bromosuccinimide (NBS) to give **4ik** in 81% yield [Eq. (3)].



It is worth noting that the present stitching reaction conditions can also accommodate imines as the 6-membered ring-forming termination moiety instead of aldehydes described above.^[17] The stitching reaction of arylboronate **1a** with *N*-tosyl imine **6** proceeded smoothly under the same conditions, and subsequent treatment with *n*Bu₄NF led to benzofluorene **7** possessing a sulfonamido group at 5-position in 82% overall yield. Unlike the reactions using aldehydes, *tert*-butyldimethysilyl group at 6position remained intact by the reaction with *n*Bu₄NF in this particular case. Furthermore, an acylmethyl group can be installed at 5-position by employing alkyne **8** having an α , β unsaturated ketone as the termination moiety [Eq. (5)].^[18]



More extended π -conjugation systems are readily accessible as well by using the present synthetic method. For example, the two-fold stitching reaction of arylboronate **1g** with dialkynyl

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dialdehyde **10** under slightly modified conditions, and subsequent treatment with *n*Bu₄NF followed by acetylation, quickly provides dihydrodiindenoanthracene **11** in a relatively high yield [60% yield; Eq. (6)]. As a different approach, benzofluorenyl triflate **5ad'**, which is obtained by triflation instead of acetylation in the final step of the present synthetic sequence,^[18] can be homo-coupled under reducing conditions to give bibenzo[*a*]fluorenyl **12** in 68% yield [Eq. (7)].^[19]



With a series of new fluorene derivatives in hand, we began to examine their optical properties. As summarized in Table 1, we chose some of these compounds for comparison (**5aa**: parent, **5ba**: 9-NMe₂, **5ah**: 3-NO₂, **5bh**: 9-NMe₂-3-NO₂, **11**: doubly fused). In both UV-vis absorption (Figure 1) and fluorescence spectra (Figure 2), introduction of an electron-donating group at 9-position (**5ba**) or an electron-withdrawing group at 3-position (**5ah**) led to a red-shift in their UV-vis absorption and emission band maxima compared to the parent compound (**5aa**), although the quantum yields did not show much difference ($\Phi_F = 0.21-0.32$). Even more significant red-shifts of UV-vis absorption and emission band

Table 1. Optical properties of compounds 5aa, 5ba, 5ah, 5bh, and 11 in CH_2Cl_2 at 25 $^\circ C.$

Compound	UV-vis absorption ^[a] $\lambda_{max}/nm (\pounds/10^4 \text{ M}^{-1} \text{cm}^{-1})$	Fluorescence ^[b] λ _{max} /nm (λ _{ex} /nm)	$arPhi_{F} \left(\lambda_{ex} / nm ight)^{[b]}$
5aa	257 (4.6), 266 (7.1), 286 (1.1), 295 (1.3), 308 (1.3), 319 (1.1), 348 (0.2)	359, 372 (266)	0.25 (266)
5ba	290 (3.2), 345 (3.5)	458 (345)	0.32 (345)
5ah	281 (2.3), 295 (2.0), 303 (1.8), 385 (1.3)	537 (384)	0.21 (384)
5bh	266 (2.9), 307 (1.9), 458 (1.9)	723 (458) ^[c]	— (458) ^[c,d]
11	257 (2.8), 309 (7.9), 321 (17.5), 358 (0.5), 378 (0.8), 398 (1.1), 415 (1.0), 440 (0.7)	456, 485 (321)	0.30 (321)
[а] At 1.0 x 10 ⁻⁵ м. [b] At 1.0 x 10 ⁻⁴ м. [c] At 2.0 x 10 ⁻⁴ м. [d] Not determined.			







Figure 2. Fluorescence spectra of compounds **5aa** (blue line; $\lambda_{ex} = 266$ nm), **5ba** (green line; $\lambda_{ex} = 345$ nm), **5ah** (dark yellow line; $\lambda_{ex} = 384$ nm), **5bh** (orange line; at 2.0 x 10⁻⁴ M, $\lambda_{ex} = 458$ nm), and **11** (red line; $\lambda_{ex} = 321$ nm) in CH₂Cl₂ (1.0 x 10⁻⁴ M except for **5bh**) at 25 °C.

maxima were observed for donor-acceptor compound **5bh**, reaching near-IR region in its emission maximum ($\lambda_{max} = 723$ nm). On the other hand, compared with benzofluorene **5aa**, doubly fused dihydrodiindenoanthracene **11** showed much larger absorption coefficient with a significant red-shift while keeping the overall absorption pattern, and the emission band maximum was also red-shifted with a similar quantum yield. These results demonstrate that the optical properties can be effectively tuned by changing the substituents and/or extending the core structure of these (benzo)fluorene derivatives.

In summary, we have developed a new and convergent synthetic method of fluorene derivatives by devising a rhodiumcatalyzed stitching reaction/alkene isomerization sequence. The reactions proceed smoothly under mild conditions for a variety of substrate combinations, and extended π -conjugation systems are also readily accessible by utilizing the present synthetic method. Future studies will be directed toward further development of new and efficient synthetic methods for various functional organic compounds.

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Keywords: fluorene • isomerization • π-conjugated compound • rhodium • stitching reaction

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