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A rapid access to fused polycyclic indolo[2,1-*a*]isoquinolins *via* Pd-catalyzed sequential Heck/C-H activation/amination reaction with diaziridinone

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ABSTRACT

Polycyclic pyrrole fused indolo[2,1-*a*]isoquinolins were efficiently generated from alkene-tethered indole derivatives and di–*tert*-butyldiaziridinone in up to 99% yield in the presence of Pd catalyst. The reaction likely proceeded *via* sequential Heck, C–H activation, and amination process. Varying *N*-substituents of indole substrates led to indolo[3,2-*b*]indoles in up to 98% yields.

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Indoles are one of the most ubiquitous nitrogen-containing heterocycles. Indolo[2,1-*a*]isoquinolines represent an important class of polycyclic fused indoles, and are present in a variety of bioactive molecules and pharmaceuticals, and materials (Fig. 1) [1–5]. In the past, considerable efforts have been made in constructing indolo[2,1-*a*]isoquinoline skeletons using various strategies, including radical [6–29] or metal-catalyzed [30–37] cascade cyclization processes. A Pd-catalyzed tandem Heck process also presents a powerful approach to this class of molecules [38–46] particularly for skeletons with additional fused ring(s) [47–50]. An introduction of a fused nitrogen heterocycle like pyrrole fused indolo[2,1-*a*]isoquinoline (**6**) would provide new structural diversities for the biological activities, and little has been reported for such type of molecules.

Previously, we have found that a five-membered azacycle can be generated by reacting a pallada(II)cycle (**7**) with di–*tert*-butyldiaziridinone (**8**) (Scheme 1) [51,52]. The reaction likely proceeded *via* four-membered pallada(IV)cycle **9** with simultaneous formation of two C–N bonds [51–64]. This bisamination process provides unique opportunities for the synthesis of structurally diverse *N*-heterocycles. Now we report that pyrrole fused indolo[2,1-*a*]isoquinolines (**6**) can be efficiently constructed from indole **12** by such reaction process *via* tandem Heck/C–H activation/amination through pallada(II)cycle intermediate **13** (Scheme 2).

Indole **12a** was used as the test substrate for initial studies. Various phosphine ligands were examined with 5 mol% $Pd(OAc)_2$, and 2.0 equiv. of di–*tert*-butyldiaziridinone (**8**) in toluene at 110 °C (Table 1, entries 1–11) (for more details, see Table S1 in Supporting information). The ligand was found to be important to the reaction. The highest yield (58%) was obtained with $PPhCy_2$ (Table 1, entry 7). Further investigation with additional Pd catalysts (Table 1, entries 12–14) and solvents (Table 1, entries 15–18) (for more details, see Table S1) did not lead to any improvement. It appeared that the reaction process was sensitive to the reaction temperature. The reaction went to completion at 130 °C while no product was observed at 90 °C (Table 1 entry 20 *vs.* entry 19). At 130 °C, the reaction was completed within 4 h (Table 1, entry 21).

With the optimized reaction conditions, the substrate scope was subsequently investigated with a variety of indole substrates. As shown in Scheme 3, the reaction can be extended to various substituted 1-methacryloyl indoles, giving the corresponding pyrrole fused indolo[2,1-*a*]isoquinolines **6a-6j** in 43%–99% isolated yields. High yield was also obtained with 1-(2-benzylacryloyl)indole (Scheme 3, **6k**). A lower yield was obtained in the case of 1-(2-phenylacryloyl) indole (Scheme 3, **6l**). 1-(2-Methylallyl) indoles were also effective for the reaction, giving

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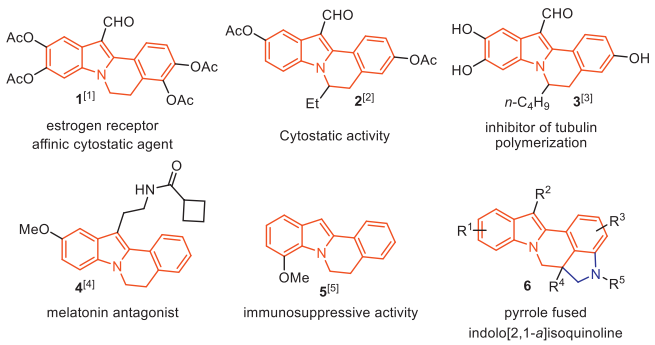
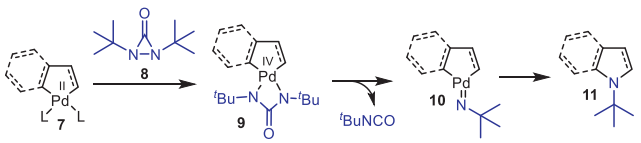
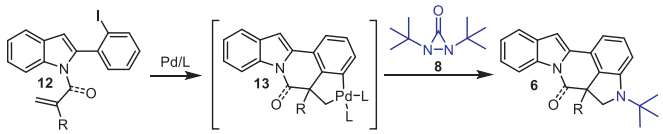


Fig. 1. Examples of bioactive indolo[2,1-*a*]isoquinolines.

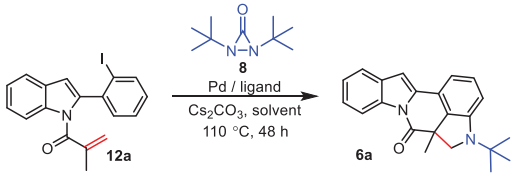


Scheme 1. Bisamination of pallada(II)cycles with diaziridinone.



Scheme 2. Pd-catalyzed sequential Heck/C–H activation/amination.

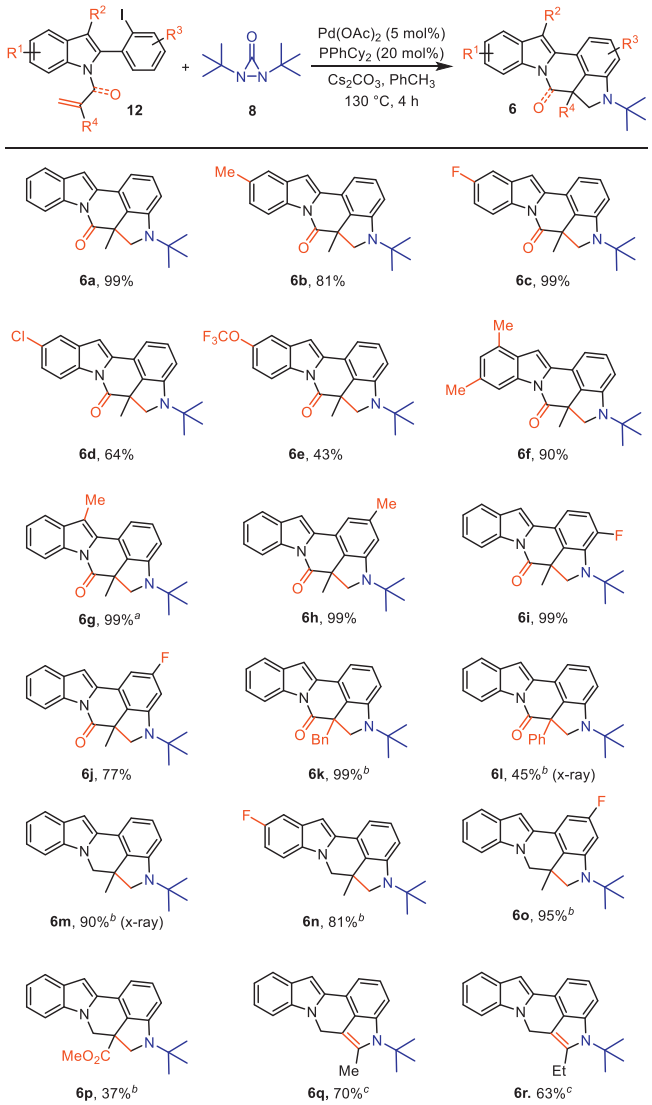
Table 1
Studies on the reaction conditions.[a]



Entry	[Pd]	Ligand	Yield (%)[b]
1	$Pd(OAc)_2$	PPh_3	43
2	$Pd(OAc)_2$	$(o\text{-MeOPh})_3P$	33
3	$Pd(OAc)_2$	$(p\text{-FPh})_3P$	26
4	$Pd(OAc)_2$	$(2\text{-thienyl})_3P$	30
5	$Pd(OAc)_2$	$(2\text{-furyl})_3P$	41
6	$Pd(OAc)_2$	PPh_2Cy	36
7	$Pd(OAc)_2$	$PPhCy_2$	58
8	$Pd(OAc)_2$	PCy_3	33
9	$Pd(OAc)_2$	dppb	19
10	$Pd(OAc)_2$	dppf	25
11	$Pd(OAc)_2$	Xantphos	31
12	$PdCl_2$	$PPhCy_2$	35
13	PdI_2	$PPhCy_2$	23
14	$Pd(PPh_3)_4$	$PPhCy_2$	26
15	$Pd(OAc)_2$ (1,4-dioxane)	$PPhCy_2$	47
16	$Pd(OAc)_2$ (hexane)	$PPhCy_2$	27
17	$Pd(OAc)_2$ (DCE)	$PPhCy_2$	27
18	$Pd(OAc)_2$ (DME)	$PPhCy_2$	27
19	$Pd(OAc)_2$ (90 °C)	$PPhCy_2$	NP
20	$Pd(OAc)_2$ (130 °C)	$PPhCy_2$	99
21	$Pd(OAc)_2$ (130 °C, 4 h)	$PPhCy_2$	99

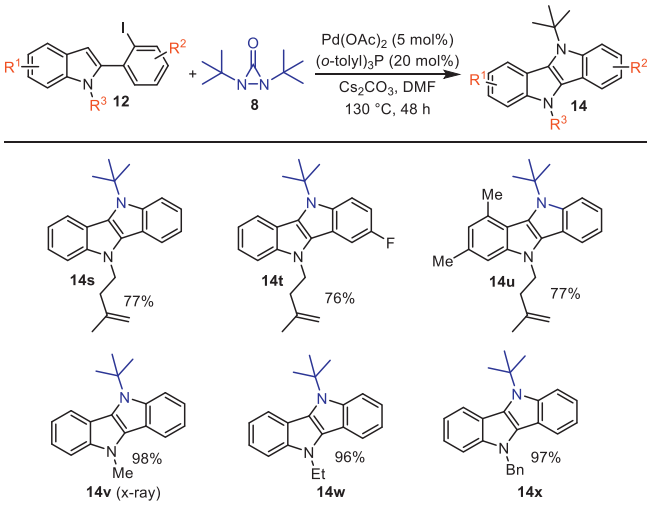
[a] All reactions were carried out with indole **12a** (0.15 mmol), Pd (0.0075 mmol), ligand (0.015–0.030 mmol, Pd/P = 1/4), di-*tert*-butyldiaziridinone (**8**) (0.225 mmol), and Cs_2CO_3 (0.225 mmol) in $PhCH_3$ (1.5 mL) at 110 °C under N_2 for 48 h unless otherwise noted.

[b] The yield was determined by ^1H NMR analysis of the crude reaction mixture with 1,1,2,2-tetrachloroethane as internal standard.

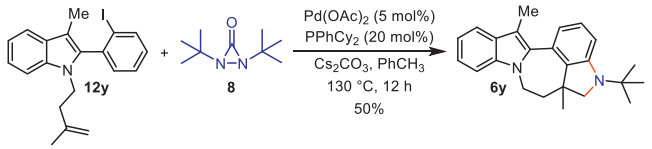


Scheme 3. The reaction scope. All reactions were carried out with **12** (0.30 mmol), $Pd(OAc)_2$ (0.015 mol), $PPhCy_2$ (0.060 mmol), di-*tert*-butyldiaziridinone (**8**) (0.45 mmol), and Cs_2CO_3 (0.45 mmol) in $PhCH_3$ (2.0 mL) at 130 °C under N_2 for 4 h unless otherwise noted, the yield was isolated yield. [a] With 1.0 mL $PhCH_3$. [b] For 12 h. [c] With $Pd(OAc)_2$ (0.015 mol), (*o*-tolyl)$_3$P (0.060 mmol), di-*tert*-butyldiaziridinone (**8**) (0.45 mmol), KOPiv (0.15 mmol), and Cs_2CO_3 (0.45 mmol) in DMF (1.0 mL) at 130 °C for 12 h.

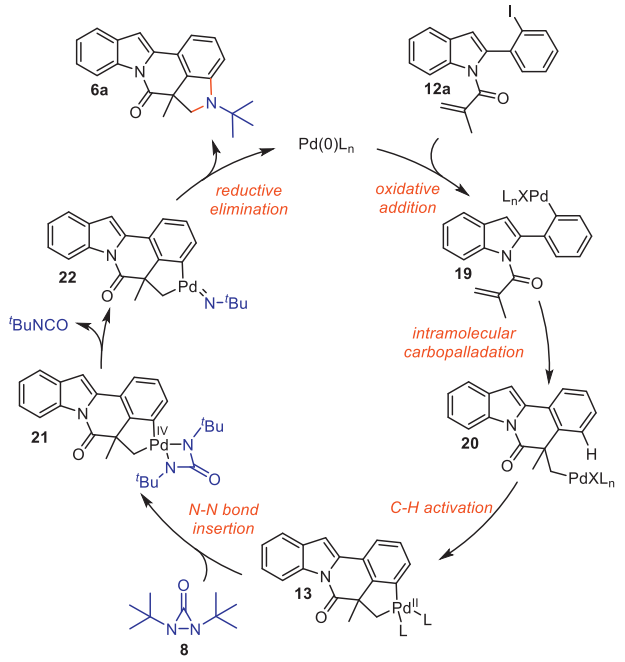
the corresponding pyrrole fused indolo[2,1-*a*]isoquinolines **6m-6o** in 81 %–95 % yields. Significantly lower yield was obtained when the methyl group on the olefin was replaced with the methyl ester group (Scheme 3, **6p**). The reaction can also be applied to propargyl indoles to give the corresponding pyrrole fused indolo[2,1-*a*]isoquinolines **6q** and **6r** in 63 %–70 % yields with modified reaction conditions. The reaction process was not effective to indole **12s** tethered with 3-methylbut-3-en-1-yl group (one carbon longer), giving a messy mixture under the reaction conditions. With modified conditions, indolo[3,2-*b*]indole **14s** was isolated in 77 % yield (Scheme 4) [65–80]. This reaction process also proceeded well with *N*-Me, Et, and Bn substituted indoles **12v-x**, giving the corresponding indolo[3,2-*b*]indole **14v-x** in 96 %–98 % yields (Scheme 4). Further studies showed that seven-membered ring product **6y** can be isolated in 50 % yield when the corresponding indole with a methyl at the indole's C-3 position (**12y**) used (Scheme 5).



Scheme 4. The indole C-3 amination reaction.



Scheme 5. The synthesis of seven-membered polycyclic indole **6y**.



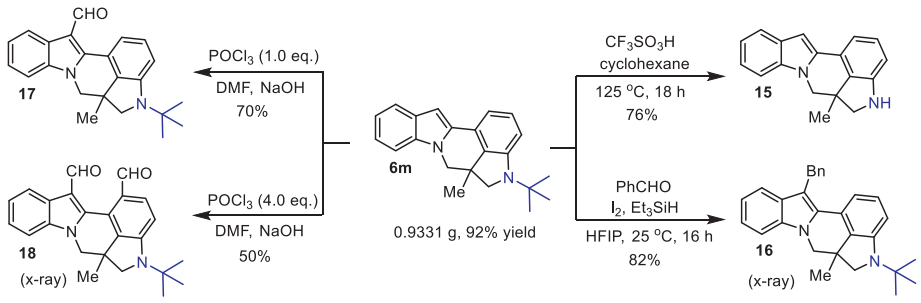
Scheme 7. Proposed catalytic pathway.

As exemplified with **6m**, the reaction proceeded well at gram scale (Scheme 6). Several synthetic transformations were also carried out with **6m**. The *tert*–butyl group can be removed with CF_3SO_3H/cyclohexane to give compound **15** in 76% yield. The benzylation of **6m** with benzaldehyde, I_2 and Et_3SiH led to compound **16** in 82% yield [81]. The formylation of **6m** with 1 equiv. of $POCl_3$ afforded aldehyde **17** in 70% yield [82]. Bisaldehyde **18** can be obtained in 50% yield when excess of $POCl_3$ used.

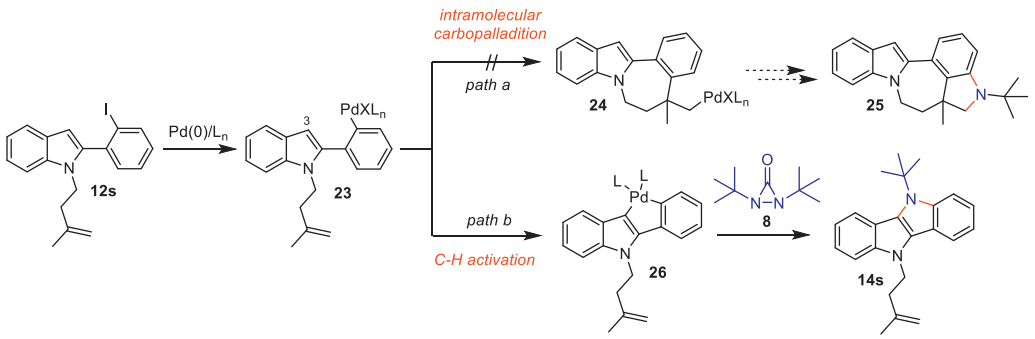
A precise understanding of the reaction mechanism awaits further study. On the basis of earlier studies [51–57], a plausible catalytic cycle is outlined in Scheme 7 (with **12a** as an example). The oxidative addition of Pd(0) to **12a** led to Pd intermediate **19**, which subsequently underwent an intramolecular carbopalladation to form alkyl Pd species **20**. Pallada(II)cycle **13** was then generated from **20** *via* aryl C–H activation [83–101]. The oxidative addition of **13** to di–*tert*-butyldiaziridinone (**8**) gave four-membered pallada(IV)cycle **21**, which afforded pallada(IV)nitrene species **22** upon release of *t*BuNCO. At the end, pyrrole fused indolo[2,1-*a*]isoquinoline **6a** was formed from **22** *via* two consecutive reductive eliminations with regeneration of the Pd catalyst.

In the case of **12s** (Scheme 8), the formation of a seven-membered ring from Pd intermediate **23** was apparently unfavorable (path a). Instead, **23** could undergo a C–H activation at C-3 under proper reaction conditions to give pallada(II)cycle **26** [102–106], which was aminated with di–*tert*-butyldiaziridinone (**8**) to form indolo[3,2-*b*]indole **14s** (path b) [53]. The introduction of a methyl group at the indole's C-3 position suppressed path b and shifted the outcome towards the formation of the seven-membered ring product.

In summary, we have developed an efficient Pd-catalyzed sequential Heck/C–H activation/amination reaction process with alkene-tethered indole derivatives and di–*tert*-butyldiaziridinone, providing a variety of pyrrole fused indolo[2,1-*a*]isoquinoline polycycles bearing various functional groups. The reaction process provides ready access to structurally diverse indolo[2,1-*a*]isoquinoline compounds for biologically studies. In addition, indolo[3,2-*b*]indoles can be efficiently formed by varying *N*-substituents of indole substrates. The current work further illustrates the versatility of the diaziridinone for the construction of *N*-heterocycles. Further expansion of its synthetic utility is currently underway.



Scheme 6. Synthetic transformations of compound **6m**.



Scheme 8. Proposed catalytic pathway.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Junhua Li: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Yu Fu:** Writing – original draft, Visualization, Validation, Software, Formal analysis, Data curation. **Yian Shi:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.111376.

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