Green Chemistry

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Cite this: *Green Chem.*, 2022, **24**, 8441

Synthesis of dihydroindazolo[2,3-*f*]phenanthridin-5(6*H*)-ones *via* Rh(III)-catalyzed C–H activation of 2-aryl indazoles and annulation with iodonium ylides†

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Received 13th August 2022,
Accepted 23rd September 2022

DOI: 10.1039/d2gc03020a

rsc.li/greenchem

An efficient synthetic route to indazole-fused dihydrophenanthridinones in excellent to almost quantitative yields under mild reaction conditions was developed. The reaction utilizes the acid-controlled Rh(III)-catalyzed C–H activation of 3-arylindazoles followed by their annulation with readily available hypervalent iodonium ylides. This methodology afforded a wide range of products that could be isolated using only a simple filtration without the need for column chromatography. In addition, the catalytic system can be recycled at least eight times with excellent yields, which may make it amenable to industrial production. Moreover, the photophysical properties of the synthesized dihydroindazolo[2,3-*f*]phenanthridin-5(6*H*)-ones indicate that they may have potential applications as new fluorescent materials.

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Introduction

The indazole skeleton has attracted great interest because it exists in numerous natural products and exhibits significant biological activity.[1] In particular, indazole-based conjugated systems and fused aromatic ring compounds have been widely utilized in drugs and materials science.[2] Consequently, much attention has been paid to methodology for the synthesis of these compounds.[3] Unfortunately, little research has been devoted to the development of methods to efficiently synthesize indazole-fused dihydrophenanthridinones. Thus, the development of an efficient and green synthetic route for indazole-fused dihydrophenanthridinones from readily available raw materials is highly desirable.

Recently, carbene precursors such as diazo compounds,[4] hydrazones,[5] sulfoxonium ylides,[6] and others,[7] have been extensively used as efficient coupling partners in transition-metal-catalyzed C–H activation/annulation reactions for the construction of various of complex heterocyclic skeletons. Compared to dangerous and explosive diazonium compounds, iodonium ylides are inexpensive and readily available hypervalent iodine reagents which show good thermal stability, safety, solubility in common organic solvents, and have been widely used in organic synthesis.[8] Some progress has also been made in using these reagents for C–H activation and for insertion into iodonium-ylide-derived carbenoids.[9] For example, Li and co-workers reported a $[Cp^*RhCl_2]_2$-AgOAc catalyzed C–H activation of a series of arenes with an annulation with iodonium ylides for the synthesis of a diverse array of heterocyclic compounds (Scheme 1a).[10] Maheswari also developed a $[Cp^*RhCl_2]_2$-$AgSbF_6$ catalyzed C–H activation of *N*-methoxybenzamides with an annulation with iodonium ylides for the synthesis of 5-methoxy-3,4-dihydrophenanthridine-1,6-(2*H*,5*H*)-diones (Scheme 1b).[11] Li and Yu independently described a method for the synthesis of cinnolines *via* a $[Cp^*RhCl_2]_2$-NaOAc catalyzed annulation of pyrazolidinones or *N*-methyl arylhydrazines that uses iodonium ylides (Scheme 1c and d).[12,13] The Kanchupalli group achieved selective access to tricyclic or tetracyclic N-heterocycles by changing the solvent of a $[Cp^*RhCl_2]_2$-AgOAc catalyzed reaction of *N*-carboxamide indoles and iodonium ylides (Scheme 1e).[14] Ji presented the synthesis of indoloquinazolinone derivatives *via* a $[Cp^*RhCl_2]_2$-AgOAc catalyzed reaction of *N*-carboxamide indole and iodonium reagents (Scheme 1f).[15] The above reactions provide attractive routes to heterocyclic derivatives using iodonium ylides, however, the synthetic potential of iodonium ylides is far from being fully exploited.

In a continuation of our interest in building heterocycles *via* rhodium-catalyzed C–H bond activations of indazoles,[16] here, an acid-controlled Rh(III)-catalyzed C–H activation of 3-arylindazoles and an annulation with iodonium ylides is presented. The reaction affords a wide range of dihydroindazolo

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†Electronic supplementary information (ESI) available. CCDC 2189691. For ESI and crystallographic data in CIF or other electronic format see DOI: https://doi.org/10.1039/d2gc03020a

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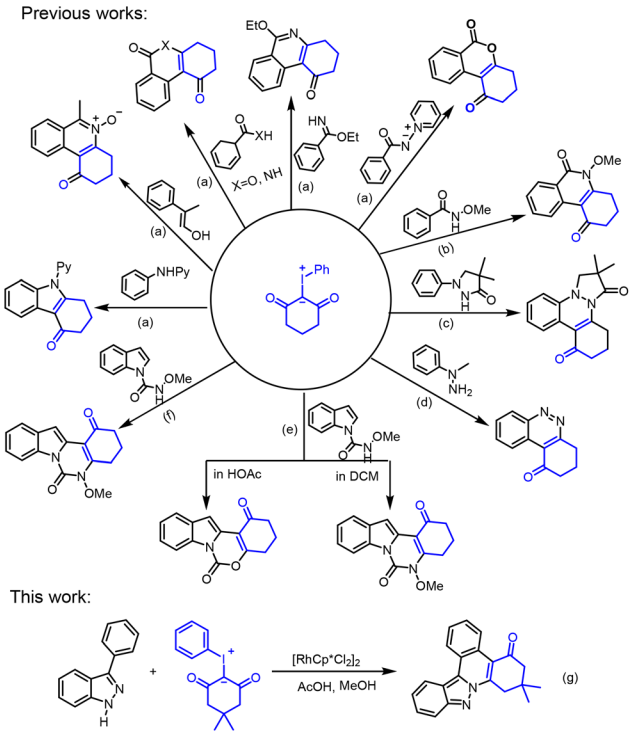


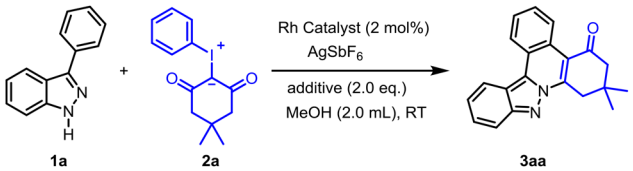
Scheme 1 The synthesis of heterocyclic derivatives using iodonium ylides.

[2,3-*f*]phenanthridin-5(6*H*)-ones with excellent to almost quantitative yields under mild reaction conditions (Scheme 1g). Furthermore, the reaction only requires simple work-up procedures and the catalyst can be recycled at least eight times whilst maintaining excellent yields. Moreover, the photophysical properties of the resulting dihydroindazolo[2,3-*f*]phenanthridin-5(6*H*)-ones indicate that they may have potential applications in the fields of new fluorescent materials.

Results and discussion

An initial experiment was conducted with 3-phenyl indazole **1a** and 5,5-dimethyl-2-(phenyl-λ^3-iodaneylidene)cyclohexane-1,3-dione **2a** as the model substrates, in the presence of 2.0 mol% of a $[RhCp^*Cl_2]_2$ catalyst, $AgSbF_6$ (8.0 mol%) and HOAc (2.0 equiv.) additives at room temperature (18 °C) under an air atmosphere. First, various common solvents including acetone, EtOAc, DCM, DCE, THF, 1,4-dioxane, toluene, MeOH, DMSO, DMF, hexafluoroisopropanol (HFIP) and Et_2O were tested. MeOH gave the best result and **3aa** was obtained in an almost quantitative yield of 99% in 6 h (Table 1, entry 1, also see ESI Table 1†). Next, a serial of additives was screened, and a quantitative yield was also obtained when using PivOH (entry 2). However, other additives such as NaOAc, $Cu(OAc)_2{\cdot}H_2O$, PivOCs, AgOAc and CsOAc failed to produce the desired **3aa** (entries 3–7). Thus, HOAc was selected as the best additive. Considering that the reaction temperature has an effect on the reaction rate, when the reaction was conducted at

Table 1 Optimization of reaction conditions[a]



Entry	Catalyst	Additive	T (°C)	t (h)	Yield[b] (%)
1	$[RhCp^*Cl_2]_2$	AcOH	18	6.0	99
2	$[RhCp^*Cl_2]_2$	PivOH	18	6.0	99
3	$[RhCp^*Cl_2]_2$	NaOAc	18	6.0	Trace
4	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2{\cdot}H_2O$	18	24.0	Trace
5	$[RhCp^*Cl_2]_2$	PivOCs	18	24.0	Trace
6	$[RhCp^*Cl_2]_2$	AgOAc	18	24.0	Trace
7	$[RhCp^*Cl_2]_2$	CsOAc	18	24.0	Trace
8	$[RhCp^*Cl_2]_2$	AcOH	30	5.0	99
9	**$[RhCp^*Cl_2]_2$**	**AcOH**	**38**	**0.7**	**99**
10	$[RhCp^*Cl_2]_2$	AcOH	50	0.4	98
11	$[RhCp^*(OAc)_2]_2$	—	38	24.0	94
12[c]	**$[RhCp^*Cl_2]_2$**	**AcOH**	**38**	**1.5**	**99**
13[d]	$[RhCp^*Cl_2]_2$	AcOH	38	26.0	91
14	$[RhCp^*Cl_2]_2$	—	38	24	55
15[c]	$[RhCp^*Cl_2]_2$	AcOH	38	1.5	99[e]

[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), $[RhCp^*Cl_2]_2$ (2.0 mol%), $AgSbF_6$ (8.0 mol%), and HOAc (2.0 eq.). [b] Yield based on isolation. [c] 4.0 mol% of $AgSbF_6$ was added. [d] Without added $AgSbF_6$. [e] Yield based on filtration.

30 °C, 38 °C and 50 °C, the reaction was completed in 5.0, 0.7 and 0.4 hours, respectively, giving **3aa** in 98–99% yields (entries 8–10). For the convenience of comparing between reactions, the temperature of the optimized reaction was set at 38 °C (room temperature in summer). When $[RhCp^*Cl_2]_2$ was utilized instead of $[RhCp^*(OAc)_2]_2$, without adding HOAc, **3aa** was isolated in a 94% yield after 24 h (entry 11). When the amount of $AgSbF_6$ was reduced to 4 mol%, the reaction required 1.5 h to furnish **3aa** in a 99% yield (entry 12). A control experiment without the addition of $AgSbF_6$ showed that the reaction required 26 h to furnish **3aa** in a 91% yield (entry 13). Only a moderate yield was achieved in the absence of HOAc (entry 14), which indicates that HOAC plays a role in this reaction. **3aa** can be easily purified through filtration and washing with methanol and does not need further purification with column chromatography (entry 15). We finally chose the optimized conditions in the entry 12, $[RhCp^*Cl_2]_2$ (2.0 mol%), $AgSbF_6$ (4.0 mol%), and HOAc (2.0 eq.) in 2.0 mL of MeOH, as the standard conditions for further scope studies.

With the optimal conditions established, the scope of indazoles in the reaction with iodonium ylide **2a** was explored (Table 2). Various kinds of 3-phenyl indazoles with either an electron-donating group (Me, *t*-Bu, and OMe) or an electron-withdrawing group (F, Cl, Br, NO_2 and CN) at the *para*-position of the 3-phenyl moiety reacted with **2a** to generate the desired products **3ba–3ja** in almost quantitative yields. Substrates with a *meta*-substituent afforded the desired products **3ka–3na** in excellent to almost quantitative yields, irrespective of the electronic properties of the substituent group. As for the reactions of the *meta*- and *ortho*-substituted 3-phenyl indazoles, it is

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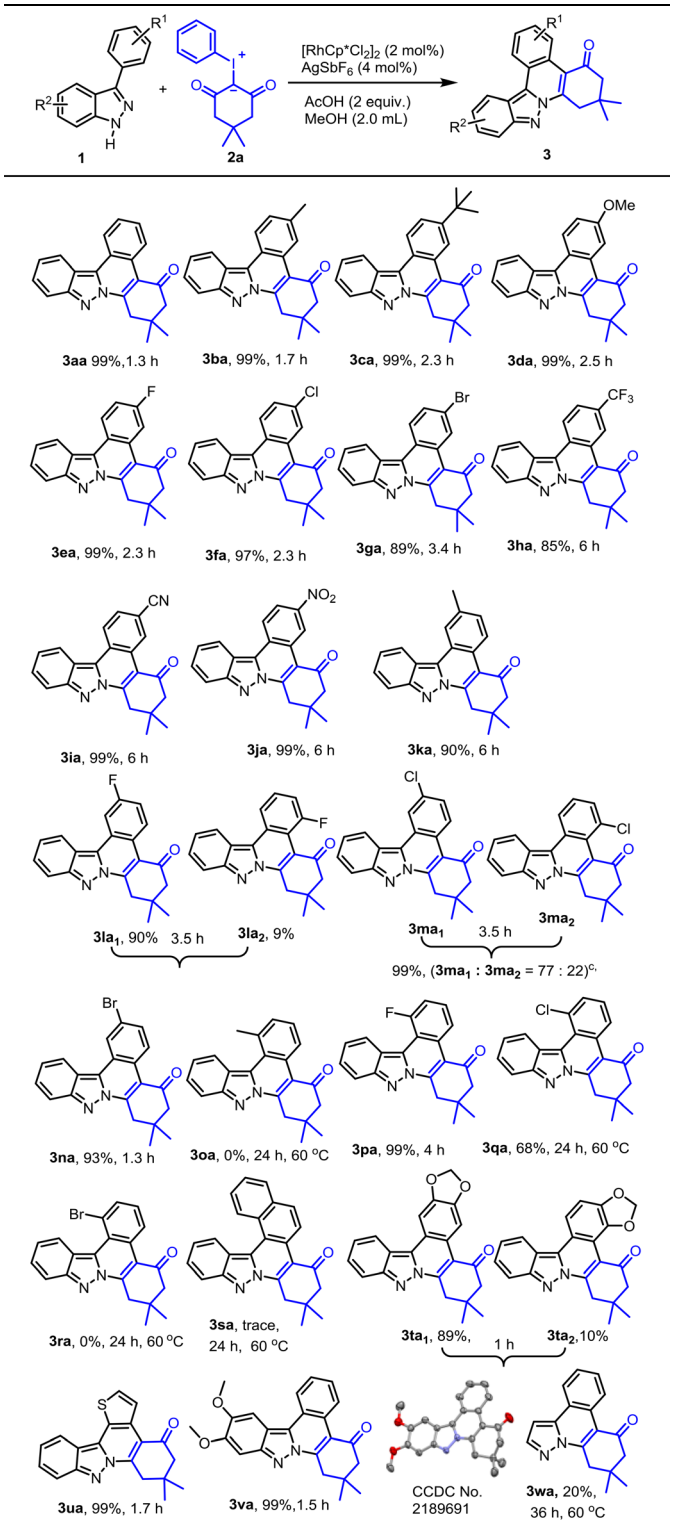
Table 2 The scope of the 3-aryl indazoles[a,b]



[a] Reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), 38 °C. [b] Yield based on isolation. [c] The ratio was determined using ^{1}H NMR.

obvious that the electronic properties of the substituents have little effect on the reaction, however, the size and position of the substituents have a greater impact on the regioselectivities. For example, substrates bearing a bulky *meta* substituent, such as a methyl or bromine group, provided the desired single products **3ka** (90%) and **3na** (93%) in excellent yields. However, the 3-phenyl indazoles with small or medium sized *meta* substituents, such as **1l** (F), **1m** (Cl) and 3-(benzo[*d*][1,3]dioxol-5-yl)-1*H*-indazole **1t**, gave the corresponding desired products in almost quantitative yields with two regioisomers obtained in each case, **3la$_1$** & **3la$_2$** (in 90% & 9% yields), **3ma$_1$** & **3ma$_2$** (in 77% & 22% yields), and **3ta$_1$** & **3ta$_2$** (in 89% & 10% yields). This may be due to the steric effect of the substituents. An almost quantitative yield was also obtained from the reaction of the substrate **1p** with a small F substituent in the *ortho*-position. A good yield (68%) of **3aq** was achieved from the reaction of the medium-sized *ortho*-Cl substituted substrate **1q** at a high reaction temperature of 60 °C. In contrast with the above success, substrates with bulky substituent in the *ortho*-position, such as CH_3 and the Br-substituted 3-phenylindazole and 3-(naphthalen-1-yl)-2*H*-indazole, failed to provide the desired annulated products **3oa**, **3ra**, and **3sa**, even at high reaction temperatures. A 3-heterocyclic substituted substrate, 3-(thiophen-2-yl)-2*H*-indazole, was also tolerated, providing the corresponding product **3au** in a 99% yield. Moreover, a substrate containing methoxy groups at the C-5 and C-6 positions of the indazole ring (**1v**) underwent annulation to furnish **3va** in a 99% yield. The structure of **3va** was confirmed by single-crystal X-ray analysis (CCDC no. 2189691†). In addition, the reaction of 3-phenyl-1*H*-pyrazole **1w** produced the corresponding product **3wa** in poor yield of 20% even at a high reaction temperature.

Subsequently, the scope of this transformation with respect to the modulation of the carbene precursors was tested (Table 3). To our delight, the reaction occurred in excellent yields in the presence of a variety of substituents at the C4 and C5 positions of the cyclohexane-1,3-dione-derived ylides (**2b–2g**). For example, Me, Ph, *p*-tolyl, and anisole were well-tolerated and afforded the desired products (**3ab–3ag**) in almost quantitative yields. The cyclopentane-1,3-dione-derived iodonium ylide (**2h**) was also well tolerated by the annulation, quantitatively giving the corresponding product **3ha**. However, (2,7-dioxocycloheptyl)(phenyl)iodonium (**2i**) only delivered the corresponding product **3ai** in a poor yield (33%) even at 60 °C.

In contrast, when 1,3-dione iodonium ylides derived from open chain compounds (**2j–2m**) were used, the reaction gave a complex mixture under the standard conditions. These results demonstrated that the transformation is sensitive to the size and nature of the dione compounds.

The recyclability of the rhodium catalyst for the synthesis of **3aa** was explored under the standard conditions (Fig. 1). It was found that the catalytic system in the organic phase can be reused at least eight times after the product has been filtered and washed with methanol. The product does need not further purification with column chromatography and the result of an ICP-MS analysis showed that the residual amount of rhodium in the product is only 20 parts per million.

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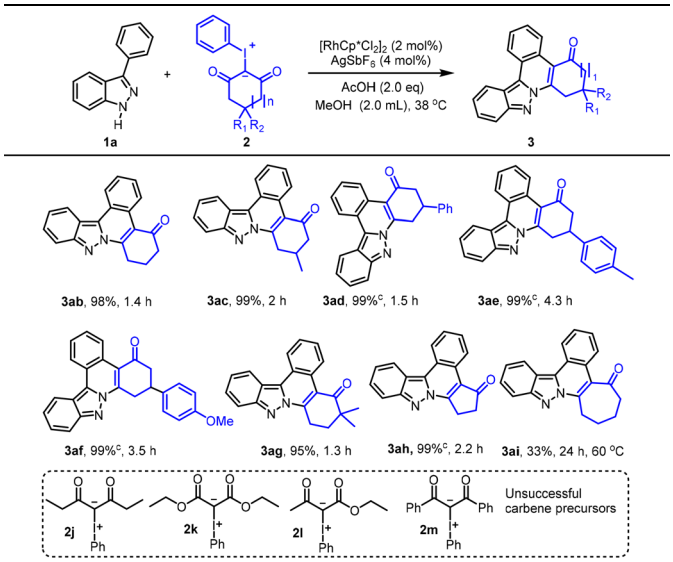
Table 3 The scope of the iodonium ylides[a,b]



[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2** (0.24 mmol, 1.2 equiv.), 38 °C. [b] Yield based on isolation. [c] Yield based on filtration and subsequent washing with methanol and petroleum. Compounds **3ac**, **3ad**, **3af**, and **3ah** are insoluble in common solvents, and are difficult to be separated and purified by column chromatography or crystallization. Therefore, these compounds were separated using direct filtration and were washed with methanol and petroleum. Due to their sparing solubility in solvents, these compounds were characterized using NMR with extended scanning times.

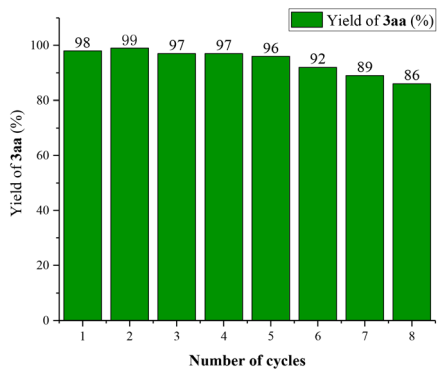
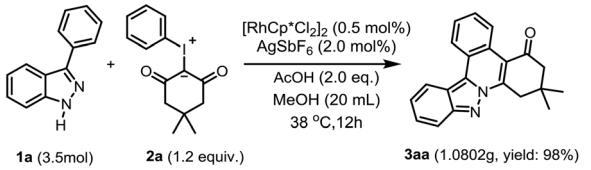


Fig. 1 The recyclability of the rhodium catalyst.

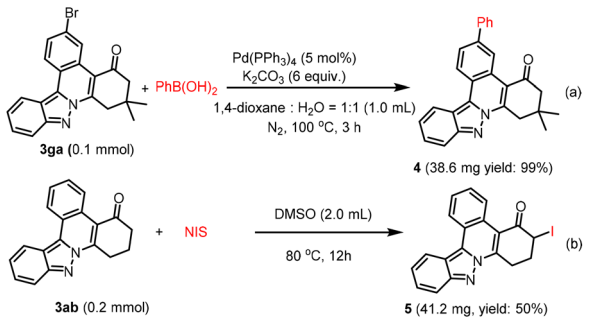
In order to verify the effective application of this protocol, a model reaction on the gram scale was performed. When 3.5 mmol of substrate **1a** was introduced with 0.5 mol% of $[RhCp^*Cl_2]_2$ and 2 mol% of $AgSbF_6$, 1.0802 g of the target product **3aa** was obtained in a 98% yield. The product was isolated *via* a simple filtration, washing with methanol and without the need for further purification *via* column chromatography. The purity of the product is sufficient for NMR, UV, and fluorescence characterization. The ^1H NMR and fluorescence data of the product were not substantially different from those of the same product purified *via* column chromatography (see Fig. S1–S3†). Thus, it was found that this strategy may be amenable to industrial production (Scheme 2).



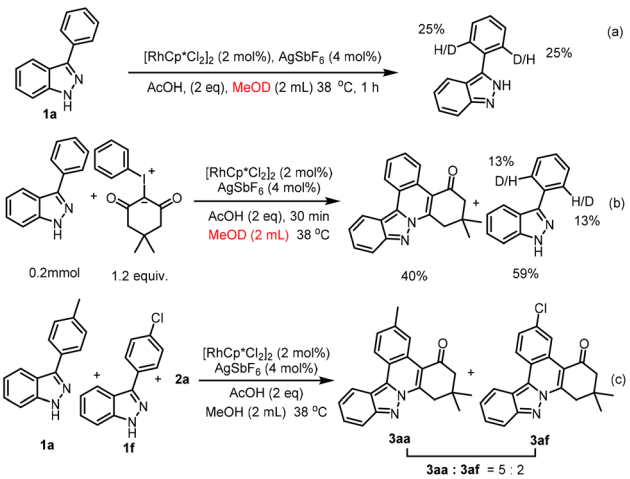
Scheme 2 A model reaction on a gram scale.

Furthermore, several derivatization reactions were conducted. The coupling reaction of **3ga** with $PhB(OH)_2$ gave product **4** in a 99% yield (Scheme 3a). In addition, the selective iodization of **3ab** with NIS gave product **5** in a 50% yield (Scheme 3b).

To gain insights into the reaction mechanism, some control experiments were carried out (Scheme 4). First, deuterium labelling experiments were performed. The reaction of **1a** in CH_3OD (2.0 mL) was conducted in the absence of an iodonium ylide under the standard conditions. 25% deuteration at the *ortho*-position of **1a** was observed based on the ^1H-NMR analysis, which reveals that the C–H bond activation step is reversible (Scheme 4a). When the full reaction was conducted in CH_3OD (2.0 mL) under standard conditions for 30 minutes, a 40% yield of **3aa** was isolated and 59% of **1a** was recycled with 13% of the *ortho*-C–H deuterated (Scheme 4b), implying



Scheme 3 Derivatization reactions.



Scheme 4 Control experiments for investigation of the mechanism.

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that the coordination and insertion of **2** is fast and irreversible. Furthermore, a competitive experiment was performed between 3-(*p*-methyl)phenyl indazole **1a**, with an electron-donating group, and 3-(*p*-chlorophenyl)indazole **1f**, with an electron-withdrawing group, with **2a** and the preferential formation of **3aa** was observed (**3aa** : **3fa** = 5 : 2, Scheme 4c). This indicated that the C–H activation process is an electrophilic process and the rate determining step.

Based on the above experimental results and literature reports,[12–14] a plausible reaction mechanism was proposed and is shown in Scheme 5. First, an active catalyst [RhCp*$(SbF_6)_2$] is generated from ligand exchange, and then undergoes direct C–H activation to afford a five-membered intermediate **A**. The coordination of iodonium ylide **2a** to the metal center in **A**, followed by the loss of iodobenzene, affords the metal carbene species **B**. Subsequently, the migratory insertion of the carbene into the Rh–C bond produces intermediate **C**, which undergoes protonation to deliver intermediate **D** and regenerate the active catalyst. Finally, nucleophilic addition and elimination occur between the amine and ketone of **D** to deliver the desired product **3aa**.

Photophysical properties

Organic compounds consisting of a fused heterocyclic π structure often show distinct photophysical properties. In our previous works, we found that indazolo[3,2-*a*]isoquinoline derivatives exhibit photoluminescence both in solution and in the solid state with high quantum yields, and that they are promising candidates for use as fluorescent materials and biological imaging reagents.[16] Thus, in addition to the synthesis of dihydroindazolo[2,3-*f*]phenanthridin-5(6*H*)-ones, we are also interested in their photophysical properties and applications.

Thus, the UV-Vis absorption and fluorescence emission spectra of selected compounds **3aa–3ja**, **3la$_1$**, **3la$_2$**, and **3pa** have been measured in dilute DCM at room temperature (Fig. 2) to evaluate their light absorption and fluorescent emission abilities. Their photophysical properties are summarized in Table S3.† From Fig. 2, we can see that there are two absorption bands in their UV-Vis spectra, one around 301 nm that originates from the n → π* transition and the next around 406 nm that may be due to intramolecular charge transfer. In comparison with **3aa**, the introduction of a typical electron-donating group (EDG) –OMe in **3da** leads to a prominent red-shift (from 406 to 424 nm).

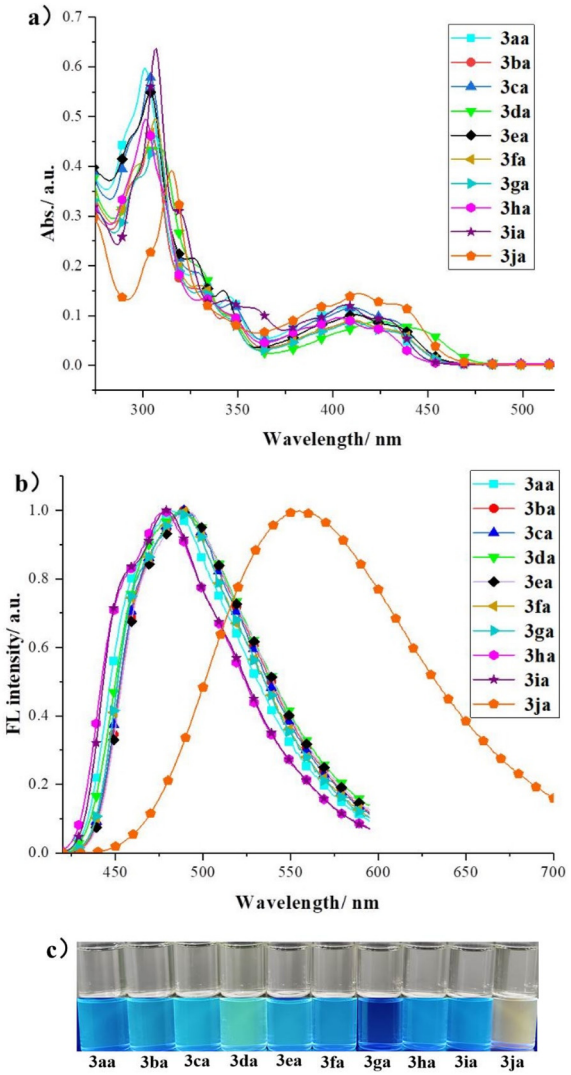
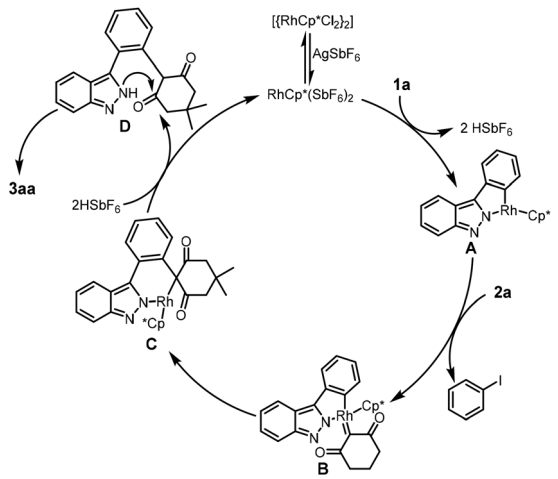


Fig. 2 (a) The UV-Vis absorption and (b) normalized emission spectra of **3aa–3ja** in DCM solution. (c) The DCM solutions of selected compounds on exposure to daylight and irradiation with 365 nm UV-Vis light.



Scheme 5 A plausible reaction mechanism.

Also from Fig. 2, we find that **3aa** has a sky-blue emission in solution, and all selected compounds exhibit greenish-blue emissions with maxima bands around 481 nm. Meanwhile, the relevant fluorescence data showed that product **3da**, bearing a typical electron-donating group –OMe, showed a green emission with the highest fluorescence quantum yield of 0.5044 (50.44%). In contrast, **3ga** with a Br atom has the lowest quantum yield due to the quenching "heavy atom

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effect". Interestingly, **3aj**, substituted with a strong electron-withdrawing group $-NO_2$, also shows a yellow-orange emission with decent quantum yield of 0.3119 (31.19%) and an emission maximum at 553 nm. The Stokes Shift of **3aj** was 140 nm (Fig. S3†), which is the largest of these compounds. This remarkable red-shift can be attributed to enhancement of the excited-state internal charge transfer of **3aj**[17] that arises from the structural features of the substrate (Fig. S4†).

From Fig. S5,† it can be seen that the emission properties of **3aa** could be fine-tuned through the incorporation of electron-withdrawing fluorine atoms at different positions on the indazole backbone. The fluorescence quantum yield of **3aa** is 0.2133 (21.33%), while that of **3pa** is 0.1730 (17.30%), in $\mathbf{3la_1}$ it is 0.1828 (18.28%), in $\mathbf{3la_2}$ it is 0.1507 (15.07%), and **3ea** has the highest quantum yield of 0.2892 (28.92%). In Fig. S6,† it can be seen that, as the position of the F atom moves, the colour of the F-containing derivatives can be tuned from blue to green. Other related parameters of these four compounds, including the CIE values, have been summarized in Table S4.†

These results demonstrate the potential applications of the dihydroindazolo[2,3-*f*]phenanthridin-5(6*H*)-ones in the fields of medicinal chemistry and materials research as probes of the cellular environment.

Conclusions

In summary, we have demonstrated an effective approach for the construction of dihydroindazolo[2,3-*f*]phenanthridin-5(6*H*)-ones under mild conditions. In this transformation, a series of excellent to almost quantitative yields were obtained *via* the Rh(III)-catalyzed C–H activation of 3-arylindazoles and their annulation with readily available hypervalent iodonium ylides. In addition, the desired products were obtained without the need for column chromatography, and only a simple filtration and washing with methanol were required. The catalyst system can be recycled at least eight times with excellent yields, which may allow this method to have potential in industrial production.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

We thank the National Natural Science Foundation of China (No. 21762020 and 22261027) for financial support.

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