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Rhodium-catalyzed formal [4 + 3] annulation reaction of *N*-methoxybenzamides with *gem*–difluorocyclopropenes: A combination of experimental and theoretical studies

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ABSTRACT

A rhodium-catalyzed [4+3] cycloaddition reaction between *N*-methoxybenzamides and *gem*–difluorocyclopropenes is described. The reaction offers a mild and efficient approach towards the synthesis of fluorinated 2*H*-azepin-2-ones with broad substrate scope. A consecutive HOAc-assisted C–N bond formation and fluorine elimination are involved as key steps for success as illustrated by detailed DFT studies.

Transition metal-catalyzed C–H bond functionalization reactions are among the most straightforward and atom-economic synthetic methodologies for the construction of complex molecules [1–4]. Typically, a directing group is need to facilitate a regioselective C–H activation event. And the use of oxidizing directing group has received tremendous attentions by offering enhanced reactivity and eliminating the employment of external oxidant [5–12]. In this respect, various five- and six-numbered rings are mildly and effectively constructed through the oxidizing directing group strategy. For example, an elegant seminal work from Fagnou reported a Rh(III)-catalyzed redox-neutral annulation of benzhydroxamic acids with alkynes towards the synthesis of isoquinolone derivatives by using N–O bond as a built-in oxidant (Scheme 1a) [13]. However, since the eight- membered rhodacycle intermediates are energetically unstable, utilizing this tactics to construct seven-membered rings is elusive [14,15].

Fluorinated organic molecules have attracted significant attention in drug discovery and agricultural chemistry due to their unique physicochemical and bioactivity properties [16–18]. Traditional methods for the incorporation of fluorine into the molecules often suffer from the need of substrate pre-activation, the use of non-readily available starting materials, low regio- or stereoselectivity and/or poor functional group tolerance due to the employment of sensitive reagents [19–21]. Compared with the above-mentioned protocols, transition metal-catalyzed C–H/C-F bond activation assisted by a directing group provides a concise and reliable alternative in an atom- and step-economic pattern. In this context, the group of Loh reported a Rh^{III}-catalysed tandem C–H/C-F activation for the synthesis of (hetero)arylated monofluoroalkenes using *gem*–difluoroalkenes as electrophiles (Scheme 1b-1) [22–24]. The group of Wang disclosed a solvent-dependent enantioselective synthesis of alkynyl and monofluoroalkenyl isoindolinones by asymmetric $CpRh^{III}$-catalyzed C–H activation with α,α-difluoromethylene alkyne as the substrate (Scheme 1b-2) [25–28]. In these two cases, metal-mediated β-fluorine elimination was observed as key step. Previously, we discovered that different directing groups (*N*–OMe and *N*–OPiv amides) enabled dictate the selectivity of C–N formation *versus* β-F elimination with 2,2-difluorovinyl tosylate as a substrate (Scheme 1c) [29–33]. With *N*–OMe benzamide being a directing group (DG), the reaction delivered a monofluorinated alkene with the retention of the to-

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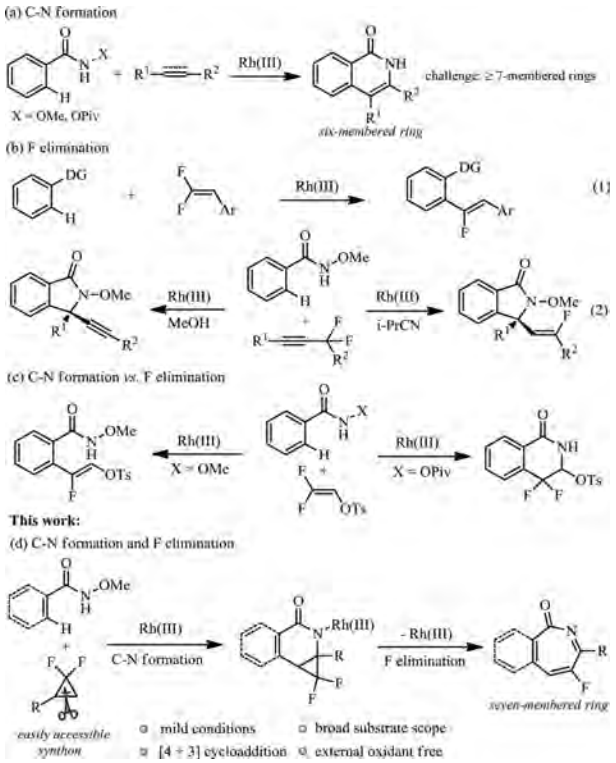


Scheme 1. Rhodium-catalyzed C–H activation reactions toward the organofluorines with an oxidizing directing group.

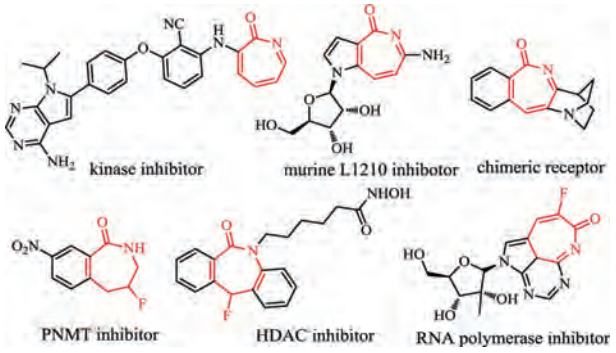
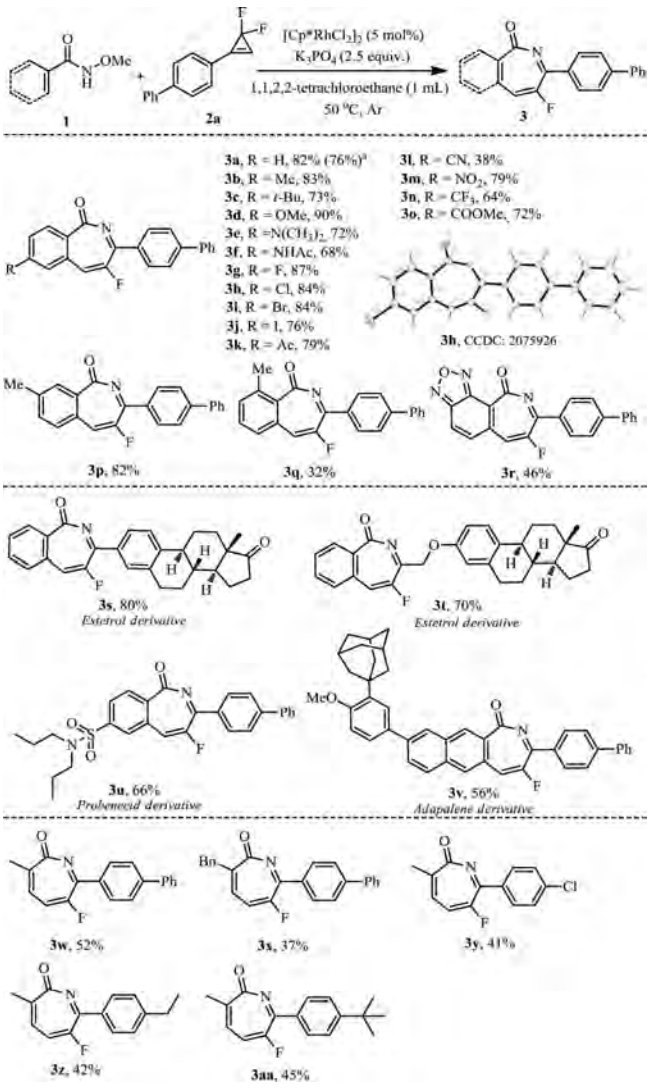


Fig. 1. Representative bioactive molecules.



Scheme 2. Substrate scope on amides. Reaction conditions: **1** (0.2 mmol), **2** (0.24 mmol), $[Cp^*RhCl_2]_2$ (5 mol%), K_3PO_4 (2.5 equiv.), 1,1,2,2-tetrachloroethane (1 mL) at 50 °C under argon atmosphere for 12 h. Isolated yield. [a] **1a** (10 mmol), **2a** (12 mmol), $[Cp^*RhCl_2]_2$ (5 mol%), K_3PO_4 (2.5 equiv.), 1,1,2,2-tetrachloroethane (10 mL) at 50 °C under argon atmosphere for 12 h.

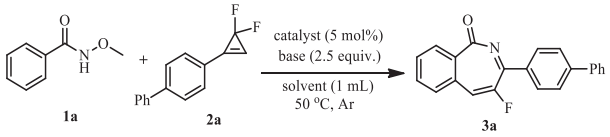
sylate functionality. When *N*–OPiv benzamides were used, however, [4 + 2] cyclization occurred to provide *gem*–difluorinated dihydroisoquinolin-1(2*H*)-ones.

Herein, we report a rhodium-catalyzed formal [4 + 3] cycloaddition reaction of *N*-methoxybenzamides with easily accessible *gem*–difluorocyclopropenes [34–49]. The reaction allows the formation of highly functionalized fluorinated 2*H*-azepin-2-one frameworks with excellent regioselectivity and functional group tolerance (Scheme 1d). Some interesting features of the transformation include: i) Both C–N bond formation and fluorine elimination occur in the reaction with *N*–OMe as an internal oxidant; ii) The combination of [4 + 2] cycloaddition and retro-[2 + 1] strategy eliminates the formation of eight-membered rhodacycle, thereby providing a feasible and reliable route for the construction of seven-membered aromatic heterocycles; iii) This reaction proceeds under rather mild conditions, and a series of bioactive fluorinated 2*H*-azepin-2-one derivatives (Fig. 1) [50–52] are obtained in moderate to good yields.

The reaction was initially investigated by using *N*-methoxybenzamide **1a** and *gem*–difluorocyclopropene **2a** as model substrates, $[Cp^*RhCl_2]_2$ as catalyst and K_3PO_4 as base in CH_2Cl_2 at 50 °C under argon atmosphere. To our delight, the desired product **3a** was obtained in 41% yield (Table 1, entry 1). Solvent screening revealed that only chlorinated alkanes promoted the transformation, with 1,1,2,2-tetrachloroethane being the solvent of choice to give a high yield of to 82% (Table 1, entries 2–7). Replacing $[Cp^*RhCl_2]_2$ with $Cp^*Rh(OAc)_2$ led to a reduced yield of 61% (Table 1, entry 8). The reaction did not proceed with other transition metal catalysts such as $[Cp^*IrCl_2]_2$, $[RuCl_2(p\text{-cymene})]_2$ and $[CoCp^*(CO)I_2]$ (Table 1, entries 9–11). Other inorganic bases were also subsequently used in the reaction, however, the yield of **3a** was not further improved (Table 1, entries 12–16).

With optimized conditions in hand, we set to examine the scope. As shown in Scheme 2, the aromatic amide substrates bearing electron-donating (such as Me, *t*-Bu, OMe and $N(CH_3)_2$) and electron-withdrawing substituents (such as Ac, CN, NO_2, CF_3, CO_2Me) at *para* position underwent reaction smoothly, delivering the cyclized products **3a**-**3f** and **3k**-**3o** in good yields. Sub-

Table 1
Optimization of reaction conditions.[a]



Entry	Catalyst	Base	Solvent	Yield of **3a** (%)[b]
1	$[Cp^*RhCl_2]_2$	K_3PO_4	CH_2Cl_2	41
2	$[Cp^*RhCl_2]_2$	K_3PO_4	CH_2ClCH_2Cl	54
3	$[Cp^*RhCl_2]_2$	K_3PO_4	$CHCl_2CHCl_2$	82
4	$[Cp^*RhCl_2]_2$	K_3PO_4	THF	n.d.
5	$[Cp^*RhCl_2]_2$	K_3PO_4	Toluene	n.d.
6	$[Cp^*RhCl_2]_2$	K_3PO_4	CH_3CN	n.d.
7	$[Cp^*RhCl_2]_2$	K_3PO_4	DMF	n.d.
8	$Cp^*Rh(OAc)_2$	K_3PO_4	$CHCl_2CHCl_2$	61
9	$[Cp^*IrCl_2]_2$	K_3PO_4	$CHCl_2CHCl_2$	n.d.
10	$[RuCl_2(p\text{-cymene})]_2$	K_3PO_4	$CHCl_2CHCl_2$	n.d.
11	$[CoCp^*(CO)I_2]$	K_3PO_4	$CHCl_2CHCl_2$	n.d.
12	$[Cp^*RhCl_2]_2$	Na_3PO_4	$CHCl_2CHCl_2$	71
13	$[Cp^*RhCl_2]_2$	Li_3PO_4	$CHCl_2CHCl_2$	47
14	$[Cp^*RhCl_2]_2$	KOAc	$CHCl_2CHCl_2$	65
15	$[Cp^*RhCl_2]_2$	NaOAc	$CHCl_2CHCl_2$	57
16	$[Cp^*RhCl_2]_2$	K_2CO_3	$CHCl_2CHCl_2$	52

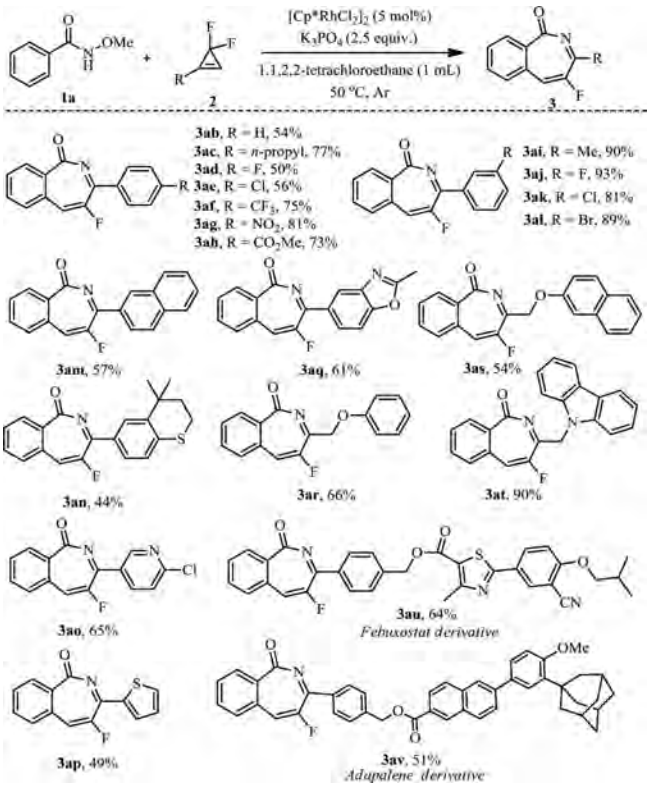
[a] Reaction conditions: **1a** (0.2 mmol), **2a** (1.2 equiv.), catalyst (5 mol%), base (2.5 equiv.), solvent (1 mL), 50 °C, under argon atmosphere, 12 h.
[b] Isolated yields.

strates bearing halogen substituents were also compatible well (**3g**-**3j**), thus providing valuable handles for follow-up transformations. When *meta*-substituted *N*-methoxyamide **1p** was used, the rhodation occurred at the less hindered site to provide exclusively the C-6 substituted regioisomer (**3p**). Specially, the 2-methyl substituted substrate did not retard the process (**3q**), although the yield was slightly reduced. To further highlight the synthetic versatility of our method, several substrates derived from complex natural products and drugs were also subjected to the reaction and the corresponding fluorination products were obtained without difficulty (**3s**-**3v**). Furthermore, various alkenyl amides with 2-alkyl substitution were also feasible substrates, producing the products **3w**-**3aa** in moderate yields. Out of our expectation, *N*–methoxy-2-phenylacrylamide showed no reactivity in the protocol. To demonstrate the scalability of this methodology, the reaction of **1a** with **2a** was performed on 10 mmol scale, affording 76% yield of the product **3a**.

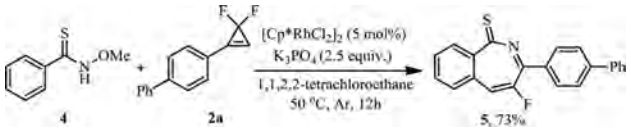
The substrate scope for *gem*–difluorocyclopropenes was also explored (Scheme 3). It was found the reaction was not sensitive to the electron nature of the substituents on the aryl ring, as a diverse of substituents such as Me, *n*-Pr, F, Cl, Br, CF_3, NO_2 and CO_2Me well survived in the reaction (**3ab**-**3al**). Interestingly, higher yields were obtained for *meta*-substituted aryl *gem*–difluorocyclopropenes (**3ai**-**3al**). The observed higher bench stability of *meta*-substituted aryl *gem*–difluorocyclopropenes could be a reason for this result. Not unexpectedly, substrates with other aromatic heterocycles, for example, thiochroman, pyridine, thiophene and benzoxazole were also tolerant, obtaining the products **3an**-**3aq** in 44%–65% yields. It was intriguing that alkyl-substituted *gem*–difluorocyclopropenes were also compatible (**3ar**-**3av**), greatly expanding the diversity of the title reaction.

Also interesting was the applicability of *N*-methoxybenzothioamide **4** in the reaction. The cyclization reaction proceeded smoothly to give the desired product **5** in 73% yield (Scheme 4).

When *N*–methoxy-2-naphthamide **6** was used in the reaction, the 3-position C–H bond with less steric hindrance was exclusively functionalized to give the product **7** in 72% yield (Scheme 5a). Interestingly, treatment of *N*-methoxybenzo[*d*][1,3]dioxole-5-carboxamide **8** with **2a** provided the 4-position annulation product **9** in 78% yield (Scheme 5b). The later could be explained by a coordination effect between the oxygen atom at the 3-position and the transition metal.



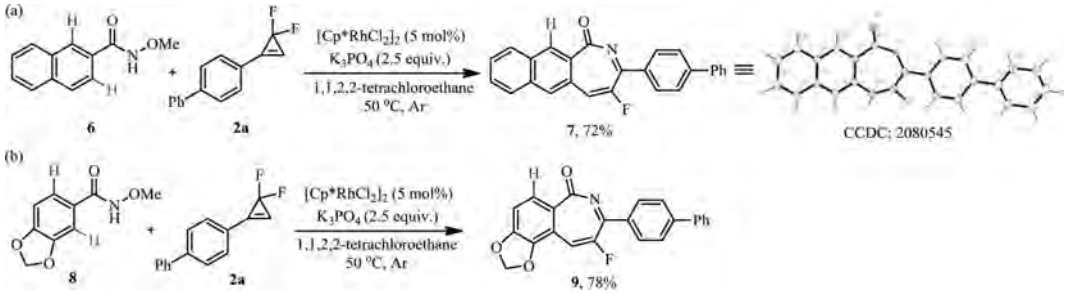
Scheme 3. *gem*–Difluorocyclopropene scope. Reaction conditions: **1** (0.2 mmol), **2** (0.24 mmol), $[Cp^*RhCl_2]_2$ (5 mol%), K_3PO_4 (2.5 equiv.), 1,1,2,2-tetrachloroethane (1 mL) at 50 °C under argon atmosphere for 12 h. Isolated yield.



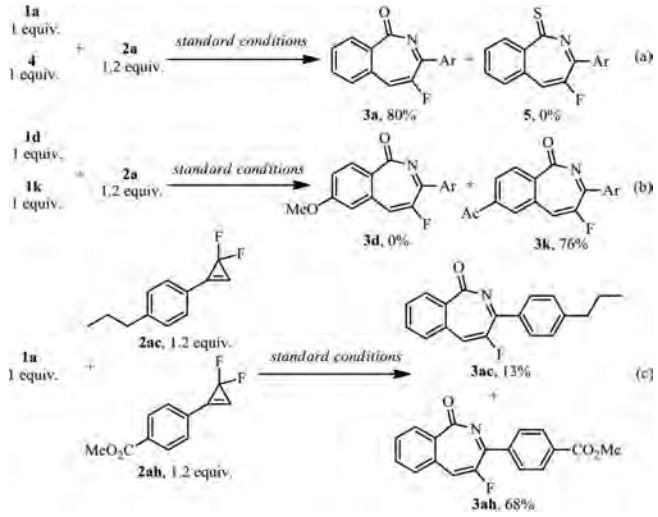
Scheme 4. Annulation reaction of **4** with **2a**.

Intermolecular competitive reactions were performed to understand the reactivity of *N*-methoxybenzamides and *gem*–difluorocyclopropenes (Scheme 6). Treatment of *N*-methoxybenzamide **1a** and *N*-methoxybenzothioamide **4** with **2a** under the standard conditions gave only **3a** in 80% yield (Scheme 6a). Competition reaction of 4–methoxy-*N*-methoxybenzamide **1d** and 4-acetyl-*N*-methoxybenzamide **1k** with **2a** gave exclusively **3k** in 76% yield (Scheme 6b). This result demonstrated that the benzamide substrates with electron-donating substituents are less reactive. Furthermore, when **1a** was treated with **2ac** and **2ah**, the corresponding products **3ac** and **3ah** were isolated in 13% and 68% yields, respectively, suggesting that electron-poorer **2ah** is good for the reaction (Scheme 6c).

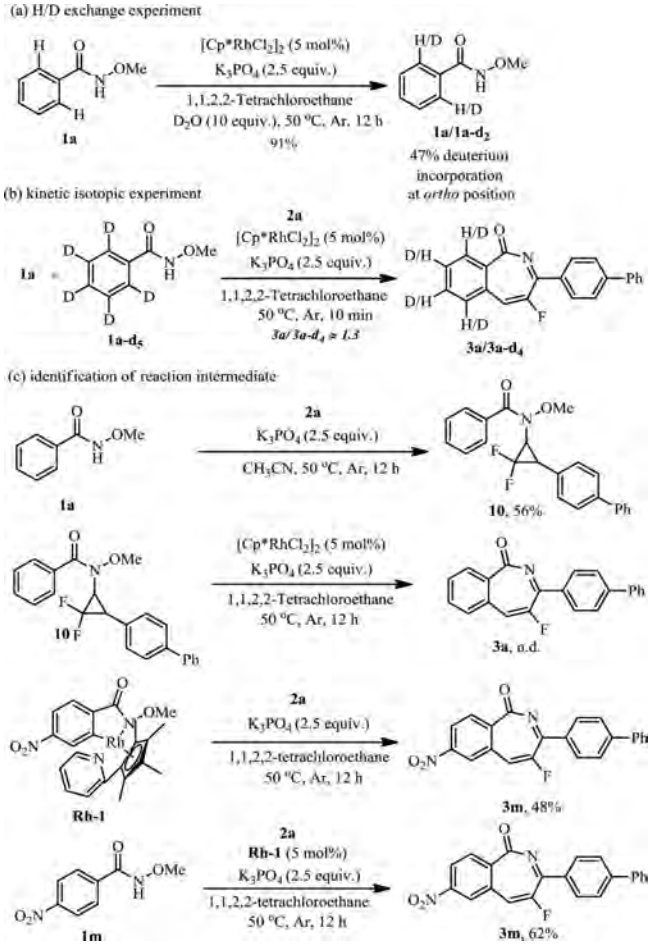
To further probe the mechanism, several control experiments were conducted (Scheme 7). When D_2O was added to the reaction in the absence of **2a**, a 47% deuterium incorporation at *ortho* position of **1a** was observed without N–O bond cleavage (Scheme 7a). And a kinetic isotope effect (KIE) value of $k_H/k_D = 1.3$ was observed (Scheme 7b). These results suggested that the C–H bond cleavage is reversible and not be involved in the turnover-limiting step. A hydroamination product **10** was unexpectedly obtained in the absence of rhodium catalyst (Scheme 7c). However, this compound



Scheme 5. Regioselective reactions.



Scheme 6. Intermolecular competitive reactions.



Scheme 7. Mechanism studies.

was demonstrated not to be an effective intermediate for the title reaction. A rhodacycle **Rh-1** was prepared and its intermediacy in the reaction was confirmed by stoichiometric and catalytic reactions, suggesting a C–H activation took place.

To further cast light on the mechanism, theoretical calculations were performed at the density functional theory level (B3LYP). For the convenience of calculation, the active catalyst $Cp^*Rh(OAc)_2$ was chosen as the starting point (zero value of energy). Using *N*–methoxy benzamide **1a** as a substrate, N–H deprotonation followed by C–H activation were performed *via* a concerted metalation-deprotonation (CMD) mechanism with acetate acting as intramolecular base, through transition states **TS-1** ($\Delta G^{\ddagger} = 13.0$ kcal/mol) and **TS-2** ($\Delta G^{\ddagger} = 19.8$ kcal/mol), respectively (Fig. 2). The intermediate verification experiments in Scheme 7c echoed the calculation results. Thereafter, the insertion of *gem*–difluorocyclopropene **2ai** into the rhodacycle **INT-5** presented two characteristic spatial arrangements, **TS-3** ($\Delta G^{\ddagger} = 27.5$ kcal/mol) and **TS-3′** ($\Delta G^{\ddagger} = 28.9$ kcal/mol), both of which had a higher activation barrier than the first two steps (Fig. 3). The computational results indicated that C–H activation was not the turnover limiting step in the reaction, consistent with the observed small experimental KIE values (Scheme 7b). Taking into account the higher energy barrier of **TS-3′**, especially **TS-4′**, therefore subsequent calculations revolved around **TS-3**. From **INT-7**, the priority of either β-fluorine elimination or C–N bond formation was discussed. The results revealed that the direct β-fluorine elimination with or without the assistance of acetic acid, followed by C–N bond formation step *via* **TS-4a** and **TS-4b**, featured a high energy barrier of 33.2 and 35.1 kcal/mol, reaspectively. Two possible pathways for C–N bond formation prior to the defluorination were then calculated. Considering the high energy barrier of **TS-4c** ($\Delta G^{\ddagger} = 68.6$ kcal/mol), the direct migration of the methoxy group from the amide to the trivalent rhodium to form **INT-9c** was tough. The migration process was more reasonable in the assistance of acetic acid, because the energy barrier of **TS-4** was reduced to 28.8 kcal/mol and a Rh(V) intermediate **INT-10** was produced with the free-energy of −54.3 kcal/mol. the synergistic effect of rhodium and acetate accelerated the ring-opening defluorination of **INT-10** to release the final product **3ai** ($\Delta G^{\ddagger} = -71.0$ kcal/mol). Overall, the computed Gibbs free-energy changes of the reaction pathway demonstrated a redox-neutral Rh(III)-Rh(V)-Rh(III) catalytic cycle for the developed protocol involving HOAc-prompted oxidative addition and unprecedented C-F bond cleavage/ring expansion processes.

On the basis of the above studies and previous reports [53–57], a plausible mechanism is proposed in Scheme 8. A ligand ex-

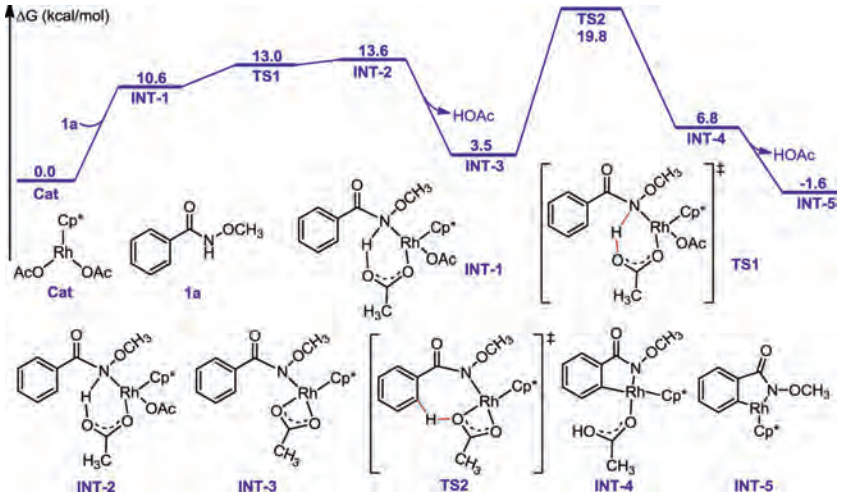


Fig. 2. Computed pathways for N–H and C–H activation.

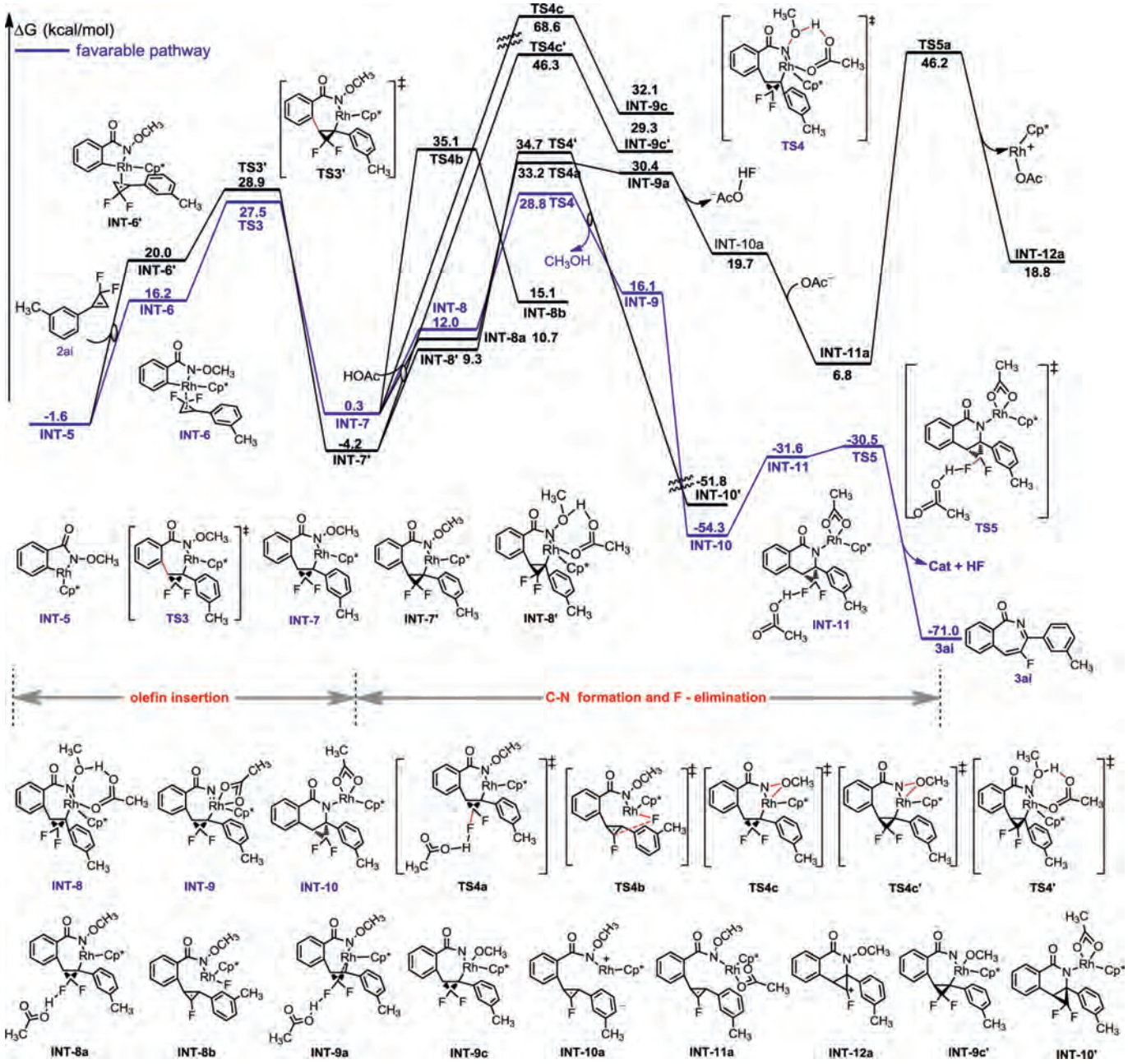
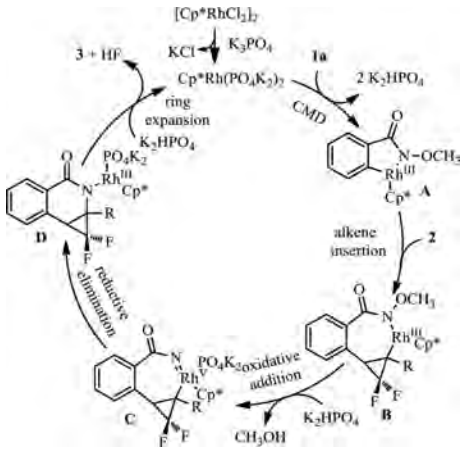


Fig. 3. Computed pathways for C–N formation and F-elimination.



Scheme 8. Possible catalytic cycle.

change between $[Cp^*RhCl_2]_2$ and K_3PO_4 forms a reactive catalyst. Rhodacycle A is then formed *via* consecutive N–H and ortho C–H bonds activation. These processes may occur *via* a CMD (concerted metalation deprotonation)-like mechanism in the aid of internal PO_4^{3-} base [29]. Afterwards, a migratory insertion of the rhodacycle **A** into *gem*–difluorocyclopropene **2** delivers the intermediate **B**. Rh(III) in intermediate **B** is oxidized to Rh(V) nitrenoid intermediate **C** in the aid of K_2HPO_4. Rh(V) intermediate **C** returns to Rh(III) through a C–N migratory insertion into the nitrenoid. Finally, the synergistic effect of rhodium and K_2HPO_4 accelerates the ring-opening defluorination to release the product **3**.

In summary, we developed a novel [4 + 3] cycloaddition reaction of *N*-methoxyamides with *gem*–difluorocyclopropenes, enabling a modular, concise and efficient approach for accessing highly functionalized fluorine-substituted 2*H*-azepin-2-ones in moderate to good yields. Other appealing features include simple and readily available substrates, mild conditions and broad substrate scope. DFT studies revealed a consecutive C–N bond formation and fluorine elimination events in the annulation reaction. Given the importance of 7-membered heterocycles as well as fluorine atom in medicinal chemistry, we anticipate this protocol will find applications. During the preparation of this work, Yi and Zhou reported a similar work [58].

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.

Acknowledgments

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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.2022.01.068.

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