Chinese Chemical Letters 34 (2023) 108014

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

Rh(III)-Catalyzed annulative aldehydic C-H functionalization for accessing ring-fluorinated benzo[*b*]azepin-5-ones

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ARTICLE INFO

Article history:
Received 26 August 2022
Revised 11 November 2022
Accepted 20 November 2022
Available online 23 November 2022

Keywords:
Rh(III) catalysis
Aldehydic C-H activation
[4+3] annulation
Benzo[*b*]azepin-5-ones
Gem-difluorocyclopropenes

ABSTRACT

A new Rh(III)-catalyzed aldehydic C-H activation/[4+3] annulation cascade of *N*-sulfonyl-2-aminobenzaldehydes with *gem*-difluorocyclopropenes is reported for the first time, and used to produce a range of hitherto unreported precedented β-monofluorinated benzo[*b*]azepin-5-ones with good yields and complete regioselectivity. This approach features a broad substrate scope, good functional group tolerance, and high regioselectivity, which may include Rh(III)-catalyzed aldehydic C−H activation, tandem site-/regioselective insertion, defluorinated ring-scission, and 1,2-elimination.

A new Rh(III)-catalyzed aldehydic C-H activation/[4+3] annulation cascade of *N*-sulfonyl 2-aminobenzaldehydes with *gem*-difluorocyclopropenes is reported for the first time, and used to produce a range of hitherto unreported precedented β-monofluorinated benzo[*b*]azepin-5-ones with good yields and complete regioselectivity.

The past two decades have witnessed the rapid development of Rh(III)-catalyzed C(sp^2)−H activation reactions as powerful synthetic tools for fabricating valuable medicinally active substances and natural products with step- and atom-economy [1–5]. Among them, Rh(III)-catalyzed annulative C(sp^2)−H functionalization has been recognized as a versatile platform for forming heterocyclic amides or ketones. A survey of the literature shows that there are two main modes in the direct activation of C(sp^2)−H bonds for the synthesis of these compounds, one is Rh(III)-catalyzed annulative amide-oriented C(sp^2)−H activation with various coupling reagents toward lactam derivatives, which include [3+3] [6], [4+1] [7–15,22], [4+2] [16–33], or [4+3] [34–38] annulations (Scheme 1a), and the other is Rh(III)-catalyzed annulative aldehydic C(sp^2)−H activations with different coupling reagents by using a β-amino group as the directing group [39], providing a direct protocol for heterocyclic ketone synthesis. However, the latter only allows [4+1] [40,41] and [4+2] [42,43] annulation processes for constructing normal 5- and 6-numbered *N*-heterocyclic ketones; in sharp contrast, a catalytic [4+3] annulation of these C–H substrates with three-carbon donors to give 7-numbered benzannulated *N*-heterocyclic ketones has rarely been explored.

Benzannulated medium-sized *N*-heterocycles are widely distributed in bioactive natural products and pharmaceutical agents [44]. In particular, representing a unique class of seven-member *N*-heterocycles, compounds featuring a benzoazepinone skeleton [45–49], such as benzazepinediones [46], Tolvaptan [47] and Lotensin [48], demonstrate a wide range of biological activities (Fig. 1). On the other hand, organic molecules with fluorine or fluorine-containing groups usually enhance the solubility and lipophilicity to improve membrane permeability and bioavailability, especially those with ring-fluorinated heterocycles [50–60]. Consistent with these findings, we think that developing new methods for synthesizing compounds incorporating both benzoazepine and fluoro fragments is a required technology. This can be understood, on the one hand, by the existence of such a skeleton in natural products with biological activities [45–49], and on the other hand, by the potential of using these molecules for downstream studies in chemical biology and medicinal chemistry. Very recently, Zhou [61] and He [62] independently reported an elegant rhodium-catalyzed [4+3] annulation reaction of *N*-methoxybenzamides and

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https://doi.org/10.1016/j.cclet.2022.108014
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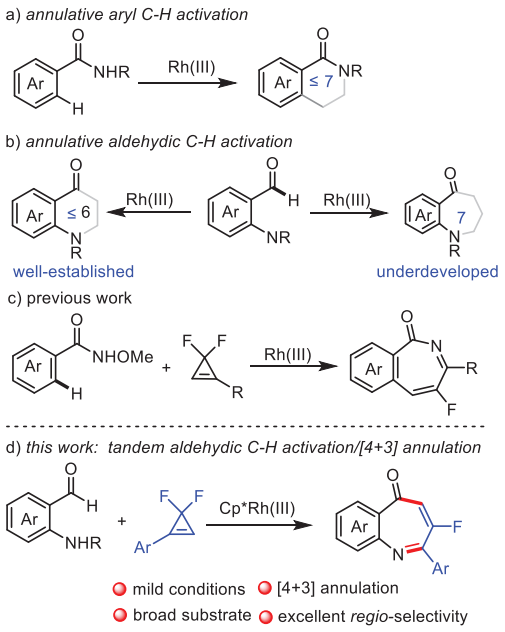


Scheme 1. Profiles of Rh(III)-catalyzed C(sp^2)–H activation.

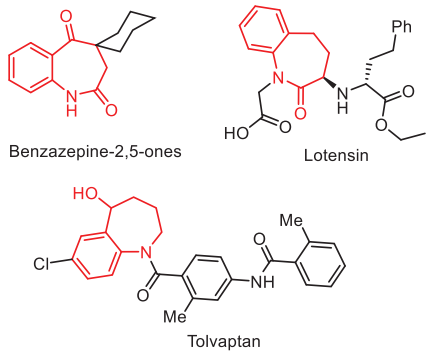
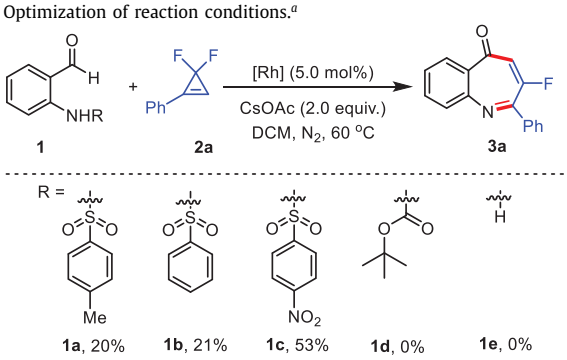


Fig. 1. Selected examples of bioactive benzoazepines.

gem–difluorocyclopropenes, leading to the straightforward formation of ring-fluorinated 2*H*-azepin-2-ones (Scheme 1c). Inspired by these impressive reports [61,62] and as part of ongoing efforts in developing metal catalysis [63–67], we questioned whether Rh(III)-catalyzed aldehyde C(sp^2)–H activation could be harnessed to establish a new [4 + 3] annulation with *gem*–difluorocyclopropenes for the synthesis of ring-fluorinated benzo[*b*]azepin-5-ones. Unfortunately, synthesizing this skeleton faces several challenges such as precise site-selective activation, control of the regiochemistry and unfavorable transannular interactions of medium-sized rings. To our delight, a Rh(III)-catalyzed [4 + 3] annulation of *N*-sulfonyl-2-aminobenzaldehydes with *gem*-difluorocyclopropenes proceeded smoothly through an aldehydic C-H activation process, leading to the formation of the hitherto unreported β-monofluorinated benzo[*b*]azepin-5-ones in good yields, with complete regiochemistry control (Scheme 1d). Herein we elaborate on this attractive observation.

We commenced our investigation on the reaction with *N*-(2-formylphenyl)benzenesulfonamide (**1a**) and (3,3-difluorocycloprop-1-en-1-yl)benzene (**2a**) in the presence of 5 mol% of $[Cp^*RhCl_2]_2$ and 2.0 equiv. of CsOAc in DCE at 60 °C under N_2 atmosphere (Table 1). A preliminary attempt gave desired product **3a** in 20% yield, and its structure was unambiguously confirmed by NMR spectra and single-crystal X-ray diffraction analysis (for details, see Supporting information). Further studies to improve the efficiency

Table 1
Optimization of reaction conditions.[a]

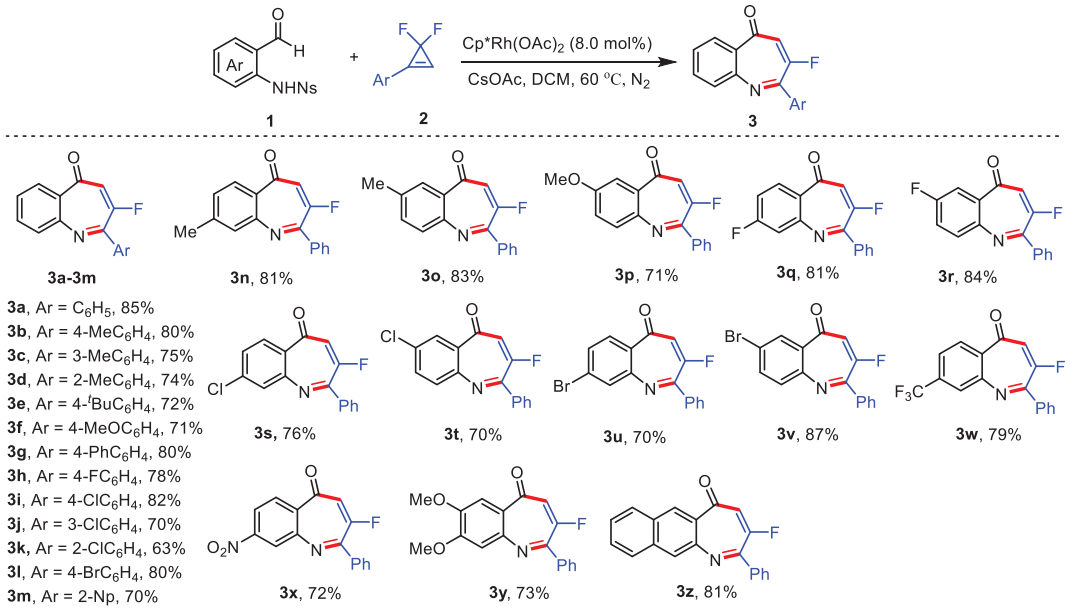


Entry	Catalyst (5 mol%)	Base	Solvent	Yield (%)[b]
1	$[Cp^*IrCl_2]_2$	CsOAc	DCE	0
2	$[(p\text{-cymene})RuCl_2]_2$	CsOAc	DCE	0
3	$Cp^*Co(CO)I_2$	CsOAc	DCE	0
4[c]	$Cp^*Rh(OAc)_2$	CsOAc	DCE	74
5[c]	$Cp^*Rh(OAc)_2$	CsOAc	DCM	77
6[c]	$Cp^*Rh(OAc)_2$	CsOAc	MeOH	0
7[c]	$Cp^*Rh(OAc)_2$	CsOAc	DMF	trace
8[c]	$Cp^*Rh(OAc)_2$	CsOAc	Dioxane	trace
9[c]	$Cp^*Rh(OAc)_2$	CsOAc	Toluene	trace
10[c]	$Cp^*Rh(OAc)_2$	CsOAc	Xylene	35
11[c]	$Cp^*Rh(OAc)_2$	CsOAc	CH_3CN	0
12[c]	$Cp^*Rh(OAc)_2$	NaOAc	DCM	69
13[c]	$Cp^*Rh(OAc)_2$	KOAc	DCM	67
14[c]	$Cp^*Rh(OAc)_2$	Cs_2CO_3	DCM	47
15[c]	$Cp^*Rh(OAc)_2$	CsF	DCM	73
16[c,d]	$Cp^*Rh(OAc)_2$	CsOAc	DCM	85
17[c,e]	$Cp^*Rh(OAc)_2$	CsOAc	DCM	73
18[d]	–	CsOAc	DCM	0
19[c]	$Cp^*Rh(OAc)_2$	–	DCM	27

[a] Reaction conditions: **1** (0.15 mmol), **2a** (0.18 mmol), catalyst (5 mol%) and base (0.30 mmol) in the solvent (0.1 mol/L) for 12 h, at 60 °C under N_2 atmosphere.
[b] Isolated yields were reported.
[c] 8 mol% catalyst loading was used.
[d] 1.0 equiv. CsOAc was used.
[e] 0.5 equiv. CsOAc was used.

of this transformation were conducted. Swapping R substituents on the nitrogen atom from 4-methylbenzenesulfonyl to benzenesulfonyl (**1b**), 4-nitrobenzenesulfonyl (Ns, **1c**) and *t*-butyloxy carbonyl (**1d**) revealed that 4-nitrobenzenesulfonyl **1c** proved to be the better choice, giving **3a** in 53% yield. The reaction did not work when free 2-aminobenzaldehyde (**1e**) was used. Subsequently, the reaction parameters, including the catalyst, solvent and base, were examined, as shown in Table 1. Other transition metal catalysts such as $[Cp^*IrCl_2]_2$, $[(p\text{-cymene})RuCl_2]_2$, $Cp^*Co(CO)I_2$ and $Cp^*Rh(OAc)_2$ were tested in the reaction of **1c** with **2a** (entries 1–4). The results indicated that the former three completely suppressed the reaction process, and no desired product was observed. Delightedly, the latter demonstrated a higher catalytic performance, enhancing the yield to 74% (entry 4). The yield was slightly increased when DCE was replaced with DCM, delivering product **3a** in 77% yield (entry 5). Next, the effect of the solvent was further evaluated by exploiting several other solvents such as MeOH, DMF, dioxane, toluene, xylene, and CH_3CN (entries 6–11). However, none of these attempts gave positive results. The use of other bases such as NaOAc, KOAc, Cs_2CO_3 and CsF could make the reaction work, but less efficient than CsOAc (entries 12–15 *vs.* entry 5). Decreasing the amount of CsOAc to 1.0 equiv. resulted in a higher yield of **3a** (85%, entry 16). Further reducing the amount of CsOAc is harmful for the reaction (entry 17). Without the Rh catalyst or the base, the reaction was ineffective (entries 18 and 19).

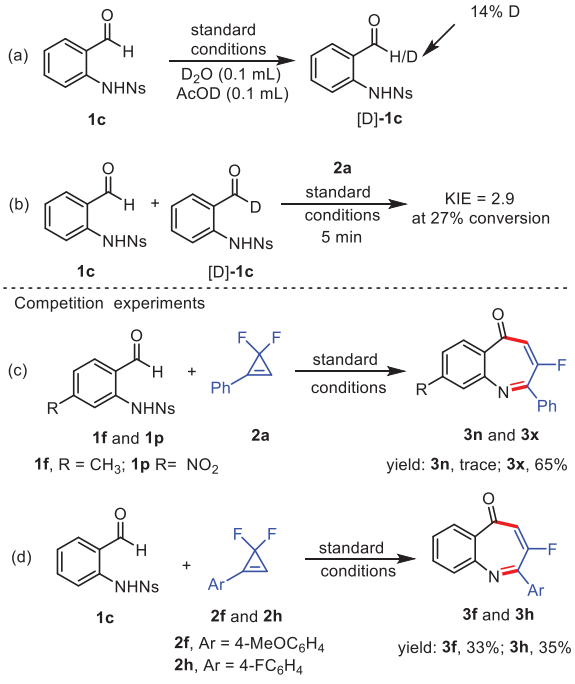
With the optimized reaction conditions in hand, we set out to examine the scope and limitations of this Rh(III)-catalyzed



Scheme 2. Substrate scope for the synthesis of products **3**. Reaction conditions: **1** (0.15 mmol), **2a** (0.18 mmol), $Cp^*Rh(OAc)_2$ (8.0 mol%), CsOAc (0.15 mmol), DCM (1.5 mL), 60 °C, N_2 atmosphere, 12 h, isolated yield. For **3p** and **3y**, Ts instead of Ns, 24 h.

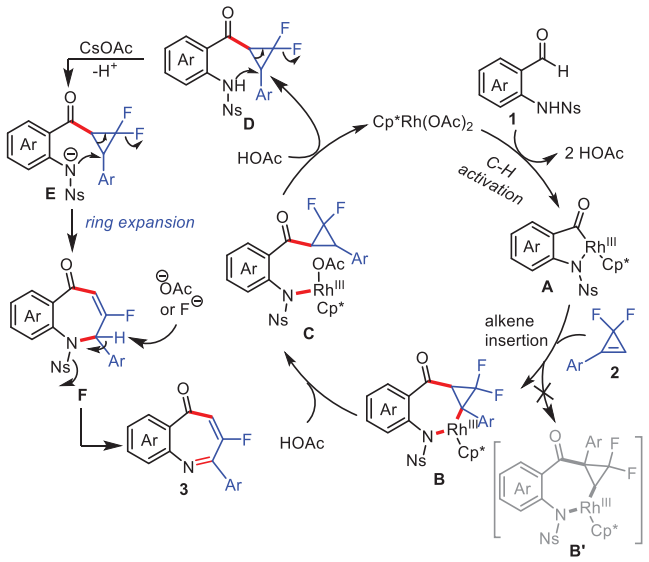
[4 + 3] annulation by exploiting a variety of *N*-sulfonyl 2-aminobenzaldehydes with *gem*-difluorocyclopropenes. As shown in Scheme 2, first, the effects of changing the electronic properties and positions of substituents in the arene ring of *gem*-difluorocyclopropenes were investigated by repeatedly reacting with substrate **1c**. Both electron-donating (methyl **2b-2d**, *tert*-butyl **2e**, methoxy **2f**, and phenyl **2g**) and electron-withdrawing (fluoro **2h**, chloro **2i-2k** and bromo **2l**) groups located at various positions (*ortho*, *meta*, or *para*) of the phenyl ring were all compatible with the standard conditions, furnishing corresponding products **3b-3l** in 63%–82% yields with complete regioselectivity. Of these functional groups, the more sterically demanding *o*-chloro and *o*-methyl substituents were examined to prove the compatibility of this transformation with high efficiency. Besides, 2-naphthyl group-substituted *gem*-difluorocyclopropene **2m** was also feasible, giving the desired benzo[*b*]azepin-5-one **3m** in 70% yield. Then, the scope of *N*-sulfonyl-2-aminobenzaldehydes was also investigated in this catalytic [4 + 3] annulation. In detail, various commonly encountered substituents, such as electronically rich (methyl, **1f–1g** and methoxy, **1h** and **1q**), and poor (fluoro, **1i–1j**; chloro, **1k–1l**; bromo, **1m–1n**; trifluoromethyl, **1o**; nitro, **1p**) at the 4- and 5-positions of the arene ring, could successfully participate in the [4 + 3] annulation, producing the corresponding benzo[*b*]azepin-5-ones **3n-3y** in 70%–87% yields. Even challenging cases where the trifluoromethyl and nitro functionalities are strong electronically deficient groups at the 4-position were functional for this catalytic protocol, providing products **3w** and **3x** in 79% and 72% yields, respectively. Alternatively, disubstituted 2-aminobenzaldehyde **1q** proved to be applicable (**3y**, 73%). Furthermore, naphthyl counterpart **1q** was also suitable for this reaction, enabling the annulation to access **3z** in 81% yield. Regrettably, the reaction of *N*-sulfonyl 2-aminobenzaldehydes bearing a methyl substituent at the 3-, or 6-position was unsuccessful, probably due to steric effects.

Further investigations were then performed to gain more insight into the mechanism of this transformation (Scheme 3). First, attempts to trace aldehydic C(sp^2)-H activation of **1c** by deuterium incorporation showed 14% deuteration at the aldehyde C-H bond (Scheme 3a), suggesting that amino-directed aldehyde C(sp^2)–H



Scheme 3. Mechanistic studies.

activation may be involved in the reaction. Subsequently, the deuterium labeling experiment was conducted to display a significant kinetic isotope effect (KIE, KIE = 2.9) in an intermolecular competitive coupling of an equimolar mixture of **1c** and [D]–**1c** with **2a** (Scheme 3b), demonstrating that the rupture of C(sp^2)-H bonds on the aldehyde fragment occurs at the rate-determining step. To understand the reactivity of *N*-sulfonyl 2-aminobenzaldehydes and *gem*-difluorocyclopropenes, two intermolecular competitive reactions were performed: 1) a competition reaction of **1f** and **1p** exclusively gave **3x** in 65% yield with a trace amount of **3n** (Scheme 3c), implying that the *N*-sulfonyl 2-aminobenzaldehyde with an



Scheme 4. Proposed mechanism for forming product **3**.

electron-donating substituent is less reactive; and 2) the other competition reaction of **2f** and **2h** generated products **3f** and **3h** in almost the same yields (Scheme 3d), suggesting that there are no apparent electronic effects in *gem*-difluorocyclopropenes.

Based on the literature survey [40–43] and our experimental observations, a plausible mechanism for this [4 + 3] annulation is outlined in Scheme 4. Initially, ligand exchange with *N*-sulfonyl 2-aminobenzaldehyde **1** *via* a similar concerted metallation-deprotonation (CMD) pathway affords rhodacycle **A** [40–43], owing to the steric factor and the stability of the intermediate, which prefers to undergo regioselective migratory insertion into *gem*-difluorocyclopropene **2** to deliver intermediate **B** without observation of its regioisomer **B'**. Then two successive ligand exchanges of **B** with HOAc release intermediate **D** and regenerate catalytically active $Cp^*Rh(OAc)_2$ species for the next catalytic cycle. Subsequently, intermediate **D** undergoes defluorinated ring-opening of cyclopropane intermediate **E** to provide seven-membered intermediate **F**, followed by 1,2-elimination [42] to give product **3** in the presence of the base. Although the detailed reaction process is not clear, the synergistic effect of rhodium and CsOAc is believed to accelerate the formation of product **3** *via* defluorinated ring-opening and 1,2-elimination.

In conclusion, by taking advantage of the unique reactivity of *gem*-difluorocyclopropenes and *N*-sulfonyl-2-aminobenzaldehydes, Rh(III)-catalyzed aldehydic C−H activation/[4 + 3] annulation was developed, and used to produce a wide range of unreported β-monofluorinated benzo[*b*]azepin-5-ones with good yields and complete regioselectivity in one-pot manner. This redox-neutral protocol holds a broad substrate scope, good functional group tolerance, and high regioselectivity, which affords a conceptually new synthesis strategy capable of the integration of C−H activation with fluorine chemistry. Further investigation and application of these fluorinated 7-membered heterocycles is underway in our laboratory.

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

We are grateful for financial support from the school-level research projects of Yancheng Institute of Technology (No. xjr2020044) and the National Natural Science Foundation of China (Nos. 22101151 and 21971090)

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.108014.

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