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Iridium-Catalyzed Asymmetric Reductive Amination of C2-Acylpyridines

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Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

Chiral 1-pyridin-2-yl-ethylamines are significant building blocks for synthetic chemistry and pharmaceutical sciences, while their stereoselective and practical synthesis remains challenging and usually troublesome. Herein, we present a novel iridium-catalyzed direct asymmetric reductive amination of 2-acylpyridines with anilines, affording a series of chiral 1-pyridin-2-yl-ethylamines with up to 97% yield and 95% ee. A 5 mmol scale reaction is also performed to demonstrate the practicality of this method.

Given the unique electronic properties and chemical reactivity conferred by the nitrogen atom in the pyridine ring, chiral molecules containing a pyridine ring hold significant positions in pharmaceutical synthesis, materials, and catalysis.^{1–7} Among these, chiral 1-(pyridin-2-yl)ethan-1-amine derivatives are critical structural units as they frequently serve as pharmacophores of drug molecules or as ligands for metal catalysis (Figure 1).⁸ For instance, chiral 1-[5-(2,2,2-trifluoroethoxy)pyridin-2-yl]ethan-1-amine is a key fragment in the T-type calcium channel modulator Suvecaltamide⁹ and the [¹⁸F]-TTA-A4¹⁰ drug molecules. Similarly, 1-(5-fluoropyridin-2-yl)ethan-1-amine serves as the core structure of a novel orally bioavailable TRK inhibitor AZ-23.¹¹ Additionally, 1-(pyridin-2-yl)ethan-1-amine is also an essential component of potential adenosine receptor antagonists LDH-E-8-2.¹² Moreover, these structural motifs are also widely found in other drug structures.^{13–15} On the other hand, due to the coordination ability of the nitrogen atom in the pyridine ring with metals, these compounds are commonly used to synthesize chiral bidentate and tridentate ligands, which are applied in the field of asymmetric transition-metal catalysis.^{16–18} Therefore, the development of efficient synthetic methods for chiral 1-

(pyridin-2-yl)ethan-1-amines is of great scientific and commercial significance.

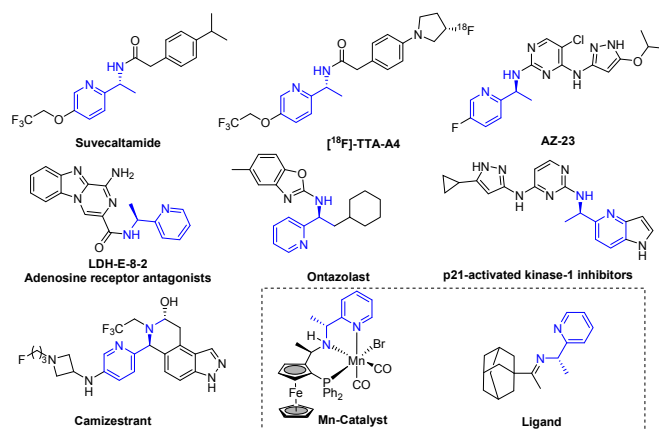


Figure 1 Selected bioactive molecules and ligand bearing a 2-pyridine-substituted chiral amine unit

So far, there are only a few reports on the catalytic asymmetric synthesis of chiral 1-pyridin-2-yl-ethylamines, while the majority rely on chiral auxiliary strategy.^{19,20} The direct catalytic asymmetric addition of MeLi to pyridyl-2-imines was poor.²¹ Alternatively, organocatalytic asymmetric reduction of ketimines with silanes as reductants was also reported, while the asymmetric control did not reach a useful level (Scheme 1a).^{22,23} On the other hand, transition-metal-catalyzed asymmetric reductive amination (ARA) of ketones has been proven as a reliable and direct method for the synthesis of chiral amines.^{24–35} Most reported ketone examples containing a pyridine ring are limited to 3 or 4-acylpyridines;^{36,37} instead, there are only very limited reports dealing with 2-acylpyridines, likely due to the strong coordinating ability of both the starting materials and the resulting products toward metal catalysts. For example, Yamada reported a Ru-catalyzed ARA of C2-acetylpyridine with ammonium salts; however, a substituent on the C6 position is required to ensure efficiency (Scheme 1b).³⁸ In addition, Zhou reported an intriguing iridium-catalyzed asymmetric stepwise amination of C2-benzoylpyridines of

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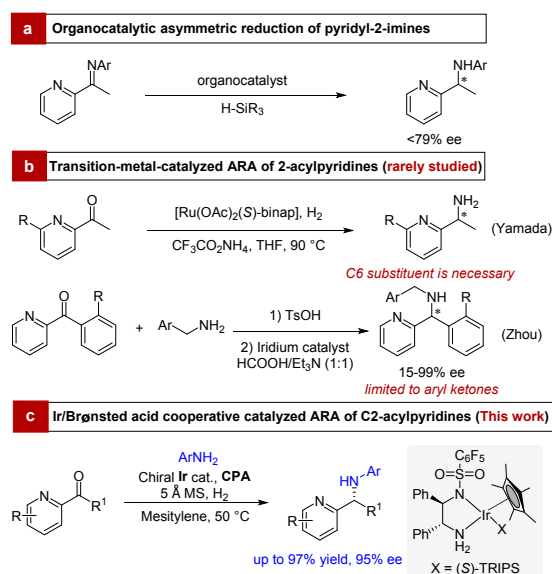
*Supplementary Information available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x



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benzylamine using transfer hydrogenation, while the scope is limited to aryl ketones.³⁹ Based on the current method limitations and the importance of the products, an efficient method towards the synthesis of enantioenriched 1-pyridin-2-yl-ethylamines is still highly desirable and rewarding. To continue our interest in ARA and drug synthesis,^{40–45} we report here an Ir-catalyzed direct and scalable ARA of C2-acetylpyridines with arylamines to synthesize a variety of chiral 1-pyridin-2-yl-ethylamine derivatives with good to excellent ee and broad scope (Scheme 1C).



Scheme 1 Background on the synthesis of chiral 2-pyridinemethanamines and our strategy

We started to explore the direct ARA reaction of C2-acetylpyridine using *p*-anisidine as the amine source. Initially, we tried several traditional catalytic systems, including various Ru or Ni catalysts,^{46,47} however, low reactivity or poor enantioselectivity were observed. Then we switched to half-sandwich Cp*Ir(III)-diamine catalysts bearing a chiral phosphate counteranion, an outstanding catalytic system for ARA reactions disclosed by Xiao's group.^{48–50} To our delight, all tested Cp*Ir(III) catalysts displayed good activity as nearly complete conversion was observed, however, the imine intermediate was also detected when using **Ir-1**, **Ir-4** or **Ir-5** (entries 1–5). The measurement of the ee of each product disclosed that the ArSO₂ group on the diamine ligand had a significant influence on the asymmetric control, with **Ir-5** providing the best ee of **3a** (80% ee, entry 5). To increase the yield of **3a** in the presence of **Ir-5**, we increased the pressure and temperature. **3a** with 85% ee and 95% GC yield was achieved at 50 °C and 6 MPa of H₂ pressure (entries 6–7). It's known that the configuration of the counteranion may affect the stereocontrol, and we thus test its influence. However, the use of (*R*)-**5a** led to a slight decrease in ee (entry 8). The use of two different chiral phosphate counteranions did not provide satisfactory results (entries 9–10). Next, we screened a series of solvents, including MeOH, THF, EA, MeCN, etc., and found that aromatic solvents were well-suited for the reaction, with

mesitylene yielding the best results, achieving 99% yield and 89% ee (entries 11–20). Finally, by adjusting the type of molecular sieves, we were able to increase the ee of the target product **3a** to 90% (entries 21–22). Contrary to expectation, a decrease in temperature did not enhance the reaction enantiocontrol (entry 23). Therefore, the optimal conditions were established as follows: **Ir-5** as the catalyst, (*S*)-**5a** and 5 Å MS as additives, mesitylene as solvent, 6 MPa H₂.

Table 1 Reaction conditions optimization of reductive amination of 2-acetylpyridine.^[a]

entry	cat.	CPA	P (MPa)	temp.	Conv.	3a/3a ^[b]	Ee ^[c]
1	Ir-1	(<i>R</i>)- 5a	4	35 °C	96%	92/8	-27%
2	Ir-2	(<i>R</i>)- 5a	4	35 °C	99%	99/1	-5%
3	Ir-3	(<i>S</i>)- 5a	4	35 °C	97%	99/1	15%
4	Ir-4	(<i>S</i>)- 5a	4	35 °C	99%	75/25	47%
5	Ir-5	(<i>S</i>)- 5a	4	35 °C	93%	19/81	80%
6	Ir-5	(<i>S</i>)- 5a	6	35 °C	99%	80/20	77%
7	Ir-5	(<i>S</i>)- 5a	6	50 °C	99%	95/5	85%
8	Ir-5	(<i>R</i>)- 5a	6	50 °C	98%	95/5	83%
9	Ir-5	(<i>S</i>)- 6a	6	50 °C	99%	78/22	57%
10	Ir-5	(<i>S</i>)- 7a	6	50 °C	95%	96/4	72%
11	Ir-5	(<i>S</i>)- 5a	6 (MeOH)	50 °C	91%	57/43	rac.
12	Ir-5	(<i>S</i>)- 5a	6 (TFE)	50 °C	71%	53/47	12%
13	Ir-5	(<i>S</i>)- 5a	6 (THF)	50 °C	72%	45/55	70%
14	Ir-5	(<i>S</i>)- 5a	6 (MeCN)	50 °C	54%	63/37	23%
15	Ir-5	(<i>S</i>)- 5a	6 (EA)	50 °C	86%	72/28	55%
16	Ir-5	(<i>S</i>)- 5a	6 (OX)	50 °C	99%	99/1	86%
17	Ir-5	(<i>S</i>)- 5a	6 (MX)	50 °C	99%	95/5	88%
18	Ir-5	(<i>S</i>)- 5a	6 (PX)	50 °C	99%	97/3	87%
19	Ir-5	(<i>S</i>)- 5a	6 (Anisole)	50 °C	99%	99/1	73%
20	Ir-5	(<i>S</i>)- 5a	6 (Mesityl)	50 °C	99%	99/1	89%
21	Ir-5	(<i>S</i>)- 5a	6 (Mesityl)	50 °C	99% (3 Å MS)	99/1	86%
22	Ir-5	(<i>S</i>)- 5a	6 (Mesityl)	50 °C	99% (5 Å MS)	99 (96)/1	90%
23	Ir-5	(<i>S</i>)- 5a	6 (Mesityl)	40 °C	99% (5 Å MS)	86/14	86%

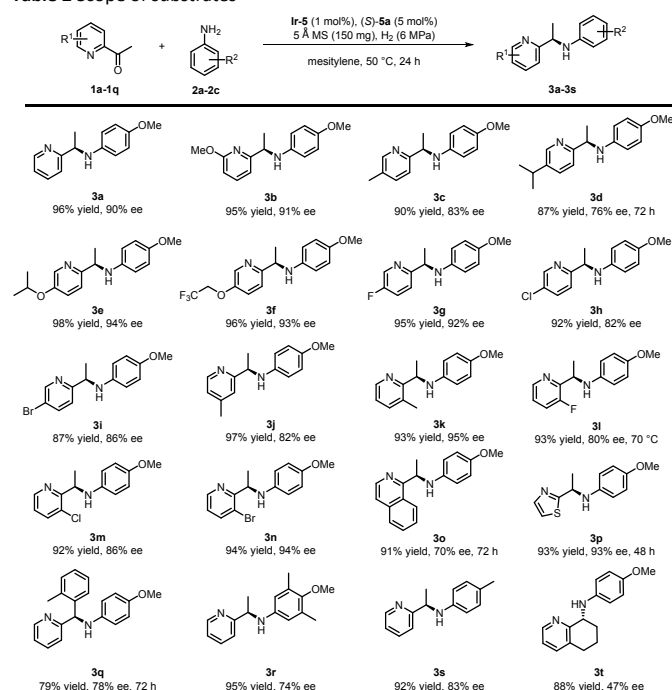
[a] Reaction conditions: **1a** (0.1 mmol), *p*-anisidine (0.12 mmol), **cat.** (1 mol%), solvent (0.5 mL), H₂ (4 MPa), 35 °C, 4 Å MS (50 mg), 24 h. [b] Determined by GC-MS. [c] The ee values were determined by chiral HPLC analysis. [d] Isolated yield. OX = *o*-xylenes; MX = *m*-xylenes; PX = *p*-xylenes; Mesityl = mesitylene

Having established the optimal conditions, we then explored the substrate applicability (**Table 1**). Initially, we examined the effect of a C6-MeO substituent in 2-acetylpyridine and found that it was well-tolerated, yielding product **3b** with 95% yield and 91% ee. Then we explored a series of easily accessed C5-substituted 2-acetylpyridines. With electron-donating groups, complete conversions were observed, supplying the corresponding products with yields ranging from 87% to 98%



and ee values between 76% and 94% (**3c-3f**). Substrates bearing a halide group also performed well, yielding **3g-3i** with yields of 87% to 95% and an ee between 82 to 92%. The compatibility of halides offers further chances for derivatizations. For C4-Me-substituted 2-acetylpyridine **1j**, the product **3j** was obtained with 82% ee but nearly quantitative yield. Interestingly, substituents (*e.g.*, Me or halide) on the C3 position have little effect on the outcome (**3k-3n**), producing the desired products with 80-95% ee. We also tested other N-hetero aromatics, such as 1-isoquinolin-1-yl-ethanone (**1o**) or 2-acetylthiazole (**1p**). While the enantiocontrol of **1o** was decreased (70% ee for **3o**), the products **3p** stemming from **1p** were achieved with a remarkable 93% yield and 93% ee after 48 hours. Notably, the sterically bulkier substrate pyridin-2-yl(*o*-tolyl)methanone **1q** yielded **3q** with 78% ee and 79% yield after 72 hours, highlighting the potential of this method on more challenging substrates. In addition, we also tested two other anilines (**3r**, **3s**), however, the asymmetric control seemed to be sensitive to the substituent on the phenyl ring, and inferior control was observed. Notably, cyclic ketone **1t** was also applicable while the corresponding product **3t** has only 47% ee.

Table 2 Scope of substrates^a

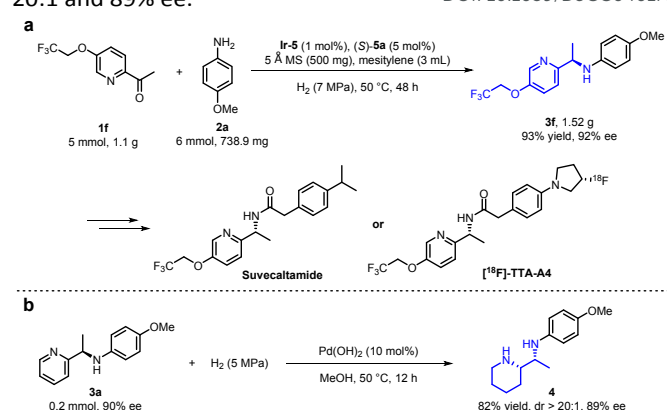


[a] Reaction conditions: ketone **1** (0.3 mmol), Ar-NH₂ (0.36 mmol), **cat.** (1 mol%), (*S*)-**5a** (5 mol%), mesitylene (1 mL), 5 Å MS (150 mg), H₂ (6 MPa), 50 or 70 °C, 24~72 h, isolated yield. Absolute configuration assigned by comparison with the literatures (see the Supporting Information).

To showcase the practicality, substrate **1f** was chosen for the scale-up reaction. At a 5 mmol scale, product **3f** was obtained with a 93% yield and 92% ee under 7 MPa hydrogen pressure for 48 hours, matching small-scale results. Product **3f** can be further converted into various drug molecules. In addition, the pyridine ring in compound **3a** can be hydrogenated in a highly diastereoselective manner using Pd(OH)₂/C as a catalyst,

yielding the piperidine-derived vicinal diamine **4** with a dr of 20:1 and 89% ee.⁵¹

DOI: 10.1039/D5CC04017E



Scheme 2 Scale-up experimentation and synthetic applications

In conclusion, the ARA reaction of C2-acylpyridines and their analogues with *p*-anisidine was achieved via synergistic catalysis using Cp*Ir(III)-diamine catalysts and chiral phosphoric acid, affording a series of synthetically useful chiral 1-(2-pyridyl)ethylamine derivatives with generally high yields and good to excellent ee. In addition, this method is scalable, offering a rapid and practical strategy for the synthesis of drug molecules containing the chiral 1-(2-pyridyl)ethylamine scaffold.

This work was supported by the National Natural Science Foundation of China (No. 22522109; 22271307; 22302048), the Shenzhen Science and Technology Innovation Program (No. JCYJ20240813161112017; JCYJ20220818100804010; GXWD20220811173736002; and KCXFZ20230731094904009), and the Shenzhen Key Laboratory of Advanced Functional Carbon Materials Research and Comprehensive Application. Q.Y. is indebted to Shenzhen University of Advanced Technology and Shenzhen Institutes of Advanced Technology, Chinese Academy of Sciences, for providing a starting grant.

Author contributions

Gang Wang: Writing—original draft, Methodology, Data curation. Zhiwen Nie: Methodology, Data curation. Hengzhi You: review&editing, Project administration. Qin Yin: Writing—review&editing, Project administration, Conceptualization.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the Supplementary Information.

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The data supporting this article have been included as part of the
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DOI: 10.1039/D5CC04017E

