Transition-Metal-Catalyzed Asymmetric Reduction of 2-Pyridine Ketones

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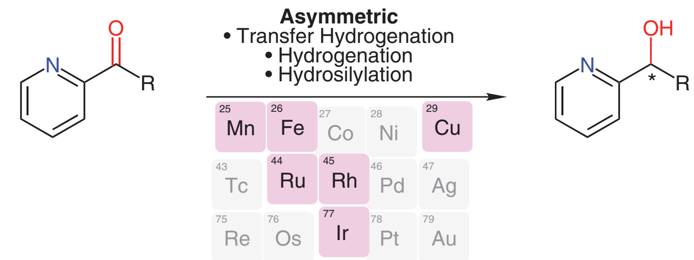

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Abstract This graphical review provides a concise overview of transition-metal-catalyzed asymmetric reduction of 2-pyridine ketones to produce enantiopure chiral 2-pyridine aryl/alkyl alcohols, which are present in many chiral ligands and pharmaceuticals. Key methods include metal-catalyzed hydrogenation, transfer hydrogenation, and hydrosilylation, with a focus on sustainable catalysts like iron and manganese. This review serves as a foundation for future advancements in sustainable and enantioselective keto group reductions.

Key words asymmetric reduction, ketones, transfer hydrogenation, hydrogenation, hydrosilylation, iron catalysis

The development of catalytic methods for the asymmetric reduction of keto groups, particularly in 2-pyridine ketones, has garnered considerable interest due to the transformative potential of these reactions in the synthesis of enantiomerically pure compounds. Enantiopure chiral 2-pyridine aryl/alkyl alcohols are not only essential intermediates in creating chiral ligands, such as Bolm's ligand, but are also foundational in the synthesis of complex, stereochemically defined molecules in fields like pharmaceuticals and materials science. As a result, there has been substantial effort to design catalysts that facilitate these reductions with high enantioselectivity, efficiency, and versatility.

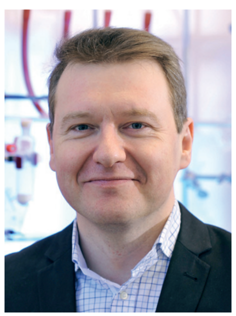
A wide array of catalytic approaches has emerged for the asymmetric reduction of 2-pyridine ketones, utilizing transition metals such as iron, manganese, ruthenium, copper, rhodium, and iridium. These systems often differ significantly in their mechanistic pathways, with some involving direct hydrogenation, others employing transfer hydrogenation, and others relying on hydrosilylation. Each method offers unique advantages, yet also presents challenges related to reaction scope, operational simplicity, cost, scalability, and environmental impact, with green chemistry principles driving much of the recent innovation in this field.

Despite these advancements, there remain open questions and unsolved challenges, particularly in the quest for more sustainable, non-precious metal catalysts and methods that maximize atom economy. Furthermore, the sheer pace of development in this area can sometimes obscure which transformations have reached maturity and which require further optimization or exploration. This graphical review seeks to clarify these developments, providing a structured overview of current catalytic systems for asymmetric reduction of 2-pyridine ketones. By highlighting well-established techniques alongside emerging approaches, it aims to illuminate future directions for research, particularly in the context of eco-friendly synthetic methodologies and the expanding role of iron-based catalysis in asymmetric synthesis.

Biosketches

Vidhul Vasudevan was born in Kerala, India, and obtained his BS-MS dual degree in chemistry from the Indian Institute of Science Education and Research, Kolkata, India. He was a MITACS Globalink Intern at Université Laval (Québec, Canada) under the supervision of Prof. Thierry Ollevier during the summer of 2023. His research was focused on the development of new bipyridine-based chiral ligands. Currently, he is a Ph.D. student at McGill University (Montréal), Canada.

Harishankar M. S was born in Kerala, India, and obtained his BS-MS dual degree in chemistry from the Indian Institute of Science Education and Research, Bhopal, India. He was a MITACS Globalink Intern at Université Laval (Québec, Canada) under the supervision of Prof. Thierry Ollevier during the summer of 2023, where he worked on the development of new bipyridine-based chiral ligands.



Thierry Ollevier was born in Brussels and obtained his B.Sc. (1991) and Ph.D. (1997) at the Université of Namur (Belgium) under A. Krief, and was a research associate at the Université catholique de Louvain (Belgium) under I. E. Markó (1997), a NATO postdoctoral fellow at Stanford University under B. M. Trost (1998–2000), then a postdoctoral fellow at the Université de Montréal under A. B. Charette (2000–2001). After an assistant professor appointment (2001) at Université Laval (Québec, Canada), he became associate professor (2006) and is currently a full professor. Current research in his group aims at designing novel catalysts, developing catalytic reactions, and applying these methods to chemical synthesis. He is active in the areas of iron catalysis, ligand design, asymmetric catalysis, fluorine chemistry, diazo and diazirine chemistry, flow chemistry, and bismuth chemistry. He has published more than 85 papers and 35 encyclopedia articles and book chapters. He served as an Associate Editor of *RSC Advances* from 2015 to 2022 and was admitted as a Fellow of the Royal Society of Chemistry (2016). After 5 years served as an Advisory Board member of *SynOpen*, he was appointed as Editor-in-Chief of *SynOpen* in 2023.

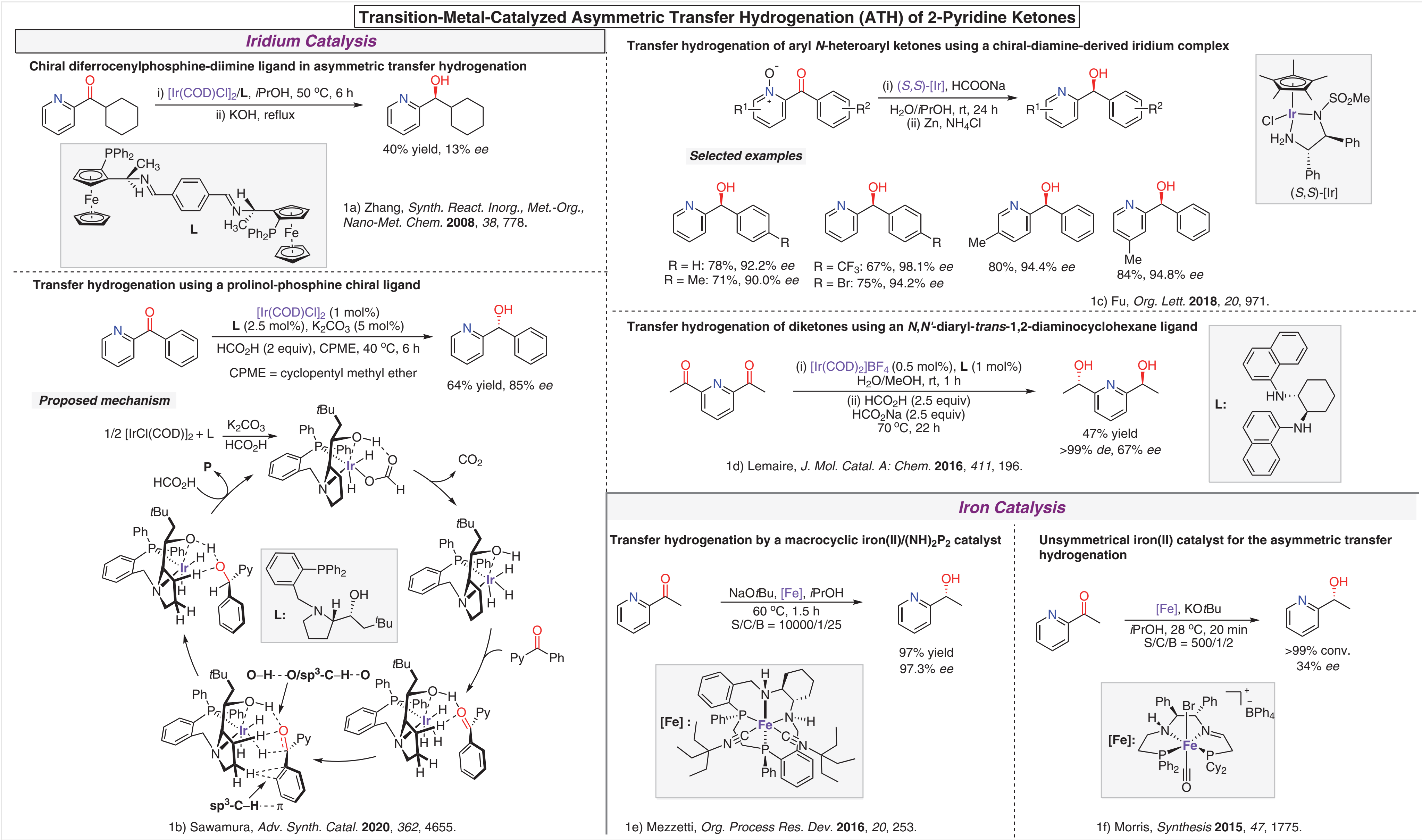


Figure 1 Catalytic asymmetric transfer hydrogenation using iridium and iron[1]

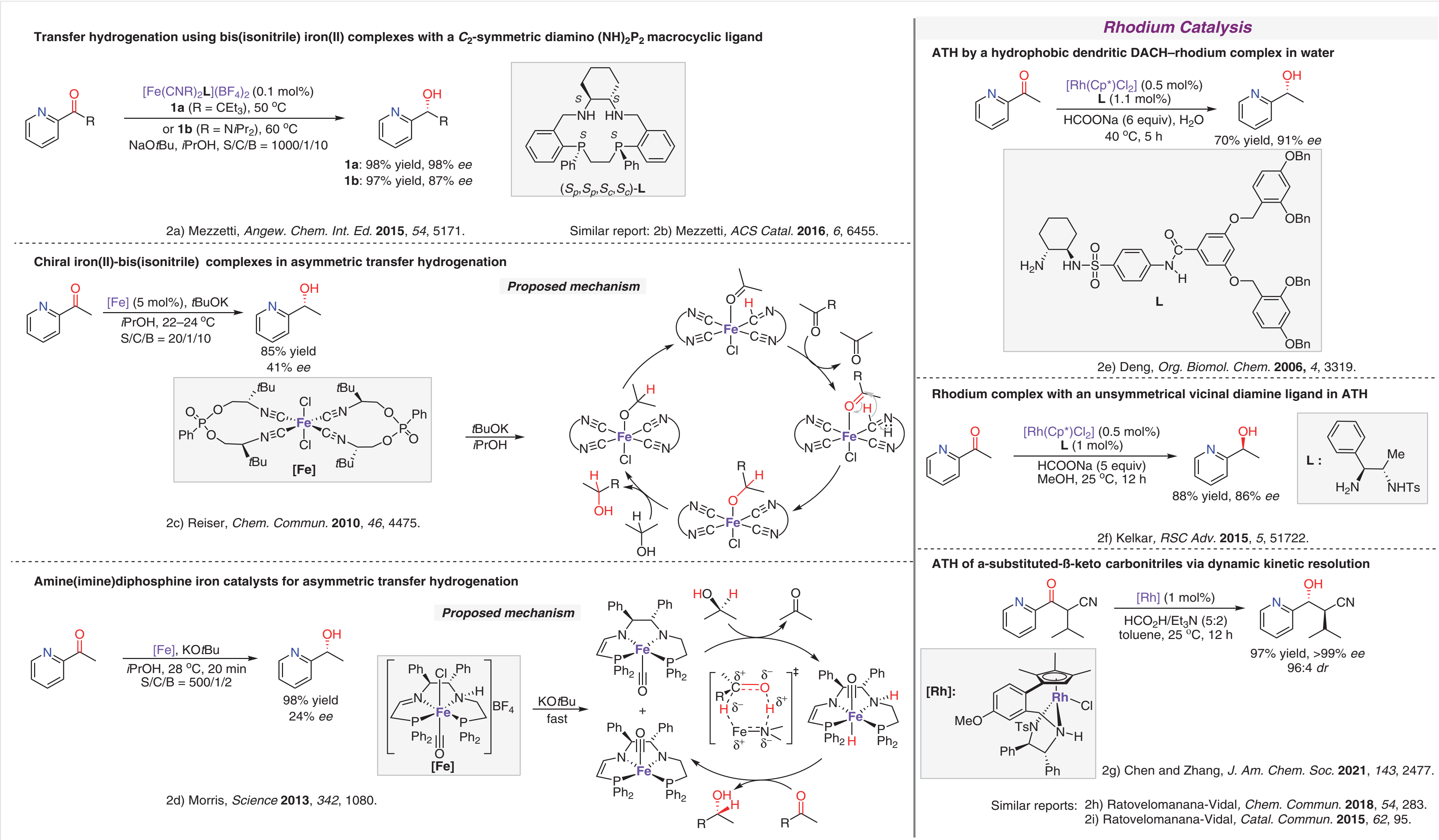


Figure 2 Catalytic asymmetric transfer hydrogenation using iron and rhodium[2]

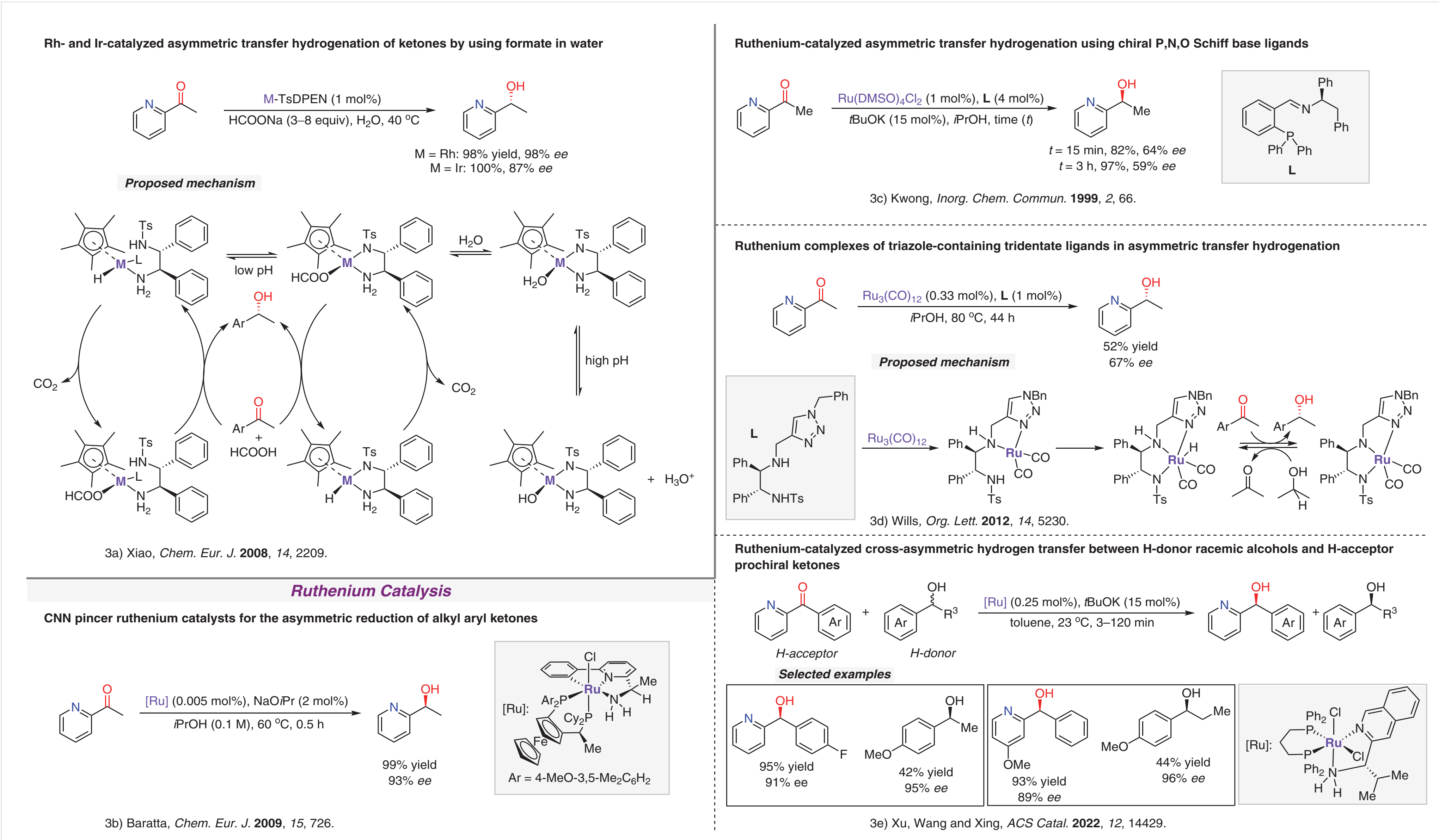


Figure 3 Catalytic asymmetric transfer hydrogenation using rhodium and ruthenium[3]

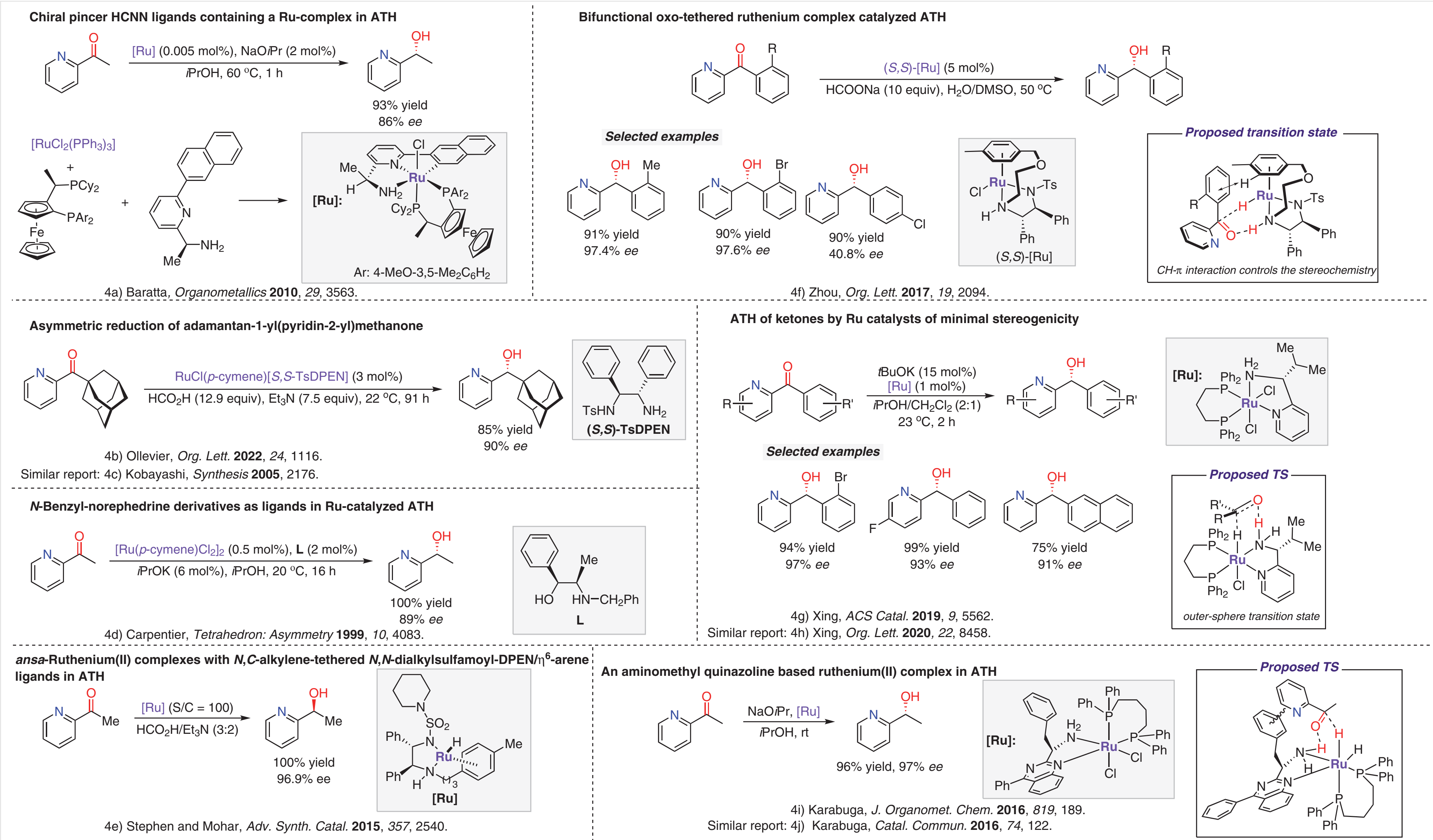


Figure 4 Ruthenium-catalyzed asymmetric transfer hydrogenation[4]

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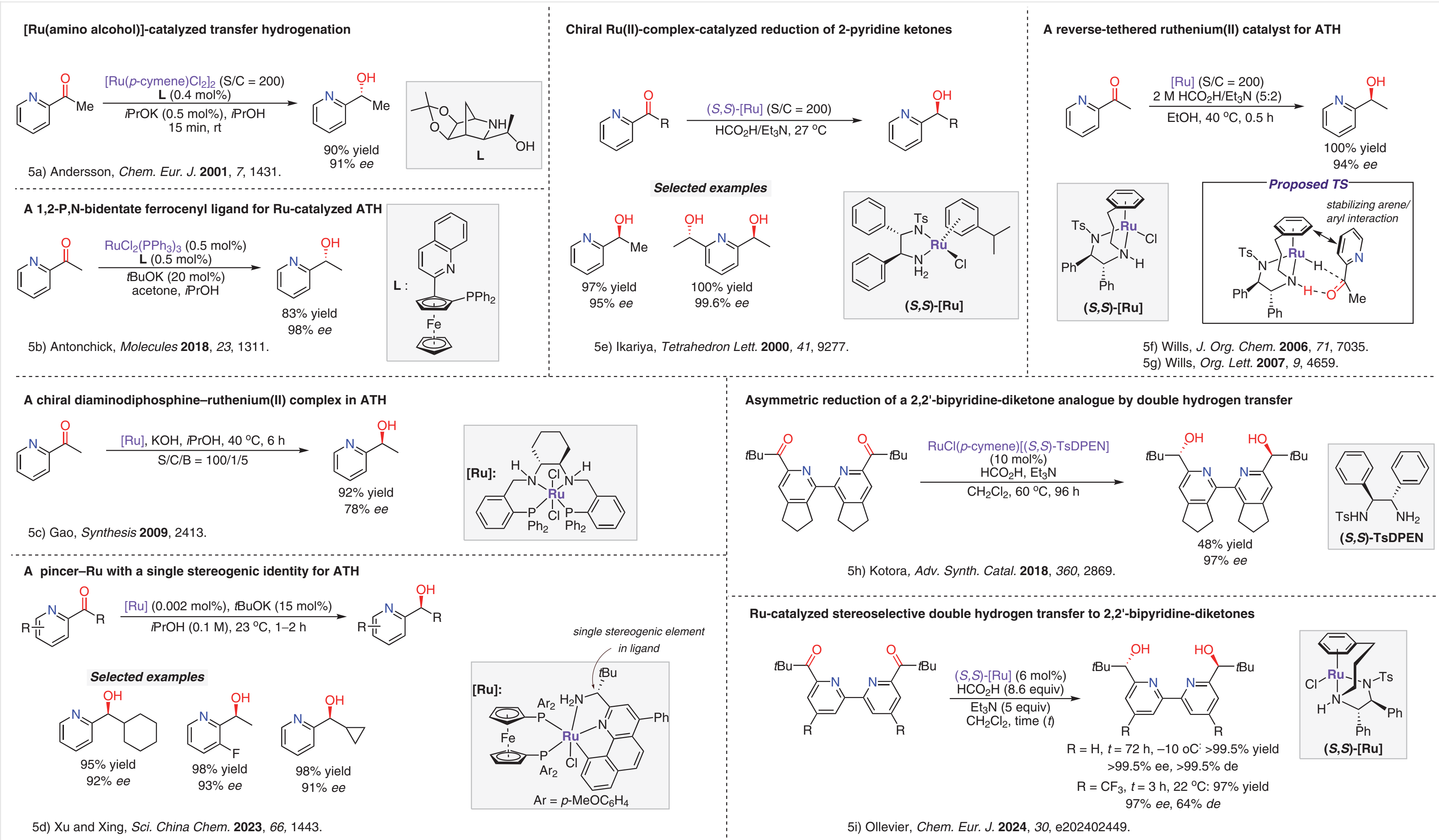


Figure 5 Ruthenium-catalyzed asymmetric transfer hydrogenation (cont.)[5]

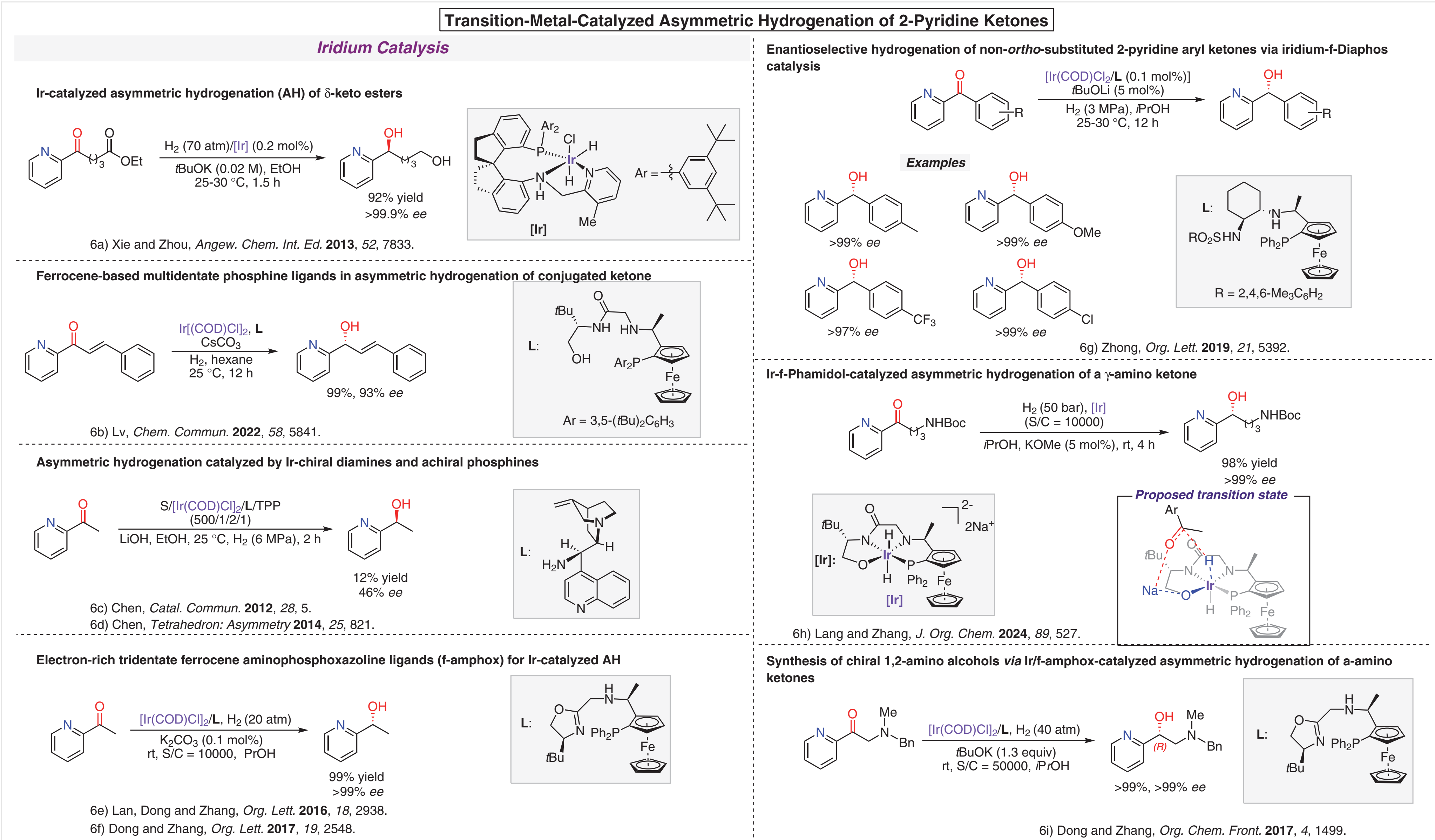
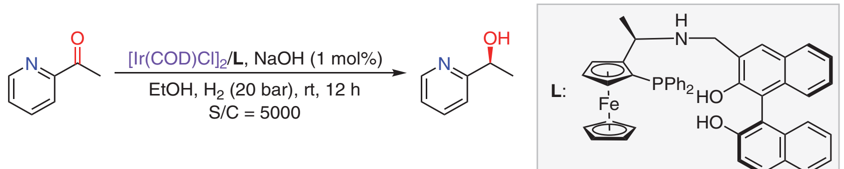


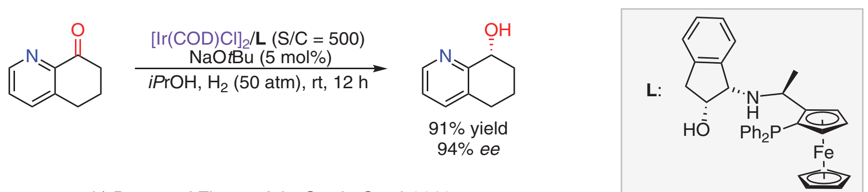
Figure 6 Iridium-catalyzed asymmetric hydrogenation[6]

Iridium-catalyzed asymmetric hydrogenation with f-Amphbinol ligand



7a) Zhou and Ji, *Org. Lett.* **2023**, *25*, 8845.

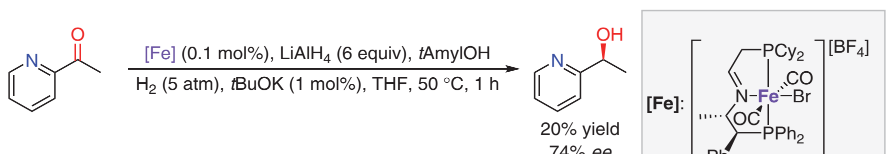
Iridium/f-Amphol-catalyzed asymmetric hydrogenation of benzo-fused cyclic ketones



7b) Dong and Zhang, *Adv. Synth. Catal.* **2018**, *360*, 4319.

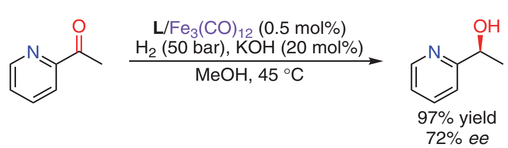
Iron Catalysis

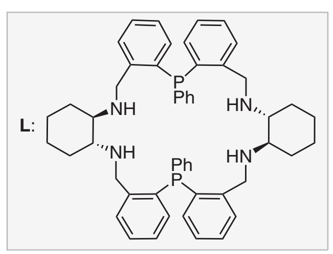
Iron(II) complexes containing unsymmetrical P–N–P' pincer ligands for asymmetric hydrogenation



7c) Morris, *J. Am. Chem. Soc.* **2014**, *136*, 1367.

Fe-catalyzed AH using a 22-membered macrocyclic ligand

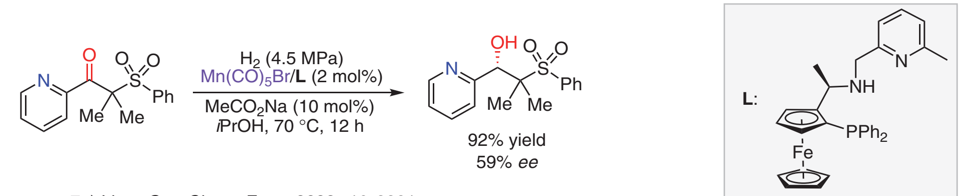




7d) Xiao and Gao, *J. Am. Chem. Soc.* **2014**, *136*, 4031.

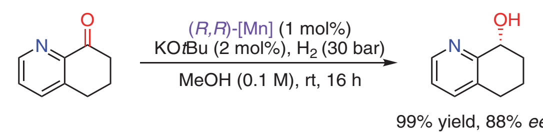
Manganese Catalysis

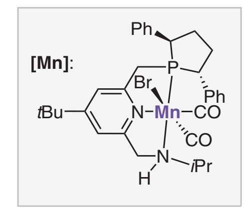
Enantioselective synthesis of chiral ß-hydroxy sulfones *via* manganese-catalyzed asymmetric hydrogenation



7e) Ling, *Org. Chem. Front.* **2023**, *10*, 3321.

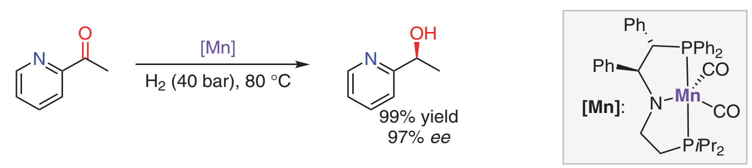
A lutidine-based chiral pincer manganese catalyst for enantioselective hydrogenation





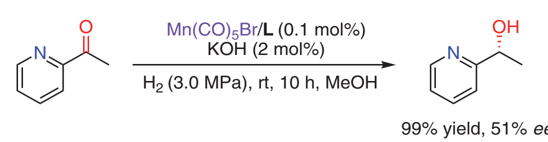
7f) Han and Ding, *Angew. Chem. Int. Ed.* **2019**, *58*, 4973.

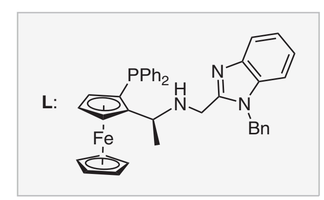
Base-free AH of ketones by a Mn-amido complex of a homochiral, unsymmetrical P–N–P' ligand



7g) Morris, *Catal. Sci. Technol.* **2021**, *11*, 3153.

An imidazole-based chiral PNN tridentate ligand in Mn-catalyzed AH

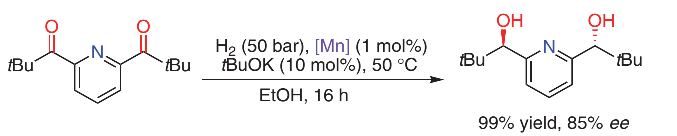


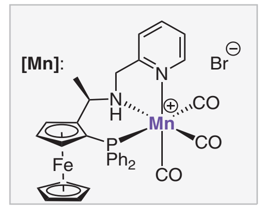


7h) Zhong, *Synlett* **2020**, *31*, 285.

Figure 7 Catalytic asymmetric hydrogenation using iridium, iron and manganese[7]

A Mn complex of a chiral P,N,N ligand in the AH of a 2,5-diketo pyridine

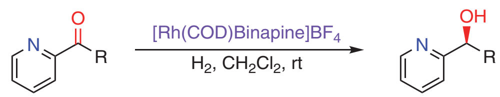




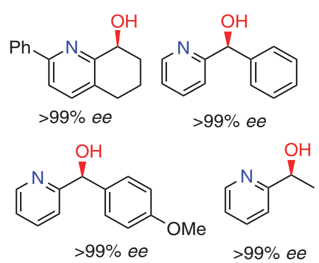
8a) Clarke, *Angew. Chem. Int. Ed.* **2017**, *56*, 5825.

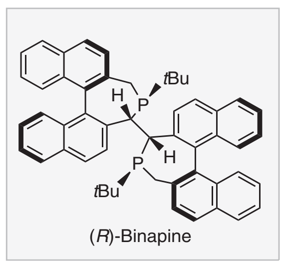
Rhodium Catalysis

Rhodium-catalyzed asymmetric hydrogenation of 2-pyridine ketones



Examples

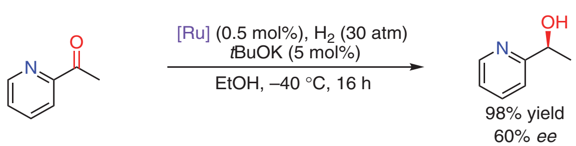


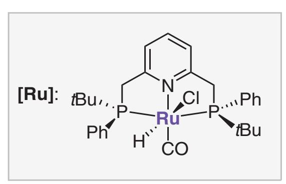


8b) Zhang, *Org. Lett.* **2015**, *17*, 4144.

Ruthenium Catalysis

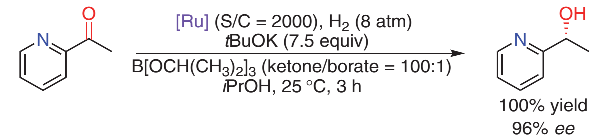
A *P*-stereogenic PNP^{tBu,Ph} Ru-complex in the AH of 2-pyridine ketones

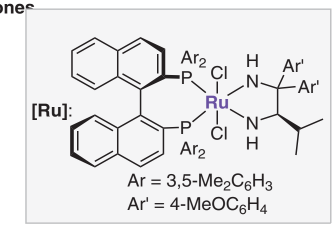




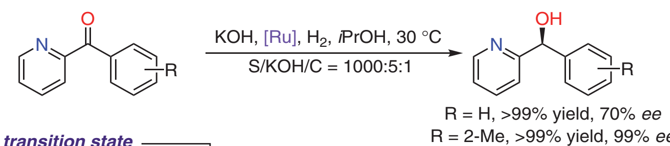
8c) Diaz and Castillón, *Eur. J. Org. Chem.* **2015**, 3666.

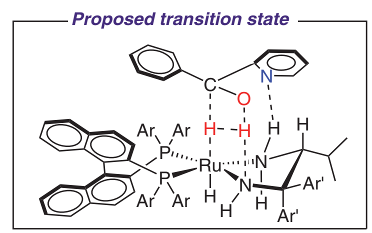
A ruthenium–diamine–diphosphine complex in the AH of pyridinyl aryl ketones

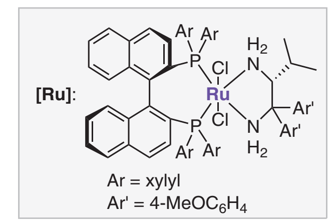




8d) Noyori, *Org. Lett.* **2000**, *2*, 1749.

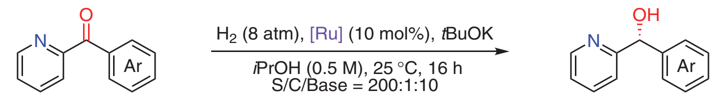






8e) Niedercorn and Castanet, *Tetrahedron* **2008**, *64*, 8700.

Enantioselective hydrogenation of aryl-pyridyl ketones with a Ru-XylSunPhos-Daipen bifunctional catalytic system



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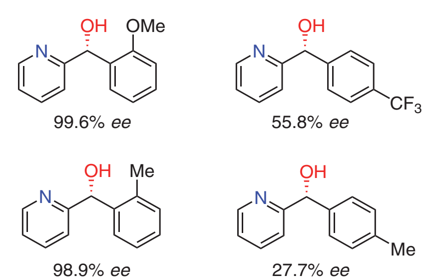
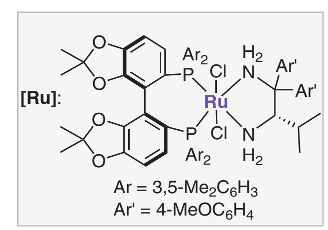
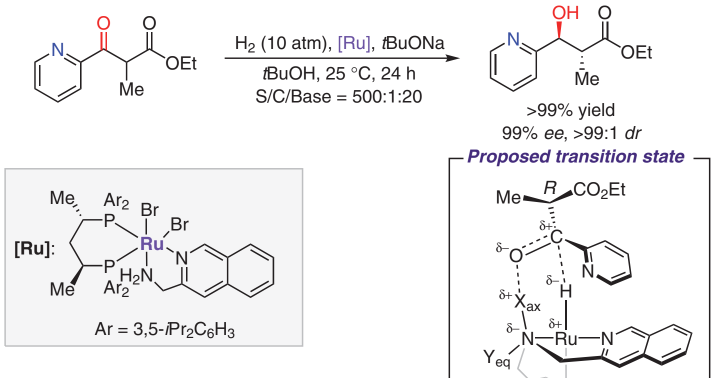




8f) Ratovelomanana-Vidal and Zhang, *J. Org. Chem.* **2012**, *77*, 612.

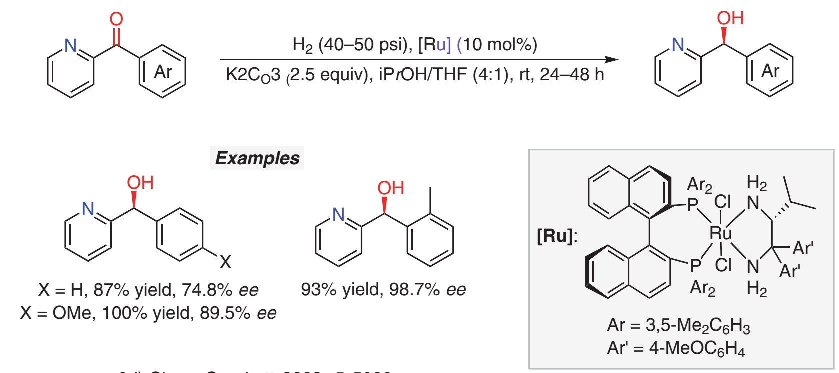
Figure 8 Catalytic asymmetric hydrogenation using manganese, rhodium and ruthenium[8]

Asymmetric hydrogenation of a-alkyl-substituted ß-keto esters through dynamic kinetic resolution



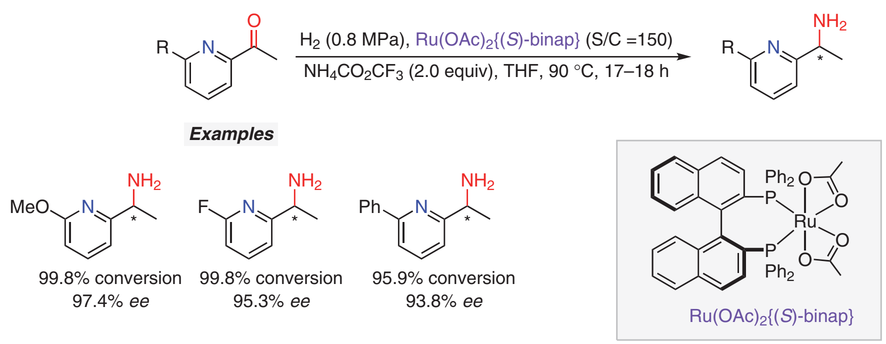
9a) Ohkuma, *Org. Lett.* **2024**, *26*, 2872.

Enantioselective hydrogenation of aromatic-heteroaromatic ketones



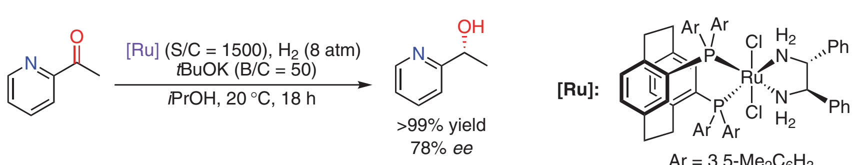
9d) Chen, *Org. Lett.* **2003**, *5*, 5039.

Enantioselective direct asymmetric reductive amination of 2-acetyl-6-substituted pyridines



9b) Yamada, *Org. Lett.* **2021**, *23*, 3364.

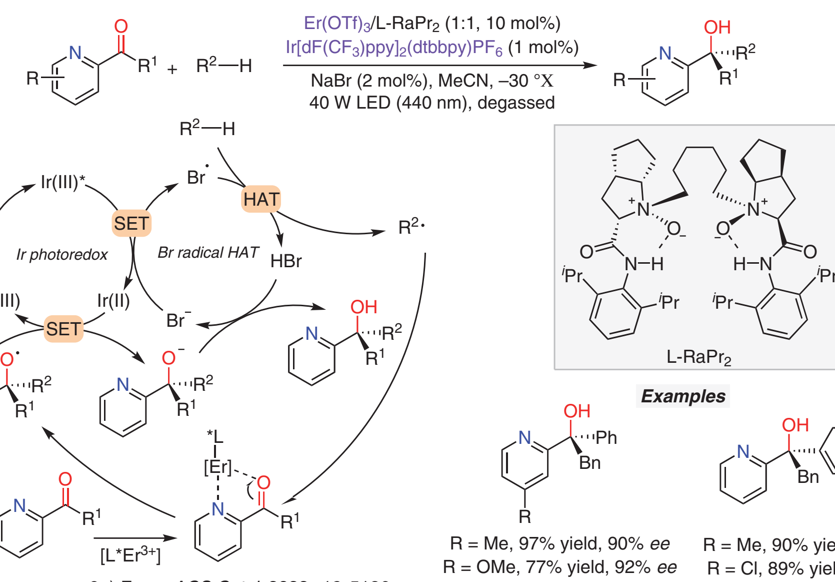
PhanePhos-Ru-diamine complexes catalyzed asymmetric hydrogenation



9c) Gerosa, *Org. Lett.* **2000**, *2*, 1749.

Other Reactions

Visible-light-activated asymmetric addition of hydrocarbons to pyridine-based ketones



9e) Feng, *ACS Catal.* **2022**, *12*, 5136.

Figure 9 Catalytic asymmetric hydrogenation using ruthenium and other reactions[9]

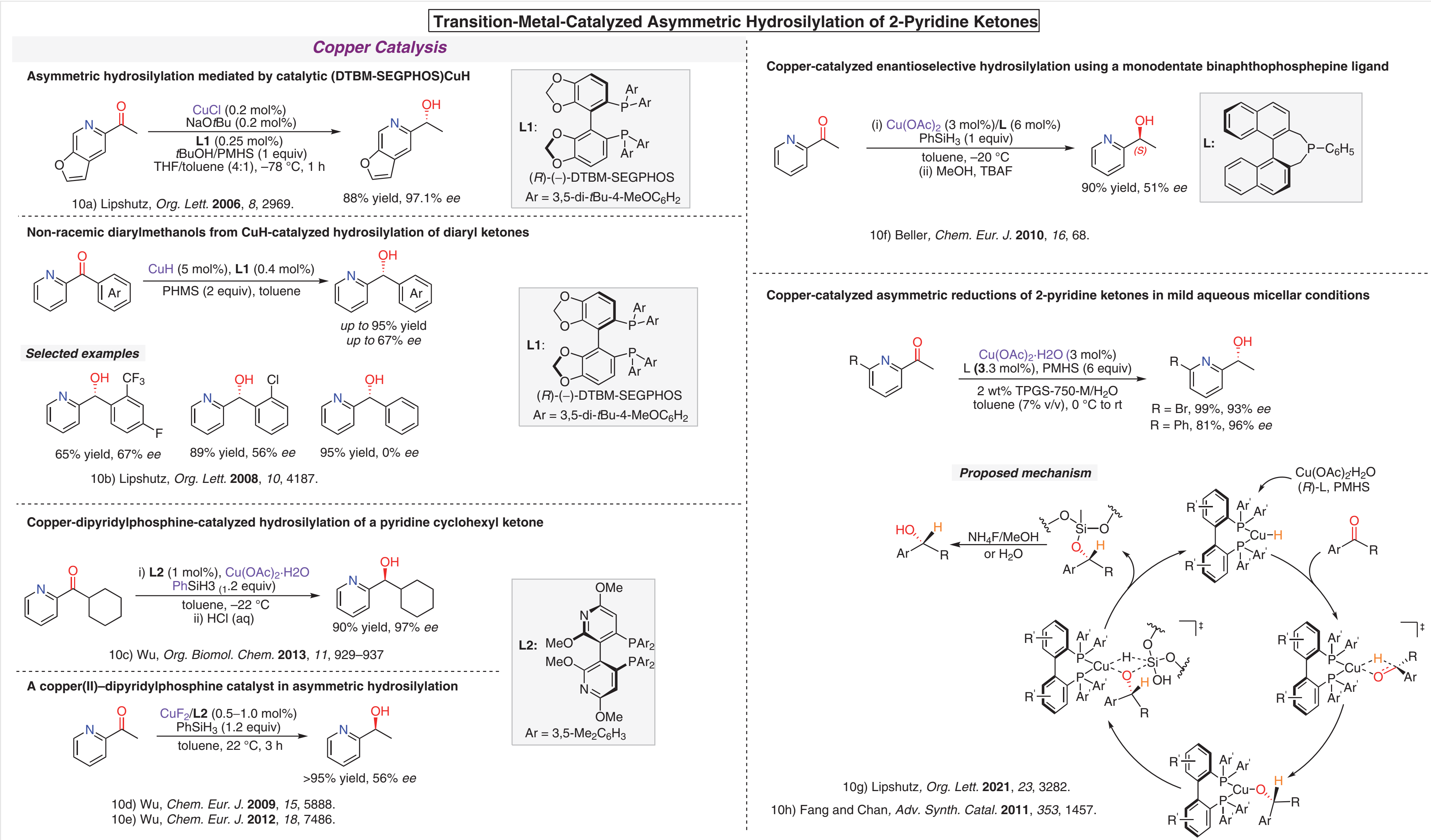
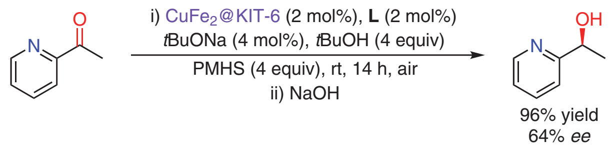
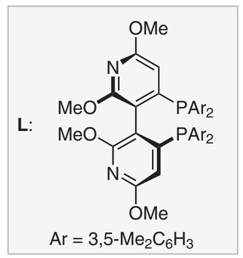


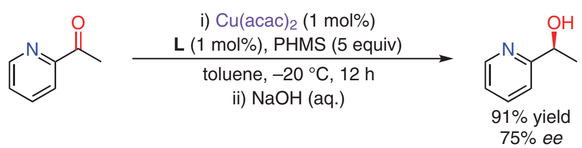
Figure 10 Copper-catalyzed asymmetric hydrosilylation[10]

Mesoporous silica KIT-6-supported superparamagnetic $CuFe_2O_4$ nanoparticles for asymmetric hydrosilylation in air



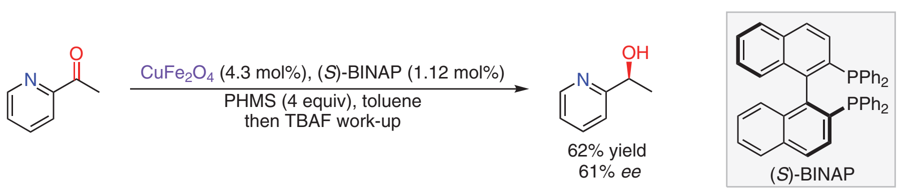
11a) Wu and Shi, *Green Chem.* **2014**, *16*, 2680.





10h) Fang and Chan, *Adv. Synth. Catal.* **2011**, *353*, 1457.

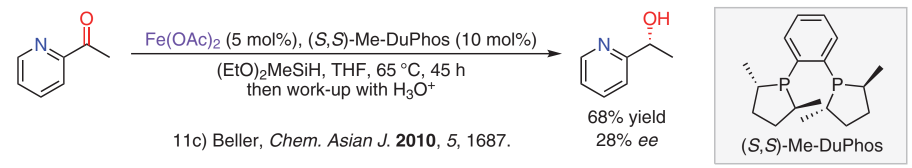
Copper-dipyridylphosphine-polymethylhydrosiloxane system for asymmetric catalytic hydrosilylation



11b) Figueras, *J. Org. Chem.* **2009**, *74*, 4608.

Iron Catalysis

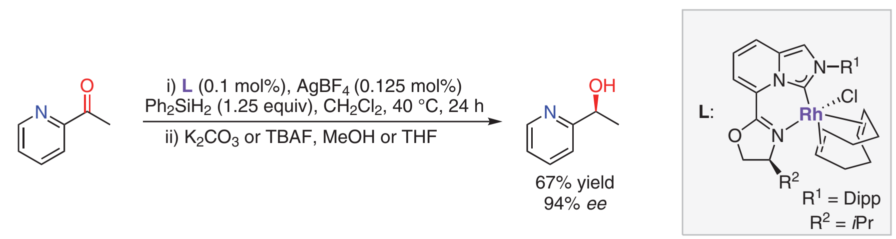
Stereoselective iron-catalyzed hydrosilylation of 2-pyridine ketones



11c) Beller, *Chem. Asian J.* **2010**, *5*, 1687.

Rhodium Catalysis

Chiral imidazo[1,5-*a*]pyridine–oxazolines as versatile NHC ligands for enantioselective hydrosilylation



11d) de Ruiter, *Organometallics* ***2020***, *39*, 247.

Figure 11 Catalytic asymmetric hydrosilylation using copper, iron and rhodium[10h,11]

Conflict of Interest

The authors declare no conflict of interest.

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