

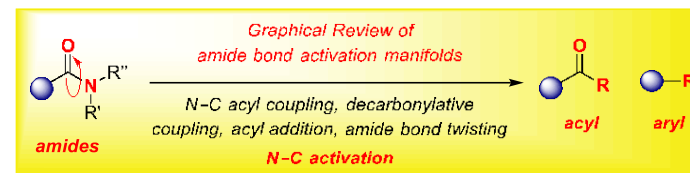
Amide N–C Bond Activation: A Graphical Overview of Acyl and Decarbonylative Coupling

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Michal Szostak^{*b}

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Abstract This Graphical Review provides an overview of amide bond activation achieved by selective oxidative addition of the N–C(O) acyl bond to transition metals and nucleophilic acyl addition, resulting in acyl and decarbonylative coupling together with key mechanistic details pertaining to amide bond distortion underlying this reactivity manifold.

Key words C–N activation, amide bond activation, acyl coupling, decarbonylative coupling, acyl addition, catalysis

The importance of amide bond is undeniable. The amide bond is the fundamental linkage of life in peptides and proteins. Due to its special dipolar character, amides are indispensable in pharmaceuticals, pesticides, polymers. At present, more than 50% of drug candidates contain amide bonds. Remarkably, reactions of amides are the most common type of reactions used in current medicinal chemistry.

Typical planar amides feature strong amidic resonance, $n_N \rightarrow \pi^*_{C=O}$ conjugation (15–20 kcal/mol), which renders amide bond cleavage extremely difficult. However, the amide bond can be sterically-twisted or electronically-activated by functionalizing the nitrogen atom of the amide bond. In this way, the amide bond resonance can be significantly decreased or diverted onto the activating group, thus enabling highly selective activation of N–C(O) amide bonds.

The recent years have seen an explosion of amide bond activation methods. Although the concept of amide bond twisting and the concurrent decrease of amidic resonance in bridged lactams was proposed as early as in 1930s, it was not until 2015 that generic acyclic twisted amides were used for the first time as cross-coupling partners in selective N–C(O) bond activation, thus effectively serving as surrogates for acyl and aryl halides and pseudohalides in transition-metal-catalysis.

Amide bond activation can be categorized as acyl coupling and decarbonylative coupling. This reactivity is triggered by selective oxidative addition of the N–C(O) acyl bond to transition metal, leading to either direct transmetalation or CO de-insertion. Furthermore, the successful use of amides as acyl halide equivalents in transition-metal-catalysis spearheaded the development of an array of highly selective methods for nucleophilic acyl addition to amide bonds, resulting in an alternative disconnection to acyl products. In many cases, the direct nucleophilic acyl addition shows advantages over transition-metal-catalyzed variants; however, it should be noted that these manifolds are broadly complementary.

This Graphical Review provides an overview of the key studies in amide bond activation covering the period of 2015 to 2022. The goal of this Graphical Review is to provide a summary of the reactions developed and manifolds established in the main areas of amide bond activation, including acyl and decarbonylative coupling as well as acyl nucleophilic addition, while highlighting the underlying mechanisms and amides that are critical to this reactivity manifold. Throughout the review we have attempted to cite the seminal reports and precedents. However, the Reader should note that due to the format of this review and the large number of contributions, the review is not comprehensive.

Throughout the review, the reactions are categorized by the type of mechanism. An important aspect that the Reader should pay special attention to is the role of specific amides that participate in each reaction of manifold. In general, sterically-twisted or electronically-activated amides can be prepared (1) from carboxylic acids or derivatives, or (2) from generic 1° or 2° amides. Both methods are valuable in terms of synthetic advantages of amide bonds in cross-coupling and acyl addition chemistry. However, for derivatization of biomolecules and late-stage functionalization of pharmaceuticals, only amides that can be generically prepared from 1° or 2° amides are useful. We hope that the review will stimulate further progress in this tremendously important field of chemistry.



Chengwei Liu received his PhD from Rutgers University with Prof. Michal Szostak in 2020. He conducted his postdoctoral research at University of Oxford with Prof. Stephen P. Fletcher from 2020 to 2021. Then he performed as an Assistant Professor at Nanjing University of Information Science and Technology from 2021 to 2022. In the summer of 2022, he joined the faculty at Shanghai University, where he started his independent career and was promoted to Full Professor in 2022. His research group is focused on amide bond activation, C–O bond activation, C–S bond activation, and lanthanide organometallic chemistry.

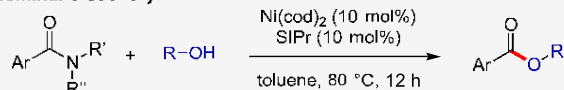


Michal Szostak received his Ph.D. from the University of Kansas in 2009. He carried out postdoctoral research at Princeton University and University of Manchester. In 2014, he joined the faculty at Rutgers University, where he is currently Professor of Chemistry. His research group developed the concept of acyclic twisted amide bond activation. In 2022, he edited the book “Amide Bond Activation: Concepts and Reactions”. His current research is focused on the development of new synthetic methodology based on transition-metal-catalysis, amide bond activation, NHC ligands, inert bond activation, and application to the synthesis of biologically active molecules. He is the author of over 240 publications.

- New concept
- Bench-stable electrophiles
- Non-toxic

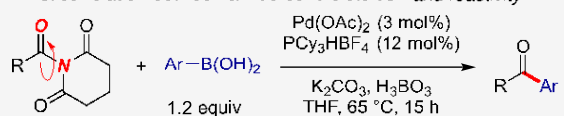
(1a) Szostak, *Amide Bond Activation: Concepts and Reactions*, Wiley, 2022.

Seminal discovery

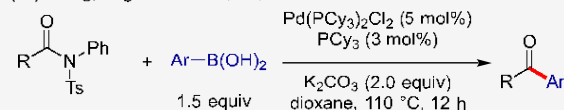


(1b) Hie, *Nature* **2015**, 524, 79.

First correlation between amide bond distortion and reactivity



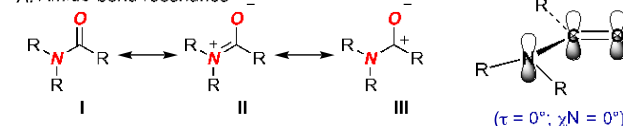
(1c) Meng, *Org. Lett.* **2015**, 17, 4364.



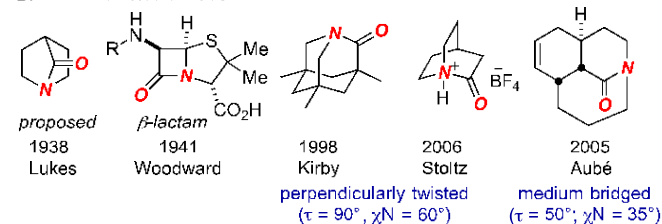
(1d) Li, *Chem. Commun.* **2015**, 51, 5089.

Concept was first introduced: C-N bond activation of twisted amides

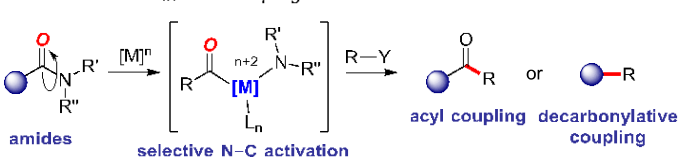
A. Amide bond resonance



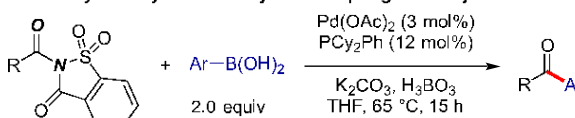
B. Classic twisted amides



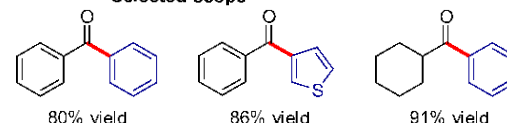
C. Twisted amides in cross-coupling reactions



Pd-catalyzed acyl Suzuki-Miyaura coupling of *N*-acylsaccharins

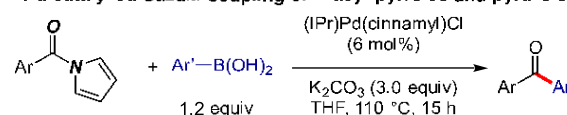


Selected scope

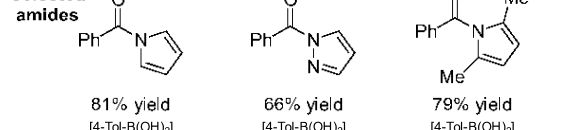


(1t) Liu, *Org. Lett.* **2016**, 18, 4194.

Pd-catalyzed Suzuki coupling of *N*-acyl pyrroles and pyrazoles

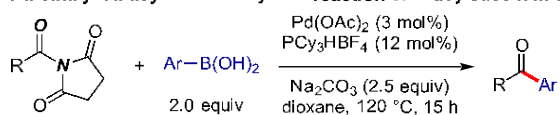


Selected amides



(1u) Meng, *Org. Lett.* **2017**, 19, 3596.

Pd-catalyzed acyl Suzuki-Miyaura reaction of *N*-acylsuccinimides



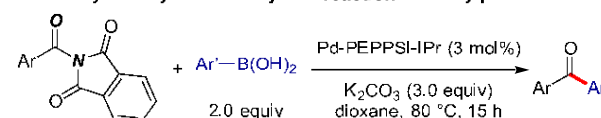
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Further reading

Reviews on twisted amide bond activation:

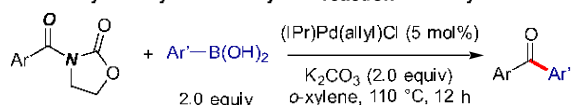
- (1e) Szostak, *Chem. Rev.* **2013**, 113, 5701.
- (1f) Meng, *Chem. Rev.* **2021**, 121, 12746.
- (1g) Meng, *Synlett* **2016**, 27, 2530.
- (1h) Liu, *Chem. Eur. J.* **2017**, 23, 7157.
- (1i) Meng, *Eur. J. Org. Chem.* **2018**, 2352.
- (1j) Takise, *Chem. Soc. Rev.* **2017**, 46, 5864.
- (1k) Kaiser, *Chem. Soc. Rev.* **2018**, 47, 7899.
- (1l) Dander, *ACS Catal.* **2017**, 7, 1413.
- (1m) Adachi, *Tetrahedron Lett.* **2018**, 59, 1147.
- (1n) Guo, *Chem. Eur. J.* **2018**, 24, 7794.
- (1o) Bourne-Branchu, *Chem. Eur. J.* **2019**, 25, 2663.
- (1p) Chaudhari, *Chem. Asian J.* **2019**, 14, 76.
- (1q) Li, *Trends Chem.* **2020**, 2, 914.
- (1r) Gao, *J. Org. Chem.* **2022**, 87, DOI: 10.1021/acs.joc.2c01094.
- (1s) Feng, *Angew. Chem. Int. Ed.* **2022**, 61, e202212213.

Pd-catalyzed acyl Suzuki-Miyaura reaction of *N*-acylphthalimides



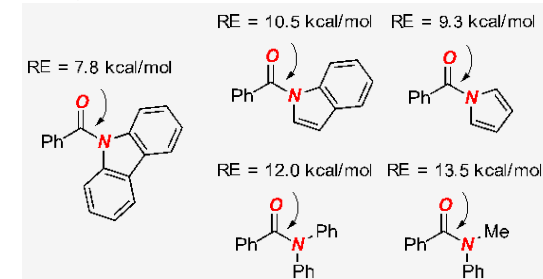
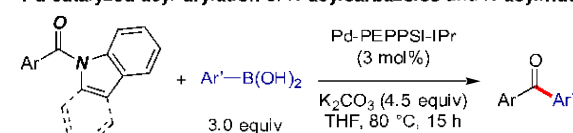
(1w) Rahman, *Catalysts* **2019**, 9, 129.

Pd-catalyzed acyl Suzuki-Miyaura reaction of *N*-acyloxazolidinones



(1x) Jian, *Eur. J. Org. Chem.* **2020**, 4176.

Pd-catalyzed acyl arylation of *N*-acylcarbazoles and *N*-acylindoles



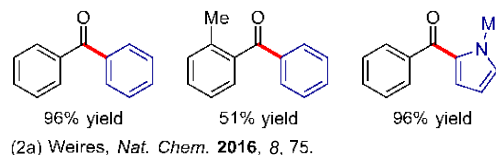
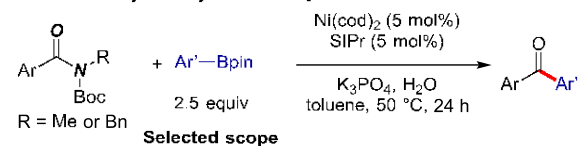
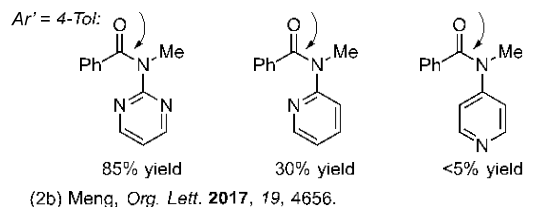
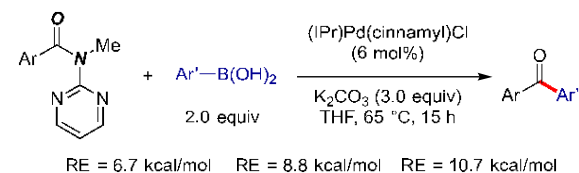
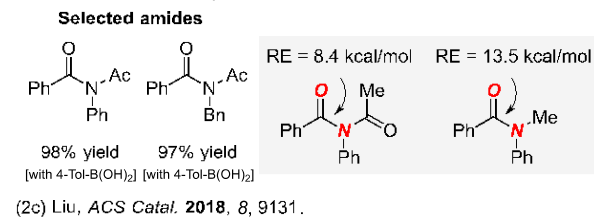
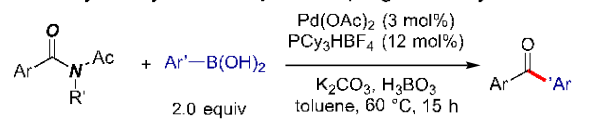
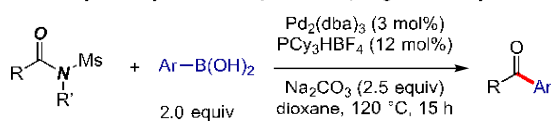
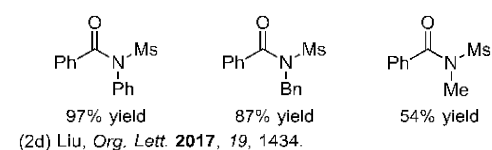
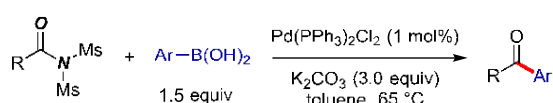
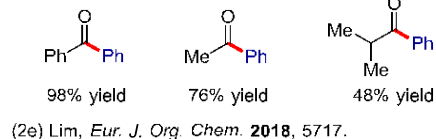
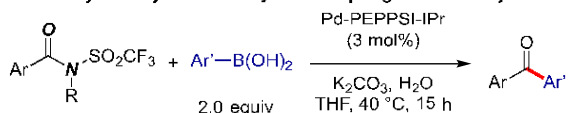
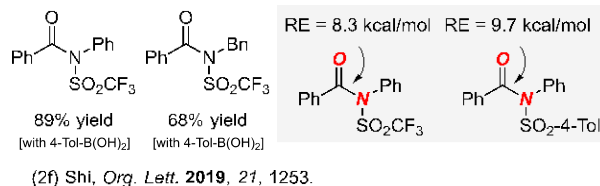
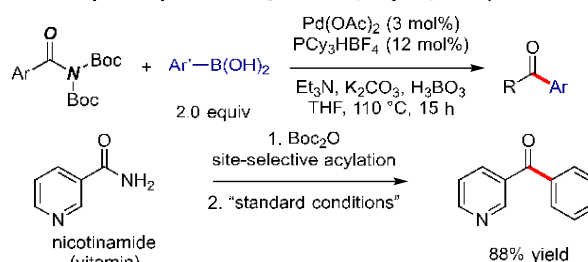
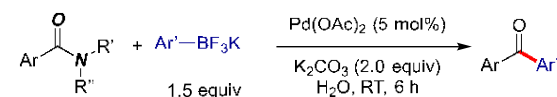
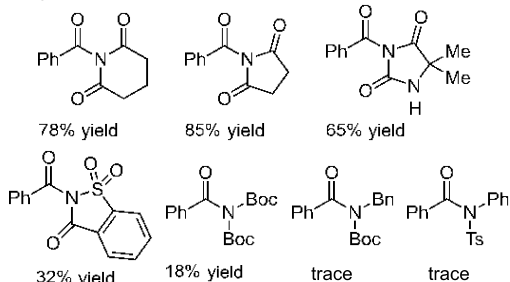
(1y) Buchspies, *Org. Lett.* **2020**, 22, 4703.

Further reading

Other selected contributions:

- (1z) Meng, *Org. Biomol. Chem.* **2016**, 14, 5690.
- (1aa) Wu, *Adv. Synth. Catal.* **2016**, 358, 3876.
- (1ab) Cui, *Tetrahedron Lett.* **2017**, 58, 3819.
- (1ac) Wang, *J. Organomet. Chem.* **2018**, 877, 80.
- (1ad) Liu, *Org. Lett.* **2019**, 21, 7976.
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- (1af) Ma, *Adv. Synth. Catal.* **2020**, 362, 1887.
- (1ag) Luo, *Org. Process Res. Dev.* **2018**, 22, 1188.
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Figure 1 Amide bond activation: concept and discoveries.¹

Nickel-catalyzed acyl Suzuki-Miyaura reaction of amides**Pd-catalyzed Suzuki coupling of *N*-methylamino pyrimidyl amides****Pd-catalyzed acyl Suzuki-Miyaura coupling of *N*-acetyl amides****Pd-catalyzed acyl Suzuki-Miyaura coupling of *N*-mesyl amides**Ar = 4-Tol: **Selected amides****Pd-catalyzed acyl Suzuki-Miyaura coupling of *N,N*-di-Ms amides****Selected scope****Pd-catalyzed acyl Suzuki-Miyaura coupling of *N*-sulfonyl amides****Selected amides****Pd-catalyzed acyl Suzuki-Miyaura coupling of primary amides****Green method for acyl Suzuki-Miyaura reaction of amides**when Ar' = Ph **Selected amides****Further reading**

Other selected contributions:

- (2i) Li, *J. Organomet. Chem.* **2015**, *794*, 136.
 (2j) Szostak, *J. Org. Chem.* **2016**, *81*, 8091.
 (2k) Pace, *Chem. Eur. J.* **2016**, *22*, 14494.
 (2l) Pace, *Chem. Commun.* **2019**, *55*, 4423.
 (2m) Szostak, *J. Org. Chem.* **2017**, *82*, 6373.
 (2n) Meng, *J. Am. Chem. Soc.* **2018**, *140*, 727.
 (2o) Szostak, *Org. Lett.* **2018**, *20*, 1342.
 (2p) Szostak, *J. Org. Chem.* **2018**, *83*, 14676.
 (2q) Ielo, *Chem. Eur. J.* **2020**, *26*, 16246.
 (2r) Liu, *Org. Lett.* **2018**, *20*, 7771.
 (2s) Boit, *ACS Catal.* **2018**, *8*, 1003.
 (2t) Mai, *Eur. J. Org. Chem.* **2019**, 7814.
 (2u) Mehta, *Org. Lett.* **2020**, *22*, 1.
 (2v) Shi, *Tetrahedron Lett.* **2020**, *61*, 152140.
 (2w) Wang, *ACS Catal.* **2022**, *12*, 2426.
 (2x) Zhang, *Angew. Chem. Int. Ed.* **2022**, *61*, e202114146.
 (2y) Li, *Chem. Eur. J.* **2021**, *27*, 2699.

Figure 2 Transition-metal-catalyzed acyl Suzuki-Miyaura coupling of amides.²

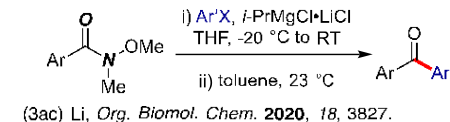
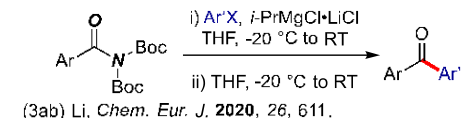
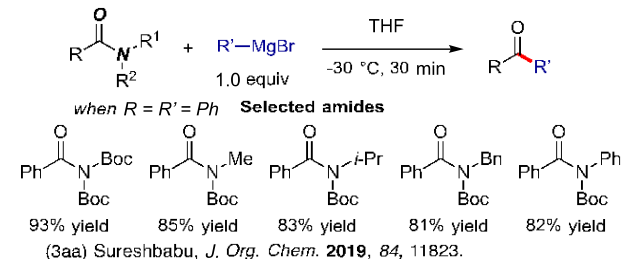
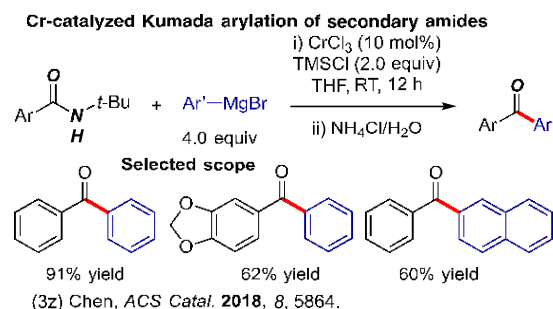
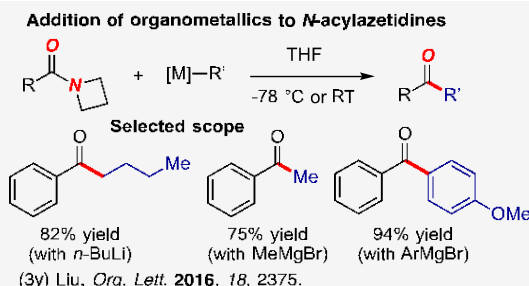
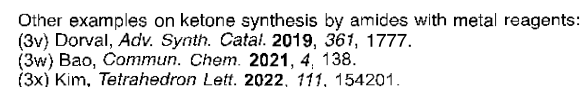
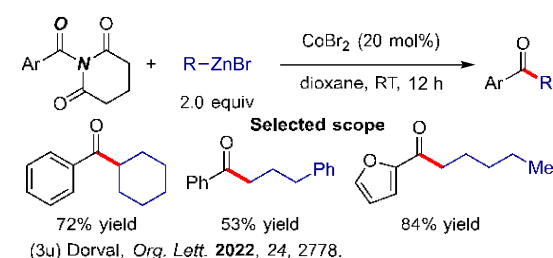
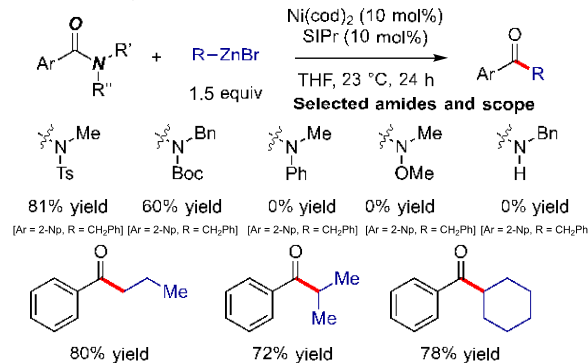
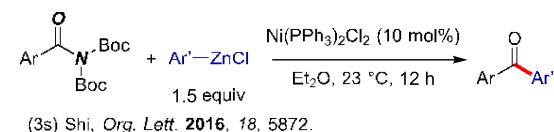
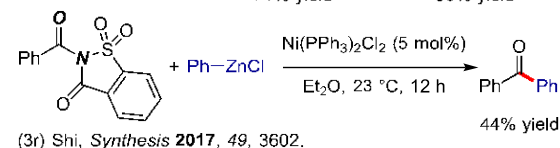
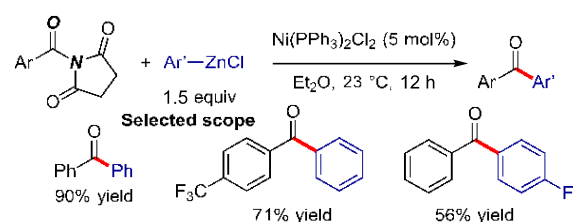
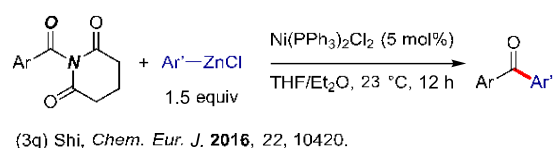
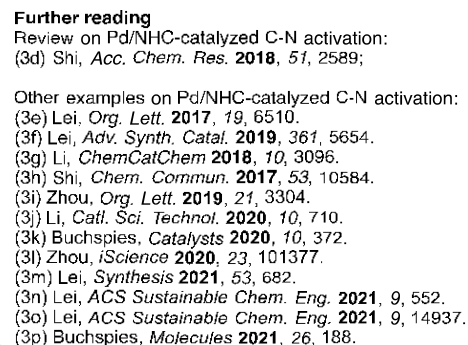
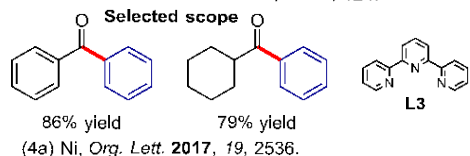
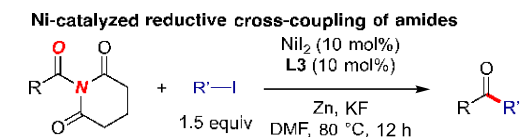
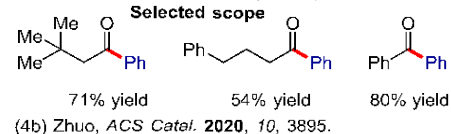
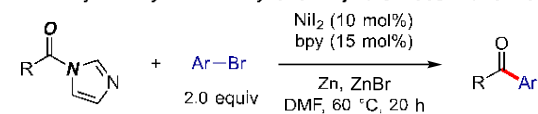


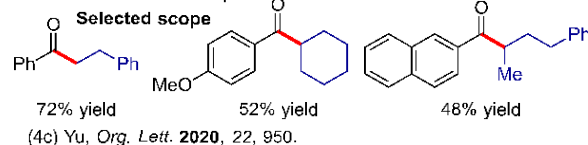
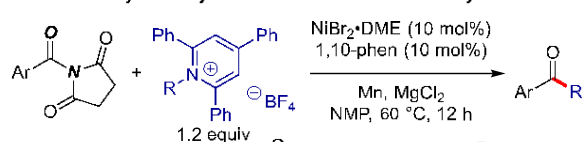
Figure 3 Synthesis of ketone via transition-metal-catalysis and metal-free conditions.³



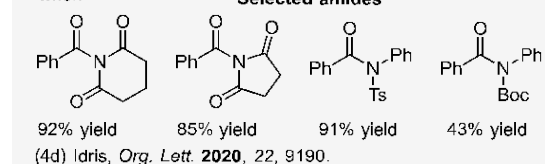
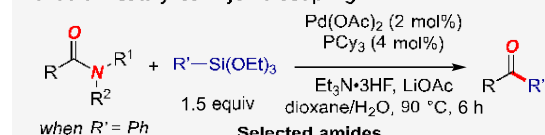
Ni-catalyzed acylation of aryl and alkyl bromides with amides



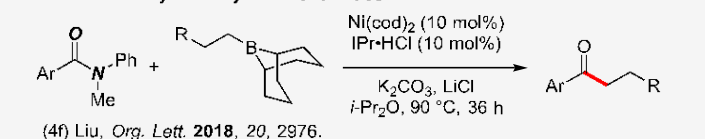
Nickel-catalyzed alkylation of amides with Katritzky salts



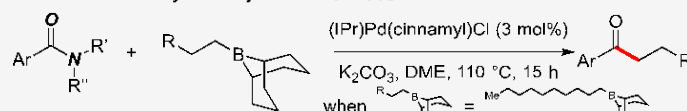
Palladium-catalyzed Hiyama coupling of amides



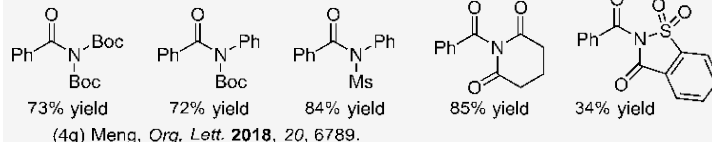
Nickel-catalyzed alkylation of amides



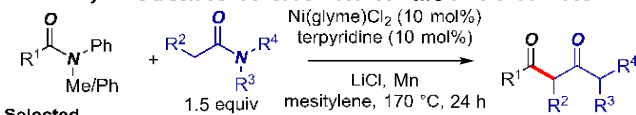
Palladium-catalyzed alkylation of amides



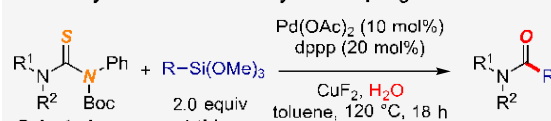
Selected amides



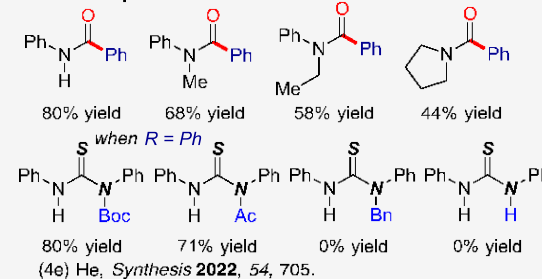
Ni-catalyzed Claisen condensation between two different amides



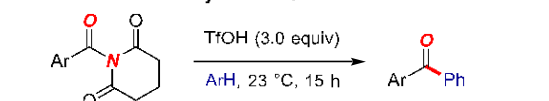
Pd-catalyzed desulfurative Hiyama coupling of thioureas



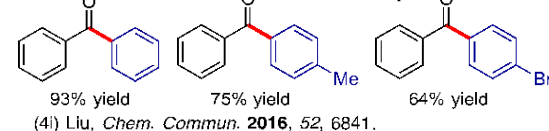
Selected scope and thioureas



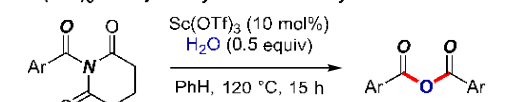
Friedel-Crafts acylation with twisted amides



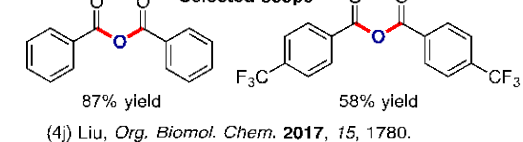
Selected scope



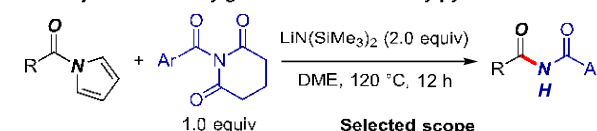
Sc(OTf)3-catalyzed synthesis of anhydrides from amides



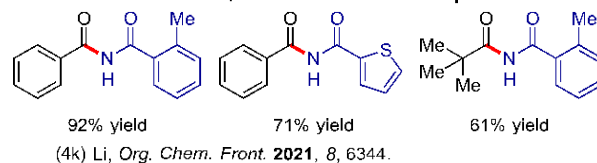
Selected scope



Acylation of N-acylglutarimides with N-acylpyrroles



Selected scope



Further reading

Other contributions on cross-coupling of amides:

(4l) Amani, *Org. Lett.* 2017, 19, 2426.

(4m) Shi, *Molecules* 2018, 23, 2412.

(4n) Mahesh, *Molecules* 2018, 23, 2615.

(4o) Wang, *Tetrahedron. Lett.* 2018, 59, 2299.

(4p) Kadam, *ACS Catal.* 2019, 9, 5651.

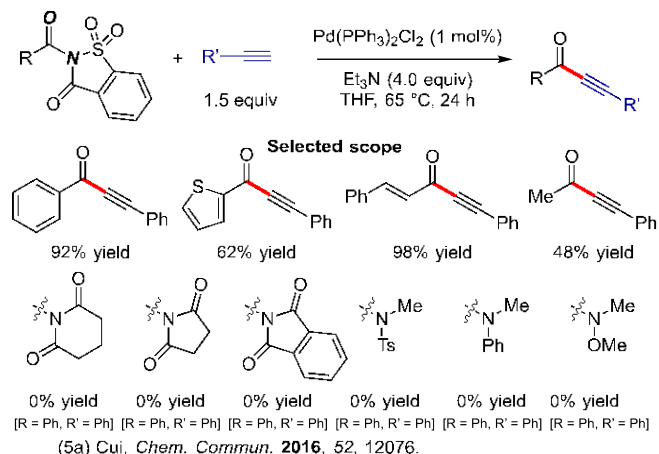
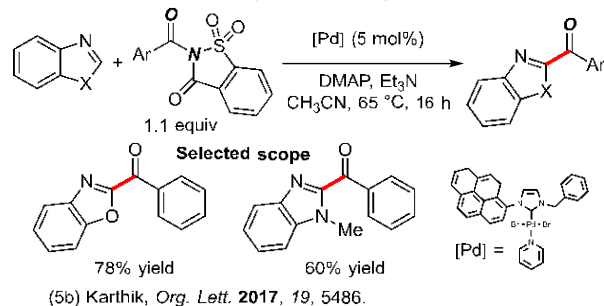
(4q) Koertiz, *Org. Lett.* 2020, 22, 5731.

(4r) Reina, *ACS Catal.* 2020, 10, 2189.

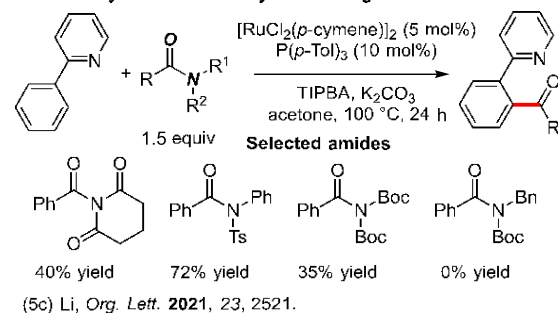
(4s) Kerackian, *Org. Lett.* 2020, 22, 2240.

(4t) Kerackian, *ACS Catal.* 2022, 12, 12315.

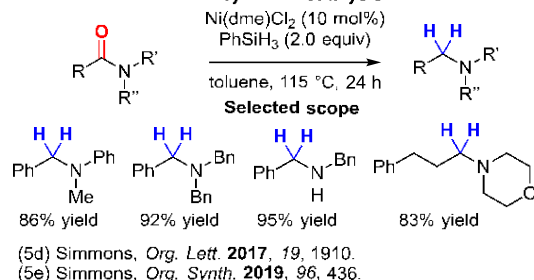
Figure 4 Amides as acylating reagents in various synthetic methodologies.⁴

Pd-catalyzed Sonogashira coupling of *N*-acylsaccharinsPd/NHC-catalyzed heteroarylation of *N*-acylsaccharins

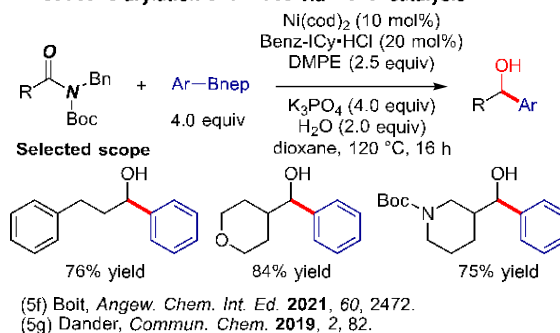
Ru-catalyzed aromatic acylation using amides



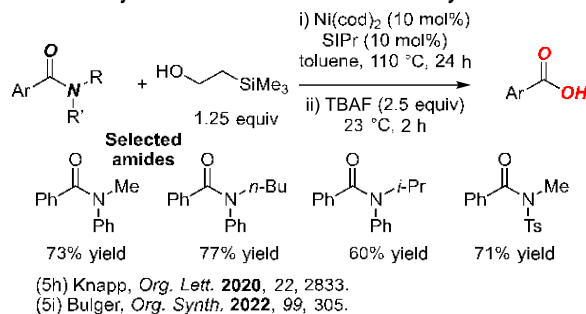
Reduction of amides by nickel catalysis



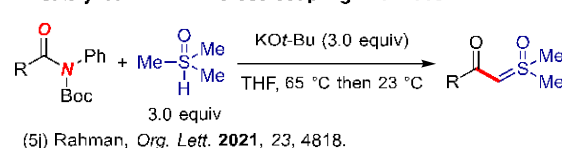
Reductive arylation of amides via nickel catalysis



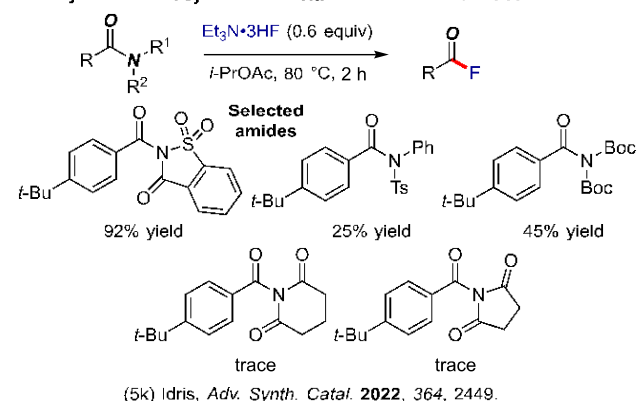
Ni-catalyzed conversion of amides to carboxylic acids



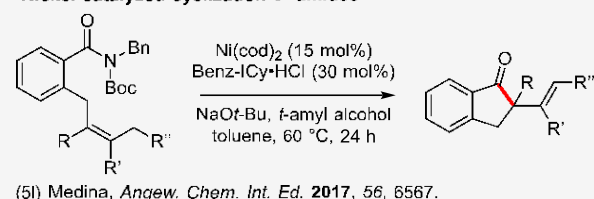
Ni-catalyzed reductive cross-coupling of amides



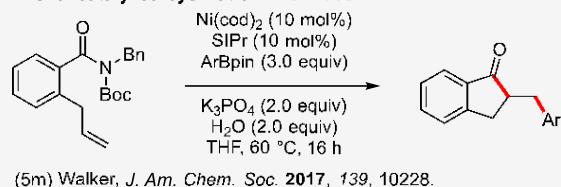
Synthesis of acyl fluorides via fluorination of amides



Nickel-catalyzed cyclization of amides



Nickel-catalyzed cyclization of amides



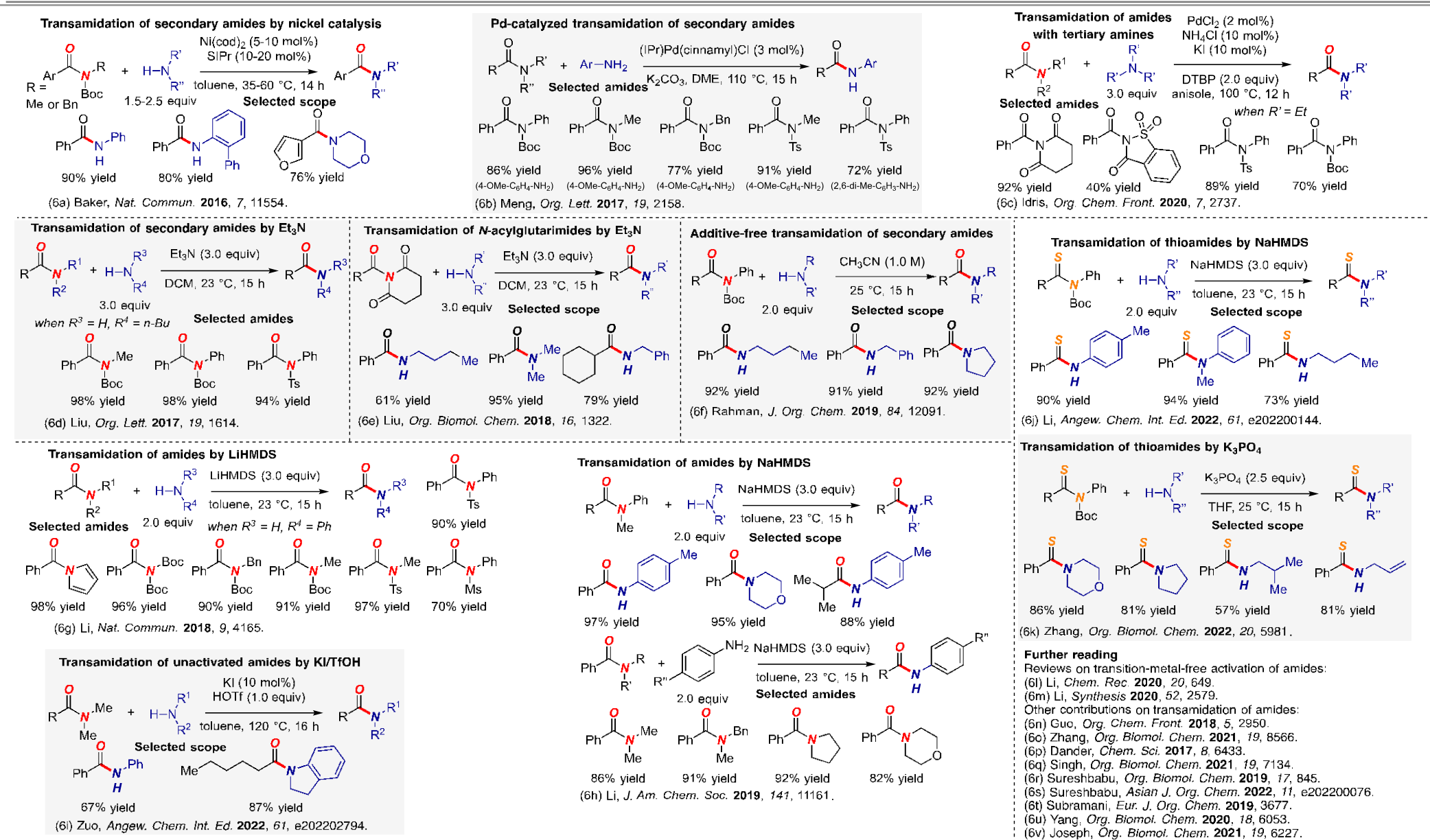
Further reading

Other contributions on synthesis of benzoxazoles from amides:
 (5n) Luo, *Adv. Synth. Catal.* **2019**, 361, 4117.

Other contributions on reduction of amides:
 (5o) Azeez, *Org. Biomol. Chem.* **2022**, 20, 2048.

Other contributions on computational studies for amides:
 (5p) Liu, *J. Org. Chem.* **2016**, 81, 11686.
 (5q) Chu, *J. Org. Chem.* **2018**, 83, 5009.
 (5r) Ji, *J. Am. Chem. Soc.* **2017**, 139, 15522.
 (5s) Wang, *Chem. Commun.* **2019**, 55, 11330.
 (5t) Tong, *Chem. Commun.* **2020**, 56, 113.
 (5u) Li, *J. Org. Chem.* **2020**, 85, 833.

Figure 5 Cross-coupling of amides with versatile partners.⁵

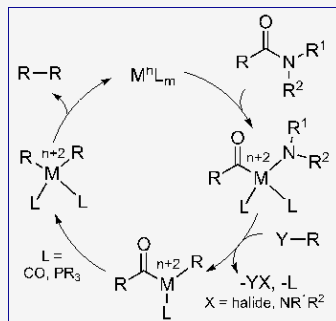
Figure 6 Transamidation of amides under metal-catalysis and metal-free conditions.⁶

(7m) Nagae, *Chem. Sci.* **2019**, *10*, 2860.

Notable features

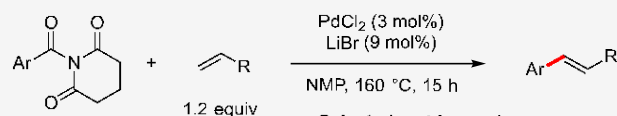
- New concept
- Bench-stable electrophiles
- Non-toxic

Reaction mechanism in decarbonylative coupling of amides

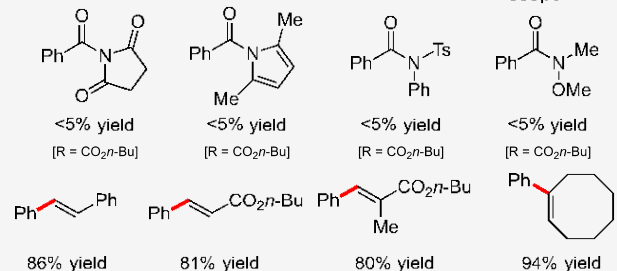
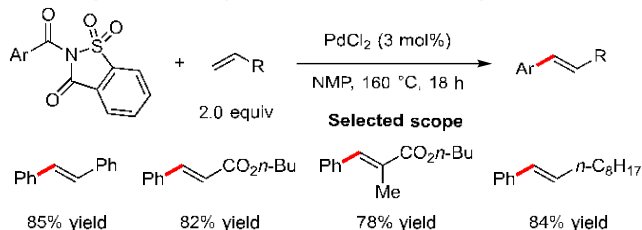


First example in decarbonylative coupling of amides:

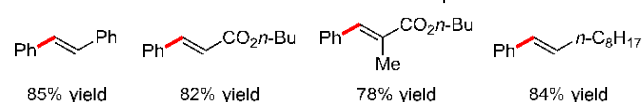
Pd-catalyzed decarbonylative Heck reaction of amides



Selected amides and scope

(8a) Meng, *Angew. Chem. Int. Ed.* **2015**, *54*, 14518.Pd-catalyzed decarbonylative Heck reaction of *N*-acylsaccharins

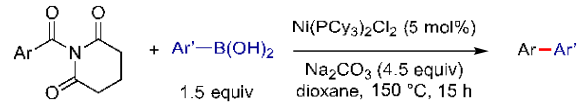
Selected scope

(8b) Liu, *J. Org. Chem.* **2016**, *81*, 12023.

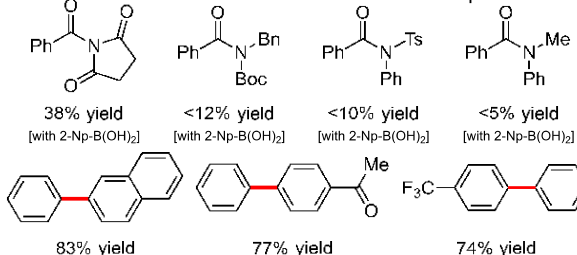
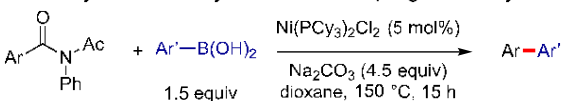
Further reading

Reviews on decarbonylative coupling of amides:
(8c) Liu, *Org. Biomol. Chem.* **2018**, *16*, 7998;

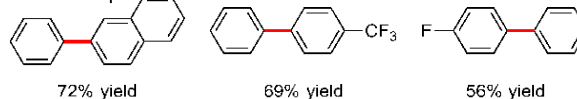
Ni-catalyzed decarbonylative Suzuki coupling of amides



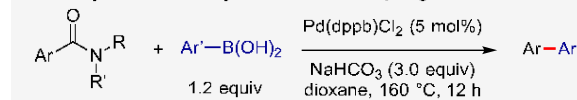
Selected amides and scope

(8d) Shi, *Angew. Chem. Int. Ed.* **2016**, *55*, 6959.Ni-catalyzed decarbonylative Suzuki coupling of *N*-acetyl amides

Selected scope

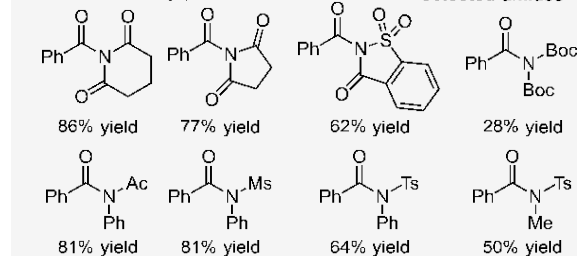
(2c) Liu, *ACS Catal.* **2018**, *8*, 9131.

Pd-catalyzed decarbonylative Suzuki coupling of amides

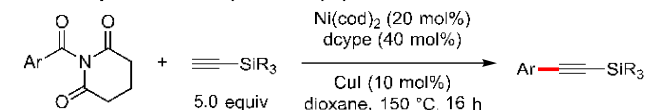


when Ar' = 4-OMe-C6H4-

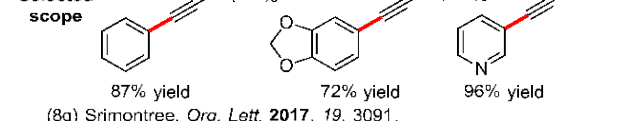
Selected amides

(8e) Zhou, *Chem. Sci.* **2019**, *10*, 9865.

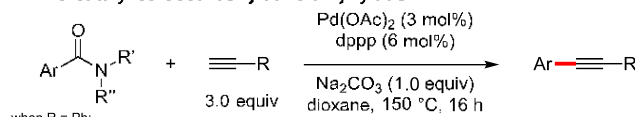
Ni-catalyzed decarbonylative alkylation of amides



Selected scope

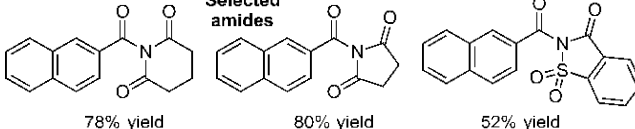
(8g) Srimontree, *Org. Lett.* **2017**, *19*, 3091.

Pd-catalyzed decarbonylative alkylation of amides

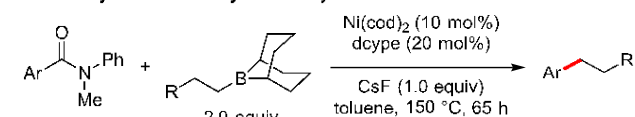


when R = Ph:

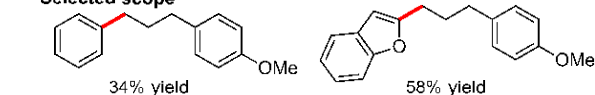
Selected amides

(8h) Liu, *Org. Lett.* **2018**, *20*, 2741.

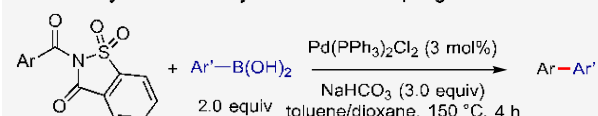
Ni-catalyzed decarbonylative alkylation of amides



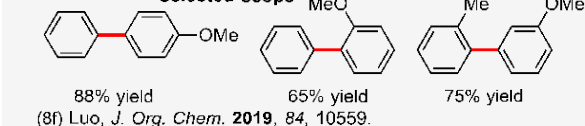
Selected scope

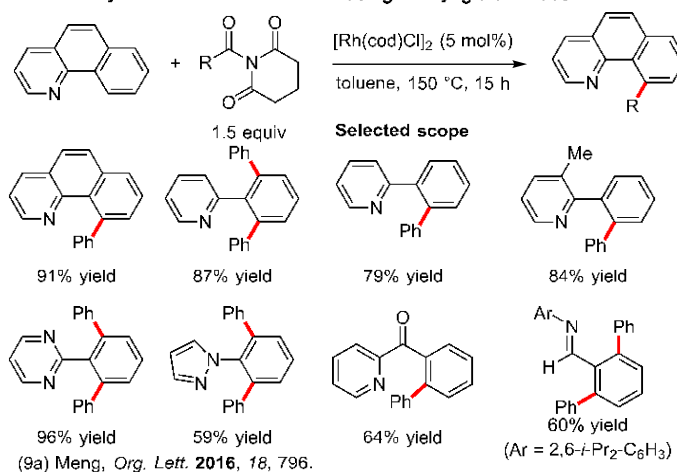
(8i) Chatupheeraphat, *J. Am. Chem. Soc.* **2018**, *140*, 3724.

Pd-catalyzed decarbonylative Suzuki coupling of amides

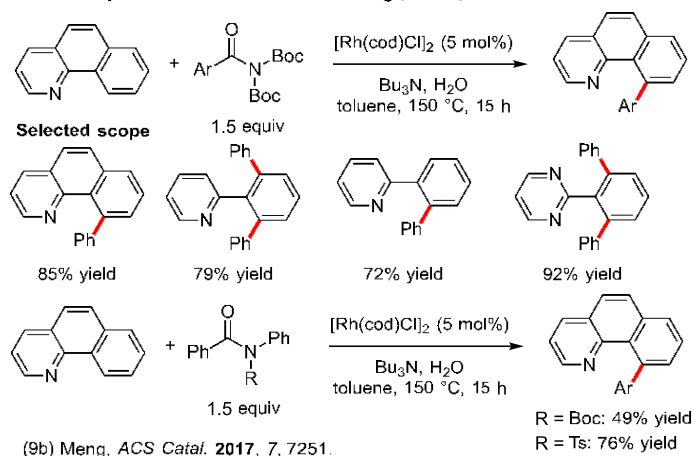


Selected scope

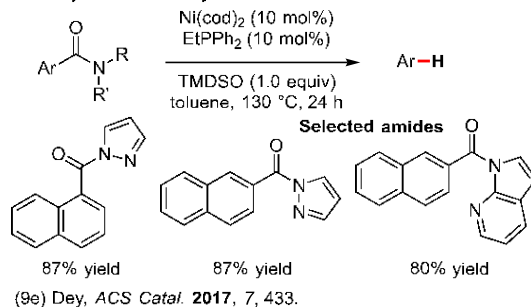
(8f) Luo, *J. Org. Chem.* **2019**, *84*, 10559.Figure 8 Decarbonylative cross-coupling of amides: discoveries and mechanism.⁸

Rh-catalyzed C-H functionalization using *N*-acyl glutarimides

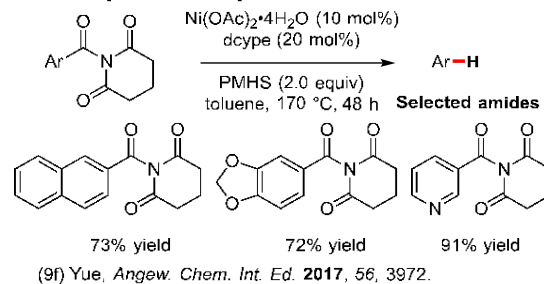
Rh-catalyzed C-H functionalization using primary amides



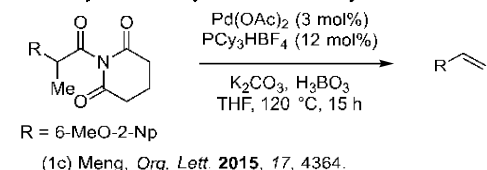
Ni-catalyzed decarbonylative reduction of amides



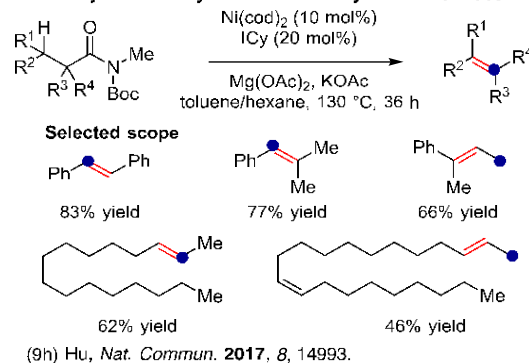
Ni-catalyzed decarbonylative reduction of amides



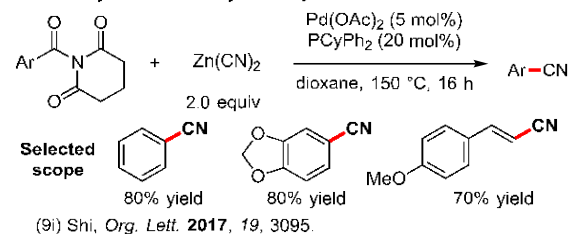
Pd-catalyzed retro-hydroamidocarbonylation of amides



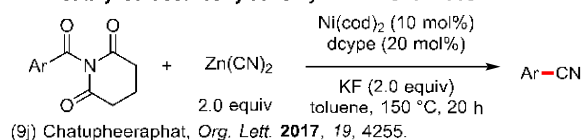
Ni-catalyzed retro-hydroamidocarbonylation of amides



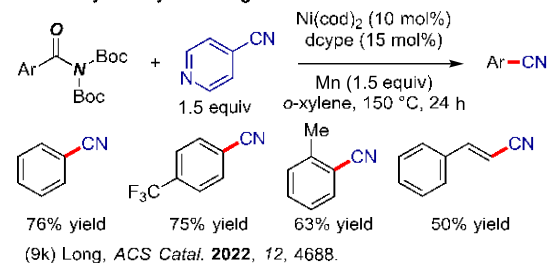
Pd-catalyzed decarbonylative cyanation of amides



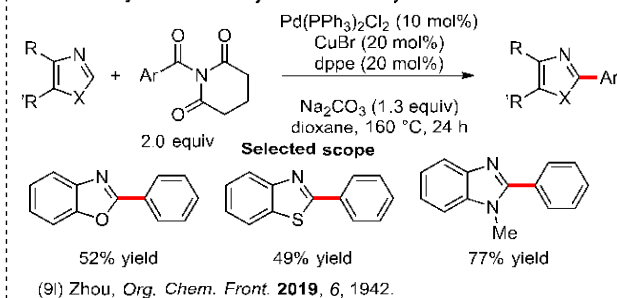
Ni-catalyzed decarbonylative cyanation of amides



Ni-catalyzed aryl exchange between amides and nitriles



Pd/Cu-catalyzed decarbonylative heteroarylation of amides

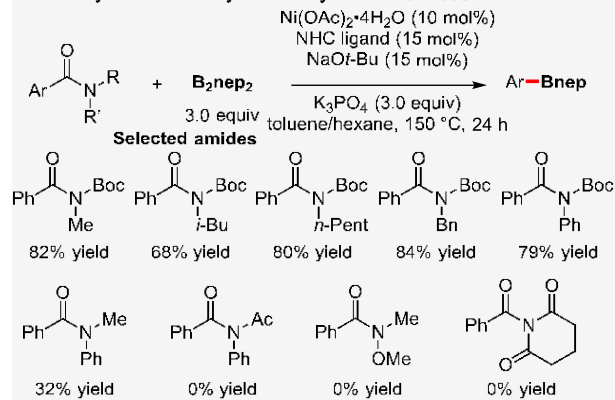
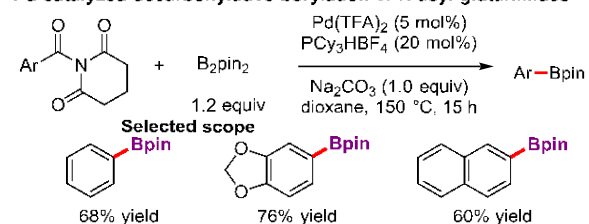


Further reading

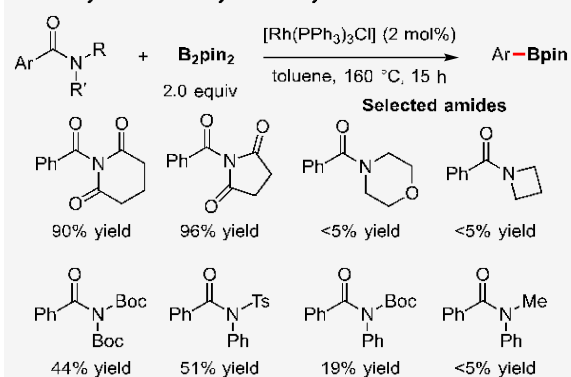
Other example on C-H functionalization of amides:
 (9c) Wu, *Org. Biomol. Chem.* **2017**, *15*, 536.
 (9d) Xiong, *Adv. Synth. Catal.* **2019**, *361*, 5383.
 Other example on reduction of amides:
 (9g) Mondal, *New J. Chem.* **2017**, *41*, 13211.

Figure 9 Decarbonylative cross-coupling of amides: construction of carbon-carbon and carbon-hydrogen bonds.⁹

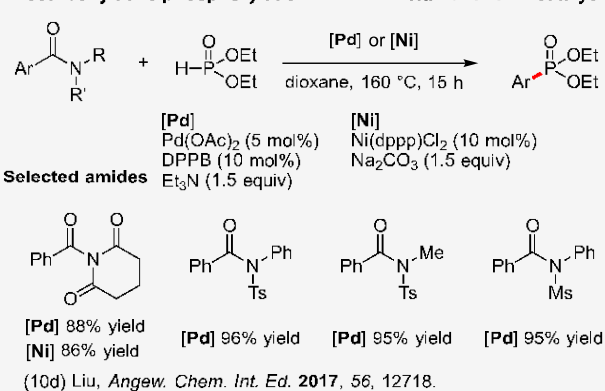
Ni-catalyzed decarbonylative borylation of amides

(10a) Hu, *Angew. Chem. Int. Ed.* **2016**, *55*, 8718.Pd-catalyzed decarbonylative borylation of *N*-acyl glutarimides(10b) Shi, *ACS Omega* **2019**, *4*, 4901.

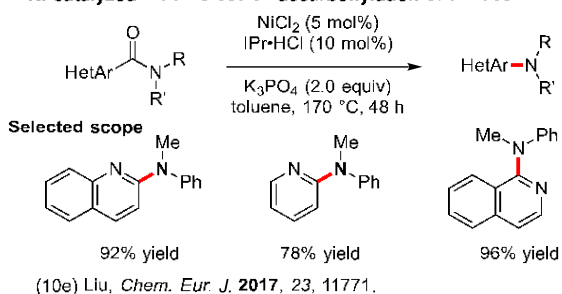
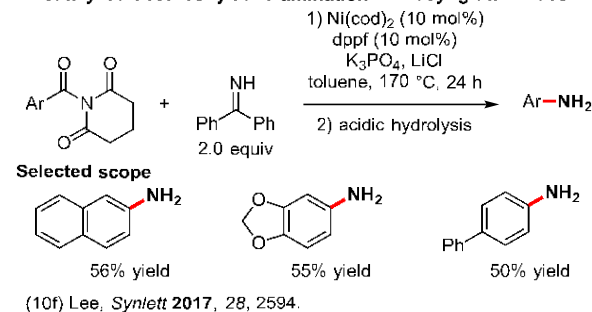
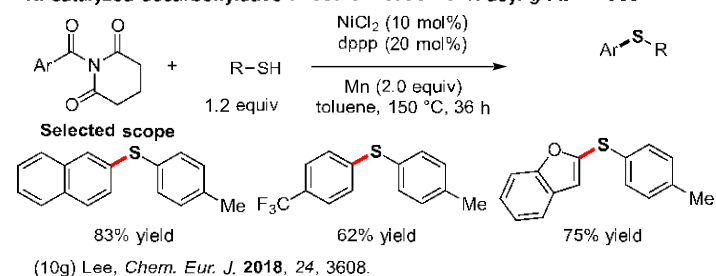
Rh-catalyzed decarbonylative borylation of amides

(10c) Bie, *J. Org. Chem.* **2020**, *85*, 15676.

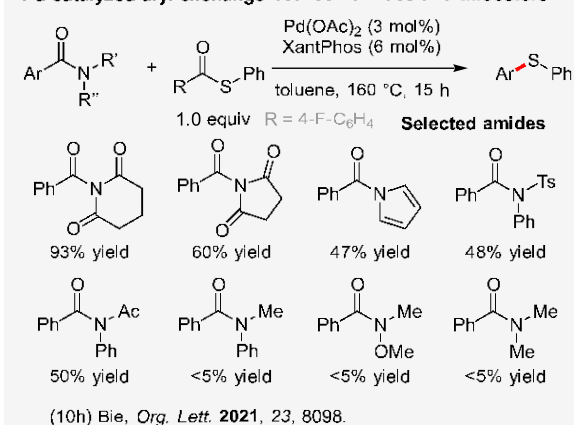
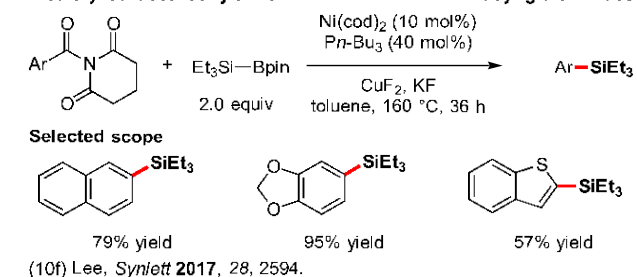
Decarbonylative phosphorylation of amides via Pd- and Ni-catalysis



Ni-catalyzed intramolecular decarbonylation of amides

Ni-catalyzed decarbonylative amination of *N*-acyl glutarimidesNi-catalyzed decarbonylative thioetherification of *N*-acyl glutarimides

Pd-catalyzed aryl exchange between amides and thioesters

Ni-catalyzed decarbonylative thioetherification of *N*-acyl glutarimides

Further reading

Example on decarbonylative amination of esters:
(10i) Yue, *Angew. Chem. Int. Ed.* **2017**, *56*, 4282.

Figure 10 Decarbonylative cross-coupling of amides: construction of carbon-heteroatom bonds.^{10,11}

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Acknowledgment

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Conflict of Interest

The authors declare no competing financial interest.

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