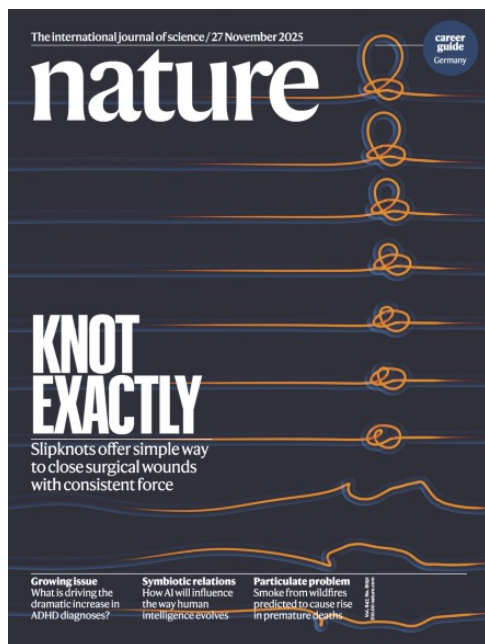


Deaminative Cross-Coupling of Amines by Boryl Radical β -Scission

Larisa P. Pop
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Article

Deaminative cross-coupling of amines by boryl radical β -scission

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Amines are among the most common functional groups in biologically active molecules and pharmaceuticals^{1–3}, yet they are almost universally treated as synthetic end points⁴. Here we report a strategy that repositions native primary, secondary and tertiary amines as handles for cross-coupling. The platform relies on in situ activation through borane coordination and exploits a copper catalytic redox system that generates amine-ligated boryl radicals, which undergo β -scission across the C(sp^3)–N bond to release alkyl radicals. These intermediates engage in copper-catalysed cross-couplings with a broad range of C-based, N-based, O-based and S-based nucleophiles. The method tolerates diverse amine classes, enables modular functionalization and supports late-stage diversification of complex drug scaffolds. Also, amides can be incorporated into the manifold through reductive funnelling. This work establishes a general approach to deaminative C–N bond functionalization and introduces a distinct approach for making and modifying drug-like molecules.

Daniele Leonori

2007 – 2010	PhD, University of Sheffield (Prof. Coldham, I.)
2010 – 2011	Postdoc, RWTH-Aachen University (Prof. Rueping, M.)
2011 – 2012	Postdoc, Max Planck Institute (Prof. Seeberger, P.)
2012 – 2014	Research Officer, University of Bristol (Prof. Aggarwal, V.)
2014 – 2017	Lecturer of Organic Chemistry, University of Manchester
2017 – 2018	EPSRC Early Career Fellow, University of Manchester
2018 – 2020	Reader in Organic Chemistry, University of Manchester
2020 – 2022	Professor, University of Manchester
from 2022	Professor, RWTH-Aachen University

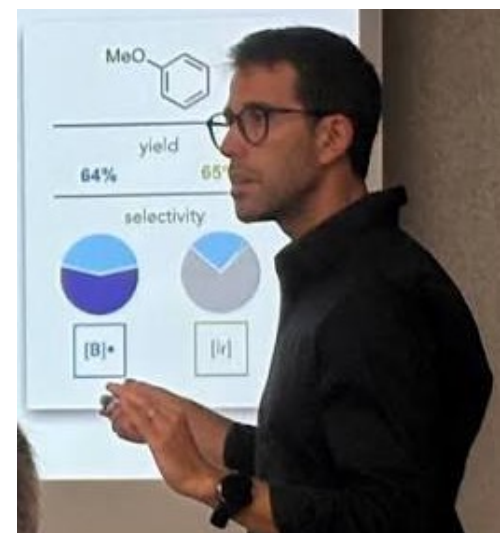


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Technische Hochschule
Aachen)*



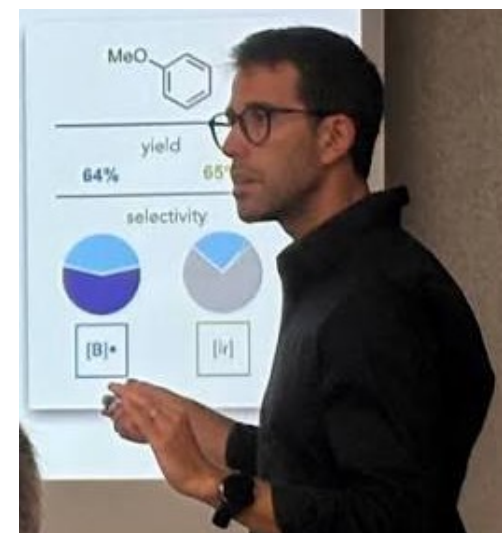
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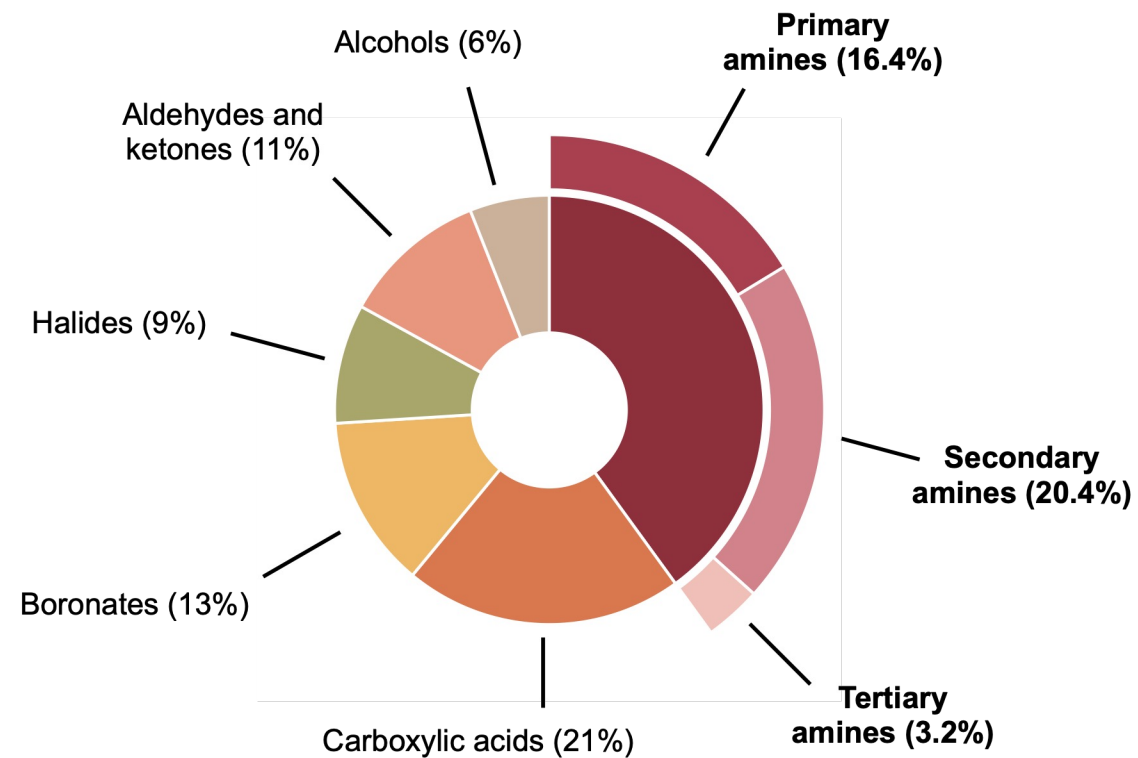
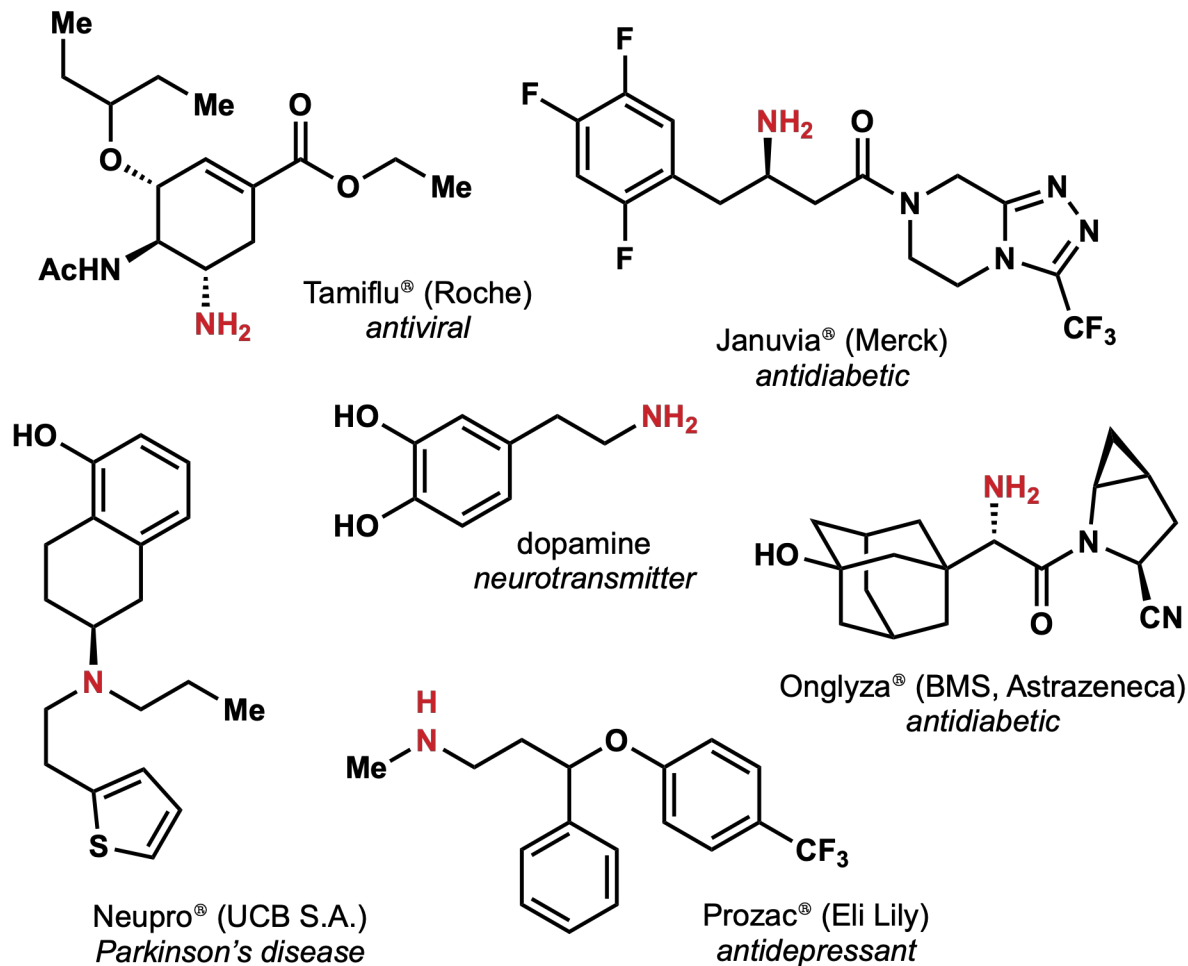


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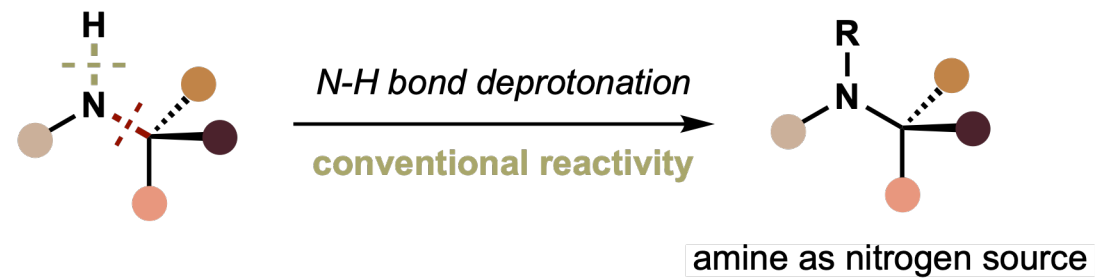
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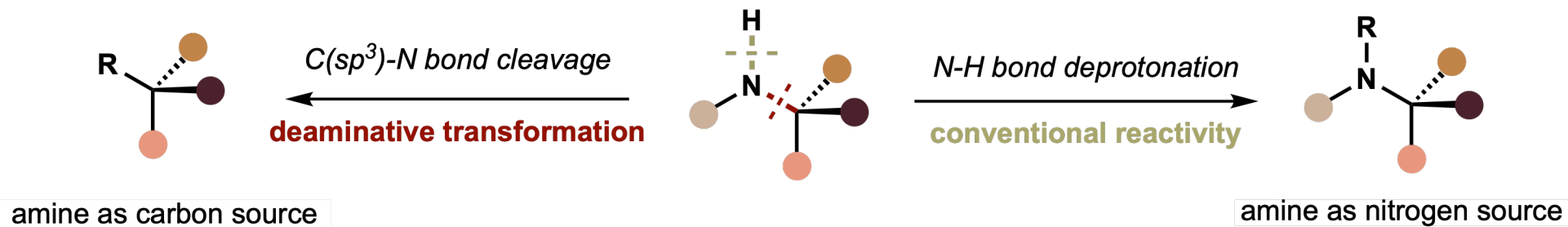
Why Starting From Amines?



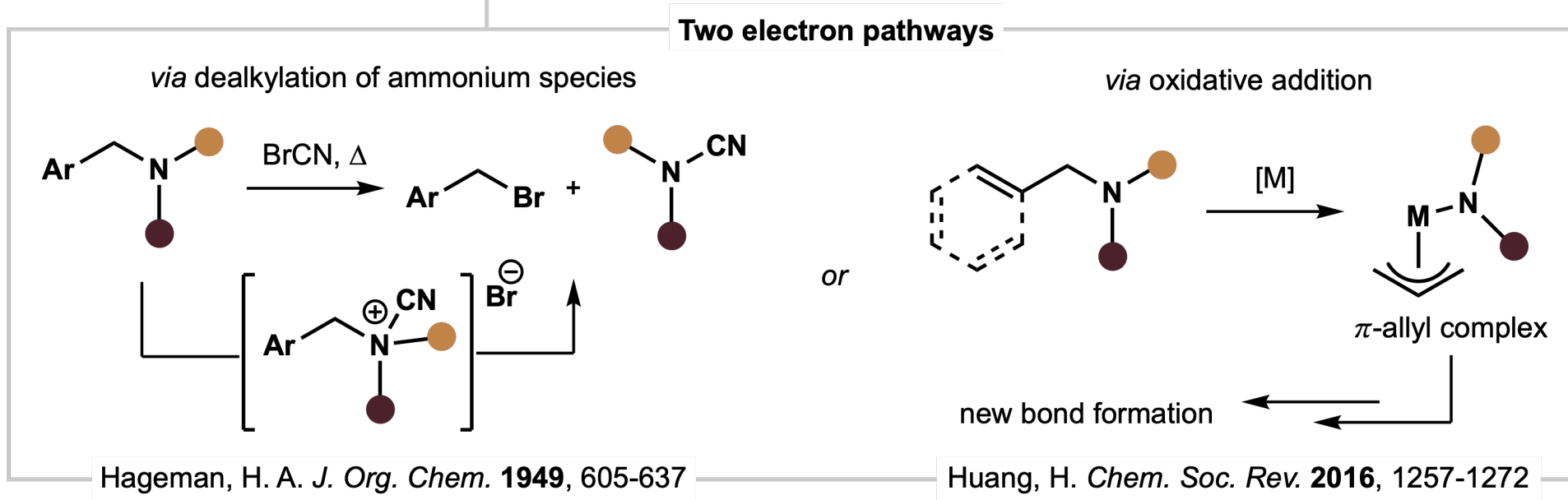
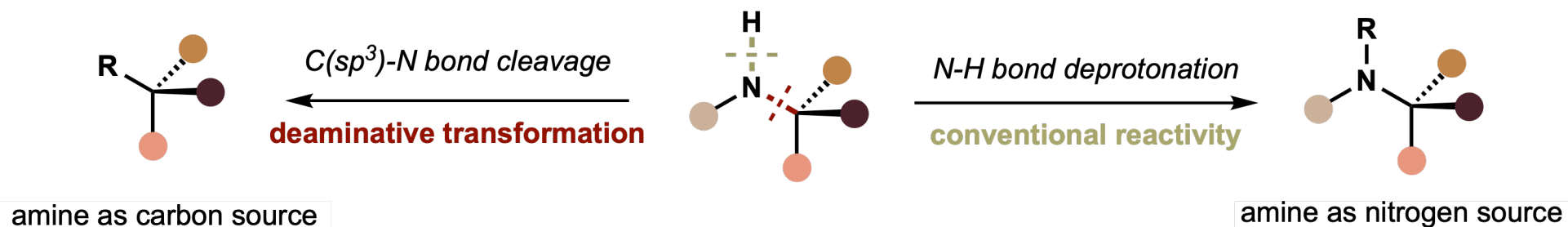
Amine As A Start For Functionalization



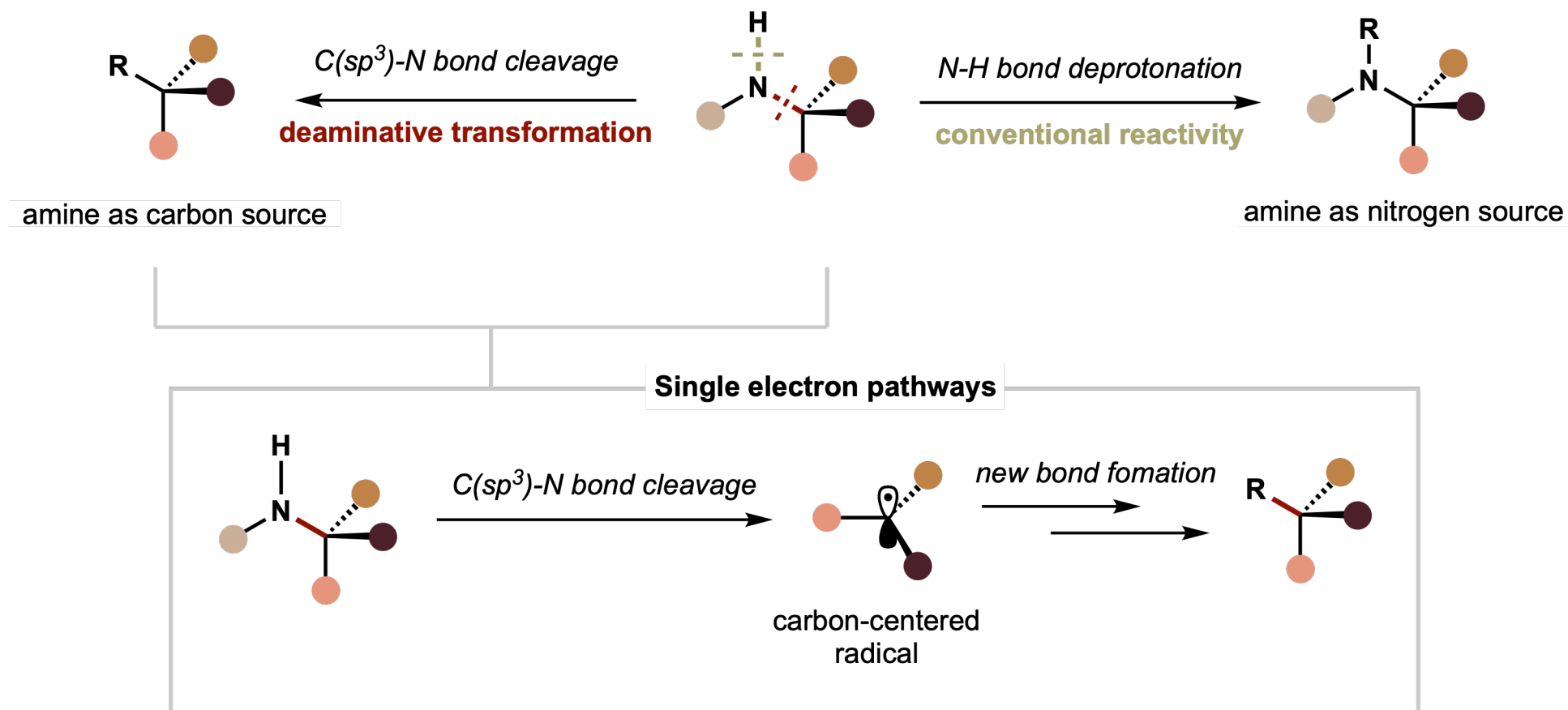
Amine As A Start For Functionalization



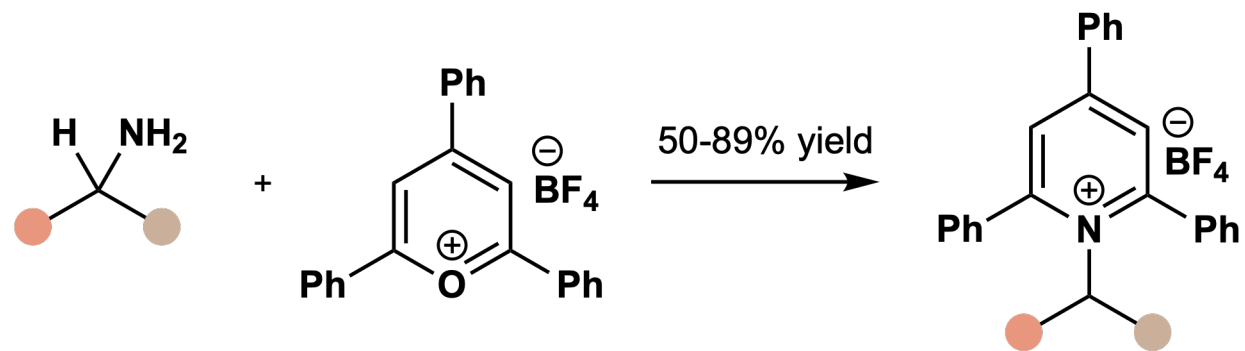
Amine As A Start For Functionalization



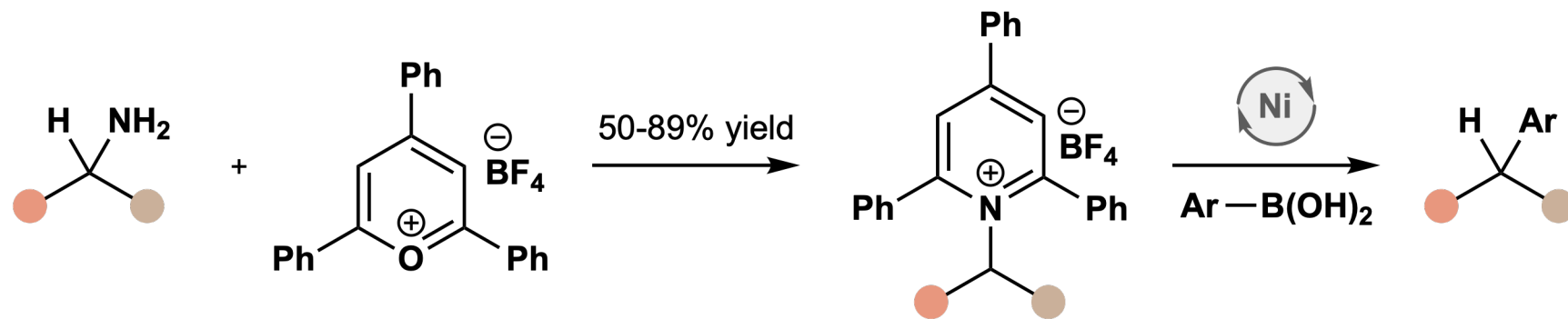
Amine As A Start For Functionalization



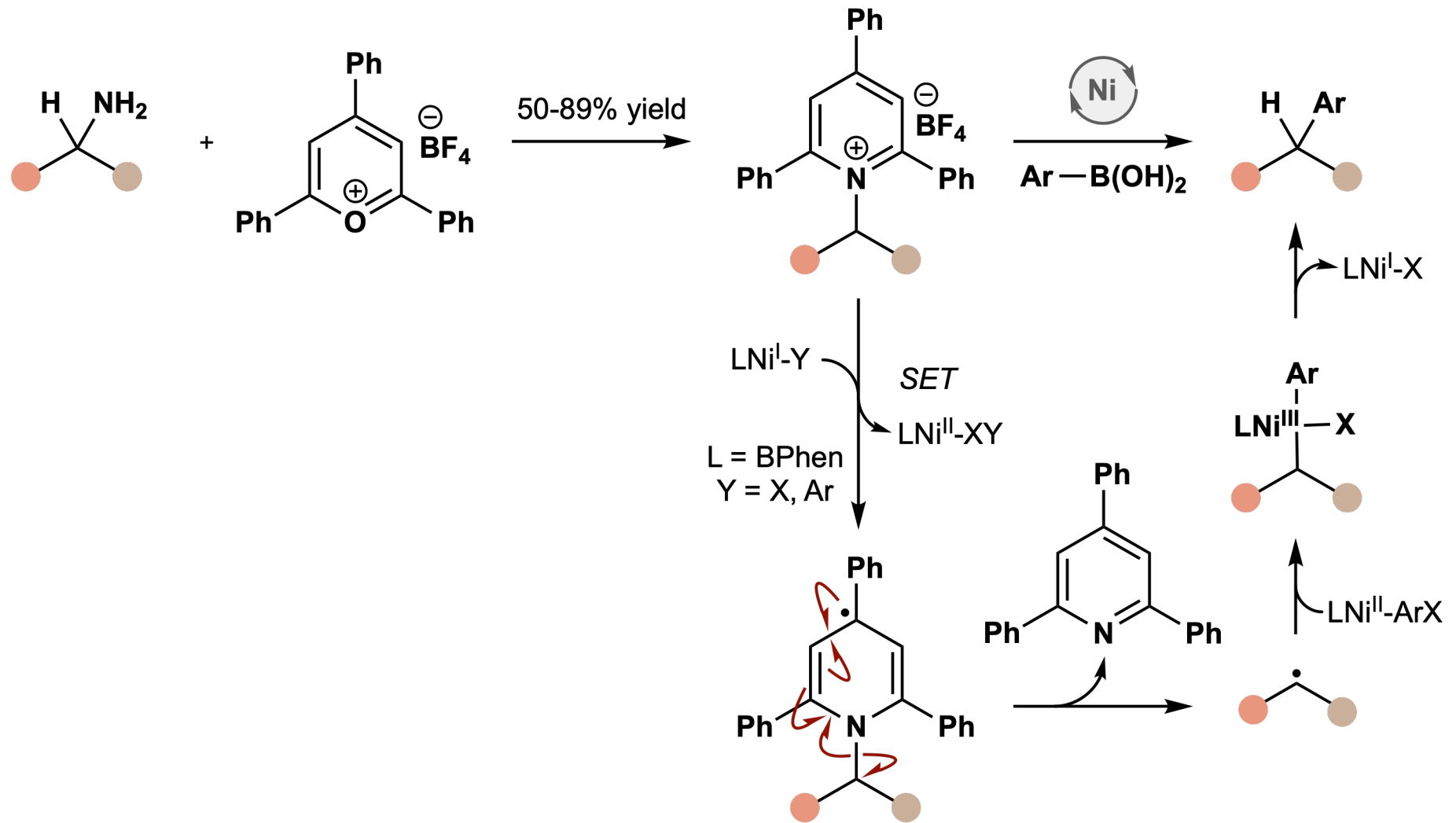
Radical Approaches Towards C-N Bond Cleavage



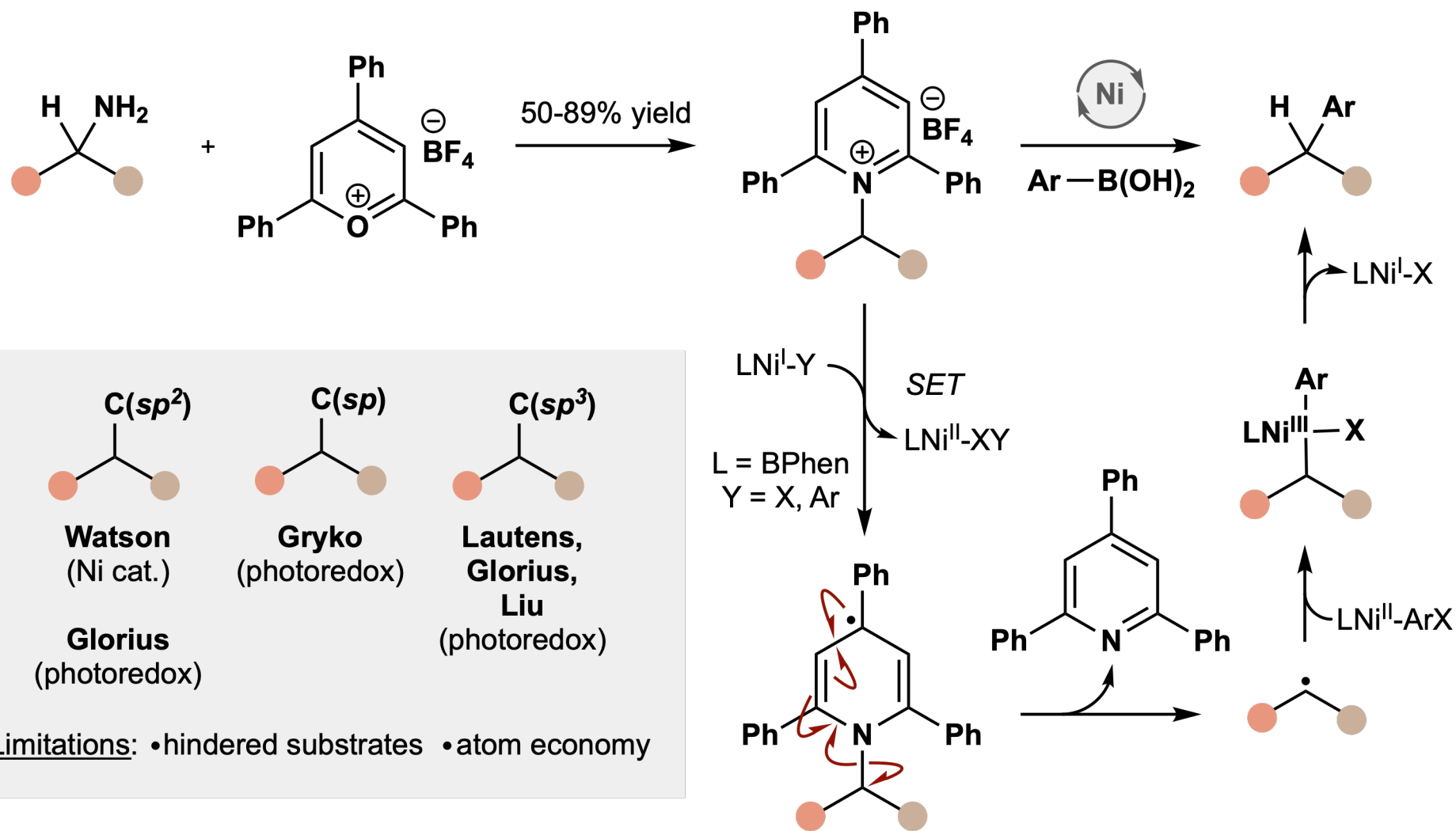
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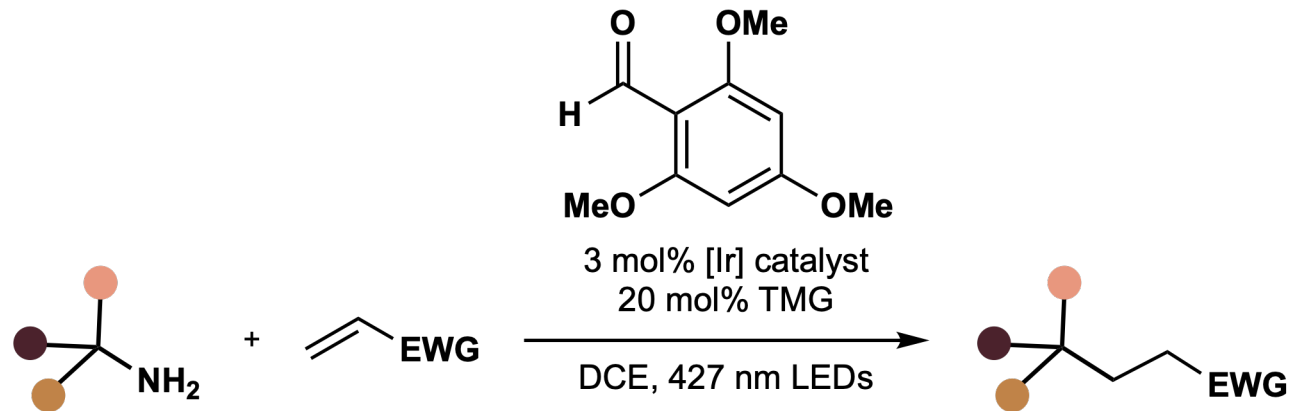
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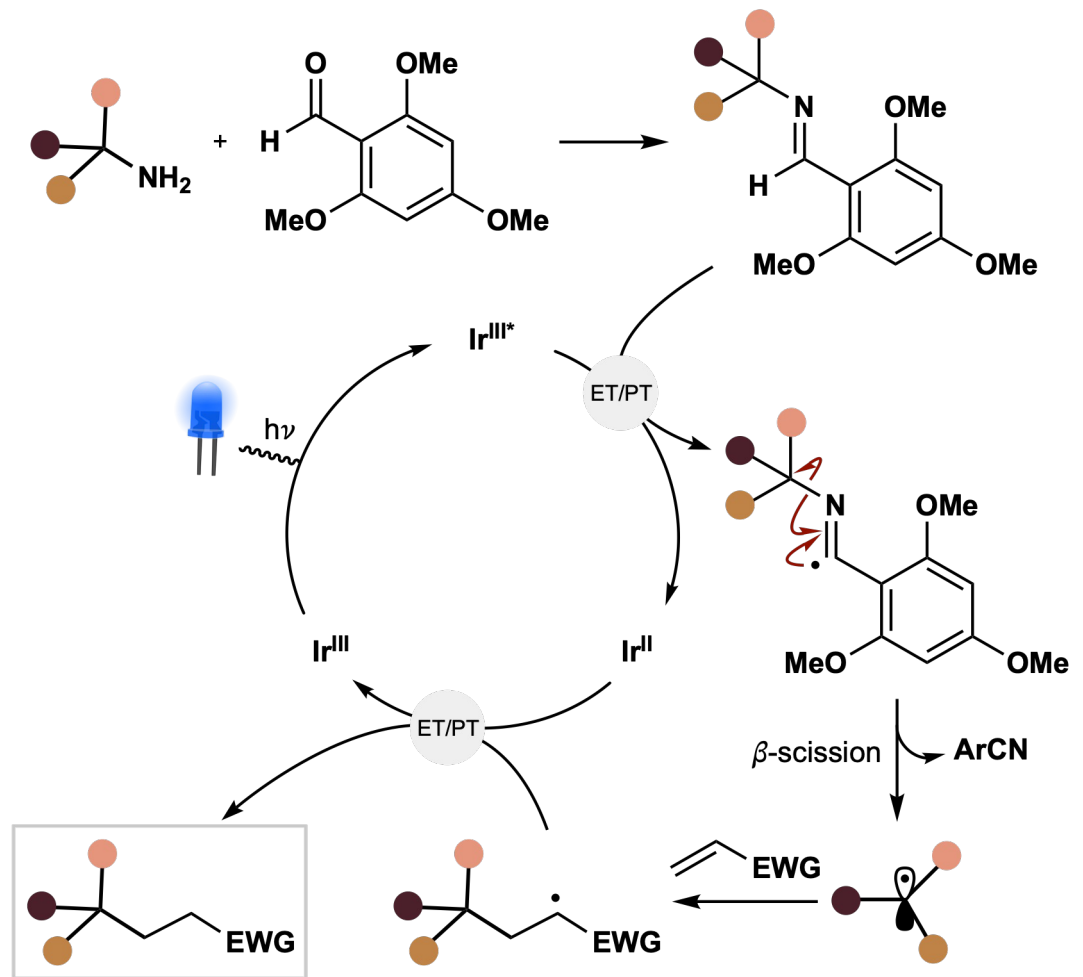
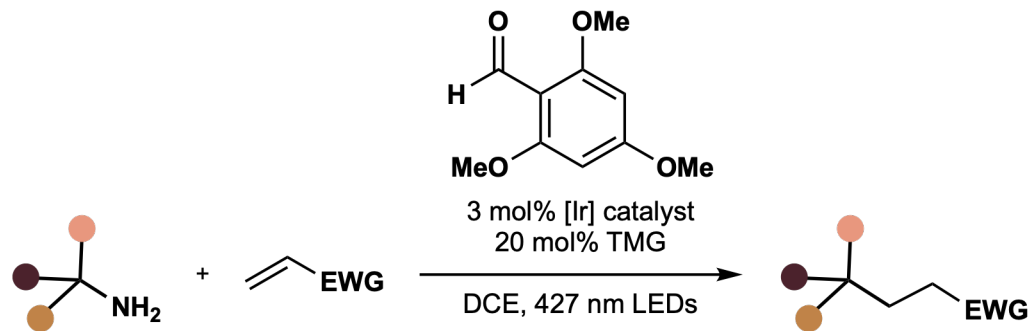
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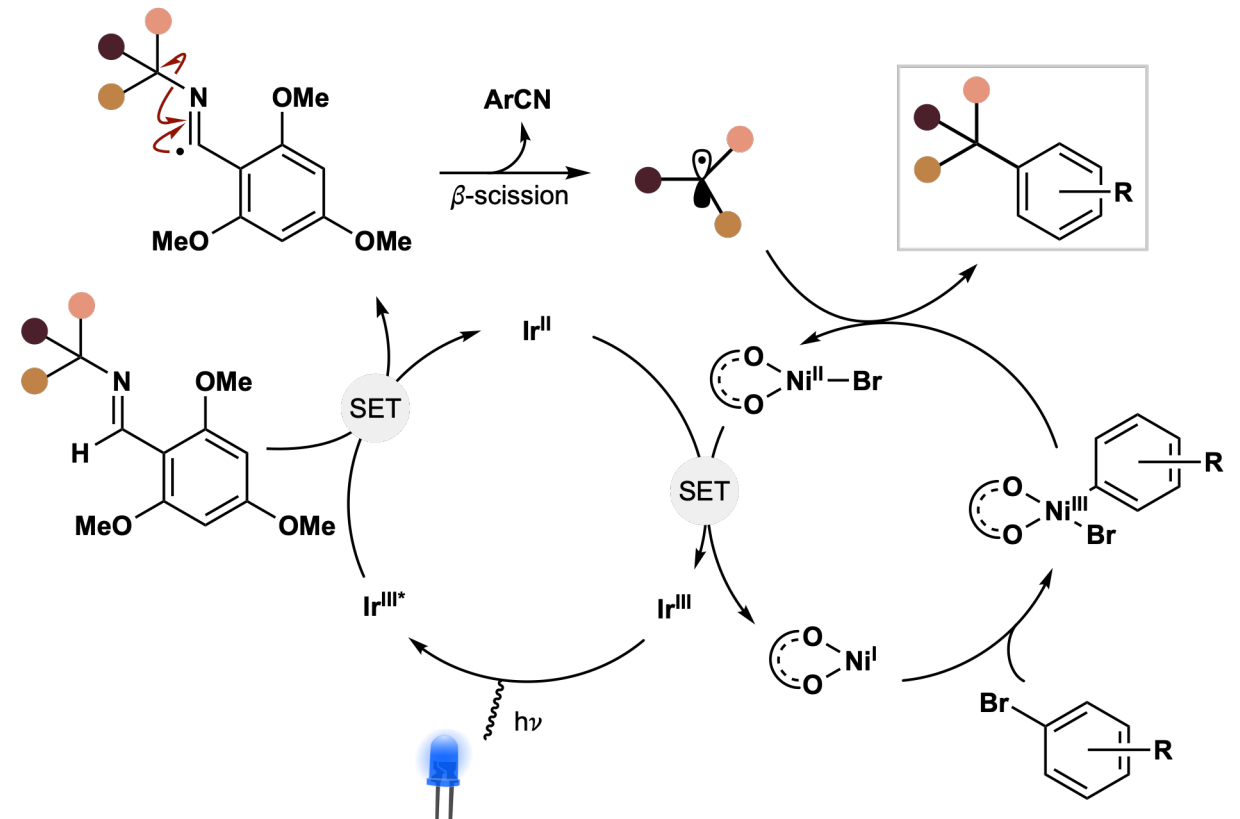
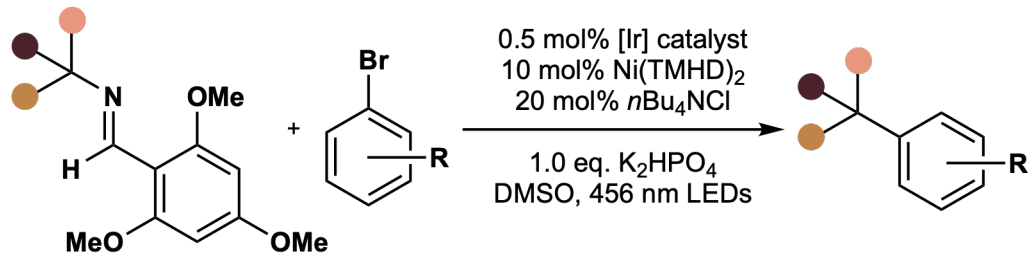
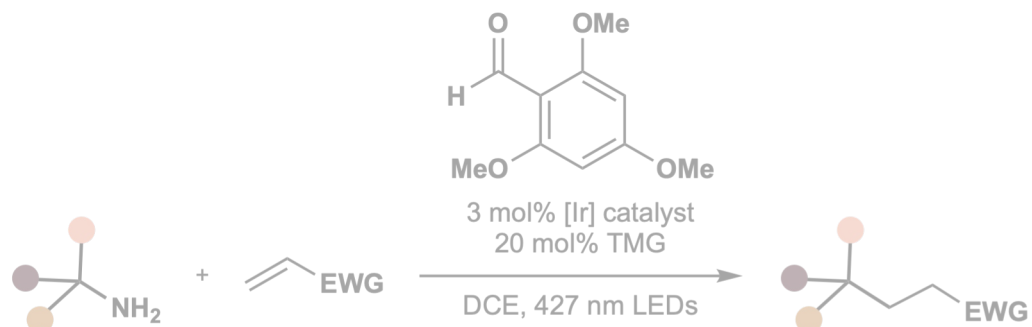
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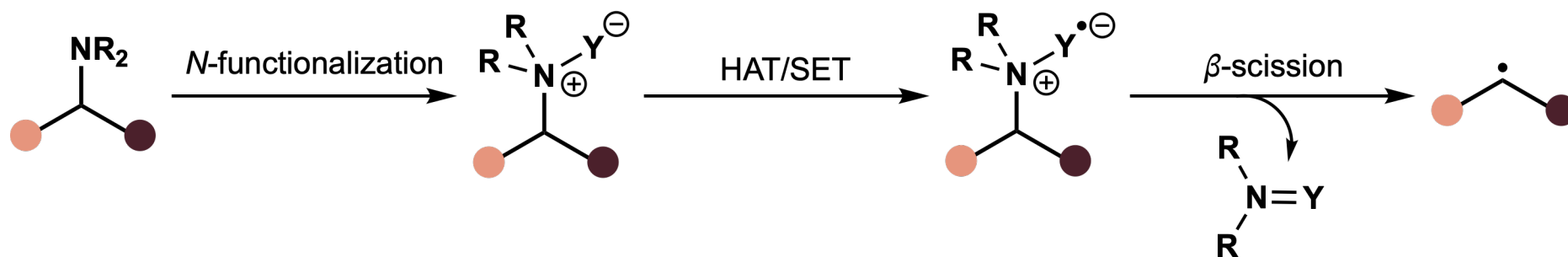
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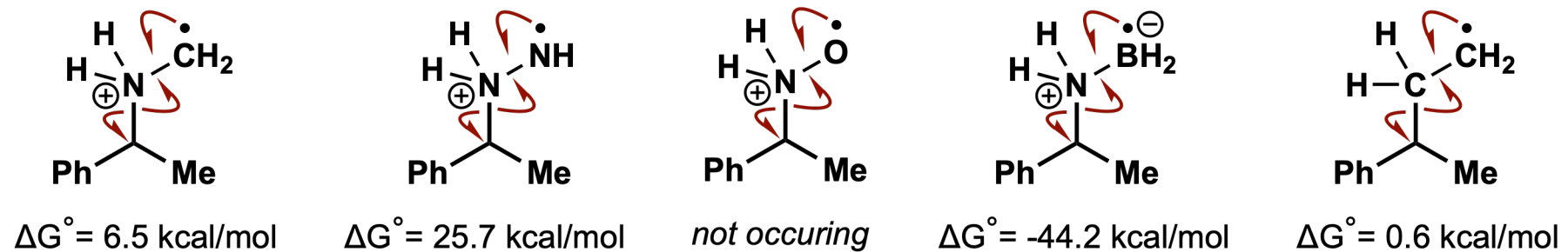
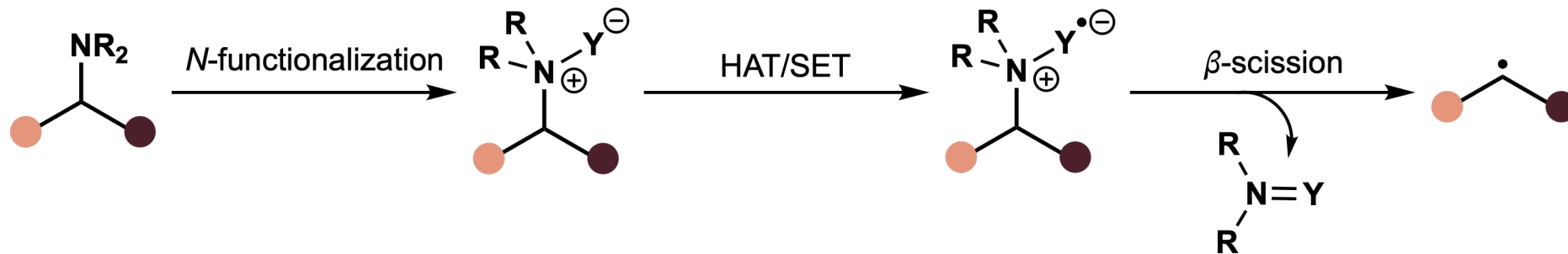
Radical Approaches Towards C-N Bond Cleavage



Leonori's Strategy

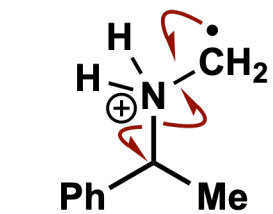
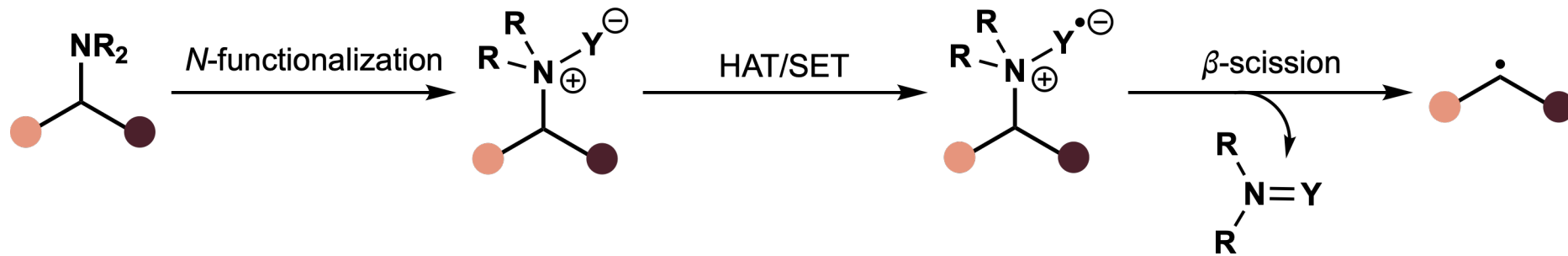


Leonori's Strategy

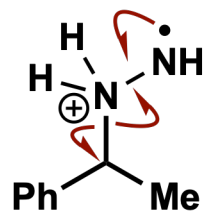


Level of theory: $\omega\text{B97X-D3/def2-TZVP//}\omega\text{B97X-D3/def2-SVP}$

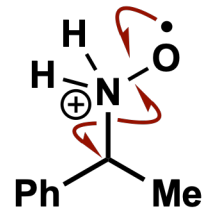
Leonori's Strategy



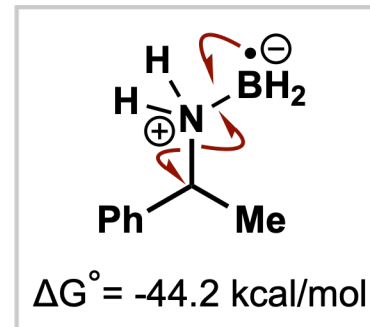
$$\Delta G^\circ = 6.5 \text{ kcal/mol}$$



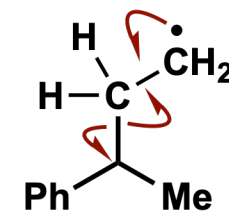
$$\Delta G^\circ = 25.7 \text{ kcal/mol}$$



not occurring



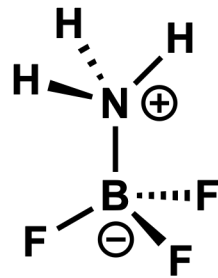
$$\Delta G^\circ = -44.2 \text{ kcal/mol}$$



$$\Delta G^\circ = 0.6 \text{ kcal/mol}$$

Level of theory: $\omega\text{B97X-D3/def2-TZVP//}\omega\text{B97X-D3/def2-SVP}$

History of B-N Containing Compounds

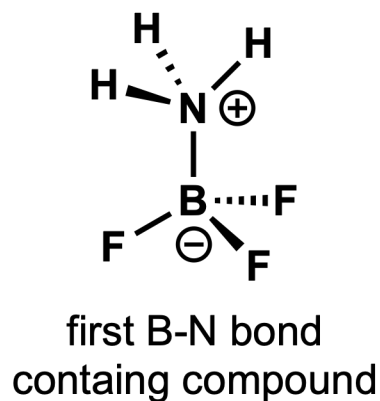


first B-N bond
containing compound

Gay-Lussac, J. L.

*Mem. Phys. Chim. Soc.
D'Arcueil* **1809**, 2, 211.

History of B-N Containing Compounds

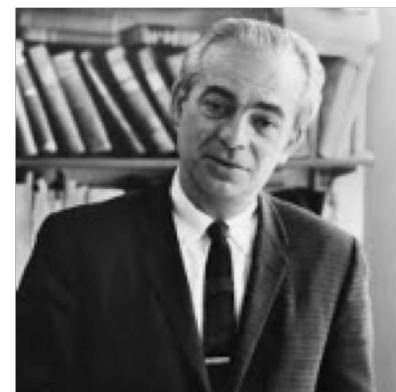


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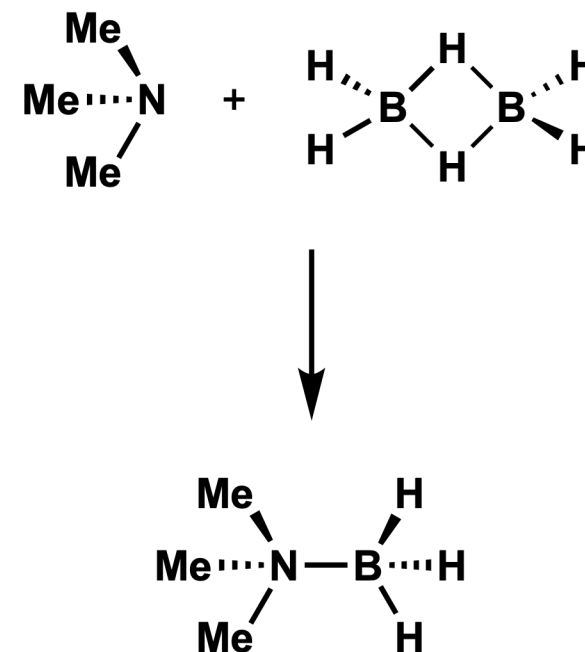
*Mem. Phys. Chim. Soc.
D'Arcueil* **1809**, 2, 211.



Burg, A. B.

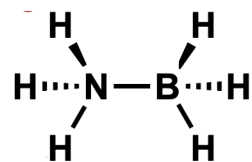


Schlesinger, H. I.

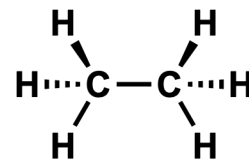


J. Am. Chem. Soc. **1937**, 59, 780.

History of B-N Containing Compounds

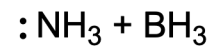
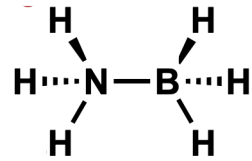


vs

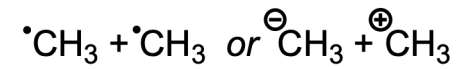
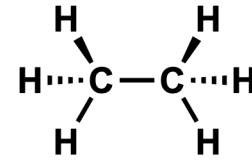


History of B-N Containing Compounds

Thermal fragmentation



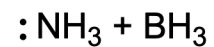
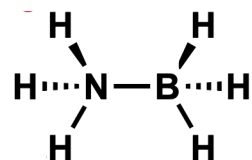
vs



History of B-N Containing Compounds

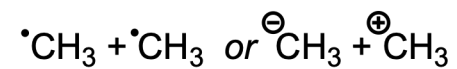
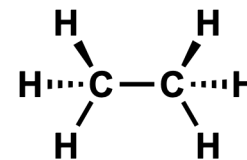
Thermal fragmentation

Central bond dissociation H



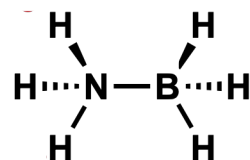
27.2 kcal/mol

vs

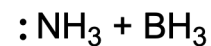


90.1 kcal/mol

History of B-N Containing Compounds



Thermal fragmentation



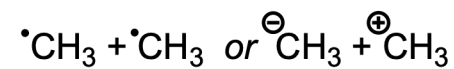
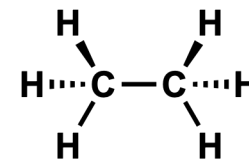
Central bond dissociation H

27.2 kcal/mol

Pauling electronegativity

Physical state

vs



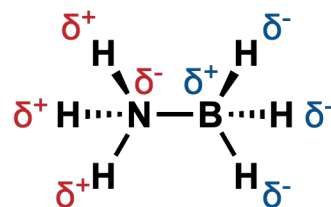
90.1 kcal/mol

C (2.5); H (2.2)

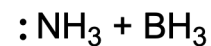


ethane gas cylinders

History of B-N Containing Compounds



Thermal fragmentation



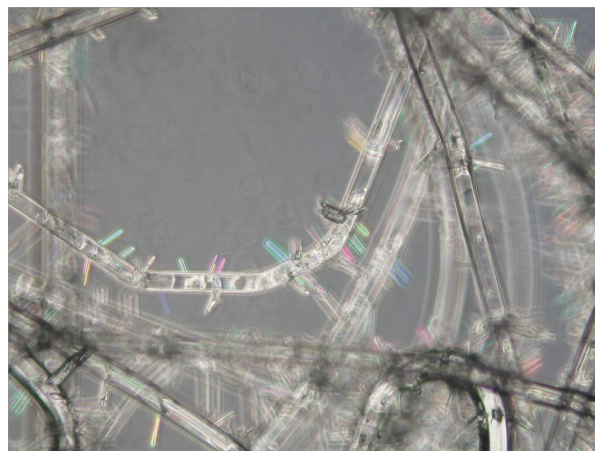
Central bond dissociation H

27.2 kcal/mol

Pauling electronegativity

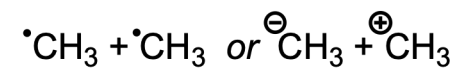
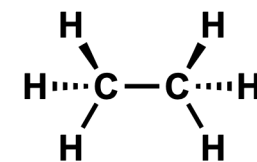
N (3.0); H (2.2); B (2.0)

Physical state



ammonia-borane crystals

vs



90.1 kcal/mol

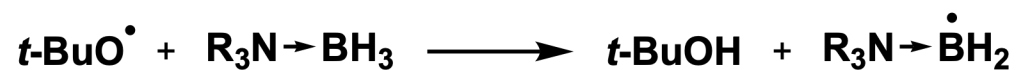
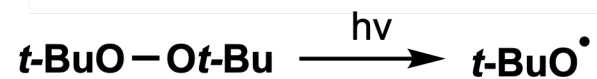
C (2.5); H (2.2)



ethane gas cylinders

General Reactivity of Amine-Borane Complexes

Generation of amine-boryl radicals



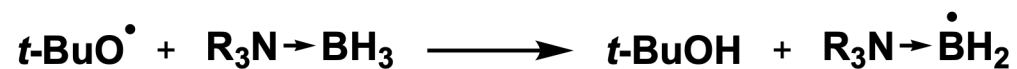
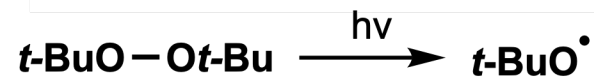
General Reactivity of Amine-Borane Complexes

Self-reaction



at low temperature (190 K)

Generation of amine-boryl radicals



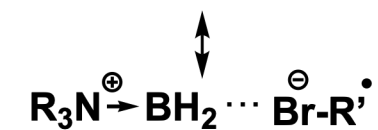
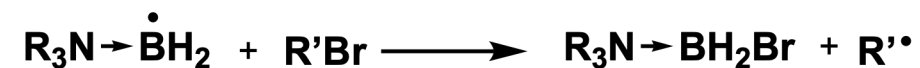
General Reactivity of Amine-Borane Complexes

Self-reaction

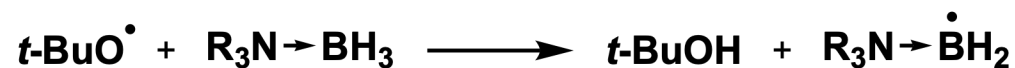


at low temperature (190 K)

Reaction with halogens



Generation of amine-boranyl radicals



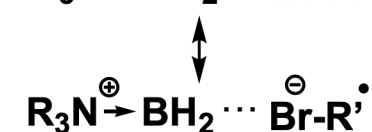
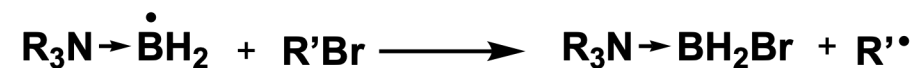
General Reactivity of Amine-Borane Complexes

Self-reaction

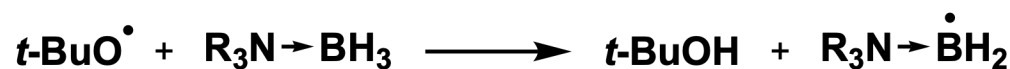


at low temperature (190 K)

Reaction with halogens



Generation of amine-boryl radicals



β -Scission

Amine-boryl radical generated



Temperature (K)

200 K

200 K

up to ca. 298 K

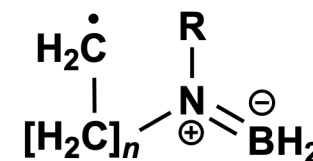
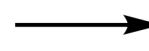
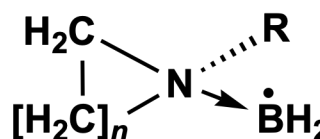
Radicals detected

$i\text{-Pr}\cdot$

$t\text{-Bu}\cdot$

no trace of $t\text{-Bu}\cdot$

Cyclic amine boryl-radical:



General Reactivity of Amine-Borane Complexes

Hypothesis: amine-boryl β -scission it's more exothermic than the C-C case, that is why it is faster.

General Reactivity of Amine-Borane Complexes

Hypothesis: amine-boryl β -scission it's more exothermic than the C-C case, that is why it is faster.

Let's calculate that! Example reaction: $\text{Me}_3\text{X}-\dot{\text{Y}}\text{H}_2 \longrightarrow \text{Me}^\bullet + \text{Me}_2\text{X}=\text{YH}_2$

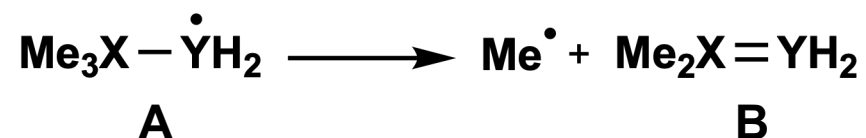
Calculate how much more exothermic this reaction is when you have X=N; Y=B compared to X=Y=C

What you know:

1. enthalpy of $\text{H}_2\overset{\oplus}{\text{N}}=\overset{\ominus}{\text{BH}}_2 + \text{H}_2 \longrightarrow \text{H}_3\text{N}-\text{BH}_3$ is + 18.7 kJ/mol
2. enthalpy of atomization $\Delta H^\ominus(\text{Me}^\bullet)$ is 1223 kJ/mol
3. enthalpy of atomization $\Delta H^\ominus(\text{H}_2)$ is 436 kJ/mol
4. bond energy terms from the literature: E(C-H) 414, E(B-H) 389, E(N-H) 391,
E(C-C) 348, E(C-N) 303, and E(C=C) 604 kJ/mol

General Reactivity of Amine-Borane Complexes

The enthalpies of atomization may be expressed as sums of bond energy terms E and, assuming that the same values of $E(\text{C-H})$, $E(\text{C-X})$, and $E(\text{Y-H})$ apply all species, you can derive:



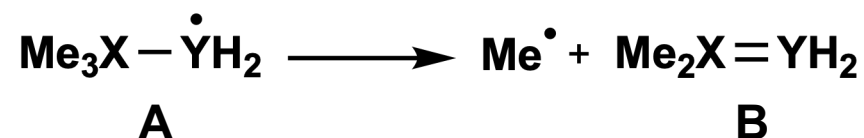
$$\Delta H_{rxn}^{\theta} = \sum (\Delta H_{SM}^{\theta} - \Delta H_{PRDT}^{\theta}) = \Delta H^{\theta}(A) - \Delta H^{\theta}(B) - \Delta H^{\theta}(\text{Me}\cdot)$$

$$[\Delta H^{\theta}(A) - \Delta H^{\theta}(B)] =$$

Bond	Species A	Species B	
C-H	9	6	3 more in SM
X-C	3	2	1 more in SM
Y-H	2	2	Terms cancel out
X-Y	one single	one double	Different terms

General Reactivity of Amine-Borane Complexes

The enthalpies of atomization may be expressed as sums of bond energy terms E and, assuming that the same values of $E(\text{C-H})$, $E(\text{C-X})$, and $E(\text{Y-H})$ apply all species, you can derive:



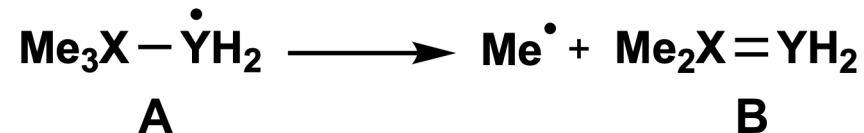
$$\Delta H_{rxn}^{\theta} = \sum (\Delta H_{SM}^{\theta} - \Delta H_{PRDT}^{\theta}) = \Delta H^{\theta}(A) - \Delta H^{\theta}(B) - \Delta H^{\theta}(\text{Me}\cdot)$$

$$[\Delta H^{\theta}(A) - \Delta H^{\theta}(B)] = 3E(\text{C-H}) + E(\text{C-X}) - [E(\text{X=Y}) - E(\text{X-Y})]$$

Bond	Species A	Species B	
C-H	9	6	3 more in SM
X-C	3	2	1 more in SM
Y-H	2	2	Terms cancel out
X-Y	one single	one double	Different terms

$$\Delta H_{rxn}^{\theta} = 3E(\text{C-H}) + E(\text{C-X}) - [E(\text{X=Y}) - E(\text{X-Y})] - \Delta H^{\theta}(\text{Me}\cdot)$$

General Reactivity of Amine-Borane Complexes



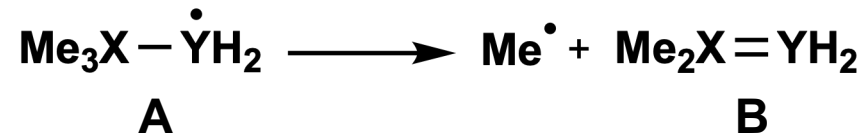
$$\Delta H_{\text{rxn}}^\theta = 3E(\text{C}-\text{H}) + E(\text{C}-\text{X}) - [E(\text{X}=\text{Y}) - E(\text{X}-\text{Y})] - \Delta H^\theta(\text{Me}^\bullet)$$

So, when X=Y=C:

E(C-H) 414, E(C-C) 348, and E(C=C) 604 kJ/mol; $\Delta H^\theta(\text{Me}^\bullet)$ is 1223 kJ/mol

$$\begin{aligned}\Delta H_{\text{rxn}}^\theta &= 3E(\text{C}-\text{H}) + E(\text{C}-\text{C}) - [E(\text{C}=\text{C}) - E(\text{C}-\text{C})] - \Delta H^\theta(\text{Me}^\bullet) \\ &= [(3 \times 414) + 348 - (604 - 348) - 1223] \text{ kJ/mol} = \mathbf{+111 \text{ kJ/mol}}\end{aligned}$$

General Reactivity of Amine-Borane Complexes



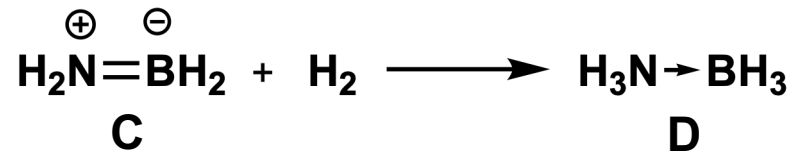
$$\Delta H_{rxn}^\theta = 3E(\text{C}-\text{H}) + E(\text{C}-\text{X}) - [E(\text{X}=\text{Y}) - E(\text{X}-\text{Y})] - \Delta H^\theta(\text{Me}^\bullet)$$

So, when X=Y=C:

E(C-H) 414, E(C-C) 348, and E(C=C) 604 kJ/mol; $\Delta H^\theta(\text{Me}^\bullet)$ is 1223 kJ/mol

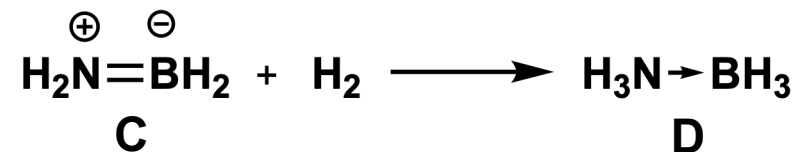
$$\begin{aligned} \Delta H_{rxn}^\theta &= 3E(\text{C}-\text{H}) + E(\text{C}-\text{C}) - [E(\text{C}=\text{C}) - E(\text{C}-\text{C})] - \Delta H^\theta(\text{Me}^\bullet) \\ &= [(3 \times 414) + 348 - (604 - 348) - 1223] \text{ kJ/mol} = \mathbf{+111 \text{ kJ/mol}} \end{aligned}$$

For X=N; Y=B, some of the energy terms are missing, so you derive them from:



$$\Delta H_{rxn}^\theta = +18.7 \text{ kJ/mol} = \Delta H^\theta(\text{C}) - \Delta H^\theta(\text{D}) - \Delta H^\theta(\text{H}_2)$$

General Reactivity of Amine-Borane Complexes

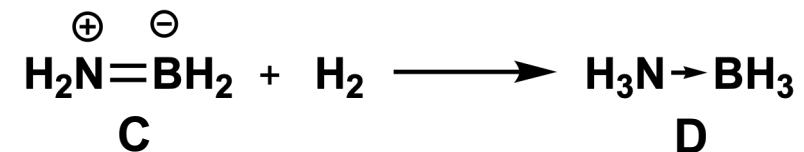


$$\Delta H_{rxn}^\theta = +18.7 \text{ kJ/mol} = \Delta H^\theta(C) - \Delta H^\theta(D) + \Delta H^\theta(\text{H}_2)$$

$$[\Delta H^\theta(C) - \Delta H^\theta(D)] =$$

Bond	Species C	Species D	
N-H	2	3	1 less in SM
N-B	one double	one single	Different terms
B-H	2	3	1 less in SM

General Reactivity of Amine-Borane Complexes

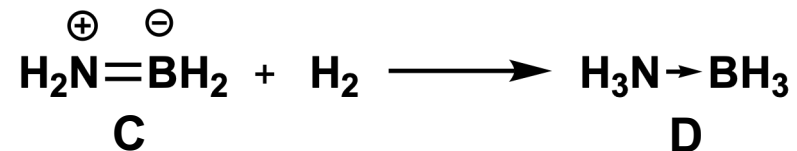


$$\Delta H_{rxn}^{\theta} = +18.7 \text{ kJ/mol} = \Delta H^{\theta}(C) - \Delta H^{\theta}(D) + \Delta H^{\theta}(\text{H}_2)$$

$$[\Delta H^{\theta}(C) - \Delta H^{\theta}(D)] = -E(N-H) - [E(N-B) - E(N=B)] - E(B-H)$$

Bond	Species C	Species D	
N-H	2	3	1 less in SM
N-B	one double	one single	Different terms
B-H	2	3	1 less in SM

General Reactivity of Amine-Borane Complexes



$$\Delta H_{rxn}^\theta = +18.7 \text{ kJ/mol} = \Delta H^\theta(C) - \Delta H^\theta(D) + \Delta H^\theta(\text{H}_2)$$

$$[\Delta H^\theta(C) - \Delta H^\theta(D)] = -E(\text{N}-\text{H}) - [E(\text{N}-\text{B}) - E(\text{N}=\text{B})] - E(\text{B}-\text{H})$$

Bond	Species C	Species D	
N-H	2	3	1 less in SM
N-B	one double	one single	Different terms
B-H	2	3	1 less in SM

$$[E(\text{N}=\text{B}) - E(\text{N}-\text{B})] = \Delta H_{rxn}^\theta - \Delta H^\theta(\text{H}_2) + E(\text{N}-\text{H}) + E(\text{B}-\text{H})$$

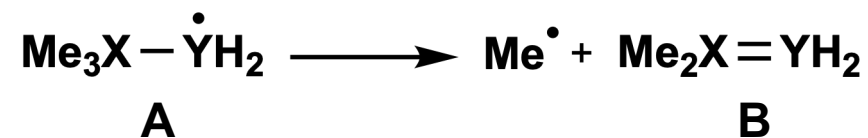
$$E(\text{N}-\text{H}) \text{ 391, } E(\text{B}-\text{H}) \text{ 389, and } \Delta H^\theta(\text{H}_2) \text{ is 436 kJ/mol}$$

$$= (18.7 - 436 + 391 + 389) \text{ kJ/mol} = 363 \text{ kJ/mol}$$

General Reactivity of Amine-Borane Complexes

$$[E(N = B) - E(N - B)] = 363 \text{ kJ/mol}$$

Now for X=N; Y=B in the previous reaction:

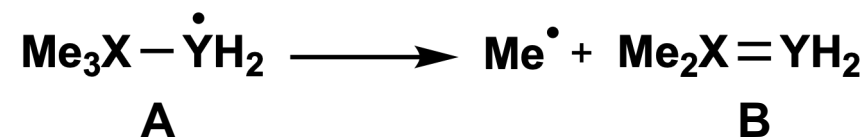


$$\begin{aligned} \Delta H_{rxn}^\theta &= 3E(C - H) + E(C - N) - [E(N = B) - E(N - B)] - \Delta H^\theta(\text{Me}^\bullet) \\ &= [(3 \times 414) + 303 - 363 - 1223] \text{ kJ/mol} = \mathbf{-41 \text{ kJ/mol}} \end{aligned}$$

General Reactivity of Amine-Borane Complexes

$$[E(N = B) - E(N - B)] = 363 \text{ kJ/mol}$$

Now for X=N; Y=B in the previous reaction:



$$\begin{aligned} \Delta H_{rxn}^\theta &= 3E(C - H) + E(C - N) - [E(N = B) - E(N - B)] - \Delta H^\theta(\text{Me}^\bullet) \\ &= [(3 \times 414) + 303 - 363 - 1223] \text{ kJ/mol} = \textbf{-41 kJ/mol} \end{aligned}$$

So, for X=Y=C it was $\Delta H_{rxn}^\theta = +111 \text{ kJ/mol}$ while for X=N; Y=B $\Delta H_{rxn}^\theta = -41 \text{ kJ/mol}$, so the difference is

152 kJ/mol

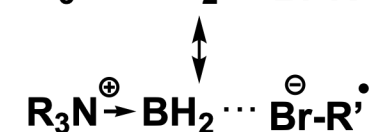
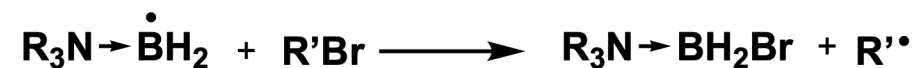
General Reactivity of Amine-Borane Complexes

Self-reaction

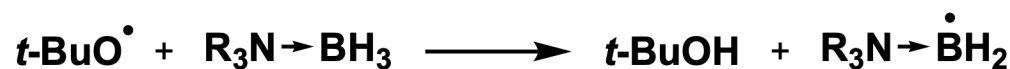


at low temperature (190 K)

Reaction with halogens



Generation of amine-boryl radicals



β -Scission

Amine-boryl radical generated



Temperature (K)

200 K

200 K

up to ca. 298 K

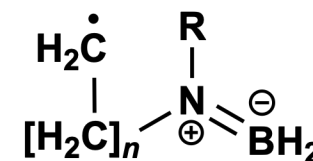
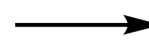
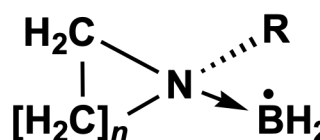
Radicals detected

$i\text{-Pr}^\bullet$

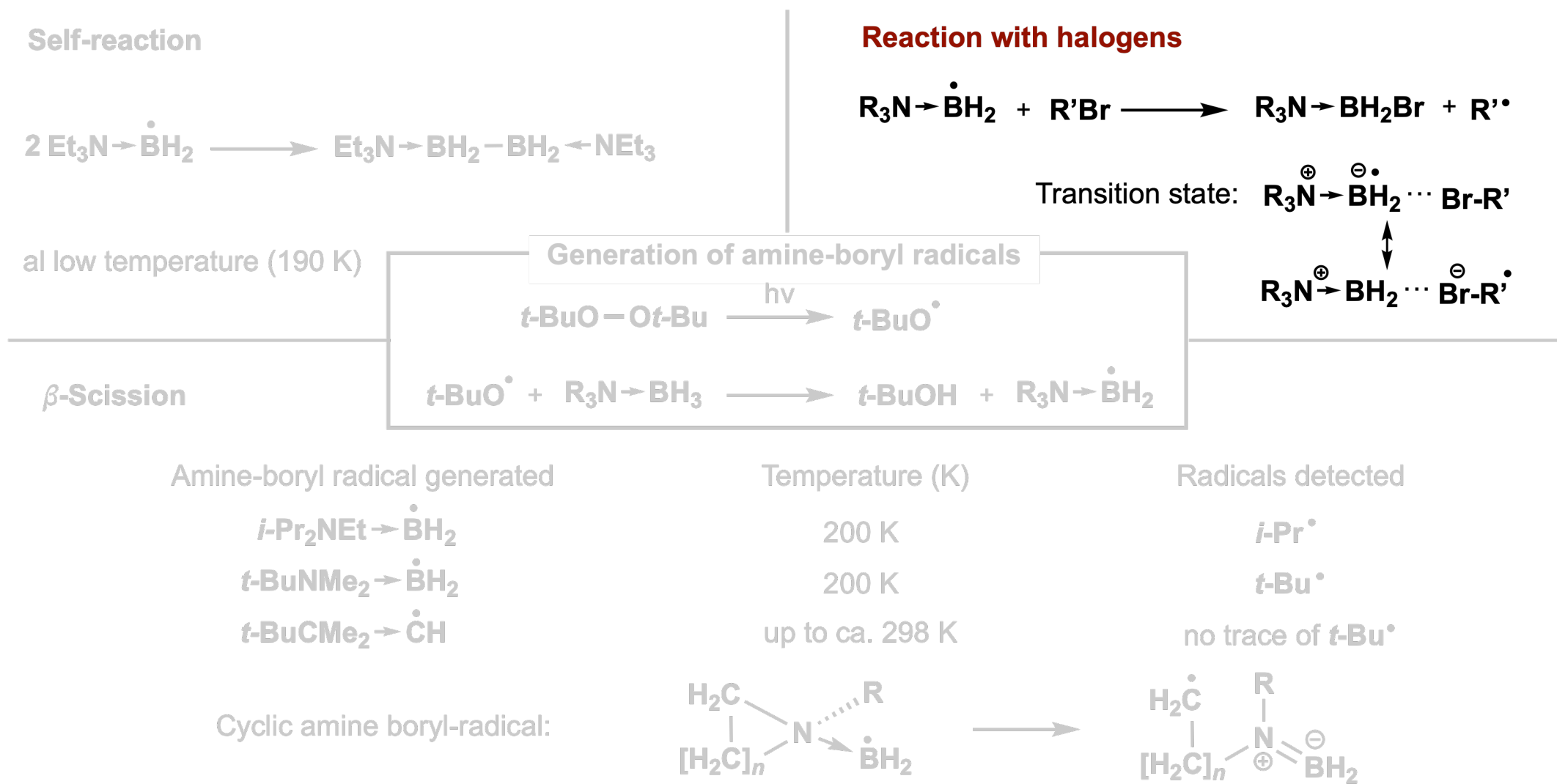
$t\text{-Bu}^\bullet$

no trace of $t\text{-Bu}^\bullet$

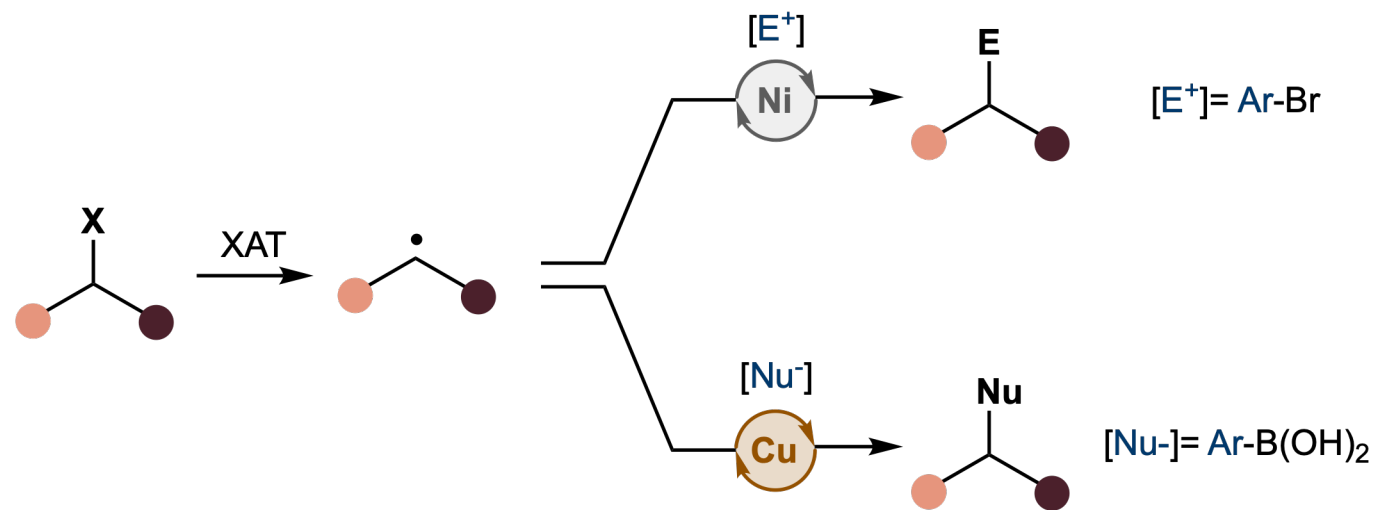
Cyclic amine boryl-radical:



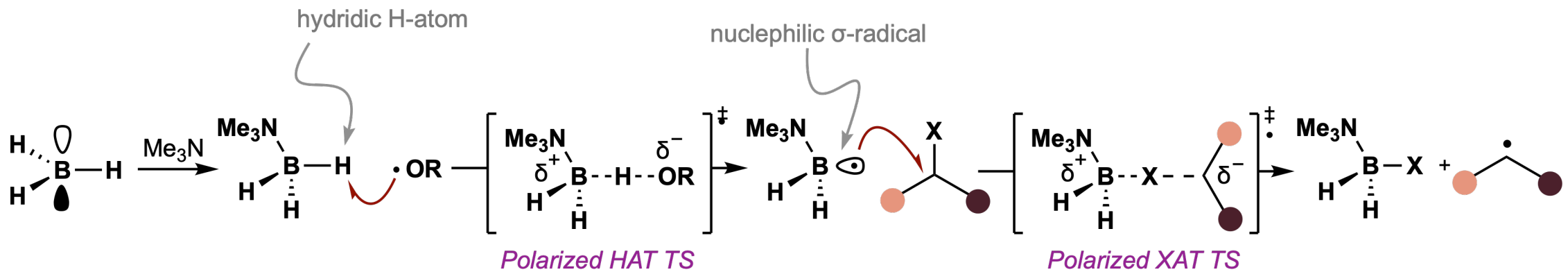
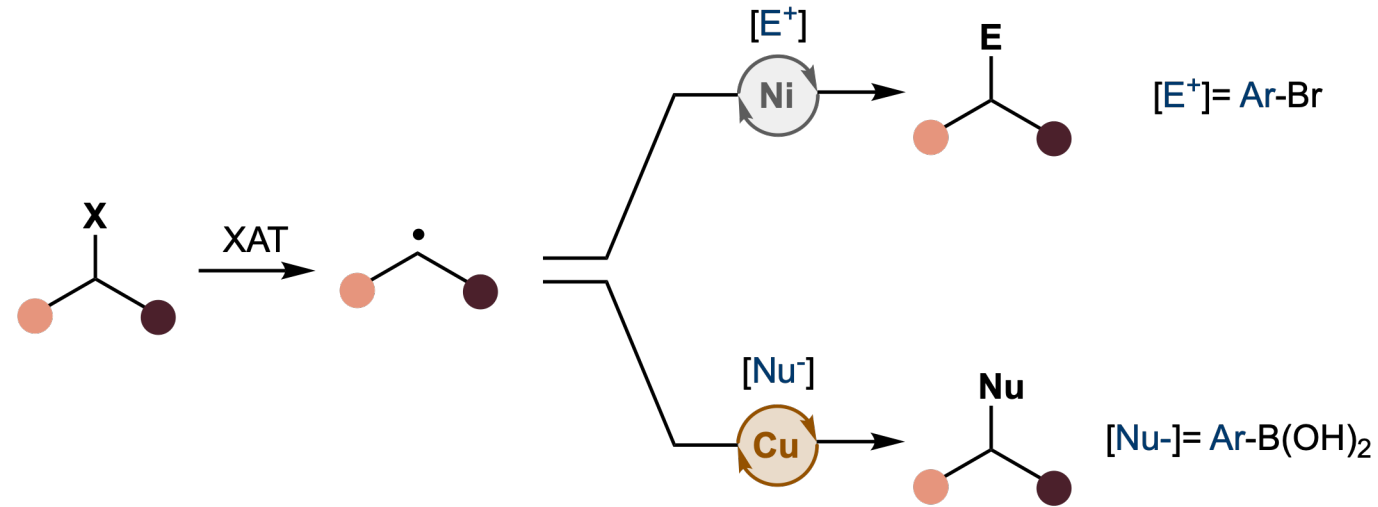
General Reactivity of Amine-Borane Complexes



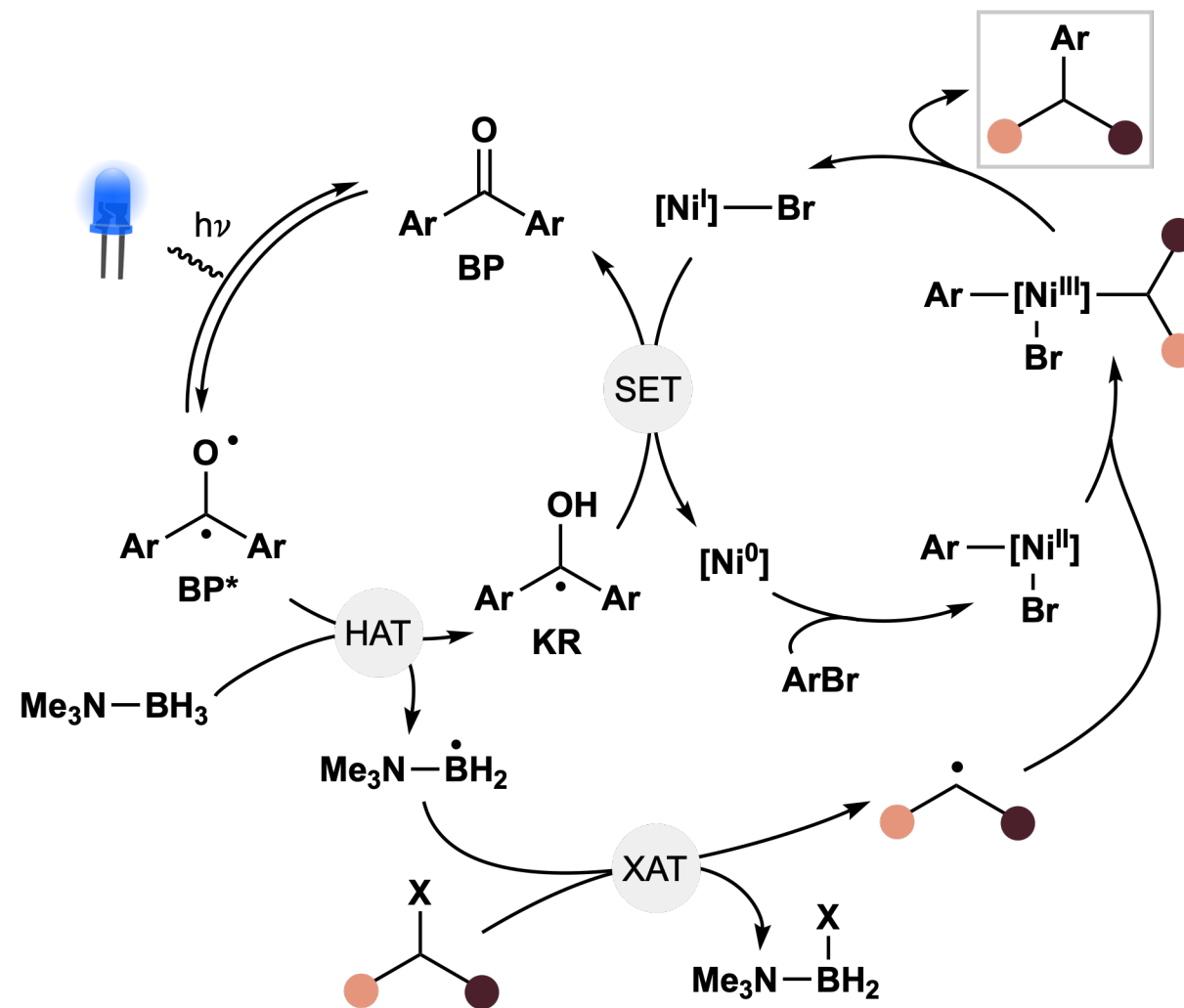
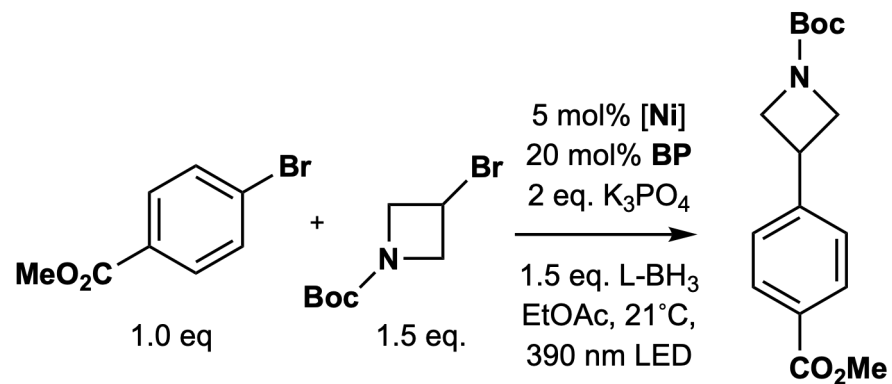
Amine-Boryl Radicals as XAT Reagents



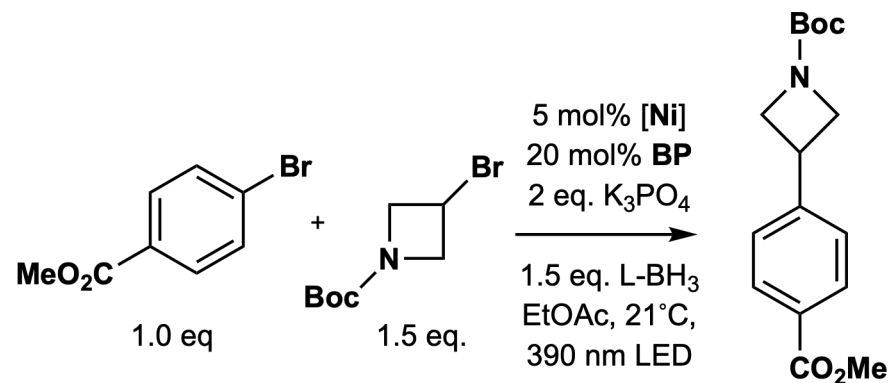
Amine-Boryl Radicals as XAT Reagents



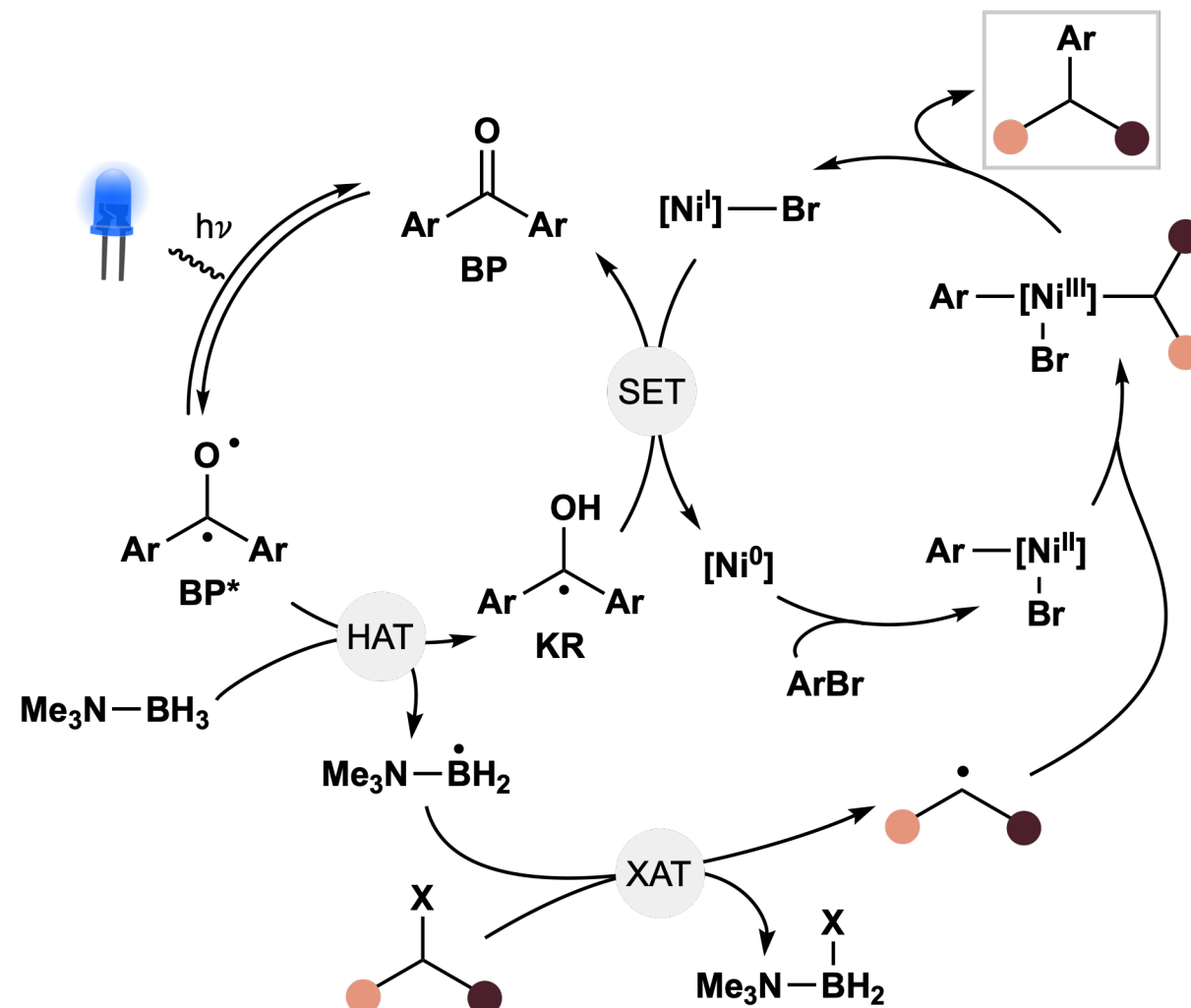
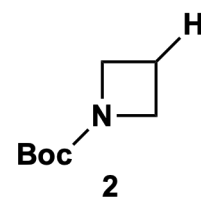
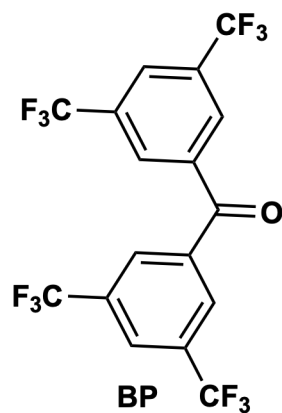
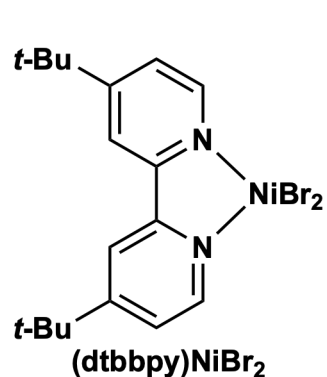
Amine-Boryl Radicals in Cross-Electrophile Coupling



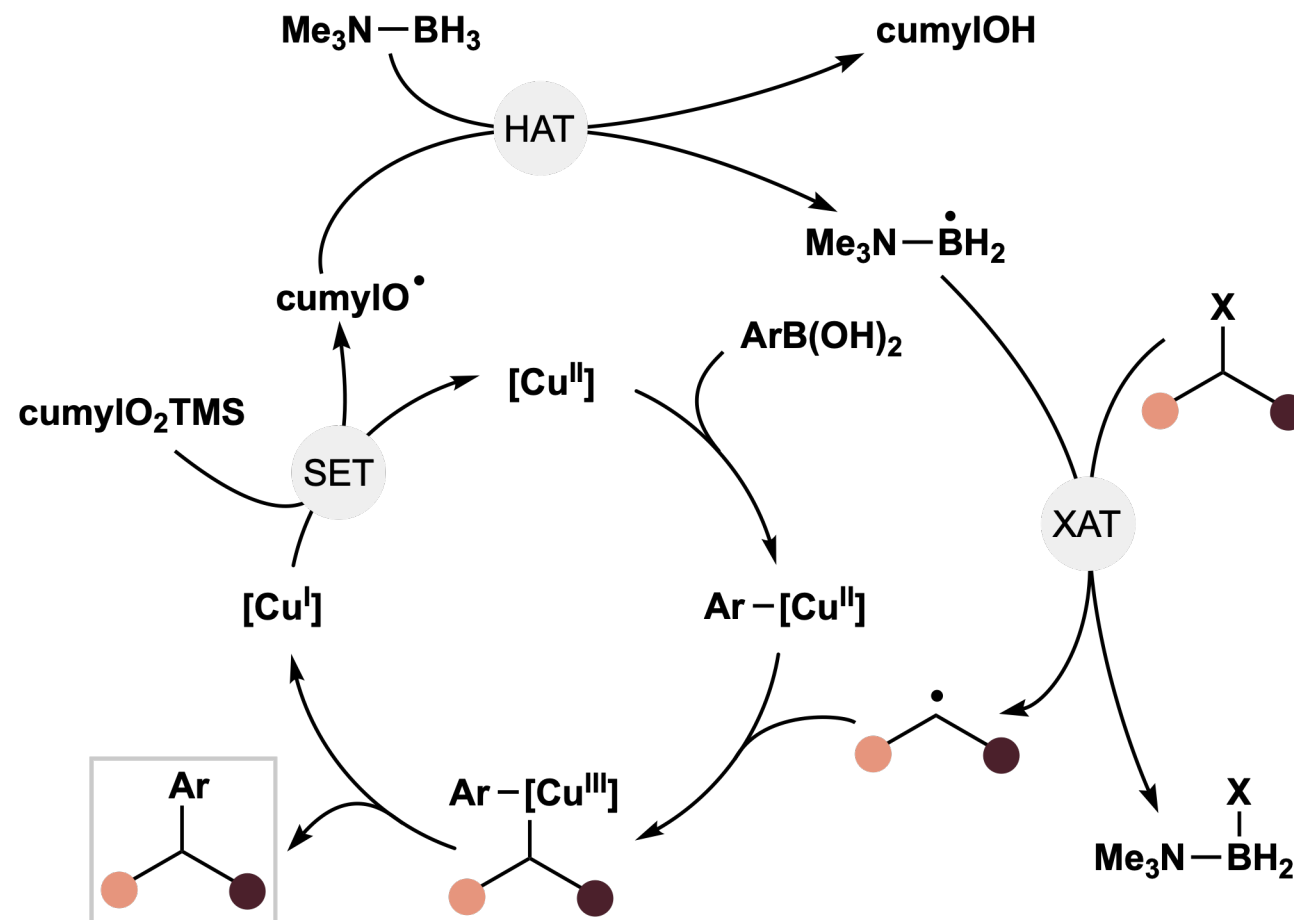
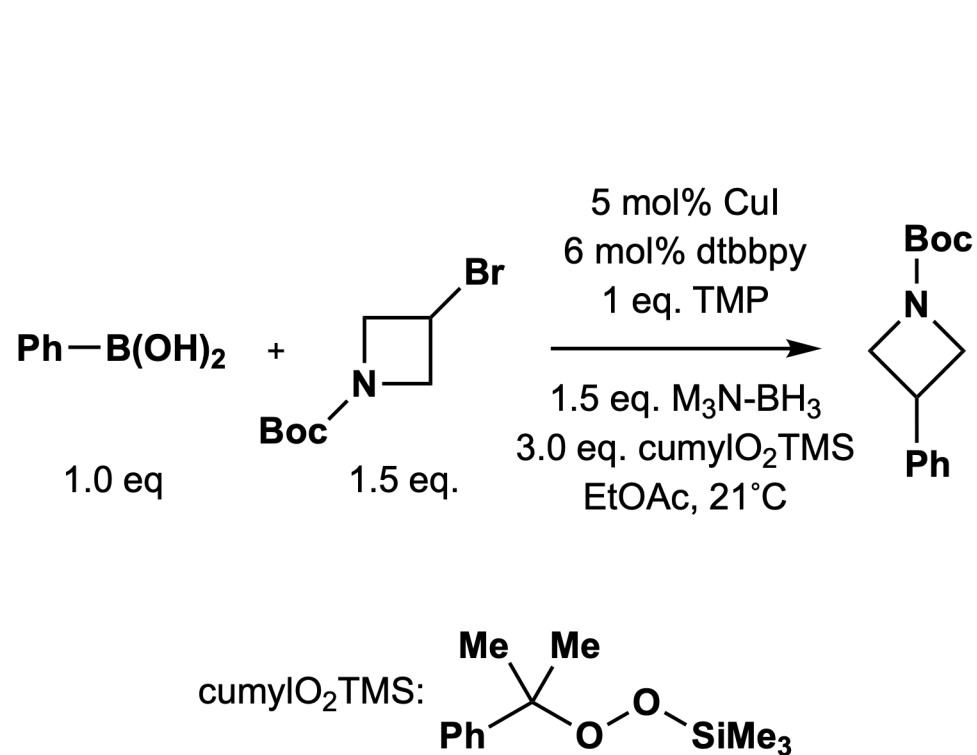
Amine-Boryl Radicals in Cross-Electrophile Coupling



Entry	L-BH ₃	1 (%)	2 (%)
1	Me ₃ N-BH ₃	84	13
2	NHC-BH ₃	27	15
3	Ph ₃ P-BH ₃	75	15
4	H ₃ N-BH ₃	-	51



Amine-Boryl Radicals in Cross Coupling with Nucleophiles



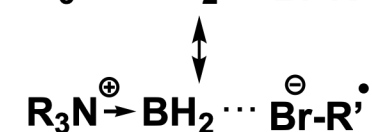
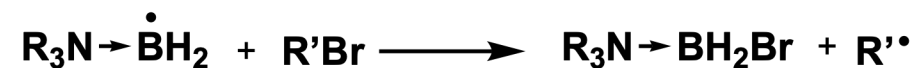
General Reactivity of Amine-Borane Complexes

Self-reaction

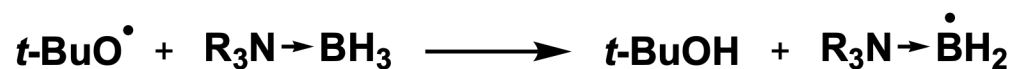


at low temperature (190 K)

Reaction with halogens



Generation of amine-boryl radicals



β -Scission

Amine-boryl radical generated



Temperature (K)

200 K

200 K

up to ca. 298 K

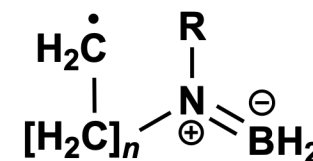
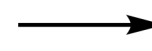
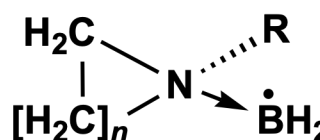
Radicals detected

$i\text{-Pr}^{\cdot}$

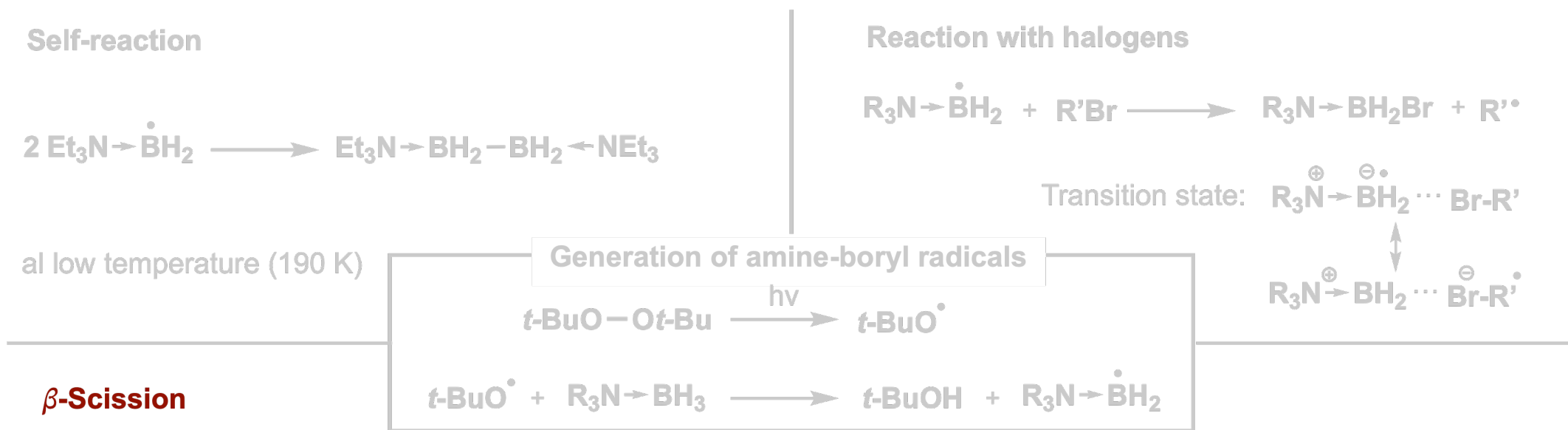
$t\text{-Bu}^{\cdot}$

no trace of $t\text{-Bu}^{\cdot}$

Cyclic amine boryl-radical:



General Reactivity of Amine-Borane Complexes



Amine-boryl radical generated



Temperature (K)

200 K

200 K

up to ca. 298 K

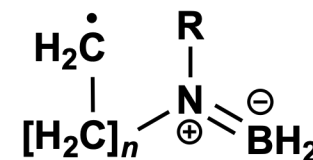
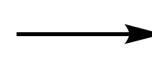
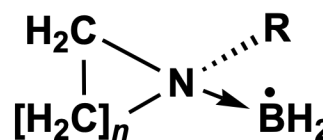
Radicals detected

i-Pr[•]

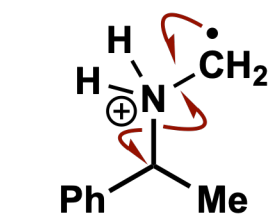
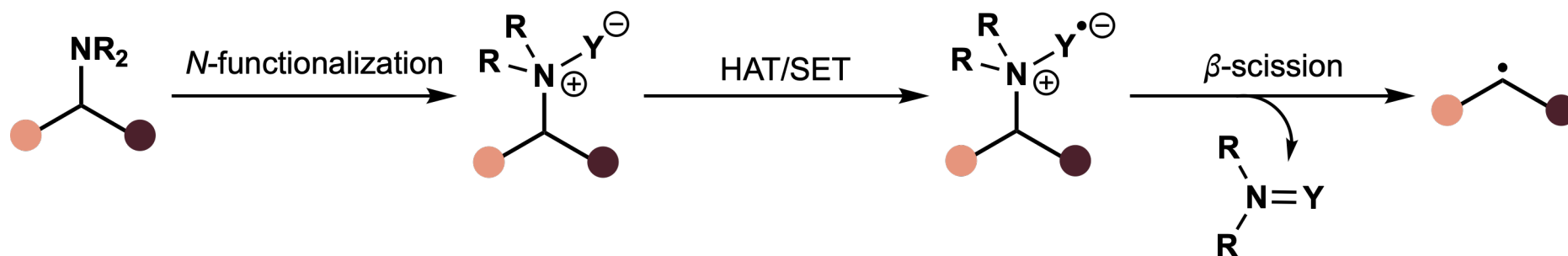
t-Bu[•]

no trace of *t*-Bu[•]

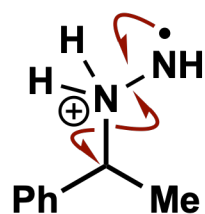
Cyclic amine boryl-radical:



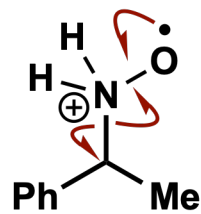
Leonori's Strategy



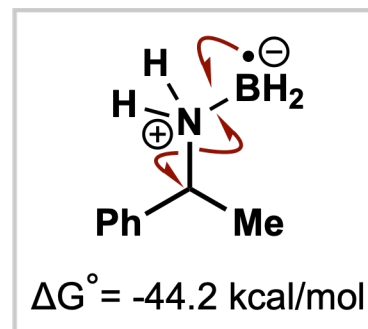
$$\Delta G^\circ = 6.5 \text{ kcal/mol}$$



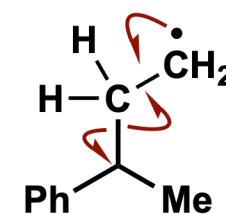
$$\Delta G^\circ = 25.7 \text{ kcal/mol}$$



not occurring



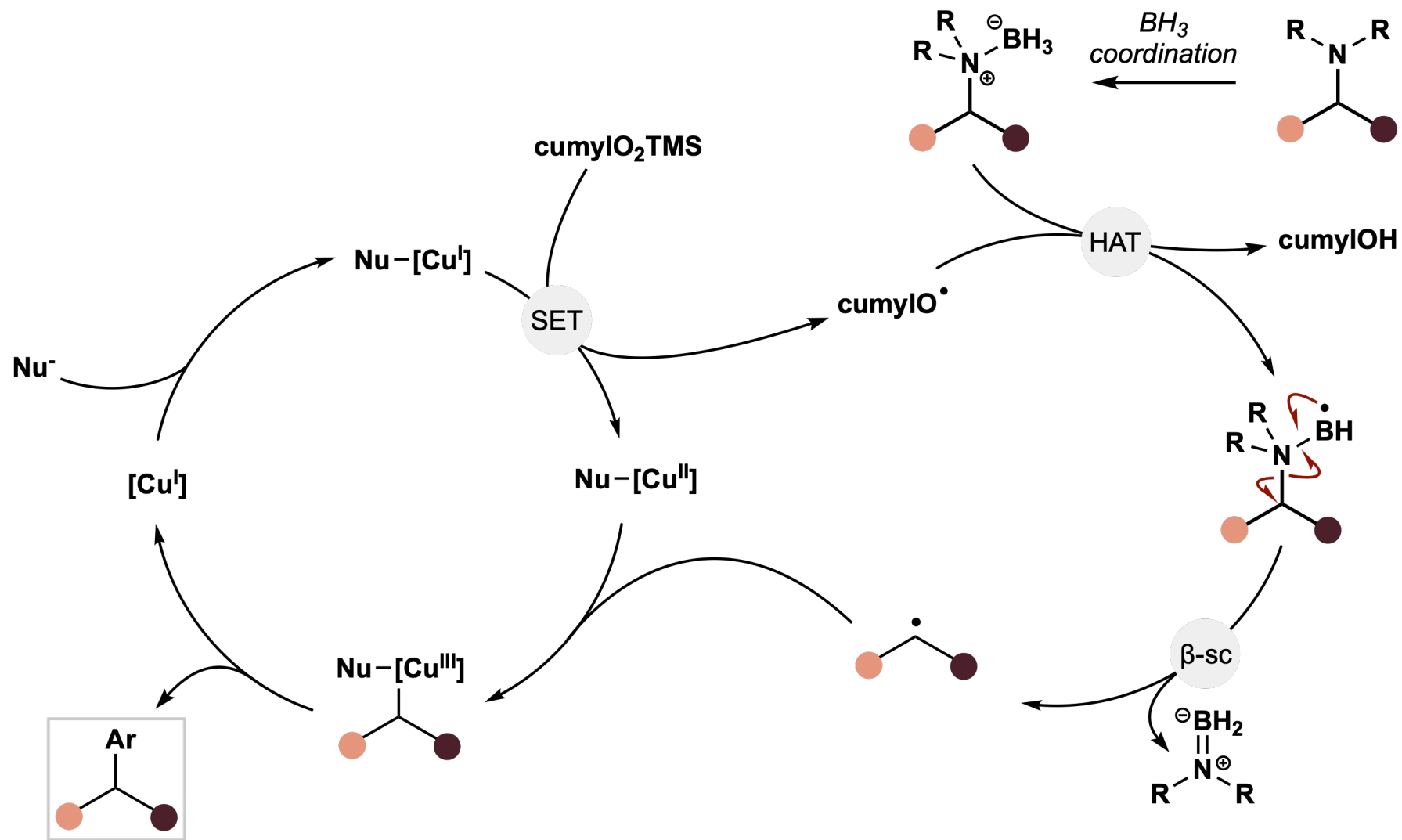
$$\Delta G^\circ = -44.2 \text{ kcal/mol}$$



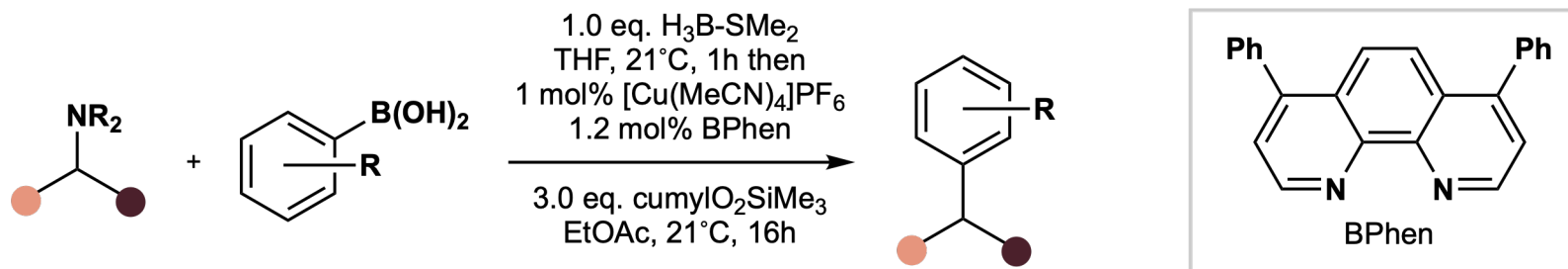
$$\Delta G^\circ = 0.6 \text{ kcal/mol}$$

Level of theory: $\omega\text{B97X-D3/def2-TZVP//}\omega\text{B97X-D3/def2-SVP}$

Deaminative Cross-Coupling Proposed Mechanism

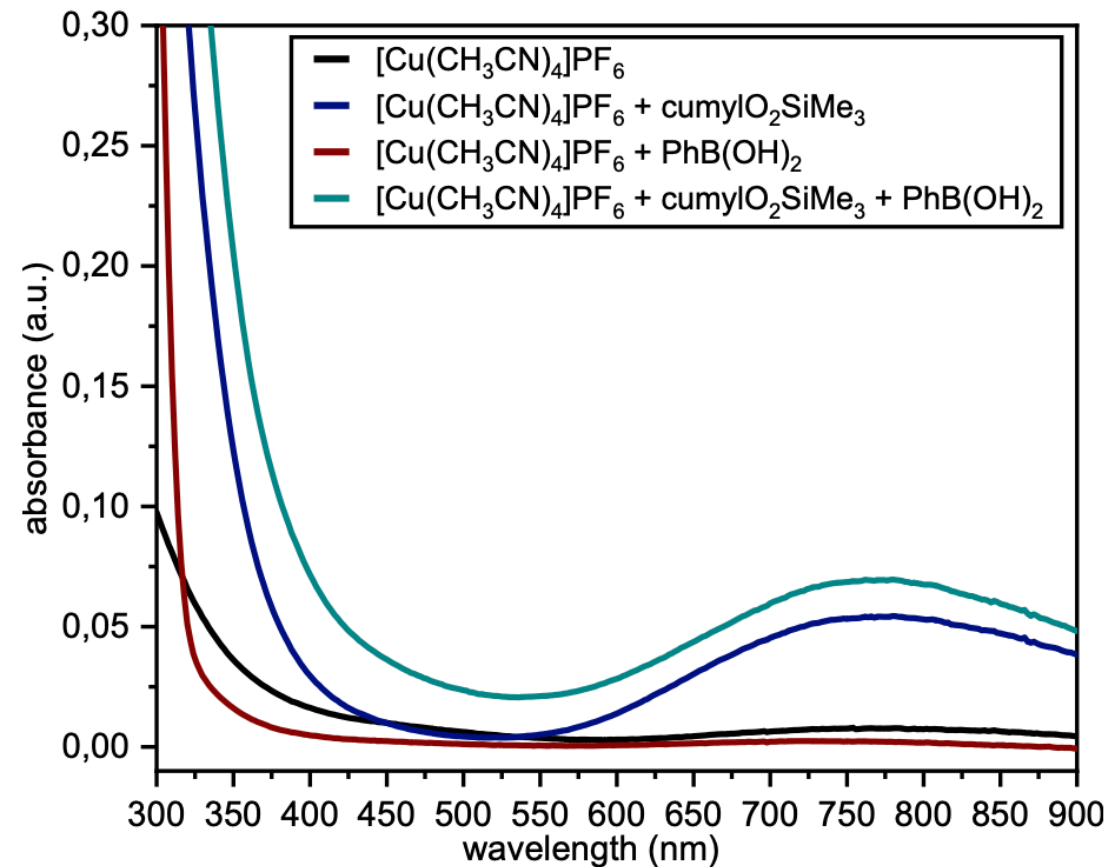
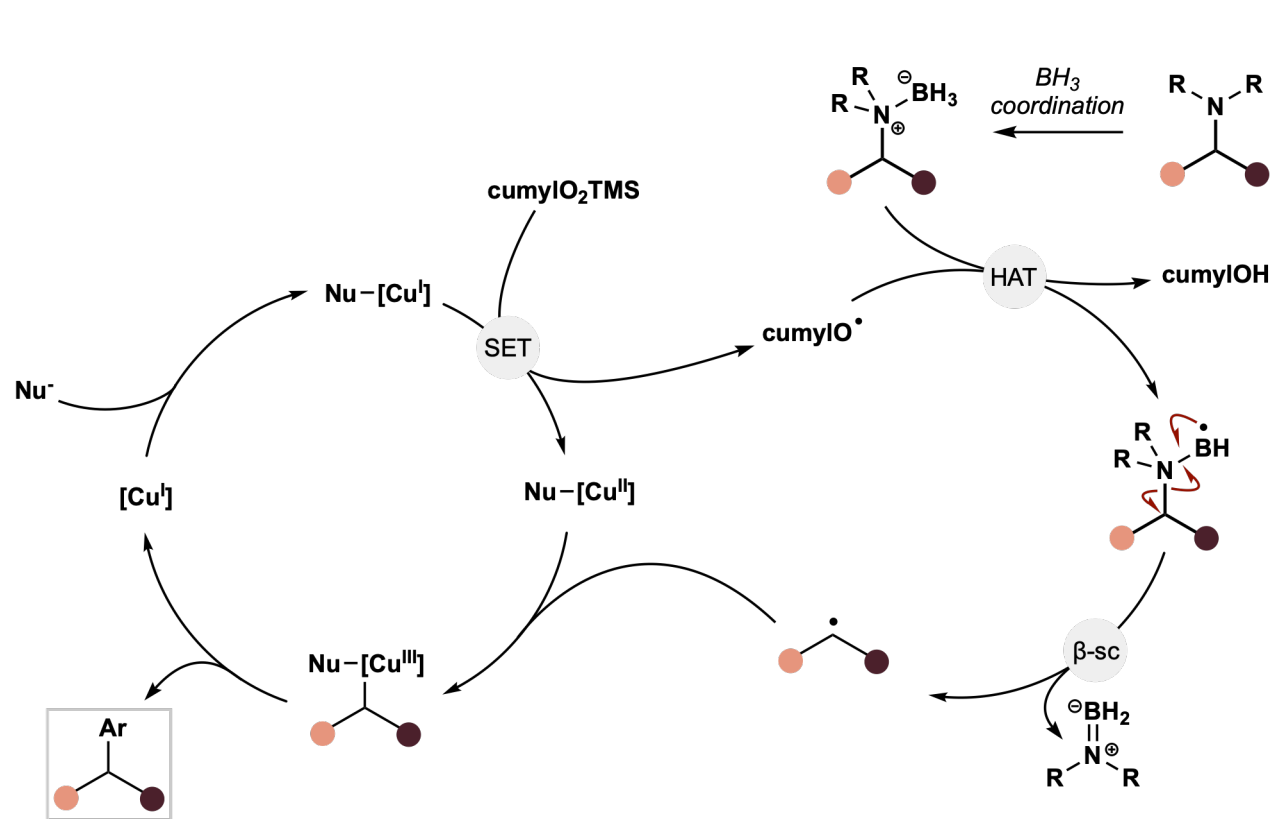


General Procedure



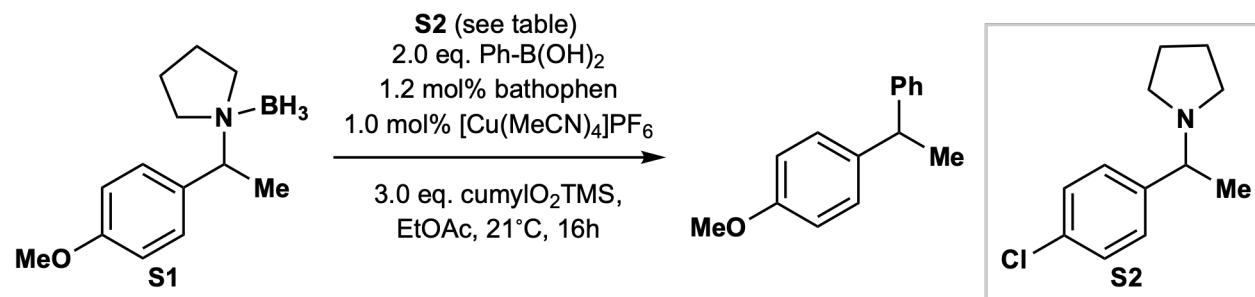
An oven-dried microwave vial equipped with a stirring bar was charged with the amine (1.0 equiv.), capped with a Supelco aluminium crimp seal with septum (PTFE/butyl), evacuated and refilled with Ar (x 3). Dry THF (0.1 M) was added, followed by $\text{H}_3\text{B-SMe}_2$ (1.0 equiv.). The mixture was stirred for 1 h at r.t. The volatiles were evaporated, and the vial was charged with $[\text{Cu}(\text{CH}_3\text{CN})_4]\text{PF}_6$ (1 mol%), **L8** (1.2 mol%) and the boronic acid (2.0 equiv.). The vial was capped with a Supelco aluminium crimp seal with septum (PTFE/butyl), evacuated and refilled with Ar (x 3). Dry and degassed EtOAc (0.1 M) was added. The mixture was stirred for 5 min and then $\text{cumylO}_2\text{SiMe}_3$ (3.0 equiv.) was slowly added. The reaction was stirred for 16 h at r.t. The volatiles were evaporated, and the crude was purified by flash column chromatography on silica gel to give the desired product.

Oxidation of Cu(I) to Cu(II) by cumyIO₂SiMe₃



Peroxide can oxidize Cu(I) to Cu(II)

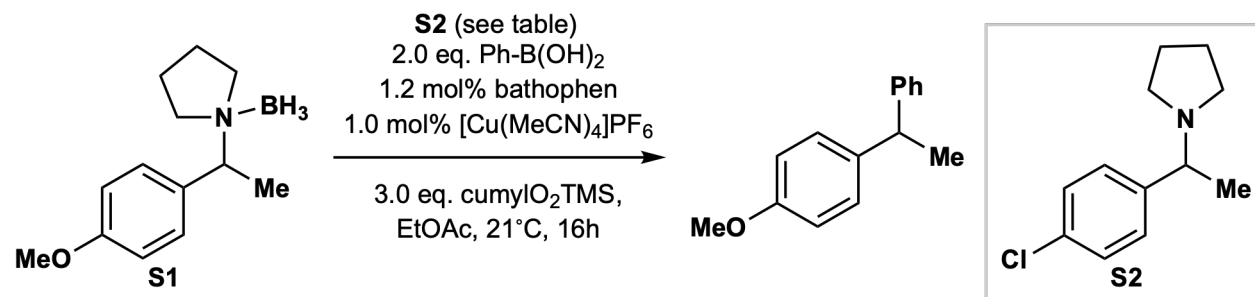
Amine Poisoning Experiment



Entry	S2 (eq.)	prdt (%)	S1 recovered (%)	S2 recovered (%)	mass balance (%)
1	0	80	12	-	92
2	0.1	65	30	-	95
3	0.25	51	39	-	90
4	0.5	32	59	-	91
5	1.0	16	77	-	93

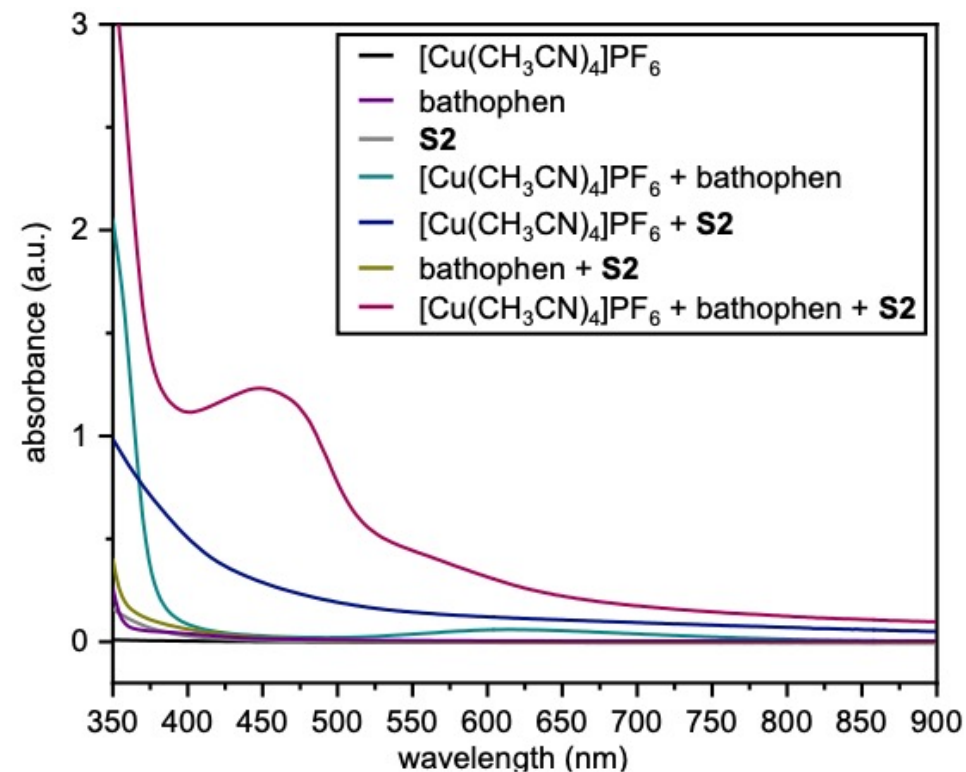
As S2 increases, recovered S1 is rising and the reaction stalls

Amine Poisoning Experiment



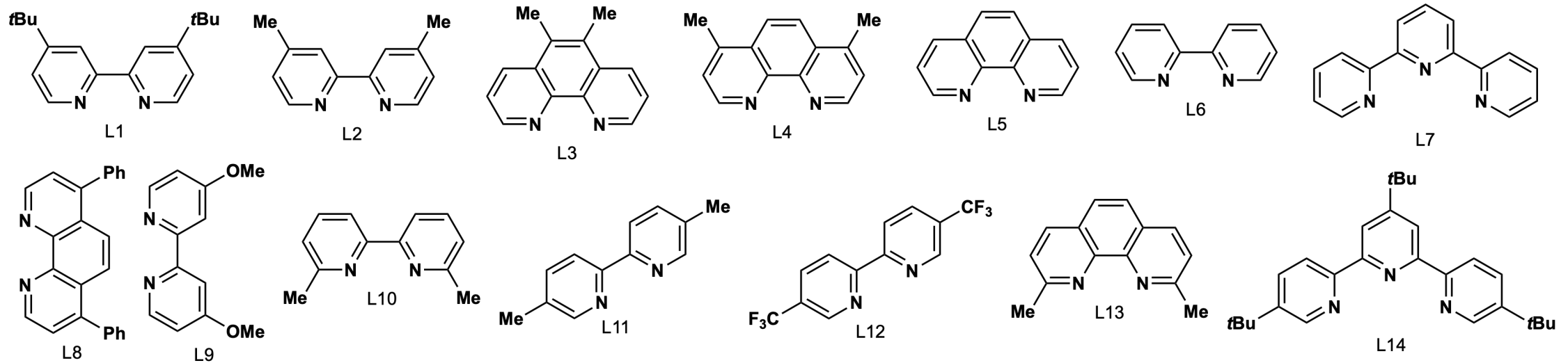
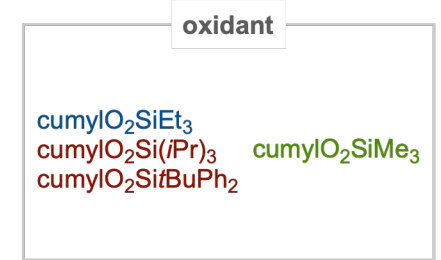
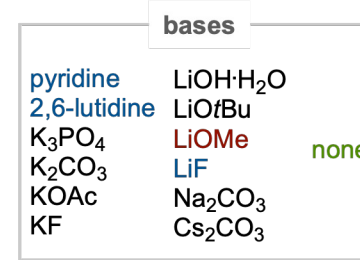
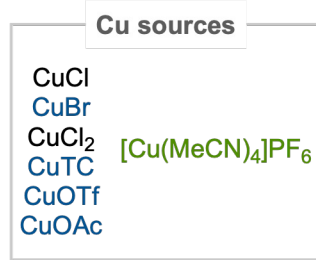
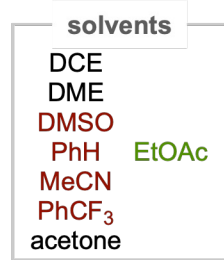
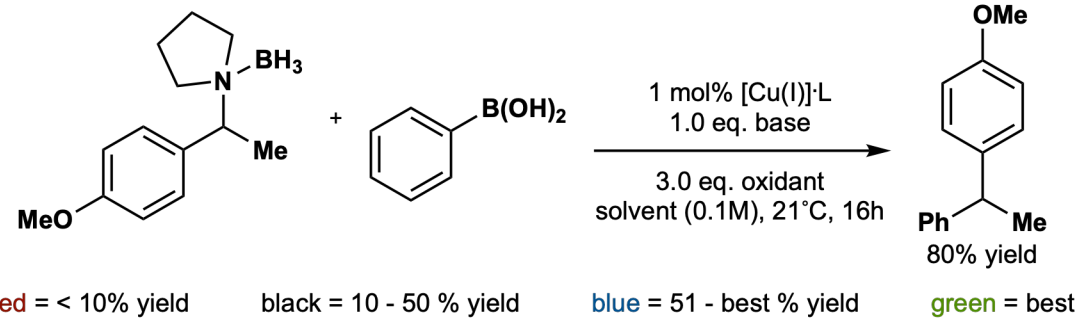
Entry	S2 (eq.)	prdt (%)	S1 recovered (%)	S2 recovered (%)	mass balance (%)
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2	0.1	65	30	-	95
3	0.25	51	39	-	90
4	0.5	32	59	-	91
5	1.0	16	77	-	93

As S2 increases, recovered S1 is rising and the reaction stalls



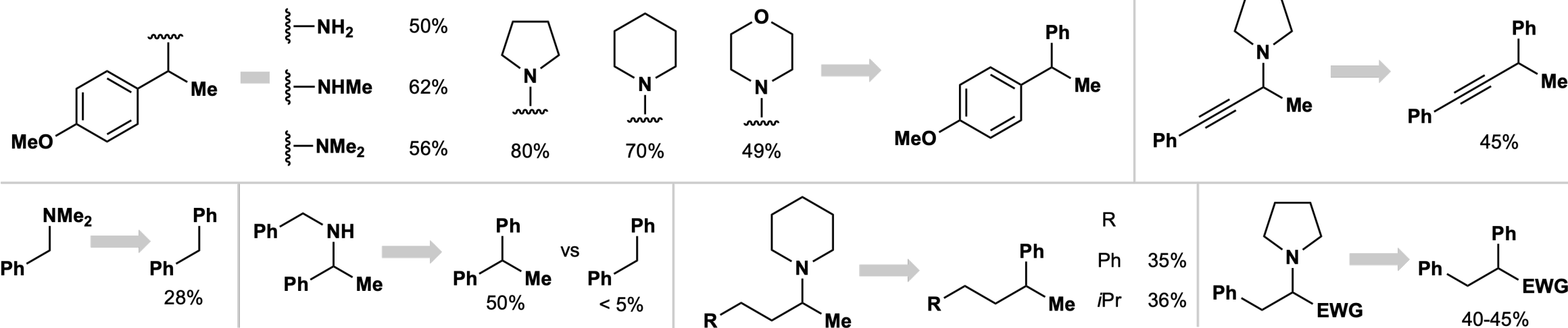
Amine DOES interact with copper-ligand complex

Reaction Optimization



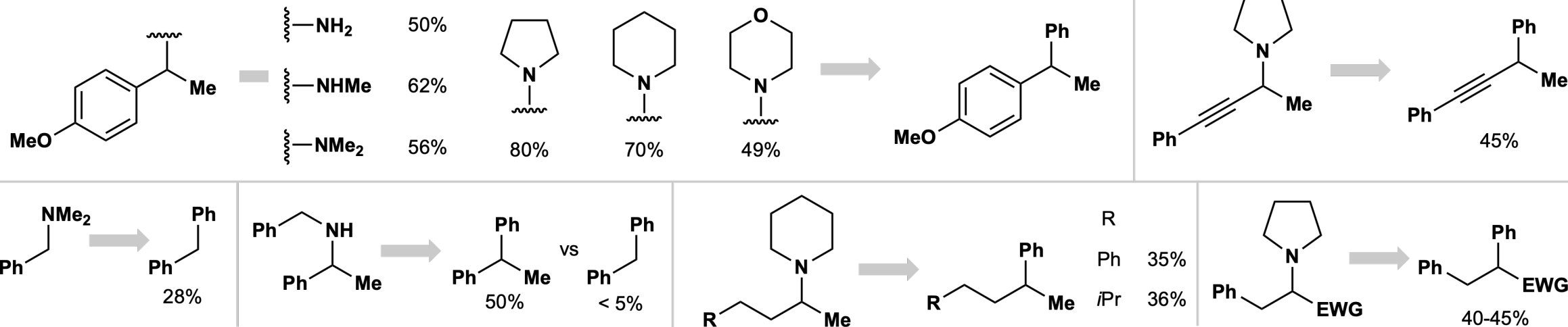
Substrate Scope

Amine scope

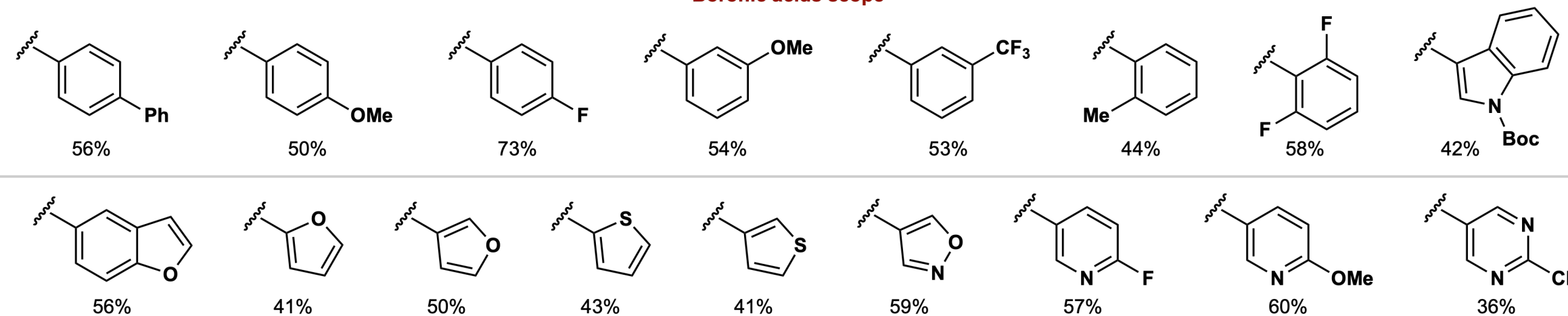


Substrate Scope

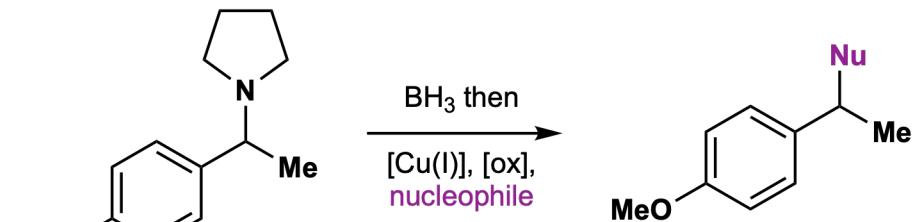
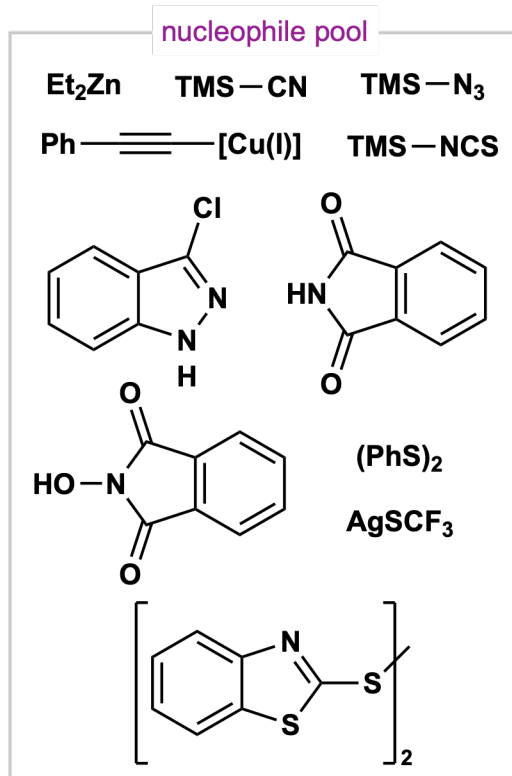
Amine scope



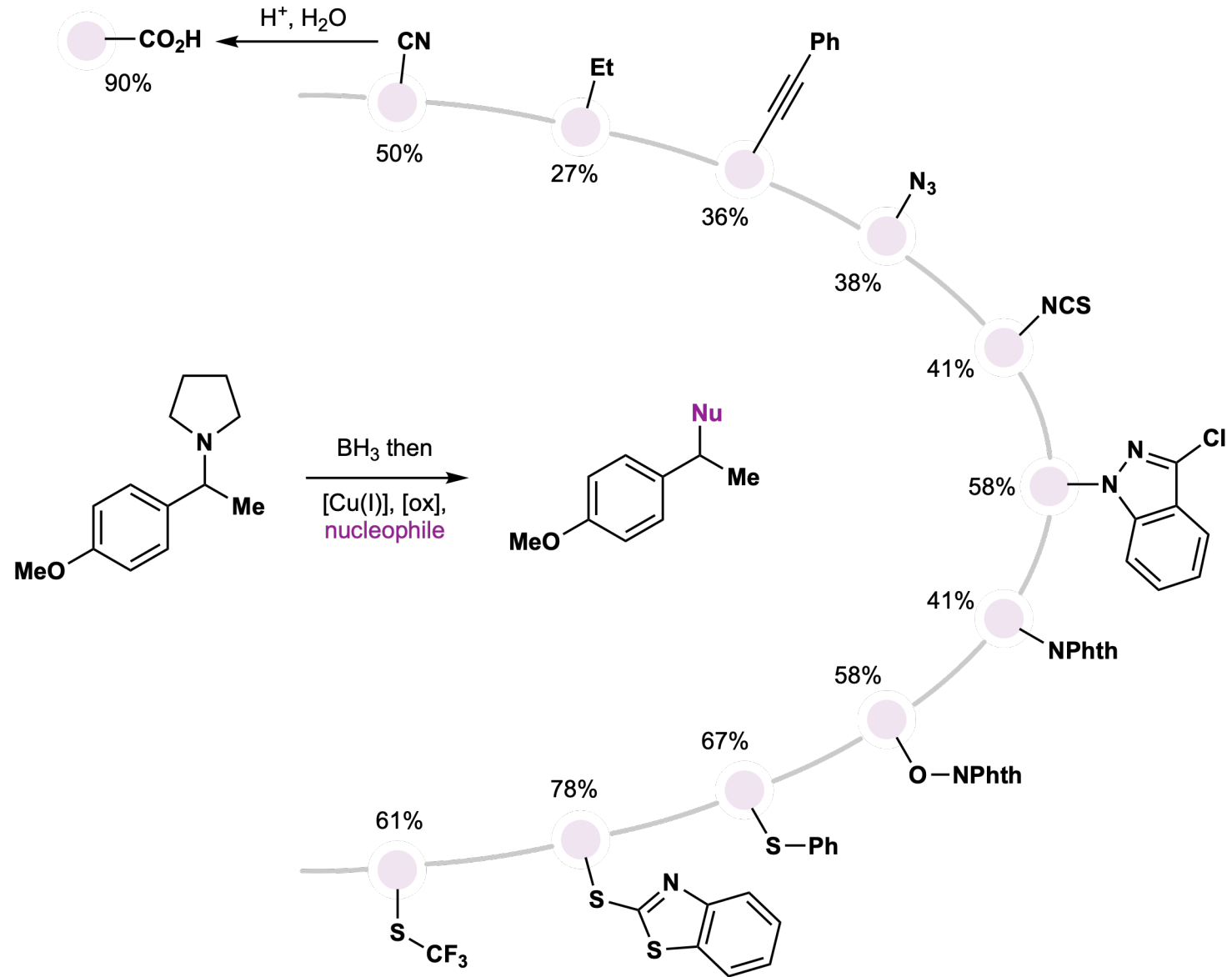
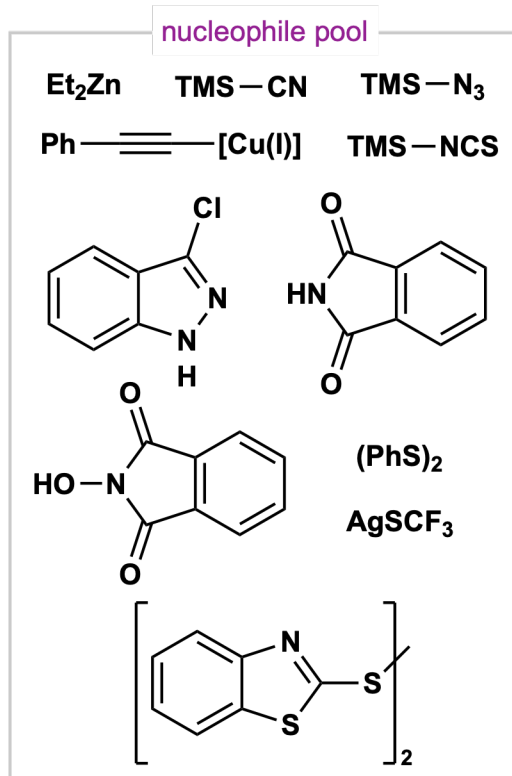
Boronic acids scope



Substrate Scope: Other Nucleophiles



Substrate Scope: Other Nucleophiles



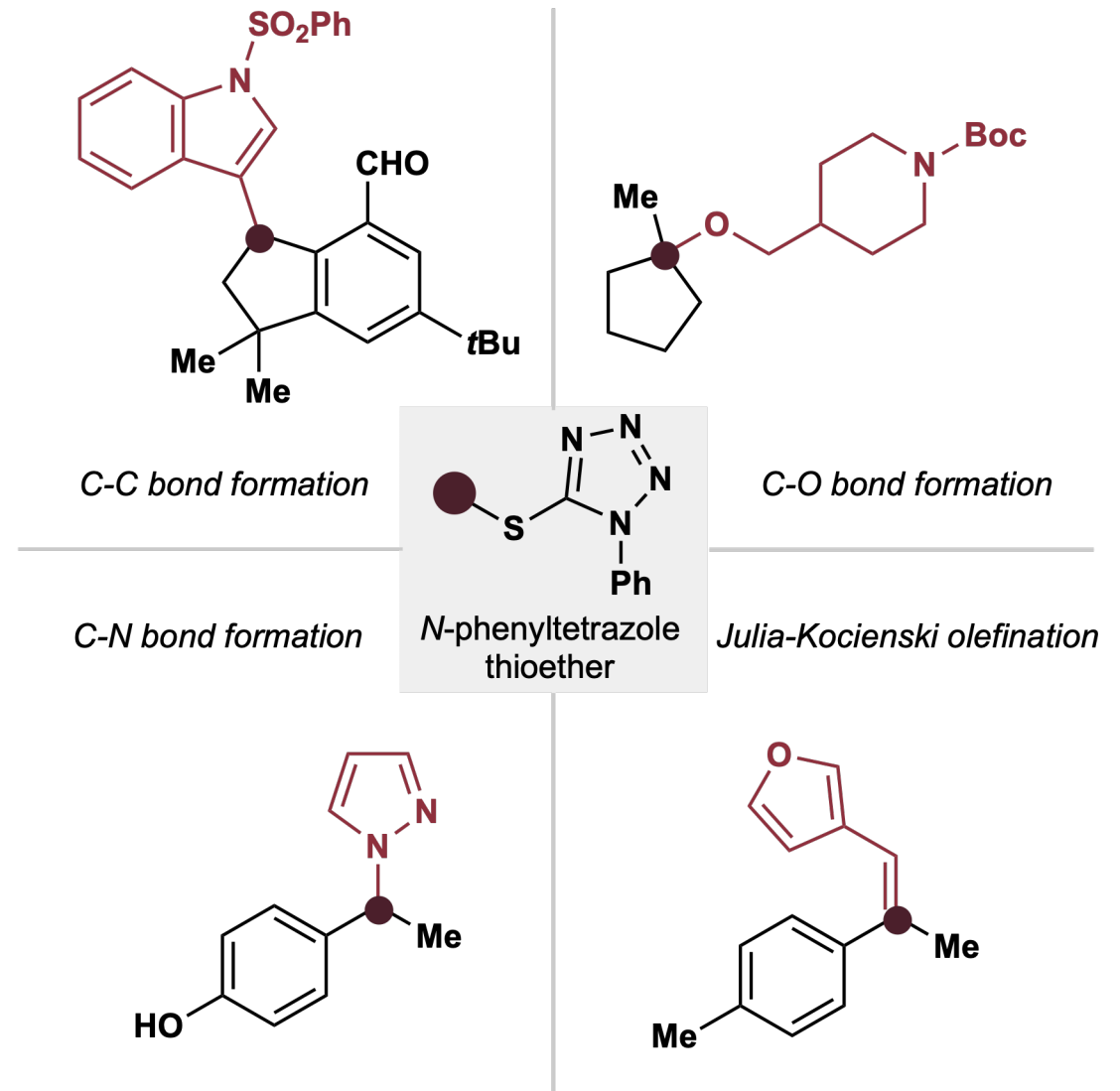
Substrate Scope Expansion

Limitations: alcohols, amides and carbamates

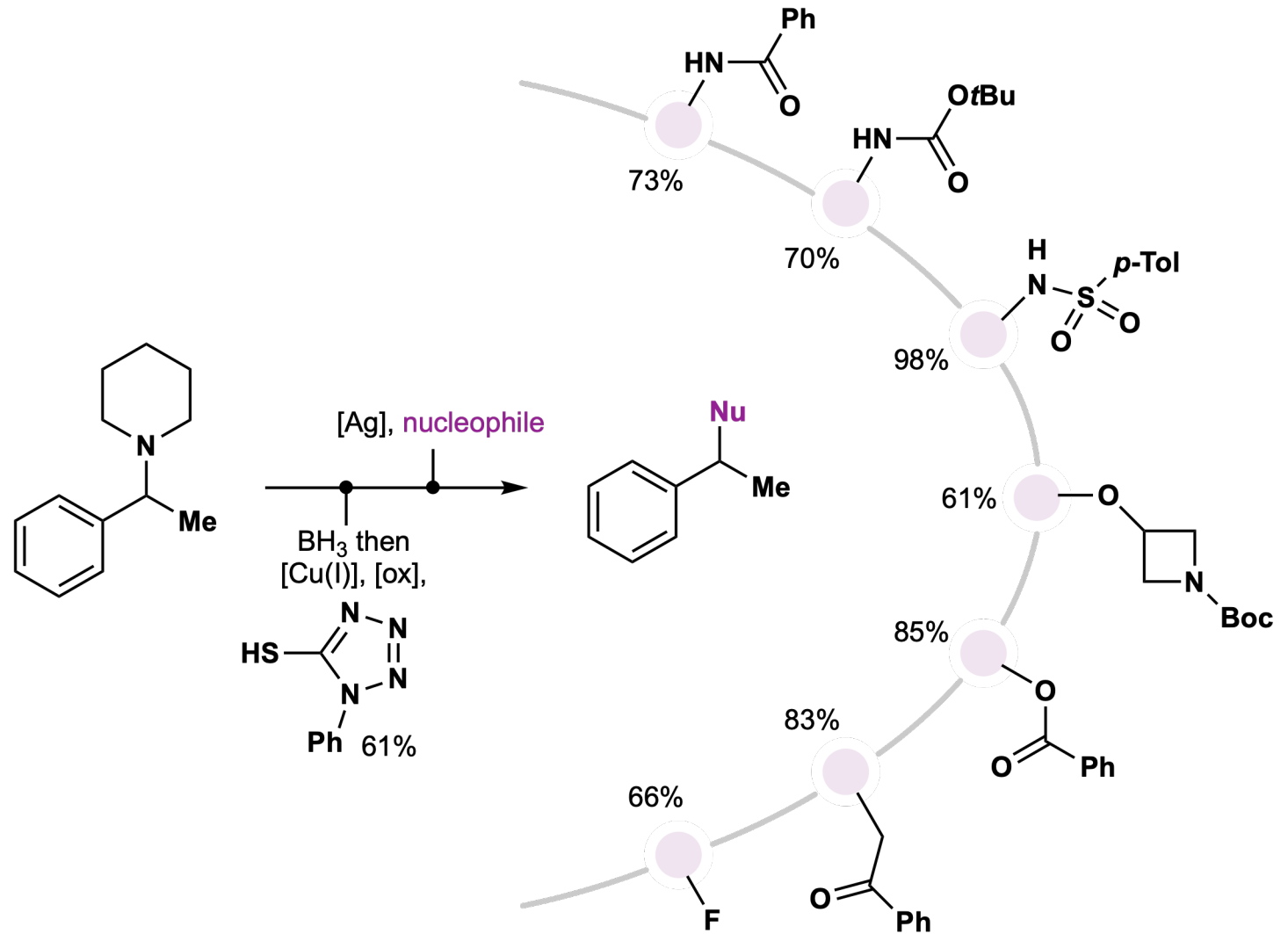
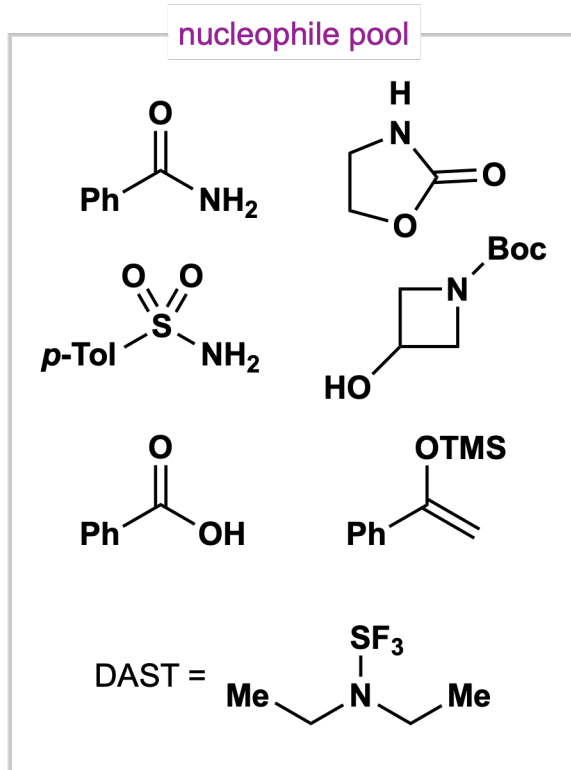
Substrate Scope Expansion

Limitations: alcohols, amides and carbamates

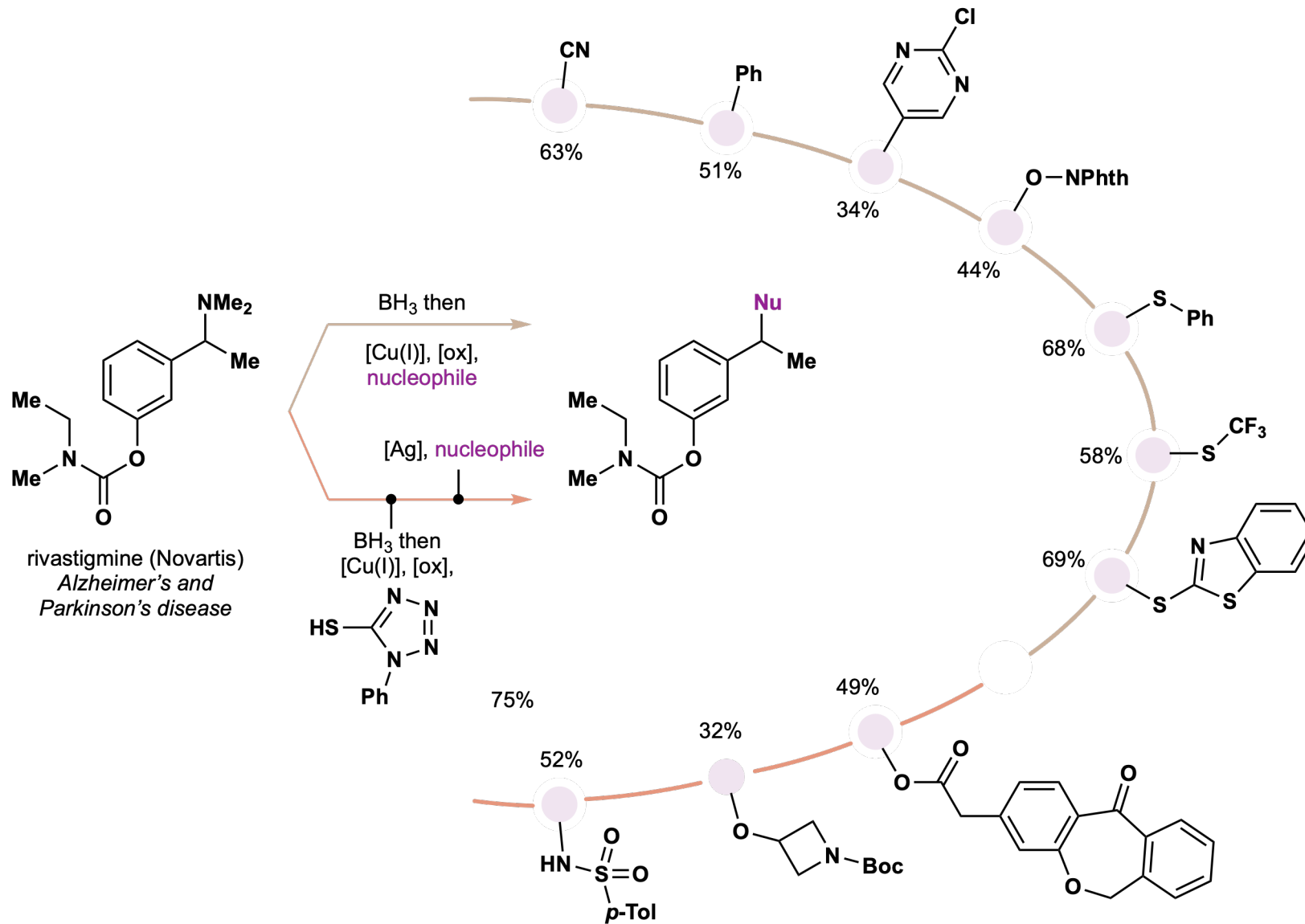
Solution: conversion of amine into a versatile intermediate



Substrate Scope Expansion



Substrate Scope: Application To Drugs



Thank you for the attention



Grazie dell'attenzione



Mulțumesc pentru atenția



Larisa P. Pop