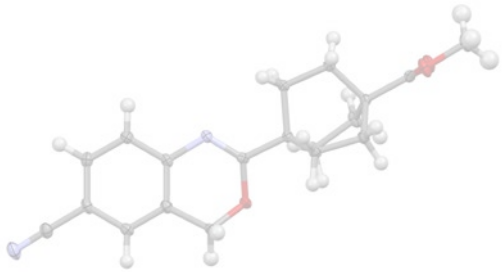
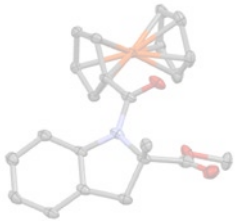
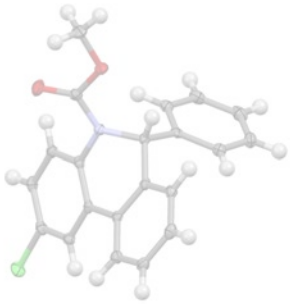
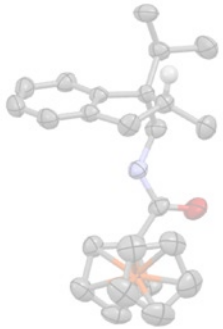
Universität Basel

Departement Chemie

Constructing rings by Pd^0-catalyzed C–H activation

Olivier Baudoin

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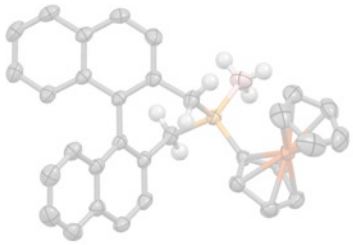
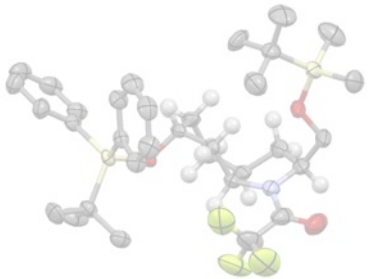
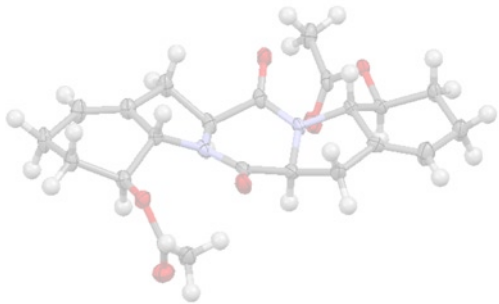
Department of Chemistry

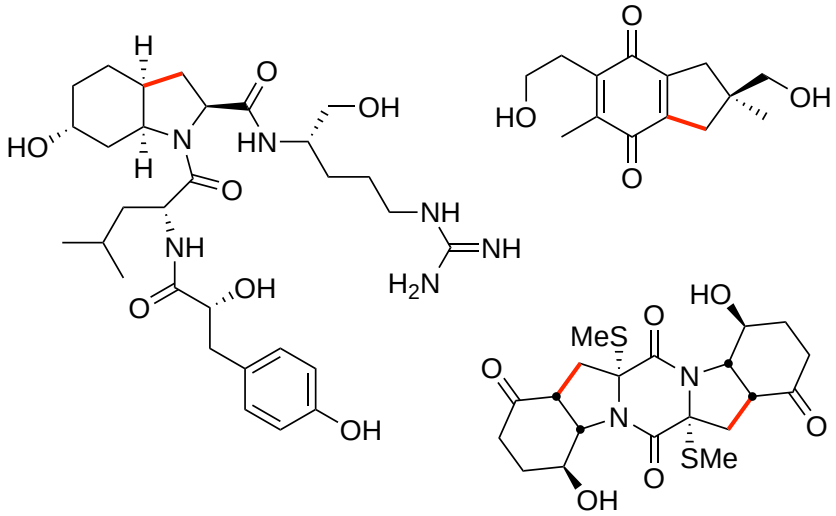




Theracat Catalysis Workshop, Basel, 25.09.2019

Current research: Pd-catalyzed C–C bond formation

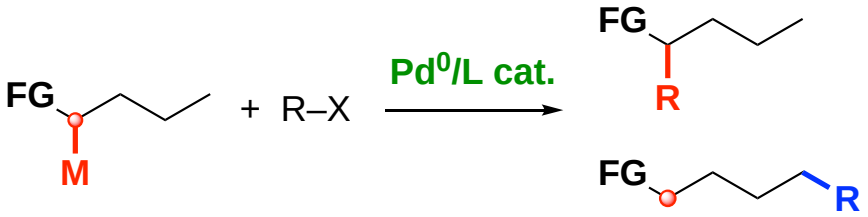
intramolecular C–H activation

- synthesis of small rings
- enantioselectivity
- application to natural products & APIs

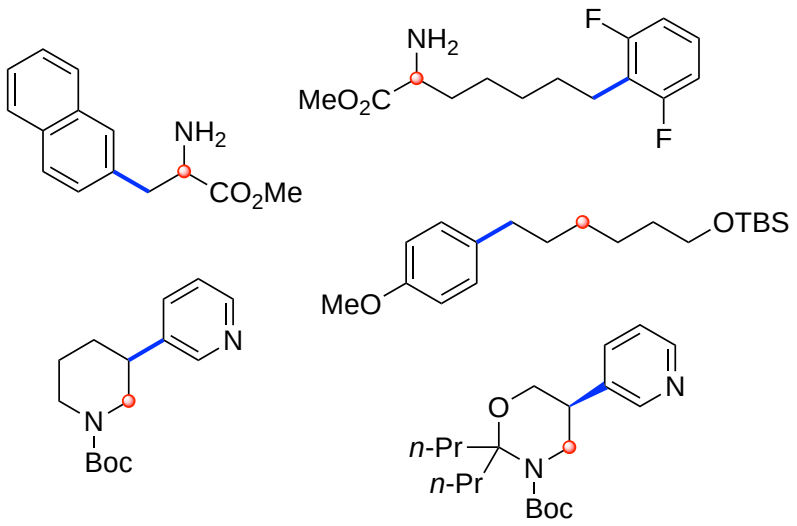


initial work: *AChIE* **2003**, 5736;
for an account: *Acc Chem Res* **2017**, 1114.

direct & migratory cross-coupling

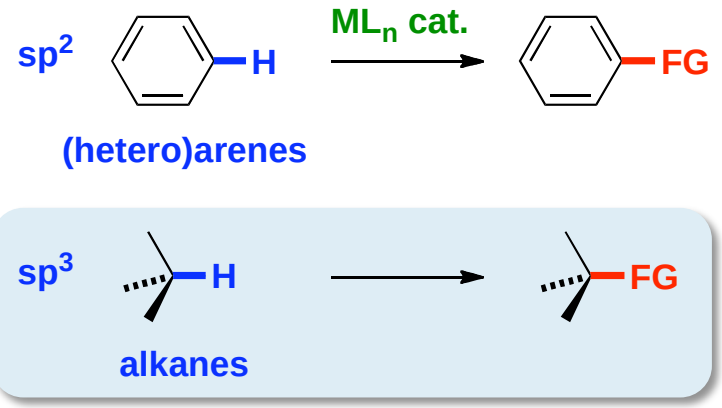


- Pd chain-walk mechanism
- ligand-controlled selectivity
- divergent access to useful intermediates



initial work: *AChIE* **2010**, 7261;
for an account: *Chimia* **2016**, 768.

Transition metal-catalyzed C–H bond functionalization

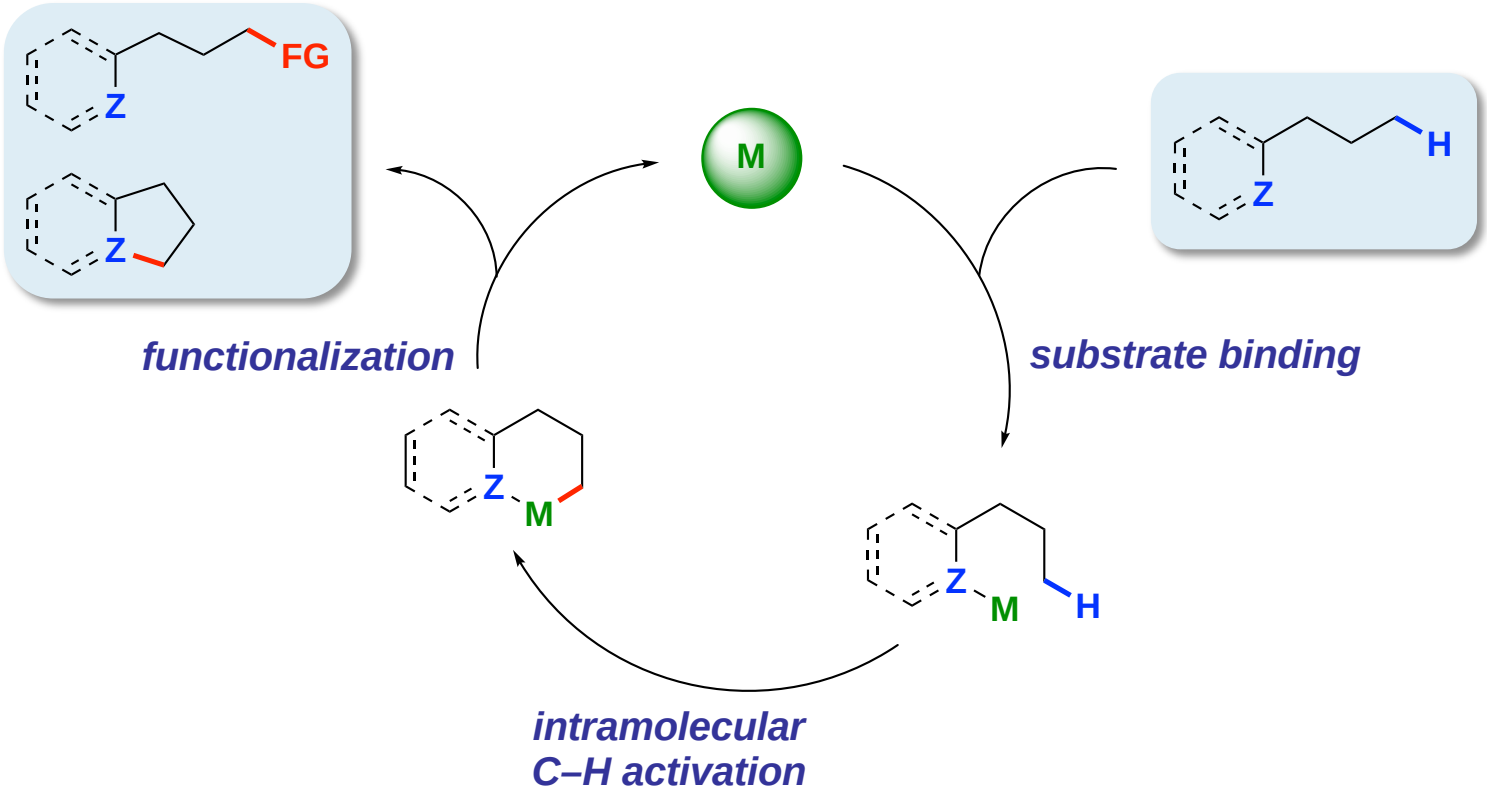


Functionalization of unactivated C(sp^3)–H bonds

- reactivity, chemo-, site- and stereoselectivity
- step-economical synthesis of useful intermediates & original scaffolds

Intramolecular organometallic C(sp^3)–H activation

General principle



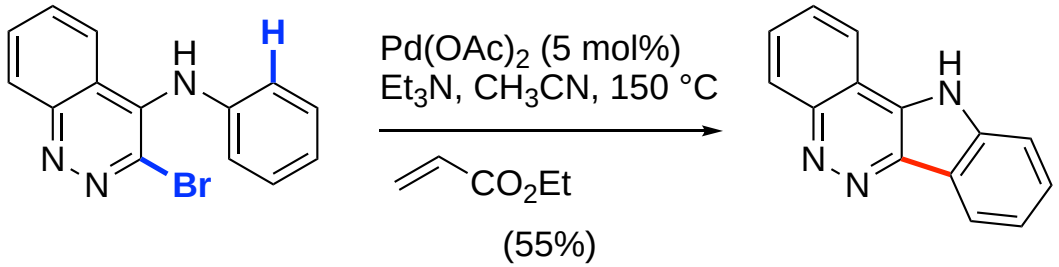
two main substrate binding modes:

Z = DG: coordination (directed C–H activation)

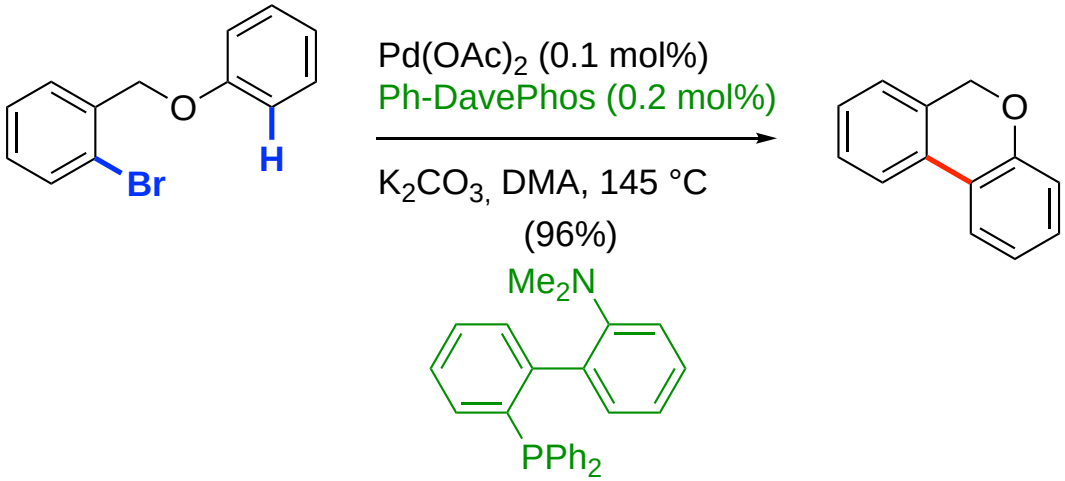
Z = C–Hal: oxidative addition

Metal-catalyzed heteroatom-directed C(sp^3)–H activation: Murai (Rh, 1997), Jun (Ru, 1998), Sanford, J.-Q. Yu, Daugulis (Pd, 2004-05), then: Corey, G. Chen, B.-F. Shi, Chatani, Chang, G. Dong, Ackermann, Gaunt, etc.

Pd^0-catalyzed intramolecular $C(sp^2)$–H activation/arylation



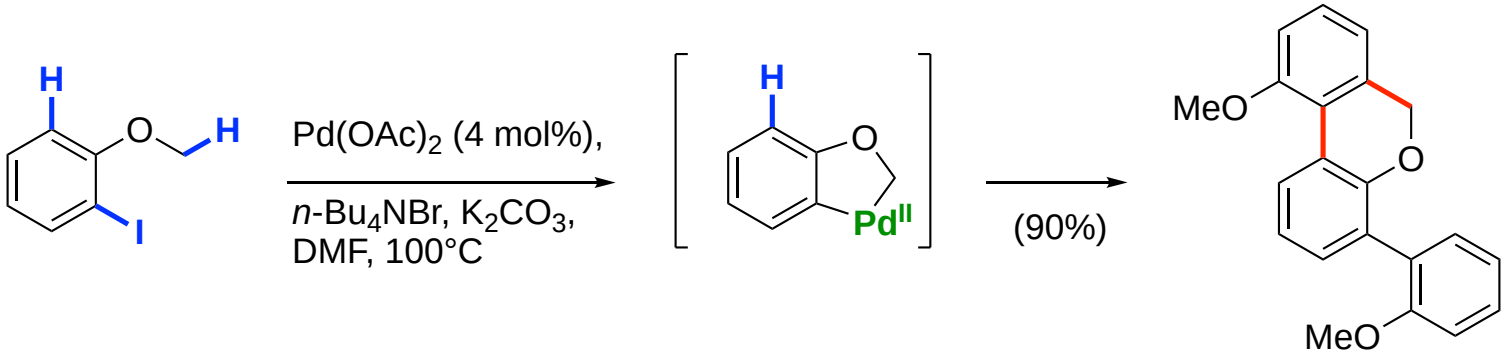
Ames *et al.*, *Tetrahedron* **1982**, 383.



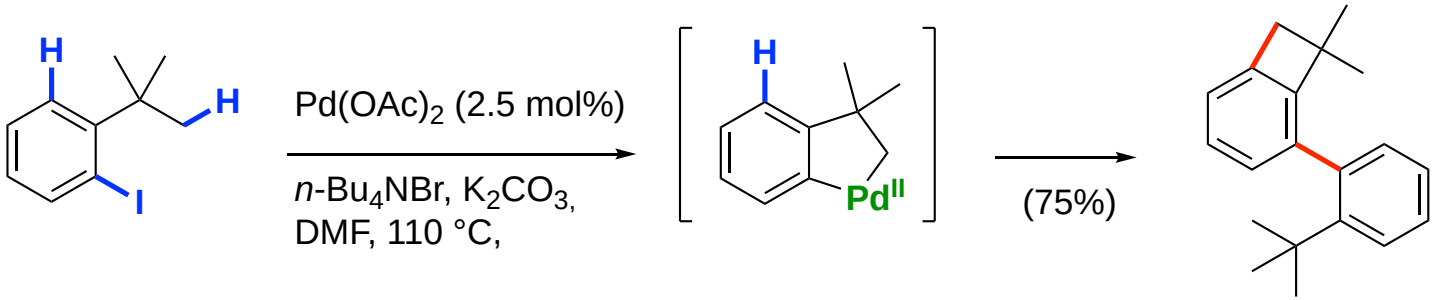
Fagnou *et al.*, *JACS* **2004**, 9186.

selected reviews: Lautens *et al.*, *Chem Rev* **2007**, 174; Ackermann *et al.*, *AChIE* **2009**, 9792.

Pd^0-catalyzed intramolecular $C(sp^3)$–H activation

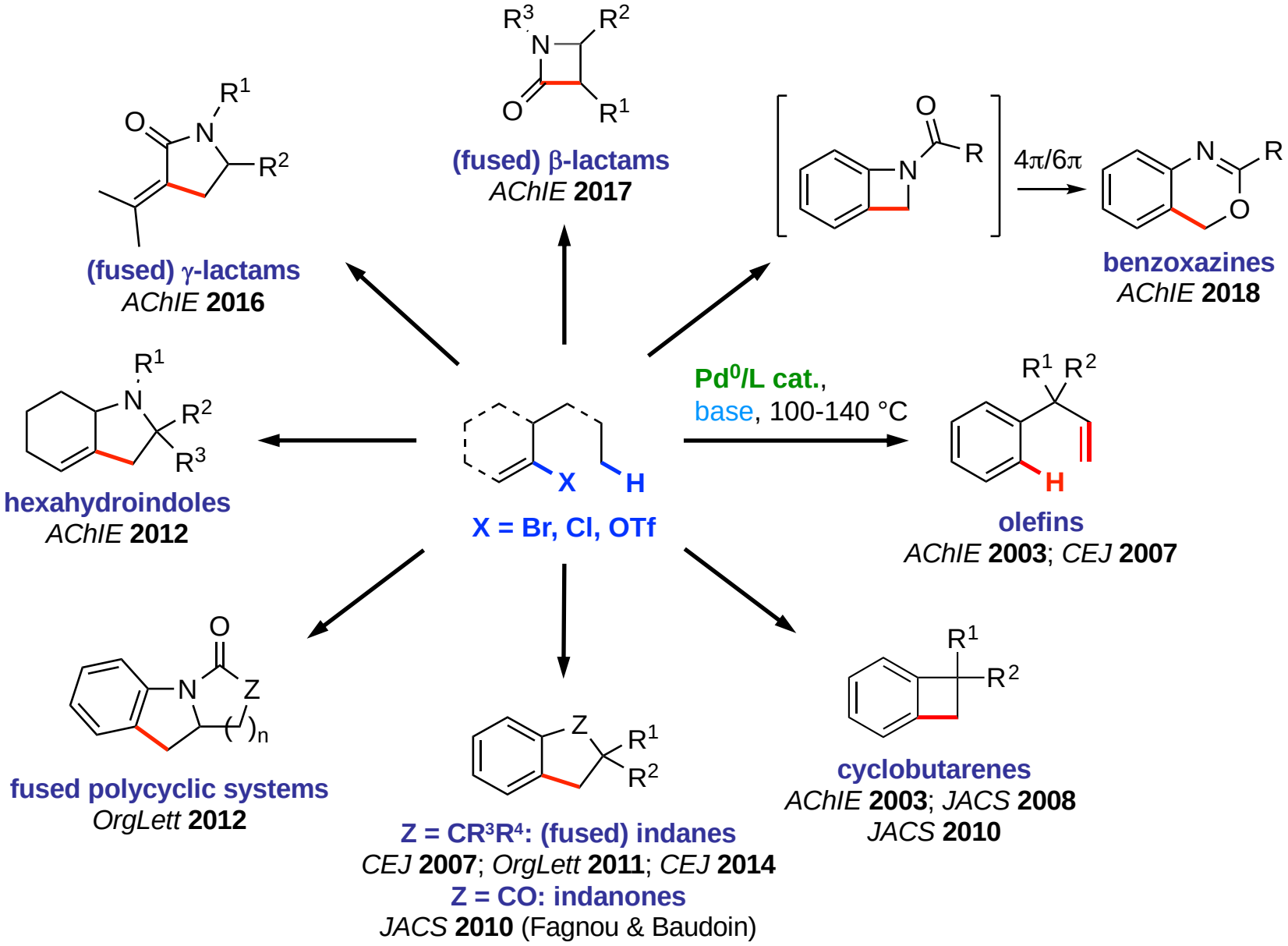


Dyker, *AChIEE* **1992**, 1023; *JOC* **1993**, 6426; *Chem. Ber.* **1994**, 739.



Dyker, *AChIEE* **1994**, 103.

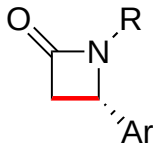
Synthesis of olefins and fused ring systems



for an account: *Acc Chem Res* **2017**, 1114.

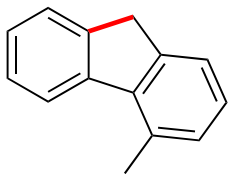
Ring construction: examples from other groups

Four-membered

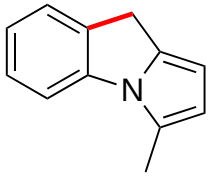


Cramer
AChIE **2014**

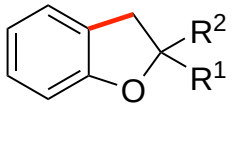
Five-membered



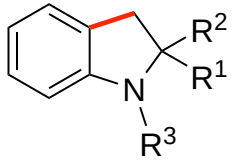
Hu
AChIE **2006**



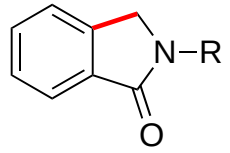
Knochel
AChIE **2006**



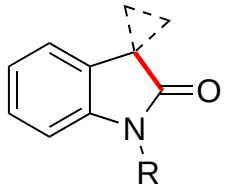
Fagnou
JACS **2007**



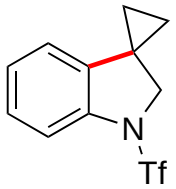
Ohno
OrgLett **2008**



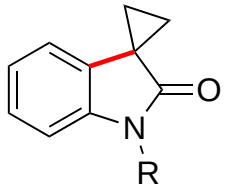
Fagnou
JACS **2010**



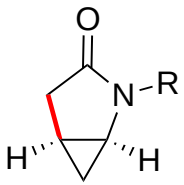
Takemoto
AChIE **2012**
ChemLett **2013**



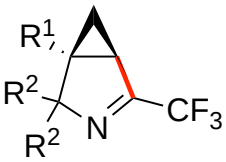
Cramer
OrgLett **2013**



Charette
OrgLett **2013**

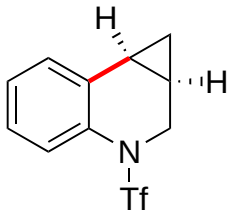


Cramer
AChIE **2015**

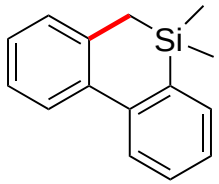


Cramer
JACS **2017**

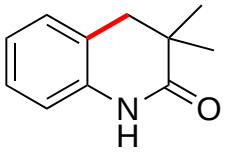
Six-membered



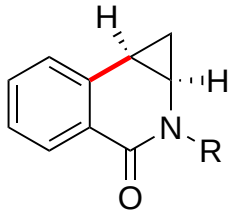
Cramer
AChIE **2012**



Xi
OBC **2012**

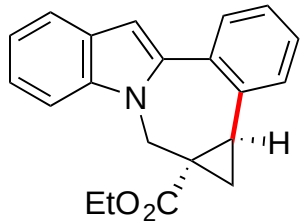


Z.-J. Shi
AChIE **2014**



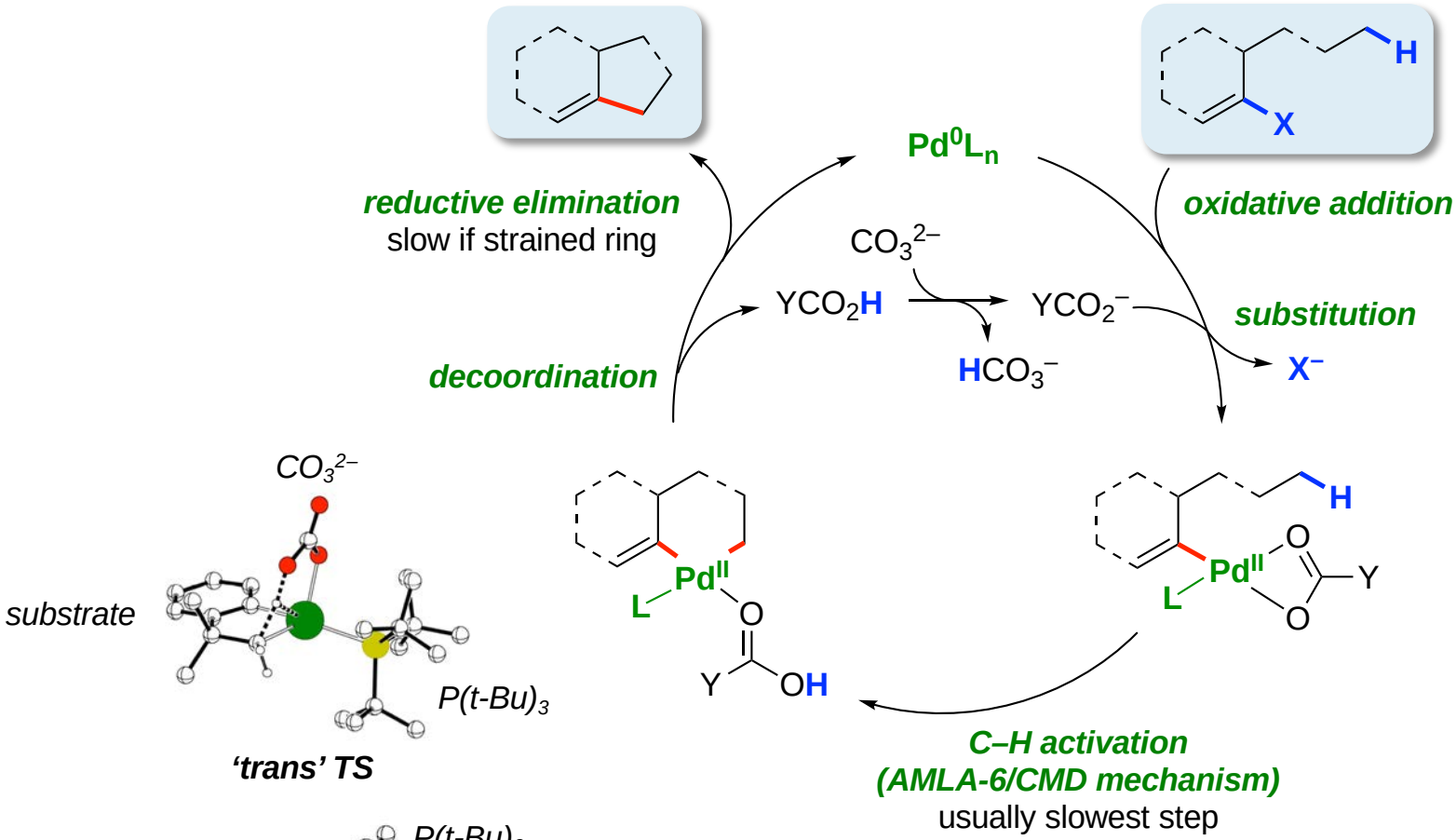
Cramer
ChemSci **2015**

Seven-membered



Cramer
ChemSci **2015**

General reaction mechanism



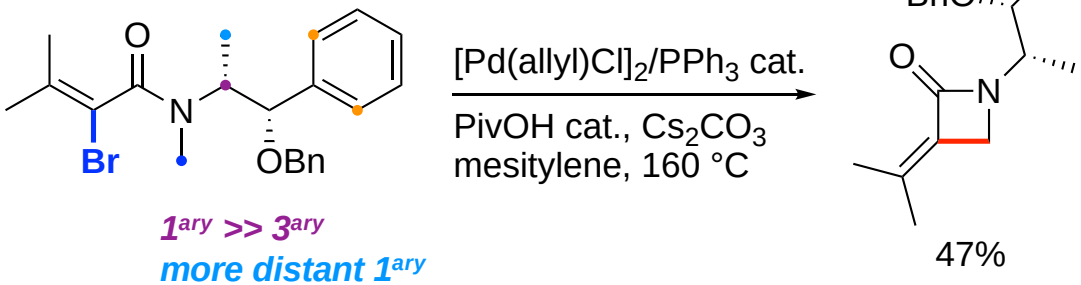
DFT calc. by E. Clot, Uni Montpellier, *JACS* **2008**, 15157; *JACS* **2010**, 10706; *Dalton Trans.* **2010**, 10528; *JOC* **2014**, 11903.

Site-selectivity of the C–H activation step

main selectivity factors

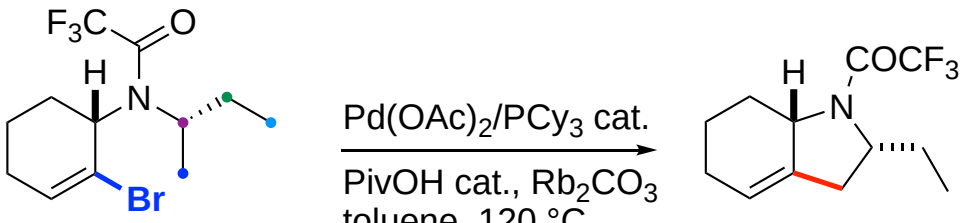
- distance of C–H bond to Pd (5 > 6 > 7-membered palladacycle)
- C–H bond type: aryl, cyclopropyl > methyl > methylene >> methine

representative examples



1^{ary} *>> 3*^{ary}
more distant 1^{ary}
sp^2 *but too far*

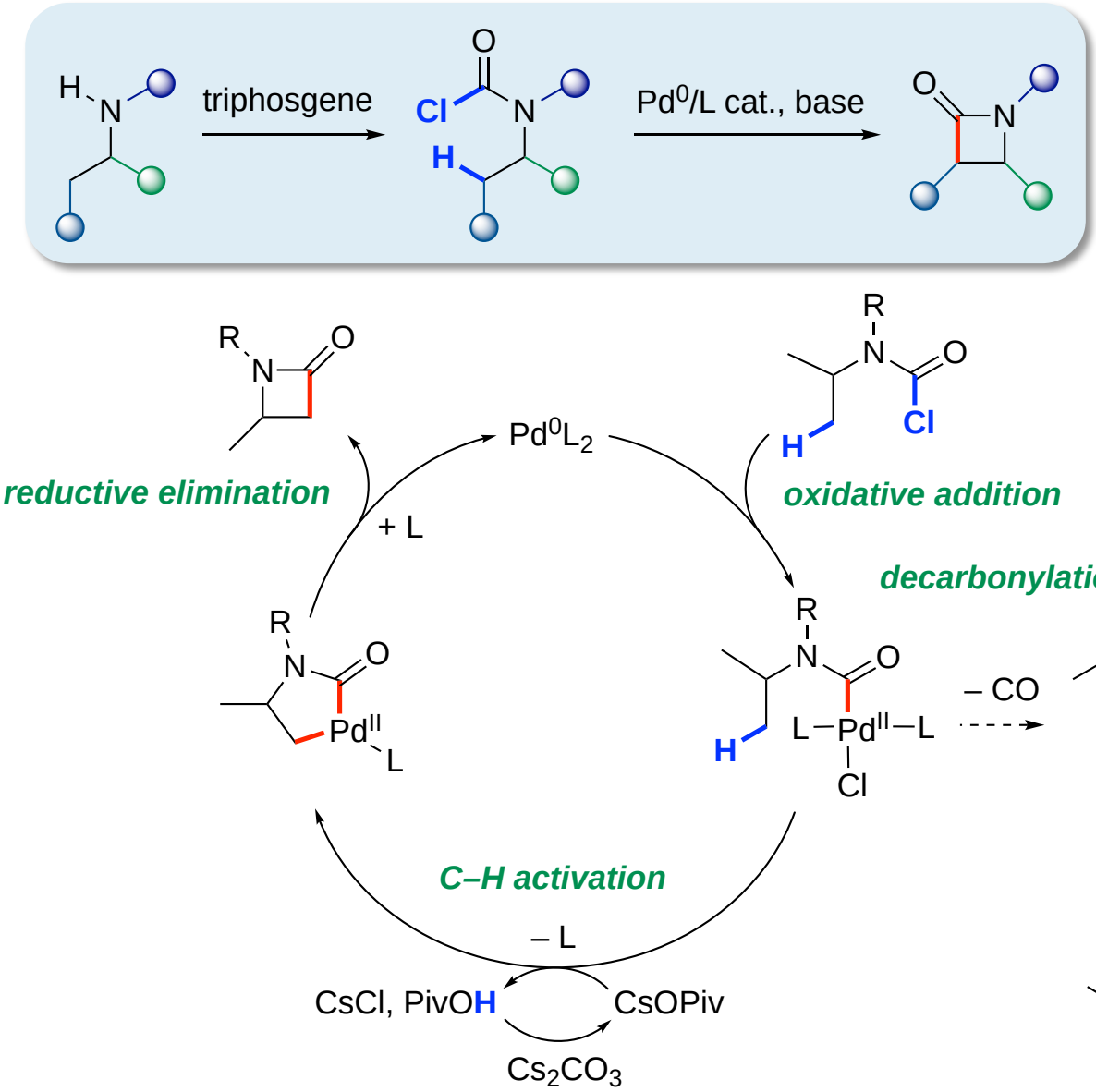
γ- and β-lactams: *AChIE* **2016**, 2805.



1^{ary} *>> 3*^{ary}
1^{ary} *> 2*^{ary}
more distant 1^{ary}

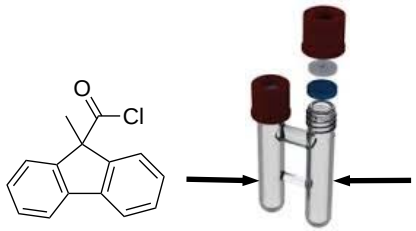
hexahydroindoles: *AChIE* **2012**, 10399.

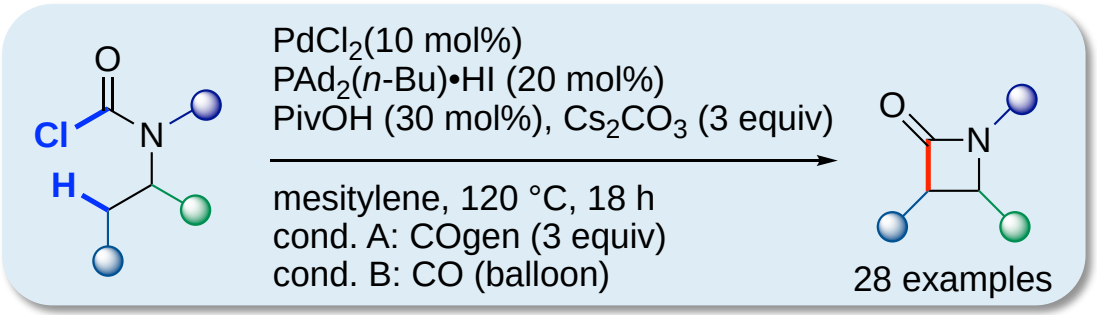
Synthesis of β-lactams

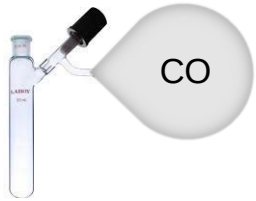


carbamoylation of benzylic C(sp³)–H bonds leading to oxindoles: Takemoto *et al.*, *AChIE* **2012**, 2763.
other C(sp³)–H activation-based syntheses of β-lactams: B.-F. Shi *et al.*, *AChIE* **2013**, 13588; Wu *et al.*, *Org Lett* **2014**, 480; Cramer *et al.*, *AChIE* **2014**, 9064; Gaunt *et al.*, *Science* **2016**, *354*, 851; *AChIE* **2017**, 11958; *Chem Sci* **2017**, 8198.

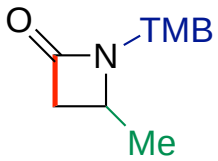
Synthesis of β-lactams



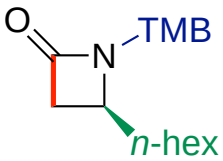




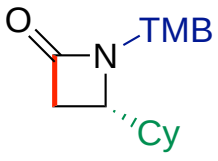
from 1ary C–H bonds



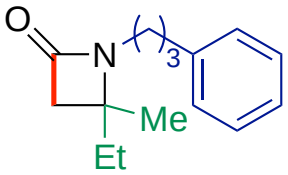
A: 92%
B: 85%
no CO: 70%



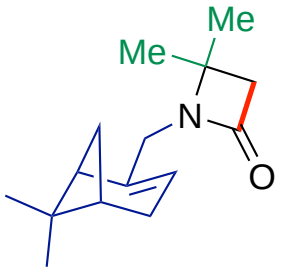
A: 92%
B: 88%



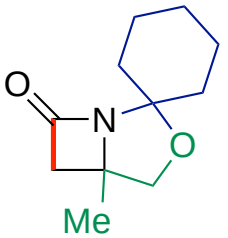
A: quant.
B: 88%



A: 73%
B: 72%

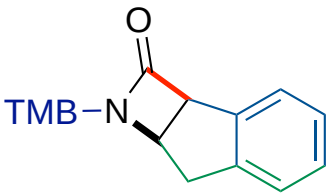


A: 53%
B: 50%
from (–)-myrtenal

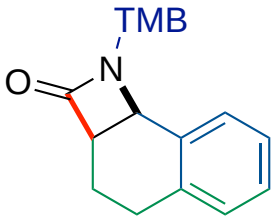


A: 70%
B: 70%

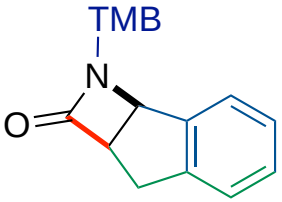
from 2ary C–H bonds



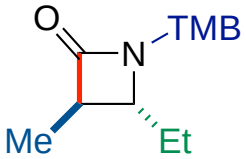
A: 93%
B: 68%



A: quant.
B: 40%

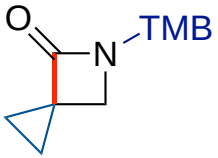


A: 47%
B: 25%

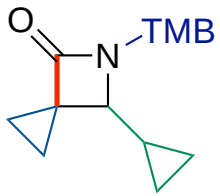


A: 51%
B: 41%
d.r. 85:15

from activated 3ary C–H bonds



A: 75%
B: 76%

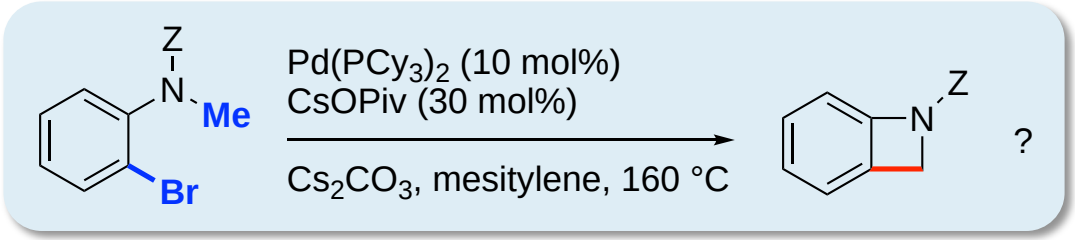


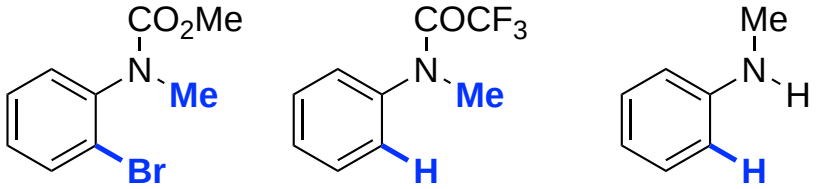
A: 81%
B: 81%

with D. Dailler & R. Rocaboy, *AChIE* **2017**, 7218.
two-chamber system: Skrydstrup *et al.*, *JACS* **2011**, 6061; *Acc Chem Res* **2016**, 594.

TMB = 2,4,6-trimethoxybenzyl

Synthesis of benzazetidines ?

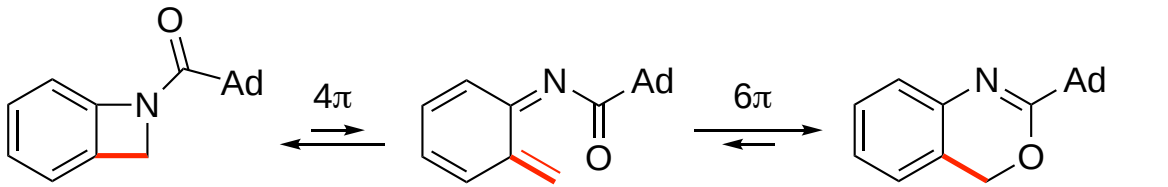




Z = CO_2Me
<5%

Z = $COCF_3$
protodebromination

Z = Me
demethylation
(45%)

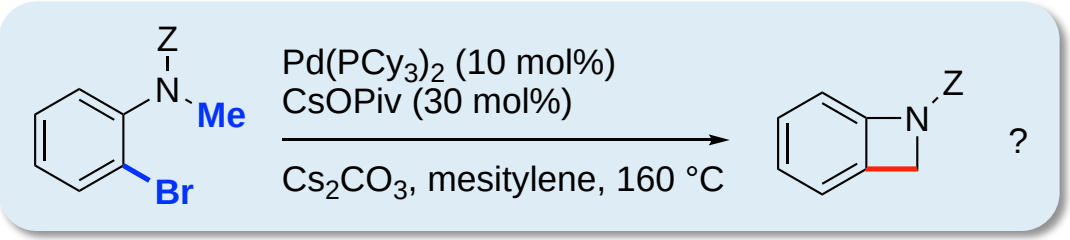


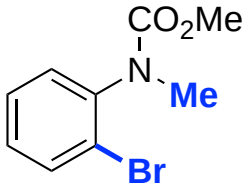
aza-*ortho*-xylylene
ΔG = 12.0 kcal mol^{-1}

ΔG = – 14.4 kcal mol^{-1}

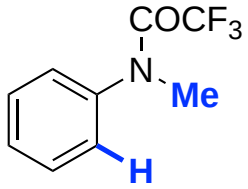
with R. Rocaboy, D. Dailler, F. Zellweger
benzocyclobutenes: *AChIE* **2003**, 5736; *JACS* **2008**, 15157; indolines: Ohno *et al.*, *Org. Lett.* **2008**,1759;
benzazetidines by directed C(sp^2)–H amination: G. Chen *et al.*, *Nat. Chem.* **2016**, 1131.

Synthesis of benzazetidines ?

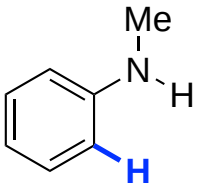




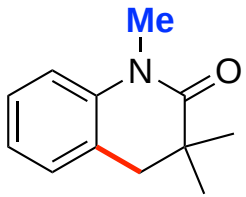
Z = CO_2Me
<5%



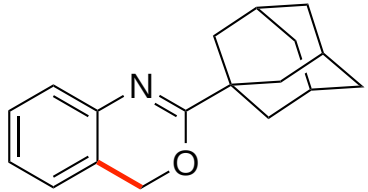
Z = $COCF_3$
protodebromination



Z = Me
demethylation
(45%)



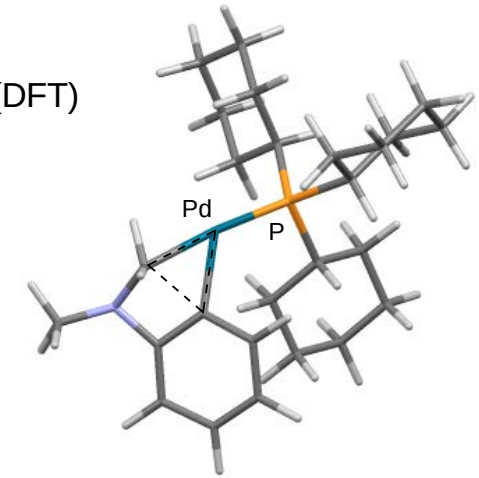
Z = Piv
wrong site-selectivity
(80%)



Z = COAd
rearranged product
(54%)

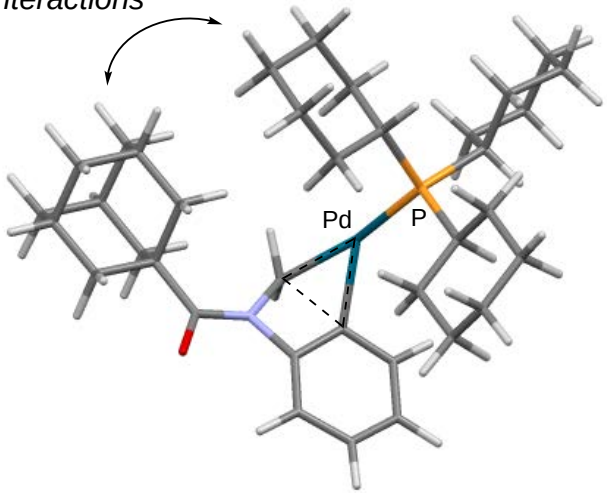
- reductive elimination = rate-limiting step (k_H/k_D = 1)
- Ad group favors reductive elimination by dispersion (DFT)

$\Delta G^{\ddagger}$ (COAd) 21.7 kcal mol^{-1}
$\Delta G^{\ddagger}$ (Me) 27.6 kcal mol^{-1}
$\Delta G^{\ddagger}$ (CO_2Me) 26.8 kcal mol^{-1}



Z = Me

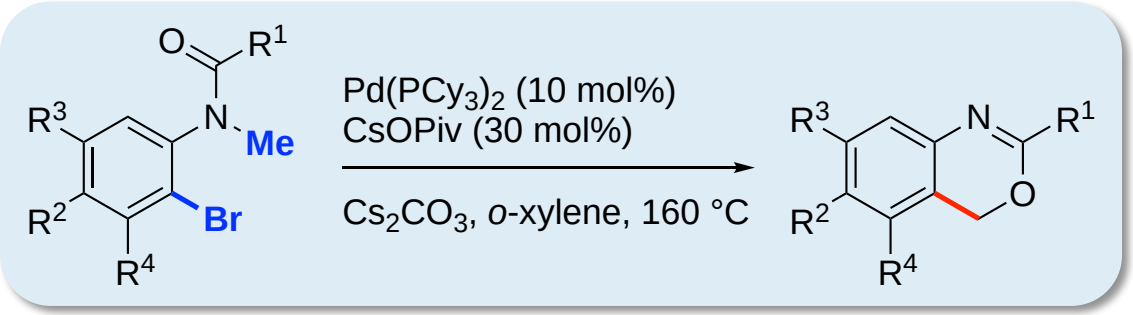
stabilizing interactions

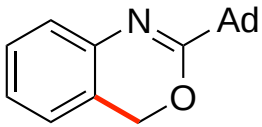


Z = COAd

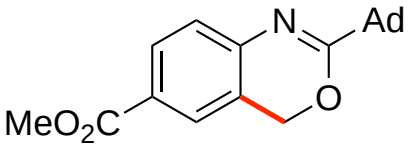
with E. Clot, Uni Montpellier, PBE0-D3(BJ)/def2-QZVP*+SMD, 433 K
review on London dispersion: Schreiner *et al.*, *AChIE* **2015**, 12274.

Synthesis of benzoxazines !

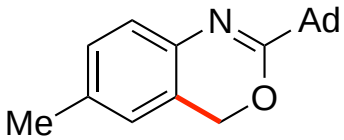




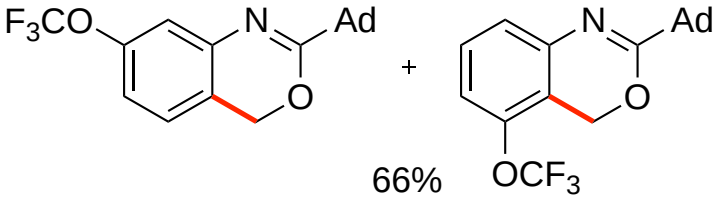
84%



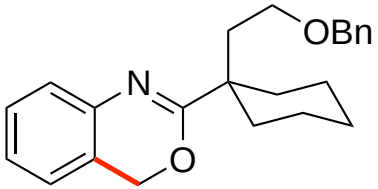
81%



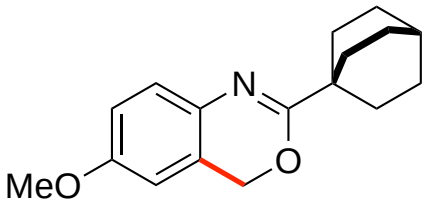
86%



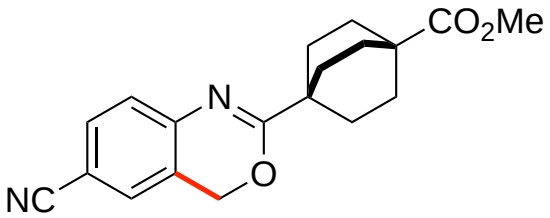
66%
6.7:1



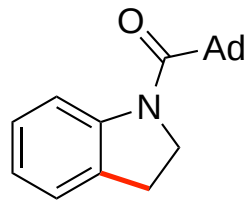
82%



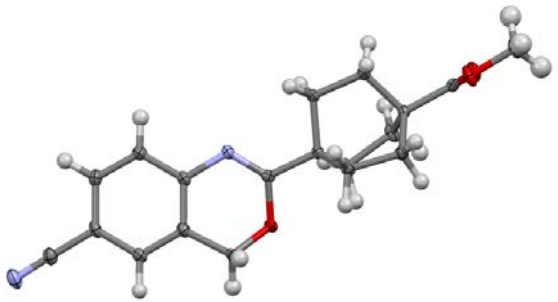
50%



45%



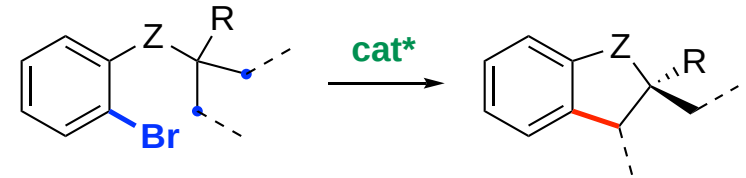
80%



with R. Rocaboy, D. Dailler, F. Zellweger, in collaboration with Spirochem, *AChIE* **2018**, 12131.

Enantioselectivity

type I: desymmetrization of enantiotopic alkyl groups

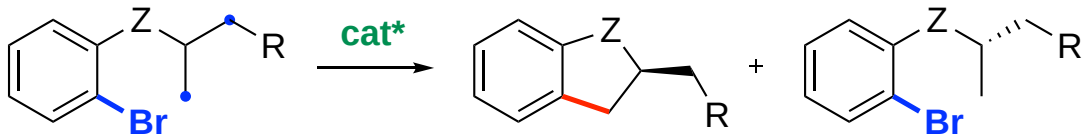


creation of stereocenter(s) remote to activated C–H bond

1^{ary}, 2^{ary} C–H bonds

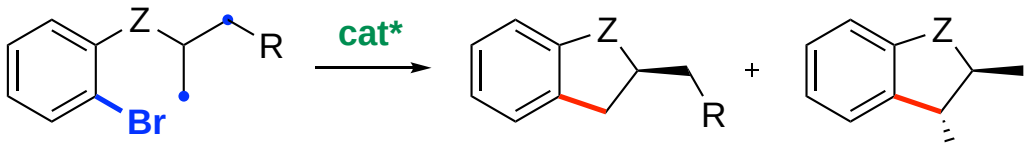
type II: kinetic resolution

- classic KR



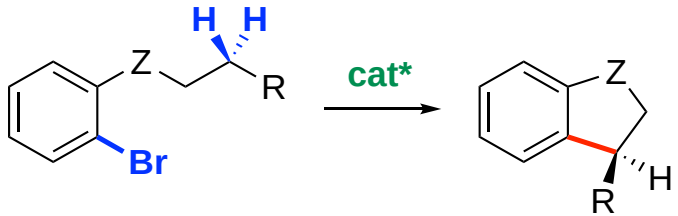
(±)

- parallel KR



(±)

type III: activation of enantiotopic 2^{ary} C–H bonds

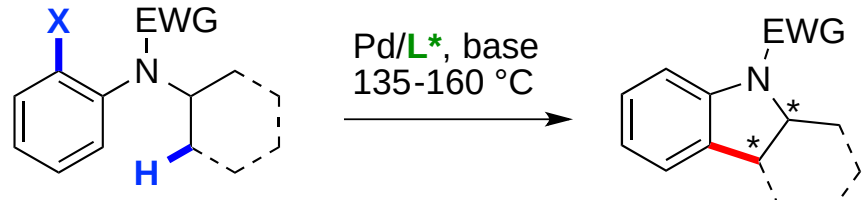


creation of stereocenter at activated C–H bond

reviews: Cramer et al., *Chem. Rev.* **2017**, 8908; J.-Q. Yu et al., *Science* **2018**, *359*, eaao4798.

Enantioselective C(sp^3)–H arylation with a chiral ligand

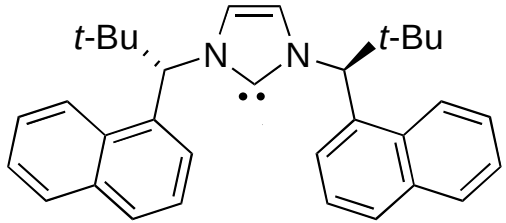
concurrent work



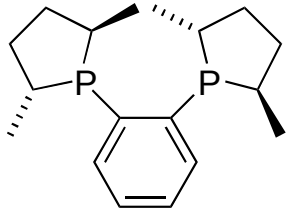
1^{ary}-2^{ary} C–H bonds

(fused) indolines

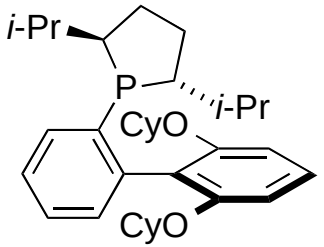
L*:



X = Br, EWG = CO_2Me
e.r. up to 99.5:0.5
Kündig et al.
AChIE **2011**, 7438; *Chem Sci* **2012**, 1422.



X = Br, EWG = CO_2Me
e. r. up to 96.5:3.5
Kagan et al.
Chem Commun **2011**, 11483.

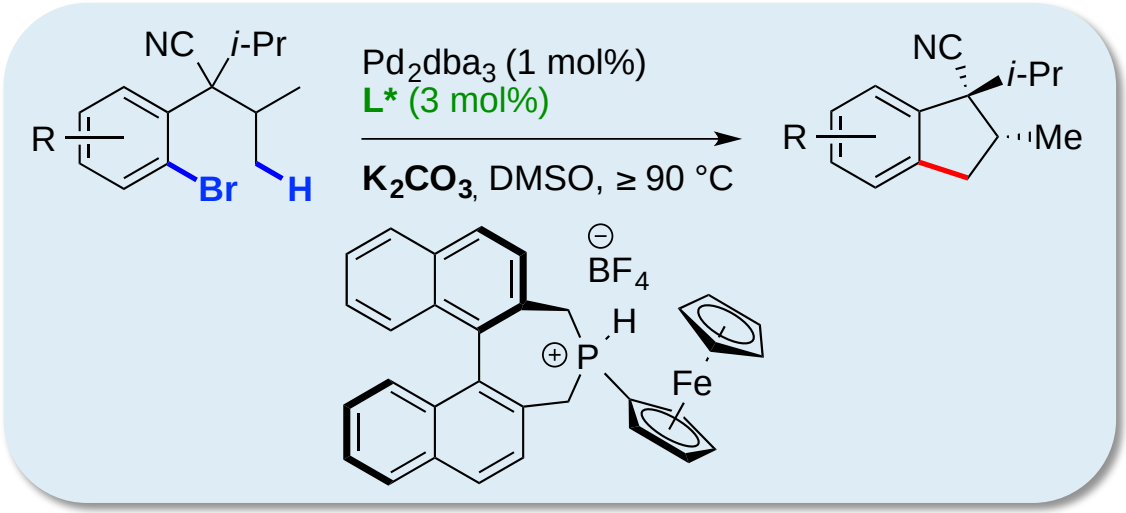


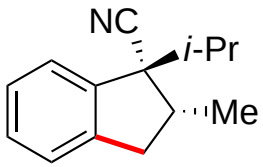
X = OTf, EWG = Tf
e. r. up to 98:2
Cramer et al.
AChIE **2012**, 2238.

racemic version: Ohno et al., *Org Lett* **2008**, 1759.

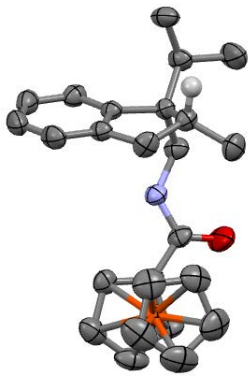
Diastereo and enantioselective C(sp^3)–H arylation

desymmetrization of methyl groups

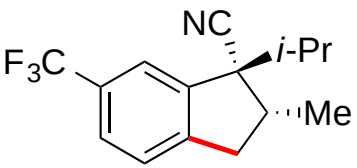




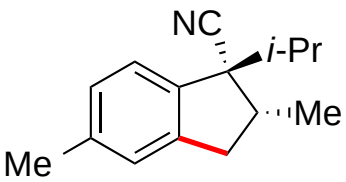
91% **(g scale)**
d.r. 89:1
e.r. 96:4
(90 °C)



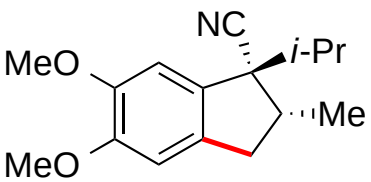
(absolute structure)



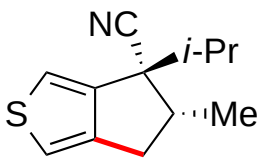
83%
d.r. 99:1
e.r. 95:5
(90 °C)



77%
d.r. 99:1
e.r. 92:8
(120 °C)



78%
d.r. 63:1
e.r. 94:6
(140 °C)

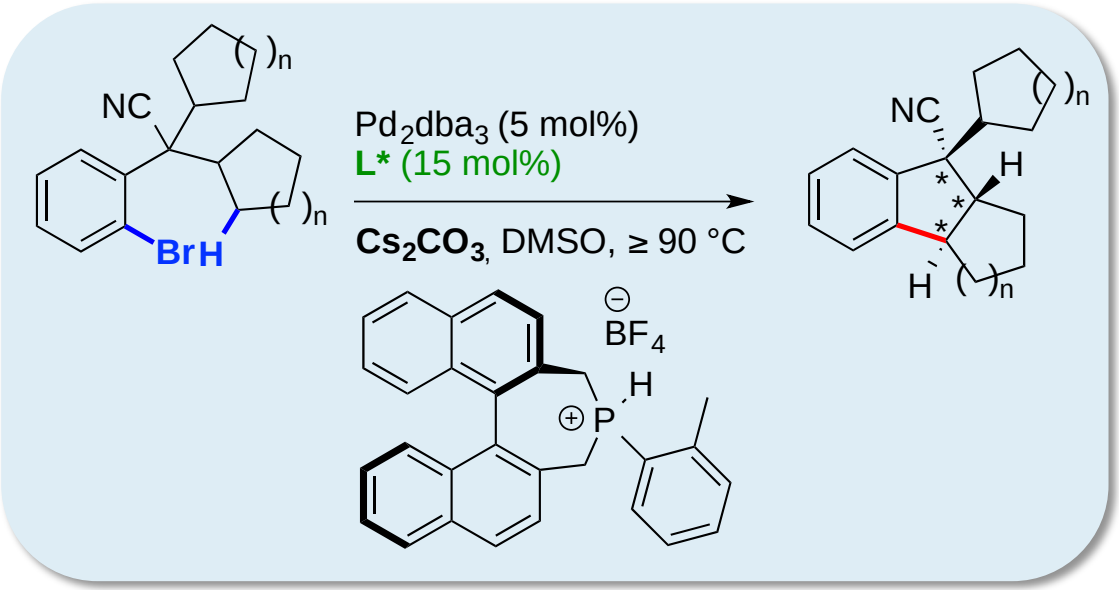


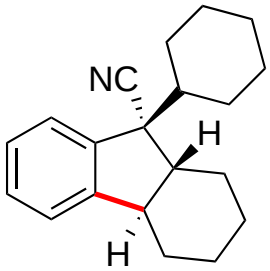
73%
d.r. 64:1
e.r. 92:8
(140 °C)

with N. Martin & C. Pierre, *Chem Eur J* **2012**, 4480; with P. Holstein, *ACS Catal* **2015**, 4300.
ferrocene derivatization method: P. and J. Holstein, *Tet Asymm* **2017**, 1321.
review on binepines: Gladiali & Beller, *Chem Soc Rev* **2011**, 3744.

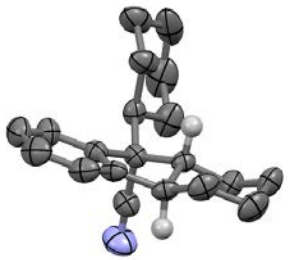
Diastereo and enantioselective C(sp^3)–H arylation

desymmetrization of cycloalkyl groups

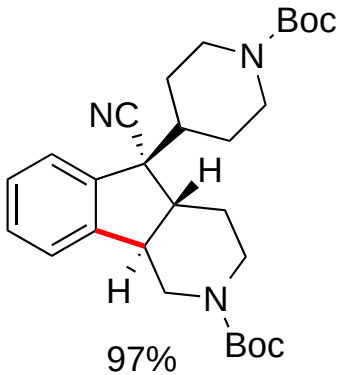




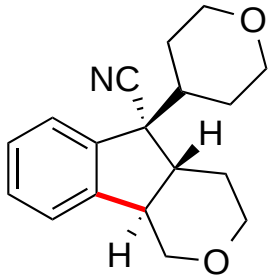
80%
d.r. >98:2
e.r. 98:2
(90 °C)



(relative structure)



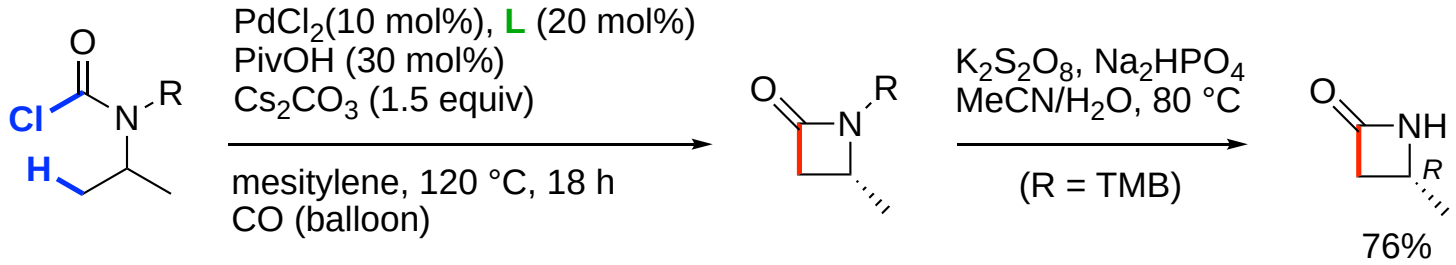
97%
d.r. >98:2
e.r. 98:2
(100 °C)

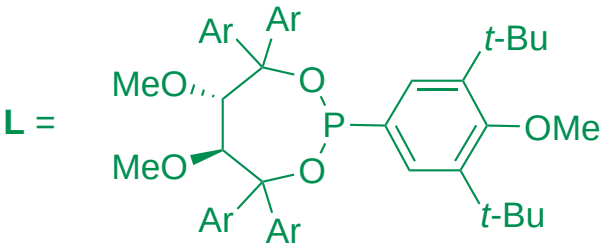


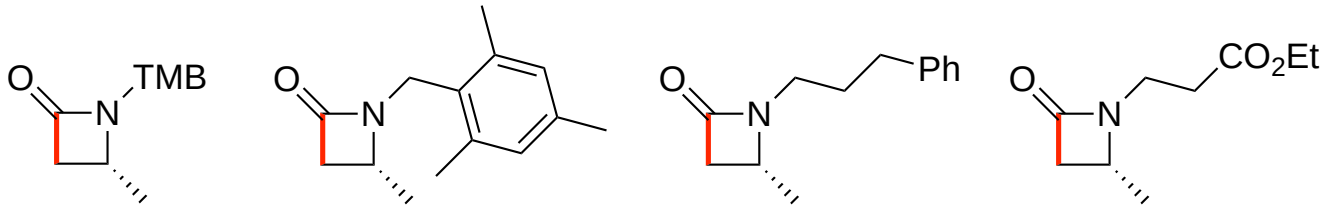
80%
d.r. >98:2
e.r. 95:5
(100 °C)

Enantioselective synthesis of β-lactams

desymmetrization of methyl groups







75%
e.r. 92:8

74%
e.r. 85:15

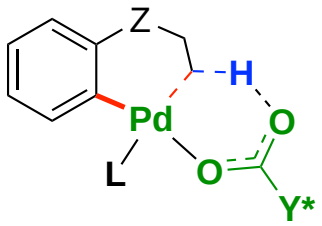
74%
e.r. 84:16

50%
e.r. 82:18

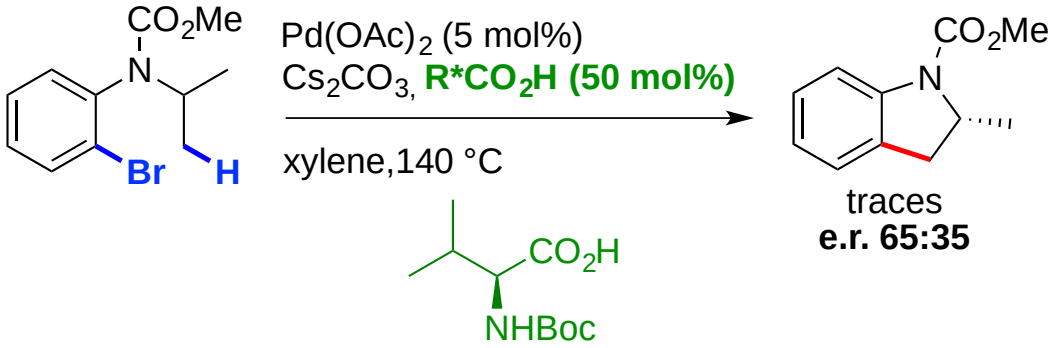
with D. Dailler & R. Rocaboy, *AChIE* **2017**, 7218.
chiral phosphonites: Cramer *et al.*, *AChIE* **2015**, 11826.

TMB = 2,4,6-trimethoxybenzyl

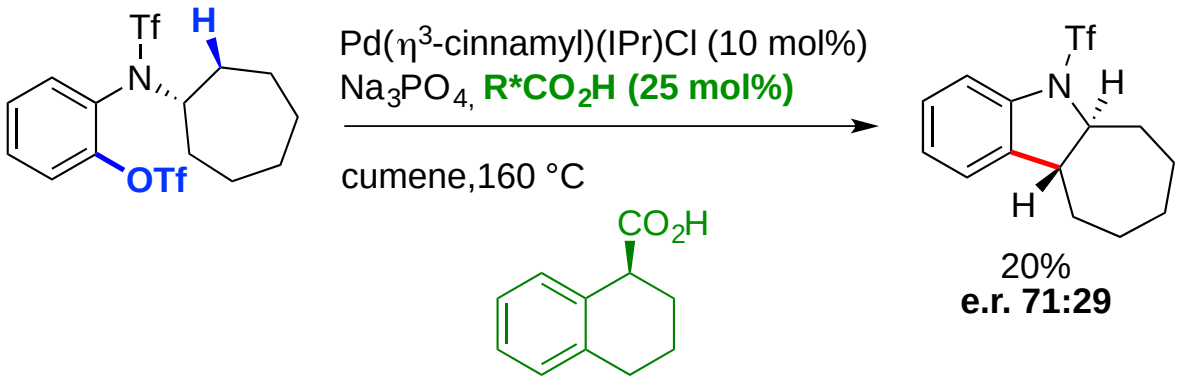
Enantioselective C(sp^3)–H arylation with a chiral base/anion



precedents (Pd^0 cat.)



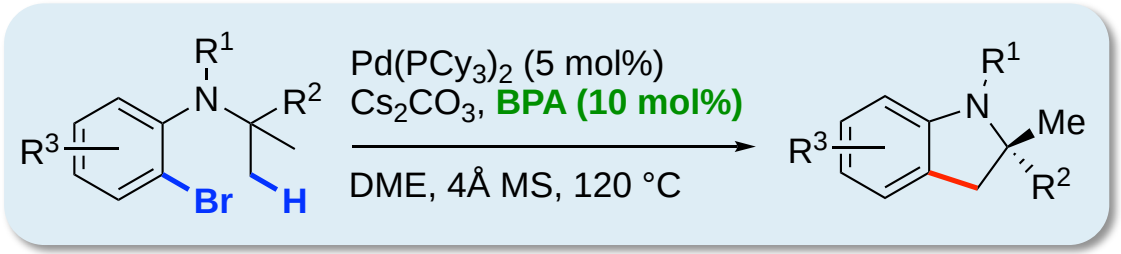
Kagan et al., *Chem Commun* **2011**, 11483.

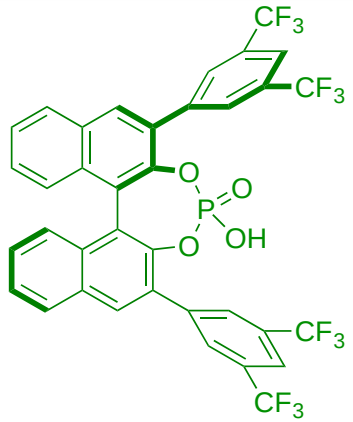


Cramer et al., *AChIE* **2012**, 2238.

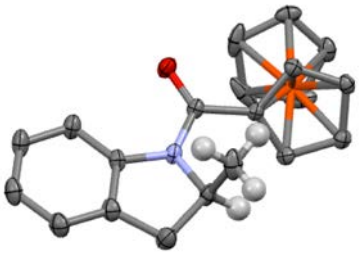
chiral mono-protected aminoacids (MPAA) in Pd^{II}-cat. enantioselective C–H activation: Yu, *AChIE* **2008**, 4882; *JOC* **2013**, 8927.

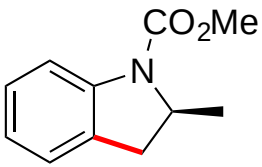
Enantioselective C(sp^3)–H arylation with a chiral phosphate



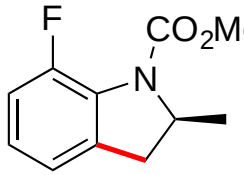


indolines with trisubstituted stereocenter

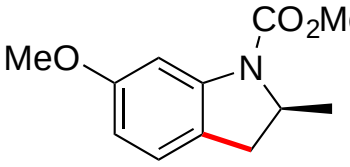




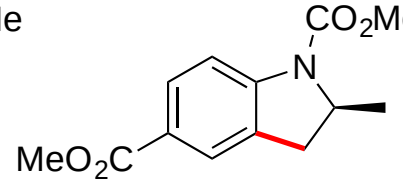
86%
e.r. 96:4
(g scale)



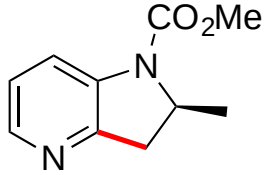
79%
e.r. 96:4



83%
e.r. 96:4

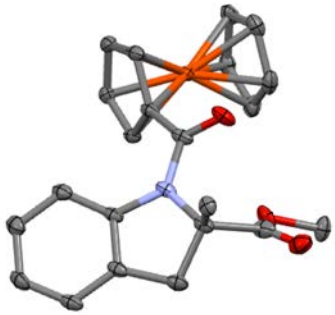


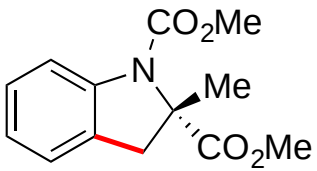
80%
e.r. 94:6



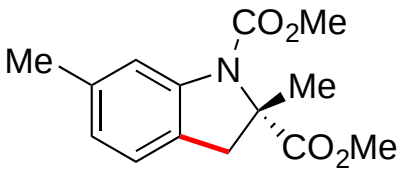
67%
e.r. 94:6

indolines with tetrasubstituted stereocenter

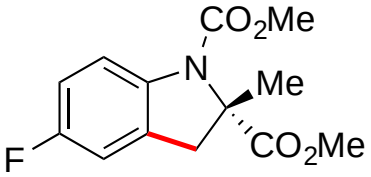




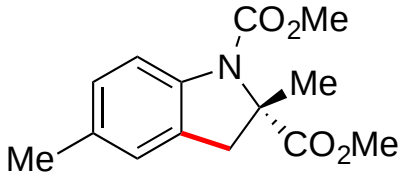
83%
e.r. 92:8



50%
e.r. 94:6



77%
e.r. 94:6

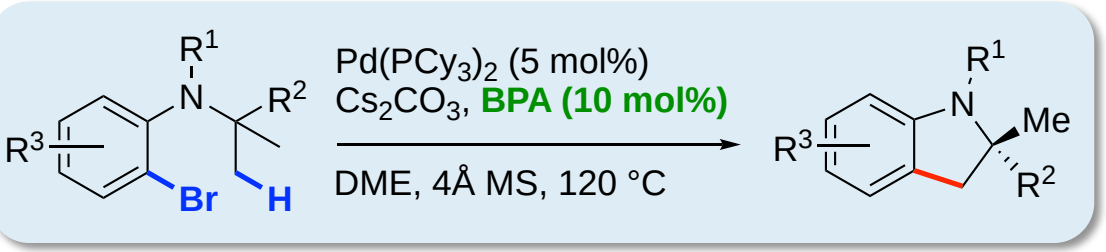


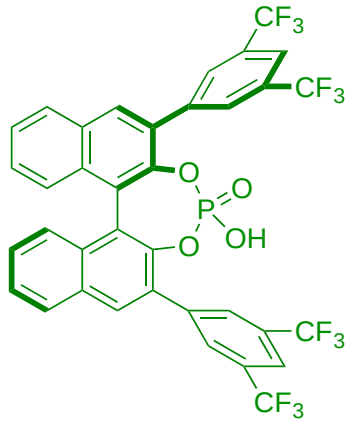
59%
e.r. 93:7

with L. Yang & R. Melot, *Chem Sci* **2017**, 1344.

Pd^{II}-cat. C(sp^3)–H activation using Binol-derived phosphates: Duan (*OrgLett* **2015**), He & Chen (*AChIE* **2016**), Yu *(NatChem* **2017**), Gaunt (*JACS* **2017**), Shi (*AChIE* **2018**).

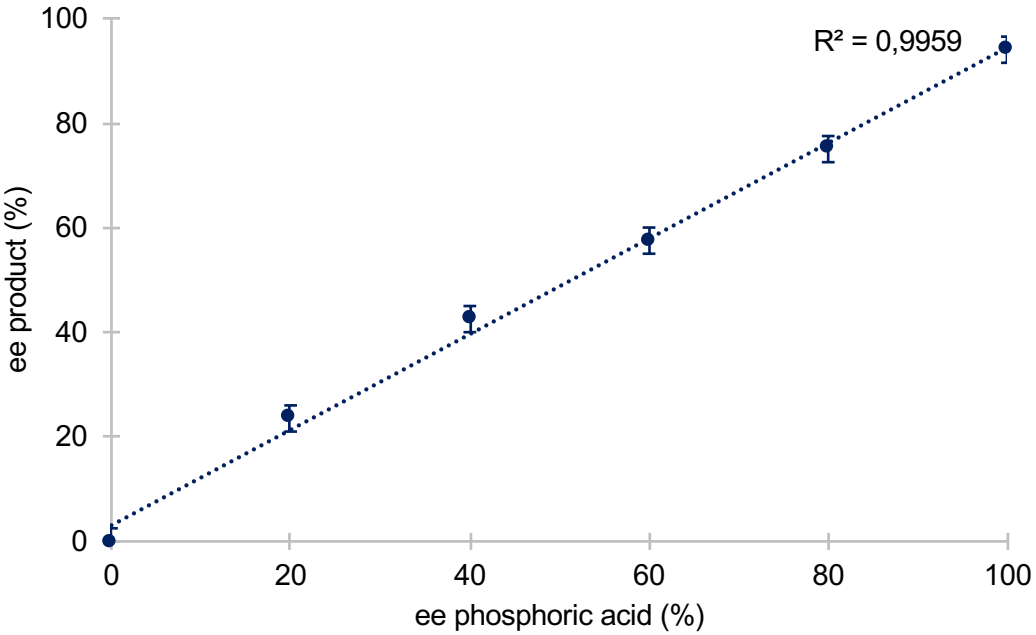
Enantioselective C(sp^3)–H arylation with a chiral phosphate

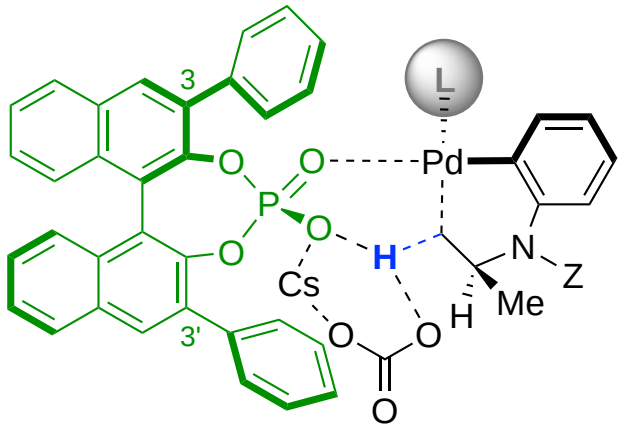




involvement of a phosphate•carbonate complex

- no reaction with stoichiometric cesium phosphate & without Cs_2CO_3
- slow background reaction without phosphoric acid
- Cs^+ is required (racemic product with K^+, Rb^+)
- linear effect (e.e. product/e.e. phosphoric acid)

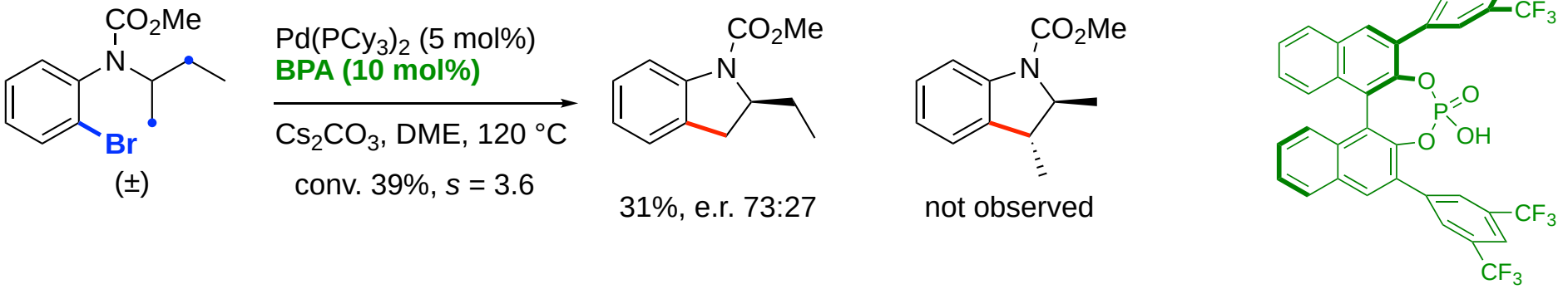




role of Cs clusters: Yu, Musaev *et al.*, *JACS* **2013**, 14206.

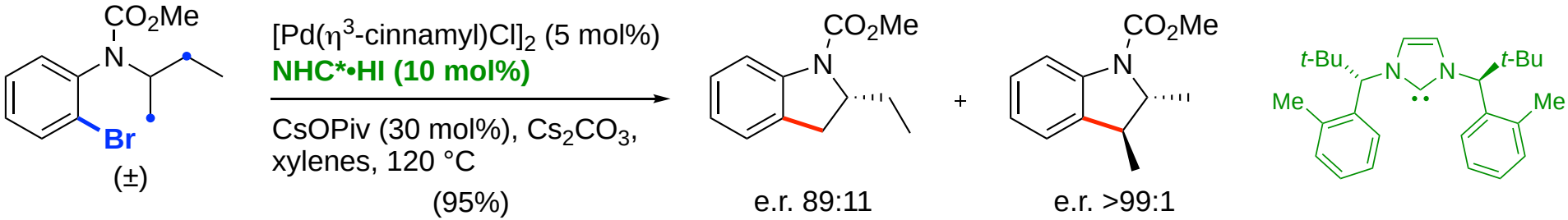
Enantioselective C(sp^3)–H arylation with a chiral phosphate

classic kinetic resolution



with L. Yang & R. Melot, *Chem Sci* **2017**, 1344.

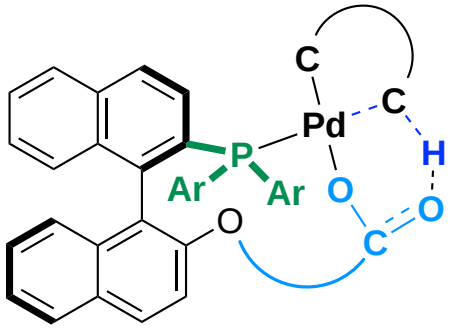
... vs. parallel kinetic resolution



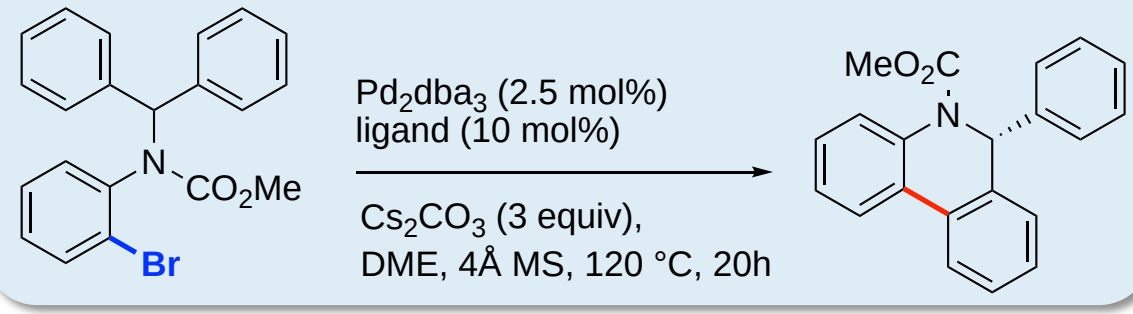
Kündig et al., *Chem Sci* **2012**, 1422.

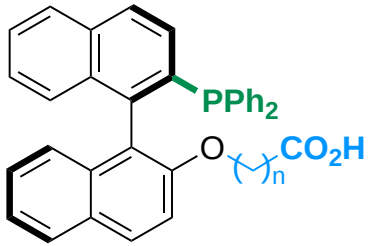
Enantioselective C(sp^2)–H arylation with a bifunctional ligand

a third way: bifunctional ligands

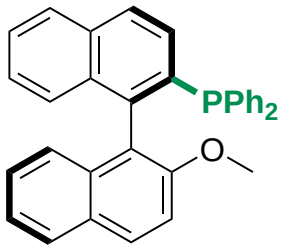


synthesis of 5,6-dihydrophenanthridines

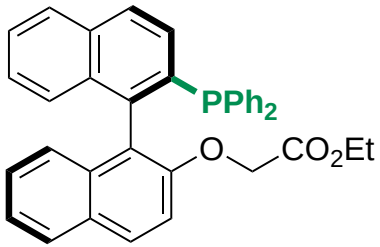




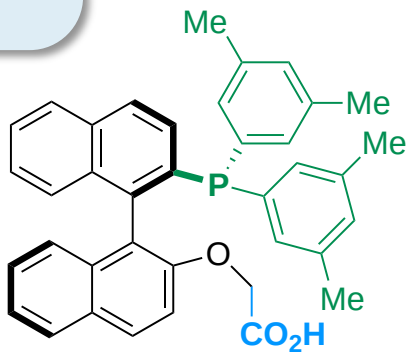
n = 1: 91%, e.r. 93.5:6.5
n = 2: 95%, e.r. 75:25
...
n = 5: 83%, e.r. 46:54



(MOP) +10 mol% PivOH
96%, e.r. 63.5:36.5



+10 mol% PivOH
87%, e.r. 58.5:41.5

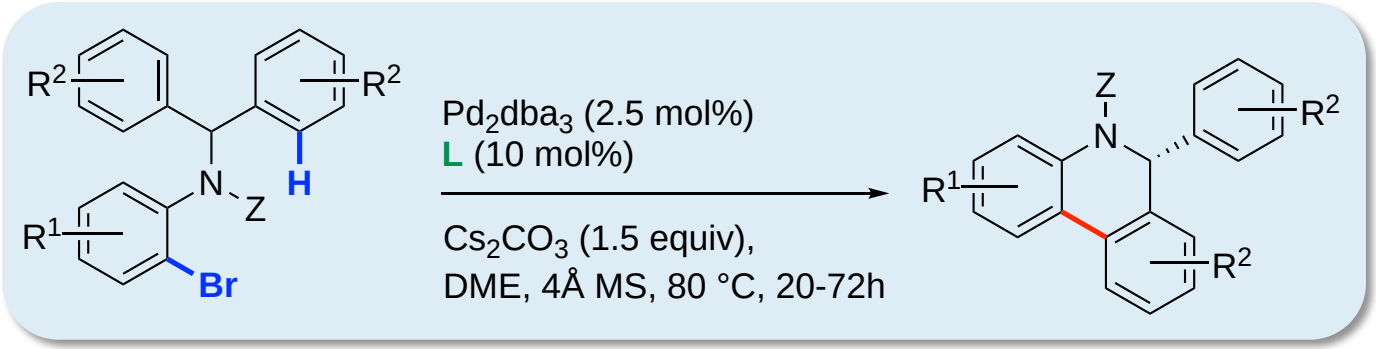


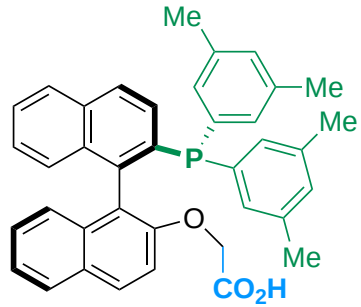
92%, e.r. 97.5:2.5
stoich. K^+ salt (no carbonate):
92%, e.r. 95.5:4.5

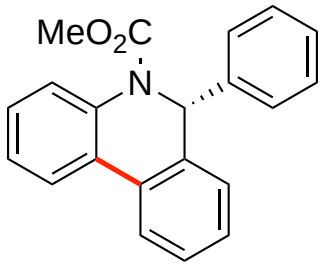
water-soluble MOPs: Uozumi, Hayashi *et al.*, *Tet Lett* **1998**, 8303.

Enantioselective C(sp^2)–H arylation with a bifunctional ligand

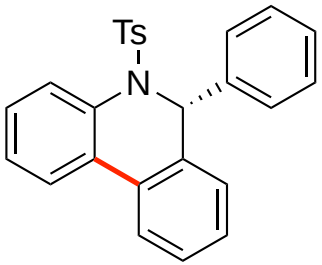
synthesis of 5,6-dihydrophenanthridines



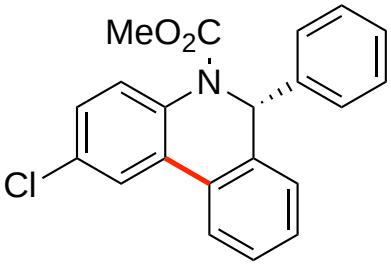




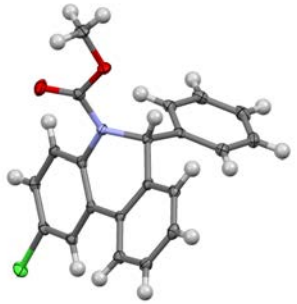
92%
e.r. 98.5:1.5

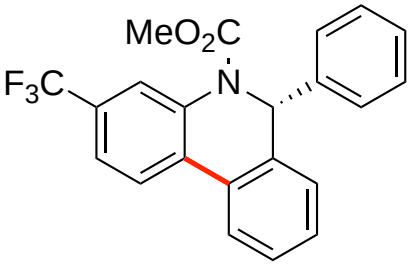


88%
e.r. 93:7

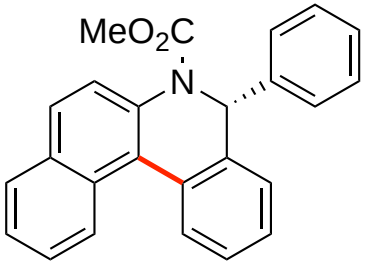


92%
e.r. 96.5:3.5

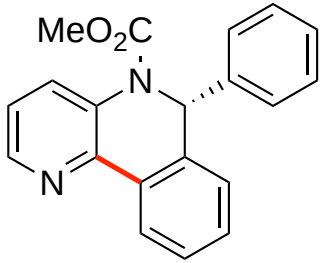




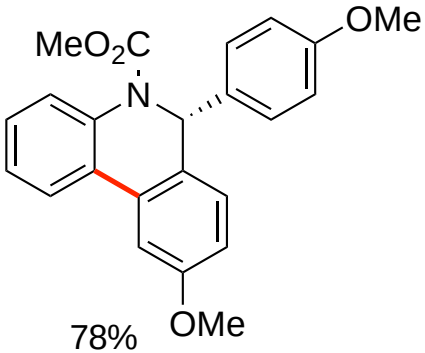
90%
e.r. 98.5:1.5



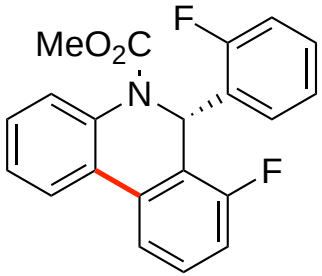
93%
e.r. 98:2



73%
e.r. 96:4



78%
e.r. 98.5:1.5

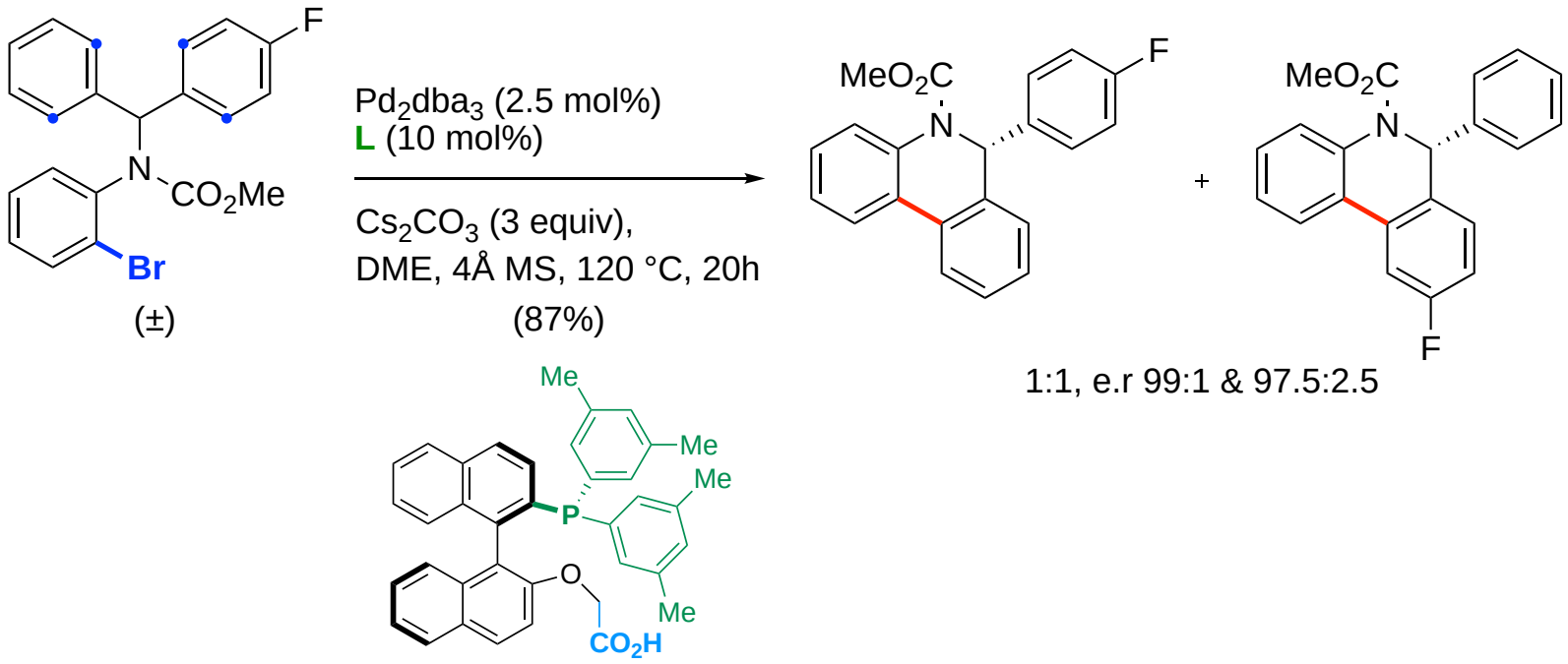


78%
e.r. 98.5:1.5

with L. Yang, *AChIE* **2018**, 1394.

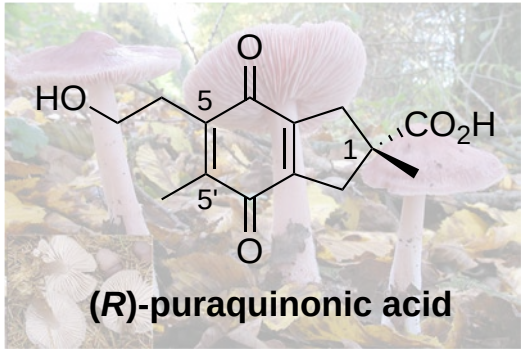
Enantioselective C(sp^2)–H arylation with a bifunctional ligand

parallel kinetic resolution



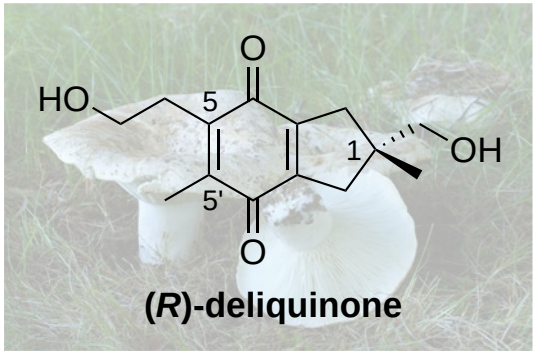
with L. Yang, *AChIE* **2018**, 1394; see also Kündig *et al.*, *Chem Sci* **2012**, 1422; Cramer *et al.*, *ACS Catal.* **2017**, 7417.

Divergent synthesis of illudalane (nor)sesquiterpenes

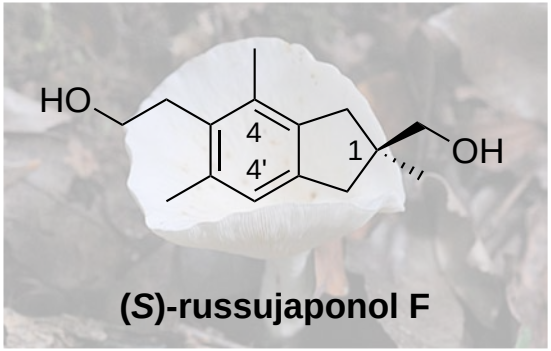


(*R*)-puraquinonic acid

(inducer of HL-60 differentiation)



(*R*)-deliquinone



(*S*)-russujaponol F

key structural aspects

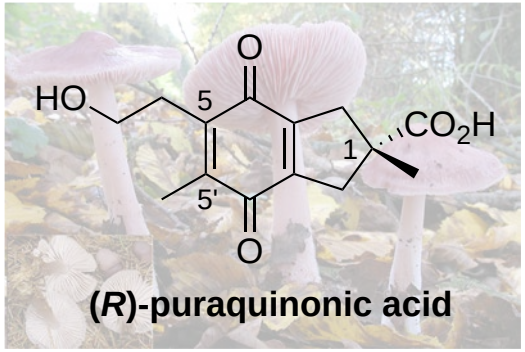
- highly symmetric quat stereocenter
- polyfunctionalized, sensitive benzoquinone

two enantioselective syntheses:

- (*S*)-puraquinonic acid: Clive, **2002**, 31 steps, Evans aldol reaction
- (*R*)-puraquinonic acid: Gleason, **2013**, 12-15 steps, 14-20% overall, asymmetric alkylation (chiral auxiliary)

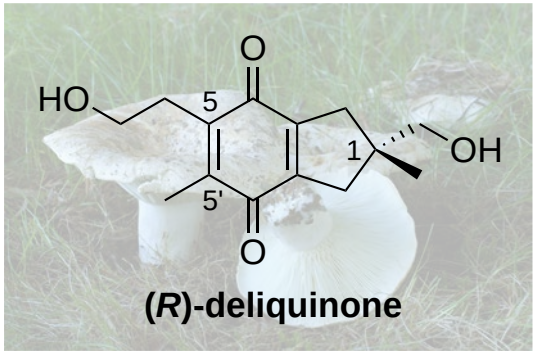
Clive *et al.*, *ChemComm* **2002**, 2380; Gleason *et al.*, *AChIE* **2013**, 3442.
racemic syntheses: puraquinonic acid: Clive, *TetLett* **2001**, 2253 (15 steps); deliquinone: Kraus, *JOC* **2002**, 5857 (10 steps).

Divergent synthesis of illudalane (nor)sesquiterpenes

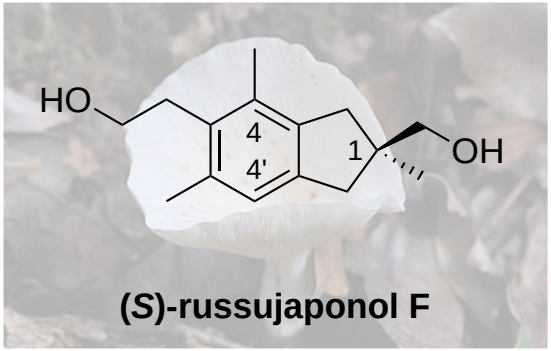


(*R*)-puraquinonic acid

(inducer of HL-60 differentiation)

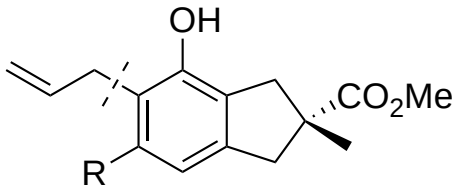


(*R*)-deliquinone



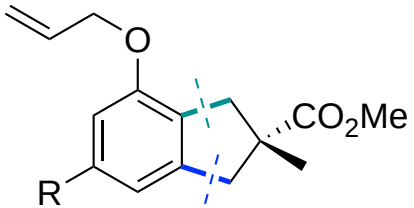
(*S*)-russujaponol F

(*R*)-illudalanes ⟹

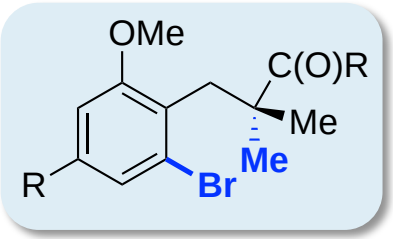


common intermediate
(R = H, Me)

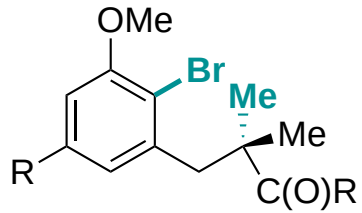
[3,3] ⟹



⇓ ***asymmetric C(sp³)–H activation***

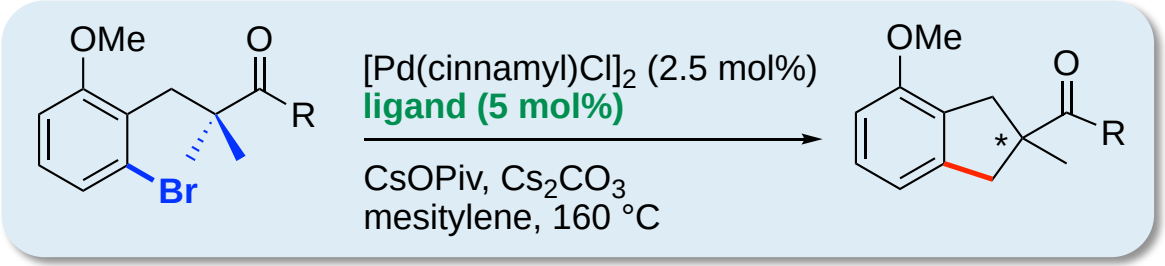


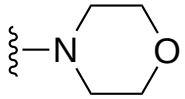
or



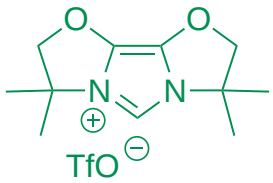
Divergent synthesis of illudalane (nor)sesquiterpenes

study of the asymmetric C(sp^3)–H activation step

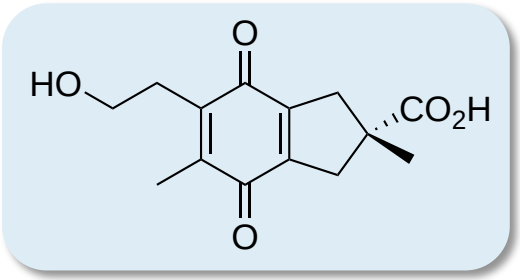


R	ligand	e. r./d. r.	yield (%)
OMe	**NHC***	66:34	92
	**NHC***	80:20	95
	IBioxMe$_4$		89

NHC*•HI



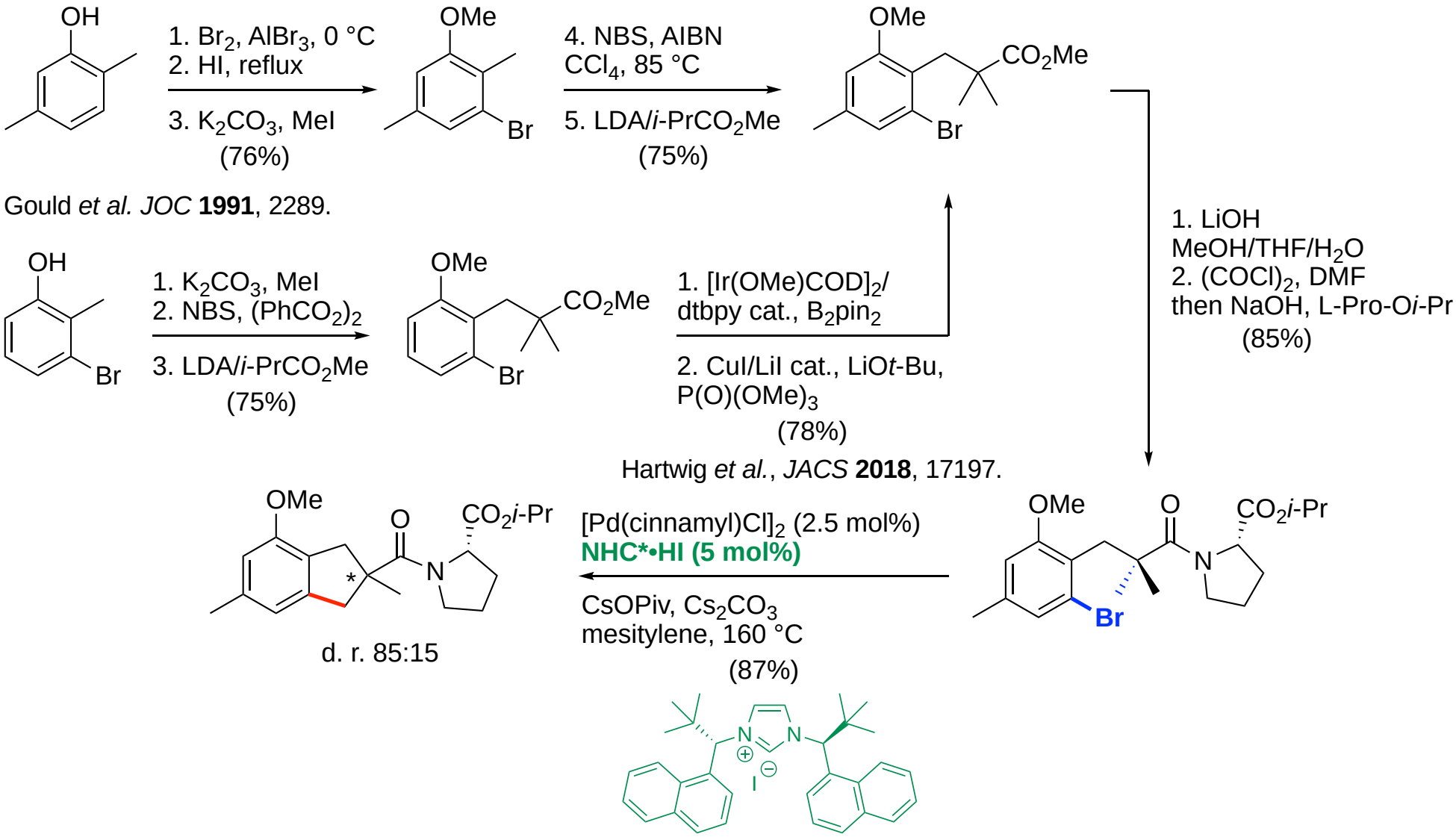
IBioxMe$_4$•HOTf



bulky NHC*: Kündig *et al.*, *AChIE* **2011**, 7438; IBioxMe$_4$: Glorius *et al.*, *JACS* **2004**, 15195.

Divergent synthesis of illudalane (nor)sesquiterpenes

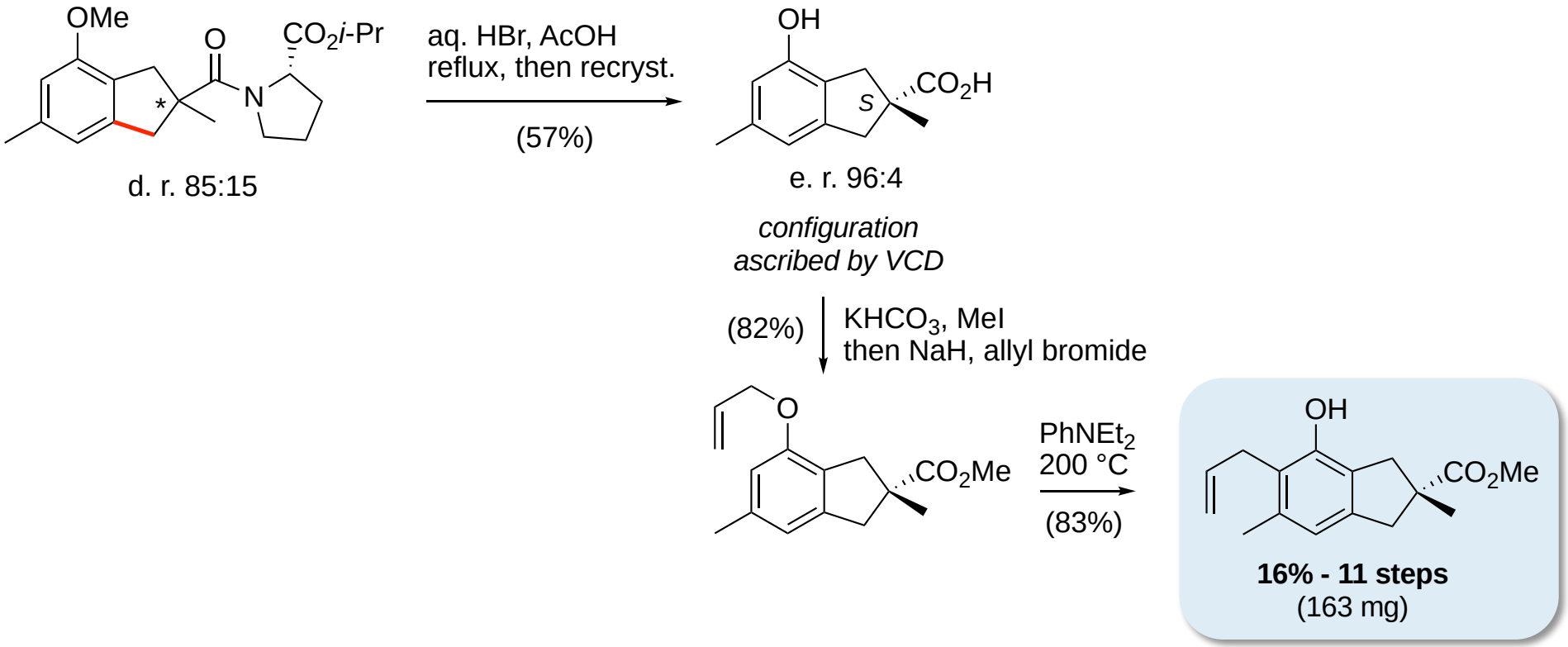
synthesis of the common intermediate



with R. Melot & M. Craveiro, *OrgLett* **2019**, 812 & *JOC* **2019**, ASAP.

Divergent synthesis of illudalane (nor)sesquiterpenes

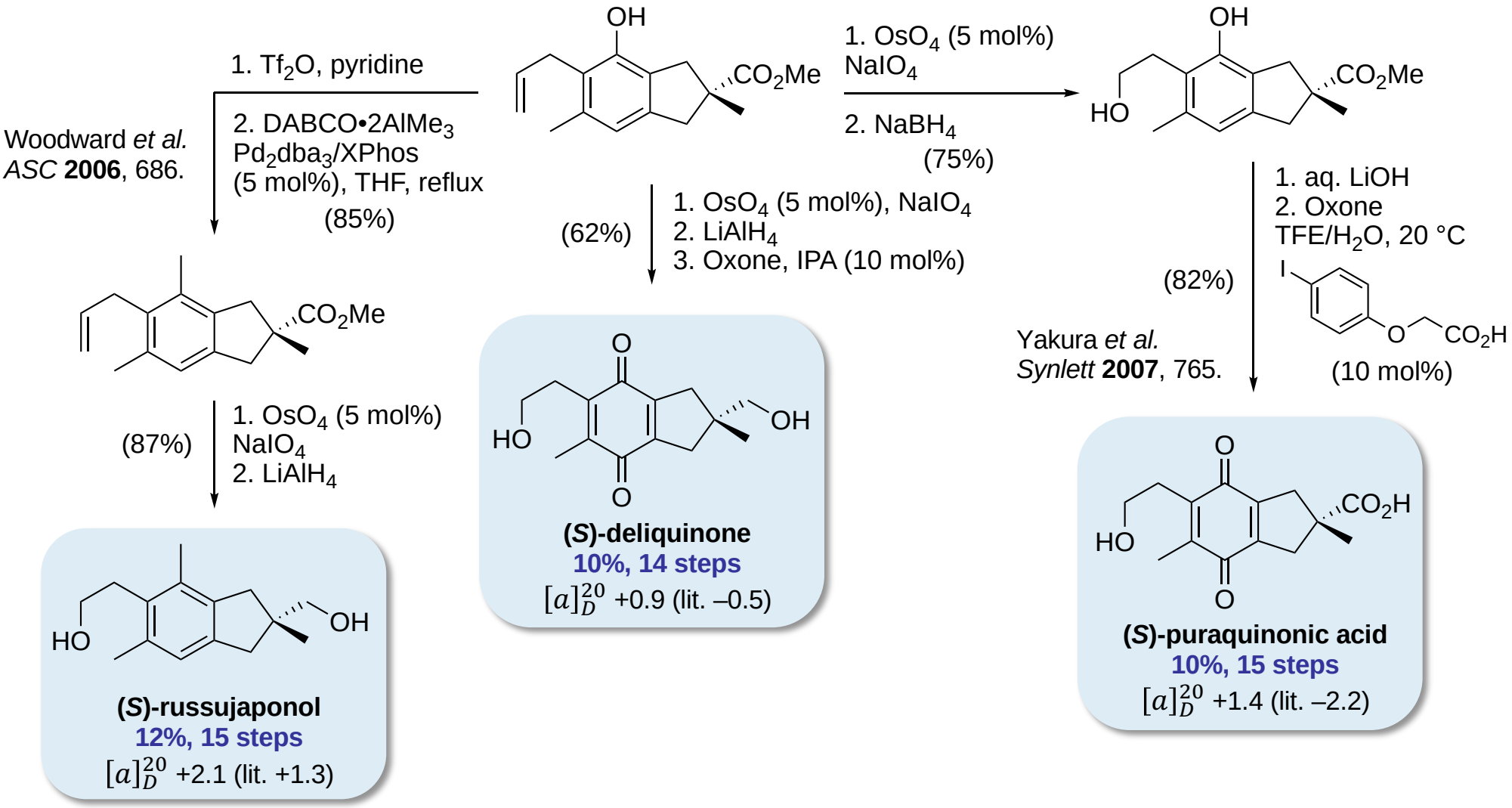
synthesis of the common intermediate



in collaboration with T. Bürgi, Uni Geneva

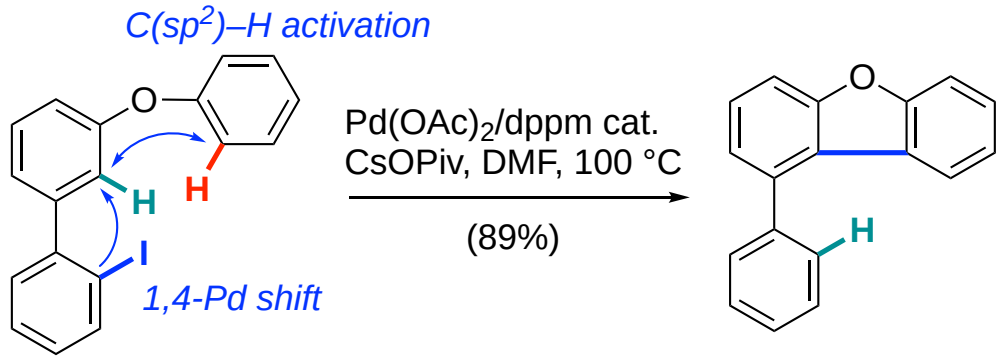
Divergent synthesis of illudalane (nor)sesquiterpenes

divergent synthesis of illudalanes

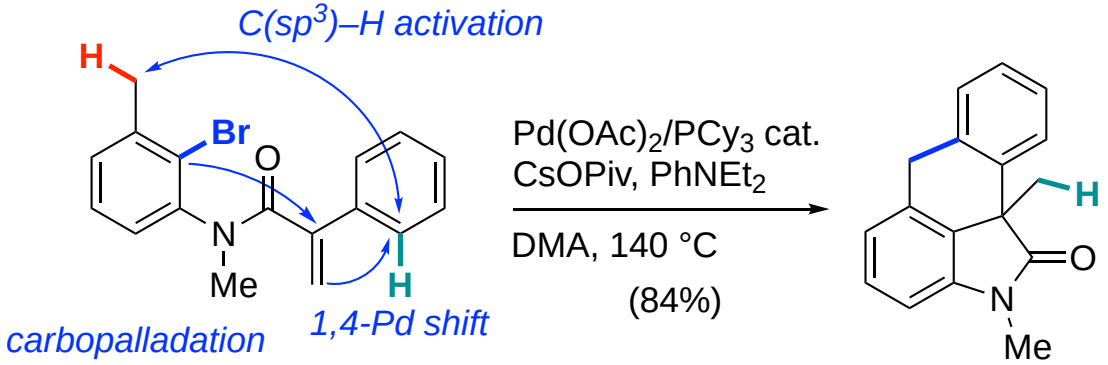


with R. Melot & M. Craveiro, *OrgLett* **2019**, 812 & *JOC* **2019**, ASAP.

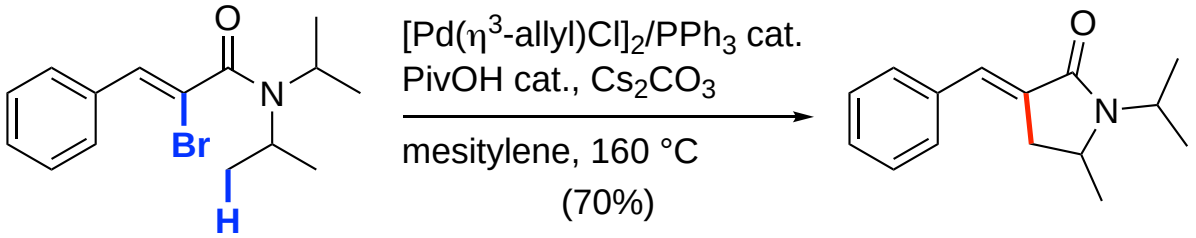
Remote ring construction via 1,4-Pd shift



Larock *et al.*, *JACS* **2003**, 11506.
seminal work: Heck, *J. Organomet. Chem.* **1972**, *37*, 389.

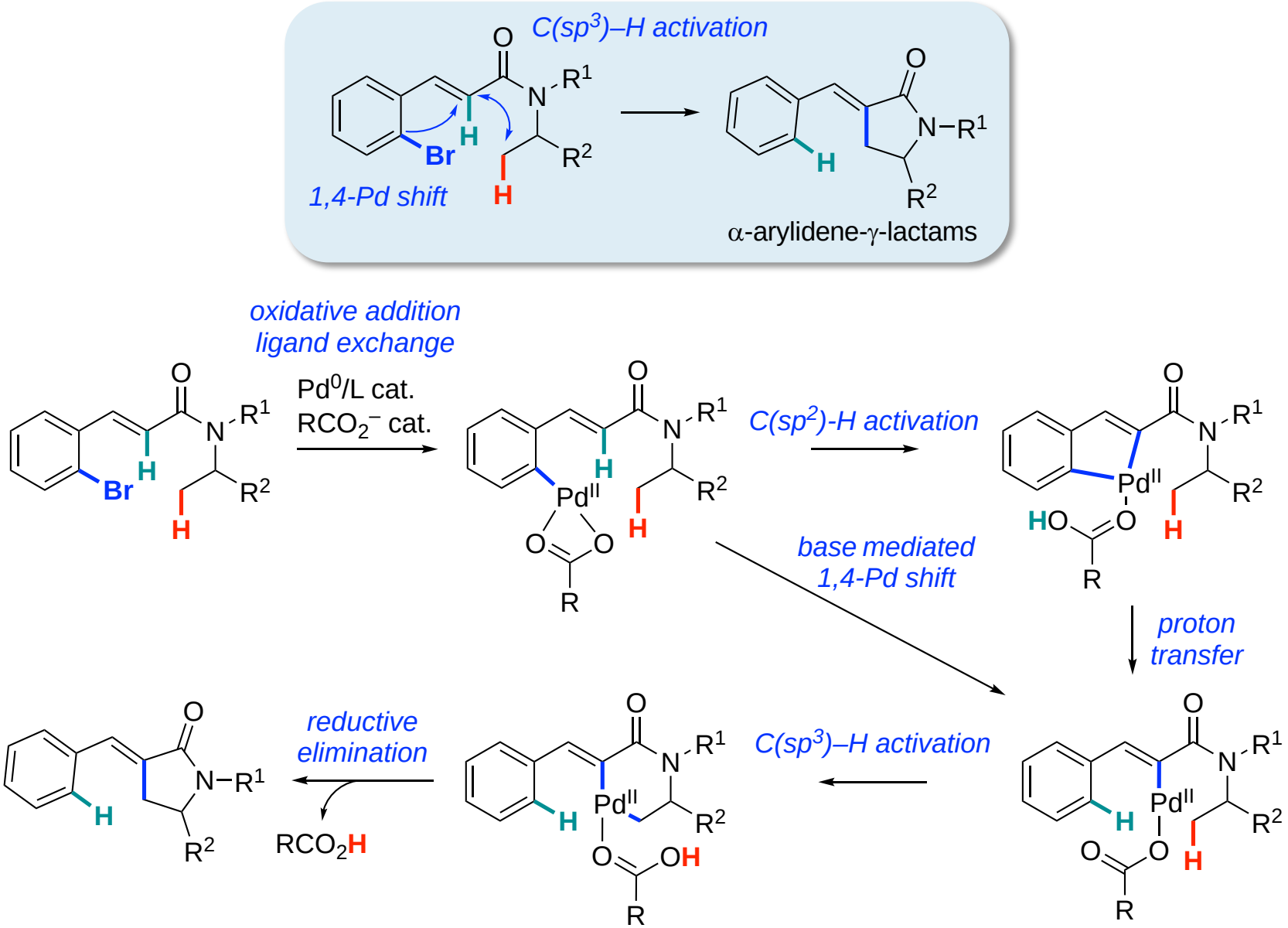


Zhu *et al.*, *AChIE* **2013**, 12385.



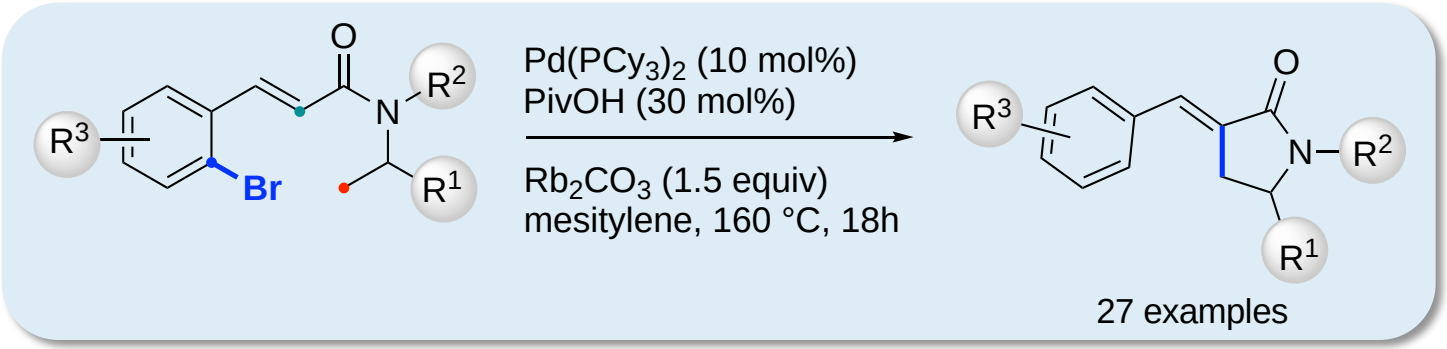
with P. Holstein, D. Dailler, J. Vantourout, *AChIE* **2016**, 2805.

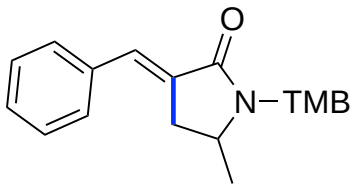
Remote ring construction via 1,4-Pd shift



C(sp^3)–H activation and proton transfer: with E. Clot, *JACS* **2008**, 15157; *JOC* **2014**, 118903.

Remote ring construction via 1,4-Pd shift





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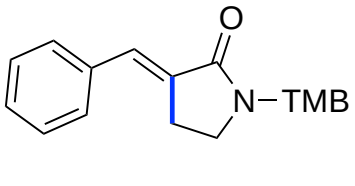

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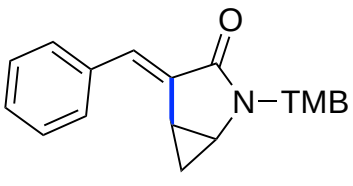

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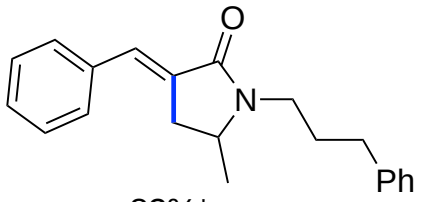

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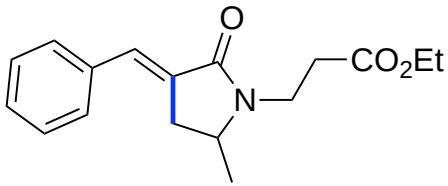

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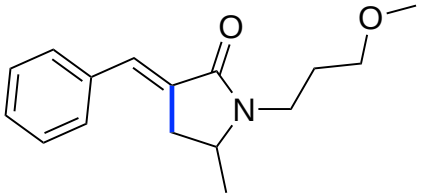

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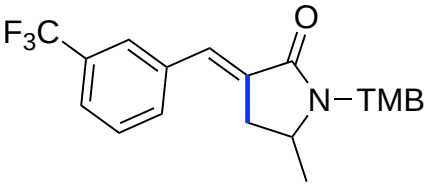

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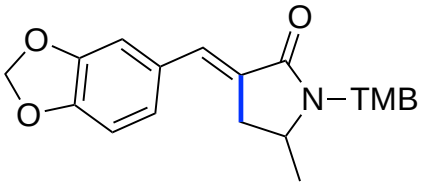

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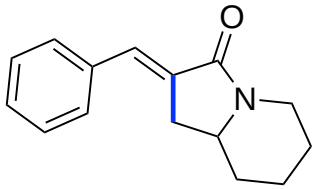

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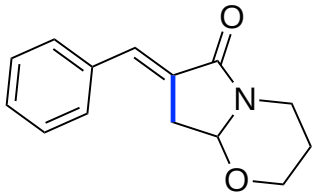

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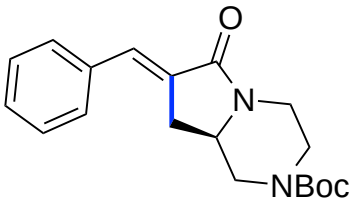

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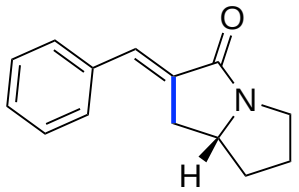

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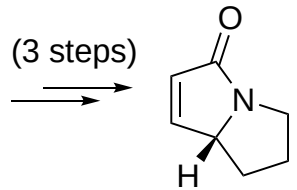
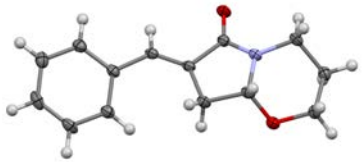


(–)-pyrrolam A

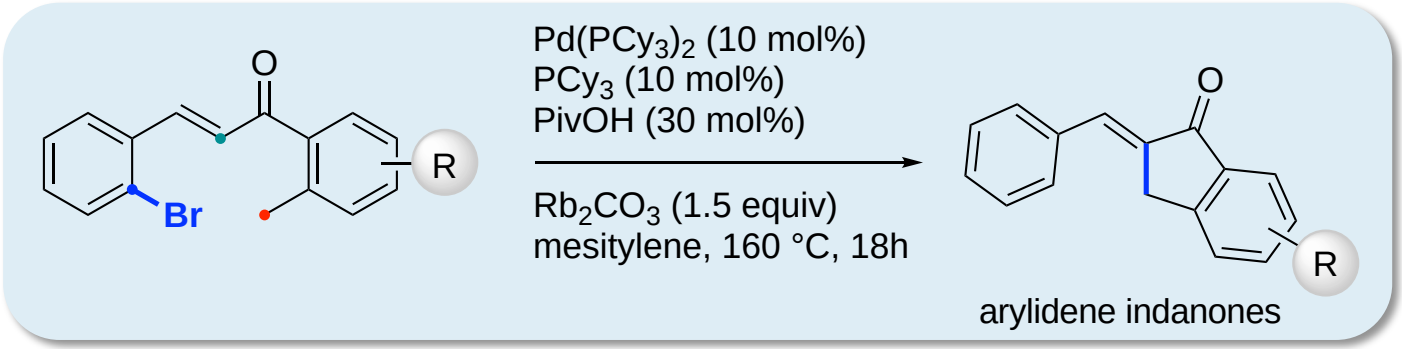


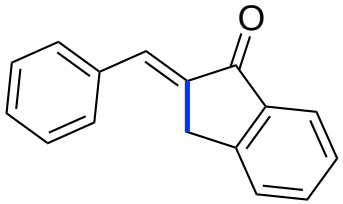
Ohta *et al.*, *Tetrahedron* **1996**, 869.

* with additional 10 mol% PCy_3

with R. Rocaboy, *Org. Lett*. **2019**, 1434.

Remote ring construction via 1,4-Pd shift





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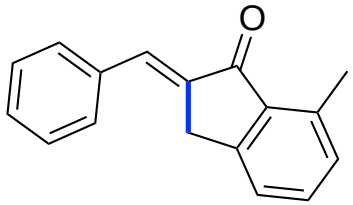

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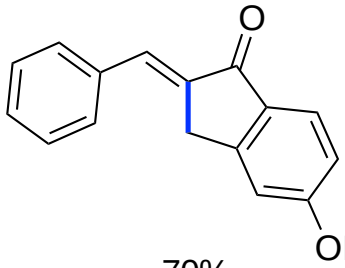

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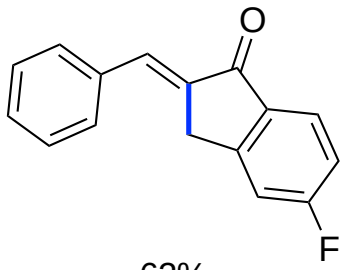

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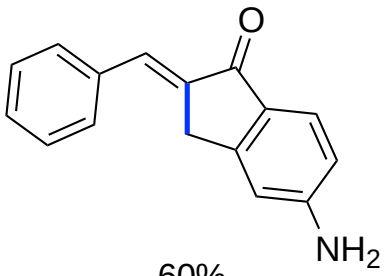

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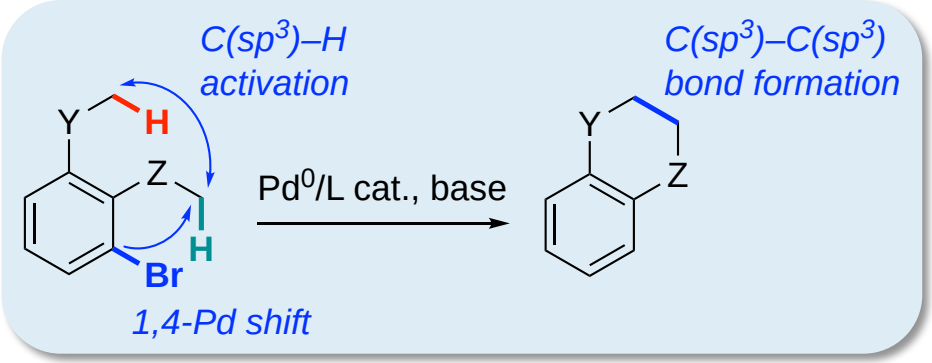
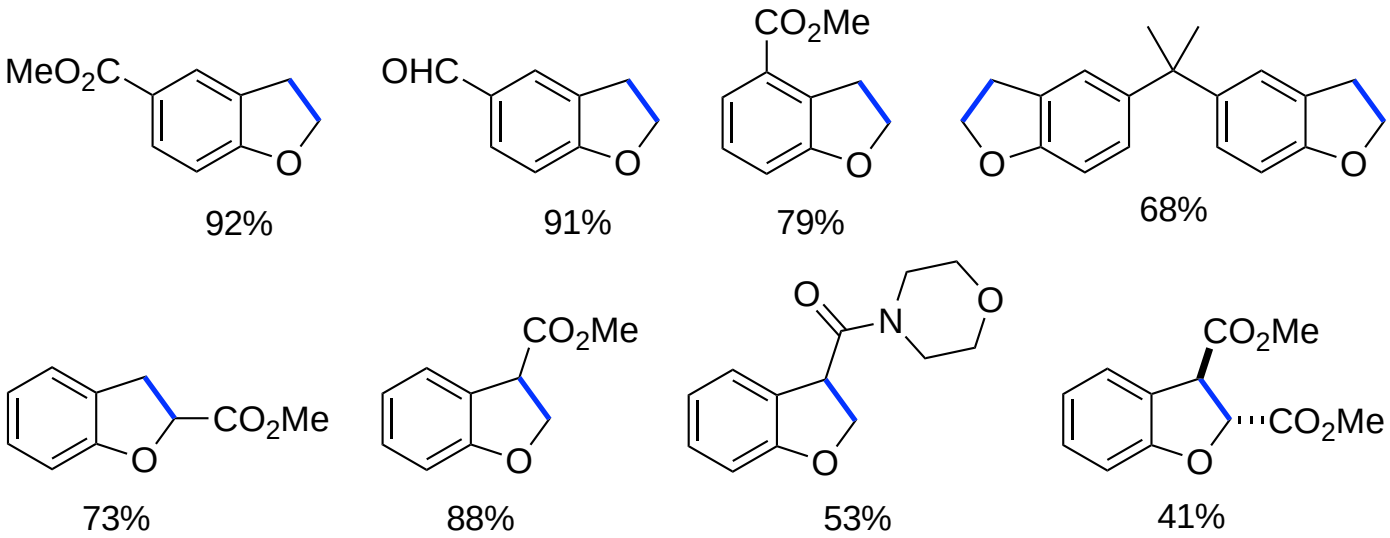
with R. Rocaboy, *Org. Lett*. **2019**, 1434.

Remote ring construction via 1,4-Pd shift



Y = –, Z = O: dihydrobenzofurans

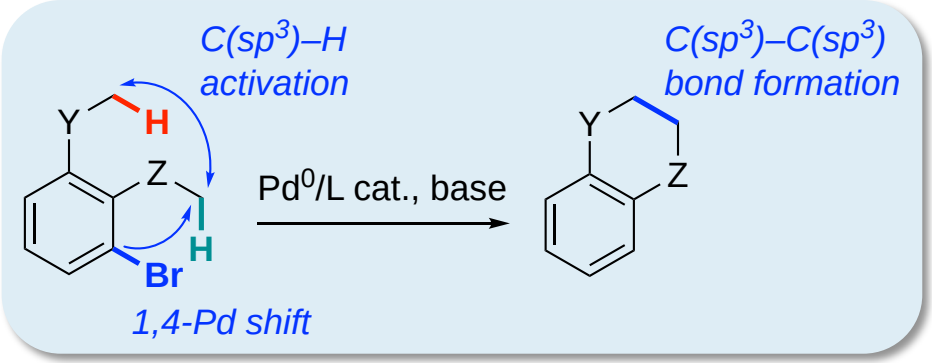


conditions: $Pd(PCy_3)_2$ (10 mol%), CsOPiv (1 equiv), toluene, 140-160 ºC.

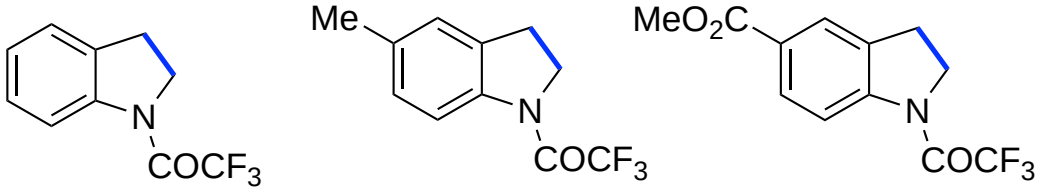
Cross-Dehydrogenative Couplings: C.-J. Li *et al.*, *AChIE* **2014**, 74; A. Lei *et al.*, *ChemRev* **2015**, 12138; seminal work: Dyker, *AChIEE* **1992**, 1023.

with R. Rocaboy, *AChIE* **2019**, ASAP.

Remote ring construction via 1,4-Pd shift

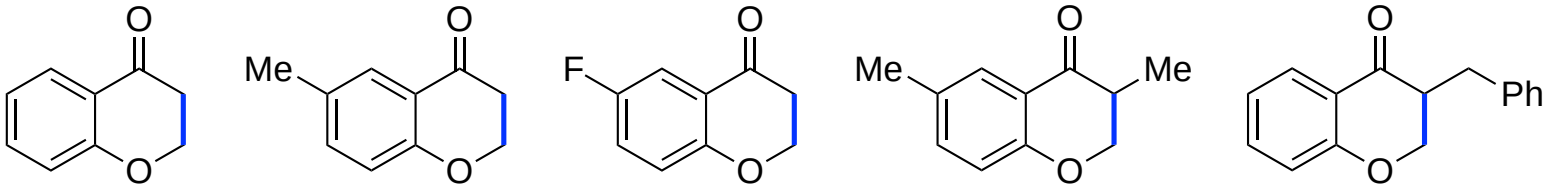


Y = –, Z = N-$COCF_3$: indolines



conditions: $Pd(PCy_3)_2$ (10 mol%), $AdCO_2H$ (30 mol%), Rb_2CO_3, toluene, 160 ºC.

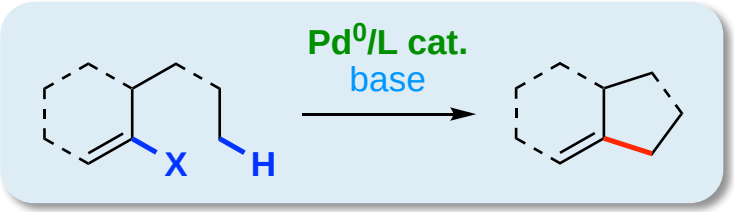
Y = CO, Z = O: chroman-4-ones



conditions: $Pd(PCy_3)_2$ (10 mol%), $AdCO_2H$ (30 mol%), Cs_2CO_3, toluene, 120 ºC.

with R. Rocaboy, *AChIE* **2019**, ASAP.

Conclusion



- Synthesis of a variety of ring systems (C–C bonds) from simple precursors (C–X/C–H bonds) *directly or remotely*
- Remarkable chemo-, site- (1^{ary}, 2^{ary}), stereo- (diastereo/enantio) selectivities
- Selectivity modulation by the ancillary ligand and the base
- Applicability to the synthesis of complex molecules

Special thanks to:

Group members

Philipp Holstein
David Dailler
Ronan Rocaboy
Lei Yang
Romain Melot
Marcus Craveiro

Department of Chemistry

Alessandro Prescimone *(X-ray)*
Daniel Häussinger *(NMR)*

Collaborations

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Christophe Salomé *(Spirochem)*
Thomas Bürgi *(Uni Geneva)*

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Takeru Miyakoshi, Anton Kudashev, Oleksandr Vyhivskyi
Weilong Lin, Antonin Clémenceau, Kevin Antien, Stefania Vergura

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National: Swiss National Science Foundation, State Secretariat for Education, Research and Innovation
Local: University of Basel
International: Marie Sklodowska-Curie Actions (EU), China Scholarship Council (CN), FAPESP (BR)
Industrial: ORIL (FR), Novartis (CH)