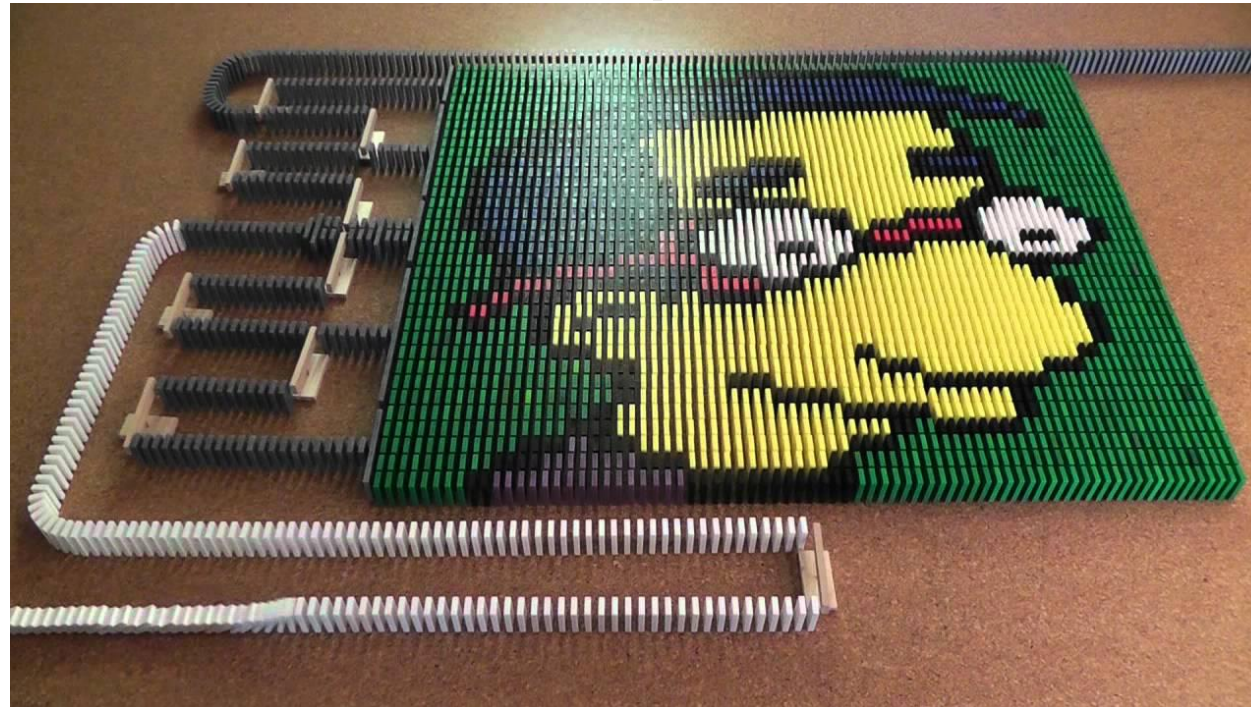


Domino Reactions in Total Synthesis



Dagoneau Dylan
Zhu Group

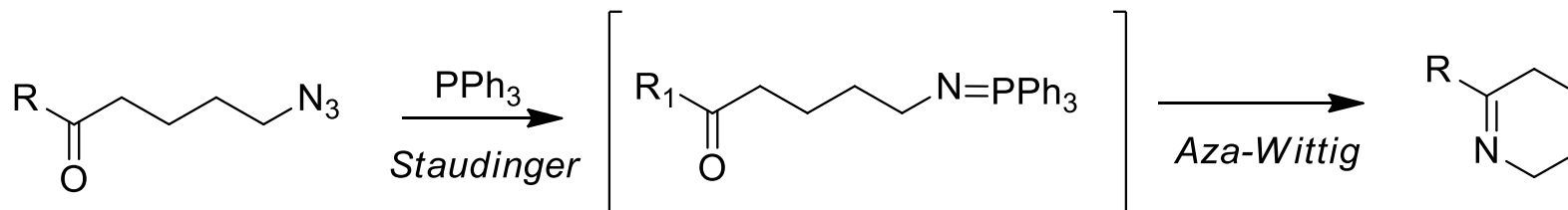
*Group
Seminar*
April 01st, 2015

Table of Contents

- Introduction and Definitions
- I/ Electrophilic Domino Reactions
- II/ Nucleophilic Domino Reactions
- III/ Radical Domino Reactions
- IV/ Pericyclic Domino Reactions
- V/ Transition Metal Catalyzed Domino Reactions
- Conclusion

Introduction and Definitions

- ▶ Domino, Cascade, Sequential and Tandem reactions : 4 different terms for the same idea
- ▶ Definition: A domino reaction is a process involving two or more bond-forming transformations (usually C-C bonds) which take place under the same reaction conditions without adding additional reagents and catalysts, and in which the subsequent reactions result as a consequence of the functionality formed in the previous step. **L. F. Tietze**
- ▶ The « Tandem » term : connotation of **2**



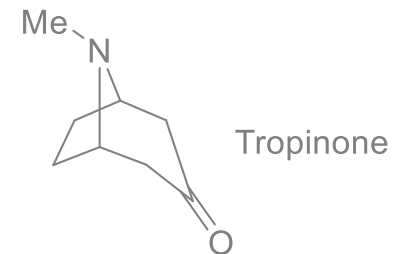
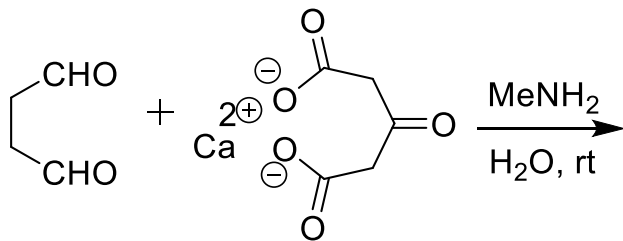
Introduction and Definitions

Why use Domino Reaction?

- One advantage: Economy
- But a lot of different economy : atom, step, time, labor, resource management and waste generation --> « Green chemistry » ?
- Synthesize fused/caged ring and formation of multiple quaternary center in a single reaction.

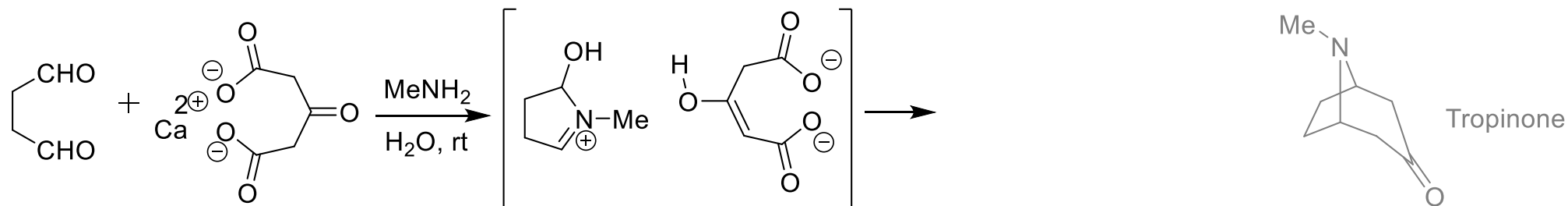
Introduction and Definitions

- First reported « domino » reaction : Robinson's total synthesis of Tropinone (1917)



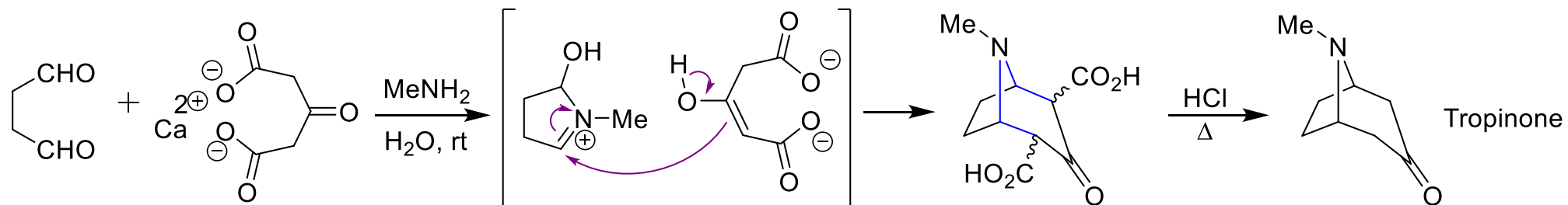
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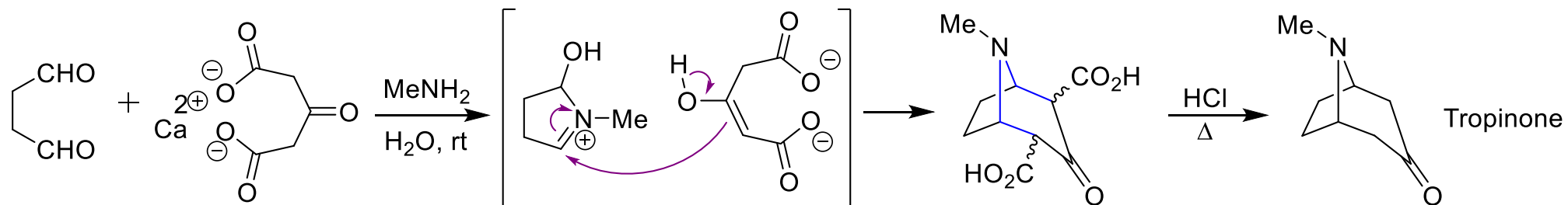
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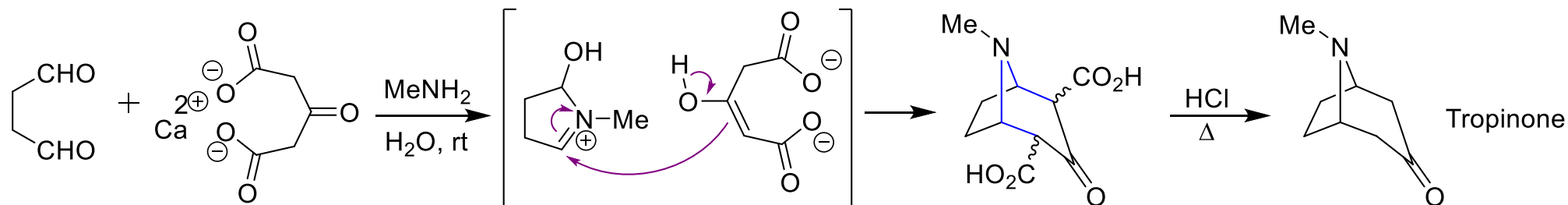


- Johnson's total synthesis of (+/-)-Progesterone by cationic polyolefin cyclization (1971)

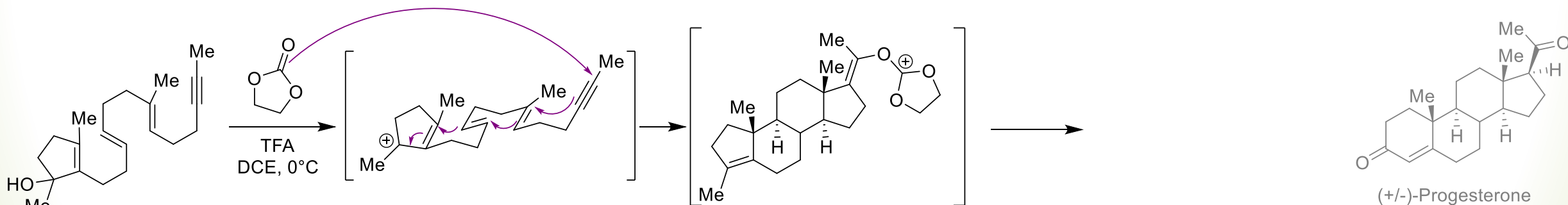


Introduction and Definitions

- First reported « domino » reaction : Robinson's total synthesis of Tropinone (1917)

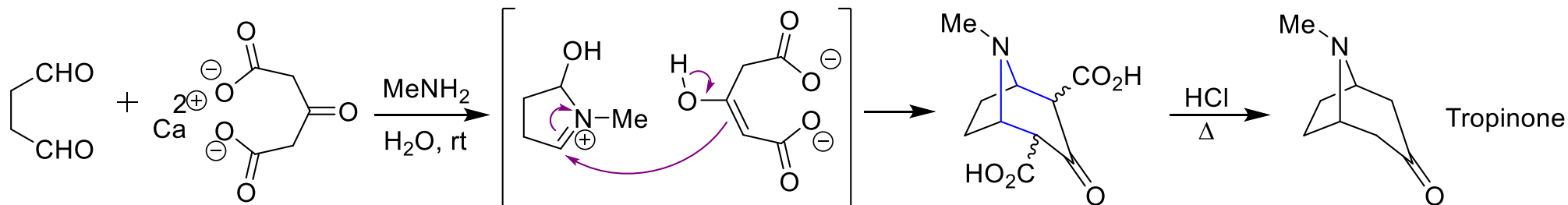


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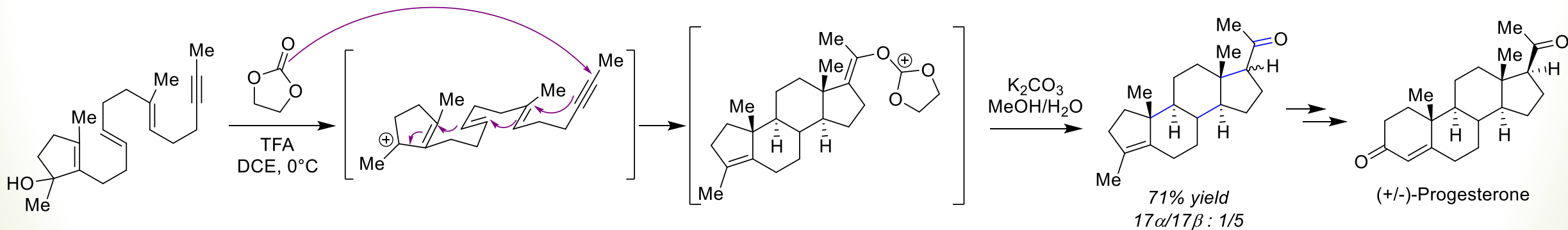


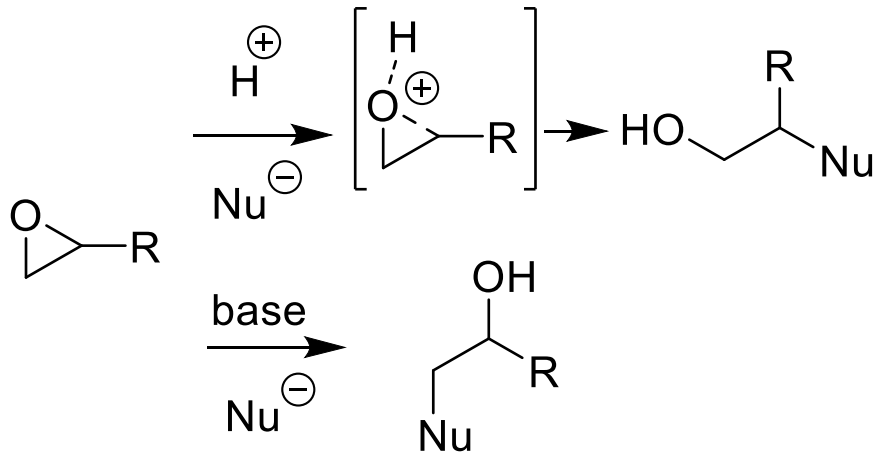
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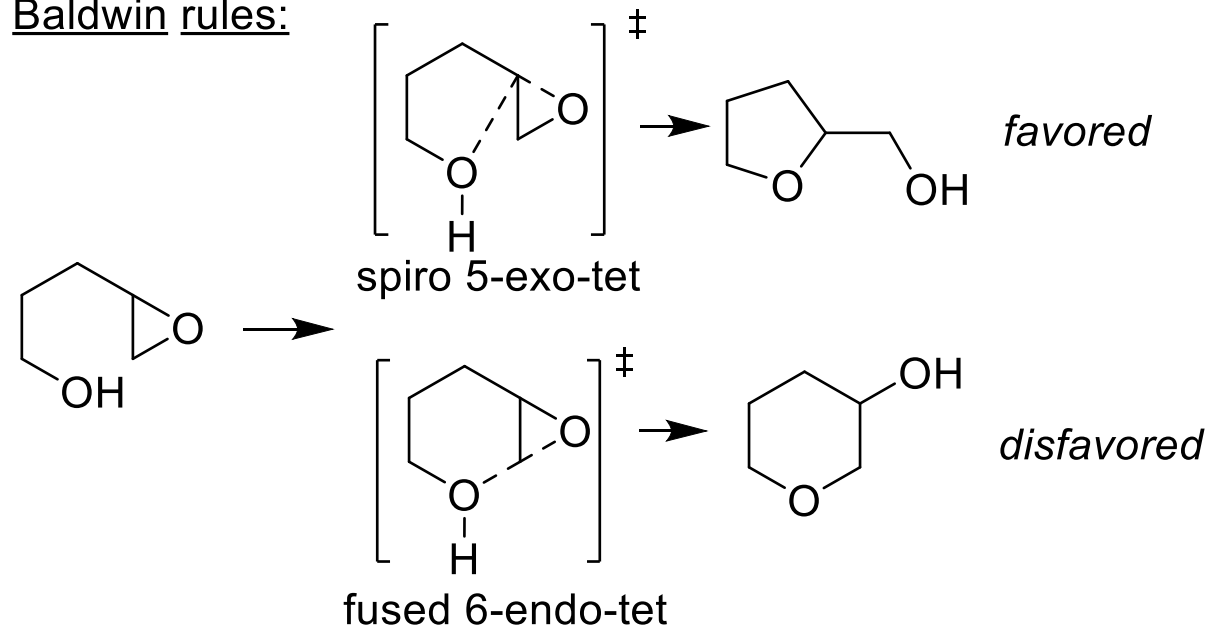
I/ Electrophilic Domino Reactions

I-1/ Epoxide opening

- Predictable opening selectivity



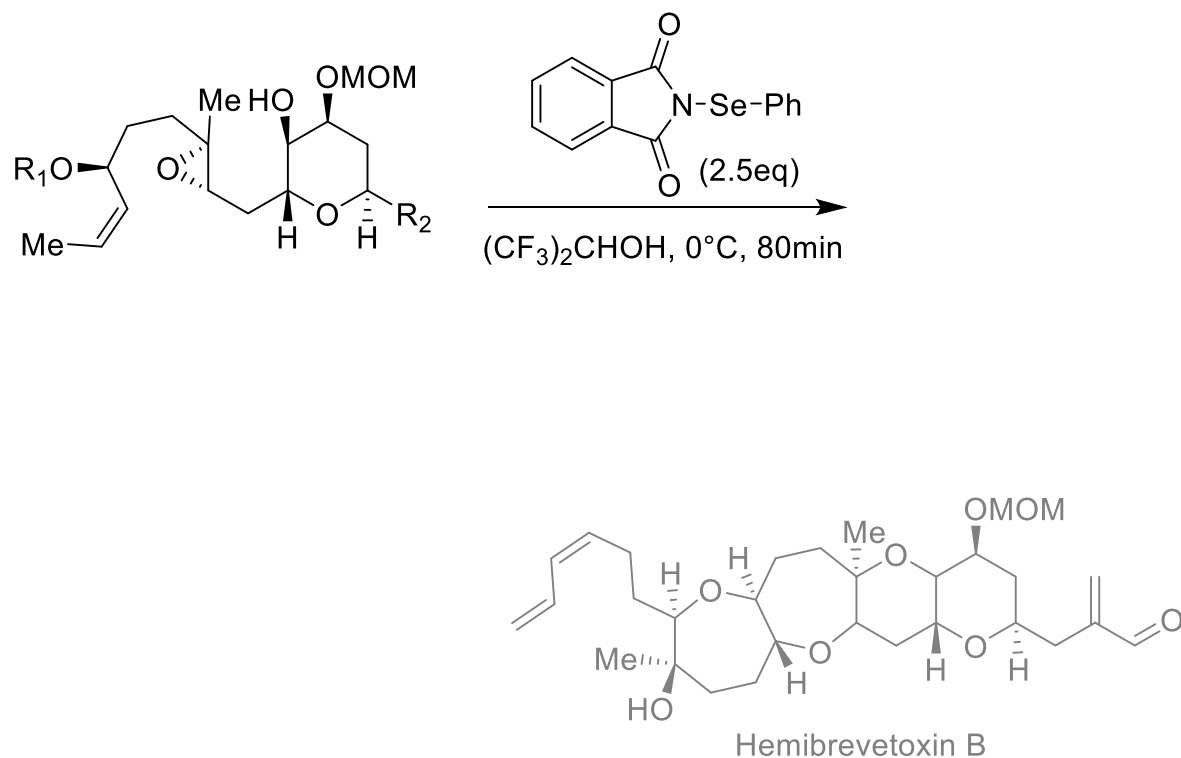
Baldwin rules:



I/ Electrophilic Domino Reactions

I-1/ Epoxide opening

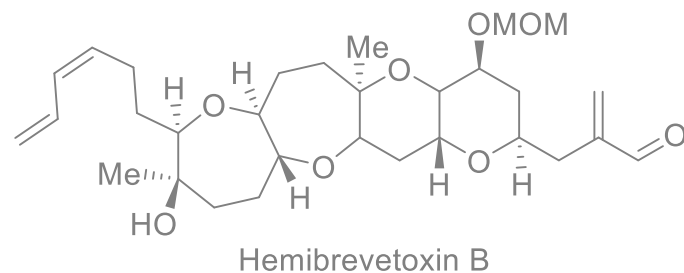
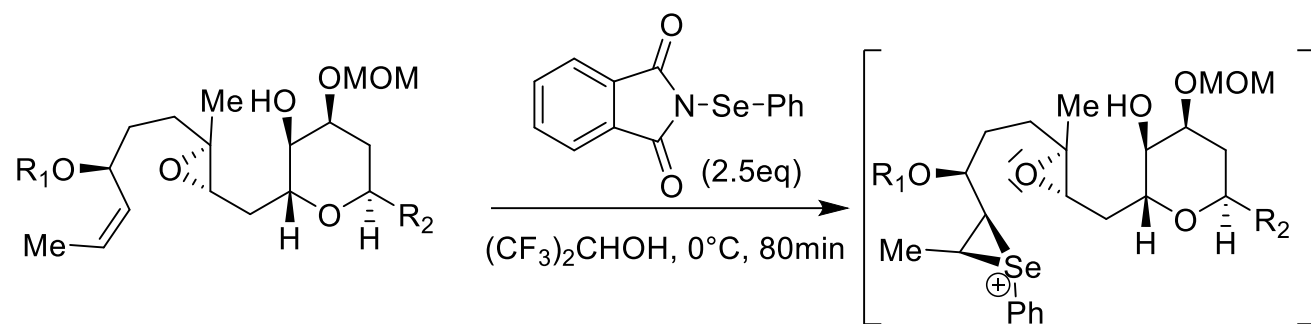
- ▶ Holton's total synthesis of Hemibrevetoxin B by epoxy-olefin cyclization (2003)



I/ Electrophilic Domino Reactions

I-1/ Epoxide opening

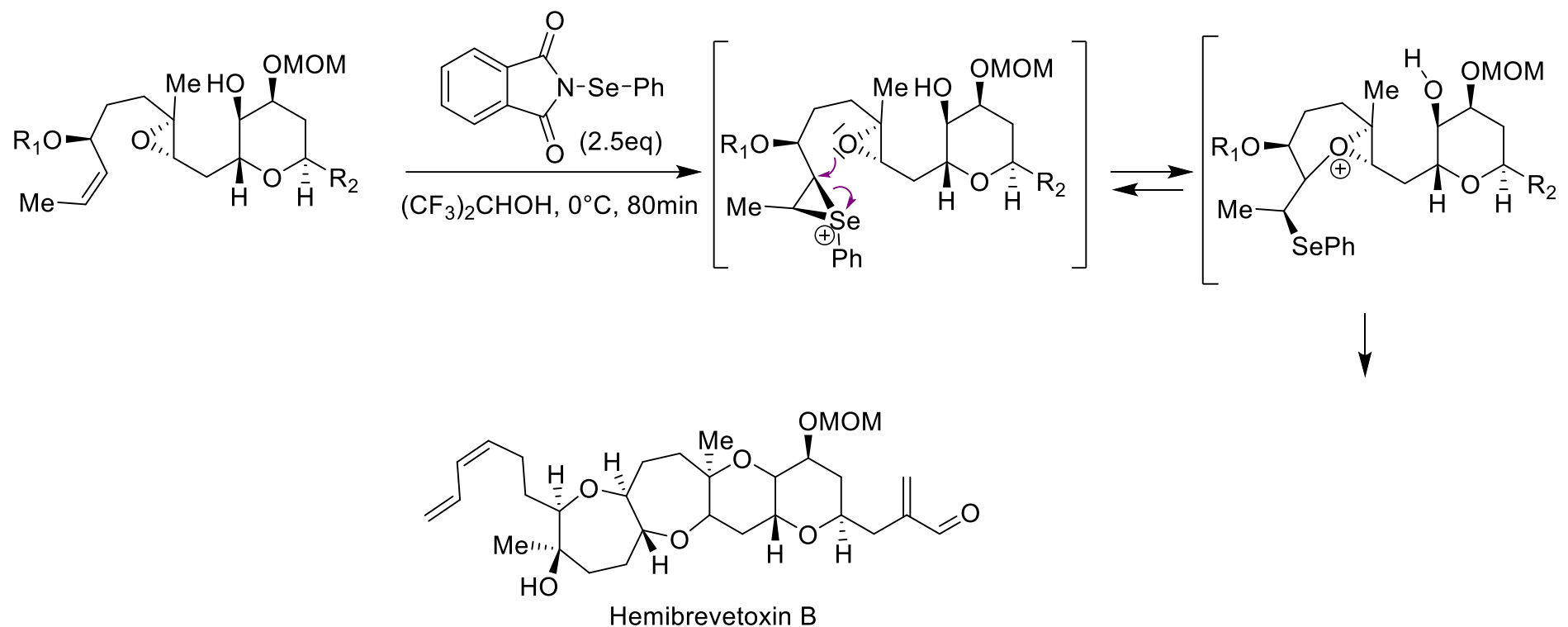
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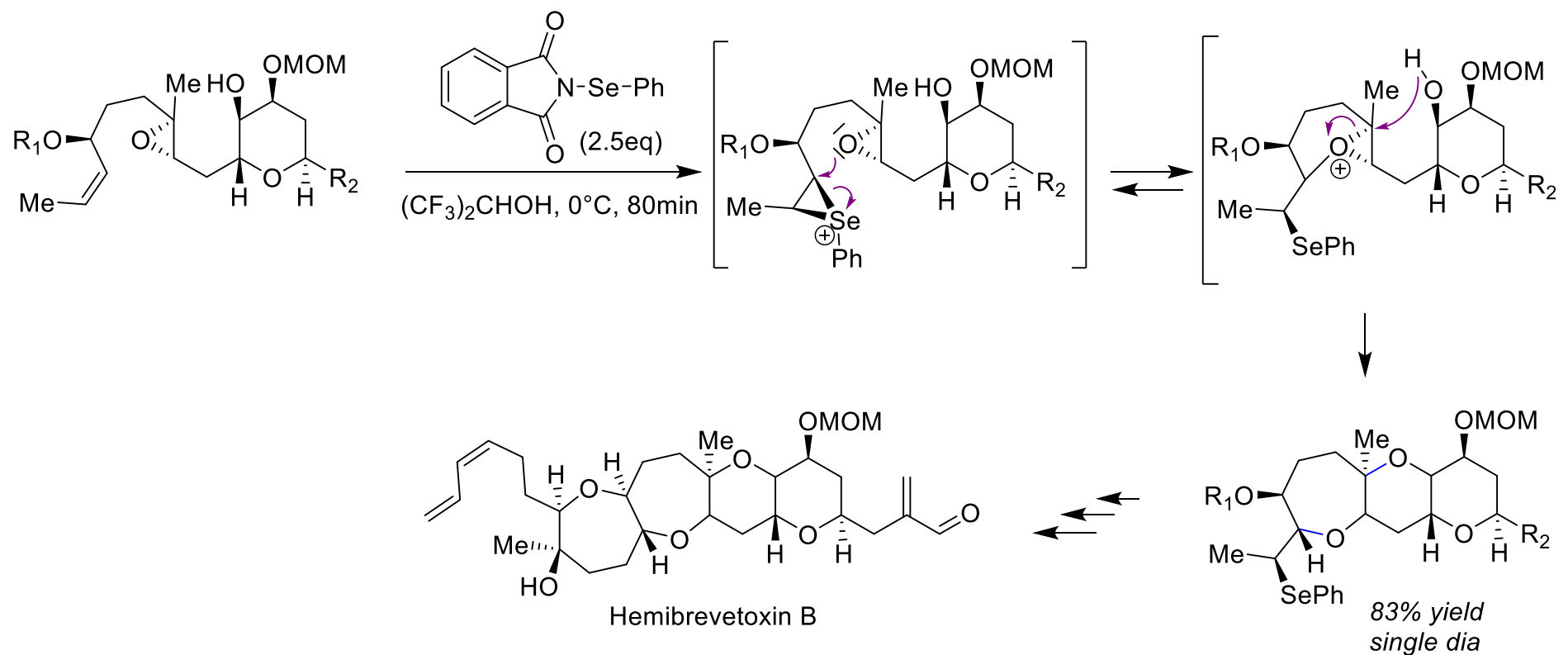
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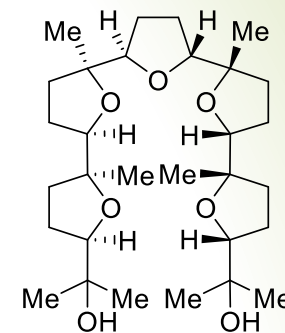
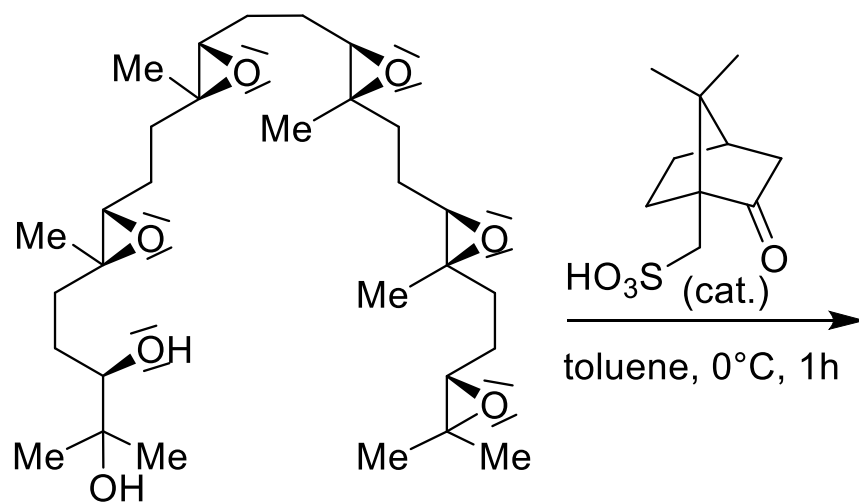
- Holton's total synthesis of Hemibrevetoxin B by epoxy-olefin cyclization (2003)



I/ Electrophilic Domino Reactions

I-1/ Epoxide opening

- Corey's total synthesis of the proposed structure of Glabrescol (2000)

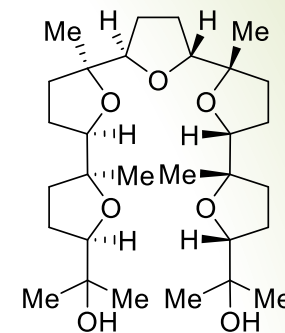
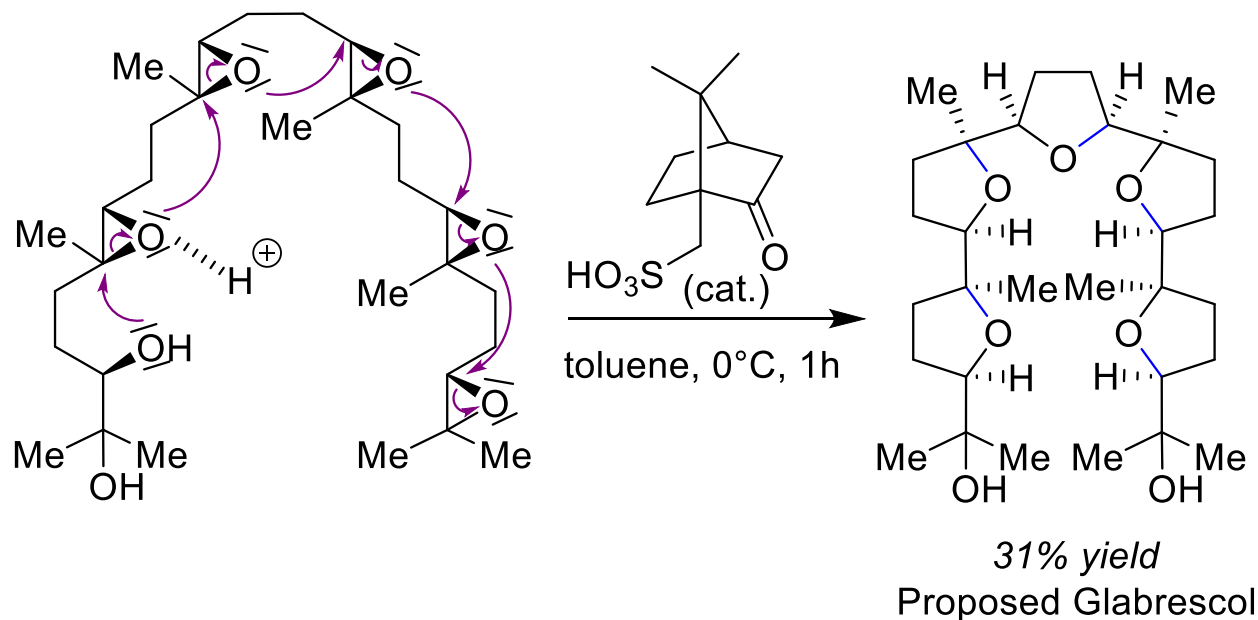


Real structure of Glabrescol

I/ Electrophilic Domino Reactions

I-1/ Epoxide opening

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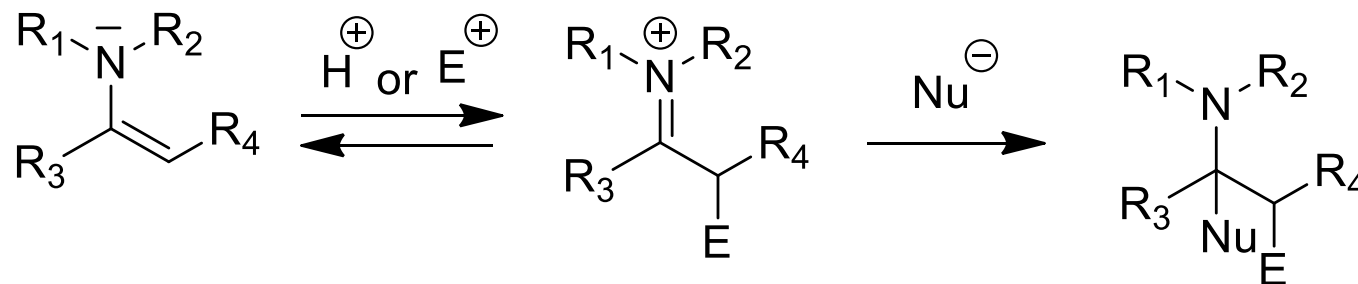


Real structure of Glabrescol

I/ Electrophilic Domino Reactions

I-2/ Enamine-Iminium

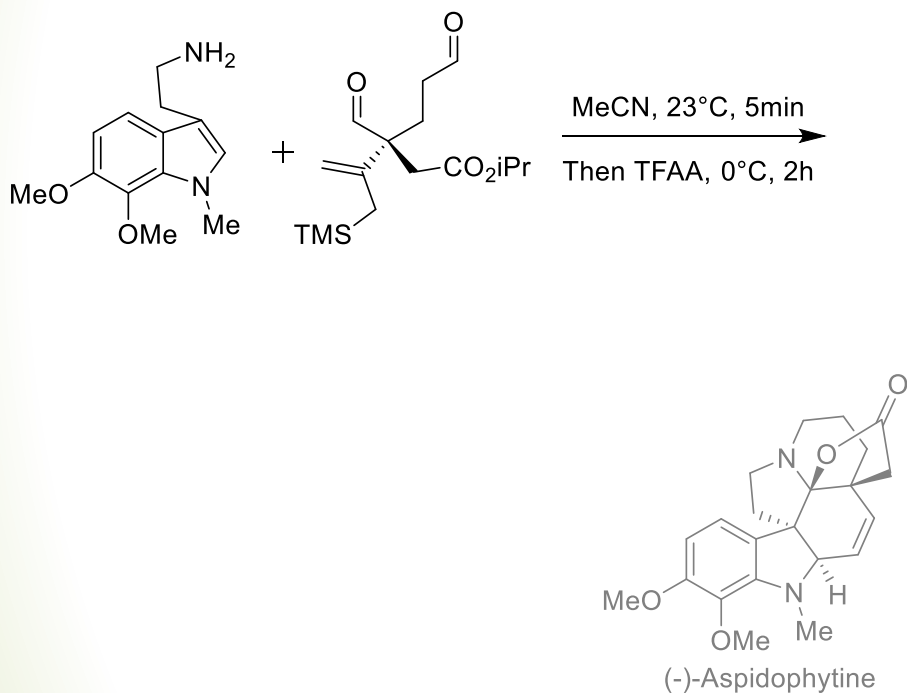
- ▶ Enamine : good nucleophile
- ▶ Iminium : good electrophile



I/ Electrophilic Domino Reactions

I-2/ Enamine-Iminium

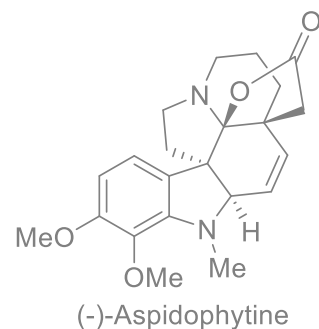
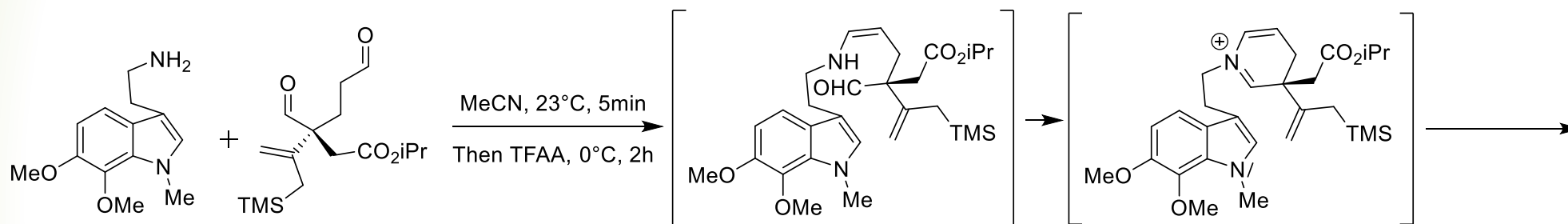
► Corey's total synthesis of (-)-Aspidophytine (1999)



I/ Electrophilic Domino Reactions

I-2/ Enamine-Iminium

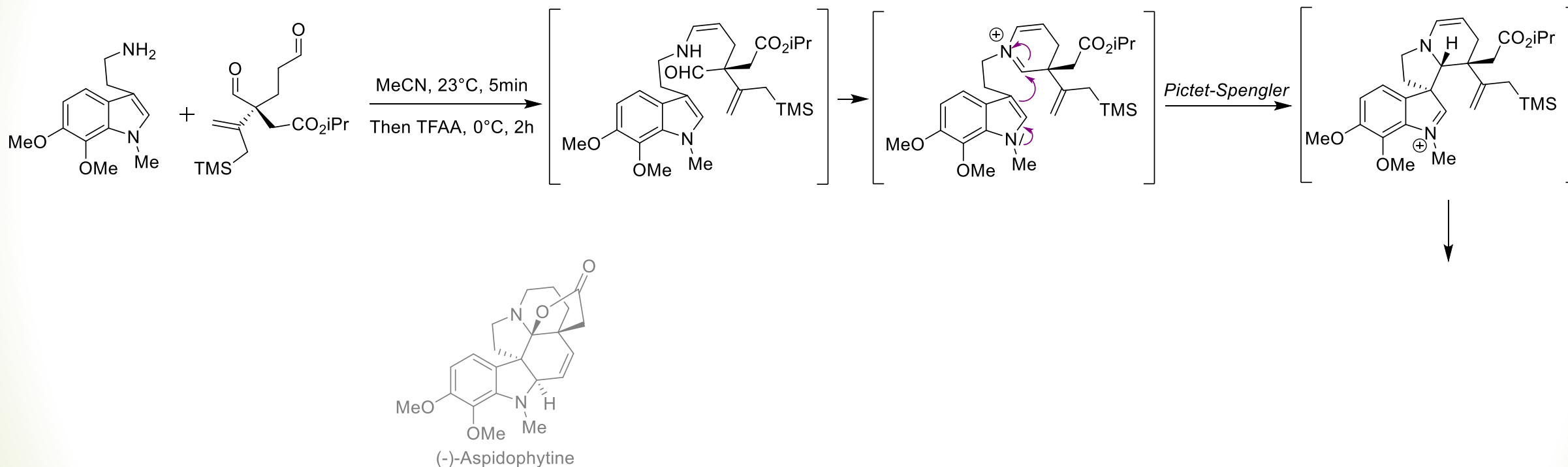
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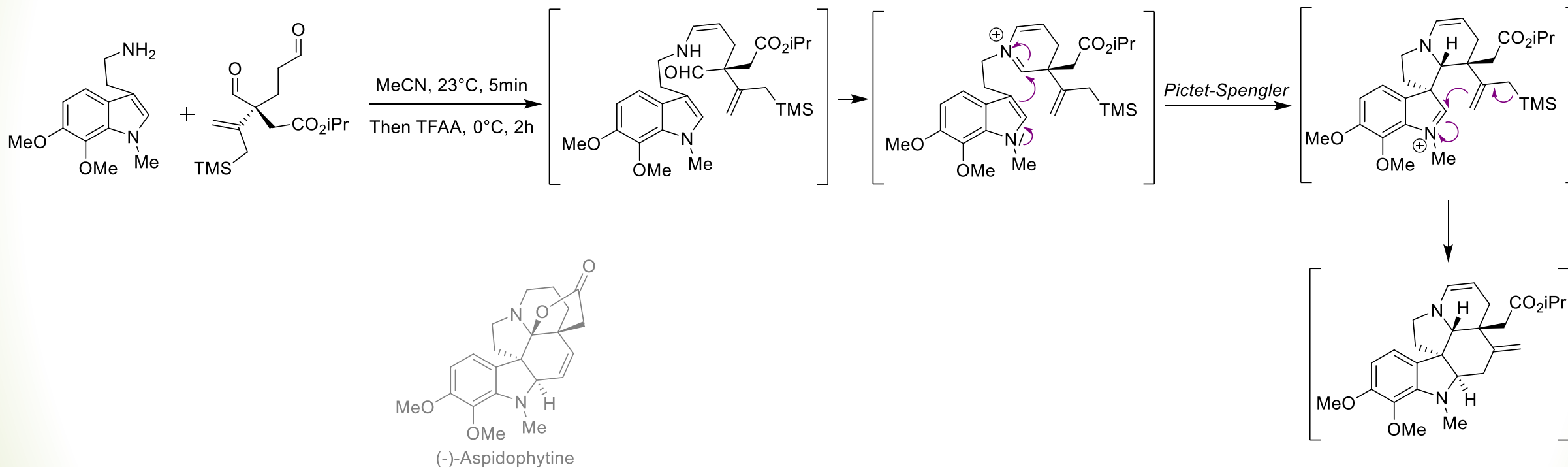
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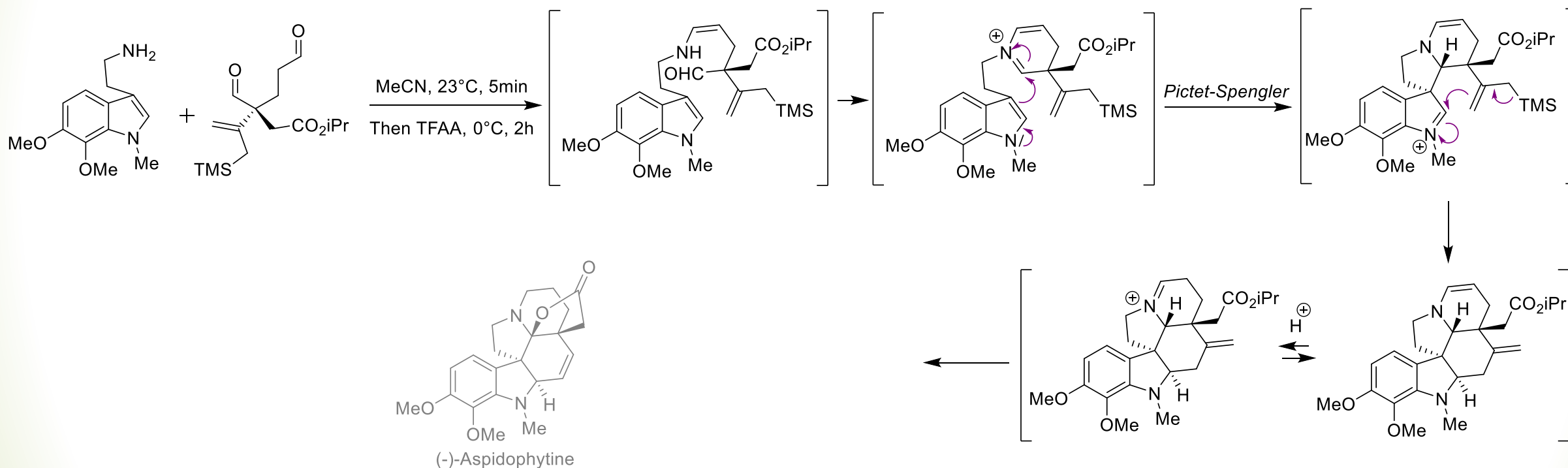
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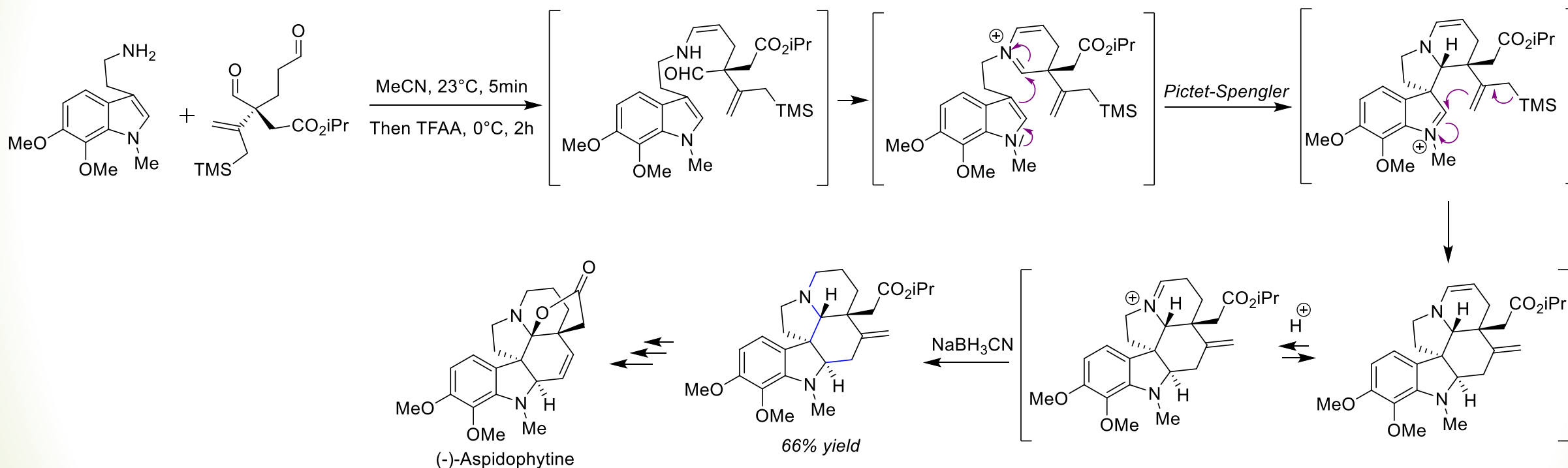
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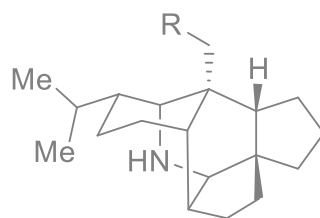
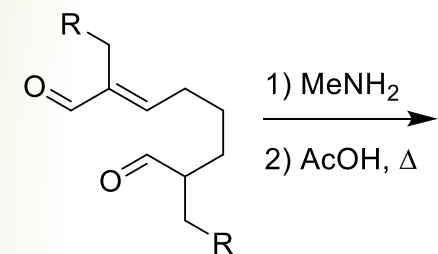
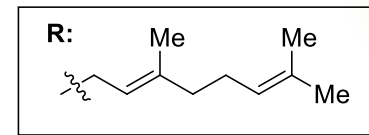
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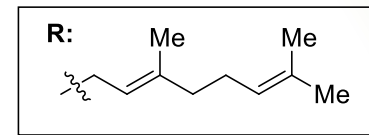
I-2/ Enamine-Iminium

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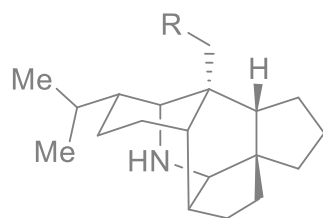
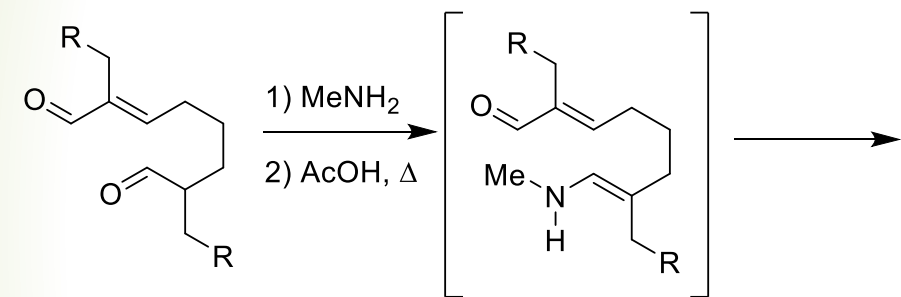
Dihydro-*proto*-daphniphylline

I/ Electrophilic Domino Reactions



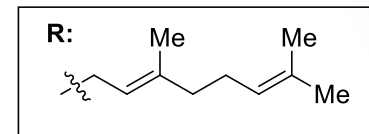
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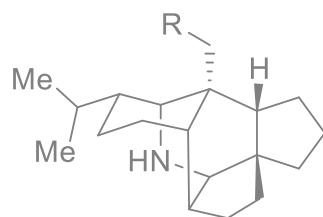
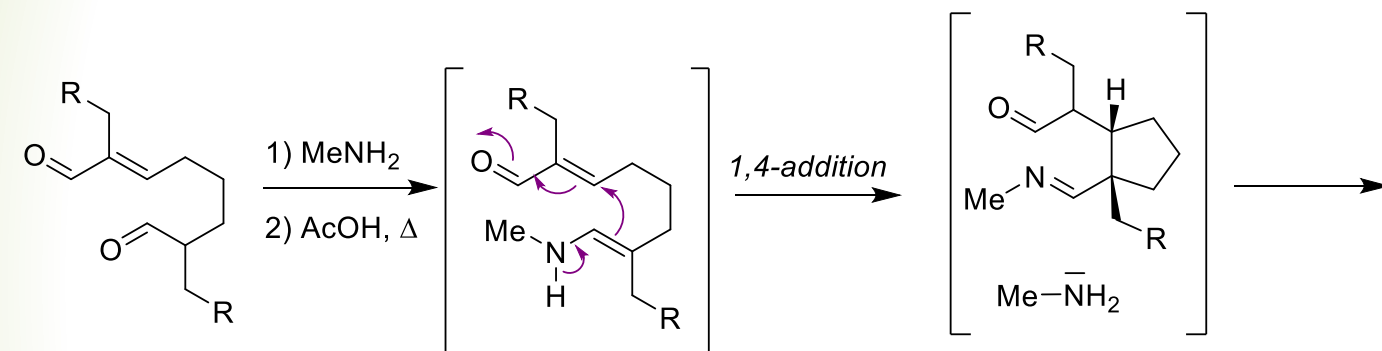
Dihydro-*proto*-daphniphylline

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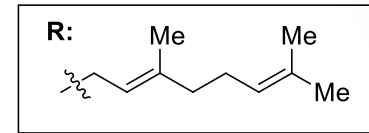
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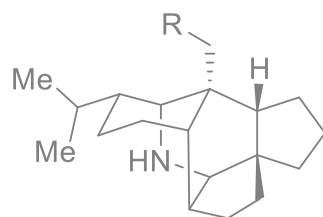
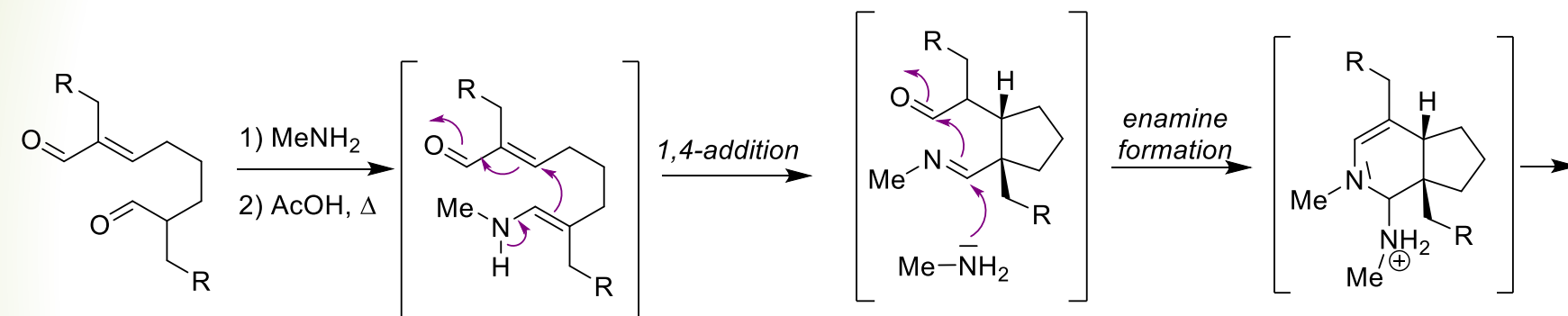
Dihydro-*proto*-daphniphylline

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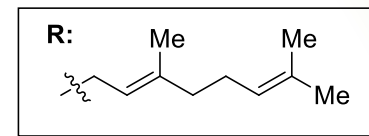
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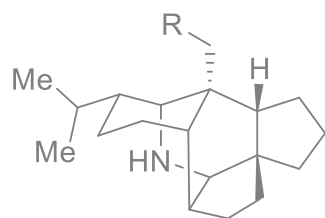
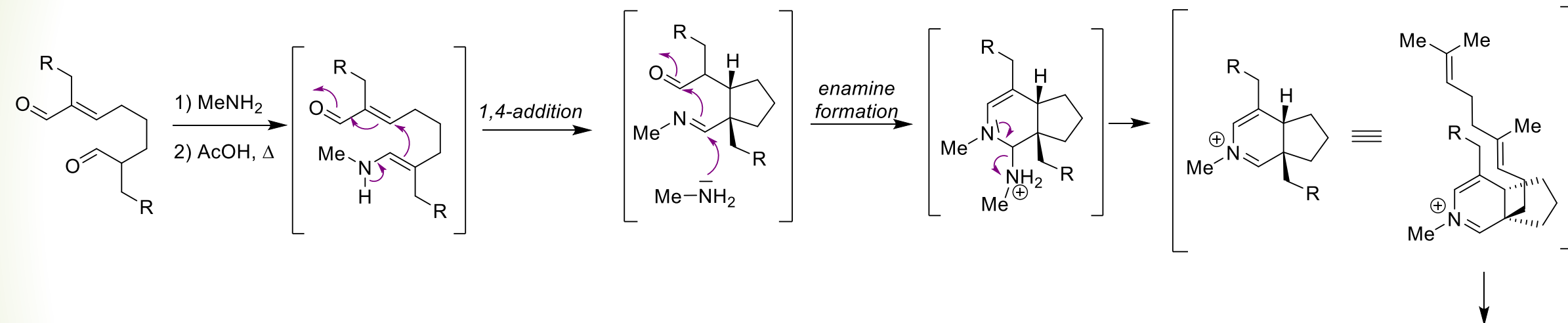
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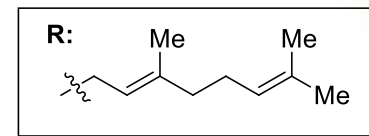
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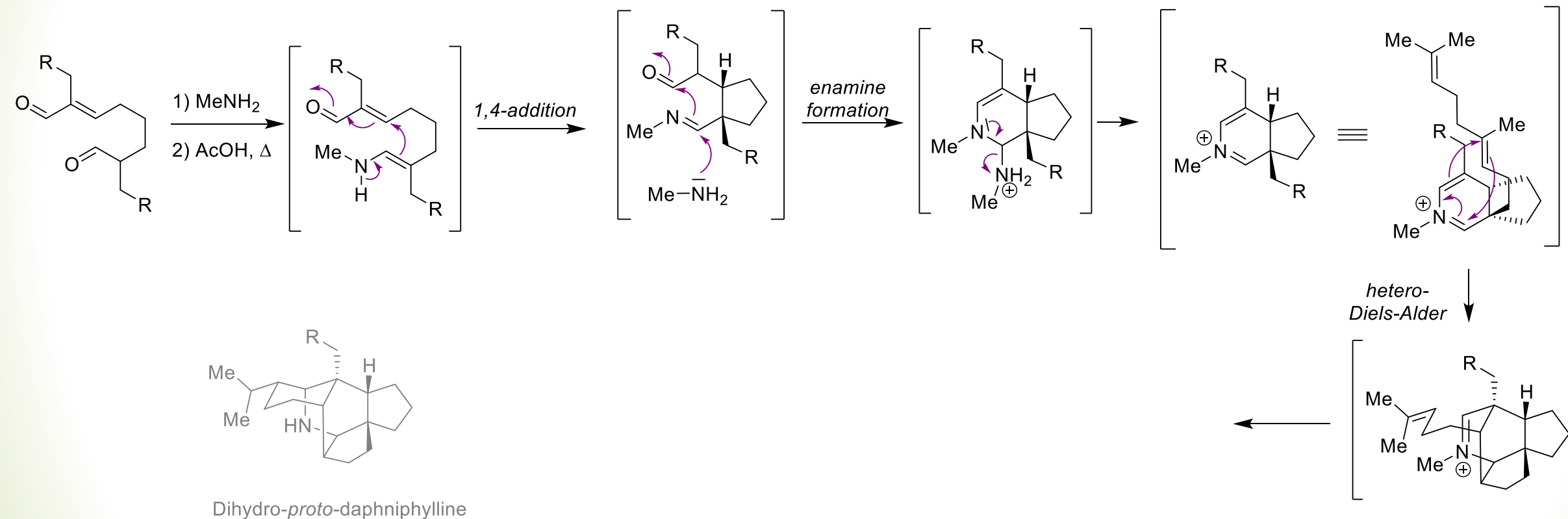
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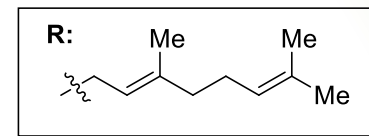
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C. H. Heathcock, M. M. Hansen, R. B. Ruggeri, J. C. Kath, *J. Org. Chem.* **1992**, 57, 2544–2553.

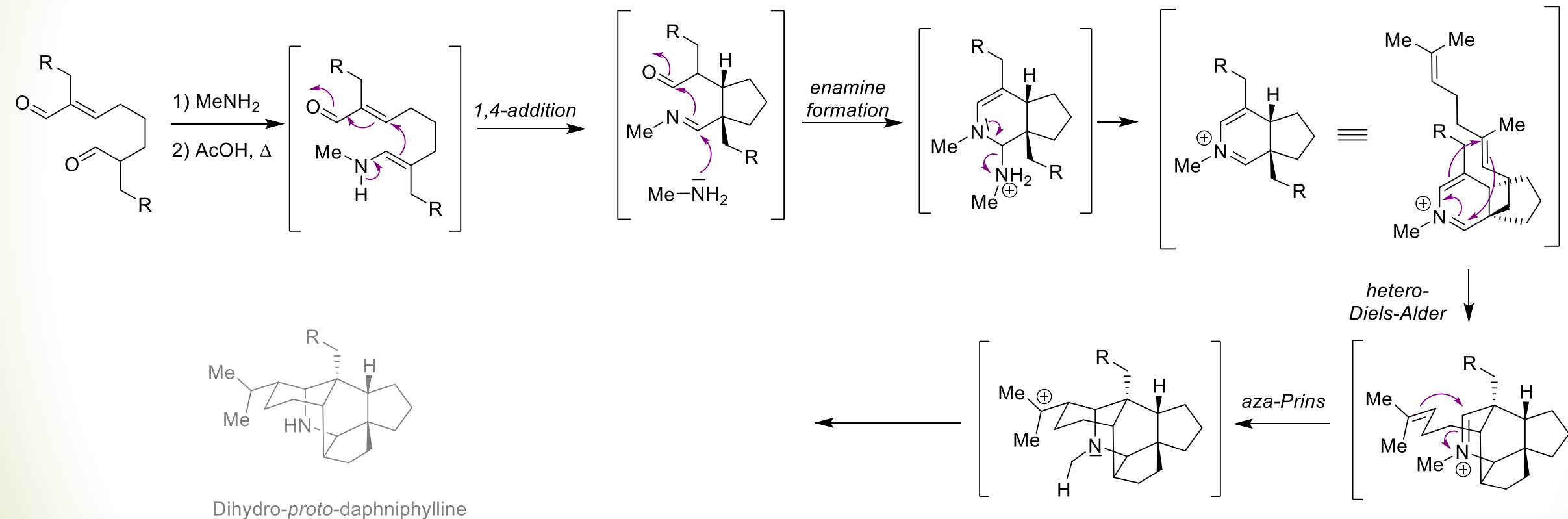
C. H. Heathcock, S. Piettre, R. B. Ruggeri, J. A. Ragan, J. C. Kath, *J. Org. Chem.* **1992**, 57, 2554–2566.

I/ Electrophilic Domino Reactions



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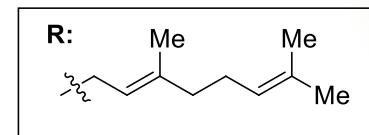
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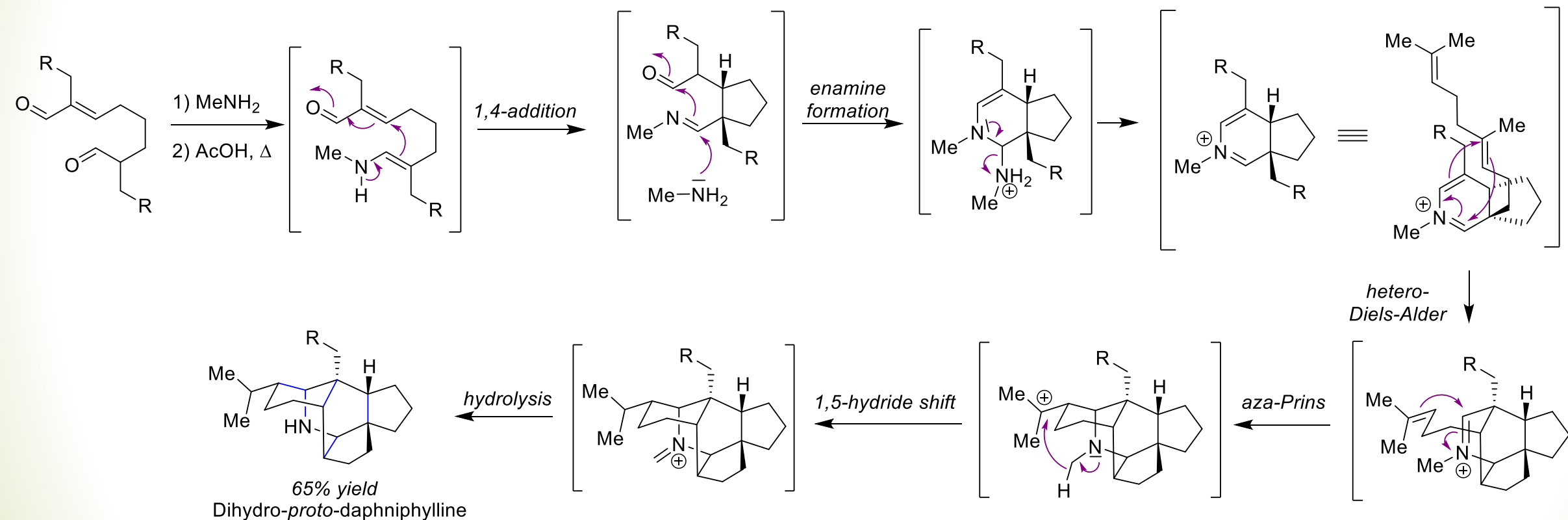


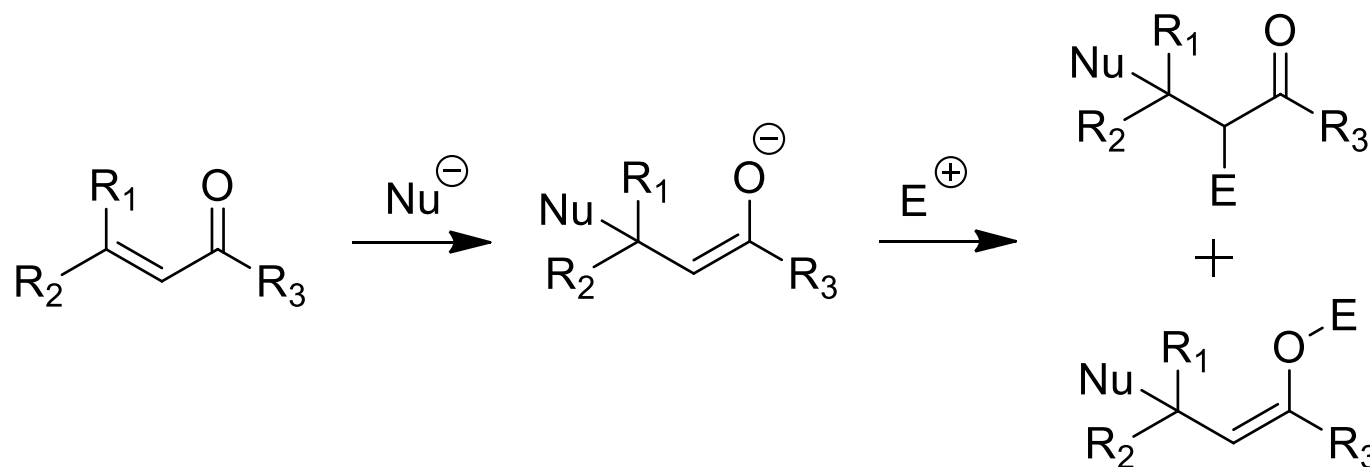
Table of Contents

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II/ Nucleophilic Domino Reactions

Michael acceptor

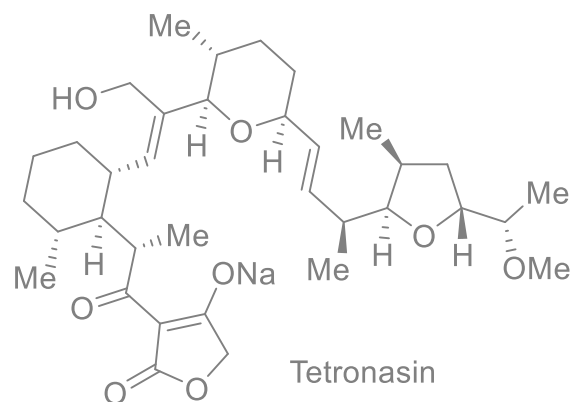
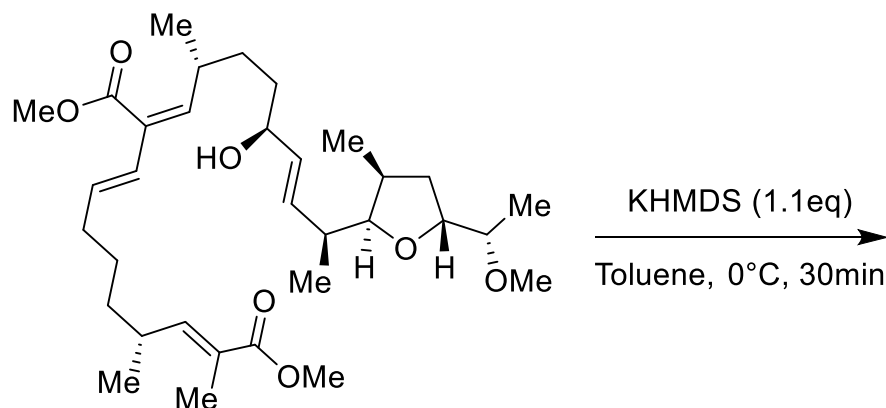
- ▶ Enone : good electrophile
- ▶ Enol : good nucleophile with competition between C- and O- attack



II/ Nucleophilic Domino Reactions

II-1/ Michael acceptor

- ▶ Ley's total synthesis of Tetronasin (1998)



G.-J. Boons, D. S. Brown, J. A. Clase, I. C. Lennon, S. V. Ley, *Tetrahedron Lett.* **1994**, 35, 319 – 322.

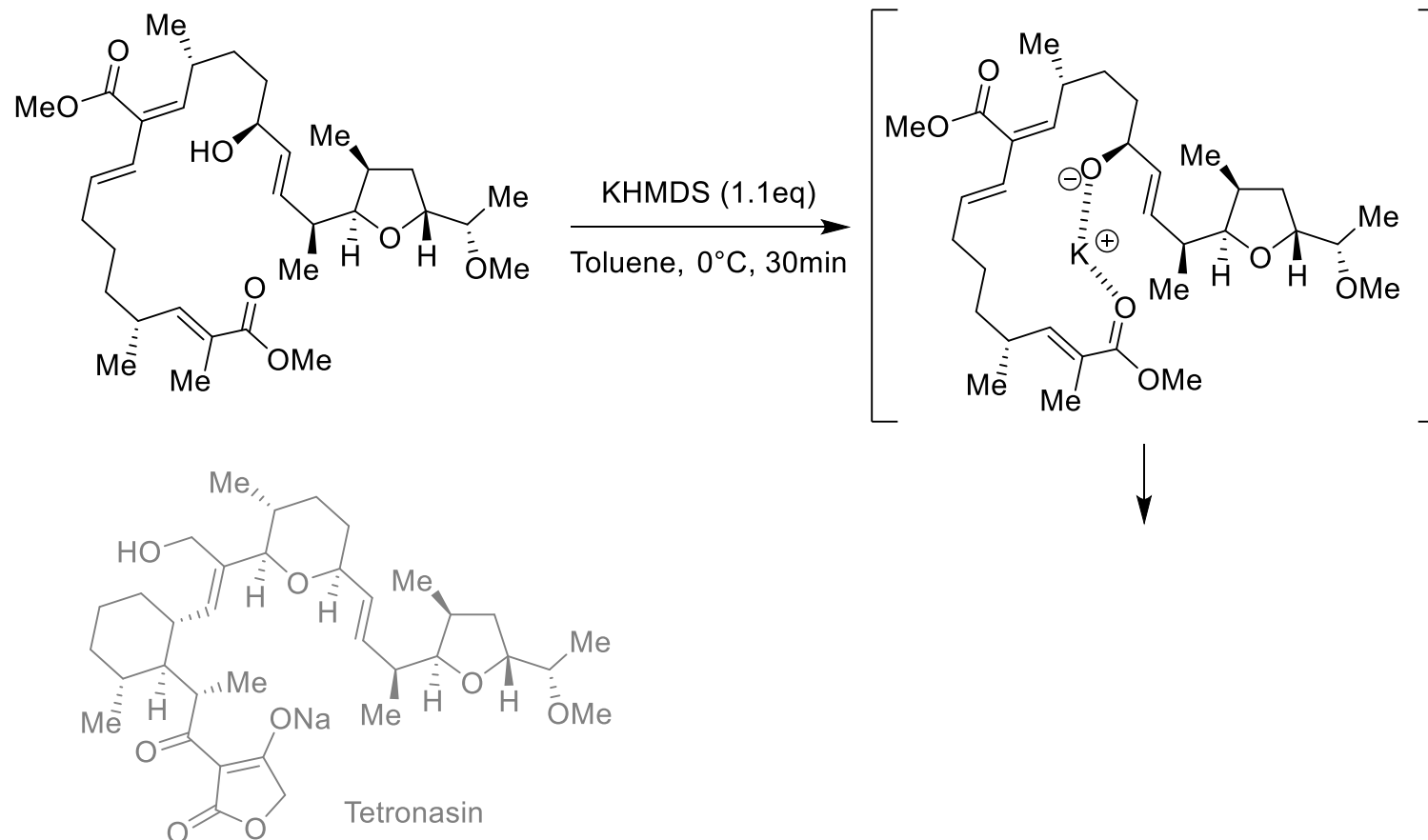
G.-J. Boons, I. C. Lennon, S. V. Ley, E. S. E. Owen, J. Staunton, D. J. Wadsworth, *Tetrahedron Lett.* **1994**, 35, 323 – 326.

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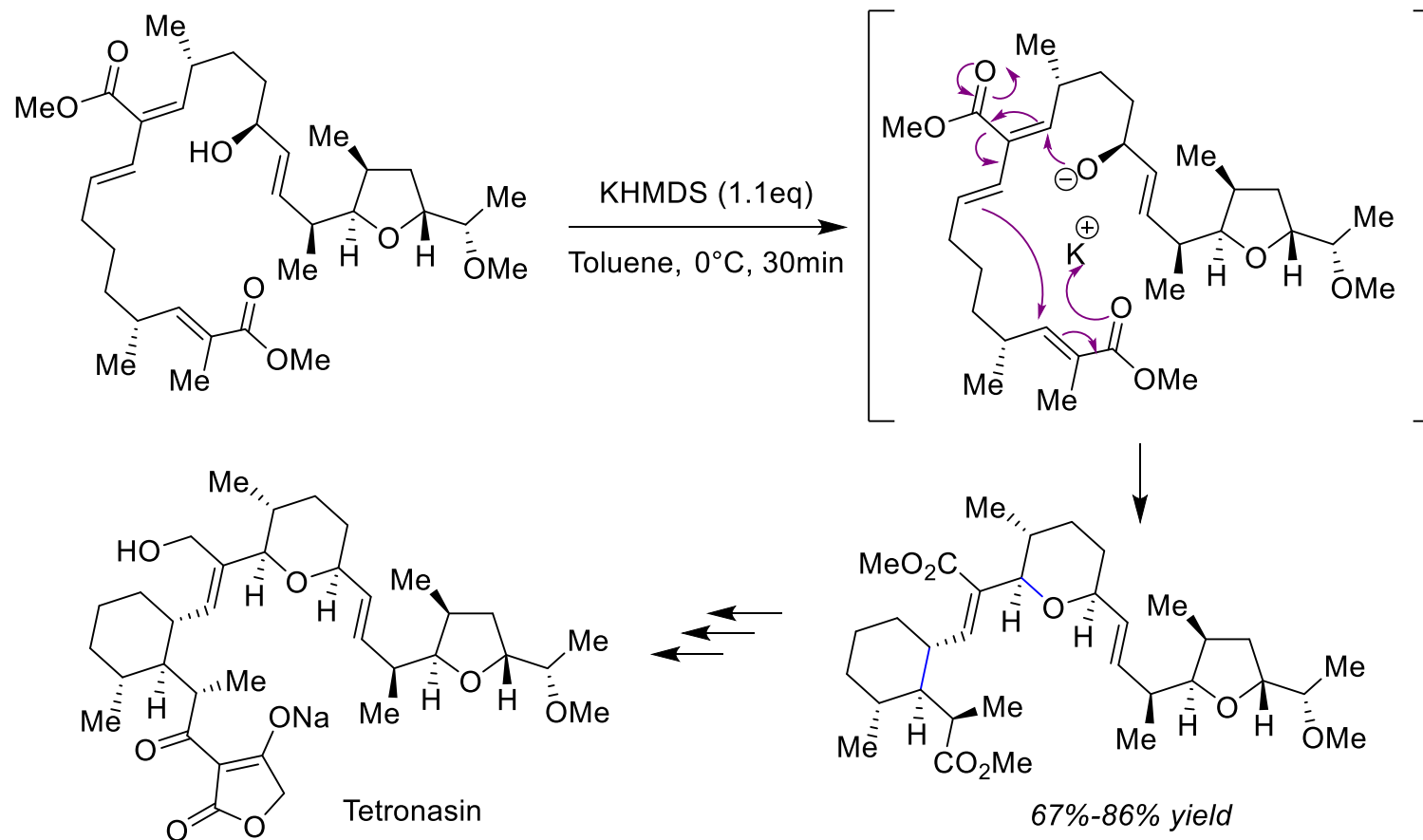
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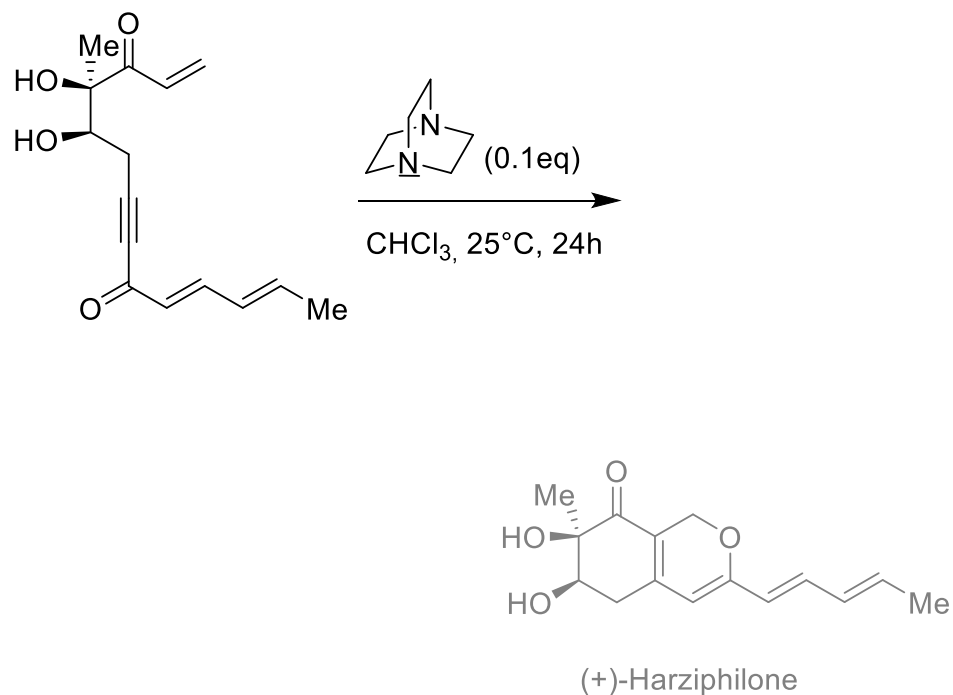
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II/ Nucleophilic Domino Reactions

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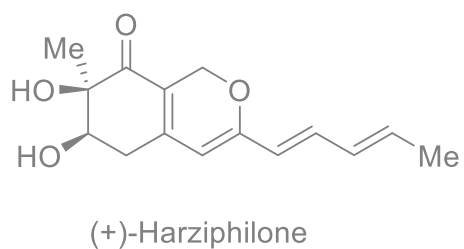
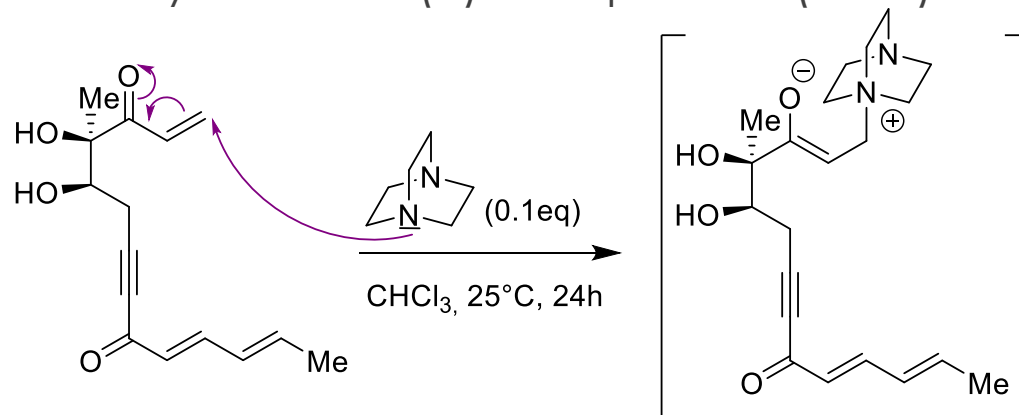
- Sorensen's total synthesis of (+)-Harziphilone (2004)



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II-1/ Michael acceptor

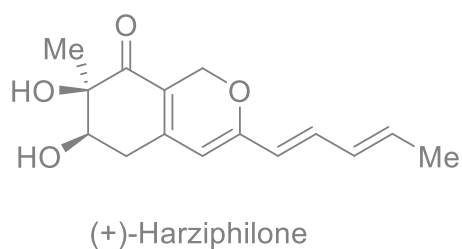
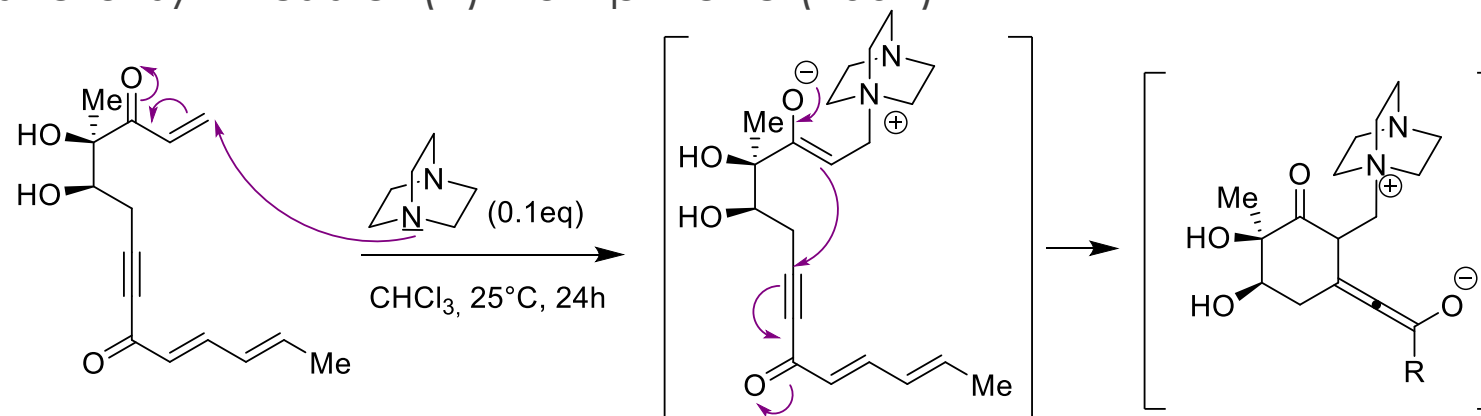
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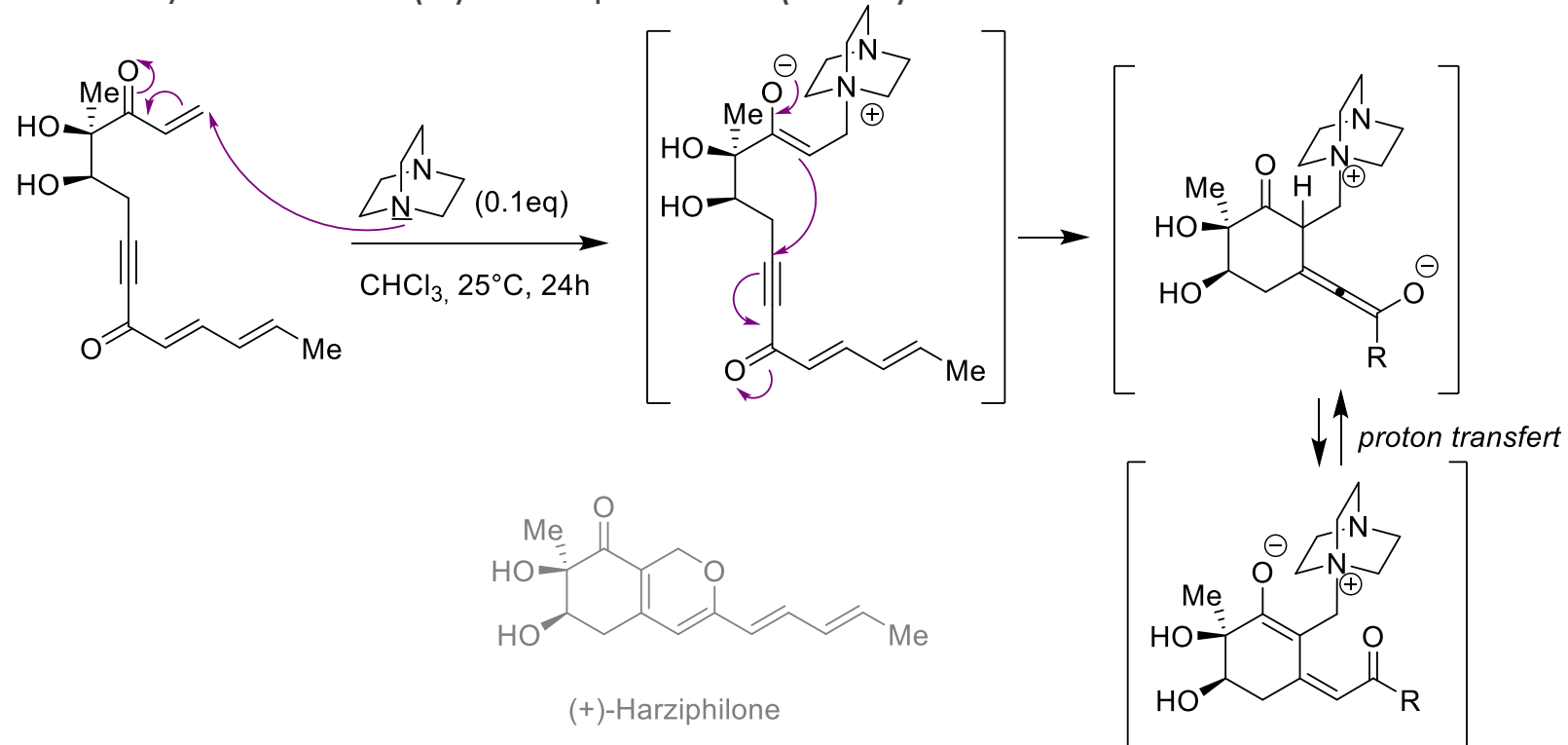
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II/ Nucleophilic Domino Reactions

II-1/ Michael acceptor

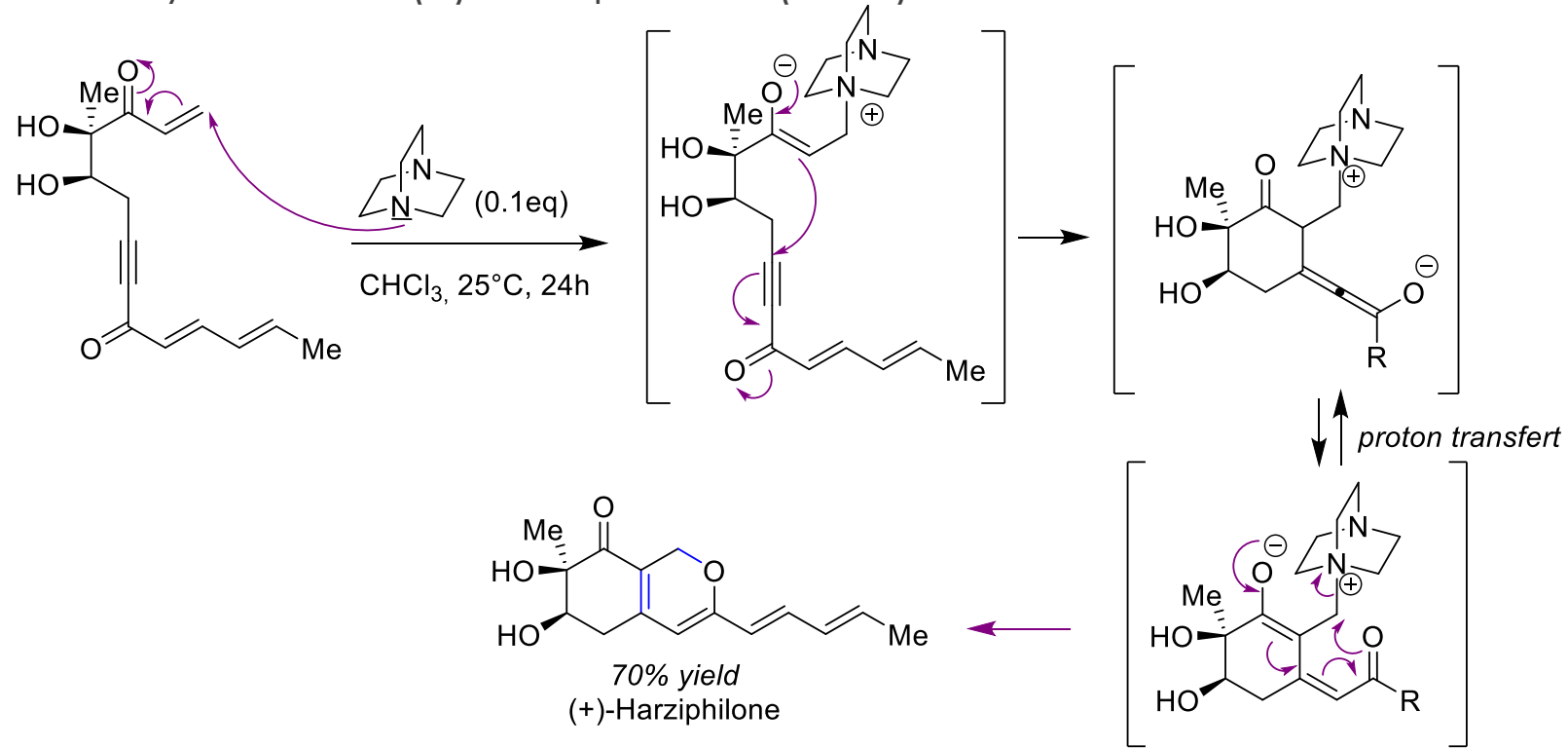
- Sorensen's total synthesis of (+)-Harziphilone (2004)



II/ Nucleophilic Domino Reactions

II-1/ Michael acceptor

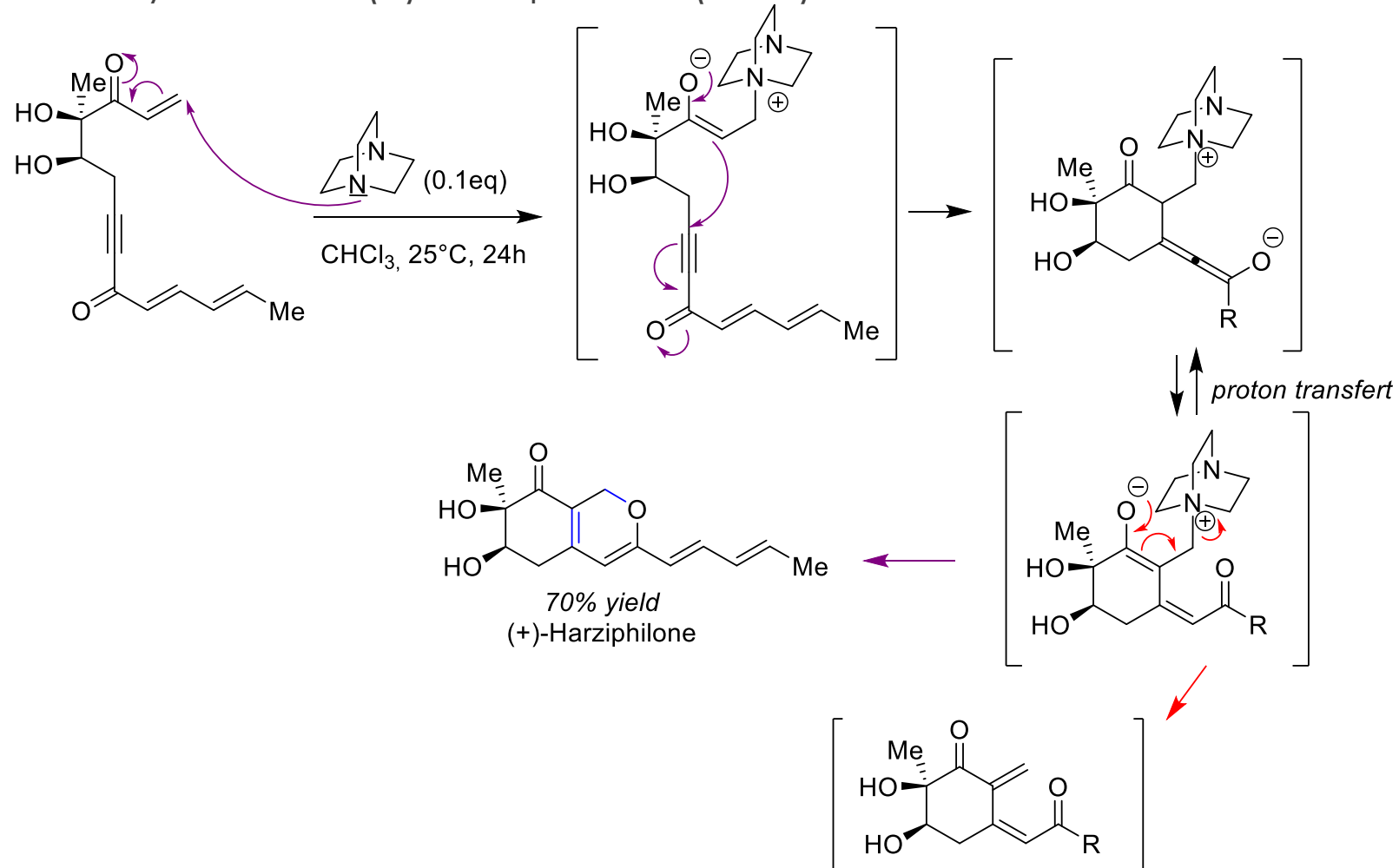
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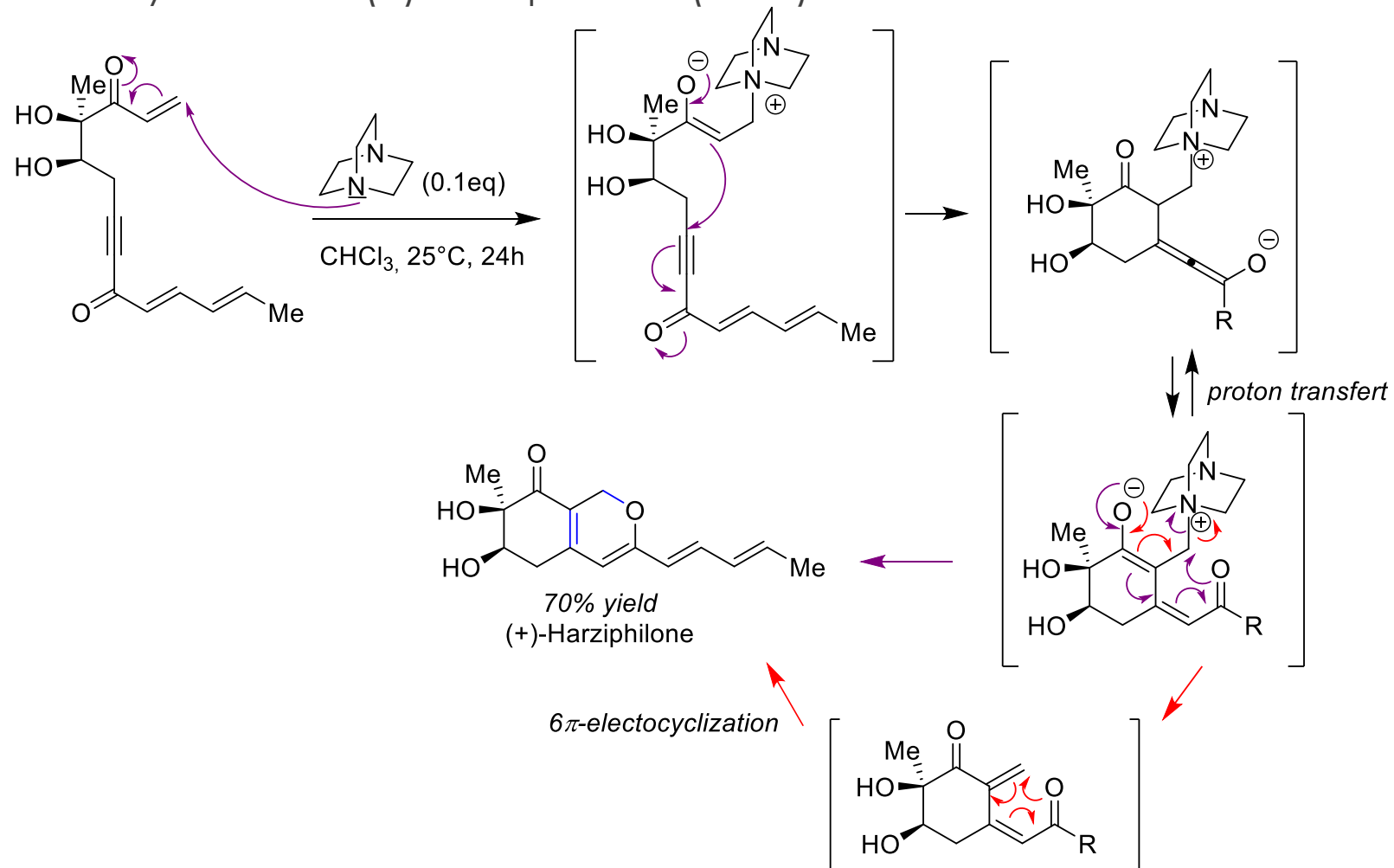
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II/ Nucleophilic Domino Reactions

II-1/ Michael acceptor

- Sorensen's total synthesis of (+)-Harziphilone (2004)



II/ Nucleophilic Domino Reactions

II-2/ Michael acceptor containing nitrogen

- Dimerization of (+)-Avrainvillamide in (-)-Stephacidin (Myers and Baran, 2005)



S. B. Herzon, A. G. Myers, *J. Am. Chem. Soc.* **2005**, 127, 5342–5344.

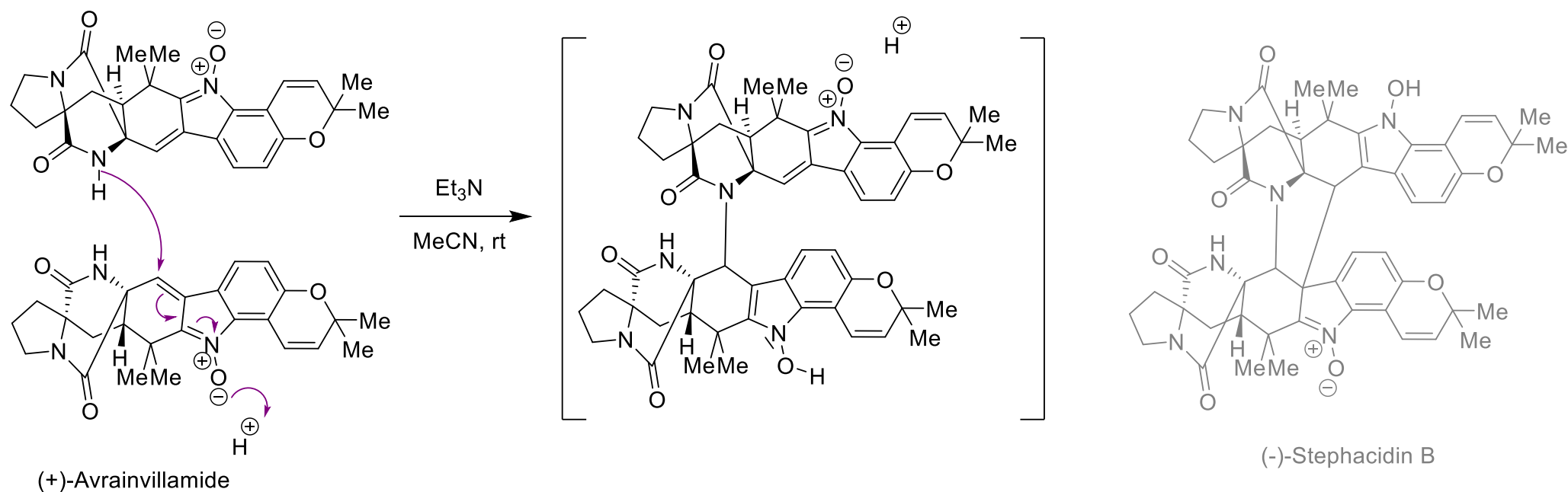
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P. S. Baran, B. D. Hafensteiner, N. B. Ambhaikar, C. A. Guerrero, J. D. Gallagher, *J. Am. Chem. Soc.* **2006**, 128, 8678–8693.

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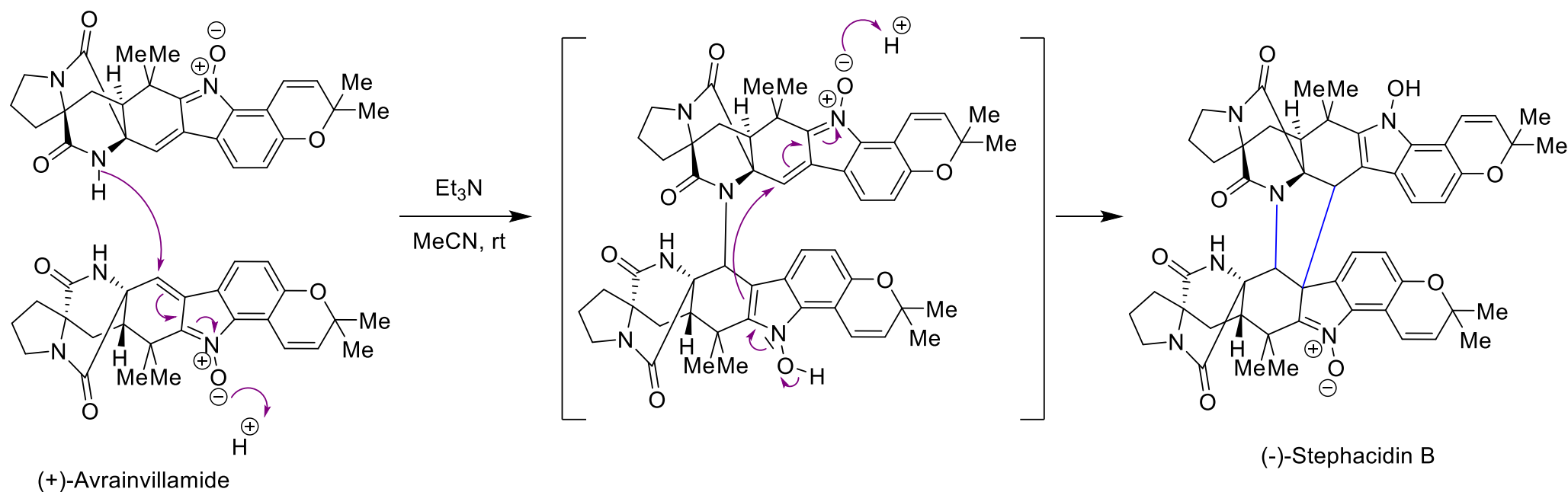
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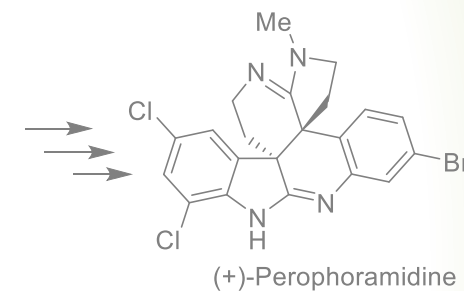
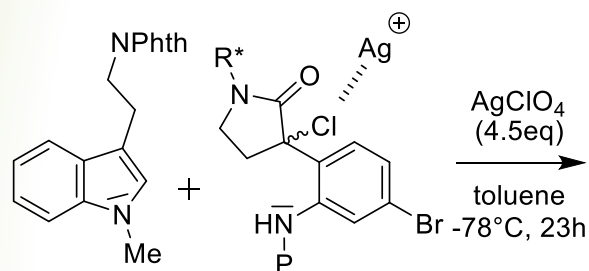
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II/ Nucleophilic Domino Reactions

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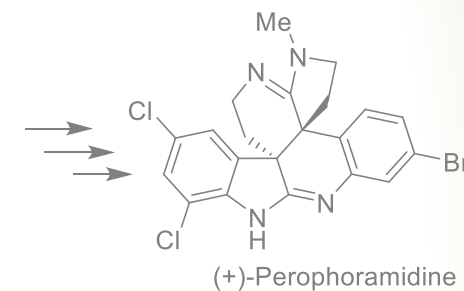
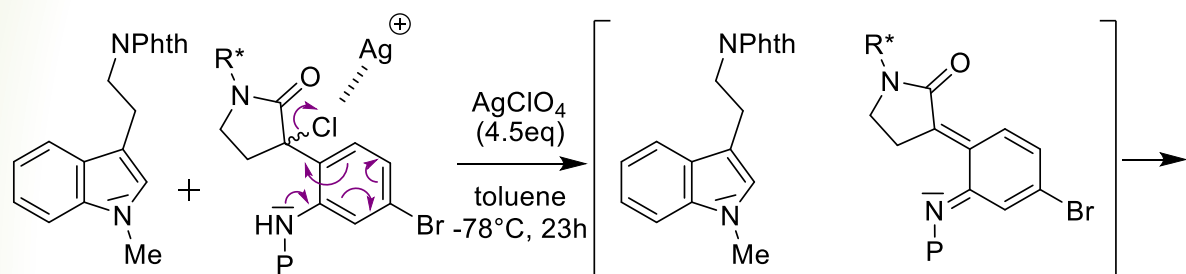
► Qin's total synthesis of (+)-Perophoramidine (2010)



II/ Nucleophilic Domino Reactions

II-2/ Michael acceptor containing nitrogen

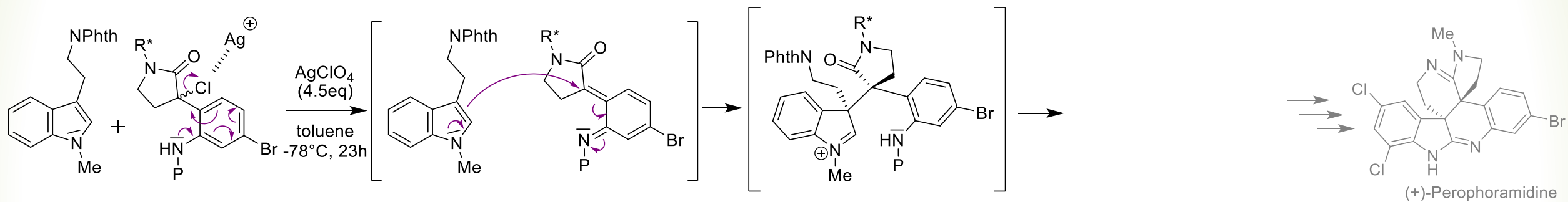
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II-2/ Michael acceptor containing nitrogen

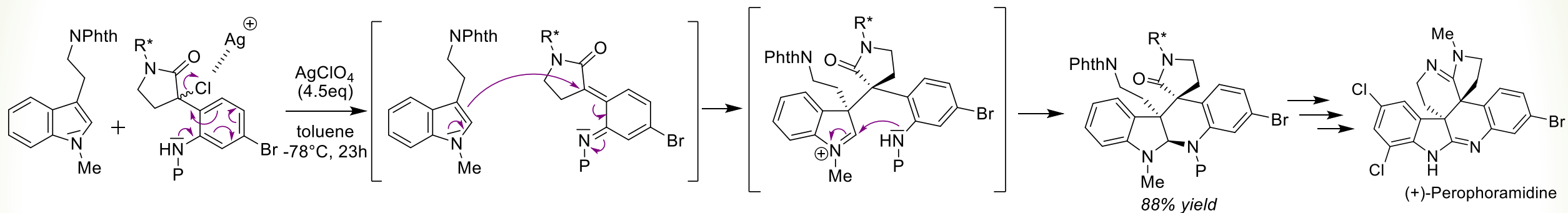
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II-2/ Michael acceptor containing nitrogen

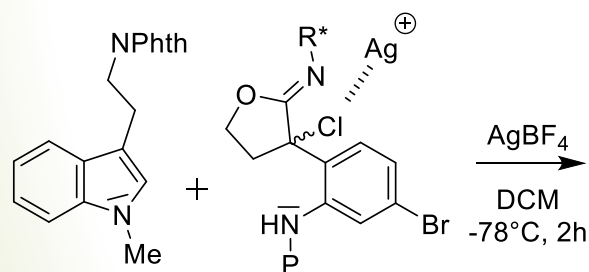
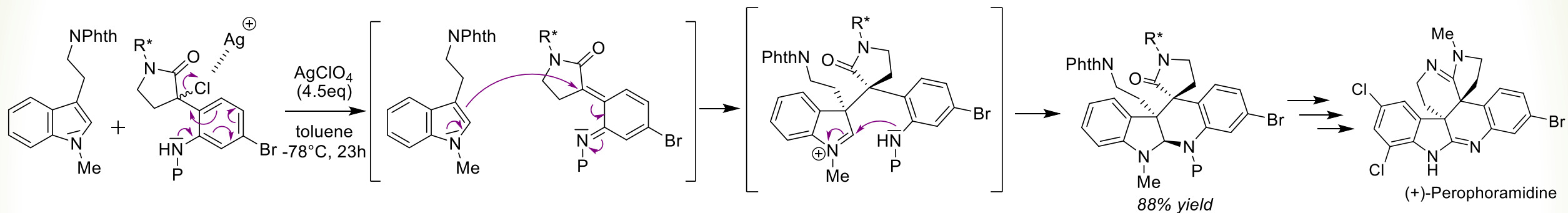
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II/ Nucleophilic Domino Reactions

II-2/ Michael acceptor containing nitrogen

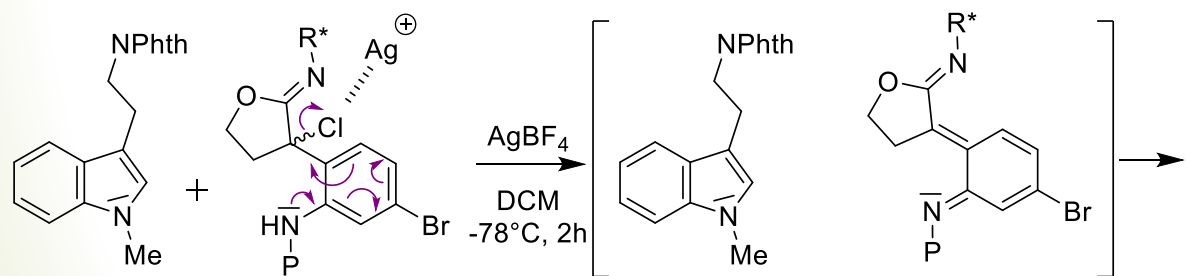
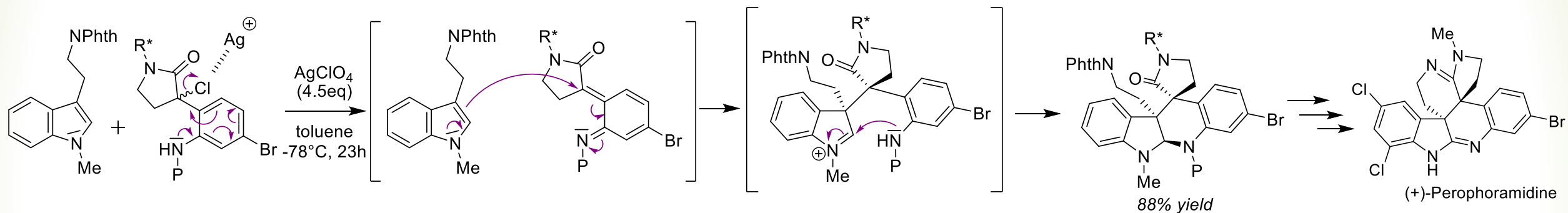
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II/ Nucleophilic Domino Reactions

II-2/ Michael acceptor containing nitrogen

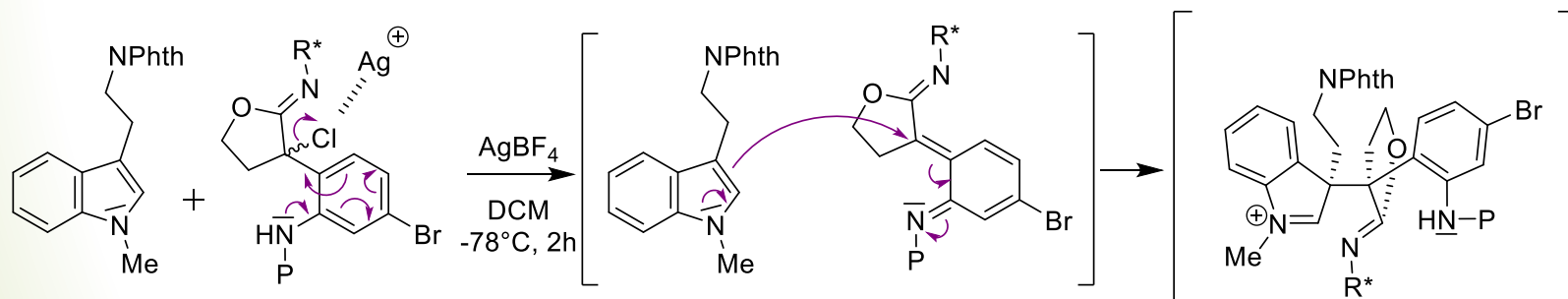
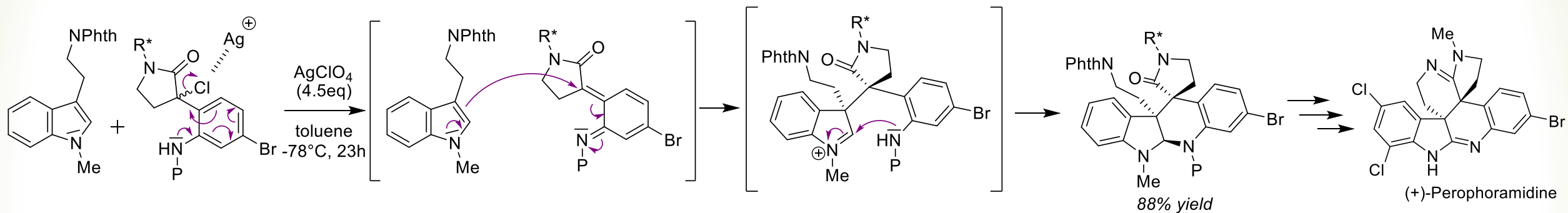
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II-2/ Michael acceptor containing nitrogen

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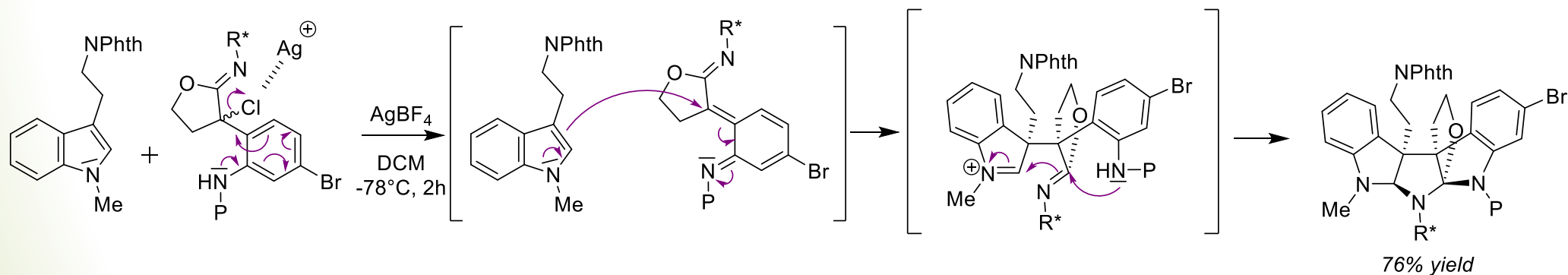
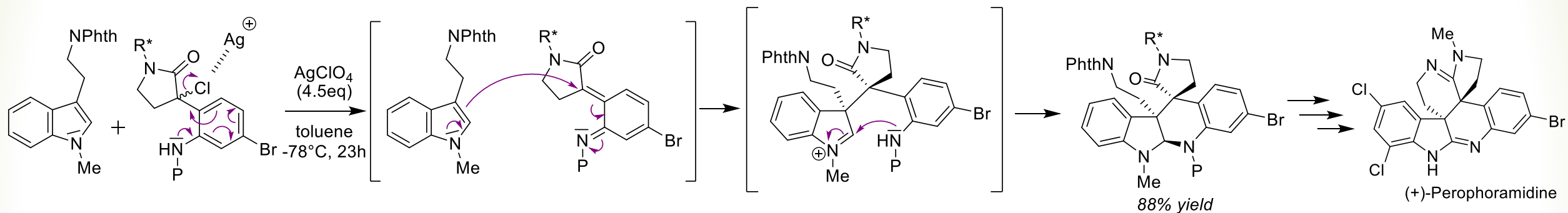


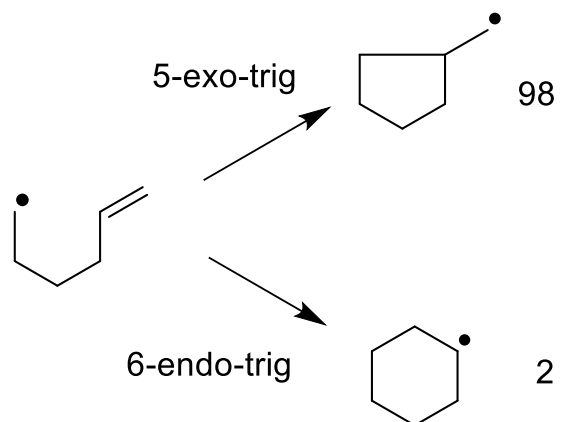
Table of Contents

- Introduction and Definitions
- I/ Electrophilic Domino Reactions
- II/ Nucleophilic Domino Reactions
- **III/ Radical Domino Reactions**
- IV/ Pericyclic Domino Reactions
- V/ Transition Metal Catalyzed Domino Reactions
- Conclusion

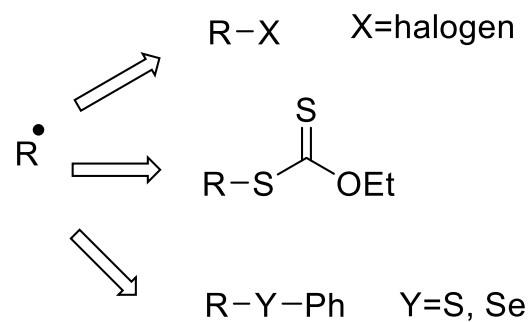
III/ Radical Domino Reactions

Radical cyclization

► Reactivity – Baldwin rules



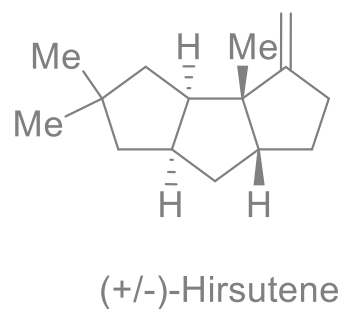
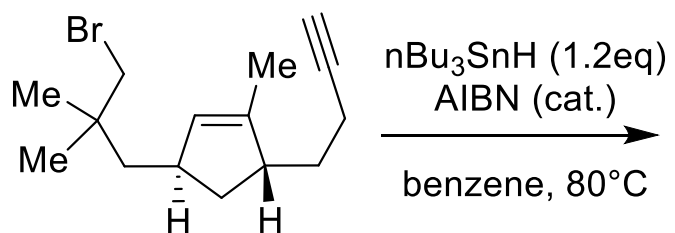
► Radical precursor



III/ Radical Domino Reactions

III-1/ Alkyl radical cyclization

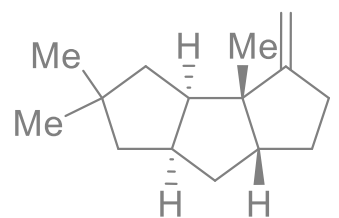
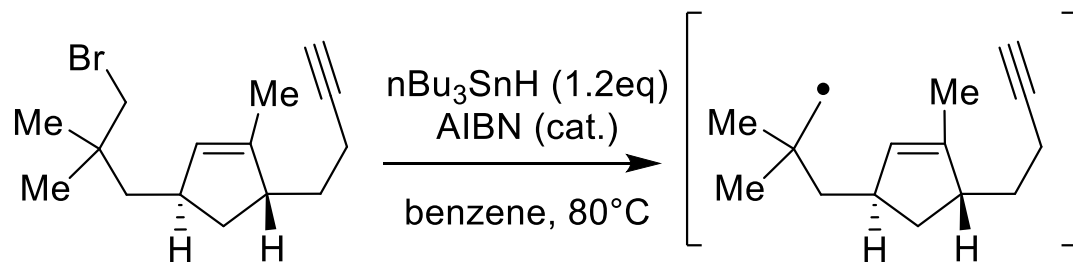
► Curran's total synthesis of (+/-)-Hirsutene (1985)



III/ Radical Domino Reactions

III-1/ Alkyl radical cyclization

► Curran's total synthesis of (+/-)-Hirsutene (1985)

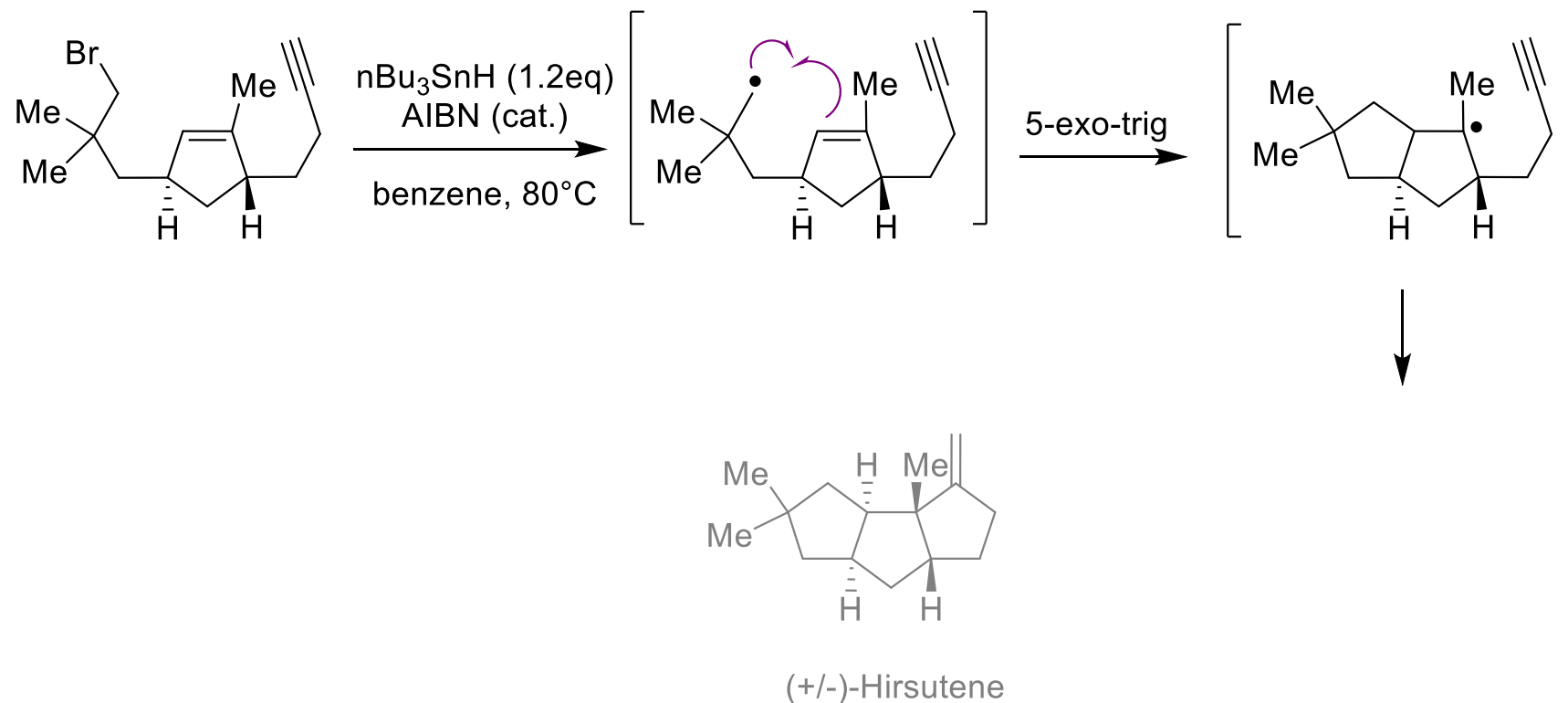


(+/-)-Hirsutene

III/ Radical Domino Reactions

III-1/ Alkyl radical cyclization

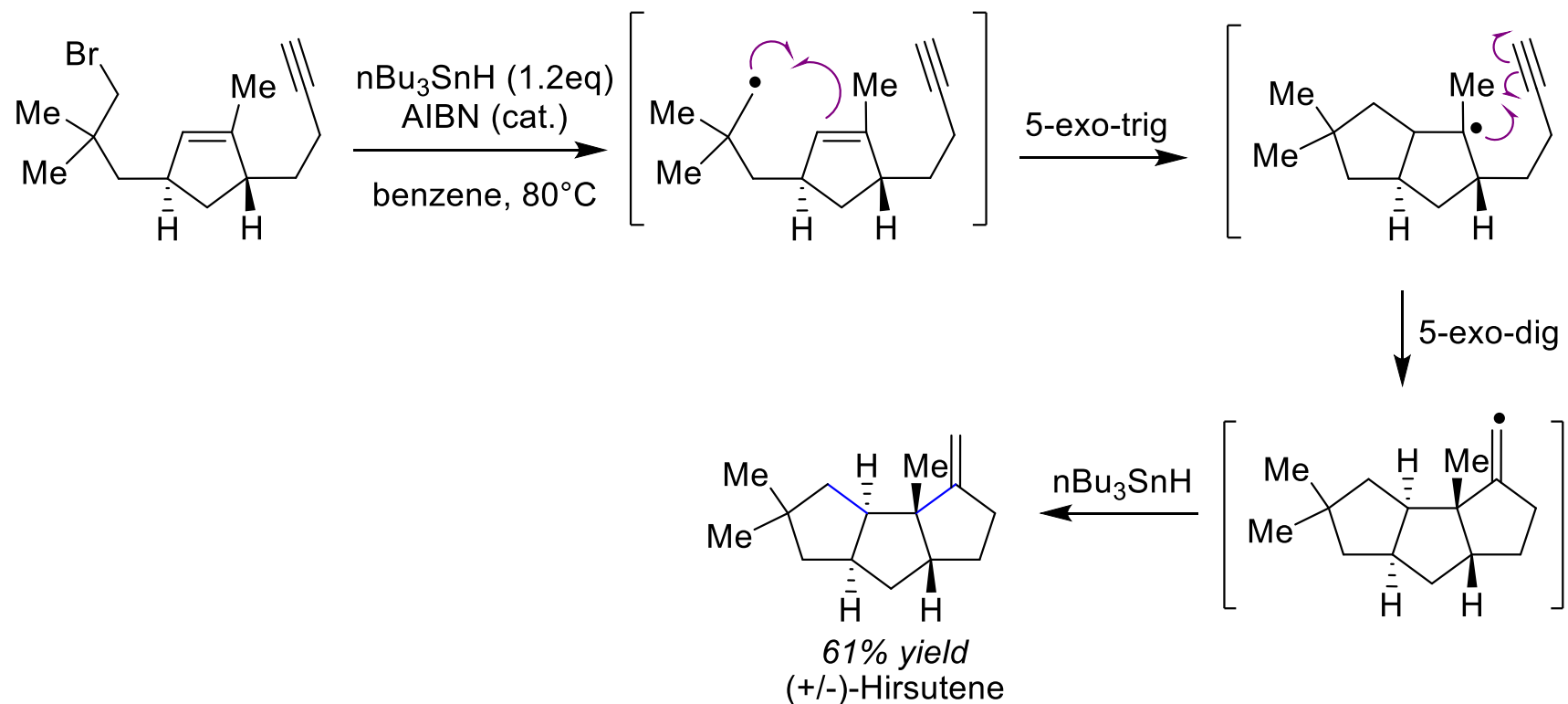
► Curran's total synthesis of (+/-)-Hirsutene (1985)



III/ Radical Domino Reactions

III-1/ Alkyl radical cyclization

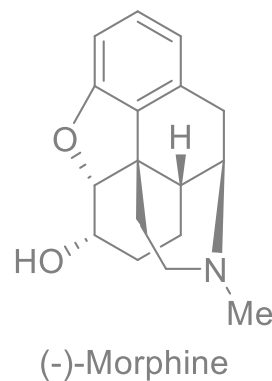
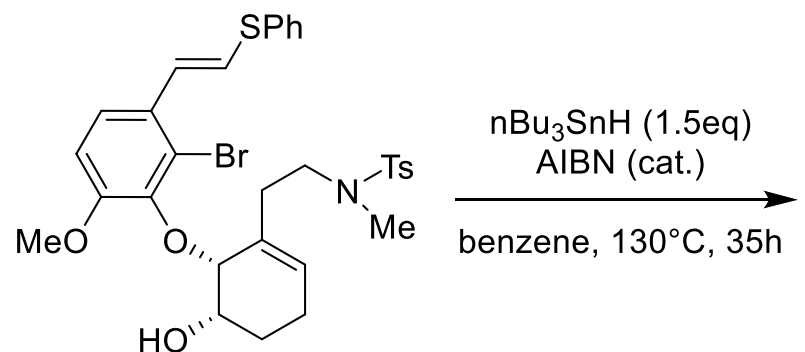
► Curran's total synthesis of (+/-)-Hirsutene (1985)



III/ Radical Domino Reactions

III-2/ Aryl radical cyclization

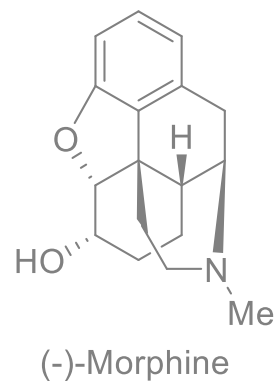
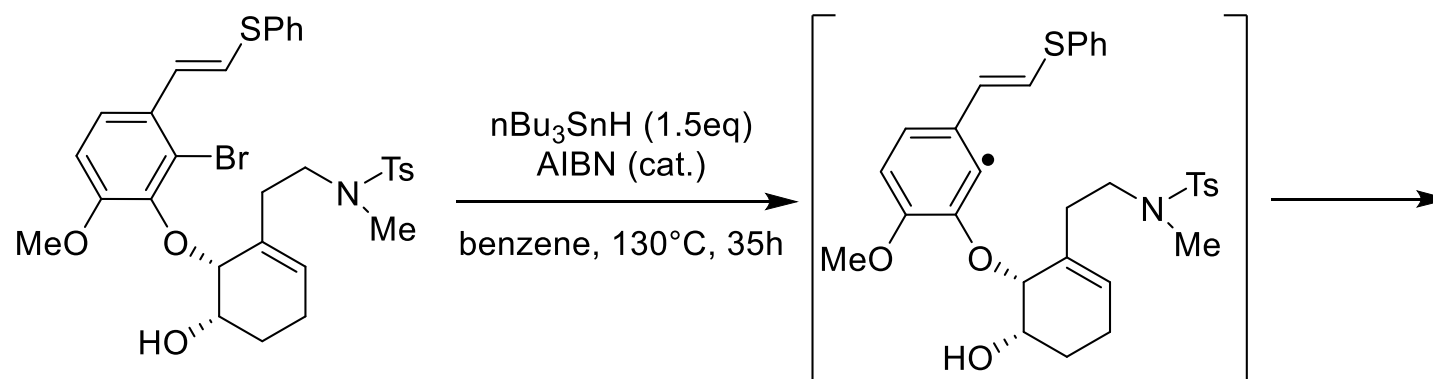
- Fokas and Parker's total synthesis of (-)-Morphine (2006)



III/ Radical Domino Reactions

III-2/ Aryl radical cyclization

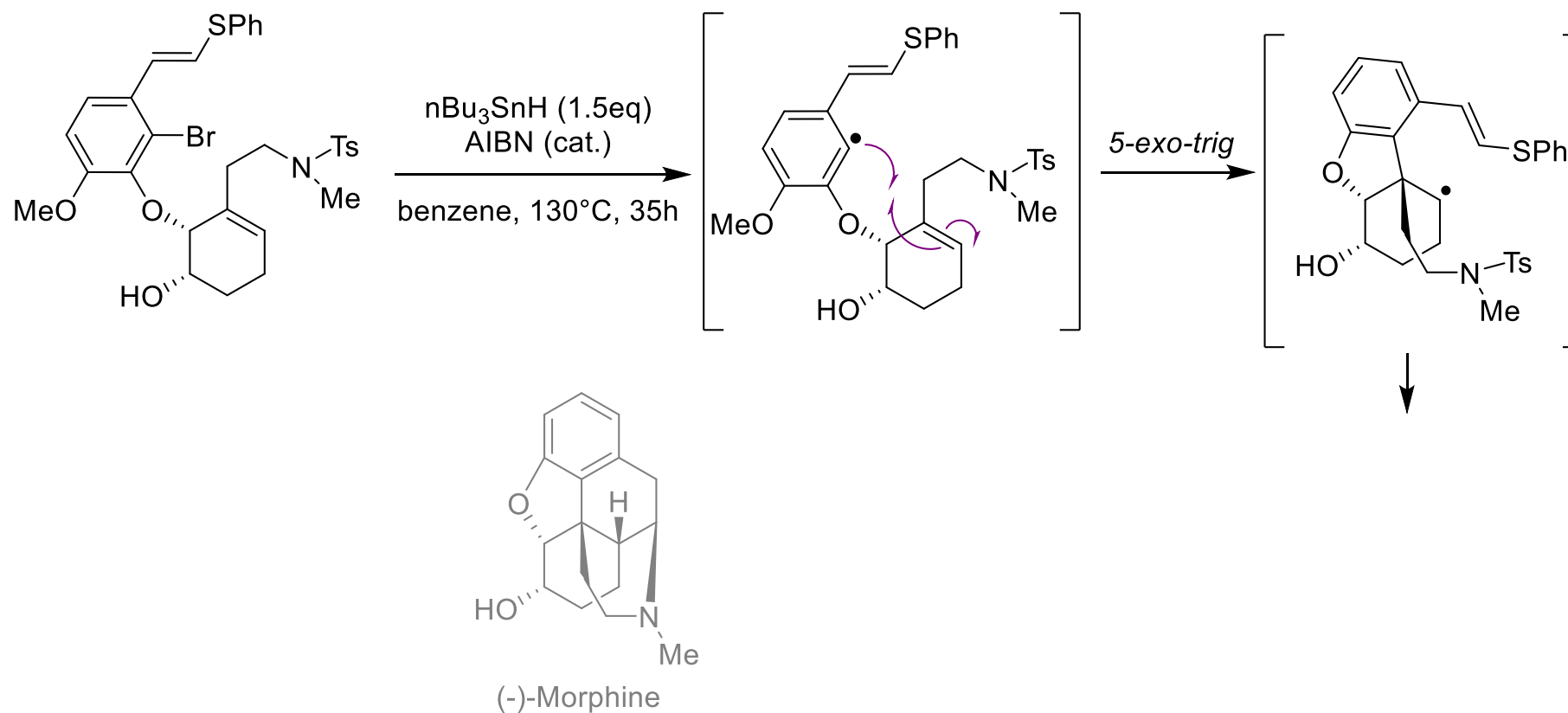
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III-2/ Aryl radical cyclization

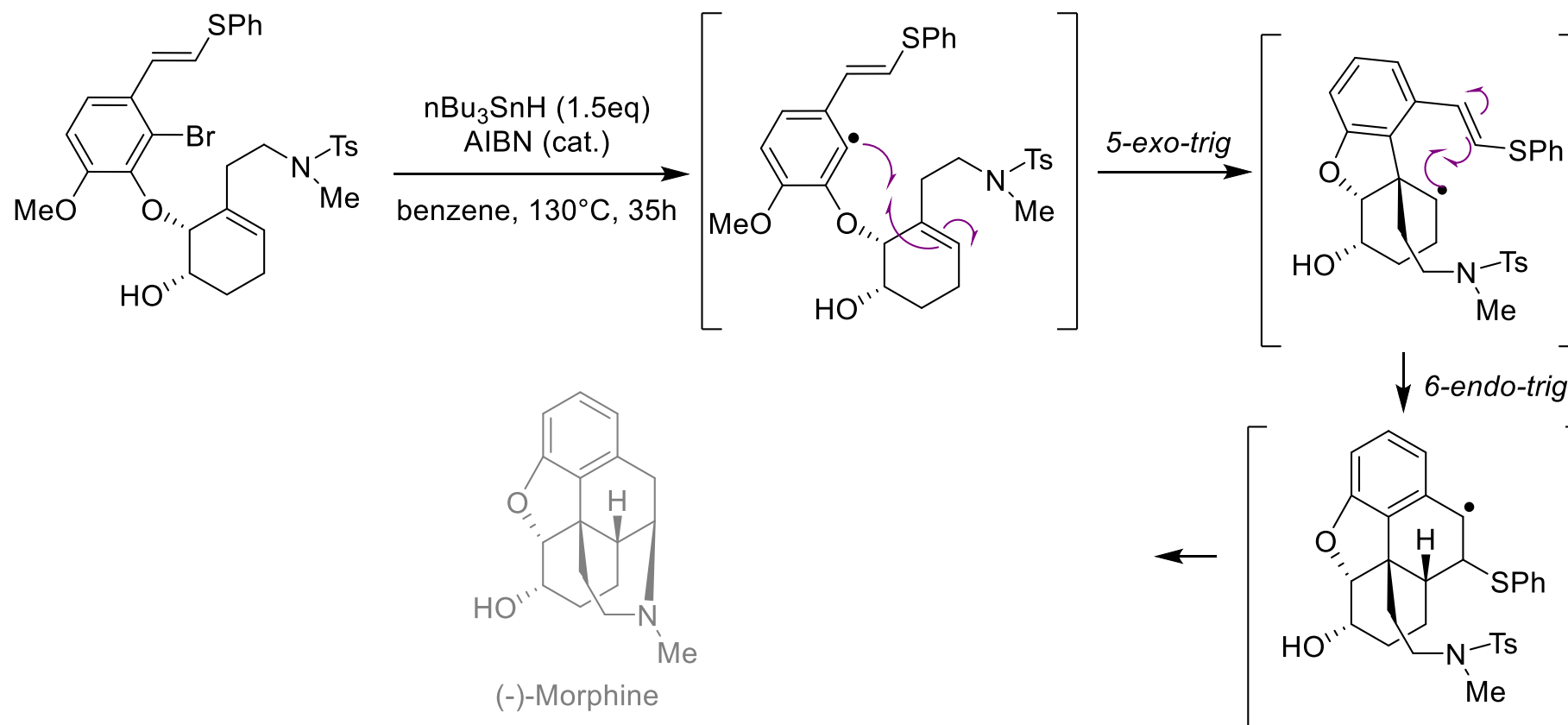
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III/ Radical Domino Reactions

III-2/ Aryl radical cyclization

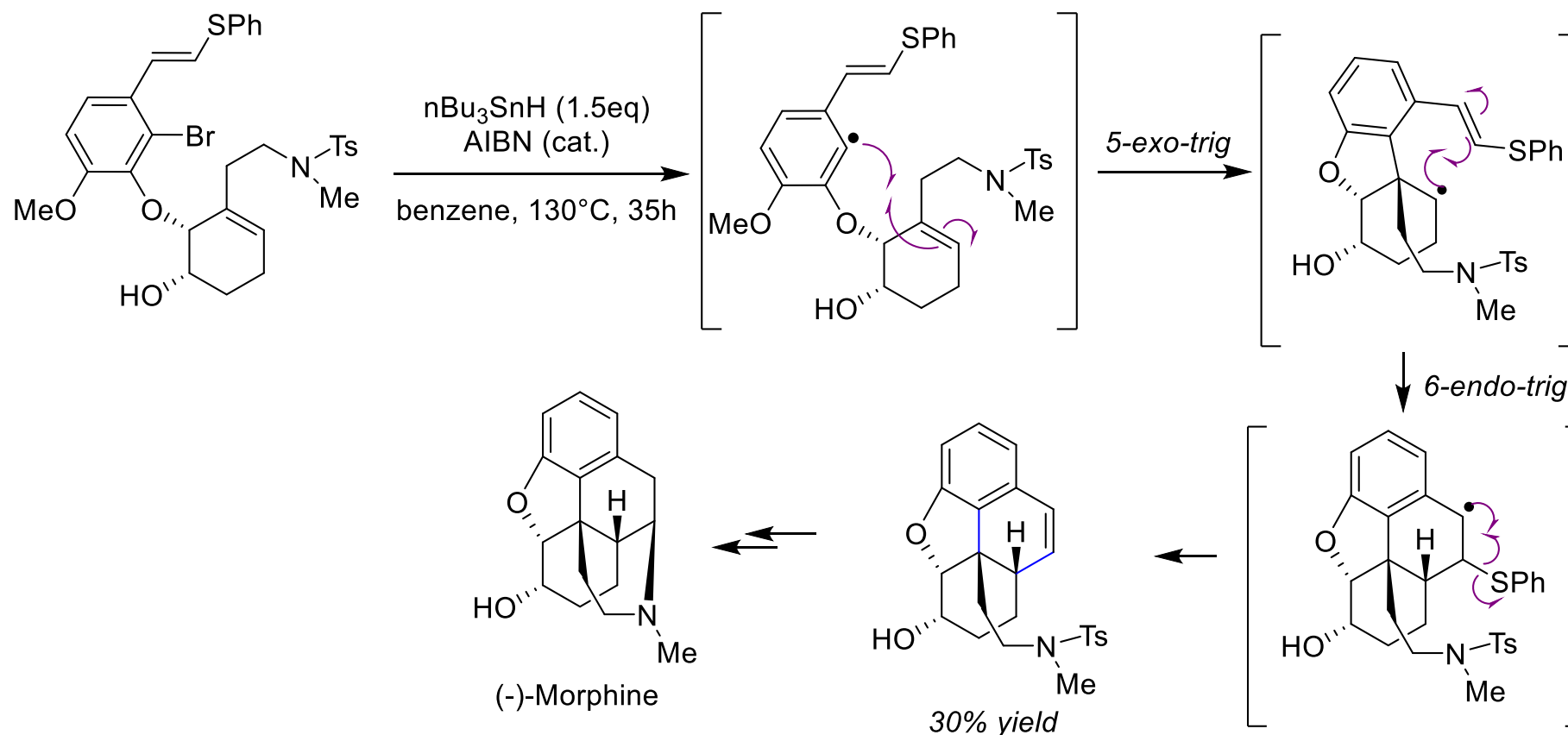
- Fokas and Parker's total synthesis of (-)-Morphine (2006)



III/ Radical Domino Reactions

III-2/ Aryl radical cyclization

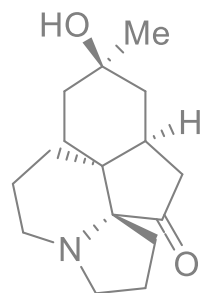
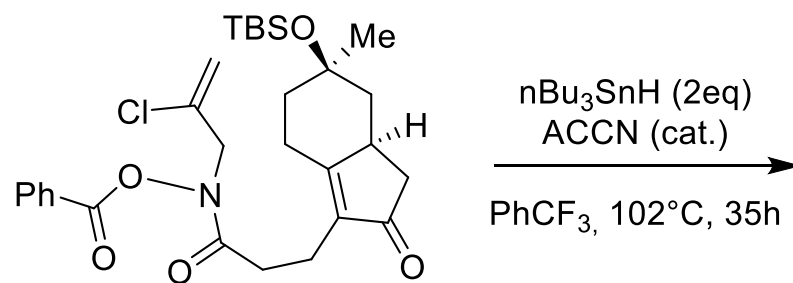
- Fokas and Parker's total synthesis of (-)-Morphine (2006)



III/ Radical Domino Reactions

III-3/ Nitrogen-centered radical cyclization

- Zard's total synthesis of 13-Deoxyserrattine (2002)

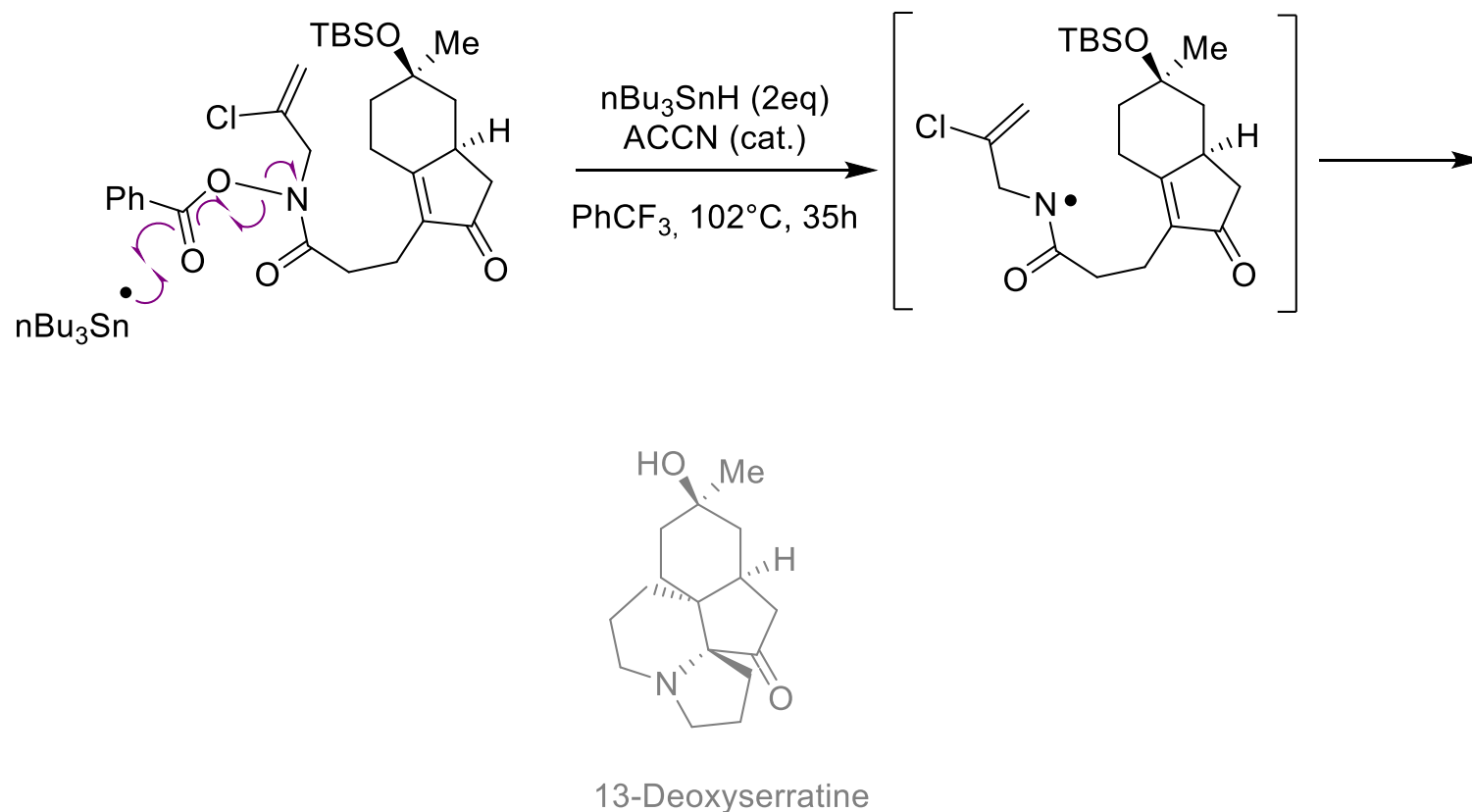


13-Deoxyserrattine

III/ Radical Domino Reactions

III-3/ Nitrogen-centered radical cyclization

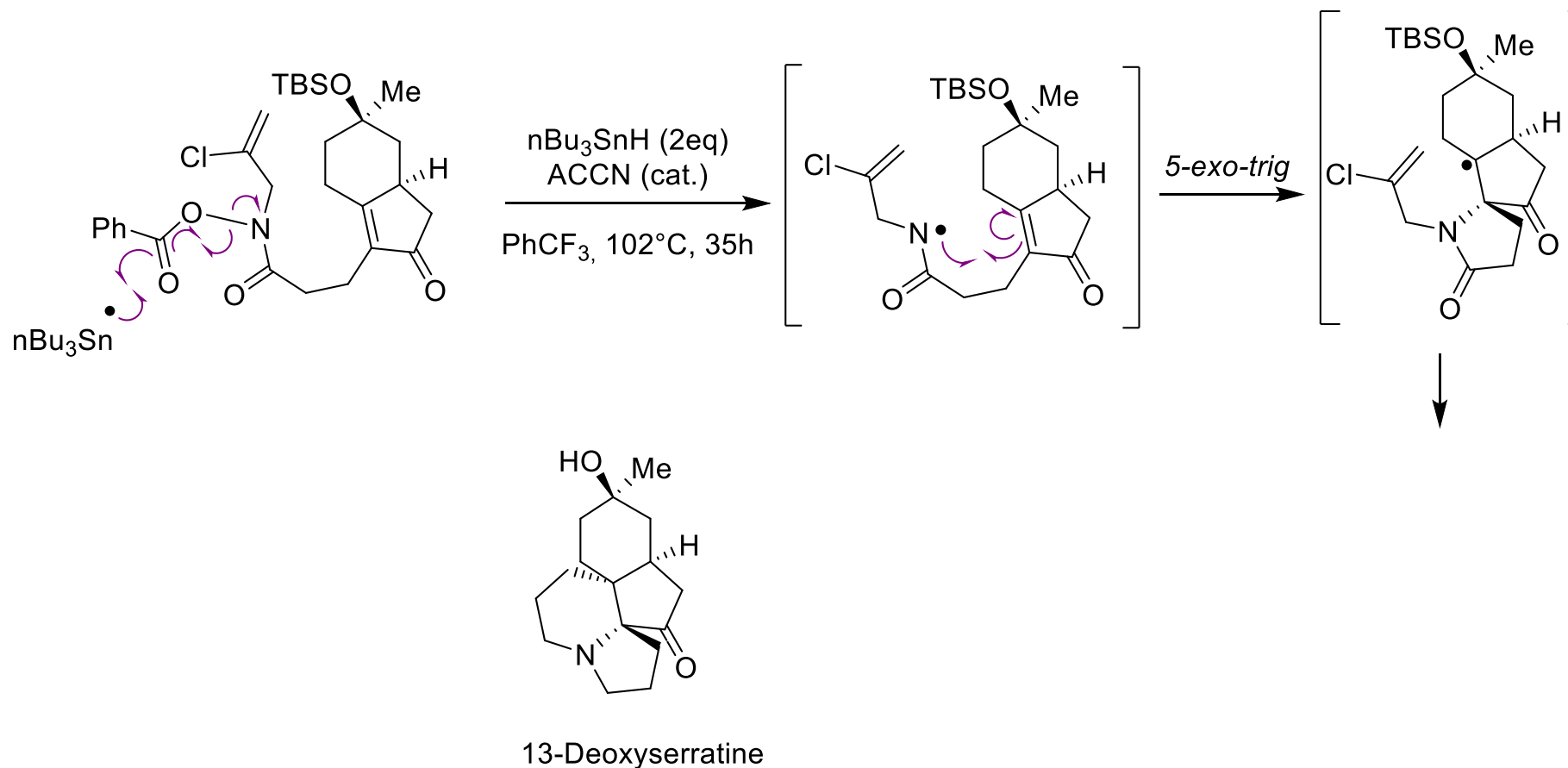
- Zard's total synthesis of 13-Deoxyserrattine (2002)



III/ Radical Domino Reactions

III-3/ Nitrogen-centered radical cyclization

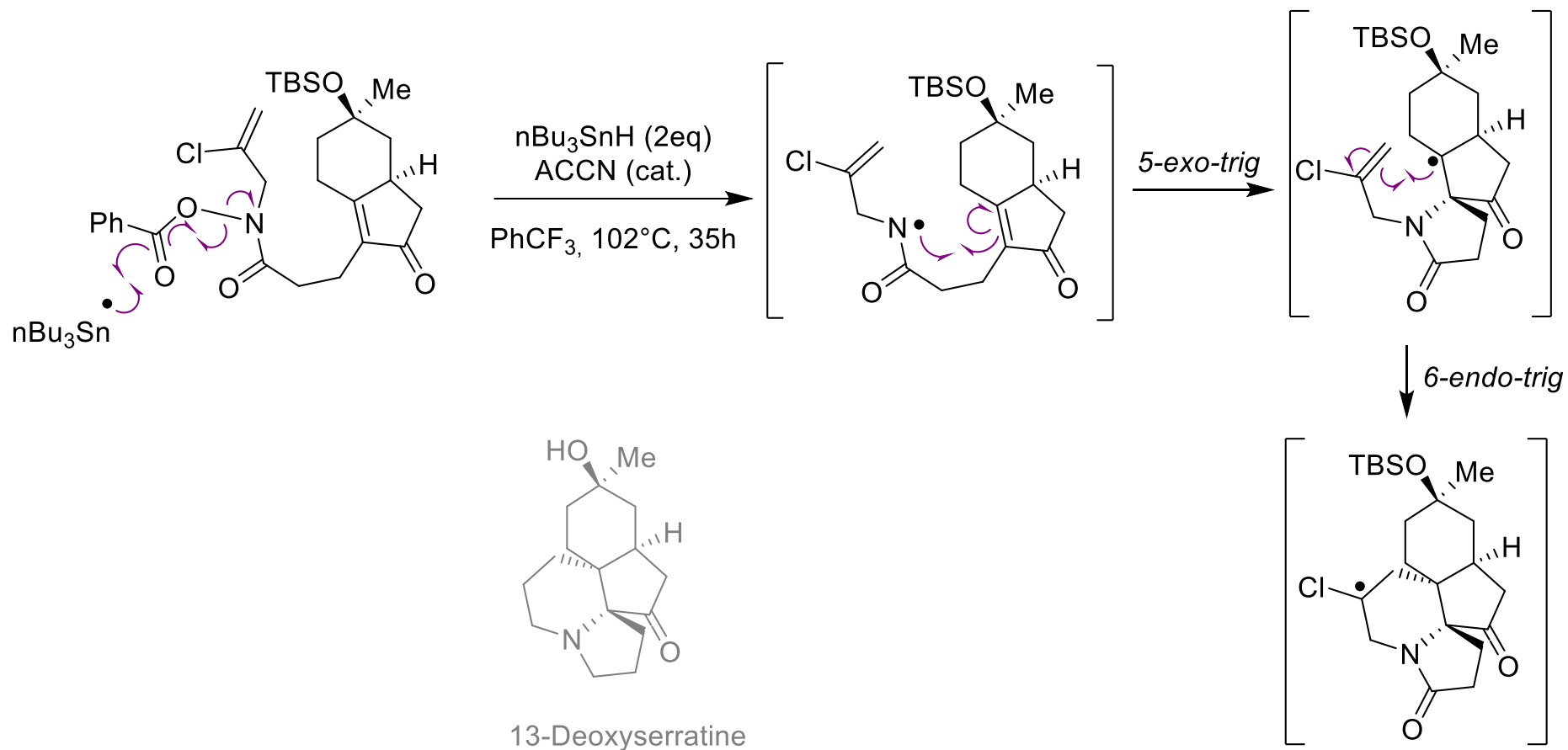
► Zard's total synthesis of 13-Deoxyserrattine (2002)



III/ Radical Domino Reactions

III-3/ Nitrogen-centered radical cyclization

► Zard's total synthesis of 13-Deoxyserrattine (2002)



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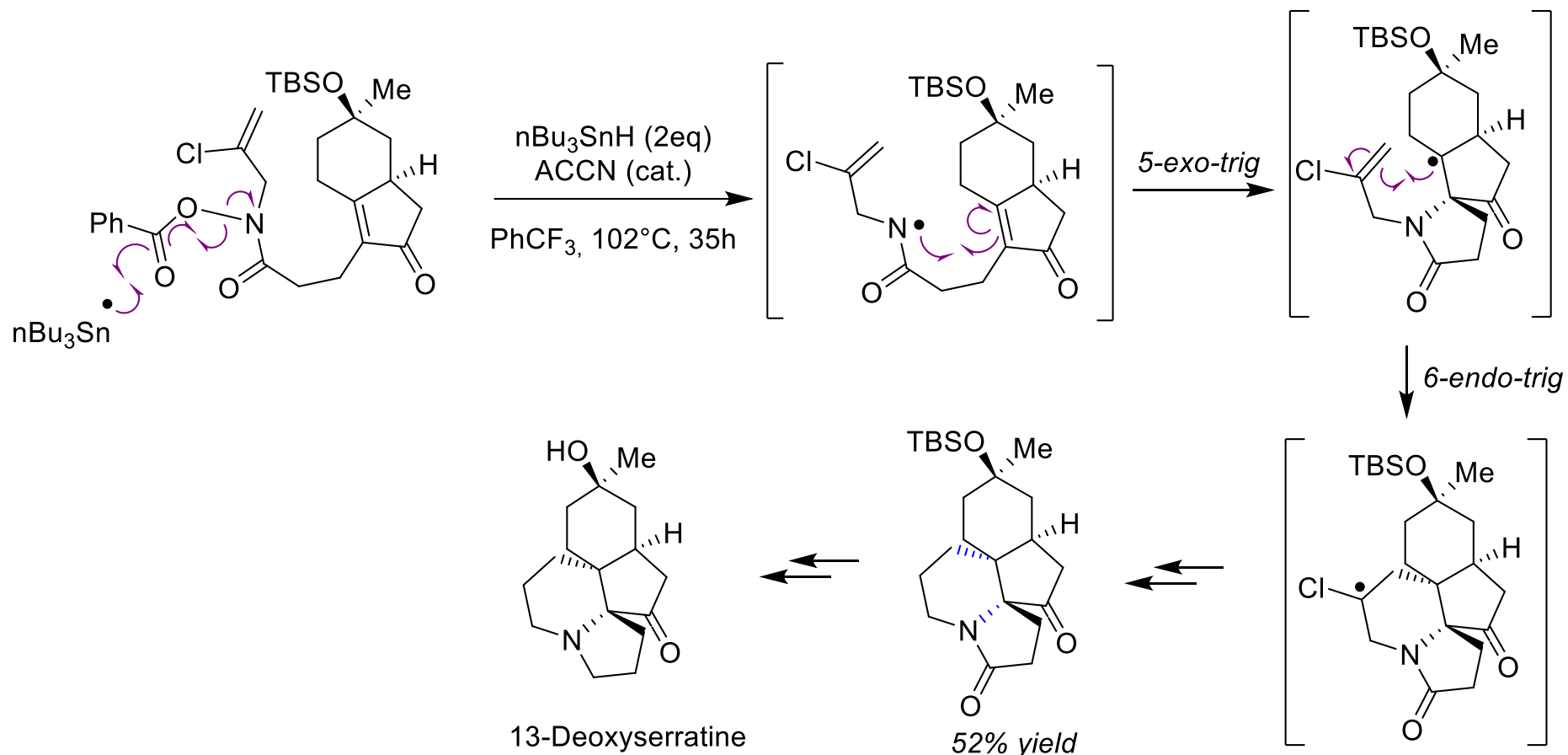
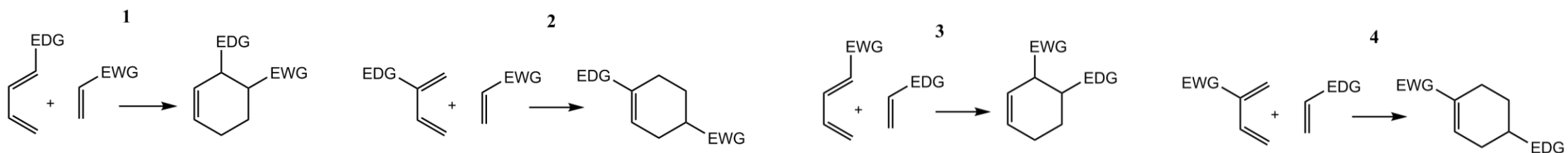


Table of Contents

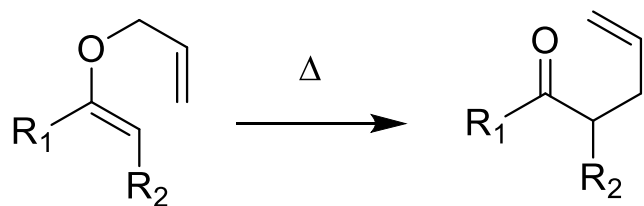
- Introduction and Definitions
- I/ Electrophilic Domino Reactions
- II/ Nucleophilic Domino Reactions
- III/ Radical Domino Reactions
- **IV/ Pericyclic Domino Reactions**
- V/ Transition Metal Catalyzed Domino Reactions
- Conclusion

IV/ Pericyclic Domino Reactions

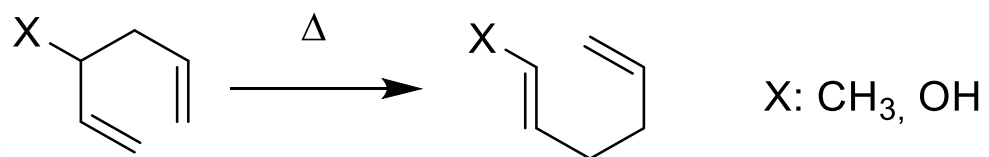
► Diels-Alder reaction



► Claisen rearrangement



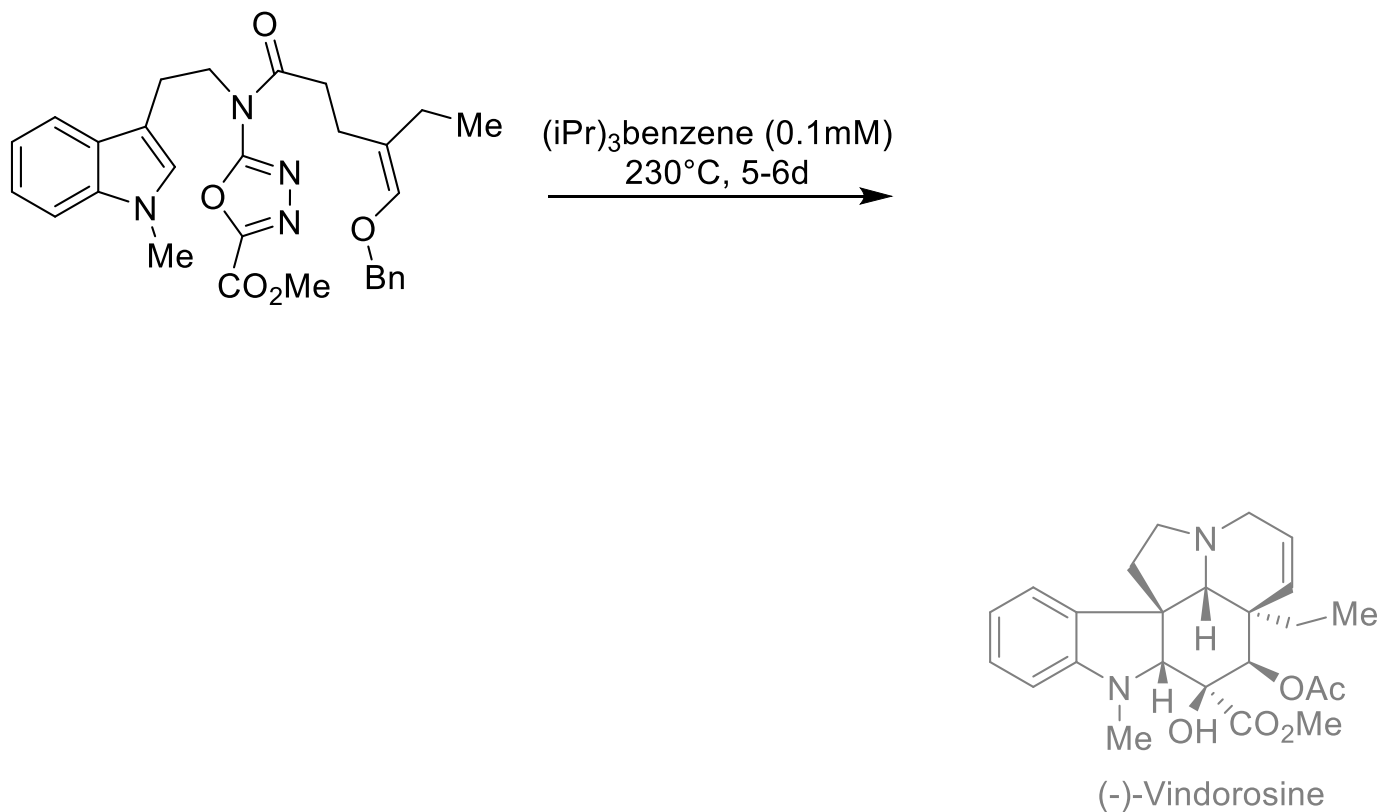
► Cope rearrangement



IV/ Pericyclic Domino Reactions

IV-1/ [4+2] / [3+2] domino process

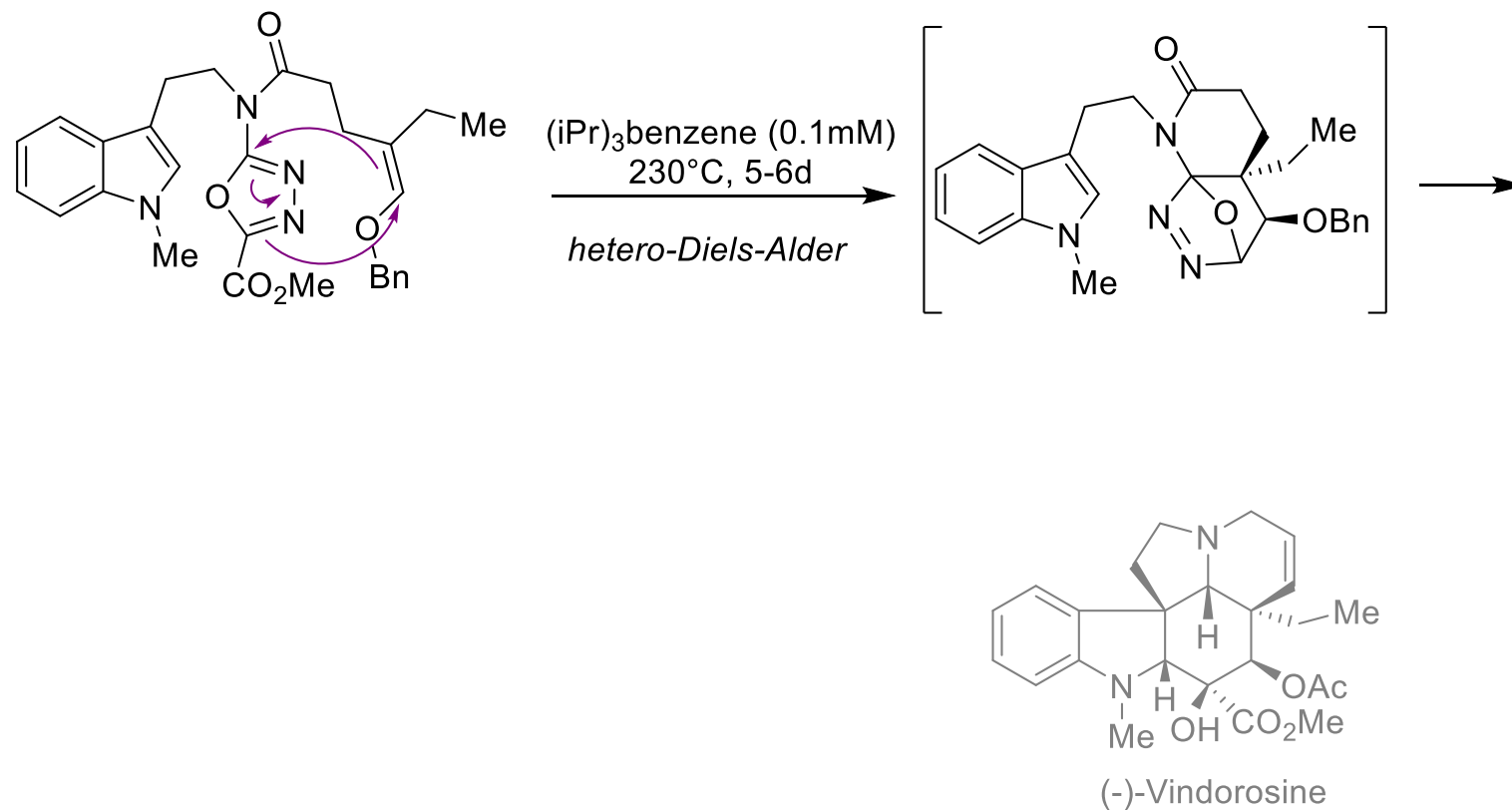
► Boger's total synthesis of (-)-Vindorosine (2006)



IV/ Pericyclic Domino Reactions

IV-1/ [4+2] / [3+2] domino process

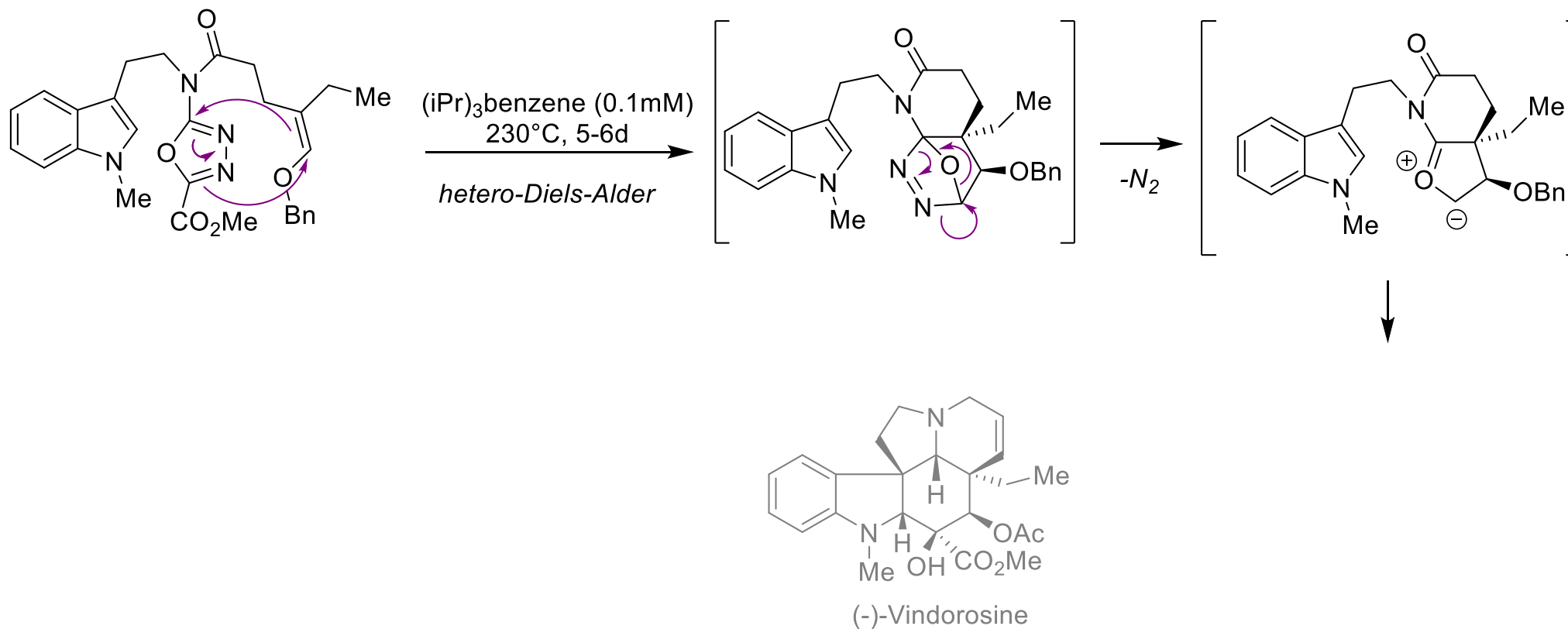
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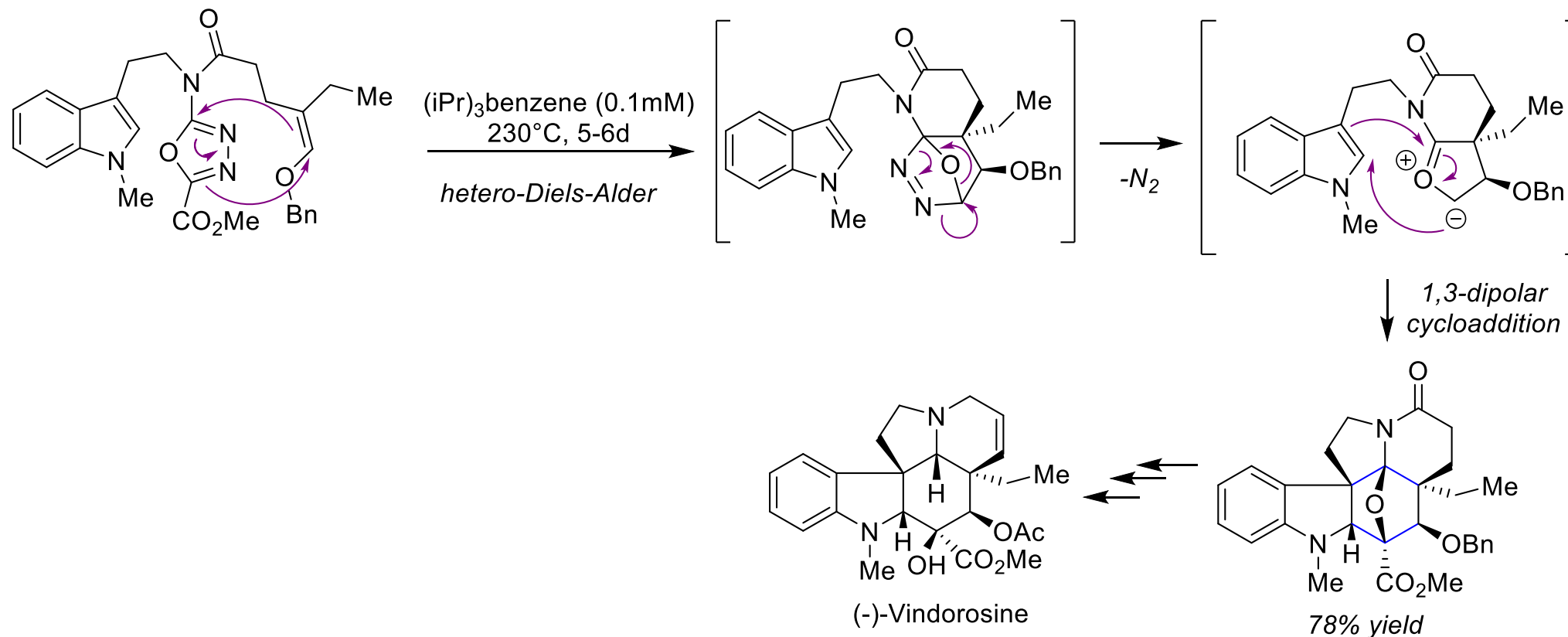
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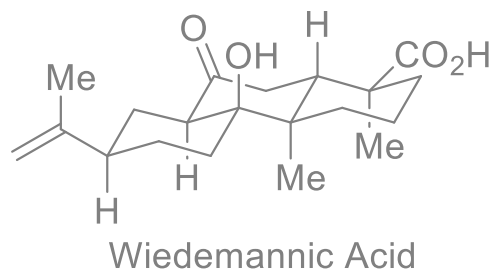
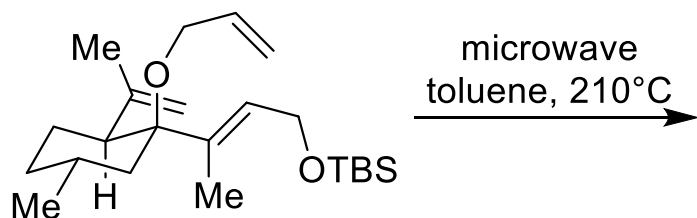
► Boger's total synthesis of (-)-Vindorosine (2006)



IV/ Pericyclic Domino Reactions

IV-2/ [3,3] / [3,3] / ene domino process

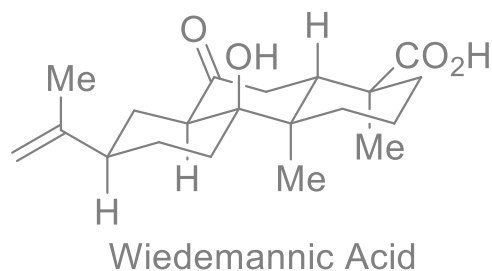
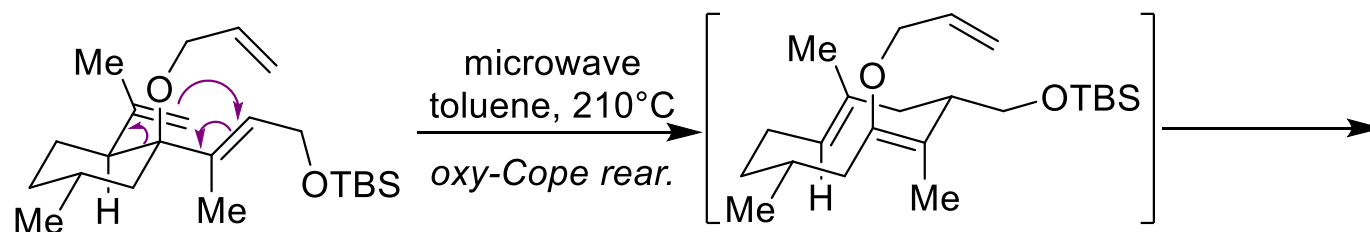
- Barriault's synthesis of Wiedemannic Acid skeleton (2004)



IV/ Pericyclic Domino Reactions

IV-2/ [3,3] / [3,3] / ene domino process

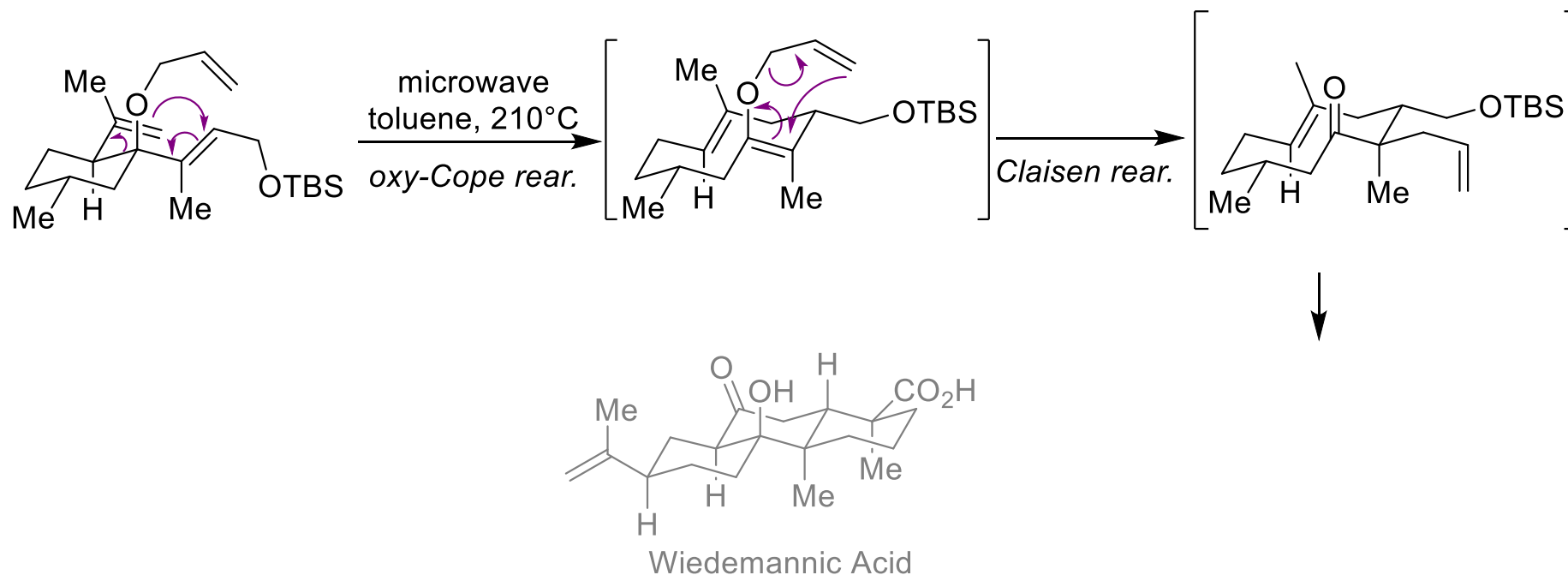
- Barriault's synthesis of Wiedemannic Acid skeleton (2004)



IV/ Pericyclic Domino Reactions

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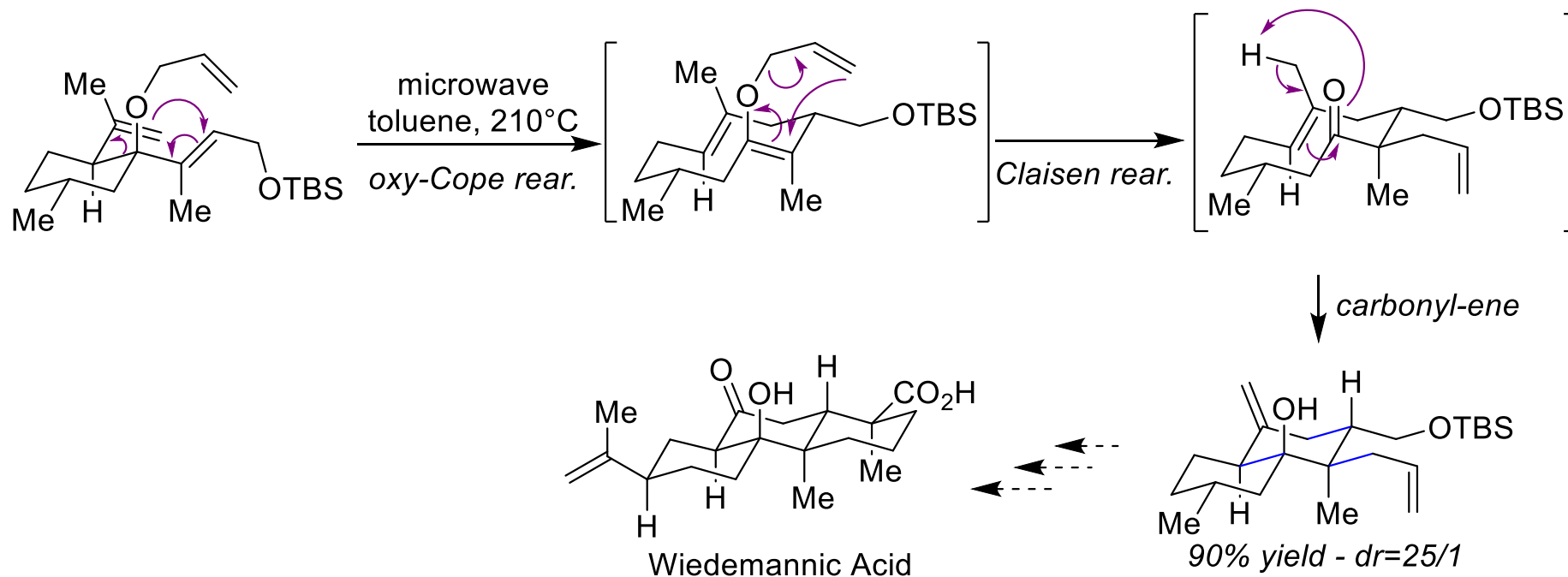


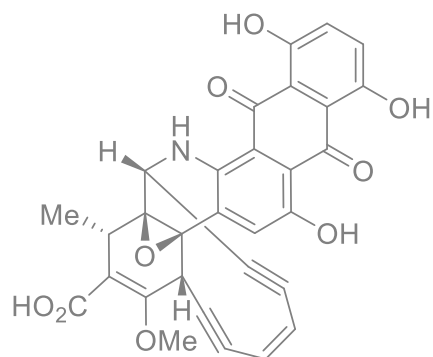
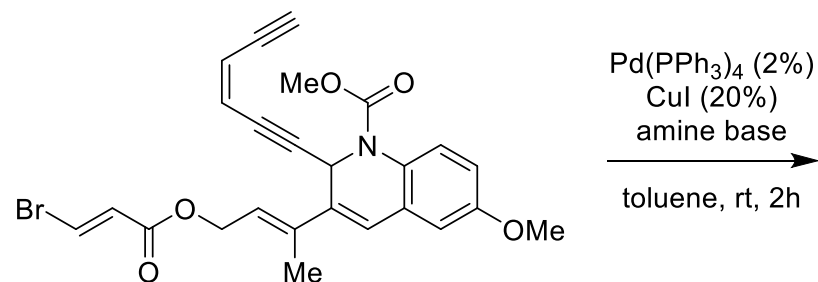
Table of Contents

- Introduction and Definitions
- I/ Electrophilic Domino Reactions
- II/ Nucleophilic Domino Reactions
- III/ Radical Domino Reactions
- IV/ Pericyclic Domino Reactions
- **V/ Transition Metal Catalyzed Domino Reactions**
- Conclusion

V/Transition Metal Catalyzed Domino Reactions

V-1/ Palladium-Copper catalyst

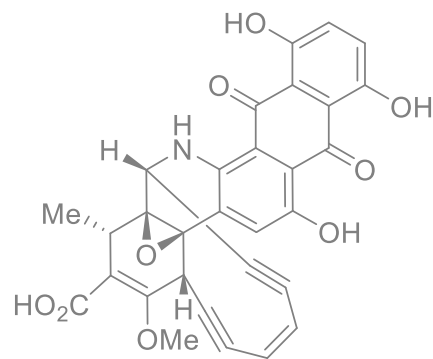
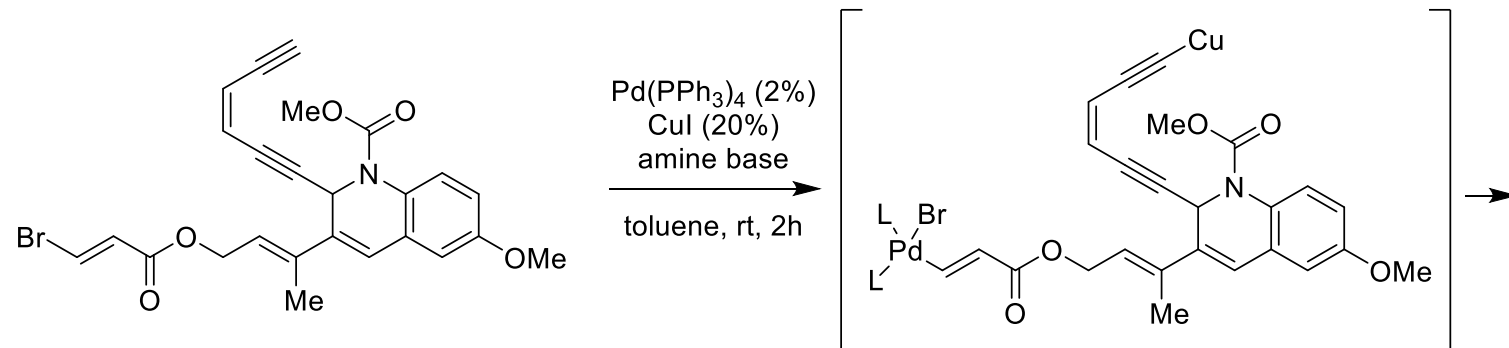
- Schreiber's total synthesis of Dynemicin A (1993)



V/Transition Metal Catalyzed Domino Reactions

V-1/ Palladium-Copper catalyst

- Schreiber's total synthesis of Dynemicin A (1993)

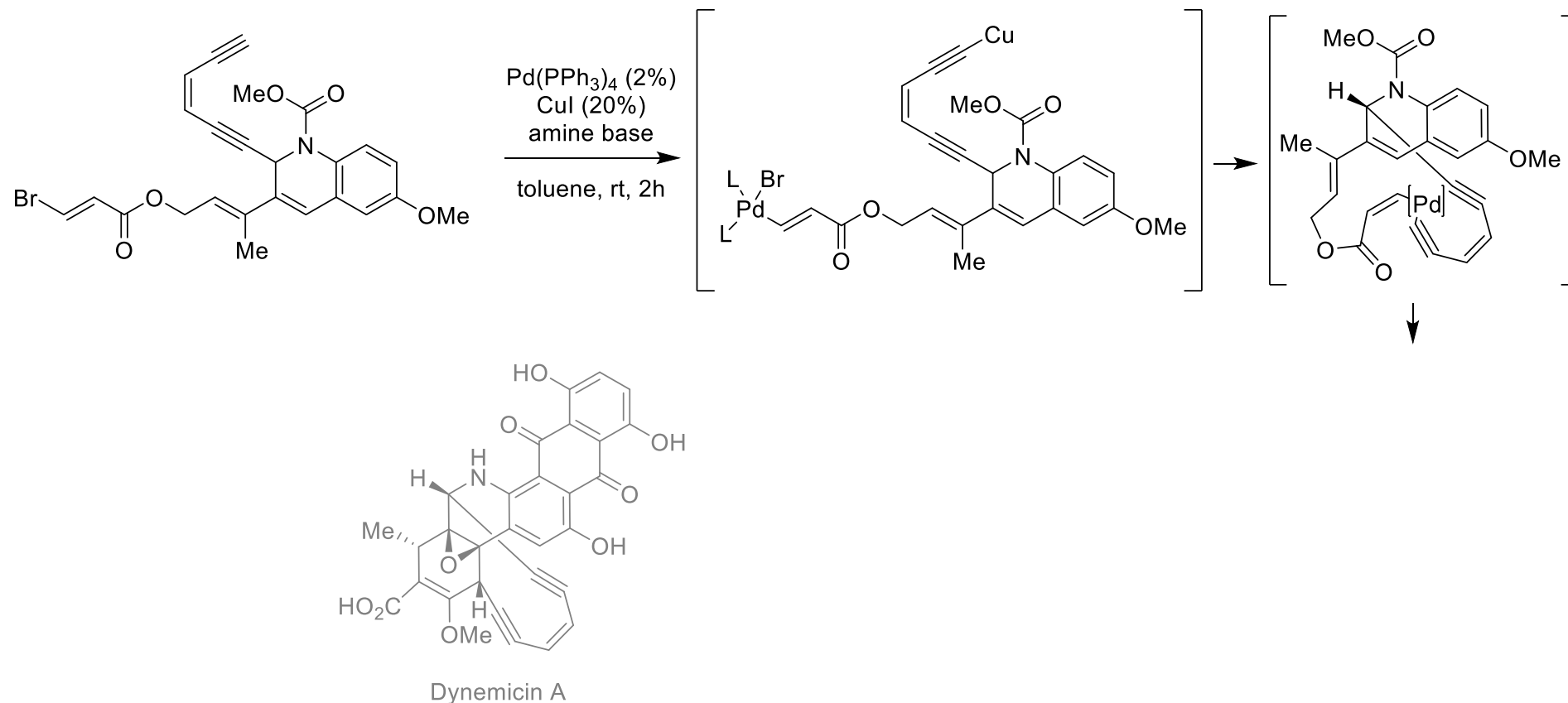


Dynemicin A

V/Transition Metal Catalyzed Domino Reactions

V-1/ Palladium-Copper catalyst

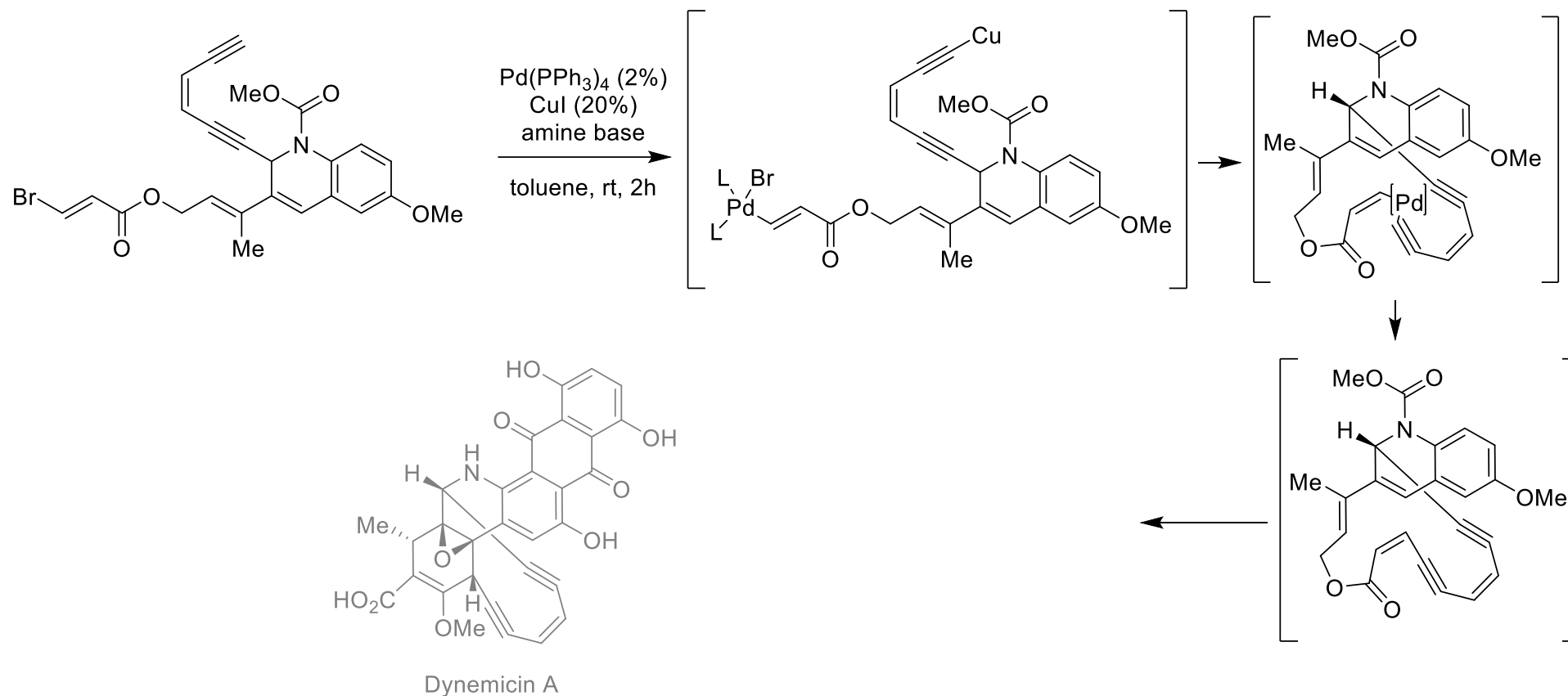
- Schreiber's total synthesis of Dynemicin A (1993)



V/Transition Metal Catalyzed Domino Reactions

V-1/ Palladium-Copper catalyst

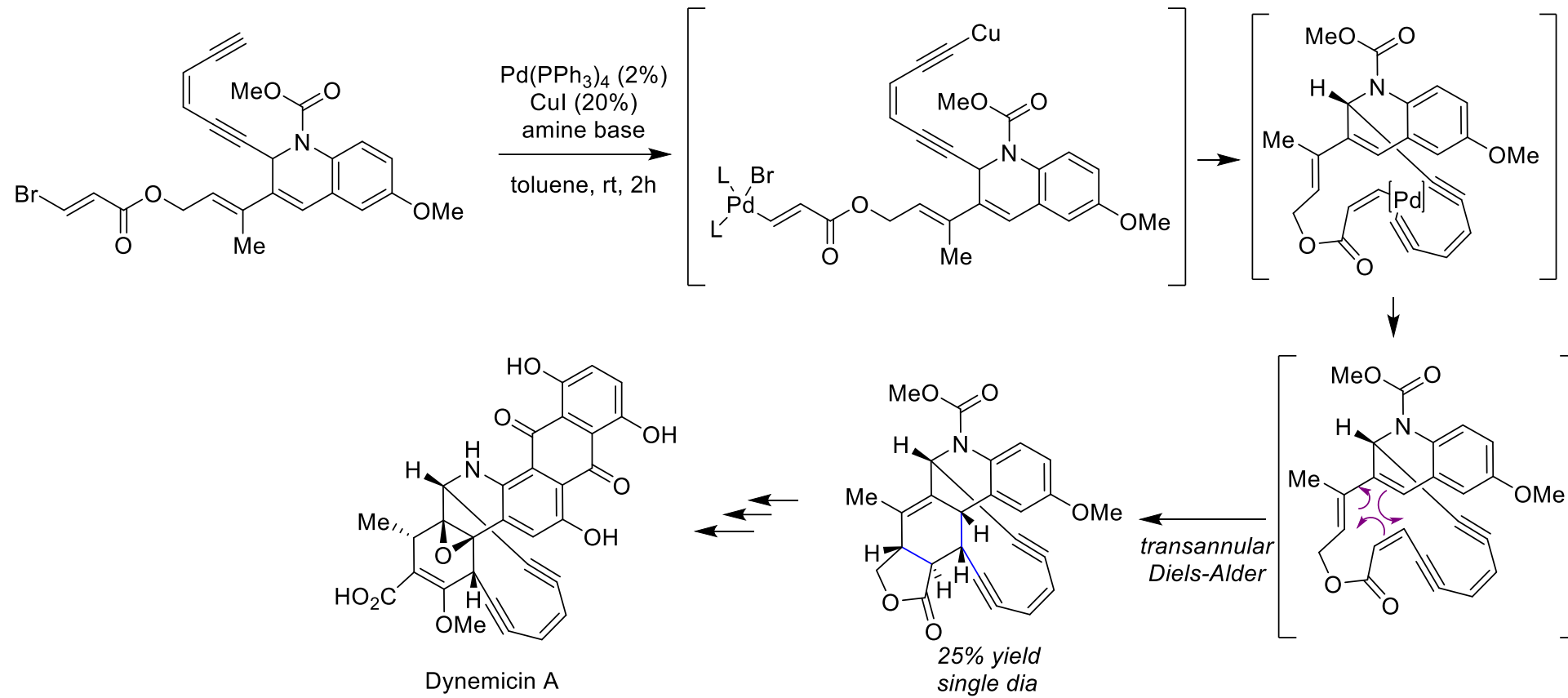
► Schreiber's total synthesis of Dynemicin A (1993)



V/Transition Metal Catalyzed Domino Reactions

V-1/ Palladium-Copper catalyst

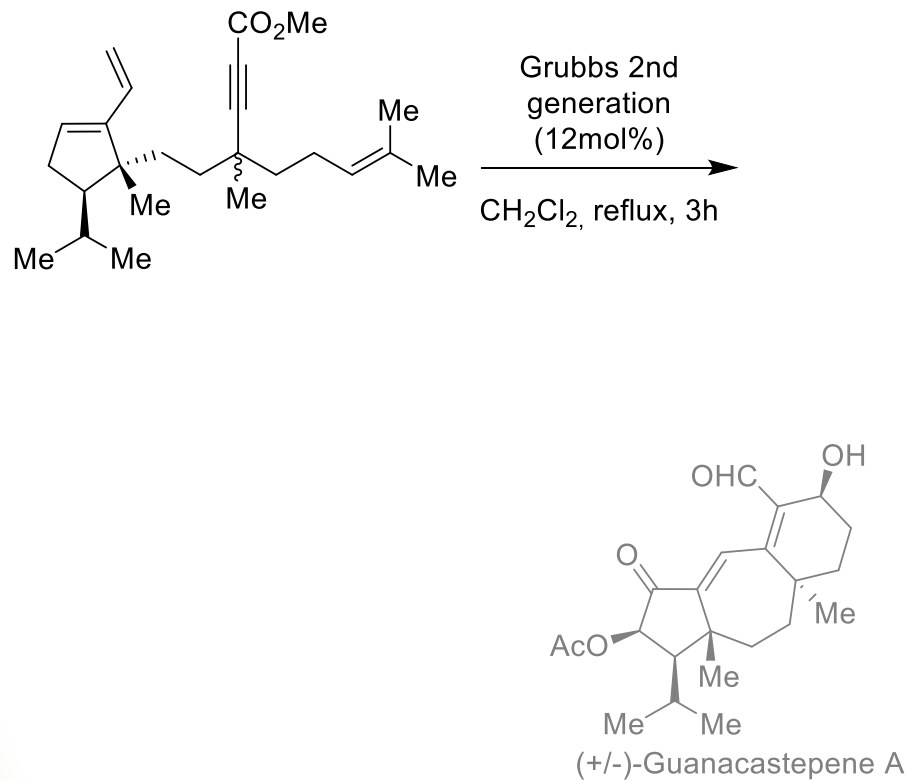
► Schreiber's total synthesis of Dynemicin A (1993)



V/Transition Metal Catalyzed Domino Reactions

V-2/ Ruthenium catalyst

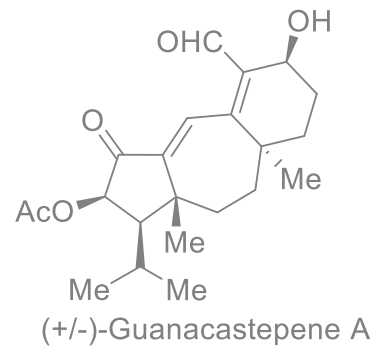
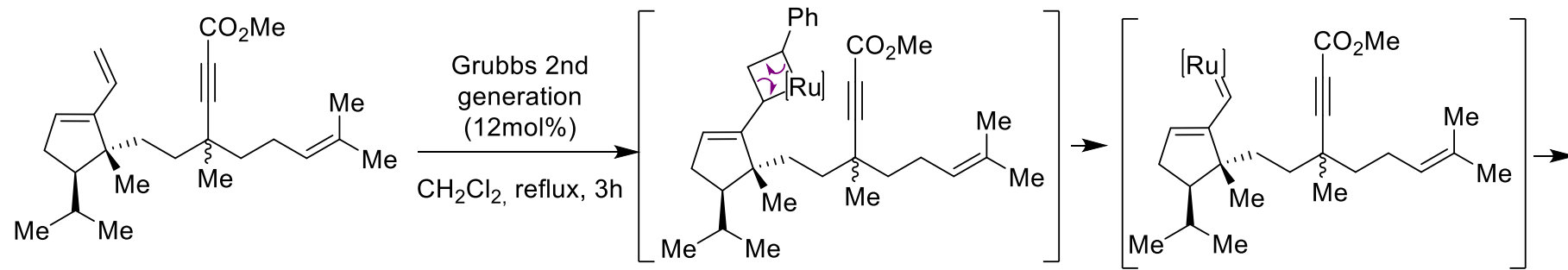
- ▶ Hanna's formal synthesis of (+/-)-Guanacastepene A (2004)



V/Transition Metal Catalyzed Domino Reactions

V-2/ Ruthenium catalyst

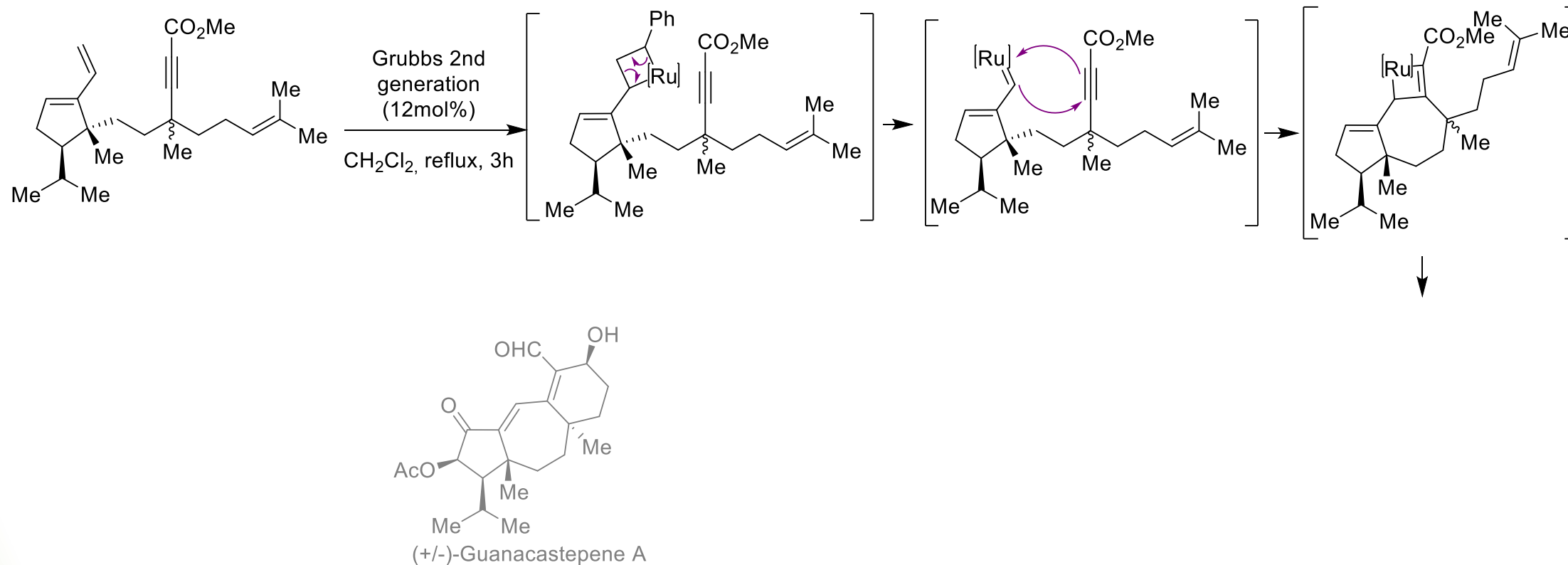
- ▶ Hanna's formal synthesis of (+/-)-Guanacastepene A (2004)



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V-2/ Ruthenium catalyst

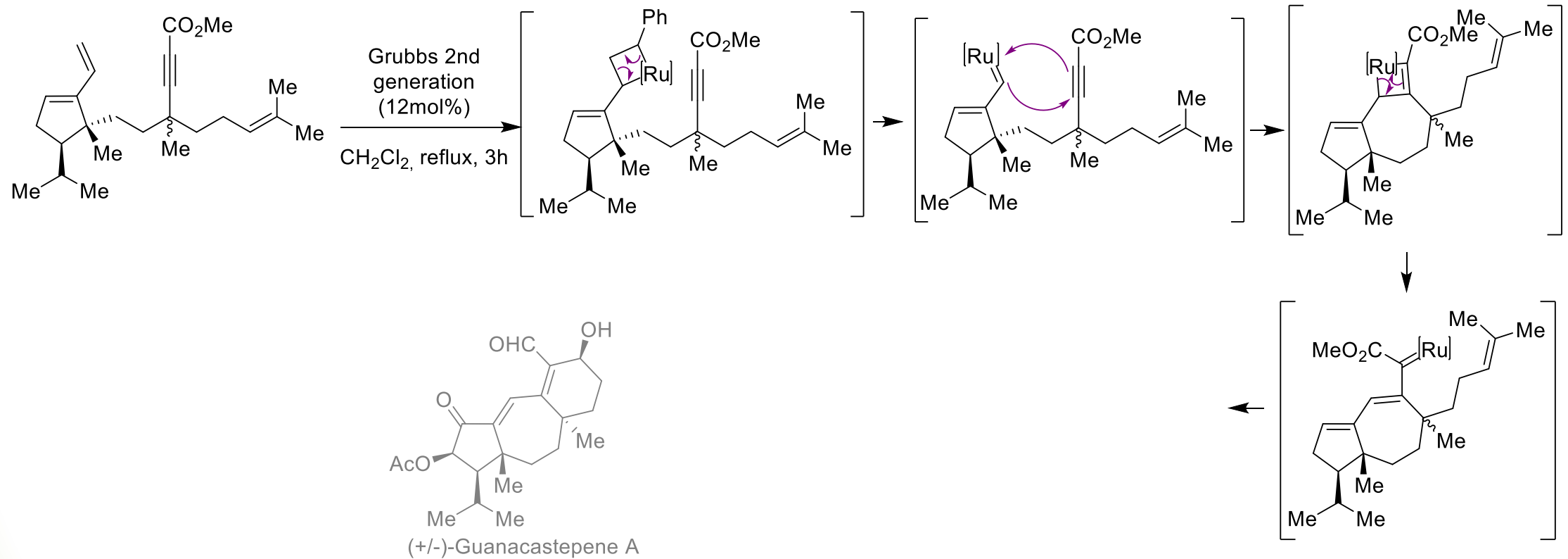
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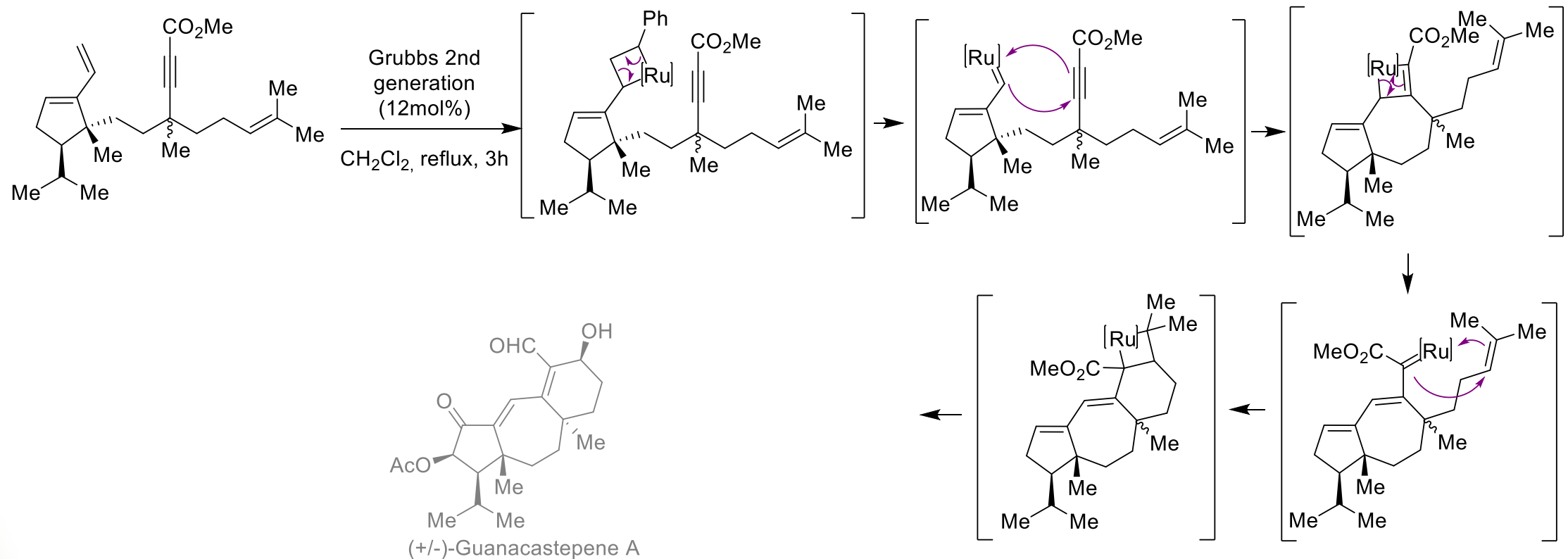
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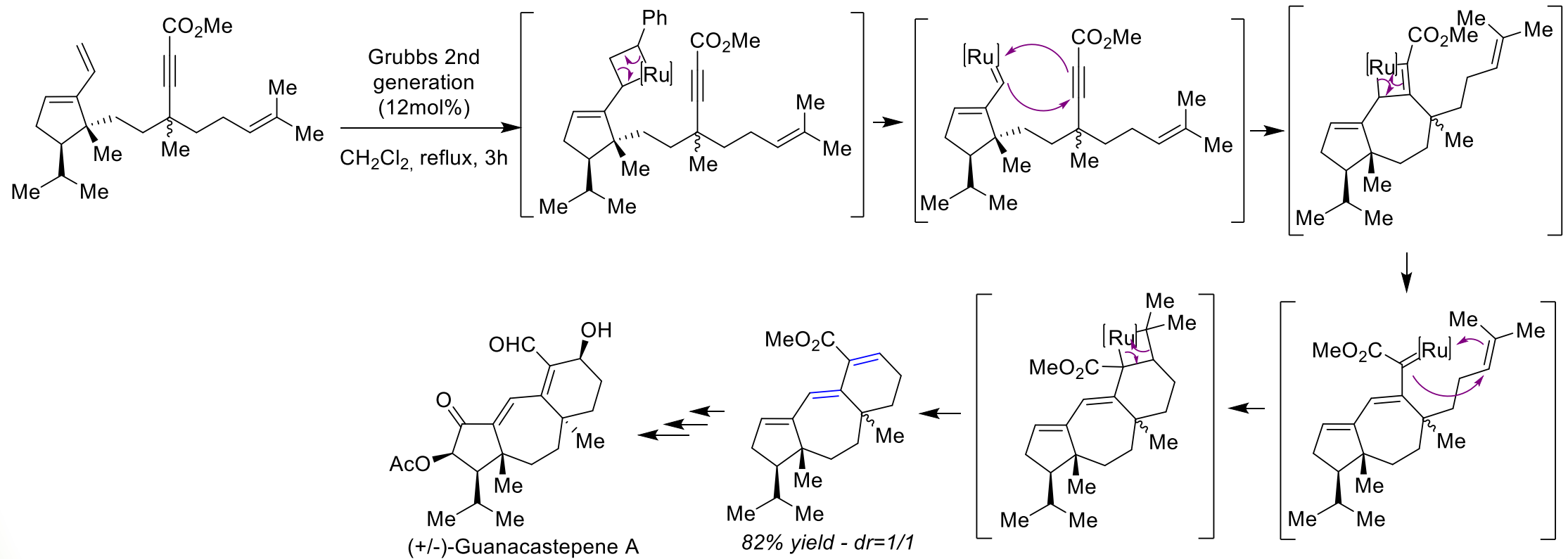
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Conclusion

- Advantages:

- Economy

- Increase of molecular complexity

- Drawbacks:

- Often take time to find suitable precursors and conditions to obtain the desired domino reaction (reduce the economy advantage...)

- Often only the thermodynamical product is obtain during a domino process

- Outlook:

- Very few example of catalytic enantioselective domino reaction --> Future of domino?

Thanks for your attention!