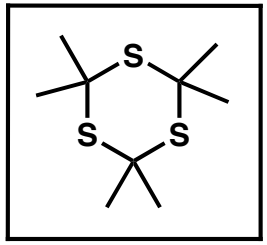
Sulfur-Facilitated Organic Synthesis

Andrew McClory
Monday March 16, 2009
8:00 pm, 147 Noyes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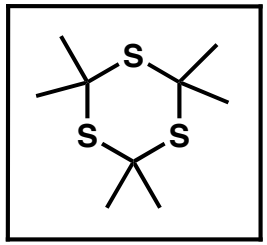
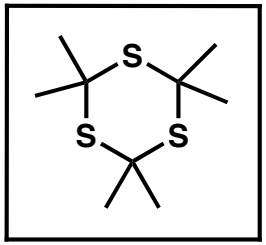


'Attempts to make thioacetone by the cracking of trithioacetone gave rise to an offensive smell which spread rapidly over a great area of the town causing fainting, vomiting and a panic evacuation'...'the laboratory work was abandoned.'

-Researcher, Freiburg, 1889



'Attempts to make thioacetone by the cracking of trithioacetone gave rise to an offensive smell which spread rapidly over a great area of the town causing fainting, vomiting and a panic evacuation'...'the laboratory work was abandoned.'

-Researcher, Freiburg, 1889

'Recently we found ourselves with an odour problem beyond our worst expectations. During early experiments, a stopper jumped from a bottle of residues, and, although replaced at once, resulted in an immediate complaint of nausea and sickness from colleagues working in a building two hundred yards away. Two of our chemists who had done no more than investigate the cracking of minute amounts of trithioacetone found themselves the object of hostile stares in a restaurant and suffered the humiliation of having a waitress spray the area around them with a deodorant. The odours defied the expected effects of dilution since workers in the laboratory did not find the odours intolerable...and genuinely denied responsibility since they were working in closed systems. To convince them otherwise, they were dispersed with other observers around the laboratory, at distances up to a quarter of a mile, and one drop of either acetone gem-dithiol or the mother liquors from crude thioacetone crystallizations were placed on a watch glass in a fume cupboard. The odour was detected downwind in seconds.'

-Esso Researcher, Oxford, 1967

Seminar Overview

1. Nomenclature

2. Selected Properties

3. Elimination

- **A. Burgess**
- **B. Martin**
- **C. Sulfoxide**
- **D. Chugaev**

4. Radical Reaction

- **A. Barton**
- **B. Zard**

5. C-C Bond Formation

- **A. Nature; Fukuyama**
- **B. Acyl Anion Equivalents**
- **C. Sulfur Ylides**
- **E. Chiral Auxiliaries**

6. Olefination

- **A. Julia**
- **B. Ramberg-Backlund**
- **C. Eschenmoser**
- **D. Corey-Winter**
- **E. Nicolaou**

7. Oxidation

- **A. Kornblum**
- **B. Moffatt-Swern**

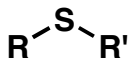
8. Functionalization

- **A. Mislow-Evans**
- **B. Pummerer**
- **C. DMTSF**
- **D. Diels-Alder**
- **E. Total Synthesis**

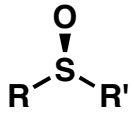
9. Allium Chemistry

Nomenclature of Organosulfur Compounds

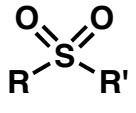
Thiol
(Mercaptan)



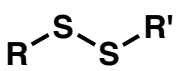
Sulfide
(Alkyl Mercaptan)



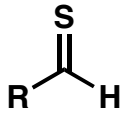
Sulfoxide



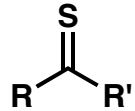
Sulfone



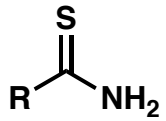
Disulfide



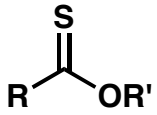
Thioaldehyde



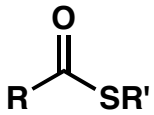
Thioketone



Thioamide



Thionoester



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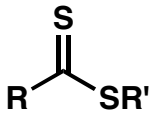
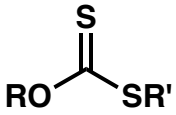
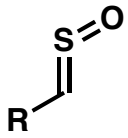


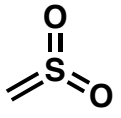
Dithioester



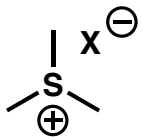
Xanthate Ester



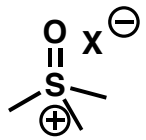
Sulfine



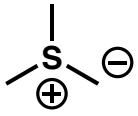
Sulfene



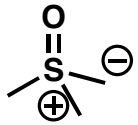
Sulfonium Salt



Sulfoxonium Salt

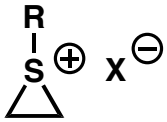


Sulfonium ylide



Sulfoxonium Ylide

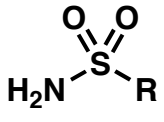
Thiirane
(Episulfide)



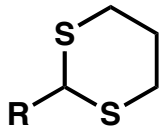
Episulfonium Salt

Episulfone

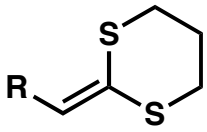
Sulfolane



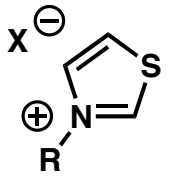
Sulfonamide



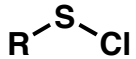
Thioacetal
(1,3-Dithiane)



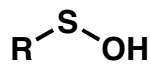
Ketene Thioacetal



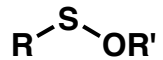
Thiazolium Salt



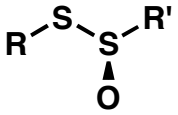
Sulfenyl Chloride



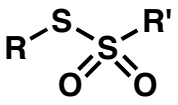
Sulfenic Acid



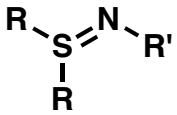
Sulfenate Ester



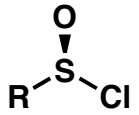
Thiosulfinate



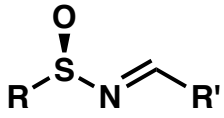
Thiosulfonate



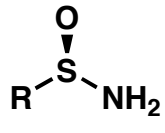
Sulfilimine



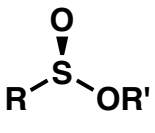
Sulfinyl Chloride



Sulfinimine



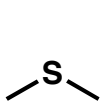
Sulfinamide



Sulfinate Ester

Selected Properties of Organosulfur Compounds

pKa Values (DMSO)



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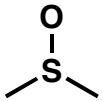

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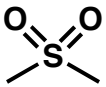

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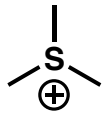

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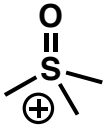

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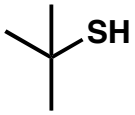

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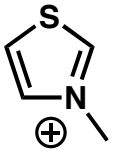

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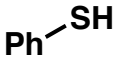

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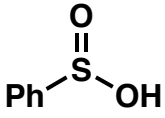

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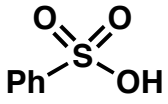


1

Bond Stengths (kcal/mol)

C=S: 120
C-S: 74
S-H: 87
S-S: 65

C=O: 177
C-O: 85
O-H: 110
O-O: 43

Bond Lengths (Å)

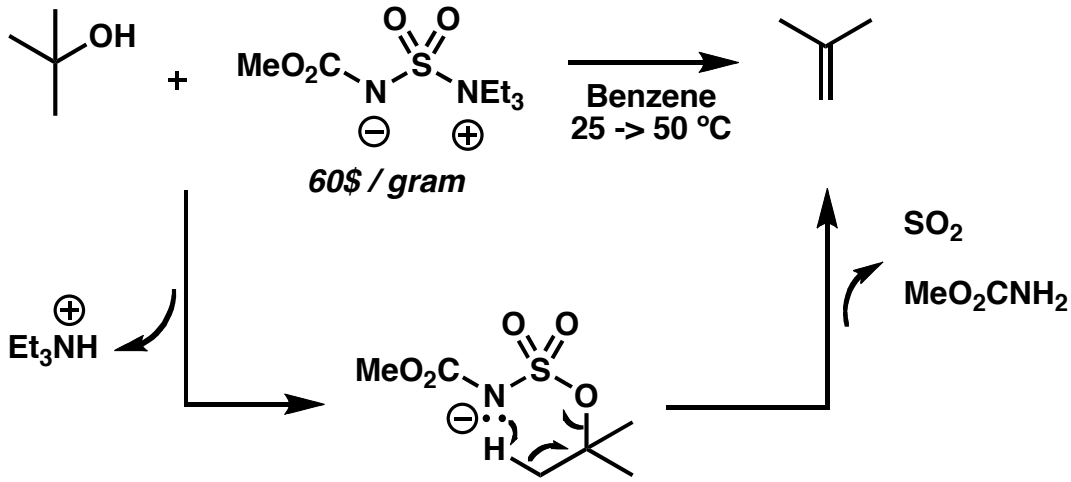
C=S: 1.6
C-S: 1.8
S-H: 1.4
S-S: 2.0
S=O: 1.5

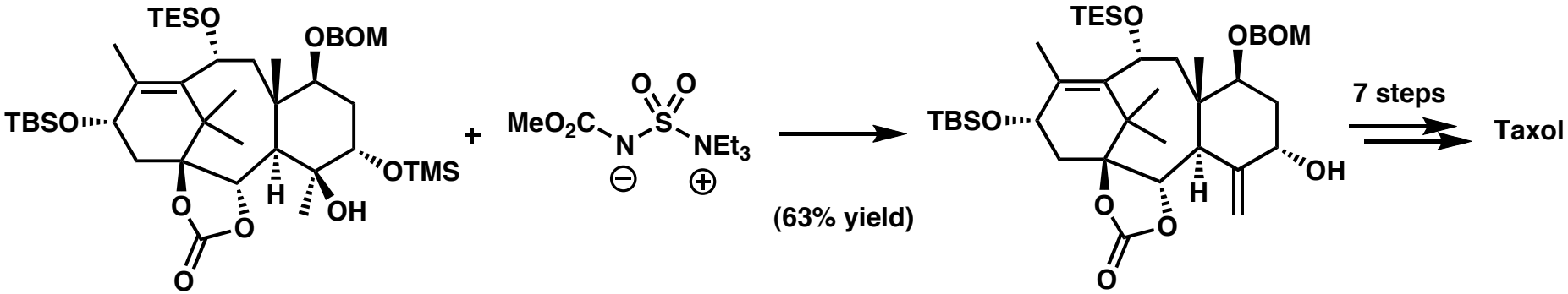
C=O: 1.2
C-O: 1.4
O-H: 1.0
O-O: 1.5

A-Values (kcal/mol)

Me: 1.7
OMe: 0.6
SMe: 0.7
SOMe: 1.2
SO_2Me: 2.5

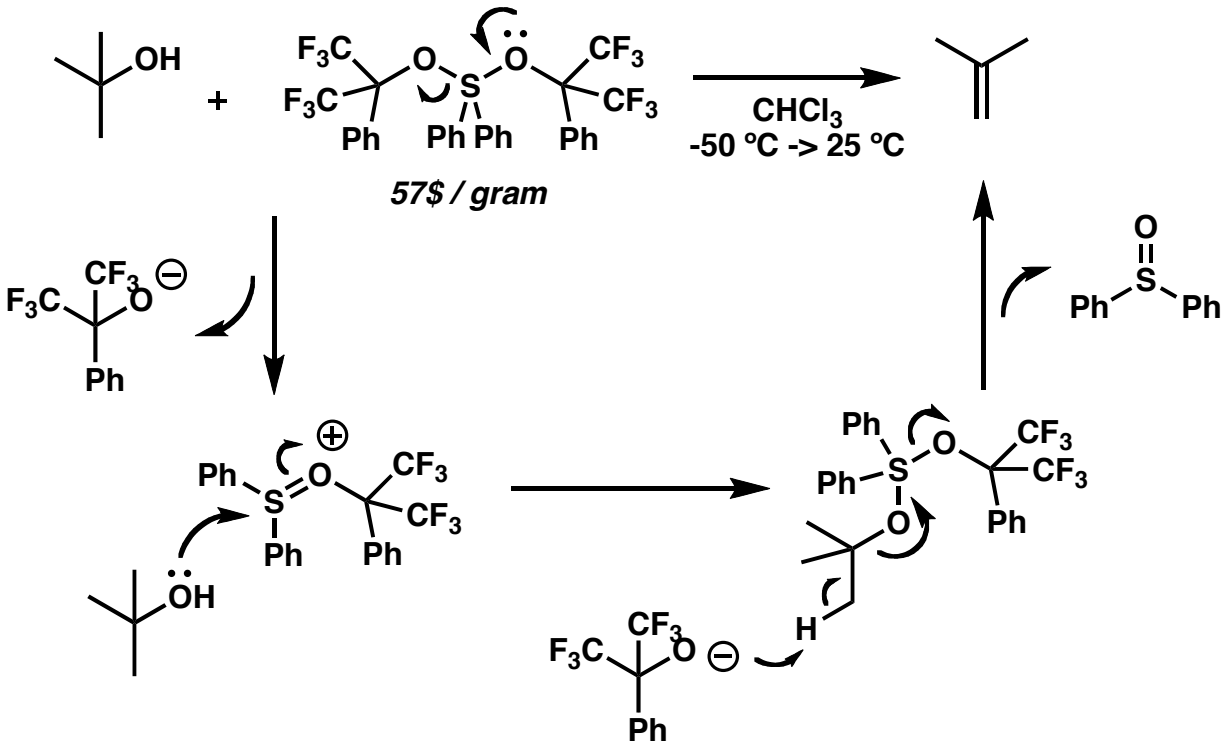
Elimination
Burgess Dehydration

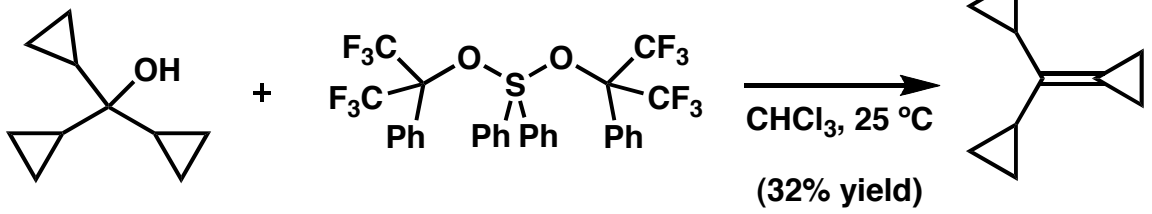




Atkins, G. M.; Burgess, E. M. *J. Am. Chem. Soc*. 1968, *90*, 4744-4745.
Burgess, E. M.; Penton, H. R.; Taylor, E. A. *J. Org. Chem*. 1973, *38*, 26-31.
Holton, R. A. et al. *J. Am. Chem. Soc*. 1994, *116*, 1599-1600.

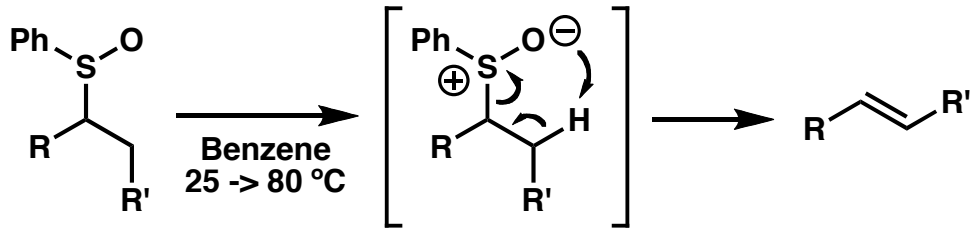
Elimination
Martin Dehydration

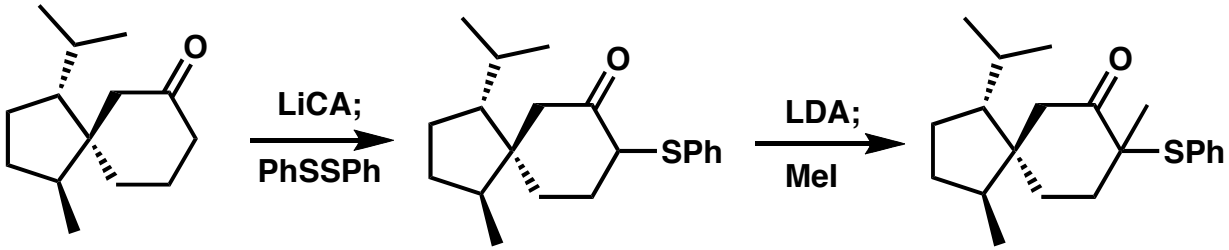


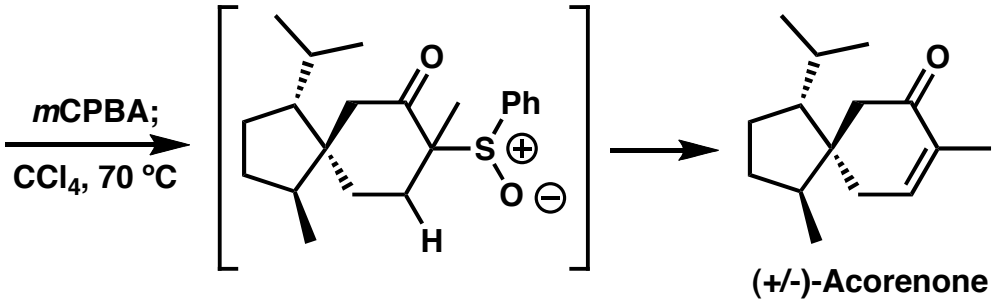


Martin, J. C. Arnhart, R. J. *J. Am. Chem. Soc.* 1971, *93*, 4327-4329.

Elimination
Sulfoxide



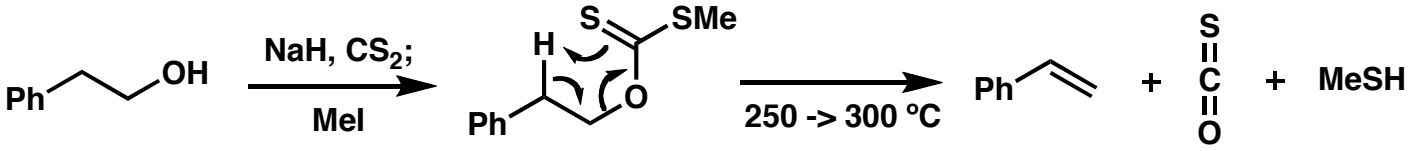


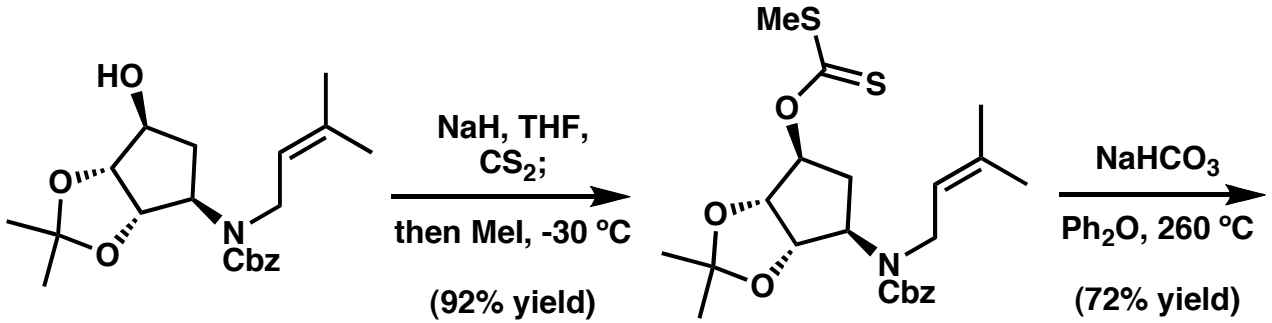


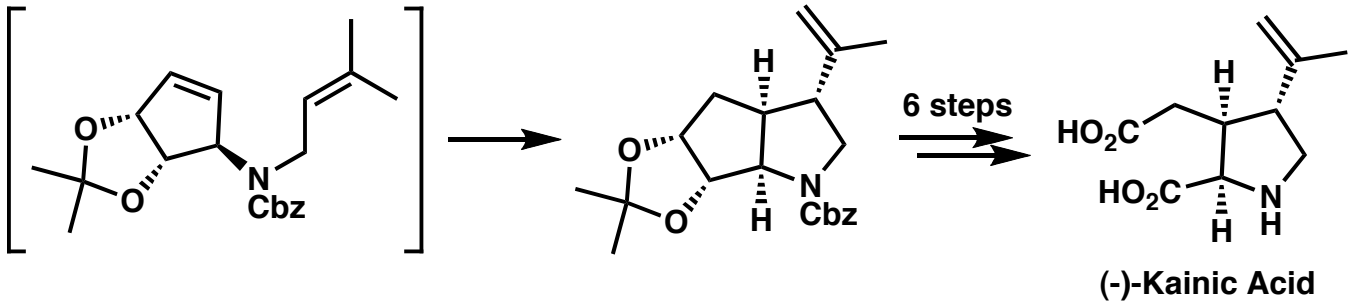
(+/-)-Acorenone

Trost, B. M. *Acc. Chem. Res*. 1978, *11*, 453-461.
Oppolzer, W.; Mahalanabis, K. K.; Bättig, K. *Helv. Chim. Acta*. 1977, *60*, 2388-2401.

Elimination
Xanthate Ester

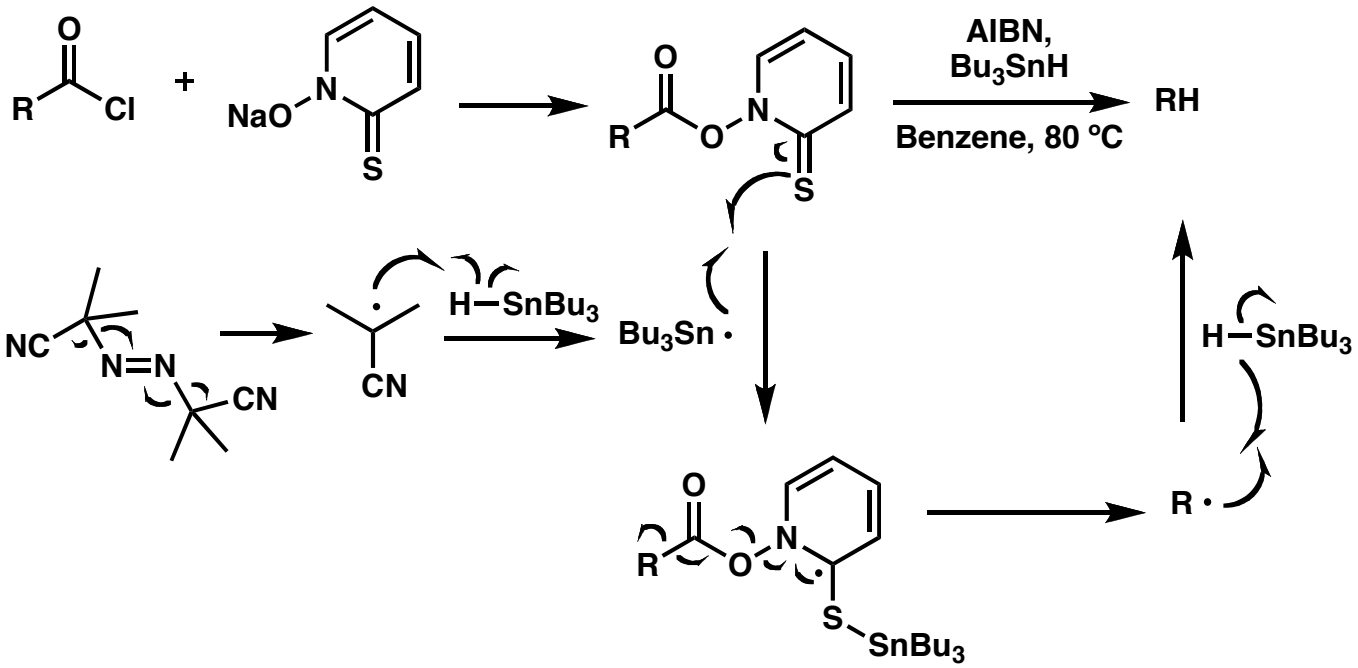


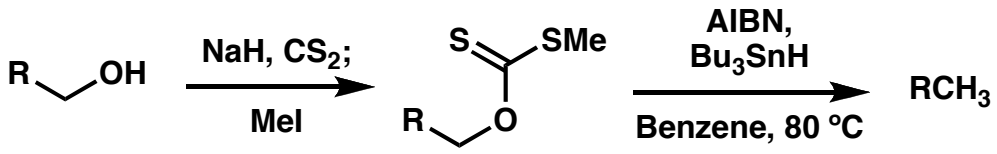




Chugaev, L. *Ber. Dtsch. Chem. Ges*. 1899, *32*, 3332.
Nakagawa, H.; Sugahara, T.; Ogasawara, K. *Org. Lett*. 2000, *2*, 3181-3183.

Radical Reaction
Barton

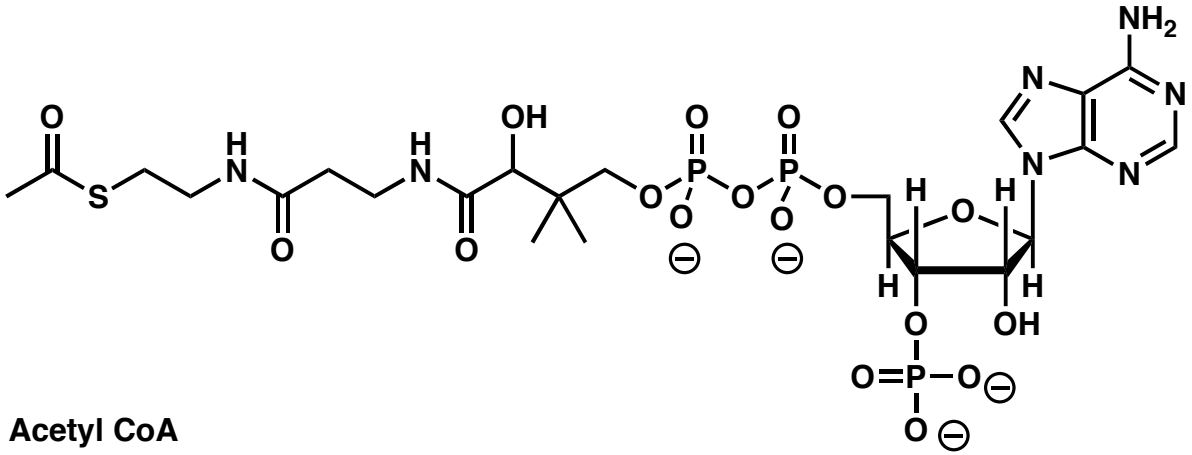




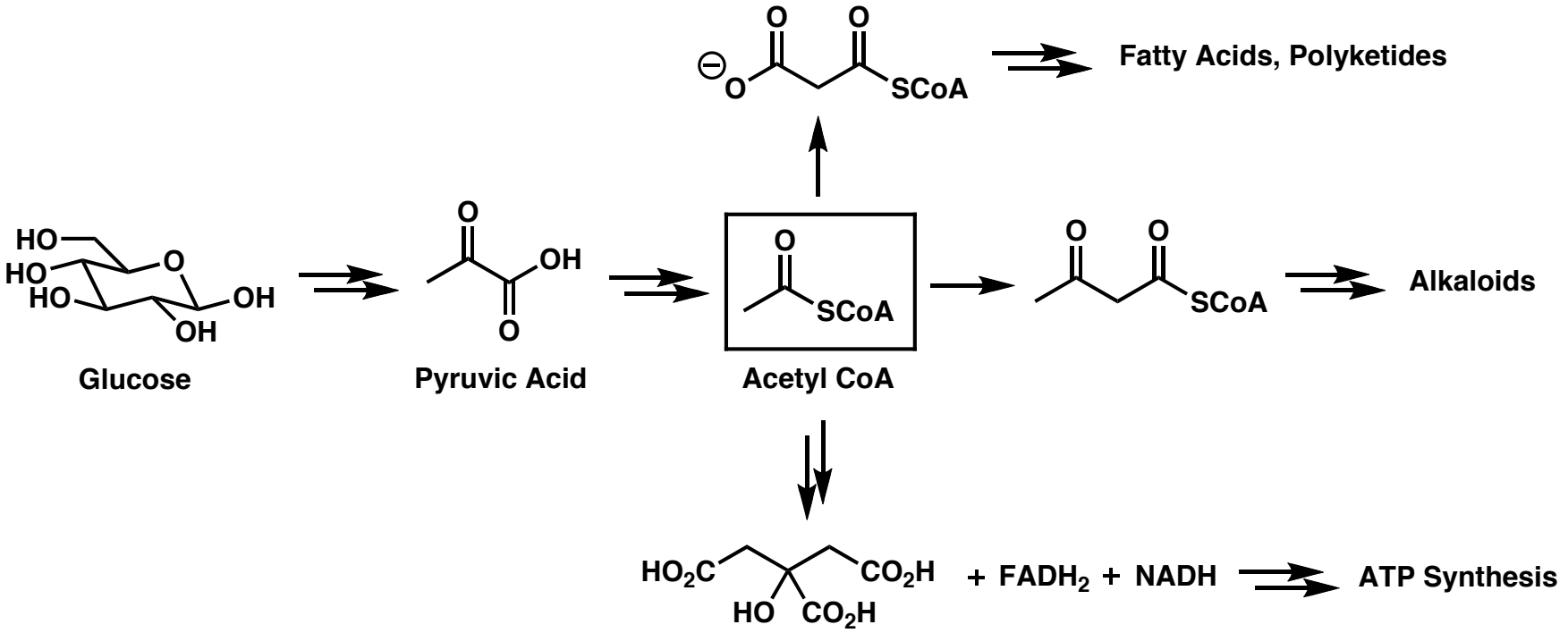
Barton, D. H. R.; Serebryakov, E. P. *Proc. Chem. Soc.* 1962, 309.
Barton, D. H. R.; McCombie, S. W. *J. Chem. Soc., Perkin Trans. 1* 1975, 1574-1585.

C-C Bond Formation

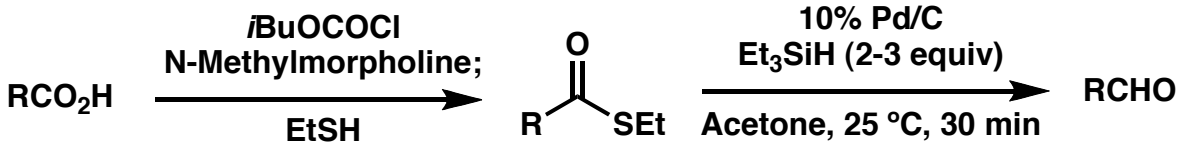
Nature - Thioester

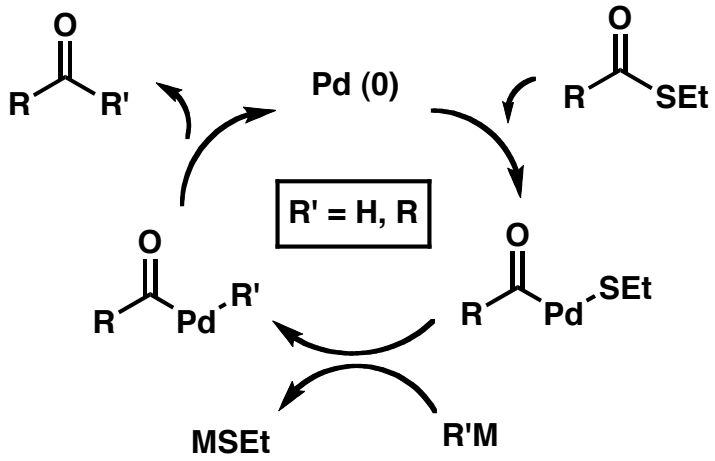


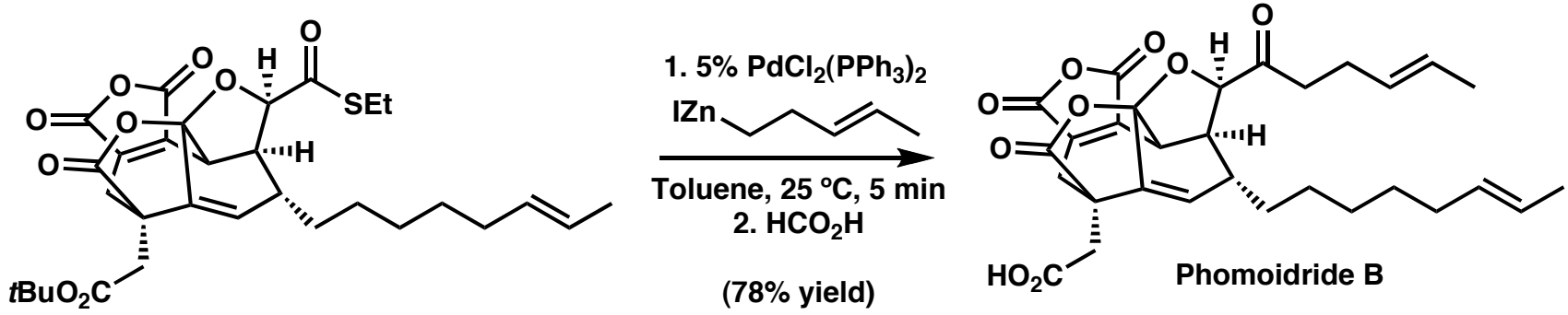
Acetyl CoA



C-C Bond Formation
Fukuyama - Thioester



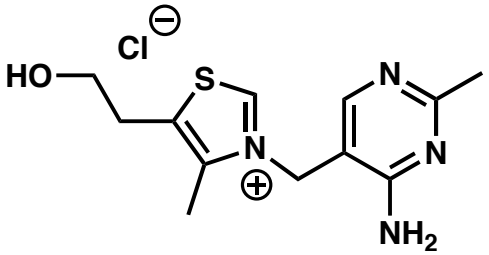




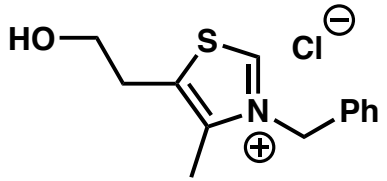
Fukuyama, T.; Lin, S-C.; Li, L. *J. Am. Chem. Soc*. 1990, *112*, 7050-7051.
Hayashi, Y.; Itoh, T.; Fukuyama, T. *Org. Lett*. 2003, *5*, 2235-2238.
Fukuyama, T.; Tokuyama, H. *Aldrich. Acta*. 2004. *37*, 87-96.

C-C Bond Formation

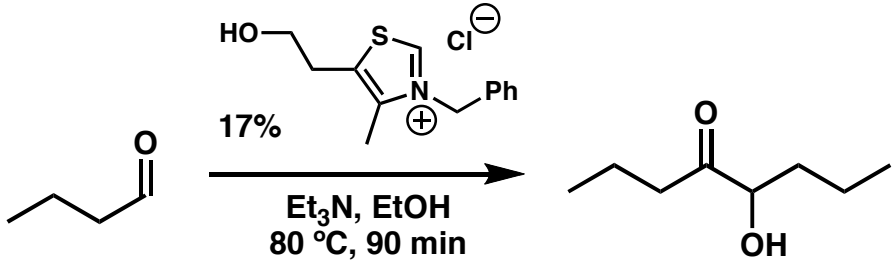
Acyl Anion Equivalent - Thiazolium Catalyst



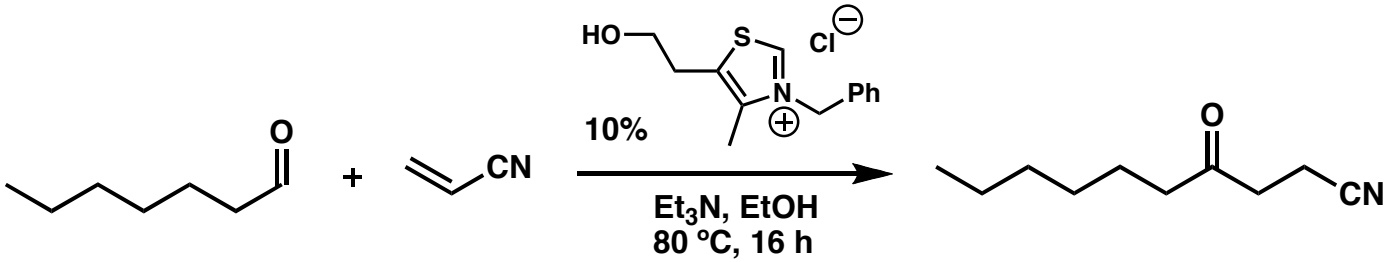
Thiamine
(Vitamin B_1)



1.30 $ / gram



(74% yield)

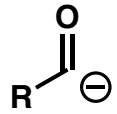


(75% yield)

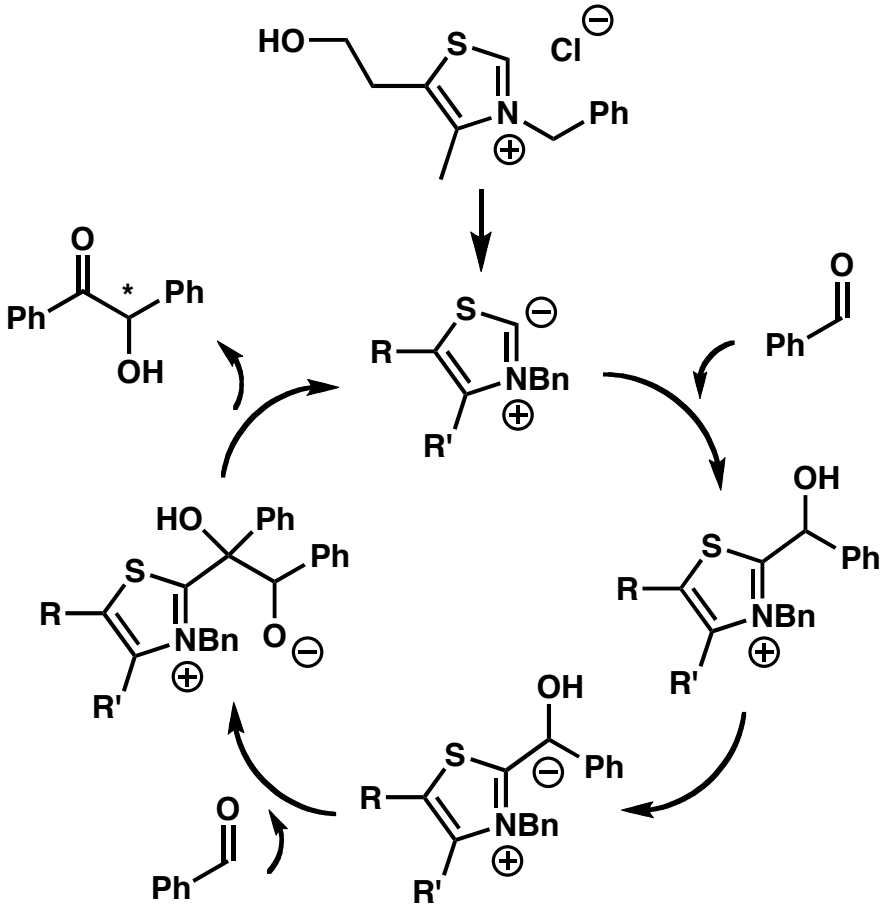
Stetter, H.; Kuhlmann, H. *Org. Syn.* 1984, *62*, 170.
Stetter, H.; Kuhlmann, H.; Haese, W. *Org. Syn.* 1987, *65*, 26.

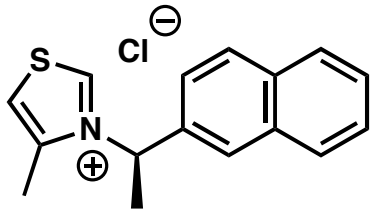
C-C Bond Formation

Acyl Anion Equivalent - Thiazolium Catalyst



Acyl Anion = synthon exhibiting reversal of normal carbonyl group reactivity



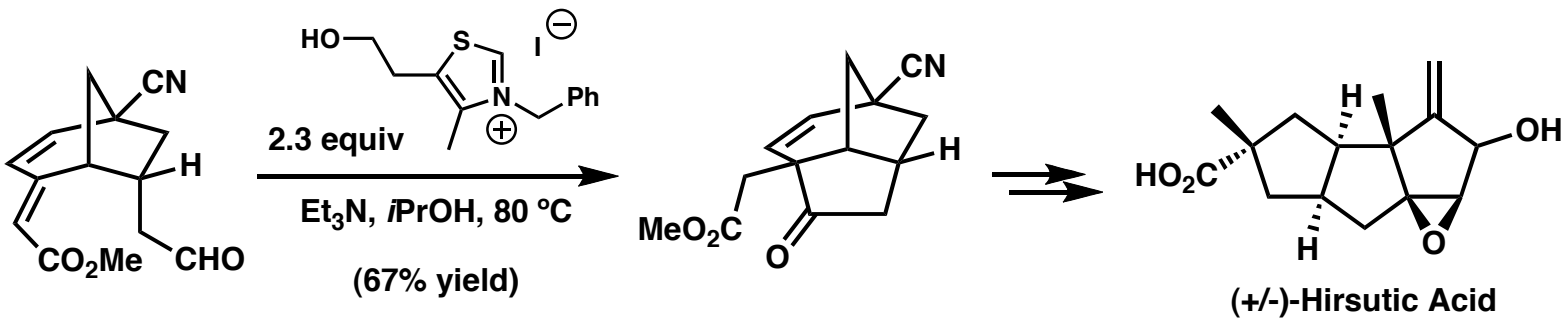


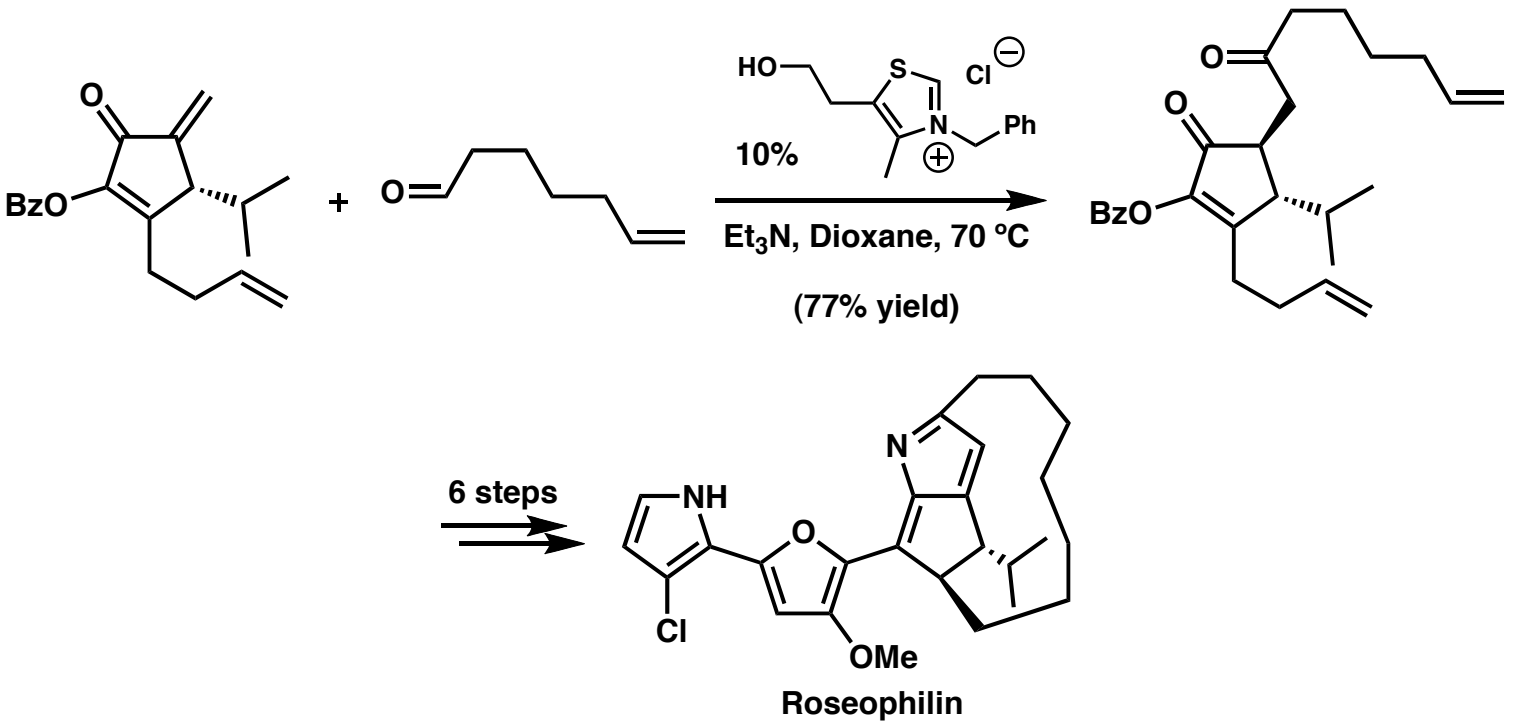
6% yield
52% ee

Breslow, R. *J. Am. Chem. Soc.* 1958, *80*, 3719-3726.
Sheehan, J.; Hunnemann, D. H. *J. Am. Chem. Soc.* 1966, *88*, 3666-3667.
Sheehan, J.; Hara, T. *J. Org. Chem.* 1974, *39*, 1196-1199.

C-C Bond Formation

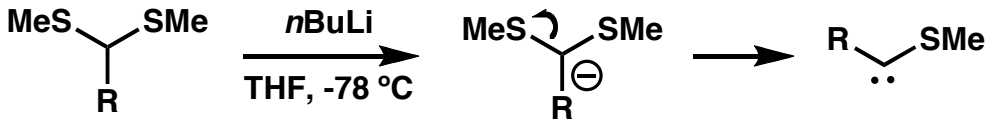
Acyl Anion Equivalent - Thiazolium Catalyst

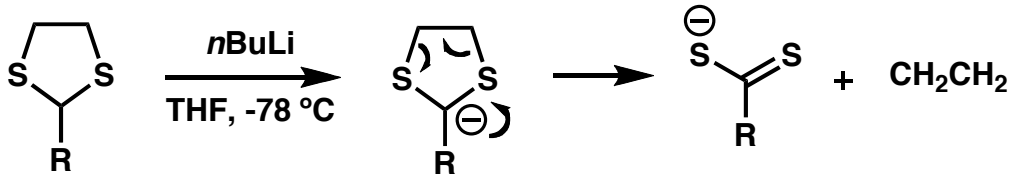


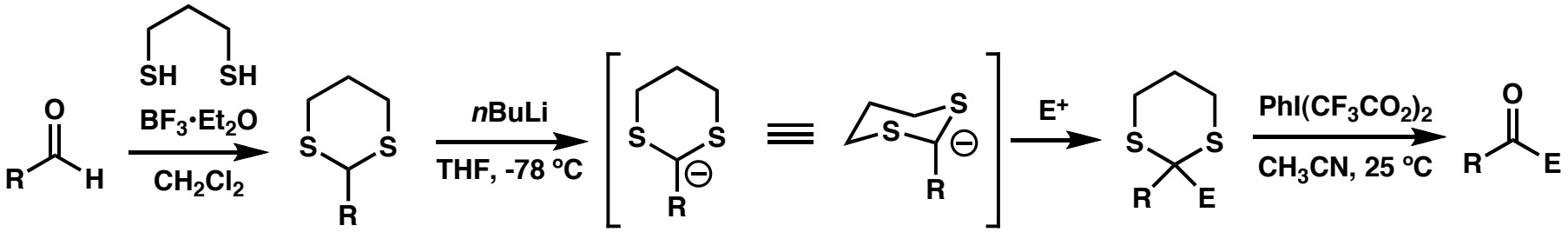


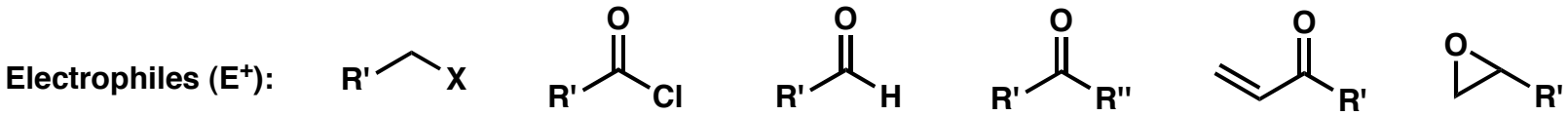
Trost, B. M.; Shuey, C. D.; DiNinno, F. *J. Am. Chem. Soc.* 1979, *101*, 1284-1285.
Harrington, P. E.; Tius, M. A. *J. Am. Chem. Soc.* 2001, *123*, 8509-8514.

C-C Bond Formation
Acyl Anion Equivalent - Dithiane



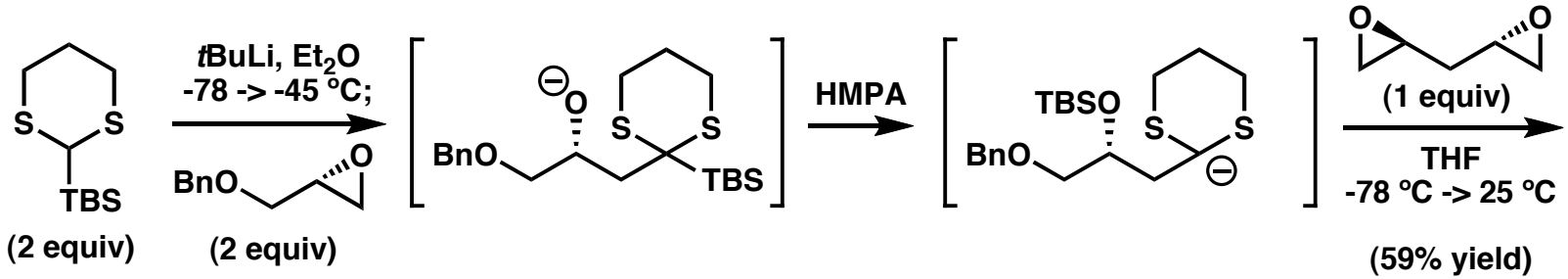


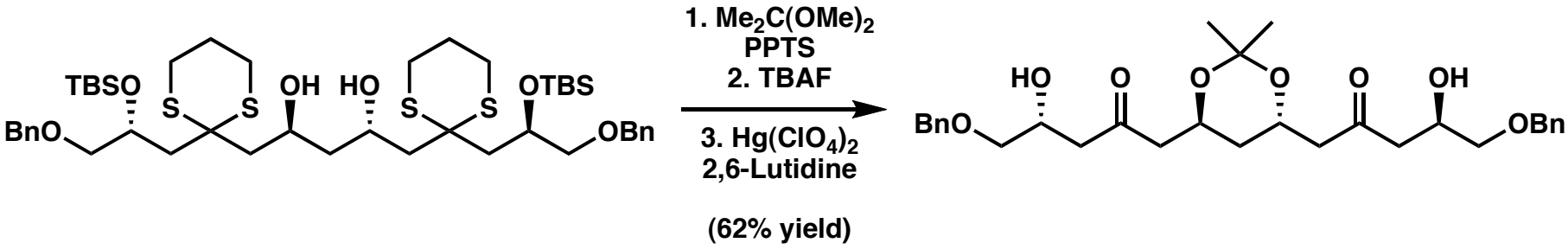


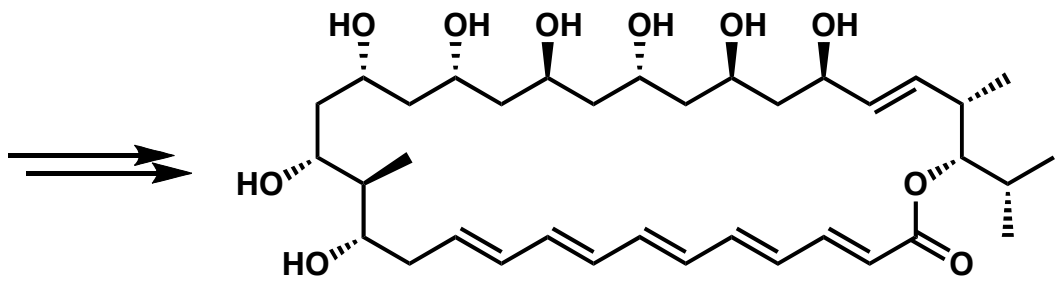


Corey, E. J.; Seebach, D. *Angew. Chem. Int. Ed. Engl.* 1965, *4*, 1075-1077.
Stork, G.; Zhao, K. A. *Tetrahedron Lett.* 1989, *30*, 287-290.

C-C Bond Formation
Acyl Anion Equivalent - Dithiane

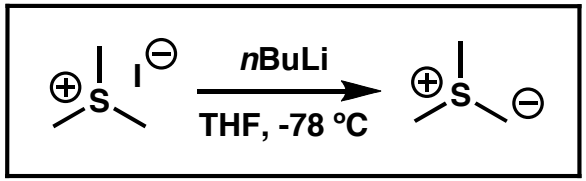






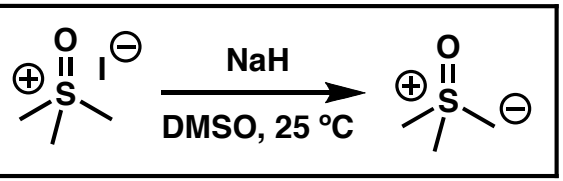
(+)-Mycoticin A

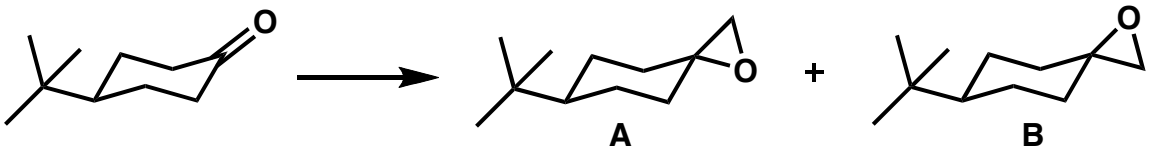
Smith, A. B. III; Pitram, S. M. *Org. Lett*. 1999, *1*, 2001-2004.
Smith, A. B. III, Adams, C. M. *Acc. Chem. Res*. 2004, *37*, 365-377.

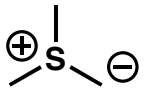
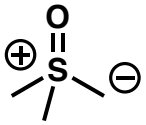


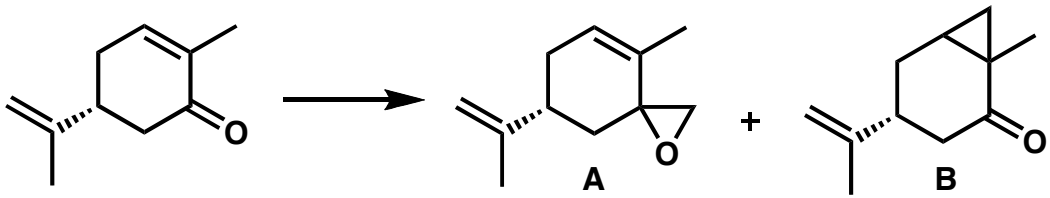
C-C Bond Formation

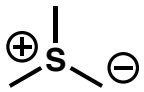
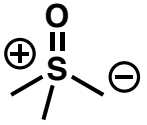
Sulfur Ylides - Epoxidation, Cyclopropanation





Reagent	Solvent	Temperature	Ratio (A/B)
	THF	0 °C -> 25 °C	87 : 13
	THF	65 °C	0 : 100

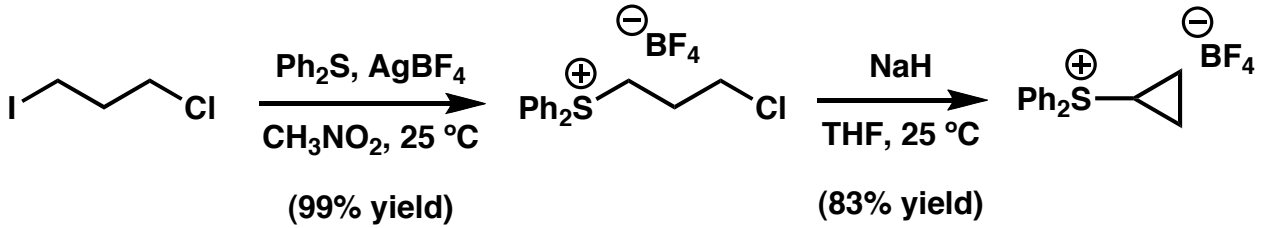


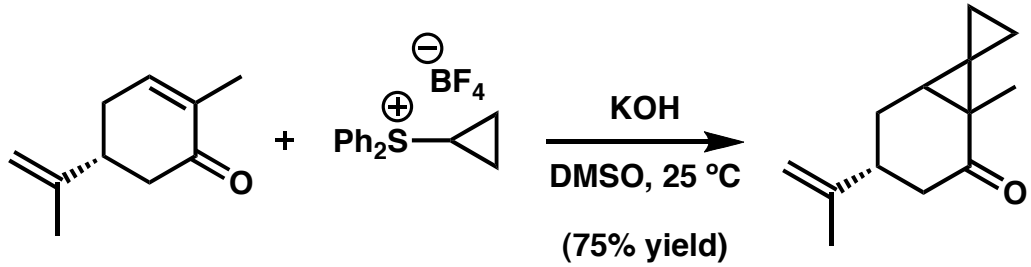
Reagent	Solvent	Temperature	Ratio (A/B)
	THF	0 °C -> 25 °C	100 : 0
	DMSO	25 °C -> 50 °C	0 : 100

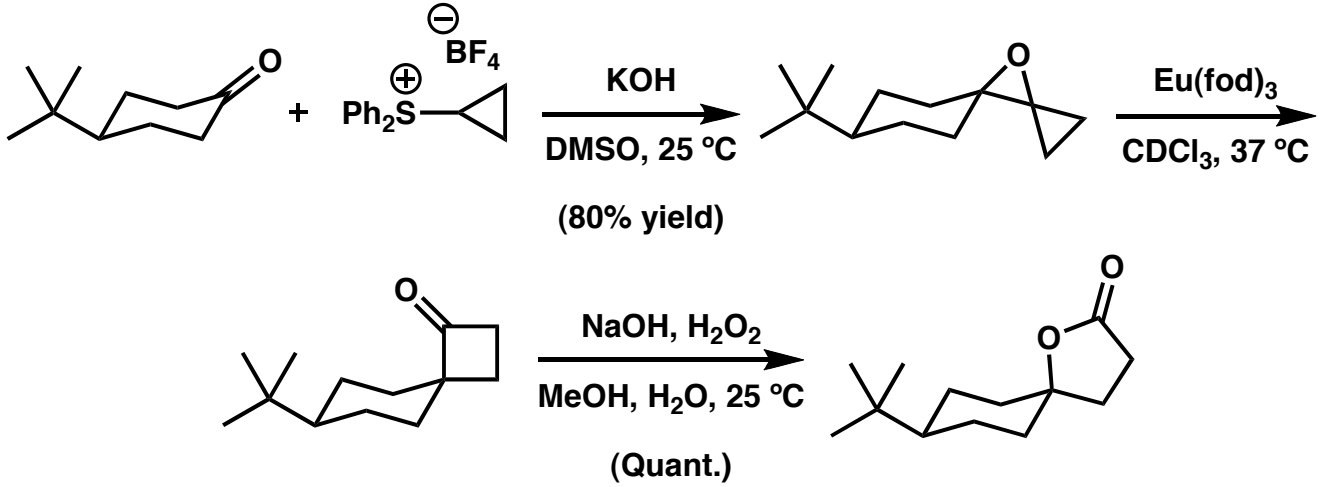
Corey, E. J.; Chaykovsky, M. *J. Am. Chem. Soc.* 1965, *87*, 1353-1364.

C-C Bond Formation

Sulfur Ylides - Epoxidation, Cyclopropanation

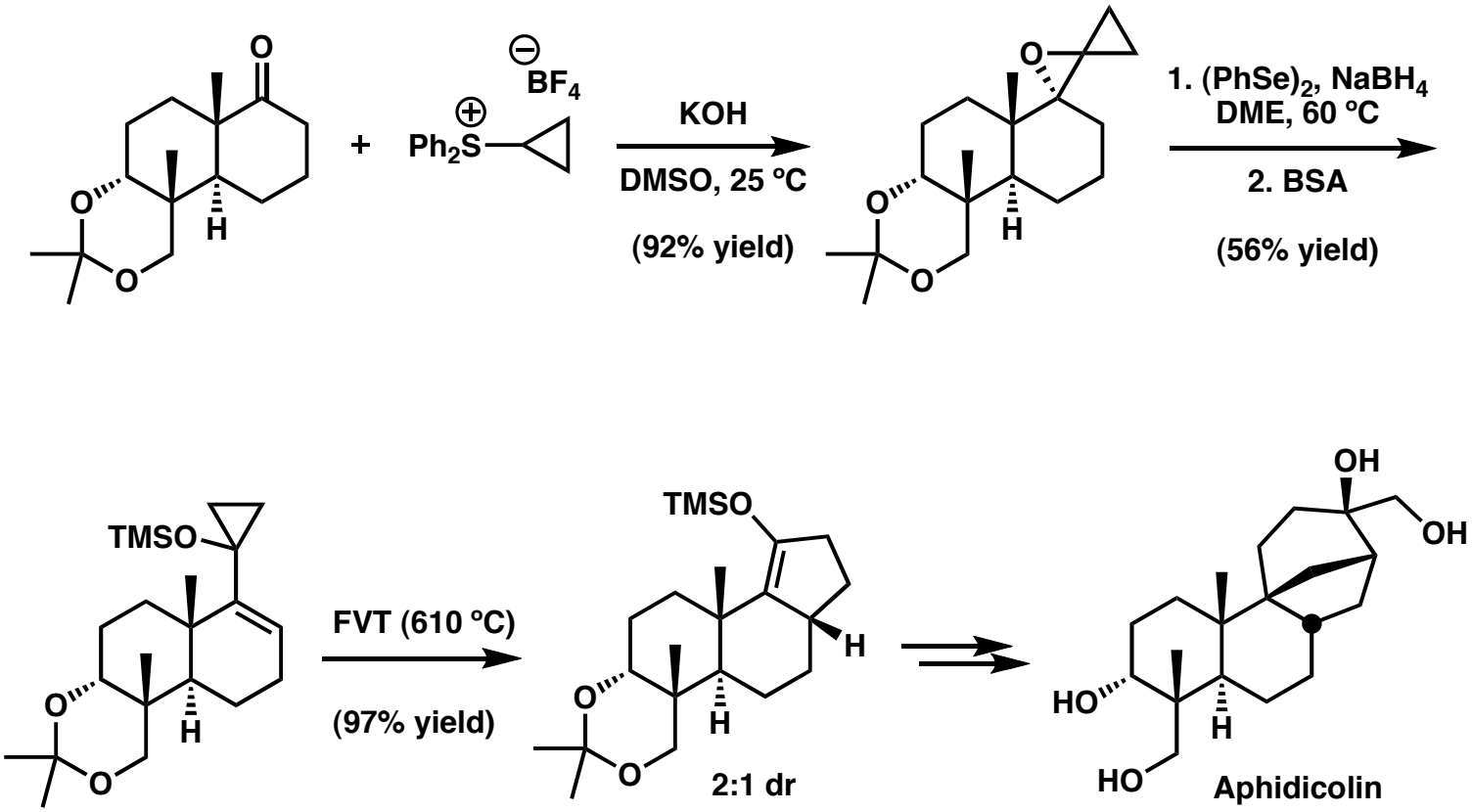






Bogdanowicz, M. J.; Trost, B. M. *Org. Syn.* 1974, *54*, 27.
Trost, B. M.; Bogdanowicz, M. J. *J. Am. Chem. Soc.* 1971, *93*, 3773-3774.
Trost, B. M.; Bogdanowicz, M. J. *J. Am. Chem. Soc.* 1973, *95*, 5311-5321.
Trost, B. M.; Bogdanowicz, M. J. *J. Am. Chem. Soc.* 1973, *95*, 5321-5334.

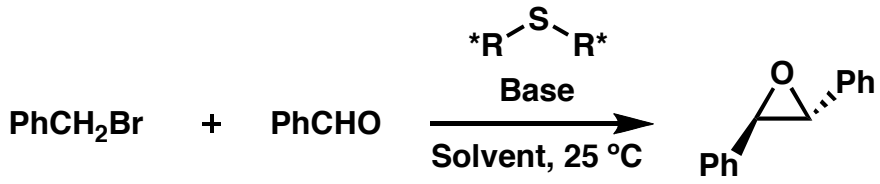
C-C Bond Formation
Sulfur Ylides - Epoxidation, Cylopropanation

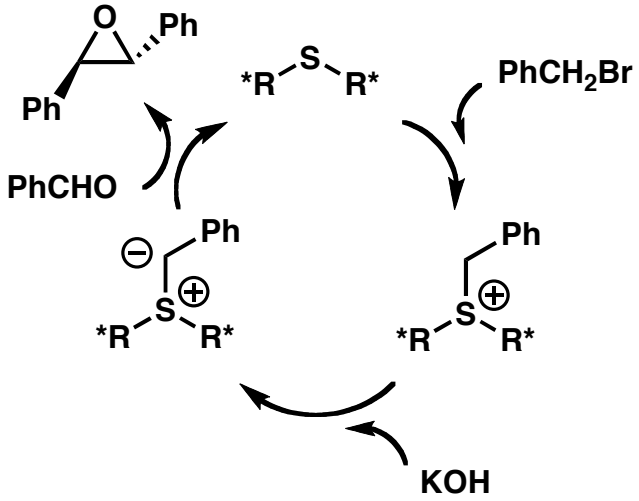


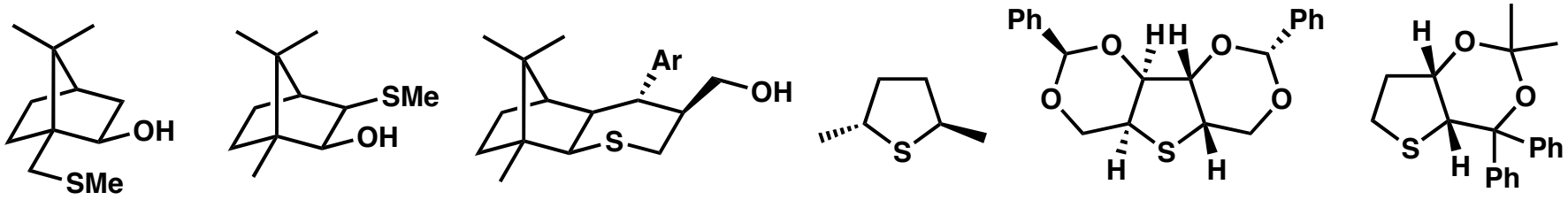
Trost, B. M.; Nishimura, Y.; Yamamoto, K.; McElvain, S. S. *J. Am. Chem. Soc*. 1979, *101*, 1328-1330.

C-C Bond Formation

Sulfur Ylides - Epoxidation, Cyclopropanation



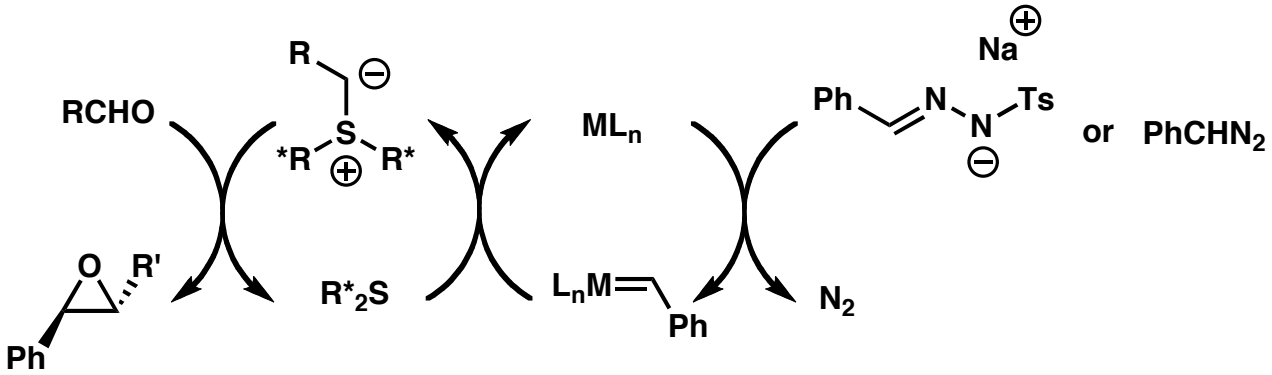


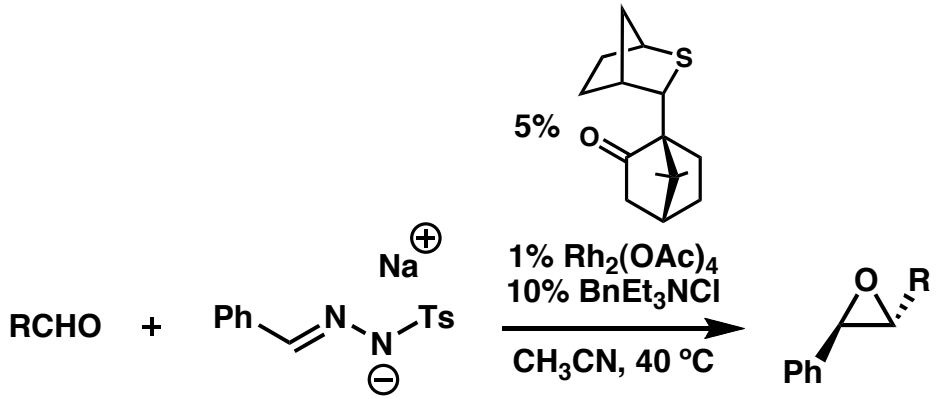


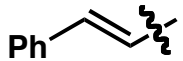
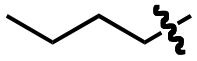
Furukawa	Dai	Saito	Metzner	Goodman	Shimizu
1989	1996	2001	1999	2002	1999
43% ee	42% ee	56% ee	85% ee	97% ee	48% ee

C-C Bond Formation

Sulfur Ylides - Epoxidation, Cyclopropanation

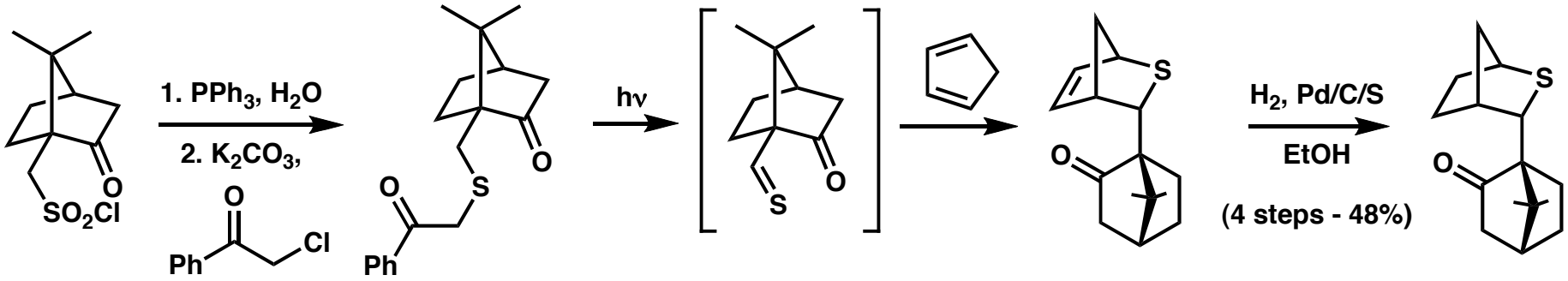


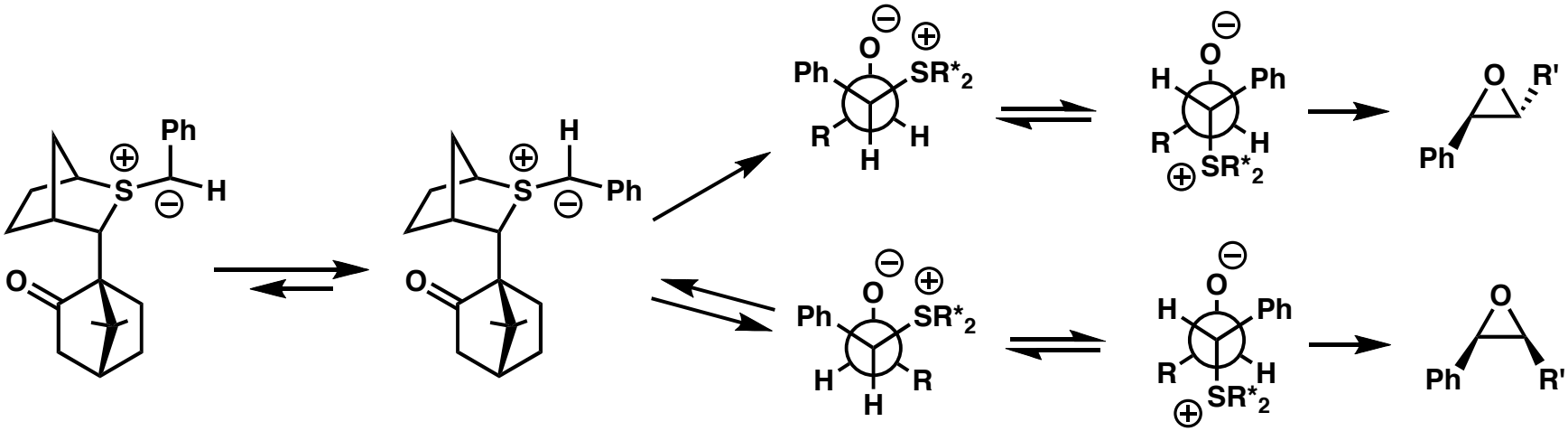


R	Yield (%)	Trans/Cis	ee (%)
	82	98:2	94
	70	98:2	87
	46	75:25	89

Aggarwal, V. K. et al. *J. Am. Chem. Soc.* 2003, *125*, 10926-10940.

C-C Bond Formation
Sulfur Ylides - Epoxidation, Cyclopropanation

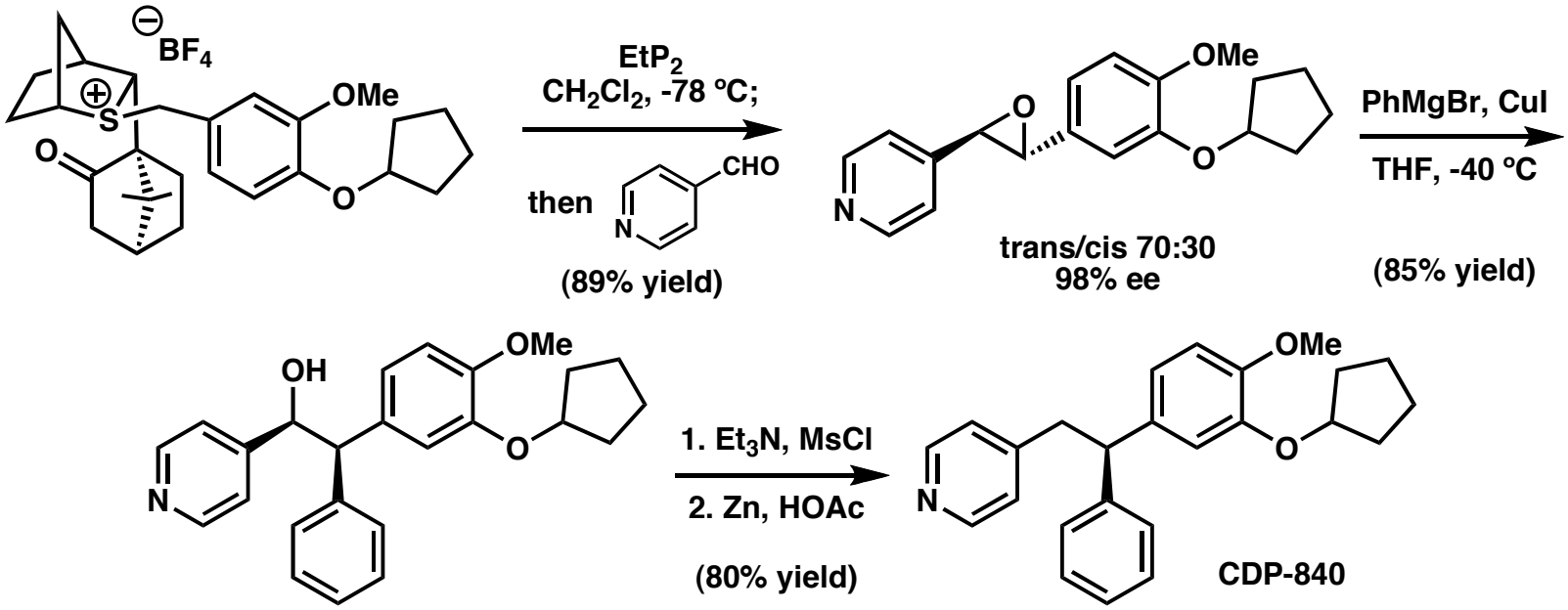


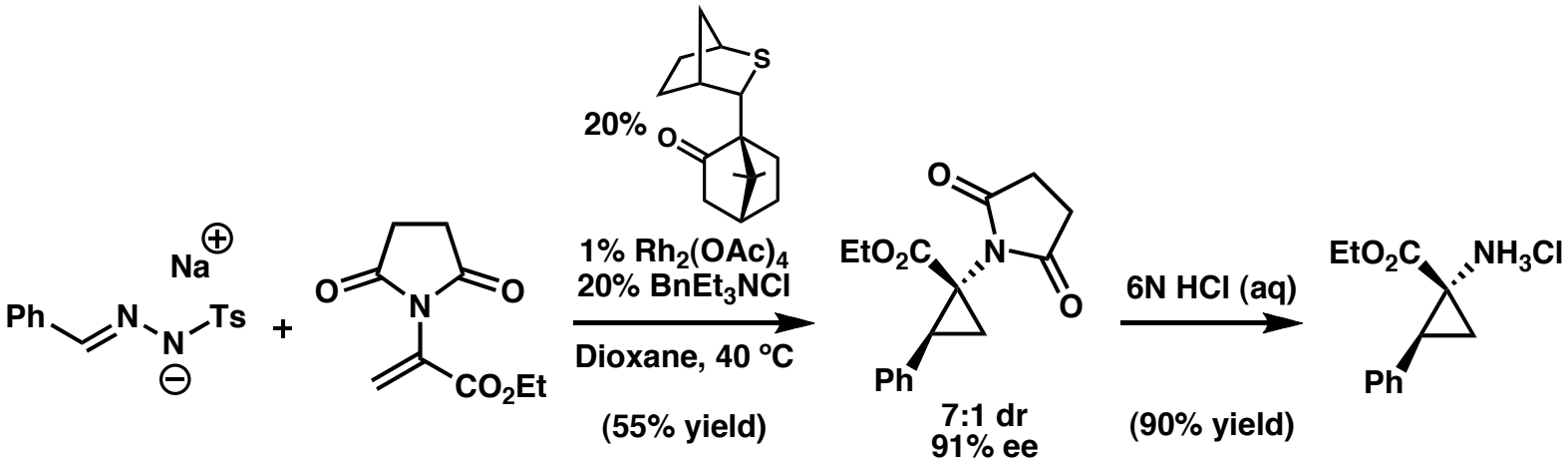


Aggarwal, V. K.; Harvey, J. N.; Richardson, J. *J. Am. Chem. Soc.* 2002, *124*, 5747-5756.

C-C Bond Formation

Sulfur Ylides - Epoxidation, Cyclopropanation





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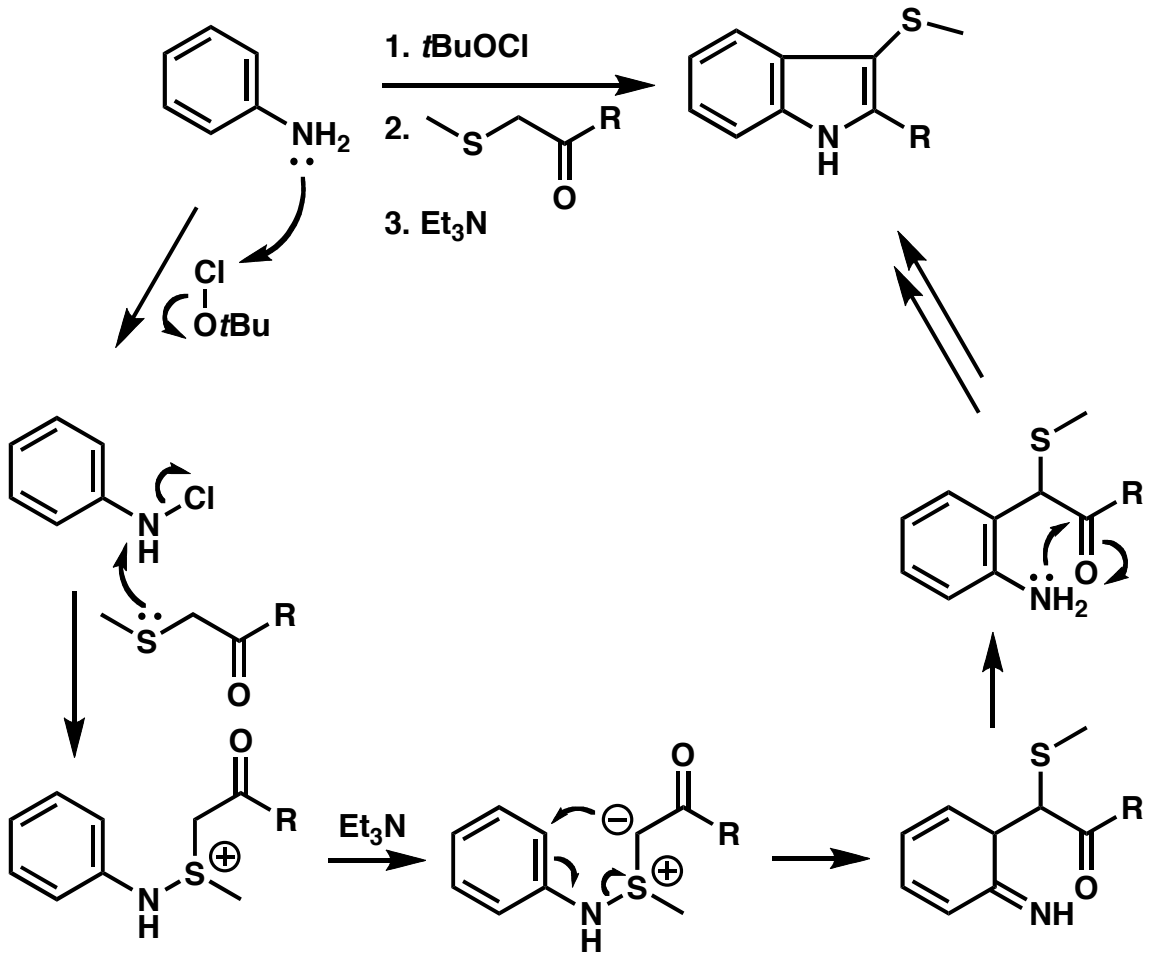
Aggarwal, V. K. et al. *Angew. Chem. Int. Ed.* 2001, *40*, 1433-1436.

C-C Bond Formation

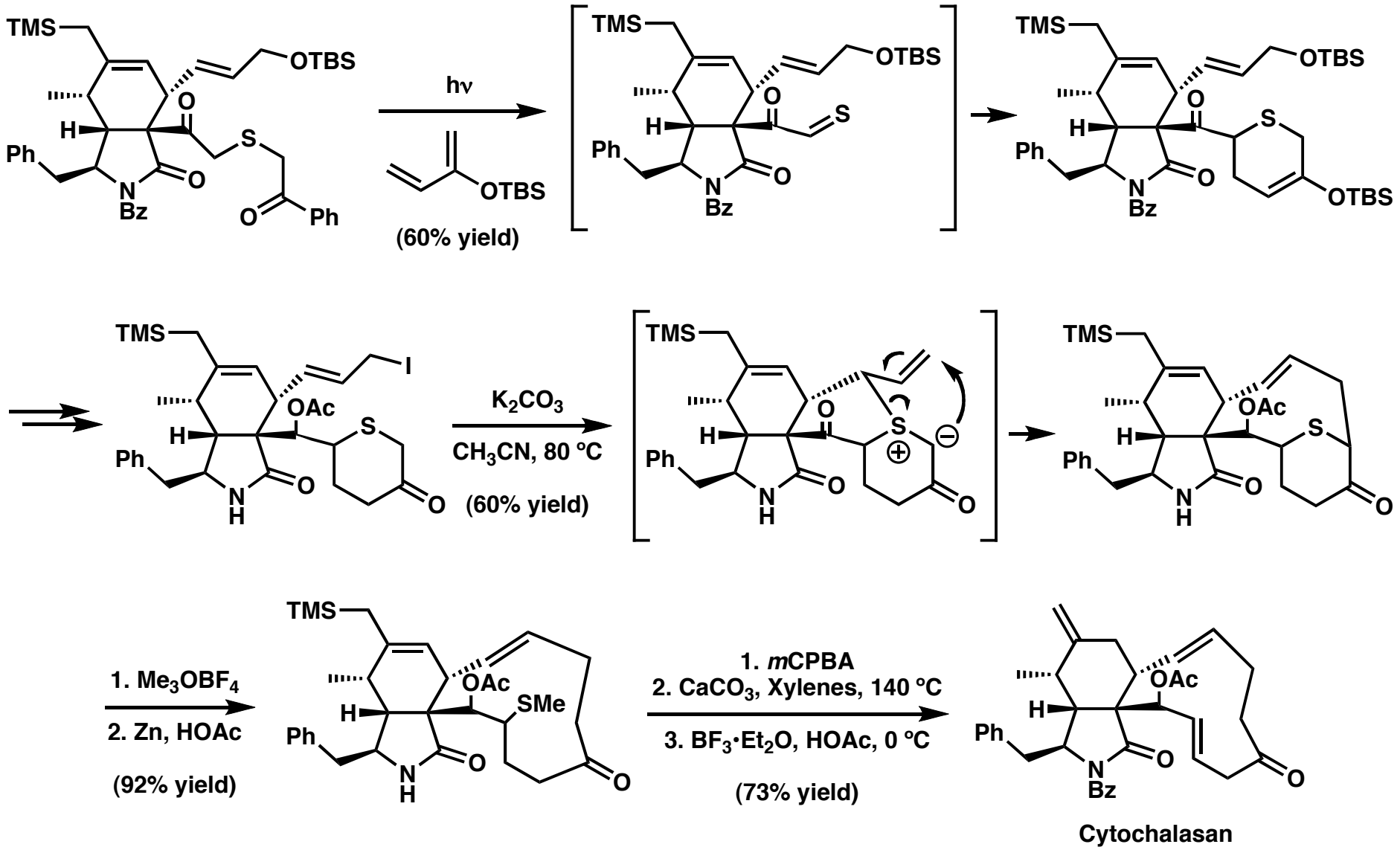
Sulfur Ylides - [2,3]-Sigmatropic Rearrangement



Gassman, P. G.; van Bergen, T. J.; Gilbert, D. P.; Cue, B. W. *J. Am. Chem. Soc.* 1974, *96*, 5495-5508.

C-C Bond Formation

Sulfur Ylides - [2,3]-Sigmatropic Rearrangement



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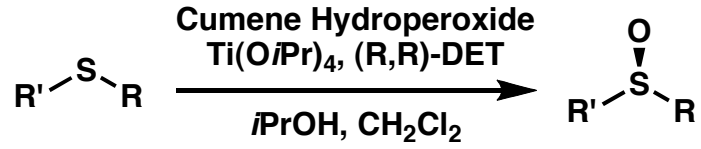
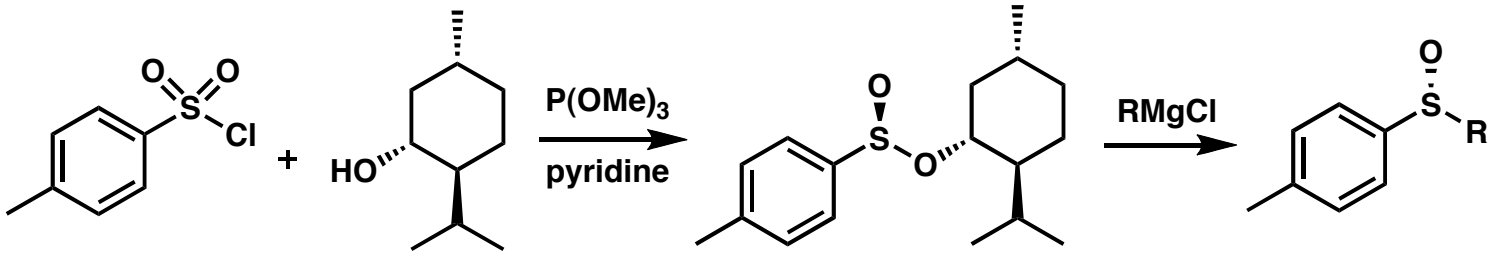
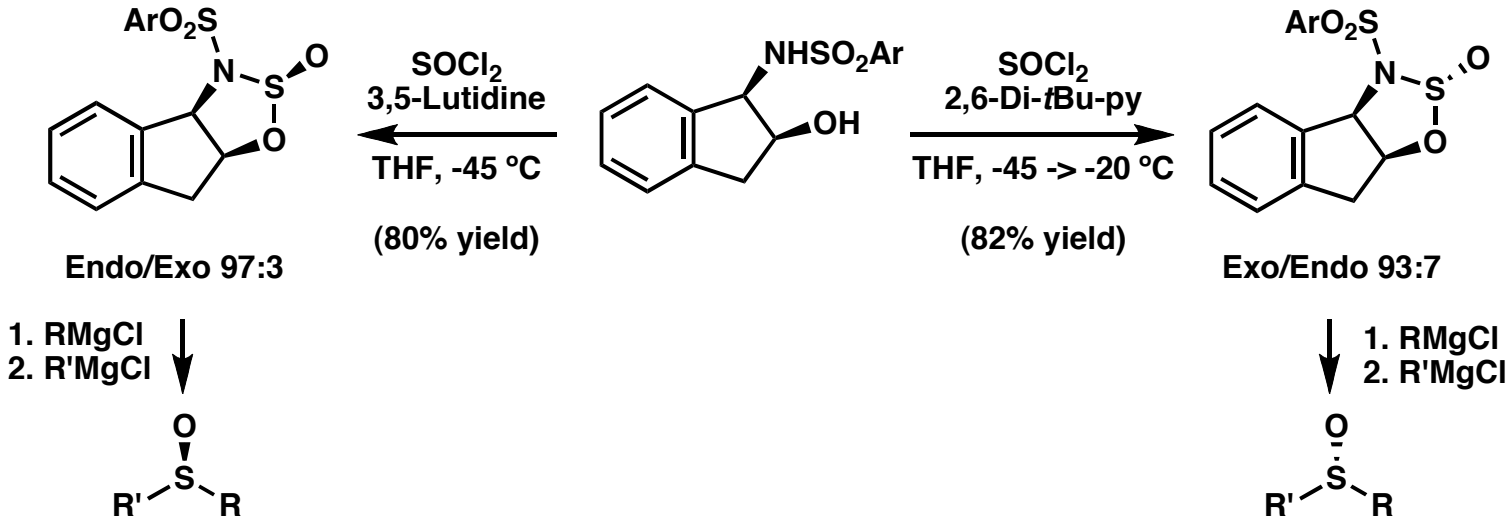
C-C Bond Formation
Chiral Sulfoxides







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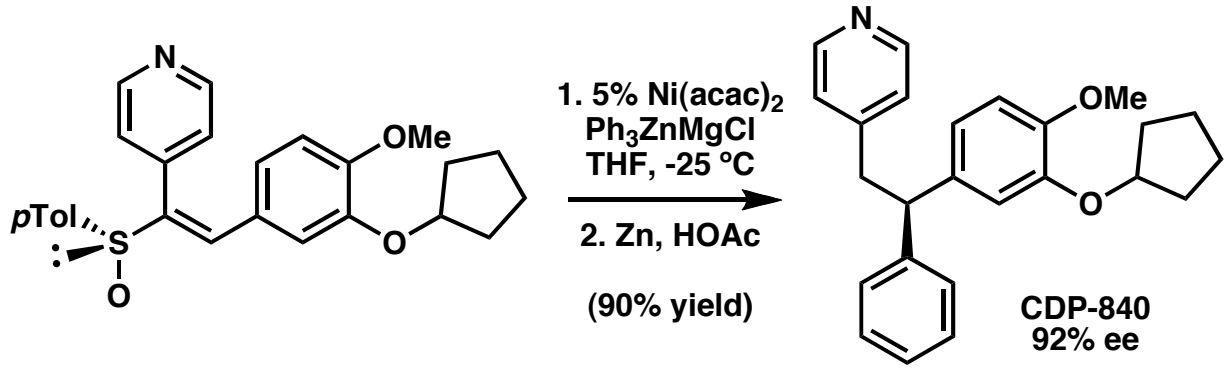
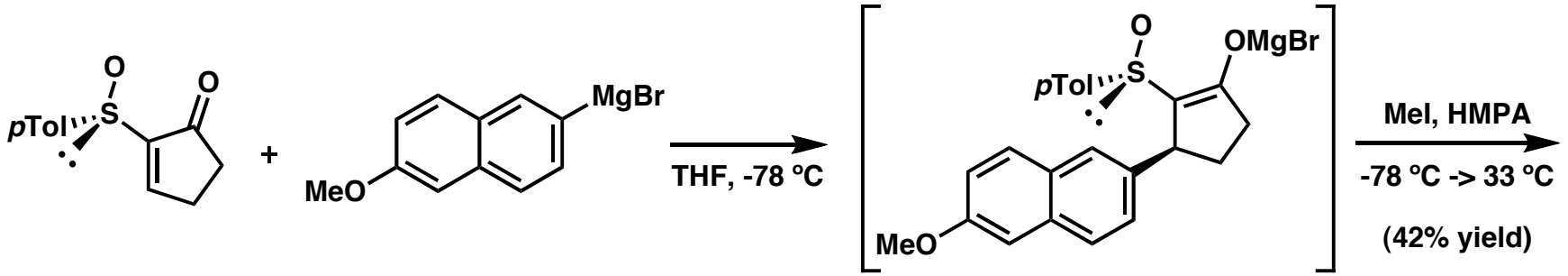
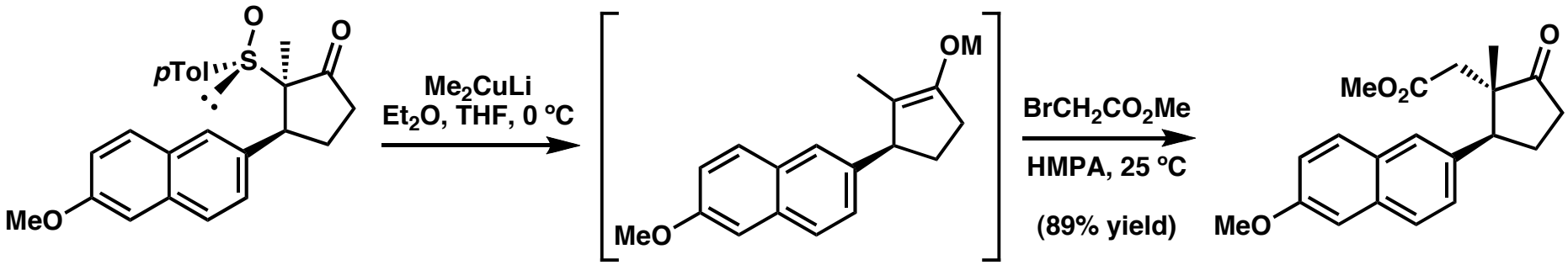
C-C Bond Formation
Chiral Sulfoxides







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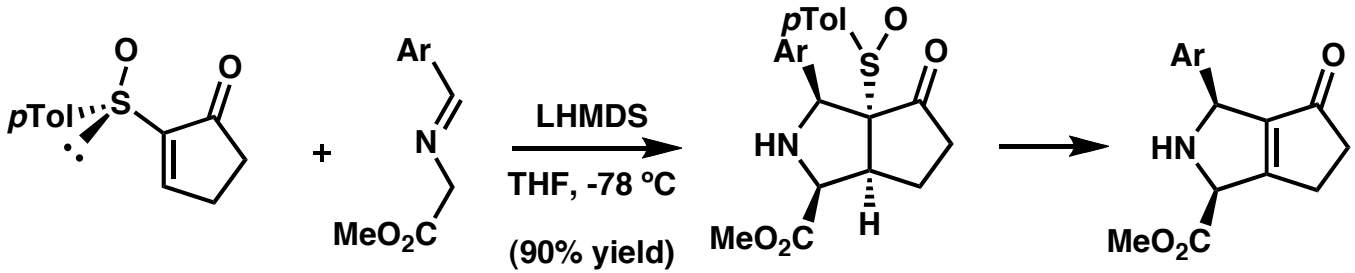
C-C Bond Formation
Chiral Sulfoxides



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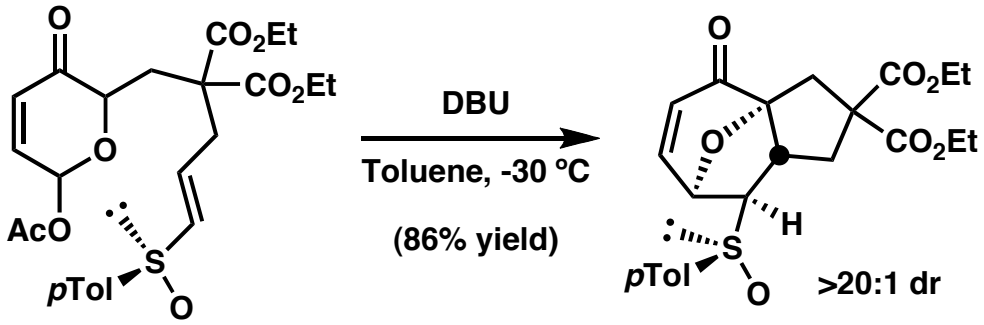

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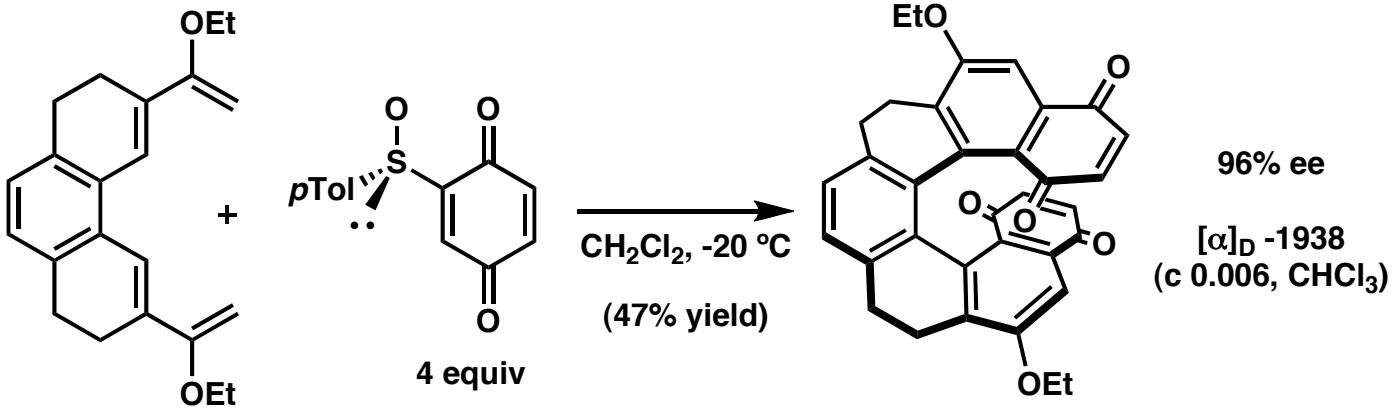

96% ee

$[\alpha]_D$ -1938
(c 0.006, $CHCl_3$)

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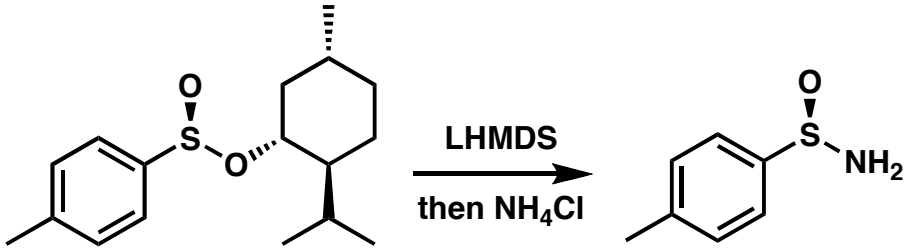
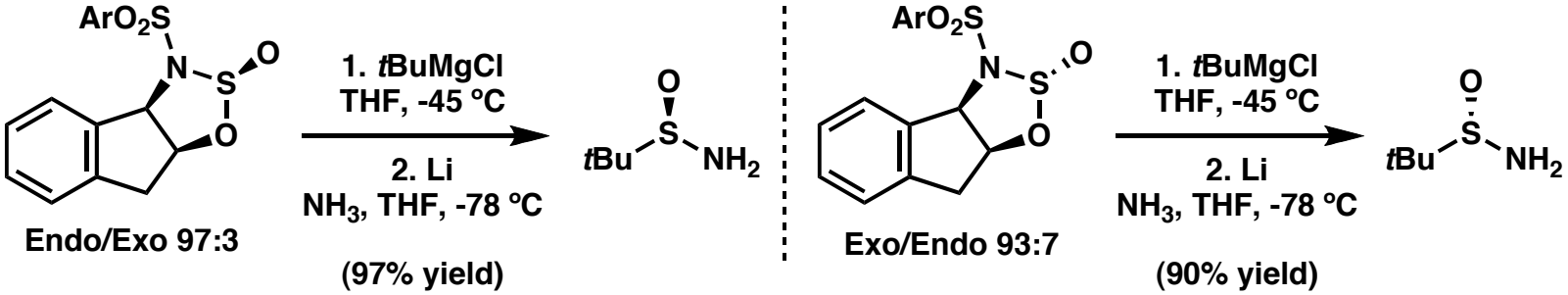
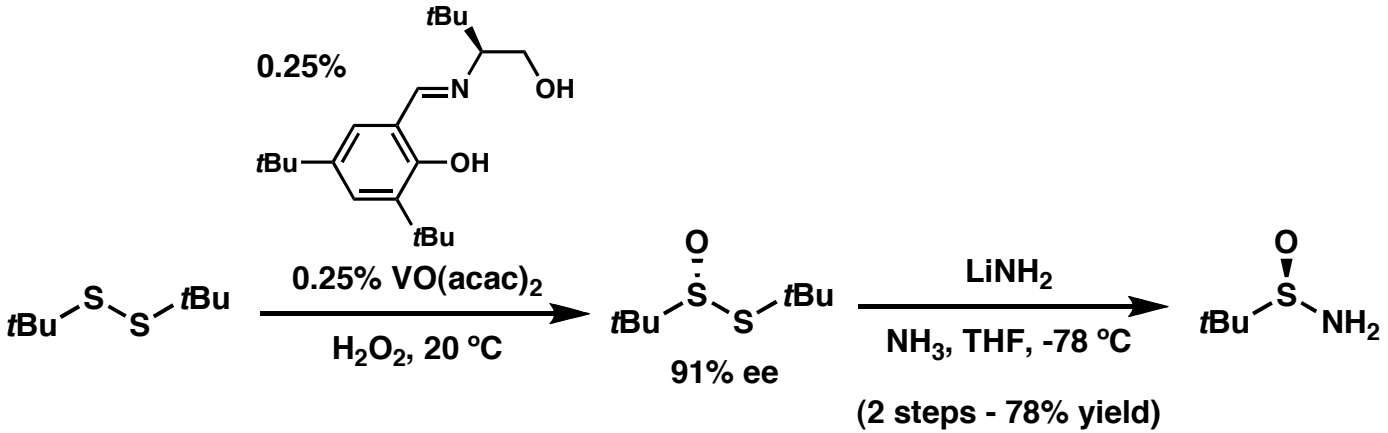
C-C Bond Formation
Chiral Sulfinamides







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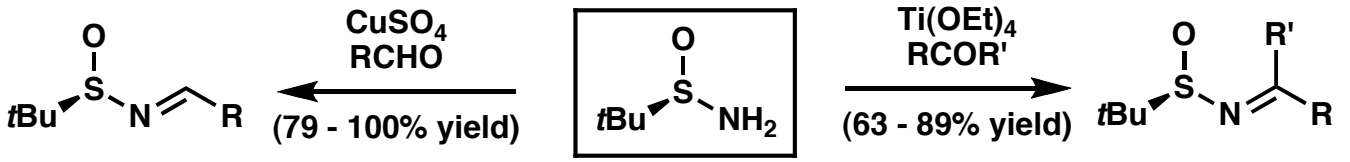
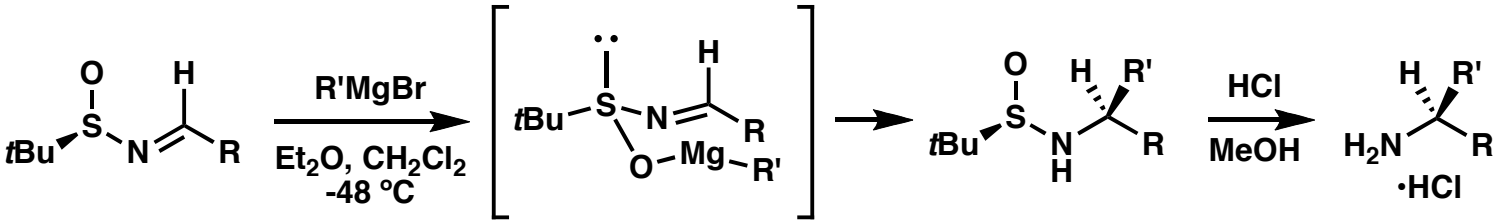
Senanayake, C. H. et al. *Aldrich. Acta*. 2005, *38*, 93-104.
Ellman, J. A.; Owens, T. D.; Tang, T. P. *Acc. Chem. Res*. 2002, *35*, 984-995.

C-C Bond Formation
Chiral Sulfinamides

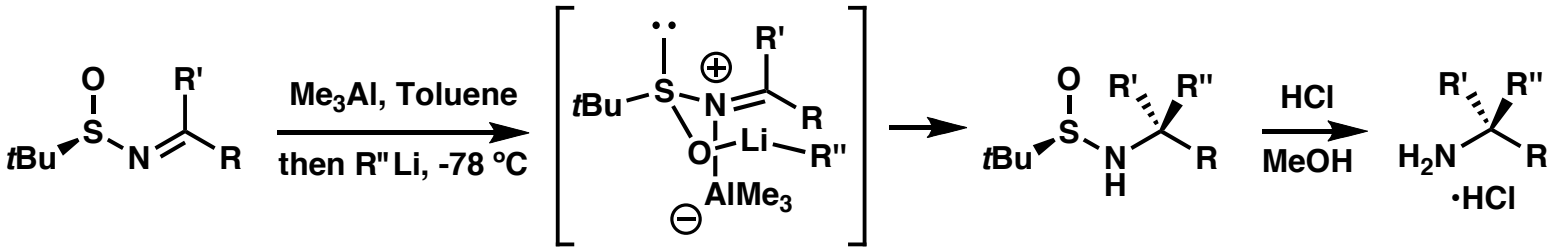




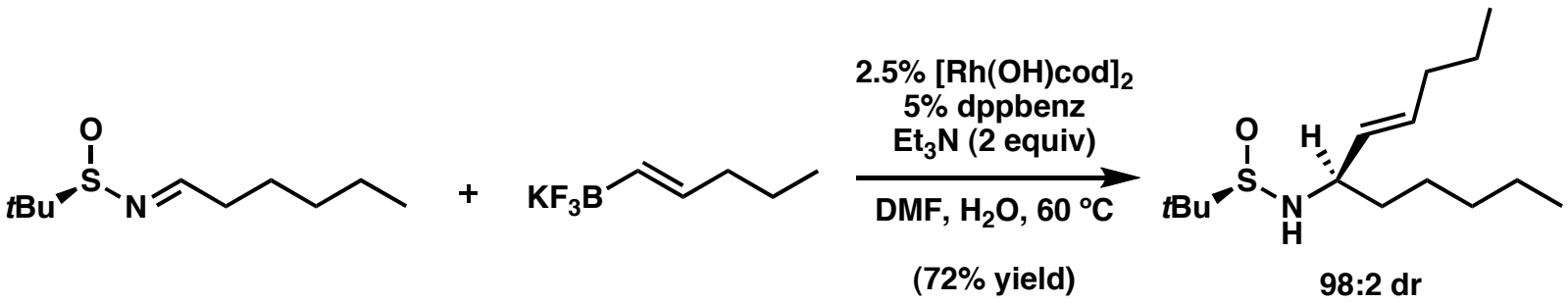
R	R'	dr	Addition Yield (%)	Cleavage Yield (%)
Et	Me	93:7	96	97
Et	iPr	98:2	97	92
Et	Ph	96:4	Quant.	90
Ph	Me	97:3	96	88

Ellman, J. A.; Owens, T. D.; Tang, T. P. *Acc. Chem. Res.* 2002, *35*, 984-995.

C-C Bond Formation
Chiral Sulfinamides



R	R'	R''	dr	Addition Yield (%)
*i*Pr	Me	*n*Bu	99:1	61
*i*Pr	Me	Ph	97:3	93
*i*Pr	*n*Bu	Me	91:9	82
CH_2OTBS	Me	*n*Bu	95:5	89



Ellman, J. A.; Owens, T. D.; Tang, T. P. *Acc. Chem. Res.* 2002, *35*, 984-995.
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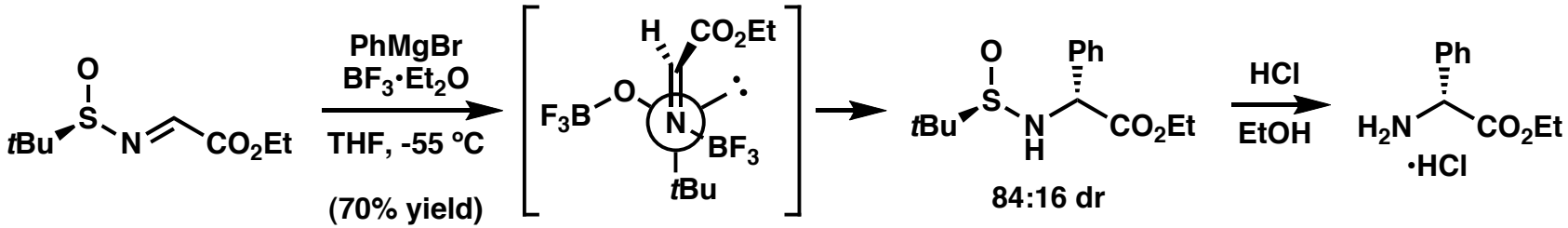
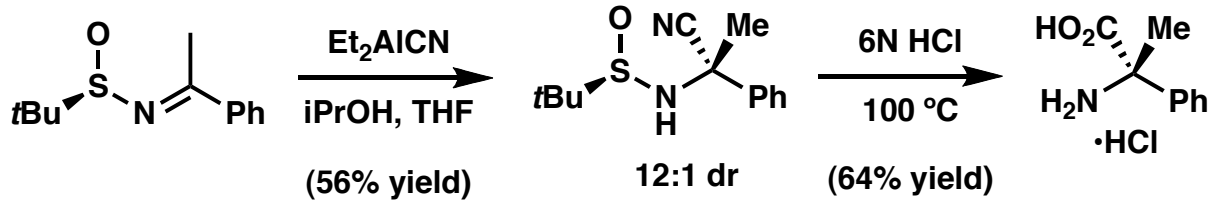
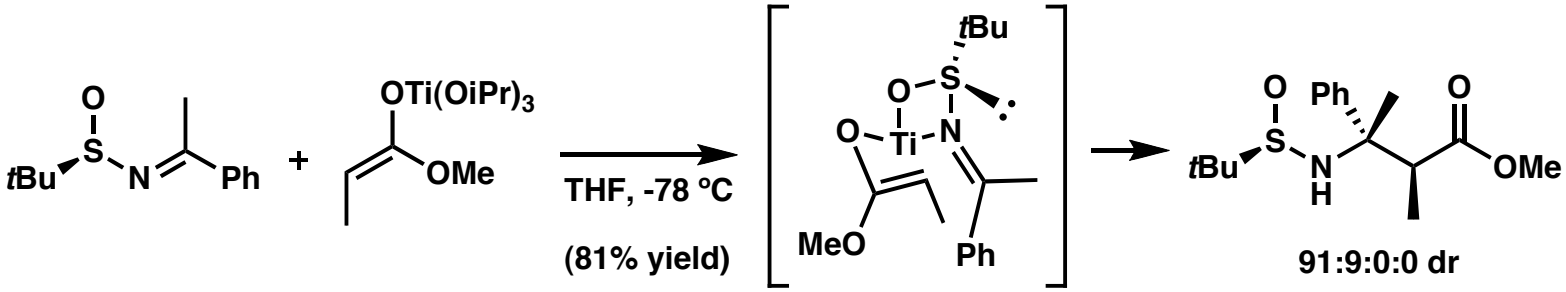
C-C Bond Formation
Chiral Sulfinamides







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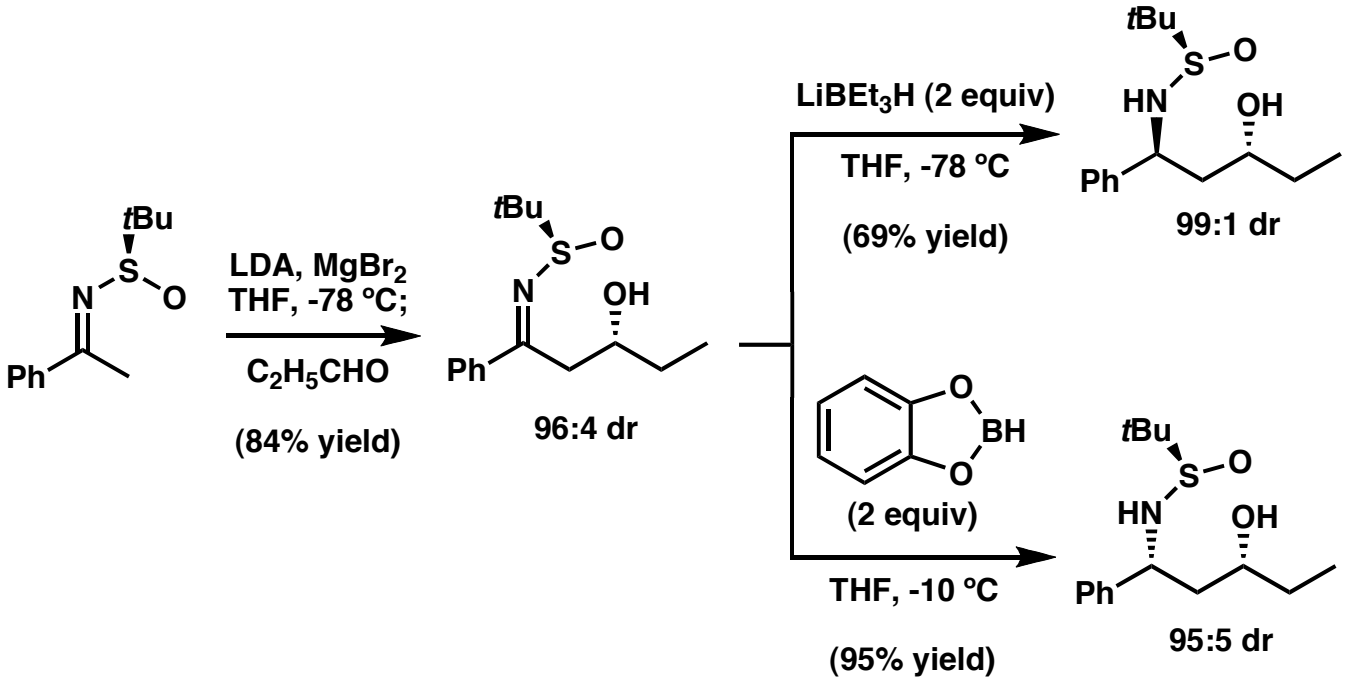
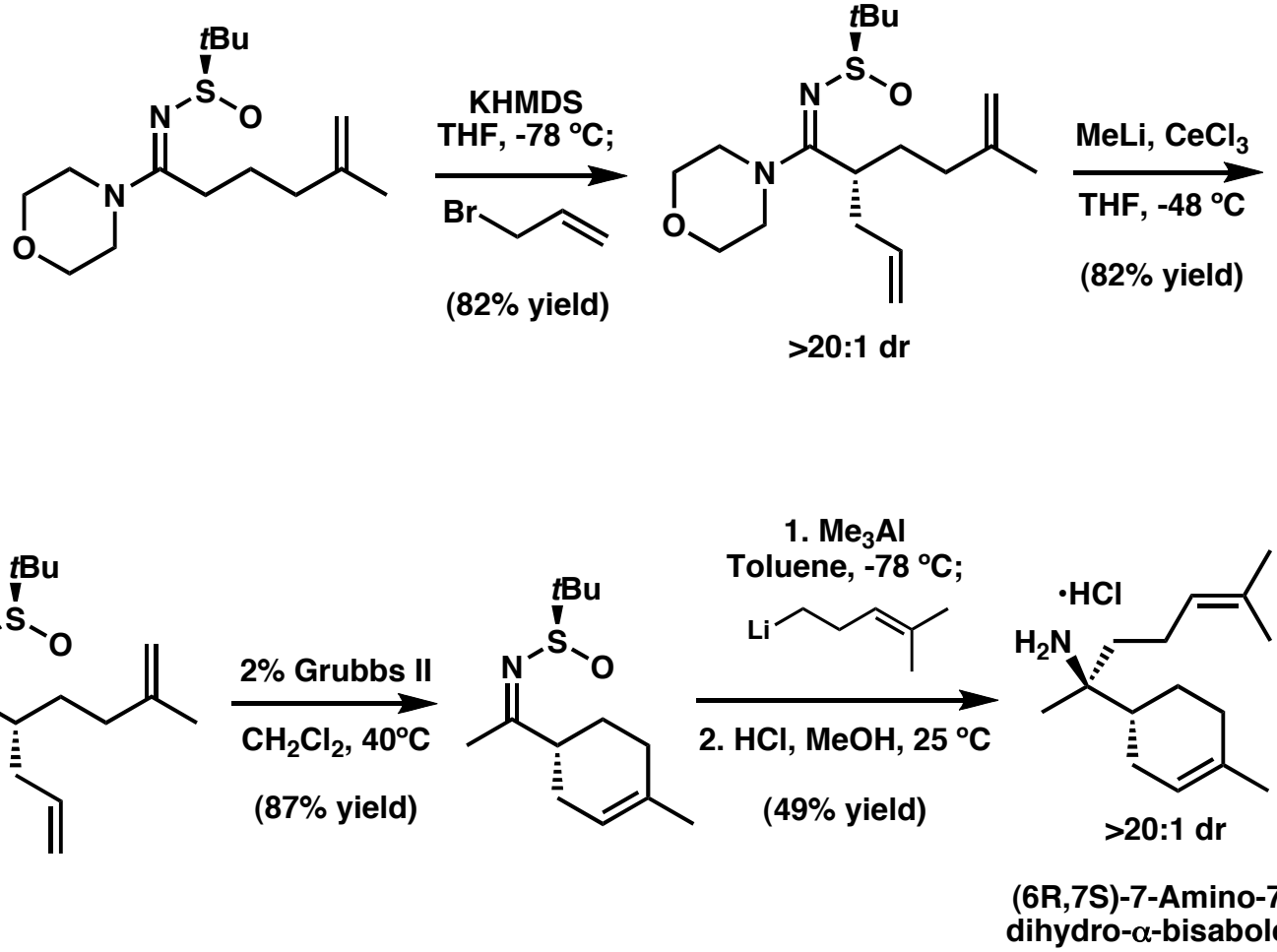
Ellman, J. A.; Owens, T. D.; Tang, T. P. *Acc. Chem. Res*. 2002, *35*, 984-995.

C-C Bond Formation
Chiral Sulfinamides



Kochi, T.; Tang, T. P.; Ellman, J. A. *J. Am. Chem. Soc*. 2002, *124*, 6518-6519.

C-C Bond Formation
Chiral Sulfinamides



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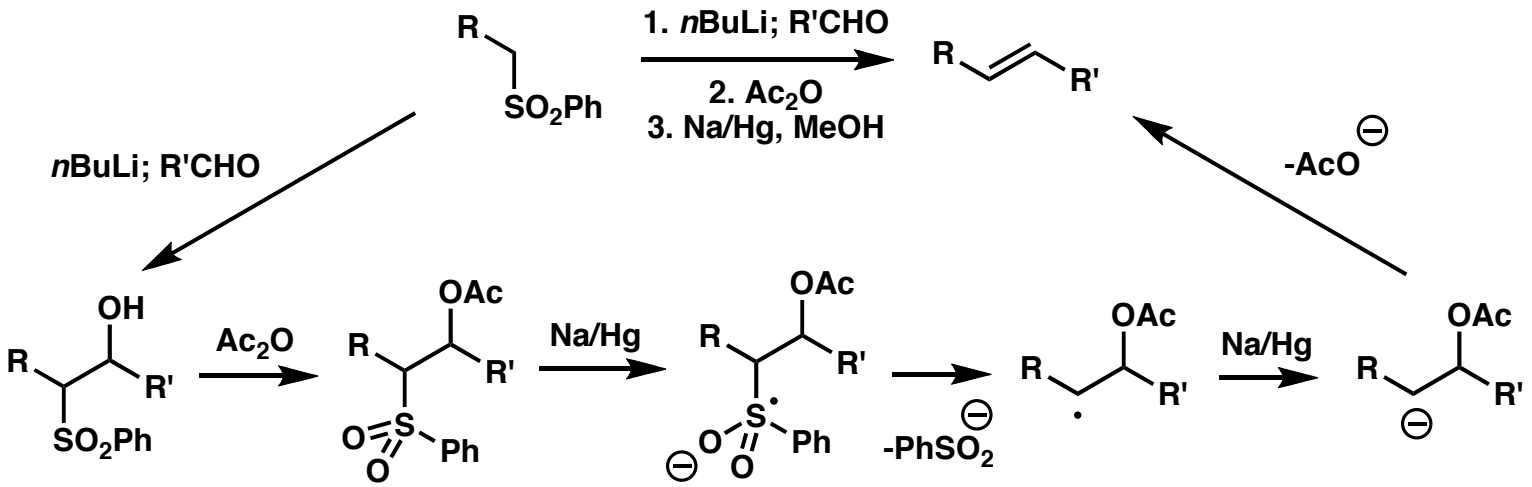
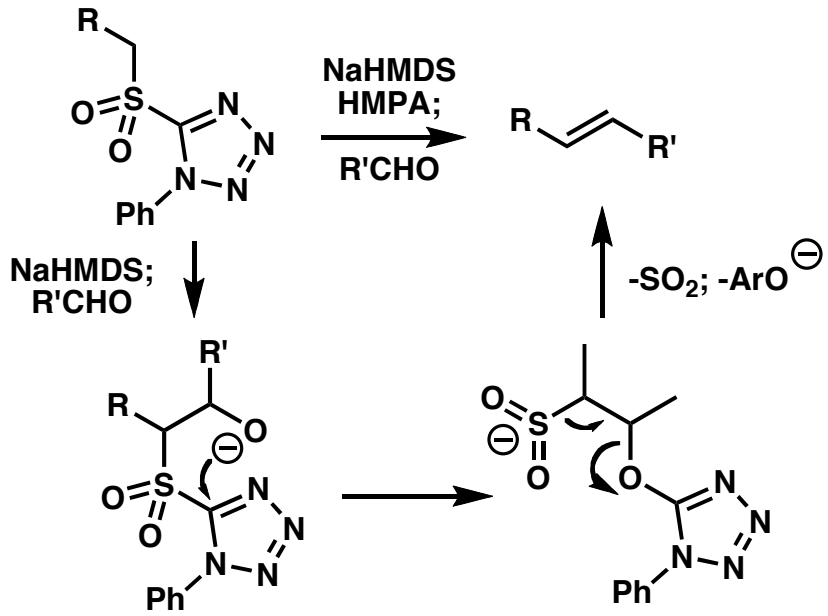
Olefination
Julia





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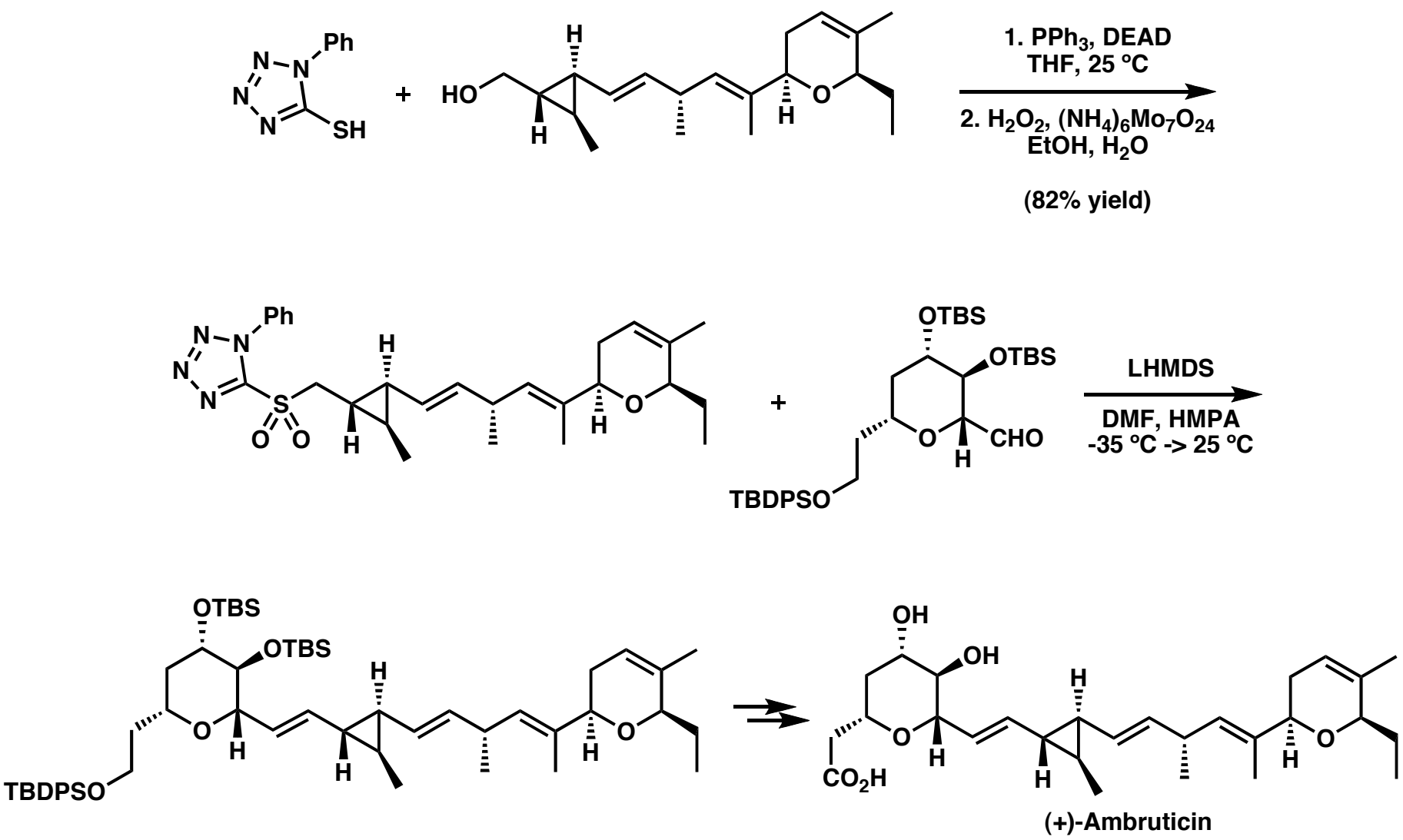
Olefination
Julia



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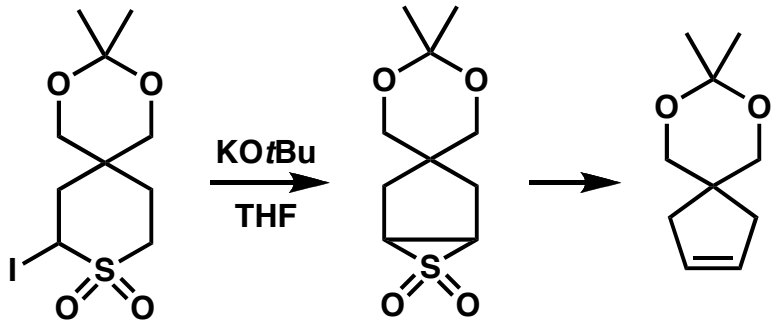
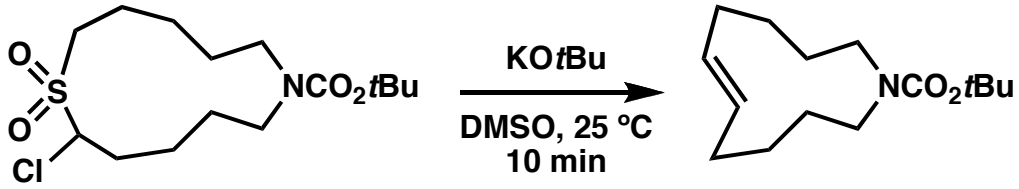
Olefination
Ramberg-Backlund





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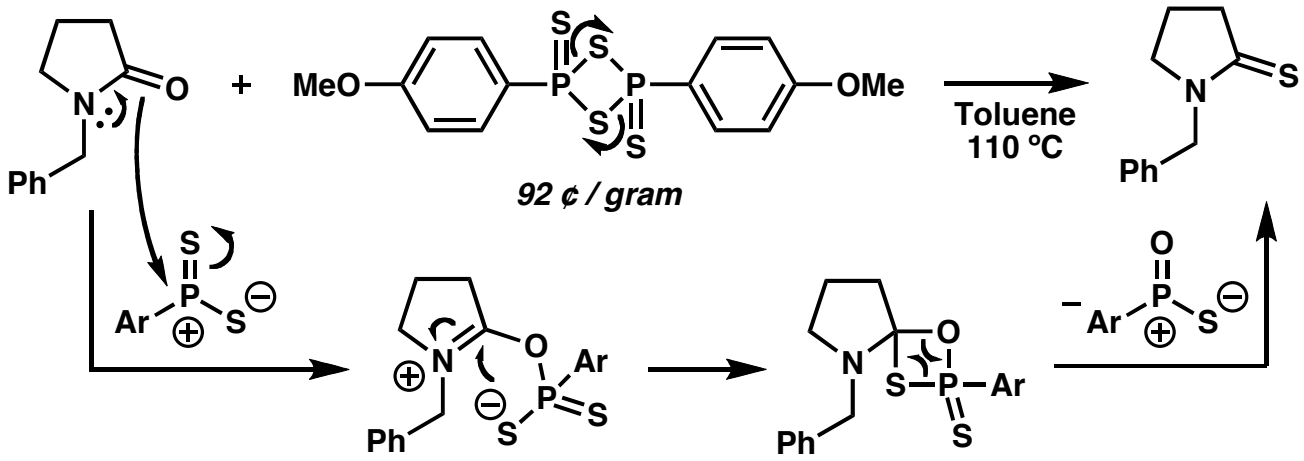
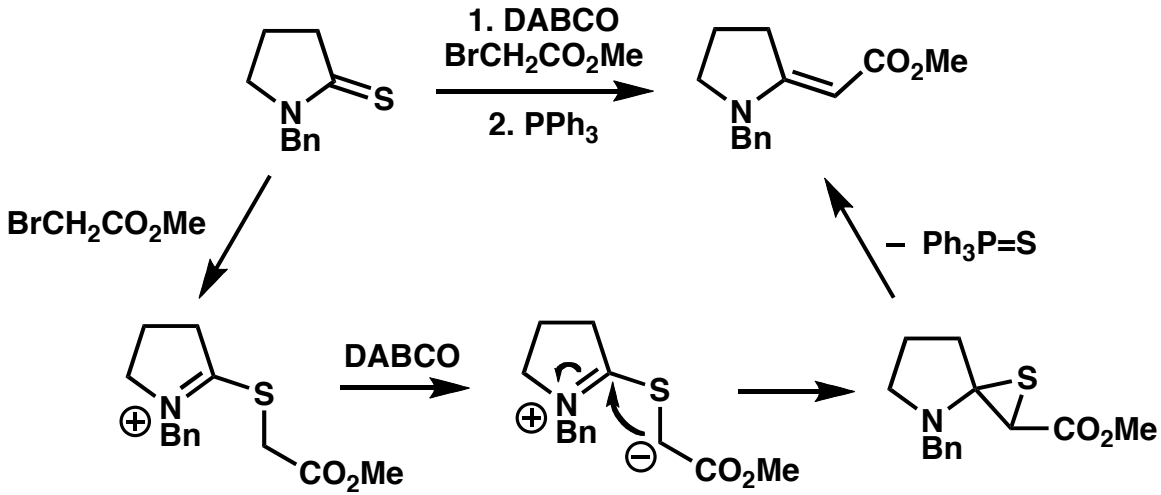
Olefination
Eschenmoser Sulfide Contraction





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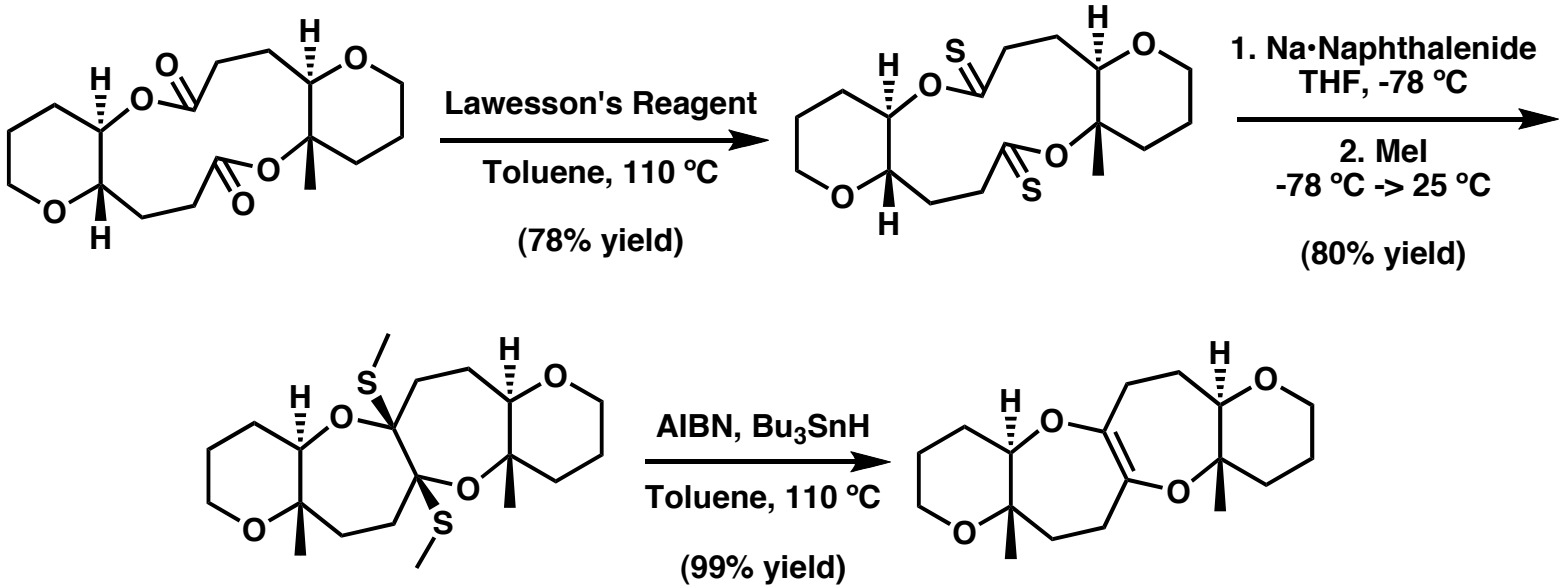
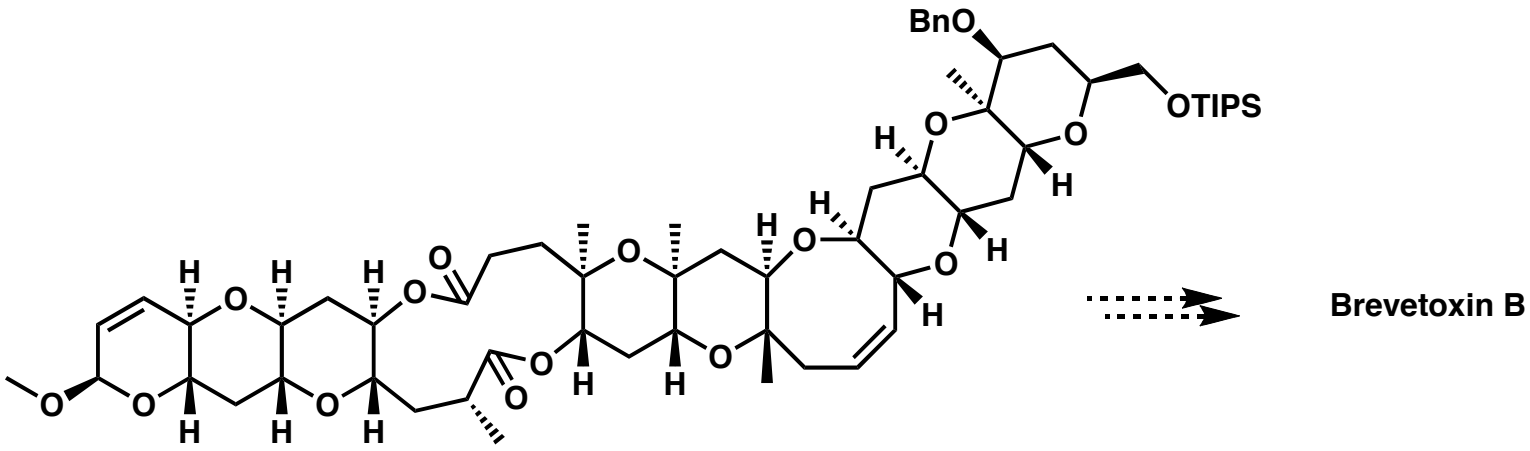
Olefination
Nicolaou Ring Contraction





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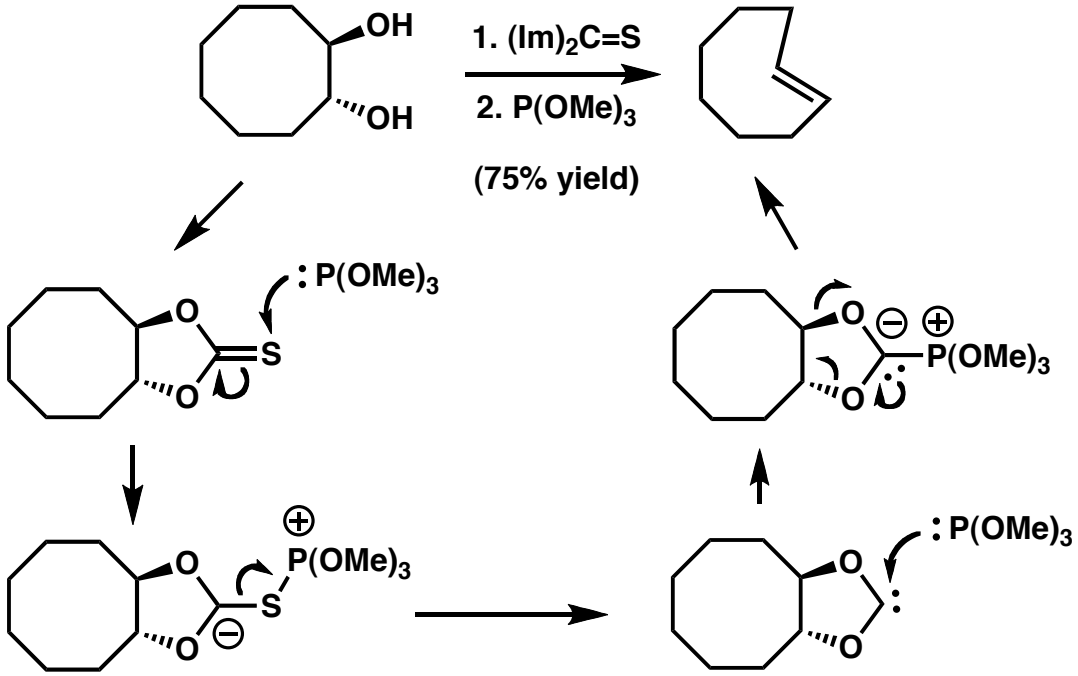
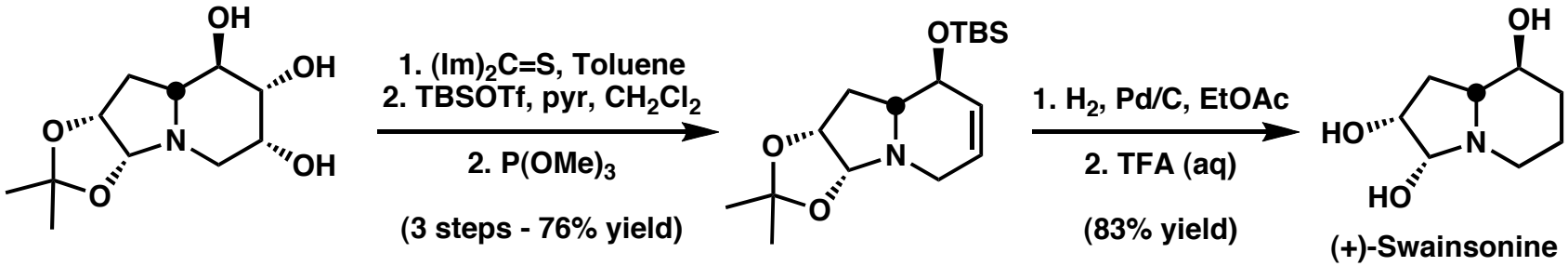
Olefination
Corey-Winter





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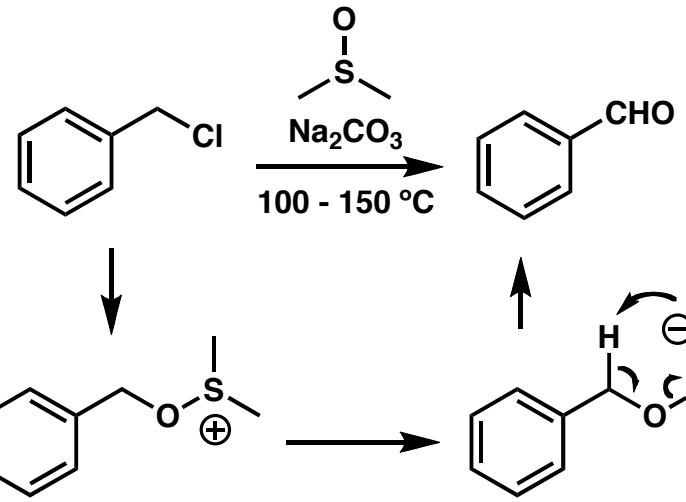
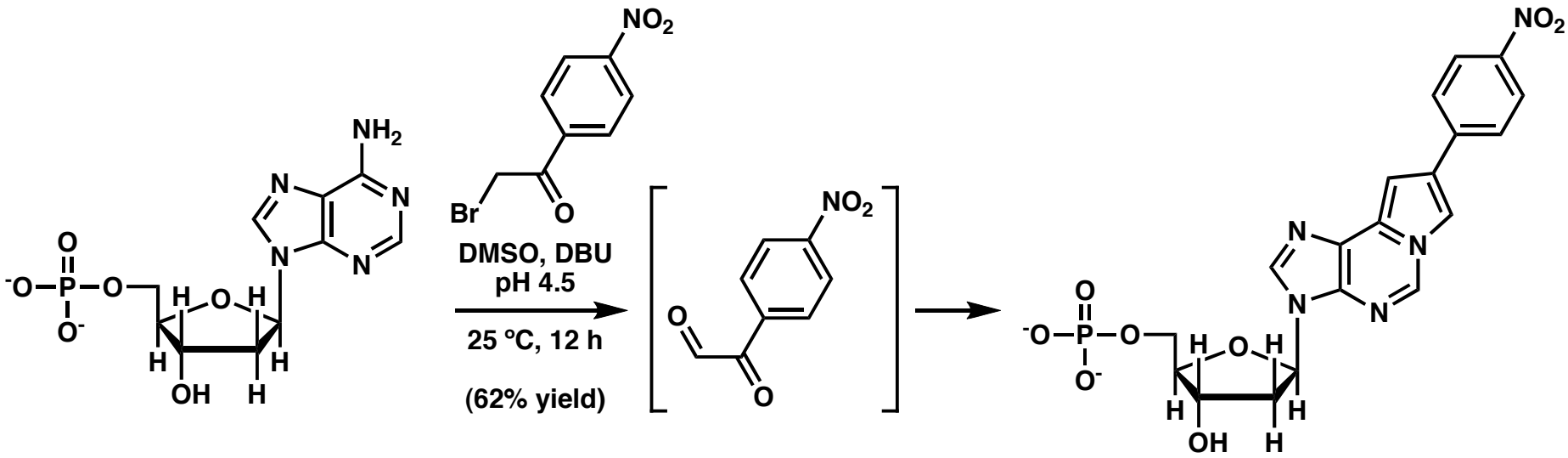
Oxidation
Kornblum





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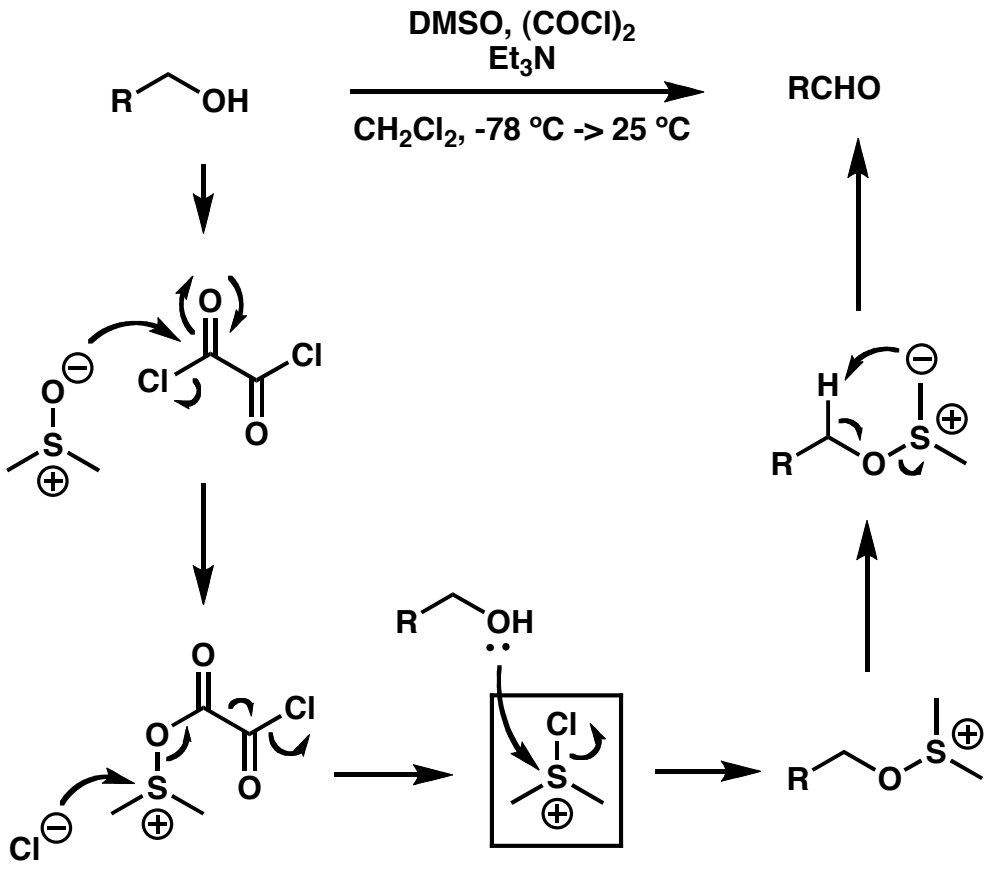
Oxidation
Moffatt-Swern



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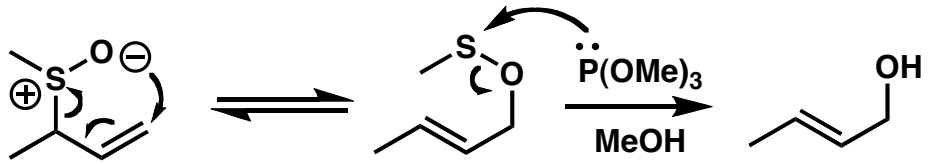
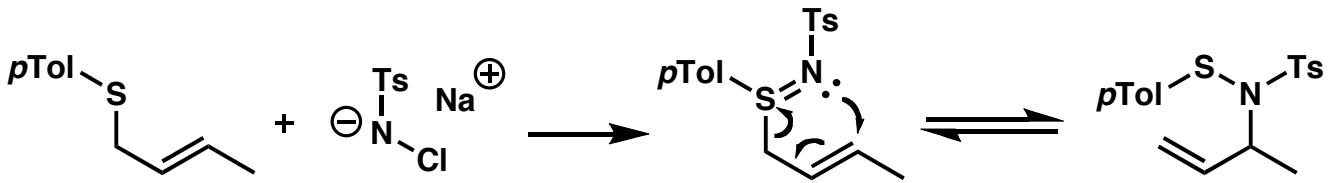
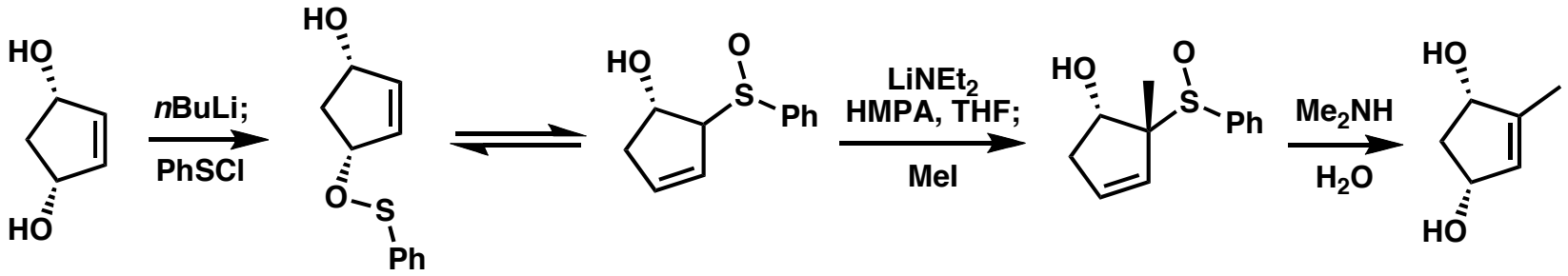
Mancuso, A. J.; Swern, D. *Synthesis*. 1981, 161-185.

Functionalization
Mislow-Evans Rearrangement







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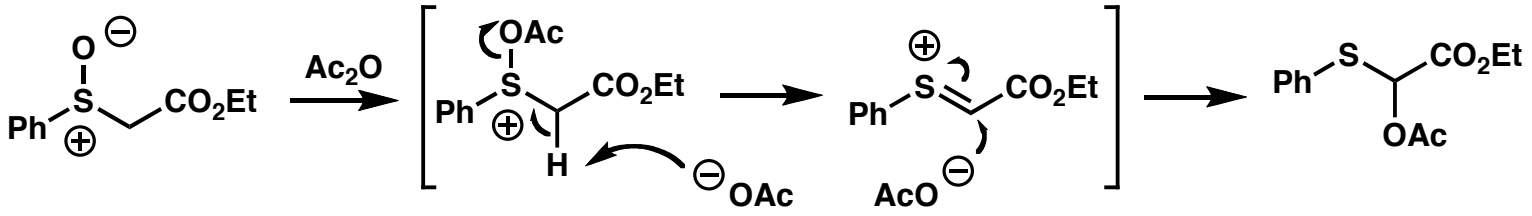
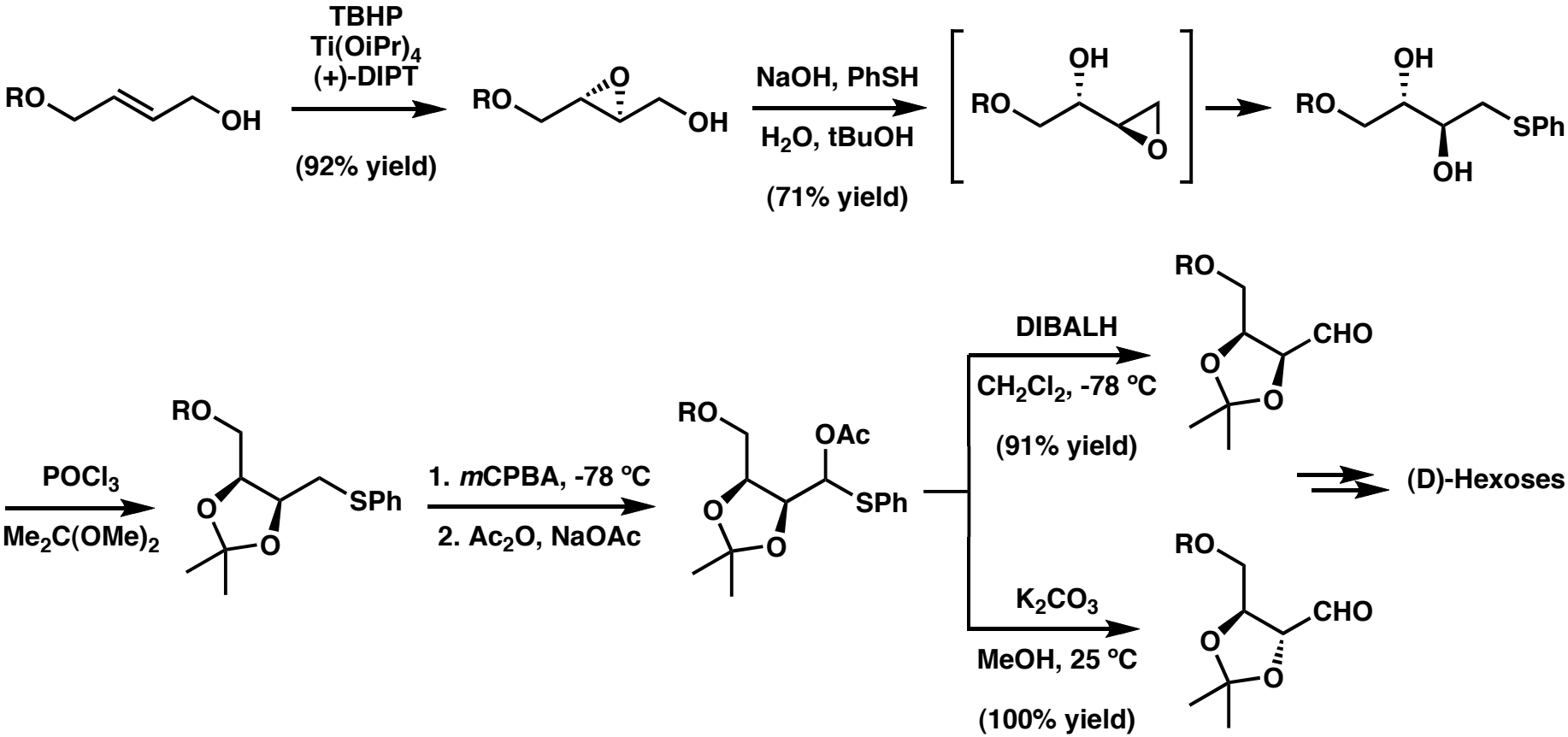
Evans, D. A.; Andrews, G. C.; Sims, C. L. *J. Am. Chem. Soc.* 1971, *93*, 4956-4957.

Functionalization
Pummerer Rearrangement





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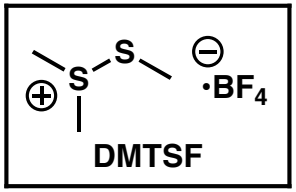
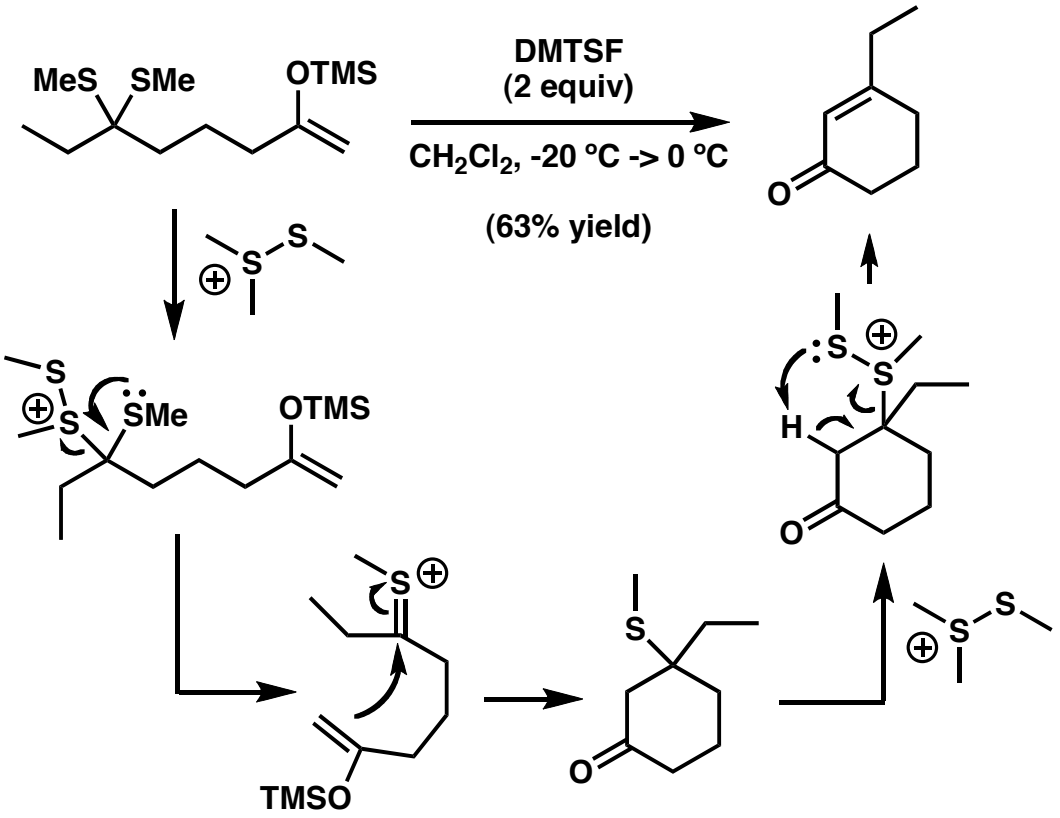
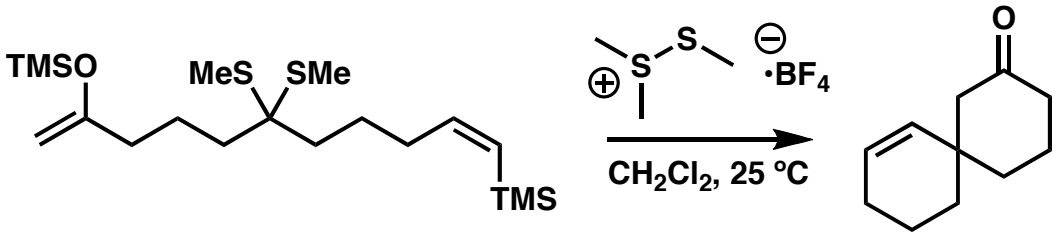
Ko, S. Y.; Lee, A. W. M.; Masamune, S.; Reed, L. A. III, Sharpless, K. B.; Walker, F. J. *Science*. 1983, *220*, 949.

Functionalization
DMTSF

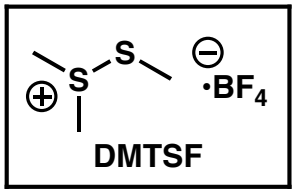


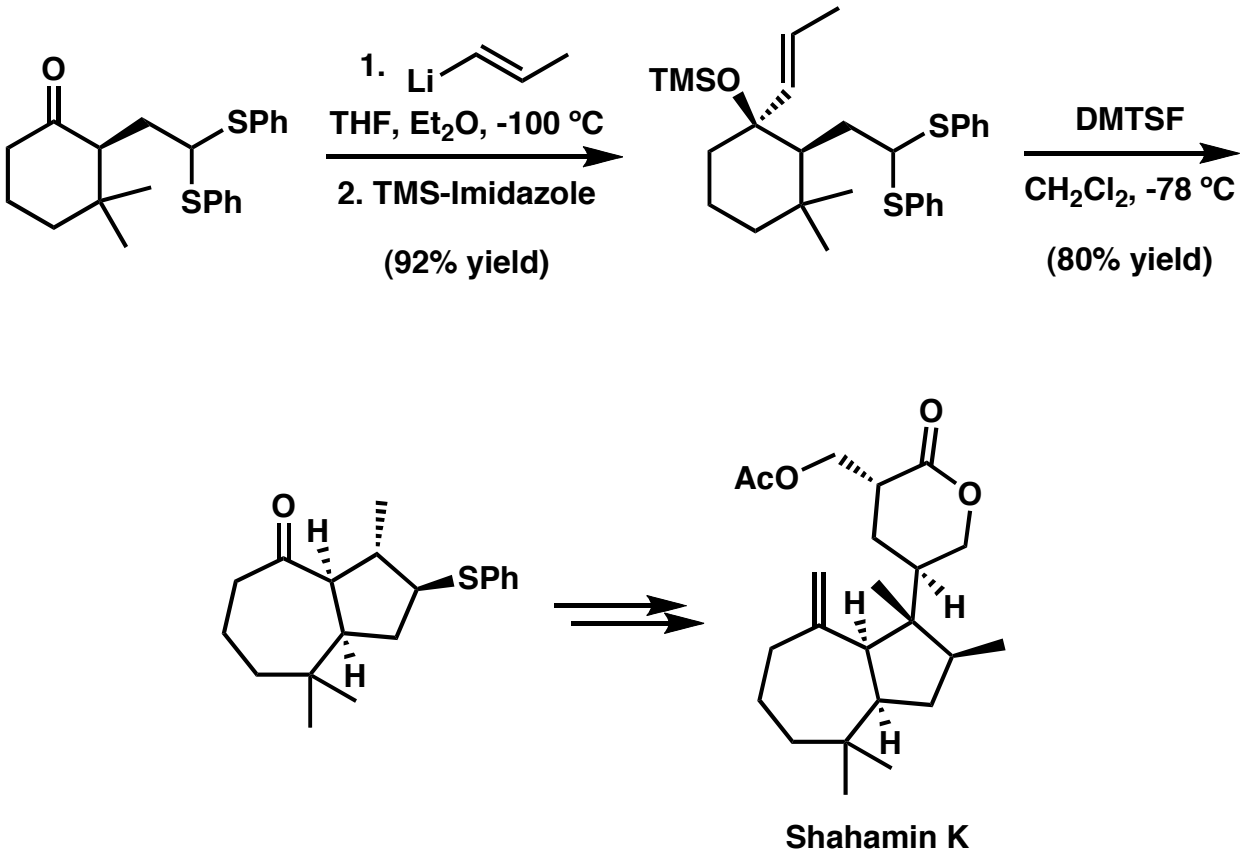




Trost, B. M.; Murayama, E. *J. Am. Chem. Soc.* 1981, *103*, 6529-6530.

Functionalization
DMTSF





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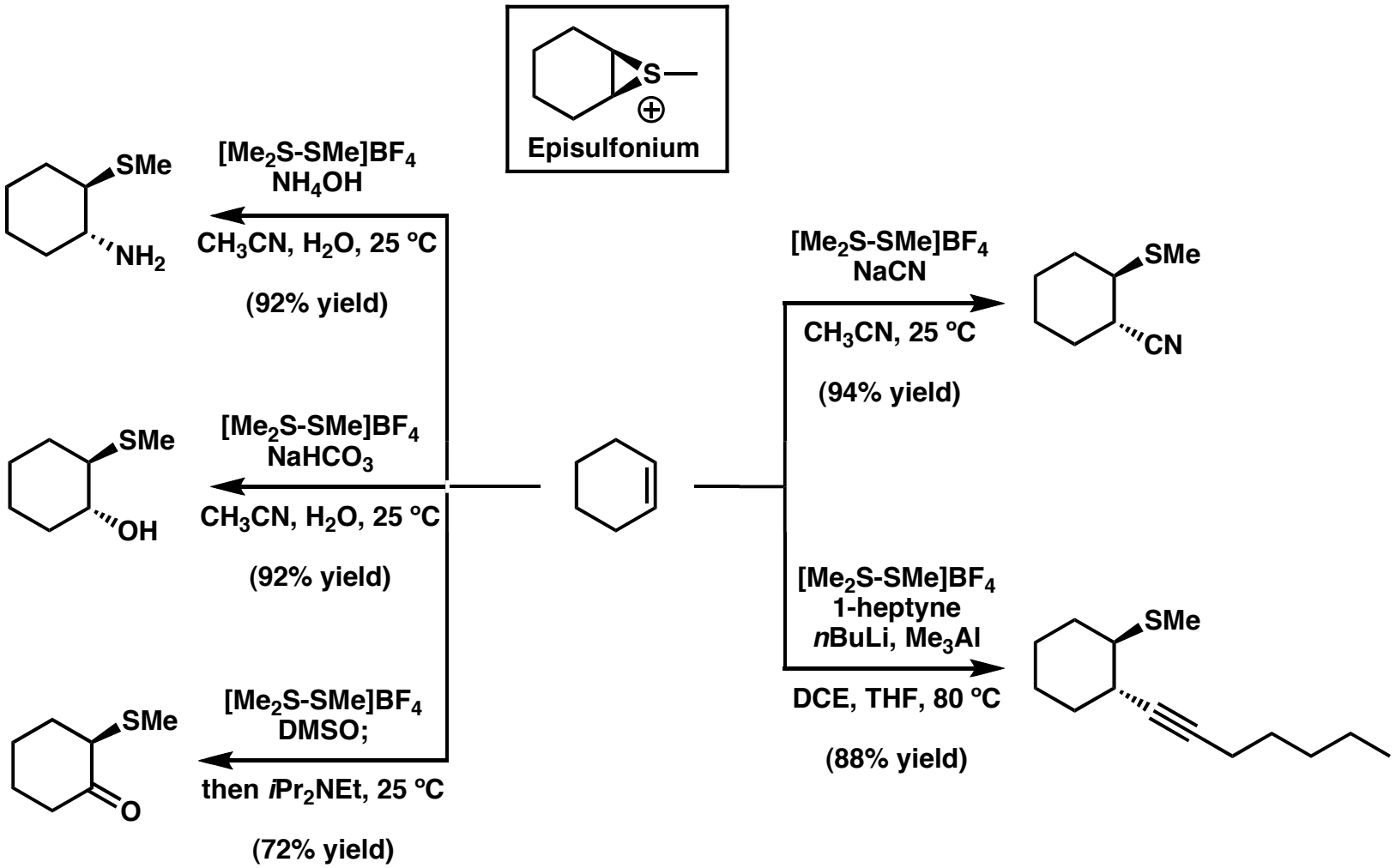
Functionalization
DMTSF



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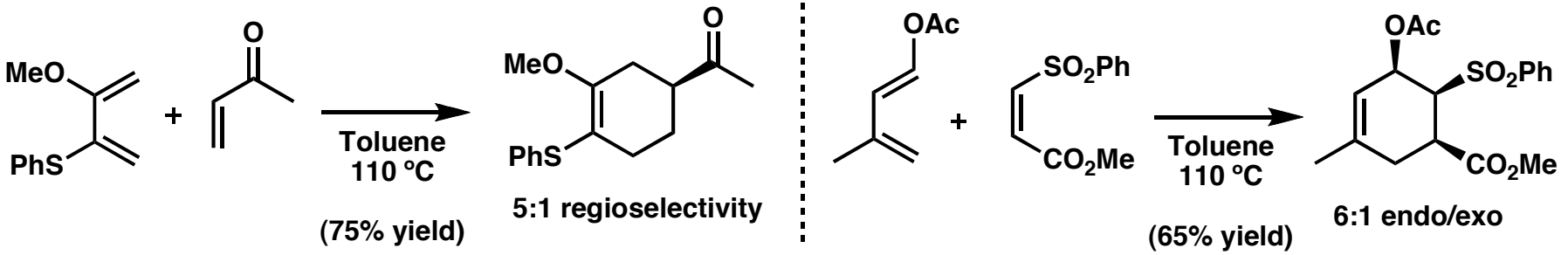
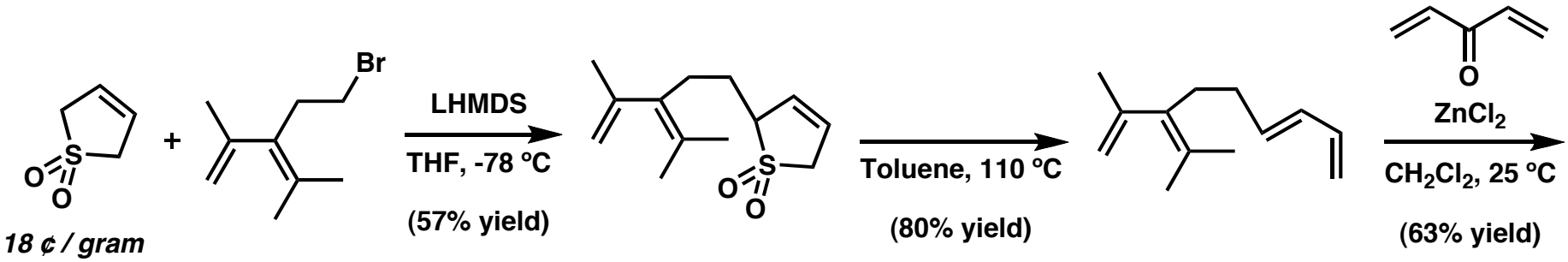
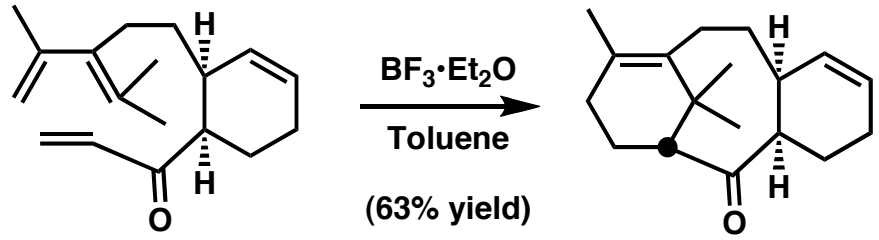
Functionalization
Diels-Alder Controlling Group







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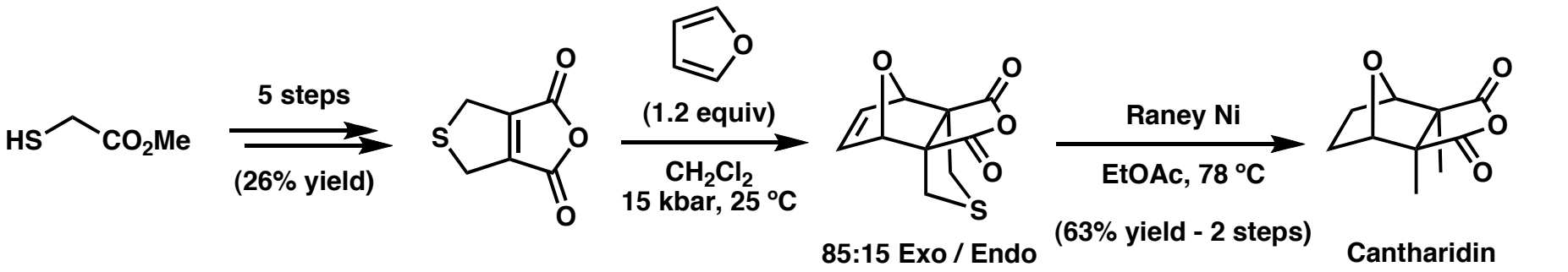
Sulfur-Templated Total Synthesis
Cantharidin



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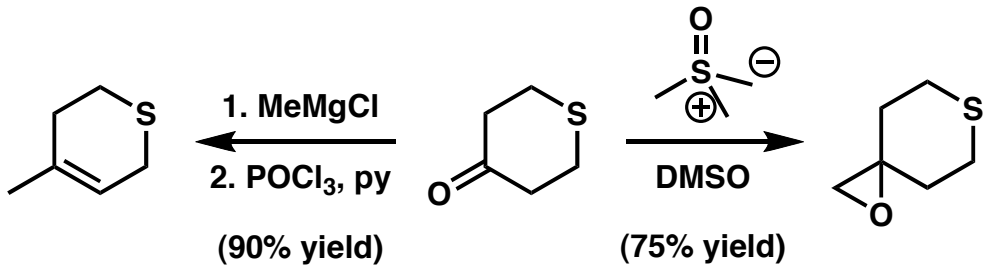
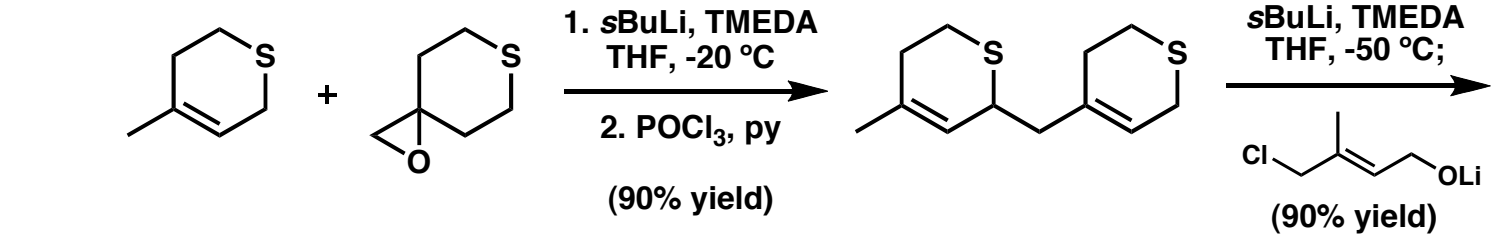
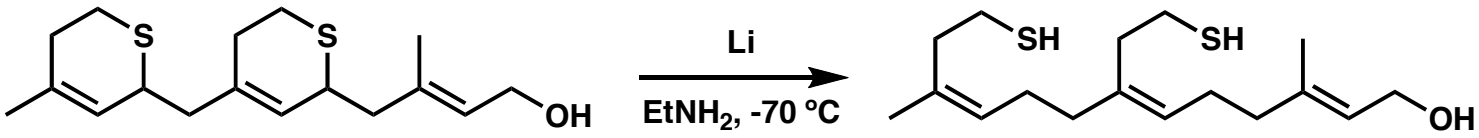
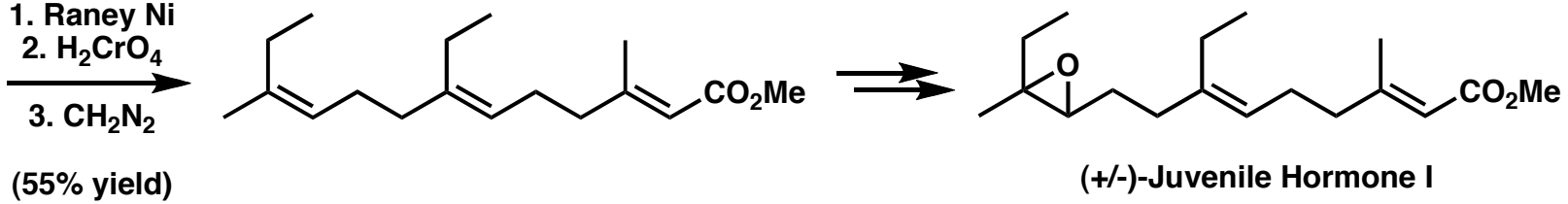
Sulfur-Templated Total Synthesis
Juvenile Hormone









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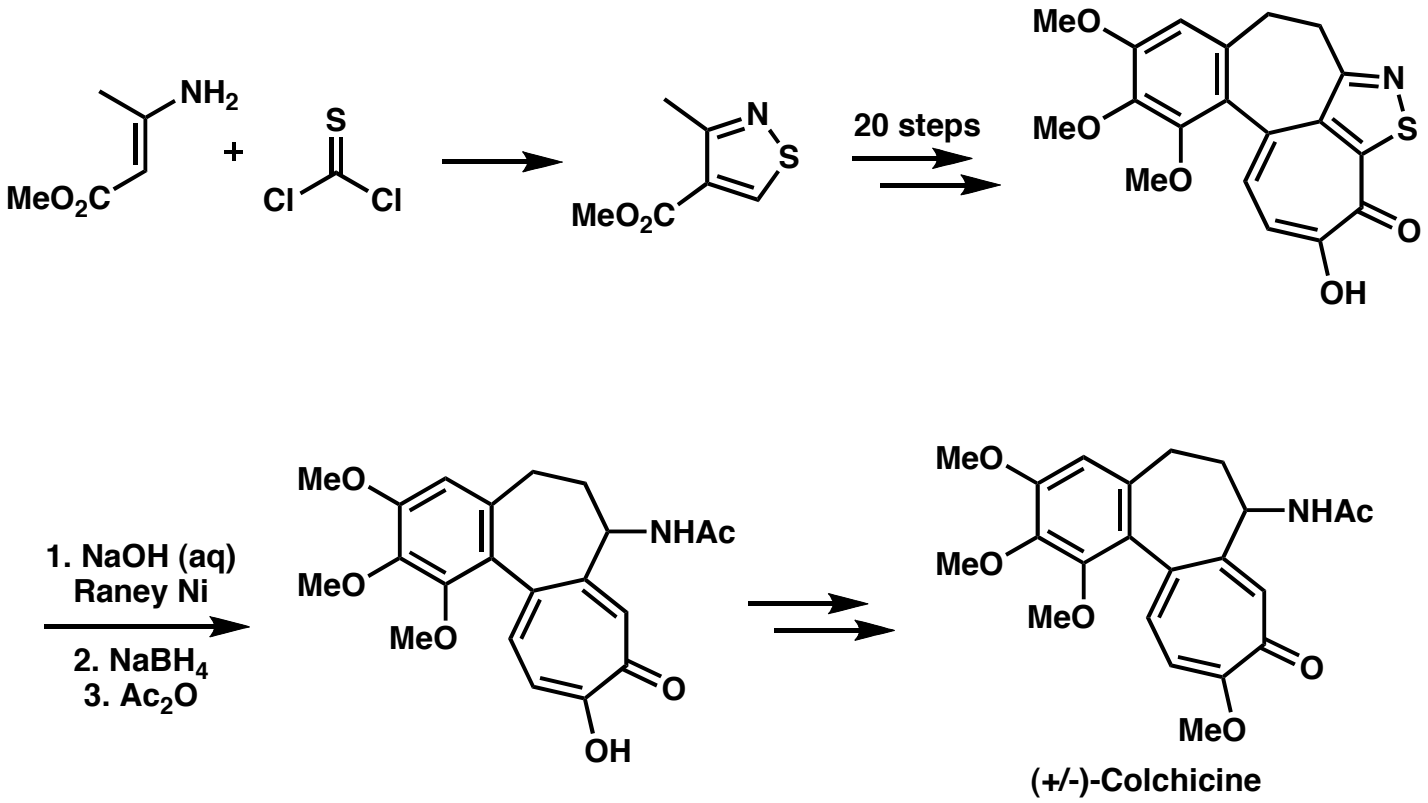
Sulfur-Templated Total Synthesis
Colchicine



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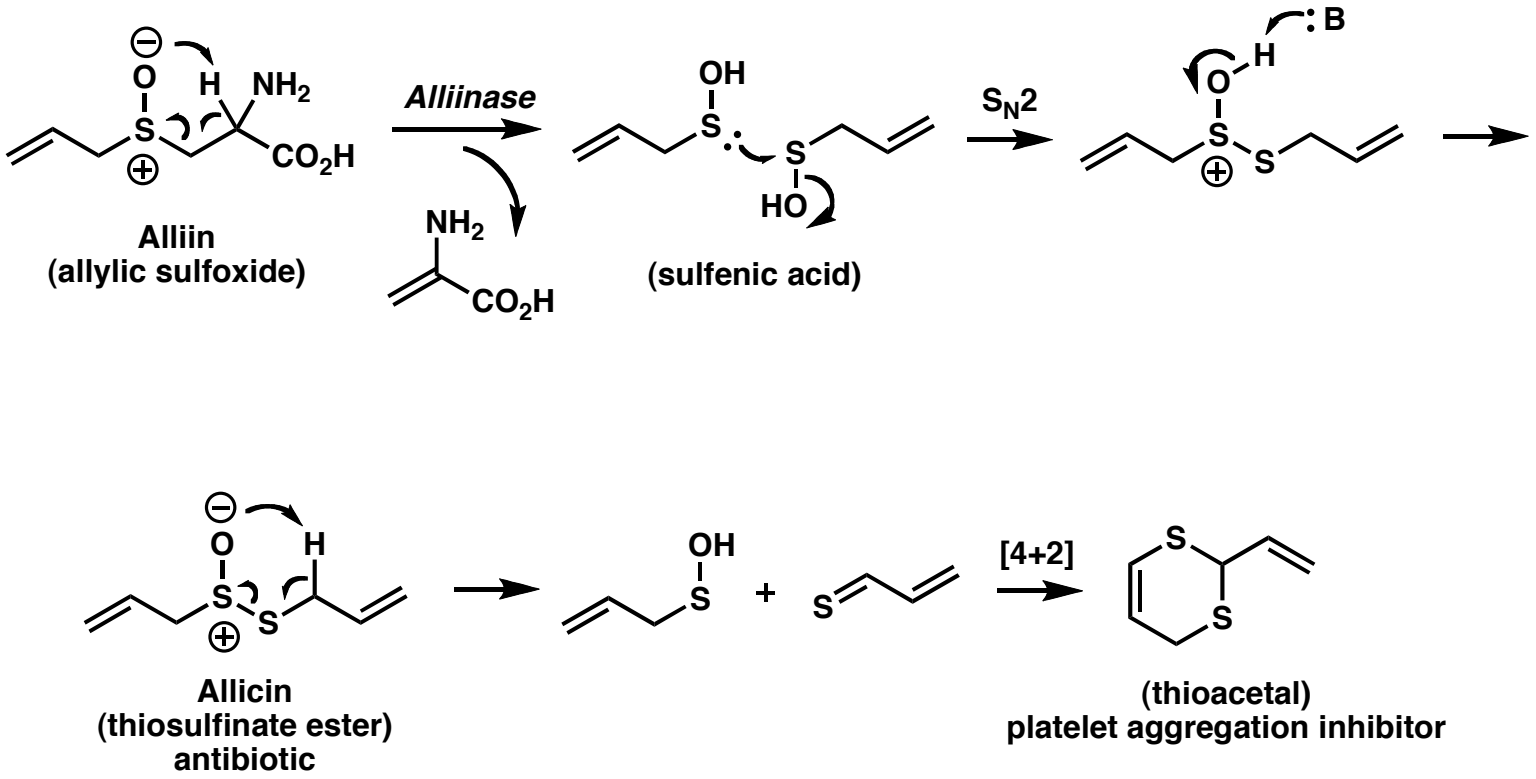
Allium Chemistry
Garlic



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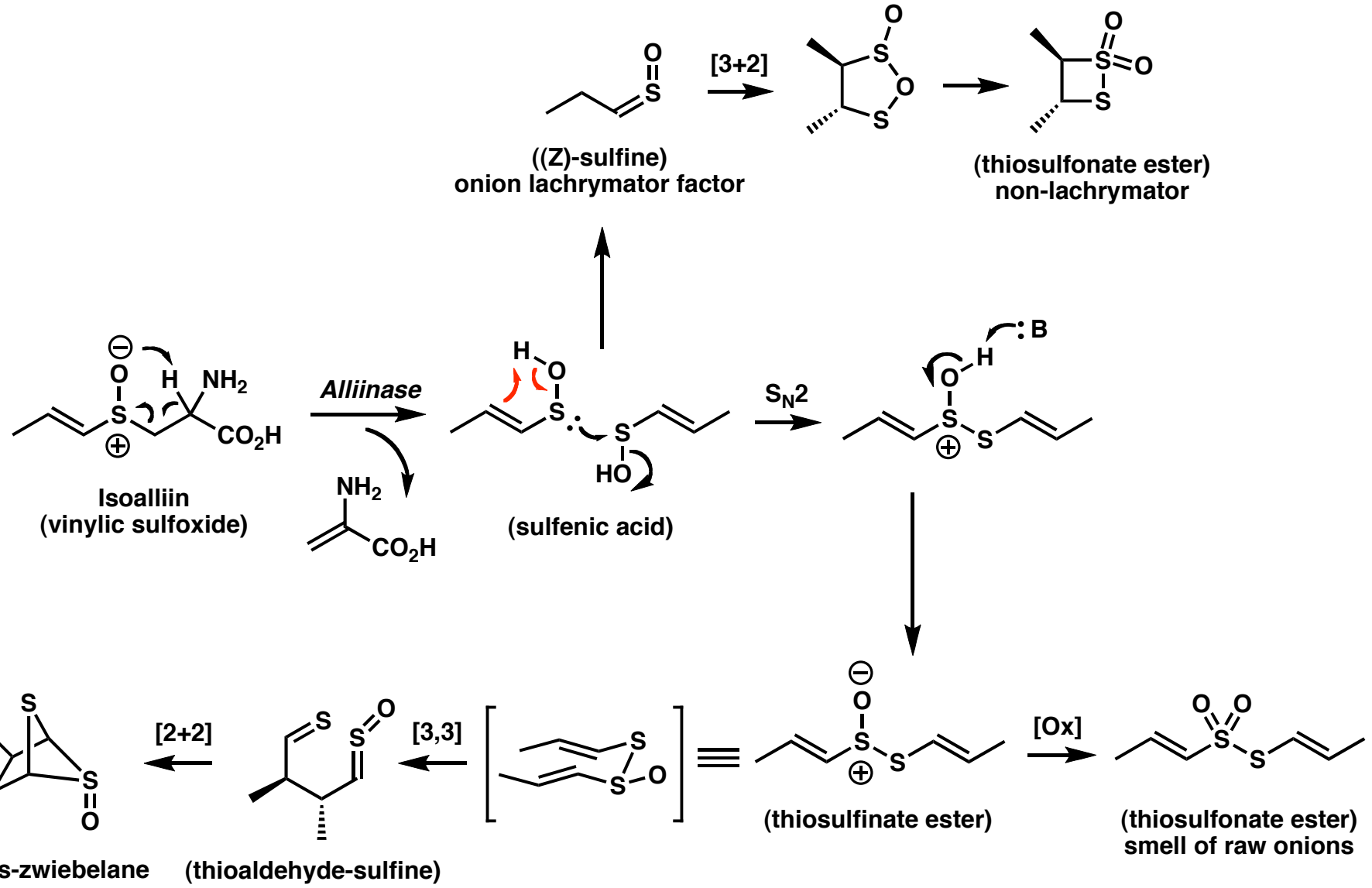
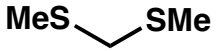
Allium Chemistry
Onion



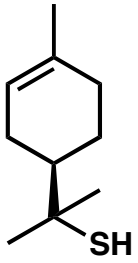
Block, E. *Angew. Chem. Int. Ed. Engl.* 1992, *31*, 1135-1178.

Organosulfur Compounds

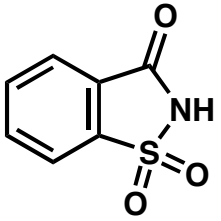
methylmercaptan
(natural gas additive)



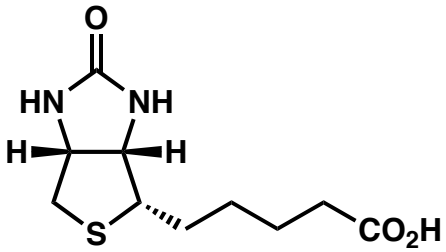
bis(methylthio)methane
(black truffles)



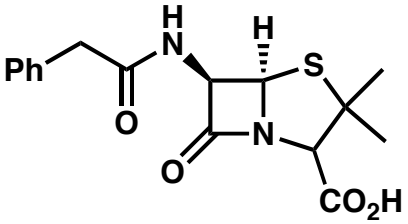
1-*p*-menthene-8-thiol
(grapefruit)



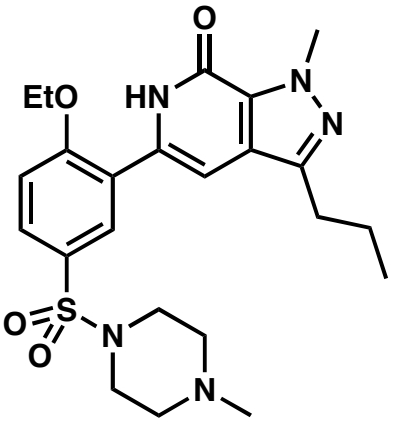
saccharin
(sweetener)



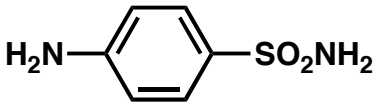
biotin



penicillin G
(antibiotic)



Viagra
(erectile dysfunction)



sulfanilamide
(antibiotic)

Summary

Nomenclature

Elimination

Radical Reaction

C-C Bond Formation

Olefination

Oxidation

Functionalization

Allium Chemistry