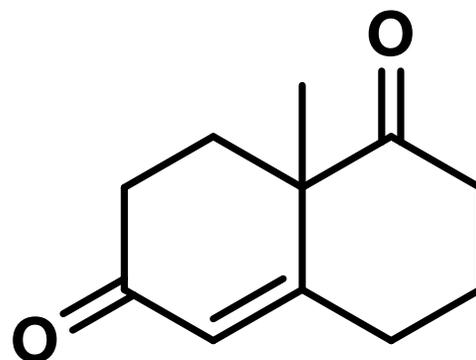


# Wieland-Miescher酮的相关合成



上海科技大学  
ShanghaiTech University



Wieland-Miescher酮 (WMK)

汇报人：吴宇翔  
时间：2024.11.20

立志成才 报国裕民





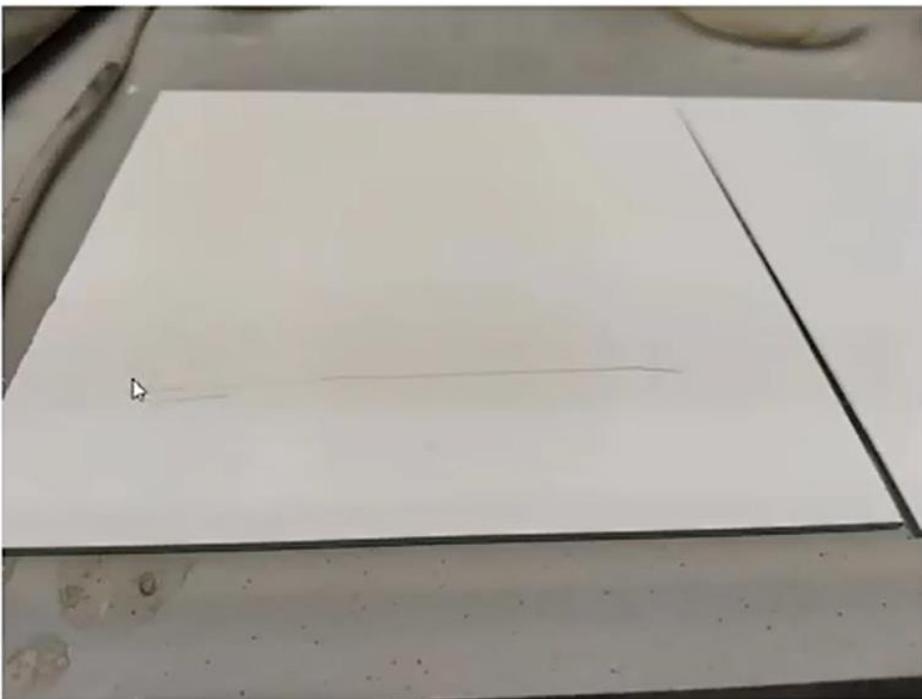
## 刮大板之前

- 1, 量小 (50 mg粗品一块大板)
- 2, TLC点太近不适合过柱子
- 3, 点TLC摸好极性





# 上大板之前



左右1.5cm，下2.5cm



松紧度以比较难抽动为佳

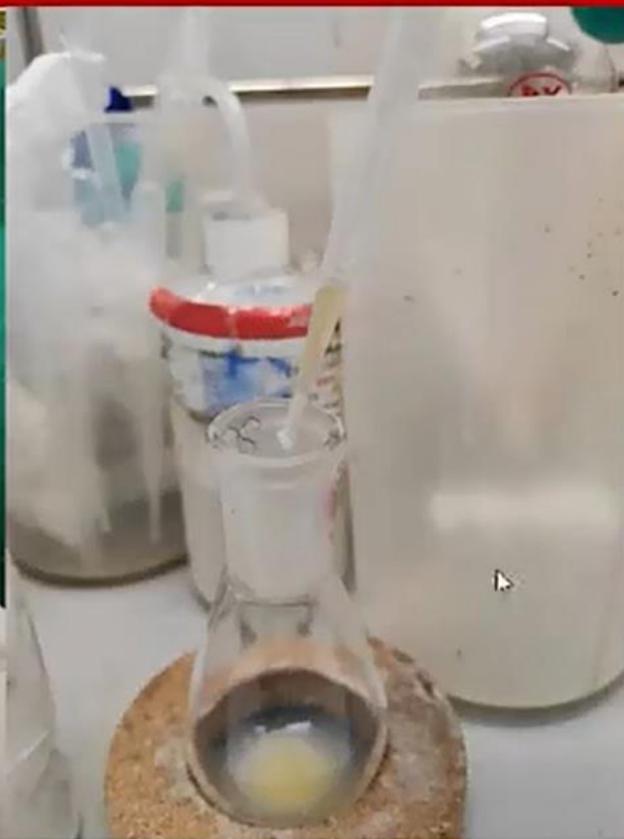




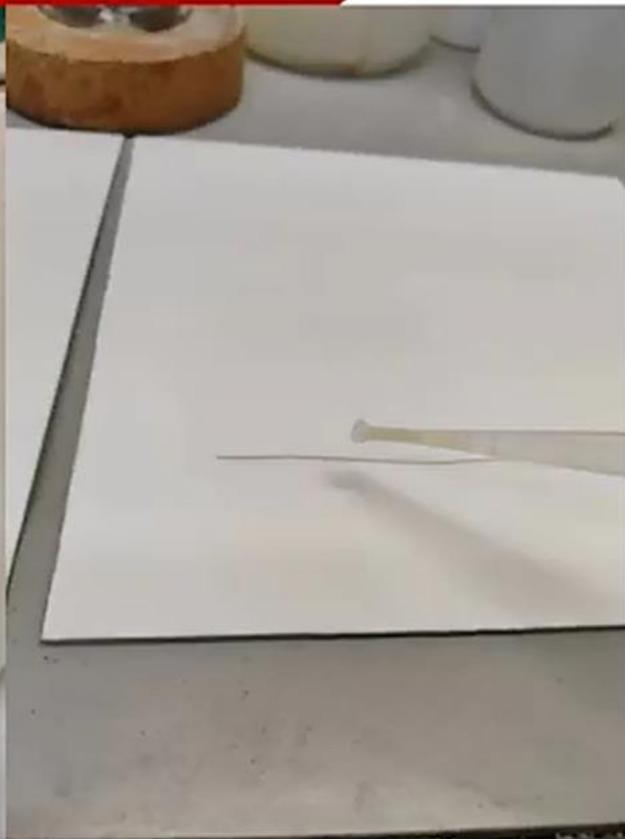
# 画板



二氯溶解



重力下不漏液



吹干



侧边点一个点





# 跑板

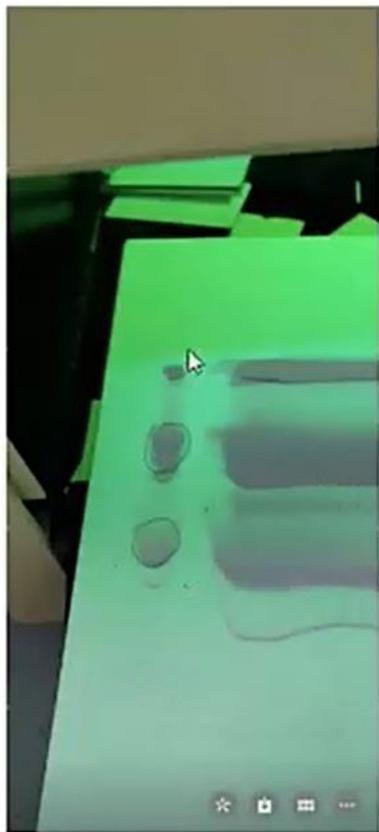


边缘效应

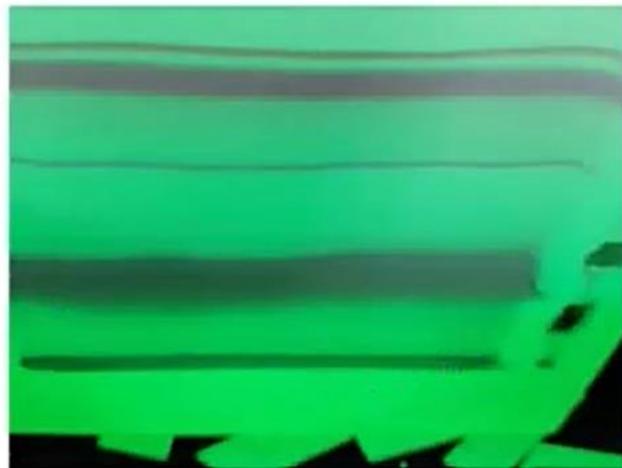




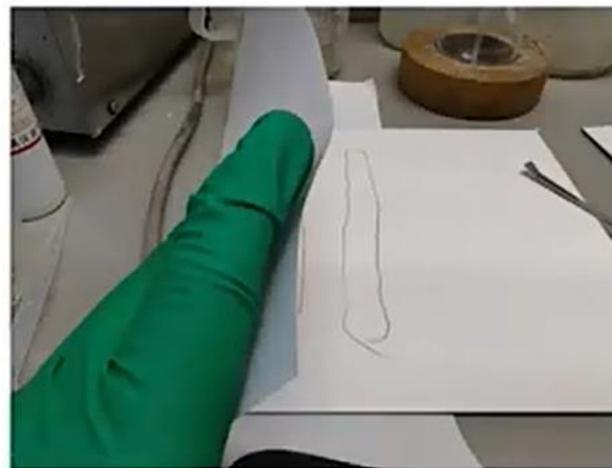
# 刮板



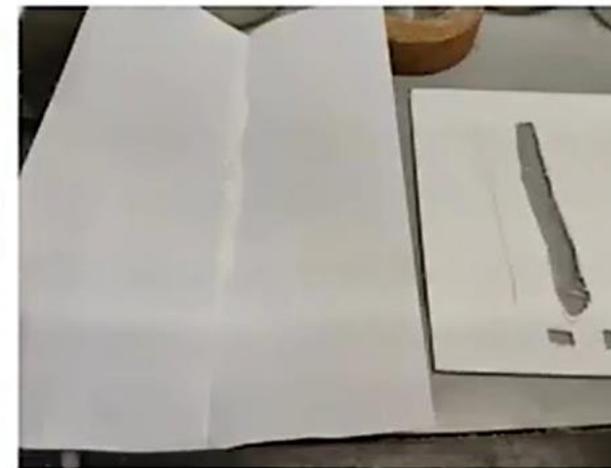
先送一个LCMS



要纯度还是要数量？



画好之后刮

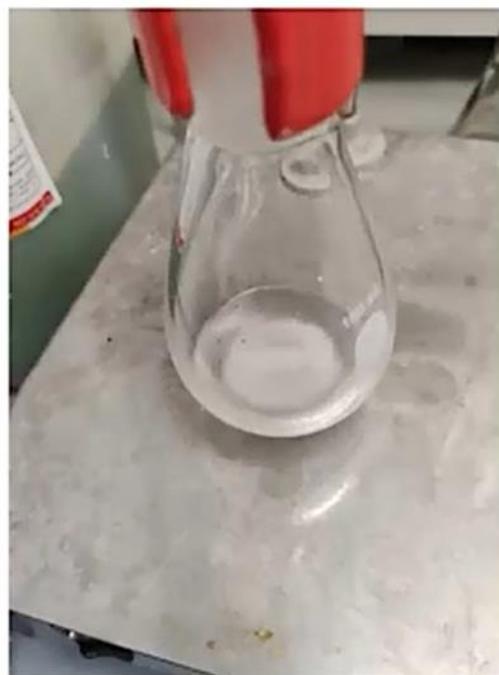


碾碎





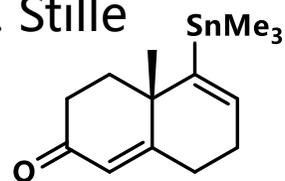
# 打浆与抽滤



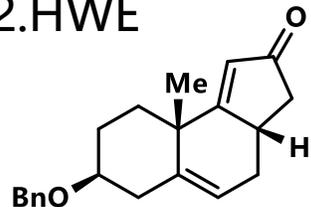
大一点搅拌子  
溶剂选择  
抽滤



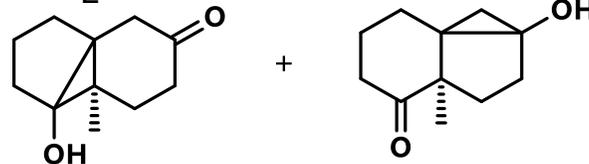
1. Stille



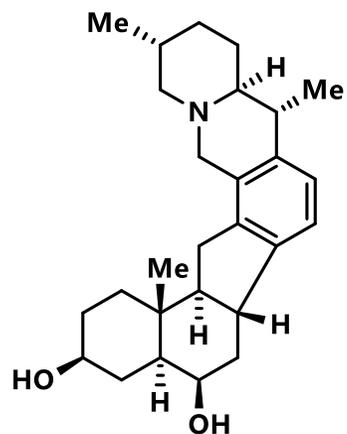
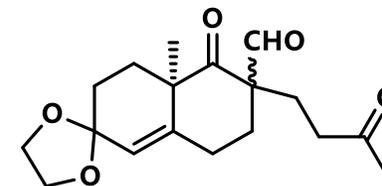
2. HWE



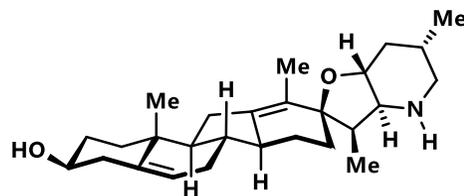
3. Sm<sub>2</sub>I



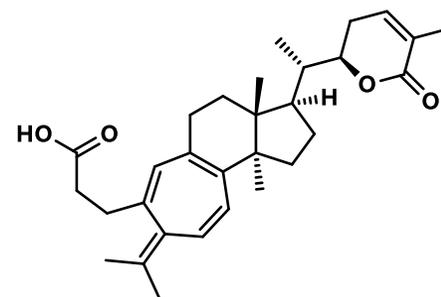
4. Robinson



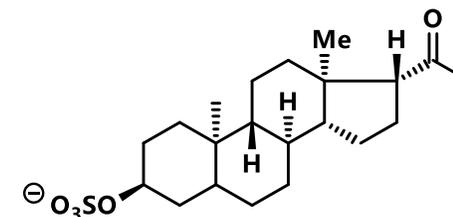
(+)-Heilonine



(-)-cyclopamine



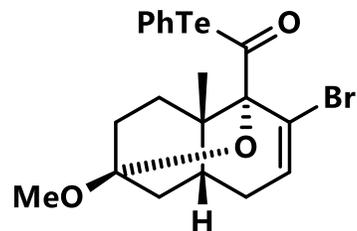
Lancilactone C



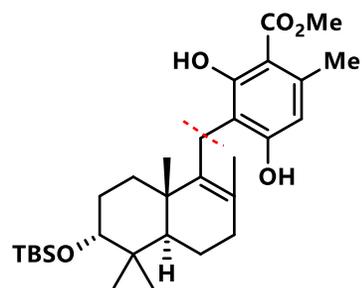
de novo



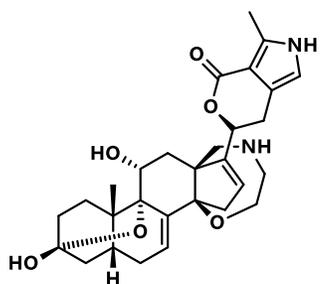
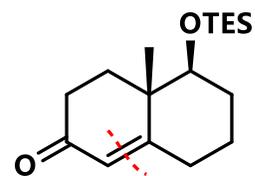
### 5. 自由基



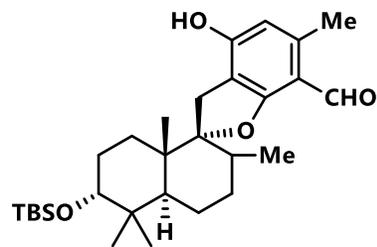
### 6. Friedel-Crafts



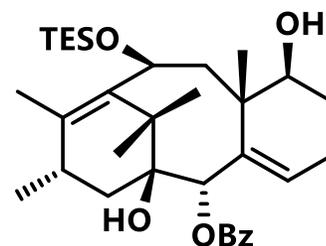
### 7. 臭氧氧化切断



batrachotoxin

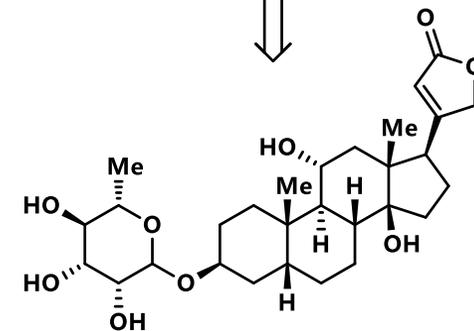
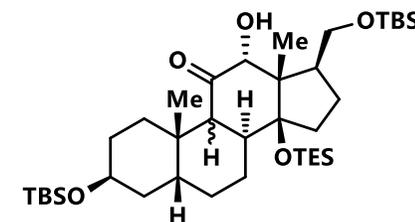
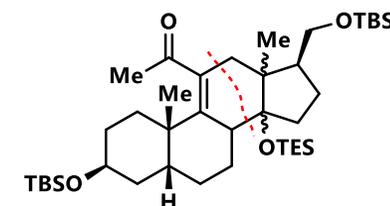


3-epi-F1839-I



taxol

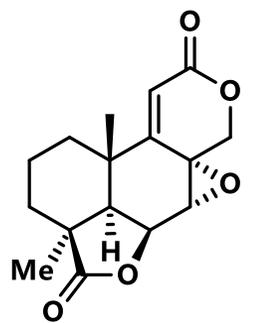
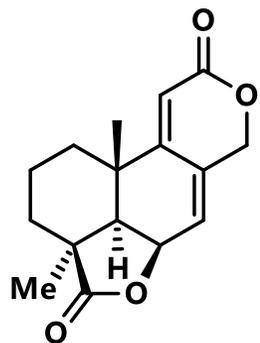
### 8. DA



Rhodexin A

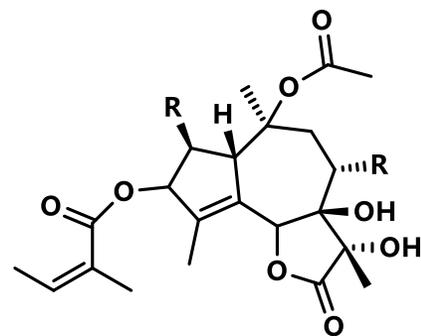
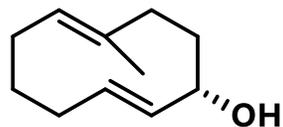


### 9. 对羰基加成



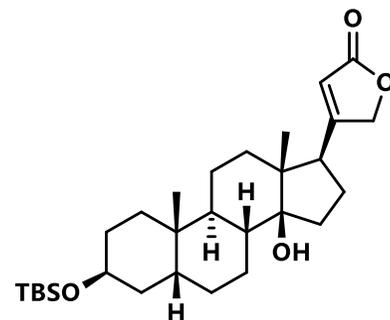
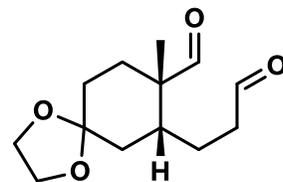
Oidiolactone C

### 10. Grob碎裂化



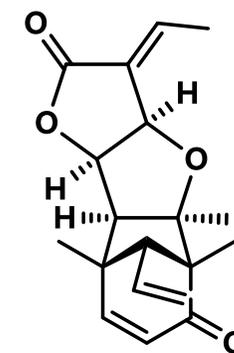
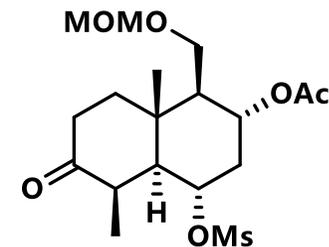
Thapsia

### 11. 臭氧化切断烯醇硅醚



(+)-Digitoxigenin

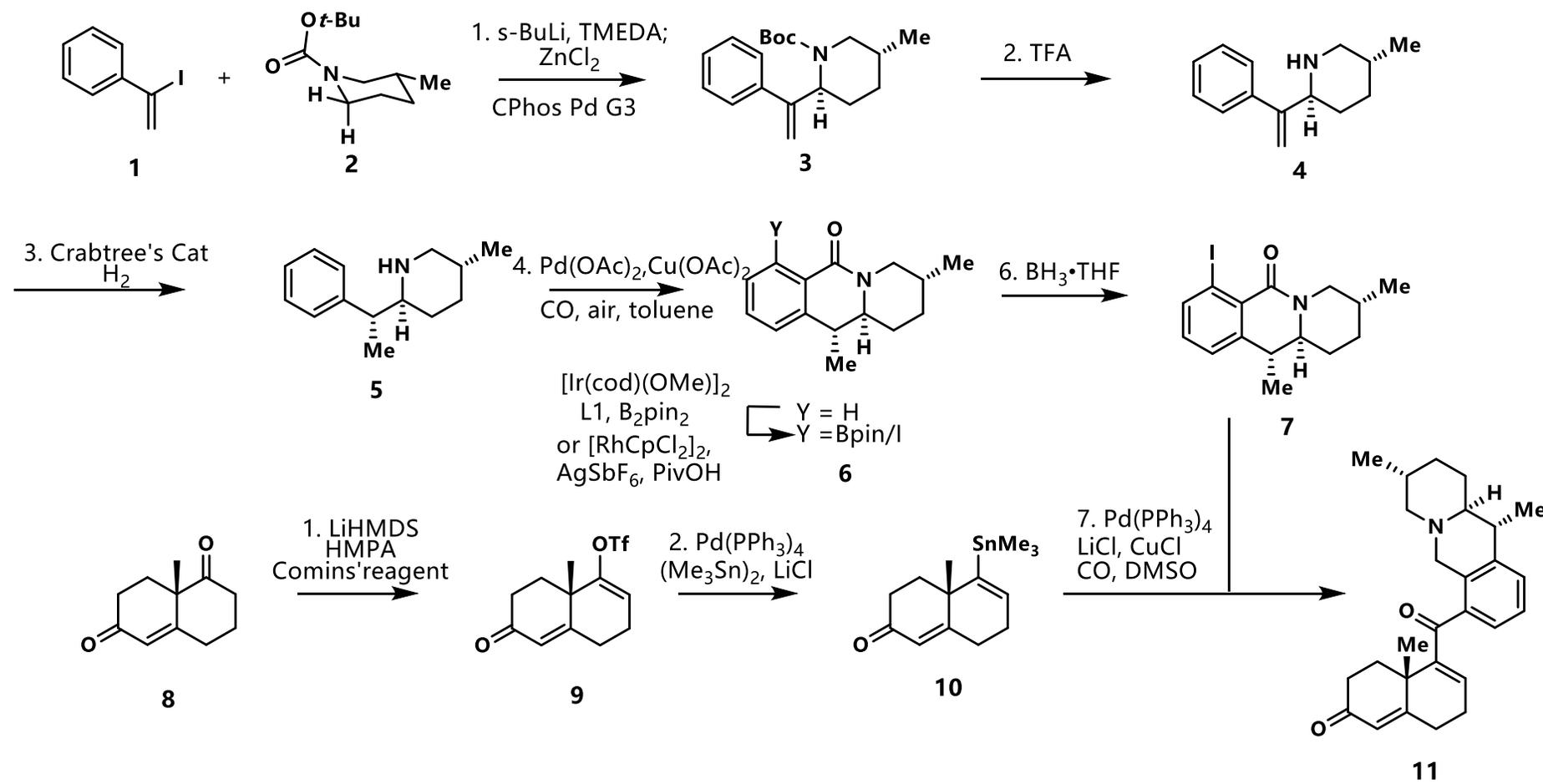
### 12. Grob碎裂化后aldol

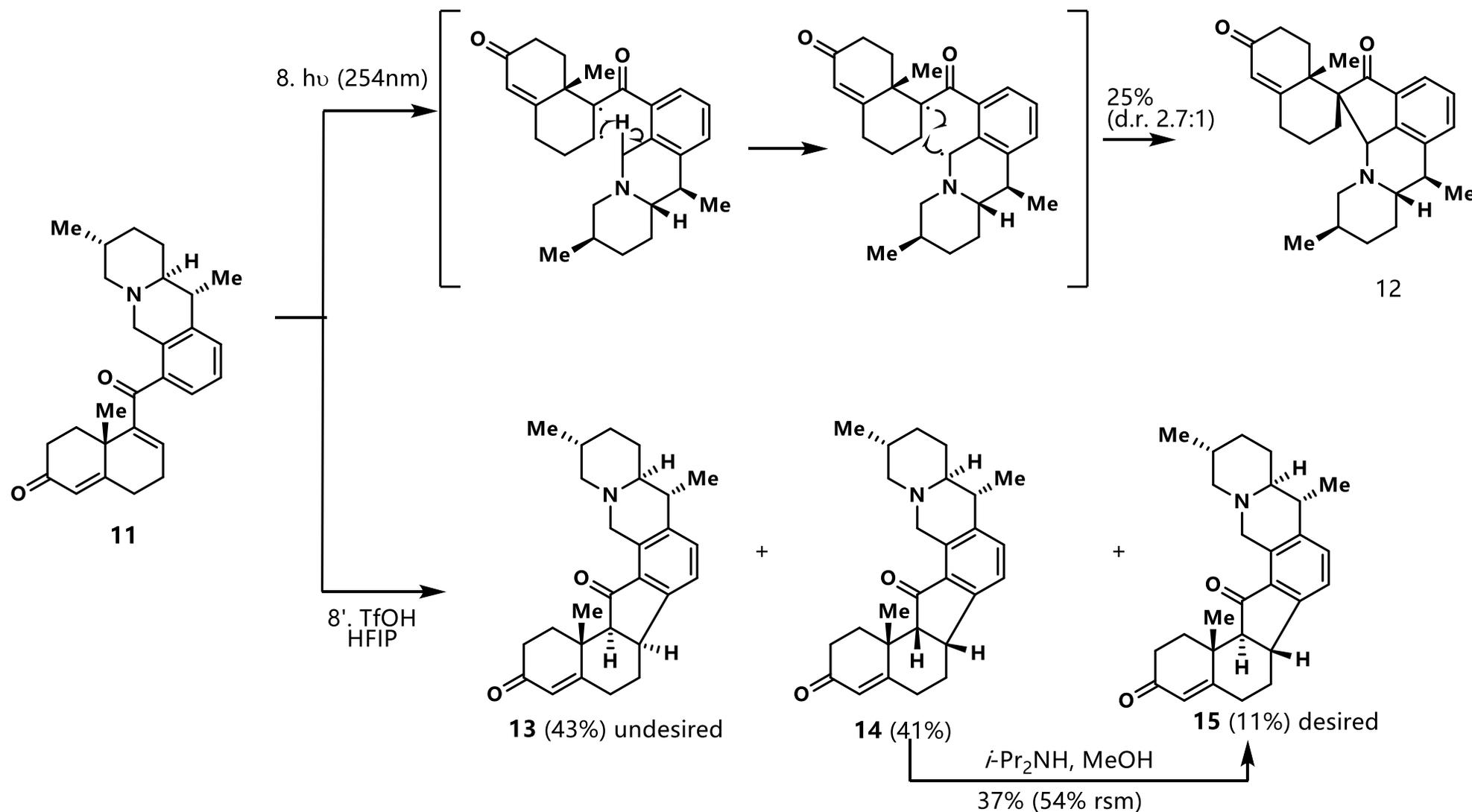


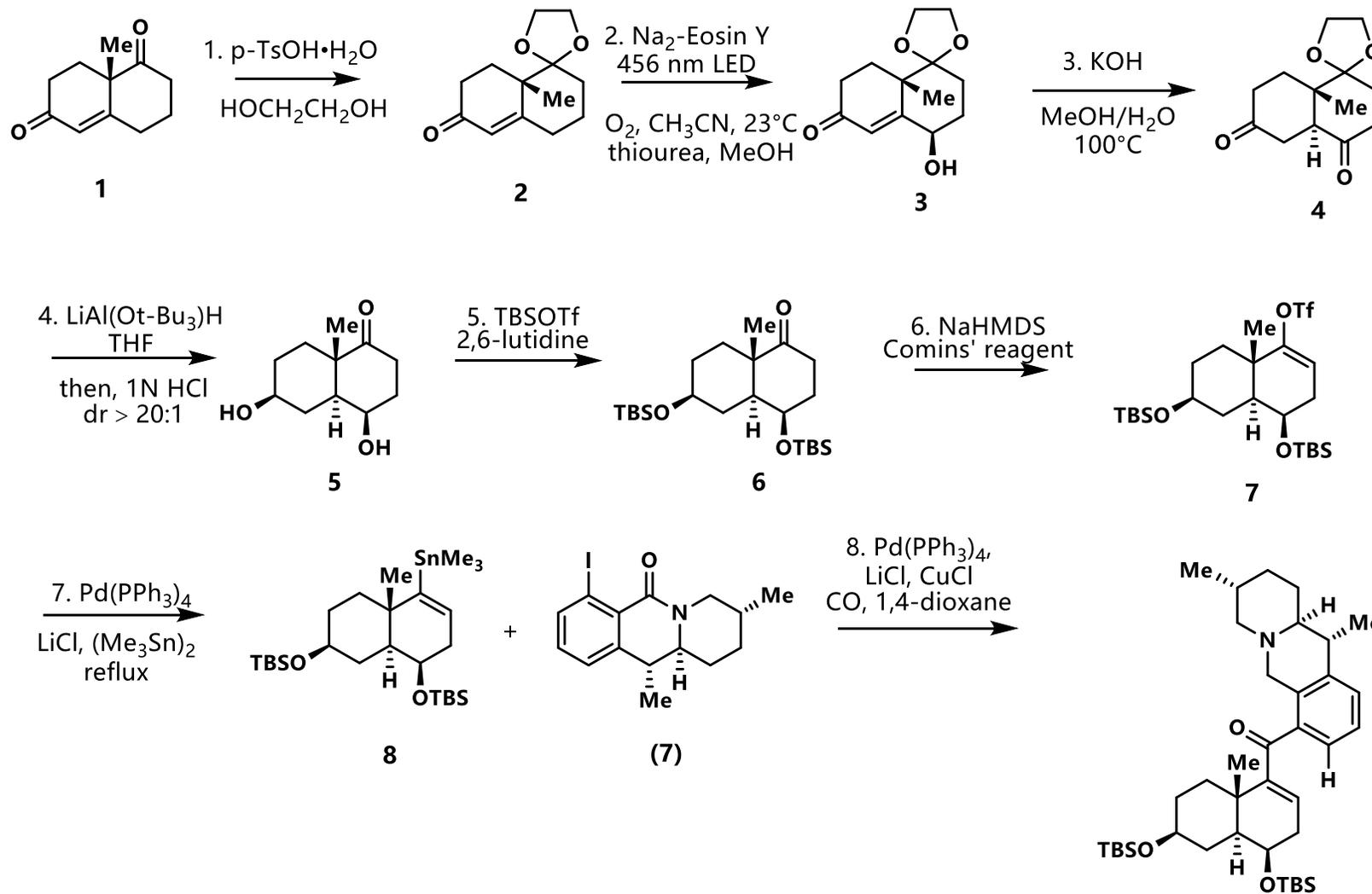
Pallambin D

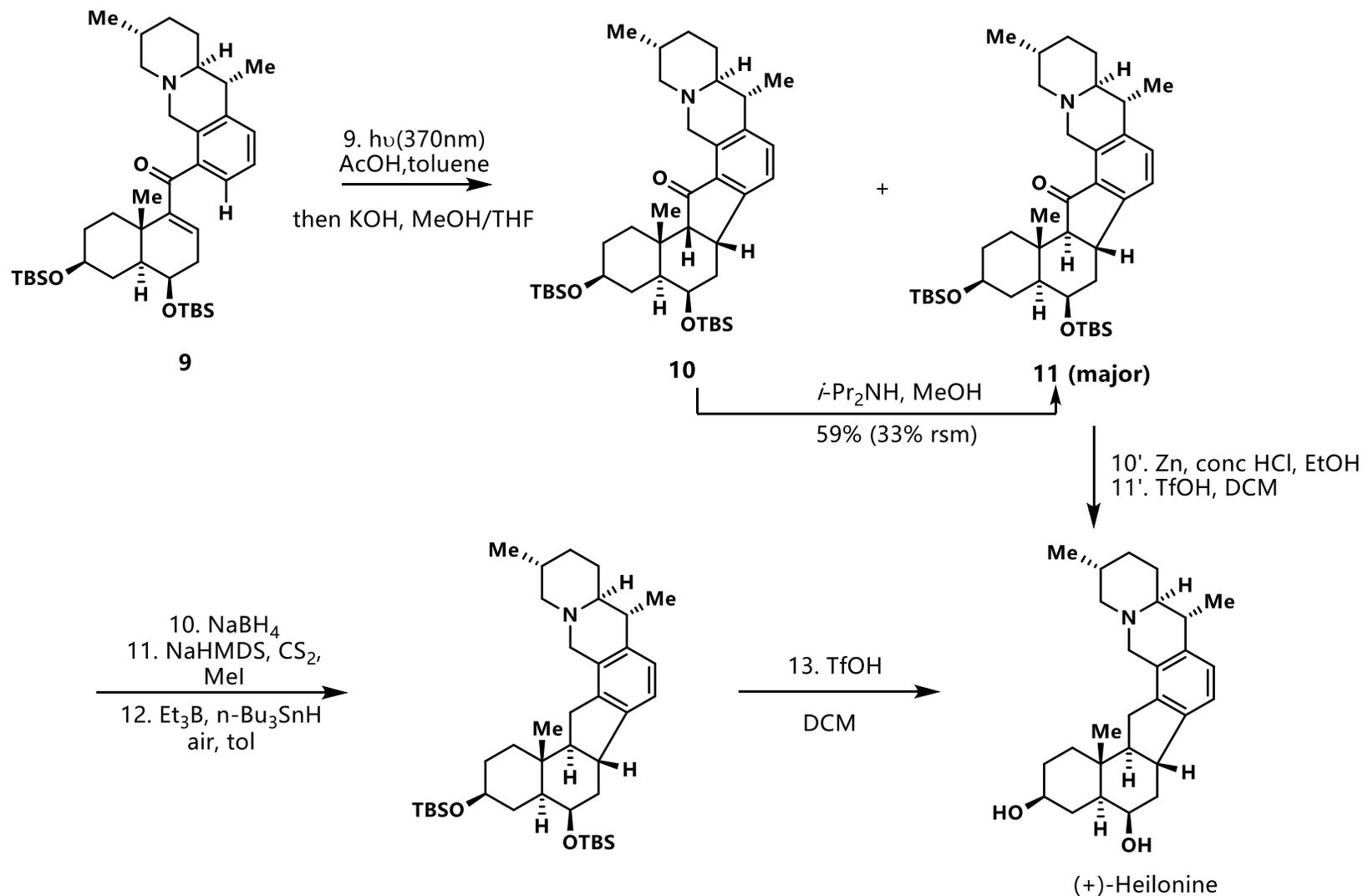
Convergent and Efficient Total Synthesis of (+)-Heilonine Enabled by C–H Functionalizations

Mingji Dai\* J. Am. Chem. Soc. 2024, 146, 1825–1831





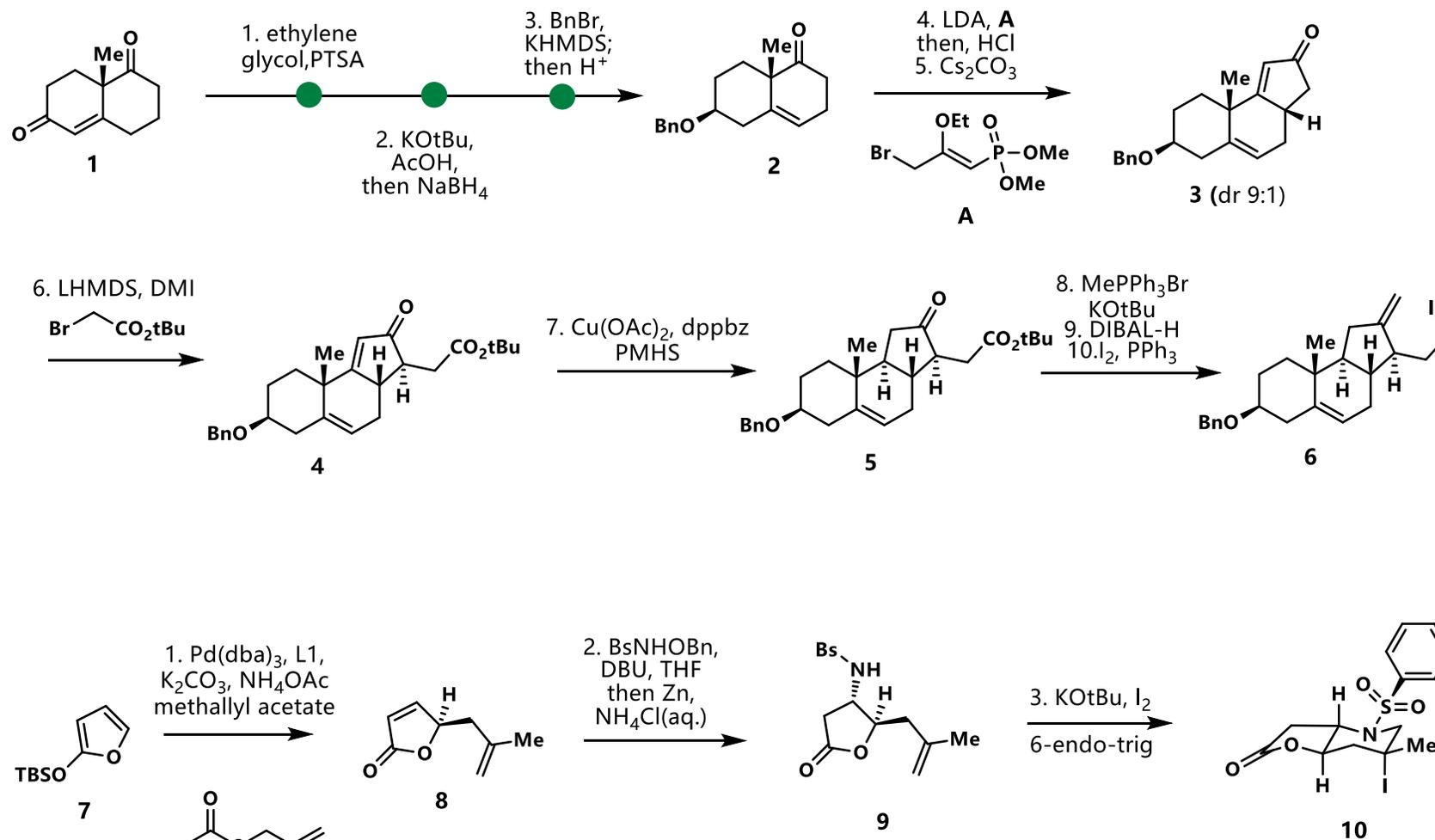


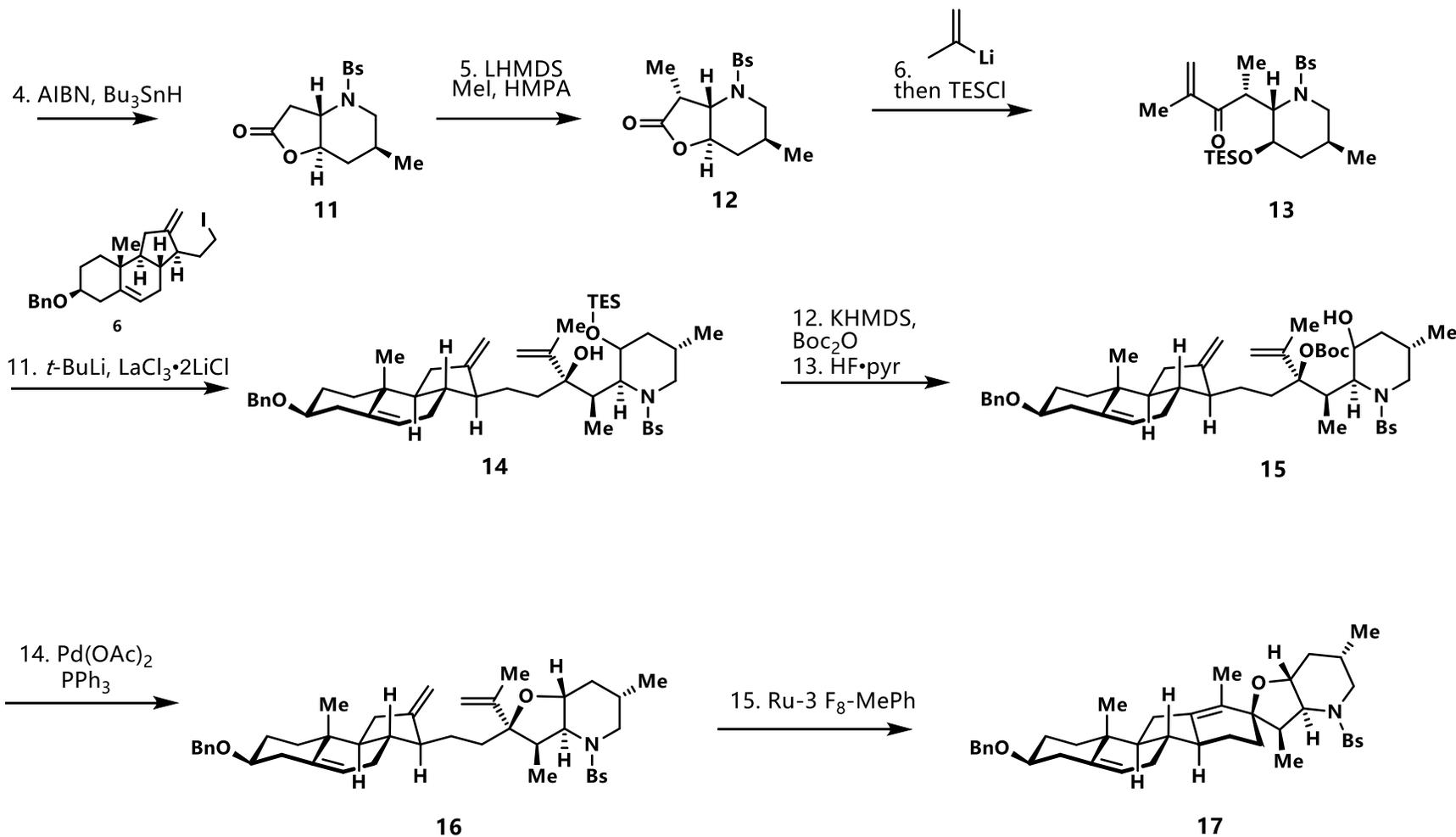


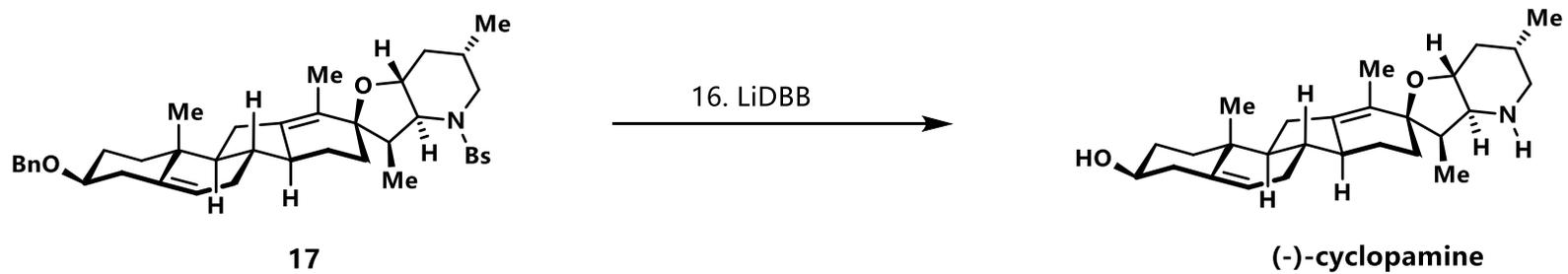


Convergent Total Synthesis of (-)-Cyclopamine

Phil S. Baran\* J. Am. Chem. Soc. 2023, 145, 40, 21760–21765



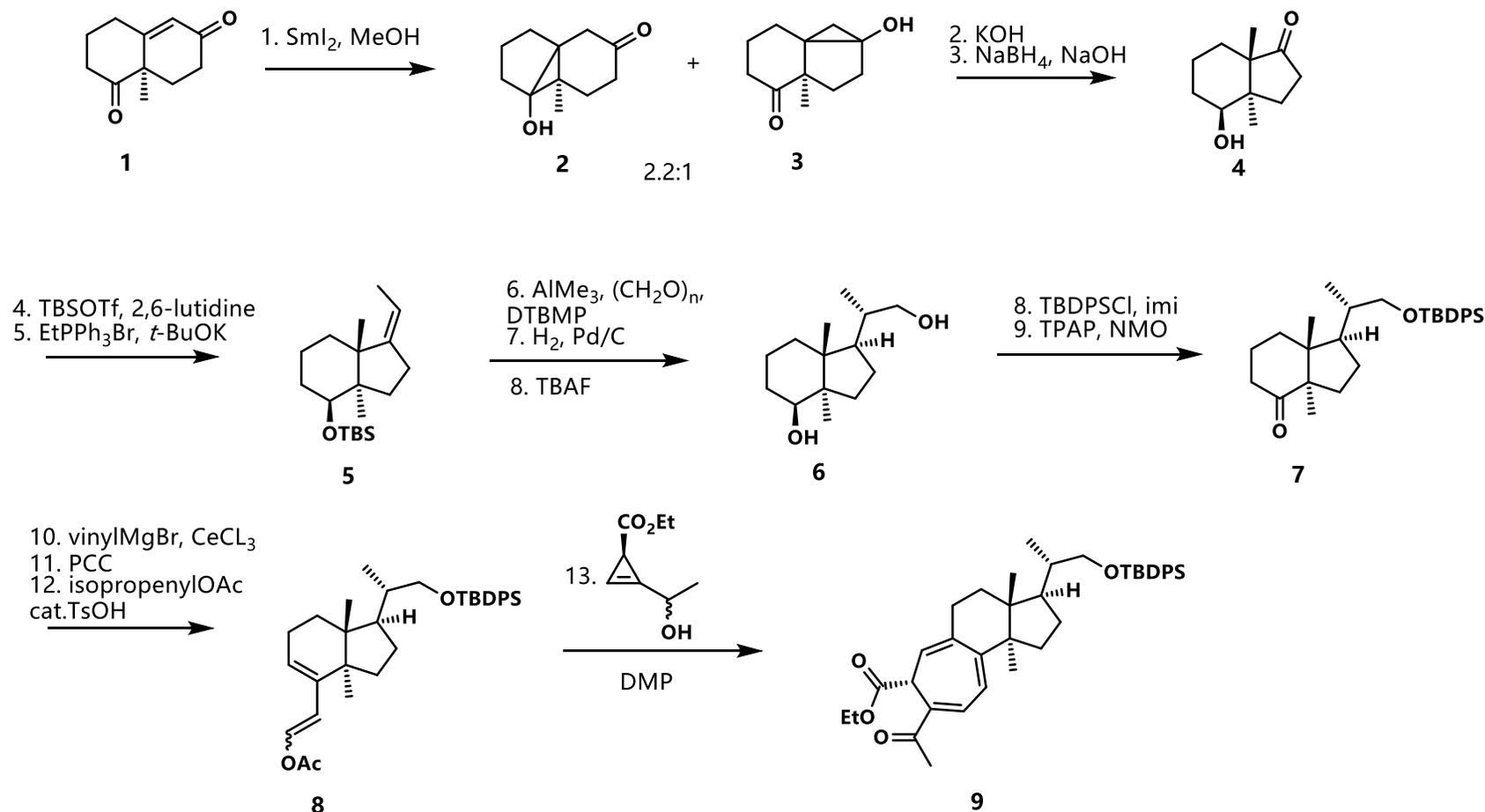


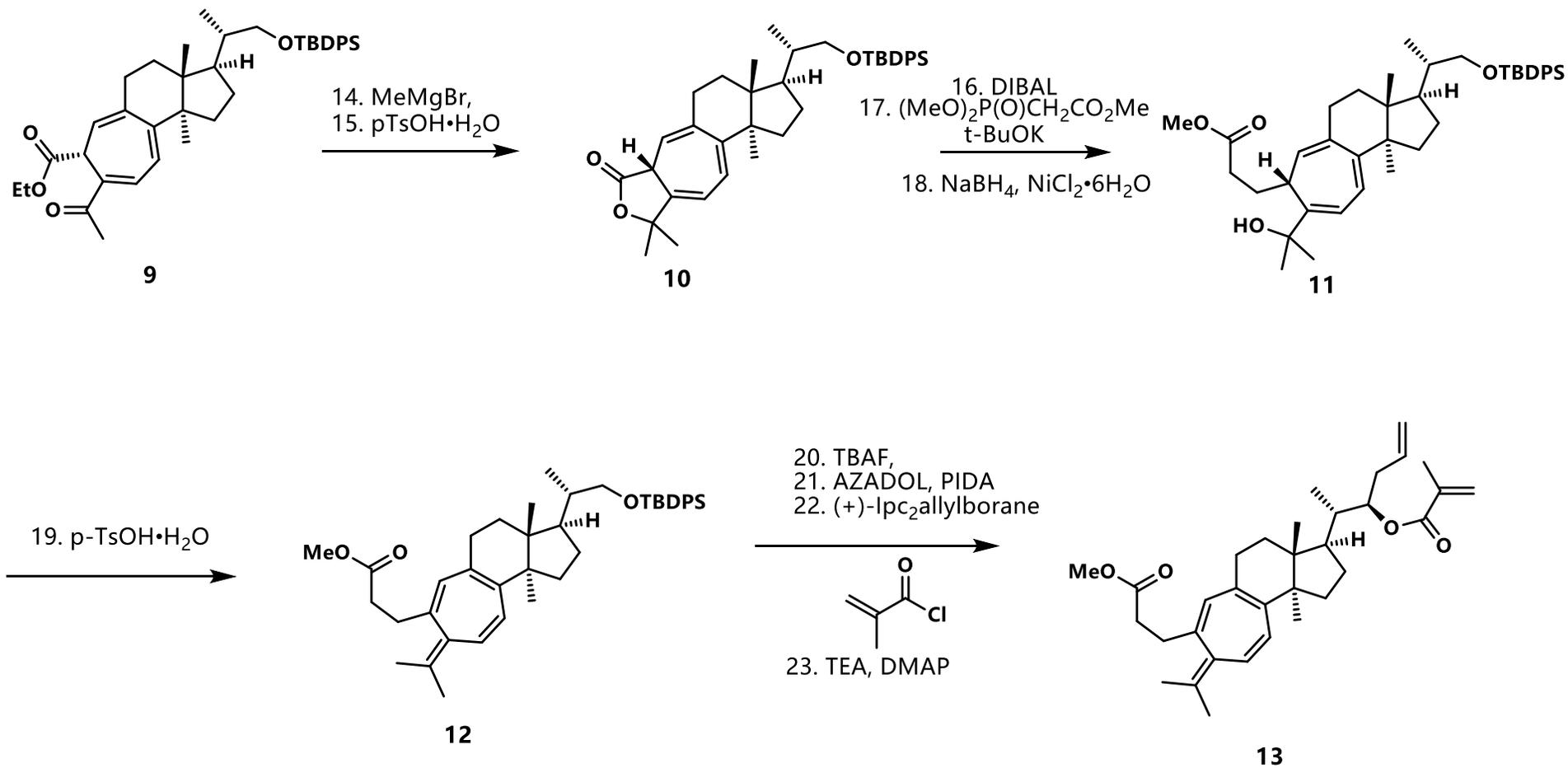


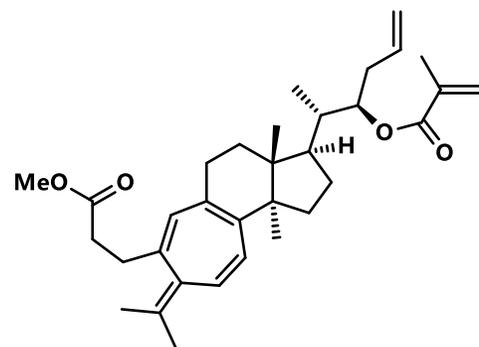


Total Synthesis and Structure Revision of (+)-Lancilactone C

Chihiro Tsukano\* J. Am. Chem. Soc. 2023, 145, 27, 14587–14591

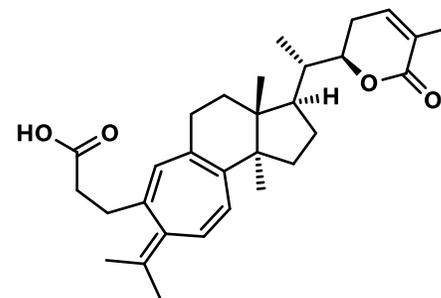




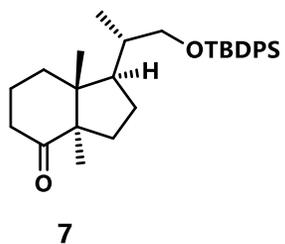


13

24. HG-II  
25. Me<sub>3</sub>SnOH, ClCH<sub>2</sub>CH<sub>2</sub>Cl



Lancilactone C

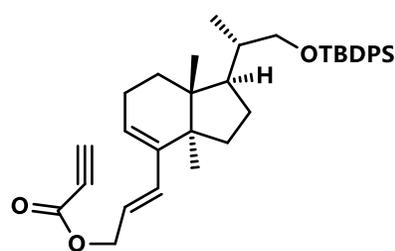


7

1. Comin's reagent  
KHMDS

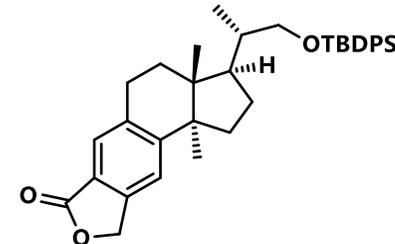
2. pinB-CH=CH-CH<sub>2</sub>-OH  
Pd(PPh<sub>3</sub>)<sub>4</sub>  
Cs<sub>2</sub>CO<sub>3</sub>

3.   
DMAP

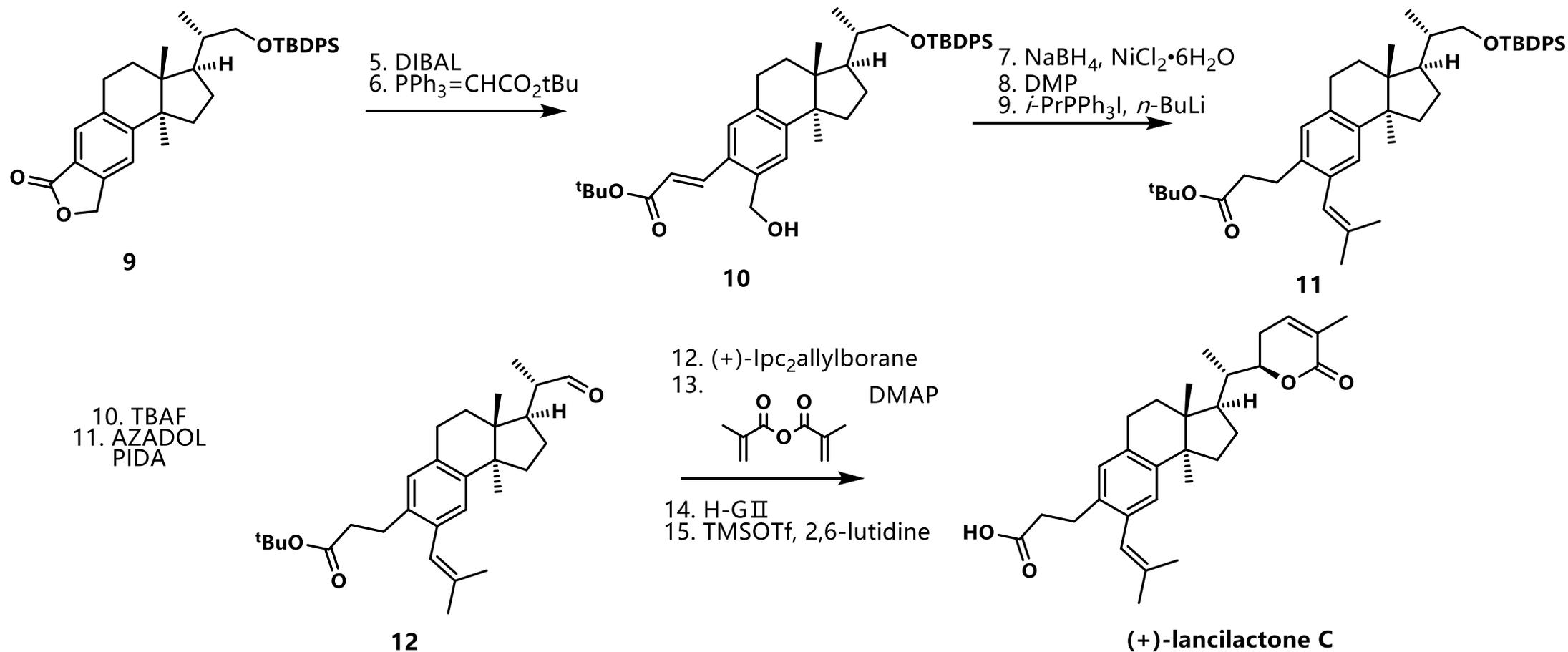


8

4. toluene  
reflux  
then DDQ



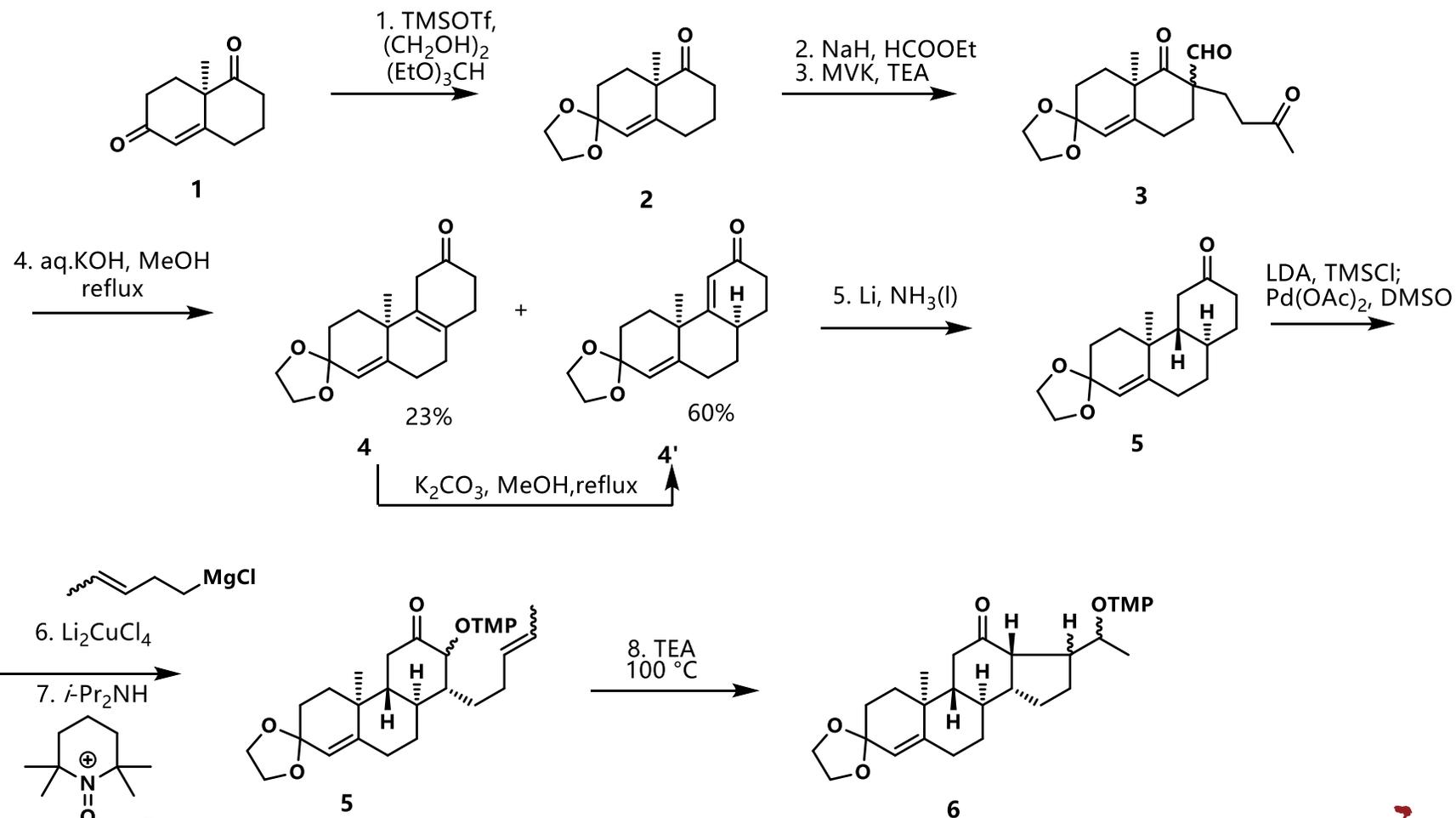
9

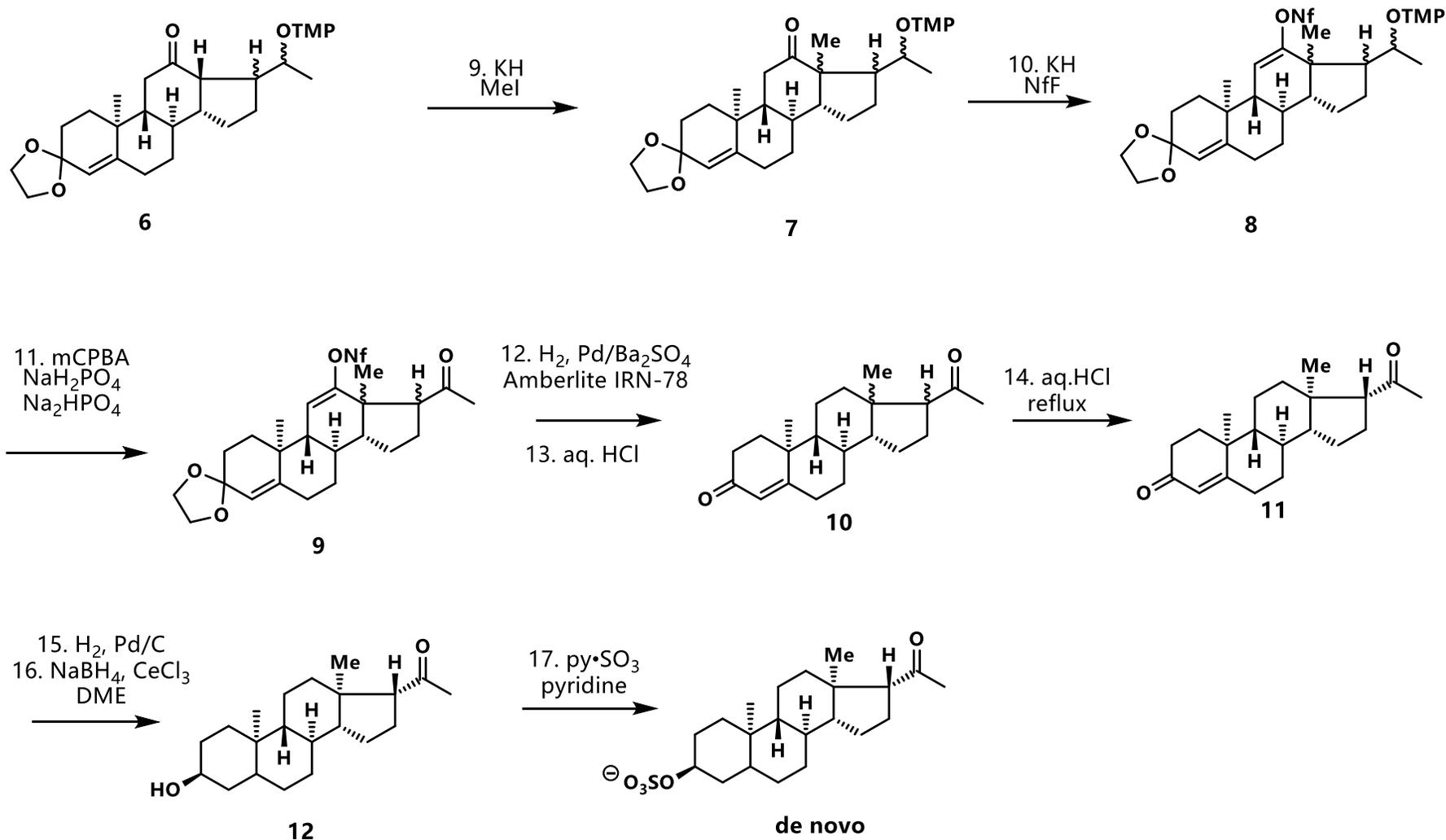




Total Synthesis of ent-Pregnanolone Sulfate and Its Biological Investigation at the NMDA Receptor

Ullrich Jahn\* Org. Lett. 2018, 20, 4, 946–949

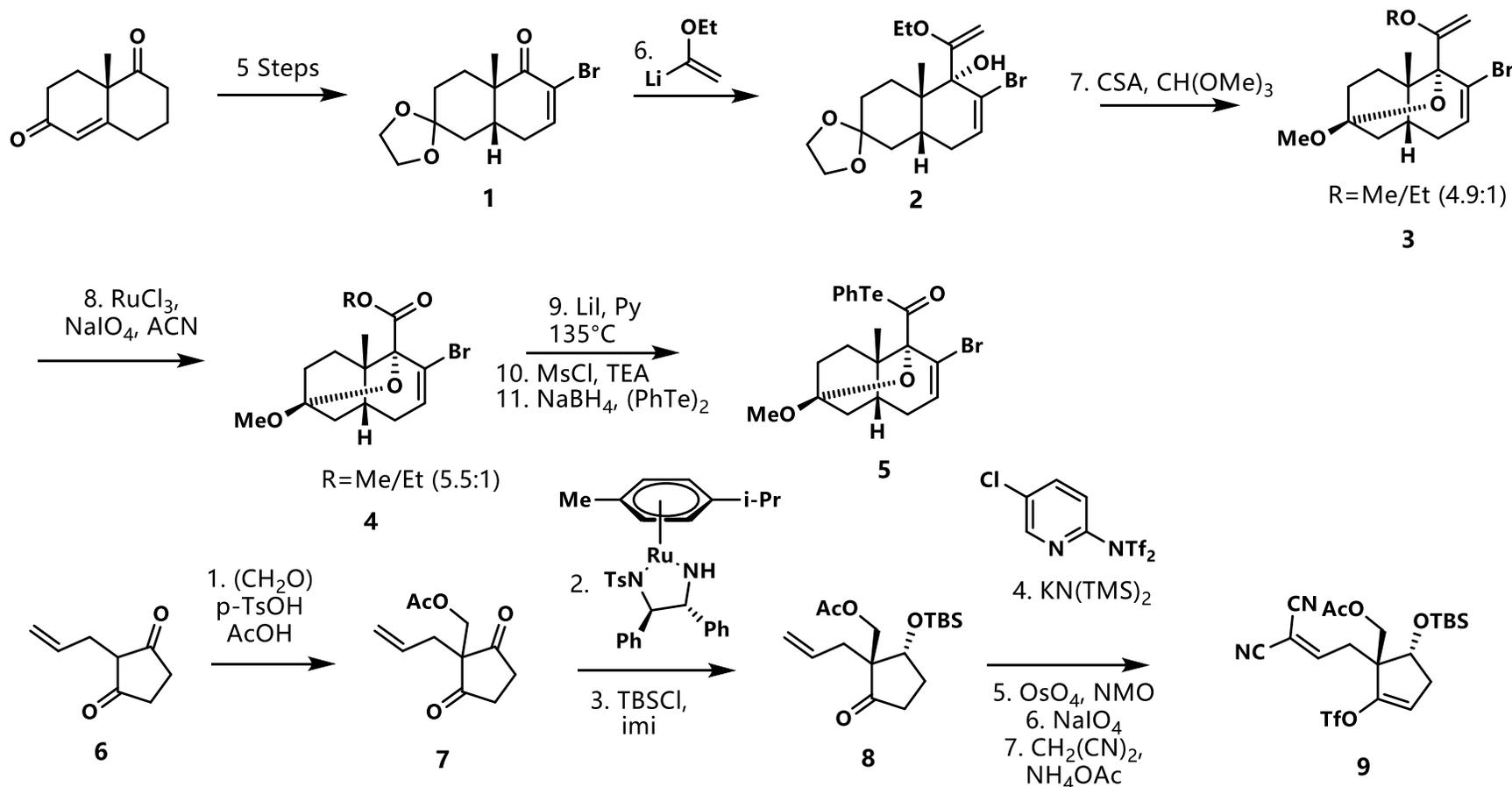


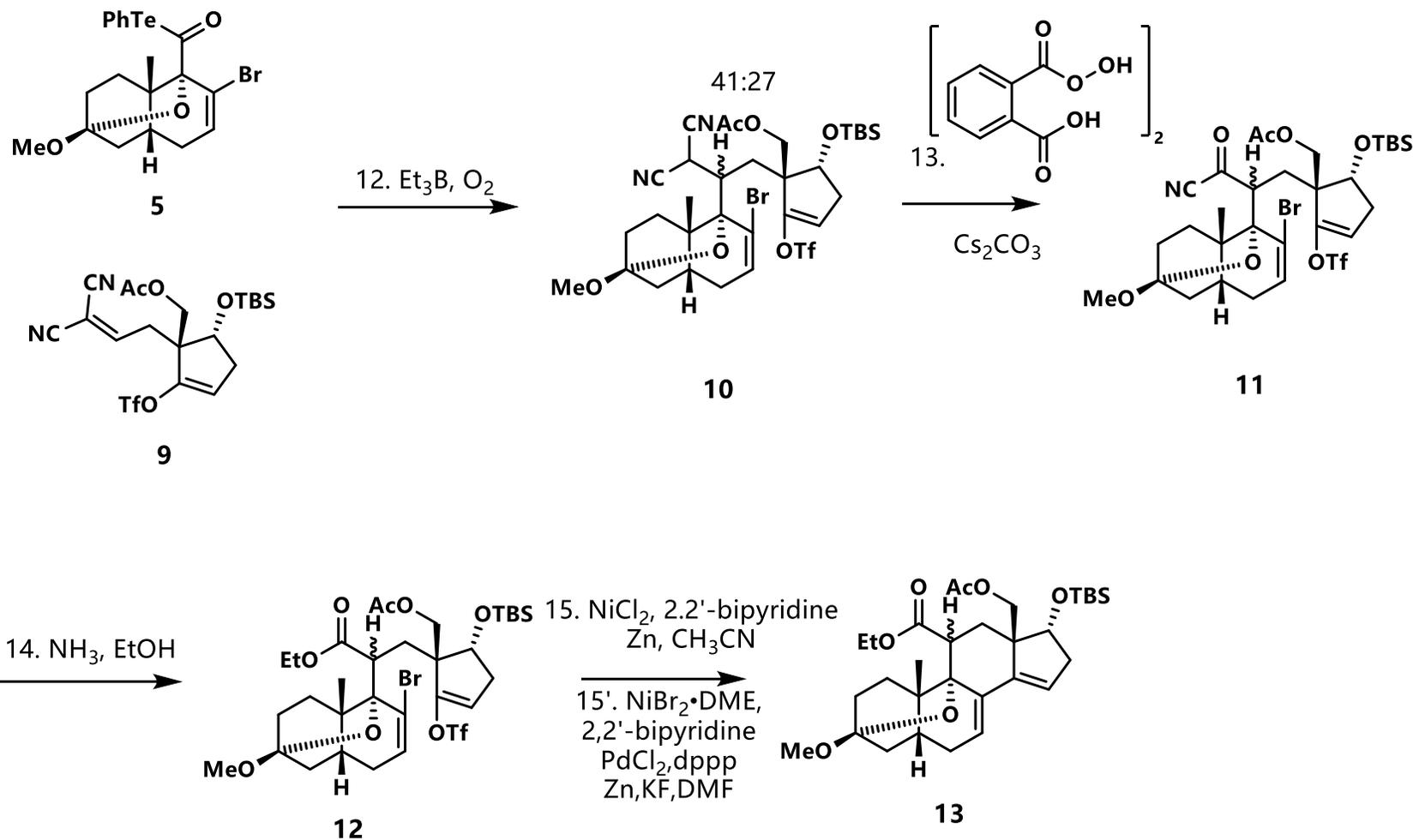




Synthesis of the Tetracyclic Structure of Batrachotoxin Enabled by Bridgehead Radical Coupling and Pd/Ni-Promoted Ullmann Reaction

Masayuki Inoue\* Org. Lett. 2018, 20, 1, 130–133

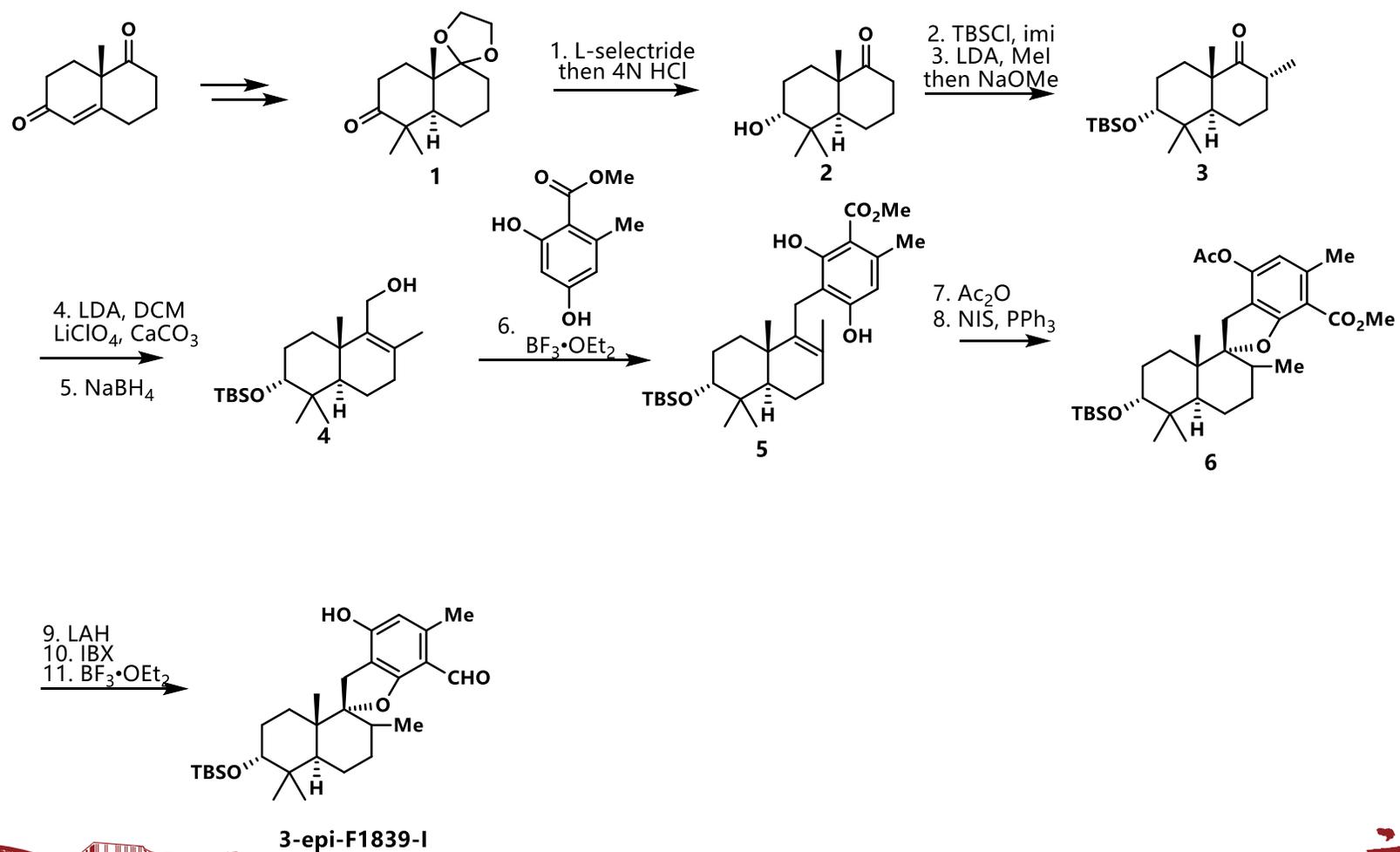






Enantiospecific total syntheses of meroterpenoids (-)-F1839-I and (-)-corallidictyals B and D

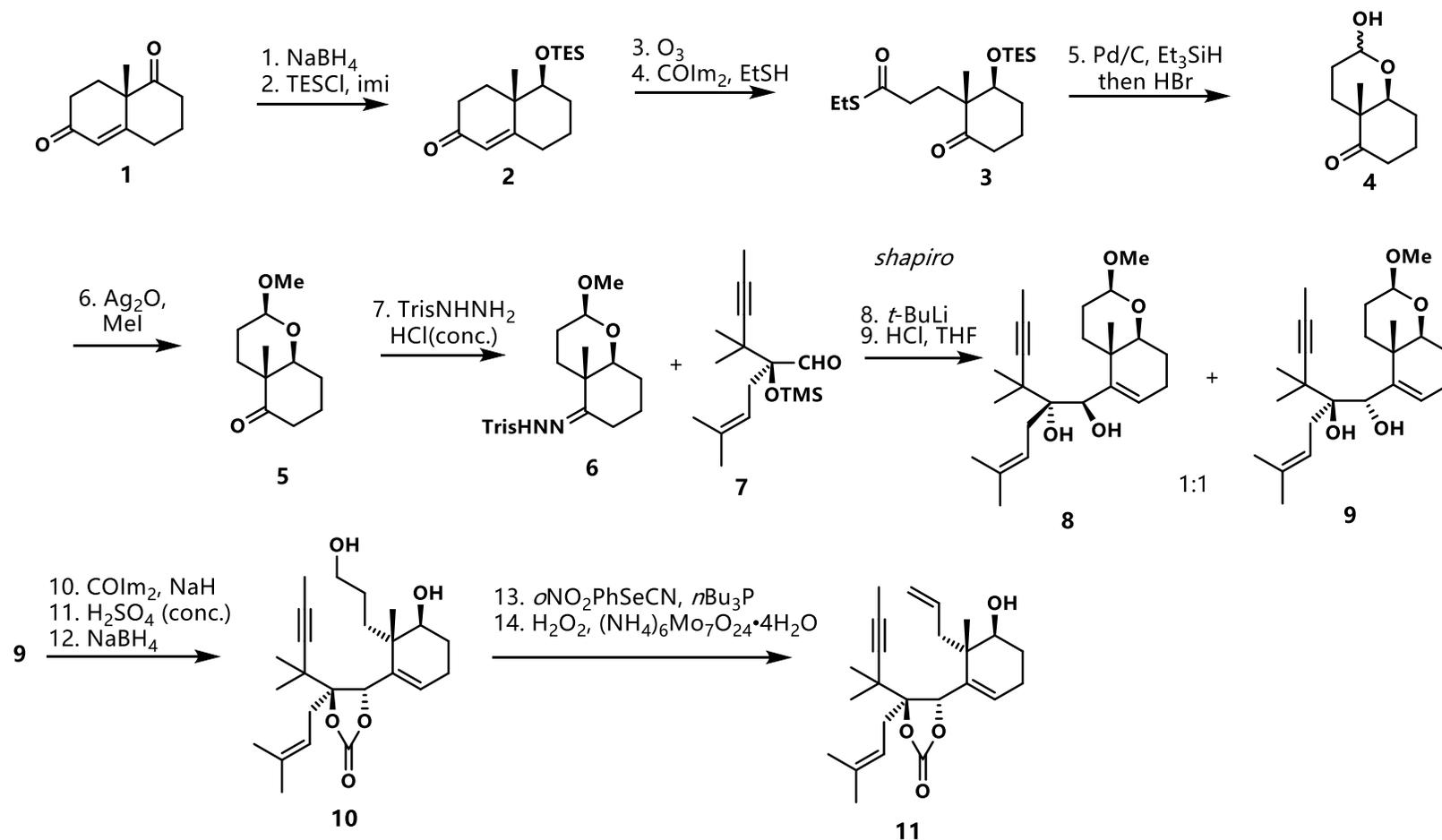
Dattatraya H. Dethe\* *Org. Biomol. Chem.*, 2017,15, 65-68

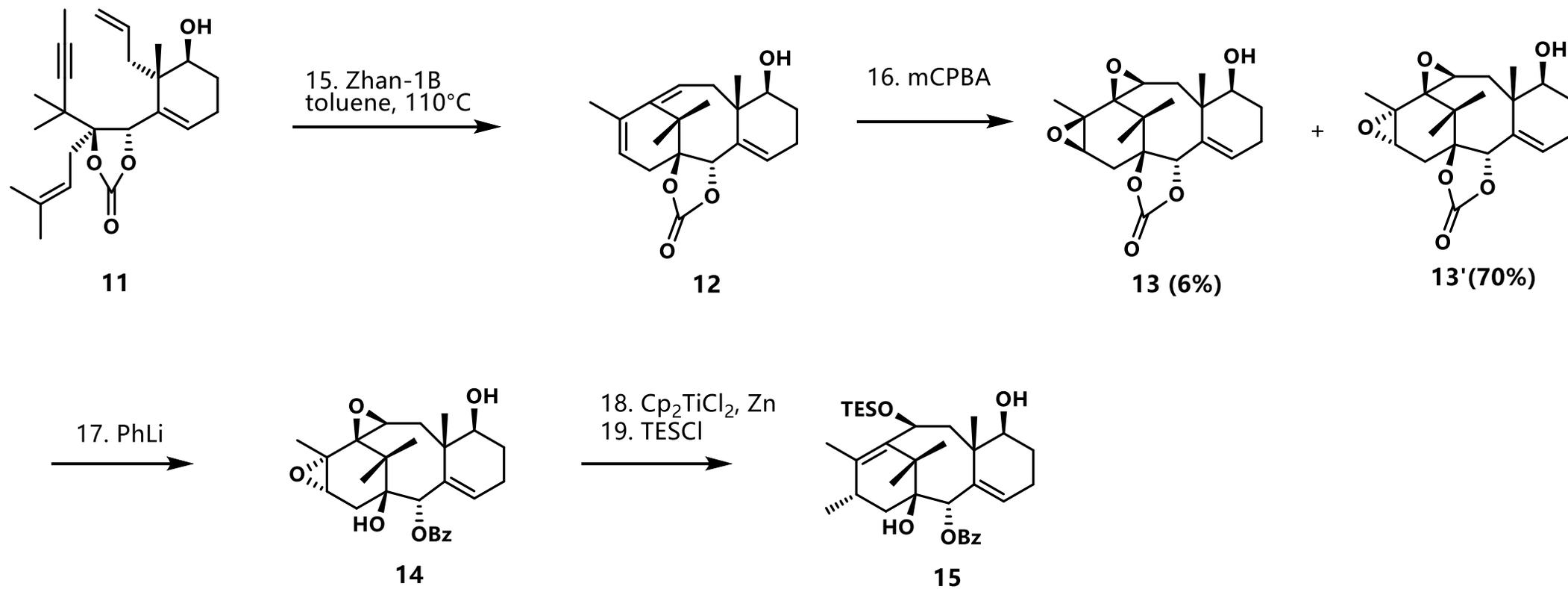




Study of Cascade Ring-Closing Metathesis Reactions en Route to an Advanced Intermediate of Taxol

Joëlle Prunet\* Org. Chem. 2016, 81, 24, 12318–12331

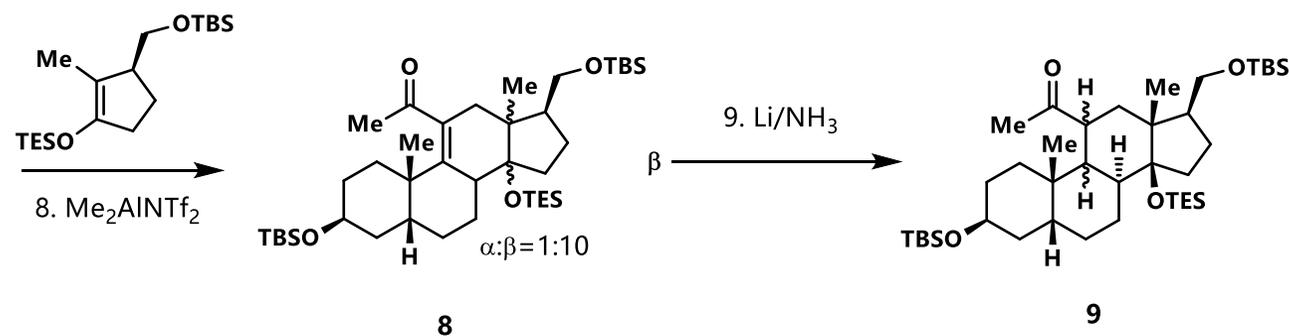
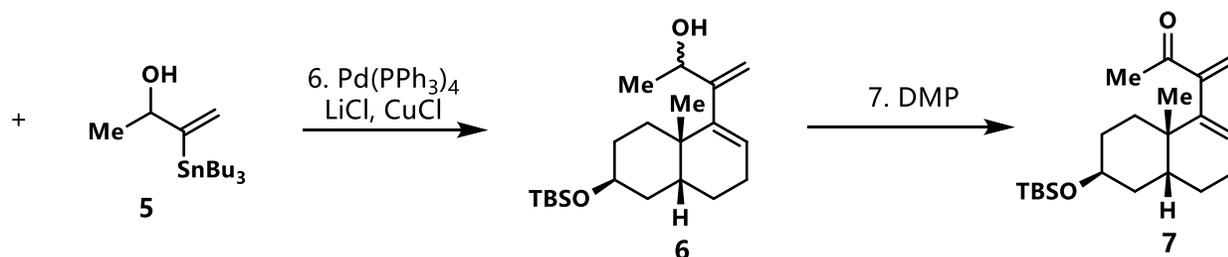
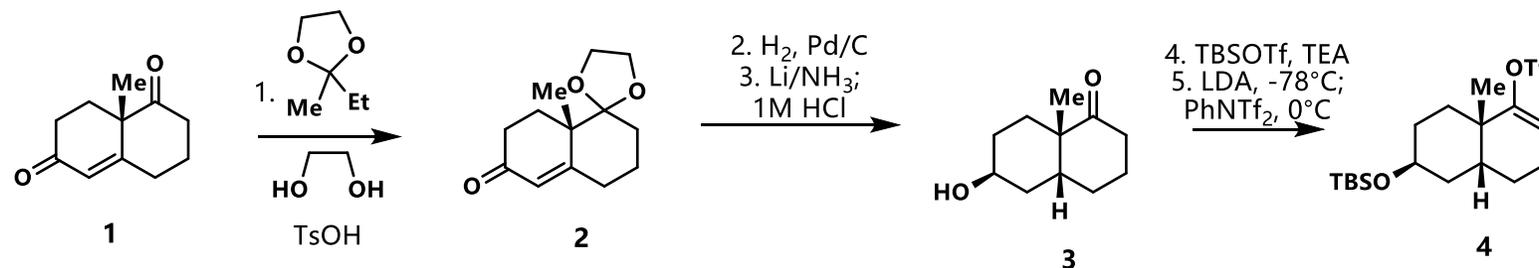


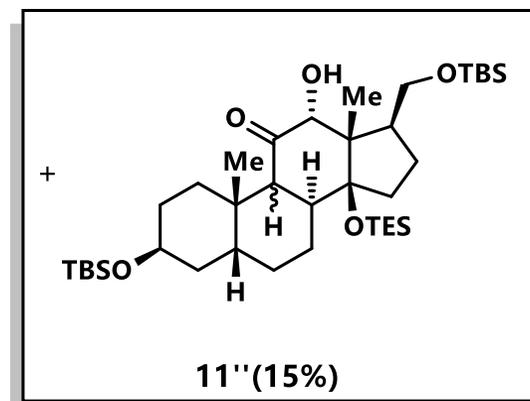
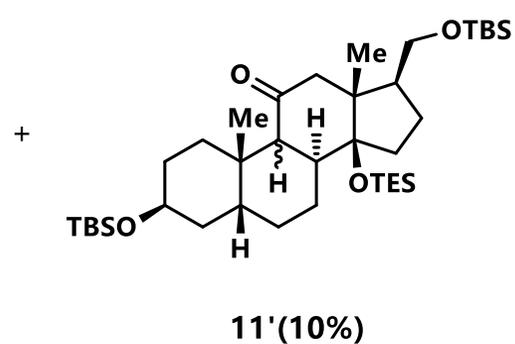
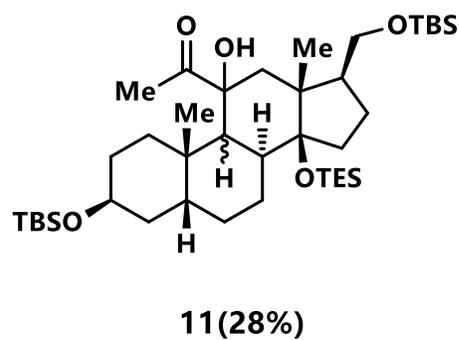
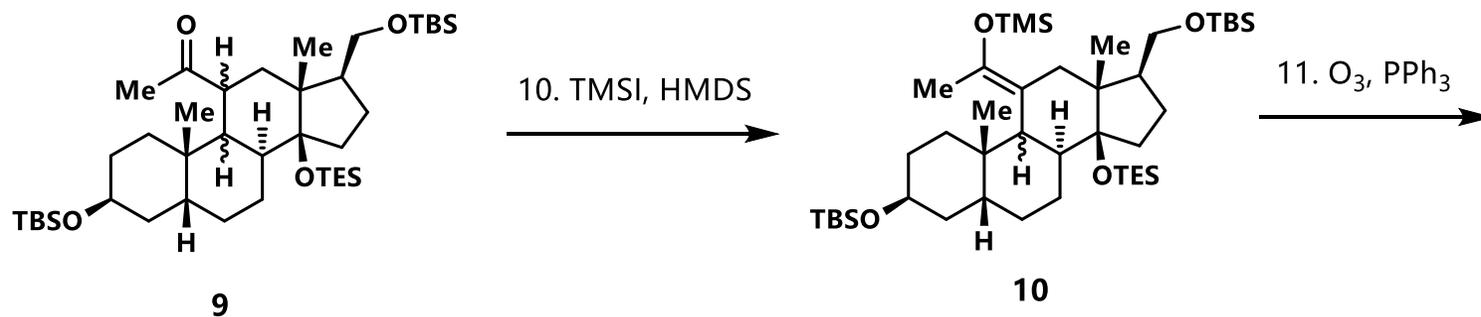




Studies Toward the Enantiospecific Total Synthesis of Rhodexin A

Michael E. Jung\**J. Org. Chem.* 2013, 78, 15, 7518–7526

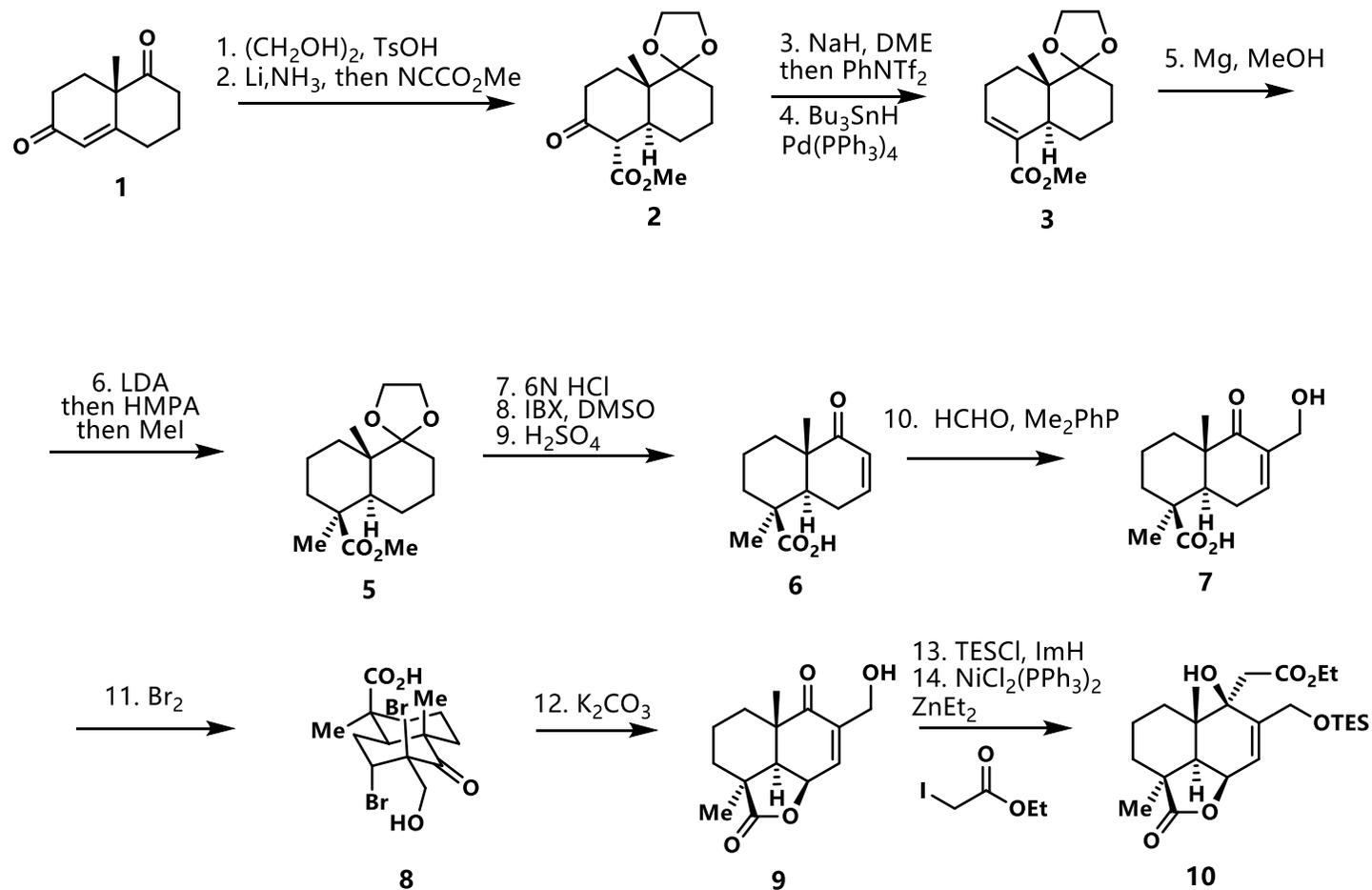


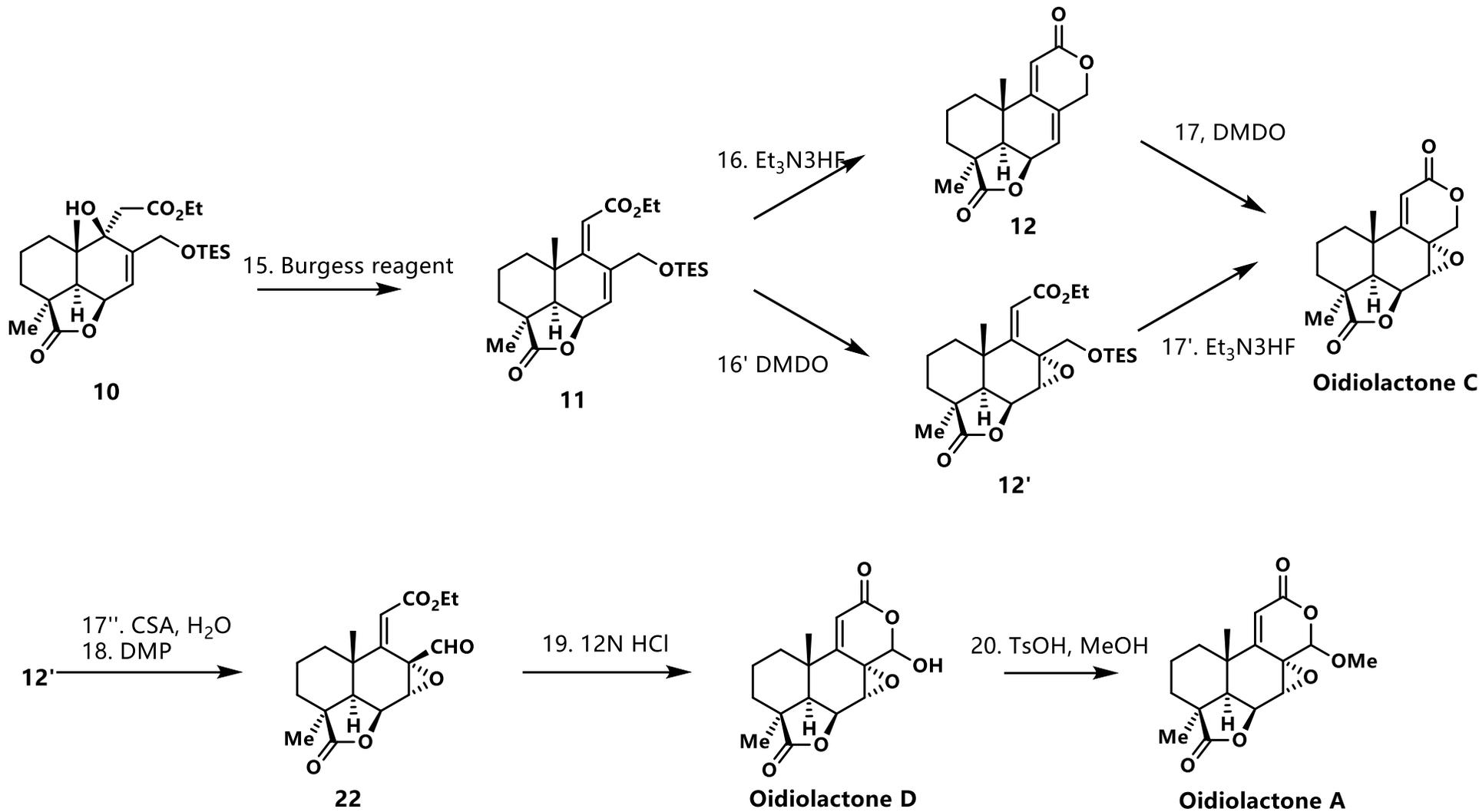


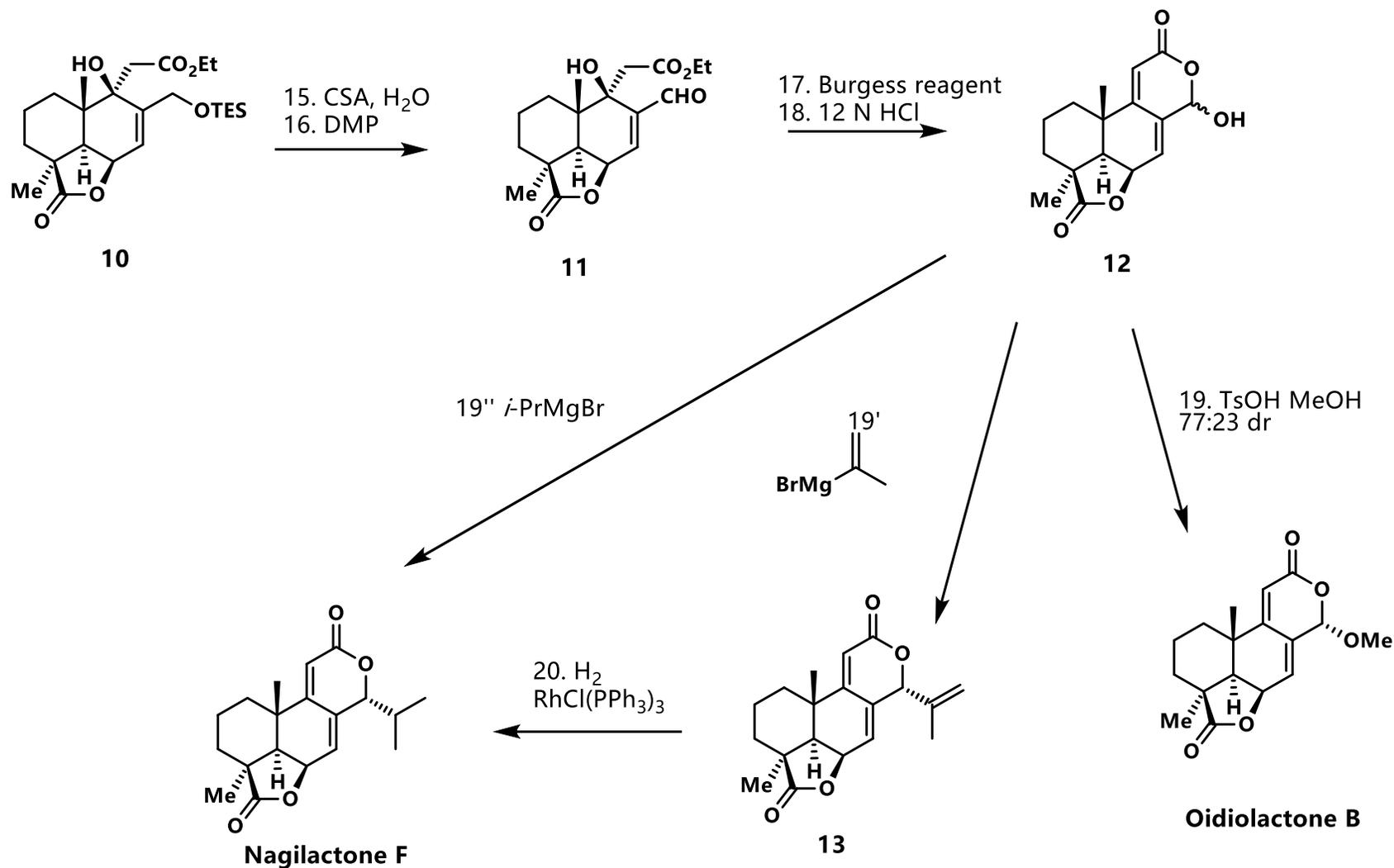


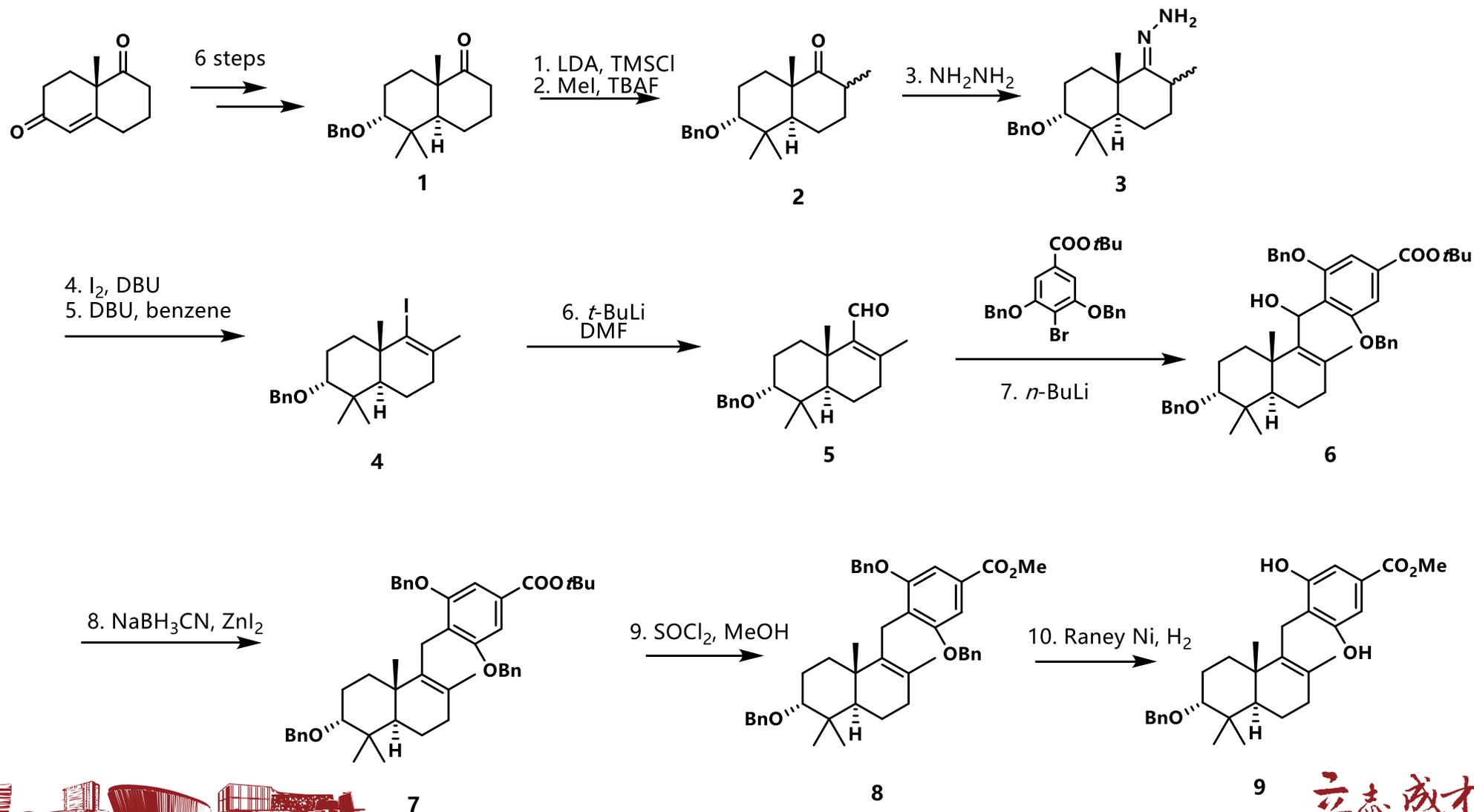
Total Synthesis of Odiiodendrolides and Related Norditerpene Dilactones from a Common Precursor: Metabolites CJ-14,445, LL-Z1271y, Odiolactones A, B, C, and D, and Nagilactone F

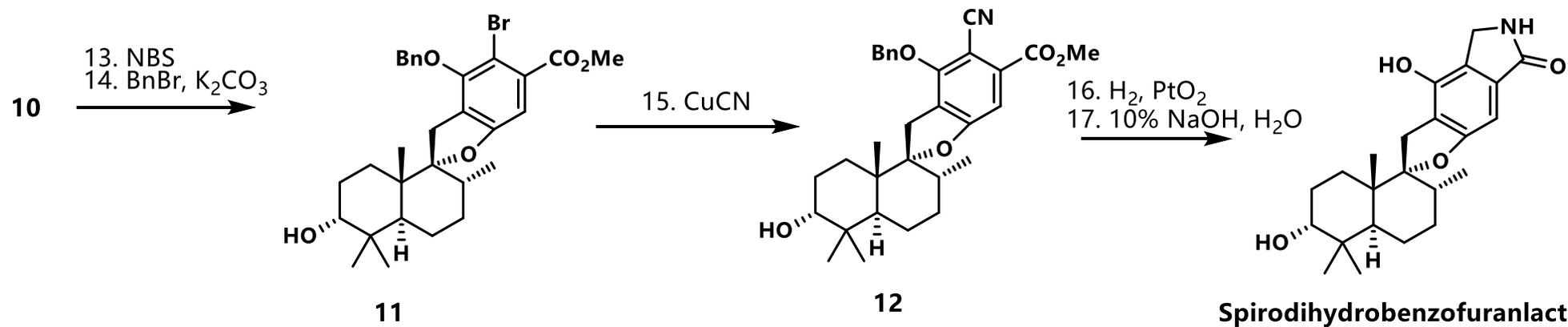
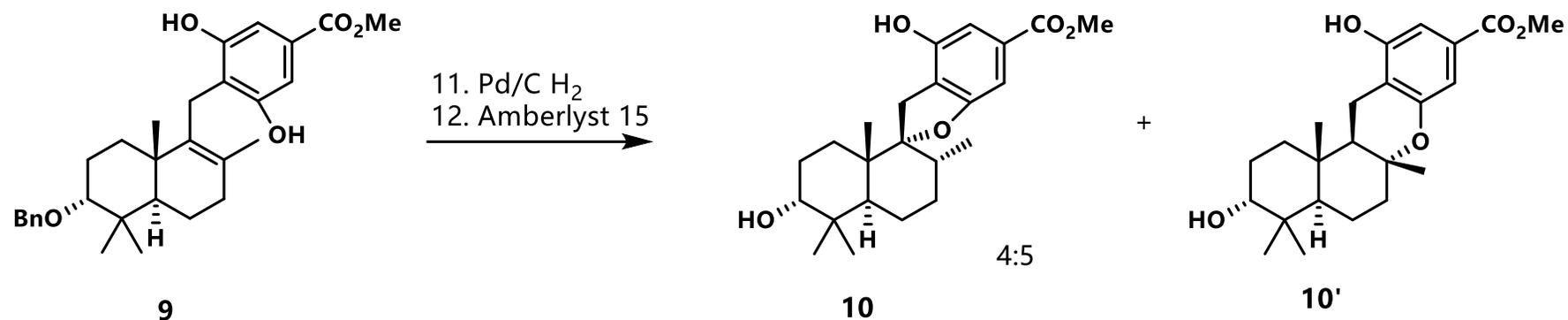
Stephen Hanessian\* Org. Lett. 2009, 11, 20, 4640–4643







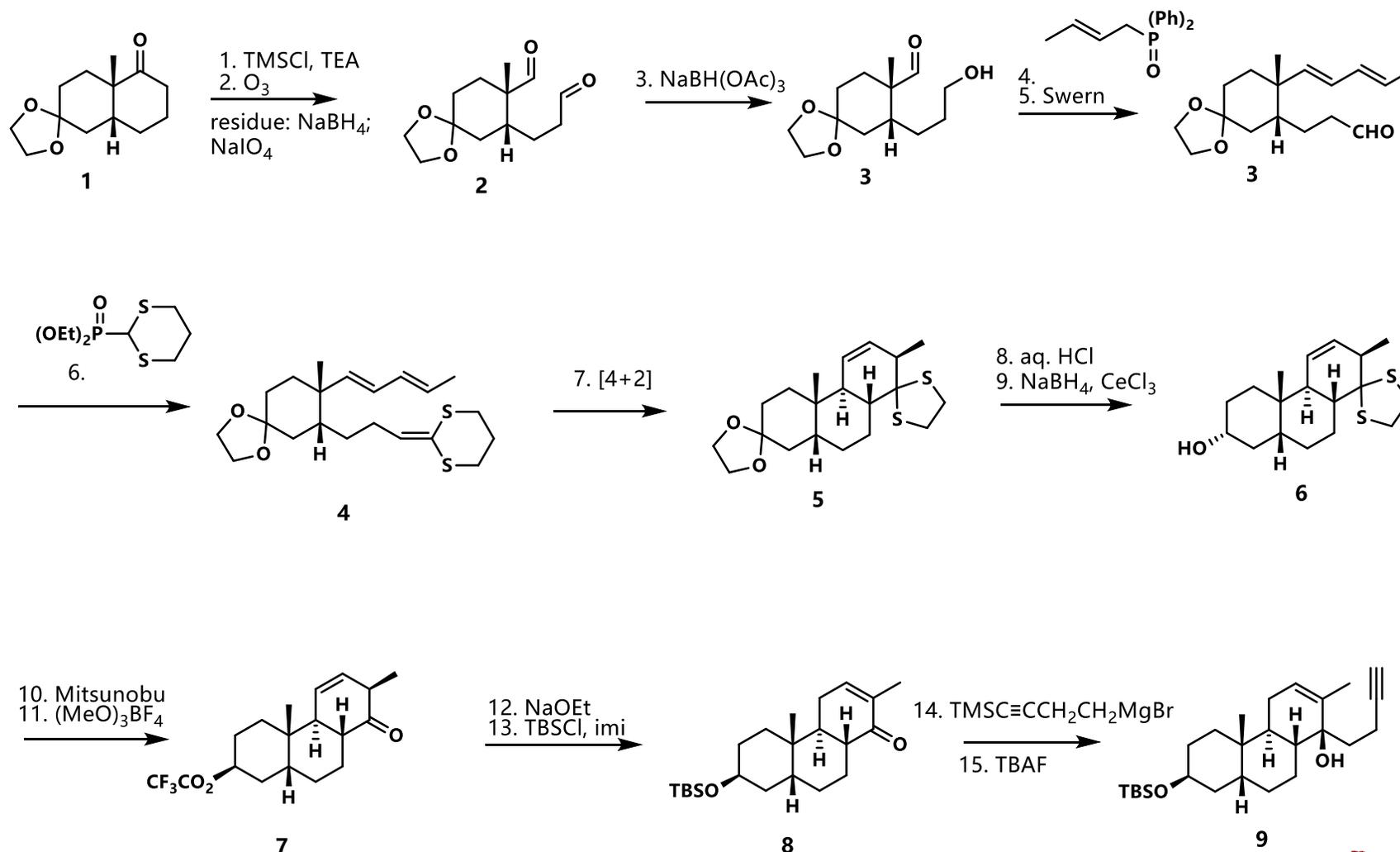


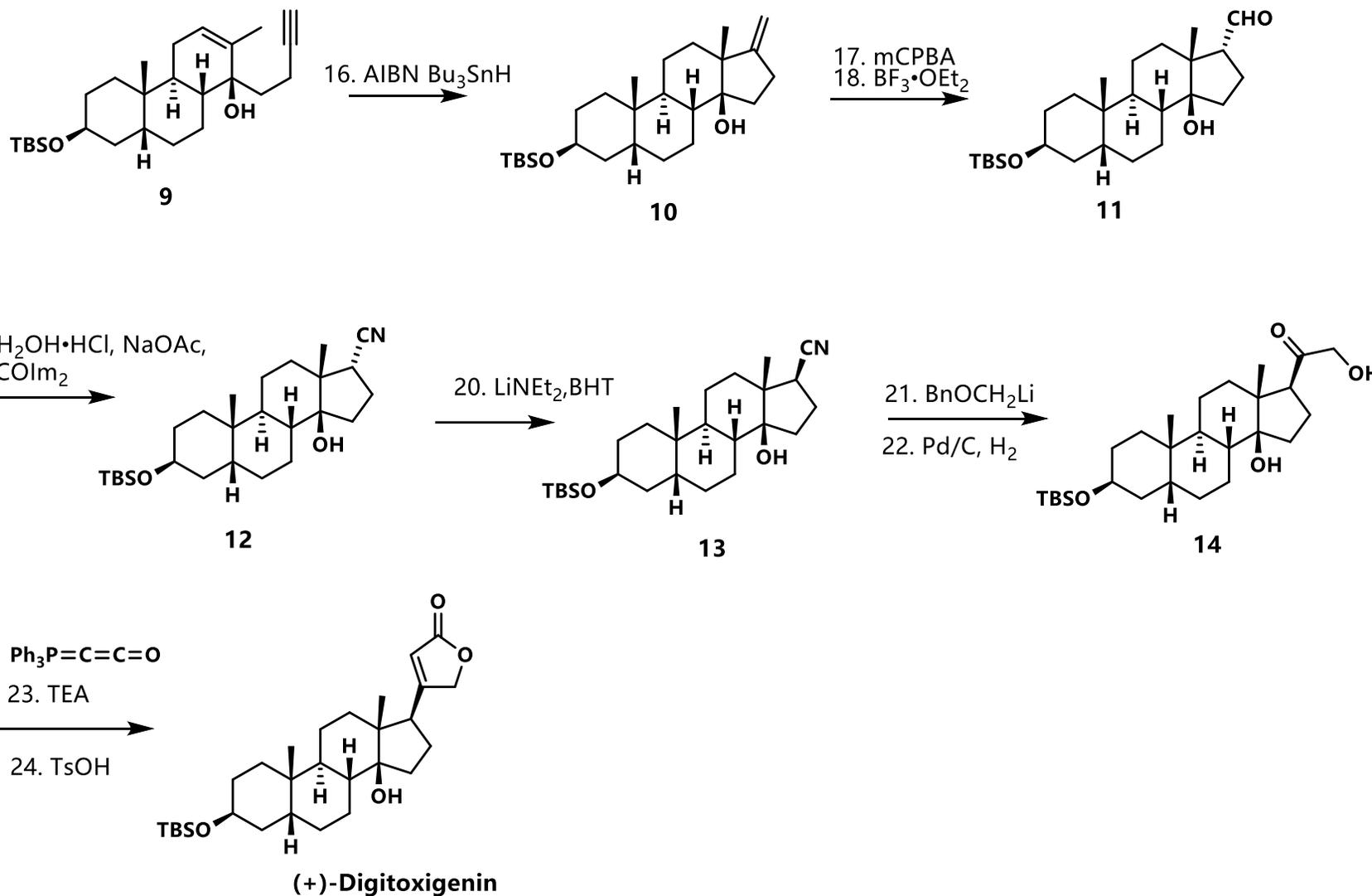




The Total Synthesis of a Natural Cardenolide: (+)-Digitoxigenin

Gilbert Stork\* J. Am. Chem. Soc. 1996, 118, 43, 10660–10661

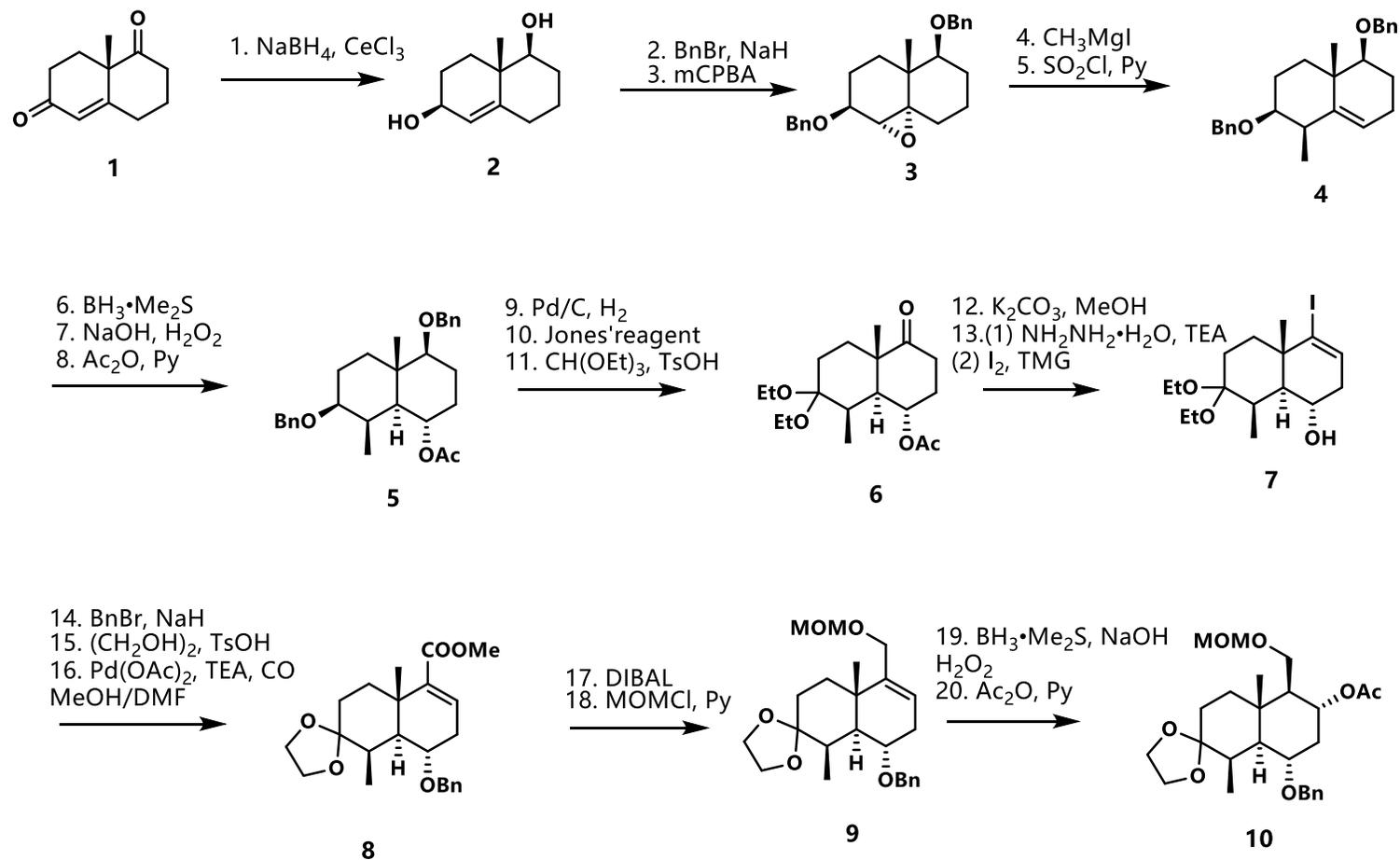


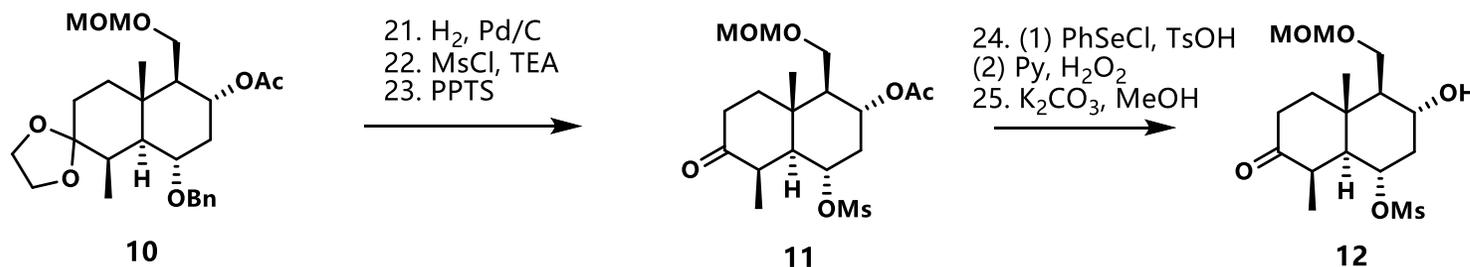




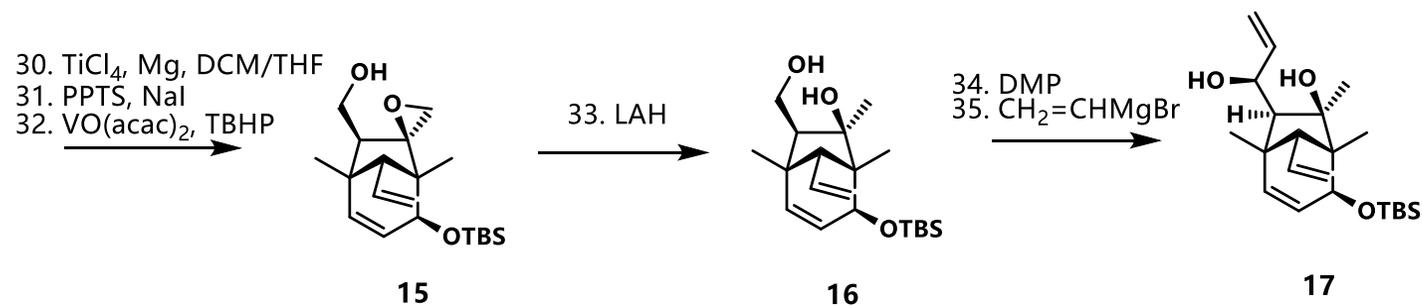
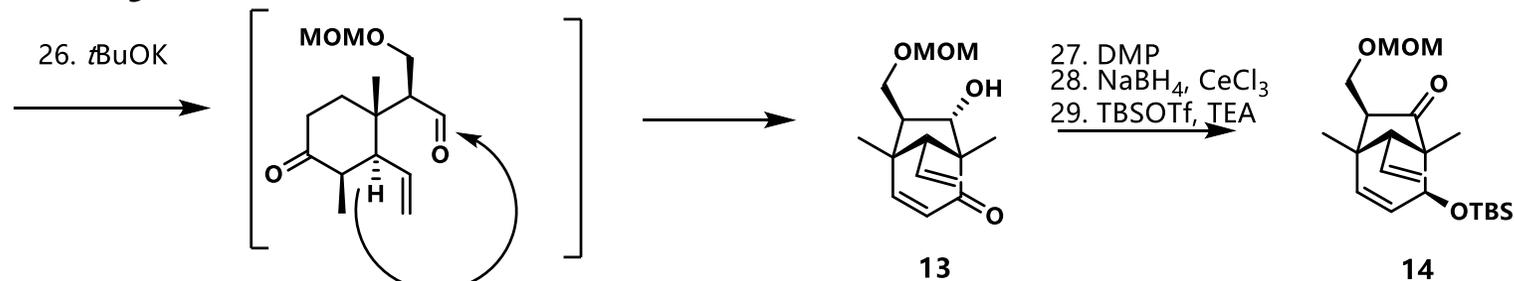
Total synthesis of (±)-pallambins C and D

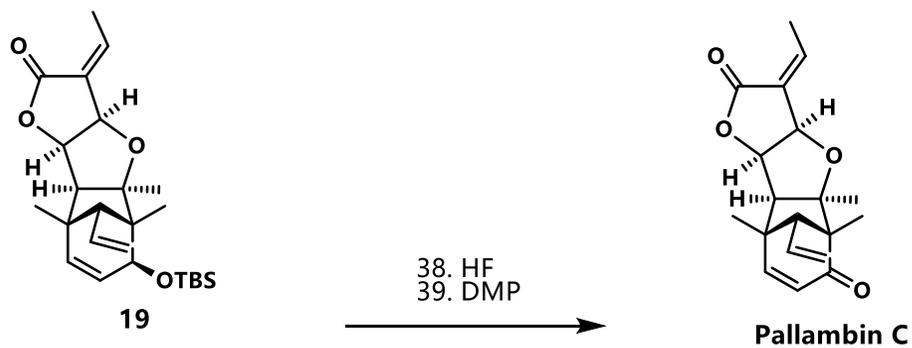
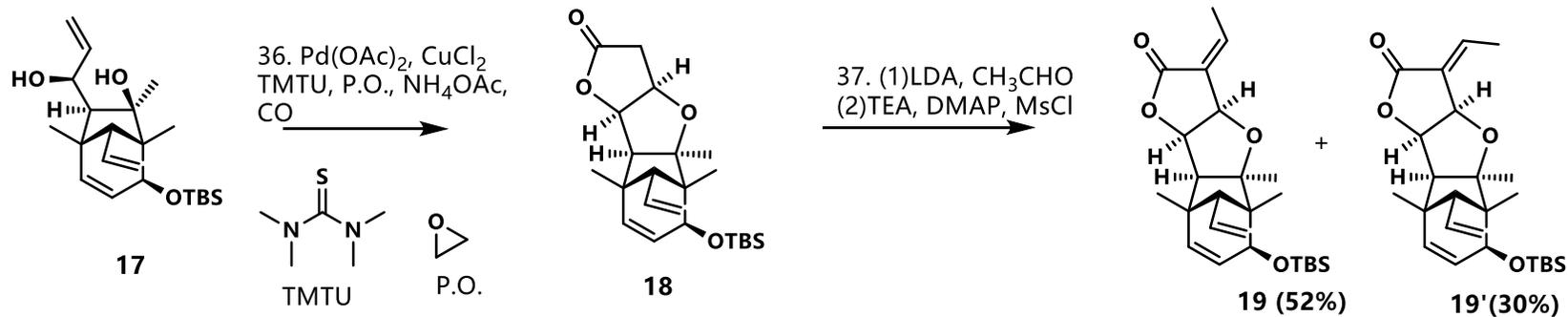
Henry N. C. Wong\* Chem. Commun., 2012,48, 8517-8519



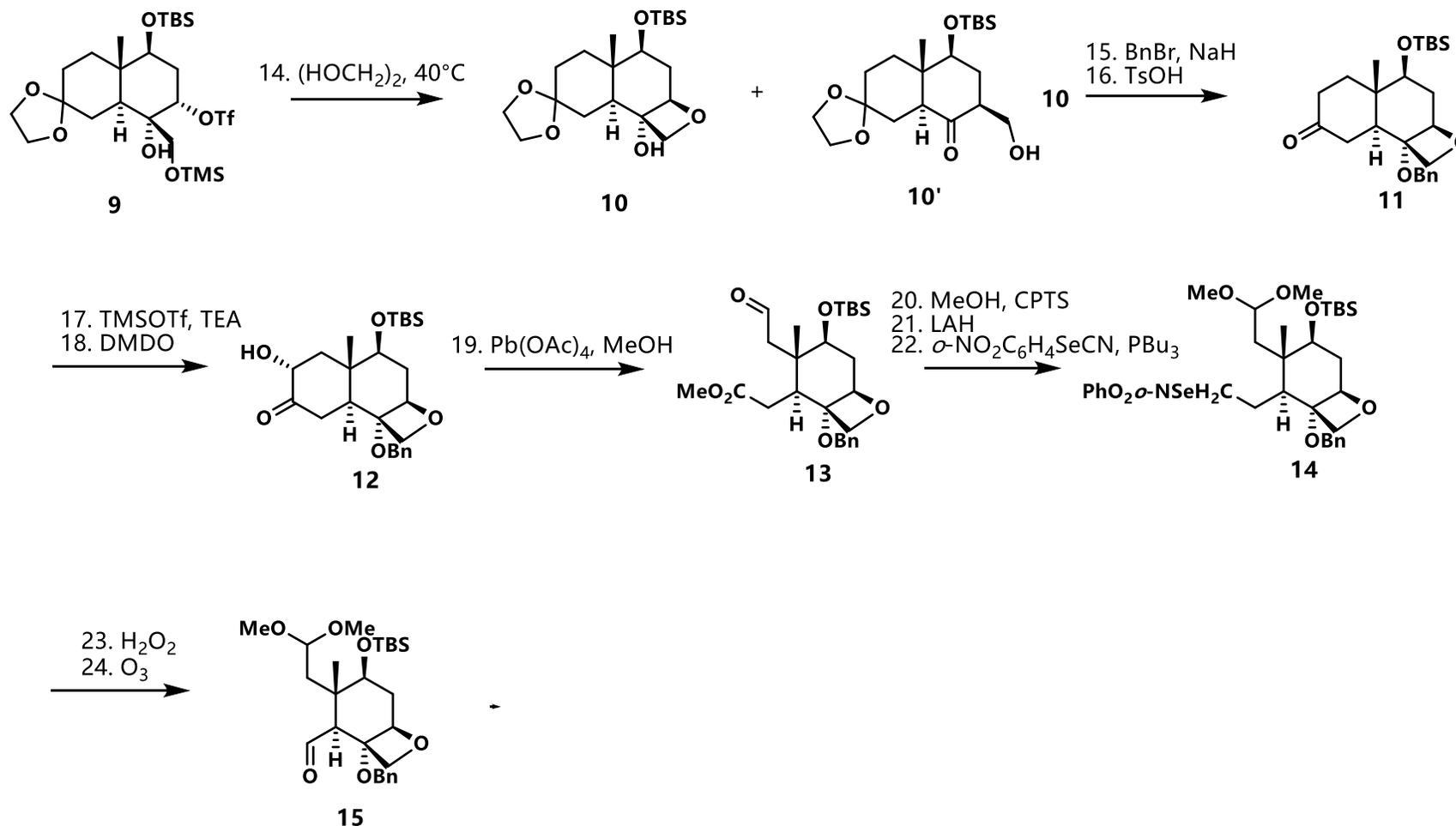


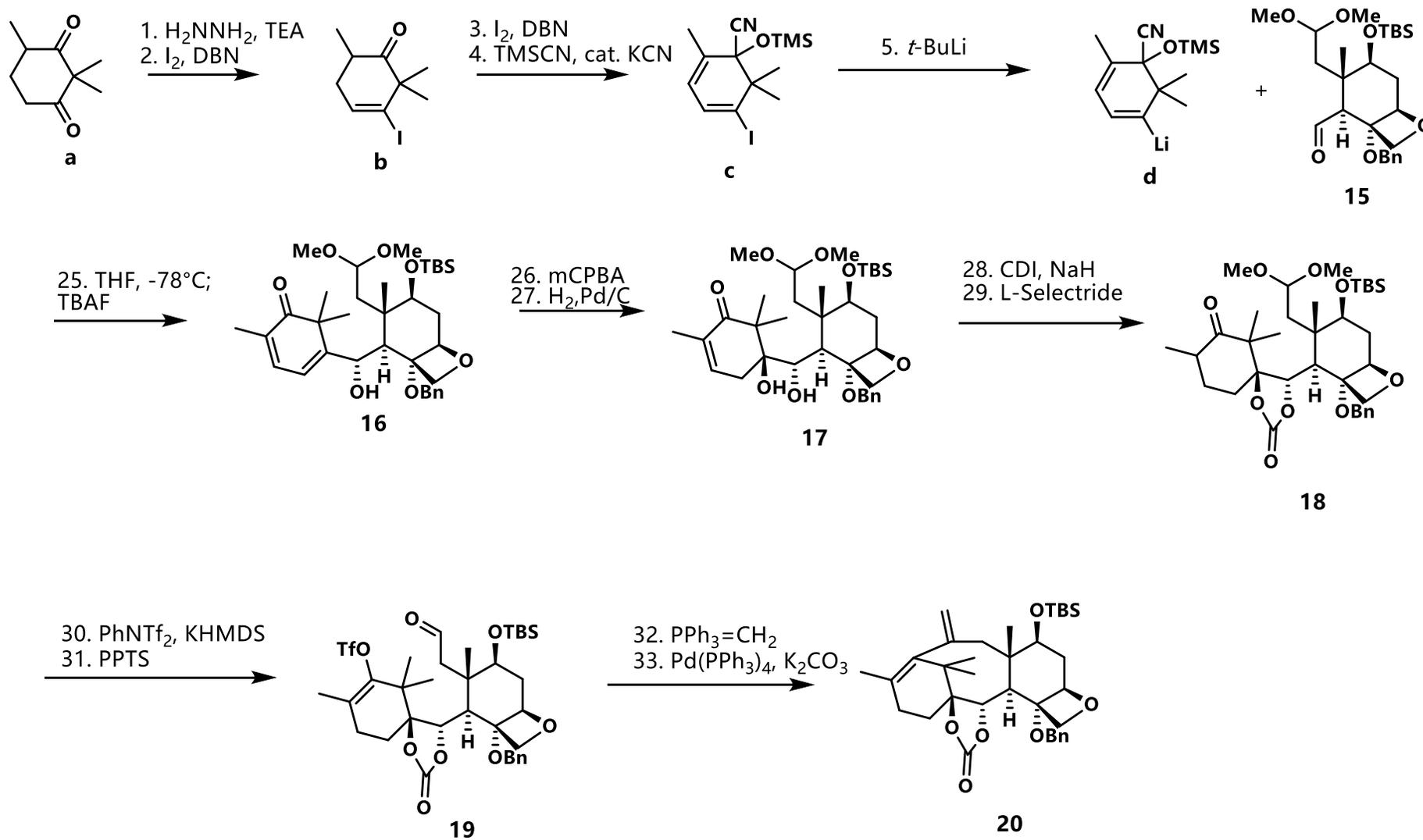
*Grob fragmentation*





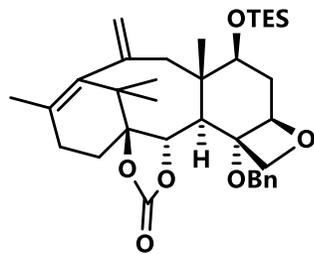






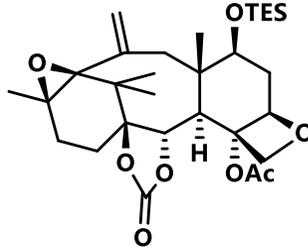


34. TBAF  
35. TESOTf, TEA



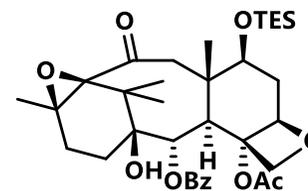
21

36. mCPBA  
37. H<sub>2</sub>, Pd/C  
38. Ac<sub>2</sub>O, DMAP



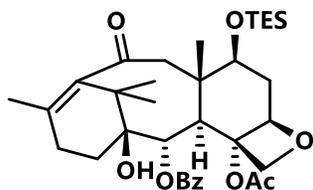
22

39. PhLi,  
40. OsO<sub>4</sub>, Py  
41. Pb(OAc)<sub>4</sub>

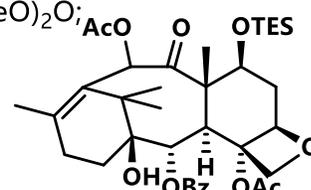
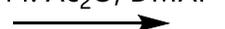


23

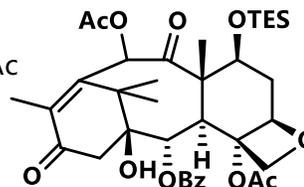
42. Sml<sub>2</sub>, Ac<sub>2</sub>O



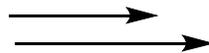
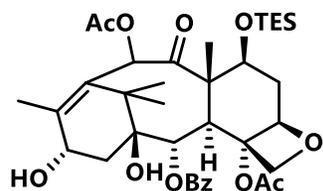
43. KOtBu, (PhSeO)<sub>2</sub>O;  
KOtBu  
44. Ac<sub>2</sub>O, DMAP



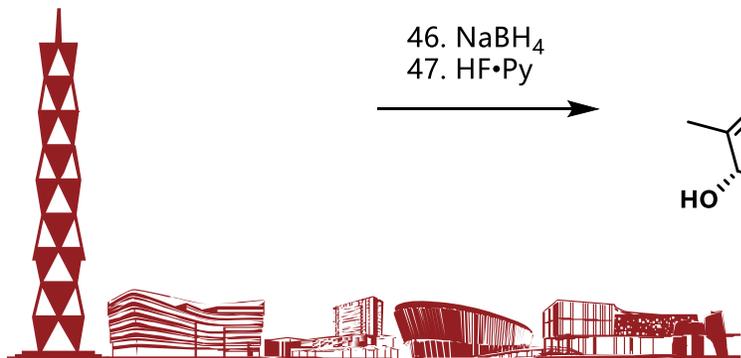
45. PCC, NaOAc



46. NaBH<sub>4</sub>  
47. HF·Py



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谢谢



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