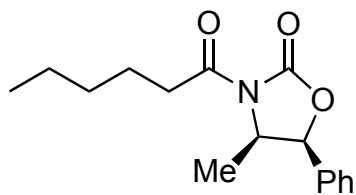
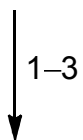


## Total Synthesis of Cylindrocyclophane F

Amos B. Smith III, Sergey A. Kozmin, Daniel V. Paone, *JACS* **1999**, 121, 7423–7424.

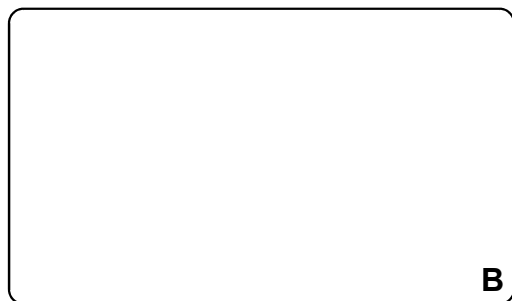


**A**

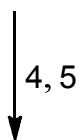


- 1) NaHMDS, allyl bromide, THF,  $-78\text{ }^{\circ}\text{C}$
- 2) LiOH,  $\text{H}_2\text{O}_2$ , THF- $\text{H}_2\text{O}$
- 3) EtI,  $\text{K}_2\text{CO}_3$ , acetone- $\text{H}_2\text{O}$

Name of reaction in step 1

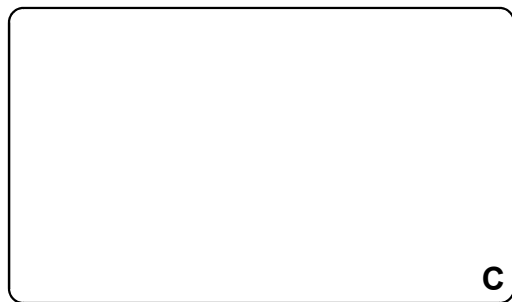


**B**



- 4)  $\text{CH}_2\text{Br}_2$ , LiTMP,  $-78\text{ }^{\circ}\text{C}$
- 5) LiHMDS, *n*-BuLi, TIPSOtF,  $-78\text{ }^{\circ}\text{C}$

Name and mechanism for the transformation in step 4 and 5



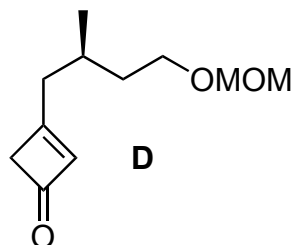
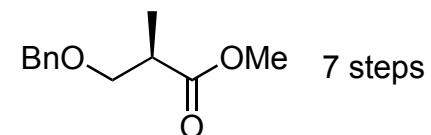
**C**

6-8  
↓

6) **D**, toluene, 80 °C  
7) TBAF, THF  
8) MeI, K<sub>2</sub>CO<sub>3</sub>

Name, mechanism of step 6  
and provide at least 2 more  
reactions of this type

How would you synthesize **D**  
from the given starting material:

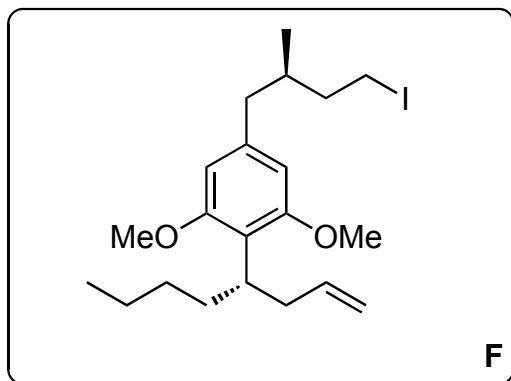


9) HCl, MeOH, 60 °C  
10) I<sub>2</sub>, PPh<sub>3</sub>, imidazole

9,10  
↓

11) OsO<sub>4</sub>, NMO  
12) NaIO<sub>4</sub>, THF  
13) TsNHNH<sub>2</sub>; TBSOTf, NEt<sub>3</sub>

11-13  
↓



name and mechanism of step 14

14) *t*-BuLi, THF,  $-78\text{ }^{\circ}\text{C}$ ;  
AcOH,  $\text{CF}_3\text{CH}_2\text{OH}$

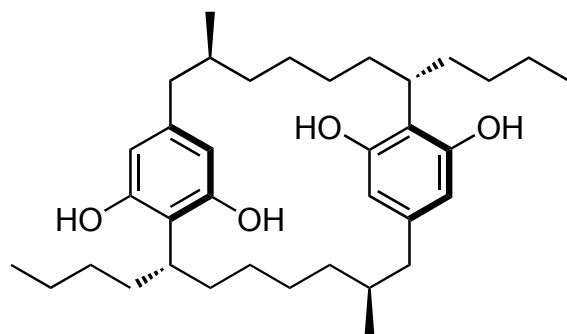
H

15–20

15) HCl, MeOH  
16) DMP  
17)  $\text{Ph}_3\text{P}=\text{CH}_2$ , THF,  $-78\text{ }^{\circ}\text{C}$   
18) Grubbs I,  $\text{CH}_2\text{Cl}_2$   
19)  $\text{H}_2$ , Pd/C, EtOAc  
20)  $\text{BBr}_3$ ,  $\text{CH}_2\text{Cl}_2$

structure and  
preparation of DMP

structure of Grubbs I



I

**(-)-cylindrocyclophane F**