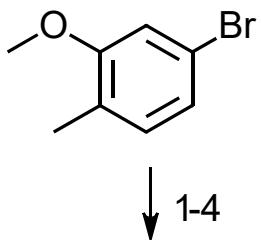


## Synthesis Challenge #73

AG Wegner

19.04.2018



A

↓ 1-4



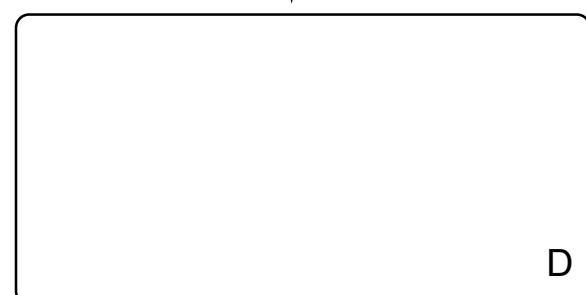
B

↓ 5-7



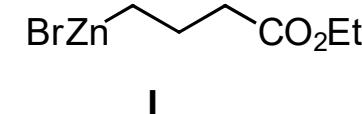
C

↓ 8-9



D

- 1) I (1.5 equiv), Pd(OAc)<sub>2</sub> (2.0 mol %), SPhos (4.0 mol %), THF, 50 °C, 8 h
- 2) AgOAc (1.0 equiv), I<sub>2</sub> (1.0 equiv), HOAc, 12 h, rt
- 3) LDA (1.5 equiv, 1.0 M in THF), HCOOMe (3.0 equiv), -78 °C → rt, 5 h
- 4) 3-penten-2-one (1.5 equiv), Cu(OTf)<sub>2</sub> (0.2 equiv), THF, 50 °C
  
- 5) NaBH<sub>4</sub> (0.8 equiv), CeCl<sub>3</sub>·7H<sub>2</sub>O (1.2 equiv), MeOH, 0 °C, 2 h
- 6) NaOH (10 equiv), MeOH/H<sub>2</sub>O (v/v = 1:1), 70 °C, 12 h
- 7) EDCI (1.2 equiv), DMAP (0.2 equiv), CH<sub>2</sub>Cl<sub>2</sub>, 0 °C → rt, 12 h
  
- 8) DIBAL-H (2.0 equiv), CH<sub>2</sub>Cl<sub>2</sub>, -78 °C
- 9) PPTS (0.1 equiv), MeOH, 50 °C, 7 h



I

↓ 10-11

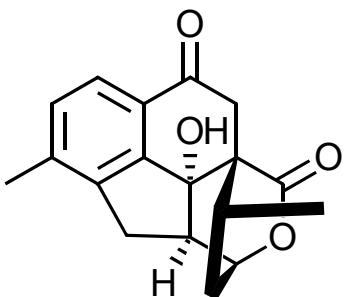
E

- 10)  $\text{Pd}(\text{OAc})_2$  (2.0 mol %),  $\text{Ph}_3\text{P}$  (4.0 mol %), CO (1 atm),  $\text{K}_2\text{CO}_3$  (2.0 equiv), PhMe, 90 °C, 18 h  
11)  $\text{BF}_3 \cdot \text{OEt}_2$  (1.2 equiv), *m*-CPBA (1.5 equiv),  $\text{CH}_2\text{Cl}_2$ , 0 °C, 1 h,  
then  $\text{Et}_3\text{N}$  (3.0 equiv), 0 °C → rt, 5 min

↓ 12-14

F

- 12) TfOH (10.0 equiv),  $\text{Et}_3\text{SiH}$  (10.0 equiv),  $\text{CH}_2\text{Cl}_2$ , rt, 40 h  
13) DDQ (2.0 equiv), 1:1  $\text{CH}_2\text{Cl}_2/\text{H}_2\text{O}$   
14) PCC (4.0 equiv), NaOAc (5.0 equiv), PhH, 70 °C, 12 h,



- 15)  $\text{BCl}_3$  (5.0 equiv), TBAI (4.0 equiv),  $\text{CH}_2\text{Cl}_2$ , -78 → -40 °C, 4 h  
16)  $\text{Tf}_2\text{O}$  (1.2 equiv), pyr. (1.5 equiv),  $\text{CH}_2\text{Cl}_2$ , 0 °C, 1 h  
17)  $\text{Pd}(\text{OAc})_2$  (10 mol %), dppp (10 mol %),  $\text{Et}_3\text{SiH}$  (2.5 equiv), DMF, 60 °C, 8 h

dppp = 1,3-bis-(diphenylphosphino)-propane