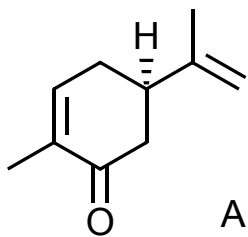


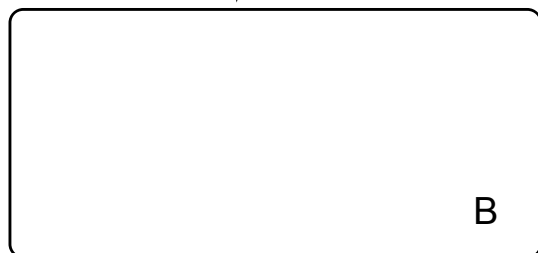
Synthesis Challenge # 47

AG Wegner

12.05.2016



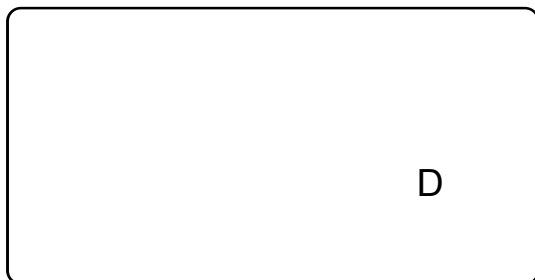
1-4



5-8



9-11



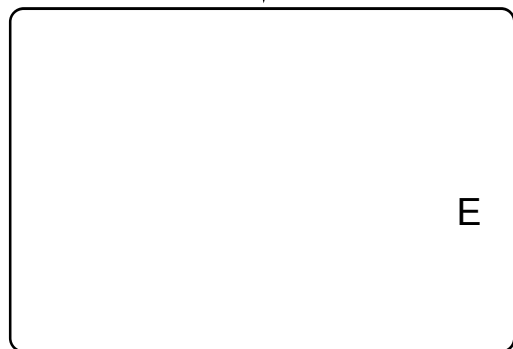
- 1) HBr, AcOH, 0 °C, 1 h
- 2) Br₂, AcOH, 23 °C, 2.5 h
- 3) *i*-PrNH₂, Et₂O, 0 °C, 12 h
- 4) 10% aq. AcOH, THF, 50 °C, 3 h

- 5) LiAlH₄, Et₂O, 0 °C, 1 h
- 6) Ac₂O, 150 °C
- 7) LiAlH₄, Et₂O, 0 °C, 1 h
- 8) DMP, NaHCO₃, H₂O, CH₂Cl₂, 23 °C,

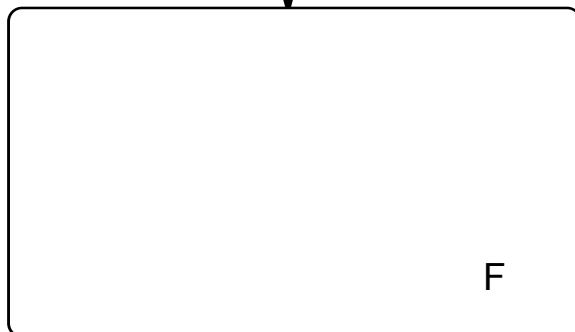
- 9) tetravinyltin, *n*-butyllithium, -78 to 23 °C, 15 min, then 10, -78 °C, 15 min, then HMPA, propargyl bromide, -78 to 23 °C, 3 h
- 10) *n*-butyllithium, -78 °C, 20 min, then: TMSCl, -78 to 23 °C
- 11) Pd(OAc)₂ (5 mol %), B₂pin₂, PhMe, MeOH, 50 °C, 15 h, then H₂O₂, NaOH, THF, 0 °C, 1 h

Hint: The synthesis has been designed to proceed on a multi gramm scale. The smallest scale on this page was 21 g.

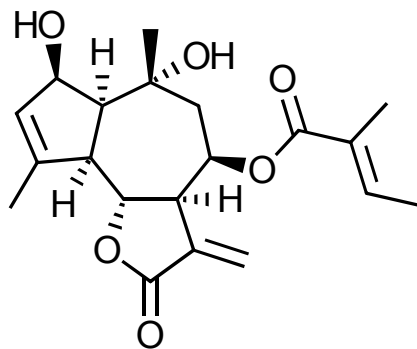
12-14



15-16



17-18



12) $(\text{COCl})_2$, DMSO, triethylamine,
 CH_2Cl_2 , -78°C , 2.5 h
13) Et_2AlCl , CH_2Cl_2 , -78°C
14) tiglic acid, 2,4,6-TCBC, triethyl-
amine, DMAP, PhMe, 80°C , 2 h

15) TFA, CH_2Cl_2 , 23°C , 2 h
16) CrO_3 , 3,5-DMP, CH_2Cl_2 , 0°C

17) $\text{Yb}(\text{OTf})_3$, NaBH_4 , MeOH/THF,
 -78°C , 2 h
18) $\text{Al}(\text{O}^{\text{sec}}\text{-Bu})_3$, TBHP, CH_2Cl_2 ,
 0 to 23°C , 40 min,
then LiCl, HCl, THF, 23°C , 5 min

3,5-DMP = 3,5-dimethylpyrazole