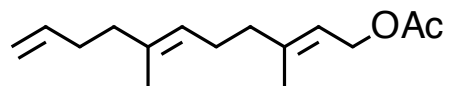


Synthesis Challenge # 33

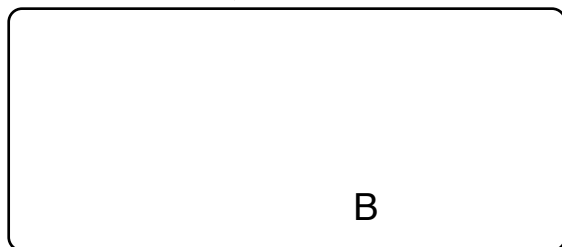
AG Wegner

30.04.2015



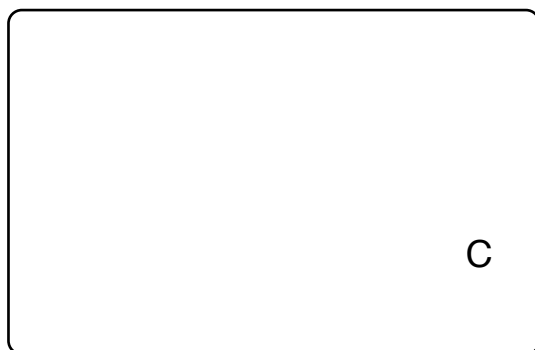
A

1-4



B

5-8



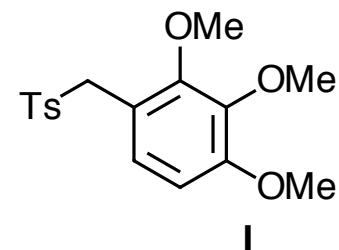
C

- 1) 9-BBN (1.05 equiv), then aq. NaHCO₃, aq. H₂O₂ (30 wt %)
- 2) DMP (1.2 equiv), CH₂Cl₂,
- 3) vinylmagnesium bromide
- 4) TBSCl, imidazole, DMF; K₂CO₃ (1.0 equiv), MeOH,

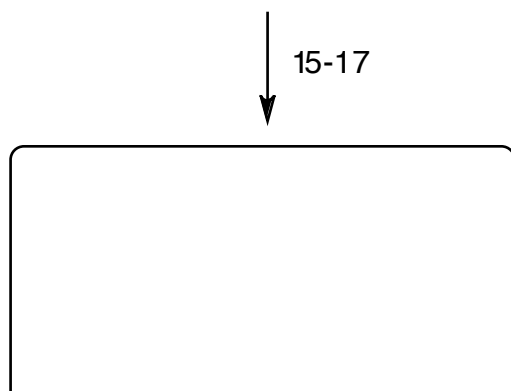
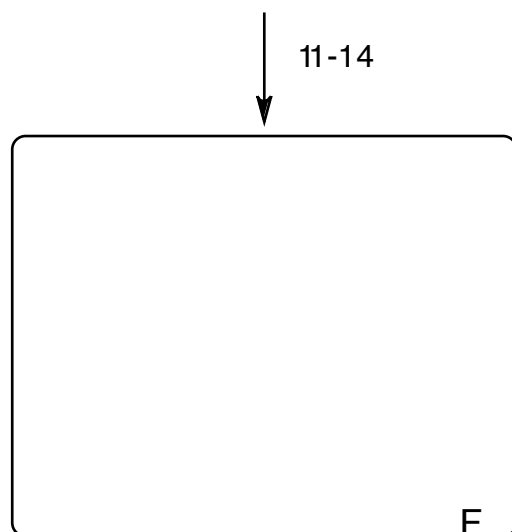
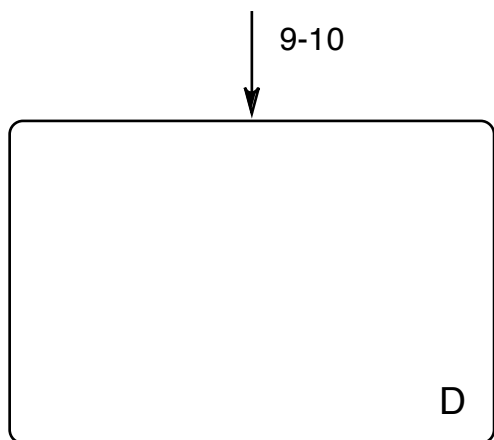
- 5) MsCl, Et₃N, LiBr, THF
- 6) I, KHMDS (1.05 equiv), THF, -78°C, then product of step 5) -78°C
- 7) Na(Hg), Na₂HPO₄, MeOH, -20°C
- 8) HF·py/ THF (1:10),

Please design a synthesis of A starting from farnesol.

Please, provide a detailed mechanism for step 1.



I



9) $[\{\text{Ir}(\text{cod})\text{Cl}\}_2]$ (4 mol%), *R-II* (16 mol%),
 $\text{Zn}(\text{OTf})_2$ (20 mol%), DCE, 22°C

10) $\text{BF}_3 \cdot \text{OEt}_2$ (2.5 equiv), CH_2Cl_2 , 0 °C,

Step 9 and 10 promote the same transformation. Please, provide a detailed mechanism.

11) $\text{K}_2\text{OsO}_2(\text{OH})_2$ (10 mol %),
2,6-lutidine (1.0 equiv), NaIO
(3.0 equiv), acetone/water (3:1)

12) *t*BuOK, MeI (10 equiv), *t*BuOH

13) $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, diethylene glycol,
160°C, 2 h, then KOH, 180°C

14) 3,5-dimethylpyrazole, CrO_3

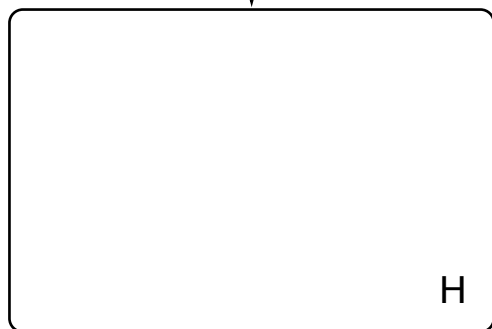
Please, provide a detailed mechanism for step 13).

15) AlCl_3 (1.1 equiv), CH_2Cl_2

16) NaBH_4 , MeOH/ CH_2Cl_2 (1:1)

17) TMSBr (20 mol %), InCl_3 (10 mol %),
TMSCN (1.2 equiv), MeCN

18-19



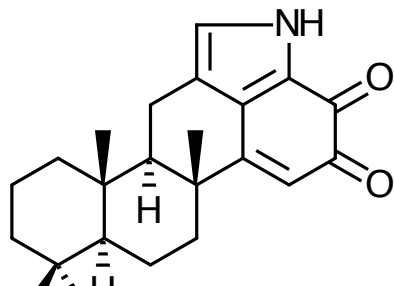
H

20-21



I

22-23



- 18) $\text{BH}_3 \cdot \text{THF}$, THF, 50 °C
19) Tf_2O , Et_3N , 4-DMAP (10 mol %)

- 20) CuI (4.0 equiv), CsOAc , NMP, 160°C, 4 h
21) DDQ (5.0 equiv), toluene, 110°C,

- 22) BBr_3 (10.0 equiv), CH_2Cl_2 , -78-22°C,
23) Mg (10.0 equiv), NH_4Cl (2.0 equiv), MeOH, sonication, 5 min, then work up under an air atmosphere,