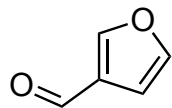
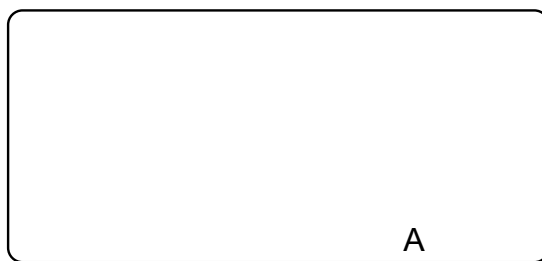


Synthesis Challenge #5 AG Wegner
JLU Giessen
7.11.2013



↓ 1,2,3



↓ 4



1) *n*-BuLi, morpholine, THF, $-78\text{ }^{\circ}\text{C}$,
then *s*-BuLi, then I_2
2) $\text{CH}(\text{OMe})_3$, $\text{TsOH}\cdot\text{H}_2\text{O}$, 3 Å
molecular sieves (MS)
3) $\text{Pd}(\text{OAc})_2$, allyl alcohol, NaHCO_3 ,
DMF, $50\text{ }^{\circ}\text{C}$

4) KHMDS, $\text{BocNH}(\text{CH}_2)_5\text{PPh}_3\text{I}$, -78
to $0\text{ }^{\circ}\text{C}$, then HCl

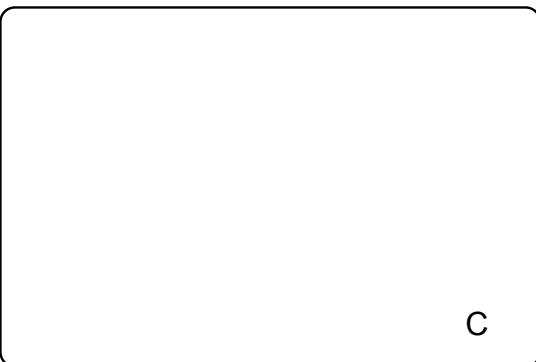
What is the name of the reaction in step 3)?

Please give a detailed mechanism of step 4)?

5



5) $\text{CH}_3\text{O}_2\text{CCH}_2\text{P}(\text{O})(\text{OCH}_2\text{CF}_3)_2$,
18-crown-6, KHMDS, THF, -78°C

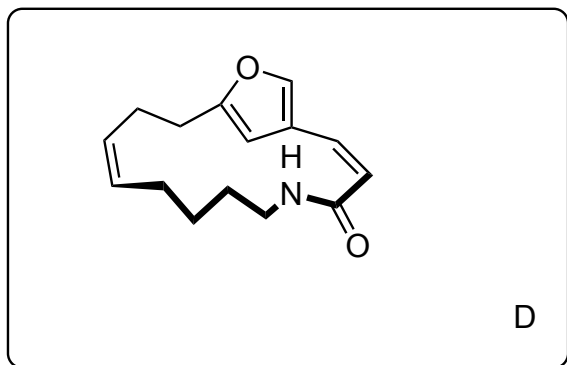


What is name of the reaction in step 5)

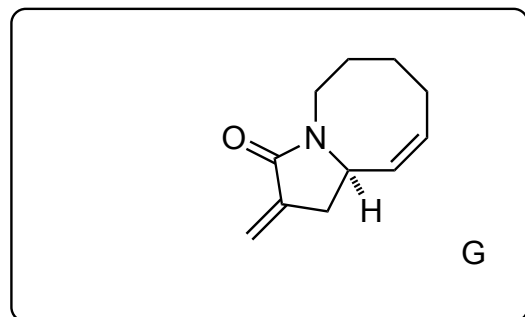
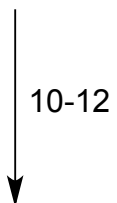
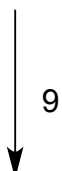
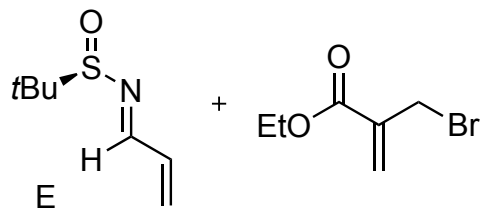
6-8



6) NaOH, MeOH, H_2O , rt
7) TFA, DCM, 0°C to rt
8) HBTU, NEt_3 , CH_3CN , 50°C



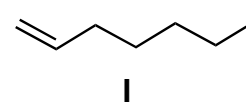
What is HBTU?



9) Zn, LiCl, DMF, H₂O (1 equiv)

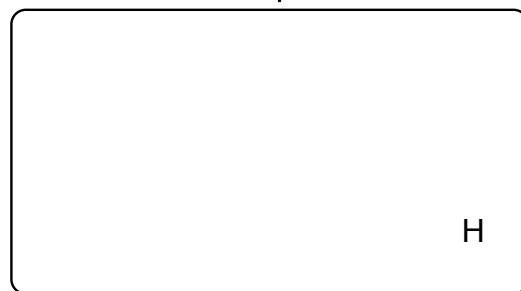
10) HCl, MeOH, then NaOH
11) NaH, I, DMF
12) 1st Grubbs

Please, determine the absolute configuration of E.

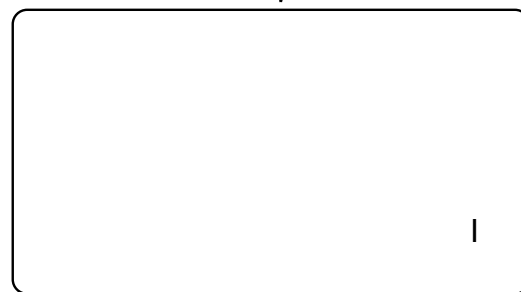


D + G

13
↓



14
↓

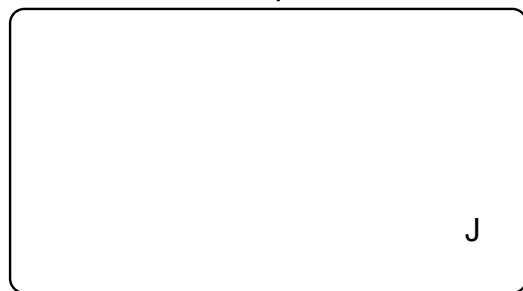


13) **D**, TBSOTf, *i*Pr₂NEt, DCE, rt,
then **G**, DCE, 14 h;

14) Me₃OBF₄, 4 Å MS, DCM, rt, 2
h, then NaBH₄, MeOH, 0 °C to r

Please give a detailed mechanism of step 13)?

15



J

15) Tf_2O , 2,6-di-*tert*-butyl-4-methylpyridine, DCM, rt, 30 min, then NaBH_3CN , MeOH, rt;