Synthesis of $( \pm)$-Vibralactone I - Working with mixtures
Q. Zhou, B.B. Snyder, Org. Lett. 2008, 10, 1401.

Diastereoselective Update
Q. Zhou, B.B. Snyder, J. Org. Chem. 2008, 73, 8049

1. $\mathrm{K}, \mathrm{NH}_{3},-78^{\circ} \mathrm{C}$



A

minor
recycled by DMP-oxidation
$+$

major

C
8. $\mathrm{O}_{3}, 1: 1 \mathrm{DCM} / \mathrm{MeOH}-78{ }^{\circ} \mathrm{C}$ then $\mathrm{PPh}_{3}-78^{\circ} \mathrm{C} \rightarrow \mathrm{rt}$
9. $\mathrm{Bn}_{2} \mathrm{NH} \cdot \mathrm{TFA}$, benzene, rt
$+$

minor
E


F
10. $\mathrm{Zn}, 4: 1 \mathrm{THF} / \mathrm{HOAc}$,
$0^{\circ} \mathrm{C}$


G
11. $\mathrm{TsCl}, \mathrm{py}, 0^{\circ} \mathrm{C}$
12. $\mathrm{NaBH}_{4}, 100: 1 \mathrm{DME} / \mathrm{H}_{2} \mathrm{O}$ $0^{\circ} \mathrm{C} \rightarrow \mathrm{rt}$

intermediate after 11

( $\pm$ )-Vibralactone

1. $(\mathrm{COCl})_{2}$, DMF, DCM, then $\mathrm{MeNH}(\mathrm{OMe}) \cdot \mathrm{HCl}$, $\mathrm{K}_{2} \mathrm{CO}_{3} \mathrm{Et}_{2} \mathrm{O} / \mathrm{H}_{2} \mathrm{O}$

2. NaHMDS, THF, 3,3-dimethylallylbromide, $-78^{\circ} \mathrm{C} \rightarrow \mathrm{rt}$


A

B

5. $\mathrm{MgBr}_{2} \mathrm{OEt}_{2} \mathrm{DCM}$, allyltributyltin, $-78^{\circ} \mathrm{C}$ 6. $\mathrm{Ms}_{2} \mathrm{O}, \mathrm{pyr}$, DMAP, DCM



E


F
3. $\mathrm{TiCl}_{4}, \mathrm{NEt}_{3}, \mathrm{DCM}, \mathrm{CH}(\mathrm{OMe})_{3}$ $\xrightarrow{\text { 4. DIBAI-H, DCM, }-78^{\circ} \mathrm{C}}$

