

C<sub>12</sub>H<sub>18</sub>O<sub>6</sub>

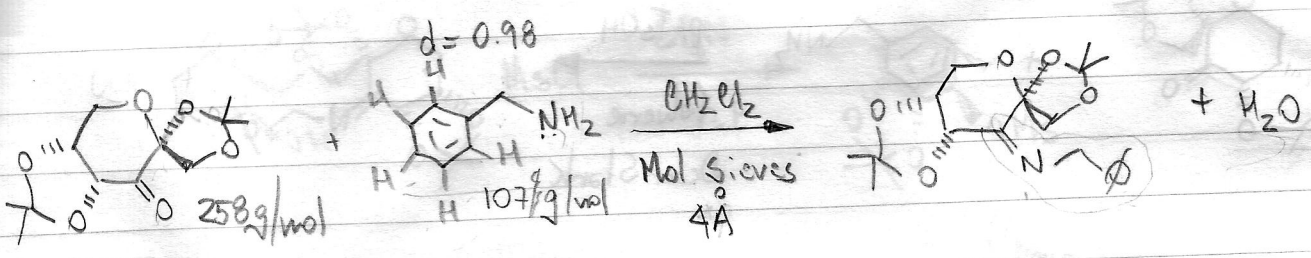
C<sub>7</sub>H<sub>9</sub>N

18  
9  
96  
258  
14  
107

2

Syntheses 01

Ref. Eur JOC 2007, 3038 ; OL 2007, 2633

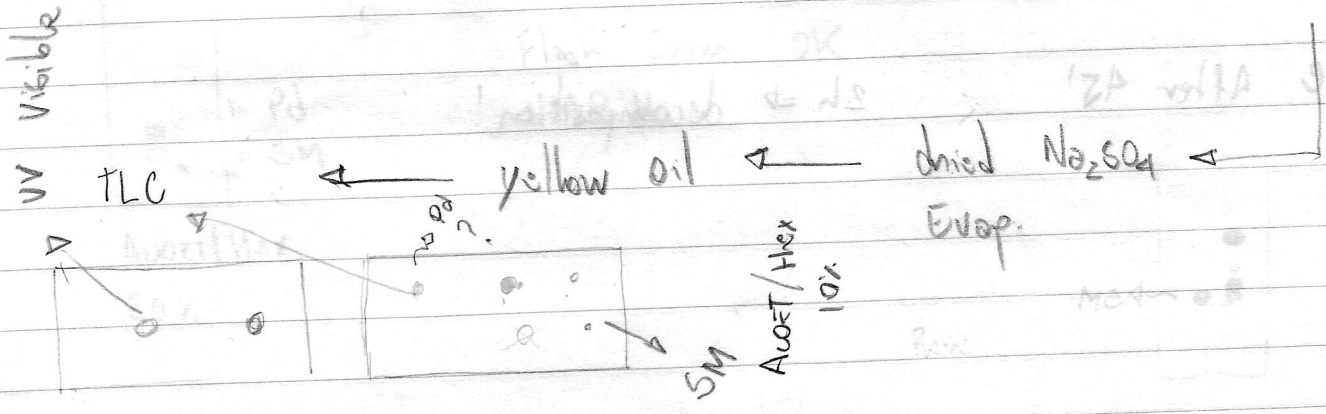


52 mg Ketone (0.2 mmol)  
 20 μL benzyl amine (0.2 mmol)  
 40 mg Mol sieves  
 0.2 mL CH<sub>2</sub>Cl<sub>2</sub>

rt  
 72h

Workup:
 

- CH<sub>2</sub>Cl<sub>2</sub> 40 mL
- HCl 5% 2x
- NaHCO<sub>3</sub> 5% 1x
- Brine 1x



NMR<sup>1</sup>H ⇒ Indicates the formation of the desired product. SM (crude) is also present. the amine is not observed.

TLC ⇒ spots stain with phosphomolibdic acid 7% EtOH  
 Flash Chrom. ~35mg Crude. AcOEt/Hex 5% 40mL then 10% 50mL

m = 25.9043 ~ 2mg 1st spot	① G1-1
58.4842 / 58.5015 last fraction ~ 15mg	③ G1-3
36.5740 / 36.5860 2nd spot	② G1-2

last fraction eluted with pure AcOEt ~100mL