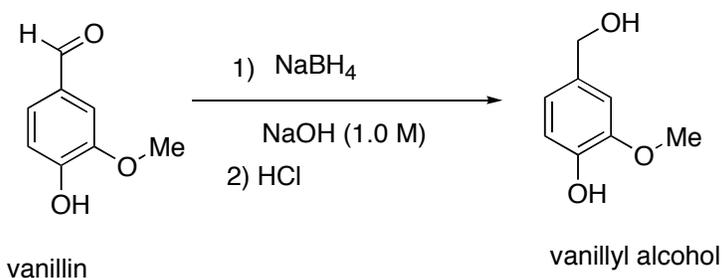


Vanillin Reduction



To a 25 mL round bottomed flask add vanillin (2.0 g), ethanol (4 mL) and a stir bar. The round bottomed flask is then clamped above the magnetic stirrer. Stir vanillin solution until it is homogeneous (5-10 min), and then cool the solution with an ice bath. During this process, prepare a sodium borohydride solution by dissolving 0.5 g of sodium borohydride in 3.8 mL of 1.0 M NaOH. Add the sodium borohydride solution dropwise to the vanillin solution. (Caution: hydrogen evolves and the reaction is exothermic. Add the sodium borohydride solution slowly.) After the addition is completed, remove the ice bath and allow stirring to continue at r.t. Monitor the reaction by TLC every 10 mins (take a small aliquot from the reaction mixture dissolve it in a 1:1 mixture of Ether:Water (2 mL) and spot the ether solution in the TLC). Once the starting material disappeared by TLC, cool down the reaction mixture using ice bath, and add 6.0 M HCl slowly until hydrogen evolution stops (the addition of HCl decomposes the excess sodium borohydride). After the addition of HCl is completed, stir the solution for 10 min. Filter the precipitate that forms, and suction dry your product and check the melting point. Further verify the formation of your product by “co-spotting” your product with authentic vanillyl alcohol, and analyzing by TLC.

For your lab report:

- comment on how you might be able to monitor the progress of this reaction by ^1H NMR and ^{13}C NMR. Which peaks are diagnostics for your starting material and product.
- explain why the product is soluble in aqueous NaOH, but precipitates upon addition of HCl.