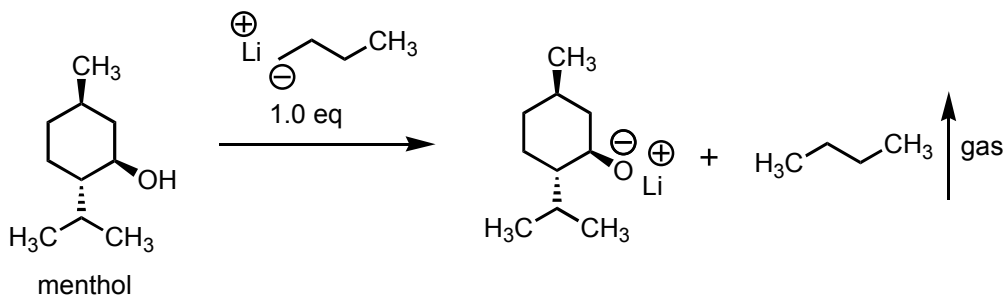


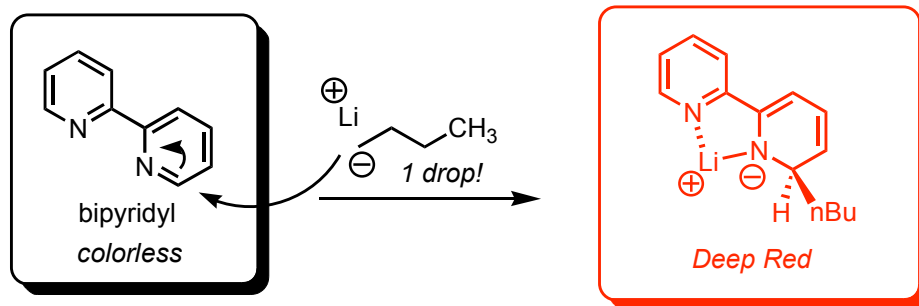
Shown below is the chemical reaction that is occurring during the titration of butyl-lithium, as well as a sample calculation for determining the molarity of the butyl lithium solution. In the first step of the process, one eq. of menthol is deprotonated by the unknown butyl lithium solution. The resulting colorless anion then sits in solution. As soon as any excess butyl lithium is added step 2 of the process begins.

Step 1:



Any of the excess butyl lithium then alkylates the bipy in solution generating

Step 2:



Titration of alkyl lithium solutions using a charge transfer complex indicator

An oven dried 25 mL two neck flask equipped with a nitrogen inlet adapter, a stirring bar and rubber septa was assembled hot and cooled under a stream of Argon (**Note 1**). Upon cooling the flask was charged with *ca.* 200 mg of menthol (**but accurately weighed out!**), *ca.* 5 mg of 2,2'-dipyridyl and 10 mL of dry THF (**Note 2**). To the resulting solution is added the unknown BuLi solution via a 1.0 mL syringe (graduated in 0.01 mL increments) in a dropwise fashion. During addition, it will be observed that periodic quantities of a red colored substance will appear in solution. **This is the charge transfer complex between the alkyl lithium and 2,2'-dipyridyl, and it will be noted that in the early stages of this titration this red**

color will disperse rapidly. As one nears the endpoint, the red coloration will require longer periods of time to disperse. At this point, it is necessary to slow the rate of addition of the alkyl lithium reagent as the endpoint is approaching. Eventually, the addition of a single drop of alkyl lithium will cause a persistent red coloration of the solution. This is the endpoint of the titration. Take note of the final volume of the syringe. The difference between the original volume of organometallic solution and this final volume of solution represents the volume of the titrant. Repeat this titration two additional times using a fresh flask, solvent, 2,2'-pyridyl, and menthol. The average of these three runs constitutes the molarity of the alkyl lithium solution.

Note 1: The flask must be oven dried for at least 2 hours, or flame dried and cooled under a stream of dry argon immediately before use.
Note 2: Exclusion of water from the transfer is integral to the success of the titration.

Use the below calculation to determine the concentration of your titrated BuLi solution.

Ex: Assume the average of your additions was 0.73 mL of BuLi solution.

Since 0.200g menthol = 1.30mmol menthol, which reacts with **1.30mmol BuLi**; this amount of RLi was present in **0.73mL** of the unknown BuLi solution. Since molarity equals moles/L, it also equals mmol/mL. Thus:

1.30 mmol RLi / 0.73mL solution = 1.78M BuLi soln.

Once you have determined the concentration, make sure to clearly label the bottle with the concentration and initials/date.