Use of a Titanocene Complex as a Colorimetic Indicator for Learning Inert Atmosphere Techniques

Before beginning a full-scale preparation, you must first conduct the following simple test to determine your synthetic skills in handling air-sensitive compounds. This simple experiment will give you a quick and easy indication of whether you can manipulate an oxygen-sensitive compound through a variety of steps such as filtration, cannula transfer, and short-term storage. Note that the compound you're making is not water sensitive.

Procedure

Stir 0.1 g of Cp_2TiCl_2 in 20 mL of acetonitrile in a 100 mL Schlenk flask using a magentic stirrer. Bubble nitrogen through the solution via a cannula inserted through a Suba Seal inserted in the 24/40 joint (you'll also need to insert a vent).
Under a nitrogen counterflow, add a few grams of Zn *dust* and continue stirring. Periodically stop the stirrer and examine the solution.
When you have a nice blue/purple solution on settling, filter this into a Schlenk tube using a cannula with a Whatman #1 filter paper wired to it.
Practice manipulating the resulting solution:

- Put a few drops in a test tube in air to see how quickly it changes color (decomposes!).
- Use a syringe to see how well you can transfer the solution without decomposition
- Add some non-degassed acetonitrile to see the effect of dissolved oxygen.
- Stopper some of the solution to see how long your sample survives – compare with your neigbors.

When you can routinely handle these solutions, you're ready to move on to the experiments in the rest of Group A.