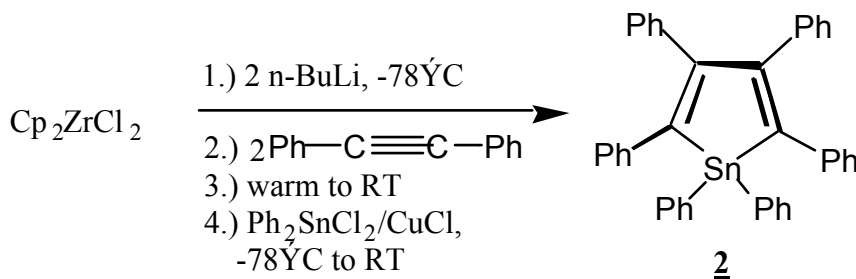


Unit 5/6M: Introduction to Organic Synthesis V & VI
Preparation of Hexaphenylstannole via a Zirconacycle Intermediate

This assignment is due at the beginning of your recitation section one week before the day that you are scheduled to perform this experiment. It is in your best interests to complete these exercises as far in advance as practical. It is not in your best interests to wait until the day you have lab. If you do not understand how to solve any of these problems, you should contact your laboratory instructor before the recitation period.

To receive maximum credit your answers must be presented clearly. Looks count. Do your preliminary work on scrap paper.

The **Introduction** to this Unit describes the formation of the intermediate zirconacycle followed by the transmetalation reaction to yield the tin-containing metallole, hexaphenylstannole (2). A condensed chemical equation for this two step reaction is shown below.



1. Complete the following table: (record formula weights to the nearest tenth of a gram)

Compound	Formula Weight
zirconocene dichloride	
diphenylacetylene	
diphenyltin(IV) dichloride	
cuprous chloride	

2. Calculate how many milligrams of zirconocene dichloride you will need to run this reaction if your 100% yield of product is 0.500 mmol.

_____ mg

3. Butyllithium is sold as a 1.60M solution in hexanes. If you start with 0.500 mmol of zirconocene dichloride, how many milliliters of 1.60M butyllithium do you need?

_____ mL

4. Again, assuming that you start with 0.500 mmol of zirconocene dichloride, how many milligrams of diphenylacetylene must you add to your reaction solution?

_____ mg

5. Calculate the weight of zirconacycle intermediate (**1**, found in the **Introduction** to this unit), in milligrams, that you will get if you have a 100% yield.

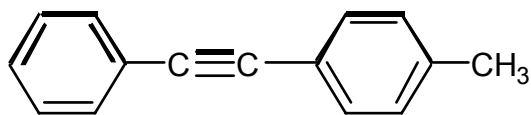
_____ mg

6. Calculate the weight, in milligrams, of the hexaphenylstammole product you could generate (i.e., theoretical yield).

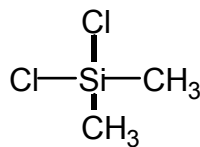
_____ mg

7. If the zirconocene dichloride, diphenylacetylene, diphenyltin dichloride, and cuprous chloride reagents are available in the dry boxes (as they should be), list every piece of equipment that you need to bring into the dry box in order to execute this reaction.

8. If two equivalents of the unsymmetrical acetylene, 1-(4-methylphenyl)-2-(phenyl)ethyne, were used to form a zirconacycle and the main group halide used was dimethylsilicon dichloride, what would the structures of the resulting metallole(s) be? (Draw all possible metalloles.)



1-(4-methylphenyl)-2-(phenyl)ethyne



dimethylsilicon dichloride

9. Suggest a spectroscopic method to distinguish between the possible products in (8) above. Explain your answer.