Chemistry 146B, Spring 2009

Experiment 2. Preparation of an Organometallic Compound Containing a Sn-Mn Bond

Synthesis Involving a M-M Bond

Assemble the apparatus that is required and flush the system with nitrogen. Weigh out 15 g of Hg and place in a 100 mL 3-neck round bottom flask. Stir the Hg magnetically and slowly add 0.15 g of sodium pieces. The sodium should be pre-weighed in hexanes and then cut into about 10 pieces while still in hexanes. Finally, each piece should be washed with hexanes, dried on a piece of filter paper and quickly dropped into the Hg. *Do not handle the sodium with your hand but use spatulas, scoopulas and tweezers (see Note 1).*

Allow the amalgamation to complete before adding the next piece. The reaction is exothermic and accompanied by a sizzling sound. When finished adding all Na, weigh out 0.5 g *(see Note 2)* of $Mn_2(CO)_{10}$ and dissolve in 25 mL of THF provided. This solvent has been distilled over sodium and under nitrogen *(see Note 3)*. Add the $Mn_2(CO)_{10}$ solution to the amalgam and stir for 30 min. Keep all systems under N_2.

Add 1.0 g of triphenyl tin chloride dissolved in 20 mL THF. Stir the mixture well for 20 minutes. Dilute the reaction mixture by adding a volume of dry THF equal to the total volume of the present reaction mixture (~ 50 mL). Filter off any residual precipitate by suction [use filter-aid (celite) in the sintered glass frit] and reduce the volume of the solution to about one-half. Very slowly, add a greater than or equal amount of water and filter off your precipitate by suction.

Next, you will recrystallize the precipitate from hot hexanes. Dissolve the complex in ~ 30 to 50 mL hot hexanes, pass through a filter pre-wet with hot hexanes, and allow the complex to crystallize. When dry, measure the MP and IR spectrum. Compare the IR spectrum of the product with that of $Mn_2(CO)_{10}$.

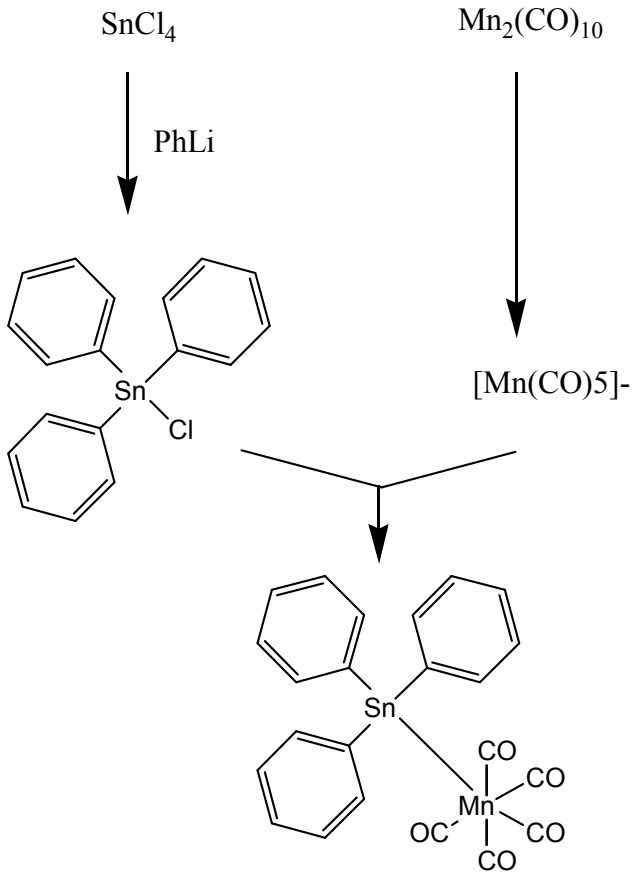
Notes

1. Weighing out sodium metal: obtain a beaker containing hexanes, and immerse several round sodium metal spheres to rinse the residual oil. Cut one sphere into 4-5 pieces (while immersed in hexanes!), then lay them on a dry paper towel. Quickly pat them dry and weigh out the ~ 0.15 g of Na. After weighing, place the Na back into hexanes (so it doesn't react further with air) until ready for use. Repeat until you have weighed out approximately 0.15 g of Na. Any small fragments of Na left on the counter or on the filter paper must be disposed of by placing into a jar of butanol in the fumehood.
2. $Mn_2(CO)_{10}$ is air sensitive and very expensive. Please weigh out exactly 0.5 g. Do not waste.
3. When you have finished with the flask containing sodium amalgam, DO NOT wash the flask with water. You should use butanol to decompose any unreacted sodium remaining in the flask first. After allowing the residue to

stand in butanol for 20 minutes, discard the residue in butanol into a jar of butanol in the fumehood.

4. Mercury should always be stored in tightly closed bottles and never allowed to spill. Once Mercury has spilled, it is best removed using a mercury vacuum. As much mercury as possible should be removed with this apparatus, although it is a tedious chore. The very small droplets of mercury remaining should be sprinkled with powdered sulfur. Mercury reacts readily with sulfur to give HgS. Since the formation of HgS occurs only on the surface of the droplets and a disturbance of the droplets produces a fresh Hg surface, treatment with sulfur is at best a temporary method of reducing the vaporization of mercury. By far the best method of avoiding mercury contamination in a lab is not to spill it.

Pentacarbonyl Manganese Adduct of Triphenyltin



Element	Mass No.	Abundance (%)
Sn	112	0.96
	114	0.66
	115	0.35
	116	14.30
	117	7.61
	118	24.03
	119	8.58
	120	32.85
	122	4.72
	124	5.94
Cl	35	75.5
	37	24.5

Propose a fragmentation pathway for the product, showing the various peaks observed and their assignments. Considering both the mass spectrum and the IR spectrum, what is your conclusion as to the structure of the product?