

# 1. Transfer and Extraction Techniques

## 1.2. Expert Experimentalist Rating: “*Into Thin Air*”

### Techniques Checklist:

- Cannula transfer
- Vacuum manifold use
- Manipulations under an inert atmosphere
- Solvent de-gassing

### Pre-Lab Discussion and Required Reading:

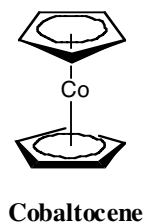
- Air sensitive techniques: LLP Ch. 9

### Equipment:

- Vacuum manifold
- Solvent trap
- Magnetic stir plate
- Cannula needle
- 25-mL Schlenk flask
- Schlenk frit
- 100-mL Round-bottomed flask - 14/20
- Small round-bottomed flask - 14/20
- Disposable purge needles
- UV-Vis cuvettes - quartz
- 14/20 rubber septa
- Smaller, tan septa (to fit cuvettes)
- Keck clips (14/20)
- Fresh bottle of tetrahydrofuran (THF) (you will only need 50 mL)
- Activated 4 Å molecular sieves

## Goal:

You will be given a small sample of cobaltocene (dicyclopentadienylcobalt) in a nitrogen-filled Schlenk flask. Your task is to carry this material through an "obstacle course" of manipulations without allowing your sample to decompose - as determined by UV-Vis spectroscopy. *Beware: This sensitive compound will quickly decompose upon exposure to air!*



## Experiment Outline:

- All of your glassware should be CLEAN and oven-dried (put the glassware in the oven the day before you perform the experiment).
- De-gas about 50 mL of THF in a 100-mL round bottom flask - *see No-Air Techniques Guide*.
- While your solvent is de-gassing, evacuate your filtration apparatus - *see No-Air Techniques Guide*.
- Attach the Schlenk flask containing your sample to a hose on the manifold (secure with copper wire), and carry out several vac-fill cycles to clear all of the air from the length of the tubing.
- At some point, you will need to purge the cuvettes, fill with degassed THF (you can use a cannula or syringe) and blank the spectrophotometer - *see UV-Vis Operation Guide*. If the spectrophotometer is not busy, you can do this while your various components are de-aerating.
- *Carefully* cannulate THF into the sample flask. You will need a dilute solution for the UV-Vis experiment, so add solvent until you get a pale solution – about 10 mL.
- *Carefully* filter the solution into the receiving Schlenk flask - *see No-Air Techniques Guide: Filtering*.
- Using a cannula, transfer the solution to a N<sub>2</sub>-purged, septum-capped cuvette. (Put a little grease over the puncture wounds on the septa to maintain their integrity.)
- Blank the UV-Vis spectrophotometer with THF, then obtain your spectrum.

## Results:

• To obtain your "EE Rating" in Transfer and Extraction Techniques, your UV-Vis spectrum must contain a single peak at 347 nm. If  $\lambda_{\text{max}}$  is below 347 nm and if a shoulder appears around 392 nm, your sample has decomposed. Go back to the start and try again!