# Implementation Notes

# Solvents

Currently, a Glass Contour solvent purification system is used to provide access to dry, de-gassed solvents at Carleton College. Before this system was installed, the laboratory experiment was performed using anhydrous solvents purchased from Aldrich in SureSeal® bottles, which were brought directly into the glove box. After removing the SureSeal® caps, the instructor passed THF through activated alumina prior to use by students, although the other solvents were used as received.

# Managing Student Work-flow

The experiment is performed over three 4-hour lab periods, although it does not require students to work for the entire period. Students also use the same lab periods to work on a different experiment or obtain spectral data, during the time that they are not using the glove box. Students are assigned to work in pairs and Carleton’s glove box has two sets of gloves. We choose to have two pairs of students perform the experiment simultaneously, with group members alternating performing tasks in the glove box, so that every student has the opportunity to develop glove box techniques during each laboratory period. By staggering the times that groups begin the experiment, two additional pairs can complete the week’s experiment within a single four-hour window. This enables a total of eight students per lab period to complete the experiment within three weeks.

In the first week, students prepare crude CpMo(CO)3(CH3). After the first two pairs of students add methyl iodide to their reaction mixtures (ca. 1-1.25 hours into the lab period), a second set of two pairs begins the synthesis. Two hours after setting up their reactions, the first two pairs return to begin evaporating THF from their crude reaction mixtures. The second two pairs also return after two hours to evaporate their solutions. The small scale enables two complete cycles of the synthesis to be completed within a four-hour window. If there is down time, it is used to prepare and obtain solution IR spectra of the starting material.

Students purify CpMo(CO)3(CH3) and prepare CpMo(CO)2(COCH3)(PR3) in the second week. After the first two pairs set up the reaction with PR3 (ca. 1.5 h), the second two pairs begin. Students prepare solution IR samples of CpMo(CO)3(CH3) in the glove box and acquire IR spectra when they are not actively using the glove box.

Students isolate (and purify) CpMo(CO)2(COCH3)(PR3) in the third week and obtain solution IR spectra of CpMo(CO)3(CH3) and CpMo(CO)2(COCH3)(PR3), as needed.

Students prepare NMR samples in Wilmad® LPV NMR tubes after purging the glove box near the end of the lab periods during weeks 2 and 3. NMR data are usually acquired outside of normal lab periods.