Substitutions in $[Mo(CO)_6]$

ABSTRACT
This experiment is an introductory exercise designed to encourage group work, while teaching the use of the ir and nmr spectrometers at the 300 level. A simple substitution of one, two or three carbonyl groups in the title compound is effected by refluxing a stoichiometric amount of ligand with $[Mo(CO)_6]$ in toluene under nitrogen.

Equipment
Characterization is done by IR & NMR spectroscopy. All reactions and columns should be done in a fumehood.

Safety Hazards
All the ligands and $Mo(CO)_6$ are toxic to varying extents. Halogenated solvents are carcinogenic. Toluene is flammable.

Year Level: 300

Student time required: two to three 3-hour lab periods.

Teaching notes? Available only to instructors upon request.

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Experiment A6
SUBSTITUTIONS IN $[Mo(CO)_6]$

This is an experiment that is designed to walk you through the basics of performing a synthesis and purification while monitoring the progress and success by spectroscopy. It typifies the kind of synthesis you will be doing in this course. Apart from learning how to run the instruments to get functionally useful spectra (!), the important message to grasp is the comparison of data that you have recorded before and after each step in a synthesis or purification.

You will be working as part of a group, although you will each have a different ligand or stoichiometric ratio to use. You will be expected to collaborate with experimental information and results but you must write your (tabulated) report independently. You will be given a group interview by your instructor during the next lab period following the first due date. You will not be required to report anything from the literature - the emphasis for this first experiment is to concentrate on reporting the data from you and the conclusions from the experiments run by your group members.

Procedure:
Deoxygenate toluene (25 mL) by bubbling nitrogen through it for 5 minutes. Weigh, in a closed vessel, $[Mo(CO)_6]$ (~0.25 g). Add this to the toluene and keep the solution under nitrogen. Add one of the following reagents and reflux under nitrogen for 1 h.

Possible reagents:
1 equivalent of tritolylphosphine
2 equivalents of tritolylphosphine
3 equivalents of tritolylphosphine
1 equivalent of 2,2'-bipyridyl
1 equivalent of 1,10-phenanthroline
1 equivalent of bis(diphenylphosphino)methane
1 equivalent of 1,2-bis(diphenylphosphino)ethane
1 equivalent of 1,2-diaminoethane (use pipettor)

Cool to room temperature and then (in air) rotovap to dryness. Record a solution (CH_2Cl_2) ir spectrum of the crude reaction mixture. Clean up the crude reaction mixture by either recrystallization or by running a silica gel column.

After the clean up procedure, collect a full set of nmr data. Recycle your sample so that you can record the solution ir spectrum again.

In the oral, you will be expected to compare your results with those from others in this lab who

used the same ligand (but different stoichiometry) and with those who used a different ligand and the same stoichiometry.

Report:
Please read the course notes for a general explanation of what is expected in the report.

Tabulate the spectroscopic data. Make sure that you construct your tables to clearly show the comparison between the starting materials and the products. Interpret the spectra as fully as possible. You are NOT required to perform a literature search but you should assign as much of the spectrum as you can rationalise. Justify why you think you have (or have not) been successful in the synthesis and separation process.

The data format that you are advised to adopt is that described in Chem 213.