Making and Characterizing New Compounds

As a synthetic lab, we will (hopefully) be synthesizing *many* new compounds. As you do this, it is *extremely* important to maintain an accurate and thorough report of all procedures and analytics associated with new compounds. The following should be performed *as soon as possible* upon the successful, clean synthesis of a compound unknown in the literature, whether it be a simple organic/ligand precursor or a target organometallic compound, and should be considered the *minimum* required characterization for any new compound:

- 1. **Full NMR characterization**. ¹H and ¹³C for all compounds. If you have common NMR active heteroatoms (³¹P, ¹⁹F for example), these should be recorded as well.
- 2. For organic compounds, the **High Res Mass Spec** of the compounds. Some inorganic/organometallic compounds are amenable to MS, but not all.
- 3. In lieu of HRMS, H, C, and N **elemental analysis** of the compound should be completed. In rare cases it may be necessary to get additional elements, but this is expensive and should be limited. Only submit spectroscopically pure material for elemental analysis, and check with lan before sending out. Our current contractor for elemental analysis is:

Midwest Microlab Inc. 7212 N. Shadeland Avenue, Suite 110 Indianapolis, IN 46250

Sample forms: http://midwestlab.com/submission-form/

Before sending out samples for EA, you <u>must</u> contact Tonks so he can quality check samples and provide proper mailing reimbursement accounts.

- 4. **X-Ray Diffraction Crystallography** should be attempted on crystalline organometallic/inorganic compounds.
- 5. The requirement of additional characterization techniques (UV-Vis, IR, cyclic voltammetry) is project-specific. If your project requires these, *do not hesitate* on collecting them.
- 6. **Journal style experimental writeup**. Do a full writeup of the experimental procedure, as you would read in *Organometallics* or *JACS*, etc. This includes experimental details, purification conditions, NMR line lists, and additional analytical data. Doing this will make your life *much* easier when paper/thesis writing time comes, and writing them while the procedure is fresh in your mind will make your experimentals more accurate and reproducible.

University of Minnesota – Twin Cities Tonks Group Standard Operating Procedures

By signing below, you indicate that you have read and understand the content of this document.	
Name:	Date: