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Incorporating Catalysis in Inorganic Chemistry 1 Lecture and Laboratory at ONU

Bradley M. Wile
Ohio Northern University, b-wile@onu.edu

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b-wile@onu.edu

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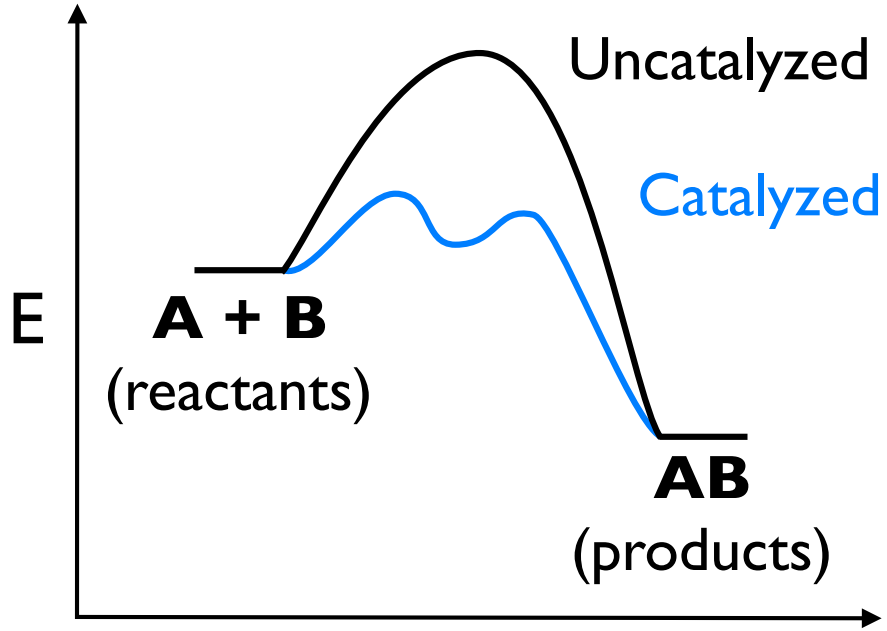
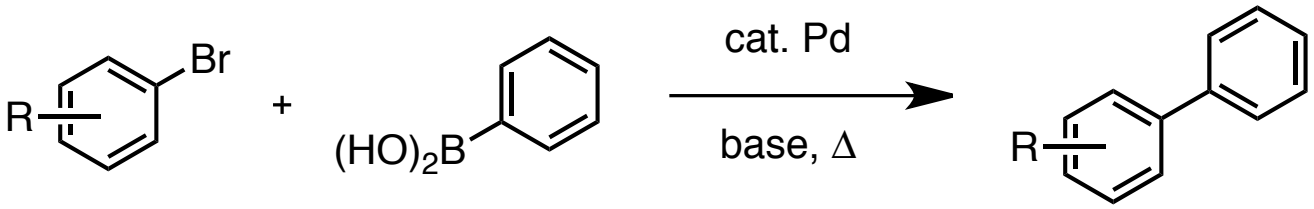
Inorganic Chemistry at ONU

- Chem 3711 (lecture) and separate lab (3751)
  - Juniors & Seniors
  - Exposure to inorganic
  - Descriptive (coordination & materials chemistry)
- Challenge: incorporate catalysis in the existing course framework
  - In class activity and laboratory exercise

Previous Experience with Catalysis

- General Chemistry
  - Not consumed, lowers E_a



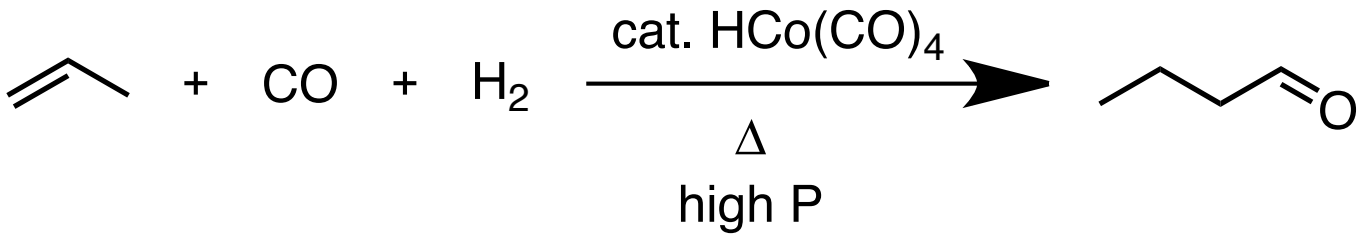


- Organic Chemistry
  - Facilitates an organic transformation

Implementation in the Lecture

- End of the "coordination chemistry" half
  - Discuss transformations of organometallic complexes
  - Mechanism for 1,1-insertion of CO
  - Emphasis on e^- count, metal O.S. and CN, and number of d e^- for each transformation
- Students should connect transformations
  - Review before midterm!

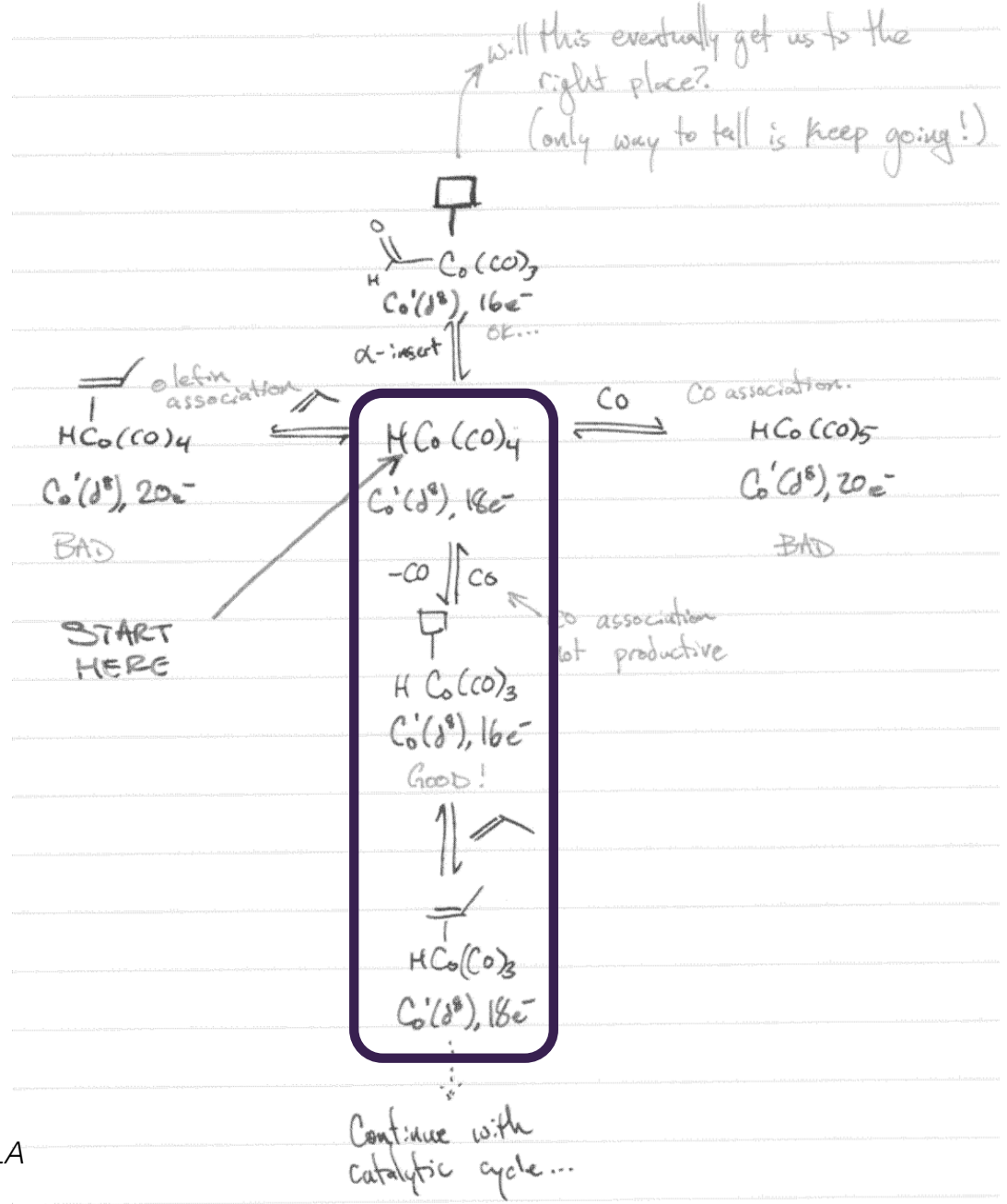
The 'oxo' process



- Propose a catalytic cycle for the above reaction
  - Break class into groups of 3
  - Start at $HCo(CO)_4$ and try things!
  - Check e^- count, metal O.S. etc.
- Instructor circulates to ask questions

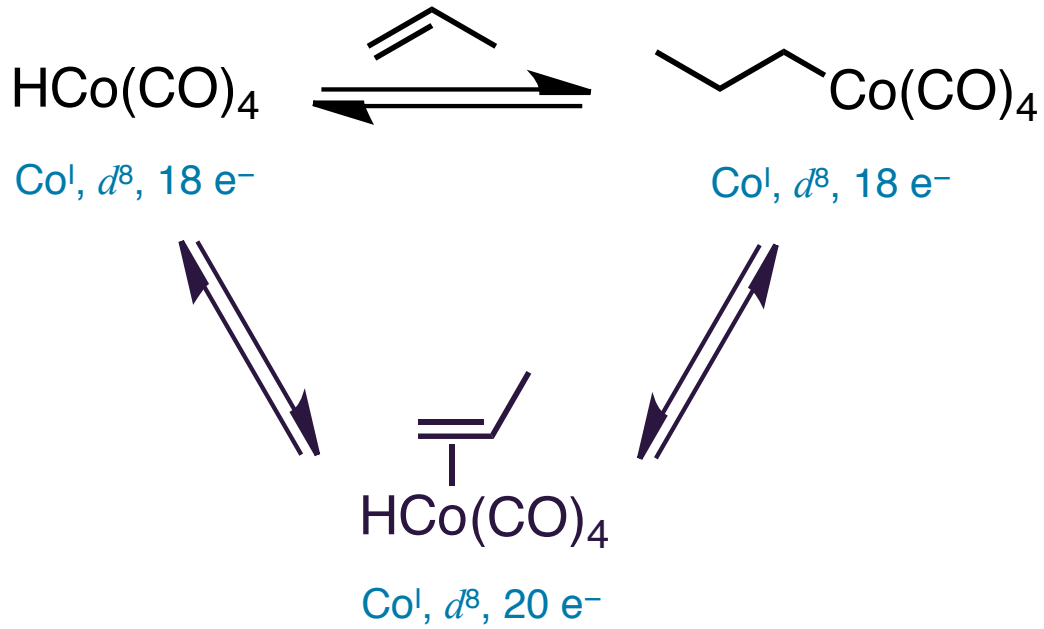
Transformations of the Co Complex

- How many valence e⁻ does this complex contain?
- How did you choose this pathway?
- What assumptions are you making when choosing one path over another?
- How does your olefin find its way over here?

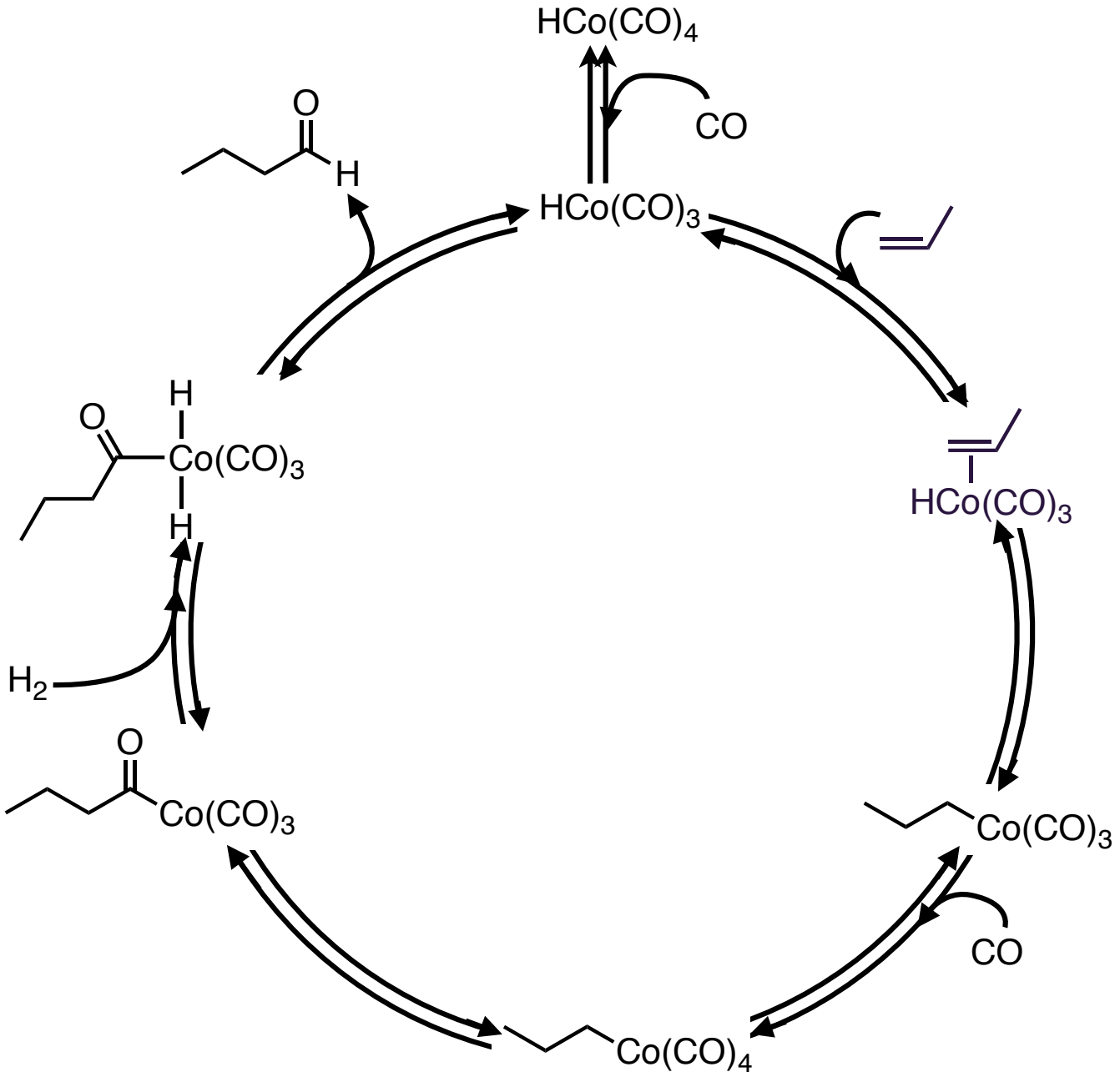


Some Problems

- Unsure about order of individual transformations
- Try to insert olefin directly
  - Miss 20 e⁻ intermediate (BAD)



The Heck-Breslow Cycle

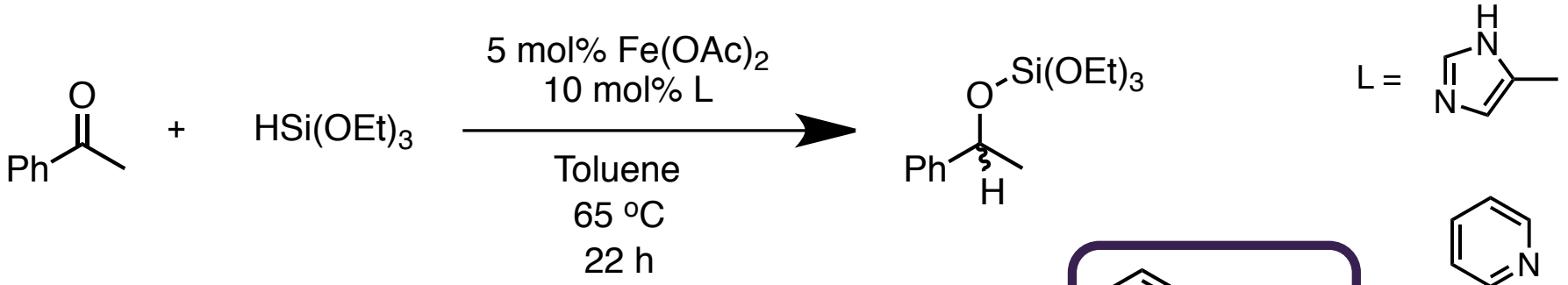


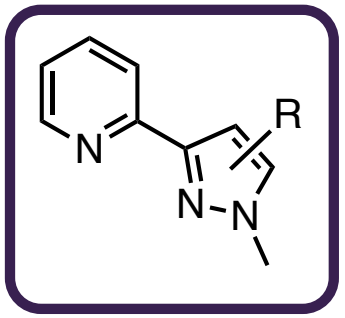
J. Am. Chem. Soc. **1961**, 8*3*, 1961.

Implementation on Lab - Wishlist

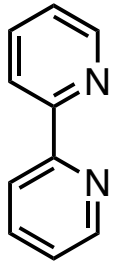
- Introduce catalysis & evaluation of reaction parameters (measurable)
- Introduce students to inert-atmosphere glovebox
  - No other specialized glassware (Schlenk, etc.)
- Does not rely on high P or T for activity
- Does not require column chromatography
- Ideally, 3 hours of 'active lab time'
  - Data analysis to follow

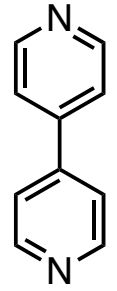
Base Metal Catalysis

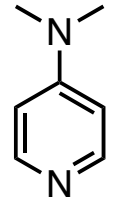




Literature





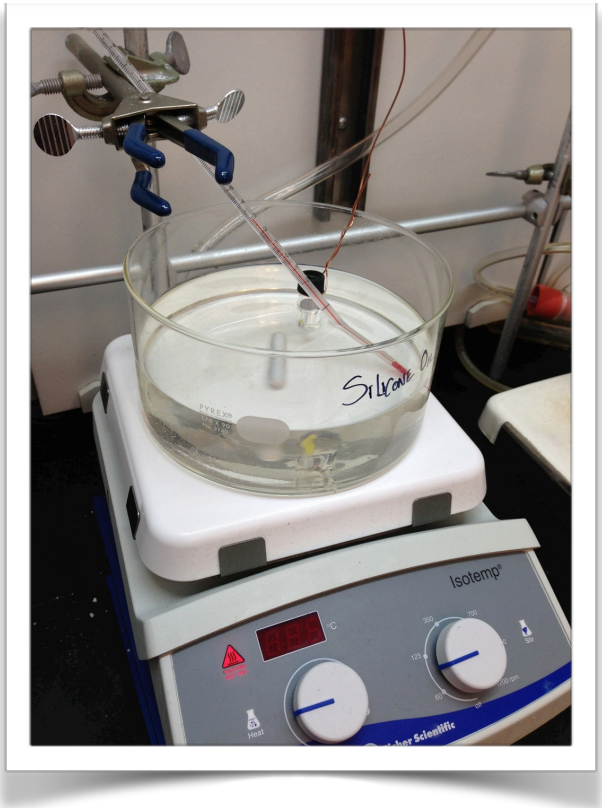


- Iron-catalyzed hydrosilylation of acetophenone
  - Conducted on industrial scale (primarily Pt)
  - Growing interest in base metal catalysis
- Groups each assigned a different ligand (L)

Muller, K.; Schubert, A.; Jozak, T.; Ahrens-Botzong, A.; Schünemann, V.; Thiel, W. R. *ChemCatChem* **2011**, *3*, 887-892.
Nishiyama, H.; Furuta, A. *Chem Commun.* **2007**, 760-762.

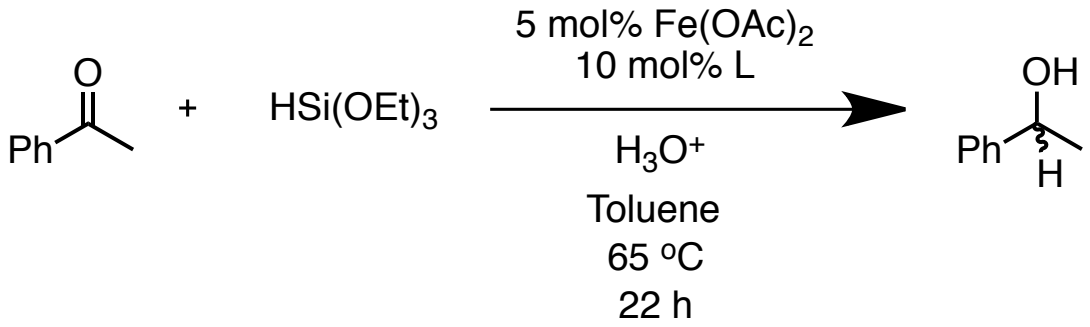
Reaction Conditions

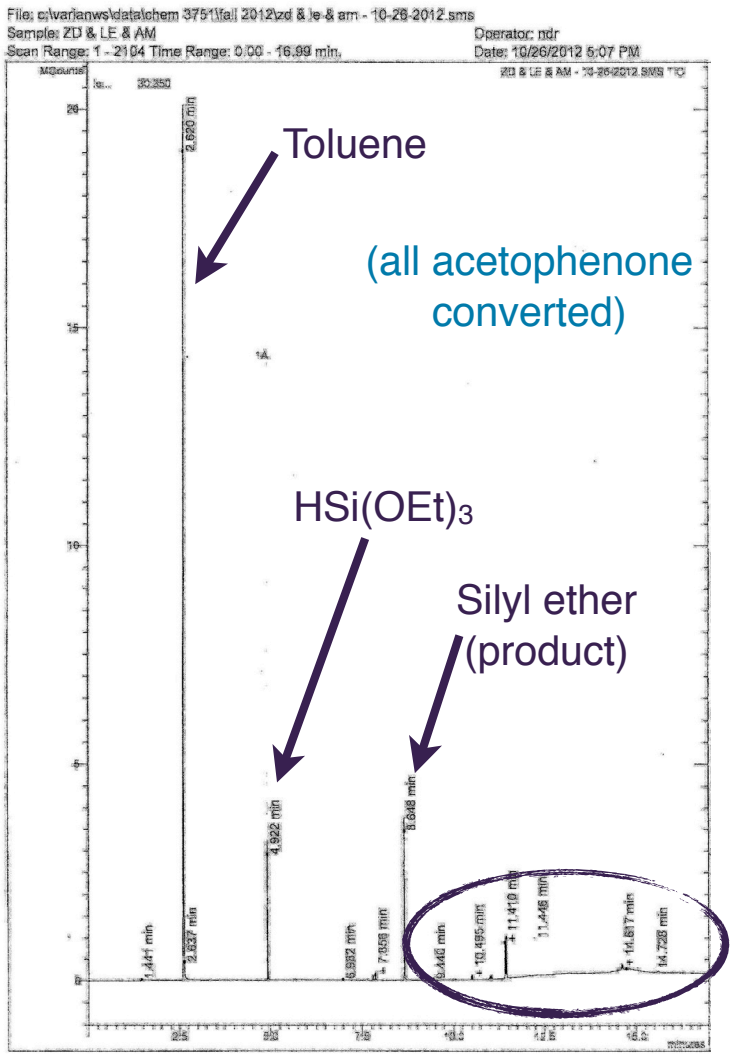
- Prepare samples in glovebox
  - Micropipette for stock solutions, reagents
  - Seal vials & immerse in 65 °C oil bath
  - Filter through silica pad to remove metal



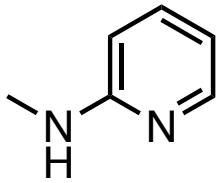
Analysis

- Students had some difficulty assigning GC-MS
  - "Bonus peaks" from ligands, silanes
  - Cleave silyl ether next time
  - Easier analysis by GC





Ligand Suggestions



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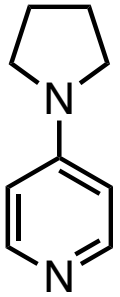

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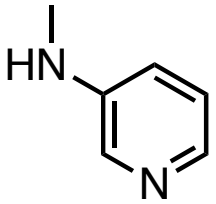

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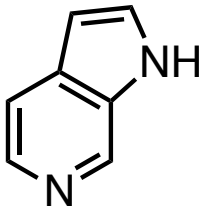

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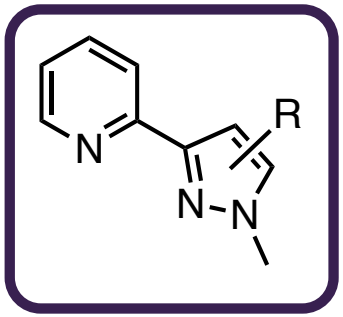

Literature

- Students asked to propose ligands for future use
  - Almost all focussed on distance between N atoms
  - Ongoing project to identify & screen catalysts

Summary

- Catalysis incorporated into Inorganic 1
  - Encouraged active learning & peer discussion
  - Biggest “problem” identified by students is the open ended nature of the tasks
- In-class activity challenged students
  - Good retention of material on final exam
- Lab activity successful, ongoing project
  - Revise expectations based on student feedback
  - Provide background as project develops

Acknowledgements

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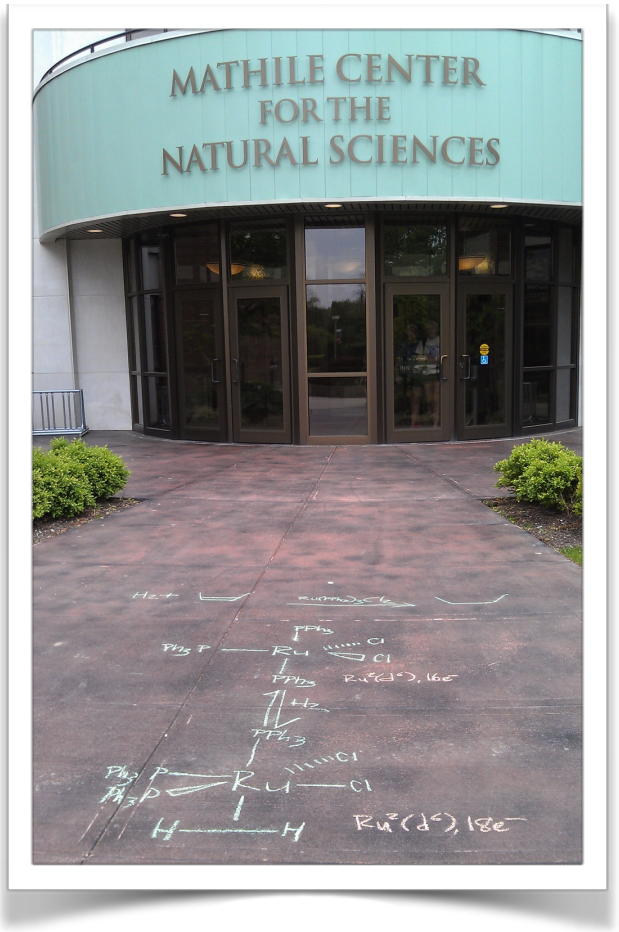

- ONU Department of Chemistry and Biochemistry
- Chem 3711/3751 Students
- Dr. Susan Bates