Lab 1 Prelab: Silicone Polymers

CHEM 411 Week 1, Day 1

Microscale Inorganic Chemistry (MIC) pages 1-17 and 176 - 181
Individual Lab, 2.5+ hours

Learning Objectives:

- Use lab safety protocols and microscale techniques to ensure a safe and well-organized experimental environment
- Demonstrate precision in preparing, handling, and measuring chemicals
- Acquire proficiency in the set up and execution of flash chromatography, emphasizing the practical application of separation techniques
- Record concise experimental procedures, observations, and data collection in a scientific notebook, cultivating appropriate documentation practices

Essential Lab Techniques:

- Use of reflux condenser
- Flash chromatography
- Polymerization

Modifications from *MIC*:

- We will use **5 times the amount of dichlorodimethylsilane** used in the MIC procedure. This has been updated in the Chemical Data Table. Please adjust the amounts of all other reagents by this factor.
- At the beginning of lab your TA will discuss safety and lab expectations. Afterwards, you will **immediately assemble the condenser** then begin experimentation.
- Diethyl ether may be labeled as ether or ethyl ether in the solvent cabinet.
- When running the column, the elute will be contained into a **weighed 20mL scintillation vial**.
- When concentrating the solution, continue to heat until an **oil-like substance** is obtained. If no oil is produced, continue heating until there **is no noticeable change in volume**.
- Once boric acid is added and stirred, the putty will still need some modification. **Knead, roll, squish** (in some fashion, manipulate) the polymer until the correct viscosity is achieved.

I. Pertinent Information:

Before every experiment this semester, a Prelab Assignment will be due at midnight the day before the lab. This is to ensure you are prepared for experimentation. Prelabs will be split into 6 Sections. Section I will contain pertinent information for that experiment. Section II will contain a Chemical Data Table. Section III will be the Synthetic Approach, which is to be filled out in your notebook. Section IV will be Prelab Questions. Section V will be Calculations. Lastly, Section VI will be Characterization. Many labs will also include a Section VII. Observations, which provides guidance on what to write in your lab notebook during and after your experiment.

Before you can begin working in the lab you must complete the necessary Safety Training. If you have not previously completed any safety training, please go to

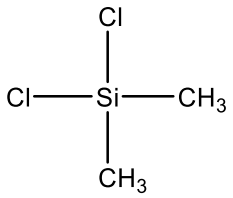
https://ehs.psu.edu/training and complete the **Initial Laboratory and Research Safety Training - University Park.** Upload your certificate to the corresponding Canvas Assignment. This training lasts for 1 year. If you have previously completed this training and it has been less than a year, please upload the **initial certificate** to Canvas. If it has been longer than a year, you must take the **Laboratory and Research Safety Refresher Training** and upload both **Initial** and **Refresher Training Certificates** to Canvas.

We will be using the textbook *Microscale Inorganic Chemistry,* by Zvi Szafran, Ronald M. Pike, and Mono M. Singh for most of our lab experiments. The text will be abbreviated as *MIC*. In addition to instructions for lab experiments, there is a wealth of information 6 chapters that you should read, refer to, and use throughout the semester, including safety, lab equipment, writing lab reports, searching the literature, microscale techniques, and spectroscopy.

For our first experiment we will be synthesizing bouncing putty via polymer chemistry. This lab allows us to explore the strange functionalities of silicone polymers while also connecting and comparing the properties of silicone-based substances to other chemicals in the same group. To prepare for this lab, please read pages 1-17 and experiment 8, on 176-181 in *MIC.* During your reading, take notes on the chemistry and how this reaction works.

II. Chemical Data Table (CDT):

In your lab notebook, you will construct a Chemical Data Table for all experiments. Start with Lab 1. Fill in the table with all the compounds you will be working with, including products, byproducts, and intermediates. You must list the name, structure, molecular weight, melting/decomposition point (for solids), boiling point (for liquids), density, quantity (mLs/mgs), mmols, hazards, and comments (color, smell, extra hazards). Reference the SDSs to help you fill in the chart. An example can be found below. Red X's mean that you don't need to fill in that part of the chart. You can print this page or scan it and attach it to your lab notebook.

Name	Structure	MW	MP/BP	Density	Quantity	mmol	Hazards	Comments
Dichlorodi-methylsilane (75-78-5)		129.06 g/mol	70°C BP	1.064 g/mL	5 mL	41.2	Highly flammable Acute tox. Skin Corr. Eye Dam.	STOT SE 3 Colorless Hydrolyzes
Polydimethyl-Siloxane (63148-62-9)		X			X	X		
Boric Acid (10043-35-5)						X		
Hydrochloric Acid (gas) (7647-01-0)			X		X	X		
Diethyl Ether (60-29-7)						X		

Name	Structure	MW	MP/BP	Density	Quantity	mmol	Hazards	Comments
Sodium Bicarbonate (10%) (144-55-8)						X		
Silica Gel (112926-00-8)	X	X		X	X	X		
Sodium Sulfate, An. (7757-82-6)					X	X		

III. Synthetic Approach:

Write the procedure for each experiment in your lab notebook before entering the lab. This procedure should be concise but thorough enough so that you can set up and run the experiment without bringing your textbook. Drawing schematics is encouraged, especially for techniques you are not familiar with. The procedure should be written in passive past tense. An example is below:

WRONG:

1. I will add 2 mL of diethyl ether to a round bottom flask (RBF).
2. To this, I will add 1 mL of dichlorodimethylsilane.

CORRECT:

1. Added to 10 mL RBF was the following:
   - 2 mL of diethyl ether
   - 1 mL (8.24 mmol) of dichlorodimethylsilane
2. A water condenser was attached to the reaction vessel

For more information on how to correctly set up a lab notebook read **pages 31 - 34 in *MIC*.**

You will not submit scans of your lab notebook with the prelab. Your TA will check your lab notebook pages every class to ensure the synthetic approach is written. Scans of the procedure and lab observations will be turned in at the end of every week in the assignment titled Lab Notebook Scan #.

IV. Prelab Questions:

1. Draw the hydrolysis reaction of dichlorodimethylsilane. Please label the reagents, the products, and the states of matter, and balance the reaction. (Hint: This is a polymerization, so the coefficients may be labeled with x)

2. Compare quartz, glass, and polysiloxane. The base empirical formula may look the same, but the composite structures differ; how do they differ? Draw the arrangement of atoms and the structure for each.

3. Compare SiO_2, CO_2, and PbO_2. Why do these three compounds have very different chemical and physical properties even though they are in the same group?

4. Silicon-oxygen single bonds are typically longer (~155-172 pm) but stronger (~450-460 kJ mol^{-1}) than carbon-oxygen single bonds (~ 143 pm, ~358 kJ mol^{-1}). Why might this be? (Clarification: The manual states that the bonds are shorter than expected but not explicitly shorter than C-O single bonds)

5. Describe the process of equilibration.

6. How do end blockers aid in the polymerization process of silicone polymers?

V. Calculations:

For each experiment there may be several calculations, such as theoretical yield, that will be performed before experimentation. Since we are synthesizing a polymer in this lab, theoretical yield does not need to be calculated. However, to reactivate your prior knowledge of dimensional analysis you will calculate the number of moles of your reagent.

1. Calculate the mmols of dichlorodimethylsilane. Please show all work with units.

VI. Characterization:

Often spectroscopy will be used to characterize the compounds synthesized in this section. Spectroscopy is explained in detail in chapter 6 of *MIC*; please use it as a reference. Before each experiment you may be asked to predict the spectra expected for the complex you are synthesizing. Some examples include NMR shifts and splitting, frequency of IR vibrations, and peaks in the UV-Vis spectrum.

For Lab 1, characterization will be qualitative material testing. Please explain the behavior that is to be expected during testing.

1. Why does bouncing putty bounce when thrown, but flatten when placed on a surface?

2. Why does bouncing putty cleave when torn apart quickly but stretch when pulled slowly?

VII. Observations:

As you perform any experiment in lab, write your observations and your data in your lab notebook. Attach data tables and plots to empty notebook pages. Have your notebook signed by your instructor at the end of each week.

You can add your discussion and analysis of the results after the lab period is done. Do not answer the MIC Questions at the end of the procedures unless noted in the prelab. Some labs will have a separate Results assignment.

Be sure to include the following in your notebook for every lab experiment, along with anything else that is important:

- Changes from the textbook procedure
- Unexpected results
- Color changes
- Gas evolution and precipitate formation
- Amount of time elapsed
- Temperatures, if known
- Tables of data obtained
- Exact mass or volume of reagents
- Mass of isolated products and calculation of percent yield
- Characterization, including melting point, IR, NMR, UV-Vis data, etc.
- Physical or chemical tests performed on reagents and products
- Graphs or plots of data
- Discussion and explanation of data or data analysis