Standard Lab Techniques & Etiquette

Enter this exercise with fresh eyes and an open mind – there is always more to learn! The idea is to *set you up for success today so that you can safely and efficiently perform experiments independently in future weeks*. Get into the habit of following lab protocols to make you a more desirable candidate for working in a research lab!

Keep your lab space neat and organized at all times. You'll learn to handle equipment properly to eliminate or minimize contamination and chemical exposure.

Everything in the lab is organized and maintained by the stockroom staff, all of which are very nice humans. Please be considerate of their time as you work and clean up! As a general rule, **leave the lab as you found it or better**.

A. Measuring Liquids by Pipet and Bulb or Pluringe

A disposable glass pipet is connected to a syringe with rubber bulb or tubing (pluringe). This allows the synthetic chemist to obtain small volumes of liquids with reasonable accuracy and precision. The pipet needs only be inserted into the rubber tubing or bulb enough to stay put; too much and it becomes difficult to remove later. The pluringe or bulb is re-usable as long as it does not come into contact with liquids.

How to avoid pipet contamination:

- **DO NOT turn upside-down when liquid is in the pipet.**
- **Make sure you are not obtaining more than the volume of the pipet itself!**

Only the glass pipet touches liquid. Dispose of any contaminated pipets in the contaminated solid waste in the fume hood. Pipets can be put in the glass waste if the pipet was used to measure water only.

Cross-contamination is avoided when delivering the second portion of water into the second container – **do not touch the tip of the pipet to the water already in the flask.**

Practice transferring portions by pipet with a bulb and both pluringes into separate labeled containers. The bulb is for transfers without specific volume. You can use any volumes to practice with the pluringes – just record them!

Measurement with 1-mL pluringe: _____±_____ mL

Measurement with 3-mL pluringe: _____±_____ mL

What would happen if…

- you quickly pulled back the syringe to obtain liquid? Or if you quickly pushed the syringe to transfer?
- you were transferring a second portion of liquid into a container and dipped the tip of the pipet into the liquid in that container?
- you dispensed more liquid into the pluringe than the capacity of the pipet? What is the volume of a short-stem glass pipet?
- you threw away a glass pipet in the regular trash?

B. Measuring Solids with a Balance

1. Bring a **spatula** or **scoopula** & **labeled container** into which to transfer the solid.
2. **Fold the weigh paper** in half on the diagonal and place on the balance pan
3. **Tare (zero) the balance** – press the tare button and wait for it to read 0.000g
4. **Add the solid** onto the paper and record the mass
5. **Place extra solid** on separate weigh paper, *not back into the original container*
6. **Close the container**
7. **Transfer the solid** into the labeled container (ex. Erlenmeyer flask or beaker)
    - **DO NOT walk around the lab with solid on weigh paper!**
8. **CLEAN**! Brush spilled solids onto **separate weigh paper** then transfer to the **solid waste**.
    - **Dispose of weigh paper** in the trash
    - **Wipe down the counter** with a wet sponge or paper towel

BALANCE PREP & PRACTICE

- The digital balances read in grams (g) so you'll need to convert to milligrams (mg).
- There are 1000 milligrams in every gram (10^3 mg / 1 g) or (1 g / 10^3 mg).
- **Convert 50 mg into grams:** 50 mg = _____ g

- Weigh **approximately 50 mg (± 10 mg)** of your assigned solid.
- Note (± 10 mg) means your measurement can be 10 mg higher or lower than 50 mg.

40 mg = _____ g 60 mg = _____ g

- **Record the exact mass measured**: _____ mg
- **Convert mass (mg) to millimoles (mmol)** using the molecular mass of the solid.

_____ mmol

- **Transfer the solid** into a small beaker or Erlenmeyer flask labeled with chemical name.

C. Separatory Funnel: Liquid-Liquid Extraction

Liquid-liquid extraction is a daily routine for the active synthetic organic chemist. Proper precautions make it easy to avoid chemical exposure and spills. Practice this in the lab with water with your TA's help.

(1) Start with the cap off. Use a glass funnel to carefully transfer 50 mL of distilled water. Place the funnel on a Kim wipe or paper towel in the fume hood. **Bench paper is not a spill mat!**

(2) Put the cap on (twist plastic caps to tighten).

(3) Hold the funnel with two fingers on the cap and the rest of the hand (or hands) holding the funnel. Slowly invert to test the cap is leak-proof.

- If it does leak, first clean up the spill. You would also change your gloves.
- Take off the cap and try again after tightening. It may be necessary to try a different cap.

(4) With a proven leak-proof cap, rotate once more (two inversions total). Hold the funnel upside-down (two fingers on the cap) with the **tip pointing away from your face and into the fume hood. Slowly open the stopcock to release the pressure.**

(5) Demonstrate step **(3)** as many times as necessary until your TA approves your technique: **2-3 inversions, vent, repeat.** A typical extraction step should take between 2-5 minutes, depending on the volume used and potential interactions taking place.

(6) Place the separatory funnel back on the support ring. Take off the cap and slowly drain the liquid into a container without spilling.

Draw a diagram of the separatory funnel set-up in the fume hood and label each component. Make a note of where each of these components are stored in the lab, including the organizational system of the ring stands.

Discussion: What would happen if…

…you forgot to test the separatory funnel's seal with water and instead performed the extraction directly with a reaction mixture?

…you inverted more than twice before venting?

…you vented by taking off the cap instead of opening the stopcock?

…you opened the stopcock too quickly when venting upside down?

Part 1. Infrared (IR) Spectroscopy: Functional Group Identification

**** Watch the IR tutorial videos on Canvas**

Bonds in organic compounds vibrate at frequencies in the IR range (400 – 4000 cm^{-1}). These stretching and bending vibrations are characteristic of the types of bonds within specific functional groups (ex. O-H bonds stretch at a different frequency in an alcohol than in a carboxylic acid). When a sample is exposed to IR radiation, the energy is absorbed by the sample at those specific frequencies and peaks are absorbed in the IR spectrum. As a general rule, the longer the bond, the slower the stretching frequency (wavenumber, cm^{-1}). For example, C-H bonds are longer than O-H bonds because of carbon's larger atomic radius. Consequentially, C-H bonds stretch at a considerably slower frequency (alkanes, 2990 – 2850 cm^{-1}) than O-H bonds (alcohols, 3550 – 3200 cm^{-1}).

Use salt plates (pure NaCl) to support your sample. Sodium chloride does not absorb IR radiation making it an ideal material to hold samples. The *NaCl plates are very fragile* and break easily so handle with care. **Prepare a nujol mull of aspirin by grinding a microspatula tip of solid with 1-2 drops of nujol for 60 seconds (count down!).** The final mull should resemble thin toothpaste. Transfer a small amount onto the plate with a rubber policeman; too much sample will cause the radiation to scatter. Spread the sample evenly by rotating the plates. Check that there is no sample in the instrument and perform a background scan. Place the plates inside the plate holder and obtain the IR spectrum, labeling necessary peaks. *Be considerate of other students and classes waiting to use the instrument (your TA may facilitate a rotation).* Use small amounts of the **acetone saturated with NaCl provided in the IR kit (NOT the GC acetone and definitely not with water!).**

Draw the structure of aspirin below and identify all functional groups.

2. Predict IR stretches before running an IR sample or interpreting a spectrum. **Complete the table below for aspirin using the IR table of values online.** Ignore any stretches expected between 1500 – 1000 cm^{-1}. Then obtain the sample and report the observed wavenumbers in the last column.

Functional Group	Bond Assignment (C=O, N-H, etc.) from IR Table	Expected Wavenumber Range from IR Table	Observed Wavenumber

General IR Spectroscopy Questions

3. Report and explain the difference in stretching frequency of **O-H vs. N-H vs. C-H bonds**.

4. What is the difference between a **saturated and conjugated ketone**? Between a **saturated and conjugated ester?** Draw an example of each. Look up the difference in IR stretching frequencies (IR table of values) and explain.

5. Why is the range between 1500 – 1000 cm^{-1} typically ignored?

6. What would happen if…

…you took the IR of your sample without knowing which peaks to look for?

… to much sample is placed on the salt plate?

NMR Refresher

1. What do you remember about **NMR spectroscopy**? Discuss with your lab mates and explain each of the following NMR terms to the best of your ability, *without using additional resources.*

- **Chemical Equivalence**
- **Chemical Shift**
- **Integration**
- **Splitting**

2. Predict the chemical shifts, integration, and splitting patterns in **3-methylbutanoic acid.** Refer to NMR tables on Canvas and/or predict its spectrum using the online ^{1}H and ^{13}C NMR predictor tool: nmrdb.org.

3. Look up the structure of any drug, medicine, or organic molecule that interests you. Draw its structure using ModelAR, the super cool molecular modeling app. **Take a screenshot or screen recording of this molecule and post in the Canvas Chem Apps Discussion.** Then, draw the structure in nmrdb.org. Sketch the spectra below and assign each signal to a H or C on the molecule.