Technique Week

The first week of lab this semester will be a lab practical of sorts. You will be asked to carry out a series of techniques independently (this means you need to try to ask as few questions as possible –if possible work totally independently) in order to review and to ensure that you have mastered basic techniques and will be able to incorporate them relatively seamlessly into the more complex second semester. You will also be introduced to a new instrument. We have two new Infrared Spectrometers. While working with the IR’s, it is OK to ask questions. We will actually be showing you how to use them and then later in the lab you will try to run a spectrum on your own.

It is important that you do not discuss your experience with your peers between labs and it is important that you not tell other students how to carry out the procedures. Generally, during lab, you should allow your peers to learn by experience. It is an important part of your and their development. Though you may never separate layers again or run an IR in your distant future, the mechanical experience, skills, confidence, problem solving skills you gain are relevant.

The class will be divided in half, into two groups lockers 1-18 and 19-30.

**Lockers 1-18** will first have to accomplish the following skills in the first third of the lab period, the first 1-1.5 hours.

1. Your group will be divided in two half of you will be first asked to build a reflux apparatus with a trap, a fractional distillation apparatus or a simple distillation apparatus.

2. The other half of you will be asked to prepare either a liquid or solid sample for IR analysis. Your instructor or TA will show you how to run the IR, but you must prepare the sample.

3. The two mini-groups will switch responsibilities after about thirty or forty minutes.

**Lockers 19-30** This will take about two thirds of the lab (2-2.5 hours)

1. You will be asked to separate a two phase system and determine which layer is organic and which is aqueous using solubility tests. The organic layer will contain an unknown solid material. The aqueous layer will also contain solid material. As you will see on the bottles that are provided, you must shake the bottle, pour out 30 mL and you must have a reasonable amount of each layer before proceeding.

2. The determination of the nature of the layers must be accomplished using solubility tests. Once the layers are separated, the organic layer has to be dried and the solvent removed by rotary evaporation.
3. The resulting solid material has to be recrystallized using an ethanol/water system (you need to review how to do this like everything – this was done in the ).

4. When the material has been isolated and is reasonably dry, you must prepare a solid IR sample and your TA or instructor will show you how to operate the new IR spectrometers.

Upon completion of the assigned tasks, the groups will switch responsibilities. Lockers 1-18 will move on to the separation/recrystallization procedures and Lockers 19-30 will build an apparatus.

It is your responsibility before this lab to go back and review all the relevant material. This means, go back to the first semester lab manual and watch all videos that are relevant and read all relevant labs again. You can also use other lab manuals or videos on the web to prepare. The point is you want to try to have everything diagramed, outlined and ready to go and then try to do what you are supposed to do independently and to hopefully understand it while you are doing it.

Again, you will not be treated in a negative manner if you need to ask a question, but it is important to reach inside and try to work on your own. Also note that safety rules have to be followed and working efficiently is somewhat important.

The grades for this exercise will be either check, check minus or check plus. A check – will translate into an 85 percent, a check into 90 percent and a check plus, a 95. Mainly, the entire class will be given some comments about

I feel that lab is like driving a car. You can drive and constantly ask for directions and never really learn how to go from point A to point B. It is time to learn how to drive independently if you just force yourself through things on your own.