6. Common Laboratory Techniques

This chapter explains the proper manner in which to carry out rudimentary chemistry laboratory techniques. More specialized techniques associated with particular analyses are covered in the on-line notes associated with the experiment in which the technique is used.

1. Measuring Mass

High precision and accuracy electronic balances are used to determine mass in organic lab. Each of these balances costs $1,000-$3,000. Treat them with care. Accuracy and precision will be lost if poor weigh techniques are used. All of the balances in the organic laboratories measure to 3 or 4 decimal places. These balances measure mass to one one-thousandth (1/1000) or one ten thousandth (1/10,000) of a gram. Always write down all digits a balance provides. Always record mass directly in laboratory notebook.

Never place a chemical directly on a balance pan. Always place chemical in a weigh boat, on weigh paper, or within another weighing vessel.

Often a target mass is stated in the procedure. Unless specified otherwise, this does not mean to obtain that exact target mass. For example if the experimental procedure states “Weigh approximately 0.4 g of impure benzoin.” This means that a mass somewhere close to 0.4 grams should be obtained, and the exact mass displayed on the balance should be recorded. This does not mean to measure out exactly 0.4000 g. It also does not mean to measure out 0.4169 gram and only record 0.4 g. It does mean to obtain a mass somewhat close to 0.4 g and to measure it precisely. The mass measured may be 0.3764 or 0.4682 g or some other reasonable value. The entire mass displayed should be recorded.

Two slightly different methods may be used to weigh a chemical, either the mass of the weigh paper (or weigh boat or flask) can be subtracted (weigh by difference), or the balance can be tared (zeroed) with the weigh paper. Each weigh boat and weigh paper has a slightly different mass, so the mass of the exact paper used must be taken.

Weighing by Difference Method. Close doors to balance and allow reading to stabilize. Press the tare button. The reading should now read 0.000 g (or 0.0000 g). Place a piece of weigh paper (or weigh boat or weigh flask) on the balance. Close balance doors and allow reading to stabilize. If the balance does not read g but N, that signifies Newtons (unit of force) and not grams (unit of mass) is being measured. If this is the case, press the mode button until “g” returns. Record this mass directly in lab notebook. Record all digits. Carefully add chemical to paper. Close doors and allow balance to stabilize. Record this mass directly in notebook. The mass of the chemical will be the second mass minus the first mass; or the mass of the chemical plus paper, minus the mass of the paper.
Tare Method Place a piece of weigh paper on balance. Close doors to balance and allow reading to stabilize. Press the tare button. The reading should now read 0.000 g (or 0.0000 g). Carefully add chemical to paper. Close doors and allow balance to stabilize. Record this mass directly in notebook. This value will be the mass of the chemical.

If chemicals are spilled on the balance pan or within the enclosure, sweep away gently using large ‘paint brush’ adjacent to each balance. Scoop up spilled chemical from counter and place in appropriate waste container.

2. Measuring Volume and Reading Scales

Measuring volumes of liquids is a common action needed within most experiments. Poor measurement techniques may result in poor results. It is important to know when it is critical to read exact volumes, and when approximate volumes are sufficient. It is rarely important to acquire exact target volumes in organic lab. The one exception to this is when making solutions with very well defined concentrations.

If you are heating water to form a hot water bath, the volume of water used does not have to be measured accurately. An approximate volume is sufficient. A volume of 50 mL or 90mL may be used and recorded. If the exact volume is not known this can be recorded as ~50mL. This means “approximately 50 mL”. The same goes for most solvents and wash solutions.

Often a target volume of a reagent will be stated in the procedure. Unless expressly stated otherwise, this does not mean that the exact target volume should be obtained. For example if the experimental procedure states “Obtain approximately 2 mL of 2-methyl-2-butanol. Record precise volume.” This means that a volume somewhere close to 2 mL should be obtained, and the precise volume obtained should be recorded. This does not mean to measure out exactly 2.00 mL. Measuring out exact target volumes when not necessary only serves to waste time (and is a telltale sign of an inexperienced chemist). The statement does mean to obtain a volume around 2 mL but measure it precisely. The volume measured may be 1.74 or 2.46 mL or some other reasonable value. The volume should be measured as accurately and precisely as the equipment will allow. This volume should be recorded directly in the laboratory notebook.

When reading scales, always measure to one digit past the value of the smallest graduation. This digit will be approximated and hence this digit has the least significance and contains the most error. Some examples follow.

The image to the left portrays a graduated cylinder. The ten mL lines are numbered and the 1 mL lines (graduations) are indicated. A precise volume measurement in this should be read to one digit past the smallest graduation or 1 mL. This means a volume of 1/10 of one mL or XX.X mL may be determined with this instrument. The correct volume measurement of this image is 36.1 or 36.2 mL.
The next image indicates another graduated cylinder. Each milliliter is numbered and the smallest graduations represent 1/10 mL or 0.1mL. Again the most precise measurement should be read to one digit past the smallest graduation of 1/10 of 1/10mL. This is 1/100 of a mL or X.XX mL. The correct volume measurement of this image is 3.66 or 3.67 or 3.68 mL. The last digit is a best approximation or a best guess.

The item to the left is part of a syringe. The entire syringe contains 1 mL. The large markings are tenths of a milliliter, the smallest graduation are hundredths of milliliters. The volume should be read between the smallest markings. In theory the volume should be able to be read to a tenth of one hundredth or 1/1000 of a milliliter, X.XXX mL. In practice it is often very difficult to interpolate between the smallest markings, and often the plunger is moved slightly to align exactly with one of the smallest lines and a value of X.XX0 is taken. The measurement is taken from the leading edge of the ring which is in contact with the inside of the syringe wall. This ring is indicated by an arrow in the image.

The last image is a portion of a ruler. If a distance is being measured, it is easiest to move the ruler until the “0” is aligned with one end of the measurement. The large markings are 1 cm. The smallest graduations are 1/10 cm. Again the measurement should be 1/10 of the smallest graduation. The distance should be read to 1/10 of 1/10 cm or 1/100 cm or X.XX cm. The correct reading is 1.94 or 1.95 or 1.96 cm. Again the last digit is a best approximation or best guess.

When reading a volume in a graduated cylinder or volumetric flask, place your eyes even with the level of the liquid. This will reduce the parallax error. Align the bottom of the meniscus to the printed scale to determine the volume. It can be easier to read the meniscus by placing a notecard or sheet of paper with a black line behind the cylinder or flask.

4. Cleaning Glassware

Adjacent to each sink in the organic laboratories is a rack of wash bottles. The rack should contain wash bottles of deionized water (D.I.), soap solution, and acetone. The acetone bottle normally has a red top to indicate a flammable material. Also adjacent to each sink is a pump dispenser of hand soap. This is for washing human hands and should not be used for washing glassware. Within the wash bottle rack is a variety of glassware brushes.

When an experiment is finished, the waste material needs to be placed in the appropriate waste container. Additional information about how to determine the correct container may be found in the safety chapter. Empty as
much of the liquid or solid into the waste container as possible. If a particularly hazardous material is being disposed of (not the norm in organic labs), then be sure to rinse the flask with a bit of appropriate solvent and also place this wash solution into the waste container. The quantities of chemicals used in organic lab are intentionally kept small to reduce the waste and environmental hazard. Minute amounts of organic waste can be broken down by the microbes contained within the sewage treatment plant, and hence can be disposed of down the sink.

Dirty glassware in which the bulk of the chemical has been removed may be cleaned by spraying soap (wash bottle) inside and scrubbing with an appropriate selected brush. Rinse with tap water, then rinse with deionized water to eliminate any spot causing mineral deposits. If organic material remains after washing with soap and water, spray acetone within container and again scrub. There is no need to rinse again with water after acetone. Acetone, ethanol and ethyl acetate are organic wash solvents that can be decomposed in reasonable amounts by sewage treatment plant microbes, therefore the small amount of acetone wash liquid used may be poured down drain.

Many organic materials have limited solubility in water but a large solubility in acetone. Acetone removes organic residue that will not easily come off with soap and water. Acetone is the major component of nail polish and nail polish remover. If one wants to make sure polished nails are not damaged while washing glassware with acetone, be sure to wear impermeable gloves.

Replace damp glassware back in drawer. It is not necessary to dry glassware with paper towels before putting away. Acetone rinsed glassware will dry rapidly. Glassware rinsed with water should air dry before 24 hours has passed and the next class needs to use it.

Ensure all of your glassware has been thoroughly cleaned and replaced in your drawer before locking your drawer and departing. Any dirty glassware found in a drawer may be replaced at the stockroom window, and the prior person having that drawer will be fined half the price of the object. Leave your glassware clean and well organized for the next student to use or you may find charges appearing on your XU account.

If you find dirty glassware upon opening your drawer, take offending items to the chemistry stockroom for replacement. Alternatively you may leave a message for the prior student in the drawer asking him or her to be more responsible and considerate.

6. Cleaning Workspace

Be sure to leave workspace clean and tidy and ready for the student in the next class to work safely. Clean up all chemicals on benches by the appropriate method. Solid chemicals can be swept up using a small dust pan and deposited in the appropriate solid chemical waste container. Aqueous solutions can be wiped up with sponge. Strong acids or bases should first be
neutralized and then wiped up with wet sponges. If you are unsure how to safely clean up a chemical, ask your instructor for clarification.

Wipe down work area with damp sponge before leaving. Place all trash in waste container. Deposit any broken glass to broken glass container. Any abandoned glassware should be placed in the “Extra Glassware” cabinet.

7. Cleaning Self

The second to last thing each of us should do after finishing an experiment and before departing the laboratory is to wash hands thoroughly. Most chemicals have a dramatic increase in toxic effects when ingested. Chemical residue on hands can easily be inadvertently transferred to one’s mouth by not washing hands thoroughly after handling chemicals. Adjacent to each sink is a pump dispenser of hand soap. Wash hands thoroughly, with plenty of soap and water (~20 seconds or the amount of time it takes to sing the happy birthday song twice) after each lab. The very last thing each of us should do before leaving lab is to remove safety goggles.

Revised April 30, 2013