

Lab meetings begin on **January 5th, 2009**. Lab methods are posted on the chemistry web site, under the CH118 course page, where you will see the link to Laboratory. You are expected to bring a printed version of the Lab Method with you each day. Prepare before coming to lab by completing the pre-lab assignment given at the start of each lab method document. Any techniques, concepts, or vocabulary that isn't understood may be asked about during the introductory lecture.

Laboratory Lecture: Each day, at the official start of lab time, there will be a brief lecture of important information for the experimental method of the day. **Be prompt** to hear this essential information. Lateness carries a penalty. You will lose points if you arrive late to lab. Please leave backpacks, cups, food, and other personal belongings outside of lab; they are not allowed in lab for safety reasons.

Eye Protection: You will be provided with safety glasses during the first lab meeting. You are expected to **wear them** each day throughout the scheduled lab meeting, including time spent writing in lab books or doing computer work. At the end of each day you can store your glasses in an assigned location.

Missed Labs: You are expected to attend your assigned lab and to complete every experiment in order to meet the CH118 course requirements. If you are sick, go to the Health Center. You can ask the Health Center to contact Lisa Miller by phone at x. 5752. Arrangements to complete the lab work will be made after you are well. Absences must be documented to be verified. Lab space is limited; therefore if you cannot attend your lab you must make **prior** arrangements. Unexcused absences will earn a grade of zero to 50 % on work completed. This grade will be determined by all CH118 instructors. For some experiments it will not be possible to make-up work as some types of evidence samples do not last indefinitely. In such a case, the unexcused absence will earn a grade of zero on all related work.

Grading: All written work accumulated in each investigator's portfolio shall count towards the final lab grade. The lab related expectations (listed below) completed will be evaluated to earn up to **150 points** towards your class grade. Any work not turned in at the conclusion of lab will be assessed a 50% penalty per 24 hours. Academic dishonesty will not be tolerated. If any submitted work is found to contain copied (electronic, written, or other form) work from any other person, then all participants shall receive a grade of ZERO and the people involved will be reported to Prof. Millard, the Chemistry Department Chairperson, and the Dean of Students.

Expectations: to be met on a daily basis

- All Colby Forensic Investigators (CFI) need to arrive **on time** and bring the completed pre-lab assignment to be collected/checked at the door.
- Each CFI needs to use appropriate safety precautions while at work.
- Working in assigned teams, CFI will complete scheduled lab experiment methods.
- Each CFI needs to clean up work area used and return any materials borrowed.
- Each CFI will record all experimental work & observations in her/his Lab Notebook.
- Follow given guidelines outlined under **Laboratory Notebook Essentials**.

- Each CFI will undergo two Lab Notebook audits (first day of lab, the last lab).
- Each CFI shall show all work to answer Post –Lab Questions and hand-in at end of day to Lab Supervisor.
- Using provided **Daily Summary Report** form, complete and give to Lab Supervisor at end of day.
- Drop off Lab Notebook at end of each workday for security storage purposes.

Laboratory Notebook Essentials: Please buy a pre-numbered, **hard-covered**, composition book to bring to the first lab to use as your laboratory notebook. On the inside front cover put your name and lab section. If you don't purchase a lab notebook with numbers, simply number the pages in your notebook before using it (it would be okay to number just the front side of each page as 1, 3, 5, etc.). Carbon paper notebooks are not acceptable.

Your notebook serves as a record and proof of each experiment that you do in the laboratory, whether it seemed to work or not. Because your notebook is to be legibly filled in as you do each experiment you are not allowed to write first on a piece of scrap paper and then recopy it into your notebook later. The notebook is not expected to be perfect and neat. To ensure the credibility of your scientific work, make lab notebook entries with a non-erasable ballpoint pen (water impervious). When you make a mistake, put a straight single line or **X** through it (NOT multiple lines that make the error unreadable), then initial the cross-out. Correctional fluids such as White Out are not allowed and it is not acceptable to rip out or leave blank pages. All references must be duly noted. Be sure that you or another person could exactly duplicate your experiment from what is written in your notebook for that experiment!

The reason scientific notebooks are kept this way is that your notebook is your legal proof of your experimental work and results. In fact, in forensic laboratories each page is signed and dated by the experimenter and a witness everyday. Any entries that look suspicious may be suspected of being added or changed at a later date. These entries would be scrutinized in court and possibly jeopardize a case.

The first page of your notebook will be for the **Table of Contents**. Write this header and then make two columns, the first will be Title and the next column will be Pages.

You will use this notebook each day that you work in lab. Each day of work will begin with a **Title**, the **Date**, your **Partner** name, and **Case number** for given evidence samples. Remember that if you skip a page you need to draw an X through it, then sign the page.

Example Title: Experiment Method #1 Mass and Volume

Example Date: January 5, 2009

Example Partner: Smar Typants

Example Case #: 123-Rinky Dink Inks

This section will be followed by an **Objective**. The **Objective** will describe your purpose for the experimental work in your own words, and can also pose a hypothesis to be tested. Sometimes you will have more than one objective. Be sure to include the means by which an analysis is

being made (what instrumentation is used, what method or technique is utilized). What is really important is that this section be concise, complete and always written in YOUR own words.

Example Objective: To determine the type of ink found on Evidence Sample #13-B, using paper chromatography and an FT-IR spectrophotometer.

The objective will be followed by the step-by step account of what you did during the experiment. This is called the **Procedure** and will include methods used, and any variations you made to the method given to you in the lab handout being used.

Example Procedure: Step 1. Weigh 3.000 g of sand/drug mixture on analytical balance #4.
 Step 2. Heat measured mixture in 20.0 mL hot deionized water, on hot plate for 10 minutes.
 Step 3. Vacuum filter dissolved solids from remaining sand.
 Step 4. Dry sand in Drying Oven at 120 degrees Celsius for one hour, (oven Model #23-8A) and weigh when cooled.

The **Data/Results** section follows the procedure. This section would include, any information about the evidence being worked on. For all evidence samples, record 1) type of package received, 2) markings on package, 3) observation of contents, and 4) descriptive information from the forensic supervisor concerning the evidence. As you process evidence, make sure that observations are recorded in detail. Observations should be written down as you proceed through lab work, not as an afterthought. Data should be recorded with appropriate units and include any contextual information necessary to easily identify what was measured. For example, if you weighed a powder substance, make sure you say what color it is, what shape the crystals are, and if the powder is homogeneous with respect to crystal size. The analytical balance number should be included for each mass recorded. In addition to data and observations, also include any graphs created and instrument generated data. Include details of the instrument used (e.g. Hewlett Packard 1050; gas chromatography instrument with a Carbowax column), as well as any other applicable results. You are required to include copies of all graphs, printouts, etc. in your notebook. All attachments must be attached with glue or tape (no staples) and fully open (no folded inserts, no layered inserts, no paper edges sticking out). It is not acceptable to reference a partner's notebook for any material, each forensic investigator is responsible for providing all data obtained in her/his own notebook.

Example Data/Results:

Evidence Description: Ballard Case #3115 exhibit 2a, left shoe of victim, white canvas Docker style, bloodstain-spattered pattern on heel, 11 Jan 2007

Balance # 5 used to weigh shoe. Mass of shoe= 23.228g

Observation: some green glass particles wedged into tread

Bloodstains respond positively to human blood type testing

Shoe treads print taken using black powder method, see results attached on page 41 of notebook

Once the data has been collected the possibility of error in the results needs to be presented in the section labeled **Discussion of Sources of Error**. This discussion shall address at least two possible sources of experimental error. As investigators we are more concerned with random

error (not the same, not reproducible, small), not so much concerned with systematic error (reproducible and same size each time). You need to explain how the reported result(s) would be influenced by these specific sources of experimental error. State the final results along with error analysis when applicable (see the following attachment titled Error Analysis in the Chemistry Laboratory).

Example of Discussion of Sources of Error: There were 53 grams of cocaine recovered from the original 200 gram “white powder” evidence sample. The low percent yield may be in part due to the experimental error of not removing enough of the cocaine crystals from the sandy mixture during the decanting procedure. This lowered the amount of final product and thus lowered the final % yield. Another source of loss of final product may have been in the multiple transfer steps from weigh paper to beaker to flask to filter. Loss of filtrate would have also lowered the amount of final product recovered and thus lowered the final % yield.

Daily Summary Report: a hand written summation made on a provided official report sheet, of what you the laboratory investigator tried to accomplish (should begin by addressing your originally stated Objective), followed by the means used to achieve your Objective (method, technique, and or instrumentation used) and includes the status of work at your time of departure from lab (any key findings & basis of said findings). If quality control samples were included in an analysis the expected versus final results shall be reported. You may reference any relevant graphs or printouts in your lab notebook, therefore eliminating the need to attach duplicate copies.

This brief summary (**less** than one side of a page) will serve to inform your Colby Forensic Investigation laboratory supervisor of what you’ve accomplished on a daily basis. Your report is for use within the investigation department, and shall be returned to you with applicable comments. Upon being return to you, attach the Daily Summary Report to your lab notebook (can refer to Laboratory Notebook Essentials, above, for proper way to make attachments).

Error Analysis in the Chemistry Laboratory¹

The goal in the Chemistry laboratory is to obtain reliable results while realizing that there are errors inherent in any laboratory technique. Some laboratory errors are more obvious than others. Replication of a particular experiment allows an analysis of the reproducibility (precision) of a measurement, while using different methods to perform the same measurement allows a gauge of the truth of the data (accuracy).

There are two types of experimental error: *systematic* error and *random* error. Systematic error arises from a flaw in experimental design or equipment and can be detected and corrected. This type of error leads to inaccurate measurements of the true value. On the other hand, random error is always present and cannot be corrected. An example of random error is that which arises from reading a burette, which is somewhat subjective and therefore varies with the person making the reading. This type of error impacts the precision, or reproducibility, of a measurement. The goal in a chemistry experiment is to eliminate systematic error and minimize random error to obtain a high degree of both accuracy and precision.

Expression of experimental results is best done after replicate trials that report the average of the measurements (the mean) and the size of the uncertainty (the standard deviation). Both are easily calculated by such programs as Excel. Because we will assume that any systematic errors have been detected and corrected, the standard deviation reflects the precision of the measurements. Whenever possible, you should provide a quantitative estimate of the precision of your measurements by reporting both the mean and standard deviation of your data. Sometimes it is most useful to report the relative uncertainty, which compares the size of the absolute uncertainty (the standard deviation) to the size of the associated measurements (the mean). The percent relative uncertainty can be calculated as follows:

$$\text{percent relative uncertainty} = 100 \times (\text{standard deviation}/\text{mean})$$

You should then reflect upon and discuss the possible sources of random error in your measurements that contribute to the observed random error. Sources of random error will differ depending on the specific experimental techniques used. Some examples might include reading a burette, the error tolerance for laboratory balances, etc. Sources of random error do not include calculational error (a systematic error that can be corrected), mistakes in making solutions (also a systematic error), or your lab partner (who might be saying the same thing about you!).

¹ Material herein taken from *Quantitative Chemical Analysis, 5th Edition* (1999) by Daniel C. Harris and *Chemistry, 5th Edition* (2000) by Steven S. Zumdahl and Susan A. Zumdahl.

CHEMISTRY LABORATORY SAFETY

You'll need to prepare for your lab time in advance by doing the following:

1. **Read** the experiment before coming to lab. The more prepared you are, the safer and more efficient you will be in lab. Completion of the pre-lab assignment will help you understand what you will be doing before your scheduled lab.
2. Think about what you need to wear to lab before you come to your lab. **No sandals** are allowed because your feet need a protective cover over them in case of spills or excess heat. Arrange long hair so that it won't be accidentally burned, pulled, or fall into chemical containers. **The wearing of contact lenses in the lab is forbidden**, even when wearing safety goggles. Contact lenses do not provide adequate eye protection, and in some cases may complicate or create an emergency situation. For example, caustic liquids splashed into the eyes may form a liquid layer beneath the contact lens; the eyewash will not rinse out the caustic liquid adequately unless the lens is removed. Also, some organic compounds can actually dissolve or be absorbed by the contact lens.
3. All coats and backpacks are to be left in the hallway, outside of the lab.
4. Drinks, food, candy, and **gum** are not allowed in the lab.

You'll be shown all of the safety features available in your work area. You will then be required to draw a sketch in your lab notebook of your laboratory that indicates the location of the following features:

shower	exits	eyewash stations
fire extinguishers	first-aid kit	phone
fire blanket	glass box	MSDS (material safety data sheets)

The lab instructor will indicate when you may begin experimentation. You may only work in the lab with authorized supervision.

The chemicals you will need for each lab assignment will be made available in your work area as needed. Several of the substances used in this laboratory are hazardous, but when used properly and with caution, exposure is avoided. All chemicals will be marked with appropriate hazard labels. If you follow the directions given during lab, the chemicals pose no threat to your health.

In the case of an accident, **report any spills or breakage** to your lab supervisor at once, so that the appropriate safety measures can be made. **DO NOT** attempt any chemical clean-up on your own.

In the case of a **fire** involving your clothing use the procedure called **STOP-DROP-and ROLL**. **STOP** what you are doing; **DROP** to the floor, and then **ROLL** over and over to extinguish the flames. Do not run to the fire blanket or safety shower if your clothes are on fire, **STOP-DROP-and ROLL** first. Someone else will get a fire blanket to further assist you.

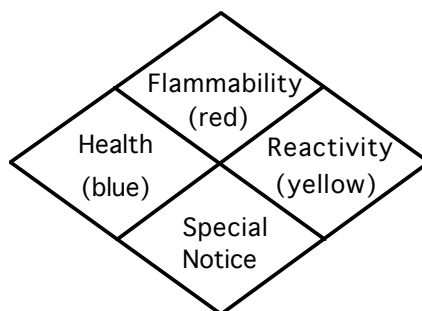
SAFETY RULES for everyone working in the chemistry lab.

1. **Put on safety glasses** as soon as you come into the room. They have to be worn the entire duration of lab! Even when you have finished your experiment, keep them on. Other people around you will still be working.
2. Keep your face well away from reactions. Never watch heating solutions from above; look instead from the sides of the container.
3. Avoid breathing dusts and vapors. Keep powders in covered beakers and work with volatile liquids in the fume hood.
4. Wash away solutions splashed onto your skin by flushing with lots of water and notify your instructor. Large corrosive spills on clothing may require use of the safety shower. In such a case, ask for help. The instructor will assist with all contaminated clothing (while you are under the shower). Go to the nearest eye wash station if solution has been splashed into your eyes. Rinse for 10-15 minutes.
5. Label any container to which you added chemicals.
6. Transfer reagents needed for your own use into a container to measure from, taking only approximately what you need. Never return reagents taken out of the original container back into the original source.
7. All left-over solutions will be collected in appropriately labeled containers provided for you in the lab. It is against the law to flush many substances down the drain. Ask if you don't know where to put your left-overs.
8. Broken glass gets put into a Glass Box.
9. When your work is completed, **clean your work area**. **Clean used glassware** before putting it away. **Return borrowed items** in better condition than originally found. **Wash your hands** before leaving lab.
10. Use common sense. Practical jokes, unnecessary noise, loud music downloaded from the WEB, etc. are not acceptable.

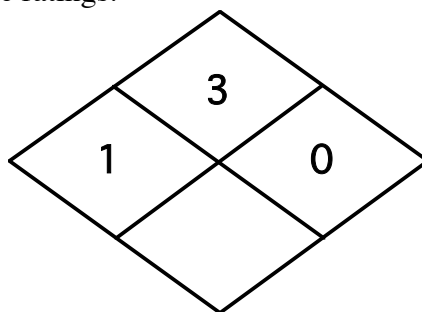
MATERIAL SAFETY DATA SHEETS

The Hazard Communication Standard gives workers the right to know the hazards to which they are exposed. In compliance with this standard, Material Safety Data Sheets (**MSDS**) are kept in lab for chemicals you will be handling. Each MSDS sheet includes the name of the chemical (or components of a mixture), common names, physical and chemical characteristics, fire and explosion hazard data, reactivity data, health hazards and precautions for safe handling of that substance. A more comprehensive collection of MSDS for all chemicals located in this building is kept on the third floor corridor of Keyes.

Stock bottles of chemicals also contain safety information. This includes chemical name, manufacturer, health, flammability and reactivity hazards. The label also includes specific hazards unique to that chemical (oxidizer, water reactive, etc.). The diamond on some bottles indicates these hazards also:



The numerals in the boxes of the diamond indicate the severity of the hazard with “0” indicating little or no hazard and “4” indicating severe hazard. For example, acetone (the major component in nail polish remover) has the ratings:



That is, the health rating of 1 means acetone can cause some irritation, but only minor residual injury. The fire rating of 3 means acetone is flammable. The reactivity rating of 0 indicates that acetone is stable under a variety of conditions, including exposure to water.

CH118 CHEMISTRY LABORATORY SAFETY AGREEMENT 2009

I have read the entire **Lab Syllabus**. I understand and will follow the safety practices and lab policies described therein.

I have been shown where the safety equipment is located in my lab room. I have drawn a map of my lab workspace in my lab notebook indicating the locations of all safety items to refer to as needed.

Please Print Your Name: _____

Your Signature: _____

Today's Date: _____ Jan 2009

Your Lab Section (circle one): A (meets Mon& Thurs) B (meets Tues & Fri)

Sometimes we like to get pictures of the investigators working in the crime lab. We might post your picture in an e-mail going out to your lab section at the end of the day or on the CH118 web page. Your picture might even be on a power point slide for a presentation at an American Chemical Society conference. We don't want to impose on anyone so just let us know if using your photo is a big no-no.

I give permission for photos taken of me or within a group during lab hours to be used only for CH118 purposes. Yes No

I give permission for photos taken of me or within a group during lab hours to be used only for professional academic purposes by the CH118 staff. Yes No

Please give this agreement to the laboratory supervisor before leaving today.
Thanks!