

Safety and Laboratory Rules and Standard Operating Procedures for Laboratory ABB 467

Please read carefully the following general guidelines, which MUST to be followed while working in the research lab. In addition to those issues reflecting on safety, we must all appreciate that the laboratory itself and much of the equipment is used in common. Proper laboratory and general maintenance practices will assure that a good environment, both scientifically and socially, is maintained in the lab. We all have responsibilities to make sure that these guidelines are followed. It is a well-known fact that bad laboratory practices lead to bad and non-reproducible results on behalf of the perpetrator as well as stress and frustration for one's co-workers. While anyone can have an accident in the laboratory, that possibility is greatly reduced when proper laboratory practices are adhered to.

Laboratory Notebook: A standard format is to be followed for maintaining the laboratory notebook. This consists of a table of reagents on the left hand page, which should be completed BEFORE the experiment is actually performed. Include the product data as well including the expected mass for 100% yield. The right hand side is used for the DATE, TITLE, REACTION SCHEME, and actual PROCEDURE followed. Details such as times, temperatures, tlc profiles should also be noted in the margin or on the back of the left hand page. It is sufficient to “sketch” the tlc profile indicating the solvent used in elution as well as the behavior to UV 254/360nm as well as any chemical development solutions used. I.e., “blue to anisaldehyde”, “UV254”, “white to KMNO₄” etc. This is very important as it proves when, where and how you made and identified a new compound. An example of this format is attached. All details should be written directly into the notebook and not on scraps of paper destined for transfer “later on”. All pages MUST be numbered. Compounds are identified according to your initials and a five digit code consisting of book # and Page #. For example RBW10057 would mean Robert Burns Woodward, Lab book 10, page 57. This number should be used to identify the compound on all spectra obtained from and flasks or vials containing the compound. Commercial compounds should be identified by the supplier name. All reaction parameters must be recorded in the notebook.

Compound Characterization: Every new compound prepared in the lab or natural product isolated should be fully characterized by ¹H, ¹³C NMR (¹⁹F or ³¹P as appropriate), IR, MS, HRMS, and optical data if the compound is chiral. Specific compounds may also require x-ray structural determination in consultation with your supervisor. The hard-copy data for each new compound should be placed in a standard brown file and fully assigned. Compounds are labeled according to the researcher’s initials and lab book/page number (see Lab notebook above). The data should be assigned and the procedure written up in standard publication format.

For example:

Dimethylmalonytributylphosphorane: Into a flame-dried round-bottom flask containing dry EtOAc (50.0 mL) was added dimethyl-2-chloromalonate (5.113 mL, 40.0 mmol, 1.0 eq) and tributylphosphine (10.0 mL, 40.0 mmol, 1.0 eq) was injected via syringe under argon. The phosphonium salt occasionally crystallizes at this point with no deleterious effects on the reaction. The mixture was cooled in an ice bath while dry Et₃N (5.575mL, 40.0 mmol, 1.0 eq) was added dropwise (mild exotherm) to the mixture via syringe. Upon the addition of base the solution develops to a viscous white slurry. The reaction mixture was stirred at room temperature for one hour under argon. The slurry was filtered under positive argon through a short column containing a cotton plug and the precipitated Et₃N-HCl washed thoroughly with

EtOAc. The clear filtrate was concentrated by removing the solvent under reduced pressure to give 6a as a colorless oil, 13.105g, 98.6%. ¹H-NMR (CDCl₃) : δ 0.75 (t, 3H, J=6.75), 1.21-1.28 (m, 12H), 1.91-1.98 (m, 6H), 3.47 (s, 6H). ¹³C-NMR: δ 13.2, 21.8 (d, J=55Hz), 23.6 (d, J=15.4Hz), 23.9 (d, J=3.8Hz), 27.3 (d, J=65Hz), 49.9, 168.8 (d, J=14.5Hz). ³¹P-NMR: δ 27.8. MS: m/z (% rel.): 332 (24.3), 303 (53.4), 301 (100), 276 (30.1), 261 (31.1), 219 (22.0), 206 (25.8), 177 (22.8). HREIMS: calcd. for C₁₇H₃₃PO₄ 332.2116; found 332.2112. FTIR (KBr): 2958, 2936, 2872, 1678, 1640, 1605, 1433, 1312 (P=C), 1076 cm⁻¹.

Safety glasses: Safety glasses must be worn at all times in the laboratory. Lab coats are recommended. Open toed shoes should not be worn in the lab, shorts are permissible however in this case the use of a lab coat is mandatory.

Fume hoods: Fume Hoods must be kept open only 10-15cm and closed when distillation or high vacuum apparatus is in use. Hoods MUST be kept clean at all times and contain only the equipment needed at that time. Spills of silica gel or chemicals must be cleaned up right away. This also applies to the stainless steel surround. Do not push flasks or beakers of material to the back of the hood for “future analysis”. The hood should be clean enough for you to eat your lunch inside at all times (but don't try this).

Benches: Benches should be kept clear at all times and contain only that equipment needed for the work in progress at the time. Any spillage including chemicals and silica gel must be cleaned up right away. Be careful with drawn-out glass tlc applicators as these sharp pieces of glass are difficult to see if left lying on benches.

Equipment: No equipment should leave the laboratory (with the exception of routine material for NMR etc) without my permission. This particularly applies to Dewar flasks, syringes, needles and chemicals.

Glassware: Do not accumulate dirty glassware. All glassware should be cleaned immediately after use. No dirty glassware should be present in the fume-hood or sink at any time. Any broken glassware should be reported to me as soon as possible and replaced through the glass shop. Simply placing it in the broken glass container and forgetting about it is completely unacceptable. Broken glass should be placed INSIDE the broken glass container not on the floor beside. Glassware is never placed in the sink unless the user is in the process of cleaning that equipment. It is a bad habit to place dirty glassware in the sink for “future” clean-up, and inconsiderate to co-workers.

Vacuum pumps and traps: Pumps should always have a liquid nitrogen trap connected when solvent is being removed or a product distilled. Traps must be rinsed out promptly after use.

Make sure you are aware of the dangers of oxygen condensation whenever using liquid nitrogen.

Solvents: Solvents should be stored in the yellow cabinet or under the fume hoods when not in use. Empty bottles should be thrown out. The use of 95% ethanol for cleaning glassware is recommended, acetone should be avoided. Waste solvent should be placed in the correct container (halogenated or non-halogenated). Full waste bottles should be taken to the disposal area and not allowed to accumulate. Great care should be taken with the larger 18-20L steel drums used for common solvents such as acetone, hexane(s) or methanol. These drums should not be tilted or lifted to dispense. The drum should be grounded and solvent removed using a siphon pump. Ensure that no sources of ignition are possible.

Dry solvents: Dry permanent stills are maintained of THF, toluene, DCM, pyridine and triethylamine. These stills are to be maintained by a designated person (see schedule) and all others must cooperate with that person to ensure a good supply of dry solvent is available to anyone when needed. Much care is needed in operating the dry solvent stills. If in doubt, ask and do not operate any of the permanent stills until you have been trained and are comfortable with the operation. Solvents should be gently refluxed only; cold water should be running through the condensers at a slow rate. Solvents must never be allowed to evaporate to dryness; this can be fatal in the case of ethers like THF. The use of diethyl ether is not recommended unless this is absolutely crucial.

Manifolds: No chemical other than solvent should ever be in contact with the manifold. Use a balloon filled with Ar or N₂ rather than a direct needle connection to the manifold. Any seized joints on the manifold should be reported as soon as possible.

Rotary evaporators: Rotary evaporators should be kept clean at all times. Solvent receivers should be emptied out immediately after use. Solvent traps should be cleaned and placed back on the rack after use, not left dangling on the rotary evaporator.

Balance: Care must be taken that chemicals not be spilled on the balance. Any spills must be cleared up right away.

TLC plates/UV Lamps: Use the minimum size on strip needed for your analysis and please discard any old plates after recording the data. Clean up any silica “dust” from the benches after handling plates. Developing solutions should be kept tightly capped and stored in order at the back of the bench. Do not leave broken parts of tlc capillary tubes on the bench as they act like a lancet. Please make sure the UV lamp is turned off after every use. If using a heat gun to develop a tlc plate, do so in the fume hood.

Doors: The two main entrance doors are to remain closed and unlocked during normal hours of operation during the day. If you are the last one to leave the lab at any time during the day, it is your responsibility to make sure the door is closed and locked at any time. Doors must be locked after 5:30 pm as a security precaution.

Carbon Monoxide: One fumehood is dedicated to reactions involving the use of CO. All chemistry is to be performed in this hood with the cylinder placed at the back of the hood. The fumehood sash must be fully down when working with CO. The CO detector must be installed on the front of this fumehood and should be tested before opening the CO cylinder.

Syringes: Syringes MUST be cleaned out with solvent and after immediately after use. Micro-Mate interchangeable syringes should be placed back in the drying oven. Analytical Hamilton-type syringes must NEVER be placed in the oven but air-dried and replaced back in the drying cabinet after use. The Hamilton syringes and mated plunger should be kept together. Remember, everyone, yourself included, relies on having access to clean dry syringes many times each day. Detachable needles must also be cleaned immediately after use and may be dried in the oven. It is completely unacceptable to leave needles lying around on benches or in fume-hoods, dirty or clean.

Sharps: Used needles, syringes, TLC applicators and any broken or drawn-out glass capillaries MUST be placed separately in the sharps waste receptacle and NEVER placed in regular rubbish containers.

NMR Tubes: NMR tubes should be rinsed out after use, washed with an appropriate solvent and water followed by a final rinsing with acetone or ethanol. NMR tubes should never be placed in the oven for drying.

Ovens (Glassware): All ovens in the lab will remain turned OFF when not in use. When the oven is required, it should be switched on by the user the day it is required and turned OFF again by the user when done. If another person is also using the oven, a clear arrangement should be made to transfer the authority for turning off the oven to the next user. In all cases, the last person out from each side of ABB 467 should do a final check to make sure the ovens are off at night.

Working alone and/or after hours: No one should carry out a chemical operation when they are the only person present in the lab. This particularly applies to potentially dangerous operations involving solvents or pyrophoric and/or reactive reagents. In addition, if you plan

to be in the lab after 6:00pm or on a weekend, you are required to inform Security (Tel. 24281) upon arrival and departure.

LAST PERSON OUT: When you are the last person to leave the lab for the day, please make sure of the following five details: All distillation units and condensers should be turned off except for the THF still (gentle reflux). Gas cylinders are turned off. UV lamps are off. Water baths for the rotary evaporators are off. Door is locked.

Safety Training: All Faculty, graduate students and postdoctoral fellows are required to have completed the following safety training courses.

Asbestos awareness:

Ergonomics:

Fire Safety:

WHMIS Core (in class training):

Slips, trips and falls:

Chemical handling & Spills:

Violence and Harrassment Prevention in the Workplace:

Accessibility (AODA):

Spills:

Gas cylinder:

Record of Safety Training:

Name: _____

<u>Training course:</u>	<u>Date taken:</u>
Asbestos awareness:	_____
Ergonomics:	_____
Fire Safety:	_____
WHMIS:	_____
Slips, trips and falls:	_____
Chemical handling:	_____
Spills:	_____
Gas cylinder:	_____
Violence and Harrassment Prevention in the Workplace:	_____
Accessibility (AODA):	_____

Declaration:

I _____ (FULL NAME) having been provided a copy of the safety and laboratory rules to be followed in Professor McNulty's laboratory, and University guidelines on safety training requirements and hereby declare my understanding and commitment to follow these regulations at all times.

Signature: _____

Date: _____