INOMAR CENTER MANUAL

Guidelines, Procedures and Requirements for Safety and Proper Laboratory Practices

Out of this nettle, danger, we pluck this flower, safety. *William Shakespeare*

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1.3 Chemical Waste Guidelines:

Liquid Waste

- **Personal Waste Bottle**
  - Recycle empty 4 L solvent bottles to use as waste containers
  - Use for contaminated solvents, i.e. containing metal salts
  - Secondary waste container is mandatory
  - Fill out waste label to identify the components of the waste and the start date of accumulation
  - Make sure cap matches bottle
  - 2/3 (66%) full, and hold for < 90 days
  - Do not cap tightly, but always have cap on
- **Organic Drums**
  - Only for PURE ORGANIC solvents. NO acids, NO metals
  - Rotovap and column solvent waste
  - Indicate amount and identity of solvent you are adding

Solid Waste

- **Solid Waste Containers**
  - For all solid chemicals, and paper towels or gloves contaminated with chemicals or solvent.
  - Do not throw any contaminated solid materials into normal trash bins, these should only go in marked solid waste containers
- **Sharps Container**
  - Only used for sharps (i.e. needles)
  - Do not exceed 75% full
  - One per lab
  - Clearly label that the container is for SHARPS ONLY!
- **Glass Disposal**
  - All unwanted glass (i.e. pipettes)
  - Rinse first – no chemicals should be on them before disposal
  - Once full, label the container as CONTAMINATED GLASS
- **Empty Reagent or Solvent Bottles**
  - Wash/rinse with water or acetone and dry in waste hood overnight (located in 305, aka Lecture Room)
  - Cross off name of chemicals
  - Open sure seal cap with cap opener for venting

1.4 GENERAL LAB SAFETY RULES (post a copy on your desk)

IN CASE OF EMERGENCY DIAL **113, 114, or 115** FROM A LAB PHONE OR FROM A CELL PHONE

GENERAL SAFETY AND WASTE RULES (INOMAR May 24)

LAB SAFETY

1) **Proper attire must be worn in lab.** This includes a lab coat, protective eyewear at all times, no shorts or open-toed shoes while in lab.

2) **Nothing is to be placed on the floor.** Solid waste containers, dewars, and chairs are the only exceptions. No items can be placed on the sills of the hoods.

3) **All solvents must be returned immediately to cabinets** once they have been used. No solvent or chemical bottles should be left in the hood or on the bench after use. They should always be returned to the proper location.

4) **All equipment must be used for its designed purpose**, and as such they should be returned to their proper storage place after use.

5) **Label all containers** with the date, their contents or the corresponding lab notebook page, and your name if they are in a public area, no matter what is in them.

6) **NEVER work alone and check your area for safety and orderliness** before leaving the lab.

WASTE MANAGEMENT

1) Waste bottles should be filled to a maximum of 70% percent of their total capacity.

2) Waste tags should be clearly labeled and have a start date.

3) Once full, waste should be transferred immediately to designated areas of the lab.

4) Waste containers must be held in an external plastic waste container and capped while not in active use.

I have read, understood and will practice these points (sign and date):

1.5 and 1.6 Fire Safety and Emergency Preparaiton

(Taken from University of California, Berkeley, College of Chemistry Health and Safety Manual, Section 10)

FIRE

Small fires can be extinguished without evacuation. However, an immediate readiness to evacuate is essential in the event the fire cannot be controlled. Fire extinguishers are only to be used by individuals who are trained in their use.

FIRE: IMMEDIATE PROCEDURES

Small Fire
Alert people in laboratory and activate alarm.
Smother fire or use correct fire extinguisher (see Fire Fighting, below).
Aim extinguisher at base of fire.
Always maintain accessible exit.
Avoid smoke or fumes.

Major Fire
Alert people in area to evacuate.
Activate nearest fire alarm.
Close door and windows to confine fire.
Evacuate to safe area or exit building through stairwell.
Call Emergency Response number, 113, 114, or 115 to provide details of the incident.
Have person knowledgeable of incident and laboratory assist emergency personnel.

FIRE: GENERAL INFORMATION

Fire Alarm Procedures for the Evacuation of INOMAR CENTER

Plan your evacuation routes before it becomes necessary, be familiar with them, and always have alternate routes in mind. Persons who are unable to walk should be carried. Always ASK someone with a disability how you can help BEFORE attempting any rescue technique or giving assistance. Ask how he or she can best be assisted or moved, and whether there are any special considerations or items that need to come with the person. DO NOT evacuate disabled people in or with their wheelchairs. If people with mobility impairments cannot exit, they should be moved to a safer area, e.g., most enclosed stairwells or an office with the door shut which is a good distance away from the hazard. Notify police or fire personnel immediately about any people remaining in the building and their locations. Keep a safe distance from the buildings. (In the event of a major catastrophe, all personnel should assemble in front of the INOMAR building

When the fire alarm sounds, it is required that all occupants evacuate the building. (If you are carrying out a procedure that would result in a hazardous condition by your immediate evacuation, you are allowed you to take a very brief time to bring your area to a safe condition before leaving. Your own safety and the safety of your neighbors should be considered at all times.)

The occupants of the building SHALL NOT re-enter the building even though the fire alarm horns/bells have been silenced. The Fire Department may deem it appropriate to silence the fire alarm immediately.

Fire Fighting

It is not the responsibility of our students or employees, including Safety Czars, to fight fires. However, if you are trained in using extinguishers and are sure that there are no hazards from which you are not protected, you may prevent further injury or damage by continuing through the following steps:

Select the proper fire extinguisher:
(1) For ordinary combustibles, such as paper or wood, use Class ABC extinguishers located by the door of each lab.

(2) For flammable liquids, use CO_2 or dry chemical extinguishers located by the door of each lab (Class: ABC or BC).

(3) For electrical fires, cut power source at main electrical panel. Extinguish with dry chemical or CO_2 extinguishers (Class ABC or BC).

Dry Chemical Fire Extinguishers (Class ABC) are effective on all types of fires except flammable metals, unlike CO_2, which is not effective on common combustibles such as paper. The dry chemical extinguishers allow you to be further from the fire and are more effective than the CO_2 units. (A 9 kg dry chemical extinguisher can extinguish ~12 times more fire than the same size CO_2 extinguisher.

Before entering an area that contains burning material, if you feel you can enter the room without putting yourself in any danger, the following must be taken into consideration:

(i) Do not enter an area if you suspect that the fire has produced toxic gases.
(ii) Feel the closed door with the back of your hand. If it is hot, leave the door closed.
(iii) If the door is cool, open it a crack to see if the fire is still confined and small. If not, close the door.
(iv) If the fire is small and you elect to enter the room, keep yourself between the fire and the door at all times.
(v) If the fire condition worsens, exit the room and close the door.
(vi) If the fire remains small, direct the contents of the appropriate extinguisher to the base of the fire but always keep yourself between the fire and the exit.

• File a written report within 24 hours on every accident involving fire.

1.7 Glove Chart

CHEMICAL	Butyl Rubber	Chlorinated Polyethylene	Viton/ Neoprene	Natural Rubber	Neoprene	Nitrile + Polyvinyl Chloride	Nitrile	Polyethylene	Polyvinyl Alcohol	Polyvinyl Chloride	Viton	Butyl neoprene	Other Materials*
Acetaldehyde	RR	NN		NN	NN	NN	NN	NN	nn	NN	NN		Yes
Acetic acid, glacial	R	rr		nn	RR	NN	RR	nr	n	NN	rr		Yes
Acetone	RR	NN		NN	NN	nn	NN	NN	NN	NN	NN		Yes
Acetonitrile	RR	rr	nn	NN	NN		NN	NN	rr	NN	rr	rr	Yes
Ammonium hydroxide	R	r		rr	rr	NN	rr	NN	n	NN	r		Yes
Amyl alcohol	rr		r	NN	RR	NN	nn	nn	rr	NN	rr	r	Yes
Aniline	RR	r	rr	NN	NN	NN	nn	NN	RR	NN	NN	rr	Yes
Benzaldehyde	rr	n	n	nn	nn	n	nn	NN	RR	N	n	r	Yes
Benzene	NN	nn	rr	NN	NN	NN	NN	NN	NN	NN	nn	rr	Yes
Butyl acetate	rr	r		NN	NN	nn	NN	NN	rr	NN	nn		Yes
Butyl alcohol	R	r		nn	RR	nn	RR	RR	nn	nn	r		Yes
Butane	r			N	R	r	n			N	r		Yes
Butyraldehyde	nn		n	R	nn	r	r		nn	R	nn	r	Yes
Calcium hypochlorite	r			R	R	r	r			R			Yes
Carbon disulfide	NN	NN		N	N	n	NN	NN	RR	N	RR		Yes
Carbon Tetrachloride	N	nn	r	NN	NN	NN	N	NN	RR	NN	rr	n	Yes
Chloroacetone		r		n	n	R	n			N		r	Yes
Chloroform	N	NN	r	NN	NN	n	NN	NN	RR	NN	rr	n	Yes
Chromic acid	n	r		NN	N	RR	N	rr		RR	r		Yes
Cyclohexane	N	r	r	NN	NN	n	RR	NN	nn	NN	RR	n	Yes
Dibenzyl ether	r		n	N	R	r	r			R		r	Yes
Diethanolamine	rr			r	rr	n	nn			r	rr		Yes
Diethyl ether	NN	r	n	NN	NN	nn	NN	NN	RR	nn	NN	n	Yes
Dimethyl sulfoxide		rr		RR	RR	rr	nn	rr		NN			Yes
Ethyl acetate	n	nn	n	NN	NN	nn	NN	NN	n	nn	n	n	Yes
Ethyl alcohol									rr				Yes
Ethylene glycol	R	r	r	RR	rr	RR	RR	RR	rr	nn	r	r	Yes
Ethylene trichloride	NN	nn		NN	NN	NN	NN	NN	NN	NN	NN	n	Yes
Formaldehyde, 37%	RR	rr	r	NN	NN	nn	NN	RR	n	NN	RR	r	Yes
Formic acid, 90%	R	r		R	R	R	r	NN		R	n		Yes
Glycerol	r		r	r	R	r	R			r		r	Yes
Hexane	NN	rr		NN	NN	NN	NN	NN	RR	NN	RR		Yes
Hydrobromic acid	r			r	R	r				R			Yes
Hydrochloric acid conc.	nn	rr	rr	rr	RR	RR	rr			NN	rr	rr	Yes
Hydrofluoric acid			r	RR	rr	NN	nn	r	n	nn	r	r	Yes

Hydrogen peroxide	nn	rr	r	r	R	r	n			nn	r	r	Yes
Isobutyl alcohol	rr		r	nn	NN	NN	RR	NN	n	NN	rr	r	Yes
Methylamine	r			nn	rr		rr		n	r			Yes
Methyl alcohol	rr	rr	rr	NN	NN	nn	NN	nn	NN	NN	nn	rr	Yes
Methyl chloride	r			N	n	n	n	n		N			Yes
Methylene chloride	NN	nn	r	NN	NN	nn	NN	NN	nr	NN	nn	n	Yes
Methyl ethyl ketone		RR	rn	NN	NN	NN	NN	NN	NN	nn	NN	NN	Yes
Naphthalene	N	rr	r	N	nn	NN	rr	NN	rr	NN	r	n	Yes
Nitric acid	r	nn		nn	n	NN	nn	nr	n	NN	rr		Yes
Perchloric acic	r		r	N	rr	rr	rr	rr		rr	r	r	Yes
													Yes
Phenol	R	nn		NN	nn	n	NN	rr	rn	NN	n		Yes
Phosphoric acid, conc	r			rr	rr	r	rr	rr	n	n			Yes
Potassium hydroxide	r			R	R	r	R			R	n		Yes
Pyridine	r			NN	NN		NN	r			n		Yes
Sodium Hydroxide	n	rr		R	R	n	R	r		rr			Yes
													Yes
Sulfuric acid	n	RR	rr	N	rr	nn	n	r		NN	rr	rr	Yes
Toluene	NN	r	rr	NN	NN	nn	NN	NN	NN	NN	nn		Yes
Trichloroethylene	NN	nn		NN	NN	NN	NN	NN	NN	NN	nn		Yes
Triethanolamine	r	r	r	N	R	r	R	r		rr	n	r	Yes
Xylene	n	n	r	NN	NN	NN	NN	NN	RR	NN	rr	n	Yes

Source: Guidelines for the selection of Chemical Protective Clothing, 1987. American Conference of Governmental Industrial Hygienists, Inc. Cincinnati, Ohio

Legend

RR= recommended based on strong data
rr=recommended based on data
R=recommended based on judgement
NN= not recommended based on strong data
nn= not recommended based on data
n= not recommended based on judgement

*other materials are recommended. Consult the Source or vendor's glove selection charts.

1.8 Chemicals of Particular Danger

Always use the minimum necessary amount

	Only in glove box	Max buy amount	Use amount
Pyrophorics			
n- butyllithium in hexanes*		100 mL	10 mL (or less)
t-butyllithium*		0 mL	10 mL (or less)
t-butyl trichlorosilane		100 mL	10 mL (or less)
diethylzinc 1.0 M in hexanes		100 mL	10 mL (or less)
methyllithium complexed with LiBr		100 mL	10 mL (or less)
lithium	X	10 g	
lithium aluminum hydride		100 g	
sodium	X	10 g	
potassium	X	10 g	
sodium hydride		100 g	
potassium hydride		100 g	
potassium borohydride		100 g	
sodium borohydride		100 g	
raney nickel slurry in water		100 mL	
triisopropyl borate			
trimethylaluminum			
Strong acids/acid formers			
boron tribromide		250 mL	
fumic sulfuric acid		2 L	
fuming nitric acid		2 L	

phosphorous oxychloride			
thionyl chloride			
titanium chloride			
trichlorosilane			
trifluoromethanesulfonic acid		100 mL	
trifluoromethanesulfonic anhydride			
Poisons/poison formers			
cupric cyanide			
phosphorous trichloride in dichloromethane			
sodium azide		100 g	
sodium cyanide		100 g	
sodium cyanoborohydride		100 g	
bromine		250 mL	30 mL

*** When using a syringe to take aliquots of BuLi the bottle MUST be clamped to prevent accidental spillage.**

1.9 Neutralization and Disposal of Dangerous Chemicals

Personal Protective Equipment (goggles, lab coat, and gloves) must be worn at all times, and all chemical procedures must be performed in a fume hood. Always place a cold bath (ice bath, liquid nitrogen etc.) nearby. Do not perform the procedure alone. Listed below are some of most commonly used dangerous materials in the lab. If you are going to dispose any dangerous materials other than the listed, please check MSDS and the references given in the bottom before taking the next step.

Sodium/potassium metal: Cover the metal with sodium carbonate or calcium carbonate. Behind a shield, add a little at a time to a large excess of dry tert-butyl alcohol (potassium) or butanol (sodium). Allow it to stand for 24 hours before disposal.

Metal hydrides:
Lithium aluminum hydride: Dropwise addition of 95% ethanol to $LiAlH_4$ solution under nitrogen in a three necked flask; alternatively, a safer procedure is to add ethyl acetate SLOWLY to the solution in a flask equipped with a stirrer. When the reaction has ceased, a saturated aqueous solution of ammonium chloride is added with stirring. Dispose the waste accordingly.

Sodium/potassium hydride: Adding enough dry hydrocarbon solvent to reduce the hydride concentration below 5% and then adding excess t-butyl alcohol dropwise under nitrogen with stirring. Cold water is then added dropwise. Dispose the waste accordingly.

Calcium hydride: Adding 25mL of methyl alcohol per gram of hydride under nitrogen with stirring. When reaction has ceased, an equal volume of water is gradually added to the stirred slurry of methoxide. The mixture is then neutralized with acid and disposed of in the sanitary sewer.

Phosphorus pentoxide: Place the phosphorus pentoxide in a large evaporating dish and cover with excess solid sodium carbonate or calcium carbonate. Add very slowly the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize with sodium carbonate or 5% sodium hydroxide solution if necessary.

Butyl lithium: Excess butyllithium solution can be destroyed by dilution with hydrocarbon solvent to a concentration of approximately 5 wt%, followed by gradual addition to water with vigorous stirring under an inert atmosphere. Alternatively, the butyllithium solution can be slowly poured (transfer by cannula for s- or t-butyllithium) into a plastic tub or other container of powdered dry ice.

References:
Prudent Practices in the Laboratory: Handling and Disposal of Chemicals, 1st ed., National Academic Press, Washington D. C., 1995
Margaret-Ann Armour, Hazardous Laboratory Chemicals Disposal Guide, 3rd ed., CRC Press LLC, Boca Raton, FL, 2003

1.10 Introductory Lab Safety Checklist (Must fill in all blanks and be checked off by Safety Czar before working in lab)

Apparel

1. Safety glasses must be worn at all times.
2. Long pants must be worn at all times.
3. Closed-toed shoes must be worn at all times.
4. Cotton or fire-retardant cotton lab coats must be worn at all times.
5. Appropriate gloves must be worn while doing lab work.
6. No headphones in the lab.
7. Gloves and coat must be removed once leaving the room.
8. No gloves should touch computer keyboards/mice.
9. Disposable nitrile gloves are stored _____
10. Non-disposable latex gloves are stored _____
11. Thermal gloves are stored _____
12. Butyl rubber gloves are stored _____
13. Will nitrite gloves protect against acetic acid ___?
14. Will nitrile gloves protect against acetone ___? dichloromethane ___?
15. Will nitrile gloves protect against methanol ___, sodium hydroxide___, toluene ___?

Training

16. Do not use any instruments, apparatus, or Schlenk lines until trained, even if you have been trained in other labs.
17. Contact the czar of an instrument for training.
18. Don't use any chemicals whose properties you do not understand. Use dangerous chemicals with supervision the first few times.
19. Read the section in the INOMAR manual that corresponds to an instrument and be checked out by that instrument's czar before using it.

Safety equipment/supplies

20. ABC (powder) fire extinguishers are located _____
21. BC (CO_2) fire extinguishers are located _____
22. D (salt) fire extinguishers are located _____
23. Spill kits are located _____
24. First aid kits are located _____
25. Eyewash stations are located _____
26. Emergency showers are located _____
27. The lab safety manual is located _____

Chemical storage

28. No glass bottles are to be placed on the floor.
29. All containers must be labeled clearly with the date they were filled, their contents or the corresponding lab notebook page, and your name if they are in a public area. This goes for anything, including water and "harmless" chemicals.
30. Solids are generally stored _____
31. Liquids are generally stored _____
32. Bulk solvents are stored _____
33. Bulk solids are stored _____
34. Oxidizers are stored _____
35. Flammable chemicals are identified by _____
36. Water reactive chemicals are identified by _____
37. Corrosive chemicals are identified by _____
38. Flammable chemicals can be stored in _____
39. Water reactive chemicals are stored in _____
40. Nonflammable liquid acids and bases are stored _____
41. Corrosive, flammable, and carcinogenic chemicals must be stored segregated from each other in some secondary containment. Chemicals that fall into more than one of these categories should be separated into their own containment.
42. Gas cylinders must be stored _____

Working in the hood/lab

43. No food or drink in the lab.
44. Chemicals on the "Dangerous Chemicals List" may not be used without supervision until you are familiar with them.
45. Solid waste containers, dewars, and chairs are the only things that can be on the floor.
46. Never do anything dangerous with no one in your lab. Do not do normal work unless another lab member is there (not necessarily in your lab).
47. All equipment must be used only for its designed purpose.
48. Oil baths must have lab jacks under them.
49. Oil should not exceed 2/3 of the bath container; flask should be smaller than half the area of the bath. They should contain only **silicone oil** (and be labeled as such; throw out unlabeled oil.)
50. When using dangerous chemicals (BuLi, Na, etc) use minimal amount.
51. Use appropriate gas-tight, Leur lock syringes for such chemicals.
52. When scaling up organic reactions from written preps consult a group member familiar with the chemistry and limit yourself to a 2x scale.
53. Solvents must be returned to storage immediately after use.
54. Clamp or tie any running water tubes when unattended.
55. If running reflux/heating overnight remove fuel sources (as possible) from hood and close the sash. Put a sign on your hood indicating that contents/conditions in case something goes

wrong while you're not there.
56. You must be trained in how to quench water-reactive chemicals before using them in stills or reactions.
57. Check your area for safety and orderliness before leaving. If you used your Schlenk line lower the Dewar, close off the valve to the pump above the Dewar, and re-pressurize the line.

Waste
58. Waste disposal procedures are located _____
59. Liquid waste is stored in _____
60. Solid waste is stored in _____
61. Waste tags should be on waste bottles at all times. When you transfer to the waste collection hood, make sure you identify the chemicals and composition. Do not abbreviate chemical names. One chemical per line except for trace chemicals (all on one line).
62. Fill waste bottles to < 75% full. Do not exceed 75% full.
63. Filled waste bottles/bags are collected _____
64. Waste bottles must be in secondary containment in the hood and closed when not in use. Take care when opening them as pressure often develops within.
65. After cleaning, glass is disposed _____
66. Glass waste should not be filled more than 66%.
67. After cleaning, sharps are disposed _____
68. Empty reagent bottles should remain capped after being washed.

In case of emergency
69. Make sure you are safe. If not, move to safety.
70. The laboratory evacuation site is _____
71. In a major emergency, call emergency services as soon as possible. If you are occupied, send someone to call and tell them to return to you after calling (if they do not return, assume no call was placed).
72. From a land line dial _____
73. From a cell phone dial _____ (program this number into your phone)
74. After emergency services are called, have someone stay on the line. Send someone to intercept them, give them a key, describe situation, etc.
75. The MSDS can be found _____
76. If a lab member needs to go to the hospital, make sure s/he is accompanied by another lab member.
77. The address of the Emergency Room is _____
78. Report any injuries or fires to the safety czar.
79. If a fire breaks out that is larger than approximately one hood area or trash can
_____

80. If a fire breaks out that is approximately the size of one hood area or trash can _____

81. If the fire contains paper, wood, or other carbon-based combustibles, you can use _____

82. If the fire contains flammable liquids (solvents, oils, etc) you can use _____

83. If the fire is in or on electrical equipment you can use _____

84. If the fire contains flammable metals, metal hydrides, or other flammable metal compounds, you can use _____

85. In the case of an acid spill use _____

86. In the case of a base spill use _____

87. In the case of a large liquid spill (not acid or base) use _____

88. If there is a gas leak (indicated by gas alarm) _____

89. If you have a minor injury, you must report it to the lab safety czar, who will relay the information to Prof. Dzung, Dr. Thi and Binh.

90. If you are absent from the lab (vacation, sickness, etc) for more than one business day, you must notify Dr. Thi two weeks in advance, or as soon as possible if unexpected.

1.11 Unattended Reaction Form

Never leave an unattended reaction without proper description

Unattended Reaction Form

Name:	**Date:**
Contact Number:	**Notebook reference:**
Solvent:	**Solvent boing point:**
Oil bath/mantle temperature:	**Water reflux (y/n)**

***Reaction Description*:**

1.12 INOMAR CENTER Safety Inspection Checklist (Mandatory before working in lab)

****NOTE: THIS SECTION THERE ARE MANY ITEMS STILL BEING WORKED OUT**

Safety Self-Inspection Checklist for Laboratories

One Inspection form per Laboratory Room

Date

Building _____ room _____

Inspector_____ PI _____

Inspector's signature _____ PI's signature _____

Answer each question by circling "YES" (satisfactory), "NO" (needs correction), or "N/A" if the question does not apply to your laboratory. After completing the self-inspection form, share the results with the Principal Investigator and other laboratory occupants. Correct each condition identified by each "No" answer as soon as possible.

SAFETY INFORMATION AND TRAINING

1. Researchers are familiar with the location and contents of:
The lab's Chemical Hygiene Plan
YES NO

2. Standard Operating Procedures for hazardous laboratory experiments or techniques have been developed and are available at all times.
YES NO

3. List one hazardous operation or technique in your laboratory that needs an SOP written for it.

4. An accurate chemical inventory for the laboratory is maintained on a general use computer.
YES NO N/A

5. Researchers are knowledgeable of how to obtain and interpret MSDS information.
YES NO N/A

GENERAL SAFETY AND HOUSEKEEPING

6. Emergency information sign is posted on the main entrance door to the laboratory.
YES NO

7. Danger/Warning/Caution signs are in place. YES NO N/A

8. Sink labels and fume hood stickers are in place. YES NO N/A

9. Food is not prepared, consumed, or stored except in "Clean Areas". YES NO

10. Floors and bench tops are free from excessive clutter.
YES NO N/A

11. Aisles and exits are unobstructed.
YES NO

12. Chemical containers are not stored on the floor in aisles or near exits.
YES NO N/A

13. All lighting systems are operational.
YES NO N/A

14. Lighting is sufficient.
YES NO N/A

PERSONAL PROTECTIVE EQUIPMENT

15. Safety glasses with side shields are available and worn by researchers at all times while in lab.
YES NO

16. Safety goggles and face shields are available and worn when appropriate.
YES NO N/A

17. Chemically resistant gloves are utilized in the lab and NOT worn in the hallways, except with a clean outer glove.
YES NO N/A

18. All personal protective equipment is inspected and maintained regularly.
YES NO N/A

FUME HOODS

19. Face velocity has been checked in the last 12 months.
YES NO N/A

20. Exhaust slots are not blocked.
YES NO N/A

21. Sash(es) are in place and functional.
YES NO N/A

22. Fluorescent lights are functioning.
YES NO N/A

23. Containers with volatile chemicals are capped.
YES NO N/A

24. Fume hoods are not overly cluttered.
YES NO N/A

VENTILATION

25. No noticeable odors in the lab.
YES NO N/A

26. Normal temperature and humidity.
YES NO N/A

27. Negative pressure is maintained between the lab and the corridor.
YES NO N/A

28. Lab windows and corridor doors are kept closed.
YES NO N/A

SEISMIC HAZARD PREVENTION

29. Furniture and equipment are adequately secured.
YES NO N/A

30. Shelves have earthquake lips/barriers.
YES NO N/A

31. Overhead storage is minimized and restrained.
YES NO N/A

ELECTRICAL

32. Permanent wiring is used in place of extension cords.
YES NO N/A

33. At least 36" clearance maintained in front of electrical panels.
YES NO N/A

34. Electrical cords are in good operating condition.
YES NO N/A

35. All equipment is grounded.
YES NO N/A

36. No exposed wiring.
YES NO N/A

LABORATORY EQUIPMENT

37. Equipment with moving parts is adequately guarded.
YES NO N/A

38. Equipment disconnects are accessible.
YES NO N/A

39. Rotovaps are NOT hooked up to water aspirators.
YES NO N/A

GAS CYLINDERS

40. Cylinders are securely fastened with TWO chains to immovable objects or wall mounts. YES NO N/A

41. Cylinders are capped when not in use.
YES NO N/A

42. Incompatible cylinders are segregated.
YES NO N/A

43. Hydrogen and other flammable gas cylinders are grounded to non-painted, bare metal surfaces.
YES NO N/A

44. Flammable gas is not used with materials that can create a spark.
YES NO N/A

45. All cylinders are currently inventoried on the Inventory database, and excess cylinders are kept to a minimum.
YES NO N/A

FIRE PREVENTION AND RESPONSE

46. Researchers are aware of the location of the nearest fire extinguisher and fire alarm pull box.
YES NO

47. Researchers are trained in the use of fire extinguishers.
YES NO

48. Access to fire extinguishers is unobstructed.
YES NO

49. Fire extinguishers have been recharged and certified in the last 12 months.
YES NO

50. Fire extinguishers are mounted.
YES NO

51. Fire alarm can be heard from anywhere in the room.
YES NO

52. Combustible materials are kept a minimum of 3 meters away from welding areas or open flames.
YES NO N/A

53. Storage of combustibles is minimized.
YES NO N/A

EMERGENCY EYEWASH/SHOWERS

54. Researchers are familiar with the location of the nearest eyewash/shower.
YES NO

55. Eyewash is operable and not blocked.
YES NO

56. Eyewash is flushed until water is clear at least once per month by a designated researcher.
YES NO N/A

57. Emergency shower(s) has been tested in the last 12 months & is not blocked.
YES NO

CHEMICAL SPILL RESPONSE AND PREPAREDNESS

56. Spill kits are readily accessible and researchers are familiar with where they are located.

YES NO N/A

57. Researchers are aware of the Emergency Assembly Areas for their buildings
YES NO

WASTE MANAGEMENT

58. Researchers (Waste Generators) are trained on the Hazardous Waste Program.
YES NO

59. Researchers are knowledgeable of the Center's drain disposal guidelines.
YES NO

60. All containers used for non-recyclable materials and contaminated lab debris are properly labeled.
YES NO N/A

61. Waste containers are kept closed and are in secondary containment trays.
YES NO N/A

62. Liquid radioactive waste is in a secondary container.
YES NO N/A

63. Red Sharps containers are used for all sharps disposal, and biohazardous sharps are collected separately from chemically contaminated sharps.
YES NO N/A

CHEMICAL HANDLING AND STORAGE

64. Researchers are aware of: The potential physical and health hazards of the chemicals in the lab
YES NO

65. The methods of handling chemicals safely.
YES NO

66. A maximum of 60 gal flammable liquid is stored inside a liquid storage cabinet.
YES NO N/A

67. A maximum of 10 L of flammable liquid is stored outside of a flammable liquid storage cabinet.
YES NO N/A

68. Incompatible chemicals are segregated.
YES NO N/A

69. Chemical storage areas are properly labeled.
YES NO N/A

70. Chemical containers are properly labeled.
YES NO N/A

71. Clean areas are labeled.
YES NO N/A

72. Chemical refrigerators are approved and appropriately labeled for holding chemicals and/or flammables.
YES NO N/A

73. Peroxide forming chemicals are dated and tested.
YES NO N/A

ADDITIONAL COMMENTS (use additional sheets, if necessary):

1.13 Further Safety Guidelines

1.13.1 YOUR MOST IMPORTANT RESPONSIBILITY IS YOUR AND YOUR CO-WORKER'S SAFETY

1.13.2 ALL SAFETY INCIDENTS OR CONCERNS NO MATTER HOW MINOR MUST BE REPORTED AND ADDRESSED IMMEDIATELY THROUGH THE SAFETY CZAR. If you have an accident, you must report it immediately. Even something that may seem minor may have profound consequences to your health. This includes cuts, puncture wounds from needles, direct contact with solvent(s) and/or reagent(s). Report all incidents to safety officers.

1.13.3 FOR ANY DEGREE OF MEDICAL ATTENTION IT IS IMPERATIVE TO INFORM DR. THI SO THAT ALL CARE THAT YOU REQUIRE IS PROVIDED AND STEPS CAN BE TAKEN TO ASSURE THAT AN INCIDENT DOES NOT OCCUR IN FUTURE.

1.13.4 If you feel you need medical attention, do the following:

a) If a major incident call 113, 114 or 115
b) If you feel you go to the hospital, make sure someone goes with you.
c) If you feel it is minor, see the Safety Czar so that proper procedures are followed.

1.13.5 YOU SHOULD NEVER WORK IN THE LAB ALONE – USE THE BUDDY SYSTEM. This is particularly important for scaling up a reaction, working with an open flame (the torch), reactions requiring very reactive materials (particularly strong oxidizing/reducing agents), high temperature reactions or when doing a reaction for the first time. Accidents happen quickly and without notice, and having another person there can assist you in dealing with the situation, or getting help if necessary. If you find yourself alone in the lab, make sure you are aware of others in neighboring labs that may be sought for help in case of emergency. For more information see the Standard Operating Procedures.

1.13.6 IF YOU ARE TIRED, DO NOT DO LAB WORK. Lab work should be performed when you are most alert, when you are a little tired this is the time to read papers, work on an X-ray structure, make figures, or go home to sleep so that you can work efficiently the next day. An alert and focused xxhr/week at the bench will be more fruitful than a hazy xx+10 hr/week.

1.13.7 YOU MUST KNOW THE LOCATIONS OF THE NEAREST SAFETY SHOWER, EYE WASH STATION, SPILL KITS, AND FIRST AID KITS.

1.13.8 Food and drink are not allowed anywhere in the lab. Bicycles and other domestic items are not allowed in the lab at any time.

1.13.9 Reactions (tubes, bombs, pressure vessels, etc.) must be clearly labeled with your name and identify which reaction is being performed. This is for your safety and the safety of others.

1.13.10 IF A REACTION IS OUT OF CONTROL OR IF THERE IS A MAJOR MALFUNCTION OF A PIECE OF EQUIPMENT

a) ASSURE THAT YOU ARE SAFE
b) Decide if you can deal with the situation safely
c) If you cannot solve the problem safely, leave the area immediately; close the fume hood sash if you feel it is safe.
d) Let your co-workers know of the incident
e) Call for help

1.13.11 You MUST wear a lab coat in the lab area all the time, no matter if you are working with chemicals or not. You MUST wear safety glasses at all times. You MUST wear gloves when handling solvents. You MUST understand the reactivity of the chemicals you are using- read MSDS sheets and consult with experienced co-workers prior to use of chemical of particular danger.

1.13.12 All waste containers must be labeled with type of waste and your name should be placed on label. Waste should be disposed of properly. See Waste Guidelines in the Standard Operating Procedures.

1.13.13 All non-MOF reactions and chemical containers, including synthesis of organic linkers, MUST be labeled with your initials, date opened or filled, a chemical description (a drawing is acceptable), and solvent. All MOF reactions including vials MUST be labeled with your initials, date, MOF description (e.g. Mg MOF-5), and solvent used. It is also recommended that you label your reactions with a reference to your notebook including the page number and sample identification. Note: You are also responsible for making sure that all labels are clear at all times, and are rewritten if they rub off.

1.13.14 Acids and bases in storage must have non-flammable spill trays.

1.13.15 Oil baths and refluxing reactions (involving potential hazards) should not be left unattended while in operation. Be conscientious of your reaction(s). Do not try to heat a solvent beyond the boiling point in anything other than a pressure vessel. If you are unsure about a particular step in a reaction, consult a senior member of the group for guidance.

1.13.16 Nothing should be stored or placed on the lab floor except bins for trash and broken glass. Floor drains should not be covered at any time.

1.13.17 Reactive chemicals should be returned to their proper storage location. No reactive chemicals or compounds of any type should be left unopened on lab benches or within hoods.

1.13.18 If you need training (torch, X-Ray equipment or other instrumentation) ask the person in charge of the equipment.

1.15.19 Any questions regarding safety should be directed to the safety officer of the lab. All

members of the lab should be familiar with and should follow The University of California, Berkeley's guidelines for safety.

1.13.20 All compounds that emit unpleasant or harmful odors must remain in hoods. Move the microscope into a hood when viewing such samples.

1.13.21 All chemicals that require special storage or handling are to be cautiously used according to the MSDS safety guidelines. Under no circumstances should chemicals stored under inert atmosphere be exposed to air/water.

1.13.22 In case of flooding or dripping water, please place plastic covering over vital equipment within harms reach. Call Group Czar and Safety Czar immediately.

1.13.23 In case of any kind of major emergency, (i.e. flooding, fire, etc.) all group members will be informed by the Safety Czar. When it is safe all members need to clear their schedules to assist in clean up.

1.13.24 YOU ARE EXPECTED TO CHECK YOUR WORKING AREA BEFORE GOING HOME FOR ANY POTENTIAL SAFETY HAZARDS.

1.13.25 IT IS PREFERABLE THAT REACTIONS NOT BE SETUP TO REMAIN RUNNING OVERNIGHT ESPECIALLY IN CASES WHERE SAFETY IS A CONCERN. If absolutely necessary, all precautions must be taken to avoid and accident: cooling water hoses clamped, heating sources at equilibrium, fume hood free of potential fuel sources (solvent bottles, paper towels) in case of file, reaction apparatus tightly clamped into position, and fume hood sash pulled down.

1.14 Read and Understood

I, _____, have read the contents of this safety booklet, understand them, and will practice them.

Signed:

Date:

2. Proper Use of Laboratory Resources

"Science begins with discovery followed by analysis. Therefore, each student must spend 6-7 hrs/day at the bench. This block of time must be protected from distractions, interruptions, teaching, and other activities such as solving crystal structures or other secondary matters not completely dedicated to discovery."

"*You can't continue to enjoy doing science unless you use resources wisely and properly*."

2.1 Personal Areas

2.1.1 To ensure that personal areas (bench, hood, drawers, sink, base bath, racks, etc.) are maintained in working order and to an acceptable level of cleanliness at all times, used glassware (pressure pipettes, vials, beakers, flasks, frits, graduated cylinders, etc.) must be cleaned daily prior to leaving at the end of the day. No glassware is disposable (vials especially), with the exception of small pipettes and glass slides. Each piece should be cleaned and reused. Glassware with hard to remove residues or particulates should be placed in a base bath for a short period of time before regular washing.

2.1.2 Proper base bath preparation includes obtaining a large or small hazardous waste bucket and layering the bottom with KOH. The salt is then dissolved in a sufficient amount of water and filled approximately one-half to two-thirds of the way full with EtOH. The base bath should be labeled properly, including name, date, and contents. Each person is responsible for cleaning the glassware out of his/her base bath. Glassware should not be kept in the base bath for more than 3-4 hours, as the harsh conditions of the base bath can dissolve the glass, making thinner glassware such as round-bottomed flasks more prone to cracking. Never place glass frits in a base bath.

2.1.3 Before you leave for the day, the following should be completed to maintain a safe, organized laboratory environment throughout the non-working hours. Make sure you have taken down your vacuum line properly (LN_2 removed from trap, trap emptied, vacuum pump isolated by closing valves, and outlet valve to trap vented). If the vacuum line must be in use unattended, ensure that LN_2 level is maintained and that the Dewar is properly insulated during the unattended period. The contents of all reaction vials, tubes, and/or flasks that are no longer running or being monitored are disposed of in properly labeled waste containers.

2.1.4 To ensure that all reactions that are running through the night are kept in safe order, all jacketed water condensers should be properly clamped and at a moderate pressure. Outlet hoses should be securely placed in the sink, and all joints with water flowing through them should be tied down tightly with copper wire. All reactions being refluxed or heated through the night should be kept away from flammable materials/chemicals, with the hood sash closed, and an unattended reaction form should be posted on your sash.

2.1.5 Clean, dry, and return to its proper drawer or cabinet all glassware you have used that day.

2.1.6 Close hood sash and turn out desk/hood lights. If you are the last person to leave your lab

module, turn out the room light.

2.1.7 If you are the last to leave the lab, make sure all doors are locked, and lights turned off.

2.2 Community Areas

2.2.1 Lab supplies are shared items that must be stored in community areas. No glassware, chemicals or supplies are permitted to remain in personal areas beyond the short length of time required for their use. They should be used and returned promptly to community areas. Keep in mind that personal areas and supplies are rented from the lab and not owned by their current users. Individuals have equal rights over each and every lab item regardless of who filled out the order form. Stocking of items in personal areas represents inefficient use of those items, disrupts the smooth operation of the lab, and is a waste that we cannot afford.

2.2.2 Clean up after yourself when using community areas such as balances, microscopes, sorption, TGA, IR, and remember to return chemicals to community areas specified in the inventory sheet.

2.2.3 Safety showers should remain completely unobstructed at all times. The back of the benches, under the showers, must remain free of all items.

2.3 Laboratory Equipment

2.3.1 All equipment (vacuum pumps, rotovaps, hot plates, furnaces, etc.) must be maintained to manufacturer's standards. It is the responsibility of the czars (see responsibility list) to make sure that a record is kept of all maintenance operations.

2.4.1 Due to the fragile nature, associated risks and specific operation of the Schlenk lines, those members new to the group must be trained and checked out by the Schlenk line czar before working with the lines. After this point, it is at the discretion of the graduate trainer working with the newer student to allow him/her to work alone. However, the czar will be directly accountable for the new student's usage and will be responsible for repairing any damages that result. When accidental damages do occur, the person in charge of the lines should be immediately notified.

2.4 Programmable Oven Guidelines

2.4.1 In order to use an oven, you must sign up on the list.

2.4.2 You may sign up again on the list after your most recent run is finished (i.e., you should not have more than one oven in use at a given time unless no one else has signed up).

2.4.3 Your "turn" consists of one program, which cannot be altered as your experiment is going.

Whatever time/temp you originally signed up for is what you get.

2.4.4 If you notice someone on the list desires similar conditions, combine your runs.

2.5 Laboratory Notebooks (Guidelines for Keeping a Clear and Prganized the Lab Notebook[1].)

Keeping a record is essential for complete reconstruction of the study and this is an important part of being a good professional. It is the only way of showing what actually went on at the time. Records must not only contain the data generated, but also prove that all the required procedures were correctly carried out at the correct time. If data are lost, or a complete record has not been made, the study validity may be seriously compromised.

Raw data are defined as original recordings made during the course of the study. These data are necessary for the "reconstruction" of the study, for example by an inspector, after the study completion date.
Thus, all the experimental data must be recorded in the Lab Notebooks exclusively in English and indicate:

2.5.1 *WHAT was done:*

1. It is a good lab practice to begin the experiment description including the target of the experiment or project.

2.The use of reaction schemes may help you to describe briefly and clearly the experiment conducted.

3. At the same time it is helpful to introduce information about CAS numbers, reference used for the reaction, commercial source of the reagents, etc.

2.5.2 *HOW it was done:*

1.Schematic representations of the glassware that was used AND detailed information about the procedure to set the reaction are essential for the reproducibility of the results.

2. Description of the molecular weight, densities of the materials (if liquids are used), solvent treatment (if degassed solvents are used), etc. are necessary while repeating the reactions.

3. During the workup procedure, detailed information of the purification method is necessary to avoid misunderstandings.

i. For instance if you used a recrystallization procedure always include the solvent system that was used and the temperature and time necessary to get the optimum results.
ii. If you perform a chromatography column, describe the length and diameter of the packed solid support as well as the amount solvents during the loading and running of the samples.

iii. If you did a solvent exchange procedure you can include if it was done under inert atmosphere, etc.

4. Document results of all analysis that have been made.

i. Example: experimental conditions (i.e., PXRD was collected with a Bragg-Brentano geometry, 0.03 degrees/step, 1s/step, with a silicon zero background sample holder on a dry sample, it shows a crystalline product with the first peak at 2theta= 5.5 degrees, it matches with MOF-5 pattern but there is presence of an extra line at 2theta = 15 degrees etc...).

5. Electronic data for the analyses should also be conserved, and it should be named consistently in a way easy to identify for other researchers.

6. A nomenclature consisting of a sequence of initials – lab notebook number – notebook pages is encouraged (i.e. FG-III-12) referring to the notebook page where the experiment / analysis is described.

7. It is recommended to document procedure and results of data analysis performed in computers.

i. Example: structure simulations or refinements (i.e., Rietveld refinement on sample FG-III-12 carried out with TOPAS v4.0, conditions are xxx or can be found in associated file named FG-IV-14.opj).

8. Filling a table of contents is encouraged.

2.5.3 *WHEN the work was performed and WHO performed the work*

1. Dating the experiments. This is an important tool to remember when a link or a MOF structure was prepared, since some of the materials may not be stable under stored conditions for a long time and the byproducts of this storage time may affect your experiments and the conclusions you can have from them.

i. In addition, the data should clearly identify who was responsible for carrying out the procedure and recording the data. If more than one person is involved in a procedure this should be recorded in the data, along with an identification of the responsibilities of each.

2. Signing the Notebook. Every page in the notebook should be signed or initialized as well as dated because it could be the only way to demonstrate the your authorship and date of the development you have achieved. Therefore, documenting the first time an idea is conceived and when it is first attempted its reduction-to-practice is essential part of your experiments.

3. Promptness. It is also important to describe the experiments in the notebook promptly. Data must be recorded as the operation is done. It is not acceptable to make the record some time after the task has been completed.

4. Legibility. Data that cannot be read are useless and records that are difficult to decipher raise

doubts as to their credibility. Data should be recorded in a logical way, and duplication should be avoided wherever possible.

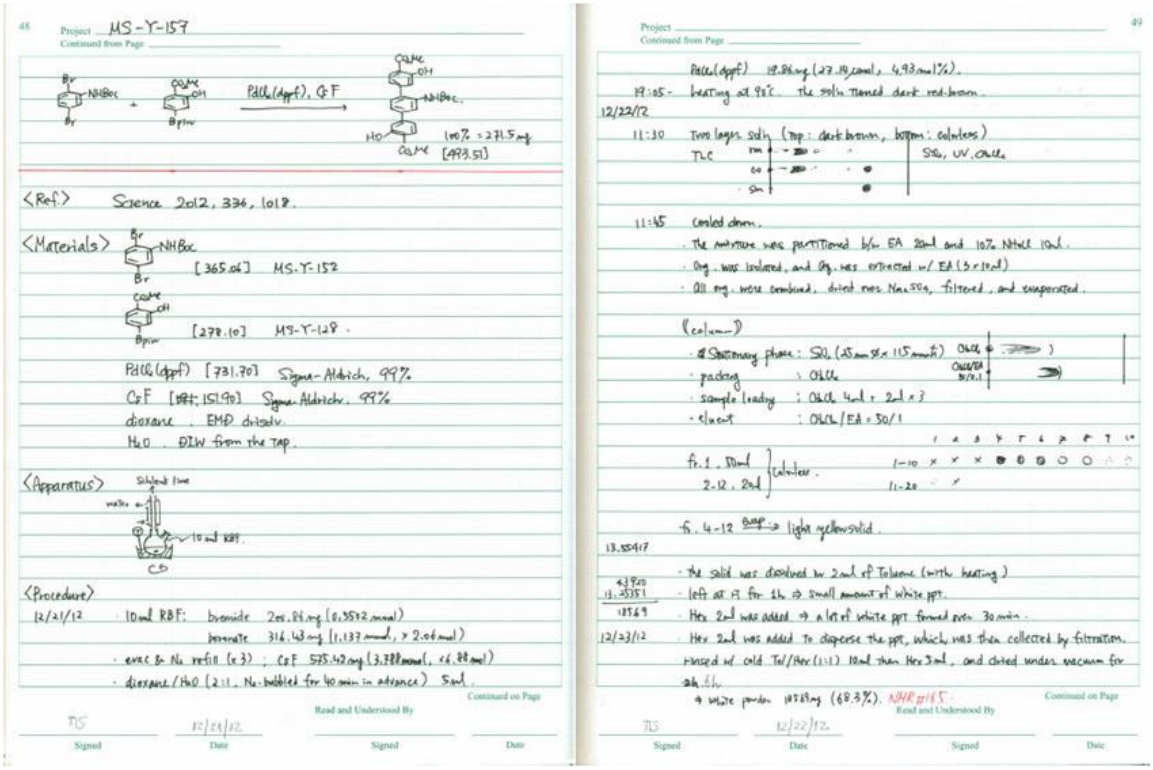

Figure 1. Example of a good notebook in the Yaghi Lab.

2.5.4 Notebooks are *legal* documentation of your experiments data, results, as well as ideas of the projects. Keep your research records as if each project were to be patented. Even though the work contained in the book may not result in a patent application, observance of these practices will provide a clear record for reports, publication or for future reference" for yourself and others. Accordingly, notebooks should be closed and put away when they are not being used.

Please take into account that the **notebook belongs to the lab**, and it will remain in the lab after the student/postdoc departure.

2.6 Computer and Telephone Usage

2.6.1 No surfing on the internet or downloading programs which are not approved by the computer czar or are not relevant to scientific research.

2.6.2 Data should be stored primarily on the machine drive. Data can be stored on your personal computer, but don't remove the data from the computer. When the disk space is short, instrument/computer czars will ask you to remove them.

2.6.3 No drives on any computer are to be shared.

2.6.4 The primary function of the laboratory phone is for business use. Personal calls should be few and ideally less than 5 minutes in order to allow incoming science-related calls.

2.7 Graduation

Upon completion in the group or graduation, the following checklist must be completed.

a) Personal area (desk and bench) must be cleaned and inspected by a supergroup czar. This includes washing all glassware in base bath and accounting for all frequently used chemicals.

b) Cleaning all personal files from INOMAR CENTER computers and transferring all pertinent data/documents to a designated "Alumni" file or to DVD.

c) Give the professor a bound copy of your final thesis (if graduating).

d) Lab notebooks must be stored in shelf/cabinets so that other groups members can refer them if necessary. If you need to keep records (e.g. writing papers), you may keep a scanned copy.

e) Upon completion of their tenure in the group all researchers (rotators, graduates, undergraduates, postdoctoral fellows and others) are required to hand in all data, research results and related records to Dr. Thi.

I, _____, have completed the departure checklist.

Signed (departing member):

Date:

Signed (supergroup czar):

Date:

3. Group Meetings

3.1.1 Group Meetings are held weekly. Be on time and ready to participate. Group meetings consist of research presentations, a question and answer session, and finally discussions of business matters and safety.

3.1.2 Presentation: The slides must have the INOMAR CENTER lab template. Make sure your slides are clear and well illustrated. Consider this as a professional presentation, which serves to highlight your progress since your last presentation.

3.1.3 The group meetings have two main functions: 1) Update the group with latest research update; 2) provide a supportive environment to get feedback from your lab mates and to tackle issues related to your research.

3.1.4 Group meetings are casual and informal but should not be treated lightly. Take advantage of them.

3.1.5 All group members should make it a priority to attend the group meeting.

3.1.6 Each student will present once every 3 weeks and can be considered in that “subgroup”.

3.1.7 The schedule is subject to change. The group meetings czar will keep the schedule updated and communicate the group with any change in them.

3.1.8 Each subgroup has a czar who is in charge of collecting the individual presentations and compiling them to a single file (with a title/name slide for each person). The subgroup czar provides a place and the required instruments for the presentation (laptop, projector, screen, etc)

3.1.9 Include informative, descriptive title. The slides must not be overly crowded in order to convey a clear message. Consider that you are presenting your lab results to your colleagues, so be clear and accurate. The presenter is encouraged to practice their talk before the meeting in order to help create a good flow of the presentation.

3.1.10 Global Meetings are held weekly. Be on time and ready to participate.

4. Collaborations

4.1 Guidelines for Interactions

4.1.1 Professor Dzung, Dr. Thi must be kept "in the loop" on how research data is being used. Data that we produce is confidential and proprietary.

4.1.2 Before sending data to an academic or industrial collaborator, you must send it to Prof Dzung via email, he will determine to decide if the collaborator is appropriate for INOMAR. After receiving his approval, send the data to the collaborator and send Prof. Dzung a copy (cc).

4.1.3 Academic and industrial collaborations have many goals. The interactions need to be monitored by the professor since he is aware of the full scope of the relationship and its history.

4.2 Sending Samples

4.2.1 Samples should be: Dry (not under solvent), in a container sealed with Parafilm, or in a sealed glass tube.

4.2.2 Labels should include: Date prepared, amount, name of student/INOMAR CENTER, and some form of identification (name or code). Include any warnings of any special care or handling.

4.2.3 Packaging: If the sample vial must contain liquid, seal the vial in a plastic bag. Surround the vial with foam. Place the foam-wrapped vial in an appropriately sized box padded with "peanuts" or bubble wrap. Include a document describing any pertinent handling instructions, explanations, or needed sample analysis. Close and securely tape the box.

4.2.4 Mailing: Obtain the appropriate (international, next-day, etc.) FEDEX, UPS, or DHL form. Fill out the form as completely as possible. Have the postal staff check the form.

5. Vacuum Pumps

5.1 Guidelines

5.1.1 It is your sole responsibility to follow these guidelines to ensure a long and healthy pump life. You are responsible for maintaining the pump that has been assigned to your work area.

5.1.2 Vacuum pump czars will keep a log of maintenance for each pump in the lab.

5.1.3 Every three months each pump will receive an oil change from the person assigned to it, regardless of usage. Lab members will have one week to carry out this simple procedure and have it checked off by a pump czar. You cannot sign off on your own pump.

5.1.4 For all other circumstances (poor pump performance, leaking, strange noises, etc.) please immediately shut down your pump and see a pump czar about the problem.

5.1.5 Familiarize yourself with your pump. Read the owners' manual: it is good experience for the future. "A clean pump is a happy pump, so keep your pump smiling"

5.2 Procedure for Pump Oil Change

5.2.1 Be sure that the vacuum pump has been running for at least 10-15 minutes before you change the oil to ensure that it is warm.

5.2.2 Turn off motor for the vacuum pump and remove all hosing attachments to the pump. Elevate the pump; this will aid in removing the last traces of oil and debris from your casing. Use clear tygon tubing to drain the oil into a properly labeled glass waste jug and place in the waste closet after you have finished. Carefully tip the vacuum pump forward to drain as much remaining oil into the waste container as possible, then close the spigot. Look for contaminants settling to the bottom of the jug or drastic darkening or discoloration of the oil. If you see contaminants or noticeable darkening of the oil, you will need to perform the flushing process (see below) until the oil comes out clear. The oil you have just drained from the pump came from the oil case only and there may be contaminants in the actual pumping mechanism. To be sure all contaminants have been removed; the pump mechanism needs to be flushed.

5.2.3 Flushing the pump is carried out by adding pink flushing oil through the intake port (IN) while the motor remains off.

5.2.4 Cover the intake port (IN) with a rubber stopper and, while leaving the exhaust port (OUT) open, turn motor on for 10-15 minutes. Turn off motor.

5.2.5 Open spigot to drain. If solid contaminants or discoloration of the flushing fluid is still apparent, perform steps (c) and (d) until oil is visibly clean.

5.2.6 Once clean flushing oil is drained from pump and spigot is shut, add the appropriate pump oil (belt or direct drive type) through the intake port (IN) until the oil level falls within the target range (shown between two lines on the oil level gauge).

5.2.7 Replace the hosing attachments to the pressure gauge and turn on motor to check the pump's performance. The pump should be pulling down to an appropriate value (10-80 mTorr) with LN2. If you are sure that you flushed and reassembled the pump properly and it is not pulling down adequately or the pump is making odd noises or not running properly, please notify those in charge of vacuum pump maintenance as soon as you notice problems. Do not continue to run the pump; this will only enhance your problem.

5.2.8 If you have completed the oil change successfully, inform the vacuum pump czars so that it can be documented and signed off.

5.2.9 The calibration of the vacuum gauge is mandatory every time pump oil is changed,. Follow the calibration procedure.

6. Calibration of Vacuum Gauges

Gauge calibration should be performed during personal group cleanup days or if a new vacuum tube is installed.

All vacuum lines should be able to attain pressures below 5 microns (5 millitorr). If your vacuum line cannot attain this pressure, attach your vacuum gauge and tube to a system that can for calibration. Ideally, a pressure of less than 1 micron should be achieved for calibration.

6.1 For Duniway (analog) Vacuum Gauges (adapted from Duniway Manual)

6.1.1 With the line power disconnected, adjust the mechanical meter zero (on the front) until the needle indicates "OFF"(Full scale).
6.1.2 Connect the thermocouple tube, matched to the control unit, to your vacuum system, which should be attaining a pressure less than 5 microns (ideally less than 1 micron).
6.1.3 Pump down the system until it is at it's lowest attainable pressure.
6.1.4 Connect the thermocouple cable of the Duniway Stockroom Corp. control unit to the thermocouple gauge tube.
6.1.5 Connect the McLeod gauge to the vacuum line, in its horizontal resting position, and slowly open it to vacuum. Allow 15 minutes for equilibration.
6.1.6 Measure the pressure with the McLeod gauge. See the vacuum Czar or DT for instructions. DO NOT operate the McLeod gauge without instructions or previous experience; the instrument must be used properly.
6.1.7 Plug the line cord into an outlet.
6.1.8 Locate the calibration control adjustment screw on the back of the control unit and adjust it until the meter reads the same pressure on the McLeod gauge. If the pressure is less than 1 micron, adjust the

calibration screw to zero.
6.1.9 Allow the system to stabilize for approximately 15 minutes, and readjust the zero if necessary.
6.1.10 CAREFULLY return the McLeod gauge to its horizontal resting point, making sure that no mercury gets stuck in the gauge. See the Czar or DT for information on how to do this. Close the vacuum to the McLeod gauge, and backfill it with N2. NEVER backfill with N2 if mercury is stuck in the gauge. Disconnect and recap the McLeod gauge.

6.2 For Digivac (Digital Gauge)

6.2.1 With the power disconnected, connect the thermocouple tube to your system.
6.2.2 Pump down the system to its lowest attainable pressure (ideally less than 1 micron, see above)
6.2.3 Connect the thermocouple cable to the gauge and the thermocouple.
6.2.4 Connect the McLeod gauge to the vacuum line, in its horizontal resting position, and slowly open it to vacuum.
6.2.5 Connect the McLeod gauge to the vacuum line, in its horizontal resting position, and slowly open it to vacuum. Allow 15 minutes for equilibration.
6.2.6 Plug the digital gauge into an outlet.
6.2.7 Let the digital gauge equilibrate.
6.2.8 Record the McLeod gauge pressure and the digital gauge pressure. The difference between the two is the calibration offset. For example, if the McLeod gauge measures 2 microns, and the digital gauge measures 10 microns, the cliabration offset for the digital gauge is -8 microns; whatever the digital gauge reads, the true pressure is 8 microns less. This offset can be safely applied for low pressures (less than 100-200 microns).

7. Glove Box Use and Etiquette

1. All products stored in the glove box must be properly labeled; otherwise they will be moved out of the box.

2. Only sensitive reagents can be kept in the glove box long-term. Products that have been in the box for more than two months should be moved out or labeled "long term storage" AND MUST BE APPROVED BY THE CZAR. Sealed glass tubes should be used for private long-term storage outside the box.

3. The bottom shelf of the glovebox is for reagents storage only. Short-term storage products go to the top shelf.

4. If a product is fully characterized and purified and needs to be kept in the glove box, it can be put on the reagent shelf, i.e. it becomes a chemical reagent for community use.

5. Flasks/Schlenk flasks should be kept in the glove box for no more than one week. Transfer your product into a labeled bottle or vial ASAP. If it is necessary to keep a flask in the box for more than one week, contact the czar.

6. Label all samples with the following information:
    a) Product ID
    b) Your name (or initials)
    c) Date the sample entered the box
    d) "long term storage" if appropriate
    e) surface area of material at time it entered box

7. When not in use, keep both antechambers on "evacuate"

8. When bringing something in, make sure to flip the sign to let other people know that the antechamber is in use.

9. Use logbook every time you use the glovebox, bring a chemical in, or bring a chemical out.

10. Please consult standard operating procedure document for details and training by the Czar.

8. X-ray Diffractometers

Preamble:

There are two x-ray diffractometers in the group: Bruker d8 Advance and Bruker d8 Venture. The care and maintenance of the three instruments are responsibility of the group. A proper and careful utilization is therefore obliged. Many group members use these instruments. In order to keep the harmony between users, basic guidelines must be followed. X-ray diffractometers are radiation-producing machines (RPM). Administrative control measures are established in order to control the operation of the machines and to prevent any safety incident. Following it is specified the procedure that each group member must follow in order to be authorized to operate any of these instruments.

8.1 General usage guidelines:

8.1.1 All users must sign on and sign off an instrument according to the policy ascribed to each instrument.

8.1.2 For each instrument, there are designated sample preparation areas. Use only those areas for sample preparation, and clean up after finished. Dispose any generated waste. Do not leave samples in the sample preparation area or inside the diffractometers.

8.1.3 Any problem or malfunctions of the instruments must be reported to the specific instrument czar and the x-ray czar.

8.1.4 If you don't know – ASK. The fast way to resolve a question is to ask someone knowledgeable, so do not attempt to do something with the instrument if you are unsure of what may result.

8.1.5 You must be clean. Any spills or careless use will not be tolerated. Any persons so doing will have to explain their behavior, and their continued use of the facility may become probationary.

8.2 Specific guidelines for Bruker D8 Advance:

8.2.1 Use of the instrument is not permitted by any person until adequate training by the instrument manager has been completed.

8.2.2 A standard operation procedure is available for the operation of the instrument. It can be found in the x-ray lab and as an appendix in this manual. It contains the basic instructions to operate the instrument.

8.2.3 Reservations are made through the on-line calendar created for this purpose.

8.2.4 For the use of the instrument for less than 10 minutes, reservations are not required. For any other period of time, a reservation has to be made. Keep in mind that users may come from other campus locations to use the diffractometer. To avoid unnecessary trips to other members, reserve the time that you will use the instrument. If you reserve time and not use it, delete the reservation.

8.2.5 The diffractometer is available during normal hours (8 am to 6 pm) for periods of time no longer than 2 hours. If a longer data collection is needed, it has to be performed overnight.

8.3 Specific guidelines for Bruker D8 Venture:

8.3.1 Use of the instrument is not permitted by any person until adequate training by the instrument manager has been completed.

8.3.2 A standard operation procedure is available for the operation of the instrument. It can be found in the x-ray lab, as an appendix in this manual, and in the group intranet. It contains the basic instructions to operate the instrument.

8.3.3 Reservations are made through the on-line calendar created for this purpose.
8.3.4 Reservations are in general limited to a maximum of 24 hours. If your data collection requires a longer time, check the availability of the instrument and consult the x-ray czar and the next users before starting it.
8.3.5 Once your data collection has started and you know the estimated completion time, update that info in the instrument calendar.
8.3.6 Each user may sign only once. Wait until your data collection has started before reserving a new time slot.
8.3.7 No trading of positions is permitted. If you cannot make use of your time you must remove your name from the calendar and communicate with the next scheduled user to optimize the utilization of the instrument.
8.3.8 Do not reserve time on behalf of other users. The name that should appear in the reservation is the one of the person who is operating the instrument.
8.3.9 Make a proper use of the log book. Include all the required information, including your name, the sample name and composition, unit cell parameters, crystal size, color and morphology, experimental conditions (temperature, radiation, exposure time, etc.). This should be clear enough for your future references of for other group members. This must be done every time the instrument is used (including just evaluation of unit cell parameters).
8.3.10 When using the low temperature controller, the user is responsible to ensure that the liquid nitrogen level is enough, and to turn the cryosystem off when the data collection is over by using the appropriate command.
8.3.11 The collected images are deleted from the instrument computer during clean up dates. Make your own back up copy if you think you will need to process the data in the future.
8.3.12 Every effort should be made to grow the best crystal possible. Often there is time to work on this while you wait for your turn in the queue. Ultimately the best crystal will yield a better structure. However, a lack of immediate success in this regard should not inhibit attempting to collect data. Seeking advice from the X-ray Czar is strongly encouraged if you feel that your sample is a borderline case.
8.3.13 Upon attaining unit cell parameters a check should be run against the Cambridge Structural Database and Yaghi-group publications so as to avoid collecting data for a known structure. Suspicions should especially arise if your cell is small (< 1000 Å3).
8.3.14 Users may not run samples for colleagues, friends, or any person outside the INOMAR Group unless they have permission from Prof. Dzung.
8.3.15 All new users should recognize that the training required for solving an X-ray structure is a significant time commitment, but it is a highly valuable and worthwhile endeavor- SXRD is the ultimate chemical characterization technique. It expected that those learning this technique be industrious for learning and trying as much as they can on their own. The first step in the process is to consult the X-ray Czar for a list of learning resources.
8.3.16 The structures generally encountered at INOMAR are among the most challenging in chemistry, so don’t be discouraged if a solution doesn’t reveal itself immediately, always keep working with the data and ask for advice.
8.3.17 An X-ray structure is not completed unless it meets IUCR (International Union of Crystallography) guidelines. Finalizing a structure can easily comprise 50-70 % of time committed to its solution. Be prepared to take the time to finish a structure. All structures must pass the IUCR cif checker (checkcif.iucr.org). Any Level A and B alerts must be eliminated and/or addressed in the .cif file.

8.3.18 All structures should be examined using PLATON to check for missed symmetry, or twinning. All unit cells should by examined using CELL_NOW and XPREP to check for rotational and/or merhohedral twinning. Finding these problems at the outset of a refinement will make your final structure resolve much faster.

9. Preliminary Characterization of Compounds and Organization of Data

9.1 Material Synthesis

a) References used in the synthesis of the materials must be recorded properly.

b) Reaction scheme showing the chemical structures, amounts, and reaction conditions for each step must be drafted using appropriate software (e.g. ChemDraw) according to JACS format.

c) Written form of the synthesis procedure for both starting materials and MOF/MOP/COF/ZIF etc. must be prepared in accordance with JACS format.

d) Synthesis procedure must be elaborated with all necessary details to the extent that "a high school student can follow the procedure".

9.2 General Characterization of Materials

a) All spectra, measurements, curves must be labeled with the compound's code, name, formula, date of collection, and history of sample if needed.

b) All spectra peaks must be clearly labeled and assigned if possible. Comparison to data from precedent literature must be made to confirm the formation and/or structure of the target materials.

c) Original data and spectra must be submitted in both hard copy and electronic copy. A Xeroxed copy must be kept for individual records.

9.3 Characterization of Organic Linkers

a) ^{1}H NMR of the ligands must be obtained and processed properly (e.g. phasing, peak labeling, assigning and integration). NMR spectra of ^{13}C, ^{11}B, ^{15}N and other nuclei must also be measured and processed if possible.

b) Elemental analysis must be performed and reported accordingly.

c) FT-IR spectra must be measured and assigned properly

d) UV-Vis, MS and other analysis must also be performed and explained if necessary.

9.4 Characterization of MOF/MOP/COF/ZIF

a) Experimental and simulated PXRD (powder X-ray diffraction) must follow the same labeling scheme as previously described (including indexing and comparison of d-spacings).

b) Differences in simulated and experimental PXRD must be explained properly.

c) SXRD (single-crystal X-ray diffraction) must be measured if possible and crystal structure must be solved accordingly (e.g. unit cell and space group determination, structure solving and refinement).

d) An ORTEP representation with all atoms labeled must be included in the structure report along with a table of crystal data and refinement.

e) Elemental analysis must be accompanied by calculated values and experimental values with an empirical formula and the relationship of this formula to that found by other techniques such as X-ray single crystal. Any differences should be reconciled.

f) Thermal gravimetric analysis (TGA) must be measured and labeled. Weight loss steps and each plateau must be analyzed and interpreted according to SXRD and elemental analysis data.

g) N_2 or Ar adsorption isotherm must be measured and surface area derived from BET and/or Langmuir isotherm model using data in an appropriate pressure range must be reported.

h) H_2, CO_2, CH_4 and other isotherms should also be measured if necessary.

i) FT-IR, solid-state ^{1}H and ^{13}C NMR, MS, UV-Vis and other analysis should also be performed if necessary.

10. SemiAnnual Cleanup Days

10. 1 Personal areas (Day 1)

10.1.1 You are not permitted to leave until checked out by the cleanup czars.

Glassware

10.1.2 Clean all glassware lying in your hood or bench area, including vials and utensils. Cleaning entails soap and water wash, base bath, then water and acetone rinses. All clean glassware must be returned to community areas. The reaction ovens are not to be used for drying glassware.

10.1.3 Ongoing reactions and stored samples must be kept to a minimum. It will not work if you pretend that a large array of such vials and containers are being monitored.

10.1.4 All stored but unused and unimportant samples must be sent to waste using proper waste deposition procedures. This also applies to tubes and other reactions. If you have unknown samples in your area, label them to the best of your knowledge and put them in the proper waste receptacle.

10.1.5 Those working with undergraduates are entirely responsible for overseeing their respective areas.

Hoods, bench tops, shelves and drawers

10.1.6 Those with a Schlenk line must clean all glass parts with soap and water, base bath and then water, ethanol/isopropanol, hexanes rinses (KEEP VITON O-RINGS FROM ACETONE).

10.1.7 If you are behind on changing your vacuum pump oil, you need to change the oil. Clean the outside of your pump and the area around it with a hexanes-soaked towel to remove excess dirty oil.

10.1.8 All items on shelves and benches must be placed in their proper places and fully accounted for. All chemicals must be returned to their original storage places.

10.1.9 Hood and bench surfaces must be thoroughly cleaned with hot soap and water and hexanes (as necessary). You should polish the surface with a small amount of mineral oil. At the end of the day, no more than 12 items should be left out on the hood. Items used for ongoing reactions are excluded, but everything must be neatly organized by the time you leave the lab.

10.1.10 All personal drawers should contain minimal items.

Desk area

10.1.11 It must be tidy. Postings of profanity, coarse language, or other material that can be construed as offensive to others is strictly prohibited.

Waste

All containers with hazardous waste must be labeled in accordance with the rules listed in the previous section.

10.1.11 Solids and liquids should be separated.

10.1.12 All filled waste containers should be placed at the waste pickup site in each room. This should be done when a container is 75% full.

Personal computer space (do this last)

10.1.13 Back up data sets and delete those data files (do not delete anything that has not been backed up). Work folders may be kept.

10.1.14 Your personal folder should not have any material unrelated to your project.

10.2 Community areas (Day 2)

10.2.1 You are not permitted to leave until checked out by the cleanup czars.

10.2.2 For each person and their respective responsibilities (according to responsibility list): Make sure the instrument or object is operational, properly standardized and cleaned. In addition, make sure that the community areas in these modules are clean and organized.

10.2.3 A designated person will lead the cleanup for each room. This person is not expected to do the entire cleanup in these rooms, but will be responsible for seeing to it that the work gets done. Food items left in the food refrigerators at the end of the second day will be discarded.

Safety Review (Both Days)

10.2.4 You are not permitted to leave on Day 2 until checked out by the safety czar(s).

10.2.5 Each group member is required to review the safety policy, answering all questions presented, and sign an updated safety checklist.

11. Access to CSD database

11.1 Cambridge structural database (CSD) is the world repository of small molecule crystal structures.

11.2 The database is installed in the x-ray computer and other lab computers. It is updated quarterly.

12. Read and understood

I have read this document in its entirety, understand and fully agree to abide by the safety guidelines and laboratory protocol contained within.

Signature:---------- Date:----------