SAFETY IN THE CHEMICAL LABORATORY

SAFETY MANUAL
DEPARTMENT OF CHEMISTRY
ILLINOIS WESLEYAN UNIVERSITY

Revised by Dr. Mohan (May 2001)

Safety is a very important component of your research career in chemistry. All workers in the laboratory must work in a manner that is in the interest of their safety and that of their co-workers.

This manual includes general guidelines on safe practices for common laboratory

1. FIRE HAZARDS

A major hazard one can face in the chemical laboratory is that of fires. Please familiarize yourself with the location and types of fire extinguishers and fire alarm pull stations. In the event of a fire, do not put yourself in danger to extinguish the fire.

- If there is a major fire, pull the fire alarm located at the end of the hall. This will alert the campus security who will then alert the local fire department. If you can call security (through the desk at 3034), do so but not at the risk of risk to your self. Do not call the fire department directly unless you are unable to reach security. If it is a small fire you feel you can control, do not pull the fire alarm.

It is important to recognize the various types of fires and methods to extinguish them.

Class A Fires: Combustible solids, paper, rubber, plastics. Combustion can result in release of toxic gases.

The most convenient method is to use water. Can also use dry chemical extinguisher, and CO_2 but these result in spreading of the ash over a large area.

Class B Fires: Solvent fires.

These pose the greatest risk, especially in the organic chemistry lab. A measure of the flammability of the liquid is given by "flash point" which is defined as the temperature at which a liquid gives rise to ignitable vapors. Any liquid with a flash point < 15 °C should be regarded as dangerously flammable.

Table 1.

Flash Point of Common Solvents:

Pentane	– 49 °C	Ethyl acetate	– 4.4 °C
Diethyl ether	– 45 °C	Toluene	4 °C
Carbon disulfide	– 30 °C	Acetonitrile	6 °C
Hexane	– 23 °C	Methanol	7 °C
Acetone	– 18 °C	1,4-dioxane	12 °C
Tetrahydrofuran	– 17 °C	Ethanol	12 °C

Carbon disulfide has the very low auto ignition point of 100 °C. The vapors can ignite up on contact with a hot water bath !! Avoid use of CS_2 !

If the fire is contained in a beaker or a small vessel, it can often be extinguished by covering the vessel with a wire gauze or addition of sand or dry ice. If the fire is in a larger vessel or has spread, dry ice is very effective. Dry chemical extinguisher is also useful. CO_2 extinguishers

produce a jet of foam which can result in overturning of the vessel and sometimes aid in spreading of the fire !

Class C Fires: Electrical equipment:

Never use water for these fires even if the main is turned off. Many equipment store charge. If power cannot be turned off, dry chemical or inert gas must be used.

Class D Fires: Metal (K, Na, Mg, Li, Al) and Metal hydrides (NaH, KH, $LiAlH_4$, etc)

These fires cannot be extinguished using water, CO_2 or volatile hydrocarbons. Inert powder (sand or talc) must be used. It is best to use Metal X extinguishers.

Some Common Fire Sources In the Lab:

A. Open Flames: Open flames should almost never be used in the lab, especially in the organic chemistry lab. If a Bunsen burner is used, it should be turned off when not in use.

B. Sparks: Volatile and flammable solvents should not stored in open containers nears sources of sparks which include vacuum pumps, drying ovens, thermostats etc.

C. Oxygen cylinders: A leaking oxygen cylinder can lead to an increase in oxygen concentration in the atmosphere which will cause a dangerous fire if a flammable substance and a ignition source are present. All compressed oxygen cylinders should be checked for leaks periodically.

D. Sodium residues: Sodium residue should never be destroyed by addition of water: even if there is only very small quantities. Large quantities are best destroyed by slow addition of 2-propanol to the sodium contained in a round-bottomed flask equipped with a water cooled reflux condenser. Smaller quantities (1-2 g) can be destroyed by very cautious addition of methanol using a similar set-up.

E. Organometallics: These pose a serious fire risk. They must always be handled in an inert atmosphere.

2. EXPLOSIONS

Explosions are usually accompanied by fire or vice-versa ! In addition to fires, there are many chemicals which can lead to an explosion when handled improperly. Some specific dangers of explosions are listed below:

A. Peroxides in ether solvents:

One of the most common cause of explosion in the organic lab is peroxides. Many a laboratory has been destroyed by this ! Simple dialkyl ethers as well as cyclic ethers such a 1,4-dioxane and tetrahydrofuran form less volatile peroxides when exposed to air and light. When these solvents are distilled, the pot residue is enriched in the peroxide and eventually a violent explosion can result. To avoid this risk:

(i) such solvents should not be stored for long in half empty containers.

(ii) it is best to buy smaller containers of ether rather than buy larger containers (which are less expensive) and store for prolonged periods.

(iii) it is best not to distill these solvents when old. But if distillation must be carried out, test for peroxide before distilling. If peroxides are detected, they must be destroyed. The process of drying these solvents with

3. Some Dangerous Inorganic Chemicals:

Strong Acids:

All of the following give off very harmful vapors and also react violently with bases. They should always be handled with gloves and in the hood. Adequate eye protection must also be worn.

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4. CHEMICAL HAZARDS:

Given below is a list of some dangerous chemicals that are often encountered in the lab. This is not a complete list of such chemicals ! It simply inludes the more common reagents. Many of these are useful compounds. While their useful properties should be taken advantage of, it is important to be aware of the risks so that adequate precautions can be taken while handling them.

The following are severe lachrymators (cause copius watering of the eyes)

acetyl chloride
acrolein
allyl alcohol
allyl chloride
benzyl chloride
diketen
dimethyl acetylenedicarboxylate
benzoyl chloride
bromoacetone (easily produced by mixing bromine with acetone)
oxalyl chloride

The following are a major explosion hazard especially when heated:

- sodium azide
- diazomethane
- acetylene salts
- p-toluenesulfonyl azide
- diazonium salts
- perchlorates

The following are extremely toxic by ingestion and can be fatal in even small doses:

- aromatic amines (aniline and its substituted derivatives)
- arsenic and its compounds
- hydrogen sulfide (this gas with the odor of rotten eggs cannot be detected at higher concentrations !)
- mercury chloride
- nitric oxide and nitrogen dioxide
- osmium tetroxide (severe hazard to eye)
- oxalic acid and its salts
- phenols and aromatic nitro compounds
- selenium and its compounds
- sodium and potassium cyanide
- thallium acetate and other thallium salts
- vanadium pentoxide

The following substances have very harmful cumulative effects which manifest after prolonged exposure over a relatively long period. If you will be working with these for long periods of time, biological monitoring of the body may be necessary.

- benzene (it has a mildly pleasant odor; if you can smell it, you are inhaling dangerous quantities)
- isocyanates (in particular toluene isocyanate)
- lead and its compounds
- mercury and mercury compounds

The following are known or suspected carcingoens. If you must use these, make every effort to wear gloves, handle them only in the hood and wear protective clothing. Label all vials and flasks containing these appropriately

Amines

1,1-dimethylhydrazine
hydrazine
methyl hydrazine
1- and 2-napthylamines

Nitroso compounds

All nitroso compounds are potential carcinogens.

Alkylating agents

aziridine
Bis(chloromethyl) ether
diazomethane
epichlorohydrin
methyl iodide
propiolactone

Aromatic hydrocarbons

benzene
benzo[a]pyrene
dibenz[a,h]anthracene
7,12-dimethylbenz[a]anthracene

Halogenated hydrocarbons

carbon tetrachloride
chloroform
1,2-dibromoethane
hexachlorobutadiene
vinyl chloride

Phosphorous and Sulfur compounds:

hexamethylphosphoramide
1,3-propanesultone (3-hydroxy-1-propanesulfonic acid)
thioacetamide and thiourea

5. The following section discusses safety aspects associated with some common lab procedures:

1. The following statements concern the use of a rotary evaporator:

(a) The body of a rotovap should not be protected with a safety net since this can obscure the vision of the condenser and the condensates. Say True or false.

This is false. Implosion is a serious hazard. Safety net does not obscure vision. The risk from an implosion is real and dangerous.

(b) What is the largest size flask that should be used in most rotovaps ?

One liter flask.

(c) Can a Erlenmeyer flask be evacuated ?

No, Flat surfaces vessels should never be evacuated unless they are specifically designed for the purpose (such as filtration flasks)

(d) Is there a greater risk in evacuating a rotovap with a vacuum pump (<0.5 mm Hg) than with a water aspirator (20 mm Hg?

No. the risk is the same. Both pressures are quite far from atmospheric pressure. It is the pressure differential that is important.

2. Why should contact lenses be never worn in the laboratory ?

Soft contact lenses will absorb organic vapors like methanol, chloroform etc. If chemicals enter the eye, then eye cannot be easily washed due to contact coverage. Only ANSI-Z87.1 approved safety glasses must be worn in the laboratory.

3. Familiarize yourself with the location of fire extinguishers,

research groups, **LAH should not be used to dry ethers**.

10. Describe the appropriate clothing that must be worn in a lab.

Loose clothing should never be worn in the lab. They pose a greater fire hazard. Long hair also poses a similar risk. Shorts and sleeveless dress offer less protection than long pants and full sleeve shirts. If a corrosive chemical is being handled, it is best to wear a laboratory coat. Shoes must be worn at all times in the lab. Open toed shoes and sandals offer little protection against spills and hence are not permitted in the lab. Hosiery should not be worn since they "melt" upon contact with acids.

11. The following section describes the use of gloves.

Leather gloves are best for handling broken glass and for inserting tubing into corks but they do not offer protection from chemicals.

There are many kinds of gloves available in the market. Check with your research advisor to see if a particular kind is more suited for the type of work you are doing. Latex surgical gloves are of little value in the chemical laboratory and should not be worn. It is important not to spread chemicals and spills with gloves: Do not handle door knobs, items in stock rooms and instruments with contaminated gloves.

12. When should a blast shield be used ?

Whenever a potentially explosive reaction is being carried out (such as generation of diazomethane) or the use of a pressurized equipment is involved, a safety shield should be used to provide added protection against an implosion.

13. When should the safety shower be used ?

It is unlikely that you will ever need to use one. But in the event of a major spill on your face, neck, eyes, head or shoulders, use the safety shower immediately. Do not worry about being modest ! Contaminated clothing should be immediately removed. Hence it is a good idea to have lab coats available in every lab !

14. What are the five hazard classes recommended by Environmental and Safety for separating chemicals ?

Flammables
Oxidizers
Acids
bases
Reactives

15. How should glass bottles containing solvents be carried around in the hallways ?

A bottle carrier should always be used in the halls, elevators and stairwells.

16. What is the best container to collect waste solvents ?

The EPA requires that waste solvents be collected in polyethylene jerri cans. Metal cans rust easily while glass bottles are easily broken.

17. Flooding: Major damage to equipment has often resulted from flooding. How can this be avoided ?

- **Do not use tubing that is too old or brittle.**
- **Don't use pure gum rubber tub**

- **<u>Absorb the spill:</u> Absorb the spill with a spill absorbent.**

- **<u>Cleanup:</u> Scoop he mixture into a plastic bag and label appropriately. Turn it into the stock room and fill up a waste disposal form.**

21. What special precautions must be observed when running overnight reactions or reactions that have to be left unattended for some length of time ?

- **An index card containing the following information should be posted clearly outside the hood or near the reaction site.**

- Your name and telephone contact number

- Contents of the flask. This is useful if someone else has to respond to an emergency situation associated with your reaction.

- If water is being used through a hose, make sure it is secured with hose clamps. Also insure that the sink is <u>not</u> clogged with debris.

- Oil baths should not be heated overnight unless a temperature controller is installed in the bath.

<u>WHAT TO DO IN CASE OF A MAJOR FIRE ?</u>

PULL THE FIRE ALARM AND EVACUATE THE BUILDING IMMEDIATELY. DO NOT PUT YOUR LIFE IN JEOPARDY.

<u>WHAT TO DO IN CASE OF A MAJOR ACCIDENT THAT SEEMS LIFE THREATENING (SUCH AS A SEVERE CUT OR A CHEMICAL BURN)</u> ?

- **IF THE ACCIDENT INVOLVES YOUR LAB MATE OR SOMEONE AROUND YOU, KEEP THEM CALM. CALL CAMPUS SECURITY THROUGH THE PHONE IN THE HALLWAY (DIAL 3034 TO REACH MEMORIAL DESK WHO WILL PUT YOU THROUGH TO SECURITY). IF THERE IS NO IMMEDIATE RESPONSE, CALL 911 BY DIALING 9-911. DO NOT HANG UP TILL THEY HAVE THE**

NECESSARY INFORMATION. TELL THEM YOU ARE IN THE SCIENCE BUILDING OF ILLINOIS WESLEYAN UNIVERSITY ON E. BEECHER STREET.

- **You should <u>not</u> be working alone in the lab. If at the moment of the accident there is no one in your vicinity, call for help by screaming ! If you can make it to the phone, dial 3034. Stay calm. If it is a burn, keep injured body part under cold water.**

- **DO NOT ATTEMPT TO MOVE THE INJURED PERSON. KEEP THEM CALM. THE ONLY EXCEPTION TO THIS IS IF THE ACCIDENT ALSO INVOLVES A FIRE.**