CHEMICAL HYGIENE PLAN

Laboratory of Dr. Emily Pentzer

Millis Hall 423 South end

December 30, 2017

Emergency: 368-3333

PI Pentzer: 224-420-2721

Other contact information

Safety Services: 368-2907

Radiation Safety: 368-2906

Health Center: 368-2450

DOES Safety Services Office	SAFETY SERVICES STANDARD OPERATING PROCEDURE CHEMICAL HYGIENE PLAN	CASE/SSO-003S Rev: Orig Rev Date: 4/6/2004

PURPOSE
To determine the scope, elements, and framework of the Chemical Hygiene Plan.

REFERENCES
29 CFR 1910.1450 (OSHA Laboratory Standard)

SCOPE
The Chemical Hygiene Plan (CHP) applies to laboratories when ALL of the following criteria are met:

1. Chemical manipulations are carried out on a "laboratory scale";
2. Multiple chemical procedures or chemicals are used;
3. The procedures involved are not part of a production process, nor in any way simulate a production process; and
4. Protective practices/equipments are available and in common use to minimize the potential for employee exposure to hazardous chemicals.

If ALL of the above criteria are not met, the lab may fall under the OSHA Hazard Communication Standard. For example, an instrument lab, using only solvents and cleaning solutions to clean instruments, will be covered by the Hazard Communication Standard and a HCP would be required.

Contact Safety Services (x2907), for any consultation regarding your work area.

PROCEDURE
1. All employees, including PI's and supervisors, must attend the DOES Lab Safety and Regulated Chemicals training. Call Safety Services (x2907) to register.

2. Complete the CHP for a lab-specific work area and send a copy to DOES.

3. The PI or Supervisor must thoroughly train employees on the contents of the Laboratory Safety Manual, the Physical Safety Manual, and the contents of the lab-specific CHP. This lab-specific training must be completed annually or whenever a new hazard is introduced into the laboratory.

4. The PI or Supervisor must document (in CHP) the lab-specific training of all relevant safety materials.

5. Update the CHP annually or whenever there is a change and submit the cover page, the review date page, and any changes to DOES.

6. All lab employees, including PI's and Supervisors, must complete an annual, on-line, retrain for the Lab Safety and Regulated Chemicals training. The on-line programs are available at http://does.cwru.edu.

NOTE: A copy of the CHP, including up-to-date information and training log sheets, shall be kept in a location known to all employees. Safety Services will review the CHP during yearly inspections and may ask for corrections.

ATTACHMENTS
Blank Chemical Hygiene Plan

Chemical Hygiene Plan

The Chemical Hygiene Plan applies to your laboratory only if you meet ALL of the following criteria:

(i) Chemical manipulations are carried out on a "laboratory scale";
(ii) Multiple chemical procedures or chemicals are used;
(iii) The procedures involved are not part of a production process; and
(iv) "Protective laboratory practices and equipment" are available and in common use to minimize the potential for employee exposure to hazardous chemical.

IF YOUR LABORATORY DOES NOT MEET ALL OF THE ABOVE CRITERIA, REFER TO THE HAZARD COMMUNICATION PLAN OR CONTACT DOES (X2907) FOR CONSULTATION.

OSHA Definitions:
Laboratory: a facility where the "laboratory use of hazardous chemicals" occurs. It is a workplace where relatively small quantities of hazardous chemicals are used on a non-production basis.

Laboratory scale: work with substances in which the containers used for reactions, transfers, and other handling of substances are designed to be easily and safely manipulated by one person. Excludes those workplaces whose function is to produce commercial quantities of materials.

Chemical Hygiene Plan

The Chemical Hygiene Plan (CHP) is a laboratory specific document that details the safety procedures in use in a specific laboratory. The goal of the CHP is to provide the necessary guidance to the laboratory staff required to maintain a safe work environment while dealing with hazardous materials.

The Primary Investigator (PI) of a laboratory is responsible for maintaining a safe work environment for the laboratory staff. As such, the PI is given the title of Chemical Hygiene Officer (CHO). The CHP is required by federal law to provide a CHP to the laboratory staff that is specific to the laboratory for which it was written. Further, the CHO is responsible for providing training in the CHP sufficient to allow the laboratory staff to implement the CHP. This training is required initially upon employment, and when there is a change in the plan or annually, whichever is the shorter time interval.

Upon employment at CASE, all employees dealing with or possibly having exposure to hazardous materials are given an overview OSHA Laboratory Standard class at the DOES office. This training is NOT substitute training for the laboratory specific training to be given by the CHO. DOES publishes a Laboratory Safety Manual and Physical Safety Manual to be used as a reference in producing a CHP. These manuals are available on the DOES website (http://does.cwru.edu).

Date: _____

Primary Investigator's (CHO) Name: _____
Please Print

Primary Investigator's Email: _____

Primary Investigator's Phone: _____ Fax: _____

Primary Investigator's (CHO) Signature: _____

Laboratory Location: Building: _____ Room: _____

Laboratory Location: Building: _____ Room: _____

Laboratory Location: Building: _____ Room: _____

Complete and send a copy to:

DOES
Service Building, First Floor
Location Code: 7227
Attention: Safety Services

Revised: 4/6/2004

Review Date

Review the CHP annually and/or whenever there are any changes in procedure. Submit a copy of the title page, this sheet, and any changes to the DOES office.

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

Review Date: _____ Changes: _____

_____

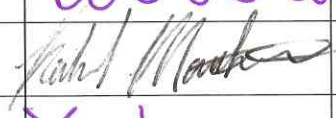
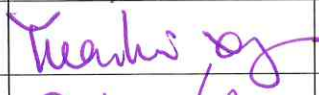
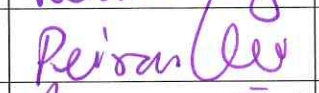
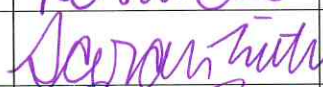
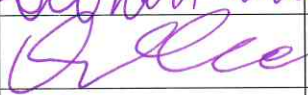
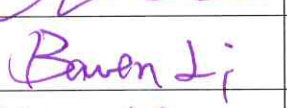
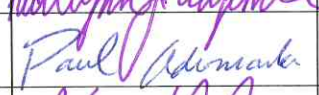
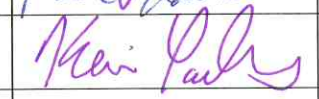
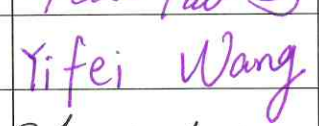
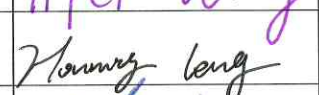
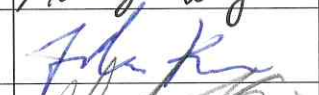
2018

Annual CHP Training Log

DATE:_____ LOCATION: Building:_____ Room:_____

Primary Investigator (CHO):_____

PLEASE CLEARLY PRINT ALL INFORMATION REQUESTED BELOW EXCEPT FOR YOUR SIGNATURE

Name	Signature	Phone	Email
Emily Pentzer		224-420-2721	EBP24
Rachael Matthews		443-546-7762	RXM453
Yuanhui Xiang		216-246-5284	YXX220
Peiran Wei		216-272-0239	PXW182
Sarah Mitchell		814-806-5643	SMM253
Qinmo Luo		216-857-8316	QXL135
Bowen Li		216-703-7630	BXL316
Katelynn Edgehouse		440-840-5280	KJE17
Paul Advincula		713-969-9003	PAA33
Kevin Pachuta		248-719-4316	KGP22 X
Yifei Wang		~~224-420-2721~~ 216-804-6174	YXW1182
Huoming Leng		206-257-8198	HXL801
John Kwon		216-333-5505	JGK50
Christina Hemmingsen		614-425-9922	CMH173
Esther Yoo		516-306-0510	EHY7

Annual CHP Training Log

DATE:_____ LOCATION: Building:_____ Room:_____

Primary Investigator (CHO):_____

PLEASE CLEARLY PRINT ALL INFORMATION REQUESTED BELOW EXCEPT FOR YOUR SIGNATURE

Name	Signature	Phone	Email
Lee Kendall		479-381-0621	LMK115
Emily Glasser		330-204-3266	EMG78
Madelyn McMillen	Madelyn McMillen	440-318-4017	mmm273
Louisa Wang	Louisa Wang	330-937-3542	lwang21@hb.edu

Outline

Lab Procedures and Safety Precautions: Work with Chemicals

To enter the Pentzer lab in a non-emergency situation, people must have permission from PI Pentzer

Protective Equipment

The safety of a lab can only be realized when all individuals take it upon themselves to create and maintain a safe laboratory environment. One obvious way for workers to realize this is by using personal protective equipment (PPE) at all times. Furthermore, no work is to be performed when in the laboratory alone.

All workers are required to wear pants or skirts that cover the entirety of their legs and close toed shoes. Long hair should be tied back and no hanging scarves, necklaces, or ties are permitted. All workers are required to wear approved lab goggles when in the laboratory, even when walking through the area. A suitably sized, buttoned lab coat of an approved flame resistant material is required when in the laboratory area. Gloves are to be worn when working in the laboratory; the material of the gloves should be compatible with the chemicals to be worked with, as determined by a fair or better rating on the chart that follows. Respirators or masks should not be used unless approved by Dr. Pentzer. Face shields are available, but not required, when working with liquid nitrogen and "base baths" (isopropanol, water, and potassium hydroxide solutions). When working with base bath, neoprene gloves are to be used.

All workers should know the location of fire extinguishers of each class, and their appropriate uses, as well as eye washes and safety showers**. <u>Do not wear lab coats or gloves in the hallway</u>.**

Visitors:
Outside traffic in the lab should be kept to a minimum. However, **any visitor is required to wear safety goggles, which are available at each entrance**. No untrained individual is to work with any chemical. This includes individuals there for maintenance and deliveries.

Glove box:
When working in the glove box, all workers should wear a lab coat, goggles, and cotton lining gloves. The butyl dry box gloves alone are not appropriate for all solvents; for this reason, when the butyl gloves are not rated for the solvent in use (see glove compatibility charts), workers are required to wear additional gloves *over* the butyl glove box gloves (i.e., inside the box). For example, viton gloves should be used with chloroform. Before each use of the glove box, workers should inspect the gloves for any signs of punctures, tears, or chemical damage. If found, these problems should be addressed by patching the gloves or replacing them before the glove box is used.

Gloves

Gloves: Please use disposable gloves compatible with the chemicals being used. A large chart of compatibility is available in lab, or can be found on the internet.

Chemical Name	NFPA Health Rating	Nitrile	Natural Rubber	PVC	Recommended Alternate Material
ACETALDEHYDE	3	P	G	P	
ACETIC ACID (GLACIAL)	3	F	G	P	
ACETIC ANHYDRIDE	3	F	G	P	
ACETONE	1	F	G	P	
ACETONITRILE	2	F	F	P	Butyl (E)
ACRYLIC ACID	3	G	G	P	
AMMONIUM ACETATE		E	E	G	
AMMONIUM CARBONATE		E	E	E	
AMMONIUM FLUORIDE, 30-70%	3	E	E	G	
AMMONIUM HYDROXIDE, 30-70%		E	E	E	
AMMONIUM HYDROXIDE, <30%		E	E	E	
AMYL ALCOHOL	1	E	G	G	
ANILINE	3	F	G	G	
AQUA REGIA		P	P	F	Neoprene (F)
AZT			G		
BENZALDEHYDE	2	P	F	P	Butyl (E)
BENZENE	2	F	P	P	Viton (G)
BORIC ACID		E	G	E	
BROMOPROPIONIC ACID		F	G	G	
BUTYL ACRYLATE	2	P	P	P	PTFE (G)
BUTYL CELLUSOLVE		G	G	P	
CALCIUM HYDROXIDE		E	E		
CARBON DISULFIDE	3	G	P	P	
CARBON TETRACHLORIDE	3	P	P	F	Viton (G)
CHLOROBENZENE	2	P	P	P	Viton (G)
CHLORODIBROMOMETHANE		P	P	F	Viton (G)
CHLOROFORM	2	P	P	P	PVA (G)
CHLORONAPTHALENES	1	P	P	P	Viton (G)
CHROMIC ACID	3	F	P	G	
CISPLATIN		G	G	P	
CITRIC ACID, 30-70%		E	E	E	
CYCLOHEXANE	1	E	P	P	
CYCLOHEXANOL	1	E	G	E	
CYCLOHEXANONE	1	P	P	P	Butyl (G)
CYCLOHEXYLAMINE	3	P	P	P	
DI-N-AMYLAMINE	3	E	P	P	
DI-N-BUTYLAMINE	3	E	P	P	
DI-N-BUTYLPHTHALATE	0	E	F	P	
DI-N-OCTYLPHTHALATE	0	E	F	P	
DIACETONE ALCOHOL	1	G	F	P	
DIALLYLAMINE		P	P	P	Viton (G)
DICHLOROACETYL CHLORIDE	3	P	P	P	Viton (G)
DIESEL FUEL	0	E	P		
DIETHANOLAMINE	1	E	E	E	
DIETHYLAMINE	3	G	F	P	
DIETHYLENE GLYCOL	1	E	E		
DIETHYLENETRIAMINE	3	P	P	P	Neoprene (G)
DISOBUTYL KETONE	1	G	P	P	
DISOBUTYLAMINE	3	E	P	P	
DIMETHYL ETHER		G	P	P	
DIMETHYL SULFOXIDE (DMSO)	1	G	E	G	
DIMETHYLACETAMIDE	2	F	G	P	
DIMETHYLFORMAMIDE (DMF)	1	P	P	P	Butyl (G)
DIOXANE	2	P	P	P	Butyl (G)
EPICHLOROHYDRIN	3	P	F	P	Butyl (G)
ETHANOL	0	G	G	G	
ETHYL ACETATE	1	P	F	P	Butyl (G)

CHEMICAL RESISTANCE AND BARRIER GUIDE

Safeskin Corporation's Nitrile, Natural Rubber Latex and Polyvinyl Chloride (PVC) gloves are thin gauge disposable products designed to provide barrier protection and tactile sensitivity to the wearer. Our gloves are not designed for applications involving prolonged, direct exposure to chemicals. Our intent in providing this chemical compatibility information is to provide a guideline for *use of our gloves in applications where incidental splash exposure to various chemicals may occur.*

The chemical compatibility information on this chart is intended to provide general information about the reaction of Nitrile, Natural Rubber Latex and Polyvinyl Chloride glove films to the commonly used chemicals listed.

The ratings scale takes into consideration three primary factors:

1) the ability of the chemical to *permeate (pass through) the glove film;*
2) the ability of the chemical to *degrade (break down) the physical structure of the glove film;*
3) the *risk* that contact exposure to the chemical poses to the glove wearer.

Safeskin recommends that you USE CAUTION AT ALL TIMES.

- Verify that your gloves are compatible with your specific applications, processes and materials before using.
- When performing processes where gloves will receive prolonged, direct exposure to chemicals, use a glove specifically designed for chemical handling.
- Avoid the risk of exposing your workers, products and facilities to chemical cross-contamination: immediately dispose of gloves after contact with chemicals.
- Double gloving provides additional barrier protection and allows the outer glove to be disposed of after contact with chemicals without exposing the hand.
- Do not use powdered gloves with substances known to pose inhalant hazards.
- If you have any questions about using Safeskin gloves or the information on this chart, please contact our Technical Affairs Department at (800) 462-9993.

Chemical Name	NFPA Health Rating	Nitrile	Natural Rubber	PVC	Recommended Alternate Material
ETHYL ETHER	1	G	P	P	
ETHYLENE GLYCOL DIMETHYL	2	F	F	P	Butyl (G)
ETHYLENE DICHLORIDE	2	P	P	P	PVA (E)
ETHYLENE GLYCOL	1	E	E	G	
FORMALDEHYDE, 30-70%	3	E	G	E	
FORMIC ACID	3	G	E	E	
FREON 113 OR TF		E	P	F	
FREON TMC		F	F	P	PVA (E)
FURFURAL	3	P	P	P	Butyl (G)
GASOLINE, 40-50% AROMATICS	1	E	P	P	
GASOLINE, UNLEADED	1	G	P	P	
GLUTARALDEHYDE, <5%		G	G	F	
GLYCEROL		E	E		
HEPTANES	1	E	P	P	
HEXANE	1	E	P	P	
HYDRAZINE	3	E	F	E	
HYDROCHLORIC ACID, <30%	3	G	E	E	
HYDROFLUORIC ACID, <10%	4	G	G	F	
ISOBUTYL ALCOHOL	1	E	P	F	
ISOOCTANE	0	E	P	P	
ISOPROPYL ALCOHOL	1	E	E	G	
ISOPROPYLAMINE	3	P	P	P	Teflon (G)
JET FUEL, <30% AROMATICS (JP-8)	1	G	P	P	
KEROSENE		E	P	F	
LACTIC ACID		E	E	E	
LAURIC ACID		E	E	F	
MALATHION, 30-70%		G			
MALEIC ACID		G	G	E	
METHANOL	1	F	F	F	Neoprene (G)
METHYL ACETATE	1	P	P	P	Butyl (G)
METHYL ETHYL KETONE	1	P	P	P	Butyl (E)
METHYL ISOBUTYL KETONE	2	P	P	P	Butyl (G)
METHYL METHACRYLATE	2	P	P	P	PVA (E)
METHYLENE CHLORIDE	2	P	P	P	PVA (G)
AMYL ACETATE	1	F	P	P	Butyl (G)
BUTYL ACETATE	1	F	P	P	Butyl (G)
BUTYL ALCOHOL	1	E	E	F	
N-METHYL-2-PYRROLIDONE (NMP)	2	P	E	P	
N-NITROSODIETHYLAMINE		P			Butyl (G)
PROPYLENE GLYCOL	1	E	E	F	
NAPHTHA, <3% AROMATICS	1	E	P	F	
NITRIC ACID, <30%	3	G	G	G	
NITRIC ACID, 30-70%	3	P	P	F	Neoprene (G)
NITROBENZENE	3	F	F	P	Butyl (G)
NITROETHANE	1	P	G	P	
1-NITROPROPANE	1	P	F	P	Butyl (G)
2-NITROPROPANE	1	P	P	P	Butyl (G)
OCTANE	0	G	P	P	
OCTYL ALCOHOL	1	E	E	F	
OLEIC ACID	0	E	G	G	
OXALIC ACID	3	E	E	E	
PALMITIC ACID		G	F	G	
PCB (POLYCHLORINATED BIPHENYLS)	2	G	P		
PENTACHLOROPHENOL	3	G	P	F	
PENTANE	1	E	P	P	
PERCHLORIC ACID, 30-70%	3	F	F	F	Neoprene (F)
PERCHLOROETHYLENE	2	G	P	P	

Chemical Name	NFPA Health Rating	Nitrile	Natural Rubber	PVC	Recommended Alternate Material
PEROXYACETIC ACID		P	P	P	Butyl (G)
PETROLEUM ETHERS, 80-110C	1	G	P	P	
PHENOL	4	F	F	F	
PHOSPHORIC ACID	3	G	F	G	
PICRIC ACID	3	E	G	E	
POTASSIUM HYDROXIDE	3	E	G	E	
POTASSIUM IODIDE		G	G	G	
PROPYL ACETATE	1	F	P	P	Butyl (F)
PYRIDINE	3	P	P	P	Butyl (G)
SODIUM CARBONATE		E	E	E	
SODIUM CHLORIDE		E	E	E	
SODIUM FLUORIDE	3	G	G	G	
SODIUM HYDROXIDE, 30-70%	3	G	E	E	
SODIUM HYPOCHLORITE		E	E	F	
SODIUM THIOSULFATE		G	G	G	
STYRENE	2	P	P	P	PVA (G)
SULFURIC ACID, <70%	3	F	G	G	
SULFURIC ACID, >70%	3	P	P	P	
TANNIC ACID	0	G	G	G	
1,1,1,2-TETRACHLOROETHANE		F	P	P	Viton (G)
TETRAHYDROFURAN	2	F	P	P	Teflon (G)
TOLUENE	2	F	P	P	Viton (G)
TOLUENE-2,4-DIISOCYANATE (TDI)	3	P	P	P	Butyl (G)
1,2,4-TRICHLOROBENZENE	2	F	P	P	Teflon (G)
1,1,1-TRICHLOROETHANE	2	P	P	P	Viton (G)
1,1,2-TRICHLOROETHANE	2	P	P	P	Viton (G)
TRICHLOROETHYLENE	2	P	P	P	Viton (G)
TRICRESYL PHOSPHATE	2	G	G	F	
TRIETHANOLAMINE	2	E	E	E	
TURPENTINE	1	E	P	F	
XYLENES	2	F	P	P	Viton (G)

The National Fire Protection Association (NFPA) has developed a system for indicating the health hazards of chemicals:

- 4 — Danger, may be fatal on short exposure. Specialized protective equipment required.
- 3 — Warning, corrosive or toxic.
- 2 — Warning, may be harmful if inhaled or absorbed.
- 1 — Caution, may be irritating.
- 0 — No unusual hazard.
- No information available. Avoid skin contact or inhalation.

The compatibility of the glove films with each chemical is color coded:

- P — POOR chemical resistance
- F — FAIR chemical resistance
- G - E — GOOD to EXCELLENT chemical resistance

Information is based upon published data. Safeskin gloves have not been individually tested against these chemicals. Variability in material thickness, chemical concentration, temperature and length of exposure to chemicals will affect specific performance.

SAFESKIN®

Chemical Resistance Guides

Chemical Resistance Guide: MAPA* Professional Chemical Protective Polymer Gloves

This guide is a condensed version of a more detailed chart prepared by MAPA Professional. When evaluating test results, remember that actual workplace conditions and a controlled test situation will affect performance of a glove differently. For the complete MAPA Professional chart or more information about a specific glove, call a Fisher Safety Customer Service Representative at **1-800-772-6733.**

Key to Degradation Ratings:

Performance Rating	Weight Change	**Puncture Resistance (based on EN 388 test procedure)
E (Excellent)	0 to 10%	>3.4 lbf
G (Good)	11 to 20%	2.2–3.4 lbf
F (Fair)	21 to 30%	1.1–2.2 lbf
P (Poor)	Over 30%	<1.1 lbf

Key to Permeation and Breakthrough Rate†:

BTT - Breakthrough Time
ND - None Detected
LDL - Lowest Detectable Level
NT - Not Tested
ppm - Parts Per Million
> - Greater Than
µg/cm²/min. - Microgram per sq. centimeter per minute
< - Less Than
- Rate too large to measure

Chemical Tested	Stansolv* Nitrile (22mil)				Stanzoil* Neoprene (22mil)				Natural Rubber (18mil)			
	Degradation Rating	Permeation Breakthrough			Degradation Rating	Permeation Breakthrough			Degradation Rating	Permeation Breakthrough		
	60 Min. Soak	BTT Min.	LDL ppm	Rate µg/cm²/Min.	60 Min. Soak	BTT Min.	LDL ppm	Rate µg/cm²/Min.	60 Min. Soak	BTT Min.	LDL ppm	Rate µg/cm²/Min.
Acetaldehyde	NT	NT			E	21	0.1	108	E	2	0.4	78
Acetic Acid 50%	E	>480	0.02	ND	E	>480	0.1	ND	E	31	0.02	18
Acetone	P	NT			E[1]	36[2]	NT	295	E	7	0.05	30
Acetonitrile	NT	NT			NT	143[1]	NT	1.71 (42)	NT	NT		
Ammonium Hydroxide 29%	E	>480	1.0	ND	E[1]	>480	NT	ND	E	58	1.0	108
Aniline††	P	72	0.001	18	E	>480	0.005	ND	E	>480	0.008	ND
Benzene††	P	27	0.03	582	NT	21[1]	NT	704	NT	NT		
Butyl Cellosolve*	E	>480	0.5	ND	E	147	1.0	30	E	12	1.0	162
Carbon Disulfide *(Carbon Bisulfide)*	F	20	0.2	516	NT	NT			NT	NT		
Carbon Tetrachloride††	G	341	1.0	48	P	31	NT	1512	NT	NT		
Chromic Acid (50%)	E	>175	0.1	—	NT	NT			NT	NT		
Cumene	G	271	0.03	48	F	41	0.4	216	NT	NT		
Cyclohexane	E	>480	0.02	ND	NT	184	NT	98	NT	NT		
Diamine	E	>480	0.7	ND	E	>480	0.7	ND	E	218	0.7	12
1,2-Dichloroethane	P	18	NT	2.94	NT	23[1]	NT	10.3	NT	NT		
Diethanolamine	E	>480	1.1	ND	E	>480	1.1	ND	E	>480	1.1	ND
Diethyl Ether	E	64	0.1	78	E	18	0.1	486	NT	NT		
Dimethyl Formamide *(DMF)*	NT	35	0.2	246	E[1]	217[1]	NT	21.4	E	67	0.1	246
Dimethyl Sulfoxyde *(DMSO)*	G	>480	0.004	ND	E	>480	0.004	ND	E	240	0.004	0
Ethyl Acetate	NT	NT			G	34	0.08	1068	NT	NT		
Ethyl Alcohol *(Ethanol)*	E	>480	0.002	ND	NT	>480[1]	NT	ND	E	>480	0.02	ND
Ethylene Glycol	E	>480	—	ND	E	>480	NT	ND	E	>480	NT	ND
Formaldehyde 37%	E	>480	8.0	ND	E[1]	>480[1]	NT	ND	E	>480	0.01	ND
Freon* TF	E	>480	0.01	ND	E	>480	0.2	ND	NT	NT		
Heptane	E	2	0.01	0.018	E	124	0.02	12	NT	NT		
Hexane	E	>480	0.08	ND	E	39	0.08	36	NT	NT		
Hydrazine	E	>480	0.7	ND	E	>480	0.7	ND	E	218	0.7	12
Hydrochloric Acid 37.5% *(HCl)*	E	>480	0.4	ND	E[1]	>480[1]	NT	ND	E	211	4.0	1308
Hydrofluoric Acid 48% *(HF)*	E	134	0.001	30	E	>480	1.0	ND	E	>480	1.0	ND

[1]Based on EN 388 procedure.
[2]BTT data have been normalized to a permeation rate of 0.1µg/cm²/min. as per ASTM F739. Normalized testing procedures produce BTTs equal to or longer than LDL testing.
††Known or suspected carcinogens.

North™ Viton™ and Butyl Gloves Chemical Resistance Guide, Contd.

Chemical	Viton (9mil) D	Viton (9mil) BT	Viton (9mil) PR	Butyl (17mil) D	Butyl (17mil) BT	Butyl (17mil) PR
Diethylamine	P	35 min.	852	P	47 min.	46
Dimethyl Formamide	P	8 min.	6.5	E	>8 hr.	ND
Dimethylsulfoxide	F	1.5 hr.	5	E	>8 hr.	ND
Ether	P	12 min.	21.5	P	8 min.	92.2
Ethyl Acetate	P	ID	ID	G	7.6 hr.	3.4
Ethyl Ether	P	12 min.	21.5	P	8 min.	92.2
Formaldehyde (37% in water)	E	>16 hr.	ND	E	16 hr.	ND
Glutaraldehyde	E	>8 hr.	ND	E	>8 hr.	ND
n-Hexane	ID	>11 hr.	ND	P	ID	ID
Hydrazine (70% in water)	P	ID	ID	E	>8 hr.	ND
Hydrochloric Acid (37%)	E	ID	ID	E	ID	ID
Hydrofluoric Acid (50%)	G	ID	ID	F	ID	ID
Methylamine (40% in water)	E	>16 hr.	ND	E	>15 hr.	ND
Methyl Ethyl Ketone	P	ID	ID	E	>8 hr.	ND
Methylene Chloride	F	1 hr.	7.32	P	24 min.	133
Nitric Acid (3 Molar)	G	ID	ID	F	ID	ID
PCB (Arochlor 1254 (50%))	E	>8 hr.	ND	P	ID	ID
n-Pentane	E	>8 hr.	ND	P	ID	ID
Perchloroethylene	E	>17 hr.	ND	P	ID	ID
Phenol (85% in water)	E	>15 hr.	ND	E	>20 hr.	ND
Sodium Hydroxide 50%	G	ID	ID	E	ID	ID
Sulfuric Acid (3 Molar)	E	ID	ID	G	ID	ID
Tetrachloroethylene	E	>17 hr.	ND	P	ID	ID
Tetrahydrofuran	P	4 min.	327	F	31 min.	112
Toluene	E	>16 hr.	ND	F	21 min.	221
Trichloroethylene	G	7.4 hr.	0.24	P	18 min.	550
1,1,1 Trichloroethane	E	>15 hr.	ND	P	ID	ID
Vinyl Chloride	G	4.4 hr.	0.098	P	ID	ID
Xylene	E	>8 hr.	ND	P	ID	ID

D = Degradation, BT = Breakthrough Time, PR = Permeation Rate, ND = None Detected, ID = Insufficient Data

Key to Degradation and Permeation Ratings:

E (Excellent) = Fluid has no effect

G (Good) = Fluid has minor effect

F (Fair) = Fluid has moderate effect

P (Poor) = Fluid has severe effect, ranging from moderate to complete destruction

ND = None Detected

ID = Insufficient Data; data either conflicting or not available

Permeation/Breakthrough Time Color Key

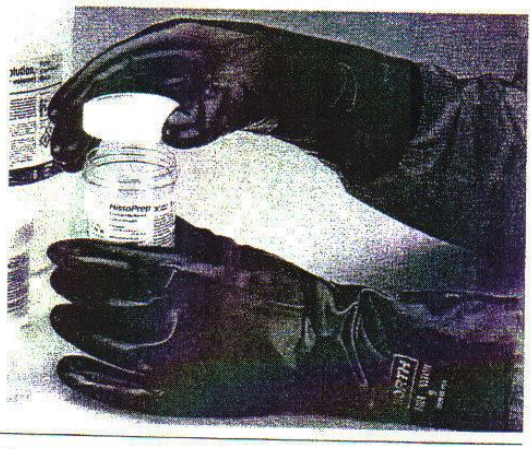
- Good for Total Immersion
- Good for Accidental Splash/Intermittent Exposure
- Not Recommended

Chemical Resistant

North Viton

Made from Viton fluoroelastomer material for protection against toxic and highly permeating chemicals like PCBs, polychlorinated triphenyls, benzene and aniline. Exhibit a high degree of impermeability to most or all chemicals that quickly deteriorate natural rubber, neoprene, nitrile and PVC gloves. See the Chemical Resistance Guide on pp. 671-672. (Chart information is based on tests performed on 9mil thick Viton gloves; 10 and 12mil thick gloves perform equivalently to those tested.) Can also be used in water-based solutions. Available in 11" (28cm) wrist or 14" (36cm) forearm lengths. Unlined. Straight cuffs. Smooth finish. Black.

Size	North No.	Cat. No.	Pack of 1 Pair
11" Long (28cm), 10mil Thick (Style F101)			
8	F101 8	11-393-62A	49.27
9	F101 9	11-393-62B	55.44
10	F101 10	11-393-62C	63.52
11	F101 11	11-393-62D	70.33
14" Long (36cm), 12mil Thick (Style F124)			
8	F124 8	11-395-46A	75.69
9	F124 9	11-395-46B	83.15
10	F124 10	11-395-46C	88.48
11	F124 11	11-395-46D	103.26



*Trademark. For ownership, see Trademark Reference.

Prices subject to change. Call your Fisher Customer Service Center for the latest information.

MAPA Chemical Resistance Guide, Contd.

Chemical Tested	Stansolv* Nitrile (22mil)				Stanzoil* Neoprene (22mil)				Natural Rubber (18mil)			
	Degradation Rating	Permeation Breakthrough			Degradation Rating	Permeation Breakthrough			Degradation Rating	Permeation Breakthrough		
	60 Min. Soak	BTT Min.	LDL ppm	Rate µg/cm²/Min.	60 Min. Soak	BTT Min.	LDL ppm	Rate µg/cm²/Min.	60 Min. Soak	BTT Min.	LDL ppm	Rate µg/cm²/Min.
Isopropyl Alcohol *(Isopropanol, IPA)*	E	>480	0.05	ND	NT	>480[1]	(0.0006)	ND	E	>60	NT	ND
Kerosene	E	>480	0.007	ND	E	>480	0.00005	ND	NT	NT		
Methyl Alcohol *(Methanol)*	E	100	0.08	18	E[1]	245[1]	NT	0.6	E	>60	NT	ND
Methyl Ethyl Ketone *(MEK)*	NT	6	NT	522	G[1]	25[1]	NT	892 (>1060)	G	6	NT	522
Methylene Chloride	NT	NT			NT[1]	12[1]	NT	>1703	NT	NT		
Mineral Spirits	E	>480	NT	ND	E[1]	>480[1]	NT	>0.1	NT	NT		
Nitric Acid 50%	G	72	0.07	1206	E	>480	0.0003	ND	E	233	0.1	0.72
Nitrobenzene	P	60	1.0	90	G	60	1.0	120	NT	NT		
Phenol (saturated)	NT	>480	NT	ND	E[1]	>480[1]	NT	ND	E	>480	0.06	ND
Phosphoric Acid 85%	E	>480	0.04	ND	E	>480	0.04	ND	E	>480	0.04	ND
Polychlorinated Biphenyls (PCBs) 50%[11]	NT	343	1.0	216	NT	>480	1.0	ND	NT	NT		
Potassium Hydroxide 50% *(KOH)*	E	>480	0.4	ND	E	>480	0.3	ND	E	>480	0.3	ND
Sodium Hydroxide 50% *(NaOH)*	E	>480	0.1	ND	E[1]	>480[1]	NT	ND	E	>480	0.1	ND
Sulfuric Acid 50%	E	>480	0.04	ND	E[1]	>480[1]	NT	ND	E	>480	0.1	ND
1,1,2,2-Tetrachloro-ethylene[11]	G	373	0.0002	27	NT	28	0.0002	453	NT	NT		
Tetrahydrofuran *(THF)*	P	17	0.08	4026	P	11	0.08	4026	NT	NT		
Toluene (toluol)	P	28	0.002	150	P[1]	19[1]	NT	>740	NT	NT		
Trichloroethylene *(TCE)*[11]	P	9	0.002	372	NT	11	NT	3966	NT	NT		
Triethanolamine *(TEA)*	E	>480	5.0	ND	E[1]	>480[1]	NT	ND	E	>480	5.0	ND
Xylenes *(xylols)*	F	92	0.002	24	F[1]	24[1]	NT	243	NT	NT		

[1]Based on EN 388 procedure.
[2]BTT data have been normalized to a permeation rate of 0.1µg/cm²/min. as per ASTM F739. Normalized testing procedures produce BTTs equal to or longer than LDL testing.
[11]Known or suspected carcinogens.

Chemical Resistance Guide: North* Viton* and Butyl Gloves

This chart is extracted and condensed from a complete guide provided by North Safety Products. The guide is designed to help you judge the right glove for use with a particular chemical. Since actual workplace conditions may vary from test conditions, always conduct your own tests to make sure you have the best possible glove for your specific need. For the complete North Safety Products Guide, call a Fisher Safety Customer Service Representative at **1-800-772-6733.**

Definitions

Degradation: A deleterious change in one or more of the glove's physical properties.

Breakthrough Time: The elapsed time between initial contact of the liquid chemical with the outside surface of the glove and the time at which the chemical can be detected at the glove's inside surface by means of analytical equipment.

Permeation Rate: The steady state flow or the highest flow of the permeating chemical, through the glove elastomer, that was recorded during the test. The rate is measured in milligrams per square meter per second.

Key to Degradation and Permeation Ratings:

E (Excellent) = Fluid has no effect
G (Good) = Fluid has minor effect
F (Fair) = Fluid has moderate effect
P (Poor) = Fluid has severe effect, ranging from moderate to complete destruction
ND = None Detected
ID = Insufficient Data; data either conflicting or not available

Permeation/Breakthrough Time Color Key

- Good for Total Immersion
- Good for Accidental Splash/Intermittent Exposure
- Not Recommended

Chemical	Viton (9mil)			Butyl (17mil)		
	D	BT	PR	D	BT	PR
Acetone	P	ID	ID	E	>17 hr.	ND
Acetonitrile	ID	ID	ID	E	>8 hr.	ND
Benzene	G	6 hr.	0.012	P	31 min.	32.3
Butyl Acetate	P	ID	ID	G	1.9 hr.	7.61
Carbon Disulfide	E	>8 hr.	ND	P	7 min.	98.4
Carbon Tetrachloride	E	>13 hr.	ND	P	ID	ID
Chlorobenzene	E	>8 hr.	ND	P	35 min.	308
Chloroform	E	9.5 hr.	0.46	P	ID	ID
Cyclohexane	E	>7 hr.	ND	P	1.1 hr.	20.3
1,1 Dichloroethane	G	1.5hr.	31	ID	ID	ID

D = Degradation, BT = Breakthrough Time, PR = Permeation Rate, ND = None Detected, ID = Insufficient Data

Contd. on next page.

Chemical Storage

Chemicals should be stored in their original bottles and the labels should be easily readable in English; lids must be well-fitting (without cracks or corrosion) and the bottles should be marked with the date the chemical was received and a label which identifies it as a fire, health or reactivity hazard. Chemicals should be stored according to compatibility; for example, oxidizing agents should not be stored with reducing agents. Any flammable material is to be stored in a flammable cabinet. Nitric acid should be stored separate from other acids. Chemicals should be stored in the following categories: inorganic acids (nitric acid separate), flammable solids, flammable liquids, flammable solvents, oxidizing agents, reducing agents, salts, organic solids, amines, and polymers. Further sub-sorting may be necessary due to incompatibility issues within these categories. **ALL CONTAINERS MUST BE PROPERLY LABELED**. The safety data sheets (MSDSs) are available to describe the chemical's properties, hazards and what to do in case of an accidental spill or exposure. The MSDSs can be consulted for storage decisions. For help with chemical spills or exposures, contact Protective Services at x3333.

Chemicals are stored in well-ventilated areas, away from direct sunlight, heat source, sparks, flames or other sources or ignition. Flammable and non-flammable volatile liquids are stored in flammable cabinets and are kept separate from the rest of the chemicals.

Liquid acids are stored in a separate cabinet from the flammables, and liquid bases are stored with the rest of the organic solvents in the flammables cabinet.

Large quantities of corrosive liquids should be stored in a corrosive liquids cabinet. Otherwise, store corrosives in easy reach, below the eyes and above the knees.

Cyanides should be stored separately. Azides should be stored separately.

Shelves on the lab bench hold both inorganic and organic solids which are relatively safe and unreactive. Hygroscopic inorganic and organic solids are stored in a desiccator on the lab bench, and air-sensitive materials are stored in the inert atmosphere box.

Chemical fume hood storage is discouraged, unless the hood is used for chemical storage only. No work should be done in a chemical storage hood.

The integrity of containers should be maintained and noted as the chemicals are present in the lab. When a crack in a container or lid is realized, the chemical should be transferred to a new container (if possible) or disposed of following appropriate protocols. An annual inspection of all containers should be performed to prevent the build-up of un-used or unnecessary chemicals.

Samples that are prepared in the lab by individual workers should be labeled with notebook number and the structure of the compound. The samples should be stored according to the guidelines above. At the end of an individual's tenure in the lab, all samples should be discarded or given to a member of the lab who assumes responsibility of them.

Chemical Waste

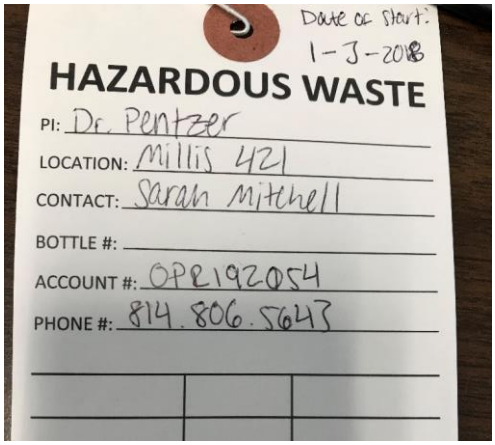
Chemical waste is to be disposed of in accordance with rules outlined in the Safety Manual supplied to our laboratory by DOES. **No chemical waste should be poured down the drain or discarded in the trash**.

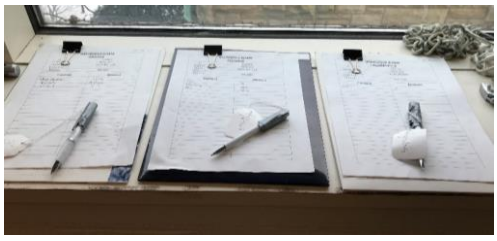
Waste must be treated no differently from the chemical constituents of which the waste is composed. Namely, flammable waste is just as flammable as flammable pure reagent grade solvent. Therefore, waste must be stored in an appropriate storage area until it is picked up. Waste containers should be checked periodically for leaks and transferred immediately to a leak-free container if needed.

The build-up of waste is not tolerated. Only one type of each waste container is to be present; when a waste container is full it is to be immediately submitted for removal following the appropriate guidelines of DOES. **If waste needs to be submitted, please contact Sarah Mitchell**.

Generally, common organic waste is separated into HALOGENATED WASTE and NON-HALOGENATED WASTE for organic liquids, in addition to ACID WASTE and BASE WASTE for aqueous solutions of corrosive materials. Solid waste, including silica gel, may also be occasionally generated. Waste containers should not be left open to the air, but the vessels should be sealed "finger tight" to prevent the build-up of pressure.

Waste Identification:
All chemical waste must be labeled with a hazardous waste label at all times, labels are provided by EHS. All chemical constituents in a hazardous waste container must be identified with chemical name and amount of compound on the hazardous waste tag. The information at the top of the tag must be filled out with the correct information (excluding the bottle number, see picture to the right). The tag is documentation for the current contents of each waste container, this must be labeled clearly and kept up-to-date. When a new tag is started, put the new start date on the tag. Once a small waste container is emptied into the larger drums a new hazardous waste tag must be filled out. For the large waste drums, each container displays a hazardous waste tag, however the logs for chemical constituents is on the window sill to the left of the waste hood.

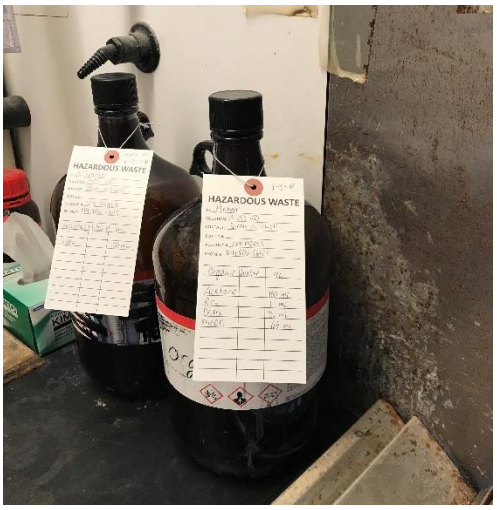




Waste Storage Requirements:

For the Pentzer lab, 20 L drums are used for halogenated, organic, and aqueous waste. These drums are kept in the solvent hood and remain there until waste removal. Small waste containers can be used in individual hoods, as long as they are identified/labeled with the requirements listed above. See pictures below for proper storage. Managing hazardous waste:

- All waste must be stored in closed containers.
- Waste containers must be in good condition and compatible with the waste they contain.
- Containers must be closed at all times, except when waste is being added or removed.
- All waste containers must be labeled clearly with contents of the waste, and marked with a hazardous waste tag.
- Avoid mixing incompatible waste in the same container. (ex., acids with bases)
- Avoid mixing halogenated and non-halogenated waste in the same container.



Waste Containers:

Waste containers are not provided to the lab. 5-gallon carboys are purchased from the stock room for aqueous waste. 20 L solvent drums are dried out and saved for organic and halogenated waste. Glass bottles can be rinsed and saved for individual hood waste. No more than 4 liters of each type of waste should be in an individual hood, besides the waste hood. Heavy plastic bags are used for solid waste, see below for more details. Waste container procedures:

- Triple rinse empty containers with a solvent capable of removing the original material.
- If a container is empty, deface all labels with a blackout marker and write empty on the container clearly.
- All containers should have cap once dried.
- Avoid obtaining a build-up of empty waste containers.

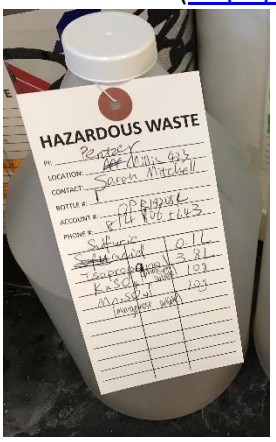
Waste Disposal Procedures:

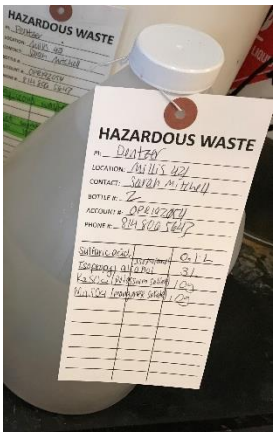
EHS provides pickup services for all chemical waste generated. A Hazardous Waste and Unwanted Chemicals form must be completed and submitted to EHS. (If form is submitted by Friday, the hazardous waste will be picked up the following week.) For disposal, let the person in charge of waste (Sarah Mitchell) know that there is waste generated and they will follow the procedure below.

Procedure for hazardous waste removal:

1. Prior to pick up, all waste must be kept in the waste hood, designated area for the Pentzer lab.
2. All waste must be placed in appropriate container(s).
3. All containers must be capped and labeled clearly.
4. Complete and submit a Hazardous Waste and Unwanted Chemicals form, obtained from EHS. (Hazardous waste tags and the removal form must match in bottle number and contents, see picture below.)

For further information regarding hazardous waste disposal, call EHS at (216) 368-2580 or visit EHS website. (https://case.edu/ehs/)





Case Western Reserve University Environmental Health and Safety
HAZARDOUS WASTE AND UNWANTED CHEMICALS FORM

ID # | PI # | Year | Pick Up #
Is this a move/termination? Yes ☐ No ☐

PAGE 1 of 1

Contact Sarah Mitchell | Primary Investigator Dr. Pentzer | Location Millis 421 | LC
Department Chemistry | Account Number OPR192054 | Phone 814.806.5643 | Date 1-3-18

List the volume or mass (L or Kg) of each component in each container. EX: A container with 10% formaldehyde in water should be documented as .010L of formaldehyde and .090L of water on the waste form. See back for complete instructions or call E.H.S. at 368-2907.

BOTTLE#	DESCRIPTION (Chemical component in each container)	QUANTITY (L or Kg)	OFFICE USE ONLY
1	formaldehyde (EXAMPLE)	.010 L	
1	water	.090 L	
1	Sulfuric acid	0.1 L	
1	Isopropanol	3.8 L	
1	potassium sulfate (K_2SO_4)	10 g	
1	manganese sulfate ($MnSO_4$)	10 g	
2	Sulfuric acid	0.1 L	
2	Isopropanol	3 L	
1	potassium sulfate (K_2SO_4)	10 g	
2	manganese sulfate ($MnSO_4$)	10 g	

I certify the above information to be true and correct
Signature Required

Picked up by:
Date:

OFFICE USE ONLY

STAGING AREA: Millis Science Center—OHD 000812330 / DOA 990—OHD 987033669
Wolstein—OHR 000120147 / The Greenhouse—OHR 000112482 / Cedar—OHD 004174660

Additional waste:

Additional waste is generated in the laboratory that follow a different procedure for disposal.

Solid Waste – Solid waste is placed in the waste hood. This mainly consists of silica gel. Solid waste is disposed in a heavy plastic bag. Once full, tie the bag and close securely with duct tape. Solid waste is removed by EHS and follows the same procedure for disposal as the hazardous waste provided above.

Trash Waste – No chemical waste or sharps waste should be discarded in the trash; this makes disposal unsafe. Gloves and Kimwipes can be discarded in the trash. All trash is picked up from the janitorial staff.

Sharps Waste – Sharps are items capable of puncturing, cutting, or abrading the skin. This includes glass or plastic pipettes, broken glass, test tubes, razor blades, needles and syringes.

- Sharps waste (needles, syringes, razor blades) contaminated with hazardous materials must be placed red rigid plastic container with a lid (purchased from the stock room). Once a container is full, call (216) 368-2580 for pick up.
- Glass waste (pipettes, broken glass, glass vials) must be placed in cardboard box designated for glass waste. These boxes are purchased from the stockroom and lined with a plastic bag provided. Once the box is full, the bag is tied and the lid is placed back on the box. Duct tape is used to secure the lid. For disposal, the box is placed in the hallway, the janitorial staff will remove.

Non-hazardous Waste – There is a cardboard box for non-sharp waste by the silica gel storage. Once full the box is taped and labeled trash. Put in the hallway for janitorial staff to pick up.

Recycling – Recyclable materials include plastic, cardboard, paper, and metal. Blue bins are provided to place recyclable material in. All cardboard boxes must be emptied and broken down. All plastic bottles and metal containers must be labeled empty and labels blacked out. Once a receptacle is full place in the hallway for pickup.

Fume Hood Use

Fume hoods are to be used for all chemical manipulations. They should be used properly and work should be done with sashes at a level approved by EHS. NEVER WORK WITH HOOD SASHES FULLY UP. Hoods should be free of clutter; the hood is not a storage place for chemicals or for paper towels and wash bottles. Electrical cords should all be in good working order (not frayed) and should not pass thru the sash, but should be passed beneath the divide. When not in use the windows should be closed. The windows should not be decorated with any non-transparent materials and one should be readily able to see into the hood.

Cooling Water

Every reaction or distillation in the department that requires a continuous flow of cooling water MUST use pressure-rated tubing and approved fittings as mandated by the College.
The Pentzer Lab has a cooler on wheels, as well as a fish pump to minimize the amount of water than can leak.

All tubing used for cooling water must have a pressure rating which exceeds 145 psi. Both Nalgene 980 Braided Clear PVC Tubing and Tygon B-44-4X I.B. Pressure Tubing have been approved for this use. A suitable stainless steel or nylon hose clamp must also be used at all connections, and are the only approved means for securing a connection. Note: twisted wire is not an appropriate method to secure hoses.

Tubing with a larger inner diameter than the hose adapter should be used, and should be clamped securely. Some typical sizes are shown below, along with the corresponding nylon hose clamps for reference.

Nalgene 980 Braided Clear PVC Tubing (14-169-10A, 1/4 in ID, 7/16 in OD) $98.15 / 50 ft
Nalgene 980 Braided Clear PVC Tubing (14-169-10G, 5/16 in ID, 1/2 in OD) $102.10 / 50 ft
Nalgene 980 Braided Clear PVC Tubing (14-169-10B, 3/8 in ID, 9/16 in OD) $105.72 / 50 ft

Fisherbrand Tight-Seal Nylon Clamps (05-815-1C, for 7/16 in OD tubing) $25.10 / pk of 100
Fisherbrand Tight-Seal Nylon Clamps (05-815-1D, for 1/2 in OD tubing) $25.10 / pk of 100
Fisherbrand Tight-Seal Nylon Clamps (05-815-1E, for 9/16 in OD tubing) $25.10 / pk of 100

If cooling water is needed for an OVERNIGHT experiment, the use of an ice bucket and CIRCULATOR is required.

Note: You will personally be held responsible for any damages caused by flooding if your reaction/distillation setup does not meet the mandated standard!

Syringes

Both gas-tight and single-use disposable syringes are available. Gas-tight syringes should be used for transfer of any pyrophoric solutions, and are encouraged for use with anhydrous solvents.

Gas tight syringes should be dried in an oven over night, then allowed to cool to room temperature in a dessicator before use. The volume of solution to be used should not exceed 75% of that which the syringe can hold. Directly after you have finished using the syringe, it must be cleaned (rinsed with an appropriate solvent). All used disposable syringes should be place in the syringe disposal box at the front of the lab. If the syringe was used to transport air sensitive materials it should be put into sand for 8h before putting into the waste container. If the syringe was used for corrosive compounds such as strong acids, the syringe should be soaked in water before disposal. For other compounds, appropriate procedures should be followed with regards to the material in the syringe before the empty syringe is disposed; if unsure consult Dr. Pentzer.

When drawing liquid into a syringe, do not use two hands to pull the plunger! Use one hand to hold the connection between the syringe and needle (at the luer lock) while using the other hand to carefully, and slowly, pull the plunger out to fill the syringe. Likewise, when dispensing from a syringe, use one hand to secure the needle and the other to slowly push the plunger. For large amounts of liquid this can take a while, be patient!

Needles

Disposable and re-usable needles of varying lengths and gauges are available. The choice of needle is dictated by the compound to be used, as well as the amount of liquid to be retrieved. Before use, you should check to ensure that the needle is not clogged. This can be done by attaching the needle to a nitrogen port of the schlenk line and ensuring that nitrogen flows.

All disposable needles should be discarded immediately after use in an approved sharps container, they should not be recapped. All re-usable needles should be rinsed immediately to prevent clogging (choice of rinsing solvent is determined by the use). Needles are sharp! Take care not to poke yourself.

If you are accidentally stuck with a needle, ask for medical help and notify the medical personnel of what chemicals may be in the syringe.

Heating Reactions

Many chemical reactions performed in the chemistry laboratory require heating of a reaction flask; most of this heating can be performed on an IKA-type hotplate/stirrer. To use this equipment, the “safety temperature” should be set no higher than 20 °C above the temperature the reaction will be performed at, and the temperature probe should be used. A reaction should not be left unattended before the set temperature is reached. Take care not to touch hot plates, oil baths, aluminum blocks, or sand baths which are hot; looks can be deceiving.

Oil bath: an oil bath can be prepared using silicon oil in a crystallization dish. MINERAL OIL SHOULD NEVER BE USED (it has a flash point of 120 °C and will catch on fire). Oil baths should never be heated above 250 °C. If chemicals are spilled into the oil bath, it should be discarded in

its own waste container. Spilled oil can be cleaned up by being absorbed by the grey packaging materials which come wrapped around chemical bottles.
Aluminum blocks: Aluminum blocks present an alternative method for heating and are especially useful for heating vials. Aluminum blocks should be used with the IKA temperature probe. Egg or pear shaped round bottom flasks should not be heated using an aluminum heating block, the expansion is not uniform and the flask can break.
Sand baths: To heat to temperatures above 175 °C, sand baths should be used. A sand bath can be made by placing dry sand in a heating mantle; the heating mantle should be plugged into a temperature regulator and the power should be slowly turned up until the desired temperature is met, as determined by a thermometer inserted into the sand. Glassware should be completely dry before placing into the sand bath, to prevent an explosion.

Glassware

When dealing with glassware take extreme caution to avoid cuts and burns. Glassware looks the same whether hot or room temperature, so use caution when drying glassware and only use tongs or oven gloves to remove glassware from the oven. Most damage to glassware occurs when the glassware is hot.

When inserting glass tubing into a septum or stopper you must use GLASS APPROVED GLOVES which prevents cuts with broken glass. Check all glassware for chips and cuts, and especially star fractures. If defects are found, the glassware should be discarded or the imperfection marked with a marker and placed in the box to be repaired by the glass blower.

Regulated Chemicals

Methylene chloride, benzene, and acrylonitrile are regulated chemicals. We must report the use of these chemicals every 6 months to EHS. These chemicals are toxic, and known or suspected carcinogens. Please read the programs for methylene chloride and benzene, available on EHS website at https://www.case.edu/ehs/ChemSafety/regchem.html .

Air-Free Techniques

Vacuum manifold:

An excellent source for using an inert gas/vacuum two-way line (Schlenk line) can be found in “Advanced Practical Organic Chemistry Techniques,” chapter 9.2.

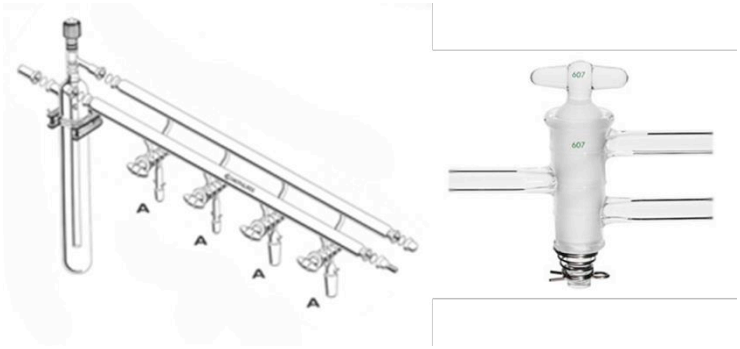
Importantly: Do not immerse a trap in liquid nitrogen unless the trap is under vacuum! If air is cooled with liquid nitrogen, liquid oxygen could condense, resulting in a violent explosion upon re-vaporization and oxidations of organic solvents. Furthermore, check all glassware for star cracks and chips before use; do not use damaged glassware, as this provides routes for implosion under vacuum. Proper set up is shown below, copied from http://www.ilpi.com/inorganic/glassware/vacline.html

Introduction

Schlenk lines and vacuum lines provide a convenient means of manipulating air and water-sensitive materials without the use of an inert ('dry') atmosphere glove box. The terms "Schlenk line", "vacuum line", "chemical transfer line", or "vacuum transfer line", refer to similar set ups, but in some general distinctions can be drawn:
Vacuum lines usually have a better vacuum (10^{-4} to 10^{-7} torr) than Schlenk lines (10^{-2} to 10^{-4}torr), because vacuum lines typically have a diffusion pump. A way of delineating these would be to use the terms "Schlenk line" and "High vacuum line". Vacuum lines usually connect to the experimental apparatus with joints, which provide a better seal and vacuum than the simple rubber pressure tubing connections used on Schlenk lines. Manipulations involving the measurement or condensation of gases are usually performed on a high vacuum line. Operations involving cannulas and counterflow techniques are usually performed on Schlenk lines.

A Schlenk or high vacuum line consists of a glass manifold that has several stopcocks, valves or openings in addition to a connection to a vacuum source (typically a mechanical and/or diffusion pump). Having several ports on the line is convenient because several different flasks or reaction vessels may be used simultaneously. For example, gases can be vacuum transferred from one flask to another or several reactions may be run at the same time. Dual manifolds have the ability to expose a flask to either nitrogen (or argon) and vacuum. Two fume hoods in the Pentzer lab are equipped with dual manifold high vacuum lines and four have dual manifold Schlenk lines. The designated vacuum line has a single manifold design. Importantly: stopcocks should be greased with Apiezon M or H type grease (this is inert and robust, yet expensive).

In the Pentzer lab, double oblique bore stopcocks are used to connect the vacuum and nitrogen in the line to the flask (see below). Each port has a designated stopcock, identified by matching numbers on the port and the plug. The glass plugs are not interchangeable! If the wrong glass plug is used, the glass will grind, leading to leaks, and ruining the glassware. To connect the line to the flask, only red rubber tubing should be used; other tubing is reactive with typical chemicals, or cannot withstand the low pressures of the schlenk line.



Using nitrogen gas:

To use the nitrogen ports, the vacuum does not have to be up and running. Flow of nitrogen to the line is controlled by a regulator on the gas tank; you should never change the set pressure. If a reaction is to be performed under nitrogen, a bubbler should be used. A "T joint" should be attached between the line and the flask, with the third arm connected to a bubbler. The bubbler

should contain mineral oil. NEVER USE SILICON OIL in a bubbler, it can undergo polymerization, clogging the bubbler and leading to an explosion. Make sure to close the stop cocks after use, so as not to waste nitrogen.

Using vacuum:

Turn on:

(1) Ensure all stopcocks are turned to the "off position" or to nitrogen (if being used). This should be a closed system.

(2) Turn on the vacuum. It will "gargle" at first, but this should subside within 20 seconds. DO NOT ADD LIQUID NITROGEN UNTIL LINE HAS BEEN EVACUATED.

(3) Place the vacuum trap in a Dewar, then add liquid nitrogen.

(4) Wrap the top of the dewar with insulating material (such as Styrofoam or glass wool)

Make sure to refill the liquid nitrogen as needed, as it evaporates over time. Lack of liquid nitrogen in the trap will allow volatile organic compounds to enter the pump, ruining the oil and corroding the parts. If more than trace organic solvents are being removed, an external trap is needed. DO NOT COLLECT SOLVENT IN THE TRAP FOR THE LINE. Note: two different lines should never be attached to the same flask!

Turn off:

(1) Ensure all stop cocks are closed (i.e., that no flask is under vacuum).

(2) Remove the liquid nitrogen from the trap.

(3) Open one line to air, then IMMEDIATELY turn off the vacuum.

(4) Keep the stopcock open to atmosphere and allow the chilled trap to warm to room temperature

Failure to vent the line before turning off the vacuum can result in suction of pump oil into the line (not fun to clean up). Any collected solvent in the trap should be allowed to warm to room temperature (while still vented). Discard the now liquid solvent from the trap into the appropriate waste container, and wash trap. It's a good idea to re-grease the trap with high vacuum silicon grease each time.

De-gassing solvents:

Solvents can be degassed by either bubbling nitrogen (or better yet argon) thru the solvent or liquid, if only a small amount is need. Alternatively, the best way to remove oxygen from a solvent is by performing three freeze-pump-thaw cycles. The vacuum manifold of the schlenk line should be running, with liquid nitrogen on the trap, as outlined above. If the solvent needs to be anhydrous and air-free, look up how to properly dry the chosen solvent in "Purification of Organic Compounds" or Vogel's "Practical Organic Chemistry."

(1) Oven dry the glassware overnight, then allow to cool to room temperature in a dessicator, or under nitrogen on the schlenk line.

(2) Add the solvent or liquid to be degassed to the flask, using air-free transfer if needed.

(3) Attach the flask to the schlenk line, but keep stopcock in the "off" position. Alternatively, a stopcock on the flask containing the compound can be used to regulate vacuum exposure.
(4) With the flas, as a closed system, freeze flask containing the solvent in liquid nitrogen. Depending on the volume this could take up to 15 minutes.
(5) *With the solvent frozen and still in liquid nitrogen,* turn on the vacuum, and wait for the pressure to drop (about 3 minutes).
(6) Close the stopcock to the off position and remove the liquid nitrogen bath.
(7) Warm the flask to room temperature by gently heating with a heat gun or placing the flask in a warm water bath. You should see bubbles escape the as the frozen liquid melts. KEEP THE FLASK AS A CLOSED SYSTEM. Do not turn on the nitrogen or vacuum. The vacuum in the head space created during the "pump" cycle is enough to pull dissolved air out of the system.

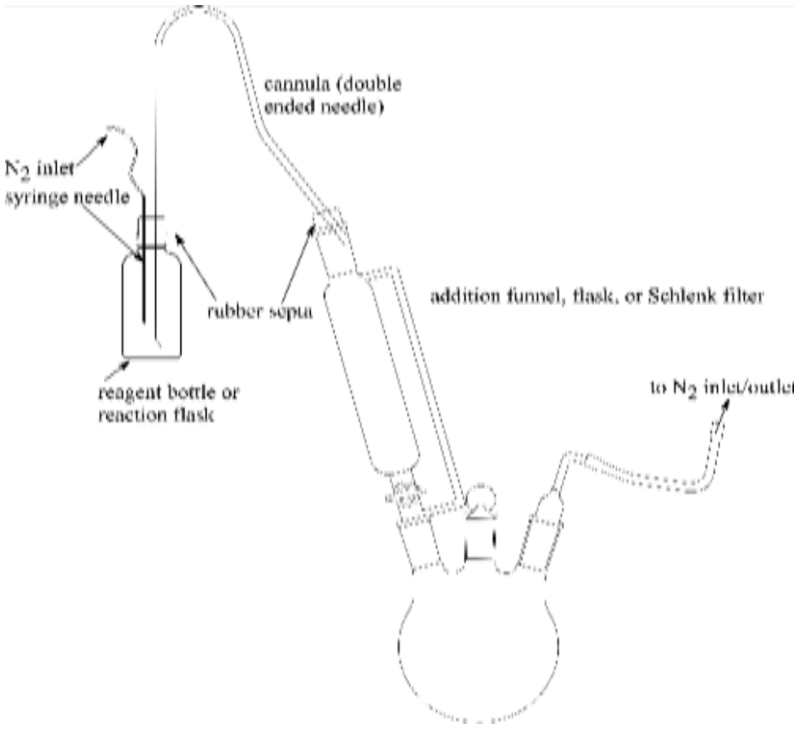
This cycle should be repeated three times. To ensure that the degassed solvent remains degassed, it should be stored in air-free glassware (specifically a Strauss flask).

Cannula Transfer:

When transferring large amounts of liquid under air free conditions, cannula transfer technique is best. A cannula is a needle which has not luer lock, but is instead sharp at both ends (or sharp at one end and blunt at the other).

(1) The flask containing the liquid to be transferred should be under nitrogen and contain a penetrable point of entry (such as a septum)
(2) A second flask should be under static nitrogen
(3) An oven-dried, but room temperature cannula should be inserted into the flask containing the liquid, but the tip should be kept above the top of the liquid.
(4) The second tip of cannula (currently exposed to air) should be inserted into the septum of the second flask, to which the solvent will be transferred.
(5) A short "exit" needle should be placed in the empty flask, but the tip kept well above the tip of the cannula. Nitrogen should now be flowing in the following order: line → flask with solvent → empty flask → out.
(6) Dip the tip of the needle in the first flask below the solvent level. The pressure imposed by the nitrogen should cause the solvent to be transferred to the second flask!
(7) When the desired amount of liquid has been transferred, lift the tip of the needle in the first flask above the level of the liquid to stop flow of solvent.
(8) The needles should be removed in the opposite order they were put in. Remove the short purge needle, then the cannula needle in the second flask, then the cannula needle in the first flask.

A schematic of the transfer can be seen below. Clean cannula immediately with a solvent in which the transferred substance is soluble. Use an aspirator to draw clean solvent through the cannula. Rinse with water, then acetone.



Vacuum Distillation

Vacuum distillation is used to isolate and purify products whose boiling points are too high to distill at room temperature.

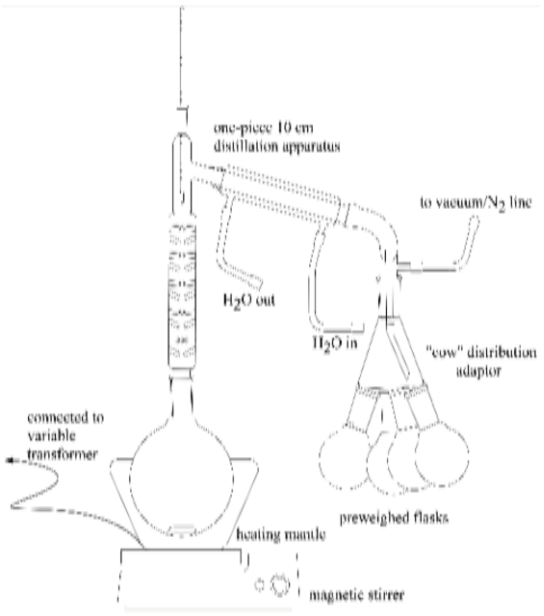
(1) Assemble the distillation apparatus shown in the figure below. All glassware should be dried in an oven overnight and cooled to room temperature in a dessicator. Before use inspect all glassware for cracks, chips, and star fractures. Only one clamp is needed, and should be placed on the flask from which distillation will occur. Make sure to grease all joints with fluorinated grease, and secure with keck clips. Make sure to turn on the cooling water.

(2) Hook up the vacuum to the adapter, and *slowly* turn it on. Note: the flask should not be heated yet. The liquid may bubble as air escapes, be careful so it doesn't bump. It's a good idea to twist all joints to seal any leaks that could be present. Leave the system under vacuum for 5-10 minutes. Note: keep the vacuum on, do not heat a closed system.

(3) In order to distill the liquid, start to slowly apply heat, increasing 15-20 °C every 20-30 minutes. After you distill a compound for the first time, you should have a better idea as to what temperature the product (and impurities) distill.

(4) Pay close attention to when distillation occurs. Record the temperature of the vapor, and do not adjust the heat applied until you are sure all of one product has finished distilling. Generally, after the fraction has finished distilling, the temperature of the vapor will drop.

(5) When distillation is complete, remove the heating source and put the system under nitrogen, allowing it to cool to room temperature. Disassemble the distillation apparatus and wash all glassware. Be cognizant of any reactive compounds or impurities that may be present.



Vacuum Pump Maintenance

These steps should be followed when changing the pump oil to prevent damage. Pumps should always be kept in a secondary container in case oil leaks. Oil is difficult to clean up and messy- take care not to spill it. Pump oil waste should have a separate waste container than organics and other oil waste.

Maintenance:

- Check oil level regularly and change the oil if the quality is not good (i.e., it's dark or cloudy). Oil should be changed about every 3 months: a good rule is when you get your car oil changed. Keep a record of oil changes on a log sheet located next to the pump.
- If solvent was sucked into the pump, change the oil immediately. You can generally tell by the smell the pump gives off when turned on.
- Use liquid Nitrogen to trap the solvents and hence prevent the solvent from entering the motor.
- Never use black oil or oil with impurities

Changing the oil:

- Locate the pump oil changing paraphernalia. This should include plastic funnels, plastic beakers, and the oil. Ensure that the pump oil you use is compatible with your pump (look on the manufactuer's website).
- Run the pump for 5-10 minutes to warm the oil, which makes it easier to drain, and to loosen up any sediment.
- Turn off the pump and open the drain valve to drain the oil into a pan that can hold twice the volume of oil in the engine's crankcase. To completely drain the pump you'll need to prop up the back edge of the pump. Ask someone to help, and keep it propped up by placing a brick under it.

Remove the brick which elevates the back and replace the drain plug. Pour in clean oil to about a quarter of a tank, using a funnel and wipe up any drips with a paper tower. MAKE SURE YOU PUT IN THE CORRECT OIL.

Run the pump oil for 10-15 minutes, then drain and discard this oil. This oil is used to rinse the remaining sediment out of the pump. To drain this oil use the same protocol as above.

Add new pump oil into the pump using a funnel to just below the ideal working level. Slowly pour in the oil, as it takes some time to reach the gauge. Also keep in mind that the pump oil will expand upon heating (use of the pump). Run the pump for 10 minutes to check the oil level. Correct as needed.

Important:

Belt guards must be in place

Service cords and plugs must be free of defects.

Do not use extension cord or power strip.

Place the pump on a tray so that spilled oil is contained

Pump oil may be contaminated and should be disposed as chemical waste.

Pump oil must be compatible with the vapors that will pass through the pump (i.e. do not use hydrocarbon pump oil with oxidizing gases or vapors).

Use a cold trap to prevent the degradation and contamination of pump oil.

Use an oil mist separator (purchased from the pump vendor) to prevent oil loss.

Extraction and Washing Guide

Standard extraction and washing protocols can be applied to virtually any crude reaction mixture. Aqueous washings are done to remove water soluble impurities from organic products, as the compound that you normally desire will be dissolved in the organic layer. Pay special attention when neutralizing acids or bases (especially when using $NaHCO_3$, and CO_2 is evolved) and also when using ether as a solvent, as its low boiling point can lead to a build-up of pressure.

Standard Aqueous Workup Protocol:

(1) Use an organic solvent that is immiscible with water and that your product is soluble in. Ether and ethyl acetate are good choices, although the higher boiling point of ethyl acetate makes it harder to remove. Dichloromethane (DCM) tends to form emulsions and many organic salts are readily soluble in DCM. The choice of organic solvent is typically dictated by the product, as well as the reaction solvent.

(2) Dillute your crude reaction with the organic solvent and transfer to a sep funnel (make sure stop cock is turned off!) which is resting in ring; the seperatory funnel should be able to hold over twice the volume of your organic phase. For 0.5 g of reactant, 100 mL or less of solvent can be used.

(3) Add water to remove the water-soluble impurities. Typically add a volume of water that is 10-50% of your organic solvent. Note: If your reaction was performed in a water-miscible organic solvent (such as DMSO or DMF), your product can be pulled into the water phase, as DMSO/DMF can make it soluble in water.

(4) To properly "wash" the organic layer, stopper the sep funell and quickly invert- *while securing the stopper with your hand*, and open the stopcock (pointed up and towards the back of the fume hood). Close stop cock and shake/rock the sep funnel, venting thru the stopcock every couple of shakes. When venting, the opening should be pointed at the back of a hood. It is very important to avoid build up of pressure in the funnel! When venting is performed a couple of time, return sep funnel to ring and remove stopper. NEVER STORE A SEP FUNNEL WITH THE STOPPER IN.
(5) Drain the aqueous layer (on the bottom usually, check out a density chart). Note: if you have the stopper in, you cannot drain the sep funnel. Repeat the washing twice more.
(6) In addition to water, it may be necessary to remove impurities by protonation/ deprotonation. This can be done using <10% HCl or saturated $NaHCO_3$. Any time you wash an ether phase with HCl, it must be washed with $NaHCO_3$, since HCl is slightly soluble in ether.
(7) Finish the extracting by washing with a solution of saturated sodium chloride (brine).
(8) If emulsions form, they can be broken up by adding more organic solvent or by the addition of brine.
(9) Dry the organic phase by swirling it over $MgSO_4$ or Na_2SO_4 (make sure your product will not reaction with/bind to the drying agent). When you swirl, it should look like a snow globe. If the drying agent is chunky, there can still be water left to be removed.
(10) Flute a filter paper for gravity filtration or set up a Buchner funnel for vacuum filtration. Filter the solution into a round bottom flask so the solvent is not more than 50% of the possible volume of the flask.
(11) Remove the solvent by rotary evaporation and remove residual solvent on a schlenk/vacuum line. The product can be transferred to a smaller vial for storage. DO NOT STORE COMPOUNDS IN ROUNT BOTTOM FLASKS FOR MORE THAN 24 h.

Flash Chromatography Guide

Flash column chromatography is a quick and (usually) easy way to separate complex mixtures of compounds. Separation occurs based on a difference in the affinity of the compounds for the solid phase versus the mobile phase. Stationary phases include silica gel (slightly acidic) and alumina (can be acidic, basic, or neutral depending on how much water is adsorbed). In essence, a glass column is packed with the stationary phase, the mixture of compounds is loaded onto the column, and solvent is run thru until the product elutes. For extremely small amounts of products, prep TLC offers a viable option. In flash chromatography compressed air is used to push the solvent through the column, providing better separation and decreasing the the amount of time required to run a column.

(1) Run a TLC to determine the appropriate solvent/solvent mixture needed to get separation. Ideally the product with have an Rf value of 0.2-0.3 (determined by dividing the distance the product travels from base line by the distance the solvent front travels). Good solvent mixtures to use are a combination of hexane/ethylacetate or methanol/DCM. These solvents are flammable and/or toxic. Flash chromatography should be performed in a fume hood. Avoid high boiling point solvents. Never use more than 10% methanol on silica gel; Never use acetone on alumina.

(2) Chose a column of an appropriate size for your reaction scale and secure it. Easy separations require ratios between 30-50:1 silica:product (by weight), while harder separations call for ratios of up to 120:1. Make sure it is vertical and level. Suspend the silica gel in the solvent to be used for your column, stirring with a glass stir rod. Add the slurry to the column with the stopcock open, rinsing the flask with more solvent. Let the silica gel slowly settle, tapping gently on the sides of the column with a wooden pencil. Let the solvent run out, gently applying pressure once the silica gel is settled. Do not let the top of the column turn dry

(3) With the solvent at the top of the silica gel, add a thin layer of sand. Then pipette your product (or product dissolved in a minimal amount of solvent) onto column evenly. Open the stopcock to let the product pass thru the sand and onto the silica gel. Add a minimal amount of solvent to wash the sand and transfer all product to the silica gel.

(4) With stopcock closed, slowly add solvent by running it down the sides of the column until a large layer has built up on the silica gel (about 5 inches). Then slowly add solvent using a funnel and long stemmed funnel, running the solvent down the sides of the column.

(5) Open the stopcock and allow the solvent to run thru. You will most likely not be able to see the product travel down the column. Compressed air can be used to increase the speed, but should never be attached without a vent! Refill solvent as needed. Never let the top of the column run dry.

(6) To determine when the product is eluting, spotting onto a TLC plate can be used. This will only work if your spot is UV active (in general conjugated). If your product is not UV active (you should know bc you did TLC on it), an appropriate stain may be used.

(7) Collect fractions which elute using small erlynmeyer flasks (25, 50, or 100 mL), or for small columns use test tubes. Keep track of the order of elution. If you invert you TLC plate this is the order your spots should come out. You can TLC every fraction, or better, ever 5th fraction, to determine where your product is.

(8) Combine the flasks containing your product in a round bottom flask and remove solvent by rotary evaporation. Let the remaining solvent run out of the column, then dry the silica gel by running solvent thru the column. Dispose of the silica gel in the solid waste.

Acknowledgements:

A BIG Thank You to Dr. Genevieve Sauve and Dr. Thomas Gray and groups for several guides and protocols used in this document.

Recommended Reading:

"Advanced Practical Organic Chemistry" by J. Leonard, B. Lygo and G. Procter, Blackie Academic & Professional, 2 edition (1995) (LLP)

"Textbook of Practical Organic Chemistry" by Vogel; 5th edition (1996).

Standard Operating Procedures

1. Azides
2. Benzene
3. Cyanide Salts
4. Dry ice/acetone
5. Hydrofluoric acid
6. Potassium Fluoride
7. Volatile Thiols
8. Nitric Acid
9. Hydrazine
10. Organometallics (organolithium and Grignards)
11. Hydrogen Peroxide (30 wt% in water)
12. Potassium Permanganate
13. Potassium metal
14. Trimethyl aluminum
15. Liquid Nitrogen
16. Aqua Regia