

DEPARTMENT OF  
CHEMICAL AND BIOLOGICAL ENGINEERING

**CBE 424**

**Operations and Process Laboratory**

Session I

May 21 – June 22, 2007

UNIVERSITY OF WISCONSIN-  
MADISON

This manual belongs to

---

Campus Address

---

---

---

---

Phone Number

---

Email

---

Lab Partner

---

Partner's phone number

---

Team Number

---

Group Partners

---

---

---

---

---

---

---

Group Number

---

---

# Operations and Process Laboratory Manual

## Contents

### Preface

Teaching Staff, Course Organization, Lab Partners,  
Email Addresses, Group Assignments, Schedule,  
Oral Reports

i - x

### Part I. Lab Policies and Procedures

A.	Safety	1
B.	Supplies and Equipment	5
C.	Report Specifications	13

### Part II. Formal Experiment Protocols

A.	Distillation	D 1
B.	Heat Transfer	HX 1
C.	Humidification	Hu 1
D.	Pump	P 1
E.	Reactors	R 1

# Operations and Process Laboratory Instructors

Session I: May 21 – June 22, 2007

---

**Professor Thatcher W. Root. (Director)**

University of Wisconsin-Madison

**Professor James Beckman**

Arizona State University

**Professor Rafael Chávez-Contreras**

University of Wisconsin-Madison  
Instituto Tecnológico de Celaya, Mexico (on leave)

**Professor Daniel Klingenberg**

University of Wisconsin- Madison

**Dr. Morris R. Schoenberg**

Amoco Oil & Chemicals, Retired

## Course Organization

Students in the laboratory are grouped into Teams of two and into Groups of four to six members. These assignments are enclosed on pages vi and viii. Groups perform the five formal experiments during the course according to the schedule on p. ix. A different instructor supervises each of the formal experiments. In addition, the Teams perform four informal experiments, which are assigned by individual instructors according to the assignment schedule on p. vii. When a Team completes one informal, and is released by the instructor for this informal, team members may contact the next instructor to get started on the next assignment. Students should anticipate spending less than one week to complete each informal experiment.

Students must prepare individual written reports on all formal and informal experiments, the sole exception being one informal that is presented orally. The first informal report must be rewritten after it has been graded; the rewritten report will be graded as a separate report. The exercise of rewriting a report is not only helpful in clarifying the style appropriate for a formal technical report, but is also a necessary condition for this course to satisfy the University's communication-skills requirement for graduation.

The oral reports will be presented during the last week of the course. One partner should present an oral report on the second informal experiment, and the other partner will present the third informal orally. In each case the other partner will present a written report, but the presenter need submit only copies of the visual aids from the talk, together with the sample calculations for evaluation.

The course grade will be based 95% on the ten reports, including the oral and the rewritten report, all equally weighted, and 5% on professionalism, as assessed by the instructors. Report grades will reflect both technical content and quality of presentation. Unacceptable reports after the first one must be rewritten, and the grade assigned for that experiment will be the average of those for the two versions.

All students are expected to be in attendance from 7:45 a.m. until 4:30 p.m. every day except for the 45-minute lunch break. Students should wear their nametags while in the laboratory for safety purposes. If you wish to leave the lab, for example, to go to the library, you must replace your nametag on the storage board in the lab to indicate you have checked out of the lab. You may also want to leave a note indicating where you may be found.

The class will meet in 1227 Engineering Hall at 7:45 a.m. each morning for announcements. Some days there will be short lectures at 12:30 p.m. in the same room. Attendance and prompt arrival are expected of all students unless one has explicit approval from an instructor to be elsewhere.

Teamwork is an important aspect of professional engineering practice. Groups conducting the formal experiments are expected to work together as a team. To clarify roles, groups will be asked to identify for each experiment one group leader and a head operator as well as assignments for other individuals, such as a data analyst, a data recorder, and operators with specific responsibilities. These roles should rotate from one

## Preface

day to another. The specific organization should be designed to meet the needs of each particular experiment.

Each team, that is, each pair of lab partners, should maintain a bound lab notebook in which notes, drawings, data, observations, and other pertinent information should be recorded. Such notebooks are used in industry for purposes of documenting patentable discoveries. They are also important for creating a clear record of what was done and what was observed in the laboratory or plant. It is good laboratory practice to also include printouts of any computer data files as both a backup and as a permanent record.

For each formal experiment, one group member should be given responsibility for compiling all of the relevant data into a master data sheet. Copies of that data sheet should be included in each student's report.

Students are responsible for arranging for electronic backups of any important computer files. Students may use CAE servers, MyWebSpace storage, flash memory devices, or any other reliable method.

A major objective of this course is to develop good communication skills. Listed below are five books that students will find useful in improving their technical-writing skills. These are on reserve at the Kurt Wendt Library. The library also has many other texts on technical writing.

Pfeiffer, W. S., *Pocket Guide to Technical Writing*, Pearson Prentice Hall, Upper Saddle River, New Jersey, 2004

Beer, D., and D. McMurrey, *A Guide to Writing as an Engineer*, John Wiley & Sons, New York, 1997.

Ulman, J. N., and J. R. Gould, *Technical Reporting*, 3rd ed., Holt, Rinehart and Winston, New York, 1972.

Eisenberg, A., *Effective Technical Communications*, McGraw-Hill, New York, 1982.

Ally, Michael, *The Craft of Scientific Writing*, 3rd ed, Springer, New York, 1996.

Strunk, W. R., and E.B. White, *The Elements of Style*, 3rd ed, Macmillan, New York, 1972. Strongly recommended for purchase.

This course also has a web page at <http://courses.engr.wisc.edu/che/424/root/> which the instructors will use to distribute messages, notes, documents, and useful computer programs.

On the last week of the course, at 9:00 a.m. on Thursday, all students must report for cleaning up the laboratory. All reports must be submitted by 9:00 a.m. on Friday, the last day of the course.

**Student Email Addresses**

<b>Name</b>	<b>Address</b>
Ballenger, Richard	rjballenger@wisc.edu
Chen, Sophia	chen1@wisc.edu
Ebeling, Andrew	apebeling@wisc.edu
Elliott, Katherine	ktstewar@wisc.edu
Gilgenbach, Anita	amgilgenbach@wisc.edu
Gore, Patrick	prgore@wisc.edu
Hsiao, Lilian	cchshiao@wisc.edu
Jensen, Lindsay	lnjensen@wisc.edu
Lambert, Corrinne	cmlambert@wisc.edu
Larsen, John	jdlarsen@wisc.edu
Lewandoski, Annie	amlewandoski@wisc.edu
Mahoney, Michelle	mrmahoney@wisc.edu
McCormick, Joshua	jmccormick@wisc.edu
Mittal, Himanshu	hmittal@wisc.edu
Nichol, John	jwnichol@wisc.edu
Page, Amber	amberpage@wisc.edu
Peykov, Alexander	peykov@wisc.edu
Pronley, Mark	mfpronley@wisc.edu
Ross, Jennifer	jenniferross@wisc.edu
Russeff, Richard	russeff@wisc.edu
Schwindt, James	jtschwindt@wisc.edu
Sharma, Himanshu	hsharma2@wisc.edu
Shuen, Tsi Felix	tsshuen@wisc.edu
Webb, Josh	jjwebb2@wisc.edu

**Student Partner, Team and Group**

<b>Team</b>	<b>Name</b>	<b>Partner</b>	<b>Group</b>
1	Ballenger, Richard	Larsen, John	I
2	Chen, Sophia	Hsiao, Lilian	II
3	Ebeling, Andrew	Schwindt, James	III
4	Elliott, Katherine	Lewandoski, Annie	III
5	Gilgenbach, Anita	Page, Amber	IV
6	Gore, Patrick	Lambert, Corrinne	V
2	Hsiao, Lilian	Chen, Sophia	II
7	Jensen, Lindsay	Ross, Jennifer	VI
6	Lambert, Corrinne	Gore, Patrick	V
1	Larsen, John	Ballenger, John	I
4	Lewandoski, Annie	Elliott, Katherine	III
8	Mahoney, Michelle	Sharma, Himanshu	V
9	McCormick, Joshua	Webb, Joshua	II
10	Mittal, Himanshu	Nichol, John	I
10	Nichol, John	Mittal, Himanshu	I
5	Page, Amber	Gilgenbach, Anita	IV
11	Peykov, Alexander	Russeff, Richard	IV
12	Pronley, Mark	Shuen, Tsi	VI
7	Ross, Jennifer	Jensen, Lindsay	VI
11	Russeff, Richard	Peykov, Alexander	IV
3	Schwindt, James	Ebeling, Andrew	III
8	Sharma, Himanshu	Mahoney, Michelle	V
12	Shuen, Tsi	Pronley, Mark	VI
9	Webb, Joshua	McCormick, Joshua	II

**Assignments to Informal Experiments**

Session I – 2007

Team	Informal 1	Informal 2	Informal 3	Informal 4
1	Root	Beckman	Klingenberg	Chavez
2	Schoenberg	Chavez	Root	Klingenberg
3	Beckman	Klingenberg	Schoenberg	Root
4	Chavez	Root	Beckman	Schoenberg
5	Klingenberg	Schoenberg	Chavez	Beckman
6	Root	Beckman	Klingenberg	Chavez
7	Schoenberg	Chavez	Root	Klingenberg
8	Beckman	Klingenberg	Schoenberg	Root
9	Chavez	Root	Beckman	Schoenberg
10	Klingenberg	Schoenberg	Chavez	Beckman
11	Root	Beckman	Klingenberg	Chavez
12	Schoenberg	Chavez	Root	Klingenberg

## **Group Assignments for Formal Experiments**

### **Group I**

Ballenger, Richard  
Larsen, John  
Mittal, Himanshu  
Nichol, John

### **Group II**

Chen, Sophia  
Hsiao, Lilian  
McCormick, Joshua  
Webb, Josh

### **Group III**

Ebeling, Andrew  
Elliott, Katherine  
Lewandoski, Annie  
Schwindt, James

### **Group IV**

Gilgenbach, Anita  
Page, Amber  
Peykov, Alexandar  
Russeff, Richard

### **Group V**

Gore, Patrick  
Lambert, Corrinne  
Mahoney, Michelle  
Sharma, Himanshu

### **Group VI**

Jensen, Lindsay  
Pronley, Mark  
Ross, Jennifer  
Shuen, Tsi Felix

## Formal Experiment Schedule

	<b>Distillation</b>	<b>Heat Transfer</b>	<b>Humidification</b>	<b>Pump</b>	<b>Reactor</b>
	Root	Beckman	Chavez	Klingenberg	Schoenberg
M May 21					
T 22	I	VI		IV	V
W 23	I		III		
R 24	II			V	VI
F 25	II		IV		
M 28	<b><u>HOLIDAY - Memorial Day</u></b>				
T 29		I			
W 30					
R 31	III	II		VI	I
F June 1	III		V		
M 4	IV		VI		II
T 5	IV	III		I	
W 6					
R 7	V			II	III
F 8	V				
M 11		IV	I		
T 12	VI			III	
W 13	VI	V	II		IV
T 14					
F 15					
M 18	Lab Open				
T 19	Oral Presentations				
W 20	Oral Presentations				
R 21	All groups report for lab clean up 9:00 AM				
F 22	<b>All Reports Due by 9:00AM</b>				

## Oral Presentations

Each student must present an oral report on one of his or her informal experiments. Unless a specific arrangement has been made in advance, a team should report on its second and third experiments. In addition to the 12-minute organized presentation, there will be approximately 3 minutes available for questions from the audience. For those students using Microsoft PowerPoint or other presentation software, a computer and video projector will be provided. A standard transparency projector will also be available.

The presentations are scheduled for Tuesday and Wednesday of the last week. There will be two concurrent sessions each morning, starting at 8:00 AM, at locations to be announced. Students should present their talk in a session attended by the instructor who supervised the informal experiment. Presenters and their team partners are expected to attend the entire session in which their team is scheduled to speak. The presenter must hand a bounded paper copy of the visual aids to the supervising instructor at the end of the presentation session.

Tuesday June 19	Session A Beckman, Chavez 1, 2, 6, 7, 11,12	Session B Klingenberg, Root, Schoenberg 3, 4, 5, 8, 9, 10
Wednesday June 20	Session A Beckman, Chavez, Schoenberg 3, 4, 5, 8, 9, 10	Session B Klingenberg, Root 1, 2, 6, 7, 11,12

## Safety Regulations in the Operations and Process Laboratory

### I. General

The operations and process laboratory introduces most chemical engineering students to experiments on a larger scale than they have experienced in previous laboratory courses. New experiences likely to be encountered involve handling increased quantities of materials, including flammable liquids used as reactants or solvents, exposure to electricity, need for ventilation, and manipulation of hand and power tools. "Acceptable safety" is mainly a matter of using common sense. The following safety rules provide an indication of considerations that prevent accidents.

Laboratory  
housekeeping  
and hygiene

The laboratory floor should be kept free of obstructions. Wet floors are extremely slippery. Water spilled on the floor should be immediately squeegeed into the nearest drain.

The work bench should at all times be kept clean and free from chemicals and equipment which are not required.

Chemical wastes from the laboratory should be disposed of in the appropriate manner (ask your instructor if you are at all uncertain as to how to proceed).

Wash your hands regularly and frequently when working with chemicals.

There is to be absolutely no consumption of food or beverages in any of the laboratories associated with ChE 424.

Protective  
clothing

Eye protection (safety glasses or goggles) is required in the laboratory at all times

Protective overalls and/or lab coats are highly recommended for wear in the laboratory. Lab coats protect you and your clothing from injury and/or contamination by chemicals.

Wear protective gloves to handle corrosive or hazardous chemicals.

Shorts, miniskirts and sandals are not allowed in the laboratory.

First Aid

Get to know where the First Aid Cabinets are located.

All corrosive acids, alkalis and other hazardous chemicals should be washed off the skin immediately.

Chemical splashes in the eye must always be washed with an abundance of water.

In cases of serious injury which need medical attention, someone else must take immediate steps to obtain medical assistance or transportation to the medical center.

**University Health Service**  
**Telephone: 262-3016**

Laboratory  
technique

Never leave a laboratory experiment unattended.

Do not fill a pipette using your mouth; use a suction filler.

Use a cloth for protection when inserting glass tubing, rods or thermometers through stopper holes. Lubricate glass/rubber interfaces with glycerol or water.

Experiments involving the use of toxic or hazardous chemicals should be conducted in fume hoods.

Remember that electricity is dangerous. Check that all wires are properly insulated.

Do not handle volatile, flammable solvents near an open flame or other source of ignition. Remember that there is absolutely no smoking in Engineering Hall.

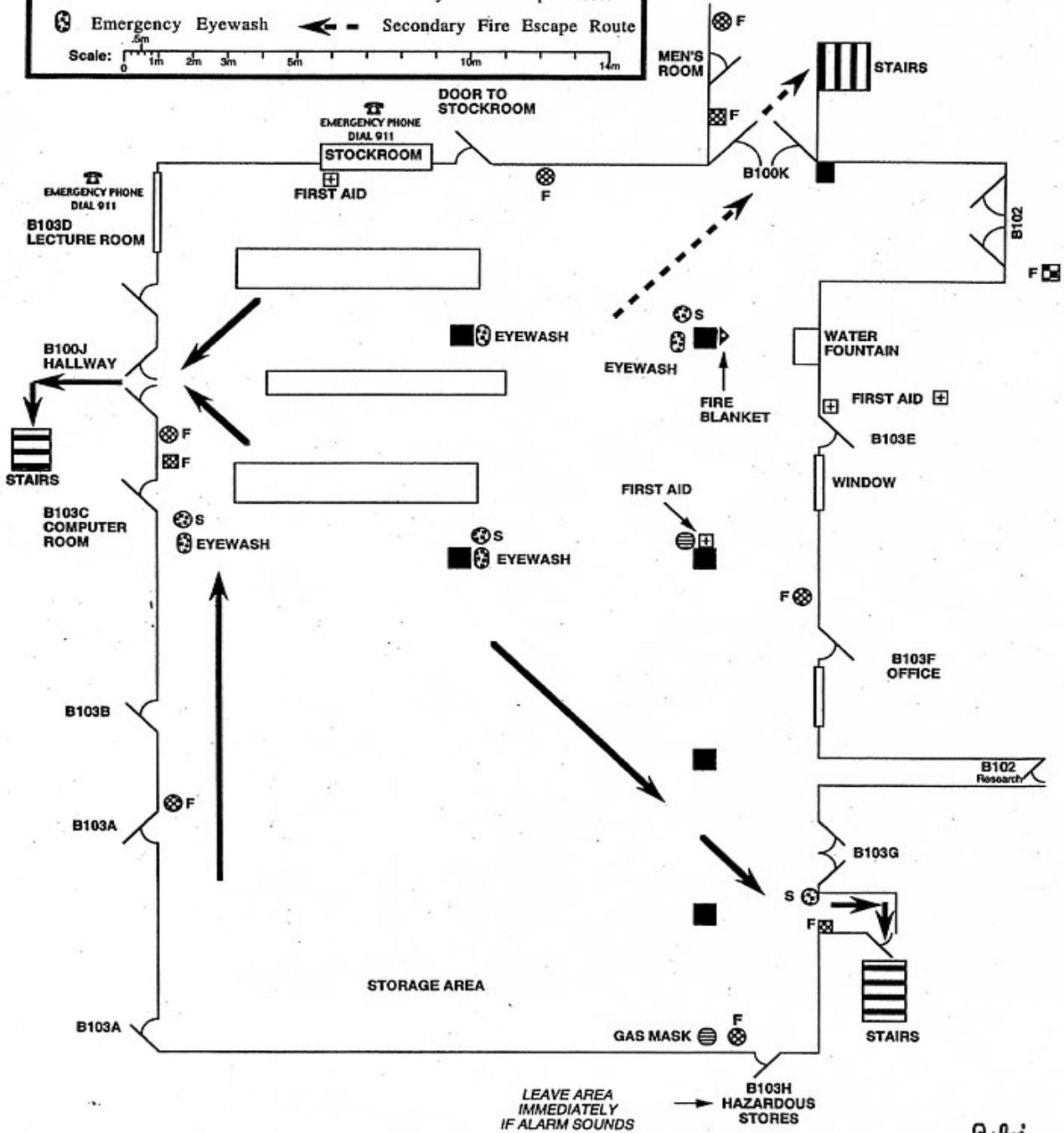
Be careful when using concentrated acids or bases; many of these materials are hazardous and corrosive and can cause burns on contact with the skin. Never add water to a concentrated acid; always add the acid slowly to the water while keeping the mixture well stirred.

**Legend**

	Gas Mask
	Fire Blanket
	First Aid Station
	Primary Fire Escape Route
	Secondary Fire Escape Route

Scale: 0 1m 2m 3m 5m 10m 14m

# Room B103 Engineering Unit Operations Lab Safety Floorplan (2003)



*R. J. Dowd*  
 Drawn by Robert Dowd  
 1/27/92

## II. Chemical Waste Disposal Procedures

(For more details, see the blue disposal notebooks in the stockroom)

<u>Chemical Waste</u>	<u>Disposal Procedure</u>
Non-halogenated organic solvents (may contain small amounts of halogenated organic solutes).	Put in correctly-labeled carboy.* Fill out waste control sheet.
Halogenated organic solvents.	Put in correctly-labeled carboy.* Fill out waste control sheet.
Non-toxic solutions of inorganic acids or bases (e.g., HCl, HNO <sub>3</sub> , NaOH, etc.).	Neutralize most acids w/ NaHCO <sub>3</sub> , very strong acids w/ NaOH solution, bases w/ dilute acetic acid. Pour down drain w/ water.
Toxic solutions of acids or bases.	Neutralize as above. Bottle, label and bring to stockroom.
Vacuum pump oil.	Bring to stockroom.
Sulfides or inorganic cyanides. Strong oxidizers or reducers. Substances that react with water (e.g., K, Na). Solids or precipitates.	Bottle, label and bring to stockroom.
Unknowns.	You are out of luck! Find out what it is <u>NOW</u> , label it and bring it to the stockroom, or dispose of it appropriately after you have identified what it is.

\* Note: DO NOT put large amounts of water in carboys. Carboys may contain only about 3 liters of water when full. If you have questions, ask Ed Paulsen or John Cannon.

## III. Materials Safety Data Sheets (MSDS)

Materials Safety Data Sheets (MSDS) play an important role in maintaining a safe environment in the laboratory. These sheets provide a great deal of information concerning physical properties, toxicity and reactivity of most common chemicals. Much information can now be found by searching the Internet. A particularly complete summary of chemical hazards is given at:

<http://msds.pdc.cornell.edu/msdssrch.asp>

## Laboratory Supplies and Equipment

Here are some guidelines to help you find your way around the lab and to locate some of the equipment you may need to use in this course. The staff is here to help you, but you can also help yourselves.

So that equipment is available to all students when they need it, it is important for all students to obey the following procedures:

1. Students using equipment for informals must sign for all equipment taken from storerooms or the instrument shop.
2. Please cross off your name and the items checked out when items are returned to the proper storeroom and made available for the next user.
3. All experimental apparatus must be labeled with the names of the students, the instructor, the topic of the experiment, the dates actively in use, and any unusual chemicals or precautions needed. Equipment left out without identified owners may be moved at instructors' discretion.

### Supplies and equipment located in the ChE Stockroom, B119, Mr. Ed Paulsen

#### A. Chemicals (see detailed lists in the stockroom)

1. Acids (Acid storage cabinet in B102)  
Some small bottles of acids are stored on the acid shelf in the stockroom.
2. Solvents (Stored in hazardous area, Summer Session solvents are located in the left hand rear shelf area). Some solvents are also stored in the hazardous refrigerator storage unit located outside the hazardous stores area in B103.

#### B. Glassware

1. Beakers, 30, 50, 100, 250, 500, 1,000, 2,000 and 4,000 ml.
2. Bottles 1, 2, 4, 8, 16 and 32 oz. sizes with screw caps. (vials in 1/2, 1, 2, 4, 6, 8 and 9.5 Dram sizes w/s. caps.
3. Flasks
  - a. Distilling (round bottom) 100, 250, 500 and 1,000 ml.
  - b. Filter (Vacuum) 50, 125, 250, 1,000, 2,000 ml.
  - c. Volumetric 10, 25, 50, 100, 250, 500, 1,000, 2,000 ml.
  - d. Erlenmeyer 25, 50, 125, 250, 500, 1,000, 2,000 ml.
4. Funnels 1, 2, 2.5, 3 and 4 in. powder; 2.5 and 4 in.; 4" polyethylene plastic  
Various sizes, in narrow and powder (wide) stem varieties.
5. Crystallizing dishes 170 x 90, 190 x 100 mm
6. Petri (Culture) Dishes 150 x 20, 100 x 15, 60 x 15 mm sizes; Plastic 100x15 mm
7. Watch glasses in assorted sizes
8. Desiccators in regular and vacuum – 2 med. vacuum, 4 in. 5 in. reg types
9. Test tubes and racks (culture tubes, 10 x 75, 15 x 85, 100 mm  
10 x 50, 10 x 75, 10 x 100, 16 x 125, 16 x 150 mm sizes
10. Tubing in Pyrex and soft grades from 3 mm up thru 12 mm;  
soft and Pyrex mixed size, rods, capillary tubes
11. Stirring rods in various lengths--8 and 12 in. are standard.

12. Beads in 0.25 inch, 1, 2, 4, 6 mm small, fine also Pyrex
13. Condensers
14. Burettes 50 and 100 ml sizes
15. Pipettes (Grad. 1/10, 1/5, 1/2, 1, 5 ml. Disposable 5 and 9 in. sizes  
Vol. 1/2, 1, 2, 5, 10, 15, 20, 25, 50, 100 ml.

C. Laboratory Bench Equipment

1. Ring stands in 20 and 36 inch heights.
2. Clamp holders, extension clamps, ring clamps  
3-claw, 4-claw extension clamps. Thermometer clamps
3. Other Types of Clamps
  - a. Hose
  - b. Pinch
  - c. Power 1/4" and up
  - d. Screw
  - e. Tubing 1/4"
4. Spatulas, Microspatulas, Scoopulas
5. Gas Burners, Bunsen (regular) and Meeker (large).  
(wing tips for Bunsen burners)
6. Tubing
  - a. Rubber-regular wall 3/16, 1/4, 5/16, 3/8, 1/2 in. ID  
thick wall for vacuum - 1/4 in. and 3/8 in. ID only
  - b. Tygon Plastic Tubing - regular wall soft polyethylene  
1/16, 1/8, 1/4, 5/16, 3/8 in. ID
  - c. High Density polyethylene - hard  
1/8, 1/4, 1/2 in. ID
  - d. Other Tubing  
Silicon 1/8 in. ID  
Tubing for MasterFlex pumps
7. Tubing Connectors and Adaptors
8. Bottles
  - a. Glass-stoppered (ground glass stoppers)
  - b. Weighing with covers
  - c. Dropping (ground glass stoppers)
  - d. Polyethylene with plastic caps - 4, 8, 16, 32 oz sizes
9. Separatory Funnels
  - a. Globe shape, 1 liter
  - b. Pear shape, 30, 50, 125, 250, 500, 1,000 ml. sizes
10. Miscellaneous Items
  - a. Aluminum foil, 12" and 18"
  - b. Parafilm, 4" dispenser and 3 ft. roll
  - c. Q-tips, Pipe cleaners, Applicator sticks, Tongue depressors.
  - d. Plastic weighing boats
  - e. Litmus paper for acid or base determinations.
  - f. Dyes and indicators. Phenolphthalein solution
  - g. Lead shot, #10, 12, 16; 1/8 in. steel balls
11. Column packing material
  - a. Raschig rings
  - b. Ceramic saddles
  - c. Shells, ceramic

- d. Hollow cylinders
12. Sand
  - a. Sea, cleaned and washed
  - b. Ottawa
  - c. White, fine
  - d. Coarse
13. Stoppers, rubber Micro, 000, 00, 0, #1 thru #14  
cork 000, 00, 0, #1 thru #28
14. Flasks, Dewar 660 ml 1,000 ml. 4,300 ml (Vacuum Jars)
15. Mortar and pestle sets – small 3 in., med. 6 in. large 7 in. huge 8 in. diameters
16. Forceps and tweezers
17. Filter papers
18. Syringes
  - a. Disposable plastic 1, 3, 6, 10, 12, 20, 60 cc. sizes
  - b. Glass Luer-lok 2, 5, 10 cc. sizes
  - c. Glass microliter, 10 and 50 microliter.
  - d. Gas tight
19. Hypodermic Needles
  - a. Disposable, Luer-lok 18, 19, 20, 22, 23, 25, and 27 ga.
  - b. Stainless, Luer-lok
20. Glass drying towers and tubes
21. Gas washing bottles (available in 500 ml. size, others possible)
22. Filter pumps (aspirators) 3/8 in. size (or use vacuum from water line)
23. Tongs, beaker, crucible, also special long lengths
24. Propipettes
25. Policeman, rubber for use with glass stirring rods.
26. Wire gauze, for use with Bunsen burners
27. Wire
  - a. Copper, bare 12, 14, 16, 18, 20, 22, 24, 26, 28, 30 ga.
  - b. Stove pipe, bare
  - c. Picture frame
  - d. Baling
28. Cords, electrical
  - a. Rubber extension 10 ft. 3-wire
  - b. Safety breaker type, multiple outlet, 3-wire w/tripper
29. Glass slides, microscope 1" x 3" also cover glass
30. Wool
  - a. Cotton
  - b. Glass (handle this with care--do not cut in stockroom)
31. Insulation
  - a. Rock wool (in Room B103A--hood area)
  - b. Glass wool
32. Valves
  - a. Glass stopcocks 2-way, 3-way, Teflon
  - b. Metal  
Needle 1/4"; Globe 1/4, 3/8, 3/4 ";  
Gate 3/8, 1, 1 1/2 "; Ball, 1/4, 3/8 " sizes
33. Funnels, Buchner
34. Cylinders, graduated
35. Flasks, volumetric

36. Glassware kit, organic chemical (call for Dr's Kit)
37. Ceramic items
  - a. Crucibles
  - b. Evaporating dishes, various sizes
  - c. Casseroles, various sizes
38. Extraction equipment
  - a. Soxlet assembly
  - b. Thimbles
39. Cork rings for round bottom flasks, etc.
40. Snoop gas leak detector
41. Gooch crucibles, various sizes, porosities
42. Screens
  - a. Copper
  - b. Aluminum
  - c. Stainless Steel, coarse, fine meshes
  - d. Galvanized
  - e. Plastic
43. Steel Pipe Fittings (for less than 1" sizes) (Machine Shop has larger sizes)
  - a. Elbows
  - b. Street els
  - c. Nipples
  - d. Caps
44. Plastic pipe fittings in smaller sizes only--less than 1"
45. Cold traps in 3 sizes
46. Wastebaskets and garbage cans, garbage bags (large)
47. Dental dam (rubber sheeting)
48. Rope, sash cord and heavy string
49. Gasket material
50. Paint brushes in various sizes 1", 2", 3"
51. Melting point tubes - open ends, closed ends
52. Posterboard in various colors  
White, Black, Lt. Blue, Green, Buff, Crimson Red, and Dark Blue
53. Construction paper in various colors
54. Food dyes in various colors  
Green, Yellow, Red, and Blue
55. Film for Polaroid Camera 230 (667 - B&W, 108 Color)
56. Various paper products
  - a. Kraft paper towels
  - b. Kim wipes, small and large sizes
  - c. Facial tissue, Kleenex, Snifles, Sneezles, etc.
  - d. Wrapping paper, brown Kraft, white drug bond.
  - e. Absorbent laboratory top soaker. Plastic backing
57. Garden hose in several sizes, also hose washers.

**Equipment Located in Room B103-Unit Operations Laboratory:**

Equipment for formal experiments  
Lab benches equipped with hot and cold water, compressed air with pressure reducing regulators, natural gas, and water seal and oil seal vacuum pump manifolds  
Fume hood  
Bench scales with weights  
Drying ovens  
Vacuum oven  
Cold water and steam service on overhead pipe lines  
Crushed ice machine and dry ice chest  
Inductively Coupled Plasma spectrometer for elemental analysis  
Spray Dryer  
Large centrifuge  
Barometer and thermometer  
Safety manual of guidelines for waste disposal and carboys for lab waste disposal

**Equipment Located in Room B103A - Instructional Fume Hoods:**

3 bench-top fume hoods  
1 walk-in fume hood  
All hoods are equipped with cold water, compressed air, natural gas, and vacuum service.

More fume hoods are available in other labs. If those in Room B103A are busy, consult with an instructor to obtain permission to use hoods in other laboratories. There is **NO EXCUSE** for carelessly exposing yourself or others to chemicals that you should know require special handling.

**Equipment Located in Rooms B103A and B103B-Unit Operations Storeroom:**

4 Centrifuges, small with tubes  
5 Chart recorders, strip chart  
5 Conductivity meters, dip and flow cells  
Constant temperature baths:  
    6 heating units  
    2 refrigerated units  
    3 immersion heater /circulating units  
Digital anemometer  
Digital pipettes  
    10-100 ml  
    100-1000 ml  
    200-1000 ml  
    disposable tips  
Digital hygrometer  
Digital multimeters  
Flowmeters and rotameters  
Heating mantles / heat tapes  
Heater / stirrers

Hot plates  
Incline gauges- 0.5", 1",2",3"  
Magnetic stirrers / stir bars  
Manometers- water and mercury  
1 Manostat cassette pump and cassettes- variable speed  
3 Masterflex tubing pumps - peristaltic variable speed  
3 pH meters / pH papers  
Pitot tubes  
Pressure transducer  
Psychrometer  
Stopwatches  
Tachometer  
Tanks, vessels, and containers  
Thermocouple indicators  
Thermocouple switches  
Variacs  
Variable speed stirrers

**Equipment Located in Room B103E-Instrument Room:**

2 Beckman UV/ visible spectrophotometers  
3 Gow-Mac thermoconductivity detector gas chromatographs with integrators  
1 Waters HPLC with UV detector  
2 Mettler digital balances  
1 Ohaus digital pan balance  
1 Sartorius digital pan balance  
1 Refrigerated constant temperature bath  
2 Magnetic stirrers with heaters

**Equipment Located in B103G:**

Storeroom for ChE 324 laboratory equipment. (Equipment checked out from this room must be kept together as used in the ChE 324 laboratory.)

**Equipment Located in B103H, Hazardous Storeroom:**

Chemical supplies- see Ed Paulsen - Stockroom B119

**Equipment Located in Room B120, Instrument Shop:**

See John Cannon, or the student helper for authorization.  
Hand tools for checkout or use in the shop on workbenches.  
Machine tools are to be used only after permission has been granted by shop personnel.  
Wood and plywood, sheet metal, plexiglass, metal stock, slotted  
Pipe lengths and tubing, pipe fittings and valves, fasteners- nuts and bolts, screws, etc.  
Angle iron

### **Note on the Machine Shop Area Related to Use by Summer Session Students**

John Cannon and our student hourly help are here to facilitate the operation of the summer laboratory. They can be helpful in locating equipment, tools, etc. They are a useful resource, as is the lab director, Dr. Eric Codner. However, all of these personnel are also involved with graduate students and professors in the design and construction of lab and research equipment. There may be times that they cannot respond immediately to your needs. Your patience will be appreciated.

- **There are hand tools which are available for summer-lab use.** The cabinet in which the tools are kept is marked, For Student Use. You must ask permission from the Machine Shop personnel to use any other shop tools.
- **The power equipment in the shop** –lathes, mills, drills, grinders, saws, welders etc.-are to be used only by shop personnel, unless you receive special permission from the Machine Shop personnel to use said equipment.
- **You may ask to use any shop supplies.** These supplies include nuts, bolts, plywood, wood, sheet metal, raw stock and other hardware.
- **Some staff member should be in the shop throughout the day to assist you.** If no one is in the shop, you must wait until someone returns. Do not assume that you are free to use the restricted machines or that you are free to take supplies without receiving the necessary authorization from shop personnel.

These guidelines are for your protection and safety as well as ours. Failure to comply could result in forfeiture of shop privileges, and a reduction in your grade for the course.

**Shop Hours are 7:45-11:45, 12:30-4:30**

## Laboratory Reports

### 1. General Instructions

Laboratory reports are to be typewritten (doubled spaced) on one side of unruled paper, 8-1/2 x 11 inches. The sheets are to be assembled in the proper order (see Item 2 below) and bound in a report folder that will be provided.

The submission deadline for reports is typically 5 – 7 days after completion of the experimental work, with the instructor in charge of the experiment setting the specific due date. Penalties for late reports will be a reduction in grade as shown in the table below. [In industry failure to meet a deadline may mean that a contract or sale is lost or that financial penalties may result. In addition, work already done may be totally wasted.]

#### Penalty Factors for Late Submission of Reports

<u>Days Late*</u>	<u>Penalty Factor</u>
0	1.00
1	0.90
2	0.85
3	0.81
4	0.77
5	0.73
6	0.70
7	0.66
8	0.63
9	0.60
10	0.57
11	0.54
12	0.50

\*Weekends after the due date count as two days.

No reports will be accepted after 9:00 AM on the last day of the course.

## 2. Contents of Reports

- a) Formal Reports
  - i) Title page
  - ii) Abstract
  - iii) Introduction
  - iv) Results and Discussion
  - v) References
  - vi) Appendix
    - a) *Sample calculations*
    - b) *Miscellaneous graphs or figures*
    - c) *Original data*
  
- b) Informal Reports
  - i) Title page
  - ii) Abstract
  - iii) Table of Contents
  - iv) Introduction
  - v) Theory
  - vi) Procedure
  - vii) Results and Discussion
  - viii) Conclusions and Recommendations
  - ix) References
  - x) Nomenclature
  - xi) Appendix
    - a) *Sample calculations*
    - b) *Miscellaneous graphs or figures*
    - c) *Original data*

## 3. Format for Reports

- a) Title page containing:
  - i) Title of the experiment
  - ii) Course number
  - iii) Experiment number
  - iv) Date of performance
  - v) Student's name
  - vi) Partner's (Partners') name(s)-indicating roles in the group, such as Group Leader, etc.
  - vii) Instructor's name
  
- b) Abstract

The abstract covers the same material as the body of the report in a concise manner and is best written after the body of the report is complete. It is usually one or two paragraphs in length (10 to 15 lines or 150 to 250 words) and should include brief statements of the following:

- i) What was done
- ii) How it was done
- iii) Major results and/or conclusions

The abstract may also indicate the experimental method if it is unusual or novel. [Sometimes only the abstract of a report may be published. Its purpose is to tell the reader whether the report contains information pertinent to his or her interests.] The abstract should be concise, precise, and informative.

c) Table of Contents (Informals only)

A listing of each section of the report and the page on which it begins.

d) Introduction

The introduction gives the background and rationale for the performance of the work being reported. It briefly states the following:

i) Subject of study

General description, feature(s) and/or application(s) of the class of equipment or process investigated.

ii) Purpose/object of the experiment and/or analysis

The reason for performance of the study as related to the particular piece of equipment or specific process studied.

iii) Preliminary appraisal of the general problem

A summary of the necessary measurements etc., which need to be made to satisfy the purpose of the study.

iv) Strategy of investigation

A discussion of the general manner in which the objective of the study is to be obtained, the measurements to be made, and the manner in which the experimental data are to be analyzed.

e) Theory (Informal experiments only)

This section connects the present work with previous studies through a review of the literature and a discussion of the pertinent theory. This section provides a basis for establishing the type of experimental data to be obtained and/or the analysis to be performed. The development also provides a basis for discussion of the results. The extent and depth of the presentation of the theory section depends on the complexity of the experiment.

f) Procedure (Equipment, Methods and/or Materials) (Informals only)

This section describes what was done and how it was done. Standard laboratory equipment and chemicals should be mentioned, but a detailed description is unnecessary. If a special piece of equipment was designed for the work, it should be described in greater detail. The description should enable a person knowledgeable in the field to repeat the experiment. Relevant flow sheets, diagrams, drawings and/or sketches are to be presented here, although in some cases it may be appropriate to relegate some of this material to an appendix.

## g) Results and Discussion

This is the most important part of the report. This section should state the findings and indicate how these are supported by the the results. Refer to tables or graphs as necessary. Indicate the limitations of the study. The discussion should show how the results have answered the question(s) posed in the introduction and elsewhere, how the results support or compare with theory and/or previously published literature, and how the results should be interpreted. A review of errors and their analysis should be included in this section.

## h) Conclusions and Recommendations

Particularly for the informal experiments, a separate section that summarizes the important results and gives recommendations for additional work may be appropriate. This section provides an opportunity to emphasize the most important outcomes of the study.

## j) References

The bibliography lists all sources of information other than the laboratory notes. Use full citations in a suitable technical format. Reference to each of these sources should appear in the text of the report where the information is used. Examples of suitable formats are available in the textbooks used in your earlier courses, e.g., Bird, Stewart and Lightfoot, McCabe and Smith, Hill, etc.

## k) Appendices (need not be typed)

- i) Sample calculations should indicate the method employed to arrive at the calculated results, including formula(s) used, units employed, and substitution of actual data. A single calculation of each type is required.
- ii) Miscellaneous graphs. These graphs are used, but are not discussed in the body of the report.
- iii) Summary of data. This appendix consists of a neat tabular presentation of all data collected.
- iv) Copies of original data book pages. If the data book is well organized, it may not be necessary to provide a separate summary.

## 4. Graphs

- a) Graphs are to be lettered so that they may be read from the bottom or right side. Each graph is to have an appropriate title and properly labeled axes (including units). Leave generous margins. Do not place any information so close to the left side of the sheet that it will be covered in the binding process.
- b) Lines and curves are to be drawn neatly with appropriate tools, not drawn freehand. Computer-plotted graphs are preferred. Each graph should occupy at least half the page.
- c) Experimental data points are to be clearly indicated.
- d) When plotting a curve representing an equation, do not indicate the points plotted to obtain the curve. These points are too easily confused with experimental data.

e) If more than one curve is drawn on a single sheet of graph paper, each curve is to be clearly labeled. Do not use colors to differentiate the various curves since most copying machines deliver black and white copy.

f) Generally, bind your graphs and tables into the report at the point where they are referred to in the text, rather than collecting them at the end. Conventional practice is to place each figure and table on the page immediately following the page on which it is first referred to.

5. Quantity does not imply quality. Reports must be both concise and complete.
6. For the most professional result, use a word processor and take full advantage of spell checkers, grammar checkers, symbol fonts, etc.
7. For most people, report writing is a skill which can only be acquired through practice. For most engineers, technical writing is an important tool for professional success.

## Lecture Notes for Distillation Formal

Date: \_\_\_\_\_

Instructor: \_\_\_\_\_

---



# DISTILLATION

## REPORT ITEMS

NOTE: Only use either accepted SI units or conventional English units in the report. Do not use non-SI metric units or nonstandard English units.

### I. Abstract

Follow by table of contents.

### II. Summary of Results

#### A. A brief introduction

#### B. Tables including the following information for each experimental run:

1. (Finite reflux ratio runs:) A reconciled material balance for the system (mole basis) for EtOH, H<sub>2</sub>O, and total moles. Use the standard format for a mass balance table. Separately, show the unreconciled "raw" mass balance numbers and the associated closure error.
2. An energy balance showing enthalpy and heat flows in and out.
3. The experimentally determined Murphree vapor efficiencies and the corresponding calculated point efficiencies.
4. A comparison of the number of actual stages with the numbers of theoretical stages determined by the McCabe-Thiele method and the stage-by-stage computer model. (At finite reflux ratio, report stage counts above and below the feed separately.)
5. Overall heat transfer coefficients and  $\Delta T_{\text{mean}}$ 's for the reboiler and condenser.

#### C. A McCabe-Thiele diagram for each run showing the experimentally determined stage conditions along with operating and "q" lines as appropriate.

### III. Discussion

Some items that might be included:

- A. Discuss the probable accuracy of the material and energy balances, and possible reasons for any major errors. How did you arrive at the reconciled balances? Determine reasonable estimates for the heat losses to the surroundings. Discuss any implications.

- B. Discuss probable errors in the tray efficiency determinations, distinguishing between systematic and random errors.
- C. What do your data show about the tray efficiencies? Compare with efficiency data in the literature, e.g. in Perry's Chemical Engineer's Handbook, 6th Ed., Chapter 18, where column efficiency data is correlated in terms of the "F" factor. How might variations in design or operating conditions explain any differences with reported values?
- D. In what other ways might the efficiencies be measured on this column? What practical improvements could be made? What effects are still unknown? How could the data obtained be used?
- E. Discuss the heat transfer coefficients obtained. Compare to expected/literature values. Are the observed values reasonable, and do the differences observed between runs make sense? Why or why not?

#### IV. Sample Calculations

##### A. Total reflux case

For one total reflux run, show the following calculations in detail:

1. Energy balances over the condenser, reboiler, and column.
2. Vapor and liquid molar flowrates in the column.
3. Tray Murphree vapor efficiencies from liquid sample analyses. Tabulate efficiencies along with tray number and mole fraction EtOH in the liquid.
4. Point efficiencies calculated from your experimental Murphree efficiencies assuming plug flow of the liquid across the tray.
5. Calculate  $\Delta T_{\text{mean}}$  and overall heat transfer coefficients for the reboiler and condenser.

##### B. Finite reflux ratio case

For one finite reflux ratio run, show the following calculations:

1. Material balance (EtOH, H<sub>2</sub>O, and total) over the column.
2. Energy balances over the condenser, reboiler, and column.
3. Vapor and liquid molar flowrates in the column.
4. Tray Murphree vapor efficiencies from liquid sample analyses. Tabulate efficiencies along with tray number and mole fraction EtOH in the liquid.

5. Point efficiencies calculated from your experimental Murphree efficiencies assuming plug flow of the liquid across the tray.
6. Calculate  $\Delta T_{\text{mean}}$  and overall heat transfer coefficients for the reboiler condenser
7. A summary of the calculation results obtained from the stage-by-stage computer model.

C. For each run

1. Plot tray temperature versus tray number, labeling curves drawn for different times before and after sampling, to show how constant operation was during the sampling period.
2. Construct a McCabe-Thiele plot to compute the number of theoretical stages required.
3. Determine predictions of  $E_{mv}$  for your run from the data correlation in Perry's based on the "F" factor.

V. Appendix

- A. Data sheets. (Only one report need have the original data sheets; other group members may submit copies.)
- B. Calibration curves and any other diagrams used in your calculations but not specifically discussed elsewhere.

VI. Additional points are reserved for professionalism in the laboratory and the general appearance of your laboratory report.

EQUIPMENT MANUAL

8-Inch Distillation Unit

- I. General description
- II. Operating instructions
  - A. Pre-startup Procedure
  - B. Start-up Procedure (Total reflux)
  - C. Finite Reflux Operation
  - D. Data Collection
  - E. Tray Sampling Technique
  - F. Sample Composition Determination
  - G. Column Shutdown Procedure
  - H. Emergency Column Shutdown Procedure
- III. Transfer of Process Liquids
- IV. Safety Considerations
- V. Appendices
  - A. Diagram of Distillation Unit
  - B. Data on Ethanol-Water System
    - Vapor-liquid equilibrium diagram
    - T-x-y diagram
    - Composition as a function of density and temperature
  - C. Rotameter Flow Rate Calibration Charts and Refractive Index Charts
    - Feed and bottoms product
    - Reflux and top product
    - Temperature corrections to rotameter flow rates
    - RI of ethanol/water mixtures, variation with mole% or mass%
  - D. Data Sheet Forms
    - Flow rates
    - Temperatures of process streams
    - Tray temperatures
    - Sample compositions

## I. General Description

The major components of the distillation unit were manufactured and assembled by APV Equipment Inc., Tonawanda, New York. Personnel from the Department of Chemical Engineering designed the sampling and control systems. Final assembly was performed by Chemical Engineering Department shop personnel and student help.

Blue prints on file in the Chemical Engineering Department Offices give specific details on the construction of the major components. The following information summarizes the construction details of the distillation unit.

### Column

The column is 8 inches in diameter by ca. 25 feet in length, constructed of stainless steel. At the base of the column is a 12-inch diameter by 2-foot long section of tube that acts as the bottom accumulator. The column is assembled from 10-inch length spool sections between which are clamped the valve trays with gaskets to form a seal. Yokes placed at several points along the column allow the column and accumulator to be suspended from U-beams attached to the supporting structure.

### Trays

The column has 28 valve trays spaced at 10-inch intervals. Each tray has five 2-inch diameter valves that are placed in an "X" pattern. (See Fig. 1) The down-comers that carry liquid from a given tray to the tray below consist of two 3/4-inch diameter tubes. These tubes project one inch above the tray to form the outlet weir. The two inlet weirs consist of a 1-inch length of 1-1/2 inch diameter tubing welded to the top surface of the tray on the opposite side of the tray from the outlet weirs. The down-comers from the tray above project into the section of 1-1/2 inch tubing to form a liquid seal that prevents vapor from going up the down-comers.

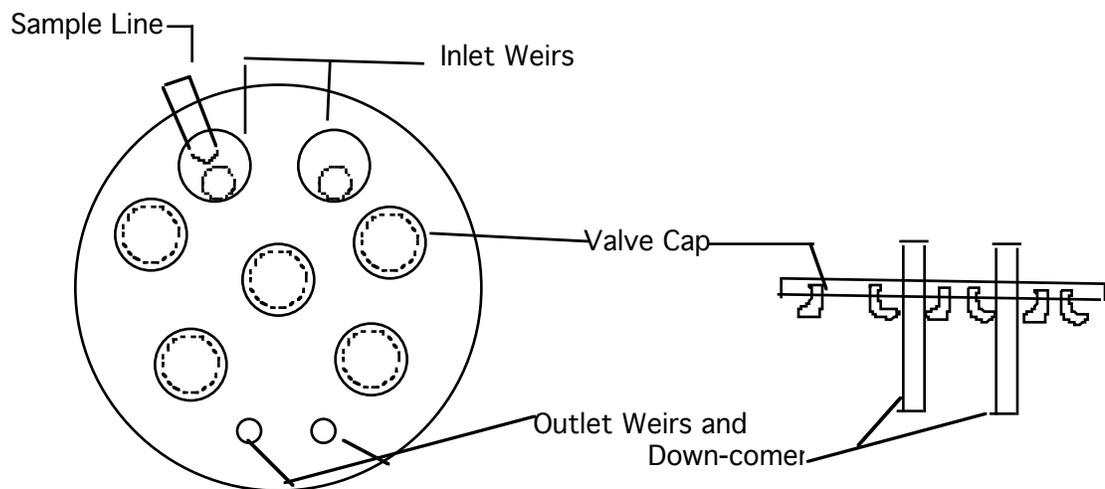


Figure 1. Valve Tray Design

Sample taps

Liquid samples may be taken from all trays except number 28 (the bottom tray). Each sample tap consists of a 1/8-inch (nominal) pipe section that passes through the column wall and projects into the inlet weir. Thus the liquid sample from tray "n" is actually taken from the inlet weir of tray "n-1". The sampling procedure is discussed in the operating instructions below.

Reboiler

The reboiler is a vertically oriented shell-and-tube heat exchanger. The exchanger has 124 tubes, each 3/8-inch O.D. x 22 ga x 24-inch long, arranged in a single pass orientation. One-half of the tubes have been blocked to reduce the reboiler capacity. Based on the outside diameter of the tubes there are 12.2 ft<sup>2</sup> (1.13 m<sup>2</sup>) of usable heat transfer area. The shell side where the steam condenses has 45% cut baffles spaced at 6-inch intervals.

Condenser

The condenser is a horizontally oriented shell-and-tube heat exchanger. The exchanger has 124 tubes, each 3/8-inch O.D. x 22 ga x 36-inch long, arranged in a 2-pass orientation with cooling water flowing through the tubes. Based on the outside diameter of the tubes there are 36.5 ft<sup>2</sup> (3.39 m<sup>2</sup>) of heat transfer area. The shell side where the vapor condenses has 45% cut baffles spaced at 6-inch intervals.

Feed Preheater and Product Coolers

All three of the heat exchangers are shell-and-tube exchangers with 64 tubes 1/4-inch O.D. x 26 ga. Specifications of the three exchangers are summarized in the following table:

	<u>Feed Preheater</u>	<u>Product Coolers</u>	
		<u>Bottoms</u>	<u>Top</u>
Tube length, inches	36	24	14
Area, ft <sup>2</sup> (m <sup>2</sup> )	12.5 (1.17)	8.4 (0.779)	4.89 (0.454)
Number of tube pass	4	4	4
Baffle spacing, inches	2-7/8	1-1/2	1-1/2
Baffle cut	45%	25%	25%
Tube-side fluid	Feed	Water	Water
Shell-side fluid	Steam	Product	Product

Storage Vessels

Three stainless steel storage vessels are supplied to store the feed, bottom product, and top product. The feed and bottom product vessels are identical and hold approximately 140 gallons (540 L). The top product vessel holds about 125 gallons (470 L). Each vessel is supplied with a gage glass to indicate the liquid level. A piping manifold located on the wall between the feed vessel and the distillation unit support structure permits process liquids to be pumped from one vessel to another using the large transfer pump located in the basement. The transfer pump may

transfer liquids into or out of the feed and bottoms product vessels. The transfer pump can only pump liquid out of the top product vessel.

Pumps – The distillation unit has four pumps.

Transfer pump: This is a centrifugal pump that is capable of pumping 20-25 gallons per minute at a head to 70 feet of fluid. The pump may be started and stopped with switches located on the distillation support structure in both the basement and the first floor. The pump can only be started if the safety locks of both switches are in the released position. As is true of all centrifugal pumps, the transfer pump should be started with the inlet valve open and the outlet valve closed. As soon as the pump is up to speed the outlet valve may be opened. The transfer pump should not be operated for extended periods of time with the outlet valve closed. VALVES ON THE MANIFOLD BETWEEN THE TOWER SUPPORT STRUCTURE AND THE FEED STORAGE VESSEL ARE FOR USE ONLY WITH THE TRANSFER PUMP.

Feed and bottom product pumps: These two pumps are identical. They are positive displacement, reciprocating pumps. Variable speed drives permit the flow rate produced by the pumps to be controlled. Since they are positive displacement pumps the outlet valves located at the rotameters should never be closed. If a pump is not in use, as is the case for total reflux operation, the inlet valve to the pump may be closed to prevent leakage flow through the pump. These pumps are supplied with an unloader valve in a by-pass line around each pump to prevent damage to the pump if an outlet valve should inadvertently be closed.

Reflux pump: This pump is a positive displacement, gear pump. It delivers liquid at a very steady rate; thus, damping chambers at the rotameters are not required. Because the pump is positive displacement, the same restriction on closing the outlet valve applies here as for the feed and bottom product pumps.

Pump controllers: All three operating pumps receive power from brown controller boxes on the main control panel (feed and reflux pumps) or at the column base (bottoms product pump). They are switched on by moving the main toggle from 'off' to the spring-loaded 'start' position, then releasing the switch to the middle 'on' position. They are switched off by returning the toggle to the bottom, 'off', position. The feed pump rate is controlled by the front-panel potentiometer. The reflux and bottoms pump rates are set by the green level controllers on the reflux accumulator and bottoms accumulator, so the front panel potentiometers on these boxes are disabled and have no effect on the pump operation.

### Rotameters

Flow rates of feed, bottom product, reflux and top product are measured using rotameters that are located on the main instrument panel.

The feed and bottom product rotameters have a flow rate range of 0.8-7.8 liters/min with a liquid of 1.0 specific gravity. Because the feed and bottom product pumps produce a pulsating flow, air-filled damping chambers are located at the inlets of the

feed and bottom product rotameters to smooth the flow. Excessive oscillation of the rotameter floats probably indicates that there is insufficient air in the damping chambers.

The top product and low-range reflux rotameters have ranges of 0.2-2.3 liters/min with a liquid of 0.81 sp. gr.

The high-range reflux rotameter has a range of 1.1-11.0 liters/min with a liquid of 0.81 specific gravity.

Correction charts are provided for determining the flow rate through the rotameters if the specific gravity and temperature differ from the standard values.

### Water meters

Three water meters are provided for measuring the flow of city water to the condenser and the top and bottom product coolers. The condenser is on the second floor while the product coolers are on the first floor. All the water meters and their associated manual control valves are located on the first floor. The meters are totalizing meters; i.e., they show the total amount of water (in cubic feet) that has flowed through the meter. To determine water flow rate the amount of water passing through the meter in a measured period of time must be recorded. The water meters are the only non-metric instruments on the distillation unit.

### Instrument Panel

The main panel-board from which almost all the operations of the distillation unit are controlled is located on the first floor. On the panel are located the five rotameters already mentioned, pump speed controls, reflux ratio valve, the tray sample switch, and digital readouts of tray temperatures, process stream temperatures and reboiler steam pressure. Use of these controls will be described in the operating instructions section.

## II. Operating Instructions

### A. Pre-startup Procedure

1. Feed preparation: Determine the composition of the mixture in the feed tank by taking a sample with a syringe at the outlet line of the vessel. If the composition is about 10 mole % ethanol, the feed is satisfactory. If it is less than 10 %, additional ethanol should be added using the procedure indicated in the section on transfer procedures. Mix the contents of the feed vessel by using the transfer pump with the valves to and from the feed vessel open.
2. Check the main electrical power switch located in the basement on the concrete column nearest to the reboiler, and turn on if necessary.
3. Check the digital readouts on the instrument panel to determine that they are operating. Do not turn on any pumps yet.

4. Start water flow to the main condenser (valve on first floor). Set flow rate at 6-8 gallons per minute. This will likely need to be reset later.
5. Start water flow to steam condensate cooler (basement). There is no meter for this water stream; set it at a moderate flow rate.
6. If operation is to be at finite reflux, start water flow to the top product cooler (first floor). Use an approximate rate of 1-2 gpm.
7. If operation is to be at finite reflux, start water flow to bottoms product cooler (first floor). Use an approximate rate of 5 gpm.
8. Check that the valves on the outlet lines from the rotameters are open. Open the valves at the inlets of the feed and bottoms product pumps. The locations of the pumps are as follows:
 

Bottoms product	Basement. Note that there is a heat exchanger between the pump and its inlet valve.
Feed	First floor.
9. Check that the steam control valve (red handwheel below the instrument panel) is closed (clockwise). Open the main steam valve located near the ceiling of the first floor. It is the valve with the large blue hand-wheel that can be reached from the ladder at the platform #3 level. Once condensate has cleared and steam is flowing clearly, the pressure gauge just downstream of the valve should show 30 to 35 psig. After steam has been flowing for 10-15 minutes, you may adjust the steam pressure regulator valve (orange colored body) to give 30 to 32 psig on the second pressure gauge if necessary. Turning the regulator valve handle clockwise increases the pressure.
10. Check that one of the feed valves located along the column is open. All other valves to the column should be closed. For total reflux, you should feed as close to the reboiler as possible. For finite reflux, you will want to choose another appropriate tray for feed.
11. Prepare for column startup by turning the rotameter selection valve (below the reflux rotameters) to the high range. Prepare for total reflux operation by turning the reflux ratio valve (below the top product rotameter) to the off position (clockwise). Do not use excessive force when closing the valve. It is a needle valve and may be damaged easily.
12. Turn on the power supply for the level controllers. This is the switch on the small grey box below the reflux pump speed control on the instrument panel.

#### B. Start-up Procedure

1. Check liquid level in the gage glass of the bottoms accumulator. If a meniscus is not visible, use the apparent width of the red indicator line on the back of the gage glass to determine if it is entirely full or empty. Alternatively, you may momentarily open the drain valve at the bottom of the gage glass. If liquid drains out, the accumulator is completely full. If very little or no liquid appears the accumulator is empty.

2. a. If the accumulator is partially or completely full with an undetermined material composition, it should be emptied and refilled with feed of known composition. The bottoms pump may be used to remove the excess liquid. Press the 'start' switch on the speed control located at the #1 platform level to the start position. The pump will start and flow will be shown on the bottoms product rotameter. When the meniscus has dropped to about the mid-point of the bottoms accumulator gage glass and the pump stops pumping, secure the bottoms pump. If you wish to empty the accumulator completely, you may drain remaining liquid through the valve at the bottom of the gage glass into a container appropriate for manual transfer of removed liquid to the feed tank.
- b. Once the accumulator is completely empty, the feed pump must be started to transfer liquid into the column. Move the feed pump speed control switch to the 'start' position and set the flow rate of feed at ~5 LPM. When the meniscus reaches the mid-point of the bottoms accumulator gage glass, you may proceed.
3. Start steam flow to the reboiler using the red handwheel below the instrument panel. Use a steam pressure of 60 to 80 kPa. Control of the steam pressure will be difficult during the startup period. An exact pressure is not necessary.
4. Observe the level of liquid in the bottoms accumulator. Add feed at 3-4 LPM as necessary to maintain the level near the mid-point of the gage glass. If the liquid level drops to the bottom of the sight glass, you should increase the feed rate, decrease the steam input, or both, as needed to keep liquid in the reboiler.
5. Monitor the heating of the column by reading the temperatures using the tray thermocouples. With this thermosiphon configuration, tray #28 will heat up first, then the bottoms accumulator and the trays proceeding up the column.
6. Before the vapor product thermocouple indicates that vapor has reached the top of the column, the reflux pump should be started. Press the switch on the speed control to the 'start' position. Monitor the liquid level in the top product accumulator (second floor). When the liquid level is seen rising in the accumulator, the reflux pump should begin pumping and liquid flow will be observed in the reflux rotameter.
7. Set the steam pressure in the reboiler to the value needed to achieve the condensate flowrate (reflux + top product) specified for the experiment. Be aware that it may take 10-15 minutes for any change in reboiler steam pressure to produce a new, steady vapor flow at the top of the column. Avoid sudden upsets that may drive composition oscillations along the column. It may be beneficial to make large changes in operating conditions in several smaller steps over a period of several minutes.
8. Monitor the temperature profile in the column by tabulating temperature versus plate number IN YOUR LAB NOTEBOOK at 10-minute intervals.

**CAUTION:** If at any time it becomes necessary to switch from the high range to low range reflux rotameter, or vice-versa, the **REFLUX PUMP MUST BE STOPPED** during the switch-over. This is necessary because the reflux pump is a positive-displacement pump and there is a momentary blocking of the flow path as the valve is switched from one position to the other.

9. Continue to monitor tray temperatures to determine that steady-state operation is being achieved. Observe the reboiler steam pressure carefully to assure that it remains constant within 2-3 kPa of your current target. Observe the reflux flowrate carefully to assure that it remains at the desired level. If the reflux flowrate shows a steady drift away from the desired level, you may choose to adjust your steam pressure setpoint up or down appropriately. Be careful not to adjust the steam setpoint excessively, as this may upset the column. Typically, after an adjustment up or down by 5 kPa, you should wait 5-10 minutes to observe the effect before deciding on another setpoint adjustment.
10. Adjust the condenser cooling water rate to give at least a 20-25°C temperature rise. Too small a temperature change results in excessive error in the energy balance calculation.
11. If the operation is a total reflux run, see section II-D for data collection instructions. For finite reflux operation see section II-C for additional steps.

### C. Finite Reflux Operation (Top and bottom products)

1. Start-up of the distillation unit is the same as for total reflux in section II-B.
2. When total reflux operation has been achieved, switch to finite reflux operation by starting feed flow at a rate specified by the instructor. Complete steady-state operation at total reflux is not necessary before this switch is made. To avoid a column upset caused by sudden influx of cold feed, you may want to reach the desired feed flowrate in a series of (1 LPM) steps separated by a few minutes, while observing the column response (tray temperatures and reflux flowrate). At higher feed flowrates, you may need to increase the reboiler steam pressure to maintain the desired vapor flow to the top of the column.
3. Start the bottom product pump.
4. Set the top product withdrawal rate using the control-panel valve located below the top product rotameter. If the column is to operate at steady state, the ethanol content of the top product stream and the ethanol content of the feed stream must balance. Recognize that the top product stream is drawn from the reflux pump outlet, and therefore the column reflux will decrease by a comparable amount. Your instructor may specify that you operate at a particular reflux ratio, or may specify other flow values at the reflux condenser. It may be necessary to switch between the two reflux rotameters to obtain desired reading accuracy. **OBSERVE** the caution regarding clear flow paths downstream from constant-volume pumps described in section II.B.8.

5. Monitor the tray temperature profile to determine that the sharp change in temperature is in the desired position and that steady state is being achieved. Change the steam pressure to move the temperature profile (and associated ethanol composition profile) in the desired direction.
6. When steady state is achieved, proceed to collect data as indicated in section II-D.

#### D. Data Collection

When steady-state operation is achieved, the data indicated below should be collected. The data sheets provided in at the section end will be helpful in assuring that all necessary data are recorded.

##### Flow rates:

- Feed, bottom product, and top product (finite reflux runs only)
- Reflux
- Condenser water
- Reboiler steam

##### Temperatures:

- Liquid at the rotameters (only reflux for total reflux runs)
- Feed to feed preheater (finite reflux only)
- Bottoms accumulator
- Vapor off top of column
- Reflux
- Condensate
- Condenser cooling water - inlet and outlet
- Trays

Pressure: Reboiler steam

Liquid compositions (duplicate samples in all cases):

Feed (finite reflux only), top and bottom accumulators

These samples are to be taken manually by attaching a 5 or 10 cm<sup>3</sup> syringe to the Luer-Lok fitting at each location. The feed sample is taken at the outlet line from the feed vessel. Do not turn the syringes on the Luer-Lok fittings with excessive torque.

Trays

Take liquid samples from 'active' trays with temperatures from 79 to 100 °C. At temperatures outside this range the composition change from tray to tray is too small to allow accurate tray efficiency calculations. See section II-E for the sampling procedure.

### E. Tray Sampling Technique

Pneumatically operated syringe drives are used to take samples from the desired trays simultaneously.

1. Open the air supply valve for the sampling system. This valve is located above the instrument panel near the sample control knob. The valve must be fully opened (counter-clockwise). Be certain that no one is working on the syringe holders while the air supply valve is opened since the plungers will operate momentarily.
2. Select the 'active' trays to be sampled (as indicated in section II-D).
3. Place the sample control knob on the instrument panel in the "sample position." All the plungers should move to their lowest position. DO NOT move the knob from this position while anyone is working on the syringe drives. The plungers operate with considerable force, and serious injury is possible if operators have their hands in the way when the plungers move.
4. At each of the selected trays insert a syringe into the drive. This is done by pulling the syringe piston partially out of the barrel. Turn the syringe onto the Luer-Lok fitting. Avoid excessive bending of the fine tube connecting the Luer-Lok valve to the column. Hold the Luer-Lok valve to prevent torque from being transmitted to the tubing, and avoid excessive torque in turning the syringe barrel on the Luer-Lok valve. Fit the tabs on the syringe barrel and the syringe piston into the two slots on the drive. Turn the brass retainer to hold the syringe in the drive. Set the Luer-Lok valve to the open position. Repeat the above procedure at each of the selected trays. Since the sampling drives alternate from one side of the column to the other, it may be advantageous to have two persons do the placing of the syringes.

The syringe drives of the trays not being sampled are left empty.

5. When the syringes have all been placed in the desired drives, and everyone is clear, turn the sample control knob on the instrument panel to the purge position. This will drive the syringe pistons in and purge the sample line. Several seconds after the syringes have moved to the purge position, return the control knob to the sample position. The samples will be withdrawn from all the desired trays simultaneously. Check to see that this has occurred.
6. Immediately close all the Luer-Lok valves. Carefully remove the syringes from the drives using a technique that is the reverse of the installation procedure. Place a suitable cap on each syringe as it is removed.

### F. Sample composition determination

Sample compositions are determined via one of the following methods, as may be recommended by the instructor:

1. **Densitometer Method:** The composition may be determined by measuring the liquid density at an accurately controlled temperature and determining the composition from the known dependence on density. A Mettler-Paar

densitometer is used, and has its own operating manual. Pay particular attention to obtaining temperature equilibrium during the measurement and avoiding any bubbles in the injected sample.

2. **Refractive Index Method:** The optical properties of ethanol-water mixtures vary systematically with composition and temperature. Using the supplied charts or fitted equations, you may determine composition from refractive index (RI) measurements under controlled conditions. This test takes only a few drops of solution, and is quick and convenient. However, the curve for RI vs. composition passes through a maximum at high ethanol concentrations and a single RI reading can indicate two different compositions in this range, so definitive measurements are possible only if i) you already know the approximate concentration, to decide which branch of the curve the sample is on, or ii) you dilute the sample enough to ensure all readings are collected from the low curve, and then obtain the initial ethanol composition from this reading and data for the dilution ratio.

**Dilution protocol:** It is NOT necessary to have exact 1:1 dilution, but you do need to record the actual masses accurately. Put a small amount of pure water in a tared vial, record the mass, re-zero the scale, add a similar amount of sample, and record the mass. Mix carefully and measure. Convert the RI of the diluted sample to mass% EtOH, and use the dilution mass data to determine the mass% (or mole%) EtOH of the original sample.

**Reading procedure:** Obtaining accurate RI values requires good experimental technique. To get a reading, match the light/dark interface at the cross-hair intersection. Adjust the focus and optical dispersion settings as needed to get the sharpest interface. Like all mechanical linkages, there is hysteresis (backlash) in the RI dial, so the most accurate readings are obtained by always approaching the match from the same direction. Each operator should set the instrument reading scale with deionized water; practice with water until you get reliable, reproducible readings. Recheck the calibration as often as necessary.

#### G. Column Shut-down Procedure

1. Shut off steam using the steam control valve.
2. Switch off the feed pump.
3. While trays drain to the bottom accumulator and the bottoms pump is conveying accumulated contents to the bottoms storage tank, check the stream temperature and increase the water flowrate to the bottoms cooler if necessary.
4. After the bottoms pump finishes transfer of accumulated liquids from the column, switch off the reflux and bottoms pumps. Turn the reflux ratio valve to the off position (clockwise) if finite reflux was being used.

5. Turn off the valve at the inlet of the feed pump if concluding operations for the day.
6. After approximately five minutes the cooling water to all heat exchangers may be turned off.
7. Close the sampling system air supply valve and close the main steam valve and main water supply valve.

#### H. Emergency Column Shut-down Procedure

1. Shut off steam using the steam control valve.
2. Switch off the feed pump.
3. Open the top product withdrawal valve fully.
4. Increase cooling water to top product and bottom product coolers to ensure all exit streams are adequately chilled before entering storage tanks.

### III. Transfer of Process Liquids

A transfer pump (20-25 gpm) is installed in the basement to transfer process liquids from one vessel to another. Switches for starting and stopping the pump are located on the distillation support structure at the basement and first floor levels. The safety latches of both switches must be released before the pump may be operated. There are basically two types of transfer operations.

1. Transfer of liquid between long-term storage drums and the distillation column feed tank. This operation is normally done at the beginning and end of the summer sessions, and is to be performed with faculty supervision. Temporary hose connections are made in the basement between the storage drum and the inlet or outlet of the pump.
2. Transfer of liquid from the top and bottom product tanks to the feed tank. These transfers are made at the end of each finite reflux run to prepare feed for the next run. It is particularly desirable to return the top product tank contents to the feed tank shortly after column shutdown (rather than waiting for the following morning), as evaporative losses will be highest from the high-ethanol storage tank.

Valves for these transfers are on the pipe manifold located between the column support structure and the feed tank. **NOTE THAT VALVES ON THE MANIFOLD ARE USED ONLY WITH THE TRANSFER PUMP.** The normal position of all the valves is closed (handles at 90° to the pipe). Arrows on the pipes indicate the direction of flow in each pipe; i.e., to or from a specified tank. As an example of a transfer consider the pumping of liquid from the bottom tank to the feed tank.

- a. Close the valve at the bottom of the gage glass on the bottom tank. This is necessary because the gage glass is on the outlet line and the suction of the pump will cause air to be drawn down the gage glass.

- b. Open the manifold valve on the bottoms line with the arrow pointing away from the bottoms tank.
- c. Start the transfer pump.
- d. Open the manifold valve on the feed line with the arrow pointing toward the feed tank.
- e. Observe the level rising in the gage glass of the feed tank.
- f. When the required amount of liquid has been transferred, close the valve on the manifold line to the feed tank and switch off the transfer pump.
- g. Close the valve on the manifold line from the bottoms product tank and open the valve on the bottoms product gage glass.

#### IV. Safety Considerations

##### A. Equipment: Safety features of the equipment are listed below:

###### 1. Pressure

The column was tested by the manufacturer at 30 psig working pressure. In the experiments performed in the CBE 424 lab the unit is operated at atmospheric pressure (~0 psig).

The heat exchangers were tested by the manufacturer at 250 psig. For the steam heating exchangers we use steam at a maximum pressure of 40 psig. For the water-cooled heat exchangers the water pressure is in the range from 50-80 psig.

The rotameters are rated at 250 psig at the temperatures used in the CBE 424 experiments.

###### 2. Electrical

Ethanol vapors are heavier than air, so vapors from ethanol spills will tend to collect along the basement floor. Thus, all electrical equipment (motors and switches) located within six feet of the basement floor is explosion proof.

All electrical equipment is connected to earth ground.

###### 3. Mechanical

Rotating shafts associated with pumps and motors are shielded.

Safety gates are placed at the ladder entries on each platform.

Gage glasses on the accumulators are shielded and provided with shut-off valves at each end of the glass tube.

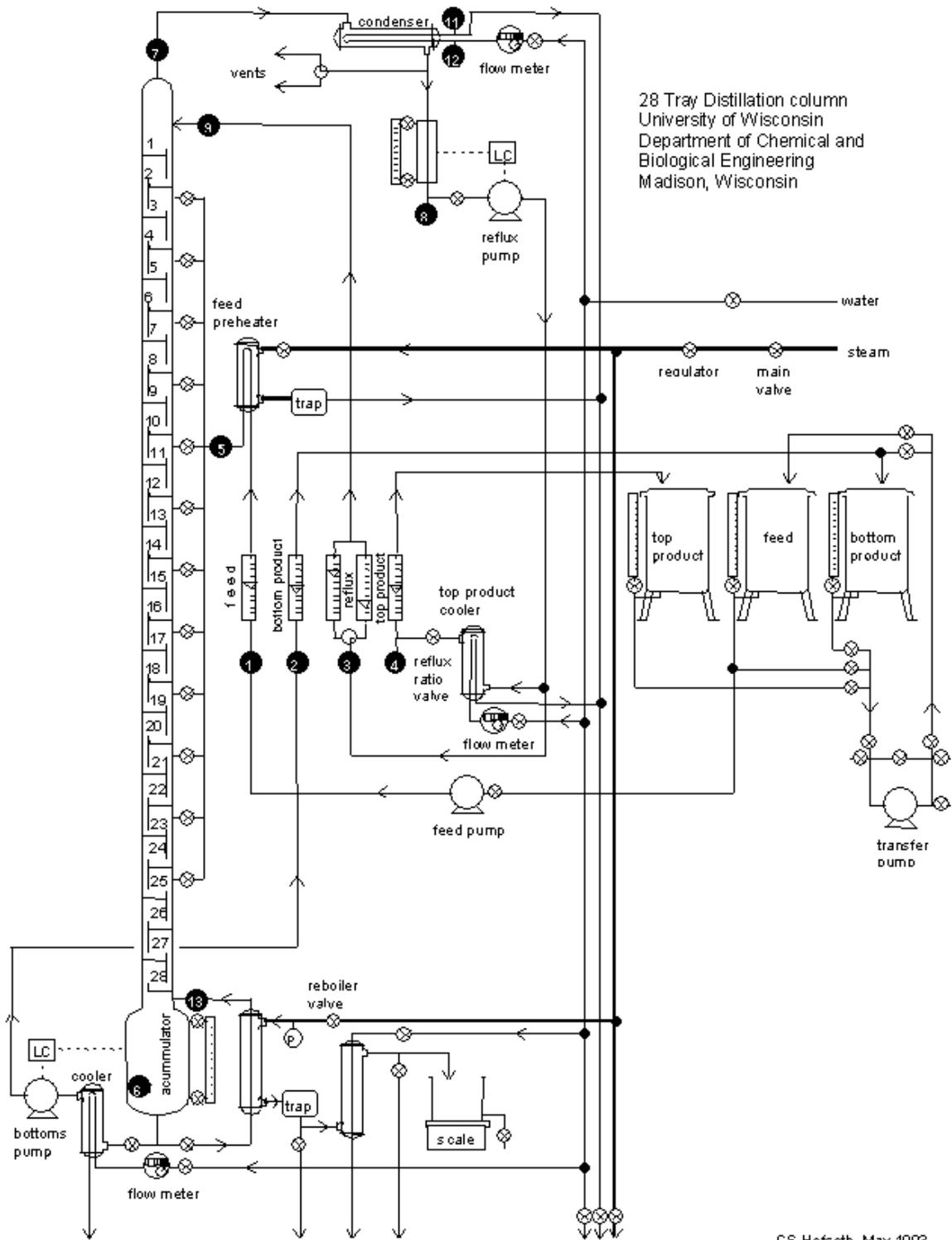
**CAUTION:** Steam lines to the reboiler and feed preheater are insulated. However, the column and the process steam lines connected to it are not insulated. Students must be careful when working near these pieces of equipment.

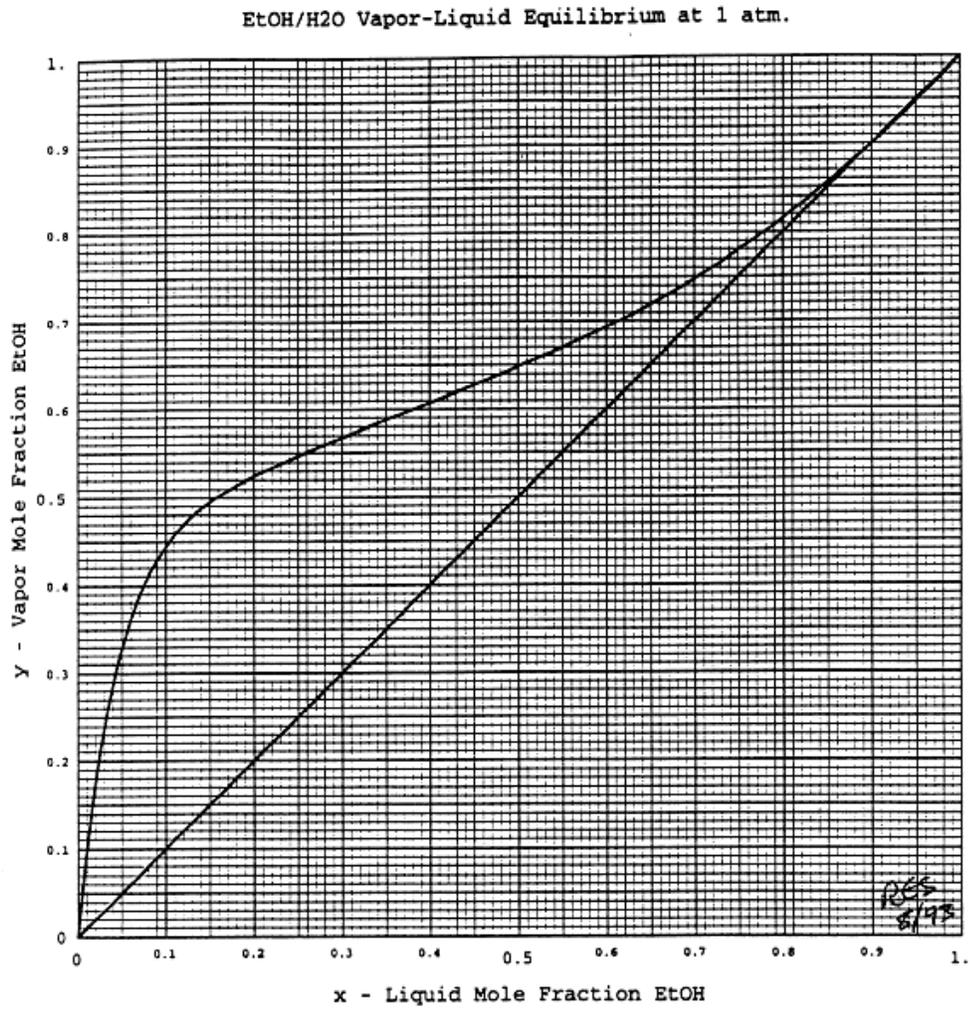
Water for the unit enters through an anti-backflow valve. This prevents water in the distillation unit from flowing into the city water mains if the pressure in those mains should drop below the pressure in the distillation unit.

#### B. Operators

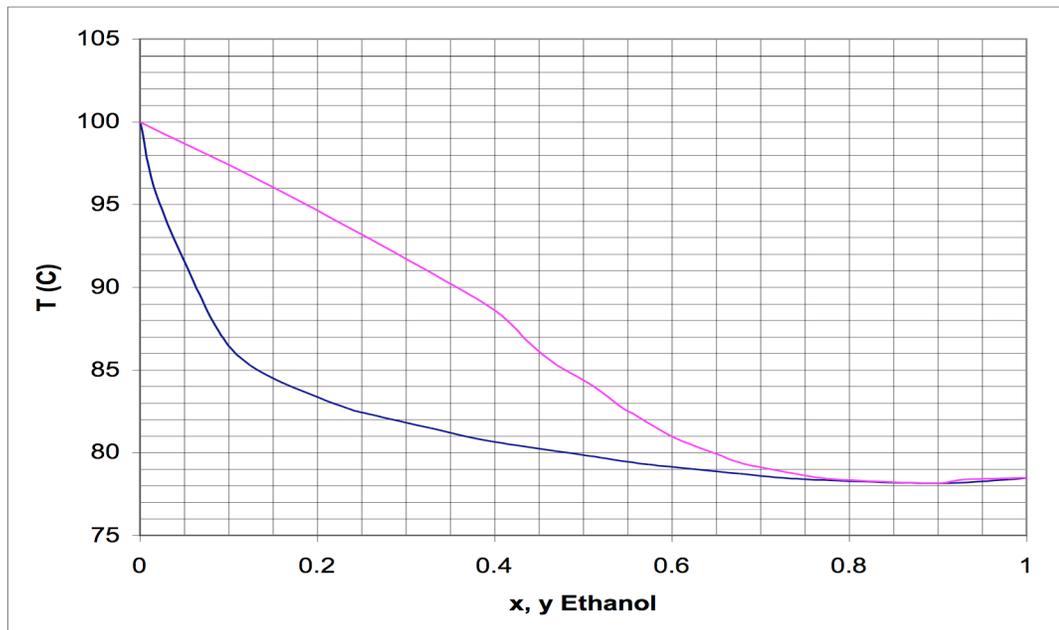
While proper design of equipment is necessary for safe operation, observance of safety rules by the operators is required if personal injuries are to be prevented. The following do's and don'ts must be observed:

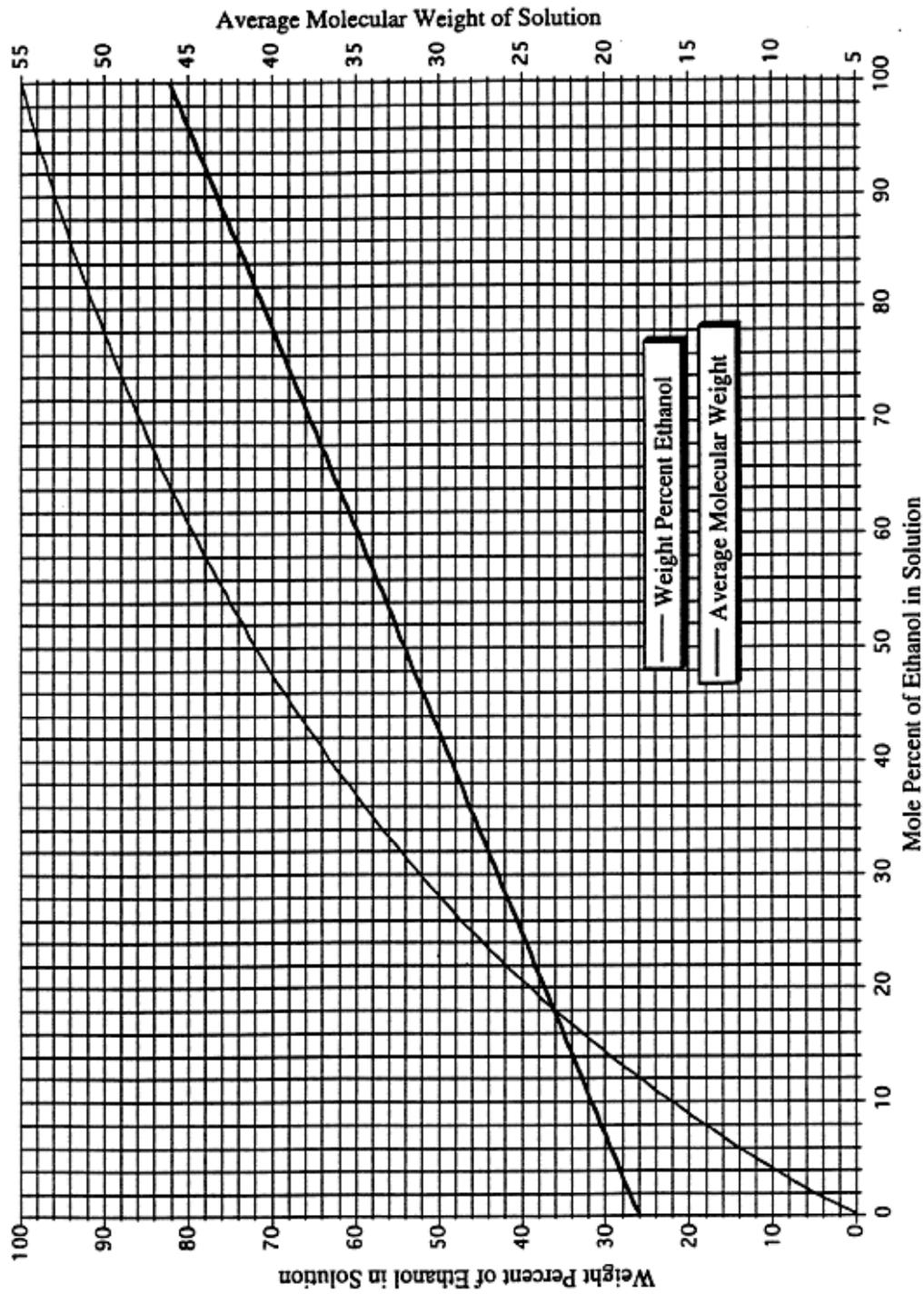
1. Do not attempt to defeat built-in safety features. Safety shields are to be removed only by faculty or shop personnel.
2. Do not block open the gates leading to the ladders on the platforms.
3. Do have a firm hold of the ladder with both hands before stepping from the platform to the ladder. Watch that you do not inadvertently grasp parts of the hot column when stepping to or from the ladder.
4. Do not operate the sampling system while anyone is inserting or removing syringes.
5. Do not lean on or step on the thermocouple wiring or wiring chases. These will not support your weight; if overloaded, you may damage the wires or hurt yourself.





(note that this plot is customarily shown with equal x and y axis scales – i.e., square)



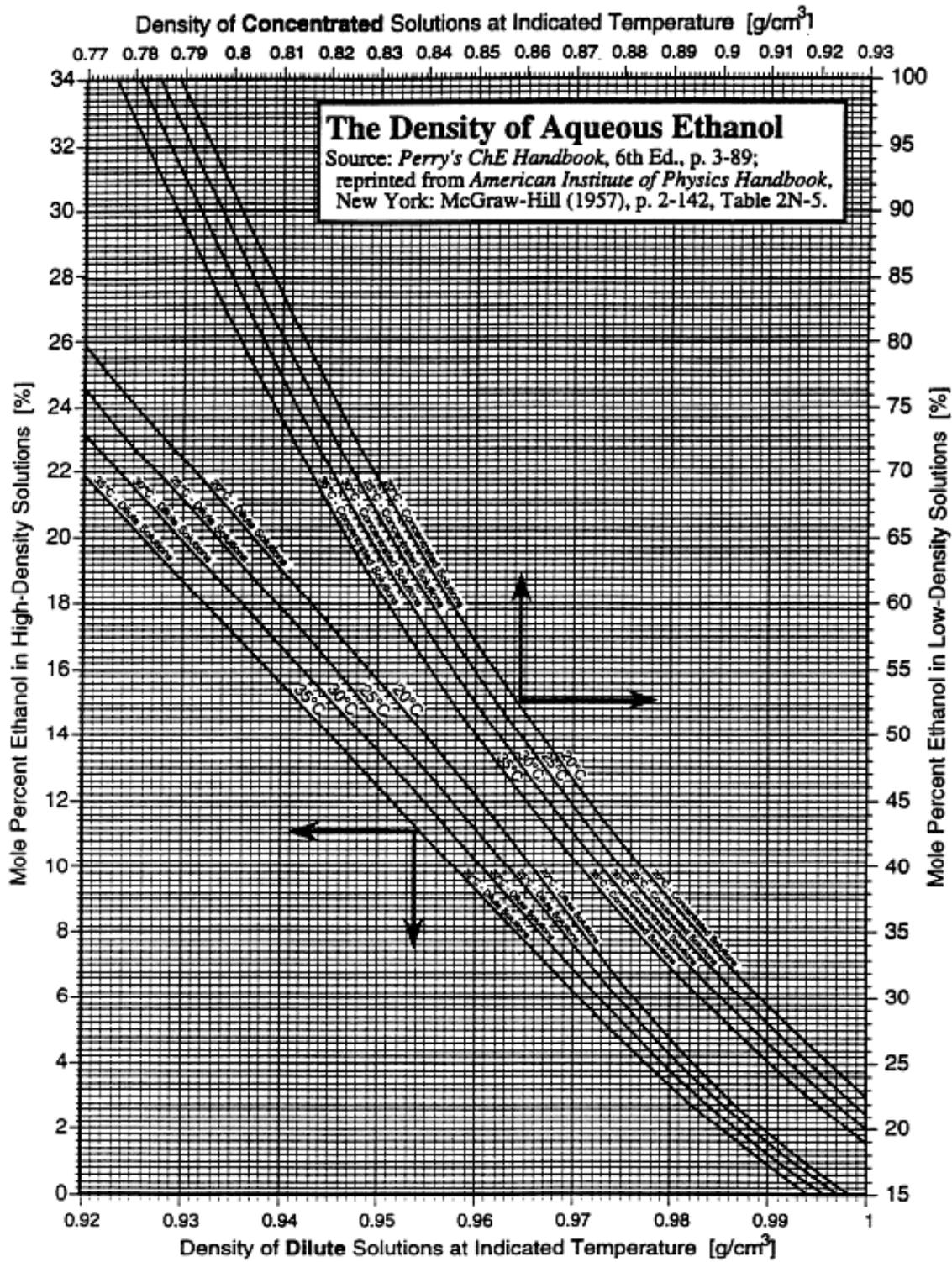


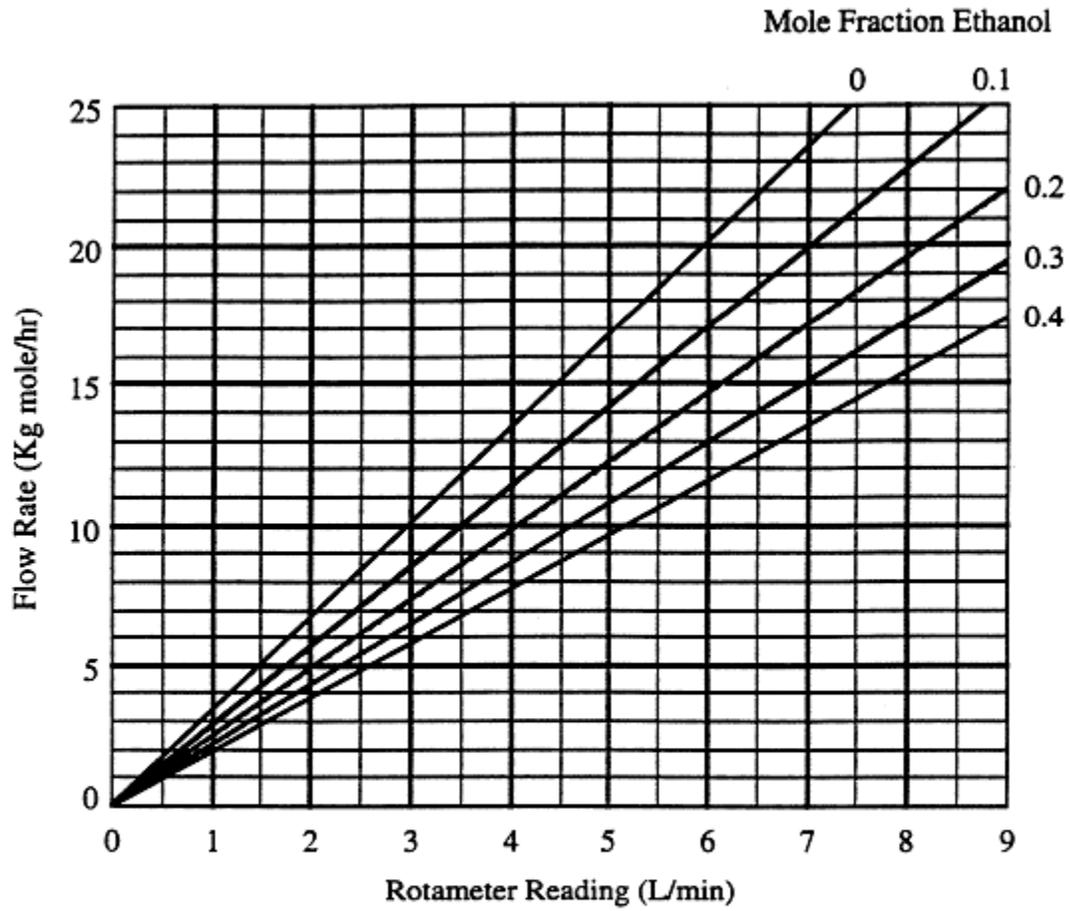
Useful Data for Ethanol/Water Mixtures

RCR - 1993

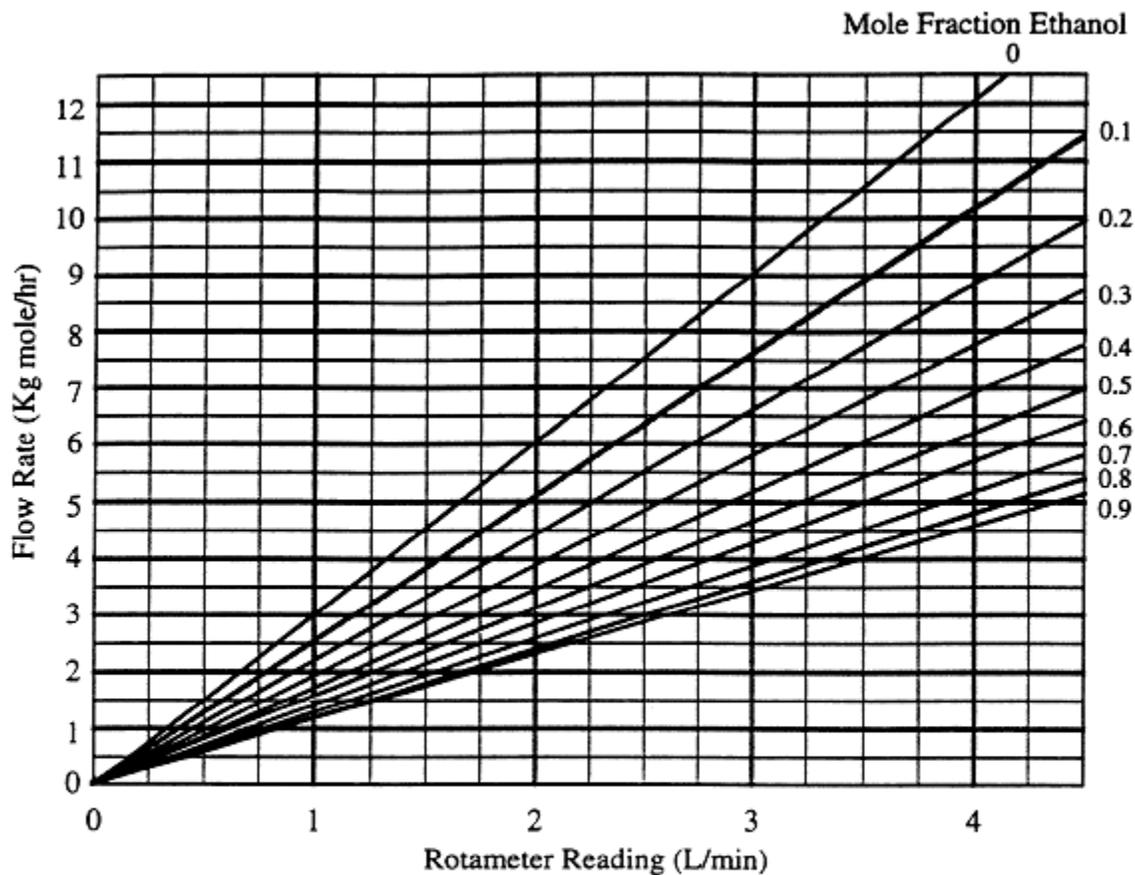
$$MW_{\text{avg}} = 46 x_{\text{EtOH}} + 18 x_{\text{H}_2\text{O}} = 18 + 28 x_{\text{EtOH}} \text{ g/g-mole}$$

relating weight and mole %:  $w_{\text{EtOH}} = 46 x_{\text{EtOH}} / (46 x_{\text{EtOH}} + 18 x_{\text{H}_2\text{O}})$



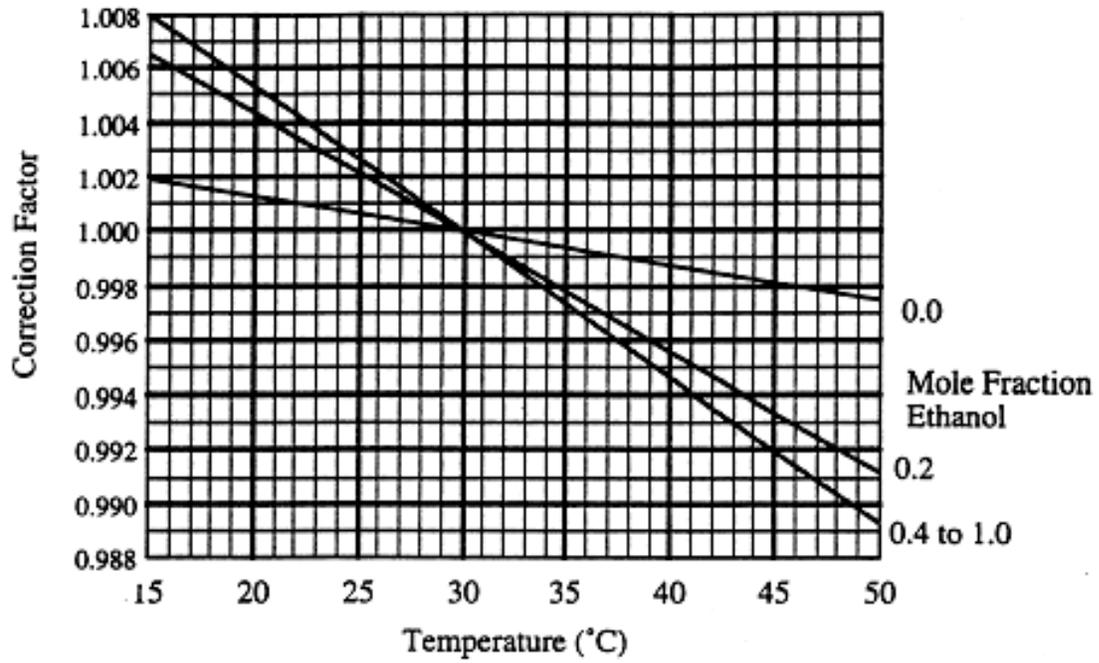


**Rotameter Calibration Curve for Feed Flow**  
(basis: 30°C, S.G. = 1.0)



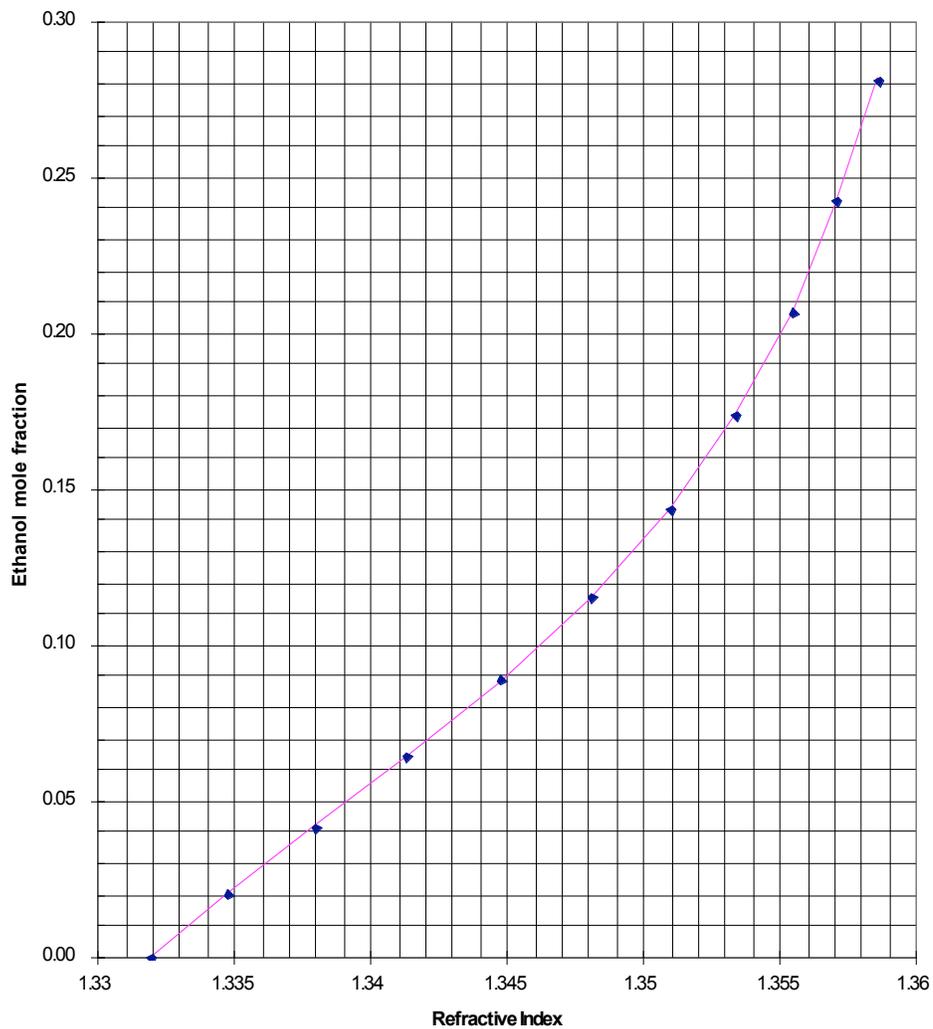
**Rotameter Calibration Curve for Reflux & Top Product Flow**

(basis: 30°C, S.G. = 1.0)



**Rotameter Flow Rate Correction for Temperature**  
(basis: 30°C)

Refractometer calibration at 30 C  
for MOLE FRACTION

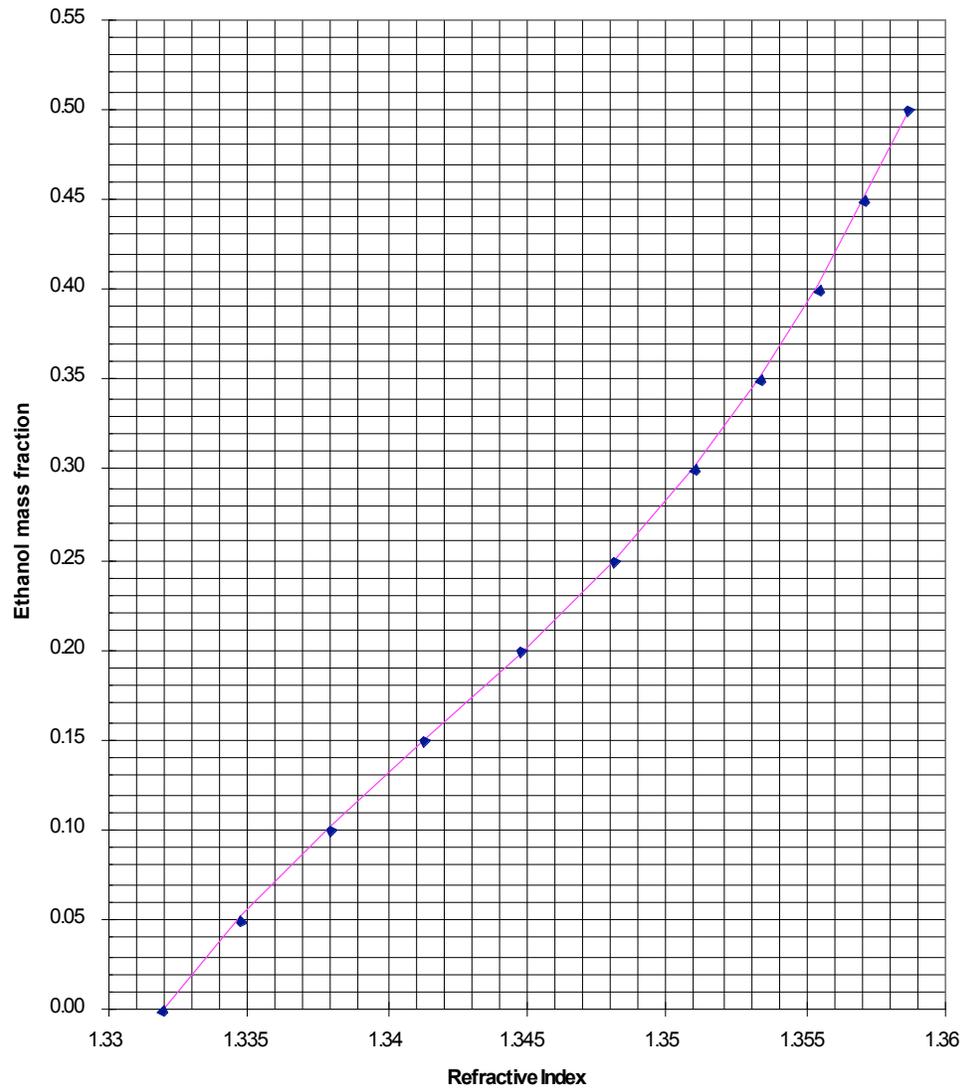


Let  $r = (RI - 1.332) * 100$

Then  $x(\text{EtOH}) = -0.217 + 0.023 * r - 0.022 * r^2 + 0.916 / (4.2 - r)$

This 3rd-order fit gives a maximum error of 0.001 and an average error of 0.0006

**Refractometer calibration at 30 C  
for MASS FRACTION**



Let  $r = (RI - 1.332) * 100$

Then  $m(\text{EtOH}) = 0.0005 + .205 * r - .0694 * r^2 + .0236 * r^3$

This 3rd-order fit gives a maximum error of 0.0025 and an average error of 0.0016





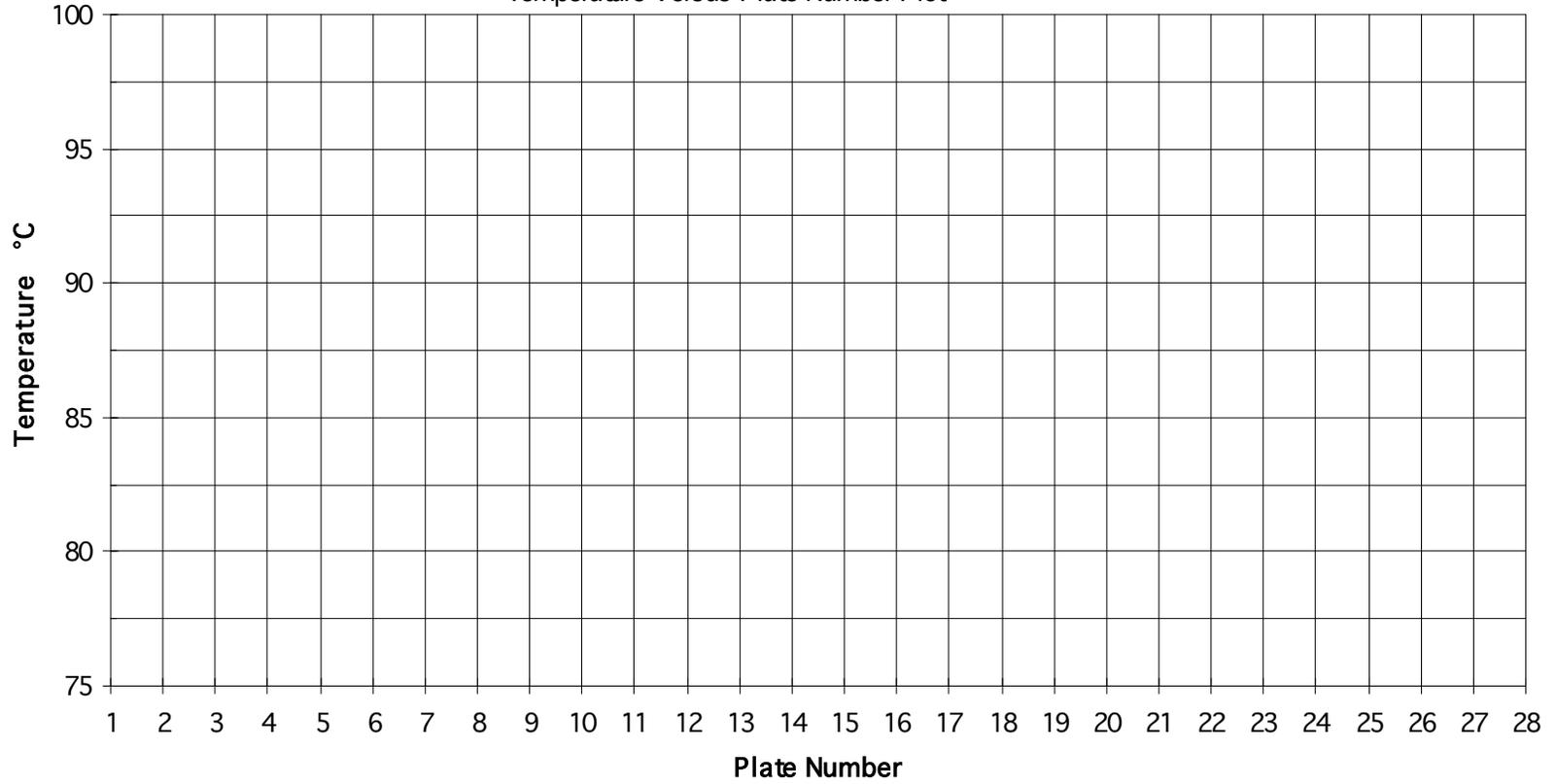
Operating Data 8" Distillation Column  
(Infinite)(Finite) Reflux Ratio

Sheet 3

ChE 220-424-6

Temperature Versus Plate Number Plot

Date\_\_\_\_\_



OPERATING DATA, 8" DISTILLATION COLUMN: SAMPLE COMPOSITIONS  
(Infinite)(Finite) reflux ratio

Sheet 4

Date: \_\_\_\_\_

Exact Time	Sample No.	Source of Sample	Density g/cm <sup>3</sup>	T-°C	y <sub>EtOH</sub>
		Feed			
		Bottoms			
		Top Prod.			
		Tray_____			
		Tray_____			
		Tray_____			

Exact Time	Sample No.	Source of Sample	Density g/cm <sup>3</sup>	T-°C	y <sub>EtOH</sub>
		Feed			
		Bottoms			
		Top Prod.			
		Tray_____			
		Tray_____			
		Tray_____			

## Lecture Notes for Heat Exchanger Formal

Date: \_\_\_\_\_

Instructor: \_\_\_\_\_

---



## Shell-and-Tube Heat Exchanger

### I. Introduction

Heat exchange is a unit operation utilized throughout a broad range of chemical engineering applications. The experimental equipment in this lab provides exposure to several common heat transfer units on a pilot-plant or small production scale, including a 1-2 shell-and-tube heat exchanger, a plate-and-frame heat exchanger, and a steam sparger. The instrumentation provided allows extensive characterization of the thermal behavior of the shell-and-tube exchanger. It is also possible to examine the performance of the other units, if the available time allows it.

### II. Modeling Heat Transfer

Heat exchangers are commonly modeled using an integral form of Newton's Law of Cooling, as

$$Q = UAF\Delta T_{LM} \quad (1)$$

The symbols are defined in the Nomenclature section. For ideal countercurrent flow with constant  $U$ ,  $F = 1$  and the appropriate average temperature driving force is the log-mean temperature difference:

$$\Delta T_{LM} = \frac{(T_{h,in} - T_{c,out}) - (T_{h,out} - T_{c,in})}{\ln\left(\frac{T_{h,in} - T_{c,out}}{T_{h,out} - T_{c,in}}\right)} \quad (2)$$

For the more complex crosscurrent or mixed flow patterns commonly used in industrial equipment,  $F$  factor values below 1.0 quantify the decrease in mean temperature driving force and thus the relative decrease in heat transfer. For a more complete description of this factor and how it varies with flow pattern and stream conditions a number of standard references are available<sup>1,2</sup>. For 1-pass/2-pass heat exchangers, the combined cocurrent and countercurrent sections provide effective heat transfer for large temperature differences but produce progressively worse behavior at close temperature approaches.

The derivation of  $F$  for a 1-2 exchanger has been presented by Bowman *et al*<sup>3</sup>. The equation is expressed in terms of a dimensionless temperature ratio  $Z$  and a dimensionless heating effectiveness factor  $\eta_H$ , defined by equations (3) and (4).

$$Z = \frac{T_{h,in} - T_{h,out}}{T_{c,out} - T_{c,in}} \quad (3)$$

$$\eta_H = \frac{T_{c,out} - T_{c,in}}{T_{h,in} - T_{c,in}} \quad (4)$$

$$F = \frac{\frac{\sqrt{Z^2 + 1}}{Z - 1} \log\left(\frac{1 - \eta_H}{1 - \eta_H Z}\right)}{\log\left(\frac{\frac{2}{\eta_H} - 1 - Z + \sqrt{Z^2 + 1}}{\frac{2}{\eta_H} - 1 - Z - \sqrt{Z^2 + 1}}\right)} \quad (5)$$

The overall heat transfer coefficient can be evaluated by considering the individual heat transfer resistances of the tube outside, tube wall, and tube inside. The resistances can be added in series to give an expression for the overall heat transfer coefficient  $U_i$  (based on the tube inside area)

$$\frac{1}{U_i} = \frac{1}{h_i} + \frac{x}{k_w} \frac{A_i}{A_w} + \frac{1}{h_o} \frac{A_i}{A_o} \quad (6)$$

where  $A_w$  is the logarithmic mean of the internal and external areas of the tube. A wide variety of correlations have been reported for estimation of the heat transfer coefficients under specific laminar or turbulent flow conditions. McCabe *et al*<sup>4</sup> describe the use of common correlations for tube-side heat transfer coefficients, such as the Dittus-Boelter, Sieder-Tate, and Colburn equations. For shell-side coefficients, the Donohue<sup>4</sup> and Kern<sup>5</sup> equations are frequently used. Careful reading of Chapter 12 of McCabe *et al*<sup>4</sup> also provides the conditions under which these correlations are applicable, as well as suggestions for treating consequences of changes in temperature and physical properties along the length of the heat exchanger. The Sieder-Tate and Kern correlations, for tube side and shell side, respectively, are shown below.

Sieder-Tate correlation, for fluid flow inside smooth cylindrical pipes:

$$\frac{h_i D_i}{k} = 0.023 \left( \frac{D_i G_i}{\mu} \right)^{0.8} \left( \frac{c_p \dot{i}}{k} \right)^{1/3} \left( \frac{\mu}{\mu} \right)^{0.14} \quad (7)$$

Kern correlation for shell flow over a bank of tubes, with triangular pitch arrangement:

$$\frac{h_o D_e}{k} = 0.36 \left( \frac{D_e G_s}{\mu} \right)^{0.55} \left( \frac{c_p \dot{i}}{k} \right)^{1/3} \left( \frac{\mu}{\mu} \right)^{0.14} \quad (8)$$

where  $G_s$  is the mass velocity based on the cross-flow area at the diameter of the shell, and  $D_e$  is the equivalent diameter, thus

$$G_s = \frac{W}{A_s}$$

If

$D_s$  = Shell inside diameter

$C'$  = tube clearance (distance between tubes)

$L_b$  = Baffle spacing

$P_T$  = Tube pitch ( distance from center to center)

then

$$N_T = D_s/P_T \text{ Number of tubes at the diameter}$$

and

$$A_s = N_T C' L_b$$

$$D_e = \frac{4 \times \text{flow area}}{\text{wetted perimeter}}$$

For a triangular pitch arrangement

$$D_e = \frac{4 \left( \frac{P_T}{2} \times 0.86 P_T - \frac{1}{2} (\pi D_o^2 / 4) \right)}{\pi D_o / 2} \quad (9)$$

The individual film heat transfer coefficients,  $h_i$  and  $h_o$ , may be graphically determined from overall heat transfer coefficient data by the Wilson method. Inspection of the Sieder-Tate correlation above indicates that if the temperature of a liquid in passing through a given heat exchange apparatus does not change markedly, the liquid film heat transfer coefficient is proportional only to  $G_i^{0.8}$ , where  $G_i$  is the mass velocity of the liquid in the tubes of the exchanger. Thus,

$$h_i = a G_i^{0.8} \quad (10)$$

where  $a =$  a constant.

If the conditions on the shell side of the heat exchange apparatus do not change appreciably, the value of  $h_o$  does not change markedly with different tube side flow rates, provided the metal wall temperature change is not too large. As an approximation, then,  $h_o$  may be assumed constant. Also, the term  $(xA_i)/(k_w A_w)$  in equation (6) will be constant. Thus, equation (6) may be written as:

$$\frac{1}{U_i} = \frac{1}{a G_i^{0.8}} + b \quad (11)$$

$$\text{where } b = \frac{x}{k_w} \frac{A_i}{A_w} + \frac{1}{h_o} \frac{A_i}{A_o}$$

If  $1/U_i$  is plotted as ordinate against  $1/G_i^{0.8}$  as abscissa, equation (11) will plot as a straight line, provided the assumptions are correct. The slope of this line will be  $(1/a)$  and the intercept will be equal to "b". From the slope, values of  $h_i$  corresponding to various  $G_i$  values may be found; from the intercept, a single average value of  $h_o$  may be evaluated for the particular conditions prevalent in the shell side. Thus, the individual film resistances may be estimated readily from measurements of the over-all heat transfer coefficient.

A similar analysis can be done for the tube side by keeping the conditions constant inside the tubes and varying the flow rate in the shell side. In this case the equation is

$$\frac{1}{U_i} = \frac{1}{a' G_s^{0.55}} + b' \quad (12)$$

where  $b' = \frac{x}{k_w} \frac{A_i}{A_w} + \frac{1}{h_i}$

### **III. Apparatus**

The flow diagram for the heat exchangers is shown in Figure 1. The shell-and-tube exchanger is supplied with water from two temperature-controlled tanks. The shell side supply is provided by one 2-1/2", 20 gpm centrifugal pump. The tube side supply has two such pumps in order to provide higher velocities and to allow a wider range of tube-flow Reynold's numbers. Resistance thermometers are used to monitor the temperatures in each inlet and exit line. Flow rates are measured with paddlewheel flowmeters in the supply lines, or by using differential pressure transducers across each path through the exchanger. The outer tube wall heat exchange area is 27.3 ft<sup>2</sup> and the inner tube wall area is 20.2 ft<sup>2</sup>, accounting for tubes which have been intentionally blocked off. In order to attain higher flow rates in the tubes, one half of the tubes in each pass have been sealed with plugs. A diagram of the internal geometry of the shell-and-tube exchanger is shown in Figure 2.

The hot tank is heated by steam injection in a sparging coil, with temperature control provided by a proportional controller on the steam supply. The cold tank temperature is maintained by cooling a constant flow side stream against the city water line in a plate-and-frame heat exchanger. A temperature controller sets the city water flow rate as required. The controllers use thermocouples in the supply tanks for input. Note that it may be possible to set operating conditions that cannot be maintained under control; in those cases the supply tank temperatures will not be at the setpoints.

### **IV. Operating Procedure**

See Figure 1 for location of components referenced in this section.

#### **A. Safety Precautions**

1. Identify shutoffs for power, steam, air, and water for emergency shutdown or end of day shutdown. For immediate emergency shutdown, turn off the pumps with the front panel buttons and then turn off the main power switch to the controllers, located on the power supply strip inside the back of the cabinet.
2. The paddlewheel flowmeters (W<sub>1</sub> & W<sub>2</sub>) are fragile and readily broken or de-calibrated. Avoid shocking the flowmeters with sudden water surges or drastic changes in water flow rates. Note that they work best with a straight run of smooth pipe supplying constant velocity flow.
3. The centrifugal pumps (pumps 1, 2, 3, 4) may be damaged by running with air, or while cavitating with restricted water feed. They will not be harmed by running at no flow, so start them with their outlet valves closed and be sure to throttle the pumps with downstream valves only. Note also that this procedure protects the paddlewheel flowmeters.

## B. Startup Procedure

1. Turn on electrical power to the controller systems with the switch located on the power supply strip accessible through the rear door of the cabinet. Provide electrical power to the 4 pumps by lifting the external levers on the circuit breaker boxes in the controller cabinet (2 in front and 2 in rear). DO NOT turn on the pumps at this time with the green power buttons on the front of the control cabinet.
2. In the front of the cabinet use the black and silver Omega<sup>®</sup> CN2000 Programmable Controller to adjust the hot tank temperature set-point a few degrees below the current tank temperature shown on the controller display. The arrow-up ( ↑ ) and arrow-down ( ↓ ) keys on the panel are used to adjust the set-point temperatures. The controller signal operating the steam valve should now be at a level that maintains the steam valve in the closed position. Because the compressed air supply to the valve has not been turned on yet, the valve will not respond to the controller. *[The above steps assume that the system is in the "automatic control" mode, i.e. the LED indicator symbols TUN and MAN are not lit up on the panel. If TUN is lit up, keep pushing the PARAM key until END OF TUNE appears, then push the RETURN key. If the MAN indicator is on, push the AUTO key.]*
3. Enter the TUNE mode by pushing the key sequence TUNE-LAST-YES on the controller key pad. The TUN indicator will illuminate and the sequence of prompts can be followed to verify/enter the control parameters and return to the automatic control mode (the PARAM/DISPLAY key advances the displays in the control loop while the LAST key will cause the display to back up).

## Tuning Sequence:

ARROW KEYS ON	⇒	Push PARAM/DISPLAY key to proceed
TUNE? OUTPUTS	⇒	Select YES
SELF TUNE?	⇒	Select NO
PR BAND 1 1 %	⇒	Enter 1% using ↑ and ↓ keys then push PARAM/DISPLAY key
RESET 2.70 R/M	⇒	Enter 2.70 using ↑ and ↓ keys then push PARAM/DISPLAY key
RATE 1 0.05 M	⇒	Enter 0.05 using ↑ and ↓ keys then push PARAM/DISPLAY key
END OF TUNE	⇒	Select RETURN

If the MAN indicator is illuminated at this point, push the AUTO/MANUAL key to enter the automatic mode.

Heat Exchanger Experiment, p.HX-8

4. Turn on the system compressed air supply valve (not on diagram). Verify that the overhead building air supply valve is also open.
5. While one person observes the steam control valve,  $V_6$ , another person should increase the set-point on the CN200 controller to a few degrees above the tank temperature using the  $\uparrow$  key. This should cause the valve indicator gauge to move up, verifying that the controller is not in the **TUN** or **MAN** mode, and to verify that there is air pressure supplied to the system. Make sure to return the setpoint temperature a few degrees below the tank temperature before proceeding.
6. The gray and blue CN3000 controller actuates the plate-and-frame cold water supply valve,  $V_5$ . While one person observes valve  $V_5$ , another person should change the setpoint a few degrees above, then a few degrees below the setpoint. The  $\uparrow$  **Y** and  $\downarrow$  **N** keys are used to adjust the setpoint. The control valve should open and close with each setpoint change. Make sure to leave the setpoint temperature a few degrees above the cold tank temperature before proceeding.

Skip the next step (7) if the hot and cold supply tanks are already  $\sim 2/3$  full of water.

7. If the hot and cold supply tanks are not filled with water at this time, place the end of the flexible drain hose from the plate-and-frame heat exchanger into the tank to be filled. Turn on the city water supply valve (labeled on vertical pipe from ceiling) and then set the cold water controller setpoint a few degrees below the cold tank temperature. The control valve,  $V_5$ , will open and allow city water to flow through the hose. Fill to a level about  $2/3$  full. Make sure to hold or secure the flexible hose in place during this step to prevent water spills.
8. Open the steam supply valve (not shown on diagram). The pressure gauge should read approximately 50 - 60 psig. Check the overhead steam supply valve to make sure that it is fully open.
9. The flexible exit hose from the plate and frame heat exchanger should be placed securely in the floor drain. If not opened yet, the city water supply valve should be opened at this time. This vertical pipe drops down from the ceiling and is labeled.
10. Check to be sure that valves  $V_1$ ,  $V_2$ ,  $V_3$ , and  $V_7$  are closed.
11. On the hot water tank controller (CN2000), adjust the setpoint up to the desired operating temperature. Then turn on pump #3 with the green button on the front of the control panel and **slowly** open valve  $V_3$  to begin recirculating the hot tank water through the exchanger at  $\sim 10$  gpm.
12. When the hot tank temperature approaches the setpoint, turn on pumps 1, 2, & 4, and fully open valves  $V_2$  and  $V_7$ . **Slowly** open valve  $V_1$  to adjust the flow rate through the tube side of the shell-and-tube exchanger. Use the cold tank temperature controller to select the desired cold tank setpoint.

13. The stream temperatures can be recorded from the front panel meter as follows:

- T1 = Hot side inlet of shell-and-tube exchanger
- T2 = Hot side outlet of shell-and-tube exchanger
- T3 = Cold side inlet of shell-and-tube exchanger
- T4 = Cold side outlet of shell-and-tube exchanger
  
- T6 = Plate-and-frame exchanger (hot side) inlet from cold supply tank
- T9 = Plate-and-frame exchanger (hot side) outlet to cold supply tank
- T8 = City water (cold side) inlet to plate-and-frame exchanger
- T7 = City water (cold side) outlet of plate-and-frame exchanger

14. Reverse the start-up sequence to shut the system down.

## **V. Experimental Outline**

The objective for this experiment is to characterize the following performance parameters of the heat exchanger:

- Experiment 1 - the  $F$  factor, as defined in equation (5).
- Experiment 2 - the shell-side coefficient,  $h_o$ , as a function of flow rate, and the tube-side coefficient,  $h_i$ , as a function of flow rate.

### Experiment 1

Set the cold stream to hot stream flow rate ratio to 1:2, or other value as indicated by the instructor (Ratio \_\_\_\_\_). Keeping the ratio fixed, change the inlet temperature of the cold fluid over the range allowed by the apparatus. Calculate the  $F$ -factor and effectiveness for each temperature.

### Experiment 2

Set the hot stream (shell side) to an inlet temperature of 120 °F, or another temperature as indicated by the instructor ( $T_{h,in}$  \_\_\_\_\_ °F). Set the cold stream (tube side) to an inlet temperature of 90 °F, or another value as indicated by the instructor ( $T_{c,in}$  \_\_\_\_\_ °F). Set the hot stream flow rate to 8 gpm. Record all relevant process variables. Change the flow rate of the cold stream from 8 gpm through the maximum obtainable from the equipment. Set at least 6 different values.

Change the shell stream flow rate and repeat the experiment for the same 6 cold stream flow rates used before. Repeat the procedure to complete 6 different hot stream flow rates.

Calculate the overall heat transfer coefficient,  $U_i$ , for each of the 36 experimental points.

## **VI. Data Analysis**

- A. Calculate the  $F$ -factor for each of the points obtained in Experiment 1. Make a plot of the  $F$ -factor versus the effectiveness factor with  $Z$  as a parameter. Compare with the theoretical graph.
- B. Compare the overall heat transfer coefficient for the 36 experimental points with the predictions obtained using Sieder-Tate for the tube side and Kern's correlation for the shell side.
- C. For each shell side flow rate, make a Wilson plot of  $1/U_i$  vs.  $1/G_i^{0.8}$  and find an estimate for the heat transfer coefficient for the shell side,  $h_o$ . Plot the six estimates of  $h_o$  so obtained versus  $G_s$  in log-log coordinates to obtain the relationship  $h_o = aG_s^n$ . Compare with the value of  $n = 0.55$  used in Kern's correlation.

For each tube side flow rate make a Wilson plot of  $1/U_i$  vs.  $1/G_s^n$ , using the  $n$  value obtained from the analysis described in the previous paragraph, and find an estimate of the heat transfer coefficient for the tube side,  $h_i$ . Plot the six estimates of  $h_i$  obtained versus  $G_i$  in log-log coordinates to obtain the relationship  $h_i = aG_i^m$ . Compare with the expected value of  $m = 0.8$  derived from the Sieder-Tate correlation.

## **VII. Report Requirements**

The summary of results section should include the  $F$ -factor and effectiveness results for the apparatus. The overall heat transfer coefficients obtained experimentally and those predicted using the Sieder-Tate and Kern correlations should also be presented.

In the discussion section, the following issues should be addressed:

- Capacity of the heat exchanger apparatus to operate at high or low effectiveness and  $F$ -factor values.
- Suitability of the Sieder-Tate and Kern correlations to predict the overall heat transfer coefficient.
- Comparison of the predictions of the Sieder-Tate correlation for  $h_i$  with the estimates obtained using Wilson's method. Include Wilson graphs ( $1/U_i$  vs  $1/G_s^{0.55}$ ), preferably a single graph with multiple lines. Include also the corresponding log-log plot of  $h_i$  vs.  $G_i$ . ( $h_i$  estimates are obtained from the intercept of  $1/U_i$  vs  $1/G_s^{0.55}$  plots.)
- Comparison the predictions of the Kern correlation for  $h_o$  with the estimates obtained using Wilson's method. Include Wilson graphs ( $1/U_i$  vs  $1/G_i^{0.8}$ ), preferably a single graph with multiple lines. Include also the corresponding log-log plot of  $h_o$  vs  $G_s$ . ( $h_o$  estimates are obtained from the intercept of  $1/U_i$  vs  $1/G_i^{0.8}$  plots.)
- Discuss the energy balance closure and the sources of error.

**VIII. Nomenclature**

$a$	Constant in Eq. (11)
$a'$	Constant in Eq. (12)
$b$	Constant in Eq. (11)
$b'$	Constant in Eq. (12)
$A$	Area, ft <sup>2</sup>
$C'$	Distance between tubes, ft
$c_p$	Specific heat at constant pressure, Btu/lb-°F
$D$	Diameter, ft
$D_e$	Equivalent diameter defined by Eq. (9), ft
$D_s$	Shell diameter, ft
$F$	Log-mean temperature correction factor
$G$	Mass velocity, lb/ft <sup>2</sup> -h
$G_s$	Mass velocity based on shell cross-flow area, lb/ft <sup>2</sup> -h
$h$	Individual heat transfer coefficient, Btu/ft <sup>2</sup> -h-°F
$k$	Thermal conductivity, Btu/ft-h-°F
$L$	Length of exchanger, ft
$L_b$	Baffle spacing, ft
$N_T$	Number of tubes at the shell diameter
$P_T$	Tube pitch, center to center, ft
$Q$	Total heat flow rate in exchanger, Btu/h
$T$	Temperature, °F
$U$	Overall heat transfer coefficient, Btu/ft <sup>2</sup> -h-°F
$W$	Mass flow rate, lb/h
$x$	Tube wall thickness, ft
$Z$	Dimensionless temperature ratio defined in Eq. (3)

Greek letters

$\Delta T$	Temperature difference, °F
$\eta_H$	Heating effectiveness factor defined in Eq (4), dimensionless
$\mu$	Viscosity, lb/ft-s
$\rho$	Density, lb/ft <sup>3</sup>

Subscripts

$a$	
$c,in$	At cold water inlet
$c,out$	At cold water outlet
$h,in$	At hot water inlet
$h,out$	At hot water outlet
$i$	Inside tube
$LM$	Log-mean
$o$	Outside tube
$s$	Shell side
$w$	Tube wall

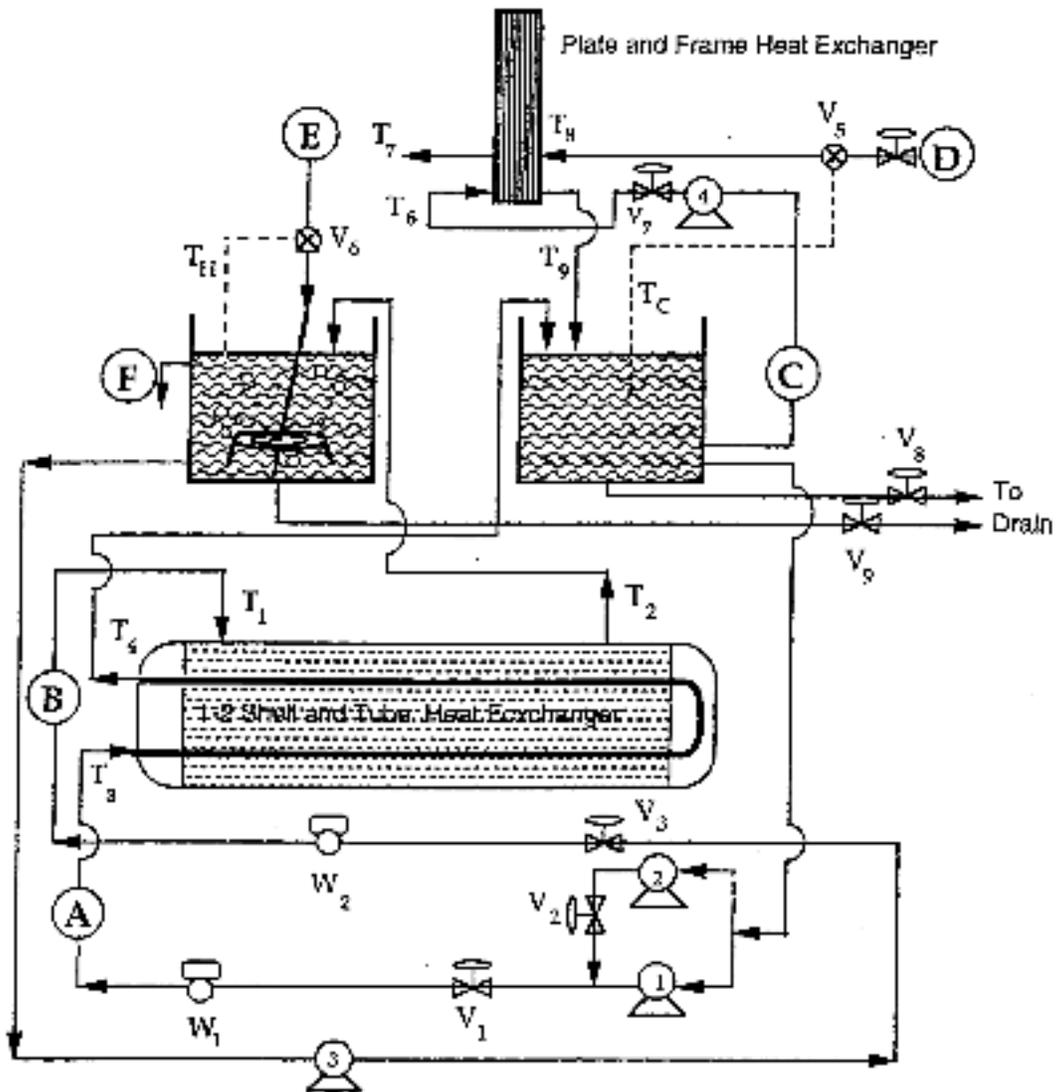
## **IX. References**

1. McCabe, W.L., Smith, J.C., and Harriott, P., "Unit Operations of Chemical Engineering," 4th ed., McGraw-Hill, N.Y., 1985, pp. 389-391.
2. Perry, R.H., Green, D.W., and Maloney, J.O., "Perry's Chemical Engineer's Handbook," 6th ed., McGraw-Hill, New York, 1984, page 10-25.
3. Bowman, R.A., Mueller, A.C., and Nagle, W.M., Mean Temperature Difference in Design, *Trans. of ASME*, May 1940, pp. 283-293.
4. McCabe, W.L., Smith, J.C., and Harriott, P., *op. cit.*, Chapter 12.
5. Hewitt, G.F., Shires, G.L., and Bott, T.R., "Process Heat Transfer", CRC Press, Boca Raton, 1994. p. 271.

## **X. Appendix**

### Shell-and-Tube Heat Exchanger Specifications

$D_s$ Internal Shell diameter	8 "
$L_b$ Distance between baffles	4 7/8" (both ends) 3" (remaining 11)
Number of tubes (external)	78 each pass
Number of tubes (open for flow)	39 each pass
$L_T$ Tube length (each pass)	42 3/4"
$D_o$ Tube external diameter	0.375"
$D_i$ Tube Internal diameter	0.277"
Pitch arrangement	Triangular
$P_T$ (Center to center distance)	0.529"
$C'$ (Clearance between tubes)	0.154"
Thermal conductivity of tubes	62 BTU/ft-h-°F



- $T_H$  = Hot side temperature and controlled variable
- $T_C$  = Cold side temperature and controlled variable
- $T$  = Stream Temperature
- $W$  = Stream flow rate, Meter symbol => 
- $V$  = Valve, Pneumatic control valve symbol =>   
Manual globe or ball valve symbol => 
-  = Pump
-  = Stream designation

Figure 1. Heat exchanger system layout

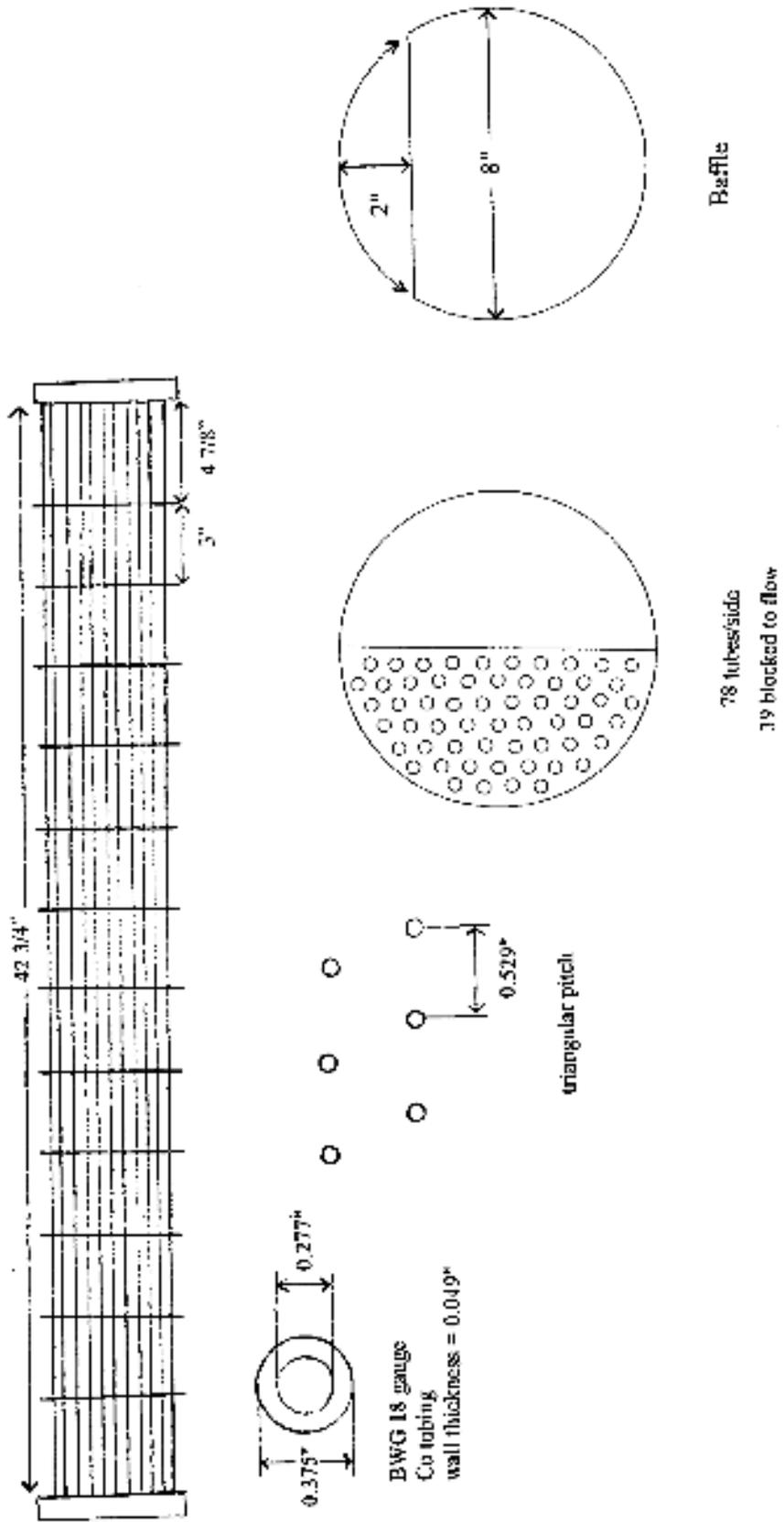


Figure 2. Internal dimensions of the shell-and-tube heat exchanger

## Lecture Notes for Humidification Formal

Date: \_\_\_\_\_

Instructor: \_\_\_\_\_

---



## Adiabatic Humidification - Water Cooling - Dehumidification

### I. OBJECTIVE

The primary objective of this experiment is to determine mass- and heat-transfer coefficients for a spray tower in which water and air are brought into direct contact. Three types of operation – adiabatic humidification, water cooling, dehumidification – will be studied, with chief attention being paid to adiabatic humidification. With this latter type of operation, the effects of air rate and of water rate on mass and heat transfer coefficients will be studied. Energy balances will be determined for the tower operating under each of the three types of operation.

### II. THEORETICAL BACKGROUND

#### II.1 General

The general mechanism of the interaction of air and water is discussed in any of the standard unit-operations textbooks [1,2,3].

- *Phase relations and definitions*

*Absolute humidity:*

$$Y = \frac{w_A}{w_B} = \frac{\text{mass of water}}{\text{mass of dry air}}$$

*Dry-bulb temperature, T.* Temperature of the water vapor-air mixture as determined by immersion of a thermometer in the mixture.

*Percentage saturation* or percentage absolute humidity,  $\phi$  :

$$\phi = \frac{Y}{Y_s} \times 100\%$$

*Humid heat,  $c_s$ .* The humid heat is the heat required to raise the temperature of a unit mass of air and its accompanying water vapor one degree at constant pressure.

$$c_s = c_B + Y c_A; \quad \text{for the air - water system} \quad c_s = 0.24 + 0.446 Y \frac{\text{BTU}}{\text{lb F}} \quad \text{or} \quad \frac{\text{cal}}{\text{gm C}};$$

$$c_s = 1 + 1.86 Y \frac{\text{kJ}}{\text{kg K}}$$

*Enthalpy, H.* The enthalpy of an air-water mixture, per unit mass of dry air, is given by:

$$H = c_s(T - T_0) + Y\lambda_0$$

*Adiabatic saturation temperature,  $T_s$ .* The adiabatic saturation temperature is the temperature reached when air is contacted with an amount of water at a temperature such that each phase experiences adiabatic conditions.

These various properties of the air-water system are commonly presented in a psychrometric chart, such as that shown in Figure 1. Values are also tabulated in Perry [4].

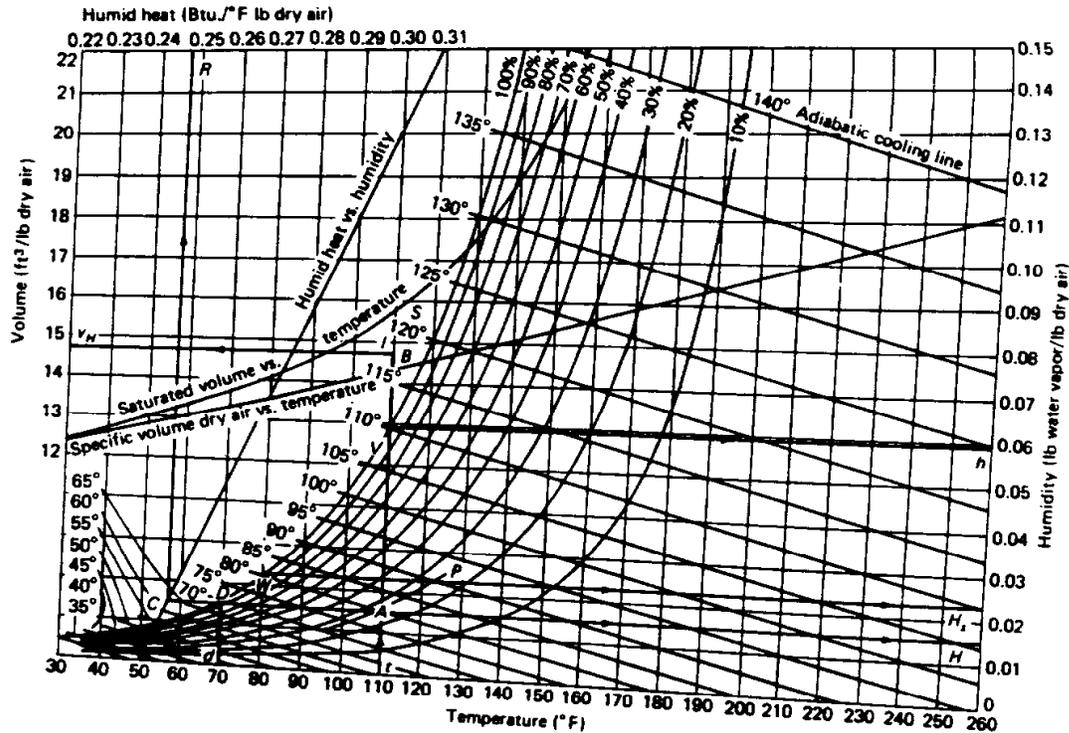


Fig. 1. The air-water system: Psychrometric chart

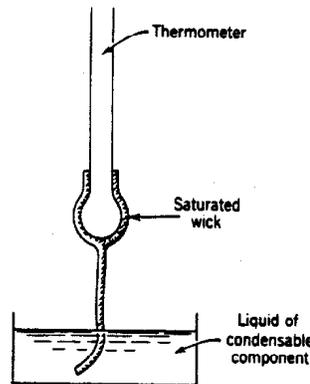
*Wet-bulb temperature.* The wet-bulb temperature,  $T_w$ , is the steady-state temperature reached by a small amount of water evaporating into a large amount of unsaturated water vapor - air mixture.

$$T - T_w = \frac{k_Y \lambda_w}{h_G} (Y_w - Y)$$

Psychrometric ratio  $\frac{h_G}{k_Y}$

Lewis relation  $\frac{h_G}{k_Y c_s} = \left( \frac{Sc}{Pr} \right)^{0.567} \approx Le^{1/3}$

For air - water mixtures:  
 $Le \approx O[1]$



Wet-bulb thermometer

**II.2. Adiabatic humidification**

The term *adiabatic humidification* is used in a special sense to designate the interaction process between air and water vapor when the water spray is admitted to the system at the wet-bulb temperature of the incoming air. Though all three of the processes investigated in the experimental program outlined above are carried out under substantially adiabatic condition for the tower as a whole (well insulated tower, with small heat losses by conduction through the walls), only one of them is designated as *adiabatic humidification*. In this special case, which is the only one easy to calculate, we have adiabatic conditions for the *local* action at the air-water interface anywhere in the tower, as well as for the tower as a whole. No sensible heat is transferred between the interface and the bulk of the water. All the latent heat of evaporation of the water comes into the interface as sensible heat from the air stream.

The theory of adiabatic humidification will be developed with the aid of the diagram shown in Figure 2. In developing the theory, it will be assumed that the ideal condition is achieved, where  $(T_L)_{top} = (T_L)_{btm} = (T_w)_{top} = (T_w)_{btm}$ . In actual operation, of course, this ideal condition is not exactly realized, and the actual values for these four temperatures should be used at the appropriate place in the formulas to be developed.

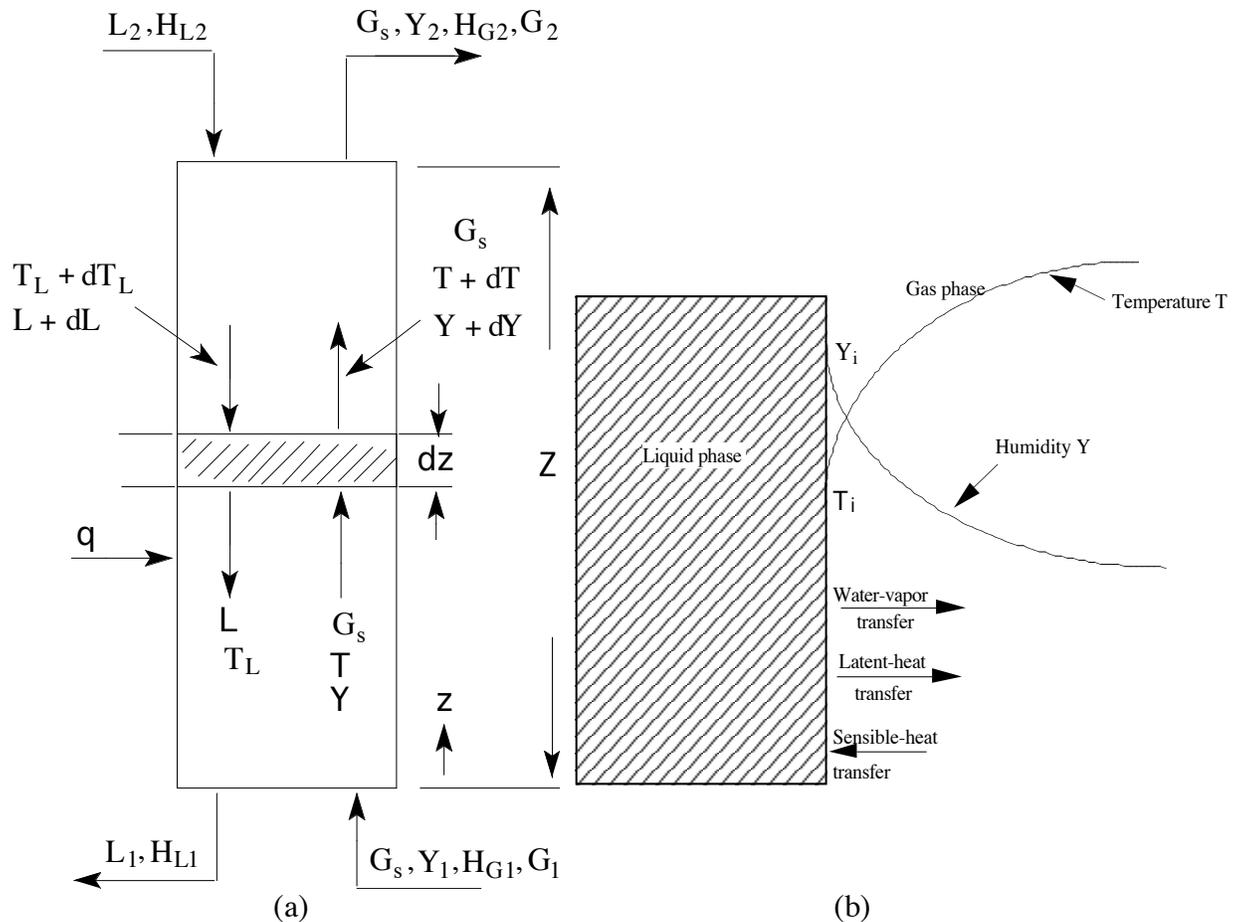


Figure 2. Adiabatic humidification process: (a) Nomenclature and process diagram, (b) Temperature and humidity profiles for the humidification experiment.

It should be understood that in adiabatic humidification, there is only *one* film, a gas film. There is no liquid film, either from the standpoint of heat transfer or from the standpoint of mass transfer. From the standpoint of heat transfer, this is true because there is no temperature difference between the water at the interface, and the main mass of water. From the standpoint of mass transfer, this is true because the liquid phase is a simple one-component system, with no dissolved material that is being absorbed from the gas phase.

In the increment of tower volume  $Adz$ , the humidity of the air changes by  $dY$  (an increase), and the total quantity of water vapor diffusing through the gas film is  $G_s dY$ . The total quantity of water vapor diffusing through the gas film can also be indicated as  $(K_Y a_M)(Y^* - Y)Adz$ , where  $Y$  represents bulk gas humidity and  $Y^*$  represents the saturated humidity corresponding to the temperature of the water (constant throughout the tower).

Assuming low mass-transfer rates and fairly low temperature level, one may express the equations for mass and heat transfer in the tower as [3]

For mass transfer:

$$G_s dY = (K_Y a_M)(Y^* - Y)Adz \quad (1)$$

$$Z = \left[ \frac{G_s / A}{K_Y a_M} \right] \int_{Y_1}^{Y_2} \frac{dY}{Y^* - Y} = \left[ \frac{G_s / A}{K_Y a_M} \right] \frac{Y_2 - Y_1}{\Delta Y_{\ln}} \quad (1a)$$

where

$$\Delta Y_{\ln} = \frac{(\Delta Y)_{\text{btm}} - (\Delta Y)_{\text{top}}}{\ln \frac{(\Delta Y)_{\text{btm}}}{(\Delta Y)_{\text{top}}}}; \quad \Delta Y = (Y^* - Y) \quad (2)$$

For heat transfer

$$G_s c_s dT_G = h_G a_H (T_i - T_G) Adz \quad (3)$$

$$Z = \left[ \frac{G_s c_s / A}{h_G a_H} \right] \int_{T_1}^{T_2} \frac{dT}{T_i - T} = \left[ \frac{G_s c_s / A}{h_G a_H} \right] \frac{T_1 - T_2}{\Delta T_{\ln}} \quad (3a)$$

where

$$\Delta T_{\ln} = \frac{(\Delta T)_{\text{btm}} - (\Delta T)_{\text{top}}}{\ln \frac{(\Delta T)_{\text{btm}}}{(\Delta T)_{\text{top}}}}; \quad \Delta T = (T_i - T_G) \quad (4)$$

### II.3. Water cooling

Water cooling with air is one of the most important unit operations. Water, warmed by passage through heat exchangers, condensers, or other process equipment, is cooled

by contact with atmospheric air for reuse in the process. The latent heat of water is so large that only a small amount of evaporation produces large cooling effects. Therefore, the water flow rate  $L$  in the cooling tower may be regarded as a constant.

The local temperature and humidity profiles for the water cooling process in the upper and lower sections of the tower are shown in Figure 3.

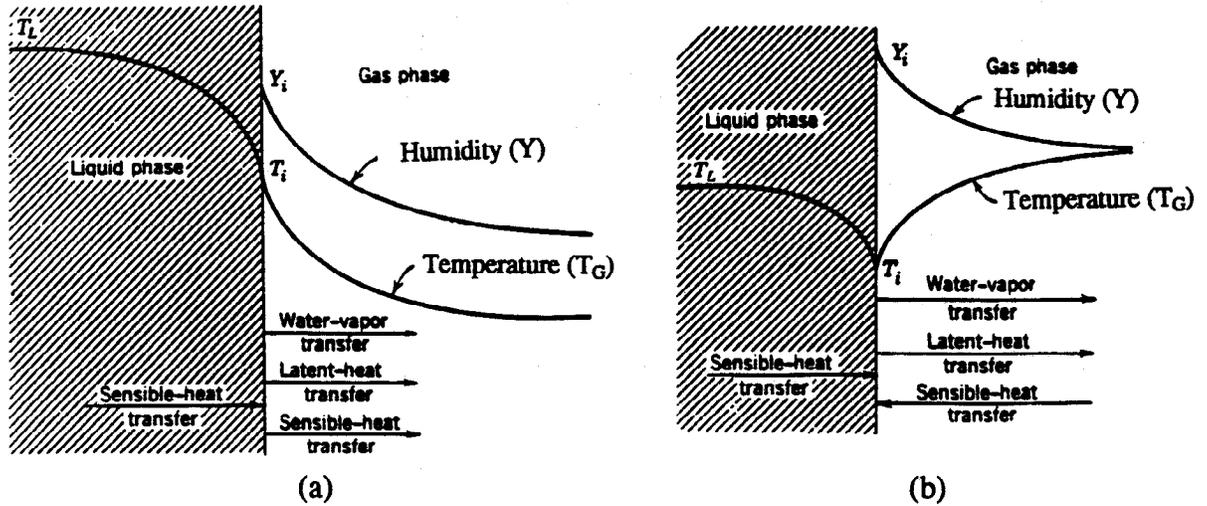


Figure 3. Water cooling process: (a) upper section of the tower, (b) lower section of the tower.

Assuming that the mass transfer rates are usually small, the temperature level is generally fairly low, and the Lewis relation applies reasonably well for the air-water system, one may derive the equations for mass transfer in the tower in terms of gas enthalpy  $H$  as

$$G_s dH = L c_L dT_L \quad (5)$$

and

$$G_s dH = K_Y a_M (H^* - H) Adz \quad (6)$$

Integration yields an operating line equation

$$G_s (H_2 - H_1) = L c_L (T_{L2} - T_{L1}) \quad (7)$$

and a rate equation

$$Z = \left[ \frac{G_s / A}{K_Y a_M} \right] \int_{H_1}^{H_2} \frac{dH}{H^* - H} = [\text{HTU}] [\text{NTU}] \quad (8)$$

The water-cooling tower industry frequently uses Equation 8 in another form which results from combining Equations 5 and 6 assuming  $c_L = 1$  for water:

$$\frac{K_Y a_M Z}{L/A} = \int_{T_{L1}}^{T_{L2}} \frac{dT_L}{H^* - H} \quad (9)$$

The left hand side of Equation 9 is known as the “tower characteristic”. A plot of  $K_Y a_M Z / (L/A)$  or  $K_Y a_M Z / (G_s / A)$  vs. the  $L/G_s$  ratio, with  $G_s$  as the varied parameter is

useful for practical purposes because it determines the position of the operating line relative to the equilibrium curve on a plot of H versus  $T_L$ .

The interfacial conditions can be determined by combining the enthalpy balance  $G_s dH_G = d(LH_L)$  with the heat transfer rate and the liquid transfer rate and assuming  $a_H = a_M$ :

$$-\frac{h_L}{k_Y} = \frac{H_G - H_i}{T_L - T_i}$$

The graphical calculation is indicated in Figure 4, which demonstrates the equilibrium curve and the countercurrent operating line. In practice, one assumes that  $h_L$  is large so that  $T_i = T_L$ .

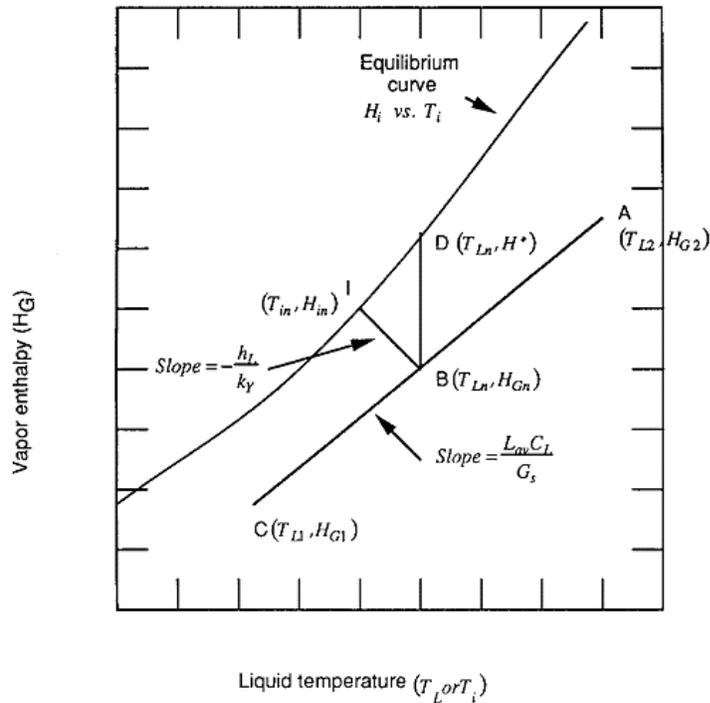


Figure 4. Operating diagram for an adiabatic water-cooling tower

#### II.4. Dehumidification

Dehumidification of air using water may seem counterintuitive, but it is equivalent to a direct contact heat exchange between warm, humid air, and cold water. The vapor condenses, carrying latent heat with it and sensible heat is also transferred from the gas to the liquid. The humidity and temperature profiles for this process are shown in Figure 5. The equations that describe the process are identical to those of water cooling, as shown in Equations 5-9, above. The operating diagram is similar to that shown above in Figure 4, except that the operating line will appear above the equilibrium line.

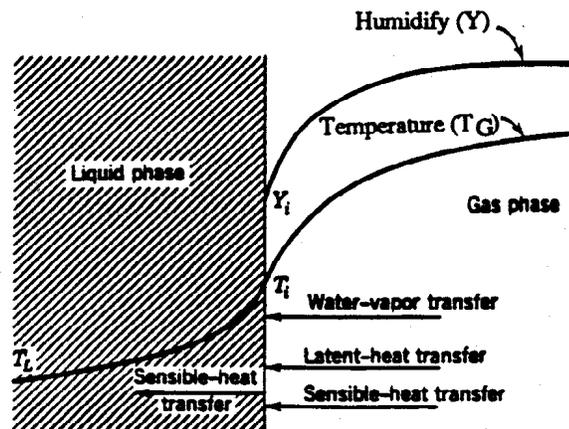


Figure 5. Dehumidification process

### III. EXPERIMENTAL

#### III.1. Equipment

A block diagram of the apparatus is shown in Figure 6 on p. Hu-19.

##### A. Tower

The spray tower consists of a steel cylinder, four feet in height and sixteen inches internal diameter. The bottom is a half-inch steel plate bolted to a flange on the main cylinder. The bottom is tapped for the air inlet and water outlet pipes. A crown bolted on top of the tower is equipped with a spray nozzle and viewing windows.

##### B. Air Supply

Air for this experiment is supplied by a U.S. Hoffman centrifugal blower with a rated capacity of 350 lbs. air per hour at 3 psig.

A one-inch, sharp-edged orifice mounted between flanges in a six-inch pipe is used to meter the air flow. The flow rate through this orifice is given by the equation:

$$G_S = 248 M \sqrt{\frac{P_o \cdot \Delta h}{T + 460}}$$

where	$G_S$	= mass rate of air flow (lbs dry air/h)
$P_o$	=	absolute pressure of the air (in of Hg) = barometric pressure + downstream static pressure
$\Delta h$	=	pressure drop across orifice (in. of H <sub>2</sub> O under air)
$T$	=	air temperature at orifice (°F)
$M$	=	moisture factor = 1.0 for flow rates less than 500 lbs/hr and dew points less than 100°F (dimensionless)

The discharge pipe from the centrifugal blower is provided with copper cooling coils to reduce the temperature of the air metered through the orifice. In operating the equipment, the cooling water to these coils is usually kept turned on.

In examining the flow diagram, observe that a steam-heat air heater is provided that makes it possible to control the dry-bulb temperature of the air supplied to the tower. Provision is also made for introducing steam into the air supply line. This is done only in the experiment on dehumidification where the inlet air should have a high humidity as it enters the tower.

##### C. Water Supply

The temperature of the water introduced into the tower for the adiabatic humidification and water cooling operations is automatically adjusted by a temperature controller connected to heating tape wrapped around the water inlet pipe to the tower. For the dehumidification operation, the tank is filled only with cold water. The rate of water leaving the tower is measured by determining the time required to collect a

convenient quantity: ca. 10-20 lbs. The water rate may be set at the desired value with the valve on the rotameter, the readings of which can be correlated to gallons per minute (gpm).

#### *D. Humidity Measurement*

Relative humidity/temperature sensors are provided at the inlet and outlet of the tower and may be monitored by using the switches and digital displays located at the base of the tower. Wet and dry-bulb thermometers are also supplied, which, when used in conjunction with a humidity chart, provide the humidity of the inlet and exit air. The humidity of the ambient air can be measured with a motor-powered psychrometer.

### ***III.2. Experimental procedure***

#### *A. Preliminary Inspection of Equipment*

It is expected that each student understands the arrangement and operation of the equipment before any experimental work is undertaken. Therefore, a complete inspection of the equipment should be made. This will be facilitated by reference to the flow diagram given in Figure 6. The valves on the equipment are tagged according to the numbering scheme shown on the flow diagram.

#### *B. General Precautions*

1. Water should not be admitted to the tower unless air is flowing; otherwise the tower may become excessively chilled or humidified, delaying attainment of steady state.
2. The air blower should not be started unless valves A1 and A2 are both closed. The slide valve at the blower suction, A7, is set so that air flow will not exceed an orifice differential of 35 inches of water. This valve should be kept at this setting at all times. Valves A1 and A2 should be used to control air flow rate.

#### *C. Preliminary Work*

1. Turn on power to the blower and to the indicator panel. DO NOT start the blower or turn on the "PUMP & HEATER" switch. The blower power throw switch is located on the manometer panel but on the opposite side from the manometers. The indicator panel power throw switch is located on the backside of the indicator panel.
2. Fill the wet bulb wells at the bottom and top of the tower with distilled water. To fill, tightly hold the metal tubing near the reservoir and pull the reservoir straight up. Replace the filled reservoir body by pushing down.
3. Drain the tower and air line (Valves W8 and W10). Be sure that these valves are closed before the tower is placed in actual operation.

Humidification Experiment, p. Hu-12

4. Open the main steam valves S1 and S2 - drain off the condensate by opening S6 until condensate is drained, then close S6; then, drain the air heater condensate by opening valves S3 and S4. Leave valves S1, S2, S3, and S4 open.
5. See that discharge air valve A6 from the tower and valve A3 are both open. Also, be sure that either air valve A4 or A5 is open.
6. Open valve W9 supplying water to the air cooling coils on the delivery line from the blower.
7. See that valves A1 and A2 at the blower intake are both closed. Then start the blower and adjust the air flow rate until the pressure drop across the orifice is approximately 20 inches of water, using the two gate valves A1 and A2.
8. By manipulating air valves A4 and A5, adjust the dry bulb temperature of the inlet air to approximately 120°F.
9. Heat the tower by allowing the warm air to blow through it until the temperature of the air leaving the tower reaches approximately 100°F without admitting any water to the tower. While waiting for the temperature to reach 100°F, proceed to Steps 10 and 11.
10. This step will purge water inlet lines and clean the tank. Open W5 and W6 to route the water to the floor drain. Open W1 and W2 completely. Completely open W3 until the cold-water temperature is constant and the water runs clear; then close W3. Next, completely open W4 until the hot-water temperature is constant and the water runs clear; then close W4. If either F1 or F2 are clogged, remove the respective filter, clean it, and reinsert the filter. Ensure that the tank is clean and debris has been washed down the drain. Close W6 and fill the tank 1/3 full with water having a temperature between 10 and 20°F below the wet bulb temperature of the incoming air. Add make-up water as needed during the experiments.
11. Open W11 and Close W12. Wait until Step 9 is complete before proceeding.
12. Reduce the air flow using valves A1 and A2 until the pressure drop across the orifice is 2 inches of water.
13. Turn on the "PUMP & HEATER" switch (located on the back of the indicator panel) and immediately ensure that there is flow through the rotameter; NEVER run the pump dry. Air trapped in the pump will inhibit performance – this can be avoided by running the pump for approximately 10 seconds, tuning it off and restarting it. Admit water to the tower at a rate of 0.2 gal/min as indicated by the rotameter.

14. The temperature of the inlet water fed to the tower should be adjusted to the wet-bulb temperature of the inlet air.

The temperature controller can be toggled between active and inactive by pressing the “Run/Stop” button. To set the temperature controller, push “Set”, use the arrow buttons to change the setpoint, press “Set” again.

Soon, the wet-bulb temperature of the exit air should approach the wet-bulb temperature of the entering air. The ideal situation, which actually can be approached quite closely, is to have the following temperatures to be identical: inlet water, outlet water, inlet wet-bulb and outlet wet-bulb.

#### *D. Experimental Conditions*

Each of the runs indicated in the following outline should be of at least 10 minutes duration, during which time the tower should be operating at substantially stable conditions. No steady-state data should be taken until all conditions (flow rates, relative humidity of the air, inlet and outlet temperatures of the water) have remained constant for a period of at least five minutes.

Prior to starting the experiments, and while the tower is warming up, verify that the relative humidity sensors are working properly. Use the wet and dry-bulb temperatures at the base of the tower to check the accuracy of the relative humidity sensors. If the tower is dry inside and no water is being added to the tower, the same humidity should be shown by air entering and air leaving. Be sure the wet-bulb thermometer wicks are wet. Misoperation of the wet bulb can be due to dirt, which keeps water from feeding properly up the wicks. New wicks may be needed. The metal tubing at the bottom of the thermometer wells is movable, after loosening the Swagelok™ nuts, to facilitate replacement of the wicks. The tubing is also marked to provide normal-operation insertion distance.

Determine the humidity of the air in the laboratory with the motor-driven psychrometer. Note that a pressure correction may be required in computing humidities since usual psychrometric charts are constructed for 29.92 in Hg total pressure. A computer program is available for aid in these calculations.

#### *1. Adiabatic Humidification*

##### *(a) Variable air rate; constant liquid rate.*

Air temperature: the dry-bulb temperature of the inlet air is to be approximately 110°F and should be held constant throughout the series.

Air rate: Start with  $\Delta h = 2''$  H<sub>2</sub>O. Observations are to be made at air rates corresponding to  $\Delta h$  values of 2, 3, 5, 10, 20, 30, and 35'' H<sub>2</sub>O.

Water rate: approximately 0.2 GPM. Hold constant throughout this series.

Water temperature: adjusted to the wet-bulb temperature of the inlet air.

*(b) Variable liquid rate; constant air rate.*

Air conditions: inlet dry-bulb air temperature is to be approximately 110°F;  $\Delta h$  across the orifice is to be held constant throughout the series at 20" H<sub>2</sub>O.

Water conditions: inlet temperature to equal wet-bulb temperature of inlet air. One liquid rate greater than and one less than 0.2 GPM.

Note: There are two major precautions that must be observed to collect usable data on adiabatic humidification:

- 1) The exit air *must not be saturated* (relative humidity < 100%). If the air leaves saturated, log-mean values for  $\Delta T$  and  $\Delta Y$  cannot be computed.
- 2) The inlet water temperature should be adjusted to within a few tenths of a degree of the inlet air wet-bulb temperature. (This will also be very close to the wet-bulb temperature of the exit air.) If the temperature of the inlet water is allowed to drift appreciably *below* the wet-bulb temperature, negative values of  $\Delta Y$  will be obtained.

2. *Water Cooling*

Air conditions: inlet dry-bulb air temperature is to be approximately 110°F;  $\Delta h$  across the orifice is to be 20" H<sub>2</sub>O.

Water conditions: rate to be approximately 0.2 GPM. Water temperature approximately 10°F above the dry-bulb temperature of the inlet air. Hot water may be added to the tank to aid the in-line heater reaching the desired temperature.

3. *Dehumidification*

Inlet air conditions: start with the same conditions as for water cooling, then admit steam to the air line by opening valve S5 until the dry-bulb temperature rises to approximately 130°F. The inlet air should not be completely saturated.

Water conditions: rate to be approximately 0.2 GPM. The temperature should be as low as possible. To facilitate low temperature operation, ensure the temperature controller is on "Stop", route the column outlet water to the floor drain (no recycle), turn off the PUMP & HEATER switch, drain tank with W6, close W6, refill tank with cold water and restart pump.

*Note: The exiting air may be saturated in this case, but there will always be a finite  $\Delta Y = (Y^* - Y)$  at the top of the tower due to the low temperature of the inlet water.*

#### IV. Calculations and Graphs

A spreadsheet program **HUMIDANAL** is available to perform most of the repetitive calculations. To determine fluid properties, see Perry's Handbook [4] for a complete psychrometric chart and corresponding tables.

- A. Calculate values of  $K_Y a_M$  with the aid of Equations 1a and 2 for the adiabatic humidification runs.
- B. Calculate values of  $h_G a_H$  with the aid of Equations 3a and 4 for those runs.

*Note on Calculation of Transport Coefficients:*

In the adiabatic humidification runs, the inlet water temperature is controlled such that there is no heat transfer into or out of the water phase. Therefore, the water temperature should not change from top to bottom of the column, and the interfacial temperature is equal to the bulk liquid temperature.

Use Equations 1a and 3a to calculate the gas-film mass and heat transfer coefficients. Actually, the computer program HUMIDA does this for you. Equations 1a and 3a use the approximation that the liquid temperature is constant.

The psychrometric chart is difficult to read very precisely. Note that extensive tables of the saturation humidity and enthalpy values and larger charts are available in *Perry's Handbook*. If necessary, numerical analysis of the wet-bulb lines may be done by recognizing that the wet-bulb lines are essentially lines of constant enthalpy for the air-water system. Thus, the wet-bulb temperature of an air stream indicates its enthalpy. Combining the definition of the gas-phase enthalpy with the measured dry-bulb temperature allows one to calculate the gas humidity.

Because of the presumably large value of  $h_L$ , we do not distinguish between the gas-film coefficient  $k_Y$  and the overall coefficient  $K_Y$  in this experiment. That is, we take  $Y_i = Y^*$  and  $H_i = H^*$  throughout, which neglects any heat-transfer resistance in the liquid phase.

- C. For the adiabatic humidification runs, calculate  $(h_G a_H)/(K_Y a_M)(c_{s,av})$ .
- D. Calculate energy balances for the water cooling and dehumidification runs, and also for one of the adiabatic humidification runs. (That for which  $\Delta h = 20''$  water is suggested.) A datum temperature of 32°F for water but 0°F for air will be convenient as it will permit use of the psychrometric tables for obtaining enthalpy values. Clearly indicate what all numerical terms used in the computations represent.
- E. For the adiabatic humidification runs, plot the following curves on separate graphs and offer appropriate comments:
  1. A log-log plot of  $(K_Y a_M)$  vs.  $G_s/A$  for the variable-air-rate series of observations. Draw a curve through these points, and then plot the values obtained for the variable liquid flow rate. Clearly differentiate these points from the others.

2. A log-log plot of  $h_G a_H$  vs.  $G_s/A$  observing the same directions as in the preceding plot.
  3. An x-y plot of  $K_Y a_M$  vs.  $L/A$  for the variable liquid rate series (air rate constant). To prevent obtaining a distorted picture, care must be taken to choose a scale for  $K_Y a_M$  that is not too "open". In general, the scale should be selected so that it is approximately as open as the logarithmic plot of  $K_Y a_M$ .
  4. An x-y plot of  $h_G a_H$  vs. liquid rate for the variable liquid rate series (air rate constant). Observe the same precautions in selecting a scale of  $h_G a_H$  as indicated for part 3 above.
- F. Using the smooth curves representing  $K_Y a_M$  vs.  $G_s/A$  and  $h_G a_H$  vs.  $G_s/A$  (Plots 1 and 2 in E above), calculate the ratio of  $h_G a_H / K_Y a_M$  and compare with the average humid heat,  $c_{s,av}$ . Do this at the highest and lowest air rates and at one intermediate air rate, approximately halfway between. Use the values of  $K_Y a_M$  and  $h_G a_H$ , *as read from the curves*, because the curve will tend to average out experimental errors.
- G. On the basis of the transport-coefficient results obtained for adiabatic humidification, calculate the outlet conditions to be expected for either water cooling or dehumidification on the assumption that the resistance to heat transfer in the aqueous phase is negligible. Compare the calculated values with your experimental observations.

*Notes on Simulation of Water Cooling or Dehumidification:*

After the volumetric transport coefficients have been determined (and correlated with flow rates), they may be used for design or simulation of general air-water contacting processes in this column or a similar one. You should do the calculations to predict the conditions of the outlet streams for either the water-cooling run or the dehumidification run by using transport-coefficient values obtained in the adiabatic humidification tests.

These more general operations are more complicated than adiabatic humidification because there are three quantities changing with position,  $T_G$ ,  $T_L$ , and  $Y$ . In principle, we need to solve three simultaneous rate equations to describe this situation. Two of the relevant equations are the differential gas-phase mass and energy balances, Equations 1 and 3. The third required equation could be the differential energy balance for the liquid phase, but that may be replaced by the enthalpy balance given in Equation 5 if the gas enthalpy is expressed in terms of  $T_G$  and  $Y$ .

Fortunately, for the air-water system, the problem can be simplified a bit by considering gas enthalpy as the gas-phase property of interest before temperature and humidity separately. This fortuitous situation arises because the Lewis number is close to unity, and there results a single rate equation for gas-phase enthalpy transport, namely Equation 6.

The enthalpy balance between the phases, Equation 5, leads to the operating diagram shown in Figure 4. The operating line is the path of coexisting gas enthalpy and liquid-temperature values along the height of the column. Its slope is determined by the ratio of flow rates, but its specific location, that is, its distance from the equilibrium curve, is determined by Equation 8 or 9. Locating the position of the operating line, which then gives the outlet liquid temperature as well as the outlet gas enthalpy, involves solving either Equation 8 or Equation 9 by trial and error, that is, by guessing one of the outlet conditions, which fixes the position of the operating line, and then integrating the driving-force integral to see whether the equation is satisfied.

As with Equations 1 and 3, one might make the approximation that the equilibrium curve in Figure 4 is linear. Then log-mean driving forces are obtained. For example, Equation 8 may be approximated as

$$\frac{K_Y a_M Z}{G_S / A} \approx \frac{H_2 - H_1}{\Delta H_{\text{ln}}} \quad (8a)$$

where the log-mean enthalpy difference is defined analogously to those quantities given in Equations 2 and 4.  $Z$  is the total column height, and the quantity  $\frac{G_S / A}{K_Y a_M} \equiv HTU$  is called the height of a transfer unit, which is a measure of column efficiency.

If Equation 8a is solved directly for the outlet gas-phase enthalpy, the result fixes the position of the operating line. Then the outlet liquid temperature  $T_{L1}$  can be calculated from Equation 7. (After these equations are solved, one should check the accuracy of the log-mean approximation by computing the integral in Equation 8 numerically.)

Although solving Equations 7 and 8 can predict the outlet liquid temperature, it does not provide the humidity or temperature of the outlet air explicitly but only the outlet-gas enthalpy value, which can correspond to any number of combinations of those two former variables. To predict their values, one must integrate the gas-phase heat and mass-transfer rate equations separately.

Consider the differential energy balance given in Equation 3. This equation may be integrated numerically to obtain  $T_G(z)$ . In the integration, we need to know  $T_i = T_L$  as a function of  $z$ , position in the column. That local liquid temperature may be obtained by returning to Equation 9, written for an arbitrary value of  $z$  rather than the total  $Z$ :

$$\frac{K_Y a_M z}{L / A} = \frac{z}{HTU} = \int_{T_L(z)}^{T_{L2}} \frac{dT_L}{H^* - H} \approx \frac{T_{L2} - T_L(z)}{\Delta H_{\text{ln}}} \quad (10)$$

where the log-mean enthalpy difference in the latter approximation is calculated for only the top portion of the column down to position  $z$ , that is, for the relevant portion of the operating line in Figure 4 between  $T_{L2}$  and  $T_L(z)$ .

By repeated application of Equation 10 one can construct a table or graph of values for  $T_L(z)$  for  $0 < z < Z$ , and then those values may be used to integrate Equation 3, with  $T_i = T_L$  at each value of  $z$ . Solving Equation 3 generates a table or graph giving  $T_G(z)$  and specifically  $T_G(Z) = T_{G2}$ , the outlet-gas temperature.

After  $T_{G2}$  is computed, the exit-gas humidity  $Y_2$  can be calculated from the known value of  $H_{G2}$  and the definition of the gas enthalpy.

There are number of ways to integrate Equation 3 in addition to finite-difference methods. For example, note that Equation 3 may be written as

$$\frac{dT_G}{dz} + \alpha T_G = \beta T_L(z) \quad (3b)$$

where  $\alpha$  and  $\beta$  are known constants and  $T_L(z)$  is not constant as in adiabatic humidification but is known in tabular form from the solution of Equation 10. Equation 3b is a first-order, linear, non-homogeneous differential equation, whose solution can be written in terms of an integrating factor. The solution can thus be computed readily by numerical evaluation of an integral containing the known values of  $T_L(z)$ .

## V. Report

In addition to the items required by Section IV., the instructor may ask some of the following questions:

1. Strictly speaking, in the water cooling and dehumidification runs it cannot be proven that the correct mean to use is a logarithmic mean. Why?
2. Why is it of interest to calculate the ratio of  $h_G a_H / K_Y a_M$  and compare it with  $c_{s,av}$ ?
3. What effect does the pattern of the spray have on the coefficients calculated?
4. How could the results of this experiment be used in the design of larger scale humidifying equipment, for example?
5. Estimate the accuracy of the coefficients determined in this experiment. What factors affect the accuracy?
6. Under what conditions is the Lewis number truly unity?
7. For one run (that for which  $\Delta h = 20$ '' water is acceptable) assume an average diameter of the drops in the spray and estimate values of  $a_H = a_M$  (neglecting wall effects) and  $k_Y$  and  $h_G$ .
8. Why are the inlet and outlet wet-bulb temperatures of the air for adiabatic saturation not the same? Is heat gained or lost through the walls of the insulated tower? Prove your answer.
9. Qualitatively discuss the results of water cooling and dehumidification.
10. Consider the spray tower to be a well mixed system. On what basis might this assumption be reasonable? Estimate the transfer coefficients for the variable

liquid rate runs and compare the results with those obtained when the system was considered in countercurrent axial flow.

## REFERENCES

1. McCabe, Smith, and Harriot, Unit Operations for Chemical Engineers, 5th ed., McGraw Hill, Inc., New York 1993.
2. Treybal, R. E., Mass Transfer Operations, 3rd ed., McGraw-Hill, Inc., New York (1980).
3. Foust, A. S., Wenzel, L. A., Clump, C. W., Maus, L., and Andersen, L. B., Principles of Unit Operations, 2nd ed., John Wiley and Sons, New York, 1980.

Additional references of interest:

4. Perry, R. H. and Green, D., Perry's Chemical Engineers' Handbook, 6th ed., McGraw Hill, Inc., New York 1984.
5. Kelly, H. W. and L. K. Swenson: "Comparative performance of cooling tower packing arrangements", Chem. Eng. Prog. 52(7), 263 (1956).

## NOMENCLATURE

<u>Symbol</u>	<u>Description</u>
a	Water/air interfacial area density, ft <sup>-1</sup>
A	Cross-sectional area of tower, ft <sup>2</sup>
c <sub>L</sub>	Specific heat of water, Btu/lb-°F
D	Diameter, ft
g	Acceleration of gravity, ft/s <sup>2</sup>
G <sub>s</sub>	Dry air flow rate, lb/hr
h <sub>G</sub> , h <sub>L</sub>	Heat transfer coefficients, Btu/ft <sup>2</sup> -hr-°F
Y	Absolute humidity, lb/lb DA
Y*	Absolute humidity at saturation, lb/lb DA
K <sub>Y</sub>	Mass transfer coefficient, lb/ft <sup>2</sup> -hr
L	Liquid flow rate, lb/hr
P	Pressure, in H <sub>2</sub> O, in Hg
c <sub>s</sub>	Humid heat capacity or air-water mixture, Btu/lb-°F
T	Dry bulb temperature, °F
T <sub>L</sub>	Liquid temperature, °F
z	Column length, ft
ρ	Density, lb/ft <sup>3</sup>
<u>Subscript</u>	<u>Description</u>
av	Average
btm	Evaluated at bottom of tower
ln	Log-mean
top	Evaluated at top of tower
w	Wet-bulb

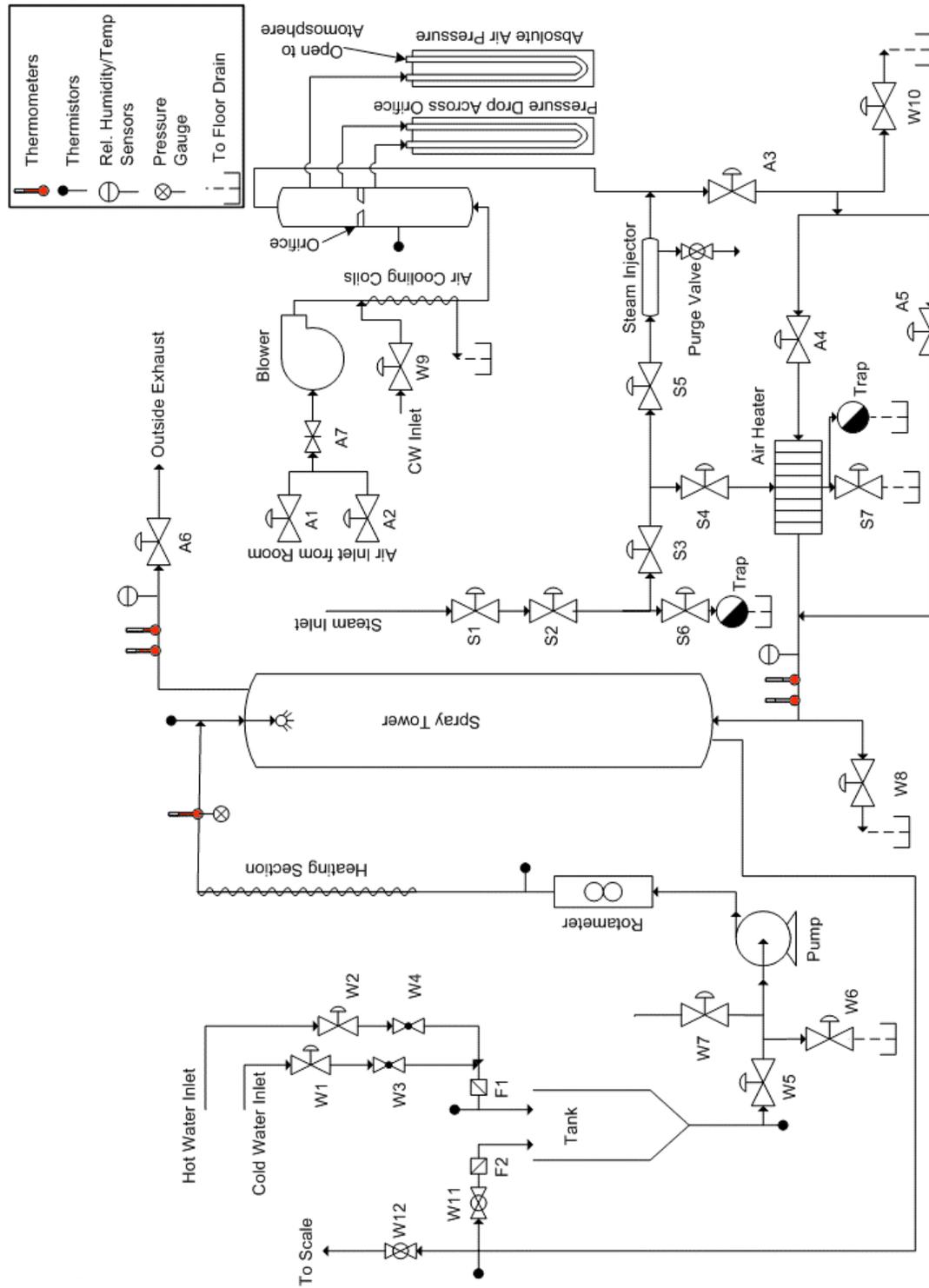


Figure 6. Humidification experiment apparatus. Overall flow diagram.



Data Sheet  
 Adiabatic Humidification-Water Cooling-Dehumidification

Date: \_\_\_\_\_ Students: \_\_\_\_\_ Barometer: \_\_\_\_\_

Run No.	Type of Operation		Orifice Data				Dry Air Rate G lb/hr	Top of Tower			Bottom of Tower			Time of Run Min	Wt. H <sub>2</sub> O from Bottom of Tower lbs	Water Rate L lb/hr
			ΔH "H <sub>2</sub> O	Gauge Press "H <sub>2</sub> O	P <sub>o</sub> "Hg	Temp T		Outlet Air		Inlet Water temp	Inlet Air		Out-let water temp			
								d.p. temp	w.b. temp		d.p. temp	w.b. temp				
		Start														
		End														
		Ave.														
		Start														
		End														
		Ave.														
		Start														
		End														
		Ave.														
		Start														
		End														
		Ave.														

ChE 424 Operations and Process Laboratory  
Humidification, Dehumidification, and Water-cooling Experiment

HumidAnal - This program is an Excel spreadsheet and also an EES document, both available on the ChE 424 web site\* to facilitate the data analysis in this experiment. For each run one should enter inlet and outlet temperatures and flow-rate data. The program calls for initial and final values taken over a 5-10 minute period. The equipment should be at steady-state, as indicated by the digital-meter readouts, but averaging over two observations accounts for fluctuations. Both programs will save the data and balance calculation results to a spreadsheet file or table, suitable for further workup and transfer to the student's computer. You may wish to try both software tools to decide which format is more suitable. Note that the Excel program uses macros, so it will be necessary to bypass any antivirus tools on your computer that automatically prevent macros from loading if you wish to use the Excel spreadsheet.

In addition to the raw data, the program requires saturation gas-enthalpy values corresponding to the measured wet-bulb temperatures. These values can be obtained from the psychrometric tables in Perry. The gas enthalpy, regardless of its humidity and dry-bulb temperature, should be the same as the enthalpy of the saturated system at the wet-bulb temperature. Then the humidity can be calculated from definition of enthalpy and the dry-bulb temperature. This is a more accurate approach for getting humidity than trying to read the psychrometric charts.

Sheet 3 of the Excel program calls for the reference temperatures used in defining air and water enthalpies. In most psychrometric tables, including those in Perry, the reference temperature for air is 0°F, whereas that for water is 32°F.

The program calculates the water mass balance and an energy balance, which indicate the accuracy and reliability of the subsequent calculations because the basic analysis assumes that there is no energy loss from the column and, of course, no loss of water at steady state. For the adiabatic-humidification experiments, the program can calculate gas-film heat and mass-transfer coefficients as well as the Lewis ratio. For the more general cases, water cooling or dehumidification, an enthalpy difference is used to calculate the mass-transfer coefficient. The latter calculation assumes that there is no temperature gradient in the liquid phase. Also, the calculation in the spreadsheet uses the log-mean enthalpy difference as the average driving force. A more accurate analysis requires numerical or graphical integration of the enthalpy difference between the enthalpy- temperature operating line and the equilibrium curve on a plot of gas enthalpy *versus* liquid temperature.

\*<http://courses.engr.wisc.edu/ecow/get/che/424/>

*this page intentionally left blank*

## Lecture Notes for Pump Formal

Date: \_\_\_\_\_

Instructor: \_\_\_\_\_

---



## Test of a Centrifugal Pump

### I. INTRODUCTION

The goal of the pump experiment is to relate the power requirement,  $P$ , and the head delivery,  $H$ , to the flow throughput,  $Q$ , and the pump speed,  $N$ ; and to efficiently represent these results. Such information can be obtained in a straightforward manner if one could obtain a solution to the velocity and pressure fields inside the pump. Solving for these fields is, unfortunately, a very difficult problem, so we must resort to dimensional analysis and experimentation.

### II. EQUIPMENT (see Pump System Flow Sheet, see Fig. 1)

- A. Single-stage, single-suction, 6.06" diameter open-impeller centrifugal pump rated at 20 ft fluid and 38 gal/min at 1725 rev/min (Pump).
- B. Flow loop consisting of a 50-gal holding tank with nominal 1-in., Schedule-40 pipe downstream of pump and nominal 2-in., Schedule-40 pipe upstream of pump with necessary valves and fittings (Tank, pipes, fittings).
- C. Impact-tube meter with nominal 1-in., Schedule-40 pipe and nominal 1/4 in. copper tube (1" pt).
- D. Impact-tube meter with nominal 1-1/2 in., Schedule-40 pipe and nominal 1/4 in. copper tube (1-1/2" pt).
- E. Venturi meter with nominal 1-in., Schedule-40 pipe upstream dimensions and a 1/2-in. throat (v).
- F. Water-over-mercury manometers (PDI).
- G. Solid-state piezoelectric differential pressure transducers, load cell, and digital readout display (PDI).
- H. Load cell scale or lab. scale (WI).
- I. Torque meter (TI).

### III. EXPERIMENTS

#### A. Calibration of Flow Meters

Use the water directly from the city water line. Be careful to open and close all valves slowly to avoid entraining air bubbles into the water. Eliminate bubbles in pressure taps by flushing water through manometers bypass. Be certain that the pressure transducer/manometer bypass valves are closed when data are being taken. Open the throttling globe valve slowly to go from zero flow to maximum ones, and look always at the manometers to avoid going over the maximum range.

1. Calibrate the venturi meter over the range of 5-20 gal/min. (Close the manometer taps across the globe valve to avoid blowing mercury out of the manometer.)

2. Calibrate the impact-tube meter over the range of 20-45 gal/min. (Close the manometer taps across the globe valve to avoid blowing mercury out of the manometer.)

#### B. Characterization of the Pump

At the four speeds of 1200, 1500, 1800, and 2000 rpm, determine the total head developed and the shaft power required for flow rates ranging from zero to the maximum possible flow at flow intervals of 5 gal/min or less as required. Make sure the tank is nearly full to avoid air entrainment. Shaft power measurements can be obtained from the load cell or the laboratory scale attached to the motor casing. Flow rates are to be measured with the calibrated flow meters.

Note: You may have to periodically readjust a given set pump speed during a fixed pump speed run.

#### C. Calibration of a Globe Valve

Changing the pump speed for given 1-1/2" globe valve stem positions, pass different flow rates through the valve. Measure the flow rates using the 1 1/2" pitot tube or the venturi meter and the pressure drop across the valve using the Pressure Differential Manometer connected to the 1 1/2" globe valve. To change the flow rate through the valve change the pump speed.

#### D. Effect of Air Entrainment on Pump Performance

At a pump speed of \_\_\_\_\_ rpm, determine the total head developed for flow rates ranging from zero to the maximum possible flow at flow intervals of 5 gal/min or less as required when air is entrained to the amount of 0, 0.5, and 1 volume percent with respect to the liquid volumetric flow rate. It is best here to fix pump speed then fix a flow rate (by means of the pressure drop across the flow meters) and then set the volumetric air flow rate at its needed value. Do not forget the run with 0 water flow rate and no air flow rate.

### IV. THEORETICAL DEVELOPMENT

- A. Develop expressions for the performance of the impact-tube and venturi meter, characterizing the relationship between the static pressures maintained at the meter taps and the flowmeter flow rates.
- B. Develop expressions for the computation of shaft power and hydraulic power in connection with pump performance.
- C. Develop an expression for the total head developed by the pump in terms of the static pressures measured at the inlet and outlet of the device.
- D. Relate the manometer reading to the static pressures measured at the inlet and outlet of the pump by application of the hydrostatic formula and combine this result with the expression for total head developed by the pump in part A in order to obtain the relationship between the pump head and the manometer readings.

## V. GRAPHICAL REPRESENTATION OF DATA AND RESULTS

- A. On a log-log scale, plot the data obtained in the calibration of the flow meters with flow rate in units of gal/min on the ordinate vs.  $\Delta P$  in units of psi on the abscissa.
- B. Make a log-log plot of (i) total head in units of feet for zero and maximum flow, (ii) maximum flow in units of gal/min, and (iii) shaft power at maximum flow rate in units of horsepower on the ordinate vs. pump speed in units of rev/min on the abscissa.
- C. Plot the pump head in units of feet and efficiency as a fraction on the ordinate vs. flow rate in units of gal/min on the abscissa with pump speed in units of rev/min as the parameter. Use a single graph.
- D. On a separate graph, plot pump head in units of feet on the ordinate vs. flow rate of water in units of gal/min on the abscissa with volume-percent entrained air as the parameter for the specified pump speed, including the 0% entrained air curve.
- E. Dimensional analysis of the equations of motion as applied to a centrifugal pump suggests, for systems of fixed geometry (see handout), that the pump dimensionless head,  $H^*$ , be represented as  $g\Delta H/(ND)^2$ , the dimensionless shaft power,  $P^*$ , be written as  $\frac{\Omega\Gamma}{\rho N^3 D^5}$ , the dimensionless flow rate,  $Q^*$ , be represented as  $Q/ND^3$  and dimensionless speed,  $Re$ , represented as  $\rho D^2 N/\mu$  with  $g$  = acceleration of gravity,  $\Delta H$  = total head of pump,  $N$  = pump speed,  $D$  = impeller diameter,  $\rho$  = liquid density,  $\mu$  = liquid viscosity,  $\Omega$  is the shaft angular velocity, and  $\Gamma$  is the shaft torque.
  1. Obtain correlations for  $H^*(Q^*)$  and  $P^*_{\text{shaft}}(Q^*)$  (see Pump Dimensional Analysis).
  2. Why does the dependence of  $H^*$  and  $P^*_{\text{shaft}}$  on  $Re$  disappear?
- F. On a log-log scale, plot the Globe Valve flow rate and  $\Delta P$  in units of gal/min and psi, respectively, for given fixed valve stem positions.

## VI. DISCUSSION (some ideas that must be included in the report)

- A. Review the experimental results for the performance of the pump relative to all operation conditions investigated, all calculations made, all characteristics determined and performance specification. Compare with accepted and reported performance characteristics as indicated in available literature sources.
- B. Review the experimental results for the performance of the flow meters. Compare with accepted and reported performance characteristics as reported in available literature sources.
- C. Consider the performance of both the meters and pump if a new liquid with a specific gravity 50 percent greater than water were used in the study.
- D. Explain why small amounts of entrained air so markedly reduce the total head of the pump at low flow rates compared to high flow rates.

- E. Explain why the maximum measurable flows delivered by the pump occur at fluid head within ca. 70-80 percent of the heads developed at zero flow rate.
- F. Error analysis.
- G. Review the results of the dimensional analysis of the performance curves for the pump with no air entrainment.
- H. Address the correctness of the values of the discharge coefficients obtained for each of the flow meters.
- I. Check that at a given pump speed and maximum flow rate the piping system has a need of a pump head (calculated by means of a mechanical energy balance to the whole pipe loop) equal to the measured pump head.
- J. Address to the variation of the pump capacity, head development and shaft power consumption with pump speed.
- K. Refer to the ratio of spatial volume-fraction of entrained air within the pump body to temporal volume-fraction of entrained air entering and leaving the pump body as a function of pump capacity for the assigned pump speed.

VII. PUMP FLOW SYSTEM (See Fig. 1)

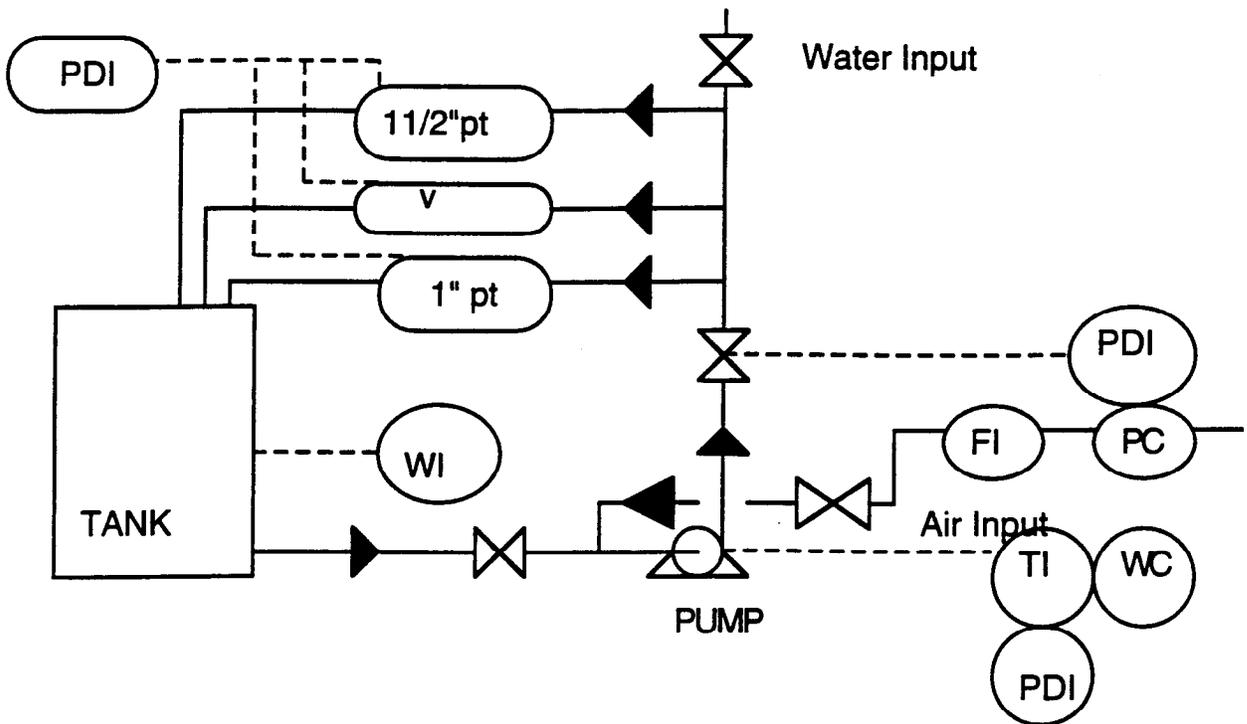


Figure. 1. Pump system flow sheet

Symbols:

- v, Venturi meter
- pt, Pitot Tube (Impact Tube) meter
- WI, Weight Indicator (scale or load cell)

FI, Flow Indicator

PC, Pressure Controller

TI, Torque Indicator

PDI, Pressure Differential Indicator (Hg-H<sub>2</sub>O manometer or solid state piezoelectric Pressure Differential Transducers)

WC, Speed Controller and Indicator

## VIII. DIMENSIONAL ANALYSIS (DJK, May 24, 1994)

### 1.1 General Analysis

For the flow of water inside a pump (see figure 2), the fluid may be assumed to be incompressible and isothermal. Hence the governing equations for the velocity and pressure fields are (BSL):

$$\nabla \cdot \mathbf{v} = 0 \quad (1)$$

$$\rho \frac{D\mathbf{v}}{Dt} = -\nabla p + \mu \nabla^2 \mathbf{v} + \rho \mathbf{g} \quad (2)$$

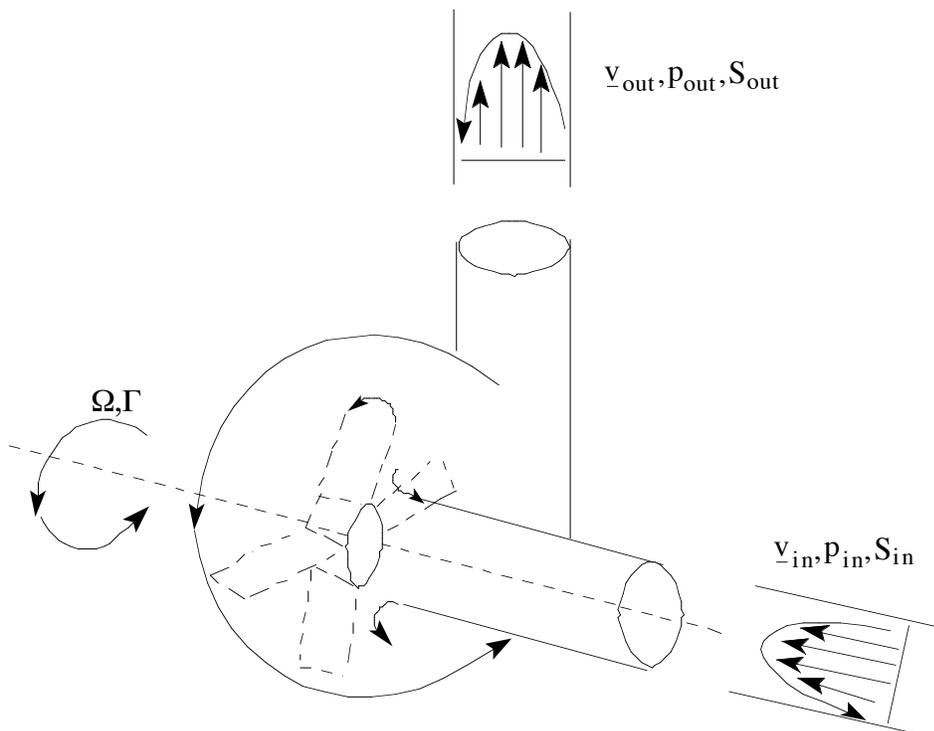


Figure 2. Pump Fluid Mechanics Detail.

The appropriate boundary conditions are

$$\mathbf{v} = \mathbf{v}_{\text{impeller}} \text{ on impeller surface,} \quad (3)$$

$$\mathbf{v} = 0 \text{ on all stationary surfaces,} \quad (4)$$

$$-\int_{S_{in}} \mathbf{v} \cdot \mathbf{n} \, dA = Q, \quad (5)$$

$$\int_{S_{out}} \mathbf{v} \cdot \mathbf{n} \, dA = Q, \text{ and} \quad (6)$$

$$p = p_{in} \text{ on } S = S_{in}, \quad (7)$$

where  $\mathbf{n}$  is the unit normal vector pointing out of the control volume, and  $Q$  is the volumetric flow rate. The complexity arises from the nonsimple geometry, and the fact that the rotating impeller generates time dependent velocity and pressure fields; hence, we resort to dimensional analysis as follows.

We choose the following dimensional scales:

$$\begin{aligned} \text{length} &= D \\ \text{velocity} &= ND \\ \text{time} &= 1/N \\ \text{pressure} &= \rho N^2 D^2 \end{aligned}$$

where  $D$  is the impeller diameter, and  $N$  is the impeller rotation rate in revolutions per unit time. The dimensionless governing equations become

$$\nabla^* \cdot \mathbf{v}^* = 0, \quad (8)$$

$$\frac{D\mathbf{v}^*}{Dt^*} = -\nabla^* p^* + \frac{1}{\text{Re}} \nabla^{*2} \mathbf{v}^* + \frac{1}{\text{Fr}} \mathbf{g}, \quad (9)$$

where the asterisks designate dimensionless quantities, and the Reynolds and Froude numbers are given by

$$\text{Re} = \frac{\rho D^2 N}{\mu},$$

$$\text{Fr} = \frac{N^2 D}{g}.$$

The boundary conditions in dimensionless form are

$$\mathbf{v}^* = \mathbf{v}_{\text{impeller}}^* \text{ on impeller surface,} \quad (10)$$

$$\mathbf{v}^* = 0 \text{ on all stationary surfaces,} \quad (11)$$

$$-\int_{S_{in}^*} \mathbf{v}^* \cdot \mathbf{n} \, dA^* = Q^*, \quad (12)$$

$$\int_{S_{out}^*} \mathbf{v}^* \cdot \mathbf{n} \, dA^* = Q^*, \text{ and} \quad (13)$$

$$p^* = \frac{p - p_{in}}{\rho N^2 D^2} = 0 \text{ on } S_{in}, \quad (14)$$

where  $Q^* = Q/(ND^3)$ . Inspection of the governing equations and boundary conditions leads us to the following results for the dependence of the dimensionless velocity and pressure fields on the variables and parameters.

$$\mathbf{v}^* = \mathbf{v}^*(\mathbf{x}^*, t^*, Q^*, Re, Fr, \text{shape}), \quad (15)$$

$$p^* = p^*(\mathbf{x}^*, t^*, Q^*, Re, Fr, \text{shape}). \quad (16)$$

Note that the dependence on the dimensionless flow rate  $Q^*$  arises from the boundary conditions. The shape dependence also arises from the boundary conditions. For most pump problems, it is reasonable to neglect the dependence of  $\mathbf{v}^*$  on  $Fr$  (i.e., gravity does not influence the velocity profile). Note that this immediately reveals the dependence of the time-averaged pressure drop across the pump on scaled parameters:

$$\Delta p^* = \Delta p^*(Q^*, Re, \text{shape}). \quad (17)$$

## 1.2 Total Head

The total head,  $H$ , is a convenient measure of the performance of pump, and is defined by

$$gH = \left( \frac{p}{\rho} + gz + \frac{1}{2}v^2 \right)_{\text{out}} - \left( \frac{p}{\rho} + gz + \frac{1}{2}v^2 \right)_{\text{in}}. \quad (18)$$

When the entrance and exit conduits are the same diameter, or when the velocity differences are small, the kinetic energy contributions vanish. Height differences are also usually negligible, and the expression for the total head reduces to

$$gH = \frac{\Delta p}{\rho}. \quad (19)$$

In dimensionless form,

$$H^* = \frac{gH}{N^2 D^2} = \Delta p^*, \quad (20)$$

and hence, using the dimensional analysis of the previous section, we find the following dependence of the head on the scaled parameters:

$$H^* = H^*(Q^*, Re, \text{shape}). \quad (21)$$

## 1.3 Power

The shaft power input is given by

$$P_{\text{shaft}} = \Omega \Gamma \quad (22)$$

where  $\Omega$  is the shaft angular velocity, and  $\Gamma$  is the torque required to turn the impeller. The torque is related to the velocity and pressure fields by

$$\Gamma = \int_{S_{\text{impeller}}} \mathbf{x} \times (p\boldsymbol{\delta} + \boldsymbol{\tau}) \cdot \mathbf{n} \, dA, \quad (23)$$

where  $\mathbf{n}$  points into the fluid. Scaling the stress and pressure with  $\rho N^2 D^2$ , and using the results from the dimensional analysis in Section 1.1, we find that the dimensionless torque can be written

$$\Gamma^* = \frac{\Gamma}{\rho N^2 D^5} = \Gamma^*(Q^*, \text{Re}, \text{shape}). \quad (24)$$

Since  $\Omega \propto N$ , the dimensionless shaft power depends on the scaled parameters as follows:

$$P_{\text{shaft}}^* = \frac{\Omega \Gamma}{\rho N^3 D^5} = P_{\text{shaft}}^*(Q^*, \text{Re}, \text{shape}). \quad (25)$$

(The dimensionless power is also known as the "power number".) Note that this is the power supplied to the fluid, and that the conversion to useful power will typically be imperfect. The hydraulic power is defined by

$$P_{\text{hyd}} = Q \Delta p \quad (26)$$

or in dimensionless form,

$$P_{\text{hyd}}^* = \frac{P_{\text{hyd}}}{\rho N^3 D^5} = Q^* \Delta p^* = P_{\text{hyd}}^*(Q^*, \text{Re}, \text{shape}). \quad (27)$$

The pump efficiency is defined by

$$\eta = \frac{P_{\text{hyd}}}{P_{\text{shaft}}} = \frac{P_{\text{hyd}}^*}{P_{\text{shaft}}^*} \quad (28)$$

which must depend on scaled parameters as follows:

$$\eta = \eta(Q^*, \text{Re}, \text{shape}). \quad (29)$$

## Lecture Notes for Reactors Formal

Date: \_\_\_\_\_

Instructor: \_\_\_\_\_

---

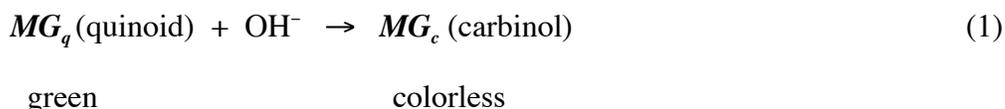


## Chemical Reactors Formal Experiment

### I. Introduction

The goals of this experiment are i) to demonstrate the behaviors of real continuous flow reactors and to compare these behaviors to those of idealized models; ii) to examine the effects of residence times and residence time distributions on the conversions obtained in a reactor network; and iii) to provide experience with computer-based data collection and use of physical properties for monitoring the progress of chemical reactions. During the course of the experiment, students will measure the kinetics of a reaction in a batch reactor, as well as predict and measure reactant conversions in various combinations of continuous flow reactors. Residence time distribution functions for the flow reactors will be used to examine the predictive behavior of models of reactor performance.

This experiment uses for a probe reaction the irreversible fading of an organic dye, malachite green, in a dilute alkaline solution because it is characterized by a simple rate expression under normal conditions. Malachite green (abbreviated  $MG$ ) changes from a green quinoid form to a colorless carbinol form in the presence of  $OH^-$  (Chen, D.T. and Laidler, K.J., 1959)



Although the reaction is first-order in each reactant, the concentration of  $OH^-$  is chosen to be in massive excess such that its concentration can be treated as constant and included in an effective first-order reaction rate constant. Thus,  $r = -r_A = k[OH^-][MG_q] = k_{\text{eff}}[MG_q]$ . This reaction has a half-life of several minutes at room temperature. This reaction is convenient for use in reactor studies because its progress may be monitored continuously using the decrease in the absorbance of the solution as the reaction proceeds.

The reactant,  $MG_q$ , exhibits a maximum absorbance peak at 617 nm and follows the Lambert-Beer law over the range of concentrations studied in this experiment. The fractional conversion,  $f$ , can be related to the consumption of  $MG_q$  as follows:

$$f = 1 - \frac{[MG_q]}{[MG_q]_0} \quad (2)$$

where  $[MG_q]$  and  $[MG_q]_0$  are the instantaneous and initial concentrations of  $MG_q$ , respectively. Because the measured absorbance,  $Abs$ , is directly proportional to the concentration of reactant  $MG_q$

$$f = 1 - \frac{Abs}{Abs_0} \quad (3)$$

In the implementation of this means of monitoring the progress of the reaction, the contributions of the solvent, inerts, and the apparatus to the total absorbance are subtracted out using the features of the computer such that the only remaining contribution is that of the malachite green. Thus, a readily-measured physical property of the solution can be used to monitor the extent of reaction.

## II. Theory

### A. Design Equations for Ideal Reactors

The conversion achieved in any continuous flow reactor depends on the reaction kinetics (rate expression, including the rate constant), the fluid flow rate, and the reactor volume, as well as details of the reactor configuration that influence internal concentration profiles and fluid mixing. The most important parameter is the reactor space time

$$\tau = \frac{V_R}{Q} = \frac{\text{reactor volume occupied by the reacting fluid}}{\text{inlet volumetric flow rate}} \quad (4)$$

This parameter is commonly used in scale up procedures to determine reactor size requirements. Different internal flow patterns can produce different conversions even for the same space time, and the Plug Flow Reactor (PFR) and Continuous Stirred Tank Reactor (CSTR) models are convenient for describing limiting cases of idealized flow patterns.

The design equation for a batch reactor is of the form  $\frac{dN_A}{dt} = V_R \cdot r_A$ . For a first-order reaction in a constant density system, the dependence of the reactant concentration on time can be shown to be  $C_A(t) = (C_{A0}) e^{-kt}$ . This result can be readily restated in terms of fractional conversion as  $f(t) = 1 - e^{-kt}$ . The best test of a model is obtained by replotting the data in a form that linearizes the expected behavior. Thus a plot of  $\ln[1-f(t)]$  against time is commonly used, although other types of plots that linearize the data may be preferable. If the data for the indicated plot of  $\ln[1-f(t)]$  fall on a straight line through the origin, the rate constant can be obtained from the slope of the best fit line through the data.

The design equation for a PFR is  $\tau = \frac{V_R}{Q} = \int_{C_{Ain}}^{C_{Aout}} \frac{dC_A}{-r_A}$  for a constant-density system. For a first-order reaction, this reduces to  $C_A(\tau) = (C_{A0})e^{-k\tau}$ , or  $f(\tau) = 1 - e^{-k\tau}$ , an expression that is of the same mathematical form as that for a batch reactor. The typical variation of fractional conversion with space time for a PFR is shown in Figure R-1.

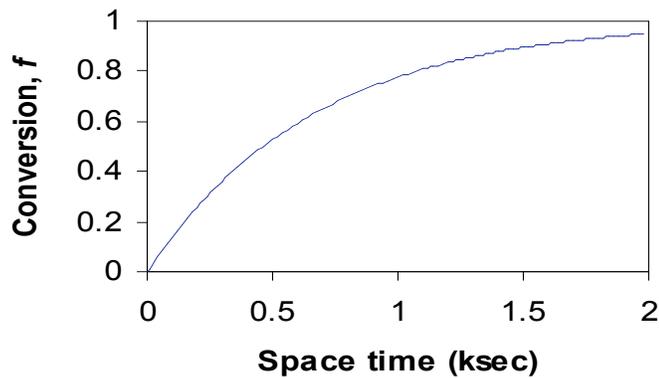


Figure R-1. Conversion in a PFR for  $k = 0.0015 \text{ s}^{-1}$ .

The design equation for a single CSTR is  $\tau = \frac{V_R}{Q} = \frac{C_{Ain} - C_{Aout}}{(-r_A)_{out}}$  for a constant-density system. This equation reduces to  $\frac{C_A(\tau)}{C_{Ao}} = \frac{1}{1 + k\tau}$ , or  $f(\tau) = 1 - \frac{C_A(\tau)}{C_{Ao}} = \frac{k\tau}{1 + k\tau}$ , for a first-order reaction. For a reactor cascade consisting of  $n$  CSTRs in series, all with identical volumes, and each with the same space time  $\tau$ , the concentration of reactant leaving the  $n$ -th CSTR is given by  $\frac{C_{An}(\tau)}{C_{Ao}} = \frac{1}{(1 + k\tau)^n}$ , where  $\tau$  refers to the space time for a single CSTR. Alternatively,  $f_n(\tau) = 1 - \frac{C_{An}(\tau)}{C_{Ao}} = 1 - \frac{1}{(1 + k\tau)^n}$ . The typical variation of fractional conversion with space time for batteries consisting of 1, 2, or 3 CSTRs in series is shown in Figure R-2.

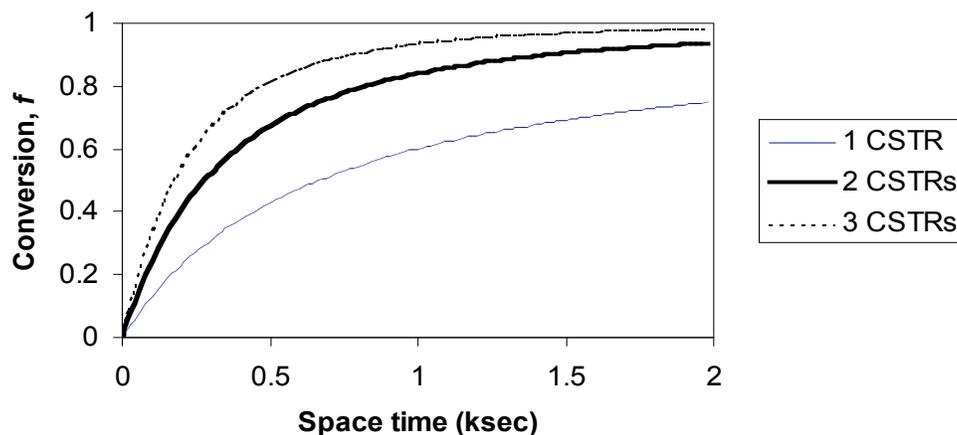


Figure R-2: Conversions in cascades of 1, 2, and 3 CSTRs in series for  $k = 0.0015 \text{ s}^{-1}$ . The indicated space time is that for one of the CSTRs that make up the cascade in question.

## **B. Residence Times in Reactor Networks**

Real and idealized reactors are characterized by different internal flow patterns and degrees of micromixing. These differences can be expressed in terms of the corresponding residence time distribution functions (RTD). The RTD can be extracted from the response of the reactor to changes (step, pulse, sinusoidal, or random) in the feed concentration of reactant. However, the simplest experiments to conduct or analyze are step and pulse variations. (The comments below apply specifically to constant-density systems and non-reactive tracers. For these systems, it can readily be shown that the space time  $\tau$  and the mean residence time  $\theta$  are numerically equal.)

The RTD data can be converted to standard curves by accounting for any background concentration of the tracer material and appropriate normalization of the response curve. The effluent response curves for step changes in the feed concentration of tracer are usually normalized in terms of the size of the step, producing a function  $F(t)$  that describes the *cumulative residence time distribution*. This  $F(t)$  curve represents the volume fraction of the reactor effluent has been in the reactor for a time less than or equal to  $t$ . All  $F(t)$  curves rise monotonically from zero at zero time to 1 at a time less than or equal to infinity. The response to a pulse input is usually normalized in terms of the total quantity of tracer injected. The relative response to a pulse is referred to as  $E(t)$ . The function  $E(t)$  describes the differential residence time distribution.  $E(t)$  indicates the fraction of the feed that entered the reactor at time  $t=0$  that is leaving at time  $t$ . The area under all  $E(t)$  curves is equal to unity. In all cases,  $E(t) = 0$  for  $t < 0$ . At long times all  $E(t)$  curves return to  $E(\infty) = 0$  because all of the tracer eventually leaves the network of reactors.

These two residence time distribution functions are related; since an ideal pulse is modeled by the Dirac delta function, which is the derivative of a step change, it can be demonstrated that  $E(t) = \frac{dF(t)}{dt}$ . Similarly, the cumulative distribution can be obtained by integrating the response to a pulse input,  $F(t) = \int_0^t E(t)dt$ . Experimentally, the pulse is easier to administer, and the  $E(t)$  curve provides a more sensitive display of RTD differences than the  $F(t)$  curve. However, when the RTD for the reactor has a long tail, it is important to collect data at times that are long enough to ensure that the tracer concentration has returned to its base value. One simple way to determine whether all of the tracer has left the reactor is to ascertain the integrated amount of tracer leaving the reactor, and compare this value with the amount originally injected.

There are several ways to determine the mean residence time from RTD data. The simplest procedure is to calculate the mean, using  $\bar{t} = \int_0^{\infty} t dF(t) = \int_0^{\infty} t E(t) dt$ . A more thorough treatment involves fitting a model for the RTD to the observed  $E(t)$  or  $F(t)$  data to obtain the parameters that give the least error in a systematic fashion.

The ideal PFR has no internal mixing, so the effluent concentration follows the inlet concentration profile but with a time delay equal to  $\tau$ . The responses to step and pulse inputs are shown in Figures R-3 and R-4.

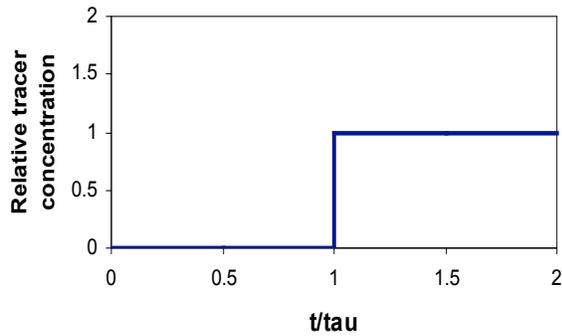


Figure R-3. Cumulative RTD,  $F(t)$ ,  
for a PFR.

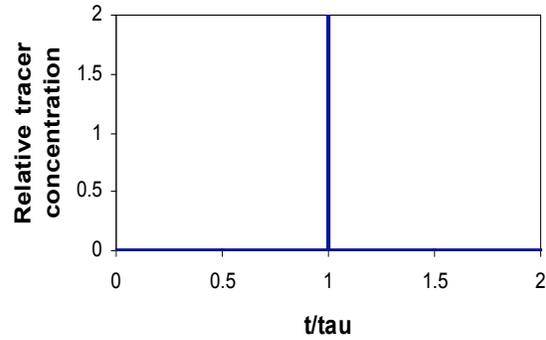


Figure R-4. Differential RTD,  $E(t)$ ,  
for a PFR.

The ideal CSTR has complete, instantaneous internal mixing as the feed enters. In this case the effluent concentration is equal to the average concentration of tracer in the reactor. The responses of a single CSTR to step and pulse inputs are presented in Figures R-5 and R-6.

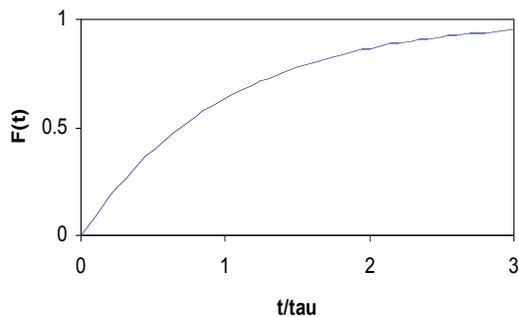


Figure R-5. Cumulative RTD,  $F(t)$ ,  
for a single CSTR.

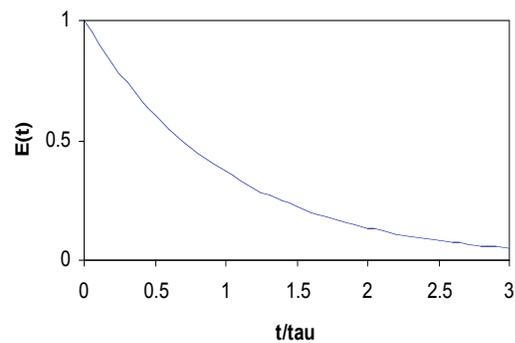


Figure R-6. Differential RTD,  $E(t)$ ,  
for a single CSTR.

For the CSTR, at  $t > 0$ ,  $F(t) = 1 - e^{-t/\tau}$  and  $E(t) = \frac{1}{\tau} e^{-t/\tau}$ .

For  $n$  CSTRs in series, each with the same nominal volume and mean residence time, the corresponding equations for  $t > 0$  are

$$F(t) = 1 - e^{-t/\tau} \left( 1 + \frac{t}{\tau} + \frac{1}{2!} \left( \frac{t}{\tau} \right)^2 + \dots + \frac{1}{(n-1)!} \left( \frac{t}{\tau} \right)^{n-1} \right) \quad (5)$$

and

$$E(t) = \frac{1}{\tau} e^{-t/\tau} \frac{1}{(n-1)!} \left( \frac{t}{\tau} \right)^{n-1} \quad (6)$$

The associated RTD curves are shown in Figures R-7 and R-8 for a cascade consisting of 1, 2, and 3 identical tanks, each with residence time  $\tau$ . Note that the relative time ( $t/\tau$ ) used as the abscissa of these plots is based on the space time ( $\tau$ ) for an individual CSTR, rather than the space time for the cascade as a whole. Also note that the responses of the downstream tanks are represented by curves that are displaced to longer times. The length of the tail increases as the number of stirred tanks increases. If students have had a course in process control, they may recognize these curves as equivalent to the responses of first-, second-, and third-order linear systems.

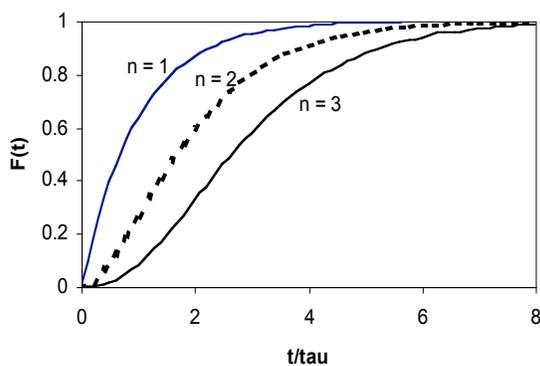


Figure R-7. Cumulative RTD curves,  $F(t)$ , for cascades of 1, 2, and 3 CSTRs.

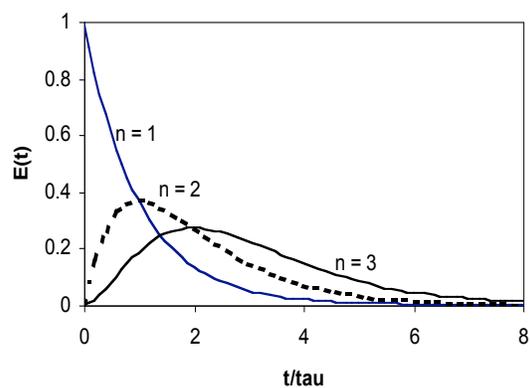


Figure R-8. Differential RTD curves,  $E(t)$ , for cascades of 1, 2, and 3 CSTRs.

Numerous other models are available for describing more complex reactor flow patterns. You may wish to consult standard textbooks for information concerning the Laminar Flow Reactor, the Axial Dispersion model, and the use of  $n$ -CSTRs as an approximation for a non-ideal tubular reactor.

### C. *Segregated Flow Analysis*

The RTD provides information on reactor behavior beyond the mean residence time. However, the RTD does not describe the degree of micromixing, and this factor may be important in considerations of reactions whose rate expressions are not first-order. For first-order reactions, a knowledge of the rate expression and the RTD alone should permit one to predict the effluent composition for any type of reactor. Since any reactor model that satisfies the observed RTD should, in principle, give the correct conversion, we can use the simple segregated flow treatment to construct an imaginary collection of PFRs that operate in parallel to produce the desired RTD. The behavior of this collection of PFRs is then obtained by calculating the weighted average conversion. To determine the effluent concentration of reactant A,

$$C_{Aout} = \int_0^1 C_A(t) dF(t) = \int_0^\infty C_A(t) E(t) dt \quad (7)$$

For the fractional conversion,

$$f_{out} = \int_0^1 f(t) dF(t) = \int_0^\infty f(t) E(t) dt \quad (8)$$

For Equations 7 and 8, one uses the appropriate batch reactor equations to determine  $f(t)$ . These equations can then be used to obtain the outlet concentration or fractional conversion.

## III. Equipment

A schematic diagram of the apparatus is shown in Figure R-9. The three stirred reactors each hold *ca.* 1.5 L of fluid, and are connected to form a gravity-driven cascade via overflow exit tubes. The tubular reactor has a volume of approximately 350 ml. [Note that these volumes are only rough estimates, and you are to determine more accurate values from your experimental results.] Temperatures of both reactor trains are controlled by recirculation of fluid from a constant temperature bath; the tubular reactor is immersed in the bath, and fluid from the bath is circulated through jackets surrounding the stirred tanks. The peristaltic pump feed line passes through heat exchange coils in a separate constant temperature bath so that the feed entering the reactors will be at the desired operating temperature.

A peristaltic pump is used to feed the NaOH solution or water to the reactors, while programmable syringe pumps are used to introduce the *MG* dye at the inlets to the reactors. In order to reduce pulsations in the flow, the peristaltic pump feed tubing is routed through two pump heads that have rotors aligned 45° out of phase. The feed line also passes through a pulse dampener. For the residence time distribution studies, the peristaltic pump feeds deionized water instead of the NaOH solution. The tracer dye is injected directly into the first tank of the CSTR

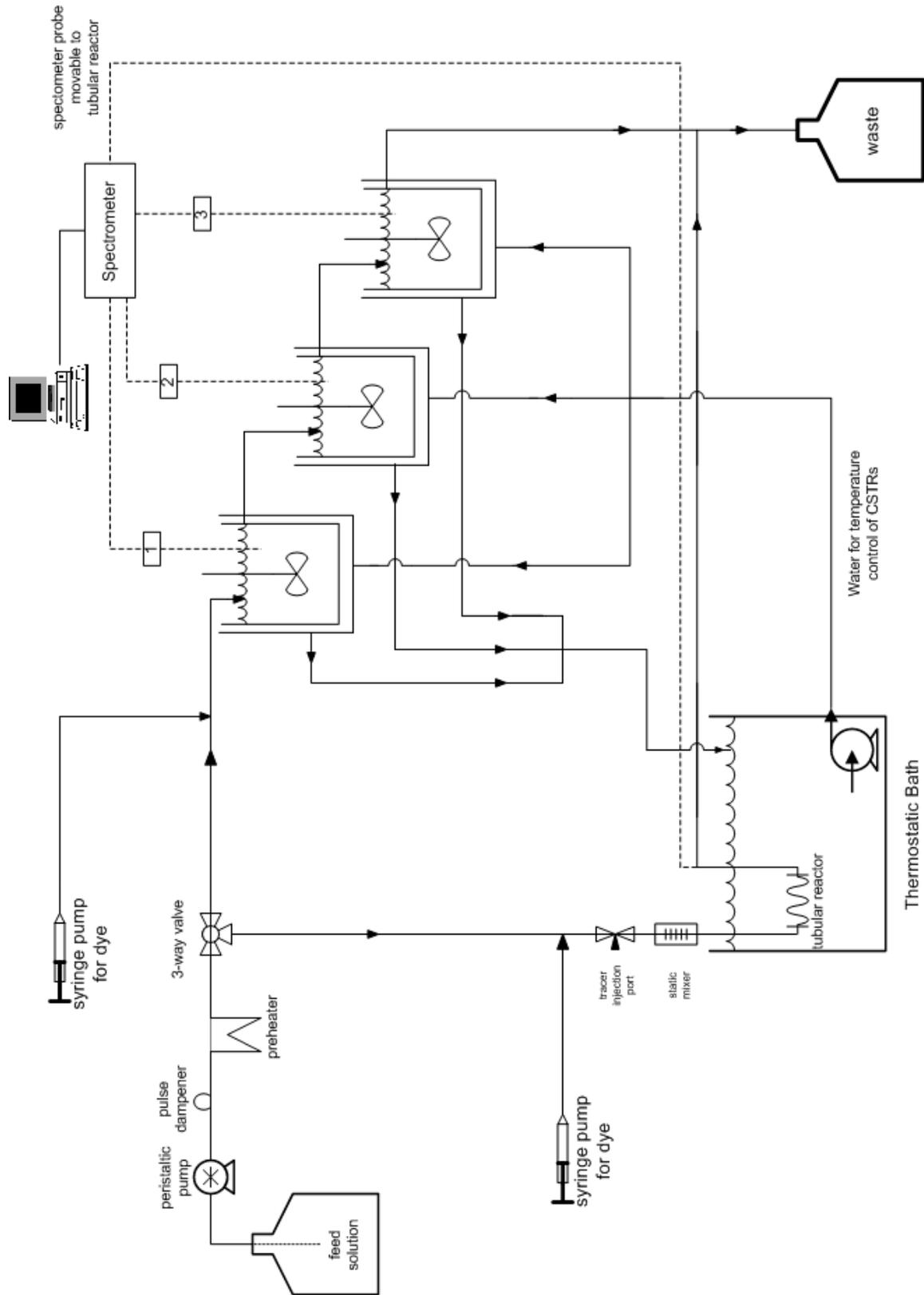


Figure R-9. Flow Diagram for CSTR and Tubular Reactors

cascade. A port is provided for injection of the tracer dye into the tubular reactor. The peristaltic pump motor has a digital display indicating the approximate flow rate through any of the three lines and pump heads. The NaOH/water lines are both size 17 tubing, so the digital readout is set at #17 to indicate the flow rates of these streams. The output of the pump is directed to the desired reactor through a three-way valve.

Each of the 3 tanks in the CSTR cascade is provided with a fiber-optic based transmittance/absorbance probe that can be used to continuously monitor the concentration of either the  $MG_q$  or any nonreacting tracer dye. This Ocean Optics spectrometer system includes three visible light sources and three diode-array detectors mounted in a rack and connected to the computer through a USB cable. Software provided by Ocean Optics (OOIBase 32) is used to calibrate the probes and to acquire the transient absorbance data. For the tubular reactor system, one of the spectrometer probes is inserted into a fixture at the exit of the reactor.

## IV. Experimental Procedures

Part A (below) should be conducted first. Subsequent parts may be conducted in any order, as indicated by your instructor. Note that reactor volumes given are only approximate, and the feed flow rate indications on the pump are also only approximate values. For both the RTD and reaction trials, you should collect and weigh samples at the reactor outlet to determine actual flow rates at each flow setting.

For the RTD measurements the supply carboy should be filled with deionized water. The NaOH solutions are also made up with deionized water. As a precaution, the liquids in the supply tanks should be degassed with helium to prevent problems with formation of oxygen bubbles on the spectrometer probes. Degas the liquid in the feed tank by bubbling helium through it for ca. 10 minutes before connecting the tank to the pump.

### A. *Familiarization with the equipment*

1. Group members should familiarize themselves with the arrangement of the equipment and with operation of the spectrometer system. You will need approximately one 20-liter carboy of water or NaOH solution for each run. A concentrated stock solution of 3.00 M NaOH is available so that dilution of 10 mL of stock sample into 20.00 kg of solution will give the proper concentration of NaOH (0.0015 M). A stock solution of the  $MG$  dye (0.0046M) is also provided. Set the constant-temperature baths at the reaction temperature indicated by your instructor.
2. Set up the spectrometer for absorbance measurements as follows:
  - a) Place the three spectrometer probes in a beaker of deionized water. Turn on the power to the visible light sources and flip the switch on each of the three light sources to the up (ON) position.
  - b) Open up the OOI software program by clicking on the desktop icon. The software should be in the "Scope" mode, indicating the number of detector counts at each wavelength. Click on the "Strobe/Lamp Enable" box on the toolbar to turn on the three light sources.

The integration time, *Integ. Time*, should be adjusted so that the maximum signal displayed is ca. 3500 counts (typically in the range of 4 msec). Set the *Average* and *Boxcar* parameter values to “3”. Leave the other settings at their default values. Because of slight differences in mirrors, fibers, and alignment, the three probes will exhibit different signal levels in the “Scope” mode. When calculating absorbance values the software will correct for these differences using the dark and reference spectra.

- c) Save a background dark spectrum, which is subtracted from all subsequent detector measurements. Turn off the light sources with the “Strobe/Lamp Enable” box. Save a dark spectrum by selecting the **Spectrum** and **Store Dark** commands. If any signal acquisition parameters (*Integ. time*, *Boxcar*, *Average*,...) are changed, or if the computer program is exited, this step must be repeated.
  - d) Prepare to take a reference spectrum by placing the probe(s) into the desired reactor(s) containing the proper fluid (water or NaOH solution) containing no dye. (For the initial familiarization experiments, just leave the probes in the beaker of deionized water.) Turn the light sources back on and let them warm up for at least 2 minutes before going to the next step.
  - e) Store a reference spectrum by selecting the **Spectrum** and **Store Reference** commands. Enter the absorbance mode by selecting the **Spectrum** and **Absorbance Mode** commands. (Alternatively, you may prefer to use the buttons on the toolbars that perform many of these tasks.) Near-zero absorbance readings on the real-time plot should be observed over the visible wavelength range. Scaling of the plot on the computer screen may be adjusted at any time using the **View** commands.
  - f) Absorbance data may be saved in Formatted Text files, Excel compatible, with the time acquisition commands. On the toolbar select the following commands: **Time Acquisition**, **Configure**, and **Configure Acquisition**. (The **Configure Time Channel** settings should be left at their default values.) Enter an appropriate file path and unique filename for each RTD or reaction run. The following boxes in the configuration window should be checked: *Streaming Data to disk*, *Show Values in Status Bar*, *Continue Until Manually Stopped*. The following parameter values should also be entered in this window: Initial Delay = 0, Frequency = 2 seconds. Close the configuration window when the proper parameters have been entered. Prepare the computer for automatic data acquisition by clicking on the clock icon in the toolbar. Make sure that the spectrometer is in the Absorbance mode. Start data acquisition by clicking on the green **Run** arrow. Select the red **Stop** button to end data acquisition. The data file may be viewed after the run is complete by opening it in an Excel spreadsheet (select Text Import Wizard defaults when opening data file in spreadsheet).
  - g) It is critical that the optical fibers are not bent excessively because they may break. The light signal is also adversely affected by tight bends.
3. An empty syringe in the syringe pump may be exchanged for a full syringe by disconnecting the needle and plastic tubing from the Luer fitting on the plastic syringe body. Twist the needle counterclockwise to remove it from the syringe body. Lift up and turn the spring-

loaded clamp that retains the syringe body in the pump in order to exchange syringes. The *MG* dye is not hazardous, but it is very tenacious and may permanently stain clothing with which it comes in contact. Promptly clean up any drops of dye that may spill on the benchtop or syringe pump.

## ***B. Reaction Kinetics in a Batch Reactor***

Use the first tank as a batch reactor. After the constant-temperature baths have reached their set point temperatures, set the NaOH pump to a flow rate of ca. 500 mL/min and fill the tank with the NaOH solution to a level about 3 cm below the overflow tube. Turn on the stirrer at a setting of “2”.

Prepare a syringe with 2 mL of the *MG* dye (0.0046M). Place a transmittance probe in the holder and set the reference absorbance level of the spectrophotometer by following the directions given above in parts A.2.d) & A.2.e). Follow the directions in part A.2.f) to start the data acquisition for the batch reactor. Shortly after data acquisition has started (after clicking on the green **Run** arrow on the toolbar), rapidly inject the dye near the center of the reactor with as little splashing as possible. Select the red **Stop** button to end data acquisition after the absorbance level decreases to a value less than 0.05 absorbance units.

Note that it is possible to conduct replicate trials by simultaneously using the other stirred tanks. Consult with your instructor to determine if replicate trials are to be conducted.

## ***C. Residence Time Measurements in Stirred Tank Reactors***

### **1. Well-mixed Tanks**

Drain the three tanks and then fill them with degassed deionized water using the peristaltic pump. Set the flow rate at 500 mL/min, and set the stirrer controls at “2”. Insert a transmittance probe into each tank and wait about 2 minutes before storing reference spectra for the three probes (see parts A.2.d) & e.). Enter a filename for the data acquisition and choose a sampling time of 2 seconds. Start the data acquisition software, and, after checking to see that the software is operating properly, rapidly inject 2 mL of the non-reactive tracer dye directly into the center of the first tank. Continue monitoring absorbance values until the values in all three tanks return to near their baseline levels.

Repeat the above process at other chosen flow rates (consult the instructor). Note that you may need to change the size of the injection if the signals go off scale or become too small to detect without significant noise.

### **2. Poorly-mixed Tanks**

Select a flow rate of 500 mL/min, and turn off the stirrer in the first tank before taking data, using the general procedures employed in the previous trial (part C.1).

## ***D. Reaction in Stirred Tank Reactors***

### **1. Determination of $C_{Ao}$**

Drain the reactor tanks and insert the pump inlet tube into a carboy of degassed deionized water. Start the pump at 500 mL/min and take reference spectra after all of the tanks are full. Then run the syringe pump at 50 mL/hr (with stirring) until a steady state absorbance is obtained in the first tank. This absorbance reading will be proportional to  $C_{Ao}$  for the 600:1 volumetric flow ratio for the reactant streams ( $\text{OH}^-$  and  $\text{MG}_q$ ).

### **2. Well-mixed Tanks**

Drain the reactor tanks and fill with 0.0015 M NaOH solution that has been degassed. Take reference spectra while the peristaltic pump is running at a flow rate of 500 mL/min. Set up the computer to record the transient approach to steady state and start the data acquisition. Start the syringe pump running at a flow rate of 50 mL/hour. As the reaction approaches steady state, check the temperature in each of the tanks with the thermistor probe provided. Watch the supply of NaOH solution and have a new carboy of degassed solution available for a rapid changeover if needed. Check the calibration of the pump by capturing some of the effluent from the third tank.

Repeat the above procedure for additional flow rates (consult the instructor). The flow rate on the syringe pump must be changed in order to maintain the reactant concentration ratio. Be sure to watch the amount of reactant dye available in the syringe so that a filled syringe may be inserted at a non-critical time.

### **2. Poorly-mixed Tanks**

Return to the poorly-mixed conditions of Part C.2 and obtain steady-state concentrations in the tanks for the 500 mL/min flow rate used above in the RTD experiment.

## ***E. Residence Time Distribution Measurements for a Tubular Reactor***

Use the 3-way valve to switch to the tubular reactor and obtain RTD data for several different flow rates, using the techniques described in section C.1. Before acquiring data, be certain to measure a new reference spectrum for the probe that is inserted into the fixture at the reactor exit. Since the RTD for this reactor is much narrower, you may wish to inject a smaller amount of tracer (0.20 ml is a suggestion). Note that the peak height now varies strongly with flow rate (dilution). Thus you may need to change the size of the injection when different flow rates are employed if the signals go off scale or become so small that the signal to noise ratio is not adequate. In addition, select a data sampling time interval of 1 sec or shorter for the tubular reactor RTD measurements.

## ***F. Reaction in the Tubular Reactor***

Obtain absorbance data for the effluent stream for same range of flow rates employed in the RTD trials, always maintaining an  $\text{OH}^-:\text{MG}_q$  volumetric ratio of 600 :1. Note how long it

typically takes for a new steady-state outlet concentration to be established after each change in flow rate. Again, check the observed flow rates to ascertain the calibration of the pump.

## V. Contents of the Report

### A. Batch Reaction

Analyze the kinetic data to determine whether the reaction can be treated as pseudo first-order in *MG*. Comment on any approximations involved in this treatment and justify their use. Report the rate constant for your conditions. What are the uncertainty limits on this parameter?

### B. Stirred-Tank Reactors

1. Analyze your RTD data. Note that you should be able to determine the RTD for systems comprised of 1, 2, and 3 stirred tanks. Determine the mean residence time for each flow rate. Report the corresponding volume you measure for the tanks. Fit the RTD data to a simple multi-tank model and comment on the quality of the fit. Consider the behavior seen at different flow rates. If these are well-mixed tanks, when the RTD curves for different runs are plotted against  $t/\tau$  they should be superimposed on one other. Is this observed? Prepare plots of  $F(t)$  versus  $(t/\tau_{\text{cascade}})$  based on the experimental data and compare these curves with the ideal curves for the corresponding number of reactors in the cascade.
2. Compare the effluent conversions observed leaving each of the three tanks at different flow rates to the predictions of the model for CSTRs in series. Show this on a plot of  $f$  vs.  $\tau$ , indicating the actual data points and showing their positions relative to the curves indicating the predictions based on the batch reactor study and the design equations for CSTRs in series.
3. Use the segregated flow approximation to predict the conversions for the actual residence time distributions reported here. Compare these results with those from the ideal CSTR cascade model in a table and also on a square plot with the actual fractional conversions on the x-axis and the predictions (ideal CSTR and Segregated Flow) on the y-axis.
4. Repeat this analysis for the runs conducted with poor stirring. Comment.

### C. Tubular Reactor

1. Analyze your RTD data. Report the mean residence time for each flow rate, and determine the actual volume of the tube. Fit the RTD data to a simple model for a Plug-Flow Reactor and also to a more accurate model (Laminar Flow Reactor, Axial Dispersion, or n-CSTR). Comment on the quality of the fits. Do the RTD curves for different runs overlay one other when plotted against  $t/\tau$ ? What does this indicate?
2. Compare the conversions observed in the tubular reactor at different flow rates to those predicted for an ideal PFR. Show this on a plot of  $f$  vs.  $\tau$ , indicating the actual data points

and showing their positions relative to a curve indicating the values predicted using the results of the batch reactor study and the design equation for a PFR.

3. Use the segregated flow approximation to predict conversions based on the measured residence time distributions. Compare these results with those based on the ideal PFR model. Show this comparison in a table, and also on a square plot with the actual fractional conversions on the x-axis and the predicted values (ideal PFR and Segregated Flow) on the y-axis.

#### ***D. Transient Behavior***

In the trials with the various flow reactors, different times were required to reach steady state each time the feed flow rate was changed. Comment on the time scales of the transients, relative to the reactor residence times and distributions. For the tubular reactor, did the transients depend on the direction of the change in the flow rate, or just the initial or final flow rate?

#### ***E. Other***

Comment on other conclusions drawn during this experiment, as appropriate. Can you draw any conclusions about the calibration of the peristaltic pump, the constancy of the mixing ratio of *MG* and NaOH, etc.? In addition, make any suggestions you have for other experiments that would extend the characterization of these reactors, improve their operation, or introduce students to new concepts.

#### ***F. Appendix and Sample Calculations***

Present a sample calculation for each of the items requested in parts A, B, and C of section V. Present the governing equations before substituting the appropriate numerical values. Include one sample calculation for the mean residence time of each type of reactor, including numerical values. Do not print out the RTD spreadsheets for inclusion in the report. Instead, have one group member (the leader on this experiment) email the spreadsheets to the instructor if requested. Do not re-derive any of the working equations presented here or in the pre-lab briefing unless you intend to show a correction or different result.

#### **Reference**

Chen, D.T. and Laidler, K.J., *Can. J. Chem.*, **37**, 599 (1959)