An-Najah National University

Department of Chemical Engineering

(Nablus/Palestine)

Unit Operation Lab, 64468

Instructor:

Dr.Husni Odeh

Eng. Maryam Hmoudah

January, 2011

An-Najah National University
Chemical Engineering Department
Course outline

Course title and number	Unit Laboratory 64468
Semester and year	Second sem. Academic year: 2010/2011
prerequisite	Unit operation
Recent textbook	**Laboratory manual**,
Suggested references	• McCabe, Smith and Harriott, Unit operations, 3rd Edition • C.J. Geankoplis, Transport processes and unit operation, 3rd Edition. • Robert E.Treybal, 3rd Edition, Masstransfer operations McGraw-Hill Book Company ISBN 0-07-065176-0 ,
Course description	The primary objectives of this class are to improve your problem-solving and self-directed learning skills coupled with an ability to work in teams. The data each team obtains, analyzes, and reports must be objectively considered and applied properly. The laboratory project should be motivated by a practical problem and must clearly demonstrate how the findings address the larger system and/or a final product. Again, problem-solving, self-directed learning, and the ability to work in teams are vital for success in professional and personal life. *Unit Ops Lab is about teaching skills as much as content.* The students carrying out complex experiments on pilot plant systems. This course provides good oppurtunity of training on the main industrial operations: distillation, absorption, liq/liq extraction. And evaporation
Instructor	Dr. Husni ODEH, E-mail: hmodeh@najah.edu
Course type	compulsory,
Course level:	4th year and 5th year students

Course content and teaching time table

	Topics	
	Gas absorption measurements: 1. Hydrodynamics measurements on packed column, first by dry column then with water/air system 2. Mass transfer measurement with carbondioxide/water system. Analysis on gas side by using gas analysis equipment provided 3. Calculation rate of absorption of carbon dioxide into water from analysis of liquid	

	solution 4. Calculation rate of absorption of carbon dioxide into caustic soda solution from analysis of liquid solution flowing down absorption column 5. Showing the amount of carbon dioxide removed from the air stream equals the amount taken by caustic soda solution. And Mass transfer measurement with carbondioxide/water system. Analysis on liquid side by using gas analysis equipment provided	
	Liquid/liquid extraction Experiments: 1. Determination of distribution coeffient 2. Basic operation of liquid /liquid absorption colpumn. 3. Overall mass balance and mass transfer coefficient with the aqueous phase as the continuous medium 4. Overall mass balance and mass transfer coefficient with the aqueous phase as the continuous medium and Determination of solvent recovery	
	Experiments on distillation unit: **Batch distillation:** 1. Determination the pressure drop over the distillation column for various boil-up rates. 2. Using refractometer for determening mixture composition 3. Determination overall column efficiency 4. Distillation at constant reflux ratio, variation of top and bottom composition with time 5. Steady state distillation of binary mixture , McCabe- Thiele/Ponchon Sarvorit determination no of theoretical plates. 6. Effect on column performance of feed plate location	
	Evaporation Demoneatration and operating of the evaporator and steam generator	
	Evaporation experiment, by deionized water	
	Final examination	**1**

		16

Intended learning outcomes of this lab credit:

1. The improvement of students problem-solving and self-directed learning skills coupled with an ability to work in teams.
2. The student will understan in depth the leared theoretical material relevant to the subject.
3. Each team of student will demonestrate on the existed an experimental system a complete process with its operational parameters
4. The student will practice writing complex experimental report with it all components (Abstract, objectives, prosedure,…)
5. The data each team obtains, analyzes, and reports must be objectively considered and applied properly.
6. Again, problem-solving, self-directed learning, and the ability to work in teams are vital for success in professional and personal life. Unit Ops Lab is about teaching skills as much as content.

7. The students will be able to operate independently
8. The students carrying out complex experiments on pilot plant systems. This course provides good oppurtunity of training on the main industrial operations: distillation, absorption, liq/liq extraction.

Grading:

Reports:	35%
Quizes and performance	10%
Mid exam	20%
Final Exam	35%

Dishonesty

Students are expected to be honest and ethical in their academic work. Academic dishonesty is defined as an intentional act of deception in one of the following areas:

- Cheating- use or attempted use of unauthorized materials, information or study aids, especially during exam writing.

When evidence of academic dishonesty comes to the instructor's attention, the instructor will document the incid permit the accused student to provide an explanation, advise the student of possible penalties, and take action. instructor may impose any academic penalty up to and including an "F" grade in the course after consulting with head department and informing the student of the action taken

An-Najah National University
Department of Chemical Engineering
Unit Operation Lab, 64468.
Second term 2011

Instructions for Preparing Laboratory Reports

The report must be prepared according to the following outline.

1. Title Page

The Title page should be separate from the rest of the report. It should contain:
a. The title of the experiment.
b. The number of course.
c. The names of the writer and his co-worker(s), their ID numbers, and group number.
d. Name of the instructor to whom the report is submitted
e. The date when the experiment was run
f. The date of submission of report
A sample title page is shown on page xv, which can be used when submitting reports.

2. Abstract

The abstract should be informative, and should be written in about three to five sentences. It should cover all phases of the investigation. It must include the following:
a. An introductory statement about the subject matter
b. Brief description of what was done and how it was done.
c. Selected results (numerical values, if available).
d. Brief assessment of the results e.g. some percentage errors in
Experimental results in comparison with theoretical values.
While writing the abstract, no reference should be made to graphs, tables or equations inside the report.

3. Introduction

This section should include few sentences discussing the physical and/or chemical principles involved in the experiment. The importance and relevance of the experiment to real applications may also be stated.

4. Theoretical Background

This section should include the theory behind the experiment. It should also contain all those equations, which are used to acquire a certain result. Theoretical correlations, which are used for comparison with experimental results, should also be included.

5. Procedure

Here, you should briefly describe the actual step-by-step procedure you followed in running the experiment. It should be written in your own words, e.g. the needle valve was manipulated in order to adjust the liquid flow rate.

6. Results

The results should be presented in the form or Tables or graphs. The Table should contain the results obtained from experiments and from theoretical knowledge.

Comparisons may also be presented in terms of percent deviation between experimental values and theoretical predictions.

7. Discussion of Results

In this section you should discuss your experimental results and observations. If the results are obtained in terms of graphs, then interpret them also. Describe observed trends and possible relationship between parameters e.g. how a change in one variable affects another. Also show how you make comparison with the values obtained theoretically and discuss the deviation of experimental results from theoretical values. The possible source of errors should also be mentioned.

8. Conclusions and Recommendations

Conclusions are the series of numbered sentences which answer the questions posed at the beginning of each experiments. Conclusions should also include main results (numerical values) and the errors between the experimental and theoretical values. What you have learned from the experiment should be mentioned as well.

Recommendations are the proposals for future work, e.g. suggested changes in equipment, Study of new variables, or possible experiments in relative fields. Like the conclusions, the recommendations are usually listed by numbers, and each consists of only a sentence or two.

9. Literature Cited

Here, you should list the books, Journal's articles, etc. used in writing your report and analyzing the experiment. The reference should be completed (name of the book, author, volume, date of publication, pages, etc.). References should be arranged alphabetically by author's names.

10. Nomenclature

The symbols, which are used in the report, should be defined in the nomenclature in alphabetical order. The accompanying definitions must include proper units.

Appendices

All appendices and graphs should be attached at the end of the report.

A1. Raw Data:

It should contain the raw data (in the log sheet) collected during the experiment.

A2. Analysis of Data and Sample Calculations:

This shows how the collected experimental data are analyzed and transformed into experimental results by using the appropriate equations. Also, how the theoretical results are obtained using theoretical in terms of percentage error. Sample calculations should contain each step, which is used to acquire certain results.

Organization and Neatness

The students must organize their reports in accordance with the format described earlier. It is required/encouraged to use computer packages such as EXCEL, SIGMAPLOT, HARVARD, GRAPHICS or any other suitable engineering software to draw graphs. The neatness will include how the student has organized his report, neat handwriting, accuracy in grammar and spelling, numbering pages, figures, tables and equations. Additional points will be given to those students who use computers in preparing their reports.

Sample Cover Page

Unit Operation Laboratory

Batch Distillation

(Experiment no. 1, Performed on: February 20, 2008)

Prepared by: Naser Jaber

Group members: N. Jaber
A. Daqa √
M. Badran √

Submitted to: Dr. Husni Odeh

Chemical Engineering Department

An-Najah National University

February 26, 2009

PART 1

Gas Absorption Column UOP7

SAFETY IN THE USE OF EQUIPMENT SUPPLIED BY ARMFIELD

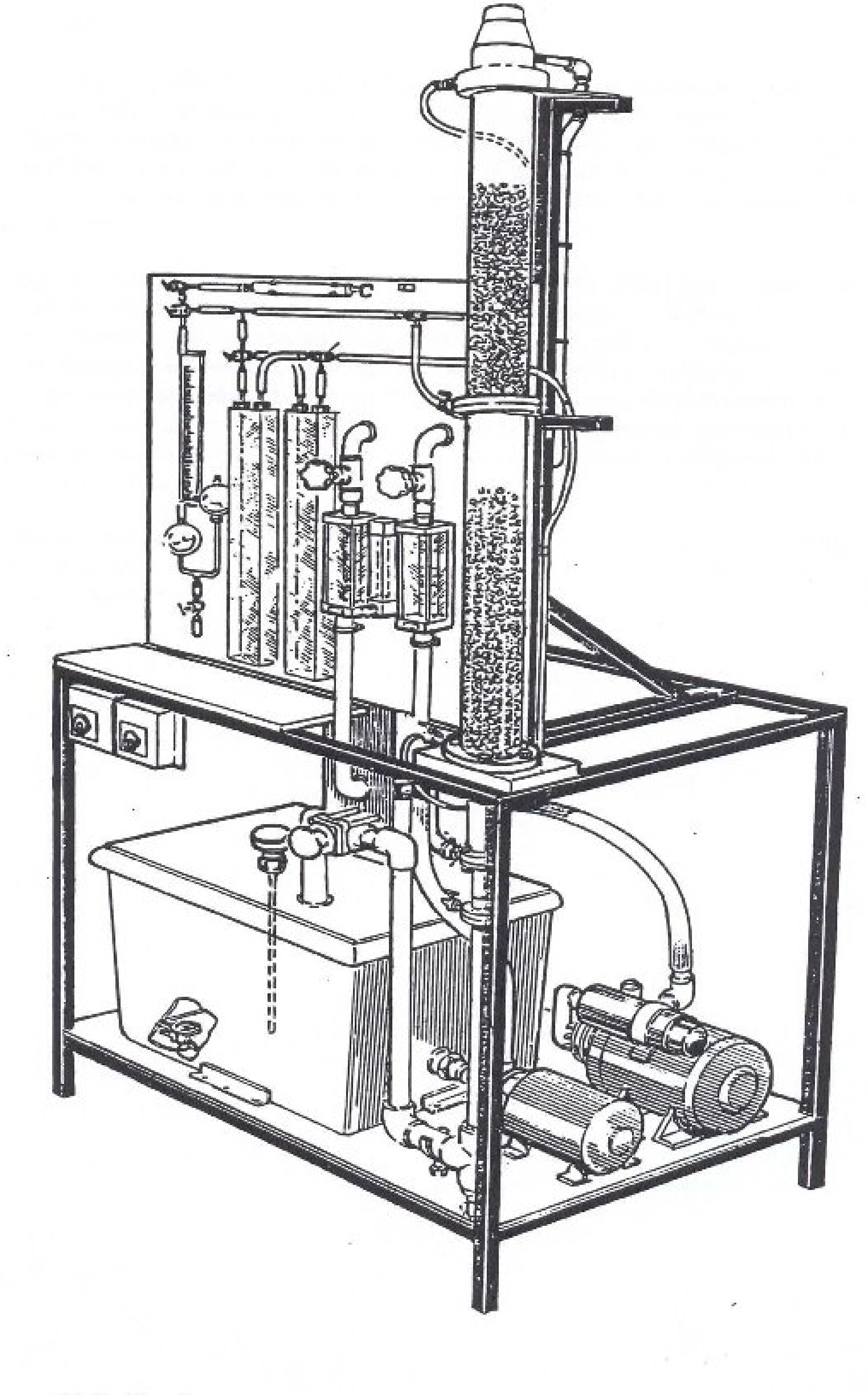
Before proceeding to install, commission or operate the equipment described in this instruction manual we wish to alert you to potential hazards so that they may be avoided.

Although designed for safe operation, any laboratory equipment may involve processes or procedures which are potentially hazardous. The major potential hazards associated with this particular equipment are listed below.

- INJURY FROM ELECTRIC SHOCK
- INJURY THROUGH MISUSE
- INJURY FROM INCORRECT HANDLING
- DAMAGE TO CLOTHING
- INJURY FROM CORROSIVE FLUIDS
- RISK OF INFECTION DUE TO LACK OF CLEANLINESS

Accidents can be avoided provided that equipment is **regularly maintained** and **staff** and **students** are made aware of potential hazards. A list of general safety rules is included in this manual, to assist **staff** and **students** in this regard. The list is not intended to be fully comprehensive but for guidance only.

Please refer to the notes overleaf regarding the Control of Substances Hazardous to Health Regulations.



UOP 7 GAS ABSORPTION COLUMN.

INTRODUCTION

The packed tower, in which two fluids flowing in opposite directions enable a chemical component to be transferred from one fluid phase to the other, occurs in almost all chemical plants. The process may be gas absorption, distillation, solvent extraction or chemical reaction. A knowledge of the characteristics of both fluid flow and of mass transfer in such towers is necessary for both plant operators and designers.

The Armfield Gas Absorption Apparatus has been designed to allow these studies to be made, and the instrumentation and layout enables students to follow both the hydrodynamic characteristics in the absence of mass transfer, and also, separately, to advise the performance of the mass transfer process involved in gas absorption. The size of the equipment has been chosen so that experiments may be completed in a typical laboratory class period, while at the same time being capable of demonstrating full-scale plant behaviour. Considerable attention is directed towards matters of safety which is of crucial importance in the process industries.

This instruction manual gives details of all the laboratory services required for operation of the column, together with commissioning procedures and instructions for the many experiments possible with the equipment.

DESCRIPTION

The equipment consists of a 75mm diameter column in which there are two lengths of Raschig ring packing material. Pressure tappings are provided at the base, centre and top of the column to determine pressure drops across the column. Sampling points are also provided for the gas at the same three points. The liquid outlet stream and feed solution are also equipped with sampling points. Suitable manometric measurement is included. Water is taken from a sump tank, and pumped to the column via a calibrated flowmeter. Gas is taken from a pressure cylinder (not supplied) through a calibrated flowmeter, and mixed with air supplied and monitored from a small compressor in a pre-determined (but variable) mixed ratio. The mixture is fed to the base of the tower, in which a liquid seal is provided. The effluent gas leaves the top of the column and is intended to be exhausted to atmosphere outside the laboratory building.

The apparatus is designed to absorb carbon dioxide/air mixture into an aqueous solution flowing down the column. Gas analysis apparatus is provided for this system.

Diluted ammonia may be used as an alternative to carbon dioxide but this is not recommended unless safe operating/disposal procedures are adopted.

COMMISSIONING

All numerical references relate to the diagram on page 10.

1. Fill sump tank (1) with clean water.
2. Connect the electrical supply cable to the appropriate mains supply.
3. Prime the water manometers (3) with clean water until the meniscus in both tubes is at mid height (500mm on the scale).
4. Fill the Hempl gas analysis apparatus (5) with water up to the '0' mark on the scale.
5. Connect a CO_2 gas cylinder, fitted with a regulator, to the inlet regulator on the equipment with a flexible tube, and set the cylinder regulator to minimum pressure.
6. Open fully the gas flow control valve (10) on the gas flowmeter (small, centre flowmeter) and open the main gas cylinder valve.
7. Increase the cylinder regulator output pressure to give maximum flow on the gas flowmeter and close the main cylinder valve.
8. Close the air (6) and water (8) flow control valves. Close the gas sampling cocks (7, 11) on the absorption column. Check that the valve (2) in the discharge pipe into the sump tank is fully open. Switch on the Water pump (13) and check that water flow is obtained through the flowmeter (9) and down the column on opening the control valve (8).
9. Switch on the air compressor (12) and check that airflow is obtained through the flowmeter (4) and up the column on opening the control valve (6).
10. Check that the two water manometers (3) indicate the pressure drop across the column (connecting valves above the manometers must be correctly set. Refer to the diagram on page G-1 for details).
11. Check that the Hempl gas analysis apparatus (5) operates correctly by following the instructions in the appropriate experiment sheet.
12. Close the flow control valves and switch off the pumps.
13. Drain the Hempl apparatus.
14. Drain the water seal with the small drain cock (14) at the bottom of the 'U' beneath the column.

The equipment is now ready for use.

LIST OF GAS/LIQUID ABSORPTION EXPERIMENTS

I. **Hydrodynamic Measurements of packed column:**
To determine the air pressure differential across the dry column and wetted column as a function of the air flow rate .

II. **Physical Absorption** :
To measure the absorption of carbon dioxide into water flowing down the tower, using the gas analysis equipment provided .

III. **Physical Absorption:**
To calculate rate of carbon dioxide into water from analysis of liquid solutions flowing down absorption column .

IV. **Chemical Absorption:**
To calculate rate of absorption of carbon dioxide into caustic soda solutions from analysis of liquid solutions flowing down absorption column

V. **Chemical Absorption:**
To show that the amount of carbon dioxide removed from the air stream equals the amount taken up a liquid stream of caustic soda solution.

VI. **Chemical Absorption:**
Determination of Overall Mass Transfer Coefficient (K)

NOMENCLATURE

a = Effective interfacial area per unit packed column (cm^{-1})

A = Cross sectional area of the tower (m^2)

C_c = Concentration of sodium hydroxide (g.moles/litre)

C_d = Concentration of dissolved 'free carbon dioxide' (g.moles/litre)

C_N = Concentration of sodium carbonate ions (g.moles/litre)

F = Flow (litres/second)

G = Gas flow rate (g.moles/second)

K_{og} = Gas-side mass transfer coefficient (g.moles/second.cm^2.atm)

L = Liquid flow (litre/second)

M = Molecular weight

P = Pressure (atm)

R = Mass transfer rate (g.moles/cm^2.second)

T = Standard acid titration volume (ml)

V_B = Volume of alkali solution added in liquid analysis (ml)

V_1 = Volume of gas sample taken in Hempl apparatus (ml)

V_2 = Corresponds to amount of gas absorbed in Hempl apparatus (ml)

X = Mole fraction of component in liquid phase

Y = Mole fraction of component in gas phase

Subscripts

T = Total

i = Inlet conditions to column

o = Outlet conditions to column

N = Rate of absorption (g.moles/second)

Hydrodynamic Measurements of packed column

Experiment I

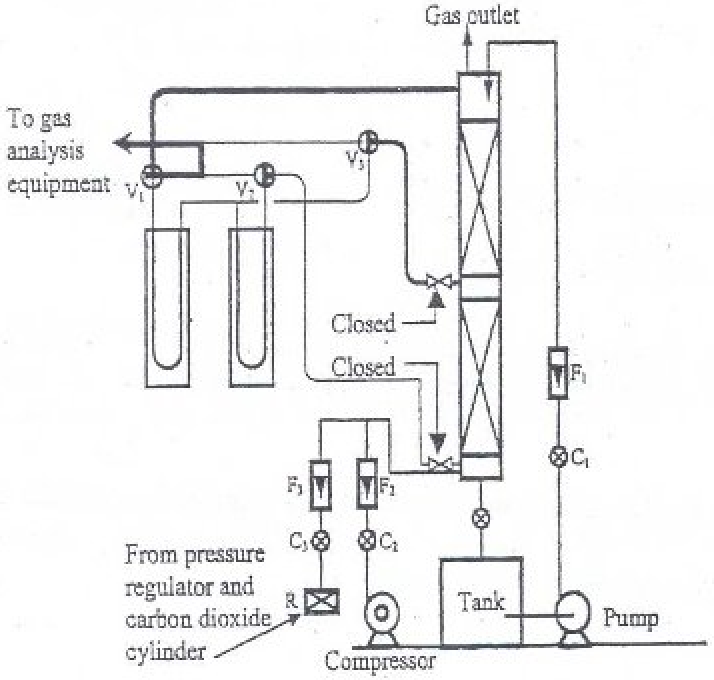
Gas absorption/ separation process

OBJECT OF EXPERIMENT:

To determine the air pressure differential across the dry column as a function of the air flow rate.
To examine the air pressure differential across the column as a function of air
Flow rate for different water flow rates on the column.

EQUIPMENT SET-UP:

Absorption column



PROCEDURE:

1. Fill the water reservoir tank to three-quarters full with tap water. Set valves V_1, V_2 and V_3 as shown on the diagram so that differential pressures in the top and bottom sections of the column are indicated on the two water manometers.

2. Switch on the water pump and set C_1 to give a flow rate of say 3 litres/minute down the column.

3. After about 30 seconds close C_1, switch off the pump and allow the column to drain for 5 minutes.

4. Measure the air pressure differential across the wet column as a function of the air flow rate.

5. Measure the air pressure differential across the column as a function of the air flow rate for different water flow rates up to say 5 litres/minute, noting the appearance of the column at each setting.

READINGS TO BE TAKEN:

Pressure Differential (mm water)

Air flow l/m Water flow l/m	20												200
0													
1.0													
2.0													
2.5													
3.0													
3.5													
4.0													

The range of possible air flow rates will decrease with increasing water flow rate due to onset of 'flooding' of the column, which should be noted.

RESULTS:

Pressure differential should be plotted as a function of air flow rate on log-log graph paper for each water flow rate. It may be necessary then, to take more readings in order to define precisely the transition points on the resulting graphs.

Physical Absorption

Experiment II

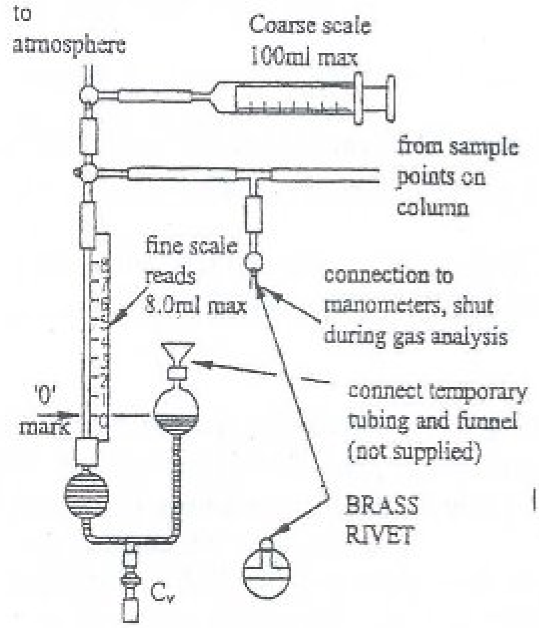
Gas absorption/ separation process

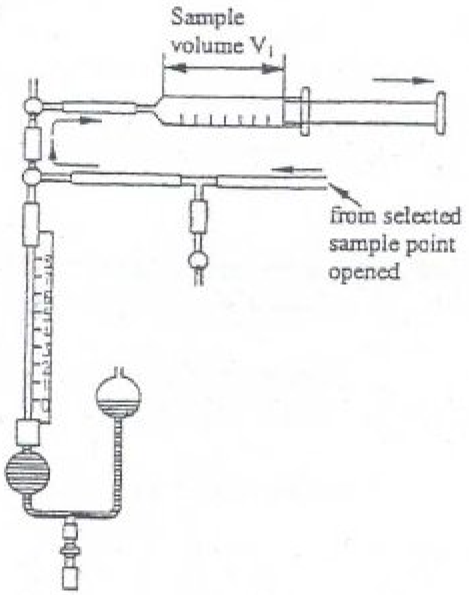
OBJECT OF EXPERIMENT:

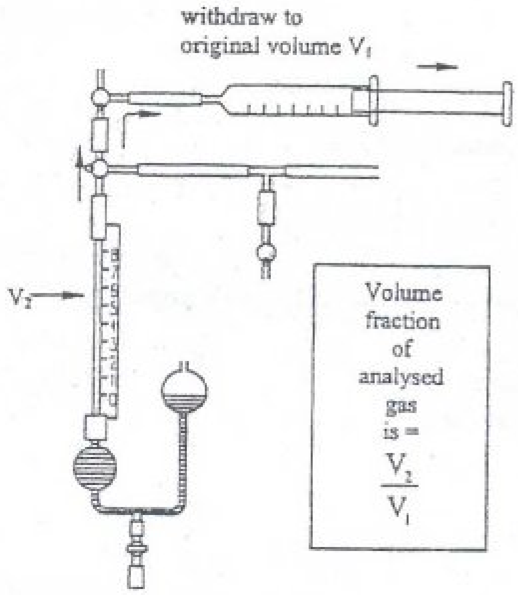
To measure the absorption of carbon dioxide into water flowing down the tower, using the gas analysis equipment provided /Hemple apparatus/.

Hempl apparatus

Hemple gas analyzer







Equipment Required:

1. Carbon dioxide cylinder with integral pressure regulator, connected to regulator R on gas inlet on the apparatus.

2. Approximately 300ml of 1.0 molar caustic soda solution with safety gloves and goggles. small funnel and tubing for filling analysis equipment .

PROCEDURE:

1. First fill the two globes of the absorption analysis equipment on the left of the panel with 1.0 Molar caustic soda. Wear gloves and goggles while doing this. Adjust the level in the globes to the '0' mark on the sight tube, using drain valve C_V into a flask to do this.

2. Fill the liquid reservoir tank to three quarters full with fresh tap water .

3. With gas flow control valve C_2 and C_3 closed, start the liquid pump and adjust the water flow through the column to approximately 6 litres / minute on flow meter F_1 by adjusting control valve C_1 .
4. Start the compressor and adjust control valve C_2 to give an airflow of approximately 30 litres /minute in flow meter F_2 .

5. Carefully open the pressure regulating valve on the carbon dioxide cylinder, and adjust valve C_3 to give a value on the flow meter F_3 approximately one half of the air flow F_2. Ensure the liquid seal at the base of the absorption column is maintained by, if necessary, adjustment of control valve C_4

6. After 15 minutes or so of steady operation, take samples of gas from the top and middle sample points. (Refer to the appropriate diagram for details of the valve positions.) Analyse these consecutively for carbon dioxide content in these gas samples as shown in the accompanying sketch and following notes .

7. Flush the sample lines by repeated sucking from the line, using the gas piston and expelling the contents of the cylinder to atmosphere. Note that the volume of the cylinder is about 100_{cc}. Estimate the volume of the tube leading to the device. Then decide how many times you need to suck and expel.

8. With the absorption globe isolated and the vent to atmosphere closed, fill the cylinder form the selected line by drawing the piston out slowly . Note volume taken into cylinder V_1, which should be approximately 20ml for this particular experiment (see WARNING note below) . Wait at least two minutes to allow the gas to come to the temperature of the cylinder.

9. Isolate the cylinder from the column and the absorption globe and vent the cylinder to atmospheric pressure. Close after about 10 second .

10. Connect cylinder to absorption globe. The liquid level should not change. If it does change, briefly open to atmosphere again.

11. Wait until the level in the indicator tube is on zero showing that the pressure in the cylinder is atmosphere.

12. Slowly close the piston to empty the cylinder into the absorption globe. Slowly draw the piston out again.

Note the level in the indicator tube.

Repeat steps E and F until no significant change in level occurs. Read the indicator tube marking = V. This represents the volume of the gas sampled.

WARNING: If the concentration of CO_2 in the gas sampled is greater than 8, it is possible to suck liquid into the cylinder. This will ruin your experiment and takes time to correct. Under these circumstances, do not pull the piston out to the end of its travel. Stop it at a particular Mark, eg .$V_1 = 20$ on the coarse scale, and read the fine scale.

READINGS AND CALCULATIONS:

A) CO_2 content of gas samples:

From use of Hempl apparatus, volume fraction of $CO_2 = V_2/V_1$

For ideal gases, volume fraction = mole fraction = Y. Check that the sample taken from the inlet to the absorption column should give the same value of CO_2 fraction as that indicated by the inlet flow meters.

Ie. $V_2/V_1 = Y_1 = F_3 / F_2 + F_3$

READINGS AT INLET				CALCULATIONS	
$F_1(CO_2)$liters /s	F_2 (air) litres/ s	V_1 ml	V_2 ml	$F_3 / F_2 + F_3$	$(V_2 / V_1) = Y_1$
From flow meters		From Hempl apparatus			

B) Calculation of amount of CO_2 absorbed in column from analysis of samples at inlet and outlet

From analysis with Hemple apparatus, volume fraction of CO_2 in gas stream at inlet = $(V_2/V_1)_I = Y_I$

And at outlet, $Y_0 = (V_2 / V_1)_0$

If F_a is litres / second of CO_2 absorbed between top and bottom, then: -

$[F_2 + F_3]\, Y_I - [F_2 + (F_3 - F_a)]\, Y_0 = F_a$

$F_a = (Y_I - Y_0)(F_2 + F_3)/1-Y_0 = (Y_I - Y_0)/(1-Y_0)$ * (total gas inlet flow)

INLET CONDITIONS				OUTLET	ABSORBED CO_2 : F_a litres/ sec
Air F_2	CO_2 F_3	TOTAL $F_2 + F_3$	GAS SAMPLE $Y_I = (V_2/V_1)_I$	GAS SAMPLE $Y_0 = (V_2/V_1)_0$	

Note: Litres / second can be converted to g. moles / second as follows: -

$G_a = F_a / 22.42$ * (av.column pressure mmHg /760)*(273/av.column temp °C + 273)
Absorbed CO_2 ______________

g.moles/second________________

The assumption implicitly made here is that the volume flow is not affected by the pressure drop through the column as this should be small in comparison with atmospheric pressure.

Physical Absorption

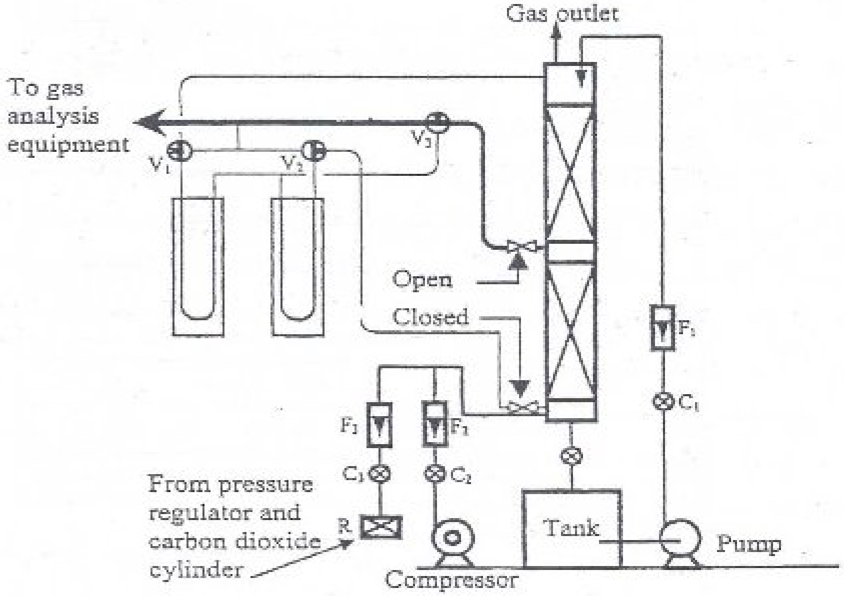
EXPERIMENT III

Gas absorption/ separation process

OBJECT OF EXPERIMENT:

To calculate rate of absorption of carbon dioxide into water from analysis of liquid solution flowing down absorption column.

EQUIPMENT SET-UP:



PROCEDURE:

1.Fill the liquid reservoir tank at the base of the column to approximately three-quarters full with (preferably) deionised water. Note the volume added [V_T litres] .

2. With gas flow control valves C_2 and C_3 closed, start the liquid pump and adjust the water flow through the column to approx. 6 litres/minute on flow meter F_1 by adjusting flow control valve C_1.

3. Start the compressor and adjust control valve C_2 to give an air flow of approx. 10% of full scale on flow meter F_2.

4. Carefully open the pressure regulating valve on the carbon dioxide cylinder, and adjust valve C_3 to give a value on the flow meter F_3 approx. one half of the air flow F_2 Ensure the liquid seal at the base of the absorption column is maintained by, if necessary, adjustment of control valve C_4.

5. After 15 minutes of steady operation, take samples at 10 minute intervals from S_4 and S_5. Take 150ml samples at known times in each case. Analyse the samples according to the procedure detailed below.

Analysis of Carbon Dioxide Dissolved in Water

Note: Water used for absorption should be deionised as presence of dissolved salts affect the analysis described below. If tap water is used, no metal ions should be present in greater quantities than 1.5 mg/litre and pH should be just alkaline: 7.1 to 7.8.
Chemical Solutions Needed

a) Phenolphthalein indicator prepared from carbon dioxide – free distilled water ie . A. R. grade.

b) Standard 0.0277M sodium hydroxide solution prepared by diluting 27.70ml 1M caustic soda standard solution to 1 litre with carbon dioxide free distilled water. Prepare daily and protect from carbon dioxide in the atmosphere by keeping in a stoppered Pyrex bottle.

c) Standard 0.01M sodium bicarbonate solution, prepared by dissolving approximately 0.1 gram of anhydrous sodium bicarbonate in carbon dioxide free distilled water to 100ml.

Procedure

1.Withdraw a sample of liquid S_5 from the sump tank with the sampler provided, approximately volume of 150ml, or from liquid outflow point S_4.

2. Discharge the sample at the base of a 100ml graduated cylinder, flicking the cylinder to throw off excess liquid above the 100ml mark.

3. Added 5-10 drops of phenolphthalein indicator solution:

a) Above; if the sample turns red immediately , no free CO_2 is present . If the sample remains colorless, titrate with standard alkali solution .
b) above Stair 30 second . This color changed is the end point – note volume V_B of alkali solution added.

For best results, use a color comparison standard, prepared by adding the identical volume of phenolphthalein solution a) to 100ml of sodium bicarbonate solution c) in a similar graduated cylinder.

Calculation

The amount of free CO_2 in the water sample is calculated from:

g.mole/litre of free $CO_2 = V_B * 0.0277/\text{ml.of sample} = C_d$

Note: Solubility of CO_2 in water is a strong function of temperature.

The accuracy of this titrimetric method is approximately 10%.

READINGS TO BE TAKEN:

F_1: ______________ litres/second

V_T: ______________ volume of water in system (litres)

TIME FROM START (minutes)	FROM SUMP TANK S_5 (Correspond to conditions at top of tower)		FROM LIQUID OUTLET SAMPLE POINT S_4	
	V_B ml	C_d in tank $[C_{di}]$ g mole /litre	V_B ml	C_d in tank $[C_{d0}]$ g mole/litre
10				
20				
30				
40				
50				
60				

CALCULATIONS:

A. CO_2 absorbed over a time period (eg. 30 mins): -

Average rate = $[C_{di}(t=40) - C_{di}(t=10)]$ **V_T/30** * 60 g.mole/second

B. CO_2 absorbed across the column at any particular time: -

Inlet flow of dissolved CO_2 $= F_{1=}.C_{di}$ g.mole/second

Outlet flow of dissolved $CO_2 = F_1 . C_{di}$ g.mole/second

Ie. Absorption rate $= F_1[C_{di} - C_{d0}]$ g.mole/second

Chemical Absorption

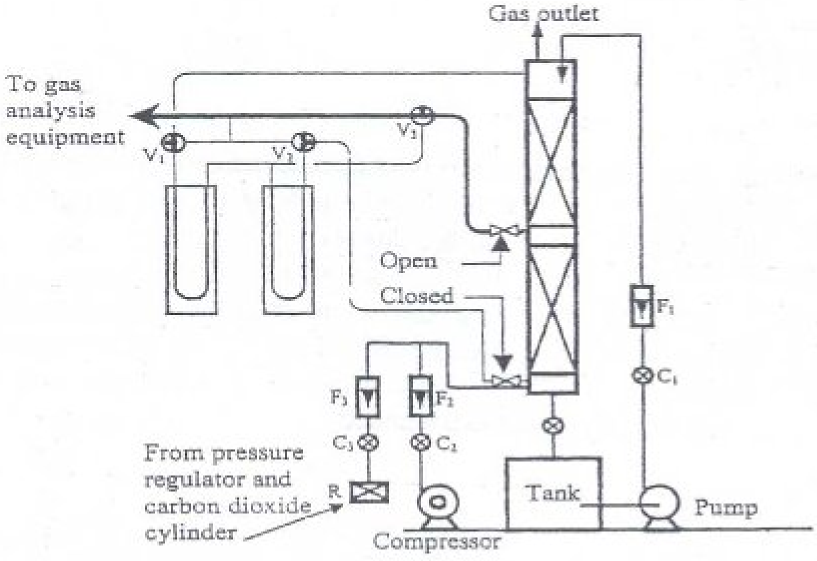
EXPERIMENT IV

Gas absorption/ separation process

Objects OF Experiment:

To calculate rate of absorption of carbon dioxide into caustic soda solution from analysis of liquid solution flowing down absorption column

EQUIPMENT SET- UP:



PROCEDURE:

1. Fill the liquid reservoir tank at the base of the column to approximately three- quarters full with 0.2M caustic soda solution. The preparation of this requires great care and **GLOVES AND GOGGLES MUST BE WORN**. Use standard 1M caustic soda solution from normal laboratory supplies and add with care to the appropriate reservoir, eg. First add 30 litres of tap water to the reservoir, followed by successive addition of 1M caustic soda until a total volume of 7.5 litres of 1M caustic soda has been added, giving 37.5 litres of 0.2M .

2. With gas flow control valves C_2 and C_3 closed, start the liquid pump and adjust the flow of caustic soda through the column to approximately 3 litres/min on flow meter F_1 by adjusting control valve C_1.

3. Start the compressor and adjust valve C_2 to give an air flow of approximately 30 litres/minute on flow meter F_2 .

CAUTION: As the solution used in this experiment is caustic it is essential that the column is not allowed to flood. The air supply should be turned off immediately if any liquid is present in the clear tube above the top of the column.

4. Carefully open the pressure regulating valve on the carbon dioxide cylinder and adjust valve C_3 to give a flow of litres/minute on flow meter F_3. Ensure the liquid seal at the base of the absorption column is maintained by, if necessary, adjustment of control valve C_4.

5. After 15 minutes of steady operation, take samples at 20minute intervals simultaneously from S_4 and S_5. Take 250ml samples at known times in each case. Analyse both samples according to the procedure detailed overleaf.

The Theory of Analysis of Carbon Dioxide Dissolved in Caustic Soda:

The absorption of carbon dioxide from mixtures with air into caustic solution is characterized by the overall reaction (for most condition) as :

$CO + 2\ NaOH \rightarrow Na_2CO_3 + H_2O$

Under the condition chosen for the absorption experiments, the amount of CO removed from the air stream can be estimated from the amounts of NaOH and Na_2CO_3 in the liquid samples, as virtually no "free" CO_2 will remain unreacted in the liquid.

In using titration techniques of analysis, acid is first used to neutralize the caustic soda and at the same time converts all sodium carbonate to bicarbonate. Continuation of the titration with acid then neutralizes all bicarbonate. The total concentration of carbonate can thus be determined, and hence the amount of CO_2 absorbed deduced.

Solution Needed:

1. Phenolphthalein indicator prepared from carbon dioxide- free distilled water.
2. Methyl orange indicator similarly prepared.
3. One litre of standardized 0.20M hydrochloric acid.
4. One litre of 5% by weight of barium chloride solution.

Procedure:

a) Take a 250ml sample of liquid from the absorption column liquid outlet or sump tank (as directed in the experiment) into a conical flask. Pipette two 50ml portions of this into separate conical flasks.

b) Flask 1-Add a drop of phenolphthalein solution 1) to the contents and titrate until the pink color just disappears with the standard hydrochloric acid 3) Note the volume of acid added $-T_1$, which is that needed to neutralize all hydroxide and convert carbonate to bicarbonate. Then add a drop of methyl orange 2) to the flask and continue to titrate with standard acid 3) until the end point is found. Note the total acid added to this second end point –T_2, which represents neutralization of all bicarbonate as (T_2-T_1).

c) Flask 2 – add about 10% more than the value of (T_2 – T_1) of the barium chloride solution 4) to the flask contents and shake well. This precipitates out all the original carbonate in the sample as barium carbonate. Now add two drops of phenolphthalein solution 1) and titrate against the standard acid solution 3) to the end point. Note the volume of acid added – T_2, which represents that needed to neutralize only the original caustic soda. (T_2-T_3) represents the difference between total acid required for carbonate and hydroxide, and that required for hydroxide alone.

Overal: $Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$

It is advisable to repeat all of the above to check reproducibility.
Calculations of Sample Composition
a) Concentration of NaOH in original sample:
$C_c = T_3/50*0.2M$ (gram-moles/litre)

b) Concentration of $NaCO_3$ in original sample:
$C_N = (T_2-T_3)/50*0.2M*0.5$

c) Amount of CO_2 removed from air mixture:

During a time interval, or between the top and bottom of the column, C_N will increase as CO_2 is absorbed in equi-molar proportions, while C_c should decrease in twice-molar proportions.

READING:

Volume of solution in system __________ litres (V_T)
Flow of solution (F_1): __________ litres/second

Time from start (mins)	ANALYSIS OF LIQUID SAMPLES									
	From sump tank (ie.column inlet) S_5					From liquid outlet S_4				
	T_1ml	T_2ml	T_1ml	C_C	C_N	T_1	T_2	T_3	C_C	C_C
0										
20										
40										
60										

Calculations:

The amount of CO_2 absorbed across the column as measured from samples taken simultaneously from the sump tank feeding the column top and at the bottom outlet, is given by:-

CO_2 absorbed = liquid flow rate x $[(C_N)_o - (C_N)_i]$

(gram-moles/sec) (litres/sec) (gram-moles/litres)

= Liquid flow rate x $\frac{1}{2}[(C_c)_i - (C_c)_o]$

Similarly, over a time period θ seconds after a first sample is taken from the sump S_5:-

CO_2 absorbed = Volume of liquid in whole system x $\left[(C_N)_{t=\theta} - (C_N)_{t=0}\right]$

Chemical Absorption

EXPERIMENT V

Gas absorption/ separation process

Objects OF Experiment:

To show that the amount of carbon dioxide removed from the air stream equals the amount taken up by a liquid stream of caustic soda solution.

EQUIPMENT SET- UP:

The same equipment of EXPERIMENT IV

SUMMARY OF THEORY

At steady state, the transfer of absorbing gas from equal that transferred to the liquid.

Let: L_I and L_o be the volume flow of liquid entering and leaving the column respectively.

Let: G_I and G_o be the total gas molal flow entering and leaving the column respectively.

Let: Y_I and Y_o be the mole fractions of CO_2 entering and leaving the column in the gas stream.

Amount of CO_2 removed from the gas stream: -

$G_I - G_o$ (gm.moles/sec) ……. (1)

As no air dissolved in the solution.

But from experiment B, the amount of CO_2 removed from the liquid stream equal the amount of carbonate ions produced: -

$L_o.C_{No} - L_i.C_{Ni}$ (gm.moles/sec) ……..(2)

The object is to check that (1) equals (2). Note that, while the liquid flows in and out are the same in this experiment ($L_o = L_I$), the gas flows are not equal because of the removal of CO_2, and also because of the pressure drop across the column.

G_o can be calculated from a molar balance on the air stream, none of which is absorbed:
$G_o (1-Y_o) = G_I (1-Y_I)$

G can be calculated from the fact that one gram mole occupies 22.42 litres at 273K and 760mm Hg pressure:

$G= F_2+F_3/22.42*760+\text{column pressure drop}/760*273/\text{column temp K}$

And Y_I and Y_o are estimated by sampling as in Experiment A.

PROCEDURE:

The same procedure should be followed as for Experiment C, except that gas samples at the inlet and outlet, as described in Experiment A, are to be taken as well.

As the liquid composition is slowly changing as progressive absorption of CO_2 takes place, the steady state can only be approximated by taking samples as close to each other in time as possible.

Thus, after 5 minutes of circulating liquid and gases at the pre-set rates, take

1. a gas outlet sample, to give Y_o,
2. a liquid outlet sample from the point S_4 as soon after 1) as possible,
3. a liquid sample from the reservoir tank at S_5 at the same time,
4. inlet gas sample, which should be constant and therefore can be taken last.

READINGS TO BE TAKEN:

QUANTITY	UNITS	SYMBOL	COMMENTS
Air flow rate	litres/min		From flowmeter
∴ air flow rate	litres/sec	F_2	(Divided by 60)
CO_2 flow rate	litres/min		From flowmeter
CO_2 flow rate	litres/sec	F_3	
Caustic soda flow rate	litres/min		From flowmeter
Caustic soda flow rate	litres/sec	$L_i = L_o$	
CO_2 outlet concentration	vol. fraction	Y_o	From Hempl analysis
CO_2 inlet concentration	vol. fraction	Y_i	From Hempl analysis (should equal F_2/F_3+F_2)
		Liquid Samples: **outlet**	
Titration with HCl	m	$T_1(o)$	From Experiment C Procedure
Titration with HCl	ml	$T_2(o)$	
Titration with HCl	ml	$T_3(o)$	
Liquid Samples: **inlet = sump**			
Titration with HCl	ml	$T_1(i)$	From Experiment C Procedure
Titration with HCl	ml	$T_2(i)$	
Titration with HCl	ml	$T_3(i)$	
Barometric pressure	mm H	ΔP	Assumed equal to outlet pressure
Column pressure drop	mm H_2O		From manometer
Pressure at base of column	mm Hg	$P + \dfrac{\Delta P}{13.6}$	
Temperature of feed gas	°C	θ_i	
Temperature of feed gas	K	$\theta_i + 273$	

SAMPLE CALCULATIONS:

Reading:	Comments:
$F_2 = 0.40$ litres/second $F_3 = 0.052$ litres/second $L = 0.051$ litres/second	Flow meter reading divided by 60
$Y_o = 0.032$ $Y_i = 0.111$ [and $Y = F_3/F_2+F_3=0.052/0.40+0.052=0.115$]	From Hemple apparatus reading (Use 0.111 for Y_i)
$T_1(o) = 33.2$ml $T_2(o) = 50.9$ml $T_3(o) = 35.0$ml $T_1(i) = 48.4$ml $T_2(i) = 47.2$ml $T_3(i) = 45.0$ml	As in experiment C, 0.20M HCl used in titration of 50ml sample As in experiment C, 0.20M HCl used in titration of 50ml sample
$P = 759$mm Hg	Lab. Barometer
$P = 100$mm H_2O	$100/13.6=7.35\approx 7$mm Hg
$\theta i = 19^{\circ}C$	292 K

Calculations:

1) Gas Flows:

→ G_i (g.moles/second of gas mixture entering column)

$= 0.40+0.052/22.42 * 759+7/759*273/292 = 0.0175$g.moles/second

and $G_o= G_i(1-Y_i)/(1-Y_i)=0.190*(1-0.111)/(1-0.032) =0.0175$g.moles/second

→Amount of CO_2 removed = 0.0015g.moles/second

Determination of Overall Mass Transfer Coefficient (K_{og}).

EXPERIMENT VI

Gas absorption/ separation process

Objects OF Experiment:

Determination of Overall Mass Transfer Coefficient (K_{og}).

EQUIPMENT SET- UP:

Using the same system and apparatus **as mentioned in the Experiment IV and V**

SUMMARY OF THEORY:

The familiar equation for packed tower absorption is

$$H=\int_{Yi}^{Yo} d[G.Y]/K_{og}.\ A\ a.(Y^*-Y)$$

Where Y* is the mole fraction of gas in equilibrium with the liquid at any point in the tower and where Y is the bulk mole fraction
A is the cross-sectional area of the tower; His the packing height and
a is the specific area of packing /unit volume of packing.

For dilute gases in an otherwise inert gas stream, the above equation can be simplified:-

$$H.aA.K_{og}/\ G\int_{Y_i}^{Y_o} dY/Y^*-Y$$

The right hand side of this equation is difficult to integrate and K_{og} is more simply (but less accurately) evaluated from the definition of K_{og} as:

N	$=K_{og}{}^*aAH$	X log mean
Rate of absorption (g.moles/second)	area for mass transfer (m^2)	driving force (pressure in atm)

$$\rightarrow K_{og} = N/aA.H.\ {}^*1NP^{Pi}o/P_i - P_o$$

PROCEDURE:

The experiment is conducted as for Experiment V, except that the liquid analyses may be omitted as the gas analysis alone can determine the rate of absorption.

RESULTS AND CALCULATIONS:

1) N is calculated as in Experiments previous experiments.
2) a is specific area of packing/unit volume of tower, which for 9mm Raschig rings is $440 m^2/m^3$.
3) A.H.is volume of the column.= volume of tower $=\pi/4*(0.075)^2*1.4=0.0062 m^3$

Area height

4) Partial pressures (of CO_2):

Partial pressure = mole fraction*total pressure

Partial pressure = volume fraction* total pressure

→ Using results of chemical absorption :

Inlet: $P_i = 0.111*(759.17)\ 0.111/760$ atm.

Outlet $P_o = 0.032*(759)0.032/760$

→ Log mean driving force $=P_i - P_o/1nP_i/P_o= 0.111- 0.032$atm $/1n.111/0.032$

5) Mass transfer coefficient:-

→K_{og}=0.0015(g.moles/second)/0.0062*440*0.064 =0.0086g.moles/atm.m^2 sec

PART 2

Liquid /Liquid Extraction Unit UOP5

SAFETY IN THE USE OF EQUIPMENT SUPPLIED BY ARMFIELD

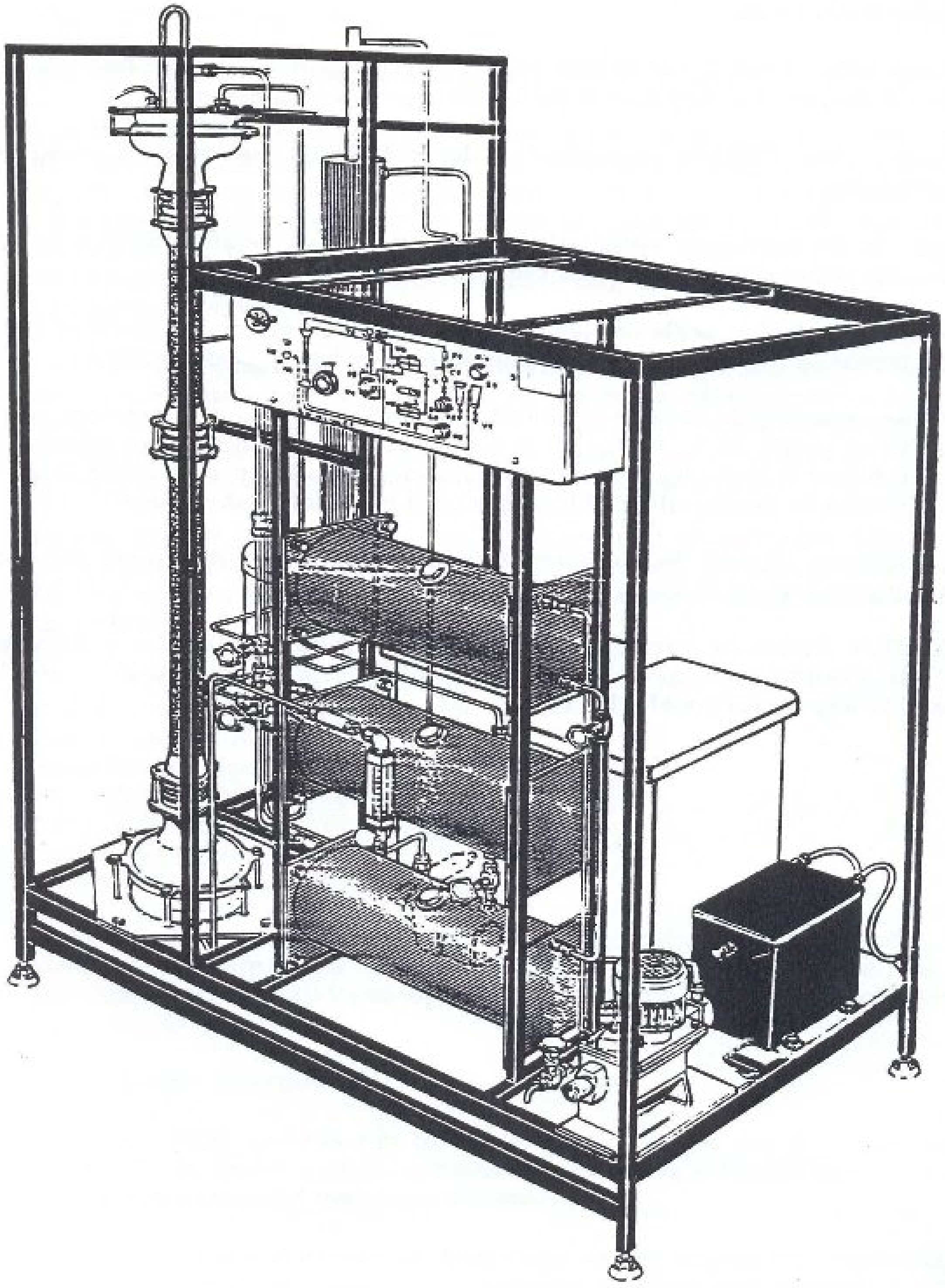
Before proceeding to install, commission or operate the equipment described in this instruction manual we wish to alert you to potential hazards so that they may be avoided.

Although designed for safe operation, any laboratory equipment may involve processes or procedures which are potentially hazardous. The major potential hazards associated with this particular equipment are listed below.

- INJURY THROUGH MISUSE
- INJURY FROM ELECTRIC SHOCK
- INJURY FROM INCORRECT HANDLING
- POISONING FROM TOXIC MATERIALS
- INJURY FROM CORROSIVE LIQUIDS
- RISK OF INFECTION THROUGH LACK OF CLEANLINESS
- DAMAGE TO CLOTHING

Accidents can be avoided provided that equipment is **regularly maintained** and **staff** and **students** are made aware of potential hazards. A list of general safety rules is included in this manual, to assist **staff** and **students** in this regard. The list is not intended to be fully comprehensive but for guidance only.

Please refer to the notes overleaf regarding the Control of Substances Hazardous to Health Regulations.



UOP 5 LIQUID / LIQUID EXTRACTION UNIT

INTRODUCTION

Many processes in chemical engineering require the separation of one or more of the components of a liquid mixture by treating the mixture with an immiscible solvent in which these components are preferentially soluble. In some cases purification of a liquid may be the function of the process, in others the extraction of a dissolved component for subsequent processing may be the important aspect. An example of the former is the preparation of pure organic liquids from products of the oil industry. Liquid/liquid extractions may also be used as energy-saving processes by, for example, eliminating distillation stages. It is possible, of course that the substance of interest may be heat-sensitive anyway and that distillation is accordingly an unacceptable process.

The rate at which a soluble component is transferred from one solvent to another will be dependent, amongst other things on the area of the interface between the two immiscible liquids. Therefore it is very advantageous for this interface to be formed by droplets and films, the situation being analogous to that existing in packed distillation columns.

The Armfield Liquid/Liquid Extraction Unit takes the form of a vertically-oriented packed column which may be operated either, by filling the column with water and allowing a solvent to flow down the column over the packing, or filling the column with solvent and allowing water to flow up the column over the packing. In either case the process is continuous, both liquids being pumped into the column. Sensing electrodes at the top and bottom of the column determine whether the column is filled with water or with solvent. This is achieved by sensing and maintaining the position of the water level at the appropriate height. A solenoid valve controlling the flow of solvent under gravity from the column is operated by the sensing electrode system.

A distillation unit with a fractionating column is included to allow the reclamation of solvent where appropriate.

DESCRIPTION

All numerical references relate to the Equipment Diagram on page 12 and the Component System Diagram on page 13 and in the experimental section of this manual.

All letter coded references relate to the Control and Instrumentation System Diagram on page 14 and in the experimental section of this manual.

The equipment is mounted in a floor-standing, welded steel framework (8) fitted with adjustable feet (12). The frame contains an extra cross-member (9) at the front and a similar one at the rear to allow the use of a fork-lift truck.

The glass liquid/liquid extraction column (19a & 19b) is fitted with enlarged end sections (14), (25), which are closed by stainless steel plates (13), (26), the lower one being bolted to the framework and supporting the column. The four sections of the column proper and the two end plates are all fastened together with flanges (15), the joints between the sections being sealed with moulded PTFE gaskets. The column is filled with Raschig rings which are supported on a perforated stainless steel plate (16) fitted between the bottom enlarged portion and the lower section of the column.

Water for the column is stored in the supply tank (37) (L2) from where it is pumped by the centrifugal pump (4), through an air bleed valve (5) (V3), a flow control valve (23) (C1) and a flowmeter (24) (F1) to an injector (28) mounted in the baseplate and with its exit about 150mm above the plate. Water leaves the top of the column through a pipe, and is collected in a polythene tank (40) (L1).

All storage tanks for solvent are constructed in stainless steel. The organic solvent supply tank (3) (L5) provides the feed for the metering pump (43) (F2), the pumping rate of which is varied by a stroke adjustment knob (1) and indicated by a digital read-out as a percentage of the maximum flow (300 ml/min). Pumped solvent enters the top of the column via an injector similar to that fitted at the base for the water (28). A sampling and drain cock (2) (V6) is fitted in the solvent feed line. Solvent from the base of the column is returned under gravity to the receiver vessel (36) (L3) via a pipeline (27) which is also fitted with a solenoid valve (6) (C3) and a sampling cock (V9).

The level of the water/solvent interface in the column is determined by the operation of the solenoid valve (6) (C3) in the solvent outlet pipeline. The operation of this valve is controlled by water-sensing electrodes, one set fitted to the top plate (31) and another set to the bottom plate (11). A switch (S2) on the system control panel (34) (see also diagram of the panel) selects the electrodes in use and hence determines whether the interface is

The three solvent tanks (36) (L3), (39) (L4), (3) (L5) are vented to atmosphere through a vent pipe which is inserted into the top of the condenser. Solvent levels in the tanks and boiler are indicated with glass sight tubes protected by clear polyethylene sleeves.

Control of the equipment is simplified with a system diagram panel (34) shown in detail on page 12. The complete solvent/water flow system is shown with electrical controls at the appropriate points on the diagram. These are mains on/off switch (S1), water pump on/off switch (S3), solvent pump on/off switch (S4), boiler heater switch (S5) and power regulator (R1), electrode changeover switch (S2), and solenoid valve 'open' indicator light. The four mains supply switches are self-illuminating in the 'on' position. A mains transformer (42) is fitted when the electrical supply is 120V, 60Hz A.C.

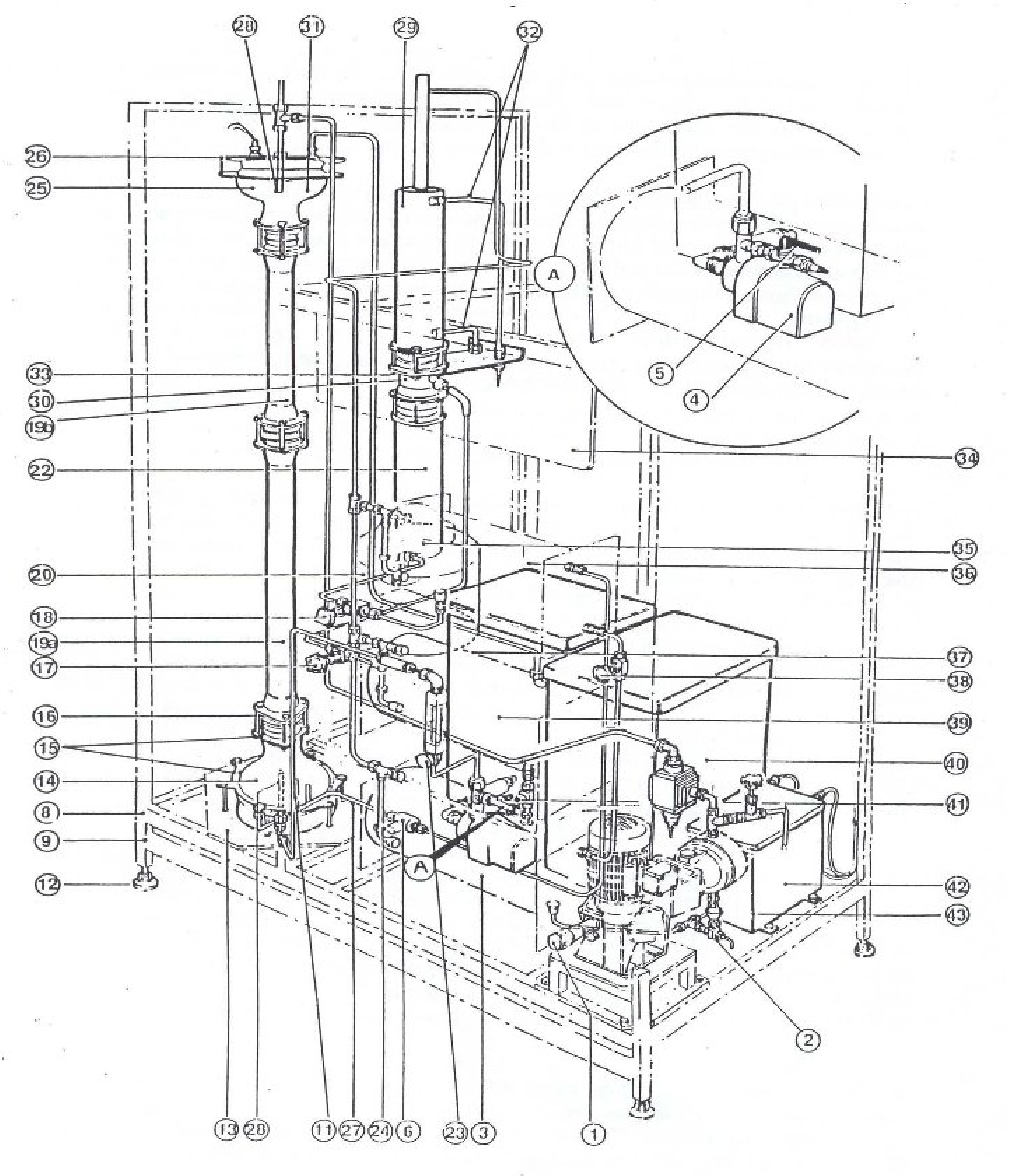
through the column once may be treated again in a batchwise fashion or the valve (38) (V4) may be left open during the extraction to provide a closed circuit, the solvent then being re-circulated continuously.

The distillation column boiler (20) (L6), mounted behind the extraction column, is fitted at such a height that liquid may be drained into it from the upper of the three solvent tanks and can be drained from it into the lowest tank. The valve (18) (V7) controls the release of solvent into the boiler. Like pipework, valves and fittings, the boiler is constructed in stainless steel. Heating is by means of two 500W cartridge elements inserted at the base of the boiler and boiler temperature is indicated on a thermometer in the side. The boiler lid is perforated where the distillation column is fitted (35) and three blind tapped holes around this area accept bolts fixing the column flange to the boiler.

The column proper is made up of a glass section (22) containing four sieve plates. The glass reflux divider (33) bolts to the column top and to the stainless steel condenser above it with bolts and flanges and all the sections are sealed together with moulded PTFE gaskets.

The reflux divider is fitted with a thermometer (30) (T2) to indicate the vapour temperature beneath the divider. The condensate outlet tube and the thermometer tube on the divider are terminated with glass screw-thread connectors and screw caps to form secure but demountable connections. The condenser (29) is vented to atmosphere at the top and its jacket is water-cooled by a supply via the copper pipes (32) at the rear. The boiler, distillation column and condenser are all lagged to minimise heat losses.

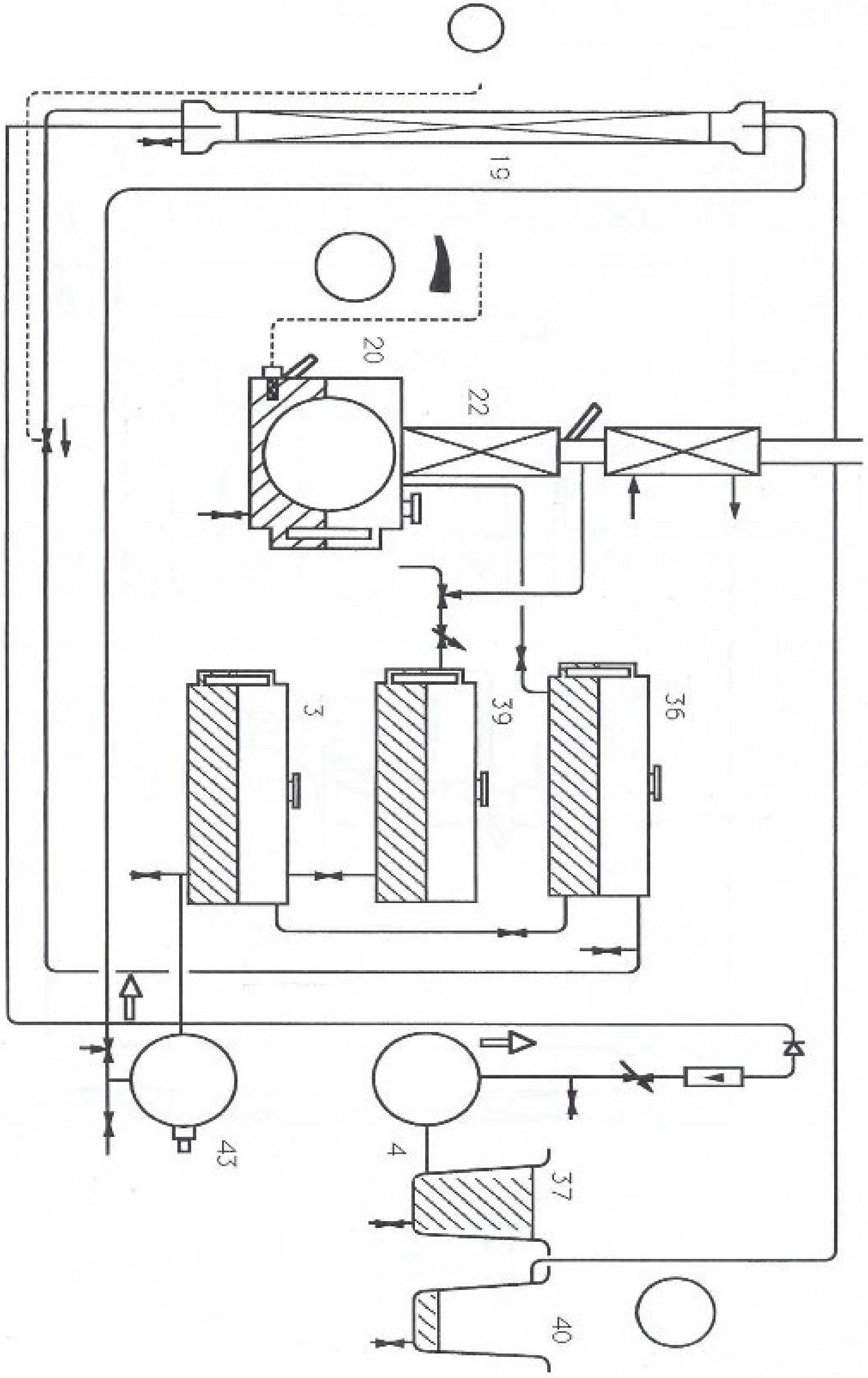
Condensate from the reflux divider flows under gravity into the middle solvent tank (39 (L4) via the flow control valve (17) (C2) which thus determines the reflux ratio. The contents of the middle tank (39) (L4) can be drained into the solvent supply tank by opening the valve (41) (V5) between the two.

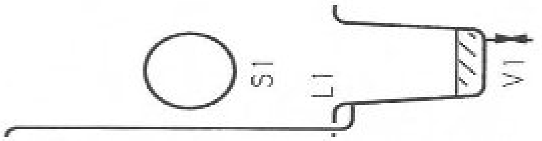


UOP5 LIQUID / LIQUID EXTRACTION UNIT

UOP5 SYSTEM DIAGRAM

(COMPONENTS)





The following procedure is intended for initially checking that the equipment is operating correctly after assembly.

Water is used in place of organic solvents on the grounds of safety.

Where the equipment is to be set up for a specific experiment, refer to the relevant experimental sheet at the rear of this manual.

Before proceeding with the commissioning ensure that the gearbox on the solvent pump has been filled with oil (indicated by the presence of oil half way up the sight gauge on the side of the gearbox).

1. Close all valves on the equipment - including drain valves. Check longest and middle electrodes at top of extraction column, and shortest and middle electrodes at bottom of column are approximately 5mm apart. Adjust if necessary.

2. Fill tank L2 with tap water.

3. Connect the equipment to the electrical supply.

4. Set electrode switch to top position (switch S2). Operate mains switch S1 - check S3, S4 and S5 are 'OFF' and S1 is illuminated. Check solenoid valve C3 is open (light on). Set the electrode switch to bottom position then check that solenoid valve C3 is still open (light on).

5. Open vent valve V3 then switch on water pump (switch S3). Allow water pump to prime (water discharges at V3), then close vent valve V3.

6. Open control valve C1. Check flow of water on flowmeter F1. Check water is delivered to injector at base of extraction column.

7. Allow column to start filling with water at a slow rate. The solenoid valve C3 will be open (light on) until water covers the short electrode when C3 will close (light off). Open the drain valve (V11) at the base of the extraction column and drain water until the tip of the short electrode is exposed and the valve will again open.

8. Close drain valve (V11) in base of column. Open control valve C1 and adjust to give full scale reading on flowmeter F1. C3 will close again when the water level reaches the short electrode then the column will gradually fill. Leave the electrode switch in the down position. Check

C3 is still closed (light off). Wait for water level to reach the top plate ie. column completely full. Make sure water flows from the top of the column to the polythene storage tank (L1).

9. Switch S2 to top electrodes position. Check C3 remains closed. Close the flowmeter control valve C1. Open the drain valve V11 at the base of the column and allow water to drain until the water level falls below the long electrode. Check valve C3 opens (light on). Water will drain into tank L3 until level in column equals level in tank L3.

10. Check pipework and column for leaks.

11. Switch off water pump (switch S3). Drain all remaining water from column through drain valve V11 in base of column.

12. Fill vessel L3 with tap water through top filler. Check sight gauge on L3 operates.

13. Half fill vessel L4 with tap water through top filler. Check sight gauge on L4 operates.

14. Open valve V7. Allow boiler L6 to 1/3rd fill with water. Close valve V7. Check sight gauge on L6 operates.

15. Open valve V4. Allow vessel L5 to fill with water - allow L3 to drain. Close valve V4. Check sight gauge on L5 operates.

16. Set calibration valve V8 to the column position, open bleed valve V13 at the solvent pump discharge and place a suitable container beneath the valve to allow the water which will discharge to be collected. Set pump delivery to 100% (F2) then switch on solvent pump (switch 54). When water flows from bleed valve V13 close valve V13 and check that water is delivered to injector at top of extraction column. Set calibration valve V8 to the calibrate position and check that water is delivered to the bypass. Close calibration valve V8. Check F2 regulates delivery to extraction column. Switch off solvent pump (switch S4). Check system for leaks. DRAIN WATER.

    NOTE: Solvent pump needs to be calibrated before experiments can take place

17. Connect cooling water to lower connection on condenser. Connect top connection on condenser to drain.

18. Turn on cooling water supply. Check flow of water to drain. Check for leaks.

19. Set power control R1 to minimum (fully anti-clockwise). Switch on heating elements (switch S5). Rotate power control R1 to maximum (fully clockwise). Check temperature of water increases (Thermometer T1). Check boiler for leaks. Allow water to boil. When condensate collects in reflux divide open control valve C2. Check water flows from reflux divider to vessel L4.

20. Set power control R1 to minimum (fully anti-clockwise) then drain the boiler by opening valve V10. When the level falls below the level switch power should be disconnected (S5 not illuminated). Switch off the boiler (S5).

21. Disconnect the electrical supply, turn off cooling water and drain all water from the equipment.

GENERAL SAFETY RULES

1 *Follow Relevant Instructions*

a Before attempting to install, commission or operate equipment, all relevant suppliers/manufacturers instructions and local regulations should be understood and implemented.

b It is irresponsible and dangerous to misuse equipment or ignore instructions, regulations or warnings.

c Do not exceed specified maximum operating conditions (e.g. temperature, pressure, speed etc.)

2 *Installation*

a Use lifting tackle where possible to install heavy equipment. Where manual lifting is necessary beware of strained backs and crushed toes. Get help from an assistant if necessary. Wear safety shoes where appropriate.

b Extreme care should be exercised to avoid damage to the equipment during handling and unpacking. When using slings to lift equipment, ensure that the slings are attached to structural framework and do not foul adjacent pipework, glassware etc. When using fork lift trucks, position the forks beneath structural framework ensuring that the forks do not foul adjacent pipework, glassware etc. Damage may go unseen during commissioning creating a potential hazard to subsequent operators.

c Where special foundations are required follow the instructions provided and do not improvise. Locate heavy equipment at low level.

d Equipment involving inflammable or corrosive liquids should be sited in a containment area or bund with a capacity 50% greater than the maximum equipment contents.

e Ensure that all services are compatible with the equipment and that independent isolators are always provided and labelled. Use reliable connections in all instances, do not improvise.

f Ensure that all equipment is reliably earthed and connected to an electrical supply at the correct voltage. The electrical supply must incorporate a Residual Current Device (RCD) (alternatively called an Earth Leakage Circuit Breaker - ELCB) to protect the operator from severe electric shock in the event of misuse or accident.

g Potential hazards should always be the first consideration when deciding on a suitable location for equipment. Leave sufficient space between equipment and between walls and equipment.

3 *Commissioning*

a Ensure that equipment is commissioned and checked by a competent member of staff before permitting students to operate it.

4 *Operation*

a Ensure that students are fully aware of the potential hazards when operating equipment.

b Students should be supervised by a competent member of staff at all times when in the laboratory. No one should operate equipment alone. Do not leave equipment running unattended.

c Do not allow students to derive their own experimental procedures unless they are competent to do so.

d Serious injury can result from touching apparently stationary equipment when using a stroboscope to \`freeze´ rotary motion.

5 *Maintenance*

a Badly maintained equipment is a potential hazard. Ensure that a competent member of staff is responsible for organising maintenance and repairs on a planned basis.

b Do not permit faulty equipment to be operated. Ensure that repairs are carried out competently and checked before students are permitted to operate the equipment.

6 *Using Electricity*

a At least once each month, check that ELCB's (RCCB's) are operating correctly by pressing the TEST button. The circuit breaker must trip when the button is pressed (failure to trip means that the operator is not protected and a repair must be effected by a competent electrician before the equipment or electrical supply is used).

b Electricity is the commonest cause of accidents in the laboratory. Ensure that all members of staff and students respect it.

c Ensure that the electrical supply has been disconnected from the equipment before attempting repairs or adjustments.

d Water and electricity are not compatible and can cause serious injury if they come into contact. Never operate portable electric appliances adjacent to equipment involving water unless some form of constraint or barrier is incorporated to prevent accidental contact.

e Always disconnect equipment from the electrical supply when not in use.

7 *Avoiding fires or explosion*

a Ensure that the laboratory is provided with adequate fire extinguishers appropriate to the potential hazards.

b Where inflammable liquids are used, smoking must be forbidden. Notices should be displayed to enforce this.

c Beware since fine powders or dust can spontaneously ignite under certain conditions. Empty vessels having contained inflammable liquids can contain vapour and explode if ignited.

d Bulk quantities of inflammable liquids should be stored outside the laboratory in accordance with local regulations.

e Storage tanks on equipment should not be overfilled. All spillages should be immediately cleaned up, carefully disposing of any contaminated cloths etc. Beware of slippery floors.

f When liquids giving off inflammable vapours are handled in the laboratory, the area should be ventilated by an ex-proof extraction system. Vents on the equipment should be connected to the extraction system.

g Students should not be allowed to prepare mixtures for analysis or other purpose without competent supervision.

8 *Handling poisons, corrosive or toxic materials*

a Certain liquids essential to the operation of equipment, for example mercury, are poisonous or can give off poisonous vapours. Wear appropriate protective clothing when handling such substances. Clean up any spillage immediately and ventilate areas thoroughly using extraction equipment. Beware of slippery floors.

b Do not allow food to be brought into or consumed in the laboratory. Never use chemical beakers as drinking vessels.

c Where poisonous vapours are involved, smoking must be forbidden. Notices should be displayed to enforce this.

d Poisons and very toxic materials must be kept in a locked cupboard or store and checked regularly. Use of such substances should be supervised.

e When diluting concentrated acids and alkalis, the acid or alkali should be added slowly to water while stirring. The reverse should never be attempted.

9 *Avoiding cuts and burns*

a Take care when handling sharp edged components. Do not exert undue force on glass or fragile items.

b Hot surfaces cannot in most cases be totally shielded and can produce severe burns even when not `visibly hot´. Use common sense and think which parts of the equipment are likely to be hot.

10 *Eye protection*

a Goggles must be worn whenever there is a risk to the eyes. Risk may arise from powders, liquid splashes, vapours or splinters. Beware of debris from fast moving air streams. Alkaline solutions are particularly dangerous to the eyes.

b Never look directly at a strong source of light such as a laser or Xenon arc lamp. Ensure that equipment using such a source is positioned so that passers-by cannot accidentally view the source or reflected ray.

c Facilities for eye irrigation should always be available.

11 *Ear protection*

a Ear protectors must be worn when operating noisy equipment.

12 *Clothing*

a Suitable clothing should be worn in the laboratory. Loose garments can cause serious injury if caught in rotating machinery. Ties, rings on fingers etc. should be removed in these situations.

b Additional protective clothing should be available for all members of staff and students as appropriate.

13 *Guards and safety devices*

a Guards and safety devices are installed on equipment to protect the operator. The equipment must not be operated with such devices removed.

b Safety valves, cut-outs or other safety devices will have been set to protect the equipment. Interference with these devices may create a potential hazard.

c It is not possible to guard the operator against all contingencies. Use common sense at all times when in the laboratory.

d Before starting a rotating machine, make sure staff are aware how to stop it in an emergency.

e Ensure that speed control devices are always set at zero before starting equipment.

14 *First aid*

a If an accident does occur in the laboratory it is essential that first aid equipment is available and that the supervisor knows how to use it.

b A notice giving details of a proficient first-aider should be prominently displayed.

c A `short list´ of the antidotes for the chemicals used in a particular laboratory should be prominently displayed.

UOP5 - LIQUID/LIQUID EXTRACTION UNIT

INDEX TO EXPERIMENTS

EXPERIMENT A

OBJECT OF EXPERIMENT:

To determine the distribution coefficient for the system Trichloroethylene-Propionic Acid-Water and to show its dependence on concentration.

EQUIPMENT SET-UP:

The following apparatus is required:

250ml Conical stoppered flask
250ml Measuring cylinder
250ml Separating funnel
Pipette with rubber bulb
Sodium Hydroxide Solution (M/10)
Phenolphthalein
Propionic acid

WARNING Concentrated Sodium Hydroxide can form explosive volatile products when in contact with Trichloroethylene. Ensure that diluted Sodium Hydroxide (NaOH) is used when performing this experiment.

SUMMARY OF THEORY:

The solvent (water) and solution (trichloroethylene/propionic acid) are mixed together and then allowed to separate into the extract phase and the raffinate phase. The extract phase will be water and propionic acid and the raffinate, trichloroethylene with a trace of propionic acid.

The distribution coefficient, K, is defined as the ratio

$$\frac{\text{Concentration of solute in the extract phase, Y}}{\text{Concentration of solute in the raffinate phase, X}}$$

It is assumed that equilibrium exists between the two phases.

At low concentrations, the distribution coefficient is dependent on the concentration and thus $Y = KX$.

READINGS TO BE TAKEN:

1. Make up in a conical flask a mixture of 50ml trichloroethylene and 50ml of de-mineralised water.

2. Add 5ml of propionic acid. 5ml can be pipetted into the flask using a pipette with a rubber bulb.

3. Place a stopper into the flask and shake for a minimum of 5 minutes.

4. Pour into a separating funnel, leave for 5 minutes and remove the lower aqueous layer.

5. Take a 10ml sample of this layer and titrate against M/10 sodium hydroxide solution using phenolphthalein as indicator.

6. Repeat the experiment for two further concentrations of propionic acid i.e. for initial additions of 3ml and 1ml of propionic acid.

RESULTS:

Propionic Acid added (ml)	Titre of M/10 NaOH	Propionic Acid Concentration in Aqueous Layer, Y	Propionic Acid Concentration in Organic Layer, X	$K=\frac{Y}{X}$
5				
3				
1				

1. ***Fill out the calculated data in the table.***
2. ***Plot the results in triangle diagram to prepare an equilibrium curve.***
3. ***Plot the results as Y versus X producing an equilibrium curve.***

EXPERIMENT B

OBJECT OF EXPERIMENT:

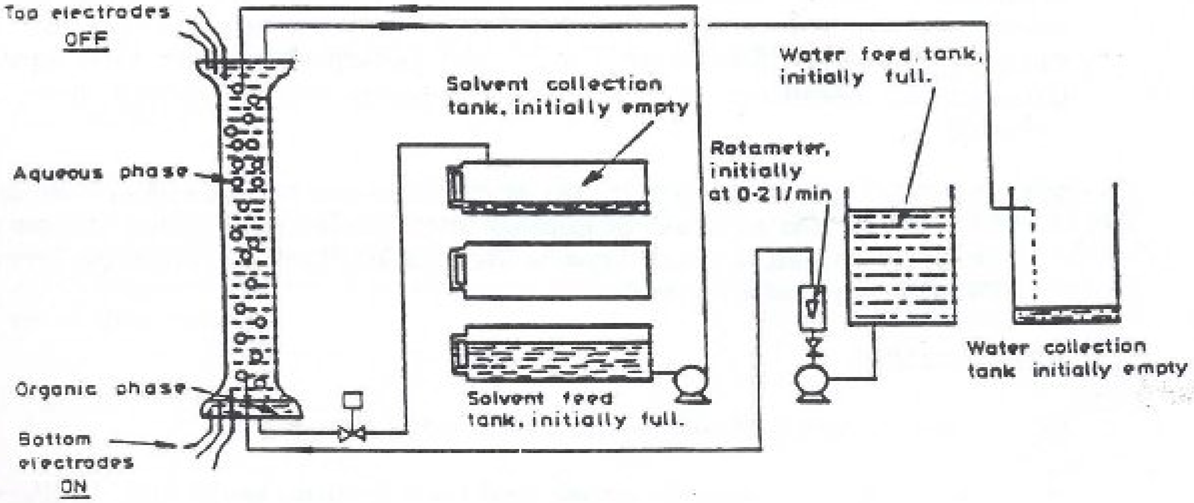
To observe the hydraulics of counter current flow in a packed column.

EQUIPMENT SET-UP:

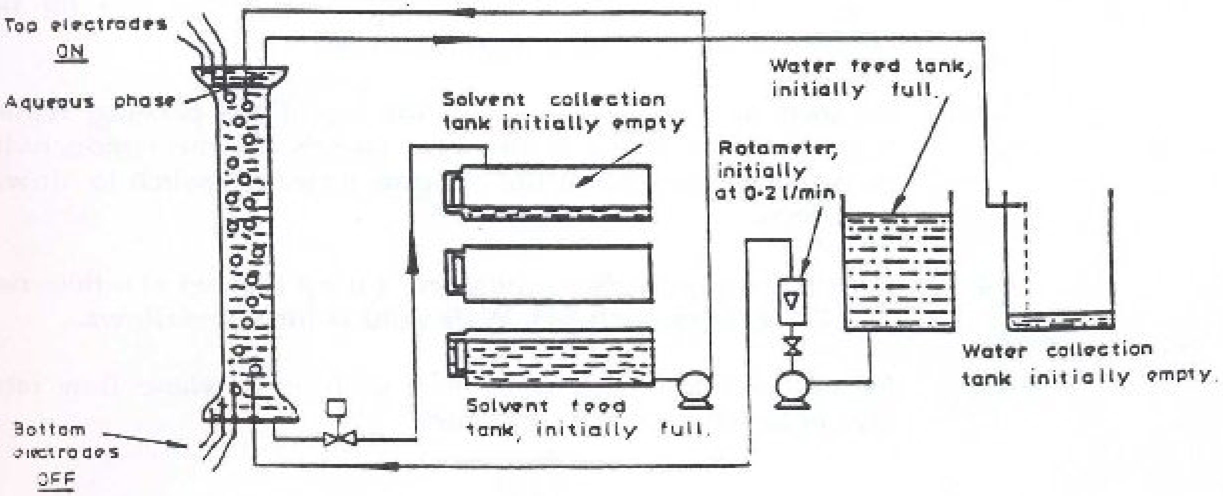
The experiment will be carried out using the two immiscible liquids trichloroethylene and water and the column will be operated in the following two modes:

(a) The aqueous phase as the continuous medium.
(b) The organic phase as the continuous medium.

(a) AQUEOUS PHASE AS CONTINUOUS MEDIUM



(b) ORGANIC PHASE AS CONTINUOUS MEDIUM



The solvent metering pump is calibrated in percentage of maximum flow which varies slightly from pump to pump. The pump should be calibrated initially by setting F2 to 100%, setting valve V8 to the calibrate position and measuring the flow from the pump, using a measuring cylinder and stopwatch. Calculate the flow rate produced settings of 10% intervals (ml per minute), then plot a graph of ml per minute against percentage of metering pump stroke. Thereafter any selected flow may be obtained by using the graph.

SUMMARY OF THEORY:

It is normal to operate a column so that the continuous phase 'wets' the packing. If the packing is 'wetted' by the dispersed phase then coalescence will be increased and the mass transfer/unit volume will be reduced. The rate of mass transfer of a solute from one phase to another is normally increased with greater throughput of material, because of increased turbulence giving better mixing. There is however, a limit to the maximum amount of material that can be fed through the column. The limit is called the 'Flooding Point' and occurs at specific flow rates of constituents resulting in one of the phases being rejected from the column.

WARNING Concentrated Sodium Hydroxide can form explosive volatile products when in contact with Trichloroethylene. Ensure that diluted Sodium Hydroxide (NaOH) is used when performing this experiment.

PROCEDURE:

(a) The Aqueous Phase as the Continuous Phase

1. Fill the organic phase feed tank (bottom tank) with 10 litres of trichloroethylene.
2. Fill the water feed tank with 15 litres of clean de-mineralised water, start the water feed pump (switch S3) and fill the column with water at a high flow rate.
3. As soon as the water is above the top of the packing, reduce the flow rate to 0.2 l/min and switch on the conductivity probe at the bottom of the column (selector switch to 'down' position).
4. Start the organic phase metering pump and set at a flow rate of 0.2 l/min (switch S4). Wait until column overflows.
5. Slowly increase both water and trichloroethylene flow rates and note where flooding occurs.

(b) The Organic Phase as the Continuous Phase

6. Empty the column and return the trichloroethylene and water into their respective feed tanks. This is achieved by allowing the weight of the water to push the trichloroethylene to the solvent collection tank (top tank), (this may mean adding more water). Then draining column and L1, L2 - refilling L2. Switch off the top conductivity probe (switch S2 in the centre position).

7. Start the metering pump (switch 4) and fill the column quickly, the regulate to a flow of 0.2 1/min when trichloroethylene reaches the top of the packing. Switch S2 to the up position.

8. Switch on the water pump (switch S3) and regulate the flow 0.2L/min

9. Slowly increase the flow rates until flooding occurs.

RESULTS:

Observe if there is any difference between operating with the aqueous phase as the continuous medium compared with the organic phase as the continuous medium. Is there any difference in droplet size? Also observe what happens as the flow rates are increased. Does flooding occur, if so at what flow rates?

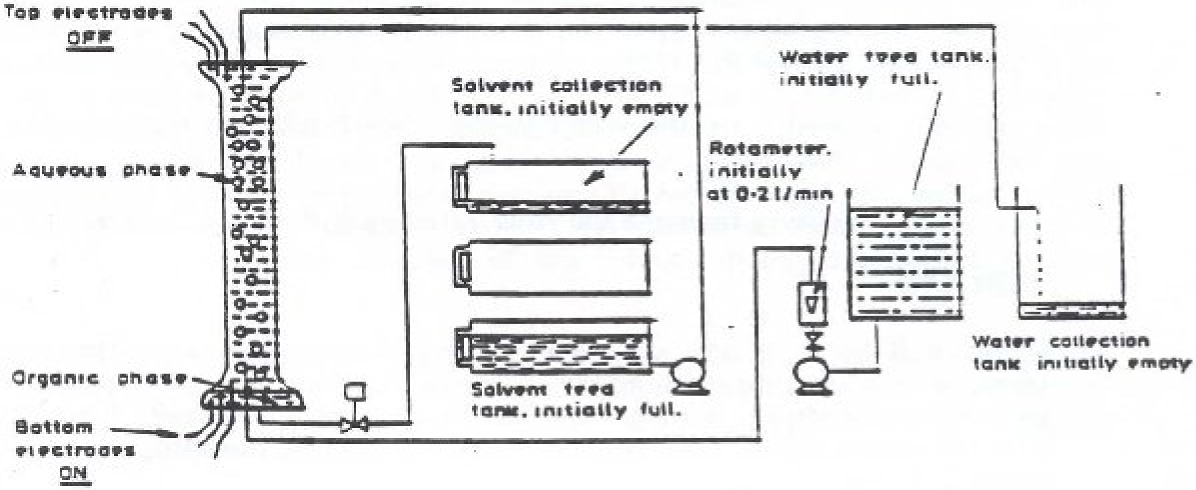
EXPERIMENT C

OBJECT OF EXPERIMENT:

To demonstrate how a mass balance is performed on the extraction column, and to measure the mass transfer coefficient and its variation with flow rate with the aqueous phase as the continuous medium.

EQUIPMENT SET-UP:

AQUEOUS PHASE AS CONTINUOUS MEDIUM



The solvent metering pump is calibrated in percentage of maximum flow which varies slightly from pump to pump. The pump should be calibrated initially by setting F2 to 100%, setting valve V8 to the calibrate position and measuring the flow from the pump, using a measuring cylinder and stopwatch. Calculate the flow rate produced settings of 10% intervals (ml per minute), then plot a graph of ml per minute against percentage of metering pump stroke. Thereafter any selected flow may be obtained by using the graph.

SUMMARY OF THEORY:

The theory is given for the system Trichloroethylene-Propionic Acid-Water.

Let V_w = Water flow rate (l/s)
V_o = Trichloroethylene flow rate (l/s)
X = Propionic Acid concentration in the organic phase (kg/l)
Y = Propionic Acid concentration in the aqueous phase (kg/l)

Subscripts: $_1$ = Top of column
$_2$ = Bottom of column

1. *Mass Balance*

Propionic Acid extracted from the organic phase (raffinate)
$= V_o (X_1 - X_2)$

Propionic Acid extracted by the aqueous phase (extract)
$= V_w (Y_1 - 0)$

Therefore $V_o(X_1 - X_2) = V_w(Y_1 - 0)$

2. *Extraction Efficiency*

Mass transfer coefficient (based on the raffinate phase)

$$= \frac{\text{Rate of Acid Transfer}}{\text{Volume of Packing x Mean Driving Force}}$$

$$\text{Log mean driving force} = \frac{\Delta x_1 - \Delta x_2}{\ln \frac{\Delta x_1}{\Delta x_2}}$$

where ΔX_1 = Driving force at the top of the column = $(X_2 - 0)$
ΔX_2 = Driving force at the bottom of the column = $(X_1 - X_1^*)$

where X_1^* is the concentration in the organic phase which would be in equilibrium with concentration Y_1 in the aqueous phase. The equilibrium values can be found using the distribution coefficient found in the first experiment.

PROCEDURE:

1. Add 100ml of proprionic acid to 10 litres of trichloroethylene. Mix well to ensure an even concentration then fill the organic phase feed tank (bottom tank) with the mixture.

2. Switch the level control to the bottom of the column (electrode switch S2).

3. Fill the water feed tank with 15 litres of clean de-mineralised water, start the water feed pump and fill the column with water at a high flow rate.

4. As soon as the water is above the top of the packing, reduce the flow rate to 0.2 l/min.

5. Start the metering pump and set at a flow rate of 0.2 l/min.

6. Run for 15-20 minutes until steady conditions are achieved, monitor flow rates during this period to ensure that they remain constant.

7. Take 15ml samples from the feed, raffinate and extract streams.

8. Titrate 10ml of each sample against M/10 NaOH using phenolphthalein as the indicator. (To titrate the feed and raffinate they may need continuous stirring using a magnetic stirrer).

9. Repeat the experiment with both the water and trichloroethylene flow rates being increased to 0.3 l/min.

WARNING Concentrated Sodium Hydroxide can form explosive volatile products when in contact with Trichloroethylene. Ensure that diluted Sodium Hydroxide (NaOH) is used when performing this experiment.

RESULTS:

Flow rate of Aqueous Phase Flow rate of Organic Phase		
	Titre of M/10 NaOH	Concentration of Propionic Acid kg/l
Feed		
Raffinate		
Extract		
Propionic acid extracted from the organic phase		
Propionic acid extracted from the aqueous phase		
Mass Transfer Coefficient		

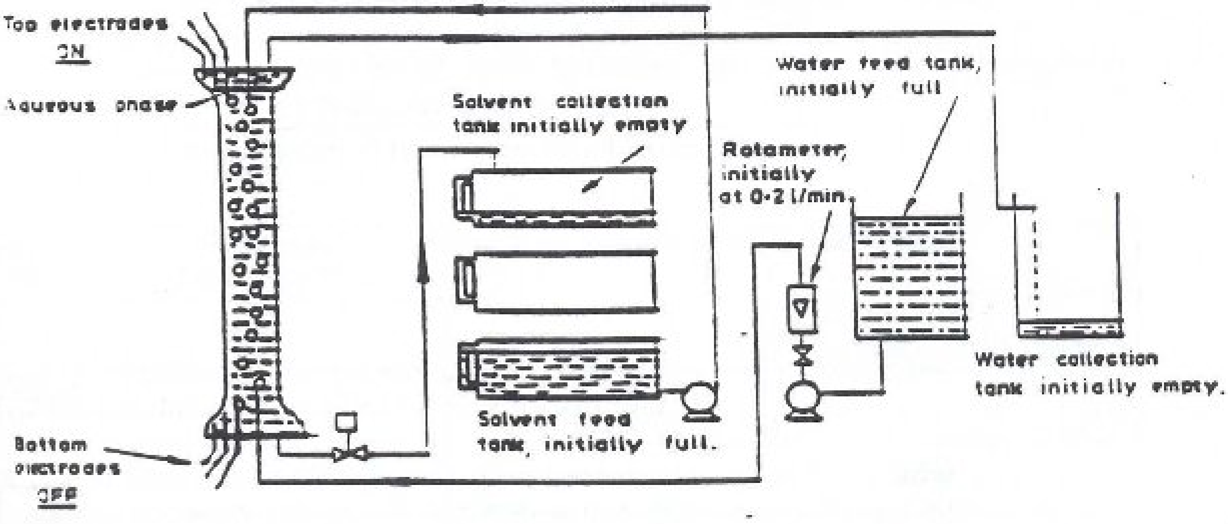
EXPERIMENT D

OBJECT OF EXPERIMENT:

To demonstrate how a mass balance is performed on the extraction column, and to measure the mass transfer coefficient and its variation with flow rate with the organic phase as the continuous medium.

EQUIPMENT SET-UP:

ORGANIC PHASE AS CONTINUOUS MEDIUM



The solvent metering pump is calibrated in percentage of maximum flow which varies slightly from pump to pump. The pump should be calibrated initially by setting F2 to 100%, setting valve V8 to the calibrate position and measuring the flow from the pump, using a measuring cylinder and stopwatch. Calculate the flow rate produced settings of 10% intervals (ml per minute), then plot a graph of ml per minute against percentage of metering pump stroke. Thereafter any selected flow may be obtained by using the graph.

SUMMARY OF THEORY:

The theory is given for the system Trichloroethylene-Propionic Acid-Water.

Let V_w = Water flow rate (l/s)
V_o = Trichloroethylene flow rate (l/s)
X = Propionic Acid concentration in the organic phase (kg/l)
Y = Propionic Acid concentration in the aqueous phase (kg/l)

Subscripts: $_1$ = Top of column
$_2$ = Bottom of column

1. *Mass Balance*

Propionic Acid extracted from the organic phase (raffinate)

$$= V_o (X_1 - X_2)$$

Propionic Acid extracted by the aqueous phase (extract)

$$= V_w (Y_1 - 0)$$

Therefore $V_o(X_1-X_2) = V_w(Y_1-0)$

2. *Extraction Efficiency*

Mass transfer coefficient (based on the raffinate phase)

$$= \frac{\text{Rate of Acid Transfer}}{\text{Volume of Packing x Mean Driving Force}}$$

$$\text{Log mean driving force} = \frac{\Delta x_1 - \Delta x_2}{1n \frac{\Delta x_1}{\Delta x_2}}$$

where ΔX_1 = Driving force at the top of the column = (X_2-0)

ΔX_2 = Driving force at the bottom of the column = $(X_1-X_1^*)$

where X_1^* is the concentration in the organic phase which would be in equilibrium with concentration Y_1 in the aqueous phase. The equilibrium values can be found using the distribution coefficient found in the first experiment.

WARNING Concentrated Sodium Hydroxide can form explosive volatile products when in contact with Trichloroethylene. Ensure that diluted Sodium Hydroxide (NaOH) is used when performing this experiment.

PROCEDURE:

1. Add 100ml of proprionic acid to 10 litres of trichloroethylene. Mix well to ensure an even concentration then fill the organic phase feed tank (bottom tank) with the mixture.
2. Switch the level control off (switch S2 in middle position).
3. Fill the water feed tank with 15 litres of clean de-mineralised water.
4. Start the metering pump and fill the column quickly then regulate to a flow of 0.2 l/min. when the trichloroethylene reaches the top of the packing.

5. Start the water pump regulating the flow to 0.2 l/min. When the water reaches the top electrodes, switch on the top electrodes (switch S2 to 'up' position).

6. Run for 15-20 minutes until steady conditions are obtained, monitor water flow rate during this period to ensure that it remains constant.

7. Take 15ml samples from the feed, raffinate and extract streams.

8. Titrate 10ml of each sample against M/10 NaOH using phenolphthalein as the indicator. (To titrate the feed and raffinate they may need continuous stirring using a magnetic stirrer).

9. Repeat the experiment with both the water and trichloroethylene flow rates being increased to 0.3 l/min.

RESULTS:

Flow rate of Aqueous Phase Flow rate of Organic Phase		
	Titre of M/10 NaOH	Concentration of Propionic Acid kg/l
Feed		
Raffinate		
Extract		
Propionic acid extracted from the organic phase		
Propionic acid extracted from the aqueous phase		
Mass Transfer Coefficient		

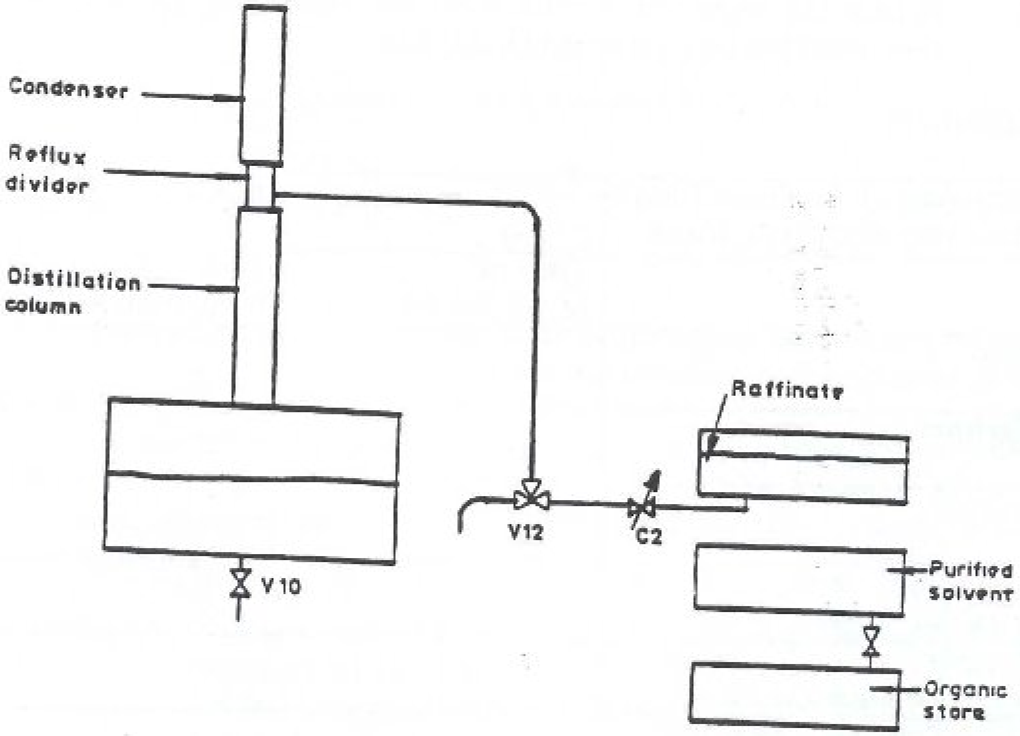
EXPERIMENT E

DEMONSTRATION OF SOLVENT RECOVERY

OBJECT OF EXPERIMENT:

To recover the solvent used in the liquid/liquid extraction experiments using the distillation column.

EQUIPMENT SET-UP:



SUMMARY OF THEORY:

In the previous experiments, water has been used as the solvent to extract propionic acid from trichloroethylene. Solvent recovery can now be demonstrated using the distillation column situated at the rear of the equipment on the left hand side.

The distillation column contains four sieve plates and is equipped with a reflux divider which is operated using valve C2. Regulating this valve will cause different quantities of condensate to be returned to the column as reflux.

PROCEDURE:

On completion of an extraction experiment, trichloroethylene contaminated with propionic acid will be contained in the solvent collecting tank. This batch can be transferred using the drain valve at the base of the tank (V7), into the distillation column. Ensure that the level in the distillation column boiler is at least halfway up the sight glass on the side of the boiler but do not overfill so that the level rises off the gauge.

Make sure the regulator R1 is on the minimum setting of 1 i.e. fully anti-clockwise and then press the red start button which will illuminate. Now turn the regulator clockwise to a setting of say, 7. The cooling water supply to the condenser should now be turned on.

Take a sample of the contaminated trichloroethylene from the sample valve V10. As in the experiment to determine the distribution coefficient, take a 10ml sample of this using a pipette with a rubber bulb and titrate this against M/10 sodium hydroxide solution using phenolphthalein as indicator. (Trichloroethylene 10ml sample will need continuous stirring using a magnetic stirrer).

When the liquid starts to boil, observe the foaming action on the distillation column plates and adjust R1 so that a gentle action takes place. Adjust reflux valve C2 so that not all of the liquid is allowed to fall into the purified solvent tank and some of the condensed liquid is allowed to reflux back into the column.

At 15 minute intervals, take samples of the liquid in the distillation column boiler (valve V10) and the purified organic tank from the flexible pipe on the reflux divider. Do titrations on both samples. Continue to distil until the level of liquid in the boiler gets low enough to operate the float switch which will turn off the power to the heater. The purified organic can be transferred from the holding tank into the organic store tank by using valve V5.

RESULTS:

Sample	Titre of NaOH boiler	Titre of NaOH holding tank	Conc. acid boiler	Conc. acid holding tank
1				
2				
3				
4				
5				

PART 3 Distillation Column UOP3cc

PART 3

Distillation Column
UOP3CC

Introduction to Distillation

Distillation has always been and will continue to be one of the most important industrial processes for separating the different components of a liquid mixture

Laboratory scale distillation columns are needed to provide adequate practical training for student engineers and plant operators in a safe environment. They may also be used to acquire process separation data, of use in full-scale plant design

The UOP3CC allows the study of both batch and continuous distillation, packed or plate column operation, operation under atmospheric pressure or under vacuum, azeotropic distillation, and manual, PID, PLC or computer control of the process. Data logging of the process is also possible.

Description of Distillation Column

All numerical referees relate to the figures (1, 2, 3,4)

The equipment comprises a 50 mm diameter sieve plate column made up of two glass sections (3) and (4) each containing four sieve plates. The columns are separated by a central feed section and arranged vertically for counter-current vapor/liquid flow. Also installed within the framework are:

Reboiler (13)
Two 5 litre feed tanks (5), (6)
Peristaltic type feed pump (7)
Condenser (8)
Bottom (9) and top (10) product tanks
Decanter (11)
Reflux valve (12)
Azeotropic dosing vessel (24)
Vacuum pump (20)

The Reboiler (13) situated at the base of the column is manufactured from 316 stainless steel and incorporators a flameproof immersion type heating element. Either each batch or continuous distillation can be carried out using this reboiler.

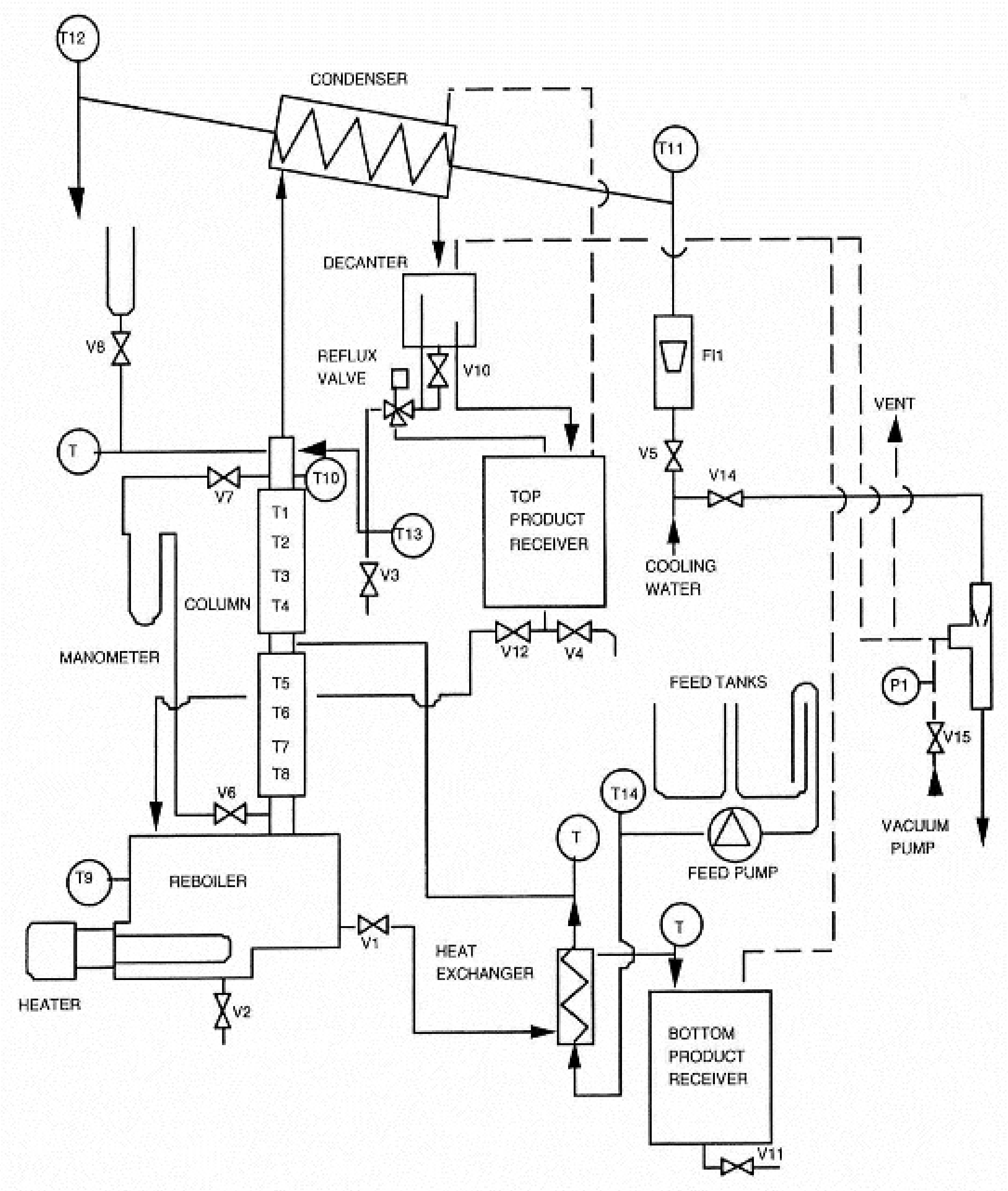
In continous operation, valve (V1) is open and bottom product flows from the reboiler through the bottom product cooler (15) to the bottom product tank (9). It is possible to preheat the feed to the column by directing the feed through a spiral coil in the bottom product cooler where heat is transferred from product leaving the reboiler at the boiling point.

For batch operation, valve (V1) remains closed so that the reboiler can be filled with initial charge (10 to 12 litres) of binary mixture.

A level sensor (17) inside the reboiler protects the heating element from overheating due to low operating level and a sign glass 18 allows the level in the reboiler to be observed.

Feed mixture from either of the feed tanks is pumped by pump (7) to the base, centre or top of the distillation column at connections (A), (B) or (C).

The glass column incorporates a total of eight sieve plates in two sections (3) and (4) each containing four plates. Each plate (D) is located by a central support rod (E) and incorporates a weir (F) and downcomer (G) to create a liquid seal between successive stages.


T12
CONDENSER
T11
DECANTER
V8
REFLUX
VALVE
V10
FI1
VENT
T
V7
T10
V5
V14
T1
T2
T3
T4
T13
TOP
PRODUCT
RECEIVER
V3
COOLING
WATER
COLUMN
MANOMETER
V12
V4
T5
T6
T7
T8
FEED TANKS
P1
V15
V6
T14
T
VACUUM
PUMP
T9
REBOILER
FEED PUMP
V1
HEAT
EXCHANGER
T
HEATER
V2
BOTTOM
PRODUCT
RECEIVER
V11

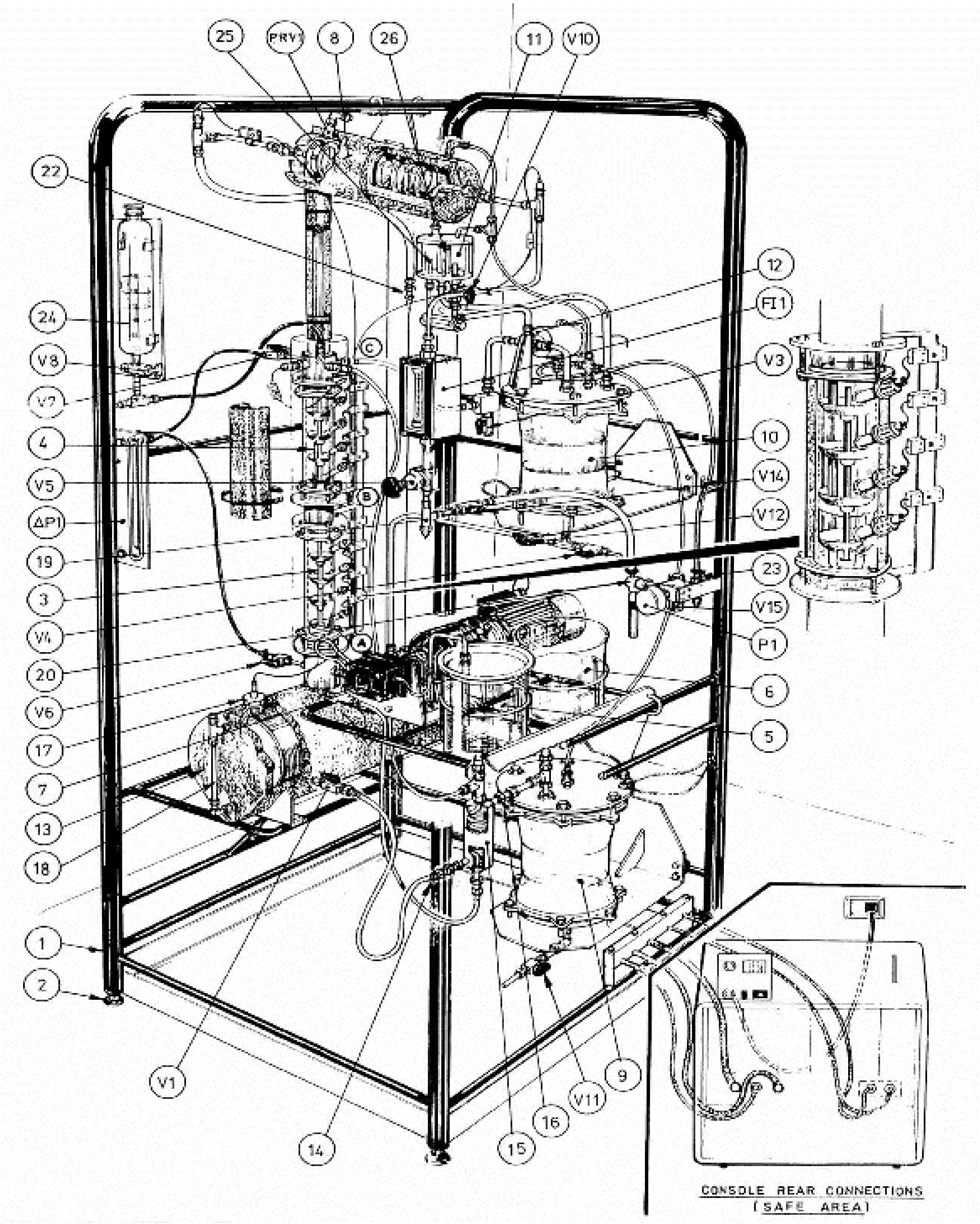


Figure 2: Distillation Column

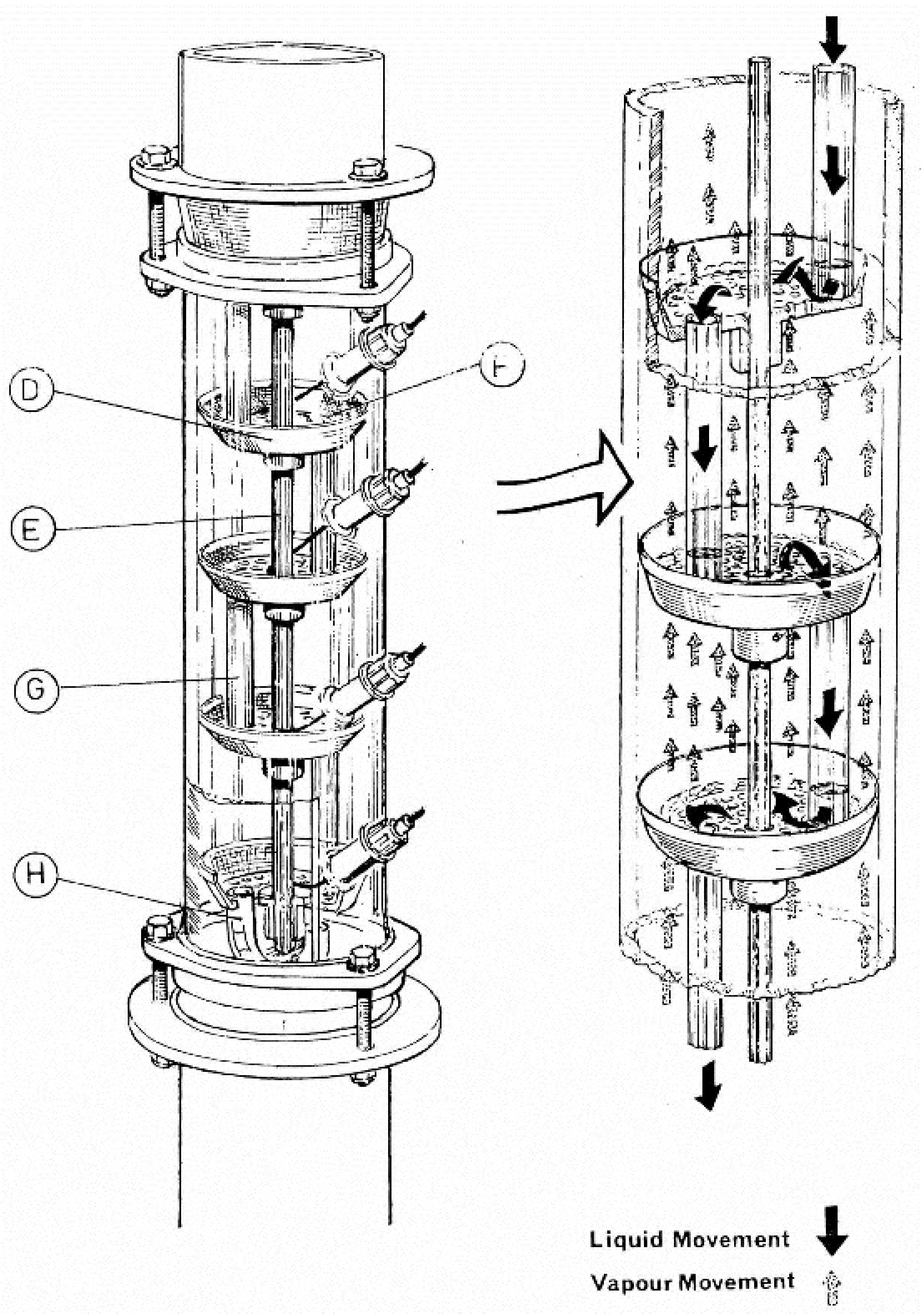


Figure 3

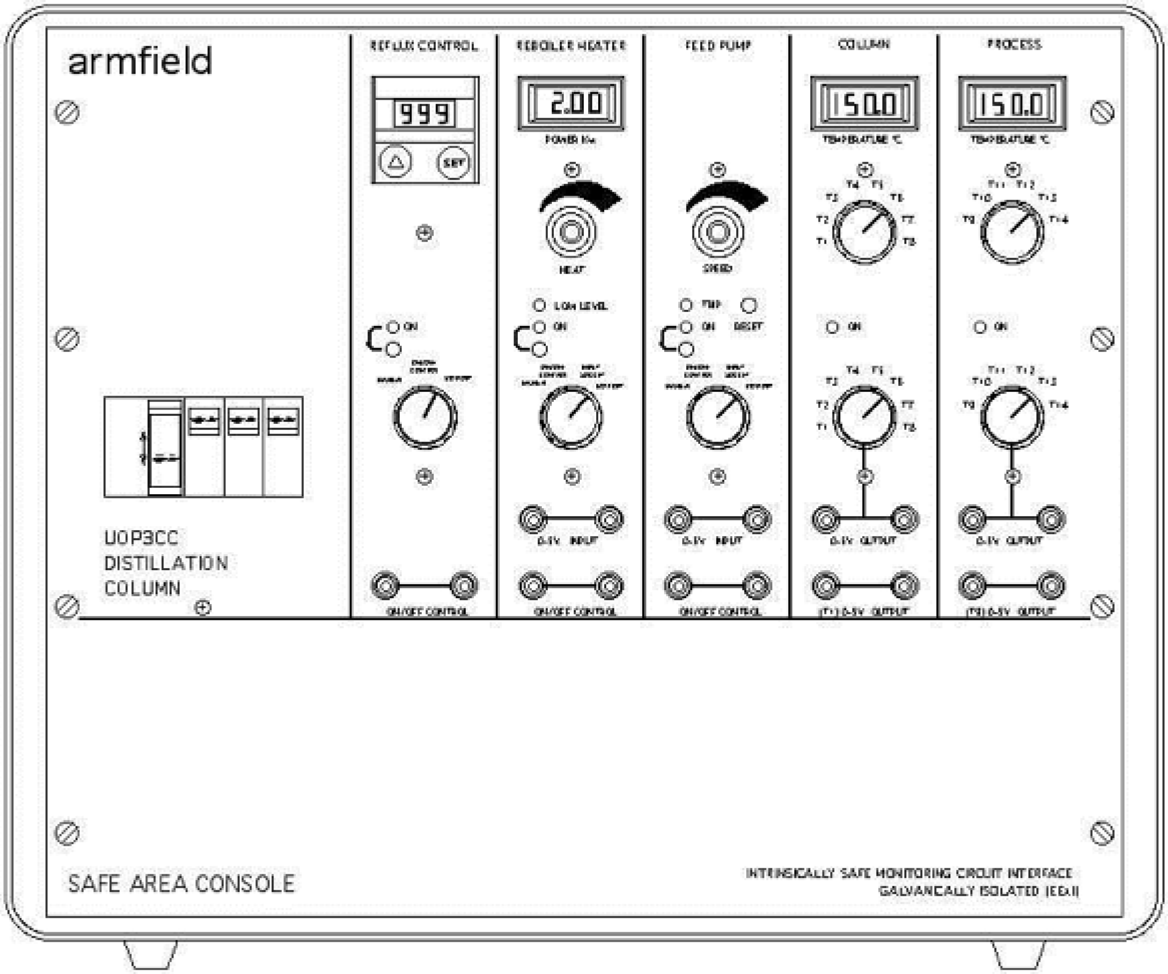


Figure 4: Control Consol

INDEX TO EXPERIMENTS

Batch Distillation

1. To determine the pressure drop over the distillation column for various boil-up rates
2. Use the refractometer for determining mixture compositions
3. To determine the overall column efficiency.
4. Distillation at constant reflux ratio-variation of top and bottom compositions with time.

Continuous Operation

1. Steady state distillation of a binary mixture, McCabe-Thiele/Ponchon Savarit determination of no. of theoretical plates.

2. Effect on column performance of feed plate location.

EXPERIMENT A

BATCH DISTILLATION

Objective

To determine the pressure drop over the distillation column for various boil-up rates

Theory

The total pressure drop across each tray is the sum of that caused by the restriction of the holes in the sieve tray, and that caused by passing through the liquid (foam) on the top of the tray.
As the velocity of the vapors passing up the column increases then so does the overall pressure drop. The velocity is controlled by varying the boil-up rate which is done by varying the power input to the broiler. Under conditions with no liquid present, the sieve trays will behave like an orifice in that the pressure drop will be proportional to the square of the velocity. Due to the fact that there is a liquid head however, this square relationship does not become apparent until the head of liquid has been overcome and foaming is taking place.

Procedure

1. Before starting, make sure all valves on the equipment are closed
2. Open valve V10 on the reflux pipe.
3. Fill the boiler with 10 liters of mixture (methanol & water) to be distilled.
4. Turn on the power to the control panel and set the temperature selector switch to T9 (the temperature in the reboiler).
5. Open valve V5 admitting the cooling water to the condenser at the flow rate on F11 of approximately 3 liters/min.
6. On the control panel turn the power controller for the reboiler heating element anti clockwise until a reading of approximately 0.5 kW is obtained on the digital wattmeter.
7. When the equilibrium condition is reached the boil up rate can be measured by operating valve V3 so that all the condensate is diverted into a measuring cylinder and the time observed to collect a set quantity.
8. Take reading of pressure drops over both the rectifying (top) and the stripping (bottom) sections by opening the valves V6 & V7 on the manometer. (When opening the valves, make sure always to open valve V6 then V7 to prevent vapor from the column to entering the manometer).close the valves in the same order, when finished the pressure drop reading.
9. Set up the boil-up rate in 250 Watt increments up to maximum 1.5 kW by adjusting the boiler heater power controller.

Results

Power (kW)	**Boil-up Rate (litres/hr)**	**Pressur Drop (cm H_2O) Top Bottom Overall**	**Degree of Foaming on Trays**

The comment " **Degree of Foaming on Trays"** should be filled in using descriptive words e.g. None, gentle Localised, Violent Localised, Foaming Gently Over Whole Tray, Foaming Violently Over Whole Tray, Liquid Flooding in Column.

- From the results, plot the curve relating pressure drop as a function of boil-up rate on log/log graph paper.

EXPERIMENT B

BATCH DISTILLATION

Objective

Use the refractometer for determining mixture composition

Theory

It is essential to have a convenient quick method of determining the composition of the binary mixture taken from the various sample points on the distillation column. Such a method involves the use of a refractometer since the refractive index of these mixtures varies with composition.

For the system Methanol/Water, mixtures of known concentration can be made up and their refractive indexes measured. The refractometer measured the critical angle of the liquid under test and each concentration will show a different critical angle.

The refractive index

The ratio of the velocity of light in vacuum to the velocity of light in a medium is referred to as the medium's **refractive index**, denoted by the letter **n**. The velocity of light in a vacuum is 3.0 x 10^8 m/s or about 186,000 miles/s. the index of refraction **n** equals the ratio of the velocities of light in vacuum (c) to that in the medium (v), that is **n = c/v**. Light slows down when it enters a substance, so the refractive index will always be greater than 1.

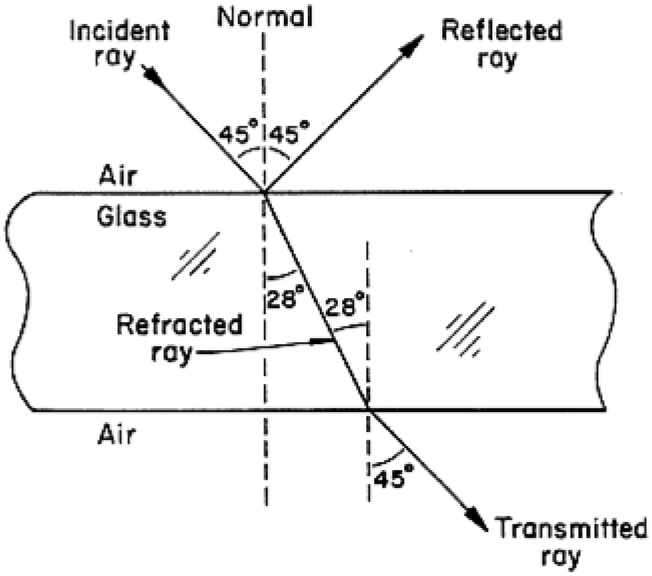


Figure 5. Light incident on a glass plate. The reflected part of the ray is shown along with the light path for the refracted component.

Procedure

1. Measure the refractive index (R.I) of pure methanol and pure water.
2. Make up small quantities of 25 mol percent, 50 mol percent, 75 mol percent methanol and measure their R.I.
3. calculate the volume of constituents to use as follows:

For 25 mol percent methanol, ie. 75 mol percent water

Molecular weight of methanol = 32
Molecular weight of water = 18

Density of methanol = 0.791 g/ml
Density of water = 1 g/ml

$$25 = \frac{\dfrac{Vol_{methanol} X \rho_{Methanol} \mathrm{X}100}{M.W_{Methanol}}}{\dfrac{Vol_{Methanol} \mathrm{X} \rho_{Methanol}}{M.W_{Methanol}} + \dfrac{Vol_{Water} \mathrm{X} \rho_{Water}}{M.W_{Water}}}$$

Thus

$$25 = \frac{100}{1 + \frac{Vol_{Water}}{Vol_{Methanol}} X \frac{M.W_{Methanol}}{M.W_{Water}} X \frac{\rho_{Water}}{\rho_{Methanol}}}$$

$$\frac{\mathrm{Vol}_{\mathrm{Water}}}{\mathrm{Vol}_{\mathrm{Methanol}}} = 3\,\mathrm{X}\,\frac{\mathrm{M.W}_{\mathrm{water}}}{\mathrm{M.W}_{\mathrm{Methanol}}}\,\mathrm{X}\,\frac{\rho_{\mathrm{Methanol}}}{\rho_{\mathrm{Water}}}$$

$$\frac{Vol_{Water}}{Vol_{Methanol}} = 3\,\mathrm{X}\,\frac{18}{32}\,\mathrm{X}\,\frac{0.791}{1}$$

$$1\,\mathrm{Vol}_{\mathrm{Water}} = 1.3348\,\mathrm{Vol}_{\mathrm{Methanol}}$$

Results

Methanol

Concentration	**R.I.**
100%	
75%	
50%	
25%	
0%	

1. Plot graph of refractive index versus mol percent methanol in methanol/water mixture.

EXPERIMENT C

BATCH DISTILLATION

Objective

To determine the overall column efficiency at varying boil-up rates

Equipment Set up

The equipment will be set up to operate at total reflux so that the charge of feed mixture can be loaded directly into the reboiler.

Equipment

1. Distillation Column
2. Refractometer
3. 250 ml measuring cylinder graduated in mls.
4. stop watch

Chemicals/materials

1. Methanol
2. Deionized Water

Theory

To calculate the number of theoretical plates for a given separation at total; reflux fenske developed the following formula:

$$n+1=\frac{\log\left[\left(\frac{x_A}{x_B}\right)_d\cdot\left(\frac{x_B}{x_A}\right)_b\right]}{\log\left(\alpha_{AB}\right)av}$$

Where n = number of theoretical plates
x_A = mole fraction of more volatile component
x_B = mole fraction of more volatile component
α_{av} = average relative volatility
Subscripts d , b indicate distillate and bottom respectively

$$\alpha_{av}=\sqrt{\alpha_d.\alpha_b}$$

the efficiency is given by,

$$\mathrm{E}=\frac{\text{Number of theoritical plates}}{\text{Number of actual plates}}\mathrm{X}\,100\%$$

Knowing the composition of distillate and bottom and the corresponding volatilities, the column efficiency can be determined.

Procedure

1. Before starting, make sure all valves on the equipment are closed
2. Open valve V10 on the reflux pipe.
3. Fill the boiler with 10 liters of mixture (methanol & water) to be distilled.
4. Turn on the power to the control panel and set the temperature selector switch to T9 (the temperature in the reboiler).

5. Open valve V5 admitting the cooling water to the condenser at the flow rate on F11 of approximately 3 liters/min.
6. On the control panel turn the power controller for the reboiler heating element anti clockwise until a reading of approximately 0.75 kW is obtained on the digital wattmeter.
7. When the equilibrium condition is reached the boil up rate can be measured by operating valve V3 so that all the condensate is diverted into a measuring cylinder and the time observed to collect a set quantity.
8. Take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line.
9. Record the refractive index for the taken overhead sample.
10. Take a sample of the bottom through valve V2.
11. Record the refractive index for the taken bottom sample.
12. Repeat this procedure for several different boil-up rates to cover the operating range of the column.
13. Determine the concentrations of the components in the taken samples by using calibration graph (see experiment B).

Results

TABLE OF RESULTS

Boil-up Rate (l/hr)	**Rectifying**				**Stripping**			
	R.I.	**(X_A)d**	**Temperature (ºC)**		**R.I.**	**(X_A)b**	**Temperature (ºC)**	
			T_1				T_5	
			T_2				T_6	

			T_3				T_7	
			T_4				T_8	
	Average				**Average**			
			T_1				T_5	
			T_2				T_6	
			T_3				T_7	
			T_4				T_8	
	Average				**Average**			

1. Calculate the number of theoretical plates.

EXPERIMENT D

BATCH DISTILLATION

Objective

To carry out a distillation at constant reflux ratio-varying top and bottom compositions with time.

Equipment Set up

- The equipment will be set up to operate at total reflux so that the charge of feed mixture can be loaded directly into the reboiler.

Equipment

1. Distillation Column
2. Refractometer
3. 250 ml measuring cylinder graduated in mls.
4. stop watch

Chemicals/materials

1. Methanol
2. Deionized Water

Theory

Since there is no feed, no bottom product, the liquid flow in the column is equal to the vapor flow in the column.

$$V = L$$

A material balance over the M.V.C. (most volatile component) gives:

$$V y_n = L x_{n+1}$$

Since V = L this gives

$$y_n = x_{n+1}$$

Procedure

1. Before starting, make sure all valves on the equipment are closed
2. Open valve V10 on the reflux pipe.
3. Fill the boiler with 10 liters of mixture (methanol & water) to be distilled.
4. Turn on the power to the control panel and set the temperature selector switch to T9 (the temperature in the reboiler).
5. Open valve V5 admitting the cooling water to the condenser at the flow rate on F11 of approximately 3 liters/min.

6. On the control panel turn the power controller for the reboiler heating element anti clockwise until a reading of approximately 0.75 kW is obtained on the digital wattmeter.
7. Set the reflux controller to the desired value i.e start with 5:1, meaning 5 sec back to column and 1 sec to top product receiver.
8. When the equilibrium condition is reached the boil up rate can be measured by operating valve V3 so that all the condensate is diverted into a measuring cylinder and the time observed to collect a set quantity.
9. Take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line.
10. Record the refractive index for the taken overhead sample.
11. Take a sample of the bottom through valve V2.
12. Record the refractive index for the taken bottom sample.
13. Record the temperatures T8 - T1 to calculate the average column temperature.
14. Repeat this procedure for different reflux ratio.
15. Repeat the whole procedure for several different boil-up rates to cover the operating range of the column.
16. Determine the concentrations of the components in the taken samples by using calibration graph (see experiment B).

Results

TABLE OF RESULTS

Boil-up Rate (l/hr)	**Rectifying**			**Stripping**		
	Reflux Ratio =					
	R.I.	**$(X_A)d$**	**Temperature (ºC)**	**R.I.**	**$(X_A)b$**	**Temperature (ºC)**

			T_1				T_5	
			T_2				T_6	
			T_3				T_7	
			T_4				T_8	
	Average				**Average**			
	T Column average (ºC) =							
	Reflux Ratio =							
	R.I.	**$(X_A)d$**	**Temperature (ºC)**		**R.I.**	**$(X_A)b$**	**Temperature (ºC)**	
			T_1				T_5	
			T_2				T_6	
			T_3				T_7	
			T_4				T_8	
	Average				**Average**			

EXPERIMENT E

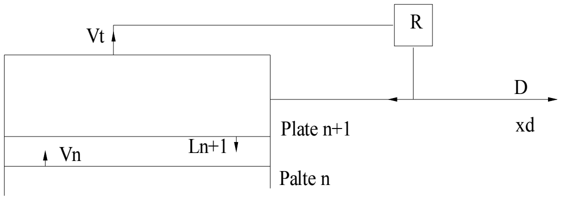
CONTINUOUS DISTILLATION

Objective

Steady state distillation of a binary mixture under continuous operation.

Equipment Set up

- The equipment will be set up to operate at total reflux so that the charge of feed mixture can be loaded directly into the reboiler.



Requirements

1. Distillation Column
2. Refractometer
3. 250 ml measuring cylinder graduated in mls.
4. stop watch
5. Calibration curve for the feed pump.
6. Calibration graph for refractometer.
7. Equilibrium data for methanol/water at atmospheric pressure.

Chemicals/materials

1. Methanol
2. Deionized Water

Theory

Calculation of number of plates using the LEWIS-SOREL method

Material Balance of top of column

$$V_n = L_{n+1} + D$$

With respect to M.V.C. this becomes

$$y_n V_n = L_{n+1} x_{n+1} + D x_d$$

$$y_n = \frac{L_{n+1}}{V} x_{n+1} + \frac{D}{V_n} x_d$$

Since the liquid overflow is constant $L_n = L_{n+1}$

$$y_n = \frac{L_{n+1}}{V_n} x_{n+1} + \frac{D}{V_n} x_d$$

Material Balance of bottom of column.

$$V_m = L_{m+1} - W$$

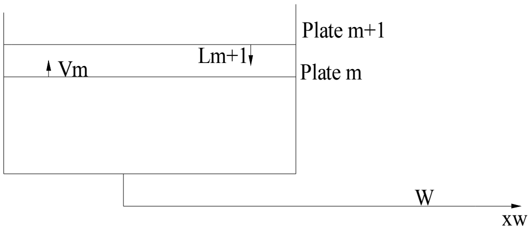
With respect to M.V.C. this becomes

$$y_m V_m = L_{m+1} x_{m+1} - W x_w$$

$$y_m = \frac{L_{m+1}}{V_m} x_{m+1} - \frac{W}{V_m} x_w$$

Since the liquid overflow is constant $Lm = L_{m+1}$

$$y_m = \frac{L_{m+1}}{V_m} x_{m+1} - \frac{W}{V_m} x_w$$



Equations (1) and (2) combined with the equilibrium curve can be used to calculate the composition on the various plates working from the condenser down to the still. The plate which has a composition nearest to that of the feed should be used as the feed plate.
Consequently the number of theoretical plates and position of entry for the feed can be calculated.

Procedure

1. the feed to the column must be admitted at the mid point (onto try 5)
2. Turn on the power to the control panel and set the temperature selector switch to T9 (the temperature in the reboiler).

3. Open valve V5 admitting the cooling water to the condenser at the flow rate on F11 of approximately 3 liters/min.
4. On the control panel turn the power controller for the reboiler heating element anti clockwise until a reading of approximately 0.75 kW is obtained on the digital wattmeter.
5. Start the experiment with total reflux.
6. Before switching on the reflux switch, Set the reflux ratio to 5:1, meaning 5 sec back to column and 1 sec to top product receiver.
7. When the column has stabilized at total reflux, the flow of feed and the reflux can be started at the same time.
8. It is advisable to set a feed flow 2 litres/hr.
9. After feeding approximately 3 litres take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line ie . partially open V3 to leave a small amount of liquid in the line all the time.
10. Take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line.
11. Record the refractive index for the taken overhead sample.
12. Take a sample of the bottom through valve V2.
13. Record the refractive index for the taken bottom sample.
14. Record the temperatures T8 - T1 to calculate the average column temperature.
15. repeat the sample taking a further three times during the experiment (before feed runs out)

Table of results

Power (kW) =
Feed Position =

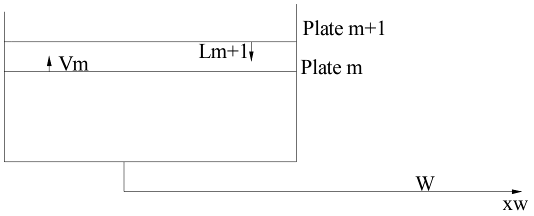
Feed Flow Rate (l/hr) =				
Reflux Ratio =				
	Rectifying		**Stripping**	
	R.I.	**(X_A)d**	**R.I.**	**(X_A)b**
Trial # 1				
Trial # 2				
Trial # 3				
Trial # 4				
Trial # 5				

EXPERIMENT F

CONTINUOUS DISTILLATION

Objective

Effect of varying the feed position under continuous operation.



Equipment Set up

Requirements

8. Distillation Column
9. Refractometer
10. 250 ml measuring cylinder graduated in mls.
11. stop watch
12. Calibration curve for the feed pump.
13. Calibration graph for refractometer.
14. Equilibrium data for methanol/water at atmospheric pressure.

Chemicals/materials

1. Methanol
2. Deionized Water

Theory

Calculation of number of plates using the LEWIS-SOREL method

Material Balance of top of column

$$V_n = L_{n+1} + D - F$$

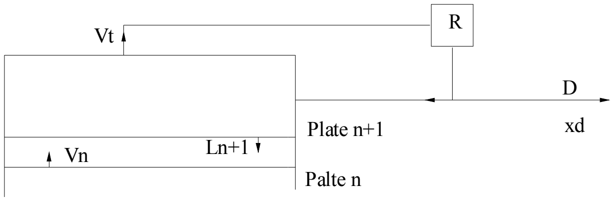
With respect to M.V.C. this becomes

$$y_n V_n = \overrightarrow{L_{n+1}x_{n+1} + Dx_d} - Fx_F$$

$$y_n = \frac{L_{n+1}x_{n+1}}{V_n} + \frac{D}{V_n}x_d - \frac{Fx_F}{V_n}$$

Since the liquid overflow is constant $L_n = L_{n+1}$

$$y_n = \frac{L_{n+1}x_{n+1}}{V_n} + \frac{Dx_d}{V_n} - \frac{Fx_F}{V_n}$$



Material Balance of bottom of column.

$$V_m = L_{m+1} - W$$

With respect to M.V.C. this becomes

$$y_m V_m = L_{m+1} x_{m+1} - W x_w$$

$$y_m = \frac{L_{m+1}}{V_m} x_{m+1} - \frac{W}{V_m} x_w$$

Since the liquid overflow is constant $Lm = L_{m+1}$

$$y_m = \frac{L_{m+1}}{V_m} x_{m+1} - \frac{W}{V_m} x_w$$

As the feed is introduced in the top of the column $L_m = L_n$ and also $V_m = V_n$

Procedure

1. The feed to the column must be admitted above the top plate.
2. Turn on the power to the control panel and set the temperature selector switch to T9 (the temperature in the reboiler).
3. Open valve V5 admitting the cooling water to the condenser at the flow rate on F11 of approximately 3 liters/min.
4. On the control panel turn the power controller for the reboiler heating element anti clockwise until a reading of approximately 0.75 kW is obtained on the digital wattmeter.
5. Start the experiment with total reflux.
6. Before switching on the reflux switch, Set the reflux ratio to 5:1, meaning 5 sec back to column and 1 sec to top product receiver.
7. When the column has stabilized at total reflux, the flow of feed and the reflux can be started at the same time.
8. It is advisable to set a feed flow 2 litres/hr.
9. After feeding approximately 3 litres take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line ie . partially open V3 to leave a small amount of liquid in the line all the time.
10. Take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line.

11. Record the refractive index for the taken overhead sample.
12. Take a sample of the bottom through valve V2.
13. Record the refractive index for the taken bottom sample.
14. Record the temperatures T8 - T1 to calculate the average column temperature.
15. repeat the sample taking a further three times during the experiment (before feed runs out)

Table of results

Power (kW) =				
Feed Position =				
Feed Flow Rate (l/hr) =				
Reflux Ratio =				
	Rectifying		**Stripping**	
	R.I.	**$(X_A)d$**	**R.I.**	**$(X_A)b$**
Trial # 1				
Trial # 2				
Trial # 3				
Trial # 4				
Trial # 5				

PART 4

Single Stage Evaporator

The falling film evaporators are used industrially to concentrate solutions, especially solutions with heat sensitive components.

Over the past 40 years the falling film evaporator has practically replaced the forced recirculation evaporator. This type of evaporator is desirable, from a product point of view, as it can be operate with very low temperature differences between the heating media and the boiling liquid, short holding time (typically just a few seconds) and a good control of the temperature of the product to be concentrated.

The falling film evaporator has a very small hold up of liquid. This is advantageous during operation (quick start up high response to changing of operative conditions), avoiding browning of sensitive products and during cleaning requiring a minimum of cleaning liquid.

Further, the amount of product in the evaporator is reduced and the surface from which the evaporation takes place is increased.

Falling film evaporators are highly responsive to alterations of parameters such as energy supply, vacuum, feed rate, concentrations, etc. When well controlled, they can produce a very consistent concentrated product.

These characteristics make the falling film evaporator particularly suitable for heat-sensitive products, and it is today the most frequently used type of evaporator.

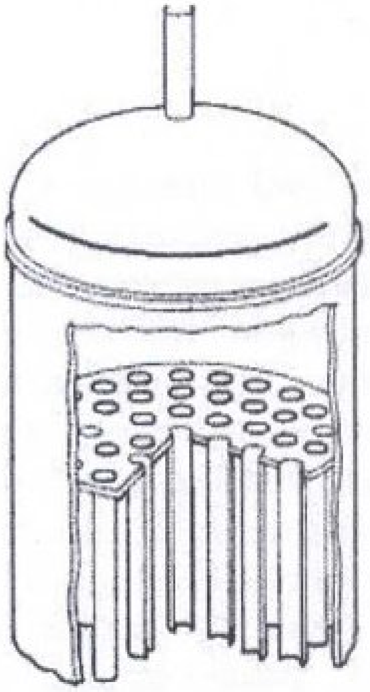
The evaporator consists of a number of tubes built together side by side. At each end the tubes are fixed to tube plates, and finally the tube bundle is enclosed by a jacket.

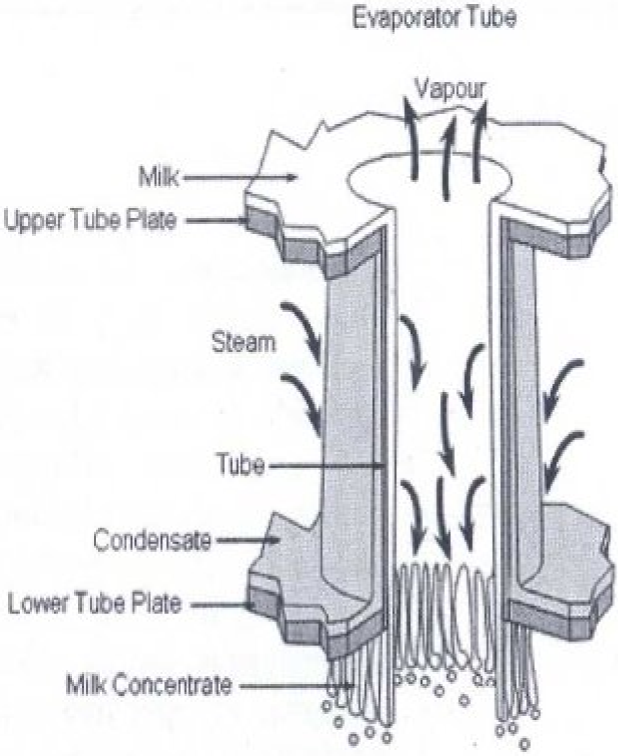
The liquid to be evaporated is evenly distributed on the inner surface of a tube and will flow downwards forming a thin film, from which the boiling/evaporation will take place because of the heat applied by the steam. This gravity-induced downward movement is increasingly augmented by the counter-current vapour flow.

The steam will condense and flow downwards on the outer surface of the tube.

The steam is introduced through the jacket. The space between the tubes is thus forming the heating section. The inner side of the tubes is called the boiling section. Together they form the so-called calandria.

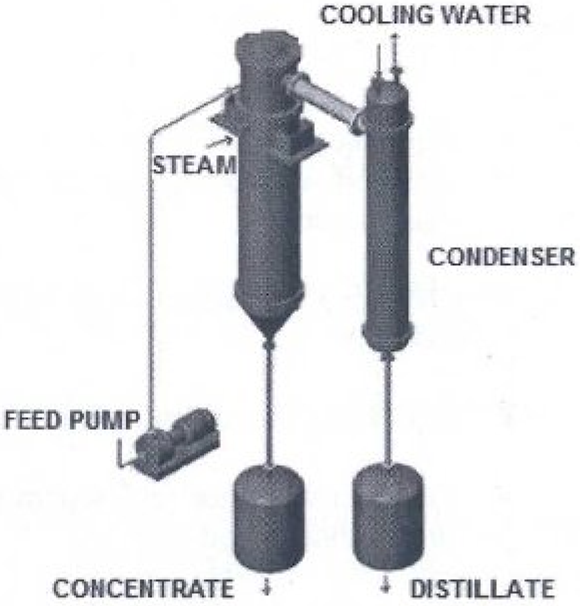
The concentrated liquid leaves the calandria at the bottom part and the vapour leaves at the top. The heating steam, which condenses on the outer surface of the tubes, is collected as condensate at the bottom part of the heating section, from where it is discharged by means of a steam trap.





The pressure in the tubes may be lower than atmospheric pressure, in order to depress the solution's boiling point.

Falling film evaporators are common in the manufacture of concentrated orange juice and condensed milk. They are attractive as a concentration process because of their energy efficiency and thermal gentleness.



1.2 Description

The system is installed within a stainless steel framework and the main components are a stainless steel falling film evaporator (E1) with a stainless steel condenser (E2).

The solution to be concentrated is contained in a glass tank (D1) and it is pumped at the top of evaporator using a metering pump (G1).

The concentrated product is cooled in an exchanger (E3) and stored in a glass tank (D2).

The vapour leaves the evaporator from the top and it is condensed in the condenser (E2) and collected in a glass tank (D3).

The system can operate under vacuum via a liquid ring vacuum pump connected with the condenser (E2).

The steam flow rate is measured, at the inlet of the evaporator jacket, using a calibrated orifice connected with a differential pressure transmitter (FT1).

The steam flow rate, and indirectly the steam pressure in the evaporator jacket, can be adjusted using a pneumatic valve (FV1)

In the low part of the jacket of the evaporator, there is a steam trap connected with a graduated tank (D4) to measure the condensate flow rate.

A safety valve (PSV1) protects the evaporator jacket from excessive pressures due to wrong operations.

A CIP pump (G2) allows to clean the piping, the evaporator and the tanks with cleaning solution.

1.3 Technical characteristics

- Metering pump in stainless steel AISI 316, range 0÷24 l/h, equipped with pneumatic actuator driving with 0.2÷1 bar signal, code G1
- CIP pump in stainless steel AISI 316, 30 l/min @ 2 bar, code G2
- Liquid ring vacuum pump, code G~~2~~ 3
- Air-water separator for vacuum pump, stainless steel AISI 304 execution, code D5
- Feed tank in borosilicate glass, capacity of 25 l, code D1
- Collection tank for the concentrated product in borosilicate glass, capacity of 10 l, code D2
- Collection tank for the condensed solvent in borosilicate glass, capacity of 10 l, code D3
- Graduated borosilicate glass collection tank for the condensed steam, capacity of 1 l, code D4
- Steam trap in stainless steel AISI 304, code SC
- Falling-film evaporator, stainless steel AISI 316 execution on the tube side and stainless steel AISI 304 on the shell side, exchange surface of 0.27 m^2, code E1
- Safety valve for steam, 2 bar, code PSV1
- Shell-and-tube condenser, stainless steel AISI 316 execution on the tube side and stainless steel AISI 304 on the shell side, exchange surface of 1.1 m^2, code E2
- Tube-tube heat exchanger in stainless steel AISI 304, code E3
- Bourdon pressure gages, range 0÷4 bar, code PI1
- Bourdon pressure gage, range of 0÷1.6 bar, code PI2
- Bourdon vacuum gage, range of 0÷-1 bar, code PI3

- Flow rate steam transmitter, differential pressure type, stainless steel AISI 316 execution, range 0 to 1000 mm H_2O, output signal of 4 to 20 mA proportional to 0 ÷15 kg/h of steam at 4 bar; code FT1

- Calibrated orifice for steam flow rate measure in stainless steel AISI 304

- Steam flow rate pneumatic control valve, stainless steel AISI 316 execution, Cv = 0.32, code FV1

- Variable area flow meter for condenser cooling water, code FI2

- 6 RTD's Pt 100, with sheath of stainless steel AISI 316, code TI1, TI2, TI3, TI4, TI5 and TI6

- 6 electronic temperature indicators, range 0÷200°C

- Absolute pressure transmitter, stainless steel AISI 316 execution, range 0÷1000 mbar, code PI3

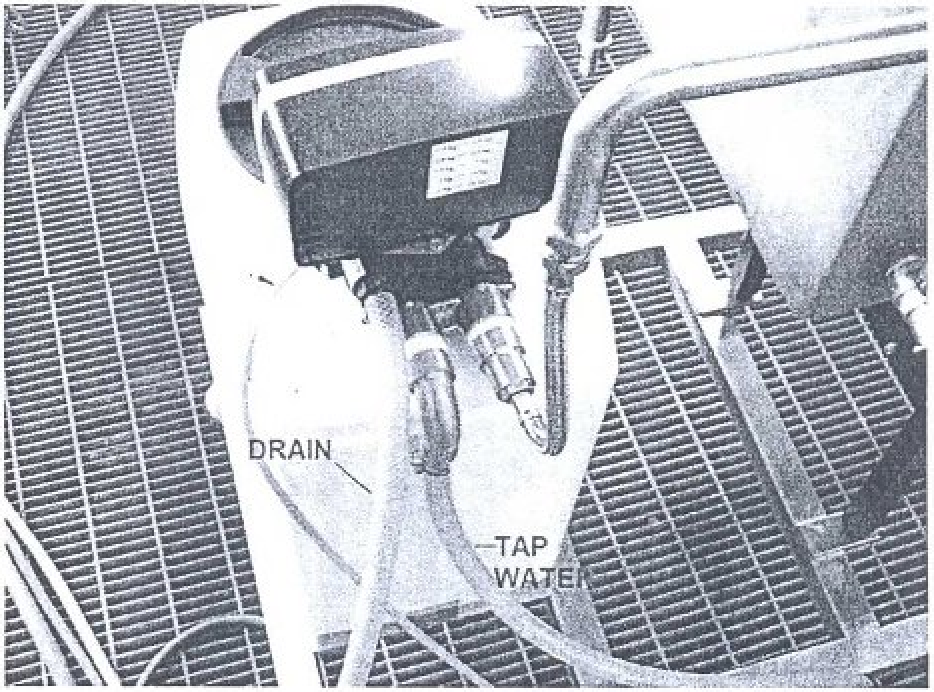
- Vacuum pump solenoid valve, code EV1

- Electric switchboard, protection level IP 55, with synoptic of the plant, E.L.C.B and interface modules for the connection of the plant with a P.C. via serial cable

- Data logging software for Windows

- Connecting lines and valves of stainless steel AISI 304 and 316

- Connect the softener to tap water and to a drain (see picture below) using the rubber pipe and the metal hose clamps supplied

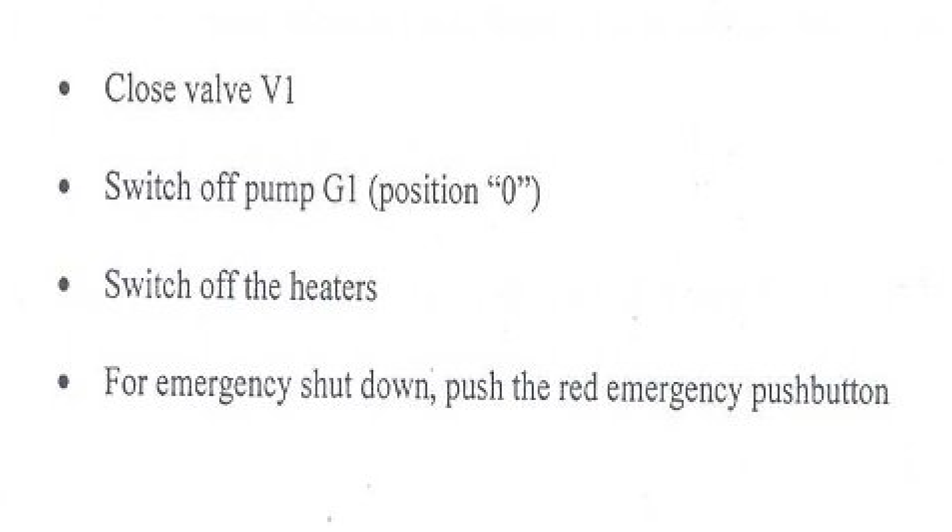


- Connect the valve V8 of the steam generator to a drain
- Open valves V4, V5, V6, V7
- Close valve V1, V2, V3, V8
- Connect the softener to the electrical supply: single-phase + G
- Connect the steam generator to the electrical supply: 3-phase + N +G, P = 16 kW
- Drain the generator opening valve V8 and V9 until to the level of the water is at the medium of the sight glass of the level gauge LG1
- Close valve V8 and V9

Start Up

- Turn on the main switch.
- Press the pushbutton "RESET"
- Switch the selector of pump G1 in position "AUT"
- Switch on the heaters
- If the heater does not turn on, press reset and try again.
- Wait that the pressure on manometer increase up to 1 bar
- Discharge the air opening partially valve V9
- Repeat the operation till to steam exits through the valve V9
- Close valve V9
- When the pressure on manometer PI1 is about 4 bar, open the valve V1

Shut Down



- Close valve V1
- Switch off pump G1 (position "0")
- Switch off the heaters
- For emergency shut down, push the red emergency pushbutton

2. Operating instruction:

2.1 Commissioning

- Close the valves V2, V3, V4, V5, V6, V7, V8, V9, V13, V15, V16, V17, V20 and V21
- Open the valves V1, V12, V14, V18 and V19
- Open partially the valves V10, V11, V22 and V23
- Connect the plant to the electrical supply: three-phase + N +G, P=3 kW
- Connect the plant to the tap water and to a water drain (see plant synoptic and labels on the pipe) using the rubber pipe and the metal hose clamps supplied
- Connect the pressure reducers under the switchboard to the compressed air line (6bar) using the pipe supplied and set the pressure following the labels
- Open the main air supply
- Close the pneumatic valve FV1 using the related knop (Ga1 flow control) positioned on the switchboard (rotating clockwise = increase the valve opening, anti clockwise = decrease the valve opening); the pressure on the related manometer is proportional to the degree of opening of the valve: 0.5 bar = valve completely close, 1 bar = valve fully open
- Connect valve V6 with the steam line using the flexible pipe for steam supplied: the steam pressure must be at maximum 4.5 bar (max. consumption about 20 kg/h)
- Open the main feed steam valve (V1 on steam generator)
- Open the main water flow line
- Set the cooling water flow rate to the condenser E2 at 150 l/h about using valve V10
- Insert the E.L.C.B (الامان)
- Pull the emergency knob out
- Press the pushbutton “ START”
- Fill the feeding tank D1 with the solution to be concentrated
- Open partially valve V22
- Start the vacuum pump G3 (switch to 1).
- Control the residual preesure, at -0.7 bar (=300 mbar) for example, with the valve V22: closing the valve the vacuum increase
- ملاحظة: للتاكد من ان المضخة سليمة بدون تنفيس يمكن اغلاق صممام 22 ومن ثم طفي مضخة3 حيث عندها يجب ان لاتزيد قراءةPI3
- Open the valve V6
- Open valves V7 and V8
- Open the valve FV1 (steam flow control) at 50% (detemined by needle position on the red FV1) using the related knop on the switchboard

- Adjust the steam flowrate using valve FV1

- The system is in stationary condition only when the condensate solvent starts to fall in tank D3; drain the solution collected until this moment in tank D2 (see below the drain instruction)

- If the temperature of the condensate is too high, increase the flow rate of water to the cooling exchanger (E3) opening the valve V11

- To increase the concentration of the product, increase the steam flow rate to the primary evaporator or decrease the feeding flow rate or increase the vacuum; adjust the vacuum very slowly (using valve V22) to avoid dragging of solvent in the tank D3

- To drain the tank D2, operate as follows:

- close the valves V12 and V14
- open the valve V13
- open the valve V16

- To enable the tank D2 again, operate as follows:

- close the valve V16

- close the valve V13
- open the valves V14 slowly and, when the pressure is stabilized, open the valve V12

- To drain the tank D3, operate as follows:

- close the valves V18 and V19
- open the valve V20
- open the valve V21

- To enable the tank D3 again, operate as follows:

- close the valve V21
- close the valve V20
- open the valves V18 slowly and, when the pressure is stabilized, open the valve V19

2.3 Shut down

- Close the valve V6

close main steam line (V1 on generator) →

- Close the valve FV1
- Stop the pump G1 turning the switch to 0
- Stop the vacuum pump G~~2~~3 turning the switch to 0
- Open the valve V22
- Drain the tank D1
- Drain the tank D2
- Drain the tank D3
- Drain the tank D4
- Drain the tank D5
- Drain the piping using the drain valves
- **Clean the plant at the end of each working cycle following the operating instruction of previous chapter**
- Disconnect the power supply
- Close the main valve of the tap water
- Close the main valve of the compressed air

2.4 Emergency stop

- Press the emergency button

2.5 Cleaning operations

IMPORTANT: During the washing operations, use safety gloves and eyewear.

If the product treated is milk, the plant must be washed, **AT THE END OF EACH WORKING CYCLE**, as follows:

- Fill tank D1 with clean tap water
- Open valves V13 and V20
- Open partially valves V5, V15, V17
- Close valve V11
- Start the pump G2 and adjust the flow rate of cleaning solution using:
    - valve V5 to clean the evaporator E1
    - valve V15 to clean the tank D2
    - valve V17 to clean the tank D3
- When the tank D1 is almost empty, stop the pump G2
- Drain the tank D2 and D3
- Fill tank D1 with $NaOH/H_2O$ (1 – 2 % wt) solution
- Close valve V17
- Start the pump G2 and adjust the flow rate of cleaning solution using valve V5, V15 and V17
- Close valve V12 and fill the evaporator E1 up to the sight glass
- Stop pump G2
- Open the valve FV1 and heat at 70 °C (TI2) for 20 minutes
- Drain the evaporator E1
- Start pump G2 and wash the evaporator
- When the tank D1 is almost empty, stop the pump G1
- Drain the tank D2
- Repeat a cleaning cycle using tap water
- Repeat the cleaning operation using a HNO_3/H_2O (0.8%) solution at 70 °C
- Repeat the cleaning operation using water till to the pH of water collected in the tank D2 is about 7
- Discharge the tanks and the piping

If the product treated is citrus juice, water sugar solution etc. (that is solution easy to wash), the plant must be washed, **AT THE END OF EACH WORKING CYCLE**, with a cleaning cycle with water at 70 °C

Note: For the cleaning operation, it is possible to use commercial detergent for food machinery but the above parameter (concentration and composition) must always be fulfilled.
This product must be compatible with stainless steel.

- Any maintenance operation must be carried out by authorized and skilled operator
- Disconnect the voltage before any maintenance operation
- Keep attention to the hot surfaces (evaporator E1, steam pipe etc.)
- Always wear leather gloves during the operations on steam pipe etc.
- During the washing operations, use safety gloves and eyewear.

4. EXPERIMENTS

4.1 Concentration of a sugar solution

1) Concentration of a sugar solution

- Prepare 10 litres of a 5% (g/100 ml) distilled water and sugar solution (sucrose, $C_{12}H_{22}O_{11}$)
- Trace, using a refractometer, a calibration curve for water/sugar solution with concentration between 0 and 50 % (wt/wt)

g/100 ml	0	5	10	15	20	30	40	50
n_D	1.331	1.3385	1.33450	1.3520	1.3590	1.3735	1.3850	1.4000

Prepare calibration curve n_D vs. concentration g/100 ml

- Start the plant and, in stationary conditions (generally after 10 – min), calculate the energy balance, mass balance and the overall heat transfer coefficient

- The operative conditions could be the following:

Feeding flow rate, F_i
Feeding concentration
Steam flow rate, FI1
Steam pressure, PI1
Steam pressure, PI2
Working pressure, PI3
Cooling water flow rate, FI2

Determination of steam consumption

Energy balance on evaporator

Total heat

$Q_T = FI1 \times \Delta H_e (PI3) = 4.8 \times 540 = 2592$ kcal/h

Sensible heat

$Q_s = F_i \times c_p \times (TI2 - TI1)$

Heat for evaporation = $Q_T - Q_s$

Efficiency = $(Q_T - Q_s) / Q_T$

Heat balance on condenser

Total heat

$Q = FI2 \times c_{p(H2O)} \times (TI6 - TI5)$

Evaporation rate $E = Q/\Delta H_e$ (PI3)

Mass balance

Inlet concentration $c_{in} = 5\%$
Outlet concentration c_{out} (to be calculated via refraction index)

$c_{in} \times F_i \approx c_{out} (F_i - E)$

4.2 Concentration of water/glicol ethylenic solution

2) Concentration of a water/glicol ethylenic solution

- Repeat the experiment n°1 using a 5% water/glicol ethylenic solution

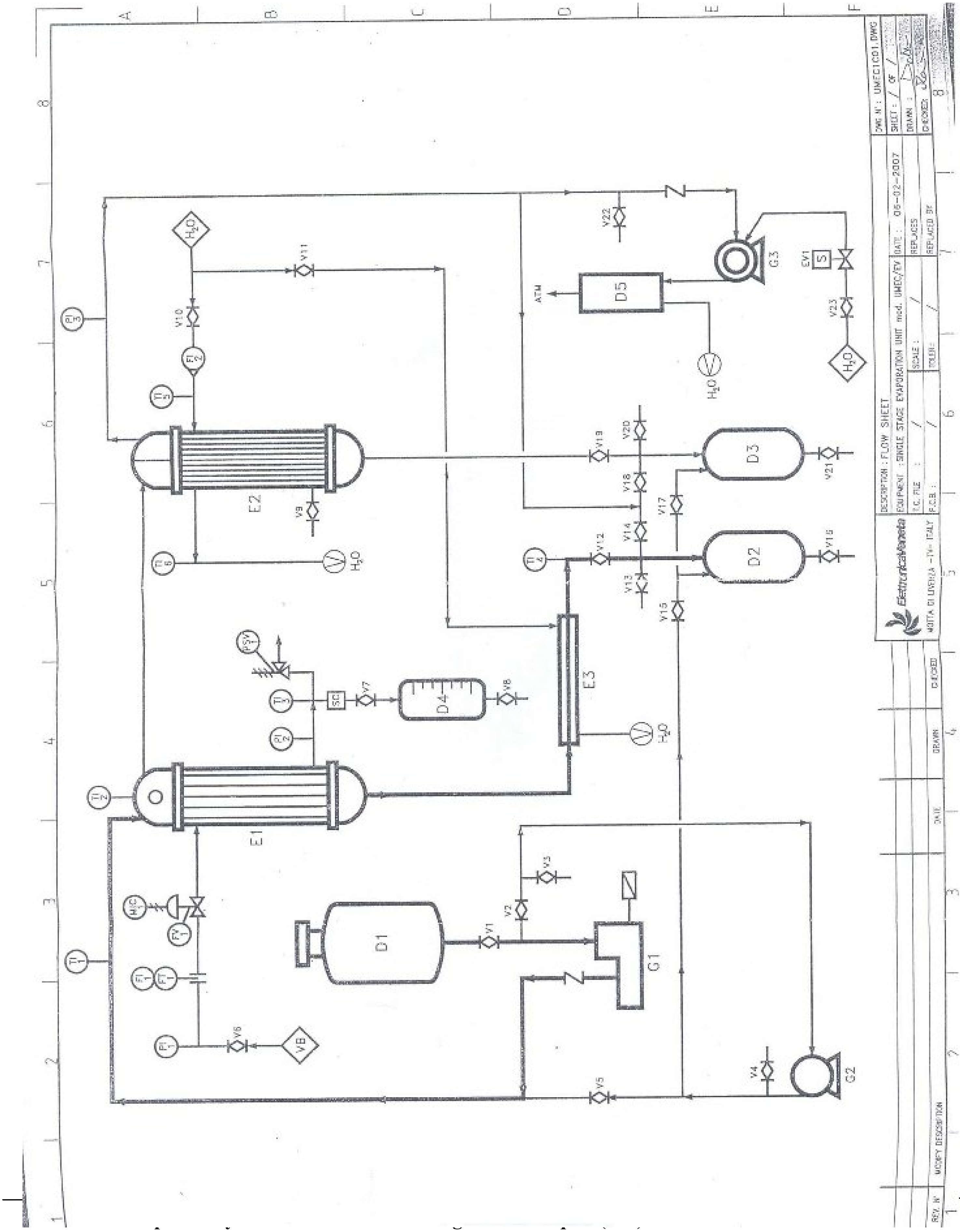
4.3 Concentration of milk

- 15 litres of whole milk are treated in the following operative conditions:

Feeding flow rate, F_i	
Steam flow rate, FI1	
Steam pressure, PI1	
Steam pressure, PI2	
Working pressure, PI3	
Cooling water flow rate, FI2	

- In stationary conditions, we measure 5 litres of concentrated product in D1 and 1 litres of water in tank D3

- The concentration then is increased of 17%


DWG N° : UMEC1C01.DWG
SHEET : 1 OF 1
DRAWN :
CHECKED :
DESCRIPTION : FLOW SHEET
EQUIPMENT : SINGLE STAGE EVAPORATION UNIT mod. UMEC/EV
DATE : 05-02-2007
Elettronica Veneta
MOTTA DI LIVENZA -TV- ITALY
D1
D2
D3
D4
D5
E1
E2
E3
G1
G2
G3
EV1
PSV 1
SC
HIC 1
FV 1
FI 1
FT 1
PI 1
PI 2
PI 3
TI 1
TI 2
TI 3
TI 4
TI 5
TI 6
FI 2
VB
H2O
ATM
V1
V2
V3
V4
V5
V6
V7
V8
V9
V10
V11
V12
V13
V14
V15
V16
V17
V18
V19
V20
V21
V22
V23
A
B
C
D
E
F
1
2
3
4
5
6
7
8